ALASKA LEGISLATURE COMMITTEE FILES 1983-1984 8672

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Does state have flexibility in

SAMPLE

SAMPLE WORK PLAN: REMEDIAL INVESTIGATION
(Sample work plans for all remedial/removal activities are under development.)

Activity: REMEDIAL INVESTIGATION   Project(s): (Name of Site)   Activity Number: I

Workyears: ______   Funds(x10^3): ______

who does work?

reimbursement?

Task No.	Description of Planned Tasks (examples)	Output	Schedule
	Comprehensively determine the nature and extent of the problem and gather data to support conceptual designs and cost estimates of alternative remedial actions.	Report	Target Date:
I-1.	Develop safety manual for Remedial Investigation tasks.	Manual	By:(date)
I-2.	Conduct appropriate community relations activities.	Several	By:(date)
I-3.	Conduct geophysical investigations.	Survey	By:(date)
I-4.	Conduct geological and hydrogeological investigations.	Investigations	By:(date)
I-5.	Conduct soil sampling and analysis.	Results of analysis	By:(date)
I-6.	Conduct hazardous waste characterization.	Survey	By:(date)
I-7.	Conduct ground water monitoring, sampling and analysis.	Results of analysis	By:(date)
I-8.	Conduct surface water monitoring, sampling and analysis.	Results of analysis	By:(date)
I-9.	Conduct air monitoring, sampling, and analysis (stability and security of site).	Results of analysis	By:(date)
I-10.	Identify physical site condition (stability and security).	Report	By:(date)
I-11.	Identify critical environmental area (flood plain, etc.).	Report	By:(date)
I-12.	Determine pathways of contamination.	Report	By:(date)
I-13.	Submit draft report to EPA for review and approval.	Draft Report	By:(date)
I-14.	Submit final report (incorporating all comments) to EPA for approval.	Final Report	By:(date)

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SAMPLE

SAMPLE WORK PLAN: COMMUNITY RELATIONS
(Sample work plans for all remedial/removal activities are under development.)

Activity: Community Relations

Project(s): (Name of Site)

Activity Number: II

Workyears: ______

Funds($x10^3$): ______

Task No.	Description of Planned Tasks (examples)	Output	Schedule
II.	Conduct community relations activities to ensure that actions taken at (site) are undertaken with the support of the local public	Plan	Target Date:
II-1.	Analyze level of citizen concern and identify constituency	Report & mailing lis	By:(date)
II-1-a	Interviews		
II-1 b	Review previous press coverage		
II-1-c	Review information on history of site		
II-2.	Develop fact sheets about the site	Fact sheets	By:(date)
II-3.	Conduct citizen's advisory committee meetings to coincide with major activities at the site	Meetings	By:(date)
II-4.	Prepare newsletters on progress and results to coincide with major activities at the site	Newsletters	By:(date)
II-5.	Press conferences	Press conferences	By:(date)
II-6.	Workshops for concerned citizens	Workshop materials	By:(date)
II-7.	Public meetings	Meetings	Before remedy is selected and before construction begins.
II-8.	Responsiveness summaries for all meetings/hearings	Report	At conclusion of remedial action.

5. Access to the Site and Permits

The State must secure the necessary rights-of-way, easements, or other authorities necessary to allow project activities to be carried out at the site as specified by the Statement of Work, and all permits required by State or Federal statutes to conduct project activities at the site.

6. Off-Site Treatment, Storage, or Disposal Facilities

The State must when EPA determines it to be necessary, assure the availability of an off-site treatment, storage, or disposal facility acceptable to EPA and in compliance with the requirements of Subtitle C of the Solid Waste Disposal Act, as amended.

7. Safety Plans

The State needs safety plans for each work plan activity (e.g., field investigation, feasibility study, design, and construction) to protect the health and safety of personnel involved in response actions. The State safety plan must be consistent with:

- Section 111(c)(6) of CERCLA.
- EPA Order 1440.2 -- Health and Safety Requirements for Employees Engaged in Field Activities.
- EPA Order 1440.1 -- Respiratory Protection.
- EPA Occupational Health and Safety Manual.
- Other EPA guidance as provided.

8. National Environmental Policy Act (NEPA)

The requirements of NEPA must be addressed through project activities described in the Statement of Work. Interim guidance and compliance with NEPA dated May 18, 1981, should be followed.

9. Community Relations Plan

A Community Relations Plan should be developed for and conducted concurrently with the response action. Plans should be consistent with interim program guidance on Community Relations Plans.

10. Laboratory Quality Assurance

States choosing to assume full responsibility for sample analysis must assure EPA that they can provide high quality in-house analytical services, capable of producing scientifically and legally defensible results. Except in cases where enforcement actions are not contemplated because a financially viable, responsible party cannot be identified, the following areas should be covered in preapplication negotiations:

- Intact chain of custody procedures;
- Adequacy of laboratory facilities to perform the work;
- An existing quality assurance/quality control (QA/QC) program;
- Participation in EPA laboratory intercomparison study and data audits;
- Adequate use of blanks, duplicates, spikes, and surrogates; and
- Use of "in process" controls to alert laboratory personnel to internal problems.

EPA's Hazardous Response Support Division, in cooperation with the Environmental Monitoring System Laboratory in Las Vegas, is developing alternative approaches to obtaining good analytical results. Guidance on using these approaches is forthcoming.

States lacking adequate analytical expertise or laboratory equipment may use EPA's existing National Analytical Contract through their Regional Project Officers. If this option is used, details for obtaining such services should be included in the Cooperative Agreement. (Regional Surveillance and Analysis Divisions are responsible for coordinating the use of the National Analytical Contract.)

11. <u>Evaluation of Potential for Response by Others</u>

Identifying and evaluating a potential for response by others are activities that the State and/or EPA may conduct. Interim guidance on this issue is being prepared by the Policy and Planning Unite of the Office of Waste Programs Enforcement.

12. Cost-Sharing Assurances

As part of the Cooperative Agreement the State must provide the cost-sharing assurances required in Section 104(c)(3) of CERCLA. The State should specify how it intends to satisfy these requirements and provide a schedule of actions that need to be accomplished. These assurances may be written as a Special Condition in the Superfund Cooperative Agreement.

The State must provide a firm commitment to contribute, or make available when needed, its share of the cost of the project (10% or 50%) before the final design activity begins. However, the State's requirement to contribute begins at the time of construction. This commitment should be made to the awarding official by the official who has the authority to guarantee that the State's contribution (including costs for all future operation and maintenance) will be provided before construction begins. Regional counsel should be consulted to determine what constitutes a firm commitment (for example, a letter from the Governor, State Attorney General, or other appropriate official). Timing of agreements, assurances, and commitments is displayed in the table at the end of Section V.

The methods the State use to meet the cost-sharing commitments will vary. Acceptable methods may include "up front" cash payments, certain in-kind services, the credit from Section 104(c)(3)(C)(ii), or a firm intent (with schedule and milestones) to seek funds. (See Section IV.)

a. Privately Owned Sites

When the site was privately owned at the time of disposal of hazardous substances and the Statement of Work includes final design and construction activities, the State must assure the following cost-sharing provisions of Section 104(c)(3) to EPA before the design activity commences:

- The State must agree to contribute (10%) of the cost of remedial action, including operation and maintenance (O&M) at the site.
- The State must agree to operate and maintain the response action for the expected life of that action as designated in the Statement of Work.

b. Publicly Owned Sites

When the site was publicly owned at the time of disposal of hazardous substance, the state must assure the following cost-sharing provisions of Section 104(c)(3) to EPA before EPA can approve any response actions.

- The State must agree to contribute at least (50%) of the cost of all response actions, including O&M, at the site.
- The State must agree to operate and maintain the response for the expected life of that response as designated in the Statement of Work.

13. Operation and Maintenance (O&M)

The Cooperative Agreement should specify if possible the terms and conditions for annual funding of)&M costs by the EPA and the State. The State and EPA should mutually agree on the minimum administrative and operational mechanisms that will be required during O&M. The Cooperative Agreement should include a schedule of milestone dates for implementation of the O&M process, and assurances for the following State requirements:

- Identification of the State's financial mechanism for annual funding O&M activities (for example, from special user fess, State or local taxes, court judgments, general revenues, bonds, special appropriations, or legislative authority).

- Identification of the organizational unit responsible for administering O&M activities. This may be an existing State or local agency or a special authority set up for this purpose (sewer or water districts, for example).

Interim guidance on how to caluculate annual estimates of O&M costs will be provided at a later date.

D. Completing the Assistance Application

In addition to EPA form 5700-33, the applicant must include a Statement of Work comprised of a work plan narrative and the work plan itself. As many of the major Superfund issues (described in C above) as possible should be incorporated in the Statement of Work; the rest should be included as Special Conditions.

E. Submitting the Completed Application

The State must submit the completed application simultaneously to the clearinghouses and the Regional Office for review. If the State notified the clearinghouses of its intent to seek assistance from the EPA, final clearinghouse reviews can be facilitated.

F. EPA Review of the Application

The Regional staff reviews the application for technical quality and completeness, and submits it to the award offical with a recommendation for funding.

G. Executing the Cooperative Agreement

The award official will execute a Superfund Cooperative Agreement if:

- The Cooperative Agreement is complete and technically satisfactory.

- The award official determines that the State is capable of accomplishing the activities specififed in the application.

- Fund monies are available.

Requirements for transferring funds to the State via letter of credit are applicable to Superfund Cooperative Agreements.

H. Special Conditions

For each State, depending on the problems at the site, some programmatic special conditions may also be required. As stated in this Section, Superfund issues that are not incorporated into the work plan may be included as Special Conditions.

1. Interim Progress Reports

Quarterly progress reports should include the following information for each site activity covered by the Cooperative Agreement:

- Expenditures to date and expenditures since the previous report.
- Estimates of work completed (as a percentage of the total work to be done on that activity), with a description of the basis for the estimates.
- Estimated variance (cost and time) expected at project completion, based on current project status.

2. Expenditures

Section II-H discusses expenditures. However, the exigencies of Fund management may require a review of all Superfund Cooperative Agreements more frequently than quarterly. To ensure consistency in reporting, the state should report quarterly its total expenditures incurred at each site. Expenditures against each Cooperative Agreement should be itemized within the categories identified on the Federal Cash Transactions Report (form SF-272) which is used by States operating on a letter of credit. In addition, the quarterly report should itemize the following expenditures:

- Personnel
- Fringe Benefits
- Travel
- Equipment
- Supplies
- Contracts
- Construction
- Direct Charges
- Indirect Charges
- Total Charges

Between the required performance reporting dates, the State shall promptly notify the Project Officer of any events, such as project overruns and underruns, having significant impact on the project.

I. Amendments

Administrative or formal amendments are likely to be required as each activity is completed and more is learned about conditions on the site. For example, if the initial activity covered by the Cooperative Agreement is the feasibility study, amendments typically will be needed prior to design and prior to construction. The procedures described in Section II-H should be followed when amending Superfund Cooperative Agreements.

J. Conclusion

The following figure displays the Superfund Cooperative Agreement process discussed in this section.

(actual Time line)

APPLICATION APPROVAL PROCESS FOR SUPERFUND COOPERATIVE AGREEMENTS

Sequence of Events	Actions Required By		
	EPA	STATE	EPA/STATE
Preapplication Phase			
1 - EPA ranks sites: o Interim priority list o National priority list	X		
2 - STATE notifies Clearinghouse(s) of intent to seek assistance: o Form 5700-30 o Summary description of project		X	
3 - EPA allocates funds for response actions at ranked sites.	X		
4 - EPA/STATE negotiate Statement of Work (including Superfund requirements): o Work plan narrative o Work plan o Schedules o Budgets o Use of State employees and equipment or o Subagreement/contract.			X
Review Phase			
5 - STATE: o Prepares assistance application package - Form 5700-33 - Statement of Work o Designates project manager.		X	

	EPA	STATE	EPA/STATE
Review Phase (con't)			
6 - STATE forwards completed application package to EPA and Clearinghouses.		X	
7 - EPA: o Reviews State assistance application for - Technical quality - Completeness o Evaluates State's capability to award and direct contracts.	X		
8 - STATE forwards Clearinghouse review(s) to EPA.		X	
9 - EPA decides: o To fund (must have clearinghouse review) or o Not to fund - STOP-	X		
10 - EPA prepares (if project to be funded): o Grant Funding Order o Commitment notice o Special conditions	X		
Award Phase			
11 - EPA: o Executes award -- Form 5700-20A -- Special Conditions o Designates project officer o Sends award to State	X		
12 - STATE: o Accepts terms and conditions o Signs agreement (Form 5700-20A) o Sends signed agreement to EPA		X	
13 - EPA transfers funds to State via letter-of-credit	X		

	EPA	STATE	EPA/STATE
Action Phase			
14 - STATE: o Negotiates subagreements o Lets contracts o Begins activities authorized in Statement of Work		X	
15 - EPA/STATE review, concur, approve outputs in work plan through completion of project.			X
16 - STATE submits quarterly reports: o Progress at site o Expenditures		X	
17 - EPA reviews quarterly reports to ensure consistency with Statement of Work.	X		
18 - STATE submits final report for budget period.		X	
19 - EPA reviews and accepts final report.	X		
20 - EPA/STATE: o Closeout or o Amend Cooperative Agreement			X

IV. STATE COSTS ELIGIBLE FOR PAYMENT BY THE FUND

A. Eligible State Costs

Eligible State costs are those that are authorized by Section 111 of CERCLA.

State costs paid from the Fund must also be:

- Directly allocable to a particular response;
- Reasonable;
- Within the scope of the response action and necessary for the accomplishment of the response; and
- Allowable in accordance with OMB Circular A-87 and EPA Policy.

Cost incurred by a State in carrying out the following activities may be paid by the fund if authorized in a Cooperative Agreement. For example, under a Cooperative Agreement, activities that have been mutually agreed upon would be eligible under CERCLA, but not all costs incurred in executing the Cooperative Agreement would be allowable. The technical preparation of a contract scope of work and the technical management of that contract would be allowable for the following eligible activities:

- Remedial Investigation
- Feasibility Study
- Final Remedial Design
- Remedial Implementation
- Operation and Maintenance
- Enforcement/Litigation
- Planned Removal

B. Direct Costs

Costs incurred in carrying out an activity at a site approved via a Cooperative Agreement are listed below. In all cases, costs must be eligible according to CERCLA and allowable, allocable, and reasonable in accordance with OMB Circular A-87 and EPA general policy.

1. Contract Services

When a State is managing a response action using subagreements, the Cooperative Agreement may cover the costs of the time (including base salary) a State employee devotes to functioning as Project Officer conducting technical review and managing a contract. The Cooperative Agreement may include necessry travel associated with carrying out a response action, as well as the costs the Project Officer incurs in preparing technical contract specifications in the Request for Proposal (RFP) or directive of work. Other costs not directly part of preparing the technical contract specification or of directing, reviewing, and managing the contract -- such as the costs of nontechnical aspects of procurement, maintenance of the State accounting system, and costs of staff supervising the Project Officer -- are covered as indirect costs subject to the requirements of OMB Circular A-87. These costs may not be charged as direct costs.

2. Response by State Employees

Where a State is taking a response action using State employees, the Cooperative Agreement may cover the costs of the time State employees devote to working directly on the response action and the portion of the time their first-level supervisor devotes on the scene to managing their work. The Cooperative Agreement may include necessary travel of the Project Officer. Other costs, such as second-level supervisory time, payroll, and accounting, are covered as indirect costs and are subject to the requirements of OMB Circular A-87 and may not be charged as direct costs.

3. Enforcement

Where a State is providing enforcement actions, the Cooperative Agreement may cover the time a State attorney or technical staff member devotes directly to the case and any litigation costs directly allocable to the enforcement case. This includes expert witnesses and court costs. The Cooperative Agreement may also include all necessary travel costs of staff working directly on the case. Supervisory time, accounting, etc. are covered as indirect costs subject to the requirements of OMB Circular A-87.

4. Materials and Supplies

Where a State is providing material and supplies, the Cooperative Agreement may cover the direct cost and materials and supplies necessary for carrying out the response action.

5. Equipment

Where the State is providing equipment, the Cooperative Agreement may cover the direct costs of equipment (subtracted from its residual value) necessary for carrying out the response action.

C. Indirect Costs

Indirect costs are those costs:

- Incurred for a common or joint purpose benefiting more than one cost objective; and
- Not readily assignable to the cost objective specifically benefited, without effort disproportionate to the results achieved.

The Fund will pay indirect State costs computed according to the principles in OMB Circular A-87 for determining the allowable indirect costs of programs administered by State or local governments. The principles are designed to provide that Federally assisted programs bear their fair share of direct and indirect costs.

As part of negotiating the Cooperative Agreement, EPA and the State will develop a fixed rate for computing indirect costs relative to the direct cost base consistent with the requirements of OMB Circular A-87.

D. Documentation of Direct Costs

For all direct costs, various logs, records, and files should be maintained by the State to establish an audit trail. The trail should begin with the site-specific financial documents such as contractor vouchers, lead to standard quarterly reports to EPA, and continue up to and through the project period of the Cooperative Agreement. Such documentation (for example, project logs, purchasing and contracting files, methods of equipment useage and/or depreciation, and periodic reconciliation of such records) should cover expenses incurred at the site from January 1978, to enactment of CERCLA (December 11, 1980). The following paragraphs describe in more detail the documentation required for different categories of costs.

1. Personnel

Compensation for employees directly employed on a response action or enforcement case may be included as a direct cost. Compensation includes all remuneration, paid currently or accrued, for services rendered during the period of performance under the Cooperative Agreement. Compensation includes wages and salaries.

State employees assigned to Superfund projects should be classified and paid according to the prescribed State personnel structure and procedures. Similarly, time records or other project documentation prepared at the time of the actual work and verified by the supervisor should establish clearly that the employees claimed under the project actually worked on the project for this time. Furthermore, the State payroll system should be capable of tracking the amount of employee's time spent on activities covered by the Cooperative Agreement, as well as on other projects, thus separating allowable personnel costs.

2. Contract Services

To properly document costs of contract service, a State agency should maintain a file of procurement requests and proposals to show that it properly procured the services at reasonable prices. For example, records should be available to show that procurement was made in accordance with established State procedures, that costs were claimed in accordance with contract provisions, and that contractors were paid.

In addition, contractor vouchers or similar documents should identify the specific site-by-site work performed and should enumerate personnel hours and travel incurred, materials and supplies consumed, and equipment used site-by-site.

3. Travel

Allowable travel and per diem of State employees directly employed on a response action should be recorded by site or by enforcement case on the document enabling the travel. Such documents include, for example, car rental receipts, preauthorized travel vouchers, per diem requests for State employees, requests for reimbursement of car mileage, and out-of-pocket expenses such as tolls or lodging.

4. Materials

The State should maintain inventory logs indicating when the materials were ordered and procured, and project logs recording when the materials were delivered and used on the site. Purchasing records should justify that the costs were charged based on actual prices less all discounts, rebates, and allowances. Withdrawals from general stores or stockrooms should be charged according to recognized methods of transfer pricing or inventory accounting -- such as first in first out (FIFO) or average cost -- if the cost is different from actual procurement cost.

5. Equipment

To properly document equipment costs, State purchasing logs must record when the equipment was procured and the cost paid in accordance with State procurement standards. The project log should indicate when the equipment was used on the project. If equipment costs are based on usage rates, the State should use a standard depreciation or usage method and document the initial costs, expected life, and residual value of the equipment. (See OMB Circular A-102, Attachment N).

6. Reconciliation

The State should have an overall accounting method and system for reconciling the records noted above. Such a method should reconcile tracking of payroll and travel; purchasing of equipment and materials and supplies; and reporting of contract-related costs (including requests for funds from the EPA). Such an accounting system should meet the specifications included in the requirements for approval of the letter-of-credit financing mechanism.

