# NETI-WEST BASIC INSPECTOR TRAINING COURSE STUDENT MANUAL May 2001

**U.S. Environmental Protection Agency**
**Office of Enforcement and Compliance Assurance**
**Office of Criminal Enforcement, Forensics, and Training**
**National Enforcement Training Institute**
**Lakewood, Colorado**

EPA NETI

NATIONAL ENFORCEMENT TRAINING INSTITUTE - West

# COURSE EVALUATION
**Basic Inspector Training Course**
**National Enforcement Training Institute**

ORGANIZATION: ____________________ NAME (Optional): ______________________________

Years of Experience as an Inspector: __________
______ Job Title: ______________________________________________________________

Course Date(s): __________________________
__________________________________

Please take a few minutes to assist us in evaluating this training program. Please comment and make recommendations for improvements in future courses.

**1. I FEEL THE OVERALL PROGRAM WAS:**

________ VERY WORTHWHILE _______ WORTHWHILE _______ OF SOME VALUE ________ OF LITTLE VALUE

**COMMENTS:**

**2. ORGANIZATION OF PROGRAM AND MATERIAL WAS:**

________ EXCELLENT _______ SATISFACTORY _______ INADEQUATE

**COMMENTS:**

**3. PRESENTATION AND MATERIAL:**

___ WERE APPROPRIATE _____ EXCEEDED NEEDS OF THE CLASS _____ WERE INSUFFICIENT FOR CLASS NEEDS

**COMMENTS:**

4. **HOW WELL DID THIS COURSE MEET YOUR PURPOSE:**

________ **EXCELLENT** _______ **TOO BASIC** _____ **TOO TECHNICAL** ___ **SATISFACTORY**

**COMMENTS:**

5. **WHICH PRESENTATION(S) DID YOU THINK WERE MOST BENEFICIAL AND WHY?**

6. **WHICH PRESENTATION(S), IF ANY, DO YOU THINK SHOULD BE REMOVED FROM THE COURSE AND WHY?**

7. **WHAT CHANGES, IF ANY, DO YOU THINK WOULD IMPROVE THIS TRAINING COURSE?**

8. **ADDITIONAL COMMENTS ON COURSE, SPEAKERS, FACILITY, OR OTHER ASPECTS OF THE COURSE**

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## SESSION 1

## TOPIC: INTRODUCTION AND PURPOSE OF COURSE

**Time: 15 minutes**

**PURPOSE**

- Provide course overview and logistics.
- Explain why course was developed.
- Conduct introductions and get-acquainted discussion.
- Identify inspection-related problems inspectors have encountered and relate them to course content.

**KEY POINTS**

- Inspectors play a crucial role in ensuring that the nation's environmental laws are implemented.
- The inspector's job is complex, requiring legal, technical, and communication skills.

**LIST OF VISUALS**

| | |
|---|---|
| 1-1 | Welcome (Title Slide) |
| 1-2 | Administrative |
| 1-3 | Introduction |
| 1-4 | Introduction/Purpose of the Course |
| 1-5 | Why this Course? |
| 1-6 | Basic Inspector Training Course |
| 1-7 | Basic Inspector Training Course |
| 1-8 | Basic Inspector Training Course |
| 1-9 – 1-12 | Summary of Course Schedule |
| 1-13 | Introductions |
| 1-14 | Inspector Profile |

**LIST OF HANDOUTS** None

## Welcome

The National Enforcement Training Institute

Presents

The Basic Inspector Training Course

1-1

## Administrative

- Facilities
- Schedule/breaks
- Telephones
- Messages
- Lunch

1-2

## Introduction

- Materials
  - Student manual
  - Reference manual
  - Evaluation form
  - KISS manual
  - CD-ROM/laws
- Questions

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## Introduction/Purpose of the Course

## Why This Course?

- Inspectors play a crucial role in ensuring that the nation's environmental laws are implemented
- Inspector's work is very complex, involving:
  - Legal aspects
  - Technical aspects
  - Communications
- Course to provide foundation

## Basic Inspector Training Course

- Course required for EPA by EPA order
- Strongly recommended for non-EPA
- Developed by EPA in 1988
- Revised by EPA senior inspectors and state senior inspectors in 1996
- Principals and policies applicable to all
- Targeted for new inspectors

## Basic Inspector Training Course

- Fulfills requirements for Basic Training in
  - Legal
  - Technical
  - Administrative
  - Communications
- Must also take Health and Safety

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## Basic Inspector Training Course

- Instructors, senior inspectors and experts from Headquarters and NETI
- May be some redundancy - all phases are interrelated
- Does not focus on any one program or set of regs - provides fundamentals for all programs

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## Summary of Course Schedule

- Day 1
  - Registration
  - Introduction/Purpose of the Course
  - Introduction to Environmental Compliance
  - Summary of Environmental Statutes
  - Inspector Issues
  - Inspection Planning

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## Summary of Course Schedule

- **Day 2**
  - Administrative issues
  - Sources of Information
  - Entry, Opening Conference & Site Tour, and Inspection Techniques
  - Evidence
  - Interviewing
  - On-Site Records Review

## Summary of Course Schedule

- **Day 3**
  - Sampling Issues
  - Closing Conference
  - Inspection Report
  - Enforcement Process Responsibilities
  - Mock trial
  - Agency/Regional Issues

## Summary of Course Schedule

- **Day 4**
  - Introduction to Criminal Investigations
  - Wrap-Up and Evaluation

## Introductions

- Ask questions
- Participate
- Provide instructors with benefit of your experience

## Inspector Profile

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## SESSION 2

## TOPIC: INTRODUCTION TO ENVIRONMENTAL COMPLIANCE

**Time: 90 minutes**

**PURPOSE**

- Provide the context for the work of inspectors.
- Explain the role of inspectors in each aspect of an enforcement case.
- Provide an overview of civil litigation
- Provide an overview of Federal, State, and Tribal relations.
- Provide an introduction to pollution prevention and waste minimization
- Introduce inspectors to criminal enforcement and explain how to recognize potential criminal violations.

**KEY POINTS**

- Knowledge of program compliance and enforcement strategies aids inspectors in making appropriate field decisions.
- Inspectors are involved in every aspect of an enforcement case.
- Inspectors should recognize potential criminal violations and refer them for investigation.
- Federal, State, and Tribal relations are important factors in environmental compliance.
- Pollution prevention and waste minimization programs and efforts impact environmental compliance

**LIST OF VISUALS**

**LIST OF HANDOUTS**

- Sample Enforcement Response Policy

## Introduction to Environmental Compliance

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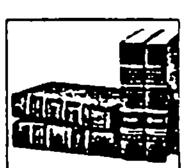
## Overview of Enforcement

- Components of a compliance program
- Civil litigation
- Role of inspector in enforcement actions
- Criminal enforcement in EPA (Will be addressed in Session 18)

3-2

## Compliance and Enforcement Program

- Laws and regulations
- Compliance and enforcement strategies
- Compliance monitoring
- Enforcement response
- Follow-up to enforcement actions



3-3

## Laws and Regulations

- Should be written clearly to indicate
  - Who is subject to them
  - What is and is not a violation

## Compliance and Enforcement Strategies

- Compliance monitoring plans
- Enforcement response policies
- Other policy and guidance documents

## Compliance Monitoring

- Source self-monitoring and reports
- Inspections

## Enforcement Response

- Based on inspector's findings and self-monitoring
- Agency policy enforcement
- Graduates with severity of violation
  - Informal administrative response
  - Formal administrative response
  - Civil judicial response
  - Criminal judicial response

## Follow-Up to Enforcement Actions

- Reports and certifications of compliance by source
- Follow-up inspections
- More severe enforcement response if facility remains in violation

## Steps in an Enforcement Action

- Violation found and documented
- Decision made on level and type of enforcement response
- Enforcement documents drafted and filed
- Settlement negotiations entered
- Hearing or trial conducted

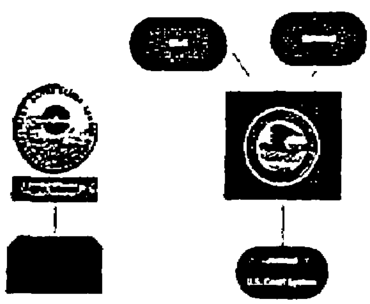
## What Can We Get From an Enforcement Action?

Depending on law, violation, and circumstances:

**Civil**
- Compliance with requirement
- Monetary penalty
- Cleanup of contamination

**Criminal**
- Monetary fine
- Prison sentence

## Types of Legal Cases

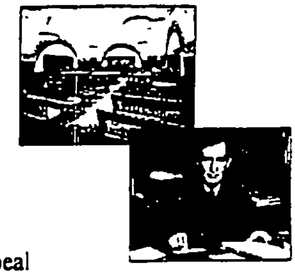


## Principal Elements of Civil Litigation

- Theory of the case
- Burden of proof
- Standards of proof

## Stages of Civil Litigation

- Conception and preparation
- Pleadings
- Discovery
- Motion practice
- Trial
- Post-trial and appeal



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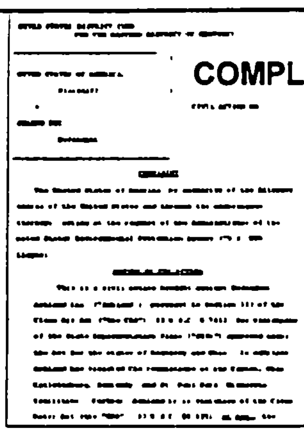

COMPLAINT

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## COMPLAINT

JURISDICTION AND VENUE

This Court has jurisdiction of the subject matter of this action pursuant to Section 113(b) of the CAA, 42 U S C § 7413(b), Section 309(b) of the CWA, 33 U S C § 1319(b), Section 3008(a) of RCRA 42 U S C § 6928(a), Section 325(b and (c) of EPCRA, 42 U S C § 11045(b) and (c), and pursuan to 28 U S C §§ 1331 1345, and 1355

Venue is proper in this District pursuant to Section 113(b) of the CAA, 42 U S C § 7413(b) Section 309(b) of the CWA, 33 U S C § 1319(b), Section 3008(a)(1) of RCRA, 4 U S C § 6928(a)(1), Section 325(b) and (c) of EPCRA, 42 U S C § 11045(b) and (c), 28 U S C § 1391(c) because Ashland does business in this District and 28 U S C § 1395(a) because this is an action for a fine or penalty and Ashland is found in this District

NOTICES

On May 19 and 20, 1998, the respective Directors of th Air Enforcement Divisions of the Environmental Protection Agency's Region IV and V offices issued Notices of Violatio to Ashland for the company's violations of certain aspects of the CAA at its Catlettsburg, Kentucky, and Canton, Ohio, facilities Pursuant to Section 113(a)(1) and (b)(1) of th CAA, 42 U S C § 7413(a)(1) and (b)(1), the Director provided a copy of the Notices of Violation to the states o Kentucky and Ohio

The 30-day period established in Section 113 of the

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## COMPLAINT

III. DEFENDANT

Ashland is a corporation incorporated under the laws o the Commonwealth of Kentucky, with its principal place of business in Russell, Kentucky

Ashland owns and operates petroleum refineries in, among other places Catlettsburg, Kentucky St Paul Park, Minnesota, and Canton, Ohio These facilities produce a variety of petroleum products The allegations set forth below apply to these three facilities only

Ashland is a "person" within the meaning of Section 302(e) of the CAA, 42 U.S.C § 7602(e), Section 502(5) of the CWA, 33 U S C § 1362(5), and Section 1004(15) of RCRA, 42 U S.C § 6903(15), and Section 329(7) of EPCRA, 42 U S C § 11049(7)

IV. CLEAN AIR ACT - STATUTORY AND REGULATORY BACKGROUND

State Implementation Plans

The Clean Air Act established a regulatory scheme designed to protect and enhance the quality of the nation's air so as to promote the public health and welfare and the productive capacity of its population 42 U S C § 7401(b)(1)

Section 110(a) of the CAA, 42 U S C § 7410(a), requires each State to adopt and submit to the Administrato for approval, a plan for the implementation maintenance, and enforcement of primary ambient air quality standards as promulgated by the Administrator

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## COMPLAINT

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## DISCOVERY MOTION

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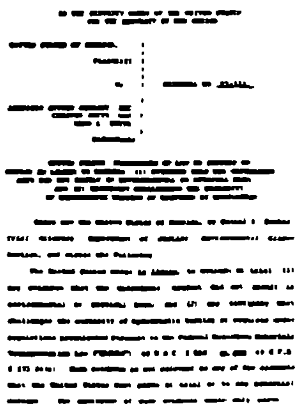

## Motion in Limine

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## Principal Differences Between Civil and Criminal Enforcement

- Warrants based on "probable cause"
- Other constitutional guarantees
- Burden of proof. "beyond a reasonable doubt"
- More severe penalties imprisonment or fine

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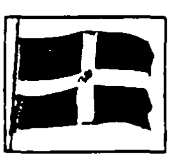
## "Red Flags" That Indicate Possible Criminal Activity



- Conflicting data
- Conflicting stories
- Unsubstantiated data
- Deliberate actions
- Claims of ignorance about requirements

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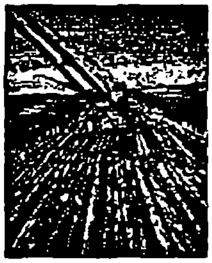
## Relationships Between Federal, State, and Tribal Authorities

- Federal Laws
- Treaties
- Tribal Rules

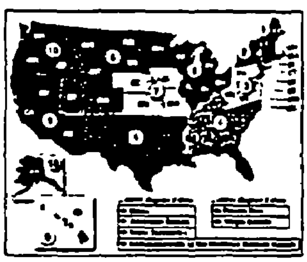


## Enforcement Agreements Between EPA and the State

(Performance Partnership Agreement)

- Establish criteria and standards for EPA oversight
- Establish criteria for direct EPA enforcement
- Specify data states will report to EPA

## Tribal Issues



- 564—Federally recognized tribes
- 227—Village groups in Alaska

## Overview of Pollution Prevention and Waste Minimization

- Pollution prevention and waste minimization
- Hazardous and Solid Waste Amendments of 1984
- Pollution Prevention Act of 1990

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**Pollution Prevention** - **works for "source reduction," and other practices that reduce or eliminate the creation of pollutants.**

**Waste Minimization** - **focuses on reducing the generation and subsequent release to the environment of the most persistent, bioaccumulative, and toxic chemicals in hazardous wastes**

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## The Environmental Management Hierarchy

- The environmental management hierarchy consists of
  - Prevention
  - Recycling
  - Treatment
  - Disposal

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## Basic Waste Minimization Requirements of HSWA

- Hazardous waste generators submit waste minimization information as part of the biennial reports
- Generators certify on manifest that waste reduction program is in effect
- As a permit requirement, all TSDFs must certify annually that waste reduction system is in place

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## Minimum Requirements for Inspectors for Waste Minimization

- Checking the manifest
- Checking the biennial report and operating record
- Other waste minimization language
- Outreach

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**HAZARDOUS WASTE**

**CIVIL ENFORCEMENT RESPONSE POLICY**

March 15, 1996

**The policies and procedures set forth in this document are intended solely for the guidance of employees of the Environmental Protection Agency and State Enforcement Agencies. They are not intended to, nor do they, constitute rulemaking by EPA. They may not be relied upon to create a right or a benefit, substantive or procedural, enforceable at law or in equity, by any person.**

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**ATTACHMENT:**

Enforcement Response Timeline

## I. INTRODUCTION

The goal of the Resource Conservation and Recovery Act (RCRA) compliance monitoring and enforcement program is to attain and maintain a high rate of compliance within the regulated community. This goal is accomplished by establishing a comprehensive monitoring and inspection program, and addressing the most serious violators with timely, visible, and effective enforcement actions. A timely and appropriate enforcement action will return the facility to compliance as expeditiously as possible, as well as deter future or potential non-compliance.

In December of 1984, the U.S. Environmental Protection Agency's (EPA's) Office of Solid Waste and Emergency Response issued the first RCRA Enforcement Response Policy (ERP). **The ERP sets forth response guidance for violations occurring pursuant to RCRA where the State or EPA intends to pursue civil action, including administrative or judicial action.**

The 1984 ERP strengthened the RCRA enforcement program by establishing guidance on timely and appropriate enforcement response, and delineating conditions for EPA enforcement actions in authorized States. The policy promoted the concept of prompt escalation of an action when compliance was not achieved. In addition, the policy directed enforcement efforts to the most serious violators. The 1984 ERP was modified in December 1987.

The 1987 Revised ERP addressed changes in the program resulting from the 1984 Hazardous and Solid Waste Amendments (HSWA) to RCRA. The HSWA Amendments necessitated modifications to the 1984 ERP in order to incorporate the broadening programmatic responsibilities, including among other things corrective action requirements, land disposal restrictions and an emphasis on hazardous waste generators, treatment and storage facilities, as well as land disposal facilities.

Since the development of the 1987 ERP, the RCRA enforcement program has evolved. The RCRA regulated universe has expanded due to the promulgation of new regulations. With the expansion of previous enforcement authorities related to federal facilities (i.e., 1992 Federal Facilities Compliance Act), the 1996 ERP will now address all violating facilities including federal facilities, in accordance with the criteria set forth in this document. In addition, EPA continues to develop a multi-media approach to facility compliance and encourages the use of national, Regional and State enforcement initiatives to address areas of non-compliance. Finally, EPA and State agencies are working together to authorize States for significant portions of the RCRA program. State primacy in implementing RCRA necessitates that the ERP accommodate the individual enforcement processes utilized by State agencies in achieving compliance with RCRA. The previous ERPs primarily reflected EPA's federal enforcement process. The 1996 ERP will address the need for

increased flexibility, as well as incorporate program developments from recent years.

The policies and procedures set forth herein are intended solely for the guidance of employees of the EPA and State enforcement agencies. They are not intended to, nor do they, constitute rulemaking by EPA. They may not be relied upon to create a right or a benefit, substantive or procedural, enforceable at law or in equity, by any person.

The revised Hazardous Waste Enforcement Response Policy will be effective on April 15, 1996.

## II. RELATIONSHIP TO OTHER AGENCY POLICY AND GUIDANCE

The ERP is one of several documents that, together, define the national RCRA Enforcement Program. The ERP provides a general framework for identifying violations and violators of concern and describing timely and appropriate enforcement responses to non-compliance. The ERP should be read in conjunction with the Office of Enforcement and Compliance Assurance (OECA) Memorandum of Agreement (MOA). The MOA establishes annual priorities for compliance monitoring and enforcement actions as identified by EPA Headquarters media programs, Regions and States. The MOA encourages use of the full range of tools to achieve compliance while emphasizing vigorous, timely, and high quality enforcement against violators of environmental statutes.

Other basic guidance utilized in the RCRA Enforcement Program include the Policy Framework for State/Federal Enforcement Agreements (revised August 1986, May 1992, February 1993, and July 1993) and the National Criteria for a Quality Hazardous Waste Management Program Under RCRA (July 1986). The Policy Framework document is an Agency-wide guidance that calls for enforcement agreements between EPA and States. It describes what the State/EPA enforcement agreements should address, including oversight criteria and measures, information needs, procedures for notification and consultation, and criteria for direct federal enforcement. The requirements of the MOA, RCRA Implementation Plan, and other RCRA guidance are made applicable to the States through the enforcement agreements.

The National Quality Criteria document establishes basic goals, objectives, and general performance expectations to assure that EPA and the States have a common understanding of what must be accomplished to effectively implement the RCRA program. The National Quality Criteria document also outlines how performance is to be measured and describes how EPA and the States should respond when criteria are not met. The enforcement program criteria modifications contained in the 1996 ERP supersede and replace all timely and appropriate criteria outlined in the Performance Expectations section of the National Quality Criteria document.[1] To the extent that a violator is deemed eligible for consideration under the Compliance Incentives for Small Businesses Policy, Small Communities Policy, the Voluntary Environmental Self-Policing and Self-Disclosure Policy, or the Audit Policy, the ERP will function as a supplement to these policies. The Audit Policy states that it "supersedes any inconsistent provisions in media-specific penalty or enforcement policies... To the extent that existing EPA enforcement policies are not inconsistent, they will continue to apply in conjunction with this policy," provided that a regulated entity may not receive additional penalty mitigation for satisfying similar conditions under other policies for the same violations. "Incentives for Self-Policing: Discovery, Disclosure, Correction and Prevention of Violations," 60 Fed. Reg. 66706 (Dec. 22, 1995).

The ERP does not address the use of an order pursuant to Section 3008(h) of RCRA, 42 U.S.C. § 6928(h), to compel corrective action; the use of an order pursuant to Section 3013 of RCRA, 42 U.S.C. § 6934, to compel monitoring, testing and analysis; or the use of an order pursuant to Section 7003 of RCRA, 42 U.S.C. § 6973, to address situations that may present an imminent and substantial endangerment to human health or the environment. In addition, the ERP does not address violations determined to be potentially criminal in nature and investigated and prosecuted pursuant to Federal or State criminal authorities. Guidance on the use of these authorities is set forth in other policy documents except, and to the extent that, the ERP applies when RCRA orders, decrees, or judgments are violated.

## III. DEFINITIONS

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[1] The 1986 National Criteria For A Quality RCRA Program permits adjustments to Regional/State Performance Expectations. The 1996 ERP modifies previous enforcement response criteria to encompass program developments, unique State authorities and individual State enforcement processes.

A. <u>Classifications of non-compliance:</u> Violators are classified based on an analysis of the facility's overall compliance with RCRA which includes prior recalcitrant behavior or a history of non-compliance. This ERP establishes two categories of violators: Significant Non-Compilers (SNC) and other Secondary Violators (SV).

1. <u>Significant Non-Compilers (SNCs)</u> are those facilities which have caused actual exposure or a substantial likelihood of exposure to hazardous waste or hazardous waste constituents; are chronic or recalcitrant violators; or deviate substantially from the terms of a permit, order, agreement or from RCRA statutory or regulatory requirements. The actual or substantial likelihood of exposure should be evaluated using facility specific environmental and exposure information whenever possible. This may include evaluating potential exposure pathways and the mobility and toxicity of the hazardous waste being managed. However, it should be noted that environmental impact alone is sufficient to cause a facility to be a SNC, particularly when the environmental media affected require special protection (e.g., wetlands or sources of underground drinking water). Facilities should be evaluated on a multi-media basis~~; however, a facility may be found to be a chronic or~~ recalcitrant violator based solely on prior RCRA violations and behavior.

2. <u>Secondary Violators</u> are violators which do not meet the criteria listed above for SNCs. Secondary Violators (SV) are typically first time violators and/or violators which pose no actual threat or a low potential threat of exposure to hazardous waste or constituents. A facility classified as a SV should not have a history of recalcitrant or non-compliant conduct. Violations associated with a SV should be of a nature to permit prompt return to compliance with all applicable rules and regulations.

B. <u>Enforceable</u> means the instrument creates an independent, affirmative obligation to comply and imposes sanctions for the prior failure to comply.

C. <u>Evaluation Date</u> is the first day of the inspection or record review during which a violation is identified, regardless of the duration of the inspection or the stage in the inspection at which the violation is identified.

D. <u>Formal Enforcement</u> is an action which mandates compliance and initiates a civil, criminal, or administrative process which results in an enforceable agreement or order.

E. Implementing Agency is the agency with responsibility for undertaking the required enforcement response.

F. Informal Enforcement are those actions other than formal enforcement that notify the facility of its non-compliance and establish a date by which that non-compliance is to be corrected.

G. Facilities will be deemed to have Returned to Compliance when they are in full physical compliance with regulatory and/or statutory requirements or when they are in full compliance with a compliance schedule established in a formal enforcement action (either an order or an agreement).

H. Sanctions include penalties as well as other tangible obligations, beyond returning to compliance, that are imposed upon the owner/operator.

## IV. APPROPRIATE ENFORCEMENT RESPONSE

The selection of an appropriate enforcement response is an integral component of the RCRA enforcement and compliance assurance program. An appropriate response will achieve a timely return to compliance and serve as a deterrent to future non-compliance by eliminating any economic advantage received by the violator. This section establishes the criteria for determining when formal and informal enforcement responses are appropriate.

### A. FORMAL ENFORCEMENT RESPONSE

The designation of Significant Non-Complier (SNC) is intended to identify non-compliant facilities for which formal enforcement is appropriate. Specifically, SNCs are those facilities which have caused actual exposure or a substantial likelihood of exposure to hazardous waste or hazardous waste constituents; are chronic or recalcitrant violators; or deviate substantially from the terms of a permit, order, agreement or from RCRA statutory or regulatory requirements.

The actual or substantial likelihood of exposure should be evaluated using facility specific environmental and exposure information whenever possible. This may include evaluating potential exposure pathways and the mobility and toxicity of the hazardous waste being managed. However, it should be noted that environmental impact alone is sufficient to categorize a facility as a SNC, particularly when the environmental media affected require special protection (e.g., wetlands or sources of underground drinking water).

Facilities should be evaluated on a multi-media basis to determine whether they are chronic violators or recalcitrant. However, facilities may also be found to be chronic or recalcitrant violators based solely on prior RCRA violations and behavior.

Due to the nature of their violations, a SNC should be addressed through a formal enforcement response. This response must mandate compliance and initiate a civil, criminal, or administrative process which results in an enforceable agreement or order. The formal enforcement response should also seek injunctive relief that ensures the non-compliant facility expeditiously returns to full physical compliance.

An enforcement response against a SNC by the implementing agency will be considered appropriate when economic sanctions in the form of penalties, or alternative punitive mechanisms, are incorporated in the formal enforcement response. Penalties incorporated in the formal enforcement response, or alternative punitive mechanisms that recover the economic benefit of non-compliance plus some appreciable amount reflecting the gravity of the violation will be considered appropriate. The portion of the penalty which does not account for the economic benefit of non-compliance may be addressed through the use of Supplemental Environmental Projects (SEPs) or Pollution Prevention Projects as deemed appropriate by the implementing agency.[3] The Agency recognizes, however, that recoupment of the full amount of economic benefit of non-compliance plus some appreciable portion of gravity may not be possible in every case. A lesser penalty amount may be appropriate where, for example, the violator demonstrates an inability to pay the full penalty. In addition, there may be circumstances where the nature of the violation(s) and the manner of correction advance important policy objectives such that substantial mitigation is warranted (e.g., where the violation was discovered by the violator during an audit or self-evaluation, and thereafter promptly and voluntarily disclosed to the government and corrected, or where the violation by a small business was disclosed and corrected pursuant to a government-approved compliance assistance program).

In addition to the injunctive relief discussed above, the implementing agency is encouraged to impose other measures

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[3] Federal enforcement actions that include a SEP or Pollution Prevention project should comply with the criteria set forth in the 1995 Interim Revised Supplemental Environmental Projects Policy.

against the non-compliant facility. Examples of non-penalty measures include, but are not limited to, SEPs, permit decisions, suspension and debarment proceedings, receivership or special masters.

### B. INFORMAL ENFORCEMENT RESPONSE

If a facility is found to be in violation but is not designated a SNC it is designated a SV. An informal enforcement response is the minimally appropriate enforcement response for all SVs. An informal enforcement response consists of a recitation of the violations and a schedule for returning the facility to full compliance with all substantive and procedural requirements of applicable regulations, permits and statutes.[3] Facilities which fail to return to compliance following an informal enforcement response should be re-classified as a SNC in accordance with Section V. A. set forth below. The appropriate enforcement response for a re-classified facility is the immediate escalation to formal enforcement.

## V. RESPONSE TIME GUIDELINES

This section establishes response time guidelines for formal and informal enforcement actions. The guidelines are designed to expeditiously return non-compliant facilities to compliance with all applicable requirements of the Federal RCRA program or the authorized State equivalent. Response times are divided into two categories, formal enforcement actions and those for informal enforcement actions. A timeline depicting these guidelines is attached. The timeline establishes response times for three types of formal enforcement. The timeline also establishes a 90 day deadline for the implementing agency to determine whether the appropriate enforcement response is a formal or informal enforcement action. Finally, the timeline establishes timeframes for the escalation from an informal response to a formal enforcement response due to the violator's failure to return to compliance.

### A. EVALUATION DATE

The evaluation date will be defined as the first day of any inspection or record review during which a violation is identified, regardless of the duration of the inspection or the

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[3] As noted in section II, above, "Relationship to other Agency Policy and Guidance," compliance assistance efforts, such as those set forth in the Compliance Incentives for Small Business policy, may be applied in conjunction with this policy.

stage in the inspection at which the violation is identified. The first day of the inspection is the evaluation date, regardless of the duration of the inspection or the stage in the inspection which the violation is discovered. For violations detected through some method other than record reviews or inspection, the evaluation date will be the date upon which the information (e.g., self-reporting violators) becomes available to the implementing agency. In the case of a State referral to EPA pursuant to Section VI. below, the evaluation date will be considered the date of the referral to EPA. In the case of SV facilities which are reclassified for failure to return to full compliance (See Section IV. B. above), the evaluation date will be considered the first day of discovery of non-compliance with the compliance schedule established through the informal enforcement response.

B. FORMAL ENFORCEMENT RESPONSE TIME

The attached timeline depicts the target response times for enforcement pursuant to RCRA. The timeline establishes target response times for three types of formal enforcement: (1) final or consent orders; (2) unilateral orders; and (3) referrals to the Department of Justice or the Attorney General's Office. The timeline delineates separate response times for formal enforcement and the escalation to formal enforcement from informal enforcement.

(1) Final or consent orders are those documents for which no appeal remains before the trier of fact. These orders represent the agreement of the parties involved or the decision of a trier of fact.

(2) Unilateral or initial orders are issued by the implementing agency and assert the agency's position that violations have occurred. However, the respondent/defendant is afforded the opportunity to appeal the agency's determination of violations to a trier of fact.

(3) For purposes of the ERP, a referral to the Department of Justice or the State Attorney General's Office occurs when the matter is officially transmitted to those offices for action. A federal referral is considered to be initiated upon the signature of the referral package by the Regional Administrator or his/her designee, or the Assistant Administrator for OECA, as appropriate. With regard to the State's referral to the Attorney General's Office, each State agency should establish a formal process for requesting that the Attorney General's Office initiate

enforcement proceedings on behalf of the State.[4] Completion of that process would then constitute referral to the Attorney General's Office as set forth in the timeline.

C. EXCEEDANCE OF FORMAL ENFORCEMENT RESPONSE TIME

Response times articulated in the ERP should be adhered to by the Regions and States to the greatest extent possible. However, there are recognized circumstances (see discussion below) which may dictate an exceedance of the standard response times. In this revision to the ERP, a ceiling of 20% per year is being established for consideration of cases involving unique factors which may preclude the implementing agency from meeting the standard response times. The 20% exceedance figure should be calculated based on the total number of civil cases existing in the Region or State at any given time.

**In cases where response times will be exceeded due to case specific circumstances, the implementing agency must prepare a brief justification for the delay and develop an alternative schedule for case resolution.** In the event that the Region does not find adequate basis within the ERP guidelines for the State's delay in enforcement, EPA reserves the right to initiate federal action. EPA will conduct periodic evaluations of Regional and State enforcement response times for the purpose of determining appropriate ceiling levels. Authorized State programs will have response time reviews performed during evaluations conducted by the Region pursuant to 40 CFR Section 35.150.

The Regions and States should strive to comply with the standard response times contained in the ERP. However, when the following considerations exist, up to 20% of the Regional/State enforcement cases may exceed the standard response times:

- Cases involving violations of two or more media; (e.g., environmental protection statutes)
- Cases involving more than one facility;
- Potential criminal conduct which is under investigation;
- National enforcement initiatives;

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[4] All references to the State Attorney General's Office in this document should be interpreted as including any State enforcement body that possesses the authority to initiate actions in State Court.

- Cases involving nationally significant issues;[5]
- Novel legal issues or defenses;
- Site abandonment;
- Additional sampling or information requests are required to confirm the violation(s); and
- Need for outside technical experts.

The Agency recognizes that circumstances may arise where the enforcement response times specified may be insufficient to prepare and initiate the appropriate enforcement response as set forth in this policy. It is also recognized that instances may occur where immediate action is appropriate. The Agency expects that the Region or State will take priority enforcement action in the following situations:

- Where a release or other violation poses an immediate threat to human health or the environment.
- Where activities of the owner/operator must be stopped or redirected, such as cases in which the Agency or the State seek to immediately halt improper construction or installation of a regulated unit.
- Where the threat of a dissipation of assets would undermine closure, post-closure, or corrective action activities.
- Where there is an imminent statute of limitations deadline or bankruptcy deadline.

## D. INFORMAL ENFORCEMENT RESPONSE TIME

Once a determination is made to utilize an informal enforcement mechanism, a violator is given notice of its non-compliance and the implementing agency will establish a date by which all violations must be corrected. The objectives of an informal enforcement response are to compel the violator to cease

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[5] Requests for exceedance of the formal enforcement response times due to existence of nationally significant issues are generally reserved for EPA enforcement responses.

its non-compliant activities and ensure that full physical compliance is achieved in the shortest possible time frame.

At the time a violator is formally notified of the violation determination it is given a compliance date which establishes a deadline for the violatior to correct all known violations. A correction period during which a violator should correct all known violations should not exceed 90 days. For a violator to be considered a candidate for informal enforcement, violations must be of a nature that will permit such a prompt return to compliance with all applicable rules and regulations. Violators addressed through an informal enforcement response should not have a history of recalcitrant or non-compliant conduct.

Violators that will require an extended compliance schedule in order to achieve full physical compliance should be addressed through a formal enforcement response. The compliance date should reflect the minimum period of time necessary for the violator to return to full physical compliance. A violator that has corrected its violations on or before the assigned compliance date is officially deemed to have returned to compliance.

If a violator is unable to meet the assigned compliance deadline it must immediately notify the implementing agency and provide that agency with documentation supporting the inability to correct violations by the prescribed compliance date. A decision to extend the compliance date should be made only when supported by sufficient documentation. Failure to achieve full physical compliance by the compliance date or a failure to notify the implementing agency of the inability to correct violations should result in an escalation to formal enforcement. The first day in exceedance of compliance date is to be considered the evaluation date for the purpose of escalating the action to a formal enforcement response. For liability and penalty assessment purposes, however, nothing in this ERP should preclude the assessment of penalties for any violations which occur during the correction period.

## VI. EPA ACTION IN AUTHORIZED STATES

States with authorized RCRA programs have the primary responsibility for ensuring compliance with the RCRA program requirements. However, EPA retains the authority to take independent enforcement action in authorized States in accordance with Section 3008(a)(2) of RCRA. Pursuant to this Section, EPA may take direct action after notice to the authorized State. EPA authority to initiate an independent enforcement action is not limited to the examples set forth, the Agency may take direct action after consideration of all pertinent factors and consultation with the State.

Notwithstanding Section 3008(a)(2) of RCRA, EPA will generally take civil enforcement actions in authorized States only under the following circumstances:

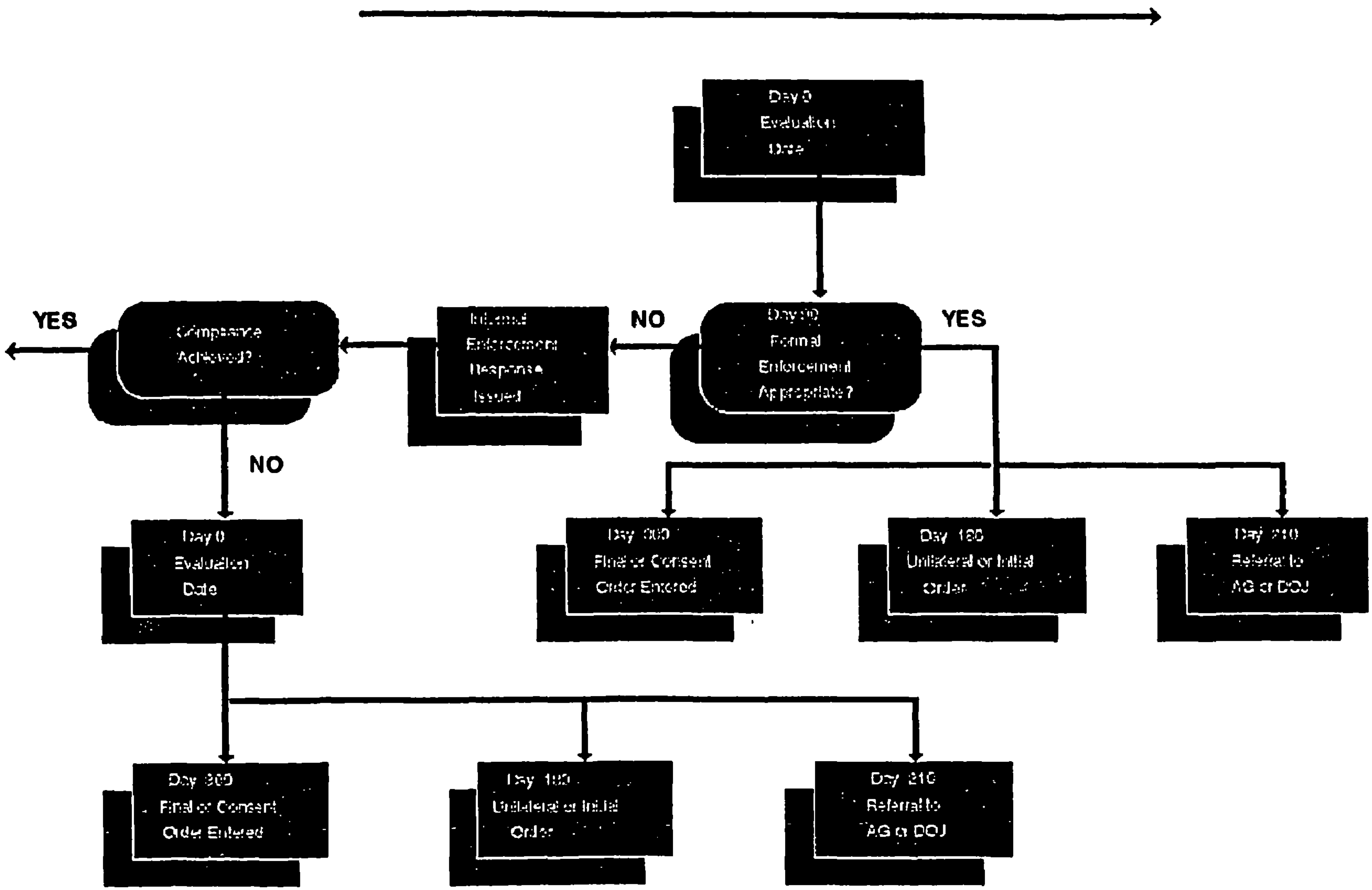
- o The State requests that EPA pursue a federal action and provides justification based on unique, case specific information;
- o The State is not authorized to take action or State authority is limited;
- o The State fails to take timely and/or appropriate action;
- o Cases involving issues that could establish a legal precedent or in which federal involvement is needed to ensure national consistency;
- o Cases involving multi-state, multi-regional "national violators;"
- o Cases involving interstate pollution problems associated with watersheds, air basins or other geographic units that cross state lines; or
- o Cases brought to prevent non complying companies from obtaining an economic advantages over their competitors, thereby maintaining a "level playing field" for the regulated community.

The previous Sections described the criteria for timely and appropriate action in response to violators in two (2) distinct categories (SNC and SV). The response times set forth in Section V. B. establish clear guidelines for a Region or State to follow during a formal enforcement process. If a State fails to take formal enforcement action within the standard response time, the State must provide the Regional office with adequate justification for consideration of an alternative schedule.

The Memorandum of Agreement (MOA), Memorandum of Understanding (MOU), or other agreement between EPA and each State should detail a process for notifying the State of EPA intent to initiate an independent enforcement action. The Regional office may need to conduct its own case development inspection, and prepare additional documentation before proceeding to initiate an action.

A State may find it necessary and advantageous to refer specific cases to the Region for federal enforcement. If a State decides to refer a case to EPA for federal enforcement, this must be completed within 90 days of the original Evaluation Date. For the purposes of establishing a new Evaluation Date, the date of the referral to EPA is considered the Evaluation Date. The State should provide all case development information to the Region as part of the referral package. This should facilitate a reduction in the time needed for Regional case development.

## Timeline For Enforcement Pursuant to The Resource Conservation and Recovery Act




Note: All days are calendar days measured from day 0.

## SESSION 3

## TOPIC: SUMMARY OF ENVIRONMENTAL STATUTES

**Time: 1 hour 45 minutes**

**PURPOSE**

- Provide an overview of the major statutes under which EPA has authority.
- Discuss inspection activities under each statute.

**KEY POINTS**

- While each statute is different, they have many features in common.
- Inspectors should be able to recognize major violations of other EPA programs and refer them, as appropriate.

**LIST OF VISUALS**

3-1 Summary of Environmental Statutes (Title Slide)
3-2 Statutory and Regulatory Process
3-3 Major Statutes Implemented by EPA
3-4 Common Statutory Features
3-5 Typical Enforcement Options
3-6 Selected Provisions of 40 CFR
3-7 Selected Provisions of 40 CFR (continued)
3-8 Authorities Granted Under Federal Environmental Laws and Regulations for Administrative Investigations
3-9 ESRC CD-ROM

**LIST OF HANDOUTS**

3-1 Summary of Authorities

# Summary of Environmental Statutes

3-1

## Statutory and Regulatory Process

- People
- Congress
- "Agency"
- Regions/states/tribes/locals

3-2

## Major Statutes Implemented by EPA

| | | Original Enactment |
|---|---|---|
| Pesticides | FIFRA | 1946 |
| Air Pollution | CAA | 1960s |
| Water Pollution | CWA | 1960s |
| Toxic Chemicals | TSCA | 1970s |
| Solid and Hazardous Waste | RCRA | 1970s |
| Abandoned Toxic Dumps | CERCLA | 1980s |
| Response Coordination | EPCRA | 1970s |

3-3

## Common Statutory Features

- Set national standards
- Administered through regulations, policies, delegation and permits
- Agency authority for inspection, monitoring, testing, information, and emergency response
- Determining violations and seeking correction, remediation, relief
- Fines, penalties, and jail

## Typical Enforcement Options

- Notice of violation
- Civil administrative and judicial complaints
- Emergency action
- Injunctive relief
- Criminal action
- Debarment

## Selected Provisions of 40 CFR

| Part | Topic |
|---|---|
| 1 | General information about and organization of EPA |
| 2 | Freedom of Information Act requests; confidential business information, testimony by employees and production of documents in civil legal proceedings in which the United States is not a party |
| 3 | Employee ethical standards |
| 22 | Rules of practice of administrative assessment of civil penalties and the revocation or suspension of permits |
| 30 | General regulations for assistance programs |
| 32 | Debarment and suspension under EPA assistance programs |

(continued)

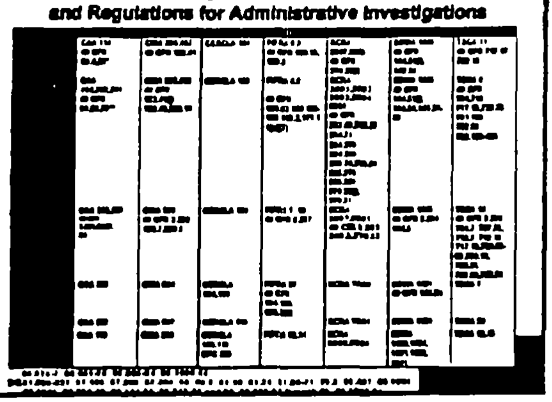
6

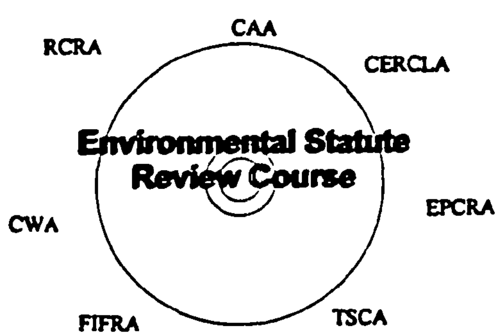
## Selected Provisions of 40 CFR
*(continued)*

| | |
|---|---|
| 100-140 | NPDES program |
| 141-147 | Drinking-water program |
| 152-180 | Pesticides program |
| 220-233 | Ocean dumping |
| 240-272 | Solid and hazardous |
| 280 | Underground storage tanks |
| 300-355 | Superfund |
| 400-471 | CWA effluent guidelines and standards |
| 702-799 | Toxic substances |

3-7 5

## Authorities Granted Under Federal Environmental Laws and Regulations for Administrative Investigations






3-9

# Authorities Granted Under Fe[illegible] nvironmental Laws and Regulations for Administrat[illegible] and Civil Investigations

| | Air | Water | Superfund | Pesticides | Solid Waste | Drinking Water | Toxics |
|---|---|---|---|---|---|---|---|
| Inspection Authority | CAA 114<br>40 CFR 80.4, 86* | CWA 308, 402<br>40 CFR 122.41 | CERCLA 104 | FIFRA 8, 9<br>40 CFR 160.15, 169.3 | RCRA 3007, 9005<br>40 CFR 270.30(i) | SDWA 1445<br>40 CFR 144.51(i), 142.34 | TSCA 11<br>40 CFR 717.17, 792.15 |
| Recordkeeping Authority | CAA 114, 208, 311<br>40 CFR 51, 60, 79** | CWA 308, 402<br>40 CFR 122.41(j), 122.48, 233.11 | CERCLA 103 | FIFRA 4, 8<br>40 CFR 160.63, 160.185-195, 169.2, 171.11(c)(7) | RCRA 3001, 3002 3003, 3004, 9003<br>40 CFR 262.40, 263.22, 264 74, 264.279, 264.309, 265.74, 265.94, 265.279, 265.309, 270.30(j), 270.31 | SDWA 1445<br>40 CFR 144.51(j), 144.54, 141.31-33 | TSCA 8<br>40 CFR 704, 710, 717.15, 720.78, 761.180, 762.60, 792.185-195 |
| Confidential Information (40 CFR 2.201-2.215) | CAA 208, 307<br>40 CFR 2.301, 53, 57, 80 | CWA 308<br>40 CFR 2.302, 122.7, 233.3 | CERCLA 104 | FIFRA 7, 10<br>40 CFR 2.307 | RCRA 3007, 9005<br>40 CFR 2.305, 260.2, 270.12 | SDWA 1445<br>40 CFR 2.304, 144.5 | TSCA 14<br>40 CFR 2.306, 704.7, 707.75, 710 7, 712.15, 717.19, 720.85-95, 750 16, 750 36, 762.60, 763.74 |
| Emergency Authority | CAA 303 | CWA 504 | CERCLA 104, 106 | FIFRA 27<br>40 CFR 164.123, 166.3(d) | RCRA 7003 | SDWA 1431<br>40 CFR 144.34 | TSCA 7 |
| Employee Protection | CAA 322 | CWA 507 | CERCLA 110 | | RCRA 7001 | SDWA 1450 | TSCA 23 |
| Penalties | CAA 113 | CWA 309 | CERCLA 103, 112<br>EPC 325 | FIFRA 12, 14 | RCRA 3008, 9006 | SDWA 1423, 1424, 1431, 1432, 1441 | TSCA 15, 16 |

* 86.078-7, 86.441-78, 86.606-84, 86.1006.84

** 51.320-327, 57.105, 57.305, 57.404, 58, 60.7, 61.10, 61.24, 61.69-71, 79.5, 85.407, 85.1086, 85.1906, 86.084-40, 86.144-78, 86.542-78 and 90, 86.609-84 through 98, 86.1009-84

## SESSION 4

## TOPIC: INSPECTOR ISSUES

**Time: 1 hour 15 minutes**

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**PURPOSE**

- Provide an overview of the roles and functions performed by inspectors that will be covered in more depth during the course.
- Provide information about issues related to confidential business information and ethics that are of concern to inspectors.
- Present techniques for handling press and other public inquiries related to an inspection.
- Provide awareness of the inspector's liability for false statements.

**KEY POINTS**

- Inspectors are involved in virtually every aspect of the compliance and enforcement program.
- The inspection is a team effort.
- Inspectors have access to confidential data and must handle such data as its confidentiality requires.
- When the inspector is faced with a question of ethics, the rule of thumb is, when in doubt, don't!
- When dealing with the press, emphasize the positive.

**LIST OF VISUALS**

| | |
|---|---|
| 4-1 | Inspector Issues (Title Slide) |
| 4-2 | Inspector Issues |
| 4-3 to 4-~~6~~ 13 | Role of the Inspector |
| 4-6 to 4-~~14~~ 22 | Responsibilities of the Team Leader and Team Members |
| 4-~~14~~ 23 32 | Follow-Up |
| 4-~~15~~ 24 to 4-~~20~~ 32 | Confidential Business Information > 4-33 to 4-34 Ethics |
| 4-~~20~~ to 4-~~25~~ 35 - 37 | Dealing with the Press and the Public |

**LIST OF HANDOUTS**

4-1 Inspector's Liability

## Inspector Issues

4-1

## Inspector Issues

Role of the inspector

Responsibilities of the team leader and team members

Confidential business information

Ethical considerations

Techniques for handling the press and the public

4-2

## Role of the Inspector

All stages and Aspects of Compliance and Enforcement Program

4-3

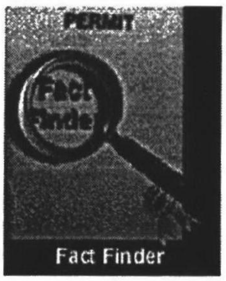
## Role of the Inspector

- **Official Representative**


4-4

## Role of the Inspector

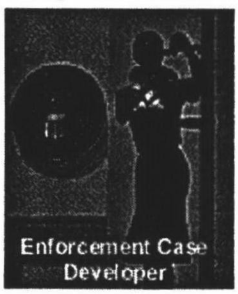
- Official representative
- **Fact-finder**




4-5

## Role of the Inspector

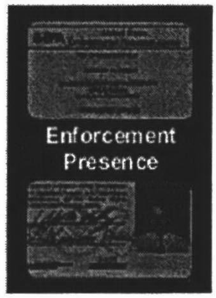
- Official representative
- Fact-finder
- **Enforcement case developer**




4-6

## Role of the Inspector

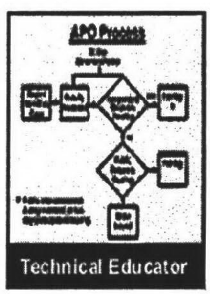
- Official representative
- Fact-finder
- Enforcement case developer
- **Provider of enforcement presence**




4-7

## Role of the Inspector

- Official representative
- Fact-finder
- Enforcement case developer
- Provider of enforcement presence
- **Technical educator**




4-8

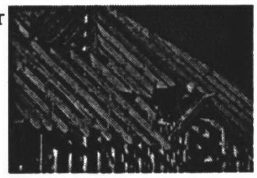
## Role of the Inspector

- Official representative
- Fact-finder
- Enforcement case developer
- Provider of enforcement presence
- Technical educator
- **Technical authority**


4-9

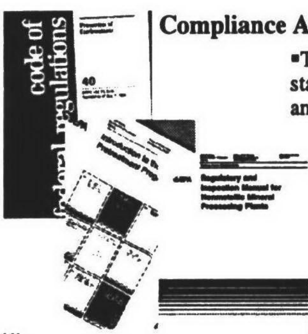
## Role of the Inspector

- Official representative
- Fact-finder
- Enforcement case developer
- Provider of enforcement presence
- Technical educator
- Technical authority
- **Compliance Assistance**



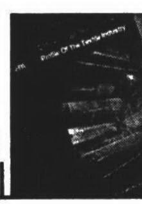
4-10

## Role of the Inspector




### Compliance Assistance

- Tier I - Sharing standardized information and references



4-11

## Role of the Inspector

### Compliance Assistance

- Tier II - More technically complex and site-specific
  - Detailed technical information/discussion of individual facility

4-12

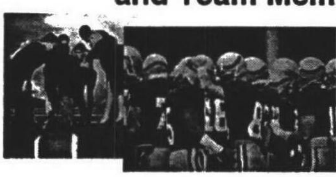
## Role of the Inspector

### Compliance Assistance

- Tier III - Most technically complex and site-specific
  - Assisting owner/operator in operating facility or in making design/operation changes
  - Consulting services to facility

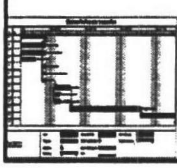
4-13

## Responsibilities of the Team Leader and Team Members



Team

Leader



Responsibilities

## Responsibilities of the Team Leader and Team Members

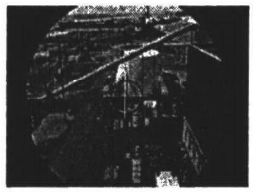
- Planning
- Specific tasks
- Broad perspective
- Work as a team
- Complete report

4-15

## Team Leader

**Working with management, technical experts, legal specialists:**

**IDENTIFY THE SCOPE!**



4-16

## Team Leader

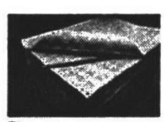
- **Identify Resources**

People

Equipment



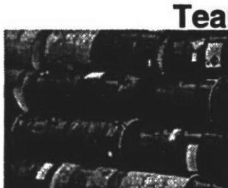
Money



Files and Data Bases

4-17

## Team Leader



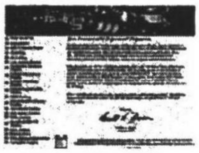
Problem

Objective

4-18

## Team Leader

Guidance



Training

Feedback

4-19

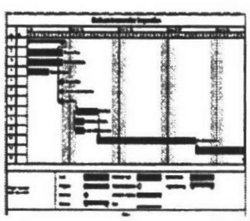
## Team Leader and Team Members

▪COMMUNICATION

4-20

## Team Leader

When developing schedule for inspection activities, keep in mind such questions as:



▪What tasks need to be performed

▪How long will each task take

▪When do the tasks need to be completed

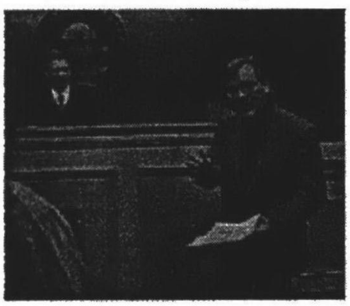
4-21

## Team Report

- Team Leader assigns writing responsibilities
  - **sections**
  - **schedules**
- Team Members prepare appropriate sections on schedule
- Team Leader prepares executive summary

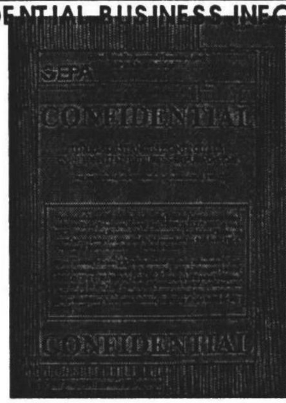
4-22

## Follow-up



4-23

## CONFIDENTIAL BUSINESS INFORMATION




4-24

## Confidential Business Information

How many have received TSCA CBI training?

