

# Water Sample Data Documentation

## Introduction

During SES a total of 68 different parameters were measured on water samples by 11 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 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.

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Counts of the dominant phytoplankton taxa for the stations selected for productivity experiments.

## **References**

A bibliography covering all the water bottle data documentation.

# Photosynthesis Parameters

## Parameter Code Definitions

ALPHPIPX Quantum yield (alpha)  
P:I incubation (whole sample counted after residual inorganic  $^{14}\text{C}$   
removed by acidification)  
mg C/( $\mu\text{E}/\text{m}^2/\text{s}$ )/mg chl/hour

PMAXPIPX Photosynthetic maximum ( $P_{\text{max}}$ )  
P:I incubation (whole sample counted after residual inorganic  $^{14}\text{C}$   
removed by acidification)  
mg C/mg chl/hour

## Originator Code Definitions

**Charles Darwin cruise CD93B and Challenger cruises CH121B, CH126B and CH128A**

34 Dr. Graham Savidge Queen's University, Belfast

## Originator Protocols

### Dr. Graham Savidge

Water samples were collected from water bottles deployed on a CTD rosette. These were inoculated with  $^{14}\text{C}$  sodium bicarbonate and incubated at a range of light levels from 1.5 to 1500  $\mu\text{E}/\text{m}^2/\text{s}$  in a photosynthetron. At the end of the incubation the remaining inorganic  $^{14}\text{C}$  was removed by acidification. The level of  $^{14}\text{C}$  incorporated by the particulate phase was then determined using an LKB scintillation counter. The complete procedure was undertaken on board ship.

# Dissolved Organic Carbon

## Parameter Code Definitions

CORGCOD1 Dissolved organic carbon  
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

## Originator Code Definitions

**Challenger cruises CH121B, CH123B, CH125B, CH126B and CH128A.**

13 Dr. Axel Miller CCMS Plymouth Marine Laboratory

## Originator Protocols

### Dr. Axel Miller

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

The analytical technique involves the direct injection of acidified and decarbonated seawater onto a platinised alumina catalyst at high temperature (680-900 °C) under an atmosphere of oxygen or high purity air. Quantitative production of CO<sub>2</sub> gas allows DOC concentrations to be determined using a CO<sub>2</sub>-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**

The data set was scrutinised by the data originator and a number of values were identified as anomalously high. These have been flagged suspect in the database.

# Particulate Organic Carbon and Nitrogen

## Parameter Code Definitions

- CORGCAP1 Particulate organic carbon (acidified)  
Acid fumed then C/N analyser (GF/F filtered)  
Micromoles/litre
- CORGCNP1 Particulate “organic” carbon (unacidified)  
Carbon/nitrogen analyser (GF/F filtered)  
Micromoles/litre
- NTOTCNP1 Particulate total nitrogen (“PON”)  
Carbon/nitrogen analyser (GF/F filtered)  
Micromoles/litre

## Originator Code Definitions

**Charles Darwin cruises CD93A and CD93B and Challenger cruises CH121B, CH121C, CH123A, CH123B, CH125A, CH125B, CH126A, CH128A and CH128B.**

24 Dr. Paul Tett University of Wales, Bangor

## Originator Protocols

### Dr. Paul Tett

Water samples were collected using Niskin bottles deployed on a CTD rosette and filtered through 25 mm GF/F filters. On most cruises, two aliquots were filtered to provide samples for the determination of particulate total carbon and particulate organic carbon. Note that this procedure resulted in duplicate determinations of total particulate nitrogen. The nitrogen data stored in the database are exclusively from the POC samples to prevent sample heterogeneity distorting organic C/N ratios. The samples were stored frozen until analysed.

The thawed filters were dried at 60°C overnight in an oven, inside a pre-cleaned open plastic dish. The POC samples were placed into a pre-cleaned hermetic plastic container inside a pre-cleaned open plastic dish. Using an excess quantity

of Analar concentrated HCl in a 50 ml beaker, the samples were exposed directly to HCl vapour for 30-48 h at room temperature. This acidification procedure removed inorganic carbon present as carbonate. The samples were removed and heated in an oven for 1 h at 60°C to drive off residual HCl and water before they were analysed. Untreated and acidified samples were handled separately to avoid residual acid vapours in the HCl-treated samples reacting with carbonates in untreated samples.

Each TPC and POC sample was folded carefully into a rectangular shape, using sterilised forceps, inside a 30x30 mm square pre-cleaned tin foil. This maximised the oxidation reaction in the combustion chamber and allowed the sample to pass through the CHN analyser oxidation furnace entrance.

The carbon and nitrogen analyses were carried out at Dunstaffnage Marine Laboratory using a LECO CHN-900 Elemental Analyser with helium as the carrier gas and pure oxygen for combustion. Simultaneous determination of the carbon and nitrogen content of the samples was achieved by measuring the products of combustion using non-dispersive infrared detection and thermal conductivity.

