1980-02-28

Analytical Chemists Meeting, Feb. 27, 28, 1980. Canada Centre for Inland Waters, Burlington, Ontario

Great Lakes Water Quality Board. Surveillance Subcommittee. Data Quality Work Group

International Joint Commission
Data Quality Work Group of the
Surveillance Subcommittee,
Great Lakes Water Quality Board

Analytical Chemists Meeting

Feb. 27, 28, 1980
Canada Centre for Inland Waters
Burlington, Ontario

PROGRAM TABLE OF CONTENTS

• WELCOME AND INTRODUCTIONS

By K. I. Aspila
Inland Waters Directorate
Burlington, Ontario

Mr. Aspila, the Data Quality Work Group Chairman, welcomed the attendees and introduced Mr. Oakley, the Director of the IJC Great Lakes Regional Office; members of the Work Group; Dr. John Clark, Senior Statistician, IJC, Great Lakes Regional Office; and Mr. Don King, Quality Assurance Officer, Ontario Ministry of the Environment, Laboratory Services Branch.

Mr. Oakley welcomed the analysts and commented on the need for adequate environmental assessment data in implementing the Great Lakes Water Quality Agreement of 1978. He stressed the need for Great Lakes chemists to be aware of the very important role they play towards the enhancement and restoration of the Great Lakes.

• WATER - A RESOURCE SHARED AND PROTECTED BY CANADA AND THE UNITED STATES

By R. E. White
International Joint Commission
Windsor, Ontario

PEOPLE AND WATER

Probably nearly no one would have predicted in 1909 that 40 million people would cluster around the Great Lakes as they do today - 14% of all of the people in the United States and 33% of the people of Canada reside in the Great Lakes Basin.

Also, probably no one 70 years ago could have predicted the multiple uses for Great Lakes waters today and the various forms of degrading impacts man has placed on these waters.

However, there were wise men in those times just as there are today. Those men of vision from our past recognized the need to formulate how the waters of Canada and the United States which flow to, through, and within our common boundaries, should be shared and protected. They recognized that there were abuses, and disputes particularly concerning the waters of Milk and St. Mary Rivers, and that there would be future problems, and therefore they sought a way to protect the rights of each nation and its people.

BOUNDARY WATERS TREATY FORMED - 1909

Their answer was a treaty between the nations, the Boundary Waters Treaty of 1909. The Treaty was proclaimed May 13, 1910.

As testimony to the clear thinking of the formulators of this Treaty is the fact it still stands today with only three paragraphs of one article no longer in force. The Treaty contains 14 articles dealing with such things as water diversion, waterway obstruction, water use and furthermore contains one of the most forward thinking and essential statements for you, me, and our nations and probably a benchmark statement for the world. A paragraph within Article IV states:

"It is further agreed that the waters herein defined as boundary waters and waters flowing across the boundary shall not be polluted on either side to the injury of health or property on the other."

A remarkable statement for 1909 and one very germane today. Without this statement, and subsequent supporting actions by our nations, our Great Lakes would not be in the useful state they are today.

INTERNATIONAL JOINT COMMISSION INSTITUTED

The formulators of the Treaty also knew that a treaty is only a piece of paper if it does not have a steward, someone or some body to ensure its implementation and continuing viability. Therefore they included within the Treaty provisions for creating a new international body, the International Joint Commission of the United States and Canada, and described its responsibilities under the Treaty.

The Commission is a binational body of six members, three members appointed by each country. They act as a single unit, not as delegates from their respective countries, and reach decisions by simple majority.

The Treaty describes 3 specific functions for the Commission; they are: 1) quasi-judicial, 2) investigative, and 3) surveillance/coordination. Their quasi-judicial function relates to approval of applications to construct dams or other facilities that will affect the natural levels or flows of waters specified in the Treaty.

The Commission has investigative responsibilities at request by the governments through referral. Recent studies such as the upper lakes condition and the pollution from land use are examples. Furthermore, the governments can extend the right to the Commission to render a binding decision for their countries. However, this has not been done and probably will never be done. However, the Commission may adopt any procedure it deems necessary to carry out the intent of the Treaty.

The IJC maintains headquarters in Ottawa, Ontario, and Washington, D.C.

LOWER LAKES STUDY CONDUCTED

Amid concern by the people residing among the lower lakes, in 1964 the Governments of Canada and the United States asked the Commission to investigate the condition of lakes Erie and Ontario and the St. Lawrence Seaway to determine the extent of international pollution, the pollutant sources, and to recommend remedial measures and estimate their costs.

The Commission formed two Boards, the International Lake Erie Water Pollution Board and the International Lake Ontario-St. Lawrence River Water Pollution Board. Membership was comprised of representatives of Federal, Provincial (Ontario) and State Governments.

The Boards reported their findings to the IJC in 1969 and public hearings were held. In 1970, after considering the Boards' reports and the findings from public hearings, the Commission made its report to the governments. The Commission concluded in part:

- o the waters were being seriously polluted;

- o the principal sources of pollution were wastes discharged by municipalities and industries, and;

- o the major source of phosphorus was municipal sewage.

Further, the Commission concluded that it should be assigned the tasks of coordinating continuous surveillance of water quality, of monitoring the implementation of pollution abatement programs, of coordinating the exchange of information on all aspects of water pollution, and of reporting and publishing the results on the effectiveness of such governmental programs. The Commission also proposed water quality objectives for lower Great Lakes.

GREAT LAKES WATER QUALITY AGREEMENT OF 1972 INSTITUTED

As a direct result of the Commission's findings the United States and Canada made a specific agreement on the Great Lakes, the 1972 Water Quality Agreement. This Agreement re-affirmed the rights of each country to use the waters of the Great Lakes, and expressed the determination of each country to restore and enhance the water quality of the Great Lakes Basin.

To assure the vitality of the Agreement, the nations assigned additional responsibilities and powers to the Commission and provided for the establishment of a regional office if deemed necessary by the Commission. Among these additional responsibilities were:

- collecting, collating, and disseminating data concerning the boundary waters and meeting Great Lakes water quality objectives;

- tendering advice and recommendations to the states, province and Parties;

- providing assistance in coordinating activities, water quality research, contingency planning, and consultation; and

- investigating pollution from land use and the actions needed to preserve and enhance the upper lakes, and other investigations as may be later assigned to the Commission.

POWERS GIVEN TO THE COMMISSION

To do these things, the Commission was given the right to exercise all the powers conferred upon it by the Boundary Waters Treaty and any legislation passed pursuant thereto, including holding public hearings, compeling testimony of witnesses, and the producing of documents.

In addition to instructing the Commission to report on progress and make recommendations to the governments annually, and giving it the right to publish reports, the Commission was also given the authority to verify independently the data submitted to it by the governments.

INTERNATIONAL JOINT COMMISSION'S GREAT LAKES REGIONAL OFFICE FORMED

To help meet its responsibilities described within the Agreement, the Commission established a Great Lakes Regional Office, a binational office comprised of equal numbers of professional people from both nations in the fields of biology, limnology, chemistry, engineering, statistics, and others. Two Boards were formed, the Water Quality Board and the Research Advisory Board along with their supporting committees and groups. The Upper Lakes and Pollution from Land Use Activities Reference Groups (ULRG and PLUARG) were also established.

Much was accomplished over the five year period of that Agreement. Billions were spent to upgrade the efficiency and completeness of sewage treatment, some states proposed bans on phosphorus in detergents, and others curtailed the amount used to 0.5% by weight as recommended by the Commission. Proper sewage treatment is in place for 99% of the Canadian sewered population and construction of treatment plants continues at a fast pace in the United States.

Spill contingency plans are in place, the Upper Lakes and Pollution from Land Use Activities Reference Groups have reported to the Commission. The Commission has forwarded its findings and recommendations on the upper lakes, and is about ready to report on pollution from land use activities. New water quality objectives have been adopted and Lake Erie no longer appears dead.

The Parties recognized their successes but also it was clear to all that much remained to be done.

Aside from past and present concern for the control of phosphorus and heavy metals, particularly organo mercury, toxic chemicals from land fills, chemical plant effluents, and other sources demanded attention.

NEW AGREEMENT - NEW EFFORTS

The governments drew up a new Agreement to attend to past unfinished business and to address new issues. On the 22nd day of November 1978, this new Agreement was signed by dignitaries representing both nations.

While still addressing the phosphorus loading issue exemplified by more stringent limitations and the requirement for future loading allocations, the new Agreement expands obligations to include the entire Basin - not just boundary waters. It also provides more focus on toxic compounds devoting two separate annexes to the subject, Annex 10, Hazardous Polluting Substances, and Annex 12, Persistent Toxic Substances.

Annex 10 commits the Parties to list toxic and hazardous materials, and to develop programs to eliminate or minimize their presence in the Great Lakes. Annex 12 requires the development of a quantitative inventory which identifies the raw materials, processes, products, by-products, waste sources, and emissions involving persistent toxic substances. It further calls for coordination between air, water, and solid waste pollution assessment and control programs, including monitoring for trends and the establishment of an early warning system to predict likely problems.

COMMISSION'S ROLE REAFFIRMED

The new Agreement reaffirms the Commission's role, the IJC Great Lakes Regional Office, and the Water Quality Board. It changed the Research Advisory Board to the Science Advisory Board and emphasized that its membership should represent managers within research. This change could have further impact in integrating research within the Basin on water quality problems.

The Water Quality Board, the principal advisor to the Commission, is charged to:

- o make recommendations on development and implementation of programs to meet the purposes of the Agreement;
- o assemble and evaluate information derived from the programs;
- o identify deficiencies in scope and funding of programs;
- o examine program appropriateness taking into account socio-economic realities; and
- o provide liaision to ensure comprehensive and coordinated approaches to plan and resolve problems.

The Science Advisory Board is the scientific advisor to the Commission and the Water Quality Board and provides:

- o recommendations on research and the development of scientific knowledge supporting the Agreement;
- o advice to jurisdictions on research needs; and
- o assessments and recommendations on pertinent ecosystem research.

The Boards report to the Commission on a periodic basis on request of the Commission.

In addition to Annexes 10 and 12 dealing with toxic and hazardous substances there are 10 other Annexes that deal with specific objectives, limited use zones, control of phosphorus, discharges of oil and other discharge of vessel wastes, review of pollution from shipping, dredging, discharges from onshore and offshore facilities, and a joint contingency plan.

Although Annexes 10 and 12 impact your work as analytical chemists, Annex 11 clearly addresses your work.

Annex 11, Surveillance and Monitoring, spells out the need to:

- assess the degree of compliance to regulations promulgated by jurisdictions;
- determine the achievement of general and specific objectives;

- evaluate water quality trends;

- identify emerging problems;

- determine inputs from tributaries, point sources, atmosphere, and connecting channels;

- develop whole lake data, nearshore information, and fish and wildlife contaminants; and

- determine pollutant levels in outflows, water intakes and outlets.

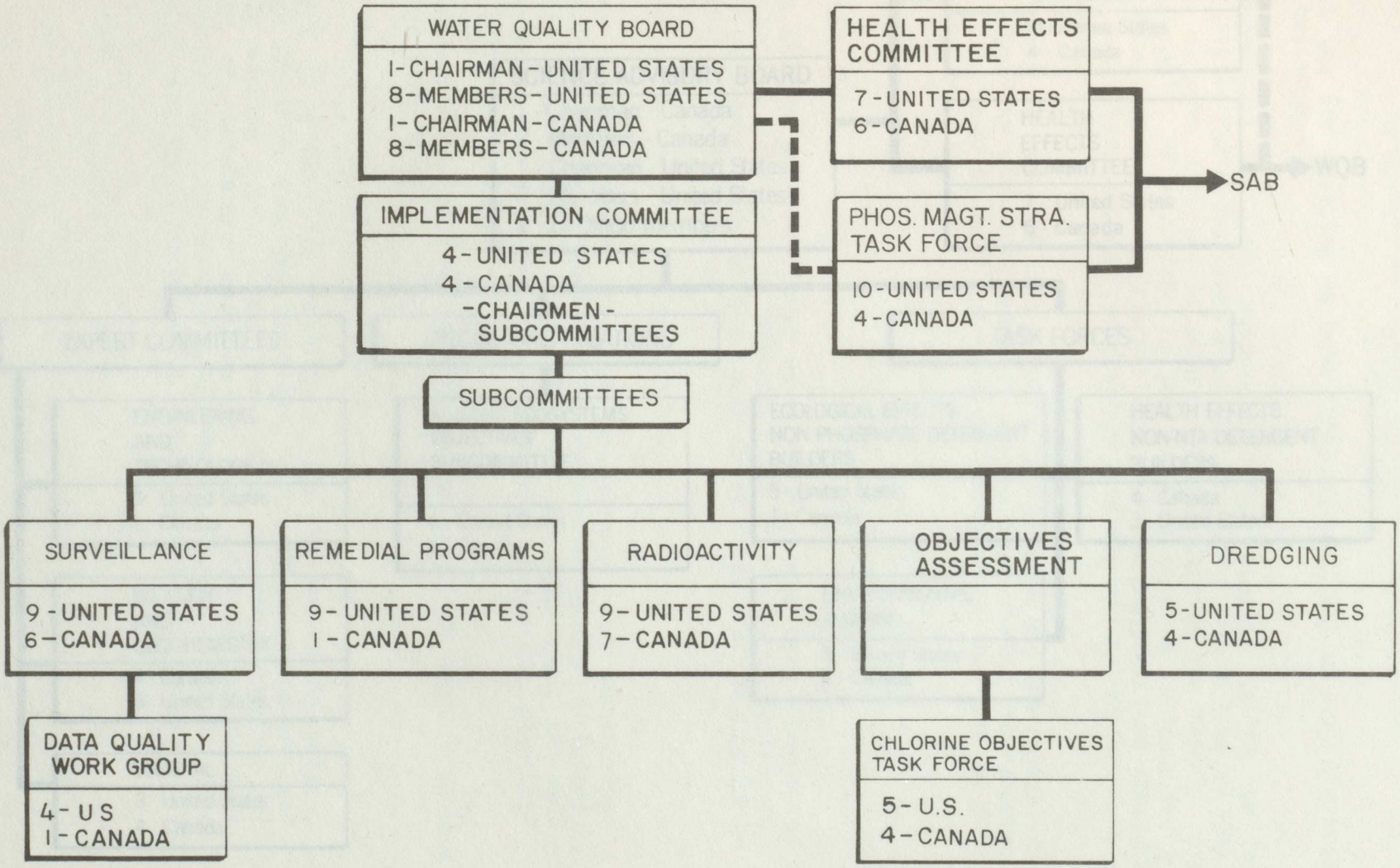
The Annex also recognizes that these assessments cannot be made without data quality assurance and specifically calls for standard sampling and analytical methodology, interlaboratory comparisons, and compatible management.

