

Memorandum

May 04, 2000

To: LeRoy Schroeder, Chief, Branch of Quality Systems

From: Greg Mohrman, Chief, National Water Quality Laboratory

Subject: Quality Assurance.—Response to the technical review of the U.S.
Geological Survey National Water Quality Laboratory, January 24 – 28, 2000

In response to your letter dated February 15, 2000, enclosed please find our response to the subject review. NWQL responses are in bold type directly following the audit team comments. There are a few instances where you used bold type for the audit comments but the font you used is a little larger. We have responded to all comments that required a specific action. In many cases steps have already been taken to resolve the comments and the responses reflect this. For those issues still requiring action, the Quality Management Program plans to track progress and submit periodic progress reports.

We appreciated the thorough, constructive comments that you provided to us. We find that this external review, along with our internal reviews, help us to continually improve the quality of our analyses.

Enclosure

cc: Janice Ward
NWQL Program Chiefs

Memorandum

February 16, 2000

To: Merle Shockey, Acting Chief National Water Quality Laboratory, Denver, CO.

From: LeRoy Schroder, Chief, Branch of Quality Systems, Denver, CO.

Subject: Technical Review of the U.S. Geological Survey National Water Quality Laboratory
January 24 – 28, 2000.

The review team was impressed with the candor of the National Water Quality Laboratory staff who was consistently helpful to the team. Also, the management and supervisory chemists are commended for their insight, helpfulness, and support to the review.

My evaluation of the reviewer's comments indicated that these are 3 significant elements in the review findings:

1. Two units are analyzing samples with unapproved methods. In the Nutrient unit, the TKN method OFR is not approved. The OWQ Technical memo 98.05 specifically states that your customers should not report these data in open-file data reports or store the data in publicly accessible databases. The Biological Section taxonomic identification and enumeration of benthic invertebrate data is similarly restricted by OWQ memo 98.05.

The Kjeldahl analytical method OFR was revised based on colleague review comments and was sent to the Office of Water Quality Chief Chemist on February 29, 2000, for method approval, prior to Headquarters review of the report. The Chief Chemist determined that the Kjeldahl method digestion procedure and analytical finish were taken directly from an approved USEPA method: Method 351.2 – Determination of Total Kjeldahl Nitrogen by Semi-Automated Colorimetry (EPA/600/R-93/100; Methods for the Determination of Inorganic Substances in Environmental Samples.) There were only two modifications to the method as implemented at the NWQL. The Chief Chemist determined that neither of the two modifications should result in a change in data quality and so are considered Standard Operating Procedure (SOP) changes; not method changes. The report received Director's approval on April 17, 2000.

An Open-File Report (OFR) titled “Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory — Processing, Taxonomy, and Quality Control of Benthic Macroinvertebrate Samples” has passed through colleague review and updates were made. The OFR was sent to Headquarters for final review and Directors Approval on May 1, 2000. Publication of the report is planned for the summer of 2000. The method SOPs were approved on April 7, 2000.

2. The Nutrient unit, in particular the 4 – channel instrumental analysis, has significant documentation problems and GLP are not the routine in some subunits.

Please see the responses to Section III C, comments 1 and 2, of the attached document.

3. The team's interpretation of the NELAC requirements suggests that the NWQL would not be certified after a NELAC review. There are two points that the team stresses:
 - a. All documentation suggested by the Assessor Checklist is not in-place in several units, and;

The NWQL contends that much of the documentation mandated by NELAC guidelines is readily available. The NWQL understands that having the data available is sufficient for NELAC audit purposes, although we recognize that there is still a great deal of speculation on how the auditors will interpret the NELAC standards. The State of Colorado auditors are scheduled for NELAC audit training this Spring. The NWQL will contact the State officials once they have completed their audit training, to discuss what they will be specifically looking for during the audits. The NWQL believes that the interpretation of NELAC requirements by State officials will be more clearly defined once they have completed this training. The NWQL has appointed a Document Custodian to define documentation needs and develop a system to meet those needs.

- b. the requirements to assess data quality using matrix spike, matrix-spike duplicate, and duplicate samples are currently outside of the NWQL culture.

The NWQL understands that requirements for matrix spike, matrix spike duplicate and duplicate samples as outlined in current NELAC requirements are expected to be discussed, and in all likelihood, eliminated by NELAC at the annual conference to be held in June. The NWQL suggests that this issue be addressed after the conference.

As always, I'm available to discuss these and other findings and suggestions from the review. Feel free to contact me at any time.

Original signed on 2-16-00
LeRoy Schroder
Chief, Branch of Quality Systems
Attachment

TECHNICAL REVIEW OF THE NATIONAL WATER QUALITY LABORATORY

Building 95, Denver Federal Center
January 24-28, 2000

The following participants performed the technical review of the National Water Quality Laboratory (NWQL) January 24-28, 2000:

<u>Reviewer</u>	<u>Organization</u>
George Aikens	National Research Program
Larry Barber	National Research Program
Bill d'Angelo	Water Quality Service Unit, Florida District
Leslie Desimone	Massachusetts District
DeWayne Kennedy-Parker	Wisconsin State Laboratory of Hygiene
Mike Meyer	North Carolina District
Colleen Rostad	National Research Program
Dave Roth	National Research Program
LeRoy Schroder	Branch of Quality Systems
Steve Sorenson	Office of Water Quality

The principle findings of the review team are provided in the NWQL Section or Unit reports. These findings are extracted from individual reports, and the reviewer reports are attached to this review.

The review team was impressed with the candor of the NWQL staff who were quite helpful. The supervisory chemists and unit supervisors are commended for their insight, helpfulness, and support to the review. Also, Al Driscoll and Tom Maloney deserve special thanks for their support before, during and after the review.

I. Login Facility Safety

A. Login/Bottle Storage (see attachment 1)

The review did not identify any significant problems with the laboratory operations/sample login/bottle storage. However, the review team has suggestions that should help improve a good operation.

1. The continuous temperature monitoring of all refrigerators (a future plan is being developed) needs to incorporate failures during non-business hours. The monitoring of the refrigerators and freezers is a good quality-control (QC) practice, but the monitoring needs to include an action plan for failures.

Installation of the first phase of a computerized temperature monitoring system at the NWQL has an estimated completion date of May 31, 2000. The instrumentation used for this first phase of installation was purchased during the past three years based on recommendations of a team that assessed the capabilities of the new computer technology to monitor and document temperatures of critical equipment, such as refrigerators or ovens. The team addressed issues regarding corrective actions for failures and drafted guidance on addressing after-hour failures. However, this guidance focused on the former Ward Road facility and needs to be revised to reflect conditions at the new laboratory.

Refrigerators and freezers in the Organic Chemistry Program are being fitted with the computerized temperature monitors during the first phase. Temperatures will be monitored 24 hours a day, seven days a week. After hour failures will be identified on the computer system that logs the temperatures, however, notification of NWQL is not possible with the current instrumentation.

The NWQL will develop plans to install computerized temperature monitors on all NWQL refrigerators and freezers during FY2001. The NWQL will also investigate improvements in technology to allow notification of failures after hours. Completion of the SOP on temperature monitoring, including a section to address corrective actions, will be planned for early FY2001.