The CHN analyser was calibrated using a known weight of a suitable standard organic compound having a known carbon and nitrogen content: acetanilide ( $\text{CH}_3\text{CONHC}_6\text{H}_5$ ) containing 71.09% of carbon and 10.36% of nitrogen. Between 1995 and 1997 five visits were made to DML to analyse the samples. To avoid any errors resulting from changes in instrument settings, a separate calibration was made on each visit. Each calibration involved analyses of standards on each day of use to check for calibration drift during a visit. Four empty tin foils were analysed on each calibration run as blanks.

The units of the data were converted from  $\text{mg/m}^3$  to  $\mu\text{M}$  at BODC by dividing the carbon values by 12.011 and the nitrogen values by 14.007.

## **Comments on Data Quality**

The data were examined at BODC and a number of obviously anomalous values were flagged suspect, including one complete profile that appeared to be inverted.

The unacidified carbon values exceeded the acidified values for a significant number of samples. The two carbon determinations were done on separate filtrations and consequently small negative differences are to be expected due to sample heterogeneity. However, in some cases the acidified carbon values were up to  $10\mu\text{M}$  greater.

In these cases, if one of the two values was clearly anomalous then it was flagged. However, users should be aware that in some cases it was impossible to ascertain which of the two values was in error and no flags were applied.

# Nutrients

## Parameter Code Definitions

AMONAATX	Ammonium (unfiltered) Colorometric autoanalysis (unfiltered) Micromoles/litre
NTRZAAD1	Dissolved nitrate + nitrite Colorometric autoanalysis (GF/F filtered) Micromoles/litre
NTRZAATX	Nitrate + nitrite (unfiltered) Colorometric autoanalysis (unfiltered) Micromoles/litre
PHOSAAD1	Dissolved phosphate Colorometric autoanalysis (GF/F filtered) Micromoles/litre
PHOSAATX	Phosphate (unfiltered) Colorometric autoanalysis (unfiltered) Micromoles/litre
SLCAAATX	Silicate (unfiltered) Colorometric autoanalysis (unfiltered) Micromoles/litre

## Originator Code Definitions

### Charles Darwin cruises CD92B, CD93A and CD93B and Challenger cruises CH120 and CH121B

115	Dr. Roger Proctor	CCMS Proudman Oceanographic Laboratory
59	Dr. Ken Jones	CCMS Dunstaffnage Marine Laboratory

### Challenger cruises CH123B, CH124, CH125B, CH126B and CH128A

59	Dr. Ken Jones	CCMS Dunstaffnage Marine Laboratory
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## Originator Protocols

### Dr. Roger Proctor

Samples were collected using either Niskin bottles deployed on a CTD rosette (CD93A, CD93B and CH121B) or NIO bottles deployed on a hydrographic wire in conjunction with a CTD cast (CD92B and CH120). The samples were immediately frozen at  $-18^{\circ}\text{C}$ . The samples were transferred to the laboratory (CCMS Dunstaffnage Marine Laboratory) in dry ice and stored frozen until analysed.

Some 2-3 months after the cruise, the samples were thawed, filtered through a GF/F filter and analysed on a Lachat autoanalyser using standard colorimetric chemistry.

### Dr. Ken Jones

Samples were collected using either Niskin bottles deployed on a CTD rosette (CD93A, CD93B, CH121B, CH123B, CH125B, CH126B and CH128A) or NIO bottles deployed on a hydrographic wire in conjunction with a CTD cast (CD92B, CH120 and CH124).

The samples were transferred to a refrigerator at  $4^{\circ}\text{C}$  immediately after collection and were analysed within a few hours on a Lachat autoanalyser using standard colorimetric chemistry.

## Comments on Data Quality

An intercalibration of the two nutrient data sets was carried out. Simple regression, excluding data values deemed suspect, yielded the following equations:

$$\begin{aligned} \text{POL NO}_3+\text{NO}_2 &= 0.93 \text{ DML NO}_3+\text{NO}_2 - 0.14 & (R^2 = 0.80) \\ \text{POL PO}_4 &= 0.84 \text{ DML PO}_4 + 0.07 & (R^2 = 0.78) \end{aligned}$$

The POL nutrient values are systematically significantly lower than the DML data. When individual data values are inspected it can be seen that in some samples the POL values are down to a half of the DML values. This nutrient loss may be attributed to biological activity during sample storage.

The POL data were primarily collected to allow estimation of dissolved organic nitrogen and phosphorus from contemporary total nitrogen and phosphorus determinations. It is therefore recommended that the DML data be used for all considerations of ambient nutrient concentrations and that the POL data be considered as metadata for the TDN and TDP data.

