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REPORT OF THE MARINE CHEMISTRY WORKING GROUP

Tenerife, Canary Islands, Spain, 9-14 March 1992

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EXECUTIVE SUMMARY

The ICES Marine Chemistry Working Group (MCWG) met in Tenerife on 9 to 14 March 1992. The meeting was hosted by Dr J. de Armas, and attended by 33 people.

This summary is confined to the tasks allotted to the Marine Chemistry Working Group by ACMP, and to major items raised by the Group itself.

Stage 3a of the CB intercomparison exercise has been completed. Data on two certified congeners, CB 52 and CB 153, and a non-certified congener, CB 156, were requested. 45 out of 58 laboratories submitted data. Compared to Stage 2 of the exercise, the between-laboratory variance had considerably improved for CB 52 and CB 153. The results were much better for the two certified congeners than for CB 156. Further analyses of the data, after the MCWG meeting, showed that the variance was caused by poor GC separation. Once this had been accounted for, the data for CB 156 had a similar variance to that of CB 52 and 153.

The planning for Stage 3b of the CB intercomparison exercise was completed. This stage will include a standard solution with unknown concentrations, a cleaned and uncleaned sediment extract, and a cleaned and uncleaned seal blubber extract. Participants will have to purchase the samples from the organizing laboratory. It is recommended that J. de Boer continue as coordinator.

Phase 2 of the ICES Fourth Round Hydrocarbon Intercomparison Exercise could not be carried out by R. Law in 1991. This phase will be conducted later in 1992, coordinated jointly by W. Cofino, F. Smedes and R. Law. A cleaned sediment extract and a standard solution will be distributed. The samples will have to be purchased.

The ACMP had requested that the overviews on chromium, nickel, atrazine and brominated flame retardants in the marine environment be completed. The MCWG recommended last year that the overview on chromium be included in an annex to the ACMP report as an overview of chromium in sea water. No further work was done on this overview. A revised document on nickel was reviewed. This paper needed some improvement and requires intersessional work. Overviews on atrazine and brominated flame retardants were reviewed. The paper on brominated flame retardants is, with minor revisions, ready for publication. It will be sent to ACMP, along with a summary. The paper on atrazine will probably be published in the open literature next year. In 1993, the MCWG will receive a copy together with an executive summary.

The procedure used by the JMG Ad Hoc Working Group on Monitoring in December 1991 to assess the quality of the data from the 1990 Supplemental Baseline Study of Contaminants in Fish and Shellfish was discussed. The MCWG fully supports the procedure used. The recommendations of the Ad Hoc Group (Annex 4 of their report) are also strongly supported. The MCWG views with regret that these recommendations are very close to those made by the MCWG after the 1985 Baseline Study, and have to be made yet again.

The paper on the design and execution of intercomparison exercises was reviewed and expanded. It is included as Annex 4 to this report.

Progress on the handling and storage of sea water for nutrient determinations was reviewed. The papers received had a very diverse nature. The handling of samples generally centred on filtration. Filtration is, when necessary, perfectly acceptable so long as systematic steps are taken to check contamination and to eliminate or minimize contaminating influences. Filtration is expected to improve the quality of data if storage is contemplated. It was suggested that the inconclusive nature of some past experiments on storage stability may be attributable to inadequate quality control in the calibration procedures.

Data on lindane in sea water were assessed for the NSTF-MMP. Dr Gaul had prepared a draft assessment of the concentrations of HCH isomers in North Sea water. In general, the NSTF-MMP data corresponded well with the data obtained by the German BSH. Dr Gaul agreed to produce a paper including the BSH and NSTF-MMP data sets for submission to the ACMP.

The plans for the ICES NUTS 5 Intercomparison Exercise were reviewed. The maximum number of participants (about 110) has been reached. Samples will be ready for distribution in the end of 1992. It is recommended that IFREMER be reimbursed for the costs of sample bottles and distribution. These costs are estimated to be 60,000 DKK.

The results of the Visby intercomparison exercise were discussed. The group agrees with the organisers that for oxygen, more attention should be paid to the purely analytical procedure, as here the possibility of systematic errors is most evident. It is recommended that the use of cadmium be avoided as a precipitating agent in the stabilization process for the determination of H_2S. Zinc is recommended as an alternative. The nutrient exercise might have benefited from a preliminary laboratory intercomparison stage. Some points of attention for nutrients are given in the report.

The EC QUASIMEME programme was discussed extensively. The MCWG recognises this unique oppor-

tunity to develop a working quality assurance programme for the European marine laboratories which is initiated by this QUASIMEME programme. The MCWG is fully positive regarding this programme, and welcomes the fact that this programme has the full support of ICES. However, there was a general feeling of concern in the group that the QUASIMEME programme may further isolate the USA and Canada from the European member countries of ICES. Presently, JMG and the NSTF activities have already led to some isolation. The group hoped that the QUASIMEME programme would not increase this sense of isolation. In addition, the present working relationship between the EC-BCR and ICES gave particular reason for concern. The MCWG instructs its Chairman, as its representative on the Steering Group of QUASIMEME, to ensure, as far as possible, that the interests of the non-EC member countries of ICES be considered in planning the activities of QUASIMEME.

Dr Uwe Harms presented the paper, "Assuring the Quality of Analytical Data from Monitoring Programmes in the Marine Environment: A Proposal". It was primarily directed towards the quality assurance programme for the future Baltic Monitoring Programmes. The Group strongly supported the concept described in the proposal.

Drs Tronczynski, Wells and de Boer presented papers on the interactions between dissolved organic matter and contaminants in relation to the transport of organics associated with particulates at a sewage sludge dumping site and non- and mono-ortho substituted chlorobiphenyls in fish and marine mammals. These papers gave rise to considerable discussion on cooperative research within the MCWG. It was agreed that, for selected topics, speakers would be invited to next year's MCWG meeting. The topics suggested include the interaction between contaminants and dissolved organic matter, the distribution in the aquatic environment of planar compounds, bioavailability, and statistical aspects of trend monitoring from an analytical point of view. All members are given the opportunity to contribute by organizing a poster session.

1 OPENING OF THE MEETING

The Chairman, Dr W. Cofino, opened the meeting at 9.45 hrs on 9 March 1992 and welcomed the participants. He thanked Dr D. de Armas for the kind invitation, who then welcomed the group on behalf of the Instituto Español de Oceanografia.

The Working Group members introduced themselves and briefly described their main areas of research interests and responsibilities in the field of marine chemistry. The list of participants is given in Annex 1.

The Chairman informed the group that Drs Ehrhardt, Reutergårdh and Yeats had written to him that they could not attend the meeting. The absence of Dr Yeats implied that the Trace Metal Sub-group had to appoint a chairman for this meeting.

2 ADOPTION OF THE AGENDA

The Working Group reviewed the draft and annotated agenda, which had been prepared and distributed by the Chairman before the meeting.

As usual, most of the tasks would be dealt with by the respective Sub-groups prior to being discussed in plenary. Sub-group sessions began in the afternoon of the first day, and were to be completed by the end of the fourth day. Each morning a short plenary session was planned. The reports from each Sub-group were to be discussed in plenary on Friday, 13 March. The reports would include any recommendations and action lists for the forthcoming intersessional period.

The agenda was amended slightly. Drs Ólaffson and Auounson had sent a letter to the Chairman drawing attention to the need for a better understanding of the relationship between the lipid content and trace metal concentrations in biota. This topic was added to the agenda of the Trace Metal Sub-group. Dr Harms had submitted a paper entitled "Assuring the quality of analytical data from monitoring programmes in the marine environment: A proposal" for consideration by the MCWG. Dr Harms is convener of the Project on Chemical Quality Assurance of the HELCOM Environment Committee. He prepared this paper with the intention of promoting the discussion on the establishment of a Quality Assurance Programme for the Contracting Parties of the HELCOM Convention. Dr Cofino proposed to organize a plenary session on quality assurance matters, discussing the QUASIMEME project and the paper of Dr Harms. This proposal was accepted. Dr Carlberg proposed a number of modifications to the agenda of the Chemical Oceanography Sub-group, which were all adopted.

The modified agenda is given in Annex 2.

The sessional chairmen for the Sub-groups on Organics and Chemical Oceanography would be, respectively, D. Wells and S. Carlberg. The Trace Metal Sub-group had to elect a chairman. The remaining members and visitors were grouped as follows:

Chemical Oceanography:
A. Aminot, J. Escanez, L. Føyn, D.S. Kirkwood, K. Mäkelä, O. Vagn Olsen, W. de Waal.

Organics:
A. Abarnou, J. de Boer, J. Biscaya, J. Boon, M. Cleemann, S. Einarsson, H. Gaul, B. Jansson, J. Klungsøyr, R. Law, E. Nixon, T. Nunes, P. Roose, F. Smedes, J. Tronczynski.

Trace Metals:
D. de Armas, G. Asmund, S. Berman, V. Besada Montenegro, L. Brügmann, U. Harms, M. Leivuori, B. Pedersen, S. Wilson.

3 REPORT OF THE 79TH STATUTORY MEETING

Relevant parts of the report of the 79th Statutory Meeting had been distributed with the agenda. The contents were in line with the report of the MCWG.

Dr Cofino drew attention to the recommendation in the 1991 MCWG report in which it was proposed "that ICES establishes as policy that overviews are to be published in the open literature, and that ICES finds a mechanism whereby overviews can be published in the ICES Journal of Marine Science (JMS)." This recommendation has not been adopted by the ACMP. Following the advice of Dr Topping, Chairman of the ACMP, Dr Cofino wrote to Prof Blaxter, editor of the ICES JMS, about this matter. Prof Blaxter replied that the ICES JMS is most willing to accept overviews for publication. The papers have to be reviewed according to normal procedures. Dr Cofino concluded that it is up to the MCWG itself to formulate a policy with regard to overviews. He proposed that this policy should entail that overviews ought to be published in a journal selected by the author(s). Assessment of the papers by the MCWG would take place using criteria also employed by journals. Publication in the ICES JMS is strongly recommended. Dr Cofino requested the sub-groups to discuss this matter.

The Chairman informed the Group that all of the tasks requested at the Statutory Meeting for attention by MCWG had been incorporated in the draft agenda.

4 REPORT OF RELATED ACTIVITIES

4.1. Joint Monitoring Group of OSPARCOM

No note concerning JMG matters was available at the meeting; requests from JMG have been included in the draft agenda.

4.2. Intergovernmental Oceanographic Commission (IOC)

No note concerning IOC activities was available at the meeting, nor was an IOC representative present.

4.3. ICES Working Groups

Dr Carlberg drew attention to a successful joint meeting of the WG on Shelf Seas Oceanography and members of the Chemical Oceanography Sub-group of the MCWG. The outcome of this meeting was to be discussed in more detail in the Sub-group meeting.