2. Analytical Sample Request (ASR) forms are available from three sources: 1) The NWQL, 2) Frame software, and 3) MS word software. These forms are not identical, and the differences among the forms cause problems for the NWQL login staff. The review team suggests that the NWQL require that Districts use a standard NWQL-designed ASR. Those Districts that choose to use a customized form should pay a surcharge for the extra effort required from the login staff.

An updated version of the ASR is currently being developed and will be sent out by the NWQL and OWQ for field review and comment. The NWQL will offer this new form in a number of electronic formats by October 2000, and it is expected that this will be the only ASR form that the Office of Water Quality will approve field personnel to use.

3. The unit estimated that 2 FTEs are needed to resolve the login problems that lead to “exception reports” and to correct the problems noted by the exception reports. Since the resolution of these programs are costly, the laboratory should develop a process that notifies the district that the occurrence of these problems will result in an increased cost for those districts that continue to submit samples that generate exception reports.

The Login Unit at the NWQL includes several staff to resolve sample submission errors. The cost of operating this Quality Assurance function is factored into the cost of analytical services. Therefore, sampling error costs are passed along to everyone. The NWQL does not believe it would be a good business practice to develop a cost recovery process for resolving sample submission errors on a sample-by-sample basis. The philosophy behind the operation of the Login Area QA function is to serve in a customer service role and to educate users on proper sample submission procedures. The NWQL believes that long term success in cost containment for sample submission errors will be achieved by training field personnel how to use appropriate sample submission procedures.

An additional process that the NWQL is developing is a email notification for exception reports generated as a result of sample submission errors. At the present time these reports are just sent to the Login Unit QA staff. Once the process is properly assessed, the NWQL hopes to send the email notifications directly to the person that submitted the sample. This notification system can be enhanced so that the notifications are forwarded to District Water Quality Specialists if an individual continues to have sample submission problems.

B. Sample bottle storage and tracking (see attachment 1)

1. The laboratory operations unit Standard Operating Procedure (SOP) needs to be updated. The review team feels this part of the unit is functioning very well.

The Standard Operating Procedures for the Login Unit and Bottle Storage will be updated after implementation of StarLIMS, and delivery of the software that will be used for sample tracking. Firm implementation dates for completion of these projects have not been established, however, the NWQL goal is to have StarLIMS operational in June. Therefore, the SOP should be updated this summer.

C. Data reporting (attachment 1)

1. The review team found no significant problems with data reporting although the use of a standardized ASR would benefit the overall efficiency of the unit.
2. A high importance needs to be placed on the implementation of the planned documentation of the STARLIMS systems. This system will need to be modified in the future, and documentation will be needed for a smooth operation.

StarLIMS is an essential part of the laboratory operations. StarLIMS is the highest priority NWQL project for FY2000. Documentation is planned at many levels – the system level, the operations level, and user level. The StarLIMS project plan is to use configuration management, change control, and complete testing is required of each step before implementation. User documentation is being written now. End users from throughout the laboratory have been selected to draft this documentation.

3. District rerun requests are retrieved hourly and an automated validation check is performed on the request. If the validation check identifies an error, several days may pass while the problem is resolved and the rerun request is declared to be valid. It appears that more rapid analysis of nutrient rerun requests could be achieved if the NWQL senior staff emphasized a policy that these analyses have the top priority.

The automated validation check in the District Rerun application compares the information from the district requestor against information stored in the NWQL database. If an exact match is not made, then it is impossible for the lab to process the request without further clarification from the requestor. If an invalid request has been made, an automated email requesting corrections or more information is sent to the district requester. The several day delay in obtaining updated information may be attributed to district response time.

NWQL senior staff has had a long-standing policy wherein reruns are given top priority. A policy statement will be written for distribution to all NWQL staff to reemphasize the priority of rerun samples. Additionally, nutrient reruns are given the highest priority of all reruns, especially considering the short turn-around times required. The Inorganic Chemistry Program Chief will work with Unit supervisors to ensure that all analysts understand these priorities.

4. Districts should be encouraged (this will probably take several encouragements) to provide information that a sample for inorganic analysis contains an analyte that will interfere with the automated ion balance routine.

The Customer Service Unit plans to re-release an updated version of the technical memorandum on shipping samples. This item will be added to the section on how to fill out an ASR. In addition, the lab will develop guidance on analytical interferences and related issues. We suggest that some of this information be presented at the QW field technique class. The lab will also work directly with districts as problem samples are identified, so that they make the desired notifications on future sample submissions.

5. Preferred values—Preferred values procedures are not documented in the current version of SPiN, and this procedure was unknown to the review team for inorganic analytes. The team recommends that this procedure be advertised.

The NWQL began the practice of releasing a preferred value for storage in NWIS at about the time that the current LIMS was put on line, approximately in May 1997. The laboratory began this practice because the NWIS system is only able to store one result for a given parameter code. However, there are common analytes (same parameter code) for many of the frequently requested lab schedules. This is particularly true of the Organic schedules. Thus, the NWQL began using the “preferred value” data release to avoid overwriting data in the NWIS database with less desirable results. The non-preferred data has been sent to the districts by email and the districts have been given the final decision on which data to store in the database. The use of “preferred values” is not thought to be very common for Inorganic analyses. If the need would arise to decide on a "preferred value" for an Inorganic determination, the NWQL would forward the value for the method with the last assigned method code. This logic was announced early on when the “preferred value” practice began but based on this audit comment it is obviously time to announce the practice again. Neither the SPiN nor catalog applications on the NWQL web site provide information regarding the “preferred value” practice. At the present time, both of these applications are being replaced with a single application that will go on line when the new LIMS (StarLIMS) comes on line. The NWQL will include information about the “preferred value” practice on the replacement application.

6. Transfer of QA data—A Web-based information-distribution system is suggested to provide QA data for inorganic and nutrient analyses that are stored in the laboratory information management system. The review team believes the districts want and will use these data, and the NWIS II may not satisfy the role.

There is currently very little Inorganic QA/QC sample data stored on LIMS. Implementation of the StarLIMS database will enable the laboratory to store a vast amount of QA/QC information such as calibration data, on-line QC, duplicates, and standard reference materials. The need to develop web based and custom report applications to release this information has been identified in the long-term project plans for the StarLIMS project. This has already been discussed with the NWIS Phoenix workgroup. The NWQL will continue to work with the Phoenix workgroup to develop and deliver the types of information and data reports required by the Districts.

D. Customer Support

1. The review team applauds the laboratory for developing the full time customer support person. The team suggests that the laboratory create a tracking system such as an electronic bulletin board that contains customer inquiries and responses. This process may help the laboratory identify commonly occurring problems and inquiries and aid the remedial-action process.

The NWQL has recognized the need to track help requests. The Computer Services Unit has a current project (FY2000) to evaluate various web based Help Desk and bulletin board software. The Customer Service Unit and other Units of the NWQL have been asked to help with the software evaluation. The goal of the project is to select a package for lab-wide use by July 2000. We will ensure that the evaluation process includes assessment of software capabilities to identify commonly occurring problems and inquiries.

