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REPORT OF THE MEETING OF THE STEERING GROUP ON QUALITY ASSURANCE OF CHEMICAL MEASUREMENTS IN THE BALTIC SEA

Gdynia, Poland, 16-19 March 1993

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1 OPENING OF THE MEETING

The Steering Group on Quality Assurance of Chemical Measurements in the Baltic Sea held its meeting at the Institute of Meteorology and Water Management in Gdynia. This meeting was preceded by a Joint Meeting with the Steering Group on Quality Assurance of Biological Measurements in the Baltic Sea, the report of which is contained in Doc. C.M. 1993/E:9.

Ms Eeva-Liisa Poutanen, Environment Secretary of the Helsinki Commission, served as Rapporteur for the meeting. The agenda is attached as Annex 1 and the list of participants as Annex 2.

2 DISCUSSION OF ORGANIZATION, PROGRAMME AND PROCEDURAL ASPECTS OF THE QUALITY ASSURANCE (QA) WORKSHOP IN GERMANY

At the opening of the meeting the Chairman, Dr Uwe Harms, informed the members that the intended purposes of the workshop are:

> To assist laboratories in the implementation of the technical aspects of quality assurance (QA) for chemical measurements in the Baltic Monitoring Programme (BMP) and to provide guidance on QA in relation to sampling, storage and pretreatment procedures prior to analysis, accurate analytical measurements, and data assessment procedures.

The Chairman stressed that the precise and complete formulation of an analytical problem is the first step to its solution. The Group discussed the aspects to be covered during the Workshop and agreed that the main aspects of the Workshop should be the items indicated in Annex 3.

Concerning the programme of the Workshop, the Group concluded that the programme should follow the structure provided in Annex 4. A more detailed programme will be elaborated by the Chairmen of the Workshop (Dr U. Harms and Dr G. Topping) together with the local organizers.

The invitation and the Workshop programme will be sent to the ICES and HELCOM Secretariats for further distribution by the end of April 1993. The Contracting Parties of HELCOM and ICES Member Countries will be requested to nominate the participants to the Workshop by the end of June 1993.

The Group welcomed the information that part of the funds allocated in the HELCOM budget for reference materials can be used to cover the travel expenses and daily subsistence allowances of some participants in the Workshop (HELCOM 14/18 Paragraph 5.25).

3 CONSIDERATION OF ANALYTICAL TECHNIQUES AND LABORATORY FACILITIES IN BALTIC COUNTRIES WITH REGARD TO ANALYSES OF THE TYPES OF DETERMINANDS AND MEDIA TO BE COVERED IN THE WORKSHOP

The Group regretted that not all countries around the Baltic Sea were represented at the meeting and, therefore, the consideration of analytical techniques and laboratory facilities could not be adequately covered. In order to be able to get some background information before the Workshop, the Chairman and the Rapporteur developed a questionnaire asking for relevant information, as attached in Annex 5 of this report. The questionnaire will be distributed by the HELCOM Secretariat to the laboratories responsible for nutrient and trace element analyses under the BMP. The answers to the questionnaire should be sent to the Chairman, Dr U. Harms, with a copy to the HELCOM Secretariat by the end of June 1993.

As requested by the HELCOM Environment Committee (EC 3/17 Paragraph 6.16), the Steering Group discussed the specific certified reference materials for the mandatory measurements of the BMP. The Group recommended that for nutrients HELCOM should purchase four sets of Sagami standards for further distribution. For trace elements in biota, five sets of the following materials should be purchased:

> DORM 1 (dogfish muscle) and DOLT 1 (dogfish liver) both from the National Research Council of Canada (NRCC), and CRM-422 (cod muscle) from the Community Bureau of Reference (BCR).

The Group recommended that the appropriate occasion to distribute the reference materials is the Workshop in Hamburg.

Concerning reference materials for the organic contaminants, the Group felt that they were not in a position to recommend any specific reference material at this time. This question will be further discussed at the Workshop.

4 EC-BCR PROJECT "QUASIMEME"

With regard to matters related to chemical QA under the HELCOM BMP, the attention of the Group was drawn to the project "Quality Assurance of Information in Marine Environmental Monitoring in Europe"

(QUASIMEME) under the Community Bureau of Reference (BCR) of the European Community.

Dr H. Gaul, a member of the QUASIMEME Steering Group, gave an overview on the present status of the QUASIMEME programme. The Group noted that most of the relevant laboratories within the European Community and EFTA countries have been invited to join in the project, but ways are also being sought to involve laboratories which are not located in either European Community or EFTA countries, but which collect data for the BMP.

According to the information available, it seems to be possible to include informally at least one laboratory from every Contracting Party to the Helsinki Convention and as an Observer Country Latvia as a participant in the programme. The laboratory representatives will be provided with all documentation and materials for intercomparisons and they will be welcome to attend the relevant meetings of the QUASIMEME project, however, non-European Community countries would need to cover their own expenses for travel and subsistence.