# **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
- NTOTWCD1 Dissolved total nitrogen  
Oxidation then autoanalysis (GF/F filtered)  
Micromoles/litre
- SETNCOD1 Dissolved total nitrogen standard error  
High temperature Pt catalytic oxidation (GF/F filtered)  
Micromoles/litre
- TPHSWCD1 Dissolved total phosphorus  
Oxidation then autoanalysis (GF/F filtered)  
Micromoles/litre

## **Originator Code Definitions**

### **Charles Darwin cruises CD92B, CD93A and CD93B and Challenger cruise CH120**

115 Dr. Roger Proctor CCMS Proudman Oceanographic Laboratory

### **Challenger cruise CH121B**

115 Dr. Roger Proctor CCMS Proudman Oceanographic Laboratory  
13 Dr. Axel Miller CCMS Plymouth Marine Laboratory

### **Challenger cruises CH123B, CH125B, CH126B and CH128A**

13 Dr. Axel Miller CCMS Plymouth Marine Laboratory

## **Originator Protocols**

### **Dr. Roger Proctor**

Samples were collected using either Niskin bottles deployed on a CTD rosette (CD93A, CD93B and CH121B) or NIO bottles deployed on a hydrographic wire in conjunction with a CTD cast (CD92B and CH120). The samples were immediately frozen at  $-18^{\circ}\text{C}$ . The samples were transferred to the laboratory (CCMS Dunstaffnage Marine Laboratory) in dry ice and stored frozen until analysed.

Some 2-3 months after the cruise, the samples were thawed, filtered through a GF/F filter. Sample oxidation was achieved by the addition of hydrogen peroxide followed by ultra-violet irradiation for three hours. Total nitrogen and phosphorus were determined on a Lachat autoanalyser using standard colorometric chemistry.

### **Dr. Axel Miller**

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

The analytical technique involves the direct injection of acidified and decarbonated seawater onto a platinised alumina catalyst at high-temperature ( $680\text{-}900^{\circ}\text{C}$ ) 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 done at sea wherever possible or the samples were stored refrigerated in flame-sealed glass vials. The instrument used was a Shimadzu TOC-5000 HTOC analyser fitted with an Antek 705-D chemiluminescence detector. The combustion products travelled through a Drierite trap (97%  $\text{CaSO}_4$ , 3%  $\text{CoCl}_2$ ) 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.

## **Comments on Data Quality**

### **Charles Darwin CD92B, CD93A and CD93B and Challenger CH120**

The TDN determinations were consistent with the associated nitrate plus nitrite data. Occasional values in both of these data sets were obviously erroneous and have been flagged suspect.

Organic phosphorus levels were consistently low and in many cases were too small to be reliably measured by the method used. Consequently, a significant number of TDP values were marginally less than their associated phosphate values. A number of TDP and phosphate measurements were obviously erroneous and have been flagged suspect.

### **Challenger CH121B**

TDN was measured on a common sample set by both wet chemistry and HTCO. The data were in good agreement as demonstrated by the intercalibration regression equation obtained:

$$\text{PML} = 1.06 * \text{POL} - 1.58 \quad (R^2 = 0.68)$$

Both data sets were consistent with the associated nitrate plus nitrite data.

Organic phosphorus levels were consistently low and in many cases were too small to be reliably measured by the method used. Consequently, a significant number of TDP values were marginally less than their associated phosphate values. A number of TDP and phosphate measurements were obviously erroneous and have been flagged suspect.

### **Challenger CH123B and CH125B**

No problems were noted with the TDN data from this cruise.

### **Challenger CH126B**

There was a serious problem with the total dissolved nitrogen data from this cruise. Without exception, the data values were significantly (often less than half) lower than the nitrate plus nitrite values determined by autoanalysis on fresh samples. No cause could be identified for this problem despite a thorough investigation by the analytical team at PML.

All total dissolved nitrogen data from this cruise have been flagged suspect.

## **Challenger CH128A**

Problems were noted with some of the TDN data from this cruise. Some profiles appeared to be problem-free whilst others (4 out of 10) consisted entirely of data values that were significantly (often less than half) lower than the nitrate plus nitrite values determined by autoanalysis on fresh samples. No cause could be identified for this problem despite a thorough investigation by the analytical team at PML.

All affected profiles have been flagged suspect.

# Pigments

## Parameter Code Definitions

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
CPHLPR01	CTD chlorophyll Calibrated in-situ fluorometer Milligrams/cubic metre
PHAEFLP1	Fluorometric phaeopigments Fluorometric assay of acetone extract (GF/F filtered) Milligrams/cubic metre
SCHLFLPF	Size-fractionated fluorometric chlorophyll-a Fluorometric assay of an acetone extract (0.2-2µm size fraction) Milligrams/cubic metre
SCHLFLPG	Size-fractionated fluorometric chlorophyll-a Fluorometric assay of an acetone extract (2-20µm size fraction) Milligrams/cubic metre
SCHLFLPQ	Size-fractionated fluorometric chlorophyll-a Fluorometric assay of an acetone extract (>20µm size fraction) Milligrams/cubic metre