7. Project Close-Out

Close-out should be consistent with instructions in EPA Letter of Credit Users Manual, which require a final report on expenditures and disposition of equipment.

V. STATE AGREEMENTS

A State agreement is developed when EPA has the lead responsibility for managing a response action. This type of agreement is a tool to exchange information, coordinate decision-making with key State and local officals, and integrate Federal and State programs to the maximum extent possible. The goal is to facilitate eventual delegation of Superfund responsibilities to the States.

A State agreement is appropriate when:

- There is no Cooperative Agreement.
- The State does not have the technical, legal, or administrative capabilities to manage Superfund activities.
- The State prefers not to assume site management responsibilities.

A. EPA Responsibilities in a State Agreement

When EPA has the lead responsibility for managing a response action, it will provide overall management and direction for activities at a site usually via one of its architectural and engineering (A&E) zone contracts. At the minimum, EPA will:

- Ensure that the proper contracting mechanism is put into effect and can be responsible for coordinating all contractual matters relating to the project.
- Desingate a Regional Project Officer who will provide overall coordination and management of the activities covered by the agreement.
- Coordinate the development and implementation of the Statement of Work and associated schedule.
- Develop necessary cost estimates.
- Develop and implement a Community Relations Plan.
- Coordinate enforcement efforts and issue demand letters when appropriate.
- Manage daily operations to the extent necessary and seek advice of the Regional Response Team (RRT) and State at key decision points.
- Obtain any Federal permits that may be required to facilitate the response action.

B. State Responsibilities in a State Agreement

A State agreement may be used to cover EPA activities up to and including conceptual design of a response action. The State must enter into a Cooperative Agreement when it is required to contribute a share of the costs of the response action and desires to use in-kind services as part of its contribution.

Activities covered by the State agreement must be consistent with the NCP, and the State should participate fully in deliberations of the RRT. At the minimum, the State should agree to:

- Designate a State Project Coordinator who will serve as the State's representative throughout the project and provide the coordination and direction desired by the State.

- Participate in the development and implementation of Statements of Work and project schedules.

- Review progress at the site with EPA as activities are performed.

- Participate in development and implementation of the Community Relations Plan and assist in coordinating the activities covered by the plan with other State and local officials.

- Provide access to necessary file information.

- Ensure inter-departmental coordination.

- Provide legal capabilities necessary to ensure site access and acquire any necessary State permits.

These requirements apply to all sites. Some requirements vary according to ownership of the site. When:

- A site was privately owned at the time of disposal of hazardous substances, the State Agreement must describe how the State will assure that it will contribute the required 10% of the cost of remedial action, including O&M.

- A site was publicly owned at the time of disposal of hazardous substances, the State agreement must describe how the State will assure that it will contribute the required 50% of all response actions, including O&M.

See the attached chart for timing of agreements and assurances.

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STATE AGREEMENTS AND ASSURANCES

SITE RESPONSE PHASES	STATE LEAD		EPA LEAD	
	Privately Owned Sites	Publicly Owned Sites	Privately Owned Sites	Publicly Owned Sites
Requirements before RESPONSE ACTIONS Other Than Planned Removals (e.g., Remedial Investigation, Feasibility Study) (State acknowledges Requirement)	o Must have Cooperative Agreement o State assures: - 10% share of remedial costs including O&M - off-site disposal.	o Must have Cooperative Agreement o State assures: -50% share of all response costs and O&M -off-site disposal. -State contributes annual share of response cost	o Must have State Agreement o State assures: - 10% share of remedial costs including O&M. - off-site disposal.	o Must have State Agreement o State assures: -50% share of costs including O&M -off-site disposal -State contributes annual share of response cost
Requirements before Starting FINAL DESIGN (State makes firm commitment) i.e letter from Gov, AG, Legislative Comm	o State provides firm commitment of 10% share of remedial costs and O&M for operable units.	o State provides firm commitment of 50% share of remedial cost and O&M for operable units o State contributes 50% share of all response costs (design)	o State provides firm commitment of 10% share of remedial costs and O&M for operable units	o State provides firm commitment of 50% share of remedial cost and O&M for operable units o State contributes 50% share of all response cos
Requirements before Starting CONSTRUCTION	o State contributes annual share of implementation cost for operable units ?	o State contributes annual share of all remedial costs for operable units	o State contributes annual share of implementation costs for operable units.	o State contributes annual share of all remedial costs for operable units

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Requirements before Starting O&M	o State contributes annual share of O&M costs.	o State contributes annual share of O&M costs.	o Must have Cooperative Agreement. o State contributes annual share of O&M costs.	o Must have Cooperative Agreement. o State contributes annual share of O&M costs.
Requirements before Starting PLANNED REMOVAL (when applicable)	o Must have Cooperative Agreement when: -$1 million or 6 months expires and -there is O&M (State contributes 10% share) (Planned removal action must be consistent with remedial action)	o Must have Cooperative Agreement. o State contributes 50% share of all response costs; including O&M. (Planned removal action must be consistent with remedial action)	o May have State Agreement. o Must have Cooperative Agreement when: -$1 million or 6 months expires and -there is O&M (State contributes 10% share) (Planned removal action must be consistent with remedial action)	o May have State Agreeme o Must have Cooperative Agreement if there is O&M. o State contributes 50% share of cost of all response costs, including O&M. (Planned removal action must be consistent with remedial action)

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VII GLOSSARY OF TERMS

To assist users of this guidance document, some terms are defined here. The definitions may differ slightly from those used in other guidance documents issued by the Office of Emergency and Remedial Response. These differences will be resolved as implementation of CERCLA proceeds and the revised National Contingency Plan is published.

Response action: A broad term covering immediate removal, planned removal, and remedial actions taken under CERCLA in response to a release. Restoration or rehabilitation of natural resources is not covered.

Immediate removal: An emergency response required within hours or days to deal with an actual or potential significant threat to human health, the environment, or real or personal off-site property. An immediate removal is meant to stablize the situation and to avert the threat of immediate harm.

Planned removal: A response that may allow several days or weeks for planning but still requires expeditious attention to reduce imminent and substantial dangers to public health or the environment. Planned removals may involve complete cleanup or measures that may be part of a subsequent remedial action.

Remedial action: An action intended to provide a permanent resolution to the release. The action probably will require a longer time and possibly more expensive efforts to implement. A remedial action seeks to prevent or minimize the release or threatened release of hazardous substances so that they do not migrate to cause substantial danger to present or future public health, welfare, and the environment.

Remedial planning: An early phase of a remedial response action, which may include remedial investigations, feasibility studies, and final remedial design.

Remedial investigation: An investigation intended to gather the data necessary to 1) determine the nature and extent of problems at the site; 2) establish cleanup criteria for the site; 3) identify preliminary alternative remedial actions; and 4) support the technical and cost analyses of the alternatives.

Feasibility study: A study intended to 1) evaluate alternative remedial actions from a technical, environmental, and cost-effectiveness prespective; 2) recommend the cost-effective remedial action; and 3) prepare a conceptual design, cost estimate for budgetary purposes, and a preliminary construction schedule.

Final Remedial Design: A phase of a remedial action where the selected remedy is clearly defined (that is, site cleanup plan, relocation plan, or engineering drawings and specificactions) in a bid package so that the remedy can be implemented in the field.

OCT 30 1981

MEMORANDUM

TO: Legislators and staff interested in Hazardous Waste Management

FROM: Donna Wise, Staff Director for the NCSL Natural Resources and Environment Committee

DATE: October 28, 1981

SUBJECT: Announcement by U.S. EPA, of the 114 top priority hazardous waste sites to be addressed with SUPERFUND monies

The attached EPA announcement, lists those 114 hazardous wastes sites in the nation which will receive attention under SUPERFUND. By law EPA must develop list of 400 priority sites, with at least one site per state falling within the top 100 to the extent practicable. The final list of 400 sites is expected to be released early in 1982.

When clean up efforts are initiated at a site, the state will be given a choice to either assume the lead responsibility for the clean up efforts, or to allow U.S. EPA to assume the major responsibility. No matter who assumes the primary responsibility, before any work can commence at a site, a state must assure EPA that it will provide 10% of the clean up and long term operation and maintenance costs if the site is privately owned, and 50% of the clean up and long term operation and maintenance costs if the site was publically owned at the time of disposal. These assurances and division of responsibility between the state and U.S. EPA will be formalized by the signing of a state/EPA cooperative agreement.

At the next NCSL State/Federal Assembly meeting in Washington, D.C. on December 9-11, members of the Natural Resources and Environment Committee will receive a briefing by U.S. EPA officials on their plans for hazardous waste site clean up under SUPERFUND.

To: Donna Wise
NCSL

United States
Environmental Protection
Agency

Press Office (A-184)
Washington DC 20460

EPA

Environmental News

FOR RELEASE FRIDAY, OCTOBER 23, 1981

EPA ANNOUNCES FIRST 114 TOP-PRIORITY SUPERFUND SITES

Woods (202) 755-0344

The U.S. Environmental Protection Agency today announced 114 top-priority hazardous waste sites targeted for action under Superfund, a five-year, $1.6 billion federal cleanup program.

"This is a milestone in the development and implementation of Superfund," said EPA Administrator Anne M. Gorsuch. "The list we have developed represents a program to which we have given the highest priority, and one we are determined to make successful."

Mrs. Gorsuch said the Reagan Administration is "committed to the cleanup of hazardous waste sites as quickly and effectively as possible."

Superfund is the name given to the Comprehensive Environmental Response, Compensation, and Liability Act that was passed by Congress in December 1980.

It provides funds from industry and the federal government to clean up hazardous waste sites where responsible parties cannot be determined or cannot afford to pay for cleanup.

(more)

The ranking of sites was based on a hazard-scoring system developed by EPA and one of its contractors, with extensive input from states and industry. The greatest emphasis was on potential threat to public health, but the threat to the environment was also taken into account.

Pollution via three "pathways' -- air, groundwater and surface water -- was measured for potential impacts. Fire, explosions, and the possibility of direct contact received separate evaluation as more appropriate for emergency action.

In some cases, EPA authorized an emergency removal action based on information uncovered during the hazard-scoring process.

The list of 114 sites was developed from an initial list of 282 sites evaluated by the states and EPA's 10 regional offices this summer. The final quality-assurance phase of the process was conducted by EPA headquarters over the past 1-1/2 months.

The sites announced today will be candidates for inclusion on the list of 400 national priority "response targets" that the Superfund law required EPA to identify.

That list will be made final after public participation and after the results of further study and data collection are incorporated into the hazard scoring.

Under Superfund, states must contribute at least 10 percent of the actual long-term costs of cleanup per

(more)

site, unless the site is publicly owned. On publicly owned sites, the state is required to pay or assure at least 50 percent of the costs.

Detailed plans for cleanup will be worked out in conjunction with the states. Cleanup can occur through three mechanisms: direct federal contracts; cooperative agreements under which the state takes the lead in directing cleanup, and private cleanup through voluntary or court-ordered action.

"The Agency will continue to press responsible parties -- through legal action, if necessary -- to clean up sites threatening public health or the environment," Mrs. Gorsuch said. "Where this cannot be done, or if it cannot be accomplished in a timely manner, EPA and the states will finance remedial action under Superfund."

To date, EPA has spent some $13 million in Superfund money on emergency action for 34 sites in 18 states. It has also awarded $17 million for design and engineering studies on 25 other sites in 19 states, almost all of which appear on the list announced today.

# # #

Priority List Facts

- 44 States and territories have sites on the list.

- 11 States and territories do not have a site on the list.

- The State with the most sites on the list is Florida with 16; next is New Jersey with 12.

- Other States with more than 1 site are:

  New York(8)
  Pennsylvania (8)
  Minnesota (5)
  Massachusetts, Texas, Arkansas, Ohio (4)
  California, New Mexico, New Hampshire, Rhode Island, Delaware (3)
  Virginia, Oklahoma, Indiana, Missouri (2)

- 26 States or territories have just 1 site on the list.

- 37 States designated a site on the list as their highest priority list.

- 18 of the 20 pre-Superfund sites are on the priority list.

- EPA is currently spending Superfund and other dollars for remedial planning and design at 25 sites, 21 of which are on the priority list.

- Of 31 sites at which EPA has conducted emergency removals, 8 are on the priority list.

State	Site Name
WA	Commencement Bay
NH	Keefe Environmental Services
NJ	Lipari Landfill
MA	Mark Phillip Trust (Woburn)
PA	McAdoo Associates
MA	Nyanza Chemical Waste Dump
NY	Pollution Abatement Services
NJ	Price Landfill
OK	Tar Creek
DE	Tybouts Corners
FL	Biscayne Aquifer (Northwest 58th Street Landfill, Miami Drum, Varsol Spill)
PA	Bruin Lagoon
NJ	Burnt Fly Bog
DE	Delaware Sand and Gravel - Llangollen Army Creek Landfill
NJ	Goose Farm
NJ	Lone Pine Landfill
TX	Motco
NJ	Pijack Farm
NJ	Spence Farm
AR	Vertac, Inc.
NJ	Bridgeport Rental and Oil Services
NJ	D'Imperio Property
TX	French Limited Disposal Site
NY	Love Canal
NY	Old Bethpage Landfill
FL	Picketville Road Landfill
FL	Reeves Southeastern Corporation
IN	Seymour Recycling Corporation
TX	Sikes Disposal Pits
SC	South Carolina Recycling and Disposal Co. (Bluff Road)
CA	Aerojet/General Corporation
FL	American Creosote Works
MA	Charles George Land Reclamation Trust
CA	Iron Mountain Mines, Inc.
NJ	Kin Buc Landfill
MN	Oakdale Dump Sites
NY	Olean Well Fields
RI	Picillo Farm Site
DE	Stauffer Chemical
FL	Taylor Road Landfill

State	Site Name
MN	Andover Sites
FL	Broward County Solid Waste Disposal Facility
PA	Butler Tunnel
NY	Facet Enterprises, Inc.
MO	Fulbright Landfill
NH	Ottati & Goss/Kingston Steel Drum
FL	Pioneer Sand Company
FL	Timber Lake Battery Disposal
FL	Whitehouse Waste Oil Pits
SD	Whitewood Creek
OH	Chem-Dyne Corporation
NJ	Chemical Control
FL	Coleman-Evans Wood Preserving Company
RI	Davis Liquid Chemical Waste Disposal Site
AR	Fritt Industries
FL	Hollingsworth Solderless Terminal Company
CT	Laurel Park Landfill
MA	Re-Solve, Inc.
MN	Reilly Tar and Chemical Corp.
CA	Stringfellow Acid Pits
AR	Allen Transformer
FL	Alpha Chemical Corp.
OH	Fields Brook
MN	Koppers Gas and Coke Plant
AR	Mid-South Wood Products
IN	Neal's Landfill
NM	United Nuclear Corporation
NJ	Upper Freehold
FL	Zellwood Ground Water Contamination Site
AZ	19th Avenue Landfill
KY	A.L. Taylor Site ("Valley of the Drums")
NY	Batavia Landfill
FL	Gold Coast Oil Corporation
NM	Homestake Mining
PA	Hranica Landfill
PA	Lord-Shope Landfill
MN	National Lead-Taracorp Site
IL	Outboard Marine Corporation
FL	Sapp Battery Salvage
FL	Tower Chemical Company

State	Site Name
PA	ABM-Wade
MO	Ellisville Area Sites
OH	Chemicals and Minerals Reclamation
MI	Gratiot County Landfill
PA	Lehigh Electric and Engineering Company
NY	Marathon Battery Corporation
VA	Mathews Electroplating
NH	Sylvester's
WV	West Virginia Ordnance Site
RI	Western Sand and Gravel Site
NM	AT&SF Railroad (Clovis)
TX	Bioecology Systems, Inc.
VA	Chisman Creek Disposal
OK	Criner Waste Disposal Site
CO	Denver Radium Sites
PA	Lindane Dump
NY	Niagara County Refuse Site
OH	Summit National Liquid Disposal Services
AL	Triana (Redstone Arsenal)
ME	Winthrop Town Landfill
IA	Aidex Corporation
KS	Arkansas City Dump Site
ND	Arsenic Trioxide Disposal Site
MD	Chemical Metals Industries, Inc.
DC	Fort Lincoln Barrel Site
GA	Luminous Processes, Inc.
TN	North Hollywood Dump
GU	Ordot Landfill
TTP	PCB Disposal
NC	PCB Spills in North Carolina
NMI	PCB Warehouse
UT	Rose Park
AS	Taputimu Farm
MS	Walcotte Chemical Company

National Conference of State Legislatures

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State
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President
William F. Passannante,
Speaker Pro Tempore,
New York Assembly

Executive Director
Earl S. Mackey

NATURAL RESOURCES & ENVIRONMENT COMMITTEE

LEGISLATIVE UPDATE

APRIL 15, 1983

(Revised April 22, 1983)

SUPERFUND

Congressional committees in both the House and the Senate are continuing investigations of U.S. EPA's administration of the Superfund Program. In a recent hearing before the Senate Environment and Public Works Committee, EPA personnel admitted that Mrs. Burford repeatedly refused to allow EPA to undertake emergency response actions due to budgetary concerns. EPA officials told committee members that recommendations would be made to Mr. Ruckleshaus for changes in program implementation.

Mr. William Hedeman, director of EPA's Superfund Program, told legislators and staff participating in a recent NCSL teleconference call, that EPA staff would recommend a number of policy changes to the new Administrator, including:

- abandoning the requirement that States provide 10% match for the feasibility and remedial investigation stage of site clean up. Thus states would only be required to match 10% of the site design and construction. EPA will also consider whether or not to provide a credit to those states which have already paid 10% of the first two stages.
- review the practicality of requiring states to pay 100% of the long term operation and maintenance costs at a site
- reviewing the lack of clean up standards in the National Contingency Plan --the "how clean is clean" issue
- reviewing the need for a more agressive EPA enforcement program
- reviewing the need for additional resources at EPA to implement the program in a timely and comprehensive fashion.

Mr. Hedeman also told teleconference participants that EPA will be publishing a handbook on alternative financing methods for states' 10% match. This handbook will be the subject of regional public meetings in the fall. (Watch for an announcement in a fall issue of <u>Capitol to Capitol</u>.)

Finally, Congress is likely to continue hearings this summer on the need for specific amendments to the Superfund Program and on the need for a victim's compensation fund. However, action on these measures is not expected until 1984.

CLEAN AIR ACT

After months of hearings last year in both Senate and House Committees on amendments to the Clean Air Act, to date in this session both houses have been silent. While it is unclear when and if hearings will be scheduled, proposals have been introduced in both the House and the Senate addressing acid rain.

These proposals range in nature from S.454 (Senator Byrd) and S.766 (Senator Randolph) which call for accelerated acid rain research to S.145 emmissions within a 31 state region east of the Mississippi over the next 10 years. Other proposals call for 8 million and 12 million ton reductions. Although not yet introduced, Representative Waxman is working on a bill which would mandate reduction nationwide thereby allocation cost equitably. As of this writing, no specific hearings have been scheduled on acid rain.

OIL SPILL LIABILITY FUND

For the fourth consecutive Congress, legislation has been introduced to create an oil spill liability and compensation fund. This central federal fund would pay for damages caused by oil spills and would establish one set of federal liability laws. The fund would be financed by a 1.3 per cent barrel tax on crude oil and petroleum products. Of major import to states is the fact the state oil spill funds would be preempted as would individual state liability schemes. States which took action to clean up oil spills would have to seek reimbursement from the federal fund. Three measures introduced in the House were the subject of hearings before the House Merchant Marine and Fisheries Committee's Subcommittee on Coast Guard and Navigation on Wednesday, April 12th. A similar measure was introduced in the Senate by Senator Chafee (RI) and referred to the Finance Committee.

