FOURTH QUARTER PROGRESS REPORT FY 87



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



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICE OF RESEARCH AND DEVELOPMENT

ENVIRONMENTAL MONITORING AND SUPPORT LABORATORY

CINCINNATI, OHIO 45268

DATE: December 28, 1987

SUBJECT: FY87 Environmental Monitoring and Support Laboratory - Cincinnati

(EMSL-Cincinnati) Fourth Quarter Progress Report

FROM: Kathie L. Fieler, Program Analyst

Environmental Monitoring and Support

Laboratory - Cincinnati

TO: SEE ADDRESSEE LIST

Attached is a copy of the EMSL-Cincinnati fourth quarter progress report. For your information, a few of the highlights of the various projects/activities this quarter include:

- * A meeting was held during this quarter to plan for the revision/updating of the manual "Microbiological Methods for Monitoring the Environment". For details, please refer to Project 02.
- * Details concerning the referee analyses, the quality control (QC) sample program, performance evaluation (PE) samples, and the water pollution (WP) studies are available in Project 04.
- * Work continued during the quarter to develop a general purpose liquid chromatography/mass spectrometry method for volatile organic compounds in water. Please refer to Project 05.
- * Further details concerning the three 304(h) notices to meet National Pollutant Discharge Elimination System (NPDES) needs are outlined in Project 06.
- * Annual Discharge Monitoring Report-Quality Assurance (DMR-QA) Study 7 was completed. See Project 08 for details.
- * A report entitled "Methods for Selected Synthetic Organic Compounds Summary Report" was completed. Further information is provided in Project 10.
- * Project 12 provides details concerning the method validation studies conducted by our Laboratory.
- * Specifications were provided to the Office of Emergency and Remedial Response for a format for inorganic data deliverables on computer readable media--see Project 22.

If you have any questions, please give me a call at FTS 684-7304 or contact the Project Officers directly.

Attachment (1): As Stated

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ADDRESSEE LIST--PAGE 2

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EMSL CINN: JULY - SEPIEMBER 198/

CODE TITLE

BUDGET SUB-ACTIVITY: B101 WATER QUALITY

ISSUE: A WQBA/PERMITTING

PPA (L): 03 REFERENCE BIOLOG. METHODS - WATER QUALITY

PPA (S): METHODS/BIOLOGICAL

RC: A EMSL CINN

PROJECT: 01 REFERENCE BIOLOGICAL METHODS - WATER QUALITY

PROJECT OFFICER: CORNELIUS I. WEBER PHONE: 513-684-7337

PROJECT DESCRIPTION:

GOAL: Provide standardized biological tools for monitoring the toxicity of fresh and marine waters and wasterwaters, for determining the effect of pollutants on aquatic ecosystems, and to support inclusion of biological methods in the regulatory processes.

RATIONALE: The project addresses Agency needs for scientifically valid and legally defensible biological methods to support the Water Quality Based Approach to NPDES permit limitations and provide information concerning the quality of ambient waters and wastewaters.

APPROACH: Work will continue to evaluate, standardize, compile, and publish biological monitoring tools supportive of wasteload allocation, water quality based NPDES permits, use-attainability studies, and site-specific water quality modifications. Methods for culturing and testing of freshwater and marine organisms will be upgraded, standardized, and published. Methods will be provided for monitoring water and wastewater toxicity to aquatic ecosystems, for evaluating toxicity of sediments and sludges to freshwater and marine organisms, for determining toxic components of complex effluents, and measurements of threshold toxicity effects such as histological damage and enzyme activity.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

Ruggedness testing of the Seven-day Fathead Minnow Larval Growth and Survival Test continued. The mean weight (0.636 mg +/-15%) of larval fish fed twice on day one was essentially the same (0.626 mg +/-13%) as for those larval fish fed beginning on day two. The lowest observable effect concentration (LOEC) for hexavalent chromium was the same for both feeding regimes. Larval fish fed beginning on day three were statistically less sensitive, indicating that feeding should begin at least on day two. Several side-by-side tests were conducted with hexavalent chromium and three different age groups of larvae.

The age groups were 24-hours, 4-days, and 7-days old. The LOEC was 3.0 mg/L using 24-hour and 4-day old larvae and 12 mg/L for the 7-day old larvae. Mean weights for control fish were 0.839 mg +/-3%, 0.971 mg +/-12%, and 1.77 mg +/-13% for the 24-hour, 4-day, and 7-day old, respectively. The data indicate that the older larvae are less sensitive to hexavalent chromium than the younger ones and suggest that the test may be initiated with larvae up to at least 4-days old.

A series of seven, seven-day, sheepshead minnow larval growth and survival tests, using copper sulfate and Forty Fathoms artificial sea salts, were completed to determine the single laboratory precision of the method. The mean weight for all the control larvae was $1.60~{\rm mg}$ +/-12%. The LOECs for survival and growth ranged from 100 to 200 ug/L and from 50 to 200 ug/L, respectively.

Nine paired 96-hour algal toxicity tests were conducted using Selenastrum capricornutum and cadmium as a reference toxicant. Algal Growth Potential Medium (AGPM) with ethylenediamine tetraacetate (EDTA) added was used in one set of the tests and AGPM without EDTA was used in the other. Mean control algal growth with AGPM plus EDTA was 1,262,565 +/-26% cells/mL and without EDTA was 349,797 +/-56% cells/mL. The mean cadmium EC50 value with EDTA was 0.447 +/-53% ug/L, and without EDTA was 0.039 +/-79% ug/L. The data indicate that addition of EDTA to the AGPM may significantly reduce the sensitivity of the test with metals.

A draft final report, "Feasibility of Culturing and Testing the Mysid, Mysidopsis bahia, Under Artificial Conditions", was received from Colorado State University, Fort Collins, Colorado. The work was done under a cooperative agreement.

The Journal of Water Pollution Control Federation Annual Literature Review of "Effects of Pollution on Freshwater Organisms," prepared in a joint effort with the Environmental Research Laboratory - Duluth, was published in June, 1987.

STATUS OF LABORATORY OUTPUTS

- B1861 "Report on Comparison of 8-Day Fathead Minnow Embryo-larval Survival and Teratogenic Test and the 7-Day Larval Growth Test". Report completed 6/24/87.
- B706 "Report on Laboratory Culture Techniques and Use of Two Species of Myriophyllum, for Effluent Testing". Report completed 6/29/87.
- B708 "Report on Rapid Tests for Biomonitoring Effects of Effluent in Receiving Waters". Report completed 6/24/87.

STATUS AND SCHEDULE OF DELIVERABLES:

2036 DUE: 09/30/87 REVISED: COMPLETED: 09/28/87
MANUAL OF SHORT- TERM CHRONIC TOXICITY TESTS FOR EFFLUENTS AND SURFACE WATERS FOR USE WITH MARINE ORGANISMS.

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CODE TITLE

BUDGET SUB-ACTIVITY: B101. WATER QUALITY

ISSUE: A WQBA/PERMITTING

PPA (L): 03 REFERENCE BIOLOG. METHODS - WATER QUALITY

PPA (S): METHODS/BIOLOGICAL

RC: A EMSL CINN

PROJECT: 02 REFERENCE BIOLOGICAL METHODS-WATER QUALITY

PROJECT OFFICER: CORNELIUS I. WEBER PHONE: 513-684-7337

PROJECT DESCRIPTION:

GOAL: Provide standardized methods and QA procedures for microbiological analysis of fresh and marine waters; wastewaters, sludges, amd sediment, and for the detection of toxicity and mutagenicity in these media. RATIONALE: This project addressess Agency needs for scientifically valid and legally defensible microbiological data on water quality in support of the Clean Water Act, Marine Protection and Sanctuaries Act, and Resource Conservation and Recovery Act.

APPROACH: Develop or modify, evaluate, standardize and publish methods for the detection and enumeration of microorganisms in environmental samples, including methods for enteric pathogens and indicator organisms in ambient water, wastewaters and sludges. Microbial tests, such as the Ames test, will be developed to measure toxicity and mutagenicity in wastewaters and sludges. Rapid instrumentation methods will be evaluated to determine the effects of toxicants on the natural aquatic biota by measuring changes in bacterial densities, population composition and metabolic activity resulting from environmental stress. Methods for detecting and identifying genetically engineered microorganisms, and recently developed techniques (e.g. gene hybidization, mono/polyclonal antibodies) will be evaluated.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

A proposal from New York University Medical Center for a cooperative agreement to evaluate the Microscreen mutagenicity test was peer reviewed and is being processed.

The report on the comparison of the hydrophobic-grid membrane filter (HGMF) procedure and USEPA approved methods for coliform analysis of water was published in the May, 1987 issue of the Journal of Applied and Environmental Microbiology. The results indicated that recoveries of total and fecal

coliforms by HGMF did not differ significantly from standard methods.

In the task to determine the relative efficiency of three effluent sample preparation methods for the Ames Test, high performance liquid chromatograph (HPLC) analyses and Ames Tests were completed on a nonmutagenic control (reagent grade water prepared by reverse osmosis) and a known carcinogen (para-nitrobenzoic acid) that had been processed through the three extraction procedures - liquid-liquid (LL), resin-packed glass with gravity flow (GF), and resin-packed stainless steel with applied pressure (AP). The HPLC results indicated recoveries of 77.8%, 72%, and 58.2% for LL, GF and AP, respectively. Results obtained with the Ames Test also indicated highest recoveries from LL and lowest from AP. The control showed no indication of contamination from the processing systems.

The report, "Precision and Relative Accuracy of the Membrane Filter and Most Probable Number with Total Coliform Methods with Several Water Types" was completed. The heterotrophic plate count (HPC) method served as the reference method for relative accuracy determinations. No difference was found between the accuracy of the two coliform methods, the analysts, or analysis times. For non-chlorinated effluents, the mean coefficients of variation for the membrane filter (MF), most probable number, and HPC tests were 20.5%, 62.7%, and 23.3%, respectively, while the values for chlorinated effluents were 40.7%, 70.3%, and 25.8%, and for groundwater samples were 43.6%, 86.5%, and 32.6%.

An interim report (2035A) was prepared on the development of methods for identification of salmonellae from sludges, wastewaters, and leachates. This report contains an extensive review of the published literature on recovery of these organisms from the environment. Many variations of qualitative procedures were found but there was only limited information on quantitative and rapid noncultural methods. The report also describes inhouse feasibility studies of potential MF media and filter aids.

An interim report containing a review of the literature and the project plan was completed for the project to prepare samples for microbial analyses and determine total populations in sludge.

EMSL-Cincinnati hosted a two-day meeting of Agency microbiologists to plan for the revision and updating of the Agency manual, "Microbiological Methods for Monitoring the Environment", held at the Andrew W. Breidenbach Environmental Research Center. Section staff participated in a meeting of the American Society for Testing and Materials, Committee D-14 on Water, and led Task Groups on methods for total coliforms and general practice procedures for counting colonies, glassware preparation, and sterilization activities.

STATUS OF LABORATORY OUTPUTS

- B1317 "Report: Evaluation of the Microbial Test for Mutagenicity of Waste Waters". Completed 1/29/87.
- B1330 "Precision and Accuracy of Coliform Methods Annual Report". Completed 6/17/87.

- B719 "Updated Procedures for Total and Fecal Coliforms in USEPA Microbiological Methods Manual". Deleted 3/9/87.
- B800 DUE: 06/87. Report on Methods of Preparation of Sludge Samples for Bacterial Analyses". In revision in Section.

STATUS AND SCHEDULE OF DELIVERABLES:

2035 DUE: 06/30/87 REVISED: COMPLETED: 06/24/87 INTERIM REPORT ON METHODS FOR SALMONELLAE IN MUNICIPAL SLUDGES, WASTE-WATERS AND LEACHATES.

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CODE TITLE

BUDGET SUB-ACTIVITY: B101 WATER QUALITY

ISSUE: A WQBA/PERMITTING

PPA (L): 03 REFERENCE BIOLOG. METHODS - WATER QUALITY

PPA (S): METHODS/BIOLOGICAL

RC: A EMSL CINN

PROJECT: 03 REFERENCE BIOLOGICAL METHODS - WATER QUALITY

PROJECT OFFICER: CORNELIUS I. WEBER PHONE: 513-684-7337

PROJECT DESCRIPTION:

GOAL: Provide standardized methodology for monitoring viruses in surface waters, wastewaters, sediments and sewage sludges as mandated in the Water Quality Act of 1987, the Marine Protection Research and Sanctuaries Act (PL 92-532), and the Resource Conservation and Recovery Act (PL 94-580). RATIONALE: Address Agency's need for collection of scientifically valid and legally defensible information concerning the quality of ambient waters and wastewaters.

APPROACH: Develop methods or adapt existing methods for detection and quantification of enteric viruses in environmental samples. Evaluate, standardize, compile and publish methods for the collection of adequate and representative samples, concentration of viruses so as to obtain manageable volumes for assay, detection of viruses as a consequence of infectivity, and identification of viruses by serological typing. Develop procedures for mammalian cytotoxicity and mutagenicity testing of environmental samples. Conduct interlaboratory collaborative tests on existing and newly proposed virus mehtods.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

A chapter update to the USEPA MANUAL OF METHODS FOR VIROLOGY has been distributed to those entered into the Manual mailing list. This update, Chapter 9, "Cell Culture Preparation and Maintenance", is the first of two replacement chapters on cell culture procedures. The second replacement, Chapter 10, "Cell Culture Procedures for Virus Assay", is currently undergoing peer review.

A revised draft has been received from Baylor College of Medicine for the Cooperative Agreement CR811258 ("Improved Methods for Hepatitis A Virus and Rotavirus Concentration and Detection in Recreational, Raw Potable and Finished Waters").

Viruses isolated from New York Bight samples have been identified and a report of the findings submitted to Region 2. Further support for their virus surveillance program on ocean dumping in the New York Bight will be provided, and the Region will expand the protocol for processing seawater to include evaluation of virus monitoring methodology.

"Status Report on Single Laboratory Evaluation of Two Methods for Virus Analysis of Soils" is in internal review.

The internal report, "Method for Mammalian Cell Toxicity Test", describing a technique for detecting threshold cytotoxicity in water, has been rewritten to incorporate the data from an internal document, "Status of in Vitro Mammalian Cell Toxicity Test Procedures".

Viral testing has been completed on samples received from phase three of studies by the Water Engineering Research Laboratory - Cincinnati (WERL-Cincinnati) on retrofitted aerobic wastewater sludge digesters at the Trumansburg, New York sewage treatment plant. Virus support provided for studies by WERL-Cincinnati on single and two-phase anaerobic digesters at the USEPA, Test and Evaluation Facility, is continuing.

