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I n d u s t r i a l U s e r I n s p e c t i o n A n d  
S a m p l i n g M a n u a l F o r P O T W ' s

Industrial User Inspection and Sampling  
Manual for POTWs

Prepared by:

The Office of Wastewater Enforcement and Compliance  
Water Enforcement Division  
U.S. Environmental Protection Agency  
Washington, D.C. 20460

April, 1994

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

MEMORANDUM

SUBJECT: Transmittal of the Final Industrial User Inspection and Sampling Manual

FROM: Michael B. Cook, Director  
Office of Wastewater Enforcement and

TO: Water Management Division Directors  
Regions I-X

I am pleased to provide the final Industrial User Inspection and Sampling Manual. The manual represents the culmination of almost two years of effort on the part of various offices within EPA Headquarters, and is the result of substantial comments from the EPA Regional Water Management Divisions, the Office of Research and Development in Cincinnati, AMSA members, and other interested parties. We appreciate the extensive effort in providing comments on the previous two drafts. Your insight significantly improved the document, and we are confident that the manual will be extremely useful to POTW inspection and sampling personnel.

The final document includes the second round of comments made by the Regions. The most significant change made to the document as a result of Regional comment is the deletion of the discussion on determining compliance with the 4-day average standard under the Electroplating regulation. The method for determining compliance with the Electroplating standard may be addressed through a policy paper at a later date. A second significant change is the deletion of the discussion on determining compliance from sample results below detection. The reason that we deleted this discussion is that the national work group addressing this issue has delayed its time frame for making a recommendation on how to address compliance in these situations. Therefore, any discussion of this matter will need to wait until the national work group has reached its conclusion.

We are expecting to conduct a mass-mailing of the document to all POTWs with approved pretreatment programs in late spring or early summer depending on the amount of time it takes to have the document printed. If you have any questions regarding the manual or its distribution, please feel free to call Lee Okster at (202) 260-8329.

cc: Cynthia Dougherty  
Regional Pretreatment Coordinators  
Fred Stiehl - OE  
Ken Kirk - AMSA

### Disclaimer

This manual has been written by the Office of Wastewater Enforcement and Compliance, U.S. Environmental Protection Agency, and has been peer reviewed both within the EPA and outside of the EPA. This guidance represents the EPA's recommended procedures to be used by POTW personnel when conducting an inspection or sampling visit at an industrial user. A failure on the part of any duly authorized POTW official, inspector, or agent to comply with the contents of the manual shall not be a defense in any enforcement action taken against an IU, nor shall a failure to comply with this guidance alone constitute grounds for rendering evidence obtained in the inspection inadmissible in a court of law. Any mention of trade names or commercial products is neither an endorsement nor a recommendation for use.

### Acknowledgements

This manual was written by the Office of Wastewater Enforcement and Compliance, U.S. Environmental Protection Agency, under the direction of Lee Okster. The Office of Wastewater Enforcement and Compliance would like to acknowledge the considerable efforts and cooperation of the following individuals, whose contribution helped to complete this document successfully: Mr. Paul Marshall (Region VII Retreatment Coordinator) for use of his checklist for inspecting industrial users; the EPA Regional Pretreatment Coordinators for insightful comments on the draft document; Mr. Sam Hadeed and members of the Association of Metropolitan Sewerage Agencies (AMSA) who provided comments on the draft document; Mr. William Potter of the EPA's Office of Research and Development in Cincinnati; Mr. Jack Stoecker of Brown and Caldwell; and Ms. Nadine Steinberg of the EPA's Office of Enforcement.

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## Definitions and Acronyms Used in the Pretreatment Program

- 1) Accuracy  
Accuracy refers to the degree of difference between observed values and known or actual value in the analysis of wastewater.
- 2) Act or "the Act"  
The Federal Water Pollution Control Act, also known as the Clean Water Act, as amended, 33 U.S.C. §125 et.seq.
- 3) Acute Effects  
When the effects of an exposure to a pollutant (over a short period of time) cause severe health effects to humans or other organisms, this condition is said to be acute (compare to chronic below).
- 4) Baseline Monitoring Report (BMR) [40 CFR 403.12(b)]  
All new source industrial users subject to categorical standards must submit a baseline monitoring report (BMR) to the Control Authority (POTW, State or EPA) at least 90 days prior to the commencement of discharge. The purpose of the BMR is to provide initial information to the Control Authority including identifying information, description of existing environmental permits, description of operations, flow measurements (estimated), and the concentration of pollutants in the waste stream (estimated). Existing sources were required to submit BMRs within 180 days after the effective date of any applicable categorical standard.

### Batch Process

A treatment or manufacturing process in which a tank or reactor is filled, the wastewater (or solution) is held or a chemical solution is prepared, and the tank is emptied, resulting in a discrete discharge to the sanitary sewer. The tank may then be refilled and the process repeated. Batch processes are also used to clean, stabilize, or condition chemical solutions for use in industrial manufacturing and treatment processes.

- 6) Biochemical Oxygen Demand (BOD)  
The quantity of oxygen utilized in the biochemical oxidation of organic matter under standard laboratory procedures for five (5) days at 20° centigrade, usually expressed as a concentration (e.g., mg/l). BOD measurements are used to indicate the organic "strength" of wastewater.
- 7) Biological Treatment  
A waste treatment process by which bacteria and other microorganisms break down complex organic or inorganic (e.g., ammonia) materials into simple, nontoxic, more stable compounds.
- 8) Blank (Bottle)  
Is an aliquot of analyte-free water which is taken through the appropriate steps of the analytic process as a means of determining if the sampling container is introducing contamination into the sample. For aqueous samples, reagent water is used as a blank matrix; however, a universal blank matrix does not exist for solid samples (e.g., sludge), and therefore, no matrix is used.
- 9) Blank (Equipment)  
Is an aliquot of analyte-free water which is taken to and opened in the field. The contents of the blank are poured appropriately over or through the sample collection device, collected in a sample container,

and returned to the laboratory as a sample to be analyzed. Equipment blanks are a check on the sampling device cleanliness.

10) Blank (Field)

Is an aliquot of analyte-free water or solvent brought to the field in sealed containers and transported back to the laboratory with the sample containers and analyzed along with the field samples.

11) Blank (Method)

Is an aliquot of analyte-free water prepared in the laboratory and analyzed by the analytical method used for field samples. Method blanks are used to test for the cleanliness of reagents, instruments, and the laboratory environment.

12) Blank (Sample Preservation)

Is an aliquot of analyte-free water (usually distilled water) to which a known quantity of preservative is added. This type of sample is a means of determining the level of contamination of acid and chemical preservatives after a period of use in the field.

13) Blowdown

The discharge of water with high concentrations of accumulated solids from boilers to prevent plugging of the boiler tubes and/or steam lines. In cooling towers, blowdown is discharged to reduce the concentration of dissolved salts in the recirculating cooling water. Clean "make-up" water is added to dilute the dissolved solids in the system. Blowdown also includes the discharge of condensate.

14) Categorical Industrial User (CIU)

A categorical industrial user is an industrial user (see IU definition below) which is subject to a categorical standard promulgated by the U.S. EPA.

15) Categorical Standards (40 CFR 405-471)

Any regulation containing pollutant discharge limits promulgated by the EPA in accordance with Sections 307(b) and (c) of the Act (33 U.S.C. §1317) which apply to a specific category of users and which appear in 40 CFR Chapter I, Subchapter N, Parts 405-471.

16) Centralized Waste Treatment Facility (CWT)

A public or private facility which treats hazardous and other wastes. These facilities are designed to handle the treatment of specific hazardous wastes from industry. The waste waters containing the hazardous substances are transported to the facility for proper storage, treatment and disposal.

17) Chain of Custody

A legal record (which may be a series of records) of each person who had possession of an environmental sample, from the person who collected the sample to the person who analyzed the sample in the laboratory and to the person who witnessed the disposal of the sample.

18) Chemical Oxygen Demand (COD)

Chemical oxygen demand is expressed as the amount of oxygen consumed from the oxidation of a chemical during a specific test (in mg/L). As such, COD is a measure of the oxygen-consuming capacity of the organic matter present in the wastewater. The results of the COD test are not necessarily related to the Biochemical Oxygen Demand (BOD) because the chemical oxidant responsible for utilizing the oxygen may react with substances which bacteria do not stabilize.

**19) Chemical Treatment Process**

A waste treatment process which involves the addition of chemicals to achieve a desired level of effluent quality.

**20) Chronic Effects**

When the effect of a single or repeated exposure(s) to a pollutant causes health effects over a long period of time in humans or other organisms this is said to be a chronic condition (compare to acute above).

**21) Code of Federal Regulations (CFR)**

A publication of the United States government which contains all of the finalized federal regulations. Federal environmental regulations are found in volume 40 of the CFR, and the General Pretreatment Regulations are found at 40 CFR Part 403.

**22) Combined Wastestream Formula (CWF) [40 CFR 403.6(e)]**

The combined wastestream formula is a means of deriving alternative categorical discharge limits in situations where process effluent is mixed with waste waters other than those generated by the regulated process prior to treatment.

**23) Composite Proportional Samples**

A composite sample is a collection of individual grab samples obtained at regular intervals, either based on time intervals or flow intervals (e.g., every two hours during a 24-hour time span or every 1000 gallons of process wastewater produced). Each individual grab sample is either combined with the others or analyzed individually and the results averaged. In time composite sampling the samples are collected after equal time intervals and combined in proportion to the rate of flow when the sample was collected. Flow composite sampling can be produced in one of two ways. The first method of obtaining a flow composite sample is to collect equal volume individual grab samples after a specific volume of flow passes the sampling point. The second manner of obtaining flow composite sample is to vary the volume of the aliquot collected in proportion to the amount of flow that passed over the time interval which the sample represents. Composite samples are designed to be representative of the effluent conditions by reflecting the average conditions during the entire sampling period (compare grab sample).

**24) Confined Space**

A space which, by design, has limited openings for entry and exit, unfavorable natural ventilation which could contain or produce dangerous air contaminants (or create an atmosphere of oxygen deprivation), and which is not intended for continuous employee occupation. A permit may be required under OSHA to enter a confined space.

**25) Conservative Pollutant**

A pollutant found in wastewater that is not metabolized while passing through the treatment processes in a conventional wastewater treatment plant. Therefore, a mass balance can be constructed to account for the distribution of the conservative pollutant. For example, a conservative pollutant may be removed by the treatment process and retained in the plant's sludge or it may leave the plant in the effluent. Although the pollutant may be chemically changed in the process, it can still be detected. Heavy metals such as cadmium and lead are conservative pollutants.

**26) Control Authority [403.12(a)]**

The Control Authority is the jurisdictional entity which oversees the implementation of the National Pretreatment Program at the local level. Usually, the Control Authority is the POTW with an approved

pretreatment program, but in some cases, the Control Authority may be the State (e.g., Vermont, Connecticut, Nebraska, Alabama, and Mississippi), or it may be the EJPA (i.e., where there is no local approved program and the state is not approved to administer the National Pretreatment Program in lieu of the EPA).

**27) *Conventional Pollutant***

A pollutant which has been designated as conventional under section 304(a)(4) of the Act. These pollutants include: BOD, TSS, pH, fecal coliform, and oil and grease.

**28) *Custody***

Custody refers to the process whereby the inspector gains and controls possession of a sample. A sample is in custody if: 1) it is in the actual possession, control, and presence of the inspector; or 2) it is in the inspector's view; or 3) it is not in the inspector's presence, but is in a place of storage where only the inspector has access; or 4) it is not in the inspector's physical presence, but is in a place of storage and only the inspector and identified others have access.

**29) *Daily Maximum***

Is the average value of all grab samples taken during any given calendar day. If only one grab sample has been taken, that grab sample becomes the daily maximum (as well as the instantaneous maximum see definition below). If more than one grab sample is taken in a given day, the daily maximum is the average of all the individual grab samples. A composite sample, by definition, becomes the daily maximum for the calendar day in which it is collected.

**30) *Duplicate Sample (Field)***

Is a precision check on sampling equipment and sampling technique. At selected stations on a random time frame duplicate samples are collected from two sets of field equipment installed at the site, or duplicate grab samples are collected from a single piece of equipment at the site.

**31) *Duplicate Sample (Laboratory)***

A sample which is received by the laboratory and divided (by the laboratory) into two or more portions. Each portion is separately and identically prepared and analyzed. The results from laboratory duplicate samples check the laboratory precision.

**32) *Effluent***

Wastewater or other liquid - raw, untreated, partially or completely treated - flowing from an IU to a reservoir, basin, treatment process, or treatment plant.

**33) *EPA***

The United States Environmental Protection Agency. The principal environmental regulatory agency established by the Congress to administer the nation's environmental laws.

**34) *Existing Source***

Any source of discharge, the construction or operation of which commenced prior to the publication by the EPA of proposed categorical pretreatment standards, which will be applicable to such source if the standard is thereafter promulgated in accordance with Section 307 of the Act.

**35) *Grab Sample***

A sample which is taken from a wastestream without regard to the flow in the wastestream and over a

period of time not to exceed 15 minutes.

**36) Hazardous Waste (40 CFR 261)**

For a waste to be considered a hazardous waste it must first be designated a solid waste. Virtually all forms of wastes are considered to be solid wastes (including solids, liquids, semi-solids, and contained gaseous materials) except those expressly excluded under the regulatory definition, e.g., industrial effluent which is mixed with sanitary wastes in the sewer. For a solid waste to be considered hazardous it must meet one of two criteria: 1) it has one of the following four characteristics – ignitibility, corrosivity, reactivity, or toxicity (according to the Toxicity Characteristic Leaching Procedure). or 2) it must be a listed hazardous waste in 40 CFR 261.31-261.33.

**37) Industrial User (IU) [40 CFR 403.3(h)]**

An industrial user is any non-domestic source which introduces pollutants into a publicly owned treatment works (POTW).

**38) Influent**

Wastewater or other liquid - raw (untreated), partially or completely treated - flowing into a reservoir, basin, treatment process, or treatment plant.

**39) Instantaneous Maximum Discharge Limit**

The maximum concentration of a pollutant allowed to be discharged at any time. determined from the analysis of a grab sample collected at the industrial user.

**40) Interference [40 CFR 403.3(i)]**

A discharge which, alone or in conjunction with a discharge or discharges from other sources, inhibits or disrupts the POTW, its treatment processes or operations or its sludge processes, use or disposal; and therefore, is a cause of a violation of the POTW's NPDES permit or of the prevention of sewage sludge use or disposal in compliance with the act or any more stringent State or local regulations.

**41) Local Limits [40 CFR 403.5(c)]**

Effluent discharge limits applicable to industrial users of the Control Authority's system developed by the Control Authority in accordance with 40 CFR 403.5(c).

**42) Monthly Average**

The monthly average is the arithmetic average value of all samples taken in a calendar month for an individual pollutant parameter. The monthly average may be the average of all grab samples taken in a given calendar month, or the average of all composite samples taken in a given calendar month.

**43) New Source [40 CFR 403.3(k)(1)]**

(l) Any building, structure, facility, or installation from which there is (or may be) a discharge of pollutants, the construction of which commenced after the publication of proposed pretreatment standards under Section 307(c) of the Act which will be applicable to such source if such standards are thereafter promulgated in accordance with that section, provided that:

(a) The building, structure, facility, or installation is constructed at a site at which no other discharge source is located; or

(b) The building, structure, facility, or installation totally replaces the process or production equipment that causes the discharge of pollutants at an existing source; or

(c) The production or wastewater generating processes of the building, structure, facility, or

installation are substantially independent of an existing source at the same site. In determining whether these are substantially independent, factors such as the extent to which the new facility is engaged in the same general type of activity as the existing source, should be considered.

(2) Construction on a site at which an existing source is located results in a modification rather than a new source if the construction does not create a new building, structure, facility, or installation meeting the criteria of Section (1)(b) or (c) above but otherwise alters, replaces, or adds to an existing process or production equipment.

(3) Construction of a new source as defined under this paragraph has commenced if the owner or operator has:

(a) Begun, or caused to begin, as part of a continuous on-site construction program;

(i) any placement, assembly, or installation of facilities or equipment; or

(ii) significant site preparation work including clearing, excavation, or removal of existing buildings, structures or facilities which is necessary for the placement, assembly, or installation of new source facilities or equipment; or

(iii) entered into a binding contractual obligation for the purchase of facilities or equipment which are intended to be used in its operation within a reasonable time. Options to purchase or contracts which can be terminated or modified without substantial loss, and contracts for feasibility, engineering, and design studies do not constitute a contractual obligation under this definition.

**44) 90-Day Compliance Report [40 CFR 403.12(d)]**

A report submitted by categorical industrial users within 90 days following the date for final compliance with the standards. This report must contain flow measurement (or regulated process streams and other streams), measurement of pollutants, and a certification as to whether the categorical standards are being met.

**45) Noncontact Cooling Water**

Water used for cooling which does not come into direct contact with any raw material, intermediate product, waste product, or finished product.

**46) NPDES Permit**

A National Pollutant Discharge Elimination System permit is the regulatory document issued by either the EPA or approved State agency. The permit is designed to control the discharge of pollutants from point sources into waters of the U.S.

**47) Pass Through [40 CFR 403.3(n)]**

A discharge which exits the POTW into waters of the United States in quantities or concentrations which, alone or in conjunction with a discharge or discharges from other sources, is a cause of a violation of any requirement of the City's NPDES permit, including an increase in the magnitude or duration of a violation.

**48) Periodic Compliance Report [40 CFR 403.12(e)]**

A report submitted at least twice annually by each significant industrial user regulated under the local pretreatment program which indicates the nature and concentration of pollutants in the effluent which are limited by applicable pretreatment standards. In addition, the periodic report must indicate a record of measured or estimated average maximum daily flows for the reporting period.

**49) pH**

pH is an expression of the concentration of hydrogen ions in solution. The measurement indicates an acid solution when the pH is <7 and an alkaline solution when the pH is >7. pH meters typically measure the pH in the range of 0 to 14. pH reflects the negative logarithm of the hydrogen ion concentration of the aqueous solution.

**50) *Physical Waste Treatment Process***

Physical wastewater treatment processes include racks, screens, comminutors, clarifiers (sedimentation and flotation), and filtration, which, through physical actions, remove pollutants from the wastewater.

**51) Pickle**

An acid or other chemical solution in which metal objects are dipped to remove oxide scale or other adhering substances.

**52) *POTW [40 CFR 403.3(o)]***

Publicly Owned Treatment Works. A sewage (or wastewater) treatment works which is owned by a state, municipality, city, town, special sewer district or other publicly owned or financed entity, as opposed to a privately owned (industrial) treatment facility. The definition includes not only the treatment works itself, but also the entire collection system leading to the treatment works.

**53) *Precision***

Precision refers to the reproducibility of laboratory analytical results.

**54) *Pretreatment***

The reduction in the amount of pollutants, the elimination of pollutants, or the alteration of the nature of pollutant properties in wastewater prior to, or in lieu of, introducing those pollutants into the POTW. This reduction or alteration can be obtained by physical, chemical, or biological processes; by process changes; or by other means, except by diluting the concentration of the pollutants unless allowed by an applicable pretreatment standard.

**55) *Pretreatment Facility***

Industrial wastewater treatment system consisting of one or more treatment devices designed to remove sufficient pollutants from waste streams to allow an industry to comply with effluent limits (i.e., categorical standards, local limits, and federal prohibitive standards).

**56) *Priority Pollutants [40 CFR 423 Appendix A]***

Is the list of pollutants designated by the U.S. EPA pursuant to section 307(a)(1) of the Act. There are 65 classes of pollutants and 126 individual pollutants currently identified.

**57) Process Wastewater [40 CFR X22.21]**

Is any water which, during manufacturing or processing, comes into direct contact with or results from the production or use of any raw material, intermediate product, finished product, byproduct, or waste product.

**58) RCRA**

The Federal Resource Conservation and Recovery Act (PL 94-580). RCRA was enacted to define a federal role in solid waste and resource management and recovery. RCRA's primary goals are: 1) to protect human health and the environment from hazardous and other solid wastes; and 2) to protect and preserve natural resources through programs of resource conservation and recovery. Its principal

regulatory focus in on the control of hazardous waste through a comprehensive system of identification, tracking, treatment, storage, and ultimate disposal.

**59) *Receiving Water***

A stream, lake, river, ocean, or other surface or groundwater into which treated or untreated wastewater is discharged.

**60) *Representative Sample***

A sample from a wastestream that is as nearly identical in composition to that in the larger volume of wastewater being discharged.

**61) *Sewer Use Ordinance (SUO)***

A sewer use ordinance is a legal instrument implemented by a local governmental entity which sets out all the requirements for the discharge of pollutants into a publicly owned treatment works.

**62) *Significant Industrial User (SIU) [40 CFR 403.3(t)]***

A significant industrial user is an industrial user (see IU definition above) which is either: 1) a categorical industrial user; 2) a user which discharges an average of 25,000 gallons per day or more of process wastewater to a POTW; 3) contributes a process wastestream which makes up 5 percent or more of the average dry weather hydraulic or organic capacity of the POTW; or 4) is designated significant industrial user by the POTW.

**63) *Significant Noncompliance (SNC) [40 CFR 403.8(f)(2)(vii)]***

An industrial user is in SNC if its violations meet one or more of the following criteria:

- (A) Chronic violations of wastewater discharge limits, defined as those in which sixty-six percent or more of all measurements taken during a six month period exceed (by any magnitude) the daily maximum limit or the average limit for the same pollutant parameter;
- (B) Technical Review Criteria (TRC) violations, defined as those violations in which thirty-three percent or more of all measurements for each pollutant parameter taken during a six month period equal or exceed the product of the daily maximum limit or the average limit multiplied by the applicable TRC (TRC = 1.4 for BOD, TSS, fats, oil, and grease, and 1.2 for all other pollutants except pH);
- (C) Any other violation of a pretreatment effluent limit (daily maximum or longer term average) that the Control Authority determines has caused, alone or in combination with other discharges, interference or pass through (including endangering the health of POTW personnel or the general public);
- (D) Any discharge of a pollutant that has caused imminent endangerment to human health, welfare or to the environment or has resulted in the POTW's exercise of its emergency authority to halt or prevent such discharge;
- (E) Failure to meet, within 90 days after the schedule date, a compliance schedule milestone contained in a local control mechanism or enforcement order for starting construction, completing construction, or attaining final compliance;
- (F) Failure to provide, within 30 days after the due date, required reports such as the baseline monitoring report (see definition above), 90-Day Compliance Report (see definition above), periodic report (see definition above), and reports on compliance with compliance schedules;
- (G) Failure to accurately report noncompliance; and
- (H) Any other violation or group of violations which the Control Authority determines will adversely affect the operation or implementation of the local pretreatment program..

**64) Sludge**

The settleable solids intentionally separated from liquid waste streams during treatment typically under quiescent conditions, and the unintentional accumulation of solids in tanks and reservoirs associated with production and manufacturing processes.

**65) *Slug Discharge [40 CFR 403.8(f)(2)(v)]***

Any discharge at a flow rate or concentration which could cause a violation of the prohibited discharge standards in the General Pretreatment Regulations.

**66) *Slug Discharge Control Plan [40 CFR 403.8(f)(2)(v)]***

A plan designed to prevent the uncontrolled discharge of raw pollutants (or materials, e.g., a dairy spill of milk may disrupt a small POTW and would have to be reported even though milk is not a "pollutant") into the POTW. Every Significant Industrial User is required to be evaluated. at least every two years, for the necessity of instituting such a control plan.

**67) *Spiked Sample (Field)***

A sample of a known amount of a particular pollutant constituent prepared in the field by adding a known amount of the analyte in question during sampling. This technique identifies potential sample matrix interference and/or problems with inadequate sample preservation.

**68) *Spiked Sample (Laboratory)***

A sample of a known amount of a particular pollutant constituent prepared in the laboratory by adding a known amount of the pollutant in question at a concentration where the accuracy of the test method is satisfactory. Spiked samples check on the accuracy of the analytical procedure.

**69) *Split Sample (Field)***

A sample which is collected and divided in the field into the necessary number of portions (e.g., 2, 3, etc.) for analysis. Equally representative samples must be obtained in the process. The split samples are then analyzed by separate laboratories (or the same laboratory) preferably using the same analytical techniques.

**70) *Technology-Based Standards***

Discharge limits for specific industrial categories established by the Federal EPA based on the use of the Best Available Technology economically achievable (BAT), the Best Practicable Control Technology available (BPT), or the Best Conventional Technology available (BCT). Such standards are based on the cost and/or availability of technology to treat the specific wastestream under consideration.

**71) *Toxic Pollutant (40 CFR 122 Appendix D)***

Those pollutants, or combination of pollutants, including disease-causing agents, which after discharge and upon exposure, ingestion, inhalation, or assimilation into any organism either directly from the environment or indirectly by ingestion through the food chain, will, on the basis of information available to the Administrator of the EPA, cause, death, disease, behavioral abnormalities, cancer, genetic mutations, physiological malfunctions (including malfunctions in reproduction) or physical deformations, in such organisms or their offspring. Such pollutants which have been identified as toxic are listed at 40 CFR 122 Appendix D.

**72) *Water Quality Standards***

Water quality standards are provisions of state or federal law which consist of a designated use or uses

for a given water body and associated water quality criteria which must be met in the stream to **achieve** these uses. Water quality standards are **effluent** standards imposed on point sources. These standards are designed to achieve the water quality criteria established for a given wata body. These standards are designed to improve and/or maintain the **quality** of the receiving water, regardless of the cost or availability of treatment technology.

## I. INTRODUCTION

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### Inspections and Sampling in the Pretreatment Program

The requirements imposed on Industrial Users (IUs) and Publicly Owned Treatment Works (POTWs) by the National Pretreatment Program have become increasingly complex. The scope and detail of the pretreatment regulations have expanded at the same time as the control on the discharge from POTWs has evolved from traditional pollutants (e.g., BOD, TSS, etc.) to water quality-based permit limits and biomonitoring requirements. In addition, the Federal Sludge Regulation, promulgated on February 19, 1993, imposes requirements on the POTW's sludge use or disposal practice based on the quality of the sludge the POTW produces. Due to this increased complexity and the strengthened controls on the POTW's effluent and sludge, it has become increasingly important for the POTW to be able to assess directly the compliance status of its IUs. The primary method for a POTW to accomplish this oversight is to use periodic inspections and sampling at the IU.

This manual is intended to acquaint POTW personnel with the well-established inspection and wastewater sampling procedures which have been used in the NPDES program for many years. The information presented will guide the POTW inspector by providing a framework for conducting inspections and wastewater sampling. The manual assumes that the POTW inspector has a basic knowledge of wastewater treatment technologies, as well as all applicable Federal, State and local pretreatment requirements. The information contained in this guidance will serve the experienced inspector as a reference, while new inspection personnel will find it useful as a logical framework for learning how to conduct inspections and sampling. This manual is also intended to assist the POTW's legal counsel and lab personnel as a reference for the legal and technical aspects of pretreatment inspections and sampling activities. This manual is not intended to provide detailed information on pollution prevention activities. For further information on pollution prevention, the POTW should consult the following EPA Manual: Guides to Pollution Prevention: Municipal Pretreatment Programs. October, 1993, EPA/625/R-93/006.

Prior to this manual, in July, 1986, the EPA issued the Pretreatment Compliance Monitoring and Enforcement (PCME) Guidance document, With the evolution of the Pretreatment Program since that time,

As used in the text and throughout this manual, the term 'inspector' includes all field personnel who collect information that may lead to or support an enforcement action. While the focus of the text is on the conduct of compliance inspection at facilities subject to regulation by the POTW, the majority of the material is also relevant to other types of compliance/enforcement investigations.

however, much of the information contained in the PCME Guidance has become outdated. Instead, POTWs should use the Guidance for Developing Control Authority Enforcement Response Plans (September, 1989), the Industrial User Permitting Guidance Manual (September, 1989) and this guidance as the principal guides for implementing their approved pretreatment programs. These three documents are intended to replace the use of the PCME Guidance because they provide the most current information for POTWs to establish appropriate pretreatment permitting, compliance monitoring and enforcement procedures.

This document creates a comprehensive and detailed framework for conducting inspections and sampling at regulated industrial users. Certain elements of this overall framework may not need to be implemented by each POTW in all cases. For example, where there are few industrial users in the system and each one is small, the POTW may never encounter issues relating to confidential business information or some of the safety precautions described in this manual. Nevertheless, each of these items should be considered when a new industrial user is identified in the POTW's system. For large POTWs with many industrial users which are complex, it will probably be necessary for the POTW to follow each of the procedures outlined in this manual.

#### Legal Authority and Regulatory Basis for Conducting Industrial User Inspections and Sampling

The General Pretreatment Regulations establish the overall framework for implementing an approved pretreatment program. The Regulations outline the minimum requirements which a POTW must perform to satisfy the obligation established in its NPDES permit. With regard to inspecting and sampling industrial users, Section 403.8(f)(1)(v) of the Regulations requires the POTW to have the legal authority to:

“carry out all inspection, surveillance and monitoring procedures necessary to determine, independent of information supplied by Industrial Users, compliance or noncompliance with applicable Pretreatment Standards and requirements. Representatives of the POTW shall be authorized to enter any premises of any Industrial User in which a discharge source or treatment system is located or in which records are required to be kept ... to assure compliance with Pretreatment Standards.”

The standard to which POTWs are held for purposes of evidence collection during an inspection or sampling event is further outlined in Section 403.8(f)(2)(vi):

“Sample taking and analysis and the collection of other information shall be performed with sufficient care to produce evidence which is admissible in enforcement proceedings or judicial actions.”

It is important that the inspector keep current on the regulations by reading the Federal Register, the Code of Federal Regulations, by subscribing to a service which summarizes the EPA regulations, or by consulting on a regular basis with the POTWs legal counsel.

The overall objectives of the General Pretreatment Regulations are to: 1) prevent the introduction of pollutants into POTWs which will interfere with the operation of the plant, including interference with the desired use or disposal of its municipal sludge; 2) prevent the introduction of pollutants into the POTW which will pass through the treatment works to receiving streams; 3) improve opportunities to reclaim and recycle municipal and industrial wastewaters and sludges; and 4) reduce the health and environmental risk of pollution caused by the discharge of toxic pollutants to POTWs.

Ultimately, the POTW must implement an overall compliance monitoring program (i.e., receiving and reviewing self-monitoring reports from IUs, and conducting inspections and sampling) that accomplishes the following objectives: 1) meets the requirements and intent of the General Pretreatment Regulations. 2) is effective and timely in determining compliance with categorical standards, local limits and prohibited discharge standards, 3) provides representative data required to meet POTW reporting requirements to the State or EPA, and 4) provides sampling data that would be admissible in court, if such an enforcement action were to be undertaken by the POTW, State or Federal government.

Inspection and sampling activities form the core of the POTW's compliance monitoring program and require POTW personnel to enter private property to gather information to assess or determine the compliance status of the facility. Therefore, these compliance monitoring activities must be performed in accordance with minimum constitutional protections (e.g., protecting against unreasonable searches and seizures), as well as other rights and "due process" considerations available to individuals under Federal, State or local law. Consequently, all POTW representatives, including any authorized agents of the POTW, who enter industrial facilities should be familiar with the section of their local ordinance that gives the Inspector (or the POTW's agent) the authority to enter an industrial user's facility to conduct an inspection or sample the wastewater. In addition, each inspector should be familiar with the POTW's standard procedures for entering industrial facilities, including how to obtain a warrant if entry is denied or withdrawn.

### *Purposes and Objectives for Inspecting and Sampling **Industrial Users***

The purposes and objectives of the POTW's compliance monitoring program (including inspections and sampling conducted by the POTW) are to:

- . Verify the completeness, accuracy and representativeness of self-monitoring data from the IU;
- . Determine compliance with IU permit conditions or Sewer Use Ordinance (SUO) provisions;
- . Support enforcement actions taken by the POTW against noncompliant IUs;
- . Generate data which can be used by the POTW in its annual report to the Approval Authority;
- . Determine if the IU has corrected problems identified in the previous inspection;

- Determine potential problems with other statutes or regulations (e.g., OSHA, RCRA);
- Evaluate Best Management Practices and pollution prevention measures;
- Identify which IUs influence the quality of the POTW's influent, effluent, and sludge quality;
- Evaluate the impacts of the POTW's influent on its treatment processes and receiving stream;
- Evaluate, the need for revised local limits;
- Inform the regulated community of pretreatment requirements;
- Maintain current data on each regulated industrial user;
- Assess the adequacy of the industrial user's self-monitoring program and the IU permit;
- Provide a basis for establishing the sampling requirements of the IU (above the Federal minimum);
- Evaluate the adequacy of the IU's operation and maintenance activities on its pretreatment system;
- Assess the potential for spills and/or slug discharges;
- Evaluate the effectiveness of slug discharge control measures;
- Gather information for IU permit development;
- Evaluate the adequacy of the IU's hazardous waste management and disposal;
- Evaluate compliance with existing enforcement actions; and
- Develop a good working relationship with the IU;

The inspector is the cornerstone of the POTW's compliance monitoring and enforcement of the pretreatment program. Without the inspector on the scene, processes that violate Federal, State, or local laws would continue to jeopardize the POTW, the environment, and public health.

For example, industrial waste can cause damage to the POTW's collection system by clogging sewers, causing corrosion, creating the potential for explosions, and contaminating the POTW's sludge. Toxic wastes from industry can upset the biological treatment processes which may take months to repair. To protect the environment, the Pretreatment Program has been designed to prevent pollutants generated at industrial sites from passing through (see the definition of Pass Through in the Definitions section of the manual) the POTW into the environment, either through the POTW's sludge or effluent, or by interfering (see the definition of Interference) with the operation of the POTW. Public health is protected by the Pretreatment Program through the regulation of industrial discharges so that treatment plant and sewer maintenance personnel are not exposed to toxic or flammable chemicals.

The inspector is usually the only person from the POTW who regularly appears at the industrial user's facility. The inspector's presence dramatically symbolizes the POTW's role as a responsible public agency observing the actions of the regulated industry. The knowledge that an inspection could occur unannounced encourages industrial plant managers to keep their operations in compliance.

*Outline of the Guidance*

The guidance is designed to lead the POTW inspector through the inspection and sampling process in a step-by-step fashion. The Introduction has laid out the overall framework for conducting site visits at industrial users. Chapter Two of the manual presents a chronological outline for conducting an inspection at an industrial user, beginning with the pre-inspection activities, such as the preparation and entry considerations; and then covers the on-site activities of the inspector, including: pre-inspection observations, the opening conference or initial meeting at the facility, the records review process, observations and illustrations, and the closing conference or exit interview conducted by the inspector. Finally, follow up activities by the POTW (e.g., inspection report writing and enforcement actions) are discussed.

Chapter Three of the manual presents a detailed framework for conducting sampling at the industrial user. This chapter covers: the objectives of sampling, pre-sampling considerations (e.g., a sampling plan), special sampling requirements (e.g., cyanide sampling at Electroplating facilities), analytical methods, use of automatic sampling devices, flow measurement, and quality assurance/quality control considerations.

## II. INSPECTING INDUSTRIAL USERS

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### INTRODUCTION

This chapter presents a framework to be used by POTW personnel when conducting on-site inspections at industrial facilities that discharge or have the potential to discharge process wastewater to the POTW. The principal intent of this chapter is to assist POTW personnel in planning, collecting, and documenting sufficient information to determine compliance or noncompliance, particularly by all SIUs, with all applicable Federal, State, and local pretreatment standards and requirements. New POTW inspection personnel are encouraged to read and understand the material presented in this chapter before beginning any inspection activities.

This chapter begins with a discussion of general inspection topics such as: developing and maintaining an industrial user survey; criteria to be used in setting the frequency of inspections and sampling activities; the types of inspections which can be used by the POTW; how to handle confidential business information; the general responsibilities of the POTW inspector; and the use of an inspector's field notebook. Once this groundwork has been laid, a detailed discussion of specific inspection activities follows. The topics covered in this discussion include: pre-inspection activities; on-site activities; and follow up activities. Also included

at the end of the chapter is an inspection checklist which can be used by the inspector as the basis for the inspection report.

Each of these activities of the inspection (i.e., general, pre-inspection, on-site, and follow up activities) are discussed in greater detail in the sections which follow. but first, the need for developing an industrial user survey is discussed.

## DEVELOPING AND MAINTAINING AN INDUSTRIAL USER SURVEY

All permitting, compliance monitoring and enforcement activities which the POTW undertakes are derived from an accurate identification of the regulated industrial users in the POTW's system. Therefore, it is imperative that the POTW maintain an up-to-date listing of each IU which discharges to the POTW. Once this list is established, the POTW should update this information periodically (sources of information and techniques for updating this information include: checking with the local Chamber of Commerce for new businesses, reconnaissance drive-throughs of the POTW district by POTW personnel, newspapers, applications for water service, yellow book advertising, building permits, etc.). For Significant Industrial Users (SIUs), the POTW is required to update this list annually as part of its Pretreatment Performance Report to the State or EPA. This list provides the basis for developing a plan for scheduling site visits at SIUs. The schedule for site visits should be contained in a neutral inspection plan (discussed later in this chapter) which should be developed by the POTW to guide its conduct of SIU site visits.

## FREQUENCY OF INSPECTION AND SAMPLING

The General Pretreatment Regulations require POTWs to inspect and sample each SIU at least once each year. This frequency was established as a minimum to represent the EPA's expectation for site visits to facilities with good compliance histories. POTWs should develop a neutral inspection plan (discussed below) to establish the criteria under which the POTW will conduct site visits at a frequency greater than the once per year minimum. These criteria should be applied when the POTW schedules routine compliance inspections for its IU universe. When considering how often to visit an industrial facility, the POTW should consider (at a minimum) the following criteria:

- The industrial user's potential to adversely affect the POTW's operations (e.g., the type and/or concentration of pollutants in the IU's discharge);
- . The volume and variability of the discharge;
- . Available resources and finances;
- . The type and reliability of control methods used to achieve compliance;
- . The quantity and nature of materials stored or in use and their relative risk of accidental spill;

- . POTW problems known or suspected to have been caused by the IU;
- . A history of complaints, if any, at the facility;
- . The facility's geographic location;
- . The compliance history of the user;
- . The period of time since the last inspection;
- . The imposition of new or additional pretreatment standards and requirements; and
- . Special considerations or circumstances such as seasonal production schedules or batch discharges at an industrial facility.

For example, a large facility with a poor compliance history may be scheduled for monthly site visits. On the other hand, a significant industrial user with a fairly consistent record of compliance, a cooperative attitude toward the pretreatment program, and a relatively constant manufacturing process may need to be inspected only once per year. When establishing an inspector's site visit schedule, adequate time must be allotted to allow inspectors to prepare for each visit, document their findings in a site visit report, and conduct other assigned duties.

The POTW should develop procedures to implement a neutral inspection plan for routine inspection and sampling visits. A neutral monitoring scheme provides some objective basis for scheduling inspections and sampling visits by establishing a system for setting priorities (whether a complex factor-based, alphabetical, or geographic system) to ensure that industrial users are not unfairly selected for inspection or sampling. The selection of which industrial users to inspect must be made without bias to ensure that the POTW can not be challenged for operating its inspection and sampling program in either an arbitrary or capricious manner. This plan should be included as part of the POTW's Enforcement Response Plan as well as included in the POTW's automated tracking system (if available).

#### *TYPES OF INSPECTIONS*

Inspection and sampling activities at industrial facilities may be: 1) scheduled, based on a neutral scheme; or 2) on demand, usually in response to a specific problem or emergency situation, such as a spill at the industry or an upset at the POTW. The POTW may use either of these inspection types when conducting a site visit at an industrial user, but to satisfy the minimum inspection frequency established by the General Pretreatment Regulations (40 CFR 403.8(f)(2)(v)), the POTW must conduct a routine compliance inspection at the IU (i.e., not a demand inspection, which may not have sufficient coverage to satisfy the regulatory requirement).

Scheduled inspections should be conducted according to the POTW's neutral inspection scheme. This does not mean that the POTW must notify the facility prior to each scheduled inspection. On the contrary, a routine compliance inspection is most effective when it is unannounced or conducted with very little advance warning (NOTE: when determining compliance with pretreatment standards, the IU should be in normal operation to ensure the representativeness of the samples taken). The neutral inspection scheme should set the criteria the POTW uses to choose which facilities to inspect, but the schedule for the actual inspection should remain confidential and may be separate from the neutral plan. Demand inspections are usually initiated in response to known or suspected violations, usually identified as a result of reviewing a self-monitoring report, a public complaint, a violation of the POTW's NPDES permit requirements, POTW operating difficulties, unusual influent conditions at the POTW, or emergency situations (e.g., sewer line blockages, fires, or explosions). When emergency situations arise in the treatment system (including the collection system), industrial inspections should be initiated immediately. Sampling is almost always a part of a demand inspection because the purpose of the inspection is to identify or verify the source of a discharge causing problems, and to gather information which might be used in a subsequent enforcement action. In some instances, the POTW may want to notify other appropriate local agencies (e.g., the fire department, State hazardous waste response team, the EPA, etc.) depending on the nature of the suspicion at the industry.

#### *CONFIDENTIAL BUSINESS INFORMATION*

The very nature of inspections involves gaining access to and collecting information that companies would not ordinarily make available to outsiders. When conducting compliance inspections, the inspector may have to deal with claims of confidentiality. These claims are authorized under Section 308 of the Federal Clean Water Act and are explained in the Code of Federal Regulations at 40 CFR Part 2. The inspector is responsible for following proper security measures when handling inspection data, both while on the road and in the office. Confidential business information includes trade secrets (including chemical identity, processes, or formulation) that could damage a company's competitive position if they became known to the public. Unauthorized disclosure of confidential information could result in criminal sanctions against the inspector.

Any business being inspected has the right to claim all or any part of the information gathered during the inspection as confidential. Information collected during an inspection is available to the public unless the IU takes measures to have the information held as confidential. This information must be held in confidence from the public, but this information must also be disclosed to the EPA upon request. The Control Authority may, as a matter of policy, notify the business through the IU permit or by providing the local SUO to the business of its right to claim confidentiality.

The affected business must assert its claim of confidentiality at the time the information is submitted to the POTW. If a claim of confidentiality is made after the fact, the POTW should make every effort to honor such a claim, but is in no position to guarantee that the information has not already been distributed to the public. The claim of confidentiality must be made in writing and signed by a responsible company official (e.g., a president, vice president, treasurer, general counsel, or chief executive officer). While the business is entitled to make a claim of confidentiality on all information which an inspector requests or has access to while on site, claims of confidentiality should be subject to review by the POTW's counsel. A business may not refuse (on the grounds that the information is considered confidential or a trade secret) to release information requested by the inspector. The claim of confidentiality only relates to the public availability of such data and is not to be used for denying access of a facility to POTW inspectors performing their duties under State or local law. Confidential business information must not be disclosed to competitors or to any other person who does not need to have access to the information to evaluate compliance with pretreatment obligations (e.g., POTW compliance personnel). A determination of confidentiality should be made when someone from the public requests the information which was claimed as confidential. At that time, the POTW's legal counsel must determine if the information is, under State law, confidential information. The POTW must have a process for safeguarding these materials until such a request is made, including having locked file cabinets, designating responsible officials, etc. Federal law requires that information described as "effluent data" (defined at 40 CFR Part 2.302(2)(i)) not be treated as confidential. Effluent data include any information regarding the nature of the discharge to the sewer system.

In some cases, entry to a facility may be denied based on a claim that there is confidential information at the facility. In such cases, the inspector should inform the industry of the relevant subsections of the State or local law regarding confidentiality so that they are clearly understood by all parties involved. The inspector should then explain the procedures used by the POTW to keep information confidential. In this instance, it would be helpful if the POTW had already notified the IU of its right to claim confidentiality and the IU's response (assuming it acknowledges this right and agrees that information must be provided to the inspector under the expectation that its claim will be honored by the POTW). If the facility representative still refuses entry, the inspector should not contest the issue but should treat the matter in the same manner as any denial of entry and follow established procedures for gaining entry (see discussion of Entry to the Industrial Facility later in this chapter).

To understand claims of confidentiality, an inspector should know the types of information considered confidential. The federal regulations specifically exclude certain types of information from confidential treatment. In particular, this "public" information includes all "effluent data." Effluent data include all

information necessary to determine the identity, amount, frequency, concentration, temperature, and other characteristics (to the extent related to water quality) of :

- . Any pollutant which has been discharged by the source (or any pollutant resulting from any discharge from the source) or any combination of these Pollutants; and
- . Any pollutant which, under an applicable standard or limitation, the IU was authorized to discharge (including a description of the manner or rate of operation of the facility).

Effluent data may also include a general description of the location and/or nature of the source of pollutants to the extent necessary to distinguish it from other sources of pollutants (e.g., a description of the device, installation, or operation of the source). In general, information which is collected to determine the compliance status of the industry is not considered confidential. It is the inspector's responsibility to handle all material claimed as confidential according to established procedures. For more information on confidentiality and the handling of confidential information, as well as on the right of entry to a facility for inspections and sampling, the inspector should consult with the POTW's legal counsel.

The inspector should not sign any pledge of secrecy or confidentiality agreements or any agreement which would limit the POTW's ability to disclose or use the information obtained while inspecting an IU. Such secrecy agreements are not a precondition of entry to the facility and should not be signed by the inspector. It is not appropriate for the inspector to determine whether an IU's claim of confidentiality is justified. Once such a claim is made, the information must be kept confidential until a determination is made by the POTW's legal counsel.

#### *RESPONSIBILITIES OF THE INSPECTOR*

The inspector's fundamental mission is to examine the environmental activities of a single regulated facility. The site visit is the basic element that determines the quality of information available for determining compliance and taking enforcement actions. The inspector must be knowledgeable about the requirements that apply to the user (i.e., the industrial user permit, the SUO, and Federal categorical standards) in order to determine the facility's compliance status. Local pretreatment program inspectors are responsible for the following areas of conduct:

#### **Legal:**

POTW inspectors must conduct all inspection activities within the legal framework established under State or local law. The inspector must be knowledgeable of the conditions established in the industrial user permit, the local SUO, applicable National Categorical Standards, local limits, the General Pretreatment Regulations, and any other applicable State or Local regulations, including any special requirements regarding entry to the industrial facility.

**Procedural:**

POTW inspectors must be familiar with general inspection procedures and evidence collection techniques to ensure accurate inspections and to avoid endangering potential legal proceedings on procedural grounds. These inspection procedures should be set forth in the local Enforcement Response Plan and should address inspections, sampling, flow monitoring, and documenting the results of these activities in a manner which enables the POTW to produce evidence which is admissible in a judicial action. The standard sequence of activities for conducting inspections are outlined in detail in Table 2-1. These procedural considerations are discussed further later in this chapter.

**Evidence Collection:**

POTW inspectors must be familiar with general evidence gathering techniques because the POTW's case in a civil or criminal prosecution depends in part on the evidence which the inspector has gathered. Inspectors must keep detailed records of each inspection. This information will serve when preparing the inspection report, determining the appropriate enforcement response, and giving testimony in an enforcement action. In particular, inspectors must know how to:

- substantiate facts with items of evidence, including: samples, photographs, document copies, statements from witnesses, and personal observations (but not opinions);
- abide by chain-of-custody procedures;
- collect and preserve data in a manner admissible in legal proceedings; and
- testify in court and administrative hearings.

Inspection and sampling procedures are discussed in detail in this chapter and in chapter three of this Manual.

**Safety:**

The POTW inspector must follow safety procedures, including: dressing appropriately and wearing safety clothing (e.g., steel toed shoes, hard hats, etc.), maintaining safety equipment in good working order, and using safety equipment in accordance with any manufacturers specifications or label procedures. In addition, the POTW inspector should follow the safety procedures established by the industrial user which is being inspected, unless these procedures prevent the inspector from conducting the inspection.

**Professional/Ethical:**

POTW inspectors must perform their duties with the highest degree of professionalism. In dealing with industry representatives, inspectors must be tactful, courteous, and diplomatic. The inspector is the representative of the POTW, and is often the initial or only contact between the IU and the POTW. A firm but responsive attitude should encourage cooperation and initiate a good working relationship with industry. Inspectors should avoid any negative comments regarding any product, manufacturer, or person while conducting their inspection. Inspectors should not accept gifts, favors, lunches, or any other benefits under any circumstances. This might be construed as influencing the performance of their duties. When evaluating the information obtained during the inspection, the facts of the inspection should be developed and reported completely, accurately, and objectively.

**Quality Assurance (QA) Responsibilities:**

The inspector must assume the primary responsibility for ensuring the quality of the compliance data obtained during the inspection. While other organizational elements play an important role in quality assurance (see the discussion in Chapter 3), it is the inspector who must assure that all effluent data generated by the POTW and introduced into the inspection file are complete, accurate, and representative of conditions at the IU. To help the inspector meet these responsibilities, the POTW should develop a QA Plan that identifies individual responsibilities and documents detailed procedures for ensuring the

**Table 2-1**  
***Procedural Responsibilities of the POH***

**Pre-Inspection Preparation:**

- Establish the purpose and scope of the inspection;
- Review all pertinent background information, including the IU permit and the permittee's compliance file;
- Contact appropriate staff personnel responsible for the permittee: e.g., pretreatment coordinator;
- Develop a plan for the inspection;
- Prepare any documents and equipment necessary for the inspection;
- Coordinate your schedule with the laboratory if samples are to be collected;
- Contact responsible party for transporting samples and for packing/shipping requirements.
- Review applicable categorical standards.

**Entry:**

- . Present official credentials and verbally identify oneself;
- . Manage denial or withdrawal of entry, if necessary.

**Opening Conference or Meeting: (if applicable)**

- . Discuss inspection objectives and scope;
- . Establish a working relationship with the industrial user

**Facility Inspection:**

- Conduct visual inspection of the entire industrial facility;
- Review industrial user records;
- Inspect monitoring equipment and operations;
- Review hazardous waste records;
- Collect samples;
- Review laboratory records for QA/QC, monitoring data (flow, pH, etc.);
- Review laboratory procedures to verify the use of approved methods;
- Document inspection activities.

**Closing Conference: (if applicable)**

- . Collect missing or additional information;
- . Clarify questions and answers with facility officials;
- . Review inspection findings and inform industry officials of follow-up procedures; and
- . Issue a deficiency notice, if appropriate.

**Inspection Report:**

- . Organize inspection findings into a useful, objective evidence package;
- . Include all deficiencies and required activities; and
- . Prepare the narrative report, checklists, and documentary information, as appropriate.

highest quality of sampling from the inspection. The objective of the QA Plan is to establish standards that will guarantee that data obtained during the inspection or sampling event meet the requirements of all users of that data (e.g., it must be able to be used by compliance personnel to determine the compliance status of the facility, and it must be able to be used by the POTW in court as admissible evidence in an enforcement proceeding). Many elements of QA are incorporated directly into the basic inspection procedures and may not be specifically identified as QA techniques by the inspector (e.g., chain-of-custody procedures). The inspector must be aware that following established inspection procedures is critical to the inspection program. These procedures should be developed to reflect the following QA elements:

- Valid data collection;
- Approved analytical methods;
- Standard data handling and reporting;
- Quality analytical techniques.

When conducting an in-depth inspection at an industrial user, POTW personnel are required to evaluate a broad spectrum of activities at the facility. In some cases, the level of expertise for this evaluation may exceed the qualifications of the inspector (e.g., when evaluating if appropriate analytical equipment is used or conformity with the analytical procedures in 40 CFR 136). In situations where such an evaluation is part of the inspection, the POTW should make available specially trained or skilled staff (e.g., analytical chemists) to either assist in the inspection or to train inspectors in their areas of expertise. The POTW inspector should have the necessary knowledge and skills for conducting effective IU inspections, but these skills and expertise may be supplemented by other POTW or contractor staff. The general skills and knowledge which POTW inspectors should have are outlined in Table 2-2.

### ***INSPECTOR'S FIELD NOTEBOOK***

providing strong documentary support of discrepancies uncovered in an inspection (e.g., conditions at the plant are found to be different than described in the permit application, the BMR, slug control plan, etc...) is one of the inspector's basic responsibilities. The core of all documentation taken by the inspector at the IU is contained in the inspector's field notebook, which is intended to provide accurate and inclusive documentation of all inspection activities. It is important for the information contained in the field notebook to relate exactly to the conditions observed by the inspector at the facility. The field notebook should *not* contain opinions or any observations not supportable from the facts of the inspection. Normally, field notes will be written in a field notebook and/or on a prepared report form developed by the POTW (or the inspector may use the enclosed checklist) to ensure that all pertinent information is collected. The field notebooks used by the inspector should be bound, to be sturdy enough to last through several inspections, and information should be recorded in permanent ink. It is important that the information obtained during the inspection be retained in the inspection notebook for a long time, because the information contained in the notebook might be used in an enforcement action years later. The POTW's legal counsel should be consulted for advice on preparing



Pretreatment inspectors should have the following knowledge and skills:

- Knowledge of Federal, State and local regulations and requirements.
- Knowledge of toxic constituents in industrial waste discharges.
- Knowledge of the Federal Categorical Pretreatment Standards.
- Knowledge of all local limits developed by the POTW.
- Knowledge of industrial processes and where wastestreams are generated.
- Knowledge of spill control procedures.
- Knowledge of wastewater treatment technology.
- Knowledge of wastewater sampling methods.
- Knowledge of wastewater analytical methods.
- Knowledge of flow measuring techniques.
- Knowledge of and ability to identify safety hazards associated with pretreatment control.
- Knowledge of and ability to practice professional ethics.
- Ability to inspect waste treatment facilities and verify conformance with specifications.
- Ability to evaluate and select monitoring locations.
- Ability to deal tactfully and effectively with industry representatives.
- Ability to maintain accurate records and write clear and concise reports.
- Ability to read and interpret mechanical construction drawings and pipeline schematics.
- Ability to keep confidential information and trade secrets.
- Ability to understand other viewpoints and work with industries and other regulatory agencies.
- Ability to prepare and maintain proper files and documentation on work performed.
- Ability to understand and carry out procedures on confidentiality developed by the POTW.

notes so that they may be read or introduced as evidence in an enforcement proceeding.

Since an inspector may be called to testify in an enforcement proceeding, it is imperative that each inspector keep detailed records of inspections, investigations, samples collected, and related inspection information. The types of information that should be entered into the field notebook include:

- **Observations:** All conditions, practices, and other observations that will be useful in preparing the inspection report or that will validate evidence should be recorded;
- **Documents and Photographs:** All documents taken or prepared by the inspector such as the completed checklists for the inspection report should be noted and related to specific inspection activities. (Photographs taken at a sampling site should be listed and described).
- **Unusual Conditions and Problems:** Unusual conditions and problems should be noted and described in detail, and
- **General Information:** Names and titles of facility personnel and the activities they perform should be listed along with statements they have made and other general information. Weather conditions should be recorded (e.g., raining or clear). This information can be used to determine if storm water is being discharged to the sanitary sewer. Information about the facility's record keeping procedures should be noted since it may be useful in later inspections. Information on who was interviewed and what those individuals said are important pieces of information for the inspector's notebook.

**PRE-INSPECTION ACTIVITIES**

Pre-inspection activities are crucial for conducting efficient and effective inspections because they provide a focus for the on-site inspection activities. By carefully planning the inspection activities, the inspector will not waste time on-site deciding what needs to be accomplished and how to obtain all of the necessary information. This background work should be completed at the POTW so that inspectors can use their time efficiently when they arrive at the facility. An inspection and sampling program begins even before the inspector goes out into the field. A good inspection begins with planning. Generally, a significant amount of time should be devoted to planning the inspection and on follow up activities to the inspection. Planning begins with the thought process by which the inspector identifies all activities relating to the inspection, from its objective (purpose) through its execution (actual conduct) and follow-up. Pre-inspection preparation is an essential element of conducting high quality inspections. By knowing “why” the facility is being inspected, “what” should be looked for, “how” it will be found, and “where” attention should be focused, the inspector will make the most efficient use of field time and ensure that the appropriate information for subsequent compliance or enforcement purposes is collected. By the time the inspector goes into the field, he or she should:

- . Have a clear idea of the objective for the inspection (e.g., investigation of a reported spill or complaint, routine compliance inspection, etc.). The objective will define the scope of the inspection (i.e., the range of activities to be conducted during the inspection). The objective will depend on what type of inspection is being conducted (i.e., scheduled, or demand);
- . Know all applicable program regulations (federal and local), compliance history, and physical layout of the site to help define the scope of activities the inspector will undertake at the facility;
- . Know the Standard Operating Procedures (SOPs) for the type of inspection activities to be conducted (again, these activities will be determined by the objective of the inspection). It is recommended that all Control Authorities adopt written SOPs. SOPs are a document or set of documents which explain, in step-by-step detail, how an inspection should be conducted (e.g., it defines who will be interviewed; what types of questions will be asked; how a specific type of sample will be collected; cleaning procedures for sample collection equipment and sample bottles; calibration methods for pH meters, D.O. meters, and conductivity meters, etc.; identification of the correct equipment, materials and techniques for conducting the inspection and for collecting, preserving, and documenting samples and other evidence; and any additional activity which the POTW conducts related to inspections or sampling). This manual will provide many of the details needed by POTWs to establish SOPs for their inspection activities; and
- . Know the safety plan for protecting all members of the inspection team from potential hazards or harmful exposures on site.

This section will describe the elements and procedures that go into pre-inspection planning, both those related to general field activity and the facility inspection itself. In this section, we will concentrate on the “why” and “what” of pre-inspection planning; detailed discussion of the “how to’s” of some of the key elements of planning (e.g., how to develop a quality assurance plan for samples) will be found in later chapters.

### ***Pre-Inspection Preparation***

Pre-inspection preparation can be broken down into the following activities: reviewing facility background information; developing an inspection plan; safety and sampling equipment preparation; and notifying the facility (if appropriate). Each of these will be discussed in turn.

#### **Review of Facility Background Information:**

To plan effectively and ensure the overall success of an inspection, it is essential that the inspector collect and analyze any available background information on the candidate facility. By reviewing background information, the inspector can minimize the inconvenience to the IU caused by requesting information which has already been submitted. Avoiding this situation increases the regulatory credibility of the POTW. The inspector must determine the amount of background information necessary for the inspection and in collecting this information should focus on the characteristics which are unique to the targeted facility, e.g., design and physical layout, historical practices and compliance status, legal requirements, etc.. The types of information which might be important for the inspector to review are listed in Table 2-3. A summary of this information may be kept in a separate file or filed with the final inspection report for each IU so that it is conveniently available for any subsequent inspection.

The inspector can find the majority of the information described in Table 2-3 in the permit application and permit of the industrial facility (for a review of permit requirements and application information, refer to the Industrial User Permitting Guidance Manual September, 1989), the POTW's Industrial Waste Survey, the BMR and 90 Day Compliance Reports, Periodic Compliance Reports, and information learned by the inspector from previous visits at the facility. The industrial user permit should clearly identify all of the responsibilities and obligations of the industrial user in a single document. The permit should provide information on all applicable effluent limitations (Federal categorical standards, prohibited discharge standards, and local limits), requirements and restrictions applicable to all discharges from the facility; slug control plans; monitoring, record keeping and reporting requirements; sampling location; type of samples to be taken; and required analytical methods (i.e., methods approved in 40 CFR Part 136). The permit application and BMR should outline the general facility information by describing the facility and providing site plans and layouts of the process areas and other areas of concern. The application should also include all contact persons as well as production levels and flow data from the facility. The previous inspection report for the facility will identify areas of concern from the last inspection which required action on the part of the IU. The inspector should review this report carefully and follow up on any progress in addressing any problems previously identified.

When the facility to be inspected is a categorical industry, the inspector should review any appropriate guidance from the U.S. EPA regarding that particular category to become familiar with the specific industrial

**Table 2-3**

***Information to Review Prior to the Inspection***

***General Facility Information:***

- Previous inspection report(s) (determine if there were any previously identified problems which needed to be addressed by the IU);
- Maps and schematics: showing the facility location, wastewater discharge pipes (i.e.. flow schematic), and geographic features (e.g.. topography);
- Names, titles, and telephone numbers of responsible facility officials;
- Any special entry requirements, e.g.. safety equipment (steel toed shoes, hard hats, etc.)
- Nature of the IU processing operations and wastewater characteristics;
- General layout of the facility;
- Production levels - past and present (especially for facilities regulated by production based categorical standards (e.g., aluminum forming);
- Changes in facility conditions since the last inspection or permit application (e.g.. expansion of the facility);
- Water use data;
- SIU slug control plan;
- Raw materials used in production processes;
- Location of storage sites for raw materials;
- Special permit conditions (e.g., peak flow restrictions, regulation of unusual substances);
- Progress toward meeting any applicable compliance schedule;
- Sources, volumes, and characteristics of the waste discharges;
- Aerial photographs (if available).

***Requirements, Regulations, and Limitations:***

- Copies of all applicable Federal, State and Local regulations and requirements, including any joint agreements or multi-jurisdictional agreements;
- Copy of the industrial user's permit and permit application; and
- Any applicable compliance schedule which the industry might be under.

***Facility Compliance and Enforcement History:***

- Any correspondence between the facility and Local, State or Federal agencies;
- Documentation on past violations of permit requirements or compliance schedules (available from the POTW's data tracking system);
- Self-monitoring data and reports;
- Post inspection reports;
- Past notices of violation (NOV), or other enforcement correspondence between the facility and the POTW; and
- Laboratory capability and analytical methods used by the industrial user's lab (if applicable).

***Wastewater Treatment Systems:***

- Description and design specifications for the wastewater treatment process employed at the industrial user;
- Process description, specifications, and schematic diagram;
- Available bypasses for existing pretreatment systems (if applicable);
- Type and amount of wastes discharged; and
- Spill control and contingency plans.

processes to be inspected. A list of all pretreatment-related guidance documents is found in Appendix XII. In addition, the inspector should become familiar with the EPA issued Development Documents associated with each industrial category to become more acquainted with the manufacturing processes, wastewater characteristics, as well as treatment technology at these categorical industries.

**Developing an Inspection Plan:**

The development of a sound inspection plan prior to going on-site is as important to the success of the POTW's inspection efforts as the on-site activities themselves or the preparation of a high-quality, well-documented inspection report. Inspection plans should be flexible enough to adapt to unanticipated situations encountered at the site, but the plan should also be designed as an organized approach to guide the conduct of the inspection. The basic purpose of the plan is to provide the inspector or inspection team (if appropriate) with a step-by-step guide to collecting relevant evidence about a facility's procedures and practices that are to be observed during the inspection. All inspection activities (i.e., its scope) are derived from the inspection's objective(s) (i.e., why the inspection is taking place). The objective(s), in turn, depend on the reason for conducting the inspection (i.e., to conduct a routine compliance evaluation, to follow up on information from a previous inspection, to investigate a complaint, or in response to an emergency situation). The inspection plan clarifies each of these areas (the inspection's objective, scope and activities) for each type of inspection. The basic components of the inspection plan should cover the following areas:

- . The purpose of (i.e., reason for) the inspection: a brief history of why the inspection is taking place and the inspection objectives (i.e., what goal is to be accomplished). This will depend on the type of inspection taking place.
- . The scope of the inspection (i.e., what range of activities need to take place to fulfill the objectives of the visit (NOTE This may change in the field, since some of the best evidence may be unanticipated by the inspector. The inspector must be flexible enough to adapt to new unanticipated situations in the field);
- . The inspection standard operating procedures (SOPs) and associated rationale for these activities (i.e., which field and analytical techniques will be used to collect what information; what record keeping systems will be reviewed; which IU personnel will be interviewed; which samples will be collected; and for each step, why).
- . The definition of team task assignments and time scheduling (if applicable);
- . Resource requirements (costs and time) based upon the planned activities and time allowances;
- . What kinds of evidence should be collected and documented in field log books.
- . A Quality Assurance Plan, where necessary; and
- . safety contingency plan, where necessary.

The investment of time required to produce a quality inspection plan is worth the effort because it constitutes a "walk-through" of the facility for the particular inspection type that should save time and

resources during the actual inspection. The inspector must be clear on what questions are appropriate to address during the inspection, and the plan provides a framework for working through these issues prior to visiting the site. POTWs should develop standard operating procedures and/or inspection checklists that are incorporated as part of the inspection plan. The checklist at the end of the chapter can be used for this purpose, and it can form the basis of the POTW's SOPs for a comprehensive compliance inspection. It is still important, however, to be clear as to which elements will be the focus of the inspection. Once this plan is developed for each facility and each type of inspection, it can be used for each subsequent inspection without revision, unless circumstances at the plant change significantly. If there are significant changes at the IU, a new inspection plan may need to be developed. The general components of such a plan are outlined in Table 2-4.

**Safety and Sampling Equipment Preparation:**

After the background information for an IU has been gathered and reviewed, and a plan for the inspection has been developed, POTW personnel should review and check the types of equipment which are necessary to meet the objectives of the site visit. An inspector must carry enough equipment to gather the necessary information during the inspection. This part of the pre-inspection process involves obtaining and preparing the equipment necessary for the inspection. The necessary types of equipment may vary with the nature of the IU and the types of activities to be performed by the POTW during the inspection. For example, if sampling is to be performed during the inspection, sampling equipment, and possibly additional safety equipment would need to be prepared. All equipment must be checked, calibrated and tested prior to each inspection. The inspector must also ensure that all materials necessary for the inspection are taken to the inspection site. Safety procedures and equipment for a facility will be based on past experience at the facility, or, for new facilities, the facility's response to the POTW's letter requesting such safety information (such a letter is recommended for all new facilities). Safety requirements must be met to ensure the inspector's safety and to help ensure that the inspector is not denied entry to the facility or parts of the manufacturing operations.

**Notification of the Facility:**

Most inspections will not involve notice to the affected facility. This is especially true when the POTW has established a working relationship with the IU, and its personnel are known at the facility. Also, notification should not be given to the facility when illegal discharges or improper records are suspected or the POTW wants an accurate picture of "normal" operations. The concern that physical conditions may be altered before the inspection or that records may be destroyed or altered justifies an unannounced visit to the facility. Likewise, a "demand" inspection (i.e., an inspection conducted as a result of a spill at the IU or upset at the POTW) can not be planned in advance. The POTW must be ready to conduct these types of inspections on very short notice and at any time of the day or night.

**Table 2-4**

***“Generic” Elements of an Inspection Plan***

- **Objectives (Purpose)**
  - What is the purpose of the inspection?
  - What is to be accomplished?
- **Tasks (Scope)**
  - What records, files, permits, regulations will be checked?
  - What coordination with laboratories, attorneys, other programs (e.g., solid waste or public health) is required?
  - What information must be collected?
- **Procedures**
  - What specific facility processes will be inspected?
  - What procedures will be used?
  - Will the inspection require special procedures?
  - Has a QA/QC plan been developed and understood?
  - What equipment will be required?
  - What are the responsibilities of each member of the team (if applicable)?
- **Resources**
  - What personnel will be required?
  - Has a safety plan been developed and understood?
- **Schedule**
  - What will be the time requirements and order of inspection activities?
  - What will be the milestones? What must get done vs. what is optional?

