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	Engineering and Design INTERLABORATORY TESTING PROGRAM FOR CHEMICAL ANALYSES	
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## DEPARIMENT OF THE ARMY U.S. Army Corps of Engineers Washington, D.C. 20314-1000

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# Engineering and Design INTERLABORATORY TESTING PROGRAM FOR CHEMICAL ANALYSES

- 1. <u>Purpose</u>. This regulation establishes an interlaboratory chemical analyses testing program, for Corps of Engineers (CE) laboratories performing water quality and sediment testing and related chemical analyses. This program is designed to increase the validity of CE analytical data by improving accuracy and precision.
- 2. <u>Applicability</u>. This regulation is applicable to all CE district, division, and research laboratories performing water quality and related chemical analyses. CE district, division and research laboratories are expected to participate in the program within the limits of their capabilities with respect to personnel and instrumentation. The Missouri River Division may use the results of the interlaboratory testing program, established by this regulation as part of its validation, in accordance with reference 3d, of CE laboratories for hazardous and toxic waste projects.

#### 3. References:

- a. ER 1110-1-8100, Laboratory Investigations and Materials Testing;
- b. ER 1130-2-415, Water Quality Data Collection, Interpretation, and Application Activities;
  - c. ER 1110-1-261, Quality Assurance of Laboratory Testing Procedures;
- d. ER 1110-1-263, Chemical Quality Management Toxic and Hazardous Wastes:
  - e. ER 1180-1-6, Construction Quality Management;
- f. Precision Measurement and Calibration, NBS Special Publication 300-Volume 1, February 1969, U.S. Department of Commerce, U.S. Government Printing Office, Washington, DC;
- g. ASTM Method D 3856, Annual Book of ASTM Standards, Vol 11.01, 1916 Race Street, Philadelphia, PA 19103;
- h. ASTM Method D 2777, Annual Book of ASTM Standards, Vol 11.01, 1916 Race Street, Philadelphia, PA 19103;

- i. ASIM Method D 3975, Annual Book of ASIM Standards, Vol 11.02, 1916 Race Street, Philadelphia, PA 19103;
- j. Statistical Manual of the Association of Official Analytical Chemists (AOAC), 1975, W. J. Youdan and E. H. Steiner, AOAC, 1111 N. 19th Street, Arlington, VA 22209.

#### 4. General:

- a. Most government agencies with responsibilities requiring the operation of chemical laboratories have instituted some type of interlaboratory testing program (e.g. National Institute for Occupational Safety and Health, United States Environmental Protection Agency, United States Geological Survey, Food and Drug Administration, Drug Enforcement Administration, Department of Agriculture, etc). The USACE programs for quality assurance and quality control (QA/QC) for water and other chemical analyses can be broadly divided into two classifications: interlaboratory and intralaboratory. An intralaboratory QA/QC program assists a single laboratory in improving the accuracy and precision of its data. It provides systematic guidelines concerning analytical methodologies and techniques to help laboratory personnel produce the highest quality data possible. An interlaboratory QA/QC program involves analysis of identical samples by multiple laboratories in order to assess the continuing capability, performance, and progress of each participating laboratory. In addition, proficiency testing indicates, on a mathematical basis, the degree of confidence that can be placed in the results of sample analysis, and identifies analytical areas needing further attention.
- b. Another positive attribute of an interlaboratory testing program is the collaborative testing of methodologies. This type of testing is the evaluation of an analytical method by the participating laboratories, under actual working conditions, by analyzing portions of carefully prepared homogeneous samples. The analytical results are examined for bias and for inter- and intralaboratory variability to determine if the method is suitable for use. Collaborative methodology testing measures the performance of a method in terms of accuracy, reproducibility and repeatability.
- c. Although standard methods are used, situations continually arise requiring new method development or modifications to existing methods. The pooling of efforts by participating USACE chemical analytical laboratories in a collaborative testing program will be advantageous.
- 5. <u>Interlaboratory QA Program Objectives</u>. The basic objectives of an interlaboratory proficiency testing program are:
- a. To provide a measure of the precision and accuracy of analytical methods run routinely by different laboratories;
  - b. To evaluate the precision and accuracy of results between laboratories;