The QUASIMEME questionnaire was distributed to the contact addresses for BMP data in non-European Community countries by the Chairman on 24 February 1993. So far, laboratories from Estonia, Latvia, Lithuania, and Poland have expressed a positive attitude towards the project. After having returned the questionnaire to Dr D. Wells, QUASIMEME project manager, they will receive a copy of the QUASIMEME QA Manual.

5 QA REQUIREMENTS FOR BMP MONITORING PARAMETERS

The Group considered QA requirements in relation to the existing guidelines for the BMP. The Group felt that the QUASIMEME QA Manual might be an appropriate instrument for introducing QA concepts and procedures into the measurement of chemical parameters within the BMP. Since the Manual has not yet been made available to all members of the Steering Group, it was recommended that the topic should be discussed in more detail at the Workshop.

Concerning the existing BMP Guidelines, the Group felt that, as the natural variation of contaminants in marine media still requires further research, it was not in a position to recommend any principal changes concerning parameters or matrices. However, the Group stressed that intercalibration and intercomparison exercises must be an essential first step before a new contaminant is introduced as an obligatory parameter in the BMP.

The Steering Group discussed what additional QA-related information should be provided with the BMP data and proposed that, *inter alia*, control charts containing information on the regular analyses of laboratory internal reference materials and appropriate certified reference materials must be included.

6 ANY OTHER BUSINESS

In order to promote international scientific cooperation, the Steering Group strongly recommended that ICES and HELCOM should encourage the exchange of scientists between Baltic laboratories.

7 DATE AND VENUE OF NEXT MEETING

The Steering Group agreed that matters relating to the quality assurance of chemical measurements will be discussed at the Workshop in Hamburg.

8 ADOPTION OF THE DRAFT REPORT

The draft report of the Joint Meeting of the Steering Group on Quality Assurance of Chemical Measurements in the Baltic Sea and the Steering Group on Quality Assurance of Biological Measurements in the Baltic Sea and the draft Report of the Steering Group on Quality Assurance of Chemical Measurements in the Baltic Sea were adopted by the Group. The draft report of the Joint Meeting of the Steering Group on Quality Assurance of Chemical Measurements in the Baltic Sea and the Steering Group on Quality Assurance of Biological Measurements in the Baltic Sea was also adopted by the Steering Group on Quality Assurance of Biological Measurements in the Baltic Sea. In addition, the two groups provided each other with a copy of their respective reports.

9 CLOSING OF THE MEETING

The members of the Steering Group on Quality Assurance of Chemical Measurements joined with the members of the Steering Group on Quality Assurance of Biological Measurements for the final closing session. On behalf of both groups, Dr Uwe Harms thanked the organizers of the meetings for their warm hospitality and helpful services. Dr Lars Hernroth also thanked the organizers for the kind invitation and the substantial efforts which were required to make the meetings a success.

Dr Uwe Harms thanked the members for their work and contributions and closed the meeting at 12.00 hrs 19 March 1993.

ANNEX 1

STEERING GROUP ON QUALITY ASSURANCE OF CHEMICAL MEASUREMENTS IN THE BALTIC SEA

Gdynia, Poland, March 16-19 1993

AGENDA

1. Opening of the meeting.
2. Discussion of the Quality Assurance Workshop in Germany.
3. Consideration of analytical techniques and laboratory facilities in Baltic countries with regard to analyses of the types of determinands and media to be covered in the Workshop.
4. EC-BCR Project "QUASIMEME".
5. QA requirements for BMP monitoring parameters.
6. Any other business.
7. Date and venue of next meeting.
8. Adoption of the draft report.
9. Closing of the meeting.

ANNEX 2

STEERING GROUP ON QUALITY ASSURANCE OF CHEMICAL MEASUREMENTS IN THE BALTIC SEA

Gdynia, Poland, 16-19 March 1993

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ANNEX 3

WORKSHOP ON QUALITY ASSURANCE OF CHEMICAL ANALYTICAL PROCEDURES FOR THE BALTIC MONITORING PROGRAMME

Aspects to be covered during the Workshop

I NUTRIENTS

Formulation of the analytical problem:

1 Nutrients in Baltic waters
   a) different concentration levels
   b) different water bodies
2 Methods available (concentration range)
3 Special problems
   a) total nitrogen
   b) ammonia
4 Calibration
   a) manually
   b) automatically
5 Blank control
6 Recovery control
7 Control by individual laboratory's internal standards
8 Control by certified reference materials
10 Control charts
11 Sampling and sub-sampling
12 General aspects of internal and external QA

II TRACE ELEMENTS

Formulation of the analytical problem:

1 Trace elements in Baltic biota
   a) different concentration levels
   b) different tissues/organs
2 Methods available (concentration range)
3 Special problems
   a) contamination through reagents
   b) vessels
   c) uncontrolled losses
4 Calibration
5 Blank control
6 Recovery control
7 Matrix interferences
8 Control by individual laboratory's internal standards
9 Control by certified reference materials
10 Control charts
11 External QA

ANNEX 4

DRAFT PRELIMINARY PROGRAMME FOR THE ICES/HELCOM WORKSHOP ON QUALITY ASSURANCE OF CHEMICAL ANALYTICAL PROCEDURES FOR THE BALTIC MONITORING PROGRAMME

5-8 October 1993, Hamburg, Germany

OPENING OF THE WORKSHOP

LECTURES ON THE FOLLOWING TOPICS on 5-7 October 1993:

1 QUASIMEME Programme,
Introduction on QA aspects by ....
2 Most important aspects of nutrient analyses (including error sources) by Prof. D. Nehring.
3 General introduction to trace element analysis (including error sources) by Dr Uwe Harms.
4 State-of-the-art analysis of organic compounds by Prof. Luckas.

ROUND TABLE DISCUSSION ON THE FOLLOWING TOPICS:

- Appropriate sampling programmes to obtain representative samples for the intended purposes.
- Laboratory facilities, labware and reagents of the necessary quality to meet the particular needs of the sampling and analytical operations.
- Appropriate collection, preservation, storage and transport procedures to maintain the integrity of samples prior to analysis.
- Suitable pre-treatment procedures, prior to analysis of samples, to prevent uncontrolled contamination, and loss of the determinand, in the samples.
- Validation of appropriate analytical procedures, by the use of relevant certified materials, to ensure that measurements are of the required accuracy and precision to meet the needs of the work.
- Conduct of regular checks on the accuracy and precision of routine measurements, by the analysis of appropriate reference materials, to assess whether the analytical procedures are remaining under control, and the documentation of the results on control charts.
- Participation in external quality assessments to provide an independent assessment of the laboratory's capability of producing reliable measurements.
- The preparation and use of written laboratory protocols so that specific analytical data can be traced to the relevant samples, and *vice versa*.

DEMONSTRATIONS RELATING TO SOME PRACTICAL ASPECTS OF ANALYSIS IN THE LABORATORIES IN HAMBURG SÜLLDORF.

8 October 1993

FINAL DISCUSSION IN PLENARY ABOUT THE OUTCOME OF THE WORKSHOP, CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE STEPS AND ADOPTION OF THE DRAFT REPORT TO BE PRESENTED TO EC 4 AND ICES.

ANNEX 5

Questionnaire to be Distributed to the Laboratories Responsible for Nutrient and Trace Element Analyses for the Baltic Monitoring Programme

1 The name of the laboratory and the person responsible for nutrient and/or trace element analysis.

A. NUTRIENTS

2 Are you using manual methods or autoanalysers?
State the name of the manufacturer and the type of the instrument.

3 Method of calibration.
Which standards are used?
What is the matrix of your standards?
What concentrations are used for stock solutions and working calibration solutions?
How often are you doing the calibration?
Give characteristic data on calibration function (slope, intercept, residual standard deviation, etc.).

4 What is the procedural blank b and its variability Sb for the analytical procedure applied?

5 Give any information about interferences.

6 Are you using any sample pre-treatment prior to analysis?
Are you preserving your samples?

7 Give a short description about methods used and relevant references.

8 Describe any special problems you may have (e.g., availability of reagents, contamination, stability of instruments).

B. TRACE ELEMENTS

9 Which instruments are used in your laboratory?

10 Method of calibration.
Which standards are used?
How do you prepare your standards?
What concentrations are used for stock solutions and working calibration solutions?
How often are you doing the calibration?
Give characteristic data on calibration function (slope, intercept, residual standard deviation, etc.).

11 What is the procedural blank b and its variability Sb for the analytical procedure applied?

12 Have you any problems with uncontrolled losses or contamination?

13 What is your sample pre-treatment (freezing, freez-drying, homogenization, etc.)?

14 Give a short description about methods used and relevant references.

15 Describe any special problems you may have (e.g., availability of reagents, contamination, stability of instruments).

16 Please refer to any special problem you may have in your laboratory (e.g., no clean benches, purified reagents, etc.).

C. POINTS RELATED TO QUALITY ASSURANCE

17 Do you check your method on a regular basis by using laboratory internal or certified reference materials?

18 Do you perform regular checks of the blanks?

19 Do you document results under items 17 and 18, above, by using control charts? If yes, include a copy.

20 Have you participated in any international intercalibration or intercomparison activities during the past 5 years? If yes, please indicate which activities or exercises.

21 Do you have any special wishes about the Workshop?