In some instances, the IU may be notified that it has been scheduled for an inspection by the POTW. The time frame for this notification is up to the POTW. Notification of the IU prior to visiting the facility is used primarily before inspecting an IU for the first time, so that plant officials are prepared to conduct a tour of the facility to familiarize the POTW with the IU's operations. If coordination with the IU is necessary for the inspection (e.g., to ensure that appropriate plant personnel are present), then the POTW may notify the facility of the exact date of the inspection and request that certain IU personnel be present. This notification may also inform the POTW of its rights to claim confidentiality of the information obtained during the inspection, but any confidentiality claim should be reviewed by the POTW's counsel at the time a request for the information is made by the public (see the Confidential Business Information discussion earlier in the chapter for a more detailed discussion of this matter).

*ENTRY TO THE INDUSTRIAL FACILITY*

The POTW's ability to conduct an inspection at a regulated facility stems from its authority to enter the IU's premises. Proper, lawful entry onto an inspection site is crucial. Failure to adhere to the requirements for exercising the POTW's entry authority could jeopardize any enforcement actions, and may subject the inspector to liability. Any evidence that may have been collected from an inspection where proper entry was not followed could be ruled inadmissible in an enforcement proceeding because it was obtained unlawfully.

This section discusses the legal basis for entry onto an IU's premises. It is always desirable to enter an IU's facility with the consent of the plant personnel, but there may be circumstances where such consent is not granted, or consent to enter particular areas of a facility may be denied. In situations where consent is denied, or where consent is withdrawn part way through an inspection, it will be necessary to follow certain procedures to ensure the legality of the inspection. This section covers both of these situations and explains what procedures should be followed by the inspector when consent is not granted to conduct or continue the inspection.

*Legal Basis for Entry:*

The authority to enter an IU's premises to conduct a compliance inspection comes from State or local law (usually the local sewer use ordinance). When a POTW is identified as required to have an approved pretreatment program, its legal authority is thoroughly reviewed by the EPA or State prior to any "approval" of that program. An approval from the EPA or State means that the POTW has the necessary authority under State and/or local law to implement all facets of its local program, including inspections. Usually, a local law authorizes the conduct of inspections and vests that authority with the Superintendent of the POTW or their designated representative. The designated representative is then allowed to enter, inspect, review records and sample at an industrial facility. If the Superintendent's designated representative (i.e., the POTW inspector) is allowed to inspect, he or she is usually required to present proper credentials prior to entering the facility. An inspector's credentials are his or her proof of authority to enter and inspect a facility, and should always be presented when entering the facility.

There may be times when the POTW may wish to inspect a non-discharging facility (e.g., if the POTW suspects that the non-discharging facility has commenced a discharge without a permit, or if the POTW desires to ensure that no discharge is occurring at the facility). Under these circumstances, the same authority which allows the POTW to enter the premises of discharging facilities should enable the POTW to gain access to the non-discharging facility. Of course, if the facility refuses entry, for whatever reason, the POTW has the same recourse as with any other facility, i.e., seek a warrant to enter the premises. In this case, the POTW should follow the Denial of Entry procedures outlined in this manual.

In some instances, the POTW employs an independent contractor to **perform** all or a **certain** aspect of the inspection or sampling at the industrial facility. POTW personnel should consult with their legal counsel to ensure that the POTW may, in fact, designate such contractor personnel as inspectors. This designation must be made in accordance with State or local law. The POTW should be aware that contractors may not perform functions which are inherently governmental (e.g., determining compliance, initiating enforcement action, etc.). All inspectors (whether POTW or contractor) must follow the **POTW's** written procedures for inspecting and sampling, and the POTW must be the ultimate authority when compliance determinations are made or policy decisions which affect the conduct of inspections or sampling.

The right of the government (Federal, State or Local) to enter an industrial facility and the protection against unlawful entry by the government has been the subject of numerous court decisions. These court decisions influence the manner in which a POTW inspector may enter a regulated facility. Several decisions by the Supreme Court pertaining to the right to enter and the use of warrants for entry have bearing on the **POTW** inspection process because these decisions define the limitations under which a POTW inspection may lawfully gain entry to a regulated industry. The principal court case dealing with these issues is *Marshall v. Barlow's*, 436 U.S. 307 (1978). Under this decision, the court concluded that where consent for the inspection was not voluntarily given by the facility, the inspector is required to obtain an administrative warrant to gain lawful entry. The court held that an inspector is not permitted to enter the non-public areas of the **worksite** without either the owner's consent or a warrant.

The court further established the conditions under which a civil or administrative warrant can be issued by a judge or magistrate. These conditions are: 1) reasonable cause to believe that a circumstance (e.g., a violation) addressed by a statute or ordinance (for a POTW this would be a State statute and/or local ordinance) had 2) that the facility to be entered was identified and selected by the **POTW** based on a pre-existing administrative plan or scheme for entries. The basis for the "plan" or "scheme" was required by the court to be "neutral". The message of the court was simply that the government (Federal, State and Local), through its field agents, cannot "pick on" regulated facilities with subtle harassing techniques or through the exercise of entry, search, inspection, investigation, or correctional rights or powers. The appropriate exercise of government authority is not to threaten an industry. This is why it is important that the **POTW** develop an inspection plan which is based on "neutral" conditions (e.g., geographical location) and to stick to this plan when conducting site visits or to conduct the inspection when there is a justified suspicion of a violation. The procedures which an inspector should follow when entry is denied are discussed in detail below.

There are two important exceptions to the limitations described above. In these two situations, there is a right to entry without a warrant, as discussed below:

**Emergency Situations:** such as potential imminent hazard situations, as well as situations where there is potential destruction of or where evidence of a suspected violation may disappear if time is permitted to elapse while a warrant is obtained. In an emergency, when there is insufficient time to **obtain** a warrant, a warrantless inspection is allowed. The POTW will have to exercise considerable judgment as to whether a warrant should be served when dealing with an emergency situation. However, even in emergency situations, the POTW would probably need the assistance of the police, sheriff, or fire department to gain entry. During the time it takes to get this assistance, a warrant could probably be obtained if there is close coordination with the POTW's legal counsel.

- **"Open Fields" and "In Plain View" situations:** Observations by an inspector of things which are in plain view (i.e., they can be seen by anyone in a lawful position or place to make such observations) do not require a warrant. For example, an inspector's observations from the public area of a facility or even from certain private property not normally closed to the public, are also proper and valid. Further, even when a warrant is obtained for entry, those areas outside of the warrant's scope are also "in plain view" so long as the warrant permits the inspector to be where they are when they make such viewing.

The inspector's authority is usually not limited to entering and examining the industry's treatment plant (effluent sources) alone. The inspector may inspect other areas of the permitted facility as well. The inspector should consult with the POTW's counsel to ensure a complete knowledge of the local law which authorizes their activity. Coordination with the POTW's counsel is also important when situations arise where entry is denied. Under these circumstances it may be necessary to contact the POTW's legal counsel to gain entry into the facility.

### *Arrival for the Inspection*

The inspector should enter the industrial facility in the following manner to avoid any "unreasonable search" or procedural problems:

- Arrive during normal business hours, unless it is an emergency situation or if other arrangement have been made with the industry;
- Enter the facility through the main gate, unless the facility has designated another point for entry;
- Locate the "person in charge" at the facility as soon as possible. Consent to enter the facility must be given by the owner or operator, or their designated representative. The inspector should learn who this individual is and develop a working relationship with that person. The inspector may want to have several industry contacts to grant entry in case the primary contact is not available. **As** long as the inspector is allowed to enter, the inspection is considered voluntary and consensual. A clear expression of consent is not necessary because an absence of an expressed denial is considered consent. If there is only a guard at the entrance, the inspector should present their credentials (if no credentials are issued by the POTW a business card should suffice) and suggest that the guard call his or her superior or the responsible industry representative. The credentials indicate that the holder is a lawful representative of the POTW and is authorized to perform pretreatment inspections. These credentials are important documents and should **never** leave the sight of the inspector.

If the facility provides a blank sign-in sheet, log, or visitor's register, it is acceptable for the inspector to sign it, as long as there is no restrictive language associated with it. The inspector must not sign any type of "waiver" or release from liability form that would limit in any way the ability of the POTW to use the information obtained during the inspection. The inspector must not agree to any such restrictive condition of entry. In addition, the inspector must not sign any safety or personal harm waiver absolving the facility of any injury which the inspector may incur while on-site. If the industry insists on such a waiver, the inspector should politely explain that they cannot sign and request a blank sign in sheet. In some instances, it may be possible to simply cross out the offensive language before signing, obtain a photocopy and make a note in your field notebook about it. If the inspector is refused entry because they do not sign the release, they should leave and immediately report all pertinent facts to their supervisor or, preferably, the POTW's legal staff. All events surrounding the refused entry should be fully documented, and problems should be discussed cordially and professionally. Officials at the regulated facility must not be subjected to any form of intimidation or threats for their failure to allow an inspector entry to the premises. The inspector's authority to inspect should not be abused, nor should the IU's right to refuse entry be attacked. Keep in mind that the inspector is at the facility to conduct an inspection, not to see a specific individual. If the normal contact is not in, the inspection should not be postponed.

The POTW inspector cannot be required to take a facility's safety training course prior to entry, but if the company has a relatively short safety briefing that will not interfere with the inspector's ability to complete the planned inspection, it may be worthwhile to attend.

Reluctance to give consent. The receptiveness of facility officials toward inspectors is likely to vary from facility to facility. Most inspections will proceed without difficulty. **Because** monitoring may be considered an adversarial proceeding to some industries, the inspector's legal authority, techniques, and competence may be challenged. If consent to enter is flatly denied, the inspector should follow the **denial** of entry procedures outlined below. In other cases, **officials** may be reluctant to give consent for entry because of misunderstandings of **responsibilities** (e.g., officials may feel that the inspection is part of an enforcement proceeding against the company), inconvenience to the firm's schedule, or other **reasons** that may be resolved through diplomacy and explanation on the part of the inspector.

One of the typical obstacles encountered by the inspector is a receptionist refusing entry because the inspector does not have an appointment. In this case, remind the receptionist that you are not there to see a specific individual but to inspect the facility. If entry is still refused, ask to speak to the **environmental** manager or owner of the facility. If that does not work, follow the denial of entry procedures outlined below.

Another common obstacle is the statement, "There is nobody here who can authorize the inspection." In this instance, ask to speak to a supervisor, or show the receptionist the section of the sewer use ordinance which authorizes the inspector's access to the facility. Do not threaten **legal** action, but clearly state your intent to inspect. Be professional, assertive and persistent, but if you still cannot gain entry, follow the denial of entry procedures outlined below.

Whenever there is **difficulty** in gaining consent to enter, inspectors should tactfully probe the reasons and work with **officials to** overcome any problems. In any instance where there is a misunderstanding or conflict due to the inspection, the inspector must avoid threats, inflammatory discussions, or language which would **deepen** the antagonism. The inspector should be aware of their personal safety during such confrontations and avoid actions which may enrage an individual who is irrational. If the situation is beyond the ability or authority of the inspector to manage, the inspector should leave and consult with the **POTW's** legal counsel.

**Uncredentialed persons accompanying the inspector.** The consent of the owner or agent in charge (i.e., industry representative) must be obtained for persons accompanying an inspector to enter a site if they do not have specific authorization (e.g., acting as an agent of the POTW). If consent is **not** given, such individuals may not enter the premises. If consent is given, these individuals may not view confidential business information unless officially authorized for access.

**Access to Federal facilities requiring security clearances.** Certain Federal facilities, including those with military, intelligence or nuclear-related activities may have special security or access requirements due to the facility's mission of national security. POTW inspectors have the right to gain access to these facilities to the same degree they have authority to enter any industrial facility which discharges to their system, but it is necessary for POTW personnel to comply with any special entry requirements. POTW inspectors must obtain the appropriate clearances for access to national security information, facilities or restricted areas at Federal facilities. Where compliance information has been classified, restricted or protected for national security, all information is to be maintained in accordance with the originating Agency's (e.g., DOD) requirements. This information should be treated as confidential business information and protected to the same degree as other CBI (e.g., access to this information should be under lock and key with only **authorized** personnel having access to the key). The POTW should contact the EPA Regional office (Pretreatment Coordinator - see the list of Regional Pretreatment Coordinators in Appendix XI) to get information on how to obtain these security clearances. Obtaining top level security clearances can take up to one year, so you should plan ahead to avoid unnecessary delays. In the interim, it may be necessary to contact the EPA Regional Office for assistance in conducting inspections at these facilities.

**Denial of Consent to Enter.** If an inspector is refused entry into a facility to conduct their inspection under an appropriate State or Local law, the following procedural steps should be taken:

- **Present Credentials.** Make sure that all credentials have been presented to the facility owner or **agent-in-charge**;
- **Tactfully Discuss the Reason for Denial.** If **entry** is not granted, courteously ask why. Diplomatically probe the reason for the denial to see if obstacles (such as misunderstandings) can be resolved. If the resolution of these conflicts is beyond the inspector's authority, he or she may suggest that the facility **officials** seek advice from their attorneys regarding a clarification of the **POTW's** inspection authority and right of entry;
- **Carefully Record Observations in Your Field Logbook.** All observations pertaining to the denial should be noted carefully in the inspector's **field** logbook. Specifically, note the following:
  - Facility name and exact address;
  - Name, title, and authority of the person who refused entry;
  - Name, address, and telephone number of the facility's attorney (if readily available);
  - Date and time of refusal;
  - Reason for the denial; and
  - Facility appearance (e.g., neat and orderly, or chaotic);

All of this information will be helpful in case a warrant is sought.

- **Avoid Threatening or Inflammatory Statements.** Under no circumstances should the inspector discuss potential penalties or do **anything** that may be construed as coercive or threatening. For example, the POTW has the right to ask for a warrant under normal circumstances. Therefore, **refusal** to permit entry to conduct the inspection is not likely to lead to any action against the industry, providing that the refusal was based on the inspector's lack of a warrant and there isn't an emergency situation as described above. If the inspector were allowed to enter the facility based on a threat of enforcement liability, it is likely that any evidence obtained through such an inspection would be deemed inadmissible in an enforcement proceeding.

On the other hand, an inspector may inform the facility representative that he or she intends to seek a warrant to compel the inspection. However, the inspector should be careful how this statement is phrased. Do not state: "I will **get** a warrant." If an enforcement action is brought against this facility using the information obtained in that inspection, a reviewing court may feel that the above statement usurped the **court's** authority to authorize a warrant and may deny the warrant. Even if the company later consents to the inspection following a statement that the inspector will get a wan-ant, there **may be** an issue as to whether consent was coerced. If the inspector decides to make a statement regarding a warrant, it should be phrased similar to: "I intend to seek (or **apply** for) a warrant."

- **Leave Premises and Contact Supervisor.** If entry is still denied after attempting to resolve the obstacles, the inspector should leave the premises **after** obtaining the information noted above in the field logbook. The inspector should contact his or her supervisor immediately **after** leaving the premises. and the supervisor should confer with the **POTW's** legal counsel regarding the desirability of obtaining a warrant. The POTW's legal counsel should attempt to resolve the conflict by contacting the facility's legal counsel prior to obtaining a **warrant**.

**Withdrawal of Consent Durinn an Inspection.** Occasionally, a facility may consent to an inspection and later withdraw the consent while the inspection is in progress. Consent for the inspection **may be**

withdrawn at any time after entry has been made. A withdrawal of consent is tantamount to a **refusal** of entry. Therefore, the inspector should follow the procedures cited above under "Denial of Consent" **unless** the inspection has progressed far enough to accomplish its purposes. All activities and evidence obtained prior to the withdrawal of consent are valid and may be used in an enforcement proceeding against the facility.

**Denial of Access to Parts of the Facility.** If, during the course of the inspection, access to some parts of the facility is denied, the inspector should make a note of the circumstances surrounding the denial of access and of the portion of the inspection that could not be completed. The inspector should then proceed with the rest of the inspection and should contact his or her supervisor **after** leaving the facility to determine whether a warrant should be obtained to complete the inspection.

**Covert Sampling in Response to Denial of Entry.** Whenever **entry** to a facility is denied, a sample should be obtained at a manhole immediately downstream of the facility, if possible (NOTE: the inspector should be aware of the potential difficulties with the sample, i.e., are other facilities connected to that part of the sewer which discharge the pollutants of concern?). This type of sampling, however, may help with any **further** enforcement actions or investigations which the POTW may undertake at the facility by uncovering activities which the industry is attempting to hide. This type of sampling is also **effective when a** demand inspection is being conducted because the POTW personnel can then compare the results of sampling from inside and just outside the plant to see if they match. This can provide evidence of any batches being dumped prior to entry to the facility.

### *CONDUCTING AN INSPECTION UNDER A WARRANT*

As an alternative to conducting an inspection with the consent of the facility, inspectors **may** conduct inspections under a search warrant issued by a magistrate or judge. If a search warrant is obtained prior to the inspection, the inspection may be conducted whether or not the facility **officials** consent to the inspection. (NOTE: Under these circumstances, it may be necessary to have the assistance of the police or sheriff to gain entry. This situation would only occur where the POTW knows that entry will be denied to the inspector). The Barlow decision from the Supreme Court (discussed earlier), **authorizes** the issuance of a warrant to inspect facilities without showing that a violation is probably occurring (probable cause requirement). When the **POTW** seeks a warrant, it must show that it has the authority to inspect industrial facilities. Obtaining such a warrant may be an appropriate part of the pre-inspection preparation process when the POTW suspects that entry may be denied, either absolutely, or temporarily until processes or records can be altered, or other actions **taken** to obscure violations of applicable pretreatment requirements.

### ***PRE-INSPECTION CHECKLIST***

No single list of documents and equipment will be appropriate for each inspection. **The majority of the** inspections which the POTW undertakes will be routine annual inspections required by the Federal Pretreatment Regulations. There may be instances, however, such as emergencies (e.g., spills at an IU, or complaints of problems at an IU), which might require an immediate site visit to assess compliance or ascertain the situation at the IU. **The POTW** must be ready to respond to such situations by having all inspection equipment and documents readily available and ready to go. **The** checklist outlined in Table 2-5 is intended to guide and aid the inspector in planning for **necessary** inspection supplies and activities.

### ***PRE-INSPECTION OBSERVATIONS***

Prior to entering the permittee's facility, the inspector should examine the facility's perimeter. By doing this, the inspector may detect leaky storage areas and other general housekeeping practices at the plant which might affect their discharge to the POTW. In addition, the inspector should **also** evaluate the environmental conditions near the plant, such as vegetation, odor problems, or direct discharges to streams. **It may also be** appropriate to take a sample at a manhole immediately downstream from the facility (**if** available) to determine the nature of the flow from the plant. This type of sampling may indicate problem areas that require further investigation. In this situation the inspector will need to know if that manhole is the only discharge from the facility.

The inspection report form and the field notebook form the basis for **the** written report made of **the** inspection and should only contain pertinent information and data. The language used in recording the inspection information should be objective, factual, and free of personal feelings or terminology. Notebooks can become an important part of the evidence package used by the POTW in an enforcement action and can be entered in court as evidence if properly maintained. All of the information developed as a result of the **inspection** should be dated so that the inspector can recall the information while writing **the** inspection report. This report should be written as soon as possible after the inspection or sampling visit.

The inspector should keep all pertinent information from the inspection in the **field** notebook. **This** information could include sampling measurements, flow measurements, production rates (if they are **needed** to determine compliance with any applicable mass based limits), process descriptions, nature of the facility, cooperativeness of the IU officials, general housekeeping observations (e.g., how clean or organized is the facility), and **any** other information learned from visiting the site. Representatives from the IU may request that any or all of the information collected be treated as confidential business information. **The POTW** must

**Table 2-5**

***Pre-Inspection Checklists***

***General Equipment:***

- Proper personal identification
- Camera
- Film and flash equipment
- Pocket calculator
- Tape measure
- Clipboard
- Waterproof pens, pencils, and markers
- Copy of the local sewer use ordinance
- Field logbook and maps
- Polyethylene bags
- Disposal towels or rags
- Flashlight and batteries
- Pocket knife
- Locking briefcase
- List of facility contacts
- Compass for direction on maps. etc.
- pH paper
- Evidence tape

***Sampling Equipment:***

- Siphoning equipment
- Weighted bottle sampler
- Liquid waste samplers
- Sample bottles/containers (certified clean bottles with teflon lids)
- Ice chest
- Flow meter (if applicable)
- Preservatives
- Thermometer
- Container for contaminated material
- Waterproof container labels
- Field test kits
- Field document records
- Vermiculite or equivalent packing
- Colorimetric gas detection tubes
- pH equipment
- Explosimeter (atmospheric testing device)
- Tubing, tape and rope

***Documents and Forms:***

- Entry warrant (if applicable)
- Notice of inspection (if applicable)
- Receipt for samples and documents
- Chain of custody for auto samplers
- Copy of IU's permit
- Chain of Custody Forms
- Hazardous sample shipping labels
- Inspection form checklist
- Copy of the local Sewer Use Ordinance

***Safety Equipment:***

- Safety glasses or goggles
- Face shield
- Ear plugs
- Rubber-soled. metal toed. non-skid shoes
- Liquid-proof gloves
- Coveralls, long sleeved
- Oxygen/combustion/H<sub>2</sub>S meter with alarm
- Air blower with 15 ft. hose
- Safety harness. tripod and hard hat
- Flashlight
- Self-contained breathing apparatus
- Confined space permit (if applicable)
- Plastic shoe covers
- Respirators and cartridges
- Self-contained breathing apparatus
- Manhole hook or pick
- Fire extinguisher
- Safety ladder (aluminum, chain, or rope)
- Safety cones
- Warning flags
- Particulate masks
- Traffic diversion devices

***Emergency Equipment:***

- Substance-specific first aid information
- Emergency telephone numbers
- First aid kit with eyewash
- Two-way communication radio
- Fire extinguisher
- Soap, waterless hand cleaner, and towels
- Supply of clean water for washing

honor this claim until or unless the POTW's legal counsel determines that the information is not **confidential**. In **some** instances it may be possible or necessary to elevate this evaluation process up the judicial ladder to the state or federal system for final determination

### **INFORMATION TO BE COLLECTED PRIOR TO AND DURING THE INSPECTION**

As mentioned earlier, the types of information that will be collected during an inspection will depend, in part, on the objective of the site visit. For a comprehensive compliance evaluation and site review most of the following information should be summarized prior to the inspection and updated during the inspection.

**The** information discussed in this section is reflected in the checklist questions found at the end of this **chapter**, and this **discussion** should be used in conjunction with **the** checklist and the checklist instructions.

- **Identifying** Information: Facility name, site address, mailing address, contact name, title, and telephone number.
- . General Background Information: Applicable categorical standards and local limits, applicable Standard Industrial Classification (SIC) code(s), number of **shifts** used, number of employees per shift, hours of operation, date facility commenced discharge to the sewer, date the categorical process commenced operation, etc.
- . Water Schematic: The schematic for water flow through the facility and the location of all wastewater discharge lines that flow to the POTW's system, along with major plant features.
- . Discharge Schematic: A description of each discharge's (including any batch discharges) amount, regulated pollutants, frequency, and destination.
- . Process Schematic: A description of each process flow from each major product line and process used within the plant, particularly processes that are subject to Federal Categorical Standards. Reactors, plating tanks and all types of process tanks can contain chemicals which maybe discharged periodically. Metal cleaning solutions are a prime example. The amounts, chemical nature, brand name, and frequency of discharge are all important. In addition, it is important to note how these wastes are disposed (i.e., discharged to the POTW or packed in drums as hazardous waste). If pretreatment of these solutions is practiced (neutralization, etc), this fact should be noted as well as the method used to determine that the waste has been treated to acceptable levels. General plant **washdown** (its frequency and quantity of water used) is also important. In many plants, the **washdown** is the largest **and/or** the most significant discharge.
- . Pretreatment **Systems**: A detailed description and appropriate sketches of each existing **pretreatment** facility, including operating data, if available.
- . List of Pollutants: The list should be divided into **two categories: 1) pollutants that come into direct** contact with the water that is discharged to the POTW; and 2) pollutants that do not come into direct contact with water discharged to the POTW, but which have the potential to enter the **wastewater** due to spills, **machinery** malfunctions, etc.
- . **Sampling** Locations: A list of all sampling locations used at the facility.
- . Chemical Storage: The proximity of chemical storage to floor drains and whether floor drains discharge to storm or sanitary sewers. The volume of all hazardous chemicals **encountered should be** listed. Any floor drains should be noted. If the chemicals stored are unknown, note the brand name, use and chemical supplier and obtain all material safety data sheets for all chemicals used **at** the plant.

The supplier's address should be noted in case it is necessary to contact them to obtain the **necessary** chemical information.

- . **Slug Control Plans:** A description of all spill control practices used by the IU, including information on all past spills, unusual discharges, or temporary problems with any of the process units that may affect the wastewater discharged to the POTW.
- . **Air Pollution Control Equipment:** A description of all air pollution control equipment that may generate a **wastestream**, the pollutants that are likely to be found in this wastestream, and the discharge or disposal method used. In some industries, the **effluent** from air scrubbing may be the principal waste source and may contain a wide variety of process chemicals which are not encountered in any other wastestreams. For example, booths for spray painting sometimes use a water column as a "water curtain" for fume control.
- . **Sludge Disposal:** A description of how waste residuals (sludge) from the pretreatment operations are handled, stored, **and/or** disposed. Many industrial processes such as cleaning, **degreasing**, grinding, or chemical pretreatment produce sludge which must be disposed. How this occurs, how **often**, and the quantities involved are all important. For example, vapor degreasers are used for cleaning metal in a wide variety of industrial applications. They almost always produce a sludge and solvent waste, and are usually water cooled, producing a steady stream of uncontaminated cooling water. The presence of these devices should always be noted as well as appropriate answers to questions concerning the wastes associated with them. As is the case with batch discharges, any waste disposal service should be recorded.
- . **Boiler Blowdown:** A description of the **biocide(s)** or **algicide(s)** used in the boiler maintenance program. The chemicals used in this process may be chemicals of concern, especially for sludge disposal by the POTW (e.g., molybdenum compounds).
- . **Operational Problems:** A description of any operational problems or shut-downs of pretreatment facilities since the previous inspection.
- . **IU Water Bills:** The inspector should be familiar with trends in the **IU's** water consumption and wastewater production. This information can be obtained by a careful review of the facility's water bills. A mass balance approach should be taken to pinpoint any areas of water loss or potential bypass.
- . **Compliance Information:** The inspector should review all previous **IU** compliance sampling data, as well as all data obtained by the POTW on the facility to be inspected.
- . **RCRA** (i.e., Hazardous) Wastes: A description of all hazardous waste generated or stored at the site and the manner of disposal for all such waste, especially any disposal to the **sewer**.

Each of these areas of a complete compliance evaluation are contained in the checklist which is presented at the end of the chapter. This checklist should provide the basis of the information collected during the inspection, unless the scope of the inspection does not require a complete compliance evaluation (e.g., a response to an emergency situation may require only very specific information and not include the general information contained in the checklist).

### *ON-SITE ACTIVITIES*

This chapter has thus far addressed the procedures which should be followed when planning and preparing for the inspection visit at a regulated **IU**. In doing so, we have covered the "why" and "what" of

inspections. We now turn to the on-site activities of the site visit. These activities can be characterized as the "who," "how," and "where" aspects of conducting inspections and form the core of the inspector's hands-on activities.

The on-site activities at the facility form the core of the inspection. Once on-site, the inspector is responsible for developing a complete picture of the IU's manufacturing and process operations, wastewater treatment operation, compliance activities, and records management. Using the industrial inspection to its fullest extent will depend upon the ability of the inspector to ask the right questions and to look closely in the appropriate locations while on-site. This will require inspectors to become thoroughly familiar with industrial treatment processes, wastewater sources and treatment technology, as well as with correct inspection procedures and techniques (e.g., interviewing and observation). The principal goal of the IU inspection is to gather information that can be used to determine compliance with all applicable requirements, including permit conditions, regulations, and other State or local requirements.

An industrial pretreatment facility consists of wastewater treatment processes designed to remove pollutants from wastestreams prior to discharge to the local sewer system. Pretreating these wastes is the method used by many industries to comply with local waste discharge ordinances and permits, and also federal and state regulations. The sources, amounts, and types of wastes generated at an industrial manufacturing or processing site depend on the age of the facility, raw materials used, production processes, and the ability to recover or recycle wastes generated as a result of industrial activity. Some industries attempt to minimize the different wastestreams by controlling them at the source (i.e., pollution prevention), while others gather all wastestreams together for treatment at one central location.

Physical, chemical, and sometimes biological treatment processes are used to separate or remove pollutants from these wastestreams. These treatment processes should be closely controlled by plant personnel to produce discharges which are acceptable to the POTW. To ensure that industrial dischargers meet all applicable requirements, the pretreatment facility inspector must inspect each of the treatment processes or facilities at any industrial site which has the potential to discharge toxic or hazardous wastes to the POTW. This section outlines the procedures used in the NPDES program for industrial inspections. These procedures should be followed by POTW inspectors when conducting their site visits.

### *Opening Conference:*

Once proper credentials have been presented and legal entry has been established, the inspector can proceed with the on-site activities of the inspection. If this is the first visit by the POTW to the IU, or if the management team at the IU has changed significantly since the last inspection, the on-site activities should

begin with an opening conference with facility officials to outline what will be covered during the inspection, the purpose of the inspection, and the procedures which will be followed. Once a rapport with facility officials has been established, subsequent inspections and sampling activities at the IU may not require a detailed opening conference as described below, or may not require a conference at all. (**NOTE:** The inspector should always inquire if any changes have been made at the plant since the **last** inspection. This can be done during an **official** opening conference or it may be done informally at the beginning of the inspection. If changes have been made which affect the discharge to the **POTW**, the **IU** is required to report such changes. If the **IU** has not reported such changes, it should be considered a reporting violation and brought to the attention of appropriate **IU** officials). **The** inspector should gauge the level of awareness of the **IU officials** with the **POTW's** role in the pretreatment program, and the inspector should continue opening conferences until the **IU** clearly understands **th** : significance of its responsibilities. The **POTW** should encourage cooperation between the facility **o** **fficials** and the inspector(s) to ensure that the activities go smoothly. This section addresses the role of the inspector in the opening conference. along with relevant meeting agenda topics. This section also describes possible mid-course adjustments which might be needed as a result of the information discussed during the opening conference.

The opening conference establishes a forum for exchanging information between the **POTW** inspector and facility personnel. This information exchange should focus on the inspection, but it does not need to be limited to the inspection itself. The inspector should use the following principles when conducting the opening meeting:

- . Gain an early rapport;
- . Start the meeting on a positive and professional note;
- . Prepare and use any supporting information that will enhance the discussion (e.g., a copy of the local regulations or statute authorizing the inspection, pollution prevention materials or technology transfer information which might allow the **IU** to operate more **efficiently**). **If you can provide the IU with information which it might find useful, the inspector will be viewed as a resource and not a burden on the facility;**
- . Acknowledge that the inspection may disrupt daily facility routines, but assert that reasonable efforts will be made to minimize such disruption;
- . Listen carefully and be willing to answer the facility **official's** questions, but do not **permit** yourself to be maneuvered into bending **POTW** policies/procedures or overstepping your authority in an attempt to accommodate facility representatives. For example, facility representatives will be understandably very curious about how they are performing vis-a-vis the requirements of the pretreatment program Do not forget that the inspector's primary objective is to inspect the facility for compliance with discharge requirements. The inspector is not there as a consultant to solve technical problems for the company, but if through the inspector's experience or technical expertise the inspector can describe how similar problems have been handled successfully. the inspector may be able to help the industry solve its problem. Be cautious about giving advice. The inspector should not "advise" the **IU** on how it could come into compliance. Such information, if followed, could be used as a defense in a future

enforcement proceeding. **The IU** has the primary responsibility to ensure compliance. It is the inspector's job to evaluate the IU based on the requirements established in its permit or in the local sewer use ordinance. It is not the inspector's job to tell the **IU** how to come into compliance.

A cooperative working relationship developed during this opening meeting can set the tone for the rest of the inspection. It can also be used as the foundation for strengthening ties between the POTW and its regulated industries. If approached properly, the opening conference provides an ideal opportunity for the inspector to function as a public relations liaison and educator. The inspector should ensure that he or she provides tactful assistance before, during and after the inspection, but does not provide information which the IU can use as a defense in a later enforcement action. During the opening conference, the inspector should attempt to cover the following topics with the facility officials:

- **Inspection Objectives:** By informing facility officials of the purpose and scope of the inspection, it may help to avoid misunderstandings and facilitate the work of the inspector.
- **Order of the Inspection:** If the inspector also discusses the order in which the inspection will be conducted, it will eliminate wasted time by allowing officials at the **IU** the time to make any requested records available.
- **Meeting Schedules:** The inspector should schedule meetings **with** key IU personnel (perhaps beforehand) to allow facility officials adequate time to spend with the inspector during the **inspection**. It is important that a facility representative accompany the inspector during the inspection, not only to answer questions about the facility and to describe the plant and its operating processes, but also for safety and liability considerations. If these needs are discussed prior to the inspection, it allows the IU an opportunity to make someone available.
- **Permit Verification:** The inspector should verify the pertinent sections of the **IU** permit, e.g., name and address of the facility, discharge points, and proper use of the Combined **Wastestream Formula** (if applicable).
- **Safety Requirements:** The inspection should verify which safety requirements (if any) are required at the facility, to ensure that appropriate preparations were made.
- **Photographs:** Photographs are a useful tool for documenting inspection information, and may prove useful in any enforcement proceedings against the facility. Facility officials, however, may object to the use **of** cameras on their property (especially Federal military facilities or defense contractors). If a mutually acceptable solution can not be reached, and if photographs are considered essential for the inspection, the inspector should conclude the inspection without photographs and consult the **POTW's** legal counsel for additional information. If facility officials request that photographs be considered confidential, as with any other information so identified, **the** POTW is obliged to comply with this request pending further legal determination. When taking photographs it may be useful for the inspector to use a camera which takes pictures with the dates imprinted on the photograph. This can prove useful when storing and retrieving photographs for enforcement purposes. Also, when taking pictures, it is often useful to include reference objects in the photograph to judge the distance and size of objects. This creates a more substantial picture of the scene and may be useful when pursuing an enforcement action.

*Inspection Procedures:*

## Physical Plant Review

**When** an inspector performs an industrial facility site review, his or her primary areas of investigation include the facility's pretreatment units, monitoring equipment, and production processes. The purpose of a facility site review is to examine the permittee's premises for problem **areas** and to verify existing **POTW** file information on the IU. This overall review allows the inspector to gain a feeling for the facility being inspected and to review areas that may indicate problems with plant operations or effluent limitations. **In** particular, the inspector should focus attention on areas of the **IU's** premises where regulated pollutants are produced, pumped, conveyed, treated, stored or recorded. This type of facility site review requires that the inspector understand **fully** the wastewater treatment processes used at the industrial facility and how each process fits in with the overall treatment scheme. The objectives of this type of comprehensive review are to:

- . Evaluate the flow of raw water used for production culminating in wastewater discharged to the POTW, the facility's water consumption and distribution usage, and the hydraulics of the facility's drainage and collection system;
- . Understand the **flow** of **raw materials** and any additives used in production **as** well as all end-products; by-products; and other liquid, gaseous, and solid wastes resulting from the production process;
- . Assess the conditions of the facility's current treatment processes and operations;
- . Evaluate the wastewater characteristics (only possible if the inspector samples the effluent);
- . Evaluate the **IU's** operation and maintenance activities;
- . Check the completeness and accuracy of the **IU's performance/compliance** records (e.g., production levels, results of self-monitoring, etc.); and
- . Determine if the treatment units are being operated as efficiently as possible.

In the course of the **site** visit, the inspector should become more knowledgeable about the facility, including areas that may indicate problems with effluent limits and overall operation and maintenance of the facility. **It** is to the inspector's advantage to conduct the facility review as soon as possible upon entering the facility. This prevents the permittee from altering any problem areas. **After** completing the preliminary discussions with the plant officials, the inspector is ready to tour the facility. During the plant tour, the inspector should be alert to and inquire about any of the following areas:

- . Vital treatment units out-of-service for repairs. (The inspector should determine when the units were taken out of service, the type of failure experienced, and when the units will be put back in service).
- . Any unusual equipment or operations such as special pumps, floating aerators in **diffused** air systems, chemical feeders, construction, temporary structures, or any rigged systems intended to correct operational problems;
- . Adequate safeguards to prevent the discharge of untreated or inadequately treated wastes during electrical failures;
- . Any evidence **of** spills and/or leaks, including proper storage of chemicals, which may enter the sewer (the inspector should ask questions of the employees to see if they are familiar with any required spill

or slug control measures at the plant and to determine if the IU provides any training on safety or slug control measures)

- Unauthorized discharge points and/or bypasses, channels, or other areas likely to experience overflows. (The inspection should determine if spills or unauthorized use has recently occurred as a result of facility staff attempting to correct operational problems; and
- Disposal of collected screenings, slurries, sludges, or other by-products of treatment. (These materials, including wastewater should be disposed of in a manner so as to prevent the materials from entering navigable waters or their tributaries);

One of the principal areas which the inspector should evaluate is the level of production at the facility.

Industries frequently make production changes because of advances in technology and the availability of new products. Therefore, during the tour of the facility, the inspector should inquire whether the permittee has made any changes to: production processes; raw material usage; amount of finished product; water use; waste treatment processes; and other such changes.

The inspector should also inquire whether the permittee has modified any production process that would change the pollutant loading to the POTW, and whether the POTW had been notified of such changes. This is especially critical if the industry has limitations which are based on the combined wastestream formula (CWF). The inspector must assess the impact which any changes in the discharge of wastewater will have on any existing limits based on the CWF. Finally, the inspector must ensure that any increases in wastewater flow is not used as dilution to meet any applicable pretreatment limits. If dilution is suspected at an industrial facility, the inspector should trace each plumbing line to determine where it originates and where it goes. This may be very time intensive, but it is the only way to accurately assess the existence of dilution at the facility, since it is unlikely that dilution lines will show up on any schematics that the company provides. The inspector should verify any changes in production processes or pollutant loadings and include the results in the Inspection Report prepared after the inspection.

In addition, the inspector should check the appropriateness of monitoring locations, the existence, condition and calibration of the permittee's self-monitoring equipment (both field and laboratory), and the facility's maintenance program for this equipment. During the physical "walk through" of the facility, the inspector should observe all areas which have current or potential problems. Each of these observations should be carefully documented in the inspector's field notebook because of their potential sensitivity during an enforcement proceeding. It is often useful when trying to understand the industrial facility's process to follow the process in a sequential order of production.

#### Self-Monitoring Review:

The site review at the industry should include an examination of the permittee's self-monitoring program. To perform this review thoroughly, it is the inspector's responsibility to be familiar with the monitoring

requirements contained in the facility's permit and with any correspondence which may have modified or replaced sampling points or analytical procedures. The inspector should also be thoroughly familiar with all approved test methods and the specified sample holding times and preservation techniques. or in the case of a complex array of methods, the inspector should have a reference list available of the approved methods for those samples required by the permit. (See Table 3-4 in Chapter 3 for an overview of appropriate holding times and analytical methods for various pollutant parameters).

There are two **objectives** of the self-monitoring review: 1) confirm that sampling and flow measurement equipment are provided as required in the permit and that they are being operated, calibrated and maintained properly; and 2) confirm that the analytical test methods used to evaluate pollutants or parameters specified in the **IU** permit conform with the EPA's regulations at 40 CFR Part 136. When conducting the self-monitoring review, the inspector should:

- . Verify that flow measurement devices are in use (if required) and are adequate to handle the expected ranges of flow rates;
- . Verify that samples are taken at the locations prescribed in the **IU** permit;
- . Verify that the sampling location specified in the permit is adequate to provide a representative sample of the regulated discharge;
- . Verify/determine that the appropriate limits are being applied at the specified sampling locations;
- . Verify that the frequency of sampling is performed in accordance with the permit's requirements and that this frequency is adequate for the nature of the facility;
- . Verify that samples are collected and preserved in accordance with 40 CFR Part 136;
- . Verify that samples are analyzed within the holding times and analyzed according to approved test methods in 40 CFR Part 136;
- . Verify that appropriate procedures are used by the industrial user when the **IU** pulls its own sample (i.e., observe the **IU** pulling a routine compliance sample), and observe the **IU** when it conducts its measurements of flow **and/or pH** (it should be a common practice for the inspector to pull a **pH** sample and compare the results with the **IU**);
- . Verify that **QA/QC** procedures used by the **IU** in its self-monitoring program are adequate; and
- . Check all sampling, monitoring, and laboratory equipment to verify that all equipment is in working order and has been operated, maintained and calibrated (including O&M and calibration logs) correctly.

#### Operations Evaluation.

The operating factors at the facility which might affect plant performance range from qualitative factors such as the skills and aptitudes of the operators (e.g., process knowledge and general aptitude), to physical deficiencies in laboratory equipment or a lack of flexibility in process equipment. The evaluation of operation activities must focus on **wastewater** treatment, and laboratory analysis. **This** evaluation should be based on the following topics:

- . Policies and Procedures;

- Staffing and Training;
- Health and Safety; and
- Management controls.

Appendix III presents the basic review questions that an inspector should ask to evaluate the operation and maintenance activities at the facility. These questions are detailed and comprehensive, and it is probably not necessary to cover all of these points with each inspection. The POTW should make sure that these areas are covered during the course of the IU's permit cycle (e.g. if the IU's permit is issued every three years, the POTW should cover the operation and maintenance questions at least every three years, unless there are suspected problems at the plant which relate to the operation and/or the maintenance of the facility. NOTE: The permit issuance cycle should never exceed five years).

**Policies and Procedures.** Written operating procedures and standard reference texts enable the operator of the process lines or wastewater treatment equipment to achieve efficient plant operation. The operations manual prepared for the facility is the most important reference that an inspector should review when evaluating plant policies and procedures. Other reference materials which should be available relating to the operation of the facility include: manufacturer's literature, publications by professional organizations (e.g., the Chemical Manufacturer's Association). and EPA publications.

**Staffing.** Even the best engineered facility cannot perform to its potential without a sufficient number of capable and qualified staff. Staff interviews are an important component of this evaluation, and the questions outlined in Appendix III can be used to ascertain the quality of the operations at the facility. The inspector should make an attempt to interview the individual in charge of overall operation at the facility, the chief operator (if different), specific unit process operators, and the laboratory staff.

**Health and Safety.** At all times, safe operating procedures should be followed by the regulated facility's personnel. Employees must be trained in emergency shut-down, fire control, and spill response procedures, as well as in the use of safety equipment. Each of these areas can adversely affect the nature of the discharge to the POTW by allowing unregulated or uncontrolled amounts of pollutants to enter the POTW's system. The authority for such an evaluation is found at 40 CFR 403.8(f)(2)(v) which requires an evaluation of each SIU for the need to adopt a slug control plan. The inspector should also verify that the Material Safety Data Sheets required by the Right-to-Know law are readily available at the facility. This law also requires a written hazard communication program (including notification to the POTW, which is also covered by the General Pretreatment Regulations). and labeling of chemicals (to ensure that incompatible chemicals are not stored together, e.g., cyanide compounds stored with acid compounds).

**Management** Controls. Monitoring practices are a good indicator of both the emphasis placed on operations and the operator's understanding of process controls. Factors affecting a facility's monitoring capabilities are:

- . **The** sampling program;
- . Performance testing;
- . Analytical capabilities; and
- . Record keeping practices.

An effective process control program is essential to a treatment facility's optimal performance. However, process control cannot be quantified easily by the inspector, therefore, in most cases, the inspector must rely on discussions with plant personnel (e.g., operators) to supplement available records and the technical evaluation. Again, the questions outlined in Appendix III can be used to evaluate the quality of the facility's operations.

Maintenance Evaluation:

Facility maintenance directly affects the ability of the facility to run efficiently and to comply with its IU permit. There are two types of facility maintenance which should be conducted at the plant:

- . Preventative Maintenance: Reduces the facility operating costs by eliminating breakdowns and the need for corrective maintenance. It improves the facility's reliability by minimizing the time the equipment is out of service. It increases the useful life of the treatment and process equipment, thus avoiding the need for costly premature replacement which may cause an interruption of wastewater treatment at the facility. Each of these items, if adequately addressed, reduces the possibility of compliance problems at the facility. Therefore, it is important that the inspector evaluate these areas to ensure compliance with all applicable program requirements. AND
- . Corrective Maintenance: Returns the malfunctioning equipment to operation. This has compliance implications because the malfunctioning equipment may be in a treatment process necessary for compliance with the pretreatment program requirements. Therefore, the inspector should evaluate the procedures the facility uses to identify and correct instances of malfunctioning equipment.

**The** principal areas of concern for both the operations and maintenance evaluation are: **staffing** and training, planning and scheduling, and management control (i.e., records systems and inventory control). Only **well-**trained, competent staff can be expected to perform adequate physical inspections, repairs, and preventative maintenance. Wastewater facility maintenance is complex and requires a variety of skills. Because many of these skills are not readily available, an ongoing **training program** is essential. The **plumbing and scheduling** of maintenance is also essential for effective preventative and corrective maintenance. Ensuring that an adequate plan and schedule is in place is an important task for the inspector. A detailed records **system** is the basis of any maintenance program. Records are used to establish the maintenance histories of equipment, diagnose problems, and anticipate (and thereby avoid) equipment failure, making records an effective tool for preventative maintenance. A central **inventory** of spare parts, equipment and supplies should be maintained. The extent of the inventory should be adequate to avoid process or treatment interruptions. A **maintenance**

cost **control system** should be an integral part of every wastewater treatment facility at the **IU**. Evaluating costs in this manner serves to control expenditures and can be used as a baseline for future budgets. This will help ensure that all **necessary** process and treatment equipment is operated continuously and effectively, thereby ensuring that the regulated facility operates within the limits specified in **its permit**.

#### Records Review at the Industrial User

The General Pretreatment Regulations require Significant Industrial Users (SIU) to submit reports **at** least twice per year on the nature and concentration of pollutants in their discharge and the flow from the plant to the **POTW**. Each SIU is required to **maintain records** of all the information obtained from their monitoring activities **for a period of at least three years**. However, many IUs, as well as many **POTWs**, maintain records for at least five years to coincide with the statute of limitations on **penalties**. These records contain **a variety** of information which may be useful to ascertain the facility's compliance status with its permit requirements. Examining these records is a key part of **the** inspection process for the POTW.

Records and files **may** be stored in a variety of information retrieval systems, including written or printed materials, computer or electronic systems, or **visual** systems such as microfilm and microfiche. Conducting an effective records review is an important investigative skill for the POTW inspector, but it is an art that is developed largely through experience and practice. No set of instructions can prepare an inspector for the variety of records and record keeping systems they are likely to encounter. This process can be **difficult** because of the complexity of the industrial processes being regulated and the infinite variety of record keeping systems which can be used to document how these **processes are operated** and maintained. This complexity makes it **difficult** for the inspector to achieve his or her goal, i.e., to verify or determine whether or not a facility is in compliance with its applicable permit requirements.

The POTW inspector should review the IU's permit prior to conducting the inspection (it may even be useful to have a copy of the permit along on the inspection) to determine the facility's record keeping requirements. Throughout the inspection, the facility's operations should be compared with the permit to verify that required permit activities are correct, current, and complete. **Some of the information needed to verify** the permit can be obtained during the opening conference and compared with the facility permit. This general information may include: correct name and address, correct name of the facility contact, number and location of discharge point(s) to the POTW, and the facility's principal products and production rates (what there are production based standards in place).

The inspector should check **for** records which will verify that proper notification was made by the facility to the POTW if: 1) discharges have changed from those stated in the **IU** permit (e.g., additional discharges,

or significant increase in flow), 2) a permit violation has occurred (e.g., the permittee is required to notify the **POTW** within 24 hours of becoming aware of an **effluent** violation and to resample the discharge within 30 days), 3) any discharge of a hazardous material (as defined in 40 CFR 261) has occurred from the **IU** (NOTE: This notification must be sent to the POTW, the U.S. EPA Regional Waste Management Division Director, and the State hazardous control authorities), or 4) a bypass has occurred. These record keeping requirements are outlined in the federal regulation at 40 CFR 403.12. The inspector should also check to ensure that the appropriate records are being kept for a minimum of three years. These records will likely include many of the following types of information:

- Sampling and Analysis Data:
  - Dates, times, and location(s) of sampling;
  - Sampling techniques (e.g., grab or composite) and analytical methods used;
  - Results of the analyses;
  - Dates of the analyses; and
  - Name(s) of analysis and sampling personnel.
- . Monitoring Records:
  - Self-monitoring reports (if applicable). including flow, pollutant parameters, etc. as required by the permit; and
  - Original charts for continuous monitoring instrumentation and bench sheets for analyses.
- . Laboratory Records:
  - Calibration and maintenance equipment and schedule;
  - Calculations (e.g., bench sheets or books); and
  - **QA/QC** analysis data.
- . Facility Operating Records:
  - Daily operating log;
  - Summary of all laboratory tests run and other required measurements (if applicable);
  - Chemicals used (e.g., pounds of chlorine per day. etc.);
  - Weather conditions (temperature, **precipitation**, etc.);
  - Equipment maintenance schedules; and
  - **Sludge/RCRA** disposal records and waste hauling manifests.
- . Slug Control Plan (if applicable);
  - When required, a properly completed Slug Control Plan should be available to the inspector.

The inspector should document all records review activities and should note all inadequacies, discrepancies, or other problems disclosed or discovered during this review. Any identified problems may warrant a more intensive investigation. This decision should be made by the inspector in conjunction with **POTW** officials.

A primary objective of **the** records review at the industrial user **includes** a comparison of the bench sheet data and laboratory report summaries to the values **reported** on the self-monitoring reports submitted by the

facility. This evaluation is critical to determine if **all** data are correctly summarized on the self-monitoring **reports** received by the POTW.

As mentioned above, the IU is required to keep complete and accurate sampling records, and a failure to do so is a violation of the Federal Pretreatment regulations. The **POTW** should treat this failure to keep records as a violation and respond with the appropriate enforcement response, as identified in its Enforcement Response Plan. A review of facility records must determine if the **IU** is complying with the sampling and the record keeping requirements of the General Pretreatment Regulations contained in its **IU** permit. In particular, the inspector should verify that the IU is keeping the following records for all samples: the date and time for each sample, the date(s) of each analysis, the exact place the sample was taken (i.e., location of the sample point), the analytical **techniques/methods** used for all samples, the name of the person who took each of the samples, the name of the person who performed the analysis, and the results of each of the analyses.

Obtaining **Copies** of Necessary Records:

**When** copies of records are necessary the IU must make these records available in accordance with 40 CFR 403.12(o). The inspector must consider how to retrieve and store the required records. The following outline may be useful in determining the appropriate means of accessing and securing **certain records**.

- **Written or printed records** generally can be photocopied on-site. Portable photocopy machines may **be** available to the **POTW** inspector, but in the absence of this equipment, inspectors should be authorized to pay a "reasonable" price for the use of facility copying equipment.
  - At a minimum, all copies made for or by the inspector should be initialed and dated for identification purposes (see identification details below).
  - When photocopying is impossible or impractical, close-up photographs may be taken to provide suitable copies.
- **Computer or electronic records** may require the generation of hard copies. Arrangements should be made at **the** time of entry or during the opening conference for these copies. Photographs of computer screens **may** provide adequate copies of these records if no other means are available.
- **Visual systems** (microfilm and microfiche) may have photocopying capacity built into the viewing machine, which can be used to generate copies. Photographs **of the viewing screen may provide** adequate copies if hard copies can not be generated.

Record Identification Procedures:

Immediate and adequate identification of the records reviewed by the inspector is essential to ensure a legally binding custody process which ensures the admissibility of the records in court. If an inspector is called to testify, he or she must be able to identify positively each particular document and state its source and the reason for its collection. This identification can be accomplished by initialing, dating, **numbering, and** entering **each** of the records in the inspector's notebook under the facility's name.

- **Initialing/Dating:** Each inspector should develop a unique system for initialing and dating the records and copies of records so that he or she can easily verify their validity. This **can** be done by initialing each document in a similar position, **or by another method, at the time of collection. Both** the original and the copy should be initialed in the same fashion. All record identification notations

should be made on the back of the document. The inspector must be able to identify positively that he or she so marked the document.

- **Numbering:** Each document or set of documents substantiating a suspected violation(s) should be assigned an identifying number unique to that document. The number should be recorded on each document and in the inspector's field notebook.
- **Logging:** Documents obtained during the inspection should be entered in the field notebook by a logging or coding system. The system should include the identifying number, date, and other relevant information, such as: the reason for copying the material (i.e., the nature of the suspected violation); the source of the record (i.e., type of file, individual who supplied the record); and the manner of collection (i.e., photocopy, other arrangement).

The originals of each document must be returned to the proper person or to their original location, and related records should be grouped together for ease of reference. Confidential business records should be handled according to the special confidential provisions discussed earlier in this chapter.

### *Closing Conference:*

To achieve the most effective results from the compliance inspection, the inspector must communicate the results of the inspection promptly to the facility's management **and/or** operating personnel. The inspector's discussion, however, should be limited to the specific findings of the site visit. **If** appropriate, the findings should be compared with the industrial user's permit requirements, consent decrees, administrative orders, **an&or** other enforcement actions. Even though a discussion of the inspection findings is important, certain precautions are essential when conveying this information. **The** inspector should keep the following guidelines in mind when presenting any findings from the site visit.

- The inspector should be cautious about discussing the compliance status or any legal enforcement consequences with the industrial user representatives or with facility operating personnel. On the other hand, if there are violations which are clearly identified during the inspection, and the inspector is confident that those violations are "actionable", then the inspector should bring these violations to the attention of the IU representative. This should be done in writing, either through a Notice of Violation (**NOV**) or **Deficiency** Notice (**DN**) (see Figure 2-1 for an example form for the deficiency notice). The inspector's purpose is to call attention to and explain the violation but **not** to predict the consequences or penalties that may occur beyond the **NOV/DN**.
- The inspector should refrain from recommending a particular consultant or consulting **firm**, or any particular treatment system, even if asked to do so. Inspectors should tell the permittee's representative to contact a professional society or approved listing for advice on how to come into compliance with all applicable permit requirements.

A deficiency notice identifies existing or potential problems specific to the permittee's self-monitoring program. Issuing a deficiency notice on-site or after the site visit provides a **swift** and simple method for improving the quality of the data submitted by the industrial user. This type of notice allows the inspector to assign formal responsibility to the permittee, and to track each stage of the compliance/enforcement process with respect to the **IU's** self-monitoring program. This notice is designed to alert the facility to deficiencies in their self-monitoring activities and to assist the industrial user in complying with its permit

## ***FOLLOW UP ACTIVITIES***

Follow up activities from the inspection are necessary because this is how the information from the inspection is translated into an action by the POTW (e.g., an enforcement action or decision to modify the IU's permit). The POTW has the primary responsibility to ensure compliance with all applicable pretreatment requirements, and the inspection is an important mechanism for achieving this goal. Once the inspection has been completed, the inspector must summarize his or her findings in a report (a standard form for this report should be developed by the POTW) so that inspection findings can be admitted in court. The POTW may also choose to use the inspection checklist at the end of the chapter as the basis of its inspection report format. This report should be placed in the IU's file for future reference as background material for subsequent inspections.

Every POTW should have systematic procedures for tracking industrial user problems, including permanent records of all problems kept in office files or computers. In this way, the information can be reviewed at any future date. If the inspector took effluent samples as part of the site visit, the results of these tests should be placed into the POTW's compliance tracking system (either manual or, preferably, automated) and appropriate action (as defined in the POTW's Enforcement Response Plan) should be taken if a violation is detected. It is in the POTW's best interest to conduct timely follow-up activities with the IU so that any identified problems can be addressed before they get out of control. Such follow-up activities will usually include some form of enforcement action, perhaps even formal enforcement action for significant violations.

## ***Inspection Report***

Once the inspection has been completed, the inspector should review his or her notes to identify areas which may require follow-up activities. The notes from the inspection should be organized into a report format. This is one of the most important points of the whole inspection procedure, yet it is often ignored. The need to write a clear and concise report which contains pertinent information to be used as a basis for future permitting, compliance, and enforcement decisions cannot be stressed enough. This chapter has outlined in detail the procedures for collecting and substantiating the information which should be used to prepare the inspection report.

The report accomplishes three objectives: 1) it organizes and coordinates all information in a comprehensive, usable manner for use by the POTW's compliance personnel, 2) it clearly identifies areas which require follow up activity, and 3) it provides significant background information on the facility which can be reviewed prior to conducting subsequent inspections at the facility. The quality of this report will, to a large degree, determine how effective these follow up activities will be at the facility. The preceding sections

have detailed the procedures for collecting and substantiating the inspection information. Once organized, this material should be translated into a report format developed by the POTW.

The information in the inspection report must be presented in a clear, concise, and well-organized manner. The information must be objective and factual; the report should not speculate **on the ultimate result** of the inspection findings (i.e., anticipate any enforcement action), but should stick with the facts as identified by the inspector. Of particular importance in the report are the following items:

- . **Accuracy:** The information in the report must be factual and based on sound inspection practices. Observations in the report must be the verifiable result of firsthand knowledge so that compliance personnel can depend on the report's accuracy when determining appropriate follow-up action (if any).
- . **Relevancy:** The information in the report must be relevant to the compliance status of the facility. Irrelevant facts and data will clutter the report and may reduce its clarity and usefulness. Personal comments and opinions must be avoided.
- . **Comprehensiveness:** All information pertinent to the industrial user's compliance status should be organized as a complete package. Documentary support (e.g., photographs, statements, sample results, etc.) accompanying the report should be referenced clearly so that anyone reading the report will get a complete, clear overview of the situation at the facility. The more comprehensive the evidence, the better and easier is the task of the compliance personnel when taking an action.

Each report should contain the following elements: the report form (developed by the POTW); supplementary narrative information; copies of the completed checklist (an example of a checklist is provided at the end of this chapter); and documentary support (e.g., photographs or sketches).

The contents of the report should be comprehensive (i.e., should include all pertinent observations from the inspection), but the report should focus on supporting or explaining the information provided in the inspector's notes. The narrative of the report should be a concise, factual summary of the observations and activities undertaken during the inspection, organized in a logical, legible manner, and supported by specific references to accompanying documentary support.

All documentation that is produced or collected by the inspector to provide evidence of suspected violations should be included in the inspection report. This type of documentation may include: the inspector's field notebook, statements, photographs, drawings and maps, printed matter, mechanical recordings, and copies of records. In general, the types of information contained in the report should reflect the type of information collected during the inspection.

FIGURE 2-1

EXAMPLE DEFICIENCY NOTICE

DEFICIENCY NOTICE

PERMITEE NAME AND ADDRESS

Pretreatment Program Administered by the (POTW Name)

PERMITEE REPRESENTATIVE (Receiving this notice)/TITLE

U# Permit No

During the compliance inspection carried out on (date) the deficiencies noted below were found. Additional areas of deficiency may be brought to your attention following a complete review of the Inspection Report and other information on file with the (Name of POTW)

DEFICIENCIES

MONITORING LOCATION (Describe)

FLOW MEASUREMENT (Describe)

SAMPLE COLLECTION/HOLDING TIME (Describe)

SAMPLE PRESERVATION (Describe)

ANALYTICAL METHODS (Describe)

RECORD KEEPING (Describe)

OTHER SELF-MONITORING DEFICIENCIES (Describe)

ADDITIONAL COMMENTS

REQUESTED ACTION - Your attention to the correction of the deficiencies noted above is requested. Receipt of the description of the corrective actions taken will be considered in the determination of the need for further Administrative or Legal Action. Your response can either be submitted with your next periodic compliance report or as directed by the inspector. Questions regarding this notice may be directed to (POTW's Name)

INSPECTOR'S SIGNATURE

INSPECTOR'S ADDRESS/PHONE

POTW'S ADDRESS

DATE

INSPECTOR'S PRINTED NAME

# Checklist Questions for POTW Inspectors

## I. General Inspection Information:

Date of inspection. Last inspection date

inspected by. Last inspected by

Type of inspection? Demand Scheduled

Did the previous inspection **identify** areas which the IU was required to correct? (Y/N)

What areas were **identified**?

What progress has the IU made in correcting the **identified** deficiencies?

Persons present **during** the inspection

	<u>Name</u>	<u>Title</u>	<u>Affiliation</u>
1.			
2.			

## II. General Facility Information

Industry name. SIC code(s): Permit on file? (Y/N)

Site address: Mailing address

Industry contacts (w/ titles) Fax #: ( ) Brief facility description

1. Phone #: ( )

2. Phone #: ( )

Applicable **categorical standards**. Employee showers on site? (Y/N)  
(e.g., 413, 433, 425, etc.)

Pollutants covered by local limits: Scheduled shutdown periods? (Y/N)  
(e.g., Cd, Cr, Cu, Pb, Ni, Zn) When?  
Are local limits technically based? (Y/N)

Number of employees. Seasonal production? (Y/N)

Number of **shifts** per day. Product(s) produced

Hours of operation per day. Amount of finished product

Work **days** per week. Raw materials used.

Manufacturing processes used.

Planned **changes** to the plant

Changes since last inspection Production level.

Use of raw materials

Amount of finished product.

Did the facility report **any** changes identified above to the POTW? (Y/N/NA)

## ***General Instructions for Using the Checklist***

### ***General:***

The checklist is intended to be used by POTW inspectors as a field guide when conducting site visits at industrial users. The checklist is intended to encompass the scope of a routine compliance inspection. If the POTW inspector follows the checklist questions, all **necessary** compliance information should be obtained during the site visit. Some of the information contained in the checklist may not change (e.g., industry name, SIC codes, etc.), but the POTW inspector should continue to gather the information in case of a change which might affect the discharge of pollutants to the POTW (e.g., a new SIC code might indicate a new industrial process which may discharge additional pollutants to the POTW). The checklist may be altered by the POTW to meet its specific needs, but the checklist, either as presented or altered, should form the basis of the inspection report prepared by the **POTW**.

### ***I. and II. General Inspection Information and General Facility Information:***

The inspector should obtain basic identifying information about the **IU** when conducting the site visit, including: industry name; standard industrial classification code number(s) (**NOTE:** there may be more than one SIC code for a given facility); site address and mailing address (the two are often different); as well as any industry contact names, fax and phone numbers, and titles. This information will facilitate routine communication with the industry. The inspector should also check to see if a copy of the IU permit is on file at the IU. If it is not, this may be an indication that the **IU** does not understand or realize its obligations under the local pretreatment program. The inspector should provide a brief description of the facility (i.e., type of operation, how long in business, nature of the products produced, length of time in business, etc.) and probe the nature of the facility's applicable limits (both categorical standards and local limits).

The nature of the business operation should be understood by the inspector. Therefore, **the** inspector should cover: how many employees the facility has (and the trend of employment over time); the number of shifts per day; the hours of operation each day; the number of work days per week; whether there is any seasonal production schedule; the nature of the products and the amount produced and raw materials used in the process; as well as a description of the manufacturing processes used. It is important for the inspector to track any changes in the plant's operation, including any changes in the items listed above. This information provides a history of the manufacturing process and will aid in future inspections at the facility.

The inspector should evaluate the **IU's** efforts at operation and maintenance of its pretreatment facilities and storage areas since this will affect the discharge of pollutants from the facility. Therefore, there are checklist questions pertaining to these issues. Production data is included and is especially important in situations where production based limits are in place for the IU. A final area of general investigation is the compliance status of the facility and a note of any enforcement actions which have been taken for the current noncompliance (past, resolved actions need not be mentioned). The inspection report should track the progress of any IU in meeting imposed compliance deadlines, and the checklist reflects this.

**II. General Facility Information (continued)**

*Date the facility commenced discharge to the POTW:*

*Current long-term average production rate: (if applicable)*

*Is the facility currently in compliance? (Y/N)*

*If not in compliance, what action has been taken ?*

*Are O&M schedules available at the facility?*

*Are there O&M policies and procedures ?*

*Is O&M training/certification adequate?*

**Comments:**

### III. Water Usage and Wastewater Production

#### 1. WATER USAGE

SOURCE      AVG. FLOW (gpd)      METERED (Y/N)?      Comments.

Water Company

Private Well

TOTAL

#### 2. WASTE WATER PRODUCTION

WASTE WATER GENERATING PROCESS	AVG. FLOW (gpd)	BATCH OR CONTINUOUS?	BATCH FREQUENCY	MEASURED/ ESTIMATED	TREATED (Y/N)	REGULATED POLLUTANTS	OUTFALL #
--------------------------------	-----------------	----------------------	-----------------	---------------------	---------------	----------------------	-----------

A.

B.

C.

D.

E.

F. Contact cooling water

SUBTOTALS

G. Boiler blowdown/Make up

H. Evaporation (loss)

N/A

I. Non-contact cooling

J. Lawn maintenance/Irrigation (loss)

N/A

K. Sanitary (loss)

L. In product/Shipped (loss)

N/A

M. Other

TOTAL

Number of **outfalls** to the POTW      Total

Number of **outfalls** to surface waters

Chemicals used in boiler blowdown.

Regulated

All **outfalls** accounted for? (Y/N)

### ***III. Water Usage and Wastewater Production***

To calculate the amount of wastewater discharged to the sewer, the inspector must obtain data on incoming water and water consumption. The inspector should request the following information from the IU regarding the IU's operation: water bills for the most **recnt** 12 months; documents showing **incomping** water from other sources (e.g., well); discharge flow meter **totalizer** charts or readings for the same 12 month period (if flowmeter is present); and production process flow meter **totalizer** charts for the **12** month period (if flowmeter is present). The inspector should understand clearly all water use and wastewater production at the IU, and the checklist is designed to allow the inspector to account for all such water use/loss and wastewater production. The chart should **be** copied and expanded if there are additional sources of wastewater at the facility. In addition, the inspector should check for such illegal activities as: piped or hosed connections which bypass a sampling point; any signs of dilution, such as rinses running during non-processing times which may be inadequately substituting for pretreatment; and any diversions of wastewater flow around the pretreatment system. One of the main reasons for establishing this water balance is to be able to compare water usage and wastewater production from one inspection to the next to determine if additional processes are being employed by the facility.

In addition, the inspector should evaluate the number of **outfalls** from the facility and identify if there are any unregulated **outfalls** in use. Any potential for by-pass needs to be investigated as soon as possible to ensure that the POTW is not receiving pollutants of a kind or an amount which it can not handle. Also, the inspector should be aware of the chemicals which are being used as biocides in the boiler **blowdown** because some of these chemicals may interfere with the operation of the treatment plant or interfere with the **POTW's** final **sludge** use or disposal (e.g.. molybdenum compounds).

