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FIFTH REPORT OF THE MARINE CHEMISTRY WORKING GROUP

Copenhagen, 7-10 February 1983

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TABLE OF CONTENTS

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1. OPENING OF MEETING AND ADOPTION OF AGENDA

1.1 The Chairman, Dr J M Bewers, opened the meeting at 9.30 hrs on 7 February 1983 and welcomed the participants. The draft agenda was considered and adopted as proposed. The agenda is attached as Annex 1. The list of participants is contained in Annex 2. The ICES Environment Officer served as Rapporteur.

2. REPORT OF THE 70th STATUTORY MEETING

2.1 The Working Group took note of a list of relevant Council Resolutions which had been adopted at the 1982 Statutory Meeting. All recommendations from the 1982 Working Group meeting had been accepted by Council.

2.2 The Chairman reported that Council had noted the lack of an adequate number of experts on CO_2 cycling in the oceans on the Working Group and had indicated that more expertise on this subject would be desirable. He further reported that the guidelines for the conduct of regional assessments of the health of the marine environment, as set forth in C.M.1982/E:22, had been accepted by the relevant Committees and a Council Resolution had been passed encouraging the conduct of such assessments (C.Res.1982/4:10). The Council had also passed a resolution (C.Res.1982/4:7) encouraging ICES member countries to commence the assessment of gross riverine inputs of trace metals and organochlorines, using the guidelines agreed by MCWG at its previous meeting and later adopted by ACMP. Finally, the Chairman pointed out that the prospectus for the Symposium on Contaminant Fluxes through the Coastal Zone (Nantes, May 1984) had been developed and he encouraged members to prepare papers for this Symposium.

3. REPORTS ON OTHER RELATED ACTIVITIES

3.1 Working Group on Marine Pollution Baseline and Monitoring Studies in the North Atlantic

3.1.1 The relevant results from the meeting of the Working Group on Marine Pollution Baseline and Monitoring Studies in the North Atlantic (WGMPNA) the previous week were reviewed. It was noted that the WGMPNA had reviewed the status of intercalibrations on the determination of contaminants in Annex 5 to the 1982 MCWG report (C.M.1982/C:1) and had supported this type of work and its further development. The WGMPNA had reaffirmed the usefulness of carrying out a baseline survey of trace metal concentrations in coastal waters and shelf seas, which has now been approved in principle by Council (C.Res.1982/4:8), and requested MCWG to develop guidelines for sampling, pretreatment and analysis of sea water for trace metals, based on the outcome of the Fifth Round Intercalibration on Trace Metals in Sea Water (5/TM/SW). It was noted that these guidelines will be needed to obtain valid information on the concentrations of dissolved trace metals in sea water during the baseline survey and would also be useful to the Joint Monitoring Group (JMG) of the Oslo and Paris Commissions in connection with the Joint Monitoring Programme. In connection with further planning for this baseline survey, the WGMPNA also requested the MCWG to consider and provide advice on whether the fresh water end member

of rivers should also be sampled in the survey of trace metal concentrations in sea water to obtain more complete information.

3.1.2 It was further reported that, in the consideration of the methods to be used for trend monitoring of the concentrations of contaminants using marine organisms, the WGMPNA had discussed the possibility of using fish bone tissue to study trends for certain trace metals. The WGMPNA requested the MCWG to determine whether there may be any matrix problems associated with the analysis of bone tissue for trace metals.

3.1.3 The WGMPNA had reviewed a paper on the toxicity of toxaphene in the marine environment and requested the MCWG to begin work on solving the analytical problems associated with the determination of toxaphene in marine samples.

3.1.4 Finally, the WGMPNA had considered the ICES Interim Reporting Format for Contaminants in Fish and Shellfish and had asked the MCWG to assist on two issues: (1) a list of methods for determination of trace metals and organochlorines in biota and for determinations of fat weight, for use in the reporting format itself, and (2) information on the possibility of developing a realistic set of criteria for evaluating a laboratory's past performance on the basis of its results in an intercalibration exercise.

3.1.5 Having taken note of these questions and other relevant items from the WGMPNA meeting, the MCWG agreed to give more detailed consideration to each request under the appropriate agenda item.

3.2 Working Group on Marine Sediments in Relation to Pollution

3.2.1 The 1982 report of the Working Group on Marine Sediments in Relation to Pollution (WGMS) was briefly considered and it was noted that the MCWG suggestion of the previous year, that the WGMS utilize radionuclide techniques when studying the geochronology of marine sediments, had been acted upon by the WGMS. The MCWG noted, however, the absence of any feedback concerning its assessment of the status of intercalibration exercises on contaminants in sediments, as contained in Annex 5 to the 1982 MCWG Report (C.M.1982/C:1).

3.3 ICES/SCOR Working Group on the Study of the Pollution of the Baltic

3.3.1 It was noted that a major activity of this Working Group is the sponsorship of a Workshop on Patchiness Experiments in the Baltic Sea (Convener, Dr B I Dybern), which will be held in Tallinn, USSR on 21-23 March 1983. The results of studies on the spatial and temporal scales of patchiness or heterogeneity of physical, chemical and biological parameters in the Baltic Sea will be discussed and the possibility of international cooperation on a joint study will be considered.

3.3.2 Other relevant activities of the ICES/SCOR Working Group include the publication of the final overview of results of the Baltic Open Sea Experiment (BOSEX) and the development of models for biogeochemical cycles of substances in the Baltic Sea, starting with nitrogen and phosphorus.

3.4 Joint Monitoring Group of the Oslo and Paris Commissions

3.4.1 It was noted that there had been some indication at the recent meeting of the Joint Monitoring Group (JMG) (Paris, 24-27 January 1983) that a new intercalibration exercise on mercury and cadmium in sea water may be necessary because several national delegations had felt that the concentrations of the metals contained in the samples distributed for 5/TM/SW were considerably lower than those normally encountered in estuarine and possibly also coastal waters. The MCWG agreed to consider this question as soon as the results of 5/TM/SW are available. The MCWG further noted that the second French-sponsored

intercalibration exercise on trace metals in sediments which is open to JMG laboratories is also open to non-JMG ICES laboratories. No detailed information on the objectives of this intercalibration or the types of samples to be used was available.

3.5 IOC/GIPME Activities and Plans

3.5.1 The representative of the IOC drew the Group's attention to the Summary Report of the Fourth Session of GEMSI (Group of Experts on Methods, Standards and Intercalibration) held in Curaçao in 1982. A brief account of the various ad hoc Groups was presented and it was noted that many of the members of GEMSI were also present at the MCWG, being common members of both groups.

3.5.2 It was reported that a group had been working on dissolved/dispersed petroleum hydrocarbons in seawater and that an intercalibration standard was soon to be distributed from the Bermuda Biological Station to IOC participating laboratories. An updated manual for the UV-fluorescence method was approaching completion and will be presented at the GEMSI V meeting this year.

3.5.3 The pilot project on Monitoring Background Levels of Selected Pollutants in Open-Ocean Waters was moving into a second phase and, pending funding, a core group would tackle the problems of intercomparison of extraction and filtration techniques for organochlorines in seawater, prior to a second intercalibration/training exercise. Further progress had been made in the isolation and analysis of individual PCBs and was to be reported jointly by the Institute for Marine Research, Bergen, and NIOZ, Texel at the next GEMSI meeting.

3.5.4 An ad hoc Group on the "Use of Marine Organisms in MARPOLMON" had produced a report at its last meeting and Dr Topping, the Chairman of this Group, reported on the follow-up activities subsequent to this report. He described the approach which had been adopted to send questionnaires to laboratories of the IOC WESTPAC (Western Pacific) region and to distribute the ICES intercalibration samples for trace metals and organochlorines and he briefly summarised the results of the first round intercalibration. The need for extensive training in the region had been noted and a training workshop for WESTPAC is envisaged for later this year.

3.5.5 The IOC representative informed the MCWG that an ad hoc Group on the Use of Sediments in MARPOLMON had been newly constituted with Dr D Schink, Texas, as its Chairman. They were to meet in April 1983 and were relying heavily on the recommendations of such groups as MCWG, JMG and the sister GEMSI groups, before submitting their report to GEMSI V.

3.5.6 The IOC representative further indicated that, following recommendations of a Sessional Group on the identification of gaps and fluxes and mass balance calculation of contaminants in marine systems, groups would be formed to consider river inputs of pollutants and that these groups would work closely with the recommendations of the SCOR Working Group 46 (RIOS).

3.5.7 In concluding, the IOC Representative offered 3 recent reports for distribution upon request: (a) IOC Summary Report GEMSI IV Curaçao 25-31 March 1982; (b) IOC Technical Series 22 , Report of the Bermuda (Pancal) Intercalibration Workshop 1980; (c) Manuals and Guides 11 - The determination of petroleum hydrocarbons in sediments.

3.6 Other Relevant Activities of Multilateral and International Agencies

3.6.1 Dr Ehrhardt provided information on the results of the Workshop on the Analysis of Hydrocarbons in Seawater, which was held at the Institut für Meereskunde an

der Universität, Kiel, on 23 March-3 April 1981, under the auspices of the Baltic Marine Environment Protection Commission (Helsinki Commission). The Workshop consisted of three parts: (1) the presentation of invited papers on subjects related to the contamination of sea water by petroleum hydrocarbons, (2) the collection of sea water samples and the measurement of the concentration of oil residues dissolved and/or finely dispersed in surface water by the UV fluorescence method recommended by IOC, and (3) the provision of an aliquot of an integrated sample for analysis at the participants' home laboratories. On the basis of the results of the Workshop, several recommendations were made concerning the analyses of hydrocarbons in sea water. A more detailed description of the Workshop and its results is attached as Annex 3. The full report is published as Baltic Sea Environment Proceedings No.6 (1982).

3.6.2 In the discussion of the results of this Workshop, it was noted that the measurement of oil in sea water will be obligatory in the second stage of the Baltic Monitoring Programme, which will begin in 1984.

3.6.3 Dr Kerkhoff informed the Working Group that a programme has been started under the European Economic Community to improve the analysis of PCBs in a number of substances, e.g., fish and milk. Twelve specialists from selected laboratories are participating in this programme.

4. REPORTS ON PROJECTS AND ACTIVITIES WITHIN ICES MEMBER COUNTRIES

4.1 In introducing this topic, the Chairman reminded the Group that it had been agreed the previous year that only information which had been submitted in writing could be considered under this agenda item. Only two papers had been submitted: (1) a paper by Dr Weichart on certain activities of the Deutsches Hydrographisches Institut, and (2) a paper by Dr Law on studies of the analysis of organotin compounds carried out by the MAFF Fisheries Laboratory in Burnham-on-Crouch, England.

4.2 Dr Weichart summarized the work of his Institute in the "Deep Water Project" organized by ICES. In this work, measurements of O_2 and CO_2 concentrations are used to identify newly formed deep water (originating from the Greenland Sea) and to observe its spreading on the sea floor. Together with current meter measurements, these chemical investigations can be used to estimate the fluxes of O_2 and CO_2 from the atmosphere to the deep layers of the oceans.

4.3 Dr Weichart also mentioned that his Institute is monitoring heavy metals, nutrients, oxygen, chlorinated hydrocarbons, petroleum hydrocarbons and radioactivity in sea water and sediments in the German Bight and the southwestern Baltic Sea. In addition, in the Baltic Sea the oxygen concentrations in the deeper layers and the petroleum hydrocarbon concentrations in the entire water column were determined along a number of sections.

4.4 Dr Law presented a short summary of the work undertaken by Dr Waldock at the MAFF Fisheries Laboratory in Burnham-on-Crouch on the analysis of organo-tin compounds in sea water and oyster tissue. This followed on from work carried out at ISTPM under C Alzieu on the link between abnormal growth and shell thickening in the Pacific oyster (Crassostrea gigas) and the presence of organo-tin compounds from anti-fouling paints used on pleasure boats. This has resulted in a 2-year ban being imposed in France on the use of organo-tin-based anti-fouling paints on boats of less than 25 m overall length.