U.S. DEPARTMENT OF INTERIOR'S COST SHARING PROPOSAL FOR WATER PROJECTS

While as of this writing the Administration has yet to announce a cost sharing policy for water resources projects, Gary Curruthers, Assistant Secretary for Land and Water Resources, U.S. DOT, recently told a group of state water resource personnel, that "future water resource projects will be viewed as business partnerships between the federal government and state and local sponsors. Within certain parameters (yet to be announced) water project cost sharing will be negotiated on a case by case basis. If state and local sponsors can meet the conditions of the contract, the business venture will proceed."

In late February of this year Gary Curruthers solicited comments on the cost sharing from legislative leaders, governors and the general public. To date approximately 18 responses have been received from legislatures and approximately and equal number from governors. A summary of those comments should be available in the near future.

CLEAN WATER ACT

The Senate Environment and Public Works in proceeding with hearings on the reauthorization of the Clean Water Act this week. A final hearing will be scheduled to allow Mr. Ruckleshaus to testify after he is confirmed as EPA Administrator. The measure under consideration is S.431 sponsored by Senator Chafee (RI). While maintaining the basic tenets of the Act, S.431 addresses a number of provisions in need of refinement. The deadlines by which industry would have to meet "best available technology" would be extended from July 1, 1984 to "three years from the date that BAT requirements were promulgated". Municipal sewage treatment works would be eligible for variance from federal pretreatment requirements if they could meet certain tests. S.431 would provide for administrative assessment of civil penalties for minor violation of the Act. Additionally the Chafee bill would extend the life of an NPDES permit from 5 years to 10 years with a reopener clause. The measure would also allow for partial delegation of the NPDES program to states. These amendments are in line with NCSL policy adopted in July 1982. To date a bill has not been introduced in the House, however, the House Public Works Committee has deemed reauthorization of the Clean Water Act a high priority.

TRANSPORTATION OF HAZARDOUS MATERIALS

On April 20th Senator Danforth (MO), Chairman of the Surface Transportation Subcommittee of the Senate Commerce Committee introduced the "Highway Safety Act of 1983" which includes as Title IV, "Hazardous Materials Transportation Act Amendments of 1983". Provisions of this title are generally in keeping with NCSL Policy adopted at the April 1983 State/Federal Assembly.

Specifically the legislation provides for: 1) DOT assistance in establishing regional training centers for enforcement and emergency response presonnel; 2) grants to states for the development and implementation of state programs to enforce hazardous materials regulations; 3) FEMA grants to state and local agencies for the development of hazardous materials emergency response programs.

Senate hearings will be held in Mid May. A companion bill HR 2693 has been introduced in the House by Congressman Lantos (D-CA).

RESOURCE CONSERVATION & RECOVERY ACT REAUTHORIZATION (Hazardous Waste Management)

The Resource Conservation & Recovery Act (RCRA) is the nations basic authority to assure the proper management of solid and hazardous waste. The Act authorizes U.S. EPA to regulate hazardous waste from its generation through its disposal. States are encouraged to seek program delegation from EPA based on state regulations and authority being as least astringent as federal regulations. States receive federal grants to administer their programs ($44 million in FY83).

Both the House and Senate are considering measures which reauthorize RCRA and would "close the loopholes" present in the current Act.

S.757 sponsored by Senator Chafee (RI) was referred to the Senate Environment and Public Works Committee. Hearing dates have not yet been scheduled. HR 2478 sponsored by Representative Florio (NJ) was referred to the House Energy and Commerce Committee on April 27th, the Subcommittee in Commerce, Transportation and Tourism favorably reported the bill to the full committee.

FY1984 BUDGET FOR U.S. EPA AND THE DEPARTMENT OF THE INTERIOR

While Congress has not completed its work to set spending levels for FY84, figures provide some assurance that environmental spending for FY84 will at least be held at the FY83 levels if not increased. Thus far the Senate Budget Committee has allocated $12 million in budget authority for natural resources and environment. This represents the $11.7 billion FY1983 level after inflation plus $300 million to accomodate an administration request for more money for the Superfund Program. While the Senate Budget committee may review this figure, it is expected to remain in a range between $11.7 - 12 billion. The House passed the first concurrent budget resolution with an FY84 level of 12.1 billion for natural resources and envrionment programs.

House appropriations hearings for both U.S. EPA and Interior are expected to continue through the end of this month, as are Senate appropriations hearings for U.S. Interior. EPA appropriations hearings in the Senate have been postponed until Mr. Ruckleshaus is confirmed.

STATE GRANTS FOR COASTAL RESOURCES MANAGEMENT

On the heals of the Administration's request to elimate funding for state coastal zone management programs and sea grant colleges, and with Administration plans to accelerate offshore oil and gas leasing, Congress has accelerated its consideration of measures to create a state block grant program for coastal resources management.

Such a program would provide coastal states, including Great Lake States, with funding derived from royalities from Outer Continental Shelf Oil and Gas leases. States could use these funds for coastal zone management activities, assessment and mitigation of OCS development impacts, and management and enhancement of natural and marine resources.

In the House HR 5, sponsored by Representatives Jones (NC) and D'Amours (NH) along with 100 co-sponsors, was reported favorably on April 27th by the full committee on Merchant Marine and Fisheries. Following action by the House Rules Committee a floor vote will be scheduled for May or June.

The Senate Commerce Committee on April 2[illegible] favorably reported S. 800 sponsored by Senators Stevens (AS), Packwood (OR), Hollings (SC), Johnston (LA), Murkowski (AS), Gorton (NA) and Lautenberg (NJ). Floor action may occur in May or June.

(for comparison of both measures see enclosed Action Alert.)

Municipality of Anchorage

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ANCHORAGE, ALASKA 99502-0650
(907) 264-4111

TONY KNOWLES, MAYOR

DEPARTMENT OF PLANNING

January 31, 1984

The Honorable William Sheffield
State of Alaska
Pouch A
Juneau, Aalaska 99811

Dear Governor Sheffield:

The Anchorage Municipal Health Commission, at its regular meeting January 25, 1984, adopted the following resolution:

WHEREAS, the Federal Hazardous Waste Act known as the Resource Conservation Recovery Act (RCRA) does not assure constant nationwide protection of human health from the potential effect of massive annual accumulation of hazardous waste, and

WHEREAS, any Waste produced in a quantity less than 2200 lbs. (1 each metric ton) is not regulated under the federal Resource Conservation Recovery Act (RCRA) (except wastes that are specifically listed as accutely hazardous), and

WHEREAS, without strict regulations and a comprehensive management plan, the creation of a hazardous waste problem is a reality now, and

WHEREAS, the State of Alaska has completed the public participation period and found overwhelming public support for the Alaska Department of Conservation's proposed regulations, and

WHEREAS, the degree of hazard approach used in the State Department of Environmental Conservation's proposed hazardous waste regulations is very effective in determining the potential danger of a substance with a greater degree of accuracy that the Resource Conservation Recovery Act (RCRA).

BE IT RESOLVED THAT, the Municipal Health Commission supports the proposed Alaska Department of Conservation's regulation, and all future, local State and Federal efforts to control hazardous and toxic materials from their point of generation to their final disposal;

FEB 6 1984

BE IT FURTHER RESOLVED THAT, this Resolution be sent to the Honorable Governor William Sheffield, the Honorable Commissioner of the Department of Environmental Conservation, Richard Neve, and members of the Legislature.

If you have any questions concerning the Commission's action you may contact the Human Resources Planning Office at (907) 264-4261.

Sincerely,

Peter T Gallagher

Peter T. Gallagher, Chairman
Municipal Health Commission

cc: Members of the State House of Representatives
    Members of the State Senate

mb2/nl2

Alaska Oil and Gas Association

505 W. Northern Lights Boulevard
Suite 219
Anchorage, Alaska 99503
(907) 272-1481

HAND DELIVERED
To be Telecopied to Governor's
Office in Juneau from Anchorage
Office.

December 5, 1983

John Shively
Chief of Staff
Office of the Governor
Pouch A
Juneau, AK 99811

AOGA Materials Regarding
Hazardous Waste Discussion
With Commissioners

Dear John:

Enclosed, as we promised in our telephone conference today with you and Commissioners Neve', Wunnicke and Lyon, are materials prepared for today's meeting. We understand you will have copies made and distributed to the Commissioners from there. Please also have a copy delivered to Attorney General Norman Gorsuch.

Our Executive Committee appreciated the opportunity to discuss our concerns with you and the Commissioners even though the weather prevented us from getting into Juneau. We look forward to meeting again in person as soon as a mutually practicable date can be arranged. We will be in touch soon with our suggestions regarding a date.

Thanks for your help.

Sincerely,

WILLIAM W. HOPKINS
Executive Director

WWH:mk

Enclosures

Information given to Administration 12/5/83

AOGA EXECUTIVE COMMITTEE BRIEFING DOCUMENT
ADEC HAZARDOUS WASTE MANAGEMENT PROGRAM
December 5, 1983

I. AOGA POSITION PAPER

II. ANTICIPATED IMPACTS OF REGULATIONS

   A. Oil and Gas Industry

      ° "And other wastes"

      ° Drill Fluids, cuttings and washwater

   B. Examples of Impacts on Other Industries

      ° Asphalt-based roadmix

      ° Treated timbers

      ° Brine Solution

      ° Other

III. CONCERNS WITH SPECIFIC PROVISIONS OF THE REGULATIONS

   A. Exclusions

   B. Characteristic Tests

   C. 62.050(d) Discarded Chemical Products

   D. HWI/HWII Chart (Persistence, Toxicity)

   E. Article 5 (62.500-590) Management of Moderate Risk Waste

IV. RECOMMENDATIONS

   A. General

   B. Specific

AOGA POSITION PAPER
ADEC HAZARDOUS WASTE MANAGEMENT REGULATIONS
DECEMBER, 1983

EXECUTIVE SUMMARY

AOGA has supported the State and has participated in planning meetings and does not object to a program which regulates truly hazardous wastes. However, AOGA does object to a program which greatly expands what previously has not been classified as "hazardous waste" without showing need or technical justification for such an expansion. The proposed regulations could add extremely costly handling and disposal costs for additional wastes not currently classified as hazardous. A person or business should not be subjected to these regulations without carefully weighing the benefit vs. the costs.

Based on the attached analysis, the State's proposed regulations have the potential for adding up to 2 billion dollars in statewide disposal costs for the oil and gas industry.

Due to the complexity of a set of regulations in which seemingly minor wording changes translate into millions of dollars and hundreds of thousands of barrels of waste inappropriately classified as hazardous, AOGA urges the state to conduct a thorough review of the AOGA oral and written comments.

It is our opinion that the proposed State regulations would:

1) fail to recognize in full the Congress' rationale for the exemptions found in the RCRA regulations, such as "other wastes associated" with the exploration, development or production of crude oil.

2) classify a waste as hazardous based upon questionable and disputed testing criteria. It is felt there is a significant possibility that the federally excluded wastes may not pass the state's proposed characteristic testing criteria and therefore drilling waste and produced waters and "other wastes associated with exploration and production" most likely would be considered hazardous upon promulgation and two years after promulgation.

3) require complex and expensive laboratory tests to determine if materials are hazardous or not; tests not intended for such a "go-no-go" determination.

4) include expansive lists of compounds and materials, which are not intended to classify materials as hazardous waste.

5) would possibly include relatively innocuous materials such as table salt, baking soda, vitamin A, nutmeg oil, cinnamon, citric acid and many other common items as hazardous by virtue of the proposed tests.

BACKGROUND

FEDERAL

When Hazardous Waste Regulations were originally enacted, Congress made no special provisions in the Resource Conservation and Recovery Act of 1976 (RCRA) for several major industries (including oil and gas exploration and production) that generated large volume, low hazard wastes. In 1978 EPA proposed regulations and designated several categories of "special wastes" including utility wastes, mining wastes and drilling muds and produced waters (43 Fed. Reg. 59, 015-16, 18 Dec. 1978). Subsequently, the industry demonstrated that no scientific justification existed for regulating drilling and production wastes as hazardous waste materials and estimated it would cost some $5.2 billion annually (with the first year costs of $14 billion) to comply. Congress agreed the EPA regulations had not been justified and the 1980 Solid Waste Disposal Act Amendments suspended RCRA hazardous regulation of "drilling muds, produced water, and other wastes associated with the exploration or production of crude oil or natural gas...". Congress then instructed EPA to study the issues within two years, and conditioned any subsequent hazardous waste regulation of these wastes on the approval of Congress.

STATE

In 1981 the State legislature passed AS 46.03, charging the Alaska Department of Environmental Conservation with the task of developing Hazardous Waste Management regulations which are "consistent with and substantially equivalent to" federal regulations. AOGA was informed on November 24, 1981 that the Department would begin preparation of a "plan" for the State's Hazardous Waste Program. Between May 1982 and June 1983 AOGA has reviewed and voiced its specific concerns with three sets of draft regulations. We believe the proposed rules continue to 1) lack basic scientific justification, 2) ignore the scientific expertise offered by members of the Hazardous Waste Advisory Work Group (HWAWG) and 3) provide little or no incremental environmental benefit over RCRA. Further, we strongly believe that the proposed regulations are in serious conflict with the enabling legislation for the proposed rules. (A recent Attorney General's opinion disagrees with industry on this issue.)

AOGA, through its membership and participation on the Hazardous Waste Advisory Work Group, provided extensive technical and conceptual input on the proposed regulations. Between March 1982 and January 1983, AOGA provided assistance to ADEC through the work group regarding a continuously changing draft of the regulations. On February 7, 1983, Mr. Stan Hungerford transmitted a copy of the January 1983 draft regulations to AOGA. On March 3, 1983 AOGA informed Mr. Hungerford that the proposed rules were in need of in-depth technical review and notified him of forthcoming technical comments. On April 6, 1983 AOGA provided ADEC an in-depth technical review of the regulations. These comments were the culmination of extensive efforts by many engineers, marine biologists, toxicologists, attorneys and corporate management level individuals. The Department informed AOGA's representative on the Advisory Group that the ADEC could not expend the money or the manpower to consider AOGA's comments prior to release of a public hearing draft. On April 11, 1983, AOGA strongly requested that the regulations not be released until AOGA's comments could be reviewed and considered.

On April 19, 1983, the regulations were moved to public notice, with no apparent review or consideration of the technical comments provided to ADEC by AOGA. On April 25, 1983 Commissioner Neve' wrote AOGA indicating that the "comments of every member of the work group... were reviewed seriously and did help significantly in the preparation of these draft regulations." On May 25, 1983 at the Public Hearing AOGA stated that we felt that there was a lack of consideration of our industry's expertise and input in the development of the April 11, 1983 draft regulations.

By June 8, 1983, AOGA had provided ADEC with extensive oral and written testimony which was developed by the Environmental Committee in conjunction with the following Industry experts:

- Joan DiNal, Senior Counsel, Environmental Affairs for Atlantic Richfield Company.
- Michael Hulse, Environmental Analyst, IMCO Services.
- James P. Ray PH.D., Manager of Environmental Sciences Support, Shell Oil Company.
- Robert E. Abbott PH.D., Coordinator of Environmental Affairs, Conoco, Inc.
- Philip Dorn PH.D., Shell Development Company.
- Ilene Danse M.D., Senior Advisor, Environmental Health Protection, Chevron Environmental Health Center, Inc.

The Department issued its latest draft in October 1983, and send a copy to EPA, which has failed again to address significant concerns which have been repeatedly expressed by our Industry. In summary, the Department has accepted AOGA's comments on three sets of draft regulations and has yet to adequately address the problem.

The following items have been provided for purposes of discussion:

- a discussion of the State's proposed Oil and Gas Exclusion
- a summary of the potential economic impacts on Industry

OIL AND GAS EXCLUSION

The proposed Oil and Gas exclusion has failed to retain the wording "other wastes associated" from the Federal exclusion. In addition, the State's proposed exemption is of little value since the basis by which an operator must evaluate the hazardous nature of a waste would be technically unsound characteristic testing. The State is rapidly approaching the regulation of wastes which were identified by Congress as being of minimal risk to human health and the environment and were exempted from Federal RCRA regulation. ADEC has provided no justification or rationale for not including "other wastes associated" in their exclusion. Possibly the Department feels this wording offers Industry a blanket exemption for all wastes generated in conjunction with exploration, development, or production of crude oil or natural gas. This is not the case as shown in the Conference Report, H. R. Rep, No. 96-1444, 96th Congress., 2nd Session. 32 (1980) which briefly explains:

> "waste materials intrinsically derived from the primary field operations associated with the exploration, development, or production of crude oil, natural gas, or geothermal energy. It would cover such substances as: Hydrocarbon bearing soil in and around the related facilities; drill cuttings, materials (such as hydrocarbons, water, sand and emulsion) produced from a well in conjunction with crude oil, natural gas, or geothermal energy, and the accumulated material (such as hydrocarbon, water, sand and emulsion) from production separators, fluid treating vessels, and production impoundments.
>
> The phrase 'intrinsically derived from the primary field operations...' is intended to differentiate exploration, development and production operations from transportation (from the point of custody transfer or of production separation and dehydration) and manufacturing operations."

The Conference Report has specifically referenced wastes which contain such natural constituents of the ground as soil, sand, water, hydrocarbons and emulsions. These wastes when handled with techniques generally employed by industry and approved by the Department should not be included within the scope of the Hazardous Waste Regulations.

Drilling associated wastes such as muds and produced waters have been provided a temporary exclusion by the Department until such time as Industry provides information to support a permanent exclusion. The Department has been provided extensive amounts of information which supports the conclusion that drilling effluents and produced waters should not be included in Hazardous Waste regulations. However, if the proposed regulations are adopted, the industry will be faced with applying existing data to criteria which has little technical merit, i.e. characteristic testing for toxicity, carcinogenicity, and persistence. This could cause these wastes to be classified as hazardous two years after promulgation.

AOGA recommends that the Department include "and other wastes associated" in its exclusion and provide language similar to the Conference Report as a definition of "other wastes". In addition, the Department should delete the characteristics of toxicity, carcinogenicity, and persistence.

POTENTIAL ECONOMIC IMPACTS ON INDUSTRY

A letter from the Executive Committee to the Governor dated September 12, 1983 stated that the impact on the oil industry would be in excess of $100 million per year.

Commissioner Neve' has been reported as saying he is skeptical regarding the 100 million dollar incremental cost estimate.

This estimate was based upon several individual operators summing their disposal cost for a portion of the wastes that would be regulated as hazardous should the State not apply the federal statutory exemption "other wastes associated". It should be emphasized that the 100 million dollar figure is not inclusive relative to the exemption i.e. does not include drilling associated wastes, and hydrocarbon bearing soil, etc.

Further, the 100 million dollar cost estimate does not include wastes which may ultimately be reclassified as hazardous based on the State's proposed regulations i.e. additional characteristic testing, etc.

Justification for the estimate is provided by way of example. Waste volume estimates were provided by one North Slope operator and one Cook Inlet operator. For this discussion, disposal costs will be those provided by Mr. DiTraglia (ADEC) at the January 31, 1983 Hazardous Waste Advisory Work Group (HWAWG) meeting and disposal costs developed by the respective operators.