**IV. Monitoring, Record Keeping and Reporting**

**I. Monitoring**

<i>Permit Sampling Location</i>	<i>Industry Sampling Location</i>	<i>Flow (gpd)</i>	<i>Permit Limit</i>	<i>Permit Sampling Frequency</i>	<i>Industry Sampling Frequency</i>	<i>Permit Sampling Method (metals)</i>	<i>Industry Sampling Method (metals)</i>	<i>Permit Sampling Method (CN, phenol, O&amp;G, pH)</i>	<i>Industry Sampling Method (CN, phenol, O&amp;G, pH)</i>
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Discrepancies between permit requirements and industry practice for Sampling location? (Y/N)  
 Sampling frequency? (Y/N)  
 Sampling method? (Y/N)

Are the permit requirements appropriate for. Sample location(s)? (Y/N) *If no, explain.*  
 Permit limit(s) ? (Y/N) *If no, explain.*  
 Sample method? (Y/N) *If no, explain.*  
 Sample frequency? (Y/N) *If no, explain.*

What changes, if any, are needed in the permit ?

Samples analyzed according to 40 CFR 136? Are samples preserved according Part 136? Samples analyzed within required holding times ?  
 Samples token during periods of process discharge only? Samples analyzed in-house or contract? Is required unanalytical certification used? <sub>1</sub>

**2. Record Keeping**

All information kept for 3 years? All required information available, current and complete? Are all sample results included in the IU's report? <sub>1</sub>

**3. Reporting**

Did the facility report results of any more frequent sampling in the last reporting period? If so, were all results reported?

POTW notified of all violations w/i 24 hours?

Do sample results match what is reported by the industry? Are there any violations which were not reported to the POTW?

#### ***IV. Monitoring, Record Keeping and Reporting***

It is crucial for the inspector to evaluate the **IU's** monitoring, record keeping and reporting practices because this information forms the basis of the **POTW's** regulation of the industry. If the IU is not monitoring correctly or if the samples are not analyzed using the required procedures, the information derived from that monitoring can not be used to establish compliance. Likewise, if the facility does not keep records, the POTW can not know the conditions at the facility during the reporting period. It is necessary for the inspector to compare the results of sampling to the actual reported values to ensure that there are no discrepancies. If a discrepancy is found, the cause should be determined at once.

In the first section the inspector should compare the industry's practice with the permit requirements for the following: sampling location (which must be specified in the permit, e.g., location 001 with a schematic indicated this location), sampling frequency (e.g., monthly, twice per year, etc.), and sampling method (i.e., grab or composite) for metals and **organics**. Each of these items should be specified in the permit, and the facility must abide by the requirements established in the permit for these items.

In addition, the inspector must determine if the current permit conditions for the facility are adequate to control the discharge to the sewer. The checklist questions are designed to give the inspector a comprehensive overview of the **IU's** monitoring, record keeping and reporting procedures. If there are any problems, the source of the problem should be determined as soon as possible.

## V. Wastewater Treatment Systems

Does the industry **treat** its process wastes prior to discharge to the POTW?

If treatment is in place, complete the following information. (If no treatment, go to the next section)

Are any treatment units out of service? (Y/N)

Inadequate system in place to correct a problem? (Y/N)

Unauthorized discharge points in service? (Y/N)

Unauthorized bypasses in place? (Y/N)

Treatment type.

Date originally installed.

Modified since installation? Describe.

Design flow (gpd).

Treatment (batch or continuous)?

Actual flow (gpd)

Discharge (batch or continuous)?

### Operating Schedule

Reagents used. (include usage rates if known)

Hours per day.

Days per week

FTEs needed to operate.

Clarifier volume.

Effluent filtration media (if applicable)

Description of overall condition.

Has the system experienced operational/upset problems since the last inspection? If yes, describe

## VI. Sludge Generation/Waste Disposal

If the facility generates sludge or hauls regulated wastes, please complete the following information. (If not, go to next section)

Sludge dewatering method.

Moisture content.

Amount generated (55 gal  
bbl/mo).

Disposal method.

Sludge Storage (bbls)

Shipment frequency.

Manifests available?

Sludge hauler(s)

Disposal location(s) :

Hazardous Sludge Generated? (Y/N/NA)

Hazardous Waste Discharged to the POTW? (Y/N/NA)

Manner of Hazardous Waste Disposal.

Are hazardous waste manifests available?

If not, verify manner of hazardous waste disposal.

## **V. Wastewater *Treatment Systems***

The wastewater treatment system at the IU must be operated and maintained in a manner which allows the system to prevent the discharge of pollutants in excess of the **IU's** permit limits. The treatment system is at the heart of the **IU's** ability to control its discharge of pollutants. Therefore, it is necessary for the inspector to spend some time to evaluate the treatment system's condition and use/operation. The inspector should check for the following items: equipment maintenance record keeping or lack of preventative maintenance; instrument calibration frequency; critical spare parts inventory; inadequate detention time or inadequate mixing in the pretreatment tanks; improper chemical dosage; improper meter settings; stale chemical use; and current operational status. (NOTE: not all of these items are contained in the checklist, but should be noted in the comment section at the end of the checklist).

## **Vi. *Sludge Generation/Waste Disposal***

How the IU handles its solid waste is an indication of its commitment to the **proper** handling of all its wastes, liquid or otherwise. The inspector should examine the **IU's** sludge disposal methods to ensure that no sludge from the treatment system is being discharged to **the** sewer (except in accordance with a permit). If the facility produces hazardous wastes (e.g., electroplating sludge), the inspector should verify where the waste is being ultimately disposed. Make sure that each applicable box is filled in (**Y/N/NA** stands for Yes, No, and Not Applicable).

## **VII. Combined Wastestream Formula/Permit Limits**

*Can flow be measured at all sampling locations?*

*Are flows measured **at** each sampling location?*

*What **type** of measuring device is used?*

*Are dilution wastestreams present at the sample location?*

*Is the C'WF used at the **facility**?*

*How **are** the flows determined?*

*Is the **facility** using dilution to meet its **effluent** limits?*

*Should the **facility** be using the combined wastestream formula?*

*Are there **any** new flows which need to be considered in the application of the **combined** wastestream formula?*

*Are there **any** dilution flows which have not been accounted for?*

## **VIII. Chemical Storage**

*What **chemicals** are used at the **facility**?*

*Can chemicals reach **floor** drains if spilled? :*

*Is chemical containment needed?*

*How often are **floors** washed? What chemicals are used?*

*How often is **equipment** washed? What chemicals are used?*

*Does the **facility** have a slug control program?*

*Has **the** facility had any past slug discharges?*

*Amount of water used in washdowns (gals).*

## **IX. Production/Process Areas .of the Industrial User**

*Are wastestreams separated at the **facility**? (Y/N)*

*Are incompatible materials separated? (Y/N)*

*Do floor drains/troughs lead to the POTW? (Y/N)*

*Are temporary hoses in place **as part** of production ?*

*Are pipes labelled/color coded for easy identification?*

*Is a piping diagram **available** at the **facility**?*

*Attach a schematic of production, wafer flow, wastewater production, and a stepwise description of the production process at the facility.*

*Attach a stepwise description of the chemicals used and/or discharged during production.*

**Overall Inspection Comments**

## ***VII. Combined Wastestream Formula/Permit Limits***

If the CWF is being used to calculate alternative discharge limits, the **flow** of each waste stream must be known and measurable. The inspector should ensure that flow can be measured at all necessary points and that flows are being measured correctly at all locations. The inspector will need to evaluate any dilute streams being discharged to the sewer and whether these streams are being used to meet any permit limits. Dilution streams for purposes of the CWF include: sanitary wastewater, boiler blowdown, non-contact cooling water or blowdown, **deionizer** backwash, cooling tower bleedoff, condensate, and **rainwater/stormwater**. If it appears that dilution or unregulated streams are being co-mingled with regulated streams prior to treatment, then the inspector should initiate the procedure to have the permit changed and new limits applied (as well as initiating any applicable enforcement action as dictated by the **POTW's** Enforcement Response Plan). In addition to dilution streams, the inspector should check for any unregulated streams at the facility. Unregulated wastestreams for purposes of the CWF include: any wastestream which is not currently regulated by a categorical pretreatment standard and does not meet the definition of a dilute stream. Determining such unregulated wastestreams requires a familiarity with the categorical industry in question, and probably will require some research into the Development Document issued by the EPA. Refer to the EPA Guidance Manual on the use of the combined wastestream formula which is listed in Appendix XII.

## ***VIII. and IX. Chemical Storage and Production/Process Areas of the Industrial User***

It is important for the inspector to trace the use of all process (and non-process) chemicals which may be discharged to the sewer. Areas of spill containment, floor drains, and the manufacturing process should be examined to determine which chemicals are (or can) find their way into the sewer. The inspector should verify that incompatible chemicals (e.g., strong acids and bases, or chemicals which may interact to form toxic compounds) are not stored near each other in the event of a spill. It is best for the inspector to follow the chronological sequence of the production process in the step wise sequence of production to comprehend the activities at the plant. (NOTE: Do not allow the IU contact to "control" the inspection or the sequence of the inspection. How the inspection is conducted is up to the inspector alone). Once the inspector understands the operation at the facility, a schematic of the production/manufacturing process, water use and wastewater production, and a **stepwise** description of all chemicals used or discharged during production should be developed and compared on subsequent inspection visits.

The inspector should also check the production area for the following conditions: excessive drag-out and/or spillage from plating lines; excessive water on the floor and its entry points to the sewer system; and labelling of all tanks used in the production process.

***General Inspection Comments (cont.)***

### III. SAMPLING INDUSTRIAL USERS

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#### INTRODUCTION

An effective local pretreatment program must include the ability to collect and analyze wastewater samples such that laboratory results are of high quality, defensible and able to support the two primary goals of the Pretreatment Program. The first goal is to determine the Impact of Industrial wastes from a particular industry or group of industries on the Publicly Owned Treatment Work's (POTW) collection and treatment system, including the impact on treatment plant operations, sludge management (including final use or disposal), and receiving stream quality. The second goal is to evaluate compliance by all industrial users with applicable pretreatment standards and requirements. In addition to these primary objectives, the POTW's sampling and analysis program is designed to satisfy one or more of the following program objectives:

- Verify the quality of self-monitoring data;
- Verify that sampling location(s) specified in the permit are adequate;
- 1 Verify compliance with daily maximum effluent limits (local limits or categorical standards);

- Support enforcement actions;
- Support local limits development; and
- Support permit development/reissuance and revision

These objectives can only be supported if the data produced by sampling are adequate. The quality of the data resulting from the POTW's sampling activities can be ensured by using the following procedures and techniques: collecting representative samples; maintaining the integrity of samples through proper handling and preservation; adhering to appropriate chain-of-custody and sample identification procedures; and practicing adequate quality assurance and quality control activities. This chapter outlines each of these areas in detail.

Sampling and analysis of wastewater can be done independently or in conjunction with a compliance inspection visit, and can be performed by the same or different POTW personnel. If sampling and analysis are performed independently and by POTW personnel different from the inspection personnel, it is strongly recommended that the sampling personnel familiarize themselves with the procedures and guidelines used by the inspection personnel. Chapter Two of this manual provides a recommended framework for conducting inspections at regulated IUs. Sampling, just like inspections, can be announced or unannounced. The goal of your sampling visit will determine whether the industry is notified. As with inspections, routine compliance sampling should be conducted based on a "neutral" scheme (see discussion in Chapter 2). These routine compliance sampling visits should be unannounced. Sampling visits which are in response to known or suspected problems or in response to a complaint should also be unannounced so that the facility does not have time to alter any of its activities.

In the pretreatment program, the vast majority of sampling will be routine compliance evaluation sampling mandated in 40 CFR 403.8. In these situations, it is necessary for the POTW to ensure that the sampling data collected will be of a quality sufficient for the POTW to draw a proper conclusion about the compliance status of the facility and to ensure that the data will be viewed as credible evidence substantiating the POTW's position should an enforcement action be pursued. This is the fundamental objective of any sampling carried out for compliance and enforcement purposes, and even, perhaps, when developing local limits, since the basis for these limits must be justifiable and defensible. But there may be situations where other types of sampling may be used by the POTW. This sampling can be conducted for a variety of reasons (e.g., operation or maintenance evaluations). These sample objectives would not involve as comprehensive a set of procedures as a compliance sampling visit, which results in evidence that may be used in court. If a sample is to be used for other than compliance evaluation purposes, it need not comply with the strict requirements of compliance sampling (i.e., to obtain results which are admissible in court). However, if a

sample is obtained using approved sampling and analytical procedures, then the results of the sampling must be reported to the POTW

**This** chapter presents a detailed and comprehensive framework to be used by POTW sampling personnel when conducting sampling and flow measurements at regulated industrial users. The purpose of the chapter is to familiarize POTW inspectors with proper sampling procedures and to establish consistent procedures for all **POTWs** with approved local programs. The goal of this chapter is to assist POTW personnel in planning and conducting sampling activities at industrial facilities which discharge to the POTW. The chapter is divided into six sections to accomplish these objectives: analytical methods, quality assurance and sampling plan, standard operating procedures, pre-sampling activities, on-site sampling activities, safety considerations during sampling, flow measurement, and quality assurance and quality control. In addition to these sections, this chapter presents a concluding section on Compliance Issues Related to Sampling and Analysis. This section discusses specific compliance issues related to the **POTW's** and **IU's** compliance monitoring program and provides recommendations for handling certain compliance information. POTW sampling personnel are encouraged to read and understand the material presented in this chapter before beginning any sampling activities.

### *ANALYTICAL METHODS*

The National Pretreatment Program requires that samples be analyzed using the approved methods listed in 40 CFR 136 (40 CFR **403.12(g)(4)**). The methods in 40 CFR **136** are derived from five different sources: (1) Methods for the Chemical Analysis of Water and Wastes, **EPA-600/4-79-020**, revised March, 1983; (2) Standard Methods for the Examination of Water and Wastewater, (specified edition); (3) **ASTM**; (4) Methods for Analysis of Inorganic Substances in Water and **Fluvial** Sediments - USGS; and (5) additional sources. Not all methods listed in each of these documents are approved in 40 CFR **136**. Therefore, the POTW (and IU) must use the methods listed in Part **136** or apply for an alternative method. **POTWs** and **IUs** can apply for such an alternate test procedure if they believe that a method not listed in 40 CFR 136 is a better method of analysis and can prove that the proposed method of analysis is comparable to the method listed in 40 CFR **136**. This alternate method must be approved by the EPA if the results are to be used to comply with 40 CFR **403.12(g)(4)**. The POTW is not allowed to grant this variance in analytical methods. The application and approval procedures for instituting an alternative analytical method are outlined in 40 CFR 136.4 and **136.5**

Choosing the appropriate **analytical** method for the samples collected is an important task. If more than one method is listed for a parameter (which is common), the method which is chosen should be based on the

**effluent** limit in the permit, Sewer Use Ordinance, **and/or** Federal Categorical Standard. An example of how to choose the correct analytical method can be illustrated using the pollutant parameter lead. Lead can be analyzed using EPA methods 200.7, 239.1, and 239.2. For our example, let's assume that the established effluent limit for lead is 15 **ug/l**. The detection limit for lead using EPA method 200.7 is 42 **ug/l**. The detection limit for lead using EPA method 239.1 is 100 **ug/l**. The detection limit for lead using EPA method 239.2 is 1 **ug/l**. Methods 200.7 and 239.1 should not be used because the detection limit for the method is higher than the permit limit. Therefore, any results reported, even a non-detect, could be viewed as an excursion from the permit limit. The appropriate method of analysis in this example is EPA method 239.2, and this method should be specified to the analytical laboratory. (NOTE: the actual detection limits for these methods will vary with different matrices). In addition, laboratories must establish their own method detection limits using Appendix B to 40 CFR 136 -- Revision 1.11. In addition, the impact of sample dilution and elevated detection limits should be evaluated by the POTW. If a sample must be diluted to get one or more analytes on-scale within the linear range of the calibration curve, the detection limit for any **non-**detected parameter must be elevated accordingly. For example, if a detection limit for analyte A was 10 **ug/l** and a sample was diluted 1:4 to get another analyte on-scale, then the detection limit for analyte A in that sample must be elevated to 40 **ug/l**. The POTW must be aware of this situation when evaluating the appropriate detection limits and analytical methods for sample analysis.

#### **QUALITY ASSURANCE AND SAMPLING PLAN**

A fundamental step in setting the objective(s) of a sample collection effort is to establish clearly the ultimate use of the data. Before sampling at an industrial user, the inspector should understand clearly the data needs (i.e., for what purpose will the data be used, e.g., compliance determination) and the data quality objectives of the site visit (e.g., a compliance **determination** will require data which are admissible in court). When the data resulting from the sampling become available, it is crucial that it be possible to assess the quality and utility of the data in meeting the sampling objective. Once the inspector understands the needs and objectives of the site visit, a complete and comprehensive quality assurance and sampling plan can be developed. The plan should be documented in written form and completed prior to initiating any sampling activities. This plan ensures that each sampling effort goes through a careful thought process before it is undertaken. The U.S. EPA Quality Assurance Management Staff (QAMS) has developed documents which will be useful to the POTW when developing their QA and Sampling Plans. It is recommended that POTWs contact their U.S. EPA Regional QAMS or QA Managers for copies of model QA and Sampling Plans to use as guidance in preparing their own plans. At a minimum, the QA and Sampling Plan should include the following elements:

- **Sampling Location(s):** Sampling locations should include all **outfalls** that appear in the IU's permit. Due to accessibility, needs, and objectives of the sampling, **and/or** safety hazards, the sampling location specified in the permit may not be adequate. Therefore, locations other than those specified

int he IU's permit may need to be sampled. The number of samples at each location should also be specified in the IU's permit. In addition, the inspector should sample any **outfalls** which are not included in the IU's permit because they may represent illegal bypasses or other illegal discharges.

- **Type of Sample:** The type of sample depends on the parameters to be measured **and/or** the discharge characteristics (e.g., batch discharge). This information may **be** specified in 40 CFR 136 and should be specified in the IU's permit.
- **Type of Flow Measurement:** The type of flow measurement is dependent on the flow rate, the condition of the wastewater, and the variability of the discharge. Flow measurements are necessary to determine the mass loadings of a discharge. The adequacy of the permittee's flow measuring device should be verified at the time of sampling.
- **Parameters for Analysis:** The IU permit should specify the pollutant parameters to be monitored by the permittee, and these parameters should be specified as either mass- or concentration-based discharge limits. These parameters must be selected for compliance sampling, but other parameters may be chosen as well, if new processes or products have been incorporated in the plant or if new or added sources of wastewater are evident. If new processes or products have been introduced in the plant, additional sampling will help provide the basis for necessary permit modifications. (**NOTE:** The IU is required to notify the POTW in advance of any substantial change in the discharge **from** the facility. Failure to do this, as discovered during the inspection or sampling visit, should be addressed with an appropriate enforcement response as specified in the **POTW's** Enforcement Response Plan).
- **Sample Volume:** The volume of sample collected depends on the type and number of analyses to be conducted. The volume of the sample obtained should be sufficient to perform all the required analyses (including laboratory **QA/QC** and repeat analyses) plus an additional amount to provide for any split samples that may be required. A summary of required sample volumes for various **pollutants** is provided in Table 3-3 at the end of this chapter, but it is still best to check with the individual laboratory to determine the sample volume which it requires.
- **Type of Sample Containers:** The selection and preparation of sample containers are based on the parameters to be measured and wastewater characteristics. Required containers are **specified** in 40 CFR 136 and are summarized in Table 3-4.
- **Sample Preservation Techniques:** To preserve samples correctly, the appropriate chemicals must be used and temperature control must be ensured. Preservation techniques and maximum allowable holding times are specified in 40 CFR 136 and are summarized in Table 3-4 at the end of this chapter.
- **Sample Identification Procedures:** Each container should have an acceptable identification label so that the sample can be tracked accurately and an uninterrupted chain-of-custody can be maintained.
- **Sample Packaging and Shipping:** Once a sample is collected, it must be delivered to **the** laboratory for analysis within the prescribed holding time. **The** manner of packaging and shipment must be addressed through the sampling plan.
- **Safety Concerns:** Sampling personnel should have complete information on any relevant plant safety regulations and safety procedures to be followed during on-site sampling activities.
- **Hazardous Waste:** Samples of potentially hazardous waste; samples with extremely high or low **pH**; and samples that may contain toxic, volatile, or explosive substances will require special handling. DOT regulations for shipping these types of wastes must be followed.
- **Chain-of-Custody Procedures:** Procedures for chain-of-custody must be followed for all samples collected by the POTW, and standard chain-of-custody forms should be used for this purpose (see Appendix X for an example Chain-of-Custody form).
- **QA/QC Procedures:** To ensure that the data collected are valid, systematic checks must be conducted to verify that the sample results are sufficiently accurate and precise to evaluate the compliance status of the facility being sampled.

Several of these items must be coordinated with the lab. Therefore, the inspector should contact the lab in advance of any sampling to discuss the sampling plan and QA/QC procedures, to allocate lab time, and obtain sample identification numbers and field trip blanks.

Once the sampling plan is developed, it should be followed when conducting sampling at the industrial users regulated by the POTW's approved pretreatment program. The procedures in the plan should be followed closely to ensure that all the information collected can be used for its intended purpose. This is especially critical when the sampling data is to be used for compliance **evaluation** (which accounts for the majority of the data collected by the POTW). However, in certain situations, the inspector may be forced to alter some items in the plan due to uncontrollable circumstances at the industrial user. **The** inspector has discretion to change some items in the QA Plan if, in the opinion of the inspector, circumstances at the facility warrant such a change. Whenever possible, however, the elements in the QA Plan should be followed.

### *STANDARD OPERATING PROCEDURES (SOPs)*

Once the sampling plan has been established, the POTW should develop **specific** standard operating procedures (SOPs) for on-site sampling activities. SOPs can be a document or set of documents which explain, in step-by-step detail, how sampling will be conducted by **the** POTW. The SOP developed by the **POTW** should include all elements of sampling, including:

- **Sample Documentation:** Documentation is an integral part of any pretreatment program. The validity of the samples collected and the data obtained both in the field (e.g., **pH** and flow) and in the laboratory (i.e., chemical analyses) is ensured through documentation and record keeping. All the information documented must be complete and accurate. Failure to maintain records and documentation according to set procedures could result in these documents being deemed inadmissible **as** evidence in court. The POTW should include the following records in their SOP:

*Field Data Record* - The Field Data Record is the primary sampling information document and should include: the sample site identification; the type of sample taken; sampler identification; settings on the sampler; results of field analyses; flow information (where applicable), and any additional information related to the site or effluent characteristics.

*Field Documentation Log* - **The** Field Documentation Log is used to record which sites are sampled each day, and any violations, conversations or other notable occurrences during the sampling visit.

*Field pH Calibration Log* - The Field **pH** Calibration Log is used to record the calibration of the field **pH** meter during the sampling event. The **field pH** meter must be calibrated at each site prior to measuring the **pH** of the effluent. Calibration and slope should **be** checked, adjusted as necessary, and recorded, along with the temperature of the buffer.

*Flow Meter Calibration Log* - The Flow Meter Calibration Log is used to record program information for the flow meter and water level calibration from the initial value shown on the flow meter to the actual measured water level.

<sup>1</sup> The discussion of Standard Operating Procedure elements was taken from **the "Standard Operating Procedures Manual: Pinellas County Sewer System Industrial Monitoring Program,"** January, 1989.

**pH Meter Calibration (Laboratory)** - The laboratory should maintain its own notebooks to record equipment calibration. When the laboratory pH meter is used, it must be calibrated. The discussion of how to calibrate the meter should be included in the SOP under the calibration procedures section. Once the meter is calibrated, the results should be recorded in the (laboratory) pH Meter Calibration Log Book.

- **pH Calibration/Spike Checklist** - The pH Calibration/Spike Checklist is used to record the date and time of field pH meter calibration, calibration data, results and true value for the known sample, and to document the buffer and fill solution changes.

**Chain-of-Custody Form** - The Chain-of-Custody Form includes sample collection information (i.e., who collected the sample, when the sample was collected, what type of sample was collected, and who received the sample after the initial sample was taken), types of analyses to be run by the lab, preservation technique used, and a space for the lab personnel to sign with the date and time the sample was received by the lab.

- **Chain-of-Custody:** The overall success of a sampling program (whether by the POTW or the industry) depends on its ability to produce valid data through the use of accepted sampling procedures and protocols, and its ability to substantiate such data through documentation. The success of documenting samples depends on the faithful use of chain-of-custody forms by all involved personnel. The SOP should include the chain-of-custody form.
- **Safety:** The SOP should outline the many safety precautions which must be followed both at the office and in the field. Industrial monitoring, by its very nature, adds additional hazardous situations to those existing in any field sampling situation. All safety procedures should be outlined in the SOP.
- **Cleaning:** Sampling equipment, grab and composite collection containers, sample bottles, and tubing should be cleaned at a specified frequency outlined in the SOP. All cleaning procedures should also be included in the SOP.
- **Maintenance:** Maintenance activities ensure the constant reliability of sampling equipment, including flow meters. The SOP should outline a maintenance schedule for all equipment related to sampling.
- **Calibration:** Calibration of field and lab equipment is crucial to the continued reliability of the sample results obtained from sampling. A regular schedule of calibration should be included in the SOP and should be adhered to strictly. This calibration should include flow meters, pH meters, and any other equipment requiring calibration, as recommended in the manufacturer's specifications.
- **Sampling Preparation:** Sampling preparation is the most important part of a successful sampling event. Standard sampling checklists should be included in the SOP and should outline all sampling preparation procedures.
- **Types of Samples/Sample Methodology:** There are two basic types of samples: grab and composite. The IU permit should establish the sample type, and this sample type should be used by the POTW when collecting samples.
- **Field Analyses:** Certain measurements are typically performed in the field (e.g., flow, pH, and temperature). Detailed procedures for conducting these analyses should be included in the POTW's SOP.

Each of these areas is discussed in greater detail later in this chapter, and a copy of the Pinellas County SOP is included as Appendix IX. Each POTW should review its SOPs to ensure that all necessary areas are covered in adequate detail using Appendix IX as a guide.

Once the SOPs are written, they should be followed closely. Any deviation from a SOP may create potential problems or weaknesses in a subsequent enforcement action taken by the POTW against a

noncompliant industrial user. If the POTW establishes clearly identified SOPs and follows those procedures when conducting on-site sampling activities, it is unlikely that an industry could challenge the results of the POTW's sampling results. If circumstances arise in the field which make it unrealistic or physically impossible for the SOPs to be followed, the inspector should document any deviation from the written SOPs and the reason for the deviation.

### *PRE-SAMPLING ACTIVITIES:*

The success of each sampling task hinges on adequate preparation. Because POTW personnel may not be familiar with the facility to be sampled, a sampling plan should be developed prior to going out into the field. Once the POTW sampling plan is in place for a particular industrial user, inspection personnel should follow the plan when conducting on-site sampling. Inspection personnel should be briefed, as well, on all field procedures, particularly safety requirements. The inspector should make sure that the appropriate sampling equipment is available and in good working order. When sample analyses are to be performed in the field (e.g., pH), the necessary instruments should also be included. Equipment must be checked prior to going into the field to ensure accurate operation and calibration. In addition, a review of necessary safety equipment should be made and the inspector should be aware of any potential hazards at the facility. The inspector and plant staff should discuss any unusual circumstances and formulate a plan for dealing with them in advance of the site visit.

A checklist of field sampling equipment should be used to ensure proper preparation. An example of such a list is outlined in Table 3-1. When the type of waste to be sampled is known ahead of time, the list can be narrowed to the actual pieces necessary for the specific sampling required.

### *Cleaning and Preparation Of Sampling Equipment*

The cleaning and preparation methods for sampling equipment will vary depending on the parameters being sampled. This section addresses the cleaning and preparation of sample collection vessels and/or sample bottles for conventional pollutants (TSS, BOD, fecal coliform, oil and grease and pH), metals, and organic pollutants. Many vendors now sell precleaned sample bottles with varied cleaning specifications. It is recommended that a bottle blank be run when analyzing the samples to verify that the bottles are not a source of sample contamination. One bottle blank is recommended per LOT number.

#### **Cleaning Procedures for Conventional Pollutant Parameters (BOD and TSS only):**

When sampling for the conventional pollutants BOD and TSS, it is necessary to clean the sample bottles and/or collection vessels prior to each sampling visit. The recommended cleaning procedure involves: (1) a detergent wash, (2) tap water rinses, and (3) deionized water rinses. This procedure forms the basis for almost all further cleaning procedures required for other pollutant parameters when preparing sampling bottles

**Table 3-1**  
**Checklist of Field Sampling Equipment**

**Sampling Equipment:**

- Siphoning equipment
- Weighted bottle sampler
- Liquid waste samplers
- Auger, trowel, or core sampler
- Scoop sampler
- Sample bottles/containers (certified clean bottles)
- Ice chest
- Flow meter (if applicable)
- Preservatives
- Chain-of-custody forms
- Custody seals and tags
- Strapping tape
- Field test kits (pH, etc.)
- Automatic or composite sampler
- Container for contaminated material
- Waterproof container labels
- Ambient air monitor
- Field document records
- Vermiculite or equivalent packing
- Thermometer
- Colorimetric gas detection tubes
- pH equipment
- Explosimeter (atmospheric testing device)
- Tubing, tape and rope
- Field sampling logs
- Sample shipping forms (w lab phone #s)
- Graduated cylinder
- Preservatives (e.g., nitric acid and NaOH)

or vessels.

**Cleaning Procedures for Metals Sampling:**

When preparing for metals analysis, it is necessary to preclean the sample bottles and or vessels (either purchased precleaned or cleaned manually). The cleaning procedures for preparing sample bottles for metal analyses is provided in 40 CFR 136, Appendix C, Section 8 (ICI' method 200.7) [NOTE: This method is not applicable for atomic absorption analysis], along with the documentation on the analytical method in Methods for Chemical Analysis of Water and Wastes, 1983. The cleaning process for metals sampling and analysis is outlined in Figure 3-1.

Chromic acid may be used to remove organic deposits on glassware. If chromic acid is used, extreme care must be taken to ensure that the glassware is thoroughly rinsed to remove all traces of the chromium. It is recommended that chromic acid not be used for cleaning sampling bottles if chromium is one of the parameters being analyzed.

Figure 3-1 Metals Cleaning Procedures

Use the following procedures when cleaning sampling bottles and/or vessels for metals sampling and analysis:

- 1) Detergent wash
- 2) Tap water rinses
- 3) (1:1) Nitric acid rinse
- 4) Tap water rinses
- 5) (1:1) Hydrochloric acid rinse
- 6) Tap water rinses
- 7) Deionized distilled water rinses.

**Cleanine Procedures for Oil and Crease:**

The approved method of analysis for Oil and Grease is gravimetric extraction (Standard Methods for the Examination of Water and Wastewater, 17th edition, 1989. method 155208 or Methods for Chemical Analysis of Water and Wastes, 1983. method #413.1). When sampling for oil and grease, it is necessary to use a wide mouthed glass jar, which has been rinsed with the solvent used in the extraction process. Currently, freon is used as the solvent in this process. A substitute for freon is currently being researched and may result in an alternative analytical method. The cleaning method for oil and grease is the following: (1) detergent wash; (2) tap water rinses; and (3) solvent rinse. It is recommended that the sample bottles have a teflon-lined cap. If this is not possible, there should be either aluminum foil or cut teflon pieces which cover the areas where the bottle and the cap meet. The teflon or aluminum foil used must also be prepared, following the same cleaning procedures as the sample bottle.

**Cleanine Procedures for Organic Analysis:****Volatile Organic Compounds**

Generally, the glassware used to collect volatile organic samples is precleaned. When glassware needs to be cleaned, the procedure in 40 CFR 136 may be followed. EPA method 624 states that the vials and the septa must be cleaned as follows: (1) detergent wash; (2) tap water rinses; (3) distilled water rinses; and (4) drying at 105°C.

**Semi-volatile Organic Compounds, Organochlorine Pesticides, and PCBs**

Semi-volatile organic samples should be collected in amber bottles according to 40 CFR 136, Methods 625 and 1625. Organochlorine pesticides and PCB samples should also be collected in amber bottles according to 40 CFR 136. Method 608. If amber bottles are not available, the samples must be **sheltered** from the light. The sample bottles (including cap liner, either teflon or foil) and the collection vessels for semi-volatile organic compounds, organochlorine pesticides, and **PCBs** must be cleaned by the following procedure: (1) detergent wash; (2) tap water rinses; (3) distilled water rinses; (4) solvent rinse (method 625 lists acetone or **methylene** chloride); and (5) drying. If one or more of the **samples** is being collected in a sampling vessel, the sampling vessel must be cleaned for all parameters. Examples of this include:

- Sampling for conventional pollutant analysis and metals analysis: the collection vessel must be detergent washed and then acid washed (following the steps for metals cleaning listed above).
- Sampling for metals analysis and semi-volatile organic analysis: the collection vessel must be acid washed (following the steps above) and then solvent rinsed. The initial steps in the semi-volatile organic cleaning do not need to be repeated in this case (only the solvent rinsing), since they were already done in the metals cleaning procedure.

**Cleaning of Automatic Sampling Equipment:**

Generally, the sampler tubing and inner parts of the sampler (e.g., distribution arm, S-tube, and compressed silicon tubing) which come into contact with the effluent are cleaned using the following procedure: (1) detergent wash, (2) tap water rinses, and (3) distilled water rinses. If the sampler is to be used to collect semi-volatile organic and/or organochlorine pesticide and PCB samples, additional cleaning is required. 40 CFR 136, Methods 625 and 608, respectively, states that the automatic sampler must be as free as possible of contaminants in the Tygon tubing and any other potential source of contamination. One suggestion for the replacement of Tygon sampler tubing is the use of teflon tubing. These methods also state **that** if the sampler has a peristaltic pump, a minimum length of compressible silicon rubber tubing may be used. Before it is used, the compressible silicon tubing must be cleaned using the following procedure: (1) methanol rinse, and (2) distilled water rinses. If the sampler used has an S-tube, it must be cleaned **by** following the same procedure as for the sample bottles for semi-volatile organic compounds, organochlorine pesticides, and **PCBs**. As an alternative, some **POTWs** may prefer to replace tubing prior to each use of the automatic sampler.

***Preparing Field Instruments***

The most common parameters tested in the field are: **pH**, residual chlorine, temperature, and dissolved oxygen. For these four parameters, 40 CFR 136 states that they must be analyzed immediately. **The** term “analyze immediately” means that the parameter should be analyzed **within 15** minutes of the sampling. These analytical parameters cannot be preserved, and therefore, must be analyzed in the field. **The electronic and** photometric instruments used to monitor these different parameters should be checked prior to leaving the **office**. The instruments should be in good condition, have charged batteries, be calibrated, and have all appropriate standards already made. **If an** instrument is calibrated in the **office** prior to going into the field, it must be recalibrated once you reach your sampling location.

**pH Meters:**

**In** the field, **pH** samples are analyzed using a portable **pH** meter. **The** meter may either analyze individual samples or do continuous readings with a recorder (e.g., strip chart). **pH** meters must have a minimum of two point calibration (see EPA method 150.1 section 7). **(NOTE :** If there are separate manufacturer’s specification for calibration, these procedures must be followed. **Otherwise,** use the procedures outlined in the remainder of the paragraph). **The pH** meter should be calibrated using two fresh buffer solutions. The buffers that are used to perform the calibration should bracket the expected **pH** range of the wastewater that is being sampled and should be at least 3 SU or more apart. If the buffer solutions are bought already made, it is important to note their shelf life and dispose of buffers **when** their expiration date has passed. A log book with calibration information for the **pH** meters should be maintained. This allows

the inspector to track when a pH probe needs to be changed or when batteries start to fail. Remember, the meter must be recalibrated once the sample location is reached.

**Residual Chlorine Meters:**

The manufacturer's specifications should be followed for calibration. Portable spectrophotometric, DPD meters are an approved method of analysis listed in 40 CFR 136. Other instruments, such as portable amperometric titrators, can also be taken into the field to detect low levels of chlorine (100 µg/l). The permits limit, local limit, or Sewer Use Ordinance limit will determine the appropriate method of analysis.

**Temperature:**

A mercury-filled thermometer, a dial type Celsius thermometer, or a thermistor must be used to make a temperature determination. The measuring device used must be routinely checked against a National Institute of Standards (NIST) traceable thermometer. This check should be recorded in a calibration log book with the date, both temperature readings (reference and actual), and any correction which was made to the temperature measuring device. The calibration log book could be an important document in an enforcement case, if temperature violations were noted during the inspection.

**Dissolved Oxygen:**

Dissolved oxygen measurements can be taken either using the Winkler or the electrode method. If the Winkler method is used, the samples must be fixed (preserved) on-site, stored in the dark, and analyzed within eight hours. JO CFR 136 requires that if the electrode method is used, the sample must be analyzed immediately (see Table 3-4 at the end of the chapter for a summary of this information). Prior to each sampling, the D.O. meter should be calibrated. The manufacturer's specifications for calibration should be followed. Generally, the D.O. meter is calibrated to a solution of known D.O. concentration (usually saturated) or to moist air. A calibration record should be maintained (including date, D.O. readings, any adjustments, date a new probe is added, etc.). If a membrane electrode is used, great care should be used when the membrane is being changed to avoid trapping air bubbles under the membrane. Trapped bubbles will result in inaccurate D.O. readings.

***Selection and Preparation of Sample Containers***

The selection and preparation of sample containers must be made prior to going out into the field. Sample containers must be made of chemically resistant material that does not affect the concentration of pollutants to be measured. The containers used should be either glass or plastic. For most analyses, the option of using either glass or plastic sample containers is open, and the selection of the sample container is based on the organization's operating procedures. It is important that the inspector become familiar with these procedures (i.e., SOPs). If either type of sample container is acceptable and available, the inspector should use plastic containers since they are less likely to break. POTWs should develop specific acceptance

criteria (i.e., **chemical\parameter** concentration) for each type of sample container to be used. An example of such criteria (as well as sample container preparation procedures) may be found in the U.S. EPA document "Specifications and Guidance for Contaminant-Free Sample Containers," EPA 540/R-93/05 1, December, 1992. Although this document was prepared for solid waste applications, it gives an idea of the types of requirements POTWs should have for sample containers. Sample containers, preservatives, and holding times are specified in Table 3-4 and in 40 CFR 136.

Glass sample bottles are required when collecting samples for organic priority pollutants, oil and grease, and phenols. while plastic sample bottles are most **often** used for Biochemical Oxygen Demand (BOD), Total Suspended Solids (TSS), metals, and nutrients. Containers with wide mouths are recommended to facilitate the transfer of samples from the sampler to sample containers (for automatic samplers). In addition, the container must be large enough to contain the required volume for laboratory analysis. The inspector should use dark containers for samples that contain constituents which will oxidize from exposure to sunlight (e.g., iron cyanide which oxidizes to hydrogen cyanide).

Container lids and closure linings must also be intact so they do not interfere with the pollutant parameters to be measured. Most containers have tight, screw-type lids. Plastic containers are usually provided with screw caps made of the same material as the container, so cap liners are usually not required. Glass containers usually come with rigid plastic screw caps. Liner materials may be polyethylene, polypropylene, neoprene, or teflon.

The inspector should make sure that all sample containers are clean and uncontaminated. The general cleaning procedure for a sample container was outlined previously and should be followed whenever samples are taken. All tubing and other sampling system parts must be scrubbed with hot water and detergent, rinsed several times with tap water, and then rinsed with distilled or deionized water. Further rinsing with acetone is advised only when the type of tubing (e.g., teflon) is not susceptible to dissolution by the solvent. In most cases, the container should be rinsed three times with the wastewater to be sampled before the sample is taken (**NOTE:** Except when sampling for oil and grease, volatile organic compounds, and coliform bacteria. In these situations, the sample container should not be rinsed with the wastewater prior to sampling). However, some sample containers, such as those used for bacteriological sampling, require special cleaning procedures. Bacteriological sample containers must be sterilized prior to sample collection. The inspector should refer to Standard Methods for the Examination of Water and Wastewater and 40 CFR Part 136 for proper procedures on sample container preparation. Table 3-4 outlines required sample containers, sample preservation and sample holding times.

### ***Type of Sample***

The type of sampling which will be undertaken at the IU must be understood clearly prior to going out into the field and should be outlined in the POTW's Quality Assurance and Sampling Plan and SOP. There are two basic types of samples: grab samples and composite samples. Each type of sample has distinct advantages and disadvantages. To obtain a complete characterization of a specific facility's effluent, the two sample types may be used in combination. However, the inspector must use the appropriate sample type for compliance monitoring. For determining compliance with all applicable requirements, the inspector must use the sample method established in the industrial user's permit. (NOTE: If the sample method is inadequate the inspector should take two samples, one with the permit sample method, and the other with the sample method which the inspector deems more appropriate). In this situation the permit should be modified to reflect the appropriate method. It is very important that the POTW establish specific procedures for collecting grab and composite samples. These procedures must be consistent with the EPA guidance on grab versus composite sampling which was distributed to all POTWs with approved pretreatment programs and all of the EPA Regional Offices (NOTE: See Appendix V for a copy of this guidance). Copies of this policy can be obtained from the Regional Pretreatment coordinators. Once the POTW has established its procedures for taking grab and composite samples, the IU permit should be modified to incorporate these specific procedures so that the IU is held to the POTW's procedures.

A grab sample is an individual sample collected over a period of time not to exceed 15 minutes. Grab samples are usually taken manually, and the sample volume depends on the number of analyses to be performed. The sampler must make sure that sufficient volume of sample is taken to conduct all necessary analytical procedures, including QA QC. Grab samples represent the conditions that exist at the moment the sample is taken and do not necessarily represent conditions at any other time. Grab sampling is the preferred method of sampling under the following conditions:

- When the effluent is not discharged on a continuous basis (i.e., batch discharges of short duration), and only when the batch is continuously stirred (i.e., well-mixed) and the pollutant can be safely assumed to be uniformly dispersed.
- When sampling wastewater from an electroplating facility regulated under 40 CFR 413, if it has been demonstrated that the single grab sample is representative of the daily discharge.
- When sampling a facility where a statistical relationship can be established between previous grab samples and composite data.
- When the effluent is being screened to see if a parameter is present (NOTE: This is only true when the sample is well-mixed and representative of the discharge).
- When the waste conditions are relatively constant (i.e., are well-mixed and homogeneous) over the period of the discharge. In lieu of complex sampling activities, a grab sample provides a simple and

- accurate method of establishing waste characteristics.
- . When a POTW or State has adopted an instantaneous local limit which is based on grab samples;
  - . When it is necessary to check for extreme conditions. For example, composite sampling would tend to conceal peaks in the **pH** of a discharge. Extreme acidic and alkaline conditions may cancel each other out, causing a composite sample to appear neutral. Therefore, composite sampling cannot be used for **pH** analyses.

When specific pollutant parameters are immediately affected by biological, chemical, or physical (e.g., **pH** sensitive compounds) interactions, or have short holding times, such as **pH**, temperature, total phenols, residual chlorine, soluble sulfide, hexavalent chromium, cyanide, volatile **organics**, and dissolved oxygen, individual grab samples must be taken. Individual grab samples or composite samples (with proper **compositing** procedures) may be taken for oil and grease, and cyanide (as described below). The sampler must be careful in sampling for oil and grease, since these pollutants tend to adhere to the sample container.

Composite samples are samples collected over a period of time greater than 15 minutes. formed by an appropriate number of discrete **samples** which **are: 1) collected at equal** time intervals and combined in proportion to the wastewater **flow**, or 2) are equal volumes taken at varying time intervals in proportion to the wastewater flow, or 3) equal *volumes* taken at equal time intervals. Composite samples are used to determine the average pollutant concentration during the compositing period. Various methods for compositing **samples** are available. Composite samples may be collected individually at equal time intervals if the flow rate of the sample stream does not vary more than plus or minus 10 percent of the **average** flow rate, or they may be collected proportional to the **flow** rate. The industrial user's **permit** may specify which composite sample method to use, either time composites or flow-proportional composites. **The** compositing methods, all of which depend on either continuous or periodic sampling, are described below:

- **Time Composite Sampline:** Composed of constant volume discrete sample aliquots collected **at** constant time intervals. This method provides representative samples when the flow of the sampled stream is relatively constant (i.e.. when the flow does not vary by more than 10% of the average **flow** rate over time).
- . **Flow-Proportional Sampline:** There are two methods used to collect **a** flow-proportional composite sample. In the **first** method, the time between samples is constant, and the volume of each **sample** is proportional to the flow at that given moment in time (i.e., **the** volume of the sample varies over time as the flow changes). This is the preferred method of sampling when taking a manually composited sample. This method requires that discrete samples be collected over the operating day **and** then be manually composited. It is crucial. when using this method, to have accurate flow data continuously recorded during the sampling period.

The second flow-proportional sampling method involves collecting a constant sample volume for each volume of wastestream flow (e.g., **200** ml sample collected for every 5,000 gallons of flow) at time intervals inversely proportional to the stream flow. This is the preferred method when taking composite samples using an automatic sampler. This method is based on taking a sample after a set amount of wastewater has been discharged. **This** method provides representative samples of all

wastestreams when the flow is measured accurately. In the other method, the sample is collected by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between aliquots.

A composite sample should be collected over a workday. If a facility operates and discharges 24 hours per day, then the composite sample should be taken as a 24-hour composite (either flow proportional or time composite). If a facility operates 24 hours per day but only discharges wastewater for six hours, a six hour composite sample should be collected. In general, composite samples should be collected to assess compliance with Categorical Standards and local limits, as long as the limits are daily, weekly, or monthly averages, except for those parameters listed above which must be taken by grab sample. If the POTW is using an automatic sampler with discrete sample containers, the inspector should keep track of any sample bottles which are empty and seek an explanation from the IU for all empty sample bottles from the composite sampler.

Appendix V contains the EPA memorandum, "The Use of Grab Samples to Detect Violations of Pretreatment Standards." This memo explains in greater detail when it is appropriate to use grab samples for determining the compliance status of industrial users with categorical standards and local limits.

As stated earlier, some pollutant parameters should be collected as grab samples, but may be collected as composite samples, if specific sampling/preservation techniques for each parameter are followed. Those parameters which may be a grab or a composite sample are oil and grease, and cyanide. Each of these is discussed in turn below.

### **Oil and Grease**

Method 5520B in Standard Methods for the Examination of Water and Wastewater, 17<sup>th</sup> edition, is an approved method of analysis for oil and grease and is listed in 40 CFR 136. Standard Methods states that a representative sample shall be collected in a wide-mouthed glass jar that has been rinsed with a solvent. It also proceeds to explain that composite samples should not be taken due to the potential losses of grease on the sampling equipment. This concept applies only to samples which are collected using automatic samplers. Oil and grease samples may be **composited**, if the following steps are taken:

- The compositing vessel is made of glass and has been precleaned and rinsed with the solvent to remove the detergent film.
- The sampling jar is made of glass and has been precleaned and rinsed with the solvent to remove the detergent film.
- Collect the sample directly into the sample jar and properly preserve the sample with HCl or H<sub>2</sub>SO<sub>4</sub> to a pH<2. Pour the sample into the compositing vessel. After each additional sample is added to the collection vessel, the pH should be **re-checked** and adjusted if necessary. Preserve the sample in the compositing vessel by cooling to 4°C and holding at a pH<2. When taking the pH, it is recommended

that the **pH** paper or probe not be put directly into the sample. To make sure that equal volumes of each sample are taken, mark the outside of the sampling jar. Repeat this process for the compositing period.

When the compositing is finished, both the compositing vessel, which has the sample in it, and the sampling jar must be submitted to the laboratory for analysis. The entire volume in the compositing vessel must be sent to the laboratory, a smaller sample may not be taken from this vessel. The sample jar is sent to the laboratory to allow the oil and grease which has adhered to the side of the container to be extracted and included as part of the sample which is analyzed.

This compositing procedure must be approved by the EPA prior to its use for determining compliance with oil and grease pretreatment standards, but is available subject to approval by the EPA Regional Office.

### ***Cyanide (Total)***

EPA methods 4500-CN-C, D, or E are approved methods from Standard Methods, 17<sup>th</sup> edition, which are listed in 40 CFR 136. Most of the sampling guidance to date has recommended that total cyanide samples be collected as grab samples (see Grab Sample memo in Appendix V). A manual composite for total cyanide may be collected if the following steps are followed. (**NOTE:** "The first 12 steps must be followed to preserve a grab sample, even if it is not being **composited**).

- 1) Collect a grab sample into either a glass or polyethylene sample bottle.
- 2) Check for oxidizing agents (**e.g.**, chlorine). If oxidizing agents are not present, then go to step #6.
- 3) If oxidizing agents are present, add **0.6g** of ascorbic acid (see NOTE below).
- 4) Repeat steps 2 and 3 until no oxidizers are present.
- 5) Add one additional dose of ascorbic acid.
- 6) Check the sample for sulfides by placing a drop of the sample on a piece of lead acetate test paper which has been moistened with acetic acid buffer solution (**pH** 4). If the lead acetate test paper is darkened, sulfides are present. If sulfides are not present, go to step 11.
- 7) If sulfides are present, add cadmium nitrate powder.
- 8) Repeat steps 6 and 7 until the moistened lead acetate test paper no longer darkens.
- 9) Add one additional dose of cadmium nitrate powder.
- IO) Filter the sample to remove the sulfides which have precipitated out of the solution. The filtrate then goes to step 11.
- 11) Preserve with **NaOH** to a **pH**>12.
- 12) Pour the sample into a compositing vessel. After each additional sample has been individually preserved (steps 1-11), add it to the compositing vessel. The **pH** of the composite should be rechecked and adjusted as needed. The sample in the compositing vessel must remain preserved at **4°C** and at **pH**>12. When taking the **pH**, it is recommended that the **pH** paper or probe not be put directly into the sample. To ensure that **equal** volumes of each sample are taken, mark the outside of the sampling jar. Repeat this process for the compositing period.

(**NOTE:** 40 CFR 136.3, Table II, footnote 5 explains that if residual chlorine is present, the sample **must** be treated with **0.6g** of ascorbic acid. Footnote 6 to this table also states that if sulfides are present in the sample, it must be **analyzed** within 24 hours. It continues to explain that samples can be tested for the

presence of sulfides using lead acetate paper. If sulfides are present, cadmium nitrate powder should be added until a negative spot test is obtained with the lead acetate paper. The sample should then be filtered and NaOH be added to a pH of 12. According to 40 CFR 136.3, checking and preserving for sulfides allows a holding time of 14 days rather than 24 hours if sulfides are present and not treated.

40 CFR 136.3 Table 1B states that Standard Methods for the Examination of Water and Wastewater, 17th edition - methods 4500-CN-C, D, E, and G are approved analytical methods for cyanide analyses. The preservation techniques for these samples are explained in the analytical methods. When conflicts arise, the information stated in 40 CFR 136.3, Table II supersedes the preservation technique in the analytical method. NOTE the preservatives in 40 CFR 136 are not the same as those stated in Standard Methods).

This process of compositing a total cyanide sample is very resource intensive. Therefore, it is not recommended for routine compliance sampling for cyanide. Table 3-2 lists the advantages and disadvantages of each compositing sampling method. Either manual or automatic sampling techniques can be used. If a sample is composited manually, sample manipulation should be minimized to reduce the possibility of contamination. The inspector must use the sampling method specified in the permit, but if the sampling method is inadequate, the inspector should pull a sample using the permit method and the method which is deemed more appropriate.

**Table 3-2**  
**Composite Sampling Methods**

<i>Method</i>	<i>Advantages</i>	<i>Disadvantages</i>	<i>comments</i>
<b>Time Composite</b>			
Constant sample volume, constant time interval between samples.	Minimal instrumentation and manual effort. Requires no flow measurement.	May lack representativeness especially for highly variable flows.	Widely used in both automatic samplers and manual handling.
<b>Flow Proportional Composite</b>			
• Constant sample volume, time interval between samples proportional to stream flow	Minimal manual effort.	Requires accurate flow measurement reading equipment.	Widely used in automatic as well as manual sampling
• Constant time interval between samples, sample volume proportional to total stream flow at time of sampling.	Minimal instrumentation.	Samples must be manually composited. Discrete samples must be taken. Chance of collecting samples which are too small or too large for a given composite volume.	Used in automatic samplers and widely used as manual method.

**ON-SITE ACTIVITIES**

Once the sampling plan has been established and pre-inspection activities have been completed, the focus of the inspection turns to the on-site activities performed by the inspector. This section outlines the procedures which POTW inspectors should follow when **conducting** on-site sampling at their industrial users. As with inspections, in many cases the **first** thing the sampler will do is conduct an opening conference with the **IU** representatives. Where the **POTW** has developed a working relationship with the **IU** or where **there** are suspected violations, the sampler should proceed immediately with sampling and then conduct a closing conference as **necessary**.

**Sampling Location**

The first step in preparing to sample is to verify that the sample location is **appropriate**. The **IU** permit must specify the sampling location for compliance sampling (40 CFR 403.8(f)(2)(iii)). This sampling location must be representative of the actual discharge from the facility. When conducting a compliance sampling site visit, the inspector should use the sample location specified in the industrial user's permit. If the sample location specified in the permit is not adequate to collect a representative sample, the inspector should determine an alternative location. This determination should be **based** on the inspector's knowledge of the plant itself, the production processes, and the outfalls. If there is a conflict between the sample location described in the permit and the location the inspector believes is most representative, samples should be collected at both sites. The reason for the conflict should be **thoroughly** documented for later resolution by the **POTW**. If necessary, the permit must be amended to reflect the correct sampling location(s).

The Federal Categorical Standards apply at the end-of-process (or at the end of **treatment**, if treatment exists), unless the standard specifies a different location to collect the sample (e.g., in 40 CFR 4.33 Metal Finishing, the sample location for cyanide is after the cyanide destruct system prior to dilution with other streams). If process effluent is mixed prior to treatment **with** unregulated wastestreams or dilution water or if local limits apply at a different point, the combined wastestream formula (**CWF**) or flow weighted average (**FWA**) formula must be used (40 CFR 403.6). Samples under this circumstance would be taken after treatment. If the samples are being taken to determine compliance, all associated flows must be measured

**The** inspector should always collect samples from a sampling location or locations that reflect the total regulated effluent flow (i.e., is representative). Convenience and accessibility are important considerations, but are secondary to the representativeness of the sample. **The** most representative samples **will be** drawn from a wastewater depth where the flow is turbulent and **well mixed** and the chance of solids settling is minimal. Depending on the sampling location, ideally, the depth of sample collection should be 40 to 60

percent of the **wastestream's** depth. Stagnant areas must be avoided as well, particularly if the wastewater contains immiscible liquids or suspended solids. To avoid contamination, the inspector should take care to collect samples from the center of the flow with the opening of the sampling device or container facing upstream. Wide channels or paths of **flow** may require dye testing to determine the most representative sampling site. If dye testing is inconclusive, multiple samples may need to be collected by cross sectional sampling.

### *Sample Collection Techniques*

To obtain a representative sample, sampling must be conducted where wastewater is adequately mixed. Ideally, a sample should be taken in the center of the **flow** where the velocity is highest and there is little possibility of solids settling. The inspector should avoid skimming the surface of the wastestream or dragging the channel bottom. Mixing of the flow is particularly important for ensuring uniformity. Sampling personnel should be cautious when collecting samples near a weir because solids tend to collect upstream and floating oil and grease accumulate downstream.

Samples can be collected either manually (grab or composite) or with automatic samplers. The following general guidelines apply when taking samples:

- Take samples at the site **specified** in the industrial user permit (**and or** at the site selected by the inspector to yield a representative sample if the inspector concludes that the site specified in the permit is not appropriate)
- **Use** the sampling method (grab, or composite) required by the IU permit. If the permit sampling method is inappropriate (e.g., if it calls for composite sampling for **pH**), then the inspector should note this in his/her field notebook and take two samples: one using the permit method and one using the method deemed most appropriate by the inspector. Samples of certain pollutant parameters may not be taken by automatic samplers, but must be taken by manual grab samples. These parameters include: dissolved oxygen, residual chlorine, **pH**, temperature, oil and grease, fecal coliforms, purgeable organics, and sulfides.

Avoid collecting large nonhomogeneous particles and objects.

- Collect the sample facing upstream to avoid contamination.

Do not rinse the sample container with the effluent when collecting oil and grease and microbiological samples, but till the container directly to within 2.5 to 5 cm from the top.

Fill the container completely if the sample is to be analyzed for purgeable organics, dissolved oxygen, ammonia, hydrogen sulfide, free chlorine, **pH**, hardness, sulfite, ammonium, ferrous iron, acidity, or alkalinity.

- Collect **sufficient** sample volume to allow for quality assurance testing. (**NOTE:** Table 3-4 provides a guide to numerous sample volume requirements but additional volume may be necessary for QA testing)

When taking a grab sample, the entire mouth of the container should be submerged below the surface of

the wastestream. A wide mouth bottle with an opening of at least two inches is recommended for this type of sampling. When using a composite sampler, the sample line should be kept as short as possible and sharp bends, kinks, and twists in the line (where solids can settle) should be avoided. The sample line should be placed so that changes in flow will not affect sample collection.

### ***Sample Volume***

The volume of samples collected depends on the type and number of analyses needed. This will be determined by the parameters to be measured and the requirements of the **analytical** laboratory being used. Sample volume must be **sufficient** for all analyses, including QA/QC and any repeat analyses used for verification. Laboratory personnel should be contacted for the sample volume required to complete all analyses, since the lab is in the best position to estimate the necessary volume of **sample**. Individual, minimum composite portions should be 100 mls. with a total composite volume of 2-4 gallons. Larger volumes may be necessary if samples are to be separated into aliquots or if bioassay tests are to be conducted.

Volume requirements for individual analyses range **from** 40 ml for **pH** and volatile organic determinations to 1,000 ml or more for BOD, oil and grease, and settleable solids. The inspector should always collect more than the minimum sample volume to allow for spillage and laboratory reruns. Table 3-3 lists minimum volume requirements for various parameters.

### ***Sample Preservation and Holding Times***

Preservation techniques ensure that the sample remains representative of the wastestream at the time of collection. Since most pollutants in the samples collected are unstable (at least to some extent), this instability requires that the sample be analyzed immediately or that it be preserved or fixed to minimize changes in the pollutant concentration or characteristics between the time of collection and analysis. Because immediate analysis is not usually possible, most samples are preserved regardless of the time of analysis.

***This preservation must take place as soon as possible after collecting the sample. This means that preservation must take place in the field*** (see 40 *CFR 136.3*). The most common procedures used for preserving samples include icing, refrigeration, **pH** adjustment, and chemical fixation. When chemical fixation is used, the chemical preservative must be added before the samples are transferred to the laboratory. Likewise, refrigeration should be supplied immediately upon taking the sample. For many samples, if preservatives are not appropriately used, bacteria can quickly degrade certain pollutant constituents (e.g., phenols and phosphorous). Other constituents may volatilize (e.g., cyanide and sulfides) or may react to form different chemical species (e.g., hexavalent chromium). Proper preservation and holding time for each parameter is essential for the integrity of the monitoring program. (See Table 3-4 and refer to 40 CFR Part

136).

Problems may be encountered, however, when N-hour composite samples are collected. Since sample deterioration can take place during the compositing process, it is necessary to preserve or stabilize the samples during the **compositing** in addition to preserving the aggregate sample before shipment to the laboratory. Preservation techniques **vary** depending on the pollutant parameter that is to be measured; therefore, the inspector must be familiar **with** the 40 CFR Part 136 preservation techniques to ensure proper sample handling and shipment. It is important to **verify** that the preservation techniques for one parameter do not affect the **analytical** results of another parameter **in** the same sample. If there is this possibility, then two discrete samples should be collected and preserved independently. Sample preservation should be provided during compositing, generally by refrigeration to 4°C (or icing) [**NOTE:** See 40 CFR 136.3 Table II. Footnote 2.]

Refrigeration is the most widely used preservation technique because it has no detrimental effect on the sample composition and does not interfere **with** any analytical methods. Refrigeration **requires** that the sample be quickly chilled to a temperature of 4°C, which suppresses biological activity and volatilization of dissolving gases and organic substances. This technique is used at the start of sample collection in the **field** and during sample shipment, and continued until the sample is **analyzed** by the **laboratory**. Sample temperature should be **verified** and recorded **by** the inspector. This is particularly important if the analytical results are to be used in an enforcement action.

In addition to preservation techniques, 40 CFR Part 136 indicates maximum holding times. Times listed are the maximum times between sample collection and analysis that are **allowed** for the sample to be considered valid. (**NOTE:** Some parameters have separate holding times from the time of sample collection to extraction preparation and from extraction preparation to analysis). A **wastewater** sample becomes a sample when the first aliquot is collected. At that point, holding time limitations begin. A detailed list of preservation methods and holding times appears in Table 3-4 at the end of this chapter. These sample preservation procedures and holding times were selected by the **EPA** because they retard sample degradation and minimize **monitoring** costs by extending holding times as long as possible.

### ***Sample Documentation***

Since many sampling reports may be used in enforcement proceedings, the inspector must keep a precise record of sample collection and data handling. All field records containing these data must be signed by the inspector at the time of collection, including all chain-of-custody forms. If required, the following

information should also be documented in the field record:

- **Unique Sample or Lon Number:** All samples should be assigned a unique identification number. If there is a serial number on the transportation case, the inspector should add this number to their field records.
- **Date and Time of Sample Collection:** The date and time of sample collection must be recorded. In the case of composite samples, the sequence of times and aliquot size should be noted.
- **Source of Sample (Facility Name and Address):** The name and address of the facility being sampled should be recorded as well as a narrative description **and/or** diagram referring to the particular site where the sample was taken should be included.
- **Name of Sampling Personnel:** The name(s) and person(s) taking the sample must be indicated. For a composite sample, the name(s) of the person(s) installing the sampler and the name(s) of the person(s) retrieving the sample must be included.
- **Sample Type:** Each sample should indicate whether it is a grab or composite sample. If the sample is a composite, volume, and frequency of individual samples should be noted.
- **Preservation Method:** Any preservatives (including the amount) added to the sample should be recorded. The method of preservation (e.g., refrigeration) should be indicated.
- **Analysis Required:** All parameters for which the sample must be analyzed should be specified.
- **Field Analysis:** Field measurements must be recorded at the time that the analysis is completed. Examples of analyses which must be conducted and recorded in the field include: **pH**, temperature, dissolved oxygen, residual chlorine, and sulfites. Field analyses should be treated the same as any other sample, i.e., the sampler must record the date of the sample, type of sample, name of the sampler, chain of custody, etc.
- **Flow:** If flow is measured at the time of the sampling, the flow measurement must be recorded. If the sample is to be used to determine compliance, flow must be measured.
- **Production Rates:** Information on products manufactured and production rates should be included since many effluent limitations are based on production rates.
- **Date, Time and Documentation of Sample Shipment:** The shipment method (e.g., air, rail, etc.) as well as the shipping papers or manifest number should be recorded.
- **Comments:** This refers to all relevant information pertaining to the sample or the sampling site. Such comments include the condition of the sample site, observed characteristics of the sample, environmental conditions that may affect the sample, and problems encountered in sampling.

Each of these items must be recorded by the POTW inspector when conducting site sampling, but this information should also be kept by the industrial user when it conducts its required self-monitoring. This information should be available for review by the POTW when conducting an on-site inspection or sampling visit.

### ***Sample Identification and Labeling***

Each sample must be accurately and completely identified. At the time a sample is collected, a waterproof, gummed label and a waterproof tag which is able to withstand field conditions should be attached to the sample container. This label and tag are necessary to prevent any misidentification of samples, since it

provides the laboratory with relevant information for analysis, such as: the name of the sample collector, the sample identification number, the date and time of sample collection, the location of the sample collection, the preservatives used, the type of sample (grab or composite) and the identification of the parameters to be analyzed. Sample identification, therefore, is a crucial **part** of chain-of-custody. Sample tags should be used as part of the chain-of-custody process because they can be removed after the sample has been transported to the lab and placed in the evidence **file** for that sample while labels are usually discarded with the sample. The tags can then be used as evidence in an enforcement proceeding. The sample identification procedures should be incorporated into the POTW's Quality Assurance and Sampling Plan (see p 56 for a further discussion of the plan).

Sample seals or cooler seals (i.e., seals placed around a cooler with similar samples inside) should be used to protect the sample's integrity from the time it is collected to the time it is opened in the laboratory. The seal should contain the collector's name, the date and time of sample **collection** and the sample identification number. Information on the seal must be identical to the information on the label and tag. In addition, the seal must be attached so it must be broken to open the sample container or gain access to the sample container in the case of a cooler seal. Caution should be observed to assure that glue on the sample seals and tag wires does not contaminate samples, particularly those containing volatile **organics** and metals.

### ***Chain-of-Custody Procedures***

Once an appropriate sample has been obtained and the sample collection methods are properly documented, a written record of the chain of possession of that sample must be made. "Chain-of-custody" refers to the documented account of changes in possession that occur for a particular sample or set of samples (see Definition section for a definition of "custody"). Chain-of-custody procedures are a **critical** aspect in monitoring industrial users, and these procedures should be incorporated into the POTW's Quality Assurance and Sampling Plan (see p. 56 for a further discussion of the plan). The chain-of-custody record allows an accurate step-by-step recreation of the sample path, from its origin through its final analysis in the **laboratory**. Every sample needs to be accompanied by a chain-of-custody tag which is properly signed and transferred to each person in the chain, from the original sampler to the final person involved in analyzing the sample. Some of the information that needs to be addressed in chain-of-custody are:

- . Name of the person collecting the sample;
- . Sample identification number(s);
- . Date and time of sample collection;
- . Parameters to be analyzed;
- . Location of sample collection; and

. and signature(s) of **all** persons handling the samples in the field and in the laboratories.

To ensure that all necessary information is documented, a chain-of-custody form should be developed by the POTW. An example of such a **form** used by the EPA is found in Appendix X. Chain-of-custody forms should be preprinted on carbonless, **multipart** paper so that all personnel handling the sample receive a copy. All sample shipments must be accompanied by the chain-of-custody record while a copy of these forms should be retained by the originator. In addition, all receipts associated with the shipment should be retained. Carriers typically will not sign for samples; therefore, seals must be used to verify that tampering has not occurred during shipment

When transferring possession of samples, the transferee must sign and record the date and time on the chain-of-custody record. In general, custody transfers are made for each sample, although samples may be transferred as a group, if desired. Each person who takes custody must fill in the appropriate section of the chain-of-custody record. Finally, the person or group at the POTW responsible for permitting **and/or** compliance and enforcement should receive a copy of the completed chain-of-custody form, particularly if the sample results are to be used for enforcement purposes.

Chain-of-custody records are crucial if the analytical data are to be used in an enforcement proceeding because they allow such data to be introduced as evidence without testimony of the persons who made the record. Therefore, it is important that all chain-of-custody records be complete and accurate. To maintain the sample's integrity, chain-of-custody records must show that the sample was properly collected, preserved, and analyzed, and was not tampered with during shipment. Since it is not possible to predict which violations will require legal action, it should be assumed that **all** data generated from sampling will be used in court. Therefore, all compliance samples taken at an industrial user should follow chain-of-custody procedures.

