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# REPORT OF THE FIRST JOINT SESSION BETWEEN THE MARINE CHEMISTRY WORKING GROUP AND THE WORKING GROUP ON MARINE SEDIMENTS IN RELATION TO POLLUTION

Rostock-Warnemünde, 22 February 1984



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## 1. WELCOME AND INTRODUCTIONS

1.1 The meeting was opened at 9.30 hrs on 22 February 1984 by the Co-Chairmen, Dr. J.M. Bewers, Chairman of the Marine Chemistry Working Group, and Dr. H.L. Windom, Chairman of the Working Group on Marine Sediments in Relation to Pollution. The meeting was held in Rostock-Warnemünde, German Democratic Republic, and was hosted by the Institut für Meereskunde of the Academy of Sciences of the German Democratic Republic.

## 2. ADOPTION OF AGENDA

2.1 The draft agenda was adopted as proposed with the addition of a new item 10 on "Future practice for joint meetings of these groups." The agenda is attached as Annex 1. The list of participants is attached as Annex 2. The ICES Environment Officer, Dr. J. Pawlak, served as Rapporteur.

## 3. REPORT OF THE INORGANIC (TRACE METAL) SUB-GROUP OF MCWG

3.1 Dr. G. Topping, Chairman of the Trace Metal Sub-Group of the Marine Chemistry Working Group, reported on the progress in the work of his Sub-Group during the past two days of meetings of the MCWG.

3.2 The Sub-Group had been requested to devise objectives and a strategy for future ICES quality control activities. The Trace Metals Sub-Group had agreed that there are three aims for studying trace metal concentrations in the marine environment:

1.) the protection of public health,

2.) determination of effects on marine life, and

3.) estimation of biogeochemical cycles.

In accordance with these aims, the Sub-Group had agreed that it was necessary to

a.) maintain the quality of data on well-studied contaminants using existing analytical techniques,

b.) develop techniques for "new" contaminants, and

c.) develop techniques for determining mass balances, inputs, etc.

3.3 The Trace Metal Sub-Group had also assessed the need for giving attention to new marine compartments in future quality control activities and studies of the marine environment. The Sub-Group noted that current ICES monitoring

activities are conducted only in relation to the analysis of contaminants in fish and shellfish. It was recognized at the beginning of this work that studies must also be done on contaminants in sea water and sediments. To this end, the Marine Chemistry Working Group has worked on the development of techniques for the measurement of trace metals in sea water for the past eight years and it will be possible, starting in 1985, to commence co-ordinated studies of trace metal concentrations in the dissolved phase in sea water, at least by a few selected laboratories. The Sub-Group felt that work should now begin on the measurement of trace metals in suspended particulate matter, starting with an intercomparison exercise on the techniques for analyzing particulate matter. The trace metals covered in these studies are: Hg, Cd, Cu, Zn, Pb plus Ni and possibly As.

3.4 The Sub-Group had considered several requests for information submitted by the Oslo and Paris Commissions and assessed the status of its work on these subjects.

3.5 In terms of the preparation of leaflets on Techniques in Marine Chemistry, the Sub-Group had agreed to produce guidelines on sampling, pre-treatment, and storage of sea water samples prior to analysis for trace metals. Leaflets will also be prepared on the analytical techniques which are shown to give the best results by the Seventh Intercalibration Exercise on the Analysis of Trace Metals in Biota (7/TM/BT).

3.6 The Sub-Group had agreed that intercalibration exercises on the analysis of trace metals in various matrices should be carried out every two or three years, depending on the need. During the time between these periodic intercalibration exercises, quality control in individual laboratories should be maintained by the use of reference materials (standard materials for which the values have been certified and are accepted as good estimates of the true concentrations). Research materials, especially "uncompromised" samples which contain undisclosed concentrations of contaminants known only to the originator of the samples, should be used for the intercalibration exercises.

