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INTERNATIONAL TECHNOLOGY CORPORATION

QUALITY ASSURANCE MANUAL
IT ANALYTICAL SERVICES

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STATEMENT OF MANAGEMENT POSITION

IT Corporation is committed to providing quality services for environmental management; services which meet the needs of our clients, satisfy regulatory requirements, and are commensurate with the current state of the art.

To satisfy our clients' quality objectives, to meet regulatory requirements, and to comply with IT Corporate-wide requirements, IT Analytical Services (ITAS) has adopted a comprehensive Quality Assurance Program. The principles and practices of the Program apply to every employee at every level within ITAS; they are fundamental to the way we do business and to the services we provide.

The Quality Assurance Manual is an overall statement of Program policy. This Manual provides guidance to ITAS personnel in fulfilling their responsibilities, and serves as a statement to external parties of ITAS' commitment to quality.

Implementation of the Program is the responsibility of all ITAS personnel. Management at every level has the commitment, duty, and authority to insist that these responsibilities are met, and that the principles and practices of the Program are followed and implemented.

Quality Assurance Coordinators are assigned in all ITAS laboratories to see that the Program is implemented on a day-to-day basis as intended. Each Coordinator has the authority and duty to stop work if and when necessary to satisfy Program requirements.

To verify that the Quality Assurance Program is successfully implemented, the laboratories are independently audited by the ITAS Director of Quality Assurance/Control, who is directly responsible to the Vice President, Analytical Services. In addition, the laboratories are subject to audits by the Environmental Projects Group Director of Quality Assurance and Discipline Management, and by various regulatory authorities and other outside agencies.

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February 1, 1988

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1.0 INTRODUCTION

Analytical data are used for many purposes, including: compliance with regulatory requirements; determination of the presence, concentration, and movement of hazardous materials in the environment; potential effects upon or protection required for persons; and the actions necessary for disposal or treatment of hazardous materials.

Analytical data may be used within IT Corporation to support a broader-based project involved with: site characterization and/or remediation; on-site treatment; treatment and/or disposal at an IT facility; or health and safety protection of IT personnel and the public. Data may also be produced for outside commercial testing and submitted directly to clients for their decision making.

It is the purpose of the ITAS Quality Assurance Program, as expressed in this Quality Assurance Manual, to provide data which are of acceptable quality. To achieve this, a system is described which controls:

- Preservation of samples
- Receipt and handling of samples
- Processing and analyses of samples
- Analytical equipment
- Data verification
- Data reporting
- Records management
- Management review.

ITAS recognizes that all laboratory personnel affect data quality. This Manual has been prepared so that staff members will be cognizant of the procedures adopted by ITAS for the production of analytical data, and so they will be aware of their responsibilities. This Manual applies to chemical analysis only; microbiology, bioassay, geotechnical and radiological analyses are described in Laboratory-Specific Attachments to this Quality Assurance Manual.

The remainder of this introductory section discusses:

- Quality Assurance objectives of ITAS, which are subsequently discussed in detail in this Manual
- The organization of ITAS
- Quality Assurance documents within ITAS
- Control, distribution, and revision of Quality Assurance documents.

Within this Manual, the terms Quality Assurance and Quality Control are defined and used as follows:

- Quality Assurance - The overall controls imposed upon laboratory operations. Actions taken by personnel and the documentation of laboratory performance are included as specified in the Quality Assurance Program.
- Quality Control - The daily, specific actions taken within the laboratory to verify sample integrity, performance of analyses, data processing, and record maintenance.

1.1 OBJECTIVES OF THE QUALITY ASSURANCE PROGRAM

As stated, the overall objective of the Quality Assurance Program for the ITAS laboratories is to provide data of known quality. To accomplish this, each laboratory must:

- Maintain an effective, ongoing Quality Control Program to measure and verify laboratory performance.
- Meet data requirements for accuracy, precision, and completeness through the use of proven methodologies.
- Provide sufficient flexibility to allow controlled changes in routine methodology to meet specific data requirements.

- Monitor operational performance of the laboratory on a routine basis and provide corrective action as needed.

- Recognize and promptly correct for any factors which adversely affect quality.

- Maintain complete records of sample submittal, laboratory performance, and completed analyses to verify resulting data.

Specifically, the following actions are taken:

- Procurement and control of instrumentation and supplies required for laboratory operation

- Sample receipt, chain-of-custody completion, and sample storage

- Calibration and preventive maintenance of instrumentation

- Establish a Quality Control sample program within the laboratory so that statistical evaluation of analyses can be done

- Analyses in accordance with recognized analytical procedures

- Data processing, validation, and reporting

- Control and maintenance of laboratory records

- Identification and resolution of nonconformances requiring corrective action

- Audits to verify laboratory performance and the reporting of audit results to management

- Training of analysts in technical and quality control procedures, including an orienetation to the ITAS Quality Assurance Program.

1.2 ITAS QUALITY ASSURANCE DOCUMENTS

Complete documentation of ITAS operations requires several types of ITAS Quality Assurance documents. This Quality Assurance Manual provides

overall policy for ITAS. Other documents provide supplementary information or project-specific requirements. Each of the document types is discussed below. Figure 1-1 illustrates the basic purpose of each document.

- Quality Assurance Manual - The Quality Assurance Manual provides ITAS Quality Assurance policy. The Manual discusses all aspects for Quality Assurance and Quality Control, both administrative and technical. However, it is not intended that the Manual provide in-depth technical discussion. The Quality Assurance Manual has precedence in policy matters over all other ITAS quality-related documents. The Manual is applicable to and provides direction for all ITAS analytical laboratories.

- Laboratory-Specific Attachments - Laboratory-Specific Attachments to the Quality Assurance Manual are prepared for two purposes:

  - If an individual ITAS laboratory provides services that are unique to that laboratory, the service and necessary Quality Assurance requirements will be described in an attachment.

  - Certain aspects of laboratory operations discussed in the Quality Assurance Manual are dependent upon laboratory facilities. Quality Assurance activities such as preventive maintenance, calibration, and data verification are functions of the equipment in a specific laboratory. Attachments will be prepared by each laboratory to document their specific operations. Laboratory-Specific Attachments are adopted as part of the Quality Assurance Manual for the preparing laboratory.

- Manuals of Practice - Manuals of Practice are developed to provide in-depth technical discussions of specific topics. For example, a Manual of Practice for the field collection, preservation, and shipment of samples to ITAS laboratories provides specific uniform direction to IT personnel. Manuals are usable across ITAS laboratories. A Manual may be a collection of SOPs.

- Standard Operating Procedures (SOPs) - These are written, detailed instructions describing the specific laboratory operations and performance of routine laboratory tasks. They specify what is done, whose responsibility it is to perform tasks and whose to verify their correctness; they are sufficiently detailed to provide data of acceptable quality and integrity, with a minimum loss of data due to out-of-control situations. They also provide for documentation to record the performance of all tasks and their results, and demonstrate the validation of the data each time the data are recorded, calculated, or transcribed. SOPs address the major elements upon which analytical quality depends.

  - Organization and personnel
  - Facilities and equipment
  - Analytical methodology
  - Sample custody procedures
  - Quality control
  - Data handling.

- Project-Specific Manuals - Frequently, contractual and regulatory demands, or uniqueness of a project's scope of work, require the preparation and implementation of a Project-Specific Quality Assurance Program and Manual, generally referred to as a Quality Assurance Project Plan (QAPP). Project demands can include:

  - The development and/or use of new testing methods

  - Different requirements for equipment calibration and maintenance

  - Different detection limits for testing

  - Different accuracy and precision limits or the statistical treatment of data

  - Additional or different document and record formats and maintenance.

  If a specific project requires a unique Quality Assurance Program, that program will be implemented. Full documentation will be provided in a Project-Specific Manual. The requirements of the project will take precedence over conventional ITAS Quality Assurance practices for that work.

1.3 DOCUMENT CONTROL, DISTRIBUTION, AND REVISION

The Quality Assurance Manual, Attachments, Manuals of Practice, SOPs, and Project-Specific Manuals are approved and controlled documents.

Approvals required before issue for the various documents follow:

- ITAS Quality Assurance Manual
  - Senior Vice President, Environmental Projects Group
  - Vice President, IT Analytical Services
  - Director, Quality Assurance/Control, ITAS
  - Director, Quality Assurance and Discipline Management, Environmental Projects Group
- Laboratory-Specific Attachment
  - Laboratory Manager
  - Regional Director, Analytical Operations
  - Director, Quality Assurance/Control, ITAS
  - Director, Quality Assurance and Discipline Management, Environmental Projects Group
- Manuals of Practice
  - Vice President, IT Analytical Services
  - Director, Quality Assurance/Control, ITAS
- Standard Operating Procedures
  - Laboratory Manager
  - Director, Quality Assurance/Control, ITAS
  - Laboratory Quality Control Coordinator
- Project-Specific Manuals
  - Laboratory Manager
  - Laboratory Quality Control Coordinator
  - Project Manager (if analytical services are part of a broader-based project, and if required by contract).

Approval of these documents shall be denoted by a signature-and-date page in each document, which includes the required signatures above and the manual author. In addition, IT prepared documents shall be reviewed and approved by regulatory agencies, as appropriate.

In general, documents will be distributed as needed to all ITAS

personnel. When the document is no longer needed or the copyholder leaves IT Corporation, it shall be returned. Copies distributed within IT and ITAS will be numbered for document control, or as uncontrolled documents. Distribution of the Quality Assurance Manual and Manuals of Practice will be by the Director, Quality Assurance/Control, ITAS. Laboratory-Specific Attachments and Project-Specific Manuals (QAPP), and SOPs will be distributed by the appropriate Laboratory Managers and QC Coordinators.

So that all Quality Assurance documents can be revised as necessary, distribution lists will be maintained, as applicable, by the ITAS Quality Assurance Staff and Laboratory Managers. Before issue, revisions will require the same approvals as the original document. Also, revisions will be consecutively numbered and each page revised shall denote the numbered revision. To facilitate revisions, each page of all manuals shall contain the following:

Title or Document Identification
Section No. _____
Revision No. _____
Date: (of issue)
Page ___ of ___

Issuing a revision requires a revised signature page.

Revisions will be issued to all holders of controlled copies. Each copyholder will sign a revision receipt verifying that the revision has been received and properly placed in the document. The receipt will be returned to the issuing manager.

Copies of Quality Assurance documents will be issued external to IT Corporation as required. These documents may be controlled (numbered) or uncontrolled (unnumbered) copies depending upon the reason for issue. Controlled copies will be revised in accordance with the above. In all cases, external parties will be asked to return Quality Assurance

documents when the need for the document has ended. Exceptions to this will be if the external party requires a copy for record purposes.

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DOCUMENT	PURPOSE
QUALITY ASSURANCE MANUAL	• Provides ITAS Quality Assurance policy • States responsibilities of laboratory staff • Describes basic Quality Control practices
LABORATORY-SPECIFIC ATTACHMENT	• States supplementary implementing practices and services of a specific laboratory • For specific laboratory; becomes incorporated with Quality Assurance Manual
MANUALS OF PRACTICE	• Detailed discussion of specific technical subjects • Provides detailed information related to technical topics discussed in the Quality Assurance Manual
STANDARD OPERATING PROCEDURES	• Detailed description of specific tasks relating to laboratory operation and procedures
PROJECT-SPECIFIC MANUAL (QAPP)	• Prepared as required for specific contract • May supplement or change ITAS Quality Assurance practices for specific project • Takes precedence over other ITAS documents for a specific project

FIGURE 1-1

ITAS QUALITY ASSURANCE DOCUMENTS

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2.0 LABORATORY ORGANIZATION

This section discusses general positions and quality-related responsibilities which each laboratory must provide for the implementation of the Quality Assurance Program and completion of Quality Control activities. Also discussed is the role of the IT Analytical Services Quality Assurance/Control Director.

2.1 ITAS QUALITY-RELATED RESPONSIBILITIES WITHIN A LABORATORY

The positions discussed below are part of ITAS laboratories. The quality-related responsibilities may be modified in the Laboratory-Specific QA Attachments' organization section:

- Laboratory Manager
  - Report directly to Vice President, IT Analytical Services, or Regional Director Analytical Operations
  - Implement the Quality Assurance Program within the laboratory
  - Periodically determine the effectiveness of the Quality Assurance Program in the laboratory
  - Approve Laboratory-Specific Attachments to the Quality Assurance Manual and Project-Specific Manuals (QAPPs), SOPs, and revisions
  - Recommends to the Quality Assurance/Control Director, ITAS, changes in the Quality Assurance Program
  - Issues laboratory reports
  - Maintain current laboratory organization chart
  - Supervise laboratory participation in inter-laboratory accreditation and proficiency programs.

- Technical Director

  - Provides technical overview of laboratory activities

  - Serves as an "in-house" consultant for the applicability of general Quality Control practices to specific needs

  - Leads the training of analysts in laboratory operations and analytical procedures

  - Evaluates analytical techniques, procedures, instrumentation and Quality Control procedures, and provide recommendations to the Laboratory Manager

  - Supervises the verification of software for data processing

  - Recommends standards for purchasing instrumentation, equipment, reagents, gases, and chemicals

  - Defines the instrument preventive maintenance schedule

  - Defines the calibration program within the laboratory.

- Quality Control Coordinator:

  - Prepares Quality Control standards, arranges insertion of Quality Control samples into the laboratory sample stream, and reviews the results

  - Performs statistical analyses utilizing results of QC samples analyses

  - Informs the Operations Manager and Group Leaders of data which lies outside of acceptable limits

  - Serves as the "focal point" for the reporting and disposition of nonconformances

  - Resolves ongoing and recurring nonconformances within the Laboratory

- Recommends corrective actions for resolution of nonconformances

- Reviews statistical data to verify the laboratory is meeting stated Quality Control goals

- Maintains current distribution lists for Laboratory-Specific Attachments and Project-Specific Manuals and Standard Operating Procedures

- Closes findings and recommendations of Quality Assurance Audits

- Notifies the Technical Director, Operations Manager, and Laboratory Manager of out-of-control situations

- Stops production of data in a laboratory area where the review of quality control data or procedures shows significant problems

- Reports nonconformances to the ITAS Quality Assurance/Control Director or the Director, Quality Assurance, Environmental Project Group if the situation is not corrected within the laboratory

- Assists in the performance of Quality Assurance audits and performs Quality Control audits

- Establishs and supervises the laboratory Quality Assurance training program.

• Operations Manager/Assistant Laboratory Manager:

- Oversees the log-in of all samples received, completion of chain-of-custody records, and maintenance of sample log books

- Manages laboratory daily analytical operations

- Supervises Quality Control activities performed as part of routine analytical operations

- Supervises sample storage facilities

- Supervises the preparation and maintenance of laboratory records

- Oversees preventive maintenance program.

- Group Leaders:

  - Serve as the Lead Analyst within the group (group meaning organic analysis, inorganic analysis, special projects, etc.)

  - Organize and schedule the analytical testing program with consideration for sample-holding times

  - Implement data verification procedures

  - Assign analysts for data processing and validation activities

  - Review and approve all analytical data and submit to Operations Manager and Laboratory Manager for issue

  - Evaluate instrument performance and supervise instrument calibration and preventive maintenance programs

  - Report out-of-control or nonconforming situations to Operations Manager, Laboratory Manager, and Quality Control Coordinator, as appropriate.

- Analysts

  - Perform analytical procedures and data recording in accordance with accepted methods

  - Perform and document calibration and preventive maintenance of instrumentation, as appropriate

  - Perform data processing and validation

  - Immediately report out-of-control situations, instrument malfunction, calibration failure, or other nonconformances to the Group Leader and Quality Control Coordinator, as appropriate.

2.2 ITAS QUALITY ASSURANCE RESPONSIBILITIES OUTSIDE OF THE LABORATORY

Ultimate responsibility for Quality Assurance within the laboratories

lies with the Vice President, IT Analytical Services. To fulfill this responsibility, the Vice President has assigned responsibility within the laboratory to the respective Regional Directors Analytical Operations and Laboratory Managers. External review of laboratory quality performance is assigned to the IT Analytical Services Quality Assurance/Control Director.

The Director, Quality Assurance/Control, reports directly to the Vice President and is independent of laboratory management. This independence allows the Director to assess laboratory quality performance without being influenced by schedule and cost considerations. Responsibilities of the Quality Assurance/Control Director are:

- Approves ITAS Quality Assurance documents
- Performs audits of ITAS laboratories (audits are discussed in Section 14.0) to verify compliance with the Quality Assurance Program
- Verifies completion of corrective action cited in audits
- Acts as the collection point for proposed changes in the Quality Assurance Program and initiate changes in the program
- As necessary, discusses with Laboratory Managers and the Vice President unresolved nonconformances brought to the Director's attention by Quality Control Coordinators.

IT Analytical Services is a Division of the Enviromental Project Group. The Director, Quality Assurance and Discipline Management for the Group provides a further independent quality assurance review by:

- Participating in laboratory audits with the ITAS Quality Assurance/Control Director

- Approving the ITAS QA Manual and its Laboratory-Specific Attachments

- Maintaining communication so that QA problems which are not readily resolved within ITAS or are detrimental to the performance of a project within the Group are brought to the Group Director's attention for resolution.

IT Analytical Services has several Regional Directors of Analytical Operations, each responsible for two or more laboratories within a particular region. Quality Assurance duties of these Directors include:

- Providing resources for each laboratory's QA Program's effective implementation

- Reporting directly to the Vice President, IT Analtyical Services

- Periodically determining the effectiveness of the QA Programs at each laboratory

- Recommending changes in the QA Program to the Director, Quality Assurance/Control, ITAS.

3.0 STANDARD LABORATORY PRACTICE

Daily activities within an analytical laboratory are directed toward the analysis of samples. However, there are many laboratory functions which precede and follow analysis that are necessary to control and verify the analysis. The functions, as a total, represent the daily implementation of the Quality Assurance Program by the laboratory staff.

This section serves as an introduction to these functions and provides an overview of them as the total system providing Quality Assurance within the laboratory. Figure 3-1 presents a flow chart of functions for laboratory analyses. The functions shown can be divided into those which directly involve a sample, laboratory functions, and those which are independent (in general) of specific sample analysis, auxiliary functions.

Each function is affected by those which precede it. Therefore, it is imperative that each function be controlled in a specified manner. It is the purpose of the Quality Assurance Program to provide control by specifying how functions are to be performed, and by providing "checks and balances" to verify performance.

Each function is discussed in detail in the sections which follow (Sections 4.0 through 13.0 as denoted in Figure 3-1). Following is a brief discussion of the activities performed for each function and their role in the Quality Assurance Program.

3.1 RECEIPT OF SAMPLES AND INITIATION OF TESTING PROGRAM

Upon receipt of samples in the laboratory, the following is done:

- Samples are examined for damage, checked for proper preservatives and temperature
- The Chain of Custody Form is signed
- Samples are placed in the proper storage environment

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- The testing program is defined by the Request for Analysis form or Purchase Order, or other appropriate means
- Acceptable holding times until samples must be analyzed are defined
- Samples are logged into the laboratory sample stream
- Appropriate laboratory personnel are notified of sample receipt.

3.2 MATERIAL AND INSTRUMENT PREPARATION

Concurrent with the performance of analysis, auxiliary functions are performed to provide appropriate materials to the Analysts, and verify and maintain instrument performance.

Material Procurement and Control

- Specifying grades of reagents, solvents, gases, and water used within the laboratory for specific analyses, and verifying adequacy of these materials before use
- Specifying material composition and volumetric tolerance of laboratory vessels
- Controlling reagents, solvents, etc., during storage
- Cleaning protocol for laboratory vessels.

Calibration

- Scheduled comparison of instrument performance against national standards for instruments which measure physical parameters, such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

- Determination of instrument response to known chemical composition and concentration. Calibration may be part of daily instrument usage to define response for post analysis data processing of analytical raw data, or as response checks during or at the completion of a instrument run. This type of calibration may be independent of or part of the analysis for a specific project.

Preventive Maintenance

- Servicing instruments on a scheduled basis to maintain performance
- Maintaining a stock of instrument parts which are known to regularly degrade because of usage
- Preparing log books so that the historical performance of an instrument can be assessed.

3.3 ANALYTICAL PROCEDURES

The actual analysis of samples occurs during this function; however, for the analysis to be complete, several activities must be coincident:

- Sample holding times and storage environment are reviewed by the Analyst to verify that sample integrity has been maintained
- Analysis is performed in accordance with standard methods adopted by ITAS or as specified by the client. Analysis results include:
  - Prescribed daily instrument calibration or instrument tuning and documentation
  - Preparation and analysis of Quality Control samples and/or standards as part of the sample stream at the level of samples required.

3.4 PROCESS QUALITY CONTROL DATA

There are four steps involved in the total verification of analytical data:

- Computation of Quality Control sample data

- Comparison of Quality Control data with analytical acceptance limits
- Computation of analytical results from the data
- Independent validation of analytical results.

In general, Quality Control data and analytical data are interconnected. However, for the purposes of this discussion, the preparation of Quality Control data and the processing and evaluation of analytical data will be discussed separately because of the difference in quality assurance requirements for each.

Section 3.4 discusses Quality Control data, and Section 3.6 discusses the treatment of analytical data.

Quality Control samples results are analyzed and the QC data are reviewed and compared against stated acceptance levels for accuracy and precision. The data are used, as appropriate, to update control charts. If the QC data meets acceptance levels, processing of the analytical data begins. If the QC data are unacceptable, corrective action must be taken.

3.5 CORRECTIVE ACTION

If Quality Control data are unacceptable, the cause must be determined. If the cause can be resolved so that the integrity of the analytical data are not affected or can be corrected, the processing of the analytical data can proceed. Nonconformances which affect the integrity of analytical data will require resolution which may include reanalysis of the affected samples. Results shall be qualified in the report.

3.6 DATA PROCESSING AND VALIDATION

- Analytical test results are calculated by the

Analyst using computational methods prescribed for the various analytical methods

- The resulting data are validated, as required, by an individual independent of the analysis. Validation demonstrates and documents that the analyses have been properly performed, that proper input parameters (such as response factors or dilutions) have been used, and that mathematical manipulation is correct.

3.7 REPORTING

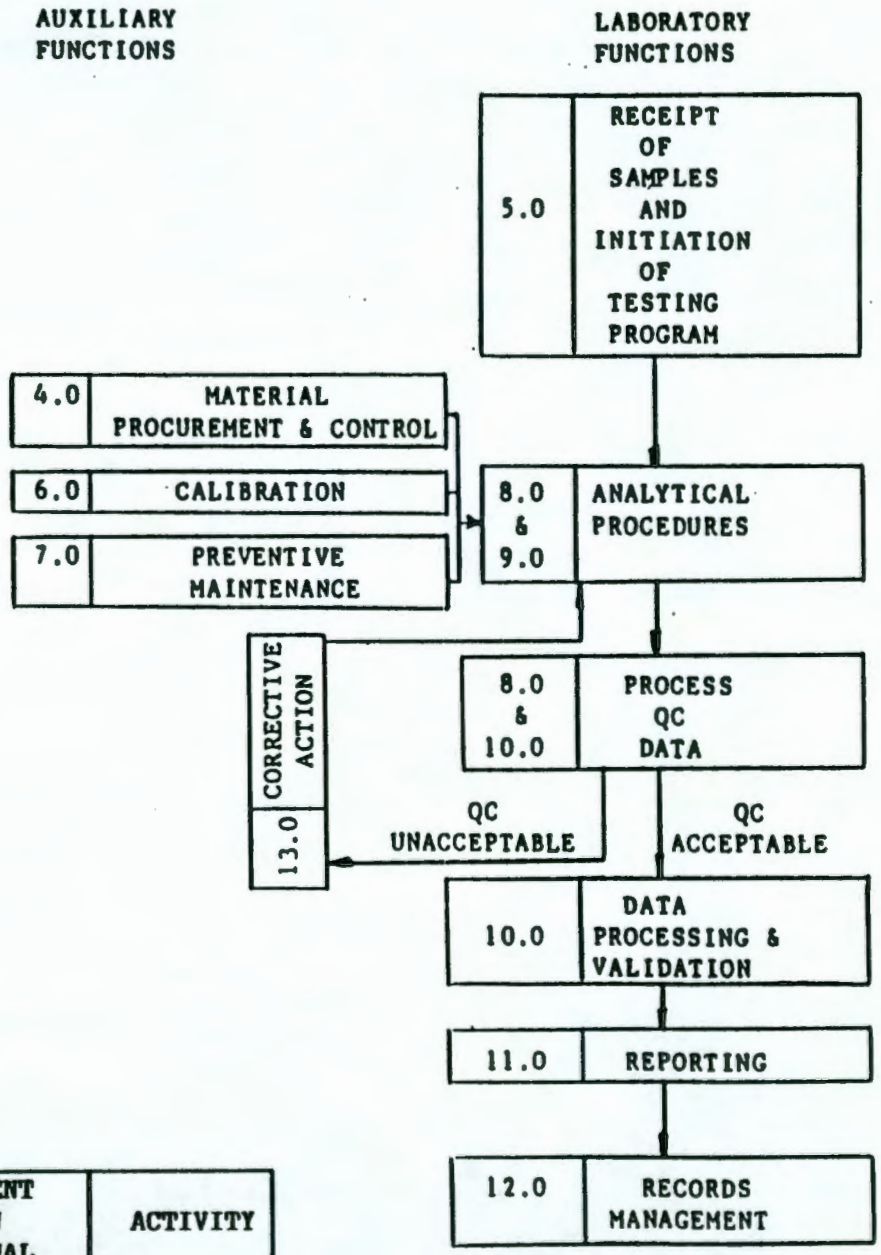
- Analytical data, and Quality Control data if appropriate, are summarized in presentation format
- The data are reviewed by Group Leaders to verify that the objectives of the analysis have been met
- After approval the data are issued by the Operations Manager and/or the Laboratory Manager.

3.8 RECORDS MANAGEMENT

There are two categories of records prepared within an ITAS laboratory:

- Project-specific records which are related solely to the analysis performed for a group of samples such as chain-of-custody and raw analytical data
- Records which demonstrate overall laboratory operation, and are, in general, independent of specific projects, such as the master laboratory sample log-in book, instrument performance, and maintenance log books and control charts.

Separate files are maintained for each category of records. However, the document control system enables cross-referencing of records (such as for instrument performance and maintenance log books) so that implementation of the Quality Assurance Program can be demonstrated.



ACTIVITIES

- Complete chain of custody
- Sample log in
- Define tests to be performed on specific samples
- Define QC sample requirements

- Analysis of samples
- Analysis of QA samples
- Summarize QC data
- Update control charts
- Statistical treatment of data

- Calculate analysis results
- Independent data review

- Summarize data
- Issue within IT or to client

- Prepare project file
- Maintain records

FIGURE 3-1

LABORATORY ANALYSIS FLOW CHART

4.0 MATERIAL PROCUREMENT AND CONTROL

The quality of reagents, solvents, gases, water, and laboratory vessels used in analyses must be known so that their effect upon analytical results can be defined. Materials purchased by an ITAS Laboratory shall meet the requirements stated below or as denoted in specific analytical procedures, and be controlled as stated. Requirements are also stated for internally prepared materials such as water and compressed air.

Each laboratory shall assign an individual responsible for purchasing materials and controlling them in the laboratory. This person can be the Quality Control Coordinator or other as assigned by the Laboratory Manager. Responsibility for this work shall be defined in the respective Laboratory-Specific Attachment to this Manual and the laboratory organization charts. Duties include:

- Specifying in purchase orders or requisitions, suitable grades of materials (grade should be defined by the QC Coordinator or Group Leaders)
- Verifying upon receipt that materials meet requirements and that, as applicable, material certificates are provided and maintained in the laboratory record system
- Identifying and storing materials
- Verifying that material storage is properly maintained, and removing materials from use when shelf life has expired.

4.1 REQUIREMENTS FOR REAGENTS, SOLVENTS, AND GASES

Chemical reagents, solvents, and gases are available in a variety of grades of purity, ranging from technical grade to ultrapure grades. The purity required varies with the type of analysis. The parameter measured and the sensitivity and specificity of the detection system are important factors in determining the purity required. For many analyses, including most inorganic analyses, analytical reagent (AR) grade is satisfactory. Other analyses, such as trace organic and radiological,

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frequently require special ultrapure reagents, solvents, and gases. If the analytical method does not specify the purity of materials, it is generally intended that analytical grade be used. Materials of lesser purity than specified shall not be used. Prior to performing an analysis, the label of the material container should be checked by the Analyst to verify that the material purity meets analytical needs.