4-25

## Confidential Business Information

WHAT IS IT?

- TSCA CBI vs confidential information claimed pursuant to 40CFR Part 2
  - (also FIFRA and proposed RCRA CBI)
- Proprietary data
  - Chemical formulations
  - Special process operations-special techniques
  - Financial information-lists of customers
  - Anything that might give another company a competitive advantage

4-26

## Confidential Business Information

WHAT IS IT - continued

- Anything the company claims as CBI
- Receipts must be provided for CBI material-TSCA receipts are preprinted
- Agency can declassify after requiring the company to justify based on four separate areas (see 40 CFR Part 2)

4-27

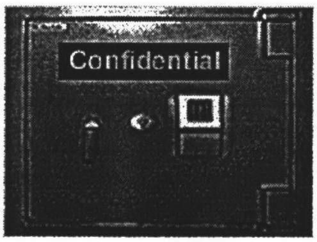
## Confidential Business Information

- TSCA CBI has very stringent handling requirements
- All CBI must be properly secured


4-28

## Confidential Business Information

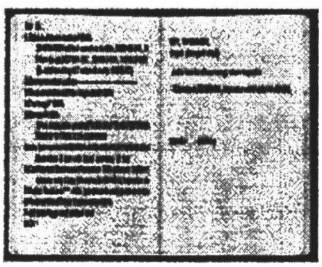
- Only authorized personnel can see the data




4-29

## Confidential Business Information

- Access log is maintained



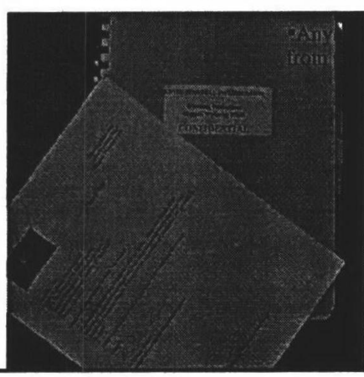
4-30

## Confidential Business Information

- Limits are imposed on copies made

4-31

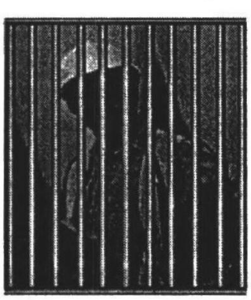
## Confidential Business Information



- Any ... ort generated from ... also is CBI

4-32

## ETHICS
## When in Doubt, Don't!



4-33

## ETHICS
## When in Doubt, Don't!

- Integrity and impartiality
- Conflict of interest
- Standards of conduct

- Consult with your ethics official first

4-34

## Dealing with the Press and the Public

# Emphasize The Positive! !

4-35

## The Reporter's Viewpoint

- Goal: A usable story
- Differences among:
  - Newspaper
  - Television
  - Radio

4-36

## Talking with the Press

- Know what you want to say
- Use the interview as an opportunity to say it
- Be professional
- Don't say too much
- Don't speculate

4-37

# INSPECTOR'S LIABILITY

Inspectors must make sure they are honest, forthright, and live up to the Boy Scout law in all their dealings with the regulated community. One senior inspector conducted an inspection at a facility and found major repeat violations. The inspector wrote the report and was responsible for either negotiating a settlement or preparing for trail. During discovery, the inspector provided information about education, work experience, and other facts, as requested by the defense attorney. The inspector's credentials were extensive and impressive; however, because of comments made during a deposition, the inspector's educational background was checked. The inspector had stated that he had a doctoral degree when, in fact, he had only taken courses toward such a degree. As a result of that dishonesty, the inspector was removed from the case, was placed under house arrest (was allowed only to go to work and return home), and ultimately was fired.

In another case, two inspectors were picked up at the airport by a courtesy shuttle. One of the inspectors claimed taxi fare, while the other did not. When questioned about the claimed taxi fare, the inspector admitted falsely claiming the fare and resigned.

During a major cleanup activity, an on-scene coordinator aided a contractor in obtaining a contract, accepted special favors, and falsified information on travel vouchers. Acting on a tip, the OIG and OCI initiated an investigation, documented the illegal activity, and had the individual arraigned in federal court. The case is still pending.

During a criminal investigation at a federal facility, evidence showed that three senior civilian employees knowingly and willingly had disposed of hazardous waste. Since it was a government facility and the government brought charges against the employees, they had to hire their own attorneys for the trial. The employees were found guilty, given suspended sentences, and fined. After being found guilty of a felony, they lost their government pensions, in addition to expending most or all of their life savings on legal fees.

Since inspectors are gathering evidence to collect fines or put people in jail, all inspectors must ensure that they are honest and above reproach; otherwise, they are subject to fines, dismissal, or imprisonment.

## SESSION 5

## TOPIC: INSPECTION PLANNING

**Time: 90 minutes**

**PURPOSE**

- Stress the importance of planning and advance preparation.
- Present information about key planning activities

**KEY POINTS**

- Plan in advance what to look for, how to look, and what documentation to collect: saves time and money and ensures a thorough inspection.
- Be Familiar with inspection options including Multimedia Inspection, Process Based Inspections, and Environmental Management System Evaluations

**LIST OF VISUALS**

| | |
|---|---|
| 5-1 | Inspection Planning (Title Slide) |
| 5-2 | Planning the Inspection |
| 5-3 | Goal of Inspection Planning |
| 5-4 | Know What To Look For |
| 5-5 | Know How To Find It |
| 5-6 | Know How To Collect, Document, and Preserve Evidence |
| 5-7 | Be Safe and Efficient in the Field |
| 5-8 | Reviewing EPA Records |
| 5-9 to 5-24 | Project Planning |
| 5-25 to 5-27 | Multimedia Inspections |
| 5-28 to 5-39 | Process Based Inspections |
| 5-40 to 5-57 | Environmental Management Systems |

**LIST OF HANDOUTS**

5-1 Generic Inspection Check List
5-2 Sample Project Plan

## Inspection Planning

3-1

## Planning

from Alice in Wonderland

- "Would you tell me, please, which way I ought to go from here?
- "That depends a good deal on where you want to go to, "said the cat
- "I don't much care where, "said Alice
- "Then it doesn't matter which way you go," said the cat.

3-2

## Planning the Inspection

- When plans went awry
- Importance of planning
- Key planning activities
- Defining scope and objectives
- Check lists
- Reviewing EPA records

3-3

## Goal of Inspection Planning

Identify all activities necessary to gather information to assess whether a facility is in compliance and to use as evidence in possible enforcement action

HOW TO ACCOMPLISH GOAL?

BE PREPARED!

## Know What To Look For

- Identify type of inspection
- Compliance evaluation
  - Routine
  - For cause
  - Oversight
- Define objective of inspection
- Decide focus of inspection

## Know How To Find It

- Review records and permits
- Know compliance history
- Talk with attorneys and other inspectors
- Contact state and local officials

## Know How To Collect, Document, and Preserve Evidence

- Know what nontechnical evidence is needed
- Identify the kind and quantity of physical samples needed
- Identify necessary equipment and check condition
- Prepare QA/QC plan for documentation, chain of custody, transportation

3-7

## Be Safe and Efficient in the Field

- Develop safety plan
- Use personal protective equipment
- Define tasks of all members of inspection team
- Arrange logistics in advance
  - Travel
  - Pay
  - Transportation
  - Lodging
  - Special equipment

3-8

## Reviewing EPA Records

- Become familiar with the facility
- Discover inadequacies in the information
- Minimize inconvenience to facility personnel
- Clarify technical and legal issues before entry
- Develop inspection plan

3-9

## Development of the Project Plan

- Foundation and purpose of the project plan
- Steps in developing the project plan
- Team-building considerations
- Review of basic elements

11

## Elements of a Project Plan

- Objectives
- Background
- Tasks
- Policies and procedures
- Safety
- Resources
- Schedules
- Modifications of the plan

12

## Identify the Overall Project Objectives:

- Focus on why a facility is targeted
- Reasons for selecting a facility
- Determine the desired outcome
- Create a statement of objectives

## Gather Background Information:

- Background information is crucial for developing a project plan

## Identification of Tasks

- What activities must be accomplished to meet the inspection objectives

## Policies and Procedures

- Organization Standard Operating Procedures (SOP's)
- Special procedures applicable to this inspection
- Policies and Procedures of other organizations
  - e g Federal EPA State environmental agency, Tribal considerations, US Attorney, DOJ, State Attorney General, other organizations

5-16

## Safety

- Safety Plan
- Requirements of Specific Organization
- Safety Equipment Required
- Special Safety Considerations

5-17

## Resources

- Team Members for onsite inspection
- Laboratory Support
- Administrative Support
- Money
- Time

5-18

## Building the Team

- Expertise needed
- Size considerations
- Development of the team
- Management support

5-19

## Necessary Skills and Qualifications

- Knowledge of policies and procedures
- Familiarity with a range of media
- Knowledge of technical issues
- Investigatory skills
- Recent experience
- Communication skills
- Experience

5-20

## Development of an Inspection Schedule

- Importance
  - To the client
  - To the team
  - To the facility
- Principles

5-21

## Milestones in Schedule Development

- Pre-inspection planning meetings
- Inspection dates
- "In" briefings
- "Out" briefings
- Due dates of reports

5-22

## Final Steps

- Reevaluate the team
- Finalize the written project plan
- Conduct a preinspection team meeting

5-23

## Notification

- Procedures (announced or unannounced)
- Necessity of a letter of notification
- Information requested in a notification letter
- Notification of state regulatory officials
- Notification of the laboratory
- Time factors

5-24

EVOLUTION OF INVESTIGATION COMPLEXITY

MULTIMEDIA COMPLIANCE INSPECTION

SINGLE-MEDIA COMPLIANCE INSPECTION

## Overview: Multimedia Inspections and Enforcement

- Define multimedia
- Define holistic approach
- Advantages and disadvantages of teams
- Advantages and disadvantages of multimedia inspections
- Four categories of inspections
- Targeting and resources
- Team skill requirements

## Industry Comments

- Reduce the number of visits by inspectors
- Easier to get questions answered and address problem issues
- Inspectors develop better understanding of facility operations or problems
- More thorough inspections

5-28

# PROCESS-BASED INSPECTIONS

"Process-Based Investigations Guide"
March 1997
EPA-330/9-97-001
NEIC Library: (303) 236-5111 x287

5-29

## EVOLUTION OF INVESTIGATION COMPLEXITY


5-30

## Process-Based Inspection

Initially focuses on/subsequently based on
- comprehensive understanding of facility processes

Includes
- tracking raw materials into and through plant
- identifying by-product, co-product and products,
- identifying wastes generated
- determining how wastes are ultimately managed

5 11

## Process-Based Inspection

Purpose
- obtain indepth knowledge of facility operations
- use knowledge to make more informed investigative evaluations and determinations

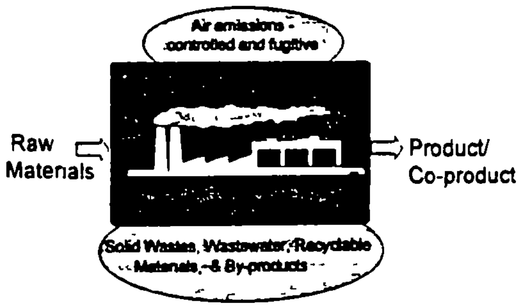
5-1

## Why Conduct Process-Based Inspection??

Often only method to
- Determine complete universe of regulated facility wastes/activities
- Evaluate accuracy and completeness of facility self-reporting data/permit application information
- View large or complex facility "holistically"

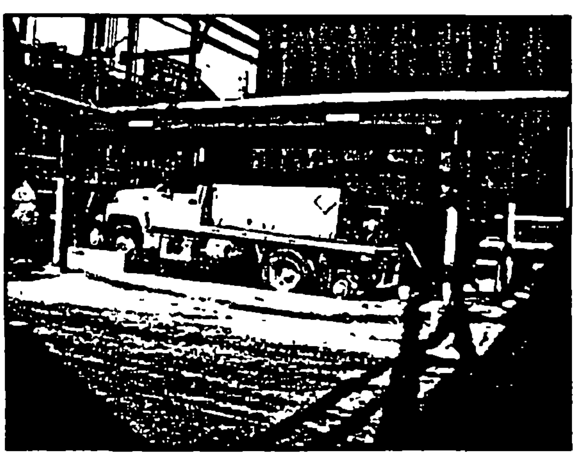
5-11

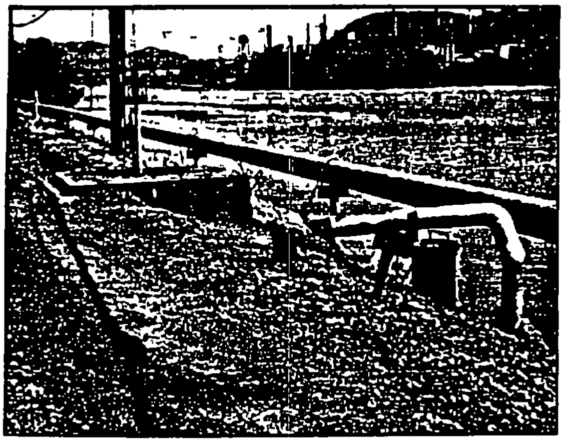
## PROCESS-BASED INSPECTION

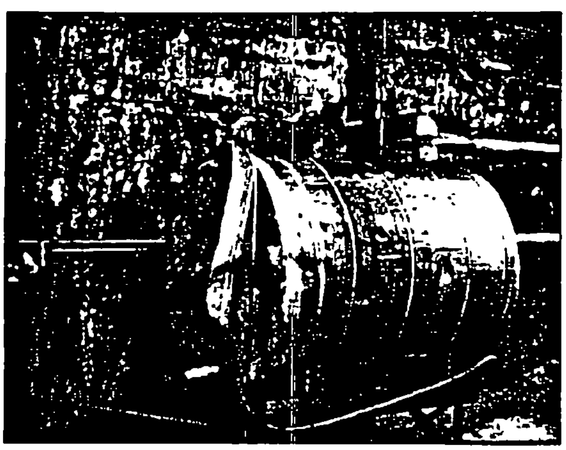


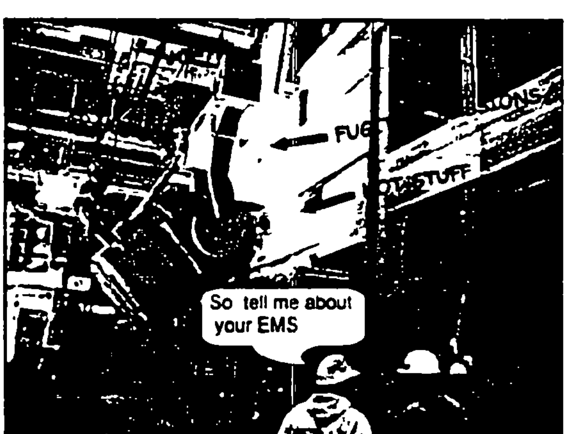

## Process-Based Inspection Tools

- Knowledge of process operations
- Process flow diagrams
- Material balance sheets
- TRI reports (EPCRA 313)
- Process observations







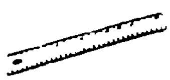

So tell me about your EMS

## EVOLUTION OF INVESTIGATION COMPLEXITY

- EMS EVALUATION
- PROCESS-BASED INSPECTION
- MULTIMEDIA COMPLIANCE INSPECTION
- SINGLE-MEDIA COMPLIANCE INSPECTION

## EPA EMS Policy Statement
**March 1998**

"Implementation of an EMS has the potential to improve an organization's environmental performance and compliance with regulatory requirements EPA supports and will help promote the development and use of EMSs "



## Innovations Task Force Report
July 1999

**Action Item 2:** Promote the use of EMSs

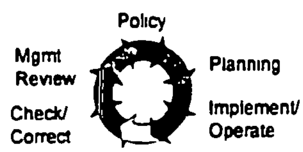
**Objective:** We will encourage organizations to use EMSs that improve compliance, pollution prevention and other measures of environmental performance

**Task 2:** We will promote the use of EMSs to address known compliance and performance problems

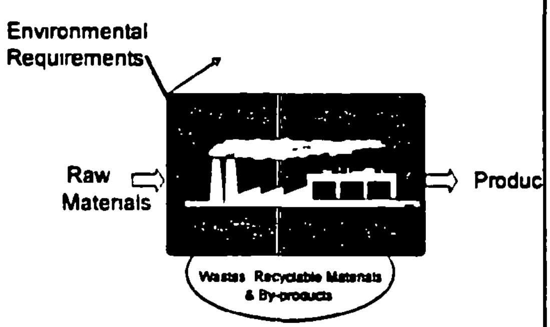
## EMS Definition

ISO 14001

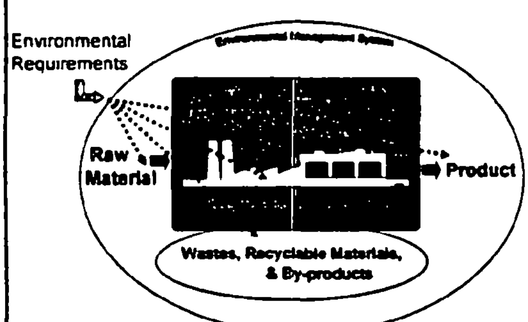
The part of the overall management system .. for .. implementing .. the environmental policy




## INDUSTRY WITHOUT AN EMS




## INDUSTRY WITH AN EMS

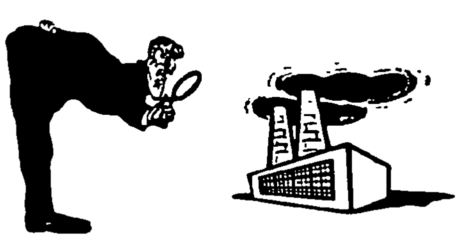


COMPLIANCE-FOCUSED
ENVIRONMENTAL MANAGEMENT SYSTEM
DEFINITION

"A system for identifying pertinent environmental requirements - statutes, regulations, permits, enforceable agreements, etc - and translating them into sustainable compliance activities at a facility."

NEIC 1996 parking lot sign

## NEIC EMS Evaluations



## Causal Analysis

Answers "why" noncompliance occurred

Aids in developing solutions to prevent recurrence

## Compliance-Focused EMS "Measuring Stick"

**Contained in August 1997 Publication**

**"NEIC Compliance-Focused EMS - Settlement Agreement Guidance"**

**ww.es.epa.gov/oeca/oceft/neic/12elemen.pdf**

## COMPLIANCE-FOCUSED EMS

**FIRST 6 ARE PRIMARY ELEMENTS**

1. **Management Policies and Procedures**
2. **Organization, Personnel, and Oversight**
3. **Accountability and Responsibility**
4. **Environmental Requirements**
5. **Assessment, Prevention and Control**
6. **Environmental Incident and Non-Compliance Investigations**

## COMPLIANCE-FOCUSED EMS

SECOND 6 ARE SUPPORT FUNCTIONS

7 Environmental Training, Awareness, and Competence

8 Environmental Planning and Organizational Decisionmaking

9 Maintenance of Records and Documentation

10 Pollution Prevention Program

11 Continuing Program Evaluation

12. Public Involvement/Community Outreach

## NEIC EMS Evaluation Objectives

Evaluate EMS vs. 12 CFEMS Elements

Determine underlying causes of noncompliance

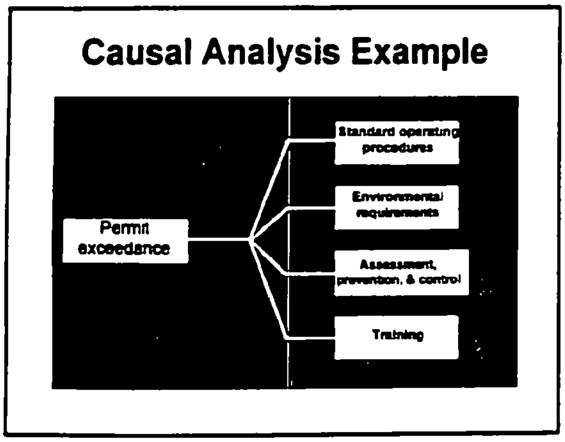
Obtain information for settlement purposes

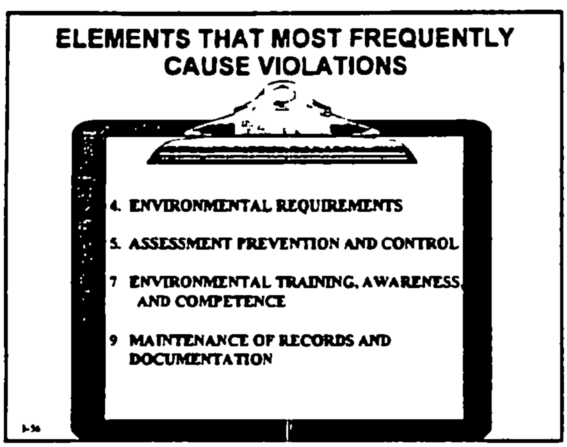
## Causal Analysis Method

Identify the problem (i.e., violation)

Identify the direct causes immediately preceding the problem

Identify the contributing or "root" causes using the 12 CFEMS elements


Causal Analysis Example
Permit exceedance
Standard operating procedures
Environmental requirements
Assessment, prevention, & control
Training


ELEMENTS THAT MOST FREQUENTLY CAUSE VIOLATIONS
4. ENVIRONMENTAL REQUIREMENTS
5. ASSESSMENT PREVENTION AND CONTROL
7 ENVIRONMENTAL TRAINING, AWARENESS, AND COMPETENCE
9 MAINTENANCE OF RECORDS AND DOCUMENTATION


Enforcement
Follow-up work

## Avoid the Engineer" Paradigm

To the engineer, all matter in the universe fits into two categories

(1) things that need to be fixed, and

(2) things that will need to be fixed after you've had a few minutes to play with them

5-11

## GENERIC INSPECTION CHECK LIST

- OBJECTIVES
  - What is the purpose of the inspection?

- TASKS
  - What records, files, permits, and regulations will be checked?
  - What coordination with laboratories, other programs, attorneys, and state or local governments is necessary?
  - What information must be collected?

- PROCEDURES
  - What specific processes of the facility will be inspected?
  - What procedures will be used?
  - Will the inspection require special procedures?
  - Has a QA/QC plan been developed, and is it understood?
  - Has a safety plan been developed, and is it understood?
  - What are the responsibilities of each member of the inspection team?

- RESOURCES
  - What personnel will be required?
  - What equipment will be required?

- SCHEDULE
  - What will be the time requirements?
  - What will be the order of inspection activities?
  - What will be the milestones (What must be done, compared with what is optional)?

PROJECT PLAN [*]

MULTI-MEDIA COMPLIANCE INVESTIGATION

XYZ COMPANY, MIDTOWN, ANYSTATE

## INTRODUCTION

The XYZ Company operates a plant at 1234 Anywhere Road in the middle part of Midtown, Anystate [Figure 1]. EPA Region XX requested that NEIC conduct a multi-media compliance investigation of the XYZ plant. The specific objectives of the investigation are to determine compliance with:

- Water pollution control regulations under the Clean Water Act (CWA), including wastewater pretreatment requirements and Spill Prevention and Control Countermeasures (SPCC) regulations
- Hazardous waste management regulations, under the Resource Conservation and Recovery Act (RCRA) and the Anystate Administrative Code (AAC)
- Underground Storage Tank (UST) regulations
- Air pollution control regulations under the Clean Air Act (CAA), Federal Implementation Plan (FIP), and the Federally approved portions of the State Implementation Plan (SIP)
- Toxic Substances Control Act (TSCA) PCB regulations
- Superfund Amendments Reauthorization Act, Title III, Emergency Planning and Community Right-To-Know Act (EPCRA) regulations

Compliance with other applicable environmental regulations may be determined by the NEIC. Region XX personnel will evaluate compliance with TSCA Sections 5, 8, 12, and 13 during the NEIC inspection, and report their findings separately.

## BACKGROUND

XYZ began operating the plant in 1492. Compounds A, B, and C; chemicals D, E, and F; pesticides G and H, and special containers for these materials have been manufactured on site. In 1942, some operations (formerly under the Middle Division) were acquired by a company known as "Newage, Inc." The remaining XYZ plant currently manufactures water soluble specialty items, and conducts research and development.

The XYZ plant employs a total of about 1,300 people, in a Primary Division, a Secondary Division, a Tertiary Division, and R and D Laboratory. The Primary Division manufactures compounds A, B, and C (240 tons in 1990). Raw materials for the compounds are purchased from

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[*] *Subject to revision*

an outside source. The Secondary Division makes chemicals and pesticides under numerous brand names (180 tons in 1990), and the Tertiary Division makes special containers for these materials (3 million containers in 1990). Research and development are conducted by R and D Laboratory.

The EPA Region XX Environmental Compliance Division, Midtown District Office (MDO), conducted a multi-media inspection of the XYZ plant during the first quarter of 1991. The MDO inspection report identified concerns with wastewater control, hazardous waste management, documentation, and spill prevention control.

Approximately 1.2 million gallons of wastewater per day are discharged to the Midtown Wastewater Treatment Plant (MWTP) of Midtown, Anystate. There are two direct National Pollutant Discharge Elimination System discharges (001 and 002) to the Midtown River at this facility. Additionally, sewered plant effluent discharge is regulated by the MWTP pretreatment standards, and the Federal effluent limitations and standards for the Compounds, Chemicals, Pesticides and Containers point source category. The R and D Laboratory conducts the Company's effluent analyses.

Violations of the MWTP pretreatment ordinance effluent limitations have occurred for solids, and the toxic standards. MWTP is concerned with data indicating the discharge of solids and toxics J, K, and L from the plant. XYZ also may have modified their pretreatment plant without obtaining a construction permit required by the Anystate Environmental Resources Department (AERD).

XYZ submitted the original RCRA Part A permit application on November 15, 1980. The application listed 19 hazardous waste management units, including 4 container storage areas, 10 storage tanks, and 5 storage surface impoundments. AERD is responsible for monitoring hazardous waste activities.

The facility's June 1990 contingency plan lists 14 above ground and 22 underground tanks on site. The tanks range in size from 2,000 to 50,000 gallons, with the majority between 5,000 and 20,000 gallons. These tanks are located in a tank farm area and near production areas.

The plant emits both volatile organics and particulates. There is no volatile organic constituent emission control equipment. Particulate emissions are controlled by three dust collectors. Five wet scrubbers are used to control fugitive particulate emissions when mixing bags of dry raw materials in reaction vessels. Air emissions are regulated by "Anystate Permits and Air Pollution regulations including AERD Operating Permits. EPA also promulgated a FIP on February 14, 1991.

On August 31, 1983, EPA Region XX conducted a PCB sampling inspection at the plant XYZ was fined for violations, including cracks in the floor of the PCB storage area, not conducting monthly inspections, no annual document, and not properly marking PCB transformers.

The Toxic Release Inventory (TRI) for this plant lists emissions of A, B, C, and D. The TRI also lists various inorganics, including E, F, G, and H.

## INVESTIGATIVE METHODS

Investigation objectives will be addressed by:

- Compilation and review of EPA, AERD, and MWTP database and file information
- Meetings with EPA Region XX personnel to discuss investigation specifics including: objectives, logistics, and potential sampling locations
- An on-site inspection

Meetings with Region XX personnel took place (date) The on-site inspection, scheduled to begin (date), will include:

- Discussing plant operations with facility personnel
- Reviewing and copying, as appropriate, facility documents including operating plans and records
- Visually inspecting plant facilities including processing, material storage, and waste handling facilities
- Sampling and analysis of appropriate waste streams and/or any unknown/unauthorized discharges to assist in compliance determination, as follows:

    (a) MWTP will collect and analyze wastewater samples for organic constituents during the week of (date). All QA/QC will be the responsibility of MWTP.

    (b) NEIC will collect wastewater samples for volatile organic constituent analysis during the on-site inspection. NEIC will conduct the associated analysis.

After completing the on-site inspection, NEIC investigators will brief appropriate EPA Region XX Program and Office of Regional Counsel per-sonnel regarding preliminary findings.

A draft report, including any analytical data, will be written by NEIC personnel and transmitted to EPA Region XX personnel for review and comment. A final report will be completed about two weeks after Region V comments are received. If analytical data are not available by (date), they will be presented in an addendum to the report.

NEIC personnel will be available for any additional support required (negotiations, litigation, etc.) until noncompliance issues are resolved.

DOCUMENT CONTROL PROCEDURES

NEIC document control procedures * will be followed during the investigation. TSCA "Notice of Inspection" and "Confidentiality" forms will be completed during the opening conference. Documents and records obtained from the Company will be uniquely numbered

* *NEIC Policies and Procedures Manual, revised August 1991*

and listed on docu-ment logs. Photograph logs will also be maintained A copy of th e document and photograph logs, w ith a Receipt For Samples/Document form, will be offered to the Company prior to completion of the on-site inspection. Any documents declared to be confidential business information pursuant to 40 CFR Part 2 will be so noted on the log and secured appropriately.

SAFETY PROCEDURES

Safety procedures to be followed during the on- site inspection will comply with those described in the attached safety p lan [Appendix A], and established NEIC safety procedures. These procedures are contained in EPA 1440 - Occupational Healt h and Safety Manual (1986 edition), Agency orders and applicable provisions of the NIOS H/OSHA/USCG/EPA Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities. Th e Company's safety policies will also be reviewed and followed.

TENTATIVE SCHEDULE

(date) Region XX will notify facility of inspection (verbally and in writing)
(date) Initiate on-site inspection
(date) Brief Region V regarding preliminary findings
(date) Draft report to Region V

APPENDIX

NEIC
SAFETY PLAN
FOR
HAZARDOUS SUBSTANCES RESPONSES AND FIELD INVESTIGATIONS

The OSHA Hazardous Waste Site Worker Standards (29 CFR 1910.120) and EPA protocols require certain safety planning efforts prior to field activities. The following format is aligned with these requirements. Extensive training and certifications are required in addition to this plan

PROJECT: ______________________ NEIC Reporting Code: ________

Project Coordinator: ______________________ Date: ____________
Branch Chief: ______________________ Date: ____________
On Scene Coordinator or Supervisor:

______________________________________________

Health and Safety Manager:

Approval: ______________________ Date: ____________

DESCRIPTION OF ACTIVITY

If any of the following information is unavailable, mark "UA"; if covered in project plan, mark "PP".

Site Name: ______________________________________________
Location and approximate size: ______________________________
Description of the response activity and/or the job tasks to be performed:
______________________________________________
______________________________________________

Duration of the Planned Employee Activity: ______________________
______________________________________________

Proposed Date of Beginning the Investigation: ______________________
Site Topography: ______________________________________________

Site Accessibility by Air and Roads: ______________________________

# HAZARDOUS SUBSTANCES AND HEALTH HAZARDS INVOLVED OR SUSPECTED AT THE SITE

***Fill in any information that is known or suspected***

| Areas of Concern | Chemical and Physical Properties | Identity of Substance and Precautions |
|---|---|---|
| Explosivity: | ________ | ________<br>________<br>________ |
| Radioactivity: | ________ | ________<br>________<br>________ |
| Oxygen Deficiency:<br>(e.g., Confined Spaces) | ________ | ________<br>________<br>________ |
| Toxic Gases: | ________ | ________<br>________<br>________ |
| Skin/Eye Contact Hazards: | ________ | ________<br>________<br>________ |
| Heat Stress: | ________ | ________<br>________<br>________ |

Pathways from site for hazardous substance dispersion: ________________________________
________________________________________________________________
________________________________________________________

## WORK PLAN INSTRUCTIONS

A. Recommended Level of Protection: A _____ B _____ C _____ D _____

Cartridge Type, if Level C: ______________________________

Additional Safety Clothing/Equipment: ______________________________
________________________________________________________________
________________________________________________________________
________________________________________________________________
________________________________________________________

Monitoring Equipment to be Used: ______________________________

CONTRACTOR PERSONNEL:

Number and Skills ______________________________

CONTRACTOR SAFETY CLOTHING/EQUIPMENT REQUIRED. ______________

Have contractors received OSHA required training and certification?
(29 CFR 1910.120)
Yes _____ Not Required _____

(If "yes", copy of training certificate(s) must be obtained from contractor)

B. Field Investigation and Decontamination Procedures:

Decontamination Procedures (contaminated protective clothing, instruments, equipment, etc.): __

Disposal Procedures (contaminated equipment, supplies, disposal items, washwater, etc.): _____

## IV. EMERGENCY CONTACTS

Hospital Phone No.: ______________________

Hospital Location: ______________________

EMT/Ambulance Phone No.: ______________________
Fire Assistance Phone No.: ______________________

NEIC Health and Safety Manager: Steve Fletcher - 303/236-5111
FTS 776-5111

Radiation Assistance: Wayne Bliss, Director
Office of Radiation Programs
Las Vegas Facility (ORP-LVF)
702/798-2476
FTS 545-2476

## SESSION 6

## TOPIC: ADMINISTRATIVE ISSUES

**Time: 30 minutes**

**PURPOSE**

- Respond to questions concerning administrative procedures

Note: This session is not long enough to cover all administrative issues. Further, this session concentrates on federal administrative issues; state employees should check with their individual administrative officers to determine their specific requirements. Nevertheless, this session is generic enough to apply in most cases to both federal and state inspectors.

**KEY POINTS**

- Anticipate administrative needs in advance and obtain the proper forms, authorizations, and signatures, and meet other requirements.
- An inspector can be held financially liable for unauthorized purchases.

**LIST OF VISUALS**

6-1 Administrative Issues (Title Slide)
6-2 Administrative Issues
6-3 Administrative Issues
6-4 Procurement
6-5 Administrative Issues
6-6 Pay Issues
6-7 Administrative Issues

## Administrative Issues

6-1

## ADMINISTRATIVE ISSUES

- Planning
- Procurement
- Travel & Reimbursement
- Pay Administration
- Special Cautions

6-2

## ADMINISTRATIVE ISSUES

- Anticipate administrative needs
- Obtain proper forms, authorizations & signatures
- Advance planning a must, but
  - Change in travel plans may be necessary
  - Unanticipated purchases may be necessary

6-3

## ADMINISTRATIVE ISSUES PROCUREMENT

- Supplies (ice, sampling equipment, etc.)
- Document copies
- Photographic processing
- Shipping
- You may be held liable for unauthorized purchases !!!

## ADMINISTRATIVE ISSUES

- Cash advance
- Reservations
  - Airlines
  - Hotel
  - Car
- Use of cash
  - Complete travel vouchers promptly
- Receipts
- Know your procedures

## ADMINISTRATIVE ISSUES PAY ISSUES

- Overtime or comp time for:
  - Work
  - Travel
  - Training
- Special pay
- Know your procedures

## ADMINISTRATIVE ISSUES

- Use of Government equipment:
  - Cars
  - Telephones
  - Computers, fax machines, copiers, etc
- Meals
- Airline benefits

6-7

7

## SESSION 7

## TOPIC: SOURCES OF INFORMATION

**Time: 45 minutes**

**PURPOSE**

- Acquaint the participants with the wide range of information that is available to the inspector before the inspection.

**KEY POINTS**

- Prior planning, including extensive review of all sources of information, is crucial to the successful and professional accomplishment of an inspection.

**LIST OF VISUALS**

7-1 Sources of Information (Title Slide)
7-2 Sources of Information (general)
7-3 Think Paper and Electronic
7-4 Federal, State, and Local Files (facility-specific)
7-5 Federal, State, and Local Files (continued)
7-6 Reconnaissance Visit
7-7 Media Program Databases
7-8 Compliance Databases
7-9 http.//es.epa.gov
7-10 IDEA
7-11 Envirofacts
7-12 Envirofacts
7-13 OTIS
7-14 OECA
7-15 Office of Compliance
7-16 EPA Inspector Information Exchange Web Site
7-16 NEIC
7-17 NETI
7-18 NEIC Library
7-19 Commercial Information Sources
7-20 RAIN home page

**LIST OF HANDOUTS**

7-1 Sources of Information
7-2 OTIS and other information based Web Sites
7-3 New EPA Inspector Information Exchange Web Site

## Sources of Information

7-1

## Sources of Information
(general)

- Statute and regulation publications
- Policies, data, and guidance from all government levels (incl. cross-agency)
- Case histories and precedent (dockets)
- Division libraries and central filings
- Trade associations

7-2

## Think Paper and Electronic

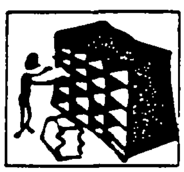
7-3

## Federal, State, and Local Files
(facility-specific)

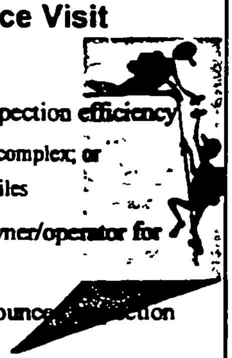
- Permits, applications, exemptions, waivers
- Process diagrams, waste stream flow charts, and facility maps
- Self-monitoring data, records, and reports
  - Including annual and per-incident reports, manifests
- Prior inspection reports
  - Including cross-program issues

## Federal, State, and Local Files
(facility-specific) (continued)

- QA documentation
  - Prior sampling test results
  - Lab certification
- Enforcement documents
- Facility responses to enforcement actions
- Citizen complaints



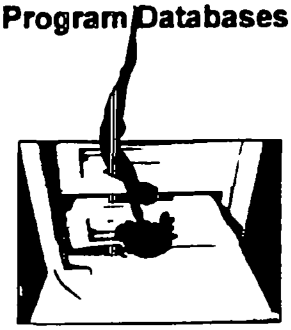
## Reconnaissance Visit



- Done to increase on-site inspection efficiency
  - Especially if facility is very complex; or
  - There is not enough info in files
- Inspector may meet with owner/operator for logistical planning
- But not used with an unannounced inspection

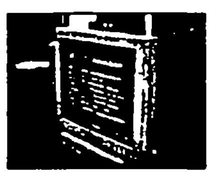
## Media Program Databases

- AIRS
- AFS
- PCS
- RCRAInfo
- TRIS
- CERCLIS



## Compliance Databases

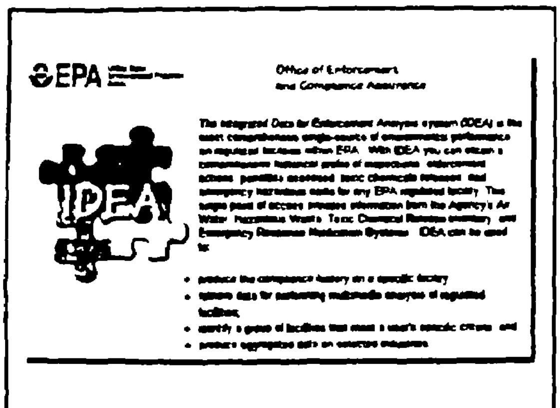
- IDEA
- ENVIROFACTS
- OTIS
- Sector Facility Indexing



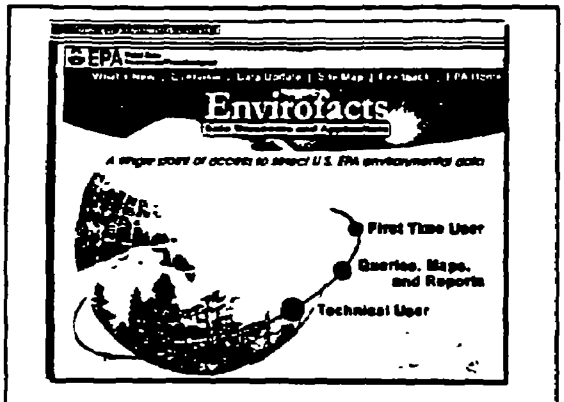
EnviroSense

SEARCH EPA FEEDBACK SITE MAP HELP ABOUT

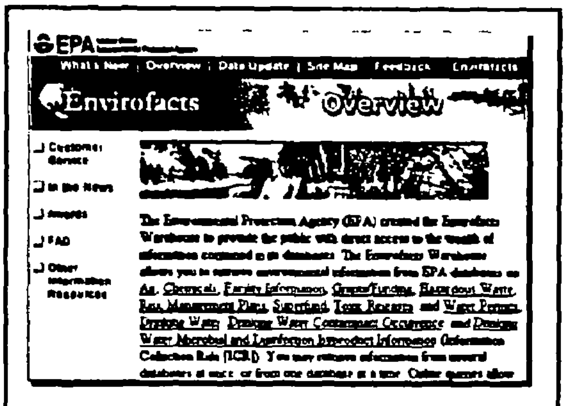
**http://es.epa.gov/**


EPA
IDEA

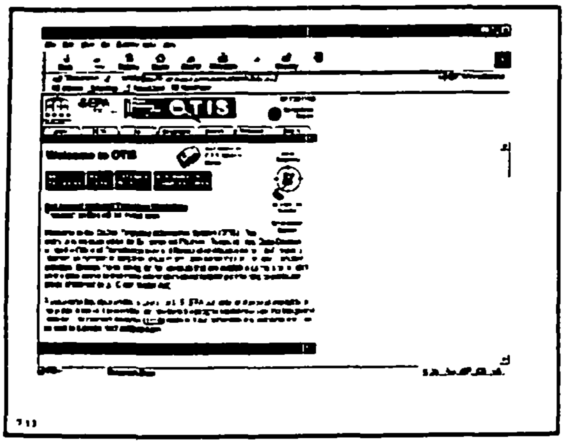
7-40

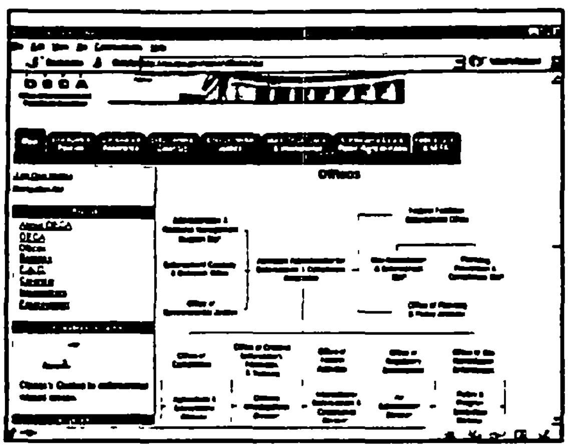

EPA
Envirofacts
First Time User
Queries, Maps, and Reports
Technical User

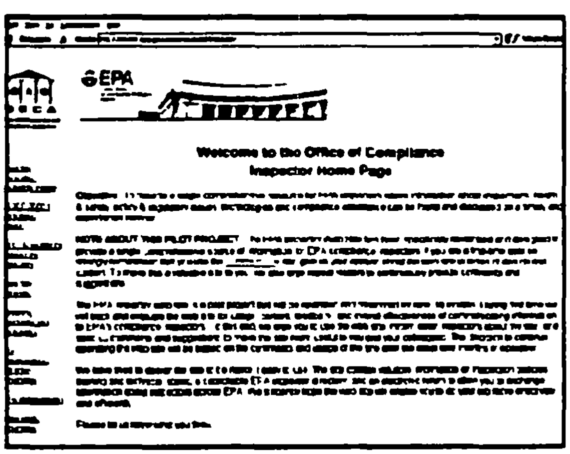
7-41

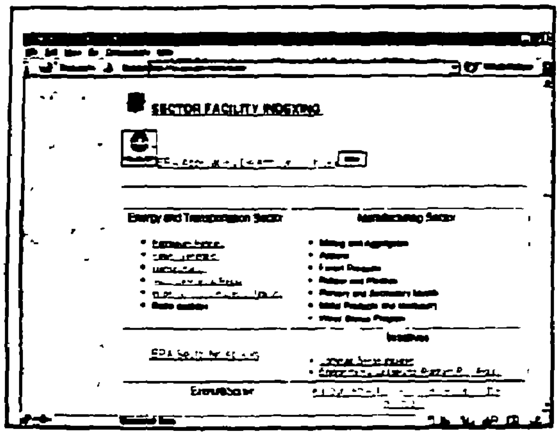

EPA
What's New | Overview | Data Update | Site Map | Feedback | Envirofacts
Envirofacts
Overview
In the News
FAQ
Other Information Resources

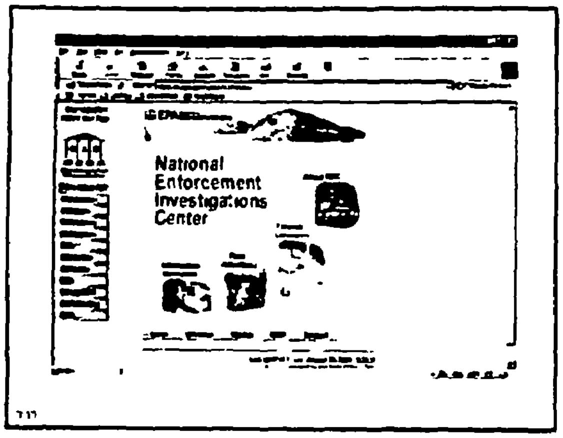
7-42

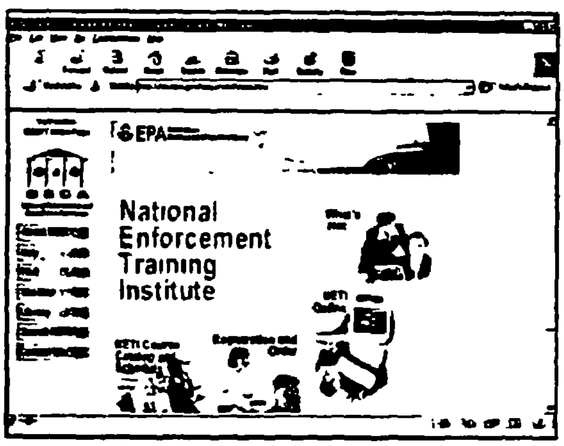

OTIS
Welcome to OTIS

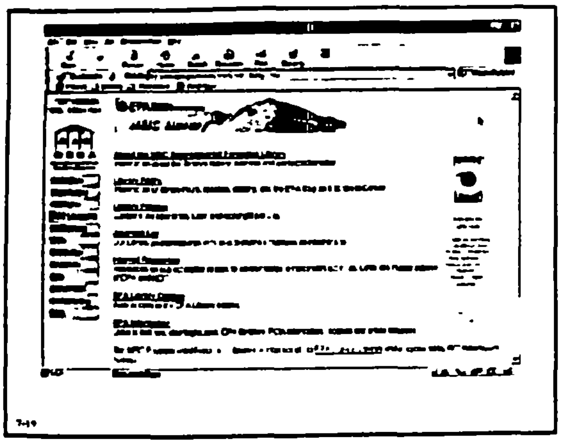

OECA
Offices


EPA
Welcome to the Office of Compliance
Inspector Home Page


SECTOR FACILITY INDEXING
Energy and Transportation Sector


National
Enforcement
Investigations
Center

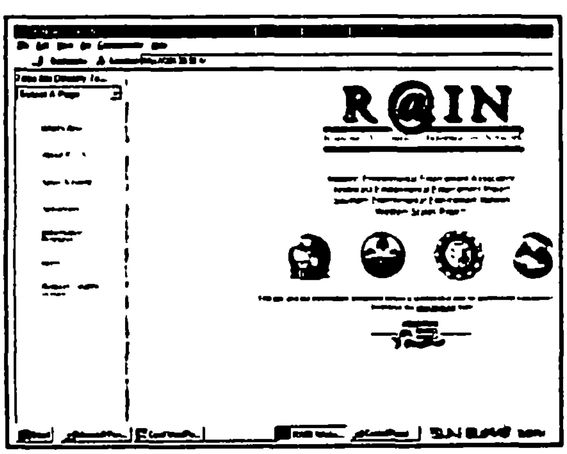

EPA
National
Enforcement
Training
Institute



## COMMERCIAL INFORMATION SOURCES

- AutoTrak
- CBDInfo
- DIALOG
- Dun & Bradstreet
- LEXIS/NEXIS
- STN
- TOMES
- Westlaw

7-20



| System | Description | Application |
|---|---|---|
| Chemical Information System (CIS) | The CIS is a collection of scientific and regulatory databases containing numeric, textual, and some bibliographic information in the areas of toxicology, environment, regulations, spectroscopy, and chemical and physical properties. | NEIC uses the CIS to locate mass spectral information; environmental fate information; formulation ingredients for commercially available products, such as pesticides; and waste disposal methods for hazardous substances. |
| Colorado Alliance of Research Libraries (CARL) | The CARL system includes the catalogs of the member libraries, an index of more than 10,000 periodicals, a full-text encyclopedia, Choice book reviews, and a bibliography of GPO publications. | CARL is searched by NEIC staff for general reference, to locate books, and to identify articles and documents. |
| DataTimes | DataTimes provides on-line access to numerous full-text databases, including newspapers, wire services, and Dow Jones News/Retrieval. | DataTimes is a source of national environmental news. Newspaper databases from all regions are updated daily. |
| Knight-Ridder Information Services, Inc. | The DIALOG system contains more than 330 databases covering a variety of disciplines: science, technology, engineering, social sciences, business, and economics. The databases contain more than 120,000,000 records and are regularly updated to provide the most recent information. | NEIC uses the DIALOG databases to obtain: (1) expert witness information, including biographies, publications, and congressional testimony; (2) up-to-date pollution control technology for hazardous waste, air, and water; and (3) business information *such as corporate officers, subsidiaries, and line of* business. |
| Dun & Bradstreet | Dun & Bradstreet, a credit-reporting firm, provides business information reports for privately and publicly owned companies and government activity reports that list federal contracts, grants, fines, and debarments for specific companies. | NEIC uses the Dun & Bradstreet system to locate corporate information, such as business done by the company, company history, financial condition, subsidiaries, and corporate officers for privately held companies. |
| Groundwater On-Line (GWOL) | The National Groundwater Information Center database is a bibliographic database containing references to materials on hydrogeology and water well technology, with emphasis on reports or projects sponsored by EPA. | NEIC accesses GWOL to locate publications on groundwater topics and to verify or locate groundwater experts. |
| NEXIS/LEXIS | NEXIS/LEXIS contains the full text of more than 600 business and general news files, including the *Washington Post* and *New York Times*. Statutory and case law are provided for computer-aided legal research. | NEIC uses NEXIS/LEXIS to keep informed of the latest EPA and environmental news stories and to track the corporate and financial status of U.S. businesses involved in environmental litigation. |

| System | Description | Application |
|---|---|---|
| National Library of Medicine (NLM) | The National Library of Medicine system contains more than 5 million references to journal articles and books in the health sciences published since 1965. | NEIC uses the NLM system to obtain: (1) information about toxicity and environmental health effects for individual chemicals or groups of chemicals, (2) physical and chemical properties of specific compounds, (3) analytical methodology references, and (4) carcinogenic bioassay information from the National Cancer Institute. |
| Scientific and Technical Information Network (STN) | The STN system contains databases covering chemistry, science, and engineering that are regularly updated to provide the most recent information. STN has strong coverage of European and Japanese scientific databases. | NEIC uses the STN databases to obtain: (1) chemical structures and synonyms for a chemical compound, (2) analytical methods and techniques, and (3) toxicity of a chemical compound.<br><br>NEIC can acquire source information and location, service areas, geographic areas, and historical information. Information on noncompliance and enforcement actions also can be obtained. |
| WESTLAW | The WESTLAW system contains legal information, including the full text of cases from the Supreme Court, U.S. courts of appeals, U.S. district courts, and state courts. It contains Shepards' Citations, regulatory information from the Code of Federal Regulations, Federal Register, and U.S. Code and expert witness information from the Forensic Services Directory. | NEIC uses WESTLAW to identify precedent cases, to locate all cases decided by a certain judge or all cases represented by a certain attorney, and to locate possible expert witnesses.. |

URL:http:/intranet.epa.gov/oeca/oc/eptdd/teb/otis

# Welcome to OTIS

Subscribe to OTIS Update News

Online Targeting Information System

What's New

Search

IDEA Web Queries | SNC Tracker | Compliance Report | Enviromapper For Compliance Analysis

**3rd Annual National Targeting Workshop**
Presentation files will be linked soon.

Welcome to the *Online Targeting Information System* (OTIS)! This website is maintained by the Enforcement Planning, Targeting, and Data Division within the Office of Compliance to assist Regional and headquarters staff in using information systems to target for inspections, enforcement priorities, and compliance activities. Browse the following list for key tools that are available on the site, or click on the tabs above to find more information about targeting pertaining to particular areas of interest (e.g., Clean Water Act).

Please note that data on this site are for U.S. EPA use only, and are not available on the public Internet. Data on the site are from the program databases via the Integrated Data for Enforcement Analysis (IDEA) system. Your comments are welcome and can be sent to barrette.michael@epa.gov.

## Available OTIS Tools

### IDEA Web Query

The IDEA Web query provides IDEA functionality on a web browser and generates compliance data in real-time based on selections including facility name, sector, compliance status, and enforcement history Data are available at the facility level that allow users to view information about the nature of violations that occurred and to rank facilities for inspection or enforcement priority Each media currently displays a separate query interface (Water-CWA, Air-CAA, and RCRA) Ongoing enhancements are planned for the IDEA Web Query tools

### SNC Tracker On-line Search

The SNC Tracker provides a quick way to view facilities in a state or Region that are currently listed as SNC (or HPV) The interface looks at the most recent information in the IDEA system, and provides either a single media or cross media result based upon selections of CAA (AFS), CWA (PCS), and RCRA (RCRIS) data The site can be searched by state, Region, or environmental program of concern and may be used to plan multimedia inspections or cases against facilities with multimedia compliance issues This feature replaces the capability of the CWA- and MultiMedia SNC Trackers as well as the planned CAA-HPV and RCRA-SNC Trackers

### Get Compliance Report

IDEA data are available in a web browser by clicking the "Get Compliance Report" button in the top right corner of the OTIS screen This feature lets you specify a facility name or permit number and generate a detailed report including data from AFS, RCRIS, PCS and TRIS New features include data on TRI releases by chemical and 5-year option for displaying enforcement actions and inspections

## BETA Version OTIS Tools

### EnviroMapper for Compliance Analysis

The newest EnviroMapper site gives users a dynamic way to use maps to target facilities by compliance status and/or time since last inspected This site will allow you to map RCRA, Clean Air Act (CAA), and Clean Water Act (CWA) facilities in a given geographic area (Beta version for comment - please provide feedback)

### Geographic Analysis Tool

Use this tool to assess environmental conditions by county, and check to see whether enforcement presence is high or low by county The tool is designed to identify geographic areas that may be ripe for more compliance monitoring Sort county rankings by media, or multimedia, and use the website to provide color maps This product is the result of a EPA/Regional Workgroup which supported the MOA process (Beta version for comment - data pulls completed in October, 1998)

### Sector Analysis Tool

Which sectors in your Region or state have the highest noncompliance rates or pollutant releases? Use this tool to view rankings that show inspection coverage, violation rates, and emissions data for 113 sectors Data was used to support FY'00 MOA national sector priorities (Beta version for comment - data pulls completed in October, 1998).

