# Water and Air Sample Data Documentation

## Introduction

During OMEX I the daunting total of 478 different parameters were measured on water or air samples by 48 principal investigators using a wide range of protocols. The aim of this document is to allow the protocol used to obtain any particular data value within the BOTDATA 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**

#### Carbon, Nitrogen and Phosphorus Assimilation

Data from <sup>14</sup>C, <sup>15</sup>N and <sup>32</sup>P short duration uptake experiments where the results having been expressed in terms of uptake per hour, together with parameterised PvI data (alpha and Pmax).

#### **Metal Assimilation Rates**

Data from assimilation experiments using gamma-emitting isotopes of trace metals.

#### **Metal Distribution Coefficients**

Trace metal distribution coefficients between the dissolved and particulate phases measured using gamma-emitting isotopes.

#### **Bacterial Production, Abundance and Characteristics**

Bacterial abundance, biomass and size data plus thymidine and leucine uptake data.

#### **Carbon and Nitrogen Isotopes**

Carbon isotope data on dissolved inorganic carbon plus carbon and nitrogen data on particulate organic matter.

#### **Dissolved and Colloidal Organic Carbon**

High temperature catalytic oxidation measurements.

#### **Dissolved Total Nitrogen and Phosphorus**

Provide the basis for the determination of dissolved organic nitrogen and phosphorus for samples that have nutrient data.

## Particulate Organic Carbon, Inorganic Carbon, Nitrogen, Phosphorus and Silica

The parameters often loosely described as 'POC' and 'PON' plus inorganic carbon, phosphorus and biogenic (opaline) silica data.

#### **Nutrients**

Nitrate plus nitrite, nitrite, phosphate, silicate, ammonia and urea data.

#### **Dissolved and Particulate Carbohydrates**

Total carbohydrate in the dissolved and particulate phases.

#### **Amino Acids and Fatty Acids**

Total free amino acids plus some seventy individual fatty acids.

#### **Carbonate System Parameters**

Dissolved total inorganic carbon,  $pCO_2$  in water and the atmosphere, alkalinity and pH.

#### **Dissolved and Colloidal Trace Metals**

Aluminium, cadmium, cobalt, copper, total iron, total manganese, nickel, lead and zinc data.

#### Particulate Trace Metals

Aluminium, calcium, cadmium, cobalt, chromium, copper, total iron, potassium, lithium, magnesium, total manganese, sodium, nickel, lead, silicon and zinc contents of suspended particulate material.

#### **Pigments**

Chlorophyll-a determined by a range of techniques, including data derived from calibrated in-situ fluorometers, plus a full suite of pigments determined by HPLC.

#### **Suspended Particulate Material Concentration and Characterisation**

Gravimetric SPM determinations plus some particle size data.

#### **Dimethylsulphide and its Precursors**

Dimethylsulphide plus DMSP and DMSO.

#### Carbonyl Sulphide.

Atmospheric concentrations, seawater concentrations and production data.

#### Methane

Dissolved and atmospheric methane data.

#### **Atmospheric Ammonia and Methylamines**

Gaseous and particulate atmospheric data.

#### **Dissolved Methylamines**

Monomethylamine, dimethylamine and trimethylamine concentrations.

#### **Dissolved Oxygen**

Dissolved oxygen concentrations including data derived from calibrated insitu oxygen probe data.

#### Hydrography

Temperature, salinity, density and attenuance data that have largely been derived from CTD data together with calibration bottle salinity and reversing thermometer data.

#### Irradiance

Light meter data at bottle firing depths.

#### Volume of Water Filtered

Records of the volume of water sampled by stand-alone pumps and underway centrifuges.

#### **Microzooplankton Biomass and Grazing**

Microzooplankton abundance, biomass and grazing together with data on heterotrophic and photosynthetic nanoflagellates.

#### **Phytoplankton Species Counts**

Abundance of phytoplankton taxa.

#### **Zooplankton and Terrestrial Detritus**

Zooplankton abundance by taxonomic group together with pollen and fungal detritus counts.

#### Radionuclides

Plutonium, barium, strontium and americium isotope data.

#### **Atmospheric Radon**

Determinations of radon through monitoring of its decay products.

#### **Current Parameters**

Parameterised data from current meters attached to a benthic water sampling system.

#### References

Full references for the papers cited in the protocol descriptions.

## Carbon, Nitrogen and Phosphorus Assimilation

## **Parameter Code Definitions**

- ALPHPIP1 Quantum yield (alpha) PvI incubation (GF/F filtered) mg C/(μE/m<sup>2</sup>/s)/mg chl/hour
- NAUPRAP1 Normalised ammonium uptake (100  $\mu$ E/m<sup>2</sup>/s) Tracer-doped constant light incubation at 100  $\mu$ E/m<sup>2</sup>/s (GF/F filtered) Nanomoles per litre per hour
- NAUPRBP1 Normalised ammonium uptake (188 μE/m<sup>2</sup>/s) Tracer-doped constant light incubation at 188 μE/m<sup>2</sup>/s (GF/F filtered) Nanomoles per litre per hour
- NAUPRDP1 Normalised ammonium uptake (dark) Tracer-doped incubation in darkness (GF/F filtered) Nanomoles per litre per hour
- NAUPRSP1 Normalised ammonium uptake (natural light) Tracer-doped incubation in natural sunlight (GF/F filtered) Nanomoles per litre per hour
- NCUPRAP1 Normalised carbon uptake (100 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 100 μE/m<sup>2</sup>/s (GF/F filtered) Milligrams/metre cube/hour
- NCUPRAP4 Normalised carbon uptake (100  $\mu$ E/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 100  $\mu$ E/m<sup>2</sup>/s (sum size fractions >0.2 microns) Milligrams/metre cube/hour
- NCUPRBP1 Normalised carbon uptake (188 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 188 μE/m<sup>2</sup>/s (GF/F filtered) Milligrams/metre cube/hour

- NCUPRBP4 Normalised carbon uptake (188 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 188 μE/m<sup>2</sup>/s (sum size fractions >0.2 microns) Milligrams/metre cube/hour
- NCUPRDP1 Normalised carbon uptake (dark) Radiotracer doped incubation in the dark (GF/F filtered) Milligrams/metre cube/hour
- NCUPRDP4 Normalised carbon uptake (dark) Radiotracer doped incubation in the dark (sum size fractions >0.2 microns) Milligrams/metre cube/hour
- NCUPRPP1 Normalised carbon uptake (azide control) Radiotracer doped azide poisoned control incubation (GF/F filtered) Milligrams/metre cube/hour
- NCUPRPP4 Normalised carbon uptake (azide control) Radiotracer doped azide poisoned control incubation (sum size fractions >0.2 microns) Milligrams/metre cube/hour
- NCUPRSP1 Normalised carbon uptake (natural light) Radiotracer doped incubation in natural sunlight (GF/F filtered) Milligrams/metre cube/hour
- NCUPRSP4 Normalised carbon uptake (natural light) Radiotracer doped incubation in natural sunlight (sum size fractions >0.2 microns) Milligrams/metre cube/hour
- NCUPRZP1 Normalised carbon uptake (188 μE/m<sup>2</sup>/s with antibiotic) Radiotracer doped constant light incubation at 188 μE/m<sup>2</sup>/s with antibiotic (GF/F filtered) Milligrams/metre cube/hour
- NCUPRZP4 Normalised carbon uptake (188  $\mu$ E/m<sup>2</sup>/s with antibiotic) Radiotracer doped constant light incubation at 188  $\mu$ E/m<sup>2</sup>/s with antibiotic (sum size fractions >0.2 microns) Milligrams/metre cube/hour
- NNUPRAP1 Normalised nitrate uptake (100 μE/m<sup>2</sup>/s) Tracer-doped constant light incubation at 100 μE/m<sup>2</sup>/s (GF/F filtered) Nanomoles per litre per hour

- NNUPRBP1 Normalised nitrate uptake (188 μE/m<sup>2</sup>/s) Tracer-doped constant light incubation at 188 μE/m<sup>2</sup>/s (GF/F filtered) Nanomoles per litre per hour
- NNUPRDP1 Normalised nitrate uptake (dark) Tracer-doped incubation in darkness (GF/F filtered) Nanomoles per litre per hour
- NNUPRSP1 Normalised nitrate uptake (natural light) Tracer-doped incubation in natural sunlight (GF/F filtered) Nanomoles per litre per hour
- NPUPRAP1 Normalised phosphorus uptake (100 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 100 μE/m<sup>2</sup>/s (GF/F filtered) Nanomoles per litre per hour
- NPUPRAP4 Normalised phosphorus uptake (100  $\mu$ E/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 100  $\mu$ E/m<sup>2</sup>/s (sum size fractions >0.2 microns) Nanomoles per litre per hour
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- NPUPRSP1 Normalised phosphorus uptake (natural light) Radiotracer doped incubation in natural sunlight (GF/F filtered) Nanomoles per litre per hour
- NPUPRSP4 Normalised phosphorus uptake (natural light) Radiotracer doped incubation in natural sunlight (sum size fractions >0.2 microns) Nanomoles per litre per hour
- NPUPRXP4 Normalised phosphorus uptake (100  $\mu$ E/m<sup>2</sup>/s with antibiotic) Radiotracer doped constant light incubation at 100  $\mu$ E/m<sup>2</sup>/s (sum size fractions >0.2 microns) Nanomoles per litre per hour
- NPUPRYP1 Normalised phosphorus uptake (dark with antibiotic) Radiotracer doped incubation in the dark with antibiotic (GF/F filtered) Nanomoles per litre per hour
- NPUPRYP4 Normalised phosphorus uptake (dark with antibiotic) Radiotracer doped incubation in the dark with antibiotic (sum size fractions >0.2 microns) Nanomoles per litre per hour
- NPUPRZP1 Normalised phosphorus uptake (188 μE/m<sup>2</sup>/s with antibiotic) Radiotracer doped constant light incubation at 188 μE/m<sup>2</sup>/s (GF/F filtered) Nanomoles per litre per hour
- NPUPRZP4 Normalised phosphorus uptake (188  $\mu$ E/m<sup>2</sup>/s with antibiotic) Radiotracer doped constant light incubation at 188  $\mu$ E/m<sup>2</sup>/s (sum size fractions >0.2 microns) Nanomoles per litre per hour
- NUUPRBP1 Normalised urea uptake (188 μE/m<sup>2</sup>/s) Tracer-doped constant light incubation at 188 μE/m<sup>2</sup>/s (GF/F filtered) Nanomoles per litre per hour
- PMAXPIP1 Photosynthetic maximum (Pmax) PvI incubation (GF/F filtered) mg C/mg chl/hour

- SNAURSPM Size-fractionated normalised ammonium uptake (natural light) Tracer doped incubation in natural sunlight (GF/F- 5µm size fraction) Nanomoles per litre per hour
- SNCURAPB Size-fractionated normalised carbon uptake (100 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 100 μE/m<sup>2</sup>/s (>2μm size fraction) Milligrams/metre cube/hour
- SNCURAPF Size-fractionated normalised carbon uptake (100 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 100 μE/m<sup>2</sup>/s (0.2-2μm size fraction) Milligrams/metre cube/hour
- SNCURBPB Size-fractionated normalised carbon uptake (188 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 188 μE/m<sup>2</sup>/s (>2μm size fraction) Milligrams/metre cube/hour
- SNCURBPF Size-fractionated normalised carbon uptake (188 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 188 μE/m<sup>2</sup>/s (0.2-2μm size fraction) Milligrams/metre cube/hour
- SNCURDPB Size-fractionated normalised carbon uptake (dark) Radiotracer doped incubation in the dark (>2µm size fraction) Milligrams/metre cube/hour
- SNCURDPF Size-fractionated normalised carbon uptake (dark) Radiotracer doped incubation in the dark (0.2-2µm size fraction) Milligrams/metre cube/hour
- SNCURPPB Size-fractionated normalised carbon uptake (azide control) Radiotracer doped azide poisoned control incubation (>2µm size fraction) Milligrams/metre cube/hour
- SNCURPPF Size-fractionated normalised carbon uptake (azide control) Radiotracer doped azide poisoned control incubation (0.2-2µm size fraction) Milligrams/metre cube/hour
- SNCURSPB Size-fractionated normalised carbon uptake (natural light) Radiotracer doped incubation in natural sunlight (>2µm size fraction) Milligrams/metre cube/hour

- SNCURSPF Size-fractionated normalised carbon uptake (natural light) Radiotracer doped incubation in natural sunlight (0.2-2µm size fraction) Milligrams/metre cube/hour
- SNCURZPB Size-fractionated normalised carbon uptake (188 μE/m<sup>2</sup>/s with antibiotic) Radiotracer doped incubation at 188 μE/m<sup>2</sup>/s with antibiotic (>2μm size fraction) Milligrams/metre cube/hour
- SNCURZPF Size-fractionated normalised carbon uptake (188 μE/m<sup>2</sup>/s with antibiotic) Radiotracer doped incubation at 188 μE/m<sup>2</sup>/s with antibiotic (0.2-2μm size fraction) Milligrams/metre cube/hour
- SNNURSPM Size-fractionated normalised nitrate uptake (natural light) Tracer doped incubation in natural sunlight (GF/F- 5µm size fraction) Nanomoles per litre per hour
- SNPURAPB Size-fractionated normalised phosphorus uptake (100  $\mu$ E/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 100  $\mu$ E/m<sup>2</sup>/s (>2 $\mu$ m size fraction) Nanomoles per litre per hour
- SNPURAPF Size-fractionated normalised phosphorus uptake (100  $\mu$ E/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 100  $\mu$ E/m<sup>2</sup>/s (0.2-2 $\mu$ m size fraction) Nanomoles per litre per hour
- SNPURBPB Size-fractionated normalised phosphorus uptake (188 μE/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 188 μE/m<sup>2</sup>/s (>2μm size fraction) Nanomoles per litre per hour
- SNPURBPF Size-fractionated normalised phosphorus uptake (188  $\mu$ E/m<sup>2</sup>/s) Radiotracer doped constant light incubation at 188  $\mu$ E/m<sup>2</sup>/s (0.2-2 $\mu$ m size fraction) Nanomoles per litre per hour
- SNPURDPB Size-fractionated normalised phosphorus uptake (dark) Radiotracer doped incubation in the dark (>2µm size fraction) Nanomoles per litre per hour

- SNPURDPF Size-fractionated normalised phosphorus uptake (dark) Radiotracer doped incubation in the dark (0.2-2µm size fraction) Nanomoles per litre per hour
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- SNPURSPF Size-fractionated normalised phosphorus uptake (natural light) Radiotracer doped incubation in natural sunlight (0.2-2µm size fraction) Nanomoles per litre per hour
- SNPURXPB Size-fractionated normalised phosphorus uptake (100  $\mu$ E/m<sup>2</sup>/s with antibiotic) Radiotracer doped incubation at 100  $\mu$ E/m<sup>2</sup>/s with antibiotic (>2 $\mu$ m size fraction) Nanomoles per litre per hour
- SNPURXPF Size-fractionated normalised phosphorus uptake (100 μE/m²/s with antibiotic)
  Radiotracer doped incubation at 100 μE/m²/s with antibiotic(0.2-2μm size fraction)
  Nanomoles per litre per hour
- SNPURYPB Size-fractionated normalised phosphorus uptake (dark with antibiotic) Radiotracer doped incubation in the dark with antibiotic (>2μm size fraction) Nanomoles per litre per hour
- SNPURYPF Size-fractionated normalised phosphorus uptake (dark with antibiotic) Radiotracer doped incubation in the dark with antibiotic (0.2-2μm size fraction) Nanomoles per litre per hour

- SNPURZPB Size-fractionated normalised phosphorus uptake (188  $\mu$ E/m<sup>2</sup>/s with antibiotic) Radiotracer doped incubation at 188  $\mu$ E/m<sup>2</sup>/s with antibiotic (>2 $\mu$ m size fraction) Nanomoles per litre per hour
- SNPURZPF Size-fractionated normalised phosphorus uptake (188  $\mu$ E/m<sup>2</sup>/s with antibiotic) Radiotracer doped incubation at 188  $\mu$ E/m<sup>2</sup>/s with antibiotic (0.2-2 $\mu$ m size fraction) Nanomoles per litre per hour

## **Originator Code Definitions**

#### Belgica cruises BG9309, BG9322, BG9412, BG9506 and BG9521

10	Ir. Marc Elskens	VUB, Brussels, Belgium
14	Dr. Lei Chou	ULB, Brussels, Belgium
72	Prof. Roland Wollast	ULB, Brussels, Belgium

#### Belgica cruise BG9522

10	Ir. Marc Elskens	VUB, Brussels, Belgium
72	Prof. Roland Wollast	ULB, Brussels, Belgium

#### **Discovery cruise DI217**

3 Dr. Ian Joint Plymouth Marine Laboratory

#### **Originator Protocols**

#### Ir. Marc Elskens

Labelled nitrate, urea and ammonia (99%  $^{15}\rm{N}$ ) were added to sea water samples in 2000 or 700 ml polycarbonate bottles. Tracer additions were kept as low as possible whilst still facilitating accurate measurements. Ambient levels were increased by 0.1 and 0.05  $\mu M$  for nitrate and ammonia respectively.

Incubation conditions were 10-20 hours in constant light of 100  $\mu$ E/m<sup>2</sup>/s on cruises BG9309, BG9322 and BG9412 and 188  $\mu$ E/m<sup>2</sup>/s on cruises BG9506, BG9521 and BG9522. Incubator temperature was controlled by continuously flushing with surface sea water.

At the end of the incubation the samples were filtered (Whatman GF/F) and converted to nitrogen gas by a modified Dumas method. Isotope detection

was carried out by emission spectrometry (Fiedler and Proksh, 1975) using either a Jasco NIA-1 or N-151 analyser. High-purity tank nitrogen gas was used as a working standard during sample analysis.

#### Dr. Lei Chou

Water samples were collected using water bottles deployed on a CTD rosette. 200 ml aliquots were doped with 11.9  $\mu$ Ci <sup>14</sup>C and 20-30 (BG9309, BG9412, BG9521) or 3.2-12 (BG9322)  $\mu$ Ci <sup>32</sup>P (as carrier-free H<sub>3</sub><sup>32</sup>PO<sub>4</sub>).

The spiked samples were then incubated for between 6 and 20 hours under one or more of the following conditions:

Constant light (100  $\mu$ E/m<sup>2</sup>/s on cruises BG9309, BG9322 and BG9412: 188  $\mu$ E/m<sup>2</sup>/s on cruises BG9506, BG9521) Full sunlight Total darkness Azide poisoned Constant light as above with antibiotic (10% polymyxin B sulphate, 10% streptomycin sulphate: 100  $\mu$ I per 200ml sample) Total darkness with above antibiotic

Note that a number of samples, some with antibiotic added, were incubated in constant light with bottles sandwiched between neutral density filters to give a light gradient. These data have not been parameterised into  $P_{max}$  and alpha. Consequently, they cannot be mapped into the BOTDATA structure and the data may be found with the other non-parameterised production profiles elsewhere in the database (tables C14HDR, C14DAT, P33HDR and P33DAT).

Temperature of incubation was controlled by a bath of pumped surface seawater. The incubation conditions for any data point may be determined from the parameter code.

At the end of the incubation the samples were filtered using GF/F or 2 micron and 0.2 micron Nuclepore filter cascade to obtain size-fractionated data. The filtration protocol may again be determined from the parameter code used.

Uptake rates were computed on the basis of the following ambient concentrations:

Cruise	Station	Depth(m)	PO4(μM)	TCO <sub>2</sub> (mM)
BG9309	GC10b	5	0.2	2.048
BG9309	GC10b	40	0.37	2.055
BG9309	GC11	5	0.07	2.091
BG9322	GC3	3	0.059	2.223
BG9322	GC5	3	0.032	2.071
BG9322	GC5	50	0.027	

Cruise	Station	Depth(m)	PO4(μM)	TCO <sub>2</sub> (mM)
BG9322	GC11	35	0.107	2.222
BG9412	OX01A	20	0.028	2.257
BG9412	OX02	20	0.167	2.261
BG9412	OX03	20	0.035	2.300
BG9412	OX04	20	0.402	2.309
BG9412	OX05	20	0.135	2.290
BG9412	OX06	20	0.436	2.293
BG9412	OX08	20	0.390	2.295
BG9412	OX15A	20	0.437	2.282
BG9412	OXHO	10	0.616	2.139
BG9412	OXCS	5	0.201	21100
BG9412	OXCS	20	0.206	
BG9412	OXCS	30	0.238	
BG9412	OXCS	40	0.272	
BG9412	OXCS	60	0.555	
BG9412	OXCS	80	0.705	
BG9506	OX01	20	0.255	2.018
BG9506	OX03	3	0.382	2.079
BG9506	OX03	20	0.389	2.077
BG9506	OX03	40	0.387	2.076
BG9506	OX04	20	0.395	2.075
BG9506	OX05	20	0.423	2.076
BG9506	OX06	20	0.425	2.077
Cruise	Station	Depth(m)	ΡΟ4(μΜ)	alkalinity(mEq/l)
BG9521	01A	3	0.041	2.309
BG9521	01A	20	0.014	2.312
BG9521	01A	45	0.790	2.312
BG9521	02A	3	0.011	2.312
BG9521	02A	20	0.005	2.311
BG9521	02A	35	0.377	2.316
BG9521	04A	3	0.027	2.331
BG9521	04A	40	0.041	2.332
BG9521	05C	3	0.081	2.318
BG9521	05C	20	0.104	2.316
BG9521	05C	35	0.099	2.319
BG9521	05C	60	0.162	2.319
BG9521	05C	75	0.162	2.317
BG9521	05E	30	0.100	2.319
BG9521	07A	3	0.050	2.335
BG9521	07A	40	0.079	2.334
BG9521	08A	3	0.072	2.347
BG9521	08A	40	0.168	2.323
BG9521	09A	3	0.043	2.331
BG9521	09A	20	0.040	
	USA	20	0.040	2.332
BG9521	09A 09A	40	0.058	2.332 2.331

Cruise	Station	Depth(m)	ΡΟ4(μΜ)	alkalinity(mEq/l)
BG9521	10A	3	0.050	2.332
BG9521	10A	20	0.024	2.331
BG9521	10A	40	0.086	2.330
BG9521	11A	3	0.067	2.332
BG9521	11A	20	0.029	2.330
BG9521	11A	35	0.168	2.330

Note that the carbon uptake rates were supplied in units of  $\mu$ M/hour. These have been converted to mg/m<sup>3</sup>/hour by multiplying the data by 12.011.

#### Prof. Roland Wollast

Constant light incubations were at a light intensity of 100  $\mu$ E/m<sup>2</sup>/s on cruises BG9309, BG9322, and BG9412. This was increased to 188  $\mu$ E/m<sup>2</sup>/s on cruises BG9506, BG9521 and BG9522. Temperature was controlled by continuously circulating surface seawater. Samples were GF/F filtered at the end of the incubation.

Photosynthesis versus irradiance experiments were performed in either 200 or 600 ml culture bottles in an artificial light gradient from 0 to 800  $\mu$ E/m<sup>2</sup>/s in a bath maintained at constant temperature by circulating surface seawater. Incubation times were limited to 6-8 hours. The relationship between <sup>14</sup>C uptake and light intensity has been parameterised following the model of Platt et al. (1980).

Data were supplied in either  $\mu$ M/hour or mg/m<sup>3</sup>/hour. Where necessary, the data have been standardised to the latter unity by multiplying by 12.011.

#### Dr. Ian Joint

Water samples were collected using GoFlo bottles deployed on the CTD rosette. Replicate samples were distributed into clear polycarbonate bottles and  $^{15}NO_3$  and  $^{15}NH_4$  were added. The concentrations of added isotope were kept as low as practicable (0.03  $\mu M$ ).

The samples were incubated for approximately 6 hours in an on-deck incubator at 97% ambient light at sea surface temperature.

The samples were filtered (<40cm Hg vacuum) through pre-ashed Whatman GF/F filters which were rinsed with filtered sea water and stored frozen until analysis back at the laboratory. The size-fractionated data were obtained by filtering through a 5 micron pore size polycarbonate filter placed over a GF/F filter in a filter cascade. The 5 micron filter was jettisoned and the GF/F filter analysed to give the GF/F to 5 micron size class.

The thawed filters were oven-dried at 50 °C before analysis. Atomic percentage <sup>15</sup>N was measured by continuous-flow nitrogen analysis mass spectrometry (Europa Scientific Ltd.) using the techniques described by Barrie et al. (1989) and Owens and Rees (1989). The rates of assimilation were calculated using the equations of Dugdale and Goering (1967).