Materials are prepared and standardized against reliable primary standards whenever possible. They are restandardized, or prepared fresh, as often as indicated by their stability.

4.1.1 General Inorganic Analyses

In general, AR grade reagents and solvents are adequate for inorganic analyses. Primary standard reagents shall be used for standardizing all volumetric solutions. All prepared reagents shall be checked for accuracy.

Individual analytical methods specify the reagents that require frequent standardization, or special treatment. The Analyst must comply with these special operations. To minimize potential deterioration, the Analyst should prepare a limited volume of such reagents, depending on the quantity required over a given period of time.

4.1.2 Trace Metals Analyses

All standards used for atomic absorption and emission spectroscopy shall be spectro-quality. It is recommended that other reagents and solvents also be spectro-quality. Standards are prepared by the Analyst, or purchased provided purchased materials meet the requirements of the analytical method.

In general, fuel and oxidant gases used for atomic absorption can be commercial grade. Compressed air can be commercially supplied, dry grade, or supplied by laboratory air compressors if adequate pressure is

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maintained and the air is filtered to remove oil, water, and possible trace metals.

4.1.3 Organic Chemical Analyses

In general, pesticide grade (nanograde) is the minimum acceptable grade for materials used for organic analyses. Reference grade standards should be used as necessary. Special note should be made of the assay of standard materials.

Some gas chromatography (GC) detectors require that solvents, standards, and samples be free of certain classes of compounds. For example, use of the flame photometric detector requires that reagents and solvents be free of sulfur and phosphorus interference.

For sample cleanup procedures, the adsorbents most commonly used for column chromotographic cleanup of sample extracts are Florisil, carbon, silica gel, and alumina. These are preactivated according to the analytical method requirements and checked for interfering constituents. The contents of each solvent lot must be checked to determine suitability for the analyses. Similarly, all analytical reagents and other chemicals must also be routinely checked.

4.1.4 Water

Deionized water is used as appropriate for dilution, preparation of reagent solutions, and final rinsing of glassware as described in Laboratory-Specific Attachments to this Quality Assurance Manual. Distilled water is usually not of sufficient purity because distillation does not remove certain contaminants. The specifications for ASTM Type II quality water shall be met: Maximum Electrical Conductivity at 25°C of 1.0 µmho/cm, or Minimum Electrical Resistivity at 25°C of 1.0 M ohm/cm. Water quality shall be determined daily by measuring either conductivity or resistivity and recording measurements in a log book.

Organic-free water is required for microbiological and volatile organic analyses. Organic-free water may be verified by the purge-and-trap technique on the GC. However, when determining trace organics by solvent extraction and gas chromatography, specialty water such as high performance liquid chromatography (HPLC) grade water with sufficiently low background must be used. Preextraction of the water with the solvent used in the analysis, or nitrogen purging, may be helpful in eliminating organic compounds from the water.

4.1.5 Compressed Air

Compressed air must be free of oil, water, and dirt. If produced in the laboratory, appropriate filters shall be used at the compressor to prevent moisture from entering the piping system. Filters to remove oil shall be installed at the point of use for atomic absorption and gas chromatographic applications.

If purchased, or as a backup to an in-house compressor system, compressed air shall be high quality, dry grade.

4.2 REQUIREMENTS FOR LABORATORY CONTAINERS

Containers used in the laboratories can affect the quality of results. Material composition and volumetric tolerances are discussed below.

4.2.1 Material Composition of Laboratory Vessels

Soft glass containers are not recommended for general use, especially for the storage of reagents. The glass recommended for general use is chemically resistant borosilicate glass, such as is manufactured under the trade names of Pyrex or Kimax. This glassware is satisfactory for analyses performed by an ITAS laboratory unless otherwise noted in the sampling or testing procedure.

The use of plastic vessels; containers; and other apparatus made of Teflon, polyethylene, polystyrene, and polypropylene is desirable for

certain specified applications.

The following guidelines should be considered when selecting the material composition of laboratory vessels:

- Borosilicate or polyethylene bottles are to be used for the storage of reagents and standard solutions, unless otherwise specified.
- Plastic containers should not be used for reagents and solvents used in organic analyses.
- Dilute metal solutions have a tendency to plate out on container walls over long periods of time; therefore, dilute metal standard solutions should be prepared at the time of analysis.
- The use of disposable glassware is satisfactory for some analyses, such as the use of disposable test tubes as sample containers for use with some automatic samplers.
- Borosilicate glassware is not completely inert, particularly to alkalis. Standard solutions of silica, boron, and the alkali metals should be stored in polyethylene bottles.

4.2.2 Volumetric Container Specifications

ITAS laboratories shall use glassware of sufficient accuracy as required for the analytical procedure for the measurement of sample or reagent volumes. This includes volumetric flasks, volumetric pipets, and accurately calibrated burets. Less accurate types of glassware, including graduated cylinders and serological and measuring pipets, have specific uses when less exact volumes are permitted by the analytical procedure.

In general, volumetric containers will not be calibrated by ITAS unless required by a specific analytical method. However, containers, primarily glassware, shall be purchased with the objective of meeting the correct end use of the container in an analytical procedure. Thus, for

example, if an analytical method requires that Class A glassware be used, the Analyst shall fully comply with the method.

4.3 STORING AND MAINTAINING REAGENTS AND SOLVENTS

The following shall apply for storing and maintaining reagents and solvents:

- Standard reagents and solvents are stored in accordance with manufacturer's recommendations
- Standard solutions shall be stored separately from samples to avoid possible cross-contamination
- Light-sensitive standard reagents or solvents are stored in a cool, dark place
- Materials used for radiological determinations are stored in dark bottles
- Organic reagent standards are stored at or below 4°C
- Organic reference materials are stored in a freezer (at or below 0°C)
- Adsorbents for thin-layer and column chromatography are stored in the containers in which they are supplied, or according to the requirements of individual analytical methods
- When fresh stock solutions are required, dilutions of the new standard are compared to the current standard or an independent Standard Reference Material to determine consistency or document traceability
- Standards are not maintained longer than recommended by the manufacturer or as specified in the analytical method.

4.4 GLASSWARE CLEANING REQUIREMENTS

Methods of cleaning glassware are selected according to the substances that are to be removed and the analytical analysis required. Water-soluble substances can be washed out with hot or cold water and the

vessel finally rinsed with successive small amounts of deionized water. Other substances more difficult to remove may require the use of a detergent, organic solvent, dichromate cleaning solution, nitric acid, or aqua regia. In all cases, it is good practice to rinse a vessel with tap water followed by deionized water as soon as possible after use. Material allowed to dry on glassware is much more difficult to remove.

Chromic acid is an effective but dangerous cleaning agent. It may be prepared by adding 1 liter of concentrated sulfuric acid slowly, with stirring, to a 35-ml saturated sodium dichromate solution. This mixture must be allowed to stand for approximately 15 minutes in the vessel to be cleaned and may then be returned to a storage bottle. Following the chromic acid wash, the vessels are rinsed thoroughly with tap water, then with small successive portions of deionized water.

For certain determinations, especially trace metals, glassware shall also be rinsed with a 1:1 nitric acid-water mixture. This is followed by thoroughly rinsing with tap water and successive portions of deionized water. This may require as many as 12 to 15 rinses, especially if chromium is determined. A nitric acid rinse is also especially important if lead is to be determined.

Glassware used for phosphate determinations should not be washed with detergents containing phosphates. This glassware must be thoroughly rinsed with tap water and deionized water. For ammonia and Kjeldahl nitrogen determination, the glassware must be rinsed with ammonia-free water.

Glassware used in the determination of trace organic constituents in water, such as chlorinated pesticides, should be as free as possible of organic contaminants. A chromic acid wash of at least 15 minutes is necessary to destroy these organic residues. Rinse thoroughly with tap water and, finally, with deionized water. Glassware may be dried for

immediate use by rinsing with redistilled acetone. Otherwise, non-volumetric glassware may be oven dried or placed in the muffle furnace at 400°C for two hours. Glassware should be stored immediately after drying to prevent any accumulation of dust and stored inverted or with the mouth of the glassware covered with foil.

Bottles used for the collection of samples for organic analyses should be rinsed successively with chromic acid cleaning solution; tap water; deionized water; and, finally, several times with a redistilled solvent such as acetone, hexane, petroleum ether, or chloroform. Caps should be washed with detergent; rinsed with tap water, deionized water, and solvent. Liners are treated in the same way as the bottles and are stored in a sealed container.

Alternate methods for cleaning may be used if it is demonstrated (such as by blank analysis) that the result is satisfactory. Also, disposable or precleaned glassware may be purchased if applicable to the analytical procedure.

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5.0 SAMPLE RECEIPT AND INITIATION OF TESTING PROGRAM

Laboratory analyses are performed to produce data representative of conditions when the sample was obtained. To provide representative samples for analysis, both field and laboratory personnel must satisfactorily perform their activities. Although the purpose of this manual is to define the laboratory Quality Assurance Program, the interrelationship of field and laboratory operations in maintaining sample integrity is briefly discussed because the effect of field operations upon resulting data quality cannot be totally separated from laboratory operations. Figure 5-1 shows the sample collection, transport, and holding process. The steps presented are the basis for the following discussion.

5.1 FIELD COLLECTION AND SHIPMENT

Prior to collecting samples, the collection team must consider the analyses to be performed and the "hazard level" of the media being sampled so that proper sample containers and shipping containers can be assembled and the proper preservatives added to samples. In addition, field logs and record sheets, Chain of Custody forms, and Request for Analysis records must be assembled.

All records required for documentation of field collection must be completed by the field team. Several of the documents that affect laboratory operations are discussed herein. The primary documenting record is the Chain of Custody form.

In addition to initiating the Chain of Custody form, field personnel are responsible for uniquely identifying (required on the Chain of Custody form) and labeling samples, providing proper preservation, and packaging samples to preclude breakage during shipment.

Referring to Figure 5-2, every sample shall be labeled to identify:

- Project number
- Unique sample number

- Sample location (such as borehole and depth, or grid coordinates)
- Sampling date and time
- Person obtaining the sample
- Method of sample preservation/conditioning.

Samples must be placed in containers compatible with the intended analysis and properly preserved to maintain sample integrity. Also, the collection of samples must consider the time interval between acquiring the sample and analysis (holding time) so that the sample is representative. Table 5-1 provides requirements for various analytical parameters with respect to the type of container, preservation method, and maximum holding time between collection and analysis.

As can be seen, polyethylene or glass containers are required; and, in most cases, samples must be cooled to four degrees Centigrade. The table also provides the recommended sample volume for a specific analysis.

High hazard (medium/high concentration) samples must be packaged according to DOT regulations. The ITAS Manual of Practice: "Sample Packaging and Shipment" should be used, in conjunction with 49 CFR, Parts 100-177, when packaging hazardous samples. The Manual also presents detailed instructions on packaging non-hazardous or environmental samples.

Shipping containers are to be sealed with custody tape prior to shipment (see Figure 5-5), whether shipped by direct transport by field personnel or commercial carrier. The only exception to this is if sufficient holding time exists so that the samples can be held in the field; then it is necessary to re-ice the containers and seal them prior to transport.

As soon as field personnel are ready to transport samples from the field to the laboratory, they shall notify the laboratory by telephone of the shipment. If the samples are transported by field personnel, the estimated time of arrival at the laboratory should be given. If the samples are shipped by commercial carrier, the laboratory should be telephoned as soon as the shipping containers are consigned to the shipper.

The final step in providing information to the laboratory is shown on Figure 5-3. The Request for Analysis form shall be completed by the field personnel and included with the Chain of Custody form. The QC level required (see Section 8.3) should be stated on this form. It is imperative that the Request for Analysis form be provided so that analytical requirements are defined and sample holding times are not exceeded. The analyses requested should be consistent with the project work plan, or Quality Assurance Project plan; the Request for Analysis form takes precedence if these documents conflict.

5.2 CHAIN OF CUSTODY

An overriding consideration for resulting data is the ability to demonstrate that the samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, laboratory custody and disposal must be documented to accomplish this. Documentation is accomplished through a Chain of Custody record that lists each sample and the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if it is:

- In a person's actual possession
- In view after being in physical possession
- Locked or sealed so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel.

Figure 5-4 is the Chain of Custody form to be used by IT personnel in collecting and shipping samples. An ITAS laboratory shall not accept samples collected by IT personnel for analysis without a correctly prepared Chain of Custody form.

The Chain of Custody form shall be signed by each individual who has the samples in their possession. Preparation of the Chain of Custody shall be as follows:

- The Chain of Custody record shall be initiated in the field by the person collecting the sample, for every sample. Every sample shall be assigned a unique identification number that is entered on the Chain of Custody form. Samples can be grouped for shipment and use a common form. The form on Figure 5-4 allows for ten samples. If more than ten samples are shipped in the same container, more than one Chain of Custody form is required.

- The record shall be completed in the field to indicate project, sampling team, etc.

- If the person collecting the sample does not transport the samples to the laboratory or deliver the sample containers for shipment, the first block for Relinquished By _____, Received By _____ shall be completed in the field.

- The person transporting the samples to the laboratory or delivering them for shipment shall sign the record form as Relinquished By _____.

- If the samples are shipped to the laboratory by commercial carrier, the Chain of Custody form shall be sealed in a watertight container, placed in the shipping container, and the shipping container sealed prior to giving it to the carrier.

- If the samples are directly transported to the laboratory, the Chain of Custody form shall be kept in possession of the person delivering the

samples.

- For samples shipped by commercial carrier, the waybill shall serve as an extension of the Chain of Custody record between the final field custodian and receipt in the laboratory.

- Upon receipt in the laboratory, the Sample Custodian, or representative, shall open the shipping containers, compare the contents with the Chain of Custody form, and sign and date the record. Any discrepancies shall be noted on the Chain of Custody form.

- If discrepancies occur, the samples in question shall be segregated from normal sample storage and the field personnel immediately notified.

- The Chain of Custody form is completed after sample disposal. Note the blocks for this purpose on Figure 5-4.

- Chain of Custody records shall be maintained with the records for a specific project, becoming part of the data package.

Multipart Chain of Custody forms may be used so that a copy (yellow) remains with the person shipping the samples.

5.3 LABORATORY SAMPLE RECEIPT

The first step in the laboratory receipt of samples is notification of the Operations Manager or Group Leaders of the incoming samples.

Upon sample receipt, the Sample Custodian, or representative shall:

- Note whether the samples were shipped as hazardous materials; if so, the sample container should be opened in a hood, the Custodian should wear gloves and a respirator in case of improper packing or breakage.

- Examine the shipping containers to verify that the custody tape is intact. If not, the sample shipper is notified. This nonconformance is

documented, together with corrective actions and notification of field personnel, and Lab and Project Management. Figure 5-5 shows examples of custody seals.

- Examine all samples and determine if proper temperature has been maintained during shipment. Document temperatures on the Chain of Custody forms. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. Any samples affected shall also be considered damaged. It will be noted on the Chain of Custody form that specific samples were damaged and that the samples were removed from the sampling program. Field personnel will be notified as soon as possible that samples were damaged and that they must be resampled, or the testing program changed, and an estimate of the cause of damage.

- Compare samples received against those listed on the Chain of Custody form.

- Verify that sample holding times have not been exceeded.

- Sign and date the Chain of Custody form and attach the waybill.

- Attach appropriate laboratory sample container labels with test and preservative information (Figure 5-6).

- Place the samples in adequate laboratory storage.

- Enter the samples in the laboratory sample log-in book, and/or the sample management computer system which contains the following information:

  - Project identification number
  - Sample numbers
  - Type of samples
  - Date received in laboratory
  - Date put into storage after analysis is completed
  - Date of disposal.

The last two items will be added to the log when the action is taken.

- Notify the Operations Manager or Group Leaders of sample arrival.

- Place the completed Chain of Custody records in the project file.

If samples collected by IT personnel arrive without Chain of Custody forms or with incorrect Chain of Custody records or broken custody seals, the following shall be done by the Sample Receiving Clerk:

- If the Chain of Custody form is incorrect or a custody seal is broken, a telephone call is made to the Project Manager and field personnel, stating the nonconformance reporting and correction. A record of the call memorandum must be signed and dated by the Sample Custodian and the QC Coordinator. This will serve as an amendment to the Chain of Custody for the project file. If the information on the Chain of Custody form cannot be corrected by the Sample Receiving Clerk, the samples affected shall be removed from the sampling program.

- If the Chain of Custody form is not shipped with the samples, the field personnel shall be called by the Sample Receiving Clerk and a memorandum prepared by field personnel which lists the persons involved in collecting, shipping, and receiving the samples and the times, dates, and events. Each person involved must sign and date this memorandum. The completed memorandum will be maintained in lieu of the Chain of Custody.

5.4 LABORATORY STORAGE OF SAMPLES

The primary considerations for sample storage are:

- Maintenance at prescribed temperature, if required, which is typically four degrees Centigrade.

- Extracting and/or analyzing samples within the prescribed holding time for the parameters of interest.

The requirements of Table 5-1 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of Sample Receiving personnel who should notify the Operations Manager, or Group Leaders, if there are any samples which must be analyzed immediately because of holding-time requirements.

5.5 INITIATION OF TESTING PROGRAM

As stated in Section 5.1, a Request for Analysis form shall be submitted with the samples to the laboratory. If the analytical program is not defined with the sample shipment, the Sample Receiving Clerk shall immediately notify the Operations Manager or designee for definition of the analysis program; if the samples were collected by IT personnel, the Project Manager and field personnel are notified. If the samples are external to IT Corporation, the Operations Manager or designee shall contact the client to determine the testing program.

The analytical program shall be entered in the laboratory Master Analytical Logbook and/or computerized information manangement system which includes the following information; project number and name; project contact name; date received; received by; submitted by; report to; requested report date; sample identifications; sample parameters; comments; special instructions.

The Operations Manager and Group Leaders are responsible for prioritizing samples on the basis of holding time and required reporting time into the laboratory sample stream.

5.6 SAMPLE DISPOSAL

The chain of custody for the sample is completed as part of sample disposal; these may take one form of a list of project samples signed upon disposition. There are several possibilities for sample disposition:

- The sample may be completely consumed during analysis.
- Samples may be returned to the client or location of sampling for disposal.
- The samples may be stored after analysis. Proper environmental control and holding time must be observed if reanalysis is anticipated. If reanalysis is not anticipated, environmental conditions for storage will not be observed.In general, ITAS will not maintain samples and extracts longer than sixty days beyond completion of analysis, unless otherwise specified.
- Disposal by the laboratory.

The Operations Manager shall determine disposition of samples if not specified in the Request for Analysis, Figure 5-3. Disposition must meet IT Corporation policies which are outlined below.

When samples are removed from storage for disposal, they become classified as hazardous wastes. Laboratory wastes include several types of material: precisely known substances from quality control analyses; unused portions of samples that may or not have been completely analyzed; and solvents, filter media, contaminated protective clothing, etc. used in the analytical process. Certain quantitites of some laboratory wastes must be identified as hazardous because of their general characteristics (ignitability, corrosivity, reactivity, or EP toxicity) or because they are specifically listed in 40 CFR Part 261 due to acute or toxic hazards. Some laboratories composite compatible waste and conduct more thorough analyses of the larger composited quantity to more completely identify their wastes. Certain laboratory waste may be disposed of in a sanitary sewer pursuant to 40 CFR 261.3(a)(2)(iv), for laboratories that connect to a facility wastewater treatment or pretreatment system. Careful review of local, state, and federal wastewater treatment regulations, and a knowledge of the material being disposed of, is required before this disposal option is taken.
A container should be chosen for the wastes in which the wastes can be

ultimately shipped, so it will not be necessary to transfer hazardous materials from one container to another. The laboratory must provide at least enough information about container contents (an inventory listing is appropriate) to properly manifest hazardous waste shipments. Federal Regulations of the Department of Transportation (49 CFR Transportation) apply to the packaging and labeling and shipment of these materials. All labels and markers must meet DOT specifications in accordance with 49 CFR 172.300 and 172.400. Shipping papers (which may be a shipping order, a bill of lading, a manifest, or other document) must contain the information required by Section 172.202, 173.203, 172.204 of the regulation. A copy of the hazardous waste manifest, identifying all wastes, must accompany each shipment of waste from pick-up to destination. Accompanying the manifest should be a contents listing describing each container's contents.

All operations should be performed in a safe manner in accordance with applicable federal, state, local, and IT requirements. Personnel who handle and pack hazardous waste should be trained regarding chemical incompatibility, spills, first aid procedures and use of personal protective equipment. Training records on current personnel must be kept until closure of the facility and training records on former employees must be kept for at least 3 years from the date the employee last worked there.

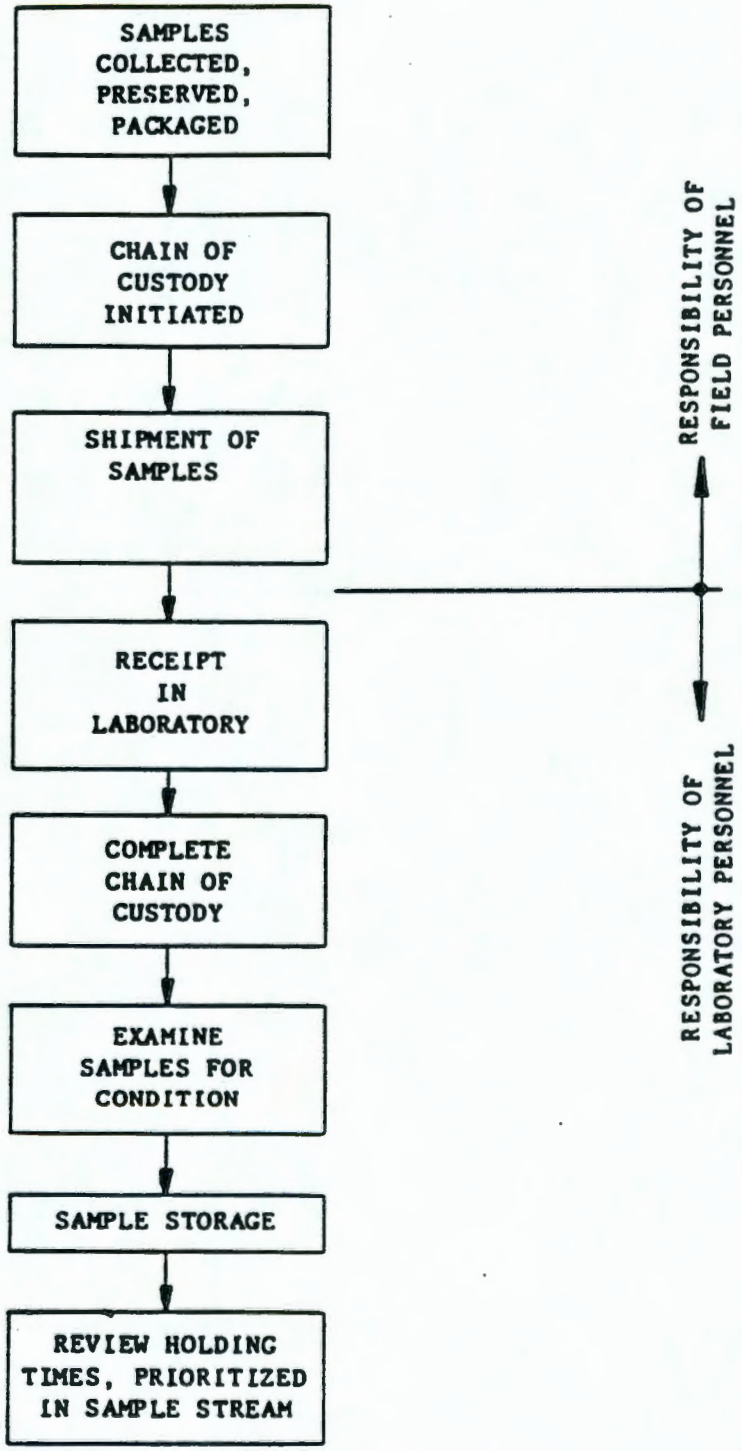

FIGURE 5-1
SAMPLE COLLECTION, TRANSPORT, AND HOLDING FLOW CHART

IT CORPORATION

Project Name _____

Project No. _____

Sample No. _____

Collection Date/Time _____

Collector's Name _____

Sample Location _____

Sample Type/Depth/Description _____

_____ Preservative _____

Analyze For _____

Bottle _____ of _____ _____ Filtered _____ Nonfiltered

FIGURE 5-2

EXAMPLE - SAMPLE FIELD LABEL

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INTERNATIONAL TECHNOLOGY CORPORATION

REQUEST FOR ANALYSIS

R/A Control No.
C/C Control No. _____

PROJECT NAME _____
PROJECT NUMBER _____
PROJECT MANAGER _____
BILL TO _____

PURCHASE ORDER NO. _____

DATE SAMPLES SHIPPED _____
LAB DESTINATION _____
LABORATORY CONTACT _____
SEND LAB REPORT TO _____

DATE REPORT REQUIRED _____
PROJECT CONTACT _____
PROJECT CONTACT PHONE NO. _____

Sample No.	Sample Type	Sample Volume	Preservative	Requested Testing Program	Special Instructions

TURNAROUND TIME REQUIRED: (Rush must be approved by the Project Manager.)

Normal _____ Rush _____ (Subject to rush surcharge)

POSSIBLE HAZARD IDENTIFICATION: (Please indicate if sample(s) are hazardous materials and/or suspected to contain high levels of hazardous substances)

Nonhazard _____ Flammable _____ Skin Irritant _____ Highly Toxic _____ Other _____ (Please Specify)

SAMPLE DISPOSAL (Please indicate disposition of sample following analysis. Lab will charge for packing, shipping, and disposal.)