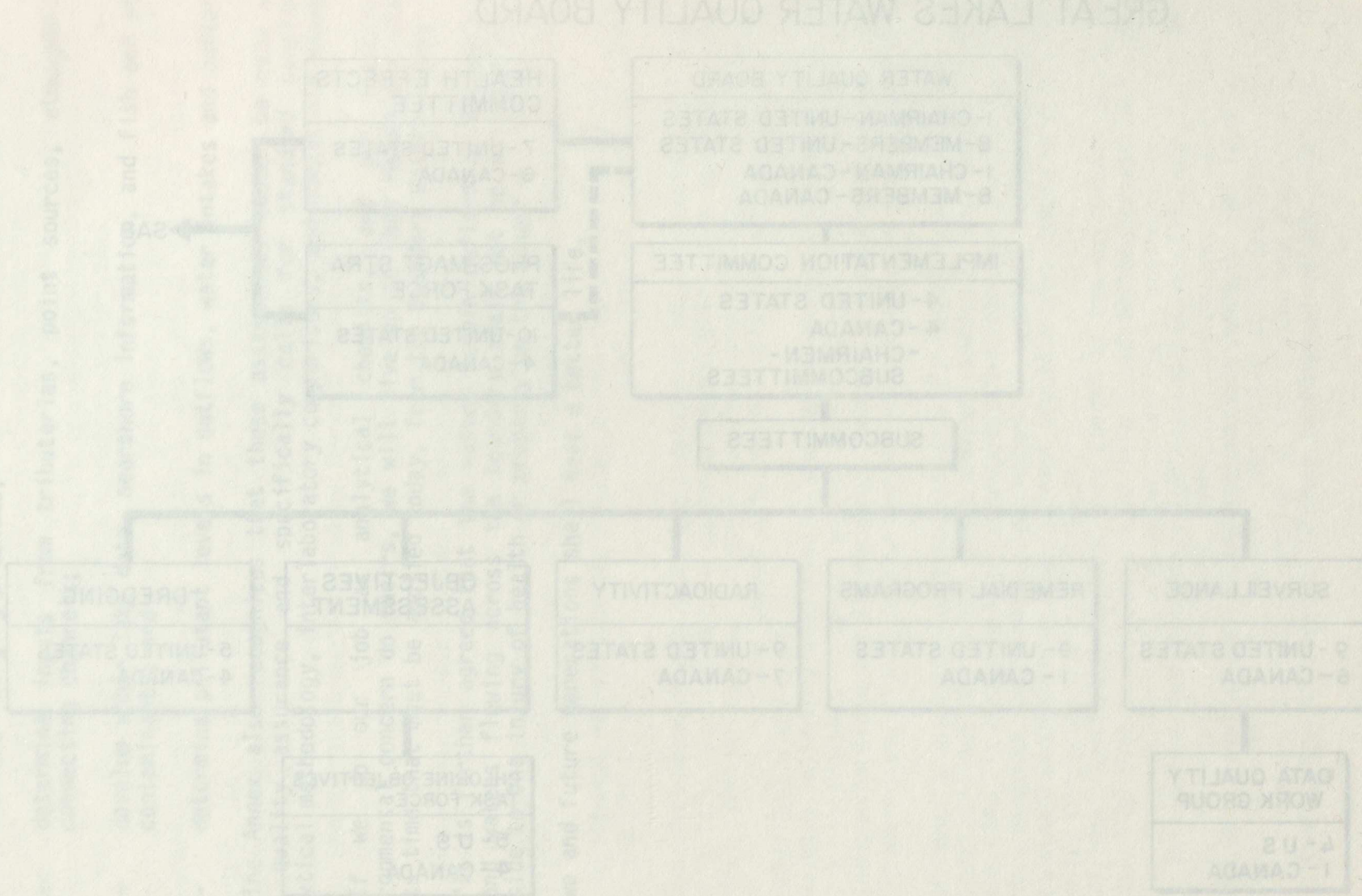
If we do our job as analytical chemists, and our associates in environmental concern do theirs, we will live up to that remarkable statement of its time that must be applied today, from the Boundary Waters Treaty -

"It is further agreed that the waters herein defined as boundary waters and waters flowing across the boundary shall not be polluted on either side to the injury of health or property on the other" -

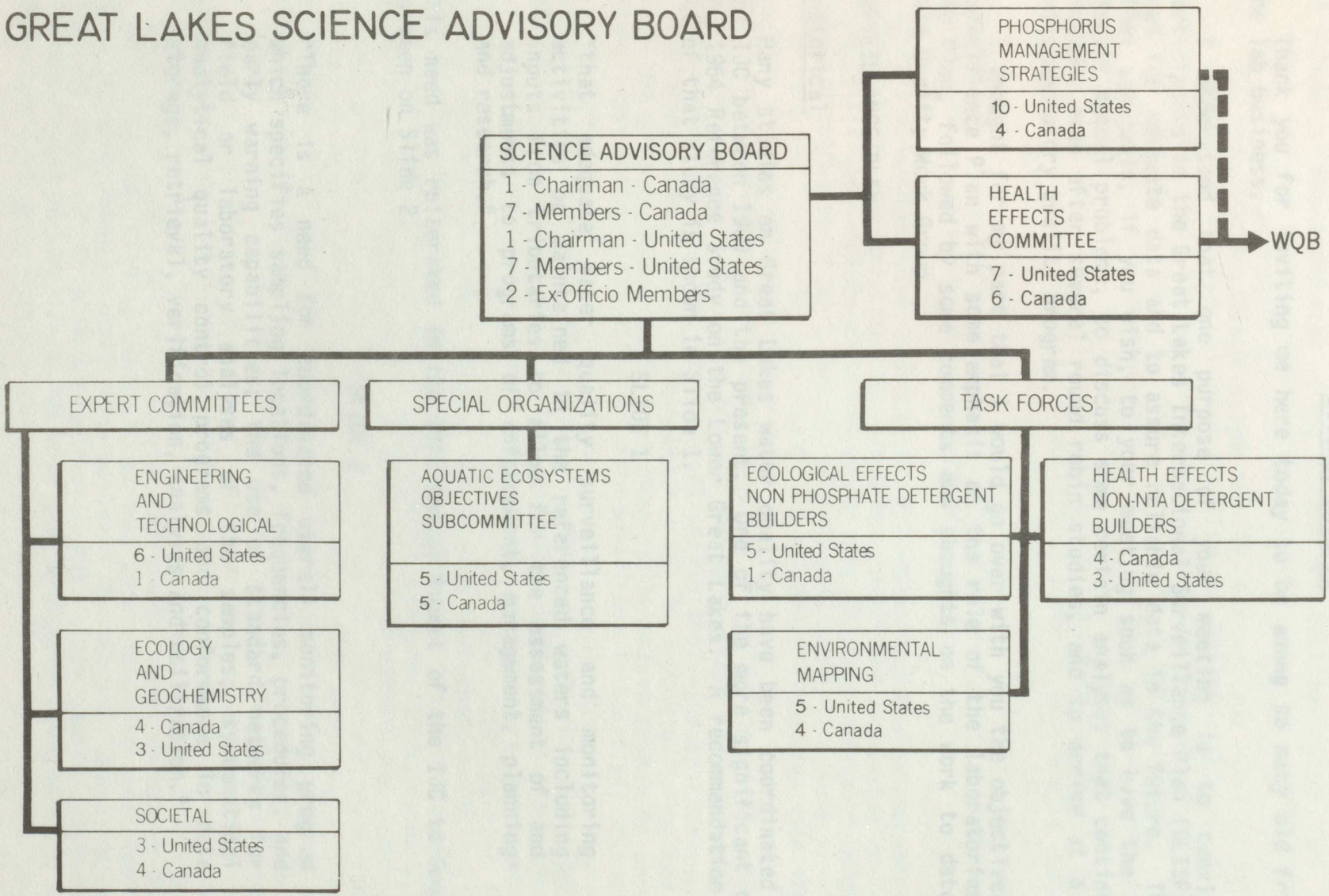
and we and future generations shall have a better life.

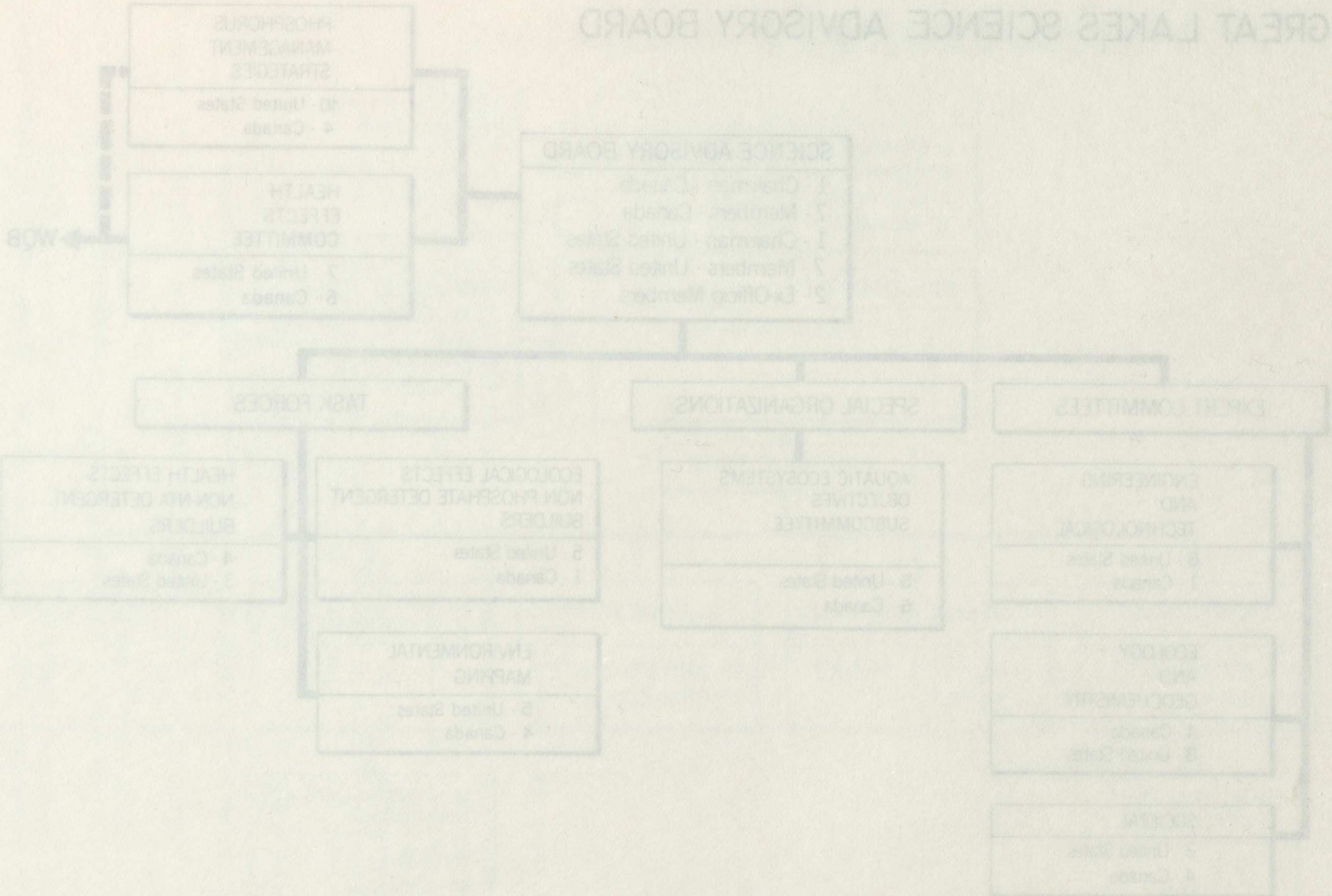
GREAT LAKES WATER QUALITY BOARD





GREAT LAKES SCIENCE ADVISORY BOARD





• THE GREAT LAKES INTERNATIONAL SURVEILLANCE PLAN

By W. J. Traversy
Water Quality Branch, Inland Waters Directorate
Ottawa, Ontario

Thank you for inviting me here today to be among so many old friends in the lab business.