## Coming Soon to OTIS

The Targeting and Evaluation Branch is currently developing a new set of capabilities for OTIS that will be launched in October, 2000 (Version 3) Please contact Michael Barrette if you would like to provide input during the process of defining new features

## Leave OTIS to Search Other Internet Sites

EPA Sector Facility Indexing Project
Provides detailed compliance and release data for 5 industry sectors

EPA EnviroFacts Warehouse
Allows basic searching for facility-level information in multiple databases

TRI Explorer
Provides web-based query and analysis functions for the 1998 Toxics Release Inventory data set

RTK Net EXIT EPA
Provides data searching from multiple EPA systems

EPA Emergency Response Notification System (ERNS)
View information on reported spills

AIRSWeb
View air pollutant release data

ORE Enforcement Alert
Is a new informational newsletter published by the Office of Regulatory Enforcement that informs and educates the public and regulated community of important environmental enforcement issues, recent trends and significant enforcement actions

EPA Surf your Watershed
Office of Water Website lists information on watershed quality

Environmental Defense Fund Scorecard EXIT EPA
Provides geographical search capabilities focused on TRI release data

Securities and Exchange Commission (EDGAR) EXIT EPA
Use this site to find corporate disclosures of environmental liability information, and to verify that EPA sanctions over $100,000 have been reported to SEC

FreeEDGAR EXIT EPA
Site allows you to perform text searches into SEC documents to look for corporate information Provides more functionality than the Security and Exchange Commission site

OPPT Environmental Indicators Model
View EPA environmental indicator data aggregated nationally or by state, county or zip code based upon the TRI-based risk screening model that OECA has adopted for enforcement targeting

OECA Data Systems and Models
Provides descriptions and links to searchable databases and models used by OECA

EPA EnviroMapper
Maps several types of environmental information, including drinking water, toxic and air releases, hazardous waste, water discharge permits, and Superfund sites

National Atlas of the United States EXIT EPA
Interactive maps with environmental, biological, geological and demographic layers

OSHA Statistics and Data EXIT EPA
Access query tools that locate OSHA inspections conducted within a particular establishment or within a particular industry

group

## Leave OTIS for Regional Enforcement, Compliance, or Targeting Data on the Internet

Region 1 Data and Software
Provides TRI data for New England States

Region 2 Geographic Information Systems
Information on Regional GIS projects, data and software

Region 3 Databases and Software
Provides links to searchable databases and environmental maps

Region 4 Geographic Information Systems and Information Resources
Regional databases and GIS applications

Region 5 Enforcement Action Database
Regional enforcements actions for FY 2000 by name, state, or statute

Region 5 Enforcement Reports
Summary of Regional enforcement activity by fiscal year (1992-1998) or type (SEP, Compliance Orders, penalties, or trends)

Region 6 Comparative Risk Publications
Contains Regional analyses covering Human Health Risk, Environmental Justice Index, Comparative Risk Report, and Federal Facilities Risk Index, plus links to national sites

Region 8 Geographic Information Systems Data
Inventory of GIS web and mapping links plus links to Region 8 state and Federal environmental data sites

## Leave OTIS for State Enforcement, Compliance, or Targeting Data on the Internet

California Geotracker
Geographic Information System with online access to regulatory data about underground fuel tanks, pipelines and drinking water systems in the State of California

Pennsylvania EFACTS
(Environment, Facility, Application, Compliance Tracking System)

*If you would like us to add a link to this site, please send a message to royer.elizabeth@epa.gov*

EPA United States Environmental Protection Agency

Office of Enforcement and Compliance Assurance

- About IDEA
- Getting Started
- Data Sources
- Building Queries
- IDEA Reports
- User Documentation
- Support Services
- FAQ

The Integrated Data for Enforcement Analysis system (IDEA) is a comprehensive single-source of environmental performance on regulated facilities within EPA. With IDEA you can obtain a comprehensive historical profile of inspections, enforcement actions, penalties assessed, toxic chemicals released, and emergency hazardous spills for any EPA regulated facility. This single point of access provides information from the Agency's Air, Water, Hazardous Waste, Toxic Chemical Release Inventory, and Emergency Response Notification Systems. IDEA can be used to:

- produce the compliance history on a specific facility,
- retrieve data for performing multimedia analysis of regulated facilities,
- identify a group of facilities that meet a user's specific criteria; and
- produce aggregated data on selected industries.

IDEA Home OECA Home EPA Home Enviro$ense
Search Site Map What's New? Contact Us

*Last Updated April 13, 2000*

URL: http://www epa gov/oeca/idea/

About IDEA
Getting Started
Data Sources
Building Queries
IDEA Reports
User Documentation
Support Services
FAQ

# Getting Started

To gain access to IDEA, all users must have a user ID and a way to access the EPA mainframe. Steps for getting started vary according to the type of user.

Select the user group that best describes you to learn more about gaining access to IDEA.

- EPA Employee
- Federal or State Employee
- Member of the Public

IDEA Home OECA Home EPA Home Enviro$ense
Search Site Map What's New? Contact Us

*Last Updated: April 13, 2000*

# Getting Started

## EPA Employees

These instructions are specifically for EPA Employees and direct support contractors who are either on-site at an EPA location or have direct access to EPA's wide area network via a local area network (LAN). Before you get started, you must:

1. Obtain a mainframe user ID and account.
2. Determine which access method and interface is appropriate for your use.
3. Determine if you qualify for enforcement sensitive data access.

Once you have selected and installed the appropriate IDEA software, you must obtain a mainframe user ID. Please note if you already have a mainframe user ID and password there are no additional requirements for accessing IDEA and/or IDEAWin as any valid mainframe user ID/account will work.



*Last Updated April 13, 2000*

URL http://www epa gov/oeca/idea/epa.html

# Getting Started

## State and Other Federal Agency Employees

These instructions are specifically for State or other Federal Agencies. Before you get started, you must:

1. Obtain a mainframe user ID and account.
2. Determine which access method and interface is appropriate for your use.
3. Determine if you qualify for state-sensitive access (state-sensitive access allows you access to more data, for example, the Occupational Safety and Health Administration (OSHA) system).

Once you have selected and installed the appropriate IDEA software, you must have a mainframe user ID. Please note if you already have a mainframe user ID and password there are no additional requirements for accessing IDEA and/or IDEAWin as any valid mainframe user ID/account will work. If you do not have an ID you must obtain one by following instructions for obtaining a mainframe user ID (see link at top of page).

Other Federal Agencies and State Environmental Enforcement Personnel may qualify for state enforcement data access. To decide whether or not you qualify for state enforcement data access, read the instructions for determining if you qualify for access (see link at top of page).



*Last Updated. April 13, 2000*

URL: http://www.epa.gov/oeca/idea/state.html

Envirofacts Text Version

EPA United States Environmental Protection Agency

WHAT'S NEW
SITE MAP
IN THE NEWS
DATA UPDATE
AWARDS
FEEDBACK
FAQ
CONNECT INFO
RELATED LINKS
EPA HOME

# Envirofacts Warehouse

A single point of access to select U S EPA environmental data

**Read about the environmental databases in Envirofacts.**

Overview of Available Data | Select an Overview

**Query the database and generate reports.**

Generate Reports from Data | Select a Query Form

**Produce maps of environmental information.**

Maps On Demand | Select a Mapping Tool

**Facility Linkage Application**

Envirofacts Warehouse Website
URL http://intranet.epa.gov/enviro
*This page was updated June 07, 2000*

URL:http:/intranet.epa.gov/enviro

# RCRAInfo Design Team Updates

Tuesday, July 11, 2000

EPA's Home Page
OSW Partnership Web

## What Can You Find on This Site?

On this site you can find all of the late-breaking news concerning the RCRAInfo project, including manuals, guides, and presentations

Work continues as we move the Resource Conservation and Recovery Information System (RCRIS) and the Biennial Reporting System (BRS) from a mainframe-based FOCUS database to a web-enabled Oracle platform

RCRAInfo are y
"point and click?"

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| | | |
|---|---|---|
| **General Documentation** | | |
| | Who is on the Design Team? | 05/23/2000 |
| | What is the RCRIS Project Scope? | |
| | What is the BRS Project Scope? | |
| | The RCRAInfo High Level Design | |
| | RCRAInfo High Level Design - Response to Comments | |
| | Cover Memo for the Response to Comments | |
| **RCRAInfo National Training Materials** | | |
| | Invitation to RCRAInfo Training | 05/23/2000 |
| | Draft Agenda | 05/23/2000 |
| | Registration Forms - PDF and WordPerfect | 05/23/2000 |
| | Hotel Logistics | 05/23/2000 |
| **Technical Documentation** | | |
| | Structure Charts | |
| | Security | 05/25/2000 |
| Upd | Handler (Part 1, Part 2) | 07/10/2000 |
| | Permitting | 07/02/2000 |
| | Corrective Action | 07/02/2000 |
| | CM&E (Part 1, Part 2) | 07/02/2000 |
| | National Biennial Report (Part 1, Part 2, Part 3) | 07/02/2000 |
| | Table Lists (English version of the structure charts) | |
| | Security | 05/25/2000 |
| | Handler | 05/25/2000 |
| | Permitting | 05/25/2000 |
| | Corrective Action | 06/28/2000 |
| | CM&E | 06/28/2000 |
| | National Biennial Report | 06/28/2000 |
| | RCRIS / BRS to RCRAInfo Crosswalks | |
| | Handler | 05/19/2000 |
| | Permitting | 05/19/2000 |
| | Corrective Action | 05/19/2000 |
| | CM&E | 06/28/2000 |
| | National Biennial Report | 06/28/2000 |
| | Guides, Guidance, and Manuals | |
| | Translator Guide - draft version 3 | 02/09/2000 |
| | RCRAInfo Final File Specification Guide for 1999 Hazardous Waste Report Submissions (Summary of Changes) | 11/1999 |

| | |
|---|---|
| RCRAInfo User Acceptance Testing Guide | 05/25/2000 |
| The Placemats - Legal Status, Operating Status, and Universes | 05/25/2000 |
| Conversion Issues for RCRIS to RCRAInfo Final Data Conversion Survey: PDF or WordPerfect | |

**Reporting in RCRAInfo**

| | |
|---|---|
| Reports List | 05/24/2000 |
| Functional Specifications for RCRAInfo National Reports | 06/08/2000 |
| Building RCRAInfo Web Reports | |

**1999 Biennial Reporting Cycle**

| | |
|---|---|
| 1999 Biennial Report Implementation Schedule | 05/23/2000 |

**RCRAInfo Status and Plans**

| | | |
|---|---|---|
| | RCRIS to RCRAInfo Final Data Conversion Schedule | 07/02/2000 |
| | Briefing for the ASTSWMO Conference | 04/27/1999 |
| | Briefing for the Regional Branch Chiefs Meeting | 04/29/1999 |
| | RCRAInfo: The Paradigm Shifts. A presentation by Design Team members Steve Cobb (AL) and Mira Neumiller (CO) | |
| New | Briefing for the ASTSWMO Conference | 07/10/2000 |

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Submit comments, suggestions, or questions about this site to the RCRAInfo HelpDesk
Unless otherwise noted, all documentation contained on this site is maintained in Adobe Acrobat's PDF format You ca
viewer at Abode's web site - http://www.adobe.com EPA personnel may already have this software installed on their P
available through their local LANs Due to the size of some of these files, it is recommended that you download the doc
rather than trying to read them on-line

TEXT VERSION
OCEFT Main Page

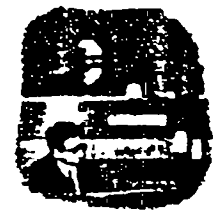
OECA
Office of Enforcement and Compliance Assurance

- NEIC Home
- Organization
- Highlights
- NEIC Library
- Publications
- FAQ
- What's New
- Contact Us
- Help
- Search NEIC
- NEIC Site Map
- EOIA

EPA United States Environmental Protection Agency

# National Enforcement Investigations Center

About NEIC



Forensics Laboratory

Field Activities

Information Resources

Search | EPA Home | Site Map | OECA | Feedback

Last Modified Friday, January 28, 2000 15 09 37
Page maintained by Kimberly O'Neill, GCI
http://www epa gov/oeca/oceft/neic/

URL:http://es.epa.gov/oeca/oceft/neic

07/11/2000 12:40 PM

# *NEIC Library*

## *Web Sites for Inspectors*

### EPA/Environment Sites

NEIC Web Site
http://www.epa.gov/oeca/oceft/neic
Information about NEIC's background and organization. Links to enforcement publications and relevant Internet information.

EPA Web Site
http://www.epa.gov/
Contains basic information about the EPA, press releases, offices, programs, databases and a calender of events.

EPA Intranet Site
http://intranet.epa.gov/agcyintr
Internal information for EPA, includes technical documents.

Envirofacts
http://www.epa.gov/docs/enviro/html/ef_home.html/
A U.S EPA relational database that integrates data from four major EPA program systems. permit compliance system (PCS),CERCLIS, TRIS, RCRIS

Environmental Information on the Internet
http://spot.colorado.edu/~jobem/envrscs.htm
This site is comprehensive listing of environmental information sites compiled by Peggy Jobe of University of Colorado-Boulder.

Regional Associations Information Network (R@IN)
http://www.epa.gov/oeca/neti/rainintr.html
An information resource designed for environmental regulatory and enforcement professionals. The site is password protected and is for use by government personnel only. You can sign up on the web.

TTN2000: TTN Bulletin Board
http://ttnwww.rtpnc.epa.gov/
Web interface for TTNBBS, includes AIRS, CHIEF, CAAA, NATICH BBS and more.

## Government/Regulatory Information

CFR-Title 40
http.//www.epa.gov/docs/epacfr40/chapt-I.info
The CFR in PDF format. Use Adobe Acrobat Reader to view and print.

FedWorld
http://www.fedworld.gov/
A searchable database of government agency web sites, as well as links to a variety of sites and databases.

FedLaw
http://www.legal.gsa.gov
References for people conducting Federal legal research.

OSHA
http://www.osha-slc.gov/
Information on OSHA standards, technical links and training.

Thomas
http://thomas.loc.gov
Congressional information on the web, including the text and legislative histories of bills from the 103-105 Congresses.

## People, Company and Place Finders

Four11
http://www.Four11.com
Find email addresses.

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http://www.mapquest.com
Allows you to locate an address by generating a street map.

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http://www.switchboard.com
Find listed phone numbers and addresses by name, city or state.

## Chemical

Chemfinder
http://chemfinder.camsoft.com
Searches various environmental databases for chemicals by name, CAS number, molecular weight, and formula.

Vermont SIRI MSDS
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| Phrase Searching | Use quotes | Use quotes or choose "the exact phrase" | Select "the exact phrase" or use quotes | Use quotes | Use quotes |
| Truncation | forest* (finds forests, forestry etc ) | n/a | Use word stemming in advance search A search of "light" will find lights, lighting, etc | Use $ to add variations to end of word | Use * to replace multiple characters, use % to replace one character |
| AND searching | Use & or AND in Adv Search cat & dog | all the words | Choose "all of the words", or use AND | Use AND, cat AND dog | Default |
| Or searching | Default | any of the words | Choose "any of the words",or use OR | OR | OR |
| Not searching | Use !/AND NOT, cat ! dog | -WORD | !/NOT, cat!dog | AND NOT | NOT |
| Proximity searching | NEAR grey NEAR hound finds grey within 10 words of hound | n/a | n/a | n/a | n/a |
| Limits | Use +/- in simple search to eliminate or add words Language time period, refine option | n/a | Date, Country, Language, Use +/- to add or eliminate terms, media type, domain name | Use +/- to add or eliminate words | Use +/- to add or eliminate words, in Power Search use date, document types |
| Ranking | Relevance ranking in simple search | n/a | Relevance ranking | Relevance ranking | n/a |
| Nesting | Nesting to set search order Use ( ) | n/a | Use ( ) to create complex searches | Use ( ) to create complex searches, (cat and dog) or (bird and fish) | Use ( ) to create complex searches, (cat and dog) or (bird and fish) |
| Special features | Use advanced option for boolean searching You control ranking by entering terms | Other searching options, see site | Search for "the person," "page title," "boolean" | Searches concepts, Using cat will find kitten | Special collection of articles from over 2000 periodicals |
| | Natural Language searching Language translator | | Set number of results or display options | Has a subject directory, Offers More like this | Puts items into custom search folders by topic, type, source and language |
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- Pipeline explosion kills at least 100 in Nigeria
- Death toll rises in Philippines garbage collapse
- Armstrong keeps Tour de France lead

more...

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- 9am ET Tour de France - stage 11
- 6 30pm Chat with rock icon Brian Wilson
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more...

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We are ir ted in your comments . , suggestions to make the EPA Inspector web site a better one. Please feel free to use the on-line comment box to make your voice heard. You may also contact Rafael Sanchez, Sanchez.rafael @epa.gov, for additional information.

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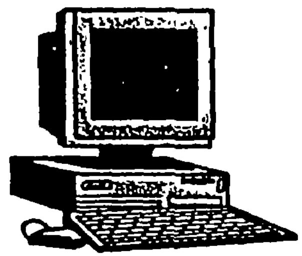


# New EPA Inspector Information Exchange Web Site

OECA

Office of Enforcement and Compliance Assurance

The EPA Inspector Web Site is a single comprehensive Intranet resource for EPA inspectors where relevant, up-to-date information about inspections, health & safety, policy & regulatory issues, technologies, compliance assistance and training can be quickly found. The inspector web site has been created as a pilot project by the Office of Compliance as way to foster and facilitate communication in the inspector community.

The pilot project will run for approximately nine months. During that time we will evaluate the web site for usage, content, number of files downloaded and in general, user involvement. The results of the evaluation process will help us identify new avenues and opportunities to better provide compliance assistance to EPA inspectors. Depending on the feedback we receive from the users we will determine whether to continue with this effort or try something else.

We are committed to provide you with the most up-to-date and relevant information about inspectors and inspection activities. With this in view, the Inspector web site has gathered a wealth of information to help you in your job as an EPA inspector. As you navigate throughout the web site, you will find a smorgasbord of inspector and inspection-related information and useful EPA and external links.

To foster timely communication, we have created a message board for you to post, respond and search for messages about inspectors and inspection-related issues. In this way, information about inspections can be shared with your colleagues in a timely and expeditious manner.

So far we have created the following discussion topic areas (Other discussion forum can be created as required):

✓ Matters of Urgency
✓ General Issues About Inspections
✓ Emerging Non-Compliance Trends
✓ Compliance Issues about Air
✓ Compliance Issues about Water
✓ Compliance Issues about RCRA
✓ Small programs & other Compliance Issues
✓ EPA ORDER 3500.1
✓ Policy and Regulatory Issues
✓ Health and Safety
✓ Training

Finally, to help you find fellow inspectors in a particular area of expertise we have created an interactive EPA Inspector Directory. The directory will allow you to pull information about EPA inspectors on the fly. The EPA directory is a user-friendly database where queries about inspector credentials and location can be made. The EPA directory can be updated by the user as often as required.

Interested? Then bookmark the following URL : intranet.epa.gov/oeca/oc/metd/inspector/

8

SESSION 8

## TOPIC: ENTRY, OPENING CONFERENCE AND SITE INSPECTION

**Time: 60 minutes**

**PURPOSE**

- Explain the extent and limits of EPA's authority to enter and inspect facilities
- Explain EPA policy and practice governing consensual entry
- Explain procedures for proper, lawful entry
- Provide guidance for handling sensitive situations regarding entry
- Discuss the role of the inspector in securing a warrant and conducting an inspection under a warrant.
- Discuss the issues and procedures to be covered during site entry.
- Discuss the issues to be covered during an opening conference.
- Discuss the issues to be covered during the site inspection
- Provide suggestions regarding inspection techniques.

**KEY POINTS**

- It is EPA policy to obtain a warrant when the owner has denied consent to entry.
- State, Local, and Tribal policy on entry and warrants may be different; discuss
- The opening conference is held to advise personnel of the facility of the objectives of the inspection and to discuss and arrange for logistics and scheduling of inspection activities
- The general site tour provides inspection team members an orientation and identifies and verifies activities that require further evaluation.

**LIST OF VISUALS**

8-1 Entry, Opening Conference and Site Inspection (Title Slide)
8-2 Entry, Opening Conference and Site Inspection—Session Overview
8-3 Entry, Opening Conference and Site Inspection (Summary of Federal Environmental Acts)
8-4 – 8-9 Entry, Opening Conference and Site Inspection—Entry
8-10 – 8-12 Entry, Opening Conference and Site Inspection—Entry
8-13 – 8-19 Entry, Opening Conference and Site Inspection—Opening Conference
8-20 – 8-21 Entry, Opening Conference and Site Inspection—Site Inspection

**LIST OF HANDOUTS**

8-1 Summary of Federal Environmental Acts

## Entry, Opening Conference, and Site Inspection

8-1

## Entry, Opening Conference, and Site Inspection—Session Overview

- Entry procedures
  - Summary of statutes
  - Legal bases or authority
  - Warrants
- General "How To" procedures for
  - Site entry
  - The opening conference
  - The site tour


8-2

## Entry, Opening Conference, and Site Inspection

### Summary of Federal Environmental Acts

| [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
|---|---|---|---|---|---|---|---|---|
| [illegible] | x | x | | x | x | | | |
| [illegible] | x | x | x | | x | | | |
| [illegible] | x | x | | x | x | | | |
| [illegible] | x | | | x | x | x | x | x |
| [illegible] | x | x | x | x | x | | | |
| [illegible] | x | x | x | | | x | | |
| [illegible] | x | | | x | x | x | x | x |

8-3

## Entry, Opening Conference and Site Inspection —Entry

- Legal basis for entry

## Entry, Opening Conference and Site Inspection —Entry

- Consensual entry

## Entry, Opening Conference and Site Inspection —Entry

- Consensual entry
  - Absence of express denial = consent

## Entry, Opening Conference and Site Inspection —Entry


- Denial of entry
  - EPA policy is to obtain a warrant

8-7

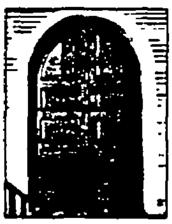
## Entry, Opening Conference and Site Inspection —Entry


- Warrantless entry
  - Emergencies
  - Heavily regulated industries
  - "Open fields" and "in plain view"

8-8

## Entry, Opening Conference and Site Inspection—Entry

- If denied entry
  - Be tactful
  - Use reason and logic
  - Record observations
  - DO NOT THREATEN
  - Contact supervisor



8-9

## Entry, Opening Conference and Site Inspection—Entry

- Warrants
  - When facility has denied access
  - If full consent is withdrawn during inspection
  - May be done in advance of inspection
  - Inspector and attorneys work as team

8-10

## Entry, Opening Conference and Site Inspection—Entry

- Warrants
  - Need to identify specific information needs
  - Limits inspection to specifics in warrant
- Other tools for gathering information

8-11

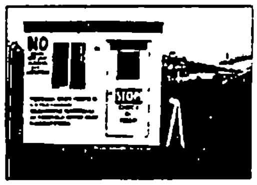
## Entry, Opening Conference and Site Inspection—Entry

- Proper site entry procedures
  - Drive-by and off-site observations
    - Look for obvious concerns
    - Familiarize yourself with layout
  - Normal working hours

8-12

## Entry, Opening Conference and Site Inspection—Entry

- Use main gate or entry
- Contact person in charge
  - Plant manager environmental manager owner etc
- Identify yourself with your credentials



8-13

## Entry, Opening Conference and Site Inspection—Opening Conference

- After initial site entry
- Introduction and credentials
- Explain
  - Authority
  - Purpose
  - Scope of inspection
  - Estimated time to complete

8-14

## Entry, Opening Conference and Site Inspection—Opening Conference

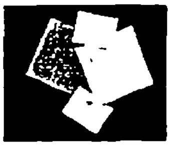
- Discuss Confidential Business Information (CBI)
  - Their right to claim CBI
    - Use good presentation technique
  - Provide paperwork
- Identify special safety concerns

8-15

## Entry, Opening Conference and Site Inspection—Opening Conference

Explain

- Documentation methods
  - Notes
  - Copies
  - Photographs
  - Samples, etc



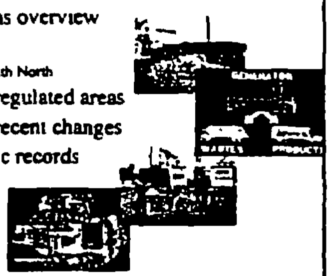
## Entry, Opening Conference and Site Inspection—Opening Conference

Explain:

- *Inspection process*
  - Facility processes
  - Records review
  - Site tour
  - Closing conference
    - Summary of findings

## Entry, Opening Conference and Site Inspection—Opening Conference

- Start of main information collection
  - General operations overview
  - Obtain map
    - Orient yourself with North
  - Identification of regulated areas
  - Identification of recent changes
  - Review of specific records



## Entry, Opening Conference and Site Inspection—Opening Conference

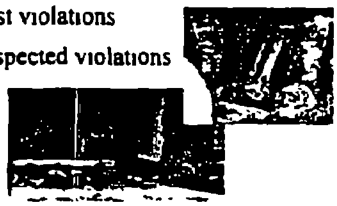
- Recommend process based approach
  - Raw materials in
  - Detailed understanding of process operations
  - Product out
  - Waste management units

8-19

## Entry, Opening Conference and Site Inspection—Opening Conference

Develop "Must See" list

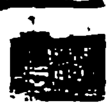
- Key process operations
- Waste management areas
- Areas with past violations
- Areas with suspected violations



8-20

## Entry, Opening Conference and Site Inspection—Site Inspection

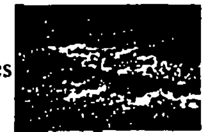
- Detailed "on-the-ground" inspection—Directed By You
- Follow process flow
- Evaluate compliance with media regulations
- Evaluate "Must See" items
- Watch for newly regulated units



8-21

## Entry, Opening Conference and Site Inspection—Site Inspection

- Review records and documents
- Identify follow-up activities
  - Interviews
  - Sampling locations
  - Additional inspection needs
- Document findings
  - Names, notes, photos, samples, etc



**SUMMARY OF FEDERAL E ONMENTAL ACTS REGARDING RIGHT OF ENTRY, INSPEC IONS, SAMPLING, TESTING, ETC.**

| Act/Section | Designated Representative | Presentation of Credentials | Notice of Inspection | Sampling Permitted | Inspection of Records | Sample Splits | Receipt for Agency's Samples | Return of Analytical Results |
|---|---|---|---|---|---|---|---|---|
| **Clean Water Act/308(a)** | Yes, authorized by Administrator | Required | Not required | Yes (effluents that the owner is required to sample) | Yes | Not required | Not required | Not required |
| **FIFRA/8(b) (books & records)** | Yes, designated by Administrator | Required | Written notice required, with reason and suspected violation noted | No | Yes | N/A | N/A | N/A |
| 9(a) (inspections of establishments) | Yes, designated by Administrator | Required | Written notice required, with reasons for inspection | Yes | See 8 | Required, if requested | Required | Required promptly |
| **Clean Air Act/114(a)** | Yes, authorized by Administrator | Required | Not required, except notification of the state for SIP sources | Yes | Yes | Not required | Not required | Not required |
| **RCRA/3007(a) 9005(a)** | Yes, designated by Administrator | Not required | Not required | Yes | Yes | Required, if requested | Required | Required promptly |
| **SDWA/1445(b)** | Yes, designated by Administrator | Required | Written notice required; also must notify state, with reasons for entry, if state has primary enforcement responsibility | Yes | Yes | Not Required | Not required | Not required |
| **TSCA/11(a,b)** | Yes, designated by Administrator | Required | Written notice required | (The act does not mention samples or sampling in this section, but it does state that an inspection shall extend to all things within the premises of conveyance) | Yes | N/A | N/A | N/A |
| **CERLCA/104** | Yes, designated by President | Not required | Not required | Yes | Yes | Required, if request | Required | Required promptly |

## SESSION 9

## TOPIC: EVIDENCE

**Time: 60 minutes**

**PURPOSE**

- Introduce the rules of evidence and their relevance to the activities of the inspector.
- Provide guidance on documenting evidence to help ensure its admissibility in a court proceeding.
- Present procedures for recording and documenting observations
- Present tips for improving techniques

**KEY POINTS**

- Many of the procedures being taught in this course are based on considerations related to evidence, they are designed to ensure the admissibility of information collected during an inspection and to enable the inspector to provide credible testimony in a court proceeding.
- Photographs are excellent evidence.

**LIST OF VISUALS**



**LIST OF HANDOUTS** None

# Evidence

## Evidence

**Session Overview**

- Identifying evidence
- Documenting evidence
- Ensuring admissibility

## Evidence Definition

"Any type of proof .. . ..legally presented at a trial .. .. for the purpose of inducing belief in the minds of the court or jury .. . ...."

## Types of Evidence

- Testimonial
- Real
- Documentary
- Demonstrative

## FRE 901: Authentication and Identification

"... a condition precedent to admissibility is satisfied by evidence sufficient to support a finding that the matter in question is what its proponent claims "

## FRE 401: Definition of "Relevant Evidence"

"... evidence having any tendency to make the existence of any fact that is of consequence to the determination of the action more probable or less probable than it would be without the evidence."

## FRE 602: Lack of Personal Knowledge

"A witness may not testify to a matter unless evidence is introduced sufficient to support a finding that the witness has personal knowledge of the matter. Evidence to prove personal knowledge may, but need not, consist of the witness's own testimony .."

## "Who Cares If..."

Who cares if what you are holding (in your hand in court) is contaminated "gunk" unless you can show that particular "gunk" came from the particular site involved in this case, not from somewhere else?

## Collection Conditions and Surroundings

- What activities were performed at the site?
- Why the sample was taken?
- How the item was discovered?
- What physical items were identified in the immediate vicinity?

Aids Notations in field logbook

Photographs of area and exact location of sample

## General Identity and Sameness

- Item has the same characteristics as the item collected by the inspector

Aids Routine documentation procedures
Sample tags with signature of the inspector and date
Notations in logbook about color, consistency, and other sensory perceptions

9-10

## Precautions To Ensure Identity

- Other precautions taken to ensure later identification

Aids Assignment of sample number
Initiation of chain-of-custody procedures

9-11

## Present Conditions That Vary

- Any features or conditions about the item being offered in evidence that vary from what was collected

Aids Full description in logbook

9-12

## Representativeness of Item

- Establishes the relationship to other items of the item offered as evidence

Aids Notations in logbook on sampling strategy used to select the particular sample plan

Sampling plan

8-17

## Connecting the Chain of Custody

- The meticulous process of showing the succession of persons who handled or had access to the evidence

Aids Chain-of-custody forms

Testimony regarding adherence to routine sampling security measures

8-18

## Inspector's Written Documentation

- Field Logbook
- Inspection Report

8-19

## FRE 612: Writing Used To Refresh Memory

"... if a witness uses a writing to refresh memory for the purpose of testifying, either --

(1) while testifying, or

(2) before testifying...

an adverse party is entitled to have the writing produced at the hearing, to inspect it, to cross-examine the witness thereon, and to introduce in evidence those portions which relate to the testimony of the witness..."

9-16

## FRE 803: Hearsay Exceptions 803(5) Recorded Recollection

"A memorandum or record concerning a matter about which a witness once had knowledge but now has insufficient recollection to enable the witness to testify fully and accurately, shown to have been made or adopted by the witness when the matter was fresh in the witness's memory and to reflect that knowledge correctly..."

9-17

## FRE 803: Hearsay Exceptions 803(6) Records of Regularly Conducted Activity

"A memorandum, report, record or data compilation, in any form, of acts, events, conditions, opinions, or diagnoses, made at or near the time by, or from information transmitted by, a person with knowledge, if kept in the course of regularly conducted business activity, and it was the regular practice of that business activity to make the memorandum, report, record, or data compilation..."

9-18

## Chain of Custody

Used to trace the possession of evidence from the time it was obtained, until the time it was introduced as evidence

9-19

## Evidence Is in "Custody" If...

- It is in the actual possession, control, and presence of the inspector
- It is in the inspector's view
- It is in a storage place to which only the inspector has access
- It is in a storage place to which only the inspector and identified others have access

9-20

## Basic Chain-of-Custody Procedures

- Establish custody
- Prepare documentation of the evidence
- Ensure custody during transit
- Note if the seal is found broken

9-21

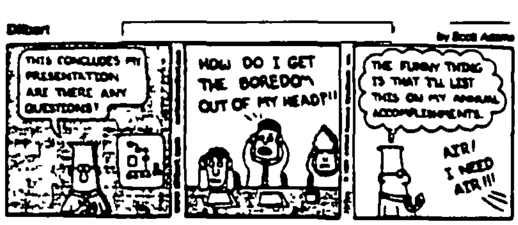
## FRE 406: Habit; Routine Practice

"Evidence of the habit of a person or of the routine practice of an organization, whether corroborated or not and regardless of the presence of eyewitnesses, is relevant to prove that the conduct of the person or organization on a particular occasion was in conformity with the habit or routine practice."

## Photographs/Video

- Keep log in notebook
- Don't take photos of sampling activities
- Film vs. Digital??
- Video recordings

## That's all



*QUESTIONS??*

## SESSION 10

## TOPIC: INTERVIEWING

**Time: 1 hour 30 minutes**

**PURPOSE**

- Present basics of planning, conducting, and documenting interviews.
- Demonstrate interview techniques through role-play, which demonstrates things not to do.
- Improve trainees' interviewing skills through discussion of techniques and practice.

**KEY POINTS**

- Interviewing is an important but often overlooked technique in an inspection.
- Good interview techniques enhance verbal and interpersonal relations skills.
- An awareness of how verbal indicators and active listening are key ingredients in effective interviewing.

**LIST OF VISUALS**

Interviewing

10-1

The Main Purpose of Interviewing

Information

10-2

Basic Interviewing Questions

- Who?
- What?
- When?
- Where?
- Why?
- How?

10-3

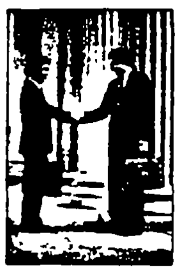
## Four General Modes of Communication

- Verbal
- Non-verbal
- Written
- Symbolic

10-4

## Five Components for More Effective Interpersonal Communications

- Self-concept (most important)
- Listening skills
- Clarity of expression
- Coping with angry feelings
- Self-disclosure



10-5

## Communication Barriers

A communication barrier is something we do or say that:

- Makes the other person less willing to communicate
- Causes the other person to become defensive
- Unnecessarily irritates the other person

10-6

## Communication Barriers (continued)

- Making premature comments and evaluations
- Making statements that are too general or excessively firm
- Interrupting others
- Talking too much
- Repeatedly telling others what to do

## Communication Barriers (continued)

- Talking down to people
- Asking loaded questions
- Administering punishment through sarcasm
- Placing emphasis on blame
- Arguing
- Displaying irritating listening habits

"It's what you learn after you know it all that counts."

John Wooden

## Time and Place

- First consideration —
  - Announced or unannounced?
- Second consideration —
  - Whose territory?
  - Ours? Theirs? Neutral?

10-10

## Time and Place (continued)

- General rules
  - Victims first, witnesses second, and suspects last
  - Separate witnesses
  - Friendly witness
  - Hostile or reluctant witness
  - Suspects
  - Attorneys present

10-11

## Physical Environment

### General Rules

- Eliminate as much noise as possible (physical, psychological, and semantic)
- Eliminate communication barriers (desks, tables, crossed arms/legs, personal items, etc.)
- Use proxemics to your advantage
- Remember—Set the stage

10-12

## Proxemics

| Zones | |
|---|---|
| Intimate | 0 to 1.5 feet |
| Personal | 1.5 to 4 feet |
| Social | 4 to 12 feet |
| Public | 12 to 25 feet |

10-13

## "Preparing" for the Interview

- What is the objective/purpose?
- What do I know?
- What do I want to know?
- What are the elements of the crime/situation?
- What is the background of the interviewee?
- Determine the potential for Miranda
- Select the lead/primary interviewer, if it is possible to have two interviewers
- Differences between one- and two-person interviews

10-14

## Five Steps of a Law Enforcement Interview

- Identification:
  - ID—yourself
  - ID—interviewee
  - ID—purpose
- Rapport
- Questions:
  - General
  - Specific
- Summary
- Close

10-15

## Types of Questions To Use

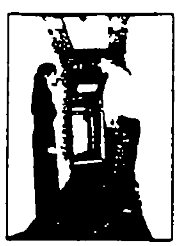
- General or open-ended
- Specific or direct
- Backward reaching
- Empathetic/sympathetic
- Opinion

?

10-16

## Avoid

- Leading questions
- Negative questions
- Compound questions
- Complex questions



10-17

## Listening

- People give meaning to words
- Feelings more important than mere words
- "Active" listening includes:
  - Hearing/noticing both verbal and non-verbal message, tone, and inflection
- The "listener" usually maintains the most eye contact

10-18

## How To Be an "Active Listener"

- Remain neutral
- Give your complete attention
- Ask about their statements
- Restate their main points
- Put their feelings into words
- Get agreement

10-19

## Non-Verbal Communication

| | |
|---|---|
| Kinesics | Body Movement |
| Oculesics/Pupillometrics | Eye Movement |
| Proxemics | Distance/Space |
| Haptics | Touch |

10-20

## Evaluating Non-Verbal Communication

- Factors to be considered
  - Culture
  - Content
  - Change from the norm
  - Clusters of behavior

10-21

## View Any Behavior in Context with the Individual's

- Social class
- Religion
- Culture
- Locale

10-22

## Best Young Achiever Quote

Elizabeth Brinton, 13-year old Girl Scout, explaining how she sold 11,200 boxes of cookies:

**"You have to look people in the eye and make them feel guilty."**

Recognize that guilt is a tool to be used. Most people want to do well

10-23

11

**SESSION 11**

**TOPIC: ON SITE RECORDS REVIEW**

**Time: 60 minutes**

| | | |
|---|---|---|
| **PURPOSE** | • Present key elements of reviewing records. | |
| **KEY POINTS** | • Ability to follow the paper trail is a key skill of the inspector. | |
| **LIST OF VISUALS** | 11-1 | On Site Records Review (Title Slide) |
| | 11-2 | Records Inspection |
| | 11-3 and 11-4 | Problem A |
| | 11-5 and 11-6 | Problem B |
| | 11-7 | Problem C |
| | 11-8 | Problem D |
| | 11-9 and 11-10 | Overview of Steps in Sampling Records |
| | 11-11 | Sampling Techniques |
| **LIST OF HANDOUTS** | 11-1 | Sample Manifests |
| | 11-2 | Problems in Sampling Strategies |

## On Site Records Review

II 1

## Records Inspection

- Conduct of records reviews
- Records sampling techniques

II 2

## Problem A

A company has not submitted any reports of exceeding the discharge limits imposed in its permit Your inspection of the facility indicates poor operation and maintenance of pollution control equipment, leading you to suspect that the equipment may have failed You want to determine whether there were any excursions that should have been reported to EPA.

II 3

## Problem A

- What records would you look at?
- What would you be looking for?
- What evidence would you attempt to collect?

11-4

## Problem B

EPA has a tip that a company has begun manufacturing and distributing a new chemical (di-benzo-horrible) before completing the remanufacture review process required under TSCA or FIFRA

11 5

## Problem B

- What kinds of records and documents would you look for?
- What other evidence might there be?
- How would you approach an inspection to determine whether a company was making a new product if EPA had no tip?

11-4

## Problem C

A company's records of storage of hazardous waste are sloppy and incomplete You easily can document the violation of recordkeeping requirements, but you suspect the company is storing waste for much longer than the period allowed

11-7

## Problem D

A citizen has notified EPA that a dredging company allegedly has disposed of dredged material into wetlands owned by the town of Clarksville

- What records might help to confirm this allegation?
- What evidence would you attempt to collect?

11-8

## Overview of Steps in Records Sampling

- Determine the objectives of the records review
- Identify the total population
- Select the sampling method

11-9

## Overview of Steps in Records Sampling

- Determine the sample size
- Conduct the sampling
- Document the sampling methodology

11 10

## Sampling Techniques

- Random sampling
- Systematic or interval sampling
- Stratified sampling
- Block sampling
- Judgmental sampling

11 11

Sample Manifest 3

TEXAS WATER COMMISSION
P.O. Box 13087, Capitol Station
Austin, Texas 78711-3087

Please print or type. (Form designed for use on elite (12-pitch) typewriter.)

Form approved. OMB No. 2050-0039, expires 09-30-91

**UNIFORM HAZARDOUS WASTE MANIFEST**

1. Generator's US EPA ID No.: C01680090071
Manifest Document No.:
2. Page 1 of 1
Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address:
U.S. E.P.A., N.E.I.C.
Building 53, Box 25227, Denver Federal Center
Denver, CO 80225

4. Generator's Phone (303) 236-5111

A. State Manifest Document Number: Nº LA 6254304
B. State Generator's ID
C. State Transporter's ID
D. Transporter's Phone
E. State Transporter's ID
F. Transporter's Phone
G. State Facility's ID
H. Facility's Phone

5. Transporter 1 Company Name: Rolling Chemrok, Inc.
6. US EPA ID Number: DED982565947

7. Transporter 2 Company Name: Matlock, Inc.
8. US EPA ID Number: DED981110166

9. Designated Facility Name and Site Address:
Rollins Environmental Service (LA), Inc.
13351 Scenic Highway
Baton Rouge, LA 70807
10. US EPA ID Number: LQD010395127

| 11A HM | 11. US DOT Description (including Proper Shipping Name, Hazard Class, and ID Number) | 12. Containers No. | Type | 13. Total Quantity | 14. Unit Wt/Vol | I. Waste No. |
|---|---|---|---|---|---|---|
| x | a. Waste Flammable Liquids, n.o.s., 8, UN1993, PG II | 002 | DE | 30 | P | F0006 |
| x | b. Waste Corrosive Liquids, n.o.s., 3, UN1760, PG II | 002 | DE | 110 | P | F0006 |
| x | c. Hazardous Waste, Liquid, n.o.s., 9, NA3082, PG II | 002 | DF | 110 | P | F0006 |
| x | d. Hazardous Waste, Solid, n.o.s., 9, NA3077, PG II | 001 | DF | 68 | P | F0006 |

J. Additional Descriptions for Materials Listed Above
K. Handling Codes for Wastes Listed Above

15. Special Handling Instructions and Additional Information
In case of emergency, contact CHEMTREC at (800)424-9300 and mention "Labpack". If undeliverable return to generator. If spilled in Louisiana contact the Department of Public Safety. Avoid ingestion, inhalation, and skin contact.

16. GENERATOR'S CERTIFICATION: I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations, including applicable state regulations.
If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment, storage or disposal currently available to me which minimizes the present and future threat to human health and the environment; OR, if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

| Printed/Typed Name | Signature | Month | Day | Year |
|---|---|---|---|---|
| Joe Badluck | Joe Badluck | 02 | 27 | 87 |

17. Transporter 1 Acknowledgement of Receipt of Materials

| Printed/Typed Name | Signature | Month | Day | Year |
|---|---|---|---|---|
| Sam Goodluck | Sam Goodluck | 02 | 27 | 87 |

18. Transporter 2 Acknowledgement of Receipt of Materials

| Printed/Typed Name | Signature | Month | Day | Year |
|---|---|---|---|---|
| | | | | |

19. Discrepancy Indication Space

20. Facility Owner or Operator Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.

| Printed/Typed Name | Signature | Month | Day | Year |
|---|---|---|---|---|
| Sam Noluck | Sam Noluck | 03 | 12 | 87 |

TWC-0311 (Rev 01/01/89)    White - original   Pink-TSD Facility   Yellow-Transporter   Green-Generator's first copy

## PROBLEMS IN SAMPLING STRATEGIES

1. Regulations require that all employees of Dumpstump, Inc., complete a training course -- within two months of being hired -- on how to handle hazardous material safely. Your task is to determine whether the company has complied with these regulations. EPA records indicate that the company employs 525 people. You plan to review records and then conduct follow-up interviews with several employees to verify those records. You have approximately one hour to complete the records review portion of this task.

   Dumpstump officials tell you that each employee's personnel file includes a record that shows that the required training has been completed. Company officials show you a file cabinet that they say contains all of the personnel files, organized in alphabetical order by name of the employee. There are 389 files.

   What factors would be important to support a reasonable conclusion about compliance at the firm? What are the potential biases in the files? How might the discrepancies between the number of personnel files and EPA's records of the number of employees be explained? Is there a particular category(ies) of employee that you want to focus on? Is there any category of employee that you want to exclude? Which sampling method or methods could you use to select specific records for review? How would you select the individuals to be interviewed?

2. The Spewforth Company is required to submit a written report to EPA if it has had a minor discharge exceeding the limit established in its permit; the reports must be submitted within five working days of the end of the month. Reports of major discharges must be filed within five days of their occurrence. Spewforth's permit requires the company to take and analyze samples of discharge from three locations twice a week and to maintain records of the sampling and results of analysis for review by EPA upon request.

   For the past 24 months, EPA has received only one monthly report about a minor exceedance; no major discharge has been reported. The last inspection of the facility took place more than two years ago. Although EPA has no direct knowledge that discharge exceedances have occurred, neighbors have complained that orange-colored, smelly liquid comes out of the pipe occasionally -- but no dates have been reported.

   The company keeps logs of its sampling activities and analytic results. Your period of interest is the previous two years. Since three samples are (supposed to be) taken each week, the logs are far too extensive to allow for review of all entries. How would you go about selecting log entries for review?

12

SESSION 12

TOPIC: SAMPLING AND LABORATORY ISSUES

**Time: 90 minutes**

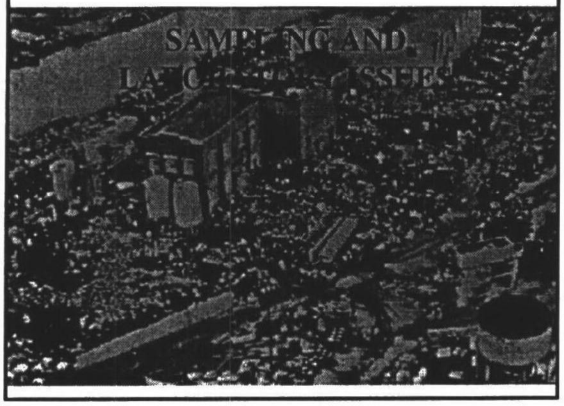
**PURPOSE**

- Explain the importance of physical samples as evidence
- Present considerations involved in ensuring that samples meet quality requirements
- Provide an overview of the sampling process from planning through disposal of contaminated equipment
- Provide a basic understanding of the functions of a laboratory and the relationship between the laboratory and the inspector

Note. **This session is not intended to teach inspectors how to sample but to provide a basic understanding regarding sampling issues**

**KEY POINTS**

- Physical samples are collected to establish the presence and concentration of regulated substances and the extent of contamination.
- Sampling data help determine whether a violation exists (for example, exceeding a standard) and, if so, the gravity of that violation (for use in determining penalties).
- Before the inspection is conducted, sampling objectives and data quality requirements and the methods by which they will be met must be established in a quality assurance project plan per EPA Order 5360.1, change 1 (July 1998)
- Standard collection should be followed or deviations noted in field notes/log book
- Chain-of-custody procedures MUST be followed.
- Inspectors should be aware of capabilities and limitations of laboratory.
- Laboratory personnel can be a valuable source of technical expertise.
- Advance planning for laboratory work helps ensure prompt analysis.



## Physical Sampling

- Policy considerations in sampling
- Technical considerations in sampling
- Common sampling errors
- Quality assurance and quality control
- Documentation of samples and chain of custody
- Management of samples in the field
- Laboratory Issues

## General Guidelines for Sampling

- Take a sample when one is needed to prove a violation
- Sample only when there is reason to suspect the substance is present
- Always attempt to verify the presence of the substance by a means in addition to sampling

## When To Sample

- No data
- Insufficient data
- Data in doubt
- Data for event
- Required

**SAMPLE COLLECTION, TRANSPORTATION, ANALYSIS IS NOT CHEAP!!!**

## Quality Assurance/Quality Control EPA Order 5360.1 chg 1 Requirements

- Scope
  - Order defines minimum requirements for quality systems supporting EPA environmental programs that encompass:
    - collection, and
    - use of environmental data by or for EPA

### Quality Assurance/Quality Control
## EPA Order 5360.1 chg 1 Requirements

- Applicability to Environmental Programs
  - Order applies to following environmental programs
    - **direct measurement of environmental conditions or releases including.**
      - **sample collection**
      - **analysis**
      - **evaluation, and**
      - **reporting of environmental data**

### Quality Assurance/Quality Control
## EPA Order 5360.1 chg 1 Requirements

- Applicability to Environmental Programs
  - Order applies to following environmental programs
    - **the use of environmental data collected for other purposes or from other sources (secondary data) including:**
      - **industry surveys**
      - **compilations from computerized data bases**

## Quality Control

- Quality control consists of a set of TECHNICAL activities that must be performed to DOCUMENT whether the desired DATA QUALITY is being produced

## Quality Assurance

- Quality assurance is a system of activities at the:
- Planning
- Implementation
- Review
- stages of a data gathering project to ensure that data collected for EPA are of the quality needed and claimed

## Quality Assurance Project Plan (QAPP)

- Must be prepared for every sampling inspection
- Two types
  - Routine
  - Nonroutine

## Quality Assurance Project Plan (QAPP)

- Verifies what and how you are conducting sampling
- ensures that data collected for EPA are of the quality needed and claimed (DATA QUALITY GOALS)

## Quality Assurance and Quality Control Samples

- Replicate
- Split
- Spiked
- Preservative blanks

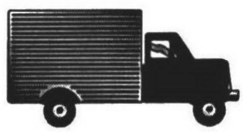
## Changes in the QAPP

- Can the original objectives still be met?
- Can the sampling still be done with the existing equipment?
- Is it safe to sample?

Can the evidence *you* collect stand up in a court of law?