E. Chain of Custody (COC)

1. The SOP for COC needs revision, e.g., the description of the locked storage area bottles is inaccurate, and there is no fume hood for the immediate storage of broken bottles.

The NWQL is in the process of transferring the Chain of Custody storage area to a location adjacent to the bottle storage warehouse. The responsibilities for the maintenance of the sample bottle storage areas will be transferred to the Inorganic Chemistry Program at the completion of the move. The estimated completion date for relocation of the Chain of Custody storage area is June 30, 2000. After the move, the Chain of Custody SOP will be updated to reflect the location of the new storage area and new responsibilities of the Inorganic Chemistry Program.

COC sample bottles received broken, or broken in Login, will be placed in a closed container and transported to the waste management area for proper disposal. This change has been approved by the Safety Unit and will be included in the Log-In SOP and the NWQL Chemical Hygiene Plan.

F. Safety

The review team felt that the safety program at the laboratory is operating well, and, for the most part, the documentation is in order. **Several reviewers noted that there seems to be a large number of people eating in the laboratory areas.**

An NWQL Policy Memorandum regarding this important safety issue was distributed via email to all NWQL employees on February 24, 2000. This policy memo is available on the NWQL internal web page and will be included in the NWQL Chemical Hygiene Plan. Supervisory personnel have been asked to emphasize the importance of this safety concern with all their staff.

1. The master file of MSDS sheets for chemicals in the building are readily accessible in the reception area. Labeling of this cabinet should be useful for after-hour emergencies.

The MSDS cabinet is now labeled so that it is easily recognized for use in after-hour emergencies. The location of the MSDS cabinet has been included in the NWQL Occupancy Emergency Plan. Signs have been posted on the cabinet, and on the wall above the cabinet, to ensure that it is easily identifiable.

2. Flammable and non-flammable liquid and gas storage rooms were unlocked when inspected by the review team (see SOP).

The storage rooms will be kept locked. Signs will be posted on the storage room doors to indicate that they are to be locked, as a reminder to warehouse employees.

3. One gas tank was reported to be unsecured in the main hall. The team suggests that NWQL personnel be reminded of the possible danger from unsecured gas tanks.

An email was sent on March 21, 2000, to all NWQL employees reminding them of the proper storage and handling of compressed gas cylinders. This important safety topic will be included in the HazCom Classes, Safety Orientations for New Employees, and State of Colorado Hazardous Materials Regulations Training.

4. The team suggests that some version of the anonymous safety-complaint book be implemented at the laboratory.

"Hazard Elimination Logs" (HEL) have been posted in the NWQL lunchroom and break rooms. Entries on the forms are made anonymously. The HEL are used to document employee suggestions and complaints regarding safety issues. Each Log station will be checked weekly by a member of the Safety Unit staff. An email message announcing the HEL was sent to all NWQL staff on May 2, 2000.

II Quality Assurance; Analytical Contracts; Sediment and Tissue Preparation (attachment 2)

A. Quality Assurance Unit

1. The introduction of in-house audits is a good approach to internal QC by the laboratory, and the review team considered the in-house audit reports to be done well.
2. QAU needs direct access to quality control charts for each analytical line. This process is very important. Review of internal control charts and external blind sample control charts between QAU and the Inorganic section is in the QA/QC manual. These meetings no longer occur.

The above comment addresses the difficulties that the QAU staff has encountered regarding access to bench-level Inorganic QC information. We believe that these difficulties, real or perceived, will be addressed in weekly Inorganic Chemistry Program QC meetings that were recently instituted by the Inorganic Chemistry Program. Agenda items for the meetings will include the following: review and discussion of BQS generated control charts, discussion of bench-level generated control charts, and discussion of reruns and associated NWQL and BQS comments for BQS Blind Samples. The Quality Assurance Unit is committed to participate in these meetings. The weekly meetings rotate through the three Inorganic Units so that each Unit is reviewed at least once each month.

3. The team is concerned about the apparent lack of interaction and communication between QAU and the Inorganic section relating to the information provided by the BQS Blind Sample program (BSP). A significant reason for the discontinuation of the internal inorganic blinds was that these data are available from the BSP and would be utilized by the NWQL (QAU). This does not appear to happen. The communication between QAU and the Inorganic section might be improved through reinstating the Inorganic Quality Assurance meetings.

As mentioned above, steps have been taken to improve communication pathways between personnel involved in quality assurance and those involved in production. Starting in March, the NWQL reinstated weekly meetings between QAU and the Inorganic Chemistry Program, and these meetings are intended to be a springboard for the discussion of several quality-related issues including the Blind Sample Program (BSP). The re-institution of the Inorganic Quality Assurance Committee may be effected to augment the Inorganic Chemistry Program quality control meetings. The Quality Assurance Unit will need to reassess functions and responsibilities regarding the continual in-house assessment of the BQS Blind Sample results. This represents an increase in core functions of the unit and staffing needs to be adjusted accordingly.

4. The NWQL QA manual indicates that most lines are to analyze duplicate and spike samples, and the team didn't find documentation of duplicate and spike sample analysis for a number of the analytical lines.

Spike samples are a fundamental QC type used for Organic Chemistry methods. Virtually every Organic method relies on spikes for assessments of preparation steps. The Inorganic Quality Control section of the current NWQL QA Manual indicates that the NWQL uses at a minimum of one QC sample for every 10 environmental samples. Spikes (and duplicates) are listed as a possible QC sample type. However, the QC sample types primarily used by the Inorganic Chemistry Program are BQS SRWS and Third-party check samples.

In 1997, the Laboratory QC Committee (including representatives from EPA, Environment Canada, and WRD Offices) released its evaluation report of QC practices at the NWQL. The report states that “By and large, the Laboratory QC Committee felt that the amount and mix of QC samples for (the Inorganic methods it evaluated) was about right.” Further, the report states that “The Laboratory QC Committee did not feel that lab-prepared inorganic spikes should be included as routine on-line QC. Although EPA and Environment Canada periodically use lab-prepared spikes as part of their on-line QC, they did not recommend the practice and generally felt the data was seldom (if ever) used for process control.”

Use of duplicates (replicates) is not common for line QC. Replicate QA samples submitted by the Branch of Quality System IBSP and OBSP projects are the basis of the method precision assessments for these QA projects. Discussion of the merits of duplicate environmental samples will be an issue brought up at the weekly Inorganic and Organic QA meetings. Duplicates may be useful for QC on Sediment and Tissue methods to supplement the use of expensive third party check samples.

The NWQL Quality Management Program is currently rewriting the QA Manual and we will be sure to clarify the use of spike and duplicate samples for line QC.

5. The NWQL QA/QC plan has not been updated for 5 years. Although OFR 95-443 is given to Districts upon request and there have been no complaints from these Districts, the manual does not portray the current laboratory method of operating.

The NWQL recognizes that the current QA/QC plan is outdated. The rewrite of the manual was intentionally delayed the past couple years in anticipation of a new standard from NELAC. The NELAC standard is now available and the NWQL has a project in place to rewrite the QA/QC manual this fiscal year. Brooke Connor made significant progress in preparation of a first draft of the QA Manual in NELAC format prior to her transfer to the Branch of Quality Systems. The Quality Management Program plans to complete the rewrite of the QA Manual this summer.

