Franklin Cruise FR 9308

Data Documentation

JGOFS Western Equatorial Pacific Process Study

[1] General

Parameters	Concentrations of total dissolvable cadmium, copper, iron, manganese and nickel.
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List of Units	nmol kg^{-1}

[2] Sampling

Gear	Samples were collected from Helmond/Byrne (6 litre) polycarbonate bottles made at CMR. The H/B bottles had external silicone rubber and nylon (Kevlar) closures and Teflon (PTFE) taps. The only metal components were external to the bottles and made from titanium.
Locations	5°N 155°E, 0° 155°E, 5°S 155°E at 10 depths from the surface to 2000 m (5°N), 1500 m (0°) and 1500 m (5°S).
Sampling Procedure	The H/B bottles were cleaned with 0.1% Triton X-100 and with 5% HCl overnight. They were then rinsed with Milli Q water and stored in polyethylene bags until they were deployed on the CTD. All Teflon fittings were acid cleaned. Nalgene LDPE sample bottles (1 L) were soaked in 2.5% Extran for a week, rinsed with Milli Q water and then acid-cleaned for 20 hours with boiling quartz-distilled 6 M HCl in an Ausdampf cleaning apparatus and rinsed again with Milli Q water. The bottles were stored in polyethylene bags / tote boxes. Gloves were used for all sampling procedures. The CTD was equilibrated at depth for about 1 minute then lowered at 10 m min^{-1} and fired after 30 sec so that samples were collected approximately 5 m below the initial depth. On recovery of the CTD, external silicone rubber bands were fitted to the H/B bottles to prevent leaking. The bottles were then removed from the CTD rosette and mounted on the outside of a portable clean laboratory. Luerlock filters (0.2 μm) were fitted to the bleed valves and Teflon (PFA) lines and taps (PTFE) were connected to the bottles. The lines were passed through ports into a laminar flow clean cabinet

inside the clean laboratory where the samples were collected by gravity feed.

[3] Analysis

Instrument	Perkin Elmer Zeeman 5000
Method	GFAAS after extraction of DDDC/APDC complexes into Freon TF and back extraction into HNO_3 (see details below).
Precision	Estimated to be ≈ 10%
Comments	The cleaning of the sample bottles (Ausdampf apparatus) was done in a general laboratory. All other cleaning of reagents and equipment, analyses and sample manipulations were performed in a Class 100 clean laboratory using standard ultra-clean procedures. All sample bottles, reagent bottles, separating funnels etc were made from Teflon or LDPE.

[4] Results

Quality of data	Blanks were measured at all stages of the procedure and the detection limits (3 × standard deviation of blanks) were Cd (0.004 nmol kg^{-1}), Cu (0.16 nmol kg^{-1}), Fe (0.31 nmol kg^{-1}), Mn (0.15 nmol kg^{-1}) and Ni (0.42 nmol kg^{-1}). Replicate analyses were performed for all 'anomalous' results and the data were verified by regular analyses of NASS certified reference materials.

[5] Brief description of analytical method

On returning to Hobart, the samples were acidified with 1 ml of Seastar HCl and stored for at least 3 months before being analysed. A subsample (50 ml) was adjusted to pH 4-5 by the addition of NH_4OH followed by a sodium acetate buffer. Trace metals in the subsample were then complexed by adding 0.3 ml of a solution containing 1% (w/v) each of diethylammonium diethyldithiocarbamate (DDDC) and ammonium pyrrolidinedithiocarbamate (APDC), extracted into 10 ml of quartz-distilled Freon TF (1,1,2 trichloro- 1,2,2 trifluoroethane) and back extracted with 25 µl of concentrated Seastar HNO_3 followed by 1 ml of Milli Q water. The dilute HNO_3 extract was then analysed for Cd, Cu, Fe and Ni by GFAAS. For Mn, a separate subsample of seawater was adjusted to pH 7.5-8.5 with NH_4OH before adding 3.0 ml of DDDC/APDC reagent and extracting into Freon. The Freon was then back extracted twice with Seastar HNO_3 and Milli Q water. The NH_4OH (≈ 6 M) was prepared by diffusion of NH_3 from concentrated NH_4OH into Milli Q water in a closed container. The mixed dithiocarbamate reagent was cleaned by repeated extraction with Freon. The citrate buffer was cleaned by adding a small amount of the DDDC/APDC reagent and extracting repeatedly with Freon. All reagents were cleaned until trace metal concentrations in the (DDDC/APDC)/Freon/HNO_3 extracts were negligible.

References

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[6] Comments

None