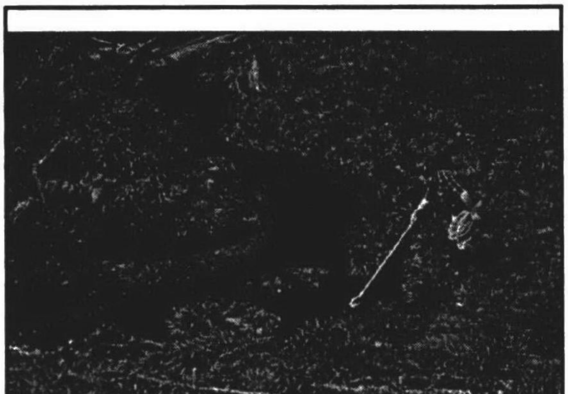
## Transportation to Site

- Driving is Easier than Flying
- Some Things just Won't Fly
- Coordinate Time of Arrival



## Representative Samples

Inspector must insure all samples are representative-taken and analyzed using appropriate SOP (QAPP)

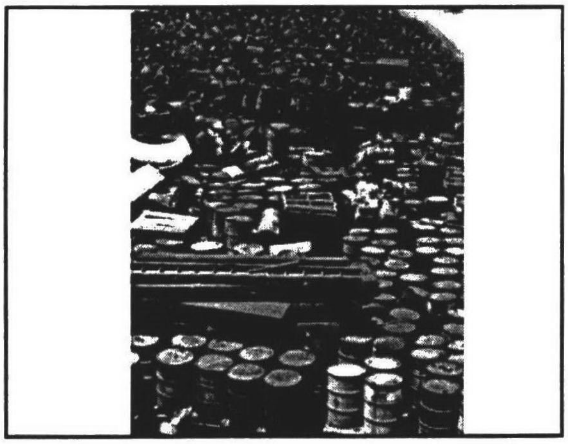


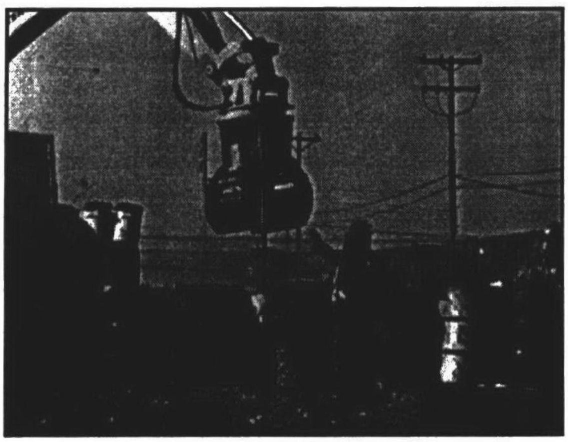
## Representative Sampling Factors

- Operating conditions
- Types of waste
- Statistical considerations
- Temporal considerations
- Spatial considerations

## Evaluating Drum Populations

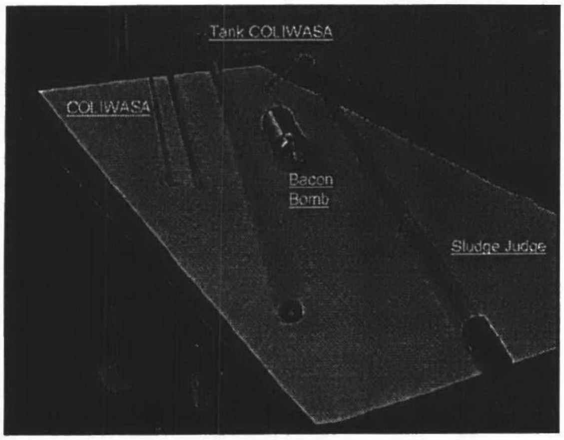
- **Monitor Ambient Air**
- **Check Accessibility**
- **Observe Labels and Markings**
- **Open Drums to Examine Contents and Perform Field Tests**

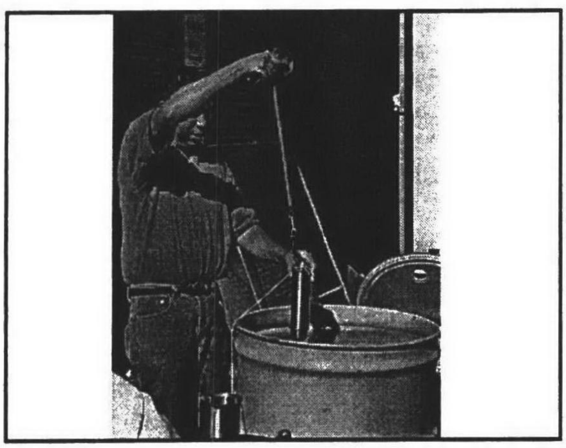


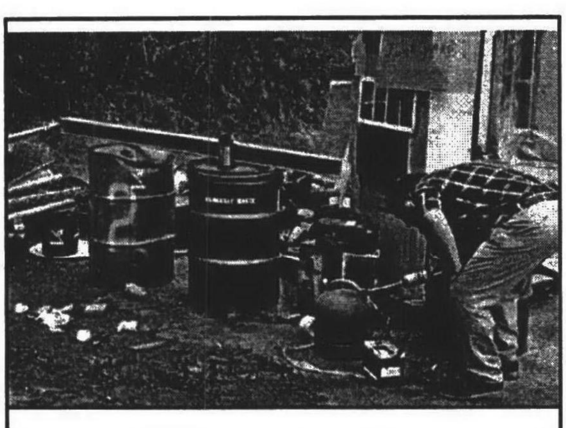


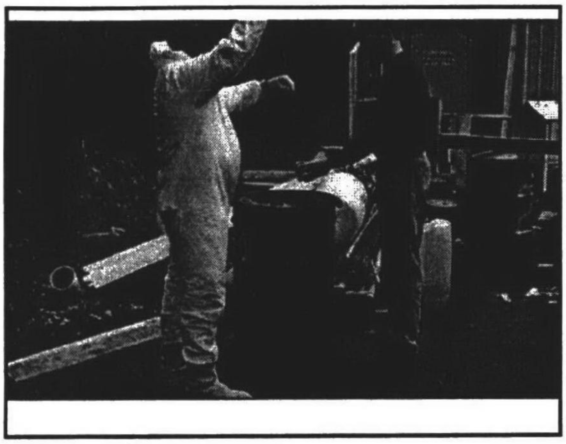
## Drum Sampling Techniques

- COLIWASA [*NO Glass thief*] for liquids
- Split Spoon Coring of Solids
- Pitard Method
- Examples


Tank COLIWASA
COLIWASA
Bacon
Bomb
Sludge Judge

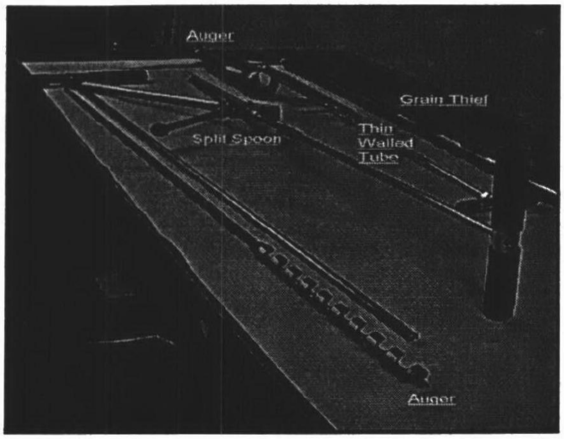






## Soil Sampling

- Review historical photos, if available
- Visually inspect site for the obvious
- Use field measurements to locate contamination
- Select a suitable sampling method




## Tools for Evaluating Wastewater Discharges

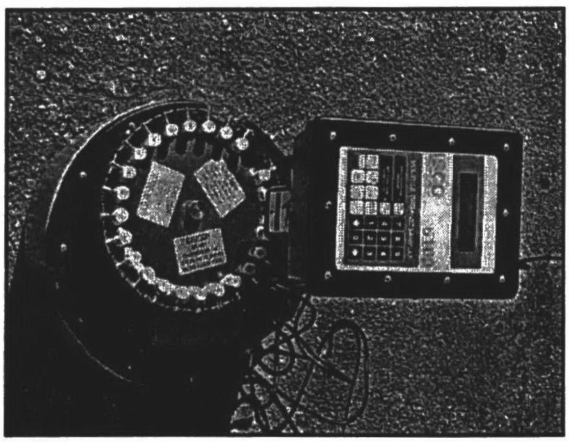
- Grab Samples
- Automatic Sampling
- Continuously Recording pH meter
- Flow Meters

## When are Grab Samples Appropriate?

- Samples of Opportunity
- Intermittent Flows
- Facility Batch Treats Wastewater
- Instantaneous Readings are Necessary

## Advantages of Using Automatic Wastewater Samplers

- May be placed hours in advance
- Monitor discharge over time
- Triggered by flow, pH, conductivity, or time



## Continuously Recording pH Meters

- Provide a record over time of instantaneous readings
- *Good method for detecting pattern of discharge*
- May be stand alone device or module of an automatic sampler

## Flow Meters

- Some permit discharge limits are flow dependent
- Another good method for detecting patterns of behavior
- Can be used to trigger automatic sampler

## Safety Plans

- Identify Team Members
- List Emergency Information
- Discuss Anticipated Hazards
- Detail Workplan and Related Safety Measures
- Describe Monitoring Plan
- Decontamination and Disposal Plans

## What is a Confined Space?

- Size and Shape Must Permit a Person to Enter
- Restricted Entry and Exit
- Not Designed for Continuous Occupancy
- Hazardous Atmosphere of Poor Ventilation
- Any Other Safety Hazard

## What are Hazardous Atmospheres?

- Oxygen Level Below 19.5% or Above 23.5%
- Flammable Gas or Vapor Level Above 10% of LEL
- Toxic Substance Level Above TLV
- Combustible Dust Level Obscuring Vision at 5 Feet or Less
- Any Atmosphere IDLH

## Atmospheric Monitoring Equipment

- Oxygen Meter
- Combustible Gas Meter
- Hydrogen Sulfide Meter
- Carbon Monoxide Meter
- Hydrogen Cyanide Meter
- Radiation Meter
- Volatile Organic Vapor Detector

## Confined Space Entry Permit

- Identifies the Site
- Describes Confined Space to be Entered
- Lists Entrants and Back-ups
- Outlines Pre-entry Preparation
- Describes Rescue Plans

## Common Sampling Errors

- Calibration

(continued)

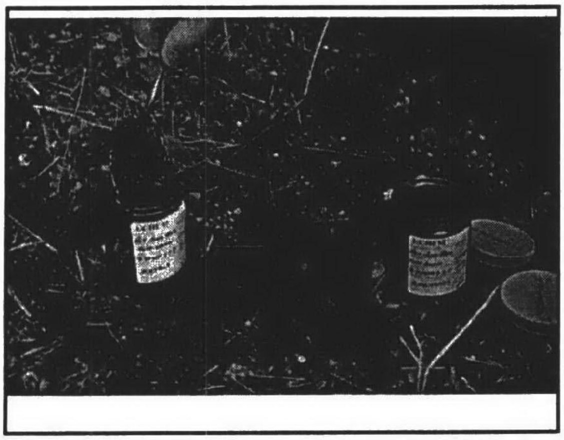
## Common Sampling Errors

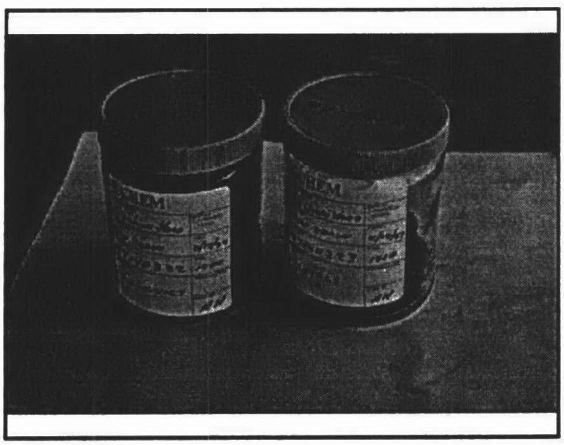
- Calibration
- Maintenance
- Forgotten equipment
- Misreading
- Miscalculations

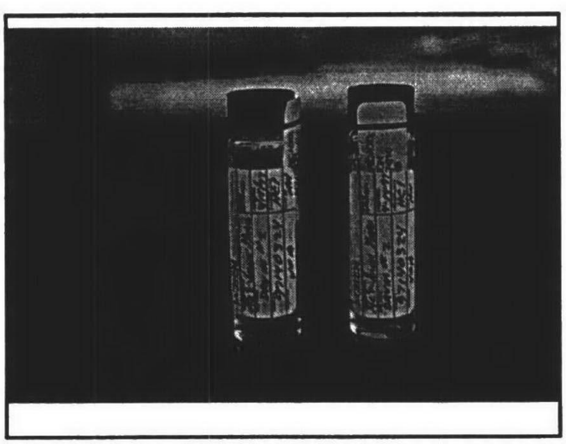
(continued)

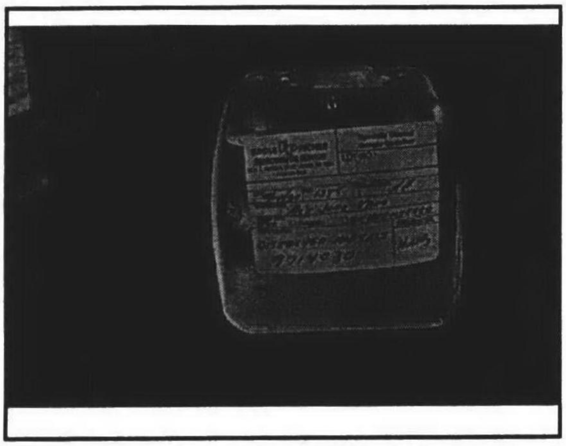
## Common Sampling Errors

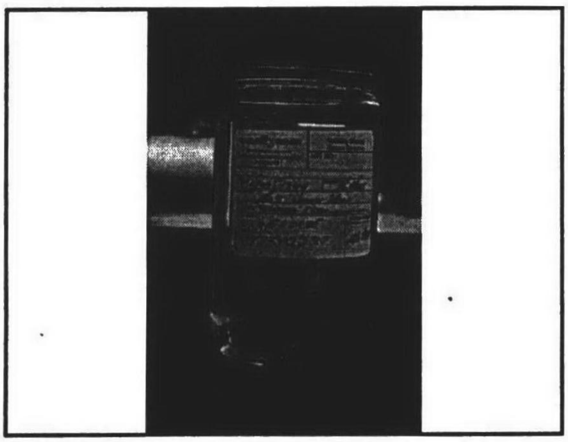
- Mislabeling
- Transposing of data
- Poor field notes
- Loss of samples

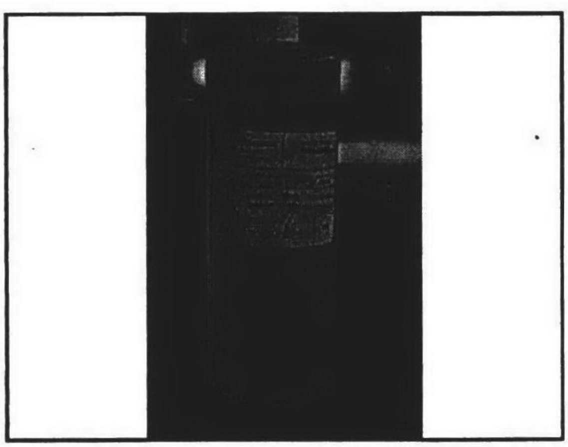






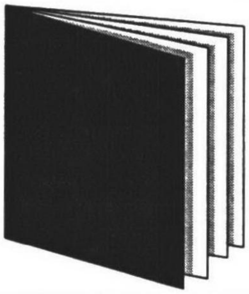


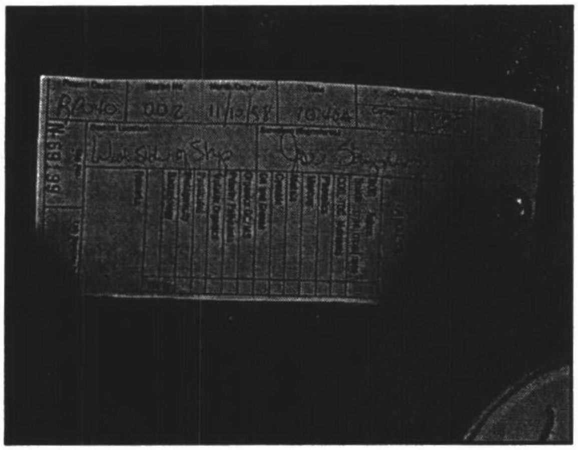


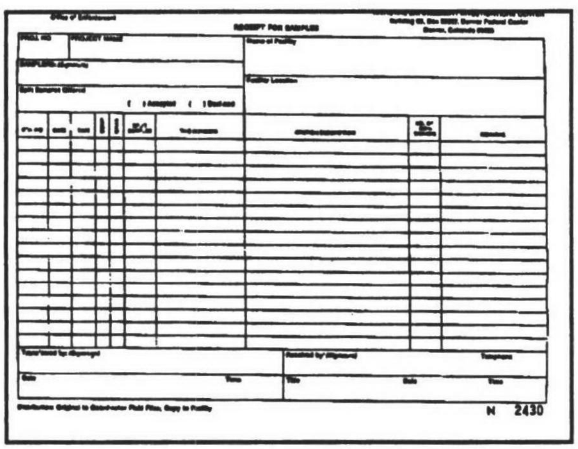


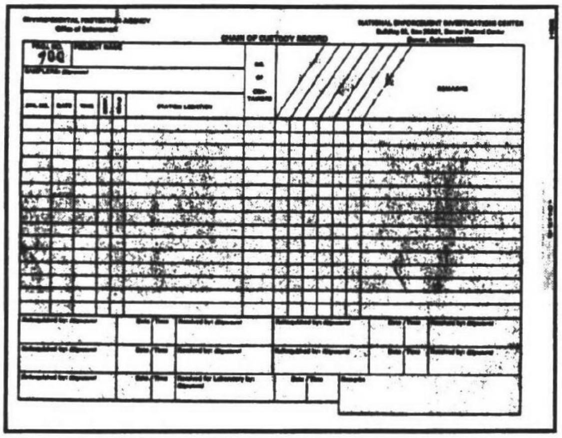
## Field Documentation

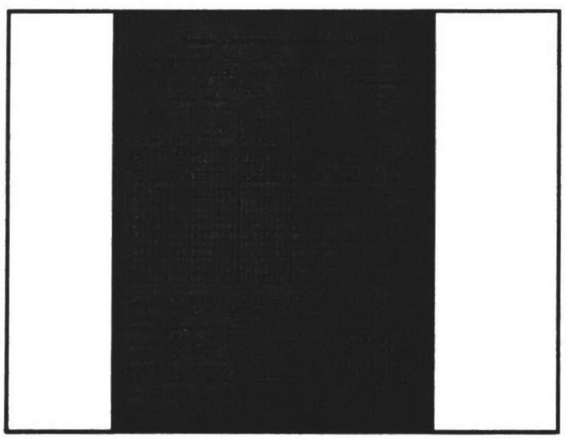
- •Sample Documentation
- •Field Notebooks
- •Photographs
- •Videotapes
- •Chain of Custody











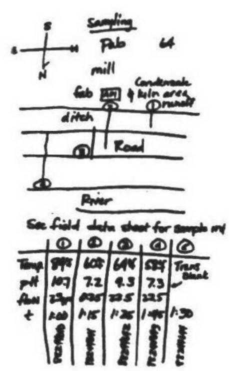
## Field Notes

- Record in a bound notebook
- Sign and date notes
- Be sure there is only one set of notes
- Remember notes may be discoverable by defense
- Don't give facility a copy of notes

## Appropriate Field Notes

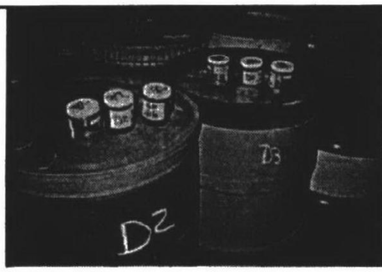
- General observations like time of entry and weather conditions
- Field instrument identification and calibration data
- Inventories of containers on site
- Detailed sample descriptions

## Field Logbook Entry: Sampling

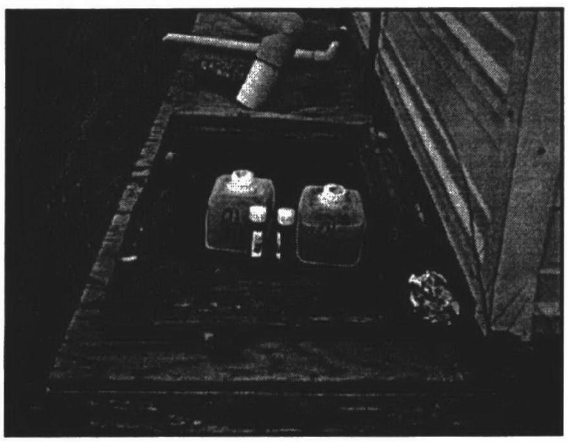


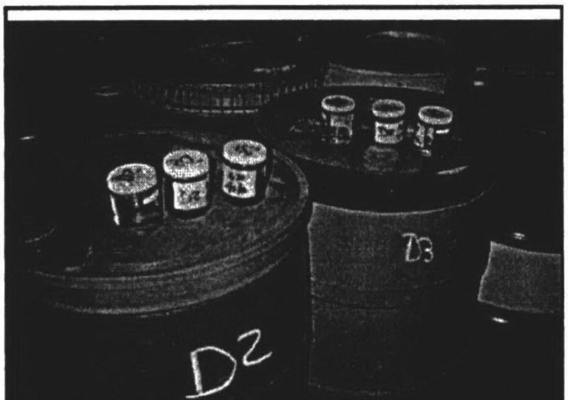

## Photo Log Reminders

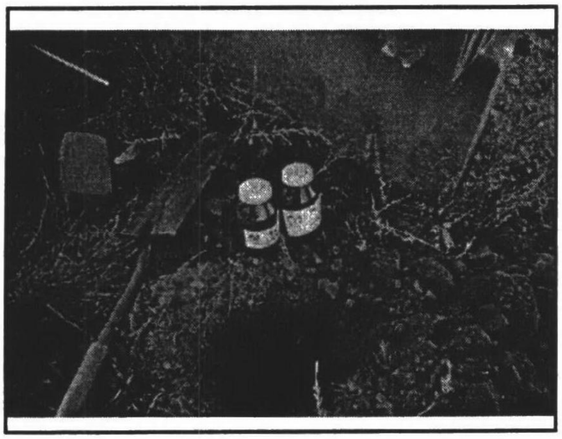
- Roll and Frame Numbers
- Subject
- Site Name
- Date
- Photographer

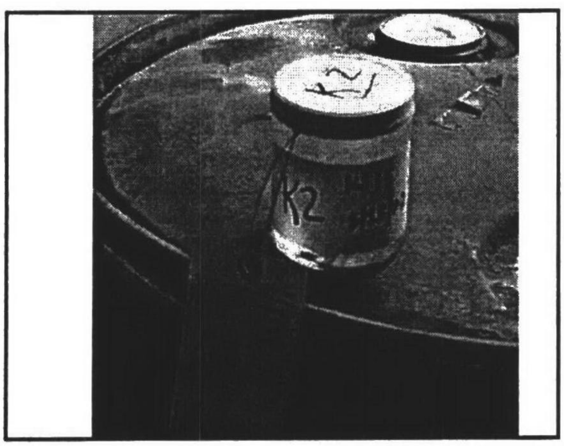


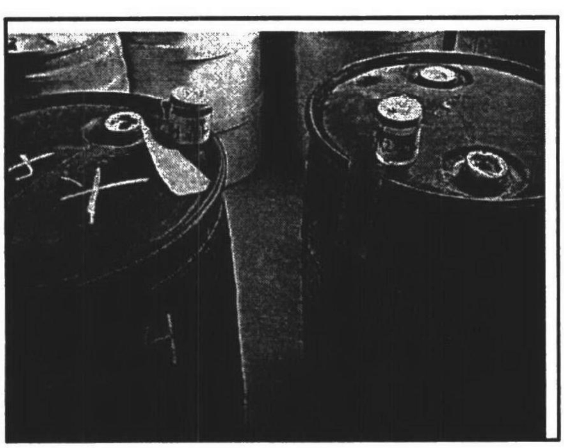

**Project:** 80G-08
**Site:** Thoro Products, Inc.
**Location:** Arvada, Colorado
**Date:** November 13, 1996
**Subject:** Samples D2 and D3
**Photographer:** D. Stephenson

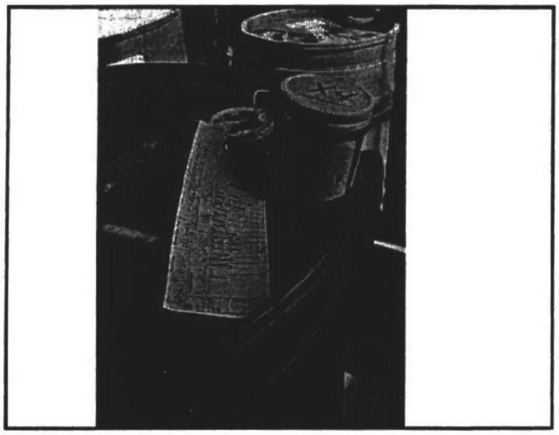







K2
K2





## Chain-of-Custody Procedures

- Establishing custody
- Preparing documentation
- Coordinating the sample and documentation
- Ensuring continuity of custody during transit

## A Sample is in Custody if

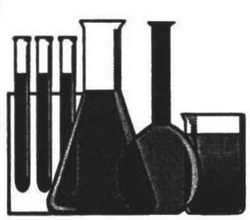
- It is in your possession
- It is in your view after being in your possession
- It was in your possession and you locked it up
- It is in a designated secure area

## Sample Holding Time

Prompt analysis is the most positive assurance against error from sample deterioration

## Preservation and Holding Times

- If preservatives are used, record in field notes
- Ice down samples before transporting to lab
- Be aware of holding times



## Packing and Shipping Samples

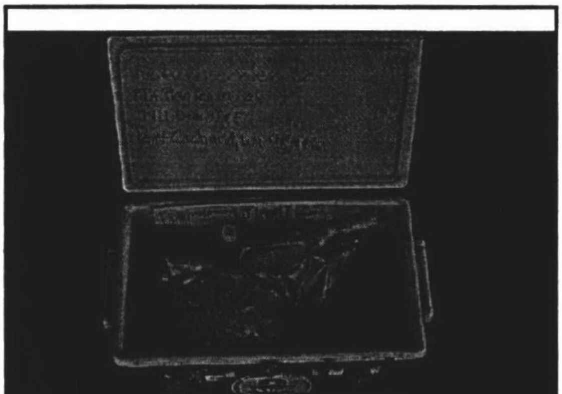
49 CFR 172.702 states in part:

Everyone "who performs any function subject to the requirements of this subchapter may not perform that function unless trained in accordance with the requirements of this subpart ..."

Each person who packs hazardous materials or offers such materials for shipment must be trained and recertified every two years

## Shipping Hazardous Samples

- Special Training
- Shipping Containers
- Labels
- Air Bill Preparation
- Common Problems

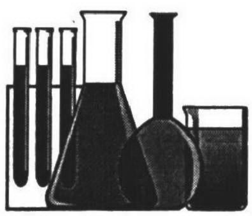


## Laboratory Analysis

- Laboratory operations
- Laboratory capabilities
- Interpretation of analytical results

## Special Analytical Considerations

- **Short Holding Times**
- **Chemist Available to Receive Samples**



## Sample Handling

- **Maintaining physical custody**
- **Chain of custody documentation**
- **Sample storage**

## Storing Samples

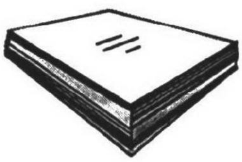
- Store samples with low levels of contamination with like samples
- Store samples with high levels of contamination with like samples
- Never store incompatibles together
- Store samples in secure area free from exposure to conditions which could alter them in any way

## Relinquishing Samples to Lab

- Coordinate time of delivery
- Check chain of custody with chemist
- Discuss field measurements
- Preservatives used

## Report of Findings

- Describe field activities
- Report analytical findings
- Site maps and sample location
- Include photos



## Litigation Support

- Exhibit Preparation
- Grand Jury Testimony
- Depositions
- Trial Support
- Trial Testimony

PROJECT NAME: ______________
PROJECT NUMBER: ____________
PROJECT COORDINATOR: ____________

ACCOUNTABLE DOCUMENTS

| | | | |
|---|---|---|---|
| RECEIPT FOR SAMPLE FORMS (#________) | | CHAIN OF CUSTODY FORMS (#________) | |
| SAMPLE TAGS (#________) | | LOG BOOKS (type & #___) | |
| CUSTODY TAPE (#_______) | | CUSTODY LOCKS (#______) | |
| TSCA DOCUMENTS (#_______) | | TSCA LOCKS (#________) | |
| | | | |

SAFETY & FIRST AID EQUIPMENT

| | | | |
|---|---|---|---|
| HARD HAT & FACESHIELD | | RESPIRATORY PROTECTION | |
| SAFETY GLASSES & INSERTS | | RESPIRATORY CARTRIDGES | |
| GOGGLES | | TYPE ________ NUMBER | |
| EAR PROTECTION | | M S A ________ | |
| RADIOS & CHARGERS | | RACAL ________ | |
| FIRST AID KIT | | SURVIAIR ________ | |
| PORTABLE EYE WASH | | NORTH ________ | |
| FIRE EXTINGUISHER | | COOLING VEST | |
| S C B A | | SPARE TANKS | |
| 5 MINUTE ESCAPE MASK | | CASCADE SYSTEM | |
| SAFETY HARNESS | | LIFTING TRIPOD | |
| TRAUMA KIT | | OXYGEN | |

PERSONAL PROTECTIVE EQUIPMENT

| TYPE | SMALL | MEDIUM | LARGE | X-LG |
|---|---|---|---|---|
| SARNEX | | | | |
| COATED TYVEK | | | | |
| NON-COATED TYVEK | | | | |
| DISPOSABLE RAIN SUITS | | | | |
| RAIN SUITS | | | | |
| NOMEX HOODS | | | | |
| BUTYL APRONS | | | | |
| NUKE BOOTS | | | | |
| RUBBER BOOTS (steel toe) | | | | |
| GLOVES | | | | |
| NEOPRENE (corrosive) | | | | |
| NITRILE (solvents) | | | | |
| BUTYL RUBBER | | | | |
| VITON | | | | |
| POLYURETHANE | | | | |
| LATEX | | | | |
| LEATHER | | | | |
| COTTON | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

## INSTRUMENTS

| | | | |
|---|---|---|---|
| V A, CHARGER & CAL. GAS | | H N U, CHARGER & CAL. GAS | |
| MONITOX SD HCN (CYANIDE) | | PHOTOVAC-TIP & CHARGER | |
| P H METER & RECORDER | | DRAGER PUMP & TUBES | |
| CONDUCTIVITY METER | | POCKET DOSIMETER | |
| RAD MINI | | BRUNTON COMPASS | |
| METAL DETECTOR | | GEIGER COUNTER | |
| E M-34 | | WELL DEPTH SOUNDER | |
| GAS TECH MODEL-86 | | GAS TECH MODEL-91 | |
| CHLORINE TESTER | | PERSONAL SAMPLING PUMP | |
| HEAT STRESS MONITOR | | SOUND LEVEL METER | |

## DECON EQUIPMENT

| | | | |
|---|---|---|---|
| WASH TUBS | | BUCKETS | |
| SCRUB BRUSHES | | DETERGENTS (type________) | |
| PLASTIC SHEETING | | SOLVENT (type__________) | |
| TRASH BAGS | | PAPER TOWELS | |
| MASK SANITIZER | | DISTILLED WATER | |
| STEAM CLEANER | | | |
| | | | |

SAMPLING EQUIPMENT

| | | | |
|---|---|---|---|
| DRUM THIEVES | | COLIWASA | |
| BAILERS | | GLASS FUNNELS | |
| GRADUATED CYLINDER | | PLASTIC SCOOPS | |
| STAINLESS SCOOPS | | STAINLESS PITCHERS | |
| STAINLESS BUCKETS | | STAINLESS PANS | |
| STAINLESS SPATULAS | | PLASTIC SPATULAS | |
| SPLIT SPOON SAMPLER | | PORTABLE SAMPLER | |
| POND SAMPLER | | MANNING SAMPLER | |
| ISCO SAMPLER | | CUBITAINER (DOSED)_____ | |
| BACON BOMBS | | SLUDGE JUDGE | |
| MANHOLE OPENER | | CAKE PANS | |
| DRUM OPENING KIT | | V O A,S | |
| ENTRENCHING TOOLS | | QUART JARS (cases) | |
| PICK & SHOVEL | | GALLON JARS (cases) | |
| P H PAPER | | 8 OZ JARS (cases) | |
| FLOW METER (Isco) | | PLASTIC BOTTLES(4oz/8oz) | |
| FLOW METER (Manning) | | CUBITAINER (size)______ | |
| | | | |
| | | | |

MISCELLANEOUS

| | | | |
|---|---|---|---|
| PORTABLE COPIER/CARTRIDGES | | DRINKING WATER CONTAINER | |
| MEASURING TAPE (cloth) | | MEASURING TAPE (steel) | |
| ELECT. EXT. CORD (lgth_____) | | GENERATOR & GAS CAN | |
| REAMS OF PAPER | | CAMERA AND FILM (35mm) | |
| PORTABLE TOILET | | FLASHLIGHT & BATTERIES | |
| MEASURING WHEEL | | BARRICADE TAPE | |
| SURVEY FLAGS | | ROPE (type____/lgth____) | |
| PLUMB BOB | | BOLT CUTTERS | |
| RHODAMINE DYE | | ISOPROPANOL | |
| NITRIC ACID | | ACETONE | |
| HEXANE | | DISPOSABLE CUPS | |
| | | | |
| | | | |

SHIPPING EQUIPMENT

| | | | |
|---|---|---|---|
| ZIP LOCK BAGS LG____SM____ | | PLASTIC BAGS LG___SM___ | |
| PAINT CANS & CLIPS | | SHIPPING LABELS (type) | |
| BUBBLE WRAP | | KIM WIPES | |
| ICE CHEST | | VERMICULITE | |
| FED X FORMS | | INK PENS | |
| MARKER PENS | | TAPE (DUCT) | |
| | | TAPE (STRAPPING) | |

## NEIC SITE HEALTH AND SAFETY PLAN

*****************************************************************************************

### General Information

1. Project Title: Project Number:

2. Location:

3. Description of Field Activities:

4. Date of Field Activities:

5. NEIC Personnel:

| | Project Leader(s) |
|---|---|
| | |

6. Contractor Personnel: Must be OSHA certified per 29CFR 1910.120.

**Obtain list of personnel, duties or work to be performed, and copies of training certificates.**

*****************************************************************************************

### Emergency Information

7. Ambulance: Phone:

8. Hospital: (Emergency Room) Phone:

9. Emergency Route:

10. Fire Department: Phone:

11. Police: Phone:

12. Poison Control Center: Phone:

13. Site Emergency Notification/Evacuation Method:

14. NEIC Health and Safety Officer: Steve Fletcher (303) 236-5111 ext. 283

15. Radiation Safety Assistance: **Jed Harrison, Director (702) 798-2476**
**Office of Radiation Programs**
**Las Vegas Facility**

*****************************************************************************************

### Hazard Evaluation

16. Check all known or potential hazards: __ Radiation __ Toxics __ Fire/Explosion

__ Corrosives __ $0_2$ Deficiency __ Noise __ Physical __ Biological

__ Dusts __ Heat/Cold Stress

**NOTE: DISCUSS HAZARDS AND PRECAUTIONS IN DETAIL IN WORK PLAN BELOW.**

17. Specify unusual working conditions/limitations (excavations, confined spaces, lagoons, elevated surface, weather, darkness, etc.)*:

* Attach specific hazard management plans, if applicable.

18. Potential Chemical Hazard:

| Chemical | TLV/ ILDH | Route of Exposure | Acute Symptoms | Odor Level | Odor/Visual Description |
|---|---|---|---|---|---|
| | | | | | |
| | | | | | |
| | | | | | |

* Potential or confirmed carcinogen

************************************************************************************

## Work Plan

19. List tasks, anticipated hazards checked above, and control measures which will be taken, including levels of protection:

| Task | Hazards | Level of Protection (A, B, C, D) and Control Measures |
|---|---|---|
| | | |
| | | |
| | | |

20. Health Hazard Monitoring Plan:

| Constituent | Type of Sample | Frequency | Instrument | Notes |
|---|---|---|---|---|
| | | | | |
| | | | | |

| | | | | |
|---|---|---|---|---|
| | | | | |

. Site Control/Security Measures:

22. Decontamination Procedures (personnel hygiene, contaminated clothing, equipment, instruments, etc.):

23. Disposal Procedures (contaminated equipment, supplies, decontamination solutions, etc.):

************************************************************************************

Approvals

This site HASP has been reviewed and constitutes the minimum anticipated safety requirements for personnel engaged in field activities at this project site. However, the Project Leader has the authority to change these requirements, based upon the conditions present at the site.

Approved by:

24. Project Leader: ______________________ Date: ____________

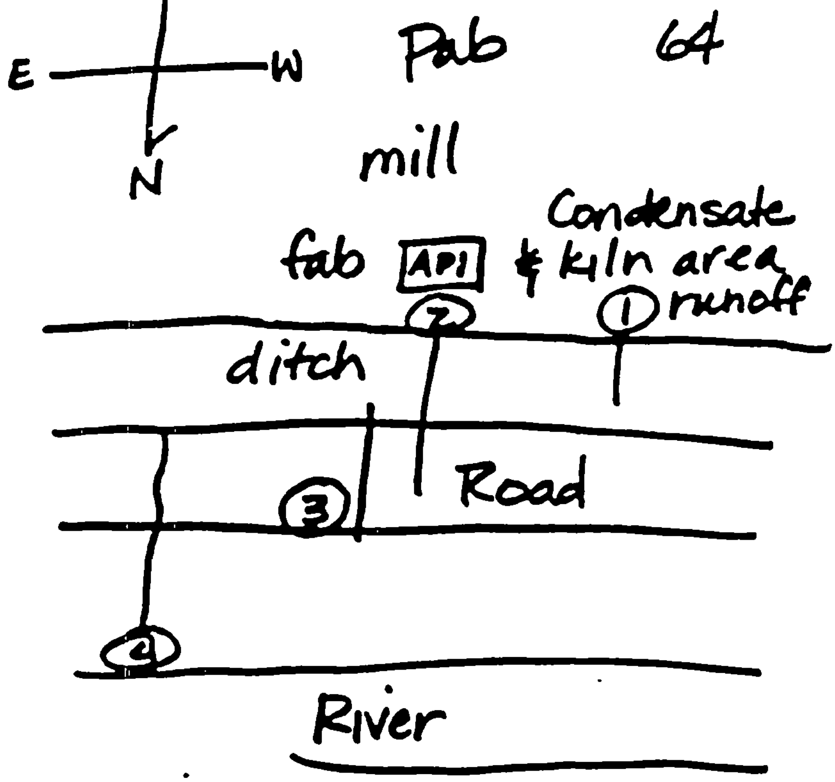
25. Branch Chief: ______________________ Date: ____________

Health & Safety Officer: ______________________ Date: ____________

SITEHASP.FRM:09/97

# Field Logbook Entry: Sampling

Sampling




See field data sheet for sample info.

| | 1 | 2 | 3 | 4 | 6 |
|---|---|---|---|---|---|
| Temp | 84°f | 60°f | 64°f | 58°f | Trans Blank |
| pH | 10.7 | 7.2 | 9.3 | 7.3 | |
| flow | 29gpm | 0.25 | 22.5 | 22.5 | |
| t | 1:00 | 1:15 | 1:25 | 1:45 | 1:30 |
| | 8824440 | 8824441 | 8824442 | 8824443 | 8824444 |

# Example of the Sample Seal

<table>
<tr><td rowspan="3">UNITED STATES<br>ENVIRONMENTAL PROTECTION AGENCY<br>INSPECTOR'S SEAL<br><br>3</td><td>Sample No.<br>1</td><td>Date<br>2</td><td rowspan="3">6</td><td rowspan="3">6</td></tr>
<tr><td colspan="2">Signature<br>4</td></tr>
<tr><td colspan="2">Print Name and Title<br>5</td></tr>
</table>

(1) Insert sample number

(2) Insert date sealed

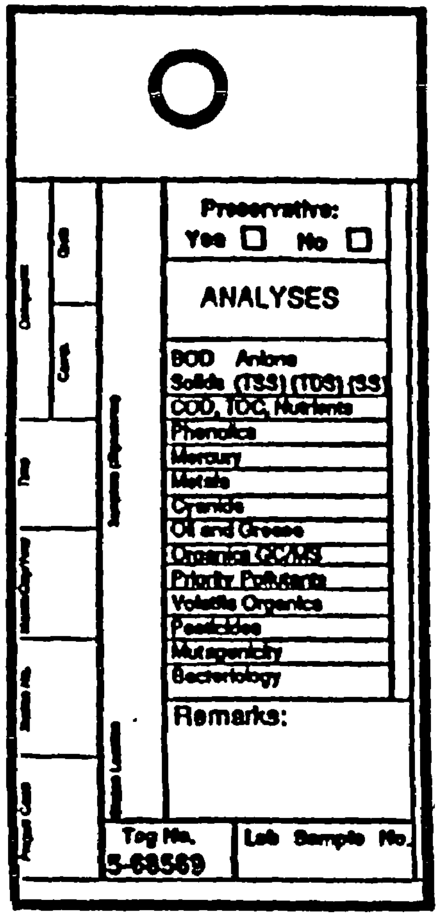
(3) Print location of collector's station

(4) Signature of persons sealing the sample

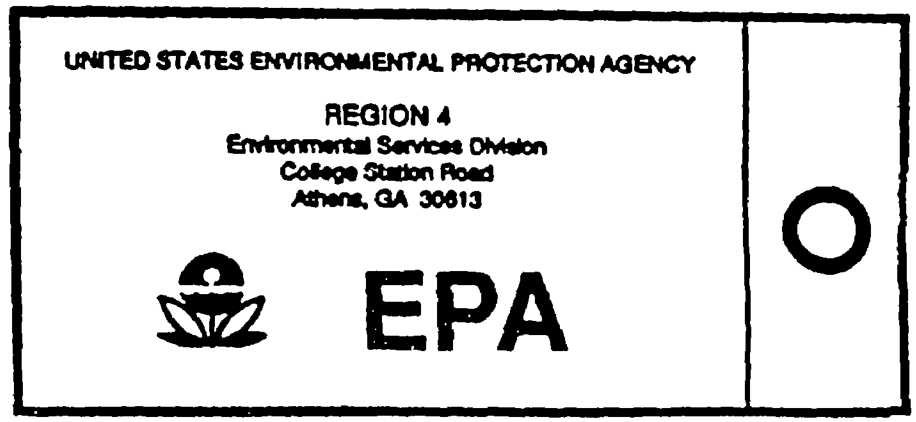
(5) Print name (same as signature) and title of sealer

(6) When a seal is broken for any purpose, initial here and enter the date broken. Submit broken seal with sample records

# Example of the Sample Tag




FRONT




BACK

Handout 12-5

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

NATIONAL ENFORCEMENT INVESTIGATIONS CENTEI
Building 53, Box 25227, Denver Federal Center
Denver, Colorado 80225

# RECEIPT FOR SAMPLES

| PROJ. NO. 90Q | PROJECT NAME | Name of Facility |
|---|---|---|
| SAMPLERS: *(Signature)* | | Facility Location |
| Split Samples Offered ( ) Accepted ( ) Declined | | |

| STA. NO. | DATE | TIME | COMP. | GRAB | SPLIT SAMPLES | TAG NUMBERS | STATION DESCRIPTION | NO. OF CON-TAINERS | REMARKS |
|---|---|---|---|---|---|---|---|---|---|
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| Transferred by: *(Signature)* | | Received by: *(Signature)* | | Telephone |
|---|---|---|---|---|
| Date | Time | Title | Date | Time |

Distributi… inal to Coordinator Field Files; Copy to Facility

CHAIN OF CUS‑‑DY RECORD

Denver Colorado 80225

Handout 12

| PROJ. NC | OJECT NAME | | | | | NO. OF CON-TAINERS | | | | | | | REMARKS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| SAMPLERS: *(Signature)* | | | | | | | | | | | | | |
| STA. NO. | DATE | TIME | COMP. | GRAB | STATION LOCATION | | | | | | | | |
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| Relinquished by: *(Signature)* | Date / Time | Received by: *(Signature)* | Relinquished by: *(Signature)* | Date / Time | Received by: *(Signature)* |
|---|---|---|---|---|---|
| Relinquished by: *(Signature)* | Date / Time | Received by: *(Signature)* | Relinquished by: *(Signature)* | Date / Time | Received by: *(Signature)* |
| Relinquished by: *(Signature)* | Date / Time | Received for Laboratory by: *(Signature)* | Date / Time | Remarks | |

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

N 3211

TABLE 2-34
CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES FOR AQUEOUS MATRICES[A]

| Name | Container[1] | Preservation | Maximum holding time[2] |
|---|---|---|---|
| **Inorganic Tests:** | | | |
| Chloride | P, G | None required | 28 days |
| Cyanide, total and amenable to chlorination | P, G | Cool to 4°C; if oxidizing agents present add 5 mL 0.1N $NaAsO_2$ per L or 0.06 g of ascorbic acid per L; adjust pH>12 with 50% NaOH. See Method 9010 for other interferences. | 14 days |
| Hydrogen ion (pH) | P, G | None required | 24 hours |
| Nitrate | P, G | Cool to 4°C | 48 hours |
| Sulfate | P, G | Cool to 4°C | 28 days |
| Sulfide | P, G | Cool to 4°C, add zinc acetate | 7 days |
| **Metals:** | | | |
| Chromium VI | P, G | Cool to 4°C | 24 hours |
| Mercury | P, G | $HNO_3$ to pH<2 | 28 days |
| Metals, except chromium VI and mercury | P, G | $HNO_3$ to pH<2 | 6 months |
| **Organic Tests:** | | | |
| Acrolein and acrylonitrile | G, PTFE-lined septum | Cool to 4°C, 0.008% $Na_2S_2O_3$[3], Adjust pH to 4-5 | 14 days |
| Benzidines | G, PTFE-lined cap | Cool to 4°C; 0.008% $Na_2S_2O_3$[3] | 7 days until extraction, 40 days after extraction |
| Chlorinated hydrocarbons | G, PTFE-lined cap | Cool to 4°C; 0.008% $Na_2S_2O_3$[3] | 7 days until extraction, 40 days after extraction |
| Dioxins and Furans | G, PTFE-lined cap | Cool to 4°C, 0.008% $Na_2S_2O_3$[3] | 30 days until extraction, 45 days after extraction |
| Haloethers | G, PTFE-lined cap | Cool to 4°C, 0.008% $Na_2S_2O_3$[3] | 7 days until extraction, 40 days after extraction |
| Nitroaromatics and cyclic ketones | G, PTFE-lined cap | Cool to 4°C, 0.008% $Na_2S_2O_3$[3], store in dark | 7 days until extraction, 40 days after extraction |
| Nitrosamines | G, PTFE-lined cap | Cool to 4°C, 0.008% $Na_2S_2O_3$[3], store in dark | 7 days until extraction, 40 days after extraction |

(continued on next page)

TABLE 2-34 (continued)

| Name | Container[1] | Preservation | Maximum holding time |
|---|---|---|---|
| Oil and grease | G | Cool to 4°C, add 5 mL diluted HCl | 28 days |
| Organic carbon, total (TOC) | P, G | Cool to 4°C, store in dark[2] | 28 days |
| Organochlorine pesticides | G, PTFE-lined cap | Cool to 4°C | 7 days until extraction, 40 days after extraction |
| Organophosphorus pesticides | G, PTFE-lined cap | Cool to 4°C[4] | 7 days until extraction, 40 days after extraction |
| PCBs | G, PTFE-lined cap | Cool to 4°C | 7 days until extraction, 40 days after extraction |
| Phenols | G, PTFE-lined cap | Cool to 4°C, 0.008% $Na_2S_2O_3$[3] | 7 days until extraction, 40 days after extraction |
| Phthalate esters | G, PTFE-lined cap | Cool to 4°C | 7 days until extraction, 40 days after extraction |
| Polynuclear aromatic hydrocarbons | G, PTFE-lined cap | Cool to 4°C, 0.008% $Na_2S_2O_3$[3], store in dark | 7 days until extraction, 40 days after extraction |
| Purgeable aromatic hydrocarbons | G, PTFE-lined septum | Cool to 4°C, 0.008% $Na_2S_2O_3$[2,3] | 14 days |
| Purgeable Halocarbons | G, PTFE-lined septum | Cool to 4°C, 0.008% $Na_2S_2O_3$[3] | 14 days |
| Total organic halides (TOX) | G, PTFE-lined cap | Cool to 4°C, Adjust to pH<2 with $H_2SO_4$ | 28 days |
| Radiological Tests: | | | |
| Alpha, beta and radium | P, G | $HNO_3$ to pH<2 | 6 months |

[A] Table originally excerpted, in part, from Table II, 49 FR 28, October 26, 1984, and revised as appropriate for SW-846. See Chapter Three, Chapter Four, or the individual methods for more information.
[1] Polyethylene (P) or Glass (G)
[2] Adjust to pH<2 with $H_2SO_4$, HCl or solid $NaHSO_4$. Free chlorine must be removed prior to adjustment.
[3] Free chlorine must be removed by the appropriate addition of $Na_2S_2O_3$.
[4] Adjust samples to pH 5-8 using NaOH or $H_2SO_4$.

TABLE 3-1.

SAMPLE HOLDING TIMES, RECOMMENDED DIGESTION VOLUMES AND RECOMMENDED COLLECTION VOLUMES FOR INORGANIC DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

| Measurement | Digestion Volume. (mL)[a,c] | Collection Volume (mL)[a,c] | Treatment/ Preservative Holding Time[b] |
|---|---|---|---|
| Inorganic Analytes (except hexavalent chromium and mercury): | | | |
| Aqueous | | | |
| Total | 100 | 600 | $HNO_3$ to pH <2<br>6 months |
| Dissolved | 100 | 600 | Filter on site;<br>$HNO_3$ to pH <2<br>6 months |
| Suspended | 100 | 600 | Filter on site<br>6 months |
| Solid | | | |
| Total | 2 g | 200 g | 6 months |
| Hexavalent Chromium: | | | |
| Aqueous | 100 | 400 | 24 hours<br>Store at 4°± 2°C<br>until analyzed |
| Solid | 2.5 g | 100 g | One month<br>to extraction, 4 days<br>after extraction<br>Store at 4°± 2°C<br>until analyzed |
| Mercury: | | | |
| Aqueous | | | |
| Total | 100 | 400 | $HNO_3$ to pH <2<br>28 days |
| Dissolved | 100 | 400 | Filter;<br>$HNO_3$ to pH <2<br>28 days |
| Solid | | | |
| Total | 0.2 g | 200 g | 28 days<br>Store at 4°± 2°C<br>until analyzed |

[a] Unless stated otherwise.
[b] Either glass or plastic containers may be used.
[c] Any sample volume reduction from the reference method's instructions must be made in the exact proportion as described in the method and representative sampling must be maintained.

## TABLE 4-1.
## SAMPLE CONTAINERS, PRESERVATION, TECHNIQUES, AND HOLDING TIMES

| VOLATILE ORGANICS | | | |
|---|---|---|---|
| Sample Matrix | Container | Preservative | Holding Time |
| Concentrated Waste Samples | Method 5035: 40-mL vials with stirring bar.<br>Method 5021: See method.<br>Methods 5031 & 5032: 125-mL widemouth glass container.<br>Use Teflon-lined lids for all procedures. | Cool to 4°C. | 14 days |
| Aqueous Samples With No Residual Chlorine Present | Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps | Cool to 4°C and adjust pH to less than 2 with $H_2SO_4$, HCl, or solid $NaHSO_4$. | 14 days |
| Aqueous Samples WITH Residual Chlorine Present | Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps | Collect sample in a 125-mL container which has been pre-preserved with 4 drops of 10% sodium thiosulfate solution. Gently swirl to mix sample and transfer to a 40-mL VOA vial. Cool to 4°C and adjust pH to less than 2 with $H_2SO_4$, HCl, or solid $NaHSO_4$. | 14 days |
| Acrolein and Acrylonitrile in Aqueous Sample | Methods 5030, 5031, & 5032: 2 X 40-mL vials with Teflon-lined septum caps | Adjust to pH 4-5. Cool to 4°C. | 14 days |
| Solid Samples (e.g. soils, sediments, sludges, ash) | Method 5035: 40-mL vials with septum and stirring bar.<br>Method 5021: See method.<br>Methods 5031 & 5032: 125-mL widemouth glass container with Teflon-lined lids. | See the individual methods. | 14 days |

TABLE 4-1 (Continued)

| SEMIVOLATILE ORGANICS/ORGANOCHLORINE PESTICIDES/PCBs AND HERBICIDES | | | |
|---|---|---|---|
| Sample Matrix | Container | Preservative | Holding Time |
| Concentrated Waste Samples | 125-mL widemouth glass with Teflon-lined lid | None | Samples extracted within 14 days and extracts analyzed within 40 days following extraction. |
| Aqueous Samples With No Residual Chlorine Present | 1-gal., 2 x 0.5-gal., or 4 x 1-L amber glass container with Teflon-lined lid | Cool to 4°C | Samples extracted within 7 days and extracts analyzed within 40 days following extraction. |
| Aqueous Samples WITH Residual Chlorine Present | 1-gal., 2 x 0.5-gal., or 4 x 1-L, amber glass container with Teflon-lined lid. | Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%). Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to 4°C. | Samples extracted within 7 days and extracts analyzed within 40 days following extraction. |
| Solid Samples (e.g. soils, sediments, sludges, ash) | 250-mL widemouth glass container with Teflon-lined lid | Cool to 4°C | Samples extracted within 14 days and extracts analyzed within 40 days following extraction. |

13

**SESSION 13**

**TOPIC: CLOSING CONFERENCE**

**Time: 30 minutes**

| | |
|---|---|
| **PURPOSE** | • Explain the purpose, scope, and limitations of a closing conference. |
| **KEY POINTS** | • Provide an opportunity for inspectors to discuss preliminary findings.<br>• Resolve any outstanding questions or issues and verify information.<br>• Provide an opportunity to complete final paperwork (for example, TSCA, CBI forms). |
| **LIST OF VISUALS** | 13-1 Closing Conference (Title Slide)<br>13-2 Closing Conference |
| **LIST OF HANDOUTS** | None |

## Closing Conference

13-1

## Closing Conference

- Format varies
- Preliminary (tentative) discussion of findings
- Resolve outstanding issues
- Complete paperwork
- Follow your agency's policy in regard to providing notice of violations

13-4

## SESSION 14

## TOPIC: INSPECTION REPORT

**Time: 90 minutes**

**PURPOSE**

- Explain the purpose and importance of the inspection report.
- Discuss and evaluate the elements of a good inspection report.
- Provide practical experience in report writing style.

**KEY POINTS**

- The inspection report should be factual, free of opinions and conclusions.
- The report should explain in detail what happened during the inspection and substantiate in full any potential violations.
- Your career may be evaluated upon how well you communicate information.

**LIST OF VISUALS**

14-1 Inspection Report (Title Slide)
14-2 Inspection Report
14-3 Inspection Report
14-4 Inspection Report
14-5 Report Heading
14-6 Outline—Executive Summary
14-7 Outline—Executive Summary (continued)
14-8 Outline—Technical Report
14-9 Description of Facility
14-10 Regulatory Status and Compliance History
14-11 Inspection Methods
14-12 Sampling Activities
14-13 Information About the Closing Conference
14-14 Compliance Findings
14-15 Other Areas of Concern
14-16 Additional Information
14-17 Outline—Appendices
14-18 Handout: Evaluation Guide for Reports
14-19 Tips for Writing Inspection Reports
14-20 Consider the Audience for (each Section of) the Report
14-21 Be Accurate and Impartial
14-22 Write To Express—Not To Impress
14-23 Avoid the "It" Habit and Other Vague References
14-24 Be Simple and Concise
14-25 Be Coherent
14-26 Use Active Voice/Action Verbs

**LIST OF HANDOUTS**

## Inspection Report

## Inspection Report

- Effective reports
  - Purpose
  - Objective
  - Importance

## Inspection Report

- Official files
- Check lists or narrative format?
  - Remember:
    - You may be asked for testimony/deposition

## Inspection Report

- Typical Outline for Report
  - Executive Summary ...
  - Technical Report ...
    - Recommendations? (discoverable)
  - Findings and Conclusions ...
  - Appendices ...

## Report Heading

- Type of inspection
- Name of site
- Address of facility
- Date of inspection
- Date of report
- Inspection team or participants

## Outline - Executive Summary

- Introduction and overview
- Project objectives and scope of the inspection
- Summary of factual findings
  - Grouped by statute or process line / wastestream
  - Potential violations (areas of noncompliance) identified
- May include (or refer to later section on) areas of concern

## Outline - Executive Summary
(continued)

- Provides a summary of information in the technical report
  - Is supported fully by information in the technical report
  - Contains no information that does not appear in the technical report

14-7

## Outline - Technical Report

- Description of the facility ...
- Regulatory status and compliance history ...
- Compliance findings
- Inspection methods ...
- Sampling activities detailed ...
- Discussion of areas of concern ...

14-8

## Description of Facility

- Length of time at location
- Ownership
- Number of employees
- Hours of operation
- Products and services
- Description of processes and operations
- Medium-specific processes
- Wastestreams and waste management methods
- Environmental management system

14-9

## Regulatory Status and Compliance History

- Includes information about applicable:
  - Regulations
  - Permits
  - Consent decrees
  - Other documents
- Includes history of compliance

## Inspection Methods

- Techniques
- Strategies
- Procedure
- Adjustments
- Chronological recount

## Sampling Activities

- Sampling plan
- Location
- Chain of custody
- Variations
- Results
- Discussion

## Information About the Closing Conference

- Answers the following questions:
  - Who was present?
  - What was discussed?
  - What did the facility agree to do?
- Identifies pollution prevention opportunities
- Discusses environmental justice issues
- Discusses compliance assistance
- Discusses other issues or concerns

## Compliance Findings

- Answers the following questions:
  - Where did you go and what did you see?
  - Who did you talk to?
  - What records did you review?
  - What did you not do?
  - What questions were asked and who responded?
- Includes photographs, copies, and all other documentation
- Includes sampling activities

## Other Areas of Concern

- Non-regulatory observations
- Environmental concerns
- Community issues

## Additional Information

- Safety issues
- Access issues
- Compliance assistance
- Agency initiatives
  - P2, EJ, EMS, 33/50
- Laws of other agencies

14-56

## Outline - Appendices

- Complex maps, diagrams
- Detailed testing, monitoring, sampling methods
- Raw data
- Raw test results and extensive analyses

14-57

## Handout: Evaluation Guide for Reports

14-58

clear

source

complete

## Tips for Writing Inspection Reports

factual

etc.

accurate

concise

audience

14-19

## Consider the Audience for (each Section of) the Report

- Audience of the Executive Summary
  - Senior management unable to read the full report
  - Non-technicians who may not understand the full report
  - Attorneys and individuals in other programs needing an overview of the inspection and findings
- Technical Report
  - Engineers, scientists, and policy writers developing the case
- Findings
  - All enforcement personnel involved with the case
- Appendices

14-20

## Be Accurate and Impartial

- Report fact as fact and hearsay as hearsay
  - Did you see actual waste oil? OR only drums labeled as "waste oil"?
  - Did you see Jimmy dump the container OR did someone else tell you that he did the dumping?
- Leave out nothing as if concealed or withheld
  - Even if it may appear to weaken the case

14-21

## Write To Express – Not To Impress

Problem:

Next I crossed the dark lot, where I discovered many drums on a forklift headed for the hidden warehouse.