A report assessing recovery efficiency of beef extract reagents for concentrating viruses from municipal wastewater sludge solids has been submitted to the Office of the Director for clearance.

Nine laboratories have submitted samples for single laboratory viral analyses in conjunction with the American Society for Testing and Materials round-robin testing of two methods for detecting virus in soil. Viral assays on half of these soil samples have been completed. Assay of the remaining samples (organic muck and clay) has been delayed pending resolution of cytotoxicity problems encountered in their original assay.

The laboratory work on the first phase of a sensitivity and precision study of virus plaque confirmation procedures has been completed. The compiled data have been statistically analyzed and the results are being drafted into a report.

Studies have been initiated for testing the applicability of serum substitutes and extenders as alternative reagents for propagation of mammalian cell lines.

STATUS OF LABORATORY OUTPUTS

- B721 "Report on the Optimization of Suspended Cell Culture Technique for Enterovirus Monitoring of Water and Wastewater". Completed 6/24/87
- B722 DUE: 3/87. "Method for Mammalian Cell Toxicity Test". In revision in Section.
- B723 "Report on the Status of Detection Methodology for Hepatitis A Virus and Rotavirus". Completed 5/27/87.
- B724 "Report on Improved Method for Virus Reconcentration by the Organic

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Flocculation Procedure". Completed 6/29/87.

STATUS AND SCHEDULE OF DELIVERABLES:

2037 DUE: 12/30/86 REVISED: COMPLETED: 12/16/86 REPORT ON MORE COST-EFFECTIVE, IN-LINE INJECTION TECHNIQUES TO CONCENTRATIVE VIRUS FROM AMBIENT WATER AND SLUDGE LEACHATES.

CODE TITLE

BUDGET SUB-ACTIVITY: B101 WATER QUALITY

ISSUE: A WQBA/PERMITTING

PPA (L): 02 PROVIDE QA SUPPORT PROGRAM OF WATER QUALITY

PPA (S): QUALITY ASSURANCE

RC: A EMSL CINN

PROJECT: 04 PROVIDE QUALITY ASSURANCE SUPPORT FOR WATER QUALITY

PLANNED START: 09/01/77 PLANNED END: 12/01/99

PROJECT DESCRIPTION:

GOAL: Assure USEPA that ambient water quality monitoring data generated either by or for regulation setting, enforcement, or compliance purposes is scientifically valid and legally defensible.

RATIONALE: This project is to maintain a sound database through use of standardized sampling and analytical methodology, applied in an ongoing Quality Assurance Program for all of USEPA's ambient monitoring programs.

APPROACH: This project consists of those needs identified as necessary to

APPROACH: This project consists of those needs identified as necessary to support a quality assurance program: quality assurance guidelines and manuals, a quality control check sample program for chemicals and biological test organisms, a Repository for Toxic and Hazardous Materials which furnishes calibration standards, and semi-annual performance evaluation studies conducted to evaluate laboratory performance. This national level quality assurance program supports the intralaboratory and interlaboratory quality control programs of USEPA Programs, USEPA contractor, and other federal, state and local laboratories conducting ambient water monitoring in the United States.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

EMSL-Cincinnati produces and distributes chemical and biological quality control (QC) samples and certified calibration standards as quality assurance (QA) support for of the Office of Water programs. Microbiological and biological samples are provided inhouse and extramurally. The National Bureau of Standards (NBS) establishes selected traceability tests for water quality analytes in performance evaluation (PE) samples. Formal PE studies are conducted semi-annually with 500/700 laboratories/study from USEPA, USEPA contractor, state, and local agencies conducting ambient water monitoring. Computer programming and data processing support are obtained through the Agency's ADP

contract with Computer Sciences Corporation. Project activities are on or ahead of schedule.

ACCOMPLISHMENTS AND RESULTS TO DATE:

USEPA Repository for Toxic and Hazardous Materials (Harry Kolde): The Repository distributed 20,300 ampuls to 420 USEPA, USEPA contract, state, and local laboratories in the third quarter.

Conduct Referee Analyses (Harry Kolde): Third quarter analyses were: QC and PE samples, 201 ordered, 93 completed. Surrogate solutions for USEPA Methods 624 and 625, 9 ordered, none completed. Future work: continue verification analyses as needed.

QC Sample Program (Ray Wesselman/Gary Collins): 46,330 chemical QC samples were distributed to USEPA, USEPA contract, state, and local laboratories. The following samples were produced to replenish depleted inventories: 5900 sets of oil and grease, 6200 sets of chlorinated hydrocarbon pesticides, and 5900 sets of polychlorinated biphenyls (PCBs) Aroclor 1260.

A total of 919 biological QC samples were shipped. These were: Algae for Identification 119; simulated plankton, 26; chlorophyll, spectrophotometric and fluorometric, 250; reference toxicants for bioassays-cadmium chloride, 95, and sodium lauryl sulfate, 21. Bacterial samples distributed were: Escherichia coli, 153; Enterobacter aerogenes, 80; Klebsiella pneumoniae, 44; Pseudomonas aeruginosa, 60; and Streptococcus faecalis, 53. Samples 3 and 4, Algae for Identification were prepared. Design of a reference toxicant (copper) was completed. It is to be prepared in the fourth quarter. Design of a sample reference food (Artemia cysts) for use in bioassays required by the National Pollutant Discharge Elimination System (NPDES) was completed and purchase of the cysts will be made in the fourth quarter if funding is available.

Preparation of PE Samples (Harold Clements): New PE samples produced for future studies include trace metals (12,000 ampuls), cyanide (6,000 ampuls), and toal phenolics (3,000 ampuls). Analyses are continuing for all WPO19 analytes to verify stability. Thirty-four ampuls containing 76 analytes are being prepared for Study 19.

Water Pollution (WP) PE Studies (Paul Britton): By April 27, 1987, the reporting deadline for WPO18, data were received from 513 laboratories, 81 percent of the 631 laboratories that received samples. A total of 35,758 analytical results were evaluated and 86.4 percent were within study acceptance limits. Individual reports for each laboratory and relevant study summaries were mailed to USEPA regional and program office personnel as scheduled on June 5 for their further distribution to participants.

WPO19 was announced as scheduled on June 29, 1987. Samples for this study will be shipped on August 10 to laboratories nominated by USEPA regional and program office personnel.

STATUS OF LABORATORY OUTPUTS:

Output 2150(B) Report on Traceability to NBS due 12/86 was completed. Output 2150[C] Annual Report on Referee Analytical Services due 12/86 was completed. Output 2150[D] Design, Develop, and Prepare PE Samples for WP Studies due 12/86 was completed.

STATUS AND SCHEDULE OF DELIVERABLES:

- 2033 DUE: 12/30/86 REVISED: COMPLETED: 12/19/86
 ANNUAL STATUS REPORT ON THE USEPA REPOSTIORY OF TOXIC AND HAZARDOUS MATERIAL FOR AMBIENT MONITORING
- 2034 DUE: 12/30/86 REVISED: COMPLETED: 12/19/86
 ANNUAL REPORT ON THE QUALTIY CONTROL SAMPLE PROGRAM FOR AMBIENT MONITOR-ING.
- 2150 DUE: 09/30/87 REVISED: COMPLETED: 07/27/87 INDIVIDUAL AND SUMMARY REPORTS ON SEMI-ANNUAL WP PERFORMANCE EVALUATION STUDIES 17 AND 18

CODE TITLE

BUDGET SUB-ACTIVITY: B101 WATER QUALITY

ISSUE: A WQBA/PERMITTING

PPA (L): 04 REFERENCE CHEMICAL METHODS WATER AND SEDIMENT ANALYSIS

PPA (S): METHODS\CHEMICAL

RC: A EMSL CINN

PROJECT: 05 REFERENCE CHEMICAL METHODS - WATER AND SEDIMENT ANALYSIS

PROJECT DESCRIPTION:

GOAL: Provide standardized analytical chemical methods for quantification of pollutants or their metabolic products in water, wastewater, biological tissue sediment and sludge.

RATIONALE: USEPA has the task of monitoring ambient water quality either directly or as the result of analyses performed by the regulatory community. USEPA therefore requires comparability of the data generated in diverse processes. Emphasis is to be given to short term event monitoring and frequency of measurement in order to ascertain patterns and assess effects. APPROACH: Collect, evaluate, standardize analytical methods for toxic materials sensitive to toxicologically significant (low) levels of contamination. Publish methods which assist controlling the levels of toxicants to subchronic levels in non-target organisms and enhance beneficial use of waters. This includes measurement of toxic concentrations in water, sediment, tissue and sludge.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

Under this project, a combination of in-house and extramural investigations are conducted by the Inorganic Analyses, Organic Analyses, Advanced Instrumentation, and Sampling and Field Measurements Sections of the Physical and Chemical Methods Branch to develop, evaluate, and standardize methods for measurement of toxic chemicals in water, sludge, sediment, and tissue. The objective is to publish methods to assist in controlling the levels of toxicants and enhance the beneficial uses of water.

IDENTIFICATION AND DETERMINATION OF METAL IONS AND THEIR ORGANOMETALLIC AND METAL CHELATE COMPOUNDS BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)--The manuscript, "Determination of Mercury (II) and Organomercury Compounds by Reversed-Phase Liquid Chromatography with Reductive Electrochemical Detection",

has gone through in-house review and has been submitted to THE ANALYST for consideration for publication. It was also sent to Headquarters as an 1869[A] deliverable in May.

The evaluation of the solid-phase extraction (SPE) of the mercury analytes from aqueous solution is continuing. Otis Evans attended a "Metal Speciation Workshop" in Jekyll Island, Georgia on May 20-22, 1987.

CHEMICAL METHOD FOR EXTRACTION OF INORGANIC POLLUTANTS FROM BIOLOGICAL TISSUE--A Quality Assurance Newsletter article was prepared announcing the development and availability of Method 200.11, "Determination of Metals in Fish Tissue by Inductively Coupled Plasma - Atomic Emission Spectrometry".

DEVELOPMENT OF METHODS FOR NONVOLATILE HAZARDOUS SUBSTANCES NOT AMENABLE TO GAS CHROMATOGRAPHY (GC)/MASS SPECTROMETRY (MS)--Work continued to develop a general purpose liquid chromatography (LC)/mass spectrometry (MS) method for nonvolatile organic compounds in water. With a liquid-liquid extraction from water followed by LC/MS, the grand mean recovery of 35 compounds was 71% with a mean relative standard deviation of 11%. With a solid phase extraction from water followed by LC/MS, the grand mean recovery was 76% with a mean relative standard deviation of 17%. Further work is needed to develop a useful internal standard and to automate the method. More instrumental stability is needed, especially in the gradient controller, and this is available in newer model commercial interfaces. Region 6 provided samples from solvent reclaimer wastes and work began to determine the applicability of analytical methods to these wastes. Similar samples from textile dye/formulators wastes are being sought. Technical and engineering support in the maintenance of instrumentation was provided to several Branches and Sections of the laboratory.

METHODS FOR RESOURCE CONSERVATION AND RECOVERY ACT COMPOUNDS IN WASTEWATER-Studies were designed to determine the optimum extraction conditions for
recovery of Appendix IX compounds from water using continuous extraction. All
of the semivolatile Appendix IX compounds amenable to gas chromatography have
been examined for recovery under various extraction schemes. Preliminary
results of these tests revealed recoveries lower than expected. The problems in
the extraction procedures were identified and the affected tests are being
repeated. Matrix studies employing the optimum extraction conditions are
scheduled to begin in the fourth quarter.

DEVELOPMENT OF DESIGN CRITERIA FOR MECHANICAL CONSTRUCTION OF A FIBER OPTIC CHEMICAL ANALYZER--The refractive indices of 13 strands of fiber optics coated with synthetic organophilic cladding were measured and found to range from 1.3650 to 1.5257. Any value less than 1.485 indicates a usable fiber. Fibers were also made for Battelle which has constructed a sensor system. Unfortunately, one of the fibers broke during installation and the project has fallen behind schedule due to this problem and lack of funds. Additional funds have since been approved so that they can complete the project.

STATUS OF LABORATORY OUTPUTS:

None for this quarter.

STATUS AND SCHEDULE OF DELIVERABLES:

- 2039 DUE: 09/30/87 REVISED: COMPLETED: 09/28/87 STATUS REPORT: ANALYTICAL METHODOLOGY APPLICABLE TO MONITORING RCRA CONSTITUENTS IN POTW EFFLUENTS
- 2040 DUE: 11/30/87 REVISED: COMPLETED: STATUS REPORT ON ANALYTICAL METHODS APPLICABLE TO CHEMICALS FROM SOLVENT RECLAIMER WASTES
- 2041 DUE: 11/30/87 REVISED: COMPLETED: STATUS REPORT ON ANALYTICAL METHODS APPLICABLE TO CHEMICALS FROM TEXTILE DYE/FORMULATORS WASTES
- 2042 DUE: 10/20/87 REVISED: COMPLETED: STATUS REPORT ON DESIGN AND EVALUATION OF FIBER OPTICS TOXIC CHEMICAL ANALYZER
- 1320 DUE: 09/01/86 REVISED: 03/30/87 COMPLETED: 03/30/87 Chemical Methods for Extraction of Inorganic Pollutants from Biological Tissue
- 1869 DUE: 05/30/87 REVISED: COMPLETED: 05/29/87 REPORT ON METHODS FOR DISTINGUISHING FORMS OF METALS

CODE TITLE

BUDGET SUB-ACTIVITY: B101 WATER QUALITY

ISSUE: C WASTE WATER TREATMENT TECHNOLOGY

PPA (L): 42 CORRECT DEFICIENCIES IN APPROVED ANALYTICAL METHODS

PPA (S): CORRECT DEFICIENCIES

RC: A EMSL CINN

PROJECT: 06 CORRECTION OF DEFICIENCIES IN APPROVED ANALYTICAL METHODS

PROJECT OFFICER: J. J. LICHTENBERG PHONE: 513-684-7306

PROJECT DESCRIPTION:

GOAL: To assure that analytical methods presently approved by the Administrator for regulatory measurement of pollutants in industrial wastewater are reliable in all wastewater matrices.

RATIONALE: When analytical methods are developed for measurement of pollutants in industrial wastewaters, they are tested in the effluents of a limited number of industrial categories. However, when approved for regulations, the methods are intended for use in discharges from all required industrial categories. Regulatory use of such methods discloses analytical deficiencies which could not possibly have been recognized in their development. The regulatory credibility of such approved methods is maintained through research which rapidly recognizes the defects and corrects them.