## **Metal Assimilation Rates**

#### **Parameter Code Definitions**

- CDRURAP2 Cadmium relative uptake rate (100  $\mu$ E/m<sup>2</sup>/s) Tracer doped constant light incubation 100  $\mu$ E/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour
- CDRURBP2 Cadmium relative uptake rate (188 μE/m<sup>2</sup>/s) Tracer doped constant light incubation 188 μE/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour
- CDRURDP2 Cadmium relative uptake rate (dark) Tracer doped incubation in the dark (0.45 micron pore filtered) Parts per thousand per hour
- CORURAP2 Cobalt relative uptake rate (100  $\mu$ E/m<sup>2</sup>/s) Tracer doped constant light incubation 100  $\mu$ E/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour
- CORURBP2 Cobalt relative uptake rate (188 μE/m<sup>2</sup>/s) Tracer doped constant light incubation 188 μE/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour
- CORURDP2 Cobalt relative uptake rate (dark) Tracer doped incubation in the dark (0.45 micron pore filtered) Parts per thousand per hour
- CSRURAP2 Caesium relative uptake rate (100  $\mu$ E/m<sup>2</sup>/s) Tracer doped constant light incubation 100  $\mu$ E/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour
- MNRURAP2 Manganese relative uptake rate (100  $\mu$ E/m<sup>2</sup>/s) Tracer doped constant light incubation 100  $\mu$ E/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour
- MNRURBP2 Manganese relative uptake rate (188 μE/m<sup>2</sup>/s) Tracer doped constant light incubation 188 μE/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour

- MNRURDP2 Manganese relative uptake rate (dark) Tracer doped incubation in the dark (0.45 micron pore filtered) Parts per thousand per hour
- ZNRURAP2 Zinc relative uptake rate (100  $\mu$ E/m<sup>2</sup>/s) Tracer doped constant light incubation 100  $\mu$ E/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour
- ZNRURBP2 Zinc relative uptake rate (188 μE/m<sup>2</sup>/s) Tracer doped constant light incubation 188 μE/m<sup>2</sup>/s (0.45 micron pore filtered) Parts per thousand per hour
- ZNRURDP2 Zinc relative uptake rate (dark) Tracer doped incubation in the dark (0.45 micron pore filtered) Parts per thousand per hour

## **Originator Code Definitions**

#### Belgica cruises BG9412, BG9521 and BG9522

72 Professor Roland Wollast ULB, Brussels, Belgium

#### **Originator Protocols**

#### **Professor Roland Wollast**

Water samples were taken from GoFlo bottles and immediately spiked with  $^{54}$ Mn,  $^{60}$ Co,  $^{65}$ Zn,  $^{109}$ Cd and (for a few of the stations)  $^{137}$ Cs. The samples were incubated for 6-8 hours at a constant light level of 100  $\mu E/m^2/s$  (BG9412), 188  $\mu E/m^2/s$  (BG9521 and BG9522) or in darkness. Incubation temperature was controlled by continuously flushing with surface sea water.

After vacuum filtration on 0.45 micron membrane filters, the radioactivity of the filters and acidified filtrates was measured with an HPGe Camberra detector, with a relative efficiency of 20%, and a series 20, model 282 multichannel spectrometer. The minimum number of counts was fixed at 1000 to reduce the standard deviation to 3%.

To ensure that perturbation of the natural conditions was kept to a minimum, concentrations of the radionuclides and their carriers were maintained at a minimum level. The activity of the spike was usually approximately 100 nCi/l for each individual radionuclide. However, to obtain this level, the concentration of the corresponding dissolved metal was raised by 5-10 nM due to the presence of the carrier. In the area investigated, the ambient

concentration of the trace metals considered was of the order of 0.02 nM for Co, 0.08 nM for Cd, 0.7 nM for Mn and 5 nM for Zn in the upper 100m of the water column. The increase in trace metal concentrations due to the addition of the tracers were therefore significant. For the 1995 field experiment (cruise BG9521 and BG9522), supplies of unsupported Mn and Cd radionuclides were obtained and used.

## **Metal Distribution Coefficients**

## **Parameter Code Definitions**

- CDKDRAX2 Cadmium fast distribution coefficient (100  $\mu$ E/m<sup>2</sup>/s) Radiotracer incubation at 100  $\mu$ E/m<sup>2</sup>/s: phases split by 0.45 micron membrane filtration Litres per kilogram
- COKDRAX2 Cobalt fast distribution coefficient (100 μE/m<sup>2</sup>/s) Radiotracer incubation at 100 μE/m<sup>2</sup>/s: phases split by 0.45 micron membrane filtration Litres per kilogram
- CSKDRAX2 Caesium fast distribution coefficient (100  $\mu$ E/m<sup>2</sup>/s) Radiotracer incubation at 100  $\mu$ E/m<sup>2</sup>/s: phases split by 0.45 micron membrane filtration Litres per kilogram
- MNKDRAX2 Manganese fast distribution coefficient (100 μE/m<sup>2</sup>/s) Radiotracer incubation at 100 μE/m<sup>2</sup>/s: phases split by 0.45 micron membrane filtration Litres per kilogram
- ZNKDRAX2 Zinc fast distribution coefficient (100  $\mu$ E/m<sup>2</sup>/s) Radiotracer incubation at 100  $\mu$ E/m<sup>2</sup>/s: phases split by 0.45 micron membrane filtration Litres per kilogram

## **Originator Code Definitions**

#### Belgica cruises BG9412, BG9521 and BG9522

72 Professor Roland Wollast ULB, Brussels, Belgium

#### **Originator Protocols**

#### **Professor Roland Wollast**

The Fast Distribution Coefficient, or  $K_{FDC}$  (Mouchel and Martin, 1990), is defined as the ratio, found after the reaction time, of the particulate activity to the dissolved activity of the added radioisotope, normalised to the suspended matter concentration.

Water samples were taken from GoFlo bottles and immediately spiked with  $^{54}$ Mn,  $^{60}$ Co,  $^{65}$ Zn,  $^{109}$ Cd and (for a few of the stations)  $^{137}$ Cs. The samples were incubated for 8 hours at a constant light level of 100  $\mu E/m^2/s$  (BG9412). Incubation temperature was controlled by continuously flushing with surface sea water.

After vacuum filtration on 0.45 micron membrane filters, the radioactivity of the filters and acidified filtrates was measured with an HPGe Camberra detector, with a relative efficiency of 20%, and a series 20, model 282 multichannel spectrometer. The minimum number of counts was fixed at 1000 to reduce the standard deviation to 3%.

To ensure that perturbation of the natural conditions was kept to a minimum, concentrations of the radionuclides and their carriers were maintained at a minimum level. The activity of the spike was usually approximately 100 nCi/l for each individual radionuclide. However, to obtain this level, the concentration of the corresponding dissolved metal was raised by 5-10 nM due to the presence of the carrier. In the area investigated, the ambient concentration of the trace metals considered was of the order of 0.02 nM for Co, 0.08 nM for Cd, 0.7 nM for Mn and 5 nM for Zn in the upper 100m of the water column. The increase in trace metal concentrations due to the addition of the tracers were therefore significant.

## Bacterial Production, Abundance and Characteristics

#### **Parameter Code Definitions**

- BATTMAPZ Proportion of total bacteria attached to particles Epifluorescence microscopy with acridine orange stain Per cent
- SDLERIP4 Standard deviation of leucine uptake rate Isotope doped, incubated, filtered (0.2 µm pore filter) and counted Picomoles/litre/hour
- SDTBMDPZ Standard deviation of total bacteria Microscopy (DAPI stain) Number per millilitre
- SDTHRIP4 Standard deviation of thymidine uptake rate Isotope doped, incubated, filtered (0.2 µm pore filter) and counted Picomoles/litre/hour
- TBBMMAPZ Total bacteria biomass as carbon Calculated from cell counts determined by epifluorescence microscopy with acridine orange stain Milligrams per cubic metre
- TBCCMAPZ Total bacteria cell numbers Microscopy (acridine orange stain) Number per millilitre
- TBCCMDPZ Total bacteria cell numbers Microscopy (DAPI stain) Number per millilitre
- TBMSIAPZ Median size of total bacteria Image analysis of acridine orange stained sample Micrometres (microns)
- UPLERIP4 Leucine uptake rate Isotope doped, incubated, filtered (0.2 µm pore filter) and counted Picomoles/litre/hour

UPTHRIP4 Thymidine uptake rate Isotope doped, incubated, filtered (0.2 µm pore filter) and counted Picomoles/litre/hour

## **Originator Code Definitions**

#### Valdivia cruise VLD137 and Discovery cruise DI217

3 Dr. Ian Joint Plymouth Marine Laboratory, UK

#### Pelagia cruises PLG93 and PLG95A

96 Dr. Laurenz Thomsen GEOMAR, Kiel, Germany

#### **Originator Protocols**

#### Dr. lan Joint

#### **Bacterial Production**

Bacterial production was estimated from the rates of incorporation of [methyl-<sup>3</sup>H] thymidine and of L-[4,5-H<sup>3</sup>] leucine (specific activities 79 Ci/mmol and 171 Ci/mmol respectively; Amersham International plc, UK). Leucine stocks were routinely diluted 1:3 with unlabelled leucine. Stock radiotracer solutions were prepared using sterilised glassware and stored in pharmaceutical-grade serum bottles that had been pre-treated by filling with 0.25 molar Analar grade HCl, left to stand for three days, rinsed with Milli-Q water, filled with Milli-Q water and left to stand for a further two days. Serum bottles and their Teflon-lined silicone seals were autoclaved before use. Stock radiotracer solutions were prepared in sterile, 0.2 micron filtered, Milli-Q water and stored at 2°C. A fresh stock bottle was used for each experiment.

Tritiated thymidine incorporation experiments followed the methods of Fuhrman and Azam (1982) and the leucine incorporation experiments followed the methods of Simon and Azam (1989), modified to include the cold trichloroacetic acid (TCA) extraction method of Chin-Leo and Kirchman (1988). Five replicate, 10 ml aliquots from each depth sampled were transferred to sterile, polystyrene, tissue-culture tubes and placed in an incubator in the dark, at in-situ temperatures and allowed to acclimatise for 15 minutes prior to the addition of the isotope. Electron microscope grade glutaraldehyde was added to one replicate sample from each depth at a final concentration of 2.5% by volume to act as controls. <sup>3</sup>H-thymidine or <sup>3</sup>H-leucine was added to each tube to give final concentrations of 5 and 10 nM respectively.

The samples were incubated for one hour, but time-course assays showed that incorporation was linear for two hours and frequently longer.

At the end of the incubation, samples were transferred to an ice/water bath and ice-cold TCA added to give a final concentration of 5% by volume. The samples were left in the water bath for 15-30 minutes and filtered through 25mm 0.2 micron pore-size, track-etched, polycarbonate membrane filters. Each filter was rinsed five times with 1ml 5% ice-cold TCA, placed in a scintillation vial and stored in a desiccator with active silica gel for 24 hours. At the end of this period, the samples were counted in an LKB Rackbeta 1219 liquid scintillation counter. Counting efficiency was determined by an external standard, channels ratio method and checked by the occasional addition of internal standards.

#### Bacterial abundance

Samples were fixed with 2.5% by volume, 0.2 micron filtered, electron microscope grade glutaraldehyde, stained immediately with DAPI (4'6-diamidino-2-phenylindole) as described by Porter and Feig (1980) and filtered.

Samples were either examined immediately or stored frozen at -20 °C until examined back at the laboratory. Fluorescent bacteria were counted with an epifluorescence microscope by the method of Hobbie et al. (1977). The microscope used was a Leitz Ortholux II equipped with a 50W HBO light source, Ploempak 2.2 fluorescence vertical illuminator with filter block A and an NPL Fluorotar 100/1.32 oil objective lens.

#### Dr. Laurenz Thomsen

Water samples were collected using the BIOPROBE benthic water sampling lander (Thomsen et al., 1994). This was deployed on a conductor cable and gently positioned on the sea bed with approximately 20m of slack cable. Penetration into the sediment was determined by a graduated rod monitored by a video camera.

After the material disturbed by the instrument deployment had been seen from transmissometer readings to have dispersed, water samples were collected by pumping into sample bottles on a command signal from the ship. Sampling inlets were positioned at different heights on the instrument enabling water at different heights from the seabed to be collected. Further samples were collected with the lander raised at different heights, generally 5m or 50m, above the sea floor.

Bacterial cell numbers were determined by the acridine orange direct counting technique of Hobbie et al. (1977) using a Zeiss 'Standard' fluorescence microscope. Cell numbers were supplied in units of 10<sup>7</sup> cells per litre and were converted to cells per millilitre by multiplying by 10,000.

Bacteria size was estimated using a Macintosh Power PC image analysis system according to the method of Thomsen (1991). A carbon conversion factor of 0.4 picograms of carbon per cubic micrometre was used to determine biomass from cell counts. Further details of the protocol may be found in Thomsen and Graf (1995).

## **Carbon and Nitrogen Isotopes**

#### **Parameter Code Definitions**

- D13CMITX Total inorganic carbon  $(TCO_2)^{13}$ C enrichment  $(\delta^{13}C)$ Mass spectrometry on acid-liberated  $CO_2$ Parts per thousand
- D13CMOPC Particulate organic carbon  $^{13}$ C enrichment ( $\delta^{13}$ C) Mass spectrometry on acidified combusted sample (centrifuged) Parts per thousand
- D15NMTPC Particulate total nitrogen ("PON")  $^{15}$ N enrichment ( $\delta^{15}$ N) Mass spectrometry on combusted sample (centrifuged) Parts per thousand

#### **Originator Code Definitions**

#### Cruises Belgica 9309, 9322, 9412

- 10 Ir. Marc Elskens VUB, Brussels, Belgium
- 30 Dr. Patrick Dauby University of Liege, Belgium

#### Cruise Meteor M27\_1

73 Prof. Robin Keir GEOMAR, Kiel, Germany

#### **Originator Protocols**

#### Ir. Marc Elskens

Suspended particulate matter was collected by continuous flow centrifugation using an Alpha-Laval oil purifier (model MAB 104) specially coated for oceanographic use. Water supply was adjusted to approximately 1 cubic metre per hour. Samples were collected both when the ship was on station and steaming between stations for about 6-10 hours.

Samples were taken from the centrifuge body using a stainless steel spatula, stored in acid-washed PET vials and immediately deep frozen. After weighing (wet weight) the sample was subdivided for C/N, trace metal and isotope analysis.

The samples for  $\delta^{15}$ N determination (~20 mg dry material) were decarbonated by acidification and the particulate nitrogen converted to dinitrogen by a modified Dumas method (Owens, 1987) using L-shaped quartz tubes. The tubes were attached to the inlet system of a Mass spectrometer Delta E Finnigan immersed in liquid nitrogen to trap carbon dioxide and water before analysis. Determinations of  $\delta^{15}$ N were undertaken using high purity nitrogen from a tank as the primary standard.

The data are expressed as:

$$\delta^{15} N = [(R_{sample}/R_{standard}) - 1] * 1000$$

where R is the <sup>15</sup>N/<sup>14</sup>N ratio. IAEA-N1 and IAEA-N2 reference materials were used as working standards. Final results are expressed relative to atmospheric nitrogen (Mariotti, 1983).

#### Dr. Patrick Dauby

Suspended particulate matter was collected by continuous flow centrifugation using an Alpha-Laval oil purifier (model MAB 104) specially coated for oceanographic use. Water supply was adjusted to approximately 1 cubic metre per hour. Samples were collected both when the ship was on station and steaming between stations for about 6-10 hours.

Samples were taken from the centrifuge body using a stainless steel spatula, stored in acid-washed PET vials and immediately deep frozen. After weighing (wet weight) the sample was subdivided for C/N, trace metal and isotope analysis.

Sub-samples for  $\delta^{13}$ C determination were decarbonated by acidification then oven dried at 55 °C. The material was ground into a fine powder and vacuum sealed in Pyrex tubes with copper oxide wire. These were heated at 550 °C for a day to complete combustion of the organic matter. The carbon dioxide generated was cryogenically trapped using liquid nitrogen.  $\delta^{13}$ C was determined either on a Varian Mat CH5 or Optima (Micromass) Isotope Ratio Mass Spectrometer, using certified CO<sub>2</sub> gas (Messer Griesheim) as an intermediate standard. All values are reported relative to the international PDB standard as:

$$\delta^{13}C = [(R_{sample}/R_{standard}) - 1] * 1000$$

where R is the  ${}^{13}C/{}^{12}C$  ratio. Routine measurements were reported as accurate to within  $\pm 0.3$  ppt.

#### Prof. Robin Keir

Water samples were drawn from the CTD rosette and returned to Kiel for analysis. The isotopic composition of the inorganic carbon was determined by acidifying with orthophosphoric acid. The  $CO_2$  liberated is stripped by high purity nitrogen and trapped in a loop immersed in liquid nitrogen under rough vacuum. The gas is separated from water vapour by distillation and trapping at controlled temperatures. The purified  $CO_2$  is then analysed using a Finnigan-MAT Delta E gas isotope mass spectrometer.

A manual sample processing line was used for the Meteor 27\_1 samples. A number of samples were contaminated during analysis by isotopically light atmospheric CO<sub>2</sub>. The data from these were removed from the data set prior to submission to BODC.

## **Dissolved and Colloidal Organic Carbon**

#### **Parameter Code Definitions**

- CORGNOD3 Dissolved organic carbon High temperature Ni catalytic oxidation (GF/C filtered) Micromoles/litre
- CORGCOC1 Colloidal organic carbon Difference in Pt HTCO determined DOC between 0.4 µm and 10<sup>4</sup> Dalton filtered samples Micromoles/litre
- CORGCOD1 Dissolved organic carbon High temperature Pt catalytic oxidation (GF/F filtered) Micromoles/litre
- CORGCOD2 Dissolved organic carbon High temperature Pt catalytic oxidation (0.4 µm pore filtered) Micromoles/litre
- CORGCOTX Total organic carbon High temperature Pt catalytic oxidation (unfiltered) Micromoles/litre
- SDOCCOD1 Dissolved organic carbon standard deviation High temperature Pt catalytic oxidation (GF/F filtered) Micromoles/litre
- SEOCCOD1 Dissolved organic carbon standard error High temperature Pt catalytic oxidation (GF/F filtered) Micromoles/litre
- SEOCCOTX Total organic carbon standard error High temperature Pt catalytic oxidation (unfiltered) Micromoles/litre

#### **Originator Code Definitions**

#### Belgica Cruise BG9309

14	Dr. Lei Chou	ULB, Brussels, Belgium
49	Dr. Jean-Marie Martin	Institut de Biogeochimie, France

#### Belgica Cruise BG9322

14 Dr. Lei Chou ULB, Brussels, Belgium

Meteor Cruises M27\_1 and M30\_1

9	Mr. Thomas Raabe	Hamburg University, Germany
51	Prof. Wolfgang Balzer	University of Bremen, Germany

#### Valdivia Cruise VLD137

9	Mr. Thomas Raabe	Hamburg University, Germany

#### **Charles Darwin Cruises CD84 and CD94**

13 Dr. Axel Miller Plymouth Marine Laboratory, UK.

## **Originator Protocols**

#### Dr. Lei Chou

Water samples were collected using either 10 I acid-cleaned polypropylene bottles deployed manually or Niskin/GoFlo bottles deployed on a CTD rosette.

Water bottle samples were filtered using pre-ashed (450 °C) GF/F filters. 10 ml samples were transferred into 15 ml glass ampoules which were poisoned with 100  $\mu$ l of 1 g/l HgCl<sub>2</sub> and sealed.

Back at the laboratory the samples were assayed using a Shimadzu TOC-5000 analyser.

#### Dr. Jean-Marie Martin

Water samples were collected using either 10 I acid-cleaned polypropylene bottles deployed manually or Teflon lined GoFlo bottles deployed on a CTD rosette. The samples were filtered under nitrogen pressure through acid-cleaned 0.4 micron Nuclepore filters. 1-2 litres were passed through the filter and discarded to guard against adsorption and/or release of organic carbon from the filter.

A 50ml sample was poisoned with  $HgCl_2$  for DOC analysis. Filtered water was then passed through a cross-flow ultra-filtration (CFF) system with a polysulphate membrane (10<sup>4</sup> Daltons) to separate colloids from the truly dissolved fraction. Quadruplate samples were collected when the concentration factor was around 4-6. All filtration was conducted using ultraclean techniques under laminar-flow clean benches. DOC was determined on the filtrate and ultra-filtrate by high temperature catalytic oxidation using a Shimadzu TOC-5000 analyser. Samples were injected into a furnace at 680 °C onto a catalyst made of 1.2% Pt coated SiO<sub>2</sub>. The CO<sub>2</sub> produced was measured by a non-dispersive infra-red (NDIR) detector. The instrument and water blanks were evaluated for each set of sample analysis as described by Cauwet (1994).

#### Mr. Thomas Raabe

Water samples were taken from the CTD rosette and filtered through Whatman GF/C filters. The filtrate was poisoned with mercuric chloride and stored in glass and polythene bottles in a cooling chamber until analysed. Samples were analysed by high temperature catalytic oxidation using a nickel catalyst.

#### Prof. Wolfgang Balzer

Water samples were taken from the CTD rosette and filtered under ultraclean conditions through pre-combusted GF/F filters. The filtrate was then acidified, sealed in brown glass ampoules and stored at 4 °C until analysed. Samples were analysed by high temperature catalytic oxidation. Samples were analysed in triplicate.

#### Dr. Axel Miller

Samples were taken from the CTD rosette and generally filtered through GF/F filters, although some samples in low particulate waters were analysed unfiltered to assess filtration as a source of contamination. Ultra-clean handling techniques were used throughout.

The analytical technique involves the direct injection of acidified and decarbonated sea water onto a platinised alumina catalyst at high temperature (680-900 °C) under an atmosphere of oxygen or high purity air. Quantitative production of  $CO_2$  gas allows DOC concentrations to be determined using a  $CO_2$ -specific infrared gas analyser (IRGA).

Analyses were undertaken at sea using a Shimadzu TOC-5000 HTCO analyser fitted with a LiCor Li6252 IRGA. This overcame the problems associated with using the standard TOC-5000 IRGA on an unstable platform.

Great care was taken to quantify blank signals generated at all stages of the analytical procedure and to correct the data for them.

A more detailed description of the protocols followed may be found in Miller et al. (1993).

## Comments on Data Quality

#### Belgica 9309

The ULB data set contains a number of high values of up to nearly 6000  $\mu$ M. Of particular concern are the data from the upper 300m from station 11 which jump from 138  $\mu$ M at 400m to 2403  $\mu$ M at 300m and some of the data from the Zodiac transects. The IBM data from station 11 (but a different cast) give values of 1-200  $\mu$ M in the upper 200m and their values for most samples from the rias are significantly lower than the ULB data.

The ULB values from the upper 300m of station 11 have been flagged suspect, together with samples from the rias in excess of 400  $\mu$ M. The possibility of contamination of some samples has been suggested by the data originator as the cause of the problem. Values in the range 200-400  $\mu$ M have been left unflagged but users should bear in mind that they may also be contaminated to some extent.

#### Meteor 27\_1 and 30\_1

The bulk of the water samples was analysed for DOC by both Bremen and Hamburg universities. The Hamburg data are systematically 2-3 times higher than the Bremen data. The cause of this is unknown. The Bremen data exhibit excellent agreement with the PML data collected on cruise Charles Darwin 84 which went to sea the day after Meteor 27\_1 docked.

The Hamburg data set included 6 samples from Meteor 27\_1, usually from rosette position 1, which showed exceptionally high DOC values. These have been flagged suspect. Three unusually high values (5-10 times the Bremen value) from Meteor 30\_1 have also been flagged suspect.

#### Valdivia 137

The DOC values from this cruise are systematically higher than those obtained by other groups working in the OMEX project. On the two Meteor cruises, Hamburg and Bremen determined DOC on the same samples. The Hamburg results were consistently 2-3 times higher than the Bremen data.

A small number of data points from VLD137 are extremely high, with the odd sample exceeding 2000  $\mu$ M. Values in excess of 400  $\mu$ M have been flagged suspect.

## Dissolved Total Nitrogen and Phosphorus (from which Dissolved Organic Nitrogen and Phosphorus may be Determined)

#### **Parameter Code Definitions**

- NTOTCOD1 Dissolved total nitrogen High temperature Pt catalytic oxidation (GF/F filtered) Micromoles/litre
- NTOTWCD3 Dissolved total nitrogen Oxidation then autoanalysis (GF/C filtered) Micromoles/litre
- SETNCOD1 Dissolved total nitrogen standard error High temperature Pt catalytic oxidation (GF/F filtered) Micromoles/litre
- TPHSWCD3 Dissolved total phosphorus Oxidation then autoanalysis (GF/C filtered) Micromoles/litre

## **Originator Code Definitions**

Meteor cruises M27\_1 and M30\_1, Valdivia cruise VLD137 and Discovery cruise DI217

9 Mr. Thomas Raabe Hamburg University, Germany

#### **Charles Darwin Cruise CD94**

13	Dr. Axel Miller	Plymouth Marine Laboratory, UK
9	Mr. Thomas Raabe	Hamburg University, Germany

#### **Originator Protocols**

#### Mr. Thomas Raabe

Water samples were taken from the CTD rosette and filtered through Whatman GF/C filters. The filtrate was poisoned with mercuric chloride and stored in glass and polythene bottles in a cooling chamber until analysed.

The samples were oxidised by peroxodisulphate in an autoclave (Eberlein and Kattner, 1987) followed by nitrate/phosphate determination.

#### Dr. Axel Miller

Samples were taken from the CTD rosette or surface sea water supply and filtered through GF/F filters. Ultra-clean handling techniques were used throughout.

The analytical technique involves the direct injection of acidified and decarbonated sea water onto a platinised alumina catalyst at high temperature (680-900 °C) under an atmosphere of oxygen or high purity air. Quantitative production of the nitric oxide radical allows total dissolved nitrogen concentrations to be determined using a nitrogen-specific chemiluminescence detector.