6. The review of QAU suggested that the rerun requests for the Nutrient Unit could encounter significant delays between the time the rerun request is made and the

actual time that the analyst receives the request. Also noted was the problem with tracking the location of sample bottles during the rerun process. The team suggests that these delays can be reduced by an emphasis on the problem by the laboratory senior management.

Each workday, rerun requests including nutrient requests are validated by QAU and incorporated into the LIMS workfile/protocol generation. In the case of non-nutrient rerun requests, validated requests are sent daily to the bottle warehouse for a bottle pull and inclusion in the District Rerun program. The Nutrient Unit has direct control of all nutrient samples, and there have been no documented occurrences of logistical difficulties in sample tracking within the Unit. Customers may have noted some delays in processing of reruns between August and October of 1999 due to staff shortages that have since been corrected. However, the Nutrients Unit is now fully staffed, and all analysts are aware that they must call-up samples daily and that rerun samples have the highest priority.

There has been a request to the StarLIMS Reports project to provide an improved rerun status management report. In addition a policy memo will be sent to laboratory staff to emphasize the need to process reanalysis requests with the highest priority. Two different rerun backlogs are distributed weekly by the QAU to all appropriate supervisors. One lists all uncompleted rerun requests from Water Resources Division personnel, and the second backlog lists all uncompleted rerun requests generated by the NWQL data review program.

7. It appears to members of the review team that the QAU personnel do not have an outside of NWQL peer group to discuss problems, find solutions, and generally discuss QA/QC of water-quality laboratories. I used the phrase "operating in a vacuum" to describe this apparent lack of interaction among the NWQL QAU members and similar units in other laboratories. The Wisconsin Hygiene Laboratory personnel indicated that they were interested in this interaction.

The Quality Assurance Unit has recently become involved with a consortium of Denver area federal laboratories to discuss quality issues. This committee, to date, has identified discussion topics including: documentation, certification, audits, training, reference materials, statistical measures, and a directory of Denver area federal laboratory resources. QAU is also becoming more involved with NELAC related activities, including plans to attend upcoming meetings. Finally, QAU is establishing contacts with the Wisconsin Hygiene Laboratory and will make efforts to improve contacts with the Ocala Water Quality Service Unit. QAU intends to establish contacts with other laboratory quality groups to improve interaction with the scientific community.

B. Analytical Contract Unit (attachment 2)

The unit was not reviewed due to changes that will affect the unit's functions. The review of the laboratories under either contract or agreement with the NWQL is required by OWQ Technical Memoranda. BQS and NWQL will arrange for these reviews during FY2000 and FY 2001. The contract unit will require significantly more quality control information from laboratories during the contracting of services, and the team believes this is the correct approach.

The NWQL will work with the Branch of Quality Systems to identify contract laboratories that require reviews. The NWQL currently administers contracts with 5 commercial laboratories.

As indicated by the review team, the NWQL Analytical Contracting Unit plans to require more quality control information prior to award of a contract. There have been significant changes made in the government contracting procedures to define goals for the performance based contracts. In addition, NELAC standards for contracting laboratory services specify documentation and QA/QC requirements. To address these changes, all future NWQL "Requests For Proposals", for laboratory contracts, require that laboratories provide a NELAC formatted QA/QC manual and pertinent QA/QC information to be considered for award of the contract.

C. Sediment and Tissue Preparation (attachment 2)

The unit is well organized. Logs were in place and when each step of the extraction is completed analysts initial the logs. The wall chart and use of color-coded labeling tape enhances the efficiency of the unit. The unit and other laboratory staff discuss procedures with the objective to minimize the chance of a problem recurring. The review team commends this communication.

1. There is a draft SOP for pipette calibration; however, it appears that the volume marks on the capillary tubes of the pipettes are accepted as correct. The draft SOP needs to be implemented, and the analysts need to check the delivery volumes of these pipettes.

The Organic Chemistry Program is currently reviewing procedures, policies, and SOP's for the dispensing of surrogates and internal standards. Further work has been done on the draft SOP and it should be ready for the first step of SOP review, within the Organic Program, by May 15, 2000. The SOP will be implemented if the procedure is found to provide accurate and valuable information.

III. Inorganic Section

A. Majors (attachment 3)

1. The review of the unit training records and explanations of the overall training program suggest the unit is doing a good job.
2. Sample results are transferred to the central database without peer review even when manual entries are needed for dilutions and sample number corrections. A GLP should be designed and included into the SOP.

The NWQL is reviewing its organizational structure to look for ways to more effectively implement a peer- or second-level review throughout the Inorganic Chemistry Program. The NWQL expects to more clearly define a peer-review process within the next six months, which will be reflected in subsequent SOP updates and in the new NWQL Quality Assurance Manual.

3. SOP changes are needed for the following:
 - a. The acceptance criterion in percent difference for the low concentration standards. The current criterion of 2% difference between the old and new standard cannot be met on most occasions.

SOP's for the Majors Unit will be revised to reflect more specific acceptance criteria for standards comparison. The Inorganic Chemistry Program acceptance criteria policy is on the agenda for the newly scheduled QAU/Inorganic meetings, and has been the subject of several recent meetings. The Program intends to redefine acceptance criteria for new standards within the next three to six months.

- b. The acceptance criteria for duplicate and spike sample results needs to be documented and not employed at the analyst's discretion.

SOP's for the Majors Unit will be revised to reflect more specific acceptance criteria for duplicate and spike results. The Inorganic Chemistry Program acceptance criteria policy will be evaluated and appropriate changes made – this will be done in cooperation with QAU and is expected to be one of the issues discussed at the weekly meetings between the Inorganic Chemistry Program and the Quality Assurance Unit. These concerns will be addressed after the annual NELAC conference in June, where it is expected that standards for duplicate and spike acceptance criteria will be addressed.

4. The Graphite furnace Atomic Absorption (GFAA) area is well organized and the SOPs seem current, as were the training records, logbooks and maintenance records.

5. The methods document recommends a monthly preparation schedule for the calibration standards. Calibration standards are prepared every 6 months within the unit. The SOP should agree with the method documentation.

The change from making new working standards monthly to semi-annually was approved in a SOP change dated February 1998. There have been no data quality issues in the GFAA area since this change was implemented. Both on-line and blind QC charts for all of the parameters involved are in control. This change will be formally documented in the SOP as a deviation from the original method.

6. A few relatively minor documentation and filing lapses were noted to the lead analyst who agreed to emphasize the need for initialed notations to be made.

This comment refers to the GFAA area and filing protocols. The need to annotate reruns on the original data was re-emphasized to the specific analyst. We believe the problem has been resolved.

B. ICP - MS, ICP-AES, Mercury, and Potassium (attachment 4)

1. The impression of the personnel, equipment, training and techniques in the unit was good.
2. ICP-MS - Pipette calibration should be checked more frequently, especially when the volume delivered is near the limits of the pipette working range.