## Originator Code Definitions

**Charles Darwin cruises CD91B and CD93A and Challenger cruises CH121A, CH121C, CH123A, CH123B, CH125A, CH125B, CH126A and CH128B**

59 Dr. Ken Jones  
16

CCMS Dunstaffnage Marine Laboratory  
British Oceanographic Data Centre

## **Charles Darwin cruise CD93B and Challenger cruises CH121B, CH126B and CH128A**

59	Dr. Ken Jones	CCMS Dunstaffnage Marine Laboratory
34	Dr. Graham Savidge	Queen's University, Belfast
16		British Oceanographic Data Centre

## **CPR Tows 388W, 31Y, 391W, 392W, 394W, 395W, 397W**

132	Mr. Tony Walne	Sir Alister Hardy Foundation for Ocean Science
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## **Originator Protocols**

### **Dr. Ken Jones**

Samples were collected from either the non-toxic surface sea water supply or from Niskin bottles fitted to the CTD rosette. They were taken in Nalgene bottles, rinsed twice with sample water prior to filling.

The samples were vacuum filtered through 2.5 cm GF/F filters. The volume of water filtered varied depending on the particulate load in the sample. The filters were folded and immediately frozen.

Back in the laboratory, the filters were extracted into 8 ml of 90% neutralised acetone and stored for between 18 and 36 hours in a refrigerator. The extracts were centrifuged at 3000 rpm for two 5-minute bursts.

The resulting chlorophyll solutions were assayed on a bench fluorometer. Three drops of 8% HCl were added and the assay was repeated. Chlorophyll-a and phaeopigment concentrations were determined from the two fluorometer readings using the equations in Tett and Grantham (1978).

### **Dr. Graham Savidge**

Water samples were collected using Niskin bottles mounted on the CTD rosette. The samples were filtered through a cascade containing 18, 2 and 0.25  $\mu\text{m}$  polycarbonate pore filters. The particulate material trapped on each filter was extracted into 90% acetone and assayed fluorometrically.

### **British Oceanographic Data Centre**

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 DML

fluorometric chlorophyll-a data by BODC. Details of the individual cruise calibrations may be found in the CTD data documentation.

**Mr. Tony Walne**

The SAHFOS Continuous Plankton Recorder was towed through the SES area of interest on a number of occasions by ships of opportunity. The instrument was fitted with a self-logging Chelsea Instruments Aquapak, including an Aquatracka fluorometer.

No extracted chlorophyll data were available due to the nature of the instrument deployment. Consequently, the logged voltages were converted into chlorophyll concentrations using the manufacturer's data based on chlorophyll standards in acetone.

The instrument sampled every 15 or 20 minutes. These data were reduced to 1 mile averages by the data originator. Times and positions assigned correspond to the mid-point of the tow.

# Dissolved Oxygen

## Parameter Codes

- DOXYPR01 Beckmann oxygen  
Beckmann oxygen probe  
Micromoles/litre
- DOXYWITX Winkler oxygen  
Winkler titration  
Micromoles/litre
- OXYBB01 Oxygen saturation (Benson & Krause/Beckmann)  
Benson & Krause algorithm from Beckmann probe data  
Per cent

## Originator Codes

**Charles Darwin cruises CD93A and CD93B and Challenger cruises CH121B, CH123B, CH125B, CH126B and CH128A**

16 British Oceanographic Data Centre  
24 Dr. Paul Tett University of Wales, Bangor

## Originator Protocols

### Dr. Paul Tett

Samples for oxygen analysis were collected from Niskin bottles on the CTD rosette using a length of silicone tube into calibrated, ground-glass stoppered borosilicate bottles. The bottles were rinsed three times and sample water was allowed to overflow for a short time to ensure that it was free from air bubbles.

Manganous sulphate and alkaline sodium iodide solutions were added to the seawater sample and the bottle was stoppered and shaken. The oxygen in the seawater oxidised some of the hydroxide to a tetravalent manganese compound. The quantity of this was determined by making the solution acid and titrating the liberated iodine with sodium thiosulphate, using potentiometric end-point detection. The protocol followed the enhanced techniques described in Bryan et al. (1976) and used a micro-burette, which enabled high precision in the titration.

The bottles were prepared as follows. After acid-washing and drying, each bottle was allocated a number and a matching stopper and weighed on an Oertling OB33 balance. The bottles were weighed dry twice, with an agreement of  $\pm 3\text{mg}$ ,

and the average weight calculated. The bottles were then filled with tap water, capped with ground glass stoppers, carefully dried and weighed. The average wet weight for each bottle was calculated once weight replicates were less than 10 mg for full bottles. The average of those two weights was then calculated. The difference between average dry and average wet weight was then calculated to give the volume of each bottle in ml.

The thiosulphate was standardised against a standard solution of potassium iodate.

### **British Oceanographic Data Centre**

The data values present in the bottle database 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.

The calibration and data processing protocols for the dissolved oxygen data were complex and varied from cruise to cruise. Further details are given in the CTD data documentation and users are strongly recommended to consult this before using the CTD-derived data.

