POMME 1 - 2 - 3

TOC - Rosette

RESPONSABLES

Carbone Organique Total (TOC)

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METHODE ANALYSE & PRELEVEMENT

Carbone Organique Total (TOC) TOC sampling and analyses during Pomme cruises

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Degré de validité des données présentées (Pomme 0 et Pomme 1): Définitif

Discrete seawater samples were collected by using a Seabird SBE 9 rosette sampler equipped with Niskin bottles from the RV Thalassa and Atalante in Atlantic Ocean at a series of stations. At the beginning of the cruise, Niskin bottles were cleaned with HCl 0.2% and distilled water. Plastic o-rings were replaced by Viton material whereas original plastic ribbons were replaced by silicone ribbons to avoid organic contamination. Samples were not filtered and were drawn as soon as possible after the rosette sampler was on the deck of the ship (before any other sampling). The samples were collected in duplicate or

triplicates in precombusted (450° C, at least 6 hours) glass ampoules (Wheaton®). All ampoules were poisoned by addition of H3PO4 (final pH ~ 2) immediately after filling of ampoules, flame-sealed, and stored in the dark for later analyses at the shore laboratory within 5 months.

HTCO analysis

The Shimadzu instrument used in this study was the commercially available Model TOC-5000 Total Carbon Analyzer with quartz combustion column filled with 1.2% Pt on silica pillows with approximate diameter of 2 mm. Several aspects of our modified unit have been already described (see relevant publications below). Briefly, the furnace temperature was maintained at 680oC and the effluents passed through a mercury trap (gold wire) to remove mercury. A magnesium perchlorate water trap has been added to the system located before the halogen scrubber, and an in-line membrane filter and the non-dispersive infrared CO2 detector. Prior to analysis, sub samples were acidified with 10 µl of 85% H3PO4 to a pH ~2 and sparged for 10 min with CO2-free pure air at a gas flow rate of 40 ml min-1 to remove inorganic carbon as CO2. TOC contamination from the preservation reagent and from H3PO4 was below the detection limit. A hundred μ l injections were repeated 3-4 times for each sample, the analytical precision of the procedure being within 3%, on average. Some variability in values from two different vials gives rise to a lower overall precision (10%).

To bring the blank down, the catalyst was pre-treated by washing in 1% HCl and gently rinsed with DW, and dried in a furnace at a temperature of about 450oC for 10-15 min. Prior to analyses of standards and samples, the catalyst bed was 'conditioned' (during 1-3 days) by injecting 100 μ l of acidified and sparged water from a high-quality water purifier, a Millipore Milli-Q Plus® System, until the lowest stable integrated area was obtained. After two seawater sample injections the column was flushed by three injections of 100 μ l of Milli-Q water. The catalyst was regenerated by using the TC catalyst function of the instrument once a week. Most of the time and after every two weeks of 25 daily samples injections, top of the catalyst (2 cm) was replaced was fresh material. The catalyst being completely renewed every three weeks.

Calibration of the instrument

Standardisation was carried out every day using potassium hydrogen phthalate (Kanto Chemical Company, Inc.) dissolved in Milli-Q water prepared just before sample analyses. The instrument response factor, measured as the slope of the standard addition to DW (r2>0.999), remained relatively constant and reproducible over the time of analysis.

The accuracy and the system blank of our instrument were determined by analysis of the Deep Sargasso Sea Water (DSR) reference and low carbon water (LCW) from ampoules provided by Denis Hansell. The average DOC concentration in the DSR reference and in the LCW were 43.1 \pm 0.9 μM C, n = 45 and 1.1 \pm 1 μM C, n = 43, respectively. Carbon level of LCW ampoules was similar and often higher than DW produced from our laboratory unit. TOC

concentration in samples was calculated by averaging all replicate injections of samples, subtracting the average DW value as the total blank and dividing by the slope of the standardisation curve.

Recent Relevant publications (DOC, TOC)

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DESCRIPTIF des FICHIERS

Fichiers en date du 09 -04- 2004