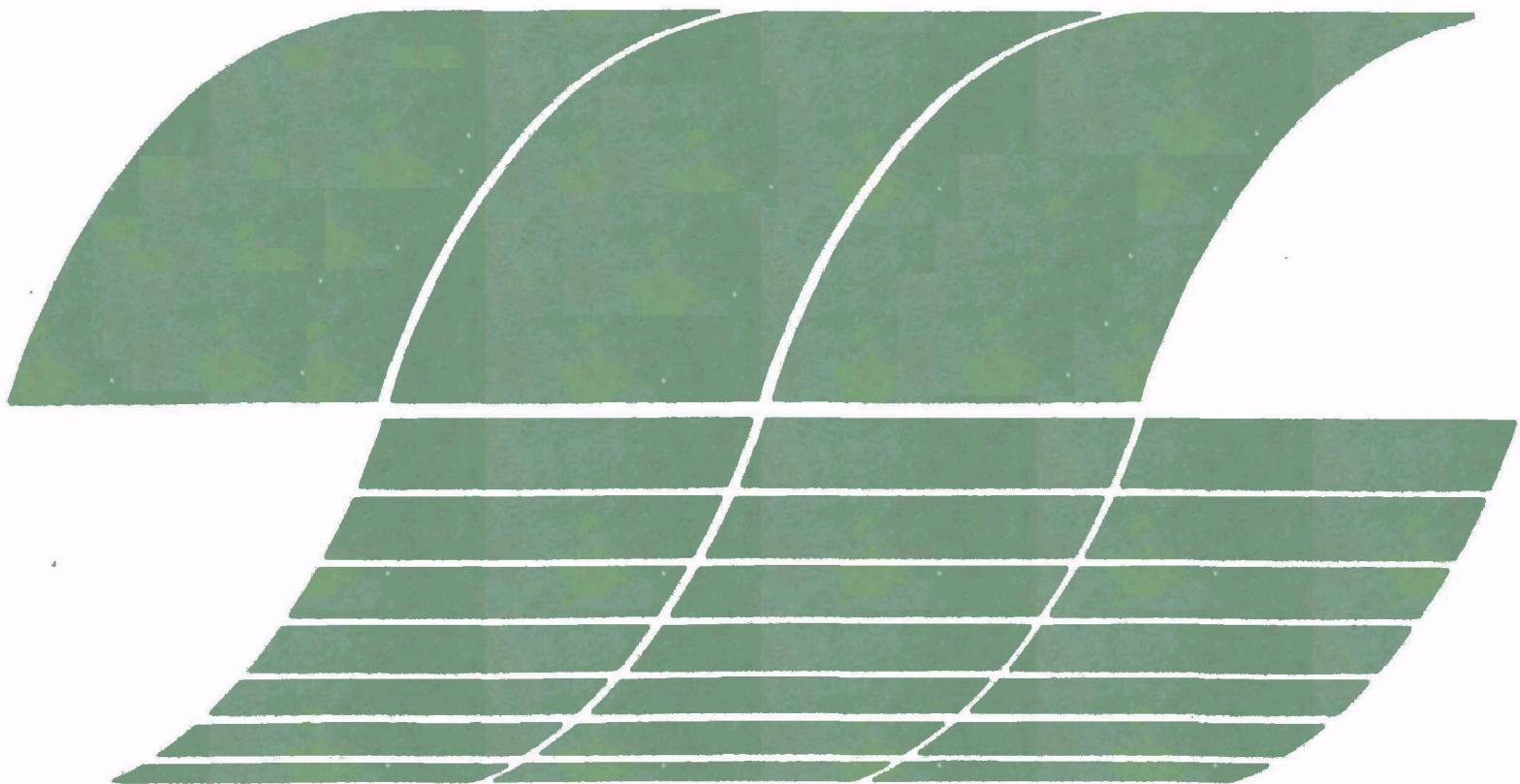


Research and Development



Evaluation of the U.S. Geological Survey Laboratory Denver, Colorado

Interagency
Energy/Environment
R&D Program
Report



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EVALUATION OF THE
U.S. GEOLOGICAL SURVEY LABORATORY
DENVER, COLORADO

by

Robert L. Booth
Office of the Director
Environmental Monitoring and Support Laboratory
Cincinnati, Ohio 45268

US EPA
Headquarters and Chemical Libraries
EPA West Bldg Room 3340
Mailcode 3404T
1301 Constitution Ave NW
Washington DC 20004
202-566-0556

ENVIRONMENTAL MONITORING AND SUPPORT LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

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FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents. The Environmental Monitoring and Support Laboratory-Cincinnati conducts research to:

- o Develop and evaluate techniques to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.
- o Investigate methods for the concentration, recovery, and identification of viruses, bacteria, and other microbiological organisms in water. Conduct studies to determine the responses of aquatic organisms to water quality.
- o Conduct an agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastewater.