I understand that one purpose of your meeting is to convince lab participants in the Great Lakes International Surveillance Plan (GLISP) of the need for adequate data and to assure reliable data in the future. There are other sub-goals, if you wish, to your meeting such as to have the analysts discuss mutual problems, to discuss some certain analyses that continue to be problems even after several round robin studies, and to arrive at a minimum intralaboratory quality program.

I thought for my part that I would go over with you the objectives of the Surveillance Plan with some emphasis on the role of the laboratories within the Plan, followed by some comments and thoughts on the work to date of the Data Quality Work Group.

SURVEILLANCE PLAN

Historical

Many studies on Great Lakes water quality have been coordinated through the IJC between 1909 and the present. One of the more significant ones was the 1964 Reference Study on the Lower Great Lakes. A recommendation arising out of that study is shown in Slide 1.

SLIDE 1

> "that adequate water quality surveillance and monitoring activities be maintained in the referenced waters including inputs from tributaries to allow for the assessment of and adjustments to programs of enforcement, management, planning and research."

This need was reiterated in the 1973 Annual Report of the IJC to Governments as seen on Slide 2.

SLIDE 2

> "There is a need for coordinated overall monitoring program which specifies sampling locations, frequencies, procedures, and early warning capabilities; the use of standard networks for field or laboratory analyses of the samples; systematical analytical quality control programs and conformance in data storage, retrieval, verification, analyses and utilization."

The first general "Program Design" of a Surveillance Plan was presented by the Surveillance Subcommittee (SSC) to the Water Quality Board in its 1974 report. This design outlined the overall goals, objectives and rationales for each plan element.

In 1977, with the consent of the Water Quality Board, the SSC began preparation of the detailed Surveillance Plan for each of the Great Lakes. This plan would contain all the details never before written down - exact sampling locations, number of samples, type of samples, parameters to be measured, etc., and the plan was to be a base plan and not a wishing list or a "pie in the sky plan" as earlier ones had been called. This work was completed and presented to the Board just last December. The plan now calls for a total expenditure of about 10 million dollars (all agencies).

PURPOSE

SLIDE 3

The fundamental objective of the Great Lakes International Surveillance Plan is to determine the impact of man's activities on the quality of the Great Lakes ecosystem, particularly with the impact of these activities on the uses of the resource. Now, this purpose is tied into three fundamental needs that basically make the program go.

FIRST - Man's activities in the Great Lakes Basin generate wastes and the disposal of these wastes into the air and water. Billions of dollars have been spent and will continue to be spent on pollution abatement programs to mitigate the impact of these substances. Surveillance, therefore, is needed to determine the effectiveness of these abatement programs and to determine the state of compliance with jurisdictional control requirements.

SECOND - Surveillance is needed to enable the countries to fulfill the requirements of the 1978 Agreement as far as monitoring and surveillance is concerned.

THIRD - Given the number of agencies in both Canada and the U.S. involved in surveillance and monitoring activities and the amount of resources being expended, it makes good sense simply from a management point of view to focus and coordinate these efforts in the most cost-effective way possible.

The primary output of the Plan then is information to assist managers and policy makers in arriving at rational and effective decisions in the overall management of the Great Lakes ecosystem quality.

DESIGN

I am not going to go into details contained in the Plan - you can all get copies and read for yourselves; however, I would like to comment on how the Plan was designed. The Plan is based on water uses and water quality concerns associated with those uses. The elements that make up the Plan design are related to those concerns - SLIDE 4. The Plan is designed to measure:

(1) Input from:

Tributaries
Point Sources (Municipal and Industrial)
Atmosphere
Connecting Channels, and

(2) To measure the impact of these inputs on:

Connecting Channels
Main Lakes
Nearshore (Problem Areas, Beaches, and Water Intake)
Fisheries; and
Wildlife.

Collectively, these elements provide a holistic assessment of the impact of man's activities on the Great Lakes aquatic ecosystem.

What I have just discussed was the philosophy and the development of the GLISP. However, there is another component of the Plan that I did not discuss and that is "Implementation". Effective implementation is an important yet complex aspect of the Plan given the number of agencies involved, the number of laboratories, differing priorities, coordinating, sampling, and reporting schedules and many other details.

Your meeting here today is a step in the implementation of the GLISP. With something like 22 laboratories involved in the program, a strong active quality assurance program is essential. As a chemist, I recognize the difficulty in obtaining compatible results, yet this is important.

Now, how has the Data Quality Work Group handled the needs for quality assurance up to this time? They have operated by preparing solutions whose concentrations are known only to them and distributing these solutions to the labs participating in the surveillance program and who are represented here today by you people. Now, presumably you or your staff carried out the required analyses on these samples and transmitted the results to IJC in Windsor (Bob White) who, in turn, analysed the results and informed each lab on an individual basis as to how well or badly they performed. Now this IJC quality assurance program should be only a part of any labs external quality assurance program, so the results should not be construed as an absolute indicator of data quality. However, this information when used along with a vigorous internal quality control and other external ones should lead to meaningful conclusions.

Now if a laboratory scores poorly, often in these quality control studies, I think that those labs should use that information as a level for additional resources needed to bring the lab in line with others. Those chemists should get after their bosses and say "Look, we don't measure up with other labs and we are of the opinion that the problem is a result of our labs deteriorating systems (old equipment, etc.)." I say this because it was my experience during my days as a quality control chemist that the problem of poor performance was usually traced to old and antiquated instruments, too few technicians, and generally run-down labs. Now, if a lab has all good, new

equipment and adequate numbers of competent staff and still scores poorly, then someting else must be wrong, requiring further investigation.

The purpose of the IJC data quality assurance program, as I see things, are twofold. The first objective is to provide the IJC with an ongoing evaluation of the validity and ensure confidence of the data being used for report preparation. The second is to give agencies involved in the surveillance program an external self-evaluation capability to complement their internal ones.

Now, how successful has this procedure been? Up to a point, I believe that the program has been and will continue to be quite adequate. Many good things have come out of the program - some labs have improved, some poor methods have been identified and discarded, and better overall data has resulted; but some problems still exist - some labs have consistently scored poorly despite repeated warnings that something is wrong, and I believe that this is an important reason why you are here today and again tomorrow in a workshop type atmosphere.

Your program to date has dealt mainly with inorganic parameters in water or sediments and there have been difficulties. I understand that you are now moving into organics and quality control for that area. Your task will be formidable to say the least - particularly as you move into wildlife and fish analyses. I wish you luck and I will be following your activities with interest.

Thank you again for inviting me and for the opportunity of sharing these thoughts with you. I sincerely hope your meetings will be beneficial to yourselves as analysts and to the surveillance program.

• INTERLABORATORY QUALITY CONTROL PROGRAMS
by K. I. Aspila

A. WHY INTERLABORATORY STUDIES ARE REQUIRED

To support the Great Lakes International Surveillance Program, the Data Quality Work Group of the Surveillance Subcommittee has been charged, in part, to develop and implement methods for conducting interlaboratory comparisons and evaluating their results. Reference to interlaboratory comparison studies are found in the Terms of Reference that were approved February 3-4, 1977 by the Surveillance Subcommittee. These terms of reference are given below.

TERMS OF REFERENCE FOR THE SURVEILLANCE SUBCOMMITTEE DATA QUALITY WORK GROUP

The Data Quality Work Group (DQWG) will provide the Surveillance Subcommittee (SS) with a recommended Quality Assurance Program whose purpose is to ensure that the analytical data submitted by each jurisdiction and agency will be as accurate and precise as necessary for the Surveillance Program. To accomplish this, the Data Quality Work Group will provide the following specific functions and information:

1. Develop a Quality Assurance Statement of broad general nature to encompass the elements of the Quality Assurance Program.

2. Develop and implement methods for conducting interlaboratory comparisons and evaluating their results.

3. Define the intralaboratory quality control program required for support of the Surveillance Program and monitor laboratory compliance.

4. Document and evaluate the suitability of various procedures used by each laboratory for each test and provide full information on the analytical characteristics.

5. Develop field sampling and handling protocols.

The Work Group recognizes that interlaboratory studies are essential mechanisms to identify the existing level of comparability among laboratories supporting the Surveillance Program and to identify bias in the laboratory measurement system.

When a group of laboratories are presented with stable natural test samples for analysis, very often, many of these laboratories are unable to agree on the concentration of the constituents. The reasons for disagreement can be attributed to the laboratory measurement system. Some variables within the measurement process responsible for interlaboratory deviations are:

a) in-lab standards (each laboratory can have different reference materials);

b) application of test method (there are subtle technical differences when various laboratories apply the same or similar methods);

c) differences between test methods and/or the influence of the test sample on the test method; and

d) stability of test samples (this area is under control since for most, if not all constituents, only stable test samples are provided).

When multi-sample check sample studies are presented to participants and data evaluated by the Work Group, the resulting reports address the laboratory measurement process and not the field related variables within the jurisdiction. Identification and control of the measurement system is recognized by the Work Group as an excellent first and positive step in interlaboratory control of bias on data being routinely obtained for the Surveillance Program.

After stating what interlaboratory studies address, it was emphasized that after sound interpretation, such studies provide valuable and constructive feedback to:

a) inform each analyst on their performance relative to a peer group of 10 to 30 other laboratories;

b) assist management by provision of documentation that identifies an ongoing basis the performance of their laboratory;

c) assist the current and future official users of surveillance data;

d) assist each laboratory manager in confirming the success of their in-lab quality control procedures.

In addition to the above four benefits derived from interlaboratory studies, it was noted that ongoing participation also provides local management the unique opportunity of providing positive feedback to successful analysts and also the opportunity to have local management constructively appraise their own measurement system with analysts should performance be consistently identified as unsatisfactory.

B. INTERLABORATORY STUDIES COMPLETED

Interlaboratory studies that were completed during (1978 and 1979) were briefly reviewed. These studies were as follows:

1978 Studies

Study No. 21 - Major Ions, Trace Metals and Nutrients in Water
Study No. 22 - Major Ions and Nutrients in Water
Study No. 23 - Trace Metals in Water
Study No. 24 - Total Phosphorus in Water
Study No. 25 - Reactive Silica in Water

1979 Studies

Study No. 26 - Arsenic and Selenium in Water
Study No. 27 - Major Ions, Nutrients and Physical Measurements in Water
Study No. 28 - Total Phosphorus in Water
Study No. 29 - Trace Metals in Water
Study No. 30 - PCBs in Ampuls and Sediments
Study No. 31 - Metals in Fish - (in preparation)

C. QUALITY CONTROL FACILITIES OF THE SPECIAL SERVICES SECTION (CCIW)

In lieu of a tour of the quality control laboratories, a brief slide show of the laboratories in the Special Services Section at CCIW was presented. Included was a description of the stock of reference waters on hand and the extensive development of sediment reference materials (for organic and inorganic constituents). Although this bank of water and sediments have been established to serve the regional requirements of the Water Quality Branch and the Canadian National Interlaboratory Quality Control Program, there has been a natural spin off benefit to serve some quality assurance components of the Great Lakes International Surveillance Program.

Design of Interlaboratory Studies

Discussion was presented on the need by the Work Group to introduce studies using complex arrays of 10 to 14 test samples comprising of blanks, dilute standards and a variety of natural samples. The complex array is required in order to have the concentrations cover the operating range of the majority of participants. These participants are quite diverse and cover those laboratories with programs involving open waters, nearshore waters, tributaries and point sources. The use of natural samples are necessary to retain more perspective on comparability (bias and precision) on real samples

which in turn relates to comparability of the real data generated in the routine surveillance programs. Problems seen with real samples in interlaboratory studies quite often cannot be identified by simple ampul concentrates. The use of several samples in a study has the added benefit that when bias in the measurement process is present, that bias becomes more rigourously defined. A single result that is deviant is a suspicious result but when 10 or 14 results from one laboratory are deviant it becomes a discussion item that warrents review by both laboratory analyst and local management.

"W" and "T" Codes (and Negative Concentrations)

This area was reviewed and analysts were complimented for their application of such coding in interlaboratory studies. Although some subtle differences in opinions were expressed on when data should be flagged "W", it appeared most analysts were applying the codes constructively to prevent the ambiguity of reporting data as simply "less than."

Subsequent group discussion on W and T codes, as well as a followup discussion in the trace metals "task group" session brought up the intriguing and almost necessary requirement that analysts in the future should be requested to consider reporting these negative concentrations when providing results at very low level concentrations. Although received with some reservations by analysts when reporting single results, the use of negative concentrations have significant impact on the appraisal of large data sets.

INTERLABORATORY TEST EVALUATION PROCEDURE

The sample results received from the laboratories are placed in tabular form by lab and sample number. The overall results of each laboratory are evaluated for bias and individual errant sample results are identified.