4.5 The work has concentrated on estuaries in eastern England, where there are large numbers of pleasure boats in traditional oyster growing areas, although oyster growing sites elsewhere have also been sampled. Analyses of total tin and organo-tin have been carried out by flameless AAS using a graphite furnace, and the presence of tri-butyl tin (TBT), the major organo-tin used in the anti-fouling paints, has been confirmed by capillary GC-MS following Grignard

methylation of the extracts. High concentrations of tin and TBT in water occurred where there were large numbers of yachts, and the highest levels coincided with the return to the water in the spring of freshly anti-fouled boats.

4.6 Samples of Crassostrea gigas and Ostrea edulis from the same area showed similar levels of total tin, but TBT levels were 3 to 9 times higher in C. gigas. All samples of C. gigas that showed signs of abnormal shell thickening also contained TBT, and samples of normal C. gigas transferred to a river with elevated levels of TBT rapidly began to develop thickened shells.

4.7 Previous work at Burnham has suggested that high suspended loads of fine sediment may also have a role in shell thickening, and laboratory experiments are underway to investigate shell growth in C. gigas exposed to sediment and TBT both alone and together. Uptake and loss experiments are also planned for the near future.

4.8 The Chairman thanked the members who presented their information and closed the discussion on this agenda item by reminding the Working Group that for the next meeting, if any members wish to present information on activities at their laboratories or in their countries, they should prepare a short paper on the subject for distribution prior to the meeting.

5. INTERCALIBRATION ACTIVITIES AND PLANS

5.1 Fifth Round Intercalibration for Trace Metals in Sea Water (5/TM/SW)

5.1.1 The Chairman summarized the nature of the Nantes Intercalibration Exercise for Trace Metals in Sea Water (ICES 5/TM/SW)in the absence of the scientific coordinator, Dr Jan Duinker. An administrative report of this experiment had been submitted to ICES in November 1982 and a copy was made available for review by members of MCWG during the meeting. The experiment comprised five components:

1) An intercomparison of filtration procedures for the measurement of dissolved Cd, Cu, Ni, Zn, Fe and Mn in turbid coastal waters;

2) The preparation of approximately 100 replicate filtered coastal water samples for Cd, Cu, Ni, Zn, Fe and Mn analysis;

3) An intercomparison of sample collection procedures for the measurement of Hg in coastal waters;

4) The preparation of approximately 100 replicate samples of coastal sea water for Hg analysis;

5) A training component involving sampling and analytical demonstrations for the measurement of metals in sea water.

5.1.2 The results of the first component experiment are being assembled by Dr Bewers for the preparation of a paper to the 1983 Statutory Meeting. The replicate samples have been distributed to those who had requested them from ICES. The results of Cd, Cu, Ni, Zn, Mn and Fe analyses of the first group of replicate samples are being collected by Dr Berman for the preparation of a paper to the 1983 Statutory Meeting. The results of Hg analyses on the second group of replicate samples and samples collected as part of the sampling intercomparison for mercury are being received and collated by Dr D Cossa, who, in collaboration with Dr P Courau, will prepare an evaluation of these components of the experiment. Some contamination problems with regard to the mercury samples

have been detected and the possibility of repeating Components 3 and 4 of the experiment will be examined during the session by a Sub-group on Trace Metals.

5.1.3 From the preliminary results of the experiment, it appears that the levels of Cd, Cu, Ni and Zn in the coastal waters off St. Nazaire are comparable to open-ocean values. It was not possible to draw similar comparisons for mercury due to the apparent contamination of the coastal water samples collected for Hg analysis.

5.2 Organochlorines in Biological Tissue (5/OC/BT)

5.2.1 Dr Uthe, Coordinator of the Fifth Intercomparison Exercise on Organochlorines in Biological Tissue (5/OC/BT), described the progress in this exercise. He reported that the intercomparison samples, consisting of (a) a fish oil, (b) the same oil spiked with four individual biphenyl compounds, and (c) small amounts of these individual biphenyls, had been distributed to thirty-three analysts. The participants had been requested to identify and quantify the individual biphenyl compounds in the samples and estimate the total PCB concentration. Of the 25 replies of results received to date, only nine analysts had been able to analyze for individual compounds. Dr Uthe stated that the overall results of the exercise will be reported in a paper to the 1983 Statutory Meeting.

5A. FORMATION OF SUB-GROUPS ON NUTRIENTS, TRACE METALS, AND ORGANICS

5A.1 The remainder of the sub-items in Agenda Item 5 were considered in detail by three Sub-Groups which met concurrently and prepared reports for discussion by the entire Working Group. The Sub-Groups were as follows: (1) Sub-Group on Nutrients (Chairman: Dr Koroleff), (2) Sub-Group on Trace Metals (Chairman: Dr Topping), and (3) Sub-Group on Organics (Chairman: Dr Knap). The Sub-Groups were given the following terms of reference:

1) To examine progress with regard to intercalibration design and logistics and formulate plans for the completion/execution of current initiatives.

2) To assess the long-term strategy for the improvement of sampling, preservation, pre-treatment and analytical methodology among ICES laboratories taking particular account of the need for data intercomparability and quality control.

3) To determine for which analytes/marine phases methods are currently sufficiently accurate, precise and intercomparable to justify their inclusion in the 1985 ICES baseline survey programme.

4) To consider what new initiatives are required to improve methodology and/or data accuracy/precision and intercomparability among ICES laboratories.

5) To consider the revision of the previously prepared summary of intercalibration activities and plans (Annex 5, MCWG Report 1982) and determine how this may be undertaken during the next intersessional period.

6) To consider, where intercalibrations have been completed on a substance, whether there is a need for uncompromised (i.e., blind) intercomparison samples and how best this should be done.

5A.2 The Sub-Groups met during the afternoon of 7 February and reported the following day on the progress in their work to the entire Working Group.

5A.3 The Sub-Group on Nutrients reported that, as a result of their deliberations, they felt that there was no need for a new multi-lateral intercalibration exercise on the determination of nutrients in sea water, except for the case of multi-ship expeditions, when informal intercalibrations should be held. The Sub-Group felt that there were generally no problems with sampling or analysis for nutrients, but recommended that samples be analyzed immediately without undergoing preservation.

5A.4 In the discussion of this preliminary report, a question was raised concerning the value of monitoring total nitrogen concentrations in sea water, because the levels are very variable and are difficult to interpret. It was generally felt, however, that measurements of total nitrogen should continue because this information is needed to obtain a full picture and better understanding of the nitrogen cycle.

5A.5 In concluding the discussion of the preliminary report on nutrients, the Sub-Group on Nutrients was requested (a) to provide a more detailed description of previous multi-lateral intercalibration exercises and their results, (b) to comment in more detail concerning the preservation of samples for nutrient analysis, including which nutrients can be preserved and under what circumstances, (c) to explain the statement that autoanalysis must be referred back to manual methods of analysis, (d) to indicate whether nutrients should be included in the 1985 baseline survey and, if so, which nutrients and why, and (e) to provide more details concerning sampling, analysis, etc. in a format similar to that used in Annex 5 to the 1982 MCWG Report.

5A.6 The Sub-Group on Trace Metals reported that the plans for the Seventh Intercomparison Exercise on Trace Metal Analysis of Biological Tissue (7/TM/BT) had been considered and it had been decided that additional reference materials would be needed to provide the appropriate information on analytical capability in connection with the 1985 baseline survey. Details of the entire exercise had been agreed and a schedule of activity had been planned. It was noted that the design of this intercomparison exercise was specifically intended to assist participants in improving their analytical techniques for the determination of trace metals in biota.

5A.7 The Working Group thanked the Sub-Group on Trace Metals for this information on the plans for 7/TM/BT and stressed the importance of obtaining widespread participation in this intercomparison exercise in order to ensure its success. Members were encouraged to distribute information on the exercise to appropriate colleagues. The Working Group then requested the Sub-Group to consider the issues related to the determination of trace metals in sea water and, especially, whether guidelines for sampling, pretreatment, and analysis should be developed in preparation for the proposed baseline survey on trace metals in sea water.

5A.8 The Sub-Group on Organics then gave a preliminary report on its deliberations. There were a number of basic problems which still need to be resolved with regard to the determination of PCBs, which should no longer be considered in terms of total PCBs but rather individual PCB compounds. The Sub-Group reported that the problems of PCB analysis were so severe that even the best laboratories in the United States cannot achieve an interlaboratory CV of 15-20% in the analysis of one mussel homogenate, so there is little hope of attaining an interlaboratory CV of less than 30% which would be needed in connection with, e.g., a baseline survey of PCBs in biota. The Sub-Group raised the question of whether laboratories could be rated according to their performance, for possible participation in the

baseline survey on contaminants in biota and other cooperative programmes. The Sub-Group also stressed the importance of having authenticated standards of individual PCB compounds available to all laboratories who wish to improve their analysis of PCBs.

5A.9 There was considerable discussion in the Working Group of the issues raised by the Sub-Group. Concerning the question of whether laboratories should be ranked or rated on the basis of their performance in an intercomparison exercise, some members felt that intercomparison exercise results should be accepted as a measure of a laboratory's past performance and that only on the basis of the intercomparison results can that laboratory's data be interpreted as to how good or intercomparable they are. Other members felt that intercomparison exercises should rather be considered as being conducted primarily to assist laboratories in learning how to conduct better analyses and that even good laboratories should be concerned about the quality of their data. After further discussion of these questions, the Working Group agreed that it would be very difficult to attempt to make statements on laboratories' performance levels at the present time, however, as was proposed for 7/TM/BT, the next intercomparison exercise on PCBs and other organochlorine residues should include two phases: (1) the distribution of an initial set of intercalibration samples, followed by a period in which laboratories can attempt to improve their analytical techniques, and (2) the distribution of a new set of uncompromised samples to be analyzed at the same time as samples which have been taken for the baseline survey. This approach will both allow laboratories to attempt to perform better before the baseline survey as well as provide a clear method of evaluating the intercomparability of data on baseline survey samples.

5A.10 Concerning the issue of the availability of authenticated individual PCB compound samples, the Working Group agreed that this was a very important issue but also very difficult in terms of which specific compounds should be synthesized and who should carry out this work. The Sub-Group on Organics was requested to consider this and the other issues in its terms of reference and develop proposals as appropriate.

5A.11 The three Sub-Groups thereafter met concurrently on 8 February and prepared written reports summarizing their conclusions and plans for future work. These reports were then considered in detail by the full Working Group and amendments were made in accordance with the results of the discussion. The final, approved Sub-Group reports are contained in their entirety in the next three sections, as follows:

5B. Report of the Sub-Group on Nutrients

5C. Report of the Sub-Group on Trace Metals

5D. Report of the Sub-Group on Organics

5B. REPORT OF THE SUB-GROUP ON NUTRIENTS

5B.1 Intercalibrations

5B.1.1 The nutrients considered here are inorganic phosphorus and nitrogen compounds, total phosphorus and total nitrogen, silicate and urea.

5B.1.2 Nutrient intercalibrations were organised for ICES countries in 1965 and 1966. A world-wide intercalibration was organised by ICES/SCOR in 1969/70, in which 45 laboratories participated from 20 countries. For this intercalibration, sample solutions were distributed containing low, medium and high concentrations of phosphate, nitrate, nitrite and silicate. Participants were requested to indicate the general analytical method used for each nutrient and the results were evaluated, inter alia, on the basis of whether all commonly used methods produced comparable results. The overall coefficients of variation obtained were as follows: (1) phosphate: 4.2% (high concentration) and 15.6% (low concentration), (2) nitrate: 9.4% (high concentration) and 20.6% (low concentration), (3) nitrite: 4.1% (high concentration) and 12.0% (low concentration), and (4) silicate: 4.1% (medium concentration) and 7.2% (low concentration). As a result of this intercalibration exercise, it was concluded that uniform standards were of great importance in the analysis of nutrients and it was recommended (C.Res.1970/3:6) that the nutrient salt standards prepared at the Sagami Chemical Research Center should be used as the primary standard for all ICES international and multi-ship expeditions. The full report on this intercalibration has been published as Coop. Res. Rep. No.67 (1977).