4. REPORT OF THE NUTRIENT SUB-GROUP OF MCWG

4.1 Dr. D. Nehring, Chairman of the Sub-Group on Nutrients and Carbonate Species of the Marine Chemistry Working Group, provided an overview of the subjects discussed by his Sub-Group. In defining its area of coverage, the Sub-Group had noted that phosphorus, nitrogen and silicate compounds, both inorganic and organic, are essential nutrients for primary production; other nutrients include, e.g., the carbonate species. Intercalibration exercises on the analysis of nutrients have shown good results when performed using mixed sea water samples, but intercalibrations performed under field conditions have yielded less satisfactory results owing to sampling problems and/or inhomogeneities in the nutrient distribution in the water mass. The Sub-Group has proposed that a trial intercalibration on determinations of alkalinity and calcium be carried out as a joint exercise between ICES and SCOR Working Group 75 (Oceanic $CO_2$ monitoring).

4.2 In considering whether measurements of nutrients should be included in a Baseline Study of Sea Water, the Sub-Group had felt that there was no need to include nutrients in the Baseline Study because measurements of nutrients are already included in a number of oceanographic programmes.

4.3 In the discussion of this Sub-Group report, it was pointed out that investigations are needed on $pCO_2$ in the atmosphere and ocean, and on the carbonate phase and alkalinity system in the ocean at all times of the year because there is a need to know the seasonal variations in these parameters and especially the conditions during the winter. Such studies are particularly important in areas of deep water formation.

4.4 Several members questioned the assessment by the Sub-Group that there is no need for additional studies of nutrients, citing the fact that only one or two institutes had obtained data in connection with the North Sea Nutrient Study (C.Res.1978/4:2) thus precluding the possibility of carrying out an overall assessment of the results of this study.

5. INTERCALIBRATION EXERCISE ON TRACE METALS IN MARINE SEDIMENTS (1/TM/MS)

5.1 Dr. D. Loring, Coordinator of the Intercalibration Exercise on the Analysis of Trace Metals in Marine Sediments (1/TM/MS), reported that at last year's WGMS meeting, it had been agreed that an intercalibration exercise should be conducted to test various acid digestion methods used in the determination of trace metals in sediments. Accordingly, an intercalibration exercise had been devised to test the following methods:

1.) determination of total metal concentrations, after digestion with hydrofluoric acid (HF),

2.) determination of partial metal concentrations, after digestion with $HNO_3$ and/or $HNO_3$/HCl.

3.) determination of "weakly bound" metal concentrations, (a) after extraction using 25% acetic acid, and (b) after extraction using 1N HCl.

Dr. Loring has also requested participants to carry out a total extraction on the residues of the samples used for weak acid extraction so that the total metal concentrations in these samples can also be calculated. Four replicate analyses of each sample should be carried out. Dr. Loring stated that samples would be distributed on 1 March 1984 to persons who had requested them.

5.2 In the discussion of the plans for this intercalibration exercise, the request was made that measurements of carbon also be included in the intercalibration, at least on one sample. Additionally, it was requested that participants be allowed to use their own methods in addition to the required methods, as an option if so desired. Finally, it was pointed out that the method for the determination of mercury given in the instructions for the exercise would be difficult for many laboratories to apply.

5.3 In response to these requests, Dr. Loring agreed to ask participants to carry out an analysis for carbon on at least one sample and to state that participants can use their own methods of acid digestion as an option in addition to using the required methods. He also agreed to look into the possibility of changing the method for mercury determinations to a simpler one.

5.4 In closing the discussion on this intercalibration exercise, the joint meeting expressed appreciation to Dr. Loring for undertaking the coordination of this exercise; this is an enormous undertaking with a great deal of work and represents a service not only to the ICES community but to the marine community in general.

## 6. JMG INTERCALIBRATION EXERCISE ON SIEVING METHODS FOR MARINE SEDIMENTS

6.1 In the absence of the Coordinator of this intercalibration exercise, Dr. M. Joanny, it was noted that intercalibration samples had been sent to 41 laboratories and results have only been received from 14 of these so far. The WGMS looked forward to reviewing the results of this intercalibration exercise at its 1985 meeting.

