ORGANIC CHEMICALS DIVISION RESEARCH *Report*

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NEWSLETTER ☆

Analytical Quality Control

ENVIRONMENTAL PROTECTION AGENCY
NATIONAL ENVIRONMENTAL RESEARCH CENTER
METHODS DEVELOPMENT and QUALITY ASSURANCE RESEARCH LABORATORY
· CINCINNATI, OHIO 45268 ·
PHONE: 513-684-2924

EDITOR
J. B. Anderson

No. 22 July 1974

The response to our request to complete a code block for confirmation and updating the mailing list has been excellent. This issue is being sent to all respondents. Former recipients and new requestors will be added to the Newsletter mailing list upon completion of a code block which will be supplied upon request. A current list of those receiving the Newsletter will be sent to each Regional coordinator in early August.

AQC COORDINATOR

A new AQC coordinator appointment has been made in Region IX. Dr. Peter K. Mueller has accepted a new position with a consulting firm. His replacement is:

Dr. Ho L. Young
Laboratory Support Branch
EPA, Region IX
620 Central Avenue, Bldg. 1
Alameda, California 94501

INTERNATIONAL JOINT COMMISSION NEWSLETTER

The Great Lakes Regional Office will soon issue a quarterly Newsletter, Great Lakes Focus. If you are interested in receiving this free Newsletter request it from:

IJC Regional Office
100 Ouelette Avenue
Suite 800
Windsor, Ontario, Canada N9A 6T3

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Radiochemistry Conference

The twentieth annual conference on Bioassay, Environmental and Analytical Chemistry will be held at Stouffer's Inn, Cincinnati, Ohio on September 24-25, 1974. The conference will be concerned with the analysis of inorganic and radiochemical content of biological samples. Further information may be obtained from:

Dr. Richard Blanchard
Radiochemistry & Nuclear Engineering Facility
National Environmental Research Center
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268
Phone: 513-684-2378

AIR NEWS

Laboratory Evaluation of Ambient NO_2 Methods

The Methods Standardization Branch, NERC/RTP, has evaluated two manual procedures for measurement of ambient nitrogen dioxide and has developed Tentative Methods based on these evaluations. The procedures are the sodium arsenite method and the triethanolamine-guaiacol sodium sulfite (TGS) method. Both methods appear to be satisfactory for measuring ambient NO_2 and the evaluations are fully described in the following reports, which are available from the Methods Standardization Branch.

"An Evaluation of the Arsenite Procedure for the Determination of Nitrogen Dioxide in Ambient Air," M. E. Beard and J. H. Margeson, March 1974.

"An Evaluation of the TGS-ANSA Procedure for the Determination of Nitrogen Dioxide in Ambient Air," R. G. Fuerst and J. H. Margeson, March 1974. (J. H. Margeson, 919-549-2196)

COLLABORATIVE TESTS OF STATIONARY SOURCE EMISSION

Test Methods

The compliance test methods for measuring SO_2 and NO_x emissions from power plants and NO_x emissions from nitric acid plants (Methods 6 and 7 of the New Source Performance Standards) have been collaboratively tested. These collaborative studies are fully described in the following reports, which are available from the Methods Standardization Branch, NERC/RTP.

"Collaborative Study of Method for the Determination of Sulfur Dioxide Emissions from Stationary Sources (Fossil Fuel-Fired Steam Generators)," H. F. Hamil and D. E. Camann (Southwest Research Institute), December 1973.

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"Collaborative Study of Method for the Determination of Nitrogen Dioxide Emissions from Stationary Sources (Nitric Acid Plants) " H. F. Hamil and R. E. Thomas (Southwest Research Institute), May 1974. (M. R. Midgett 919-549-2196)

QUALITY ASSURANCE GUIDELINES FOR STATIONARY SOURCE EMISSION

Test Methods

The first in a series of quality control guideline documents for stationary source emission test methods specified with Standards of Performance for New Stationary Sources is now available from the Quality Control Coordinator in each Region. This first document is titled, "Guidelines for Development of a Quality Assurance Program: Volume I - Determination of Stack Gas Velocity and Volumetric Flow Rate (Type-s Pitot Tube)," EPA-650/4-74-0050. (S. M. Bromberg 919-549-2220)