The oxygen saturation values have been computed from calibrated temperature and salinity data using the algorithm of Benson and Krause (1984).

### **Comments on Data Quality**

In most cases, the calibrated CTD oxygen data are in good agreement with the water bottle data. It should be noted that bottle measurements were only made on a small proportion of the casts but the resulting calibration has been applied to all casts from the cruise.

However, this is not the case for cruise CD93B where the CTD oxygen values are some 10-20% higher than the bottle values. The problem has been investigated but no processing errors could be identified. It is therefore recommended that the CTD-derived data from this cruise be used with extreme caution.

# Iodine

## Parameter Code Definitions

IATEAATX	Iodate Photometric autoanalysis (unfiltered) Nanomoles per litre
IXXAATX	Total iodine Catalysis of Ce-As reaction by autoanalysis (unfiltered) Nanomoles per litre

## Originator Code Definitions

### Challenger cruises CH125B, CH126B and CH128A

122 Dr. Vic Truesdale Oxford Brookes University

## Originator Protocols

Water samples were taken from the non-toxic surface seawater supply and were analysed unfiltered. Samples were stored in glass, at 4° C in the dark for up to 2 months, a mode which has been shown to introduce negligible error (Truesdale, 1968)

Total iodine (iodate plus iodide) was determined catalytically using the  $\text{Ce}^{\text{IV}} - \text{As}^{\text{III}}$  reaction optimised for the Technicon AutoAnalyser II system (Truesdale and Smith, 1975; Truesdale and Chapman, 1976). In essence, seawater was mixed with arsenious acid to reduce all iodine to iodide. Wellow ceric ammonium sulphate was then added, whence the reaction between the cerium and the arsenious acid occurs at a rate dependant upon the iodide concentration. The extent of decolorisation after a fixed time was used to monitor the rate of the reaction.

Iodate was determined photometrically as the tri-iodium ion using a Technicon Auto-analyser II system (Truesdale, 1978).

The precision of both analytical methods is high, with a coefficient of variation of less than 1% being reported during analysis (Truesdale, 1995).

# Suspended Particulate Material

## Parameter Code Definitions

TSEDGVP3 Total SPM (gravimetry)  
Gravimetric analysis (GF/C filtered)  
Milligrams per litre

## Originator Code Definitions

**Charles Darwin cruises CD91B and CD93B and Challenger cruises CH121B, CH123B, CH125B, CH126B and CH128A**

21 Dr. Sarah Jones University of Wales, Bangor

## Originator Protocols

### Dr. Sarah Jones

Near-surface, mid-water and near-bed water samples were obtained from the CTD Niskin bottles. Because of the generally low concentrations at the shelf break large volumes (~10 litres) were required for each sample. The water sample was removed from the bottle by opening the bottom of the bottle into a bucket, ensuring that 'dregs' or rapidly settling particles, which might have settled below the spigot, were included in the sample. The sample was then poured into a clean container to await filtration.

Samples were filtered, noting the volume filtered, by gentle vacuum through pre-weighed 47 mm diameter Whatman GF/C glass microfibre filters with 1.2  $\mu\text{m}$  retention capabilities. When the whole sample had passed through the filter, ~150 ml of distilled water was filtered through in order to dissolve and rinse through any salt crystals. Every tenth sample (approx.) had an additional GF/C filter inserted beneath (i.e. two filters were used, one on top of the other) thereby acting as a blank. As much moisture as possible was removed from the filter before switching off the vacuum. The used filters were frozen until their return to the laboratory.

In the laboratory, the GF/Cs were oven dried overnight at 60°C, then brought to room temperature under ambient humidity before re-weighing. The weight of material on the filter was determined using the pre and post filtration filter weights

(having first subtracted the mean blank weight) and then converted into SPM mass concentrations using the filtered sample volumes.

# Hydrography

## Parameter Code Definitions

ATTNZR01	Red light attenuation (unspecified beam) 661nm unspecified path length transmissometer per metre
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
TEMPRTNX	RT temperature Reversing thermometer Degrees centigrade
TEMPPR01	Sea temperature (unspecified) Unspecified temperature probe Degrees centigrade
TEMPST01	Sea temperature (CTD/STD) CTD or STD measurement Degrees centigrade
TOKGPR01	$\mu\text{M}$ to $\mu\text{moles/kg}$ conversion (CTD) CTD measurement Dimensionless



The attenuation data were obtained using a SeaTech 25cm path length red light (661 nm) transmissometer fitted to the CTD cage. 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.

Further details of CTD data processing and quality control procedures may be found in the CTD data documentation.

### **Mr. Tony Walne**

The SAHFOS Continuous Plankton Recorder was towed through the SES area of interest on a number of occasions by ships of opportunity. The instrument was fitted with a self-logging Chelsea Instruments Aquapak, including a thermistor and a conductivity cell.