4.4. EC-BCR QA Pilot Project "QUASIMEME"

Dr D. Wells presented the proposal for the BCR QUASIMEME Programme. An outline of this programme was first presented to the MCWG during its meeting in Copenhagen in 1990, but the beginning of the programme was held up due to some financial restrictions within BCR. It is the intention to start the programme in May 1992 after a positive decision of the Council of Ministers. The proposed QA steering group met in January 1992. Several members of the MCWG belong to this group.

The pilot project will commence with an initial one-year proficiency exercise focusing on CBs in fish oil, trace metals in sediment, and nutrients in sea water. The programme for the next three years will be elaborated in March 1993.

The QUASIMEME programme consists of a management programme, an operational programme and a communication programme. Standards and reference materials for the first year are presently being prepared. A list of participants will be finalised in May 1992. Laboratories will receive an invitation to participate in May/June. A questionnaire will be distributed in order to obtain information on the quality assurance procedures implemented in each laboratory. In June 1992 a workshop for all participants will be held in Brussels.

The programme was extensively discussed by MCWG. There were several questions regarding the possibilities of participation by non-EC member countries. Dr Wells stressed that the QUASIMEME programme was primarily for laboratories from EC countries taking part in marine monitoring programmes. Officially, non-EC laboratories may receive information on the programme, receive materials, and submit data, but travel costs for attending meetings cannot be refunded. However, beyond the official scheme, several ways may be found to include non-EC countries in the QUASIMEME programme. It was agreed that Dr L. Brügmann, who acts as a coordinator for the Baltic countries, would provide Dr Wells with a list of laboratories in non-EC Baltic Sea states which should be involved in the programme as much as possible. This list will include the fields of interest.

Dr Cofino will provide Drs Brügmann and Pedersen information on the EC-PHARE programme. This programme may provide opportunities to obtain finances so that non-EC/EFTA countries can take part.

Dr Wells informed MCWG that all requests relating to participation and other questions concerning QUASIMEME should be sent to him.

The MCWG recognises this unique opportunity to develop a working quality assurance programme for the European marine laboratories which is initiated by this QUASIMEME programme. The MCWG is fully positive regarding this programme and it welcomes the fact that this programme has the full support of ICES.

However, there was a general feeling of concern in the group that the QUASIMEME programme may further isolate the USA and Canada from the European member countries of ICES. Presently, JMG and the NSTF activities have already led to some isolation. The group hoped that the QUASIMEME programme would not increase this sense of isolation. In addition, the present working relationship between the EC-BCR and ICES gave particular reason for concern. The MCWG instructed its Chairman, as its representative on the Steering Group of QUASIMEME, to ensure, as far as possible, that the interests of the non-EC members of ICES be considered in planning the activities of QUASIMEME. Copies of the transparencies presented by Dr Wells are included in Annex 3 of this report.

4.5. Other Activities

The Chairman stated that no matters had been raised by members under this agenda item.

5 REPORTS ON PROJECTS AND ACTIVITIES IN MEMBER COUNTRIES

The Chairman informed the Group that no matters had been raised by members under this agenda item.

6 REQUESTS FROM ACMP AND REGULATORY AGENCIES

The Chairman informed the Group that all requests had been incorporated into the agenda.

7 SUB-GROUP ACTIVITIES AND DISCUSSIONS

7.1 Trace Metal Sub-Group

Dr Berman was elected Chairman; Dr Pedersen agreed to act as rapporteur.

7.1.1 The overviews on chromium and nickel

No revised version of the chromium overview was received. Last year, the MCWG recommended that the overview should be sent to ACMP as a review on chromium in sea water only. The group sees no reason to modify this point of view, and instructs its Chairman to sort out this matter with Dr Topping, Chairman of the ACMP.

A new draft of the nickel overview was received from Mr Jones at the meeting. The group feels that there were many improvements made since the first version, but that there are still some shortcomings with the paper, e.g., some of the tables should include more recent data and the structure should be more firm.

The group also discussed (again) the large differences between writing a review about a well-described contaminant, such as Ni, and a "new" contaminant, where the amount of information is much more limited.

It was suggested that a review of a well-known contaminant should mainly include a guide (reference list) as to where to look, a description regarding concentrations found, the most recent pertinent information, and conclusions and recommendations.

It was agreed that Dr Brügmann would pass on comments to the author. It was the opinion of the group that this could be handled intersessionally.

7.1.2 Revision of Outline of Guidelines for the Conduct of Intercomparison Exercises

The outline of Guidelines for the Conduct of Intercomparison Exercises was discussed. The group felt that much information was to be found in the literature, and that there was no need for a very comprehensive document on this subject. The revised guidelines are given in Annex 4 of this report.

7.1.3 The procedure used by the JMG Ad Hoc Group in December 1991 to assess the quality of the 1990 baseline data

The Trace Metal Sub-group has examined the procedures used by the JMG Ad Hoc Working Group on Monitoring and supports the approach taken by the assessors in evaluating the data from the 1990 Supplementary Baseline Study of Contaminants in Fish and Shellfish. There is general improvement over earlier studies by the participants.

However, the Trace Metal Sub-group regrets that sufficient QA information was not submitted to the assessors in time to enable an easier and possibly more valid assessment.

In view of the difficulties encountered by the assessors, the Sub-group affirms its strong support of the recommendations of the *Ad Hoc* Working Group (AHWG) in December 1991, to the JMG (Annex 4). Also, ICES should ensure that all pertinent data are delivered to the assessors at least two months prior to the scheduled assessment meeting. Otherwise, it is recommended that the assessors do not participate in the assessment process.

The Sub-group especially emphasizes that the participating laboratories must submit, along with their data, sufficient QA information, to be determined by the coordinator, to allow the assessors to make valid decisions regarding the quality of the data. This includes, in addition to what has already been recommended by the AHWG, that the managers must ensure that participating laboratories will be supplied with reference materials whose metal concentrations are unknown to the participants, to be analysed along with the programme samples during the course of the exercise. These data must be submitted along with the sample data.

The Sub-group views with regret that the above recommendations are very close to those made by the MCWG after the 1985 Baseline Study on Contaminants in Fish and Shellfish.

7.1.4 Exploration of the estuarine data collected in the 1985-1987 ICES Baseline Study on Trace Metals in Sea Water

At the 1991 MCWG meeting, the Trace Metals Subgroup suggested, as an initiative for intersessional work, that the estuarine data, collected during the Baseline Study of Trace Metals in Coastal and Shelf Sea Waters, be further investigated in particular respect to trace metal/salinity relationships. It had been agreed that S. Wilson would distribute relevant data sets to some members of the sub-group for preliminary investigation. Unfortunately, due to other work priorities leading to delays in reorganising the sea water data within ICES, it had not been possible to prepare the requested data during the intersessional period. S. Wilson apologised for these delays and informed that data for the Western Scheldt had been compiled and were available for distribution or investigation during the meeting, if time allowed. He further informed that the other data sets could be made available shortly. The group agreed that

this project would form an ongoing task in the period prior to the 1993 MCWG meeting.

7.1.5 Review of "new" contaminants

The sub-group did not identify a contaminant for which reviews or overviews needed to be prepared.

7.1.6 Problems involving high lipid Materials

The Trace Metal Sub-group discussed problems associated with the determination of trace metals in materials containing high concentrations of lipids. It soon became evident that this was a possible area for collaborative research generated within laboratories of the Sub-group. Four possible problems were discussed:

(1) The need for a practical determination of "lipid weight" which would be relatively independent of procedure. This may turn out to be a more stringent requirement for trace metals than for trace organics.

(2) The need to understand the speciation of the metals in the fatty tissue, and also in the protein. The laboratories of Drs Berman, Harms and Pedersen are already involved with studying speciated metals. Uwe Harms agreed to attempt to outline a collaborative programme of research initially involving either alkyltins, methylmercury or organoarsenics.

(3) The problem of normalization of trace metal concentrations in lipids is a contentious one. Simon Wilson, Gerd Asmund and Britta Pedersen agreed to examine existing ICES data in order to ascertain whether there is sufficient information in the databases to warrant an intensive effort to determine correlations between various parameters relating to the trace metal and lipid concentrations. They will report intersessionally, recommending further action.

(4) Is there a problem in determining trace metals in fatty tissue vis-à-vis muscle tissue? It was felt that this issue might be answered in due course in the QUASIMEME Programme.

7.1.7 The paper on quality assurance in the framework of the Baltic Sea Monitoring Programme

Dr Uwe Harms presented the paper, "Assuring the Quality of Analytical Data from Monitoring Programmes in the Marine Environment: A Proposal". It was primarily directed towards the quality assurance programme for the future Baltic Monitoring Programmes.

The group strongly supported the concept described in the proposal. They also noted that the content was in line with similar recommendations made earlier by the group concerning this subject, and wished Dr Harms all the best with the project in the future.

7.1.8 Any business phoned and found

Dr Yeats was prepared to act intersessionally as Chairman of the Trace Metal Sub-group. Drs Berman and Cofino agreed to give him an update on the outcome of this meeting.

7.2 Organic Sub-group

7.2.1 Report on the Second Phase of the Intercomparison Programme for CBs, and make recommendations for Phase 3 of this exercise

Dr de Boer presented the results of Stage 3a of the exercise and pointed out the following, in particular:

- The deadline for sending in the results had been extended from 31 January to 29 February 1992, primarily because some US-based laboratories experienced difficulties in obtaining the necessary reference material on cod liver oil (BCR 349).

- The main aim of Stage 3a was to establish the long-term precision of the participating laboratories. For this purpose, the reference sample was analyzed 6 times with one-week intervals between the individual analyses. The results for three individual CB congeners had to be reported, as follows;

- CB52, representing a certified and usually well-separated congener;

- CB153, belonging to the same category;

- CB156, representing an uncertified congener which is more difficult to separate (CB202 and, especially, CB171 may co-elute on an SE-54 type column). This congener shows a "TCDD-type" mechanism of toxicity.

Participants had to analyze the samples on two columns (minimum length 50m and maximum internal diameter 0.25mm) with different stationary phases and could choose to report the most appropriate results.

45 out of 58 laboratories reported their results before the deadline. Ten laboratories did not submit any results, while three laboratories withdrew from the exercise. An overview of the results is given in Table 1. For comparison with a former stage of the exercise, the results for CBs 52 and 153 in the seal blubber extract used in Stage 2 are also given.

Table 1. Repeatability, reproducibility and their ratios of stage 3a and stage 2 (seal extract only) of the ICES/IOC/OSPARCOM I/C exercise on individual CBs in seal blubber and sediments.