Currently the pipettes are verified on a quarterly basis with the information entered into a logbook. The Rainin pipettes used by the Plasma Unit have demonstrated excellent accuracy and precision over time and rarely, if ever, have required adjustments. However, pipettes used for dilutions near the limits of the working range will be calibrated monthly to ensure accuracy and precision.

3. ICP-MS - Daily-operating parameters of the instruments should be in logbooks. Also, a record of basic periodic maintenance should be in a logbook. Entries to these logbooks should include the analysts' initials.

Daily operating parameters of the instruments are generated electronically and entered into the logbooks that also document periodic maintenance. ICP-MS historical logbook entries are readily available in-house for the last two years. Older logbook records are archived. ICP-MS analysts will initial all entries.

4. ICP-MS - To reduce the influence of the high calibration standards, additional lower concentration standards should be included in the calibration curve. A NELAC requirement is that calibrations are to be verified by analyzing a blank and mid-level standard immediately following the calibration curve. Blanks, matrix spikes, and matrix-spike duplicates should be an integral part of analysis procedure.

Although low level QC is analyzed during daily sample analyses, the Plasma Unit will reassess its calibration procedure with the expectation of adding an additional standard at the low end of the calibration curve. Implementation of NELAC and related QC requirements will be discussed and clarified further as the NWQL moves towards NELAC accreditation.

5. ICP-AES - Both the horizontal and vertical adjustments should be re-optimized to maximize performance after maintenance on the nebulizer, torch, or the torch box.

The Plasma Unit will routinely re-optimize the horizontal and vertical adjustments on the torch after maintenance. In addition, a vertical alignment and optimization is performed, on a daily basis, so that any changes in the sample transport from the sample introduction system (nebulizer and spray chamber) are compensated for.

6. ICP-AES - The review team believes that the use of a multi-point calibration curve must be implemented. The NELAC requirements list in section B.4 also pertain to ICP-AES.

As a result of the recommendation, the NWQL expects to implement a multi-point calibration curve within the next year. The Plasma Unit has traditionally used a 2-point method of calibration according to the Technique of Water Resources Investigations method. In addition, ASTM method D 1976 is validated using a two-point calibration procedure. The 2-point calibration procedure has been employed for over 20 years at the NWQL with no apparent adverse effect on data quality. Nevertheless, the NWQL understands that prevailing thought on this subject has changed in recent years, and multi-point calibration curves have become more common.

7. Mercury - Samples should be poured into sample vials in a clean environment (laminar flow hood) to minimize the possibility of contamination.
 - a. The CVAFS instrumentation needs to become useable as soon as possible because the current instrumentation is not adequate for uncontaminated surface-water samples.

The Inorganic Chemistry Program agrees that the Cold Vapor Atomic Fluorescence Spectroscopy development and implementation should be a priority. Prior to the audit, the NWQL had no immediate plans to develop the CVAFS system; however, as a result of the recommendation, the NWQL will get the project prioritized and scheduled for development. The Plasma Unit will work with the MRDP staff on replacing the present Cold Vapor Atomic Absorption with new CVAA and/or CVAFS instrumentation. An initial meeting has been held regarding this project.

The current CVAA method does not have low-level detection capabilities, and therefore does not require the use of a laminar flow hood for the sample pour-up. However, because of the low-level detection capability of the CVAFS methodology, the use of a clean environment will be addressed when the CVAFS method is implemented.

- b. The NELAC requirements mentioned earlier pertain to mercury determinations.

As a result of this recommendation, the Plasma Unit will collaborate with Quality Management Program to develop quality-monitoring systems that meet NELAC requirements. Currently, after each calibration curve is established, a blank and a reference are analyzed to validate the curve. In addition, known Standard Reference Water Sample's are analyzed every ten samples. Additionally, a variety of known SRWS's that cover the analytical range are employed. However, due to water-volume limitations, matrix spikes and duplicates may be difficult to implement for whole-water mercury determinations.

8. Potassium - Linear concentration ranges need to be determined, checked, and documented on a regular basis.

The Plasma Unit will implement a procedure to verify the linear concentration range for each calibration. The Plasma Unit currently employs reference materials at various concentrations, including blanks, to check and document the analytical range. NELAC and QC requirements including spikes, duplicates, spike duplicates, use of mid-point check standards, and frequency of blanks, will be discussed further at future Inorganic Chemistry Program QA/QC meetings.

C. Nutrient Unit (attachment 5)

1. As noted in the 1995 review of this unit, the SOPs are usually not current and inconsistently used by the analysts except for the 2-channel line (TKN, TP).

New SOP's have been written to reflect current practices. The SOP for suspended solids has been completed and approved. The SOP for silica has been through colleague review, and is now in NWQL senior staff review. The SOP for the 2-Channel (TKN/TP) has been through colleague review and is now in NWQL senior staff review. The SOP for the 4-Channel is in colleague review. New SOP's have been written to reflect current practices. The SOP for suspended solids has been completed and reviewed. The SOP for the 4-channel is in colleague review; the SOP's for the 2-channel (TKN/TP) and Silica have been through colleague review and are presently in NWQL senior staff review. SOP's for the Low Level Phosphorus, Low Level 4-channel and, Ion Chromatography (chloride and sulfate) will be out for review no later than June 1, 2000.

2. GLP are not always followed in the unit, for example:

- a. Electronic pipette calibration documentation was not readily available.

New pipette logbooks and a new calibration process are in place. Specific individuals are assigned the responsibility of specific logbooks and senior personnel check the logbooks monthly.

- b. The labeling of calibration standards was incomplete and inconsistent.

1. Some labels did not contain the year prepared.

2. Other labels did not contain the initials of who made the standard.

Analysts have been retrained. Proper procedures are detailed in the new training forms that are used to train each analyst. Senior personnel will check the standards bottles monthly.

- c. Standard Preparation schedule documentation was not available.

Standard preparation schedules are different on individual lines based on the stability of the analytes of interest. Standard preparation schedules are in the new SOP's.

3. Standard preparation does not appear to be in agreement with the SOP. For example, calibration standards and QC references are prepared by one-step dilution of the primary stock solution. The SOP discusses serial dilution. The SOP needs to be updated or the one-step dilution needs to be discontinued.

New SOP's have been written to reflect current practices. As indicated in the response to III.C.1 above, Nutrient Unit SOP's will be completed and approved in the next 4 months.

4. Duplicate and spiked samples, acceptance criteria for blanks, and a formal corrective action plan for the use of QC data would increase the data quality.

The Inorganic Chemistry Program agrees that acceptance criteria and corrective actions need to be clearly defined. These issues will be addressed at weekly meetings between QAU and the Inorganic Chemistry Program, with a long-term goal of developing Program-wide standards that can be incorporated into a comprehensive training program.

Preliminary acceptance criteria have been included in the new SOPs and in the training forms.

5. **The TKN determination method used on the 4-channel instrument is not an approved method. The unit and the NWQL are not in compliance with OWQ Technical Memorandum 98.03, and should notify their customers that these data are not to be reported in the annual open-file report or put in public accessible databases.**

The OFR for the method has been completed; method approval by OWQ was obtained March 21, 2000; and Directors approval as OFR 00-170 was received April 17, 2000. Data quality has been consistent since the method was introduced and is well documented. Additionally, the method in use is the same as a documented and published USEPA method by Andrea Jirka (Jirka, and Carter, et.al., 1976, Environmental Science and Technology, v.10, no. 10, p. 1038-1044).