No water bottle or reversing thermometer data were available due to the nature of the instrument deployment. Consequently, the logged voltages were converted into temperature and salinity using the manufacturer's calibrations.

The instrument sampled every 15 or 20 minutes. These data were reduced to 1 mile averages by the data originator. Times and positions assigned correspond to the mid-point of the tow.

### **Research Vessel Services**

Temperature measurements were made using SIS digital reversing thermometers. Two or three instruments were mounted together in a reversing cage to provide duplicate data and an indication of occasions when the cage failed to reverse cleanly. Each thermometer was periodically calibrated at the RVS laboratory facility and a correction, in the form of a third order polynomial, determined. These corrections were 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) had 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 Autosol 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.

## **Comments on Data Quality**

The CTD temperature and salinity data are believed to be of good quality. The target accuracy for temperature and salinity was 0.02 but this has been significantly exceeded and the vast majority of the data are believed to be within 0.005.

Quality of attenuation data is variable due to a malfunctioning instrument being used on several cruise legs. Significant efforts have been made to enhance the data through empirical recalibration. The detailed procedures used and the degree of success achieved are documented in detail in the CTD data documentation.

# Optics

## Parameter Code Definitions

- IRRDP01 Downwelling 2-pi PAR scalar irradiance  
Hemispherical photodiode light meter  
Micro-Einsteins per square metre per second
- IRRUP01 Upwelling 2-pi PAR scalar irradiance  
Hemispherical photodiode light meter  
Micro-Einsteins per square metre per second

## Originator Code Definitions

**Charles Darwin cruises CD91B, CD93A and CD93B and Challenger cruises CH121A, CH121B, CH121C, CH123A, CH123B, CH125A, CH125B, CH126A, CH126B, CH128A and CH128B.**

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British Oceanographic Data Centre

## Originator Protocols

### British Oceanographic Data Centre

2-pi PAR (350-700 nm) sensors fitted to the CTD instrument package measured upwelling and downwelling scalar irradiance. Jim Aiken of the Plymouth Marine Laboratory designed and built the instruments. The data were logged as voltages and calibrated into scientific units at BODC. Details of the calibrations applied may be found in the CTD data documentation. An empirical conversion factor to convert the data into units of Watts per square metre has been determined for these instruments. Divide the values by 3.75 to implement the conversion.

The irradiance values at the bottle firing depths were determined from **downcast** data. This ensures an internally consistent data set across all cruises regardless of whether or not the upcast data were made available. The data were log transformed prior to interpolation to eliminate errors resulting from interpolation of exponential profiles. It must be noted that no geometrical correction was included in the algorithm to allow for the fact that the downwelling sensor was

located some 2m above the upwelling sensor. Consequently, upwelling irradiance values are slightly underestimated.

# Radioactive Isotopes

## Parameter Code Definitions

CS37GSD5	Dissolved caesium-137 Gamma-ray spectroscopy (0.2 µm filtered) Bequerels per litre
ET34SPD9	Dissolved thorium-234 activity standard error Alpha and beta liquid scintillation spectroscopy (0.2 µm filtered) Millibecquerels per litre
ET34SPP9	Particulate thorium-234 activity standard error Alpha and beta liquid scintillation spectroscopy (0.2 µm filtered) Millibecquerels per litre
PB10IGD2	Dissolved lead-210 Isotope spiking and ingrowth (0.4/0.45 µm pore filtered) Microbecquerels per litre
PB10IGP2	Particulate lead-210 Isotope spiking and ingrowth (0.4/0.45 µm pore filtered) Microbecquerels per litre
PO10IGD2	Dissolved polonium-210 Isotope spiking and ingrowth (0.4/0.45 µm pore filtered) Microbecquerels per litre
PO10IGP2	Particulate polonium-210 Isotope spiking and ingrowth (0.4/0.45 µm pore filtered) Microbecquerels per litre
SE37GSD5	Dissolved caesium-137 standard error Gamma-ray spectroscopy (0.2 micron filtered) Bequerels per litre
T234SPD9	Dissolved thorium-234 activity Alpha and beta liquid scintillation spectroscopy (0.2 µm filtered) Millibecquerels per litre
T234SPP9	Particulate thorium-234 activity Alpha and beta liquid scintillation spectroscopy (0.2 µm filtered) Millibecquerels per litre

## Originator Code Definitions

### Charles Darwin cruises CD92A and CD92B and Challenger cruises CH120 and CH128B

78 Dr. A. MacKenzie Scottish Universities Reactor Centre

### Charles Darwin cruise CD93B and Challenger cruises CH123B and CH128B

40 Dr. Graham Shimmield CCMS Dunstaffnage Research Laboratory

## Originator Protocols

### Dr. Graham Shimmield

The samples were collected using 10 litre Niskin bottles attached to the CTD rosette, using two bottles per depth. Surface samples were obtained from the surface non-toxic seawater supply.