APPROACH: Deficiencies brought to the attention of EMSL-Cincinnati through promulgated channels by NPDES permittees or others, will be addressed to improve the method validity as required by regulation. The Agency will review existing approaches for minimum detectable level (MDL) and develop MDLs for methods which do not have MDL data.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

This project involves a combination of extramural and in-house investigations conducted by the Inorganic Analyses, Organic Analyses, Advanced Instrumentation, and Sampling and Field Measurements Sections of the Physical and Chemical Methods Branch. This work is designed to correct reported deficiencies in approved National Pollutant Discharge Elimination System (NPDES) analytical methods for specific applications. Several projects are for the evaluation and standardization of methods for hazardous and toxic chemicals, including development of method detection limits (MDLs) for those analytes that do not have published MDLs.

FY 87 REPORT ON DEFICIENCIES AND REVISED METHODS--(Item #2047[A] and #2048[A]) Three 304(h) notices are being processed. Two, the Virginia Edison Power Company (VEPCO) agreement and the nationwide approval of the direct current plasma emission method, have undergone Red Border review and have received Office of Management and Budget (OMB) approval. Red Border review comments are being incorporated into these notices and they will be sent to the Federal Register by June 30, 1987, for publication. The third notice is to establish standard test procedures for: Toxicity Tests for Aquatic Organisms, Enteroviruses in Sludge, Mutagens in Wastewater, and Viruses in Water. Work is continuing to determine precision, accuracy, and MDL data for Fluoride Method 340.1, 340.2, and 340.3. The Start Action Request (SAR) has been forwarded, Vaun Newill has signed the SAR, and it is being forwarded to the Steering Committee.

METHODS FOR PESTICIDES IN WASTEWATER--Battelle has completed the review of the National Pesticide Survey (NPS) and Part 455 Methods. The entire consolidated list of pesticides has been received now and experiments with traditional adsorption cleanup procedures (Florisil, alumina, silica gel) have begun to establish elution profiles. For polar pesticides, new column packings such as Extralut and reverse phase packings will be evaluated.

STANDARDIZE Gas Chromatography/Mass Spectrometry (GC/MS) METHODS FOR SLUDGE--This project is designed to be a follow-up to the study in B101/PPA 04, and will not be initiated until the fourth quarter. If delays occur on the Status Report: Analytical Methodology Applicable to Monitoring Resource Conservation and Recovery Act (RCRA) Constituents in Publicly-Owned Treatment Works (POTW) Effluents, deliverable 2039[A], they will also affect this project.

REPORT TO CONGRESS ON 304(h) METHODS--After a six-week delay finding a suitable contract mechanism, the Rocky Mountain Analytical Laboratory Division of Enseco, Incorporated, was tasked to prepare the chemical methods portion and consolidated report for Section 518 of the Clean Water Act. Meetings were held on June 17 and 25 to review the technical details and timing of the report.

FLOW MEASUREMENTS FOR THE UNDERGROUND INJECTION AND CONTROL (UIC) PROGRAM--An Evaluation of the Doppler Flowmeter (Hydra) has begun. The Hydra was selected for evaluation since it is a portable flowmeter that is claimed to be insensitive to particles and air bubbles. Initial studies showed that the apparatus had serious defects and it was returned to the factory for major repairs.

ACCUMULATOR COLUMNS FOR SAMPLING WATERS--Work on this project has been delayed due to reassignment of personnel on a technical assistance project for the Drinking Water Research Program and also to help in planning of an instrument evaluation program.

OPEN CHANNEL FLOW MEASUREMENTS--Preliminary plans have been made to transfer, ship, and reassemble an open channel flow test stand from the National Bureau of Standards (NBS). The test stand will be used to develop a quality assurance program for open channel measurements.

ALTERNATE TEST PROCEDURE (ATP) ACCOMPLISHMENTS--During the third quarter, a total of 18 requests for approval of ATPs were received. The following summarizes the applications reviewed by this laboratory and the actions recommended: Received National Pollutant Discharge Elimination System (NPDES): Nationwide (7); Limited Use (11). Action Taken: Recommended Approval (1); Recommended Disapproval (0); Not ATP (0); Requested Data (0); Pending (17).

STATUS OF LABORATORY OUTPUTS:

____ [B]/EMSL 930 DUE: 2/87 REVISED: 1/87 COMPLETED: 5/18/87 "2nd Quarterly Report-Equivalency Applications Received, Notice and Comments Published in Federal Register, Final Notice Published in Federal Register"

STATUS AND SCHEDULE OF DELIVERABLES:

- 2047 DUE: 11/30/87 REVISED: COMPLETED: ANNUAL REPORT: ITEMIZING DEFICIENCIES IDENTIFIED (FY 87)
- 2048 DUE: 11/30/87 REVISED: COMPLETED:
 ANNUAL REPORT: REVISED ANALYTICAL METHODS WITH DEFICIENCIES REMOVED (FY 87)
- 1854 DUE: 11/30/86 REVISED: COMPLETED: 11/25/86 ANNUAL REPORT ITEMIZING DEFICIENCIES IDENTIFIED FY 86
- 1855 DUE: 11/30/86 REVISED: COMPLETED: 11/25/86
 ANNUAL REPORT REVISED ANALYTICAL METHODS WITH DEFICIENCIES REMOVED FY 86
- 1859 DUE: 11/30/86 REVISED: COMPLETED: 11/25/86
 ANNUAL REPT FED REGISTER NOTICES FOR EQUIVALENCY APPLICATIONS RECD NOTICE AND COMMENTS PUB IN FED REGISTER FINAL NOTICE IN FED REGIS

CODE TITLE

BUDGET SUB-ACTIVITY: B101 WATER QUALITY

ISSUE: C WASTE WATER TREATMENT TECHNOLOGY

PPA (L): 40 QUALITY ASSURANCE AND SAMPLE REPOSITORY

PPA (S): QA AND SAMPLE REPOS.

RC: A EMSL CINN

PROJECT: 07 QUALITY ASSURANCE AND SAMPLE REPOSITORY

PROJECT OFFICER: JOHN WINTER PHONE: 513-684-7325

PLANNED START: 07/01/72 PLANNED END: 12/01/99

PROJECT DESCRIPTION:

GOAL: Assure USEPA that all monitoring data which it requires is sufficiently documented for quality to both support its primary data quality objective and its secondary uses--i.e., that intercomparable data are obtained.

RATIONALE: Data which meet uniform data quality criteria are intercomparable

RATIONALE: Data which meet uniform data quality criteria are intercomparable allowing comparison with similar data generated by different analysts and/or at separate times. Knowledge of confidence levels associated with each data set helps prevent misuse of the data as well as with the decision-making process.

APPROACH: Provide quality control samples and calibration materials from which analyst can establish and document data accuracy. Provide an opportunity for analysts to document their performance semi-annually through the analysis of unknown samples. Evaluate and revise data quality criteria and additional standards as needed. Monitor/evaluate the adequacy of existing procedures for maintaining the Agency's QA support program.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

EMSL-Cincinnati is responsible for producing and distributing quality control (QC) samples and calibration standards and conducting performance evaluation (PE) studies for ambient monitoring and point source discharges to support the quality assurance (QA) program of the Office of Water. The projects which are conducted inhouse and extramurally are on or ahead of schedule.

ACCOMPLISHMENTS AND RESULTS TO DATE:

USEPA Repository for Toxic and Hazardous Materials (Harry Kolde): Distributed 20,400 ampuls to 420 USEPA, USEPA contract, state, and local laboratories.

Three new compounds and five other volatile organic chemicals (VOCs) and synthetic organic chemicals (SOCs) were ampuled.

Conduct Referee Analyses (Harry Kolde): Third quarter analyses were: spiking/calibration solutions, 76 ordered, none completed and QC and PE samples, 201 ordered, 93 completed. Future work: continue verification analyses as needed.

Traceability to the National Bureau of Standards (NBS) (Ed Berg): A priority list of PE and QC samples requiring NBS analyses was agreed upon. Analyses have begun on selenium in fish samples. NBS will produce 3,000 sets of polychlorinated biphenyl (PCB) congeners.

QC Sample Program (Ray Wesselman): In the third quarter, 46,330 QC samples were distributed to USEPA, USEPA contract, state, and local laboratories. The following samples were produced to replenish depleted inventories: 5900 sets of oil and grease, 5900 sets of PCBs and 6200 sets of chlorinated hydrocarbon pesticides.

Water Pollution (WP) PE Studies (Paul Britton): By April 27, 1987, the reporting deadline for WP018, data were received from 513 laboratories, 81 percent of the 613 laboratories that received samples. A total of 35,758 analytical results were evaluated and 86.4 percent were within study acceptance limits. Individual reports for each labortory and relevant study summaries were mailed to USEPA regional and program office personnel as scheduled on June 5 for their further distribution to participants.

WP019 was announced as scheduled on June 29, 1987. Samples for this study will be shipped on August 10 to laboratories nominated by USEPA regional and program office personnel.

Prepare PE Samples (Harold Clements): New PE samples produced for future studies include trace metals (12,000 ampuls), cyanide (6,000 ampuls), and total phenolics (3,000 ampuls). Analyses are continuing for all WP019 analytes to verify stability. Study 19 involves 34 ampuls covering 76 analytes.

Projection of Calibrant Requirement Necessary to Support Domestic Sewage Study, Resource Conservation and Recovery Act (RCRA) Section 3013(A), Section 4C of Consent Decree (John Winter): Contaminated soil, and industrial and municipal sludge QC samples are being prepared. Presentations by Bill Whittington and Al Rubin (Office of Water Regulations and Standards) were attended on June 17. The analytical methods and QA requirements are being sent to EMSL-Cincinnati for consideration of calibrant QC needs.

STATUS OF LABORATORY OUTPUTS:

Output 2044[B] Annual Report on Traceability to NBS due 12/86 was completed. Output 2044[C] Annual Report on Referee Analytical Services due 12/86 was completed. Output 1850[B] Design, Develop, and Prepare PE Samples due 12/86 was completed.

STATUS AND SCHEDULE OF DELIVERABLES:

- 2044 DUE: 09/30/87 REVISED: COMPLETED: 07/27/87 INDIVIDUAL AND SUMMARY REPORT OF SEMI-ANNUAL WP PERFORMANCE EVALUATION STUDIES 17 AND 18
- 2045 DUE: 10/30/87 REVISED: COMPLETED: PROJECTION OF QA/QC REQUIREMENT NECESSARY TO SUPPORT DOMESTIC SEWAGE STUDY, RCRA SECTION (3018(A), SECTION 4C OF CONSENT DECREE
- 1851 DUE: 01/30/87 REVISED: COMPLETED: 12/19/86 ANNUAL REPORT OF QA DISTRIBUTION OF RESPOSITORY STANDARDS AND QC SAMPLES

CODE TITLE

BUDGET SUB-ACTIVITY: B101. WATER QUALITY

WASTE WATER TREATMENT TECHNOLOGY ISSUE:

PPA (L): 41 DISCHARGE MONITORING REPORT (DMR) QUAL. ASSURANCE SUPPORT

PPA (S): DMR OA SUPPORT

RC: Α EMSL CINN

PROJECT: 08 DISCHARGE MONITORING REPORT (DMR) QUALITY ASSURANCE SUPPORT

PHONE: 513-684-7325

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PLANNED START: 10/01/79 PLANNED END: 12/01/99

PROJECT DESCRIPTION:

GOAL: Measure the ability of discharger laboratories to generate good quality DMR data submitted by the 8000 major industrial wastewater dischargers to show compliance with their National Pollutant Discharge Elimination (NPDES) permits. RATIONALE: The NPDES is one of the most extensive monitoring networks operated by the Agency. Within this system, dischargers monitor themselves and are required to immediately report any NPDES permit violations. Enforcement actions initiated by reported self-monitoring data are legally and technically equitable only if the data generated within the NPDES system are of known quality and are intercomparable. The DMR QA program provides a cost-effective mechanism by which this data quality is monitored. APPROACH: Performance evaluation samples are prepared containing basic analytes listed on the NPDES permits. Acceptance criteria are derived from analyses of

samples by 300 USEPA and state enforcement laboratories. Samples are distributed to 8000 discharger laboratories. Results are used to evaluate quality of self-monitored data. Quality control samples are provided on request and other corrective actions are taken to bring poor performers back to acceptable levels.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

Clerical administration and conduct of the Discharge Monitoring Report -Quality Assurance (DMR-QA) studies are conducted by the Bionetics Corporation. A five-person staff prepares, analyzes, and distributes samples; maintains study records; and handles most direct contacts with study participants. Computer programming and data processing support are administered by the QA Branch through the Agency's ADP contract with Computer Sciences Corporation (CSC). These activities are all on or ahead of schedule for the current study. Inhouse activities include overall management of the project and direction of the Bionetics and CSC efforts, statistical programs, computer operations, study design, and policy; with direct responsibility for producing the final laboratory performance evaluation (PE) reports for each study. These activities are on or ahead of schedule for the current study.

ACCOMPLISHMENTS AND RESULTS TO DATE:

Annual DMR-QA Study 6 (Paul Britton): A memorandum was sent to James Elder, Director, Office of Water Enforcement and Permits (OWEP) on November 16, 1986, reporting completion of Study 6.

Annual DMR-QA Study 7 (Paul Britton): By the final deadline for receipt of data effectively May 19, 1987, 6697 responses had been received from NPDES permittees designated as "major dischargers", by USEPA's OWEP. This constitutes an 89.3 percent response of the 7502 permittees that received samples. To facilitate a rapid evaluation of performance by the regions or states in high-priority situations, the true values and PE limits for the study were sent to the designated regional and state DMR-QA Coordinators on May 28, 1987. Because of the volume of data and reports involved, the first copy of reports will be mailed to the regions by mid-July. By mid-August, the regions will receive another copy and the state will receive two copies. Participants will be mailed their copy two weeks after the state copies have been mailed.

STATUS OF LABORATORY OUTPUTS: None.

STATUS AND SCHEDULE OF DELIVERABLES:

- 2046 DUE: 09/30/87 REVISED: 11/30/86 COMPLETED: 11/12/86 ANNUAL REPORT TO OWEP OF DMR-QA PERFORMANCE EVALUATION STUDY (FY 86)
- 2188 DUE: 11/30/87 REVISED: COMPLETED:
 ANNUAL REPORT TO OWEP OF DMR-QA PERFORMANCE EVALUATION STUDY -- FY 87

CODE TITLE

BUDGET SUB-ACTIVITY: C104 DRINKING WATER

ISSUE: E DRINKING WATER TECHNOLOGY

PPA (L): 72 QUALITY CONTROL AND PERFORMANCE EVALUATION SAMPLES DW AND QA

PPA (S): QC PE SAMPLES DW/QA

RC: A EMSL CINN

PROJECT: 09 QUALITY CONTROL AND PERFORMANCE EVALUATION SAMPLES

PROJECT OFFICER: JOHN WINTER PHONE: 008-684-7325

PLANNED START: 09/01/85 PLANNED END: 12/30/99

PROJECT DESCRIPTION:

GOAL: Provide Quality Assurance Support to the Drinking Water Program. RATIONALE: Quality control samples provide an independent check on the analyst's technique and application of the USEPA approved methodology. Calibration materials provide verified source of calibration standards, and performance evaluation samples provide the bases for judging USEPA, state, and local laboratory capabilities to conduct the required analyses under the certification program of the Drinking Water Regulations.