5B.1.3 After these general intercalibrations, several regional initiatives have been taken in the North Sea (e.g., FLEX intercalibration) and the Baltic Sea areas. Standard reference materials for phosphate, nitrate, nitrite and silicate have become available during recent years. At present there seems to be no necessity to carry out a new multi-lateral intercalibration. However, it is strongly recommended that ad hoc intercomparisons (including exchange of samples) be conducted between ships sampling the same water mass whenever suitable opportunities arise.

5B.2 Methodological Considerations

5B.2.1 In general, there is no problem in sampling sea water for nutrient determinations. However, it is recognised that sampling and analysis in the continuous mode will yield vital information on the distribution of nutrients in sea water and, therefore, such procedures should be encouraged.

5B.2.2 Based on individual studies, it can be concluded that preservation of samples for the analysis of various nutrient parameters can be done in certain cases. The general conclusion is, however, that preservation of samples for nutrient analysis cannot be recommended as a routine procedure (see Table 1 for further details).

5B.2.3 In order to facilitate comparisons of nutrient data, it is recommended that when reporting such data it be stated whether analysis was carried out immediately after sampling or later on preserved samples.

5B.2.4 In waters of visible turbidity, pretreatment is necessary. In such cases, centrifugation is recommended as it is superior to filtration owing to the greater possibility of contamination occurring with filtration.

5B.2.5 The analytical methodology seems to be well established for most of the nutrients. The outstanding parameters are urea in sea water and total phosphorus and total nitrogen in sediments, for which more methodological work and intercalibrations may be needed.

5B.2.6 There has been an increased application of automatic analysis systems, which have allowed improved precision and increased throughput of samples. However, the Sub-Group regards the manual methods as the reference procedures and, therefore, recommends that checks against these procedures be performed when automated techniques are being introduced in order to assure comparability of the results with those obtained using the previous methodology.

5B.3 Baseline Studies

5B.3.1 Studies and analyses of nutrients have for many years been included in standard hydrochemistry programmes. Consequently, large volumes of nutrient data are readily available. At least for the Baltic Sea area, the present situation does not call for the inclusion of extended nutrient studies in the 1985 baseline study. Whether or not this situation is representative for the entire North Atlantic area could not be fully assessed by the Nutrient Sub-Group.

5B.3.2 The Sub-Group is convinced, however, that large volumes of nutrient data are available at the various North Atlantic institutes although the specific reporting of such data, as called for in C.Res.1976/4:6, has not been implemented. This was clearly shown in the paper MCWG 1983/10/1 by Drs Dooley and Topping.

5B.4 Input Studies

5B.4.1 In order to understand the fluxes and fate of, e.g., nutrients in the marine environment, input studies are required as contributions to mass-balance studies. In this connection, it is important to identify the major sources and study them in detail so as to assess their qualitative as well as quantitative contribution.

5B.4.2 It cannot be the responsibility for the marine chemists alone to carry out such an inventory for riverine and other point sources as well as for atmospheric input. Therefore, cooperative work with colleagues in other disciplines is advocated, making use of data that have been or will be collected by other organisations, e.g., the regional pollution regulatory commissions.

Table 1. Status of Nutrient Determinations in Marine Samples.

D I S S O L V E D

	Satisfactory	Problematic
Sampling	PO_4, ΣP, SiO_4, NH_4	
Pretreatment	NO_2, NO_3, urea, ΣN	
Storage	ΣP, urea, ΣN	PO_4, SiO_4, NH_4, NO_2, NO_3
Analysis	PO_4, ΣP, SiO_4, NH_4 NO_2, NO_3, urea* ΣN*	

* The analytical techniques seem to be well established but our knowledge of the nitrogen cycle is incomplete. Therefore, it can be foreseen that in the future these techniques may need to be refined.

P A R T I C U L A T E

	Satisfactory	Problematic
Sampling, including filtration or centrifugation		ΣP, ΣN
Pretreatment	not applicable	not applicable
Storage	ΣP, ΣN	
Analysis	assessment incomplete	

S E D I M E N T S

	Satisfactory	Problematic
Sampling	ΣP, ΣN	
Pretreatment	not applicable	not applicable
Storage	ΣP, ΣN	
Analysis		ΣP, ΣN

5C. REPORT OF THE SUB-GROUP ON TRACE METALS

5C.1 Four topics were identified for discussion by the Sub-Group, which were:

(1) Present status of trace metal intercalibration of fish tissues and availability/preparation of uncompromised intercomparison samples.

(2) Status of trace metal intercalibration in sea water - Are we in a position to conduct monitoring programmes with ICES laboratories?

(3) Discuss and propose recommendations for indexing of methodologies for ICES data forms.

(4) Discuss/propose new or additional contaminants to be considered (i.e., for overviews, etc.).

5C.2 Intercomparison exercises for metals in biological tissue

5C.2.1 The Sub-Group discussed the results of the analyses of the specimen intercomparison samples which had been prepared by some members of the Sub-Group during the intersessional period. It was agreed that in general the range of concentrations in these samples fulfilled the criteria laid down in the paper presented to the 1982 Statutory Meeting by the Sub-Group Chairman. It was felt, however, that additional intercomparison samples should be prepared to provide an adequate range of metal concentrations in shellfish tissue, and mussels (*Mytilus edulis*)were identified as a suitable material for these additional intercomparison samples. In this respect, it was agreed that two samples, based on background level and contaminated mussel samples, should be collected for conversion into intercomparison samples. Following discussion of these proposals at the plenary session, the Sub-Group was asked to consider whether it was possible to run the intercomparison exercise for metals in biological tissue in two phases. The first phase of the exercise should be organised prior to the 1985 baseline study with the aim to assess the current analytical performance of laboratories conducting monitoring programmes. The second phase should be conducted in parallel to the 1985 baseline study in order to provide an estimate of how well participants in the study had performed in terms of analytical performance. It was suggested that the first phase of the intercomparison exercise would identify those laboratories with relatively poor analytical performance and so afford these laboratories an opportunity to improve their analytical approach (on the basis of advice given to them by the Sub-Group) before they began the monitoring activities in relation to the 1985 baseline survey programme.

5C.2.2. The Sub-Group agreed to consider this proposal in their discussions. Following these discussions the Sub-Group proposed the following design and schedule for the intercalibration exercise (7/TM/BT) in relation to the 1985 ICES baseline study. The overall coordinator of this intercalibration exercise is Dr Berman. The plans are as follows:

(1) Four intercomparison samples based on fish muscle (plaice), lobster hepatopancreas, and two scallop samples (sampled from "clean" and "contaminated" environments in Canada) would be used in the first phase of the exercise. Sufficient quantities of these samples were currently available for distribution to laboratories which were likely to participate in the 1985 baseline study. These samples would be dispatched to participants (see

notes for identification of these laboratories) once homogeneity tests had been completed by Dr S Berman (see notes below).

(2) In regard to the second phase of the exercise, the Sub-Group agreed that five other samples would be prepared and distributed at the start of the ICES 1985 baseline study. These samples would be prepared from dogfish muscle, dogfish liver, cod liver and mussels ("clean" and "contaminated"). These samples would be distributed to the laboratories referred to in (1) above, and other laboratories who had expressed an interest in participating in this particular exercise.

5C.2.3 The timetable for the above two phases of the exercise would be as follows:

Phase 1

(1) Dr U Harms (Federal Republic of Germany) would dispatch the bulk plaice intercomparison sample to Dr S Berman (Canada) by the end of March 1983.

(2) Dr S Berman would arrange for the above sample plus the samples of lobster hepatopancreas and scallops (provided by Dr J Uthe (Canada)) to be reground, mixed and sub-divided into 20 g samples. These aliquots would be stored in glass or high density plastic phials.

(3) A representative number of phials for each intercomparison sample would be subjected to homogeneity testing for the metals under examination.

(4) On completion of (3), Dr S Berman would dispatch samples of each of the intercomparison materials to participants in the exercise. This task would be completed by 1 October 1983.

(5) Participants would be asked to report analytical results by the end of January 1984.

(6) Dr S Berman would prepare a preliminary report on the results of this exercise by 1 April 1984. This report would be distributed to participants and members of the trace metal Sub-Group for comment and evaluation, respectively.

(7) On receipt of these comments, Dr S Berman would prepare a final report on the exercise for the 1984 ICES Statutory Meeting and for tabling at the 1985 Meeting of MCWG. (Should the 1984 MCWG meeting occur after 1 April 1984, it is suggested that the preliminary report be discussed by the Sub-Group so that members can identify laboratories with poor performance and discuss what advice they could pass on to those particular laboratories before they commence their baseline studies in 1985).

Phase 2

(1) The intercomparison samples based on dogfish muscle and liver, cod liver and mussel samples would be prepared during 1983 and the bulk quantities sent to Dr Berman for homogeneity testing by December 1983.

(2) During 1984, Dr Berman would complete the homogeneity testing and the sub-division of samples into 20 g aliquots.

(3) Dr Berman would distribute the above samples to participants in early 1985. Participants would be instructed to run these samples at the same time as they analyse the fish and shellfish samples collected during the baseline study.

(4) Dr Berman would prepare a report on this intercomparison exercise for presentation to MCWG and WGMPNA at their respective 1986 Working Group meetings.

5C.2.4 Action by Members of MCWG in relation to the Intercomparison Exercises

(1) The General Secretary should write to ICES Delegates and the Secretariats of the Oslo, Paris and Helsinki Commissions during March/April 1983 informing them of the proposed phases of the intercomparison exercise and requesting a list of participants for each of these phases. This letter would include a description of the two phases (aims, design, timetable, deadlines, etc.) together with the names of people to whom participants should write to confirm their participation in the exercises. The above information will be provided to Dr Pawlak by Dr Topping shortly after the 1983 MCWG meeting.

(2) Dr Harms will distribute guidance notes on analytical techniques, primarily in relation to the analyses of lead in tissue, to participants before the samples for the first phase of the exercise are distributed in October 1983.

(3) Dr Topping, Dr Jensen and Dr Law will arrange for their respective intercomparison samples of cod liver, mussels and dogfish to be prepared during 1983 and the bulk material to be dispatched to Dr Berman before December 1983.

5C.3 Trace Metals in Sea Water

5C.3.1 The following questions were considered by the Sub-Group on this issue:

(1) Can we include measurements of trace metals in sea water as a part of the 1985 baseline survey program?

(2) If so, would the collection of trace metal data on sea water concentrations provide useful information regarding the objectives of the baseline survey program?

(3) Can the MCWG provide guidelines to ICES laboratories (having little or no experience) regarding trace metal sampling, storage, and analysis for the purpose of the baseline study?

5C.3.2 Concerning the first question, the Sub-Group felt that several ICES laboratories could conduct baseline measurements for soluble Cd, Hg, Zn, Cu, Ni, Fe, and Mn (Al, Co, Mo, V and Cr could probably be added to the list as well). Two metals of interest, Pb and Se, could probably not be measured adequately by these laboratories for the purposes of the baseline survey at the present time. It must be pointed out that this conclusion is based on the results of ICES intercalibrations which show that there are few ICES laboratories with adequate overall capability.

5C.3.3 In answering the second question, the Sub-Group felt that results of baseline measurements of dissolved metals would provide useful information in the vicinity of marine dumpsites, river mouths, outfalls or other potential point sources of metal inputs. Such information would define gradients, indicating sources and transport. In general, however, the measurements of soluble trace metals would be most useful only when ancillary data, such as on salinity, nutrients, regional physical oceanography, river and atmospheric trace metal inputs and sediment metal distributions, are also obtained. This additional information would provide the framework from which assimulative capacities of coastal waters could be assessed.

5C.3.4 Regarding the third question, the Sub-Group felt that guidelines in the form of written instructions regarding sampling and analysis would only be useful to fairly experienced laboratories. In relation to inexperienced laboratories, the Sub-Group, therefore, came up with the following recommendations regarding further training of ICES laboratories in trace metal determinations of sea water samples:

(1) All inexperienced laboratories will require further training or experience before commencing baseline studies.

(2) These training exercises should not be coupled with any other intercalibration activity.

(3) Laboratories identified by past ICES exercises as "expert laboratories" should be used for training of inexperienced laboratories.