Return to Client _____ Disposal by Lab _____

FOR LAB USE ONLY

Received By _____ Date/Time _____

WHITE - Original to accompany samples

FIGURE 5-3 REQUEST FOR ANALYSIS FORM

INTERNATIONAL TECHNOLOGY CORPORATION

CHAIN-OF-CUSTODY RECORD

R/A Control No. _____

C/C Control No. _____

PROJECT NAME/NUMBER _____ LAB DESTINATION _____

SAMPLE TEAM MEMBERS _____ CARRIER/WAYBILL NO. _____

Sample Number	Sample Location and Description	Date and Time Collected	Sample Type	Container Type	Condition on Receipt (Name and Date)	Disposal Record No

Special Instructions _____

Possible Sample Hazards: _____

SIGNATURES (Name, Company, Date and Time)

1 Relinquished By: _____ 3. Relinquished By: _____

Received By: _____ Received by: _____

2 Relinquished By: _____ 4. Relinquished By: _____

Received By _____ Received By: _____

FIGURE 5-4

CHAIN OF CUSTODY RECORD FORM

WHITE - To accompany samples
YELLOW - Field copy

SAMPLES
INTERNATIONAL TECHNOLOGY ANALYTICAL SERVICES

INTERNATIONAL TECHNOLOGY CORPORATION	LABORATORY SAMPLES Do Not Tamper — Date:
	Signature:
	Name & Title (PRINT):

FIGURE 5-5

EXAMPLE - CUSTODY SEALS

9112364092

International Technology Corp.
165 Fieldcrest Ave.
Edison, New Jersey 08818-7809

CLIENT	WORK-ORDER	FRAC	DATE-RECIVED
	E702144	02AS	02/27/87

CLIENT NAME:
SAMPLE ID: A1-0-E"
DATE/TIME COLLECTED: 02/26/87
Old Lot # 3194

TESTS:

3550X	60SAB	624	625	625A	AS
AS	BE	CD	CNT_R	CNT_S	CR
CU	DSST_S	DSS_AS	DSS_CU	HG	NI
PB	PL_R	PL_S	SB	SE	TL
TS	TH				

No Preservative [] White
Sulfuric Acid [] Yellow
Nitric Acid [] Red
Phosphoric Acid [] Dark Blue
Sodium Hydroxide [] Green
Zinc Acetate [] Black
Sodium Thiosulfate [] Light Blue
Acetone Rinsed [] Brown
Other [] Orange

COLOR KEY

JOB# LOT# SAMPLE DATE SAMPLE# BOTTLE#

PROJECT NAME:

SAMPLE ID:

No Preservative	☐	White
Sulfuric Acid	☐	Yellow
Nitric Acid	☐	Red
Phosphoric Acid	☐	Dark Blue
Sodium Hydroxide	☐	Green
Zinc Acetate	☐	Black
Sodium Thiosulfate	☐	Light Blue
Acetone Rinsed	☐	Brown
Other	☐	Orange

COLOR KEY	SAMPLE NUMBER

FIGURE 5-6

EXAMPLE - LABORATORY SAMPLE CONTAINER LABELS

9 1 1 2 3 6 4 0 0 9 3

TABLE 5.1
SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
Bacterial Tests				
o Coliform, fecal and total	P,G	200	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	6 hours
o Fecal streptococci	P,G	200	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	6 hours
Classical Wet Chemistry Tests				
o Acidity	P,G	50	Cool 4°C	14 days
o Alkalinity	P,G	50	Cool 4°C	14 days
o Ammonia	P,G	100	Cool 4°C, H_2SO_4 to pH <2	28 days
o Biochemical Oxygen Demand	P,G	1,000	Cool 4°C	48 hours
o Biochemical Oxygen Demand (carbonaceous)	P,G	1,000	Cool 4°C	48 hours
o Bromide	P,G	200	None required	28 days
o Chemical Oxygen Demand	P,G	75	Cool 4°C, H_2SO_4 to pH <2	28 days
o Chloride	P,G	50	None required	28 days
o Chlorine, Total Residual	P,G	200	None required	Analyze immediately
o Color	P,G	50	Cool 4°C	48 hours
o Cyanide, Total and Amenable to Chlorination	P,G	1,500	Cool 4°C, NaOH to pH ≥12, 0.6g ascorbic acid[d]	14 days[e]
o Fluoride	P	300	None required	28 days
o Hardness	P,G	100	HNO_3 to pH <2, H_2SO_4 to pH <2	6 months

MAN:sampling

See footnotes at end of table.

TABLE 5.1

(Continued)

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
o Hydrogen Ion (pH)	P,G	25	None required	Analyze immediately
o Kjeldahl and Organic Nitrogen	P,G	500	Cool 4°C, H_2SO_4 to pH <2	28 days
Metals[f]				
o Chromium VI	P,G	50	Cool 4°C	24 hours
o Mercury	P,G	100	HNO_3 to pH <2	28 days
o Metals, Except Chromium VI and Mercury	P,G	200	HNO_3 to pH <2	6 months
o Nitrate	P,G	100	Cool 4°C	48 hours
o Nitrate-Nitrite	P,G	100	Cool 4°C, H_2SO_4 to pH <2	28 days
o Nitrite	P,G	50	Cool 4°C	48 hours
o Oil and Grease	G	1,000	Cool 4°C, H_2SO_4 to pH <2	28 days
o Orthophosphate	P,G	50	filter immediately, cool 4°C	48 hours
o Oxygen, Dissolved Probe	G bottle and top	300	None required	Analyze immediately
o Phenols	G	500	Cool 4°C, H_2SO_4 to pH <2	28 days
o Phosphorus (Elemental)	G	50	Cool 4°C	48 hours
o Phosphorus, Total	P,G	50	Cool 4°C, H_2SO_4 to pH <2	28 days
o Residue, Total	P,G	100	Cool 4°C	7 days
o Residue, Filterable	P,G	100	Cool 4°C	7 days
o Residue, Nonfilterable	P,G	250	Cool 4°C	7 days

MAN:sampling See footnotes at end of table.

TABLE 5.1

(Continued)

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
o Residue, Settleable	P,G	1,000	Cool 4°C	48 hours
o Residue, Volatile	P,G	100	Cool 4°C	7 days
o Silica	P	50	Cool 4°C	28 days
o Specific Conductance	P,G	100	Cool 4°C	28 days
o Sulfate	P,G	100	Cool 4°C	28 days
o Sulfide	P,G	500	Cool 4°C, add zinc acetate plus sodium hydroxide to pH >9	7 days
o Sulfite	P,G	50	None required	Analyze immediately
o Surfactants	P,G	250	Cool 4°C	48 hours
o Temperature	P,G	1,000	None required	Analyze immediately
o Turbidity	P,G	100	Cool 4°C	48 hours
<u>Organic Tests</u>[g]				
o Organic Carbon	P,G	25	Cool 4°C, HCl or H_2SO_4 to pH <2	28 days
o Organic Carbon (TOC)	G, Amber bottle Teflon-Lined Cap	4 X 15	Cool 4°C, 1 ml of 1.1 M $Na_2S_2O_3$	28 days
o Purgeable Halocarbons	G,Teflon-lined septum	2 X 40	Cool 4°C, 0.008% $Na_2S_2O_3$[d] HCl pH 2[h]	14 days

MAN:sampling

See footnotes at end of table.

TABLE 5.1

(Continued)

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
o Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	2 X 40	Cool 4°C, 0.008% $Na_2S_2O_3$[d], HCl to pH 2[h]	14 days
o Acrolein and Acrylonitrile	G, Teflon-lined septum	2 X 40	Cool 4°C, 0.008% $Na_2S_2O_3$[d], adjust pH to 4-5[i]	14 days
o Phenols[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	7 days until extraction, 40 days after extraction
o Benzidines[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	7 days until extraction[l] 40 days after extraction
o Phthalate Esters[j]	G, Teflon-lined cap	1,000	Cool 4°C	7 days until extraction, 40 days after extraction
o Nitrosamines[j,m]	G, Teflon-lined cap	1,000	Cool 4°C, store in dark, 0.008% $Na_2S_2O_3$[d]	7 days until extraction, 40 days after extraction
o PCBs[j]	G, Teflon-lined cap	1,000	Cool 4°C	7 days until extraction, 40 days after extraction
o Nitroaromatics and isophorone[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d], store in dark	7 days until extraction, 40 days after extraction
o Polynuclear Aromatic Hydrocarbons[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d], store in dark	7 days until extraction, 40 days after extraction
o Haloethers[j]	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	7 days until extraction, 40 days after extraction

MAN:sampling

See footnotes at end of table.

TABLE 5.1
(Continued)

PARAMETER	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
o Chlorinated Hydrocarbons[j]	G, Teflon-lined cap	1,000	Cool 4°C	7 days until extraction, 40 days after extraction
o TCDD[j]---------- Tetrachlorodibenzo-p-dioxin	G, Teflon-lined cap	1,000	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	7 days until extraction, 40 days after extraction
o Total Organic Halogens	G, Teflon-lined Cap, Amber Bottle	4 X 15	Cool 4°C, H_2SO_4 to pH ≤ 2	14 days
Pesticides				
o Pesticides	G, Teflon-lined cap	1,000	Cool 4°C, pH 5-9[n]	7 days until extraction, 40 days after extraction
Radiological Tests				
o Alpha, Beta, and Radium	P,G	1,000	HNO_3 to pH <2	6 months

MAN:sampling

See footnotes at end of table.

TABLE 5.1

(Continued)

Reference: This table includes the requirements of the U.S. Environmental Protection Agency, as published in the Code of Federal Regulations, Vol. 49, No. 209, 40 CFR 136, October 26, 1984, pg, 43260.

(a) Polyethylene (P) or glass (G).

(b) 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.

(c) Samples should be analyzed as soon as possible after collection. The times listed are maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time. 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 if knowledge exists to show this is necessary to maintain sample stability.

(d) Should only be used in the presence of residual chlorine.

(e) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustment to determine if sulfide is present.

TABLE 5.1

(Continued)

(f) Filtration, acid preservation, etc., are to be performed at the time the sample is collected or as soon as possible thereafter. For the determination of dissolved elements the sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. Acidify the filtrate with (1+1)HNO_3 to a pH of 2 or less. For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required. For the determination of total or total recoverable elements, the sample is acidified with (1+1)HNO_3 to pH 2 or less as soon as possible, preferably at the time of collection. The sample is not filtered before processing.

(g) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

(h) Aqueous sample receiving no pH adjustment must be analyzed within seven days of sampling. Soils, sediments and sludge samples do not require acidification.

(i) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.

(j) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard 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 six to nine; samples preserved in this manner may be held for seven days before extraction and 40 days after extraction. Exceptions to the optional preservation and holding time procedure are noted in footnote (d) (re the requirement for thiosulfate reduction of residual chlorine) and footnotes (k) and (l) (re the analysis of benzidine).

TABLE 5.1

(Continued)

(k) If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

(l) Extracts may be stored up to seven days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

(m) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to seven to ten with NaOH within 24 hours of sampling.

(n) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extacted within 72 hours of collection. For the analysis of aldrin add 0.008% $Na_2S_2O_3$.

6.0 CALIBRATION PRACTICES

Instruments and equipment used in an ITAS laboratory are controlled by a formal calibration program. The program verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. All instruments and equipment which measure a quantity, or whose performance is expected at a stated level, are subject to calibration. Calibration may be performed by ITAS personnel using reference standards, or externally by calibration agencies or equipment manufacturers.

This section of the QA Manual prescribes the practices used by an ITAS laboratory to implement a calibration program. Specifics are not provided herein because the requirements for the calibration of instruments and equipment are dependent upon the type and expected performance of individual instruments and equipment. Therefore, an ITAS laboratory shall use the guidelines provided herein to develop a calibration program as part of its Laboratory-Specific Attachment to the Quality Assurance Manual. Development and documentation of the laboratory calibration program is the responsibility of the Technical Director. Implementation is the responsibility of the Group Leaders and Analysts. The Quality Control Coordinator shall review the implementation of the program, as discussed in Section 14.0.

Two types of calibration are discussed in this section:

- Operational calibration which is routinely performed as part of instrument usage, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operational calibration is generally performed for instrument systems.

- Periodic calibration which is performed at prescribed intervals for equipment, such as balances and ovens. In general, equipment which can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance.

6.1 CALIBRATION SYSTEM

Following is a discussion of the elements comprising the calibration system.

6.1.1 Calibration Procedures

Written procedures shall be developed by an ITAS laboratory within the requirements of this manual for all instruments and equipment subject to calibration. Whenever possible, recognized procedures, such as those published by ASTM or the USEPA, or procedures provided by manufacturers shall be adopted. If established procedures are not available, a procedure shall be developed considering the type of equipment, stability characteristics of the equipment, required accuracy, and the effect of operational error on the quantities measured. As a minimum, the procedures shall include:

- Equipment to be calibrated
- Reference standards used for calibration
- Calibration technique and sequential actions
- Acceptable performance tolerances
- Frequency of calibration
- Calibration documentation format.

6.1.2 Equipment Identification

Equipment that is subject to calibration shall be uniquely identified so that calibration records can be designated with a specific instrument. Equipment identification shall be by manufacturer's serial number, IT inventory control number, or a unique number assigned by an ITAS laboratory.

6.1.3 Calibration Frequency

Instruments and equipment shall be calibrated at prescribed intervals and/or as part of the operational use of the equipment. Frequency shall be based on the type of equipment, inherent stability, manufacturer's recommendations, values provided in recognized standards, intended data use, specified analytical methods, effect of error upon the measurement process, and prior experience.

6.1.4 Calibration Reference Standards

Two types of reference standards are used within ITAS laboratories for calibration:

- Physical standards, such as weights for calibrating balances and certified thermometers for calibrating working thermometers and ovens, which are generally used for periodic calibration

- Chemical standards such as Standard Reference Materials (SRMs) provided by the National Bureau of Standards (NBS) or the United States Environmental Protection Agency (USEPA). These may include vendor certified materials traceable to NBS or EPA SRMs. These are primarily used for operational calibration.

Whenever possible, physical reference standards shall have known relationships to nationally recognized standards (e.g., NBS) or accepted values of natural physical constants. If national standards do not exist, the basis for the reference standard shall be documented.

Physical reference standards shall be used only for calibration and shall be stored separately from equipment used in analyses. In general, physical reference standards shall be at least four to ten times as accurate as the requirements for the equipment which they are used to calibrate. In general, physical standards should be recalibrated every three years by a certified external agency.

Whenever possible, chemical reference standards shall be directly traceable to NBS SRMs. If SRMs are not available, compounds of vender-certified high purity will be used to prepare calibration standards.

6.1.5 Calibration Failure

Equipment that fails calibration or becomes inoperable during use shall be removed from service and segregated to prevent inadvertent use, or shall be tagged to indicate it is out of calibration. Such equipment shall be repaired and satisfactorily recalibrated before reuse. For equipment that fails

calibration, a record such as shown on Figure 6-1 shall be prepared and maintained with the equipment calibration file.

Scheduled calibration of equipment does not relieve the laboratory staff of the responsibility for using properly functioning equipment. If an equipment malfunction is suspected, the equipment shall be tagged and removed from service and recalibrated. If it fails recalibration, the above process shall apply.

6.1.6 Calibration Records

Records shall be prepared and maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of preparation, stability, and proof of continuity of reference standards shall also be maintained.

Records for periodically calibrated equipment shall include, as appropriate:

- Identification number of equipment and type of equipment, or assigned unique equipment number
- Calibration frequency and acceptable tolerances
- Identification of calibration procedure used
- Date calibration was performed
- Identity of ITAS personnel and/or external agencies performing calibration
- Reference standards used for calibration
- Calibration data
- Certificates or statements of calibration provided by manufacturers and external agencies, and traceability to national standards
- Information regarding calibration acceptance or failure and any repair of failed equipment.

Records for periodically calibrated equipment shall be maintained in the Laboratory Operations Records as discussed in Section 12.0. Records for each instrument/equipment and physical reference standard shall be kept in a separate folder. The title sheet for each file shall be a summary of calibrations performed, such as shown in Figure 6-2. It is recommended that an index precede the equipment files which lists in matrix form all equipment and physical standards, calibration frequency, and dates for upcoming calibration. The use of a calibration due date matrix provides ready reference so that calibration can be maintained by the Group Leaders.

For instruments and equipment that are calibrated on an operational basis, calibration generally consists of determining instrumental response against compounds of known composition and concentration or the preparation of a standard response curve of the same compound at different concentrations. Records of these calibrations can be maintained in several ways:

- The calibration data can be kept with analytical sample data
- A log book can be prepared for each instrument which contains all calibration data.

The former method provides response factor information, etc., directly with analytical data so that the analytical data can be readily processed and verified. Also, the raw data package is complete as a unit. However, if samples from several projects are processed together, the calibration data must be copied and included with each group of data.

The latter method provides an ongoing record of the calibration undertaken for a specific instrument; however, to process and verify the analytical data the log must be used in conjunction with the raw data.

For operational calibration, the following is recommended:

- Calibration data be included with the raw analytical data. If samples from different projects are processed together, calibration data should be copied and included with each group of samples.

- A file is maintained for each instrument in the Laboratory Operating Records which includes:

  - Calibration instructions (curve preparation, linear range, etc.)

  - Procedures for chemical standards preparation

  - A bound calibration log book prepared by the Analyst which contains:

    a. Notice of calibration failure and repairs

    b. Midpoint standard response (as discussed in Section 8.0)

    c. Brief notice of calibrations performed.

The log book should be indexed in the Laboratory Operations Records as discussed in Section 12.0, but should be maintained at the instrument by the Analyst. All entries should be signed and dated by the Analyst.

6.2 OPERATIONAL CALIBRATION

Operational calibration is generally performed as part of the analytical procedure. Included may be the analysis of a method blank and the preparation of a standard response (standard calibration) curve. Operational calibration is dependent upon the instrumentation within an ITAS laboratory, and as previously discussed, each ITAS laboratory shall prepare a Laboratory-Specific Attachment to this manual which includes the Laboratory program for calibration.

Following is a brief discussion of the analysis of method blanks and preparation of standard curves. Guidelines for the major instrument systems within ITAS laboratories follow. (See also Table 6-1).

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6.2.1 General Calibration Procedures

The initial phase of a laboratory testing program requires the selection and certification of the method best suited for an individual parameter. Certification, or verification, is the elimination, or minimizing, of determinate errors which may be due to Analyst error, the use of less-than-optimum equipment, reagents, solvents, or gases. The quality of materials, even though they are AR grade or better, may vary from one source to another. The Analyst must determine, through the use of reagent and/or solvent blanks, if materials are free from interfering substances which could affect the analysis. Other steps in certifying the method include the determination of a method blank and the preparation of a standard calibration curve.

6.2.1.1 Method Blank

After determining the individual reagent or solvent blanks, the Analyst defines the method blank to determine if the cumulative blank interferes with the analysis. The method blank is prepared by following the procedure step by step, including the addition of all of the reagents and solvents, in the quantity required by the method. If this cumulative blank interferes with the determination, steps must be taken to eliminate or reduce the interference to a level that will permit the combination of solvents and reagents to be used. If the blank interference cannot be eliminated, the magnitude of the interference must be considered when calculating the concentration of specific constituents in the samples analyzed.

A method blank should be determined whenever an analysis is made. The number of blanks is determined by the method of analysis and the number of samples analyzed at a given time. (See also Section 8.1.4.)

6.2.1.2 Preparation of Standard Calibration Curve

Concurrent with the preparation of reagent and method blanks, a standard calibration curve is prepared for the instrumentation. Preparation of a

standard calibration curve is accomplished by using calibration standards. The process is summarized as:

- Preparation of a standard calibration curve is accomplished by using calibration standards prepared by mixing the species to be analyzed into the solvent that is to be introduced into the instrument.
- The concentrations of the calibration standards are chosen to cover the working range of the instrument.
- All sample measurements are made within this working range.
- The calibration curve is prepared by plotting instrument response versus concentration of the species analyzed.
- Concentrations of the sample prepared with the same procedure are read directly from the calibration curve or determined by interpolation.

6.2.2 Instrument Calibration Procedures

This section outlines the minimum operations necessary to satisfy analytical requirements associated with the determination of specific parameters in water, soil/sediment, and hazardous waste samples. Table 6-1 presents summary calibration requirements for analytical instrumentation. Specific methods may have slightly different requirements. Details of calibration are described in Laboratory-Specific Manuals or in Standard Operating Procedures.

6.3 PERIODIC CALIBRATION

Periodic calibration shall be performed for equipment such as balances, thermometers, ovens, and furnaces that are required in analytical methods, but which are not routinely calibrated as part of the analytical procedure. Documentation of calibration shall be kept for each equipment item as discussed in Section 6.1.6.

Calibration requirements are determined within each ITAS laboratory

depending upon the equipment used and its operating function. Following are brief example discussions for the calibration of balances and thermometers with examples of calibration data sheets to serve as a guideline for the preparation of laboratory-specific procedures. (See also Table 6-2).

6.3.1 Balances (Example Procedure)

All balances shall be calibrated at least every three months using weights traceable to the National Bureau of Standards (NBS). Calibration weights shall be Class S or better and shall be recertified every three years. If balances are calibrated by an external agency, verification of their weights shall be provided.

Calibration of balances shall be to approximately 95 percent of balance capacity. Testing intervals over the operational range of the balance shall be approximately 5, 10, 15, 20, 25, 30, 40, 50, 60, 70, 80, 90, and 95 percent of capacity. Acceptance for balances which are direct reading to 0.01 gram shall be ±0.01 g for 0 to 100 g and ±0.1 percent of the applied weight over 100 g.

Figure 6-3 provides an example data sheet that can be used for balance calibration. Figure 6-4 provides a data form which can be used for calibration weight certification.

6.3.2 Thermometers (Example Procedure)

Certified, or reference, thermometers shall be maintained for use in calibrating working thermometers. Reference thermometers shall be provided with NBS traceability for initial calibration and shall be recertified every three years with equipment directly traceable to the NBS.

Working thermometers shall be compared with the reference thermometers every 12 months. In addition, working thermometers shall be visually

inspected by laboratory personnel prior to use.

Calibration temperatures and acceptance criteria shall be based upon the working range of the thermometer and the accuracy required for its use.

Figure 6-5 shows an example of a calibration record sheet for thermometer calibration.

IT ANALYTICAL SERVICES

NOTICE OF EQUIPMENT
CALIBRATION FAILURE

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

REASON FOR FAILURE TO PASS CALIBRATION _____

_____

_____

WAS EQUIPMENT REPAIRED OR REPLACED? _____

_____

IF REPAIRED, WAS THE CORRECTIVE ACTION ADEQUATE? _____

_____

_____

_____

_____

SIGNED _____

DATE _____

FIGURE 6-1
EXAMPLE CALIBRATION FAILURE NOTICE

IT ANALYTICAL SERVICES

LABORATORY EQUIPMENT CALIBRATION RECORD

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

_____

REQUIRED CALIBRATION PERIOD _____

DATE CALIBRATION PERFORMED	CALIBRATION PERFORMED BY	DATE CALIBRATION PERFORMED	CALIBRATION PERFORMED BY

FIGURE 6-2
EXAMPLE EQUIPMENT CALIBRATION RECORD

9 1 1 2 3 6 4 0 1 1 3

BALANCE CALIBRATION

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

DATE _____ DATE LAST CALIBRATED _____

CALIBRATION PERIOD _____

WEIGHTS APPLIED TO BALANCE	BALANCE READING	DOES BALANCE MEET STANDARD	WEIGHTS APPLIED TO BALANCE	BALANCE READING	DOES BALANCE MEET STANDARD

Signed _____

FIGURE 6-3

EXAMPLE - BALANCE CALIBRATION RECORD

9112160114

WEIGHT CALIBRATION BY EXTERNAL AGENCY*

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

WEIGHT CLASS _____

NAME OF EXTERNAL AGENCY PERFORMING CALIBRATION _____

_____

DATE _____

PROVIDE IN THE TABLE BELOW THE LABELED WEIGHTS VERSUS THE CERTIFIED WEIGHT DETERMINATION. ALL CERTIFICATION RECORDS ARE TO BE ATTACHED TO THIS FORM.

WEIGHT AS LABELED	WEIGHT AS CERTIFIED	DOES WEIGHT MEET CLASS STANDARD ?	WEIGHT AS LABELED	WEIGHT AS CERTIFIED	DOES WEIGHT MEET CLASS STANDARD ?

* WEIGHT CALIBRATION IS TO BE PERFORMED EVERY THREE YEARS

FIGURE 6-4

EXAMPLE - WEIGHT CERTIFICATION RECORD

9112260115

THERMOMETER CALIBRATION

EQUIPMENT NUMBER _____

EQUIPMENT NAME _____

DATE _____ DATE LAST CALIBRATED _____

CALIBRATION PERIOD _____

TEMPERATURE ACCORDING TO REFERENCE	THERMOMETER BEING CALIBRATED

WORKING RANGE _____

ACCEPTANCE CRITERIA _____ ± _____ °C

Signed _____

FIGURE 6-5

EXAMPLE - THERMOMETER CALIBRATION RECORD

9112756101116

Table 6.1. Summary of Operational Calibration Requirements

Instrument	Calibration Standards Used, Initial & Daily Minimum	Acceptance Limits	Corrective Actions	Reference[1]
Atomic Absorption Spectrophotometer	Initial: 3 levels + blank Daily: 1 check standard (midrange) per 10 samples;	Linear regression correlation coefficient >0.995; ±5% of true value	Make new standards or establish new calibration curve	1,2
Gas Chromatograph	Initial: 5 levels + blank Daily: 1 level of check standards standards	Calibration factor % RSD < 10%; ±10% of original curve	Make new standards, or establish new calibration curve	1
Inductively Coupled Plasma Emission Spectrophotometer	Initial: Manufacturer's instructions; Daily: Instrument check standard and calibration blank every 10 samples Weekly: QC sample, EPA/NBS source	Maximum response <0.006 nm of reference line, ±5% of original curve; blank within 2 standard deviations of limits. ±5% of true value	Establish new curve " Repeat twice; if average is outside control limit, then: Make new standards, and recalibrate	3 3 3
Ion Chromatograph	Initial: 3 levels + blank Daily: 1 level of check standards every 20 samples (midrange)	±10% of original curve	Make new standards recalibrate	4
pH Meter	Daily: 3 levels	±0.05 pH unit	Clean or replace electrode; recalibrate	1
UV-Visible Spectrophotometer	Initial: 3 levels + blank Daily: Check standard Quarterly: Wavelength accuracy and photometric linearity	±10% of original curve Manufacturer specifications ±10% previous observations	Recalibrate Service Service	

MAN:PM-t-6-1

Table 6.1. (Continued)

Instrument	Calibration Standards Used, Initial & Daily Minimum	Acceptance Limits	Corrective Actions	Reference[1]
Infrared Spectrophotometer	Daily: Wavelength calibration and calibration curve, 6 levels + blank	±5 cm^{-1} true (Sadtler Index)	Service Make new standards, and recalibrate	5
Total Organic Carbon	Daily: 3 levels + blank	±10% of original curve	Make new standards	1
GC/MS	Initial: 5 levels + blank Daily: 1 level	%RSD <30% ±5% of initial curve	Make new standard recalibrations	1
Total Organic Halogen	Daily: 3 levels + blank	±10% of original curve	Make new standards	1

References:
1) EPA SW-846, "Test Methods for Evaluating Solid Waste"
2) USEPA Methods for Chemical Analysis of Water and Wastes, 1979
3) USEPA 200.7,1982
4) USEPA 300.0, 1984
5) USEPA "Procedure for the Evaluation of Environmental Monitoring Laboratories", 1978

Table 6.2. Summary of Periodic Calibration Requirements

Instrument	Calibration Frequency	Acceptance Limit	Corrective Actions
Analytical Balance	Daily: Sensitivity (with a Class "S" weight)	0.001 gm	Adjust, sensitivity
	Monthly: Reproducibility	Mean, Std. Dev. less than 0.1 mg	Service balance
	Monthly: Consistency	Difference less than 0.1 mg	Replace weights
	Quarterly: Class "S" weights Check	Difference less than 0.1 mg	Service balance
Autoclaves and Sterilizers	(a) Sterilization effectiveness check daily		Service
	(b) Temperature-recording device calibrated semi-annually		Service
	(c) Automatic timing mechanism checked monthly	±0.5 minutes	Service
Thermometers	Calibrate in constant temperature baths at two temperatures against precision thermometers certified by NBS annually	±0.5°C	Tag and remove from service, replace
Pipettors	Gravimetric check quarterly	High volume (>100 µL): ≤ 1.0% relative error and RSD Low volume (<100 µL): ≤ 2.0% relative error and RSD	Service or replacement

7.0 PREVENTIVE MAINTENANCE

Within ITAS laboratories, preventive maintenance is an organized program of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance and to prevent instruments and equipment from failing during use. An adequate preventive maintenance program increases reliability of a measurement system. A preventive maintenance program considers the following:

- Instruments, equipment, and parts thereof that are subject to wear, deterioration, or other change in operational characteristics without periodic maintenance.
- Spare parts that should be available within the laboratory to minimize downtime.
- Frequency that maintenance is required.

The implementation of a preventive maintenance program is dependent upon the specific instruments and equipment used within an ITAS laboratory; therefore, this manual does not designate specific practices for instruments and equipment. Each ITAS laboratory shall prepare a preventive maintenance program which meets the guidelines presented in this section. The program shall be included in the Laboratory Specific Attachment to this Quality Assurance Manual.

Within a laboratory, the Operations Manager is responsible for preparation and documentation of the program. Group Leaders shall implement the program, and the QC Coordinator shall review implementation to verify compliance.

Each ITAS laboratory preventive maintenance program shall include the following:

- A listing of the instruments and equipment that are included in the program

- The frequency of maintenance considering manufacturer's recommendations and/or previous experience with equipment. The listing and maintenance frequency should be provided on a schedule such as Figure 7-1. Frequency should be stated in terms of monthly, quarterly, etc. following guidance in Table 7-1

- For each instrument in the program provide:

  - A list of spare parts maintained by the laboratory

  - External service contracts

  - Items to be checked and/or serviced during maintenance and directions for performing maintenance (if external service is not provided, or if not stated in manufacturer's instrument manuals).