This report summarizes the results of an evaluation of the capabilities of the U.S. Geological Survey Laboratory, Denver, Colorado, to provide valid data for the analyses of water and waste samples on a variety of contaminants and parameters commonly requested by the U.S. Environmental Protection Agency.

Dwight G. Ballinger
Director
Environmental Monitoring and Support
Laboratory - Cincinnati

ABSTRACT

An onsite evaluation was made of the capabilities of the U.S. Geological Survey Laboratory at Denver, Colorado. Particular emphasis was placed on determining their ability to meet the monitoring requirements connected with their contractual efforts with the U.S. Environmental Protection Agency. This monitoring is a major part of the Environmental Protection Agency's quality assurance program in support of energy-related activities in the western United States.

Overall, water analyses for parameters related to the broad areas of chemistry, organics and pesticides, and radiochemistry are being conducted by approved sampling/preservation techniques and laboratory methods. Results on unknown performance samples and cross-check samples were well within acceptable ranges, thus documenting their ability to generate valid data. Specific recommendations for areas of improvement are given.

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SECTION 1

EXECUTIVE SUMMARY

An onsite evaluation was made of the capabilities of the U.S. Geological Survey Laboratory at Denver, Colorado. Particular emphasis was placed on determining their ability to meet the monitoring requirements connected with their contractual efforts with the U.S. Environmental Protection Agency (U.S.EPA). This monitoring is a major part of EPA's quality assurance program in support of energy-related activities in the western United States.

Team members consisted of Dwight G. Ballinger, Director, and Robert L. Booth, Deputy Director of the Environmental Monitoring and Support Laboratory - Cincinnati, Office of Monitoring and Technical Support, Office of Research and Development. A separate visit was made by David G. Easterly of the Environmental Monitoring and Support Laboratory - Las Vegas to determine their radiochemistry capabilities.

Overall, water analyses for parameters related to the broad areas of chemistry, organics and pesticides, and radiochemistry are being conducted by approved sampling/preservation techniques and laboratory methods. Results on unknown performance samples and cross-check samples were well within acceptable ranges, thus documenting their ability to generate valid data. The laboratory has ample space, services, and equipment. The present staff is composed of well-qualified, conscientious scientists and technicians. There is a need, however, for more senior professional staff to conduct project studies concurrently with the technicians responsible for laboratory analyses. Additional manpower is also needed in the organics program to establish the precision and accuracy of their methodology and to institute a daily program of quality control techniques.

Although approved sampling/preservation techniques are followed, holding times are often exceeded even before arrival at the laboratory. This problem must be corrected. In addition, a thorough review at the management level concerning more effective use of standard reference materials, duplicates, and spikes is strongly recommended.

SECTION 2

BACKGROUND

An evaluation was made of the U.S. Geological Survey (USGS) Laboratory at Denver, Colorado. The evaluation consisted of preliminary questionnaires and onsite visits. Radiochemistry was reviewed January 10-12, 1977, and water chemistry, January 31 through February 2, 1977. The visits included an initial briefing session attended by the USGS senior staff and chaired by Russell McAvoy, Laboratory Director; a general laboratory tour; two days of detailed discussions with their staff (ranging from the respective section chiefs to the personnel working at the bench and responsible for processing the collected samples); and a debriefing attended by senior staff and members of the evaluation team to discuss the preliminary findings. Subsequently, unknown performance samples were provided by the Quality Assurance Branch, Environmental Monitoring and Support Laboratory - Cincinnati to document their ability to generate valid data in the areas of trace organics, minerals, nutrients, demand, and trace metal analyses.

SECTION 3

WATER CHEMISTRY

(Reported by Dwight G. Ballinger and Robert L. Booth, EMSL-CI)

Sampling, Preservation, and Holding Times

Most samples are collected by personnel from the USGS District Offices/Laboratories and shipped to the Denver Facility for processing and analyses. Approximately 80,000 samples are received, and 20 to 30 tests per sample are made, generating 3 to 4 million pieces of data. Minnesota and all states west of the Mississippi are serviced. Approximately 75% of the samples are surface waters, 20% are groundwater, and 5% are other. At present, no samples are being analyzed for either the Safe Drinking Water Act (SDWA) or the National Pollutant Discharge Elimination System (NPDES).