CB No.	Repeatability (S_r%) (within laboratory)	Reproducibility (S_R%) (between laboratories)	S_r%/$S_{R\%}$
Stage 3a			
52	8.5	19	0.45
153	7.0	19	0.37
156	25.0	78	0.32
Stage 2 (Seal extract)			
52	18.0	37	0.49
153	7.0	30-35	0.20

The following conclusions may be made from Table 1:

- The results were much better for the two certified congeners than for CB 156.
- Compared to Stage 2 of the exercise, the reproducibility of the laboratories for the analysis of CBs 52 and 153 had improved considerably.
- Because analyses were carried out with one-week intervals, the contribution of the repeatability to the total variance increased from Stage 2 to Stage 3a for CB 153.

As an additional remark, Dr de Boer stated that laboratory No. 87 had reported values no higher than 50% of the target values. There was an uncertainty about the reason for these low values. A matrix effect on the sensitivity of the system was suggested as a possible cause. Such matrix effects may occur in spite of a straight baseline in an ECD-chromatogram.

More details about the results of this exercise are given in the (draft) report of Stage 3a.

The Chairman and the members of the Organic Subgroup thanked Dr de Boer for the extensive amount of work done and were very appreciative of the detailed report completed just prior to the meeting. Dr de Boer requested all members to read the draft report carefully, and give comments. It was agreed that the report on the results of Stage 3a should be recommended for publication in the ICES Cooperative Research Report series at a later stage, together with the results of Stage 3b.

The design of the following stage of the exercise was then discussed. The Chairman stated that, with the increasing amounts of time spent on intercomparison exercises organised by different national and international bodies, it was necessary to have a very efficient design for the next stage. At present, measurements for the JMG involve fish tissue, measurements for NSTF involve sediments, while those for ICES involve sediments, fish tissue and seal blubber.

After an extensive discussion, it was agreed that Stage 3b should involve the following samples:

- A standard solution with unknown concentrations;
- A cleaned and an uncleaned sediment extract;
- A cleaned and an uncleaned seal blubber extract.

The use of seal blubber was preferred to fish oil, because it has been a primary matrix of the exercise from the beginning, and because congener patterns and concentrations are significantly different from those of fish oil and sediment. The samples will be analyzed on two columns of different polarity for CBs 28, 31, 52, 101(84/90), 105(132), 118(123/149), 138(163), 153, 156(171/202), and 180 (possible co-elutants on SE-54/CPSil8 or similar column types are given in brackets following the primary determinands.

The participants in Stage 3b will be requested to purchase the samples from the organizing laboratory, presumably the Netherlands Institute for Fisheries Research (RIVO). Laboratories are free to choose either one or both environmental matrices, but must analyze the standard solution. It was agreed that participation in Stage 3b is only open to laboratories which have performed successfully in the previous stages of the exercise. Other laboratories that are involved in marine monitoring will be informed of other intercomparison exercises which will commence in 1992, such as the QUASIMEME programme of BCR.

Dr R.F. Addison (Canada) has offered to press seal blubber for the next stage (4) of the intercomparison exercise. It was suggested that a large amount of homogenate be prepared, so that after the exercise this can be used as a well-characterized reference material for this matrix until a certified reference material becomes available.

7.2.2 ICES Fourth Round Hydrocarbon Intercomparison Exercise

Mr R. Law presented this item and announced that no further progress had been made since MCWG 1991, and that it would not be possible for him to continue with the coordination of the exercise. A proposal was made that Dr W.P. Cofino, Mr F. Smedes, and Mr Law jointly take on the task of coordinating the second stage of this intercomparison exercise. This proposal was accepted by MCWG. The samples, a cleaned sediment extract and a standard solution, will be prepared by Dr Cofino,

assisted by Mr Smedes, and the report will be prepared by the joint co-ordinators. In the near future, the coordinators will notify the participants that the exercise will take place shortly, but that in order to cover the costs associated with sample preparation, the samples must be purchased at a cost of DFL 1500. The limited number of expected participants (17) is the reason for the relatively high cost. The second stage can take place on a short time scale. The concentrations of the PAHs in the standard (and sample) will be between 10 and 100 times lower than in the previous exercise, which will make the levels more realistic. It was suggested that the cleaned sediment extract could be prepared either from a certified reference material, or from a well-characterised material which might be available from NRC or BCR; thus more information on the levels of PAHs would be available. The coordinators will investigate these possibilities. The group thanked Mr Law for the work carried out during the first stage of this intercomparison exercise.

7.2.3 Assessment of the data on lindane (γ-HCH) in sea water for the NSTF MMP

Dr Gaul had prepared a draft assessment of the concentrations of HCH isomers in sea water in the North Sea. The usage of the insecticide γ-HCH (lindane) in EC countries currently amounts to ca. 3000 tonnes per annum. Environmental Quality Objectives (EQOs) have been set for lindane as:

fresh water	100 ng l^{-1}
estuaries	20 ng l^{-1}
coastal waters	10 ng l^{-1}

HCHs are widely distributed in marine waters, and the distribution pattern in the North Sea is the result of freshwater input, primarily in the German Bight, residual currents and water exchange. Concentrations of α- and β-HCH are low throughout the area. Concentrations of γ-HCH in coastal and offshore water are below the EQO, and no real trend in concentration was apparent off the Elbe river between 1982-1991, or in the Arkona Basin in the Baltic proper during the period 1975-1990. In addition to riverine inputs there is a contribution from atmospheric inputs, which give rise to a background concentration in open North Atlantic water of around 0.2 ng l^{-1}, and may contribute to the concentrations observed in coastal waters. All of these data are comparable, having been produced by a single laboratory using the same method.

A summary of the data collected under the NSTF MMP for γ-HCH in sea water was then circulated. Dr Gaul's data, as presented in the paper described above, will be submitted to the ICES databank in the near future. The data submitted to the MMP contained a maximum value of 107ngl^{-1}, close to the fresh water boundary in the Scheldt. Higher concentrations and strong gradients were observed in the estuaries sampled. Lower concentrations, similar to those obtained in the earlier studies, were seen in coastal and offshore areas. These anomalous values were noted:

(a) The high value off northeast Norfolk (9.4ngl^{-1}) was believed to be a mistake; Robin Law should confirm to ICES that 0.94 ng l^{-1} is the correct value.

(b) Two low values were submitted by FRUK; these were believed to result from an error in units of 1000x; FRUK has been approached with a view to checking these data.

Dr Gaul agreed to produce an updated text incorporating the MMP data (1985-1991) and to comment on the agreement among the many data. The text should include the caveats introduced by Dr Gaul during his presentation and the subsequent discussion, regarding the dangers of combining data from different laboratories and a number of years, given the year-to-year variability seen in the earlier data sets.

7.2.4 Review the methods used by ICES/JMG laboratories for the determination of lipids and, on the basis of this review, consider the need for an intercomparison exercise to assess the comparability of measurements

A lively discussion on this topic was held. The use of lipid concentrations for the normalization of data on organic contaminants was questioned. It was stressed that laboratories should provide data on a wet or dry weight basis, along with water and lipid contents, mentioning the method of the lipid determination.

An intercomparison exercise to assess the comparability of lipid determinations was deemed premature at present. Drs de Boer and Nixon agreed to prepare a paper on this subject. This paper should provide insight into the degree of variability which can be attributed to the use of different methods for lipid determinations, taking the lipid contents of the tissues into account.

7.2.5 Overviews on atrazine and brominated flame retardants

Dr de Boer and Dr Boon were asked to finalize the overview on brominated flame retardants, which was first presented during the MCWG meeting last year in Brussels. The authors have modified the paper according to the remarks that were made last year and have included, as was requested, a paragraph on analytical aspects and a section containing risk assessment and recommendations.

During the discussion, the Sub-group proposed small changes, which will be incorporated by the authors. The

general opinion of the Sub-group was, however, very positive and it was felt that the paper is ready for publication, bearing in mind the additional remarks of the WGBEC. Therefore, a copy of the draft will be sent to Dr Addison, Chairman of the WGBEC. It was stated that the authors should receive the final remarks by the end of May. The summary presented at the beginning of the overview will be submitted to the ACMP this year. Dr Jansson promised to send comments from his coworkers on the overview and to supply recent data on the subject from his laboratory.

Dr Tronczynski was asked to finalize his overview on atrazine in the estuarine environment. The author stated that he included the remarks that were made during the MCWG meeting last year and that he already had submitted the paper to the Chairman of the WGBEC. A paragraph on analytical methods was not included but, according to the author, it could be added in the form of an overview of the existing methods. The Sub-group suggested that it might be appropriate to present this overview in the form of a critical assessment. Apart from that, a number of additional remarks were made during the discussion, which will be included by the author.

Since the author expects the paper to be published in the open literature by next year, it was proposed that he present a copy of the paper at next year's MCWG meeting, together with an executive summary of the overview.

7.2.6 The procedure used by the JMG Ad Hoc Group on December 1991 to assess the quality of the 1990 baseline data

J. Klungsøyr informed the group about the assessment of the quality of the data submitted for the 1990 supplementary Baseline Study of Contaminants in Fish and Shellfish. The possibility of making a judgement about the quality of the data was quite limited because detailed information on quality control from the laboratories was lacking. Results from an ongoing intercomparison on the analysis of CBs (step 2) could be applied because 6 out of 7 laboratories submitting data on CBs had participated. Results from an intercomparison (from 1985) of other components were, however, considered too old to be representative. Only two laboratories had submitted data on the use of reference materials. Other information was not available at the meeting of the ad hoc group (QA information is requested by the JMG, but the deadline for submission is later). The evaluation was therefore based mainly on the data as submitted. Many of the data were rejected because of improper sample size and improper sampling time. The comparability of the results was not considered to be much better than for data submitted in the 1985 Baseline Study, which implied an accepted variance of a minimum of 30-40%.

The group fully supports the procedure used to assess the quality of the data. The quality of the information obtained from the monitoring was considered reasonable with regard to the information asked for. The data were not considered suitable to be used for temporal trends. The totally insufficient QA data supplied with the analytical results was due to insufficient guidelines for the submission of data. Forms for data submission should be reviewed for the inclusion of requests for specific QA data. A check should be included in monitoring programmes by sending a blind sample to the participating laboratories. Before a monitoring study is undertaken a framework should be set in place giving target values for the accuracy and precision required to meet the aims of the study.

7.2.7 Review of "new" contaminants

The Sub-group did not identify a contaminant for which reviews or overviews needed to be prepared. Dr Boon drew attention to a comprehensive review on toxaphene which appeared recently (M.A. Saleh, "Toxaphene: Chemistry, Biochemistry, Toxicity and Environmental Fate", Rev. Environ. Contam. Toxicol. 118 (1991), 1-85).