Preventive maintenance should be documented as discussed below and the records stored in accordance with Section 12.2 of this manual. (See examples Figure 7-3 through 7-9). The master schedule (Figure 7-1) should be kept at the beginning of the maintenance records. Behind the schedule, a separate file should be maintained for each instrument. The instrument file should include:

- Spare parts list

- External service contracts

- Checklist of items to be serviced and directions for maintenance or manufacturer's instrument manuals

- Record/results of periodic maintenance.

The record of maintenance can be documented using Figure 7-2 or equivalent. Comments should note any parts which were replaced, observed deterioration, etc.

PREVENTIVE MAINTENANCE SCHEDULE

INSTRUMENT/EQUIPMENT	SERIAL NUMBER	MAINTENANCE FREQUENCY	MAINTENANCE REQUIRED	TO BE PERFORMED BY

FIGURE 7-1

EXAMPLE - PREVENTIVE MAINTENANCE SCHEDULE

INSTRUMENT _____

SERIAL NUMBER _____

MAINTENANCE FREQUENCY _____

MAINTENANCE PERFORMED BY	DATE	COMMENT

FIGURE 7-2

EXAMPLE - PREVENTIVE MAINTENANCE RECORD

9112356 0123

pH METER/SIE

Maintenance Performed	Date/Initials				
Clean Electrode					
Calibrate / Check Slope					
Calibrate Meter. Electronics					

Clean Electrode Each Use

Check Slope Each Use

Calibrate Meter Electronics Monthly

FIGURE 7-3

EXAMPLE - pH METER/SIE
PREVENTIVE MAINTENANCE RECORD

9 1 1 2 7 4 0 1 2 4

CONDUCTIVITY METER

Maintenance Performed	Date/Initials				
Clean Cell					
Meter Span Check					

Clean Cell Each Use

Meter Span Check Monthly

FIGURE 7-4

EXAMPLE - CONDUCTIVITY METER PREVENTIVE MAINTENANCE RECORD

ATOMIC ABSORPTION SPECTROPHOTOMETER

Maintenance Performed	Date / Initials				
Clean Burner Head					
Empty Waste Receptacle					
Clean Nebulizer/ Burner Chamber					
Optics/Gen. Cleaning					
Gases Checked					
Air Purifier Drained					

Burner Head Cleaned Each Use

Waste Reservoir Emptied as Needed

Nebulizer/Burner Chamber Cleaned Weekly

Optics Cleaned by Contract Personnel Quarterly

Gases Checked Each Change of Tank

Air Purifier Drained Quarterly

FIGURE 7-5

EXAMPLE - ATOMIC ABSORPTION SPECTROPHOTOMETER
PREVENTIVE MAINTENANCE RECORD

9 1 1 2 7 6 4 0 1 2 6

GC PREVENTIVE MAINTENANCE

Maintenance Performed	Date / Initials				
Detector Cleaned					
Hall					
FID					
Septa Changed					
Gases Changed					
Other (Specify) _____ _____ _____ _____					

Detector Cleaned as Needed

Septa Changed as Needed

Gases Changed as Needed

Others as Needed

FIGURE 7-6

EXAMPLE - GC PREVENTIVE MAINTENANCE RECORD

GC/MS PREVENTIVE MAINTENANCE

Maintenance Performed	Date / Initials				
Source Cleaned					
Septa Changed					
Turbo Oil Changed					
Pump Oil Changed					
Gases Changed					
Other (Specify) _____ _____ _____ _____					

Source Cleaned as Needed

Septa Changed as Needed

Turbo Oil Changed Quarterly

Pump Oil Changed Quarterly

Gases Changed as Needed

Others as Needed

FIGURE 7-7

EXAMPLE - GC/MS PREVENTIVE MAINTENANCE RECORD

9 1 1 2 3 6 1 0 1 2 8

SPECTROPHOTOMETER MAINTENANCE FORM

Maintenance Performed	Date / Initials				
Lamp Changed					
Wavelength Checked					
Cell Changed / Cleaned					
Service					

Lamp Changed as Needed

Wavelength Checked Quarterly

Cell Changed/Cleaned as Needed

Service as Needed

FIGURE 7-8

EXAMPLE - SPECTROPHOTOMETER PREVENTIVE MAINTENANCE RECORD

9112340129

TOTAL ORGANIC CARBON ANALYZER

Maintenance Performed	Date / Initials				
Perchlorate Tubes Changed					
Ascarite Changed					
Clean Cell / Detachable Parts					
Insure Gas Quality					

Perchlorate and Copper Tubes - as Needed

Ascarite - as Needed

Cleaning Detachable Parts - Each Use

Gas Quality - Each Tank

FIGURE 7-9
EXAMPLE - TOTAL ORGANIC CARBON ANALYZER
PREVENTIVE MAINTENANCE RECORD

9 1 1 2 7 6 4 0 1 3 0

TABLE 7-1
PREVENTIVE MAINTENANCE GUIDANCE

INSTRUMENT	ITEM CHECKED/SERVICED	FREQUENCY
Atomic Absorption Spectrophotometer	3 point calibration performed	When serviced by repairman
	Burner head	Each shift
	Electrical	Each shift
	Lamps	Each shift
	Nebulizer	Each shift
	Optics	Quarterly
	Pump	Each shift
	Tygon tubing	Each shift
	Graphite tube	As necessary
	Replace graphite tube	As needed, or 80-100 Samples
	Replace contact rings	Quarterly
	Replace quartz windows	Semi annual
	Clean optics	Quarterly
	Align background lamp	Quarterly
	Check wave length	Quarterly
Gas Chromatograph	EC (Ni-63) wipe test	Semi annual
	Replace column packing	< 2 months
	Clean detector	As needed or EC Semi annually; FID monthly
	Change column	< 6 weeks
	Change glass wool plug	Weekly
	Clean insert	Weekly
	Replace septum	Daily
	Gases purity checks	Upon receipt of new cylinders
	Flow controller	Semi annually
	Purge and trap	As needed
	Change fuses	As needed
	Reactivate external carrier gas filler dryers	Weekly
	Reactivate flow controller filter dryers	Semi annually
	Clean and silanize or replace glass liners on injectors	As needed or quarterly
	Clean Detectors a) ECD	As needed
	b) FID/NPD	As needed or annually
	Clean Purge Vessel	As needed or monthly
	Replace Purge Vessel	As needed
	Bake Trap	As needed
	Replace Trap	Semi annually
	Replace carbon filter	Annually

Table 7-1 continued

Inductively Coupled Plasma Spectrophotometer	Sample introduction system	Daily
	Clean optics	Daily
	Clean burner heads	Daily
	Check pumps	Daily
	Check electronics	Daily
	Clean, realign torch	Monthly
	Clean nebulizer	Monthly
	Clean mixing chamber	Monthly
	Check nebulizer press	Monthly
	Replace pump tubing	Monthly
	Clean air filters	Monthly
Ion Chromatograph	Change plunger seals	Six months
	Check plumbing	Daily
	Oil pumps	Weekly (leak check monthly)
	Check filter (inlet)	Weekly
	Change column	Replaced < 2 weeks
	Check bed support	Change when pressure drops
	Change fuses	As needed
	Change pump motor	As needed
	Replace plunger seals	Semi annually
	Clean check valve	Semi annually
	Clean, replace solvent reservoir filter	Semi annually
	Degas pump head	As needed
	Clean, replace cell packing, degas floor cell	As needed or quarterly
Refrigerators	Temperature checked and logged	Daily
Walk-in Coolers	Temperature checked and logged	Daily
Balances	Service representative calibration	Annually
Deionized/Organopure Water	Conductivity check	Daily
	Ion exchange bed changed	Weekly
	Replace filters	As needed
Vacuum Pumps and Air Compressor	Check performance	Weekly
	Lubrication, belts, etc.	As needed
Analytical Balance	Internal Weight Train, Gears, Electronics	Annual Service

Table 7-1 continued

GC/MS	GC/MS maintenance is the same as GC with the following additions:	
	Diffusion pump oil	Bi-Weekly
	Mechanical pump oil	Quarterly
	Power Con. air filter	Bi-Weekly
	QEM filter	Bi-Weekly
	Water bay filter	Bi-Weekly
	Interface Box	Bi-Weekly
	Vacuum chaff filter	Monthly
	Turbo pump oil	Quarterly
	Water filter (if applicable)	Observe and change as needed
	Computer air filter	Monthly
	Card cage air filter	Monthly
	Source-clean ceramics, polish lenses	As needed
	Clean poles and ceramics on the poles	As needed
	Clean contacts on the component boards	As needed
	Vacuum the component boards	As needed
	Clean all fan screens	Weekly
	Vacuum outside of instrument	Weekly
	Clean grob and replace quartz insert	As needed
	Replace septum	Daily (each shift)
	Injection port liner checked	Daily
	Column maintenance	As needed
	Disk Drive	Semi-Annual (Service the Engineer) or as needed
	Printer	Quarterly

Table 7-1 continued

Infrared Spectrophotometer	Clean cells	Daily
pH Meter	Electronics checked	Daily
	Electrolyte changed	Checked weekly, changed when low
Threshold Limit Valve Meter (HCVP)	Check valves	Weekly
	Check pump	Weekly
	Check electronics	Weekly
	Check detector	Weekly
Total Organic Carbon Instrument	Check oxygen purity	Each new cylinder
	Check heater	
	Add phosphoric acid	Monthly
UV/VIS Spectrophotometer	Lamp	As needed
	Wavelength checked	Quarterly
	Serviced	As needed
HPLC	Carbon check	Daily
	Oil levels	Weekly
	Filters	Monthly
	Pump Drives	Quarterly

8.0 ANALYSIS OF QUALITY CONTROL SAMPLES

This section discusses samples which are routinely added to the normal laboratory sample stream to demonstrate that the laboratory is operating within prescribed requirements for accuracy and precision. Quality control samples are of known content and concentration (with the exception of field blanks) so that accuracy and precision can be determined and control charts can be prepared. Evaluation of these data are discussed in Section 10.1.

Table 8.1 summarizes the quality control samples which are analyzed in ITAS laboratories. Included in the table are:

- Type of sample.
- Purpose of the sample.
- Frequency with which the sample is to be analyzed within the normal sample stream.
- Applicability of the sample to organic or inorganic analyses, with a citation if the sample is for GC/MS analysis only.
- Whether the sample is used for the statistical evaluation of accuracy and/or precision.
- Person responsible for introducing the quality control sample into the sample stream. If the sample is introduced by the Quality Control Coordinator, the content and/or concentration of the sample and its occurrence is unknown to the Analyst. Samples unknown to the analyst provide independent verification of laboratory operation.

Following is a discussion of the major types of quality control samples. QC samples will be analyzed as recommended herein, unless analytical procedures prescribe specific sample analysis. If the procedure is specific, the procedural requirements will be met. A discussion of the ITAS interlaboratory testing program follows. Figures 8.1 and 8.2 show how different QC samples may be used to provide bias and precision estimates.

9 1 1 2 7 4 0 1 3 5

As stated, Section 10.1 presents the statistical analyses of these samples.

8.1 TYPES OF QUALITY CONTROL SAMPLES

Type of Quality Control Sample	Section No.
Trip Blank Analyses	8.1.1
Field Blank Analyses	8.1.2
Rinsate Blank Analyses	8.1.3
Method Blank Analyses	8.1.4
Reagent Blank Analyses	8.1.5
Bottle Blank Analyses	8.1.6
Duplicate Sample Analyses	8.1.7
Blind Duplicate Analyses	8.1.8
Check Standard Analyses	8.1.9
Surrogate Standard Analyses	8.1.10
Laboratory Matrix Spike Analyses	8.1.11
Laboratory Matrix Spike Duplicate Analyses	8.1.12
Verification/Reference Standard Analyses	8.1.13
Blank Spike Analyses	8.1.14
Field Matrix Spike Analyses	8.1.15
Analysis Matrix Spike Analyses	8.1.16
Internal Standard Spike Analyses	8.1.17
Collocated Samples Analyses	8.1.18
Replicated Sample Analyses	8.1.19
Split Sample Analyses	8.1.20

8.1.1 Trip (Travel) Blank Analyses

Volatile organics samples are susceptible to contamination by diffusion of organic contaminants through the teflon-faced silicone rubber septum of the sample vial; therefore, trip blanks (also referred to as travel blanks) shall be analyzed to monitor for possible sample contamination during shipment. Trip blanks will be prepared by filling two VOA vials (40 ml) with organic-free water and shipping the blanks with the field kit. Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. Following the analyses, if the trip blanks indicate possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may be corrected for the trip blank concentration or th sources resampled. Results of trip blank analyses should be maintained with the corresponding sample analytical data in the project file.

9 1 1 2 1 5 1 0 1 3 6

8.1.2 Field Blank Analyses

A field blank is a volume of water (or soil) that is provided by the sample collectors to demonstrate the absence of contamination during sampling. Deionized, distilled laboratory water, or previously-prepared solid materials (lab sand, etc.) is placed into sample containers by the sample collection crew, packaged, and shipped with the other field samples. If analysis of the field blanks indicates possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may be corrected for the field blank concentration or the sources resampled. Contamination sources to be checked include: Containers; sample storage facilities; field handling procedures; sampling tools (also see rinsate blanks). Results should be maintained with the corresponding sample analytical data in the project file.

8.1.3 Rinsate Blank Analyses

A rinsate blank is a volume of rinse solution (deionized, distilled lab water or organic solvent) used to rinse a sampling tool which contacts multiple samples. The rinse solution is collected after the tool has collected a sample and has been cleaned, to demonstrate that there is no residual contamination remaining on the tool to carry over into the next sample. If the rinsate blank indicates possible contamination of the succeeding samples, the samples may be corrected for the rinsate blank concentration or the sources resampled. Results of rinsate blank analyses should be maintained with the corresponding sample analytical data in the project file.

8.1.4 Method Blank Analyses

A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or sample weight processed. A method blank should be performed with each group of samples. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and

9 1 1 2 7 5 0 1 3 7

minimized. Optimally, a method blank should contain no greater than two times (2X) the method detection limit for the parameter. Results of method blank analyses should be maintained with the corresponding analytical data in the project file. (See also Section 6.2.1.1).

8.1.5 Reagent Blank Analyses

A reagent blank is composed of the materials which will be added to client samples during preparation, and then will be analyzed for specific parameters. It is run prior to the use of the materials on "real" samples, to verify that no contaminants are present at levels which would affect sample results. If such contaminants are found, corrective actions must be taken. Records of reagent blanks, including solvent lots and column adsorbent test results, are stored in Quality Operation files. The reagent blank may be run as a method blank with client samples, but if contamination is present, samples, will require reanalysis with a clean blank demonstrating reagent purity.

8.1.6 Bottle Blank Analyses

At a frequency of 1 percent or greater, laboratory-prepared sample containers are tested to verify that the glassware cleaning procedure is performed acceptably. Parameters of concern for the particular container are tested (metals for plastic containers, organics for glove, etc.). Results of the tests from each batch are filed with the shipping/receiving clerk or sample custodian. Records of traceability of these containers through each project must be maintained. Vendors must supply certificate of cleanliness for purchased, pre-cleaned, containers, and these records must also be maintained.

8.1.7 Duplicate Sample Analyses

Duplicate analyses are performed to evaluate the precision of an analysis. Results of the duplicate analyses are used to determine the relative percent difference between replicate samples. Criteria for evaluating duplicate sample results are provided in Section 10.1. A duplicate analysis should be performed whenever a group of five or more samples are analyzed at one time.

9 1 1 2 3 6 1 0 1 3 8

Duplicate analysis results should be summarized on the quality control data summary form.

8.1.8 Blind Duplicate Analyses

A blind duplicate sample is a duplicate sample which has been introduced as a separate sample by the Quality Control Coordinator during the log-in process or prior to analysis. Evaluation of the replicate is discussed in Section 10.1. This data is reported to and summarized by the Quality Control Coordinator.

8.1.9 Check Standard Analyses

Because standards and calibration curves are subject to change and can vary from day to day, a midpoint standard or check standard should be analyzed with each group of samples. Analysis of this standard is necessary to verify the standard curve and may serve in some cases as sufficient for calibration. This value should be entered in the instrument calibration log whenever performed. Check standard analyses results should be summarized on the quality control data summary form.

8.1.10 Surrogate Standard Analyses

Surrogate standard determinations should be performed on all samples and blanks for GC/MS analyses. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction to monitor preparation and analysis of samples. Recoveries should meet EPA acceptance criteria which are established as laboratory results become available. Surrogate standard data should be summarized on the surrogate standard recovery form.

8.1.11 Laboratory Matrix Spike Analyses

To evaluate the effect of the sample matrix upon analytical methodology, a separate aliquot sample should be spiked with the analyte of interest and analyzed with the sample. The percent recovery for the respective compound will then be calculated. If the percent recovery falls outside established

quality control limits the data should be evaluated and the sample reanalyzed if criteria are not met. Matrix spike results should be summarized on the quality control data summary sheets. This type of matrix spike does not necessarily reflect the behavior of the field-collected target analyte, especially if the target analyte is not stable during shipping or storage.

8.1.12 Laboratory Matrix Spike Duplicate Analyses

Similar in concept to the matrix spike sample above, it is a separate aliquot sample that is spiked with the analyte(s) of interest and analyzed with the associated sample and sample matrix spike. If the percent recovery falls outside established quality control limits, that data should be evaluated and the sample reanalyzed if criteria are not met. The comparison of the recoveries of the spiked compounds in the matrix spike and the matrix spike duplicate samples is made to determine the relative percent difference between the MS/MSD samples. RPD criteria and results should be summarized on the quality control data summary form.

8.1.13 Verification/Reference Standard Analyses

On a quarterly basis, the Quality Control Coordinator should introduce a group of prepared verification samples, or standard reference materials, into the analytical testing regime. Results of these data will be summarized, evaluated, and presented to laboratory management for review and corrective actions, if appropriate. The data are reported to and summarized by the Quality Control Coordinator.

8.1.14 Blank Spike Analyses

A blank spike is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples which is spiked with parameters of interest and carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of spiked materials demonstrates that the laboratory techniques for this method are in control. This sample is recommended in conjunction with matrix spike/matrix spike duplicate (MS/MSD) samples on those sample matrices which are

9 1 1 2 7 6 1 0 1 4 0

anticipated to cause analytical difficulties due to matrix interferences. If the MS/MSD pair shows poor recoveries due to interferences, yet the blank spike sample is acceptable, this is strong evidence that the method has been performed correctly by the laboratory for these samples, but matrix interferences have affected the results. Results of blank spike analyses should be maintained with the corresponding MS/MSD and sample analytical data in the project file.

8.1.15 Field Matrix Spike Analyses

This is a sample created by spiking target analytes into a portion of a sample in the field at the point of sample acquisition. This sample provides information on the target analyte stability after collection and during transport and storage.

8.1.16 Analysis Matrix Spike Analyses

This is a sample created by spiking target analytes into a prepared portion of a sample just prior to analysis. It only provides information on matrix effects encountered during analysis, i.e., suppression or enhancement of instrument signal levels. It is most often encountered with elemental analyses involving the various forms of atomic spectroscopy and is often referred to as "standard additions."

8.1.17 Internal Standard Spike (or Recovery Standard) Analyses

This is an analyte which has the same characteristics as the surrogate, but is added to a sample just prior to analysis. It provides a short-term indication of instrument performance, but it may also be an integral part of the analytical method in a non-quality control sense, e.q., to normalize data for quantitation purposes.

8.1.18 Collocated Samples Analyses

Collocated samples are independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. Examples of collocated samples include: samples

from two air quality analyzers sampling from a common sample manifold or two water samples collected at essentially the same time and from the same point in a lake.

Collocated samples when collected, processed, and analyzed by the same organization provide intralaboratory precision information for the entire measurement system including sample acquisition, handling, shipping, storage, preparation, and analysis. Both samples can be carried through the steps in the measurement process together to provide an estimate of short-term precision for the entire measurement system. Likewise, the two samples, if separated and processed at different times or by different people, and/or analyzed using different instruments, provide an estimate of long-term precision of the entire measurement system.

Collocated samples when collected, processed and analyzed by different organizations provide interlaboratory precision information for the entire measurement system.

8.1.19 Replicated Sample Analyses

A replicated sample is a sample that has been divided into two or more portions, at some step in the measurement process. Each portion is then carried through the remaining steps in the measurement process (see also duplicate sample 8.1.5).

8.1.20 Split Sample

A split sample is a sample divided into two portions, one of which is sent to a different organization or laboratory and subjected to the same environmental conditions and steps in the measurement process as the one retained.

A replicated or split sample can be divided into portions (or split) at different points in the sampling and analysis process to obtain precision information on the various components of the measurement system. For example, a field replicated, or field split sample, provides precision information

about all steps after sample acquisition including effects of storage shipment, analysis, and data processing; whereas, information on the intra- and interlaboratory precision of sample preparation and analysis steps of the measurement system is provided by samples subdivided once they are received in the laboratory, i.e., laboratory replicated or laboratory split samples, respectively. A sample divided into two portions just prior to analysis, i.e., an analysis replicate, provides information on the precision of the analytical instrumentation.

The replicated sample can provide short-term or long-term precision estimates by processing the two portions together or separating them for processing at different times and under different conditions as discussed above for collocated samples.

8.2 INTERLABORATORY (ROUND ROBIN) VERIFICATION SAMPLES

On an annual basis, ITAS will conduct an interlaboratory round robin testing program for organic and inorganic parameters which are common to all laboratories. Samples will be prepared on an annual rotating basis by the Quality Control Coordinator of an ITAS laboratory. Results of the round-robin program will be determined by the Quality Control Coordinator preparing and distributing the samples. A report of the "true" values, acceptable statistical ranges and values determined by a specific laboratory will be issued to the respective Laboratory Manager. Results of all ITAS laboratories will be presented to the Vice President, Analytical Services for use in the annual management laboratory review.

8.3 QC LEVELS

There are several types of QC samples which may be applied to different projects at varying frequencies; these may also be reported in summary fashion, or in detail with all raw and associated data provided. The costs to the laboratory and the client will vary depending upon the types of samples, analyses, and data reported. This section describes the levels available from ITAS laboratories and their applications. The QC level should be specified

9 1 1 2 1 5 4 0 1 4 3

where requesting analyses (use the Request for Analysis form).

Level I -- ITAS standard practice. Use available analytical procedures. Fifteen percent QC samples (blank/spike/duplicate) for every 20 samples. QC samples may not be performed for a specific project, but as part of compiled sets of samples. QC data are not reported as part of analytical results. ITAS published rates apply to client samples tested.

Level II - Use available analytical (reference) methods. Fifteen percent QC samples minimum (blank, duplicate, spike, or duplicate spike). QC samples are project or client-specific. QC summary report is included as part of the analytical results reported. No raw data are included. Each QC sample is billed as a "real" analytical sample.

Level III - The highest level requires use of referenced regulatory procedures, and/or established/verified procedures using confirmatory type techniques (i.e., GC/MS, dual column GC). Method blank plus 20 percent or two QC samples minimum per each matrix. A QC summary report is supplied with supporting data. Where applicable, this level is the USEPA Contract Laboratory Program (CLP) package. A surcharge is added and/or QC samples are billed at sample rates. Costs will be based on the analytical program required.

Project-specific requirements must be defined in a specific QA Project Plan or Work Plan. Project specific documentation must be submitted to the laboratory before beginning work. Project requirements for QC samples cannot be less than Level I.

Table 8.2 provides a quick reference for the QC levels and their types of QC sample information.

TABLE 8.1. QUALITY CONTROL SAMPLES

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
Trip Blank	40-ml VOA vial filled with organic free water and taken with field sample collection kit. Used to verify that contamination of soil/ water VOA samples has not occurred due to shipment and sample containers being in field.	Two trip blank vials with every set of collection vials sent to field; each VOA cooler, returned to the lab should contain two trip blanks.		X	Accuracy	Supplier of Containers
Field Blank	A volume of "clean" collection media is added to the container to verify absence of field contamination. May be water, soil, or other material.	Specified in Project Work Plan	X	X	Accuracy	Field Sampler
Rinsate Blank	Rinse of field sample collection equipment to verify cleanliness, eliminate carryover of contamination to later samples.	Specified in Project Work Plan	X	X	Accuracy	Field Sampler
Method Blank (Process Blank)	The analysis is performed using only the reagents and solvents used in the method. Determines cumulative interference. If interference cannot be eliminated, it must be	With each group of samples, or beginning and end of each run. For GC/MS, method is spiked with surrogates 1 out of 10.	X	X	Accuracy	Analyst

TABLE 8.1. (Continued)

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
	considered when computations are performed.					
Reagent Blank	Determine the background of each reagent/solvent to be used in an analyses. Must use identical conditions to actual analyses including detection system. Background must not interfere with intended analyses.	Can be done as part of method blank, determine separately with each new batch of reagent/solvents.	X	X	Accuracy	Analyst
Bottle Blank	Rinse of cleaned sample container, analyzed prior to field shipment to demonstrate lack of contaminants.	At least 1% of each container batch; does not apply to vendor-certified containers	X	X	Accuracy	QC Coordinator or Shipping/Receiving Clerk
Duplicate	An aliquot of a sample known to Analyst. Calculate Relative Percent Difference.	1 out of 20 or at least 1/month/run	X	X	Precision	Analyst
Blind Duplicate	An aliquot of a sample introduced unknown to analyst at time of sample log in by QC Coordinator (known to QC Coordinator).	1 out of 100	X	X	Precision	QC Coordinator

TABLE 8.1. (Continued)

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
Check Standard	Analysis of standard with concentrations at mid-point or low end of standard curve to verify standard calibration curve.	With each group of samples.	X		Accuracy	Analyst
Surrogate Standards	For GC/MS and GC analy-, sis the addition of non-priority pollutants as spikes in standards, method blanks and samples. 3-point calibration curve, When used with daily determines Mean and Warning and Control Limits.	All standards, method blanks, and samples.		X GC/MS only	Accuracy	Analyst
Spiked Samples (Laboratory Matrix Spikes)	A known concentration of a specific parameter is added to an aliquot of a sample with the matrix of interest. Percent recovery is determined and spike is compared against an unspiked aliquot.	1 out of 20 or at least 1/month/ run	X	X	Accuracy	Analyst
Matrix Spike Duplicate	Percent recovery is determined and compared against matrix spiked sample.	1 out of 20 or at least 1/batch		X	Both	Analyst

TABLE 8.1. (Continued)

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
Standard Reference Materials	Standards prepared by recognized external agency and used to determine performance of entire system, instrumentation and Analysts.	Scheduled periodic basis such as quarter or semi-annually.	X	X	Accuracy	Analyst or QC Coordinator
Blank Spike	Blank material spiked with parameters of interest. Recovery evaluated for proper execution of technique or analytical method.	1 out of 20 or at least 1/batch with MS/MSD pair		X	Accuracy	Analyst
Field Matrix Spike	Sample spiked in field to show target analyte stability after collection, shipment, storage, preparation, and analysis	5 percent recommended	X	X	Accuracy	Field sampler
Analysis Matrix Spike	Sample or extract spiked just prior to analysis. Shows matrix effects during analysis. Also known as "Method of Standard Additions" spike	As specified in methods, or as needed	X		Accuracy	Analyst
Internal Standards	Nonpriority pollutant spiked into sample and method blank after extraction to monitor instrument performance and sensitivity should surrogate standards indicate a problem.	Each sample and standard		GC/MS VOA and Base-Neutral/ Acid Analysis Base-Neutral	Accuracy	Analyst

TABLE 8.1. (Continued)

TYPE	PURPOSE OF SAMPLE	FREQUENCY	APPLICABILITY		ACCURACY AND PRECISION APPLICATION	INTRODUCED BY FIELD SAMPLER/ ANALYST/QC COORDINATOR
			INORGANIC	ORGANIC		
Collocated samples	Independent samples collected to be equally representative of the variables of interest	5 percent recommended	X	X	Precision	Field sampler
Replicated sample	Collected sample that has been split into two or more parts in the field, sent to the same laboratory for analysis	5 percent recommended	X	X	Precision	Field sampler
Split sample	Sample split into two or more portions, and sent to several laboratories for analysis	1 percent recommended	X	X	Precision	Field sampler or QC coordinator

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Table 8.2 QC Samples and QC Levels

	QC Samples*					
QC level	Method Blanks (%)	Matrix Spikes (%)	Duplicates and/or Duplicate Spikes (%)	Are QC Results Reported?	Are QC Samples Project-specific?	QC Sample Costs
I (15%)	5	5	5	No	No	Unbilled
II (15%)	5	5	5	Yes, summary	Yes	Per sample
III (blank +20%)	5	10	10	Yes, summary plus raw data	Yes	Surcharge and/or per sample
Project-specific	To be established individually on project-specific basis			Project-specific	Project-specific	Project-specific

*Frequencies listed are minimum goals but may vary depending upon rate of sample receipt and number of samples per analytical batch. Other types of samples, such as "blank spikes," which may be analyzed to demonstrate laboratory technique is in control despite matrix interferences in samples received, may also be billed.