### ***Sample Packaging and Shipping***

After the samples are properly **labelled**, they should be placed in a transportation case along with the chain-of-custody form, pertinent field records, and analysis request forms. Glass bottles should be wrapped in foam rubber, plastic bubble wrap, or other material to prevent breakage during shipment. The wrapping can be secured around the bottle with tape. Samples should be placed in ice or a synthetic ice substitute that will maintain sample temperature at 4°C throughout shipment. Ice should be placed in double-wrapped watertight bags to ensure the water will not drip out of the shipping case. Metal or heavy plastic chests make good sample transportation cases. Filament tape wrapped around each end of the ice chest ensures that it will not open during transport. Sampling records should be placed in a waterproof bag, envelope or airtight sample bottle and taped to the inside lid or other appropriate place inside the transported container to prevent

tampering or breach of custody. Shipping containers should also be sealed to prevent tampering. Sample Packaging and Shipping considerations should be included in the POTW's Quality Assurance and Sampling Plan (see p. 56 for a further discussion of the plan).

Most samples will not require any special transportation precautions except careful packaging to prevent breakage and or spillage. If the sample is shipped by common carrier or sent through the U.S. mail, it must comply with DOT Hazardous Waste Materials Regulations (49 CFR Parts 171 -177). Air shipment of hazardous waste materials may also be covered by requirements of the International Air Transport Association (IATA). Before shipping a sample, the inspector should be aware of, and follow, any special shipping requirements. Special packaging and shipping rules apply to substances considered hazardous as defined by IATA rules. Wastewater samples are not generally considered hazardous materials (see Footnote #3 in Table 3-4).

#### **Quality Control**

Control checks should be performed during the actual sample collection to determine the performance of sample collection techniques. In general, the most common monitoring errors are caused by improper sampling, improper preservation, inadequate mixing during compositing, and excessive holding time. The following samples should be used to check the sample collection techniques:

- Duplicate Samples (Field): Duplicate samples are collected from two sets of field equipment installed at the site, or duplicate grab samples are collected from a single piece of equipment at the site. These samples provide a precision check on sampling equipment and techniques.
- Equipment Blank: Is an aliquot of analyte-free water which is taken to and opened in the field. The contents of the blank are poured appropriately over or through the sample collection device, collected in a sample container, and returned to the laboratory as a sample to be analyzed. Equipment blanks are a check on the sampling device cleanliness.
- Field Blank: Is an aliquot of analyte-free water or solvent brought to the field in sealed containers and transported back to the laboratory with the sample containers. The purpose of the trip blank is to check on sample contamination originating from sample transport, shipping and from site conditions.
- Preservation Blanks: Is an aliquot of analyte-free water (usually distilled water) to which a known quantity of preservative is added. Preservation blanks are analyzed to determine the effectiveness of the preservative, providing a check on the contamination of the chemical preservatives.

The quality control measures taken by the POTW should be included in the POTW's Quality Assurance and Sampling Plan (see p. 56 for a further discussion of the plan). Quality control is discussed in greater detail later in this chapter. This full discussion includes a review of laboratory as well as sampling quality assurance and quality control techniques.

**SAFETY CONSIDERATIONS DURING SAMPLING ACTIVITIES**

Inspection and sampling activities are **often** carried out under hazardous situations. In developing the sampling plan, the inspector should not include sampling locations which pose a threat to health or safety. It is recommended that sampling and inspection teams include at least two people for safety purposes. Under known hazardous conditions, a two-person team should be **mandatory**. The sampling team should use all required safety equipment and protective clothing. Appendix IV lists specific hazards which are associated with various industrial facilities. The inspector should use this appendix as a reference when conducting sampling activities at any of the listed industries.

Continuous education is essential to a successful safety program. The inspector should be familiar with the hazards associated with sampling, in addition to the safety measures to be followed. For example, if the inspector is required to enter a manhole or other confined space, training in confined space entry and rescue procedures should be required. In addition, a **permit** for confined space entry is now required **under OSHA**, and the permit must be obtained prior to field personnel entering a confined space. Potential **hazards** in a confined space include toxic gases, such as hydrogen sulfide, chlorine, and carbon monoxide; or **explosive** gases, such as gasoline vapors or methane. In addition, an atmosphere may be hazardous because **there** is not enough oxygen to support life due to the presence of other gases. A confined space, such as a **manhole**, should not be entered until the atmosphere has been tested for sufficient oxygen and the lack of toxic or explosive gases. Such a confined space should never be entered alone or without a lifeline.

In general, the potential hazards that POTW personnel will encounter while performing inspection or sampling at industrial users can be divided into two areas: physical hazards and atmospheric hazards. The ability to recognize these hazards and follow proper procedures will eliminate most accidents.

***Physical Hazards***

A sampling location can present several potential hazards. Sampling activities are often carried out in locations that meet the criteria set forth in the definition of **confined** space. A confined space is defined as a space having limited means of entry or exit which is subject to a deficiency of oxygen, and the accumulation of toxic or combustible gases. Such locations include manholes, pumping stations, wetwells, storm drains, and water meter vaults.

Care must be exercised when removing manhole covers and entering manholes or other confined spaces. Manhole covers should be opened and removed with a properly designed hook. Manhole covers should **never** be opened with fingers. An acceptable tool can be made from 3/4 hex or round stock. **Two** inches of one

end should be bent at a right angle and the opposite end should be formed into a handle wide enough for placement of both hands. The two inch hook can be inserted into one of the holes in the cover and **lifted** by straightening the legs. Improper **lifting** of a manhole cover may result in back injury. Caution must be exercised when lowering and lifting sampling equipment. A sampler is much heavier when it is full and is sometimes **difficult** to **lift**. Tools should be lowered into and **lifted** out of the manhole in a bucket to prevent the tools from falling on someone below.

Generally, the top of a manhole is flush with the surrounding surface. Therefore, a person entering the manhole **may** not have anything to hold on to for support. ladders and steps leading into manholes and other types of **confined** spaces are subject to corrosion and may not be well maintained. These structures should be examined prior to entry. If there is any doubt regarding the soundness of the manhole steps, a portable ladder should be used. Other physical hazards in a confined space include the following: excessive depths; excessive flow; poor visibility; wet/slippery surfaces; harmful animals, insects or organisms (spiders, snakes, bacteria); protruding or sharp objects; and falling objects. Other physical hazards are listed in Appendix IV.

**Atmospheric Hazards**

Atmospheric hazards are comprised of three primary types: oxygen deficient atmospheres; explosive, flammable atmospheres; and toxic atmospheres. These types of hazards require air monitoring and ventilation before entering a manhole or other confined space. Air monitoring equipment is discussed later in this chapter.

**Oxygen Deficient Atmosphere:**

The minimum OSHA requirement for oxygen concentration in the atmosphere is 19.5%. A **Self-Contained Breathing Apparatus** is necessary to enter an atmosphere with less than 19.5% oxygen. The oxygen enriched atmosphere, which exists when the oxygen concentration is greater than 25%. is also considered hazardous because of its ability to support combustion. Normal air consists of the gases listed in Figure 3-2. Other gases, such as nitrogen and carbon dioxide, which are harmless under normal conditions, may build up in confined spaces in quantities large enough to displace the oxygen necessary to support life. When the concentration of oxygen in the atmosphere falls to 10-16%. a person will experience shortness of breath. Loss of consciousness will occur at a **6-10%** oxygen concentration and death will occur rapidly when the concentration of oxygen falls

<u>Gas</u>	<u>Volume</u>
Nitrogen	78.09%
<b>Oxygen</b>	20.95%
Argon	0.93%
Carbon Dioxide	0.03%

**Figure 3-2 Atmospheric Constituents**

below 6%.

**Explosive Atmosphere:**

Gasoline is the most common flammable liquid found in the sewer system. **The** major source of gasoline is leaking underground storage tanks and accidental discharge or spills. Gasoline is lighter than water, enabling it to float on top of the wastestream, and the vapors spread out in the collection system. It takes a very small amount of gasoline to generate an explosive atmosphere in a confined space.

Methane is the most common flammable gas encountered in the sewer system. Methane gas is the product of waste decomposition and is the primary component of natural gas. A leak in a natural gas pipe may result in the gas seeping into the collection system. Methane is lighter than air, which allows it to collect at the top of a confined space.

**Toxic Atmosphere:**

There are various guidelines for assessing chemical hazards in the atmosphere. The Threshold Limit Values (TLVs) are guidelines developed and published by the American Conference of Governmental Industrial Hygienists (ACGIH) to be used for identifying and controlling potential hazards. One **form** of the TLS - the time weighted average (TWA) - refers to the vapor phase concentration a worker may be exposed to for an eight hour day or 40 hour work week without chronic or acute health effects. TLV-TWA numbers are sometimes used to calculate IU discharge screening levels for volatile organic compounds. The EPA has issued a guidance document entitled "Guidance to Protect POTW Workers from Toxic and Reactive Gases and Vapors," (EPA 812-B-92-001). **This** document should be used to evaluate the potential for exposure to toxic atmospheres and necessary steps for avoiding contact with such atmospheres.

Hydrogen sulfide ( $H_2S$ ) is the most common gas found in the collection system. **The** gas, which is formed by anaerobic decomposition of organic matter, is heavier than air and tends to collect at the bottom of an enclosed space. At low concentrations, hydrogen sulfide has an odor of rotten eggs, at higher concentrations, however, the olfactory system becomes impaired **and** the gas cannot be detected by smell. The TLV-TWA for hydrogen sulfide is 100 **ppm**. At higher concentrations of  $H_2S$ , damage can occur to the eyes, nervous system, and respiratory system. A caustic solution (sodium sulfide) is formed when the gas comes into contact with moist tissue, such as in the eyes and respiratory tract, which causes the irritation and danger from the chemical. At concentrations of 500-1,000 ppm the respiratory system is paralyzed and death will occur.

Hydrogen cyanide gas may be generated when cyanide salts react with an acidic wastestream. Cyanide salts are **often** found in plating baths and metal finishing facilities. The gas causes death by preventing the transfer of oxygen within the bloodstream.

Chlorine (Cl<sub>2</sub>) gas may be encountered at plating facilities where it is used for cyanide destruction. Aside from the irritating odor, chlorine gas is corrosive in the presence of moisture. It **combines** with moisture in the lungs and the respirator) system to form hydrochloric acid. Pulmonary edema (fluid in the lung may occur at 50 ppm and at 1,000 ppm death occurs rapidly.

Carbon monoxide (CO) is another gas that is generated in a collection system from anaerobic decomposition of organic materials. Asphyxiation occurs from exposure to this gas because the hemoglobin of the blood has 300 times more **affinity** for carbon monoxide than for oxygen. Carbon monoxide combines with hemoglobin to form **carboxy** hemoglobin. As a result, blood cells with CO cannot transport **oxygen** to body tissues, and death occurs.

Toxic vapors also present a hazard to inspectors and sampling personnel. Vapors are the volatile form of substances that are normally in a solid or liquid state. Chlorinated solvents used in degreasing or photoresist developing operations generate vapors that may accumulate in the collection **system**. Some of the vapors have an anaesthetic effect when inhaled. In addition, the vapors are generally heavier than air causing oxygen in a confined space to be displaced which may create an oxygen deficiency in that space.

## ***Safety Equipment***

Recognizing the physical and atmospheric hazards associated with sampling and inspections is important. Simply acknowledging the hazards, however, does not guarantee safe working conditions. An employer is obligated to provide safety equipment and to establish a training program for employees. ‘The important thing to remember is that the responsibility for using the equipment and following safety procedures rests **with** the individual inspector. The following discussion will acquaint the inspector with proper safety techniques,

### **Protective Clothing:**

Protective clothing is an important aspect of safety, and the guidelines presented below should be followed when conducting inspections and sampling at industrial users.

- **Hard Hat** - All persons entering a confined space are required to wear a hard hat. A full strength hard hat with a brim and chin strap provides protection from head injuries.
- **Coveralls** - A person’s skin should be covered **as** much as possible to prevent scrapes and cuts and to avoid skin contact with hazardous substances.
- **Gloves** - Hand protection is necessary when collecting **and/or** handling wastewater samples. In addition to preventing absorption of hazardous chemicals through the skin, gloves will protect the hands from cuts and scratches.
- **Shoes** - Rubber-soled, non-skid, steel-toed shoes and boots must always be **worn** in or around a confined space. Safety shoes are designed to protect against impact **and/or** hazardous chemicals.
- **Ear Plugs** - Ear plugs should be **worn** when working in areas with high noise levels (e.g., general

manufacturing areas). This equipment will protect the inspector from the cumulative effects of loud noises in the work place.

- **Safety Goggles** - Safety goggles are necessary during inspections and sampling to prevent eye contact with hazardous substances. Contact lenses are **often** prohibited around some industrial processes such as plating operations. If a hazardous substance comes into contact with a person's eye through splashing or exposure to mists or vapors, that substance may become trapped behind the lens where it would be **difficult** to flush out and could cause severe eye damage.
- **Safety Vests** - Safety vests are necessary warning devices in areas with vehicle traffic.

#### **Traffic Control:**

Traffic diversions are necessary when inspecting and/or sampling in areas subject to vehicle traffic. For protection of the public as well as employees, the devices must be installed immediately upon arrival at the site and must not be removed until the work is completed. The following devices may be used to route traffic away from an open manhole.

- **Warning Devices** - Rotating/flashing lights and arrow boards should be placed between the work area and oncoming **traffic** to alert drivers and **pedestrians**.
- **Barricades** - A vehicle or heavy piece of equipment should be placed between traffic and the working area. It should not, however, interfere with traffic.
- **High-Level Warning Flag/Cones** - Should be used to route traffic through a job-site. Flagmen should be used whenever possible and must wear reflective clothing, such as safety vests, hard hats, and safety shoes.

#### **Radio:**

A two-way radio is the most effective way to maintain contact with a main office. A sampling team should make radio contact upon arrival and departure from each sampling location. Sampling sites are often located in unpopulated areas; therefore, it is important that the radio be kept in working order in the event it is necessary to call for help. If an accident should occur, the rescuer must call for help before any assistance is given to the victim.

#### **Air Monitorine Devices:**

Before sampling in a confined space, tests should be **done** for: (1) explosive gases; (2) the presence of toxic gases; and (3) oxygen deficiency. The most effective method for detecting these conditions is with an atmospheric monitor. The gas detectors discussed below are the most commonly used for atmospheric monitoring.

- **Single Purpose Detector** - Designed to detect **specific** gases, such as carbon monoxide, methane, or hydrogen sulfide. **These** gases are commonly present in collection systems and confined spaces. Single detection units or tubes are available for measuring gases that are less common.
- **Dual Purpose Detector** - Capable of detecting lack of oxygen and explosive conditions in an area.
- **Combination Detector** - Capable of detecting a lack of oxygen, explosivity and the presence of toxic gases. **This** type of meter provides maximum protection by detecting the presence of all three hazards. **Hydrogen** sulfide and carbon monoxide are the gases usually measured because they occur most

frequently in a collection system

The atmosphere in a confined space can change suddenly; therefore, a detector that continuously monitors the atmosphere is recommended. In addition, detectors should be equipped with an audible and visual alarm which is activated in response to specific hazardous conditions or a low battery, thus eliminating the need for taking the time to read a dial or gauge.

It is important to remember that using an atmospheric tester does not ensure safe conditions. Gas detectors are only one source of information pertaining to a potentially hazardous situation. Most gas detectors are designed to test for common gases such as hydrogen sulfide, and are not effective for detecting less common substances such as trichloroethylene vapors. There are test kits available for detecting the less common gases. **The kits** consist of a bellows-type pump and glass tubes containing an indicator chemical which are sealed at the ends until they are used. The indicator chemical in the detector tube is specific to a particular contaminant or group of gases. A predetermined volume of air is drawn through the tube and the contaminant reacts **with** the indicator chemical, producing a color change that can be compared to a **color-calibrated** chart to determine an approximate concentration.

When measuring explosivity, gas meters measure the percentage of the Lower-Explosive Limit (LEL) of a calibration gas, which is usually methane. Gases are combustible throughout a range of air mixtures. The meters do not differentiate between gases, but only indicate explosivity relative to the calibration gas. The range begins **with** the LEL, which is the lowest concentration of a combustible gas or vapor in air that is necessary to support combustion. The explosive range extends upward to the Upper Explosive Limit (UEL) which is the maximum concentration that will support combustion. If the concentration of gas is below the LEL, there is insufficient fuel to support ignition. Alternatively, if the concentration is above the UEL, there is insufficient oxygen to support combustion. These limitations in the atmospheric monitoring equipment emphasize the need for constant ventilation and awareness of potential **hazards**. (**NOTE:** Combustible gas meter alarms are usually set a point well below the LEL of the gas or atmosphere being measured)

#### **Ventilation Devices:**

Few confined spaces have adequate natural or mechanically induced air movement, and in most spaces, it is **necessary** to remove harmful gases or vapors by ventilation with a blower or fan. The most common method of ventilation uses a large flexible hose attached at one end to a blower with the other end lowered into the space. The blower will push fresh air into the space to purge the area of hazardous substances. The blower allows the fresh air to enter the space at the lowest point possible. Because the atmosphere in a confined space can change quickly, ventilation should be continuous.

**Safety Harness and Retrieval System:**

Any entry into a confined space must always be performed by a team consisting of at least two **people**. A standby person must be stationed outside of the confined space and must remain in visual and radio contact with the person inside. **All** personnel required to enter the confined space must wear a safety harness. A full body parachute type harness with a lifeline attached at the shoulders is recommended. This type of harness will keep a body vertical and prevent a limp body **from** falling out when being pulled out of an area.

The lifeline should be attached to a retrieval system which includes a fall arrest mechanism. This type of safety system works in a similar manner as an automobile safety **belt** where a centrifugal locking mechanism is activated when a fall occurs. If a rescue attempt is necessary, this type of retrieval system eliminates the need to enter the space. Approximately one half of all fatalities that occur in confined spaces are unplanned rescue attempts where a worker instinctively rushes into the confined space to assist an injured co-worker.

The retrieval system should be purchased **from** a reputable manufacturer or authorized distributor as complete systems, including, repair, and training for proper use. Most components of a retrieval system must meet certain manufacturer specifications and substitution of these components may result in liability for personal injury.

Safety equipment must be maintained and inspected on a regular basis. A safety harness and rescue rope should be examined for the following: frayed strands of fibers, cuts or tears, chemical damage, decay, and kinks or extreme stiffness. Visual inspection of this equipment should be made prior to each use, and **formal** procedures should be implemented for periodic inspection and maintenance.

**Respirators:**

The primary function of a respirator is to prevent exposure to hazardous atmospheres. It is important to choose a respirator based on the job to be performed and the potential hazards to which an employee may be exposed. The basic types of respirators are:

- . Air **Purifying** - Masks which filter dangerous substances **from** the air; and
- Air **Supplying** - Devices which provide a supply of safe breathing air from a tank.

An air-purifying respirator will remove particles of dust and light concentrations of gas or vapors, but it will not protect against heavy gas concentrations. In addition, this type of respirator provides no oxygen other than what is filtered through the mask. Air-purifying respirators include the following types:

- . Gas and Vapor Respirators - Contaminated air is passed through charcoal which traps gases and vapors.
- . **Particulate** Respirator - Contaminated air is passed through a filter for removal of particles.
- . Powered Air-Purifying Respirator - A blower passes contaminated air through a device which removes

- . Testing. **All** confined spaces must be tested prior to entry.
- . Evaluation. Tests must be evaluated for oxygen level, explosivity, and potentially toxic substances. Sampling personnel should also consider necessary safety equipment.
- Monitoring. The atmosphere in a confined space is subject to change. Therefore, the area should be continuously monitored during the sampling activities.

Rescue procedures must be developed for each type of confined space that may be encountered by the sampling personnel. A written record of training and safety drills should be kept. Rescue procedures should be practiced frequently enough to ensure proficiency in any necessary rescue situations. In developing a successful training program, the POTW is encouraged to call on other agencies (e.g., local fire and rescue department) with expertise in any of the areas described above.

## ***FLOW MEASUREMENT***

Pollutant limits in the industrial user's permit are **often** expressed in terms of mass loadings to the POTW (e.g., OCPSF categorical standards). To determine a mass loading and thereby evaluate compliance with the permit limits, it is necessary for the inspector to obtain accurate **flow** data. "Flow measurement" is the commonly used **term** for this process, and this section briefly describes the types of devices and **procedures** used to measure flow. For further information, the inspector should consult two other EPA guidance manuals, the 1988 NPDES Compliance Inspection Manual and the **1981 NPDES Compliance Flow Measurement Manual**. In situations where flow measuring devices such as those described in the following sections are not available, the POTW may need to rely on the use of water consumption records at the facility. However, when a mass loading needs to be determined for assessing compliance, the POTW should have the ability to assess directly the **flow** at the facility during the sampling event. Relying on water consumption records when determining compliance with mass-based limits is not an acceptable practice and should not be used by the POTW.

### ***Open Channel Flow***

Open channel flow, where the flow occurs in conduits that are not **full** of liquid, is the most prevalent type of flow at industrial user discharge points regulated by the pretreatment program. Partially full pipes that are not under pressure are classified as open channels as well. Open channel flow is measured using both primary and secondary devices (as described below).

#### **Primary Devices:**

Primary devices are calibrated, hydraulic structures installed in the channel so flow measurements can be obtained by measuring the depth of liquid at a specific point in relationship to the primary device. Weirs and flumes are examples of primary devices.

The most common type of weir consists of a thin, vertical plate with a sharp crest that is placed in a stream, channel, or partially filled pipe. Figure 3-4 shows a profile of a sharp-crested weir and indicates the appropriate nomenclature. Four common types of sharp-crested weirs are shown in Figure 3-5. The crest is the upper edge of the weir to which water must rise before passing over the structure. The depth of water above the crest of the weir is termed the “head.” To determine **flow** rate, the inspector must measure the hydraulic head. The rate of flow over a weir is directly related to the height of water (hydraulic head) above the crest. To measure the hydraulic head, a measuring device is placed upstream of the weir at a distance of at least four times the head. To approximate the head, the inspector can measure at the weir plate. However, this value will provide only a rough estimate of flow.

The flume is an artificial channel constructed so the wastestream flows through it. The wastestream’s flow is proportional to the depth of water in the flume and is calculated by measuring the head. A flume is composed of three sections: (1) a converging upstream section; (2) a throat or contracted section; and (3) a diverging or dropping downstream section. The two principal types of flumes are the **Parshall** Flume and the **Palmer-Bowlus** Flume.

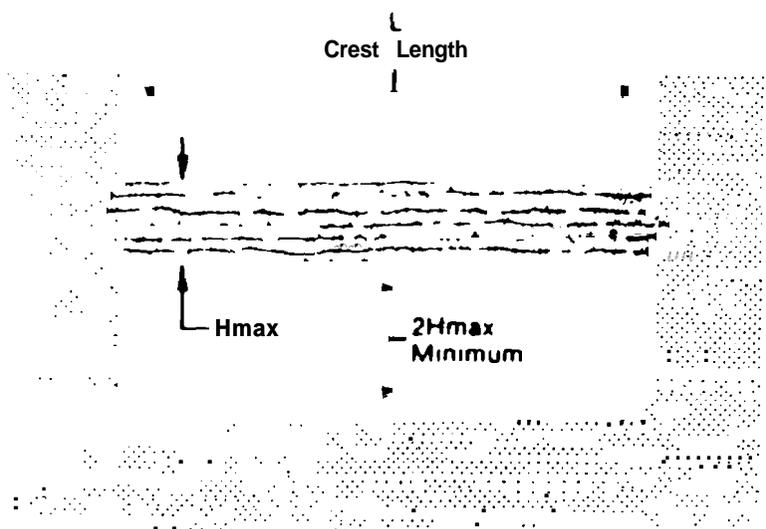
Figure 3-6 presents a sketch of the **Parshall** Flume, identifying its different parts. In the **Parshall** Flume, the floor level of the converging section is higher than the floor of the throat and diverging section. The Flume operates on the principle that when water flows through a constriction in the channel, a hydraulic head is produced that is proportional to the flow. **Flumes** are good for measuring open channel waste flow because they are self-cleaning. Sand and suspended solids are unlikely to affect the device’s operation.

A **Palmer-Bowlus** Flume, which may or may not have a constriction, has a level floor in the throat section and is placed in a pipe for approximately the length of the pipe’s diameter. The depth of water above the raised step in the throat is related to the discharge rate. The head should be measured at a distance  $(d/2)$  upstream of the throat, where (d) is the size (width) of the flume. The height of the step is usually unknown until the manufacturer’s data **are** consulted, and it is difficult to **measure** manually the height of water above the step at an upstream point. The dimensions of each **Palmer-Bowlus** Flume are different. Therefore, the manufacturer’s data must be consulted to establish a relationship between the head and the discharge rate. Figure 3-7 contains a sketch of a free-flowing **Palmer-Bowlus** Flume.

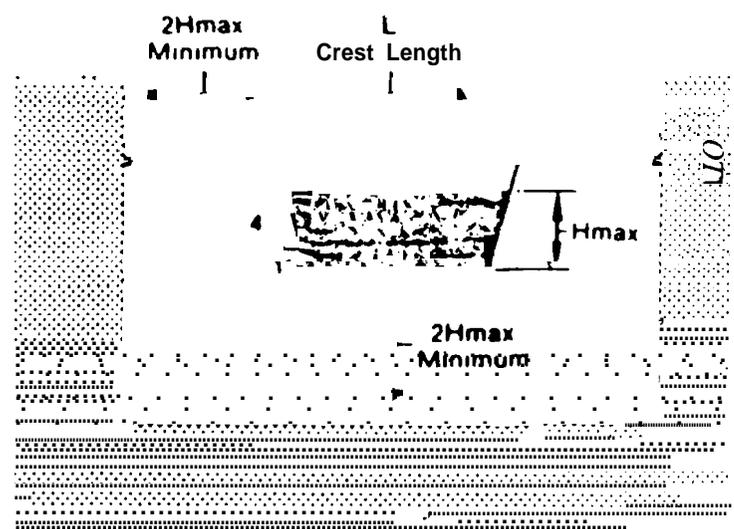
#### **Secondary** Devices:

Secondary devices are used in conjunction with primary devices to determine the actual flow passing the measuring point. Typically, secondary devices measure the depth of water in the primary device and convert

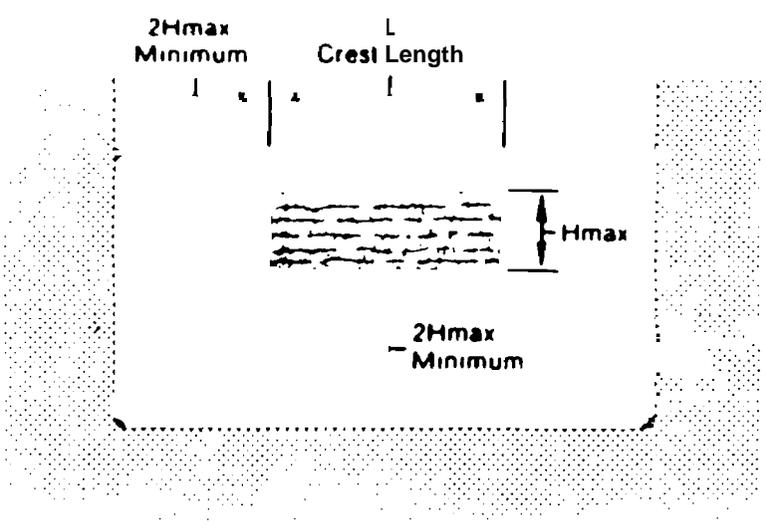




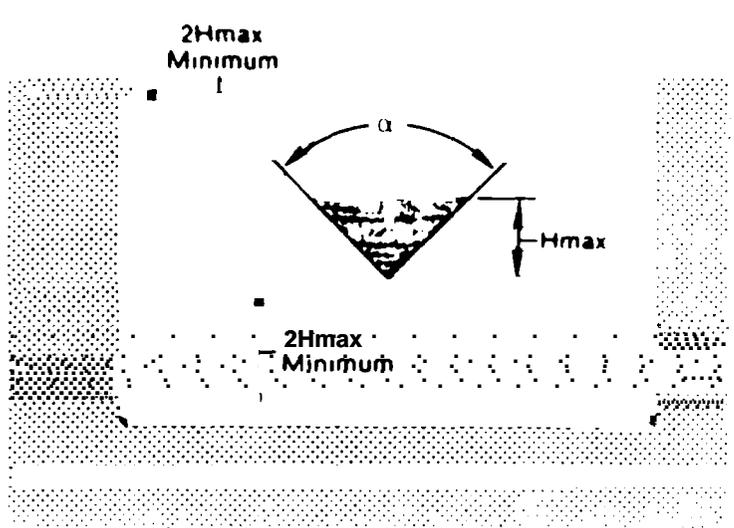
Suppressed (Without End Contractions) Rectangular Weir



Trapezoidal (Cipolletti) Sharp-Crested Weir



Contracted (With end Contractions) Rectangular Weir



V-Notch (Triangular) Sharp-Crested Weir

Figure 3-5

Four Common Types of Sharp-Crested Weirs  
(Taken from NPDES Compliance Inspection Manual, EPA, May 1988) 91

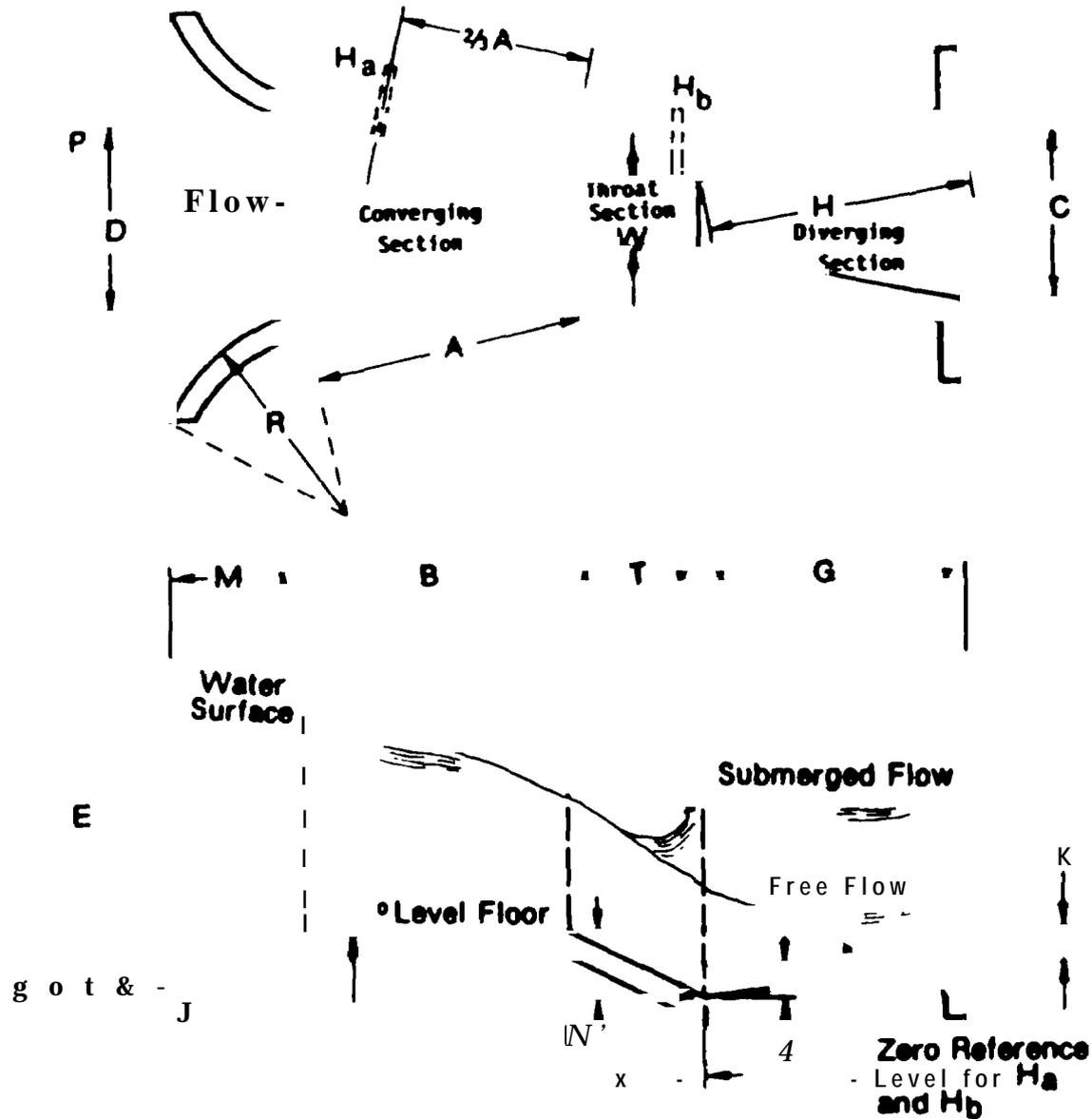
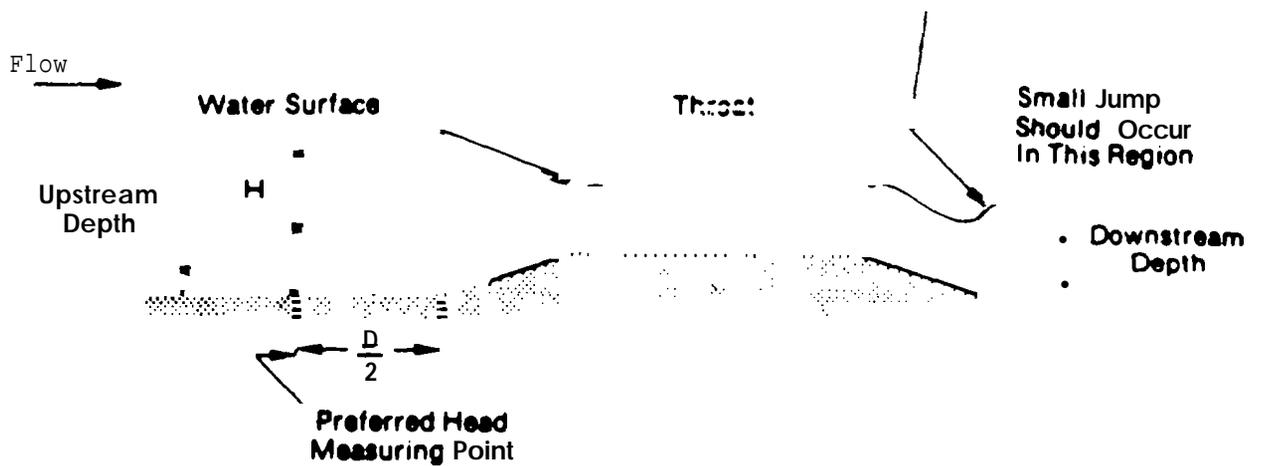


Figure 3-6

Plan View and Cross Section of a Parshall Flume  
 (Taken from NPDES Compliance Inspection Manual, EPA, May, 1988)

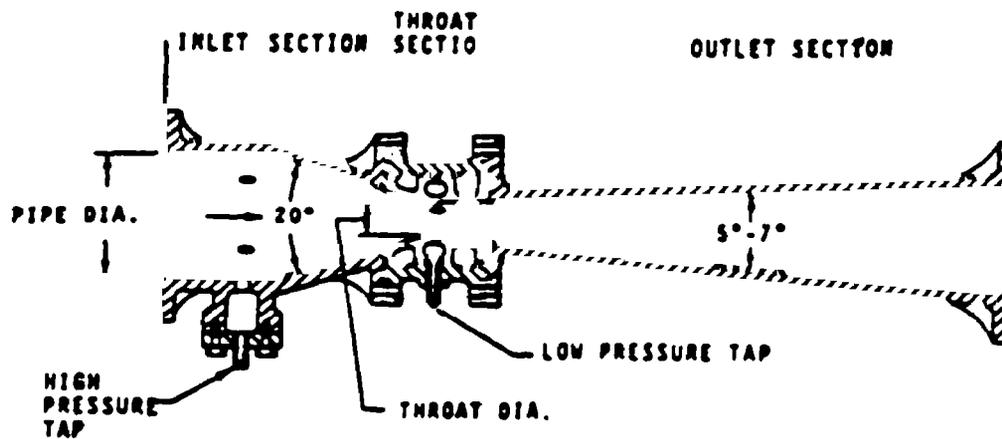


**D = Conduit Diameter**

**Figure 3-7**

**Free-Flowing Palmer-Bowlus Flume**

(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)



**Figure 3-8**

**Configuration and Nomenclature of a Venturi Meter**

(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)

the depth measurement to a corresponding flow, using established mathematical formulas. The output of the **secondary** device is generally transmitted to a recorder **and/or totalizer** to provide instantaneous and historical flow data to the operator. Outputs may also be transmitted to sampling systems to facilitate flow proportioning. Secondary devices can be organized into two broad categories:

- A non-recording type with direct readout (e.g., a staff gauge) or indirect readout from **fixed** points (e.g., a chain, wire weight, or float); or
- A recording type with either digital or graphic recorders (e.g., float in well, float in flow, bubbler, electrical, or acoustic).

### ***Closed Channel Flow***

Closed channel flow is normally encountered between treatment units in a wastewater treatment plant and after lift stations, where liquids and/or sludges are pumped under pressure. It is also encountered in submerged **outfalls**. Flow in closed channels is usually measured by a metering device inserted into the conduit. Examples of closed channel flow measuring devices are the Venturi Meter and the electromagnetic flow meter.

The Venturi Meter is one of the most accurate primary devices for measuring flow in closed channels. It is basically a pipe segment consisting of an inlet with a converging section, a throat, and a diverging outlet section, as illustrated in Figure 3-8. The water velocity is increased in the constricted portion of the inlet section which results in a decrease in static pressure. **The** pressure difference between the inlet pipe and the throat is proportional to the flow.

**Electromagnetic** flow meter operation is based on the fact that the voltage induced by a conductor moving at right angles through a magnetic field will be proportional to the velocity of that conductor as it moves through the field. In the case of the electromagnetic flow meter, the conductor is the stream of water to be measured, and the magnetic field is produced by a set of electromagnetic coils. A typical electromagnetic flow meter is shown in Figure 3-9.

### ***QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROCEDURES***

**Quality** Assurance and Quality Control are tools which are necessary to maintain a specified level of quality in the measurement, documentation, and interpretation of sampling data. To produce evidence which is admissible in an enforcement action, QA and QC procedures are necessary both in the **field** (during sampling) and in the laboratory. The QA/QC procedures used in the field are separate from those used in the laboratory, but both are crucial for obtaining reliable data. Both laboratory and field QA/QC are discussed in this section. QA/QC procedures are used to obtain data that are both precise (degree of closeness between

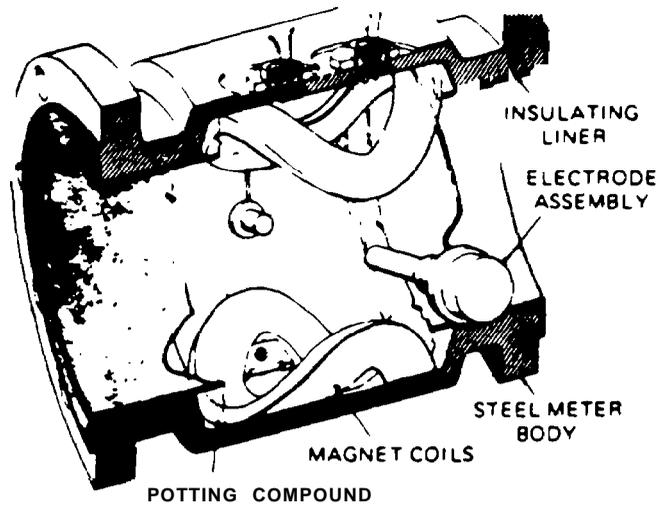


Figure 3-9

**Electromagnetic Flow Meter**

(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)

two or more samples) and accurate (degree of closeness between the results obtained from the sample analysis and the true value that should have been obtained). By following QA/QC procedures, the POTW's confidence in the validity of the reported analytical data is increased.

All data generated or used by the POTW must be of known, defensible, and verifiable quality. This includes data which are generated through self-monitoring at the industrial facility. Therefore, the IU should also have QA/QC procedures in place to ensure the adequacy of the data submitted as part of its periodic compliance report (NOTE: All inspections, and the data obtained as a result of the inspection, have the potential to be used in an enforcement proceeding and should be treated as potential evidence to be admitted in court).

QA is the program functions specified to assure the quality of measurement data while QC is the process of carrying out those procedures stated in the QA program. The QA program should be general while QC activities are specific. Also, the specific QC procedures used to assure data of good quality should be specified in the Quality Assurance and Sampling Plan developed for individual sampling events (e.g., the use of duplicate samples in the field). A QA program has two primary functions. First, it is designed to *monitor and evaluate* continuously the reliability (i.e., accuracy and precision) of the analytical results reported by each industrial user. This is how the quality of the data received from the IU is judged for acceptability. Second, QA should *control* the quality of the data to meet the program requirements. A QC program is designed to ensure the routine application of procedures necessary for the measurement process to meet prescribed standards of performance (e.g., through instrument calibration and analysis of reference unknowns). A program describing the schedule for calibration is QA, while the actual calibration procedures are QC.

QA/QC functions fit into two categories, field procedures and laboratory procedures. Each of these items is discussed in greater detail in the subsections which follow.

### ***Quality Assurance Procedures for Sampling***

A QA program for sampling equipment and for field measurement procedures (for such parameters as temperature, DO, pH, and conductivity) is necessary to ensure data of the highest quality. The inspector should recognize the importance of implementing quality assurance in sample collection to minimize such common errors as improper sampling methodology, poor sample preservation, and lack of adequate mixing during compositing and testing. Again, each of these activities should be a part of the POTW's SOP, so that all POTW sampling personnel are familiar with the proper sampling procedures. Quality assurance checks will help the inspector determine when sample collection techniques are inadequate for the intended use of the

data. A field quality assurance program should contain the following elements:

- The required analytical methodology for each regulated pollutant; special sample handling procedures; and the precision, accuracy, and detection limits of all required analytical methods.
- The basis for selecting the analytical and sampling method. For example, each analytical method should consist of approved procedures. Where the method does not exist, the QA plan should state how the new method will be documented, justified, and approved for use.
- The number of analyses for QC (e.g., the percentage of spikes, blanks, or duplicates), expressed as a percentage of the overall analyses, (e.g., one duplicate sample per 10 samples) to assess data validity. Generally, the QA program should approximate 15 percent of the overall program, with 10 percent and 5 percent assigned to laboratory QC and field QC respectively. The QA plan should include **shifting** these allocations or decreasing these allocations depending on the degree of confidence established for collected data.
- Procedures to calibrate and maintain field instruments and automatic samplers.
- A performance evaluation system which allows sampling personnel to cover the following areas:
  - Qualifications of personnel for a particular sampling situation;
  - Determining the best representative sampling site;
  - Sampling techniques, including the location of sampling points within the wastestream, the choice of grab or composite samples, the type of automatic sampler, special handling procedures, sample preservation procedures, and sample identification.
  - Flow measurement, where applicable.
  - Completeness of data, data records, processing, and reporting.
  - Calibration and maintenance of field instruments and equipment.
  - Use of QC samples, such as field duplicates, or splits to assess the validity of the data.
  - Training of all personnel involved in any function affecting data quality.

By following these QA procedures, the inspector can ensure the proper quality data from the industrial user.

### **Quality Control Procedures for Sampling**

Sampling QC begins with calibration and preventative maintenance procedures for sampling equipment. The inspector should prepare a calibration plan and documentation record for all **field** sampling and analysis equipment. A complete document record should be kept in a QC logbook, including equipment specifications, calibration date, and calibration expiration data, and maintenance due date. All of these activities should be reflected in the POTW's Standard Operating Procedures (see the beginning of the chapter for a discussion of SOPs). The sampler should keep in mind that field analytical equipment should be recalibrated in the field prior to taking the sample. In addition to calibration procedures, the person conducting field sampling should complete the various types of QC samples outlined on page 77.

Personnel conducting sampling should be well-trained in the use, cleaning, calibration, and maintenance of all instruments or samplers used. Automatic sampler tygon tubing, bottles, and the sampler itself should be cleaned prior to each sampling event. Automatic samplers should be calibrated for sample quantity, line purge, and the timing factor, if applicable. This calibration can and should be checked in the field to verify

draw. The manufacturer's directions should be reviewed and followed for cleaning and calibrating all equipment.

### **Laboratory Quality Assurance/Quality Control**

Laboratory QA/QC procedures ensure high-quality analyses through instrument calibration and the processing of control samples. The precision of laboratory findings refers to the reproducibility of results. In a laboratory QC program, a sample is analyzed independently (more than once) using the same methods and set of conditions. Precision is estimated by comparing the measurements. Accuracy refers to the degree of difference between **observed** values and known or actual values. The accuracy of a method may be determined by **analyses** of samples to which known amounts of a reference standard have been added,

Four specific laboratory QA procedures can be used to determine the confidence in the validity of the reported analytical data: duplicates, blanks, splits, and spiked samples. Each of these are described below:

- **Duplicate Samples (Laboratory):** Laboratory duplicate samples are samples which are received by the laboratory and divided (by the laboratory) into two **or** more portions. **Each** portion is **separately** and **identically** prepared and **analyzed**. Duplicate samples check for precision. These samples provide a check on sampling equipment and sampling techniques.
- **Method Blanks:** Method blanks are samples of analyte-free water that are prepared in the **laboratory** and analyzed by the analytical method used for field samples. The results from the analyses are used to check on the cleanliness of reagents, instruments, and the laboratory environment.
- **Split Samples (Field):** Field splits are collected and divided in the field into the necessary number of portions (e.g., 2, 3, etc) for analysis. Equally representative samples must be collected in this process, and then the samples are usually sent to different **analytical** laboratories. Field splits allow the comparison of analytical techniques and procedures from separate laboratories. Sampling personnel should exercise caution when splitting samples to avoid producing large differences in TSS. All widely divergent results should be investigated and the cause identified. **NOTE:** Oil and grease samples cannot be split due to the nature of the pollutant.
- **Laboratory Spiked Samples:** These samples provide a proficiency check for the accuracy of **analytical** procedures. A known amount of a particular constituent is added to separate aliquots of the sample or to distilled/deionized water at a concentration where the accuracy of the test method is satisfactory. **The** amount added should be coordinated with the laboratory.

Laboratory QA/QC procedures can be quite complex. Often, the analytical procedures specify QA/QC requirements for calibration, interference checks (for ICP analyses), control samples, spiking (including the method of standard additions), blank contaminant level, and instrument tuning. Accuracy is **normally** determined through the analysis of blanks, standards, blank spikes, laboratory control samples, and spiked samples. Precision is determined through the comparison of duplicate results or duplicate spiked results for organic analysis. For more information on laboratory QA/QC, the POTW should contact their Regional Quality Assurance Management Staff or Quality Assurance Manager.

The methods used by in-house or contract laboratories to analyze industrial **user** samples must be methods which are EPA approved under 40 CFR 136 and thus are acceptable to a court of law as the most reliable and accurate methods of analyzing water and **wastewater**. Although some field test kits are useful as indicators of current conditions (and, thus, may be used for process control considerations), they are not appropriate for sampling that is conducted to verify or determine compliance. If non-EPA approved methods are used for analysis, it is likely that the data will **be** considered inadmissible as evidence. When choosing a contract lab. **POTWs** should obtain and review a copy of the lab's **QA/QC** plan.

### ***Compliance Issues Related to Sampling and Analysis***

The compliance monitoring program of the **POTW** (i.e., the process of receiving **IU** self-monitoring reports, and conducting inspections and sampling at regulated industrial users) is a crucial aspect of the local pretreatment program. The compliance monitoring program forms the basis of the information the **POTW** must use to enforce the federal and local requirements established as part of the **POTW's** approved pretreatment program. The purpose of this manual has **been** to ensure the ability of the **POTW** to use the information gained in compliance monitoring (e.g., through proper chain-of-custody, sample **QA/QC**, and legal entry procedures). We now turn to certain specific issues of how the **POTW** should use this information to determine the compliance status of its industrial users **after** it has **been** appropriately gathered. For example, we have already discussed certain laboratory **QA/QC** measures. These measures are **necessary** to ensure the validity and reproducibility of the sample. **One** of these measures is the use of **duplicate** samples, but how should the **POTW** determine compliance from **these** samples if their results are different (e.g., one shows compliance and the other shows noncompliance)? This issue and others like it are addressed in this section of the manual. The **POTW** should follow the procedures and information outlined in this section when establishing the compliance status of their **IUs**.

#### ***The Use of Duplicate Samples to Evaluate Compliance***

In the previous section, the use of duplicate samples (both field and laboratory) was discussed as a means of determining if the sample collection and laboratory analyses are adequately precise for compliance determination. In most cases, if proper **QA/QC** procedures are followed, the analyses from the duplicates should be very close together. This indicates that the sample collection technique is **sufficiently** precise and that the lab has a high degree of precision in its analysis of samples. If the duplicate sample results are very close to one another (i.e., within the **QC** range established by the laboratory) but one is above the limit and the other is below the limit, the **POTW** should average these results together to determine the compliance status of the **IU**. Remember, this can only be done if the sample results are within the **QC** range of the

laboratory

In other cases, the analytical results from the duplicate samples, even though pulled from the same sample (or a simultaneous second sample), may yield significantly different analytical results. If this happens, the POTW needs to make a judgment as to whether the sample can be used for determining the compliance status of the IU (e.g., for determining SNC). If duplicate samples produce significantly different analytical results, the POTW should follow the procedures outlined below:

Investigate the **Analytic Methodology**. The POTW should review the procedures used by the laboratory personnel when analyzing the sample to ensure that all steps were followed properly. The POTW should also evaluate the nature of the samples themselves and whether the samples may be responsible for contributing to any analytical discrepancies (e.g., duplicate samples of very high concentration, i.e., requiring significant dilutions, may produce high relative percent differences which may be due to sampling techniques in the field, sampling of the aliquot in the laboratory, dilution technique, or a combination of these factors). **POTWs** must be aware of this situation when evaluating whether any duplicate sample results may be used for determining compliance.

- Check the **Analytic Time Sequence**. Very often the sample analysis for duplicate samples is done sequentially. This is especially true when the sample analysis requires the use of **GC/MS** equipment. **In** these situations, it is possible to have the sample analyses far apart in time. If this happens, the sample holding time may be exceeded. If this happens, the sample which was analyzed after the holding time is not valid, and a new sample must be taken. Even if the sample holding time has not been exceeded, it is possible that two analyses from the same sample will produce different results if they are far enough apart in time. Therefore, make sure that all duplicate samples are analyzed as close together as possible to ensure sample integrity through the entire analytic process. If the sample has not exceeded its allotted holding time, the POTW can re-analyze two new samples from the original sample collection vessel to obtain valid duplicate results.
- Check the **Laboratory Equipment**. The POTW should make sure that all glassware is properly washed and rinsed to make sure that it is clean and free of contaminants. Dirty glassware can cause interference with the sample analysis. In addition, make sure that all lab equipment is properly calibrated, operated and maintained. This should ensure consistent sample results.
- Review the **Sampling Methodology**. It is possible that a duplicate sample, if taken as two discrete samples, will have **very** different characteristics. For example, when taking a duplicate sample for oil and grease, it is **usually** necessary to take two discrete samples because it is not possible to split an **oil** and grease sample. When the two samples are taken, the sampler may not take each sample in exactly the same way (e.g., one sample may skim the top of the wastestream and the other may be taken from the bottom of the wastestream). This can produce two radically different samples, even though they were taken at the same time from the same place. If duplicate samples are taken from the same sample collection vessel, make sure that the sample is well mixed and homogeneous so that each sample is as close as possible to each other.
- Check the **Laboratory QA/QC**. The laboratory and sampling **QA/QC** procedures should be reviewed when duplicate samples produce different analytical results. The lab should check to see if blank and spike sample analyses give appropriate results. If the blanks **and/or** spikes do not produce expected values, it is highly likely that there is a problem with the analytical procedures. If the blanks and spikes indicate analytic problems, it may be necessary to discard the sample and disregard the results when determining the compliance status of the industrial user.

If the source of the discrepancy is identified, the POTW should run another analysis from the same

sample batch (this is one good reason to take an adequate sample amount when in the field) making sure to avoid the mistakes on the original duplicate sample.

The POTW also has the option of sending the sample in question to an independent laboratory. This “referee” laboratory can **serve** to give impartial analysis of the sample so that the sample results can be used to evaluate compliance. If the POTW chooses this option it should keep in mind that while the “referee?” laboratory will give independent results, it will not necessarily give the “right” result. The POTW should evaluate the referee lab in terms of the equivalency of its analytical procedures, **QA/QC** etc., in relation to 40 CFR 136 as well as equivalency to the **POTW's** and/or the **IU's** lab.

#### **Compliance with Monthly Average Limitations**

**POTWs** are faced with the issue of how to assess an appropriate penalty for various types of violations of the local pretreatment program. In evaluating this situation, the POTW should follow the reasoning of the courts when assessing the number of violations which accrue as a result of a violation of a monthly average. In *Chesapeake Bay Foundation v. Gwaltney of Smithfield Ltd.* 791 F.2d. 304 (4th Cir. 1986) a violation of the monthly average was deemed to be a violation of each of the days of the month (not necessarily operating days). The court reasoned that the language in the Clean Water Act “strongly suggests that where a violation is defined in terms of a time period longer than a day, the maximum penalty assessable for that violation should be defined in terms of the number of days in that time period.” (*Id* at 314). This means that if there is a violation of a monthly average, the largest penalty assessable is the maximum penalty authority of the POTW (which should be, at a minimum, 1,000 dollars per day) multiplied by the number of days in the month of violation. This is not necessarily the penalty which the POTW will impose on the IU, but it is the maximum amount which the POTW may legally assess.

There has been some confusion on the part of some **POTWs** as to how many samples are required to demonstrate a violation of a monthly average. At a minimum, the POTW needs only one valid sample from the month to assess compliance with the monthly average. If the POTW has only one sample from an IU in the six month reporting period and that sample is in violation of the monthly average, the maximum liability the IU faces for that effluent violation in that six month period is the maximum penalty authority of the POTW multiplied by the number of days in the month the sample was taken. This process should be used by the POTW when evaluating the appropriate penalty amount to assess in situations where the POTW's Enforcement Response Plan indicates the need to assess a penalty.

***Closed Cup Flashpoint Sampling and Compliance***

The General Pretreatment Regulations at 403.5(b)(1) require that no discharge to a POTW shall “create a fire or explosion hazard in the POTW.” The regulation further explains this requirement by setting a closed cup flashpoint limit of 140°F (60°C) on wastestreams discharged to the POTW. Since this prohibitive limit is an instantaneous limit, the POTW must use a grab sample to evaluate compliance with the closed cup **flashpoint** requirement.

The POTW should monitor the **IU's** wastestream periodically for the potential of creating a fire hazard, but this frequency should be based on the nature of the wastestream. If the POTW has reason to believe that the **IU** has a strong potential to create a fire hazard, monitoring for the closed cup flashpoint should **be** conducted regularly. If the **POTW** has no reason to believe that the **IU** poses a fire hazard, minimum closed cup flashpoint monitoring can be done. At a minimum, the POTW should evaluate the **IU's** potential to cause pass-through or interference, as well as the **IU's** potential to violate any of the prohibitive discharge limits, at least once every permit cycle (e.g., every **five** years if the **IU's** permit duration is five years long).

***Frequency of POTW Sampling In Lieu of industrial User Sampling***

The General Pretreatment Regulations allow for the POTW to take over the periodic sampling and analysis activities for the industrial user. When the POTW chooses to exercise this option, the **IU** is not required to submit the periodic sampling report required in 403.12(e). The General Pretreatment Regulations also require **POTWs** to “inspect and sample the **effluent** from each Significant Industrial User at least once a year.” (403.8(f)(2)(v)). The purpose of this inspection and sampling is to provide information, independent of the **IU**, on the compliance status of the **SIU**. If the POTW is already conducting the periodic sampling for the **IU**, however, the POTW is already evaluating compliance independent of the **IU**. Therefore, if the POTW is conducting the required sampling for the **IU** under 403.12, the POTW is satisfying the 403.8 requirement of annually sampling the **IU**. The POTW is still required to inspect each **SIU** annually, but the POTW would only be **required** to conduct two sampling events at the **IU**, as required by 403.12.

The General Pretreatment Regulations require **IUs** to notify the Control Authority and conduct follow-up sampling if the previous sample taken by the **IU** indicates a violation. The notification to the Control Authority must be conducted within 24 hours of becoming aware of the violation, and the results of resampling must be submitted to the Control Authority within 30 days. If the POTW is conducting the sampling for the **IU**, however, there is no regulatory requirement for the **POTW** to resample after detecting a violation. As a matter of policy, though, we strongly recommend that the POTW conduct the required repeat sampling, or require the **IU** to conduct the repeat sampling, because this provides further information on the

nature of the IU's impact on the treatment plant and may provide further support in an enforcement action taken by the POTW for **effluent** violations.

### *SNC in Situations With Multiple Outfalls*

**POTWs often** encounter situations where industrial users have multiple **outfalls** (i.e., connections to the collection system) from the same categorical process line, and the question has been raised of how the POTW should evaluate such situations for the purpose of determining the compliance status of the facility, especially with respect to SNC. **When** the POTW encounters a multiple outfall situation, compliance with applicable standards should be determined in the following manner.

Multiple outfall situations can arise in three ways: 1) multiple categorical operations with multiple outfalls, 2) a single categorical operation with multiple outfalls, and 3) a wastestream regulated by local limits with multiple outfalls. Each of these circumstances are discussed below. If an industrial user has several **outfalls** to the POTW from separate categorical operations, each of these **outfalls** and each pollutant parameter per outfall must be evaluated separately for the purpose of determining whether the facility meets the criteria for Significant Noncompliance. For example, if the IU has three **outfalls** from three separate categorical operations and **each** outfall is regulated for chromium, cadmium and zinc, and any of the data from each separate outfall exceeds either the chronic or TRC criterion, then the IU meets the criteria for SNC and should be published in the newspaper by the **POTW**. When evaluating the compliance status of the IU keep in mind that the IU must **be evaluated** on a **categorical** operation-by-categorical operation, **parameter-by-parameter**, and outfall-by-outfall basis. However, if the IU has more than one outfall *from the same categorical* operation (e.g., several lines from the same metal finishing operation), the POTW should treat those different categorical operations as a single, aggregate line for purposes of determining compliance. For example, if a metal finisher discharges categorical process wastewater generated from different categorical operations in the same process line through two different sewer connections (without any intermediate treatment), compliance with the categorical standard should be determined by using a flow weighted average of the two lines. Finally, the POTW should be aware of how to evaluate compliance in situations where local limits control the nature of the discharge and there is more than one discharge point to the POTW. If there is more than one discharge point to the POTW which is regulated by a local limit (even if the separate **outfalls** come from the same process line), then the facility must meet the local limit *at the end of each pipe*. Likewise, the federal prohibitive standards in 403.5 must be met *for each discharge point* to the POTW no matter where the discharge point is derived.

*Violation Date*

If a sample taken at an **IU** indicates a violation, the date of the violation is the date the sample was taken, not the date the sample was analyzed in the laboratory. For a long-term composite sample, the date of violation is the date the ~~sample was completed~~ a sampler is placed at an industrial user at SAM on Tuesday and picked up at 8AM on Wednesday (the following day), but the sampler stopped taking samples at 5PM Tuesday, the date of violation is 5PM Tuesday (not 8AM Wednesday). (**NOTE:** In this example, the required holding time for this sample would commence at 8AM on Tuesday). This can be important when the POTW goes to apply the rolling quarters method for determining SNC (as outlined in the EPA Memorandum "Application and Use of the Regulatory Definition of Significant Noncompliance for Industrial Users," September 9, 1991). When tracking the compliance status of each **IU**, the **POTW** should have an automated system which tracks the date each sample was taken at each **IU** regulated by the **POTW**.

*Compliance With Continuous **Monitoring of pH***

The question of how to determine compliance with a **pH** limit when there is continuous monitoring has **often** been raised by **POTWs**. The EPA has formally responded to this question in a letter to the State of New Jersey. This letter is provided in Appendix VIII for your reference. The underlying concern of how to determine compliance with continuous monitoring centers on the length of time allowed for **pH** to be out of compliance with the limit. **POTWs** have been placed in the situation of having to determine what is an acceptable amount of time to be out of compliance under a continuous monitoring scheme, and many **POTW** have turned to the federal regulations at 401.17 to provide the answer. **Part 401 .17** addresses the allowed excursions for **pH** when applied to facilities which discharge directly to a receiving water. The question has been asked whether or not this same excursion policy should be adopted for facilities which discharge to the sanitary sewer. In general, **POTWs** may apply an excursion policy (although not necessarily the one found at 40 CFR 401.17), with three important restrictions. First, the **POTW** may not allow an industrial user to discharge waste below a **pH** of 5 (**403.5(b)(2)**) unless the **POTW** is specifically designed to accommodate such waste. Second, a **POTW** may not grant a variance from a categorical standard for an upper **pH** limit, if such an upper limit exists in the standard. Finally, a **POTW** may not grant a waiver from a local **pH** limit if the waiver would cause pass through or interference at the plant. If the **POTW** observes these restrictions, it can establish a policy of allowing the **pH** discharge of a facility to go outside the range established in the limit without the facility incurring any enforcement liability. **If** a **POTW** establishes a policy allowing **pH** discharges outside the established range, the **POTW** should document such a policy in the **POTW's** Enforcement Response Plan, Sewer Use Ordinance, or **IU** permit to ensure the enforceability of the policy.

## ***Summary***

This chapter discussed the procedures and protocols which should be used when sampling the effluent from industrial users or when observing a permittee's self-monitoring procedures. The need for a sampling plan and for coordinating with the laboratory performing the analyses were stressed in order to promote consistency between samplers and to ensure that laboratory requirements are met during all sampling events. The chapter also emphasized the importance of using proper sample collection techniques, including the selection of an appropriate sample location and sample type, the preparation of sample containers, and the preservation, labeling, and handling of samples **after** collection to establish the validity of each sample result should violations be identified that lead to **an** enforcement action. The chapter further explained several instances in which special sampling requirements must be followed. Finally, this chapter described various chain-of-custody and quality assurance procedures that should be practiced during all sampling events to ensure the accuracy, integrity, and reliability of each sample and of the corresponding **analytical** results. Inspectors must conduct all sampling activities on the premise that each may lead to an enforcement action.

**Table 3-3**  
**Volume of Sample Required for Analyzing Various Industrial Pollutants**  
*(Associated Water and Air Resource Engineers, Inc., 1973*  
**Handbook for Monitoring Industrial Wastewater. U.S. EPA Technology Transfer**

<i>Analytical Tests</i>	<i>Volume of Sample<sup>1</sup> (ml)</i>
<b>PHYSICAL</b>	
Color and Odor <sup>2</sup> .....	100 to 500
Corrosivity <sup>2</sup> .....	flowing sample
Electrical Conductivity' .....	100
pH, electrometric' .....	100
Radioactivity' .....	100 to 1,000
Specific gravity' .....	loo
Temperature' .....	flowing sample
Toxicity' .....	1,000 to 20,000
Turbidity' .....	100 to 1,000
<b>CHEMICAL</b>	
Dissolved Gases:	
Ammonia', NH <sub>3</sub> ' .....	500
Carbon Dioxide' .....	200
Chlorine' ..	200
Hydrogen <sup>1</sup> .....	1,000
Hydrogen Sulfide' .....	500
Oxygen' .....	500 to 1,000
Sulfur dioxide' .....	100
Miscellaneous	
Acidity and alkalinity .....	loo
Bacteria, iron .....	500
Bacteria, sulfate reducing .....	100
Biochemical Oxygen Demand (BOD) .....	100 to 500
Chemical Oxygen Demand (COD) .....	50 to 100
Chloroform extractable matter .....	1,000
Detergents .....	100 to 200
Hardness .....	50 to 100
Hydrazine .....	50 to loo
Microorganisms .....	100 to 200
Volatile and filming amines .....	500 to 1,000
Oil and Grease .....	3,000 to 5,000
Organic Nitrogen .....	500 to 1,000
Phenolic compounds .....	800 to 4,000
pH, colorimetric .....	10 to 20
Polyphosphates .....	loo to 200
Silica .....	50 to 1,000
Solids, dissolved .....	100 to 20,000
Solids, suspended .....	50 to 1,000
Tannin and Lignin .....	loo to 200

Table 3-3

**Volume of Sample Required for Analyzing Various Industrial Pollutants (cont.)**

<i>Anal&amp;al Tests:</i>	<i>Volume of Sample (ml)</i>
Cations:	
Aluminum, Al <sup>+++</sup> .....	100 to 1,000
Ammonium <sup>+</sup> , NH <sub>4</sub> <sup>+</sup> .....	500
Antimony, Sb <sup>+++</sup> to Sb <sup>++++</sup> .....	100 to 1,000
Arsenic, As <sup>+++</sup> to As <sup>++++</sup> .....	100 to 1,000
Barium, Ba <sup>++</sup> .....	100 to 1,000
Cadmium, Cd* .....	100 to 1,000
Calcium, Ca <sup>++</sup> .....	100 to 1,000
Chromium, Cr <sup>+++</sup> to Cr <sup>++++</sup> .....	100 to 1,000
Copper, Cu <sup>++</sup> .....	200 to 4,000
Iron <sup>+</sup> , Fe <sup>++</sup> to Fe <sup>+++</sup> .....	100 to 1,000
Magnesium, Mg <sup>++</sup> .....	100 to 4,000
Manganese, Mn <sup>++</sup> to Mn <sup>++++</sup> .....	100 to 1,000
Mercury, Hg <sup>+</sup> to Hg <sup>++</sup> .....	100 to 1,000
Potassium, K <sup>+</sup> .....	100 to 1,000
Nickel, Ni <sup>++</sup> .....	100 to 1,000
Silver, Ag <sup>+</sup> .....	100 to 1,000
Sodium, Na <sup>+</sup> .....	100 to 1,000
Strontium, Sr <sup>++</sup> .....	100 to 1,000
Tin, Sn <sup>+++</sup> to Sn <sup>++++</sup> .....	100 to 1,000
Zinc, Zn <sup>++</sup> .....	100 to 1,000
Anions:	
Bicarbonate, HCO <sub>3</sub> <sup>-</sup> .....	100 to 200
Bromide, Br <sup>-</sup> .....	100
Carbonate, CO <sub>3</sub> <sup>-</sup> .....	100 to 200
Chlorine, Cl <sup>-</sup> .....	25 to 100
Cyanide, Cn <sup>-</sup> .....	25 to 100
Fluoride, F <sup>-</sup> .....	200
Hydroxide, OH <sup>-</sup> .....	50 to 100
Iodide, I <sup>-</sup> .....	100
Nitrate, NO <sub>3</sub> <sup>-</sup> .....	10 to 100
Nitrite, NO <sub>2</sub> <sup>-</sup> .....	50 to 100
Phosphorous, ortho, PO <sub>4</sub> <sup>-</sup> , HPO <sub>4</sub> <sup>-</sup> , H <sub>2</sub> PO <sub>4</sub> <sup>-</sup> .....	50 to 100
Sulfate, SO <sub>4</sub> <sup>-</sup> , HSO <sub>4</sub> <sup>-</sup> .....	100 to 1,000
Sulfide, S <sup>-</sup> , HS <sup>-</sup> .....	100 to 500
Sulfite, SO <sub>3</sub> <sup>-</sup> , HSO <sub>3</sub> <sup>-</sup> .....	50 to 100

<sup>1</sup> Volumes specified in this table should be considered as guides for the approximate quantity of sample necessary for a particular analysis. The exact quantity used should be consistent with the volume prescribed in the standard method of analysis, whenever a volume is specified.