APPROACH: Design and prepare QC and PE samples and standards series inhouse and on contract for all contaminants and monitoring analytes regulated under the SDWA. Verify sample true values, stability and homogeneity through analyses by referee laboratories, and by traceability to NBS. Conduct formal performance evaluation studies and report results to regulating authorities and participant laboratories.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS:

EMSL-Cincinnati is responsible for the production and distribution of quality control (QC) samples, performance evaluation (PE) samples and calibration standards and for conducting PE studies for certification of drinking water (DW) laboratories. PE studies are conducted with inhouse and extramural resources.

ACCOMPLISHMENTS AND RESULTS TO DATE:

QC Samples (Ray Wesselman): A total of 11,650 QC samples were distributed in the third quarter to USEPA, USEPA contract, state, and local laboratories. 6400 sets of trihalomethanes were produced and verified to replenish depleted

stock.

Prepare PE Samples (Harold Clements): PE samples were produced for the volatile organic chemicals (VOCs) in the next DW study (WS021). Ampul 1 contains the eight specific VOCs and Ampul 2 contained some of the regulated and unregulated VOCs (1445 compounds) (3,000 ampuls). Residual free chlorine and pesticide ampuls were also produced for upcoming WS021 (6,000 ampuls). Day zero verification confirmed the sample designs and concentrations.

USEPA Repository for Toxic and Hazardous Materials (Harry Kolde): 20,400 ampuls were distributed to 420 USEPA, USEPA contract, state, and local laboratories. The VOCs to be regulated are now available as standards. Three new DW compounds were ampuled as analytical standards and five other VOCs and synthetic organic chemicals (SOCs) were re-ampuled to replenish low stocks.

Traceability to the National Bureau of Standards (NBS) (Ed Berg): A priority list of PE and QC samples requiring NBS analyses was agreed upon. Analyses have begun on Water Supply (WS) VOCs, WS herbicides, WS trihalomethanes, and WS chlorinated hydrocarbon pesticides.

Conduct Referee Analyses (Harry Kolde): Third quarter verification analyses were as follows: (1) spiking/calibration solutions of DW compounds, 76 ordered, none completed; (2) QC and PE samples, 1005 ordered, 242 completed; and (3) neat surrogates for USEPA Methods 624 and 625, 9 ordered, none completed. Future work - continue verification analyses, as needed.

STATUS OF LABORATORY OUTPUTS:

Output 1653[B] Annual Report on Referee Analytical Services due 12/86 was completed.

STATUS AND SCHEDULE OF DELIVERABLES:

1653 DUE: 12/30/86 REVISED: COMPLETED: 12/19/86
ANNUAL REPORT ON PRODUCTION AND DISTRIBUTION OF QC/PE SAMPLES
AND CALIBRATION STANDARDS

1654 DUE: 12/30/86 REVISED: COMPLETED: 12/22/86 REPORT ON TRACEABILITY TO NBS

CODE TITLE

BUDGET SUB-ACTIVITY: C104 DRINKING WATER

ISSUE: E DRINKING WATER TECHNOLOGY

PPA (L): 73 METHODS VALIDATION- DRINKING WATER QUALITY ASSURANCE

PPA (S): METHOD VALID. DW/QA

RC: A EMSL CINN

PROJECT: 10 METHODS VALIDATION - DRINKING WATER QUALITY ASSURANCE

PROJECT OFFICER: J. J. LICHTENBERG PHONE: 513-684-7306

PLANNED START: 09/01/82 PLANNED END: 12/01/99

PROJECT DESCRIPTION:

GOAL: The mandate for laboratory certification is expressed in the SDWA. The objective of this program is to develop less expensive methods for which maximum contaminant levels have been set. The methods should contain accuracy standards, and be able to produce reliable data which can be used to support regulations promulgated by the Agency.

RATIONALE: Conduct methods validation studies for trihalomethanes, purgeables and total organic halogens. Produce quality control check samples to support the methods validation program. Conduct GC/MS analysis for priority pollutants. Improve analytical methods for organic, inorganic, and microbiological analyses for drinking water. Improve sampling and holding procedures. APPROACH: Conduct analyses of raw and finished drinking water using the validated methods. Evaluate alternate test procedures to meet requirements of the SDWA. Technical reviews, external and/or internal reviews will be conducted and coordinated to provide a technically sound recommendation on approval or denial of the proposed alternate test procedures.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

This project is assigned in the Organic Analyses Section for the purpose of developing and evaluating methods for synthetic organic chemicals (SOCs) and volatile organic chemicals (VOCs). The Environmental Monitoring and Support Laboratory - Cincinnati (EMSL-Cincinnati) is cooperating with the Technical Support Division (TSD) to evaluate pesticide methods proposed for use in the future groundwater survey. This work is carried out both in-house and under contract.

METHODS FOR VOCS--Preservation of volatile organics in chlorinated tap water can be affected by dechlorination with ascorbic acid or thiosulfate followed by acidification to pH 2 with hydrochloric acid. Ascorbic acid is preferred for

gas chromatography/mass spectrometry (GC/MS) methods, as sulfur dioxide can be formed by acidification of thiosulfate and interfere with determination of some early eluting peaks. No similar interference is found in the non-MS methods. Draft journal articles on the two capillary methods were completed. The GC/MS paper is being revised and will be completed early next quarter; the photoionization detection (PID)/Hall paper is being sent out for peer review. Final editing is being completed on the drinking water methods prior to typesetting by the Center for Environmental Information (CERI).

METHODS FOR SOCs--(Item #2060[A]) Under the Safe Drinking Water Act (SDWA) Amendments of 1986, regulations call for some specific SOCs for which suitable analytical methods are not available. These include endothall and diquat. The present method of choice for diquat (and paraquat) is a high-performance liquid chromatographic (HPLC) technique with ultraviolet (UV) photometric detection and confirmation with a photodiode array detector. Liaison has been established with personnel in the State of South Carolina and other locations where similar approaches are being employed. A trip has been scheduled next quarter to South Carolina to observe a real application and the subsequent sampling and analyses. The project was initiated on derivatization of endothall with pentafluorobenzyl bromide for subsequent gas chromatographic analysis. Because of the hazardous nature of this reactant, present plans are to examine alternative derivatization techniques for HPLC.

METHODS FOR DISINFECTION BY-PRODUCTS--An interim procedure was previously provided for the haloacetic acids, chloral hydrate and 1,1,1-trichloropropanone. Application of the approach to real world samples has shown that this technique needs more refinement before it can be considered for routine application. A micro-extraction procedure with methyl-t-butyl ether has been examined for the dihaloacetonitriles, chloropicrin, chloral hydrate, and 1,1,1-trichloropropanone. The preliminary results look promising in terms of recoveries and detection limits. These two methods cover most of the higher priority disinfection by-products.

NATIONAL PESTICIDE SURVEY (NPS)-- The EMSL-Cincinnati is participating in the NPS as a referee for Battelle on the analysis of phenoxyacid herbicides in groundwater. Battelle's Method 3 is a modified version of the original EMSL-Cincinnati procedure. Calibration standards were exchanged with Battelle to ensure that all field mesurements are related to a common basis.

A total of 8 field samples have been analyzed, 5 of which were also spiked samples. The data were reviewed during the June 8-10 NPS meeting with representatives from Battelle, Bay St. Louis, EMSL-Cincinnati, and TSD. As a result of the meeting, a number of changes were recommended in the application of Method 3, the major change being the accurate measurement of pH before extractions. Future samples will be analyzed with additional quality control including laboratory control standards, quantitation on both the primary and confirmation column, and perhaps the use of weighed standards for some of the analytes.

TECHNICAL ASSISTANCE TO DRINKING WATER RESEARCH PROGRAM--Modification of automatic capping mechanism in an automatic sampler to be used for sampling volatile organics from a force main of drinking water distribution system. An

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American Sigma sampler has been modified with an EMSL-Cincinnati patented VOC sampling device and the unit is now undergoing laboratory evaluation. Preliminary indications are that the unit is working satisfactorily in sampling VOCs automatically.

STATUS OF LABORATORY OUTPUTS:

None for this quarter.

STATUS AND SCHEDULE OF DELIVERABLES:

2060 DUE: 09/30/87 REVISED: COMPLETED: 09/28/87 METHODS FOR SELECTED SYNTHETIC ORGANIC COMPOUNDS - SUMMARY REPORT

CLIENT OFFICE 054

FOURTH QUARTER STATUS REPORT ON FY'87 PROJECT DESCRIPTIONS
Office of Acid Deposition, Environmental Monitoring and Quality Assurance
EMSL CINN: JULY - SEPTEMBER 1987

BUDGET SUB-ACTIVITY: C104 DRINKING WATER

ISSUE: E DRINKING WATER TECHNOLOGY

PPA (L): 73 METHODS VALIDATION- DRINKING WATER QUALITY ASSURANCE

PPA (S): METHOD VALID. DW/QA

RC: A EMSL CINN

PROJECT: 11 METHODS VALIDATION --DRINKING WATER QUALITY ASSURANCE

PLANNED START: 10/01/86 PLANNED END: 12/01/99

PROJECT DESCRIPTION:

GOAL: The Equivalency Program encourages analytical methods development and/ or improvement by the regulated community and by manufacturers of pollution measuring instruments. These alternate test procedures (ATPs) may be approved for case-by-case or nationwide use.

RATIONALE: 40 CFR Part 141.27 requires that the Agency consider and approve use of appropriate ATPs for the NIPDWR.

APPROACH: After receipt of unsolicited ATP applications, necessary internal and/or external technical reviews are conducted and coordinated to provide a technically sound recommendation of approval or denial of the proposed ATP. Items considered during review include, but are not limited to: availability of a well documented test procedure; documentation of the applicability of the proposed procedure; performance characteristics such as accuracy, precision, and method detection limits; equivalency data to an approved test procedure.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

During the fourth quarter of FY87, six requests for approval of alternate test procedures (ATPs) were received and processed. Two of the requests were inquiries by LaMotte Chemical Products, Incorporated, concerning nationwide use of test kits for free and total chlorine. Recommendations are pending for both requests. The remaining four requests were for limited approvals. The City of Chicago requested and received a recommendation for approval of a modified extraction procedure for total trihalomethanes (TTHMs). The State of Connecticut has requested approval to use high performance liquid chromatography (HPLC) for determination of herbicides. The request is under review. The U. S. Environmental Protection Agency (USEPA), Region 6, has requested comments on a two-step titrimetric test procedure for total chlorine and residual oxidants. Additional information is being forwarded so that the review may be completed. CSMRI Analytica has requested limited approval to

proceed with the use of inductively coupled argon plasma (ICAP) spectrometry, although final nationwide approval is expected soon. A recommendation for approval has been made.

STATUS OF LABORATORY OUTPUTS: None.

STATUS AND SCHEDULE OF DELIVERABLES:

- 2057 DUE: 12/30/87 REVISED: COMPLETED: RECOMMENDATIONS TO REGIONAL OFFICES FOR CASE-BY-CASE APPLICATIONS -ANNUAL REPORT (FY 87)
- 2058 DUE: 12/30/87 REVISED: COMPLETED: RECOMMENDATIONS TO THE DIRECTOR, OFFICE OF DRINKING WATER FOR NATIONWIDE APPLICATION ANNUAL REPORT FY 87
- 1656 DUE: 12/30/86 REVISED: COMPLETED: 12/01/86 Recommendations to Regional offices for case-by-case applications Annual Report (FY 86)
- 1657 DUE: 12/30/86 REVISED: COMPLETED: 12/01/86 Recommendations to the Director, Office of Drinking Water FOR NATIONWIDE APPLICATION--ANNUAL REPORT (FY 86)

CODE TITLE

BUDGET SUB-ACTIVITY: C104 DRINKING WATER

ISSUE: E DRINKING WATER TECHNOLOGY

PPA (L): 73 METHODS VALIDATION- DRINKING WATER QUALITY ASSURANCE

PPA (S): METHOD VALID. DW/QA

RC: A EMSL CINN

PROJECT: 12 METHODS VALIDATION - DRINKING WATER/QUALITY ASSURANCE

PROJECT OFFICER: JOHN WINTER PHONE: 513-684-7325

PLANNED START: 09/01/82 PLANNED END: 12/01/99

PROJECT DESCRIPTION:

GOAL: Provide Quality Assurance Support to the Drinking Water Program. RATIONALE: Precision and accuracy statements are needed for all analytical methodology used to monitor MCLs and RMCLs in drinking water. Such statistics shall be used for laboratory quality control charts, acceptance limits for performance evaluations, and confidence limits for quality control samples. APPROACH: A prime contractor is selected to obtain 10-15 participating laboratories. The prime contractor prepares ampul concentrates which are spiked into reagent and drinking water and subsequently analyzed by the participating laboratories using the specified USEPA drinking water methodology. Youden's non-replicate design of three sample pairs spread over the linear response range is used in the method study. Data are processed using EMSL-Cincinnati's specially designed computer programs (IMVS).

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

EMSL-Cincinnati conducts method validation studies (MVSs) inhouse and extramurally for drinking water (DW) analytes. The prime contractor, Bionetics, Inc., was assigned the task order for preparation of spiking solutions, water samples, instructional materials, verification analyses, formal conduct of the study, and screening of the study data from ten participating laboratories. EMSL-Cincinnati obtains the ten participating laboratories through the competitive procurement process based on technical qualifications, performance evaluation, and costs. EMSL-Cincinnati evaluates the results, conducts computerized data treatment, and prepares the final report.

ACCOMPLISHMENTS AND RESULTS TO DATE:

Status Report - MVSs (Chemical and Microbiological) (Ray Wesselman): At the request of the Office of Drinking Water (ODW), preliminary collaborative studies were conducted to determine the interlaboratory method detection limits (IMDLs) for analytes in Method 504 (1,2-Dibromoethane (EDB) and Dibromochloropropane (DBCP)), Method 505 (Pesticides), Method 515 (Herbicides) and Method 531 (Aldicarbs). The IMDLs were provided to ODW for use in drafting Phase 2, synthetic organic chemicals (SOC) regulations.