Analyses were undertaken at sea using a Shimadzu TOC-5000 HTCO analyser fitted with an Antek 705-D chemiluminescence detector. The combustion products travelled through a Drierite trap (97% CaSO<sub>4</sub>, 3% CoCl<sub>3</sub>) and a membrane (permeation tube) drier to remove any trace of water. The dried nitric acid radical was then reacted with ozone to produce the excited chemiluminescent nitrogen species and passed to the detector. Each sample was injected four times with each injection cycle taking 5.5 minutes.

Great care was taken to quantify blank signals generated at all stages of the analytical procedure and to correct the data for them.

## Particulate Organic Carbon, Inorganic Carbon, Nitrogen, Phosphorus and Silica

#### **Parameter Code Definitions**

- CINGWLP1 Particulate inorganic carbon Weight loss on acidification (GF/F filtered) Micromoles/litre
- CORGCAP1 Particulate organic carbon (acidified) Acid fumed then C/N analyser (GF/F filtered) Micromoles/litre
- CORGCAP4 Particulate organic carbon (acidified) Acid fumed then C/N analyser (30 µm pore filtered) Micromoles/litre
- CORGCNP3 Particulate organic carbon (unacidified) Carbon/nitrogen analyser (GF/C filtered) Micromoles/litre
- ICCNCNP2 Inorganic carbon content (filtered SPM) Difference between C/N analyser results on total and acidified samples (0.4/0.45 μm pore filtered) Per cent
- ICCNCNPC Inorganic carbon content (centrifuged SPM) Difference between C/N analyser results on total and acidified samples (centrifuged) Per cent
- NTOTCNP1 Particulate total nitrogen ("PON") Carbon/nitrogen analyser (GF/F filtered) Micromoles/litre
- NTOTCNP4 Particulate total nitrogen ("PON") Carbon/nitrogen analyser (30 µm pore filtered) Micromoles/litre
- NTOTCNP3 Particulate total nitrogen ("PON") Carbon/nitrogen analyser (GF/C filtered) Micromoles/litre
- OCCNCAP1 Organic carbon content (GF/F filtered SPM) Acidification then carbon/nitrogen analyser (GF/F filtered) Per cent
- OCCNCAP2 Organic carbon content (0.45 micron pore filtered SPM) Acidification then carbon/nitrogen analyser (0.4/0.45 µm pore filtered) Per cent
- OCCNCAPC Organic carbon content (centrifuged SPM) Acidification then carbon/nitrogen analyser (centrifuged) Per cent
- OPALWCP7 Particulate opaline silica NaOH hydrolysis of material trapped on a cellulose acetate filter Micromoles/litre
- TNCNCNP2 Total nitrogen content (0.45 micron pore filtered SPM) Carbon/nitrogen analyser (0.4/0.45 µm pore filtered) Per cent
- TNCNCNPC Total nitrogen content (centrifuged SPM) Carbon/nitrogen analyser (centrifuged) Per cent
- TPHSWCP3 Particulate total phosphorus Oxidation then autoanalysis (GF/C filtered) Micromoles/litre

## **Originator Code Definitions**

#### Belgica cruises BG9309, BG9322 and BG9412

10	Ir. Marc Elskens	VUB, Brussels, Belgium
14	Dr. Lei Chou	ULB, Brussels, Belgium
30	Dr. Patrick Dauby	University of Liege, Belgium

#### **Belgica cruise BG9506**

10	Ir. Marc Elskens	VUB, Brussels, Belgium
14	Dr. Lei Chou	ULB, Brussels, Belgium

#### Belgica cruises BG9521 and BG9522

10	Ir. Marc Elskens	VUB, Brussels, Belgium
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## **Charles Darwin cruise CD84**

14 Dr. Lei Chou ULB, Brussels, Belgium Meteor cruises M27 1 and M30 1 9 Mr. Thomas Raabe Hamburg University, Germany 96 Dr. Laurenz Thomsen GEOMAR, Kiel, Germany Charles Darwin cruise CD94 and Valdivia cruise VLD137 9 Mr. Thomas Raabe Hamburg University, Germany Pelagia cruises PLG93 and PLG95A and Charles Darwin cruise CD86 96 Dr. Laurenz Thomsen GEOMAR, Kiel, Germany **Charles Darwin cruise CD85** 7 Dr. Avan Antia University of Kiel, Germany **Discovery cruise DI217** 7 Dr. Avan Antia University of Kiel, Germany 9 Mr. Thomas Raabe Hamburg University, Germany Jan Mayen cruises JM1-JM11 61 Dr. Paul Wassmann University of Tromsø, Norway **Charles Darwin cruise CD83** 39 Mr. Bob Head Plymouth Marine Laboratory, UK Auriga cruises PLUTUR2-PLUTUR5 and Andromeda cruise PLUTUR6 91 Dr. Aurora Rodrigues Instituto Hidrografico, Portugal

## **Originator Protocols**

## Ir. Marc Elskens

Sea water samples from Niskin or GoFlo bottles were filtered on precombusted Whatman GF/F filters. The filters were air dried at 60 °C and kept at room temperature until analysed. The samples were treated with HCl vapour to remove carbonates and analysed using a Carlo Erba NA1500 elemental analyser. The CO<sub>2</sub> and N<sub>2</sub> were separated by means of a gas chromatographic column (Poropak QS) and measured by thermal conductivity detection.

## Dr. Lei Chou

Samples were obtained using one of two protocols. The protocol used may be identified by the gear code in the EVENT entry for the data (SAP or GPCENT).

## SAP collection

Challenger Oceanics in-situ stand-alone pumps (SAPs) were used to sample particulate material. The instruments are operated by a programmable timer to ensure that the pump only operates when in position at the desired depth. Membrane filters with a 0.4 micron pore size were used to collect the particulate material.

On recovery the filters were rinsed and dried in clean conditions.

## GPCENT collection.

Suspended particulate matter was collected by continuous flow centrifugation using an Alpha-Laval oil purifier (model MAB 104) specially coated for oceanographic use. Water supply was adjusted to approximately 1 cubic metre per hour. Samples were collected both when the ship was on station and steaming between stations for about 6-10 hours.

Samples were taken from the centrifuge body using a stainless steel spatula, stored in acid-washed PET vials and immediately deep frozen. After weighing (wet weight) the sample was subdivided for C/N, trace metal and isotope analysis.

If sufficient material was available, a sample for carbon and nitrogen determination was acidified to remove carbonates and then assayed in an Interscience NA-2000 elemental particulate analyser.

Inorganic carbon content was determined where sufficient material was available by analysing both acidified and unacidified samples and computing the difference.

## Dr. Patrick Dauby

Suspended particulate matter was collected by continuous flow centrifugation using an Alpha-Laval oil purifier (model MAB 104) specially coated for oceanographic use. Water supply was adjusted to approximately 1 cubic metre per hour. Samples were collected both when the ship was on station and steaming between stations for about 6-10 hours.

Samples were taken from the centrifuge body using a stainless steel spatula, stored in acid-washed PET vials and immediately deep frozen. After weighing (wet weight) the sample was subdivided for C/N, trace metal and isotope analysis.

Back in the laboratory, sub-samples were dried and weighed, slightly acidified to remove carbonates, rinsed, oven dried and ground into a fine powder. Determinations of C and N were performed with a Carlo Erba NA1500 elemental analyser.

The data were supplied to BODC in the form of organic carbon content in parts per thousand and molar C/N ratio. The units were converted to percentages and the nitrogen contents computed from the molar ratio assuming atomic weights for carbon and nitrogen of 12.011 and 14.007 respectively.

## Mr. Thomas Raabe

Water samples taken from the CTD rosette were filtered through Whatman GF/C filters and kept at -17 °C until analysed back at the laboratory using a high combustion CHN-analyser.

Phosphorus was determined by persulphate digestion followed by determination as orthophosphate using the methods of Kattner and Brockmann (1980).

## Dr. Avan Antia

Water samples were taken from either the bottles on the CTD rosette or from large (30 litre) GoFlo bottles deployed from the hydrographic winch. Aliquots were filtered through GF/F filters for carbon determinations and cellulose acetate filters for biogenic silica determinations. Inorganic carbon was measured gravimetrically through weight loss on acidification. Organic carbon was determined on samples with the inorganic carbon removed using a CHN analyser. Biogenic silica was determined by wet chemical methods after hydrolysis of the sample.

The data were supplied in various units. For Charles Darwin CD85, organic carbon was supplied in units of  $\mu$ g/l. This was converted to  $\mu$ M through division by 12.011.

For Discovery DI217, organic carbon was supplied in mg/l and inorganic carbon was supplied in mg/l of CaCO<sub>3</sub>. Inorganic carbon in  $\mu$ M was computed by multiplying the CaCO<sub>3</sub> value by 1000 then dividing by 100.0892. The organic carbon was converted by multiplying by 1000 and dividing by 12.011.

## Dr. Paul Wassmann

Water samples were taken from the CTD rosette and filtered through precombusted Whatman GF/F filters and analysed on a Leeman lab CHN analyser after removal of carbonate.

The data were supplied in units of  $\mu$ g/l. Carbon and nitrogen were converted to  $\mu$ M by dividing by 12.011 and 14.007 respectively.

## Mr. Bob Head

Replicate 500 ml aliquots were taken from CTD rosette bottles or the underway non-toxic sea water supply. After an initial screening through a 200 micron mesh, to prevent spurious results caused by large zooplankton, the samples were filtered through 25mm GF/F filters. Additional aliquots were taken on some stations and filtered through 30 micron pore filters to give additional data for the >30 micron size fraction. Samples were frozen at -20 °C until analysed back at the laboratory.

The samples were acidified with sulphur dioxide to remove carbonates and then dried at 50 °C for 2 days. The samples were then encapsulated in squares of pre-combusted aluminium foil in a 4.5mm press.

The samples were analysed in a Carlo Erba NA1500 elemental analyser at a reactor temperature of 1030 °C and a helium carrier flow rate of 120 ml per minute. Calibration was effected with standards of acetanilide assayed on a calibrated Cahn 25 balance. Filter and sea water blanks were analysed and used to correct the data.

The data were supplied in units of  $\mu$ g/l. Carbon and nitrogen were converted to  $\mu$ M by dividing by 12.011 and 14.007 respectively.

## Dr. Aurora Rodrigues

Water samples were collected using a portable pump and filtered through Whatman GF/F filters. Organic carbon was determined at the University of Bordeaux using the method of Strickland and Parsons (1972) as adapted by Etcheber (1982). Samples were treated with 2N HCl to remove carbonates and assayed using a LECO CS-125 analyser.

The POC values were supplied in units of  $\mu$ g/l and converted to  $\mu$ M by dividing by 12.011.

## Dr. Laurenz Thomsen

Water samples were collected using the BIOPROBE benthic water sampling lander (Thomsen et al., 1994). This was deployed on a conductor cable and gently positioned on the sea bed with approximately 20m of slack cable.

Penetration into the sediment was determined by a graduated rod monitored by a video camera.

After the material disturbed by the instrument deployment had been seen from transmissometer readings to have dispersed, water samples were collected by pumping into sample bottles on a command signal from the ship. Sampling inlets were positioned at different heights on the instrument enabling water at different heights from the seabed to be collected. Further samples were collected with the lander raised at different heights, generally 5m or 50m, above the sea floor.

The water samples were filtered on GF/F filters, acidified to remove carbonates and analysed using a Heraeus CHN analyser. Further details of the protocol are given in Thomsen and Graf (1995).

The data were supplied in units of  $\mu g/l$ . Carbon and nitrogen were converted to  $\mu M$  by dividing by 12.011 and 14.007 respectively.

## **Comments on Data Quality**

## **Belgica Cruises**

Comparison of the carbon and nitrogen contents between the centrifuged samples and shallow SAP samples was possible at two stations. The values compared well.

Both ULB and Liege determined organic carbon and total nitrogen content on the centrifuged samples. An intercalibration of the two data sets by BODC showed excellent agreement. Regressing one data set against the other gave the following results:

Carbon	ULB = 1.0051 * Liege + 0.7038 (R <sup>2</sup> = 92%; n=37)
Nitrogen	ULB = $1.0062 * \text{Liege} + 0.225$ (R <sup>2</sup> = 90%; n=34)

# **Nutrients**

# Parameter Code Definitions

AMONAAD2	Dissolved ammonium Colorometric autoanalysis (0.4/0.45 µm pore filtered) Micromoles/litre
AMONAATX	Dissolved ammonium Colorometric autoanalysis (unfiltered) Micromoles/litre
AMONMATX	Ammonium (unfiltered) Manual colorometric analysis (unfiltered) Micromoles/litre
NTRIAAD2	Dissolved nitrite Colorometric autoanalysis (0.4/0.45 µm pore filtered) Micromoles/litre
NTRIAAD5	Dissolved nitrite Colorometric autoanalysis (0.2 µm pore filtered) Micromoles/litre
NTRIAATX	Nitrite (unfiltered) Colorometric autoanalysis (unfiltered) Micromoles/litre
NTRZAAD2	Dissolved nitrate + nitrite Colorometric autoanalysis (0.4/0.45 µm pore filtered) Micromoles/litre
NTRZAAD5	Dissolved nitrate + nitrite Colorometric autoanalysis (0.2 µm pore filtered) Micromoles/litre
NTRZAATX	Nitrate + nitrite (unfiltered) Colorometric autoanalysis (unfiltered) Micromoles/litre
PHOSAAD2	Dissolved phosphate Colorometric autoanalysis (0.4/0.45 µm pore filtered) Micromoles/litre

- PHOSAAD5 Dissolved phosphate Colorometric autoanalysis (0.2 µm pore filtered) Micromoles/litre
- PHOSAATX Phosphate (unfiltered) Colorometric autoanalysis (unfiltered) Micromoles/litre
- PHOSMATX Phosphate (unfiltered) Manual colorometric analysis (unfiltered) Micromoles/litre
- SLCAAAD2 Dissolved silicate Colorometric autoanalysis (0.4/0.45 µm pore filtered) Micromoles/litre
- SLCAAD5 Dissolved silicate Colorometric autoanalysis (0.2 µm pore filtered) Micromoles/litre
- SLCAAATX Silicate (unfiltered) Colorometric autoanalysis (unfiltered) Micromoles/litre
- SLCAMATX Silicate (unfiltered) Manual colorometric analysis (unfiltered) Micromoles/litre
- UREAMDTX Urea (unfiltered) Manual analysis using the diacetylmonoxime method Micromoles/litre

## **Originator Code Definitions**

## Belgica cruise BG9309

10	Ir. Marc Elskens	VUB, Brussels, Belgium
14	Dr. Lei Chou	ULB, Brussels, Belgium
66	Dr. Ricardo Prego	IIM, CSIC, Vigo, Spain

## Belgica cruises BG9322, BG9412, BG9506, BG9521 and BG9522

10	Ir. Marc Elskens	VUB, Brussels, Belgium
14	Dr. Lei Chou	ULB, Brussels, Belgium

## Cruises Pelagia PLG93, Charles Darwin CD86 and Pelagia PLG95B

11	Dr. Wim Helder	NIOZ, Texel, the Netherlands		
	Meteor cruises M27_1 and M30_1, Valdivia Cruise VLD137 and Discovery cruise DI217			
9	Mr. Thomas Raabe	Hamburg University, Germany		
Char	les Darwin cruise CD83			
39	Mr. Bob Head	Plymouth Marine Laboratory, UK		
Char	les Darwin cruise CD84			
12 14	Dr. David Hydes Dr. Lei Chou	Southampton Oceanography Centre, UK ULB, Brussels, Belgium		
Char	les Darwin cruise CD85			
3	Dr. Ian Joint	Plymouth Marine Laboratory, UK		
Char	les Darwin cruise CD94			
9 53	Mr. Thomas Raabe Prof. Mike Orren	Hamburg University, Germany University College Galway, Ireland.		
Disco	overy cruise DI216			
12 14 53	Dr. David Hydes Dr. Lei Chou Prof. Mike Orren	Southampton Oceanography Centre, UK ULB, Brussels, Belgium University College Galway, Ireland.		
Jan M	Mayen cruises JM1-JM11			
61	Dr. Paul Wassmann	University of Tromsø, Norway		
Poseidon cruise PS211				
70	Dr. Ludger Mintrop	IfM Kiel, Germany		
Heincke cruise HEINK68 and Valdivia cruise VLD153				
90	Dr. Pete Bowyer	University College Galway, Ireland.		

# Madorniña cruises MD0994, MD0495, MD0695, MD0795, MD0995, MD1095, MD1195

66 Dr. Ricardo Prego IIM, CSIC, Vigo, Spain

## **Originator Protocols**

#### Ir. Marc Elskens

Water samples were taken from manually filled bottles deployed from an inflatable boat away from Belgica (ria surveys) or taken from water bottles deployed on a CTD rosette. On two cruises (9322 and 9412) continuous underway measurements were made by drawing discrete samples at frequent intervals from the continuous seawater supply. Note that these data are stored in the underway binary merge files and not in the BOTDATA table.

Nutrient determinations were carried out on board ship, immediately after sampling. Nitrate plus nitrite and phosphate were determined using a Technicon AA2 autoanalyser as described by Elskens and Elskens (1989).

Ammonia was determined according to the manual method using indophenol blue described in Koroleff (1969) using a Baush and Lomb Spectronic 21 spectrophotometer.

Urea was determined using the diacetymonoxime method of Mulvena and Savidge (1992) modified to allow precise analyses when strict control of the reaction temperature is impossible as described by Goeyens et al. (submitted 1996).

## Dr. Lei Chou

Manual spectrophotometric analyses for phosphate, nitrite and silicate were done using the methods specified in Grasshoff et al. (1983). These analyses were usually carried out on board ship as soon after sampling as possible. Samples were kept refrigerated and dark between collection and analysis.

Samples for nutrient determination by autoanalysis were kept frozen until analysed. A separate set of samples were usually taken specifically for silicate analysis and stored in the dark, chilled but not frozen. Samples were analysed on a SKALAR autoanalyser.

## Dr. Ricardo Prego

Nitrate plus nitrite was determined using a Technicon AAII autoanalyser with the adaptation described in Mouriño and Fraga (1985). Phosphate and silicate were determined using a Technicon AAI autoanalyser following the method described by Hansen and Grasshoff in Grasshoff et al. (1983).

## Dr. Wim Helder

Samples were taken from water bottles deployed on a CTD rosette and analysed at sea using a TRAACS 80 autoanalyser, usually within 4 hours of collection. Samples were stored in cool and dark conditions between collection and analysis.

On cruise Pelagia 93, the samples from the CTD rosette were analysed unfiltered. On Charles Darwin 86 and Pelagia 95, the samples were filtered through a 0.45 micron acrodisc filter to improve the quality of the ammonium results.

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<sub>2</sub>, 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 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. In cases where multiple bottles were fired at a single depth, nutrient values were reported from each bottle. These have been averaged, excluding any bottles flagged as leaking, to give a single nutrient value for each depth.

## Mr. Thomas Raabe

Water samples were taken from bottles deployed on a CTD rosette and analysed immediately on board ship. Samples were analysed unfiltered, provided the particulate content was not considered too high in which case the samples were GF/C filtered. Parameter coding has assumed analysis of unfiltered samples.

Samples were analysed using a Technicon autoanalyser system using the method of Murphy and Riley (1962) as modified by Eberlein and Kattner (1987) for phosphate, the method of Grasshoff (1983) for silicate, the method of Koroleff (1969) for ammonia and the methods of Armstrong et al. (1967) for nitrate and nitrite.

## Dr. David Hydes

Samples were collected from either bottles on the CTD rosette or the continuously pumped surface sea water supply and immediately analysed unfiltered using a Chemlab autoanalyser. Samples were analysed in triplicate and the mean value is stored in the database.

## Professor Mike Orren

Samples were collected from either bottles on the CTD rosette or the continuously pumped surface sea water supply and analysed using an Alpkem autoanalyser. This machine and the chemistries employed for phosphate and silicate were progressively modified during the project in an attempt to obtain reasonable performance. The following modifications were described:

The length of all tubing was reduced to the absolute minimum.

The instrument was thoroughly cleaned with Decon90 before each procedure.

The phosphate determination wavelength was switched to 760 nm, with wash and sample times switched to 60 and 30 seconds respectively.

The wavelength used for silicate was switched to 795 nm. The ascorbic acid reagent was prepared without the recommended acetone addition, the sulphuric acid concentration was doubled and the ammonium molybdate was filtered prior to each procedure.

Samples were generally analysed on board ship but some samples taken towards the end of a cruise had to be analysed back in the laboratory. These were kept in the dark and as cool as possible between collection and analysis.

## Dr. Paul Wassmann

Water samples were taken from the CTD rosette, fixed with 0.2 ml of chloroform and kept cold (4  $^{\circ}$ C) and dark until analysed by autoanalyser following the protocols of Føyn et al. (1981).

## Dr. Ian Joint

Standard autoanalyser methods were used as described in Rees et al. (1995). Nitrite corrected nitrate data were supplied to BODC. The nitrite corrections were removed and the data stored as nitrate+nitrite in the database.

## Mr. Bob Head

Standard autoanalyser methods were used, with a 3-channel (nitrate+nitrite, silicate and phosphate) instrument logged onto chart recorders.

## Dr. Ludger Mintrop

Water samples were taken from the ship's 'moon pool' and immediately frozen. The samples were transferred to the Polarstern and analysed several months after collection using standard photometric methods on a four channel autoanalyser.

Nitrate and nitrite were supplied as separate channels. These were summed by BODC to give the nitrate+nitrite channel stored.

## Dr. Pete Bowyer

Samples were taken from the CTD rosette bottles, filtered using a syringe through 0.2 micron pore filters and immediately frozen. Back in the laboratory, the samples were analysed on an Alpkem autoanalyser (the same instrument used by Professor Mike Orren) with four channels (nitrate, nitrate+nitrite, phosphate and silicate).

The nitrate data supplied to BODC had been corrected by subtraction of nitrite. These were restored to nitrate+nitrite for storage in the database.

## **Comments on Data Quality**

## Belgica cruise BG9309

The SKALAR autoanalyser phosphate data were supplied with a warning that there may be problems. On a number of stations all three laboratories provided phosphates and for a number of stations there were also manually analysed phosphates from ULB. Comparing these data it can be clearly seen that the SKALAR values are frequently way too high. Consequently, the SKALAR phosphate data set has been flagged 'L'.

For the stations where inter-comparison of  $NO_3+NO_2$  data is possible, the ULB data are generally higher than the VUB data which are, in turn, generally higher than the CSIC data. None of the data have been flagged. Users are

advised to retrieve all three data sets and reach their own conclusions about which data to use.

## Belgica cruise BG9412

On this cruise the ULB  $NO_3+NO_2$  data, with the exception of a handful of points, are significantly lower than the VUB data. Differences of 10 per cent and more are predominant throughout the overlapping data set.

The phosphate and nitrite data sets show excellent agreement.

## **Charles Darwin cruise CD83**

Problems with the colorimeter were reported for this cruise, giving rise to variable sensitivity and non-linear calibrations. The problem was circumvented by careful calibration for each individual CTD cast and is not believed to have affected data quality.

## Charles Darwin cruise CD84

Both ULB and SOC measured the nitrate+nitrite profile at the Belgica station. The two data sets show very good agreement.

## Charles Darwin cruise CD85

The nitrate+nitrite data for station 04\_09 exhibited a curious gradient from 7 to 10  $\mu$ M increasing towards the surface in the top 10m. The data points concerned have been flagged suspect as has a single anomalously high ammonium value. Other than these, no problems could be identified in the data set.

## **Charles Darwin cruise CD94**

A subset of the nutrient channels ( $NO_3+NO_2$ ,  $PO_4$  and silicate) were measured by both Hamburg and Galway universities. Both data sets included a small number of anomalous data values. These have been flagged suspect ('M') together with data from bottles where there is strong evidence of contamination through leakage.

The nitrate+nitrite and silicate data from the two groups compare extremely well and no systematic difference between the two data sets could be established. On some casts the Hamburg data were slightly higher whilst on other casts it was the Galway data that were slightly higher.

Regressing the two data sets gave the following results:

Nitrate+nitrite	Galway = Hamburg * 0.9591 + 0.4471	$(R^2 = 98\%)$
Silicate	Galway = Hamburg * 1.0188 - 0.1091	(R <sup>2</sup> = 99%)

The results for phosphate were not as good. The Galway values were systematically significantly lower than the Hamburg data, sometimes by as much as 50%. The intercalibration plot exhibited much more scatter than the plots for the other two nutrients.

Regressing the two data sets gave the following result:

Phosphate Galway = Hamburg \* 
$$0.9234 - 0.0939$$
 (R<sup>2</sup> = 83%)

The Hamburg data compare more favourably with data from other cruises where the phosphate values are believed to be good quality. It is therefore recommended that the Galway phosphates be used with caution, bearing in mind that they are probably low. However, either nitrate+nitrite or silicate data set may be used with confidence.

## **Discovery cruise DI216**

Nutrients were measured by three groups on this cruise: nitrate+nitrite, silicate and phosphate were measured by SOC; phosphate was determined manually by ULB; nitrate+nitrite and silicate were determined by the Galway group.

The ULB and SOC phosphate data show very good agreement. ULB reported some phosphate samples contaminated and these have been flagged 'L' in the database.