## 7. REPORT OF THE ORGANIC SUB-GROUP OF MCWG

7.1 Dr. M. Ehrhardt, Chairman of the Sub-Group on Trace Organics of the Marine Chemistry Working Group, reported on the discussions taking place within his Sub-Group. In terms of measurement of petroleum hydrocarbons in sea water, the Sub-Group had agreed that the use of the UV fluorescence (UVF) method had been successfully intercalibrated at the Kiel Intercalibration Workshop in 1981, so it was possible to use this method to measure petroleum hydrocarbons in sea water in connection with the Baseline Study, as well as other investigations. The initial use of the UVF method in an area should be accompanied by a detailed analysis of the individual compounds this technique is measuring. As a good correlation has been found between the results using UVF and a more detailed analysis, the UVF method can be used on a general basis with only occasional checks as to the specific compounds being measured.

7.2 Of the two main systems for detailed analysis of petroleum hydrocarbons, namely, GC/MS with electron ion detector and HPLC with fluorescence detection, the Sub-Group agreed that GS/MS was the better system both because better results are obtained and because this equipment can be used to measure other types of organic compounds. The Sub-Group had also recommended that, given the high costs for collecting marine samples using research vessels, laboratories should attempt to obtain these sophisticated analytical systems when possible in order to gain the most information from their samples.

7.3 The Sub-Group noted that while petroleum hydrocarbons are presently mainly determined in sea water, it could be useful to consider the measurement of these compounds in surface sediments and suspended particulate matter in the future. In this connection, two sediment reference materials for petroleum hydrocarbons will be available from the National Research Council of Canada later in 1984.

7.4 Dr. Ehrhardt reported that his Sub-Group had discussed the measurement of chlorinated hydrocarbons, such as PCBs, and had agreed that the use of capillary column gas chromatography with electron capture detection was the preferred method. While the use of packed columns was not entirely discouraged, laboratories were encouraged to obtain a capillary column system whenever possible. The Sub-Group encouraged the study of chlorinated hydrocarbons in sediments and suspended particulate matter. In terms of the study of such chemicals as chlorinated dibenzodioxins and chlorinated dibenzofurans, the Sub-Group decided that the methodology for determining these compounds was not sufficiently developed to warrant coordinated work, but individual laboratories were encouraged to work on the development of their own techniques. The situation is similar in terms of the analysis of phenols and chlorinated phenols and plasticizers.

7.5 In discussing the problems of PCB analysis brought out by the Sub-Group report, it was noted that work is being carried out by the Bureau of Community References of the European Community to examine in detail the problems associated with the various steps in PCB measurements. The results of this work will be forwarded to ICES for further application appropriate to ICES programmes. Despite the problems of analysis of PCBs, however, it was felt that baseline and monitoring studies of PCBs in marine biota should be continued, realizing that data from different laboratories may not be comparable.

## 8. INTERCALIBRATION EXERCISE ON BALTIC SEA SEDIMENTS

8.1 Dr. L. Brügmann, Coordinator of the Intercalibration Exercise on the Analysis of Baltic Sea Sediments, provided a preliminary report on the results of the first part of this exercise. This intercalibration is being conducted in preparation for a Pilot Study of Sediments in the Baltic Sea in relation to pollution, coordinated by the ICES/SCOR Working Group on the Study of the Pollution of the Baltic. Dr. Brügmann stated that the intercalibration exercise has been divided into two parts, with the first part comprising the distribution of two dried, homogenized samples of muddy sediment, one sample from the Arkona Basin and the other from the Mecklenberg Bight. These samples were distributed to 67 laboratories, who were requested to analyze them for inorganic and organic carbon and a suite of major and trace metals. While the number of laboratories returning results had been low to date, Dr. Brügmann reported that the results received had been fairly good for Zn, Cu, Pb, Mn, Fe, Ni and organic carbon; the results for Cd, Hg and inorganic carbon had not been good.

8.2 Dr. Brügmann stated that the second part of the intercalibration exercise involves the analysis of sediment cores to determine pollution history. The samples consist of 20-cm cores sliced into 1-cm slices; participants have been requested to conduct dating analyses on every second slice and trace metal analyses on the remaining slices. Seventeen core samples have been distributed so far, but many samples are still available.