Formal Interlaboratory MVSs are being conducted on DW Methods: 502.2 (Volatile Halogenated and Aromatics using the photoionization detector and the Hall detector in series) 504 (EDB and DBCP) Organics - gas chromatograph/electron capture (GC/EC) microextraction), and 505 (Organohalide Pesticides with microextraction), 515 (Herbicides - GC derivatization), 524.2 (Volatiles by GC/mass spectrometer (MS) and 531 (N-methyl carbamayloxines and N-methyl carbamates by post column derivatization high performance liquid chromatography (HPLC)). Award of contract was made in June, 1987 to ten laboratories.

STATUS OF LABORATORY OUTPUTS: None

STATUS AND SCHEDULE OF DELIVERABLES:

DUE: 12/30/86 REVISED: COMPLETED: 12/19/86
STATUS REPORT; INTERLAB METHOD VALIDATION STUDIES (CHEMICAL AND MICROBIOLOGICAL) FOR DRINKING WATER METHODS

CODE TITLE

BUDGET SUB-ACTIVITY: C104 DRINKING WATER

ISSUE: E DRINKING WATER TECHNOLOGY

PPA (L): 74 INTERLABORATORY PERFORMANCE EVAL. - DRINKING WATER QA

PPA (S): INTERLAB. PE DW/QA

RC: A EMSL CINN

PROJECT: 13 INTERLABORATORY PERFORMANCE EVALUATION - DRINKING WATER QA

PROJECT OFFICER: PAUL BRITTON PHONE: 513-684-7325

PROJECT DESCRIPTION:

GOAL: Monitor the ability of the USEPA regional, primary state and intrastate laboratories to perform chemical and microbiological analyses for contaminants regulated under the NIPDWR.

RATIONALE: The regions and states can only effectively operate the Certification Program under the SDWA if they themselves have the knowledge and ability to perform all of the analyses required for certification.

APPROACH: Design, prepare and verify by analyses samples for performance evaluation studies, analyses of trace metals, nitrate/fluoride, chlorinated pesticides, herbicides, turbidity, residual chlorine, sodium and corrosivity. Conduct annual interlaboratory performance evaluation studies for principal state (50 states) and USEPA regions required to conduct chemical and microbiological analyses of public drinking water supplies. Evaluate regional laboratories for capabilities to provide quality data in support of the NIPDWR monitoring program. Compare the analytical results from participating laboratories against acceptance limits generated from previous PE studies.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS:

EMSL-Cincinnati is responsible for providing support for the Drinking Water Certification Program. This support consists of preparing and verifying performance evaluation (PE) samples and conducting PE studies with 1100+ water supply (WS) laboratories across the United States twice annually (WS and WS Microbiology studies). The returned data are evaluated and individual laboratory reports of performance computed and returned to interested parties. The studies include all maximum contaminant level (MCL), monitoring and control analytes. Bionetics, Inc., was selected as the contractor to supply microbiological PE samples for full-volume and/or lyopholized cultures on

July 1, 1985.

Computer programming and data processing support are obtained through the Agency's ADP contract with Computer Sciences Corporation. All project activities are on or ahead of schedule.

ACCOMPLISHMENTS AND RESULTS TO DATE:

Microbiological PE Study (Gary Collins): On April 13, 1987, as scheduled, samples for WSM10 were mailed to 155 USEPA and state laboratories. Results were obtained from 137 laboratories by the deadline, an 88 percent response rate. Reports will be provided to the USEPA regional Quality Assurance Coordinators/Officers by July 27, 1987. All activities are on schedule.

Conduct PE Studies for Drinking Water Laboratories (Paul Britton): Samples for WS020 and WSM10 were shipped to 1248 laboratories on April 6, 1987, as scheduled. For the first time, samples were available for certification for volatile organic chemicals (VOCs). Data were returned by 1041 laboratories before the deadline on June 8 for a good 83.4 percent response. Three copies of the individual laboratory PE reports will be returned by July 27 to the involved regional Quality Assurance Coordinators and contract project officers for their distribution and use to support certification decisions.

STATUS OF LABORATORY OUTPUTS: None.

STATUS AND SCHEDULE OF DELIVERABLES:

2189 DUE: 09/30/87 REVISED: COMPLETED: 08/14/87
PERFORMANCE EVALUATION STUDY REPORT FOR WATER SUPPLY STUDIES WSM09,
WSM 10, WSO19 AND WSO20 FOR FY 87

CLIENT OFFICE 054

FOURTH QUARTER STATUS REPORT ON FY'87 PROJECT DESCRIPTIONS
Office of Acid Deposition, Environmental Monitoring and Quality Assurance
EMSL CINN: JULY - SEPTEMBER 1987

CODE TITLE

BUDGET SUB-ACTIVITY: C104 DRINKING WATER

ISSUE: E DRINKING WATER TECHNOLOGY

PPA (L): 75 LABORATORY CERTIF. PROGRAM FOR DRINKING WATER QA

PPA (S): LAB. CERT. DW/QA

RC: A EMSL CINN

PROJECT: 14 LABORATORY CERTIFICATION PROGRAM FOR DRINKING WATER

PROJECT OFFICER: J. J. LICHTENBERG PHONE: 513-684-7306

PROJECT DESCRIPTION:

GOAL: Certification of laboratories to do drinking water analyses. Section 141.28 of the NIPDWR states that for compliance purposes, samples may be considered only if they have been analyzed by an approved laboratory. RATIONALE: The "Manual of Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies" EPA 600/8-78-008 states that the Environmental Monitoring and Support Laboratory - Cincinnati is responsible for evalutation of the Regional Laboratories by conducting on-site visits at each laboratory once annually and conducting performance evaluation studies annually.

APRROACH: Evaluate regional laboratories and personnel for capabilities to provide quality data in support of the NIPDWR's monitoring program. Approve regional laboratories to perform analyses on the regulated contaminants covered under NIPDWR. Certify regional staff. Review regional certification program and laboratory performance records. Evaluate ten regions annually.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

The Environmental Monitoring and Support Laboratory - Cincinnati (EMSL-Cincinnati) evaluates and certifies regional laboratories and personnel for capability to provide analytical data in support of drinking water regulations. Extramural activity for this project involves preparation of performance evaluation samples and support for the microbiology laboratory certification training course. The majority of activity under this project is in-house. The effort includes: development and presentation of drinking water certification training courses, including course manual, for chemistry and microbiology; on-site visits to the regional laboratories for the purpose of certifying them to do the analyses required under the National Interim Primary Drinking Water Regulations (NIPDWR); and quality assurance support.

LABORATORY CERTIFICATION TRAINING COURSES--The chemistry course was held twice during the weeks of June 15 and 22. One session was held for microbiology during the week of June 8. These provided regional, state, and some other agency chemists and biologists with the opportunity of becoming certified to audit state and local laboratories for drinking water analyses.

REGIONAL LABORATORY ON-SITE VISITS--(Items #2062[A] and 2063[A]). Plans are being made to begin the next round of regional on-site evaluations during the second half of FY 87. EMSL-Cincinnati staff are continuing to work with the Office of Drinking Water (ODW) in revising the Drinking Water Certification Manual. A workshop was conducted on April 1 and 2. Approximately 110 state, local, U. S. Environmental Protection Agency (USEPA), and other government laboratory representatives attended. Several EMSL-Cincinnati staff members assisted in presenting the workshop, preparing reports, and again revising the certification based on input during the workshop.

STATUS OF LABORATORY OUTPUTS; None.

- 2062 DUE: 12/30/87 REVISED: COMPLETED: CONDUCT LABORATORY CERTIFICATION FOR 10 REGIONS SUMMARY REPORT FY 87
- 2063 DUE: 12/30/87 REVISED: COMPLETED: LABORATORY CERTIFICATION FOR FY 1987 -- ANNUAL REPORT
- 1663 DUE: 12/30/86 REVISED: COMPLETED: 12/22/86 Conduct Laboratory Certification for 10 Regions Summary Report (FY 86)
- 1664 DUE: 12/30/86 REVISED: COMPLETED: 12/22/86 Laboratory Certification for FY 1986 Annual Report

CODE TITLE

BUDGET SUB-ACTIVITY: C104 DRINKING WATER

ISSUE: E DRINKING WATER TECHNOLOGY

PPA (L): 70 RADIOCHEMICAL PROC. - DRINKING WATER - QUAL. ASSURANCE

PPA (S): RAD. CHEM. DW/QA

RĆ: A EMSL CINN

PROJECT: 15 RADIOCHEMICAL PROCEDURES - DRINKING WATER-QUALITY ASSURANCE

PROJECT OFFICER: J. J. LICHTENBERG PHONE: 513-684-7306

PROJECT DESCRIPTION:

GOAL: The ODW is conducting an occurrence survey for radionuclides and inorganic chemicals. This survey is called the National Inorganic Radionuclide Survey (NIRS).

RATIONALE: Analyses will be conducted for the six radiochemical analytes and 33 inorganic elements.

APPROACH: These analyses will be conducted at a rate of 600 samples per year.

Referees will analyze radionuclides in drinking water.

The analyses will be conducted under highly controlled quality assurance conditions.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

The Environmental Monitoring and Support Laboratory - Cincinnati (EMSL-Cincinnati) is participating in the National Inorganics and Radionuclide Survey. Drinking water samples are delivered by the Technical Support Division (TSD) to EMSL-Cincinnati for analysis of 33 inorganic and 6 radionuclide analytes. All efforts under the project are being carried out in-house. Activities include: working closely with TSD in project planning and selection of analytical methods for the survey, performing appropriate sample analyses; reviewing and analysis of data; and report writing. The main part of the survey has been completed. Special samples for Ra 228 are now being analyzed.

NATIONAL INORGANICS AND RADIONUCLIDES SURVEY--EMSL-Cincinnati continues to provide analytical technical assistance to other U. S. Environmental Protection Agency (USEPA) laboratories and state and local laboratories. During the quarter, 6 samples were analyzed for gross alpha and beta, 64 for uranium, 6 for Ra 226, and 95 for radon.

The analyses of samples for Ra-228 analysis has started. The data from this

study are to be used for the Ra-228 Occurrence Model. The status of these analyses is noted in the following table.

Analysis	Samples Received	Samples Started	Samples Completed	Completed- This Quarter	Quality Control Samples This Quarter
Ra-228	255	255	224	102	14

STATUS OF LABORATORY OUTPUTS: None.

STATUS AND SCHEDULE OF DELIVERABLES:

COMPLETED: 09/14/87

2051 DUE: 09/30/87 REVISED: COMPLETED: 09/14/87 Radio-chemical analytical data - Report of occurrance study

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CODE TITLE

BUDGET SUB-ACTIVITY: C104 - DRINKING WATER

ISSUE: E DRINKING WATER TECHNOLOGY

PPA (L): 71 COLIFORM ANALYSIS-DRINKING WATER-OUALITY ASSURANCE

PPA (S): COLIFORM ANAL. DW/QA

RC: A EMSL CINN

PROJECT: 16 COLIFORM ANALYSIS - DRINKING WATER - QUALITY ASSURANCE

PROJECT OFFICER: CORNELIUS I. WEBER PHONE: 513-684-7337

PROJECT DESCRIPTION:

GOAL: Provide improved methods for the microbiological analysis of drinking water, correct method deficiencies, evaluate sample holding conditions, and develop quality assurance procedures.

RATIONALE: This project addresses Agency needs for scientifically valid and legally defensible microbiological data on water quality in support of the Safe Drinking Water Act.

APPROACH: Develop or modify, evaluate, select, and standardize methods for the detection and enumeration of indicator organisms and pathogens in drinking water. Alternate methods for total coliforms will be evaluated against standard methods. Appropriate methodologies will be recommended for use in revised drinking water regulations. As a result of inhouse and extramural research, recommendations will be made on holding limitations for drinking water samples to assure reliable analytical results. Rapid methods, such as gene probes and monoclonal and polyclonal antibodies, will be developed for the detection and verification of pollution indicators and waterborne pathogens.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

There are no FY-87 extramural funds for this project. However, two tasks from previous years are still active: one with McNeese State University to determine sample holding effects on microbial populations, and one with the University of Rhode Island, to develop an improved membrane filter (MF) medium for total coliforms (TCs). The draft final reports from both projects were received and are being peer reviewed.

The report, "Comparison of the Hydrophobic-Grid Membrane Filter Procedure and Standard Methods for Coliform Analysis of Water", was published in the May, 1987 issue of the Journal of Applied and Environmental Microbiology. The study

found that total and fecal coliform recoveries by the hydrophobic-grid membrane filter (HGMF) and standard MF methods did not differ significantly over all sample types, and the HGMF method offers no advantage for the analysis of drinking water.

A report on the results of a preliminary multilaboratory evaluation of new alternative media for the TC MF test was completed. The results indicated that the proposed alternative media and the standard M-Endo medium provided similar recoveries. Improved formulations and a lyophilized natural sample will be used in a formal collaborative testing procedure (2054A).

The Section staff directed a course, "Drinking Water Laboratory Certification for Microbiology", June 8-15, 1987, attended by twenty regional and state laboratory certification officers. Several Agency microbiologists participated in lectures and panel discussions.

The Microbiology Section sponsored a seminar at the Andrew W. Breidenbach Environmental Research Center, May 26, 1987, on "The Importance of Membrane Filter Manufacturing and Characterization of their Use" by Ricardo Alfonso, Sartorius Filters, Inc.

STATUS OF LABORATORY OUTPUTS

The extramural report on an improved MF medium for coliforms in drinking water from the University of Rhode Island is being peer reviewed.

A revision of the final report on the research project, "The Effects of Storage on the Bacteriologic Quality of Potable Water Samples", from MCNeese State University has been peer reviewed and is being revised again.

B812 DUE: 03/87 "Improved Membrane Filter Medium for Coliforms in Drinking Water". Report distributed for peer review.

STATUS AND SCHEDULE OF DELIVERABLES:

2054 DUE: 06/30/87 REVISED: COMPLETED: 06/30/87 REPORT ON MULTI-LABORTORY EVALUATION OF NEW ALTERNATIVE MEDIA FOR TOTAL COLIFORM MF TEST

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CODE TITLE

BUDGET SUB-ACTIVITY: D109 HAZARDOUS WASTE

ISSUE: Q QUALITY ASSURANCE

PPA (L): 01 QA TO SUPPORT WASTE CHARACTERIZATION

PPA (S): QUALITY ASSURANCE

RC: A EMSL CINN

PROJECT: 17 QA TO SUPPORT WASTE CHARACTERIZATION IN SW 846

PROJECT OFFICER: JOHN WINTER PHONE: 513-684-7325

PLANNED START: 09/01/84 PLANNED END: 09/01/88

PROJECT DESCRIPTION:

GOAL: Section 3001 of the RCRA requires the Agency to determine the chemical composition of wastes, detect trace levels of toxic constituents and to be able to rapidly screen for hazardous characteristics of spilled materials. The lack of standardized and validated methods emphasizes the immediate need for a comprehensive program to assure that data of known quality are being collected.