<sup>2</sup> Aliquot may be used for other determinations.

<sup>3</sup> Samples for unstable constituents must be obtained in separate containers, preserved as prescribed, completely filled, and sealed against all exposure.

Table 3-4

**Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR PM 136 Tab& I and II)**

<b>Pollutant Parameter</b>	<b>Container<sup>(1)</sup></b>	<b>Preservative<sup>(2,3)</sup></b>	<b>Maximum Holding Time<sup>(4)</sup></b>	<b>EPA Tar Method (1979) (unless otherwise noted)<sup>(10)</sup></b>
<b>BACTERIAL TESTS</b>				
Colifonn, Fecal and Total	P,G	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(6)</sup>	6 Hours	Standard Methods 15th Ed.: 908
Fecal Streptococci	P,G	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(6)</sup>	6 Hours	Standard Methods 15th Ed.: 910
<b>INORGANIC TESTS</b>				
Acidity	P,G	Cool to 4°C	14 days	305.1
Alkalinity	P,G	Cool to 4°C	14 days	310.1
Ammonia	P,G	Cool to 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	350
BOD	P,G	Cool to 4°C	48 Hours	405. I
BOD. carbonaceous	P,G	Cool to 4°C	48 Hours	Standard Methods 16th Ed.:507(5.e.6)
Bromide	P,G	None required	28 Hours	320. I
Chemical Oxygen Demand	P,G	Cool to 4°C HSO, to pH<2	28 Hours	410
Chloride	P,G	None required	28 days	325
Chlorine, total residual	P,G	None required	Analyze immediately	330
Color	P,G	Cool to 4°C	48 Hours	110
Cyanide, total and amenable to chlorination	P,G	Cool to 4°C NaOH to pH>12 0.6g ascorbic acid <sup>***</sup>	14 days <sup>(6)</sup>	335
Fluoride	P	None required	28 days	340
Hardness	P,G	HNO, to pH<2, H <sub>2</sub> SO <sub>4</sub> to pH<2	6 months	130

**Table 3-4 (Cont)**

**Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR Part 136 Table I and II)**

<i>Parameter</i>	<i>Container<sup>(1)</sup></i>	<i>Preservative<sup>(2,3)</sup></i>	<i>Maximum Holding Time<sup>(4)</sup></i>	<i>EPA Test Method (1979) (unless otherwise noted)</i>
Hydrogen Ion (pH)	P,G	None required	Analyze immediately	150.1
TKN, organic nitrogen	P,G	Cool to 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	351
METAL!?"				
Hexavalent Chromium	P,G	Cool to 4°C	24 Hours	218.4
Mercury	P,G	HNO <sub>3</sub> to pH<2	28 days	245
Metals. (except above)	P,G	HNO <sub>3</sub> to pH<2	6 months	(Various methods)
Nitrate	P,G	Cool to 4°C	48 Hours	352.1
Nitrate-nitrite	P,G	Cool to 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	353
Nitrite	P,G	Cool to 4°C	48 Hours	354
Oil and Grease	G	Cool to 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	413
Organic Carbon	P,G	Cool to 4°C. HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	415
Orthophosphate Phosphorous	P,G	Filter immediately Cool to 4°C	48 Hours	365
Dissolved Oxygen Probe	G bottle/top	None required	Immed. analysis	360
Winkler	G bottle/top	Fix on-site and store in the dark	8 Hours	
Phenols	G	Cool to 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	24 Hours	420
Phosphorous (elemental)	G	Cool to 4°C	48 Hours	Note <sup>(16)</sup>

**Table 3-4 (Cont.)**

**Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR Part 136 Table I and II)**

<b>Parameter</b>	<b>Container<sup>1,2</sup></b>	<b>Preservative<sup>1,3</sup></b>	<b>Maximum Holding Time<sup>4</sup></b>	<b>EPA Test Method (1979) (unless otherwise noted)</b>
Phos. (total dissolved)	P,G	Cool to 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days	365
Residue, total	P,G	Cool to 4°C	7 days	160.3
Residue, filterable	P,G	Cool to 4°C	7 days	160.1
Residue, nonfilterable (TSS)	P,G	Cool to 4°C	7 days	160.2
Residue, settleable	P,G	Cool to 4°C	48 Hours	160.5
Residue, volatile	P,G	Cool to 4°C	7 days	160.4
Silica	P	Cool to 4°C	28 days	370.1
Specific conductance	P,G	Cool to 4°C	28 days	120.1
Sulfate	P,G	Cool to 4°C	28 days	375
Sulfide	P,G	Cool to 4°C add zinc acetate plus NaOH to pH>9	7 days	376
Sulfite	P,G	None required	Analyze immediately	377
<b>Surfactants</b>	P,G	Cool to 4°C	48 Hours	425.1
Temperature	P,G	None required	Analyze immediately	170.1
Turbidity	P,G	Cool to 4°C	48 Hours	180.1
<b>ORGANIC TESTS<sup>1,2</sup></b>				
<b>Purgeable</b> halocarbons	G, teflon lined septum	Cool to 4°C 0.008Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>1,5</sup>	14 days	601 (40 CFR 136 Appendix A, 1984)

**Table 3-I (Cont.)**

**Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR Part 136 Table I and II)**

<i>Parameter</i>	<i>Container<sup>(1)</sup></i>	<i>Preservative<sup>(2,3)</sup></i>	<i>Maximum Hold Time<sup>(4)</sup></i>	<i>EPA Method (1979) unless otherwise noted</i>
<b>Purgeable Aromatic Hydrocarbons</b>	G, teflon-lined septum	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup> HCl to pH 2 <sup>(9)</sup>	14 days	601 (40 CFR 136 Appendix A, 1984)
Acrolein and acrylonitrile	G, teflon-lined septum	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup> Adjust pH/4.5 <sup>(10)</sup>	14 days	602 (40 CFR 136 Appendix A, 1984)
Phenols	G, teflon-lined cap	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction.	603 (40 CFR 136 Appendix A, 1984)
Benzidines <sup>' 11)</sup>	G, teflon-lined cap	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction	604 (40 CFR 136 Appendix A, 1984)
Phthalate esters <sup>' 13)</sup>	G, teflon-lined cap	Cool to 4°C	7 days until extraction; 40 days after extraction	605 (40 CFR 136 Appendix A, 1984)
<b>Nitrosamines<sup>(11 14)</sup></b>	G, teflon-lined cap	Cool to 4°C Store in the dark 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction	606 (40 CFR 136 Appendix A, 1984)
Polychlorinated Biphenyls <sup>(17)</sup>	G, teflon-lined cap	Cool to 4°C	7 days until extraction; 40 days after extraction	607 (40 CFR 136 Appendix A, 1984)
Nitroaromatics and isophorone <sup>' 11)</sup>	G, teflon-lined cap	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup> Store in the dark	7 days until extraction; 40 days after extraction	608 (40 CFR 136 Appendix A, 1984)
<b>Polynuclear aromatic hydrocarbons<sup>(11)</sup></b>	G, teflon-lined cap	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup> Store in the dark	7 days until extraction; 40 days after extraction	609 (40 CFR 136 Appendix A, 1984)

**Tab&? 3-4(Cont.)**

**Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR Part 136 Tab& I and II)**

<i>Parameter</i>	<i>Container</i>	<i>Preservative</i> <sup>(2,3)</sup>	<i>Maximum Hold Time</i> <sup>(4)</sup>	<i>EPA Method (1979) (unless otherwise noted)</i>
<b>ORGANIC TESTS (cont.)</b>				
Haloethers	G, teflon-lined cap	Cool to 4°C	7 days until extraction; 40 days after extraction	611 (40 CFR I36 Appendix A, 1984)
Chlorinated Hydrocarbons <sup>(11)</sup>	G, teflon-lined cap	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction	612 (40 CFR I36 Appendix A, 1984)
TCDD (2,3,7,8-Tetrachlorodibenzo-p-dioxin)	G, teflon-lined cap	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction	613 (40 CFR I36 Appendix A, 1984)
<b>PESTICIDES TESTS</b>				
Organochlorine pesticides	G, teflon-lined cap	Cool to 4°C pH 5-9 <sup>(15)</sup>	7 days until extraction; 40 days after extraction	608 (40 CFR I36 Appendix A, 1984)
<b>RADIOLOGICAL TEST</b>				
Alpha, beta, and radium	P,G	HNO <sub>3</sub> to pH<2	6 months	Meth. 900-903 <sup>(17)</sup>

**KEY**

- (1) Polyethylene (P) or Glass (G)
- (2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- (3) When any sample is to be shipped by common carrier or sent through the United States mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the

Table 3-4 (Conk)

**Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR Part 136 Table I and II)**

preservation requirements of this table, the Office of Hazardous Materials. Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following **materials**: hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (**pH** about 1.96 or greater); nitric acid ( $\text{HNO}_3$ ) in water solutions at concentrations of 0.15% by weight or less (**pH** about 1.62 or greater); sulfuric acid ( $\text{H}_2\text{SO}_4$ ) in water solutions at concentrations of 0.35% by weight or less (**pH** about 1.15 or greater); and sodium hydroxide (NaOH) in water solutions at concentrations of 0.80% by weight or less (**pH** about 12.3 or less).

- (4) Samples should be analyzed as soon as possible **after** collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or laboratory, has data on file to show that the specific types of samples under study are stable for the longer time and has received a variance from the Regional Administrator under §136.3(e). Some samples may not **be** stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter period of time if knowledge exists to show this is necessary to maintain sample stability and integrity.
- (5) Should only be used in the presence of residual chlorine.
- (6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead **acetate** paper before **pH** adjustments **to** determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. **The** sample is filtered, then **NaOH** is added to raise the **pH** to 12.
- (7) Samples should be filtered immediately on-site before adding preservative for dissolved metals.
- (8) Guidance applies to samples to be analyzed by GC, LC, GC/MS for **specific** organic compounds.
- (9) Sample receiving no **pH** adjustment must be analyzed within 7 days of sampling.
- (10) **The pH** adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no **pH** adjustment must be analyzed within 3 days of sampling.
- (11) When extractable analytes of concern fall within a single chemical category, the specified **preservation** and maximum holding times should be observed for optimum safeguarding of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved **by** cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the **pH** to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days **after** extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote (5) (re: the requirement for thiosulfate reduction of residual chlorine and footnotes (12), (13) (re: the analysis of benzidine).
- (12) If 1,2-diphenylhydrazine is likely to be present, adjust the **pH** of the sample to 4.0 ±0.2 to prevent rearrangement of the benzidine.
- (13) Extracts **may** be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

**Table 3-4 (Cont.)**

**Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Except from 40 CFR Part 136 Table I and II)**

- (14) For the analysis of diphenylnitrosamine, add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$  and adjust pH to 7-10 with NaOH within 24 hours of sampling
- (15) The pH adjustment may be performed upon receipt of the sample at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$ .
- (16) K.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorous by Gas-Liquid Chromatography," Journal of **Chromatography**, 47 (3): 421-426, 1970.
- (17) Reference: "Prescribed Procedures for Measurement of Radioactivity in Drinking Water" EPA-600/4-80-032 (1980 Update) U.S. EPA, August, 1980.
- (18) **This** list does not represent an exhaustive list of all approved test methods. When determining the appropriate analytical method, always refer to 40 CFR Part 136. **NOTE:** See **Appendix XIII** for details on 40 CFR Part 136.

**Table 3-4 (Cont.)**

**Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR Part 136 Table I and II)**

<i>Parameter</i>	<i>Container<sup>(1)</sup></i>	<i>Preservative<sup>(2,3)</sup></i>	<i>Maximum Hold Time<sup>(4)</sup></i>	<i>EPA Method (1979) (unless otherwise noted)</i>
<b>ORGANIC TESTS (cont.)<sup>(11)</sup></b>				
Haloethers <sup>(11)</sup>	G, teflon-lined cap	Cool to 4°C	7 days until extraction; 40 days after extraction	611 (40 CFR 136 Appendix A, 1984)
Chlorinated Hydrocarbons <sup>(11)</sup>	G, teflon-lined cap	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction	612 (40 CFR 136 Appendix A, 1984)
TCDD (2,3,7,8-Tetrachlordibenzo-p-dioxin) <sup>(11)</sup>	G, teflon-lined cap	Cool to 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> <sup>(5)</sup>	7 days until extraction; 40 days after extraction	613 (40 CFR 136 Appendix A, 1984)
<b>PESTICIDES TESTS</b>				
Organochlorine pesticides <sup>(11)</sup>	G, teflon-lined cap	Cool to 4°C pH 5-9 <sup>(15)</sup>	7 days until extraction; 40 days after extraction	608 (40 CFR 136 Appendix A, 1984)
<b>RADIOLOGICAL TEST</b>				
Alpha, beta, and radium	P,G	HNO <sub>3</sub> to pH<2	6 months	Meth. 900-903 <sup>(17)</sup>

**KEY**

- (1) Polyethylene (P) or Glass (G)
- (2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- (3) When any sample is to be shipped by common carrier or sent through the United States mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the

**Table 3-4 (Cont.)****Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR Part 136 Table I and II)**

preservation requirements of this table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and sodium hydroxide (NaOH) in water solutions at concentrations of 0.80% by weight or less (pH about 12.3 or less).

- (4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or laboratory, has data on file to show that the specific types of samples under study are stable for the longer time and has received a variance from the Regional Administrator under §136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter period of time if knowledge exists to show this is necessary to maintain sample stability and integrity.
- (5) Should only be used in the presence of residual chlorine.
- (6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered, then NaOH is added to raise the pH to 12.
- (7) Samples should be filtered immediately on-site before adding preservative for dissolved metals.
- (8) Guidance applies to samples to be analyzed by GC, LC, GC/MS for specific organic compounds.
- (9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
- (10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- (11) When extractable analytes of concern fall within a single chemical category, the specified preservation and maximum holding times should be observed for optimum safeguarding of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote (5) (re: the requirement for thiosulfate reduction of residual chlorine and footnotes (12), (13) (re: the analysis of benzidine).
- (12) If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ±0.2 to prevent rearrangement of the benzidine.
- (13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

**Table 3-4 (Cont.)****Required Containers, Preservation Techniques, Holding Times, and Test Methods  
(Excerpt from 40 CFR Part 136 Table I and II)**

- (14) For the analysis of diphenylnitrosamine, add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$  and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- (15) The pH adjustment may be performed upon receipt of the sample at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$ .
- (16) K.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorous by Gas-Liquid Chromatography," *Journal of Chromatography*, 47 (3): 421-426, 1970.
- (17) Reference: "Prescribed Procedures for Measurement of Radioactivity in Drinking Water" EPA-600/4-80-032 (1980 Update) U.S. EPA, August, 1980.
- (18) This list does not represent an exhaustive list of all approved test methods. When determining the appropriate analytical method, always refer to 40 CFR Part 136. **NOTE: See Appendix XIII for details on 40 CFR Part 136.**

*Appendix I*  
*General Industrial Inspection Questions*

## ***General Industrial Inspection Questions***

### **GENERAL QUESTIONS**

#### **A. Usage of Chemicals, Cleaners, and Location of Drains**

1. Check the proximity of any chemical storage areas to floor drains. What kind of chemicals are stored? Chemicals might include, paint, thinner, solvents, etc. Are the chemicals stored in a way that they could reach the floor drains if spilled?
2. Check the floor washdown procedures (frequency, water usage, detergents). What is the frequency (daily, periodical) of the washdown? Are high pressure sprays used? Are detergents used? How is the wash water disposed?
3. If floor drains are sealed, do employees have access?
4. Check for the use of detergents and chemical cleaners for equipment washdown. Acids (e.g., muriatic, sulfuric, phosphoric, acetic, etc.), surfactants, caustic soda, soda ash, and phosphates are commonly used as cleaners. How are these materials stored? How are working concentrations of chemical prepared, and who prepares them?

#### **B. Cooling Waters**

1. Are there any sources of uncontaminated cooling water in the plant? Are there any sources of recirculated or once-through cooling waters? What is the disposal method of the cooling water?
2. If contact cooling water is used, is it treated in any way before discharge? What contaminants would be in the water? Are conditioning chemicals added to cooling waters?
3. Is there any water cooled machinery used by the facility? What contaminants would be in the water? What is the volume and how is the cooling water disposed?

#### **C. Solvents**

1. Does the facility use any solvents or degreasing agents?
2. Are any solvent wastes handled separately from the other cleaning solution wastes?
3. Is there any batch pretreatment prior to discharge?
4. How are any residual materials, sludges at the bottom of the tank disposed?
5. Is there a solvents management plan to reduce solvent waste at the facility?
6. If solvents are used, are they redistilled on-site? Does this process generate uncontaminated cooling water? Where is it discharged?

#### **D. Boiler Discharge**

1. Check the frequency and volume of any boiler blowdown. Check on the usage of additives to the boiler make-up waters. Do the additives contain any metals or priority pollutants?
2. What types of boiler pretreatment is used (e.g., ion exchange, chemical addition, etc.)? Are there any boiler wastes generated?
3. What is the frequency and volume of boiler blowdown?
4. Is the waste stream acting as a dilution stream at a process monitoring point?
5. Are there air pollution control devices which use water? How is the water disposed?

**E. Discharge Locations and Sampling Points**

1. Are the facility's domestic and process wastewaters segregated?
2. What method is used to determine domestic and process discharge volumes?
3. Are dilution streams accounted for at the monitoring point?
4. Does the facility have a sampling point available which is representative of the process wastewaters discharged?

**F. Food Processing**

1. What are the products processed at the facility? What is the production rate?
2. Does the facility use flow equalization prior to discharging into the sewer? Does the facility have any provisions to respond to a produce spill into the wastewater system?
3. Does the facility generate any byproducts which have associated wastewaters?
4. Is there any coloring added to the product? Is there any treatment for removal of the color?
5. Check for the usage of chemical cleaners for equipment washdown. Acids (e.g., muriatic, acetic, sulfuric, and phosphoric), surfactants, caustic soda, soda ash, and phosphates are commonly used as cleaners. How are these materials stored and used?
6. Check the floor washdown procedures. Are equipment and floors washed down with water?
7. What is the water consumption rate of the operation (total gallons per day and the number of pounds of product or pounds of material processed)? How much water is generated by or incorporated into the product?
8. What percent of water use is recycled? Does this include any uncontaminated water (for refrigeration, machinery, etc.)? **NOTE:** Single pass cooling water is common in food processing and should be checked or verified.
9. What kind of containers does the facility use to package the product? Are containers made on-site? Are they washed or sterilized?
10. Are acidic and caustic solutions used and when are pH samples taken by the facility?

**G. Pretreatment**

1. What kind of treatment systems does the facility have in place for each of the various types of process wastewaters discharged? What chemicals are added? How often are monitoring equipment calibrated?
2. Are any of the process wastewaters subject to National categorical pretreatment standards? If so, are dilution waste streams accounted for during monitoring?
3. Does the facility combine its waste from the various sources prior to treatment or discharge? Is the combined wastestream formula applicable? If so, are proposed waste stream volume determination method accurate?

**H. Radioactive Materials**

1. Determine the maximum quantity of each radionuclide used, stored, and discharged at the facility. Does the storage area have adequate spill control?
2. How are liquid and solid radioactive wastes being disposed?
3. Are they being hauled away? If so, what is the name of the hauler and what is the destination of the waste?

4. Are they being discharged to the sanitary sewer? If so, how often and what are the maximum concentrations in Curies?
5. Obtain a copy of the industrial user's radioactive user's license(s).
6. Obtain a copy of any protocols for handling radioactive materials at the facility.
7. Obtain a copy of any logs pertaining to radioactive discharges.

*Appendix II*  
*Industry Specific Questions*

## ***Industry Specific Questions***

### ***Adhesives and Sealants:***

1. What is the product manufactured at the facility? Are the adhesives water-based or organic solvent base materials? What kind of binder material is used?
2. Are there any product washing operations? Are reactor vessels washed down between batches? Is water or a solvent used? Would these wastes be discharged to the sewer? What is the frequency and volume of washing operations?
3. Check the general questions on solvents listed below.
4. Check the usage of cooling waters. See general questions on cooling waters listed below.

### ***Aluminum Forming:***

1. What is the production rate of this facility in terms of mass of aluminum or aluminum alloy processes per year? Is there an accurate method for determining off-lbs from individual processes?
2. What are the forming processes at the facility? Is there a waste stream generated from any air pollution control unit present?
3. What kind of metal forming lubricating compounds are used? Is water recycling feasible?
4. How often are the lubricant-wastewater emulsions changed and discharged?
5. Is there a continuous overflow from quenching water baths? What is the disposal method for the quench waters?
6. Is any casting done on site? If yes, see questions under Metal Refineries and Foundries listed below.
7. Are any solvents used as part of the cleaning processes? Refer to the general questions on Solvents.
8. Are wastewaters from desmutting and deoxidizing pretreated before discharge to the sewers? What volume is discharged?
9. Are there any metal finishing processes (anodizing, chemical conversion coatings, coloring, dyeing, chemical sealing, chemical or electrochemical brightening, or etching) done on site? If yes, refer to questions under Electroplating and Metal Finishing listed below.

### ***Auto Repair and Paint Shops:***

1. Do the paint booths use a water curtain? If so, how often is it discharged? How is the bottom sludge disposed? Are water conditioning chemicals used?
2. Check the paint spray gun cleaning procedures and the method of disposal of any cleanings. Are employees trained, supervised, and rewarded to paint efficiently, thus reducing contaminants to the sewer?
3. How does the facility dispose of old unwanted paint?
4. Is there any other disposal of any chemicals at this site? What is the disposal method?

### ***Auto Parts and Suppliers: Wholesale and Retail***

1. If floor drains are present, is there any storage of oils, paint, anti-freeze, transmission and brake fluids, or any other fluids within the proximity of the drains? What is the quantity of fluids stored?
2. Check the location and manner of storage of batteries and battery acid.
3. Check for used crankcase oil and return facilities.
4. Check for any machining or repair. See Auto Repair questions.

***Auto Repair (Mechanical) - Engine and Transmission Work***

1. Look for drains under service bays. What is the destination of the drain?
2. Identify the location of any gas/oil interceptors or separators. What kind of unit is it and what is the general operating condition of the unit?
3. Check on the use of solvents and parts degreasers. Check the general questions on solvents.
4. Check for the storage of fluids such as oil, transmission fluid, brake fluid, and anti-freeze.
5. Check on the quantity and method of waste oil storage and the manner and location in which it is disposed.

***Auto Wash***

1. Check for any system for water reuse or reclamation such as settling tank.
2. If a settling tank exists, check how the sludge from it is handled and by whom. what is the ultimate disposal method?
3. Check what types of cleaners are used. Do any specialty cleaners such as tire cleaners contain solvents? Do waxes contain solvents? If yes, check the general questions on solvents.
4. Check for an oil and grease separator on the discharge line.
5. Check for the storage of any liquids near the floor drains.
6. Check and identify the water consumption level.
7. Can the facility handle trucks? If so, what kinds of materials might be contained in the trucks and what the washwater contamination from those materials?

***Bakeries - Retail***

1. Check the washdown and cleanup procedures.
2. Check the storage of cleaning agents.
3. Check the storage of baking ingredients.
4. Check the quantity of deep fry grease generated. Note how it is disposed.
5. Check for the presence of any grease interceptor. Describe the size and general condition of the unit. How often and by whom is it serviced? How is the grease disposed?

***Battery Manufacturing***

1. What is the production rate of the facility (number of units manufactured, amp per hour output, etc.)?
2. What is the primary reactive anode material (cadmium, calcium, lead, leclanche, lithium, magnesium, nuclear, zinc) used for the batteries produced at the facility?
3. What volume of wastewater from electrodeposition rinses, scrubber bleed off and caustic removal is discharged to the sewer?
4. Are depolarizers used in the manufacturing process? What type? What is the final disposal method of these materials?
5. What kind of electrolytes does the facility use? Check the general questions on Usage of Chemicals, Cleaners and Location of Drains.

***Beverages***

1. Check the general questions on Food Processing.

**Blueprinting and Photocopying**

1. Check to see if they do any offset printing (related questions).
2. What type of blueprinting machines are being used? With some, the total ammonia is totally consumed while other will have spent ammonia solution to dispose.
3. Is there a significant amount of ammonia stored? Check the floor drains.
4. Check the usage of other cleaning agents and solvents. Are there any chemicals of concern?

**Canned and Preserved Fruits and Vegetables**

1. Check what detergents and techniques are used in washing fruits and vegetables before rinsing.
2. In addition to checking the water usage for washing, rinsing, and cooling, check to see if water is also used for conveyance and the amounts used.
3. Is peeling done chemically (i.e., caustic soda, surfactants to soften the cortex)? Is there any discharge from the peeling operation?
4. Check the floor washdown procedures. Are equipments and floors washed down with water?
5. Does the facility have a grease and solids recovery system? Is there any other pretreatment before discharge? Describe the system. How often is monitoring equipment calibrated?
6. Are there any processing brines used by the facility? How are these brines disposed of? Check the kind of treatment given the brines prior to discharge to the sewer.
7. How are the larger remains of processed fruit and vegetables disposed (ground up and flushed down the sewer? used as byproducts?)?
8. Check the refrigeration system for possible leaks.
9. Are there any fungicides or other similar chemicals used in the processing? Are they discharged to the sewer?
10. Check the general questions on Food Processing.

**Coil Coating (Including Canmaking)**

1. What is the average square footage of metal sheeting processed at the facility (either on a daily or annual basis)?
2. What is the base metal processed (aluminum, galvanized steel, and/or steel)?
3. Check the general questions on solvents.
4. What sort of conversion coating is used at the facility (chromating, phosphating, complex oxides)?
5. What solvents are used to control viscosity?
6. Is there a continuous overflow from quenching water baths? What is the disposal method from the quench waters?

**Canmaking (in addition to the questions above)**

1. What kind of metal forming lubricants does the facility use?
2. What is the volume of rinse waters discharged to the sewer? Have the wastewaters been characterized?

**Eating Establishments: (Restaurants)**

1. Check for the presence of any grease interceptor. Describe the size and general condition of the unit. How often and by whom is the unit serviced? How is the grease disposed?

2. How does the facility dispose of spent cooking grease?
3. How does the facility dispose of edible garbage material?
4. Check what types of janitorial cleaners are used. How are they stored?
5. Does the facility use an automatic dishwasher? Approximately how many hours per day does it operate? What is the discharge temperature and the water consumption rate? Is the dishwasher connected to any grease interceptors?
6. How many sinks does the facility have and how are they used?
7. How are grill cleaning residuals disposed?

***Electric Services***

Steam Electric Power Generation

1. Are the plants run on coal, oil, or gas?
2. What is the source of condenser cooling water (e.g., city, river, wells)? Are there any water treatment chemicals added by the facility? How is the cooling water disposed?
3. What is done with the waste oils?

Substations

1. Check the location of any floor drains.
2. Is there any contact cooling water discharge?
3. Look for signs of leaking transformer oil and to where it would go if leakage occurred. Look for a label on the transformer for the identification of PCBs. What percentage of PCBs are in the oil?

***Electronic Components***

1. What is the product that is manufactured at this facility?
2. Does the facility use any solvents or degreasing agents? If yes, check the general questions under Solvents.
3. Does the facility use any cooling water? Check the general questions under Cooling Waters.
4. Does the facility have a clean air room for which is must scrub air? Are there any chemical wastes generated from the scrubber? How is the waste disposed?
5. Does the facility conduct any electroplating activities? Check the questions under Electroplating and Metal Finishing.
6. Is there any water recycle/reuse within the plant? Does the plant employ pretreatment for the recycle streams?
7. Does the facility employ any photographic processes?
8. Check the general questions under Solids Disposal.

***Electroplating and Metal Finishing***

1. Try to determine the quantity of plating done by the facility in terms of surface area (sq. ft., etc.) plated.
2. How often does the facility change its cleaning solutions, both acidic and alkaline cleaners? What is the volume for each change and how is the old material disposed? Is there any batch pretreatment prior to discharge? How are any residual materials, i.e., sludges, at the bottom of the tank disposed?
3. Does the facility use any solvents or degreasing agents? If yes, check the general questions under Solvents.
4. Does the facility use any cooling water? If yes, check the general questions under Cooling Waters.

5. What types of chemicals make up the plating baths? Is cyanide used in the plating operations? Is there any chromium used? Is there any ammonium persulfate used in an etching process? Are employees aware of water conservation/drag out?
6. How often are plating bath solutions changed? What is the volume for each change and how is the old material disposed? Is there any batch pretreatment prior to discharge? How are any residual materials at the bottom of the tank disposed?
7. Is there any water recycle/reuse within the plant? Does the plant employ any pretreatment for the recycle streams?
8. If masking is employed, are photographic processes involved (circuit boards)?
9. Are there metal finishing operations (e.g., machining, grinding, coloring, brightening, etc.) associated with the plating operations?
10. If metal coloring is present, are organic dyes used?
11. Check the plumbing of the process wastewater from the plating room to the pretreatment system or sewers. Are floor drains in the plating room directed to the pretreatment units? Are floors washed down regularly? How much water is used and discharged? Where is the discharge location for dilution streams?
12. Does the facility use running water systems for rinsing? Are the units set up for countercurrent flows? Are any still or dead rinses used? Check if the rinsewaters are pretreated prior to being discharged.
13. Has the facility been checked against any interconnections of the public water supply with process lines (cross connections)? Are there backflow preventers in place?
14. Check the general questions under Solids Disposal.

***Explosives***

1. What are the products manufactured at this facility?
2. Does the facility blend these products into end-use products?
3. Is ammonium nitrate used in the product and if so, how is it monitored for in the wastewater?
4. Does the facility have a disposal area for obsolete, off-grade, contaminated, or unsafe explosives and propellants?
5. Are the products produced for private sector usage or military usage? Are there any security clearances necessary to enter the facility?
6. Check the general questions for Solids Disposal.

***Fiberglass Insulation***

1. What methods are used to bind and cool the glass after it has been drawn into fibers? What wastes are generated from this phase? Are these wastes pretreated prior to being discharged to the sewer?
2. What method is employed for collecting the glass fibers (i.e., wire mesh conveyors, flight conveyors, etc.)? What methods are used to clean the conveyors or any glass fibers? Is this process shut down or in service while being cleaned? What type of cleaning agent is used? Is the wastewater discharged to the sewer?
3. Are wet air scrubbers used? Is wastewater discharged to the sewer? Is the wastewater treated prior to discharge (e.g., sedimentation for particulate matter)?
4. How are any backings applied (heat, adhesives, etc.)?

***Fuel Oil Dealers***

1. Record storage capacity (above ground and underground).

2. Are the above ground storage and the loading areas diked or bermed? Is there any leakage or spillage access to storm or sanitary sewers?
3. Are any oils or fuels stored inside the building? Note the proximity of floor drains to these areas.
4. What types of absorbent is used for spills? How much is stored for immediate availability? Note the proximity of the material to any floor drains.
5. Does the facility have a spill prevention plan? Do employees receive spill plan training?

***Funeral Services***

1. Check what kind of chemicals are used and how they are stored. What is the storage proximity to the floor drains?
2. Check how much formalin is used for embalming. What percentage of usage is discharged to the sewer? How much blood is discharged per day? Are there any other chemicals involved in the embalming process?
3. Check the washing and cleaning procedures at the embalming table. What kind of detergents and disinfectants are used?
4. How are infectious wastes disposed?

***Gasoline Service Stations***

1. Check the location of any floor drains. Look for drains under service bays. What is the destination of the drain?
2. Identify the location of any gas/oil interceptors or separators. What kind of unit is it and what is the general operating condition of that unit?
3. Check what kind of chemicals are used and how they are stored. Chemicals might include fluids such as oil, transmission and brake fluids, anti-freeze, or solvents. What is the proximity of the storage area to floor drains?
4. Check the quantity and method of waste oil storage and the manner and location in which it is disposed. Is there a waste oil receptacle (drum or tank)?
5. Check the disposal method used for radiator flushing.

***Gum and Wood Chemicals Manufacturing***

1. What volume of product is produced on a yearly basis?
2. If gum resin, turpentine, or pine oil are produced, what is the volume of process wastewater from stripping, vacuum jet stream condensates, and unit washdown?
3. If tall oil resin, pitch, or fatty acids are produced, what is the volume of wastewater from the acid treatment system, overflow from the evaporative cooling system, process washdowns and quality control lab wastes?
4. Check the general questions on Cooling Water.
5. If essential oils are produced, what is the volume of contaminated condensate that is discharged from the batch extraction of oil of cedarwood?
6. If rosin derivatives are produced, what is the volume of wastewater from the water of reaction; sparge stream, if used; and the vacuum jet stream?

***Hospitals***

1. Note the general layout of the facility (e.g., types of labs, equipment, morgue, laundry, food services, etc.).

2. Check what kind of chemicals are used and how they are stored. What is the storage proximity to the floor drains?
3. Any special procedures used for handling infectious or hazardous wastes? Identify the mode of disposal and names of any haulers of such wastes.
4. What are the types and quantities of cleaners and germicides utilized in cleaning procedures?
5. Disposal of spent photoprocessing chemicals (i.e., fixer) from X-ray departments?
5. See general questions on Radioactive Materials.

***Inorganic Chemicals***

1. What is the product that is manufactured at this facility?
2. Are any brine muds generated by the facility's production or inorganic compounds? Do these brines contain any known heavy metals? How are these brine muds disposed?
3. Does the facility generate any air scrubber wastewater? What is the chemical quality of this water and how is it disposed?
4. Are any cyanide (CN) compounds generated by the facility? Are the CN wastestreams segregated and/or pretreated prior to discharge?
5. Check the general questions under Cooling Waters.
6. Check the general questions under Solids Disposal.

***Laundries***

1. Is dry cleaning done? If so, what is the solvent used?
2. Is sludge generated? What is the disposal method?
3. If solvents are used, are they redistilled on-site? Does this process generate uncontaminated or contaminated cooling water? Where is it discharged? What is the discharge volume?
4. Do washers have lint traps and settling pits?
5. What is the temperature of the effluent? Is a heat exchange system used?
6. Are printers rags, shop rags, or other industrial materials cleaned?
7. What types of detergents and additives are used? What is the pH of the effluent? How are chemicals added?
8. Are laundry trucks maintained and washed on-site? If so, how are the waste oils, etc. handled? Are there any floor drains leading to the sewer in the vicinity of the vehicle wash or vehicle storage area?
9. Is there any loss of water as a result of evaporation? What is the estimated volume of the loss?
10. What is the water consumption at the facility? What is the source of the water used at the facility?

***Leather Tanning and Finishing***

1. What method is used to preserve the received hides? (Note: hides preserved with salt will result in a high dissolved solids count in the effluent).
2. What types of skins and/or hides are tanned? (Note: if sheepskins or goatskins are tanned, there will be a separate solvent or detergent degreasing operation.)
3. Is hair saved or pulped (i.e., chemically dissolved)? (Note: in a save hair operation with food recovery of hair, the contribution to the effluent strength is substantially lower than in pulp hair operations).

4. Is deliming accomplished by treating with mild acids or by bating? What is the destination of these waters?
5. What types of tannin are used? (Note: chrome and vegetable tannins are the most common. A combination of tannins may also be used).
6. Are chemicals stored near floor drains? (This is a very appropriate question to ask since many liquid chemicals are used in the leather tanning industry).
7. Are tannins recycled and/or chemically recovered? What happens to this wastewater?
8. Are any pretreatment units employed? What is the calibration of monitoring equipment?
9. If sludge is generated, how is it disposed?
10. Check the general questions under Cooling Waters.

***Lumber and Building Materials: Retail***

1. Check for the storage of paint, thinner, and other solvents, adhesives, roofing materials, etc.
2. Does the facility mix paint? Is the paint mixing dry or does it involve water? Check the nearby sinks for evidence of water usage. How is the waste paint disposed?
3. Are cutting oils used and are they water soluble?
4. Are hydraulic oils used?
5. Would any of these oils ever be discharged to the sewer?

***Machine and Sheet Metal Shops***

1. What type of product is manufactured?
2. What kind of material is machined?
3. Are cutting oils used and are they water soluble?
4. Are hydraulic oils used?
5. Would any of these oils ever be discharged to the sewer?
6. Are any degreasing solvents or cleaners used? What are the chemical make up and/or brand names of the degreasers and how are they used? How are the spent degreasing chemicals or sludge disposed? Is degreasing rinse water discharged to the sewer?
7. Is there any water cooled equipment such as a vapor degreaser or air compressor? What is its discharge frequency and volume?
8. Is any painting done on the premises? How are waste thinners or paints disposed? Is a water curtain used for control of solvents entering the air and is contaminated water discharged?
9. Is any type of metal finishing done, such as anodizing, chromating, application of black oxide coating or organic dye? What are the chemicals used, volumes consumed, and destinations of the finishing chemicals?
10. What is the water consumption at the facility?
11. Are there any pretreatment units, traps, etc.?

***Meat Products/Poultry Products***

1. What type of livestock are slaughtered and/or processed?
2. What are the principal processes employed?

3. What methods are used to dehair? Is the hair recovered from the wastewater stream?
4. Does the facility cure hides? What brine solution is used specifically (i.e., sodium chloride)? Are hides cured in vats? Are vats ever discharged to the sewer? What is the frequency and volume of such discharges?
5. What are the by-product processes?
6. Is rendering practiced at the plant? How (i.e., catch basins, grease traps, air flotation, etc.)? How often are the systems cleaned out?
7. What methods are used for clean-up operations? What detergents are used (i.e., caustic, alkaline, etc.)?
8. Which wastestreams, if any (i.e., uncontaminated water) bypass all treatment and discharge directly to the sewer?
9. If poultry processing is done, how are the feathers removed? How are they disposed? How are chicken parts disposed? How is blood disposed?

***Metal Heat Treating Shops***

1. What kinds of metal are heat treated?
2. What fluids are used for quenching metals? Are these ever discharged to the sewer?
3. Are sludge ever removed from the quenching tanks? How are the sludge disposed?
4. Is any of the metal cleaned before or after heat treating? Are any degreasing solvents or cleaners used and how are they used?
5. Are there any water cooled quenching baths, vapor degreasers, or other equipment? Where are they discharged? What volumes are discharged?
6. What is the water consumption?

***Metal Refineries and Foundries***

1. What is the product that is manufactured at the facility?
2. Does the facility use any solvents or degreasing agents? Check the general questions under Solvents.
3. Does the facility use any cooling water? Check the general questions under Cooling Waters.
4. Is there any water recycle/reuse within the plant? Does the plant employ any pretreatment for the recycle streams?
5. Does the facility have a spill prevention plan developed? Does the plan include spills to the sewer of highly acidic or caustic materials?
6. Check the general questions under Solids Disposal.

***Nursing Care Facilities***

1. Food service (see Restaurant questions).
2. Any chemical usage (e.g., lab facility)?
3. Janitorial chemicals - usage, destination, and storage or germicides and disinfectants.

***Organic Chemicals***

1. Are processes batch or continuous?
2. If batch processes are used, how frequent is clean-up and how are wastes disposed?

3. Are waste disposal services or scavengers used? If so, are they licensed? Which wastes are hauled?
4. What types of solvents are stored in bulk?
5. Check the points for the discharge of cooling water. Check general questions under Cooling Waters.
6. Is there water in contact with catalysts used at the plant (e.g., in cleaning catalyst beds)?
7. List all products and raw materials.
8. Are there laboratories for research and for product testing? How are laboratory wastes disposed?
9. Is the water used in boiler feed or in processing pre-created? How are laboratory wastes disposed?
10. Are storage areas near drains leading to the sewer?
11. Are there any chemical reaction or purification techniques, such as crystallization, filtration, or centrifugation, which produce wastewater and/or sludge wastes? What is the destination of these wastestreams and/or sludge?
12. Are there any pretreatment units at the facility?
  
13. Is deionized water used, how is it generated (on-site)? Are columns regenerated on-site? Does the facility use acids or caustics? Is there a discharge from the deionization process? Where does the discharge go?
14. Is a water tower used? What is the frequency and volume of the discharge? Where does the discharge go? Are any additives such as chromates used by the facility?

***Paint and Ink Formulation***

1. What types of inks are made (i.e., oil-based or water-based)?
2. What types of paints are manufactured (i.e., water-based or solvent-based)?
3. What are the pigments made of?
4. Are extenders used?
5. Are any solvents used? Check the general questions under Solvents.
6. What are the resin types?
7. What other ingredients are used in formulating the product?
8. Is there any discharge to the sewer system (washdown and/or bad batches)? Are any chemicals used to clean product equipment?
9. Are there any floor drains in chemical storage and mixing areas?
10. Is there a scavenger service? If yes, for which wastes?
11. Is there on-site disposal of solids by burial? If not, where do the solids go?
12. Check the general questions on Cooling Waters.

***Paper Mills***

1. What are the products manufactured at the plant?
2. Which specific chemicals are used in the process?
3. Is pulp bleached? If yes, what is the process and what chemical are used?
4. Are any chemicals manufactured on-site (e.g., chlorine dioxide, hypochlorites, etc.)? Are any chemicals discharged from these operations?

5. Are recovery streams (white water recycle, cooking liquor regeneration, cooling water reuse, etc.)?
6. Where is cooling water used in the plant (e.g., condensers, vacuum pumps, compressors)? Where is it discharged?
7. Describe the types of fillers, coatings, finishes, etc., in paper making.
8. What happens to bad batches or liquids in case of equipment failure? Are they discharged to the sewer?
9. On the average, how much water is consumed in the process of making paper? What is the source of the water?

### ***Paving and Roofing***

#### **Tar and Asphalt**

1. Does the wastewater from wet air scrubbers used on the oxidizing tower discharge directly to the sewer? Is it treated and recycled?
2. What method(s) are used to control the temperature of the oxidizing tower (i.e., water)? Is this waste discharged or recycled?
3. What treatment methods are used to remove suspended solids or oil from the water (i.e., catch basins, grease traps, sedimentation, oil skimmers)?
4. Is water or air used to cool asphalt products? If water, is it contact or noncontact? If contact, is this water discharged directly to the sewer or does it undergo treatment? (Note: mist spray used alone causes the largest amount of solids present in wastewater.)
5. Check the general questions under Cooling Waters.
6. Are any solvents used? Check the general questions under Solvents.

### ***Pesticides***

1. Does the facility manufacture or blend pesticides at this location?
2. What pesticides are manufactured or formulated at the facility? What volumes of product is produced on a yearly basis?
3. Check the chemical storage areas.
4. How are chemical containers rinsed? Is the rinse water discharged to the sewer?
5. What is the volume of wastewater from the final synthesis reaction or the dilution water step used directly in the process?
6. Check the procedures for floor and/or equipment washes.
7. Check the general questions under Solids Disposal.

### ***Petroleum Refining***

1. What are the processes employed by the facility and what is the throughput (in barrels per day) of each of the following processes:

Topping: The term includes basic distillation processes;

Cracking: The term cracking includes hydrocracking, fluid catalytic cracking, and moving bed catalytic cracking processes;

Petrochemical: This includes the production of second generation petrochemicals (i.e., alcohols, ketones,

cumene, styrene, etc.), first generation petrochemicals, and isomerization products (i.e., BTX, olefins, cyclohexanes, etc.); and

Lube: This term includes hydrofining, white oil manufacturing, propane dewaxing, solvent extractions and dewaxing, naphthenic lubes, phenol extraction, SO<sub>2</sub> extraction, etc.

2. Identify the location of any oil interceptors or separators. What kind of unit is it and what is the general operating condition of the unit?
3. Does the facility employ any biological treatment prior to discharging to the sewer?
4. Are there any controls in place for phenols, sulfides, hexavalent chromium, and/or ammonia? How does the facility dispose of any spent caustic which it might generate?
5. Is storm water runoff isolated from the sewer discharge? How is the contaminated storm water runoff disposed? Does the facility have a NPDES permit for stormwater?
6. Check the general questions for Cooling Waters.
7. Check the general questions for Sludge Disposal.

#### **Pharmaceuticals Manufacturing**

1. What type of processes are used to manufacture the product (e.g., fermentation, biological, or natural extraction, chemical synthesis, mixing/compounding and formulation).
2. If processes include fermentation and/or chemical synthesis, are these continuous or batch discharge?
3. If chemical synthesis is involved, what processing steps (crystallization, distillation, filtration, centrifugation, vacuum filtration, solvent extraction, etc.) produce wastewater? Are these wastewaters discharged to the sewer system?
4. What types of solvents are used? Check the general questions on Solvents.
5. Is raw water intake purified? If yes, by what method (e.g., ion exchange, reverse osmosis, water softening, etc.)? What types and volumes of wastes are generated? What is the frequency of discharge?
6. What is done with the spent beer generated by fermentation?
7. Check the general questions on use of cleaners and location of drains.
8. Is there any chance of spills or batch discharges?
9. Check the usage of cooling waters. See general questions on Cooling Waters.
10. Is there a research lab in the plant? What are the wastes generated by the facility? How are they controlled?

#### **Photographic Process**

1. Determine what type of chemistry is used. This is important because some of the chemicals may be toxic while others are not.
2. What types of films are developed? Are prints made? Give an estimate of how much total processing is done per day. How many automatic processors are utilized and how long are they in operation per day?
3. What chemical brands are used? What type of process chemistry is used: C-41, E-6, CP-30, etc.? What are the names of each chemical used in the process? What are the volumes used? Which chemicals are discharged to the sewer? Do any of the chemicals contain cyanide?
4. What is the square footage of the material being processed?
5. Is silver recovery practiced? Is bleach regeneration practiced, and if yes, is it done within the lab? What are the processes and wastes involved? Does the silver recovery system have a maintenance schedule?

6. What is the wastewater flow from each of the photographic processing operations? Does the rinse water on the processors run continuously or does it shut off when film is not being processed? How often are the processors cleaned and the chemicals changed? How often, if ever, are these chemicals discharged to the sewer? What chemicals, if any, are used to clean the processor rollers and trays? Are there any floor drains where the chemicals are mixed or stored?
7. Is there any type of pretreatment or pH control?
8. What is the frequency of silver recovery maintenance?
9. What is the water consumption of the facility?

***Plastic and Synthetic Materials Manufacturing***

1. What is the product that is being manufactured?
2. What are the raw materials used, including any accelerators and inhibitors? Are there any known toxics (such as cyanide, cadmium, or mercury) utilized in manufacturing the product?
3. What type of polymerization process is employed? Does the process use a water or solvent suspension? What are the wastes generated from the process? What are the possible contaminants? How are the wastes disposed?
4. Are there any product washing operations? Are reactor vessels washed down between batches? Is water or a solvent used? Would these wastes be discharged to the sewer?
5. Check the usage of cooling water. Check the general questions on Cooling Waters.

***Porcelain Enameling***

1. What is the square footage of material enameled at the facility on annual basis?
2. How is the base metal prepared for enameling?
3. Is any electroplating done on-site? If yes, check the general questions on Electroplating and Metal Finishing.
4. What coating application method is used?
5. Check the general questions under Usage of Chemicals, Cleaners and Location of Drains.

***Printing***

Some of the following questions may apply and others may not; experience will be the best judge. The SIC number for offset and silkscreen printing is 2732 and letterpress is 2751. Other types of printing are listed in the 27XX group.

1. Note the kind of printing done (i.e., offset, letterpress, silkscreen, or other types of printing).
2. If offset printing is done, is film processing and plate developing done in the shop?
3. If film processing is done, is an automatic film processor used or are trays used? Does the processor's rinse water run continuously or does it shut off after processing is completed? How often are the processor's chemical tanks cleaned out and what volume is discharged to the sewer? How much developer, fixer, and stop bath (if applicable) are used and are these discharged to the sewer? Is silver reclamation practiced? Is cyanide used at all for further reducing of negatives? Are phototypesetters used, and if yes, what chemicals are discharged?
4. If plate development is done, what type of plates are used? If they are aluminum plates, are they developed with a subtractive color key additive developer? What are the names of the developers, and what quantities are used? Is the developed washed off the plates to the sewer or wiped off with a rag? Where do the rags go? How many plates are developed?

5. If paper plates are used, what ~~type~~ of processor is employed and what are the names, volumes, and destination of the chemicals used? If a silver process is used, is silver reclamation practiced?
6. In the press room, what type of fountain solution is used and would this ever be discharged during normal use or cleanup operation? What type of solvent is used to clean the presser and how is this applied? Would this solvent ever be discharged or does it become associated with the rags? Are these rags washed on the premises or are they picked up by a commercial laundry? What is the name of the laundry? Are there any floor drains where the solvent or ink is stored? Are any of the presser waters cooled? Are there any waste oils from the presser?
7. If letter press printing is done, is old lead type smelted in the shop, and if yes, are the molds water cooled? What type of solvent is used to clean the presser and type? Check the general questions on Solvents.
8. Is silkscreen printing done? What kind of photosensitive coating is used and what volume of coating is used? What kind of developer is used and is it discharged? Is a solvent or other cleaner used to clean the screen after printing? Check the general questions for Solvents. Are the screens used over again for making new stencils or are they thrown away?
9. If a different type of printing is done, what kind is it and what are the names and volumes of the chemicals used? Are these discharged to the sewer? Are the screens used over again for making new stencils or are they thrown away?
10. Check the general questions under Cooling Waters.

***Rubber Processing***

Synthetic or Natural

1. What are the products manufactured at the facility?
2. Is the rubber natural or synthetic? If synthetic rubber is used, is it polymerized on-site and would it be a water or solvent suspension? Is there a discharge associated with the process?
3. What are the ingredients of the rubber, including all additives? What kind of anti-tack agents are used? Are any known toxics used at the plant?
4. Are there any waste oils from rubber mixers or other processes which required disposal, and if yes, how are they disposed?
5. What type of forming process is used? Is cooling water used? Check the general questions on Cooling Waters.
6. Is there any wastewater associated with the curing process (e.g., steam condensate) and what would the contaminants be in the water?
7. Is rubber reclaimed, and if yes, what types of processes are used? Are any chemical agents used and how are spent agents disposed?
8. Are any final coatings applied to the rubber, paint, plastics, etc.? Are there any wastes or wastewater associated with the process and how are they be disposed?
9. Does the plant have air pollution control equipment? Does it use water as a scrubbing medium and is this discharged?
10. Check the general questions under Cooling Waters.

***Schools and Universities***

General

1. Cafeteria (see Restaurant questions).
2. Janitorial chemicals (usage, destination and storage).

Junior High and High Schools

1. Labs (chemical usage and disposal).
2. Art department (note any agents disposed to the sewer and their amounts - e.g., paint thinners).
3. Wood/metal shop (refer to questions under Woodworking Shops).

Universities

1. Is a map of the campus available to inspectors?
2. Can a master list of chemicals used on campus be provided? Which chemicals are used most?
3. Is there an organized waste chemicals pickup program? How many pickups per year? How many gallons picked up per year? Are there any central storage locations?
4. Are radioactive materials handled on campus? If yes, in what capacity? Are any wastes generated which are discharged to the sewer?
5. Any photo developing facilities on campus? Any printing facilities?
6. Any prototype PC board work in the electronics labs on campus?
7. How are pathogenic organisms disposed?
8. Any pretreatment facilities?
9. Has study been done to account for all water uses at the university (e.g., cooling water, lab wastes, cooling tower and boiler blowdowns, etc.)? What is the total campus population, including support staff?
10. Are there any floor drains near liquid chemical storage areas?

***Scrap and Waste Materials***

1. Is there any processing of the material (e.g., welding or smelting)?
2. Check the general questions under Cooling Waters.
3. Describe oil storage, including capacity. Is there a potential for discharge to the sewer?
4. Is there any other liquid storage or reclamation?

***Soap and Detergent Manufacturing***

General

1. Are only soaps manufactured, detergents, or both? Classify the plant.
2. Is there any cooling water used? Refer to the general questions under Cooling Waters.
3. How are the liquid materials stored? Are there floor drains nearby leading to the sewer?
4. Are air scrubbers used? Is water used? Caustics?
5. In product purification steps, how are filter backwashes handled?

Soap

1. What is the basic process employed for manufacturing soap (e.g., batch kettles)? What amount for fatty acid neutralization? Other?
2. Is process batch or continuous? If batch, what is the frequency and volume of reactor cleanout?
3. Is waste soap from processing sewerage?
4. Are defoamers added prior to sewer discharge?

5. Are perfumes and additives used? If so, what are they?

**Detergent**

1. What are the additives used in the product?
2. How are the spray drying towers cleaned?

***Steam Supply and Noncontact Cooling***

**Steam Supply**

1. Is the system high or low pressure steam?
2. Check the general questions under Boiler Discharges.
3. Is major cleaning and maintenance done? How often?
4. Are ion exchange systems used for boiler feed water? If yes, what types of wastes are generated?

**Noncontact Cooling Water**

1. Are cooling towers used? If yes, what are the chemical additives?
2. How frequently are towers blown down? Where does the blowdown go?
3. Are closed system ever by-passed? Under what circumstances?

***Sugar Processing***

1. Are both liquid and crystalline sugar produced?
2. What type of system exists for "sweet water" recovery?
3. Are ion exchange systems used? If yes, what are the backwashing systems likely to produce as wastes? How frequent is the backwash?
4. Are trucks or other heavy equipment maintained? Washed? Any floor drains leading to sewers? Any traps?
5. What bulk chemicals are stored and how? (Examples are acids used in liquid sugar production).
6. What happens to filter sludges in the plant? What type of filter aids are used?
7. Is cooling water used? Refer to the general questions on Cooling Waters.
8. From the cleaning of the equipment, what wastes are sewered and what wastes are recycled through the plant (e.g., filters, evaporation plans screens, etc.)

***Textile Mills***

1. What are the products manufactured at the mill? What is the approximate production of the mill?
2. What types of fibers are used in the fabric?
3. Does the raw fiber require cleaning before spinning and weaving?
4. Are the fibers or fabrics scored, mercerized, fulled, carbonized or bleached? What chemicals and rinsing operations are used and what is the destination of these wastes?
5. Is any kind of sizing applied, and if yes, what kind is it?
6. Is desizing practiced and what are the chemicals used? Are these chemicals discharged to the sewer?
7. Is dye applied to fabrics? What are the types and chemical constituents of the dyes and are the spent dye solutions and rinse waters discharged to the sewer?
8. Are any antistatic agents applied to synthetic fibers before spinning and weaving operations? Would these

be removed from the fabric and subsequently enter the wastewater discharged to the sewer?

9. Are any further finishing operations practices such as printing or application or various coatings?
10. What is the volume of wastewater generated by each chemical process?
11. Are there any methods of pretreatment employed before discharge of wastewater to the sewer?
12. Check the general questions for Cooling Waters.
13. Any liquids stored near floor drains leading to the sewer?

***Veterinary Services***

1. Check on chemical (including alcohol, germicides, pesticides, cleaners, and medicines) usage and storage. Refer to the general questions under Usage of Chemicals, Cleaners, and Location of Floor Drains.
2. Are detergents used and discharged for animal washing baths? Are there any hair clogging problems?
3. What is done with excreta material for any animals boarded?
4. Are there any special procedures taken for infectious wastes?

***Woodworking Shops***

1. Check chemical usage at the facility (e.g., look for solvents, thinners, paints, stains, cutting oils, adhesives, etc.). Check the general questions under Solvents.
2. Check the general questions under Usage of Chemicals, Cleaners, and Location of Drains.
3. How are brushes cleaned? Are any spray guns used? If yes, how are they cleaned?
4. Are cutting oils discharged?
5. Is any cooling water used? Check the general questions under Cooling Waters.

*Appendix III*

*General Operations and Maintenance Questions*

**Operations and Maintenance Questions for Industrial Users  
Policies and Procedures:**

**General Questions:**

- |     |    |     |   |
|-----|----|-----|---|
| Yes | No | N/A | 1. Is there a formal or informal set of policies for facility operations? |
|     |    |     | 2. Do policies address any of the following:                              |
| Yes | No | N/A | • Remaining in compliance?  |
| Yes | No | N/A | • Maintaining process controls?   |
| Yes | No | N/A | • Quality control?  |
| Yes | No | N/A | • Preventative maintenance?   |
| Yes | No | N/A | 3. Is there a set of standard procedures to implements these policies?    |
| Yes | No | N/A | 4. Are the procedures written or informal?                                |
|     |    |     | 5. Do the procedures consider the following areas?                        |
| Yes | No | N/A | • Safety?   |
| Yes | No | N/A | • Emergency?  |
| Yes | No | N/A | • Laboratory?   |
| Yes | No | N/A | • Process control?  |
| Yes | No | N/A | • Operating procedrues?   |
| Yes | No | N/A | • Monitoring?   |
| Yes | No | N/A | • Labor relations?  |
| Yes | No | N/A | • Energy conservation?  |
| Yes | No | N/A | • Collection system?  |
| Yes | No | N/A | • Pumping stations?   |
| Yes | No | N/A | • Treatment processes?  |
| Yes | No | N/A | • Sludge disposal?  |
| Yes | No | N/A | • Equipment record system?  |
| Yes | No | N/A | • Maintenance planning and scheduling?                                    |
| Yes | No | N/A | • Work orders?  |
| Yes | No | N/A | • Inventory management?   |
| Yes | No | N/A | 6. Are the procedures followed?   |

**Organization:**

- |     |    |     |   |
|-----|----|-----|---|
| Yes | No | N/A | 1. Is there an Organizational Plan (or Chart) for operations?                           |
|     |    |     | 2. Does the plan include:   |
| Yes | No | N/A | • Delegation of responsibility and authority?   |
| Yes | No | N/A | • Job descriptions?   |
| Yes | No | N/A | • Interaction with other functions (such as maintenance)?                               |
| Yes | No | N/A | 3. Is the Plan formal or informal?  |
| Yes | No | N/A | 4. Is the Plan available to and understood by the staff?                                |
| Yes | No | N/A | 5. Is the Plan followed?  |
| Yes | No | N/A | 6. Is the Plan consistent with policies and procedures?                                 |
| Yes | No | N/A | 7. Is the Plan flexible (i.e., can it handle emergency situations)?                     |
|     |    |     | 8. Does the Plan clearly define lines of authority and responsibility in such areas as: |
| Yes | No | N/A | • Laboratory?   |
| Yes | No | N/A | • Process control?  |
| Yes | No | N/A | • Instrumentation?  |
| Yes | No | N/A | • Sludge disposal?  |
| Yes | No | N/A | • Collection system?  |
| Yes | No | N/A | • Pumping stations?   |
| Yes | No | N/A | • Monitoring practices?   |
| Yes | No | N/A | • Mechanical?   |

- Yes No N/A • Electrical?
- Yes No N/A • Buildings and grounds?
- Yes No N/A • Automotive?
- Yes No N/A • Supplies and spare parts?

**Staffing:**

- Yes No N/A 1. Is there an adequate number of staff to achieve the policies and procedures established in the plan?
- Yes No N/A 2. Are staff members adequately qualified for their duties and responsibilities by demonstrating:
  - Yes No N/A • Certification?
  - Yes No N/A • Qualification?
  - Yes No N/A • Ability?
  - Yes No N/A • Job performance?
  - Yes No N/A • Understanding of treatment processes?
- Yes No N/A 3. Is staff effectively used?
- Yes No N/A 4. Has the potential for borrowing personnel been considered?
- Yes No N/A 5. Are training programs followed for:
  - Yes No N/A • Orientation of new staff?
  - Yes No N/A • Training new operators?
  - Yes No N/A • Training new supervisors?
  - Yes No N/A • Continuing training of existing staff?
  - Yes No N/A • Cross training?
- Yes No N/A 6. Which of the following training procedures are used?
  - Yes No N/A • Formal classroom?
  - Yes No N/A • Home study?
  - Yes No N/A • On-the-job training?
  - Yes No N/A • Participation in professional conferences or organizations?
- Yes No N/A 7. Does the training program provide specific instruction for the following operations and maintenance activities?
  - Yes No N/A • Safety?
  - Yes No N/A • Laboratory procedures?
  - Yes No N/A • Treatment processes?
  - Yes No N/A • Instrumentation?
  - Yes No N/A • Equipment troubleshooting?
  - Yes No N/A • Handling personnel problems?
  - Yes No N/A • Monitoring practices?
  - Yes No N/A • Handling emergencies?
  - Yes No N/A • Mechanical?
  - Yes No N/A • Electrical?
  - Yes No N/A • Automotive?
  - Yes No N/A • Building maintenance?
  - Yes No N/A • Inventory control?
- Yes No N/A 8. Does management encourage staff motivation?
- Yes No N/A 9. Does management support its first-line supervisors?
- Yes No N/A 10. Is staff motivation maintained with:
  - Yes No N/A • Encouragement for training?
  - Yes No N/A • Job recognition?
  - Yes No N/A • Promotional opportunities?
  - Yes No N/A • Salary incentives?
  - Yes No N/A • Job security?
  - Yes No N/A • Working environment?

**Operations:**

- Yes No N/A 1. How are operating schedules established?
- Yes No N/A 2. Do schedules attempt to attain optimum staff use?
- Yes No N/A 3. Are line supervisors included in manpower scheduling?
- Yes No N/A 4. Are staff involved in and/or informed of manpower planning?
- Yes No N/A 5. Is there sufficient long-term planning for staff replacement and system changes?
- Yes No N/A 6. Are there procedures in manpower staffing for emergency situations?
- Yes No N/A 7. How are process control changes initiated?
- Yes No N/A 8. How do process control changes interact with management?
- Yes No N/A 9. How effectively are laboratory results used in process control?
- Yes No N/A 10. Are there emergency plans for treatment control?
- Yes No N/A 11. Is there an effective energy management plan? Is the plan used?
- Yes No N/A 12. To what extent are operations personnel involved in the budget process?
- Yes No N/A 13. Do budgets adequately identify and justify the cost components of operations?
- Yes No N/A 14. Are future budgets based on current and anticipated operating conditions?
- Yes No N/A 15. Do operating and capital budget limits constrain operations?
- Yes No N/A 16. Can budget line items be adjusted to reflect actual operating conditions?

**Maintenance:**

- Yes No N/A 1. Are maintenance activities planned? Is the planning formal or informal?
- Yes No N/A 2. Does the facility have sufficient management controls to affect realistic planning and scheduling? If the controls exist, are they used?
- Yes No N/A 3. Are operating variables exploited to simplify maintenance efforts?
- Yes No N/A 4. To what extent are the supply and spare parts inventories planned in conjunction with maintenance activities?
- Yes No N/A 5. Have minimum and maximum levels been established for all inventory items?
- Yes No N/A 6. Does the facility have a maintenance emergency plan?
- Yes No N/A 7. Is the maintenance emergency plan current? Is the staff knowledgeable about emergency procedures?
- Yes No N/A 8. Does a plan exist for returning to the preventative maintenance mode following an emergency?
- Yes No N/A 9. Are preventative maintenance tasks scheduled in accordance with manufacturer's recommendations?
- Yes No N/A 10. Is adequate time allowed for corrective maintenance?
- Yes No N/A 11. Are basic maintenance practices (preventative and corrective) and frequencies reviewed for cost-effectiveness?
- Yes No N/A 12. Do the management controls provide sufficient information for accurate budget preparation?
- Yes No N/A 13. To what extent are maintenance personnel involved in the budget process?
- Yes No N/A 14. Do budgets adequately identify and justify the cost components of maintenance?
- Yes No N/A 15. Are future budgets based on current and anticipated operation and maintenance conditions?
- Yes No N/A 16. Do maintenance and capital budget limits constrain preventative maintenance (equipment replacement and improvement)?
- Yes No N/A 17. Does the maintenance department receive adequate feedback on cost performance?
- Yes No N/A 18. Can budget line items be adjusted to reflect actual maintenance conditions?

**Management Controls:**

- 1. Are current versions of the following documents maintained:
  - Yes No N/A • Operating reports?
  - Yes No N/A • Work schedules?
  - Yes No N/A • Activity reports?
  - Yes No N/A • Performance reports (labor, supplies, energy)?

- Yes No N/A • Expenditure reports (labor, supplies, energy)?
- Yes No N/A • Cost analysis reports?
- Yes No N/A • Emergency and complaint calls?
- Yes No N/A • Process control data, including effluent quality?
- Yes No N/A 2. Do the reports contain sufficient information to support their intended purpose?
- Yes No N/A 3. Are the reports usable and accepted by the staff?
- Yes No N/A 4. Are the reports being completed as required?
- Yes No N/A 5. Are the reports consistent among themselves?
- Yes No N/A 6. Are the reports used directly in process control?
- Yes No N/A 7. Are the reports reviewed and discussed with operating staff?
- Yes No N/A 8. What types of summary reports are required?
- Yes No N/A 9. To whom are reports distributed and when?

**Management Controls (Maintenance):**

- 1. Does a maintenance record system exist? Does it include:
  - Yes No N/A • As-built drawings?
  - Yes No N/A • Shop drawings?
  - Yes No N/A • Construction specifications?
  - Yes No N/A • Capital and equipment inventory?
  - Yes No N/A • Maintenance history (preventative and corrective)?
  - Yes No N/A • Maintenance costs?
- 2. Is the base record system kept current as part of daily maintenance practices?
- 3. Is there a work order system for scheduling maintenance? Is it explicit or implicit?
- 4. Do work orders contain the following:
  - Yes No N/A • Date?
  - Yes No N/A • Work order number?
  - Yes No N/A • Location?
  - Yes No N/A • Nature of the problem?
  - Yes No N/A • Work requirements?
  - Yes No N/A • Time requirements?
  - Yes No N/A • Assigned personnel?
  - Yes No N/A • Space for reporting work performed, required supplies, time required, and cost summary?
  - Yes No N/A • Responsible staff member and supervisory signature requirements?
- 5. When emergency work must be performed without a work order, is one completed afterward?
- 6. Are work orders usable and acceptable by staff as essential to the maintenance program? Are they actually completed?
- 7. Is work order information transferred to a maintenance record system?
- 8. Does a catalog or index system exist for controlling items in inventory?
- 9. Are withdrawal tickets used for obtaining supplies from the inventory?
- 10. Do the tickets contain cost information and interact well with inventory controls and the work order system?
- 11. Is the cost and activity information from work orders aggregated to provide management reports?
- 12. Is this information used for budget preparation?
- 13. Is the maintenance performance discussed regularly with staff?
- 14. How is the cost of contract maintenance or the use of specialized assistance recorded?
- 15. Are safeguards and penalties adequate to prevent maintenance cards from being returned without the work being done?
- 16. Is the preventative maintenance record checked after an emergency equipment failure?