The SOC data are believed to be of extremely high quality. Indeed the data were used successfully to identify CTD rosette misfires due to the close proximity of the values from unintentional 'blind duplicates'. The only problem encountered with the SOC data were the nitrate+nitrite values for one cast (CTD4) which were obviously low. This was attributed to the reduction column being poisoned by mercury in an internal standard and the data have been flagged.

The Galway data from CTD bottles were compared with the SOC data and flagged if they deviated from the SOC values by more than 10 per cent. The same 'blind duplicates' described above were analysed by Galway but the replication was very poor. Users are recommended to use the SOC data rather than the Galway data whenever possible.

Samples from the continuous sea water supply were not analysed by SOC. The Galway data are erratic and in many cases incredibly high. With the exception of samples taken on a section up the Channel right up to the Solent, surface nitrate+nitrite values in excess of 0.75  $\mu$ M and silicate values in excess of 1.0  $\mu$ M have been flagged suspect by BODC. The remaining data should be used with caution.

## Poseidon cruise PS211

A small number of the nutrient values were obviously anomalously high for oceanic surface sea water. Nitrites in excess of 0.5  $\mu$ M (plus the associated nitrate+nitrite values), phosphates in excess of 1.5  $\mu$ M and silicates in excess of 5  $\mu$ M were flagged suspect. This affected between 1 and 4 data values in each channel.

There is, however, some concern about the remaining data, particularly the silicates and, to a lesser extent, the nitrate+nitrite channel. The pattern of the data is more uneven than one would expect for surface values, particularly in the lower nutrient waters encountered south of 52 °N. Users are advised to examine the data carefully and make their own judgements on whether further data should be rejected before making use of this data set.

## Heincke cruise HEINK68

A small number of the nitrite values were anomalously high. All values in excess of  $0.5 \mu$ M (four in total) were flagged suspect in the database.

## Valdivia cruise VLD153

A number of isolated values that were obviously anomalous have been flagged suspect in the database.

However, the main problem with the data from this cruise were the nitrites. The values for stations 40-58 and 93-104 were consistently and unrealistically high (0.9-5  $\mu$ M) whereas the values from the remaining stations, apart from a couple of high spikes, were normal. Consultation with the data originator revealed a calibration scaling problem, by a factor of 10, for these samples. On the basis of this information, the nitrite data in the database for the affected stations have been divided by 10.

Note that the uncorrected nitrites were added to the nitrate data to give nitrate+nitrite so as to accurately reverse the correction made by the data originator.

A number of the silicate profiles, particularly stations 76, 82, 83, 86, 88, 90, exhibit oscillating values rather than a progressive increase from depth to surface. The fact that this phenomenon was confined to consecutive samples from one of the three sections raised a question as to whether this was real and not an analytical artefact. Consequently, the profiles have not been flagged.

## **Dissolved and Particulate Carbohydrates**

## **Parameter Code Definitions**

- TOCHWCD3 Total dissolved carbohydrate Photometry (GF/C filtered) Micromoles/litre
- TOCHWCP3 Total particulate carbohydrate Photometry on hydrolysed sample (GF/C filtered) Micromoles/litre

## **Originator Code Definitions**

# Meteor cruises M27\_1 and M30\_1, Valdivia cruise VLD137, Charles Darwin cruise CD94 and Discovery cruise DI217

9 Mr. Thomas Raabe Hamburg University, Germany

## **Originator Protocols**

## Mr. Thomas Raabe

Water samples were taken from the CTD rosette and a quantity sufficient to yield measurable amounts of particulate matter were filtered through Whatman GF/C filters. The filtrate was poisoned with mercuric chloride and stored refrigerated until analysed. The filters were stored at -17 °C until analysed. After hydrolysis, the samples were analysed following the anthrone method (Eberlein and Schutt, 1986).

## **Comments on Data Quality**

## Meteor cruise M27\_1

A number of the dissolved carbohydrate values were negative. These have been flagged suspect.

## Valdivia cruise VLD137

A small number of deep values showed dramatically high values compared to those immediately above and below them. These have been flagged suspect.

# **Amino Acids and Fatty Acids**

## **Parameter Code Definitions**

- F001GCP3 Particulate dodecanoic acid (lauric acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F002GCP3 Particulate 11-methyldodecanoic acid (11-methyllauric acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F003GCP3 Particulate 10-methyldodecanoic acid (10-methyllauric acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F004GCP3 Particulate tridecanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F005GCP3 Particulate 12-methyltridecanoic acid (12-isomyristic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F006GCP3 Particulate 11-methyltridecanoic acid (11-isomyristic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F007GCP3 Particulate tetradecanoic acid (myristic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F008GCP3 Particulate 13-methyltetradecanoic acid (13-methylmyristic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F009GCP3 Particulate 7-tetradecenoic acid (7-myristoleic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F010GCP3 Particulate 12-methyltetradecanoic acid (12-methylmyristic acid) GCMS (GFC filtered) Nanomoles of carbon per litre

- F011GCP3 Particulate 9-tetradecenoic acid (9-myristoleic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F012GCP3 Particulate pentadecanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F013GCP3 Particulate 14-methylpentadecanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F014GCP3 Particulate hexadecanoic acid (palmitic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F015GCP3 Particulate trans-9-hexadecenoic acid (trans-9-palmitoleic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F016GCP3 Particulate cis-9-hexadecenoic acid (cis-9-palmitoleic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F017GCP3 Particulate 14-methylhexadecanoic acid (14-methylpalmitic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F018GCP3 Particulate cis-11-hexadecenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F019GCP3 Particulate heptadecanoic acid (margaric acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F020GCP3 Particulate cis-10,cis-13-hexadecadienoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F021GCP3 Particulate cis-9-heptadecenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F022GCP3 Particulate octadecanoic acid (stearic acid) GCMS (GFC filtered) Nanomoles of carbon per litre

- F023GCP3 Particulate cis-7,cis-10,cis-13-hexadecatrienoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F024GCP3 Particulate trans-6-octadecenoic acid (petroselaidic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F025GCP3 Particulate trans-9-octadecenoic acid (elaidic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F026GCP3 Particulate trans-11-octadecenoic acid (vaccenic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F027GCP3 Particulate cis-6-octadecenoic acid (petroselinic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F028GCP3 Particulate cis-9-octadecenoic acid (oleic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F029GCP3 Particulate cis-11-octadecenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F030GCP3 Particulate 17-methyloctadecanoic acid (17-methylstearic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F031GCP3 Particulate nonadecanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F032GCP3 Particulate cis-9,cis-12-octadecadienoic acid (linoleic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F033GCP3 Particulate cis-12,cis15-octadecadienoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F034GCP3 Particulate cis-9-nonadecenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre

- F035GCP3 Particulate cis-11-nonadecenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F036GCP3 Particulate methylnonadecanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F037GCP3 Particulate eicosanoic acid (arachidic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F038GCP3 Particulate cis-6,cis-9,cis-12-octadecatrienoic acid (gammalinolenic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F039GCP3 Particulate cis-3-eicosenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F040GCP3 Particulate cis-9,cis-12,cis-15-octadecatrienoic acid (linolenic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F041GCP3 Particulate cis-11-eicosenoic acid (gondoic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F042GCP3 Particulate cis-13-eicosenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F043GCP3 Particulate 19-methyleicosanoic acid (19-methylarachidic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F044GCP3 Particulate cis-3,cis-6,cis-9,cis-12-octadecatetranoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F045GCP3 Particulate heneicosanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F046GCP3 Particulate cis-6,cis-9,cis-12,cis-15-octadecatetranoic acid GCMS (GFC filtered) Nanomoles of carbon per litre

- F047GCP3 Particulate cis-11,cis-14-eicosadienoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F048GCP3 Particulate cis-8,cis-11,cis-14-eicosatrienoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F049GCP3 Particulate docosanoic acid (behenic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F050GCP3 Particulate cis-11,cis-14,cis-17-eicosatrienoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F051GCP3 Particulate cis-11-docosenoic acid (cetoleic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F052GCP3 Particulate cis-5,cis-8,cis-11,cis-14-eicosatetraenoic acid (arachidonic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F053GCP3 Particulate cis-13-docosenoic acid (erucic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F054GCP3 Particulate 21-methyldocosanoic acid (21-methylbehenic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F055GCP3 Particulate cis-15-docosenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F056GCP3 Particulate 20-methyldocosanoic acid (20-methylbehenic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F057GCP3 Particulate tricosanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F058GCP3 Particulate cis-13,cis-16-docosadienoic acid GCMS (GFC filtered) Nanomoles of carbon per litre

- F059GCP3 Particulate cis-5,cis-8,cis-11,cis-14,cis-17-eicosapentaenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F060GCP3 Particulate 22-methyltricosanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F061GCP3 Particulate 21-methyltricosanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F062GCP3 Particulate tetracosanoic acid (lignoceric acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F063GCP3 Particulate cis-13,cis-16,cis-19-docosatrienoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F064GCP3 Particulate cis-13-tetracosenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F065GCP3 Particulate 23-methyltetracosanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F066GCP3 Particulate cis-7,cis-10,cis-13,cis-16-docosatetraenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F067GCP3 Particulate 22-methyltetracosanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F068GCP3 Particulate cis-7,cis-10,cis-13,cis-16,cis-19-docosapentaenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F069GCP3 Particulate pentacosanoic acid GCMS (GFC filtered) Nanomoles of carbon per litre

- F070GCP3 Particulate hexacosanoic acid (cerotic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F071GCP3 Particulate cis-4,cis-7,cis-10,cis-13,cis-16,cis-19docosahexaenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F072GCP3 Particulate cis-15-hexacosenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- F073GCP3 Particulate cis-17-hexacosenoic acid (ximenic acid) GCMS (GFC filtered) Nanomoles of carbon per litre
- F074GCP3 Particulate heptacosenoic acid GCMS (GFC filtered) Nanomoles of carbon per litre
- TFAAWCD3 Total dissolved free amino acids Orthopthalic acid reaction / photometry (GF/C filtered) Micromoles/litre

## **Originator Code Definitions**

## Meteor cruises M27\_1 and M30\_1 and Valdivia Cruise VLD137

9 Mr. Thomas Raabe Hamburg University, Germany

## **Originator Protocols**

## Mr. Thomas Raabe

Water samples were taken from the CTD rosette and filtered through Whatman GF/C filters. The filtrate was poisoned with mercuric chloride and stored in glass and polythene bottles in a cooling chamber until analysed. The autoanalyser method of Hammer and Eberlein (1981) was used to determine total free amino acids.

The filters were stored at -17 °C until analysed as follows for a range of fatty acids. All solvents were of analytical grade and re-distilled in all-glass apparatus before use. Glass fibre thimbles were heated at 550 °C in a muffle furnace overnight. All glassware was rinsed with organic solvent before use.

The samples were Soxhlet-extracted in a glass fibre thimble. Water content was reduced by repeated mixing with methanol, settling and extraction of the super-natant into the extraction flask. The samples were Soxhlet-extracted by chloroform/methanol/water azeotrope (42:7:1 v:v:v) under nitrogen within 16 hours. Fatty acid methyl ester (FAME) was added to the sample as an internal standard prior to extraction.

The extracts were rotary-evaporated, transferred into 10-ml Sovirel tubes and evaporated to dryness. 2 ml of 2% sulphuric acid in dry methanol and 0.2 ml of toluene were added and the lipids trans-methylated under argon at 80 °C for 12 hours. The reaction mixture was diluted with 1 ml of double-distilled water and extracted with hexane three times. The organic phases were combined and evaporated to dryness.

The FAME components were isolated from the extracts by thin-layer chromatography on silica gel plates with n-hexane-diethylether-acetic acid (90:10:1 v:v:v) as mobile phase. FAME were recovered by scraping a 3-cm band from the 4-8 cm region (detected by a reference mixture developed on each plate) and eluted with 5 ml of dichloromethane.

GC analysis was performed using a Hewlett-Packard 5710A gas chromatograph, employing a 50 m by 0.3mm internal diameter Silar 10C WCOT glass capillary column with helium carrier gas (0.8 bar), split 1:10, temperature programmed from 120 °C to 220 °C at 4 °C per minute and held at 220 °C for 20 minutes. Quantification was done via the internal standard with a Merck-Hitachi D2000 integrator,

Fatty acid contamination throughout the procedure, as detected by blank analyses, was negligible. Deviation in total fatty acid content was below 2% in duplicate analyses.

FAME components were identified from former results, electron-impact mass spectrometric analyses and ammonia chemical-ionisation mass spectrometry. Double bond location was performed with the 2-amino-2-methylpropanol derivatives of polyunsaturated fatty acids and dimethyl-disulphide derivatives of monounsaturated FAME. Unsaturated and polyunsaturated FAME were separated from the saturated FAME by argentation chromatography.

## **Comments on Data Quality**

## Meteor cruise M27\_1

The dissolved amino acid data set contained a small number of exceptionally high (>10  $\mu$ M) values. These have been interpreted as contaminated and flagged suspect.

## Valdivia cruise VLD137

Dissolved amino acid values in excess of 2  $\mu$ M do not occur consistently in profiles but as isolated spikes. These have been interpreted as contaminated and flagged suspect. 23 out of 428 determinations were affected.

## **Carbonate System Parameters**

## **Parameter Code Definitions**

- ACO2GC01 Atmospheric carbon dioxide partial pressure Gas chromatography Parts per million
- ALKYPOTX Total alkalinity Potentiometry Micro-equivalents per litre
- PCO2C101 pCO<sub>2</sub> Computed from pH and alkalinity Parts per million
- PCO2GC01 pCO<sub>2</sub> Gas chromatography Parts per million
- PHXXPR01 pH pH electrode pH scale per litre
- TCO2C1TX Total dissolved inorganic carbon (TCO<sub>2</sub>) Computed from pH and alkalinity Micromoles/litre
- TCO2MATX Total dissolved inorganic carbon (TCO<sub>2</sub>) Manometrically (unfiltered) Micromoles/litre

## **Originator Code Definitions**

# Belgica cruises BG9309, BG9322, BG9412, BG9506, BG9521 and BG9522

69	Dr. Michel Frankignoulle	University of Liege, Belgium
Mete	eor cruise M27_1	
9 73	Mr. Thomas Raabe Prof. Robin Keir	Hamburg University, Germany GEOMAR, Kiel, Germany

## Meteor cruise M30\_1, Valdivia cruise VLD137 and Discovery cruise DI217

9	Mr. Thomas Raabe	Hamburg University, Germany
Pose	eidon cruise PS211	
73	Prof. Robin Keir	GEOMAR, Kiel, Germany
• •		

# **Originator Protocols**

## **Dr. Michel Frankignoulle**

pH was measured using a combined ROSS electrode and is calibrated on the total proton scale using buffers proposed by Dickson (1993). The error on the pH is estimated to 0.005 pH units.

Total alkalinity was determined by electrotitration (Gran method). Errors on measured alkalinity are estimated to 4  $\mu$ Eq/kg.

Carbon dioxide speciation (TCO<sub>2</sub> and  $pCO_2$ ) has been calculated from alkalinity and pH using CO<sub>2</sub> constants from Goyet and Poisson (1989) for the 1993 and 1994 data and Roy et al. (1993) for the 1995 data. The borate constant is from Hansson (1973) for the 1993 and 1994 data and Dickson (1990) for the 1995 data. The carbon dioxide solubility coefficient is from Weiss (1974) The error on  $pCO_2$  is estimated to be 8-10 ppm.

Further details of the methods used are given in Frankignoulle et al. (1986, 1996).

The alkalinity and TCO<sub>2</sub> data were supplied in units of mEq/kg and millimoles/kg. BODC standard practice is to store parameters in units per litre together with a conversion factor derived from in-situ pressure, temperature and salinity (TOKGPR01) that effects the conversion from litres to kilograms. The database units for these parameters are micromoles rather than millimoles. Consequently, the data supplied had the following transform applied:

Database value = (Original value \* 1000)/TOKGPR01

The pH units supplied were also in terms of per kilogram. The following transform was applied to convert the data into a concentration per litre:

Database value =  $-1.0 \times \log_{10}(10 \times ((\text{Original value} \times -1)/\text{TOKGPR01}))$ 

## Mr. Thomas Raabe

pH values were determined using a WTW pH meter. Note that these are not high precision measurements.

## Prof. Robin Keir

## Total Inorganic Carbon

Samples were taken from CTD rosette bottles and returned to Kiel for analysis. Carbon dioxide was liberated by adding orthphosphoric acid, trapped at liquid nitrogen temperatures and purified by distillation and trapping at controlled temperature. The quantity of  $CO_2$  liberated was determined by a manometer attached to the sample processing line.

The data were supplied in units of micromoles/kg. These were converted to micromoles/litre through application of the following transform:

Database value = Original value/TOKGPR01

 $pCO_2$ 

Surface sea water (from Poseidon's moon pool) was continuously pumped through the gas equilibrator where the dissolved gases exchanged with a closed loop of air. The gas loop was sampled periodically (approximately every 10 minutes) and the  $CO_2$  separated from the methane by the GC column. The  $CO_2$  was then reduced by hydrogen over a nickel catalyst and analysed as methane using FID detection. Calibration samples were analysed approximately every hour as part of an automated sampling cycle.

Air samples were collected periodically (approximately hourly) from an inlet mounted on the bow of the ship and analysed in the same way as the equilibrated gases.

The surface  $pCO_2$  data were reduced by the data originator to average values for each one degree square traversed by the ship. The data in this reduced form has been loaded into BOTDATA. The full resolution data were also provided and these may be found in the underway data file for Poseidon PS211.

## Comments on Data Quality

## Meteor 27\_1

The manometric  $TCO_2$  measurements are systematically lower than results obtained coulometrically on the same samples by non-OMEX participants on the cruise and literature values from the same area, again determined

coulometrically. The difference is approximately 1-2 per cent with the higher differences observed in samples collected from below 1000m depth. This has been attributed to uncertainty in the calibration volume and the data originator cites the coulometric values as more reliable.

There were a number of low value spikes reported in the data set which have been attributed to loss of gas volume during transfer operations on the line. These were removed from the data set prior to submission to BODC.

# **Dissolved and Colloidal Trace Metals**

## **Parameter Code Definitions**

- ALXXLGD2 Dissolved aluminium Lumogallion (0.4/0.45 µm pore filtered) Nanomoles per litre
- ALXXLGTX Dissolved + reactive particulate aluminium Lumogallion (unfiltered) Nanomoles per litre
- CDXXFXC1 Colloidal cadmium Difference between freon extract/AA analyses on 0.4  $\mu$ m pore and 10<sup>4</sup> Dalton filtered water Nanomoles per litre
- CDXXFXD2 Dissolved cadmium Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomoles per litre
- COXXFXD2 Dissolved cobalt Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomoles per litre
- CUXXCVDX Dissolved copper Cathodic stripping voltammetry (UV digested) Nanomoles per litre
- CUXXFXC1 Colloidal copper Difference between freon extract/AA analyses on 0.4  $\mu$ m pore and 10<sup>4</sup> Dalton filtered water Nanomoles per litre
- CUXXFXD2 Dissolved copper Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomoles per litre
- FEXXFXC1 Colloidal total iron Difference between freon extract/AA analyses on 0.4  $\mu$ m pore and 10<sup>4</sup> Dalton filtered water Nanomoles per litre

- FEXXFXD2 Dissolved total iron Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomoles per litre
- MNXXFXD2 Dissolved total manganese Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomoles per litre
- NIXXCVDX Dissolved nickel Cathodic stripping voltammetry (UV digested) Nanomoles per litre
- NIXXFXC1 Colloidal nickel Difference between freon extract/AA analyses on 0.4  $\mu$ m pore and 10<sup>4</sup> Dalton filtered water Nanomoles per litre
- NIXXFXD2 Dissolved nickel Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomoles per litre
- PBXXFXD2 Dissolved lead Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomoles per litre
- ZNXXFXD2 Dissolved zinc Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomoles per litre

## **Originator Code Definitions**

## Belgica cruise BG9309

8 49	Dr. Peter Statham Dr. Jean-Marie Martin	Southampton Oceanography Centre, UK Institut de Biogeochimie, France
Belg	gica cruise BG9322	
8	Dr. Peter Statham	Southampton Oceanography Centre, UK

## **Charles Darwin cruises CD84**

8	Dr. Peter Statham	Southampton Oceanography Centre, UK
12	Dr. David Hydes	Southampton Oceanography Centre, UK.
14	Dr. Lei Chou	ULB, Brussels, Belgium
49	Dr. Jean-Marie Martin	Institut de Biogeochimie, France

## **Charles Darwin cruise CD94**

12	Dr. David Hydes	Southampton Oceanography Centre, UK.
49	Dr. Jean-Marie Martin	Institut de Biogeochimie, France

#### Discovery cruise DI216

8	Dr. Peter Statham	Southampton Oceanography Centre, UK
14	Dr. Lei Chou	ULB, Brussels, Belgium
54	Dr. Eric Achterberg	University of Plymouth

## **Originator Protocols**

## Dr. Jean-Marie Martin

Samples were collected using either manually filled 10 litre acid-washed polypropylene bottles from an inflatable boat in the rias or Teflon coated GoFlo bottles deployed on a CTD rosette. On Charles Darwin cruise CD94, lever action Niskin bottles with external springs were used instead of GoFlo bottles.

The samples were filtered through acid-cleaned 0.4 micron Nuclepore filters under nitrogen pressure under laminar-flow clean benches using clean working practices. For colloidal metal determinations, an aliquot of filtrate was further filtered through a cross-flow ultra-filtration (CFF) system with a polysulphate membrane (10<sup>4</sup> Daltons) to separate colloids.

Hydrophobic metal-organic complexes were extracted on board from both filtered and ultra-filtered samples through pre-conditioned  $C_{18}$  Sep-Pak cartridge (Millipore) via a peristaltic pump and then eluted by a 50% methanol solution using a method modified from Mills et al. (1987).

The metal contents were measured by GFAAS (Perkin Elmer 3030) after extraction in a Class 100 clean room using a method modified from Danielsson et al. (1982). The extraction was carried out at pH 4-5 for CD, Fe, Cu and Ni with 1% of APDC/DDDC as complexant. For Mn the extraction (adapted from Statham, 1985) was run at a pH range of 9-9.5 using 2% APDC/DDDC as complexant. Detection levels have been determined as:

Cd	0.003 nM
Cu	0.05 nM
Ni	0.06 nM
Fe	0.09 nM
Mn	0.06 nM

Extraction efficiency was checked with standard seawater reference material (NASS-4, Canada). 88-120% recoveries were obtained (140% for Fe). The reagent blanks were close to the detection limits and represent less than 5% of the lowest sample concentration.

Colloidal concentrations were determined as the difference between filtered and ultra-filtered dissolved metal determinations.

## Dr. Peter Statham

Trace metal clean procedures developed for open ocean work were used throughout the sample collection and processing. Seawater samples were filtered directly from the CTD rosette bottles (Teflon lined GoFlo on cruises BG9309, BG9322A and CD84: lever-action Niskin bottles on cruises CD94 and DI216) under about 1 bar nitrogen pressure through acid-cleaned 0.4 micron Nuclepore filters mounted in PTFE holders.

Samples were acidified by addition of 1 ml sub-boiling distilled  $HNO_3$  per litre of seawater (except samples for Al analysis) and stored in acid-cleaned (Morley et al. 1988) low density polythene bottles.

This was undertaken using the specialised clean facilities in the Department of Oceanography, University of Southampton and (from 1994) Southampton Oceanography Centre. Dissolved metals were extracted and preconcentrated following the dithiocarbamate complexation-freon extraction method of Danielsson et al. (1982), as modified by Statham (1985) and Tappin (1988), and were determined by graphite furnace atomic absorption spectrophotometry (GFAAS). Within batch analytical precision of the method is generally less than 10% (coefficient of variation) for each metal. More details of the method are given in Tappin et al. (1992).

Quality control (i.e. accuracy and between batch analytical precision) of the data was assessed by regularly analysing aliquots of the CASS-1 coastal seawater reference sample for dissolved trace metals and a bulk filtered acidified sea water sample which was used for batch-to-batch quality control. Results of these analyses were satisfactory, with very few exceptions, and ensure that the data are of high quality.

The method used for aluminium analysis by Peter Statham's student (Ruth Parker) on Discovery 216 is exactly as described below for David Hydes and Lei Chou.

## Dr. David Hydes and Dr. Lei Chou

Water samples were collected by drawing samples off an all-plastic continuous pumped seawater supply fed from a towed fish or from Teflon coated GoFlo (cruise CD84) or from lever-action Niskin (CD94 and DI216) bottles deployed on a CTD rosette.

An aliquot of water was either vacuum filtered through a 0.4 micron Nuclepore membrane or analysed unfiltered for aluminium using the method of Hydes and Liss (1976) with the modification that the sample size was reduced to 20 ml and the samples were left in the dark at laboratory temperature for 24 hours to allow the fluorescence signal to develop. Fluorescence at 500 nm was measured using a Perkin Elmer LS fluorometer using an excitation wavelength of 485 nm.

The complete analytical procedure was undertaken at sea.