Onboard, the twenty litre samples were filtered through 142mm diameter, 0.45 µm Asypor membrane filters to separate the particulate and dissolved phases. The filtrate was acidified with concentrated HCl to a pH of about 2.  $^{208}\text{Po}$  tracer, with activity of 2 dpm (disintegrations per minute), and stable lead were added.

After allowing around 24 hours for equilibration, the dissolved material (<0.45µm) was precipitated via APDC and cobalt (II) nitrate and collected on a 142mm diameter, 3µm Asypor membrane filter.

In the laboratory these filters were digested in a mixture of nitric, hydrofluoric, and perchloric acids and the  $^{210}\text{Po}$  and  $^{208}\text{Po}$  tracer were auto-deposited onto silver discs. The discs were counted using alpha spectrometry and silicon surface barrier detectors (EG &G Ortec 576s).

Silver scraps were added to remove any final traces of Po and the sample solutions were stored for a year to allow new  $^{210}\text{Po}$  to grow into equilibrium with the  $^{210}\text{Pb}$  in solution. Chemical yield was determined by measurement of stable lead. These measurements allowed the calculation of initial  $^{210}\text{Pb}$ .

Both  $^{210}\text{Po}$  and  $^{210}\text{Pb}$  were decay corrected back to the time of sampling.

The data were supplied to BODC in units of dpm/100 litres. These were converted to microbecquerels per litre by multiplying by 166.6667.

## Dr. A. MacKenzie

25-litre water samples were taken from either the ship's non-toxic seawater supply or water bottles deployed on the hydrographic wire.

Each  $^{137}\text{Cs}$  sample was acidified to pH 1.5 by the addition of 12M HCl and filtered through 0.22 $\mu\text{m}$  filter paper. The filtered sample was then passed through a 5cm by 1cm column of KCFC (potassium hexacyanocobalt(11)-ferrate(11)) inorganic ion exchanger contained in disposable plastic tubing. Thereafter, the columns were dried, sealed and analysed by gamma spectroscopy analysis using a HPGe gamma photon detector. Combined extraction and detection efficiency was determined using seawater spiked with a known activity of  $^{137}\text{Cs}$ .

Further details of the method are given in Boni (1966) and MacKenzie et al. (1979).

$^{234}\text{Th}$  activities were measured in the aqueous and solid phase of 25-litre samples. Each sample was filtered through a 0.2 $\mu\text{m}$  filter immediately after collection (noting the date and time) and the filtered sample acidified to pH 2 with hydrochloric acid to maintain thorium solubility. At this point a  $^{230}\text{Th}$  spike (50 dpm total activity) was added and the samples were thoroughly mixed.

500 mg of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and 50 mg  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  were added as carriers after which Th and U were scavenged from solution by adjusting the pH to 9 to precipitate a mixed Fe, Al hydroxide. Following stirring for 20 minutes, the precipitate was separated by filtering through a 0.2 $\mu\text{m}$  filter. The filter paper was washed with 9 M HCl to redissolve the precipitate and the solution passed through a 1 by 6 cm column of Bio Rad AG1 X8 (100-200 mesh) chloride form resin. This separated the Fe and U from the Th and the time and date of this separation were noted.

The eluate containing Th and Al was then reduced in volume to 20-30 ml and the pH raised to 9 by the addition of  $\text{NH}_4\text{OH}$  to precipitate Al and Th. The solution was spun in the centrifuge for a maximum of two minutes at 3000 rpm and decanted. The precipitate was then washed, dissolved in  $\text{HNO}_3$  and the process repeated to remove all traces of  $\text{Cl}^-$ . Finally the precipitate was dissolved in 8M  $\text{HNO}_3$  and the solution passed through a 1X6 cm column of Bio Rad AG1 X8 (100-200 mesh) (preconditioned to nitrate form) resin to retain the Th.

Following two 20 ml washes with 8 M  $\text{HNO}_3$ , Th was eluted using 9 M HCl. The solution was heated to dryness in a 7 ml scintillation vial and a fixed volume (0.5 ml) of dilute HCl (0.1 M) was added. 5 g of Ultima Gold AB scintillation cocktail was added and the  $^{234}\text{Th}$  and  $^{230}\text{Th}$  activities analysed using a Packard 2550TR/AB liquid scintillation counter.

For the analysis of the particulate material,  $^{230}\text{Th}$  spike was added to the moist filter papers which were then digested in acid. 50 mg  $\text{Al}(\text{NO})_3 \cdot 9\text{H}_2\text{O}$  was added to the resultant 9 M solution and the above procedure followed for thorium analysis.