Better:

I observed drums being transported to the warehouse.

## Avoid the "It" Habit and Other Vague References

Problem:

I inspected tank A, still B, and lagoon C. It was overflowing and its secondary containment was inadequate.

Better:

I inspected tank A, still B, and lagoon C. Lagoon C was overflowing. The secondary containment around lagoon C was not capturing all the overflow.

## Be Simple and Concise

Problem:

Write all your sentences as short as you possibly can, and always avoid the use of complicated terminology when possible.

Better:

Use short sentences. Avoid complicated terms.

## Be Coherent

Problem:

It is considered that a detailed examination is unnecessary at this time

Better:

Detailed examination is not necessary.

## Use Active Voice / Action Verbs

Problem:

It is recommended that ...

Better:

I recommend ...

Who?

What?

When?

## Essentials of Good Report Writing

Which?

Why?

Where?

How?

## Who?

**Problem:**

*The degreaser mechanic*

**Better:**

Elvis Ferguson, the junior degreaser mechanic working during the inspection

## What?

**Problem:**

If there had been a fire, no firefighter equipment could have been carried down the narrow aisle.

**Better:**

The aisle space between the drums was 16 inches.

## When?

**Problem:**

Mr. Ferguson called me and told me the hose had ruptured at 10:30. When I arrived, it was overflowing.

**Better:**

Mr. Ferguson called me at 10:30 a.m. He told me that the hose had ruptured at 10:15 a.m. When I arrived at 11:00 a.m., the secondary containment was overflowing.

## Where?

**Problem:**

The tank behind the warehouse

**Better:**

The 1,000-gallon tank used to store chloride catalyst before it is recycled

## Why?

**Problem:**

They ship drums to the warehouse because the storage area is so small.

**Better:**

Mr. Ferguson stated that 2 drums per day are taken to the warehouse because storage area B is too small. Measurements indicate that storage area B is large enough to contain 6 drums. Plant records indicate that 8 drums per day are generated by the #2 production line.

## How?

**Problem:**

Appropriate drums are used to avoid corrosion.

**Better:**

The facility neutralizes hydrochloric acid from detergent line #1 in plastic "poly" drums.

**Exercise:**
**Review Example Reports**

# *INSPECTION REPORT EVALUATION GUIDE*

The organization and format of an inspection report can vary, according to the practice of the office or program under which the inspection is conducted, the particular circumstances of the inspection, and the individual writing style of the report writer. No matter what form the report takes, however, the report and its attachments should answer the following questions.

## Basic Inspection Information

Who prepared the inspection report?

Who signed the inspection report, and on what date?

Who performed the inspection (all participants)?

What is the name and location of the facility or site?

What is the mailing address and telephone number of the facility or site?

What is the name and title of the responsible official who was contacted?

What was the reason for the inspection (for example, routine, response to a complaint, or for cause)?

What are the names and titles of all of the government personnel who participated in the inspection?

## *Entry and Opening Conference*

What are the facts about the entry (date, time, entry location, and agent in charge)?

Is there documentation that proper entry procedures were followed?

Were all required notices and credentials presented?

Is there documentation that facility officials were informed of their right to claim that information is confidential?

Were there any unusual circumstances concerning gaining consent to enter (for example, reluctance, attempts to limit the scope of the inspection, or attempts to place special requirements on inspectors)? How were such circumstances handled?

Who was present at the opening conference? What topics were discussed?

**Background of the Facility or Site**

What is the type of facility or site?

What types of activities and operations take place at the facility or site?

Who owns the facility or site (for example, a corporation, an individual, a partnership, a federal or state agency, or a nonprofit organization)?

How many years has the facility been in existence?

How many employees work at the site?

Have any major modifications been made at the facility? Are any modifications or expansions planned?

At what level of capacity is the facility operating? For how many shifts does it operate, and how many hours per day and days per week? What relationship does this information have to the inspection that was performed?

Which operations, processes, and activities at the facility were examined during the inspection?

Which operations, processes, and activities at the facility were not examined?

**Inspection Activities**

- **Records Inspection**

  Is there a general description of how records are kept at the facility?

  What was the purpose of reviewing records?

  What records of the facility were reviewed?

  How were the specific records selected for review (was an auditing technique used or were all records reviewed)?

  Are photocopied records or data manually copied from records adequately identified and documented?

  Were any suspected violations found? (Each should be fully documented, making sure that all the information required for the section set forth below on suspected violations is included.)

- **Physical Sampling**

  What was the inspector's sampling plan for the facility or site?

What physical samples were collected at the site?

Are the sampling techniques used explained adequately?

Are all samples clearly linked to an identification number, location, and purpose?

Are sampling conditions and other physical aspects of the sample (for example, color, texture, and viscosity) described?

Were any deviations from the sampling plan or SOPs explained and documented adequately?

Are chain-of-custody procedures documented?

Are the results of laboratory analysis presented clearly?

How do the results of analysis of samples compare with limits set forth in the facility's permit?

- **Illustrations and Photographs**

Are photographs taken during the inspection included and properly documented?

Is there some information about the inspection that could be made easier to understand through the inclusion of a diagram or sketch?

If sketches, diagrams, or maps are used, is the scale or other relationship shown clearly?

- **Interviews**

What are the names and titles of officials of the facility and other personnel who were interviewed?

Are their statements summarized clearly?

What are the names and addresses of any other individuals who were interviewed or who were witnesses?

- **Closing Conference**

Does the report include documentation that required receipts for samples and documents were provided?

Does the report include documentation that officials of the facility were given an opportunity to make confidentiality claims?

Does the report note statements the inspector made to officials of the facility about compliance status, recommending actions to take, or other matters?

**Documentation of Suspected Violations**
The heart of the inspection report is really the documentation and substantiation of suspected violations, which allows EPA to determine whether a violation occurred, how and why it occurred, and its seriousness. This substantiating information includes all the evidence of various kinds that has been collected. In an actual inspection report, some of the questions on the preceding pages might be answered in the portion of the report that discusses the evidence collected and other particulars of each suspected violation.

- **Documentation of Suspected Violation**

  For each suspected violation, the inspection report should answer the following questions:

  What regulation does the inspector suspect has been violated?

  What information proves that the cited regulation applies to the facility or site?

  According to the elements of the regulation, what information proves that the suspected violation occurred?

  What sampling methods (if appropriate) were used to determine that the violation occurred? Are any deviations from sampling methods adequately explained?

  What information shows that possible exemptions to the rule do not apply?

- **Cause of Violation**

  Note: Not all programs require this information, but it may be useful, even when it is not required, for such purposes as negotiating an appropriate remedy and penalty and planning future inspections. Causal information must be stated carefully so that it does not provide the violator with an excuse for the violation.

  What information documents the possible cause of the violation (for example, direct observations of gauge readings, production logs, physical appearance of materials, or statements by facility personnel)?

  Is there any supporting information that confirms or disproves a possible claim of an upset or other exempt activity?

- **Other Mitigating and Aggravating Factors**

  The level of enforcement response is based on the seriousness of the violation. Amounts of civil penalties are based on the gravity and circumstances of the violation, which is usually a calculation of the extent of the violation (amount of material involved) and the extent of the actual or potential harm that was or could be caused by the violation. The base penalty can be adjusted upward or downward because of such factors as past compliance history or efforts made by the facility to correct the violation.

The inspection report should contain information that will support the appropriate determination of the seriousness and extent of the violation, as well as other information that might be useful in calculating a penalty.

What is the seriousness of the violation (for example, amount of emissions, length of time of excess emissions, nature of emissions, location of source, and perceived effect on the public)?

What harm resulted or could result from the violation?

What efforts did the facility make to correct the violation?

How difficult will it be to comply (considering such factors as availability of technology, cost of complying, and time required to correct the violation)?

What is the facility's past compliance history?

# SAMPLE INSPECTION REPORTS

This handout contains samples of actual inspection reports; only the names have been changed. Each report is of acceptable quality, although each has both strong and weak points.

Using the Inspection Report Evaluation Guide, evaluate the sample reports.

- How well does each of these reports meet the criteria in the evaluation guide?
- What are the strengths of each report? What problems can you identify?
- Which report provides the strongest support for case development? The weakest? Why?
- If you were the supervisor of report writer A, what comments would you make on the report? To writer B? To writer C?

# INSPECTION REPORT A: CASTINGS MANUFACTURING, INC.

## RCRA SAMPLING INSPECTION

This company manufactures steel castings for the railroad industry. The manufacturing facility occupies forty acres on the northeast side of Bigcity. The 12-1/2 acre landfill owned by the company is located in Rural County near Bubbatown to the southeast of the intersection of 4th and Main.

The purpose of this sampling inspection was to determine if waste generated and disposed of by this facility at its Bubbatown landfill is RCRA hazardous waste. The main wastestream in question is a mixture of electric arc furnace (EAF) dust and sand wash slurry. This dust/slurry mixture is claimed to be nonhazardous by the facility. Other wastestreams of interest are from the five other dust collectors at the facility.

On August 6, 1986, Jim Sleuth, Sam Tweed, and Mike Heard of the USEPA Regional Office and Jean Parker of the USEPA Regional Waste Management Division, conducted an unannounced sampling inspection at the above-mentioned company. We arrived at the company landfill on 8/6/86 at 0915 to wait for a truck to arrive from the company's manufacturing facility in Bigcity. A truck did arrive at 0925 but it was not hauling the type of waste desired to sample during the dump. It was later determined that this load contained dust collector fines. At 0945 another truck arrived at the landfill but it also was not hauling the dust/slurry mixture desired to sample. At this time, Mr. Apple and Mr. Banana of the company arrived at the landfill to inquire about the purpose of our inspection. We informed them that we wished to sample the dust/slurry mixture as it was being dumped into the landfill. Mr. Banana did not know if any of this mixture would be disposed of that day and he asked us to return with him to the Bigcity facility where he could determine when disposal of that material would occur. Before leaving for the landfill, samples 86EF10S01 and S02 (see Table 1) were collected of the two loads that were dumped that morning. These samples were split with the facility.

At 1115 a meeting was held with Mr. Cake, assistant works manager and Mr. Donut at the Bigcity facility. It was determined that the sand wash system was not generating any slurry that day and that we could not sample the EAF dust/sand slurry mixture until the following day. We then proceeded to collect samples S03-S06.

On August 7, 1986, Mr. Sleuth, Ms. Parker, and Mr. Heard returned to the facility in Bigcity and collected samples S07-S10 (see Table 1). It was observed that a tanker truck of sand wash slurry was mixed with a load of EAF dust at the facility. This truck was followed to the landfill, where it was sampled while it was dumped. Before being dumped a core of the top ten to twelve inches of the load was taken in a 2" diameter plastic tube; eight to ten inches of this material all appeared to be dry EAF dust. The bottom two inches were damp EAF dust (mixed with slurry). The contents of this core were used for sample S14. As the truck was dumping, five jars (one quart in size) were collected of the material coming out of the truck. The first two (chronologically) were composited and split as sample S13, the third jar was discarded and the last two jars were composited and split as sample S12. At the very end of the dump a quantity of dry EAF dust was observed to float out on top of the discharge. A sample of this dry material (S11) was collected from the top of the dumped material after it was on the ground. All samples collected except D09, S14, and S15 were split with the facility. The sampling results can be found in Attachment 1. Samples D09, S11, and S14 were all found to exceed the EPA toxicity limit of 1 ppm for cadmium and 5 ppm for lead.

Figure 1 - Landfill

August 6&7, 1986

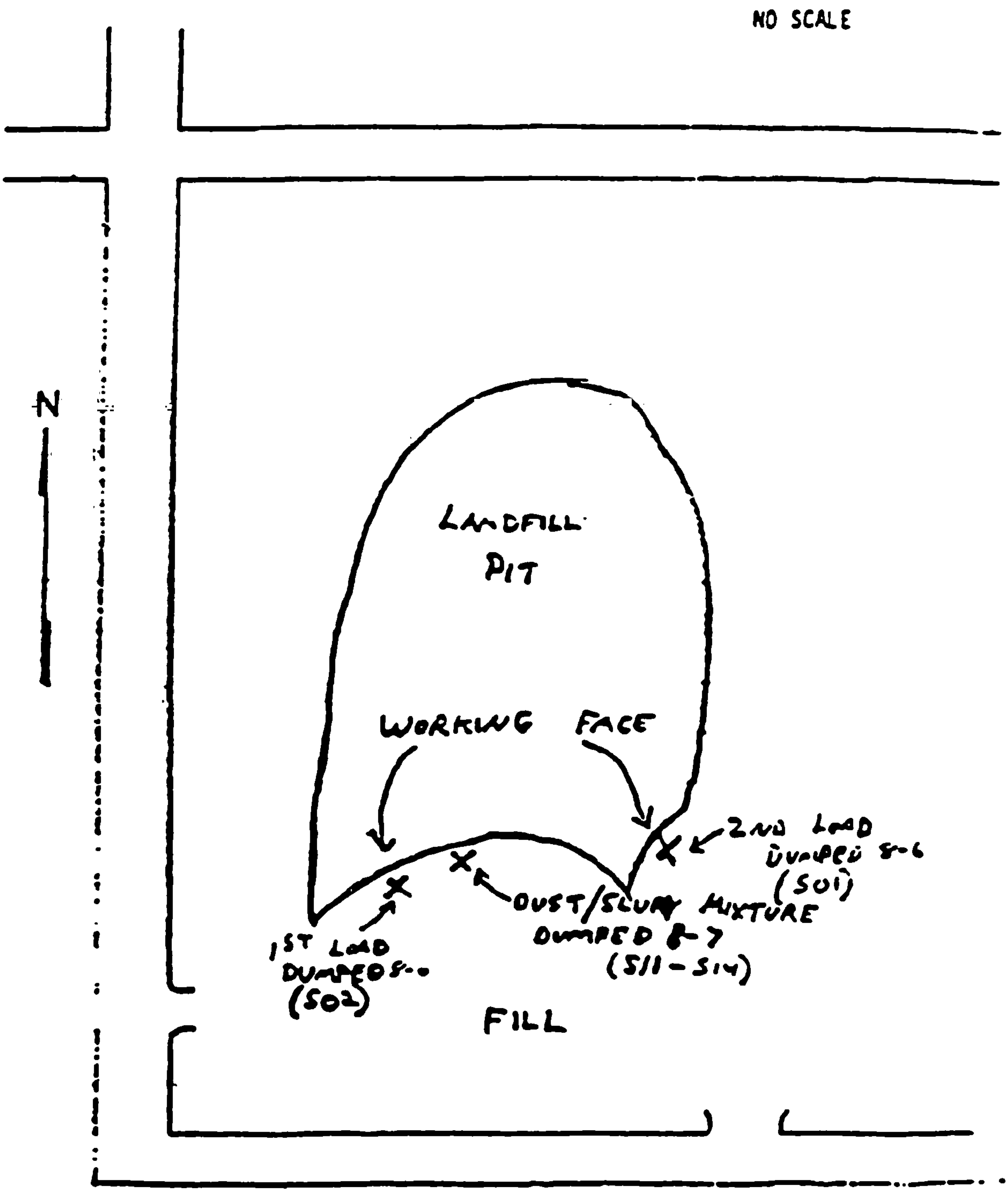


TABLE I

Sample Locations
August 6 & 7, 1986

| STA. NO. | DATE 1986 | TIME | COMPOSITE | GRAB | STATION LOCATION |
|---|---|---|---|---|---|
| S01 | 8-6 | 1006 | X | | Waste pile from load #2 |
| S02 | 8-6 | 1030 | X | | Waste pile from load #1 |
| S03 | 8-6 | 1327 | | X | Carrier blast dust collector |
| S04 | 8-6 | 1338 | | X | Knockout dust collector |
| S05 | 8-6 | 1415 | | X | Cabinet blast dust collector |
| S06 | 8-6 | 1425 | | X | Tumblast dust collector |
| S07 | 8-7 | 1045 | | X | South End sand system |
| S08 | 8-7 | 1100 | | X | Sand wash and wet scrubber slurry |
| S09 | 8-7 | 1100 | | X | EAF dust (duplicate) |
| S10 | 8-7 | 1300 | | X | EAF dust |
| S11 | 8-7 | 1420 | | X | After dump (dust/slurry mixture) |
| S12 | 8-7 | 1418 | X | | Last half of dump * |
| S13 | 8-7 | 1414 | X | | First half of dump * |
| S14 | 8-7 | 1410 | | X | Core of load * |
| S15 | 8-7 | 1700 | | | Blank |

Attachment 1 - Analytical Results of Sampling

## ANALYTICAL RESULTS OF AUGUST 6 AND 7, 1986, SAMPLING
(all results are in ppm)

| Sample Number | Ag | Ba | Cd | Cr | Pb | As | Se | Hg | Fluoride | Phenol | Cyanide |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Maximum Contaminant Level for EP Toxicity: | 5.0 | 100.0 | 1.0 | 5.0 | 5.0 | 5.0 | 1.0 | 0.2 | | | |
| 86ER10 | | | | | | | | | | | |
| S01 | <0.006 | 0.047 | <0.01 | <0.008 | <0.07 | <0.002 | <0.02 | <0.0001 | 0.04 | 2.7 | <1.0 |
| S02 | <0.006 | 0.058 | <0.01 | <0.008 | <0.07 | <0.002 | <0.02 | <0.0001 | 0.07 | 6.8 | <1.0 |
| S03 | <0.006 | 0.028 | <0.01 | <0.008 | <0.07 | <0.002 | <0.02 | <0.0001 | 0.1 | 4.6 | <1.0 |
| S04 | <0.006 | 0.150 | <0.01 | <0.008 | <0.07 | <0.002 | <0.02 | <0.0001 | 0.1 | 37.8 | 2.0 |
| S05 | <0.006 | 0.049 | <0.01 | <0.008 | <0.07 | <0.002 | <0.02 | <0.0001 | 0.2 | 15.2 | <1.0 |
| S06 | <0.006 | 0.114 | <0.1 | 0.08 | <0.7 | <0.002 | <0.002 | <0.0001 | 0.1 | 7.3 | <1.0 |
| S07 | <0.006 | 0.118 | <0.01 | 0.00892 | <0.7 | 0.006 | <0.002 | <0.0001 | 0.2 | 68.2 | 2.0 |
| S08 | <0.006 | 0.114 | <0.01 | <0.008 | <0.07 | <0.002 | <0.002 | <0.0001 | 0.2 | 15.6 | <3.0 |
| D09 | <0.006 | 0.950 | 4.32* | <0.08 | 14.1* | <0.002 | 0.012 | 0.0005 | 2.0 | 3.2 | <1.0 |
| S10 | <0.06 | 0.796 | 4.3* | <0.08 | 14.5* | <0.002 | 0.04 | 0.0004 | 1.5 | 3.3 | <1.0 |
| S11 | <0.06 | 0.329 | 1.01* | <0.08 | 24.4* | <0.002 | 0.007 | 0.0004 | 1.0 | 10 | <3.0 |
| S12 | <0.006 | 0.130 | 0.0269 | 0.009 | 0.229 | <0.002 | <0.002 | <0.0001 | 0.28 | 16.5 | <4.0 |
| S13 | <0.006 | 0.836 | <0.01 | <0.008 | <0.7 | <0.002 | <0.002 | <0.0001 | 0.2 | 30.7 | <3.0 |
| S14 | <0.006 | 0.715 | 3.13* | <0.08 | 11.3* | <0.002 | 0.025 | 0.0036 | 2.0 | 2.2 | 2.0 |
| S15 | | | | | | <0.002 | <0.002 | | | | |

*Concentration over maximum contaminant level.

**INSPECTION REPORT B: GRITTY WRECKING**

December 2, 1987

NESHAP Asbestos Demolition Inspection -- Gritty Wrecking,
Urban, Michigan (A24535:00)

K. Eagle, Environmental Engineer
THRU: J. Medium

Engineering Section 5AC
ATTB: S. Boss

This is the first in a series of inspection reports intended to provide a complete discussion of a NESHAP inspection (asbestos removal) at the former Consolation Company ("the facility") located at 1521 East First St., in Smallerville, MI. The inspection was conducted on October 22, 23, 26-30, and November 2, 1987. An initial inspection of demolition occurring at the facility was conducted on October 22 and 23, 1987. On October 26-27, additional visits were made to the site to obtain measurements of the amount of suspected friable asbestos containing material (FACM) still in the facility and to obtain correct information as to which buildings were involved in the demolition project. Mr. Linus Lip of the EDO was on site October 29, 30 and November 2, 1987, to oversee the entire asbestos abatement and to observe deposition of the ACM waste after removal.

Background information about the facility, notification, amount of asbestos present, work practices, worker safety and equipment, and waste handling at the facility are discussed in this report. Actual ACM removal by an asbestos abatement contractor occurred October 29, 30, and November 2, 1987, and all discussion of work practices, worker safety and equipment, amount of asbestos removed, waste handling at the facility and waste transport and disposal by the abatement contractor will be forwarded in a second report by Mr. Lip. Information about samples taken, sample analyses, and pictures of the site is provided in the attachments (Attachment 4 - Summary of Samples Taken; Attachment 5 - Sample Analyses from Laboratory; Attachment 6 - Pictures of Site).

**Background**

It is estimated that the facility was constructed in the early 1900s. The entire complex includes approximately 35 buildings and encompasses 365 acres. The portion of the facility inspected included buildings 2 through 8 at the west end of the complex, located at the corner of East First and East Front Streets (Attachment 1 - Diagram of Complex). East of the facility is an industrial area and approximately one quarter mile to the west begins a residential neighborhood. Downtown Smallerville is located approximately one mile west of the facility. The city of Smallerville owns the complex and was contracting out groups of buildings for demolition.

On the morning of October 22, 1987, a call was made to the EDO from a contractor who had bid on this particular job but did not win the contract. The contractor stated that he had bid $90,000 to do the project, which would have included the asbestos removal necessary. He continued that Gritty Wrecking of Urban, MI, has won the contract with the city with a bid of only $24,000. He said the buildings were "full of asbestos" and that the project could not possibly be completed properly at that low a cost. I left that afternoon to inspect the site in Smallerville for possible noncompliance with NESHAP regulations regarding asbestos removal prior to demolition.

**Notification**

Attached is the building permit (Attachment 2) obtained from Michael Edifice, Director of Building and Zoning for the city of Smallerville, stating the buildings to be demolished by Gritty Wrecking per the contract between Gritty Wrecking and the city of Smallerville (Attachment 3), and including the terms for the handling of asbestos in the subject buildings.

No notification of demolition or intent to remove asbestos was submitted to any appropriate Michigan agency or the federal government by Gritty Wrecking prior to beginning work.

***Amount of Asbestos Present***

Upon my initial inspection on October 22, 1987, I observed approximately 45-50 linear feet of pipe lagged with dry, suspected FACM in Building 2. In addition, I observed large amounts of dry, suspected FACM lagging and debris in the rubble below pipes in the same building. On October 23, 1987, I returned to the site and entered Building 2 with P. Gradey, Superintendent for Gritty Wrecking, to observe one of Gritty's employees removing asbestos. Inside I observed approximately 25 linear feet of suspected ACM in the immediate area. Mr. Lip and I returned on October 26, 1987, and entered what was left of buildings 2 through 7. Within these buildings we measured an additional 230 linear feet of suspected FACM lagging. There also was an open labeled asbestos waste bag filled with dry pipe lagging in Building 3, and dry, suspected FACM lagging and debris on the floors, walls, and fixtures in the buildings. We also observed seven bags of pipe lagging and three bags of a dry, suspected FACM sheet material outside against a fence on the site. The asbestos abatement contractor (Scrub Abatement) later estimated that there was at least 100 linear feet of lagging in those bags. When we entered the building again on October 28, 1987, we found an additional 8 feet of pipe lagged with suspected FACM in Building 8.

Scrub Abatement had later been contracted by Gritty Wrecking to properly remove all of the asbestos in the buildings involved in the demolition project. Their notice of intent to remove asbestos stated that 397 linear feet had been found which did not include the material in Building 2 noted previously since most of that building had been demolished prior to Scrub's assessment.

**Inspection**

Thursday, October 22, 1987
As stated previously, a call was made to the EDO on the morning of October 22, 1987, regarding the possibility that violations of the NESHAP, specifically asbestos removal, might have been occurring.

Acting on the information obtained, I went to Smallerville, Michigan, to inspect the facility in question. I arrived at the site at 1615 EDT on October 22, 1987, and found no demolition occurring although there was a front-end loader there. I immediately observed pipes with dry, suspected FACM lagging in the partially demolished Building 2. As I walked closer to Building 2, I observed large amounts of white, friable, suspected ACM in the rubble. I estimated that 45-50 feet of pipe contained suspected FACM lagging in part of the Building 2 that was visible from the outside.

Five samples were taken from the material in the rubble and still on pipes (88EH01S01-S05), and four were analyzed as positive for asbestos (Attachment 5). Several pictures were taken of the building and the suspected FACM (Attachments 4, 5, and 6).

Friday, October 23, 1987
On October 23, 1987, at approximately 0915 EDT, I returned to the site to see if there was, in fact, demolition in progress. I observed a man operating a front-end loader, knocking down Building 2. I also observed a second man using a torch to cut pipes in the same building. As the front-end loader was knocking down Building 2, I observed visible emissions. Sample 88EH01S06 was taken here later. ~~Shortly after, the loader operator left Building 2 and~~ proceeded to begin demolition on Building 4A.

I entered the site at 1140 EDT and spoke with the loader operator. He informed me that there was a man inside the building at that time removing asbestos from the pipes, but there was no foreman on the site at that time and he did not know when he'd return. I looked into the open end of Building 2 and saw a man using a torch but I did not enter at that time. I inspected the area of Building 2 where I had seen the loader working and found much more pipe lagging in the rubble there than on October 22, but no more lagging on the pipes above, which I had observed on those pipes the previous afternoon. I concluded it had been knocked down during demolition.

A short time later, the man that had been inside emerged from Building 2 wearing brown coveralls. There was no sign of a respirator. He introduced himself as Ernie McDoogle and produced his certification paper for asbestos handling in Michigan. He informed me that he was not removing asbestos but rather cutting down the pipes containing suspected ACM, which were to be disposed of in sections, pipe and lagging all together. He stripped off his coveralls, coated with white dust, hung them over the back of his truck, and prepared for lunch.

I sampled the material (88EH01S06) where I had previously seen the visible emissions; it was later found by the Central Regional Laboratory to contain 25%-35% amosite (Attachment 5). No foreman returned to the site and I left at 1300 EDT.

I returned to the site at approximately 1430 EDT, and spoke with Paul Gradey, Superintendent from Gritty Wrecking. He informed me that Ernie McDoogle was inside removing asbestos. I asked him what would be done about all of the pipe lagging laying in and around the rubble of Building 2 and he said he did not know about that. I asked him where the bags of asbestos that were in the back of his pickup truck were going and he said "in the river." Then he said they would be taken to Gritty's shop in Urban until they had a full load to transport to Payne Disposal in Oldville, MI. He asked me if I wanted to see the removal in progress inside and I followed him into Building 2. Inside I observed Mr. McDoogle removing suspected asbestos from piles (contrary to what he'd told me) wearing his brown coveralls, gloves, and dust mask. He had the pipes laying on the floor. He sliced open the dry lagging with a knife, peeled the two halves off of the pipe, and stuffed them into a labeled asbestos waste bag. He was not wetting the material and when I asked why, he said it was "wet enough." I observed visible emissions when he removed the lagging from the pipe, but I did not sample the material. In that immediate area, I observed approximately 25 feet of suspected ACM on pipes. I asked Mr. McDoogle if he had learned about wetting the ACM, the glove bag technique, and protective equipment worn during asbestos handling in his training course and he said yes. I left Building 2. I spoke more with Mr. Gradey outside and a short time later I left the site.

The five samples I obtained on Thursday, October 22 and the sample obtained on October 23, from where visible emissions were observed during demolition, were express mailed to the Central Regional laboratory in Chicago at approximately 1630 EDT on Friday, October 23, 1987. As indicated previously, analytical results are included in Attachment 5.

<u>Monday, October 26, 1987</u>
On October 26, 1987, at 1320 EST, Linus Lip of the EDO and I returned to the site. It was apparent that a considerable amount of demolition work had occurred between Friday (October 23) evening and Monday (October 26) morning despite Mr. Edifice's order to stop, because a large portion of Building 2 had been leveled. No one was on site but a claw was present beside the front-end loader which was at the site on Friday.

Mr. Lip and I proceeded to enter the facility. We entered through Building 2 and there we observed a 102-foot pipe that appeared to have recently had the suspected ACM removed. There were thread-like pieces of white material hanging from the pipe and pieces of dry, suspected ACM hanging on the wall and laying on the floor below the pipe. A sample was taken (88EH01S07) and confirmed to be 25%-35% amosite and 1%-5% tremolite-actinolite (Attachment 5). We soon found another 13 foot piece of pipe that matched the cut of the 102 foot pipe. We found four feet of pipe, with lagging, laying on the floor in a small room marked "Bathroom," but could not discern the area from which it had fallen.

We continued into the facility, into Building 3. In Building 3, we found what appeared to be a type of printing unit with suspected ACM-lagged pipes running from it and around it. There also were two vessels in that area wrapped with asbestos insulation (sample 88EH01S08). An open marked bag, containing dry, friable asbestos lagging (sample 88EH01S10A) sat near the printer. On a catwalk that ran along the east wall of Building 3, there was an asbestos sludge (sample 88EH01S09) that apparently had dumped off of the pipes above. Mr. Lip and I measured 197 linear feet of pipe lagging in Building 3. We exited the building and observed many more areas where suspected ACM lay in the rubble. Against the fence on the west end of the site, we observed ten marked clear, asbestos bags; two of them were open and they were accessible to the public. Seven of the bags contained pipe lagging and three contained chunks of dry sheet (sample 88EH01S11) about one-quarter inch thick. We did not find any more of the sheet material inside the building, and left the site.

At 1620 Mr. Lip and I returned to the site to obtain samples of the materials described above (Attachment 4 - Summary). We left the site at 1730 EST.

<u>Wednesday, October 28, 1987</u>
On October 28, 1987, at approximately 0800 EST, Linus Lip, Joe Lawstruck of the Office of Regional Counsel, and I returned to the site on the corner of East First and East Front Streets. We entered the facility through Building 2 to re-measure the amount of suspected FACM contained in all of the buildings (2-8) that Gritty Wrecking was contracted to demolish. Including the 197 feet Mr. Lip and I previously observed, we measured 286 linear feet of suspected ACM pipe lagging.

At 1030, Mr. Lip and I met with Mr. Lawstruck and Caroline Bernoose of the Air Compliance Branch; Kenneth Chalk, Vice President of Operations for Gritty Wrecking and his lawyer Frank Gradey; Mr. Edifice, Building Director for the city of Smallerville; the city of Smallerville's lawyer, Oliver Twist; and the Assistant U.S. Attorney, Harry Marvel, at the U.S. Attorney's office in Urban.

At the meeting, all parties discussed potential violation of the NESHAP that occurred at the demolition site and recommendations for the immediate correction of and compliance with NESHAP regulations governing asbestos removal as it applied to this demolition.

Mr. Chalk agreed to contact Scrub Abatement, an asbestos abatement contractor, to begin removal the following morning (October 29, 1987) of all ACM in the buildings concerned. Mr. Lawstruck, Mr. Lip, and I agreed under the condition that Mr. Lip or I were present throughout the ACM removal and disposal.

At 1530 EST, Larry Lip spoke with Mr. Chalk and confirmed that Scrub Abatement would arrive on site at 0730 on October 29 to assess the abatement job and begin removal of the ACM. Mr. Lip agreed to be present on site for the entire ACM removal period which occurred on October 29, 30 and November 2, 1987. The waste was transported to a landfill at 1530 EST on November 2, 1987.

A subsequent report will follow from Mr. Lip describing the actual amount of asbestos removed, work practices, worker safety and equipment, waste handling at the facility, waste pickup, and waste transport and disposal at the landfill. Also, analyses of samples taken during ACM removal, and the field data collection checklists, will follow in Mr. Lip's report.

ENVIRONMENTAL PROTECTION AGENCY
FOR THE [illegible]

DIVISION/BRANCH ESD/EDO SAMPLING DATE 10/26/87 LAB ARRIVAL DATE 10-28-87 DUE DATE 11-7-87/ASAP ✕

[illegible] NUMBER A306 DATASET NUMBER 4480 STUDY METRO WRECKING E. FIRST & E. FROST STS. PRIORITY #1 CONTRACTOR CRL

| CRL LOG NUMBER 88EH01 | SAMPLE DESCRIPTION | ANALYTICAL REQ'D PTS ASBESTOS UG/L 01020137 | TAG. NO: |
|---|---|---|---|
| S07 | HANGING ON WALL & ON FLOOR INSIDE BLDG | ✓ [illegible] Tremolite | 5-30783 |
| S08 | LARGE VESSEL | ✓ [illegible] 10-15 Chrysotile | 5-30782 |
| S09 | SLUMPED ON CATWALK INSIDE BLDG. | ✓ 35 PCT. Amosite | 5-30784 |
| S10A | OPEN BAG INSIDE BLDG. NEAR PRINTING UNIT - [illegible] | ✓ [illegible] | 5-30785 |
| S11 | OPEN [illegible] OUTSIDE BLDG SHEET MATERIAL | ✓ [illegible] PCT. [illegible] | 5-30786 |
| S10B | OPEN BAG INSIDE BLDG NEAR PRINTING UNIT. DRY | ✓ Not Analyzed | 5-30787 SG * |

11/24/87

* User did not have this sample on the request form. I took the information from the Chain-of-Custody Sheet.
Sylvia Griffin

Form

HANDOUT 14-28

# Summary of Samples Taken

| SAMPLE NUMBER | TIME | SAMPLE DESCRIPTION | SAMPLE ANALYSIS RESULTS |
|---|---|---|---|
| OCTOBER 22, 1987 | | | |
| 88EH01S01 | 1647 EDT | On red bricks | 33%-40% Amosite |
| S02 | 1655 EDT | On pipe | 32%-35% Amosite;<br>8%-12% Tremolite-Actinolite |
| S03 | 1705 EDT | On boards in rubble | 20%-25% Amosite;<br>5%-10% Tremolite-Actinolite |
| S04 | 1730 EDT | In rubble | 35%-40% Amosite |
| S05 | 1736 EDT | Brown fluffy | Negative |
| OCTOBER 23, 1987 | | | |
| 88EH01S06 | 1210 EDT | Where visible emissions observed during demolition | 25%-35% Amosite |
| OCTOBER 26, 1987 | | | |
| 88EH01S07 | 1330 EST | Hanging on wall and on floor in building 2 | 25%-35% Amosite;<br>1%- 5% Tremolite-Actinolite |
| S08 | 1343 EST | Large vessel | 5%-10% Amosite;<br>10%-15% Chrysotile |
| S09 | 1630 EST | Slumped on catwalk in building 3 | 25%-30% Amosite |
| S10A | 1640 EST | Open bag in building 3 | 15%-25% Amosite |
| S10B | 1640 EST | Open bag in building 3 | Not Analyzed-Dry |
| S11 | 1710 EST | Open bag outside - sheet material | 15%-20% Amosite;<br>1%- 5% Chrysotile |

# Summary of Pictures Taken

**October 22, 1987:**

EDT

| | | |
|---|---|---|
| Picture 1 | 1647 | On bricks, outside Building 2, Sample S01. |
| Picture 2 | 1647 | Outside Building 2, in rubble. |
| Picture 3 | 1655 | Pipe lagging in Building 2, Sample S02. |
| Picture 4 | 1705 | On boards, in rubble, in Building 2, Sample S03 |
| Picture 5. | 1710 | In rubble, in Building 2. |
| Picture 6 & 7 | 1712 | On ground in Building 2. |
| Picture 8 | 1715 | Pipe lagging in Building 2. |
| Picture 9 & 10 | 1720 | Building 2. |
| Picture 11 | 1730 | On ground in Building 2, Sample S04. |
| Picture 12 & 13 | 1732 | Pipes in Building 2. |
| Picture 14 - 21 | 1735-1815 | Rubble in and around Building 2. |
| Picture 22 | 1820 | View of demolition site from street. |

**October 23, 1987:**

EDT

| | | |
|---|---|---|
| Picture 23 & 24 | 1210 | Building 2, where visible emissions were observed during demolition, Sample S06. |
| Picture 25 | 1515 | Truck driven by Paul Garvaglia, asbestos bags in back. |

**October 26, 1987:**

EST

| | | |
|---|---|---|
| Picture 26 - 31 | 1320 | Appearance of Building 2 after the weekend. |
| Picture 32 & 33 | 1330 | Inside Building 2, Sample S07. |
| Picture 34 | 1335 | Inside Building 3. |
| Picture 35 | 1343 | Large vessel inside Building 3, Sample S08. |
| Picture 36 & 37 | 1345 | Pipes in Building 3. |
| Picture 38 | 1400 | Rubble outside Building 2. |
| Picture 39 & 40 | 1405 | Outside Building 5A. |
| Picture 41 - 45 | 1410 | Bags outside, against fence, Sample S11. |
| Picture 46 | 1630 | ACM slumped on catwalk in Building 3, Sample S09. |
| Picture 47 & 48 | 1640 | Open bag of dry FACM in Building 3, Sample S10. |
| Picture 49 | 1655 | Pallets in Building 5. |

15

## SESSION 15

## TOPIC: ENFORCEMENT PROCESS RESPONSIBILITIES

**Time: 60 minutes**

**PURPOSE**

- Describe what to expect when appearing as a witness
- Present tips for testifying at a deposition or trial
- Demonstrate questioning, examination, and cross-examination
- Discuss role of inspectors in settlement and other negotiations
- Provide tips for effective negotiations

**KEY POINTS**

- Prepare well in advance. Don't volunteer information
- Prepare carefully, know all the facts and options Resolve differences internally, not in front of the other side
- Understand negotiation; most cases are settled through negotiation, not trials

**LIST OF VISUALS**

| | |
|---|---|
| 15-1 | Enforcement Process Responsibilities (Title Slide) |
| 15-2 | Trial/Hearing or Deposition |
| 15-3 | Common Misconceptions |
| 15-4 | The Legal Framework |
| 15-5 | Our Adversary System |
| 15-6 | Discovery Rules |
| 15-7 | Rules of Evidence |
| 15-8 | Hearsay |
| 15-9 | Keys to Effective Testimony |
| 15-10 | Documenting Investigative Activities |
| 15-11 | Role of the Inspector/Witness |
| 15-12 | Expert Witness |
| 15-13 and 14 | Preparation for Testimony |
| 15-15 | Depositions |
| 15-16 | Depositions/Setting |
| 15-17 to 19 | Hearings/Trials |
| 15-20 | Trick Questions |
| 15-21 and 22 | Universal Witness Guidelines |
| 15-23 | Negotiations |
| 15-24 | Preparation for Negotiations· Role of the Inspector |
| 15-25 | Negotiation Fundamentals |
| 15-26 and 27 | Supplemental Environmental Projects |
| 15-28 | Categories of SEPs |
| 15-29 | Unallowable SEPs |

## Enforcement Process Responsibilities

Trial/Hearing or Deposition Testimony

Negotiations

Supplemental Environmental Projects (SEPs)

15-1

## Trial/Hearing or Deposition Testimony

- Common Misconceptions
- The Legal Framework
- Keys to Effective Testimony
- Depositions/Trials/Hearings

15-2

## Common Misconceptions

- "This case will never go to trial, so why worry about the details"
- "I know what I'm testifying on, so I don't need to prepare"
- "If I get into trouble, my lawyer will protect me"

15-3

## The Legal Framework

- Adversary System of Justice
- Discovery Rules
- Rules of Evidence
- Burden of Proof

15-4

## Our Adversary System

- Investigation
- Complaint/Indictment
- Discovery
- Trial/Hearing

15-5

## Discovery Rules

- Civil Judicial Cases
  - Documents
  - Interrogatories
  - Expert reports
  - Depositions
- Civil Administrative Cases
- Criminal cases
  - Rule 16/Jencks Act
  - Brady Rule

15-6

## Rules of Evidence

- All Evidence Must Be
  - Relevant
  - Competent
  - Authentic
- Expert Opinions Must Be
  - Relevant
  - Reliable

15-7

## "Hearsay"

- Out of court statement offered in court to prove the truth of the matter asserted
- General rule hearsay is not admissible.
- Numerous exceptions allow for admission of reliable hearsay.
- Exceptions may apply to scientific data, laboratory reports and public records.

15-8

## Keys to Effective Testimony

- From outset, fully understand case and role
- *Carefully document work* during investigation
- Know limits of expertise
- Prepare for testimony

15-9

## Documenting Investigative Activities

- Assume you will need to reconstruct events at trial--<u>from first day</u> on case
- As appropriate, include detailed information on facility operations and processes
- Fully describe protocols used, field measurements, sampling and equipment

## Role of the Inspector/Witness

- Fact witness
- "Summary" witness
- Expert witness
- Consultant

## Expert Witnesses

- Experts qualified based upon knowledge, skill, experience, training or education
- Testimony permitted if it would be <u>helpful</u> to trier of fact
- Proposed rules require that expert provide opinions on scientific or technical matters

## Preparation for Testimony

- Work closely with counsel
- Review pertinent records
  - Government files
  - Discovery from defendant
- Understand case
  - Theory of case/ elements of proof
  - Your role in proving elements
  - Weaknesses in evidence

15-13

## Preparation for Testimony-2

- Understand nature of proceedings
  - Deposition
  - Trial/Hearing
- Know audience/understand impact of demeanor
- Be familiar with exhibits
- Rehearse, but **do not memorize** testimony

15-14

## Depositions

- **Purpose of Deposition**
  - Obtain explanation of basis for case
  - Discover expert opinions
  - Prepare for cross-examination
- **Legal Standards**
  - "[R]easonably calculated to lead to the discovery of admissible evidence"
  - Privileges/instructions not to answer
  - Depositions taken subject to objections

15-15

## Depositions /Setting

- Conference room
- Court reporter/no judge
- Counsel control conduct of deposition
- Witness can confer with counsel
- Testimony is under oath

15-16

## Hearings/Trials

- **In general**
  - First moment "an out of body experience"
  - relax, listen to questions carefully
  - Simplify presentation/translate into plain English
  - use visual aids /refer to documents
- **Direct examination (government's case)**
  - Witness qualifications/expertise
  - Description of work
  - Opinion testimony (if an expert)

15-17

## Hearings/Trials-2

- **Cross-examination--purpose**
  - Impeach/discredit
  - Expose inconsistencies/weaknesses
  - Limit effect of direct testimony
- **Handling Cross-Examination**
  - Know case (both sides)
  - Expect convoluted/leading questions
  - Expect disruptive antics by counsel
  - Listen carefully/ stay calm
  - DO NOT VOLUNTEER!
  - Admit lack of knowledge

15-18

## Hearings/Trials-3

- **Objective is to show that:**
  - Personnel were qualified
  - Chain-of-custody was intact
  - Appropriate sampling/analytical methods were used
  - Data obtained were reliable
  - Expert opinions were reliable

15-19

## Trick questions

- Is there "anything else" or "is that all?"
- Did you talk with your attorney (i e the government's attorney) before testifying?
- Have you ever made a mistake?
- Isn't it possible that .. ?

15-20

## Universal Witness Guidelines

- Tell the truth
- Listen carefully
- DO NOT VOLUNTEER
- Do not answer convoluted /ambiguous questions

15-21

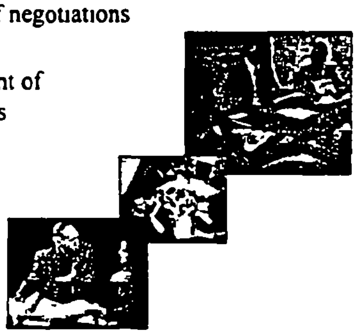
## Universal Witness Guidelines-2

- Do not speculate
- Be wary of opposing counsel's restatement of testimony
- Use words not gestures
- Remain calm and courteous

15-22

## Negotiations

- Purposes of negotiations
- Management of negotiations



15-23

## Preparation for Negotiations: Role of the Inspector

- Complete investigation, collect information
- Assist in the analysis of the information
- Participate in internal negotiations
- Assist in developing strategy and credible proposals

15-24

## Negotiation Fundamentals

- Plan roles and approach
- Plan for caucuses/external communications
- Have inspector attend negotiations

15-25

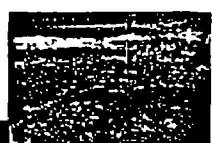
## Supplemental Environmental Projects

- To further EPA's goals to protect and enhance public health and the environment
- To obtain protection that might not occur otherwise

15-26

## Supplemental Environmental Projects

- Pollution prevention
- Environmental justice



15-27

## Categories of SEPs

- Public health
- Pollution prevention
- Pollution reduction
- Environmental restoration and protection
- Assessments and audits
- Environmental compliance promotion
- Emergency planning and preparedness

15-28

## Unallowable SEPs

- General public education
- University research
- Projects unrelated to environmental protection
- Studies without commitment
- Projects funded by low-interest federal loans or grants

15-29

## THE CASE OF THE UNMANAGED NEGOTIATION

Sam Acosta has just become acting chief of the RCRA Enforcement Section. Previously, he was in the RCRA State Programs Section. He finds a short action memorandum on his desk from Bill Sanders, a RCRA inspector. The memorandum recommends that EPA issue an administrative order to a hazardous waste treatment facility called Treatment Supreme (TS) for violations of interim status requirements related to security of the site and manifests. Sanders wrote that during an inspection he observed that a 25-foot segment of fencing along a highway at the back of the TS facility was missing. He also noted that TS had failed to note discrepancies on manifests for 75 shipments of one waste stream, analysis of which performed by TS did not confirm that the waste was as represented by the generator. He attached a copy of the draft administrative order requiring restoration of the fence, prohibiting further receipt of the waste stream, and assessing a $25,000 penalty. Sanders noted that copies of the manifests and laboratory reports were in the inspection file. Sanders has been detailed under an Intergovernmental Personnel Agreement (IPA) to the state for a year.

Acosta signs off on the action memorandum and sends it to his boss. It eventually is sent to the Regional Counsel's office for legal review and is assigned to Laura Smith. Smith's main job at EPA has been to handle the legal aspects of construction grants for sewerage treatment facilities. This is her first enforcement case. She is instructed that the program office is responsible for substantive determinations and her role is to ensure the order is legally sustainable, to assist the program office in any resultant negotiations, and to represent the program office in any subsequent appeals. She reviews the order and action memorandum and determines that the violations alleged are sufficient to support the remedies sought and are supported in the action memorandum. She compares the draft order with agency guidance and makes some changes to conform it to the guidance. She signs off on the order, and it is eventually issued.

Guy Larado, attorney for TS, calls Smith to request a conference on the order; he hopes to negotiate a mutually acceptable resolution. Smith indicates she must check Acosta's calendar; they arrange three possible times, depending on Acosta's availability. She calls Acosta, settles on a date three weeks hence, and makes arrangements to meet with Acosta that afternoon to review the case.

When Smith and Acosta meet, they review the action memorandum and order. Smith asks to see copies of Sander's inspection report and the manifests at issue. She asks whether they can talk to Sanders, but Sam says he has been detailed under an IPA to the state. They agree that the case seems open and shut and that, under EPA's penalty guidance, they can agree to mitigate the penalty only to $18,000. They agree that Smith will be the spokesperson in the negotiations. They tell both of their superiors that they intend to settle for the substantive relief set forth in the order and a penalty of from $18,000 to $25,000. Their supervisors concur.

As the date of the meeting approaches, Smith attempts to meet again with Acosta, but, because they are both out of the office much of the time, they do not connect. The day before the meeting, Smith attempts to arrange for a conference room, but they already have been claimed. Instead, she arranges to meet in Acosta's office, which is larger than hers.

On the date of the meeting, Smith goes to Acosta's office five minutes before the meeting, telling the receptionist to ring her there when Larado and TS arrive. Larado, however, is familiar with the EPA office and proceeds directly to Smith's office, never coming near the receptionist. Smith's secretary is not there, and no one knows where she is. Both negotiating teams remain in splendid isolation until Smith's secretary returns, discovers the situation, and calls Smith. Smith returns to her office, meets the TS group -- the plant manager, chief chemist, staff attorney, consulting attorney, and customer's plant manager and attorney -- and escorts them to Acosta's office. There are only four chairs in Acosta's office. Acosta and Smith scurry around to find four more chairs. Acosta sits at the desk, Smith sits beside it, and the TS group crowds in front of the desk, filling all the space between it and the door. The room is not large enough to hold them all comfortably.

Smith opens the meeting by introducing herself and Acosta and inviting the TS group to do the same. She apologizes for the confusion and for the cramped quarters. She then outlines the violations alleged and the enforcement procedures. She emphasizes EPA's view of the importance and gravity of the violations and states that EPA would like to determine whether there is a basis for settling the matter. She indicates the substantive violations must be corrected expeditiously. Finally, she states that EPA "really would like to get a penalty of around $18,000 to $20,000."

Guy Larado, the consulting attorney for TS, opens his argument by stating that TS explained both situations to the EPA inspector when he was on site and that TS believes the complaint is a mistake. He asks whether the inspector is coming to the meeting. Acosta says the inspector has been detailed under an IPA to the state and is not available. Larado expresses dismay, since TS already has been through the issue with the inspector.

Larado then said there was indeed a 25-foot section of fence missing the day the inspector was there, as a result of an automobile accident on the highway. The fence was scheduled for repair within the week and, in fact, was repaired two days later. The fence, incidentally, was a 10-foot-high, electrified, chain-link fence, topped with concertina wire, a far more protective fence than was required or was customary in the trade. He produced pictures of the fence; a notarized affidavit from the repair company stating when it was repaired; and a copy of a letter to the inspector, with copies of the pictures and affidavit. Acosta stated he was satisfied that the violation had been corrected. Smith said that a penalty might be authorized legally, but that, equitably, it should be mitigated to zero, since the hole in the fence had been caused by a third party beyond the control of TS. TS had scheduled its repair before the inspection and had repaired the fence immediately after the inspection, and the fence was far better than required by EPA's regulations. Acosta then said, "Let's talk about the manifest violation."

Larado said TS also had discussed that issue with the inspector. The waste stream in question was being delisted when the inspection took place and subsequently was delisted, so it was not a hazardous waste at all. TS's customer produced a copy of the delisting document and the accompanying Federal Register notice. Larado said that TS had written the inspector, enclosing a copy of the delisting document and notice. Smith asked Acosta whether copies of TS's letters to the inspector were in his files, because there were none in hers. Acosta answered that he did not know, but looked through his file and found both letters.

Larado said that TS could argue that, since the waste stream had been delisted by EPA, EPA acknowledged that it was never really hazardous waste, and, therefore, TS never really violated the manifest requirements. He said TS would forgo that argument for the sake of settlement, if EPA would acknowledge that, because the waste stream was not hazardous, the violations were technical and there was no damage done to the environment or the regulatory scheme and a de minimis penalty, if any, was appropriate. He offered $2,500. Acosta said that settlement was acceptable but that the violations found raised a question about the integrity of TS's system for handling manifests and its waste analysis plan. Larado answered that EPA's inspector had found no other problems, but TS would hire an auditor to review its system and would follow the auditor's recommendations if defects were found. Acosta asked whether TS would agree to put a requirement for that action in a consent order, and Larado agreed. Larado said that there appeared to be agreement: TS would settle for a $2,500 penalty and an agreement to audit TS's manifest system and correct any deficiencies. At that point, Smith said she thought EPA's penalty policy would require more than $2,500 for the admitted violations. Larado said Acosta already had agreed to the $2,500 figure. Acosta said he had not agreed to the figure, and that he had agreed only that, because of the facts, a relatively low penalty seemed appropriate. Larado asked how much, and Acosta asked Smith whether she thought $5,000 would be enough. She said she did not know. Larado said TS would write a check for $4,000 and deliver it immediately to settle the matter. Acosta said he did not think a settlement could be reached so quickly, since it would require concurrence of senior staff. That process usually took at least two weeks, he said.

Larado, who, until this point, had been soft-spoken, polite, and charming, became red in the face and began speaking in a louder voice, touched with anger. He protested that he had spoken at length with the inspector about the importance of a quick resolution of the matter. TS was about to close major financing to construct three new state-of-the-art incinerators in another EPA region and was required to certify a clean regulatory bill of health to secure the financing. He said the inspector had assured him that, if EPA's negotiators could sign off on a settlement, the matter could be handled in a matter of days. Larado said he was dumbfounded that EPA would hold up so important a matter when it agreed the violations were trivial and of no consequence.

Acosta asked Smith whether she saw any reason not to agree to the settlement outlined. She said she had not seen enough of the problems to be sure. Acosta said that, as far as he was concerned, the violations, as explained, were technical; the solutions were adequate; and the penalty appropriate. Smith said he was the client and if he was satisfied, she was. Larado then drew up a letter of agreement, which both parties initialed, and Smith agreed to turn into a consent order that afternoon. That afternoon, Smith talked to Sanders, the inspector, by phone. He confirmed that he had indicated the possibility of quick action if agreement was reached, but said the agreement was inappropriate. The break in the fence indeed had been caused by an automobile accident and had been repaired immediately after the inspection. But the break had occurred four months earlier, and the repair was not ordered until after TS knew an inspection had been scheduled. The fence was indeed far better than those around most disposal facilities. But installation of the fence had been ordered by the state after previous fencing had proven inadequate to prevent repeated damage by vandals. TS was correct that the waste stream involved in the manifest violations had been delisted. But the real question was whether the shipments received really were of that waste stream, or whether TS had been accepting a nonpermitted waste. Indeed, Sanders wondered whether he had made a mistake in not recommending action against TS's customer for sending a waste to a disposal facility not permitted to take the waste. He was surprised that Smith was unaware of those facts, because most of the information was in the handwritten notes that he was sure were in the file somewhere.

At this point, EPA's negotiating team recognized that it was in an embarrassing situation.