North Slope:

Wastes:
- materials produced from a well such as sand and emulsions

- accumulated material from production separators, fluid treating vessels, storage vessels and production impoundments

Waste Volume: The referenced waste (includes Prudhoe Bay and Kuparuk) is generated at a current rate of approximately 262,000 bbls/yr

Disposal Costs:

- State's estimate of $271 per bbl (analysis included)

Cost = 262,000 bbls/yr X $271/bbl = 71 million dollars

- Operators' estimate of $350 per bbl (analysis not included)

Cost = 262,000 bbls/yr X $350/bbl = 92 million dollars

- Operators' estimate with $100 (JRB report) analysis fee included

Cost = 262,000 bbl/yr X $450/bbl = 118 million dollars

Cook Inlet:

Waste:

- materials produced from a well such as sand and emulsions

Waste Volume:

- The reference waste is generated at a rate of approximately 20,000 bbls/yr

Disposal Costs:

- JRB's estimated disposal cost of $271 per bbl (includes analysis)

Cost = 20,000 bbls/yr X $271/bbl = $5 million per year

- Operators' estimated disposal cost of $350 per bbl (analysis not included)

Cost = 20,000 bbls/yr X $350/bbl = $7 million per year

- Operators' estimate with $100 (JRB report) analysis fee included

Cost = 20,000 bbls/yr X $450/bbl = $9 million per year

Summary

	JRB Estimated Disposal Cost With Analysis	Operators Estimated Disposal Cost Without Analysis	Operators Estimated Disposal Cost Costs with Analysis
North Slope	$71 million	$92 million	$118 million
Cook Inlet	$5 million	$7 million	$9 million
TOTAL	$76 million	$99 million	$127 million

As was previously stated, drilling muds and cuttings were not included in the aforementioned economic impact analysis.

At this time AOGA offers the following example as a range by which to estimate additional potential economic impacts. AOGA is uncertain as to the percentage of drilling wastes which may ultimately be reclassified as hazardous based upon the proposed characteristic tests for toxicity, carcinogenicity, and persistence. However, AOGA is certain that the proposed characteristic criteria and tests have technical flaws and are overly restrictive.

Waste:

° Drilling muds and cuttings and washwater

Waste Volume: a typical well will generate about 36,000 barrels of muds and cuttings and washwater. In 1982, 203 wells were drilled in Alaska (API Basic Petroleum Data Book Vol. III, No. 3, Sept. 83). Waste volume (estimate) = 36,000 bbls X 203 wells

= 7.3 million bbls in 1982

Disposal Costs:

° JRB's estimated disposal cost of $271 per bbl (includes analysis)

Cost = 7.3 million bbls X $271/bbl = $2 billion in 1982

AOGA does not anticipate the total volume of drilling muds and cuttings to be classified as hazardous. However, based on the preceding analysis, the State's proposed regulations have the potential for adding up to 2 billion dollars in statewide disposal costs for drilling muds and cuttings.

MM3:901

COMMENTS:

ADEC HAZARDOUS WASTE MANAGEMENT PROGRAM

DECEMBER 5, 1983

The information contained herein is provided as a supplement for AOGA's Position Paper of December, 1983, and addresses the following topics:

I. ANTICIPATED IMPACTS OF REGULATIONS

II. CONCERNS WITH SPECIFIC PROVISIONS OF THE REGULATIONS

III. AOGA RECOMMENDATIONS

ADEC HAZARDOUS WASTE MANAGEMENT PROGRAM
DECEMBER 5, 1983

I. ANTICIPATED IMPACTS OF REGULATIONS

A. Oil and Gas Industry (support-industry waste disposal costs not included)

°"And other wastes" (materials produced from a well such as sand and emulsion, accumulated materials from production separators, fluid treating vessels, etc.)

- $100,000,000/yr. in disposal costs

° Drill fluids, cuttings and washwater

- Estimated volume 7.3 million bbls. based on 1982 drilling activities

- Potential of up to $2 billion/yr. cost to Industry

B. Examples of Impacts on Other Industries

° Asphalt-based roadmix

- Asphalt-based roadmix would be classified as a Class II hazardous waste under the characteristic of persistence.

- Generation of 400 lbs. or more of hazardous waste per month would require a generator to comply with the proposed Hazardous Waste Regulations.

- All waste asphalt (including discarded pavement) must be transported to a permitted hazardous waste disposal facility.

° Treated timbers (railroad ties, power poles, etc.)

- Treated timber would be classified as a Class II hazardous waste by the characteristic of toxicity (equivalent concentration/creosote).

- More than 10,000 used railroad ties alone are generated in this state per year and would be subject to these regulations.

- Used railroad ties are currently sold at approximately $10 each to the public. Would the generator still be able to sell these to the public?

- Brine Solution (fish processing industry)

    - More than 45 gallons of a brine solution containing 10% or greater of salt (NaCl) would be a Class I Hazardous Waste subject to the requirements of these regulations.

    - Crab processors, using brine freezers, generate from 2,000 to 9,400 gallons per month per processor of used brine containing greater than 10% salt.

    - Would EPA continue to allow a state-designated hazardous waste to be discharged under an NPDES permit?

C. <u>Other Industries with Concerns</u>

- Large volume, low hazard waste

    - Lumber and Pulp Industry

- Small quantity generators

    - independent gas and service stations
    - dry cleaning

- Previously-regulated industries with additional impacts

    - Paint Manufacturers
    - Pest Control

NOTE: These examples are not meant to draw attention to a specific industry or provide the basis for another permanent exclusion. Rather, they point to examples of why the additional characteristic tests are inappropriate.

II. CONCERNS WITH SPECIFIC PROVISIONS OF THE REGULATIONS

The following comments are only shown as specific examples of major concerns. Please refer to the extensive AOGA written comments and testimony for further details.

A. Exclusions

The regulations fail to recognize in full the exemptions found in the RCRA regulations, such as "other wastes associated" with the exploration, development or production of crude oil as envisioned by Congress.

° 62.140 (b) Temporary Exclusion

The proposed regulations reverse the roles of the regulating agency and permittee. The permittee will have to prove that certain wastes are not hazardous, rather than the State having to prove that they are. Further the testing criteria in the regulations are inadequate to provide such proof, not because the wastes are hazardous but rather because the testing procedures are too stringent to identify truly hazardous waste.

B. Characteristic Tests

The proposed testing procedures either are inappropriately applied or have not been verified by the scientific community, do not give reproducible results and are subject to inherent errors. Some of the tests have no relevance to the true hazardous nature of the compounds.

° 62.060 Toxicity

This system proposes on overly simplistic scheme whose validity has not been established. For example, the oral (rat) LD_{50} criteria is 100 times more stringent than RCRA. What is state's justification or rationale?

° 62.070 Persistence

Classifies as hazardous many stable products which are not a hazard to people or the environment. The state has not provided the rationale used in proposing this test.

° 62.080 Carcinogenicity

The regulations provide no guidance of how to approach the problem of determining the cancer-producing agents other than the International Agency for Research on Cancer (IARC) list. How does one demonstrate that a waste sample contains less than 1% of all <u>known or suspected</u> carcinogens? What is the basis for classifying as hazardous, 1% concentration of these listed substances when contained within a waste? Why not 0.1% or 10%? What is the rationale for making less than 1% of various carcinogens additive?

° 62.120 Waste Extraction Procedure (WEP) Test

The list has been expanded and maximum concentrations levels changed from that which is listed in RCRA without justification. ADEC has therefore "listed" wastes as hazardous without following requirements in enabling legislation (AS 46.03.299(b)(2)).

C. <u>62.050(d) Discarded Chemical Products</u>

This regulation should not regulate the use or intended <u>use</u> of a chemical material prior to its intended disposal. This section currently discourages recycling and reuse as was the intent of RCRA. The proposed regulation would classify a waste as hazardous if it contained any of the listed generic names regardless of their concentration or intended use whereas 40 CFR 261.33 only deals with formulations with listed chemical as their "sole active ingredients."

D. <u>HWI/HWII Chart (Persistence, Toxicity)</u>

What is the documented source on which the hazardous waste graphs were based?

E. Article 5 (62.500-590) Management of Moderate Risk Waste

This is a new article on which the public has not had an opportunity to comment. Classification of a designated hazardous waste as a Class I (moderate risk waste) vs. a Class II hazardous waste is irrelevant. Once a waste is labeled as hazardous, landfill operators may not accept the (designated hazardous) waste and EPA and the public will likely require disposal only at approved hazardous waste disposal sites.

Summary:

Hazardous waste programs by nature are complex and minor wording changes can translate into millions of dollars and hundreds of thousands of barrels of waste inappropriately classified as hazardous. According to current and past surveys, the management of hazardous waste within the state is appropriately regulated under the federal program and is not a problem in the State of Alaska. Therefore, it is more appropriate to implement regulations which address hazardous wastes (as defined by RCRA) that are similar to the currently enforced regulations. If expansions are needed, ADEC should take the time to properly research and document the problem and evaluate the financial impact vs. the benefit to the people and the environment of the State of Alaska. This could be accomplished by deleting controversial provisions now and scheduling further review along with other provisions such as recycling in the years following final authorization of the basic RCRA program.

III. RECOMMENDATIONS

A. General

° The state must conduct and document an objective interagency review of all comments received to date. Verify why after extensive, well-documented comments prior to and during the public comment period, the final draft regulations presently submitted to EPA remain substantially the same as previous drafts.

° We suggest a nationally-recognized scientific body, such as the National Academy of Sciences or Batelle, review the technical merits and cost/benefit implications associated with the proposed hazardous waste management program.

° That a multi-year phased program must be developed whereby various concerns could be addressed and implemented. Such a program may be as follows: (1) Phase 1 would utilize a basic RCRA program (drop the additional characteristic tests, include RCRA exclusions, drop Section 5, etc.); (2) Phase 2 would address in appropriate detail reuse and recycling programs suitable for Alaska; and (3) Phase 3--Based upon first-hand experience, would review additional measures that may be necessary to deal with wastes improperly identified as hazardous. This approach would allow the State to gain regulatory experience in implementing a well thought out and manageable program.

B. SPECIFIC

° Temporary exemption--adopt RCRA exemption and include definition of "and other wastes" as found in the Congressional Conference Report.

° Delete characteristics of carcinogenicity, persistence and toxicity until scientific justification can be provided for appropriate hazard classification.

° Delete additional items which have been added to the WEP test from the federal EP toxicity test until such time as proper justification is provided.

° Section 62.050(d) should not regulate the intended use or reuse of a chemical material and should reflect the footnote contained in 40 CFR 261.33.

° The addition of a new Article 5 is a major revision which should be subject to public review and comment. Suggest deletion of the section until it can be shown that it is necessary to regulate moderate risk wastes as hazardous.

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I. INTRODUCTION: Narrative of ADEC Tests for Criteria Covering Persistent Classes of Chemical Compounds

There are two classes of chemical compounds that present a long-term threat because they tend to persist, bioaccumulate and present a chronic threat to human health and the environment. These classes are halogenated hydrocarbons and polycyclic aromatic hydrocarbons.

It is important for the reader to understand that analysis for these compound classes is intentionally broad and inclusive rather than a compound-by-compound analysis. As such, the analysis should be viewed as a screening test which alerts the analyst to the presence of potentially hazardous materials. At threshold concentrations of such persistent materials, further analysis for the presence of genetically active chemicals or the use of short-term bioassays for mutagenicity is adivsed. The Department of Environmental Conservation Hazardous Waste Section should be consulted before any such tests are undertaken. In addition, these methods are designed to determine semivolatile materials. Therefore, if the presence of solvents such as benzene or methylene chloride is suspected, the Department of Environmental Conservation may require a volatile analysis by GC or GC/MS.

A. Sample Extraction for Halogenated and Polycyclic Aromatic Hydrocarbon Analysis

This method is applicable to nearly all types of samples, regardless of water content, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The extraction procedure involves sonification of the sample with methylene chloride, neutralization to pH 7, and the addition of anhydrous sodium sulfate to remove the water. The amount of acid or base required for the neutralization is determined by titration of the sample. For halogenated hydrocarbon analysis, the methylene chloride extraction solvent is exchanged to hexane. For polycyclic aromatic hydrocarbon analysis, the methylene chloride extraction solvent is exchanged to cyclohexane. Specific procedures for a sample extraction are contained in Section III.

B. Halogenated Hydrocarbon Analysis

Halogenated hydrocarbons can be determined in the final extract by a "Parr" bomb analysis followed by a titrimetric determination of chloride ion, or by analysis with a Dohrmann MCTS 20 Microcoulometric analyzer, or by an equivalent method.

The Parr bomb analysis consists of combusting the extract in a "Parr" bomb to convert the organic compounds to simpler chemical forms. Halogenated (i.e., contains chlorine, fluorine, iodine or bromine) compounds break down to form the respective ion forms (i.e., chlorides, fluorides, iodides and bromides). These are water soluble. The Parr bomb residue is then rinsed with water and the solution analyzed for the respective halide. Since the most common halogen is chlorine, these procedures include a colorimetric test for the chloride ion in water, commonly used in water quality analyses. Similar tests for the iodide, fluoride and bromide ions are available upon request from the department and taken from similar analytical sources. The Parr bomb and colorimetric test procedure for the chloride ion is described in Attachment 1.

When the analyst knows what particular halogenated compounds are present, chromatographic methods may be used to determine threshold concentrations. An example of such a method is described in the EPA Method 612 for chlorinated hydrocarbons.

C. Polycyclic Aromatic Hydrocarbon Analysis

Polycyclic aromatic hydrocarbons (PAHs) with 4 or more fused rings are of interest because of their persistence, bioaccumulation, and chronic disease-causing properties. Following the procedures of EPA Method 610 (Attachment 2), the methylene chloride extract is exchanged to cyclohexane and a silica gel cleanup is employed to separate PAHs from coextracted interferences. The PAHs with 4 or more rings are then identified and quantitated by HPLC.

II. Crude Sample Extraction for Halogenated and Polycyclic Aromatic Hydrocarbon Analysis

The crude sample may be in several physical forms and mixtures of these forms as follows:

1. Dry granular solid.
2. Solid with liquid organic phase.
3. Solid with water phase.
4. Large chunks of a tar-like solid material.
5. Liquid organic phase only.
6. Mixed aqueous and organic phases.
7. Solid granular phase that is damp with water.
8. Combinations of the above.

Each of these physical forms may require different extraction procedures. Therefore, in many instances the extraction procedure or combination of extraction procedures that will be used are the chemist's best judgment when he receives the sample.

The extractions of single phase sample systems are covered in the following written discussion. The multiple phase systems would be separated by decantation, filtration or centrifuging and each phase then treated as a single phase system.

This method is applicable to nearly all types of samples, regardless of water content, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. It is presumed that reactivity and corrosivity have been tested prior to this analysis. DO NOT PROCEED WITH THIS EXTRACTION PROCEDURE IF CYANIDE, SULFIDE, EXPLOSIVES, OR OTHER SUCH HAZARDOUS MATERIALS ARE PRESENT UNLESS PROPER PRECAUTIONS ARE TAKEN.

The extraction procedure involves sonification of the sample with methylene chloride, neutralization to pH 7, and the addition of anhydrous sodium sulfate to remove the water. The amount of acid or base required for the neutralization is determined by titration of the sample. The particle size of all samples, except those comprised of nonporous inorganic particles (e.g. soils and sediments), should be reduced to less than 0.1 mm diameter before extraction if possible. A glass mortar and pestle is recommended for grinding the sample.

Extraction Procedure:

1. Thoroughly mix the sample to enable a representative sample to be obtained. Weigh 3.0 g (wet weight) of sample into a 200 ml centrifuge tube. Add 15 ml methylene chloride and 15 ml of water.

2. Sonify the mixture for two minutes by inserting the sonifier horn 0.5-1.0 cm below the surface and using a power setting of 5 and a 50% pulsed duty cycle.

3. Transfer the contents of the centrifuge tube to a 400 ml beaker using 50 ml of methylene chloride followed by 150 ml of water as rinses.

4. Adjust the pH of the mixture to 7.0 ± 0.2 by titration with 0.4 M H_3PO_4 or 0.4 M K_3PO_4 using a pH meter to measure the pH. Record the volume of acid or base required.

5. The extraction with methylene chloride is performed using a fresh portion of the sample. Weigh 3.0 g (wet weight) of sample into a 200 ml centrifuge tube. Add 15 ml of methylene chloride. Add 1.0 ml of 4 M phosphate buffer pH 7.0, and an amount of 4 M H_3PO_4 or 4 M K_3PO_4 equal to one tenth of the pH 7 acid or base volume requirement determined in Section 4. For example, if the acid requirement in Section 4 was 2.0 ml of 0.4 M H_3PO_4, the amount of 4 M H_3PO_4 needed would be 0.2 ml.

6. Sonify the mixture for 1 minute by inserting the sonifier horn 0.5-1.0 cm below the surface and using a power setting of 5 and a 50% pulsed duty cycle. Cool the mixture in an ice bath or cold water bath, if necessary, to maintain a temperature of 20-30°C. Add 135 ml of methylene chloride, adjust the position of the sonifier horn to

0.5-1.0 cm below the surface and repeat the sonification for 1 minute. Some samples, especially those that contain much water, may not disperse well in this step but will disperse after sodium sulfate is added. Add all at once an amount of anhydrous sodium sulfate powder equal to 15.0 g plus 3.0 g/ml of the 4 M H_3PO_4 or 4 M K_3PO_4 added in Section 5. Immediately cap the centrifuge tube and shake it vigorously for 1 minute. Insert the sonifier horn 0.5-1.0 cm below the surface and sonify for 2 minutes as descried above. Allow the mixture to stand until a clear supernatant is obtained. Centrifuge if necessary to facilitate the phase separation.

7. Transfer the supernatant to a K-D flask fitted into a 25 ml concentrator tube. Add two boiling chips, attach a three-ball macro Snyder column to the K-D flask, and concentrate the extract using a water bath at 60 to 65°C. Place the K-D apparatus in the water bath so that the concentrator tube is about half immersed in the water and the entire rounded surface of the flask is bathed with water vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation the balls of the column actively chatter but the chambers do not flood. When the liquid has reached an apparent volume of 5 to 6 ml, remove the K-D apparatus from the water bath and allow the solvent to drain for at least 5 minutes while cooling. Remove the Snyder column and rinse the flask and the lower joint of the flask into the concentrator tube with 1-2 ml of methylene chloride.