(a) These laboratories should be identified by ICES and their cooperation should be officially solicited and encouraged.

(b) Individual trainees should be encouraged to visit "expert labs" and to work with them to gain the necessary training and experience.

5C.3.5 The Sub-Group then considered a telex from Dr D Cossa informing the MCWG that the mercury intercalibration samples collected during the Nantes Intercalibration exercise (5/TM/SW) were probably compromised by contamination. In view of this, Dr Cossa had offered to repeat the intercalibration with samples collected from the Canadian coastal zone. The Sub-Group recommended acceptance of this offer and considered that it might be used to satisfy the needs of JMG for replicate intercalibration samples derived from a contaminated coastal area, rather than open-ocean or uncontaminated coastal areas as has been the case in previous intercalibrations. It was suggested that samples from a mercury contaminated area such as the Saguenay Fjord might be most appropriate but it should be left to Dr Cossa to choose an appropriate location on the basis of his own research in the area. With regard to the sea water sampling intercalibration conducted at Nantes to determine the best method for the collection of sea water samples for mercury analysis, it was decided to await the results of analyses of these samples and then to consider the repetition of this experiment on a multi-laboratory basis at some future opportunity.

5C.4 Recommendations on Indexing of Methodologies for ICES Data Forms

The recommendation of the Sub-Group is discussed in Section 6 of this report.

5C.5 Future Activities

5C.5.1 Regarding future activities, the Sub-Group recognized a continuous need for intercalibration samples for trace metals in biological tissues. Needs for samples of various matrices will continue and efforts to improve and expand abilities for analyses of additional trace metals will be required. At some point in the near future, efforts to develop intercalibration samples for various metal species should be encouraged.

5C.5.2 It is recommended that during the intersessional period the two following projects be undertaken and the results presented at the next Working Group meeting:

(1) A review of the status of trace metal sampling and analysis of sea water by ICES laboratories based on past ICES intercalibration exercises (to be prepared by J M Bewers and J Olafsson).

(2) Review of past studies of atmospheric transport of trace metals to coastal waters, evaluating methods used and information gaps, and, where possible, emphasizing studies in ICES regions (to be prepared by H Windom and A Knap).

5D. REPORT OF THE SUB-GROUP ON ORGANICS

5D.1 Organochlorine Compounds

5D.1.1 Problems with intercomparability of organochlorine analyses in tissue samples suggest that inclusion in the 1985 Baseline Survey of packed column analysis of organochlorines and PCBs based on technical formulations is not justified. In order to improve the quality of analysis, it is necessary to change the method of analysis for PCBs to determine concentrations of a few specific chlorobiphenyl compounds (CBs) by capillary gas chromatography. To this end it is proposed that ICES explore the possibilities of making available, possibly in conjunction with other organizations, a number of authentic reference CBs that may be used by ICES laboratories to improve their methodology. Clearly, this will require funds to permit the acquisition and banking of these reference materials and JMG was suggested as being a possible interested party to be approached in this respect. It is currently felt that only a small number of CBs will be needed and they will be identified later on the basis of the results of MCWG activities.

5D.1.2 The Sub-Group was informed about a group of specialists within the European Economic Community who are carrying out work intended to improve methods of PCB analysis with capillary columns based on the determination of individual components. Several ICES countries are already involved in this exercise and the Sub-Group urged ICES to approach the EEC Bureau of the Community on Reference Materials (BCR) to inquire whether there is a possibility for a few specialists from non-EEC member countries to participate on a voluntary basis. Noting that the BCR will also prepare individual PCB components, it was anticipated that this work might be of help to ICES in obtaining certified standards of CB compounds.

5D.1.3 The Group recognized the need for future IC exercises for organochlorines, but felt that it was premature to formulate plans prior to being able to review the results of 5/OC/BT. If necessary, some intersessional work will be carried out by a few laboratories immediately after the completion and reporting of this exercise at the 1983 Statutory Meeting, and plans may then be formulated for a further exercise. In addition, it will be necessary to study the results of this fifth exercise before recommending the inclusion of some organochlorine compounds in the 1985 Baseline Survey. For this reason, it was felt that in 1984 the MCWG meeting should be held before that of WGMPNA.

5D.1.4 If the fifth exercise proves satisfactory for some organochlorines, and they are recommended for inclusion in the 1985 Baseline Survey, then there will be a need for a new intercomparison exercise including also the distribution of uncompromised reference tissue samples to be analyzed by participating laboratories at the same time as samples for the baseline survey, in order to validate their data. Dr Uthe of the Halifax laboratory is currently considering the feasibility of preparing such materials, at least for those laboratories which did not perform satisfactorily in the most recent intercomparison exercise (5/OC/BT).

5D.1.5 It was noted that intersessional studies by Dr Kerkhoff concerning the possible development of a sodium sulphate/fish flesh homogenate as a material for intercomparison exercise samples had indicated that this type of material was not suitable for this purpose. Thus, extracted oils will be used as substrates for the next intercomparison exercise.

5D.1.6 Laboratories which will participate in the 1985 Baseline Survey and determine organochlorine compounds in fish and shellfish should be encouraged to take samples from as wide an area as possible, so as to achieve an overlap with other laboratories and provide the opportunity to compare results.

5D.1.7 The Sub-Group then discussed the issue of reporting the results of the determination of the fat content of fish and shellfish according to whether extractable or total lipids have been determined, as brought up by Dr Kerkhoff. It was noted that, because the methods used for organochlorine analysis are not designed for the extraction of total fat content, it is important to be able to distinguish between the methods used for the extraction of lipids in the reporting and storage of data on organochlorines in organisms. Further work on the relationship between extractable fat and total fat and organochlorine content will be carried out intersessionally.

5D.1.8 The Sub-Group noted that the analysis of organochlorines in sea water presents an added complication over tissue samples because of the extremely low levels of organochlorines present, necessitating the collection of very large samples. In addition, the differing partitions existing between particulate material and solution for the different organochlorine compounds further complicates the analysis. The advances in methodology proposed in connection with tissue analysis should also lead to improvements in sea water analysis, and specific aspects of appropriate sampling methodology are under investigation by an IOC Sub-Group. No analysis of organochlorines in sea water is proposed for the 1985 Baseline Survey.

5D.1.9 The Sub-Group recognized the importance of sediments as a link in the study of organochlorines in the marine environment, and felt that there is a need for analysts to improve their analytical techniques with respect to sediments, particularly with reference to the determination of specific CB components.

5D.1.10 In the absence of an intercomparison exercise on organochlorines in sediments, the attention of laboratories wishing to improve their methodology is drawn to the three standard reference sediments now available from the National Research Council of Canada. These three materials were initially certified only with respect to their total PCB content. Recently, however, about ten individual chlorobiphenyls have been identified and quantified in these materials. Information concerning this will be released in the near future. The Halifax laboratory of the National Research Council of Canada is currently attempting to build up a stock of individual chlorobiphenyls, mainly by contracting their synthesis by expert laboratories.

5D.1.11 The Group felt strongly that there is a need for an intercomparison exercise in the near future and hoped that a volunteer coordinating laboratory will be forthcoming when definite plans can be formulated.

5D.1.12 Recognizing the complexity of marine sediments, the Group requested advice from the Working Group on Marine Sediments in Relation to Pollution on the collection of representative samples for organochlorine analysis. Specific information requested includes advice on the type of sampling equipment that will maintain surface samples intact and questions of sample homogeneity within a monitoring context.

5D.1.13 The Group recognized that information on the identification of by-products (such as dioxins) and metabolites of organochlorine compounds should be encouraged to be submitted by ICES laboratories to the MCWG or the Statutory Meetings.

5D.1.14 The Group noted that, analytically, toxaphene presents similar problems to CBs. Authentic materials are difficult to obtain, but various laboratories are working to identify single components which may be used to develop methods similar to those proposed for PCBs. It was agreed that toxaphene is of great interest and concern within ICES laboratories, and the development of analytical methodology is encouraged by the Working Group.

5D.1.15 The Group then turned to a question from WGMPNA on whether DDT and its metabolites should be analyzed in the 1985 Baseline Survey of Contaminants in Fish and Shellfish. To answer this question, the MCWG proposed that an overview be prepared on the toxicological aspects of DDT relative to other "new contaminants" in the marine environment. It was felt that WGMPNA was the appropriate body to prepare this overview. The MCWG felt, however, that from a geochemical point of view, it is important to continue measurement of DDT in the marine environment.

5D.2 Hydrocarbons

5D.2.1 Progress towards the second intercalibration exercise on petroleum hydrocarbons in biological tissue (2/HC/BT) was described in detail by Dr Farrington. Approximately 400 samples of homogenized mussel (Mytilus

edulis) tissue have been prepared, and ten randomly selected samples have been analyzed for hydrocarbons by spectrofluorimetry and capillary GC. Concentrations of PAHs in the mussels are in the middle of the expected range for tissue. Fluorescence analyses showed an RSD of 7-10% for the ten replicates, whilst capillary GC analyses of aliphatic and aromatic hydrocarbons showed an RSD of 14 and 28%, respectively. The samples seem sufficiently homogeneous for use in the exercise, but the Group decided that the samples should be freeze-dried to ease shipment problems. This will require re-analysis to check the homogeneity of the samples as distributed and Dr Farrington agreed to carry this out.

5D.2.2 The suggestion was then made that the two planned hydrocarbon intercomparison exercises should be combined, i.e., that both hydrocarbons and PAHs in biological tissue should be determined in one joint exercise so as to economize on distribution costs. This was agreed by the Group, and it is therefore now proposed to distribute mussel homogenates (2/HC/BT), prepared lobster extracts (3/HC/BT) and aliphatic and aromatic standards as one intercalibration kit. It was also agreed that, to prevent duplication and consequent waste of samples, participating laboratories or analysts should request samples on laboratory letterhead stationery.

5D.2.3 It was noted that there is no facility within ICES for the operation of a tissue banking service from which analysts could request standard reference tissue, however, this is under investigation in several ICES member countries and Sub-Group members will report on progress in this matter at the 1984 MCWG meeting. It was recognised that this is a difficult undertaking, requiring of necessity a long-term commitment of funds. The Group discussed the long-term storage problem and it was felt that, as the second round petroleum intercomparison exercise is producing a large amount of samples, some progress may be made in this regard during this programme.

5D.2.4 Considering the question of an intercalibration of hydrocarbon analyses of water samples, it was noted that there is an exercise currently under consideration within IOC and it was felt that ICES should await the results of this exercise before planning further exercises. The Group discussed the results of the Baltic Intercomparison Workshop on Hydrocarbons in Sea Water and recognized the relatively good precision of the water analyses using UVF.

5D.2.5 It was agreed that no recommendations can be made about inclusion of hydrocarbons in the 1985 Baseline Survey until the results of the second round intercalibration are available next year. In order to promote methodological improvements in this field, as many ICES laboratories as possible are encouraged to take part in this exercise.

5D.2.6 Attention was drawn to the difficulty of obtaining a full range of hydrocarbon standards. Analysts are encouraged to submit details of standards used in their laboratories and the suppliers from which they are available to Dr Knap, who will compile a comprehensive information list to be available in 1984.

5D.2.7 Sediments were recognized as a very important environmental compartment for hydrocarbons, and a further intercalibration exercise for sediments is necessary. The rationale for concentrating on tissue sample intercalibration at the present time is that there is a more critical need to assess analysis of hydrocarbons (particularly PAH) in marine tissues because of the public health implications, but a further sediment intercalibration should be carried out as soon as possible. The Group would

also like to request that the Working Group on Marine Sediments in Relation to Pollution consider the sampling of sediments for hydrocarbons, with the same questions addressed as for organochlorines, and report their findings to MCWG.

5D.2.8 For information, it was noted that a shale sample certified for 5 or 6 PAH compounds is available from the U.S. National Bureau of Standards.