*Appendix IV*  
*Hazards Associated with Specific*  
*Industrial Categories*

## *Hazards Associated With Specific Industrial Categories*

<b><i>Industry</i></b>	<b><i>Physical</i></b>	<b><i>Atmospheric</i></b>	<b><i>Corrosive</i></b>	<b><i>Suggested Protective Gear</i></b>
Electroplating Metal Finishing	<ul style="list-style-type: none"> <li>• Cluttered areas</li> <li>• Sloppy housekeeping</li> <li>• Wet floors/loose boards</li> <li>• Heated plating baths</li> <li>• High amperage in plating baths</li> </ul>	<ul style="list-style-type: none"> <li>• Flammable vapors</li> <li>• Exposure to chlorine</li> <li>• Sulfur dioxide vapors</li> <li>• Cyanide vapors</li> <li>• Alkaline vapors/mist</li> <li>• Acid vapor/mist</li> </ul>	<ul style="list-style-type: none"> <li>• Corrosive chemicals used in plating process</li> <li>• Heavy metal baths</li> <li>• High voltage</li> </ul>	<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Neoprene gloves</li> <li>• Boots</li> <li>• organic vapor/gas mask</li> </ul>
Chemical Blending Manufacturing	<ul style="list-style-type: none"> <li>• Slippery floors</li> </ul>	<ul style="list-style-type: none"> <li>• Leaking mixing or blending equipment</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to chemicals due to leaking equipments</li> </ul>	
Semiconductor Manufacturing		<ul style="list-style-type: none"> <li>• Exposure to freon and chlorinated solvents</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to acids (hydrofluoric and fluoroboric)</li> </ul>	
Pulp, Paper and Paperboard Manufacturing	<ul style="list-style-type: none"> <li>• Slippery floors</li> <li>• Steam heated tanks</li> <li>• Moving equipment (fork lifts)</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to:                             <ul style="list-style-type: none"> <li>- Ammonia (pulping process)</li> <li>- Chlorine (bleaching process)</li> </ul> </li> <li>• Arsenic fumes</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to caustic soda (pulping process)</li> </ul>	<ul style="list-style-type: none"> <li>• Ear plugs</li> <li>• Safety glasses</li> <li>• Latex gloves</li> <li>• Dust and mist mask</li> <li>• Boots</li> </ul>
Battery Manufacturing	<ul style="list-style-type: none"> <li>• Heat from steam curing of pasted plates</li> <li>• Spills of washwater</li> <li>• Exposure to lead metal particles during anode production</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to sulfuric acid vapors.</li> <li>• Lead fumes</li> <li>• Acid vapors</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to sulfuric acid (pickling and bleaching process)</li> </ul>	<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Boots</li> <li>• Latex gloves</li> <li>• Lead fume mask</li> <li>• Organic vapor/gas mask</li> </ul>
Leather Tanning	<ul style="list-style-type: none"> <li>• Slippery floors</li> <li>• Moving equipment in wringing operation.</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to:                             <ul style="list-style-type: none"> <li>- sulfuric acid</li> <li>- chromium</li> <li>- cyanide</li> <li>- naphthalene</li> <li>- phenol</li> <li>- pentachlorophenol</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to sulfuric and hydrochloric acids (acid pickling)</li> </ul>	
Iron and Steel Manufacturing	<ul style="list-style-type: none"> <li>• Hot ovens</li> <li>• Hot baths</li> <li>• Particulates</li> <li>• Moving equipment</li> <li>• Slippery floors</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to toxic vapors from hot baths</li> </ul>		
Industrial Laundries	<ul style="list-style-type: none"> <li>• Heated equipment</li> <li>• Slippery floors</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to toxic vapors from solvents.</li> </ul>		
Aluminum Formers	<ul style="list-style-type: none"> <li>• Moving machinery</li> </ul>	<ul style="list-style-type: none"> <li>• Metal particulates</li> </ul>	<ul style="list-style-type: none"> <li>• Nitric acid</li> <li>• Caustic solutions</li> </ul>	<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Boots</li> <li>• Latex gloves</li> <li>• Metal fume mask</li> <li>• Organic vapor mask</li> </ul>
Electrical and Semiconductors	<ul style="list-style-type: none"> <li>• Wet floors</li> <li>• Loose boards</li> </ul>	<ul style="list-style-type: none"> <li>• Acid vapors/mists</li> <li>• Ammonia vapors</li> <li>• Alkaline vapors</li> <li>• Metal fumes</li> <li>• Freon chlorinated solvents</li> <li>• Borane gas</li> </ul>	<ul style="list-style-type: none"> <li>• Chlorinates</li> <li>• Aromatic solvents</li> <li>• Hydrofluoric acid</li> <li>• Fluoroboric acid</li> </ul>	<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Boots</li> <li>• Latex gloves</li> <li>• Lead fume mask</li> <li>• Organic vapor mask</li> </ul>

Metal Molding and Casting	<ul style="list-style-type: none"> <li>• Tripping hazards</li> <li>• Hot liquid metals</li> </ul>	<ul style="list-style-type: none"> <li>• Metal particulates</li> <li>• Degreasing solvents</li> </ul>		<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Boots</li> <li>• Latex gloves</li> <li>• Hard hat</li> <li>• Metal fume mask</li> </ul>
Nonferrous Metals	<ul style="list-style-type: none"> <li>• Tripping hazards</li> <li>• Burn danger from hot metals</li> </ul>	<ul style="list-style-type: none"> <li>• Cyanide gas</li> </ul>	<ul style="list-style-type: none"> <li>• Cyanide liquid</li> </ul>	<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Boots</li> <li>• Latex gloves</li> <li>• Hard hat</li> <li>• Organic vapor/gas mask</li> </ul>
Organic Chemicals	<ul style="list-style-type: none"> <li>• Tripping hazards</li> </ul>	<ul style="list-style-type: none"> <li>• Organic vapors/mists</li> <li>• Phenols</li> <li>• Ammonia formaldehyde</li> <li>• Chlorine</li> <li>• Hypochlorite solvents</li> <li>• Plastic monomers</li> </ul>	<ul style="list-style-type: none"> <li>• Monomers</li> </ul>	<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Boots</li> <li>• Rubber gloves</li> <li>• Organic vapor/gas mask</li> </ul>
Pharmaceutical	<ul style="list-style-type: none"> <li>• Tripping hazards</li> </ul>	<ul style="list-style-type: none"> <li>• Radioactive materials</li> <li>• Biological materials</li> </ul>		<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Boots</li> <li>• Ear protection</li> <li>• Latex gloves</li> <li>• Dust mask</li> </ul>
Soap and Detergent	<ul style="list-style-type: none"> <li>• Slippery floors</li> </ul>	<ul style="list-style-type: none"> <li>• Detergent dust</li> </ul>	<ul style="list-style-type: none"> <li>• Acidic or alkaline solutions</li> <li>• High pH</li> <li>• Slippery oils</li> </ul>	<ul style="list-style-type: none"> <li>• Safety glasses</li> <li>• Boots</li> <li>• Latex gloves</li> <li>• Particulate mask</li> </ul>
Welding	<ul style="list-style-type: none"> <li>• Electric shock</li> <li>• Burns</li> <li>• Radiant energy/light.</li> </ul>	<ul style="list-style-type: none"> <li>• Exposure to toxic fumes</li> <li>• Potential for explosions caused by sparks.</li> </ul>		

*Appendix V*  
*EPA's Policy on Grab Samples vs.*  
*Composite Samples*



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

OCT 1 1992

MEMORANDUM

OFFICE OF  
WATER

SUBJECT: The Use of Grab Samples to Detect Violations of  
Pretreatment Standards

FROM: Michael B. Cook, Director *Michael B. Cook*  
Office of Wastewater Enforcement & Compliance (WH-546)

Frederick F. Stiehl *Frederick F. Stiehl*  
Enforcement Counsel for Water (LE-134W)

TO: Water Management Division Directors, Regions I - X  
Environmental Services  
Division Directors, Regions I - X  
Regional Counsels, Regions I - X

The primary purpose of this Memorandum is to provide guidance on the propriety of using single grab samples for periodic compliance monitoring to determine whether a violation of Pretreatment Standards has occurred. More specifically, the Memorandum identifies those circumstances when single grab results may be used by Control Authorities, including EPA, State or publicly owned treatment works (POTW) personnel, to determine or verify an industrial user's compliance with categorical standards and local limits. Please be aware that the concepts set out below are applicable when drafting self-monitoring requirements for industrial user permits.

REGULATORY BACKGROUND

The General Pretreatment Regulations require Control Authorities to sample all significant industrial users (SIUs) at least once per year [see 40 CFR 403.8(f)(2)(v)]. In addition, the Regulations, at 40 CFR 403.12(e), (g) and (h) require, at a minimum, that all SIUs self-monitor and report on their compliance status for each pollutant regulated by a Pretreatment Standard at least twice per year unless the Control Authority chooses to conduct all monitoring in lieu of self-monitoring by its industrial users.

<sup>1</sup> The POTW should conduct more frequent sampling and/or require more frequent self-monitoring by an industrial user if deemed necessary to assess the industry's compliance status (e.g., a daily, weekly, monthly or quarterly frequency as appropriate).

The Regulations, at 40 CFR 403.12(g) and (h), also specify that pollutant sampling and analysis be performed using the procedures set forth in 40 CFR Part 136. Part 136 identifies the proper laboratory procedures to be used in analyzing industrial wastewater (including the volume of wastewater necessary to perform the tests and proper techniques to preserve the sample's integrity). However, with certain exceptions, Part 136 does not specifically designate the method to be used in obtaining samples of the wastewater. Rather, section 403.12(g) and (h) require sampling to be "appropriate" to obtain "representative" data; that is, data which represent the nature and character of the discharge.

#### DISCUSSION OF BASIC SAMPLING TYPES

Sampling may be conducted in two basic ways. Both types of sampling provide valid, useful information about the processes and pollutants in the wastewater being sampled. The first is an "individual grab sample." An analysis of an individual grab sample provides a measurement of pollutant concentrations in the wastewater at a particular point in time. For example, a single grab sample might be used for a batch discharge which only occurs for a brief period (e.g., an hour or less). Such samples are typically collected<sup>2</sup> manually but are sometimes obtained using a mechanical sampler.

The second type of sample is a "composite sample." Composite samples are best conceptualized as a series of grab samples which, taken together, measure the quality of the wastewater over a specified period of time (e.g., an operating day). Monitoring data may be composited on either a flow or time basis. A flow-proportional composite is collected after the passage of a defined volume of the discharge (e.g., once every 2,000 gallons). Alternatively, a flow-proportional composite may be obtained by adjusting the size of the aliquots to correspond to the size of the flow. A time-proportional composite is collected after the passage of a defined period of time (e.g., once every two hours).

Generally, composite samples are collected using a mechanical sampler, but may also be obtained through a series of manual grab samples taken at intervals which correspond to the wastewater flow or time of the facility's operations. In some cases, composite data is obtained by combining grab samples prior

<sup>2</sup> Mechanical samplers may not be used to sample for certain pollutants (e.g., those which could adhere to the sampler tubing, volatilize in the sampler, or pollutants with short holding times).

to transmittal to a laboratory. At other times, the samples remain discrete and are either combined by the laboratory prior to testing or are analyzed separately (and, mathematically averaged to derive a daily maximum value).<sup>3</sup>

#### **DETERMINING APPROPRIATE COMPLIANCE SAMPLING METHODS**

EPA policy on appropriate compliance sampling types has been articulated in several pretreatment guidance manuals and regulatory preambles, and continues to be as follows:

##### **A. Compliance With Categorical Standards**

- Most effluent limits established by categorical standards are imposed on a maximum daily-average and a monthly-average bases. Generally, wastewater samples taken to determine compliance with these limits should be collected using composite methods.

- There are exceptions to the general rule. Composite samples are inappropriate for certain characteristic pollutants (i.e., pH and temperature) since the composite alters the characteristic being measured. Therefore, analysis of these pollutants should be based on individual grab samples. Alternatively, continuous monitoring devices may be used for measuring compliance with pH and temperature limits. Any exceedance recorded by a continuous monitoring device is a violation of the standard.

- Sampling wastewater from electroplating facilities regulated under 40 CFR Part 413 may be conducted using single grab samples [(assuming that the grab samples are representative of the daily discharge for a particular facility); see also preamble discussion at 44 Fed. Reg. 52609, September 7, 1979]

- A series of grab samples may be needed to obtain appropriate composite data for some parameters due to the nature of the pollutant being sampled. Examples of this situation include:

<sup>3</sup> Daily maximum discharge limits are controls on the average wastewater strength over the course of the operating day. They are not intended to be instantaneous limits applied at any single point during that operating day.

- Sampling for parameters which may be altered in concentration by compositing or storage. These pollutants include pH-sensitive compounds (i.e., total phenols, ammonia, cyanides, sulfides); and volatile organics such as purgeable halocarbons, purgeable aromatics, acrolein, and acrylonitrile.
- Sampling for pollutants with short holding times such as hexavalent chromium and residual chlorine; and
- Sampling for pollutants which may adhere to the sample container or tubing such as fats, oil and grease. Individual analysis for these parameters ensures that all the material in the sample is accounted for.

#### B. Compliance With Local Limits

- Local limits may be established on an instantaneous, daily, weekly or monthly-average basis. The sample type used to determine compliance with local limits should be linked to the duration of the pollutant limit being applied.
- Compliance with instantaneous limits should be established using individual grab samples. Exceedances identified by composite sampling are also violations.
- Compliance with daily, weekly or monthly average limits should be determined using composited sampling data, with the same exceptions noted in A, above.
- Measurements of wastewater strength for non-pretreatment purposes (e.g., surcharging) may be conducted in a manner prescribed by the POTW.

#### GRAB SAMPLING AS A SUBSTITUTE FOR COMPOSITE SAMPLING

EPA is aware that a number of Control Authorities currently rely on a single grab sample to determine compliance, particularly at small industrial users, as a way of holding down monitoring costs. It is EPA's experience that the process activities and wastewater treatment at many industrial facilities may not be sufficiently steady-state as to allow for routine use

<sup>4</sup> Certain pH-sensitive compounds can be automatically composited without losses if the collected sample is only to be analyzed for a single parameter. Additionally, a series of grab samples may be manually composited if appropriate procedures are followed.

of single grab results as a substitute for composite results. Therefore, the Agency expects composited data to be used in most cases. However, there are several circumstances when a single grab sample may be properly substituted for a single composite sample. These situations are:

- Sampling a batch or other similar short term discharge, the duration of which only allows for a single grab sample to be taken;
- Sampling a facility where a statistical relationship can be established from previous grab and composite monitoring data obtained over the same long-term period of time; and
- Where the industrial user, in its self-monitoring report, certifies that the individual grab sample is representative of its daily operation.

Except for these circumstances, Control Authorities should continue to use composite methods for their compliance sampling.

#### **GRAB SAMPLES AS A COMPLIANCE SCREENING TOOL**

Control Authorities may consider using grab samples as a compliance screening tool once a body of composite data (e.g., Control Authority and self-monitoring samples obtained over a year's time), shows consistent compliance. However, in the event single grab samples suggest noncompliance, the Control Authority

<sup>5</sup> Grab sampling may provide results that are similar to composite sampling. See for example, a March 2, 1989, Office of Water Regulations and Standards (OWRS) Memorandum to Region IX describing the results of a statistical analysis of sampling data from a single industrial facility. These sampling data included both individual grab and flow-proportional, composite sampling obtained during different, non-overlapping time periods. After reviewing the data, OWRS concluded that the composite and grab sample data sets displayed similar patterns of violation for lead, copper, and total metals. In fact, the analyses did not find any statistically significant difference in the concentration values measured between the grab and composited data. Furthermore, additional statistical tests of the two data sets indicated that the means and variances for each pollutant were similar. The statistical conclusion was that the plant was judged to be out of compliance regardless of what data were analyzed.

and/or the industrial user should resample using composite techniques on the industrial users effluent until consistent compliance is again demonstrated.<sup>6</sup>

Control Authorities may also rely on single grab samples, or a series of grab samples for identifying and tracking slug loads/spills since these "single event" violations are not tied to a discharger's performance over time.

Any time an SIU's sample (either grab or composite) shows noncompliance, the General Pretreatment Regulations, at 40 CFR 403.12(g)(2), require that the SIU notify the Control Authority within twenty four (24) hours of becoming aware of the violation and resample within 30 days. Furthermore, EPA encourages Control Authorities to conduct or require more intensive sampling in order to thoroughly document the extent of the violation(s). Of course, the use of grab samples should be reconsidered in the event the SIU changes its process or treatment.

#### **SUMMARY**

The collection and analysis of sampling data is the foundation of EPA's compliance and enforcement programs. In order for these programs to be successful, wastewater samples must be properly collected, preserved and analyzed. Although the Federal standards and self-monitoring requirements are independently enforceable, Control Authorities should specify, in individual control mechanisms for industrial users, the sampling collection techniques to be used by the industry. Generally, pretreatment sampling should be conducted using composite methods wherever possible, to determine compliance with daily, weekly or monthly average limits since this sampling technique most closely reflects the average quality of the wastewater as it is discharged to the publicly owned treatment works. Grab samples should be used to determine compliance with instantaneous limits. There are circumstances when discrete grab samples are also an appropriate, cost effective means of screening compliance with daily, weekly and monthly pretreatment standards.

<sup>6</sup> Where grab samples are used as a screening tool only (i.e., consistent compliance has been demonstrated by composite data), the results should not be used in the POTW's calculation of significant noncompliance (SNC).

<sup>7</sup> When POTWs choose to allow the SIU to collect single grab samples, the POTW should draft the SIU's individual control mechanism to clearly indicate that grab samples are to be obtained thereby preventing any uncertainty at a later date.

In summary, there are limited situations in which single grab sample data may be used in lieu of composite data. Assuming adequate quality control measures are observed, analyses of these grab samples can indicate noncompliance with Federal, State and Local Pretreatment Standards and can form the basis of a successful enforcement action. Grab sampling can also be useful in quantifying batches, spills, and slug loads which may have an impact on the publicly owned treatment works, its receiving stream and sludge quality.

Should you have any further comments or questions regarding this matter, please have your staff contact Mark Charles of OWEC at (202) 260-8319, or David Hindin of OE at (202) 260-8547.

cc: Frank M. Covington, NEIC  
Thomas O'Farrell, OST  
Regional and State Pretreatment Coordinators  
Lead Regional Pretreatment Attorneys, Regions I - X  
Approved POTW Pretreatment Programs

*Appendix VI*  
*Flow Measurement Techniques*

## ***Flow Measurement Techniques and Inspection Procedures:***

### ***Basic Hydraulic Calculations***

The relationship between flow rate (Q), the average velocity (V), and the cross-sectional area of the flow (A) is given by the following equation:

$$Q = VA$$

where: Q = flow in cubic feet per second  
V = the velocity in feet per second  
A = the cross-sectional area in square feet

To convert flow in cubic feet of water per second to flow in gallons of water per minute, the following proportionality is used:

$$\text{cubic feet} \times \frac{7.48 \text{ gallons water}}{\text{cubic foot of water}} \times \frac{60 \text{ seconds}}{\text{minute}} = \frac{\text{gallons}}{\text{minute}}$$

To convert from cubic feet per second to million gallons per day, multiply the number of cubic feet per second by 0.6463. The cross-sectional area of the pipe is described by the equation:  $A = 1/4 \pi d^2$ , where d is the diameter of the pipe in feet.

### ***Flow Measurement Devices***

Flow data may be collected instantaneously or continuously. Instantaneous flows must be measured when samples are taken so that the pollutant concentration can be correlated to the flow data. In a continuous flow measurement system, flow measurements are summed to obtain a value for the total flow to verify IU permit compliance.

A typical flow measurement system consists of a flow device, a flow sensor, transmitting equipment, a recorder, and a totalizer. Instantaneous flow data can be obtained without using such a system. The primary flow device is constructed to yield predictable hydraulic responses related to the rate of wastewater or water flowing through the device. As previously mentioned, examples of such devices include weirs and flumes, which relate water depth (head) to flow; Venturi meters, which relate differential pressure to flow; and electromagnetic flow meters, which relate induced electric voltage to flow. In most cases, a standard primary flow device has undergone detailed testing and experimentation and its accuracy has been verified.

Flow is measured by many methods; some are designed to measure open channel flows, and others are designed to measure flows in pipelines. A complete discussion of all available flow measurement methods, their supporting theories, and the devices used are beyond the scope of this manual. The most commonly used flow measurement devices and procedures for inspecting them are described in this Appendix.

**Primary Devices:**

**Weirs.** A weir consists of a thin vertical plate with a sharp crest that is placed in a stream, channel, or partly filled pipe. Figure 3-4 (in Chapter 3) shows a profile of a sharp-crested weir and indicates the appropriate nomenclature. Four common types of sharp-crested weirs are shown in Figure 3-5. This figure illustrates the difference between suppressed and contracted rectangular weirs as well as illustrates Cipolletti (trapezoidal) and V-notch (triangular) weirs.

To determine the flow rate, it is necessary to measure the hydraulic head (height) of water above the crest of the weir. For accurate flow measurements, the crest must be clean, sharp, and level. The edge of the crest must not be thicker than 1/8 inch. The rate of flow over the weir is directly related to the height of the water (head) above the crest at a point upstream of the weir where the water surface is level. To calculate the discharge over a weir, the head must first be measured by placing a measuring device upstream of the weir, at a distance of at least 4 times an approximate measurement of the head. A measurement can be taken at the weir plate to approximate the head. However, if this measurement is used to calculate the discharge, this value will provide only a rough estimate of the discharge. Therefore, when evaluating compliance with mass-based permit limits, it is essential that a more refined method of determining flow be used.

The head-discharge relationship formulas for nonsubmerged contracted and suppressed rectangular weirs, Cipolletti weirs, and 90° V-notch weirs are provided in Table VI-1. Discharge rates for the 90-degree V-notch weir (when the head is measured at the weir plate) are included in Table VI-2. Flow rates for 60- and 90-degree V-notch weirs can be determined from the nomograph in Figure VI-1. Minimum and maximum recommended flow rates for Cipolletti weirs are provided in Table VI-3. Figure VI-2 is a nomograph for flow rates for rectangular weirs using the Francis formulas.

**Parshall Flume:** The Parshall Flume is composed of three sections: a converging upstream section, a throat or contracted section, and a diverging or dropping downstream section. When there is free fall out of the throat of a Parshall flume, no diverging downstream section is required. It operates on the principle that when open channel water flows through a constriction in the channel, it produces a hydraulic head that is proportional to the flow. The hydraulic head is used to calculate the flow. Flow curves are shown in Figure VI-3 to determine free flow through 3 inches to 50 feet Parshall flumes.

The Parshall flume is good for measuring open channel flow because it is self-cleaning. Therefore, sand or suspended solids are unlikely to effect the operation of the device. The flume is both simple and accurate. The flume size is given by the width of the throat section. Parshall flumes have been developed with throat widths from 1 inch to 50 feet. The configuration and standard nomenclature for Parshall flumes is provided in Figure 3-6. Strict adherence to all dimensions is necessary to achieve accurate flow measurements. Figure VI-4

**Table VI-1**  
**Head-Discharge Relationship Formulas for Nonsubmerged Weirs\***  
**(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)**

Weir Type	Contracted	Suppressed	Remarks	Reference
<b>Rectangular</b>				
Francis formulas	$Q = 3.33 (L - 0.1nH)H^{1.5}$	$Q = 3.33 L H^{1.5}$	Approach velocity neglected	King 1963
	$Q = 3.33 [(H + h)^{1.5} - h^{1.5}](L - 0.1nH)$	$Q = 3.33 L [(H + h)^{1.5} - h^{1.5}]$	Approach velocity considered	King 1963
Cipolletti	$Q = 3.367 L H^{1.5}$	NA	Approach velocity neglected	King 1963
	$Q = 3.367 L (H + h)^{1.5} - h^{1.5}$	NA	Approach velocity considered	EPA 1973
<b>V-notch</b>				
Formula for 90° V-notch only	$Q = 2.50 H^{2.5}$	NA	Not appreciably affected by approach velocity	King 1913
	$Q = 3.01 H_w^{2.48}$	NA	Head measured at weir plate	Eli and Peterson 1979 EPA-61809A-2B

Q = discharge in cubic feet per second  
H = head in feet  
NA = not applicable  
 $H_w$  = head in feet at weir plate  
n = number of end contractions

L = crest length in feet  
h = head in feet due to the approach velocity =  $V^2/2g$   
V = approach velocity  
g = gravity (32.2 ft/sec<sup>2</sup>)

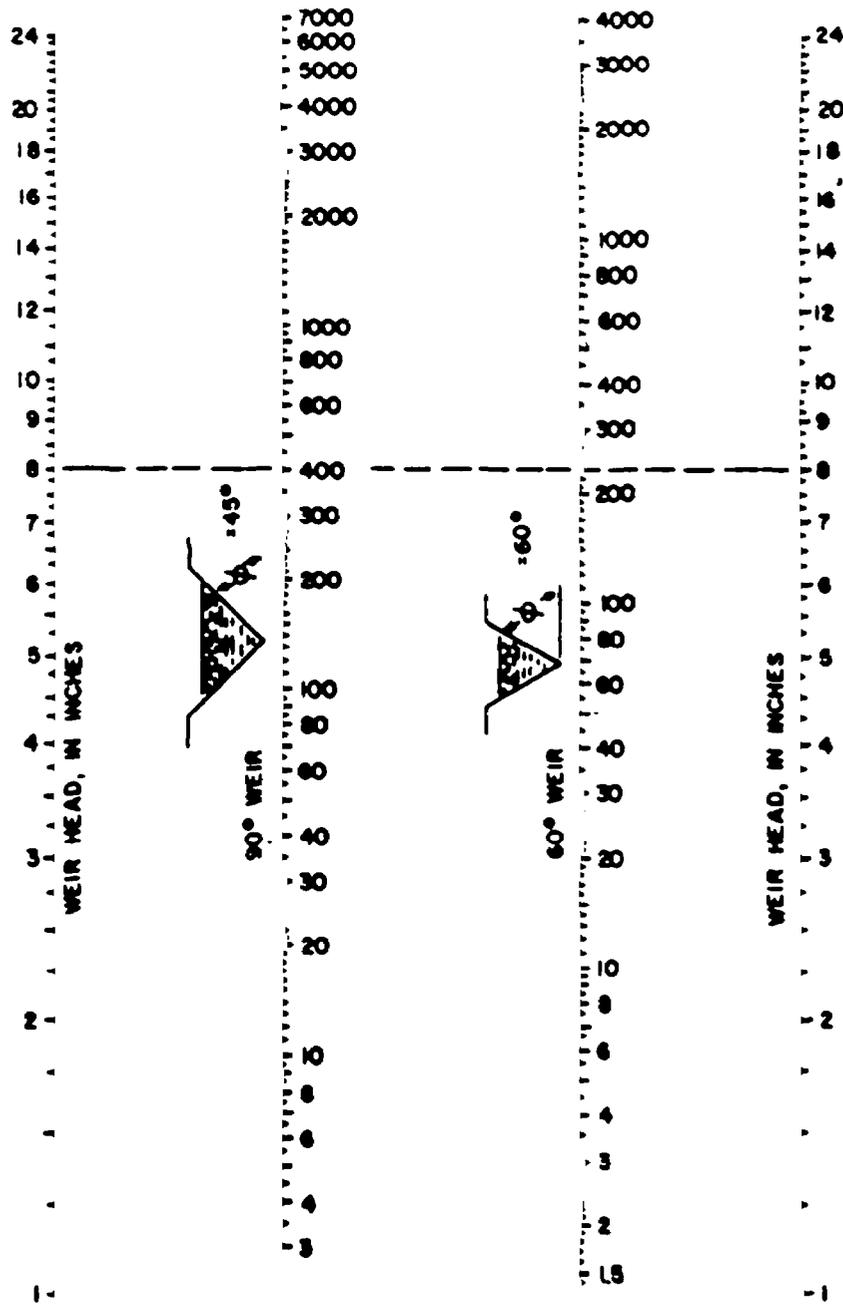
\*Selection of a formula depends on its suitability and parameters under consideration.

Table VI-2

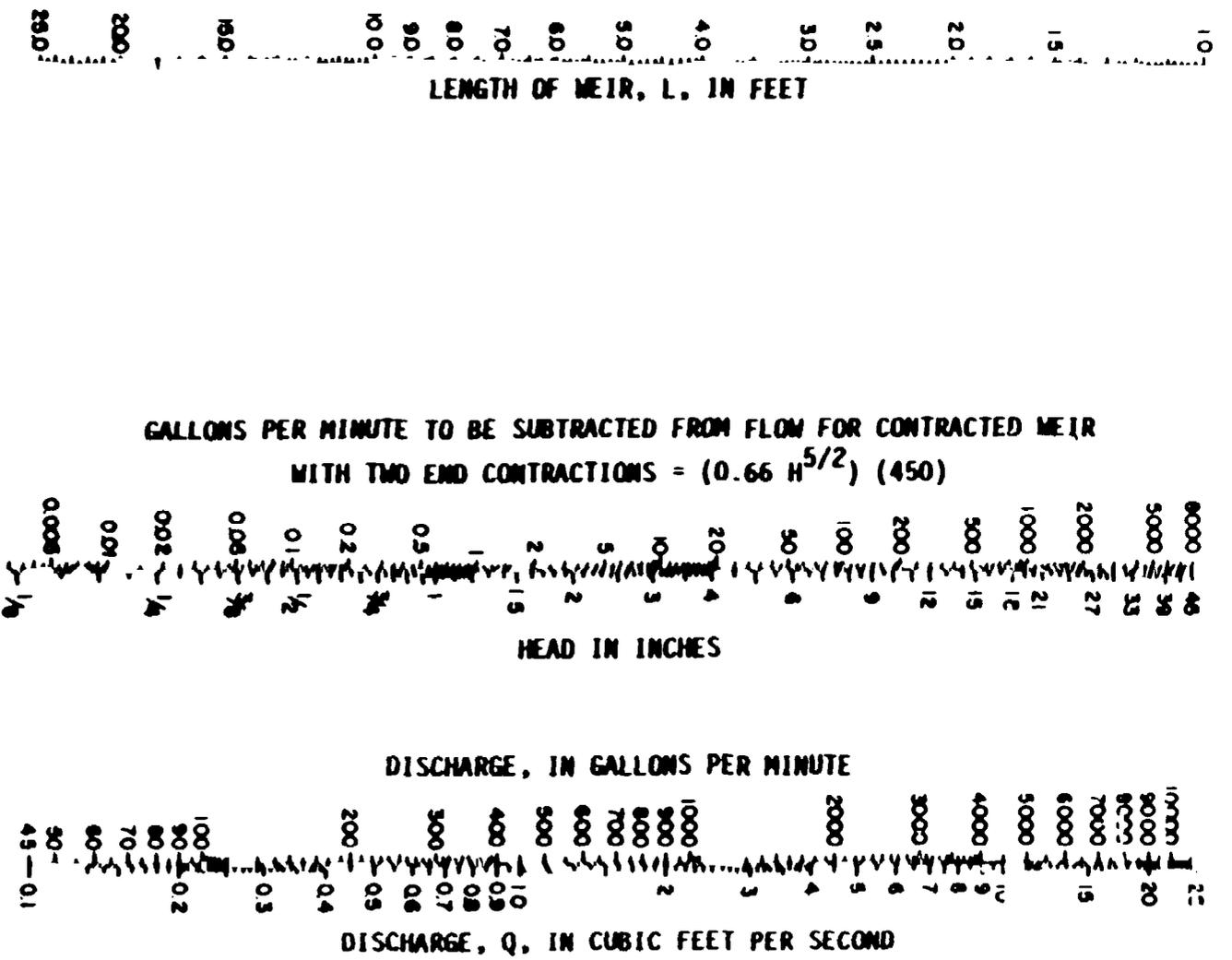
**Discharge of 90° V-Notch Weir -- Head Measured at Weir Plate**  
 (Taken from: NPDES Compliance Inspection Manual, EPA, May 1988)

<u>Head@</u> <u>Weir</u> <u>in</u> <u>Feet</u>	<u>FLOW</u> <u>RATE</u> <u>in</u> <u>CFS</u>	<u>HEAD@</u> <u>WEIR</u> <u>in</u> <u>FEET</u>	<u>FLOW</u> <u>RATE</u> <u>in</u> <u>CFS</u>	<u>HEAD@</u> <u>WEIR</u> <u>in</u> <u>FEET</u>	<u>FLOW</u> <u>RATE</u> <u>in</u> <u>CFS</u>
0.06	0.003	0.46	0.439	0.86	2.071
0.07	0.004	0.47	0.463	0.87	2.140
0.08	0.006	0.48	0.488	0.88	2.192
0.09	0.008	0.49	0.513	0.89	2.255
0.10	0.010	0.50	0.540	0.90	2.318
0.11	0.013	0.51	0.567	0.91	2.382
0.12	0.016	0.52	0.595	0.92	2.448
0.13	0.019	0.53	0.623	0.93	2.514
0.14	0.023	0.55	0.653	0.94	2.582
0.15	0.027	0.55	0.683	0.95	2.650
0.16	0.032	0.56	0.715	0.96	2.720
0.17	0.037	0.57	0.747	0.97	2.791
0.18	0.043	0.58	0.780	0.98	2.863
0.19	0.049	0.59	0.813	0.99	2.936
0.20	0.056	0.60	0.848	1.00	3.010
0.21	0.063	0.61	0.883	1.01	3.085
0.22	0.070	0.62	0.920	1.02	3.162
0.23	0.079	0.63	0.957	1.03	3.239
0.24	0.087	0.64	0.995	1.04	3.317
0.25	0.097	0.65	1.034	1.05	3.397
0.26	0.107	0.66	1.074	1.06	3.478
0.27	0.117	0.67	1.115	1.07	3.556
0.28	0.128	0.68	1.157	1.08	3.643
0.29	0.140	0.69	1.199	1.09	3.727
0.30	0.152	0.70	1.243	1.10	3.813
0.31	0.165	0.71	1.287	1.11	3.889
0.32	0.178	0.72	1.333	1.12	3.987
0.33	0.193	0.73	1.379	1.13	4.076
0.34	0.207	0.74	1.426	1.14	4.166
0.35	0.223	0.75	1.475	1.15	4.257
0.36	0.239	0.76	1.524	1.16	4.349
0.37	0.256	0.77	1.574	1.17	4.443
0.38	0.273	0.78	1.625	1.18	4.538
0.39	0.291	0.79	1.678	1.19	4.634
0.40	0.310	0.80	1.730	1.20	4.731
0.41	0.330	0.81	1.785	1.21	4.829
0.42	0.350	0.82	1.840	1.22	4.929
0.43	0.371	0.83	1.896	1.23	5.030
0.44	0.393	0.84	1.953	1.24	5.132
0.45	0.415	0.85	2.012	1.25	5.235

EQUATION  $Q = 3.01 H_w^{2.48}$  where  $H_w$  is head in feet at the weir and  $Q$  is in cubic feet per second



**Figure VI-1**  
**Flow Rates for 60° and 90° V-Notch Weirs**  
 (Taken from NPDES Compliance Inspection Manual, EPA, May 1988)



Note: Based on Francis weir formulas as follows:

$Q = 3.33 LH^{3/2}$  (for suppressed weir)

or  $Q = 3.33 (L - 0.2H)H^{3/2} - 0.66H^{5/2}$  (for contracted weir with two end contractions)

Where:

Q = discharge, in cubic feet per second

L = length of weir, in feet

H = head, in feet

Figure VI-2

Nomograph for the Capacity of Rectangular Weirs  
 (Taken from NPDES Compliance Inspection Manual, EPA, May 1988)

**Table VI-3**

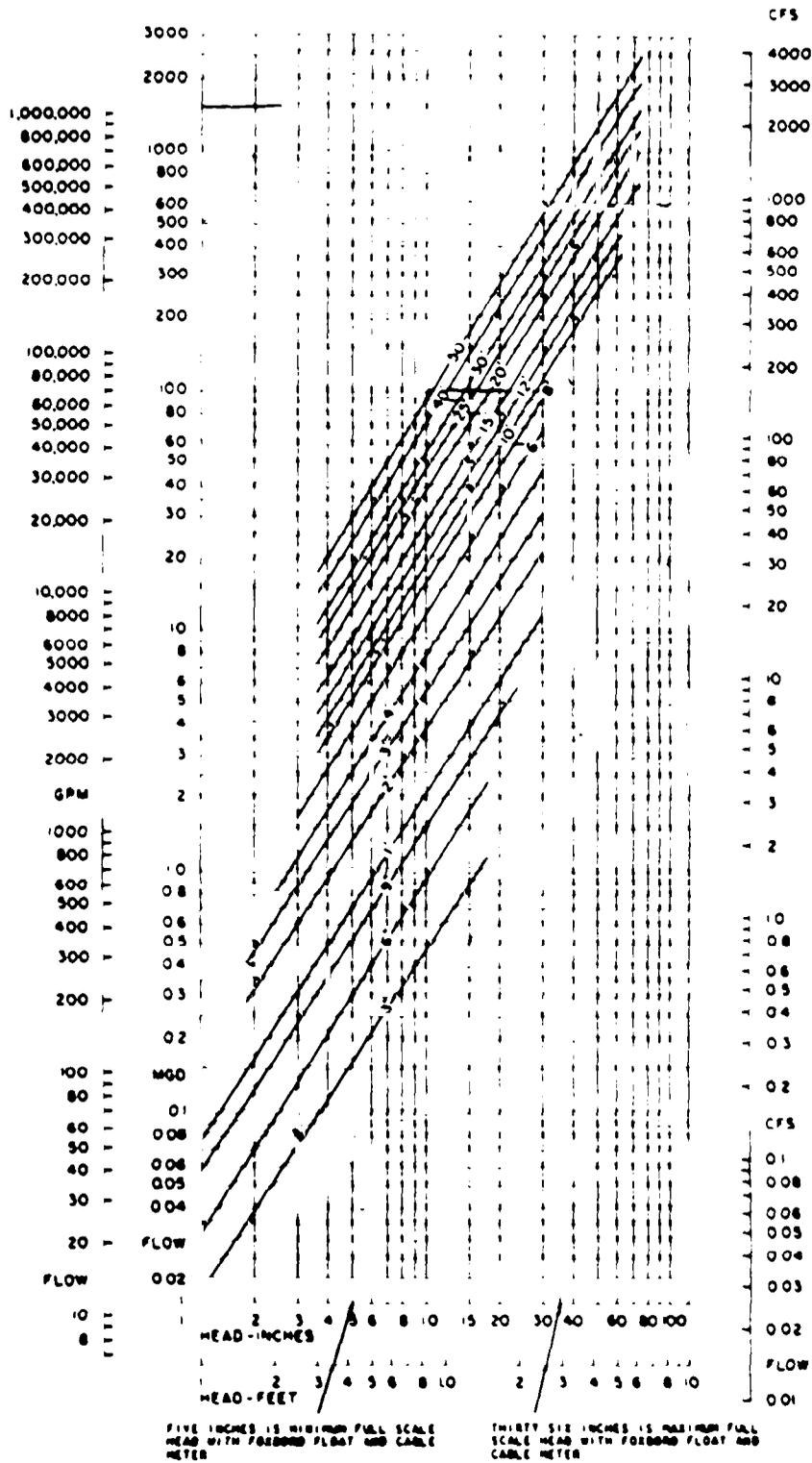
**Minimum and Maximum Recommended Flow Rates for Cipolletti Weirs  
(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)**

Crest Length, ft.	Minimum Head, ft.	Minimum Flow Rate		Maximum Head, ft.	Maximum Flow Rate	
		MGD	CFS		MGD	CFS
1	0.2	0.195	0.301	0.5	0.769	1.19
1.5	0.2	0.292	0.452	0.75	2.12	3.28
2	0.2	0.389	0.602	1.0	4.35	6.73
2.5	0.2	0.487	0.753	1.25	7.60	11.8
3	0.2	0.584	0.903	1.5	12.0	18.6
4	0.2	0.778	1.20	2.0	24.6	38.1
5	0.2	0.973	1.51	2.5	43.0	66.5
6	0.2	1.17	1.81	3.0	67.8	105.0
8	0.2	1.56	2.41	4.0	139.0	214.0
10	0.2	1.95	3.01	5.0	243.0	375.0

**Table VI-4**

**Minimum and Maximum Flow Rates for Free Flow Through Parshall Flumes  
(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)**

Throat Width, W	Minimum Head, Ha ft.	Minimum Flow Rate		Maximum Head, Ha ft.	Maximum Flow Rate	
		MGD	CFS		MGD	CFS
1 in.	0.07	0.003	0.005	0.60	0.099	0.153
2 in.	0.07	0.007	0.011	0.60	0.198	0.306
3 in.	0.10	0.018	0.028	1.5	1.20	1.86
6 in.	0.10	0.035	0.054	1.5	2.53	3.91
9 in.	0.10	0.059	0.091	2.0	5.73	8.87
1 ft.	0.10	0.078	0.120	2.5	10.4	16.1
1-1/2 ft.	0.10	0.112	0.174	2.5	15.9	24.6
2 ft.	0.15	0.273	0.423	2.5	21.4	33.1
3 ft.	0.15	0.397	0.615	2.5	32.6	50.4
4 ft.	0.20	0.816	1.26	2.5	43.9	67.9
5 ft.	0.20	1.00	1.55	2.5	55.3	85.6
6 ft.	0.25	1.70	2.63	2.5	66.9	103
8 ft.	0.25	2.23	3.45	2.5	90.1	139
10 ft.	0.30	3.71	5.74	3.5	189	292
12 ft.	0.33	5.13	7.93	4.5	335	519



**Figure VI-3**  
**Flow Curves for Parshall Flumes**  
 (Taken from NPDES Compliance Inspection Manual, EPA, May 1988)

W	A	$\frac{2}{3}A$		B	C	D	E	T	G	H	K	M	N	P	R	X	Y	Free-flow Capacity (Second-Foot <sup>3</sup> )													
		Ft. In.	Ft. In.															Mini- mum	Maxi- mum												
0 3	1	6 $\frac{3}{8}$	1	$\frac{1}{4}$	2	6	0	7	0	10 $\frac{3}{16}$	1-1 $\frac{1}{2}$	0	0	6	1	0	1	$\frac{5}{32}$	1	0	0	2 $\frac{1}{4}$	0	0	0	0	1	1 $\frac{1}{2}$	0.03	1.9	
0 6	2	$\frac{7}{16}$	1	4 $\frac{5}{16}$	2	0	1	3 $\frac{1}{2}$	1	3 $\frac{5}{8}$	2	0	1	0	2	0	0	0	0	3	1	0	4 $\frac{1}{2}$	2	11 $\frac{1}{2}$	1	4	2	3	0.05	3.9
0 9	2	10 $\frac{5}{8}$	1	11 $\frac{1}{8}$	2	10	1	3	1	10 $\frac{5}{8}$	2	6	1	0	1	6	0	0	3	1	0	6 $\frac{1}{2}$	3	6 $\frac{1}{2}$	1	4	2	3	0.09	8.9	
1 0	4	6	3	0	4	4 $\frac{7}{8}$	2	0	2	9 $\frac{1}{4}$	3	0	2	0	3	0	0	0	3	1	3	9	4	10 $\frac{3}{4}$	1	0	2	3	0.11	16.1	
1 6	4	9	3	2	4	7 $\frac{7}{8}$	2	6	3	4 $\frac{3}{8}$	3	0	2	0	3	0	0	0	3	1	3	9	5	6	1	0	2	3	0.15	24.6	
2 0	5	0	3	4	4	10 $\frac{7}{8}$	3	0	3	11 $\frac{1}{2}$	3	0	2	0	3	0	0	0	3	1	3	9	6	1	1	0	2	3	0.42	33.1	
3 0	5	6	3	0	5	4 $\frac{3}{4}$	4	0	5	1 $\frac{7}{8}$	3	0	2	0	3	0	0	0	3	1	3	9	7	3 $\frac{1}{2}$	1	0	2	3	0.61	50.4	
4 0	6	0	4	0	5	10 $\frac{5}{4}$	5	0	6	4 $\frac{1}{4}$	3	0	2	0	3	0	0	0	3	1	6	9	8	10 $\frac{3}{4}$	2	0	2	3	1.3	67.9	
5 0	6	6	4	4	6	4 $\frac{1}{2}$	6	0	7	4 $\frac{5}{8}$	3	0	2	0	3	0	0	0	3	1	6	9	10	1 $\frac{1}{4}$	2	0	2	3	1.6	85.6	
6 0	7	0	4	8	6	10 $\frac{3}{8}$	7	0	8	9	3	0	2	0	3	0	0	0	3	1	6	9	11	3 $\frac{1}{2}$	2	0	2	3	2.6	103.5	
7 0	7	6	5	0	7	4 $\frac{1}{4}$	8	0	9	11 $\frac{3}{8}$	3	0	2	0	3	0	0	0	3	1	6	9	12	6	2	0	2	3	3.0	121.4	
8 0	8	0	5	4	7	10 $\frac{1}{8}$	9	0	11	1 $\frac{3}{4}$	3	0	2	0	3	0	0	0	3	1	6	9	13	0 $\frac{1}{4}$	2	0	2	3	3.5	139.5	

\*Equals 1 cu. ft. per sec.

**LEGEND:**

W	Width of flume throat.	G	Axial length of diverging section.
A	Length of side wall of converging section.	B	Length of side wall of the diverging section.
$\frac{2}{3}A$	Distance back from end of crest to gage point.	K	Difference in elevation between lower end of flume and crest.
B	Axial length of converging section.	M	Length of approach floor.
C	Width of downstream end of flume.	N	Depth of depression in throat below crest.
D	Width of upstream end of flume.	P	Width between ends of curved wing walls.
E	Depth of flume.	R	Radius of curved wing wall.
T	Length of flume throat.	X	Horizontal distance to $H_g$ gage point from low point in throat.
		Y	Vertical distance to $H_g$ gage point from low point in throat.

Figure VI-4

Dimensions and Capacities of Parshall Flumes for Various Throat Widths  
(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)

provides Parshall flume dimensions for various throat widths and Table VI-4 provides the minimum and maximum flow rates for free flow through Parshall flumes.

For free, nonsubmerged flow in a Parshall flume of throat and upstream head ( $H_u$  in feet), the discharge relationship for flumes of 8 feet or less is given by the general equation:  $Q = CWH_u^n$ , where  $Q$  = flow. Table VI-5 provides the values of  $C$ ,  $n$ , and  $Q$  for different sizes (widths) of the Parshall flumes. Nomographs, curves, or tables are readily available to determine the discharge from head observations. Flow through a Parshall flume may also be submerged. The degree of submergence is indicated by the ratio of the downstream head to the upstream head ( $H_d/H_u$ ) which is the submergence ratio.  $H_d$  is the height of water measured above the crest. The flow is submerged if the submerged ratio is:

- Greater than 0.5 for flumes under 3 inches;
- Greater than 0.6 for flumes 6 to 9 inches;
- Greater than 0.7 for flumes 1 to 8 feet; and
- Greater than 0.8 for flumes larger than 8 feet.

If submerged conditions exist, the inspector should apply a correction factor to the free flow determined using the relationship  $Q = CWH^n$ . These correction factors are shown in Figure VI-5 for different sizes of the Parshall flume.

**Palmer-Bowlus Flume.** The Palmer-Bowlus flume is also composed of three sections: a converging upstream section, a contracted section or throat, and a diverging downstream section (see Figure 3-7). The upstream depth of the water (head) above the raised step in the throat is related to the discharge rate. The head should be measured a distance  $d/2$  upstream of the throat where  $d$  is the size (width) of the flume. The height of the step is usually unknown until the manufacturer's data are consulted, since it is difficult to manually measure the height of water above the step at an upstream point. The dimensions for Palmer-Bowlus flumes are not standardized as they are for Parshall flumes. Therefore, no standard flow equation exists. Instead, rating curves are provided by the manufacturers of these flumes to relate the head to the discharge rate.

The flume must be installed with a minimum channel slope downstream to maintain critical flow through the flume and prevent the flume from becoming submerged. A small jump or rise in the water surface below the throat indicates that critical flow through the flume has probably occurred and submerged conditions exist. Accurate flow measurements can usually be obtained with upstream depths that are up to 95 percent of the pipe diameter. Table VI-6 provides a table of the maximum slopes recommended for installation of Palmer-Bowlus flumes. The advantages of this type of flow measurements device are: easy installation, insignificant head loss, and self-cleaning.

**Table VI-5**  
**Free Flow Values of C and N for Parshall Flume**  
**Based on the Relationship  $Q = CWH_a^n$**   
**(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)**

Flume Throat, W		C	n	Max. Q cfs
1	in	0.338	1.55	0.15
2	in	0.676	1.55	0.30
3	in	0.992	1.55	1.8
6	in	2.06	1.58	3.9
9	in	3.07	1.53	8.9
1	ft	4 W *	1.522W <sup>0.026</sup>	16.1
1.5	ft	"	"	24.6
2	ft	"	"	33.1
3	ft	"	"	50.4
4	ft	"	"	67.9
5	ft	"	"	85.6
6	ft	"	"	103.5
7	ft	"	"	121.4
8	ft	"	"	139.5

Where:

W = Flume throat width

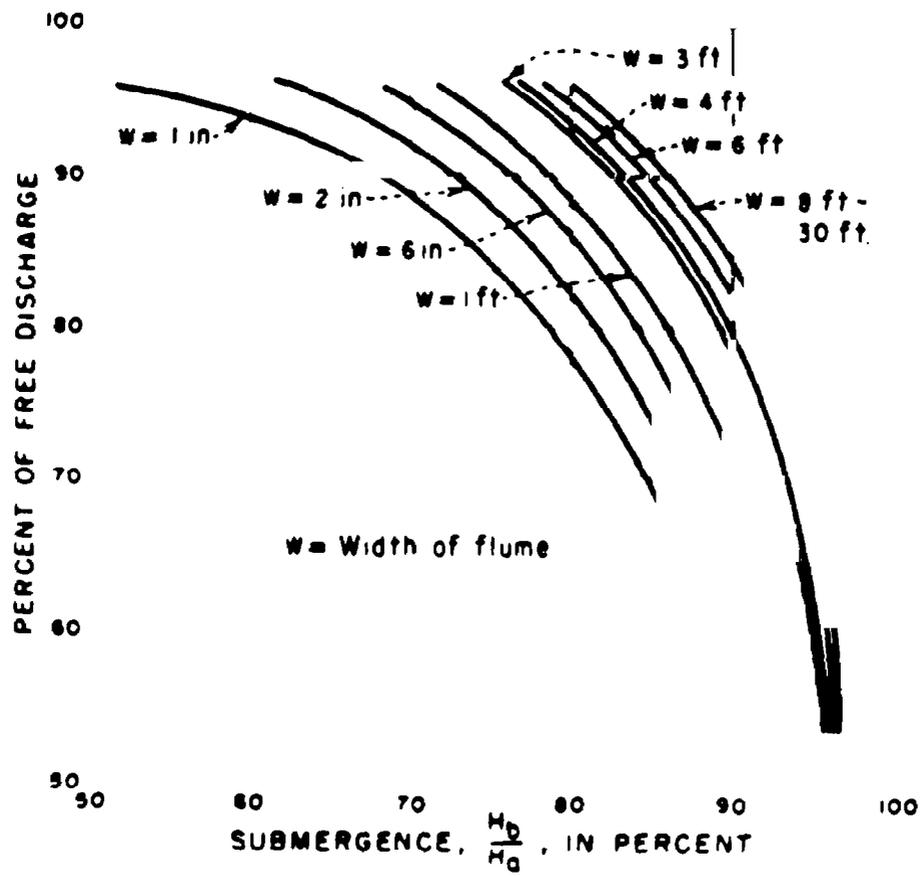
Q = Flow (cfs)

C = Constant

H = Head upstream of the flume throat (feet)

n = Constant

\* = W should be represented in feet to calculate C



**Figure VI-5**  
**Effect of Submergence on Parshall Flume Free-Discharge**  
 (Taken from NPDES Compliance Inspection Manual, EPA, May 1988)

Table VI-6

**Minimum and Maximum Recommended Flow Rates for Free Flow  
Through Plast-Fab Palmer-Bowlus Flumes  
(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)**

D Flume Size, (in.)	Maximum Slope for Upstream, Percent	Minimum Head (ft.)	Minimum Flow Rate		Maximum Head (ft.)	Maximum Flow Rate	
			MGD	CFS		MGD	CFS
6	2.2	0.11	0.023	0.035	0.36	0.203	0.315
8	2.0	0.15	0.048	0.074	0.49	0.433	0.670
10	1.8	0.18	0.079	0.122	0.61	0.752	1.16
12	1.6	0.22	0.128	0.198	0.73	1.18	1.83
15	1.5	0.27	0.216	0.334	0.91	2.06	3.18
18	1.4	0.33	0.355	0.549	1.09	3.24	5.01
21	1.4	0.38	0.504	0.780	1.28	4.81	7.44
24	1.3	0.44	0.721	1.12	1.46	6.70	10.4
27	1.3	0.49	0.945	1.46	1.64	8.95	13.8
30	1.3	0.55	1.26	1.95	1.82	11.6	18.0

**Venturi Meter.** The Venturi (differential pressure) meter is one of the most accurate primary devices for measuring flow rates in pipes. The Venturi meter is basically a pipe segment consisting of an inlet section, a converging section, a throat, and a diverging outlet section as illustrated in Figure 3-8. The water velocity is increased in the constricted portion of the inlet section resulting in a decrease in the static pressure. The pressure difference between the inlet pipe and the throat is proportional to the square of the flow. The pressure difference can easily be measured with great accuracy, resulting in an accurate flow measurement. One of the advantages of the Venturi meter is that it causes insignificant head loss.

The formula for calculating the flow in a Venturi meter is as follows:

$$Q = cKd_2^2 \sqrt{h^1 - h^2}$$

- Where: Q = volume of water, in cubic feet per second
- c = discharge coefficient, obtain from Table VI-5
- h<sup>1</sup> = pressure head at center of pipe at inlet section, in feet of water
- h<sup>2</sup> = pressure head at throat, in feet of water
- K = constant which relates d<sub>2</sub> to d<sub>1</sub> for Venturi meters. Values for K can be obtained from Table VI-8 or calculate according to the following formula:

$$K = \frac{1}{4} \sqrt{\frac{2g}{1 - [d_2/d_1]^4}}$$

- Where: d<sub>2</sub> = throat diameter, in feet
- d<sub>1</sub> = diameter of inlet pipe, in feet

**Electromagnetic Flowmeter.** The electromagnetic flowmeter operates according to Faraday's Law of Induction: the voltage induced by a conductor moving at right angles through a magnetic field will be proportional to the velocity of the conductor through the field. In the electromagnetic flowmeter, the conductor is the liquid stream to be measured and the field is produced by a set of electromagnetic coils. A typical electromagnetic flowmeter is shown in Figure 3-9. The induced voltage is transmitted to a converter for signal conditioning. The meter may be provided with recorder and totalizer using electric or pneumatic transmission systems. This type of flowmeter is useful at sewage lift stations and for measuring raw wastewater flow or raw or recirculated sludge flow.

Electromagnetic flowmeters are used in full pipes and have many advantages, including: accuracies of ±1 percent, a wide flow measurement range, a negligible pressure loss, no moving parts, and rapid response time. However, they are expensive and buildup of grease deposits or pitting by abrasive wastewaters can cause errors. Regular checking and cleaning of the electrodes is necessary. The meter electronics can be checked for proper operation with devices especially made for this purpose. The meter should be checked at least annually. The calibration of an electromagnetic flow meter can not be verified except by returning it to the factory or be the

**Table VI-7**  
**Coefficients of Discharge c for Venturi Meters**  
**(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)**

Diameter of Throat, in.	Throat velocity, ft. per sec									
	3	4	5	10	15	20	30	40	50	
1	0.935	0.945	0.949	0.958	0.963	0.966	0.969	0.970	0.972	
2	0.939	0.948	0.953	0.965	0.970	0.973	0.974	0.975	0.977	
4	0.943	0.952	0.957	0.970	0.975	0.977	0.978	0.979	0.980	
8	0.948	0.957	0.962	0.974	0.978	0.980	0.981	0.982	0.983	
12	0.955	0.962	0.967	0.978	0.981	0.982	0.983	0.984	0.985	
18	0.963	0.969	0.973	0.981	0.983	0.984	0.985	0.986	0.986	
48	0.970	0.977	0.980	0.984	0.985	0.986	0.987	0.988	0.988	

**Table VI-8**  
**Values of K in Formula for Venturi Meters**  
**(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)**

$\frac{d^2}{d^1}$	K								
0.20	6.31	0.33	6.34	0.46	6.45	0.59	6.72	0.72	7.37
0.21	6.31	0.34	6.34	0.47	6.46	0.60	6.75	0.73	7.45
0.22	6.31	0.35	6.35	0.48	6.47	0.61	6.79	0.74	7.53
0.23	6.31	0.36	6.35	0.49	6.49	0.62	6.82	0.75	7.62
0.24	6.31	0.37	6.36	0.50	6.51	0.63	6.86	0.76	7.72
0.25	6.31	0.38	6.37	0.51	6.52	0.64	6.91	0.77	7.82
0.26	6.31	0.39	6.37	0.52	6.54	0.65	6.95	0.78	7.94
0.27	6.32	0.40	6.38	0.53	6.54	0.66	7.00	0.79	8.06
0.28	6.32	0.41	6.39	0.54	6.59	0.67	7.05	0.80	8.20
0.29	6.32	0.42	6.40	0.55	6.61	0.68	7.11	0.81	8.35
0.30	6.33	0.43	6.41	0.56	6.64	0.69	7.17	0.82	8.51
0.31	6.33	0.44	6.42	0.57	6.66	0.70	7.23	0.83	8.69
0.32	6.33	0.45	6.43	0.58	6.69	0.71	7.30	0.84	8.89

**Table VI-9**

**Advantages and Disadvantages of Secondary Devices  
(Taken from NPDES Compliance Inspection Manual, EPA, May 1988)**

Device	Advantages	Disadvantages
Hook gauge	Common	Require training to use, easily damaged
Stage board	Common	Needs regular cleaning, difficult to read top of meniscus
Pressure measurement		
a. Pressure bulb	Since no compressed air is used, source can be linked directly to sampler	Openings can clog, expensive
b. Bubbler tube	Self-cleaning, less expensive, reliable	Needs compressed air or other air source
Float	Inexpensive, reliable	Catches debris, requires frequent cleaning to prevent sticking and changing bouyancy, and corroding hinges
Dipper	Quite reliable, easy to operate	Oil and grease foul probe causing possible sensor loss
Ultrasonic	No electrical or mechanical contact	Errors from heavy turbulence and foam, calibration procedure is more involved than for other devices

dye dilution method. Secondary devices are the devices in the flow measurement system that translate the interaction of primary devices in contact with the fluid into desired records or readouts. They can be organized into two types:

- Nonrecording type with direct readout (e.g., a staff gauge) or indirect readout from fixed points (e.g., a chain, wire weight, or float); and
- Recording type with either digital or graphic recorders (e.g., float in well, float in flow, bubbler, electrical, and acoustic).

The advantages and disadvantages of various secondary devices are provided in Table VI-9.

### ***Flow Measurement System Evaluation***

The current strategy for assessing compliance by industrial users in the Pretreatment Program depends heavily on the IU's submittal of self-monitoring data. When the POTW inspector is on-site, he or she should evaluate the IU's flow measurement system and techniques if there are any effluent limitations (local limits or categorical standards) which are mass-based. The flow measured during the compliance inspection should verify the flow data submitted by the IU as part of its periodic report to the POTW, support any enforcement action that may be necessary, and provide a basis for reissuing or revising the IU's permit.

The POTW inspector must check both the IU's flow data and the flow measurement system to verify the IU's compliance with mass-based categorical standards or local limits. When evaluating the flow measurement system, the POTW inspector should consider and record his or her findings on the following:

- Whether the system measures the entire discharge flow.
- The system's accuracy and good working order. This will include a thorough physical inspection of the system and comparison of the system's readings to those obtained with calibrated portable instruments.
- The need for new system equipment.
- The existence or absence of a routine calibration and maintenance program for flow measurement equipment.

If the IU's flow measurement system is accurate to within  $\pm 10$  percent, the inspector is encouraged to use the installed system. If the flow sensor or recorder is found to be inaccurate, the inspector should determine whether the equipment can be corrected in time for use during the inspection. If the equipment cannot be repaired in a timely manner, the portable flow sensor and recorder used to assess the accuracy of the IU's system should be used for the duration of the inspection. If nonstandard primary flow devices are being used by the IU, the IU should provide data on the accuracy and precision of the method being employed.

For flow measurement in pipelines, the inspector may use a portable flowmeter. The inspector should select a flowmeter with an operating range wide enough to cover the anticipated flow to be measured. The

selected flowmeter should be tested and calibrated before use. The inspector should select the site for flow measurement according to the permit requirements and install the selected flowmeter per the manufacturer's specifications. The inspector should use the proper tables, charts, and formulas as specified by the manufacturer to calculate the flow rate.

Four basic steps are involved in evaluating the IU's flow measurement system:

- Physical inspection of the primary device;
- Physical inspection of the secondary device and ancillary equipment;
- Flow measurement using the primary/secondary device combination of the IU; and
- Certification of the system using a calibrated, portable instrument.

In the following section, procedures are presented for inspecting the more common types of primary and secondary devices, for measuring flow using common permanent and portable systems, and for evaluating flow data. It must be emphasized that the number of primary/secondary device permutations is limitless. Therefore, it is not feasible to provide procedures for all system configurations. When system other than those discussed are encountered, the inspector is strongly encouraged to consult the manufacturers for advice before preparing a written inspection procedure.

### ***Primary Device Inspection Procedures***

The two most common open channel primary devices are sharp-crested weirs and Parshall Flumes. Common sources of error when using these devices include:

- Faulty Fabrication - The weirs may be too narrow or not "sharp" enough. Flume surfaces may be rough or critical dimensions may exceed tolerances or throat walls may not be vertical.
- Improper Installation - The weirs and flumes may be installed too near pipe elbows, valves, or other sources of turbulence. The devices may also be out of level or out of plumb.
- Sizing Errors - The primary device's recommended applications may not include the actual flow range.
- Poor Maintenance - The primary devices corrode and deteriorate and debris or solids may accumulate.

### ***Sharp-Crested Weir Inspection Procedures***

- Inspect the upstream approach to the weir
  - Verify that the weir is perpendicular to the flow direction.
  - Verify that the approach is a straight section of conduit with a length at least 20 times the maximum expected head of liquid above the weir crest.
  - Observe the flow pattern in the approach channel. The flow should occur in smooth stream lines without the velocity gradients and turbulence.
  - Check the approach, particularly in the vicinity of the weir, for accumulated solids, debris, or oil and grease. The approach must not have any accumulated matter.
- Inspect the weir
  - Verify that the crest of the weir is level across the entire conduit traverse.
  - Measure the width of the weir crest. The edge of the weir crest should be no more than 1/8-inch thick.

- Make certain the weir crest corresponds to zero gauge elevation (zero output on the secondary device).
- Measure the angle formed by the top of the crest and the upstream face of the weir. This angle must be 90 degrees.
- Measure the chamfer on the downstream side of the crest. The chamfer should be approximately 45 degrees.
- Visually survey the weir-bulkhead connection for evidence of leaks or cracks which permit by-pass.
- Measure the height of the weir crests above the channel floor. The height should be at least twice the maximum expected head of liquid above the crest.
- Inspect the weir for evidence of corrosion, scale formation, or clinging matter. The weir must be clean and smooth.
- Observe flow patterns on the downstream side of the weir. Check for the existence of an air gap (ventilation) immediately adjacent to the downstream face of the weir. Ventilation is necessary to prevent a vacuum that can induce errors in head measurements. Also ensure that the crest is higher than the maximum downstream level of water in the conduit.
- Verify that the nappe (see Figure 3-4) is not submerged and that it springs free of the weir plate.
- If the weir contains a V-notch, measure the apex angle. The apex should range from 22.5 degrees to 90 degrees. Verify that the head is between 0.2 and 2.0 feet. The weir should not be operated with a head of less than 0.2 feet since the nappe may not spring clear of the crest.

***Parshall Flume Inspection Procedures***

- Inspect the flume approach.
  - The flow pattern should be smooth with straight stream lines, be free of turbulence, and have a uniform velocity across the channel.
  - The upstream channel should be free of accumulated matter.
- Inspect the flume
  - The flume should be located in a straight section of the conduit.
  - Flow at the entrance should be free of "white" water.
  - The flume should be level in the transverse and translational directions.
  - Measure the dimensions of the flume. Dimensions are strictly prescribed as a function of throat width (see Figure 3-6 for critical dimensions).
  - Measure the head of liquid in the flume and compare with the acceptable ranges in Figure 3-6.
- Inspect the flume discharge
  - Verify that the head of water in the discharge is not restricting flow through the flume. The existence of a "standing wave" is good evidence of free flow and verifies that there is no submergence present.
  - Verify whether submergence occurs at or near maximum flow.

***Palmer-Bowlus Flume Inspection Procedures***

- Inspect the flume approach as outlined above (these flumes are seldom used for effluent flow measurement).
- Inspect the flume.
  - The flume should be located in a straight section of the conduit.
  - The flow at the entrance should be free of any "white water."
  - Observe the flow in the flume. The profile should approximate that depicted in Figure 3-7.
  - The flume should be level in the transverse direction and should not exceed the translational

slope in Table VI-1 at the end of the Appendix.

- Measure the head of water in the flume. The head should be within the ranges specified in Table VI-1.
- Inspect the flume discharge
  - Verify that free flow exists. Look for the characteristic "standing wave" in the divergent section of the flume.

#### ***Venturi Meter Inspection Procedures***

- Verify that the Venturi Meter is installed according to the manufacturer's specifications.
- Verify that the Venturi Meter is installed downstream from a straight and uniform section of pipe, at least 5 to 20 diameters, depending on the ratio of pipe to throat diameter and whether straightening vanes are installed upstream. (Installation of straightening vanes upstream will reduce the upstream piping requirements).
- Verify that the pressure measuring taps are not plugged.
- Calibrate the Venturi Meter in place by either the volumetric method or the comparative dye dilution method to check the manufacturer's calibration curve or to develop a new calibration curve.

#### ***Secondary Device Inspection Procedures***

Common sources of error in the use of secondary devices are:

- Improper Location - The gauge is located in the wrong position relative to the primary device.
- Inadequate Maintenance - The gauge is not serviced regularly.
- Incorrect Zero Settings - The zero setting of the gauge is not the zero point of the primary device.
- Operator Error - There is human error in the reading.

Specific inspection procedures follow.

#### **Flow Measurement in Weir Applications**

- Determine that the head measurement device is positioned 3 to 4 head lengths upstream of the weir.
- Verify that the zero or other point of the gauge is equal to that of the primary device.