- c. To provide documentation of precision and accuracy data to clients of laboratories;
  - d. To identify weak methodologies;
  - e. To detect training need;
- f. To upgrade the overall quality of performance of all participating water quality laboratories; and
  - g. To improve communication among laboratory personnel.
- 6. <u>Interlaboratory Testing Program for Water and Chemical Analysis</u> Laboratories.
- a. References 3 (a)-(d) provide requirements for a continuing effort to improve testing. As part of these efforts, this interlaboratory testing program has been developed. Implementation of this program will emphasize to each USACE district, division, and research laboratory the importance placed upon a quality product. This program will demonstrate to other government agencies and the public the total commitment to quality maintained by the USACE.
- b. The interlaboratory program is administered by a coordinating laboratory which sends uniform samples to each participating laboratory for analysis. The coordinating laboratory will be CEWES-EE. The resulting data is returned to the coordinating laboratory on a special report form. The coordinating laboratory then processes the data, performs a statistical analysis, prepares a report and distributes the final results to each participating laboratory.
- c. This program will be initiated in FY 90, and division laboratories should program funds in anticipation of participating in this program.
- 7. <u>Coordinating Laboratory Responsibilities</u>. The US Army Engineer Waterways Experiment Station (CEWES-EE) Analytical Laboratory Group, as the coordinating laboratory, will be responsible for the following:
- a. Preparing homogenous samples and distributing them to the participating CE laboratories for analysis using criteria outlined in 3(g) (i).
- b. Providing report forms, methodologies and other instructions. When methodologies are not specified each laboratory must specify the methodology which was used:

- c. Providing instructions pertaining to maximum sample holding times and requirements for data submission times back to the coordinating organization.
- d. Collating the data, performing a statistical analysis, and sending a report to the participating laboratories; and
- e. Sample test results not within 2 standard deviations of the grand average values will be considered a discrepancy, and quidance and assistance in improving the laboratory performance will be provided.
  - f. Maintaining the confidentiality of the laboratories.

# 8. Types and Preparation of Sample Media.

- a. Various types of routinely tested samples will be used. In addition to samples prepared by WES, reference materials obtained from United States Environmental Protection Agency, National Bureau of Standards, and the private sector may be used. The sources and types of reference samples are varied so that laboratories are not able to anticipate the results of analyses. Procedures for preparing and distributing samples are provided in Appendix A.
- b. Along with the samples, each participant will receive a cover letter providing the protocol for handling the sample. The time allowed for analyzing and reporting results corresponds to the normal time for processing a similar routine sample.

#### 9. Evaluation of Reported Data.

a. The completed report forms from each participating laboratory will be sent to CEWES-EE and the quantitative results will be entered on a summary sheet. If any results appear obviously and grossly erroneous, the responsible laboratory will be immediately contacted and given an opportunity to review its work and change the report if a simple error was made in computation and/or transcription. After all results are recorded, a statistical analysis of the results is performed using procedures described in 3(j). The reports, statistical analyses, and any specific comments concerning particular irregularities will be mailed to participating laboratories and to CEEC-EH-W and CEEC-EB. Reports of data for laboratories providing QA for Superfund and DERA will also be submitted to CEMRD-ED-L for use as an ongoing QA check on laboratory performance. A private critique of performance will be sent to each laboratory exhibiting a special need for help.

FOR THE COMMANDER:

ALBERT J. ÆNETTI, JR:/ Colonel, Corps of Engineers

Chief of Staff

Appendix A - Program procedures

#### Appendix A

# Interlaboratory Quality Assurance Testing Program For Corps Laboratories

# Samples To Be Submitted For Testing

<u>Media</u>	<u>Chemical Parameters</u>
Water	Metals
Water	Nutrients, NH4-N, NO3-N
Water	PCB's
Water	
Water	Volatile Organics Base Neutral Acid Extraction.
Water	Chlorinated Pesticides
Water	Chlorinated Herbicides
Water	Oil & Grease/Total recoverable hydrocarbons
Water	Specialty Parameters as requested
Sediment/Soil	Total metals
Sediment/Soil	Base Neutral Acid Extraction
Sediment/Soil	PCB's
Sediment/Soil	Specialty Parameters as requested
Tissue	Parameters as requested

- a. Samples will be submitted on a quarterly basis, and sample media and parameters will be rotated through the program annually. However, the schedule for submission will be flexible to respond to the types of analyses being performed most frequently by the Division Laboratories or those most sensitive to current demands for proficiency. A poll will be taken of Corps laboratories each year to assure the coverage of pertinent analyses. The schedule may be altered to meet specific needs. The submission of samples for specialty analyses may also be used to validate methods or investigate new methods of analysis needed for Corps projects. Detailed procedures for analysis will be submitted with these samples.
- b. One month prior to the submission of samples CEWES-EE will send information to the Corps laboratories concerning the availability of the QA samples. A form will be provided which must be returned to CEWES-EE stating the laboratory's ability to participate. This will allow the CEWES-EE to prepare sufficient sample for analyses.

ER 1110-1-1401 15 May 1989

c. Since the primary purpose of this program is to improve the quality of Corps data, sample size will be large enough so that duplicate or triplicate analyses can be performed. This will allow an evaluation of precision as well as accuracy. Acceptable methods and detection limits for performing the analyses will be provided with the samples. Reporting requirements such as calibration curves, method blanks, internal standards, surrogates, chromatographic, mass spectra, etc., will also be included together with a time table for data submission.