RATIONALE: With the transfer of permitting and enforcement from USEPA to the states, standardized methods and QA support are needed to assist the states performing the required measurement and monitoring functions and in obtaining legally defensible data.

APPROACH: Develop, evaluate and distribute analytical standards and reference materials for Appendix VIII compounds. Perform validation studies on analytical methods. Develop and distribute quality control and performance evaluation samples for Appendix VIII compounds Provide quality assurance support to Regions.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

EMSL-Cincinnati produces and distributes quality control (QC) samples and calibration standards and conducts performance evaluation (PE) studies to support the Office of Solid Waste (OSW) monitoring program. A contract was awarded to Bionetics, Inc., in July 1985, to produce and distribute QC samples for analytes of interest to OSW and other programs of the Agency. Sample concentrates and full-volume PE samples are produced and distributed to evaluate contract laboratories performing analyses and for the Ground Water Monitoring Survey being conducted by the Office of Solid Waste and Emergency Response (OSWER). Approximately 58 sites will be sampled in FY86 and FY87.

Northrop Services, Inc., produces and distributes organic calibration standards for the Resource Conservation and Recovery Act (RCRA) and other Agency programs. Contracts were awarded for four organic and four inorganic referee laboratories to verify the true concentration of analytes in QC and PE samples and calibration standards produced inhouse and by contractors. The National Bureau of Standards (NBS) is in the fourth year of an interagency agreement (IAG) to establish traceability for RCRA - Appendix VIII analytes in PE and QC samples.

ACCOMPLISHMENTS AND RESULTS TO DATE:

USEPA Repository for Toxic and Hazardous Materials (Harry Kolde): Eleven new RCRA chemicals were ampuled and four others re-ampuled to replenish stocks. The Repository distributed 20,400 standard solutions to 420 USEPA, USEPA contract, state, and local laboratories. Sixteen RCRA chemicals were sent to referee laboratories for purity assay.

QC Samples (Ray Wesselman): A total of 46,330 QC samples were sent to USEPA, USEPA contractor, regional, state, and local laboratories. Approximately 24,000 sets of various polychlorinated biphenyls (PCBs), PCBs in oils, and pesticides were produced and verified.

Conduct Referee Analyses (Harry Kolde): (1) spiking/calibration solutions for OSW compounds, 199 ordered, none completed; (2) QC and PE samples, 861 ordered, 249 completed; and (3) surrogates for USEPA Methods 624 and 625, 9 ordered, none completed.

Traceability to NBS (Ed Berg): A priority list of PE and QC samples requiring NBS analyses was agreed upon. Analyses have begun on selenium in fish samples. NBS will produce 3,000 sets of polychlorinated biphenyls (PCBs) congeners for USEPA.

Preparation of PE Samples for RCRA Contract Laboratories (Harold Clements): Solid Waste Interlaboratory Comparison Study X was initiated April, 1987. Organic and inorganic samples were sent to 54 participating laboratories on April 16, 1987. Reference values and statistical estimates were generated by the QA Branch and its referee laboratories and supplied to Office of Solid Waste (OSW) for use in final evaluation of results.

Ground Water Monitoring Task Force (GWMTF) (Ray Wesselman): No activity to report this quarter.

SW Guidance Manual on Organization and Management of Laboratories QA Program (John Winter): Target dates have been set. A text outline was presented to OSW and approved. Additional source materials were being reviewed for appropriateness. A first draft is in preparation with deadline for August.

STATUS OF LABORATORY OUTPUTS:

Output 1688[B] Annual Report on Traceability to NBS due 12/86 was completed. Output 1688[C] Annual Report on PE Studies for RCRA Contract Laboratories due 12/86 was completed. Output 1688[D] Annual Report on Referee Analytical

Services due 12/86 was completed. Output 1688[E] Status Report on PE Studies for Ground Water Monitoring Program due 12/86 was completed.

- 2152 DUE: 09/30/87 REVISED: COMPLETED: 09/28/87 GUIDANCE MANUAL ON ORGANIZATION AND MANAGEMENT OF LAB QA PROGRAM
- 1986 DUE: 12/31/86 REVISED: COMPLETED: 12/19/86
 ANNUAL REPORT ON CALIBRATION STANDARDS FROM THE USEPA REPOSITORY OF TOXIC AND HAZARDOUS MATERIALS (APPENDIX VIII COMPOUNDS)
- 1987 DUE: 12/31/86 REVISED: COMPLETED: 12/19/86
 ANNUAL REPORT ON QUALITY CONTROL AND SAMPLE PROGRAM FOR RCRA (APPENDIX VIII) COMPOUNDS

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CODE TITLE

BUDGET SUB-ACTIVITY: D109 HAZARDOUS WASTE

ISSUE: I WASTE IDENTIFICATION

PPA (L): 12 VALIDATE METHODS FOR WASTE CHARACTERIZATION IN SW 846

PPA (S): EVAL/VALIDATE METHOS

RC: A EMSL CINN

PROJECT: 18 VALIDATE METHODS FOR WASTE CHARACTERIZATION IN SW 846

PROJECT OFFICER: ED BERG PHONE: 513-684-7325

PLANNED START: 10/01/82 PLANNED END: 10/01/87

PROJECT DESCRIPTION:

GOAL: Evaluate/validate protocols and procedures for solid waste characterization to assure legally defensible and scientifically accurate data.

RATIONALE: Provide precision and accuracy statements for OSW analytical methodology.

APPROACH: EMSL-Cincinnati will obtain 10-12 laboratories to participate in a collaborative study, will prepare samples for spiking into selected matrices, process analytical data, and will develop precision and accuracy statements for metals in water and sludge and GC\MS Methods 8240 and 8270.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

This is an extramural project to conduct interlaboratory method validation studies (IMVSs) for SW-846 analytical methodology. Request for proposals (RFPs) are to be developed to conduct studies for SW-846 Methods 3005, 3006 (Trace Metals in Ground Water) 3010 (Trace Metals in Ambient Water), 3050 (Trace Metals in Sludges), 8270 (Base/Neutrals), and 8240 (Purgeables). The studies use Bionetics, Inc., as a prime contractor to prepare samples and instructions and conduct the study for ten participating laboratories selected by EMSL-Cincinnati. The resulting data from the participants will be processed by the Quality Assurance (QA) Branch, EMSL-Cincinnati, using the customized IMVS computer programs which provide standard data treatments to reject outliers and generate regression equations for precision and bias statements.

ACCOMPLISHMENTS AND RESULTS TO DATE:

IMVS for SW-846, Methods 8240/5030 Purge and Trap (100 Compounds) (Ray

Wesselman): Contracts were awarded to ten study participant laboratories in late June, 1987. The analytes will include all Appendix IX compounds which are analyzable with the method.

IMVS for SW-846, Methods 8270/3510 (59 Compounds) (Ray Wesselman): Data from all ten laboratories was received in late June, 1987. Statistical review of data to occur in July with rough draft of report in August.

IMVS for SW-846, Methods 3005, 3050, 3010; for Metals in Ambient and Ground Water, and in Sludges (Ed Berg): Nine contractors were selected to participate in the IMVSs for SW-846 Methods 3005, 3010, and 3050. Participants have completed analyses of samples by Method 3005 and returned data is being processed by IMVS computer program. Bionetics, Inc., is currently producing the solid samples for use in the study for Method 3050 which will commence next quarter.

STATUS OF LABORATORY OUTPUTS:

Output 1696[B] Conduct Interlaboratory Method Validation Studies for SW 846 - Methods 8240 and 8270 due 12/87, completed 12/86. Output 1695[B] Conduct Interlaboratory Method Validation Studies for SW 846 - Metals Dissolution due 12/87, completed 12/86. Output 1360[B] Report on Status of Reference Materials for Chlorinated and Brominated Dibenzo-p-Dioxins due 12/86, completed 12/86.

- 2068 DUE: 12/30/87 REVISED: 09/30/87 COMPLETED: 09/17/87 STATUSREPORT-INTERLABORATORY METHOD VALIDATION STUDY FOR SW-846, METHOD 8240/5030 PURGE AND TRAP (100 COMPOUNDS)
- 2069 DUE: 12/30/87 REVISED: 09/30/87 COMPLETED: 09/17/87 STATUSREPORT INTERLABORATORY METHOD VALIDATION STUDY FOR SW-846, METHOD 8270/3510 (59 COMPOUNDS)
- 2071 DUE: 09/30/87 REVISED: COMPLETED: 09/17/87
 STATUS REPORT: INTERLABORATORY METHOD VALIDATION STUDY FOR SW-846 METHOD 3006, 3050, 3010; FOR METALS IN AMBIENT AND GROUND WATER, AND IN SLUDGES

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CODE TITLE

BUDGET SUB-ACTIVITY: D109 HAZARDOUS WASTE

ISSUE: I WASTE IDENTIFICATION

PPA (L): 12 VALIDATE METHODS FOR WASTE CHARACTERIZATION IN SW 846

PPA (S): EVAL/VALIDATE METHOS

RC: A EMSL CINN

PROJECT: 19 VALIDATE METHODS FOR WASTE CHARACTERIZATION IN SW-846

PROJECT OFFICER: J. J. LICHTENBERG PHONE: 513-684-7306

PLANNED START: 10/01/82 PLANNED END: 12/01/99

PROJECT DESCRIPTION:

GOAL: -RCRA requires the Agency to determine the chemical composition of waste and to detect trace levels of toxic constituents in groundwater and to be able to rapidly screen for hazardous constituents.

RATIONALE: The lack of standardized validated methods emphasizes the immediate need for a comprehensive program to assure that data of known quality are being collected. Improved laboratory methods will be required to furnish the quality assured data.

APPROACH: Evaluate and validate protocols and procedures to assure legally defensible and scientifically accurate data for hazardous waste measurement. This includes protocols for both organic and inorganic analyses. Protocols in "Test Methods for Evaluating Solid Waste" Document (SW-846) will be evaluated for precision, accuracy, and reliability on a wide variety of solid waste matrices.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

The major portion of work under this project is conducted extramurally. Two contractors, Battelle-Columbus and S-Cubed, are responsible for performing evaluation and single laboratory validation (development of accuracy and precision data) of the SW-846 solid waste methods. The contracts are flexible task order type contracts covering inorganic and organic chemical and physical methods.

GAS CHROMATOGRAPH (GC)/MASS SPECTROMETER (MS) SUITABILITY STUDIES FOR METHODS 8240 AND 8270--Previous reports have identified 39 additional compounds that could be chromatographed with 31 priority pollutants using the conditions specified in 8240 and 148 compounds that could be added to the priority pollutants that chromatograph using the conditions of 8270 (Item # 2153[A]). The final report on this work is planned for delivery 9/87.

PURGEABLES BY METHODS 8010, 8015, AND 8020--In response to a request by the Office of Solid Waste, Battelle prepared a special report for inclusion into the public record for the Appendix IX final regulation. The revised final report is expected early in the fourth quarter. A request will be made to modify the delivery date.

CAPILLARY COLUMN UPDATE OF METHOD 8240--Experimental work has been completed but the draft final report has been delayed by the need to prepare pesentations for the Solid Waste Symposium. The draft report is expected in August. SINGLE-LABORATORY VALIDATION OF METHOD 8240/5030--A task to generate single laboratory validation data for Method 8240 with a wide-bore capillary column has been initiated. Preliminary retention time and response factor data have been gathered and matrix validation has begun. The project is expected to stay on schedule.

EVALUATION OF SW-846 METHODS FOR VOLATILES IN SOLID MATRICES--Due to emergency personal leave taken by the principal investigator, start of the project is delayed until October.

EVALUATION OF METHOD 3510/3520 WITH GC--Those Appendix VIII/Michigan List compounds that could be chromatographed using the Superfund conditions for Method 8270 were screened for extractability and aqueous stability to provide information to support Appendix IX. The revised final report was received in early June. The project summary is due in July.

EVALUATION OF ALTERNATE EXTRACTION ph LEVELS--A comparative study of the four extraction procedures (ph variations) is being conducted on the Appendix IX list of compounds. The extraction procedures being examined are: (1) Acid/Neutral + Bases (A/N+B) - Initial extraction at ph 2 followed by extraction at ph 11; (2) Base/Neutral + Acids (B/N+A) - Initial extraction at ph 11 followed by extraction at ph 2; (3) Neutral + Acids (N/A) - Initial extraction at ph 7 followed by extraction at ph 2; (4) Extraction at ph 4 only. The results will be statistically evaluated. A status report has been written on existing data for presentation at the Solid Waste Symposium in Washington, D.C. in July. Data to complete the report will be collected in the fourth quarter.

EVALUATION OF GEL PERMEATION CHROMATOGRAPHY (GPC) CLEANUP--Elution profiles of the Appendix VIII compounds were completed. Acetone showed a dramatic influence on the gel as it causes shrinkage. Other ketones and similar laboratory solvents will be tested for column effects in the next quarter. EVALUATION OF SW-846 EXTRACTION METHOD FOR SEMIVOLATILES IN SOLIDS--(Item #2155[A]) The Appendix VIII and Michigan List analytes will be spiked into solid Resource Conservation and Recovery Act (RCRA) wastes and extracted to determine the accuracy and precision of the method(s) in SW-846. The original plans to conduct this activity in parallel with the validation of 8270 in water have been modified due to the delays in establishing the final pH for extraction of water and the need to complete the 8270 validation in a timely manner. It will now be a follow-on task at S-Cubed to commence in October, when the 8270 study will be finished.

EVALUATION OF GENERIC HEATED PURGE METHOD--(Item #2154[A]) METHODS 8320 AND 8330--Single laboratory testing of the GC/MS method for selected Appendix VIII analytes has been completed and will be incorporated into a report at the Solid Waste Symposium. The draft final report is to be submitted in August. FORMALDEHYDE AND OTHER ALDEHYDES--The task to complete referee validation of the method was discontinued until final experiments testing effects of filtraion on waste samples using adsorption cartridges are completed. Experiments in

comparing extractions using MTBE and methylene chloride for monitoring background levels using carbon filtered tap water and commercially available water matrices have been completed. Spiked real-world samples were also analyzed.

STATUS OF LABORATORY OUTPUTS: 1695[C] GC/MS Suitability Testing of RCRA Appendix VIII and Michigan List Analytes--DUE: 4/87 STATUS: To be delivered July 1987. 1695[D] Validation of SW-846 Methods 8010, 8015, and 8020--DUE: 5/87 STATUS: To be delivered 9/87. 1695[E] Screening of Semivolatile Organic Compounds for Extractability by EPA-Method 3510 and Aqueous Stability--DUE: 5/87 STATUS: To be delivered 9/87.