The inspector should use an independent method of measuring head, such as with a yardstick or carpenter's rule (be sure to measure at least 4  $H_{max}$  upstream and convert to the nearest hundredth of a foot). To determine flow rate, use the appropriate head discharge relationship formula (see Table VI-2).

#### **Flow Measurement in Parshall Flume Applications**

##### ***Flow Measurement - Free-Flow Conditions***

- Determine the upstream head ( $H_u$ ) using a staff gauge.
  - Verify the staff gauge is set to zero head. A yardstick or carpenter's rule can be used for this.
  - Verify the staff gauge is at the proper location (two-thirds of the length of the converging section back from the beginning of the throat).
  - Read to the nearest division the gauge division at which the liquid surface intersects the gauge.
  - Read  $H_u$  in feet from the staff gauge.

- To determine the flow rate, use Table VI-3 in the units desired, or use tables published in flow management standard references, or calculate using the coefficients in Table VI-4.

#### ***Flow Measurement - Submerged Flow Conditions***

Generally, it is difficult to make field measurements with submerged-flow conditions. In cases when measurements can be obtained (using a staff or float gauge), the procedures listed below should be followed:

- Determine the upstream head using a staff or float gauge.
  - Read to the nearest division and, at the same time as for  $H_u$ , the gauge division at which the liquid surface intersects the gauge.
  - Calculate  $H_u$  from the gauge reading.
- Determine the downstream head ( $H_d$ ) using a staff or float gauge.
  - $H_d$  refers to a measurement at the crest.
  - Read to the nearest division, and at the same time as for  $H_u$ , the gauge division at which the liquid surface intersects the gauge.
  - Calculate  $H_d$  from the staff reading.
- Determine the flow rate
  - Calculate the percent submergence  $\left[ H_d/H_u \right] \times 100$
  - Consult Table VI-XXX
  - When a correction factor is obtained, use  $H_u$  and find the free-flow from Figure VI-XXX.
  - Multiply this free-flow value by the correction factor to obtain the submerged flow.

The inspector may use an independent method of measuring head, such as a yardstick or carpenter's rule at the proper head measurement point. Due to the sloping water surface in the converging section of a flume, it is essential that the proper head measurement point be used.

#### ***Flow Measurement in Palmer-Bowlus Flume Applications***

- Obtain head measurements as in the Parshall Flume application, using the secondary device. The head is the height of the water above the step. The total depth upstream of the step is not the head.
- Refer to the manufacturer-supplied discharge tables to convert head measurements to flow data. Palmer-Bowlus flumes, unlike Parshall Flumes, are not constructed to rigid dimensional standards. The inspector must not use discharge tables supplied by other manufacturers.

#### ***Verification***

Most flow measurement errors result from inadequate calibration of the flow, totalizer and recorder. If the inspector has determined that the primary device has been installed properly, verification of the IU's system is relatively simple. The flow determined from the inspector's independent measurement is compared to the flow of the IU's totalizer or recorder. The inspector's flow measurements should be within 10 percent of the IU's measurements to certify accurate flow measurement. Optimally, flow comparisons should be made at various flow rates to check the system's accuracy.

When the IU's permit requires daily average flow to be measured by a totalizer meter, the inspector should verify that the totalizer is accurate, i.e., properly calibrated. This can be done during a period of steady flow by reading the totalizer and at the same time starting a stop watch. The stop watch should be started just as a new digit starts to appear on the totalizer. After ten to thirty minutes, the totalizer should be read again, just as a new digit begins to appear, the stop watch is read. By subtracting the two totalizer readings, the total flow over the measured time period can be obtained. The flow rate in gallons per minute can be calculated by using the time from the stop watch. This flow rate should be compared to the flow determined by actual measurement of the head made at the primary device at the time interval. The calibration of the totalizer should be considered satisfactory if the two flows are within 10 percent of each other, when the actual measured flow is used as the known value, or divisor, in the percent calculation.

*Appendix VII*

*EPA's Policy on Split Samples*



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

JAN 21 1992

OFFICE OF WATER

MEMORANDUM

**SUBJECT:** Determining Industrial User Compliance Using Split Samples

**FROM:** Richard G. Kozlowski, Director *by Henry Heng*  
Enforcement Division

**TO:** Mary Jo M. Aiello, Acting Chief  
Bureau of Pretreatment and Residuals

This memo is a response to your letter of September 30, 1991, where you requested written clarification regarding the use of split samples for determining industrial user (IU) compliance under the Pretreatment Program. Specifically, you requested guidance on how to use the data from split samples for determining IU compliance in situations where split samples yield different analytical results. The fundamental question posed by your inquiry is whether all analytical results must be used when evaluating the compliance status of IUs and how to use those results for determining compliance. In situations where split samples exist and both samples were properly preserved and analyzed, POTWs should evaluate compliance with applicable Pretreatment Standards in the manner described below.

When evaluating the compliance status of an industrial user, the POTW must use all samples which were obtained through appropriate sampling techniques and analyzed in accordance with the procedures established in 40 CFR Part 136<sup>1</sup>. The Environmental Protection Agency (EPA) has consistently encouraged Publicly Owned Treatment Works (POTWs) to periodically split samples with industrial users as a method of verifying the quality of the monitoring data. When a POTW splits a sample with an IU, the POTW must use the results from each of the split samples.

A legitimate question arises, however, when a properly collected, preserved and analyzed split sample produces two different analytical results (e.g., one which indicates compliance and the other shows noncompliance, or where both indicate either compliance or noncompliance but the magnitudes are substantially different). In these instances, questions arise regarding the compliance status of the IU, and what should be done to reconcile the results.

<sup>1</sup> See Memorandum, "Application and Use of the Regulatory Definition of Significant Noncompliance for Industrial Users," U.S. EPA, September 9, 1991.

There is inherent variation in all analytical measurements, and no two measurements of the same analyte (even when drawn from the same sample) will produce identical results. When a split sample is analyzed using appropriate methods, there is no technical basis for choosing one sample result over the other for determining the compliance status of a facility. Since this is the case for all split samples which have been properly analyzed, the POTW should average the results from the split and use the resulting average number when determining the compliance status of an IU. Using the average of the two sample results avoids the untenable situation of demonstrating compliance and noncompliance from the same sample.

If the split sample produces widely divergent results or results which are different over a long period of time, then the cause of the discrepancy between the analytical results should be reconciled. When this happens, the POTW should investigate Quality Assurance and Quality Control (QA/QC) procedures at each laboratory involved. For example, the POTW could submit a spiked sample (i.e., a sample of known concentration) to the laboratories involved (preferably blind) to determine which laboratory may be in error.

In situations where one or both of the analytical results is determined to be invalid, there are compliance and enforcement consequences. If one of the analytical results is determined to be invalid, the average value for that sample is also invalid. In this situation, the value for this sample should be the value of the sample which was not determined to be invalid (e.g., if the IU's results are determined to be invalid, the POTW should use its sample for assessing compliance, and vice versa). If both samples are determined to be invalid, the averaged result from that sample should be discarded and not used for compliance assessment purposes. In either case, the POTW must recalculate the compliance status of the IU using all remaining valid sample results.

In summary, whenever split samples are taken and both are properly preserved and analyzed, the POTW should average the results from each sample and use the averaged value for determining compliance and appropriate enforcement responses. Where the sample results are widely divergent, the POTW should instigate QA/QC measures at each of the analytical laboratories to determine the cause of the discrepancy. If one or both of the samples are invalid, the POTW must recalculate the compliance status of the IU using all valid results.

If you have any further questions regarding these questions, please feel free to call me at (202) 260-8304. The staff person familiar with these issues is Lee Okster. Lee can be reached at (202) 260-8329.

cc: Cynthia Dougherty  
Regional Pretreatment Coordinators  
Approved State Pretreatment Coordinators  
Bill Telliard



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

APR 12 1993

OFFICE OF  
WATER

Mr. Harold R. Otis  
Chairman, Split Sampling Task Force  
Greater Fort Wayne Chamber of Commerce  
826 Ewing Street  
Fort Wayne, IN 46802-2182

Re: Using Split Samples to Determine Industrial User Compliance

Dear Mr. Otis:

In response to your letter of January 12, 1993, and your phone conversation of February 9, 1993, with Lee Okster, I am providing a further discussion of the issues surrounding the use of split samples to determine industrial user (IU) compliance with Pretreatment Standards. In your letter and your phone conversation, you requested clarification from the Environmental Protection Agency (EPA) on three issues. First, you requested a firm definition of what constitutes "widely divergent results" when comparing split sample results. Second, when a publicly owned treatment works (POTW) splits a sample with an IU, you inquired whether a POTW must use the industrial user's data to determine compliance with pretreatment standards. Finally, you requested written authorization from the EPA to incorporate the language from our existing guidance memorandum on split samples into the Rules and Regulations of the Water Control Utility for the City of Fort Wayne.

***What are Widely Divergent Results?***

As you are aware, the EPA issued a memorandum on January 21, 1992, entitled "Determining Industrial User Compliance Using Split Samples." The "widely divergent results" criterion established in this memo is to be used as an indication that a problem exists with the laboratory analysis. We did not include an indication of what constitutes "widely divergent" in our memorandum because the amount of "normal" analytical variability depends on the pollutant parameter being tested and the method being used to analyze the sample. With appropriate QA/QC, this "normal" analytical variability is small. In general, though, metals analyses have a smaller variation than organics analyses, but the magnitude of the variability depends on the pollutants being tested. Therefore, no hard and fast rules exist for determining what is widely divergent. This determination is left to the discretion of the local authority.

### ***Must the POTW Use All Sample Results?***

In the January, 1992, memorandum we state that "the POTW must use all samples which were obtained through appropriate sampling techniques and analyzed in accordance with the procedures established in 40 CFR Part 136." The memo further states "[w]hen a POTW splits a sample with an IU; the POTW must use the results from each of the split samples."

The POTW is required to sample the IU at least once per year to determine, independent of information supplied by the IU, the compliance status of that facility. If the POTW does not wish to be in a position of comparing its own data with the IU when it samples the IU's discharge, it is not required to split its samples with the IU. Furthermore, we do not recommend that the POTW use a split sample with the industry to satisfy its annual sampling requirement. The POTW should pull its own sample so that it has data which are truly independent of the IU's results.

The POTW also has the primary responsibility to ensure compliance by the IU with all applicable pretreatment standards and requirements. One way the POTW can satisfy its requirement to ensure compliance is to split a routine sample taken by the IU. If a POTW splits a routine sample taken by the IU, it must use the IU's data, in conjunction with its own, to determine the compliance status of the facility (assuming all of the data are sampled and analyzed appropriately). We encourage POTWs to split samples in this manner to verify the IU's data. In a similar fashion, if the POTW chooses to split its own sample with the IU, it must use all of the data to determine the compliance status of the facility (assuming all of the data are appropriately analyzed).

When the POTW splits a sample with an IU (whether it is a routine sample by the IU or an annual sample by the POTW) the POTW has the responsibility to determine whether the IU's results from the split sample are valid. Where an IU's results are different than the POTW's, the burden is on the IU to show that all preservation, chain-of-custody, and analytical and QA/QC methods were followed. If the IU cannot make this showing, then the analytical results from the IU should be discarded when determining the compliance status of the facility. If the IU establishes that it followed all appropriate procedures, then the POTW should review its own QA/QC program. If both the IU and POTW have followed appropriate procedures, and there is still a wide divergence, then follow-up sampling should be conducted. If follow-up sampling consistently shows IU noncompliance, or if the POTW is otherwise satisfied with the validity of its own results, it should proceed to follow its enforcement procedures.

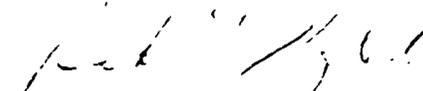
### ***Authorization From the EPA***

In regard to your final request, the City of Fort Wayne has the authority to incorporate these procedures into its Rules and Regulations without any authorization from the EPA. As long as the City has the minimum legal authorities to implement its

approved program, it has satisfied its requirements under the Federal regulations. As always, the City is encouraged to adopt the EPA's Pretreatment Guidance whenever possible.

I hope this letter responds to your questions and concerns. If you have any further questions, please feel free to call me at (202) 260-8304 or you can call Lee at (202) 260-8329.

Sincerely yours,



**Richard G. Kozlowski, Director  
Water Enforcement Division  
U.S. Environmental Protection Agency**

cc: Cynthia Dougherty  
Regional Pretreatment Coordinators  
Approved State Pretreatment Coordinators

*Appendix VIII*

*Compliance with Continuous Monitoring of pH*



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

MAY 13 1993

OFFICE OF  
WATER

Mary Jo M. Aiello, Chief  
Bureau of Pretreatment and Residuals  
Wastewater Facilities Regulation Program (CN 029)  
New Jersey Department of Environmental  
Protection and Energy  
Trenton, NJ 08625-0029

Dear Ms. Aiello:

Thank you for your letter of January 25, 1993, to Jeffrey Lape of my staff regarding the New Jersey Department of Environmental Protection and Energy's (the Department) proposed policy on waivers from pH limits applicable to industrial discharges to Publicly Owned Treatment Works (POTWs). Subject to the qualifications stated below, your proposed policy is consistent with the federal regulations.

Your letter relates to the application of 40 CFR 401.17, which allows facilities that employ continuous pH monitoring to exceed certain pH limits one percent of the time. Your letter correctly notes that 40 CFR 401.17 applies only to discharges to surface waters, but inquires whether an analogous policy could be applied to discharges to POTWs.

We believe an analogous policy could be applied to discharges to POTWs, subject to several restrictions. First, the federal pretreatment regulations contain a specific prohibition against discharges with a pH below 5.0, from which no waivers are allowed unless the treatment works is specifically designed to accommodate such discharges (40 CFR 403.5(b)(2)). Your letter correctly acknowledges that, except for such specifically designed treatment works, waivers below this minimum limit would not be consistent with federal regulations. Second, although federal pretreatment regulations do not include an upper pH limit applicable to all discharges, some categorical pretreatment standards do so. Waivers from the requirements of those categorical standards would not be allowed unless expressly permitted by the standards themselves.

Third, a POTW may not grant a waiver from a local limit if such waiver would cause pass through or interference. Since local limits are based on considerations at each POTW, it would not be appropriate to institute a waiver of local limits that applies statewide regardless of conditions at individual POTWs.

So long as POTWs act consistently with their obligations not to allow pass through or interference, however, they might implement waivers that apply either more or less frequently than the 1% you propose. Of course, if it wishes, the State could cap all waivers at 1% and thereby be more stringent than Federal law, which requires no cap.

We note that, if a POTW wishes to provide waivers from pH limits that are technically-based and are part of the POTW's Approved Pretreatment Program, the POTW will have to modify its Approved Pretreatment Program accordingly. The Department should consider for each POTW whether the adoption of this policy is a "change to local limits, which result in less stringent local limits" and therefore requires a formal modification under 40 CFR 403.18(c)(1)(ii), or whether it constitutes a clarification of the POTW's existing local limits.

I hope that this response addresses your concerns. If you have any questions or would like to discuss this further, please call me at (202) 260-5850 or Louis Eby at (202) 260-2991.

Sincerely,

  
Cynthia C. Dougherty, Director  
Permits Division

***Appendix IX***

***Example Standard Operating Procedure***

**STANDARD OPERATING PROCEDURES MANUAL  
PINELLAS COUNTY SEWER SYSTEM  
INDUSTRIAL MONITORING PROGRAM**

**JANUARY 1989**

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## 1.0 INTRODUCTION

The Pinellas County Sewer System (PCSS), Industrial Monitoring Program, is responsible for monitoring industrial discharges to the sewer system. There are four primary goals in industrial monitoring: to protect worker health and safety, to prevent inhibition to the wastewater treatment facilities (WWTF), to control the quality of effluent discharged from the WWTF, and to limit sludge contamination. This Standard Operating Procedures Manual (SOPM) explains the procedures used to collect samples of industrial wastewater, including all quality control (QC) protocols, equipment cleaning and maintenance, and safety considerations.

## 2.0 DOCUMENTATION

Documentation is an integral part of any pretreatment program. The validity of samples collected and data obtained both in the field and the laboratory is ensured through documentation and record keeping. All information must be complete and accurate. Record management and methodology must be consistent throughout the program to support the validity of data gathered. Failure to maintain records and documentation according to set procedures could result in these documents being inadmissible as evidence in court. In addition to the Field Data Record, which is the primary sampling information document, there are logs for equipment calibration, maintenance, chain-of-custody, and cleaning.

### 2.1 FIELD DATA RECORD

The Field Data Record includes sample site identification, type of sample, sampler and battery identification, settings on the sampler, results of field analyses, flow information (where applicable), and any additional information related to the site or effluent characteristics.

### 2.2 FIELD DOCUMENTATION LOG

The Field Documentation Log is used to record which sites are sampled each day, and any violations, conversations, or notable occurrences during the sampling event.

### 2.3 FIELD pH CALIBRATION LOG

The Field pH Calibration Log is used to record calibration of the field pH meter during the sampling event. The field pH meter is calibrated at each site prior to measuring the pH of the effluent. Calibration and slope are checked, adjusted as necessary, and recorded, along with the temperature of the buffer.

### 2.4 FLOW METER CALIBRATION LOG

The Flow Meter Calibration Log is used to record program information for the flow meter and water level calibration from the initial value shown on the meter to the actual measured water level.

## 2.5 pH METER CALIBRATION - LABORATORY

The laboratory maintains their own notebooks to record equipment calibration. When the laboratory pH meter is used, it must be calibrated as discussed in Section 7, and the results recorded in the (laboratory) pH Meter Calibration Log Book.

## 2.6 pH CALIBRATION/SPIKE CHECKLIST

The field pH meter is calibrated in the laboratory on a weekly basis. The buffers and internal fill solution are also changed at this time. Once a month, an EPA known sample for pH is checked after meter calibration in order to verify the accuracy of the pH meter. The pH Calibration/Spike Checklist is used to record the date and time of field pH meter calibration, calibration data, results and true value for the known sample, and to document the buffer and fill solution changes.

## 2.7 CHAIN-OF-CUSTODY FORM

When samples are brought in to the laboratory for analyses, they must be logged in and received by the laboratory personnel. The Chain-of-Custody Form includes sample collection information, types of analyses to be run, preservation, and a space for the laboratory personnel to sign with the date and time the sample was received by the laboratory. Any comments which may be important for the analysts to know prior to running the sample, or in reviewing the results of analyses, are also included on this form. A more detailed discussion of the Chain-of-Custody Form is presented in Section 3.5.

There will occasionally be samples collected which need to be sent to an outside laboratory for analyses, either for confirmation of a number, or for analysis of parameters which we cannot do in-house. The laboratory is responsible for filling out additional Chain-of-Custody Forms for sending shipments to an outside laboratory. The Chain-of-Custody Form (discussed above) will still be used by sampling personnel, with the addition of a statement that the samples are to be sent to an outside laboratory. The laboratory documents all shipment information on the Custody Form.

## 2.8 SAMPLE STORAGE FORM

When a sample is in violation of applicable limits, it is saved for evidence. The caps are sealed off with tape prior to long-term storage. The Sample Storage Form is used to record all samples which are currently being stored, as well as holding times and disposal dates for those samples.

## 2.9 ISCO CONTROL LOG

It is crucial to the integrity of the samples collected that all equipment used in sample collection be clean and free of contamination. Those parts of the sample equipment which do not come into contact with the wastewater are cleaned with soap and water for sanitary reasons. Any piece of equipment which will come into contact with the wastewater must be cleaned according to the procedures outlined in Section 5. The ISCO Control Log lists the ISCO number, the site at which the sampler was used, the date(s) used, the date cleaned, the cleaning method used, and the initials of the personnel cleaning the equipment.

## 2.10 CONTAINER CONTROL LOG

The container(s) used to collect the sample must also be cleaned thoroughly and that cleaning documented. Actual sample bottles are cleaned by the laboratory according to their quality assurance protocols. Composite and sequential bottles are cleaned by Program personnel, according to the procedures outlined in Section 5. The Container Control Log lists the container number, the site at which the container was used, the date(s) used, the date cleaned, the cleaning method used, and the initials of the personnel cleaning the container.

### 3.0 CHAIN-OF-CUSTODY

The overall success of a monitoring program depends on its capability to produce valid data through the use of accepted sampling procedures and protocol, and its ability to substantiate such data through documentation. This begins with properly trained personnel and continues with sampling preparation, the sampling event, transfer of sample custody, laboratory analyses, equipment cleaning and data management. The importance of this concept is realized when sampling data is used as evidence in court against non-compliant Industrial Users.

#### 3.1 FIELD DATA RECORD

The Field Data Record is a permanent record of the information gathered during the sampling round. An example of the Field Data Record is included as Form 1. The sheet should be accurate, legible and complete. INFORMATION RECORDED SHOULD NEVER BE FALSIFIED. A few points to consider:

- 1) When identifying the facility sampled, record the complete name of the industry and street address. This line also includes the Industry Login Code.
- 2) The description of the sample site location (point where sample is actually taken) should be concise.
- 3) The last name of each person from PCSS that is actively participating in the sampling round is recorded by that person for Day 1. If the same people are present the next day, they place their name or initials by Day 2. If a different person is present, he records his name by Day 2. It is preferable that sampling personnel remain consistent throughout the round to assure continuity.
- 4) Monitoring is considered unscheduled unless the industry is notified of a specific date and time that they will be sampled, in which case it would be a scheduled event. Surcharge sampling is marked as such. A sample would be considered a demand sample if it were taken in response to a complaint or an emergency situation. A sample will either be a grab, a composite, or a sequential sample.
- 5) When collecting samples for metals analyses, a deionized (DI) water blank is collected using the tubing and ISCO to be used at that site. This will confirm that the equipment was not contaminated at setup. The time collected is also noted.

PINELLAS COUNTY SEWER SYSTEM  
INDUSTRIAL MONITORING PROGRAM  
FIELD DATA RECORD

GENERAL DATA

Facility Sampled: \_\_\_\_\_ Login Code: \_\_\_\_\_  
 Facility Address: \_\_\_\_\_  
 Sample Site Location: \_\_\_\_\_  
 Persons Sampling: Day 1) \_\_\_\_\_ Day 2) \_\_\_\_\_  
 Type Monitoring:  Scheduled  Unscheduled  Demand  Surcharge  
 Grab  \_\_\_-Hr Composite  \_\_\_-Hr Sequential  
 Blank Lab Number: \_\_\_\_\_ Time Collected: \_\_\_\_\_  
 Sample Lab Number: \_\_\_\_\_

ISCO SETTINGS

- 1) ISCO I.D. #: \_\_\_\_\_ Actuator Used:  Yes
- 2) ISCO Battery #: \_\_\_\_\_
- 3) Sample Interval (minutes/gallons): \_\_\_\_\_
- 4) Suction line: Length (ft) \_\_\_\_\_ Diameter (in)  3/8"  1/4"
- 5) Mode:  Time  Flow
- 6) Volume Selector: Volume per sample \_\_\_\_\_ ml.; Head feet \_\_\_\_\_
- 7) Volume of Measured Grab: \_\_\_\_\_ ml.

TIME OF SAMPLING

Initial: \_\_\_\_\_:\_\_\_\_ Date: / /  
 Final: \_\_\_\_\_:\_\_\_\_ Date: / /  
 Volume Collected: \_\_\_\_\_ Liters. Number of Containers: \_\_\_\_\_

FLOW MEASUREMENTS

Flow Meter I.D. #: \_\_\_\_\_ Battery I.D. #: \_\_\_\_\_  
 Initial: \_\_\_\_\_ Time: : Date: / /  
 Final: \_\_\_\_\_ Time: : Date: / /  
 Total gallons used: \_\_\_\_\_

FIELD PARAMETERS

	pH	Temp. (C)	Time	Date	Initials
Initial				/ /	
Final				/ /	
Sample				/ /	

PRESERVATION/ANALYSIS

Dairies:  Chilled  Sulfuric Acid  
 Metals:  Nitric Acid (Cd, Cr, Cu, Pb, Ni, Ag, Zn, \_\_\_\_\_, \_\_\_\_\_, \_\_\_\_\_)  
 Cyanide:  Sodium Hydroxide  
 V. Organics:  Sodium Thiosulfate  
 Oil & Grease:  Sulfuric Acid  
 Other: \_\_\_\_\_

COMMENTS

Split sample received by, \_\_\_\_\_ Date / /

Duplicate Sample Collected:  Yes  No Lab Number: \_\_\_\_\_  
 Record Reviewed: \_\_\_\_\_

- 6) Each sample receives a lab number. This number, when complete, always has eight digits. The first two digits refer to the year, as in "87", the second two digits refer to the month, and the third pair to the day. The last two digits are assigned by the lab when the samples are checked in and are known as the sequence number. A sample collected on March 13, 1987, with sequence number 05, would read 87031305. If a field blank is collected, it is also assigned a lab number, and recorded on the Field Data Record.
- 7) It is important to record the ISCO ID number. If there is any question as to the cleaning procedure used or the cleanliness of the equipment, it can easily be traced back to the Cleaning Documentation Log.
- 8) ISCO settings must be recorded completely. In most cases, sample intervals will be set every 15 minutes for timed composites. Gallons are used for flow composites. Time or Flow must be indicated. If any settings are not applicable, they should be marked as such. "Volume of Measured Grab" is the actual volume collected when using the recorded ISCO settings.
- 9) Time of sampling is important to note. On a regular monitoring basis, 24-hour composite samples are collected after the last sample interval, and before the 24th hour. Example: Sample interval is every 15 minutes, and the initial sample is at 9:00 am. The sampler is to be pulled after the 8:45 am sample is collected. Note: This applies also to an 8-hour, 12-hour, 16-hour, etc. composite. Composite samples may be collected and composited over any time or flow interval, depending upon the purpose of the sampling.
- 10) When a flow meter is used, the time of flow meter readings and total flow is to be recorded along with the flow meter identification. Flow meter settings are recorded along with calibration information in the Flow Meter Calibration Log. If a flow meter is not used, this section is to be crossed out.
- 11) Data values resulting from analyses performed in the field are to be recorded accurately and neatly, and initialed after test completion. This area is extremely important, as pH and temperature violations could result in the issuance of citations.

- 12) The preservatives which are added to each sample container must be marked off, along with the parameters to be analyzed. If the preservative/parameter is not listed, it should be specified.
- 13) If an industry requests a "split" (a portion of our sample for their own analytical purposes), make sure to have the person receiving the sample sign and date the Field Data Record.
- 14) Any unusual occurrences should be recorded under the Comments section, along with the date, time and duration.
- 15) Any conversations with industry personnel that would be of interest are documented in the Field Documentation Log and referenced in the Comments section.
- 16) For every 10 samples collected, one duplicate sample is taken. If less than 10 samples are collected, one duplicate sample is still collected. When a duplicate sample is taken at a site, the lab number is recorded on the Field Data Record for that site.

It is important that all field records be completed using black waterproof pen. If errors are made, corrections should be made by drawing a single line through the error, and entering the correct information. Such corrections should be initialed and dated. Under no circumstances should a statement be crossed out so that the information is not legible.

### 3.2 EQUIPMENT DOCUMENTATION

Properly cleaned, maintained and handled equipment will help ensure the integrity of the Monitoring Program. Documentation is vital to support these efforts, especially in the event of litigation. Documentation is discussed in Section 2. Proper cleaning techniques should be implemented according to the analyses to be performed. This involves the ISCO head itself, tubing, sample containers and any other equipment that may come in contact with the wastewater discharge at the sampling point. Tubing is site specific to alleviate the possibility of cross contamination. For proper equipment cleaning techniques and use of sample containers see Section 5.

### 3.3 SAMPLE COLLECTION

Once a sample is collected, precautions must be taken to ensure sample validity and security. It is possible that the sample collected might be in violation and the data could be used in court. THIS SHOULD ALWAYS BE KEPT IN MIND.

Every sample collected, regardless of type, should be handled in the same manner. Once a sample is collected, the following procedures should be used:

- 1) Sample containers must be flushed with a portion of the effluent sample prior to filling. In the case of containers with preservatives, i.e., cyanide and volatile organic samples, this step is omitted.
- 2) Sample should be well agitated (to prevent settling) and then poured into the sample container, filling to about 1/2 inch from the top (See specific instructions for purgeable organic samples). If filling more than one sample container from a composite jug, make sure to shake the jug before each pour to prevent settling.
- 3) Field tests on a 24-hour composite sample are taken from the sample remaining after all sample containers are filled. If collecting a grab sample for laboratory analysis, field tests are taken on a separate portion of the same grab. The sample container is filled completely to the top, and then a small portion is poured into a separate container for field analysis.
- 4) Field tests on the initial and final grab samples that are collected when conducting 24-hour composite sampling are taken from grab samples collected immediately prior to the initial sample in the composite round and immediately after the final sample.
- 5) Sample containers should be labeled with:
  - Industry login code for site at which sample is collected.
  - Type of sample collected (grab or 24-hr composite).
  - Date sample collected, written as process number.
  - Type of preservative added to sample, i.e., HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>.
  - Initials of sampling personnel.
- 6) Sample should be properly preserved, and sample pH checked to confirm that pH is adjusted appropriately.
- 7) Sample should be placed on ice.

### 3.4 SAMPLE TRANSPORT

Once the samples are in the van, it is important to take every precaution to make sure that the samples are secure. When away from the van, make sure it is kept locked. If away for an extended period of time, the cooler should be taped shut and initialed in such a manner that tampering would be noted. The samples must be kept in sight, or in a secure place, at all times.

### 3.5 TRANSFER OF CUSTODY

When delivering the samples to the laboratory, they should be placed on the central counter by the sink. The Chain-Of-Custody Form should be filled out. When the Form is completed, the lab personnel must sign in the samples. At this point, the responsibility for the custody of the samples is transferred to the lab. An example of the Chain-of-Custody Form is included as Form 2, and an explanation of the information to be completed is provided below.

- 1) **SOURCE:** Type of location sample was collected from, i.e., industry, POTW, transmission system.
- 2) **COLLECTED BY:** Identification of personnel involved with sampling.
- 3) **DATE:** Date that sample was collected. If it is a 24 hour composite sample, the date the sampler was set up is the sample date.
- 4) **DELIVERED BY:** Personnel that delivered samples to the lab.
- 5) **DATE:** Date samples are signed in.
- 6) **TIME:** Time samples are signed in.
- 7) **PRESERVATIVE:** Circle letter for preservative(s) used.
- 8) **RECEIVED BY:** Lab personnel receiving sample accepts transfer by signing.
- 9) **DATE:** Date lab personnel receives sample.
- 10) **TIME:** Time that lab personnel signs in sample.
- 11) **PROCESS NUMBER:** This is a six digit number, the first two digits refer to the year, as in "87", the second two digits refer to the month, and the third pair to the day. A sample collected on March 13, 1987 would be process number 870313.

FORM 2

INDUSTRIAL WASTE PROGRAM  
CHAIN-OF-CUSTODY FORM

SOURCE: Industry  
COLLECTED BY:  
DELIVERED BY:  
RECEIVED BY:  
PROCESS NUMBER:

DATE: \_\_\_\_\_  
DATE: \_\_\_\_\_ TIME: \_\_\_\_\_  
DATE: \_\_\_\_\_ TIME: \_\_\_\_\_

PRESERVATIVE:  
Chilled C  
H<sub>2</sub>SO<sub>4</sub> H  
HNO<sub>3</sub> V  
NaOH S  
Other O

SEQUENCE NO.  
DESCRIPTION  
TIME  
TYPE  
PRESERVATIVE  
GROUP NUMBER

NO <sub>3</sub>	NO/1
Cl <sub>2</sub>	NO/1
TS <sub>2</sub>	NO/1
VS <sub>2</sub>	-P/1
FS <sub>2</sub>	-P/1
TS	-P/1
VE	NO/1
PS	NO/1
Turbidity	NO/1
CO <sub>2</sub>	NO/1
NO <sub>2</sub>	NO/1
Ca	NO/1
Cr	NO/1
Cl	-P/1
Pb	-P/1
Mn	NO/1
As	NO/1
Zn	NO/1
Cu	NO/1

Laboratory     
P1-14    Initial  
P1-14    Final  
P1-14    Composite  
Kit Number(s)

COMMENTS:

PAGE NO.

ccofom:890614

- 12) SEQUENCE NUMBER: This is a two digit number assigned by the lab which, combined with the process number, completes the sample lab number.
- 13) DESCRIPTION: Five-digit Industry login code.
- 14) TIME: Time sample is taken (use military time). If it is a 24-hour composite sample, put down times for the initial and final samples.
- 15) TYPE: Type of sample, i.e., grab or 24-hour composite.
- 16) PRESERVATIVE: Letter(s) indicating preservative(s) used.
- 17) PARAMETERS: Mark squares with a yellow felt tip marker, indicating tests to be run on sample. Blank spaces are provided to request additional test(s). This space is most commonly used when shipping samples to an outside laboratory.
- 18) Field pH information is transferred from the Field Data Record. This includes initial, final and sample pH.
- 19) Cyanide or other test kit numbers are to be placed directly under the column that they correspond to.

### 3.6 SAMPLING QUALITY CONTROL

Control checks should be performed during the sampling round to ensure proper sampling and cleaning techniques. These documented checks can also be used as supportive evidence in court.

A sample blank is run at each sample site, where metal or organic samples are collected, at the beginning of the sampling round as a QC check on the equipment being used at that site. DI water is pumped through the ISCO sampler and tubing, and preserved in the same manner as the sample. This ensures that any contamination of the equipment or lab chemicals will be detected. Blanks are not run for biological samples (BOD, solids).

Split samples are sometimes taken by industries that PCSS monitors. A split sample is a portion of a sample collected by PCSS which is given to the industry for testing. That portion of the sample is then analyzed by the industry or sent out to a private laboratory. Comparison of the data is useful in identifying discrepancies in analytical methodologies. Duplicate samples are also collected and analyzed for at least ten percent of the samples collected as a check on our laboratory.

## 4.0 SAFETY

There are many safety precautions which must be followed both at the office and in the field. Industrial monitoring, by its very nature, adds additional potentially hazardous situations to those existing in any field sampling situation. On the job safety can be broken down into three broad categories: cleaning, traffic, and sampling. Safety hazards associated with doing chemical analyses in the field are discussed in the Hach Portable Laboratory Procedures Manual.

### 4.1 CLEANING

The major hazard associated with cleaning sampling equipment and containers is the use of chemicals. Nitric acid, hydrochloric acid, and/or acetone are used in cleaning certain types of equipment. The hazards come from 1) mixing the acid/DI water solution, and 2) using the solution for cleaning.

Acid, when mixed with water, creates fumes which may be hazardous if inhaled, and can cause irritation to the eyes, nose and throat. Acid can cause skin burns if it is spilled on the skin. This can occur during mixing or cleaning. Upon working with acid, rubber gloves, a rubber apron and goggles should be worn. Acid solutions should always be prepared under the hood with the vent fan turned on. It is also advisable to wear rubber boots when cleaning, to protect your feet and lower legs from spilled acid, as well as to keep them dry.

NOTE : ALWAYS ADD ACID TO WATER - NEVER ADD WATER TO ACID.

Acetone is used to clean equipment that is used for collecting organic samples. It is extremely flammable and ignitable. Acetone is incompatible with nitric and sulfuric acids and should not be used where these acids are present. While the primary hazard is ignition, acetone is also a skin and mucous membrane irritant. Gloves and a respirator with organic vapor cartridges must be worn when working with acetone. Goggles must be worn to protect against any possible splashing.

PREVENT OR AVOID ALL IGNITION SOURCES.

Other hazards associated with cleaning are the potential for slipping on wet ground, and strains from lifting and moving equipment. Lifting and moving equipment will be discussed under sampling. Slipping, tripping or falling are problems which can occur during any activity, and can be prevented by wearing proper footwear (good fit and condition) and by using extra caution in areas of potential hazards.

#### 4.2 TRAFFIC

All County employees who are authorized to drive a County vehicle are required to take a driving safety course. In addition to following safe driving techniques, wearing seatbelts and observing traffic laws, there are other concerns related to working on or near the road and/or parking lots. The county requires that cones be placed in front of and/or behind the vehicle whenever it is parked. More cones may be advisable when parking in a heavily trafficked area. When parking on the road, hazard lights should be used in addition to cones. If an employee has to work on or near the road or where cars may pass in close proximity, a safety vest must be worn and the work area should be blocked off with cones and/or barricades. Before leaving the site, the area should be checked to ensure that everything was reloaded into the van, and a circle-of-safety check should be performed (walk around vehicle, look under, and check area).

#### 4.3 SAMPLING

Because sampling is conducted at industrial locations, often in confined or remote areas, there are so many potential hazards which must be recognized. Hazards associated with sampling include; working around unfamiliar chemicals/equipment; handling contaminated wastewater; lifting and moving equipment; and opening and/or entering manholes and flumes. A list of the safety equipment kept in the industrial van is included below:

acid-sill pads	barricades
eye-wash station	fire extinguisher
first aid kit	gas/vapor detector
goggles	hard hats
latex gloves	respirators
safety cones	safety vests

#### 4.3.1 Safety Clothing

Steel toe shoes or boots must be worn at all times when sampling. Steel toes are protection against manhole lids and flume doors or sampling equipment dropping on your feet. They can also be of aid in moving equipment or knocking a manhole cover back on. Steel toes are especially important when accessing an industrial facility's process or pretreatment area where you are unfamiliar with the layout, equipment used, and associated hazards.

If it is necessary to enter a manhole/flume, you must wear a hardhat to protect yourself against falling objects (manhole cover/flume door). A hardhat is also protection against injuries to the head from bumping into the walls or roof of the manhole or flume. A respirator with the appropriate cartridges should also be available anytime you enter a manhole/flume (see Confined Space Policy).

Gloves must be worn at all times when sampling. This is not only to protect yourself from contamination by the effluent being sampled, but also to protect the sample from contamination by oils or dirt on your hands.

Goggles may be worn to protect against splashing or fumes, if the situation warrants it.

#### 4.3.2 Opening Manholes

Opening manholes and flumes have some common hazards. Fumes and/or gases may accumulate within the manhole, and possibly could overcome someone opening the lid. The covers of manholes and flumes should always be opened slightly and allowed to vent prior to opening them completely. A vapor/gas detector probe should be inserted through the opening to check the quality of the air in the manhole/flume. If the probe indicates the presence of hazardous gases, a respirator should be worn while working near the manhole. The gas detector should be kept on as long as anyone is working near the manhole. Be sure to stand upwind of the opening so any releases will be blown away from you. Manhole covers are usually very heavy and unwieldy. Opening them can be dangerous. Possible injuries include: catching your hands or feet under the cover; hitting your legs or feet with the hook if it slips loose of the cover; falling in; or being overcome by fumes.

The proper procedure for opening a manhole is to first pry the manhole cover open slightly with the manhole hook. The manhole cover has a small indentation where the hook can be inserted and used to pry the cover up. Some manholes have a larger opening and you will be able to slide the hook completely under the edge of the cover. If not, use a screwdriver, alternating with the hook, to pry the lid up until you can slip the manhole hook all the way under. Use the hook to slide the manhole cover free of the rim, so that it is ajar. Test the quality of the air as discussed above. Allow the manhole to vent for several minutes before opening further. Use the hook to drag the cover off the manhole, making sure that your hands and feet are clear.

#### 4.3.3 Lifting and Moving

Do not attempt to lift or move anything that is uncomfortably heavy, i.e., causes you to strain yourself. Pinellas County policy requires that two people work together to lift anything that weighs over 50 pounds. Be sure to practice safe lifting procedures; bend your knees and use your legs and arms to support the weight, not your back.

## 5.0 CLEANING

ISCO samplers, composite and sequential collection containers, sample bottles, and suction tubing are cleaned specific to the parameters to be analyzed. Equipment which do not come into contact with the effluent (ISCO base and cover, flow meter, etc.) is either wiped off or washed with soapy water and rinsed well with tap water.

Cleaning must be done near a source of potable water (a spigot with a hose) and should be done in sunlight, when possible, to facilitate drying. ISCO sampler, tubing and container cleaning must be documented in the appropriate log books.

### 5.1 ISCO SAMPLER

Two types of ISCO samplers are used, Model 1680 and Model 2710. ISCO Model 2710 does not have a control panel plate, and does not use a discharge tube: the sample collection tubing extends all the way through the ISCO head into the base. The following procedures are used to clean the ISCO samplers:

- 1 ) Clean two 3-gallon polyethylene jugs, fill one jug with tap water and fill the other jug with soapy water.
- 2 ) The sampler should be completely assembled, i.e.: the base, head, and head cover are all connected, and a charged battery installed. Remove the head cover.
- 3 ) Leave the suction line attached to the sampler and wipe off the suction line with a sponge, using soapy water. Rinse the suction line off with tap water.
- 4 ) Place the end of the suction line into the jug containing soapy water. Turn the pump to "Forward", and pump at least 2 liters of soapy water through the sampler.
- 5 ) Remove the suction line from the soapy water. Place the end of the line into the jug containing tap water and thoroughly rinse (use at least 2 liters). Make sure that there is no detergent residue remaining in the tubing. Pull the end of the suction line out of the jug, turn the pump to "Reverse" (this purges the line) until the line is free of water, and then turn the pump to "Off".

- 6) Disconnect the suction line at the white polyethylene connector barbs. Wipe the barb connected to the sampler clean, or replace if necessary (there are extras in the van).
- 7) Disconnect the battery. With a soapy sponge, wash down the ISCO head and carefully rinse it, being sure not to get the control panel wet. Wipe off the control panel with a slightly damp paper towel or cloth, and then wipe it dry with a clean paper towel.

NOTE: THE CONTROL PANEL IS SENSITIVE TO MOISTURE AND OTHER THAN THIS MILD CLEANING, THE CONTROL PANEL MUST BE KEPT DRY AT ALL TIMES TO AVOID DAMAGE TO THE CIRCUITRY.

- 8) Remove the head from the base. Wash the base and head cover with soap and water. Rinse clean with tap water and let dry.
- 9) After all parts are dry, reassemble the sampler (base, head, control panel plate-black ISCOs only, head cover) and store in the shed.
- 10) If the ISCO sampler is to be used for collecting metals samples, follow the cleaning procedures outlined below for metals tubing.

## 5.2 TUBING

### 5.2.1 Dairies

Tubing to be used for dairy sampling (or other biological analyses) should be soaked in a composite jug with soapy water and bleach for at least 24 hours after use, or replaced if necessary. The tubing should then be cleaned with soap and water again, and thoroughly rinsed with tap water, followed by a DI water rinse.

### 5.2.2 Metals

Tubing to be used for metals or cyanide sampling should be acid washed and then rinsed with DI water as detailed below:

- 1) Get the styrofoam cooler from the shed that holds four 2-liter containers: two clear containers labeled "ACID" (1:3 HNO<sub>3</sub>:H<sub>2</sub>O) and two brown containers labeled "DI water". The cooler should also contain a length of tubing with a connector barb on one end. This tubing is used as an extension to the discharge tube/hose when cleaning.
- 2) In the laboratory, prepare the acid solution in one of the clear containers, filling it first with 1500 ml DI water, and then adding 500 ml HNO<sub>3</sub>. Fill both brown containers with DI water (you may want to fill an acid washed composite jug with DI water and use this to refill your brown DI water containers as they are emptied). Keep the DI water containers capped when they are not being used for rinsing to avoid acid contamination.

**CAUTION: USE EXTREME CARE WHEN WORKING WITH ACID. IT WILL BURN. GOGGLES, GLOVES, AND AN APRON SHOULD BE WORN AT ALL TIMES.**

- 3) Release the ISCO head from the base of the sampler and place it on one of the PVC stands. Attach the discharge tube extension to the discharge tube on the underside of the ISCO head and place the end of the extension into the empty acid container. Turn the pump to "Forward". Put the end of the suction line in the full acid solution container and pump the acid through the sampler. When all the acid has passed through, pull the end of the suction line from the acid container and hold it above the sampler head, allowing any residue in the tubing to be pumped through. Turn the sampler to "Off". Repeat this sequence. Cap both acid containers. Disconnect the discharge tube extension and store in the cooler.
- 4) Turn the pump to "Forward", then put the end of the suction line in one of the brown DI water containers. Pump two full containers of DI water through the sampler allowing it to run onto the ground. Hold the tubing above the sampler head as discussed above to ensure that the tubing is flushed completely through. Turn the sampler to "Off".
- 5) Disconnect the suction line at the white polyethylene connector barb, and connect the two ends of the suction line together. Hang the line in the storage shed until ready to use.
- 6) Re-assemble the ISCO and store in the shed.

- 7) Empty the used acid solution out in the laboratory sink; flush with copious amounts of water. Rinse the containers with DI water and cap. Store the empty acid and DI water containers in the styrofoam cooler. Replace the cooler in the storage shed.

### 5.2.3 Organics

Teflon tubing must be used for collecting organic samples, and should be cleaned according to the procedures outlined below:

- 1) Wash the exterior of the tubing with soap and water, and rinse thoroughly with tap water. Pump at least 2 liters of soapy water through the tubing, followed by a tap water rinse until no detergent residue remains.
- 2) Pump 2 liters of DI water through the tubing.
- 3) Pump an interference free redistilled solvent such as acetone or methylene chloride through the tubing; stop the sampler when the tubing is filled with the solvent. Cap the ends of the tubing and allow it to soak for 10 - 15 minutes. Flush the solvent through the tubing until it is completely purged. Place the tubing under the laboratory hood with the vacuum on and allow to dry thoroughly.

## 5.3 CONTAINERS

### 5.3.1 Composite Jugs

Composite jugs should be cleaned in the same parameter-specific manner as the tubing. "Dairy" jugs should be soap and water washed, soaked with soap and bleach for at least 24 hours, soap and water washed again, and rinsed out thoroughly with DI water.

"Metal" jugs should be acid washed with a 1:3 solution of  $\text{HNO}_3$ , and triple rinsed with DI water.

MAKE SURE ACID WASHED ITEMS ARE LABELED AND STORED AS SUCH.

### 5.3.2 Discrete Sample Bottles

Each set of discrete sample bottles consists of 28 500-ml bottles which are to be used when conducting sequential sampling. These bottles should be acid-washed as outlined below:

- 1) Soap and water wash, followed by a tap water rinse.
- 2) Rinse with a 1:1 solution of  $\text{HNO}_3$ , followed by a triple rinse with DI water.

### 5.3.3 Sample Containers

Sample containers used for collecting metal samples are prepared by the laboratory according to standard methods. Dairy and other biological samples are collected in cubitainers, which are rinsed with effluent prior to filling, and are disposed of after use. Cyanide and organic samples are collected in bottles which are supplied and prepared by an outside laboratory.

## 6.0 MAINTENANCE

### 6.1 ISCO SAMPLERS

Change desiccants weekly, or more frequently if needed (ISCO Model 1680). The desiccant face should be blue. If the blue is faded, or is turning to pink or white, it is time to Change the desiccant. Unscrew the desiccant cartridge from the sampler and replace the desiccant as discussed below:

- 1) Take the cartridges into the laboratory. Inside the laboratory oven, there are two beakers with colored desiccant. Using insulated gloves, take the beakers out of the oven.
- 2) Unscrew the top of the cartridge and pour the old desiccant into the least filled beaker. Pour fresh desiccant into the cartridge, screw the top on, and put the beakers, as well as the cartridge, back in the oven.
- 3) Remove the cartridge after the face is a rich blue color (approximately 5 minutes), let it cool, and screw it back into the sampler.

Other maintenance for the ISCO includes routine checks of the base, head and cover for cracks, and checking/repairing the seal on the control plate cover. The pump housing should be opened and lubricated once a month, and the tubing checked for cracks. The locks and cables should also be cleaned and lubricated to prevent rusting.

### 6.2 FLOW METER

Flow meter desiccants also need to be changed weekly. There are two plastic desiccant cartridges on the outside of the flow meter, and one metal desiccant cartridge inside the cover. Replacement metal cartridges are kept in the laboratory oven. The whole cartridge should be replaced - DO NOT OPEN. The desiccant from the exterior plastic cartridges should be emptied into the least filled beaker from the laboratory oven and refilled with fresh desiccant. DO NOT HEAT THE PLASTIC CARTRIDGES IN THE OVEN. Place the cartridges back in the flow meter.

### 6.3 BATTERIES

Batteries should be kept fully charged. The batteries should be kept hooked up to the chargers when not in use. Return the batteries to the chargers after each use. When a battery is plugged into a charger, it should be labeled with the data and time plugged in. Always use the batteries that have been charged for the longest time first. Keep batteries clean and dry.

### 6.4 pH METER

There are two types of pH meters used: Orion and Corning. The maintenance procedures for both meters are similar, but cleaning procedures should be checked in the appropriate pH probe manual prior to use.

The protective cap that comes with the pH electrode should be kept filled with pH 7 buffer between measurements. The reference buffer solutions in the field pH kit should be changed weekly.

The solution level in the pH probe should be maintained above the internal element at all times. The fill solution should be changed when erratic readings or slow response/stabilization is observed. After being refilled, the electrode should be allowed to soak in pH 7 buffer before being used again.

If the readings continue to be slow/erratic, the ceramic junction may be clogged. This may be tested by wiping off the electrode tip and observing it after an hour of air drying. A failure of KC1 crystals to appear at the junction indicates a clogged junction. If the junction is clogged, soak the pH probe in a warm solution of DI water. If the junction still does not function properly, it must be replaced (Corning meter only). Instructions for replacing the ceramic junction are presented in the pH meter manual.

Another cause of slow/erratic readings is build-up on the glass bulb or ceramic junction of the probe. This may cause interference in the measurements. Check the pH probe manual for proper cleaning solution depending upon the cause of the build-up.

## 7.0 CALIBRATION

### 7.1 FLOW METER

The flow meter should be calibrated at the sampling site as part of the setup activities. After programming the flow meter (see setup instructions), there are two calibrations which must be done. The bubbler tube should be adjusted to put out one bubble per second. The adjustment knob is located on the outside of the flow meter, near the bubbler tube outlet. The water level should also be checked and calibrated, if necessary. Set the meter readout to level. Check the actual water level in the flume. Adjust the flow meter level readout to match the actual water level.

### 7.2 pH METER

The pH meter is calibrated once a week in the laboratory. The pH meter is also calibrated in the field at each site, prior to taking a pH reading. The procedure for calibrating the meter is the same in the field and the laboratory, but the documentation varies, and the buffers are replaced during the weekly laboratory calibration.

#### 7.2.1 Field Calibration

##### 7.2.1.1 Corning pH meter

Rinse the pH probe and the thermometer with DI water, and shake the excess water off. Place the probe in buffer 7 and wait for the readout to stabilize. At the same time, check the temperature of the buffer, and adjust the pH meter temperature dial to that temperature. Once the meter has stabilized, write down the initial reading in the pH calibration log book. Also fill in the date, time, location, and buffer temperature. Calibrate the meter to 7.00 using the calibration dial, and record the reading in the log book. Turn the meter off. Remove the probe and thermometer and rinse with DI water. Shake the excess water off. Place the probe in buffer 4 or buffer 10 and allow to stabilize. Once the meter has stabilized, write down the initial reading in the log book. If the reading varies from 4.00 (or 10.00) by greater than 0.04, slope the meter to exactly 4.00 (or 10.00) using the small screwdriver in the slope hole at the bottom of the meter. Record the reading in the log book. If sloping was necessary, rinse off the probe and recheck the meter with buffer 7 as above, to be sure that the calibration held. If it did not, repeat the procedures above until the meter returns to 7.00 ( $\pm 0.02$ ).

### 7.2.1.3 Orion pH meter

At the beginning of each day, an overall meter check should be performed. With the shorting plug in, and no probes attached, turn the meter on with the "type of measurement" selector in the mV position. mV should read 0. How the selector to Temperature, and check that it is reading 25.0. If not, use the scroll button to correct the temperature and press Enter. Move the selector to pH 0.01. Press ISO, and check that the reading is 7.00. If not, correct as above and press Enter. Press the Slope button and check that the reading is 100. Correct as necessary. Press the Sample button and check that pH is reading 7.00 ±0.05. Correct as necessary. The meter is now ready for calibration.

Rinse the pH and temperature probes with DI water, and shake the excess water off. Place the probes in buffer 7. Move the selector to pH .01. Turn the pH motor on, and press the "ISO" button. The readout should show 7.00. Press the "CAL" button. The readout will flash from ".1." to the pH value. Wait for the readout to stabilize. Once the meter has stabilized, write down the initial reading in the pH calibration log book. Press the Enter button, and record the pH that the meter is calibrated to. Also fill in the data, time, and location. Remove the probes and rinse with DI water. Shake the excess water off. Place the probes in buffer 4 or buffer 10 and allow to stabilize. The readout will flash from ".2." to the pH value. Once the meter has stabilized, write down the initial reading in the log book. Press the Enter button, and record the sloped value in the log book. If buffer 4 or 10 were off by more than 0.1, rinse off the probes and recheck the meter with buffer 7 as above, to be sure that the calibration held. If it did not, repeat the procedures above until the meter returns to the calibrated value for buffer 7.00 (± 0.02). Move the selector to temperature, and record the buffer temperature in the log book.

### 7.2.2 Laboratory Calibration

Get the pH/Standard Calibration Check Sheet. Change the buffers in the field pH meter kit and mark the Check Sheet accordingly. Replace the internal fill solution in the pH probe. Calibrate the field pH meter as described above, logging the information onto the Check Sheet. This laboratory calibration should be done once a week, and/or before each sampling round to verify the accuracy and operation of the pH meter. Once a month, a pH sample with a known value is measured with each pH meter. This check confirms that the pH meter is functioning properly at pH values other than the buffers.

## 8.0 SAMPLING PREPARATION

Sampling preparation is the most important part of a successful sampling event. Careful attention must be given to both equipment and handling in order to collect a valid sample. Sampling site(s) and type(s) of samples will determine the equipment needed and the method of collection. Standard sampling checklists are included as Tables 1 and 2. Sampling preparation procedures are as follows:

Day before the sampling round:

- 1) Go over the sample checklist and the site specific data sheet to determine what will be used for each site.
- 2) Check the van to make sure it is properly stocked. Load into the van:
  - Properly cleaned ISCO samplers, site specific tubing and composite jug/sequential bottles;
  - Site specific equipment;
  - Proper sample containers (metals, cyanides, cubitainers, etc.); and
  - Flow meters, if needed.
- 3) Change the buffers and calibrated the pH meter.

Day of the sampling round:

Load into the van:

- Charged batteries, one for each ISCO plus a backup,
- Ice for ISCO bases (approx. 1/2 cooler per sampler),
- Ice to chill samples (if picking up samples that day).

TABLE 1  
DAIRY SAMPLING CHECKLIST

Data:

Sites to be sampled:

ISCO Sampler(s) #  
battery(s) #  
desiccant(s) in good condition  
site specific tubing with weights  
12 liter composite container(s) (dairy)

ISCO Flow Meter(s) #  
battery(s) #  
desiccant(s) in good condition  
program box

pH meter calibrated w/fresh buffer  
cooler(s) w/ice #  
cubitainers for samples #  
DI water (lowboy)  
latex gloves  
Sulfuric acid (for acid kit)  
field data sheets  
field notebook  
grab sampler  
ISCO stand  
camera with film

TABLE 2  
METAL/CYANIDE SAMPLING CHECKLIST

Date:

Sites to be sampled:

- \_\_\_ ISCO Sampler (composite or discrete) #
- \_\_\_ battery(s) #
- \_\_\_ dessicant(s) in good condition #
- \_\_\_ actuators #
- \_\_\_ site specific tubing
- \_\_\_ serving plate base
- \_\_\_ discrete bottles (glass or plastic) #
- \_\_\_ 12 liter composite container(s) (acid-washed) #
  
- \_\_\_ ISCO Flow Meter(s) #
- \_\_\_ battery(s) #
- \_\_\_ dessicant(s) in good condition
- \_\_\_ program box
  
- \_\_\_ pH meter calibrated w/fresh buffer
- \_\_\_ DI water for blanks
- \_\_\_ cooler(s) w/ice #
- \_\_\_ acid-washed bottle(s) for blanks #
- \_\_\_ acid-washed bottle(s) for samples #
- \_\_\_ DI water (lowboy)
- \_\_\_ latex gloves
- \_\_\_ Nitric acid (for acid kit)
- \_\_\_ field data sheets
- \_\_\_ field notebook
- \_\_\_ grab sampler
- \_\_\_ ISCO stand
- \_\_\_ camera with film
  
- \_\_\_ cyanide kit(s) #
- \_\_\_ Sites to be tested:

## 9.0 TYPES OF SAMPLES/METHODOLOGY

There are two basic types of samples: grab and composite. The method of collection is determined by flow, sampling location and analyses to be performed. Obtaining a representative sample should be of major concern.

### 9.1 GRAB SAMPLES

A grab sample is defined as "an individual sample collected over a period of time not exceeding 15 minutes". A grab sample is collected when:

- 1) Setting up a sampler is not feasible due to flow or site arrangement.
- 2) There is unusual flow of short duration.
- 3) The flow is not continuous (batch discharge).
- 4) Waste characteristics are relatively constant.
- 5) Analytical parameters require a grab sample: i.e., pH, cyanide, organics, oil and grease, sulfides, temperature, and residual chlorine.

A grab sample can be taken either manually or with an automatic sampler. To obtain a manual grab sample; a clean grab container, sample container, and a sampling pole are needed. The grab and sample containers must be properly cleaned according to test specifications. The grab container is attached to the pole and lowered into the wastestream. The sample container is then rinsed with the effluent to be sampled. This is known as "seeding". The container is then filled with effluent. Several grabs might be needed to fill the sample container completely. It is important to remember that in order for a sample to be considered a grab, it must be collected within a 15 minute time frame.

An automatic sampler is used to obtain a grab sample where pipes or "cleanouts" are used as sampling sites. In most cases, the diameter of the pipe prevents easy access, allowing only a tube to be placed into the flow. To obtain a grab sample in this manner, follow procedures for obtaining a pH grab sample for a 24-hour composite with an automatic sampler. See Section 11 for instructions on setting up the equipment.

## 9.2 COMPOSITE SAMPLES

A composite sample is made up of a number of individual grab samples which are combined based on either time or flow. A time composite sample consists of equal volume grab samples collected at equal time intervals. A flow composite sample may consist of equal volume grab samples taken at varying time intervals; samples of variable volume (in proportion to flow) collected at equal time intervals; or one sample continuously collected proportional to flow. A composite sample is taken when:

- 1) Determining average pollutant concentrations during the compositing period.
- 2) Calculating mass/unit time loadings.

The use of an automatic sampler with a composite base simplifies implementing this type of collection. In the event of a timed composite, the sampler can be programmed with the desired time interval. For a flow proportional composite, a flow meter can be used in conjunction with the sampler (provided that the sampling site is constructed for this type of sampling). In the event of industrial batch discharges at varying time intervals, an actuator should be used. See Section 11 for instructions on rrating up the equipment.

If an automatic sampler is not available, grab samples can be collected and manually composited. (NOTE; IN ANY MANUAL COMPOSITING METHOD. SAMPLE MANIPULATION SHOULD BE MINIMIZED TO REDUCE THE POSSIBILITY OF CONTAMINATION).

A second type of compositing is known as "sequential" sampling. This type of compositing consists of a series of individual grab samples collected into different bottles. The ISCO sequential base holds 28 500-ml bottles. Depending on sample volume, from one to four grab samples can be composited into each of the bottles, at time intervals programmed into the sampler. This type of sampling is valuable in isolating discharge values that may vary over a period of time or from one batch discharge to another. It is also useful in situations where chemical constituents at a particular time are of interest.

If sequential sampling is to be done manually, procedures for grab sampling can be followed. An automatic sampler may also be used with a sequential base (ISCO Model 1680 only). See Section 11 for instructions on setting up the equipment.

## 10.0 FIELD ANALYSES

Two types of measurements are routinely performed in the field: pH and temperature. These tests are done in conjunction with one another on the initial and final grabs, and on the composite sample during the 24-hour composite sampling event. The pH and temperature are also measured on a portion of the grab sample during a grab sampling event. Other analyses may be conducted in the field for special investigations/projects. These analyses are performed using a Hach portable laboratory.

During composite sampling, the "pH grab" is usually collected in a graduated cylinder at the beginning and end of the sampling want. The cylinder should be rinsed out three times with DI water, and then seeded with effluent before collecting the pH grab. The pH can then be measured directly from the cylinder.

The pH of a grab sample is measured off of a separate portion of the sample. The grab container should be seeded with effluent before being used to fill the sample container; the sample container should also be seeded with effluent before filling. The sample container should be filled to the top, and then a portion of the sample should be poured into a separate 25-ml container for pH measurement. The cylinder or pH container should be rinsed out three times with DI water after the pH grab is analyzed and disposed of.

The pH meters used in the field are stored in cases which contain the following supplies:

- 1) 1 pH meter.
- 2) 1 pH probe.
- 3) 1 thermometer or temperature probe.
- 4) Buffers - 4, 7 and 10.
- 5) 1 slope adjustment screwdriver (Corning only).
- 6) A supply of paper towels.

To obtain a pH measurement, the following procedures are used

- 1) Connect the probe(s) to the meter.
- 2) Remove the cap from the pH probe; rinse the probe and the thermometer (or temperature probe) with DI water.
- 3) Calibrate the meter following instructions in Section 11.

- 4) After meter calibration, rinse the pH probe and the thermometer/temperature probe with DI water and place in the sample solution.
- 5) Turn the meter on to start stabilization; read the thermometer and make the temperature adjustment (Corning only). Record the temperature on the Field Data Record. When using the Orion meter, it is generally faster to check the pH first, and check the temperature afterwards (switch steps 5 and 6).
- 6) Let the pH reading stabilize; remember to periodically stir the sample with the probe. Record the results on the Field Data Record.
- 7) If the pH value is out of compliance, recheck the buffer 7 solution to verify proper calibration.
- 8) Rinse the pH probe and the thermometer/temperature probe with DI water and store properly.
- 9) Pour the pH sample back into the wastestream, unless the pH value is out of compliance. If the pH value is out of compliance, take the sample in to the laboratory/contact person so that they may check the pH value themselves.

## 11.0 EQUIPMENT SETUP

### 11.1 ACTUATOR

At some sites where there is intermittent flow, it may be advisable to use an actuator. The actuator has an open electronic circuit, which is "closed" by the water when there is flow. When the circuit closes, it sends a signal to the sampler to collect a sample. Once the programmed time interval between samples passes, the ISCO is in the "ready" position and waits for a signal from the actuator. If there is flow at that time, the circuit will be closed; a signal will be sent and a sample will be collected. If there is no flow, the ISCO will remain in the "ready" position until the actuator circuit is again closed by the recurrence of flow. To use the actuator:

- 1) Connect the actuator and the sampling hose to a firm support (such as a short piece of PVC pipe) so that the end of the actuator is resting on the bottom of the manhole or flume floor in a vertical position. Be sure the hole in the side of the plastic cap at the end of the actuator is not covered and is open in the direction affluent flow will be coming from (facing upstream). The tubing should be positioned so that the end of the tubing is slightly above the floor (about 1/4-inch).
- 2) Attach the actuator to the battery by putting the control box clamp over the top of the battery and pressing down firmly until it clamps on.
- 3) Set the switch on the actuator control block to "Toggle/Reset".
- 4) Set the ISCO switch to time (not flow).
- 5) Plug the actuator into the flow meter socket on the ISCO head next to the battery socket; you are ready to start sampling or proceed with the ISCO setup.

### 11.2 FLOW METER

The settings on the ISCO flow meter will vary with site characteristics. Be sure you have the programming data for your site, or the flow meter manual which has all the program settings.

Settings for sites which we currently monitor using a flow meter are listed in front of the Flow Meter Calibration Log. To set up the flow motor:

- 1) Hook up the battery.
- 2) Hook up the bubbler tube.
- 3) Set the program disk by inputting the device number (based on width of flume and maximum expected depth of flow) and the scaling constant (determined by the device number). Use the scaling constant for gallons per minute. The scaling constant equals, the flow at the maximum expected depth, and is expressed in the format "#.##". If the maximum flow is greater than 9.99 or less than 1.00, an exponent will be included in the scaling constant (i.e., #.## ± 1).
- 4) Place the program disk into the flow meter.
- 5) Turn the flow meter on.
- 6) Adjust the bubble rate from the bubbler tube to one bubble per second.
- 7) Adjust/calibrate the flow level. Set the meter readout to level. Measure the actual flow level. Unlock the calibration knob and adjust the level on the readout to match the actual flow level. Relock the calibration knob.
- 8) Set the recorder mode/span. This determine the span (total level variation) that will show up on the recorder paper. Generally, you want the average flow level to be near the center of the page.
- 9) Set the flow meter to purge every 60 minutes, except in a flow with lots of fats or solids, than set it for 30 minutes. "Purge" causes a burst of air to be blown out through the bubbler tube, thereby clearing any obstructions from the and of the bubbler tube.
- 10) The sampler initiation signal sends a signal a to the ISCO for way 10, 100, or 1000 gallons of effluent that flow part the bubbler tube. The ISCO will be set to take a sample after it receives a certain number of signals. For example, the flow meter signals every 10

gallons, and the ISCO samples every 10 signals, equaling one sample every 100 gallons, or it can signal every 100 gallons and sample every signal equaling one sample every 100 gallons. If there is an exponent in the scaling constant (+/-), multiply/divide the sampler initiation signal by that exponent. For example, if there is an exponent of +1, setting the sampler initiation signal at 10 will result in a signal being sent every 100 gallons. The flow reading on the total flow dial is also to be multiplied/divided by the exponent in the scaling constant.

- 11) Set the chart speed at one inch per hour.
- 12) On the display knob, the bottom setting is calibrate recorder 0. This will move your recorder pen to the bottom of the page. Manually move the paper and see where the pen writes. If it is not on the 0 line, unscrew the knob holding the pen and adjust the pen's position up or down. Recheck this until the pen lines up evenly on 0. Relock the knob holding the pen. Reset the display knob to level.
- 13) Hook up the connect cable to the flow meter and to the ISCO sampler.

### 11.3 WATER METERS

Water meters are used to estimate the flow at facilities where effluent flow measurement is not practical or possible. Flow data is necessary in order to calculate contaminant/organic loading based on measured concentrations; this data is then used as a basis for surcharge fees, mass balance equations, and/or the combined wastestream formula.

Water meter readings should be taken as close as possible to the same time each day so as to be representative of a 24-hour period. Readings may also be taken at the beginning and end of the month for monthly average and total flows, or at the beginning and end of a sampling period of variable duration (i.e., operational hours only) for special loading studies.

There are two types of water meters which may be encountered in the field. One type uses a series of dials, while the other has a digital readout with the last digit(s) being determined by a single exterior dial. The number of fixed 0's at the end of the digital readout indicates the value of the exterior dial (1, 10,

100). Figure 1 illustrates these two types of meters. It is important to take accurate readings. When a value is about to change, it may appear to be the higher value, while it is actually near the top of the lower value. For example, on a digital readout, the "4" in "49" may appear to be a "5" because the "4" has almost rolled up and the "5" is almost in its new position. Therefore, meters should be read from right to left, or lowest to highest placement, so that the value of the lower digit may give an indication of the value of the higher digit. Another consideration when reading a multiple-dial meter is which dial is "first", or "lowest". This dial will be marked in some way, and will generally be moving at a faster rate than the others.