## Dr. Eric Achterberg

Underway sampling was performed for sample collection and took place using a peristaltic pump (Watson Marlow; fitted with Marprene tubing (i.d. 2 mm)) and a polyethylene hose (length: 15 m; i.d. 13 mm). The hose was hung overboard and attached to an NERC fish (a torpedo-like object, weight ca. 80 kg) which was towed at approximately 3 m distance from the hull from a cable attached to a winch. The design and weight ensured that the fish stayed at a constant depth (3-4 m), even at speeds over 10 knots. The inlet of the hose was pointed forward, which not only reduced the risk of contamination caused from the fish or the metal cable from which this device was towed, but also assisted pumping by forcing water in. Contamination risk was reduced further by wrapping PVC tape around the bottom 2.5 metres of the metal cable. The underway pumping system had a seawater flow of approximately 2 I min<sup>-1</sup>. The hose for the sample collection is rapidly equilibrated with the water as pumping was continuous during sample collection. The outlet of the hose was positioned in a sink in the ship's laboratory and seawater was sub-sampled continuously from the outlet for trace metal analysis.

Trace metal analyses were performed using an automated voltammetric system (Achterberg and van den Berg, 1994). The metal monitor consisted of a Personal Computer (Opus 386), a voltammetric analyzer (mAutolab; Eco Chemie), an HMDE (hanging mercury drop electrode; 663 VA Stand, Metrohm) and a syringe pump (Cavro) for metal standard addition. A peristaltic pump (Eco Chemie) was used to sub-sample from the outlet of the hose and transport the sample (pumping speed: ~3 ml min<sup>-1</sup>) into a sample loop (11.3 ml) and the sample loop was emptied into the voltammetric cell with the use of nitrogen gas. After analysis, the sample was pumped out of the voltammetric cell by a second peristaltic pump (Eco Chemie) and the cell was rinsed twice with de-ionised water (Milli-Q, Millipore) using a third peristaltic pump (Eco Chemie). A fourth peristaltic pump (Minipuls 3, Gilson) was used for the addition of reagent to the seawater sample. Trace metal concentrations were thus monitored at a rate of one complete measurement approximately every 15 minutes. Voltammetric scans were evaluated using 'intelligent' software; sub-quality scans were selected on basis of a standard deviation above a pre-set value of 5% and deleted, and additional scans were carried out when necessary (Achterberg and van den Berg, 1994). The software calculated the concentration which was subsequently stored on the computer's hard disk with the time of the determination.

A mixed reagent consisting of a pH buffer (Tris, pH 8.35, BDH), dimethyl glyoxime (DMG) and 8-hydroxyquinoline (BDH) was used for the Ni and Cu determinations, which were performed simultaneously using a multi-elemental method (Pihlar *et al.*, 1981; Nimmo *et al.*, 1989; Colombo and van den Berg, 1997). Copper and Ni contamination due to addition of the mixed reagent was <0.1 nM. Nickel contamination due to addition of the mixed reagent was <0.06 nM. Final concentrations in the voltammetric cell were 0.01 M Tris and either 0.1  $\mu$ M 8-hydroxyquinoline or 0.2 mM DMG.

The voltammetric cell was de-aerated for 3 min by purging with watersaturated nitrogen. Two mercury drops were discarded and after the extrusion of the third mercury drop the adsorption period was initiated. Adsorption of Cu-(8-hydroxyquinoline) and Ni-DMG complexes on the HMDE was carried out for a period of 60 seconds, whilst stirring the solution with the potential set at -0.2 V. The stirrer was then stopped and a quiescence period of 10 seconds was allowed at -0.2 V, followed by a potential scan using a square-wave modulation at a frequency of 200 Hz, a modulation amplitude of 25 mV and a step height of 2.4 mV. The scan direction was negative and the reduction peaks corresponding with Cu and Ni appeared at -0.40 and -1.0 V, respectively.

## **Comments on Data Quality**

## Charles Darwin cruise CD84

In the second OMEX annual report, IBM reported that an intercalibration exercise between IBM and SOC on this cruise showed the IBM Cd concentrations to be about 20% higher, Cu determinations from both laboratories to be virtually identical and IBM Ni determinations to be lower.

A small number of very high values in both the IBM and SOC data sets that may only be explained in terms of contamination have been flagged suspect ('M') by BODC. Other values flagged 'M' which otherwise look reasonable are from bottles for which there is strong evidence of contamination of deep water samples by shallow water through leakage.

A number of data values in the ULB aluminium data set have been flagged either 'L' or 'M' in the database. The data values flagged 'L' were reported as contaminated by the originator. The values flagged 'M' are from bottles where there is strong evidence of contamination of deep water samples by shallow water through leakage.

## Charles Darwin cruise CD94

IBM data values identified as contaminated by the data originator (through notes on the spreadsheet supplied) have been flagged 'L' in the database.
# **Discovery cruise DI216**

A number of data values in the ULB aluminium data set have been flagged either 'L' or 'M' in the database. The data values flagged 'L' were reported as contaminated by the originator. The values flagged 'M' are from bottles where there is strong evidence of contamination of deep water samples by shallow water through leakage.

# **Particulate Trace Metals**

# **Parameter Code Definitions**

- ALCNAAP2 Particulate aluminium content Atomic absorption (0.45/0.4 µm pore filtered) Per Cent
- ALCNAAPC Particulate aluminium content Atomic absorption (centrifuged) Per Cent
- CACNICP2 Particulate calcium content ICP after acid digestion (0.45/0.4 µm pore filtered) Per Cent
- CACNICPC Particulate calcium content ICP after acid digestion (centrifuged) Per Cent
- CDCNAAP2 Particulate cadmium content Atomic absorption (0.45/0.4 µm pore filtered) Parts per million
- CDCNAAPC Particulate cadmium content Atomic absorption (centrifuged) Parts per million
- COCNAAP2 Particulate cobalt content Atomic absorption (0.45/0.4 µm pore filtered) Parts per million
- COCNAAPC Particulate cobalt content Atomic absorption (centrifuged) Parts per million
- CRCNAAP2 Particulate chromium content Atomic absorption (0.45/0.4 µm pore filtered) Parts per million
- CRCNAAPC Particulate chromium content Atomic absorption (centrifuged) Parts per million

- CUCNAAP2 Particulate copper content Atomic absorption (0.45/0.4 µm pore filtered) Parts per million
- CUCNAAPC Particulate copper content Atomic absorption (centrifuged) Parts per million
- FECNAAP2 Particulate total iron content Atomic absorption (0.45/0.4 µm pore filtered) Per Cent
- FECNAAPC Particulate total iron content Atomic absorption (centrifuged) Per Cent
- KXCNICP2 Particulate potassium content ICP after acid digestion (0.45/0.4 µm pore filtered) Per Cent
- KXCNICPC Particulate potassium content ICP after acid digestion (centrifuged) Per Cent
- LICNAAP2 Particulate lithium content Atomic absorption (0.45/0.4 µm pore filtered) Parts per million
- MGCNICP2 Particulate magnesium content ICP after acid digestion (0.45/0.4 µm pore filtered) Per Cent
- MGCNICPC Particulate magnesium content ICP after acid digestion (centrifuged) Per Cent
- MNCNAAP2 Particulate total manganese content Atomic absorption (0.45/0.4 µm pore filtered) Per Cent
- MNCNAAPC Particulate total manganese content Atomic absorption (centrifuged) Per Cent
- NACNICP2 Particulate sodium content ICP after acid digestion (0.45/0.4 µm pore filtered) Per Cent

- NACNICPC Particulate sodium content ICP after acid digestion (centrifuged) Per Cent
- NICNAAP2 Particulate nickel content Atomic absorption (0.45/0.4 µm pore filtered) Parts per million
- NICNAAPC Particulate nickel content Atomic absorption (centrifuged) Parts per million
- PBCNAAP2 Particulate lead content Atomic absorption (0.45/0.4 µm pore filtered) Parts per million
- PBCNAAPC Particulate lead content Atomic absorption (centrifuged) Parts per million
- SICNICP2 Particulate silicon content ICP after acid digestion (0.45/0.4 µm pore filtered) Per Cent
- SICNICPC Particulate silicon content ICP after acid digestion (centrifuged) Per Cent
- ZNCNAAP2 Particulate zinc content Atomic absorption (0.45/0.4 µm pore filtered) Parts per million
- ZNCNAAPC Particulate zinc content Atomic absorption (centrifuged) Parts per million

# **Originator Code Definitions**

Belgica cruises BG9309, BG9322, BG9412 and BG9506, Charles Darwin cruises CD84 and CD94 and Discovery cruise DI216.

14 Dr. Lei Chou ULB, Brussels, Belgium

# **Originator Protocols**

# Dr. Lei Chou

Samples were obtained using one of two protocols. The protocol used may be identified by the gear code in the EVENT entry for the data (SAP or GPCENT).

#### SAP collection

Challenger Oceanics in-situ stand-alone pumps (SAPs) were used to sample particulate material. The instruments were deployed on kevlar rope from an auxiliary winch and were switched on and off by a programmable timer to ensure that the pump only sampled when in position at the desired depth. Membrane filters with a 0.4 micron pore size were used to collect the particulate material.

On recovery the filters were rinsed and dried in clean conditions. Back at the home laboratory, the suspended particulate material was ultrasonically detached from the filter for analysis.

## GPCENT collection.

Suspended particulate matter was collected by continuous flow centrifugation using an Alpha-Laval oil purifier (model MAB 104) specially coated for oceanographic use. Water supply was adjusted to approximately 1 cubic metre per hour. Samples were collected both when the ship was on station and steaming between stations for about 6-10 hours.

Samples were taken from the centrifuge body using a stainless steel spatula, stored in acid-washed PET vials and immediately deep frozen. After weighing (wet weight) the sample was subdivided for C/N, trace metal and isotope analysis.

The samples were analysed for Al, Cu, Fe, Mn, Cr, Ni, Co, Zn, Cd and Pb by direct injection of solid samples as slurries using electrothermal atomic absorption spectroscopy in a Varian Spectraa-300 spectrometer with Zeeman correction.

The analytical conditions were as follows:

AI	Wavelength	394.4nm
	Slit width	0.5nm
	Atomisation support	Platform
	Drying	110 °C to 300 °C in 60 seconds
	Ashing	1000 °C, 10 second ramp, 20 second hold
	Atomisation	2600 °C, maximum power, 4 second hold,
		no gas stop
	Modifier	Pd, Si, Ca, K, Mg, Fe, Na, P

Cd	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	228.8nm 0,5nm Platform 110 °C to 280°C in 70 seconds 600 °C, 5 second ramp, 20 second hold 2300 °C, maximum power, 2 second hold, gas stop Mg/PO <sub>4</sub>
Co	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	240.7nm 0.2nm Tube 50 °C to 160 °C in 70 seconds 300 °C, 5 second ramp, 5 second hold, cool to 100 °C 2600 °C, maximum power, 2 second hold, gas stop None
Cr	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	357.9nm 0.2nm Tube 50 °C to 165 °C in 60 seconds 1050 °C, 5 second ramp, 20 second hold, cool to 100 °C 2650 °C, maximum power, 3 second hold, gas stop None
Cu	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	324.7nm 0.5nm Tube 50 °C to 150 °C in 70 seconds 950 °C, 5 second ramp, 30 second hold 2500 °C, maximum power, 2 second hold, gas stop Pd/Mg
Fe	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	302.1nm 0.2nm Tube 50° to 130 °C in 50 seconds 200 °C, 5 second ramp, 20 second hold 2400 °C, maximum power, 2 second hold, no gas stop None

Mn	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	403.1nm 0.2nm Platform 110 °C to 400 °C in 80 seconds 1500 °C, 5 second ramp, 20 second hold, cool to 100 °C 2700 °C, maximum power, 3 second hold, gas stop Pt
Ni	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	232.0nm 0.2nm Tube 50 °C to 150 °C in 70 seconds 1100 °C, 5 second ramp, 20 second hold, cool to 100 °C 2600 °C, maximum power, 3 second hold, gas stop Pd/Mg
Pb	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	217.0nm 1.0nm Platform 110 °C to 370 °C in 70 seconds 800 °C, 5 second ramp, 20 second hold 2600 °C, maximum power, 3 second hold, gas stop Pd/Mg
Zn	Wavelength Slit width Atomisation support Drying Ashing Atomisation Modifier	213.9nm 1.0nm Platform 110 °C to 300 °C in 60 seconds 950 °C, 5 second ramp, 20 second hold 2600 °C, maximum power, 2 second hold, gas stop Mg

Peak area measurement mode was used for all the above elements.

Major elements were determined by Inductively Coupled Plasma emission spectroscopy after complete digestion of the samples by an HNO<sub>3</sub>/HCI/HF mixture in a Teflon bomb in a microwave oven.

If there was insufficient material for the direct injection technique, trace elements were also determined on the digested samples either by ICP, if

present in sufficient concentration, or by AA. The parameter codes have been set up to indicate the predominant method for the element.

# **Comments on Data Quality**

It is reported in the OMEX I final report that ULB participated in the QUASIMEME intercalibration exercise and obtained the following results. The certified values are given in brackets.

1	North Sea	Baltic
Cd (ppb) Cu (ppb) Pb (ppb)	3.46 (3.5) 1.487 (1.425) 26.02 (24) 44.6 (45) 172 (170)	6.1 (6.4) 0.120 (0.123) 20.8 (19.1) 45.7 (44.9) 112 (123)

The good agreement with the certified values gives confidence in the ULB particulate trace metal data.

#### **Charles Darwin cruise CD94**

A number of the filters were reported by the data originator to have anomalously high concentrations for some metals that have been attributed to sample contamination. These have been flagged as 'L' in the database.

## **Discovery cruise DI216**

The CTD cable used on this cruise was in very poor condition and a clearly visible plume of rust could be seen in the water each time the CTD was deployed. Although the SAPs were deployed on kevlar rope, the SAP sampling had to be preceded by a CTD cast to determine the water column structure for assigning the sampling depths.

There is clear evidence in the iron data that most of the SAP samples were polluted by the plume generated by this cast. Consequently, the metal data have been heavily flagged 'L' to reflect the comments of the data originator.

In view of this obvious contamination problem any of the SAP data from this cruise, whatever its associated flag value, should be used with caution.

# **Pigments**

# **Parameter Code Definitions**

- ACARHPP1 Alpha-carotene HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- ALLOHPP1 Alloxanthin HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- BCARHPP1 Beta-carotene HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- BUTAHPP1 Butanoyloxyfucoxanthin HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- C1C2HPP1 Chlorophyll-c1c2 HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- CAROSSP1 Spectrophotometric carotenoid pigments (SCOR) Spectrophotometric assay of acetone extraction (GF/F filtered) milligrams/cubic metre
- CHLBHPP1 Chlorophyll-b HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- CHLBSSP1 Spectrophotometric chlorophyll-b (SCOR) Spectrophotometric assay of acetone extraction (GF/F filtered) Nanograms per litre
- CHLCSSP1 Spectrophotometric chlorophyll-c (SCOR) Spectrophotometric assay of acetone extraction (GF/F filtered) Nanograms per litre
- CLC3HPP1 Chlorophyll-c3 HPLC assay of acetone extract (GF/F filtered) Nanograms per litre

- CLPHHPP1 Chlorophyll-a plus phaeophorbides HPLC assay of acetone extract (GF/F filtered) milligrams/cubic metre
- CPHLFLP1 Fluorometric chlorophyll-a Fluorometric assay of acetone extract (GF/F filtered) Milligrams/cubic metre
- CPHLFLP4 Fluorometric chlorophyll-a Fluorometric assay of acetone extraction (sum of size fractions >0.2 microns) Milligrams/cubic metre
- CPHLFMP1 Fluorometric chlorophyll-a Fluorometric assay of methanol extract (GF/F filtered) Milligrams/cubic metre
- CPHLHPP1 HPLC chlorophyll-a HPLC assay of acetone extract (GF/F filtered) Milligrams/cubic metre
- CPHLPR01 CTD chlorophyll Calibrated in-situ fluorometer Milligrams/cubic metre
- CPHLPRTX Bench fluorometer chlorophyll Bench fluorometer measurement on unfiltered water sample Milligrams/cubic metre
- CPHLSPP1 Spectrophotometric chlorophyll-a (Lorenzen) Spectrophotometric assay of acetone extraction (GF/F filtered) Milligrams/cubic metre
- CPHLSSP1 Spectrophotometric chlorophyll-a (SCOR) Spectrophotometric assay of acetone extraction (GF/F filtered) Milligrams/cubic metre
- CPHLSSPC Spectrophotometric chlorophyll-a (SCOR) Spectrophotometric assay of acetone extraction (centrifuged) Milligrams/cubic metre
- DIADHPP1 Diadinoxanthin HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- DVCAHPP1 Diavinyl chlorophyll-a HPLC assay of acetone extract (GF/F filtered) Nanograms per litre

- FUCXHPP1 Fucoxanthin HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- HEXOHPP1 Hexanoyloxyfucoxanthin HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- LUTNHPP1 Lutein HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- PBA1HPP1 Phaeophorbide-a1 HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- PBA2HPP1 Phaeophorbide-a2 HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- PBA3HPP1 Phaeophorbide-a3 HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- PERIHPP1 Peridinin HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- PHAEFLP1 Fluorometric phaeopigments Fluorometric assay of acetone extract (GF/F filtered) Milligrams/cubic metre
- PHAEFLP4 Fluorometric phaeopigments Fluorometric assay of acetone extract (sum of size fractions >0.2 microns) Milligrams/cubic metre
- PHAEFMP1 Fluorometric phaeopigments Fluorometric assay of methanol extract (GF/F filtered) Milligrams/cubic metre
- PHAESPP1 Spectrophotometric phaeopigments (Lorenzen) Spectrophotometric assay of acetone extract (GF/F filtered) Milligrams/cubic metre
- PTA1HPP1 Phaeophytin-a1 HPLC assay of acetone extract (GF/F filtered) Nanograms per litre

- PTA2HPP1 Phaeophytin-a2 HPLC assay of acetone extract (GF/F filtered) Nanograms per litre
- SCHLFLPA Size-fractionated fluorometric chlorophyll-a Fluorometric assay of acetone extract (>5 micron size fraction) Milligrams/cubic metre
- SCHLFLPB Size-fractionated fluorometric chlorophyll-a Fluorometric assay of acetone extract (>2 micron size fraction) Milligrams/cubic metre
- SCHLFLPC Size-fractionated fluorometric chlorophyll-a Fluorometric assay of acetone extract (2-5 micron size fraction) Milligrams/cubic metre
- SCHLFLPF Size-fractionated fluorometric chlorophyll-a Fluorometric assay of acetone extract (0.2-2 micron size fraction) Milligrams/cubic metre
- SPHAFLPA Size-fractionated phaeopigments Fluorometric assay of acetone extract (>5 micron size fraction) Milligrams/cubic metre
- SPHAFLPB Size-fractionated phaeopigments Fluorometric assay of acetone extract (>2 micron size fraction) Milligrams/cubic metre
- SPHAFLPC Size-fractionated phaeopigments Fluorometric assay of acetone extract (0.2-2 micron size fraction) Milligrams/cubic metre
- SPHAFLPF Size-fractionated phaeopigments Fluorometric assay of acetone extract (>2 micron size fraction) Milligrams/cubic metre
- TCPEFLP1 Total chloroplastic pigment Fluorometric assay of acetone extract (GF/F filtered) Milligrams/cubic metre
- ZEOXHPP1 Zeoxantin HPLC assay of acetone extract (GF/F filtered) Nanograms per litre

# **Originator Code Definitions**

# Cruises Belgica BG9309 and BG9322

5	Dr. Ray Barlow	Plymouth Marine Laboratory, UK
30	Dr. Patrick Dauby	University of Liege, Belgium

# Cruise Belgica BG9412 and BG94ZB

14	Dr Lei Chou	ULB, Brussels, Belgium
30	Dr. Patrick Dauby	University of Liege, Belgium
74	Ir. Andre Pollentier	BMM, Ostend, Belgium

# Cruise Belgica BG9506

30	Dr. Patrick Dauby	University of Liege, Belgium
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# Cruises Belgica BG9521 and BG9522

14	Dr. Lei Chou	ULB, Brussels, Belgium
30	Dr. Patrick Dauby	University of Liege, Belgium

# **Cruises Pelagia PLG93**

76	Dr. Hendrik van Aken	NIOZ, Texel, the Netherlands
95	Dr. Peter de Wilde	NIOZ, Texel, the Netherlands
96	Dr. Laurenz Thomsen	GEOMAR, Kiel, Germany

#### **Cruise Charles Darwin CD86**

76	Dr. Hendrik van Aken	NIOZ, Texel, the Netherlands
95	Dr. Peter de Wilde	NIOZ, Texel, the Netherlands

# Cruise Pelagia PLG95A

95	Dr. Peter de Wilde	NIOZ, Texel, the Netherlands
96	Dr. Laurenz Thomsen	GEOMAR, Kiel, Germany

# Cruise Pelagia PLG95B

76 Dr. Hendrik van Aken	NIOZ, Texel, the Netherlands
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# **Cruise Charles Darwin CD83**

39	Mr. Bob Head	Plymouth Marine Laboratory, UK
94	Dr. Robin Pingree	Plymouth Marine Laboratory, UK

# **Cruise Charles Darwin CD97**

39	Mr. Bob Head	Plymouth Marine Laboratory, UK
Cruis	es Charles Darwin CD84 a	and Valdivia VLD154
5	Dr. Ray Barlow	Plymouth Marine Laboratory, UK
Cruis	es Discovery DI216 and C	harles Darwin CD94
5 16	Dr. Ray Barlow	Plymouth Marine Laboratory, UK British Oceanographic Data Centre, UK
Cruis	e Valdivia VLD137	
5 3 9	Dr. Ray Barlow Dr. Ian Joint Mr. Thomas Raabe	Plymouth Marine Laboratory, UK Plymouth Marine Laboratory, UK Hamburg University, Germany
Cruis	e Charles Darwin CD85	
3	Dr. Ian Joint	Plymouth Marine Laboratory, UK

<ul> <li>5 Dr. Ray Barlow</li> <li>7 Dr. Avan Antia</li> <li>Plymouth Marine Laboratory, Kiel University, Germany</li> </ul>	UK
7 Dr Avan Antia Kiel University Germany	UK
16 British Oceanographic Data C	Centre, UK

# Cruise Discovery DI217

5	Dr. Ray Barlow	Plymouth Marine Laboratory, UK
3	Dr. Ian Joint	Plymouth Marine Laboratory, UK
9	Mr. Thomas Raabe	Hamburg University, Germany
37	Dr. Mike Fasham	Southampton Oceanography Centre

# Cruise Poseidon PS211

# Cruises Jan Mayen JM1-JM8 and JM11

61	Dr. Paul Wassman	University of Tromsø, Norway
97	Prof. Kurt Tande	University of Tromsø, Norway

# Cruises Jan Mayen JM9 and JM10

61	Dr. Paul Wassman	University of Tromsø, Norway
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# **Originator Protocols**

# Dr. Ray Barlow and Dr. Stuart Gibb

Water samples were either collected from water bottles deployed on a CTD rosette, bottles deployed on a hydrographic wire or taken from a continuous surface seawater supply.

1-2 litres of water were filtered through a 25mm GF/F filter, flash frozen and stored in liquid nitrogen until analysed either on board or back in the laboratory.

Pigment concentrations were determined by reverse phase HPLC following the protocols described in Barlow et al. (1993a). Frozen filters were extracted in 90% acetone, sonicated and centrifuged to remove debris. An aliquot (300  $\mu$ l) of clarified extract was mixed with an equal volume of 1M ammonium acetate and 100  $\mu$ l of this mixture was injected into a Shimazdu HPLC system incorporating a 3 micron C18 Pecosphere column (3.3 x 0.45 cm, Perkin Elmer) heated to 30°C.

Pigments were separated by a linear binary gradient changing from 0% B to 100% B over 10 minutes, followed by an isocratic hold at 100% B for 7.5 minutes, at a flow rate of 1 ml per minute. Solvent A consisted of 80:20 (v/v) MeOH : ammonium acetate. Solvent B contained 60:40 (v/v) MeOH : acetone.

Chlorophylls and carotenoids were detected by absorbance at 440nm and phaeopigments by fluorescence detection at 405nm excitation, 670nm emission. Data collection and integration was performed with the Philips PU6000 chromatography software. Diavynyl chlorophyll *a* was determined on some samples using a C8 column as described by Barlow et al. (1996).

Pigments were identified and calibrated by comparison with retention times of pigments isolated from well-documented microalgal species in the Plymouth Culture Collection and with standards obtained from the Water Quality Institute, Denmark. Peak identity was further confirmed on selected samples by on-line diode array visible spectroscopy. Chlorophyll *a* and *b* were calibrated using authentic standards (Sigma Chemical Co.) in acetone and quantified spectrophotometrically using the extinction coefficients of Jeffrey and Humphrey (1975). Diavynyl chlorophyll *a* standard was obtained from R. Bidigare, University of Hawaii. Phaeopigment concentrations were estimated from peak areas and calibrations performed by simultaneous absorbance (667nm) and fluorescence detection of phaeopigments extracted from copepod and mussel faeces as detailed by Barlow et al. (1993b).

All pigments were supplied in units of ng/l. Chlorophyll *a* values were converted to  $mg/m^3$  by dividing by 1000 to unify units for this parameter in the database.

# Dr. Patrick Dauby

Water samples were taken from CTD rosette bottles or the continuously pumped surface seawater supply. To provide pigment data to accompany the centrifuged samples, water samples were taken regularly from the centrifuge input and the results averaged (pigment parameter codes ending in 'PC').