Please refer to the response to comment number 1 on the cover letter for further discussion on this issue.

6. 4-Channel SOP is actually an instrument operating procedure. The review team recommends a new SOP similar to the SOP for the 2-channel instrumentation needs to be prepared as soon as possible.

A comprehensive SOP for the 4-Channel method has been written and was submitted for colleague review on 03/03/00.

7. 4-Channel - The QC sample used is an SRWS, and there doesn't appear to be an action plan if the QC result is unacceptable. Acceptance limits need to be developed for all QC sample types (continuing calibration verification sample (CCV), duplicate samples, spiked samples and purchased reference materials,) and these limits need to be enforced.

Preliminary acceptance criteria have been developed and documented and are in the new SOP and in the training forms. Acceptance limits for QC throughout the Inorganic Chemistry Program will be discussed in the weekly meetings with QAU.

8. 2-Channel - There is a training need for the analyst who was unaware of the duplicate sample acceptance limits documented in the SOP.

The analyst was re-trained and a training certification was made using a newly developed training form. Acceptance criteria are in the new SOP and in the training forms used for cross training.

9. 2-Channel - Even though the spikes to nanopure water have a positive bias and have had a positive bias since mid-November, no corrective action taken because the "spikes are still within limits". Again, this is not GLP and would not withstand a NELAC review.

Immediately following the audit, the bias problem was investigated and was found to be the result of contamination in the spike solution. Spike solutions are now made more frequently and all analysts have been reminded that any changes in an analytical line (even if QC is still within limits) should be reported to the Unit supervisor immediately and investigated.

10. 2-channel - The documentation of calibration standard for old standards verses new standards was not available. These criteria need to be developed and started as soon as possible.

Establishing program-wide criteria for the acceptance of calibration standards will be a topic for discussion at the Inorganic Chemistry Program and QAU weekly meetings. As a result of the audit, calibration standard validation forms are now in use throughout the Nutrient Unit. Preliminary acceptance criteria are documented in each of the new SOP's and in analyst training forms. Also, although the previous checks did not use a specific form, comparison runs were kept and reviewed. The unit supervisor reviews verification forms, QC charts, and maintenance logs monthly.

11. 2-Channel - Samples are diluted if the concentration exceeds 10% of the highest calibration standard. However dilution verification documentation was not available. The normal check is to multiply the new results by the dilutions value and compares this value with the non-diluted result.

Results are multiplied by the reported dilution factor automatically by the software package in use. The new SOP and the training forms will point out that diluted samples must yield results above the highest calibration standard.

12. 2-Channel - The QC charts for ERA references and SRS were maintained and looked good.

13. LL Phos - Again, the main point of the review team is that GLP is not followed for this method, for example:

- a. Duplicate and spike sample are analyzed but no QC limits exist; and data are not used to access the data reported.

Establishment of program-wide acceptance criteria for duplicate and spike samples will be a topic for discussion at the Inorganic Chemistry Program and QAU weekly meetings. Preliminary acceptance criteria are documented in the new SOP and in training forms. The analyst has been retrained in this area.

- b. No documentation is available for the comparison of old calibration standards to new calibration standards. However, the analyst does examine the CCV data, but reviewers are not positive about how these data are used by the analyst.

Establishing program-wide criteria for the acceptance of calibration standards and the use of CCV data will be topics for discussion at the Inorganic Chemistry Program and QAU weekly meetings. Calibration standard validation forms are now in use. Preliminary acceptance criteria for calibration standards are documented in the new SOP and in training forms. The unit supervisor reviews verification forms monthly.

14. LL 4-Channel - The calibration standards are made daily and Cd-column efficiency is checked daily. Also, the acceptance limits for the column efficiency are used to regenerate the column. All are good QC activities.
15. LL 4-Channel - The pipette calibration verification is done but not documented. The pipette calibration is a mass determination. Reviewers suggest the verification kits recommended by Charlie.

New pipette calibration logbooks are in place. Specific individuals are assigned the responsibility of specific logbooks and senior personnel check the logbooks monthly. Additionally, Unit personnel are investigating the use of colorimetric verification for micropipettes. Quality Assurance Unit staff demonstrate the colorimetric system and provide training if desired to appropriate personnel.

16. LL 4-Channel - The computer program used to transfer the data to the central database does not work correctly, and the macro needs to be fixed.

As a result of the audit, data transfer problems were brought to light and were given priority status. Computer problems were corrected on February 7, 2000.

17. IC for Cl and SO₄ . The analysts is knowledgeable, experienced, and regularly reviews the QC charts. However, a duplicate sample is included in each sample set, but the data are not used to evaluate data quality.

Duplicate samples are used to evaluate method precision. Establishing program-wide criteria for the acceptance of duplicate samples will be a topic for discussion at the Inorganic Chemistry Program and QAU weekly meetings. Preliminary acceptance criteria have been established and are documented in the new SOP and in training forms.

18. IC for Cl and SO₄ . As noted in earlier reviews, the calibration range is from 0.1 to 300 mg/L; reviewers believe this is an excessive range. A quadratic curve fit is used to evaluate the calibration curve, but a point-to-point curve fit is used to calculate analyte concentrations. Again, this does not seem to be a GLP.

The method report recommends an analytical range of .01 to 10 mg/L. The current range of .1 to 300 mg/L has a span of approximately the same magnitude as recommended in the analytical method. No problems have been reported for these lines based on internal or external QA assessments. BQS Blind Sample data for these analytes for the past couple years indicate that the methods are, and have been generating excellent quality data throughout the analytical range. However, as a result of this audit comment, the Nutrients Unit will work with MRDP and QMP personnel will further examine the analytical range.

The point-to-point curve is used according to the method prove-out and the TWRI method (TWRI I-2057-85) for this line. Chloride and sulfate are not linear over the calibration range used. The quadratic curve fit is only used to provide analysts a general feeling for the linearity and shape of the curve. It is a gross measurement but the NWQL believes it provides the analysts' with a tool to assess the overall calibration.

19. IC for Cl and SO₄ . The SOP is out of date and the automatic diluter is not regularly calibrated. Reviewers could not find any dilution verification documentation

A new SOP has been written and was submitted for colleague review on April 3, 2000. Analysts have been trained to perform a login check on diluted results such that the final result should exceed the highest calibration standard if a dilution has been made. The analysts have been instructed to ensure that the corrected data is entered into the computer. It is understood that the StarLIMS software will include an application to automate this check.

New pipette calibration logbooks are in place. Specific individuals are assigned the responsibility of specific logbooks and senior personnel check the logbooks monthly.

20. Acceptance limits for duplicate and spike sample results needs to be developed, used to evaluate data quality, and documented in the SOP.

Establishing program-wide criteria for the acceptance of spike and duplicate sample will be topics for discussion at the Inorganic Chemistry Program and QAU weekly meetings. Preliminary spike and duplicate sample acceptance criteria are documented in the new SOP. There remain some computer limitations that need to be addressed before the results can be fully analyzed and evaluated. Procedures will begin as soon as the data handling software can be modified.