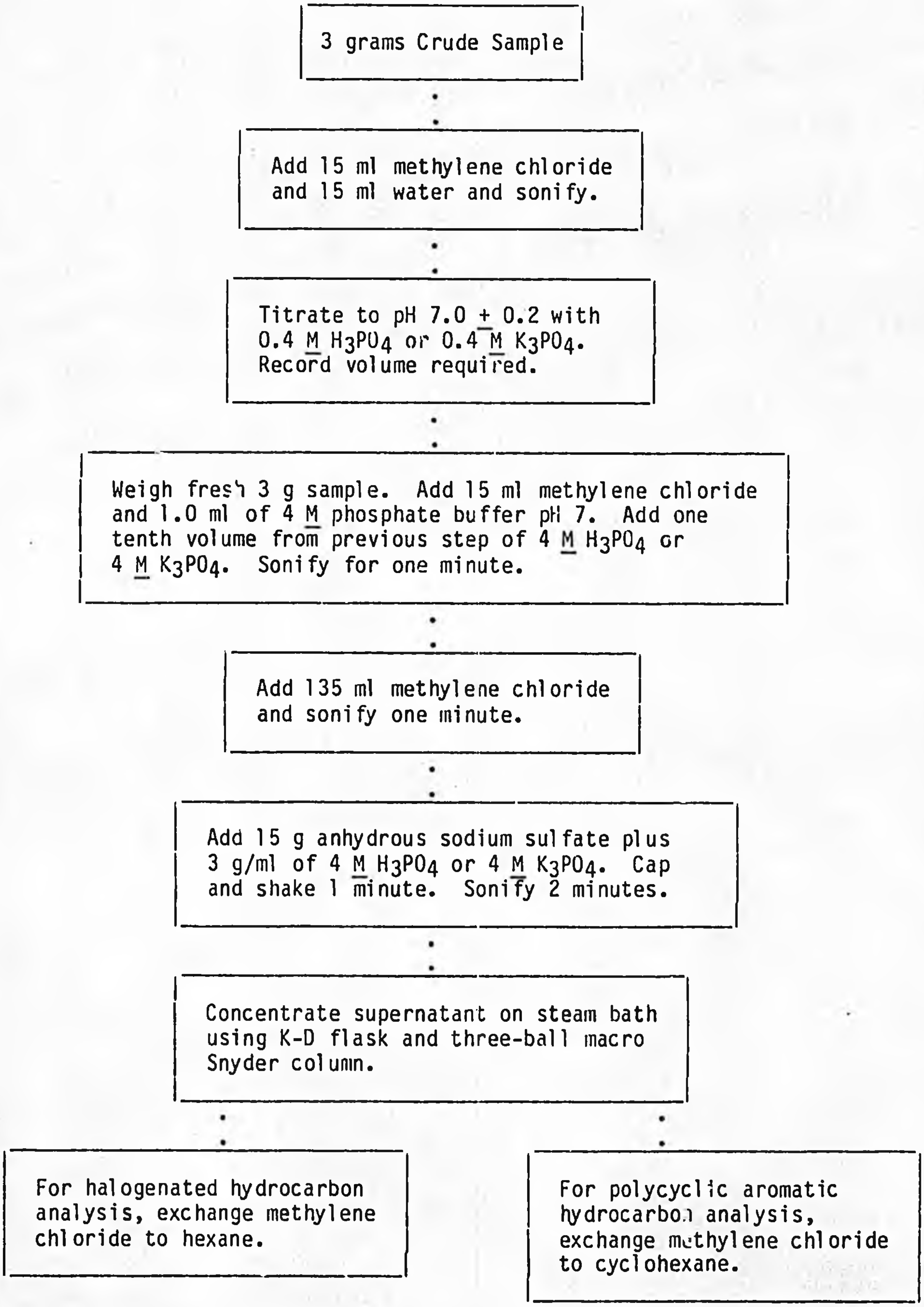
8. For analysis of polycyclic aromatic hydrocarbons, proceed to Section 10 of EPA Method 610, Polynuclear Aromatic Hydrocarbons (Attachment 2).

9. For analysis of halogenated hydrocarbons, the methylene chloride extraction solvent must be exchanged to hexane. Add 5 ml of hexane to the extract in the concentrator tube. Evaporate the extract to 1.0 ml with a gentle stream of nitrogen with the concentrator tube placed in a water bath maintained at 40°C.

10. Total halogenated hydrocarbons may be determined by using a Parr bomb analysis followed by a titrimetric determination of chlorine (Attachment 1) or by analysis with a Dohrmann MCTS 20 Microcoulometric Analyzer, or by equivalent method.

Reference: EPA, EMSL-Las Vegas Procedure No. 2 for analysis of hazardous waste for semivolatile organic compounds.

HAZARDOUS WASTE EXTRACTION PROCEDURE for ANALYSIS of HALOGENATED HYDROCARBONS and POLYCYCLIC AROMATIC HYDROCARBONS



3 grams Crude Sample

Add 15 ml methylene chloride and 15 ml water and sonify.

Titrate to pH 7.0 ± 0.2 with 0.4 M H_3PO_4 or 0.4 M K_3PO_4. Record volume required.

Weigh fresh 3 g sample. Add 15 ml methylene chloride and 1.0 ml of 4 M phosphate buffer pH 7. Add one tenth volume from previous step of 4 M H_3PO_4 or 4 M K_3PO_4. Sonify for one minute.

Add 135 ml methylene chloride and sonify one minute.

Add 15 g anhydrous sodium sulfate plus 3 g/ml of 4 M H_3PO_4 or 4 M K_3PO_4. Cap and shake 1 minute. Sonify 2 minutes.

Concentrate supernatant on steam bath using K-D flask and three-ball macro Snyder column.

For halogenated hydrocarbon analysis, exchange methylene chloride to hexane.

For polycyclic aromatic hydrocarbon analysis, exchange methylene chloride to cyclohexane.

ATTACHMENT 1

PARR BOMB ANALYSIS

ANSI/ASTM D808-63 (REAPPROVED 1976)

American Society for Testing and Materials Standards are protected by copyright. Copies of ASTM standards can be obtained by writing to the following address:

ASTM
1916 Race Street
Philadelphia, PA 19103

Steps 6.5, 6.6 and 7 in the above referenced method are replaced by the chloride determination described in Appendix F, Attachment 1a.

ATTACHMENT 1a

CHLORIDE

Method 325.3 (Titrimetric, Mercuric Nitrate)

STORET NO. 00940

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The method is suitable for all concentration ranges of chloride content; however, in order to avoid large titration volume, a sample aliquot containing not more than 10 to 20 mg Cl per 50 ml is used.
   - 1.3 Automated titration may be used.
2. Summary of Method
   - 2.1 An acidified sample is titrated with mercuric nitrate in the presence of mixed diphenylcarbazone-bromophenol blue indicator. The end point of the titration is the formation of the blue-violet mercury diphenylcarbazone complex.
3. Comments
   - 3.1 Anions and cations at concentrations normally found in surface waters do not interfere.
   - 3.2 Sulfite interference can be eliminated by oxidizing the 50 ml of sample solution with 0.5 to 1 ml of H_2O_2.
4. Apparatus
   - 4.1 Standard laboratory titrimetric equipment including a 1 ml or 5 ml microburet with 0.01 ml graduations.
5. Reagents
   - 5.1 Standard sodium chloride, 0.025 N: Dissolve 1.4613 ±0.0002 g (dried at 600°C for 1 hour) in chloride-free water in a 1 liter volumetric flask and dilute to the mark.
   - 5.2 Nitric acid, HNO_3 solution (3 + 997)
   - 5.3 Sodium hydroxide solution, NaOH, (10 g/l)
   - 5.4 Hydrogen peroxide (30%), H_2O_2
   - 5.5 Hydroquinone solution (10 g/liter): Dissolve 1 g of purified hydroquinone in water in a 100 ml volumetric and dilute to the mark.
   - 5.6 Mercuric nitrate titrant (0.141 N): Dissolve 25 g $Hg(NO_3)_2 \cdot H_2O$ in 900 ml of distilled water acidified with 5.0 ml conc. HNO_3 in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.141 N and check. Store in a dark bottle. A 1.00 ml aliquot is equivalent to 5.00 mg of chloride.
   - 5.7 Mercuric nitrate titrant (0.025 N): Dissolve 4.2830 g $Hg(NO_3)_2 \cdot H_2O$ in 50 ml of distilled water acidified with 0.5 ml conc. HNO_3 (sp. gr. 1.42) in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard

Approved for NPDES
Issued 1971
Editorial revision 1978

sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.025 N and check. Store in a dark bottle.

5.8 Mercuric nitrate titrant (0.0141 N): Dissolve 2.4200 g $Hg(NO_3)_2 \cdot H_2O$ in 25 ml of distilled water acidified with 0.25 ml of conc. HNO_3 (sp. gr. 1.42) in a 1 liter volumetric flask and dilute to the mark with distilled water. Filter if necessary. Standardize against standard sodium chloride solution (5.1) using procedure 6. Adjust to exactly 0.0141 N and check. Store in a dark bottle. A 1 ml aliquot is equivalent to 500 ug of chloride.

5.9 Mixed indicator reagent: Dissolve 0.5 g crystalline diphenylcarbazone and 0.05 g bromophenol blue powder in 75 ml 95% ethanol in a 100 ml volumetric flask and dilute to the mark with 95% ethanol. Store in brown bottle and discard after 6 months.

5.10 Alphazurine indicator solution: Dissolve 0.005 g of alphazurine blue-green dye in 95% ethanol or isopropanol in a 100 ml volumetric and dilute to the mark with 95% ethanol or isopropanol.

6. Procedure

6.1 Place 50 ml of sample in a vessel for titration. If the concentration is greater than 20 mg/l chloride, use 0.141 N mercuric nitrate titrant (5.6) in step 6.6 or dilute. If the concentration is less than 2.5 mg/l of chloride, use 0.0141 N mercuric nitrate titrant (5.8) in step 6.6, a 1 ml or 5 ml microburet, and determine an indicator blank on 50 ml chloride-free water using procedure 6.6. If the concentration is less than 0.1 mg/l of chloride concentrate an appropriate volume to 50 ml.

6.2 Add 5 to 10 drops of mixed indicator reagent (5.9), shake or swirl solution.

6.3 If a blue-violet or red color appears add HNO_3 solution (5.2) dropwise until the color changes to yellow.

6.4 If a yellow or orange color forms immediately on addition of the mixed indicator, add NaOH solution (5.3) dropwise until the color changes to blue-violet; then add HNO_3 solution (5.2) dropwise until the color changes to yellow.

6.5 Add 1 ml excess HNO_3 solution (5.2).

6.6 Titrate with 0.025 N mercuric nitrate titrant (5.7) until a blue-violet color persists throughout the solution. See 6.1 for choice of titrant normality. Alphazurine indicator solution (5.10) may be added with the indicator to sharpen the end point. This will change color shades. Practice runs should be made.

6.7 Additional steps to eliminate particular interferences:

6.7.1 If chromate is present at < 100 mg/l and iron is not present, add some alphazurine indicator solution (5.10) and acidify to pH 3 (indicating paper). End point will then be an olive-purple color.

6.7.2 If chromate is present at > 100 mg/l and iron is not present, add 2 ml of fresh hydroquinone solution (5.5).

6.7.3 If ferric ion is present use volume containing no more than 2.5 mg of ferric ion or ferric ion plus chromate ion. Add 2 ml fresh hydroquinone solution (5.5).

6.7.4 If sulfite ion is present, add 0.5 ml of H_2O_2 solution (5.4) to 50 ml sample and mix for 1 minute.

7. Calculation

$$\text{mg chloride/l} = \frac{(A - B)N \times 35{,}450}{\text{ml of sample}}$$

where:
A = ml titrant for sample
B = ml titrant for blank
N = normality mercuric nitrate titrant

mg NaCl/l = mg chloride/l x 1.65

8. Precision and Accuracy

8.1 Forty two analysts in eighteen laboratories analyzed synthetic water samples containing exact increments of chloride, with the following results:

Increment as Chloride mg/liter	Precision as Standard Deviation mg/liter	Accuracy as	
		Bias, %	Bias, mg/liter
17	1.54	+2.16	+0.4
18	1.32	+3.50	+0.6
91	2.92	+0.11	+0.1
97	3.16	-0.51	-0.5
382	11.70	-0.61	-2.3
398	11.80	-1.19	-4.7

(FWPCA Method Study 1, Mineral and Physical Analyses)

8.2 In a single laboratory (EMSL), using surface water samples at an average concentration of 34 mg Cl/l, the standard deviation was ±1.0.

8.3 A synthetic unknown sample containing 241 mg/l chloride, 108 mg/l Ca, 82 mg/l Mg, 3.1 mg/l K, 19.9 mg/l Na, 1.1 mg/l nitrate N, 0.25 mg/l nitrite N, 259 mg/l sulfate and 42.5 mg/l total alkalinity (contributed by $NaHCO_3$) in distilled water was analyzed in 10 laboratories by the mercurimetric method, with a relative standard deviation of 3.3% and a relative error of 2.9%.

Bibliography

1. Annual Book of ASTM Standards, Part 31, "Water", Standard D512-67, Method A, p 270 (1976).

ATTACHMENT 2

United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

Test Method

Polynuclear Aromatic Hydrocarbons — Method 610

1. Scope and Application

1.1 This method covers the determination of certain polynuclear aromatic hydrocarbons (PAH). The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Acenaphthene	34205	83-32-9
Acenaphthylene	34200	208-96-8
Anthracene	34220	120-12-7
Benzo (a) anthracene	34526	56-55-3
Benzo (a) pyrene	34247	50-32-8
Benzo (b) fluoranthene	34230	205-99-2
Benzo (ghi) perylene	34521	191-24-2
Benzo (k) fluoranthene	34242	207-08-9
Chrysene	34320	218-01-9
Dibenzo (a, h) anthracene	34556	53-70-3
Fluoranthene	34376	206-44-0
Fluorene	34381	86-73-7
Indeno (1, 2, 3-cd) pyrene	34403	193-39-5
Naphthalene	34696	91-20-3
Phenanthrene	34461	85-01-8
Pyrene	34469	129-00-0

1.2 This is a chromatographic method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for many of the parameters listed in Section 1.1, using the extract produced by this method.

1.3 This method provides for both high performance liquid chromatographic (HPLC) and gas chromatographic (GC) approaches to the determination of PAHs. The gas chromatographic procedure does not adequately resolve the following four pairs of compounds: anthracene and phenanthrene; chrysene and benzo (a) anthracene; benzo (b) fluoranthene and benzo (k) fluoranthene; and dibenzo (a, h) anthracene and indeno (1, 2, 3-cd) pyrene. Unless the purpose for the analysis can be served by reporting the sum of an unresolved pair, the liquid chromatographic approach must be used for these compounds. The liquid chromatographic method does resolve all 16 of the PAHs listed.

1.4 The method detection limit (MDL, defined in Section 15)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ depending upon the nature of interferences in the sample matrix.

1.5 The sample extraction and concentration steps in this method are essentially the same as in methods 606, 608, 609, 611 and 612. Therefore, a single sample may be extracted to measure the parameters included in the scope of each of these methods, provided the concentration is high enough to permit selecting aliquots of the extract for cleanup, when required. Selection of the aliquots must be made prior to the solvent exchange steps of this method. The analyst is allowed the latitude, under Gas Chromatography (Section 13), to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.6 Any modification of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.7 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC and GC and in the interpretation of liquid and gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately one-liter, is solvent extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and concentrated to a volume of 10 mL or less. The solvent is exchanged to cyclohexane prior to cleanup. Following cleanup, when using HPLC for determination of the PAHs, the solvent is exchanged to acetonitrile. Ultraviolet (UV) and fluorescence detectors are used with HPLC. When cleanup is not required and when flame ionization detector GC is used for determination, the methylene chloride extract may be analyzed directly. When cleanup is required, the cyclohexane exchange is made. Instrumental conditions are described which permit the separation and measurement of the PAH compounds[2].

2.2 A silica gel column cleanup procedure is provided to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.5.

3.1.1 Glassware must be scrupulously cleaned[3]. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water and distilled water. It should then be drained dry, and heated in a muffle furnace at 400°C for 15 to 30 minutes. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

3.3 The extent of interferences that may be encountered using liquid chromatographic techniques has not been fully assessed. Although the HPLC conditions described allow for a unique resolution of the specific PAH compounds covered by this method, other PAH compounds may interfere.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2 The following paramenters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens; benzo (a) anthracene, benzo (a) pyrene and dibenzo (a, h) anthracene.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle - Amber glass, one-liter or one-quart volume, fitted with screw caps lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The container must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional) - Must incorporate glass sample containers for the collection of a minimum of 250 mL. Sample containers must be kept refrigerated at 4°C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only).

5.2.1 Separatory funnel - 2000-mL, with Teflon stopcock.

5.2.2 Drying column - Chromatographic column 400 mm long x 19 mm ID with coarse frit.

5.2.3 Concentrator tube, Kuderna-Danish - 10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.4 Evaporative flask, Kuderna-Danish - 500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.5 Snyder column, Kuderna-Danish - three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.6 Snyder column, Kuderna-Danish - two-ball micro (Kontes K-569001-0219 or equivalent).

5.2.7 Vials - Amber glass, 10- to 15- mL capacity, with Teflon-lined screwcap.

5.2.8 Chromatographic column - 250 mm long x 10 mm ID with coarse fritted disc at bottom and Teflon stopcock.

5.3 Boiling chips - approximately 10/40 mesh. Heat to 400°C for 30 minutes or Soxhlet extract with methylene chloride.

5.4 Water bath - Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5 Balance — Analytical, capable of accurately weighing 0.0001g.

5.6 High performance liquid chromatographic apparatus (modular):

5.6.1 Gradient pumping system, constant flow.

5.6.2 Reverse phase column, 5 micron HC-ODS Sil-X, 250 mm x 2.6 mm ID (Perkin-Elmer No. 089-0716 or equivalent).

5.6.3 Fluorescence detector, for excitation at 280 nm and emission greater than 389 nm cutoff (Corning 3-75 or equivalent). Fluorometers should have dispersive optics for excitation and can utilize either filter or dispersive optics at the emission detector.

5.6.4 UV detector, 254 nm, coupled to fluorescence detector.

5.6.5 Strip-chart recorder compatible with detectors. Use of a data system for measuring peak areas and retention times is recommended.

5.7 Gas chromatograph - An analytical system complete with temperature programmable gas chromatograph suitable for on-column injection or splitless injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.7.1 Column - 1.8 m long x 2 mm ID pyrex glass packed with 3% OV-17 on Chromosorb W-AW-DCMS (100/120 mesh) or equivalent. This column was used to develop the retention time data in Table 2. Guidelines for the use of alternate column packings are provided in Section 13.

5.7.2 Detector - Flame ionization. This detector has proven effective in the analysis of wastewaters for the compounds listed in the scope excluding the four pairs of unresolved compounds listed in Section 1.3. Guidelines for the use of alternate detectors are provided in Section 12.2.

6. Reagents

6.1 Reagent water - Reagent water is defined as a water in which an interferent is not observed at the MDL of each parameter of interest.

6.2 Sodium thiosulfate - (ACS) Granular.

6.3 Cyclohexane, methanol, acetone, methylene chloride, and pentane - Pesticide quality or equivalent.

6.4 Acetonitrile, high purity HPLC quality, distilled in glass.

6.5 Sodium sulfate - (ACS) Granular, anhydrous. Purify by heating at 400°C for four hours in a shallow tray.

6.6 Silica gel - Grade 923 (100/200 mesh) dessicant (Davison Chemical or equivalent). Before use, activate for at least 16 hours at 130°C in a shallow glass tray, loosely covered with foil.

6.7 Stock standard solutions (1.00 μg/μL) - Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in HPLC quality acetonitrile, dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. If compound purity is certified at 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can be used to determine the accuracy of calibration standards will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicate a problem.

7. Calibration

7.1 Establish liquid or gas chromatographic operating parameters to produce resolution of the parameters equivalent to that indicated in Tables 1 or 2. The chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with acetonitrile. One of the external standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Analyze each calibration standard (5 to 25 μL for HPLC and 2 to 5 μL for GC), and tabulate peak height or area responses against the mass injected. The results may be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.2.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that compound.

7.3 Internal standard calibration procedure. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with acetonitrile. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Analyze each calibration standard (5 to 25 μL for HPLC and 2 to 5 μL for GC) and tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

Eq. 1 $RF = (A_s C_{is})/(A_{is} C_s)$

where:

A_s = Response for the parameter to be measured.
A_{is} = Response for the internal standard.
C_{is} = Concentration of the internal standard, (μg/L).
C_s = Concentration of the parameter to be measured, (μg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.3.3 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than 10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

7.4 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of the rapid advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 The laboratory must spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 Select a representative spike concentration for each compound to be measured. Using stock standards, prepare a quality control check sample concentrate in acetronitrile 1000 times more concentrated than the selected concentrations. Quality control check sample concentrates, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

8.2.2 Using a pipet, add 1.00 mL of the check sample concentrate to each of a minimum of four 1000-mL aliquots of reagent water. A representative wastewater may be used in place of the reagent water, but one or more additional aliquots must be analyzed to determine background levels, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 10.