# CRUSH AND DESTROY NEGOTIATION

## I. General Instructions

The facts presented below are based on actual EPA cases, but the information has been modified and supplemented to facilitate this exercise. Participants should use only the information provided in this fact sheet, along with their knowledge of the Clean Air Act and EPA regulations. While participants may have only limited specific knowledge of the requirements, they can use the general principles of negotiation to develop a strategy. Logical inferences may be made from the facts. The objective of this exercise is to reach agreement on a plan for conducting a negotiation with the defendant in the case described below. In developing the strategy, consider:

- What items are negotiable and nonnegotiable for EPA
- Strengths and potential weaknesses in EPA's position
- The role (if any) of the state
- Options for specific remedial steps to be required
- Factors that could be considered in adjusting the amount of the penalty

## II. Facts

### A. The Company

Crush and Destroy, Inc. (C&D) demolishes industrial and commercial structures. It has been in business about 20 years, operating in the state of Maryland. C&D is a closely held, family-operated business that employs 10 people. C&D's gross revenues are approximately $400,000 per year, and its assets are slightly less than $100,000. Two years ago, the company earned $40,000. Last year, C&D lost $20,000.

### B. The Violations

Several months ago, C&D was demolishing sections of an apartment building. In doing so, it uncovered friable (crumbly) asbestos material. This fact became known to the tenants of the standing portions of the building who, concerned about the well-publicized effects of asbestos, contacted EPA. Several days later, EPA sent an inspector to the site. The inspector observed C&D's operation and noted several violations. First, C&D workers threw dry asbestos waste material onto the back of an open truck and transported it to a local landfill, where they dumped the material. These actions violated 40 CFR 61.147(e), which requires that asbestos waste material be kept wet until it is collected for disposal; and 40 CFR 61.152(b), which requires that asbestos waste material be properly contained, transported, and disposed of. Finally, in violation of 40 CFR 61.146, C&D failed to notify EPA in advance of its demolition work at the apartment building.

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## SESSION 16

## TOPIC: MOCK TRIAL

**Time: 45 minutes**

| | | |
|---|---|---|
| **PURPOSE** | • | Introduce participant to the realities of what can result from their inspection. |
| **KEY POINTS** | • | Inspectors must be accurate and thorough in their inspections and follow-up record keeping. |
| **LIST OF VISUALS** | 16-1 Mock Trial (Title Slide) | |

## Mock Trial

**SESSION 17**

**TOPIC: AGENCY/REGENCY INITIATIVES**

**Time: 60 minutes**

**PURPOSE**

- Allow the region or host organization to address EPA or Regional initiatives not addressed elsewhere in the course

Agency/Regency Initiatives

PS-2

18

**SESSION 18**

**TOPIC: INTRODUCTION TO CRIMINAL INVESTIGATIONS**

**Time: 180 minutes**

**PURPOSE**

- Introduce inspectors to criminal enforcement and explain how to recognize potential criminal violations.
- Provide an overview of criminal litigation

## Introduction to Criminal Investigations

## Principal Differences Between Civil and Criminal Enforcement

- Warrants based on "probable cause"
- Other constitutional guarantees
- Burden of proof: "beyond a reasonable doubt"
- More severe penalties: imprisonment or fine

## "Red Flags" That Indicate Possible Criminal Activity

- Conflicting data
- Conflicting stories
- Unsubstantiated data
- Deliberate actions
- Claims of ignorance about requirements

## SESSION 19

## TOPIC: WRAP-UP AND EVALUATION

**Time: 60 minutes**

| | |
|---|---|
| **PURPOSE** | • Answer any questions outstanding and to obtain an evaluation of the overall course. |
| **KEY POINTS** | • Students and instructors will identify any key points or areas that should be addressed. |
| **LIST OF VISUALS** | • 19-1 Wrap-Up and Evaluation (Title Slide) |
| **LIST OF HANDOUTS** | • Course Evaluation |

**Wrap-Up and Evaluation**

NOT USED

Method 1624 Revision B

# VOLATILE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

Method 1625 Revision B

# SEMIVOLATILE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

EPA

Office of Water Regulations and Standards
Industrial Technology Division

U.S. ENVIRONMENTAL PROTECTION AGENCY WASHINGTON, D.C. 20460

# Method 1624 Revision B
# VOLATILE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

## 1 SCOPE AND APPLICATION

1 1 This method is designed to determine the volatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to purge and trap gas chromatography-mass spectrometry (GC/MS)

1 2 The chemical compounds listed in table 1 may be determined in municipal and industrial discharges by this method The method is designed to meet the survey requirements of EPA's Industrial Technology Division (ITD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR 136 1 and 136 5 Any modifications 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 3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations The limits in table 2 represent the minimum quantity that can be detected with no interferences present

1 4 The GC/MS portions of this method are for use only by analysts experienced with GC/MS or under the close supervision of such qualified persons Laboratories unfamiliar with the analyses of environmental samples by GC/MS should run the performance tests in reference 1 before beginning

## 2 SUMMARY OF METHOD

2 1 Stable isotopically labeled analogs of the compounds of interest are added to a 5 mL water sample The sample is purged at 20-25 °C with an inert gas in a specially designed chamber The volatile organic compounds are transferred from the aqueous phase into the gaseous phase where they are passed into a sorbent column and trapped After purging is completed, the trap is backflushed and heated rapidly to desorb the compounds into a gas chromatograph (GC) The compounds are separated by the GC and detected by a mass spectrometer (MS) (references 2 and 3) The labeled compounds serve to correct the variability of the analytical technique

**TABLE 1**

**Volatile Organic Compounds Analyzed by Isotope Dilution GCMS**

| | POLLUTANT | | | | LABELED COMPOUND | | |
|---|---|---|---|---|---|---|---|
| COMPOUND | STORET | CAS | EGD | NPDES | ANALOG | CAS | EGD |
| acetone | 81552 | 67-64-1 | 516 V | | $d_6$ | 666-52-4 | 616 V |
| acrolein | 34210 | 107-02-8 | 002 V | 001 V | $d_4$ | 33984-05-3 | 202 V |
| acrylonitrile | 34215 | 107-13-1 | 003 V | 002 V | $d_3$ | 53807-26-4 | 203 V |
| benzene | 34030 | 71-43-2 | 004 V | 003 V | $d_6$ | 1076-43-3 | 204 V |
| bromodichloromethane | 32101 | 75-27-4 | 048 V | 012 V | $^{13}C$ | 93952-10-4 | 248 V |
| bromoform | 32104 | 75-25-2 | 047 V | 005 V | $^{13}C$ | 72802-81-4 | 247 V |
| bromomethane | 34413 | 74-83-9 | 046 V | 020 V | $d_3$ | 1111-88-2 | 246 V |
| carbon tetrachloride | 32102 | 56-23-5 | 006 V | 006 V | $^{13}C$ | 32488-50-9 | 206 V |
| chlorobenzene | 34301 | 108-90-7 | 007 V | 007 V | $d_5$ | 3114-55-4 | 207 V |
| chloroethane | 34311 | 75-00-3 | 016 V | 009 V | $d_5$ | 19199-91-8 | 216 V |
| 2-chloroethylvinyl ether | 34576 | 110-75-8 | 019 V | 010 V | | | |
| chloroform | 32106 | 67-66-3 | 023 V | 011 V | $^{13}C$ | 31717-44-9 | 223 V |
| chloromethane | 34418 | 74-87-3 | 045 V | 021 V | $d_3$ | 1111-89-3 | 245 V |
| dibromochloromethane | 32105 | 124-48-1 | 051 V | 008 V | $^{13}C$ | 93951-99-6 | 251 V |
| 1 1-dichloroethane | 34496 | 75-34-3 | 013 V | 014 V | $d_3$ | 56912-77-7 | 213 V |
| 1,2-dichloroethane | 32103 | 107-06-2 | 010 V | 015 V | $d_4$ | 17070-07-0 | 210 V |
| 1,1-dichloroethene | 34501 | 75-35-4 | 029 V | 016 V | $d_2$ | 22280-73-5 | 229 V |
| trans-1,2-dichlorethene | 34546 | 156-60-5 | 030 V | 026 V | $d_3$ | 42366-47-2 | 230 V |
| 1,2-dichloropropane | 34541 | 78-87-5 | 032 V | 017 V | $d_6$ | 93952-08-0 | 232 V |
| trans-1,3-dichloropropene | 34699 | 10061-02-6 | 033 V | | $d_4$ | 93951-86-1 | 233 V |
| diethyl ether | 81576 | 60-29-7 | 515 V | | $d_{10}$ | 2679-89-2 | 615 V |
| p-dioxane | 81582 | 123-91-1 | 527 V | | $d_8$ | 17647-74-4 | 627 V |
| ethylbenzene | 34371 | 100-41-4 | 038 V | 019 V | $d_{10}$ | 25837-05-2 | 238 V |
| methylene chloride | 34423 | 75-09-2 | 044 V | 022 V | $d_2$ | 1665-00-5 | 244 V |
| methyl ethyl ketone | 81595 | 78-93-3 | 514 V | | $d_3$ | 53389-26-7 | 614 V |
| 1,1,2,2-tetrachloroethane | 34516 | 79-34-5 | 015 V | 023 V | $d_2$ | 33685-54-0 | 215 V |
| tetrachlorethene | 34475 | 127-18-4 | 085 V | 024 V | $^{13}C_2$ | 32488-49-6 | 285 V |
| toluene | 34010 | 108-88-3 | 086 V | 025 V | $d_8$ | 2037-26-5 | 286 V |
| 1,1,1-trichloroethane | 34506 | 71-55-6 | 011 V | 027 V | $d_3$ | 2747-58-2 | 211 V |
| 1,1,2-trichloroethane | 34511 | 79-00-5 | 014 V | 028 V | $^{13}C_2$ | 93952-09-1 | 214 V |
| trichloroethene | 39180 | 79-01-6 | 087 V | 029 V | $^{13}C_2$ | 93952-00-2 | 287 V |
| vinyl chloride | 39175 | 75-01-4 | 088 V | 031 V | $d_3$ | 6745-35-3 | 288 V |

TABLE 2

Gas Chromatography of Purgeable Organic Compounds by Isotope Dilution GC/MS

| EGD NO (1) | COMPOUND | REF EGD NO | MEAN RETENTION TIME (SEC) | MINIMUM LEVEL (2) (µg/L) |
|---|---|---|---|---|
| 181 | bromochloromethane (internal standard) | 181 | 730 | 10 |
| 245 | chloromethane-$d_3$ | 181 | 147 | 50 |
| 345 | chloromethane | 245 | 148 | 50 |
| 246 | bromomethane-$d_3$ | 181 | 243 | 50 |
| 346 | bromomethane | 246 | 246 | 50 |
| 288 | vinyl chloride-$d_3$ | 181 | 301 | 50 |
| 388 | vinyl chloride | 288 | 304 | 50 |
| 216 | chloroethane-$d_5$ | 181 | 378 | 50 |
| 316 | chloroethane | 216 | 386 | 50 |
| 244 | methylene chloride-$d_2$ | 181 | 512 | 10 |
| 344 | methylene chloride | 244 | 517 | 10 |
| 616 | acetone-$d_6$ | 181 | 554 | 50 |
| 716 | acetone | 616 | 565 | 50 |
| 002 | acrolein | 181 | 566 | 50 |
| 203 | acrylonitrile-$d_3$ | 181 | 606 | 50 |
| 303 | acrylonitrile | 203 | 612 | 50 |
| 229 | 1,1-dichloroethene-$d_2$ | 181 | 696 | 10 |
| 329 | 1,1-dichloroethene | 229 | 696 | 10 |
| 213 | 1,1-dichloroethane-$d_3$ | 181 | 778 | 10 |
| 313 | 1,1-dichloroethane | 213 | 786 | 10 |
| 615 | diethyl ether-$d_{10}$ | 181 | 804 | 50 |
| 715 | diethyl ether | 615 | 820 | 50 |
| 230 | trans-1,2-dichloroethene-$d_2$ | 181 | 821 | 10 |
| 330 | trans-1,2-dichloroethene | 230 | 821 | 10 |
| 614 | methyl ethyl ketone-$d_3$ | 181 | 840 | 50 |
| 714 | methyl ethyl ketone | 614 | 848 | 50 |
| 223 | chloroform-$^{13}C_1$ | 181 | 861 | 10 |
| 323 | chloroform | 223 | 861 | 10 |
| 210 | 1,2-dichloroethane-$d_4$ | 181 | 901 | 10 |
| 310 | 1,2-dichloroethane | 210 | 910 | 10 |
| 211 | 1,1,1-trichloroethane-$^{13}C_2$ | 181 | 989 | 10 |
| 311 | 1,1,1-trichloroethane | 211 | 999 | 10 |
| 527 | p-dioxane | 181 | 1001 | 10 |
| 206 | carbon tetrachloride-$^{13}C_1$ | 182 | 1018 | 10 |
| 306 | carbon tetrachloride | 206 | 1018 | 10 |
| 248 | bromodichloromethane-$^{13}C_1$ | 182 | 1045 | 10 |
| 348 | bromodichloromethane | 248 | 1045 | 10 |
| 232 | 1,2-dichloropropane-$d_6$ | 182 | 1123 | 10 |
| 332 | 1,2-dichloropropane | 232 | 1134 | 10 |
| 233 | trans-1,3-dichloropropene-$d_4$ | 182 | 1138 | 10 |
| 333 | trans-1,3-dichloropropene | 233 | 1138 | 10 |
| 287 | trichloroethene-$^{13}C_2$ | 182 | 1172 | 10 |
| 387 | trichloroethene | 287 | 1172 | 10 |
| 204 | benzene-$d_6$ | 182 | 1200 | 10 |
| 304 | benzene | 204 | 1212 | 10 |
| 251 | chlorodibromomethane-$^{13}C_1$ | 182 | 1222 | 10 |
| 351 | chlorodibromomethane | 251 | 1222 | 10 |
| 214 | 1,1,2-trichloroethane-$^{13}C_2$ | 182 | 1224 | 10 |
| 314 | 1,1,2-trichloroethane | 214 | 1224 | 10 |
| 019 | 2-chloroethylvinyl ether | 182 | 1278 | 10 |
| 182 | 2-bromo-1-chloropropane (internal standard) | 182 | 1306 | 10 |
| 247 | bromoform-$^{13}C_1$ | 182 | 1386 | 10 |
| 347 | bromoform | 247 | 1386 | 10 |
| 215 | 1,1,2,2-tetrachloroethane-$d_2$ | 183 | 1525 | 10 |
| 315 | 1,1,2,2-tetrachloroethane | 215 | 1525 | 10 |
| 285 | tetrachloroethene-$^{13}C_2$ | 183 | 1528 | 10 |
| 385 | tetrachloroethene | 285 | 1528 | 10 |
| 183 | 1,4-dichlorobutane (internal standard) | 183 | 1555 | 10 |
| 286 | toluene-$d_8$ | 183 | 1603 | 10 |
| 386 | toluene | 286 | 1619 | 10 |
| 207 | chlorobenzene-$d_5$ | 183 | 1679 | 10 |
| 307 | chlorobenzene | 207 | 1679 | 10 |
| 238 | ethylbenzene-$d_{10}$ | 183 | 1802 | 10 |
| 338 | ethylbenzene | 238 | 1820 | 10 |
| 185 | bromofluorobenzene | 183 | 1985 | 10 |

(1) Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method, reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method, reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution

(2) This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points Column 2 4 m (8 ft) x 2 mm i d glass packed with one percent SP-1000 coated on 60/80 Carbopak B Carrier gas helium at 40 mL/min Temperature program 3 min at 45°C 8°C per min to 240°C hold at 240°C for 15 minutes

Note The specifications in this table were developed from data collected from three wastewater laboratories

2 2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and the background corrected characteristic spectral masses with those of authentic standards

2 3 Quantitative analysis is performed by GC/MS using extracted ion current profile (EICP) areas Isotope dilution is used when labeled compounds are available, otherwise, an internal or external standard method is used

2 4 Quality is assured through reproducible calibration and testing of the purge and trap and GC/MS systems

## 3 CONTAMINATION AND INTERFERENCES

3 1 Impurities in the purge gas, organic compounds outgassing from the plumbing upstream of the trap, and solvent vapors in the laboratory account for the majority of contamination problems The analytical system is demonstrated to be free from interferences under conditions of the analysis by analyzing blanks initially and with each sample lot (samples analyzed on the same 8 hr shift), as described in section 8 5

3 2 Samples can be contaminated by diffusion of volatile organic compounds (particularly methylene chloride) through the bottle seal during shipment and storage A field blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination

3 3 Contamination by carry-over can occur when high level and low level samples are analyzed sequentially To reduce carry-over, the purging device and sample syringe are rinsed between samples with reagent water When an unusually concentrated sample is encountered, it is followed by analysis of a reagent water blank to check for carry-over For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds, or high levels of purgeable compounds, the purge device is washed with soap solution, rinsed with tap and distilled water, and dried in an oven at 100-125°C The trap and other parts of the system are also subject to contamination, therefore, frequent bakeout and purging of the entire system may be required

3 4 Interferences resulting from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being sampled

## 4 SAFETY

4 1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined, however, each chemical compound should be treated as a potential health hazard Exposure to these compounds should be reduced to the lowest possible level 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 data handling sheets should also be made available to all personnel involved in these analyses Additional information on laboratory safety can be found in references 4-6

4 2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens benzene, carbon tetrachloride, chloroform, and vinyl chloride Primary standards of these toxic compounds should be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled

## 5 APPARATUS AND MATERIALS

5 1 Sample bottles for discrete sampling

5 1 1 Bottle—25 to 40 mL with screw cap (Pierce 13075, or equivalent) Detergent wash, rinse with tap and distilled water, and dry at >105°C for one hour minumum before use

5 1 2 Septum—Teflon-faced silicone (Pierce 12722, or equivalent), cleaned as above and baked at 100-200°C for one hour minumum

5 2 Purge and trap device—consists of purging device, trap, and desorber Complete devices are commercially available

5 2 1 Purging device—designed to accept 5 mL samples with water column at least 3 cm deep The volume of the gaseous head space between the water and trap shall be less than 15 mL The purge gas shall be introduced less than 5 mm from the base of the water column and shall pass through the water as bubbles with a diameter less than 3 mm The purging device shown in figure 1 meets these criteria

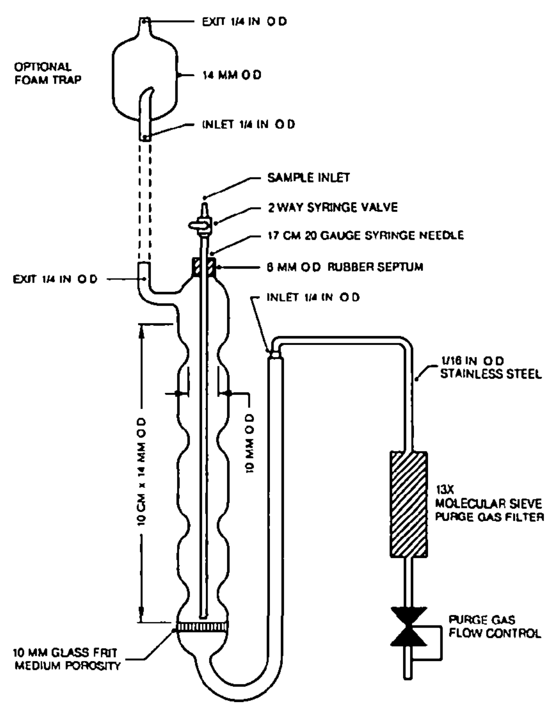


**FIGURE 1 Purging Device.**

5 2 2 Trap—25 to 30 cm x 2 5 mm i d minimum, containing the following

5 2 2 1 Methyl silicone packing—one ± 0 2 cm, 3 percent OV-1 on 60/80 mesh Chromosorb W, or equivalent

5 2 2 2 Porous polymer—15±1 0 cm, Tenax GC (2,6-diphenylene oxide polymer), 60/80 mesh, chromatographic grade, or equivalent

5 2 2 3 Silica gel—8±1 0 cm, Davison Chemical, 35/60 mesh, grade 15, or equivalent The trap shown in figure 2 meets these specifications

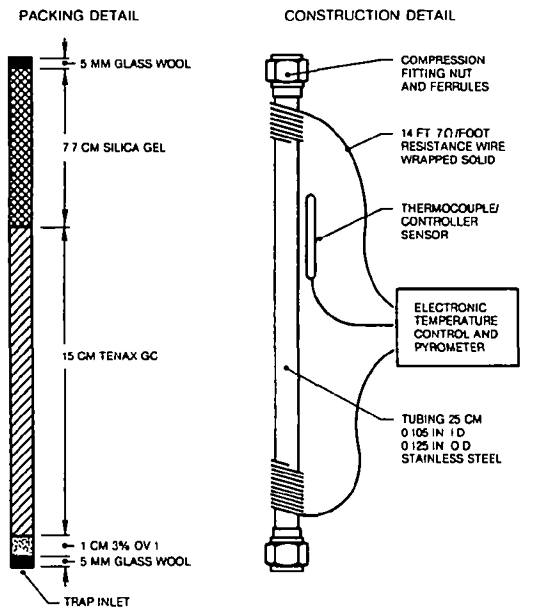


**FIGURE 2 Trap Packings and Construction to Include Desorb Capability.**

5 2 3 Desorber—shall heat the trap to 175±5°C, in 45 seconds or less The polymer section of the trap shall not exceed 180°C, and the remaining sections shall not exceed 220°C The desorber shown in figure 2 meets these specifications

5 2 4 The purge and trap device may be a separate unit or coupled to a GC as shown in figures 3 and 4

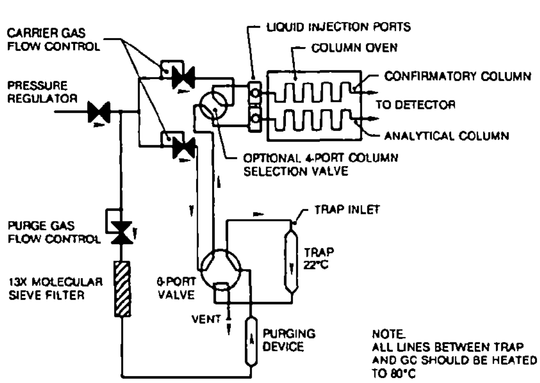


**FIGURE 3 Schematic of Purge and Trap Device-Purge Mode**

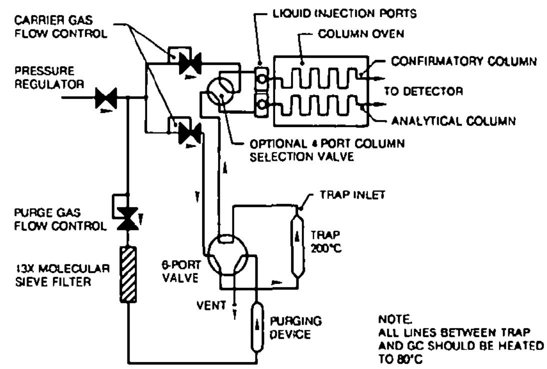


**FIGURE 4 Schematic of Purge and Trap Device-Desorb Mode.**

5 3 Gas chromatograph—shall be linearly temperature programmable with initial and final holds, shall contain a glass jet separator as the MS interface, and shall produce results which meet the calibration (section 7), quality assurance (section 8), and performance tests (section 11) of this method

5 3 1 Column—2 8±0 4 m x 2±0 5 mm i d glass, packed with one percent SP-1000 on Carbopak B, 60/80 mesh, or equivalent

5 4 Mass spectrometer—70 eV electron impact ionization, shall repetitively scan from 20 to 250 amu every 2-3 seconds, and produce a unit resolution (valleys between m/z 174-176 less than 10 percent of the height of the m/z 175 peak), background corrected mass spectrum from 50 ng 4-bromofluorobenzene (BFB) injected into the GC The BFB spectrum shall meet the mass-intensity criteria in table 3 All portions of the GC column, transfer lines, and separator which connect the GC column to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds

**TABLE 3**

**BFB Mass-Intensity Specifications**

| MASS | INTENSITY REQUIRED |
|---|---|
| 50 | 15 to 40 percent of mass 95 |
| 75 | 30 to 60 percent of mass 95 |
| 95 | base peak, 100 percent |
| 96 | 5 to 9 percent of mass 95 |
| 173 | <2 percent of mass 174 |
| 174 | >50 percent of mass 95 |
| 176 | 95 to 100 percent of mass 174 |
| 177 | 5 to 9 percent of mass 176 |

5 5 Data system—shall collect and record MS data, store mass intensity data in spectral libraries, process GC/MS data and generate reports, and shall calculate and record response factors

5 5 1 Data acquisition—mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device

5 5 2 Mass spectral libraries—user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GC/MS runs for the compounds of interest (section 7 2)

5 5 3 Data processing—the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GC/MS analysis Software routines shall be employed to compute retention times and EICP areas Displays of spectra, mass chromatograms, and library comparisons are required to verify results

5 5 4 Response factors and multipoint calibrations—the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and generate multi-point calibration curves (section 7) Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity Statistics on initial and on-going performance shall be maintained (sections 8 and 11)

5 6 Syringes—5 mL glass hypodermic, with Luer-lok tips

5 7 Micro syringes—10, 25, and 100 $\mu$L

5 8 Syringe valves—2-way, with Luer ends (Teflon or Kel-F)

5 9 Syringe—5 mL, gas-tight, with shut-off valve

5 10 Bottles—15 mL, screw-cap with Teflon liner

5 11 Balance—analytical, capable of weighing 0 1 mg

## 6 REAGENTS AND STANDARDS

6 1 Reagent water—water in which the compounds of interest and interfering compounds are not detected by this method (section 8 5 2) It may be generated by any of the following methods

6 1 1 Activated carbon—pass tap water through a carbon bed (Calgon Filtrasorb-300, or equivalent)

6 1 2 Water purifier—pass tap water through a purifier (Millipore Super Q, or equivalent)

6 1 3 Boil and purge—heat tap water to 90-100°C and bubble contaminant free inert gas through it for approx one hour While still hot, transfer the water to screw-cap bottles and seal with a Teflon-lined cap

6 2 Sodium thiosulfate—ACS granular

6 3 Methanol—pesticide quality or equivalent

6 4 Standard solutions—purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition If compound purity is 96 percent or greater, the weight may be used without correction to calculate the concentration of the standard

6 5 Preparation of stock solutions—prepare in methanol using liquid or gaseous standards per the steps below Observe the safety precautions given in section 4

6 5 1 Place approx 9 8 mL of methanol in a 10 mL ground glass stoppered volumetric flask Allow the flask to stand unstoppered for approximately 10 minutes or until all methanol wetted surfaces have dried In each case, weigh the flask, immediately add the compound, then immediately reweigh to prevent evaporation losses from affecting the measurement

6 5 1 1 Liquids—using a 100 $\mu$L syringe, permit 2 drops of liquid to fall into the methanol without contacting the neck of the flask Alternatively, inject a known volume of the compound into the methanol in the flask using a micro-syringe

6 5 1 2 Gases (chloromethane, bromomethane, chloroethane, vinyl chloride)—fill a valved 5 mL gas-tight syringe with the compound Lower the needle to approx 5 mm above the methanol meniscus Slowly introduce the compound above the surface of the meniscus The gas will dissolve rapidly in the methanol

6 5 2 Fill the flask to volume, stopper, then mix by inverting several times Calculate the concentration in mg/mL ($\mu$g/$\mu$L) from the weight gain (or density if a known volume was injected)

6 5 3 Transfer the stock solution to a Teflon sealed screw-cap bottle Store, with minimal headspace, in the dark at −10 to −20°C

6 5 4 Prepare fresh standards weekly for the gases and 2-chloroethylvinyl ether All other standards are replaced after one month, or sooner if comparison with check standards indicate a change in concentration Quality control check standards that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio

6 6 Labeled compound spiking solution—from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution to contain a concentration such that a 5-10 $\mu$L spike into each 5 mL sample, blank, or aqueous standard analyzed will result in a concentration of 20 $\mu$g/L of each labeled compound For the gases and for the water soluble compounds (acrolein, acrylonitrile, acetone, diethyl ether, and MEK), a concentration of 100 $\mu$g/L may be used Include the internal standards (section 7 5) in this solution so that a concentration of 20 $\mu$g/L in each sample, blank, or aqueous standard will be produced

6 7 Secondary standards—using stock solutions, prepare a secondary standard in methanol to contain each pollutant at a concentration of 500 $\mu$g/mL For the gases and water soluble compounds (section 6 6), a concentration of 2 5 mg/mL may be used

6 7 1 Aqueous calibration standards—using a 25 $\mu$L syringe, add 20 $\mu$L of the secondary standard (section 6 7) to 50, 100, 200, 500, and 1000 mL of reagent water to produce concentrations of 200, 100, 50, 20, and 10 $\mu$g/L, respectively If the higher concentration standard for the gases and water soluble compounds was chosen (section 6 6), these compounds will be at concentrations of 1000, 500, 250, 100, and 50 $\mu$g/L in the aqueous calibration standards

6 7 2 Aqueous performance standard—an aqueous standard containing all pollutants, internal standards, labeled compounds, and BFB is prepared daily, and analyzed each shift to demonstrate performance (section 11) This standard shall contain either 20 or

100 $\mu$g/L of the labeled and pollutant gases and water soluble compounds, 10 $\mu$g/L BFB, and 20 $\mu$g/L of all other pollutants, labeled compounds, and internal standards It may be the nominal 20 $\mu$g/L aqueous calibration standard (section 6 7 1)

6 7 3 A methanolic standard containing all pollutants and internal standards is prepared to demonstrate recovery of these compounds when syringe injection and purge and trap analyses are compared This standard shall contain either 100 $\mu$g/mL or 500 $\mu$g/mL of the gases and water soluble compounds, and 100 $\mu$g/mL of the remaining pollutants and internal standards (consistent with the amounts in the aqueous performance standard in 6 7 2)

6 7 4 Other standards which may be needed are those for test of BFB performance (section 7 1) and for collection of mass spectra for storage in spectral libraries (section 7 2)

## 7 CALIBRATION

7 1 Assemble the gas chromatographic apparatus and establish operating conditions given in table 2 By injecting standards into the GC, demonstrate that the analytical system meets the detection limits in table 2 and the mass-intensity criteria in table 3 for 50 ng BFB

7 2 Mass spectral libraries—detection and identification of the compounds of interest are dependent upon the spectra stored in user created libraries

7 2 1 Obtain a mass spectrum of each pollutant and labeled compound and each internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components That only a single compound is present is determined by examination of the spectrum Fragments not attributable to the compound under study indicate the presence of an interfering compound Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic m/z's or introduce other distortion

7 2 2 The authentic reference spectrum is obtained under BFB tuning conditions (section 7 1 and table 3) to normalize it to spectra from other instruments

7 2 3 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10 percent of the base peak This spectrum is stored for reverse search and for compound confirmation

7 3 Assemble the purge and trap device Pack the trap as shown in figure 2 and condition overnight at 170-180°C by backflushing with an inert gas at a flow rate of 20-30 mL/min Condition traps daily for a minimum of 10 minutes prior to use

7 3 1 Analyze the aqueous performance standard (section 6 7 2) according to the purge and trap procedure in section 10 Compute the area at the primary m/z (table 4) for each compound Compare these areas to those obtained by injecting one $\mu$L of the methanolic standard (section 6 7 3) to determine compound recovery The recovery shall be greater than 20 percent for the water soluble compounds, and 60-110 percent for all other compounds This recovery is demonstrated initially for each purge and trap GC/MS system The test is repeated only if the purge and trap or GC/MS systems are modified in any way that might result in a change in recovery

### TABLE 4
### Volatile Organic Compound Characteristic Masses

| LABELED COMPOUND | ANALOG | PRIMARY M/Z'S |
|---|---|---|
| acetone | $d_6$ | 58/64 |
| acrolein | $d_2$ | 56/60 |
| acrylonitrile | $d_3$ | 53/56 |
| benzene | $d_6$ | 78/84 |
| bromodichloromethane | $^{13}C$ | 83/86 |
| bromoform | $^{13}C$ | 173/176 |
| bromomethane | $d_3$ | 96/99 |
| carbon tetrachloride | $^{13}C$ | 47/48 |
| chlorobenzene | $d_5$ | 112/117 |
| chloroethane | $d_5$ | 64/71 |
| 2-chloroethylvinyl ether | $d_7$ | 106/113 |
| chloroform | $^{13}C$ | 85/86 |
| chloromethane | $d_3$ | 50/53 |
| dibromochloromethane | $^{13}C$ | 129/130 |
| 1,1-dichloroethane | $d_3$ | 63/66 |
| 1,2-dichloroethane | $d_4$ | 62/67 |
| 1,1-dichloroethene | $d_2$ | 61/65 |
| trans-1,2-dichloroethene | $d_2$ | 61/65 |
| 1,2-dichloropropane | $d_6$ | 63/67 |
| trans-1,3-dichloropropene | $d_4$ | 75/79 |
| diethyl ether | $d_{10}$ | 74/84 |
| p-dioxane | $d_8$ | 88/96 |
| ethylbenzene | $d_{10}$ | 106/116 |
| methylene chloride | $d_2$ | 84/88 |
| methyl ethyl ketone | $d_3$ | 72/75 |
| 1,1,2,2-tetrachloroethane | $d_2$ | 83/84 |
| tetrachloroethene | $^{13}C_2$ | 164/172 |
| toluene | $d_8$ | 92/98 |
| 1,1,1-trichloroethane | $d_3$ | 97/102 |
| 1,1,2-trichloroethane | $^{13}C_2$ | 83/84 |
| trichloroethene | $^{13}C$ | 95/136 |
| vinyl chloride | $d_3$ | 62/65 |

7 3 2 Demonstrate that 100 ng toluene (or toluene-$d_8$) produces an area at m/z 91 (or 98) approx one-tenth that required to exceed the linear range of the system The exact value must be determined by experience for each instrument It is used to match the calibration range of the instrument to the analytical range and detection limits required

7 4 Calibration by isotope dilution—the isotope dilution approach is used for the purgeable organic compounds when appropriate labeled compounds are available and when interferences do not preclude the analysis If labeled compounds are not available, or interferences are present, the internal standard method (section 7 5) is used A calibration curve encompassing the concentration range of interest is prepared for each compound determined The relative response (RR) vs concentration ($\mu$g/L) is plotted or computed using a linear regression An ex-

ample of a calibration curve for toluene using toluene-$d_8$ is given in figure 5 Also shown are the ±10 percent error limits (dotted lines) Relative response is determined according to the procedures described below A minumum of five data points are required for calibration (section 7 4 4)

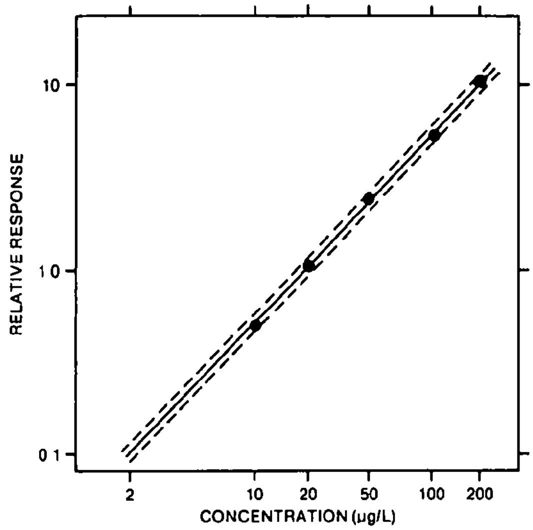


**FIGURE 5 Relative Response Calibration Curve for Toluene. The Dotted Lines Enclose a ±10 Percent Error Window.**

7 4 1 The relative response (RR) of pollutant to labeled compound is determined from isotope ratio values calculated from acquired data Three isotope ratios are used in this process

$R_x$ = the isotope ratio measured in the pure pollutant (figure 6A)

$R_y$ = the isotope ratio of pure labeled compound (figure 6B)

$R_m$ = the isotope ratio measured in the analytical mixture of the pollutant and labeled compounds (figure 6C)

The correct way to calculate RR is

$$RR = \frac{(R_y - R_m)(R_x + 1)}{(R_m - R_x)(R_y + 1)}$$

If $R_m$ is not between $2R_y$ and $0\ 5R_x$, the method does not apply and the sample is analyzed by the internal standard method (section 7 5)

7 4 2 In most cases, the retention times of the pollutant and labeled compound are the same and isotope ratios (R's) can be calculated from the EICP areas, where

$$R = \frac{(\text{area at } m_1/z)}{(\text{area at } m_2/z)}$$

If either of the areas is zero, it is assigned a value of one in the calculations, that is, if

area of $m_1/z$ = 50721, and
area of $m_2/z$ = 0, then

$$R = \frac{50721}{1} = 50720$$

The m/z's are always selected such that $R_x > R_y$ When there is a difference in retention times (RT) between the pollutant and labeled compounds, special precautions are required to determine the isotope ratios

$R_x$, $R_y$, and $R_m$ are defined as follows

$$R_x = \frac{[\text{area } m_1/z \text{ (at } RT_1)]}{1}$$

$$R_y = \frac{1}{[\text{area } m_2/z \text{ (at } RT_2)]}$$

$$R_m = \frac{[\text{area } m_1/z \text{ (at } RT_1)]}{[\text{area } m_2/z \text{ (at } RT_2)]}$$

7 4 3 An example of the above calculations can be taken from the data plotted in figure 6 for toluene and toluene-$d_8$ For these data,

$$R_x = \frac{168920}{1} = 168900$$

$$R_y = \frac{1}{60960} = 0\ 00001640$$

$$R_m = \frac{96868}{82508} = 1\ 174$$

The RR for the above data is then calculated using the equation given in section 7 4 1 For the example, RR = 1 174 NOTE Not all labeled compounds elute before their pollutant analogs

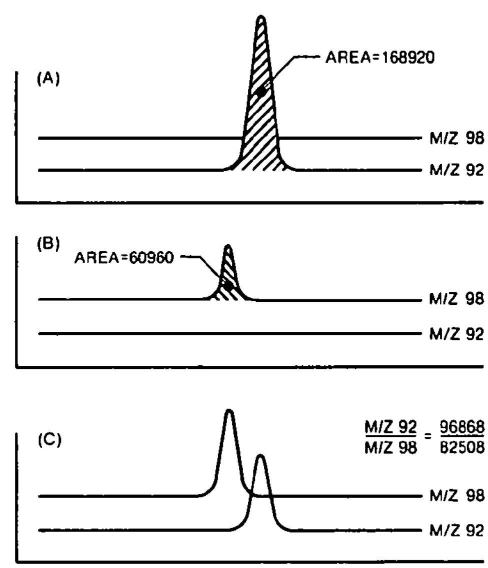


**FIGURE 6 Extracted Ion Current Profiles for (A) Toluene, (B) Toluene-$d_8$, and (C) a Mixture of Toluene and Toluene-$d_8$.**

7 4 4 To calibrate the analytical system by isotope dilution, analyze a 5 mL aliquot of each of the aqueous calibration standards (section 6 7 1) spiked with an appropriate constant amount of the labeled compound spiking solution (section 6 6), using the purge and trap procedure in section 10 Compute the RR at each concentration

7 4 5 Linearity—if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, an averaged relative response/concentration ratio may be used for that compound, otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range

7 5 Calibration by internal standard—used when criteria for isotope dilution (section 7 4) cannot be met The method is applied to pollutants having no labeled analog and to the labeled compounds The internal standards used for volatiles analyses are bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane Concentrations of the labeled compounds and pollutants without labeled analogs are computed relative to the nearest eluted internal standard, as shown in table 2

7 5 1 Response factors—calibration requires the determination of response factors (RF) which are defined by the following equation

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}, \text{ where}$$

$A_s$ is the EICP area at the characteristic m/z for the compound in the daily standard

$A_{is}$ is the EICP area at the characteristic m/z for the internal standard

$C_{is}$ is the concentration ($\mu$g/L) of the internal standard

$C_s$ is the concentration of the pollutant in the daily standard

7 5 2 The response factor is determined at 10, 20, 50, 100, and 200 $\mu$g/L for the pollutants (optionally at five times these concentrations for gases and water soluble pollutants—see section 6 6), in a way analogous to that for calibration by isotope dilution (section 7 4 4) The RF is plotted against concentration for each compound in the standard ($C_s$) to produce a calibration curve

7 5 3 Linearity—if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound, otherwise, the complete calibration curve for that compound shall be used over the 5 point range

7 6 Combined calibration—by adding the isotopically labeled compounds and internal standards (section 6 6) to the aqueous calibration standards (section 6 7 1), a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods These curves are verified each shift (section 11 5) by purging the aqueous performance standard (section 6 7 2) Recalibration is required only if calibration and on-going performance (section 11 5) criteria cannot be met

## 8 QUALITY ASSURANCE/QUALITY CONTROL

8 1 Each laboratory that uses this method is required to operate a formal quality assurance program The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method

8 1 1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method This ability is established as described in section 8 2

8 1 2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met Each time a modification is made to the method, the analyst is required to repeat the procedure in section 8 2 to demonstrate method performance

8 1 3 Analyses of blanks are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis (section 3) The procedures and criteria for analysis of a blank are described in sections 8 5

8 1 4 The laboratory shall spike all samples with labeled compounds to monitor method performance This test is described in section 8 3 When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (section 14 2)

8 1 5 The laboratory shall, on an on-going basis, demonstrate through the analysis of the aqueous performance standard (section 6 7 2) that the analysis system is in control This procedure is described in sections 11 1 and 11 5

8 1 6 The laboratory shall maintain records to define the quality of data that is generated Development of accuracy statements is described in sections 8 4 and 11 5 2

8 2 Initial precision and accuracy—to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations

8 2 1 Analyze two sets of four 5-mL aliquots (8 aliquots total) of the aqueous performance standard (section 6 7 2) according to the method beginning in section 10

8 2 2 Using results of the first set of four analyses in section 8 2 1, compute the average recovery ($\overline{X}$) in $\mu$g/L and the standard deviation of the recovery (s) in $\mu$g/L for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog

8 2 3 For each compound, compare s and $\overline{X}$ with the corresponding limits for initial precision and accuracy found in table 5 If s and $\overline{X}$ for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin If, however, any individual s exceeds the precision limit or any individual $\overline{X}$ falls outside the range for accuracy, system performance is unacceptable for that compound NOTE The large number of

compounds in table 5 present a substantial probability that one or more will fail one of the acceptance criteria when all compounds are analyzed To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows

8 2 4 Using the results of the second set of four analyses, compute s and $\overline{X}$ for only those compounds which failed the test of the first set of four analyses (section 8 2 3) If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin If, however, any of the same compounds fail again, the analysis system is not performing properly for the compound (s) in question In this event, correct the problem and repeat the entire test (section 8 2 1)

8 3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix

8 3 1 Spike and analyze each sample according to the method beginning in section 10

8 3 2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (section 7 5)

8 3 3 Compare the percent recovery for each compound with the corresponding labeled compound recovery limit in table 5 If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample Therefore, the sample matrix is complex and the sample is to be diluted and reanalyzed, per section 14 2

8 4 As part of the QA program for the laboratory, method accuracy for waste-water samples shall be assessed and records shall be maintained After the analysis of five wastewater samples for which the labeled compounds pass the test in section 8 3 3, compute the average percent recovery (P) and the standard deviation of the percent recovery ($s_p$) for the labeled compounds only Express the accuracy assessment as a percent recovery interval from $P - 2s_p$ to $P + 2s_p$ For example, if $P = 90\%$ and $s_p = 10\%$, the accuracy interval is expressed as 70-110% Update the accuracy assessment for each compound on a regular basis (e g after each 5-10 new accuracy measurements )

**TABLE 5**

**Acceptance Criteria for Performance Tests**

| | ACCEPTANCE CRITERIA AT 20 µg/L | | | |
|---|---|---|---|---|
| | INITIAL PRECISION AND ACCURACY SECTION 8 2 3 | | LABELED COMPOUND RECOVERY SECTION 8 3 AND 14 2 | ON-GOING ACCURACY SECTION 11 5 |
| COMPOUND | s (µg/L) | $\overline{X}$ (µg/L) | P (%) | R (µg/L) |
| acetone | note 1 | | | |
| acrolein | note 2 | | | |
| acrylonitrile | note 2 | | | |
| benzene | 9 0 | 13 0 - 28 2 | ns - 196 | 4 - 33 |
| bromodichloromethane | 8 2 | 6 5 - 31 5 | ns - 199 | 4 - 34 |
| bromoform | 7 0 | 7 4 - 35 1 | ns - 214 | 6 - 36 |
| bromomethane | 25 0 | d - 54 3 | ns - 414 | d - 61 |
| carbon tetrachloride | 6 9 | 15 9 - 24 8 | 42 - 165 | 12 - 30 |
| chlorobenzene | 8 2 | 14 2 - 29 6 | ns - 205 | 4 - 35 |
| chloroethane | 14 8 | 2 1 - 46 7 | ns - 308 | d - 51 |
| 2-chloroethylvinyl ether | 36 0 | d - 69 8 | ns - 554 | d - 79 |
| chloroform | 7 9 | 11 6 - 26 3 | 18 - 172 | 8 - 30 |
| chloromethane | 26 0 | d - 55 5 | ns - 410 | d - 64 |
| dibromochloromethane | 7 9 | 11 2 - 29 1 | 16 - 185 | 8 - 32 |
| 1,1-dichloroethane | 6 7 | 11 4 - 31 4 | 23 - 191 | 9 - 33 |
| 1,2-dichloroethane | 7 7 | 11 6 - 30 1 | 12 - 192 | 8 - 33 |
| 1,1-dichloroethene | 11 7 | d - 49 8 | ns - 315 | d - 52 |
| trans-1,2-dichloroethene | 7 4 | 10 5 - 31 5 | 15 - 195 | 8 - 34 |
| 1,2-dichloropropane | 19 2 | d - 46 8 | ns - 343 | d - 51 |
| trans-1,3-dichloropropene | 14 5 | d - 40 2 | ns - 284 | d - 44 |
| diethyl ether | note 1 | | | |
| p-dioxane | note 1 | | | |
| ethyl benzene | 9 6 | 15 6 - 28 5 | ns - 203 | 5 - 35 |
| methylene chloride | 9 7 | d - 49 8 | ns - 316 | d - 50 |
| methyl ethyl ketone | note 1 | | | |
| 1,1,2,2-tetrachloroethane | 9 6 | 10 7 - 30 0 | 5 - 199 | 7 - 34 |
| tetrachloroethene | 6 6 | 15 1 - 28 5 | 31 - 181 | 11 - 32 |
| toluene | 6 3 | 14 5 - 28 7 | 4 - 193 | 6 - 33 |
| 1,1,1-trichloroethane | 5 9 | 10 5 - 33 4 | 12 - 200 | 8 - 35 |
| 1,1,2-trichloroethane | 7 1 | 11 8 - 29 7 | 21 - 184 | 9 - 32 |
| trichloroethene | 8 9 | 16 6 - 29 5 | 35 - 196 | 12 - 34 |
| vinyl chloride | 27 9 | d - 58 5 | ns - 452 | d - 65 |

d = detected result must be greater than zero
ns = no specification limit would be below detection limit

note 1 Specifications not available for these compounds at time of release of this method
note 2 Specifications not developed for these compounds, use method 603

8 5 Blanks—reagent water blanks are analyzed to demonstrate freedom from carry-over (section 3) and contamination

8 5 1 The level at which the purge and trap system will carry greater than 5 $\mu$g/L of a pollutant of interest (table 1) into a succeeding blank shall be determined by analyzing successively larger concentrations of these compounds When a sample contains this concentration or more, a blank shall be analyzed immediately following this sample to demonstrate no carry-over at the 5 $\mu$g/L level

8 5 2 With each sample lot (samples analyzed on the same 8 hr shift), a blank shall be analyzed immediately after analysis of the aqueous performance standard (section 11 1) to demonstrate freedom from contamination If any of the compounds of interest (table 1) or any potentially interfering compound is found in a blank at greater than 10 $\mu$g/L (assuming a response factor of 1 relative to the nearest eluted internal standard for compounds not listed in table 1), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level

8 6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state The standards used for calibration (section 7), calibration verification (section 11 5) and for initial (section 8 2) and ongoing (section 11 5) precision and accuracy should be identical, so that the most precise results will be obtained The GC/MS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of volatiles by this method

8 7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used

## 9 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

9 1 Grab samples are collected in glass containers having a total volume greater than 20 mL Fill sample bottles so that no air bubbles pass through the sample as the bottle is filled Seal each bottle so that no air bubbles are entrapped Maintain the hermetic seal on the sample bottle until time of analysis

9 2 Samples are maintained at 0-4 °C from the time of collection until analysis If the sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL) to the empty sample bottles just prior to shipment to the sample site EPA Methods 330 4 and 330 5 may be used for measurement of residual chlorine (reference 8) If preservative has been added, shake the bottle vigorously for one minute immediately after filling

9 3 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined Collect about 500 mL of sample in a clean container Adjust the pH of the sample to about 2 by adding HC1 (1 + 1) while stirring Check pH with narrow range (1 4 to 2 8) pH paper Fill a sample container as described in section 9 1 If residual chlorine is present, add sodium thiosulfate to a separate sample container and fill as in section 9 1

9 4 All samples shall be analyzed within 14 days of collection

## 10 PURGE, TRAP, AND GC/MS ANALYSIS

10 1 Remove standards and samples from cold storage and bring to 20-25 °C

10 2 Adjust the purge gas flow rate to 40 ± 4 mL/min Attach the trap inlet to the purging device and set the valve to the purge mode (figure 3) Open the syringe valve located on the purging device sample introduction needle (figure 1)

10 3 Remove the plunger from a 5-mL syringe and attach a closed syringe valve Open the sample bottle and carefully pour the sample into the syringe barrel until it overflows Replace the plunger and compress the sample Open the syringe valve and vent any residual air while adjusting the sample volume to 5 0 mL Because this process of taking an aliquot destroys the validity of the sample for future analysis, fill a second syringe at this time to protect against possible loss of data Add an appropriate amount of the labeled compound spiking solution (section 6 6) through the valve bore, then close the valve

10 4 Attach the syringe valve assembly to the syringe valve on the purging device Open both syringe valves and inject the sample into the purging chamber

10 5 Close both valves and purge the sample for 11 0 ± 0 1 minutes at 20-25 °C

10 6 After the 11 minute purge time, attach the trap to the chromatograph and set the purge and trap apparatus to the desorb mode (figure 4) Desorb the trapped compounds into the GC column by heating the trap to 170-180 °C while backflushing with carrier gas at 20-60 mL/min for four minutes Start MS data acquisition upon start of the desorb cycle, and start the GC column temperature program 3 minutes later Table 2 summarizes the recommended operating conditions for the gas chromatograph Included in this table are retention times and detection limits that were achieved under these conditions An example of the separations achieved by the column listed is shown in figure 8 Other columns may be used provided the requirements in section 8 can be met If the priority pollutant gases produce GC peaks so broad that the precision and recovery specifications (section 8 2) cannot be met, the column may be cooled to ambient or sub-ambient temperature to sharpen these peaks

10 7 While analysis of the desorbed compounds proceeds, empty the purging chamber using the sample introduction syringe Wash the chamber with two 5-mL portions of reagent water After the purging device has been emptied, allow the purge gas to vent through the chamber until the frit is dry, so that it is ready for the next sample

10 8 After desorbing the sample for four minutes, recondition the trap by returning to the purge mode Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap Maintain the trap temperature at 170-180°C After approximately seven minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap When cool, the trap is ready for the next sample

## 11 SYSTEM PERFORMANCE

11 1 At the beginning of each 8 hr shift during which analyses are performed, system calibration and performance shall be verified for all pollutants and labeled compounds For these tests, analysis of the *aqueous performance standard (section 6 7 2) shall* be used to verify all performance criteria Adjustment and/or recalibration (per section 7) shall be performed until all performance criteria are met Only after all performance criteria are met may blanks and samples be analyzed

11 2 BFB spectrum validity—the criterai in table 3 shall be met

11 3 Retention times—the absolute retention times of all compounds shall approximate those given in table 2

11 4 GC resolution—the valley height between toluene and toluene-$d_8$ (at m/z 91 and 98 plotted on the same graph) shall be less than 10 percent of the taller of the two peaks

11 5 Calibration verification and on-going precision and accuracy—compute the concentration of each pollutant (table 1) by isotope dilution (section 7 4) for those compounds which have labeled analogs Compute the concentration of each pollutant (table 1) which has no labeled analog by the internal standard method (section 7 5) Compute the concentration of the labeled compounds by the internal standard method These concentrations are computed based on the calibration data determined in section 7

11 5 1 For each pollutant and labeled compound, compare the concentration with the corresponding limit for ongoing accuracy in table 5 *If all compounds meet the* acceptance criteria, system performance is acceptable and analysis of blanks and samples may continue If any individual value falls outside the range given, system performance is unacceptable for that compound NOTE The large number of compounds in table 5 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed To determilne if the analytical system is out of control, or if the failure may be attributed to probability, proceed as follows

11 5 1 1 Analyze a second aliquot of the aqueous performance standard (section 6 7 2)

11 5 1 2 Compute the concentration for only those compounds which failed the first test (section 11 5 1) If these compounds now pass, system performance is acceptable for all compounds and analyses of blanks and samples may proceed If, however, any of the compounds fail again, the measurement system is not performing properly for these compounds In this event, locate and correct the problem or recalibrate the system (section 7), and repeat the entire test (section 11 1) for all compounds

11 5 2 Add results which pass the specification in 11 5 1 2 to initial (section 8 2) and previous on-going data Update QC charts to form a graphic representation of laboratory performance (figure 7) Develop a statement of accuracy for each pollutant and labeled compound by calculating the average percent recovery (R) and the standard deviation of percent recovery ($s_r$) Express the accuracy as a recovery interval from $R - 2s_r$ to $R + 2s_r$ For example, if R = 95% and $s_r$ = 5%, the accuracy is 85-105 percent

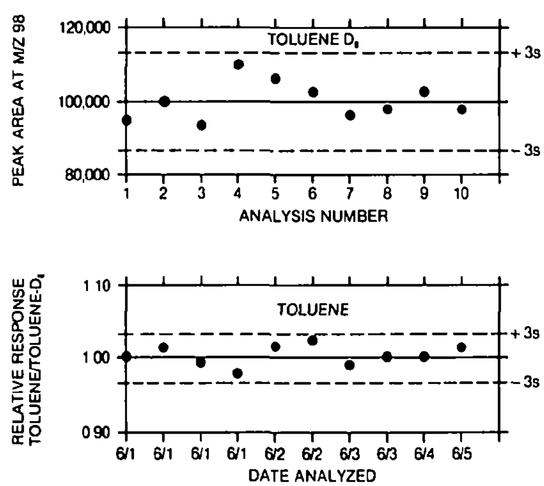


**FIGURE 7 Quality Control Charts Showing Area (top graph) and Relative Response of Toluene to Toluene-$d_8$ (lower graph) Plotted as a Function of Time or Analysis Number.**