A set of a laboratory's results is said to be biased when the set exhibits a tendency to be either higher or lower than some standard - the standard which has been used in the analysis of work group studies thus far has been the performance of all other participating laboratories. The ranking procedure employed in testing for bias is described in W. J. Youden's paper, "Ranking Laboratories by Round-Robin Tests" from Precision Measurement and Calibration, H. H. Ku, Editor, NBS Special Publication 300-Volume 1, U.S. Government Printing Office, Washington, D.C., 1969. In this paper, Youden establishes the rationale for evaluating laboratories' performance by ranking results. In the Work Group's use of the procedure there is about 1 change in 20 of deeming a set of results biased when in fact it is not, that is, $\alpha = 0.05$.

To determine if a laboratory is using a method of sufficient sensitivity or applying a method appropriately, the Work Group has requested that results which a laboratory might report in an ambiguous way, such as less than values, be replaced by two codes, W and T. The W code is used with a result when no measurement was possible due to no response of the instrument to the sample. The W is preceded by the smallest determinative division that can be used in the units used in reporting. The T code is used for results with values between Criterion of Detection and the W value. The Criterion of Detection is commonly thought of by many as the limit of detection.

Errant results are values of unacceptable deviation from the median value obtained on the sample from all results. The median values were selected as the target values because the true values of the samples were not verified by a panel of reference methods known to be bias free. An errant result is flagged as either high (#) or low (b). A sample result is not flagged when it falls within an acceptable deviation range. This allowable range is determined from the values chosen for the basic acceptable error (BAE) and the concentration error increment (CEI). These values (BAE and CEI) are derived primarily from the results received for the range of samples analyzed, augmented by the work group's judgement of reasonable performance. The underlying concept is that if several laboratories are found to perform adequately with the values chosen, then all laboratories participating should be capable of that level of performance. In a sense, the values represent the present state of the art for analysis of this kind of round robin samples.

Since for almost all substances it appears that the variability of results increases with increases in concentration an allowance is made for the increased variability for those samples whose target values are above the lower limit for use of basic acceptable error. The allowance is added to the basic acceptable error, and it is calculated by multiplying the concentration error increment by the difference between the target value and the lower limit for use of basic acceptable error.

For example: The lower limit for use of basic acceptable error for a measured constituent is 15 mg/L, the basic acceptable error is 1.5 mg/L, and the concentration error increment is 0.05 mg/L. The target value for the sample was determined to be 33.0 mg/L. The difference between the target value and the lower limit for use of basic acceptable error is 33 - 15 = 18 mg/L. Multiplying this difference (18 mg/L) by the concentration error increment (0.05) equals 0.9 mg/L. This allowance is added to the basic acceptable error of 1.5 mg/L to determine the acceptable difference of 2.4 mg/L for the sample. Therefore, any reported result within the range 33 $\pm$ 2.4 or 30.6 to 35.4 mg/L would be considered acceptable and not flagged.

A result is flagged high # when its value is greater than the target value plus the acceptable difference but not greater than the target plus 1.5 times; a result greater than 1.5 times the acceptable difference is flagged with ##. Similarly, a result less than the target minus the acceptable difference but not less than the target minus 1.5 times the acceptable difference is flagged b; a lower result is flagged bb.

The absolute values of the maximum difference between a result and the target value which will not be flagged is the Acceptable Difference or Acceptable Deviation.

Laboratory results are judged satisfactory when they are quite acceptable, "good results." Results are judged erratic when the laboratory set displays both high and low flags. An out of control designation is given when a laboratory demonstrates the ability to perform adequately and produces an extreme result or results. For example, consider the set of results by laboratory #3 on total phosphorus in Study #24.

Sample No.	Reported Value	Median	Difference
1	9	9.5	-.5
2	5	4.5	.5
3	2T	3	-1
4	8	8	0
5	2T	2.5	-.5
6	9	8	1
7	28	28	0
8	18	17	1
9	23	23.7	-.7
10	16	15	1
11	35	35.8	-.8
12	75	78.7	-3.8
13	58	59	-1
14	110	90	20

Given the excellent results obtained on samples 1 through 13, the result on sample 14 indicates that the analytical system was out of control.

Upon completion of the study, each laboratory receives from the Work Group general comments on the study, e.g. source of samples, overall performance, and specific comments germane to the particular laboratory. An evaluation by laboratory with specific comments for this study follows:

SUMMARY OVERVIEW OF INTERLABORATORY STUDIES

In closing it was noted that Data Quality Work Group had evolved rather well in developing its interlaboratory program over the previous two years. Emphasis was made on the constructive concepts of these studies and it was recommended that such quality control programs by the principal agencies be maintained.

Insofar as a negative overview, it was noted that some laboratories do have their share of difficulties and that although some laboratories were not able to participate when provided samples, this situation was improving.

Although not necessarily valid for all jurisdictions, it was noted that rapport between the analyst, the users of data and the program managers could be improved.

On the positive frame it is encouraging to recognize that a number of the laboratories within the Surveillance Program have consistently provided data in interlaboratory studies that have been unbiased and frequently unflagged for a majority of constitutents. In an equally positive tone it was noted that some laboratories have shown improvements over the past two years and several laboratories (excellent and those less well endowed) have acknowledged the DQWG effort as beneficial. Also noted was the existence of an improving referral process between the excellent laboratories and those performing poorly.

Although not specifically expressed in the closing comments to the interlaboratory program it is the Chairman's personal opinion that before a laboratory produces data to support the Great Lakes International Surveillance Program, it should be made necessary that it demonstrate that its measurement process is in control (in-lab) and that the data so produced is suitable for the needs of the users of data and meets the objectives of the Surveillance Program and the managers who oversee them. The issue of control in advance can be initially a hard pill to swallow in the area of "expensive" data such as for toxic organics but it is felt this strategy is necessary if year to year or lab to lab bias is to be controlled and the work cost effective. Interlaboratory studies carried out before and during the field season are also recommended. Inherent in the above comment is the need for closer liaison between analysts, the management overseeing the program, and the users of data (within or between jurisdictions).

• GUIDELINES FOR CONTROL OF ANALYTICAL PROCEDURES IN AN INTRALABORATORY QUALITY CONTROL PROGRAM

By J. L. Clark
International Joint Commission
Windsor, Ontario

Scope:

The following guidelines are applicable to laboratories for all data provided to the International Great Lakes Surveillance Program.

Assumptions:

1. The analytical methods used are appropriate for the surveillance tasks; they are essentially bias free, are capable of being brought into a state of statistical control at the precision required, and have adequate sensitivity to analyze environmental samples at the levels of interest.

2. Quality assurance procedures for field operations such as sample collection, container selection, preservation, transportation and storage have been satisfactorily implemented and are therefore not addressed herein. However, for ship laboratories and other field laboratories, it is understood that intralaboratory quality control may include analysis of field blanks and field duplicates.

3. The laboratory has designated the person or persons responsible for quality control together with development of an adequate reporting system such that the laboratory director and any other senior managers are kept apprised of the laboratory's performance and can substantiate it.

General Considerations:

Any analytical procedure that is in a state of statistical control will have an inherent variability as one of its characteristics. For a given procedure this variability is irreducible, that is, there is no identifiable factor which contributes to procedure variation (no assignable cause).

The measure of procedure variability which we will use is the estimate of the population standard deviation. The specific population of interest is the population of between run analyses; between run analyses are chosen rather than within run analyses because we are interested in monitoring performance across runs. However, with highly labile constituents it may be necessary to use an estimate of the standard deviation of the population of within run analyses.

To obtain a reliable initial estimate of the population standard deviation 40 to 50 data are needed. They may be either duplicates analysed in separate runs or analyses of a stable standard in separate runs; examples of both will be given. Highly labile constituents may, however, require an estimate based on duplicates analysed in the same run.

Once the estimate is obtained, control limits can be set for the analytical procedure which, if exceeded, indicate that the procedure is probably out of control. The control limits are commonly set at 3 standard deviations (3σ limits). These limits imply an $\alpha = 0.0027$ or about 3 chances in 1000 of judging an in control procedure to be out of control.

Control limits are generally incorporated into control charts which provide an immediate visual record of performance. If a procedure goes out of control, the point(s) at which control is lost can be easily identified.

Two types of control charts can be differentiated: those that monitor accuracy and those that monitor precision. An example of the former is a chart that monitors results on a known, stable standard; violation of a control limit indicates that the analytical procedure is not producing accurate results. The difficulty may be due to bias, may be due to a loss of precision, or may stem from a combination of the two. An example of the latter is a chart that monitors the range of duplicate analyses on a sample whose value is unknown; violation of a control limit indicates that precision has been lost. However, information regarding possible bias is not provided either by control limit violation or the lack thereof.

Estimating Analytical Procedure Variability:

The essential first step in developing a control system for an analytical procedure is to acquire a sound estimate of procedure variability when the procedure is in a state of statistical control. Once the estimate has been obtained, it can be used to set control limits for the monitoring of both accuracy and precision.

Example 1 - Using Duplicates

Consider the following 50 pairs of results, in µg/L, on duplicates which were analysed in different runs.

1st Result	2nd Result	Range	1st Result	2nd Result	Range
50	46	4	39	42	3
37	36	1	25	25	1
22	19	3	20	18	2
17	20	3	12	10	2
32	34	2	28	32	4
46	46	0	35	40	5
26	28	2	22	22	0
26	30	4	26	25	1
61	58	3	41	40	1
44	45	1	20	21	1
40	44	4	22	40	18
36	35	1	37	35	2
29	31	2	29	26	3
26	38	12	34	35	1
36	36	0	17	19	2
47	45	2	43	44	1
16	20	4	56	53	3
18	21	3	30	32	2
26	22	4	20	21	1
35	36	1	36	32	4
26	25	1	43	39	4
49	51	2	22	21	1
33	32	1	35	36	1
40	38	2	53	50	3
16	13	3	47	47	0

Two of the ranges obtained, 12 and 18, strongly suggest that the analytical system was out of control; these two values are discarded. The remaining 48 ranges are summed and the average range, $\bar{R}$, found.

$$4 + 1 + 3 \ldots + 3 + 0 = 101$$
$$\bar{R} = 101/48 = 2.104$$

An estimate of the standard deviation, s, is obtained from the average range of duplicate analyses by dividing by 1.128, the proper factor for acquiring a standard deviation estimate from ranges derived from duplicates.

$$s = \frac{2.104}{1.128} = 1.865 \text{ µg/L}$$

Example 2 - Using a Stable Standard

Consider the following 50 results, in µg/L, obtained by analysing a stable standard in separate runs.

35.1	31.8	36.4	33.8	33.0	34.2
33.2	35.0	32.1	34.3	37.2	33.7
33.7	31.4	24.7	32.9	34.3	33.9
35.9	35.6	38.2	34.2	32.7	35.6
33.5	30.2	33.1	35.6	34.1	40.1
34.5	32.7	34.9	31.5	35.8	34.6
34.4	31.1	36.2	36.4	33.9	33.8
49.6	34.8	34.0	32.6	35.5	33.0
34.3	35.3				

Mean of all values = 34.368
Mean of 48 values (omitting 24.7 and 49.6), $\bar{x}$ = 34.252083

The two values 24.7 and 49.6 clearly indicate that the procedure was out of control; they are discarded. The value 40.1 is marginal and represents a more difficult decision; in this example it is left in, provisionally.

The estimate of the standard deviation, s, is obtained in the usual way.

$$s^2 = \frac{\Sigma X_i^2 - N\bar{X}^2}{N-1}$$

$$s^2 = \frac{56{,}470.35 - 48\ (34.252083)^2}{47}$$

$$s^2 = 3.32978$$

$$s = 1.825\ \mu g/L \quad \text{(provisional value, see Example 7 below)}$$

If the two omitted values had been included in the calculation, the estimated standard deviation would have been a badly inflated 3.138 µg/L.

It should be noted that s is expressed in absolute rather than relative terms. If variability were proportional to concentration, then the relative standard deviation (coefficient of variation) would be appropriate, but we are not aware of any analytical procedures so characterized. It appears that for any given practical working range variability may be treated as a constant with minimal ill effects. However, if very different ranges are employed to determine the same constituent an estimate of the standard deviation will be required for each range. One would not expect the variability which characterizes analyses in the range 0-100 µg to also pertain to analyses in the range 0-10 mg.

As additional data are obtained initial estimates of variability can be put on a sounder footing by pooling with estimates from the new information, assuming that no substantial change is apparent. If a procedure's variability appears to change significantly, the procedure should be carefully reviewed to ascertain the cause.

The following method may be used to test for change in procedure variability.

Example 3 - Testing for Change in Variability

Suppose an initial estimate of an analytical procedure's standard deviation is obtained, s_1 = 1.796 µg/L, based on a data set of 61 items and therefore having associated with the estimate 60 degrees of freedom. A new estimate, s_2 = 2.145 µg/L, is then obtained based on 41 additional measurements, and thus having 40 degrees of freedom.

The ratio of the two estimates of the <u>variance</u> is found,

$$\frac{s_1^2}{s_2^2} = \frac{1.796^2}{2.145^2} = \frac{3.225616}{4.601025} = 0.701$$

and the ratio compared to appropriate values of the F distribution.