Between 0.5 and 5 litres of water were passed through a GF/F filter which was then frozen. Pigments were subsequently extracted and assayed spectrophotometrically. The data were determined both as chlorophyll *a*, chlorophyll *b*, chlorophyll *c* and carotenoids using the SCOR equation (Strickland and Parsons, 1972) and chlorophyll *a* and phaeopigment using the equations of Lorenzen and Jeffrey (1978).

All data were supplied to BODC in units of  $\mu g/l$  (equivalent to mg/m<sup>3</sup>) but the chlorophyll *b* and chlorophyll *c* were converted to ng/l to conform with the database standard units.

# Dr. Lei Chou

Water samples were filtered through GF/F filters. The filters were placed in plastic vials and flash frozen in liquid nitrogen. Back in the laboratory, the pigments were extracted into 90% acetone and the resulting extracts were assayed fluorometrically.

## Ir. Andre Pollentier

Calibrated fluorometer values from the CTD **downcasts** at depths corresponding to the bottle firings have been determined by BODC and stored in the database. A Chelsea Instruments fluorometer was used and converted to nominal chlorophyll using the SeaBird processing software. These were then calibrated against University of Liege SCOR equation spectrophotometric chlorophyll *a* data by BODC.

## Dr. Hendrik van Aken

Fluorometer values from the CTD **downcasts** at depths corresponding to the bottle firings have been determined by BODC and stored in the database. A Chelsea Instruments fluorometer was used and converted to nominal chlorophyll using the SeaBird processing software.

## Mr. Bob Head

Samples (100 ml) were taken from the non-toxic supply and filtered through 25mm GF/F filters. Pigments were extracted on board ship using 90% acetone and assayed fluorometrically before and after the addition of acid on a Turner 111 fluorometer.

Some size fractionated data were obtained by successive filtration through 5 micron and 2 micron pore filters. These determinations were made in addition to the total chlorophyll measurements described above.

The fluorometer was calibrated before and after the cruise using known concentrations of chlorophyll *a* standard in 90% acetone.

# Dr. Ian Joint

Samples were taken from CTD rosette Niskin bottles, GoFlo bottles deployed on a hydrographic (kevlar) wire or from the continuous pumped seawater supply.

Samples taken for chlorophyll determinations (1-2 litres) were filtered through Whatman GF/F filters. The filters were quickly frozen and returned, continuously frozen, to the laboratory for analysis. Back at the laboratory, the samples were extracted in 90% acetone for approximately 12 hours in the dark at 4 °C. In most cases the resulting extract was assayed spectrophotometrically for chlorophyll *a* and phaeopigments following the procedures outlined in Lorenzen and Jeffrey (1978). On Discovery 217, the extracts were assayed both spectrophotometrically and fluorometrically.

Additional samples were analysed for chlorophyll as part of the sizefractionated production experiments. Aliquots of 100-200 ml were filtered through a cascade of membrane filters (the pore sizes may be determined from the parameter codes). Usually, they were immediately extracted into 90% acetone and assayed fluorometrically on board ship but on occasions the filters were frozen and returned to the laboratory for analysis. The sums of the individual size fraction values have been computed and included in the database.

## Mr. Thomas Raabe

Water samples taken from the CTD rosette bottles had their chlorophyll concentrations estimated by measuring the untreated sample in a Turner Designs bench fluorometer.

For Valdivia 137, BODC have calibrated these estimates against a set of 48 extracted chlorophylls assayed by HPLC. The resulting equation:

chlorophyll 
$$(mg/m^3)$$
 = Fluorometer\_reading \* 0.667 + 0.0657

has been applied to the data set loaded into the database.

For Discovery DI217, a similar calibration against a set of 60 HPLC extracted chlorophyll values gave the relationship:

chlorophyll (mg/m<sup>3</sup>) = Fluorometer\_reading \* 5.0804 - 0.0004

Again, this has been applied to the data loaded into the database. It should be noted, however, that the maximum fluorometer reading corresponding to an HPLC value was 0.147, whereas the maximum raw fluorometer reading in the data set was 0.43. Users should therefore be wary of chlorophyll values from this cruise with the code CPHLPRTX in excess of 1 mg/m<sup>3</sup> as they are the result of an extrapolated calibration.

Calibration has not proved possible for other cruises as no extracted sample data were available. As the fluorometer to chlorophyll relationships for the two cruises are above, it was considered unwise to load the raw readings into the database. The original data have, however, been archived in the BODC system and are available on request.

# Dr. Robin Pingree

Calibrated fluorometer values from the CTD **downcasts** at depths corresponding to the bottle firings have been determined by BODC and stored in the database. A Chelsea Instruments fluorometer was used, calibrated against PML fluorometric chlorophyll *a* data by BODC.

# Dr. Peter de Wilde

Water samples were taken from the CTD rosette or the bottles on the BOLAS lander, filtered through GF/F filters and analysed by means of HPLC. The eluents, gradient and column were similar to those described in Wright et al. (1991) with minor modifications. Pigments were detected by a photodiode array coupled with a fluorometer and quantified according to Tahey et al. (1994).

## British Oceanographic Data Centre

## Cruise Charles Darwin CD85

Calibrated fluorometer values from the CTD **downcasts** at depths corresponding to the bottle firings have been determined by BODC and stored in the database. A Chelsea Instruments Aquatracka fluorometer was used, calibrated against size-fractionated fluorometric chlorophyll data by BODC. Note that the chlorophyll samples were taken from GoFlo bottle casts either immediately before or immediately after the CTD cast used for calibration. No chlorophylls were done on the CTD bottles.

A small HPLC data set was available for this cruise but the subset missed a strong bloom that occurred during this cruise. Attempts to use this calibration to calibrate the fluorometer failed because an exponential extrapolation was required for high chlorophyll casts that gave wildly inaccurate results.

# Cruises Discovery DI216 and Charles Darwin CD94

Calibrated fluorometer values from the CTD **downcasts** at depths corresponding to the bottle firings have been determined by BODC and stored in the database. A Chelsea Instruments Aquatracka fluorometer was used, calibrated against PML HPLC chlorophyll *a* data by BODC.

## Dr. Paul Wassman

Water samples were taken from bottles on the CTD rosette and filtered through Whatman GF/F filters. The filter papers were extracted into methanol and fluorometrically assayed following the protocols of Holm-Hansen et al. (1965) on board ship.

## **Professor Kurt Tande**

Calibrated fluorometer values from the CTD **downcasts** at depths corresponding to the bottle firings have been determined by BODC and stored in the database. Fluorometer data and converted to nominal chlorophyll values were supplied to BODC. These were calibrated on a cruise by cruise basis against Paul Wassman's fluorometrically-assayed extracted chlorophyll data to give the values near the database.

## Dr. Avan Antia

Water samples were collected using large (30 litre) GoFlo bottles deployed from the hydrographic winch and filtered through GF/F filters. The filters were extracted into acetone and assayed fluorometrically.

## Dr. Mike Fasham

Calibrated fluorometer values from the CTD **downcasts** at depths corresponding to the bottle firings have been determined by BODC and stored in the database. A Chelsea Instruments fluorometer was used, calibrated against PML fluorometric chlorophyll *a* data by SOC.

## Dr. Laurenz Thomsen

Water samples were collected using the BIOPROBE benthic water sampling lander (Thomsen et al., 1994). This was deployed on a conductor cable and gently positioned on the sea bed with approximately 20m of slack cable. Penetration into the sediment was determined by a graduated rod monitored by a video camera.

After the material disturbed by the instrument deployment had been seen from transmissometer readings to have dispersed, water samples were collected by pumping into sample bottles on a command signal from the ship. Sampling inlets were positioned at different heights on the instrument enabling water at different heights from the seabed to be collected. Further samples were collected with the lander raised at different heights, generally 5m or 50m, above the sea floor.

Water samples were filtered on GF/F filters and assayed fluorometrically using a Turner Designs fluorometer. Pigment concentrations were computed using the equations of Lorenzen (1967). Further details of the protocol are given in Thomsen and Graf (1995).

# **Comments on Data Quality**

# Cruises Pelagia PLG93, Charles Darwin 86 and Pelagia PLG95B

The values quoted are based on a nominal fluorometer calibration. The data should therefore only be used for relative comparisons. The absolute values are meaningless.

## **Cruise Charles Darwin CD83**

It was reported that during the cruise the supply of GF/F filters for total chlorophyll determinations ran out and 0.45 micron pore filters were used instead. It is not known which samples were affected. All the total chlorophyll data for the cruise have been coded to indicate GF/F filtration. Users should be aware that this parameter code is incorrect for an unknown proportion of the data.

## Jan Mayen Cruises JM1-JM11

The fluorometer signal on many casts shows a strong increasing gradient from the surface to a depth of 10-15m even when temperature and salinity show the water to be well mixed. This has been attributed to quenching which cannot be calibrated out as no light sensors were carried on the CTD package. The effect was seen to be particularly dramatic for cruises in May and June. Calibrated fluorometer values from the upper 15m should therefore be used with caution, especially from spring cruises.

## **Cruise Discovery DI217**

The CTD pressure sensor gave rise to problems during this cruise and for the first three stations only wire out data were available. Whilst every effort has been made to correct the pressure channel and accurately match bottle firing pressures, the possibility for error should be borne in mind by users of the data.

# Suspended Particulate Material Concentration and Characterisation

# **Parameter Code Definitions**

- ABAGIXPZ Aggregate abundance Particle camera image analysis Number per litre
- MDGSCCXX Median grain size Coulter counter analysis on disaggregated samples Micrometres (microns)
- MSAGIXPZ Median aggregate size Particle camera image analysis Micrometres (microns)
- TURBTNTX Nephelometer turbidity (Turner Designs) Through-flow nephelometer Standard turbidity units
- TSEDGVP1 Total suspended particulate material (gravimetry) Gravimetric analysis (GF/F filtered) Milligrams per litre
- TSEDGVP2 Total suspended particulate material (gravimetry) Gravimetric analysis (0.4/0.45 µm pore filtered) Milligrams per litre
- TSEDGVPC Total suspended particulate material (gravimetry) Gravimetric analysis (centrifuged) Milligrams per litre

# **Originator Code Definitions**

# Belgica cruises BG9309 and BG9322

30 Dr. Patrick Dauby University of Liege, Belgium

# Belgica cruise BG9412

30	Dr. Patrick Dauby	University of Liege, Belgium
72	Professor Roland Wollast	ULB, Brussels, Belgium

#### Pelagia cruise PLG93 and Charles Darwin cruise CD86

75	Dr. TCE van Weering	NIOZ, Texel, the Netherlands
96	Dr. Laurenz Thomsen	GEOMAR, Kiel, Germany

#### Pelagia cruise PLG95A

96	Dr. Laurenz Thomsen	GEOMAR, Kiel, Germany
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#### Meteor cruises M27\_1 and M30\_1

9	Mr. Thomas Raabe	Hamburg University, Germany
96	Dr. Laurenz Thomsen	GEOMAR, Kiel, Germany

#### Valdivia cruise VLD137 and Discovery cruise DI217

	9	Mr. Thomas Raabe	Hamburg University, Germany
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#### Charles Darwin cruises CD84, CD94 and Discovery cruise DI216

15	Prof. Nick McCave	Cambridge University, UK
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#### **Discovery cruise DI217**

7 Dr. Avan Antia Kiel University, Germany

## Auriga cruises PLUTUR1-PLUTUR5 and Andromeda cruise PLUTUR6

91 Dr. Aurora Rodrigues Instituto Hidrografico, Portugal

# **Originator Protocols**

#### **Dr. Patrick Dauby**

Suspended particulate matter was collected by continuous flow centrifugation using an Alpha-Laval oil purifier (model MAB 104) specially coated for oceanographic use. Water supply was adjusted to approximately 1 cubic metre per hour. Samples were collected both when the ship was on station and steaming between stations for about 6-10 hours.

Samples were taken from the centrifuge body using a stainless steel spatula, stored in acid-washed PET vials and immediately deep frozen. Wet weight

was determined back in the laboratory and the spm concentration computed from this and the volume of water centrifuged measured by a flow meter on the inlet line.

# Professor Roland Wollast

Determinations of suspended particulate material concentration were made to support radiotracer fast distribution coefficient determinations. Filtration was through 0.45 micron Sartorius membrane filters.

# Dr. T.C.E. van Weering

Between 2 and 6 litres of water collected by a water bottle deployed on a CTD rosette were filtered through a pre-weighed 47mm Nuclepore polycarbonate filter (0.4 micron pore size). Filtrations were performed under pressure applied by a vacuum pump. Salt was removed from the filter residues by rinsing with demineralised water. The filters were oven dried at 50 °C in the laboratory and subsequently re-weighed to determine the SPM concentration.

# Mr. Thomas Raabe

Water samples were assayed on board ship using a through flow Turner Designs nephelometer.

## Prof. Nick McCave

Water samples were taken from Niskin bottles deployed on the CTD rosette or from the continuous non-toxic surface seawater supply. Up to 20 litres of water were filtered through a pre weighed 47mm Cyclopore polycarbonate filter (0.4 micron pore size). Wherever possible, the entire contents of the Niskin bottles were filtered to prevent loss of settling suspended particles through sub-sampling.

Filtrations were performed under pressure applied by a vacuum pump. Salt was removed from the filter residues by rinsing with 8, 15ml aliquots of demineralised water. The samples were air dried and stored in sealed polystyrene petri dishes. Back at the laboratory the samples were further air dried and stored at a constant humidity until they were re-weighed to determine the SPM concentration.

Weighing was done to  $10^{-6}$  grams using a Mettler MT5 balance with a 125  $\mu$ Ci <sup>241</sup>Am alpha foil ionising source mounted in the balance for static reduction. All critical manipulations were carried out within a Class-100 laminar flow hood.

Data were supplied in units of  $\mu$ g/l and converted to mg/l through division by 1000.

# Dr. Avan Antia

Water samples from the CTD rosette were filtered through clean, preweighed GF/F filters, rinsed with MilliQ water and dried at 40 °C for 24 hours. The filters were then re-weighed to determine the SPM concentration.

## Dr. Aurora Rodrigues

Samples were collected using a portable pump and pressure filtered through 142 mm Millipore or Sartorius 0.45 micron pore filters. These were washed, dried and gravimetrically assayed to determine the suspended particulate material concentration.

## Dr. Laurenz Thomsen

## Aggregate concentration and size.

Two particle cameras were fitted to the BIOPROBE lander (Thomsen et al., 1994) mounted such that they were 20 and 40 cm above the sea floor when the instrument was on the bottom. The lander was deployed on a conductor cable and gently positioned on the sea bed with approximately 20m of slack cable. Penetration into the sediment was determined by a graduated rod monitored by a video camera.

After the material disturbed by the instrument deployment had been seen from transmissometer readings to have dispersed, images of the suspended material were collected by each camera.

The instrument was then raised to 5m above the sea floor and further pictures were taken. This process was repeated at 50m above the sea bed.

Back in the laboratory, samples of surface sediment collected using a multicore were resuspended and photographed by the aggregate camera to obtain estimates of median aggregate size of the surface fluff.

The resulting images were analysed on a Macintosh Power PC image analysis system following the method of Thomsen and Ritzrau (1996) to obtain the aggregate abundance and median size.

## Total particulate matter

Water samples were collected using the BIOPROBE benthic water sampling lander (Thomsen et al., 1994). This was deployed on a conductor cable and gently positioned on the sea bed with approximately 20m of slack cable. Penetration into the sediment was determined by a graduated rod monitored by a video camera.

After the material disturbed by the instrument deployment had been seen from transmissometer readings to have dispersed, water samples were

collected by pumping into sample bottles on a command signal from the ship. Sampling inlets were positioned at different heights on the instrument enabling water at different heights from the seabed to be collected. Further samples were collected with the lander raised at different heights, generally 5m or 50m, above the sea floor.

Total suspended matter was determined gravimetrically by filtering through pre-weighed GF/F filters. Median grain size was determined by Coulter counter analyses of disaggregated samples.

# Comments on Data Quality

#### **Belgica cruises**

The Liege SPM determinations by centrifugation are based on wet weighing and an approximately determined volume. In addition, no matter how carefully the sample is extracted from the centrifuge, it is impossible to recover it all. The data should therefore only be used for comparative purposes within the centrifugation data set and should not be used for quantitative calculation of particulate constituent concentrations like POC.

# **Dimethylsulphide and its Precursors**

# **Parameter Code Definitions**

- DMSOGCD4 Dissolved dimethylsulphoxide Gas chromatography on DMS released by enzyme digestion (Millipore pre-filtered) Nanomoles per litre
- DMSPGCD4 Dissolved DMSP Gas chromatography (Millipore pre-filtered) Nanomoles per litre
- DMSPGCP4 Particulate DMSP Gas chromatography (Millipore pre-filtered) Nanomoles per litre
- DMSPGCTX Total DMSP Gas chromatography (unfiltered) Nanomoles per litre
- DMSXGCD4 Dissolved dimethylsulphide Gas chromatography (Millipore pre-filtered) Nanomoles per litre
- DMSXGCDX Dissolved dimethylsulphide Gas chromatography on gases purged from unfiltered water Nanomoles per litre
- DMSXGCDZ Dissolved dimethylsulphide Gas chromatography (unspecified filter type) Nanomoles per litre

# **Originator Code Definitions**

## **Cruises Belgica 9412 and Charles Darwin 83**

85 Dr. Ba Cuong Nguyen CFR, CNRS-CEA, France

## Cruise Poseidon PS211

68	Dr. Angela Hatton	University of East Anglia, UK
85	Dr. Ba Cuong Nguyen	CFR, CNRS-CEA, France

# Cruises Meteor M30\_1 and Valdivia VLD154

58 Dr Guenther Uher Max Planck Institute, Germany

# **Originator Protocols**

## Dr. Ba Cuong Nguyen

Samples were collected from either water bottles deployed on a CTD rosette or the continuously pumped surface seawater supply. Comparative measurements were made which showed that there is no significant difference in the data obtained from these two water sources.

The dimethylsulphide was purged from about 60-180 ml of seawater by bubbling high grade helium at 100 ml per minute through the sample. The gases liberated were cryogenically trapped at -90 °C before being analysed by gas chromatography with flame photometric detection. The precision of the analysis is reported to be about 10% and the detection limit 0.4nM.

Dissolved plus particulate DMSP was determined as DMS after hydrolysis by sodium hydroxide. The detection limit is reported as 2 nM.

Calibration was achieved using a weight-loss calibrated DMS permeation device and a standard DMS solution in ethylene glycol.

## Dr. Angela Hatton

Water samples were collected from the continuous pumped sea water supply, drawn from the ship's moon pool, using 500 ml ground glass bottles sealed with ground glass stoppers to leave minimal head space. The water was supplied to the bottom of the bottle using a silicate tube and the water allowed to overflow to prevent bubble entrapment.

Samples were transferred to the ship's laboratory for immediate DMS analysis by purge and trap gas chromatography. A volumetric aliquot of water was injected into the purging vessel through a Millipore pad pre-filter. Trace gases were extracted from the water by a stream of high grade nitrogen which subsequently passed through a cold trap (-150°C) where the gases were concentrated before being heated and injected into the gas chromatograph.

The gases were analysed using a Chromosil 330 column, isothermal at 40 °C with flame photometric detection (FPD). Full details are given in Turner et al. (1990). All DMS analyses were completed on board ship. The instrument was calibrated at the start and end of each sampling run using a stock DMS standard.

An aliquot of the purged water sample was treated with 10M NaOH (to decompose DMSP to DMS) The dissolved DMSP was quantified as DMS as described above. The filter pad was also treated with 10M NaOH and the DMS released was quantified to give particulate DMSP.

A second aliquot of purged water was treated with a solution containing purified DMSO reductase, ethylenediaminetetraacetic acid (EDTA) and flavin mononucleotide (FMN) to convert DMSO into DMS. Dissolved DMSO was then determined as DMS as described above. Further details of this technique may be found in Hatton et al. (1994).

## Dr. Guenther Uher

Continuous underway measurements of dimethylsulphide were made using an automated gas chromatograph with a flame photometric detector. This allowed samples to be analysed automatically at intervals of between 20 and 60 minutes.

Sea water from the ship's continuous surface pumped supply was drawn through a filter into a purging vessel. Dissolved gases were purged by bubbling purified helium, passing through a potassium carbonate drying tube into a cryogenic trap. This automatically went through a liquid nitrogen cooling cycle followed by a heating cycle. After release, the trapped gases were analysed using a gas chromatograph with flame photometric detection.

# Carbonyl Sulphide. Atmospheric Concentrations, Seawater Concentrations and Production

# **Parameter Code Definitions**

- COSAGCXX Atmospheric carbonyl sulphide Gas chromatography Parts per billion by volume
- COSSGCXX Carbonyl sulphide saturation Computed from atmospheric and seawater concentrations Per cent
- COSXGCXX Dissolved carbonyl sulphide Gas chromatography Picomoles per litre
- CSDEGCD1 Carbonyl sulphide dark production standard error Gas chromatography on GF/F filtered water incubated in darkness Picomoles per cubic metre per second
- CSDPGCD1 Carbonyl sulphide dark production Gas chromatography on GF/F filtered water incubated in darkness Picomoles per cubic metre per second
- CSPEGSD1 Carbonyl sulphide photo-production standard error Gas chromatography on GF/F filtered water incubated in natural sunlight Picomoles per metre per Watt per second
- CSPPGSD1 Carbonyl sulphide photo-production Gas chromatography on GF/F filtered water incubated in natural sunlight Picomoles per metre per Watt per second

# **Originator Code Definitions**

# Meteor cruises 27\_1 and 30\_1

55	Dr. Veit Ulshofer	Max Planck Institute, Germany
65	Dr. Otmar Flock	Max Planck Institute, Germany

# **Originator Protocols**

# Dr. Veit Ulshofer

Seawater was drawn from approximately 7m depth using a non-contaminating pumping system comprising a Teflon pump and PVC tube mounted inside a stainless steel shaft submerged in the ship's 'moon pool'. Atmospheric samples were drawn from the bow of the ship.

The automated analytical system alternately analysed air samples and fixed volume air samples equilibrated with seawater with a cycling time of 1-2 hours. The air samples were cryogenically trapped, separated by chromatography and then analysed by flame photometric detection.

The air value associated with each water sample measurement was obtained by linearly interpolating between the air samples measured before and after the water sample.

Carbonyl sulphide saturation was computed by:

Saturation = (H \* C<sub>seawater</sub>)/C<sub>air</sub>

where H is the Henry's Law constant for carbonyl sulphide in seawater.

Precision and accuracy are estimated at  $\pm 15\%$  for Meteor 27\_1 and  $\pm 10\%$  for Meteor 30\_1.

# Dr. Otmar Flock

Water samples were taken using non-contaminating, gas-tight GoFlo water bottles. The samples were pressure filtered through pre-ashed (400 °C for 2 hours) Whatman GF/F filters and transferred into volume-calibrated glass flasks (approximately 300 ml) and stored in the dark at 4 °C for not longer than 6 hours.

Carbonyl sulphide production was estimated by taking samples from the clean surface pumping system. Samples were GF/F filtered and transferred into glass flasks that were incubated in natural sunlight for approximately 10 hours, together with dark controls, at sea surface temperature. The carbonyl sulphide concentration was monitored at regular intervals during the incubation.

Carbonyl sulphide was analysed by gas stripping the seawater, followed by cryogenic trapping, gas chromatographic separation and flame photometric detection. All results were corrected for sample losses due to hydrolysis.

# Methane

# **Parameter Code Definitions**

- CH4AGCXX Atmospheric methane Gas chromatography Nanomoles per litre
- CH4CGCXX Dissolved methane Gas chromatography Nanomoles per litre

# **Originator Code Definitions**

# Cruises Meteor M27\_1, Belgica BG9506 and Poseidon PS211

73 Prof. Robin Keir

GEOMAR, Kiel, Germany

# **Originator Protocols**

## Prof. Robin Keir

Water samples were collected from the CTD rosette and analysed on board ship for dissolved methane. The gas phase was obtained by two methods. The first involved a partial separation of gas and water phases under vacuum using repeated application of ultrasound. The second utilised equilibration of the water sample with a small volume of added pure nitrogen head space. Dissolved methane was then computed from the measured gas phase mixing ratio and the methane solubility at the laboratory conditions of temperature and salinity.

Measurements of surface dissolved methane were obtained as a by-product of the underway  $pCO_2$  determination. Surface sea water (from an inlet in the bow of Belgica or the 'moon pool' of Poseidon) was continuously pumped through the gas equilibrator where the dissolved gases exchanged with a closed loop of air. The gas loop was sampled periodically (approximately every 10 minutes) and the  $CO_2$  separated from the methane by the GC column. The  $CO_2$  was then reduced by hydrogen over a nickel catalyst and analysed as methane. This resulted in two peaks that could be quantified separately for methane and carbon dioxide. Air samples were collected periodically (approximately hourly) from an inlet mounted on the bow of the ship and analysed in the same way as the equilibrated gases.

The surface methane data were reduced by the data originator to average values for each one degree square traversed by the ship. The data in this reduced form could be loaded into the database for Poseidon PS211 because the cruise track was a simple straight line. However, Belgica passed through a number of squares several times during the cruise making loading of the spatially averaged data impossible. Consequently, the full raw data set (water samples every 10 minutes with an air sample approximately every hour) has been loaded for this cruise.