## 12 QUALITATIVE DETERMINATION-accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (section 11 1) Identification is confirmed when spectra and retention times agree per the criteria below

12 1 Labeled compounds and pollutants having no labeled analog

12 1 1 The signals for all characteristic masses stored in the spectral library (section 7 2 3) shall be present and shall maximize within the same two consecutive scans

12 1 2 Either (1) the backgound corrected EICP areas, or (2) *the corrected relative intensities of the mass spectral* peaks at the GC peak maximum shall agree within a factor of two (0 5 to 2 times) for all masses stored in the library

12 1 3 The retention time relative to the nearest eluted internal standard shall be within ± 7 scans or ± 20 seconds, whichever is greater of this difference in the shift standard (section 11 1)

12 2 Pollutants having a labeled analog

12 2 1 The signals for all characteristic masses stored in the spectral library (section 7 2 3) shall be present and shall maximize within the same two consecutive scans

12 2 2 Either (1) the backgound corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library

12 2 3 The retention time difference between the pollutant and its labeled analog shall agree within ± 2 scans or ± 6 seconds (whichever is greater) of this difference in the shift standard (section 11 1)

12 3 Masses present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or backgound ions If the experimental mass spectrum is contaminated, an experienced spectrometrist (section 1 4) is to determine the presence or absence of the compound

## 13 QUANTITATIVE DETERMINATION

13 1 Istope dilution—by adding a known amount of a labeled compound to every sample prior to purging, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon purging, desorption, and gas chromatography Relative response (RR) values for sample mixtures are used in conjunction with calibration curves described in section 7 4 to determine concentrations directly, so long as labeled compound spiking levels are constant For the toluene example given in figure 6 (section 7 4 3), RR would be equal to 1 174 For this RR value, the toluene calibration curve given in figure 5 indicates a concentration of 31 8 $\mu$g/L

13 2 Internal standard—calculate the concentration using the response factor determined from calibration data (section 7 5) and the following equation

$$\text{Concentration} = \frac{(A_S \times C_{IS})}{(A_{IS} \times RF)}$$

where the terms are as defined in section 7 5 1

13 3 If the EICP area at the quantitation mass for any compound exceeds the calibration range of the system, the sample is diluted by successive factors of 10 and these dilutions are analyzed until the area is within the calibration range

13 4 Report results for all pollutants and labeled compounds (table 1) found in all standards, blanks, and samples, in $\mu$g/L, to three significant figures Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation mass is within the calibration range (section 13 3) and the labeled compound recovery is within the normal range for the Method (section 14 2)

## 14 ANALYSIS OF COMPLEX SAMPLES

14 1 Untreated effluents and other samples frequently contain high levels (>1000 $\mu$g/L) of the compounds of interest and of interfering compounds Some samples will foam excessively when purged, others will overload the trap and/or GC column

14 2 Dilute 0 5 mL of sample with 4 5 mL of reagent water and analyze this diluted sample when labeled compound recovery is outside the range given in table 5 If the recovery remains outside of the range for the diluted sample, the aqueous performance standard shall be analyzed (section 11) and calibration verified (section 11 5) If the recovery for the labeled compound in the aqueous performance standard is outside the range given in table 5, the analytical system is out of control In this case, the instrument shall be repaired, the performance specifications in section 11 shall be met, and the analysis of the undiluted sample shall be repeated If the recovery for the aqueous performance standard is within the range given in table 5, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes

14 3 Reverse search computer programs can misinterpret the spectrum of chromatographically unresolved pollutant and labeled compound pairs with overlapping spectra when a high level of the pollutant is present Examine each chromatogram for peaks greater then the height of the internal standard peaks These peaks can obscure the compounds of interest

## 15 METHOD PERFORMANCE

15 1 The specifications for this method were taken from the interlaboratory validation of EPA Method 624 (reference 9) Method 1624 has been shown to yield slightly better performance on treated effluents than method 624 Additional method performance data can be found in Reference 10

15 2 A chromatogram of the 20 $\mu$g/L aqueous performance standards (sections 6 7 2 and 11 1) is shown in figure 8

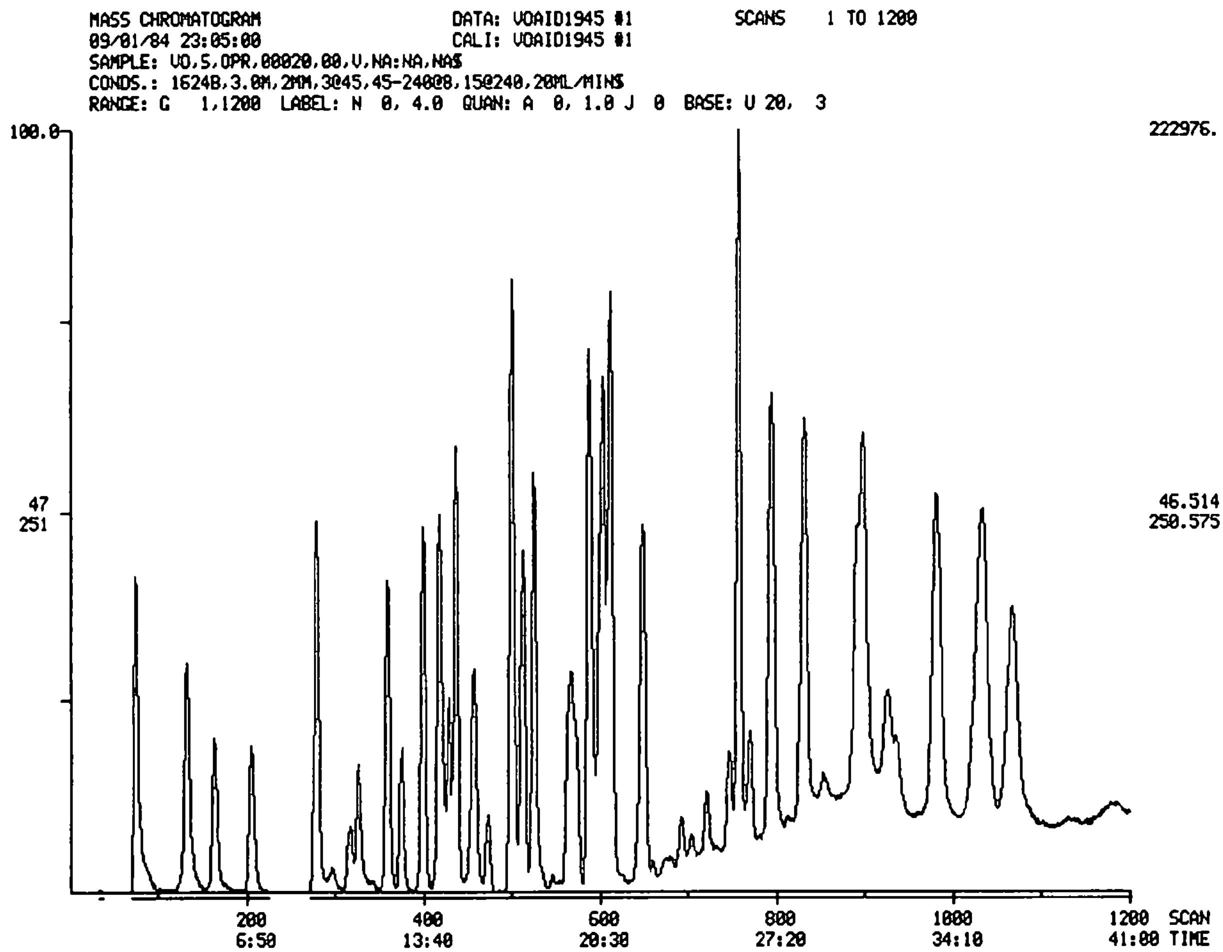


FIGURE 8 Chromatogram of Aqueous Performance Standards

## 16 REFERENCES


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10 Colby, B N, Beimer, R G, Rushneck, D R, and Telliard, W A, "Isotope Dilution Gas Chromatography-Mass Spectrometry for the Determination of Priority Pollutants in Industrial Effluents," USEPA, Effluent Guidelines Division, Washington, DC 20460 (1980)

# Method 1625 Revision B
# SEMIVOLATILE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

## 1 SCOPE AND APPLICATION

1.1 This method is designed to determine the semivolatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to extraction and analysis by capillary column gas chromatography-mass spectrometry (GC/MS)

1.2 The chemical compounds listed in tables 1 and 2 may be determined in municipal and industrial discharges by this method The method is designed to meet the survey requirements of EPA's Industrial Technology Division (ITD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR 136 1 Any modifications 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

**Table 1**
**Base/Neutral Extractable Compounds**

| | POLLUTANT | | | | LABELED COMPOUND | | |
|---|---|---|---|---|---|---|---|
| COMPOUND | STORET | CAS | EGD | NPDES | ANALOG | CAS | EGD |
| acenaphthene | 34205 | 83-32-9 | 001 B | 001 B | $d_{10}$ | 15067-20-2 | 201 B |
| acenaphthylene | 34200 | 208-96-8 | 077 B | 002 B | $d_{8}$ | 93951-97-4 | 277 B |
| anthracene | 34220 | 120-12-7 | 078 B | 003 B | $d_{10}$ | 1719-06-8 | 278 B |
| benzidine | 39120 | 92-87-5 | 005 B | 004 B | $d_{8}$ | 92890-63-6 | 205 B |
| benzo(a)anthracene | 34526 | 56-55-3 | 072 B | 005 B | $d_{12}$ | 1718-53-2 | 272 B |
| benzo(b)fluoranthene | 34230 | 205-99-2 | 074 B | 007 B | $d_{12}$ | 93951-98-5 | 274 B |
| benzo(k)fluoranthene | 34242 | 207-08-9 | 075 B | 009 B | $d_{12}$ | 93952-01-3 | 275 B |
| benzo(a)pyrene | 34247 | 50-32-8 | 073 B | 006 B | $d_{12}$ | 63466-71-7 | 273 B |
| benzo(ghi)perylene | 34521 | 191-24-2 | 079 B | 008 B | $d_{12}$ | 93951-66-7 | 279 B |
| biphenyl (Appendix C) | 81513 | 92-54-4 | 512 B | | $d_{10}$ | 1486-01-7 | 612 B |
| bis(2-chloroethyl) ether | 34273 | 111-44-4 | 018 B | 011 B | $d_{8}$ | 93952-02-4 | 218 B |
| bis (2-chloroethoxy) methane | 34278 | 111-91-1 | 043 B | 010 B | $d_{8}$ | 93966-78-0 | 243 B |
| bis(2-chloroisopropyl) ether | 34283 | 108-60-1 | 042 B | 012 B | $d_{12}$ | 93951-67-8 | 242 B |
| bis(2-ethylhexyl) phthalate | 39100 | 117-81-7 | 066 B | 013 B | $d_{4}$ | 93951-87-2 | 266 B |
| 4-bromophenyl phenyl ether | 34636 | 101-55-3 | 041 B | 014 B | $d_{5}$ | 93951-83-8 | 241 B |
| butyl benzyl phthalate | 34292 | 85-68-7 | 067 B | 015 B | $d_{4}$ | 93951-88-3 | 267 B |
| n-$C_{10}$ (Appendix C) | 77427 | 124-18-5 | 517 B | | $d_{22}$ | 16416-29-8 | 617 B |
| n-$C_{12}$ (Appendix C) | 77588 | 112-40-3 | 506 B | | $d_{26}$ | 16416-30-1 | 606 B |
| n-$C_{14}$ (Appendix C) | 77691 | 629-59-4 | 518 B | | | | |
| n-$C_{16}$ (Appendix C) | 77757 | 544-76-3 | 519 B | | $d_{34}$ | 15716-08-2 | 619 B |
| n-$C_{18}$ (Appendix C) | 77804 | 593-45-3 | 520 B | | | | |
| n-$C_{20}$ (Appendix C) | 77830 | 112-95-8 | 521 B | | $d_{42}$ | 62369-67-9 | 621 B |
| n-$C_{22}$ (Appendix C) | 77859 | 629-97-9 | 522 B | | | | |
| n-$C_{24}$ (Appendix C) | 77886 | 646-31-1 | 523 B | | $d_{50}$ | 16416-32-3 | 623 B |
| n-$C_{26}$ (Appendix C) | 77901 | 630-01-3 | 524 B | | | | |
| n-$C_{28}$ (Appendix C) | 78116 | 630-02-4 | 525 B | | | | 625 B |
| n-$C_{30}$ (Appendix C) | 78117 | 638-68-6 | 526 B | | $d_{62}$ | 93952-07-9 | 626 B |
| carbazole (4c) | 77571 | 86-74-8 | 528 B | | $d_{8}$ | 38537-24-5 | 628 B |
| 2-chloronaphthalene | 34581 | 81-58-7 | 020 B | 016 B | $d_{7}$ | 93951-84-9 | 220 B |
| 4-chlorophenyl phenyl ether | 34641 | 7005-72-3 | 040 B | 017 B | $d_{5}$ | 93951-85-0 | 240 B |
| chrysene | 34320 | 218-01-9 | 076 B | 018 B | $d_{12}$ | 1719-03-5 | 276 B |
| p-cymene (Appendix C) | 77356 | 99-87-6 | 513 B | | $d_{14}$ | 93952-03-5 | 613 B |
| dibenzo(a,h)anthracene | 34556 | 53-70-3 | 082 B | 019 B | $d_{14}$ | 13250-98-1 | 282 B |
| dibenzofuran (Appendix C & 4c) | 81302 | 132-64-9 | 505 B | | $d_{8}$ | 93952-04-6 | 605 B |
| dibenzothiophene (Synfuel) | 77639 | 132-65-0 | 504 B | | $d_{8}$ | 33262-29-2 | 604 B |
| di-n-butyl phthalate | 39110 | 84-74-2 | 068 B | 026 B | $d_{4}$ | 93952-11-5 | 268 B |
| 1,2-dichlorobenzene | 34536 | 95-50-1 | 025 B | 020 B | $d_{4}$ | 2199-69-1 | 225 B |
| 1,3-dichlorobenzene | 34566 | 541-73-1 | 026 B | 021 B | $d_{4}$ | 2199-70-4 | 226 B |
| 1,4-dichlorobenzene | 34571 | 106-46-7 | 027 B | 022 B | $d_{4}$ | 3855-82-1 | 227 B |
| 3,3-dichlorobenzidine | 34631 | 91-94-1 | 028 B | 023 B | $d_{6}$ | 93951-91-8 | 228 B |
| diethyl phthalate | 34336 | 84-66-2 | 070 B | 024 B | $d_{4}$ | 93952-12-6 | 270 B |
| 2,4-dimethylphenol | 34606 | 105-67-9 | 034 B | 003 B | $d_{3}$ | 93951-75-8 | 234 B |
| dimethyl phthalate | 34341 | 131-11-3 | 071 B | 025 B | $d_{4}$ | 93951-89-4 | 271 B |
| 2,4-dinitrotoluene | 34611 | 121-14-2 | 035 B | 027 B | $d_{3}$ | 93951-68-9 | 235 B |
| 2,6-dinitrotoluene | 34626 | 606-20-2 | 036 B | 028 B | $d_{3}$ | 93951-90-7 | 236 B |
| di-n-octyl phthalate | 34596 | 117-84-0 | 069 B | 029 B | $d_{4}$ | 93952-13-7 | 269 B |
| diphenylamine (Appendix C) | 77579 | 122-39-4 | 507 B | | $d_{10}$ | 37055-51-9 | 607 B |

## Table 1 (Continued)
## Base/Neutral Extractable Compounds

| COMPOUND | POLLUTANT STORET | POLLUTANT CAS | POLLUTANT EGD | POLLUTANT NPDES | LABELED COMPOUND ANALOG | LABELED COMPOUND CAS | LABELED COMPOUND EGD |
|---|---|---|---|---|---|---|---|
| diphenyl ether (Appendix C) | 77587 | 101-84-8 | 508 B | | $d_{10}$ | 93952-05-7 | 608 B |
| 1,2-diphenylhydrazine | 34346 | 122-66-7 | 037 B | 030 B | $d_{10}$ | 93951-92-9 | 237 B |
| fluoranthene | 34376 | 206-44-0 | 039 B | 031 B | $d_{10}$ | 93951-69-0 | 231 B |
| fluorene | 34381 | 86-73-7 | 080 B | 032 B | $d_{10}$ | 81103-79-9 | 080 B |
| hexachlorobenzene | 39700 | 118-74-1 | 009 B | 033 B | $^{13}C_6$ | 93952-14-8 | 209 B |
| hexachlorobutadiene | 34391 | 87-68-3 | 052 B | 034 B | $^{13}C_4$ | 93951-70-3 | 252 B |
| hexachloroethane | 34396 | 67-72-1 | 012 B | 036 B | $^{13}C$ | 93952-15-9 | 212 B |
| hexachlorocyclopentadiene | 34386 | 77-47-4 | 053 B | 035 B | $^{13}C_4$ | 93951-71-4 | 253 B |
| ideno(1,2,3-cd)pyrene | 34403 | 193-39-5 | 083 B | 037 B | | | |
| isophorone | 34408 | 78-59-1 | 054 B | 038 B | $d_8$ | 93952-16-0 | 254 B |
| naphthalene | 34696 | 91-20-3 | 055 B | 039 B | $d_8$ | 1146-65-2 | 255 B |
| beta-naphthylamine (Appendix C) | 82553 | 91-59-8 | 056 B | 040 B | $d_7$ | 93951-94-1 | 602 B |
| nitrobenzene | 34447 | 98-95-3 | 056 B | 040 B | $d_5$ | 4165-60-0 | 256 B |
| N-nitrosodimethylamine | 34438 | 62-75-9 | 061 B | 041 B | $d_6$ | 17829-05-9 | 261 B |
| N-nitrosodi-n-proplylamine | 34428 | 621-64-7 | 063 B | 042 B | $d_{14}$ | 93951-96-3 | 263 B |
| N-nitrosodiphenylamine | 34433 | 86-30-6 | 062 B | 043 B | $d_6$ | 93951-95-2 | 262 B |
| phenanthrene | 34461 | 85-01-8 | 081 B | 044 B | $d_{10}$ | 1517-22-2 | 281 B |
| phenol | 34694 | 108-95-2 | 065 B | 010 B | $d_5$ | 4165-62-2 | 265 B |
| alpha-picoline (Synfuel) | 77088 | 109-06-8 | 503 B | | $d_7$ | 93951-93-0 | 503 B |
| pyrene | 34469 | 129-00-0 | 084 B | 045 B | $d_{10}$ | 1718-52-1 | 284 B |
| styrene (Appendix C) | 77128 | 100-42-5 | 510 B | | $d_5$ | 5161-29-5 | 610 B |
| alpha-terpineol (Appendix C) | 77493 | 98-55-5 | 509 B | | $d_3$ | 93952-06-8 | 609 B |
| 1,2,3-trichlorobenzene (4c) | 77613 | 87-61-6 | 529 B | | $d_3$ | 3907-98-0 | 629 B |
| 1,2,4-trichlorobenzene | 34551 | 120-82-1 | 008 B | 046 B | $d_3$ | 93952-16-0 | 208 B |

## TABLE 2
## Acid Extractable Compounds

| COMPOUND | POLLUTANT STORET | POLLUTANT CAS | POLLUTANT EGD | POLLUTANT NPDES | LABELED COMPOUND ANALOG | LABELED COMPOUND CAS | LABELED COMPOUND EGD |
|---|---|---|---|---|---|---|---|
| 4-chloro-3-methylphenol | 34452 | 59-50-7 | 022 A | 008 A | $d_2$ | 93951-72-5 | 222 A |
| 2-chlorophenol | 34586 | 95-57-8 | 024 A | 001 A | $d_4$ | 93951-73-6 | 224 A |
| 2,4-dichlorophenol | 34601 | 120-83-2 | 031 A | 002 A | $d_3$ | 93951-74-7 | 231 A |
| 2,4-dinitrophenol | 34616 | 51-28-5 | 059 A | 005 A | $d_3$ | 93951-77-0 | 259 A |
| 2-methyl-4,6-dinitrophenol | 34657 | 534-52-1 | 060 A | 004 A | $d_2$ | 93951-76-9 | 260 A |
| 2-nitrophenol | 34591 | 88-75-5 | 057 A | 006 A | $d_4$ | 93951-75-1 | 257 A |
| 4-nitrophenol | 34646 | 100-02-7 | 058 A | 007 A | $d_4$ | 93951-79-2 | 258 A |
| pentachlorophenol | 39032 | 87-86-5 | 064 A | 009 A | $^{13}C_6$ | 85380-74-1 | 264 A |
| 2,3,6-trichlorophenol (4c) | 77688 | 933-75-5 | 530 A | | $d_2$ | 93951-81-6 | 630 A |
| 2,4,5-trichlorophenol (4c) | | 95-95-4 | 531 A | | $d_2$ | 93951-82-7 | 631 A |
| 2,4,6-trichlorophenol | 34621 | 88-06-2 | 021 A | 011 A | $d_2$ | 93951-80-5 | 221 A |

1 3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations The limits listed in tables 3 and 4 represent the minimum quantity that can be detected with no interferences present

1 4 The GC/MS portions of this method are for use only by analysts experienced with GC/MS or under the close supervision of such qualified persons Laboratories unfamiliar with the analyses of environmental samples by GC/MS should run the performance tests in reference 1 before beginning

## 2 SUMMARY OF METHOD

2 1 Stable isotopically labeled analogs of the compounds of interest are added to a one liter wastewater sample The sample is extracted at pH 12-13, then at pH <2 with methylene chloride using continuous extraction techniques The extract is dried over sodium sulfate and concentrated to a volume of one mL An internal standard is added to the extract, and the extract is injected into the gas chromatograph (GC) The compounds are separated by the GC and detected by a mass spectrometer (MS) The labeled compounds serve to correct the variability of the analytical technique

2 2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and the background corrected characteristic spectral masses with those of authentic standards

2 3 Quantitative analysis is performed by GC/MS using extracted ion current profile (EICP) areas Isotope dilution is used when labeled compounds are available, otherwise, an internal or external standard method is used

2 4 Quality is assured through reproducible calibration and testing of the extraction and GC/MS systems

## 3 CONTAMINATION AND INTERFERENCES

3 1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms and spectra All materials shall be demonstrated to be free from interferences under the conditions of the analysis by running method blanks initially and with each sample lot (samples started through the extraction process on a given 8 hr shift, to a maximum of 20) Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required Glassware and, where possible, reagents are cleaned by solvent rinse and baking at 450 °C for one hour minimum

3 2 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being sampled

**TABLE 3**

**Gas Chromatography of Base/Neutral Extractable Compounds**

| EGD NO (1) | COMPOUND | RETENTION TIME MEAN (SEC) | EGD REF | RELATIVE | DETECTION LIMIT (2) (μg/L) |
|---|---|---|---|---|---|
| 164 | 2,2'-difluorobiphenyl (internal standard) | 1163 | 164 | 1 000-1 000 | 10 |
| 061 | N-nitrosodimethylamine* | 385 | 164 | 0 264-0 398 | 50 |
| 603 | alpha picoline-$d_7$ | 417 | 164 | 0 326-0 393 | 50 |
| 703 | alpha picoline | 426 | 603 | 1 006-1 028 | 50 |
| 610 | styrene-$d_5$ | 546 | 164 | 0 450-0 488 | 10 |
| 710 | styrene | 549 | 610 | 1 002-1 009 | 10 |
| 613 | p-cymene-$d_{14}$ | 742 | 164 | 0 624-0 652 | 10 |
| 713 | p-cymene | 755 | 613 | 1 008-1 023 | 10 |
| 265 | phenol-$d_5$ | 696 | 164 | 0 584-0 613 | 10 |
| 365 | phenol | 700 | 265 | 0 995-1 010 | 10 |
| 218 | bis(2-chloroethyl) ether-$d_8$ | 696 | 164 | 0 584-0 607 | 10 |
| 318 | bis(2-chloroethyl) ether | 704 | 218 | 1 007-1 016 | 10 |
| 617 | n-decane-$d_{22}$ | 698 | 164 | 0 585-0 615 | 10 |
| 717 | n-decane | 720 | 617 | 1 022-1 038 | 10 |
| 266 | 1,3-dichlorobenzene-$d_4$ | 722 | 164 | 0 605-0 636 | 10 |
| 326 | 1,3-dichlorobenzene | 724 | 226 | 0 998-1 008 | 10 |
| 227 | 1,4-dichlorobenzene-$d_4$ | 737 | 164 | 0 601-0 666 | 10 |
| 327 | 1,4-dichlorobenzene | 740 | 227 | 0 997-1 009 | 10 |
| 225 | 1,2-dichlorobenzene-$d_4$ | 758 | 164 | 0 632-0 667 | 10 |
| 325 | 1,2-dichlorobenzene | 760 | 225 | 0 995-1 008 | 10 |
| 242 | bis(2-chloroisopropyl) ether-$d_{12}$ | 788 | 164 | 0 664-0 691 | 10 |
| 342 | bis(2-chloroisopropyl) ether | 799 | 242 | 1 010-1 016 | 10 |
| 212 | hexachloroethane-$^{13}C$ | 819 | 164 | 0 690-0 717 | 10 |
| 312 | hexachloroethane | 823 | 212 | 0 999-1 001 | 10 |
| 063 | N-nitrosodi-n-propylamine* | 830 | 164 | 0 701-0 721 | 20* |
| 256 | nitrobenzene-$d_5$ | 845 | 164 | 0 706-0 727 | 10 |
| 356 | nitrobenzene | 849 | 256 | 1 002-1 007 | 10 |
| 254 | isophorone-$d_8$ | 881 | 164 | 0 747-0 767 | 10 |
| 354 | isophorone | 889 | 254 | 0 999-1 017 | 10 |
| 234 | 2,4-dimethylphenol-$d_3$ | 921 | 164 | 0 781-0 803 | 10 |
| 334 | 2,4-dimethylphenol | 924 | 234 | 0 999-1 003 | 10 |
| 043 | bis(2-chloroethoxy) methane* | 939 | 164 | 0 799-0 815 | 10 |
| 208 | 1,2,4-trichlorobenzene-$d_3$ | 955 | 164 | 0 813-0 830 | 10 |
| 308 | 1,2,4-trichlorobenzene | 958 | 208 | 1 000-1 005 | 10 |
| 255 | naphthalene-$d_8$ | 963 | 164 | 0 819-0 836 | 10 |
| 355 | naphthalene | 967 | 255 | 1 001-1 006 | 10 |
| 609 | alpha-terpineol-$d_3$ | 973 | 164 | 0 829-0 844 | 10 |
| 709 | alpha-terpineol | 975 | 609 | 0 998-1 008 | 10 |
| 606 | n-dodecane-$d_{26}$ | 953 | 164 | 0 730-0 908 | 10 |
| 706 | n-dodecane | 981 | 606 | 0 986-1 051 | 10 |
| 529 | 1,2,3-trichlorobenzene* | 1003 | 164 | 0 855-0 870 | 10 |
| 252 | hexachlorobutadiene-$^{13}C_4$ | 1005 | 164 | 0 856-0 871 | 10 |
| 352 | hexachlorobutadiene | 1006 | 252 | 0 999-1 002 | 10 |
| 253 | hexachlorocyclopentadiene-$^{13}C_4$ | 1147 | 164 | 0 976-0 986 | 10 |
| 353 | hexachlorocyclopentadiene | 1142 | 253 | 0 999-1 001 | 10 |
| 220 | 2-chloronaphthalene-$d_7$ | 1185 | 164 | 1 014-1 024 | 10 |
| 320 | 2-chloronaphthalene | 1200 | 220 | 0 997-1 007 | 10 |
| 518 | n-tetradecane* | 1203 | 164 | 1 015-1 026 | 10 |
| 612 | biphenyl-$d_{10}$ | 1205 | 164 | 1 016-1 027 | 10 |
| 712 | biphenyl | 1195 | 612 | 1 001-1 006 | 10 |
| 608 | diphenyl ether-$d_{10}$ | 1211 | 164 | 1 036-1 047 | 10 |
| 708 | diphenyl ether | 1216 | 608 | 0 997-1 009 | 10 |
| 277 | acenaphthylene-$d_8$ | 1265 | 164 | 1 080-1 095 | 10 |

## TABLE 3 (Continued)
## Gas Chromatography of Base/Neutral Extractable Compounds

| EGD NO (1) | COMPOUND | RETENTION TIME MEAN (SEC) | EGD REF | RELATIVE | DETECTION LIMIT (2) (µg/L) |
|---|---|---|---|---|---|
| 377 | acenaphthylene | 1247 | 277 | 1 000-1 004 | 10 |
| 271 | dimethyl phthalate-$d_4$ | 1269 | 164 | 1 083-1 102 | 10 |
| 371 | dimethyl phthalate | 1273 | 271 | 0 998-1 005 | 10 |
| 236 | 2,6-dinitrotoluene-$d_3$ | 1283 | 164 | 1 090-1 112 | 10 |
| 336 | 2,6-dinitrotoluene | 1300 | 236 | 1 001-1 005 | 10 |
| 201 | acenaphthene-$d_{10}$ | 1298 | 164 | 1 107-1 125 | 10 |
| 301 | acenaphthene | 1304 | 201 | 0 999-1 009 | 10 |
| 605 | dibenzofuran-$d_8$ | 1331 | 164 | 1 134-1 155 | 10 |
| 705 | dibenzofuran | 1335 | 605 | 0 998-1 007 | 10 |
| 602 | beta-naphthylamine-$d_7$ | 1368 | 164 | 1 163-1 189 | 50 |
| 702 | beta-naphthylamine | 1371 | 602 | 0 996-1 007 | 50 |
| 280 | fluorene-$d_{10}$ | 1395 | 164 | 1 185-1 214 | 10 |
| 380 | fluorene | 1401 | 281 | 0 999-1 008 | 10 |
| 240 | 4-chlorophenyl phenyl ether-$d_5$ | 1406 | 164 | 1 194-1 223 | 10 |
| 340 | 4-chlorophenyl phenyl ether | 1409 | 240 | 0 990-1 015 | 10 |
| 270 | diethyl phthalate-$d_4$ | 1409 | 164 | 1 197-1 229 | 10 |
| 370 | diethyl phthalate | 1414 | 270 | 0 996-1 006 | 10 |
| 619 | n-hexadecane-$d_{34}$ | 1447 | 164 | 1 010-1 478 | 10 |
| 719 | n-hexadecane | 1469 | 619 | 1 013-1 020 | 10 |
| 235 | 2,4-dinitrotoluene-$d_3$ | 1359 | 164 | 1 152-1 181 | 10 |
| 335 | 2,4-dinitrotoluene | 1344 | 235 | 1 000-1 002 | 10 |
| 237 | 1,2-diphenylhydrazine-$d_8$ | 1433 | 164 | 1 216-1 248 | 20 |
| 337 | 1,2-diphenylhydrazine (3) | 1439 | 237 | 0 999-1 009 | 20 |
| 607 | diphenylamine-$d_{10}$ | 1437 | 164 | 1 213-1 249 | 20 |
| 707 | diphenylamine | 1439 | 607 | 1 000-1 007 | 20 |
| 262 | N-nitrosodiphenylamine-$d_6$ | 1447 | 164 | 1 225-1 252 | 20 |
| 362 | N-nitrosodiphenylamine (4) | 1464 | 262 | 1 000-1 002 | 20 |
| 041 | 4-bromophenyl phenyl ether | 1498 | 164 | 1 271-1 307 | 10 |
| 209 | hexachlorobenzene-$^{13}C_6$ | 1521 | 164 | 1 288-1 327 | 10 |
| 309 | hexachlorobenzene | 1522 | 209 | 0 999-1 001 | 10 |
| 281 | phenanthrene-$d_{10}$ | 1578 | 164 | 1 334-1 380 | 10 |
| 520 | n-octadecane* | 1580 | 164 | 1 335-1 381 | 10 |
| 381 | phenanthrene | 1583 | 281 | 1 000-1 005 | 10 |
| 278 | anthracene-$d_{10}$ | 1588 | 164 | 1 342-1 388 | 10 |
| 378 | anthracene | 1592 | 278 | 0 998-1 006 | 10 |
| 604 | dibenzothiophene-$d_8$ | 1559 | 164 | 1 314-1 361 | 10 |
| 704 | dibenzothiophene | 1564 | 604 | 1 000-1 006 | 10 |
| 528 | carbazole* | 1650 | 164 | 1 180-1 660 | 20 |
| 621 | n-eicosane-$d_{42}$ | 1655 | 164 | 1 184-1 662 | 10 |
| 721 | n-eicosane | 1677 | 621 | 1 010-1 021 | 10 |
| 268 | di-n-butyl phthalate-$d_4$ | 1719 | 164 | 1 446-1 510 | 10 |
| 368 | di-n-butyl phthalate | 1723 | 268 | 1 000-1 003 | 10 |
| 239 | fluoranthene-$d_{10}$ | 1813 | 164 | 1 522-1 596 | 10 |
| 339 | fluoranthene | 1817 | 239 | 1 000-1 004 | 10 |
| 284 | pyrene-$d_{10}$ | 1844 | 164 | 1 523-1 644 | 10 |
| 384 | pyrene | 1852 | 284 | 1 001-1 003 | 10 |
| 205 | benzidine-$d_8$ | 1854 | 164 | 1 549-1 632 | 50 |
| 305 | benzidine | 1853 | 205 | 1 000-1 002 | 50 |
| 522 | n-docosane* | 1889 | 164 | 1 578-1 671 | 10 |
| 623 | n-tetracosane-$d_{50}$ | 1997 | 164 | 1 671-1 764 | 10 |
| 723 | n-tetracosane | 2025 | 612 | 1 012-1 015 | 10 |
| 067 | butylbenzyl phthalate* | 2060 | 164 | 1 724-1 818 | 10 |
| 276 | chrysene-$d_{12}$ | 2081 | 164 | 1 743-1 837 | 10 |
| 376 | chrysene | 2083 | 276 | 1 000-1 004 | 10 |
| 272 | benzo(a)anthracene-$d_{12}$ | 2082 | 164 | 1 735-1 846 | 10 |
| 372 | benzo(a)anthracene | 2090 | 272 | 0 999-1 007 | 10 |
| 228 | 3,3'-dichlorobenzidine-$d_6$ | 2088 | 164 | 1 744-1 848 | 50 |
| 328 | 3,3'-dichlorobenzidine | 2086 | 228 | 1 000-1 001 | 50 |
| 266 | bis(2-ethylhexyl) phthalate-$d_4$ | 2123 | 164 | 1 771-1 880 | 10 |
| 366 | bis(2-ethylhexyl) phthalate | 2124 | 266 | 1 000-1 002 | 10 |
| 524 | n-hexacosane* | 2147 | 164 | 1 791-1 901 | 10 |
| 269 | di-n-octyl phthalate-$d_4$ | 2239 | 164 | 1 867-1 982 | 10 |
| 369 | di-n-octyl phthalate | 2240 | 269 | 1 000-1 002 | 10 |
| 525 | n-octacosane* | 2272 | 164 | 1 880-2 004 | 10 |

## TABLE 3 (Continued)
## Gas Chromatography of Base/Neutral Extractable Compounds

| EGD NO (1) | COMPOUND | RETENTION TIME MEAN (SEC) | EGD REF | RELATIVE | DETECTION LIMIT (2) (µg/L) |
|---|---|---|---|---|---|
| 274 | benzo(b)fluoranthene-$d_{12}$ | 2281 | 164 | 1 902-2 025 | 10 |
| 354 | benzo(b)fluoranthene | 2293 | 274 | 1 000-1 005 | 10 |
| 275 | benzo(k)fluoranthene-$d_{12}$ | 2287 | 164 | 1 906-2 033 | 10 |
| 375 | benzo(k)fluoranthene | 2293 | 275 | 1 000-1 005 | 10 |
| 273 | benzo(a)pyrene-$d_{12}$ | 2351 | 164 | 1 954-2 088 | 10 |
| 373 | benzo(a)pyrene | 2350 | 273 | 1 000-1 004 | 10 |
| 626 | n-triacontane-$d_{62}$ | 2384 | 164 | 1 972-2 127 | 10 |
| 726 | n-triacontane | 2429 | 626 | 1 011-1 028 | 10 |
| 083 | indeno(1,2 3-cd)pyrene* | 2650 | 164 | 2 119-2 356 | 20 |
| 082 | dibenzo(a,h)anthracene* | 2660 | 164 | 2 121-2 358 | 20 |
| 279 | benzo(ghi)perylene-$d_{12}$ | 2741 | 164 | 2 187-2 524 | 20 |
| 379 | benzo(ghi)perylene | 2750 | 279 | 1 001-1 006 | 20 |

(1) Reference numbers beginning with 0 1 or 5 indicate a pollutant quantified by the internal standard method, reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution

(2) This is a minimum level at which the entire GC/MS system must give recognizable mass spectra (background corrected) and acceptable calibration points

(3) detected as azobenzene

(4) detected as diphenylamine

* specification derived from related compound

Column 30 ± 2 m x 0 25 ± 0 02 mm i d 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary

Temperature program 5 min at 30°C 30 – 280°C at 8°C per min isothermal at 280°C until benzo(ghi)perylene elutes

Gas velocity 30 ± 5 cm/sec

## TABLE 4
## Gas Chromatography of Acid Extractable Compounds

| EGD NO (1) | COMPOUND | RETENTION TIME MEAN (SEC) | EGD REF | RELATIVE | DETECTION LIMIT (2) (µg/L) |
|---|---|---|---|---|---|
| 164 | 2,2'-difluorobiphenyl (internal standard) | 1163 | 164 | 1 000-1 000 | 10 |
| 224 | 2-chlorophenol-$d_4$ | 701 | 164 | 0 587-0 618 | 10 |
| 324 | 2-chlorophenol | 705 | 224 | 0 997-1 010 | 10 |
| 257 | 2-nitrophenol-$d_4$ | 898 | 164 | 0 761-0 783 | 20 |
| 357 | 2-nitrophenol | 900 | 257 | 0 994-1 009 | 20 |
| 231 | 2 4-dichlorophenol-$d_3$ | 944 | 164 | 0 802-0 822 | 10 |
| 331 | 2 4-dichlorophenol | 947 | 231 | 0 997-1 006 | 10 |
| 222 | 4-chloro-3-methylphenol-$d_2$ | 1086 | 164 | 0 930-0 943 | 10 |
| 322 | 4-chloro-3-methylphenol | 1091 | 222 | 0 998-1 003 | 10 |
| 221 | 2,4 6-trichlorophenol-$d_2$ | 1162 | 164 | 0 994-1 005 | 10 |
| 321 | 2,4,6-trichlorophenol | 1165 | 221 | 0 998-1 004 | 10 |
| 531 | 2,4,5-trichlorophenol* | 1170 | 164 | 0 996-1 016 | 10 |
| 530 | 2,3,6-trichlorophenol* | 1195 | 164 | 1 016-1 140 | 10 |
| 259 | 2,4-dinitrophenol-$d_3$ | 1323 | 164 | 1 127-1 149 | 50 |
| 359 | 2,4-dinitrophenol | 1325 | 259 | 1 000-1 005 | 50 |
| 258 | 4-nitrophenol-$d_4$ | 1349 | 164 | 1 147-1 175 | 50 |
| 358 | 4-nitrophenol | 1354 | 258 | 0 997-1 006 | 50 |
| 260 | 2-methyl-4,6-dinitrophenol-$d_2$ | 1433 | 164 | 1 216-1 249 | 20 |
| 360 | 2-methyl-4,6-dinitrophenol | 1435 | 260 | 1 000-1 002 | 20 |
| 264 | pentachlorophenol-$^{13}C_6$ | 1559 | 164 | 1 320-1 363 | 50 |
| 364 | pentachlorophenol | 1561 | 264 | 0 998-1 002 | 50 |

(1) Reference numbers beginning with 0 1 or 5 indicate a pollutant quantified by the internal standard method, reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method, reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution

(2) This is a minimum level at which the entire GC/MS system must give recognizable mass spectra (background corrected) and acceptable calibration points

* specification derived from related compound

Column 30 ± 2 m x 0 25 ± 0 02 mm i d 94% methyl 4% phenyl, 1% vinyl bonded phase fused silica capillary

Temperature prog am 5 min at 30°C 30 – 250°C or until pentachlorophenol elutes

Gas velocity 30 ± cm/sec

## 4 SAFETY

4 1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined, however, each chemical compound should be treated as a potential health hazard Exposure to these compounds should be reduced to the lowest possible level 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 data handling sheets should also be made available to all personnel involved in these analyses Additional information on laboratory safety can be found in references 2-4

4 2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens benzo(a)anthracene, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, N-nitrosodimethylamine, and beta-naphthylamine Primary standards of these compounds shall be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled

## 5 APPARATUS AND MATERIALS

5 1 Sampling equipment for discrete or composite sampling

5 1 1 Sample bottle, amber glass, 1 1 liters minimum If amber bottles are not available, samples shall be protected from light Bottles are detergent water washed, then solvent rinsed or baked at 450°C for one hour minumum before use

5 1 2 Bottle caps—threaded to fit sample bottles Caps are lined with Teflon Aluminum foil may be substituted if the sample is not corrosive Liners are detergent water washed, then reagent water (section 6 5) and solvent rinsed, and baked at approximately 200°C for one hour minumum before use

5 1 3 Compositing equipment—automatic or manual compositing system incorporating glass containers for collection of a minimum 1 1 liters Sample containers are kept at 0 to 4°C during sampling Glass or Teflon tubing only shall be used If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only Before use, the tubing is thoroughly rinsed with methanol, followed by repeated rinsings with reagent water (section 6 5) to minimize sample contamination An integrating flow meter is used to collect proportional composite samples

5 2 Continuous liquid-liquid extractor—Teflon or glass connecting joints and stopcocks without lubrication (Hershberg-Wolf Extractor) one liter capacity, Ace Glass 6841-10, or equivalent

5 3 Drying column—15 to 20 mm i d Pyrex chromatographic column equipped with coarse glass frit or glass wool plug

5 4 Kuderna-Danish (K-D) apparatus

5 4 1 Concentrator tube—10mL, graduated (Kontes K-570050-1025, or equivalent) with calibration verified Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts

5 4 2 Evaporation flask—500 mL (Kontes K-570001-0500, or equivalent), attached to concentrator tube with springs (Kontes K-662750-0012)

5 4 3 Snyder column—three ball macro (Kontes K-503000-0232, or equivalent)

5 4 4 Snyder column—two ball micro (Kontes K-469002-0219, or equivalent)

5 4 5 Boiling chips—approx 10/40 mesh, extracted with methylene chloride and baked at 450°C for one hr minimum

5 5 Water bath—heated, with concentric ring cover, capable of temperature control (± 2°C), installed in a fume hood

5 6 Sample vials—amber glass, 2-5 mL with Teflon-lined screw cap

5 7 Analytical balance—capable of weighing 0 1 mg

5 8 Gas chromatograph—shall have splitless or on-column injection port for capillary column, temperature program with 30°C hold, and shall meet all the performance specifications in section 12

5 8 1 Column—30 ± 5 m x 0 25 ± 0 02 mm i d 5% phenyl, 94% methyl, 1% vinyl silicone bonded phase fused silica capillary column (J & W DB-5, or equivalent)

5 9 Mass spectrometer—70 eV electron impact ionization, shall repetitively scan from 35 to 450 amu in 0 95 to 1 00 second and shall produce a unit resolution (valleys between m/z 441-442 less than 10 percent of the height of the 441 peak), background corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet The spectrum shall meet the mass-intensity criteria in table 5 (reference 5) The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within one centimeter of the ion source but does not intercept the electron or ion beams All portions of the column which connect the GC to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds

**TABLE 5**

**DFTPP Mass-intensity Specifications**

| MASS | INTENSITY REQUIRED |
|---|---|
| 51 | 8-82 percent of mass 198 |
| 68 | <2 percent of mass 69 |
| 69 | 11-91 percent of mass 198 |
| 70 | <2 percent of mass 69 |
| 127 | 32-59 percent of mass 198 |
| 198 | base peak, 100 percent abundance |
| 199 | 4-9 percent of mass 198 |
| 275 | 11-30 percent of mass 198 |
| 441 | 44-110 percent of mass 443 |
| 442 | 30-86 percent of mass 198 |
| 443 | 14-24 percent of mass 442 |

5 10 Data system—shall collect and record MS data, store mass intensity data in spectral libraries, process GC/MS data, generate reports, and shall compute and record response factors

5 10 1 Data acquisition—mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device

5 10 2 Mass spectral libraries—user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GC/MS runs for the compounds of interest (section 7 2)

5 10 3 Data processing—the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GC/MS analysis Software routines shall be employed to compute retention times and peak areas Displays of spectra, mass chromatograms, and library comparisons are required to verify results

5 10 4 Response factors and multipoint calibrations—the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multipoint calibration curves (section 7) Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity Statistics on initial (section 8 4) and ongoing (section 12 7) performance shall be computed and maintained

## 6 REAGENTS AND STANDARDS

6 1 Sodium hydroxide—reagent grade, 6N in reagent water

6 2 Sulfuric acid—reagent grade, 6N in reagent water

6 3 Sodium sulfate—reagent grade, granular anhydrous, rinsed with methylene chloride (20 mL/g) and conditioned at 450°C for one hour minimum

6 4 Methylene chloride—distilled in glass (Burdick and Jackson, or equivalent)

6 5 Reagent water—water in which the compounds of interest and interfering compounds are not detected by this method

6 6 Standard solutions—purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition If compound purity is 96 percent or greater, the weight may be used without correction to compute the concentration of the standard When not being used, standards are stored in the dark at −20 to −10°C in screw-capped vials with Teflon-lined lids A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected The vials are brought to room temperature prior to use Any precipitate is redissolved and solvent is added if solvent loss has occurred

6 7 Preparation of stock solutions—prepare in methylene chloride, benzene, p-dioxane, or a mixture of these solvents per the steps below Observe the safety precautions given in section 4 The large number of labeled and unlabeled acid, base/neutral, and Appendix C compounds used for combined calibration (section 7) and calibration verification (12 5) require high concentrations (approx 40 mg/mL) when individual stock solutions are prepared, so that dilutions of mixtures will permit calibration with all compounds in a single set of solutions The working range for most compounds is 10-200 $\mu$g/mL Compounds with a reduced MS response may be prepared at higher concentrations

6 7 1 Dissolve an appropriate amount of assayed reference material in a suitable solvent For example, weigh 400 mg naphthalene in a 10 mL ground glass stoppered volumetric flask and fill to the mark with benzene After the naphthalene is completely dissolved, transfer the solution to a 15 mL vial with Teflon-lined cap

6 7 2 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards Quality control check samples that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268

6 7 3 Stock standard solutions shall be replaced after six months, or sooner if comparison with quality control check samples indicates a change in concentration

6 8 Labeled compound spiking solution—from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution at a concentration of 200 $\mu$g/mL, or at a concentration appropriate to the MS response of each compound

6 9 Secondary standards—using stock solutions (section 6 7), prepare a secondary standard containing all of the compounds in tables 1 and 2 at a concentration of 400 $\mu$g/mL, or higher concentration appropriate to the MS response of the compound

6 10 Internal standard solution—prepare 2,2'-difluorobiphenyl (DFB) at a concentration of 10 mg/mL in benzene

6 11 DFTPP solution—prepare at 50 $\mu$g/mL in acetone

6 12 Solutions for obtaining authentic mass spectra (section 7 2)—prepare mixtures of compounds at concentrations which will assure authentic spectra are obtained for storage in libraries

6 13 Calibration solutions—combine 0 5 mL of the solution in section 6 8 with 25, 50, 125, 250, and 500 $\mu$L of the solution in section 6 9 and bring to 1 00 mL total volume each This will produce calibration solutions of nominal 10, 20, 50, 100 and 200 $\mu$g/mL of the pollutants and a constant nominal 100 $\mu$g/mL of the labeled compounds Spike each solution with 10 $\mu$L of the internal standard solution (section 6 10) These solutions permit the relative response (labeled to unlabeled) to be measured as a function of concentration (section 7 4)

6 14 Precision and recovery standard—used for determination of initial (section 8 2) and on-going (section 12 7) precision and recovery This solution shall contain the pollutants and labeled compounds at a nominal concentration of 100 $\mu$g/mL

6 15 Stability of solutions—all standard solutions (sections 6 8-6 14) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation Standards will remain acceptable if the peak area at the quantitation mass relative to the DFB internal standard remains within ±15 percent of the area obtained in the initial analysis of the standard

## 7 CALIBRATION

7 1 Assemble the GC/MS and establish operating conditions in table 3 Analyze standards per the procedure in section 11 to demonstrate that the analytical system meets the detection limits in tables 3 and 4, and the mass-intensity criteria in table 5 for 50 ng DFTPP

7 2 Mass spectral libraries—detection and identification of compounds of interest are dependent upon spectra stored in user created libraries

7 2 1 Obtain a mass spectrum of each pollutant, labeled compound, and the internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components That only a single compound is present is determined by examination of the spectrum Fragments not attributable to the compound under study indicate the presence of an interfering compound

7 2 2 Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic masses or introduce other distortion

7 2 3 The authentic reference spectrum is obtained under DFTPP tuning conditions (section 7 1 and table 5) to normalize it to spectra from other instruments

7 2 4 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10 percent of the base peak This edited spectrum is stored for reverse search and for compound confirmation

7 3 Analytical range—demonstrate that 20 ng anthracene or phenanthrene produces an area at m/z 178 approx one-tenth that required to exceed the linear range of the system The exact value must be determined by experience for each instrument It is used to match the calibration range of the instrument to the analytical range and detection limits required, and to diagnose instrument sensitivity problems (section 15 4) The 20 $\mu$g/mL calibration standard (section 6 13) can be used to demonstrate this performance

7 3 1 Polar compound detection—demonstrate that unlabeled pentachlorophenol and benzidine are detectable at the 50 $\mu$g/mL level (per all criteria in section 13) The 50 $\mu$g/mL calibration standard (section 6 13) can be used to demonstrate this performance

7 4 Calibration with isotope dilution—isotope dilution is used when 1) labeled compounds are available, 2) interferences do not preclude its use, and 3) the quantitation m/z (tables 6 and 7) extracted ion current profile (EICP) area for the compound is in the calibration range If any of these conditions preclude isotope dilution, the internal standard method (section 7 5) is used

### TABLE 6
### Base/Neutral Extractable Compound Characteristic Masses

| COMPOUND | LABELED ANALOG | PRIMARY M/Z'S |
|---|---|---|
| acenaphthene | $d_{10}$ | 154/164 |
| acenaphthylene | $d_8$ | 152/160 |
| anthracene | $d_{10}$ | 178/188 |
| benzidine | $d_8$ | 184/192 |
| benzo(a)anthracene | $d_{12}$ | 228/240 |
| benzo(b)fluoranthene | $d_{12}$ | 252/264 |
| benzo(k)fluoranthene | $d_{12}$ | 252/264 |
| benzo(a)pyrene | $d_{12}$ | 252/264 |
| benzo(ghi)perylene | $d_{12}$ | 276/288 |
| biphenyl | $d_{10}$ | 154/164 |
| bis(2-chloroethyl) ether | $d_8$ | 93/101 |
| bis(2-chloroethoxy) methane | | 93 |
| bis(2-chloroisopropyl) ether | $d_{12}$ | 121/131 |
| bis(2-ethylhexyl) phthalate | $d_4$ | 149/153 |
| 4-bromophenyl phenyl ether | | 248 |
| butyl benzyl phthalate | | 149 |
| n-$C_{10}$ | $d_{22}$ | 55/66 |
| n-$C_{12}$ | $d_{26}$ | 55/66 |
| n-$C_{14}$ | | 55 |
| n-$C_{16}$ | $d_{34}$ | 55/66 |
| n-$C_{18}$ | | 55 |
| n-$C_{20}$ | $d_{42}$ | 55/66 |
| n-$C_{22}$ | | 55 |
| n-$C_{24}$ | $d_{50}$ | 55/66 |
| n-$C_{26}$ | | 55 |
| n-$C_{28}$ | | 55 |
| n-$C_{30}$ | $d_{62}$ | 55/66 |
| carbazole | $d_8$ | 167/175 |
| 2-chloronaphthalene | $d_7$ | 162/169 |
| 4-chlorophenyl phenyl ether | $d_5$ | 204/209 |
| chrysene | $d_{12}$ | 228/240 |
| p-cymene | $d_{14}$ | 119/130 |
| dibenzo(a,h)anthracene | | 278 |
| dibenzofuran | $d_8$ | 168/176 |
| dibenzothiophene | $d_8$ | 184/192 |
| di-n-butyl phthalate | $d_4$ | 149/153 |
| 1,2-dichlorobenzene | $d_4$ | 146/152 |
| 1,3-dichlorobenzene | $d_4$ | 146/152 |
| 1,4-dichlorobenzene | $d_4$ | 146/152 |
| 3,3'-dichlorobenzidine | $d_6$ | 252/258 |
| diethyl phthalate | $d_4$ | 149/153 |
| 2,4-dimethylphenol | $d_3$ | 122/125 |
| dimethyl phthalate | $d_4$ | 163/167 |
| 2,4-dinitrotoluene | $d_3$ | 165/168 |
| 2,6-dinitrotoluene | $d_3$ | 165/167 |
| di-n-octyl phthalate | $d_4$ | 149/153 |
| diphenylamine | $d_{10}$ | 169/179 |
| diphenyl ether | $d_{10}$ | 170/180 |
| 1,2-diphenylhydrazine* | $d_{10}$ | 77/82 |
| fluoranthene | $d_{10}$ | 202/212 |
| fluorene | $d_{10}$ | 166/176 |
| hexachlorobenzene | $^{13}C_6$ | 284/292 |
| hexachlorobutadiene | $^{13}C_4$ | 225/231 |
| hexachloroethane | $^{13}C$ | 201/204 |
| hexachlorocyclopentadiene | $^{13}C_4$ | 237/241 |
| ideno(1,2,3-cd)pyrene | | 276 |
| isophorone | $d_8$ | 82/88 |
| naphthalene | $d_8$ | 128/136 |
| beta-naphthylamine | $d_7$ | 143/150 |
| nitrobenzene | $d_5$ | 123/128 |
| N-nitrosodimethylamine | | 74 |
| N-nitrosodi-n-propylamine | | 70 |
| N-nitrosodiphenylamine** | $d_6$ | 169/175 |
| phenanthrene | $d_{10}$ | 178/188 |