Sample Spiking Point			Data Interpretation
Sample Acquisition	Preparation	Analysis	
Field Matrix Spike			Provides a best case estimate of bias based on recovery; includes matrix effects associated with sample preservation, shipping, preparation and analysis.
	Lab Matrix Spike		Provides an estimate of bias based on recovery. Incorporates matrix effects associated with sample preparation and analysis only.
		Analysis Matrix Spike	Provides an indication of matrix effects associated with the analysis process only.

Figure 8-1 The Use of Target Analyte Spikes for Bias Estimation

from "Chapter 5, Calculation of Precision, Bias, and Method Detection Limit for Chemical and Physical Measurements"; issued by Quality Assurance Management and Special Studies Staff, Office of Monitoring Systems and Quality Assurance, Office of Research and Development, USEPA, Washington, D.C. 20460

9 1 1 2 7 4 0 1 5 1

Data Quality Assessment Sample Point of Origination			Data Interpretation
Sample Acquisition	Preparation	Analysis	
Collocated Samples			Best estimate of intra- or interlaboratory precision of the entire measurement system
Field Replicate			Second best estimate of intralaboratory precision of measurement system from sample acquisition through analysis.
Field Split			Second best estimate of interlaboratory precision of the measurement system from sample acquisition through analysis.
	Lab Replicate		Best estimate of intralaboratory precision of sample preparation and analysis.
	Lab Split		Best estimate of interlaboratory precision of sample preparation and analysis.
		Analysis Replicate	Estimate of intralaboratory precision of analysis.
		Analysis Split	Estimate of interlaboratory precision of analysis.

Figure 8-2 Precision Evaluation Samples.

from "Chapter 5, Calculation of Precision, Bias, and Method Detection Limit for Chemical and Physical Measurements"; issued by Quality Assurance Management and Special Studies Staff, Office of Monitoring Systems and Quality Assurance, Office of Research and Development, USEPA, Washington, D.C. 20460

9 1 1 2 7 6 1 0 1 5 2

9.0 ANALYTICAL PROCEDURES

Analytical procedures for the analysis of samples include the following:

- Prescribed method for sample preparation, including observance of stated sample holding times (such as shown on Table 5.1) and necessary extractions, dilutions, etc.
- Instrument standardization, including calibration and preventive maintenance
- Analytical techniques to be used in processing the sample
- Prescribed format on prepared data sheets for recording raw data which include:
  - Identification of project(s)
  - Identification of sample number(s)
  - Identification of Analyst
  - Identification of Analyst performing data validation
  - Dates for sample analysis and data validation
  - Raw data resulting from the analysis with appropriate calibration standards and blanks.
- Method for computation of analytical results, which can be included on the data sheet.

Documentation of the above as an analytical procedure is dependent upon the specific instrumentation and data collection and reduction methods used within a laboratory. Therefore, ITAS laboratories shall prepare and maintain as a Laboratory Specific Attachment to this manual an Analytical Procedures Manual which includes the above for all analyses performed. The requirements of this Quality Assurance Manual for activities such as calibration, preventive maintenance, and quality control sample analysis shall be incorporated into the laboratory specific manuals as appropriate.

9.1 ANALYTICAL METHODS

Whenever possible, ITAS laboratories utilize industry recognized analytical methods from source documents published by agencies such as the U.S. Environmental Protection Agency (USEPA), American Public Health Association (APHA), American Society for Testing and Materials (ASTM), and the National Institute for Occupational Safety and Health (NIOSH).

Tables 9.1 through 9.8 provide a compendium of the analytical methods typically used throughout ITAS. The format presented in Tables 9.1A through 9.1E and Table 9.2 has been adopted from the referenced USEPA document. Tables 9.3 through 9.8 provide analytical procedures for analyses not cited in Tables 9.1A through 9.1E and procedures for different matrices. Table 9.9 lists detection limits for the USEPA's Toxicity Characteristic Leaching Procedure (TCLP).

9.2 DETECTION LIMITS

All analytical methodologies have an associated method detection limit below which an analyte present in the sample cannot be accurately measured.

The detection limit of a method is generally defined as the quantity of analyte which results from the lowest differential between a signal caused by the analyte and that of random noise. A factor of 2 is generally used. Analytes present in concentrations below the detection limit WILL NOT appear as present in the sample when using this analytical technique. A detection limit quantity is reported as a less than value(<). This less than value does not indicate that an analyte is not present in a sample but only that it is present at levels below the detection limit. For results produced by USEPA CLP methods, values which are below required detection limits, but can still be quantified, are reported inside brackets [ ] as "estimated concentrations."

The detection limits attained by the EPA methods used in the laboratory are equal to or better than required by EPA regulations. The accompanying pages

indicate these detection limits. It is important to remember that specific detection limits are highly matrix dependent and the detection limits listed herein are provided for guidance and may not always be achievable.

9.2.1 Inorganics

Wet chemical techniques for nutrient, demand, and mineral constituents are relatively free from interferences in the analysis of aqueous samples. Analyses of these constituents in solid phase samples are not routinely performed. High concentrations of any of these analytes require dilution of the sample, with corresponding changes to detection limits.

9.2.2 Metals

The detection limit for metals in aqueous samples is affected by the salt content total dissolved solids (TDS). If the TDS is above 5,000 mg/liter, the sample may require dilution to prevent background light-scattering effects and false positives. Solids and sludges for analysis require a digestion prior to analysis. The detection limit for the analyte of interest is equal to the detection limit in water times a digestion factor equal to the final volume of digestate divided by the weight of solids digested.

9.2.3 Organics

Organic compounds analyzed by GC or GC/MS are analyzed as either volatile or extractable constituents. Factors which affect the detection limit of these constituents fall into the following categories.

- Contaminated samples (samples containing the analytes of interest). Samples containing as little as 0.5 ppm of chromatographable organics may require dilution. The analyst must make a dilution decision based on the knowledge of the sample and the observation of the sample.

- Background contamination, that is, a sample may have a high organic loading but not of the compounds of interest. This background may mask the compounds of interest and must be diluted from the matrix.

- Sample preparation techniques--Water samples are extracted at a 1000:1 concentration factor. One liter of sample is extracted in triplicate and concentrated to 1 ml. Soil samples are prepared by extracting 20 g of soil and concentrating to 1 ml. The resultant detection limits are 50 times greater than for water.

- Clean-up preparations--All analytical techniques for organics allow for the use of column chromatographic clean-up techniques to eliminate background and non-chromatographic organic loadings. This preparation step also dilutes the sample with the resultant rise in detection limit.

In short, many factors affect the specific detection limit of sample and no absolute may be made except for the use of average conditions. The preceding factors all affect the detection limit from sample to sample. The following tables provide the detection limits for various parameters analyzed within the analytical laboratory.

9.3 VARIANCE FROM STATED ANALYTICAL METHODS

Analyses will be performed in accordance with the methods cited herein unless specific project requirements or needs dictate adoption of an alternate method or modification of the cited methods. For example, GC/MS procedures may be "CLP-modified", as specified by the USEPA's Contract Laboratory Program (CLP).

Based on laboratory experience with a procedure and matrix type, ITAS has developed for some analytes in-house Standard Operating Proceudres (SOPs) based on regulatory methods but which may include modifications to improve reproducibility and/or accuracy.

If analysis is performed in an alternate manner, the method shall be documented in the project records as discussed in Section 12.1 of this manual.

Table 9.1
Method Summary for Organic Compounds in Water and Wastewater

Parameter	Method No.	Preferred Equipment	Detection Limit	Reference
Purgeable Hydrocarbons	601	Purge and Trap Gas Chromatography	0.005 to 0.04)g/l, 0.02 to 1.81)g/l	Federal Register[1] Federal Register[4]
Purgeable Aromatics	602	Purge and Trap Gas Chromatography	0.01 to 0.04)g/l 0.02 to 0.04)g/l	Federal Register[1] Federal Register[4]
Acrolein/Acrylonitrile	603	Purge and Trap Gas Chromatography	10 to 100)g/l 0.5 to 0.7)g/l	Federal Register[1] Federal Register[4]
Phenols	604	Gas Chromatography	1 to 10)g/l 0.14 to 16.0)g/l	Federal Register[1] Federal Register[4]
Benzidines	605	High Performance Liquid Chromatography	0.5 to 0.1)g/l 0.08 to 0.13)g/l	Federal Register[1] Federal Register[4]
Phthalate Esters	606	Gas Chromatography	2.0 to 3.0)g/l FID, 0.02 to 0.20)g/l ECD 0.29 to 3.0)g/l	Federal Register[1] Federal Register[4]
Nitrosamines	607	Gas Chromatography	0.3 to 1.0)g/l 0.15 to 0.81)g/l	Federal Register[1] Federal Register[4]
Organochlorine Pesticides and PCBs	608	Gas Chromatography	0.1 to 0.10)g/l 0.002 to 0.24)g/l	Federal Register[1] Federal Register[4]
Nitroaromatics and Isophorone	609	Gas Chromatography	5.0)g/l FID, 0.05)g/l ECD 3.6 to 5.7)g/l	Federal Register[1] Federal Register[4]
Polynuclear Aromatic Hydrocarbons	610	High Performance Liquid Chromatography	0.1 to 5.0)g/l 0.013 to 2.3)g/l	Federal Register[1] Federal Register[4]

Table 9.1 (Continued)
Method Summary for Organic Compounds in Water and Wastewater

Parameter	Method No.	Preferred Equipment	Detection Limit	Reference
Haloethers	611	Gas Chromatography	0.4 to 1.1)g/l 0.3 to 3.9)g/l	Federal Register[1] Federal Register[4]
Chlorinated Hydrocarbons	612	Gas Chromatography	0.001 to 0.015)g/l 0.03 to 1.34)g/l	Federal Register[1] Federal Register[4]
2,3,7,8-Tetrachloro-dibenzo-p-dioxin	613	Gas Chromatograph, Gas Chromatography Mass Spectrometry	0.003)g/l GC, 0.003)g/l GC/MS 0.002)g/l	Federal Register[1] Federal Register[4]
Organics Screen	503.1	Gas Chromatography	0.1 to 100)g/l	EPA, May 1980[3]
Purgeable Organics	624	Gas Chromatography/ Mass Spectrometry	10 to 100)g/l 1.6 to 7.2)g/l	Federal Register[1] Federal Register[4]
Base-Neutral Extractables and Acid Extractables	625	Gas Chromatography/ Mass Spectrometry	10 to 25)g/l 1.6 to 36)g/l	Federal Register[1] Federal Register[4]
Chlorophenoxy Acid Herbicides	615	Gas Chromatography	0.05 to 0.50)g/l	EPA, 1978[2]
Safe Drinking Water Act Pesticides	-	Gas Chromatography	0.02 to 1.0)g/l	EPA, 1978[2]
Organophosphorous Pesticides	-	Gas Chromatography	0.1 to 1.0)g/l	EPA, 1978[2]
Polychlorinated Biphenyls (PCBs)	-	Gas Chromatography	10)g/l	EPA, 1978[2]
Triazine Pesticides	-	Gas Chromatography	10 to 20)g/l	EPA, 1978[2]

Table 9.1 (Continued)

(1) Federal Register, Volume 44, No. 233, Monday, December 3, 1979, 40 CFR Part 136, pp. 69464 to 69575.

(2) "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, U. S. Environmental Protection Agency, Cincinnati, Ohio, September, 1978.

(3) "The Analysis of Aromatic Chemicals in Water by the Purge and Trap Method," Method 503.1, U.S. Environmental Protection Agency, Physical and Chemical Methods Branch, Cincinnati, Ohio, May, 1980.

(4) "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," 40 CFR Part 136, Federal Register, Friday, October 26, 1984.

Table 9.1A
List of Approved Biological Test Procedures

Parameter and Units	Method[1]	Reference (Method Number or Page) EPA[2]	Standard Methods 16th Ed.	ASTM	USGS[3]
Bacteria:					
1. Coliform (fecal), number per 100 ml	MPN, 5 tube, 3 dilution;	p. 132	908C	-	-
	or membrane filter (MF),[4] single step	p. 124	909C	-	B-0050-7
2. Coliform (fecal) in presence of chlorine, number per 100 ml	MPN, 5 tube, 3 dilution	p. 132	908C	-	-
	or membrane filter (MF), single step [4A]	p. 124	909C	-	B-0050-7
3. Coliform (total), number per 100 ml	MPN, 5 tube, 3 dilution; or MF[4] single step or two step	p. 114	908A	-	-
		p. 108	909A	-	B-0025-7
4. Coliform (total) in presence of chlorine, number per 100 ml	MPN, 5 tube, dilution; or MF[4] with enrichment	p. 114	908A	-	-
		p. 111	909 (A+A.5c)	-	-
5. Fecal streptococci, number per 100 ml	MPN, 5 tube, 3 dilution; MF;[4] or plate count	p. 139	910A	-	-
		p. 136	910B	-	B-0055-7[5]
		p. 143	910C	-	-

Table 9.1A Notes

[1] The method must be specified when results are reported.

[2] "Microbiological Methods for Monitoring the Environment, Water and Wastes, 1978," EPA-600/8-78-017, U.S. Environmental Protection Agency."

[3] Greeson, P.E. et al., "Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A4, Laboratory Analysis, 1977.

[4] 0.45 m membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and free of extractables which could interfere with their growth and development.

[4A] Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPV method will be required to resolve any controversies.

[5] Approved only if dissolution of the KF Streptococcus Agar (Section 5.1, USGS Method B-0055-77) is made in a boiling water bath to avoid scorching of the medium.

Table 9.1B
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
1. Acidity, as $CaCO_3$, mg/l: Electrometric end point or phenolphthalein end point.	305.1	402(4.a)	D1067-82(E)	-	-
2. Alkalinity, as $CaCO_3$, mg/L: Electrometric or colorimetric:					
Titration to pH 4.5, manual	310.1	403	D1067-82(B)	I-1030-84	33.014[2]
Or automated	310.2	-	-	I-2030-84	-
3. Aluminum--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	202.1	303C	-	I-3051-84	-
AA furnace	202.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Eriochrome cyanine R)	-	306B	-	-	-
4. Ammonia (as N), mg/L: Manual distillation[5] (at pH 9.5) followed by:	350.2	417A	-	-	33.057[2]
Nesslerization	350.2	417B	D1426-79(A)	I-3520-84	33.057[2]
Titration	350.2	417D	-	-	-
Electrode	350.3	417E or F	D1426-79(D)	-	-
Automated phenate, or	350.1	417G	D1426-79(C)	I-4523-84	-
Automated electrode	-	-	-	-	(6)
5. Antimony--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	204.1	303A	-	-	-
AA furnace, or	204.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
6. Arsenic--Total,[3] mg/L:					
Digestion[3] followed by					
Hydride	206.5	-	-	-	-
AA furnace	206.3	303E	D2972-84(B)	I-3062-84	-
Inductively coupled plasma	206.2	304	-	-	-
Or, colorimetric (SDDC)	-	-	-	-	Method 200.7[4]
	206.4	307B	D2972-84(A)	I-3060-84	-
7. Barium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	208.1	303C	-	I-3084-84	-
AA furnace, or	208.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
8. Beryllium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	210.1	303C	D3654-84[A]	I-3095-84	-
AA furnace	210.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (aluminon)	-	309B	-	-	-
9. Biochemical oxygen demand (BOD_5), mg/L:					
Dissolved oxygen depletion	405.1	507	-	I-1578-78[7]	33.019[2], p. 17[8]
10. Boron--Total, mg/L:					
Colorimetric (curcumin) or	212.3	404A	-	I-3112-84	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
11. Bromide, mg/L: Titrimetric	320.1	-	D1246-82(C)	I-1125-84	P.544[9]

PIT:PM-9-1B

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
12. Cadmium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	213.1	303A or 303B	D3557-84(A or B)	I-3135-84 or I-3136-84	33.089[2]
AA furnace	213.2	304	-	-	Pg. 37[8]
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Voltametry[10] or	-	-	D3557-78(C)	-	-
Colorimetric (Dithizone)	-	310B	-	-	-
13. Calcium--Total,[3] mg/L: Digestion[3] followed by:					
Atomic absorption	215.1	303A	D511-84(B)	I-3152-84	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or EDTA titration	215.2	311C	D511-84(A)	-	-
14. Carbonaceous Biochemical oxygen demand[11] ($CBOD_5$), mg/L: Winkler (Azide modification) or electrode method with nitrification inhibitor	-	507(5,6)	-	-	-
15. Chemical oxygen demand (COD), mg/L:					
Titrimetric colorimetric	410.1	508A	D1252-83	I-3560-84 or	33.034[2]
Manual or	410.2	-	-	I-3562-84	P. 17[8]
	410.3	-	-	-	-
Automated	410.4	-	-	-	-
Spectrophotometric	-	-	-	I-3561-84	(12,13)
16. Chloride, mg/L:					
Titrimetric (silver nitrate) or	-	407A	D512-81(B)	I-1183-84	-
Mercuric nitrate	325.3	407B	D512-81(A)	I-1184-84	33.067[2]
Colorimetric (ferricyanide) manual or	-	-	D512-81(C)	I-1187-84	-
Automated	325.1 or 325.2	407D	-	I-2187-84	-

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
17. Chlorine--Total residual, mg/L:					
Titrimetric-amperometric,	330.1	408C	D1253-76(A)	-	-
Starch end point direct,	330.3	408A	D1253-76(B)	-	-
Back titration either end point [14] or	330.2	408B	-	-	-
DPD-FAS	330.4	408D	-	-	-
Spectrophotometric, DPD, or	330.5	408E	-	-	-
Electrode	-	-	-	-	(15)
18. Chromium VI dissolved, mg/L: 0.45 micron filtration with:					
Extraction and atomic absorption, or	218.4	312B	-	I-1232-84	-
Colorimetric (Diphenylcarbazide)	-	-	-	I-1230-84	307B[16]
19. Chromium--Total,[3] mg/L:					
Digestion[3] (optional extraction) followed by:	218.3	303B	-	-	-
AA direct aspiration	218.1	303A	D1687-84(D)	I-3236-84	33.089[2]
AA furnace	218.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Diphenylcarbazide)	-	312B	D1687-77(A)	-	-
20. Cobalt--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	219.1	303A or 303B	D3558-84(A or B)	I-3239-84 or I-3240-84	P. 37[8]
AA furnace, or	219.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
21. Color, platinum Cobalt units or dominant wavelength hue, luminance, purity:					
Colorimetric, ADMI	110.1	204D	-	-	(17)
Platinum cobalt; or	110.2	204A	-	I-1250-84	-
Spectrophotometric	110.3	204B	-	-	-

PIT:PM-9-1B

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
22. Copper--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	220.1	303A or 303B	D1688-84(D or E)	I-3271-84 or I-3270-84	33.089[2],P.37[8]
AA furnace	220.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Colorimetric (Neocuproine)	-	313B	D1688-84(A)	-	-
Bicinchoninate	-	-	-	-	(18)
23. Cyanide--Total, mg/L:					
Manual distillation with $MgCl_2$, followed by:		412B	-	-	-
Titrimetric,		412C	-	-	P.22[8]
Manual or	335.2	412D	D2036-82(A)	-	-
Automated[19] spectrophotometric	335.3	-	D2036-82(A)	I-3300-84	-
24. Cyanide amenable to chlorination, mg/L: Manual distillation with $MgCl_2$; followed by titrimetric, manual or automated[15] spectrophotometric	335.1	412F	D2036-82(B)	-	-
25. Fluoride--Total, mg/L:					
Manual distillation[3]	-	413A	-	-	-
Followed by manual or	340.2	413B	D1179-80(B)	-	-
Automated electrode	-	-	-	I-4327-84	-
SPADNS	340.1	413C	D1179-80(A)	-	-
Or automated complexone	340.3	413E	-	-	-
26. Gold--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	231.1	303A	-	-	-
Or AA furnace	231.2	304	-	-	-
27. Hardness--Total as $CaCO_3$, mg/L:					
Automated colorimetric	130.1	-	-	-	-
EDTA titration, or Ca plus Mg as their carbonates, by inductively coupled plasma	130.2	314B	D1126-80	I-1338-84	33.082[2]
Or atomic absorption (see parameters 13 and 33)	-	-	-	-	-

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Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
28. Hydrogen ion (pH), pH units:					
Electrometric measurements	150.1	423	D1293-84(A or B)	I-1586-84	33.062[2]
Or automated electrode	-	-	-	-	(20)
29. Iridium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	235.1	303A	-	-	-
Or AA furnace	235.2	304	-	-	-
30. Iron--Total[3] mg/L:					
Digestion[3] followed by	-			-	-
AA direct aspiration	236.1	303A or 303B	D1068-84 (C or D)	I-3381-84	33.089[2]
AA furnace	236.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Phenanthroline)	-	315B	D1068-84(A)	-	(21)
31. Kjeldahl nitrogen--Total (as N), mg/L:					
Digestion and distillation	351.3	420A or B	D3590-84(A)	-	33.051[2]
Followed by titration	351.3	417D	D3590-84(A)	-	-
Nesslerization or	351.3	417B	D3590-84(A)	-	-
Electrode	351.3	417E or F	-	I-4551-78	-
Automated phenate	351.1	-	-	I-4552-78	-
Semi-automated block digestor	351.2	-	D3590-84(A)	-	-
Or potentiometric	351.4	-	D3590-84(A)	-	-
32. Lead--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	239.1	303A or 303B	D3559-85(A or B)	I-3399-84	33.089[2]
AA furnace	239.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Voltametry[9] or	-	-	D3559-85(C)	-	-
Colorimetric (Dithizone)	-	316B	-	-	-

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Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
33. Magnesium--Total,[3] mg/L: Digestion[3] followed by:					
Atomic absorption	242.1	303A	D511-84(B)	I-3447-84	33.089[2]
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or gravimetric	-	318B	D511-77(A)	-	-
34. Manganese--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	243.1	303A or 303B	D858-84(B or C)	I-3454-84	33.089[2]
AA furnace	243.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (persulfate), or	-	319B	D858-84(A)	-	33.126[2]
Periodate	-	-	-	-	(22)
35. Mercury--Total,[3] mg/L:					
Cold vapor, manual or	245.1	303F	D3223-80	I-3462-84	33.095[2]
Automated	245.2	-	-	-	-
36. Molybdenum--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	246.1	303C	-	I-3490-84	-
AA furnace, or	246.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
37. Nickel--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	249.1	303A or 303B	D1886-84(C or D)	I-3499-84	-
AA furnace	249.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Heptoxime)	-	321B	-	-	-
38. Nitrate (as N), mg/L:					
Brucine sulfate, or	352.1	-	D092-71	-	33.063[2] 4190
Nitrate-nitrite N minus Nitrite N	See parameters 39 and 40	See parameters 39 and 40	See parameters 39 and 40	See parameters 39 and 40	P. 28[8]

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Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
39. Nitrate-nitrite (as N), mg/L: Cadmium reduction,					
Manual or	353.3	418C	D3867-85(B)	-	-
Automated; or	353.2	418F	D3867-85(A)	I-4545-84	-
Automated hydrazine	353.1	-	-	-	-
40. Nitrite (as N), mg/L: Spectrophotometric,					
Manual or	354.1	419	D1254-67	-	(24)
Automated (Diazotization)	-	-	-	I-4540-84	-
41. Oil and grease--total recoverable, mg/L: Gravimetric (extraction)	413.1	503A	-	-	-
42. Organic carbon--total (TOC), mg/L: Combustion or oxidation	415.1	505	D2579-85(A or B)	-	33.044[2] Note 23
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 or	365.1	424G	-	I-4601-84	33.116[2]
Manual single reagent or	365.2	424F	D515-82(A)	-	33.111[2]
Manual two reagents	365.3	-	-	-	-
45. Osmium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration, or	252.1	303C	-	-	-
AA furnace	252.2	304	-	-	-
46. Oxygen, dissolved, mg/L:					
Winkler (Azide modification)	360.2	421B	D888-81(C)	I-1575-78	33.028[2]
Or electrode	360.1	421F	-	I-1576-78	-

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Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
47. Palladium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	253.1	-	-	-	P.527[9]
Or AA furnace	253.2	-	-	-	P.528[9]
48. Phenols, mg/L:					
Manual distillation[25]	420.1	-	D1783-80 (A or B)	-	(26)
Followed by manual	420.1	-	-	-	(26)
Or automated[15] colorimetric (4AAP)	420.2	-	-	-	-
49. Phosphorus (elemental), mg/L: Gas-liquid chromatography	-	-	-	-	(27)
50. Phosphorus--Total, mg/L:					
Persulfate digestion	365.2	424C(III)	-	-	33.111[2]
Followed by manual or	365.2 or 365.3	424F	D515-82(A)	-	-
Automated ascorbic acid reduction;	365.1	424G	-	I-4600-78	-
or semi-automated block digestor	365.4	-	-		33.116[2]
51. Platinum--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	255.1	303A	-	-	-
Or AA furnace	255.2	304	-	-	-
52. Potassium-Total,[3] mg/L: Digestion[3] followed by:					
Atomic absorption,	258.1	303A	-	I-3630-84	33.103[2]
Inductively coupled plasma,	-	-	-	-	Method 200.7[4]
Flame photometric, or	-	322B	D1428-82(A)	-	-
Colorimetric (Cobaltinitrate)	-	-	-	-	317B
53. Residue--total, mg/L: Gravimetric, 103-105°C	160.3	209A	-	I-3750-84	-
54. Residue--filterable, mg/L: Gravimetric, 180°C	160.1	209B	-	I-1750-84	-

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Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
55. Residue--nonfilterable, (TSS), mg/L: Gravimetric, 103-105°C post washing of residue	160.2	209C	-	I-3765-84	-
56. Residue--settleable, mg/L: Volumetric (Imhoff cone) or gravimetric	160.5	209E	-	-	-
57. Residue--volatile, mg/L: Gravimetric, 550°C	160.4	209D	-	I-3753-84	-
58. Rhodium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	265.1	303A	-	-	-
Or AA furnace	265.2	304	-	-	-
59. Ruthenium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	267.1	303A	-	-	-
Or AA furnace	267.2	304	-	-	-
60. Selenium--Total,[3] mg/L: Digestion[3] followed by:					
AA furnace	270.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or AA gaseous hydride	270.3	303E	D3859-84(A)	I-3667-84	-
61. Silica--Dissolved, mg/L: 0.45 micron filtration followed by:					
Colorimetric, manual,	370.1	425C	D859-80(B)	I-1700-84	-
Automated (Molybdosilicate), or	-	-	-	I-2700-84	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
62. Silver--Total,[28] mg/L: Digestion[3] followed by:					
AA direct aspiration	272.1	303A or 303B	-	I-3720-84	33.089[2] and 37[8]
AA furnace, or	272.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]

Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page)				
	EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
63. Sodium--Total,[3] mg/L: Digestion[3] followed by:					
Atomic absorption	273.1	303A	-	I-3735-84	33.107[2]
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or flame photometric	-	325B	D1428-82(A)	-	-
64. Specific conductance, mhos/cm: Wheatstone bridge	120.1	205	D1125-82(A)	I-1780-84	33.002[2]
65. Sulfate (as SO_4), mg/L:					
Automated colorimetric (barium thoranilate),	375.1	-	-	I-2822-78	-
Gravimetric, or	375.3	426A or 426B	D516-82(A)	-	33.124[2]
Turbidimetric	375.4		D516-82(B) (15th Ed.)	-	426C[29]
66. Sulfide (as S), mg/L:					
Titrimetric (iodine) or	374.1	427D	-	I-3840-84	228A[30]
Colorimetric (methylene blue)	376.2	427C	-	-	-
67. Sulfite (as SO_3), mg/L: Titrimetric (iodine iodate)	377.1	428A	D1339-84(C)	-	-
68. Surfactants, mg/L: Colorimetric (methylene blue)	425.1	512B	D2330-82(A)	-	-
69. Temperature, °C: Thermometric	170.1	212	-	-	(31)
70. Thallium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	279.1	303A	-	-	-
AA furnace, or	279.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
71. Tin--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration or	282.1	303A	-	I-3850-78	-
AA furnace	282.2	304	-	-	-

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Table 9.1B (Continued)
List of Approved Inorganic Test Procedures

Parameter, Units, and Method	Reference (Method No. or Page) EPA 1979	Standard Methods 16th Ed.	ASTM	USGS[1]	Other
72. Titanium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration or	283.1	303C	-	-	-
AA furnace	283.2	304	-	-	-
73. Turbidity, NTU: Nephelometric	180.1	214A	D1889-81	I-3860-84	-
74. Vanadium--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	286.1	303C	-	-	-
AA furnace	286.2	304	-	-	-
Inductively coupled plasma	-	-	-	-	Method 200.7[4]
Or colorimetric (Gallic acid)	-	327B	D3373-84(A)	-	-
75. Zinc--Total,[3] mg/L: Digestion[3] followed by:					
AA direct aspiration	289.1	303A or 303B	D1691-84(C or D)	I-3900-84	33.089[2]
AA furnace	289.2	304		-	
Inductively coupled plasma, or	-	-	-	-	Method 200.7[4]
Colorimetric (Zincon or Dithizone)	-	328C	-	-	(32)

TABLE 9.1B NOTES

[1] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 85-495, 1986.