# **Atmospheric Ammonia and Methylamines**

# **Parameter Code Definitions**

ADMAFIGI	Gaseous atmospheric dimethylamine Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
ADMAFIPT	Atmospheric particulate dimethylamine Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air
AEAXFIGI	Gaseous atmospheric ethylamine Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
AEAXFIPT	Atmospheric particulate ethylamine Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air
AMMAFIGI	Gaseous atmospheric monomethylamine Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
AMMAFIPT	Atmospheric particulate monomethylamine Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air
ANH3FIGI	Gaseous atmospheric ammonia Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
ANH3FIPT	Atmospheric particulate ammonium Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air
ATMAFIGI	Gaseous atmospheric trimethylamine Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
ATMAFIPT	Atmospheric particulate trimethylamine Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air

- DDMAFIGI Gaseous atmospheric dimethylamine standard deviation Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
- DDMAFIPT Atmospheric particulate dimethylamine standard deviation Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air
- DEAXFIGI Gaseous atmospheric ethylamine standard deviation Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
- DEAXFIPT Atmospheric particulate ethylamine standard deviation Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air
- DMMAFIGI Gaseous atmospheric monomethylamine standard deviation Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
- DMMAFIPT Atmospheric particulate monomethylamine standard deviation Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air
- DNH3FIGI Gaseous atmospheric ammonia standard deviation Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
- DNH3FIPT Atmospheric particulate ammonium standard deviation Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air
- DTMAFIGI Gaseous atmospheric trimethylamine standard deviation Flow injection gas diffusion analysis of acid impregnated filter Picomoles per cubic metre of air
- DTMAFIPT Atmospheric particulate trimethylamine standard deviation Flow injection gas diffusion analysis of Teflon filter Picomoles per cubic metre of air

# **Originator Code Definitions**

## Poseidon cruise PS211

# **Originator Protocols**

# Dr. Stuart Gibb

Atmospheric particulate and gaseous samples were collected using a tandem filter system, equipped with cyclone separators in series with Teflon and acid-impregnated filter papers. The system was modified from the equipment presented in Quinn et al. (1987, 1988). The samples were exposed for periods of 1 or 2 days whilst the vessel was underway and then frozen for subsequent laboratory extraction and Flow Injection Gas Diffusion coupled to Ion Chromatography (FIGD-IC) analysis.

The FIGD-IC procedure is described in Gibb et al. (1995). This is a novel technique that allows the simultaneous measurement of methylamines and ammonia at nanomolar levels. Briefly, the ammonia and methylamines were deprotonated to their free, volatile forms through alkali admixing (NaOH, pH>12) and selectively transferred by diffusion across a gas-permeable membrane into a dynamic, acidic acceptor stream in which they were enriched in their cationic forms. Chelation of the alkali earth metals in the samples with EDTA, under thermodynamically optimised conditions, was used to prevent the precipitation of their hydroxides under the elevated pH conditions. The enriched acceptor stream was then transferred directly into an ion chromatograph in which the  $NH_4^+$  and methylamines were separated within 15 minutes in an acidic eluent and quantified by chemically suppressed conductimetric detection.
# **Dissolved Methylamines**

### **Parameter Code Definitions**

DIMAFITX	Dissolved dimethylamine Flow injection gas diffusion (unfiltered) Nanomoles per litre
MOMAFITX	Dissolved monomethylamine Flow injection gas diffusion (unfiltered) Nanomoles per litre

TRMAFITX Dissolved trimethylamine Flow injection gas diffusion (unfiltered) Nanomoles per litre

### **Originator Code Definitions**

#### Poseidon cruise PS211

71 Dr. Stuart Gibb Plymouth Marine Laboratory, UK

### **Originator Protocols**

#### Dr. Stuart Gibb

Water samples were collected from the continuous pumped sea water supply using either 250 ml gas-tight polythene bottles or 100 ml glass syringes. The methylamine concentrations were determined on board using Flow Injection Gas Diffusion coupled to Ion Chromatography (FIGD-IC).

The FIGD-IC procedure is described in Gibb et al. (1995). This is a novel technique that allows the simultaneous measurement of methylamines and ammonia at nanomolar levels. Briefly, the ammonia and methylamines were deprotonated to their free, volatile forms through alkali admixing (NaOH, pH>12) and selectively transferred by diffusion across a gas-permeable membrane into a dynamic, acidic acceptor stream in which they were enriched in their cationic forms. Chelation of the alkali earth metals in the samples with EDTA, under thermodynamically optimised conditions, was used to prevent the precipitation of their hydroxides under the elevated pH conditions. The enriched acceptor stream was then transferred directly into

an ion chromatograph in which the  $NH_4^+$  and methylamines were separated within 15 minutes in an acidic eluent and quantified by chemically suppressed conductimetric detection.

# **Dissolved Oxygen**

### **Parameter Code Definitions**

- DOXYPR01 Beckman oxygen Beckman oxygen probe Micromoles/litre
- DOXYWITX Winkler oxygen Winkler titration Micromoles/litre
- OXYSBB01 Oxygen saturation (Bens.Kr./Beckman) Benson & Krause algorithm from Beckman data Per Cent

## **Originator Code Definitions**

# Belgica cruises BG9309, BG9322, BG9412, BG9506, BG9521 and BG9522

69 74	Dr. Michel Frankignoulle Ir. Andre Pollentier	University of Liege, Belgium MUMM, Ostend, Belgium
Pelag	ia cruises PLG93 and PL	G95B and Charles Darwin cruise CD86
11 76	Dr. Wim Helder Dr. Hendrik van Aken	NIOZ, Texel, the Netherlands NIOZ, Texel, the Netherlands
Meteor cruise M27_1, Valdivia cruise VLD137 and Discovery cruise DI217		
9	Mr. Thomas Raabe	Hamburg University, Germany
Meteo	or cruise M30_1	
9 51	Mr. Thomas Raabe Prof. Wolfgang Balzer	Hamburg University, Germany University of Bremen, Germany
Charles Darwin cruise CD83		
48 94	Mr. Pablo Serret Dr. Robin Pingree	University of Oviedo (Spain) Plymouth Marine Laboratory, UK

#### **Charles Darwin cruise CD84**

8	Dr. Peter Statham	Southampton Oceanography Centre, UK
16		British Oceanographic Data Centre, UK

#### Charles Darwin cruise CD94

9	Mr. Thomas Raabe	Hamburg University, Germany
16		British Oceanographic Data Centre, UK

#### **Cruise Discovery DI216**

8	Dr. Peter Statham	Southampton Oceanography Centre, UK
12	Dr. David Hydes	Southampton Oceanography Centre, UK
16		British Oceanographic Data Centre, UK

# Madorniña cruises MD0994, MD0495, MD0695, MD0795, MD0995, MD1095 and MD1195

66	Dr. Ricardo Prego	IIM, CSIC, Vigo, Spain
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### **Originator Protocols**

#### Dr Michel Frankignoulle

Water samples taken from CTD rosette bottles were analysed using the classical Winkler technique using a Metrohm automatic titration system.

#### Ir Andre Pollentier

The oxygen probe concentrations and saturations in table BOTDATA have been obtained by extracting the data from the CTD **downcasts** corresponding to the bottle firing depths. The CTD system used was a SeaBird SBE 9 or 9 *plus* with a non-pulsed membrane oxygen sensor. On cruises BG9506, BG9521 and BG9522 the CTD was fitted with a YSI pulsed oxygen electrode. Oxygen data were calibrated against the University of Liege water bottle data.

Oxygen saturations were computed using calibrated temperature and salinity data and the algorithm of Benson and Krause (1984).

#### Dr. Wim Helder

Water samples from CTD rosette bottles were analysed using the classical Winkler technique with minor modifications. Calibrated 100 ml oxygen bottles with ground glass stoppers were thoroughly flushed then filled without trapping any air. 1 ml of solution A ( $600g \text{ MnCl}_2.4H_2\text{O}$  per litre) and 2 ml of solution B (250g NaOH, 350g KI per litre) were added immediately and the closed bottles shaken vigorously. The samples were shaken again once the precipitate had settled. The bottles were stored under water with the stoppers held closed by elastic bands.

Prior to analysis, about 25 ml of the supernatant was removed by syringe and then 1 ml of 20N sulphuric acid was added. Titration was carried out with 0.01N sodium thiosulphate in a Brand Digital Burette.

When the solutions in the bottle turned light yellow, 0.5 ml of 1% starch solution was added and titration continued until the solution became colourless. The sodium thiosulphate solution was made from a 0.1N stock solution in ampoules (Merck) and its strength was regularly checked by titration with 0.01N KIO<sub>3</sub>. Blank corrections were applied.

All samples were determined in duplicate at least. Accuracy is reported to be within 1%.

For Pelagia 95 a modified automatic Metrohm titration unit was introduced with spectrophotometric end point detection.

#### Dr. Hendrik van Aken

The oxygen probe concentrations and saturations in table BOTDATA have been obtained by extracting the data from the CTD **downcasts** corresponding to the bottle firing depths. The CTD system used was a SeaBird SBE 911 *plus* with a non-pulsed membrane oxygen sensor. Oxygen data were calibrated against the NIOZ water bottle data.

Oxygen saturations were computed using calibrated temperature and salinity data and the algorithm of Benson and Krause (1984).

#### Mr. Thomas Raabe

Water samples were taken from the CTD bottle rosette. Dissolved oxygen was determined by Winkler titration with a Metrohm titration stand. Data were supplied in units of mg/l and have been converted to  $\mu$ M by multiplying by 31.251172.

#### Prof. Wolfgang Balzer

Water samples taken from the hydrocast rosette bottles were analysed using the classical Winkler technique without any modifications of note.

#### Mr. Pablo Serret

Water samples were taken from water bottles deployed on the CTD rosette and analysed by the Winkler procedure with a potentiometric end-point detector (Metrohm 716 DMS).

#### Dr. Robin Pingree

The oxygen probe concentrations and saturations in table BOTDATA have been obtained by extracting the data from the CTD **downcasts** corresponding to the bottle firing depths. The CTD system used was a Neil Brown Mk3B with a non-pulsed membrane oxygen sensor. Oxygen data were calibrated against University of Oviedo water bottle data.

Oxygen saturations were computed using calibrated temperature and salinity data and the algorithm of Benson and Krause (1984).

#### Dr. Peter Statham and Dr. David Hydes

The protocols used were very similar and so are described together.

Water samples were taken from the CTD rosette into glass-stoppered bottles that were filled from the bottom and allowed to overflow. After addition of the Winkler reagents, the bottles were shaken vigorously for some considerable time. The samples were then titrated against thiosulphate.

The difference between the two methods was in the technique used for endpoint detection. Peter Statham's method was photometric with the end point taken as the transmittance maximum as thiosulphate was added from a 1ml Dosimat unit controlled by a manual switch. David Hydes's method used two bright platinum electrodes which measured the depolarisation caused by iodine and iodide in solution. Thiosulphate was dispensed by an automatic Titrino unit with the results logged by a PC.

#### British Oceanographic Data Centre

#### Charles Darwin CD84

The oxygen probe concentrations and saturations in table BOTDATA have been obtained by extracting the data from the CTD **downcasts** corresponding to the bottle firing depths. The CTD system used was a Neil Brown Mk3B with a non-pulsed membrane oxygen sensor. Oxygen data were calibrated against Peter Statham's water bottle data.

Oxygen saturations were computed using calibrated temperature and salinity data and the algorithm of Benson and Krause (1984).

#### Charles Darwin CD94

The oxygen probe concentrations and saturations in table BOTDATA have been obtained by extracting the data from the CTD **downcasts** corresponding to the bottle firing depths. The CTD system used was one of two Neil Brown Mk3Bs with a non-pulsed membrane oxygen sensor. Oxygen data were calibrated against University of Hamburg's water bottle data. The two CTDs were calibrated independently using different methods. See the CTD data documentation for more details.

Oxygen saturations were computed using calibrated temperature and salinity data and the algorithm of Benson and Krause (1984).

#### Discovery DI216

The oxygen probe concentrations and saturations in table BOTDATA have been obtained by extracting the data from the CTD **downcasts** corresponding to the bottle firing depths. The CTD system used was a Neil Brown Mk3B with a non-pulsed membrane oxygen sensor. Oxygen data were calibrated against David Hydes's water bottle data. This was chosen over the alternative data set because coverage was more complete (Peter Statham's data set is confined to the ten casts used for the intercalibration exercise).

Oxygen saturations were computed using calibrated temperature and salinity data and the algorithm of Benson and Krause (1984).

#### Dr. Ricardo Prego

Water samples were taken from the CTD rosette and analysed using the Winkler technique as described in Aminot and Chaussepied (1983).

#### Comments on Data Quality

#### Cruise Belgica BG9506

The Winkler oxygen data from this cruise are exceptionally low with surface saturations of 90 per cent or less and values in the oxygen minimum below 180  $\mu$ M. Compared to data from the same area at the same time of year (1993 and 1994), the data are systematically low by 10 per cent. The CTD data, prior to calibration against the bottle data, look as expected with surface saturations scattered around 100 per cent and values in the oxygen minimum of 190-200  $\mu$ M.

The conclusion from examination of the data is that there is a systematic error in the bottle data from this cruise. Consequently, all bottle data have been flagged suspect and no bottle calibration has been applied to the CTD data.

#### Cruises Belgica BG9521 and BG9522

The CTD oxygen data from these cruises were detrimentally affected by temperature gradients in the water column and the oxygen sensor appeared slow to equilibrate. Empirical correction on a cast by cast basis was attempted for BG9521 but there were insufficient bottle data to do this for BG9522 and all shallow (above 200m) CTD oxygen data from this cruise have been jettisoned.

The shallow CTD derived values from BG9521 should be used with caution and wherever possible the Winkler data should be used instead. The deep CTD data from both cruises are of better quality. See the CTD data documents from these cruises for further details.

#### Cruise Meteor M30\_1

The Hamburg and Bremen dissolved oxygen data, where common samples have been analysed, are generally within 5-10  $\mu$ M and often in closer agreement. The Bremen values tend to be lower than the Hamburg values near the surface but slightly higher at depth.

The Hamburg data set includes some values from the oxygen minimum on the Goban Spur transect that are some 5-10  $\mu$ M lower than those observed on other cruises where the dissolved oxygen data are considered to be high quality.

#### **Cruise Charles Darwin CD84**

The bottle data from this cruise are good quality, with the exception of one obvious wild point that has been flagged suspect in the database. A significant number of other points have been flagged suspect. These are samples that have been taken from bottles identified through salinity and/or nutrient evidence as contaminated by surface waters through leakage.

#### **Cruise Charles Darwin CD94**

A number of isolated dissolved oxygen values were clearly anomalously high and these have been flagged 'M' in the database. Values from the oxygen minimum on the Goban Spur are slightly higher on some casts than values observed on other cruises where the oxygen data are believed to be good quality.

It is stated in the OMEX I final report that the CTD oxygen calibration from CD94 appears incorrect when the data are compared to DI216 and CD84. As a result, further quality control (more samples flagged 'M' and excluded from the calibration) was carried out on the bottle data set and the CTD was recalibrated. The result has been a systematic reduction in the CD94 deep

oxygen values by approximately 5  $\mu$ M, bringing them significantly closer to the values obtained on the other two cruises

#### **Cruise Discovery DI216**

Two groups measured dissolved oxygen on this cruise using different endpoint detection methods. The primary purpose of this was to intercalibrate the two systems and to improve procedures to ensure that WOCE precision standards (<0.1%) may be obtained regularly.

Duplicate samples were taken by the two groups from the first 10 casts of the cruise and produced results showing good agreement between the two data sets (slope = 0.9947: R<sup>2</sup> = 98.74%). A small negative offset (-0.88) showed that David Hydes's method produced results that were systematically slightly lower.

Precision of each method was tested by taking duplicate samples from two bottles on each of the casts. This showed that 16% of the samples analysed by Peter Statham's method and 46% of the samples analysed by David Hydes's method achieved WOCE precision.

It may therefore be seen that the oxygen water bottle data from this cruise are of very high quality.

# Hydrography

### **Parameter Code Definitions**

- ATTNZR01 Red light attenuance (unspecified beam) 661nm unspecified path length transmissometer per metre
- NEPHAQ01 Nephelometer turbidity (Aquatracka) CI Aquatracka in-situ nephelometer Arbitrary units
- POTMCV01 Potential temperature (UNESCO) Computed using UNESCO function POTEMP Degrees Centigrade
- PSALBSTX Bench salinometer salinity Salinometer Practical Salinity Units
- PSALPR01 Practical salinity (unspecified probe type) Unspecified conductivity probe Practical Salinity Units
- PSALST01 Practical salinity (CTD) CTD conductivity measurement Practical Salinity Units
- SIGTPR01 Sigma-theta (CTD data) Computed by UNESCO SVAN function Kilograms/cubic metre
- TEMPDTNX Sea temperature (thermometer) Hand-held digital thermometer Degrees centigrade
- TEMPPR01 Sea temperature (unspecified) Unspecified temperature probe Degrees centigrade
- TEMPRTNX RT temperature Reversing thermometer Degrees centigrade

- TEMPST01 Sea temperature (CTD/STD) CTD or STD measurement Degrees centigrade
- TOKGPR01 µM to µmoles/kg conversion (CTD) CTD measurement Dimensionless

# **Originator Code Definitions**

Belgica cruises BG9309, BG9322, BG9412, BG9506, BG9521 and BG9522

74	Ir. Andre Pollentier	MUMM, Ostend, Belgium
Pelag	ia cruise PLG93 and Char	les Darwin cruise CD86
76 96	Dr. Hendrik van Aken Dr. Laurenz Thomsen	NIOZ, Texel, the Netherlands GEOMAR, Kiel, Germany
Pelag	ia cruise PLG95A	
96	Dr. Laurenz Thomsen	GEOMAR, Kiel, Germany
Pelag	ia cruise PLG95B	
6 76	Dr. Hendrik van Aken	NIOZ, Texel, the Netherlands NIOZ, Texel, the Netherlands
Posei	don cruise PS200_7	
7 88	Dr. Avan Antia	Kiel University, Germany IfM Kiel, Germany
Meteo	or cruise M27_1	
7 16	Dr. Avan Antia	Kiel University, Germany British Oceanographic Data Centre, UK
Meteo	or cruise M30_1	
7 96	Dr. Avan Antia Dr. Laurenz Thomsen	Kiel University, Germany GEOMAR, Kiel, Germany

### Valdivia cruise VLD137

3 16	Dr. Ian Joint	Plymouth Marine Laboratory, UK British Oceanographic Data Centre, UK
Heind	ke cruise 68	
90	Dr. Pete Bowyer	University College Galway, Ireland
Charl	es Darwin cruise CD85	
103 16		Defence Research Agency, UK British Oceanographic Data Centre
Disco	overy cruise DI217	
37	Dr. Mike Fasham	Southampton Oceanography Centre
Valdiv	via cruise VLD154	
92		IfM Hamburg, Germany
Charl	es Darwin cruise CD83	
94	Dr. Robin Pingree	Plymouth Marine Laboratory, UK
Charl	es Darwin cruises CD84 a	nd CD94
1 16		Research Vessel Services British Oceanographic Data Centre
Disco	overy cruise DI216	
1 12 16	Dr. David Hydes	Research Vessel Services, UK Southampton Oceanography Centre, UK British Oceanographic Data Centre, UK
Jan N	layen cruises JM1-JM11	
97	Prof. Kurt Tande	University of Tromsø, Norway
	rniña cruises MD0994, 95, MD1195	MD0495, MD0695, MD0795, MD0995,
66	Dr. Ricardo Prego	IIM, CSIC, Vigo, Spain

#### Poseidon cruise PS211

16

British Oceanographic Data Centre, UK

### **Originator Protocols**

In most cases where the parameter code ends in '01', the values have been obtained by BODC software which extracts CTD **downcast** data corresponding to the bottle firing depths. This ensures an internally consistent data set across all cruises regardless of whether or not the upcast data were made available. The method is prone to errors if significant changes occur to water column structure during the cast. In all cases, further details about the CTD data may be obtained from the CTD document for the relevant cruise.

The conversion factor TOKGPR01 is 1000/(1000+sigma-theta) and is stored to allow sample data stored in concentration per litre to be converted to concentration per kilogram.

#### Ir. Andre Pollentier

CTD data were taken using a SeaBird SBE 9 or 9 *plus* CTD with, on some cruises, a SeaTech 25cm transmissometer. The instrument is well maintained and frequently recalibrated.

Water samples are collected in cleaned beer bottles and sealed with crown corks. Back in the laboratory, salinity is determined using a Beckman RB7 bench salinometer.

On the Belgica cruise BG9309, a series of samples were collected using a small boat working away from the mother ship. Water temperatures for these samples were measured using a hand-held thermometer and salinities determined using a refractometer.

#### Dr. Hendrik van Aken

CTD data were taken using a SeaBird SBE 911 *plus* CTD with a SeaTech 25cm transmissometer. The instrument is well maintained and frequently recalibrated. Data were recalibrated against bottle salinities and reversing thermometer data or high quality deep T/S profiles at NIOZ.

#### Dr. Avan Antia

Digital reversing thermometer data from instruments of unknown make were written onto CTD log sheets. Between one and three thermometers were fitted per cage and one or two cages were fitted to the CTD rosette on each

cast. Replicate readings were invariably within 0.1 degrees and have been averaged to give the values in the database.

Bottle salinity data for cruise Meteor 27\_1 were analysed on a Guildline Autosal. Standard sea water samples were analysed at intervals and came out 0.002 to 0.003 PSU low. The samples were analysed in July 1994 but were collected in January of that year.

On Meteor 30\_1, the CTD rosette was inoperative and the water samples for salinity were taken from a bottle cast immediately following the CTD cast.

#### IfM Kiel

CTD data were taken using a Neil Brown CTD believed to be a Mk 3. Temperature calibration was confirmed by digital reversing thermometer data and salinity was back calibrated against bottle data assayed by salinometer.

Attenuance values were measured using a SeaTech 25cm path length red light (661nm) transmissometer. On Poseidon 200\_7 a normalisation correction was required to get reasonable clear water data. See the CTD data document from this cruise for more details.

#### British Oceanographic Data Centre

Meteor M27\_1

CTD data were taken by a Neil Brown MK 3 CTD. 16 Hz ASCII data in engineering units were supplied to BODC where they were reduced to 1Hz. Data were screened on a graphics workstation to accurately delimit the downcast and flag out any spikes. Salinity was calibrated against the bottle data set and by comparison of deep T/S curves on stations OMEX2 and OMEX3 with known good quality data from other cruises.

#### Valdivia VLD137

CTD data were taken by a Neil Brown Mk 2 CTD. Raw EG&G binary files at 16 Hz were supplied to BODC on archive tapes. ASCII data in engineering units reduced to 1db were obtained using the Chelsea Instruments Aquasoft software. Data were screened on a graphics workstation to accurately delimit the downcast and flag out any spikes. Salinity and temperature were initially calibrated against classical reversing thermometer and bottle salinity data. However, comparison of deep T/S curves with other cruises showed the calibration data set to be unreliable and the final calibration has been determined from these curves.

#### Charles Darwin CD85

A Neil Brown Mk 3 CTD provided by the Defence Research Agency was used. Temperatures were checked against SIS digital reversing thermometers and salinity calibrated against bottle salinity data.

#### Cruises Charles Darwin CD84, CD94 and Discovery DI216

An RVS Neil Brown Mk 3B CTD was used with a SeaTech 25cm path length red light (661 nm) transmissometer fitted to the cage. On cruise CD84, the package also included a Chelsea Instruments Aquatracka configured as a nephelometer. Temperatures were checked against SIS digital reversing thermometers and salinity calibrated against bottle salinity data. Transmissometer data were corrected for source decay using the air readings during the cruise and the air reading taken from the instrument with a new source. No additional processing has been done on the nephelometer data.

#### Poseidon PS211

Conversion factors from  $\mu M$  to  $\mu mol/kg$  were computed for the non-toxic sampling times using thermosalinograph data. The data used had been despiked using a graphics editor but the calibrations could not be checked or modified as no CTD or bottle data were available.

#### Dr. Mike Fasham

Reversing thermometer temperatures were recorded using SIS digital reversing thermometers.

Salinity samples were taken in medicine bottles. After rinsing, the bottle was filled up to the shoulder, carefully dried off and then sealed with a plastic stopper under the cap. Salinities were determined by taking triplicate readings on a Guildline Autosal bench salinometer as soon as the samples had come to laboratory temperature (generally 24-36 hours after sampling). The instrument was standardised against OSI standard seawater.

CTD profiles were taken using a Neil Brown Mk 3B CTD with a SeaTech 1m path length red light (661 nm) transmissometer. Temperatures were based on a pre-cruise tank calibration. Salinity was calibrated against bottle data. Transmissometer data were corrected for source decay using the air readings during the cruise and the air reading taken from the instrument when new.

#### Dr. lan Joint

Classical mercury in glass protected and unprotected reversing thermometers were deployed on CTD bottle mounts. Water samples were taken from a subset of the CTD bottles and analysed using a bench salinometer of unknown type.

#### Dr. Pete Bowyer

CTD data were collected using an Applied Microsystems STD 12 plus CTD system incorporating a Keller stainless steel pressure transducer, a precision thermistor and a platinised four-electrode conductivity cell. Salinity has been calibrated against bottle data on one SEFOS cruise and the resulting correction applied to all other cruises where the instrument was used.