7.2.8 Any other business

7.2.8.1 Non- and mono-ortho substituted chlorobiphenyls in fish and marine mammals

Dr de Boer presented a paper entitled "Non- and mono-ortho substituted chlorobiphenyls in fish and marine mammals". This paper describes recent developments in the analysis of planar CBs and on toxicological knowledge of non- and mono-ortho substituted CBs. Due to the toxicological similarities of planar CBs to 2,3,7,8,-tetrachlorodibenzo-p-dioxin (TCDD), toxic effects can be expressed as a ratio to 2,3,7,8,-TCDD. Analytical methods for the determination of non-ortho CBs, which are found in low concentrations in PCB technical mixtures and environmental samples, are described. Extra separation by HPLC using porous graphitic carbon columns was performed for the separation of non-ortho substituted CBs 77, 126 and 169. Results of analyses of marine and freshwater fishes from the Netherlands and some marine mammals, along with the ratio of planar CBs to the metabolically stable CB 153, are presented. Total CB-TEQs (dioxin equivalents) in cod liver from all parts of the North Sea were above the Canadian tolerance level of 20 ng/kg. In this paper Dr de Boer poses a number of questions:

1) What are the uncertainties in TEFs (toxic equivalency factors)?

2) What are the TEFs, toxic properties, and synergistic and antagonistic effects of other CBs?

3) What are the consequences of the relatively high planar CB concentrations on marine organisms and for human consumption?

4) Which CBs should be analyzed in the future?

Dr Boon pointed out that it would be inadvisable to restrict monitoring to CBs with high TEFs, as many other CBs have neurotoxic effects, are tumor promoters, and produce cytochrome P450 responses.

It was felt that the CBs required to be analyzed in the next stage of the CB intercalibration will give good coverage of the various toxic groups of PCBs, with the exception of the planar CBs. Attention should be paid to the intercomparability of the results of planar CB analyses and to the difference in the use of the TEFs.

7.2.8.2 Future work plan of the Organic Sub-group

For the next meeting, it is proposed to evaluate the current practice of marine monitoring programmes under the headings:

1) Analytical capabilities of the participating laboratories;

2) The practice of pooled data from different laboratories;

3) Statistical aspects of trend monitoring from an analytical point of view.

It is proposed to invite Dr J. Uthe to participate in these discussions.

For the 1993 meeting, a number of papers will be submitted:

- Distribution in the aquatic environment of planar compounds - Dr D. Wells.
- Chlorinated naphthalenes - Dr B. Jansson.
- Contaminants and dissolved organic matter - Dr J. Tronczynski and Dr P. Yeats.
- Measurements of sediment-water distribution coefficients of PCBs excluding the influence of dissolved organic matter -Dr F. Smedes.

It was suggested that other members would be given the opportunity to present results by organizing a poster session.

Dr Wells was prepared to act intersessionally as Chairman of the Sub-group.

7.3 Chemical Oceanography Sub-group

7.3.1 (ACMP) Progress on the handling and storage of sea water for nutrient determination

Several contributions on this topic were received by D. Kirkwood, intersessionally and at the meeting. Individual papers were considered in some detail, but because of their diverse nature it proved difficult for the Sub-group to identify a unifying rationale. 'Handling' generally centred on the necessity or otherwise of filtration. The filtration step is widely recognised as a potential source of contamination, and is, of course, best avoided if possible. However, if filtration, for whatever reason, is considered necessary, it is perfectly acceptable so long as systematic steps are taken to check possible contamination and eliminate or minimise contaminating influences. There can be no hard-and-fast rules that apply to every situation; each individual worker must satisfy himself that his procedures are valid and fully applicable to his particular situation.

These caveats apply equally well to all aspects of 'storage'. The majority of the papers contributed indicated that, for most of the water types studied, it is expected that the removal of particles will improve the quality of the data, particularly if storage is contemplated. It was suggested that the inconclusive nature of some past experiments on storage stability may be attributable to inadequate quality control in calibration procedures. Further progress continues to be hampered by the lack of Certified Reference Materials in this field.

7.3.2 Review the progress of the BCR Pilot Programme QUASIMEME and discuss its implications for the Sub-group

In response to questions from sub-group members not involved in QUASIMEME, Drs Aminot and Kirkwood assured the sub-group that their own involvement in QUASIMEME would have no negative effect on the proposed plans for the ICES Intercomparison Exercise 'NUTS I/C 5'.

The nutrients aspect of QUASIMEME is not an intercomparison exercise as such, but is intended to be a quality assurance programme which will address, among other things, long-term variance within laboratories.

The coordinators of NUTS I/C 5 pointed out that they are fully in support of BCR's intention to insist that QUASIMEME laboratories accept that participation in NUTS I/C 5 should be a pre-condition for their taking part in QUASIMEME.

The list of laboratories to be invited to join QUASIMEME is still under preparation, but it is likely that the majority of those under consideration are already included in the list of provisional participants for ICES NUTS I/C 5.

7.3.3 Review of plans for ICES 'NUTS I/C 5' Intercomparison Exercise

The list of provisional participants now stands at 110 laboratories and to prevent overloading IFREMER's capacity to produce the required quantity of sample materials, the Chemical Oceanography Sub-group agreed that there should be no further attempts to publicise the exercise. Any additional laboratories which show an interest in participation can comprise a reserve list pending final confirmation of the participation of those on the 110 primary list. Dr Aminot now anticipates that samples will be ready for distribution in late 1992 rather than in early 1993, as was previously stated. The Sub-group sees no reason to postpone the distribution and recommends that the extra time available should be to the benefit of the participants and should enable them to meet the reporting deadline that much more comfortably. Drs Kirkwood and Aminot will revise the schedule accordingly. Participants can expect a newsletter in mid-1992 requiring them to confirm their intention to participate.

The Sub-group recommends that IFREMER be reimbursed for the costs of packaging and distribution. These costs are estimated to be 60,000 DKK.

The Sub-group welcomes the document "Comments on the Evaluation of Intercomparison Study Results" (MCWG 1992/7.1.2) by Shier Berman, and is in a position to assure the MCWG that its plans for the conduct of 'NUTS I/C 5' are fully consistent with the recommendations and views expressed therein. It is anticipated that a preliminary report on the exercise will be available for discussion by the MCWG at its 1994 meeting.

Based on the experience from 'NUTS I/C 4 and 5', the Sub-group is planning to contribute to an MCWG document on how to organise intercomparison exercises.

7.3.4 (ACMP) Review the results of the Visby intercomparison exercise for dissolved oxygen (and materials) in sea water and consider the need for and methods of conducting further QA work on the measurement of dissolved oxygen and hydrogen sulphide

The Sub-group reviewed the report of the HELCOM Intercomparison Exercise held in Visby and made several comments and observations. The sub-group found it rather discouraging that several of the laboratories reporting data to the Baltic Monitoring Programme had abstained from participating in the exercise.

OXYGEN:

The sub-group concurs with the conclusions presented by the organisers that there are no significant differences due to sampling equipment or sampling staff and, therefore, suggests that in future exercises more time and attention could be given to the purely analytical part of this determination, in individual laboratories prior to the field exercise, as this is the part where the possibility of systematic errors is most evident.

Samples low in oxygen require particular attention, as systematic errors will bias these determinations more seriously.

HYDROGEN SULPHIDE:

Future Baltic initiatives should avoid the use of cadmium as a precipitating agent in the stabilisation process for the determination of H_2S. In the Visby exercise, the resultant precipitate proved impossible to re-dissolve, and zinc is recommended as an alternative.

NUTRIENTS:

The Sub-group discussed the results in some detail and offered explanations for some of the deviations. The sub-group noted that in future work the following points should be incorporated:

a) inclusion of organic N and P compounds for a test of digestion procedures in the determination of TN and TP. The substance proposed, riboflavin 5'-phosphate, is not adequate, but the sub-group is not in a position to propose something better at present. Dr Kirkwood agreed to look into this matter intersessionally.

b) careful consideration of background levels of nutrients in blanks and calibration standards, as well as in wash water for the automated techniques.

The exercise was effectively a field intercomparison, and, as in previous exercises of this kind, might have benefited from some preliminary laboratory intercomparison studies. The exercise was a useful demonstration of the kind of interlaboratory variability that can be expected in field work, but a lack of emphasis on the resolution of this variability prevents such exercises from achieving their full potential.

7.3.5 Outcome of the discussions with the Working Group on Shelf Seas Oceanography (February 1992) concerning the sampling protocol and the trend analysis of nutrients in sea water

Following the 1991 Statutory Meeting, members of the Chemical Oceanography Sub-group of the MCWG were invited to the February 1992 meeting of the WGSSO, but only three of those present (at Tenerife), Carlberg, Føyn and Vagn Olsen, had been able to attend the (Copenhagen) meeting.

Protocol for sampling and analysis: The Chairman reviewed the background to the original request for advice on these matters and made it clear that the 'Guidelines' document as produced in 1991 was a relevant document, although the task had not been well specified. The analytical chemistry section of the document has since been expanded and was now before the Sub-group for its final consideration with a view to publication in the ICES Techniques in Marine Environmental Sciences 'TIMES' series.

The Sub-group shares the view of the WGSSO that, rather than a continued elaboration of the basic Guidelines document, a more direct involvement by group members in the development of monitoring programmes is required. Therefore, the Sub-group concurs with both of the WGSSO recommendations, namely:

1) The WGSSO or a sufficient number of its national members (and consequently MCWG members) should be involved in the design of a programme for the monitoring of nutrients in the North Sea within the framework of the revised Oslo and Paris Commissions; and

2) The WGSSO should have a meeting back-to-back with the MCWG in 1993 to discuss matters of common interest, e.g., nutrients and other interdisciplinary aspects of shelf seas oceanography.

Joint initiatives of this kind are seen as a useful way forward, and the Sub-group takes note of the discussions on the evaluation of nutrients data held within the WGSSO.

7.3.6 Any other business

The Sub-group took note of the WOCE intercalibration exercise carried out on board R/V Vernadsky and discussed the results pertaining to the high precision determination of dissolved oxygen contained in the draft report. The Sub-group will welcome the opportunity to consider the final report at a future date.

The Sub-group takes note of the tendency of workers in the nutrients field to continue to express concentrations in a variety of ways, e.g., g-at/l, M, etc., and reminds contributors that md/l is the preferred and most generally used version among oceanographers, and is consistent with IAPSO recommendations. The Sub-group concurs with the continued use of ml/l for dissolved oxygen, but suggests that it should be accompanied by the conversion factor to mol/l.

The future work programme was agreed as follows:

1) Determination and characterization of dissolved organic matter. (Føyn)

2) Carbon dioxide, its distribution and relation to the cycling of nutrients in the sea. (Jon Olaffson)

3) Distribution patterns of nutrients for the explanation of regional phenomena, e.g., algal blooms. (Føyn)

4) What is a representative sampling programme to characterize a sea area. (Carlberg)

5) Continue work with intercomparison exercises. (Aminot, Kirkwood)

6) Problems related to chemical analysis of constituents in anoxic waters. (Kalervo Mäkelä)

The Sub-group re-elected Stig Carlberg as its Chairman for the intersessional period and the next meeting.