## TABLE 6 (Continued)
## Base/Neutral Extractable Compound Characteristic Masses

| COMPOUND | LABELED ANALOG | PRIMARY M/Z'S |
|---|---|---|
| phenol | $d_5$ | 94/71 |
| alpha-picoline | $d_7$ | 93/100 |
| pyrene | $d_{10}$ | 202/212 |
| styrene | $d_5$ | 104/109 |
| alpha-terpineol | $d_3$ | 59/62 |
| 1,2,3-trichlorobenzene | $d_3$ | 180/183 |
| 1,2,4-trichlorobenzene | $d_3$ | 180/183 |

*detected as azobenzene
**detected as diphenylamine

## TABLE 7
## Acid Extractable Compound Characteristic Masses

| COMPOUND | LABELED ANALOG | PRIMARY M/Z'S |
|---|---|---|
| 4-chloro-3-methylphenol | $d_2$ | 107/109 |
| 2-chlorophenol | $d_4$ | 128/132 |
| 2,4-dichlorophenol | $d_3$ | 162/167 |
| 2,4-dinitrophenol | $d_3$ | 184/187 |
| 2-methyl-4,6-dinitrophenol | $d_2$ | 198/200 |
| 2-nitrophenol | $d_4$ | 139/143 |
| 4-nitrophenol | $d_4$ | 139/143 |
| pentachlorophenol | $^{13}C_6$ | 266/272 |
| 2,3,6-trichlorophenol | $d_2$ | 196/200 |
| 2,4,5-trichlorophenol | $d_2$ | 196/200 |
| 2,4,6-trichlorophenol | $d_2$ | 196/200 |

7 4 1 A calibration curve encompassing the concentration range is prepared for each compound determined The relative response (pollutant to labeled) vs concentration in standard solutions is plotted or computed using a linear regression The example in figure 1 shows a calibration curve for phenol using

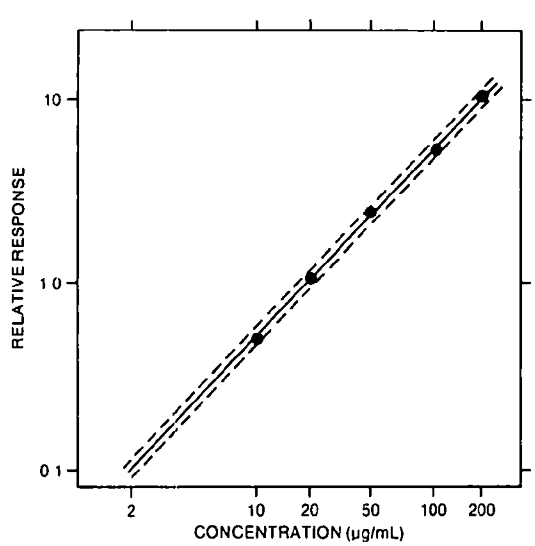


**FIGURE 1 Relative Response Calibration Curve for Phenol. The Dotted Lines Enclose a ± 10 Percent Error Window**

phenol-$d_5$ as the isotopic diluent Also shown are the ±10 percent error limits (dotted lines) Relative Response (RR) is determined according to the procedures described below A minumum of five data points are employed for calibration

7 4 2 The relative response of a pollutant to its labeled analog is determined from isotope ratio values computed from acquired data Three isotope ratios are used in this process

$R_x$ = the isotope ratio measured for the pure pollutant

$R_y$ = the isotope ratio measured for the labeled compound

$R_m$ = the isotope ratio of an analytical mixture of pollutant and labeled compounds

The m/z's are selected such that $R_x > R_y$ If $R_m$ is not between $2R_y$ and $0\ 5R_x$, the method does not apply and the sample is analyzed by the internal standard method (section 7 5)

7 4 3 Capillary columns usually separate the pollutant-labeled pair, with the labeled compound eluted first (figure 2) For this case,

$$R_x = \frac{[\text{area } m_1/z]}{1},$$

at the retention time of the pollutant ($RT_2$)

$$R_y = \frac{1}{[\text{area } m_2/z]},$$

at the retention time of the labeled compound ($RT_1$)

$$R_m = \frac{[\text{area } m_1/z \text{ (at } RT_2)]}{[\text{area } m_2/z \text{ (at } RT_1)]},$$

as measured in the mixture of the pollutant and labeled compounds (figure 2), and RR = $R_m$

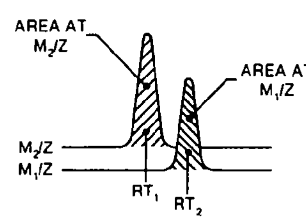


**FIGURE 2 Extracted Ion Current Profiles for Chromatographically Resolved Labeled ($m_2/z$) and Unlabeled ($m_1/z$) Pairs.**

7 4 4 Special precautions are taken when the pollutant-labeled pair is not separated, or when another labeled compound with interfering spectral masses overlaps the pollutant (a case which can occur with isomeric compounds) In this case, it is necessary to determine the respective contributions of the pollutant and labeled compounds to the respective EICP areas If the peaks are separated well enough to permit the data system or operator to remove the contributions of the compounds to each other, the equations in section 7 4 3 apply This usually occurs when the height of the valley between the two GC peaks at the same m/z is less than 10 percent of the height of the shorter of the two peaks If significant

GC and spectral overlap occur, RR is computed using the following equation

$$RR = \frac{(R_y - R_m)(R_x + 1)}{(R_m - R_x)(R_y + 1)}$$

where $R_x$ is measured as shown in figure 3A, $R_y$ is measured as shown in figure 3B, and $R_m$ is measured as shown in figure 3C For the example,

$$R_x = \frac{46100}{4780} = 9\ 644$$

$$R_y = \frac{2650}{43600} = 0\ 0608$$

$$R_m = \frac{49200}{48300} = 1\ 019$$

$$RR = 1\ 114$$

7 4 5 To calibrate the analytical system by isotope dilution, analyze a 1 0 $\mu$L aliquot of each of the calibration standards (section 6 13) using the procedure in section 11 Compute the RR at each concentration

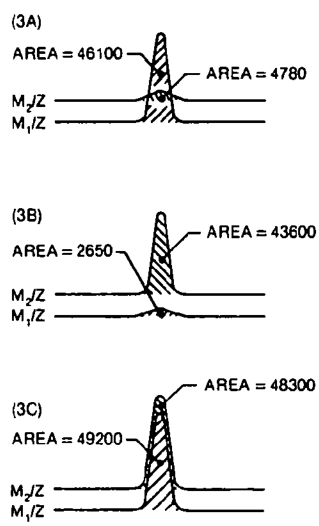


**FIGURE 3 Extracted Ion Current Profiles for (3A) Unlabeled Compound, (3B) Labeled Compound, and (3C) Equal Mixture of Unlabeled and Labeled Compounds.**

7 4 6 Linearity—if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, an averaged relative response/concentration ratio may be used for that compound, otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range

7 5 Calibration by internal standard—used when criteria for isotope dilution (section 7 4) cannot be met The internal standard to be used for both acid and base/neutral analyses is 2,2'-difluorobiphenyl The internal standard method is also applied to determination of compounds having no labeled analog, and to measurement of labeled compounds for intra-laboratory statistics (sections 8 4 and 12 7 4)

7 5 1 Response factors—calibration requires the determination of response factors (RF) which are defined by the following equation

$$RF = \frac{(A_s \times C_{is})}{(A_{is} \times C_s)}, \text{ where}$$

$A_s$ is the area at the characteristic mass for the compound in the daily standard

$A_{is}$ is the area of the characteristic mass for the internal standard

$C_{is}$ is the concentration of the internal standard ($\mu$g/mL)

$C_s$ is the concentration of the compound in the daily standard ($\mu$g/mL)

7 5 1 1 The response factor is determined for at least five concentrations appropriate to the response of each compound (section 6 13), nominally, 10, 20, 50, 100, and 200 $\mu$g/mL The amount of internal standard added to each extract is the same (100 $\mu$g/mL) so that $C_{is}$ remains constant The RF is plotted vs concentration for each compound in the standard ($C_s$) to produce a calibration curve

7 5 1 2 Linearity—if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound, otherwise, the complete calibration curve for that compound shall be used over the 5 point range

7 6 Combined calibration—by using calibration solutions (section 6 13) containing the pollutants, labeled compounds, and the internal standard, a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods These curves are verified each shift (section 12 5) by analyzing the 100 $\mu$g/mL calibration standard (section 6 13) Recalibration is required only if calibration verification (section 12 5) criteria cannot be met

## 8 QUALITY ASSURANCE/QUALITY CONTROL

8 1 Each laboratory that uses this method is required to operate a formal quality assurance program The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method

8 1 1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method This ability is established as described in section 8 2

8 1 2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met Each time a modification is made to the method, the analyst is required to repeat the procedure in section 8 2 to demonstrate method performance

8 1 3 Analyses of blanks are required to demonstrate freedom from contamination The procedures and criteria for analysis of a blank are described in sections 8 5

8 1 4 The laboratory shall spike all samples with labeled compounds to monitor method performance This test is described in section 8 3 When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (section 15)

8 1 5 The laboratory shall, on an on-going basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (section 6 14) that the analysis system is in control These procedures are described in sections 12 1, 12 5, and 12 7

8 1 6 The laboratory shall maintain records to define the quality of data that is generated Development of accuracy statements is described in section 8 4

8 2 Initial precision and accuracy—to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations

8 2 1 Extract, concentrate, and analyze two sets of four one-liter aliquots (8 aliquots total) of the precision and recovery standard (section 6 14) according to the procedure in section 10

8 2 2 Using results of the first set of four analyses, compute the average recovery ($\bar{X}$) in $\mu$g/mL and the standard deviation of the recovery (s) in $\mu$g/mL for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog

8 2 3 For each compound, compare s and $\bar{X}$ with the corresponding limits for initial precision and accuracy in table 8 If s and $\bar{X}$ for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin If, however, any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, system performance is unacceptable for that compound NOTE The large number of compounds in table 8 present a substantial probability that one or more will fail one of the acceptance criteria when all compounds are analyzed To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows

8 2 4 Using the results of the second set of four analyses, compute s and $\bar{X}$ for only those compounds which failed the test of the first set of four analyses (section

**TABLE 8**

**Acceptance Criteria for Performance Tests**

| EGD NO (1) | COMPOUND | ACCEPTANCE CRITERIA: INITIAL PRECISION AND ACCURACY SECTION 8 2 3 (µg/L) s | $\bar{X}$ | LABELED COMPOUND RECOVERY SECTION 8 3 AND 14 2 P (%) | CALIBRATION VERIFICATION SECTION 12 5 (µg/mL) | ON-GOING ACCURACY SECTION 12 7 R (µg/L) |
|---|---|---|---|---|---|---|
| 301 | acenaphthene | 21 | 79 - 134 | | 80 - 125 | 72 - 144 |
| 20 | acenaphthene-$d_{10}$ | 38 | 38 - 147 | 20 - 270 | 71 - 141 | 30 - 180 |
| 377 | acenaphthylene | 38 | 69 - 186 | | 60 - 166 | 61 - 207 |
| 277 | acenaphthylene-$d_8$ | 31 | 39 - 146 | 23 - 239 | 66 - 152 | 33 - 168 |
| 378 | anthracene | 41 | 58 - 174 | | 60 - 168 | 50 - 199 |
| 278 | anthracene-$d_{10}$ | 49 | 31 - 194 | 14 - 419 | 58 - 171 | 23 - 242 |
| 305 | benzidine | 119 | 16 - 518 | | 34 - 296 | 11 - 672 |
| 205 | benzidine-$d_8$ | 269 | ns - ns | ns - ns | ns - ns | ns - ns |
| 372 | benzo(a)anthracene | 20 | 65 - 168 | | 70 - 142 | 62 - 176 |
| 272 | benzo(a)anthracene-$d_{12}$ | 41 | 25 - 298 | 12 - 605 | 28 - 357 | 22 - 329 |
| 374 | benzo(b)fluoranthene | 183 | 32 - 545 | | 61 - 164 | 20 - ns |
| 274 | benzo(b)fluoranthene-$d_{12}$ | 168 | 11 - 577 | ns - ns | 14 - ns | ns - ns |
| 375 | benzo(k)fluoranthene | 26 | 59 - 143 | | 13 - ns | 53 - 155 |
| 275 | benzo(k)fluoranthene-$d_{12}$ | 114 | 15 - 514 | ns - ns | 13 - ns | ns - 685 |
| 373 | benzo(a)pyrene | 26 | 62 - 195 | | 78 - 129 | 59 - 206 |
| 273 | benzo(a)pyrene-$d_{12}$ | 24 | 35 - 181 | 21 - 290 | 12 - ns | 32 - 194 |
| 379 | benzo(ghi)perylene | 21 | 72 - 160 | | 69 - 145 | 58 - 168 |
| 279 | benzo(ghi)perylene-$d_{12}$ | 45 | 29 - 268 | 14 - 529 | 13 - ns | 25 - 303 |
| 712 | biphenyl (Appendix C) | 41 | 75 - 148 | | 58 - 171 | 62 - 176 |
| 612 | biphenyl-$d_{10}$ | 43 | 28 - 165 | ns - ns | 52 - 192 | 17 - 267 |
| 318 | bis(2-chloroethyl) ether | 34 | 55 - 196 | | 61 - 164 | 50 - 213 |
| 218 | bis(2-chloroethyl) ether-$d_8$ | 33 | 29 - 196 | 15 - 372 | 52 - 194 | 25 - 222 |
| 043 | bis(2-chloroethyoxy) methane* | 27 | 43 - 153 | | 44 - 228 | 39 - 166 |
| 342 | bis(2-chloroisopropyl) ether | 17 | 81 - 138 | | 67 - 148 | 77 - 145 |
| 242 | bis(2-chloroisopropyl) ether-$d_{12}$ | 27 | 35 - 149 | 20 - 260 | 44 - 229 | 30 - 169 |
| 366 | bis(2-ethylhexyl) phthalate | 31 | 69 - 220 | | 76 - 131 | 64 - 232 |
| 266 | bis(2-ethylhexyl) phthalate-$d_4$ | 29 | 32 - 205 | 18 - 364 | 43 - 232 | 28 - 224 |
| 041 | 4-bromophenyl phenyl ether* | 44 | 44 - 140 | | 52 - 193 | 35 - 172 |
| 067 | butyl benzyl phthalate* | 31 | 37 - 183 | | 22 - 450 | 35 - 195 |

TABLE 8 (Continued)
Acceptance Criteria for Performance Tests

| EGD NO (1) | COMPOUND | ACCEPTANCE CRITERIA: INITIAL PRECISION AND ACCURACY SECTION 8 2 3 (µg/L) s | $\bar{X}$ | LABELED COMPOUND RECOVERY SECTION 8 3 AND 14 2 P (%) | CALIBRATION VERIFICATION SECTION 12 5 (µg/mL) | ON-GOING ACCURACY SECTION 12 7 R (µg/L) |
|---|---|---|---|---|---|---|
| 717 | n-$C_{10}$ (Appendix C) | 51 | 24 - 195 | | 42 - 235 | 19 - 237 |
| 617 | n-$C_{10}$-$d_{22}$ | 70 | ns - 298 | ns - ns | 44 - 227 | ns - 404 |
| 706 | n-$C_{12}$ (Appendix C) | 74 | 35 - 369 | | 60 - 166 | 29 - 424 |
| 606 | n-$C_{12}$-$d_{26}$ | 53 | ns - 331 | ns - ns | 41 - 242 | ns - 408 |
| 518 | n-$C_{14}$ (Appendix C)* | 109 | ns - 985 | | 37 - 268 | ns - ns |
| 719 | n-$C_{16}$ (Appendix C)* | 33 | 80 - 162 | | 72 - 138 | 71 - 181 |
| 619 | n-$C_{16}$-$d_{34}$ | 46 | 37 - 162 | 18 - 308 | 54 - 186 | 28 - 202 |
| 520 | n-$C_{18}$ (Appendix C)* | 39 | 42 - 131 | | 40 - 249 | 35 - 167 |
| 721 | n-$C_{20}$ (Appendix C) | 59 | 53 - 263 | | 54 - 184 | 46 - 301 |
| 621 | n-$C_{20}$-$d_{42}$ | 34 | 34 - 172 | 19 - 306 | 62 - 162 | 29 - 198 |
| 522 | n-$C_{22}$ (Appendix C)* | 31 | 41 - 184 | | 40 - 249 | 39 - 195 |
| 723 | n-$C_{24}$ (Appendix C) | 11 | 80 - 139 | | 65 - 154 | 78 - 142 |
| 623 | n-$C_{24}$-$d_{50}$ | 28 | 27 - 211 | 15 - 376 | 50 - 199 | 25 - 229 |
| 524 | n-$C_{26}$ (Appendix C)* | 35 | 35 - 193 | | 26 - 392 | 31 - 212 |
| 525 | n-$C_{28}$ (Appendix C)* | 35 | 35 - 193 | | 26 - 392 | 31 - 212 |
| 726 | n-$C_{30}$ (Appendix C) | 32 | 61 - 200 | | 66 - 152 | 56 - 215 |
| 626 | n-$C_{30}$-$d_{62}$ | 41 | 27 - 242 | 13 - 479 | 24 - 423 | 23 - 274 |
| 528 | carbazole (4c)* | 38 | 36 - 165 | | 44 - 227 | 31 - 188 |
| 320 | 2-chloronaphthalene | 100 | 46 - 357 | | 58 - 171 | 35 - 442 |
| 220 | 2-chloronaphthalene-$d_7$ | 41 | 30 - 168 | 15 - 324 | 72 - 139 | 24 - 204 |
| 322 | 4-chloro-3-methylphenol | 37 | 76 - 131 | | 85 - 115 | 62 - 159 |
| 222 | 4-chloro-3-methylphenol-$d_2$ | 111 | 30 - 174 | ns - 613 | 68 - 147 | 14 - 314 |
| 324 | 2-chlorophenol | 13 | 79 - 135 | | 78 - 129 | 76 - 138 |
| 224 | 2-chlorophenol-$d_4$ | 24 | 36 - 162 | 23 - 255 | 55 - 180 | 33 - 176 |
| 340 | 4-chlorophenyl phenyl ether | 42 | 75 - 166 | | 71 - 142 | 63 - 194 |
| 240 | 4-chlorophenyl phenyl ether-$d_5$ | 52 | 40 - 161 | 19 - 325 | 57 - 175 | 29 - 212 |
| 376 | chrysene | 51 | 59 - 186 | | 70 - 142 | 48 - 221 |
| 276 | chrysene-$d_{12}$ | 69 | 33 - 219 | 13 - 512 | 24 - 411 | 23 - 290 |
| 713 | p-cymene (Appendix C) | 18 | 76 - 140 | | 79 - 127 | 72 - 147 |
| 613 | p-cymene-$d_{14}$ | 67 | ns - 359 | ns - ns | 66 - 152 | ns - 468 |
| 082 | dibenzo(a,h)anthracene* | 55 | 23 - 299 | | 13 - 761 | 19 - 340 |
| 705 | dibenzofuran (Appendix C) | 20 | 85 - 136 | | 73 - 136 | 79 - 146 |
| 605 | dibenzofuran-$d_8$ | 31 | 47 - 136 | 28 - 220 | 66 - 150 | 39 - 160 |
| 704 | dibenzothiophene (Synfuel) | 31 | 79 - 150 | | 72 - 140 | 70 - 168 |
| 604 | dibenzothiophene-$d_8$ | 31 | 48 - 130 | 29 - 215 | 69 - 145 | 40 - 156 |
| 368 | di-n-butyl phthalate | 15 | 76 - 165 | | 71 - 142 | 74 - 169 |
| 268 | di-n-butyl phthalate-$d_4$ | 23 | 23 - 195 | 13 - 346 | 52 - 192 | 22 - 209 |
| 325 | 1,2-dichlorobenzene | 17 | 73 - 146 | | 74 - 135 | 70 - 152 |
| 225 | 1,2-dichlorobenzene-$d_4$ | 35 | 14 - 212 | ns - 494 | 61 - 164 | 11 - 247 |
| 326 | 1,3-dichlorobenzene | 43 | 63 - 201 | | 65 - 154 | 55 - 225 |
| 226 | 1,3-dichlorobenzene-$d_4$ | 48 | 13 - 203 | ns - 550 | 52 - 192 | ns - 260 |
| 327 | 1,4-dichlorobenzene | 42 | 61 - 194 | | 62 - 161 | 53 - 219 |
| 227 | 1,4-dichlorobenzene-$d_4$ | 48 | 15 - 193 | ns - 474 | 65 - 153 | 11 - 245 |
| 328 | 3,3'-dichlorobenzidine | 26 | 68 - 174 | | 77 - 130 | 64 - 185 |
| 228 | 3,3'-dichlorobenzidine-$d_6$ | 80 | ns - 562 | ns - ns | 18 - 558 | ns - ns |
| 331 | 2,4-dichlorophenol | 12 | 85 - 131 | | 67 - 149 | 83 - 135 |
| 231 | 2,4-dichlorophenol-$d_3$ | 28 | 38 - 164 | 24 - 260 | 64 - 157 | 34 - 182 |
| 370 | diethyl phthalate | 44 | 75 - 196 | | 74 - 135 | 65 - 222 |
| 270 | diethyl phthalate-$d_4$ | 78 | ns - 260 | ns - ns | 47 - 211 | ns - ns |
| 334 | 2,4-dimethylphenol | 13 | 62 - 153 | | 67 - 150 | 60 - 156 |
| 234 | 2,4-dimethylphenol-$d_3$ | 22 | 15 - 228 | ns - 449 | 58 - 172 | 14 - 242 |
| 371 | dimethyl phthalate | 36 | 74 - 188 | | 73 - 137 | 67 - 207 |
| 271 | dimethyl phthalate-$d_4$ | 108 | ns - 640 | ns - ns | 50 - 201 | ns - ns |
| 359 | 2,4-dinitrophenol | 18 | 72 - 134 | | 75 - 133 | 68 - 141 |
| 259 | 2,4-dinitrophenol-$d_3$ | 66 | 22 - 308 | ns - ns | 39 - 256 | 17 - 378 |
| 335 | 2,4-dinitrotoluene | 18 | 75 - 158 | | 79 - 127 | 72 - 164 |
| 235 | 2,4-dinitrotoluene-$d_3$ | 37 | 22 - 245 | 10 - 514 | 53 - 187 | 19 - 275 |
| 336 | 2,6-dinitrotoluene | 30 | 80 - 141 | | 55 - 183 | 70 - 159 |
| 236 | 2,6-dinitrotoluene-$d_3$ | 59 | 44 - 184 | 17 - 442 | 36 - 278 | 31 - 250 |
| 369 | di-n-octyl phthalate | 16 | 77 - 161 | | 71 - 140 | 74 - 166 |

## TABLE 8 (Continued)
## Acceptance Criteria for Performance Tests

| EGD NO (1) | COMPOUND | ACCEPTANCE CRITERIA: INITIAL PRECISION AND ACCURACY SECTION 8.2.3 (µg/L) s | X̄ | LABELED COMPOUND RECOVERY SECTION 8.3 AND 14.2 P (%) | CALIBRATION VERIFICATION SECTION 12.5 (µg/mL) | ON-GOING ACCURACY SECTION 12.7 R (µg/L) |
|---|---|---|---|---|---|---|
| 269 | di-n-octyl phthalate-$d_4$ | 46 | 12 - 383 | ns - ns | 21 - 467 | 10 - 433 |
| 707 | diphenylamine (Appendix C) | 45 | 58 - 205 | | 57 - 176 | 51 - 231 |
| 607 | diphenylamine-$d_{10}$ | 42 | 27 - 206 | 11 - 488 | 59 - 169 | 21 - 249 |
| 708 | diphenyl ether (Appendix C) | 19 | 82 - 136 | | 83 - 120 | 77 - 144 |
| 608 | diphenyl ether-$d_{10}$ | 37 | 36 - 155 | 19 - 281 | 77 - 129 | 29 - 186 |
| 337 | 1,2-diphenylhydrazine | 73 | 49 - 308 | | 75 - 134 | 40 - 360 |
| 237 | 1,2-diphenylhydrazine-$d_{10}$ | 35 | 31 - 173 | 17 - 316 | 58 - 174 | 26 - 200 |
| 339 | fluoranthene | 33 | 71 - 177 | | 67 - 149 | 64 - 194 |
| 239 | fluoranthene-$d_{10}$ | 35 | 36 - 161 | 20 - 278 | 47 - 215 | 30 - 187 |
| 380 | fluorene | 29 | 81 - 132 | | 74 - 135 | 70 - 151 |
| 280 | fluorene-$d_{10}$ | 43 | 51 - 131 | 27 - 238 | 61 - 164 | 38 - 172 |
| 309 | hexachlorobenzene | 16 | 90 - 124 | | 78 - 128 | 85 - 132 |
| 209 | hexachlorobenzene-$^{13}C_6$ | 81 | 36 - 228 | 13 - 595 | 38 - 265 | 23 - 321 |
| 352 | hexachlorobutadiene | 56 | 51 - 251 | | 74 - 135 | 43 - 287 |
| 252 | hexachlorobutadiene-$^{13}C_4$ | 63 | ns - 316 | ns - ns | 68 - 148 | ns - 413 |
| 312 | hexachloroethane | 227 | 21 - ns | | 71 - 141 | 13 - ns |
| 212 | hexachloroethane-$^{13}C_1$ | 77 | ns - 400 | ns - ns | 47 - 212 | ns - 563 |
| 353 | hexachlorocyclopentadiene | 15 | 69 - 144 | | 77 - 129 | 67 - 148 |
| 253 | hexachlorocyclopentadiene-$^{13}C_4$ | 60 | ns - ns | ns - ns | 47 - 211 | ns - ns |
| 083 | ideno(1,2,3-cd)pryene* | 55 | 23 - 299 | | 13 - 761 | 19 - 340 |
| 354 | isophorone | 25 | 76 - 156 | | 70 - 142 | 70 - 168 |
| 254 | isophorone-$d_8$ | 23 | 49 - 133 | 33 - 193 | 52 - 194 | 44 - 147 |
| 360 | 2-methyl-4,6-dinitrophenol | 19 | 77 - 133 | | 69 - 145 | 72 - 142 |
| 260 | 2-methyl-4,6-dinitrophenol-$d_2$ | 64 | 36 - 247 | 16 - 527 | 56 - 177 | 28 - 307 |
| 355 | naphthalene | 20 | 80 - 139 | | 73 - 137 | 75 - 149 |
| 255 | naphthalene-$d_8$ | 39 | 28 - 157 | 14 - 305 | 71 - 141 | 22 - 192 |
| 702 | beta-naphthylamine (Appendix C) | 49 | 10 - ns | | 39 - 256 | ns - ns |
| 602 | beta-naphthylamine-$d_7$ | 33 | ns - ns | ns - ns | 44 - 230 | ns - ns |
| 356 | nitrobenzene | 25 | 69 - 161 | | 85 - 115 | 65 - 169 |
| 256 | nitrobenzene-$d_5$ | 28 | 18 - 265 | ns - ns | 46 - 219 | 15 - 314 |
| 357 | 2-nitrophenol | 15 | 78 - 140 | | 77 - 129 | 75 - 145 |
| 257 | 2-nitrophenol-$d_4$ | 23 | 41 - 145 | 27 - 217 | 61 - 163 | 37 - 158 |
| 358 | 4-nitrophenol | 42 | 62 - 146 | | 55 - 183 | 51 - 175 |
| 258 | 4-nitrophenol-$d_4$ | 188 | 14 - 398 | ns - ns | 35 - 287 | ns - ns |
| 061 | N-nitrosodimethylamine* | 198 | 21 - 472 | | 40 - 249 | 12 - ns |
| 063 | N-nitrosodi-n-propylamine* | 198 | 21 - 472 | | 40 - 249 | 12 - ns |
| 362 | N-nitrosodiphenylamine | 45 | 65 - 142 | | 68 - 148 | 53 - 173 |
| 262 | N-nitrosodiphenylamine-$d_6$ | 37 | 54 - 126 | 26 - 256 | 59 - 170 | 40 - 166 |
| 364 | pentachlorophenol | 21 | 76 - 140 | | 77 - 130 | 71 - 150 |
| 264 | pentachlorophenol-$^{13}C_6$ | 49 | 37 - 212 | 18 - 412 | 42 - 237 | 29 - 254 |
| 381 | phenanthrene | 13 | 93 - 119 | | 75 - 133 | 87 - 126 |
| 281 | phenanthrene-$d_{10}$ | 40 | 45 - 130 | 24 - 241 | 67 - 149 | 34 - 168 |
| 365 | phenol | 36 | 77 - 127 | | 65 - 155 | 62 - 154 |
| 265 | phenol-$d_5$ | 161 | 21 - 210 | ns - ns | 48 - 208 | ns - ns |
| 703 | alpha-picoline (Synfuel) | 38 | 59 - 149 | | 60 - 165 | 50 - 174 |
| 603 | alpha-picoline-$d_7$ | 138 | 11 - 380 | ns - ns | 31 - 324 | ns - 608 |
| 384 | pyrene | 19 | 76 - 152 | | 76 - 132 | 72 - 159 |
| 284 | pyrene-$d_{10}$ | 29 | 32 - 176 | 18 - 303 | 48 - 210 | 28 - 196 |
| 710 | styrene (Appendix C) | 42 | 53 - 221 | | 65 - 153 | 48 - 244 |
| 610 | styrene-$d_5$ | 49 | ns - 281 | ns - ns | 44 - 228 | ns - 348 |
| 709 | alpha-terpineol (Appendix C) | 44 | 42 - 234 | | 54 - 186 | 38 - 258 |
| 609 | alpha-terpineol-$d_3$ | 48 | 22 - 292 | ns - 672 | 20 - 502 | 18 - 339 |
| 529 | 1,2,3-trichlorobenzene (4c)* | 69 | 15 - 229 | | 60 - 167 | 11 - 297 |
| 308 | 1,2,4-trichlorobenzene | 19 | 82 - 136 | | 78 - 128 | 77 - 144 |
| 208 | 1,2,4-trichlorobenzene-$d_3$ | 57 | 15 - 212 | ns - 592 | 61 - 163 | 10 - 282 |
| 530 | 2,3,6-trichlorophenol (4c)* | 30 | 58 - 137 | | 56 - 180 | 51 - 153 |
| 531 | 2,4,5-trichlorophenol (4c)* | 30 | 58 - 137 | | 56 - 180 | 51 - 153 |
| 321 | 2,4,6-trichlorophenol | 57 | 59 - 205 | | 81 - 123 | 48 - 244 |
| 221 | 2,4,6-trichlorophenol-$d_2$ | 47 | 43 - 183 | 21 - 363 | 69 - 144 | 34 - 226 |

(1) Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method, reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method, reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution

* measured by internal standard, specification derived from related compound

ns = no specification, limit is outside the range that can be measured reliably

8 2 3) If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin If, however, any of the same compounds fail again, the analysis system is not performing properly for the compounds In this event, correct the problem and repeat the entire test (section 8 2 1)

8 3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix

8 3 1 Analyze each sample according to the method beginning in section 10

8 3 2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (section 7 5)

8 3 3 Compare the labeled compound recovery for each compound with the corresponding limits in table 8 If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample Therefore, the sample matrix is complex and is to be diluted and reanalyzed per section 15 4

8 4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained After the analysis of five wastewater samples for which the labeled compounds pass the tests in section 8 3, compute the average percent recovery (P) and the standard deviation of the percent recovery ($s_p$) for the labeled compounds only Express the accuracy assessment as a percent recovery interval from $P - 2s_p$ to $P + 2s_p$ For example, if $P = 90\%$ and $s_p = 10\%$, the accuracy interval is expressed as 70-110% Update the accuracy assessment for each compound on a regular basis (e g after each 5-10 new accuracy measurements )

8 5 Blanks—reagent water blanks are analyzed to demonstrate freedom from contamination

8 5 1 Extract and concentrate a blank with each sample lot (samples started through the extraction process on the same 8 hr shift, to a maximum of 20 samples) Analyze the blank immediately after analysis of the precision and recovery standard (section 6 14) to demonstrate freedom from contamination

8 5 2 If any of the compounds of interest (tables 1 and 2) or any potentially interfering compound is found in a blank at greater than 10 µg/L (assuming a response factor of 1 relative to the internal standard for compounds not listed in tables 1 and 2), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level

8 6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state The standards used for calibration (section 7), calibration verification (section 12 5), and for initial (section 8 2) and ongoing (section 12 7) precision and recovery should be identical, so that the most precise results will be obtained The GC/MS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of semivolatiles by this method

8 7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used

## 9 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

9 1 Collect samples in glass containers following conventional sampling practices (reference 7) Composite samples are collected in refrigerated glass containers (section 5 1 3) in accordance with the requirements of the sampling program

9 2 Maintain samples at 0-4 °C from the time of collection until extraction If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water EPA Methods 330 4 and 330 5 may be used to measure residual chlorine (reference 8)

9 3 Begin sample extraction within seven days of collection, and analyze all extracts within 40 days of extraction

## 10 SAMPLE EXTRACTION AND CONCENTRATION (See figure 4)

10 1 Labeled compound spiking—measure 1 00 ± 0 01 liter of sample into a glass container For untreated effluents, and samples which are expected to be difficult to extract and/or concentrate, measure an additional 10 0 ± 0 1 mL and dilute to a final volume of 1 00 ± 0 1 liter with reagent water in a glass container

10 1 1 For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 1 00 ± 0 01 liter aliquots of reagent water in glass containers

10 1 2 Spike 0 5 mL of the labeled compound spiking solution (section 6 8) into all samples and one reagent water aliquot

10 1 3 Spike 1 0 mL of the precision and recovery standard (section 6 14) into the two remaining reagent water aliquots

10 1 4 Stir and equilibrate all solutions for 1-2 hr

10 2 Base/neutral extraction—place 100-150 mL methylene chloride in each continuous extractor and 200-300 mL in each distilling flask

10 2 1 Pour the sample(s), blank, and standard aliquots into the extractors Rinse the glass containers with 50-100 mL methylene chloride and add to the respective extractor

10 2 2 Adjust the pH of the waters in the extractors to 12-13 with 6N NaOH while monitoring with a pH meter Begin the extraction by heating the flask until the methylene chloride is boiling When properly adjusted, 1-2 drops of methylene chloride per second will fall from the condenser tip into the water After 1-2 hours of extraction, test the pH and readjust to 12-13 if required Extract for 18-24 hours

10 2 3 Remove the distilling flask, estimate and record the

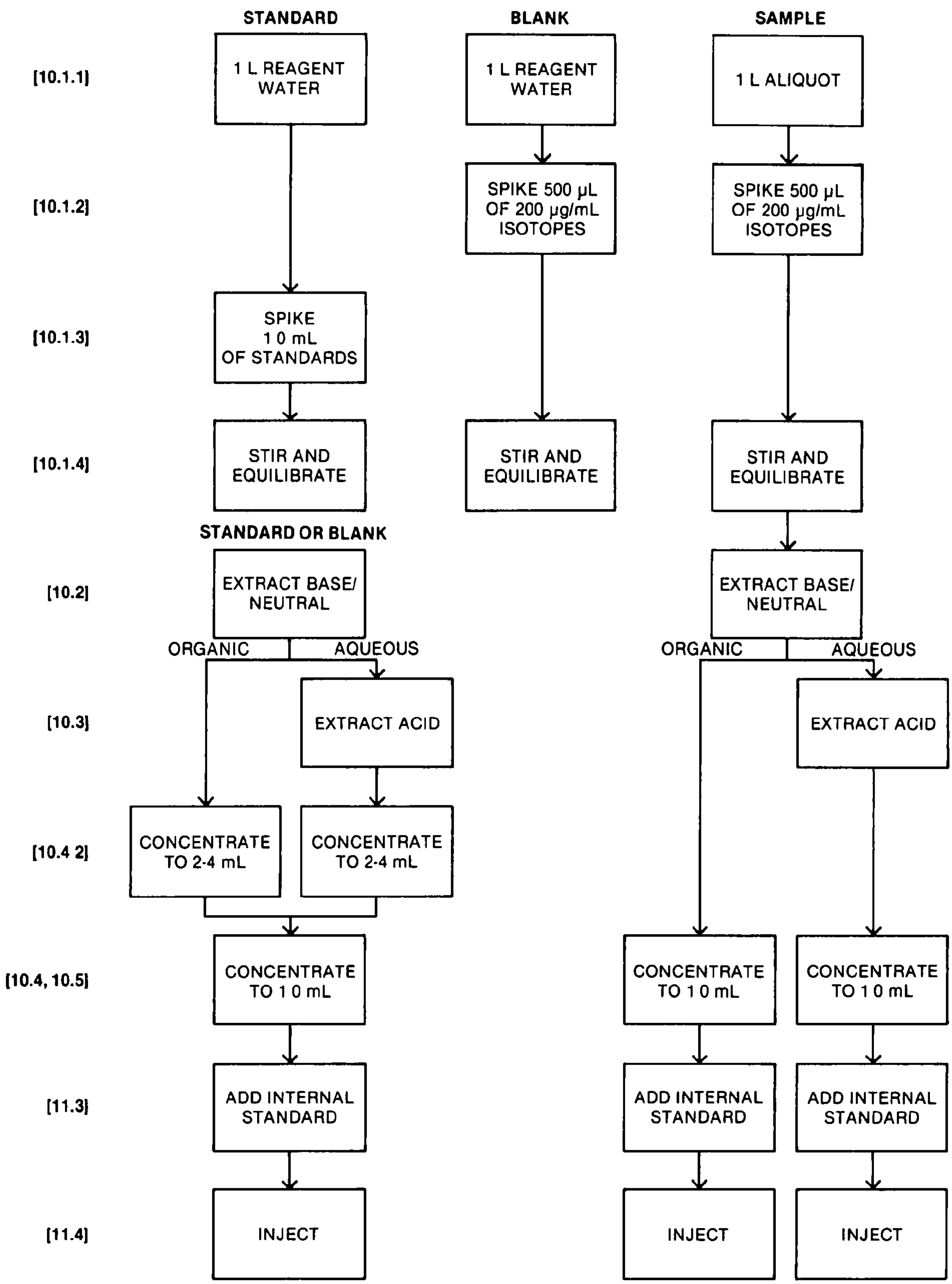


FIGURE 4 Flow Chart for Extraction/Concentration of Precision and Recovery Standard, Blank, and Sample by Method 1625. Numbers in Brackets [ ] Refer to Section Numbers in the Method

volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7 to 10 cm anhydrous sodium sulfate Rinse the distilling flask with 30-50 mL of methylene chloride and pour through the drying column Collect the solution in a 500 mL K-D evaporator flask equipped with a 10 mL concentrator tube Seal, label as the base/neutral fraction, and concentrate per sections 10 4 to 10 5

10 3 Acid extraction—adjust the pH of the waters in the extractors to 2 or less using 6N sulfuric acid Charge clean distilling flasks with 300-400 mL of methylene chloride Test and adjust the pH of the waters after the first 1-2 hr of extraction Extract for 18-24 hours

10 3 1 Repeat section 10 2 3, except label as the acid fraction

10 4 Concentration—concentrate the extracts in separate 500 mL K-D flasks equipped with 10 mL concentrator tubes

10 4 1 Add 1 to 2 clean boiling chips to the flask and attach a three-ball macro Snyder column Prewet the column by adding approximately one mL of methylene chloride through the top Place the K-D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam 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 When the liquid has reached an apparent volume of 1 mL, remove the K-D apparatus from the bath and allow the solvent 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 1-2 mL of methylene chloride A 5-mL syringe is recommended for this operation

10 4 2 For performance standards (section 8 2 and 12 7) and for blanks (section 8 5), combine the acid and base/neutral extracts for each at this point Do not combine the acid and base/neutral extracts for samples

10 5 Add a clean boiling chip and attach a two ball micro Snyder column to the concentrator tube Prewet the column by adding approx 0 5 mL methylene chloride through the top Place the apparatus in the hot water bath Adjust the vertical position and the water temperature as required to complete the concentration in 5-10 minutes At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood When the liquid reaches an apparent volume of approx 0 5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes Remove the micro Snyder column and rinse its lower joint into the concentrator tube with approx 0 2 mL of methylene chloride Adjust the final volume to 1 0 mL

10 6 Transfer the concentrated extract to a clean screw-cap vial Seal the vial with a Teflon-lined lid, and mark the level on the vial Label with the sample number and fraction, and store in the dark at − 20 to − 10 °C until ready for analysis

## 11 GC/MS ANALYSIS

11 1 Establish the operating conditions given in tables 3 or 4 for analysis of the base/neutral or acid extracts, respectively For analysis of combined extracts (section 10 4 2), use the operating conditions in table 3

11 2 Bring the concentrated extract (section 10 6) or standard (sections 6 13-6 14) to room temperature and verify that any precipitate has redissolved Verify the level on the extract (sections 6 6 and 10 6) and bring to the mark with solvent if required

11 3 Add the internal standard solution (section 6 10) to the extract (use 1 0 µL of solution per 0 1 mL of extract) immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction Mix thoroughly

11 4 Inject a volume of the standard solution or extract such that 100 ng of the internal standard will be injected, using on-column or splitless injection For 1 mL extracts, this volume will be 1 0 µL Start the GC column initial isothermal hold upon injection Start MS data collection after the solvent peak elutes Stop data collection after the benzo (ghi) perylene or pentachlorophenol peak elutes for the base/neutral or acid fraction, respectively Return the column to the initial temperature for analysis of the next sample

## 12 SYSTEM AND LABORATORY PERFORMANCE

12 1 At the beginning of each 8 hr shift during which analyses are performed, GC/MS system performance and calibration are verified for all pollutants and labeled compounds For these tests, analysis of the 100 µg/mL calibration standard (section 6 13) shall be used to verify all performance criteria Adjustment and/or recalibration (per section 7) shall be performed until all performance criteria are met Only after all performance criteria are met may samples, blanks, and precision and recovery standards be analyzed

12 2 DFTPP spectrum validity—inject 1 µL of the DFTPP solution (section 6 11) either separately or within a few seconds of injection of the standard (section 12 1) analyzed at the beginning of each shift The criteria in table 5 shall be met

12 3 Retention times—the absolute retention time of 2,2'-difluorobiphenyl shall be within the range of 1078 to 1248 seconds and the relative retention times of all pollutants and labeled compounds shall fall within the limits given in tables 3 and 4

12 4 GC resolution—the valley height between anthracene and phenanthrene at m/z 178 (or the analogs at m/z 188) shall not exceed 10 percent of the taller of the two peaks

12 5 Calibration verification—compute the concentration of each pollutant (tables 1 and 2) by isotope dilution (section 7 4) for those compounds which have labeled analogs Compute the concentration of each pollutant which has no labeled analog by the internal standard method (section 7 5) Compute the concentration of the labeled compounds by the internal standard method These concentrations are computed based on the calibration data determined in section 7

12 5 1 For each pollutant and labeled compound being tested, compare the concentration with the calibration verification limit in table 8 If all compounds meet the acceptance criteria, calibration has been verified and analysis of blanks, samples, and precision and recovery standards may proceed If, however, any compound fails, the measurement system is not performing properly for that compound In this event, prepare a fresh calibration standard or correct the problem causing the failure and repeat the test (section 12 1), or recalibrate (section 7)

12 6 Multiple peaks—each compound injected shall give a single, distinct GC peak

12 7 On-going precision and accuracy

12 7 1 Analyze the extract of one of the pair of precision and recovery standards (section 10 1 3) prior to analysis of samples from the same lot

12 7 2 Compute the concentration of each pollutant (tables 1 and 2) by isotope dilution (section 7 4) for those compounds which have labeled analogs Compute the concentration of each pollutant which has no labeled analog by the internal standard method (section 7 5) Compute the concentration of the labeled compounds by the internal standard method

12 7 3 For each pollutant and labeled compound, compare the concentration with the limits for on-going accuracy in table 8 If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed If, however, any individual concentration falls outside of the range given, system performance is unacceptable for that compound NOTE The large number of compounds in table 8 present a substantial probability that one or more will fail when all compounds are analyzed To determine if the extraction/concentration system is out of control or if the failure is caused by probability, proceed as follows

12 7 3 1 Analyze a second aliquot of the pair of precision and recovery standards (section 10 1 3)

12 7 3 2 Compute the concentration for only those pollutants or labeled compounds that failed the previous test (section 12 7 3) If these compounds now pass, the extraction/concentration processes are in control and analyses of blanks and samples may proceed If, however, any of the same compounds fail again, the extraction/concentration processes are not being performed properly for these compounds In this event, correct the problem, re-extract the sample lot (section 10) and repeat the on-going precision and recovery test (section 12 7)

12 7 4 Add results which pass the specifications in 12 7 3 to initial and previous on-going data Update QC charts to form a graphic representation of continued laboratory performance (figure 5) Develop a statement of laboratory accuracy for each pollutant and labeled compound by calculating the average percent recovery (R) and the standard deviation of percent recovery ($s_r$) Express the accuracy as a recovery interval from R − $2s_r$ to R + $2s_r$ For example, if R = 95% and $s_r$ = 5%, the accuracy is 85-105%

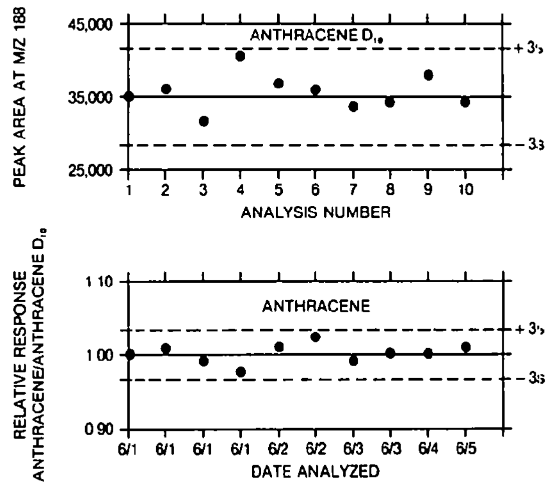


**FIGURE 5 Quality Control Charts Showing Area (top graph) and Relative Response of Anthracene to Anthracene-$d_{10}$ (lower graph) Plotted as a Function of Time or Analysis Number**

## 13 QUALITATIVE DETERMINATION

13 1 Qualitative determination is accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (section 12 1) and with data stored in the spectral libraries (section 7 2 4) Identification is confirmed when spectra and retention times agree per the criteria below

13 2 Labeled compounds and pollutants having no labeled analog

13 2 1 The signals for all characteristic masses stored in the spectral library (section 7 2 4) shall be present and shall maximize within the same two consecutive scans

13 2 2 Either (1) the backgound corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (0 5 to 2 times) for all masses stored in the library

13 2 3 The retention time relative to the nearest eluted internal standard shall be within ± 15 scans or ± 15 seconds, whichever is greater of this difference in the shift standard (section 12 1)

13 3 Pollutants having a labeled analog

13 3 1 The signals for all characteristic masses stored in the spectral library (section 7 2 4) shall be present and shall maximize within the same two consecutive scans

13 3 2 Either (1) the backgound corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library

13 3 3 The retention time difference between the pollutant and its labeled analog shall agree within ± 6 scans or ± 6 seconds (whichever is greater) of this difference in the shift standard (section 12 1)

13 4 Masses present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or backgound ions If the experimental mass spectrum is contaminated, an experienced spectrometrist (section 1 4) is to determine the presence or absence of the compound

## 14 QUANTITATIVE DETERMINATION

14 1 Istope dilution—by adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon extraction, concentration, and gas chromatography Relative response (RR) values for sample mixtures are used in conjunction with calibration curves described in section 7 4 to determine concentrations directly, so long as labeled compound spiking levels are constant For the phenol example given in figure 1 (section 7 4 1), RR would be equal to 1 114 For this RR value, the phenol calibration curve given in figure 1 indicates a concentration of 27 $\mu$g/mL in the sample extract ($C_{ex}$)

14 2 Internal standard—compute the concentration in the extract using the response factor determined from calibration data (section 7 5) and the following equation

$$C_{ex}\ (\mu g/mL) = \frac{(A_s \times C_{is})}{(A_{is} \times RF)}$$

where $C_{ex}$ is the concentration of the compound in the extract, and the other terms are as defined in section 7 5 1

14 3 The concentration of the pollutant in water is computed using the volumes of the original water sample (section 10 1) and the final extract volume (section 10 5), as follows

$$\text{Concentration in water}\ (\mu g/L) = \frac{(C_{ex} \times V_{ex})}{V_s}$$

where $V_{ex}$ is the extract volume in mL, and $V_s$ is the sample volume in liters

14 4 If the EICP area at the quantitation mass for any compound exceeds the calibration range of the system, the extract of the dilute aliquot (section 10 1) is analyzed by isotope dilution, otherwise, the extract is diluted by a factor of 10, 9 $\mu$L of internal standard solution (section 6 10) are added to a 1 0 mL aliquot, and this diluted extract is analyzed by the internal standard method (section 14 2) Quantify each compound at the highest concentration level within the calibration range

14 5 Report results for all pollutants and labeled compounds (tables 1 and 2) found in all standards, blanks, and samples, in $\mu$g/L, to three significant figures Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation mass is within the calibration range (section 14 4) and the labeled compound recovery is within the normal range for the method (section 15 4)

## 15 ANALYSIS OF COMPLEX SAMPLES

15 1 Untreated effluents and other samples frequently contain high levels ( >1000 $\mu$g/L) of the compounds of interest, interfering compounds, and/or polymeric materials Some samples will not concentrate to one mL (section 10 5), others will overload the GC column and/or mass spectrometer

15 2 Analyze the dilute aliquot (section 10 1) when the sample will not concentrate to 1 0 mL If a dilute aliquot was not extracted, and the sample holding time (section 9 3) has not been exceeded, dilute an aliquot of the sample with reagent water and re-extract (section 10 1), otherwise, dilute the extract (section 14 4) and analyze by the internal standard method (section 14 2)

15 3 Recovery of internal standard—the EICP area of the internal standard should be within a factor of two of the area in the shift standard (section 12 1) If the absolute areas of the labeled compounds are within a factor of two of the respective areas in the shift standard, and the internal standard area is less than one-half of its respective area, then internal standard loss in the extract has occurred In this case, use one of the labeled compounds (preferably a polynuclear aromatic hydrocarbon) to compute the concentration of a pollutant with no labeled analog

15 4 Recovery of labeled compounds—in most samples, labeled compound recoveries will be similar to those from reagent water (section 12 7) If the labeled compound recovery is outside the limits given in table 8, the dilute extract (section 10 1) is analyzed as in section 14 4 If the recoveries of all labeled compounds and the internal standard are low (per the criteria above), then a loss in instrument sensitivity is the most likely cause In this case, the 100 $\mu$g/mL calibration standard (section 12 1) shall be analyzed and calibration verified (section 12 5) If a loss in sensitivity has occurred, the instrument shall be repaired, the performance specifications in section 12 shall be met, and the extract reanalyzed If a loss in instrument sensitivity has not occurred, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes

## 16 METHOD PERFORMANCE

16 1 Interlaboratory performance for this method is detailed in references 5 and 9

16 2 A chromatogram of the 100 $\mu$g/mL acid/base/neutral calibration standard (section 6 13) is shown in figure 6

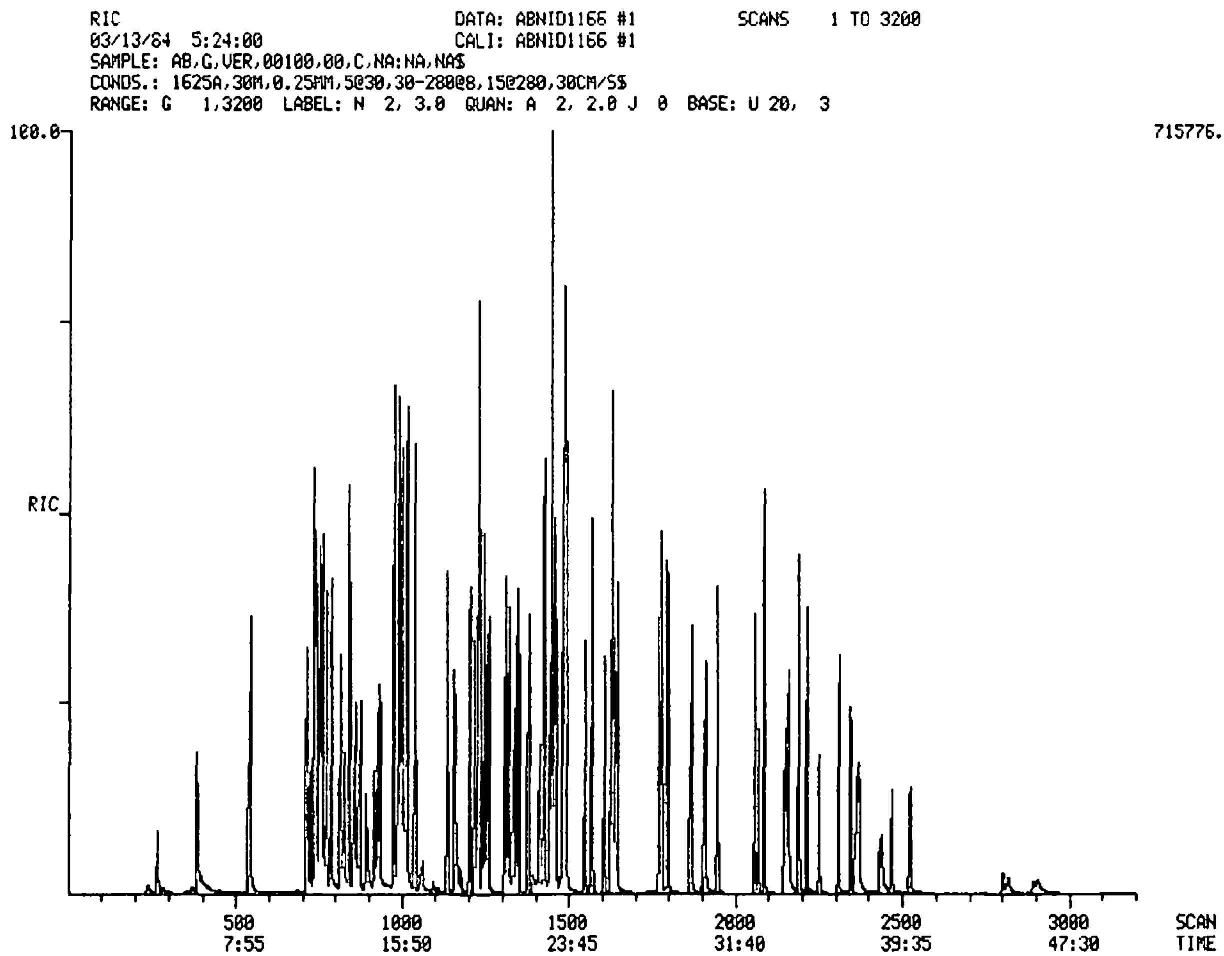


FIGURE 6 Chromatogram of Combined Acid/Base/Neutral Standard.

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