Benthic Organic Chemistry

On one of the Humber Plume process cruises, Challenger 69, a worker from Rikjwatersaat collected surface sediment samples for organic chemical analysis.

Surface sediment samples were collected using a Day grab or the SMBA (now DML) box corer. If a relatively undisturbed and muddy top layer existed (0-2 cm), it was subsampled using a PTFE spatula; otherwise, the top 5 to 10 cm were sampled. The subsamples were transferred to pentane-washed polyethylene bottles. All samples were sieved on board, using a specially designed sieving apparatus with 63µm nylon mesh (Klamer et al., 1990). The collected fine fractions were kept frozen until further treatment.

After freeze-drying, chlorobiphenols (CBs) 29 and 155 and benzo(b)chrysene were added to this sediment, for recovery measurements. The sediments were Soxhlet-extracted with hexane-acetone (3:1). The crude extracts were cleaned up and the CB and PAH fractions were fractionated by adsorption chromatography using aluminium oxide and silica gel. CBs were determined with capillary gas-chromatography, using two columns with different polarity, and ECD detection at both columns. A laboratory standard of a mixture of CB congeners was used for identification and quantification, using CB 143 as internal standard. CBs determined were: 18, 26, 28, 31, 44, 52, 101, 118, 138, 153, 170 and 180. Detection limits were about 0.1 µg/kg, with an analytical error of about 10% and a recovery of 90-100%.

Polycyclic aromatic hydrocarbon (PAH) compounds were determined using high-performance liquid chromatography with fluorescence detection, using 2-methylchrysene as internal standard. PAH compounds determined were: fluoranthene (FLAN), phenantrene (PHEN), pyrene (PYRE), benzo(a)anthracene (BAA), chrysene (CHRY), benzo(e)- and benzo(a)pyrene (BEP and BAP), benzo(b)- and benzo(k)fluoranthene (BBF and BKF), benzo(g,h,i)perylene (BGP) indeno(1,2,3-c,d)pyrene (ICDP) and dibenzo(ah)anthracene (DAAH). Detection limits were about 5 µg/kg, with an analytical error of about 10% and a recovery of 80-95%.

Biogeochemical markers (e.g. ^{13}C) were analysed in the organic carbon and carbonate fractions of the particulate samples, measuring the $^{13}C/^{12}C$ ratio of the combustion product (CO_2) with mass spectrometry (Laane et al., 1990).

References

Klamer, J.C., W.J.M. Hegeman & F. Smedes (1990). Comparison of grain size correction procedures for organic micropollutants and heavy metals in marine sediments. Hydrobiologia 26(1), 83-87.

Laane, R.W.P.M., E. Turkstra and W.G. Mook (1990). Stable carbon isotope composition of pelagic and benthic organic matter in the North Sea and adjacent estuaries. Ittekkot, V. et al. (Eds.), Springer Verlag, Berlin, pp.214-224, ISBN: 3-540-50145-2.