8 PLENARY DISCUSSIONS

The proposal of Dr Cofino regarding the preparation of overviews was approved. This entails that overviews will be assessed using the criteria also employed by journals. Publication in the ICES Journal of Marine Science is recommended.

The reports of the Sub-groups were discussed in plenary and approved. The MCWG was satisfied to note that the Trace Metal and Organic Sub-groups independently reached the same conclusions with respect to the data assessments of JMG. The discussions led to a recommendation given in Annex 5.

Dr Tronczynski presented a paper on the interactions between dissolved organic matter and contaminants. He also passed on a number of comments from Dr Yeats on this subject. Dr Wells contributed with a paper on the transport of organics associated with particulates at a sewage sludge dumping site.

The papers gave rise to discussion. At this stage it was not clear how to arrive at a cooperative programme in this area. It was suggested that speakers be invited to elaborate more on some subjects. In addition, members of the group might be willing to present the results of work next year in the form of posters.

After a lively discussion, it was decided that:

- Drs Cofino, Boon and de Boer would investigate whether Dr R. Norstrom could present a paper at the 1993 MCWG meeting;

- J. Tronczynski, P. Yeats and S. Berman would identify a person to present a paper on the interaction between DOM and TM at the 1993 MCWG meeting;

- W. Cofino would invite the persons identified above on behalf of the Group. To this end, J. Boon and J. Tronczynski will inform W. Cofino as soon as their attempts have been successful.

In the Organic Sub-group it was proposed to look into statistical aspects of trend monitoring from an analytical point of view. A similar topic was raised in the Chemical Oceanography Sub-group. It was proposed to invite Dr J. Uthe, Chairman of WGSATM, to participate in these discussions. This proposal was accepted.

9 ANY OTHER BUSINESS

No matters were raised under this agenda item.

10 RECOMMENDATIONS AND ACTION LIST

The action list and recommendations are given in Annex 5.

11 DATE AND VENUE OF NEXT MEETING

MCWG discussed the venue and time of the next meeting. Dr Berman offered to host the meeting. MCWG thanked Dr Berman and recommends that the meeting be held in Ottawa in February 1993.

12 CLOSURE OF THE MEETING

Dr. D de Armas and his staff joined the closing session of the Working Group. On behalf of MCWG, the Chairman thanked them for their warm hospitality and all the substantial efforts and services they provided.

The Chairman thanked the members for their hard work, and closed the meeting at 18.00 hrs on 13 March 1992.

ANNEX 1

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ANNEX 2

ICES MARINE CHEMISTRY WORKING GROUP

Santa Cruz de Tenerife, March 9-14 1992

Agenda

1. Opening

2. Adoption of the agenda

3. Report of the 79th ICES Statutory Meeting

4. Reports on related activities
   - 4.1. Joint Monitoring Group of OSPARCOM
   - 4.2. Intergovernmental Oceanographic Commission (IOC)
   - 4.3. ICES Working Groups
   - 4.4. EC-BCR QA pilot project "QUASIMEME"
   - 4.5. Other activities

5. Reports on projects and activities in member countries

6. Requests from ACMP and regulatory agencies

7. Sub-group activities and discussions

   7.1. Trace Metal Sub-Group
   - 7.1.1. (ACMP) Complete the overviews on chromium and nickel
   - 7.1.2. (ACMP) Complete the paper on the design and execution of intercomparison exercises
   - 7.1.3. (ACMP) Examine and comment on the procedure used by the JMG Ad Hoc Group in December 1991 to assess the quality of the 1990 baseline data
   - 7.1.4. Discuss the progress on the more detailed exploration of the estuarine data collected in the 1985-1987 ICES Baseline Study on Trace metals in Sea Water
   - 7.1.5. Review "new" contaminants and determine where reviews or overviews would be warranted
   - 7.1.6. Problems involving high lipid materials
   - 7.1.7. Paper on quality assurance in the framework of the Baltic Sea Monitoring Programme
   - 7.1.8. Any other business raised by the Sub-group.

   7.2. Organic Sub-group
   - 7.2.1. (ACMP) Finalise the results of Phase 2 of the CB intercomparison, prepare a report on the results of Phase 3a of the CB intercomparison exercise, and complete the planning for Phase 3
   - 7.2.2. (ACMP) Review the results of Phase 2 of the PAH intercomparison and complete the design of Phase 3
   - 7.2.3. (ACMP) Assess the data on lindane (γ-HCH) in sea water for the NSTF MMP
   - 7.2.4. (ACMP) Review the methods used by ICES/JMG laboratories for the determination of lipids and, on the basis of this review, consider the need for an intercomparison exercise to assess the comparability of measurements
   - 7.2.5. (ACMP) Complete the overviews on atrazine and brominated flame retardants

7.2.6. (ACMP) Examine and comment on the procedure used by the JMG Ad Hoc Group in December 1991 to assess the quality of the 1990 baseline data

7.2.7. Review "new" contaminants and determine where reviews or overviews would be warranted

7.2.8. Any other business raised by the Sub-group

7.3. Chemical Oceanography Sub-group

7.3.1. (ACMP) Review the progress on the handling and storage of sea water samples for nutrient determination and report accordingly

7.3.2. Review the progress in the BCR Pilot Project QUASIMEME and discuss its implications for the activities of the Sub-group

7.3.3. Review the state of the plans for the "ICEC NUTS I/C 5"

7.3.4. (ACMP) Review the results of the Visby intercomparison exercise for dissolved oxygen (and materials) in sea water and consider the need for and methods of conducting further QA work on the measurement of low levels of DO and H_2S

7.3.5. Consider the outcome of the discussions with the WG on Shelf Seas Oceanography (February 1992) concerning the sampling protocol and the trend analysis of nutrients in sea water