21. The analysis of duplicates, spikes, etc., and the non-use of these data are bad laboratory practice. The review team feels this unit should not be exposed to a NELAC review because the documentation is not available. Problems in the QC practice of the unit were stated in the 1995 review and need to be corrected.

The Inorganic Chemistry Program agrees that the correct use of spike and duplicate data needs to be clearly defined, and tools need to be developed to support the use of these data. As mentioned above, the first step is the establishment of acceptance criteria for the use of this QC data. The Inorganic Chemistry Program will work with both CSU and QAU to develop appropriate tools and methods of data interpretation. These issues will be addressed at weekly meetings between QAU and the Inorganic Chemistry Program, with a long-term goal of developing Program-wide policies and procedures.

The NWQL understands the current NELAC requirements for matrix spike, matrix spike duplicate, and duplicate samples are expected to be discussed, and in all likelihood, eliminated by NELAC at the annual conference to be held in June. The NWQL suggests that this issue be addressed after the conference.

The Quality Management Program and Nutrient Unit will work together to correct the various problems noted by the review team.

IV. Biological Unit (attachment 6)

A. Analytical Methods

The Reviewer was impressed with the unit from the supervisor to the data manager. One comment from the reviewer seems to sum the reviewer's thoughts: "All of the members of the (Biological) Unit appear to work well together and communication among the taxonomists is very effective at promoting the consistent integrity of the data produced".

1. The unit supervisor should continue to work towards a larger customer or client base. The senior staff of the NWQL should recognize this need. The potential for these services seems to be large.

The Biology Group (BG) supervisor continues to be proactive in marketing the biology services with the water and biology disciplines in the USGS. The BG is also exploring cooperative projects with other federal agencies, especially the U.S. Environmental Protection Agency. NWQL Senior Staff is continuing to support these efforts.

2. **The methods used by this unit are not approved. This fact can cause a significant problem for the district clients and customers. The unit and the NWQL are not in compliance with OWQ Technical Memorandum 98.03 and should notify their customers that these data are not to be reported in the annual open file report or put in public accessible databases.**

An Open-File Report (OFR) titled "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory — Processing, Taxonomy, and Quality Control of Benthic Macroinvertebrate Samples" has had colleague review and has been revised. The report was forwarded to Headquarters for final review and Directors approval on May 1, 2000. Publication of the report is expected this summer. The NWQL Biological Group has a series of communications with NAWQA leadership regarding protocols that have been used during the development of the invertebrate sample processing methods. The Biological Group plans to draft a Technical Memorandum summarizing this information so the NAWQA Study Units can publish their findings in interpretive reports.

Please refer to the response to comment number 1 in the cover memo for further discussion on this issue.

3. The SOPs need to be formalized as soon as possible.

NWQL SOPs on the processing of benthic macroinvertebrate samples have been written and were approved on April 7, 2000.

4. The data handling procedures are good and a very important part of the unit's ability to serve the customer. The review team suggests that the unit investigate the automation of the data handling to make the process easier for another unit personnel. The incorporation of these data-handling procedures with the STARLIMS would be a plus for the unit and the NWQL.

During FY99, the Biological Group met with a NWQL STARLIMS representative to define data-handling needs. The data-handling requirements were significantly different from other current NWQL operations, so that the Biological Group functions could not be addressed by the current version of StarLIMS. The Biological Group supervisor met with the NWQL StarLIMS coordinator during April 2000 to develop the request for proposal to LIMS USA, for incorporating Biological functions into the StarLIMS system. A goal was set to have the RFP developed by June.

5. A long-term need for the unit is to investigate and improve the procedures for evaluating the variability in sub-sampling procedures. Plans for this investigation should be developed, so activities can begin when time and resources are available.

The Biological Group supervisor agrees that being able to address sub-sampling variability is an important question. However, this question has not been satisfactorily addressed in the industry. Studies to answer this question, and other related questions, would be better developed through some biological representation of the Methods Research and Development Program (MRDP), or through existing working relationships with National Research Program personnel in Menlo Park, California. Discussion with Menlo Park NRP staff have been ongoing regarding the need to evaluate sub-sampling techniques. In addition, a conversation between the Biological Group Supervisor and the MRDP Chief addressed the possibility that the MRD Program may develop biological expertise.

V. Organic Section.

The review team found that the section has made a substantial effort to improve the method and SOP documentation compared to the 1995 review. Most of the units received a review that examined many aspects of the NELAC requirements, including using review checklist guides prepared for NELAC. As noted earlier, an attachment is provided for each unit that details the findings of the review team.

A. Chromatography and Liquid Chromatography / Mass Spectrometry (attachment 7)

The staff of the unit seems to interact and communicate well with each other. Analysts are familiar with SOPs and the QA/QC Organic Chemistry guidelines. The review indicated that "E codes" are consistently applied, and analysts are well trained in Chemstation and Target data reduction software. The review team has a few suggestions for a good unit.

1. Analysts are working at the limit of personnel and instrumentation to analyze the 3,000 schedule 6090 samples. This unit will probably need more resources if the sample load remains constant at the present level.

The NWQL purchased two additional LC/MS units and is currently training additional preparation and analytical personnel to meet the needs of our customers.

2. Analysts are aware of possible compound degradation in samples. Compounds, such as atrazine, can degrade at room temperature. Because of the possible degradation of compounds at room temperature, the large multi-day sample sets may not be a good practice. The team was told at the unit debriefing that single-day sets are loaded in the autosamplers.

The analysts will be reminded to add only single-day sets into the autosamplers, and to make sure the autosamplers are kept at 4 degrees Celsius +/- 2 degrees Celsius. These steps will ensure data integrity and minimize analyte degradation.

3. The team suggests that the unit set guidelines to 1) decide if a peak needs reintegration and 2) record the area of the peaks that an analyst reintegrates. These guidelines could be used to determine if analysts agree when to reintegrate and how to draw the baseline for manual reintegration.

The Organic Chemistry Program is working with the Quality Management Program to develop guidelines for the identification and quantification of all analyses in the Organic Chemistry Program. These guidelines would include secondary data review policies and all associated documentation. A recommendation on the above topic will be made no later than the end of this calendar year.

B. Gas Chromatography and Gas Chromatography / Mass Spectrometry
(attachment 8)

The review team found that there was enthusiasm for improvement and initiative to follow through in both units. The team found the units well organized, and the laboratory used by the units is well kept. The documentation is thorough, and the reviewer was able to follow an implementation process. The team has a few comments to help improve two good units.

1. No SOPs are available for Schedules 1324, 1396, 1364, and 1319. These procedures need to be documented as soon as possible.

Standard Operating Procedures (SOP) are currently being written for schedules 1324, 1398, 1364 and 1319. (Note that schedule 1396 stated in the review is really schedule 1398.) The SOPs should be approved for use in approximately 4 months.

2. The GC methods should be revised to reduce the amounts of solvents and labor needed.

One of the long-term goals of the Organic Chemistry Program is to minimize the use and exposure of solvents used in the program. This reduction would include the use of solid-phase extraction techniques, the use of robotic systems, and the optimization of available extraction techniques. The Organic Chemistry Program relies on MRDP to develop and prove out method revisions such as this. The NWQL cannot identify specific dates for reducing solvent use, but we are committed to achieve this goal.