[2] "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 14th ed. (1985).

[3] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes," 1979. One (§4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (§4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all samples types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic 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 this vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations 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 instructions and/or cautions.

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Table 9.1B (Continued)

List of Approved Inorganic Test Procedures

NOTE: If the digestion procedure for direct aspiration or graphite furnace atomic absorption analysis 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 for dissolved 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 visibility 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.

(4) The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of 40 CFR Part 136 as published in the October 26, 1984, Federal Register.

(5) Manual distillation is not required if comparability data on representative 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.

(6) Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

(7) The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluid Sediments," USGS TWRI, Book 5, chapter A1 (1979).

(8) American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

(9) "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).

(10) The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

(11) Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data obtained using the nitrification inhibitor.

(12) OIC COD Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840.

(13) Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

(14) The back titration method will be used to resolve controversy.

Table 9.1B (Continued)

List of Approved Inorganic Test Procedures

(15) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, Massachusetts 02138.

(16) The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976.

(17) National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 253, December 1971.

(18) Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

(19) After the manual distillation is completed, the auto-analyzer 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, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

(20) Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon Auto-Analyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

(21) Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

(22) Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, Colorado 80537.

(23) Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3, p.4 (1972).

(24) Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

(25) Just prior to distillation, adjust the sulfuric acid preserved sample to pH 4 with 1 + 9 NaOH.

(26) The approved method is that 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 page 576-81 of the 14th edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedures.

Table 9.1B (Continued)

List of Approved Inorganic Test Procedures

[27] 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.

[28] Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where 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 a 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 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[29] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th. Edition.

[30] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 13th Edition.

[31] Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature-Influential Factors, Field Measurement and Data Presentation," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 1, Chapter D1, 1975.

[32] Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, Colorado 80537.

Table 9.1C
List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7] GC	GC/MS	HPLC	Other
1. Acenaphthene	610	625, 1625	610	
2. Acenaphthylene	610	625, 1625	610	
3. Acrolein	603	[4]624, 1624	-	
4. Acrylonitrile	603	[4]624, 1624	-	
5. Anthracene	610	625, 1625	610	
6. Benzene	602	624, 1624	-	
7. Benzidine	-	[5]625, 1625	605	Note 3, p. 1
8. Benzo(a)anthracene	610	625, 1625	610	
9. Benzo(a)pyrene	610	625, 1625	610	
10. Benzo(b)fluoranthene	610	625, 1625	610	
11. Benzo(ghi)perylene	610	625, 1625	610	
12. Benzo(k)fluoranthene	610	625, 1625	610	
13. Benzyl Chloride	-	- -	-	Note 3, p. 130; Note 6, p. S102
14. Benzyl Butyl Phthalate	606	625, 1625	-	
15. Bis(2-chloroethoxy) methane	611	625, 1625	-	
16. Bis(2-chloroethyl) ether	611	625, 1625	-	
17. Bis(2-ethylhexyl) phthalate	606	625, 1625	-	
18. Bromodichloromethane	601	624, 1624	-	
19. Bromoform	601	624, 1624	-	
20. Bromomethane	601	624, 1624	-	
21. 4-Bromophenylphenyl ether	611	625, 1625	-	

Table 9.1C (Continued)

List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7] GC	GC/MS	HPLC	Other
22. Carbon tetrachloride	601	624, 1624	-	Note 3, p. 130
23. 4-Chloro-3-methylphenol	604	625, 1625	-	
24. Chlorobenzene	601, 602	624, 1624	-	Note 3, p. 130
25. Chloroethane	601	624, 1624	-	
26. 2-Chloroethylvinyl ether	601	624, 1624	-	
27. Chloroform	601	624, 1624	-	Note 3, p. 130
28. Chloromethane	601	624, 1624	-	
29. 2-Chloronaphthalene	612	625, 1625	-	
30. 2-Chlorophenol	604	625, 1625	-	
31. 4-Chlorophenylphenyl ether	611	625, 1625	-	
32. Chrysene	610	625, 1625	610	
33. Dibenzo(a,h)anthracene	610	625, 1625	610	
34. Dibromochloromethane	601	624, 1624	-	
35. 1,2-Dichlorobenzene	601, 602, 612	624, 625, 1625	-	
36. 1,3-Dichlorobenzene	601, 602, 612	624, 625, 1625	-	
37. 1,4-Dichlorobenzene	601, 602, 612	625, 1624, 1625	-	
38. 3,3'-Dichlorobenzidine	-	625, 1625	605	
39. Dichlorodifluoromethane	601	- -	-	
40. 1,1-Dichloroethane	601	624, 1624	-	

Table 9.1C (Continued)

List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7] GC	GC/MS	HPLC	Other
41. 1,2-Dichloroethane	601	624, 1624	-	
42. 1,1-Dichloroethene	601	624, 1624	-	
43. trans-1,2-Dichloroethene	601	624, 1624	-	
44. 2,4-Dichlorophenol	604	625, 1625	-	
45. 1,2-Dichloropropane	601	624, 1624	-	
46. cis-1,3-Dichloropropene	601	624, 1624	-	
47. trans-1,3-Dichloropropene	601	624, 1624	-	
48. Diethyl phthalate	606	625, 1625	-	
49. 2,4-Dimethylphenol	604	625, 1625	-	
50. Dimethyl phthalate	606	625, 1625	-	
51. Di-n-butyl phthalate	606	625, 1625	-	
52. Di-n-octyl phthalate	606	625, 1625	-	
53. 2,4-Dinitrophenol	604	625, 1625	-	
54. 2,4-Dinitrotoluene	609	625, 1625	-	
55. 2,6-Dinitrotoluene	609	625, 1625	-	
56. Epichlorohydrin	-	- -	-	Note 3, p. 130; Note 6, p. S102
57. Ethylbenzene	602	624, 1624	-	
58. Fluoranthene	610	625, 1625	610	

Table 9.1C (Continued)

List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7] GC	GC/MS	HPLC	Other
59. Fluorene	610	625, 1625	610	
60. Hexachlorobenzene	612	625, 1625	-	
61. Hexachlorobutadiene	612	625, 1625	-	
62. Hexachlorocyclopentadiene	612	[5]625, 1625	-	
63. Hexachloroethane	612	625, 1625	-	
64. Ideno(1,2,3-cd)pyrene	610	625, 1625	610	
65. Isophorone	609	625, 1625	-	
66. Methylene Chloride	601	624, 1624	-	Note 3, p. 130
67. 2-Methyl-4,6-Dinitrophenol	604	625, 1625	-	
68. Naphthalene	610	625, 1625	-	
69. Nitrobenzene	609	625, 1625	-	
70. 2-Nitrophenol	604	625, 1625	-	
71. 4-Nitrophenol	604	625, 1625	-	
72. N-Nitrosodimethylamine	607	625, 1625	-	
73. N-Nitrosodi-n-propylamine	607	[5]625, 1625	-	
74. N-Nitrosodiphenylamine	607	[5]625, 1625	-	
75. 2,2-oxybis(1-chloropropane)	611	625, 1625	-	
76. PCB-1016	608	625	-	Note 3, p. 43
77. PCB-1221	608	625	-	Note 3, p. 43

Table 9.1C (Continued)

List of Approved Test Procedures for Non-Pesticide Organic Compounds

Parameter[1]	EPA Method Number[2,7] GC	GC/MS	HPLC	Other
78. PCB-1232	608	625	-	Note 3, p. 43
79. PCB-1242	608	625	-	Note 3, p. 43
80. PCB-1248	608	625	-	Note 3, p. 43
81. PCB-1254	608	625	-	Note 3, p. 43
82. PCB-1260	608	625	-	Note 3, p. 43
83. Pentachlorophenol	604	625, 1625	-	Note 3, p. 140
84. Phenanthrene	610	625, 1625	610	
85. Phenol	604	625, 1625	-	
86. Pyrene	610	625, 1625	610	
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	-	[5a]613	-	
88. 1,1,2,2-Tetrachloroethane	601	624, 1624	-	Note 3, p. 130
89. Tetrachloroethene	601	624, 1624	-	Note 3, p. 130
90. Toluene	602	624, 1624	-	
91. 1,2,4-Trichlorobenzene	612	625, 1625	-	Note 3, p. 130
92. 1,1,1-Trichloroethane	601	624, 1624	-	
93. 1,1,2-Trichloroethane	601	624, 1624	-	Note 3, p. 130
94. Trichloroethene	601	624, 1624	-	
95. Trichlorofluoromethane	601	624	-	
96. 2,4,6-Trichlorophenol	604	625, 1625	-	
97. Vinyl Chloride	601	624, 1624	-	

Table 9.1C Notes

(1) All parameters are expressed in micrograms per liter ()g/L).

(2) The full text of Methods 601-613, 624, 625, 1624, and 1625, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of 40 CFR 136 as published in the October 26, 1984 Federal Register. 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 40 CFR 136 as published in the October 26, 1984 Federal Register.

(3) "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.

(4) Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, Method 603 or Method 1624 are preferred methods for these compounds.

(5) Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625,are preferred methods for these compounds.

(5a) 625, Screening only.

(6) "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).

(7) Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601-613, 624, 625, 1624, and 1625 (See Appendix A of 40 CFR 136 as published in the October 26, 1984 Federal Register) in accordance with procedures in Section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for Methods 1624, and 1625) 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 unspiked sample are suspect and cannot be reported.

Table 9.1D
List of Approved Test Procedures for Pesticides[1]

Parameter (µg/L)	Method	EPA[2,7]	Standard Methods 15th Ed.	ASTM	OTHER
1. Aldrin	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
2. Ametryn	GC	-	-	-	Note 3, p.83; Note 6, p.S68
3. Aminocarb	TLC	-	-	-	Note 3, p.94; Note 6, p.S16
4. Atraton	GC	-	-	-	Note 3, P.83; Note 6, P. S68
5. Atrazine	GC	-	-	-	Note 3, p.83; Note 6, p. S68
6. Azinphos methyl	GC	-	-	-	Note 3, p.25; Note 6, p.S51
7. Barban	TLC	-	-	-	Note 3, p.104; Note 6, p. S64
8. a-BHC	GC	608	509A	D3086	Note 3, p.7
	GC/MS	[5]625	-	-	
9. b-BHC	GC	608	-	D3086	
	GC/MS	625	-	-	
10. w-BHC	GC	608	-	D3086	
	GC/MS	[5]625	-	-	
11. g-BHC (Lindane)	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
12. Captan	GC	-	509A	-	Note 3, p.7
13. Carbaryl	TLC	-	-	-	Note 3, p.94; Note 6, p.S60
14. Carbophenothion	GC	-	-	-	Note 4, p.30; Note 6, p.S73
15. Chlordane	GC	608	509A	D3086	Note 3, p.7
	GC/MS	625	-	-	
16. Chloroprapham	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
17. 2,4-D	GC	-	509B	-	Note 3, p.115; Note 4, p.35
18. 4,4'-DDD	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
19. 4,4'-DDE	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
20. 4,4'-DDT	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
21. Demeton-O	GC	-	-	-	Note 3, p.25; Note 6, p.S51
22. Dementon-S	GC	-	-	-	Note 3, p.25; Note 6, p. S51
23. Diazinon	GC	-	-	-	Note 3, p.25; Note 4, p.30; Note 6, p. S51

Table 9.1D (Continued)
List of Approved Test Procedures for Pesticides[1]

Parameter (µg/L)	Method	EPA[2,7]	Standard Methods 15th Ed.	ASTM	OTHER
24. Dicamba	GC	-	-	-	Note 3, p.115
25. Dichlofenthion	GC	-	-	-	Note 4, p.30; Note 6, p.S73
26. Dichloran	GC	-	509A	-	Note 3, p.7
27. Dicofol	GC	-	-	D3086	
28. Dieldrin	GC	608	509A	-	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
29. Dioxathion	GC	-	-	-	Note 4, p.30; Note 6, p.S73
30. Disulfoton	GC	-	-	-	Note 3, p. ; Note 6, p. S51
31. Diuron	TLC	-	-	-	Note 3, p. 104; Note 6, p. S64
32. Endosulfan I	GC	608	509A	D3086	Note 3, p.7
	GC/MS	[5]625	-	-	
33. Endosulfan II	GC	608	509A	D3086	Note 3, p.7
	GC/MS	[5]625	-	-	
34. Endosulfan sulfate	GC	608	-	-	
	GC/MS	625	-	-	
35. Endrin	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	[5]625	-	-	
36. Endrin aldehyde	GC	608	-	-	
	GC/MS	625	-	-	
37. Ethion	GC	-	-	-	Note 4, p.30; Note 6, p.S73
38. Fenuron	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
39. Fenuron-TCA	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
40. Heptachlor	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
41. Heptachlor epoxide	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30; Note 6, p. S73
42. Isodrin	GC/MS	625	-	-	
43. Linuron	GC	-	-	-	Note 4, p.30; Note 6, p.S73
44. Malathion	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
45. Methiocarb	GC	-	509A	-	Note 3, p.25; Note 4, p.30; Note 6, p.S51
46. Methoxychlor	TLC	-	-	-	Note 3, p.94; Note 6, p.S60
	GC	-	509A	D3086	Note 3, p.7; Note 4, p.30

Table 9.1D (Continued)
List of Approved Test Procedures for Pesticides[1]

Parameter (µg/L)	Method	EPA[2,7]	Standard Methods 15th Ed.	ASTM	OTHER
47. Mexacarbate	TLC	-	-	-	Note 3, p.94; Note 6, p.S60
48. Mirex	GC	-	509A	-	Note 3, p.7
49. Monuron	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
50. Monuron-TCA	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
51. Neburon	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
52. Parathion methyl	GC	-	509A	-	Note 3, p.25; Note 4, p.30
53. Parathion ethyl	GC	-	509A	-	Note 3, p.25
54. PCNB	GC	-	509A	-	Note 3, p.7
55. Perthane	GC	-	-	D3086	
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
60. Propoxur	TLC	-	-	-	Note 3, p.94; Note 6, p.S60
61. Secbumeton	TLC	-	-	-	Note 3, p.83; Note 6, p.S68
62. Siduron	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
63. Simazine	GC	-	-	-	Note 3, p.83; Note 6, p.S68
64. Strobane	GC	-	509A	-	Note 3, p.7
65. Swep	TLC	-	-	-	Note 3, p.104; Note 6, p.S64
66. 2,4,5-T	GC	-	509B	-	Note 3, p.115; Note 4, p.35
67. 2,4,5-TP (Silvex)	GC	-	509B	-	Note 3, p.115
68. Terbuthylazine	GC	-	-	-	Note 3, p.83; Note 6, p.S68
69. Toxaphene	GC	608	509A	D3086	Note 3, p.7; Note 4, p.30
	GC/MS	625	-	-	
70. Trifluralin	GC	-	509A	-	Note 3, p.7

Table 9.1D Notes

(1) Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 9.1C, where entries are listed by chemical name.

(2) The full text of methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of 40 CFR 136 as published in the October 26, 1984 Federal Register. The standardized test procedure to be used to determine the method detection limit (MDL) for these procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of 40 CFR 136 as published in the October 26, 1984 Federal Register.

(3) "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.

(4) "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3 (1972).

(5) The method may be extended to include a-BHC, w-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

(6) "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).

(7) Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of 40 CFR 136 as published in the October 26, 1984 Federal Register) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 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 unspiked sample are suspect and cannot be reported.

Table 9.1E
List of Approved Radiological Test Procedures

Parameter and Units	Methods	Reference (Method No. or Page) EPA[1]	Standard Methods 16th Ed.	ASTM	USGS[2]
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900.0	703	D1943-81	pp. 75 and 78[3]
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	703	D1943-81	p. 79
3. Beta-Total, pCi per liter	Proportional counter	900.0	703	D1890-81	pp. 75 and 78[3]
4. Beta-Counting error, pCi per liter	Proportional counter	Appendix B	703	D1890-81	p. 79
5. (a) Radium-Total, pCi per liter	Proportional counter	903.0	705	D2460-70	
(b) ^{226}Ra, pCi per liter	Scintillation counter	903.1	706	D3454-79	p. 81
6. Radium -228, pCi per liter	Beta counter	9320[4]	-	-	-
7. Gross Alpha and Gross Beta	Proportional counter	9310[4]	-	-	-
8. Alpha-Emitting Radium Isotopes	Proportional counter	9315[4]	-	-	-

Table 9.1E Notes

(1) "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980) update), U.S. Environmental Protection Agency, August 1980.

(2) Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).

(3) The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

(4) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA, September 1986.

Table 9.1F References and Sources

Table	Parameters	References and Sources
9.1A - EPA	1-5	"Microbiological Methods for Monitoring the Environment, Water and Wastes," United States Environmental Protection Agency, EPA-600/8-78-017, 1978. ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268
9.1A - Standard Methods	1-5	Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 16th Edition, 1986. Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, DC 20036.
9.1B - Standard Methods	1-46, 48-75	
9.1D - Standard Methods	1, 8, 11, 12, 15, 17-20, 26, 28, 32, 33, 35, 40, 41, 44, 46, 48, 52-54, 64, 66, 67, 69, 70	
9.1E - Standard Methods	1-5	
9.1C - Other (Standard Methods Supplement)	13, 56	"Selected Analytical Methods Approved and Cited by the the United States Environmental Protection Agency," Supplement to the 15th Edition of Standard Methods for the Examination of Water and Wastewater (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036.
9.1D - Other (Standard Methods Supplement)	2-7, 13, 14, 16, 21-23, 25, 29-31, 37, 38, 39, 41-45, 47, 49, 50, 51, 56-63, 65, 68	
9.1A - U.S. Geological Survey (USGS)	1, 2, 3, 5	"Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples," edited by P. E. Greeson, T. A. Ehlke, G. A. Irwin, B. W. Lium, and K. V. Slack: U.S. Geological Survey, Techniques of Water-Resources Investigation (USGS TWRI), Book 5, Chapter A4 (1977). Revised edition, 332 pages. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Government Printing Office.

Table 9.1F (Continued)

Table	Parameters	References and Sources
9.1B - EPA	1-13, 15-48, 50-75	"Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, United States Environmental Protection Agency, March, 1979. Available from: ORD Publications, CERI, U.S. Enviornmental Protection Agency, Cincinnati, Ohio 45268.
9.1B - ASTM	1, 2, 4, 6, 8, 11-13, 15-17, 19, 20, 22-25, 27, 28, 30-35, 37-40, 42-44, 46, 48, 50, 52, 60, 61, 63-65, 67, 68, 73-75	"Annual Book of Standards, Part 31, Water," American Society for Testing and Materials, (1979-1984 as noted in Table 9.1B). Available from: American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.
9.1D - ASTM	1, 8-11, 15, 18-20, 27, 32, 33, 35, 40, 41, 46, 55, 69	
9.1E - ASTM	1-5	
9.1B - USGS	2, 3, 4, 6-13, 15, 16, 18-23, 25, 27, 28, 30-40, 44, 46, 50, 52-55, 57, 60-66, 71, 73, 75	"Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, Open File Report, 85-495 (1986). Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Government Printing Office.
9.1B - Other (AOAC)	2, 4, 9, 12, 15, 16, 19, 22, 30-35, 38, 42-44, 46, 50, 52, 62-65, 75	Official Methods of Analysis of the Association of Official Analytical Chemists, methods manual, 14th Edition (1985). The Association of Official Analytical Chemists, 1111 N. 19th St., Suite 210, Arlington, VA 22209.
9.1B - Other (ANSI)	9, 12, 15, 20, 22, 23, 38, 62	"American National Standard on Photographic Processing Effluents," April 2, 1975. American National Standards Institute, 1430 Broadway, New York, New York 10018.

Table 9-1F. (Continued)

Table	Parameters	References and Sources
9.1B - Other (EPA)	3, 5-8, 10, 12, 13, 19, 20, 22, 27, 30, 32-34, 36, 37, 52, 60-63, 70, 74, 75	The full text of the inductively coupled plasma optical emission spectroscopic test procedure, Method 200.7, is found in Appendix C of 40 CFR Part 136 as published in the October 26, 1984 Federal Register.
9.1B - Other	21	"An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color," NCASI Technical Bulletin No. 253, December, 1971. National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, New York.
9.1B - Other	4	Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon AutoAnayzer II. Technicon Industrial Systems, Tarrytown, New York 10591.
9.1B - Other	15	Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	15	OIC Chemical Oxygen Demand Method. Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840.
9.1B - Other	17	ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Orion Research Incorporated, 840 Memorial Drive, Cambridge, Massachusetts 02138.

Table 9-1F (Continued)

Table	Parameters	References and Sources
9.1B - Other	22	Bicinchoninate Method for Copper, Method 8506, Hach Handbook of Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	28	Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon AutoAnalyzer II. Technicon Industrial Systems, Tarrytown, New York 10591.
9.1B - Other	30	1,10-Phenanthroline Method for Iron, Hach Method 8008. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	34	Periodate Oxidation Method for Managanese, Method 8034, Hach Handbook for Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	40	Nitrite Nitrogen, Hach Method 8507. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	75	Zincon Method for Zinc, Method 8009, Hach Handbook for Water Analysis, 1979. Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.
9.1B - Other	49	"Direct Determination of Elemental Phosphorous by Gas-Liquid Chromatography," by R. F. Addison and R. G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421-426, 1970. Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.

Table 9-1F (Continued)

Table	Parameters	References and Sources
9.1B - Other (USGS)	69	"Water Temperature-Influential Factors, Field Measurement, and Data Presentation," by H. H. Stevens, Jr., J. Ficke, and G. F. Smoot: USGS-TWRI Book 1, Chapter D1, 1975, 65 pages. U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202.
9.1D - Other (USGS)	1, 11, 14, 17-20, 23, 25, 28, 29, 35, 37, 40-42, 44, 46, 52, 66, 69	"Methods for Analysis of Organic Substances in Water," by D. F. Goerlitz and Eugene Brown: USGS-TWRI Book 5, Chapter A3, 1972, 40 pages. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202.
9.1C - EPA	1-12, 14-55, 57-97	The full texts of Methods 601-613, 624, 625, 1624 and 1625 are found in Appendix A of 40 CFR Part 136 as published in the October 26, 1984 Federal Register.
9.1D - EPA	1, 8-11, 15, 18, 19, 20, 28, 32-36, 40, 41, 69	
9.1C - Other (EPA)	7, 13, 22, 24, 27, 56, 66, 76-83, 88, 89, 91, 93	"Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,"
9.1D - Other (EPA)	1-8,11-13, 15-24, 26, 28, 30-33, 35, 38-41, 43-54, 56-70	Environmental Monitoring and Support Laboratory, United States Environmental Protection Agency, Cincinnati, Ohio 1978. ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.
9.1E - EPA	1-5	"Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 Update), United States Environmental Protection Agency, 1980. ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.
9.1E - USGS	1-5	"Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," by M. J. Fishman and Eugene Brown; U.S. Geological Survey Open File Report 76-77 (1976). U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202.

Table 9.2
Method Summary for Organic Compounds in
Transformer Fluid, Waste Oils, Soil, Sediment, Solid and Liquid Wastes

Parameter	Method No.	Preferred Equipment	Detection Limit	Reference	Sample Matrix
Polychlorinated Biphenyls (PCBs)	-	Gas Chromatography	1.0 mg/kg	EPA, 1981[1]	Transformer Fluid and Waste Oil
Polychlorinated Biphenyls (PCBs)	-	Gas Chromatography	0.1 mg/kg	EPA, 1981[2]	Solid, Sediment
Organochlorine Pesticides	-	Gas Chromatography	0.002 mg/kg	EPA, 1981[2]	Solid, Sediment
Purgeable Organics	-	Gas Chromatography/ Mass Spectrometry	0.1 mg/kg	EPA, 1981[2]	Solid, Sediment, Tissue
Base-Neutrals and Acid Extractables	-	Gas Chromatography/ Mass Spectrometry	1.0 mg/kg	EPA, 1981[2]	Solid, Sediment, Tissue
Halogenated Volatile Organics	8010	Gas Chromatography	0.03 to 0.52)g/l[a]	EPA, 1986[3]	Solid Waste
Nonhalogenated Volatile Organics	8015	Gas Chromatography	Not cited	EPA, 1986[3]	Solid Waste
Aromatic Volatile Organics	8020	Gas Chromatography	0.2 to 0.4)g/l[a]	EPA, 1986[3]	Solid Waste
Acrolein, Acrylonitrile, and Acetonitrile	8030	Gas Chromatography	0.5 to 0.7)g/l[a]	EPA, 1986[3]	Solid Waste
Phenols	8040	Gas Chromatography	0.14 to 16)g/l[b]	EPA, 1986[3]	Solid Waste
Phthalate Esters	8060	Gas Chromatography	0.29 to 31)g/l[b]	EPA, 1986[3]	Solid Waste
Organochlorine Pesticides and PCBs	8080	Gas Chromatography	0.002 to 0.24[b]	EPA, 1986[3]	Solid Waste

Table 9.2 (Continued)
Method Summary for Organic Compounds in
Transformer Fluid, Waste Oils, Soil, Sediment, Solid and Liquid Wastes

Parameter	Method No.	Preferred Equipment	Detection Limit	Reference	Sample Matrix
Nitroaromatics and Cyclic Ketones	8090	Gas Chromatography	0.01 to 15.7)g/l[b]	EPA, 1986[3]	Solid Waste
Polynuclear Aromatic Hydrocarbons	8100	High Performance Liquid Chromatography	Not cited	EPA, 1986[3]	Solid Waste
Chlorinated Hydrocarbons	8120	Gas Chromatography	0.03 to 1.34)g/l[b]	EPA, 1986[3]	Solid Waste
Organophosphorous Pesticides	8140	Gas Chromatography	0.03 to 5.0)g/l[b]	EPA, 1986[3]	Solid Waste
Chlorinated Herbicides	8150	Gas Chromatography	0.07 to 249)g/l[b]	EPA, 1986[3]	Solid Waste
Volatile Organics	8240	Gas Chromatography/ Mass Spectrometry	(c)	EPA, 1986[3]	Solid Waste
Semivolatile Organics; Packed Column Technique	8250	Gas Chromatography/ Mass Spectrometry	(d)	EPA, 1986[3]	Solid Waste
Semivolatile Organics Capillary Column Technique	8270	Gas Chromatography/ Mass Spectrometry	(d)(e)	EPA, 1986[3]	Solid Waste
Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans	8280	Gas Chromatography/ Mass Spectrometry	Not cited	EPA, 1986[3]	Solid Waste
Polynuclear Aromatic Hydrocarbons	8310	HPLC	0.013 to 2.3)g/l[b]		Solid Waste

Table 9.2 (Continued)

(1) "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils," Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, U. S. Environmental Protection Agency, Cincinnati, Ohio, April, 1981.