The Sample Receiving Department is responsible for providing proper containers for sample collection in the field:

1. For trace metals, use polyethylene that has been acid-rinsed; it may be reused.
2. For inorganics, use polyethylene containers direct from the manufacturer once only.
3. For TOC and organics, use glass. Filtration and preservation are done in the field per approved techniques. All samples are processed as soon as possible and are held at least 14 days after data are stored in the terminal.

Recommended holding times are not being observed. There was ample evidence of samples with holding times that were exceeded even before arrival at the laboratory. Internal lab tour also surfaced TOC samples that were 2 to 3 weeks old, boron samples over 6 months old, and pesticide samples approximately 3 months old.

RECOMMENDATION: Institute an immediate program to rectify problems in (1) sample shipment (adhere to recommended holding times), (2) sample receiving (process samples in shortest turn-around time possible), and (3) the laboratory (guarantee timely analyses of received samples).

Analytical Methodology

The laboratory has its own methods that do not necessarily check completely with USGS Techniques of Water Resources Investigations series. For the most part, however, methodology appeared to be by approved methods manuals. Potential exceptions to 304(g) test procedures are boron, antimony, molybdenum (need to do extraction techniques), arsenic, COD, cyanide, color, and turbidity.

RECOMMENDATION: Document the methodology used in more formal fashion, determine where variances are present, and submit as proposed alternative test procedures for EPA consideration.

Personnel

The Denver facility has 47 permanent and 35 temporary positions. The ratio of technicians to professionals is approximately 2:1. They are mandated to contract 0.5 million in FY 77 and 1.5 million in FY 78 for analytical services that will require strict quality control. Overall, the staff is composed of well-qualified, conscientious scientists and technicians. There are at least two areas, however, that need additional manpower.

RECOMMENDATION:

1. Provide additional senior staff to conduct project studies.
2. Provide additional professional staff to the Organics Section to prevent backlog of samples, document quality control programs, and institute a daily quality control practice.

Space and Services

The Denver facility is a comparatively new one-floor structure. The space has been utilized in a most efficient manner, and services are excellent. Except for some minor first aid items that were verbally noted in the debriefing, no problem areas were noted.

Equipment

The Denver facility is exceptionally well equipped and capable of performing all the routine analyses in water and waste monitoring. The facility has the latest equipment, particularly, in atomic absorption spectroscopy instrumentation, Technicon equipment, and gas chromatography instrumentation.

Data Handling and Review

The Denver facility is currently expanding its computer handling of

all data by going to an online lab automation system via contract with Lawrence Livermore Laboratories that will be similar to EPA's system operational at EMSL-CI and Region V's Central Laboratory. Their Quality Assurance Section has the responsibility for reviewing all data before it is reported and checking back with the respective Section Chiefs on any problem areas. Results of performance on standard reference materials (USGS water samples used as Water Quality checks) and computer logic checks are the two basic mechanisms used for determining the validity of the reported data. The Quality Assurance Section does not routinely utilize results of performance on replicates (precision) and spiked (accuracy) samples. Thus for measurements that do not have unknown Standard Reference Materials (SRMs), there are no quality control checks other than what the analyst does in preparation of calibration curves.

RECOMMENDATION: A thorough review should be made at the management level to determine the most effective ways of utilizing quality control data generated from SRMs, duplicates, and spikes.

Quality Control

The older version of their quality control program was available for review, but a newly documented plan should be available in the near future. We understand it will be coordinated by Bernard Malo of USGS. Key elements of the present system are:

1. Use of standard water reference samples furnished by Marv Fishman at least 1 to 3 times per day for major cations/ions.
2. Use of these samples at least twice per week for metals.
3. Submission of samples as unknowns to analysts.
4. Next-morning computer evaluation: Any value that exceeds $1.5 \times SD^*$ is flagged and reviewed by the section responsible for analysis.
5. A new Quality Assurance Section that submits samples to District Offices, who in turn ship into Denver on a 1-per-week basis. Use of dummy stations again, the following morning results are reviewed. The computer simply states, for example, "chloride missed."
6. Use of standard water reference samples as known values by section chiefs.
7. Use of nutrients standard only (used only as a check on standard curve).
8. Rejection rate of approximately 12%.

*SD from Skougstad's/Fishman's RR studies.

9. Use of demand Analyses Sets not as quarterly samples but only as checks on standard curve.
10. Request for pesticides/organics quality control samples through Harold Clements, EMSL-CI. (Note: These have since been made available).
11. Performance of approximately 100 checks, including ion/cation balance checks, that are done automatically by the computer; (ditto for total versus dissolved, etc.).