6. CODES FOR ANALYTICAL METHODS FOR INTERIM REPORTING FORMAT FOR CONTAMINANTS IN FISH AND SHELLFISH

6.1 The Environment Officer presented for information the ICES Interim Reporting Format for Contaminants in Fish and Shellfish, which will be used for reporting data to ICES for the Cooperative Monitoring Studies programme, to the Helsinki Commission for the Baltic Monitoring Programme and possibly to the Oslo and Paris Commissions for the Joint Monitoring Programme. She stated that in the development of this format, a three-column field had been allocated for a code for the method used to analyze each contaminant and a one-column field had been allocated for a method code for the determination of fat weight. The question now was on what basis a method code should be devised and how the methods should be listed for code development purposes.

6.2 This question was considered during the meetings of the Sub-Group on Trace Metals and the Sub-Group on Organics. These Sub-Groups thereafter jointly recommended that common indexing of methodologies for use on the ICES data reporting forms should not be pursued. Discussions during the session of the full Working Group pointed out the difficulty of establishing a method coding scheme that would adequately take into account the many variations in techniques and their continual change. It was felt that any limited coding system that indexed methods in a generic way, such as by instrumentation, would be of little use to anyone evaluating the data. The Working Group generally felt that any interested reviewer of the data held by ICES would need to contact the originator of the data to obtain the necessary analytical details. Accordingly, the Working Group recommended that each laboratory submitting data on contaminants in marine samples to ICES should maintain detailed records of the methods used to analyze these contaminants and should assign numbers to each of these methods that can be recorded when data are submitted to the ICES Environment Officer.

7. OVERVIEWS ON FLUXES AND TRANSPORT OF CONTAMINANTS IN THE MARINE ENVIRONMENT

7.1 Division Of Responsibilities With WGMPNA

7.1.1 The Chairman reminded the Working Group that, at its 1982 meeting, it had agreed on section headings to be used when overview papers on new contaminants are prepared for MCWG use and, further, that section headings had been proposed for WGMPNA to consider in the preparation of overviews for WGMPNA use (see Annex 7 to C.M.1982/C:1). The Chairman reported that the WGMPNA had considered these proposed section headings at its meeting the previous week and had generally agreed to them, but had made certain modifications to reduce overlap in the subjects covered in the two types of overviews. As agreed by the WGMPNA, the MCWG should cover the physico-chemical aspects of new contaminants in the marine environment, with section headings as follows: (1) production and discharge, (2) transport mechanisms and deposition in the marine environment,

and (3) movement and fate within the marine environment. The WGMPNA would handle the biological aspects of a new contaminant, under the following subject headings: (1) distribution and concentrations in sea water, sediments, and marine trophic levels, (2) toxicology, and (3) implications and public health aspects.

7.2 Furans and Dioxins

.2.1 Dr O'Sullivan presented her paper, entitled "Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans: An overview" (Doc.MCWG 1983/7/2). This paper provided an overview of information on the structure, sources, methods of analysis, occurrence and effects of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). She pointed out that very little information had been available concerning the distribution of these substances in the marine environment.

7.2.2 In the discussion of this paper, it was noted that the analytical procedures, particularly those in the clean-up steps, are complex and no two laboratories use the same procedures. It was thus felt that the analytical difficulties in the determination and quantification of PCDDs and PCDFs in the environment should be stressed more in the paper. Additionally, it was felt that Table 2, showing a correlation between concentrations of TCDD and those of PCBs in several species of fish, could be clarified for readers with a less specialized background in this area.

7.2.3 Dr Reutergårdh stated that there had been several Swedish studies on PCDDs in the marine environment and he would attempt to obtain appropriate information from them which could be added to the paper.

7.2.4 The Working Group agreed that this was a very good paper, containing precisely the kind of information needed in an overview, and thanked Dr O'Sullivan for her conscientious work. The Working Group further agreed that the paper should be transmitted to ACMP, after amendment as agreed above and with additional information supplied by Dr Reutergårdh.

'.3 Polycyclic Aromatic Hydrocarbons

7.3.1 Two papers were available on this subject: (1) Dr Law's amended version of his paper from the previous year, entitled "Polycyclic Aromatic Hydrocarbons in the Marine Environment: An Overview" (Doc. MCWG 1983/7.3/1), and (2) a paper by Dr A Moinet and Dr J Puize entitled "Contamination de l'environnement marin par les hydrocarbures aromatiques polycycliques" (Doc. MCWG 1983/7.3/2). As neither of the authors of this latter paper were present, it was opened to discussion without formal presentation.

7.3.2 Dr Law presented his paper and stated that it was an expanded version of the previous year's paper and that he had taken into account most of the comments and suggestions he had received on his earlier paper. In the discussion, a specific question was raised concerning a statement in Dr Law's paper that fish and shellfish are probably the major source of PAHs in the diet of humans. Dr Law was requested to expand this statement to include additional information on which it is based.

7.3.3 Other questions raised with regard to these papers included whether information could be added on (1) the concentrations of PAHs in fish, (2) the results of work on PAH metabolites in fish, and (3) the results of autoradiographic studies showing the organs in the fish body where the different PAH compounds accumulate. In response to these questions, it

was pointed out that a very large paper would be needed to cover all this information for all the various PAH compounds which have been studied. The Working Group agreed that, rather than attempting to produce a long, comprehensive overview on the subject, the overview should be kept short and concise, focussing in on the major points of concern regarding PAHs in the marine environment. This overview should then contain references to other, more detailed overviews and important literature on PAHs in the marine environment. Working Group members were requested to send references on the most important literature, especially broad overviews, on PAHs to Dr Law for inclusion in the final version of his paper.

7.3.4 Dr Law agreed to work with Dr Piuze to synthesize their two papers into one overall document, incorporating the most important information from both papers and taking into account comments from MCWG as well as from WGMPNA. It was requested that the major points of concern be covered, as given in the section headings for contaminant overviews, and that the references sent by MCWG members be included.

7.3.5 In conclusion, the Working Group thanked the authors of both papers for their work, and especially Dr Law with apologies for asking him to continue this work for another year. The Working Group looked forward to reviewing the final document at its next meeting.

7.4 Polychlorinated Terphenyls

7.4.1 Dr Jensen presented Doc. MCWG 1983/7.4 on PCTs, which is a synopsis of a paper entitled "Polychlorinated Terphenyls (PCTs). Use, Levels and Biological Effects" by Allen Astrup Jensen and Kjeld F Jørgensen. He reported that the full paper will be published in "The Science of the Total Environment". The synopsis paper contained information on concentrations of PCTs detected in various species of organisms and concluded that the most important environmental hazard of PCTs is the possible disturbance of the reproductive system of higher animals. Dr Jensen pointed out that the full report also contained information on the production, discharge and uses of PCTs.

7.4.2 The Working Group felt that the synopsis paper contained useful information, but agreed that a more expanded summary of the full paper should be requested, including a summary of the information on uses, production and discharges of PCTs. Dr Jensen agreed to convey to the authors the gratitude of the Working Group for their paper, along with the request that it be expanded into a fuller summary of the main paper, along the lines that have been agreed for overview papers.

7.4.3 In the discussion of this paper, the great difficulties in analyzing for PCTs were pointed out. It was noted that there are over 8 000 congenitors of PCTs, thus making them considerably more difficult to determine than PCBs. Additionally, it was reported that the concentrations of PCTs determined so far in fish tissues have been very low.

7.5 Photo-Degradation Products of Petroleum Hydrocarbons

7.5.1 Dr Palmork presented a paper which he had prepared with Dr K Tjessem entitled "An Overview of Auto/Photo-oxidation of Petroleum in the Marine Environment" (Doc. MCWG 1983/7.5). He stated that he had prepared this paper because it appeared that the amounts of photo-oxidation products produced were large and their toxicities appeared to be greater than those of the parent petroleum hydrocarbons. The paper contained background

information on why this subject was considered important, information on the techniques used to analyze for these substances, and a discussion of the importance of auto/photo-oxidation processes of oil degradation in relation to other oil degradation processes in the marine environment. On the basis of the information in this overview, Dr Palmork concluded that the photo-oxidation of petroleum hydrocarbons in the marine environment exceeds by far biological degradation by micro-organisms.

7.5.2 In discussing this paper, Dr Ehrhardt reported that studies carried out at Kiel University had indicated that photo-oxidation is a relatively slow process. It was further pointed out that studies of photo-oxidation products are difficult because their quantities are very small and many of these compounds are very transient in the marine environment. These compounds are often difficult to identify even using GCMS.

7.5.3 A question was raised regarding the statement in the paper that photo-oxidation is the main weathering mechanism in dissipating oil residues. It was felt that volatilization is also very important in weathering and that in certain sea areas, microbial degradation can occur at fairly high rates. Thus, it was felt that while photo-oxidation may be an important weathering mechanism, it should not be categorized as the main mechanism.

7.5.4 In conclusion, the Working Group thanked Dr Palmork for his paper and requested him to amend it in accordance with the comments given, for reconsideration at the 1984 meeting of the Group.

7.6 Carbon Dioxide

7.6.1 The Chairman reported that Dr A Poisson had written stating that the paper he would produce on the marine chemical aspects of CO_2 in the environment would not be ready until the 1984 meeting of the Working Group. In the intersessional period, Dr Poisson would be developing plans for an intercalibration exercise on TA, TCO_2, pCO_2 and Ca in sea water and was interested in a list of laboratories in ICES member countries who may be interested in taking part in this intercalibration.

7.6.2 The Working Group thanked Dr Poisson for his work in this regard and looked forward to reviewing this report at the next meeting.

7.6.3 Dr Farrington informed the Group that the Carbon Dioxide Information Center (CDIC), established at the Oak Ridge National Laboratory in Tennessee, USA, intended to issue an informal newsletter entitled "CDIC Communications". This newsletter will be issued free of charge to persons engaged in research related to the CO_2 problem.

7.6.4 In concluding consideration of this topic, the Chairman urged all members to provide papers or other information on the results of work on CO_2 processes in the marine environment for presentation at the 1984 Working Group meeting so that a useful discussion can be held on this subject.

7.7 Selenium

7.7.1 Dr Knap presented the paper "Selenium in the Marine Environment" (Doc. MCWG 1983/7.7) which had been prepared on his request by Dr C I Measures, Massachusetts Institute of Technology, and Dr J J Wrench, University of

British Columbia. In this paper the authors, who have conducted pioneering work on selenium in the oceans, summarized their observations on the marine geochemistry of selenium and indicated the questions which are open for research.

7.7.2 The Working Group expressed their gratitude to the authors for preparing this very thorough review of the geochemistry of selenium in the marine environment. In the discussion, it was noted that no figures were given for the concentration of selenium in sea water, but rather, given the controversy over the true levels of selenium in sea water, the reader was left to consider the data in the references cited and judge for himself.

7.7.3 The Working Group agreed that the overview should first be submitted to the WGMPNA for the addition of sections on the toxicological aspects of selenium and thereafter be submitted to the ACMP as an addition to the series of overviews on the transport and fluxes of substances in the marine environment. In transmitting the paper to the WGMPNA, that Group was requested to consider the question of the reduction of selenium to selenium sulfonite in anoxic sediments and the associated toxicological consequences. The Chairman agreed to write to the authors expressing the gratitude of the MCWG for their paper.

7.8 Zinc

7.8.1 The Chairman presented the paper "Zinc in the Marine Environment - An Overview" (Doc. MCWG 1983/7.8) by Dr P A Yeats. This paper was a geochemical overview of zinc in the marine environment and covered the speciation and concentrations of zinc in the various marine and related compartments, the transport and distribution of zinc in the ocean and an estimate of the fluxes of zinc through the marine environment. The Chairman reported that the paper had been considered by the WGMPNA the previous week and a member of that Group had agreed to supplement the paper with a section on the toxicological aspects of zinc in the marine environment, although zinc was not considered to have serious toxicological effects in the marine environment.

7.8.2 The Working Group requested the Chairman to convey thanks to the author for preparing the paper and agreed that, after amendment in accordance with comments and the addition of the section on toxicology, the paper should be transmitted to ACMP.

7.9 Other Contaminants

7.9.1 The Working Group then considered two papers which had been prepared mainly for the Working Group on Marine Pollution Baseline and Monitoring Studies in the North Atlantic and had been considered in detail by that Group at its meeting the previous week. One of the papers was on toxaphene and the other was on hexachlorobutadiene.