NATIONAL ENVIRONMENTAL RESEARCH CENTER - LAS VEGAS, NEVADA

Biological Reference Material Containing Plutonium

The Monitoring Systems Research and Development Laboratory of NERC-Las Vegas is involved in the preparation of selected types of biological reference materials containing various radionuclides. Recently some 50 pounds of liver was obtained from dairy animals from the Nevada Test Site and prepared as a reference sample. Aliquots of the sample were taken and will be submitted to various laboratories for plutonium-239 analysis to obtain a certified concentration. It is anticipated that this reference sample will be available for distribution by January 1, 1975. (E. W. Bretthauer, 702-736-2969)

REGION IV, SURVEILLANCE AND ANALYSIS DIVISION, ATHENS, GEORGIA

Sample Preparation for Organic Chemicals

The Region IV sample preparation technique "Procedure for the Extraction of Industrial Organic Chemicals in Effluents and Streams" has recently been modified to improve the extraction efficiency. The primary changes are: (1) adding 5 gm of sodium chloride to improve the extraction efficiency of hexane for non-polar compounds and (2) changing the extraction solvent for acid compounds from methylene chloride to a combination of ethyl ether-methylene chloride. A copy of the procedure is available. (B. Loy, 404-546-3165)

REGION V, CENTRAL REGIONAL LABORATORY, CHICAGO, ILLINOIS

Quality Control

An interlaboratory collaborative testing program has been developed in the Twin Cities area. The Twin Cities Round Robin Program is a volunteer program, composed of governmental, independent and industrial laboratories involved in the analysis of waters and wastewaters.

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The purpose of the program is to determine the correlation of results between laboratories and to give validity to each laboratory's in-process quality control program. (D. A. Payne, 312-353-8370)

REGION VII LABORATORY, KANSAS CITY, KANSAS

Coliform Recoveries on Membrane Filters

The comparative study of Gelman and Millipore membrane filters by Presswood and Brown (Appl. Microbiol. 26:332-336) prompted our microbiology laboratory to evaluate the two brands of membrane filters employing routine samples. The study included a total of 100 samples from a variety of unchlorinated aquatic sources. Gelman filters averaged 2.5 times greater recovery of fecal coliform than Millipore filters. A comparative study, with fewer samples, was also made utilizing total coliform. Tentative data indicate that total coliform recovery is similar with the two brands. Colonies from both brands of filters were transferred to confirmatory media. Also, different lots of each brand of filter were used. (F. L. Harris, 816-374-4285)

REGION X LABORATORY, REDMOND, WASHINGTON

Analysis of Suspended Solids in Pulp Wastes

The EPA Region X Laboratory, the Port Angeles Division and the Research Division of ITT Rayonier, Inc., have completed a cooperative study to determine the best techniques for analyzing suspended solids in pulp wastes. Extensive studies conducted by the company have shown the EPA method, which uses a glass fiber filter and a 100 ml sample, to be the superior method. In those cases where filter-clogging materials result in extended filtration times, they have further shown that a combination filter using a mat of coarse asbestos fibers over the glass fiber disk is preferred. (A. R. Gahler, Coml. 206-883-0833)

Method for Total Dissolved Gas Pressure

Atmospheric gas supersaturation of natural waters is responsible for the gas-bubble disease of aquatic organisms. The problem is particularly important in the Columbia River basin. Methods for the measurement of dissolved nitrogen include the Van Slyke procedure and gas chromatography. A new, simple device for measurement of total dissolved gas pressure has been developed which permits field sampling. The saturometer, now in use in Region X, was developed by Dr. Ray Weiss, Scripps Institute of Oceanography, and is described in a paper by Fickelsen, Schneider, and Montgomery of Battelle Northwest. Copies of the paper and specifications for the instrument are available. (R. L. Rullfson, 206-442-1216)

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NATIONAL ENVIRONMENTAL RESEARCH CENTER-CINCINNATI
INDUSTRIAL WASTE TREATMENT RESEARCH LABORATORY, EDISON, NJ

Effect of Fluorescence Quenching on Oil Identification

Questions have been raised by several investigators about the effects of quenching on fluorescence spectra when used for passively tagging oils. Quenching as defined for the fluorescence analysis of oil, occurs at high concentrations and is characterized by supression of the oil's emission radiation. The exact concentrations at which quenching becomes apparent was determined for four oils. Solutions having known concentrations of the oils in 99 Mol % pure cyclohexane were prepared. Emission spectra were obtained in the range 220-600 nm, by exciting at 290 and 340 nm. Graphs were then constructed by plotting fluorescence intensities versus concentrations. The points at which the plots began to deviate from linearity marked the "onset of quenching." Those points corresponded to the following concentrations:

OIL	CONCENTRATION OF "ONSET OF QUENCHING" mg/l	
	Ex 290 nm	Ex 340 nm
#2 Fuel Oil	30	No Emission
#6 Fuel Oil	6	7
Bachaquero Crude	6	18
Iran - Gach Crude	16	25

By inspection of the spectral envelopes it was found that above the onset of quenching the shapes of the envelopes varied with concentration, while below the onset (region of linarity) the shapes remained constant. In addition, at concentrations above 100 mg/l the envelopes were sometimes distorted beyond recognition. It is therefore vital to prepare oil solutions with concentrations below the onset of quenching when passively tagging oils by the fluorescence technique. (U. Frank, FTS 201-548-3510, Coml. 201-548-3347)

METHODS DEVELOPMENT AND QUALITY ASSURANCE RESEARCH LABORATORY
CINCINNATI, OHIO

Reference Standard for Vinyl Chloride

The current widespread interest in the analysis of water for vinyl chloride has pointed out a serious problem in preparing a reference standard of this compound. The method of analysis most commonly employed for the determination has been direct aqueous injection gas chromatography using a flame ionization detector. For this analysis, most analysts presently prepare calibration standards gravimetrically in an organic solvent or in water.

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Although vinyl chloride is soluble in many organic solvents, losses due to the high vapor pressure of these solvents are a problem. Clearly, there is a need for an accurate and reliable standard for calibration of vinyl chloride measurements in water. Accurate results and valid interlaboratory comparison of data require a stable and accurate reference standard. MDQARL has undertaken a study to determine the feasibility of preparing such a standard. Investigations to date have included dissolving vinyl chloride in acetone or methyl alcohol at a low temperature and sealing the solution in glass ampuls. The ampuls are stored in a freezer. When ready for use, the ampuls are opened (cold) and the vinyl chloride solution is immediately dosed into water and the concentration determined by microcoulometric gas chromatography (MCT-GC). This assumes that the MCT-GC can be used as a primary analytical method without reference to a known standard. It is too early to tell whether or not this approach will provide adequate reference standards of vinyl chloride. (T. Bellar, 513-684-2911 or J. Eichelberger, 513-684-2918)

Laboratory Computerization Progress Report No. 3

In the last issue of this Newsletter is was reported that bids would be received for the computer hardware for the pilot laboratory computerization system. These bids were evaluated and the Data General Corporation, Southboro, Massachusetts was the successful bidder. The laboratory automation system will employ a Model 840 mini-computer and operate under a real time disk operating system. Other hardware components include a medium speed line printer, an industry standard magnetic tape unit, five cathode ray tube/keyboard terminals for the instrument sites, two printer/keyboard terminals, and a direct electrical connection analogue to digital conversion system for input of signals from the instrumentation. The principal programming language for the automation project will be multi-user (time-sharing) extended Basic. At present the Lawrence Livermore Laboratory of the AEC is writing machine language "patches" for the basic operating system. This will allow real time data acquisition and control of laboratory instrumenation from the high level Basic language. This work is being done under an interagency agreement with EPA. The computer system is expected to be delivered to the Livermore Laboratory this fall. It will undergo extensive testing and some modifications of hardware. The final debugging of the computer programs is expected by the end of the year when it will be shipped to MDQARL. Final testing and extensive tune-up will be completed at MDQARL with typical water quality instrumentation and environmental samples. Data on accuracy, precision, cost savings, sensitivity, and quality control concepts will be acquired. The major goal of the project is to develop methodology for application in Regional and other EPA water monitoring laboratories. Very significant development cost savings are expected for EPA laboratories that follow the same basic design as MDQARL. The instrumentation chosen for automation was selected as typical of non-computerized equipment that is widely used in EPA water laboratories. The computer

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system selected is sufficient to allow the addition of other instruments in future years. Computerization at MDQARL will substantially improve its ability to develop superior analytical methodology by permitting measurements that are not possible with non-computerized instrumentation. (J. Teuschler/W. L. Budde, 513-684-2918)

Benzidine Analysis

An improved version of the colorimetric method based on oxidation of benzidine with Chloramine T has been developed. The detection limit of the method is 0.18 ppb. At 1.8 ppb the recovery from dosed river water and sewage was only 70% but the standard deviation was an excellent 5%. (J. E. Longbottom, 513-684-2911)

Oil and Grease Analysis

Revised gravimetric methods and a new infrared method for oil and grease have been prepared for the revised EPA Methods Manual to be released this fall. Freon is used exclusively as the extracting solvent for these methods. An extension of these methods, involving the addition of silica gel to Freon solutions of oil and grease is recommended for removing edible oils and measuring the "hydrocarbon" content of the extract. (J. E. Longbottom, 513-684-2911)