#### 11.4 GRAB SAMPLER

Grab samples may be collected manually or by using an ISCO sampler. The manual sampler is a pole with two clamps used to hold the collection bottle. Place the collection bottle in the clamps, and tighten until snug. Dip the bottle into the flow, facing upstream, and rinse (or seed) it several times with effluent. Redip the collection bottle and pour the effluent collected into your sample container. Seed the sample container with this effluent, and then continue to collect grabs until the sample bottle is full. When collecting grab samples from a holding tank (as opposed to a stream-flow), follow the same procedures as above, except that the collection bottle should be dipped into the flow upside down and then turned so as to collect effluent from below the surface. Depending upon the size and characteristics of the tank, it may be necessary to use grabs from several different locations and depths of the tank in order to obtain a representative sample.

NOTE: A GRAB SAMPLE MAY BE COLLECTED OVER A PERIOD OF TIME UP TO, BUT NOT EXCEEDING, 15 MINUTES.

When collecting a grab sample with the ISCO, connect properly cleaned tubing to the sampler, and then turn the ISCO to "Forward". Pump enough effluent through the ISCO to fill the sample bottle, rinse the bottle with this effluent, and then refill the sample container. Turn the ISCO to "Reverse" to purge the line when you are finished sampling.

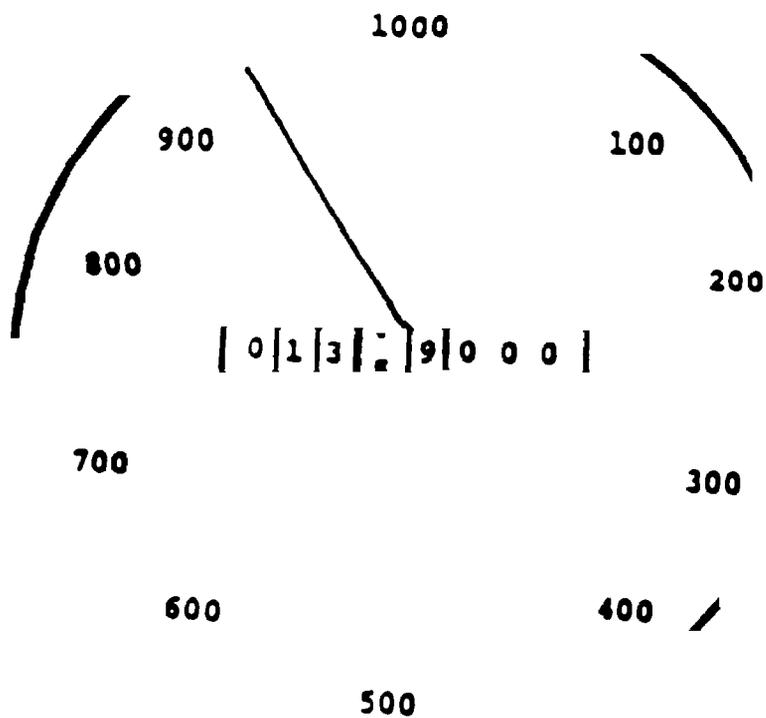
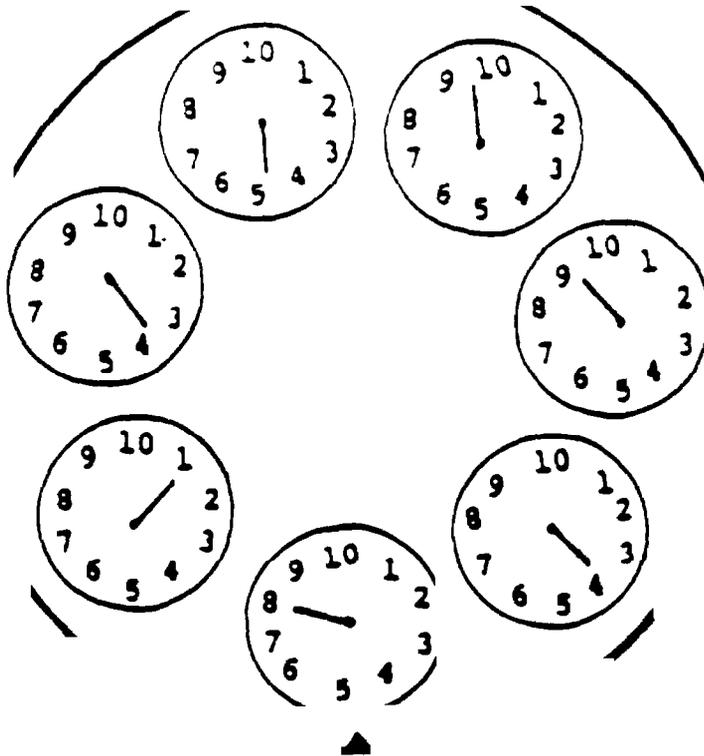


FIGURE 1. WATER METERS

## 11.5 ISCO SAMPLER

The ISCO is an automatic sampler composed of a base, which holds the sample collection container(s) and ice; the head, which is the control unit; and a cover to help keep the head dry. It operates by utilizing a peristaltic pump to draw effluent up through a length of tubing extending from the ISCO to the flow, and discharges this effluent into the collection container(s) in the base of the ISCO. The ISCO is designed to collect two types of samples: composite and sequential. Sequential samples can only be collected with ISCO Model 1680.

For composite samples, ISCO Model 1680 uses a discharge tube that extends to the middle of the head so as to discharge to a container with a center opening. For sequential sampling, a short discharge tube is used to feed the sample into the appropriate discrete container through a distribution funnel (see Sequential Sampling). Glass discharge tubes are used when collecting metal or organic samples. To set up an ISCO sampler:

- 1) Open the manhole, flume, cleanout cap, etc. to access the flow following the procedures outlined in Section 4 (Opening manholes/flumes).
- 2) Attach a battery to the ISCO.
- 3) Attach the site specific tubing.
- 4) If collecting metal or organic samples, run some DI water through the tubing and rinse the sample blank container; then collect a DI water blank.
- 5) Set up the actuator or flow meter, if necessary, but do not connect the cable to the ISCO.
- 6) Lower the tubing into the flow, setting it up with a weight or pole as necessary.
- 7) Set the ISCO controls for the tubing length and diameter (3/8 or 1/4-inch).
- 8) Set the ISCO controls for the desired volume to be collected at the appropriate head feet for the site.
- 9) For ISCO Model 1680, put the sampler on "Auto", set the time interval to next sample to "000", and collect a "measured grab". For Model 2710, press Manual Sample. This will allow you to see how much volume will be

collected with each grab. If the volume is higher or lower than you wish to collect, reset the volume and/or head feet and collect another measured grab until you collect the desired volume.

- 10) Collect another grab (approximately 400 ml) in the graduated cylinder to use for pH analysis.

#### 11.5.1 Composite Samples

For composite sampling, a long discharge tube should be used with a piece of polyethylene tubing attached to the end (Model 1680). The tail base, ISCO head and cover are used.

- 11) Place the composite jug (with lid on) in the base and pack with ice; remove the lid from the jug.
- 12) Put the ISCO head on the base, making sure the discharge tube feeds into the composite jug.
- 13) Set the toggle switch to disable (Model 1680 only).
- 14) Set the interval to next sample at "2" or "3", then set the time setting to the desired composite time interval (15, 20 or 30 minutes) or the number of signals if using a flow meter.
- 15) Lock the ISCO, and position it where it will remain for the composite duration (suspended in the manhole, lowered into the flume, or located away from traffic/ activity areas).

NOTE: BE SURE YOU OBSERVE THE FIRST SAMPLE BEING COLLECTED BEFORE LEAVING THE SITE.

#### 11.5.2 Sequential Samples

For sequential sampling, you must use ISCO Model 1680. A short discharge tube should be used with no tubing attached. The distribution funnel should be attached to the bottom of the head using the center bolt to hold it in place. The black serving plate (with 28 holes in it) should be placed over the bottles in the base.

- 11) Use the shorter "discrete" base that has 28 bottles which are held in place by a stainless steel metal circle.

- 12) Pack the center of the discrete base with ice; remove the lids from the bottles.
- 13) Set the toggle switch to normal or enable.
- 14) Hold the manual advance button on the ISCO until the word "Standby" appears in the small hole in the base of the ISCO head, then press manual advance again one time so that the number "1" appears in the hole.
- 15) Put the ISCO head on the base.
- 16) Set the switch for number of bottles per sample, or number of samples per bottle as appropriate for the sampling requirements.
- 17) Set the interval to next sample at "2" or "3", then change the time setting to the desired time interval or number of flow signals.
- 18) Lock the ISCO, and position it where it will remain for the sampling duration (suspended in the manhole, lowered into the flume, or located away from traffic/activity areas).

NOTE: BE SURE YOU OBSERVE THE FIRST SAMPLE BEING COLLECTED BEFORE LEAVING THE SITE.

## 12.0 INDUSTRIAL VAN MAINTENANCE AND SUPPLY

### 12.1 MAINTENANCE

The industrial van is serviced by Pinellas County Fleet Maintenance (PCFM) every six months or 5000 miles, whichever comes first. PCFM does a complete vehicle inspection, and changes fluids and filters, as necessary. The industrial van is also taken to PCFM for repair if the drivers' routine checks indicate any problems. The van is checked by the drivers for fluid level, tire pressure, light and horn function, and proper operation on a routine basis. The Vehicle Maintenance Schedule is included as Form 3. Per trip mileage is also recorded and turned in monthly.

### 12.2 SUPPLY

The industrial van is kept stocked with the supplies and equipment that are commonly used or that may be necessary in an emergency. These supplies include: first aid and safety equipment, at least one sampler, sampling accessories, miscellaneous tools, paperwork, DI water and disinfectants. The complete van inventory checklist is included as Table 3. Many of the items on the list are kept in the van at all times, others are depletable (i.e., gloves, DI water). The van is checked and restocked, as necessary, at the end of each sampling run and/or prior to going out in the field.

FORM 3

VEHICLE MAINTENANCE SCHEDULE

INSPECTION ITEM	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC
FLUIDS:												
OIL												
STEERING												
BATTERY												
TRANSMISSION												
RADIATOR												
WIPER												
LIGHTS:												
HEADLIGHTS												
TAIL LIGHTS												
BRAKE LIGHTS												
TURN SIGNALS												
HAZARD LIGHTS												
TIRES:												
AIR PRESSURE												
TREAD/APPEARANCE												
OPERATION:												
HORN												
BRAKES												
WIPERS												
TRANSMISSION												
OTHER:												

NOTE: Check off items inspected; use an asterisk(\*) to indicate items needing repair. Report any needed repairs immediately.

vehicle: 881221

TABLE 3

## INDUSTRIAL VAN INVENTORY CHECKLIST

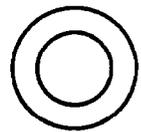
- \_\_\_ acid preservation kit
- \_\_\_ acid-washed tubing
- \_\_\_ actuator
- \_\_\_ alcohol
- \_\_\_ as-builts
- \_\_\_ battery booster cable
- \_\_\_ batteries for pH meter
- \_\_\_ bubbler tubing
- \_\_\_ buckets
- \_\_\_ calibration log books (flow meter, pH meter)
- \_\_\_ clamps and ISCO hardware
- \_\_\_ cubitainers
- \_\_\_ DI water for 5 gallon container
- \_\_\_ extension cords
- \_\_\_ flashlight
- \_\_\_ flow meter connector cables
- \_\_\_ flow meter manual
- \_\_\_ flow meter supplies (paper/pens)
- \_\_\_ flume depth measurer
- \_\_\_ foot pump
- \_\_\_ graduated cylinder
- \_\_\_ hand wash
- \_\_\_ incident documentation log
- \_\_\_ industrial sampling sheets
- \_\_\_ inflatable line blocker
- \_\_\_ ISCO SAMPLER (1)
- \_\_\_ ladder (20 ft. extension)
- \_\_\_ latex gloves
- \_\_\_ manhole hook
- \_\_\_ manhole spiders (2)
- \_\_\_ metal sampling bottles
- \_\_\_ mileage sheets
- \_\_\_ misc. bottles
- \_\_\_ misc. paperwork (accident forms, etc.)
- \_\_\_ paper towels
- \_\_\_ permanent markers and ball point pens
- \_\_\_ rvc pens
- \_\_\_ rope
- \_\_\_ strainers and weights
- \_\_\_ suspension cables
- \_\_\_ tape (electrical, filament, fluorescent, masking)
- \_\_\_ thermometers
- \_\_\_ tool box
- \_\_\_ volatile organic bottles
- \_\_\_ volt-ohm meter
- \_\_\_ work gloves

*Appendix X*

*Example Sample Tag and Chain-of-Custody Form for Use by POTWs*

EXAMPLE SAMPLE TAG

ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF ENFORCEMENT  
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER  
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER  
DENVER, COLORADO 80025



Project Code      Station No.      Mo./Day/Year      Time      Designate:      )  
Comp.      Grab

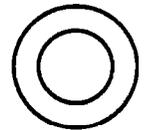
Station Location

Samplers: (Signature)

Remarks:  
Tag #      Lab Sample #

- Bacteriology
- Mutagenicity
- Pesticides
- Volatile Organics
- Priority Pollutants
- Organics GC/MS
- Oil and Grease
- Cyanide
- Metals
- Mercury
- Phenolics
- COD, TOC, Nutrients
- Soilds (TSS) (TDS) (SS)
- BOD      Anions

Analyses:



Preservative:  
Yes:  No:

**United States  
Environmental Protection  
Agency**

**Chain-of-Custody Form**

3. *Sample Number*

5. *Task Number*

4. *Inspection Number*

1. *Inspector Name and Address*

6. *Sample Description*

7. *Sample Date*   8. *Sample Time*   9. *Duplicate Requested?*  
( ) Yes   ( ) No

2. *Inspector Signature*

10. *Sample Location*

11. *Analysis/Testing Required*

*Laboratory*

*Date Received*

*Received By*

*Sent Via*

*Sample Condition*

*Condition of Seals*

*Units Received*

*Storage Location*

*Assigned By*

*Assigned To*

*Delivered By*

*Date Delivered*

*Number of Units Received*

*Units Analyzed*

*Date Seal Broken*

*Date Received*

*Resealed By*

*Storage Location*

*Date Results of Analysis*

*Issued to the POTW*

*Remarks:*

### ***Instructions for the Use of the Example Chain-of-Custody Form***

1. Enter the Inspector's Name and POTW's (or the POTW's office) address.
2. Sign the Chain-of-Custody Record Form.
- 3 & 4. Each inspection and sampling event should be identified with a unique number to track the information from the inspection.
5. Task numbers are used when the POTW uses contractors to perform inspections. Inspectors may disregard this space.
6. Describe the sample, including: size, container, contents (e.g., 8 oz. bottle of electroplating effluent).
- 7 & 8. List the date (7) and time (8) the sample was taken at the facility.
9. Indicate if duplicate (or splits) were performed at the time of sampling.
10. Enter the name and address of the facility and the sample location identification number.
11. List all required analytic tests (e.g., Cd, Cr, Ni, and Zn testing).

*Appendix XI*

*List of Regional Pretreatment Coordinators*

## ***Regional Pretreatment Coordinators***

### **REGION I**

(CT, ME, MA, NH, RI, & VT)

**Mark Spinale** (WCM-2103)

U.S. EPA - Region I

JFK Federal Building

Boston, MA 02203

Phone: (617) 565-3554

Fax: (617) 565-4940

### **REGION II**

(NJ, NY, Puerto Rico & VI)

**Virginia Wong** (2WM-WPC)

U.S. EPA - Region II

26 Federal Plaza, Room 825

New York, NY 10278

Phone: (212) 264-1262

Fax: (212) 264-9597

### **REGION III**

(DE, DC, MD, PA, VA, & WV)

**John Lovell** (3WM-52)

U.S. EPA - Region III

841 Chestnut Street

Philadelphia, PA 19107

Phone: (215) 597-6279

Fax: (215) 597-3359

### **REGION IV**

(AL, FL, GA, KY, MS, NC, SC, & TN)

**Al Herndon** (FPB-3)

U.S. EPA - Region IV

345 Courtland Street, N.E.

Atlanta, GA 30365

Phone: (404) 347-3973

Fax: (404) 347-1797

### **REGION V**

(IL, IN, MI, MN, OH, & WI)

**Matt Gluckman** (5 WQP-16J)

Implementation Coordinator

U.S. EPA - Region V

77 W. Jackson Blvd.

Chicago, IL 60604

Phone: (312) 886-6089

Fax: (312) 886-7804

**Mike Mikulka** (WCC-15J)

Enforcement Coordinator

U.S. EPA - Region V (WCC-15J)

77 W. Jackson Blvd.

Chicago, IL 60604

Phone: (312) 886-6760

Fax: (312) 886-7804

### **REGION VI**

(AR, LA, NM, OK, & TX)

**Lee Bohme** (6W-PM)

Implementation Coordinator

U.S. EPA - Region VI

1445 Ross Avenue, Suite 1200

Dallas, TX 75202-2733

Phone: (214) 655-7175

Fax: (214) 655-6490

**Bob Goodfellow** (6W-EO)

Enforcement Coordinator

U.S. EPA - Region VI

1445 Ross Avenue, Suite 1200

Dallas, TX 75202-2733

Phone: (214) 655-6444

Fax: (214) 655-6490

### **REGION VII**

(IA, KS, MO, & NE)

**Paul Marshall** (WACM)

U.S. EPA - Region VII

726 Minnesota Avenue

Kansas City, KS 66101

Phone: (913) 551-7419

Fax: (913) 551-7765

### **REGION VIII**

(CO, MT, ND, SD, UT & WY)

**Curt McCormick** (8WM-C)

Implementation Coordinator

U.S. EPA - Region VIII

999 18th Street, Suite 500

Denver, CO 80470

Phone: (303) 293-1592

Fax: (303) 293-1647

**Bruce Kent** (8WM-C)

Enforcement Coordinator

U.S. EPA - Region VIII

999 18th Street, Suite 500

Denver, CO 80470

Phone: (303) 293-1592

Fax: (303) 293-1647

### **REGION IX**

(AZ, CA, HI, & NV)

**Keith Silva** (W-5-2)

Implementation Coordinator

U.S. EPA - Region IX

75 Hawthorn Street

San Francisco, CA 94105-3901

Phone: (415) 744-1907

Fax: (415) 744-1235

**Bob Wills** (W-5)

Enforcement Coordinator

U.S. EPA - Region IX

75 Hawthorne Street

San Francisco, CA 94105-3901

Phone: (415) 744-1910

Fax: (415) 744-1235

### **REGION X**

(AK, ID, OR, & WA)

**Sharon Wilson** (WD-134)

U.S. EPA - Region X

1200 Sixth Avenue

Seattle, WA 98101

Phone: (206) 553-0325

Fax: (206) 553-0165

*Appendix XII*

*List of Available Pretreatment Guidance Documents*

This appendix provides a comprehensive list of documents available from the U.S. EPA. To obtain any of these documents, please contact either of the following references:

- U.S. EPA Water Resources Center  
(202) 260-7786  
Please reference the EPA Identification Number provided in the following table when ordering through the Water Resources Center.
- The National Technical Information Service  
U.S. Department of Commerce  
5285 Port Royal Rd.  
Springfield, VA 22161  
(800) 553-6847  
Please reference the NTIS number provided in the following table when ordering through NTIS.

Document Safes Desk:	(703) 487-4650
General Information:	(703) 487-4600
Customer Services:	(703) 487-4660
Document Identification:	(703) 487-4780

	<i>Title</i>	<i>Year</i>	<i>EPA Document #</i>	<i>NTIS #</i>
1	Guidance to Protect POTW Workers From Toxic and Reactive Gases and Vapors	1992	EPA 812/B-92/001	PB92- I 73-236
2	Model Pretreatment Ordinance	1992	EPA 833/B-92/003	PB93-122-414
3	National Pretreatment Program - Report to Congress (519 Report)	1991	EPA 505/2-91/001	PB91-228-726
4	Control of Slug Loadings to POTWs - Guidance Manual	1991	EPA 21W-4001	PB92- 11 1-897
5	Supplemental Manual on the Development and Implementation of Local Discharge Limitations Under the Pretreatment Program	1991	EPA 21W-4002	PB93-209-872
6	PRELIM Version 4.0 User's Guide	1991	EPA 21 W-4003	PB93-505-980GEI
7	Guidance Manual for POTWs to Calculate the Economic Benefit of Noncompliance	1990		PB91-148-445
8	Industrial User Permitting Guidance Manual	1989	EPA 833/B-89/001	PB92-123-OI7
9	Aluminum, Copper, and Nonferrous Metals Forming and Metal Powders Pretreatment Standards	1989	EPA 833/B-89/201	PB91-145-441
10	Guidance for Developing Control Authority Enforcement Response Plans	1989		PB90-185-083/AS
11	Guidance Manual for Preventing Interference at POTWs	1987	EPA 833/B-89/201	PB92-117-969/AS
12	Guidance Manual for Battery Manufacturing Pretreatment Standards	1987	EPA 833/B-87/200	PB92-117-951/AS
13	Guidance Manual on the Development and Implementation of Local Discharge Limits Under the Pretreatment Program	1987	EPA 833/B-87-202	PB92-129-188
14	Guidance Manual for the Identification of Hazardous Wastes Delivered to POTWs by Truck, Rail, or Dedicated Pipe	1987		PB92-149-251
15	Environmental Regulations and Technology - The National Pretreatment Program	1986	EPA 625/10-86/005	PB90-246-521
16	Guidance Manual for Leather Tanning and Finishing Pretreatment Standards	1986		PB92-232-024
17	Guidance Manual for the Use of Production-Based Standards and the Combined Wastestream Formula	1985	EPA 833/B-85/201	PB92-114-438
18	Guidance Manual for Iron and Steel Manufacturing Pretreatment Standards	1985		PB92-114-388
19	RCRA Information on Hazardous Wastes for Publicly Owned Treatment Works	1985	EPA 833/B-85/202	PB92-114-396/AS
20	Guidance Manual for Preparation and Review of Removal Credit Applications	1985	EPA 833/B-85/200	PB92-114-446
21	Guidance Manual for Implementing the Total Toxic Organics (TTO) Pretreatment Standards	1985	EPA 440/1-85/009g	PB93-167-005
22	Guidance Manual for Electroplating and Metal Finishing Pretreatment Standards	1984	EPA 440/1-84/091g	PB87-192-597
23	Guidance Manual for Pulp, Paper, and Paperboard and Builder's Paper and Board Mills Pretreatment Standards	1984		PB92-231-638
24	Procedures Manual for Reviewing a POTW Pretreatment Program Submission	1983	EPA 833/B-83/200	PB93-209-880

Appendix XIII

40 CFR Part 136 - Tables IA, IB, IC, ID, IE and II

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, and IE. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, and IE. The references and the sources from which they are available are given in paragraph (b) of this section. These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the FEDERAL REGISTER. The discharge parameter values for which reports are required must be determined

by one of the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, ID, and IE, or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and §§ 136.4 and 136.5 of this part 136. Under certain circumstances (§ 136.3 (b) or (c) or 40 CFR 401.13) other test procedures may be used that may be more advantageous when such other test procedures have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing the Director of the State in which such discharge will occur does not object to the use of such alternate test procedure.

TABLE IA. - LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and methods	Reference (Method No. or Page)		ASTM	USGS <sup>a</sup>	Other
	EPA <sup>b</sup> #	Std. methods 17th Ed.			
11. Bromide, mg/L: Titrimetric	320.1		D1346-82(C) (1988)	I-1125-85	p. 844.10
12. Cadmium—Total <sup>a</sup> , mg/L, digestion <sup>a</sup> followed by: AA direct aspiration <sup>20</sup>	213.1	2111 B or C	D3657-80 (A or B)	I-3135-85 or I-3138-85	974.27 p.37
AA furnace	213.2	3113 B		I-1472-85	
ICP <sup>20</sup>	200.7	2120 B			
DCP <sup>20</sup>			D4180-88		Note 34.
Voltammetry <sup>11</sup> , or Colorimetric (Dithizone)	3657-90C	3600-Cd D			
13. Calcium—Total <sup>a</sup> , mg/L, Digestion <sup>a</sup> followed by: AA direct aspiration	215.1	3111 B	D611-88(B)	I-3152-85	
ICP	200.7 <sup>a</sup>	3120 B			
DCP, or Titrimetric (EDTA)	215.2	3600-Ca D 8210 B	D611-88(A)		Note 34.
14. Calcitropic biochemical oxygen demand (CBOD <sub>5</sub> ), mg/L D solved Oxygen Depletion with nitrification inhibitor					
15. Chemical oxygen demand (COD), mg/L, Titrimetric, or % dichromate, manual or automated	410.1 410.2 or 410.3 410.4	8220 B	D1252-88	I-3560 or I-3562-85 I-3561-85	973.46 <sup>a</sup> p. 17.9 Notes 13 or 14.
16. Chloride, mg/L: Titrimetric (silver nitrate) or (Mercuric nitrate), or Colorimetric, manual or Automated (Ferricyanide)	325.3 325.1 or 325.2	4600-Cl B 4600-Cl C 4600-Cl C	D612-88(B) D612-88(C) D612-88(C)	I-1183-85 I-1184-85 I-1187-85 I-2187-85	973.51.9
17. Chlorine—Total residual, mg/L, Titrimetric: Ar/Cl or A <sub>2</sub> direct Starch end point direct	330.1 330.3	4600-Cl D 4600-Cl B	D1253-79(A) D1253-79(B) (1985) Part 18.3.		
Back titration either end point <sup>16</sup> , or DPD-FAS % dichromate / L DPD or Electrode	330.2 330.4 330.5	4600-Cl C 4600-Cl F 4600-Cl G			Note 18.
18. Chromium VI dissolved, mg/L, 0.45 micron filtration followed by: AA direct aspiration Colorimetric (DiAs <sub>2</sub> / L) solids	218.4	2111 A		I-1232-85 I-1230-85	307B.17
19. Chromium—Total <sup>a</sup> , mg/L, digestion <sup>a</sup> followed by: AA direct aspiration <sup>20</sup> AA furnace AA furnace ICP <sup>20</sup> DCP <sup>20</sup> , or Voltammetry <sup>11</sup> , or	218.1 218.3 218.2 200.7	2111 B 3111 C 3112 B 3120 B	D1687-88(D)	I-3236-85	974.27
Colorimetric (DiAs <sub>2</sub> / L) solids		3600-Cr D	D1687-84(A)		
20. Cobalt—Total <sup>a</sup> , mg/L, Digestion <sup>a</sup> followed by: AA direct aspiration AA furnace ICP, or DCP	218.1 or C 218.2 200.7 <sup>a</sup>	3111 B (A or B) 3113 B 3120 B	D3656-80	I-3238-85	p. 37.9
21. Color platinum cobalt units or dominant wavelength, hue, lum- inance purity Colorimetric (ADM), or (Platinum cobalt), or % dichromate / L	1101 1102 1102	2120 E 2120 B 2120 C		I-1380-85	Note 18.
22. Copper—Total <sup>a</sup> , mg/L, digestion <sup>a</sup> followed by: AA direct aspiration <sup>20</sup>	PO.1	3111 B or C	D1688-80 (A or B)	I-3270-85 or I-3271-85	974.27 p. 37.
AA furnace	220.2	3113 B			
ICP <sup>20</sup>	200.7	3120 B			
DCP <sup>20</sup> , or Colorimetric (Neocuproine), or Automated <sup>20</sup>		3600-Cu D or E	D4180-88 1688-84 (88)(A)		Note 34. O 18.
23. Cyanide—Total, mg/L: Manual distillation with MgCl <sub>2</sub> followed by Titrimetric, or % dichromate / L, manual or Automated <sup>20</sup>	335.2 335.3	4600-CN-C 4600-CN-D 4600-CN-E	D8036-88(A) D8036-88(A)	I-3300-85	p. 22.9
24. Cyanide amenable to chlorination, mg/L: Manual distillation with MgCl <sub>2</sub> followed by titrimetric or % dichromate / L	335.1	4600-CN-G	D8036-88(B)		
25. Fluoride—Total, mg/L: Manual distillation <sup>a</sup> followed by Electrode, manual or Automated Colorimetric (SPADNS) or Automated complexone	340.2 340.1 340.3	4600-F-B 4600-F-C 4600-F-D 4600-F-E	D1179-88(B) (1988)	I-4327-85	
26. Gold—Total <sup>a</sup> , mg/L, Digestion <sup>a</sup> followed by: AA direct aspiration AA furnace, or DCP	231.1 231.2	3111 B			Note 34
27. Hardness—Total, as CaCO <sub>3</sub> , mg/L: Automated colorimetric Titrimetric (EDTA), or Ca plus Mg as their carbonates, by induc- tively coupled plasma or AA direct aspiration. (See Parameters 13 and 33)	130.1 1302	2340 C	D1126-88 (1988)	I-1338-85	973.52B.9
28. Hydrogen ion (pH), pH units Electrometric, measurement, or Automated electrode	150.1	4600-H. B	D1283-84 (A or B) (1980)	I-1588-85	973.41.9 Note 21
29. Iodine—Total <sup>a</sup> , mg/L, Digestion <sup>a</sup> followed by: AA direct aspiration or AA furnace	235.1 235.2	3111 B			
30. Iron—Total <sup>a</sup> , mg/L, digestion <sup>a</sup> followed by: AA direct aspiration <sup>20</sup>	236.1	3111 B or C	D1088-80 (A or B)	I-3381-85	974.27

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TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and methods	Reference (Method No. or Page)		ASTM	UBS <sup>2</sup>	Other
	EPA 1.1 <sup>3</sup>	Std. methods 17th Ed.			
AA furnace	236.2	3113 B			
ICP <sup>26</sup>	*200.7	3120 B			
DCP <sup>26</sup> , a					Note 34.
Colorimetric (Phenanthroline)		3600—Fe D	D1068—90(D)		Note 22.
31. Kjeldahl nitrogen—Total, (as N), mg/L; digestion and distillation followed by:		4600—N org B a c	3690—84(A)		
Titration	361.3	4600—NH, E	D3690—89(A)		973.48. <sup>9</sup>
1% solution	361.3	4600—NH, c	D3690—89(A)		
Electrode	361.3	4600—NH, F a G			
Automated phenate	361.1	4600—NH, H	I-4661-78 <sup>9</sup>		
Semi-automated block digester, or	361.2		D3690—89(B)		
Polysulfate	361.4		D3690—89(A)		
32. Lead—Total <sup>4</sup> , mg/L; digestion <sup>4</sup> followed by:					
AA direct aspiration <sup>26</sup>	239.1	3111B or c	D3659—80 (A or B)	I-3399—86	*974.27.
AA furnace	239.2	3113 B			
ICP <sup>26</sup>	*200.7	3120 B			
DCP <sup>26</sup>					Note 34.
Voltammetry <sup>11</sup> , a			D4190—88		
Colorimetric (Dithizone)		3600—Pb D	D3659—90(C)		
33. Magnesium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:					
AA direct aspiration	242.1	3111 B	D611—88(B)	I-3447—86	974.27. <sup>9</sup>
ICP	200.7 <sup>9</sup>	3120 B			
DCP, or					Note 34
Gravimetric		3600—mg D	D611—77(A)		
34. Cadmium—Total <sup>4</sup> , mg/L; digestion <sup>4</sup> followed by:					
AA direct aspiration <sup>26</sup>	243.1	3111 B or c	D668—80 (A or B)	I-3464—86	*974.27.
AA furnace	243.2	3113 B			
ICP <sup>26</sup>	200.7	3120 B			
DCP <sup>26</sup> , a	wo0.7				Note 34.
Colorimetric (Parasulfate), a		3600—Mn D	D4190—88		Note 34.
(Periodate)			D668—84(A) (1988)		*920.203 Note 23.
35. Mercury—Total <sup>4</sup> , mg/L:					
Cold vapor, manual or	246.1	3112 B	D3223—86	I-3482—86	977.22. <sup>9</sup>
Automated	246.2				
36. Molybdenum—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:					
AA direct aspiration	246.1	3111 D		I-3490—86	
AA furnace	246.2	3113 B			
ICP, a	200.7 <sup>9</sup>	3120 B			
DCP					Note 34.
37. Nickel—Total <sup>4</sup> , mg/L; digestion <sup>4</sup> followed by:					
AA direct aspiration <sup>26</sup>	249.1	3111B or c	D1898—80 (A or B)	I-3498—86	
AA furnace	249.2	3111 B			
ICP <sup>26</sup>	*200.7	3120 B			Note 34.
DCP <sup>26</sup> , a			D4190—88		
Colorimetric (Hexamine)		3600—Ni D			
38. Nitrate (as N), mg/L:					
Colorimetric (Brucine sulfate) or Nitrate-nitrite N minus Nitrite N	362.1		D992—71		973.50 <sup>9</sup> , 4190 <sup>17</sup> , p. 28. <sup>9</sup>
(See parameters 39 and 40)					
39. Nitrite-nitrite (as N), mg/L:					
Cadmium reduction, Manual a	363.3	4600—NO <sub>2</sub> E	D3967—80(B)		
Automated, or	363.2	4600—NO <sub>2</sub> F	D3967—80(A)	I-4646—86	
Automated hydrazine	363.1	4600—NO <sub>2</sub> H			
40. Nitrite (as N), mg/L; % of sulfanilic acid:					
Manual or	364.1	4600—NO <sub>2</sub> B	D1254—87		Note 26.
Automated (Diazotization)				I-4640—86	
41. Oil and grease—Total recoverable, mg/L:					
Gravimetric (extraction)	413.1	5620 B			
42. Organic carbon—Total (TOC), mg/L:					
Combustion or oxidation	416.1	6310—B	D2579—86 (A or B)		973.47 <sup>9</sup> , p. 14. <sup>24</sup>
43. Organic nitrogen (as N), mg/L:					
Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4)					
44. Orthophosphate (as P), mg/L; Ascorbic acid method:					
Automated, a	3n.1	4600—P F		I-4801—86	973.56. <sup>9</sup>
Manual single reagent, or	365.2	4600—P E	D615—89(A)		973.55. <sup>9</sup>
Manual two reagent	365.3				
45. Cesium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:					
AA direct aspiration, or	262.1	3111 D			
AA furnace	262.2				
46. Oxygen dissolved, mg/L:					
Winkler (Azide modification), a	360.2	4600—O c	D699—81(C) (1988)	I-1576—78 <sup>9</sup>	973.468. <sup>9</sup>
Electrode	50.1	4600—O G		I-1576—78 <sup>9</sup>	
47. Palladium—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:					
AA direct aspiration, or	263.1	3111 B			p. 827. <sup>10</sup>
AA furnace	263.2				p. 828. <sup>10</sup>
DCP					Note 34.
48. Phenols, mg/L; Manual distillation <sup>26</sup>	420.1		D1783—80 (A or B)		Note 27.
Followed by:					
Colorimetric (4AAP) manual, or	420.1				Note 27.
Automated <sup>19</sup>	420.2				
49. Phosphorus (elemental), mg/L; Gas-liquid chromatography					Note 28.
50. Phosphorus—Total, mg/L:					
Parasulfate digestion followed by:	366.2	4600—P—B.5			973.56. <sup>9</sup>
Manual, a	366.2 or 366.3	4600—P—E	D615—89(A)		
Automated ascorbic acid reduction, or	366.1	4600—P—F		I-4800—86	973.56. <sup>9</sup>
Semi-automated block digester	am.4				
51. Platinum—Total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed by:					
AA direct aspiration	266.1	3111 B			
AA furnace	266.2				Note 34.
DCP					
52. Potassium—total <sup>4</sup> , mg/L; Digestion <sup>4</sup> followed					
AA direct aspiration	268.1	3111 B		I-3630—86	973.53. <sup>9</sup>
ICP	200.7 <sup>9</sup>				
Flame photometric, or		3600—K D	D1429—82(A)		
Colorimetric (Cobaltinitrate)					3178. <sup>17</sup>

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TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and methods	Reference (Method No. or Page)		ASTM	USECS <sup>2</sup>	Other
	EPA <sup>1</sup>	Std. methods 17th Ed.			
53 Residue—Total, mg/L, Gravimetric, 103-105*	180.3	2540 B		I-3750-85	
54 Residue—Total, mg/L, Gravimetric, 180*	180	2540-C		I-1750-85	
55 Residue—Total, mg/L, Gravimetric, 103-105* post washing of residue.	180.2	2540-D		I-3785-85	
56 Residue—Total, mg/L, Volumetric, (Imhoff cone), or gravimetric.	180.5	2540 F			
57 Residue—Volatile, mg/L, Gravimetric, 550*	180.4	2540 E		I-3753-85	
58 Rhodium—Total*, mg/L, Digestion* followed by: AA direct aspiration, or AA furnace	285.1 285.2	3111 B			
59 Ruthenium—Total*, mg/L, Digestion* followed by: AA direct aspiration, or AA furnace	287.1 287.2	3111 B			
60 Selenium—Total*, mg/L, digestion* followed by: AA furnace ICP <sup>3</sup> , or AA gaseous hydride	2702 *200.7 270.3	3113 B 3120 B 3114 B	D3858-88(A)	I-3887-85	
61 Silica—Dissolved, mg/L, 0.45 micron filtration followed by: Colorimetric Manual or Automated (Autoanalyzer), or ICP	370.1 200.7*	4600-Si D	D859-88(B)	I-1700-85 I-2700-85	
62 Silver—Total*, mg/L, Digestion* AA direct aspiration AA furnace Colorimetric (Mum) ICP, or DCP	272.1 72.2  20D.7	3111 B or C 3113 B		I-3720-85	973.27*, p 37*
63 Sodium—Total*, mg/L, Digestion* followed by: AA direct aspiration ICP DCP, or Fluorometric	273.1 200.7*	3111 B 3120 B	3600-Na D	I-3736-85	973.54*
64 Specific conductance, micromhos/cm at 25°C Wheatstone bridge	120.1	110 B	D1125-82(A)	I-1780-85	973.40*
65 Sulfate (as SO <sub>4</sub> ), mg/L, Automated colorimetric (barium chloride) Gravimetric, or Turbidimetric	375.1 375.3 375.4	4600-SO <sub>4</sub> C or D	D516-82(A) (1988) D516-88	925.54*	4aC, Q
66 Sulfide (as S), mg/L, Titrimetric (iodine), or Colorimetric (methylene blue)	376.1 376.2	4600-S E 4600-S D		I-3840-85	228A, 21
67 Sulfite (as SO <sub>3</sub> ), mg/L, Titrimetric (iodine-iodate)	377.1	4600-SO <sub>3</sub> B	D1338-84(C)		
68 Surfactants, mg/L, Colorimetric (methylene blue)	425.1	5540 C	D2330-88		
69 Temperature, C Thermometric	170.1	2550 B			Note 32.
70 Thallium—Total*, mg/L, Digestion* followed by: AA direct aspiration AA furnace, or ICP	278.1 278.2 200.7*	3111 B			
71 Tin—Total*, mg/L, Digestion* followed by: AA direct aspiration, or AA furnace	282.1 282.2	3111 B 3113 B		I-3850-78*	
72 Titanium—Total*, mg/L, Digestion* followed by: AA direct aspiration AA furnace DCP	283.1 283.2	3111 D			Note 34.
73 Turbidity, NTU Nephelometric	180.1	2130 B	D1889-88a	I-3880-85	
74 Vanadium—Total*, mg/L, Digestion* followed by: AA direct aspiration AA furnace ICP DCP, or Colorimetric (Gallie acid)	286.1 286.2 200.7*	3111 D 3120 B	D4190-88 D3373-84(A) (1988)		Note 34.
75 Zinc—Total*, mg/L, digestion* followed by: AA direct aspiration <sup>4</sup> AA furnace ICP <sup>3</sup> DCP <sup>3</sup> , or Colorimetric (Dimhizone) or (Zincron)	39.1 289.2 *200.7	3111 (B or C) 3120 b	D1891-80 (A or B)	I-3900-85	*974.27, *p. 37

Table 1B notes

<sup>1</sup> "Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI), EPA-800/4-78-020. Revised March 1983 and 1979 where applicable.

<sup>2</sup> Fehman, M. J., et al, "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, Techniques of Water-Resources Investigations, U.S. Geological Survey, Denver, CO, Revised 1988, unless otherwise stated.

<sup>3</sup> "Official Methods of Analysis of the Association of Official Analytical Chemists," methods manual, 16th ed. (1980).

<sup>4</sup> For the purpose of this test, the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979 and 1983." One (section 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (section 4.1.4) is preferred; however, the analyst should be cautioned that the mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all metal-organic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by the vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as ICP for certain elements such as arsenic, the noble metals, mercury, selenium, and titanium require a modified digestion and in all cases the method write-up should be consulted for specific instruction and/or cautions. Note: if the digestion included in one of the other approved references is different than the above, the EPA procedure must be used. NOTE: if the digestion included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals, (or digestion of the original sample solution for total metals) may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample solution to be analyzed meets the following criteria:

- a. has a low COD (<20)
- b. is visibly transparent with a turbidity measurement of 1 NTU or less
- c. is colorless with no perceptible odor, and
- d. is of one liquid phase and free of particulate or suspended matter following acidification.

<sup>5</sup> The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometry Method for Trace Element Analysis of Water and Wastes," is given at appendix C of this part 136

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- Manual distillation is not required if comparability data on effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.
- Ammons, Automated Electrode Method, Industrial Method Number 378-78 WE, dated February 19, 1978, (Bran & Luebbe (Technicon) AutoAnalyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmford, N.Y. 10523.
- The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979).
- American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.
- "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981)
- The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.
- C<sub>50</sub> is biochemical oxygen demand (CBOD<sub>5</sub>) must not be confused with the traditional BOD<sub>5</sub> test which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD<sub>5</sub> parameter. A discharger whose permit requires reporting the traditional BOD<sub>5</sub> may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD<sub>5</sub> is required, can the permittee report data using the nitrification inhibitor.
- OC Chemical Oxygen Demand Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, TX 77840
- Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537
- The back titration method will be used to resolve controversy.
- Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138. The calibration graph for the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 ml 0.00281 N potassium iodate/100 ml solution, respectively.
- The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976
- National Council of the Paper Industry for Air and Stream Improvement, (Inc.) Technical Bulletin 253, December 1971
- Cooper, Bismutholite Method, Method 8508, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2
- Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1978, Bran & Luebbe (Technicon) AutoAnalyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmford, N.Y. 10523.
- Iron, 1,10 Phosphoantimonate Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537
- Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, CO 80537.
- Wershaw, R.L., et al., "Methods for Analysis of Organic Substances in Water," Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14
- Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537
- Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH
- The approved method is cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0±0.2. The approved methods are given on pp 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual flow injection procedure.
- R.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, vol. 47, No. 3, pp. 421-426, 1970.
- Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate when silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L, the approved method is satisfactory.
- The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition.
- The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 13th Edition.
- Stevens, H.H., Ficke, J.F., and Smoot, G.F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation", Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 1, Chapter D1, 1975.
- Zinc, Zincin Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, CO 80537.
- "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1935 Rev. 1/1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923.
- Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the electrothermal atomic absorption method for arsenic are provided in appendix D of this part used. "Precision and Recovery Statements for Methods for Measuring Metals"
- "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, P.O. Box 200, Matthews, North Carolina 28106-0200, April 16, 1982. Available from the CEM Corporation

TABLE 1C.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter <sup>1</sup>	EPA Method Number <sup>2</sup>					ASTM	Other
	GC	GC/MS	HPLC	Standard methods 17th Ed			
1. Acetophenone	610	625, 1625	610	6410 B, 6440 B		D4857-87	
2. Acenaphthylene	610	625, 1625	610	6410 B, 6440 B		D4857-87	
3. Acroline	603	604, 1624					
4. Acrylonitrile	610	625, 1625	610	6410 B, 6440 B		D4857-87	
5. Anthracene	602	624, 1624		6210 B, 6220 B			
6. Benzene		625, 1625	605				Note 3, p. 1
7. Benzidine	610	625, 1625	610	6410 B, 6440 B		D4857-87	
8. Benzo(a)anthracene	610	625, 1625	610	6410 B, 6440 B		D4857-87	
9. Benzo(a)pyrene	610	625, 1625	610	6410 B, 6440 B		D4857-87	
10. Benzo(b)fluoranthene	610	625, 1625	610	6410 B, 6440 B		D4857-87	
11. Benzo(g,h,i)perylene	610	625, 1625	610	6410 B, 6440 B		D4857-87	
12. Benzo(k)fluoranthene	610	625, 1625	610	6410 B, 6440 B		D4857-87	
13. Benzyl chloride							Note 3, p. 130 Note 6, p. S102
14. Benzyl butyl phthalate	606	625, 1625		6410 B			
15. Bis(2-chloroethyl) methane	611	625, 1625		6410 B			
16. Bis(2-chloroethyl) ether	611	625, 1625		6410 B			
17. Bis(2-ethylhexyl) phthalate	606	625, 1625		6410 B, 6230 B			
18. Bromodichloromethane	601	624, 1624		6210 B, 6230 B			
19. Bromoform	601	624, 1624		6210 B, 6230 B			
1. 1,1,1-Trichloroethane	601	624, 1624		6410 B			
21. 4-Bromobiphenyl ether	611	625, 1625		6410 B			
1 Carbon tetrachloride	601	624, 1624		6230 B, 6410 B		Note 3, p. 130	
23. 4-Chloro-3-methylphenol	604	625, 1625		6410 B, 6420 B			
24. Chlorobenzene	601, 602	624, 1624		6210 B, 6220 B, 6230 B		Note 3, p. 130	
25. Chloroethene	601	624, 1624		6210 B, 6230 B			
26. 2-Chloroethyl ether	601	624, 1624		6210 B, 6230 B			
27. Chloroform	601	624, 1624		6210 B, 6230 B		Note, p. 130	
28. Chlorobenzene	601	624, 1624		6210 B, 6230 B			
29. 2-Chlorophenol	612	625, 1625		6410 B			
1. 2-Chlorophenol	604	625, 1625		6410 B, 6420 B			
31. 4-Chlorobiphenyl ether	611	625, 1625		6410 B			
32. Chrysene	610	625, 1625	610	6410 B, 6440 B		D4857-87	
33. Dibenz(a,h)anthracene	610	625, 1625	610	6410 B, 6440 B		D4857-87	
34. Dibenz(a,h)anthracene	601	624, 1624		6210 B, 6230 B			
35. 1, 2-Dichlorobenzene	601, 602, 612	624, 625, 1625		6410 B, 6230 B, 6220 B			
36. 1, 3-Dichlorobenzene	601, 602, 612	624, 625, 1625		6410 B, 6230 B, 6220 B			
8. 1, 4-Dichlorobenzene	601, 602, 612	625, 1624, 1625		6410 B, 6230 B, 6220 B			
38. 3-Dichlorobenzene		625, 1625	905	6410 B			
39. Dichlorodifluoromethane	601			6230 B			
40. 1, 1-Dichloroethene	601	624, 1624		6230 B, 6210 B			
41. 1, 2-Dichloroethene	601	624, 1624		6230 B, 6210 B			

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TABLE 1C.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter <sup>1</sup>	GC	EPA Method Number <sup>2,3</sup>			ASTM	Other
		GCMS	HPLC	Standard methods 17th Ed.		
42. 1,1-Dichloroethene	801	824, 1824		8230 a, 8210 B		
43. trans-1,2-Dichloroethene	801	824, 1824		8230 B, 8410 B		
44. 2,4-Dichlorophenol	804	825, 1825		8420 B, 8410 B		
45. 1,2-Dichlorobenzene	801	824, 1824		8230 B, 8210 B		
46. cis-1,2-Dichloroethene	801	824, 1824		8230 B, 8210 B		
47. trans-1,2-Dichloroethene	801	824, 1824		8230 B, 910 B		
48. Diethyl phthalate	806	825, 1825		8410 B		
49. 2,4-Dichlorophenol	804	825, 1825		8420 B, 8410 B		
50. Dimethyl phthalate	806	825, 1825		8410 B		
51. Di-n-butyl phthalate	806	825, 1825		8410 B		
52. Di-n-octyl phthalate	806	825, 1825		8410 B		
53. 2,3-Dinitrophenol	804	825, 1825		8420 B, 8410 B		
54. 2,4-Dinitrotoluene	809	825, 1825		8410 B		
55. 2,6-Dinitrotoluene	809	825, 1825		8410 B		
56. Ethylbenzene	802	824, 1824		8220 A, 8210 B		Note 3, p.130 Note 6, p.8102.
57. Ethylbenzene	802	824, 1824		8220 A, 8210 B		
58. Fluoranthene	810	825, 1825	810	8410 B, 8440 B	D4857-87	
59. Fluorene	810	825, 1825	810	8410 B, 8440 B	D4857-87	
60. Fluorene	812	825, 1825		8410 B		
61. Hexachlorobutadiene	812	825, 1825		8410 B		
62. Hexachlorobutadiene	812	825, 1825		8410 B		
63. Hexachlorobutadiene	816	825, 1825		8410 B		
64. Indeno (1,2,3-cd)pyrene	810	825, 1825	010	8410 B, 8440 B	D4857-87	
a. Isophorone	809	825, 1825		8410 B		
66. Methylene chloride	801	824, 1824		8230 B		Mm 3, p.130.
67. 2-Methyl-4,6-dimethylmu	804	825, 1825		8420 B, 8410 B		
68. Naphthalene	810	825, 1825	810	8410 B, 8440 B		
69. 1-Naphthol	809	825, 1825		8410 B	D4857-87	
70. 2-Nitrophenol	804	825, 1825		8410 B, 8420 B		
71. 4-Nitrophenol	804	825, 1825		8410 B, 8420 B		
72. N-Nitrosodimethylamine	807	825, 1825		8410 B		
73. N-Nitrosodimethylamine	807	825, 1825		8410 B		
74. N-Nitrosodimethylamine	807	825, 1825		8410 B		
75. 2,2-Dimethyl-1,3-dioxane	811	825, 1825		8410 B		
76. PCB-1016	808	825		8410 B		Note 3, p.43
77. PCB-1221	808	825		8410 B		Note 3, p.43
78. PCB-1232	808	825		8410 B		Note 3, p.43
79. PCB-1242	808	825		8410 B		Note 2, p.43
80. PCB-1248	808	825		8410 B		
81. PCB-1254	808	825		8410 B		Mm 3, p.43
82. PCB-1260	808	825		8410 B, 8830 B		Note a p.43
83. Polychlorinated biphenyls	804	825, 1825		8410 B, 8830 B		Note 3, p.140
84. Polychlorinated biphenyls	810	825, 1825		8410 B, 8440 B	D4857-87	
85. Phenol	804	825, 1825		8420 B, 8410 B		
86. Pyrene	810	825, 1825	010	8410 B, 8440 B	D4857-87	
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	801	824, 1824		8230 B, 8210 B		Note 3, p.130.
88. 1,1,2,2-Tetrachloroethane	801	824, 1824		8230 B, 8210 B		Note 3, p.130.
89. Toluene	802	824, 1824		8210 B, 8220 B		
90. Toluene	812	825, 1825		8410 B		Note 3, p.130.
91. 1,2,4-Trichlorobenzene	801	824, 1824		8210 B, 8230 B		
92. 1,1,1-Trichloroethane	801	824, 1824		8210 B, 8230 B		Note 3, p.130.
93. 1,1,2-Trichloroethane	801	824, 1824		8210 B, 8230 B		
94. Trichloroethene	801	824		8210 B, 8230 B		
95. Trichlorofluoromethane	801	824		8210 B, 8230 B		
96. 2,4,6-Trichlorophenol	804	825, 1825		8410 B, 8240 B		
97. Vinyl chloride	801	824, 1824		8210 B, 8230 B		

Table 1C notes:

- <sup>1</sup>All parameters are expressed in micrograms per liter (µg/L).
- <sup>2</sup>The full text of Methods 801-813, 824, 825, 1824, and 1825, are given at appendix A, "Test Procedures for Analysis of Organic Pollutants," of this part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B, "Definition and Procedure for the Derivation of the Method Detection Limit" of this part 136.
- <sup>3</sup>Methods for Benzidine, Chlorinated Organic Compounds, Polychlorinated Biphenyls and Pesticides in Water and Wastewater, U.S. Environmental Protection Agency, September, 1978.
- <sup>4</sup>Method 824 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 803 or Method 1824.
- <sup>5</sup>Method 825 may be extended to include benzidine, 1,2-dichloroethane, N-nitrosodimethylamine, and N-nitrosodimethylamine. However, when they are known to be present, Methods 806, 807, and 812, or Method 1825, are preferred methods for these compounds.
- <sup>6</sup>825, Screening only.
- <sup>7</sup>Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency, Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).
- <sup>8</sup>Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 801-803, 824, 825, 1824, and 1825 (See appendix A of this part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must split and analyze 10% (5% for Methods 824 and 825 and 100% for Methods 1824 and 1825) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unsplit sample are suspect and cannot be reported to demonstrate regulatory compliance.

NOTE: These warning limits are promulgated as an "interim final action with a request for comments."

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TABLE 1 D.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES<sup>1</sup>

Parameter µg/L	Method	EPA method number			Other
		EPA <sup>2,3</sup>	Standard methods 17th Ed.	ASTM	
1 Aldrin	GC GCMS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
2. Atracyln	GC				Note 3, p. 83; Note 6, p. S68.
3. Aminocarb	GC				Note 3, p. 94; Note a, p. S16.
4. Atraton	GC				Note 3, p. 83; Note 6, p. S68.
5. Atrazine	GC				Note 3, p. m; Note 6, p. S68.
a Azinphos methyl	GC				Note 3, p. 25; Note 6, p. S51.
7. Barban	GC				Note 3, p. 104; Note 6, p. S64.
8. α-BHC	GC GCMS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7.
9. β-BHC	GC GCMS	608 625	6630 C 6410 B	D3086-90	
10. δ-BHC	GC GCMS	608 625	6630 C 6410 B	D3086-90	
11. γ-BHC (Lindane)	GC GCMS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
12. Captaf	GC				Note 3, p. 7.
13. Carbaryl	TLC	D6630 B	D3086-90		Note 3, p. 94; Note 6, p. S60.
14. Carbaryl II	GC				Note 4, p. 30; Note 6, p. S73.
15. Chlordane	GC GC-MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7.
16. Chlorpyrifos	GC				Note 3, p. 104; Note 6, p. S64.
17. 2,4-D	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
18. 4,4'-D-DDD	GC GC-MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
19. 4,4'-DDE	GC GC-MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
20. 4,4'-DDT	GC GC-MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
21. Demeton-O	GC				Note 3, p. 25; Note 6, p. S51.
22. Deltamethrin	GC				Note 3, p. 25; Note 6, p. S51.
23. Diazinon	GC				Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
24. Dicamba	GC				Note 3, p. 115.
25. Dieldrin	GC				Note 4, p. 30; Note 6, p. S73.
26. Dieldrin	GC		6630 B & C		Note 3, p. 7.
27. Dieldrin	GC			D3086-90	
28. Dieldrin	GC GC-MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
29. Dioxathion	GC				Note 4, p. 30; Note 6, p. S73.
30. Disulfoton	GC				Note 3, p. 25; Note 6, p. S73.
31. Dursin	TLC				Note 3, p. 104; Note 6, p. S73.
32. Endosulfan I	GC GC-MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7.
33. Endosulfan II	GC GC-MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7.
34. Endosulfan Sulfate	GC GC-MS	608 625	6630 C 6410 B		
35. Endrin	GC GC-MS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
36. Endrin aldehyde	GC	608			
37. Ethion	GC				Note 4, p. 30; Note 6, p. S73.

TABLE 1 D.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES<sup>1</sup>—Continued

Parameter µg/L	Method	EPA method number			Other
		EPA <sup>2,3</sup>	Standard methods 17th Ed.	ASTM	
a Fenuron	TLC				Note a, p. 104; Note 6, p. S64.
38. Fenuron-TCA	GC				Note 3, p. 104; Note 6, p. S64.
40. Heptachlor	GC GCMS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
41. Heptachlor epoxide	GC	608	6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30; Note 6, p. S73.
42. Iodrin	GCMS GC	625	6410 b		Note 4, p. 30; Note 6, p. S73.
43. Linuron	GC				Note 3, p. 104; Note 6, p. S64.
44. Malathion	GC		6630 C		Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
45. Methiocarb	GC				Note 3, p. 94; Note 6, p. S60.
46. Methoxychlor	GC		6630 B & C	D3086-90	Note 3, p. 7; Note 4, p. 30.
47. Methyl parathion	TLC				Note a, p. 94; Note a, p. S60.
a Mirex	GC		6630 B & C		Note 3, p. 7.
Monuron	GC				Note 3, p. 104; Note a, p. S64.
50. Monuron	GC				Note 3, p. 104; Note 6, p. S64.
61. Nuburon	TLC				Note a, p. 104; Note 6, p. S64.
52. Parathion methyl	GC		6630 C		Note 3, p. 25; Note 4, p. 30.
Parathion ethyl	GC		6630 C		Note 3, p. 25.
PCMB	GC		6630 B & C		Note 3, p. 7.
55. Permethrin	GC			D3086-90	
56. Prometon	GC				Note 3, p. 83; Note 6, p. S68.
57. Prometryn	GC				Note 3, p. 83; Note 6, p. S68.
58. Propazine	GC				Note 3, p. 83; Note 6, p. S68.
59. Propham	TLC				Note 3, p. 104; Note 6, p. S64.
Propoxur	GC				Note a, p. 94; Note 6, p. S60.
61. Pyrethrin	GC				Note 3, p. 83; Note 6, p. S68.
62. Pyrethrin	GC				Note 3, p. 104; Note 6, p. S64.
63. Simazine	GC				Note 3, p. 83; Note 6, p. S68.
64. Strobil	GC		6630 B & C		Note 3, p. 7.
Sweep	GC				Note 3, p. 104; Note 6, p. S64.
66. 2,4,5-T	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
67. 2,4,5-TP (Silver)	GC		6640 B		Note 3, p. 115.
68. Terbutylazine	GC				Note 3, p. 83; Note 6, p. S68.
69. Toxaphene	GC GCMS	608 625	6630 B & C 6410 B	D3086-90	Note 3, p. 7; Note 4, p. 30.
m. Trifluralin	GC		6630 B		Note 3, p. 7.

Table 1D Notes:  
<sup>1</sup> Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 1C, where entries are listed by chemical name.  
<sup>2</sup> The full text of Methods 608 and 625 are given at appendix A, "Test Procedures for Analysis of Organic Pollutants," of this part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this part 136.  
<sup>3</sup> Methods for Benzidine, Chlorinated Organic Compounds, Pesticides, and Pesticides in Water and Wastewater, U.S. Environmental Protection Agency, September, 1978. This EPA publication includes then-layer chromatography (TLC) methods.  
<sup>4</sup> Methods for Analysis of Organic Compounds in Water and Fluvial Sediments, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).  
<sup>5</sup> The method may be extended to include α-BHC, β-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.



from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Cost: 313.50 (subject to change). Table IE, Note 2.

(18) "Official Methods of Analysis of the Association of Official Analytical Chemists", Methods manual, 15th Edition (1980). Price: \$240.00. Available from: The Association Of Official Analytical Chemists, 2200 Wilson Boulevard, Suite 400, Arlington, VA 22201. Table IB, Note 3.

(19) "American National Standard on Photographic Processing Effluents," April 2.1415. Available from: American National Standards Institute, 1439 Broadway, New York, New York 10018. Table IB, Note 9.

(20) "An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color." NCASI Technical Bulletin No. 253, December 1961. Available from: National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, NY 10016. Cost available from publisher. Table IB, Note 19.

(21) Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976. Technicon Auto Analyzer II. Method and price available from Technicon Industrial Systems, Tarrytown, New York 10591. Table IB, Note 7.

(22) Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Method price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 14.

(23) OIC Chemical Oxygen Demand Method, 1978. Method and price available from Oceanography International Corporation, 512 Wat Loop, P.O. Box 2980, College Station, Texas 77840. Table IB, Note 13.

(24) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Method and price available from ORION Research Incorporation, 840 Memorial Drive, Cambridge, Massachusetts 02138. Table IB, Note 16.

(25) Bicinchoninate Method for Copper. Method 8506, Hach Handbook of Water Analysis, 1979. Method and price available from Hach Chemical Com-

pany, P.O. Box 300, Loveland, Colorado 80537. Table IB, Note 19.

(26) Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976. Bran & Luebbe (Technicon) Auto Analyzer II. Method and price available from Bran & Luebbe Analyzing Technologies, Inc. Elmsford, N.Y. 10523. Table IB, Note 21.

(27) 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water, Hach Method 8008, 1980. Method and price available from Hach Chemical Company, P.O. Box 369 Loveland, Colorado 80537. Table LB, Note 22.

(28) Periodate Oxidation Method for Manganese, Method 8034, Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 23.

(29) Nitrogen, Nitrite—Low Range, Diazotization Method for Water and Wastewater, Hach Method 8507, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 25.

(30) Zincon Method for Zinc, Method 8009, Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 33.

(31) "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," by R.F. Addison and R.G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164. Cost available from publisher. Table IB, Note 28.

(32) "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes", Method AES 0029, 1986-Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923. Table B, Note 34.

(33) "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals, CEM Corporation, P.O. Box 200, Matthews, North Carolina 28105-0200, April 16, 1982. Available from

the CEM Corporation. Table IB, Note 36.

(c) Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director upon the recommendation of the Director of the Environmental Monitoring Systems Laboratory—Cincinnati.

(d) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring Systems Laboratory—Cincinnati, additional alternate test procedures for nationwide use.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters cited in Tables IA, IB, IC, ID, and IE are prescribed in Table II. Any person

may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded, by the Regional Administrator, to the Director of the Environmental Monitoring Systems Laboratory—Cincinnati, Ohio for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Director of the Environmental Monitoring Systems Laboratory, the Regional Administrator may grant a variance applicable to the specific charge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./Name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum holding time <sup>4</sup>
<b>Table IA—Inorganic Tests:</b>			
1-4. Coliform, fecal and total	P, G	Cool, 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> *	6 hours.
5. Fecal coliforms	P, G	do	Do.
<b>Table IB—Organic Tests:</b>			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	Do.	Do.
4. Ammonia	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
9. Dissolved oxygen demand	P, G	Cool, 4°C	48 hours.
11. Bromide	P, G	None required	28 days.
14. Dissolved oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, G	do	Analyze within 7 days.
21. Color	P, G	Cool, 4°C	48 hours.
23-24. Cyanide, total and amenable to alkali <sup>5</sup>	P, G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid <sup>6</sup>	14 days.*
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO <sub>3</sub> to pH<2, H <sub>2</sub> SO <sub>4</sub> to pH<2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
<b>Metals:<sup>7</sup></b>			
18. Chromium VI	P, G	Cool, 4°C	24 hours.
35. Mercury	P, G	HNO <sub>3</sub> to pH<2	28 days.
3, 6-B, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 38, 37, 46, 47, 51, 62, 59-60, 62, a, 70-72, 74, 75. Metals, except chromium VI and mercury.	P, G	do	6 months.
38. Nitrate	P, G	Cool, 4°C	4-6
39. Nitrite	P, G	Cool, 4°C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
42. Organic carbon	P, G	Cool, 4°C, HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2	Do.
44. Total suspended solids	P, G	Filter, 1.2 µm, Cool, 4°C	48

TABLE II—POLLUTANT CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Table with 4 columns: Parameter No./Name, Container, Preservation, Maximum holding time. Includes parameters like Oxygen, Dissolved Probe, Wobler, Phenols, Pesticides, and various acids/bases.

Table II Notes: 1. P, A, T, or G (P) or Glass (G). 2. Sample should be preserved at the time of collection. 3. When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation's Hazardous Materials Regulations (49 CFR part 172).

1. Samples should be filtered immediately on-site before adding preservatives for dissolved metals. 2. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds. 3. Samples receiving no pH adjustment must be analyzed within seven days of sampling.

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure. (b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

studies of the applicability of the alternate test procedure to the effluents in question.

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. Any application for an alternate test procedure under this paragraph (d) shall:

- (1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge aerial number. (2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested. (3) Provide justification for using testing procedures other than those specified in Table I. (4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

- (1) Provide the name and address of the responsible person or firm making the application. (2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested. (3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified Industrial or other categories. (4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976]

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will occur has final responsibility for ap-

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## Tables IA, IB, IC, ID, IE

and

## Table II

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and shall state the full name, address, and telephone number of the person giving notice.

(c) *Identification of counsel.* All notices shall include the name, address, and telephone number of the legal counsel, if any, representing the person giving notice.

#### § 136.13 Timing of notice.

No action may be commenced under section 1449(a)(1) or (a)(2) until the plaintiff has given each of the appropriate parties sixty days notice of intent to file such an action. Actions concerning injection wells disposing of hazardous waste which allege jurisdiction solely under section 7002(c) of the Resource Conservation and Recovery Act may proceed immediately after notice to the appropriate parties.

### PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

#### Sec.

136.1 Applicability.

136.2 Definitions.

136.3 Identification of test procedures.

136.4 Application for alternate test procedures.

136.5 Approval of alternate test procedures.

APPENDIX A TO PART 136—METHODS FOR ORGANIC CHEMICAL ANALYSIS OF MUNICIPAL AND INDUSTRIAL WASTEWATER

APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11

APPENDIX C TO PART 136—INDUCTIVELY COUPLED PLASMA—ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES METHOD 200.7

APPENDIX D TO PART 136—PRECISION AND RECOVERY STATEMENTS FOR METHODS FOR MEASURING METALS

AUTHORITY: &a. 301, 304(h), 307 and 501(a), Pub. L. 95-217, 91 Stat. 1568, et seq. (33 U.S.C. 1251, et seq.) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

#### § 136.1 Applicability.

The procedures prescribed herein shall, except as noted in § 136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(a) An application submitted to the Administrator, or to a State having an approved NPDES program for a permit under section 402 of the Clean Water Act of 1977, as amended (CWA), and/or to reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under parts 122 to 125 of Title 40, and,

(b) Reports required to be submitted by discharges under the NPDES established by parts 124 and 125 of this chapter, and,

(c) Certifications issued by States pursuant to section 401 of the CWA, as amended.

(38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 28, 1984)

#### § 136.2 Definitions.

As used in this part, the term:

(a) Ad means the Clean Water Act of 1977, Pub. L. 95-217, 91 Stat. 1568, et seq. (33 U.S.C. 1161 et seq.) (The Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

(b) *Administrator* means the Administrator of the U.S. Environmental Protection Agency.

(c) *Regional Administrator* means one of the EPA Regional Administrators.

(d) *Director* means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) *National Pollutant Discharge Elimination System (NPDES)* means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) *Detection limit* means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth at appendix B of this part.

(38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 28, 1984)