7.9.2 Dr Uthe presented the review paper on toxaphene which he and Dr Reutergårdh had prepared. He stated that they had prepared the paper in response to concerns expressed over toxaphene in the marine environment and to determine whether monitoring for toxaphene should be considered. The paper provided information on the production of toxaphene, its distribution in the environment, and its toxicology. It was pointed out that there are severe problems in analyzing for toxaphene because there are many different components in toxaphene and it is difficult to separate these compounds from other chlorinated hydrocarbons.

7.9.3 Dr Kerkhoff then summarized the main points of her overview paper on hexachloro-1, 3 - butadiene (Doc. MCWG 1983/7.9/1). The paper covered the major sources of HCBD emission, its toxicity and bioaccumulation, and its occurrence and distribution in the marine environment. Based on this information, Dr Kerkhoff stated that HCBD appears only to be a problem in the vicinity of factories emitting HCBD and, thus, HCBD is not expected to cause problems in marine waters. Concerning monitoring for HCBD, she pointed out that because HCBD contamination is accompanied by HCB contamination, one can monitor for HCB and only study HCBD when the occurrence of high levels of HCB has been found.

7.9.4 The Working Group thanked Drs. Uthe and Reutergårdh and Dr Kerkhoff for presenting their excellent overview papers. It was agreed that any members with comments on these papers should write directly to the authors.

7.9.5 The Working Group then discussed whether there were other contaminants for which the preparation of overviews should be initiated. It was noted that several substances have been under discussion in some of the pollution regulatory commissions as to whether these substances should be "black listed", "gray listed" or not contained at all in the convention's annexes. It was felt that overviews on these substances could help to determine from a scientific standpoint their importance in the marine environment. The possibility of preparing overviews on chromium, fluorides, or organosilicon compounds was considered and Dr Kerkhoff agreed to see whether one of her colleagues in the Netherlands could prepare an overview paper on organosilicon compounds in the marine environment for the next Working Group meeting.

7.9.6 Recalling the discussion under Agenda Item 4 on the effects of tributyltin on oysters and noting that a considerable amount of work has been done on this subject in France, it was queried whether a French scientist could prepare an overview paper on alkyl-tin compounds. Dr Thibaud agreed to request one of his colleagues to prepare this overview.

7.9.7 It was also suggested that it would be useful to have an overview prepared on nutrients and hypertrophication in the marine environment, given the possible relationships between high nutrient concentrations and unusual plankton blooms and low dissolved oxygen concentrations in bottom waters of certain areas. The Working Group agreed that this was an important topic for an overview but felt that, given the vastness of the subject area and the many open questions, it would be better to wait at least one year before beginning work on an overview on nutrients.

7.9.8 In concluding the discussion on this entire agenda item, the Chairman, on behalf of the Working Group, thanked all the authors who had prepared or will prepare overviews. He reminded members to keep aware of discussions in regulatory commissions concerning whether certain contaminants should or should not be included in annexes to their conventions and requested members to bring these subjects up when applicable.

8. PREPARATIONS FOR THE ICES BASELINE SURVEYS

8.1 The Working Group recalled that at the 1982 Statutory Meeting the Council had approved the conduct of a baseline survey on contaminant levels in fish and shellfish (C.Res.1982/4:6), subject to prior satisfactory intercalibration of analyses of the contaminants. The Council had also given approval in principle to the conduct of a baseline study of trace metals

in coastal and shelf sea waters of the North Atlantic (C.Res.1982/4:8), pending a satisfactory outcome of the Fifth Round Intercalibration on Trace Metals in Sea Water (5/TM/SW).

8.2 With regard to the baseline study of contaminants in fish and shellfish, the Working Group noted that the WGMPNA had established a planning group at its 1982 meeting to prepare detailed guidelines for the conduct of this baseline survey. These plans had been completed at the 1983 WGMPNA meeting, subject to endorsement by the ICES/SCOR Working Group on the Study of the Pollution of the Baltic, which is also participating in this study. The WGMPNA had agreed that the analysis of the following substances should be considered mandatory: (1) metals: mercury, copper, zinc, cadmium, lead, and arsenic; (2) organochlorine compounds: HCH, HCB, dieldrin, PCBs, op and pp DDT, DDE and DDD. Several other substances were included on an optional list of analytes, to attempt to establish their general baseline distribution. These are: (1) metals: chromium, nickel, selenium, vanadium, tin and manganese; (2) organic compounds: petroleum hydrocarbons and polycyclic aromatic hydrocarbons, toxaphene and chlordane. The WGMPNA had then requested the MCWG to coordinate appropriate intercalibration exercises on the analysis of as many of these contaminants as possible, noting that plans were already in hand for an intercalibration exercise on trace metals in biota.

8.3 In response to this request, the MCWG noted that its Sub-Group on Trace Metals had reported that from an analytical standpoint there would be no problem analyzing at least the mandatory trace metals in the fish and shellfish tissues proposed. Furthermore, the Seventh Intercomparison Exercise on Trace Metals in Biological Tissue (7/TM/BT) has been specifically planned to assist laboratories participating in this baseline survey to develop their methods and, later, to provide information on the quality of analysis of baseline survey samples. In contrast to the situation with regard to metals, the MCWG noted that the Sub-Group on Organics had not been in a position to state that any organic contaminants could be analyzed sufficiently well to be included in a multi-laboratory baseline survey. At present, no new intercalibration exercise on organochlorine residues was being planned, pending an analysis of the results of the Fifth Intercalibration Exercise on PCBs in Biota (5/OC/BT). Concerning the optional organic contaminants, petroleum hydrocarbons and polycyclic aromatic hydrocarbons, intercalibration exercises on the analysis of these substances in biota would be underway shortly and no statements concerning the possible inclusion of these contaminants in the baseline survey could be made until these intercalibrations have been completed. Thus, the Working Group concluded that, at the present time, it could not recommend that any organic contaminants be included in the 1985 baseline survey of contaminant concentrations in fish and shellfish.

8.4 Turning to the 1985 baseline survey of trace metals in sea water, the Working Group noted that the Sub-Group on Trace Metals had stated that trace metals in sea water could be measured under the conditions indicated in the Sub-Group report (see Section 5C.3 above)but had not made a firm recommendation that these measurements actually be done. In specific, the Sub-Group had indicated that several ICES laboratories could conduct baseline measurements of soluble cadmium, mercury, zinc, copper, nickel, iron and manganese (and probably also aluminium, cobalt, molybdenum, vanadium and chromium). The Sub-Group had also indicated steps which could be taken to assist less experienced laboratories to develop better

capabilities in the measurement of trace metals in sea water. With regard to the WGMPNA request for guidelines for sampling, pretreatment, and analysis of sea water, it was noted that the Sub-Group had agreed that such guidelines would only be useful to fairly experienced laboratories. However, it would be possible to develop guidelines for the sampling procedures (see para.12.2, below).

8.5 With regard to a possible inclusion of nutrients or organochlorine compounds in the sea water baseline survey, the Working Group noted that the Sub-Group on Nutrients had indicated that it felt that there was no need to include nutrient measurements in the baseline survey. The Sub-Group on Organics had stated that it was not yet possible for analytical reasons to include organochlorines in such a survey.

8.6 The Working Group then considered the question of where the sea water samples should be taken in the context of a coastal water baseline survey, including the question from the WGMPNA as to whether river mouths should be included. The Working Group noted that the Sub-Group on Trace Metals had stated that it would be useful to study dissolved metals in the vicinity of marine dumpsites, river mouths, and outfalls or other point sources of metal discharge. To obtain the greatest use of these data, the Sub-Group had recommended that data also be obtained on the salinity, concentrations of nutrients, regional physical oceanography, river and atmospheric trace metal inputs and sediment metal distributions.

8.7 Having considered this information, the Working Group agreed that there was value in including trace metal riverine input measurements in the baseline survey of metals in sea water to the extent that input measurements have already been established on a given river and the methods used are acceptable. In this connection, it was recalled that the Working Group had approved a methodology for the assessment of riverine inputs of metals at its 1982 meeting and that this methodology had been accepted by the Advisory Committee on Marine Pollution and published as Annex 6 to the 1982 ACMP Report (Coop.Res.Rep.No.120 (1983)). It was, however, felt that to make the measurements of trace metals in river water meaningful, an intercalibration exercise on the analysis of trace metals in fresh water is needed to assist in interpreting the quality of river water data.

8.8 Finally, noting that the detailed plans for the baseline survey on trace metals in sea water would be developed by a small planning group consisting of representatives of the WGMPNA, the MCWG and also the Working Group on Shelf Seas Hydrography, the MCWG asked its Chairman, Dr Bewers, to serve on this planning group along with either Dr Duinker or Dr Kremling.

9. STORAGE CONDITIONS FOR BIOLOGICAL SAMPLES

9.1 The Environment Officer reported that the ACMP had not been satisfied with the MCWG's treatment of its question concerning the most appropriate conditions and procedures for short-term storage of biological samples prior to analysis, as contained in Section 8 of the 1982 MCWG Report (Doc. C.M.1982/C:1). The ACMP had accordingly requested the MCWG to consider this subject again to determine whether the best storage methods are actually being used and also to look at possible new methods of storage.

9.2 The Working Group felt that it was not clear enough what advice the ACMP wished, but agreed to provide additional details to its advice of the previous year. The Working Group generally felt that storage of biological materials in a deep freezer at -20°C is adequate for the subsequent analysis of trace metals, but that the storage issue was not so clear for organic contaminants. One important and well-recognized problem, however, concerns the determination of the wet weight basis for samples which will be frozen. After considering several procedures for accounting for the problems of determining the wet or fresh weight basis in terms of deep freeze storage of samples, it was agreed that no commonly accepted procedure could be documented because there were differences of opinion on this matter. The Group finally agreed, however, to accept the proposals made by Drs Topping and Uthe on the basis of the practice adopted in their respective laboratories. This approach consists of the following options or procedural steps:

1) Whenever possible, the analyst should process the samples of fish and shellfish immediately after collection and perform analysis on a representative portion of these samples (in some cases this would be an homogenate of the tissue) within a day of processing.

2) In the event that analysis cannot be performed in this specified period, a representative, weighed sample of the tissue (i.e., a sufficient weight to allow a complete analysis of the sample for the contaminants in question) should be deep frozen in a suitable container until the analytical work can be done.

3) In cases in which samples of entire fish have arrived at the laboratory in a deep frozen condition, the following action is suggested:

   a) The sample should be semi-thawed, filleted, and a representative sample of the fillet homogenized and a sub-sample taken for immediate analysis.

   b) In the event that these homogenates cannot be analyzed immediately, they should be deep frozen. Prior to analysis, these homogenates should be thawed, re-homogenized, and then sub-sampled.

The above procedural steps are considered necessary to avoid artifacts in analysis caused by wet weight changes in fish and shellfish samples which have been frozen, and thereby influencing estimates of contaminant concentrations in samples of frozen organisms versus estimates of concentrations in freshly-caught fish or shellfish.

10. RELEVANT NUTRIENT STUDIES

10.1 It was noted that most of the issues related to this agenda item had been discussed by the Sub-Group on Nutrients and the results reported in Section 5B, above.

10.2 Among additional items of information mentioned were that the results of a joint study among laboratories in Sweden, the Federal Republic of Germany,

and the USSR on patchiness in the distribution of nutrients in the Baltic Sea will be presented at the Workshop on Patchiness Experiments in the Baltic Sea (Tallinn, 21-23 March 1983). These papers will be referred back to the MCWG for information at the 1984 meeting. Additionally, an intercalibration of analyses of nutrients in sea water had been carried out during the Biological Intercalibration Workshop, held under the auspices of the Helsinki Commission in Rønne in August 1982. The report of the results of this nutrient intercalibration will be presented for information at the next MCWG meeting.

11. INFORMATION ON "NEW" CONTAMINANTS

11.1 It was noted that this subject had already been considered during the discussion under Agenda Item 7 on the overviews on contaminants in the marine environment and had also been considered by the Sub-Group on Trace Metals and the Sub-Group on Organics. There were no additional suggestions of new contaminants to be considered by the Working Group at the present time. The Working Group, however, agreed that there should be a re-assessment of the measurement of DDT in relation to other anthropogenic organics within baseline and monitoring programmes.