- 2153 DUE: 09/30/87 REVISED: 05/30/88 COMPLETED: FINAL REPORT ON SINGLE LAB VALIDATION OF METHODS 8240 AND 8270 FOR APPENDIX IX COMPOUNDS
- 2154 DUE: 12/30/87 REVISED: COMPLETED: FINAL REPORT ON SINGLE LAB VALIDATION OF METHOD 8240 AND HEATED PURGE AND TRAP METHODS FOR APPENDIX VIII PLUS THE MICHIGAN COMPOUNDS LIST
- 2155 DUE: 09/30/88 REVISED: COMPLETED: FINAL REPORT ON SINGLE LAB VALIDATION OF METHOD 8270 FOR APPENDIX VIII PLUS THE MICHIGAN COMPOUND LIST
- 1695 DUE: 12/30/86 REVISED: COMPLETED: 12/22/86
 PROGRESS REPORT GENERIC GC/MS METHODS FOR VOLATILE AND SEMIVOLATILE ORGANIC COMPOUNDS

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CODE TITLE

BUDGET SUB-ACTIVITY: D109 HAZARDOUS WASTE

ISSUE: I WASTE IDENTIFICATION

PPA (L): 14 DEVELOP NEW METHODS FOR SW 846

PPA (S): DEVELOP METHODS

RC: A EMSL CINN

PROJECT: 20 DEVELOP NEW METHOD FOR SW-846

PROJECT OFFICER: J. J. LICHTENBERG PHONE: 513-684-7306

PLANNED START: 10/01/82 PLANNED END: 12/01/99

PROJECT DESCRIPTION:

GOAL: Develop methodologies and protocols for analyzing and monitoring hazardous wastes including Appendix VIII compounds incorporating advanced techniques for more rapid and accurate methods. Develop and evaluate extractive procedures for organic wastes. Develop GC(LC)MS and GC fourier transform infrared/MS for cost effective analysis of groundwater and wastes. RATIONALE: Monitoring methods require developing, adopting and assessing technologies applicable to monitoring groundwater and complex wastes containing a significant portion of nonvolatile components. A broad spectrum of volatile, semivolatile compounds and landfill leachates containing intact, biodegraded or chemically altered components. Various wastes experience storage, treatment and disposal differences which require rapid, cost effective and/or unique monitoring methods.

APPROACH: Evaluate techniques and procedures for extraction, isolation, and measurement for rapid screening of organic chemicals in solid wastes. Compare existing methods for cost effectiveness.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

Cornell University, under a cooperative agreement, has a project titled "The Determination of Intractable Organic Compounds by Direct Liquid Injection (DLI)/Thermospray Liquid Chromatography (LC)/Mass Spectrometry (MS) and LC/MS/MS". In-house efforts consist of research to develop analytical procedures to identify and to measure nonvolatile and non-gas chromatographable hazardous compounds in environmental samples.

The monoclonal antibody immunoassay for chlorinated dibenzodioxins (CDDs) and chlorinated dibenzofurans (CDFs) was extensively tested using spiked California soils and contaminated Missouri soils provided by Region 7. During this testing unacceptably variable results were obtained, and it was necessary to

find the source of the problem before field testing could begin. The sample extraction and preparation procedure was reviewed and modified to eliminate sources of variability and to allow automation of the procedure to further reduce variability. The testing resumed late in the quarter and will continue until consistent assays with good precision can be obtained.

STATUS OF LABORATORY OUTPUTS: None.

STATUS AND SCHEDULE OF DELIVERABLES:

2074 DUE: 12/30/87 REVISED: COMPLETED: ORGANIC CHEMICAL SCREENING METHOD EVALUATION

1676 DUE: 12/30/86 REVISED: COMPLETED: 12/22/86 REPORT ON DEVELOPMENT OF ANALYTICAL METHODS FOR DIOXINS AND

FURANS

> CODE TITLE

BUDGET SUB-ACTIVITY: Y105 HAZARDOUS SUBSTANCES

> ISSUE: A PROVIDE TECHNIQUES&PROCEDURES FOR SITE&SITUATION ASSESSMENT

PPA (L): 04 TECHNIQUE FOR SITE ASSESSMENT

PPA (S): SITE ASSESSMENT

> RC: A EMSL CINN

21 PROJECT: Monitoring for Site Assessment

PLANNED START: 06/01/81 PLANNED END: 12/30/99

PROJECT DESCRIPTION:

GOAL: The success of removal and remedial actions depends on an accurate definition of the kinds and severity of the problem. The latest protocols, techniques, instrumentation, for sampling and analysis and remote monitoring must be applied to provide the decision maker with scientifically accurate information.

RATIONALE: Methods modifications and evaluations of methods are needed to increase the efficiency of site and situation assessment and cost-effective-Systems are required to enhance the quality of data acquisition and the speed and reliability of data analysis. Water monitoring represents over 50% of the analytical work at most uncontrolled hazardous waste sites. APPROACH: Protocols, techniques and methods will be prepared, tested, modified, and documented. Emphasis will be on automated methods to ensure cost effectiveness, speed, consistency, and reliability. Water and water related media will be given high priority along with CERCLA analytes published in the Federal Register.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

Work began on a multilaboratory test of a gas chromatography (GC)/mass spectrometry (MS) method for semivolatiles, pesticides, and polychlorinated biphenyls (PCBs) in a single extraction and analysis. Included in the method is a modified pH adjustment sequence and a gel permeation chromatography cleanup step with detailed directions and required quality control. Included in the multilaboratory test is the use of software to automate the interpretation of some of the data, software to automate all the computations, and software to place all the results in the proposed Agency standard format for delivery of monitoring data on computer readable media. The participating laboratories, which include seven Superfund contract laboratories, Region 7, and Battelle Columbus, received spiked water and sediments, sediments environmentally

contaminated, all calibration solutions, and all software. The results will be evaluated during the last quarter.

STUDY TO OPTIMIZE EXTRACTION PROCEDURES--Preliminary results of these tests revealed recoveries that were lower than expected. Problems in the extraction procedures were identified and the affected tests are being repeated. This study in reagent water is scheduled for completion next quarter.

STATUS OF LABORATORY OUTPUTS: None.

- 1623 DUE: 12/30/86 REVISED: COMPLETED: 12/19/86 MONITORING METHODS FOR CERCLA SITE ASSESSMENT -- FY 86
- 2200 DUE: 11/30/87 REVISED: COMPLETED: SUMMARY REPORT MONITORING METHODS FOR CERCLA SITE ASSESSMENT
- 0501 DUE: 09/30/87 REVISED: COMPLETED: 09/28/87
 PRESENTATION ON RESULTS OF STUDY TO OPTIMIZE EXTRACTION PROCEDURES FOR CERCLA ANALYTES USING THE CLP GC/MS METHOD

CODE TITLE

BUDGET SUB-ACTIVITY: Y105 HAZARDOUS SUBSTANCES

ISSUE: F PROVIDE TECHNICAL SUPPORT TO ENFORCEMENT, PROGRAM & REGIONS

PPA (L): 06 MONIT. FOR ENFORCE. & TECH. SUPPORT

PPA (S): TECH SUPPORT

RC: A EMSL CINN

PROJECT: 22 Monitoring for Enforcement and Other Technical Support

PROJECT OFFICER: J. Lichtenberg PHONE: 008-684-7306

PLANNED START: 06/01/81 PLANNED END: 12/30/99

PROJECT DESCRIPTION:

GOAL: All projects under CERCLA have potential enforcement requirements.

RATIONALE: The ORD is frequently required to provide scientific data for case

preparation.

APPROACH: The ORD will continue to provide oversight on settlement agreements for example, Hyde Park, Love Canal. Services covering sampling, analytical techniques, quality assurance samples, and review of monitoring plans will be provided.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

Inhouse: The Quality Assurance (QA) Branch of EMSL-Cincinnati provides performance evaluation (PE) samples to the officials in charge of the Hyde Park-USEPA agreement, periodically, upon request. Hooker Chemical Company is required to analyze for 21 target compounds in the PE samples to prove proficiency in their analytical techniques used in managing the dump site. The manager in Region 2 and the New York Department of Health officials. Inhouse and extramural activities are conducted to cover sampling, analytical service needs, and review of monitoring plans.

ACCOMPLISHMENTS AND RESULTS TO DATE:

Sampling and Analytical Techniques (Bill Budde): Specifications were provided, in response to a request from the Office of Emergency and Remedial Response (OEER), for a format for inorganic data deliverables on computer readable media. These specifications are compatible with the proposed Agency standard format for delivery of monitoring data on computer readable media. In response to another request, a file containing 44,205 Chemical Abstracts Service

Registry Numbers and corresponding chemical nomenclature was made available on the National Computer Center mainframe at Research Triangle Park. This file is needed by the contract laboratory program to verify information provided by contract laboratories. A revised reference data base of 44,205 electron ionization mass spectra was prepared and distributed. This collection contains 1944 new spectra, and 1700 old spectra were replaced with higher quality spectra. Participated on a peer review panel to evaluate 28 proposals received in response to an U.S. Environmental Protection Agency (USEPA) request for applications for grants to develop measurement and monitoring methods for Superfund sites. This Superfund research effort by non-profit research organizations and education institutions was authorized under the Superfund Amendments and Reauthorization Act of 1986. The peer review panel evaluated the proposals according to their technical merit, and the probability that the research could produce results that would support USEPA's hazardous waste site clean-up program.

Evaluation of Performance/Hyde Park Litigation (Harold Clements): Samples were analyzed by Occidental Chemical Company and data were returned to the QA Branch for evaluation last quarter. All data were acceptable. No new activity on this project this quarter.

STATUS OF LABORATORY OUTPUTS:

Output 1632[B] Annual Report of PE on Hyde Park Remedial Project of Contractor/ Enforcement Performance, due 12/86 was completed.

STATUS AND SCHEDULE OF DELIVERABLES:

1632 DUE: 11/30/86 REVISED: COMPLETED: 11/26/86 INTERNAL REPORT Water Monitoring Support Programs

2199 DUE: 11/30/87 REVISED: COMPLETED:

SUMMARY REPORT - ANALYTICAL TECHNICAL ASSISTANCE TO WATER MONITORING

SUPPORT PROGRAMS

CODE TITLE

BUDGET SUB-ACTIVITY: Y105 HAZARDOUS SUBSTANCES

ISSUE: Q PROVIDE QUALITY ASSURANCE - SUPERVISE PROGRAM REQUIREMENTS

PPA (L): 01 QUALITY ASSURANCE SUPPORT FOR CERCLA

PPA (S): QUALITY ASSURANCE

RC: A EMSL CINN

PROJECT: 23 Quality Assurance Support for CERCLA

PROJECT OFFICER: Ed Berg PHONE: 008-684-7325

PROJECT DESCRIPTION:

GOAL: The success of removal and remedial actions depends on an accurate definition of the kinds and severity of the problem. The latest protocols, techniques, instrumentation, quality assurance for sampling and analysis and remote monitoring must be applied to provide the decision maker with scientifically accurate information. A quality assurance program is needed to provide support to the National Contract Laboratory Program which is responsible for all contract chemical analyses under Superfund. RATIONALE: Quality control samples and reference calibration standards are provided to Superfund contractors as independent checks on the analyst's technique and application of the analytical procedure. Additionally performance of Superfund contractors evaluated quarterly to assure the legal defensibility of their monitoring data. APPROACH: Quality control and performance evaluation samples are prepared and distributed to Superfund contractors by Bionetics, Inc. Reference calibration standards are prepared and distributed by Northrop Services. Inc. control samples and reference calibration standards are verified by referee

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

laboratories.

EMSL-Cincinnati provides quality assurance (QA) support to EMSL-Las Vegas which is the lead laboratory responsible for QA support to the Superfund program. EMSL-Cincinnati's QA program portion follows.

Quarterly performance evaluation (PE) studies are conducted on the Contract Laboratory Program (CLP) laboratories which are providing analytical support for Superfund site assessments. Bionetics, Inc., was awarded a new contract on

July 1, 1985, and assigned this task. A unit was set up to provide full-volume PE samples containing about 20 volatiles, semi-volatiles, and pesticides. Samples are prepared, packed in coolers, iced, and shipped to regional personnel who label the samples and then mail them to the CLP laboratories as double-blind samples. Bionetics, Inc., analyzes each sample set and reports the true concentration and the analyzed concentration of each analyte to EMSL-Las Vegas. CLP laboratory data are returned to EMSL-Las Vegas for evaluation. Comprehensive Environmental Response Compensation and Liability Act (CERCLA) compounds. Northrop Services operates the Repository for Toxic and Hazardous Materials for EMSL-Cincinnati, which provides calibration standards for the same list. Referee laboratories verify the true concentrations of analytes in the QC samples and calibration standards.

Special series of sample concentrates and full-volume PE samples are produced and distributed by Bionetics, Inc., to evaluate contract laboratories performing analyses on samples from the Ground Water Monitoring Survey of hazardous waste sites being conducted by the Office of Solid Waste and Emergency Response (OSWER). Approximately 58 sites will be sampled in FY86 and FY87.

ACCOMPLISHMENTS AND RESULTS TO DATE:

Evaluation of Superfund Contractors (Ed Berg): Approximately 70-80 sets of PE samples are currently being prepared for the fourth quarter evaluation of CLP laboratories.

USEPA Repository for Toxic and Harardous Materials (Harry Kolde): Eleven new CERCLA chemicals were prepared as analytical standards and two were re-ampuled to replenish low stocks. The Repository distributed 20,400 ampuls to 420 USEPA regional, state, local, and USEPA contract laboratories this quarter. Ten sets of mixed polychlorinated biphenyl (PCB) congener standards were supplied to support the New Bedford Harbor Study. Ten CERCLA chemicals were sent to referee laboratories for purity verification.

QC Samples (Ray Wesselman): A total of 46,330 samples were distributed to USEPA, USEPA contract, state, and local laboratories. Due to depletion of inventories, 6200 ampuls of chlorinated hydrocarbon pesticides and 5900 PCBs Aroclor 1260 were produced and are being verified.

Conduct Referee Analyses (Harry Kolde): Third quarter analyses were as follows: (1) spiking/calibration solutions for Superfund compouds, 187 ordered, none completed; (2) QC and PE samples, 813 ordered, 249 completed; and (3) surrogates for USEPA GC/MS Methods, 9 ordered, none completed. Future work - continue verification analyses.

Ground Water Monitoring PE Samples (Ed Berg): No activity for this quarter.