8.3 The Joint Meeting noted this information with interest and expressed its appreciation to Dr. Brügmann and his Institute for the substantial amount of work which has been carried out in the coordination and conduct of this intercalibration exercise.

## 9. ANALYSIS OF ORGANIC COMPOUNDS IN SEDIMENTS

### 9.1 Procedures of Sediment Analyses for Organics

9.1.1 It was noted that the situation regarding analysis of petroleum hydrocarbons in sediments is better than that for analysis of PCBs and other organochlorines. A number of laboratories are engaged in the study of petroleum hydrocarbons in sediments and an intercalibration exercise on the analysis of PHCs in marine sediments (1/HC/MS) coordinated under ICES in 1980 showed fairly good results for the analysis of overall PHCs (cf. Coop. Res. Rep. No. 117 (1982)). Reference methods for the analysis of PHCs in sediments have been published by IOC.

9.1.2 In terms of the analysis of PCBs in sediments, Dr. M. Marchand reported that he has studied PCBs in French coastal sediments and has found PCBs in concentrations ranging from 1 ppb to 1 ppm, with some sediments at levels below the detection limit. He stated that a French intercalibration exercise on analyses of organochlorines in sediments had resulted in a coefficient of variation of 40-60% for analyses of PCBs, which was better than that for the analysis of DDT. Dr. M. Perttilä reported that Finnish studies of PCBs in sediments in the Baltic Sea have shown a wide range in concentrations on a geographical basis and also variations in PCB concentrations in vertical sections of cores. He stated that it is important to study PCBs in dated cores so that concentrations can be related to the age of the sediments.

9.1.3 Having carried out a general overview of this topic, the Joint Session decided that a small sub-group should consider this subject in greater depth and, in particular, should decide which compounds can adequately be measured in sediments and whether we are in a position to develop an intercalibration exercise to compare the results of analysis among laboratories. This sub-group was thereafter set up and the results of its deliberations are contained in the following section.

9.2 Planning for a Future Intercalibration Exercise for Organics in Sediments

9.2.1 As mentioned in the previous paragraph, a sub-group to consider the development of an intercalibration exercise on organic compounds in sediments was set up, with the following members:

R. Law (Chairman), J. Calder (Rapporteur), M. Ehrhardt, M. Kerkhoff, M. Marchand, K. Palmork, M. Perttilä, L. Reutergårdh, J. Skei. The questions it was requested to handle are as follows:

1. Which organic compounds are presently being measured in sediments, and how comparable are the data from these studies?

2. Would the comparability of data from a sediment monitoring programme be improved by participation in a suitable intercalibration exercise and, if so, what form should this exercise take?

3. Would it be practical to conduct such an exercise in the near future and, if so, could a timetable be drawn up?

9.2.2 The organic compounds which the sub-group identified as currently being determined in sediments within various programmes were:

organochlorine compounds and PCBs

PCDDs, PCDFs and chlorinated butadienes

PAHs

specific aliphatic hydrocarbons

total PAHs by fluorescence spectroscopy

total aliphatic and total aromatic hydrocarbons by gravimetry or gas chromatography

chlorophenols

humic and fulvic acids

total organic carbon

coprostanol

9.2.3 Of these, total PAH by fluorescence spectroscopy has been well intercalibrated by ICES using a dried sediment sample. The other determinands have not been sufficiently well intercalibrated to date. Sampling variations will also be important, and these have not yet been studied.

9.2.4 It is unlikely that an intercalibration exercise for organochlorine compounds and PCBs in sediment could be successfully carried out before satisfactory improvement has been achieved for analysis of these compounds in biological tissue. Considerable effort is already being put into solving this problem by members of MCWG, and it is not desirable to add to their workload and dilute that effort this year.