12. LEAFLETS ON "TECHNIQUES IN MARINE CHEMISTRY"

12.1 The Working Group considered the status in the preparation of leaflets on methods for publication in the "Techniques in Marine Chemistry" series. The Environment Officer reported that, to date, draft leaflets had been submitted for determinations of (1) silicate in sea water, (2) dissolved aluminium in sea water, (3) mercury in sea water using cold vapour AAS, and (4) cadmium and lead in biota. She reminded the Group that to begin the publication of this new series, about ten leaflets should be published at the same time. Thereafter, publication would occur when two or three manuscripts were in hand.

12.2 Each editorial board then reviewed the progress in preparation of leaflets in their subject area. The Editorial Board for Nutrients stated that, with the exception of the two methods already submitted, the methods for all the other nutrients were in the process of being published in the second edition of "Chemical Analysis of Sea Water". Thus, no work could be done regarding further leaflets until this book has appeared. The Editorial Board for Trace Metals stated that it would be possible to prepare leaflets on the analysis of trace metals in biota, in addition to the leaflet on cadmium and lead in biota which has already been prepared by Dr Harms, if this is agreed by the Working Group. Concerning trace metals in sea water, it was noted that Dr D Schmidt had recently submitted a draft leaflet for the determination of mercury. In addition, Dr Bewers and Mr Olafsson agreed to prepare a leaflet on sampling methods for studying trace metals in sea water. The Editorial Board for Organics stated that no leaflet could be prepared on the analysis of PCBs in biota until the results of the Fifth Intercalibration on PCBs in Biological Tissues (5/OC/BT) are available, because the analysis of PCBs has changed completely over the past two years and it is not yet clear how comparable the new methods are. Concerning the leaflet on the analysis of PCBs in sea water which had been promised by Drs. Palmork and Duinker, it was noted that this method would now be published in "Chemical Analysis of Seawater" and thus was affected by the same copyright problems as the nutrients methods.

12.3 In the general discussion, the Working Group agreed that leaflets should only be prepared for methods which have been shown to be accurate and give intercomparable results, indicating the coefficients of variation attainable, when possible. In the interim before guidelines on particular methods are available, publications by laboratories using applicable methods should be consulted.

12.4 Later in the meeting, the Working Group decided that, as methods are constantly changing and each laboratory has its own special variations on any particular method, it would not be useful to publish leaflets on methods in a series such as "Techniques in Marine Chemistry". The Working Group agreed that, instead of such leaflets, each laboratory should develop a detailed description of the methods it uses for sampling, sample preparation and preservation, and analysis and make such descriptions available to persons requesting them. When preparing these detailed descriptions, the approach outlined for the leaflets at the last meeting (Doc. C.M.1982/C:1, para. 13.1) should be followed. Thus, if a scientist wishes to have a detailed description of a method, he should request this directly from an ICES laboratory. The Working Group agreed to propose to the ACMP that this new system be used as an alternative to the leaflets and, thus, that the plans for the "Techniques in Marine Chemistry" series be dropped.

13. ANY OTHER BUSINESS

13.1 The Working Group considered the subject of the chemistry of the surface layer of the oceans, particularly the surface microlayer, and especially with respect to the atmospheric deposition of contaminants, noting that there was an interest in these issues by the WGMPNA, the Oslo and Paris Commissions and the Helsinki Commission. It was noted that for some sea areas, a lot of work has been done to determine the atmospheric deposition of trace metals, while some work has been done on organochlorines and only very little on petroleum hydrocarbons. The results of these studies indicate that for certain coastal areas, the input of trace metals to the sea by atmospheric deposition can exceed the input of trace metals from land. It was noted, however, that there are many methodological difficulties involved in the study of atmospheric deposition to the sea and that these left many open questions, one important question being the exact contribution of dry fallout to the overall atmospheric deposition.

13.2 The Working Group agreed that, to obtain a better picture of the relative importance of atmospheric deposition, an overview paper should be prepared. Dr Windom together with Dr Knap volunteered to write an overview on the atmospheric deposition of trace metals to coastal waters. Dr Windom stated that they would review the studies which have attempted to quantify the atmospheric deposition of trace metals and review the types of methods used and try to evaluate these methods. Dr Farrington offered to contact a colleague to obtain information on the deposition of PAHs to the sea.

13.3 On the questions regarding the surface microlayer, it was noted that a GESAMP working group is preparing a report on air-sea interaction which may contain relevant information. Accordingly, the Working Group agreed to wait until it has reviewed this GESAMP report and the overview papers agreed in the preceeding paragraph before deciding whether it should do any work on this subject.

13.4 The Working Group then considered a series of papers prepared by Belgian scientists for the 1982 Statutory Meeting. These papers were "Boundary Conditions for Heavy Metals at the Air-Sea Interface" by F Dehairs, et al. (C.M.1982/E:33) and "Distribution, Transport and Fate of Bi, Cd, Cu, Hg, Pb, Sb and Zn in the Belgian Coastal Marine Environment" (C.M.1982/E:34, E:35, E:36, E:37, E:38 and E:39). Dr Vandamme highlighted the main points from the first of these papers, on atmospheric deposition of metals, and pointed out that the results of these studies showed that the atmospheric deposition of copper, zinc, lead and cadmium in the Belgian coastal waters is larger than the input of these metals by the River Scheldt. The study also showed that wet precipitation is more important than dry deposition in the overall atmospheric deposition of most metals to this sea area.

13.5 The Working Group agreed that the authors of these papers had done an excellent job in bringing together information from many sources concerning the distribution, transport and fate of the trace metals studied in the Belgian coastal area. The Chairman then opened the papers to discussion, focussing on the philosophy of the approach used to put the information together. Several members commented on the results in the papers, but a large number of members had been unable to obtain copies of the papers, and so were unable to contribute to the discussion.

13.6 Accordingly, it was agreed that all members should obtain copies of these papers in the intersessional period so that a discussion of the concepts and approach used in the papers could be held at the 1984 Working Group meeting. The Working Group asked Dr Vandamme to convey to his Belgian colleagues and authorities the Group's appreciation of this very interesting work and also to inquire whether it might be possible to have one of the authors attend the next Working Group meeting to present the results of these studies in greater detail.

13.7 The Working Group then considered information on an intercalibration exercise on the analysis of sediments which will be coordinated by Dr L Brügmann (German Democratic Republic) in connection with the Pilot Sediment Study in the Baltic Sea, under the ICES/SCOR Working Group for the Study of the Pollution of the Baltic. It was noted that there would be the possibility for a number of laboratories (about 20) from outside the Baltic Sea area to participate in this intercalibration exercise and that requests for participation should be forwarded to the ICES Secretariat before 1 May 1983. It was expected that the Working Group on Marine Sediments in Relation to Pollution would consider this proposal in detail and respond to any questions asked.

14. APPROVAL OF RECOMMENDATIONS AND DEADLINES

14.1 The Working Group noted the requests made to other Working Groups during the course of the meeting and agreed to attach a short statement of each request in Annex 4.

14.2 The Working Group then considered and approved the Action List for intersessional work; this is attached as Annex 5.

14.3 Noting that the only recommendation which should be brought to the attention of Council at the next Statutory Meeting was that for the next meeting of the Working Group, the Group discussed this in some detail. It was agreed that, given the interest of the MCWG in issues related to the chemistry of sediments and the role of sediments in the biogeochemical cycling of substances in the marine environment, it would be very useful to have a one-day joint

meeting with the Working Group on Marine Sediments in Relation to Pollution. The MCWG felt that it would be useful to have both groups meeting during the same week, with the MCWG meeting beginning on Monday and the WGMS meeting beginning on Tuesday or Wednesday, depending on how long that meeting should last. The joint meeting could take place on Wednesday. The MCWG meeting should be scheduled for four days, and it should preferably be held near the end of February 1984 in a location other than Copenhagen, depending on offers received to host the two meetings. The terms of reference for this meeting were agreed as follows:

1) to review the results of the following intercalibration exercises:
   a) the Fifth Intercomparison Exercise on PCBs in Biota (5/OC/BT),
   b) the Fifth Round Intercalibration for Trace Metals in Sea Water (5/TM/SW);

2) to review the status of the intercomparison exercises on petroleum hydrocarbons (2/HC/BT) and polycyclic aromatic hydrocarbons (3/HC/BT) in biological tissue;

3) to review the overviews on atmospheric deposition of substances to the marine environment and on the status of sampling and analysis of sea water for trace metals;

4) to re-examine the list of contaminants to be included in the baseline studies on contaminants in fish and shellfish and on trace metals in sea water; and

5) to consider the overview on CO_2 cycling in the oceans and other relevant information.

14.4 The terms of reference for the joint meeting with WGMS would be to consider matters of common interest on the chemistry of marine sediments, including questions on the analysis of contaminants in sediments and possible intercalibration exercises on the analytical methods. The full recommendation is contained in Annex 6.

14.5 The Working Group also considered the timing of its meeting in relation to that of the WGMPNA and agreed that it was preferable for MCWG to meet before WGMPNA. Given the existing level of good communications between these two Working Groups, it was felt that it was not necessary for their respective meetings to be held during consecutive weeks in 1984.

14.6 As there was no further business, the Chairman thanked the members for their participation and expressed special gratitude to the Chairmen of the three Sub-Groups and their rapporteurs for the excellent work they had done. The Chairman then closed the meeting at 13.00 hours on 10 February 1983.

ANNEX 1

MARINE CHEMISTRY WORKING GROUP

Fifth Meeting

Copenhagen, 7-10 February 1983

AGENDA

1. Opening of meeting and adoption of agenda
2. Report of the 70th Statutory Meeting
3. Report on other related activities
   - 3.1 Working Group on Marine Pollution Baseline and Monitoring Studies in the North Atlantic
   - 3.2 Working Group on Marine Sediments in Relation to Pollution
   - 3.3 ICES/SCOR Working Group on the Study of Pollution in the Baltic
   - 3.4 Joint Monitoring Group of the Oslo and Paris Commissions
   - 3.5 IOC/GIPME activities and plans
   - 3.6 Other relevant activities of multilateral and international agencies
4. Reports on projects and activities within ICES countries
5. Intercalibration activities and plans
   - 5.1 Fifth Round Intercalibration for Trace Metals in Seawater (5/TM/SW)
   - 5.2 Organochlorines in biological tissue (5/OC/BT)
   - 5.3 Metals in biological tissue (7/TM/BT) - progress
   - 5.4 Polycyclic aromatic hydrocarbons in shellfish tissue (3/HC/BT)
   - 5.5 Petroleum hydrocarbons in biological tissues and marine sediments (2/HC/BT and 2/HC/MS respectively)
   - 5.6 Progress with sodium sulphate/tissue blend matrices
   - 5.7 Other intercalibrations
   - 5.8 Revision/amendment of philosophy/notation drafted at Fourth Meeting (Annex 5)
6. Codes for analytical methods for Interim Reporting Format for Contaminants in Fish and Shellfish
7. Overviews on fluxes and transport of contaminants in the marine environment
   - 7.1 Division of responsibilities with WGMPNA
   - 7.2 Furans and Dioxins
   - 7.3 Polycyclic Aromatic Hydrocarbons
   - 7.4 Polychlorinated Terphenyls
   - 7.5 Photo-degradation products of petroleum hydrocarbons
   - 7.6 Carbon Dioxide
   - 7.7 Selenium
   - 7.8 Zinc
   - 7.9 Other contaminants (including proposals for new overviews).

8. Preparations for ICES baseline surveys
9. Storage conditions for biological samples
10. Relevant nutrient studies
11. Information on "new contaminants"
12. Leaflets on "Techniques in Marine Chemistry"
13. Any other business
14. Approval of recommendations and deadlines

ANNEX 2

MARINE CHEMISTRY WORKING GROUP

Copenhagen, 7-10 February 1983

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ANNEX 3

WORKSHOP ON THE ANALYSIS OF HYDROCARBONS IN SEAWATER

The Workshop was held at the Institut für Meereskunde an der Universität Kiel, March 23 - April 3, 1981.