7.3.6. Any other business raised by the Sub-group

8. Plenary discussion of Sub-group work

9. Any other business

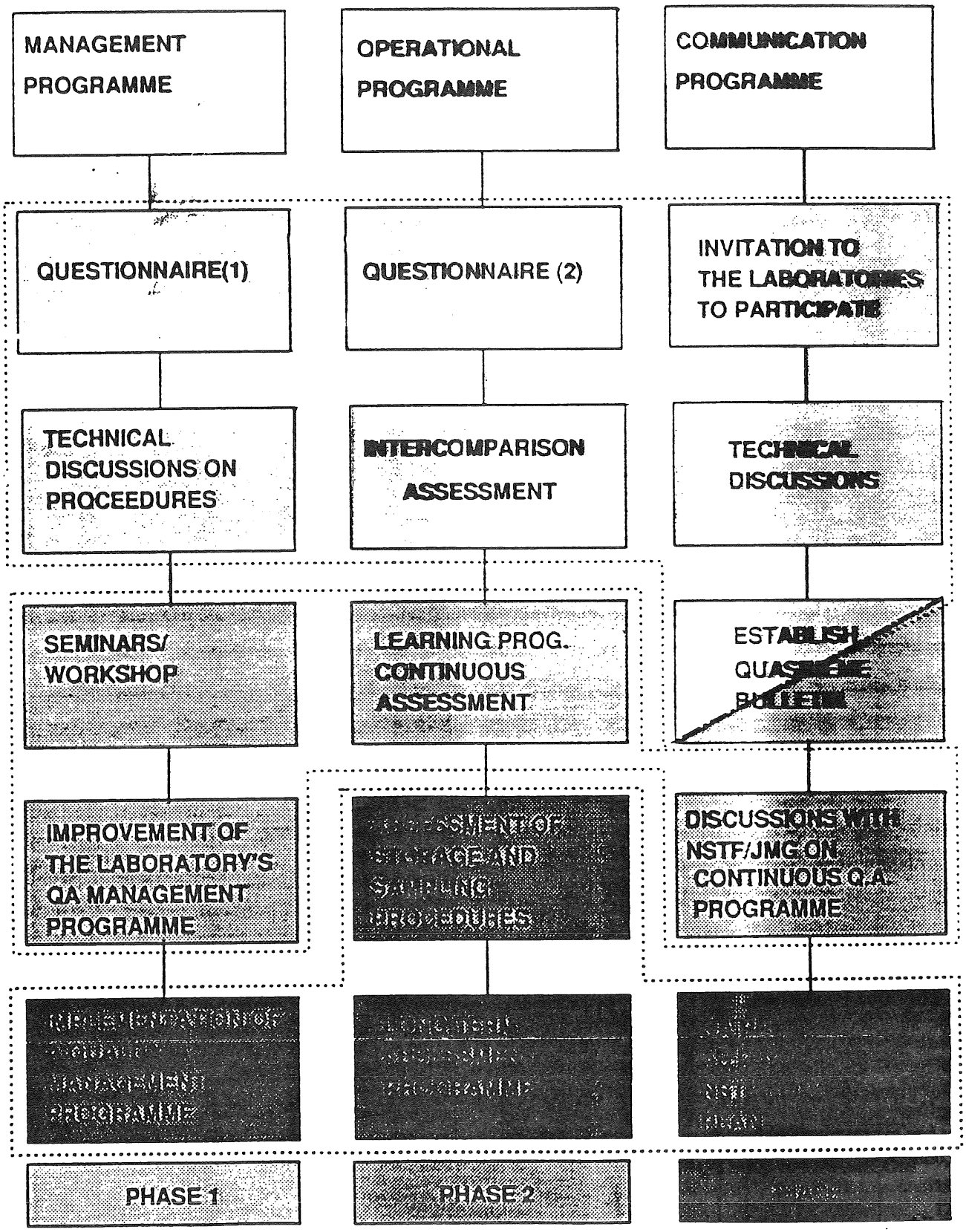
10. Recommendations and action list

11. Data and venue of next meeting

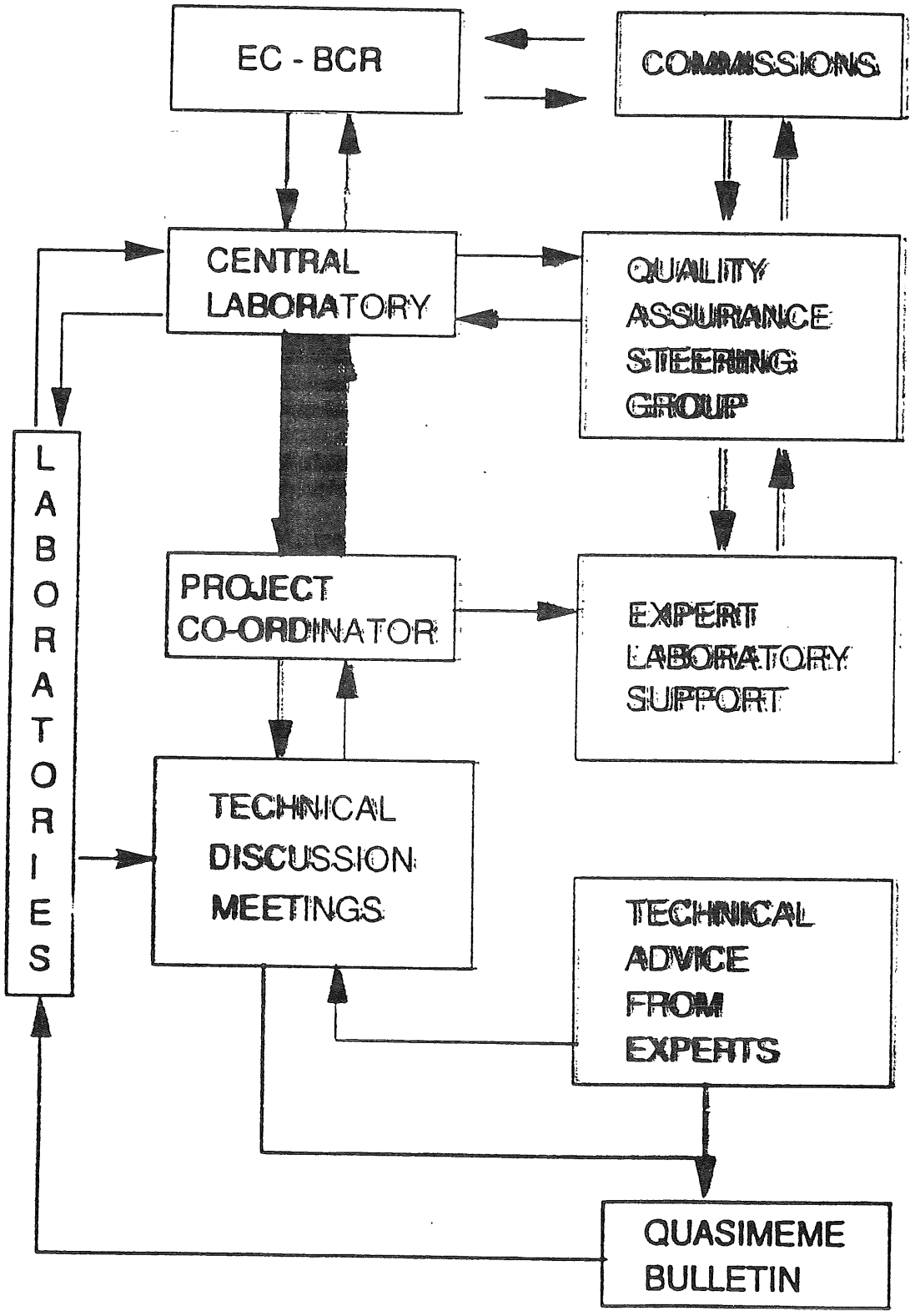
12. Closure of meeting

ANNEX 3

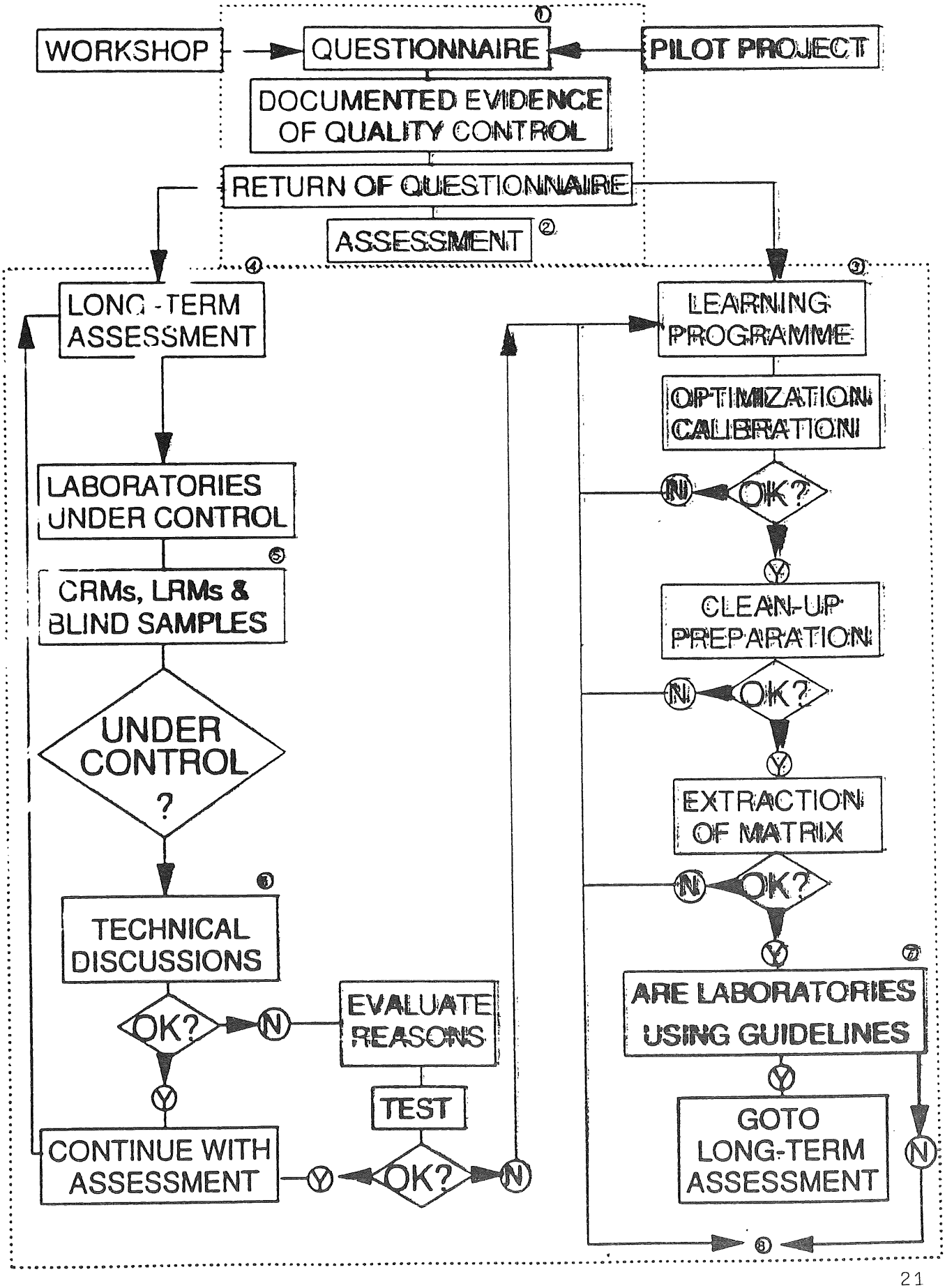
OVERVIEW OF THE QUASIMEME PILOT PROJECT



FLOW CHART FOR THE MANAGEMENT OF THE PROJECT



FLOWCHART FOR THE OPERATIONAL PROGRAMME



ANNEX 4

ICES MARINE CHEMISTRY WORKING GROUP

Santa Cruz de Tenerife, 9-14 March 1992

AN ANNOTATED OUTLINE OF GUIDELINES FOR THE CONDUCT OF INTERCOMPARISON EXERCISES

1. Introduction

Intercomparison exercises are coordinated by different laboratories for any of a range of parameters, compartments and objectives. An intercomparison exercise may be organized among a small group of laboratories in order to study particular methodological problems (a research type objective), but may also be held as a means to qualify laboratories (e.g., acceptance of data in monitoring programmes).

These guidelines do not describe a rigid structure for the conduct of intercomparison exercises. They primarily aim at proficiency testing, for instance, schemes which are employed to qualify laboratories with respect to the acceptance of data. The objectives of these guidelines are to ensure that the design, execution and evaluation of the intercomparison exercise are performed in a valid manner so that proper inferences are made, and that the presentation of information is lucid for both the laboratories and the community of users of laboratory data (e.g., monitoring agencies). The contents are, however, in principle generally valid.

2. Assessment and Description of Objectives

It is necessary that a clear understanding regarding the objectives of the intercomparison exercise exists between the parent organisation, the coordinators and the participants. The coordinators have to assess the stated and implied needs to conduct the exercise, describe the objectives fully, and ascertain that the objectives have the approval of the parent organisation. The coordinators must ensure that sufficient resources to carry out the exercise have been allocated. There must also be agreement regarding the selection of participants.

3. Communication with the Participants

A proper scheme for communication with the participants should be established. When laboratories are invited to participate in an exercise, information has to be provided on the following subjects:

* the objectives of the exercise;
* the way the results of the exercise will be used;
* the fact that laboratories are identified in MCWG exercises;
* the availability of the report for non-participants;
* the time schedule for the whole process;
* the input which is expected of the laboratory;
* measures which will be taken if a laboratory subscribes but does not send in data;
* the CRM(s) to be used during the course of the study;
* the possible financial obligations of the participating laboratory;
* the nature, origin and processing of the samples carried out by the coordinator;
* the descriptions of the forms for reporting the data.

The participants should be notified of any delay in the programme, and should receive a report describing the results of the exercise.

4. Design of the Exercise

4.1. Project team

A project team has to be established. A profound statistical competence is necessary. This input can be provided by a chemist with long experience in this field, or by a statistician who is a coworker of the coordinating laboratory. Alternatively, a member of the ICES WGSATM can be identified.

4.2. Selection of samples

In principle, the concentration levels of the parameters of interest and the characteristics of the samples should be as representative as possible of the materials studied in the project. It is recommended that different samples with different concentration levels or sample characteristics be included in the exercise. Mandatory CRM(s) with appropriate matrix and analyte concentrations should be recommended.

4.3. The preparation and distribution of samples

The preparation and distribution of the samples should be performed in such a manner that their integrity is maintained. It should be assured that the samples are sufficiently homogeneous and stable. Assessment of stability should also take the conditions during transport into account. In international exercises, samples may be inspected by customs, resulting in a loss of integrity. Therefore, clear sealing of the samples is required, so that any manipulations with samples by customs will be observed.

4.4. Statistical considerations

Generally applicable guidelines for the selection of a statistical model cannot be provided. The approach to the design may be based on available national or international standards or on previous approaches deemed to generate successfully the required information. The selected model and any other tests which are used must be described in detail and should be readily understandable by the participants. The underlying assumptions for the model must be described.

4.5. Assigned values.

Where appropriate, an estimate of the true concentration ("the assigned value") for accuracy assessment should be established. For some contaminants/matrices this can be done by using the results from one or more expert laboratories preferably using several independent analytical procedures critically evaluated by the coordinator. It is also possible to derive assigned values from the evaluation of results from all or from a subset of the participating laboratories. The assigned values must have uncertainties (usually standard deviations) associated with them.

5. Statistical evaluation

The raw data should be sent to each participant in order to ensure that there have been no errors in the transposition of data by the coordinator. Data must not be changed unless there has been an error by the coordinator.