3. The team recommends that the unit strongly consider changing from liquid – liquid extraction to solid phase extraction (SPE). A method change to SPE would reduce the labor and solvents required for the process.

See response to V.B.2.

4. The team suggests that review of data by another analyst improves the consistency of data and provides for exchange and increased communication and is commendable.

The secondary data review policy, in place throughout the Organic Chemistry Program, helps to minimize errors and does facilitate communication between analysts.

5. As in other units, there is an expectation that Starlims will provide another higher level of QC documentation; for example, more efficient control charts. The team suggests that supervisors determine that Starlims will meet their and the unit staff's expectations. If not, the units need to examine ways to provide the analysts the QC data the analysts feel they need.

The Organic Chemistry Program currently has detailed two people (including one supervisor) to the Computer Services Unit in order to facilitate the implementation of StarLIMS into the Program. This detail and the input of the analysts will be used to determine if StarLIMS will fulfill expectations regarding QA/QC data assessments.

6. It was suggested that some sample mix-ups have occurred; and, in the data-set examine by the review, an internal standard was added at twice the normal concentration. Error correction is a time-consuming process. The team suggests that the unit staff discuss possible improvements.

The Organic Prep Unit has separate vials to store internal standard and surrogate solutions. The vials for these 2 solutions used to be identical in appearance. In the past year the unit changed this so that the vials for the 2 solutions are different. This change should help the Prep unit staff to easily select the correct solutions. In addition, the Prep Unit staff is now being rotated through the prep areas for the various methods on a 3 to 6 month interval. This will result in more frequent cross-training of staff and review of the SOPs

7. The addition of an internal standard prior to the sample concentration seems to be a practice that is outside of the norm for most EPA protocols, and this practice needs to be evaluated as soon as possible. (See last paragraph on page 3 of attachment 8 for complete details.)

A team from the Organic Chemistry program, the Methods Research and Development Program, and the Quality Assurance Unit is currently examining the internal standard issue. The NWQL believes that a consensus decision needs to be made that includes representation from throughout the Water Resources Division because the current practice on use of internal standards varies from lab to lab in WRD. A Policy Memo on the use of internal standards should be released from the Office of Water Quality. The NWQL does not believe that implementation of changes could be made before a consensus decision is reached. Probably not before the start of the 2002 Water Year. The organic and Quality Management Program Chiefs will further define the issues regarding the use of internal standards and advance the issue with OWQ staff.

8. The review team suggests that congener PCB analysis is the current procedure that is prevalent in the literature and the procedure of choice for tissues. The team realized that currently the customer base for these analyses are limited. This might be because the District project staff needs training about congener analysis to develop additional programs and enlarge the customer base.

The Organic Chemistry Program and the Methods Research and Development Program are currently developing PCB congener methods for water, sediment, and tissue. Progress on method development effort will be assessed prior to the start of the next water year

C. Volatile Organic Carbon (VOC) (attachment 9)

The VOC unit has both the skill and experience needed to generate data of high quality. The analysts are very familiar with the SOP and follow it closely. The records and documentation are well kept, and there is accurate and traceable handling of the standards. The review team has a few suggestions that should help a good unit.

1. The unit should request more detailed information from the commercial standard providers. The documentation should include:
 - a. Neat standards – actual volumetric and gravimetric amounts add to the mix and the measurement technique used.
 - b. Diluting methods – source, purity, and lot number.
 - c. Individual compounds – original source, purity, and lot number of each compound.

The Volatile Organic Carbon Unit evaluated vendors to ensure they could provide this information. An order for new VOC standards was made in March that included a requirement to provide the documentation suggested by the review team. The documentation provided was very helpful and didn't add to the cost of the standards. The Unit plans to share the information on requesting standards documentation with other units throughout the laboratory so that everyone can obtain required documentation from the vendors.

2. The preparation of stock solutions, storage of standards and stocks, and documentation in the standard notebook are all GLP. Calibration curves should be defined on a set schedule even though the CCV solution analysis indicates the relative standard deviation of the response factors are within the acceptance criteria in the SOP.

The Organic Chemistry Program is currently developing a policy for the use of CCV's for calibration. A policy of the time frame, criteria, and use will be forth coming in the next 4 months, and will be included in the Organic QA Manual

3. The review team was impressed with the efforts to qualitatively and quantitatively evaluate the analytical data. Also, the wealth of QC information available to the analysts is substantial. Again, much of these data are not directly available to the customers and clients of the NWQL, and the review team suggests that these data become available to the customers as soon as practical.

The Organic Chemistry Program recognizes that QA/QC data generated is valuable to its customers. The STARLIMS database will allow the NWQL to store a vast amount of the online QC data. An important objective of the StarLIMS implementation effort is to work with the laboratory customers to assess ways to provide this QA/QC data. It is envisioned that the QA/QC data will be provided through a combination of web based applications and data reports similar to those offered for regulatory analyses. The NWIS database is not capable of storing laboratory QA/QC data at the present time.

D. Carbon, UV absorbance, and Methylene blue active substance
(attachment 10)

The reviewer was impressed with the effort, knowledge, and skill available in this part of the unit. For the most part, the SOPs were available and were being followed by the analysts. The carbon analysts have participated in the intercomparison managed by the NRP and compared well with other laboratories. A few suggestions are provided to improve a well-functioning unit.

1. CCV sample results for the Dohrmann Phoenix instrument need to be evaluated on a defined schedule, and a formalized process to assess the stability of the calibration curve should be in the SOP.

The Organic Chemistry Program is currently developing a policy for the use of CCV's for calibration. A policy of the time frame, criteria, and use of CCV data will be forth coming in the next 4 months and will be included in the Organic QA Manual.

2. The team recommends that the CCV include a compound that is more difficult to digest such as caffeine. This procedure should allow the analyst to better assess the method.

The Carbon Unit made a preliminary evaluation of the digestion capabilities for the TOC and DOC methods with caffeine and found that there was better recovery for the more rigorous TOC digestion. The Carbon Unit supervisor attended the PittCon Meeting this spring and evaluated 4 new instruments to better oxidize difficult DOC compounds. A project plan has been developed to run 7 replicate caffeine samples for both methods. The testing will be conducted once modifications are made for the new DOC filtration process. It is expected that the replicate caffeine sample tests will be conducted in June.

3. Customers obtaining UV absorbance data should be made aware that some samples have precipitated, and these precipitates were removed from the sample before analysis. The precipitate may contain UV absorbing compounds.

Analysts currently keep handwritten notes for samples requiring filtration to allow the analysis to be done. It has been difficult to share this information with our customers. The next release of NWIS and the STARLIMS implementation should allow the NWQL to store notes in our databases and inform our customers when a sample has been filtered.

4. The methylene blue active substance analysis SOP needs to be consistent with current practices in the unit.

Upon review of the SOP a few minor changes were required to include acceptance criteria for the standard curve. The analyst is currently making updates to the SOP (May 2000).