Participants in the Workshop came from the United Kingdom and from all countries bordering the Baltic Sea, except the USSR.

The Workshop was divided into three sections, starting with presentations of invited papers on subjects related to the contamination of sea water with petroleum hydrocarbons. Part two included the collection of sea water samples and the measurement of concentrations of oil residues dissolved and/or finely dispersed in surface water by the UV fluorescence method recommended by IOC.

Each participant used his or her own samplers, sampling, and work-up procedure, but all concentrates were measured in the same way.

As part three of the Workshop, each participant was provided with an aliquot of one integrated sample obtained by concentrating dissolved lipophilic material from approximately 760 dm^3 of sea water by sorption onto Amberlite XAD-2 resin. These samples were taken back to each participants' home laboratory and analyzed by individually-developed methods.

The following is a short summary of results of discussions following the oral presentations:

Excitation at 310 nm and measurement of emission at 360 nm as recommended by IOC is fairly sensitive for residues of whole crudes, but less suitable for assessing concentrations of light fuels such as diesel containing relatively high concentrations of monocyclic aromatics. In case such material is to be measured, excitation at 270 nm and emission at 330 nm result in higher sensitivity.

Extraction of petroleum hydrocarbons from sea water can be done effectively at the natural pH of sea water. As was confirmed by the ensuing practical work, hexane or cyclohexane are just as effective for extracting low concentrations of petroleum residues from sea water as the originally recommended carbon tetrachloride. Added advantages of hexane and cyclohexane are low toxicity and the fact that the same solvent can be used both for extraction and for the fluorescence measurement. Thus, samples do not have to be taken to dryness in order to change from carbon tetrachloride to hexane, thus eliminating the hazard of losing sample material by evaporation.

It was found that more information on the composition of the extracted material could be obtained by simultaneous scanning of excitation and emission wavelengths with a wavelength difference of 25 nm.

A study on the composition of fluorescent material from Baltic sea water showed that organisms do not appear to contribute to it. However, the 5-20 times higher concentrations of unsubstituted PAHs relative to alkyl-substituted compounds appears to indicate a considerable input of pyrogenic PAH's, presumably via the atmosphere.

Since UV fluorescence is insensitive to the presence of biogenic saturated and olefinic hydrocarbons and PAH's have never been related unambiguously to biological sources, the method can be used to measure selectively non-biogenic hydrocarbon concentrations in sea water. It cannot differentiate, however, between fossil and pyrogenic PAH's. Detailed analyses on the single compound level are necessary to provide this information.

Concerning sub-lethal effects of petroleum hydrocarbons, results of laboratory experiments were reported which showed that crude oil at concentrations as low as 0.2 - 15 µg . dm^{-3} interfered with the attraction of Fucus gametes to eggs.

Results of the practical field work were rather encouraging. In spite of different sampling and work-up procedures, a standard deviation of ± 37% was obtained with a mean value of 0.98 µg . dm^3 (extractant: carbon tetrachloride). With hexane as extractant, the standard deviation of results was ± 14.8% with a mean value of 1.35 µg . dm^3. Assuming an equal concentration of oil residues in the water 5 days after the first round of sampling, the higher concentration may be due to the simplified work-up procedure. Dichloromethane was also tested as an extractant but was found to extract an undue amount of material interfering with the measurement of petroleum residues.

The comparison of results obtained from the integrated sample at the participants' home laboratories showed a standard deviation of ± 28.6% with a mean value of 0.42 µg. dm^3.

In the light of these results, the selection of a reference crude oil does not appear to be critical, since a comparison of the specific fluorescence intensities of 21 different crude oils gave a standard deviation of ± 17.94% with a mean value of 0.856 when the response of a light Iranian crude was taken as unity.

Nevertheless, it was recommended by the Workshop participants to use an artificially weathered Ekofisk crude as reference oil for investigations in the Baltic and North Sea.

Other recommendations are: (1) to compare the intensities of the Rámán peaks of pure solvent and solution as a test for self-absorption and (2) to use either n-hexane or cyclohexane as extractant.

A copy of the Workshop Proceedings may be obtained from:

Baltic Marine Environment Commission
Eteläesplanadi 22 C 43
SF-00130 Helsinki 13
FINLAND

ANNEX 4

ISSUES RELATED TO OTHER ICES WORKING GROUPS

1. WORKING GROUP ON MARINE POLLUTION BASELINE AND MONITORING STUDIES IN THE NORTH ATLANTIC

   A. The overview paper on selenium should be submitted to the WGMPNA for the addition of sections on the toxicological aspects of selenium and thereafter be submitted to the ACMP as an addition to the series of overviews on the transport and fluxes of substances in the marine environment. In transmitting the paper to the WGMPNA, that Group was requested to consider the question of the reduction of selenium to selenium sulfonite in anoxic sediments and the associated toxicological consequences (see paragraph 7.7.3).

   B. The MCWG has proposed that an overview be prepared on the toxicological aspects of DDT relative to other "new contaminants" in the marine environment. It was felt that WGMPNA was the appropriate body to prepare this overview. The MCWG felt, however, that from a geochemical point of view, it is important to continue measurement of DDT in the marine environment (see paragraph 5D.1.15).

2. WORKING GROUP ON MARINE SEDIMENTS IN RELATION TO POLLUTION

   A. Recognizing the complexity of marine sediments, the Group requested advice from the Working Group on Marine Sediments in Relation to Pollution on the collection of representative samples for organochlorine analysis. Specific information requested includes advice on the type of sampling equipment that will maintain surface samples intact and questions of sample homogeneity within a monitoring context. (see paragraphs 5D.1.9 - 5D.1.12).

   B. Sediments were recognized as a very important environmental compartment for hydrocarbons, and a further intercalibration exercise for sediments is necessary. The rationale for concentrating on tissue sample intercalibration at the present time is that there is a more critical need to assess analysis of hydrocarbons (particularly PAH) in marine tissues because of the public health implications, but a further sediment intercalibration should be carried out as soon as possible. The Group would also like to request that the Working Group on Marine Sediments in Relation to Pollution consider the sampling of sediments for hydrocarbons, with the same questions addressed as for organochlorines, and report their findings to MCWG (see paragraph 5D.2.7).

   C. It was agreed that, given the interest of the MCWG in issues related to the chemistry of sediments and the role of sediments in the biogeochemical cycling of substances in the marine environment, it would be very useful to have a one-day joint meeting with the Working Group on Marine Sediments in Relation to Pollution. The MCWG felt that it would be useful to have both groups meeting during the same week, with the MCWG meeting beginning on Monday and the WGMS meeting beginning on Tuesday or Wednesday, depending on how long that meeting should last. The joint meeting could take place on Wednesday (see paragraph 14.3).

ANNEX 5

ACTION LIST

The following intersessional activities have been agreed by the Working Group.

1. All members who wish to report on the results of relevant research activities in their laboratories or countries should prepare a short written statement, giving references and contact persons (paragraph 4.8).

2. Dr Bewers, Dr Berman, Dr Cossa and Dr Coureau have agreed to write papers giving the results of the various parts of 5/TM/SW for presentation at the 1983 Statutory Meeting (paragraph 5.1.2).

3. Dr Uthe will prepare a paper on the results of 5/OC/BT for the 1983 Statutory Meeting (paragraph 5.2.1).

4. All members are encouraged to check with colleagues to obtain relevant data on nutrients, as called for in C.Res.1976/4:6, and send them to Dr H D Dooley, Marine Laboratory, P.O.Box 101, Victoria Road, Aberdeen AB9 8DB, Scotland, as soon as possible (paragraph 5B.3.2).

5. Dr Berman, Dr Harms and Dr Uthe should carry out the preparations for 7/TM/BT as given in paragraph 5C.2.3.

6. Dr Topping should provide information for the announcement of 7/TM/BT to the Environment Officer by 15 April 1983 (paragraph 5C.2.4(1)).

7. Dr Harms will distribute guidance notes on analytical techniques, primarily in relation to the analyses of lead in tissue, to 7/TM/BT participants before the samples for the first phase of the exercise are distributed in October 1983 (paragraph 5C.2.4(2)).

8. Dr Topping, Dr Jensen and Dr Law will arrange for the respective intercomparison samples of cod liver, mussels and dog fish to be prepared during 1983 and the bulk material to be dispatched to Dr Berman before December 1983 (paragraph 5C.2.4.(3)).

9. Dr Bewers and Dr Olafsson should prepare a review of the status of trace metal sampling and analysis of sea water by ICES laboratories based on past ICES intercalibration exercises (paragraph 5C.5.2(1)).

10. Dr Windom and Dr Knap should prepare a review of past studies of atmospheric transport of trace metals to coastal waters, evaluating methods used and information gaps, and, where possible, emphasizing studies in ICES regions (paragraph 5C.5.2(2)).

11. All members and their colleagues are encouraged to submit details of hydrocarbon standards used in their laboratories and the suppliers from which they are available to Dr Knap, who will compile a comprehensive information list to be available in 1984 (paragraph 5D.2.6).

12. Each laboratory submitting data on contaminants in marine samples to ICES should maintain detailed records of the methods used to analyze these contaminants and should assign numbers to each of these methods that can be recorded when data are submitted to the ICES Environment Officer (paragraph 6.2).

13. Dr O'Sullivan will revise her paper on PCDDs in accordance with comments supplied and attempt to finalize it by 15 May 1983 (Section 7.2).

14 Members were requested to send references of the most important papers on PAHs to Dr Law, who will add them to his overview on PAHs; Dr Law will also work with Dr Piuze to attempt to amalgamate their respective PAH overviews into one, for presentation at the 1984 Working Group meeting (Section 7.3).

15. Dr Jensen is requested to ask the authors of the PCT overview to provide a more comprehensive summary of their full-length paper on PCTs for the 1984 Working Group meeting (Section 7.4).

16. Dr Palmork will revise his paper on photo-oxidation of petroleum hydrocarbons for presentation at the 1984 Working Group meeting (Section 7.5).

17. All members are urged to provide papers or other information on the results of work on CO_2 processes in the oceans for the 1984 Working Group meeting (Section 7.6).

18. Dr Bewers will forward the selenium overview to the Chairman of WGMPNA to obtain the addition of a section on the toxicological aspects of selenium (Section 7.7).

19. Dr Kerkhoff will attempt to find a colleague to prepare an overview on organosilicon compounds in the marine environment (paragraph 7.9.5).

20. Dr Thibaud will ask a French colleague to prepare an overview paper on alkyl-tin compounds in the marine environment (paragraph 7.9.6).

21. All members should obtain copies of papers C.M.1982/E:33, E:34, E:35 E:36, E:37, E:38, and E:39 and read them prior to the 1984 Working Group meeting (paragraphs 13.4 - 13.6).

22. Drs Uthe, Topping, Farrington and Berman should prepare a paper for the 1983 Statutory Meeting on the implementation of C.Res.1982/4:3, regarding the role of the new Intercalibration Coordination Center.

23. Dr Berman should prepare a list of international standards appropriate to the measurement of contaminants in sea water, biological tissues, and sediments.

ANNEX 6

RECOMMENDATIONS

The Marine Chemistry Working Group recommends that the next meeting of the Group be held for four days at the end of February 1984 with the following terms of reference:

1) to review the results of the following intercalibration exercises:

   a) the Fifth Intercomparison Exercise on PCBs in Biota (5/OC/BT),

   b) the Fifth Round Intercalibration for Trace Metals in Sea Water (5/TM/SW);

2) to review the status of the intercomparison exercises on petroleum hydrocarbons (2/HC/BT) and polycyclic aromatic hydrocarbons (3/HC/BT) in biological tissue;

3) to review the overviews on atmospheric deposition of substances to the marine environment and on the status of sampling and analysis of sea water for trace metals;

4) to re-examine the list of contaminants to be included in the baseline studies on contaminants in fish and shellfish and on trace metals in sea water; and

5) to consider the overview on CO_2 cycling in the oceans and other relevant information.

The meeting should be arranged so that a one-day joint meeting may be held with the Working Group on Marine Sediments in Relation to Pollution.

The Environment Officer should take part in this meeting.

Palmark har vært med i denne gruppen