The evaluation of the results of interlaboratory studies must be done with care. For instance, problems may arise owing to the statistical distribution of the results, the make-up of the group of participating laboratories, the concentration levels and specific matrix problems. Different statistical models may be employed for the handling of the results (e.g., rugged statistics, different oulier tests, multivariate models, etc). The evaluation and presentation of the results should be attuned to the objective of the study. In addition, the evaluation and the presentation ought to be transparent for the users of the results.

The assessment of the accuracy of a laboratory is the most important goal of ICES intercomparison studies. The accuracy is an estimate of the bias of the participating laboratory with respect to the assigned value for the concentration of the analyte. A report should contain information regarding the assigned value and the way it has been obtained. The assessment of accuracy provides each laboratory valuable feedback with respect to its performance. It is also important to obtain insight into the overall performance of the group of participants. This insight is obtained by looking into the characteristics of the whole population, the major parameters being the consensus concentration and a parameter describing the range of data (e.g., the relative standard deviation, reproducibility).

Currently, the use of the z-score is advocated as a simple parameter to quantify bias. The z-score is defined as $z=(x-X)/s$, where x is the analyte concentration determined by the participant, X is the assigned value, and s is the target representing the maximum allowed variation consistent with valid data. Preferably, a value is assigned to the precision based on accuracy requirements resulting from the information need. Performance is considered acceptable if the z-score is less than two (ISO/REMCO).

The appraisal of the consensus concentration and parameters to describe the range is subject to considerable discussion. Statistical methods employed for interlaboratory studies often assume a normal distribution, a condition which is frequently not met. A detailed discussion of the possible statistical methods and their merits is not included here. In the literature, several approaches are described, and it is the responsibility of the coordinating laboratory to make a sensible choice. Often, ISO 5725 is employed, sometimes with some modifications (e.g., with log-transformation of the data). An elegant approach has been applied in ICES 7/TM/BT. This method entails that the data are successively subjected to a t-test at the 95 percent confidence level until a population remains which represents a fair approximation of a normal distribution. The consensus mean and standard deviation are calculated for this population. An example of this approach is given in MCWG 1992/7.1.2, which is attached hereto.

It is strongly recommended that in every intercomparison exercise the consensus concentration and a parameter describing the range (e.g., the standard deviation, reproducibility) is given for each parameter investigated. The statistics applied should be clearly explained.

Presently, intercomparison exercises are also organized specifically in learning-type programmes. More sophisticated statistical techniques, often involving multivariate models, are applied in order to identify analytical problems. The potential of such approaches is clearly recognized. The results of these calculations should be given, in addition to the outcome of the techniques described above, and explained in detail.

6. Presentation of the information

The presentation of the results and the evaluation should be in the simplest form possible without loss of relevant information. Diagrams should be used to display the overall data for an analyte in a particular matrix. Rejected data should be marked. Various graphs may be used when applicable (e.g., Youden plots, data distribution) and when they enhance the understanding of the report. All data received must be listed and identified in the report. The presentation should also include an evaluation of the submitted CRM data. If an apparent

relationship is observed between various parameters (e.g., performance using a particular methodology), a relevant statistical test should be carried out to determine whether the relationship is statistically significant.

7.Every report must have a section of conclusions and recommendations.

Literature

ISO 5725, Precision of test methods, International Organization for Standardization, 1986.

AOAC Guidelines for Collaborative Study Procedure to Validate Characteristics of a Method of Analysis, J. Assoc. Off. Anal. Chem. 72 (1989) 694-709.

IUPAC Protocol for the Design, Conduct and Interpretation of Collaborative Studies, Pure and Appl. Chem. 60 (1988) 855-864.

IUPAC/ISO/AOAC, Harmonised proficiency testing protocol, June 1991.

International Union of Pure and Applied Chemistry (IUPAC), Analytical Chemistry Division, Commission on General Aspects of Analytical Chemistry (V.1), project 27/87: Nomenclature for Interlaboratory Studies, Fourth Draft (April 1991).

Shier Berman, NRC, Canada

COMMENTS ON THE EVALUATION OF INTERCOMPARISON STUDY RESULTS

The purpose of an intercomparison study is to provide the participating laboratories and the intercomparison study organizers with a means of objectively assessing the reliability of results produced by those laboratories. There are three parameters which are assessed most frequently:

1. Accuracy

The assessment of **accuracy** is usually the most important goal of an ICES intercomparison study. This is an estimate of the **bias** of the participating laboratory with respect to the **assigned value** for the concentration of the analyte. In the best of cases the assigned value will have been predetermined by the coordinator and will be a practical estimate of the true value of the concentration of the analyte in the matrix. In some instances this is not possible and the assigned value will be a consensus value established by the coordinator by a **critical evaluation** of the set of results returned by the participants.

The assigned value can not be merely the consensus value of the participants because there may not be a consensus, or the consensus may be biased due to widespread use of faulty methodology.

The **bias** is equal to (x-X) where

x is the analyte concentration determined by the participant , and

X is the analyte concentration value assigned by the coordinator.

The **relative bias** is (x-X)/X. The relative bias is usually used as the measure of accuracy rather than the absolute bias.

If the user community is able to estimate the precision s needed in order to ensure proper data interpretation, the quotient $z = (x-X)/s$ is a very valuable indicator. If z exceeds 2 there is only a 5 percent probability that the laboratory can produce reliable data.

2. Intralaboratory Precision

This is an estimate of the repeatability of a procedure within the individual participating laboratory. Repeatability for a particular analyte concentration can be assessed by the analysis of replicate samples and is usually described by the **standard deviation** (s) of a single determination. The computation is simple:

$$s=\sqrt{\frac{\sum_{x=1}^{N}(x_i-\bar{x})^2}{N-1}}$$

where x_i is the is the determined concentration of an individual replicate,

$\bar{x}$ is the determined mean of the replicate analyses, and

N is the number of replicate analyses.

The **relative standard deviation** (RSD) is $s/\bar{x}$. This number is often multiplied by 100 to yield the **percent standard deviation.**

An estimate of the repeatability can also be calculated from a set of samples of different analyte concentrations. This is done by a linear regression procedure and yields an overall value of the standard deviation for the range of concentrations tested.

The calculation of the intralaboratory precision is always done in intercomparison exercises but, except for identifying a laboratory with serious precision problems, is of limited value. An intercomparison study is usually a snapshot in time and only provides an estimate of the true standard deviation. The number of replicate samples analyzed is usually rather small and the errors in this estimate can be very large as indicated in Figure 1 below.

The confidence limits for the estimation of a standard deviation are not symmetrical and are surprisingly large for small numbers of replicates[1]. The standard deviation calculated from the results of five replicate analyses has a 95 percent confidence interval ranging from 0.6 to 2.4 times its calculated value. The probability of a "bad" result is quite high. Also, it is obvious that studies based on only one or two measurements may produce misleading results.

A far superior estimate of the standard deviation for a particular analytical procedure is acquired from long term control chart data maintained by any laboratory employing good laboratory practices.

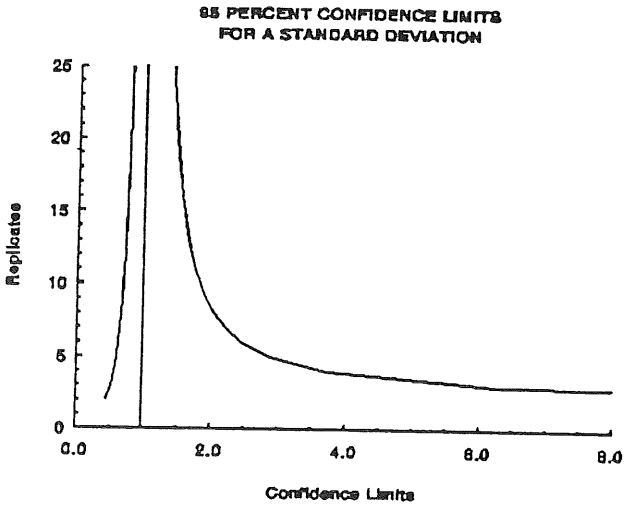


Figure 1

3. Interlaboratory Precision

This is an estimate of the reproducibility of submitted analyte concentrations between the participating laboratories. If there is acceptable accuracy and intralaboratory precision, then the interlaboratory precision can be used to determine whether a cooperative project is feasible between the set of laboratories. It is usually described by a standard deviation and the calculation is identical to that shown above but here

x_i is the determined concentration of an analyte from a single participating laboratory.

$\bar{x}$ is the assigned value for the analyte concentration, and

N is the total number of laboratories whose results are being intercompared.

Other information may be acquired from an intercomparison study such as the efficacy of various analytical procedures. Also, the distribution of laboratory results about the assigned values could lead to a better understanding of the causes of laboratory bias.

There may be a tendency to try to describe the population of results by a rigorous multivariate model which assumes that the determined values of the analyte concentrations are interdependent. This is a difficult concept for an experienced analytical chemist to accept. The response is, that if this is indeed the case, the analytical procedures are inadequate. However, it is possible that a portion of the population is distorting the distribution. If the former is true then this area of analysis has severe problems. If the latter is true then it would be best to find a means of isolating the group whose results may be of an acceptable calibre from the group which is distorting the distribution.

Experiences over the last decade with respect to the analysis of trace metals in various matrices indicate that, as long as the analyte concentrations are above their quantitative limits of determination (at least twice the limit of detection), a group of competent laboratories will produce a set of results homogeneously distributed about a mean which is seldom significantly different from the assigned value. There is no basic reason to believe that organic analytes would produce a dissimilar distribution. The fundamental problem is that, at the current state of the practice of analytical chemistry, the quantitative analysis of materials for trace organic constituents is a much more difficult and challenging task.

Figure 2 is an example taken from a recent intercomparison study regarding the determination of 13 trace metals in sewage treatment plant (STP) effluents[2]. Thirty-five sets of zinc concentrations were submitted by the participants for this sample. The distribution of their mean values is shown in the diagram. The consensus mean is 59.3 micrograms zinc per litre. Aside from what is probably a high biased mean the group can not distinguish concentration differences from between 29 to 115 micrograms zinc per litre. The standard deviation can not be used to calculate this range.

The distribution is obviously skewed towards the higher concentrations and does not appear to be normally distributed. However, what we have here are some quite good laboratories and some poor laboratories. The poor laboratories generally produce high results in trace analysis because they do not have their blanks and contamination under control. They also may produce both high and low results because of poor calibration techniques, improper instrument usage, poor choice of methods and poorly trained staff. The problem is to find a relatively simple method to separate the underachievers from the good performers (i.e, get rid of the outliers).