Testing at an α-level = 0.05, the appropriate upper value is simply the tabulated value for the upper 2.5% point of the F distribution with 60 and 40 degrees of freedom; this tabulated value is 1.80. Obtaining the appropriate <u>lower</u> value requires a little arithmetic. The tabulated value for the upper 2.5% point of the F distribution with 40 and 60 degrees of freedom (note the reversal) is found and its reciprocal taken, 1/1.74 = 0.575, to give the required value.

Since the ratio of the two estimates of the analytical procedure variance, 0.701, lies between the values 0.575 and 1.80, we would <u>not</u> conclude that the variability of the procedure had changed.

This test differs from the usual F test in that it is two-tailed, there being no a priori reason for assuming that one variance estimate would be greater than the other.

When it appears that the variability of an analytical procedure has not changed, a pooled estimate of variability may be obtained as follows:

Example 4 - Pooling Estimates of Variability

The pooling method consists of weighing the two <u>variance</u> estimates by the size of the respective data sets from which they were obtained, summing the weighed variance estimates, and dividing the sum by the sum of the degrees of freedom associated with the two estimates. The quotient which results is the pooled variance estimate, s^2, from which the new, pooled estimate of the standard deviation, s, is obtained.

Using the data of Example 3 we have

$$s^2 = \frac{61(1.796)^2 + 41(2.145)^2}{60 + 40}$$

$$s^2 = \frac{196.7626 + 188.6420}{100}$$

$$s^2 = 3.854$$

$$s = 1.963 \text{ µg/L}$$

When a pooled estimate of the procedure standard deviation is obtained, new control limits should be calculated using the revised estimate.

Setting Control Limits:

There are two goals in setting control limits. They should be close enough to signal when there is trouble with a system, and they should be distant enough to discourage tinkering with a system that is operating within its capabilities. Since these two goals are antithetical, a compromise is necessary. The compromise which has been found satisfactory in a great many applications is the use of 3σ control limits, and they are illustrated here.

Example 5 - Use of a Known

A known sample whose concentration is 32.7 µg/L is analysed by a procedure whose estimated standard deviation is 2.131 µg/L. The control limits are 32.7 ± 3 x 2.131 or 26.31 and 39.09. Assuming that results can be read to tenths of a microgram, a result ≥26.3 and ≤39.1 is judged acceptable.

Example 6 - Use of an Unknown Duplicate

An unknown duplicate sample is analysed in separate runs by a procedure whose estimated standard deviation is 1.537 µg/L. The control limit for the range of the two analyses is 1.537 x 3.686 or 5.67; 3.686 is the proper factor for duplicate ranges. Assuming that results can be read to tenths of a microgram, a pair of results whose range is ≤5.7 is judged acceptable.

Example 7 - Correcting an Initial Estimated Standard Deviation

In Example 2 the value 40.1 µg/L was provisionally allowed to remain in the data set for which an estimated standard deviation of 1.825 µg/L was obtained. We now determine whether the 40.1 should remain in the data set.

From the results of Example 2 we can calculate the 3σ control limits 34.252 ± 3 x 1.825 or 28.8 and 39.7.

Since 40.1 is larger than the upper control limit 39.7, there is sufficient evidence to discard this value also.

The estimate of the standard deviation is now recalculated from the 47 item data set to give s = 1.626 µg/L. The new sample mean is 34.128, resulting in new control limits of 29.3 and 39.0 which encompass the 47 values remaining in the data set.

Example 8 - A Special Case, Use of Recovery Data

The use of recovery data for control purposes presents some special problems which are dealt with in this example. We begin with estimation of the variability associated with the determination of recoveries.

Consider the following data set, values in mg/L:

1. Spiked Recovery	2. Unspiked Result	3. Apparent Recovery	4. True Spike	5. Deviation From Expected	6. % Recovery
1.91	0.68	1.23	1.30	-0.07	94.615
1.78	0.57	1.21	1.30	-0.09	93.077
1.53	0.23	1.30	1.30	0	100
1.74	0.15	1.59	1.30	0.29	122.308
2.10	0.53	1.57	1.30	0.27	120.769
1.82	0.61	1.21	1.30	-0.09	93.077
2.07	0.54	1.53	1.30	0.23	117.692
1.39	0.14	1.25	1.30	-0.05	96.154
1.16	0.20	0.96	1.30	-0.34	73.846
1.55	0.19	1.36	1.30	0.06	104.615
2.02	0.41	1.61	1.30	0.31	123.846
1.58	0.36	1.22	1.30	-0.08	93.846
13.01	11.97	1.04	1.30	-0.26	80
1.46	0.17	1.29	1.30	-0.01	99.231
1.63	0.31	1.32	1.30	0.02	101.538
11.95	10.98	0.97	1.30	-0.33	74.615
1.68	0.27	1.41	1.30	0.11	108.462
1.83	0.47	1.36	1.30	0.06	104.615
1.62	0.43	1.19	1.30	-0.11	91.538
5.04	3.96	1.08	1.30	-0.22	83.077
2.53	1.22	1.31	1.30	0.01	100.769
2.69	1.09	1.60	1.30	0.3	123.077
1.50	0.25	1.25	1.30	-0.05	96.154
2.73	0.24	2.49	1.30	1.19	191.538
2.86	0.23	2.63	1.30	1.33	202.308
1.77	0.51	1.26	1.30	-0.04	96.923
1.88	0.55	1.33	1.30	0.03	102.308
0.90	0.57	0.33	1.30	-0.97	25.385
2.22	0.95	1.27	1.30	-0.03	97.692
1.99	0.85	1.14	1.30	-0.16	87.692
1.54	0.26	1.28	1.30	-0.02	98.462
1.47	0.15	1.32	1.30	0.02	101.538
1.43	0.09	1.34	1.30	0.04	103.077
1.65	0.35	1.30	1.30	0	100
1.91	0.68	1.23	1.30	-0.07	94.615
2.06	0.93	1.13	1.30	-0.17	86.923
5.24	4.02	1.22	1.30	-0.08	93.846
1.58	0.27	1.31	1.30	0.01	100.769
1.63	0.28	1.35	1.30	0.05	103.846
1.52	0.23	1.29	1.30	-0.01	99.231
1.70	0.35	1.35	1.30	0.05	103.846
1.77	0.31	1.46	1.30	0.16	112.308
1.93	0.49	1.44	1.30	0.14	110.769
2.30	1.13	1.17	1.30	-0.13	90

In column 5 there are 3 deviations from expected recoveries which appear extreme: 1.19, 1.33 and -0.97; these results are discarded. From the remaining 41 results in the 5th column of the data set an estimate of the standard deviation of the spiking recovery procedure is calculated in the usual way and found to be s = 0.1532 mg/L. (Since the deviations from expected results represent the difference between two analytical determinations, we would expect the standard deviation of the spiking recovery procedure to be greater than the standard deviation of a single determination by a factor of $\sqrt{2}$.)

The mean of the deviations from the expected results is -0.0061 mg/L. Since the absolute value of this mean is less than the standard error of the mean of the spiking recovery procedure, s_m (= $0.1532/\sqrt{41}$ = 0.024 mg/L), the spiking recovery procedure appears to be unbiased with complete recovery a reasonable expectation. Control limits may therefore be set around the expectation of complete recovery with allowable deviations of $0 \pm 3 \times 0.1532$ or -0.46 mg/L and 0.46 mg/L. The remaining 41 members of the data set are all within these limits.

Had the spiking recovery procedure demonstrated a bias, the control limits would have been calculated from the estimate of the bias.

In this example the data in column 6 may be used to obtain equivalent control limits in terms of percent recovery. With the omission of the 3 questionable results, the estimate of the standard deviation of the spiking recovery procedure is 11.782% on a spike of 1.3 mg/L; 11.782% of 1.3 mg/L is 0.1532 mg/L, which is the same estimate as obtained from column 5. However, the equivalency holds because identical spikes were employed in all recoveries. If variable spikes are used, then the estimate of the standard deviation and the ensuing control limits must be made in absolute units such as mg/L and not in percent recovery.

Frequency of Control Sample Use:

The following minimum frequencies are recommended for the use of control samples:

To monitor accuracy, 1 quality control sample of known value should be included with every 15 analyses or with each batch, whichever results in the greater frequency.

To monitor precision, 1 quality control sample should be included with every 15 analyses or with each batch, whichever results in the greater frequency. If duplicates are used to monitor precision, they should be analysed in different runs when a between run measure of variability is employed in setting control limits.

Concluding Remarks:

The control charts which employ control limits as illustrated above are examples of Shewhart control charts. The reference used in preparing these guidelines is:

ASTM Manual on Presentation of Data and Control Chart Analysis
ASTM Special Technical Publication 15D, 1976

The factors 1.128 and 3.686 used in examples 1 and 6, respectively, were taken from this manual.

Recognition that problems exist is, of course, but the essential first step toward their solution; one authority on quality control has suggested that it represents only 10% of the effort which will be required. For the intermittent problems which often occur in analytical chemistry, the identification of causes will typically be arduous. For such recurrent problems, careful record keeping will be required to determine whether rates of occurrence have in fact diminished when putative causes are addressed.

• TASK GROUP ON MAJOR IONS, NUTRIENTS & PHYSICAL MEASUREMENTS IN WATER

Chairman: C. Ross

Delivered Report By Secretary: J. Peck

Twenty-six people participated in the Major Ions, Nutrients and Physical Measurements in Water task group meeting. No papers were presented for distribution.

- Round Robin Design Problems

Design problems with past interlaboratory studies conducted by the Data Quality Work Group were identified: some samples have been much too high for normal operating conditions thereby causing large dilutions to be performed while other samples were frequently lower than present laboratory capabilities necessitating non-positive results and many W and T codes due to the variety of laboratories which participate. The variety of such laboratories spans open water, nearshore, tributary, and point source discharge analyses.

The Data Quality Work Group has tried to supply a variety of samples and constituent levels in each study to meet the needs of the laboratories. Because of this the above described problems are noted.

- Study #27 - Associated Problems

Study #27, Major Ions and Nutrients in Water, was discussed. The samples consisted of rain water, open lake water, harbor, tributary, reference, and standard sources; lead to the following expressed conclusion: the attempt to have some samples for every type of laboratory resulted in inappropriate samples for all. However, it was suggested that this problem could be solved by identifying sample (type) source with a preliminary information package so that a laboratory might analyze its normal type sample and code other samples as inappropriate.

Laboratories may have more than one analytical method depending on sample source and therefore apply a different instrument manifold, glassware, or apparatus. Some laboratories need versatile methods because of the range of programs they are responsible for supporting, and IJC participation may be as low as 1% of their total laboratory workload.

- Phosphorus

The relative merits of using ascorbic acid versus stannous chloride were discussed. The ascorbic acid reduction procedure is the U.S. EPA approved

procedure. Deterioration of reducing reagents, particularly stannous chloride, was pointed out. Stannous chloride reagent provides a 5 to 10-fold increased sensitivity for the measurement of phosphorus, about 0.2 µg/L vs. 1 or 2 µg/L.

Some chemists stated that their laboratories performing the analyses were unaware of the purpose of various programs including IJC surveillance work and therefore:

- o there is a need for better program planning by communication including a description of needed method sensitivity, accuracy, precision, and so on;
- o needs should be evaluated before many resources are spent rather than collecting reams of worthless data; and
- o usefulness of data must be established with respect to program needs and laboratory capability.

It was felt that if possible program planners should identify need before a project is initiated so that adjustments could be made. Also, some constituent levels, particularly in Lake Superior, are so low that they are nearly impossible to measure by present technology.

Several attendees expressed concern over the Data Quality Work Group's intention to report performance by laboratory to the Chairman of the Surveillance Subcommittee.

Many viewed participation in Data Quality Work Group round robin studies primarily as an education process for its own value in that they:

- o explain the need of surveillance to know which laboratories are probably supplying data which would be compatible and from which accurate loading and trend analysis could be evaluated;
- o may assist poor performing laboratories in identifying the cause of poor performance; and
- o identify if additional laboratory personnel, equipment or better methods are needed.

- Laboratory to Sample or Sample to Laboratory?

For non-conservative constituents the laboratory should be brought to the sample, such as ship-board analyses for open lake work.

For toxic materials the safety of the field personnel is paramount and therefore a mobile laboratory should be used to conduct preliminary analyses so that proper precautions can be taken.

- Contracting Out Laboratory Work?

It takes at least a year to build up analytical expertise, then frequently the contract expires. Attendees expressed there should be:

- o no blanket contracts;
- o a need identification before initiation;

- o a thorough review of a laboratory's capability, personnel; and
- o an evaluation sample set to identify which laboratories are competent and might bid.

- Laboratory Automation

Various computer printouts were supplied by Messrs. Philbert (CCIW) and Ross (U.S. EPA). Automated laboratory analysis was discussed. CCIW and EPA are using automated systems for data collection, on-line instruments, and data management. Ontario Ministry of the Environment has a data management system (Mr. Rawlings). Mr. Tupy of Minnesota Department of Health, described their system.

The benefits of automated systems are considered to be:

- o cost saving;
- o elimination of errors;
- o improved quality control; and
- o more time for the chemist to be a chemist.

However, in placing such systems in laboratories, some draw-backs have been noted; they follow:

- o possible poor acceptance for change; and
- o unfamiliar with computers due to lack of exposure.

The draw-backs are being overcome and computer systems for analytical work are being introduced even at the college level.