8.2.3 Calculate the average percent recovery, (R), and the standard deviation of the percent recovery (s), for the results. Wastewater background corrections must be made before R and s calculations are performed.

8.2.4 Using Table 3, note the average recovery (X) and standard deviation (p) expected for each method parameter. Compare these to the calculated values for R and s. If $s > 2p$ or $|X-R| > 2p$, review potential problem areas and repeat the test.

8.2.5 The U.S. Environmental Protection Agency plans to establish performance criteria for R and s based upon the results of interlaboratory testing. When they become available, these criteria must be met before any samples may be analyzed.

8.3 The analyst must calculate method performance criteria and define the performance of the laboratory for each spike concentration and parameter being measured.

8.3.1 Calculate upper and lower control limits for method performance:

Upper Control Limit (UCL) = R + 3 s
Lower Control Limit (LCL) = R — 3 s

where R and s are calculated as in Section 8.2.3.

The UCL and LCL can be used to construct control charts[7] that are useful in observing trends in performance. The control limits above must be replaced by method performance criteria as they become available from the U.S. Environmental Protection Agency.

8.3.2 The laboratory must develop and maintain separate accuracy statements of laboratory performance for wastewater samples. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analysis of four aliquots of wastewater as described in Section 8.2.2, followed by the calculation of R and s. Alternately, the analyst may use four wastewater data points gathered through the requirement for continuing quality control in Section 8.4. The accuracy statements should be updated regularly[7].

8.4 The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. The frequency of spiked sample analysis must be at least 10% of all samples or one sample per month, whichever is greater. One aliquot of the sample must be spiked and analyzed as described in Section 8.2. If the recovery for a particular parameter does not fall within the control limits for method performance, the results reported for that parameter in all samples processed as part of the same set must be qualified as described in Section 14.3. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

8.5 Before processing any samples, the analyst should demonstrate through the analysis of a one-liter aliquot of reagent water, that all glassware and reagents interferences are under control. Each time a set of samples is extracted or there is a change in reagents, a laboratory reagent blank should be processed as a safeguard against laboratory contamination.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as chromatography with a dissimilar column or detector must be used. This may include the use of a mass spectrometer. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[8] should be followed, except that the bottle must not be prewashed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 The samples must be iced or refrigerated at 4°C from the time of collection until extraction. PAHs are known to be light sensitive, therefore, samples, extracts and standards should be stored in amber or foil wrapped bottles in order to minimize photolytic decomposition. Fill the sample bottle and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample. U.S. Environmental Protection Agency methods 330.4 and 330.5 may be used for measurement of residual chlorine[9]. Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days, and analysis completed within 40 days of extraction[2].

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a two liter separatory funnel.

10.2 Add 60 mL methylene chloride to the sample bottle, seal, and shake 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of ten minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle, rinse and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5 mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extracts will be stored longer than two days, they should be transferred to Teflon-sealed screw-cap bottles and protected from light.

10.7 Determine the original sample volume by refilling the sample bottle to the mark and transferring the water to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. The cleanup procedures recommended in this method have been used for the analysis of various clean waters and industrial effluents. If particular circumstances demand the use of an alternative cleanup procedure, the analyst must determine the elution profile and demonstrate that the recovery of each compound of interest is no less than 85%.

11.2 Before the silica gel cleanup technique can be utilized, the extract solvent must be exchanged to cyclohexane. Add a 1- to 10- mL aliquot of sample extract (in methylene chloride) and a boiling chip to a clean K-D concentrator tube. Add 4 mL cyclohexane and attach a micro-Snyder column. Prewet the micro-Snyder column by adding 0.5 mL methylene chloride to the top. Place the micro-K-D apparatus on a boiling (100 C) water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5 to 10 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of the liquid reaches 0.5 mL, remove the K-D apparatus and allow it to drain for at least 10 minutes while cooling. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum of cyclohexane. Adjust the extract volume to about 2 mL.

11.3 Silica gel column cleanup for PAHs.

11.3.1 Prepare a slurry of 10g activated silica gel in methylene chloride and place this in a 10-mm ID chromatography column. Gently tap the column to settle the silica gel and elute the methylene chloride. Add 1 to 2 cm of anhydrous sodium sulfate to the top of the silica gel.

11.3.2 Preelute the column with 40 mL of pentane. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, transfer the 2 mL of cyclohexane sample extract onto the column, using an additional 2 mL of cyclohexane to complete the transfer.

11.3.3 Just prior to exposure of the sodium sulfate layer to the air, add 25 mL pentane and continue elution of the column. Discard the pentane eluate.

11.3.4 Elute the column with 25 mL of methylene chloride/pentane (4 + 6) (V/V) and collect the eluate in a 500-mL K-D flask equipped with a 10-mL concentrator tube. Elution of the column should be at a rate of about 2 mL/min.

11.3.5 Concentrate the collected fraction to less than 10 mL by K-D techniques as in Section 10.6, using pentane to rinse the walls of the glassware. Proceed with HPLC or GC analysis.

12. High Performance Liquid Chromatography (HPLC)

12.1 To the extract in the concentrator tube, add 4 mL of acetonitrile and a new boiling chip, then attach a micro-Snyder column. Increase the temperature of the hot water bath to 95 to 100°C. Concentrate the solvent as in Section 10. After cooling, remove the micro-Snyder column and rinse its lower joint into the concentrator tube with about 0.2 mL acetonitrile. Adjust the extract volume to 1.0 mL.

12.2 Table 1 summarizes the recommended HPLC column materials and operating conditions for the instrument. This table includes retention times, capacity factors, and MDL that were obtained under these conditions. The UV detector is recommended for the determination of naphthalene, acenaphthylene, acenapthene, and fluorene, and the fluorescence detector is recommended for the remaining PAHs. Examples of the parameter separations achieved by this HPLC column are shown in Figures 1 and 2. Other HPLC columns, chromatrograpic conditions or detectors may be used if the requirements of Section 8.2 are met.

12.3 Calibrate the system daily as described in Section 7.

12.4 If the internal standard approach is being used, the internal standard must be added to sample extract and mixed thoroughly, immediately, before injection into the instrument.

12.5 Inject 5 to 25 μL of the sample extract using a high pressure syringe or a constant volume sample injection loop. Record the volume injected to the nearest 0.1 μL, and the resulting peak size in height or area units. Re-equilibrate the liquid chromatographic column at the initial gradient conditions for at least 10 minutes between injections.

12.6 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.7 If the peak height or area exceeds the linear range of the system, dilute the extract with acetonitrile and reanalyze.

12.8 If the peak area measurement is prevented by the presence of interferences, further cleanup is required.

13. Gas Chromatography

13.1 The packed column GC procedure will not resolve certain isomeric pairs as indicated in Section 1.3 and Table 2. The liquid chromatographic procedure (Section 12) must be used for these materials. Capillary (open-tubular) columns may be used if the relative standard deviations of responses for replicate injections are demonstrated to be less than 6% and the requirements of Section 8.2 are met.

13.2 To achieve maximum sensitivity with this method, the extract must be concentrated to 1.0 mL. Add a clean boiling chip to the methylene chloride extract in the concentrator tube. Attach a two-ball micro-Snyder column. Prewet the micro-Snyder column by adding about 0.5 mL of methylene chloride to the top. Place the micro K-D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation the balls will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus. Drain and cool for at least 10 minutes. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a small volume of methylene chloride. Adjust the final volume to 1.0 mL and stopper the concentrator tube.

13.3 Table 2 describes the recommended GC column and operating conditions for the instrument. This table includes retention times that were obtained under these conditions. An example of the parameter separations achieved by this column is shown in Figure 3. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met. Capillary (open-tubular) columns may also be used if the relative standard deviations of responses for replicate injections are demonstrated to be less than 6% and the requirements of Section 8.2 are met.

13.4 Calibrate the GC system daily as described in Section 7.

13.5 If the internal standard approach is being used, add the internal standard to sample extract and mix thoroughly, immediately, before injection into the instrument.

13.6 Inject 2 to 5 μL of the sample extract using the solvent-flush technique(10). Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, and the resulting peak size in area or peak height units.

13.7 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

13.8 If the response for the peak exceeds the working range of the system, dilute the extract and reanalyze.

13.9 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

14. Calculations

14.1 Determine the concentration of individual parameters in the sample.

14.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Eq. 2. Concentration, } \mu g/L = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

A = Amount of material injected, in nanograms.
V_i = Volume of extract injected (μL).
V_t = Volume of total extract (μL).
V_s = Volume of water extracted (mL).

14.1.2 If the internal standard calibration procedure was used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Eq. 3. Concentration, } \mu g/L = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

A_s = Response for the parameter to be measured.
A_{is} = Response for the internal standard.
I_s = Amount of internal standard added to each extract (μg).
V_o = Volume of water extracted, in liters.

14.2 Report results in micrograms per liter without correction for recovery data. When duplicate and spiked samples are analyzed, report all data obtained with the sample results.

14.3 For samples processed as part of a set where the laboratory spiked sample recovery falls outside of the control limits established in Section 8.4, data for the affected parameters must be labeled as suspect.

15. Method Performance

15.1 Method detection limits - The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero(1). The MDL concentrations listed in Table 1 were obtained using reagent water(1). Similar results were achieved using representative wastewaters. MDL for the GC approach were not determined.

15.2 This method has been tested for linearity of recovery from spiked reagent water and has been demonstrated to be applicable over the concentration range from 8 x MDL to 800 x MDL(1), with the following exception: benzo(ghi)perylene recovery at 80 x and 800 x MDL were low (35% and 45% respectively).

15.3 In a single laboratory (Battelle Columbus Laboratories), using spiked wastewater samples, the average recoveries presented in Table 3 were obtained(2). Each spiked sample was analyzed in triplicate on two separate days. The standard deviation of the precent recovery is also included in Table 3.

15.4 The U.S. Environmental Protection Agency is in the process of conducting an interlaboratory method study to fully define the performance of this method.

References

1. See Appendix A.
2. "Determination of Polynuclear Aromatic Hydrocarbons in Industrial and Municipal Wastewaters," Report for EPA Contract 68-03-2624 (In preparation).
3. ASTM Annual Book of Standards, Part 31, D 3694. "Standard Practice for Preparation of Sample Containers and for Preservation," American Society for Testing and Materials, Philadelphia, PA, p. 679, 1980.
4. "Carcinogens - Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug. 1977.
5. "OSHA Safety and Health Standards, General Industry," (29CFR-1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).
6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.
7. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," EPA-600/4-79-019, U.S. Environmental Protection Agency Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.
8. ASTM Annual Book of Standards, Part 31, D 3370, "Standard Practice for Sampling Water," American Society for Testing and Materials, Philadelphia, PA, p. 76, 1980.

9. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA 600-4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

10. Burke, J. A., "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48*, 1037 (1965).

11. Cole, T., Riggins, R., and Glaser, J., "Evaluation of Method Detection Limits and Analytical Curve for EPA Method 610 - PNAs," International Symposium on Polynuclear Aromatic Hydrocarbons, 5th, Battelle Columbus Laboratory, Columbus, Ohio (1980).

Table 1. High Performance Liquid Chromatography Conditions and Method Detection Limits

Parameter	*Retention Time (min)*	*Capacity Factor (k')*	*Method Detection Limit (μg/L)*[a]
Naphthalene	*16.6*	*12.2*	*1.8*
Acenaphthylene	*18.5*	*13.7*	*2.3*
Acenaphthene	*20.5*	*15.2*	*1.8*
Fluorene	*21.2*	*15.8*	*0.21*
Phenanthrene	*22.1*	*16.6*	*0.64*
Anthracene	*23.4*	*17.6*	*0.66*
Fluoranthene	*24.5*	*18.5*	*0.21*
Pyrene	*25*[illegible]	*19.1*	*0.27*
Benzo(a)anthracene	[illegible]	*21.6*	*0.013*
Chrysene	*2*[illegible]*.3*	*22.2*	*0.15*
Benzo(b)fluoranthene	*31.6*	*24.0*	*0.018*
Benzo(k)flouranthene	*32.9*	*25.1*	*0.017*
Benzo(a)pyrene	*33.9*	*25.9*	*0.023*
Dibenzo(a,h)anthracene	*35.7*	*27.4*	*0.030*
Benzo(ghi)perylene	*36.3*	*27.8*	*0.076*
Indeno(1,2,3-cd)pyrene	*37.4*	*28.7*	*0.043*

HPLC conditions: Reverse phase HC-ODS Sil-X 2.6 mm x 250 mm Perkin-Elmer column; isocratic elution for 5 min using acetonitrile/water (4 : 6), then linear gradient elution to 100% acetonitrile over 25 minutes; flow rate is 0.5 mL/min. If columns having other internal diameters are used, the flow rate should be adjusted to maintain a linear velocity of 2 mm/sec.

[a] The method detection limit for naphthalene, acenaphthylene, acenaphthene, and fluorene were determined using a UV detector. All others were determined using a fluorescence detector.

Table 2. Gas Chromatographic Operating Conditions and Retention Times

Parameter	*Retention Time (min)*
Naphthalene	*4.5*
Acenaphthylene	*10.4*
Acenaphthene	*10.8*
Fluorene	*12.6*
Phenanthrene	*15.9*
Anthracene	*15.9*
Fluoranthene	*19.8*
Pyrene	*20.6*
Benzo(a)anthracene	*20.6*
Chrysene	*24.7*
Benzo(b)fluoranthene	*28.0*
Benzo(k)fluoranthene	*28.0*
Benze(a)pyrene	*29.4*
Dibenzo(a,h)anthracene	*36.2*
Indeno(1,2,3-cd)pyrene	*36.2*
Benzo(ghi)perylene	*38.6*

GC conditions: Chromosorb W-AW-DCMS (100/120 mesh) coated with 3% OV-17, packed in a 1.8 m long x 2 mm ID glass column, with nitrogen carrier gas at a flow rate of 40 mL/min. Column temperature was held at 100°C for 4 min, then programmed at 8°/minute to a final hold at 280°C.

Table 3 Single Operator Accuracy and Precision

Parameter	*Average Percent Recovery*	*Standard Deviation %*	*Spike Range (μg/L)*	*Number of Analyses*	*Matrix Types*
Acenaphthene	88	5.7	11.6-25	24	4
Acenaphthylene	93	6.4	250-450	24	4
Anthracene	93	6.3	7.9-11.3	24	4
Benzo(a)anthracene	89	6.9	0.64-0.66	24	4
Benzo(a)pyrene	94	7.4	0.21-0.30	24	4
Benzo(b)fluoranthene	97	12.9	0.24-0.30	24	4
Benzo(ghi)perylene	86	7.3	0.42-3.4	24	4
Benzo(k)fluoranthene	94	9.5	0.14-6.2	24	4
Chrysene	88	9.0	2.0-6.8	24	4
Dibenzo(a,h)anthracene	87	5.8	0.4-1.7	24	4
Fluoranthene	116	9.7	0.3-2.2	24	4
Fluorene	90	7.9	6.1-23	24	4
Indeno(1,2,3-cd)pyrene	94	6.4	0.96-1.4	24	4
Naphthalene	78	8.3	20-70	24	4
Phenanthrene	98	8.4	3.8-5.0	24	4
Pyrene	96	8.5	2.3-6.9	24	4

Column: HC-ODS SIL-X
Mobile phase: 40% to 100% Acetonitrile in water
Detector: Ultra violet at 254nm

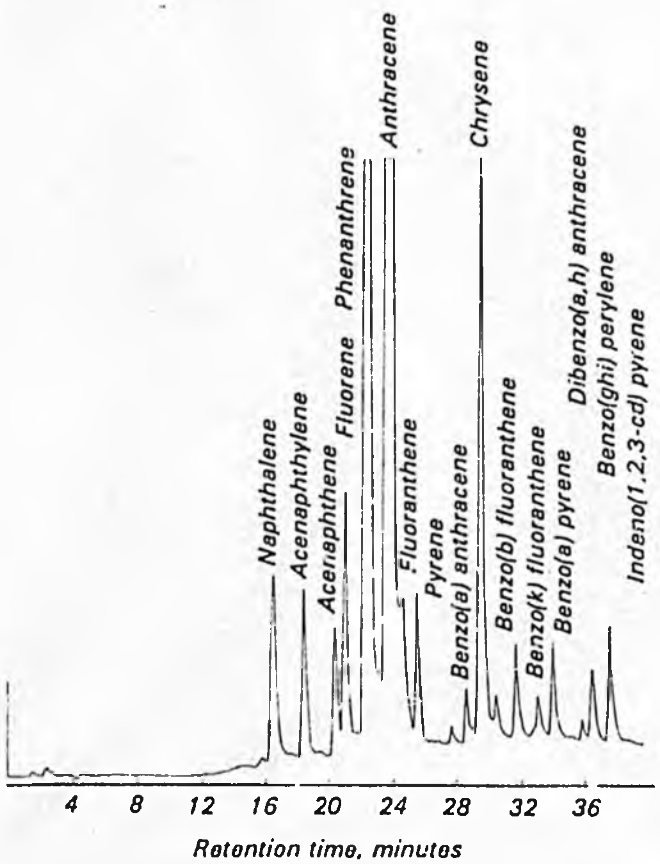


Figure 1. *Liquid chromatogram of polynuclear aromatic hydrocarbons.*

Column: HC-ODS SIL-X
Mobile phase: 40% to 100% Acetonitrile in water
Detector: Fluorescence

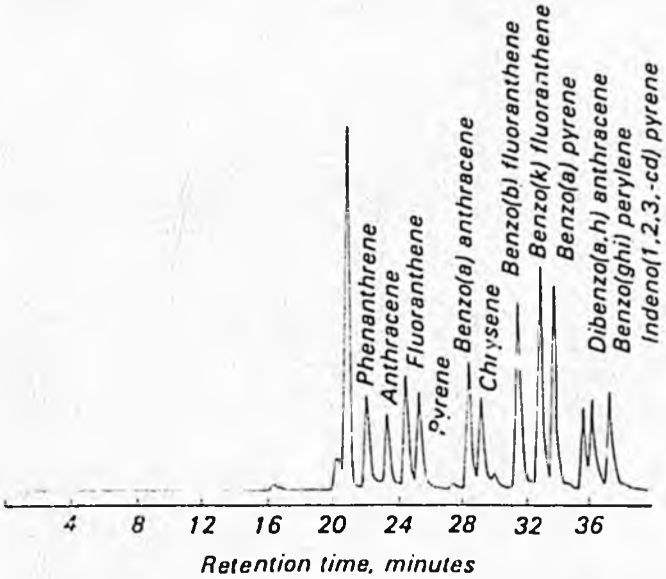


Figure 2. *Liquid chromatogram of polynuclear aromatic hydrocarbons.*

Column: 3% OV-17 on Chromosorb W-AW-DCMS
Program: 100°C. 4 min..8° per min. to 280°C.
Detector: Flame ionization