(2) "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue," Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, U. S. Environmental Protection Agency, Cincinnati, Ohio, April, 1981.

(3) "Test Methods for Evaluating solid Waste, Physical/Chemical Methods," Waste Characterization Branch, Office of Solid Waste, U. S. Environmental Protection Agency, Washington, D.C., July 1982.

(a) Practical quantitation limits, for various matrixes are provided for guidance and may not always be achievable. Methods detection unit listed in column of table is multipled by the following factors to determine PQL(s):

Ground water	10
Low-level soil	10
Water miscible liquid waste	500
High level soil and sludge	1250
Non-water miscible waste	1250

(b) Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable. Multiply the listed method detection limit times the following factors for these matrixes:

Ground water	10
Low level soil by sonication with GPC cleanup	670
High level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

(c) PQL of Method 8240 for an individual compound is approximately 5)g/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5)g/l for ground water. PQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector. Sample PQLs are highly matrix dependent. The PQLs listed are provided for guidance and may not always be achievable. Multiply the listed method detection limit times the following factors for these matrixes:

Water miscible liquid waste	50
High-level soil and sludges	125
Non-water miscible waste	500

Table 9.2 (Continued)

(d) The PQL of Method 8250 for determining an individual compound is approximately 1 mg/kg (net weight) for soil/sediment samples, 1-200 mg/kg for wastes (dependent on matrix and method of preparation), and 10)g/l for ground water samples. PQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector. Sample PQLs are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable. Multiply the listed method detection limit times the following factors for these matrixes:

Ground water	10
Low-level soil by sonication with GPC cleanup	670
High-level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

(e) The PQLs listed are highly matrix dependent. The PQLs listed herein are provided for guidance and may not always be achievable. Multiply the listed method detection limit times the following factors for these matrixes:

Medium level soil and sludges by sonicator	7.5
Non-water miscible waste	75

Table 9.3
Method Summary for Trace Metals by Atomic Absorption Spectrophotometry and Inductively Coupled Argon Plasma Emission Spectrometry

Parameter	Direct Aspiration Detection Limit (mg/l)	Direct Aspiration Reference	Graphite Furnace Detection Limit (mg/l)	Graphite Furnace Reference	ICAP[1],[8] Detection Limit (mg/l)
Trace Metals Analysis					
Aluminum	0.1	EPA 202.1[2]; 7020[7]	0.005	EPA 202.2	0.05, 0.045[8]
Antimony	0.2	EPA 204.1; 7040	0.005 0.003	EPA 204.2 7041	0.03, 0.032
Arsenic	1.0	EPA 206.2	0.001	EPA 206.2; 7060	0.05, 0.053
Barium	0.1	EPA 208.1; 7080	0.002	EPA 208.2	0.002, 0.002
Beryllium	0.01 0.005	EPA 210.1 7090	0.001 0.0002	EPA 210.2 7091	0.0003, 0.0003
Boron	0.1	SM 404 A[4]; EPA 212.3			0.005, 0.005
Cadmium	0.005	EPA 213.1; 7130	0.0001	EPA 213.2; 7131	0.004, 0.004
Calcium	0.01	EPA 215.1; 7140	-	-	0.01, 0.01
Chromium	0.05	EPA 218.1; 7190	0.005 0.001	EPA 218.2 7191	0.007, 0.007
Cobalt	0.1 0.05	EPA 219.1 7200	0.001	EPA 219.2; 7201	0.007, 0.007
Copper	0.02	EPA 220.1; 7210	0.001	EPA 220.2	0.006, 0.006
Iron	0.03	EPA 236.1; 7380	0.005	EPA 236.2	0.007, 0.007
Lead	0.1	EPA 239.1; 7420	0.005 0.001	EPA 239.2 7421	0.040, 0.042
Lithium	0.01	Perkin-Elmer[3]	-	-	-
Magnesium	0.001	EPA 242.1; 7450	-	-	0.03, 0.30
Manganese	0.01	EPA 243.1; 7460	0.0005	EPA 243.2	0.002, 0.002
Mercury	0.5	Perkin-Elmer[3]	0.0002[6]	EPA 245.1[6]; 7471[7]	-

Table 9.3 (Continued)

Method Summary for Trace Metals by Atomic Absorption Spectrophotometry and Inductively Coupled Argon Plasma Emission Spectrometry

Parameter	Direct Aspiration Detection Limit (mg/l)	Direct Aspiration Reference	Graphite Furnace Detection Limit (mg/l)	Graphite Furnace Reference	ICAP[1],[8] Detection Limit (mg/l)
Molybdenum	0.1	EPA 246.1; 7480	0.005 0.001	EPA 246.2 7481	0.008, 0.008
Nickel	0.04	EPA 249.1; 7520	0.001	EPA 249.2	0.015, 0.015
Osmium	1.0	7550			
Potassium	0.01	EPA 258.1; 7610	-	-	-
Selenium	0.1	Perkin-Elmer[3]	0.002	EPA 270.2; 7240	0.08, 0.075
Silicon	0.1	SM 303C[4]	-	-	0.005, 0.058
Silver	0.01	EPA 272.1; 7760	0.001	EPA 272.2	0.007, 0.007
Sodium	0.005 0.002	EPA 273.1 7770	-	-	0.03, 0.029
Strontium	0.1	SM 303A[4]	-	-	-
Thallium	0.1	EPA 279.1; 7840	0.005 0.001	EPA 279.2 7841	0.04, 0.040
Tin	1.0 0.8	EPA 282.1 7870	0.005	EPA 282.2	-
Titanium	0.5	EPA 283.1	0.005	EPA 283.2	-
Tungsten	10	Perkin-Elmer[3]	-	-	-
Uranium	-	-	0.1	Perkin-Elmer[5]	-
Vanadium	0.2	EPA 286.1; 7910	0.005 0.004	EPA 286.2 7911	0.008, 0.008
Zirconium	10	Perkin Elmer[3]	-	-	-
Zinc	0.01 0.005	EPA 289.1 7950	0.001	EPA 289.2	0.002, 0.002

Table 9.3 (Continued)

(1) Samples to be analyzed according to EPA Method 200.7 as per manufacturer's specifications.

(2) "Methods for Chemical Analysis of Water and Wastes," Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, March, 1979. The numerical designation following the EPA prefix indicates the pertinent method number in the above reference.

(3) "Analytical Methods for Atomic Absorption Spectrophotometry," Perkin-Elmer Corporation, Norwalk, Connecticut, September 1976, or as prescribed by other instrument manufacturers.

(4) Standard Methods for the Examination of Water and Wastewater, 16th edition, American Public Health Association, American Waterworks Association, and the Water Pollution Control Federation, 1986. The numerical designation following the SM prefix indicates the pertinent method number in the above reference. The Standard Methods reference for boron is curcumin colorimetric procedure. All other references to Standard Methods are atomic absorption spectrophotometry procedures.

(5) "Analytical Methods for Atomic Absorption Spectrophotometry Using the HGA Graphite Furnace," Perkin-Elmer Corporation, Norwalk, Connecticut, 1978.

(6) The indicated analytical procedure for mercury is the cold vapor technique described in the referenced EPA procedure.

(7),(8) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, USEPA, September 1986.

Table 9.4
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Acidity				
- Available	Titrimetric	Buret, pH meter	1 mg/l	SM 402[1]
- Total				EPA 305.1[3]; SM 402[1]
• Alkalinity	Titrimetric	Buret, pH meter	1 mg/l	SM 403[1], EPA 310.1[3]
- Phenolphthalein				
- Total				
- Bicarbonate				
- Carbonate				
- Hydroxide				
• Ammonia	Colorimetric	Spectrometer	0.1 mg/l	SM 417B[1]
	Titrimetric	Buret	1.0 mg/l	SM 417D[1]; 350.2[3]
	Specific Ion Electrode	Specific Ion Meter	0.1 mg/l	EPA 350.3[3]; SM 417E or F[1]
• BOD	Electrometric	Dissolved Oxygen Meter/ Electrode	1.0 mg/l	SM 507[1], 405.1[3]
• Bromide	Colorimetric	UV/VIS Spectrometer	1.0 mg/l	320.1[3]
• Carbon				
- Total	Combustion, Infrared	TOC Analyzer	1 mg/l	EPA 415.1[3]
- Inorganic	Combustion, Infrared	TOC Analyzer	1 mg/l	EPA 415.1[3]
- TOC	Combustion, Infrared	TOC Analyzer	1 mg/l	EPA 415.1[3]
• Carbon Dioxide (CO_2)				
- Free	Titrimetric	Buret/pH Meter	1 mg/l	SM 406B[1]
- Total	Calculation			SM 406C[1]
• Chloride	Titrimetric	Buret/Indicator	0.5 mg/l	EPA 325.3[3]; SM 407[1]

Table 9.4 (Continued)
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
(Mercuric Nitrate)				
• Chlorine Demand	Colorimetric	UV/VIS Spectrometer	1 mg/l	SM 409A[1]
• Chlorine Residual	Colorimetric	UV/VIS Spectrometer	1 mg/l	SM 408E[1]; EPA 330.5[3]
• Chromium (Hexavalent)	Colorimetric	UV/VIS Spectrometer	0.1 mg/l	SM 312B
• COD	Dichromate Reflux/ Titration	Buret/Indicator	1 mg/l	SM 508A[1];410.1[3]
• Color				
- Apparent	Dominant Wavelength	UV/VIS Spectrometer	-	SM 204B[1]; EPA 110.3[3]
- True				SM 204B[1]
• Cyanate (CNO)*	Specific Ion Electrode	Specific Ion Analyzer	0.1 mg/l	SM 412J[1]
• Cyanide				
- Free	Distillation/Colorimetric	UV/VIS Spectrometer	0.02 mg/l	EPA 335.2[3]; SM 412D[1]
- Total	Distillation/Colorimetric	UV/VIS Spectrometer		EPA 335.2[3]; SM 412D[1]
- Amenable and Total	Distillation/Colorimetric	UV/VIS Spectrometer		EPA 335.1[3]; SM 412F[1]
• Eh (Redox potential)	Electrometric	Specific Ion Meter	1 mV	ASTM 31-D1498
• Fluoride				
- Electrode (CDTA)	Specific Ion Electrode	Specific Ion Meter	0.1 mg/l	EPA 340.2[3]; SM 413B[1]
- Distillation				EPA 340.1[3]; SM 413C[1]
• Halogen (Total Organic)	Combustion/Electrolytic	TOX Analyzer	0.01 mg/l	EPA 450.1 Interim[3]
(TOX)	Conductivity Detector			
• Hardness				
- Total	Titrimetric	Buret/Indicator	1.0 mg/l	EPA 130.2[3]; SM 314B[1]
- Calcium			1.0 mg/l	ASTM 31-D511
- Magnesium			1.0 mg/l	ASTM 31-D511

Table 9.4 (Continued)
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Iodide	Colorimetric	UV/VIS Spectrometer	0.1 mg/l	EPA 345.1[3]
• Iron	Specific Ion	Specific Ion Meter	1.0 mg/l	-
- Ferrous	Colorimetric	UV/VIS Spectrometer	0.1 mg/l	SM 315.B[1]
- Ferric	Spectrometric	AAS	0.1 mg/l	SM 315A[1]
• Nitrate				
- Electrode	Specific Ion Electrode	Specific Ion Meter	1.0 mg/l	SM 418B, 419[1]
- Colorimetric	Brucine Sulfate	UV/VIS Spectrometer	0.1 mg/l	EPA 352.1[3]
• Nitrite	Colorimetric	UV/VIS Spectrometer	0.05 mg/l	EPA 354.1[3]
• Nitrogen				
- Organic (Kjeldahl-Ammonia)	Digestion/Distillation/ Specific Ion	Digestion Rack Specific Ion Meter	1.0 mg/l	SM 420[1]; EPA 351.3[3]
- Kjeldahl	Digestion/Distillation/ Specific Ion	Digestion Rack Specific Ion Meter	0.1 mg/l	EPA 351.3[3], SM 417E or F[1] EPA 351.3[3], SM 420[1]
• Oil and Grease	Titrimetric	Buret	0.1 mg/l	EPA 351.3[3]; SM 417D[1]
- Gravimetric	Extraction-Gravimetric	Separatory Funnel/Balance	0.5 mg/l	EPA 413.1[3]; SM 503A[1]
• Oxygen, Dissolved	Elecrometric	Dissolved Oxygen Meter	0.1 mg/l	EPA 360.1[3]; SM 421F[1]
Phenolics - Distillation/Extraction	Extraction/Colorimetric	UV/VIS Spectrometer	0.01 mg/l	EPA 420.1; EPA 420.2[3]
• Phosphorus				
- Total (Persulfate)	Persulfate Digestion/ Colorimetric	UV/VIS Spectrometer	0.01 mg/l	SM 424C (III); F or G[1]

Table 9.4 (Continued)
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Residue				
- Filterable, Dissolved (TDS)	Filtration/Gravimetric	Analytical Balance	1.0 mg/l	EPA 160.1[3]; SM 209B[1]
- Nonfilterable, Suspended (TSS)	Filtration/Gravimetric	Analytical Balance	1.0 mg/l	EPA 160.2[3]; SM 209C[1]
- Total (TS)	Gravimetric	Analytical Balance	1.0 mg/l	EPA 160.3[3]; SM 209A[1]
- Settleable (volumetric)	Settling Procedure	Imhoff Cone	1.0 mg/l	EPA 160.5[3]; SM 209E[1]
- Volatile (TVS,VSS)	Filtration/Gravimetric	Analytical Balance	1.0 mg/l	EPA 160.4[3]; SM 209D[1]
• Specific Conductance	Conductimetric	Conductivity Meter	1 0 mho/cm	EPA 120.1[3]; SM 205[1]
• Specific Gravity	Gravimetric	Analytical Balance	0.1 mg/l	SM 213E[1]
• Sulfate	Turbidimetric	UV/VIS Spectrometer	1.0 mg/l	EPA 375.4
• Sulfide	Titrimetric	Buret/Indicator	0.5 mg/l	EPA 376.1[3]; SM 427D[1]
• Sulfite	Titrimetric	Buret/Indicator	0.5 mg/l	EPA 377.1[3]; SM 427C[1]
• Surfactants (MBAS)	Extraction/Colorimetric	UV/VIS Spectrometer	0.05 mg/l	SM 512B[1]; EPA 425.1[3]
• Thiocycanate	Colorimetric	UV/VIS Spectrometer	1.0 mg/l	SM 412L[1]
• Thiosulfate	Titrimetric	Buret Indicator	0.1 mg/l	Guo, Phm & Jank, B. E. Wastewater Technology Center Environmental Protection Service, Environmental Canada, May, 1980.

Table 9.4 (Continued)
Method Summary
General Chemical Analysis of Water and Wastewater

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Turbidity	Nephelometric	Turbidimeter	1 NTU	EPA 180.1[3]; SM 214A[1]
• Viscosity	Saybolt Method	Viscometer/Water Bath	-	USGS Methods for Collection and Analysis of Water Samples, 1968.

References

1 American Public Health Association. Standard Methods for the Examination of Water and Wastewater (16th ed.). APHA: Washington D.C., 1986.
2 Environmental Protection Agency. Methods for the Chemical Analysis of Water and Wastes. EPA: Cincinnati, OH, 1974.
3 Environmental Protection Agency. Methods for the Chemical Analysis of Water and Wastes. EPA: Cincinnati, OH, 1979.
4 Federal Register. Volume 44 (75050). December 18, 1979.
5 Federal Register. Volume 44 (69464). December 3, 1979.
6 Federal Register. Volume 41 (57280-86). December 1, 1976.
7 Perkin Elmer Methods Manual, 1975.

Table 9.5
Analytical Procedures Summary
Soil, Sediment, Sludge Analysis

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Acid/Base Potentials	Calculation from Neutralization Potential and % Sulfur Determination	Sulfur Analyzer	0.1%	(1)
• Neutralization Potential	Titration to pH 7.0	Buret, pH Meter	0.1%	(1)
• Maximum Potential Acidity	Calculation from % Total Sulfur Determination	Sulfur Analyzer	0.1%	(1)
• Leaching Testing				
- TEP	Aqueous Extraction at controlled pH	Rotary Extractor, pH Meter	-	(2)
- ASTM	Aqueous Extraction	Agitator/Extractor	-	(3)
- CAM-WET Procedure	30 day Cumulative Extraction Two Extracting Solutions; Water and Citrate-NaCl Buffer	Agitator/Extractor	-	(4)
• pH (1:1)	1:1 Soil to Water Paste	pH Meter	0.05	(1)
• Electrical Conductivity	Extract from Saturated Soil Paste	Conductivity Meter	1 mhos/cm	(1)
• Saturation Paste Determinations (Sodium Absorption Ratio)	Atomic Absorption Spectrophotometry of Extract from Saturated Soil Paste of Calcium, Magnesium, Sodium	Atomic Absorption Spectrophotometer	1 ppm	(1)

Table 9.5
Analytical Procedures Summary
Soil, Sediment, Sludge Analysis

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Particle Size Determinations	Hydrometer or Pipette, Drying Oven	Hydrometer, Oven	1% of size	(1)
• Exchangeable Cations (Cation Exchange Capacity)	Atomic Absorption Spectrophotometry of Calcium Magnesium, Potassium, or Sodium in Salt Extract	Atomic Absorption Spectrophotometer	1 ppm	(1)
• Organic Matter (Carbon)	Walkley-Black Dichromate Oxidation, Titration	Digester, Buret	0.1%	(1)
• Heavy Metals	Digestion followed by Atomic Absorption Spectrophotometry	Atomic Absorption Spectrophotometer	Variable	(1)
• Proximate Analysis				
- Percent Ash	Gravimetric	Analytical Balance, Muffle Furnace	1%	(5)
- Percent Moisture	Gravimetric	Analytical Balance, Drying Oven	1%	(5)
- Percent Solids	Gravimetric	Analytical Balance, Drying Oven	1%	(5)
• Sulfur Determinations				
- Total	Sulfur Analyzer	Sulfur Analyzer	0.05%	(6)
- Sulfate	Extraction, Sulfur Analyzer	Sulfur Analyzer	-	(6)
- Pyritic	Extraction, Sulfur Analyzer	Sulfur Analyzer	0.05%	(6)
- Pyritic + Organic	Extraction, Sulfur Analyzer	Sulfur Analyzer	0.05%	(6)
• Specific Gravity	Pycnometer	Pycnometer	-	(7)

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Table 9.5
Analytical Procedures Summary
Soil, Sediment, Sludge Analysis

Parameter	Method	Instrumentation or Equipment	Detection Limit	Reference Method
• Oil and Grease	Drying with $MgSO_4 \cdot H_2O$ followed by Freon extraction	Soxhlet Extactor, Vacuum Pump	0.5 ppm	(8)
• Total Organic Carbon	Oxidation to CO_2 and IR Detection	Total Carbon Analyzer	1 ppm	(9)
• Bomb Preparation	Elevated Temperature and Pressure Acid Digestion	Digestion Bomb	-	(10)
• NH_3-N	KCl Extraction Ion Meter	Specific Ion Meter	0.1 ppm	(11)
• NO_3-N	Colorimetric	Spectrometer	0.1 mg/l	(8)
• TKN-N	Kjeldahl Digestion, Specific Ion Electrode	Kjeldahl Digestion Apparatus, Specific Ion Meter	1 ppm	(12)
• Phosphorus	Persulfate Digestion, Colorimetric	Spectrophotometer	0.01 ppm	(12)
• Phosphate-Phosphorus	NH_4F or $NaHCO_3$ Extraction (Dependent on pH <7.00 or >7.00	Spectrophotometer	0.05 ppm	(11)
• Potassium	SAR	AAS	0.1 ppm	(11)
• Potassium	Double-Acid Extraction	AAS	0.1 ppm	(11)
• Lithium Metaborate Fusion	Muffle Furnace Digestion	Muffle Furnace	0.1 ppm	(1)

Table 9.5 (Continued)

Method References

(1) Field and Laboratory Methods Applicable to Overburdens and Minesoil, by Andrew A. Sobek, William A. Schuller, John R. Freeman, and Richard M. Smith, College of Agriculture and Forestry, West Virginia University, Morgantown, West Virginia, March, 1978.

(2) Federal Register, Volume 45, No. 98, Book 2, May 19, 1980.

(3) 1982 Annual Book of ASTM Standards, Part 31, "Standard Test Method for Shake Extraction of Solid Waste with Water," American Society for Testing and Materials, Philadelphia, PA.

(4) California Assessment Manual for Hazardous Wastes, Hazardous Materials Management Section, California Department of Health Services, Sacramento, CA, June 1981.

(5) Methods of Soil Analysis, Part I, Physical and Mineralogical Properties, Including Statistics of Measurement and Sampling, C.A. Black, Monograph 9, American Society of Agronomists, Madison, WI, 1965.

(6) Methods of Analyzing and Testing Coal and Coke, Bulletin 638, U. S. Bureau of Mines, United States Department of the Interior, Washington, D.C. 1967.

(7) 1978 Annual Book of ASTM Standards, Part 19," Natural Building Stones; Soil and Rock," American Society for Testing and Materials, Philadelphia, PA.

(8) Standard Methods for the Examination of Water and Wastewater, 16th Edition, Method 503A, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1986, pp. 464 to 465.

(9) Operating Procedures Manual for 0524B Total Carbon System, Oceanography International Corporation, College Station, Texas, no date, pp. 10-2 to 10-4.

(10) "Perkin Elmer Autoclave-3 Instructions," Instrument Division, Perkin-Elmer Corporation, Norwalk, CT, no date.

(11) Methods of Soil Analysis, Part II, Chemical and Microbiological Properties, C. A. Black, Monograph 9, American Society of Agronomists, Madison, WI, 1965, pp. 1191 to 1195.

(12) Soil Testing and Plant Analysis, L. M. Walsh and J. D. Beaton, Soil Science Society of America, Madison WI, 1973.

Table 9.6
Method Summary
Waste Disposal Analyses
of Water, Wastewater, Soil and Waste Samples

Parameter	Method	Reference	Detection Limit	Equipment Description
BTU (Heat Content)	D240(L)(1) D271(S)(2)	5 5	0.05 Wt.%	Bomb Calorimeter
% Moisture (Water Content)	E203(L) D2216(S)	5 5	1 mg/ℓ 0.05 wt.%	Karl Fischer Titrator
% Solids (Solid Content)	160.3	6	1% (wt.)	Filtration/Oven
Organic Halogen Content	D808(L) D1305(S)	5	0.05 wt.%	Bomb Calorimeter
Organic Sulfur Content	D129(L) D3177(S)	5	0.05 wt.%	Bomb Calorimeter
% Ash Content	160.4	2	0.05 wt.%	Muffle Furnace, Balance
Ignitability	1010	2	±2°F	Pensky Martens Flash Point Tester
	1020	2	±1°F	Setaflash Method Closed Cup
Reactivity	8.3	2	1 ppm	Reaction Vessel
EP Toxicity	1310	2	0.1 mg/ℓ	Extractors/AAS Instrumentation
Corrosivity	1110	2	0.1 pH unit	pH Meter
Organochlorine Pesticides/PCBs	608(L) 8080(S)	1,3 2	0.1 µg/ℓ See Table 9.2	Gas chromatograph
% Acidity	402	4	1 mg/ℓ	Titration
% Alkalinity	403	4	1 mg/ℓ	Titration
Conductivity (Specific Conductance)	120.1	6	1 µmho/cm	Conductivity Meter
Ammonia Nitrogen	350.3	6	1 mg/ℓ	Distillation/Specific Ion Meter

Table 9.6 (Continued)
Method Summary
Waste Disposal Analyses
of Water, Wastewater, Soil and Waste Samples

Parameter	Method	Reference	Detection Limit	Equipment Description
TOC (Total Organic Carbon)	415.1	6	1 mg/ℓ	TOC Analyzer
TOX (Total Organic Halogen)	450.1 (9020)	6 2	0.05 mg/ℓ 5 µg/ℓ	TOX Analyzer
Chloride	325.3	6	1 mg/ℓ	Titration
Density (Specific Gravity)	213E	4	-	Pycnometer
Oil and Grease	413.1	6	0.5 mg/ℓ	Extraction/ Gravimetric
Viscosity	D88	5	1 CPS	Viscometer (Water Bath)
pH	9040	2	0.1 pH unit	pH Meter
Sulfate	375.4	6	1 mg/ℓ	Titration
Cyanide	9010	2	0.05 mg/ℓ 1 µg/g	Colorimetric

Table 9.6 (Continued)

REFERENCES:

1. Federal Register, Volume 44, December 3, 1979, 40 CFR Part 136, pp. 69464 to 69575.
2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Waste Characterization Branch, Office of Solid Waste, 3rd Edition U.S. Environmental Protection Agency, Washington, D.C., September 1986.
3. Manual of Analytical Methods for Analysis of Pesticides in Human and Environmental Samples, U.S. Environmental Protection Agency, EPA 600/8-80-038, June 1980.
4. Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1986, Washington, D.C.
5. American Society for Testing and Materials, Philadelphia, PA.
6. Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1979.

FOOTNOTES:

(1) L indicates the method for liquid samples.

(2) S indicates the method is for semisolid and solid samples.

Table 9.7
Method Summary
Compatibility/Characterization Analyses
of Hazardous Wastes

Parameter	Method No.	Reference	Limit	Equipment Description
PCBs	608 8080 Section 8	1 2 3	0.1)g/P (water) See Table 9.2 1.0 ppm (air)	Gas Chromatograph
Water Reactivity	15.5.11	6	-	Reaction Vessel
pH	9040 -	2 4	0.01 pH unit 1 pH Unit	pH meter Test Paper
Flammability	7.1	2	±2°F	Flash Point Tester
Radioactivity	-	4 6	- Sensitivity 800 Counts/Minute	Survey Meter Geiger-Mueller Counter
Water Solubility	15.5.1	6	-	Beaker
Cyanide	412E Hach (spot test)	6 4	0.1 ppm 2.0 ppm	Specific Ion Electrode Colorimetric (semiquantitative)
Sulfide	-	4	N/P(1)	Test Paper
Oxidizers	-	4	N/P	Test Paper
Peroxides	-	4	N/P	Test Paper

Table 9.7 (Continued)
Method Summary
Compatibility/Characterization Analyses
of Hazardous Wastes

Parameter	Method No.	Reference	Limit	Equipment Description
Oxidation/Reduction Potential (Eh)	D1498	7	1mV	Ion Analyzer
Specific Gravity	213E	5	-	Pycnometer
Hexane Solubility	-	6	-	Reaction Vessel

References:

1. Federal Register, Volume 44, December 3, 1979, 40 CFR Part 136, pp. 69464 to 69575.
2. Test Method for Evaluating Solid Waste, Physical/Chemical Methods, Waste Characterization Branch, Office of Solid Waste, U.S. Environmental Protection Agency, Washington, D.C., 3rd Edition, September 1986.
3. Manual of Analytical Methods for Analysis of Pesticides in Human and Environmental Samples, U.S. Environmental Protection Agency, EPA 600/8-80-038, June 1980.
4. Analysis performed as per manufacturer's recommended procedure.
5. Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1986, Washington, D.C.
6. Drum Consolidation Protocol (Draft), U.S. Environmental Protection Agency, August 1981.
7. American Society for Testing and Materials, Philadelphia, PA.

Footnotes:

(1) N F indicates results are qualitative, and the quantitative results for precision are not applicable.