Evaluation of a Continuous Cell Line for Enteric Viruses

Evaluations have been completed on a continuous cell line derived from primary African green monkey kidney cells. We have demonstrated through comparative titrations that this cell line, designated BGM, is more sensitive than primary Rhesus or African green monkey kidney cells to many enteroviruses and to reovirus 1, even if the viruses had previously been adapted to primary cells. In comparative studies with sewage, sewage effluents, river and ocean waters, we usually recovered many more viruses in BGM cultures than in primary Rhesus or in African green monkey kidney cultures. The availability of the BGM cell line could greatly reduce the need for monkey sacrifice and thus bring about substantial cost savings to research and clinical laboratories requiring such cells. (D. R. Dahling, 513-684-8274)

New Staff Member in Aquatic Biology Section

Donald Klemm recently joined the staff of the MDQARL Aquatic Biology Section, and will be in charge of macroinvertebrate methods development. Mr. Klemm is completing the requirements for a doctorate from the University of Michigan, Ann Arbor, and has authored several publications, including "Freshwater Leeches (Annelida:Hirudinea) of North America," Manual No. 8 of the EPA BIOTA OF FRESHWATER ECOSYSTEMS series.

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Current List of World-Wide Algal Virus Publications

The past decade has seen algal virology emerge from the speculative to the factual. The first definite evidence of phycoviral infection rose from an interest in underlying factors responsible for algal degeneration. Motivated by increased concern over eutrophication and associated algal blooms, attention has continued to be focused on the degree of control that phycoviruses could exercise over the fluctuations of algae in nature. A current bibliography of world-wide publications on phycoviruses has been compiled and is available for distribution. This "Bibliography--Phycoviruses" can be obtained from: Dr. Robert Safferman, EPA, National Environmental Research Center, Cincinnati, Ohio 45268. (R. S. Safferman, 513-684-8277)

New Report on Mayfly Taxonomy and Ecology

A manual on the taxonomy and ecology of the genus Stenonema is in press and will be available in the near future. The manual contains species descriptions, an illustrated species key, and information on the pollution tolerance of the organisms. (P. Lewis, 513-684-2980)

Test Methods for Membrane Filters

Two subcommittees of D-19 Technical Committee on Water, American Society for Testing and Materials, have been organized to develop standard test methods for (1) microbiological and (2) chemical and physical properties of filtration membranes. The subcommittee on microbiological properties composed of representatives from industry, universities and government agencies, has completed a preliminary round-robin test to determine the feasibility of proposed procedures for recovery of fecal coliforms and evaluation of inhibitory effects upon fecal coliforms. At the annual ASTM meeting in Washington, D. C., June 24, the subcommittee reviewed the round-robin results and decided to prepare the recovery test for vote and to further develop the test for inhibition. The subcommittee on chemical and physical properties is developing standard test methods for flow rate, porosity and pore size. Additional test proposals and results will be reviewed at the next meeting in January 1975. (R. H. Bordner, 513-684-2928)

New Edition of Chemical Methods Manual

The EPA "Methods for Chemical Analysis of Water and Wastes, 1971" is being revised. This second edition is the result of input by several Federal and State laboratories, AQC Coordinators, the staff of the Physical and Chemical Methods Branch of MDQARL, and constructive criticism by many analysts as well as new developments in analytical chemistry. It will contain a number of revisions as well as new analytical methods. Delivery of the manual is expected by October 1974. All persons on the

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Newsletter mailing list will be sent a copy of the manual. Additional copies will be available from Regional AQC coordinators and MDQARL.

Onsite Evaluation of EPA Facilities

As part of the1EPA interlaboratory quality control program senior staff members of MDQARL participated in the first of a series of onsite evaluations of EPA Regional laboratory facilities. The inspection team consisted of air, water, pesticides, and where necessary, radiation-oriented personnel from NERC-Cincinnati, NERC-Research Triangle Park, NERC-Las Vegas, and the Quality Assurance Division, Office of Monitoring Systems, EPA Headquarters. In the last quarter of FY 74, visits were made to the Alameda Facility, Region IX, and to the Surveillance and Analysis Laboratory at Athens, Region IV. Reports are in preparation for the involved laboratories and Office of Research and Development. During FY 75, visits are planned to the laboratory facilities in the remaining eight Regions. (J. A. Winter, 513-684-2983)

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