- Chlorophyll Analyses

The merits of various glass and membrane filters were discussed. The use of acetone vs. methanol for extraction, and the grinding of sample vs. no grinding vs. ultrasonic destruction were discussed.

DMSO was described as an excellent solvent because it provides good filter dissolution. However, special handling is required since it is absorbed through the skin.

- Round Robin Samples Treated Differently?

It was suggested that round robin samples should be handled identically as for others, resulting in a typical report neither emphasizing or lowering priority on W & T codes. Further, the possibility of a one week analytical period for the round robin samples to be completed by all laboratories was agreed to be desirable but not practical.

Several chemists suggested that a previous notification of samples being sent would be helpful so that they could be planned into the analytical system. The notification should include expected date of shipment, the kind of preservation used, and recommend holding times not to be exceeded. The Task Group Chairman commented that the samples distributed for nutrients were generally already 2 years old, unpreserved, and therefore should cause little concern for preservation techniques and sample constituent changes over time.

- Measurement of Total Kjeldahl Nitrogen

Various digestion techniques for TKN measurements were discussed. Some analysts use $K_2S_2O_8$ others K_2SO_4 or K_2SO_4, with HgO as catalyst. Contaminants within reagents were noted, particularly $K_2S_2O_8$, and contaminated reagents should be returned to the supplier.

Most agreed that particulate TKN should be measured separately from dissolved.

Topics brought up but not discussed:

- Sediment analytical reproducibility problems mostly related to analytical technique. This underlines the need for standard methodology applicable to sediment analyses.

- Use of ICP for metal analyses and data comparability problems

- Sample volume in IJC Studies insufficient

- Use of ion chromatography

• TASK GROUP ON METALS IN WATER, SEDIMENT, AND BIOTA

Delivered Report by Chairman: B. Loescher
Secretary: J. Clark

The Task Group Subgroup consisted of approximately 35 participants. Over the four hour discussion period certain common themes emerged.

- Specific analytical problems
- Interlab comparisons
- Low level metals analysis
- Sediment analysis

1. Specific problems were discussed relating to arsenic and selenium analyses, vanadium by graphite furnace, anomalously high copper values, etc. There was sufficient expertise within the group to provide plausible solutions to most difficulties. Exchange of methodologies was often the most ready solution. Several participants requested the CCIW Analytical Methods Manual and the new EPA procedures on bottom sediment and elutriate testing. The EPA procedure can be obtained from:

   NTIS, U.S. Department of Commerce
   5285 Fort Royal Road
   Springfield, VA 22161

   Order by asking for

   Chemistry Laboratory Manual for Bottom Sediments and Elutriate Testing
   Author: U.S. EPA
   #EPA 905-4-79-014, PB-294 596/2 WP. Cost $8.00 (U.S. Funds).

2. Almost all participants agreed to the usefulness and/or necessity of round robin studies. There were criticisms of the laboratory ranking scheme in that laboratories with poor detection limits might not be flagged while laboratories with more sensitive methodologies might be. Some participants related ensuing difficulties with their management as a result of flags. Keijo Aspila suggested that poor or inadequate performance might be used as a lever to obtain better equipment. It was also agreed that an indication of analytical technique to accompany the data was also necessary and in keeping with the concept of intercomparisons as an information tool. The enclosure of a vial of concentrate standard to accompany the regular samples was requested as a distilled water check.

3. It was agreed that the extremely low levels of metals in most Great Lakes watersheds presented major analytical and data evaluation problems.

   Acid washed glassware, laminar flow hoods, and if possible clean rooms are necessary parts of any analytical preparation. Various preconcentration procedures (solvent extraction, ion exchange) are being evaluated as a means of achieving adequate sensitivity to measure ambient levels in the Great Lakes.

   Currently, most data is at or below detection limits for most elements which is unsatisfactory for data evaluation. John Clark stated that reporting all results, including negative numbers, would be superior to reporting either "zero" or "less than."

   All data below the laboratory detection limit would be designated with a "T" code and negative results would be reported. This would hopefully eliminate much of the high biases currently encountered in the analysis of large data blocks.

   Identical preparation procedures for water samples are unlikely because of the need to tailor the preparation to the analytical finish. Different laboratories use different analytical techniques. Preconcentration-flame atomic absorption, flameless atomic absorption, preconcentration-inductively coupled plasma emission spectroscopy, and anodic stripping voltammetry being the most common.

4. Almost every analyst used a different preparative technique for sediment analysis and acknowledged that different results are to be expected for certain elements such as aluminum and titanium. There was consensus that common preps that would provide consistent interlab data were required and that some client as well as analyst responsibility was required. A sediment intercomparison to address the above was designed. There will be 10 samples, 6 dry and 4 wet. One would be the NBS standard sediment. They would contain a range of clay and organic contents and would be analyzed by a specified leaching technique, a "total" procedure and the laboratory's routine method. Various agencies agreed to provide samples.

As an overview most participants felt that there had not been enough time to adequately discuss methodologies and mutual interest, but that contacts made would be helpful for future reference.

• TASK GROUP ON ORGANICS IN WATER, SEDIMENT, AND BIOTA

Delivered Report By Chairman: J. Daly
Secretary: R. E. White

There were approximately 50 people attending the Task Group meeting. Interests varied, with the majority citing the analysis of chlorinated hydrocarbon pesticides, PCBs, PAHs, and purgeable organics to be the major concern, within general trace organics.

Methodology was initially discussed in general terms. Some inquired about what approved methods were available. It was pointed out that U.S. EPA has promulgated methods for water and wastewater. While methods for other matrices such as fish, wildlife and sediment samples are suggested methods, available from U.S. EPA, FW&S, and FDA, and from the Canadian Wildlife Service.

Further, the U.S. EPA has published within the Federal Register, Dec. 3 and 18, 1979, proposed methods and identifies about 20% of laboratory efforts to be devoted to quality assurance matters.

Then the discussion turned to recoveries and what is an acceptable recovery from a data user's point of view. It was mentioned that recoveries anywhere from 50% for herbicides in water to 95% for pesticides in water are routinely achieved. For HCB and other similar materials "keepers" have been used to enhance recovery.

The question of how to handle recoveries was then brought up. It was pointed out that in the case of organic analysis data are not usually corrected for recovery, whereas results for most inorganic or trace metal procedures account for recovery losses in the reported value. To complicate matters further recoveries for water and recoveries in matrices such as fish and sediment don't usually mean the same thing.

In general it was obvious that there is no consistent policy for handling recovery in organic analyses. In some cases a recovery figure may be reported with result, results in some instances may be corrected for losses or no effort may be made to indicate recovery.

I think you can begin to appreciate some of the problems just associated with chemical recoveries for organic analyses relative to other analytical procedures.

The session yesterday afternoon closed on that note after some reprints and papers of interest were distributed to the attendees.

This morning we tried to get into some specific areas. We started off with a discussion of capillary column GC analysis. Several of the attendees were working in this area while others were in the process of buying equipment for this type of analysis. There was considerable interest in this area and the pros and cons of capillary analysis were extensively discussed.

For those of you who aren't familiar with capillary GC analysis relative to packed column GC analysis, the resolution or separation of peaks is much greater on a capillary column. When you might have 15 or 20 peaks on a packed column, a capillary column will yield 120 peaks or more. Messrs. Sturino, Won, Onuska, and Mullin, among others, use capillary column for much of their work.

A concern which was voiced was that the capillary column would decrease productivity because of the longer elution times. It was pointed out that this was not always the case and even then the selectivity and increased sensitivity offset the longer running time. Some have shortened analysis time through using shorter columns - about 15 meters. However, some reported less precision using capillary columns.

It was kind of summed up by the comments of one of the attendees that once you went to a capillary column you would never go back to a packed column. Fused silica glass capillary columns were highly recommended because of their flexibility. Also for mass spectrometery work one can go directly into the ion source.

Then there was a short discussion on toxaphene analysis and some of the problems associated with the cleanup and quantification of toxaphene. It was pointed out that the capillary column solved some of the problems of quantifying toxaphene but for PCBs one finds it a difficult choice to report individual isomer or total. Finally it was pointed out that toxaphene is probably going to be more important then the PCB problem since PCB levels are on the decrease.

There was a fairly lengthy discussion on the relative merits of the various extraction procedures for water, fish and sediment. The most general extraction techniques were Soxhlet, blend, shakers, and column elution. Several methods were discussed and the importance of depicting the relative recovery for each of the methods stressed.

From these discussions, it became apparent that a standard reference material for fish tissue and sediment would be extremely useful in comparing extraction procedures.

The discussion next turned to quality control. Several attendees described the quality control procedures used in their lab. The use of reference materials and the analysis of duplicates seemed to be the most common quality control measures. Spiked recoveries were also used in many cases for water, fish and sediment. Some of the laboratories used primarily system quality control activities, e.g. things like linearity check.

Finally we had a brief discussion on the problem, peculiar to mass spectrometery analysis. There was some interest in what others in the field were doing particulary regarding the storage of data for unidentified pecks and the need to circulate information among analysts on unknown compounds found.

In closing we discussed ways we could improve future meetings. It was pointed out that concentrating on specific areas of interest in depth would be more useful to most people.

• CONCLUDING COMMENTS
By K. I. Aspila

The Chairman, in his closing comments, noted that the Data Quality Work Group had evolved rather well over the previous two years and it is now constructively providing valuable feedback to the analysts and the present and future users of data. Regarding the analysts meeting, he noted that it was indeed a successful exercise for many analysts and that analysts are recognizing why excellent data are essential for the Surveillance Program and how they and the Work Group inter-relate to constructively identify and improve quality in the analytical measurement systems.

The Chairman also reminded analysts that the Work Group would appreciate receiving from them a copy of a précis of their intralaboratory quality control measures that they currently utilize when supporting the Surveillance Program. He also reminded analysts they should review and comment on the intralaboratory quality control procedure guidelines presented during the analysts' meeting. Establishing evidence of control, prior to initiating analysis of routine surveillance samples, was noted as essential even if it is initially expensive. To begin an analysis program when it is not known that control exists in the measurement system is unwise as it can lead to significant embarrassements when the final data are reviewed by data users or the laboratory is evaluated with negative comments through interlaboratory testing procedures such as those provided by the Data Quality Work Group.

Regarding future interlaboratory studies, the Chairman was unable to confirm specific dates and specific studies. He did indicate a total phosphorus, total trace metals and major ion studies might be delivered in the spring and summer portions of the 1980 field program. The high level of interest in establishing interlaboratory comparability in metals in sediment had been expressed in the metals task group session. A number of analysts volunteered assistance in providing sediments. Cooperation is essential and with management support in the respective jurisdictions the cost sharing will improve the international effort in data quality assurance. The sediment study (metals) may possibly be distributed in the fall of 1980.

The intriguing matter of having laboratories recognize that negative concentrations may now need to be implemented was raised again. It was expressed as a natural followup from the successful application of "W" and "T" coding and that although this concept is initially difficult to appreciate, it will when implemented, have significant impact for the users of data.

The Chairman then thanked all the analysts for their contribution to the meeting.

APPENDIX I

SURVEILLANCE SUBCOMMITTEE
DATA QUALITY WORK GROUP

Mr. Keijo I. Aspila (Chairman)
Head, Quality Control and Special Analysis Laboratory
Special Services Section, Ontario Region
Inland Waters Directorate
Environment Canada
P. O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4645 - Lab.
(416) 637-4638 - Office

Mr. James H. Adams, Jr.
Head, Quality Assurance Office (5SQA)
U.S. Environmental Protection Agency
Region V
Surveillance and Analysis Division
536 South Clark Street
Chicago, Illinois 60605
(312) 353-9604 (FTS) 353-9604

Mr. J. N. Bishop
Assistant Director
Laboratory Services Branch
Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3346

Mr. James C. Daly
Quality Assurance Director
New York State Dept. of Health
Tower Building, Room D527
Empire State Plaza
Albany, New York 12201
(518) 474-7999 (FTS) 564-7999

Dr. M. D. Mullin
Chemist Manager
Large Lakes Research Station
U.S. Environmental Protection Agency
9311 Groh Road
Grosse Ile, Michigan 48138
(313) 226-7811 (FTS) 226-7811

Mr. John H. Peck
Environmental Laboratory
Michigan Department of Natural Resources
315 North Clippert Street
Lansing, Michigan 48912
(517) 373-2197 (FTS) 253-2197

Mr. C. Ross
Director
Central Regional Laboratory
U.S. Environmental Protection Agency
Region V
536 South Clark Street
Chicago, Illinois 60605
(312) 353-8370 (FTS) 353-8370

Secretariat Responsibilities

Mr. R. E. White
Senior Scientist
Great Lakes Regional Office
International Joint Commission
100 Ouellette Avenue, 8th Floor
Windsor, Ontario N9A 6T3
(519) 256-7821 - Windsor
(313) 963-9041 - Detroit

APPENDIX II

LIST OF INVITEES

Dr. B. K. Afghan
Head
Analytical Methods Research Section
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4661 (O)*

Mr. Haig Agemian
Water Quality Branch
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4557 (M)**

Mr. Harry Alkema
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (M)

Dr. David E. Armstrong
Water Chemistry Programs
University of Wisconsin-Madison
Madison, Wisconsin 53706