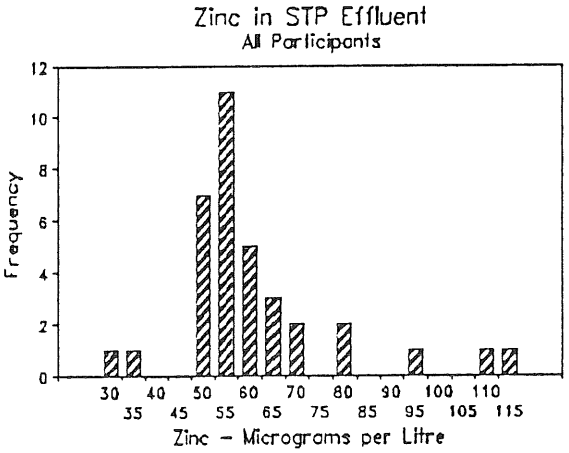


Figure 2

There are many suggestions on how to do this. ISO/REMCO, for example, supports a procedure based on the successive application of the Cochran test and the Grubbs tests[3]. At NRC we prefer a more statistically transparent method involving the successive application of a *t*-test at the 95 percent confidence level to isolate what we believe is a fair approximation of a normal distribution. The results of this procedure on the popuiation of Figure 2 are shown in Figure 3 on the next page.

Eight laboratories were eliminated from the distribution in this example, a larger than usual number. The excluded mean is 55.7 ± 9.8 micrograms zinc per litre. The mean is no longer biased and the range of indiscrimination is reduced to 36 to 75 micrograms zinc per litre with 95 percent confidence.

This method may not be statistically rigorous. One or two laboratories may have been rejected (or accepted) when they should not have been. However, we have found that this type of evaluation of the results is readily understandable to the participants and to the user community of the data, most of whom have a rather unsophisticated understanding of even elementary statistics.

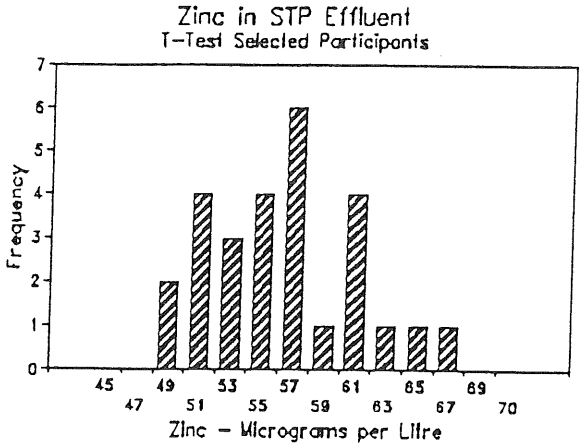


Figure 3

The main purpose of the study has been achieved. A subset of the participants has been identified as a homogeneous group and its performance has been characterized. The organizers of the study and the user community are aware of the possible consequences of using any one of the participants in a future project. They are also aware of the limitations on the quality of the data which can be produced by the group as a whole or any subset of laboratories they may choose from this group. This knowledge should be incorporated in their planning. They should be wary of any laboratory, regardless of reputation, which has not participated in an intercomparison study or which has not been accredited through some harmonized proficiency testing program related to their project interests.

The participating laboratories have gained in that they are aware of their own capabilities, based on an objective assessment. The "rejected" laboratories must examine their procedures in order to improve their capabilities, seeking outside advice if necessary. The others must also continually seek to improve. The range of indiscrimination between laboratories is still too large to produce the necessary quality of data for many environmental projects.

References

1. Statistics Manual, Crow, E.L., Davis, F.A. and Maxfield, M.W., Dover Publications, New York, 1960.

2. MOE/CAEAL Pre MISA Laboratory Assessment of Sewage Treatment Plant Effluents for Trace Metals, Berman, S. and Willie, S., Ontario Ministry of the Environment Report PIBS 1741, 1991.

3. Protocol for the Design, Conduct and Interpretation of Collaborative Studies, Ed. Horowitz, W., J. Pure and Appl. Chem. 60, 855-864 (1988).

ANNEX 5

RECOMMENDATIONS AND ACTION LIST

ACTION LIST

Chairman

W. Cofino	Contact G. Topping about the review on Cr in the marine environment and ensure that this topic is removed from the MCWG agenda.
W. Cofino	Send a copy of the Guidelines for Intercomparison Exercises to the ICES WGSATM for consideration.
W. Cofino	Invite speakers on behalf of the MCWG to the meeting (Ross Norstrom and others to be identified).
W. Cofino	Work intersessionally with the new chairman of WGSSO concerning a back-to-back meeting MCWG/WGSSO.
W. Cofino	Distribute action and recommendation list to participants in the 1992 MCWG meeting.
S. Wilson	Contact J. Pawlak on funding NUTS 5, inform A. Aminot on the outcome as soon as possible.

Trace Metal Sub-group

U. Harms	Prepare an outline for a collaborative research programme on alkyltins, organo-mercury and organo-arsenic compounds.
S. Wilson, Asmund, B. Pedersen	Examine ICES/JMG data in ICES databank with respect to (possible) relationships between lipid G. G. content and trace metal concentrations in biota.
S. Wilson	Distribute data on trace metals in (estuarine) water to B. Pedersen, P. Yeats, L. Brügmann, P. Balls, S. Westerlund.
P. Balls, L. Brügmann, B. Pedersen, S. Westerlund, P. Yeats	Continue interpretation of estuarine data in the ICES data bank.
S. Berman	Distribute information on silver to members of the Trace Metal Sub-group.
S. Berman	Inform P. Yeats on the achievements of the 1992 MCWG meeting.
S. Berman	Request P. Yeats to expand the paper on the interactions between dissolved organic matter and trace metals.
J. Tronczynski, S.Berman	Identify a person who can present a paper on the interaction between DOM and TM at next year's MCWG meeting. Pass the name on to W. Cofino.

Organics Sub-group

H. Gaul — Expand the lindane assessment for the NSTF before 1 May, 1992.

J. Boon — Provide information on toxaphene overview for report.

F. Smedes, D. Wells, B. Jansson, J. Tronczynski — Distribute papers one month before the meeting.

J. de Boer — Prepare a report on setp 3b of the CB intercomparison exercise.

W. Cofino, F. Smedes, R. Law — Prepare a report on progress in the PAH intercomparison exercise

F. Smedes — Prepare a paper on variations in lipid concentrations in relation to contaminant concentrations.

F. Smedes — Send a copy of sheets (possibly with explanation) to B. Pedersen and S. Wilson.

F. Smedes — Measurements of sediment-water distribution coefficients of PCBs excluding the influence of dissolved organic matter.

E. Nixon, J. de Boer — Prepare a paper on the variability of lipid determinations.

D. Wells — Prepare a paper on the distribution in the aquatic environment of planar compounds.

B. Jansson — Prepare a paper on chlorinated naphthalenes.

J. Tronczynski, P. Yeats — Prepare a paper on contaminants and dissolved organic matter.

Chemical Oceanography Sub-group

D. Kirkwood — Prepare a note concerning alternatives for riboflavin.

José Escanez — Report on the WOCE intercalibration to the Chemical Oceanography Sub-group.

D. Wells — Provide information on SI/IUPAC units to S. Carlberg.

L. Føyn — Prepare a paper on the determination and characterization of dissolved organic matter.

Jon Olaffson — Prepare a paper on carbon dioxide, its distribution and relation to cycling of nutrients in the sea.

L. Føyn — Prepare a paper on distribution patterns of nutrients for the explanation of regional phenomena, e.g., algal blooms.

S. Carlberg — Prepare a paper on what is a representative sampling programme to characterize a sea area.

K. Mäkelä — Prepare a paper on the problems related to chemical analysis of constituents in anoxic waters.

All

All	Ensure that information which has to be assessed arrives before the meeting, so that members can have a look at the data prior to the meeting.
All	All members are invited to present papers. If possible, please distribute these one month prior to the meeting. Posters are welcome too.
All	Contact delegates regarding funding of NUTS 5.

RECOMMENDATIONS

The Marine Chemistry Working Group recommends that:

Recommendation 1

The ACMP critically review the procedures and practises currently applied in the data assessment activities of both ICES and the Commissions, taking into account the comments of MCWG and the JMG's ad hoc Working Group on Monitoring in this respect. The ACMP should prepare appropriate and detailed advice for transmission to the Commissions with a recommendation that it be adopted and implemented at the earliest possible time, paying particular attention to the timetables for the provision of relevant information for use by the data assessment groups (validated monitoring data, QA information, national comments, etc.) well in advance of any proposed assessment meeting.

Recommendation 2

ICES should publish the report of on the results of the Seventh Intercalibration Exercise on the Analysis of Trace Metals in Biological Tissue (7/TM/BT) Part 2 (by Dr S.S. Berman *et al.*) in the ICES Cooperative Research Report Series.

Recommendaton 3

ICES should publish the report on the results of Stage 3a of the ICES/IOC/OSPARCOM Intercomparison Programme on the Analysis of Chlorobiphenyls in Marine Media at a later time, with the (future) report of stage 3b, in the ICES Cooperative Research Report Series.

Recommendation 4

Stage 3b of the ICES/IOC/OSPARCOM Intercomparison Programme on the Analysis of Chlorobiphenyls in Marine Media should be organised in 1992 under the coordination of Dr J. de Boer; a fee will be levied on each participant to cover the costs of the samples.

Recommendation 5

The second phase of the Intercomparison Programme on the Analysis of Polycyclic Aromatic Hydrocarbons (PAHs) in Marine Media should be organised in 1992, under the coordination of F. Smedes, R. Law and W. Cofino; a fee will be levied on each participant to cover the costs of the samples.

Recommendation 6

The Fifth ICES Intercomparison Exercise on the Analysis of Nutrients in Sea Water (5/NUT/SW) should be organised under the coordination of A. Aminot and D. Kirkwood in 1992/1993; IFREMER (brest) should be reimbursed for the costs of bottles and distribution, the cost of which is estimated to be 60,000 DKK.

Recommendation 7

The Marine Chemistry Working Group (Chairman: Dr W. Cofino) should accept the offer made by Dr. S. Berman to host the next meeting in Ottawa. This meeting should be held for six days in February 1993 to carry out the following tasks:

a) to review the progress in NUTS 5;

b) to review the results of stage 3b of the CB intercomparison exercise;

c) to review the results of stage 2 of the PAH intercomparison exercise;

d) to complete planning for stage 3 of the PAH intercomparison exercise;

e) to review the progress in the evaluation of estuarine data held in the ICES data bank;

f) to review the programme for cooperative research in the field of metal speciation;

g) to review the information on the relationship between trace metal concentrations and lipid content in biological tissues;

h) to review the documents on lipid variability;

i) to consider the information on the interaction of contaminants with dissolved organic matter;

j) to review the current practice and statistical design of current monitoring programmes;

k) to consider the papers and posters submitted by MCWG members;

l) to consider any other matters raised by ACMP.