9.2.5 During 1984, an intercalibration exercise for hydrocarbons will be carried out. Sediment samples will be analyzed for total aliphatic and total aromatic hydrocarbons, specific aliphatic hydrocarbons and PAH. The exercise will be organized by the Oil Pollution Research Unit, Wales, and it is intended primarily for laboratories belonging to central government, oil companies and contract analysis companies in the UK, who have carried out surveys around North Sea oil fields. The MAFF Fisheries Laboratory at Burnham-on-Crouch will be participating, and Dr. Law will contact the coordinator regarding possible further involvement of ICES laboratories. If this is possible, Dr. Law will circulate information to ICES laboratories who may be interested. Dr. Law will also endeavour to present the results of this exercise to the MCWG at its 1985 meeting; it may serve as a model for any future exercise to be undertaken by ICES.

9.2.6 The sub-group felt that it was premature to carry out an intercalibration exercise for organic compounds in sediments, although in principle it was felt to be feasible. The sub-group would like to reconsider this question in 1985 when information from ongoing intercalibration exercises will be available.

9.2.7 The Joint Session took note of the above report at a brief session on the morning of Thursday, 23 February, and endorsed it.

9.2.8 Dr. S. Berman pointed out that relevant sediment reference materials are available from the National Research Council of Canada. Three sediment reference materials have been issued earlier which are certified for total PCB concentration and two of these have recently been certified for ten PCB congeners. In addition, three sediment reference materials are being prepared, with certification for a total of 15 to 16 individual PAHs.

## 10. FUTURE PRACTICE FOR JOINT MEETINGS OF THESE GROUPS

10.1 The Joint Session discussed the issue of whether the MCWG and the WGMS should meet in joint sessions or in back-to-back meetings on a regular basis. While it was agreed that such joint meetings can be useful, both Working Groups decided that they should meet separately but hold a joint session occasionally, when the topics to be discussed warranted it.

## 11. ANY OTHER BUSINESS

11.1 The Joint Session then discussed the topic of studies of suspended particulate matter in terms of which of the two Working Groups should consider suspended matter and how the work should be divided. It was agreed that each Working Group had an interest in suspended particulate matter (SPM) and looked at SMP from a different standpoint. Accordingly, each Group should be encouraged to carry out work on this subject, but major duplications in work, especially in terms of intercalibration exercises, should be avoided.

11.2 The Joint Session felt that it could be useful to devise an intercalibration exercise on the analysis of trace metals in SPM and that this exercise should be a joint project between WGMS and MCWG. Accordingly, the Chairmen of both Working Groups agreed to work together to develop plans for future work on the analysis of trace metals in suspended particulate matter.

NOTE: On the basis of this discussion, a small group consisting of two members of MCWG and two members of WGMS met on the evening of 23 February to discuss specific activities. The results of this discussion and the consideration of the issue by WGMS are contained in paragraphs 9.1 to 9.6 of the report of the WGMS meeting, Doc. ICES C.M.1984/E:2.

11.3 As all business was completed, the Co-Chairmen thanked the participants for their contributions and closed the meeting.

ANNEX 1

JOINT SESSION BETWEEN THE MARINE CHEMISTRY WORKING GROUP AND THE WORKING GROUP ON MARINE SEDIMENTS IN RELATION TO POLLUTION

Rostock-Warnemünde, 22 February 1984

A G E N D A

1. Welcome and Introductions
2. Adoption of Agenda
3. Report of the Inorganic (Trace Metal) Sub-Group of MCWG
4. Report of the Nutrient Sub-Group of MCWG
5. Intercalibration Exercise on Trace Metals in Marine Sediments (1/TM/MS)
6. JMG Intercalibration Exercise on Sieving Methods for Marine Sediments
7. Report of the Organic Sub-Group of MCWG
8. Intercalibration Exercise on Baltic Sea Sediments
9. Analysis of Organic Compounds in Sediments
   - 9.1 Procedure of Sediment Analyses for Organics
   - 9.2 Planning for a Future Intercalibration Exercise for Organics in Sediments
10. Future Practice for Joint Meetings of These Groups
11. Any Other Business

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ANNEX 2

JOINT SESSION BETWEEN THE MARINE CHEMISTRY WORKING GROUP AND THE WORKING GROUP ON MARINE SEDIMENTS IN RELATION TO POLLUTION

Rostock-Warnemünde, 22 February 1984

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