STATUS OF LABORATORY OUTPUTS:

Output 1626[B] Annual Summary Report - QA to Support CLP - PE Studies due 2/87 was completed. Output 1628[B] Annual Report on Referee Analytical Services due 12/86 was completed. Output 1626[C] Status Report - PE Study to Ground Water

Monitoring Program due 2/87 was completed. Output 1628[C] Annual Report - Repository for CERCLA Hazardous and Toxic Compounds due 12/86 was completed.

- 1628 DUE: 12/31/86 REVISED: COMPLETED: 12/19/86 Annual Report Quality Assurance to Support CERCLA
- 0509 DUE: 12/30/87 REVISED: COMPLETED:
 ANNUAL REPORT QUALITY CONTROL SAMPLE PROGRAM FOR CERCLA (FY 87)
- 0510 DUE: 12/30/87 REVISED: COMPLETED: ANNUAL REPORT -- QUALITY ASSURANCE TO SUPPORT CONTRACT LAB PROGRAM (FY 87)
- O511 DUE: 12/30/87 REVISED: COMPLETED:
 ANNUAL REPORT USEPA REPOSITORY FOR TOXIC AND HAZARDOUS MATERIALS,
 ACTIVITIES FOR CERCLA (FY 87)

CODE TITLE

BUDGET SUB-ACTIVITY: L104 TOXIC CHEMICAL TESTING F ASSESSMENT

ISSUE: A TEST METHOD DEVELOPMENT

PPA (L): 02 ENVIRONMENTAL HAZARD ANALYSIS FOR TOXIC SUBSTANCES

PPA (S): HAZARD ANALYSIS

RC: A EMSL CINN

PROJECT: 24 ENVIRONMENTAL HAZARD ANALYSIS FOR TOXIC SUBSTANCES

PROJECT OFFICER: J. J. LICHTENBERG PHONE: 513-684-7306

PROJECT DESCRIPTION:

GOAL: To evaluate chemicals which are of specific interest to the regulatory programs in the Office of Toxic Substances. Improving the quality of scientific and technical assessment is a major goal of the Office of Toxic Substances.

RATIONALE: Application of new or improved analytical methods to identify and quantitate chemicals of concern in the environment and to develop reliable methods for routine monitoring associated with regulatory reviews is an important component of OTS regulatory programs. This research provides biological and chemical methods for identification and quantification of of chemical compounds being reviewed by OTS.

APPROACH: Sampling and analytical procedures used to identify and quantitate the presence of chemical compounds in the environment and the biota will be assessed to meet regulatory needs. Application of immunochemical techniques including monoclonal antibodies will be investigated. Influence of extraction and clean-up procedures for multi-residue methods will be evaluated.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

Cornell University, under a cooperative agreement, has a project titled "The Determination of Intractable Organic Compounds by Direct Liquid Injection (DLI)/Thermospray Liquid Chromatography (LC)/Mass Spectrometry (MS) and LC/MS/MS". The first budget period of the three-year cooperative agreement was extended to September 1986. This first budget period was funded in FY 85 with L104 research and development resources to investigate methods for azodyes. FY 87 funds are now available for continuation of this project.

ACCOMPLISHMENTS AND RESULTS TO DATE:

During the quarter a funding package was provided for a cooperative agreement

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with Cornell University, and funds (\$49K) were obligated (Item #2190[A]). The principal investigator at Cornell began the search for a post doctoral research associate.

Two requests for information on the analytical chemistry of several large volume commercial chemicals were received from the Office of Toxic Substances, and brief status reports were provided within one week in each case. A presentation was made at the program review for the Chemical Testing and Assessment Research Committee.

STATUS AND SCHEDULE OF DELIVERABLES:

2190 DUE: 09/30/87 REVISED: COMPLETED: 09/25/87 REPORT ON ADVANCED ANLAYTICAL TECHNIQUES APPLIED TO MONITORING TOXIC SUBSTANCES

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FOURTH QUARTER STATUS REPORT ON FY'87 PROJECT DESCRIPTIONS Office of Acid Deposition, Environmental Monitoring and Quality Assurance EMSL CINN: JULY - SEPTEMBER 1987

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CODE TITLE

BUDGET SUB-ACTIVITY: L104 TOXIC CHEMICAL TESTING F ASSESSMENT

> ISSUE: J **SUPPORT**

PPA (L): 28 **OUALITY ASSURANCE FOR TOXIC SUBSTANCES**

PPA (S): QUALITY ASSURANCE

> RC: EMSL CINN

PROJECT: 25 QUALITY ASSURANCE FOR TOXIC SUBSTANCES

PHONE: 513-684-7325 PROJECT OFFICER: JOHN WINTER

> PLANNED END: 12/01/99 PLANNED START: 10/01/80

PROJECT DESCRIPTION:

GOAL: Provide quality assurance to support implementation of sections 4 and 6 of the Toxic Substances Control Act

RATIONALE: Quality assurance support for biological monitoring networks is needed to ensure their application to regulatory reviews. Such data are needed to conduct various analyses including exposure and risk assessments required under the Toxic Substances Control Act.

APPROACH: Quality control samples and calibration standards will be prepared and distributed for chemicals of interest to the Office of Toxic Substances. Traceability to NBS will be addressed under the current IAG.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

EMSL-Cincinnati is responsible for the production and distribution of quality control (QC) and performance evaluation (PE) samples and calibration standards to support the quality assurance (QA) program of the Office of Toxic Substances (OTS). Contracts were awarded to Bionetics, Inc., to produce and distribute QC and PE samples, to Northrop Services, Inc., to produce and distribute calibration standards and to eight laboratories to serve as organic and inorganic referee laboratories. The National Bureau of Standards (NBS) is on the second year of an interagency agreement (IAG) to establish traceability for QA products where practical.

ACCOMPLISHMENTS AND RESULTS TO DATE:

USEPA Repository for Toxic and Hazardous Materials (Harry Kolde): A total of total of 20,400 ampuls were distributed to 420 USEPA, USEPA contract, state, and local laboratories. Four 13C-polychlorinated biphenyl (PCB) calibration

standards were sent to OTS contractors.

Traceability to NBS (Ed Berg): A priority list of PE and QC samples requiring NBS analyses was agreed upon. NBS will begin production of 3,000 sets of PCB congeners for USEPA. Analyses have begun on selenium in fish samples.

QC Samples (Ray Wesselman): A total of 46,330 QC samples were distributed to USEPA, USEPA contract, state, and local laboratories. To replace depleted inventories, 24,000 ampuls of chlorinated hydrocarbon pesticides, PCBs, and PCBs in oils were produced and analyzed.

STATUS OF LABORATORY OUTPUTS:

Output 1941[B] Annual Report on Traceability to NBS due 12/86 was completed. Output 1941[C] Annual Report on Referee Analytical services due 12/86 was completed.

- 2094 DUE: 12/30/88 REVISED: 12/30/87 COMPLETED:
 ANNUAL REPORT ON QC SAMPLE PROGRAM -- EPA REPOSITORY FOR HAZARDOUS MATERIALS (FY 87)
- 1941 DUE: 12/30/86 REVISED: COMPLETED: 12/19/86 ANNUAL REPORT QC SAMPLE PROGRAM EPA REPOSITORY FOR TOXIC AND HAZARDOUS MATERIALS (FY 86)

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EMSL CINN: JULY - SEPIEMBER 1907

CODE TITLE

BUDGET SUB-ACTIVITY: H109 INTERMEDIA

ISSUE: N MANAGE THE MANDATORY QUALITY ASSURANCE PROGRAM

PPA (L): 01 MANAGEMENT SUPPORT FOR MANDATORY AGENCYWIDE QA PROGRAM

PPA (S): MGMT SUPPORT FOR QA

RC: A EMSL CINN

PROJECT: 26 MANAGEMENT SUPPORT FOR MANDATORY AGENCY-WIDE QA PROGRAM'

PROJECT OFFICER: DANIEL BENDER PHONE; 513-684-7301

PROJECT DESCRIPTION:

GOAL: To provide the Administrator with central management support for the Mandatory Agency-wide Quality Assurance (QA) Program.

RATIONALE: It is necessary to develop policy, management procedures, and technical procedures; coordination and direction for program implementation and to review, evaluate, and audit Agency-wide QA activities in order to respond to the QA mandate.

APPROACH: As the developmental mode proceeds to completion, the implementation and evaluation activities will be increased via reviewing program plans, conducting management system audits, reviewing QA audits, developing and improving technical guidance, reviewing standard methods of various media, assuring Agency allocation of adequate resources for Agency-wide QA programs, and preparing a QA program status report for senior Agency management.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

PROJECT STATUS

This project is an ongoing support function involving the supplying of technical and policy information and technical services such as document review, committee and work group (WG) membership, document and report preparation, and the coordination of the input into these activities of regional, headquarters, and national laboratory Quality Assurance Officers (QAOs).

ACCOMPLISHMENTS AND RESULTS TO DATE.

Policy statements concerning quality assurance (QA) positions of the Office of Acid Deposition, Environmental Monitoring, and Quality Assurance (OADEMQA) and its four laboratories were written and/or reviewed. Progress toward the

implementation of these policies will be reported at an OADEMQA QAO meeting during the first quarter of fiscal year (FY) 1988. Another round of regional, national program office, and Office of Research and Development (ORD) QAOs conference calls were held to plan for the national QAO meeting to be held in early FY88.

Information concerning the routinely-used measurement method (RUMM) project was sent to the contractor for the Section 518 report. The Physical and Chemical Methods Branch performance in studies WPO18 and WSO20 was examined. An audit of EMSL-Cincinnati sample handling and analysis procedures of the pilot study for the National Pesticide Survey was performed.

The regional QAOs were contacted regarding the responses of the Environmental Services Divisions Director to a memorandum from the Quality Assurance Management Staff (QAMS) concerning the RUMM project.

Reviews were made of manuscripts concerning management systems audits in ORD laboratories, QA Program Plan Guidance, Intralaboratory Quality Control, Data Quality Objectives in ORD laboratories, the LaMotte Residual Chlorine Titratjon Kit as an alternate test procedure, and the EMSL-Cincinnati recommendation for an analytical method for waters disinfected by chlorine dioxide.

STATUS OF LABORATORY OUTPUTS: None.

STATUS AND SCHEDULE OF DELIVERABLES:

NO DELIVERABLES AVAILABLE

CODE TITLE

BUDGET SUB-ACTIVITY: N104

MULTI-MEDIA ENERGY

ISSUE: E

UNDERSTAND & QUANTIFY AQUATIC EFFECTS

PPA (L): 01

NATIONAL SURFACE WATER SURVEY

PPA (S):

NATL SURF WATER SRVY

RC: A

EMSL CINN

PROJECT: 27

EVALUATION OF ANALYTICAL METHODOLOGY FOR ALUMINUM

PROJECT OFFICER: J. LICHTENBERG

PHONE: 513-684-7306
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PLANNED END: 09/01/89

PROJECT DESCRIPTION:

GOAL: To evaluate existing methods used to determine the various species of aluminum.

RATIONALE: The potential for aluminum toxicity is related to the chemical form of aluminum present in freshwater systems. Current methods may not be measuring the various species. Consequently, knowledge as to which methods produce data for these species is needed.

APPROACH: Sampling and handling protocols and the existing methods will be compared using developed standard reference materials and real world samples.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

This project appears in the work plans of the Environmental Monitoring Systems Laboratory - Research Triangle Park (EMSL-Research Triangle Park) and an [A] deliverable is listed in their Project Output Plan. The development and evaluation of "Methods for the Analysis of Ions in Atmospheric Deposition" is being conducted under a cooperative agreement with Illinois State Water Survey. The contractor is responsible for acquiring available published methods and delivering them to the Environmental Monitoring and Support Laboratory - Cincinnati (EMSL-Cincinnati) for review prior to selection of methods for evaluation and validation.

EVALUATION OF METHODS FOR REACTIVE ALUMINUM--(Item #2111[A]) The second year funding package was prepared for the cooperative agreement to evaluate methods for determination of aluminum in acid rain.

Jack Pfaff attended the ASTM meeting of ASTM Committee D-22 on Atmosphere (Acid Rain) in Cincinnati on April 29-30. He chaired the discussion on the method to determine calcium, magnesium, potassium, and sodium by atomic absorption in acid rain samples.

STATUS OF LABORATORY OUTPUTS: None.

STATUS AND SCHEDULE OF DELIVERABLES:

2111 DUE: 12/30/87 REVISED: COMPLETED: REPT ON EVALUATION OF ALUMINUM SPECIATION USING SYNTHETIC AND NATURAL

SAMPLES

CODE TITLE

BUDGET SUB-ACTIVITY: N104 MULTI-MEDIA ENERGY

> UNDERSTAND & QUANTIFY AQUATIC EFFECTS ISSUE: Ε

EFFECTS OF SNOWMELT & STORM EPISODES ON SURFACE WATER ACIDIF PPA (L): 08

PPA (S): EPISODIC RESEARCH

> RC: EMSL CINN

EFFECTS OF SNOWMELT AND STORM EPISODES ON SURFACE WATER PROJECT: 28

PHONE: -684-7337

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> PLANNED START: 10/01/86 PLANNED END: 09/30/92

PROJECT DESCRIPTION:

GOAL: Provide information on the effects of acidification of surface waters during snowmelt and storm episodes on fish and other aquatic life. RATIONALE: Episodic acidification of surface waters from snowmelt and storms has been cited as a major factor affecting aquatic organisms. Although considerable data exist regarding the extent of chronic acidification, there are very little data as to the extent, magnitude, duration and frequency of short-term, episodic acidification in potentially sensitive regions, and the biological effects of episodic acidification. APPROACH: Field and laboratory studies of the aquatic biology effects of episodic acidification on fish and other aquatic life will begin in FY-87 at single site in New England or the mid-Atlantic states. Toxicity tests of surface waters containing acidic runoff from manipulated catchments will be performed with indigenous and/or surrogate fish and invertebrates on-site in a mobile laboratory and in at EMSL-Cincinnati. Field studies of the standing crop, species composition, and condition of indigenous fish and invertebrates will be carried out prior to and following application of acid materials to experimental catchments. Biological efects will be related to pH, aluminum, calcium, alkalinity and other relevant water quality parameters.

PROJECT STATUS AND ACCOMPLISHMENTS TO DATE:

A contract has been awarded for development of the detailed work plan for the field bioassay portion of the Episodic Response Project.

STATUS OF LABORATORY OUTPUTS: None

STATUS AND SCHEDULE OF DELIVERABLES:

DUE: 08/30/87 REVISED: 09/30/87 COMPLETED: 09/28/87 FIELD BIOASSAY PLAN FOR THE EPISODIC RESEARCH PLAN (ERP) (TO OADEMOA)