The Organics Section has a quality assurance program that operates independently of the above general practice. Key elements of its present system are:

1. Quality assurance practice
 - a. Dosed samples (spiked)
 - (1) Once/month
 - (a) Use levels from lower (0.5 to 1.0 ppt) detection on up to 0.5 to 1.0 ml acetone.
 - (b) Spike into distilled water only; want to expand to natural waters.
 - b. Check samples. Use Applied Science and their own; will want to use EMSL's.
 - c. Blind samples. Hope eventually to have SRM samples (still some question as to how real values will be determined).
 - d. Duplicates. Don't use on water; some in sediments.
 - (1) Have done limited sample splitting with Atlanta facility.
 - e. Multi-lab studies. ASTM roundrobins.
2. Performance evaluation
 - a. No established criteria for rejection of results (Need SRM's or equivalent).
 - b. Unestablished precision of methods (strive for $\pm 10\%$ in standards).
 - c. No bias data.

d. No formal program for repeatability (again, strive for $\pm 10\%$).

- RECOMMENDATION:
1. National Bureau of Standards (NBS) thermometer is needed to verify readings on ovens.
 2. Formal use of class S weights is needed to check out balances.
 3. Methodology needs to be documented by establishing the precision and accuracy of methods.
 4. More frequent use needs to be made of duplicate and spiked samples rather than almost total dependence on SRMs.

SECTION 4

(Reported by David G. Easterly, EMSL-LV)

On the afternoon of January 10, 1977, I met with Russell L. McAvoy, Vic Janzer, and Bernard Malo of the USGS. Doug Skie of EPA also attended the meeting. A general discussion on the purpose and mode of operation of the visit was held with the above personnel followed by a tour of the USGS laboratory.

On January 11, a more detailed discussion as well as an onsite inspection of the USGS facilities was conducted. Topics of discussion included staffing, facilities, equipment, quality assurance program responsibilities, and general laboratory operations.

The present staffing appears very knowledgeable in the field of radio-chemistry, and all are very well experienced in this field. For the present workload at the USGS laboratory, and particularly for any expansion, additional professional personnel should be considered. Even though all personnel are well experienced, it is felt that additional periodic training would be advantageous. The laboratory facilities and equipment are excellent, and the general laboratory operation appears excellent.

All of the USGS personnel I talked with seemed aware of the importance of good quality control practices and indicated a sincere desire to produce data that are scientifically defensible. These observations are also supported by the good results obtained on samples they have analyzed from the EMSL-LV cross-check program. The following suggestions are offered for consideration, however:

1. A written quality control plan should be developed. Each branch and, in some cases, each project should have such a plan.
2. A routine performance check should be conducted on all balances, including documentation in log books.
3. Although background tables are kept on a continuing basis quality control charts should be employed in the laboratory operation.
4. A planned service maintenance program should be considered for all counting equipment.

On the morning of January 12, a debriefing was held with Russell McAvoy, Vic Janzer, and Bernard Malo, and the above topics were discussed.

Again, it is felt that the personnel at the USGS laboratory are doing a good job and are aware of the importance of good quality control practices.

SECTION 5

PERFORMANCE EVALUATION

(Conducted by John A. Winter, EMSL-CI)

Performance evaluation samples were analyzed by the laboratory staff for the following groups: trace metals, minerals, nutrients, demands, and trace organics. Results of these analyses were reported directly to the Quality Assurance Branch, EMSL-CI, and processed by means of a computer program that compared reported values to actual values in relation to results from previous EPA method studies or from similar interlaboratory data.

Overall, the laboratory staff had an outstanding performance. With the exception of values for low-level specific conductance, low-level sulfate, and high-level methoxychlor, all reported values were well within the warning limits (95% confidence level). The values for conductance and sulfate were still well within the acceptance limits (99% confidence level), and the potential problem with methoxychlor was resolved in subsequent analyses of additional check and performance samples. Thus the laboratory ultimately showed satisfactory performance for all of the parameters cited in the following major groupings:

Trace Metals

Aluminum
Arsenic
Beryllium
Cadmium
Cobalt
Chromium
Copper
Iron
Mercury
Manganese
Nickel
Lead
Selenium
Vanadium
Zinc

Nutrients

Ammonia-N
Nitrate-N
Orthophosphate
Kjeldahl-N
Total Phosphorus

Demands

COD
TOC

Organics

Endrin
Lindane

Minerals

pH
Specific Conductance
TDS
Total Hardness
Calcium
Magnesium
Sodium
Potassium
Total Alkalinity
Chloride
Fluoride
Sulfate

Organics (Continued)

Methoxychlor
Toxaphene
PCB (1254)
PCB (1016)
PCB (1016 and 1254)

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