# Dissolved 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) Nanomaoles per litre
CDXXFXD2	Dissolved cadmium Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomaoles per litre
CUXXFXD2	Dissolved copper Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomaoles per litre
MNXXFXD2	Dissolved manganese Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomaoles per litre
NIXXFXD2	Dissolved nickel Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomaoles per litre
PBXXFXD2	Dissolved lead Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomaoles per litre
ZNXXFXD2	Dissolved zinc Freon extract/atomic absorption (0.4/0.45 µm pore filtered) Nanomaoles per litre

## **Originator Code Definitions**

### **Charles Darwin cruise CD93B and Challenger cruise CH123B**

8 Dr. Peter Statham Southampton Oceanography Centre

## **Originator Protocols**

### **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 (lever-action Niskin bottles) 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<sub>3</sub> per litre of seawater (except samples for Al analysis) and stored in acid-cleaned (Morley et al. 1988) low-density polythene bottles.

Analysis was undertaken using the specialised clean facilities in the Southampton Oceanography Centre. Dissolved metals (Cd, Cu, Mn, Ni, Pb and Zn) were extracted and pre-concentrated following the dithiocarbamate complexation-freon extraction method of Danielsson et al. (1982), as modified by Statham (1985) and Tappin (1988). They 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. A bulk-filtered acidified seawater sample was used for batch-to-batch quality control. The results of these analyses (with very few exceptions) were satisfactory and indicate that the data are of high quality.

For aluminium, an aliquot of water was either vacuum filtered through a 0.4 micron Nuclepore membrane or analysed unfiltered. The method of Hydes and Liss (1976) was used 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.

# Particulate Chemistry

## Parameter code definitions

ALXXXFP2	Particulate aluminium Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
BAXXXFP2	Particulate barium Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
CAXXXFP2	Particulate calcium Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
CLXXXFP2	Particulate chlorine Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
FEXXXFP2	Particulate total iron Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
KXXXXFP2	Particulate potassium Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
MGXXXFP2	Particulate magnesium Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
MNXXXFP2	Particulate total manganese Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
PXXXXFP2	Particulate phosphorus Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre

SIXXXFP2	Particulate silicon Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
SXXXXFP2	Particulate sulphur Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre
TIXXXFP2	Particulate titanium Thin film X-ray fluorescence (0.4/0.45µm pore filtered) Nanomoles per litre

## **Originator Code Definitions**

### **Challenger cruises CH123B and CH128B**

40 Dr. Graham Shimmiel CCMS Dunstaffnage Research Laboratory

## **Originator Protocols**

### **Dr. Graham Shimmiel**

Water samples were collected using 10 litre Niskin bottles attached to the CTD rosette and the suspended particulate material collected was analysed for major elements by thin-film X-ray spectrometry.

Between 1 and 10 litres of water were filtered through 37mm diameter, 0.4 µm pre-weighed Nuclepore polycarbonate membrane filters. These were rinsed with deionised water which had a pH adjusted to ~8. The filters were stored in plastic petri dishes, allowed to dry and then re-weighed.

These particulate samples were analysed for major elements (Si, Al, Ti, Mg, Ca, K, Fe, Mn, P, S, and Ba) using a Phillips PW 1450 sequential automatic X-ray spectrometer using protocols and calibrations developed at the University of Edinburgh, Department of Geology & Geophysics.

The data were supplied to BODC in units of µg/l and were converted to the standard database units of nM by multiplying by 1000 and dividing by the atomic weight. The atomic weights used were as follows:

Al	26.98	Cl	35.45
Si	28.09	Fe	55.85
Mg	24.31	Ca	40.08
K	39.10	Ti	47.90
Mn	54.94	P	30.97
S	32.06	Ba	137.33

# Phytoplankton Counts

## Parameter Code Definitions

P030M00Z	Chaetoceros spp. Optical microscopy Number per millilitre
P073M00Z	Navicula spp. Optical microscopy Number per millilitre
P093M00Z	Rhizosolenia spp. Optical microscopy Number per millilitre
P110M00Z	Thalassionema spp. Optical microscopy Number per millilitre
P111M00Z	Thalassiosira spp. Optical microscopy Number per millilitre
P197M00Z	Pseudo-nitzschia spp. Optical microscopy Number per millilitre
P200M00Z	Dinoflagellates Optical microscopy Number per millilitre
P213M01Z	Ceratium spp. Optical microscopy Number per millilitre
P400M00G	Flagellates <5 µm Optical microscopy Number per millilitre
P400M00H	Flagellates >5 µm Optical microscopy Number per millilitre

## **Originator Code Definitions**

**Charles Darwin cruise CD93B and Challenger cruises CH121B, CH126B and CH128A**

34 Dr. Graham Savidge Queen's University, Belfast

## **Originator Protocols**

### **Dr. Graham Savidge**

Water samples were collected using Niskin bottles deployed on the CTD rosette and the phytoplankton preserved using Lugol's iodine solution. Back in the laboratory, the samples were sedimented onto glass slides and the dominant phytoplankton groups were counted. The data are based upon counts along a single transect.

Please note that this exercise was undertaken to identify the broad structure of the phytoplankton community. Identification was aimed at the group or genus level and only the dominant organisms were counted. It should not be considered as a detailed taxonomic study of the phytoplankton community or as a complete inventory of the organisms present.

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