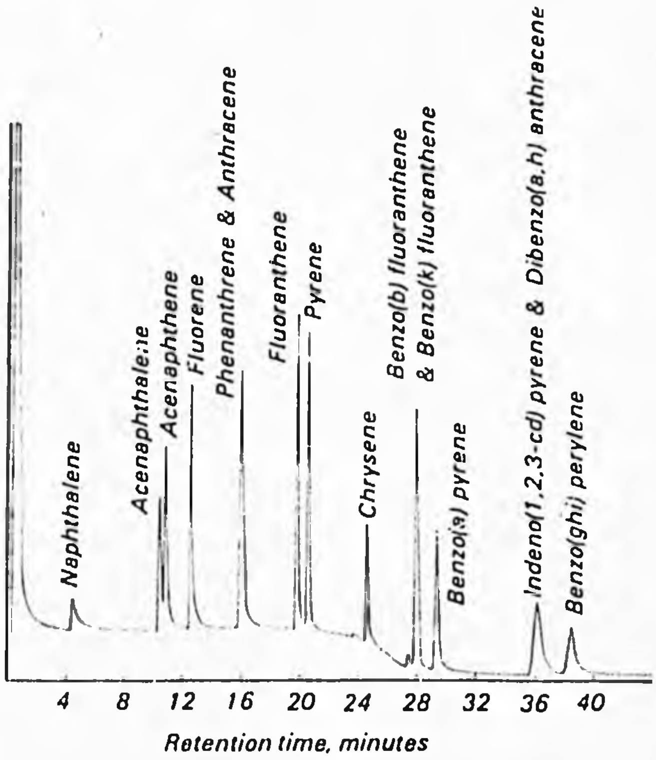
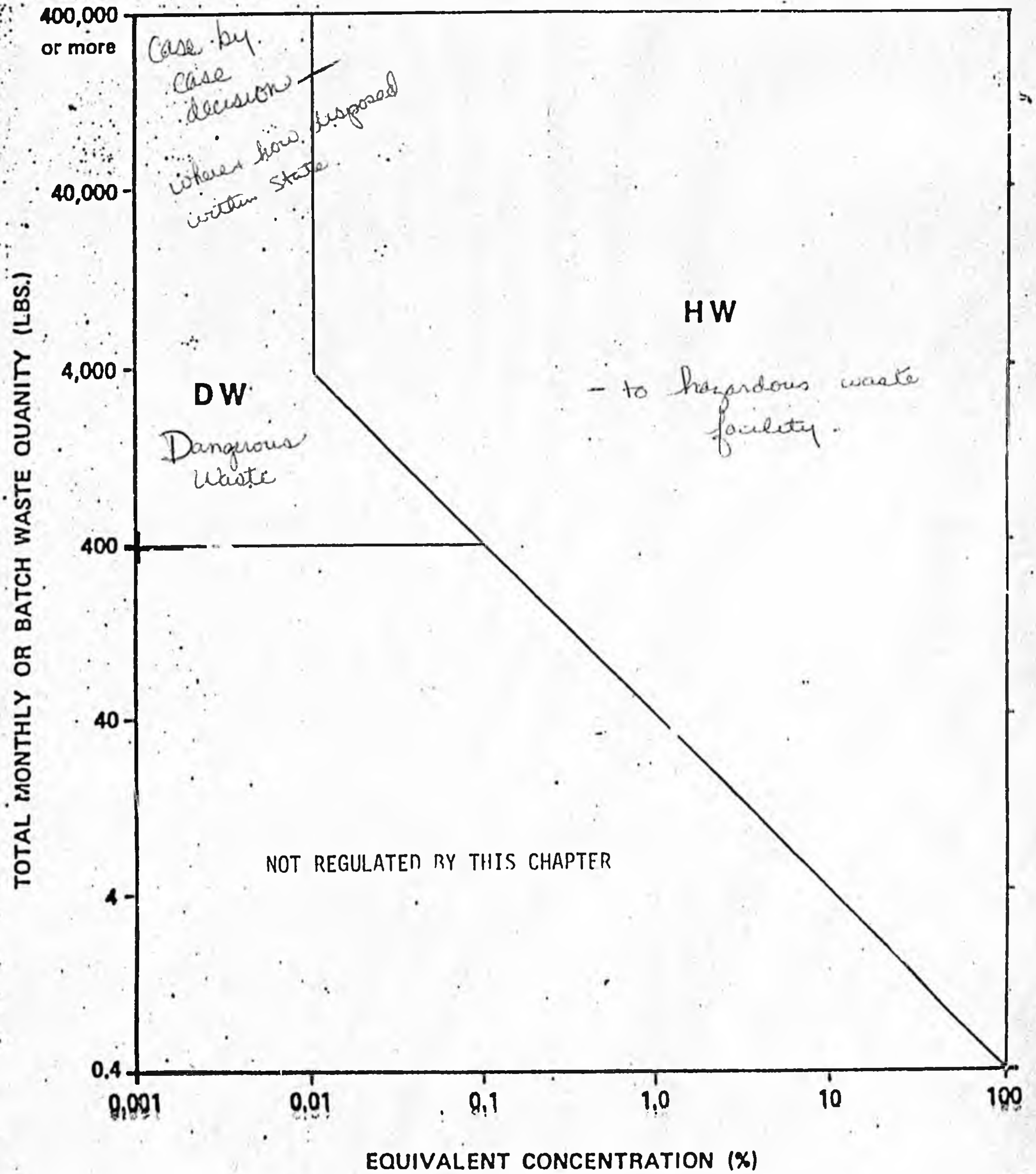


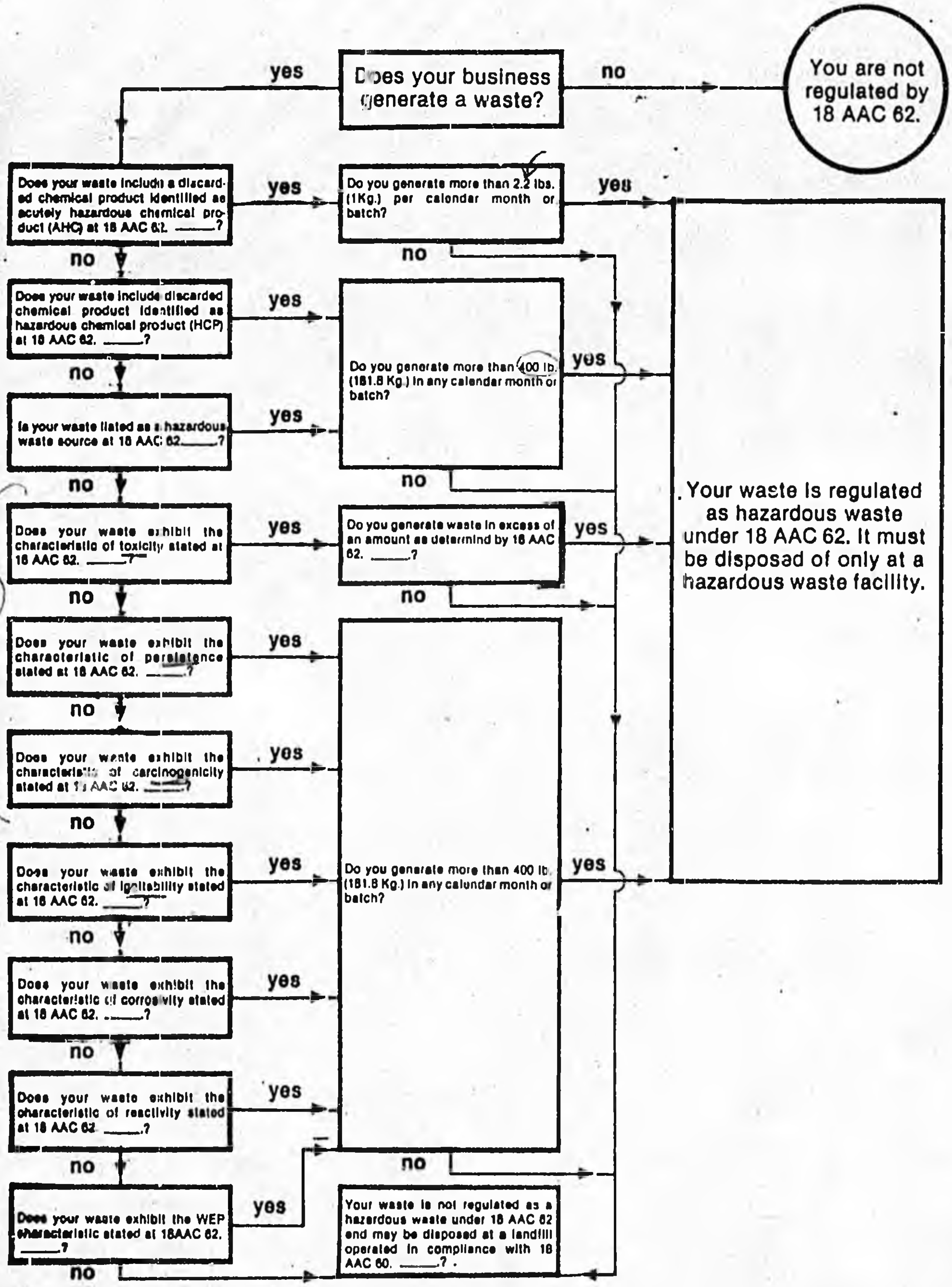
Figure 3. *Gas chromatogram of polynuclear aromatic hydrocarbons.*

TOXIC WASTE MIXTURES GRAPH.



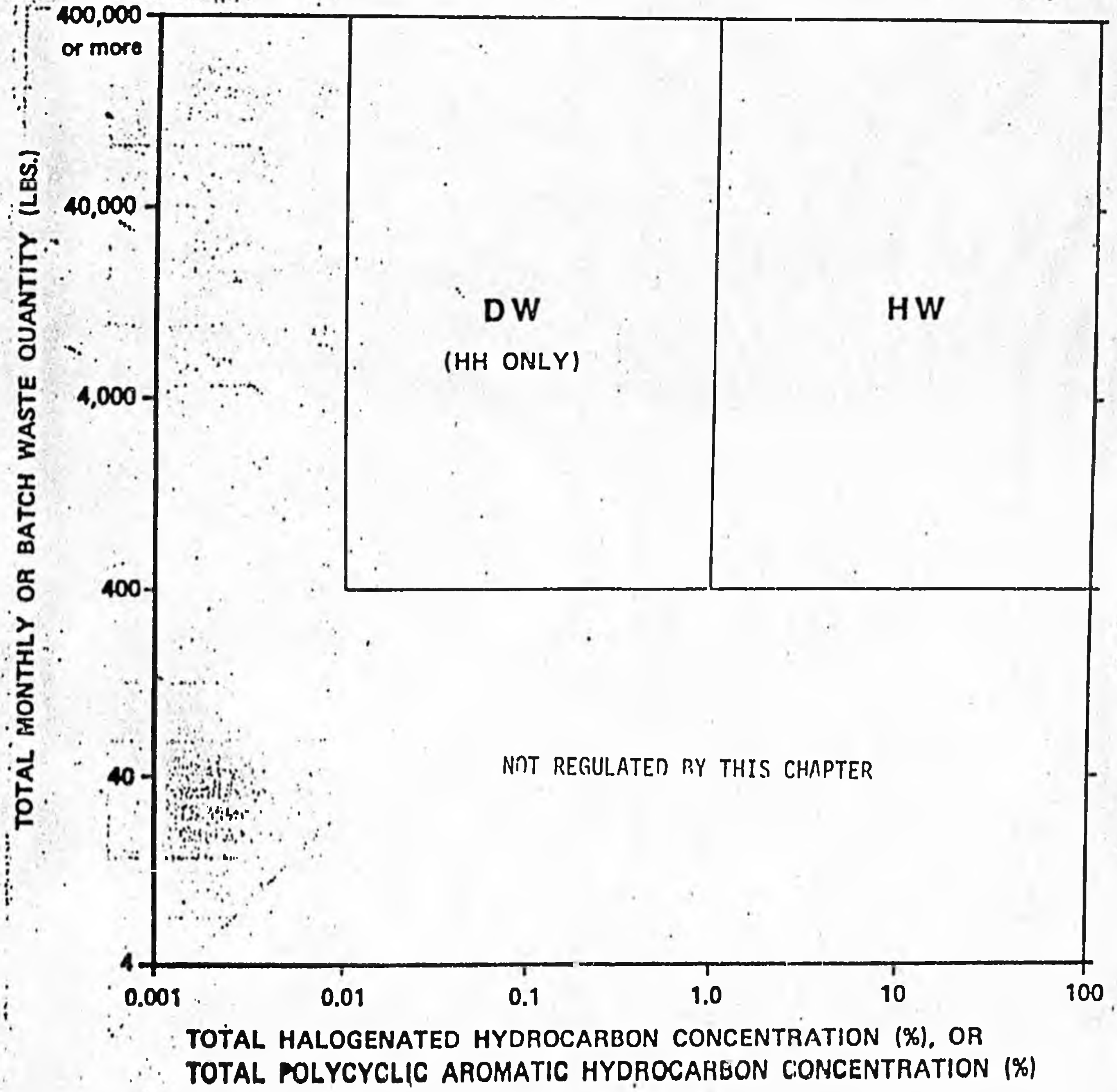
CHAPTER 62 HAZARDOUS WASTE

APPENDIX III *FLOW CHART FOR DETERMINING WHETHER WASTE IS HAZARDOUS.*



different than EPA

PERSISTENT WASTE MIXTURES GRAPH.



STATE OF ALASKA

DEPT. OF ENVIRONMENTAL CONSERVATION

BILL SHEFFIELD, GOVERNOR

POUCH O
JUNEAU, ALASKA 99811
PHONE:

465-2600

March 30, 1983

Mr. James A. Palmer
Joint Committee on Oil & Gas
Pouch V
Juneau, Alaska 99811

Dear Mr. Palmer:

Upon the request of several Hazardous Waste Advisory Work Group (HWAWG) members, we are scheduling our next meeting for April 11, 1983. Looking at the enclosed RCRA Authorization milestone dates submitted to the U.S. EPA, you will see it would be very difficult to accommodate any future delays without jeopardizing our proposed authorization schedule.

Our April 11, 1983, meeting will focus on the HWAWG members' comments to the January 28, 1983, draft hazardous waste regulations. Since the department is planning to publish notice of the draft regulations in mid-April, your comments are important at this time for possible inclusion into our final draft.

The meeting is scheduled to begin at 9:00 a.m. at the Community Center Building, Sixth and G Streets, Anchorage. We are appreciative of your past cooperation and assistance and look forward to a productive April 11, 1983, HWAWG meeting.

Sincerely,

Richard A. Nevé

Richard A. Nevé
Commissioner

Enclosure

Joe C

STATE OF ALASKA

DEPT. OF ENVIRONMENTAL CONSERVATION

BILL SHEFFIELD, GOVERNOR

465-2640

POUCH O — JUNEAU 99811

February 28, 1983

Mr. Ron Kreizenbeck
Director, Alaska Operations Office
U.S. Environmental Protection Agency
Pouch O
Juneau, Alaska 99811

Dear Mr. Ron Kreizenbeck:

As we discussed in our February 23, 1983, meeting, the department would appreciate the services of the U.S. EPA contractor in developing Alaska's Resource Conservation and Recovery Act (RCRA) Authorization application. We will be working with your office through March 1983 to specify the contractor's scope of work. In addition to the above discussion, we outlined the RCRA authorization milestones to be as follows:

1.	U.S. EPA contractor to begin assisting ADEC in developing authorization application	April 1, 1983
2.	Next Hazardous Waste Advisory Work Group meeting to discuss the group's draft regulation comments	April 8, 1983
3.	Draft Hazardous Waste Regulations Public Notice	April 15, 1983
4.	Public hearings	May 16-20, 1983
5.	Evaluate public hearing comments and revise regulations	June 17, 1983
6.	Department regulation adoption order	July 15, 1983
7.	Send RCRA authorization package to U.S. EPA	July 15, 1983

If you have any questions, please call me or David DiTraglia of my staff.

Sincerely,

Joe Cladouhos
Director, EQM

Commissioner Neve'
Stan Hungerford

EXXON COMPANY, U.S.A.
POUCH 6601 • ANCHORAGE, ALASKA 99502 (907) 276-4552

ALASKA OPERATIONS
WESTERN DIVISION

RICHARD H. WEAVER
OPERATIONS MANAGER

March 9, 1983

Mr. Richard Neve, Commissioner
Alaska Department of
Environmental Conservation
Pouch O
Juneau, AK 99811

Dear Mr. Neve:

As you are probably aware, the recent newspaper article relating to the toxicity of drilling muds (Anchorage Daily News, March 5, 1983) concerns me a great deal. We understand that you were unfortunately misquoted in this article. Regrettably, the implication of the article is that all drilling muds "are very toxic", including those that may be used for exploratory drilling in the St. George Basin lease sale area.

The drilling muds which Exxon uses in offshore operations may be classified in the eight "generic mud" types developed in conjunction with the Environmental Protection Agency administered National Pollutant Discharge Elimination System (NPDES) permitting process. These "generic muds" have been extensively tested and shown to have very low toxicity, even though some of the minor components may be more toxic than the whole mud. Discharges of "generic muds" are rapidly dispersed and have only a minor and temporary local effect. No significant impact on open ocean water quality has ever been noted.

Studies of the effects of drilling mud discharge have produced a large body of knowledge about the nature of these discharges and their environmental impact. It is, of course, an ongoing process and Exxon as well as others in the petroleum industry and government are continually adding to the available information. This information is available to the public, as well as to state and federal agencies. Our affiliate, Exxon Production Research Company, has senior research personnel who are experts in this field. If you, your staff, and any others desire, I would be pleased to arrange for a discussion with our technical experts on the environmental effects of drilling related discharges. For your information, I have attached a paper by Dr. R. C. Ayers, Jr., Senior Research Associate of Exxon Production Research Company, entitled "The Fate and Effects of Drilling Discharge in the Marine Environment." This paper was presented at the American Society for Environmental Education's "Technical Conference on Georges Bank Hydrocarbon Exploration and Development." I have also attached another recent paper by Dr. Ayers entitled "The Generic Mud Concept of Offshore Drilling for NPDES Permitting" which provides additional pertinent information regarding the toxicity of the "generic muds."

I believe that a meeting to discuss the fates and effects of drilling fluids has great potential for the exchange of information on this important topic. I will be in touch with you to discuss your desire for this exchange.

R. H. Weaver

RHW:jlp
CMR:ENGR039

Attachment

xc: Senator Bettye Fahrenkamp
Senate Resource Committee

IADC/SPE

IADC/SPE 11399

The Generic Mud Concept for Offshore Drilling for NPDES Permitting

by R.C. Ayers Jr. and T.C. Sauer Jr., *Exxon Production Research Co.*, and P.W. Anderson. *U.S. Environmental Protection Agency*

This paper was presented at the IADC/SPE 1983 Drilling Conference held in New Orleans, Louisiana, February 20-23, 1983. The material is subject to correction by the author. Permission to copy is restricted to an abstract of not more than 300 words. Write SPE, 6200 North Central Expressway, Drawer 64706, Dallas, Texas 75206.

ABSTRACT

U. S. Environmental Protection Agency (EPA), Region II granted the first offshore drilling NPDES permits to the mid-Atlantic Operators in 1978. The generic or standard mud concept was developed by the Offshore Operators Committee with EPA Region II in order to provide the agency with an understanding of and control over mud components and discharges without requiring the operators to perform redundant bioassay and chemical tests every time mud was discharged. The generic mud concept has subsquently been incorporated into permits issued by EPA Regions I and IX. Regions III and X are considering the use of generic muds in future permits.

Eight basic mud types were defined which encompassed virtually all water base muds used on the Outer Continental Shelf (OCS). A bioassay test procedure, adapted from the EPA/Corp of Engineers' Procedure for dredged material, was developed and a test program conducted on samples of field muds, representative of each of the eight basic types. Operators were then allowed to discharge muds of the eight types without conducting additional bioassays. Results of the test program indicate that all eight generic muds demonstrated low toxicity.

The eight generic muds contain primarily major components and do not consider specialty chemicals sometimes required to remedy special drilling problems (i.e., lost returns materials, lubricants, defoamers, etc.). This situation is being handled by the use of an "approved additive list" naming additives for use which do not significantly affect mud toxicity.

The API is developing a standard bioassay procedure based on the Region II protocol for testing of specialty chemicals in standard muds, and service companies are already using parts of this procedure to gain approval for use of their products.

