4. ANALYTICAL PROCEDURES

Inorganic nutrient (NH_4, NO_2, NO_3, PO_4, $Si(OH)_4$) concentrations were determined by standard wet chemical methods (Treguer and LeCorre 1975) implemented on a segmented flow analyser (Ryle et al. 1981). Frozen samples were thawed in a microwave oven immediately prior to analysis. While this method results in lower blanks and variability for inorganic nitrogen and phosphorus species, it appears that silicate in the samples does not uniformly revert to a form detectable by the SFA chemistry within the short time between thawing and analysis. Accordingly, variability in the silicate values should be viewed with some caution. Dissolved organic nitrogen (DON) and dissolved organic phosphorus (DOP) concentrations were calculated by difference after oxidation (>= 7 hrs) of the organic matter in the water samples with UV radiation (Armstrong et al. 1966; Walsh 1989). Irradiated water samples were re-frozen until analysis, which results in negligible losses of inorganic nitrogen and phosphorus (Nowicki 1986). Total nitrogen was calculated from the sum of NO_3 and NH_4 in the irradiated samples (Walsh 1989).

Particulate nitrogen was determined by high-temperature combustion of the organic matter collected on glass-fibre filters using an ANTEK Nitrogen Analyser. The sample was ramp-heated (150°C min^{-1}) to 650°C in the primary combustion oven, with the combustion gases being passed through an oxygenated secondary oven (1050°C). The analyser was standardised with AR grade EDTA weighed out on an electronic microbalance. PON samples were lyophilised prior to analysis and stored in a dessicator. Procedure ('wet filter') blanks were analysed to correct for dissolved organic and inorganic nitrogen blotted into the filters plus systematic contamination introduced during storage and processing. Several drops of filtered seawater were blotted into clean, combusted filters, which were then sucked dry, stored and processed in parallel with sample filters. This correction was on the order of 0.25 µg nitrogen per filter. Freshly combusted glass-fibre filters were not measurably contaminated with nitrogen at the instrument attentuation settings used. Sediment nitrogen was determined by weighing ground (agate pestle) sediment (ca. 100 mg) into pre-combusted aluminium sample boats, which were then processed in a manner similar to filters.

Particulate phosphorus was determined by colorimetric means (Strickland and Parsons 1972) following acid-persulfate digestion (Menzel and Corwin 1965) of the organic matter in the samples (based upon suggestions from Smith et al. 1981). Filters were placed in acid-washed scintillation vials with 5 ml of five percent (w/v) potassium persulfate. The persulfate was refluxed to dryness by heating the vials in an aluminium heating block, using an acid-washed marble for a stopper. Following the digestion, the filter and residue were resuspended in 5 ml of deionized water and the filter pulverised to dissolve all soluble material. The solution was cleared by centrifugation and the inorganic phosphorus determined colormetrically in aliquots of the supernatant. Organic and inorganic phosphorus standards were run in parallel with each batch of filters. For sediments, weighted subsamples of agate-ground sediment were acidified with 25% (v/v) HCl in acid-cleaned scintillation vials and refluxed to dryness to remove all carbonates and extraneous volatile acid. The residue was then redissolved in five percent persulfate and re-refluxed again to dryness. The residue was redissolved in deionized water, cleared by centrifugation and the phosphorus determined colormetrically as above.

Chlorophyll on filters was determined fluorometrically after grinding in 90% (v/v) acetone (Strickland and Parsons 1972). Most samples were analysed at sea within days of collection and all samples were analysed within three weeks of collection.

Suspended solids concentrations were determined gravimetrically from the difference between loaded and unloaded filter weights after the filters were dried overnight at 80°C.

Filtered zooplankton biomass samples were dried for several days at 60°C. The zooplankton and netting were weighed, then zooplankton was scraped off and the filter re-weighed.

Grain size analysis of sediment samples was carried out following Folk (1974). Discriminations were made only to gravel (> 2 mm), sand (2-0.063 mm) and mud (< 0.063 mm) size fractions. Sediment samples were first treated overnight with hydrogen peroxide to remove organic matter. Percent gravel, sand and mud were determined gravimetrically following wet and dry sieving. Clay composition (expressed as a percent of mud) was determined by pipette analysis. Subsamples of the gravel size fraction were hand sorted to fragments derived from molluscs, echinoderms and segments of the calcareous green alga, *Halimeda*. A weighted 20 ml subsample of the mud fraction was acidified to dissolve all carbonate materials. The residue was collected on a pre-weighed filter, rinsed with deionized water, re-dried and re-weighed to estimate the contribution of $CaCO_3$ to the mud fraction.