#### **Research Vessel Services**

Temperature measurements are made using SIS digital reversing thermometers. Two or three instruments are mounted together in a reversing cage to provide duplicate data and an indication of occasions when the cage has failed to reverse cleanly. Each thermometer is periodically calibrated at the RVS laboratory facility and a correction, in the form of a third order polynomial, determined. These corrections are routinely applied. Data in the database are the averages of the readings from all thermometers in the cage after fliers (such as caused by the reading being written down incorrectly) have been eliminated.

Salinity samples were taken in medicine bottles. After rinsing, the bottle was filled up to the shoulder, carefully dried off and then sealed with a plastic stopper under the cap. Salinities were determined by taking triplicate readings on a Guildline Autosal bench salinometer as soon as the samples had come to laboratory temperature (generally 24-36 hours after sampling). The instrument was standardised against OSI standard seawater.

#### IfM Hamburg

CTD profiles were taken using a SeaBird SBE 9 CTD. Temperatures were checked using digital reversing thermometers and salinity was corrected against salinometer determinations on bottle samples.

Surface underway temperatures were taken using a bucket on the end of a rope to collect water samples. The sample temperature was taken using a WTW LF191 temperature and salinity probe. The originators reported that the LF191 was reading 0.2 °C low. This correction has been applied to the data held in the database by BODC.

#### NIOZ Texel

Water samples from CTD rosette bottles were analysed on board ship using a Guildline model 8400 Autosal together with software developed at NIOZ. The instrument was calibrated against standard sea water.

#### Dr. Robin Pingree

CTD data were collected using a Neil Brown Mk3B CTD belonging to RVS. The temperature sensor was calibrated immediately prior to cruise Charles Darwin CD83. Salinity was calibrated against salinometer determinations on bottle samples.

#### Dr. David Hydes

Salinity samples were taken in medicine bottles. After rinsing, the bottle was filled up to the shoulder, carefully dried off and then sealed with a plastic stopper under the cap. Salinities were determined by taking triplicate readings on a Guildline Autosal bench salinometer as soon as the samples had come to laboratory temperature (generally 24-36 hours after sampling). The instrument was standardised against OSI standard seawater.

#### Professor Kurt Tande

On cruises JM1 to JM5 a Neil Brown Mk3B CTD was used. This was replaced by a Meerestechnik OTS-1200 for the remaining cruises.

#### Defence Research Agency

Samples were taken from the CTD rosette in medicine bottles sealed with plastic stoppers and returned to shore for analysis using a Guldline Autosal bench salinometer standardised against OSI standard seawater.

#### Dr. Ricardo Prego

CTD profiles were measured using a SeaBird SBE-19 CTD package. No calibration sample data or information on calibration accuracy are available. Inspection of the data during quality control showed remarkably smooth temperature profiles but very noisy salinity data that required heavy flagging, especially on temperature gradients. The bottle equivalent values were computed after the data had been quality controlled by BODC.

#### Dr. Laurenz Thomsen

Temperature and salinity were determined very close (within 50cm) to the sea bed using the BIOPROBE lander (Thomsen et al., 1994). The lander was deployed on a conductor cable and gently positioned on the sea bed with approximately 20m of slack cable. Penetration into the sediment was determined by a graduated rod monitored by a video camera.

After the material disturbed by the instrument deployment had been seen from transmissometer readings to have dispersed, a pumped water sample was taken in response to a command signal from the ship through one of a group of inlets at varying distances from the seabed. An aliquot of the water collected was used for a bench salinometer salinity determination. Temperature was measured at the time of sample collection by a thermistor located at the water inlet.

### **Comments on Data Quality**

#### Meteor 27\_1

The scatter in the difference between CTD readings and the bottle data was much larger than is usually encountered, even for deep samples. For two of the casts, all bottle values are clearly about 0.02 PSU low and these have been flagged suspect. It is possible that the samples had degraded between collection and analysis. The CTD calibration would have been significantly different had the T/S curve comparison not been done.

#### Valdivia VLD137

The reversing thermometer and bottle salinity data from this cruise were shown to be unreliable by comparison with high quality data from other cruises. They have therefore been flagged suspect in the database.

#### Charles Darwin CD84

A significant number of the bottle salinity data have been flagged suspect in the database. These are from water bottles where there is strong evidence for contamination by surface waters through bottle leakage.

#### Charles Darwin CD85

The salinity samples were analysed on land some time after the cruise. The data were accompanied by a note from the analyst reporting that salt crystals were observed in some of the bottles. However, in cases where several samples were taken within a well defined mixed layer all salinities were within 0.005 PSU and no problems were encountered with the data during CTD calibration. Consequently, it is believed that this observation is not associated with any serious data quality problems.

#### Jan Mayen JM1-JM5

Temperature and salinity records from the instrument used on these cruises were unusually noisy to the extent that absolute accuracy was compromised.

#### **Cruise Discovery DI217**

The CTD pressure sensor gave rise to problems during this cruise and for the first three stations only wire out data were available. Whilst every effort has been made to correct the pressure channel and accurately match bottle firing

pressures, the possibility for error should be borne in mind by users of the data.

Only limited reversing thermometer data were available and these did not compare well with the CTD data. The reversing thermometer data have all been flagged suspect and the CTD temperatures have been assumed to be accurate.

# Irradiance

### **Parameter Code Definitions**

- IRRDPP01 Downwelling 2-pi PAR irradiance Hemispherical photodiode light meter MicroEinsteins/square metre/second
- IRRUPP01 Upwelling 2-pi PAR irradiance Hemispherical photodiode light meter MicroEinsteins/square metre/second

### **Originator Code Definitions**

#### **Charles Darwin cruise CD84**

16		British Oceanographic Data Centre
Disco	very cruise DI217	
37	Dr. Mike Fasham	Southampton Oceanography Centre

### **Originator Protocols**

#### Dr. Mike Fasham and British Oceanographic Data Centre

The data presented in the BOTDATA table are derived from CTD **downcast** data at the bottle firing depths. Note that the interpolation was done on log transformed data to allow a linear technique to be used.

The data were collected by Plymouth Marine Laboratory designed light meters based on a photodiode under a hemispherical translucent white plastic cap. The sensors were designed to collect light across the visible portion of the spectrum.

The light meters were fitted to the CTD frame with the downwelling instrument projecting above the top of the bottle rosette and the upwelling instrument attached to the base of the cage. This gave a physical separation of approximately two metres.

The data were logged as voltages and converted to W/m<sup>2</sup> using laboratory calibrations. Note that the instruments were rebuilt and recalibrated during the summer of 1995, shortly prior to DI217. The calibrations used for CD84 were over five years old. The data were converted to  $\mu E/m^2/s$  using an empirically derived conversion factor of 3.75.

# **Volume of Water Filtered**

#### **Parameter Code Definitions**

VOLFFMXX Volume filtered Flow meter Litres

### **Originator Code Definitions**

#### Belgica cruise BG9309

14	Dr. Lei Chou	ULB, Brussels, Belgium
30	Dr. Patrick Dauby	University of Liege, Belgium

#### Belgica cruises BG9322, BG9412 and BG9506

30	Dr. Patrick Dauby	University of Liege, Belgium
	,	

#### Pelagia cruise PLG95B

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

#### Charles Darwin cruise CD84 and Discovery cruise DI216

14 Dr. Lei Chou ULB, Brussels, Belgium

#### **Originator Protocols**

#### Dr. Lei Chou

The volume filtered during a stand-alone pump (SAP) deployment is measured by reading the flow meter on the instrument before and after deployment.

#### Dr. Patrick Dauby

Flow rate to the centrifuge is controlled and monitored. The volume filtered may therefore be estimated from the flow rate and the sampling duration.

#### Dr. Tjeerd van Weering

The volume filtered during a stand-alone pump (SAP) deployment is measured by reading the flow meter on the instrument before and after deployment.

### **Comments on Data Quality**

The volume filtered parameter is only included as an indication of the quantity of water sampled and should not be used for quantitative purposes. In particular, SAP filters are prone to bursting. A burst filter will use perfectly adequate material for determination of suspended particulate material composition, but the volume filtered will be a gross overestimate.

### **Parameter Code Definitions**

- C400E00A Autotrophic nanoflagellate (2-20 micron) biomass as carbon Epifluorescence microscopy with DAPI/Proflavine stain milligrams/cubic metre
- C400E00B Heterotrophic nanoflagellate (2-20 micron) biomass as carbon Epifluorescence microscopy with DAPI/Proflavine stain milligrams/cubic metre
- MZBCMITX Total microzooplankton biomass (expressed as carbon) Calculated from cell counts determined by optical microscopy milligrams/cubic metre
- MZBNMITX Total microzooplankton abundance (cell numbers) Optical microscopy Number per millilitre
- MZCTDDTX Daily chlorophyll turnover by microzooplankton grazing Dilution experiment (shipboard incubation) Per cent
- MZCTDITX Daily chlorophyll turnover by microzooplankton grazing Dilution experiment (in-situ incubation) Per cent
- P400E00A Autotrophic nanoflagellates (2-20 micron) Epifluorescence microscopy with DAPI/Proflavine stain Number per millilitre
- P400E00B Heterotrophic nanoflagellates (2-20 micron) Epifluorescence microscopy with DAPI/Proflavine stain Number per millilitre

### **Originator Code Definitions**

Belgica cruises 9322 and 9506, Pelagia cruise PLG93, Poseidon cruise PS200\_7, Charles Darwin cruises CD84, CD85, CD86 and CD94, Valdivia cruise VLD137, Discovery cruises DI216 and DI217

84	Dr. Peter Burkill	Plymouth Marine Laboratory, UK
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### **Originator Protocols**

#### Dr. Peter Burkill

Water samples were obtained from water bottles deployed on a CTD rosette. These were fixed with 1% Lugol's iodine and the microzooplankton were counted using an image analysis system coupled to an inverted microscope. Fixed samples were gently mixed and sub-samples of 30-100 ml were concentrated overnight in sedimentation chambers. Each sample was examined at a magnification of x300 and all grazers > circa 10 microns were counted. Cells were identified to genus level whenever possible.

Live video work together with fluorescence microscopy at sea enabled the separation of heterotrophic dinoflagellates from phototrophic forms. All ciliates were assumed to be heterotrophic. In order to obtain a more accurate identification of some ciliates, Protargol silver staining was carried out on a number of samples.

The biomass was determined using methods detailed in JGOFS protocols (Burkill et al., 1994). The image analysis system was used to generate data on the surface area of each cell. These were converted to cell volume using geometric formulae and standard volume to carbon conversion factors were applied for different taxa. Individual cell carbon volumes were integrated for discrete taxa to determine the biomass of those taxa in each water sample.

Samples were also collected for the determination of nanoplankton (2-20 micron) abundance and biomass. Samples were fixed in 0.3% glutaraldehyde, dual stained with DAPI and proflavine and filtered onto 0.4 micron black polycarbonate filters. Cells were counted by epifluorescence microscopy. Heterotrophs were distinguished from autotrophs by the presence or absence of chlorophyll autofluorescence. 1-200 flagellate cells were counted per filter and cell dimensions were measured with an ocular micrometer. Flagellate cell volumes were calculated assuming they were ellipsoids. Biovolumes were converted to biomass using appropriate carbon conversion factors.

Natural microbial populations were incubated either in-situ or on board ship using the 'dilution technique described by Landry and Hassett (1982). Time course experiments were run under different dilutions and the specific growth of phytoplankton determined. Water samples were collected at dawn from a depth of 10m using 30 litre Niskin bottles. Half of this water was filtered using a Gelman 0.2 micron mini capsule filter. A known volume of this 'predator and prey free' water was added to polycarbonate bottles. Each bottle was gently topped up with 200 micron screened, unfiltered water generating triplicate dilutions of 100%, 70%, 40% and 10%. Incubation was carried out over 24 hours. Sub-samples were taken from each bottle at T0 and T24 for determination of chlorophyll and fixation in Lugol's iodine for estimation of microzooplankton abundance. Chlorophyll was determined by extraction of 90% acetone, using a highly sensitive fluorometer. Phytoplankton mortality due to grazing was determined from alteration in the specific growth rate.

Qualitative analysis of microzooplankton herbivory was determined by incubating natural microbial populations with different types of fluorescently labelled algae (FLA). Cultured algae were heat stained with a fluorochrome, 5-(4,6-dichlorotriazan-2-yl) aminofluorescein (DTAF). Water samples were innoculated with FLA stock and experiments run for 60 minutes. Subsamples were collected over the experimental period and fixed in 1% Lugol's iodine. Up to 110 ml of sample were settled for 24 hours prior to analysis and the Lugol's coloration cleared with sodium thiosulphate. The FLAs present in each microzooplankton cell were counted and FLA uptake rates were calculated from the change in the average number of FLAs per individual with time. The mean cellular uptake rates were calculated for those taxa taking up FLAs in each experiment.

# **Phytoplankton Species Counts**

### **Parameter Code Definitions**

- P000M00Z Diatoms Optical microscopy Number per millilitre
- P017M02Z Bacillaria paradoxa Optical microscopy Number per millilitre
- P018M03Z Bacteriastrum delicatulum Optical microscopy Number per millilitre
- P028M01Z Cerataulina pelagica Optical microscopy Number per millilitre
- P030M00A Chaetoceros spp. (hyalochaete) Optical microscopy Number per millilitre
- P030M00B Chaetoceros spp. (phaeoceros) Optical microscopy Number per millilitre
- P067M01Z Lauderia borealis Optical microscopy Number per millilitre
- P068M01Z Leptocylindrus danicus Optical microscopy Number per millilitre
- P068M03Z Leptocylindrus minimus Optical microscopy Number per millilitre
- P073M00Z Navicula spp. Optical microscopy Number per millilitre

- P074M18Z Nitzschia delicatissima Optical microscopy Number per millilitre
- P074M61Z Nitzschia seriata Optical microscopy Number per millilitre
- P074M72Z Nitzschia longissima Optical microscopy Number per millilitre
- P093M02Z Rhizosolenia alata Optical microscopy Number per millilitre
- P093M13Z Rhizosolenia fragilissima Optical microscopy Number per millilitre
- P093M24Z Rhizosolenia styliformis Optical microscopy Number per millilitre
- P110M01Z Thalassionema nitzschiodes Optical microscopy Number per millilitre
- P111M00Z Thalassiosira spp. Optical microscopy Number per millilitre
- P112M02Z Thalassiothrix longissima Optical microscopy Number per millilitre
- P200M00Z Dinoflagellates Optical microscopy Number per millilitre
- P213M08Z Ceratium furca Optical microscopy Number per millilitre
- P213M09Z Ceratium fusus Optical microscopy Number per millilitre

- P213M15Z Ceratium lineatum Optical microscopy Number per millilitre
- P213M18Z Ceratium macroceros Optical microscopy Number per millilitre
- P213M26Z Ceratium tripos Optical microscopy Number per millilitre
- P219M28Z Dinophysis punctata Optical microscopy Number per millilitre
- P219M30Z Dinophysis rotundata Optical microscopy Number per millilitre
- P228M10Z Gonyaulax grindleyi Optical microscopy Number per millilitre
- P228M17Z Gonyaulax polygramma Optical microscopy Number per millilitre
- P230M01Z Gyrodinium aureolum Optical microscopy Number per millilitre
- P236M01Z Heterocapsa triquetra Optical microscopy Number per millilitre
- P236M99Z Heterocapsa minima Optical microscopy Number per millilitre
- P240M02Z Mesoporus perforatus Optical microscopy Number per millilitre
- P257M02Z Prorocentrum balticum Optical microscopy Number per millilitre

- P257M04Z Prorocentrum compressum Optical microscopy Number per millilitre
- P257M05Z Prorocentrum dentatum Optical microscopy Number per millilitre
- P257M08Z Prorocentrum micans Optical microscopy Number per millilitre
- P349M01Z Oxytoxum scolopax Optical microscopy Number per millilitre
- P349M98Z Oxytoxum sphaeroideum Optical microscopy Number per millilitre
- P349M99Z Oxytoxum caudatum Optical microscopy Number per millilitre
- P358M20Z Protoperidinium depressum Optical microscopy Number per millilitre
- P366M99Z Peridinium faeroense Optical microscopy Number per millilitre
- P400M00E Silicoflagellates Optical microscopy Number per millilitre
- P400M00Z Flagellates Optical microscopy Number per millilitre
- P426M01Z Distephanus speculum Optical microscopy Number per millilitre
- P427M04Z Halosphaera viridis Optical microscopy Number per millilitre

- P436M00Z Phaeocystis spp. Optical microscopy Number per millilitre
- P490M00Z Coccolithophores Optical microscopy Number per millilitre
- P498M00Z Dinobryan spp. Optical microscopy Number per millilitre
- P499M00Z Monoraphis spp. Optical microscopy Number per millilitre
- P530M00Z Protozoa Optical microscopy Number per millilitre
- P999M01Z Paleophalacroma unicinctum Optical microscopy Number per millilitre
- PU01M00Z Undifferentiated picophytoplankton Optical microscopy Number per millilitre
- PU02M00Z Undifferentiated algal fragments Optical microscopy Number per millilitre

### **Originator Code Definitions**

#### Cruises Valdivia VLD137, Charles Darwin CD85 and Discovery DI217

3 Dr. Ian Joint Plymouth Marine Laboratory, UK

#### Cruises Jan Mayen JM1-JM8

61 Dr. Paul Wassmann University of Tromsø, Norway

### **Originator Protocols**

#### Dr. Ian Joint

Water samples were taken from bottles deployed on a CTD rosette or a kevlar hydrographic wire and preserved in Lugol's lodine. Back in the laboratory, sedimented samples were examined by optical microscopy and the dominant species in the >5 micron size fraction were quantified.

Data were supplied in units of cells per litre and were converted to cells per ml at BODC by dividing by 1000.

#### Dr. Paul Wassmann

Water samples were taken from the CTD rosette and approximately 50 ml samples were fixed with glutardialdehyde-Lugol solution. Back in the laboratory, the samples were counted using a non-inverted light microscope furnished with a counting stage. The whole sample was gently mixed using a large bore pipette which was slowly emptied into the bottle as it was moved from bottom to top, keeping the end barely immersed.

Counting of the picoplankton and most abundant nanoplankton was carried out in a Fuchs-Rosental counting chamber with 400 times magnification.

After these had been counted, the sample was allowed to settle for about a week before being slowly decanted through a glass tube covered with two layers of fine-mesh nylon gauze. After gentle mixing, the portion of the sample that remained was removed using a glass tube and placed into a 0.05 ml chamber. Cells were counted using 200 times magnification.

Rare, usually larger, forms were counted in a special 1 ml chamber using 100 times magnification.

# **Zooplankton and Terrestrial Detritus**

### **Parameter Code Definitions**

- EXUVMIXX Exuvia Optical microscopy Number per litre
- TD00M00Z Pine plus birch pollen Optical microscopy Number per litre
- TD10M00Z Fungal threads plus acsospores Optical microscopy Number per litre
- Z000M00Z Copepodites Optical microscopy Number per litre
- Z500M00Z Gastropoda Optical microscopy Number per litre
- Z510M00Z Bivalvia Optical microscopy Number per litre
- Z520M00Z Ostracoda Optical microscopy Number per litre
- Z530M00Z Nematoda Optical microscopy Number per litre
- Z540M00Z Pteropoda Optical microscopy Number per litre
- Z601M01Z Fritillaria borealis Optical microscopy Number per litre

- Z701M00Z Testacida spp. Optical microscopy Number per litre
- ZU00M00Z Undifferentiated zooplankton Optical microscopy Number per litre
- ZU01M00Z Undifferentiated eggs Optical microscopy Number per litre
- ZU02M00Z Undifferentiated faecal pellets Optical microscopy Number per litre
- ZU03M00Z Undifferentiated naupli Optical microscopy Number per litre

### **Originator Code Definitions**

#### Cruises Jan Mayen JM1-JM11

61 Dr. Paul Wassmann University of Tromsø, Norway

### **Originator Protocols**

#### **Dr. Paul Wassmann**

Water samples were taken from the CTD rosette and approximately 50 ml samples were fixed with glutardialdehyde-Lugol solution. Back in the laboratory, the samples were counted using a non-inverted light microscope furnished with a counting stage. The whole sample was gently mixed using a large bore pipette which was slowly emptied into the bottle as it was moved from bottom to top, keeping the end barely immersed.

Counting of the picoplankton and most abundant nanoplankton was carried out in a Fuchs-Rosental counting chamber with 400 times magnification.

After these had been counted, the sample was allowed to settle for about a week before being slowly decanted through a glass tube covered with two layers of fine-mesh nylon gauze. After gentle mixing, the portion of the sample that remained was removed using a glass tube and placed into a 0.05 ml chamber. Cells were counted using 200 times magnification.

Rare, usually larger, forms were counted in a special 1 ml chamber using 100 times magnification.

# Radionuclides

# Parameter Code Definitions

AM41GSD2	Dissolved americium-241 Gamma-ray spectroscopy (0.4/0.45 µm pore filtered) Microbequerels per litre
CS37GSD2	Dissolved caesium-137 Gamma-ray spectroscopy (0.4/0.45 µm pore filtered) Bequerels per litre
PU09GSD2	Dissolved plutonium-239 plus plutonium-240 Gamma-ray spectroscopy (0.4/0.45 µm pore filtered) Microbequerels per litre
PU38GSD2	Dissolved plutonium-238 Gamma-ray spectroscopy (0.4/0.45 µm pore filtered) Microbequerels per litre
SE09GSD2	Dissolved plutonium-239 plus plutonium-240 standard error Gamma-ray spectroscopy (0.4/0.45 $\mu m$ pore filtered) Microbequerels per litre
SE37GSD2	Dissolved caesium-137 standard error Gamma-ray spectroscopy (0.4/0.45 µm pore filtered) Bequerels per litre
SE38GSD2	Dissolved plutonium-238 standard error Gamma-ray spectroscopy (0.4/0.45 µm pore filtered) Microbequerels per litre
SE90GSD2	Dissolved strontium-90 standard error Gamma-ray spectroscopy (0.4/0.45 µm pore filtered) Bequerels per litre
SR90GSD2	Dissolved strontium-90 Gamma-ray spectroscopy (0.4/0.45 µm pore filtered) Bequerels per litre

### **Originator Code Definitions**

#### **Cruise Charles Darwin CD84**

99 Dr. M. Thouard DIRCEN-CEA/SMSR, Monthlery, France

### **Originator Protocols**

#### Dr. M. Thouard

300-litre water samples were collected using a custom-made large water bottle or were drawn from the non-toxic supply. The samples were transferred to 30-litre polythene bottles and filtered through 293mm diameter 0.45 micron porosity membranes as quickly as possible. The filtrate was acidified to pH 1.7 with hydrochloric acid to stabilise the radionuclides in the polythene bottles.

Back at the laboratory, radiochemical treatments specific to each radionuclide were applied followed by low-level counting.

# **Atmospheric Radon**

### **Parameter Code Definitions**

- BI14APAX Atmospheric bismuth-214 APIA monitoring of continuously pumped air Microbequerels per litre
- PO18APAX Atmospheric polonium-218 APIA monitoring of continuously pumped air Microbequerels per litre

### **Originator Code Definitions**

#### Meteor cruise M30\_1

55 Dr. Veit Ulshofer Max Planck Institute, Germany

### **Originator Protocols**

#### Dr. Veit Ulshofer

Air was continuously pumped from an inlet located on a beam extending into the air flow just above the flying bridge, approximately 30 m above sea level. A tube, less than 5 m in length, carried the sample into the ship's air chemistry laboratory.

<sup>222</sup>Rn was recorded continuously via the decay products <sup>214</sup>Bi and <sup>218</sup>Po using an APIA monitor. The counts were integrated over periods of two hours.

The data were provided in units of Bequerels per cubic metre and have been converted to Microbequerels per litre by multiplying them by 1000.

# **Current Parameters**

### **Parameter Code Definitions**

- BSHVESXX Estimated bottom shear velocity Determined from surface roughness determined using bottom photography Centimetres/second
- CSERTFBL Current speed standard error Thermistor flow meter mounted on a benthic lander Centimetres/second
- LCSATFBL Current speed Thermistor flow meter mounted on a benthic lander Centimetres/second
- TINTTFBL Turbulence intensity Thermistor flow meter mounted on a benthic lander Per cent

### **Originator Code Definitions**

Pelagia cruises PLG93 and PLG95A, Charles Darwin cruise CD86 and Meteor cruise M27\_1 and M30\_1

96 Dr. Laurenz Thomsen GEOMAR, Kiel, Germany

### **Originator Protocols**

#### Dr. Laurenz Thomsen

The inclusion of current parameters in a water bottle data set may initially seem surprising. However, these data were collected in conjunction with a number of conventional water bottle parameters by a benthic water sampling lander. Their management as bottle parameters therefore enables all elements of a related data set to be maintained as a single entity.

Water samples were collected using the BIOPROBE benthic water sampling lander (Thomsen et al., 1994). This was deployed on a conductor cable and gently positioned on the sea bed with approximately 20m of slack cable.

Penetration into the sediment was determined by a graduated rod monitored by a video camera.

The lander was equipped with one or more ADM instruments thermistor flow meters. The data were transmitted up the umbilical cable and logged on the ship. Mean current speed, standard error of the current speed and turbulence intensity were computed over the duration of the lander deployment.

Cameras on the lander provided seabed photographs from which bottom roughness and hence shear velocity could be estimated.

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