References and illustrations at end of paper.

INTRODUCTION

In 1978 the U.S. Environmental Protection Agency (EPA), Region II granted the first offshore drilling NPDES permits to operators drilling on leases in the Baltimore Canyon. As a permit condition, the operators were required to perform a jointly funded drilling mud bioassay program.

Prior to initiating the test program, the Offshore Operator Committee Task Force on Environmental Science with EPA Region II developed an acceptable drilling mud bioassay procedure and a spectrum of eight general mud types that included essentially all water base compositions.[1] Previously, the agency had not recognized differences in water base mud systems and had classified all muds in only two categories, oil or water based. The generic mud concept and the EPA Region II bioassay procedure evolved from this effort.

The approach has proven to be practical and the generic mud concept, supported by the Region II bioassay results, has subsequently been incorporated into permits issued by EPA Regions I and IX. EPA Regions III and X are considering the use of generic muds in future permits.

The Mid-Atlantic Bioassay Program was funded by the Mid-Atlantic Operators. The bioassay tests were conducted by Energy Resources Company and Normandeau Associates under the supervision of Exxon Production Research Company.

GENERIC MUD CONCEPT

The generic muds were identified by reviewing the permit requests and selecting the minimum number of mud systems which would cover all those named by the prospective permittees. Eight different mud systems were identified which encompass virtually all water based muds used on the OCS (Table 1). As opposed to naming a set concentration for each component in each mud system, concentration ranges were specified to allow the operators sufficient flexibility to drill safely.

In the eight generic mud systems, only major components are specified. Specialty additives (e.g. lost circulation materials, lubricity agents, etc.) needed for special drilling situations are not named. If an unanticipated need for an additive of this type arose, the operator was required to submit to the agency chemical composition, usage rates, and toxicity data on the additive prior to its use. Based on this information, the EPA Regional Administrator would either approve or disapprove discharge of mud containing the additive on a case by case basis. If there was a continuing need for the additive, the operator could then submit bioassay data on mud containing the additive. Discharge would be allowed if the additive did not greatly increase mud toxicity. Once an additive became "approved" in this way, future discharge of muds containing the additive would be allowed without conducting additional bioassays. It should be noted that any of the generic muds may contain one or more specialty additives; however, the presence of an approved specialty additive in a generic mud does not change the generic mud type.

BIOASSAY TESTS

Ninety-six hour bioassay tests are a standard method for determining acute toxicity. In these tests, sets of test organisms are subjected to different concentrations of toxicant for 96 hours and the number of organisms surviving at each concentration within the 96-hour time frame is noted. A plot of mortality as a function of toxicant concentration is prepared and the concentration required to kill 50% of the test organisms is taken off the plot (Figure 1). This is called the 96-hour LC$_{50}$. Bioassay tests are useful because they are comparatively rapid and inexpensive, and produce a single value to help make a judgement on toxicity.

Note that a high LC$_{50}$ implies low toxicity because a high concentration is required to kill 50% of the test organisms. On the other hand, a low LC$_{50}$ implies high toxicity because a small concentration of the toxicant is needed to kill 50% of the test organisms. Table 2 presents a classification of toxicity grades that is commonly used.[3,4] LC$_{50}$ values in excess of 10,000 ppm are considered practically nontoxic.

Bioassay data provides an estimate of toxic concentrations; however, this information alone is insufficient to predict environmental impact. It is equally important to know what concentrations actually exist in the marine environment.

EPA REGION II BIOASSAY PROCEDURE

The vast majority of bioassays on marine organisms have been conducted on toxicants that are soluble in seawater. Because drilling mud contains solid particles, a special procedure had to be developed. The EPA Region II test procedure[1] was adapted from the U.S. Army Corps of Engineers/EPA Bioassay procedure[5] for determining the toxicity of dredged materials and compliance with the ocean dumping criteria.

The test divides the drilling fluid into three phases: the liquid phase, the suspended particulate phase, and the solid phase. These phases are designed to represent the anticipated conditions that organisms would be exposed to when drilling mud is discharged into the ocean. Certain drilling fluid components are water soluble and will dissolve in the water column, others are fine particulates which would stay suspended, and still other material would settle rapidly to the bottom.

The procedure for phase separation follows the schematic in Figure 2. To prepare the three test phases, a 1:4 ratio by volume of mud to seawater is mixed for 30 minutes. The pH is adjusted to that near seawater (pH=7.8-9.0) by the addition of acetic acid. The slurry is allowed to settle for one hour. A portion of the supernatant is filtered through a 0.45-micron filter. The filtrate is designated as the "liquid phase". The remaining unfiltered supernatant of the slurry is the "suspended particulate phase", while the "solid phase" is the settled solid material at the bottom of the mixing vessel.

Two species of test organisms are used in the bioassays. The mysid shrimp, Mysidopsis bahia, is the test organism for the liquid and suspended particulate phases. This species has been shown to be exceptionally sensitive to toxic substances and is considered to be a representative marine organism for bioassay testing by EPA.[6] An LC$_{50}$ is determined in both the liquid and suspended particulate phase bioassay tests. The solid phase test organism is the hard shell clam, Mercenaria mercenaria. No LC$_{50}$ is determined in the solid phase test. In these tests, clams are exposed to a 15 mm layer of drilling mud solids (solid phase) placed on top of a reference sediment. After 10 days of exposure, percent survival is determined, and the results are statistically compared with those obtained from the same test with no mud solids present.

BIOASSAY TEST RESULTS

The composition, properties, metals analyses, and oil and grease analyses of representative samples of the eight generic muds tested are shown in Table 3. The corresponding bioassay test results are shown in Table 4.[7,8] During the bioassay program, only two muds (mud #2 and mud #7) were used in wells drilled in the Baltimore Canyon. The other mud samples were obtained from wells drilled in the Gulf of Mexico, Utah, Texas, Mississippi, Wyoming, and Louisiana. In some of the muds tested (Table 1), a few of the component concentrations fall slightly outside the range of those listed in the eight generic muds. Also in some of the test muds, low concentrations of specialty additives were present.

In Table 4 the LC$_{50}$s for the liquid and suspended particulate phases are expressed as a function of concentration of the phase tested. Two different laboratories (Energy Resources Company and Normandeau Associates) were used to perform the tests. The differences in the LC$_{50}$ results between the laboratories are typical of what one might expect from two independent laboratories and are an indication of the precision of this type of test. In the solid phase tests only mud #2 demonstrated any statistically significant toxicity. In general, the suspended particulate phase of each mud is somewhat more toxic than the liquid phase.

The LC_{50} values expressed in Table 4 are shown as parts per million of phase tested. The phases were prepared by diluting the mud with seawater in a 1:4 ratio prior to testing. To determine a measure of whole mud toxicity for a particular phase, the LC_{50} values shown in Table 4 need to be divided by five. This is perhaps a more realistic indication of mud toxicity. According to the toxicity grade classification (Table 2), all the muds fall in the slightly toxic to practically non-toxic range.

IMPLICATIONS OF BIOASSAY TEST RESULTS

A comparison of liquid and suspended particulate phase bioassay results with results from field dispersion studies permits an estimate of possible toxic effects of mud on pelagic organisms in the open ocean. Figure 3 is a plot of steady state suspended solids concentration versus transport time during a high rate (1,000 bbl per hour) mud discharge study conducted in the Gulf of Mexico.[9] Transport time (distance where concentration was measured/current velocity) represents the time required for a parcel of fluid to travel from the discharge source to a point where the concentration was measured. This type of curve indicates how water column solids concentration decreases with time due to settling and dilution. Also plotted on Figure 3 are the lowest 96-hour LC_{50} values for the suspended particulate phase shown in Table 4, corrected to whole mud values ($LC_{50}/5$). In addition to the values, we have indicated the lowest 96-hour LC_{50} values for drilling muds presented in a recent literature review.[10]

To be a threat to biota in the water column, the LC_{50}s should fall at or below the curve at a time of 96 hours. Note that even the lowest mud 96-hour LC_{50} value (100 ppm) is more than two orders of magnitude above the curve. The only concentrations that exceed even this value are those at low transport times (<20 minutes) which are representative of concentrations near the discharge source. These concentrations exist only for a short time while the discharge is actually occurring. The implication is clear that even the more toxic muds will not cause significant biological effects in the water column.

Field studies[9,11] have shown that most of the solid material settles rapidly near the well site. Concentrations in the sediment at the well site may remain elevated for some time depending on environmental factors (storm frequency, water depth, bottom current velocity) that govern resuspension and bottom transport. Of the three phases tested the solid phase bioassay is the best indicator of impact on benthos; however, the solid phase test does not yield an LC_{50} and interpretation of the results is difficult. There is some concern over the test species used for the solid phase test. The hard shell clam Mercenaria used in the solid phase test is considerably less sensitive than the mysid shrimp used in the liquid or suspended particulate test. Both API and EPA are investigating ways to improve the solid phase test for water base muds.

OIL BASE MUD AND CUTTINGS BIOASSAY

Discharge of oil base muds on the OCS is prohibited; however, discharge of cuttings generated while using oil base muds is allowed provided the cuttings are washed and do not cause a sheen.

In recent years service companies have developed so called "nontoxic" oil base muds. These are paraffinic oil base formulations that show considerably less toxicity in liquid and suspended phase bioassays than oil muds containing diesel. Some companies are suggesting that it may be safe to discharge cuttings generated using these mud systems, even without washing. Since cuttings discharges primarily impact the benthos, a solid phase bioassay would be the most appropriate laboratory test to estimate potential harm. At this time, however, no solid phase bioassay test acceptable to EPA and the operators is available which will accurately measure toxicity of this type of discharge. There is a strong need to develop a solid phase bioassay test that will assist regulators in making accurate judgements regarding the discharge of oily cuttings.

CONCLUSIONS

- The use of generic muds along with approved specialty additives has proved to be a useful concept for both regulators and operators. Regulators have control over toxicity of discharged mud and operators have sufficient flexibility to drill safely.
- The liquid and suspended particulate phase bioassay tests are adequate to evaluate mud toxicity in the water column. Improvements in the procedure can be made, but they are expected to be minor.
- Because of rapid settling and dispersion along with the relatively high 96-hour LC_{50}s observed in the liquid and suspended particulate phase bioassays, it is clear that drilling mud discharges do not cause acute water column biological effects in the open ocean. This in true even for the more toxic muds.
- An improved solid phase bioassay test is needed for water base muds. The test should employ a more sensitive test organism and yield an LC_{50}.
- A bioassay test for oily cuttings generated while drilling with oil muds needs to be developed. This is especially important today due to the rapid development of the 'low toxicity' oil muds.

ACKNOWLEDGEMENTS

The Offshore Operators Committee Task Force on Environmental Science made a major contribution to this entire effort. We also wish to recognize the contribution of Messrs. O. C. Rath and C. L. Prokop of Exxon U.S.A. who were instrumental in developing the generic mud concept, Dr. Royal Naudau of EPA who helped develop the bioassay procedure, of Drs. Curt Rose and Tim Ward of ERCO and Ken Simon of Normandeau Associates who performed the bioassay tests, and Mike Foster of SCR Inc., Houston who conducted the metals analyses. We are also indebted to Dr. Maurice Jones of IMCO and Bill Salins of the Petroleum Equipment Suppliers Association who provided most of the mud samples for testing.

REFERENCES

1. EPA Region II. 1978. Drilling mud bioassay test procedures to be employed under EPA, Region II, offshore exploratory drilling permits.

2. Jones, M. and M. Hulse. 1982. Drilling fluid bioassays and the OCS. Oil and Gas Journal, June 21, 1982: 241-244.

3. Sprague, J. B. 1973. The ABCs of pollutant bioassays using fish. In Cairns, J. and K. L. Dickson (eds), Proc., Biological methods for the assessment of water quality. Amer. Soc. of Testing and Materials, ASTM Tech. Publ. 528: p. 6-30.

4. IMCO/FAO/UNESCO/WMO. 1969. Group of experts on the scientific aspects of marine pollution, first session report. Water Res. 3:995-1005.

5. U.S. Environmental Protection Agency/Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material. 1977. Ecological evaluation of proposed dredged material into ocean waters. Environmental Effects Laboratory, U.S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi.

6. Nimmo, D. R. and T. L. Hamaker. 1982. Mysids in toxicity testing - a review. Hydrobiologia 93: 171-178.

7. ERCO. 1980. Acute toxicity results for EPA Region II generic muds; reports #1-8. Prepared for Mid-Atlantic Operators as part of Joint Bioassay Program, EPA, Region II. Energy Resources Co., Cambridge, Massachusetts.

8. Normandeau Associates. 1980. Acute toxicity results for EPA Region II generic muds; reports #1-8. Prepared for Mid-Atlantic Operators as part of Joint Bioassay Program, EPA Region II. Normandeau Associates, Inc., Bedford, New Hampshire.

9. Ayers, R. C., R. P. Meek, T. C. Sauer, and D. O. Stuebner. 1980. An environmental study to assess the effect of drilling fluids on water quality parameters during high rate, high volume discharges to the ocean, p. 351-381. In Proceedings of a symposium on research on environment fate and effects of drilling fluids and cuttings. Lake Buena Vista, Florida, January 21-24, 1980.

10. Petrazzuolo, G. 1981. Preliminary report: An environmental assessment of drilling fluids and cuttings released onto The Outer Continental Shelf. Draft submitted March 26, 1981. Prepared for the Office of Water Enforcement and Office of Water and Waste Management, U.S. Environmental Protection Agency, Washington, D.C., Vol. I, II.

11. EG&G. 1982. A study of environmental effects of exploratory drilling on the Mid-Atlantic Outer Continental Shelf - final report of the Block 684 monitoring program. Prepared for the Offshore Operators Committee under the supervision of Exxon Production Research Company, Houston. EG&G, Environmental Consultants, Boston, Massachusetts.

Table 1. Generic Mud Systems for EPA, Region II, Mid-Atlantic Operators Bioassay Program

1. Potassium/Polymer Mud

Components	lbs/bbl
KCl	5-50
Starch	2-12
Cellulose Polymer	0.25-5
XC Polymer	0.25-2
Drilled Solids	20-100
Caustic	0.5-3
Barite	0-450
Seawater or Freshwater	As needed

2. Seawater/Lignosulfonate Mud

Components	lbs/bbl
Attapulgite or Bentonite	10-50
Lignosulfonate	2-15
Lignite	1-10
Caustic	1-5
Barite	25-450
Drilled Solids	20-100
Soda Ash/Sodium Bicarbonate	0-2
Cellulose Polymer	0.25-5
Seawater	As needed

3. Lime Mud

Components	lbs/bbl
Lime	2-20
Bentonite	10-50
Lignosulfonate	2-15
Lignite	0-10
Barite	25-180
Caustic	1-5
Drilled Solids	20-100
Soda Ash/Sodium Bicarbonate	0-2
Freshwater or Seawater	As needed

4. Non-dispersed Mud

Components	lbs/bbl
Bentonite	5-15
Acrylic Polymer	0.5-2
Barite	25-180
Drilled Solids	20-70
Freshwater or Seawater	As needed

5. Spud Mud (slugged intermittently with seawater)

Components	lbs/bbl
Attapulgite or Bentonite	10-50
Lime	0.5-1
Soda Ash/Sodium Bicarbonate	0-2
Caustic	0-2
Barite	0-50
Seawater	As needed

6. Seawater/Freshwater Gel Mud

Components	lbs/bbl
Attapulgite or Bentonite	10-50
Caustic	0.5-3
Cellulose Polymer	0-2
Drilled Solids	20-100
Barite	0-50
Soda Ash/Sodium Bicarbonate	0-2
Lime	0-2
Seawater or Freshwater	As needed

7. Lightly Treated Lignosulfonate Freshwater/Seawater Mud

Components	lbs/bbl
Bentonite	10-50
Barite	0-180
Caustic	1-3
Lignosulfonate	2-6
Lignite	0-4
Cellulose Polymer	0-2
Drilled Solids	20-100
Soda Ash/Sodium Bicarbonate	0-2
Lime	0-2
Seawater to Freshwater Ratio	1:1appx

8. Lignosulfonate Freshwater Mud

Components	lbs/bbl
Bentonite	10-50
Barite	0-450
Caustic	2-5
Lignosulfonate	4-15
Lignite	2-10
Drilled Solids	20-100
Cellulose Polymer	0-2
Soda Ash/Sodium Bicarbonate	0-2
Lime	0-2
Freshwater	As needed

Table 2. Classification of Toxicity Grades.[3,4]

Toxicant Classification	LC_{50} Value (ppm)
Practically Nontoxic	>10,000
Slightly Toxic	1,000-10,000
Moderately Toxic	100-1,000
Toxic	1-100
Very Toxic	<1

Table 3. Components, Properties, Metals, and Oil Content in Field Drilling Fluids Used in Mid-Atlantic Bioassay Program

	Generic Mud Types							
	#1 KCl/Polymer	#2 SW Lignosulfonate	#3 Lime	#4 Non-dispersed	#5 SW Spud	#6 SW/FW Gel	#7 LT Lignosulfonate SW/FW	#8 Lignosulfonate FW
Components (lbs/bbl)								
Barite	18.0	176	64.0	10.8*	2	21.2	9.0	15.1
Bentonite/Drill Solids	18.0	32.1	20.0/30.0	20.0*/49.0	22.0/52.0	9.7*/14.1*	25.0/48.0	15.1/28.1
Chrome Lignosulfonate	0	1.8*(2.8)[3]	3.5	0	0	0	4.0	1.7*
Lignite	0	0.9*	1.8	0.1	0	0	5.0*	2.8
Polyanionic cellulose	1.0	0.2*	0	1.0	0	0.5	0.5	0
Caustic	2.0	0.9*	1.5	n.m.	n.m.	0.4*	n.m.	1.2*
Other	KCl (10.0) XC Polymer (12.0)[1]	Salt (10.0)	Lime (1.5)*			CMC (0.1)		Lime (<0.1)
Properties								
Mud Density (lbs/gal)	9.3	12.1	10.4	9.4	9.2	9.1	9.6	9.3
Percent Solids (wt%)	18.1	43.5	27.8	21.0	21.7	11.6	24.1	16.4
pH	11.5	n.m.	10.0	n.m.	n.m.	n.m.	10.8	9.0
Chlorides (mg/l)	38,000	n.m.	n.m.	1,200	n.m.	250	7,500	1,800
Calcium (mg/l)	n.m.[2]	650	lime	n.m.	n.m.	40	n.m.	40
O/G (ppm-whole mud)[4]	2,200	1,800	180	290	70	40	50	80
Metals (ppm-whole mud)[4]								
Arsenic	1	2	3	2	3	2	<1	3
Barium	24,800	141,000	76,200	13,300	2,800	25,600	11,500	14,000
Cadmium	<1	<1	<1	<1	<1	<1	<1	<1
Chromium	14	227	192	10	16	2	265	48
Copper	2	11	8	7	5	2	26	4
Lead	2	<1	4	2	4	<1	24	9
Mercury	<1	<1	<1	<1	<1	<1	<1	<1
Nickel	6	8	3	4	6	1	6	8
Vanadium	9	18	27	22	35	6	30	18
Zinc	20	181	58	16	21	12	82	15

1 - Other components in KCl mud; - Soda Ash (4.0), Aluminum Stearate (0.5), Sawdust (<.1), Lime (<.1), Surfactant (<.01), no paraformaldehyde.
2 - n. m. - not measured
3 - Chrome lignosulfonate concentration estimate from chromium content calculation (3% Cr in chrome lignosulfonate)
4 - Oil and grease analyses conducted by Energy Resources Co., Cambridge; Metals analyses conducted by SCR Inc., Houston
* Estimated concentration outside range designated in generic mud systems (Table 1)