Dr. Eugene J. Aubert
Director
Great Lakes Environmental Res. Lab.
National Oceanic & Atmospheric Admin.
2300 Washtenaw Avenue
Ann Arbor, Michigan 48104

Mr. Keith Austen
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (M)

Dr. Jim Bedford
Michigan Department of Natural Resources
315 North Clippert Street
Lansing, Michigan 48912
(517) 373-2197 (FTS) 253-2197 (O)

Mr. Don Bennie
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (O)

Mr. Dale S. Boshart
Division of Laboratories and Research
N.Y. State Department of Health
Empire State Plaza
Albany, New York 12201
(518) 474-4934 (M)

Mr. R. J. Bowden
Chief, Surveillance & Research Staff
Great Lakes National Program Office
U.S. Environmental Protection Agency
536 South Clark Street
Chicago, Illinois 60605

Mr. Heinz E. Braun
Provincial Pesticide Residue Testing Lab.
Ontario Department of Agriculture and Food
c/o University of Guelph
Guelph, Ontario N1G 2W1
(519) 824-4120 (O)

Mr. Patrick G. Calway
Fisheries Service
Department of Fisheries and Oceans
590 Keele Street, 4th Floor
Toronto, Ontario M6N 3E3
(416) 763-1161 (O)

---

*(O) Task Group Participants - Organics in Water, Sediment, and Biota
**(M) Task Group Participants - Metals in Water, Sediment, and Biota
***(INP) Task Group Participants - Major Ions, Nutrients & Physical Measurements in Water

Mr. John M. Carron
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (O)

Mr. Matthew Caruso
Division of Laboratories & Research
New York State Department of Health
Central New York Laboratory
677 South Salina Street
Syracuse, New York 13202
(315) 473-8351 (INP)***

Mr. Alfred S. Y. Chau
Water Quality Branch
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4653 (O)

Dr. Ven Cheam
Water Quality Branch
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4645 (M)

Dr. John Christison
Pesticides & Trace Analytical Laboratory
Ontario Research Foundation
Sheridan Park Research Community
Mississauga, Ontario L5K 1B3
(416) 822-4111 (M)

Dr. John L. Clark
Senior Statistician
Great Lakes Regional Office, IJC
100 Ouellette Avenue, 8th Floor
Windsor, Ontario N9A 6T3
(519) 256-7821 (M)

Mr. John A. Coburn
Water Quality Branch, Ontario Region
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4660 (O)

Dr. Robert L. Collin
Chief, Toxic Substances Control Unit
Division of Water
NY State Dept. of Environmental Conservation
50 Wolf Road
Albany, New York 12233

Mr. Walter Cook
Ontario Ministry of the Environment
London Regional Laboratory
985 Adelaide Street South
London, Ontario N6E 1V3
(519) 681-3600 (INP)

Mr. Terry Cooper
Pesticides and Trace Analytical Laboratory
Ontario Research Foundation
Sheridan Park Research Community
Mississauga, Ontario L5K 1B3
(416) 822-4111 (O)

Mr. George Crawford
Pesticide Section
Laboratory Services Branch
Ontario Ministry of the Environment
P. O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3743 (O)

Ms. Joan Crowther
Laboratory Services Branch
Water Quality Section, MOE
P.O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3512 (INP)

Dr. Joseph J. Delfino
State Laboratory of Hygiene
University of Wisconsin
465 Henry Mall
Madison, Wisconsin 53706
(608) 262-3458 (FTS) 262-3458 (O)

Mr. Douglas J. Dube
Assistant Section Chief
Wisconsin Department of Natural Resources
State Laboratory of Hygiene
465 Henry Mall
Madison, Wisconsin 53706
(608) 262-3458 (FTS) 262-3458 (M)

Mr. Carlton Duke
City of Chicago
Bureau of Water
Water Purification Division
Central Water Filtration Plant
1000 East Ohio Street
Chicago, Illinois 60611

Mr. Brian J. Eadie
Great Lakes Environmental Res. Lab.
National Oceanic & Atmospheric
Administration
2300 Washtenaw Avenue
Ann Arbor, Michigan 48104

Dr. Steven J. Eisenreich
University of Minnesota
Department of Civil & Mineral Engineering
112 Mines and Metallurgy
221 Church Street, S.E.
Minneapolis, Minnesota 55455

Mr. Omar Elkie
Water Quality Branch
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4643 (INP)

Mr. Chuck Elley
Central Regional Laboratory
536 So. Clark Street
Chicago, Illinois 60605
(312) 353-8370 (FTS) 353-8370 (INP)

Mr. David E. Erdmann
National Water Quality Laboratory-Atlanta
U.S. Geological Survey
6481 Peachtree Industrial Boulevard, Ste. H
Doraville, Georgia 30360

Ms. Ellen Ewing
Water Quality Laboratory
Heidelberg College
Tiffin, Ohio 44883
(419) 448-2998 (M,INP)

Ms. Laura Fay
Center for Lake Erie Area Research
Ohio State University
484 West 12th Avenue
Columbus, Ohio 43210
(614) 422-8949 (FTS) 940-8949 (INP)

Mr. Milton Feldman
Environmental Research Laboratory
U.S. Environmental Protection Agency
200 S.W. 35th Street
Corvallis, Oregon 97330

Mr. David Ferguson
Ministry of Environment
Southeast Regional Laboratory
P.O. Box 820
Kingston, Ontario K7L 4X6
(613) 549-4000 (INP)

Mr. John Filkins
Cranbrook Institute of Science
c/o U.S. EPA
9311 Groh Road
Grosse Ile, Michigan 48138
(313) 226-7811 (FTS) 226-7811 (O)

Mr. Robert L. Flint
Laboratory Coordinator
U.S. EPA, Region II
Rochester Field Office
100 State Street
Rochester, New York 14614

Ms. Glenys Foster
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3755 (O)

Dr. Richard Frank
Ontario Ministry of Agriculture & Food
Pesticide Laboratory
University of Guelph, Building 43
Guelph, Ontario N19 2W1

Mr. R. E. Frazier
Minnesota Department of Health
Section of Health Risk Assessment
and Analytical Services
717 Delaware Street, S.E.
Minneapolis, Minnesota 55414

Ms. Mary Gessner
Center for Lake Erie Area Research
Ohio State University
484 West 12th Avenue
Columbus, Ohio 43210
(419) 285-4754 (O)

Dr. G. E. Glass
Environmental Research Laboratory-Duluth
6201 Congdon Blvd.
Duluth, Minnesota 55804

Mr. Norman R. Glass
Environmental Research Laboratory
U.S. Environmental Protection Agency
200 S.W. 35th Street
Corvallis, Oregon 97330

Dr. Edward Glod
Chief, Environmental Laboratories
Ohio Department of Health
1571 Perry Street
Columbus, Ohio 43210

Mr. D. Glutek
Ontario Ministry of the Environment
London Regional Laboratory
985 Adelaide Street South
London, Ontario N6E 1V3

Mr. David A. Gruber
Water Quality Specialist
Milwaukee Metropolitan Sewerage District
735 North Water Street
Milwaukee, Wisconsin 53202

Dr. D. J. Hallett
Research Scientist
Environment Canada, Ontario Region
National Wildlife Research Centre
Ottawa, Ontario K1A 0E7

Ms. Marilyn Hendzel
Industry Services Branch
Dept. of Fisheries and Oceans
Western Region
Freshwater Institute
501 University Cresc.
Winnipeg, Manitoba R3T 2N6
(204) 269-7379 (O)

Dr. E. Herdendorf
Ohio State University
Center for Lake Erie Area Research
484 West 12th Avenue
Columbus, Ohio 43210

Mr. Robert Hesselberg
U.S. Department of the Interior
Fish and Wildlife Service
Great Lakes Fishery Laboratory
1451 Green Road
Ann Arbor, Michigan 48105
(313) 994-3331 (FTS) 378-1331 (O)

Mr. Robert Hoffman
New York State Dept. of Health
Division of Lab. & Research
Tower Building
Empire State Plaza
Albany, New York 12201
(518) 474-8293 (FTS) 564-8293 (O)

Mr. Sam Jackling
Senior Analytical Chemist
Hale Creek Field Station
NYS Dept. of Environmental Conservation
7235 Steele Ave. Extension, R.D. #2
Gloversville, New York 12078
(518) 773-7318 (O, M)

Mr. Adrian Jackson
Ontario Ministry of the Environment
Northwest Regional Laboratory
421 James Street South
Thunder Bay, Ontario P7E 2V6
(807) 475-1275 (INP)

Mr. Steve Jenkins
Beak Consultants Limited
6870 Goreway Drive
Mississauga, Ontario L4V 1L9
(416) 671-2600 (INP)

Mr. A. F. Johnson
Water Resources Planning Unit
Ontario Ministry of the Environment
135 St. Clair Avenue West
Toronto, Ontario M4V 1P5

Mr. Robert A. Jung
Central New York Laboratory
New York State Department of Health
677 S. Salina Street
Syracuse, New York 13202

Dr. Klaus L. E. Kaiser
Environmental Quality Coordinating Unit
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (O)

Mr. Stephen R. Kin
Director
Water & Sewage Laboratory Division
Indiana State Board of Health
1330 West Michigan Street
Indianapolis, Indiana 46206

Mr. D. E. King
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3015

Mr. S. J. Kleinhert
Chief, Surveillance Section
Department of Natural Resources
P.O. Box 7921
Madison, Wisconsin 53707

Mr. J. Kohli
Beak Consultants Limited
6870 Goreway Drive
Mississauga, Ontario L4V 1L9
(416) 671-2600 (O)

Mr. Jack W. Kramer
Laboratory Manager
Water Quality Laboratory
Heidelberg College
Tiffin, Ohio 44883
(419) 448-2998 (M, INP)

Dr. Joseph Kutkuhn
Director
U.S. Department of the Interior
Fish and Wildlife Service
Great Lakes Fishery Laboratory
1451 Green Road
Ann Arbor, Michigan 48105

Ms. Muriel Lawson
Pesticides & Trace Analytical Laboratory
Ontario Research Foundation
Sheridan Park Research Community
Mississauga, Ontario L5K 1B3
(416) 822-4111 (M)

Mr. Russell Leacock
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (M)

Dr. Bill Lee
Water Quality Branch
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4606 (O)

Mr. Paul Letki
Great Lakes Laboratory
State University College at Buffalo
1300 Elmwood Avenue
Buffalo, New York 14222
(716) 878-5821 (O)

Ms. Julie Letterhos
Center for Lake Erie Area Research
Ohio State University
484 West 12th Avenue
Columbus, Ohio 43210
(419) 285-4754 (INP)

Dr. Wan Chi Li
Barringer Magenta Limited
304 Carlingview Drive
Rexdale, Ontario M9W 5G2
(416) 675-3870 (M)

Ms. Jane Lindsay
Pesticides & Trace Analytical Laboratory
Ontario Research Foundation
Sheridan Park Research Community
Mississauga, Ontario L5K 1B3
(416) 822-4111 (INP)

Mr. Alan J. Lipski
Barringer Magenta Limited
304 Carlingview Drive
Rexdale, Ontario M9W 5G2
(416) 675-3870 (INP)

Mr. Barry Loescher
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1 (M)

Mr. Dave Long
Michigan Dept. of Natural Resources
315 North Clippert Street
Lansing, Michigan 48912
(517) 373-2197 (FTS) 253-2197 (O)

Dr. Larry Ludke
Columbia National Fishery
Research Laboratory
Route #1
Columbia, Missouri 65201

Mr. Andrew Lutz
Analytical Chemistry Group
Freshwater Institute
501 University Crescent
Winnipeg, Manitoba R3T 2N6
(204) 269-7379 (M)

Mr. S. E. MacBeth
Ministry of the Environment
Kingston Regional Office
Southeast Regional Laboratory
P.O. Box 820
Kingston, Ontario K7L 4X6

Mr. Gerald O. Mack
Division of Laboratories
State of Illinois Dept. of Public Health
139 North 19th Street
Springfield, Illinois 62701

Mr. John M. Malczyk
Great Lakes Environmental Res. Laboratory
National Oceanic & Atmospheric Admin.
2300 Washtenaw Avenue
Ann Arbor, Michigan 48104
(313) 668-2235 (INP)

Mr. John Marsden
Chief, Dept. of Environmental Resources
Division of Water Quality
Erie Laboratory
712 Maryland Avenue
Erie, Pennsylvania 16505

Mr. John A. McKinney
Director, Analytical Laboratory
NYS Dept. of Environmental Conservation
Building 47, Rte. 9W
P.O. Box 200
West Coxsackie, New York 12192
(518) 756-2188 (O, M)

Dr. Otto Meresz
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1

Mr. Jerry Mirsch
Ontario Ministry of the Environment
London Regional Laboratory
985 Adelaide Street South
London, Ontario N6E 1V3
(519) 681-3600 (INP)

Mr. Donald A. Muentener
Chief, Laboratory Division
Michigan Department of Agriculture
1615 S. Harrison Road
East Lansing, Michigan 48823

Dr. Thomas J. Murphy
DePaul University
Department of Chemistry
1036 West Belden Avenue
Chicago, Illinois 60614

Mr. Andrew Murray
Barringer Magenta Limited
304 Carlingview Drive
Rexdale, Ontario M9W 5G2
(416) 675-3870 (M)

Mr. R. Nordlund
Michigan Department of Natural Resources
315 North Clippert Street
Lansing, Michigan 48912
(517) 373-2197 (FTS) 253-2197 (M)

Mr. K. A. Oakley
Director
International Joint Commission
100 Ouellette Avenue, 8th Floor
Windsor, Ontario N9A 6T3
(519) 256-7821

Mr. J. Osborne
Pesticide Section
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3743 (O)

Mr. Guy M. Paquette
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (INP)

Mr. Ronald Pause
New York State Dept. of Health
Division of Lab. & Research
Tower Building
Empire State Plaza
Albany, New York 12201
(518) 474-8293 (FTS) 564-8293 (O)

Mr. Harry M. Pawlowski
Engineer of Water Purification
Water Purification Division
City of Chicago
1000 East Ohio Street
Chicago, Illinois 60611

Mr. Art Pethybridge
Great Lakes Laboratory
State University College at Buffalo
1300 Elmwood Avenue
Buffalo, New York 14222
(716) 878-5821 (M)

Mr. Francis J. Philbert
Chief, Water Quality Branch, Ont. Region
Inland Waters Directorate
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4663 (INP)

Mr. Russell H. Plumb
Great Lakes Laboratory
State University College at Buffalo
1300 Elmwood Avenue
Buffalo, New York 14222
(716) 878-5821 (O)

Dr. J. E. Poldoski
Environmental Research Laboratory-Duluth
6201 Congdon Blvd.
Duluth, Minnesota 55804

Dr. Mian A. Rashid
Acres Consulting Services Limited
5259 Dorchester Road
P.O. Box 1001
Niagara Falls, Ontario L2E 6W1
(416) 354-3831 (O, M, INP)

Mrs. Jarka Rasper
Provincial Pesticide Residue
Testing Laboratory
Ontario Department of Agriculture & Food
c/o University of Guelph
Guelph, Ontario N1G 2W1
(519) 824-4120 (O)

Mr. Michael Rawlings
Supervisor, Rivers & Lakes Laboratory
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3512 (INP)

Mr. G. A. V. Rees
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L2

Dr. Lincoln M. Reynolds
Principal Scientist
Pesticide & Trace Analytical Lab.
Department of Applied Chemistry
Ontario Research Foundation
Sheridan Park Research Community
Mississauga, Ontario L5K 1B3
(416) 822-4111 (O)

Dr. Clifford P. Rice
Great Lakes Research Division
The University of Michigan
Institute of Science & Technology Bldg.
Ann Arbor, Michigan 48109
(313) 764-2420 (O)

Dr. R. Peter Richards
Water Quality Laboratory
Heidelberg College
Tiffin, Ohio 448831
(419) 448-2198 (O, M, INP)

Dr. Ronald Rossmann
Great Lakes Research Division
The University of Michigan
Institute of Science & Technology Bldg.
Ann Arbor, Michigan 48109
(313) 764-7527 (M)

Mr. Kenneth Rygwelski
Cranbrook Institute of Science
c/o Large Lakes Research Station
U.S. Environmental Protection Agency
9311 Groh Road
Grosse Ile, Michigan 48138

Mr. Gino Sardella
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (INP)

Dr. G. L. Schelske
Great Lakes Research Division
University of Michigan
2200 Bonisteel Blvd.
Ann Arbor, Michigan 48109

Dr. James G. Seelye
U.S. Department of the Interior
Fish and Wildlife Service
Great Lakes Fishery Laboratory
1451 Green Road
Ann Arbor, Michigan 48105
(313) 994-3331 (FTS) 378-1236 (O)

Mr. Yousuf W. Sheikh
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4650 (M, INP)

Mr. R. F. Showalter
Assistant Director
Water & Sewage Laboratory Division
Indiana State Board of Health
1330 West Michigan Street
Indianapolis, Indiana 46206

Mr. John Sliwinski
Beak Consultants Limited
6870 Goreway Drive
Mississauga, Ontario L4V 1L9
(416) 671-2600 (O)

Dr. Ronald Sloan
Senior Scientist
Division of Fish and Wildlife
NY Bureau of Environmental Protection
50 Wolf Road, Room 526
Albany, New York 12233
(518) 457-1769 (FTS) 567-1769 (O, M)

Dr. R. Dean Smillie
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3755 (O)

Mr. Mike Smith
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (INP)

Dr. John J. Spikes
Toxicology Section
Illinois Department of Public Health
2121 West Taylor Street
Chicago, Illinois 60612

Mr. Leroy Stratton
Associate Director, MSPH
Office of Environmental Health
Illinois Department of Public Health
535 West Jefferson Street
Springfield, Illinois 62761

Dr. W. M. J. Strachan
Chemical Limnology Subdivision
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (O)

Dr. Emilio Sturino
Chief, Organic Lab. Section
Central Regional Laboratory
U.S. Environmental Protection Agency
536 So. Clark Street
Chicago, Illinois 60605
(312) 353-9065 (FTS) 353-9065 (O)

Mr. Doug F. Sturtevant
Water Quality Branch
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-5639 (M)

Mr. Lawrence E. Sullivan
Food, Drug & Dairy Lab. Division
Indiana State Board of Health
1330 West Michigan Street
Indianapolis, Indiana 46206

Mr. K. Suns
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1

Dr. Robert A. Sweeney
Director, Great Lakes Laboratory
State University College at Buffalo
1300 Elmwood Avenue
Buffalo, New York 14222

Mr. Richard Szawioca
Beak Consultants Limited
6870 Goreway Drive
Mississauga, Ontario L4V 1L9
(416) 671-2600 (M)

Mr. Vince Tabone
Great Lakes Laboratory
State University College at Buffalo
1300 Elmwood Avenue
Buffalo, New York 14222
(716) 878-5821 (INP)

Mr. Jeff Taylor
Ontario Ministry of the Environment
Northwest Regional Laboratory
421 James Street North
Thunder Bay, Ontario P7E 2V6
(807) 475-1275 (INP)

Mr. Ken A. Terry
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (O)

Mr. William J. Traversy
Chief, Monitoring and Surveys Division
Water Quality Branch
Inland Waters Directorate, EMS
Place Vincent Massey, 7th Floor
Ottawa, Ontario K1A OE7
(613) 997-3422

Mr. Frank Tomassini
Laboratory Services Branch
Water Quality Section, MOE
P.O. Box 213
Rexdale Ontario M9W 5L1
(416) 248-3512 (M)

Mr. Hung Tse
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4304 (O)

Mr. Allen C. Tupy
Minnesota Department of Health
Section of Health Risk Assessment
and Analytical Services
717 S.E. Delaware St.
Minneapolis, Minnesota 55414
(612) 296-5516 (INP)

Dr. G. Veith
Research Chemist
Environmental Research Laboratory-Duluth
6201 Congdon Blvd.
Duluth, Minnesota 55804

Mr. Prem Vijan
Laboratory Service Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1

Dr. Serge Villard
Manager, Water Quality Section
Laboratory Services Branch
Ontario Ministry of the Environment
P.O. Box 213
Rexdale, Ontario M9W 5L1

Mr. Rick L. Wales
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
Burlington, Ontario L7R 4A6
(416) 637-4303 (INP)

Dr. Lester J. Walters
Associate Professor
Department of Geology
Bowling Green University
Bowling Green, Ohio 43403
(419) 372-2886 (M)

Mr. Bob Weinbloom
Division of Laboratories and Research
N.Y. State Department of Health
Empire State Plaza
Albany, New York 12201
(518) 474-4934 (M)

Mr. W. A. Willford
Research Chemist
Great Lakes Fishery Laboratory
U.S. Fish & Wildlife Service
1451 Green Road
Ann Arbor, Michigan 48105

Mr. Ray Wimmer
Department of Natural Resources
Stevens T. Mason Building
Lansing, Michigan 48926

Mr. Stan Wisz
Laboratory Services Branch
Water Quality Section, MOE
P.O. Box 213
Rexdale, Ontario M9W 5L1
(416) 248-3512 (INP)

Ms. Ann Wojcieszak
City of Chicago, Bureau of Water
Water Purification Division
Central Water Filtration Plant
1000 East Ohio Street
Chicago, Illinois 60611
(312) 744-3722 (M)

Mr. Henry Won
Chemist
Wildlife Toxicology Division
Environment Canada, Ontario Region
National Wildlife Research Centre
Ottawa, Ontario K1A OE7
(613) 997-1410 (O)

Mr. W. Wong
Bondar Clegg and Co. Limited
765 Belfast Road
Ottawa, Ontario K1G OZ5

Mr. Tung Kai Wu
Laboratory Director
Michigan Dept. of Natural Resources
Environmental Laboratory
315 North Clippert Street
Lansing, Michigan 48912

Dr. Robert K. Wyeth
Recra Research, Inc.
P.O. Box 448
Tonawanda, New York 24250
(716) 692-7620 (O)

Dr. Matthew Zabik
Pesticide Research Center
Michigan State University
East Lansing, Michigan 48824

APPENDIX III

REFERENCE MATERIAL

(1) Analysis of Organochlorine Residues in Fish. Authors: L. M. Reynolds and T. Cooper. Water Quality Parameters, ASTM STP 573, American Society for Testing Materials, 1975, pp. 196-205.

(2) Analytical Reference Materials: Organochlorine Residues in CWS-79-1, A Herring Gull Egg Pool from Lake Erie, 1979. Authors: H. T. Won and R. J. Norstrom. Canadian Wildlife Service, Manuscript Reports, 41, Jan. 1980.

(3) Analytical Reference Materials. I. Preparation and Purification of Photomirex. Authors: Alfred S. Y. Chau and Rod Thomson. Journal of the Assoc. of Off. Chem., Vol. 62, 6, Nov. 1979, pp. 1302-1308.

(4) Analytical Reference Materials. II. Preparation and Sample Integrity of Homogeneous Fortified Wet Sediment for Polychlorinated Biphenyl Quality Control Studies. Authors: Alfred S. Y. Chau, John Carron, and Hing-Biu Lee. Journal of Assoc. Off. Chem., Vol. 62, 6, Nov. 1979, pp. 1312-1314.

(5) Capillary Column Gas Chromatography: Twenty Years Old and Still in the Developing Stage? Author: Francis I. Onuska. Canadian Res., Vol. 12, 2, April 1979, pp. 26-33.

(6) Chemistry Laboratory Manual for Bottom Sediments and Elutriate Testing, Mar. 1979. Author: U.S. EPA. NTIS, U.S. Dept. of Commerce, 5285 Fort Royal Rd., Springfield, VA 22161. EPA 905-4-79-014, PB-294 596/2 WP, cost $8.00.

(7) Computations with Approximate Numbers. Author: D. B. Delury. The Mathematics Teacher, Vol. LI, 7, Nov. 1958, pp. 392-521 to 401-530.

(8) Great Lakes Cleanup - An International Success Story. Author: K. H. Walker, International Joint Commission, Windsor, Ont., Oct. 1977.

(9) Interlaboratory Study of the Determination of Polychlorinated Biphenyls in a Paper Mill Effluent. Authors: Joseph J. Delfino and Dwight B. Easty. Analytical Chem., Vol. 51, Nov. 1979, pp. 2235-39.

(10) Investigation of the Integrity of Seven Herbicidal Acids in Water Samples. Authors: Alfred S. Y. Chau and Karen Thomson. Journal of the Assoc. of Off. Analytical Chem., Vol. 61, Nov. 1978, pp. 1481-1485.

(11) Organochlorine Contaminants and Trends in Reproduction in Great Lakes Herring Gulls, 1974-1978. Authors: D. Vaughn Woseloh, Pierre Mineau and Douglas J. Hallett. Transactions of the 44th North American Wildlife and Natural Resources Conference 1979, pp. 543-557. Wildlife Management Institute, Washington, DC.

(12) Organochlorine Pesticides & PCBs. Author: P. Calway, Fisheries Services, Dept. of Fisheries & Oceans, Toronto, Ont., June 28, 1979.

(13) Quality Control Protocol, WPL Chemistry Unit. Prepared by: Ann L. Wojcieszak, City of Chicago, Bureau of Water, Chicago, IL 60611, Sept. 1979.

(14) Separation of Polychlorinated Biphenyls, Chlordane, and p.p'-DDT from Toxaphene by Silicic Acid Column Chromatography. Authors: Terry F. Bidleman, Janis, R. Matthews, Charles E. Olney and Clifford P. Rice. Assoc. of Off. Analytical Chemists, Inc., Vol. 61, 4, 1978, pp. 820-826.

(15) Toxic Substances in the Great Lakes. Author: Joseph J. Delfino. Environ. Sc. & Tech., Vol. 13, Dec. 1979, pp. 1462-1468.

(16) Water Pollution Control - An International Perspective. Author: K. A. Oakley, International Joint Commission, Windsor, Ont., June 13, 1977.

(17) Wildlife Contaminants Program Standard Procedures. Canadian Wildlife Service, Ottawa, Ont. K1A OE7

(18) Wildlife Contaminants Surveillance Program (IJC Great Lakes Water Quality Board, Data Quality). Authors: Douglas J. Hallett and Ross J. Norstrom. Canadian Wildlife Service, Ottawa, Ont. K1A OE7.