TABLE 9.8
LIST OF APPROVED TEST PROCEDURES
FOR SOIL AND SOLID WASTES(1)

Parameter	Section No.	Method No.
CHARACTERISTICS OF SOLID WASTE	8.0	
• Ignitability	8.1	
- Pensky-Martens Closed Cup Method	8.1	1010
- Setaflash Closed Cup Method	8.1	1020
• Corrosivity	8.2	
- Corrosion Towards Steel	8.2	1110
• Reactivity	8.3	
• Extraction Procedure Toxicity	8.4	
- Method and Structural Integrity Test	8.4	1310
- Multiple Extraction Procedure	6.0	1320
- Extraction Procedure for Oily Wastes	6.0	1330
SAMPLE PREPARATION TECHNIQUES	4.0	
• Inorganic Techniques	3.0	
- Acid Digestion of Waters for Flame AAS or ICP	3.0	3005
- Acid Digestion of Aqueous Samples and Extracts Flame AAS or ICP	3.0	3010
- Acid Digestion of Aqueous Samples and Extracts for Total Metals for Furnace AAS	3.0	3020
- Dissolution Procedure for Oil, Grease, or Wax	3.0	3040
- Acid Digestion of Sediments, Sludges, and Soils	3.0	3050
• Organic Techniques, Extractions and Preparations	4.2.1	
- Organic Extraction and Sample Preparation	4.2.1	3500
- Separatory Funnel Liquid-Liquid Extraction	4.2.1	3510
- Continuous Liquid-Liquid Extraction	4.2.1	3520
- Soxhlet Extraction	4.2.1	3540
- Sonication Extraction	4.2.1	3550
- Waste Dilution	4.2.1	3580
- Purge and Trap	4.2.1	5030
- Protocol for Analysis of Sorbent Cartridges from Volatile Organic Sampling Train	4.2.1	5040
• Cleanup		
- Cleanup	4.2.2	3600
- Alumina Column Cleanup	4.2.2	3610
- Alumina Column Cleanup and Separation of Petroleum Wastes	4.2.2	3611
- Florisil Column Cleanup	4.2.2	3620
- Silica Gel Cleanup	4.2.2	3630

Table 9.8
(Continued)

Parameter	Section No.	Method No.
- Gel-Permeation Cleanup	4.2.2	3640
- Acid-Base Partition Cleanup	4.2.2	3650
- Sulfur Cleanup	4.2.2	3660
• Screening	4.4	
- Headspace	4.4	3810
- Hexadecane Extraction and Screening if Purgeable Organics	4.4	3820
METHODS FOR DETERMINATION OF METALS	3.0	
• Inductively Coupled Plasma Atomic Emission Spectroscopy	3.3	6010
• Atomic Absorption Methods	3.0	7000
• Aluminum (AA, Direct Aspiration)	3.0	7020
• Antimony (AA, Direct Aspiration)	3.0	7040
• Antimony, (AA, Furnace Technique)	3.0	7041
• Arsenic, (AA, Furnace Technique)	3.0	7060
• Arsenic, (AA, Gaseous Hydride)	3.0	7061
• Barium, (AA, Direct Aspiration)	3.0	7080
• Beryllium, (AA, Direct Aspiration)	3.0	7090
• Beryllium, (AA, Furnace Technique)	3.0	7091
• Cadmium, (AA, Direct Aspiration)	3.0	7130
• Cadmium, (AA, Furnace Technique)	3.0	7131
• Chromium, (AA, Direct Aspiration)	3.0	7190
• Chromium, (AA, Furnace Technique)	3.0	7191
• Chromium, Hexavalent, (Chelation/Extraction)	3.0	7197
• Chromium, Hexavalent, (Colorimetric)	3.0	7196
• Chromium, Hexavalent, (Coprecipitation)	3.0	7195
• Chromium, Hexavalent, (Differential Pulse Polarography)	3.0	7198
• Cobalt (AA, Direct Aspiration)	3.0	7200
• Cobalt (AA, Furnace Technique)	3.0	7201
• Copper, (AA, Direct Aspiration)	3.0	7210
• Iron, (AA, Direct Aspiration)	3.0	7380
• Lead, (AA, Direct Aspiration)	3.0	7420
• Lead, (AA, Furnace Technique)	3.0	7421
• Magnesium (AA, Direct Aspiration)	3.0	7450
• Manganese, (AA, Direct Aspiration)	3.0	7460
• Mercury, in Liquid Waste (Manual Cold Vapor Technique)	3.0	7470

Table 9.8
(Continued)

Parameter	Section No.	Method No.
• Mercury, in Solid or Semisolid Waste (Manual Cold Vapor Technique)	3.0	7471
• Molybdenum (AA, Direct Aspiration)	3.0	7480
• Molybdenum (AA, Furnace Technique)	3.0	7481
• Nickel, (AA, Direct Aspiration)	3.0	7520
• Osmium, (AA, Direct Aspiration)	3.0	7550
• Potassium, (AA, Direct Aspiration)	3.0	7610
• Selenium, (AA, Furnace Technique)	3.0	7740
• Selenium, (AA, Gaseous Hydride)	3.0	7741
• Silver, (AA, Direct Aspiration)	3.0	7760
• Sodium, (AA, Direct Aspiration)	3.0	7770
• Thallium, (AA, Direct Aspiration)	3.0	7840
• Thallium, (AA, Furnace Technique)	3.0	7841
• Tin (AA, Direct Aspiration)	3.0	7870
• Vanadium, (AA, Direct Aspiration)	3.0	7910
• Vanadium, (AA, Furnace Technique)	3.0	7911
• Zinc, (AA, Direct Aspiration)	3.0	7950
ORGANIC ANALYTICAL METHODS	4.3	
• Gas Chromatographic Methods	4.3.1	8000
- Acrolein, Acrylonitrile, Acetonitrile	4.3.1	8030
- Aromatic Volatile Organics	4.3.1	8020
- Chlorinated Herbicides	4.3.1	8150
- Chlorinated Hydrocarbons	4.3.1	8120
- Halogenated Volatile Organics	4.3.1	8010
- Nitroaromatics and Cyclic Ketones	4.3.1	8090
- NonHalogenated Volatile Organics	4.3.1	8015
- Organochlorine Pesticides and PCBs	4.3.1	8080
- Organophosphorus Pesticides	4.3.1	8140
- Phenols	4.3.1	8040
- Phthalate Esters	4.3.1	8060
- Polynuclear Aromatic Hydrocarbons	4.3.1	8100
• Gas Chromatographic/Mass Spectroscopy Methods (GC/MS)	4.3.2	
- GC/MS for Volatile Organics	4.3.2	8240
- GC/MS for Semivolatile Organics, Packed Column Technqiue	4.3.2	8250
- GC/MS for Semivolatile Organics, Capillary Column Technqiue	4.3.2	8270
- Analysis of Polychlorinated Dibenzo-P-Dioxins and Polychlorinated Dibenzofurans	4.3.2	8280
Appendix A: Signal-to-Noise Determination Methods		
Appendix B: Recommended Safety and Handling Procedures for PCDD's/PCOFI		

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Table 9.8
(Continued)

Parameter	Section No.	Method No.
• High Performance Liquid Chromatographic Methods (HPLC)	4.3.3	
- Polynuclear Aromatic Hydrocarbons	4.3.3	8310
MISCELLANEOUS TEST METHODS	5.0	
• Cation Exchange Capacity of Soils (Ammonium Acetate)	6.0	9080
• Cation Exchange Capacity of Soils (Sodium Acetate)	6.0	9081
• Chloride (Colorimetric, Automated Ferricyanide AAI)	5.0	9250
• Chloride (Colorimetric, Automated Ferricyanide AAII)	5.0	9251
• Chloride (Titrimetric, Mercuric Nitrate)	5.0	9252
• Total Coliform: Multiple Tube Fermentation Technique	5.0	9131
• Total Coliform: Membrane Filter Technique	5.0	9132
• Total and Amendable Cyanide (Colorimetric, Manual)	5.0	9010
• Total and Amendable Cyanide (Colorimetric, Automated UV)	5.0	9012
• Paint Filter Liquids Test	6.0	9095
• Gross Alpha and Gross Beta	6.0	9310
• Nitrate	5.0	9200
• pH Electrometric Measurement	6.0	9040
• pH Paper Method	6.0	9041
• Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)	5.0	9065
• Phenolics (Colorimetric, Automated 4-AAP with Distillation)	5.0	9066
• Phenolics (Spectrophotometric, MBTH with Distillation)	5.0	9067
• Alpha-Emitting Radium Isotopes	9.0	9315
• Radium, 228	5.0	9320
• Soil pH	6.0	9045
• Specific Conductance	6.0	9050
• Sulfate (Colorimetric, Automated, Chloranilate)	9.0	9035
• Sulfate (Colorimetric, Automated, Methylthymol, Blue,AA II)	9.0	9036
• Sulfate (Turbidimetric)	9.0	9038
• Sulfides	5.0	9030
• Total Recoverable Oil and Grease (Gravimetric, Separatory Funnel Extraction)	5.0	9070
• Oil and Grease Extraction Method for Sludge Samples	5.0	9071

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Table 9.8
(Continued)

Parameter	Section No.	Method No.
• Total Organic Carbon	5.0	9060
• Total Organic Halides (TOX)	5.0	9020
• Total Organic Halides (TOX) by Neutron Activation	5.0	9022
• Saturated Hydraulic Conductivity, Saturated Leachate Conductivity and Intrinsic Permeability	6.0	9100

FOOTNOTES:

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, SW-846, U.S. Environmental Protection Agency, 1986.

Table 9.9 Toxicity Characteristic Leaching Procedure (TCLP) Detection Limits

Compound	Regulatory Level* (mg/liter)	Detection Limit: Low Samples (mg/liter)	Detection Limit: High Samples (mg/liter)
	Volatile Organics		
Acrylonitrile	5.0	0.2	1.0
Benzene	0.07	0.01	0.05
Carbon disulfide	14.4	0.01	0.05
Carbon tetrachloride	0.07	0.01	0.05
Chlorobenzene	1.4	0.01	0.05
1,2-Dichloroethane	0.4	0.01	0.05
1,1-Dichloroethylene	0.1	0.01	0.05
Isobutanol	25	0.5	2.5
Methylene chloride	8.6	0.05	0.25
Methyl ehtyl ketone	7.2	0.01	0.05
1,1,1,2-Tetrachloro-ethane	10	0.01	0.05
1,1,2,2-Tetrachloro-ethane	1.3	0.01	0.05
Tetrachloroethylene	0.1	0.01	0.05
Toluene	14.4	0.01	0.05
1,1,1-Trichloroethane	25	0.01	0.05
1,1,2-Trichloroethane	1.2	0.01	0.05
Trichloroethylene	0.7	0.01	0.05
Vinyl chloride	0.05	0.01	0.05

*Test Methods for Evaluating Solid Waste, USEPA SW-846, Third Edition, September 1986.

Table 9.9 (Continued)

Compound	Regulatory Level (mg/liter)	Detection Limits (mg/liter)
	Extractable Organics	
Bis(2-chloroethyl)ether	0.05	0.02
Cresols (total)	10	0.10
1,2-Dichlorobenzene	4.3	0.02
1,4-Dichlorobenzene	10.8	0.02
2,4-Dinitrotoluene	0.13	0.02
Hexachlorobenzene	0.13	0.02
Hexachlorobutadiene	0.72	0.02
Hexachloroethane	4.3	0.02
Nitrobenzene	0.13	0.02
Pentachlorophenol	3.6	0.10
Phenol	14.4	0.02
Pyridine	5.0	0.10
2,3,4,6-Tetrachlorophenol	1.5	0.10
2,4,5-Trichlorophenol	5.8	0.05
2,4,6-Trichlorophenol	0.3	0.05
	Extractable Pesticides	
Chlordane	0.03	0.005
2,4-D	1.4	0.05
Endrin	0.003	0.003
Heptachlor	0.001	0.001
Lindane	0.06	0.005
Methoxychlor	1.4	0.01
Toxaphene	0.07	0.05
2,4,5-TP (Silvex)	0.14	0.05

Table 9.9 (Continued)

Metal	Regulatory Level (mg/liter)	Detection Limit (mg/liter)
Arsenic	5.0	0.005
Barium	100.0	1.0
Cadmium	1.0	0.01
Chromium	5.0	0.05
Lead	5.0	0.05
Mercury	0.2	0.0002
Selenium	1.0	0.005
Silver	5.0	0.01

10.0 DATA VERIFICATION

Data verification is the steps taken within an analytical laboratory so that reported results correctly represent the analyses performed. There are two basic verification activities:

- The processing of quality control sample results to demonstrate that analyses are within laboratory-prescribed bounds for accuracy, precision, and completeness.
- Data validation to demonstrate that numerical computation of data is correct and that it is correctly reported.

This section discusses how data verification is performed. Section 12.0 discusses maintenance of resulting records.

10.1 PROCESSING OF QUALITY CONTROL DATA

This section discusses the analytical treatment of the data resulting from the quality control samples discussed in Section 8.0.

10.1.1 Specific Routine Procedures Used to Assess Data Precision and Accuracy

Following are the procedures recommended for evaluating the precision and accuracy of all data generated within ITAS Laboratories. Quality control sample analyses are performed as appropriate for organic or inorganic sample analyses as discussed in Section 8.0. The protocol used will be in accordance with specific analytical procedures if QC requirements are stated in the procedure.

- A reagent and/or method blank is prepared and analyzed with each set of samples.
- Field and trip blanks are analyzed to determine possible sample contamination during collection and shipment to the laboratory. Trip blanks are applicable to volatile organics analysis (VOA) where volatile contaminants can be introduced from ambient air during shipment.

- Bottle blanks are prepared and analyzed to demonstrate that clean containers were shipped to the field for sample collection. If purchased, vendor certificates should be retained to doucment cleanliness and lot numbers.

- Rinsate blanks from the field are analyzed to show sampling equipment was properly cleaned between samples.

- A daily calibration curve consisting of at least three standards and a reagent blank is prepared for each parameter. If the standard curve is known to be stable, the standard curve can be verified daily by the analysis of a midpoint standard.

- One sample in every sample set of 20 samples is analyzed in duplicate, or as a matrix sample duplicate.

- One sample in every sample set of 20 samples is spiked at a level to provide a final concentration equivalent to the original concentration of the sample before dilution.

- A blind duplicate, unknown to the Analyst, is introduced by the Quality Control Coordinator at least once monthly. Blind duplicates are routinely used for the analysis of metals, water quality parameters, and organics analyses.

- Standard Reference Materials (SRMs) are introduced periodically into the testing scheme as verification or reference standards, by the Quality Control Coordinator to evaluate the accuracy of standards, the testing procedure, and the analyst's performance.

- A blank spike consisting of deionized water or a purified solid matrix spiked with the parameter of interest is analyzed. Blank spikes are used to show the analytical technique was in control, although matrix effects may have been present in associated matrix spike cycles.

- Every sample is spiked with the appropriate surrogate standards prior to extraction and analysis

for volatile organic compounds, base-neutrals, acids, and pesticides/PCBs.

- Internal standards are added to all samples or extracts prior to GC/MS analysis.

- A check standard or midpoint standard is run to verify the continued acceptability of a calibration curve.

- Analysis matrix spikes (or Method of Standard Additions) of target analytes added to samples or extracts just prior to analysis to give information on matrix effects encountered during analysis.

- Field matrix spikes are sample portions spiked with target analytes spiked into samples in the field to provide data on stability.

- Collocated samples provide information on precision of intralaboratory or interlaboratory analytical systems when sent to the same or different laboratories, respectively.

- A replicated or split sample provides information about precision depending on the location in the sampling and analysis process where the split occurs. An analysis replicate gives information about the analytical precision; a field replicated or field split sample provides precision information for all steps after sample collection.

When the analyses of a sample set are completed, the results will be reviewed as described in Laboratory-Specific Attachments and/or SOPs, and evaluated to assess the validity of the data set. Review is based on the following criteria:

- Reagent Blank Evaluation - The reagent and/or method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents should be checked for contamination and the analysis halted until the system can be brought under control before

further sample analysis proceeds. A high background is defined as a background value sufficient to result in a difference in the sample value, if not corrected, greater than or equal to smallest significant digit known to be true. A reagent blank should contain no greater than two times (2X) the parameter detection limit for most parameters (1,2,3,4).*

- Field Trip, Bottle, and Rinsate Blank Evaluation Field blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high field blank readings are encountered, the procedure for sample collection, shipment, and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the trip blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. In the case of VOAs, ambient air in the laboratory and reagents should be checked as possible sources of contamination. High field blank readings for other parameters may be due to contaminated sample bottles (unless bottle blanks are clean) or cross-contamination due to sample leakage and poorly sealed sample containers, or cross-contamination between samples collected if the rinsate blank is contaminated (1, 2, 3, 4).

- Calibration Standard Evaluation - The daily calibration curve is evaluated to determine linearity through its full range, and that sample values are within the range defined by the low and high standards. If the curve is not linear ($r \geq 0.995$), sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm

*The criteria are denoted as applicable to:

1 - Volatile organic compounds

2 - Base-neutral, acid, and pesticide organic compounds

3 - Metals

4 - Water quality.

9 1 1 2 7 6 1 0 2 2 4

to fit a nonlinear curve to the standards (1,2,3,4).

- Duplicate Sample Evaluation - Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the relative percent difference (RPD). The precision value, RPD, should be plotted on control charts for the parameter determined. If the precision value exceeds the warning limit (Section 10.1.2.2) for the given parameter, the appropriate Group Leader, Operations Manager, or the Quality Control Coordinator is notified. If the precision value exceeds the control limit, the reason for the nonconformance must be determined; corrective action may include reanalysis for the parameter in question. Attainable precision limits will be specified by the Quality Control Coordinators and updated periodically following review of data (1,2,3,4).

- Matrix and Blank Spike Evaluations - The observed recovery of the matrix spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. The accuracy value, the percent recovery, may be plotted on a control chart for the parameter determined. If the accuracy value exceeds the warning limit for the given parameter, the appropriate Group Leader, Operations Manager, or the Quality Control Coordinator is notified. If the accuracy value exceeds the control limit, the reason for nonconformance must be determined; corrective actions may include reanalysis for the parameter in question. If interferences are present in the samples spiked, a blank spike is used to demonstrate that the laboratory technique is in control. The results of matrix spike and matrix spike duplicate parts of samples are compared as duplicate samples described above (RPD) (1,2,3,4).

- QC Check, or Laboratory Control, Standard - Prepared as blank spikes, these samples are used for initial proficiency demonstration of analysts, methods, and equipment. Material is obtained with known values (such as river sediment); it may be analyzed without spiking as

a Laboratory Control Standard (LCS), similiar to a Reference Standard (1,2,3,4).

- Blind Duplicate Evaluation - The blind duplicate analysis is evaluated in the same manner as described above for the duplicate sample analysis and is treated as a duplicate result for purposes of evaluating the precision of the analytical method. This evaluation is performed independently by the Quality Control Coordinator (1,2,3,4).

- Reference Standard Evaluation - Standard Reference Materials analyses are compared with true values and acceptable ranges. Values outside the acceptable ranges require corrective action to determine the source of error and provide correction. Analyses data reporting should be halted pending this evaluation. Following correction of the problem, the Standard Reference Material should be reanalyzed (1,2,3,4).

- Check Standard Evaluation - The results of check standard analysis are compared with the original calibration curve, and the relative percent difference of the check standard is calculated to determine if the calibration system is in control. If correction is required, the check standard should be reanalyzed to demonstrate that the corrective action has been successful (1,2,3,4).

- Surrogate Standard Evaluation - The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards are determined. Percent recoveries attained shall be in accordance with current EPA recommendations/ requirements, or in their absence, laboratory-generated control limits (1,2).

- Analysis Matrix Spikes Evaluation - Suppression or enhancement of instrument signal levels is demonstrated when recoveries are lowered or raised, respectively. Matrix-suppressing agents may be added to the sample or the extract to reduce such effects in routine sample analysis when the evaluation indicates samples are affected in this manner. One to three levels of

9 1 1 2 7 6 0 2 2 6

analysis matrix spike concentrations are used to determine the unaffected concentration level native to the sample. The observed recovery versus the theoretical recovery is used to calculate the accuracy as defined by the percent recovery. Percent recovery may be plotted on control charts. If the accuracy value exceeds the warning limits for a given parameter, the appropriate Group Leader, Operation Manager, or QC Coordinator is notified. The reason for nonconformance must be determined, and appropriate corrective actions taken. These may include reanalysis of the sample(s) for the parameter in question(3).

- Field Matrix Spike Evaluation - Target analytes spiked into a portion of a field sample are measured to determine the amounts recovered after collection, shipment, storage, preparation and analytical processes have acted upon the analytes. Decreased recoveries may be due to bacteriological action on the poorly preserved sample, to photodegradation, to leaks from improperly capped/sealed containers, or degradation during physical treatment for analysis such as evaporation of organic solvents to reduce the sample extract volumes. The observed recovery versus the theoretical recovery is used to calculate the accuracy as defined by the percent recovery. Percent recovery may be plotted on control charts. If the accuracy value exceeds the warning limits for a given parameter, the appropriate Group Leader, Operation Manager, or QC Coordinator is notified. The reason for nonconformance must be determined, and appropriate corrective actions taken. These may include reanalysis of the sample(s) for the parameter in question (1,2,3,4).

- Collocated Sample Evaluation - Two or more samples processed independently by different people at different times using different techniques, or combinations of these variables, will yield results which provide an estimate of long-term precision of measurement systems. Or, if processed identically together, short-term precision is estimated (1,2,3,4).

- Replicate Sample Evaluation - Replicate sample analysis for the sample set is used to determine

the precision of the sampling and analytical method for the sample matrix. Field splits provide precision information about all steps after collection; analytical splits, or laboratory duplicates, provide information about instrument precision. The replicate results are used to calculate precision as RPD. Precision values exceeding warning limits require the appropriate Group Leader, Operations Manager, or QC Coordinator to be notified. The reason for the nonconformance must be determined and corrective actions taken. Laboratory duplicate precision limits are specified by the QC Coordinator and updated periodically (1,2,3,4).

10.1.2 Statistical Evaluation of Quality Control (QC) Data

As part of the analytical quality control program, ITAS laboratories will determine precision and accuracy for each parameter or class of parameter analyzed. Initially, when these data are compiled, the evaluation is applied over a broad concentration range. As more data are accumulated, precision and accuracy determinations will be updated and criteria developed to define precision and accuracy over specific concentration ranges.

10.1.2.1 Evaluation of Data Using Control Charts

ITAS laboratories will apply precision and accuracy criteria to each parameter that is analyzed. When analysis of a sample set is completed, the quality control data are reviewed and evaluated through the use of control charts to validate the data set.

Control charts are established for all major analytical parameters. This discussion of control charts is a guideline which shall be used unless other provisions are stated. A minimum of 20 measurements of precision and accuracy are required before control limits can be established. In general, warning limits of two standard deviations shall be used; three standard deviations should be used as control limits. Once established, control limits are updated as additional

precision and accuracy data become available by the Quality Control Coordinator. Other methods may be used as described in laboratory-specific attachments to this manual.

10.1.2.2 Evaluation of Analytical Precision

General Considerations

To determine the precision of the method and/or laboratory analyst, a routine program of duplicate analyses is performed. These may also be matrix spike/matrix spike duplicate pairs. The results of the duplicate analyses are used to calculate the relative percent difference (RPD), which is the governing quality control parameter for precision.

The RPD for replicate analyses is defined as the difference (range) of each replicate set, divided by the average value (mean) of the replicate set, times 100 percent. For replicate results D_1 and D_2, the RPD is calculated from Equation 10-1:

$$RPD\% = \frac{D_1 - D_2}{\frac{D_1 + D_2}{2}} \times 100\% \qquad (10\text{-}1)$$

When the RPD is obtained for at least 20 replicate pairs, the average RPD and the standard deviation are calculated using:

$$\bar{m} = \frac{\sum_{i=1}^{n} m_i}{n} \qquad (10\text{-}2)$$

and

$$Sm = \sqrt{\frac{\sum_{i=1}^{n} (m-\bar{m})^2}{n-1}} \qquad (10\text{-}3)$$

where

m = the RPD of a replicate pair,

$\bar{m}$ = the average of the Relative Percent Difference determinations,

Sm = the standard deviation of the data set of RPD determinations, and

n = the number of RPD determinations.

When constructing a control chart for a specific parameter, the Warning and Control Limits are then calculated from the following:

Upper Control Limit = $\bar{m} + 3\ Sm$
Lower Control Limit = $\bar{m} - 3\ Sm$
Upper Warning Limit = $\bar{m} + 2\ Sm$
Lower Warning Limit = $\bar{m} - 2\ Sm$

A control chart is established by plotting the RPD of each replicate pair on a graph generated as follows:

- The average of the RPD determinations for the original data set is established as the midpoint on the Y axis of the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the RPD determinations.
- The Lower Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis below the mean of the RPD determinations.
- The calculated RPD of each replicate pair is plotted on the graph to determine whether the RPD is within the Warning and Control Limits of the Control Chart.
- If the RPD plots between the Warning and Control

Limits, the Group Leader, Operations Manager, or Quality Control Coordinator is notified for a decision as to how to proceed.

- If the RPD plots outside the Control Limits, the data set must be reviewed and the results of the analysis considered preliminary until the source of error has been determined and corrective action taken.

- Factors that influence precision include: Analyst skill; instrument stability; sample homogeneity; reagents quality; environmental fluctuations.

10.1.2.3 Evaluation of Analytical Accuracy

In addition to the evaluation of analytical precision, ITAS laboratories evaluate accuracy.

When a program for evaluation of analytical accuracy is established, the evaluation is applied over the entire range of spiking concentrations. As more data are accumulated, the evaluation procedure is refined to define the analytical accuracy of the method over specific concentration ranges.

To determine the accuracy of an analytical method and/or the laboratory analyst, a periodic program of sample and blank spiking is conducted. The results of matrix, matrix spike duplicate, and blank spikes are used to calculate the quality control parameter for accuracy evaluation, the Percent Recovery (%R).

The %R is defined as the observed concentration, minus the sample concentration, divided by the true concentration of the spike, times 100 percent.

$$\%R = \frac{O_i - O_s}{T_i} \times 100\% \qquad (10\text{-}4)$$

9 1 1 2 7 6 4 0 2 3 1

where

$\%R$ = the Percent Recovery,

O_i = the Observed Spiked Sample Concentration,

O_s = the Sample Concentration, and

T_i = the True Concentration of the Spike.

The True Concentration is calculated from Equation 10-5:

$$T_i = \frac{\text{Spike Concentration [c] (mg/L)} \times \text{Volume of Spike (in mL)}}{\text{Volume of Sample [in mL]} + \text{Volume of Spike [in mL]}} \qquad (10\text{-}5)$$

When the Percent Recovery is obtained for at least twenty spiked samples, the mean percent recovery and the standard deviation are calculated using the formula:

$$\overline{\%R} = \frac{\sum_{i=1}^{n} \%R_i}{n} \qquad (10\text{-}6)$$

and

$$S_R = \sqrt{\frac{\sum_{i=1}^{n} (\%R_i - \overline{\%R})}{n-1}} \qquad (10\text{-}7)$$

where

$\overline{\%R}$ = the Mean Percent Recovery,

$\%R_i$ = the Percent Recovery of a Single Spiked Sample,

n = the number of results, and

S_R = the Standard Deviation of the data set of Percent Recovery determinations.

The Warning and Control Limits are then calculated from the following equations:

$$\text{Upper Control Limit} = \overline{\%R} + 3\ S_R$$
$$\text{Lower Control Limit} = \overline{\%R} - 3\ S_R$$
$$\text{Upper Warning Limit} = \overline{\%R} + 2\ S_R$$
$$\text{Lower Warning Limit} = \%R - 2\ S_R$$

A control chart (as shown in Figure 10-1) is generated by plotting the Percent Recovery data on a graph as follows:

- The average of the Percent Recovery determinations for the original data set is established as the midpoint on the Y axis on the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the Percent Recovery determinations.
- The Lower Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis below the mean of the Percent Recovery determinations.
- The calculated Percent Recovery of each spiked sample is plotted on the graph to determine whether the Percent Recovery is within the Warning and Control Limits of the Control Chart.
- If the Percent Recovery plots between the Warning and Control Limits, the Group Leader, Operations Manager, or Quality Control Coordinator is notified for a decision as to how to proceed.
- If the Percent Recovery plots outside the Control Limits, the data set must be reviewed and the results of the analysis considered preliminary until the source of error has been determined and corrective action taken.
- The Warning and Control Limits are recalculated for the data set on a periodic basis and the Control Chart for the corresponding parameter is updated.

- Factors that influence bias (accuracy) include: interference, calibration, contamination, losses during preparation, instrument drift, sensitivity resolution, analyst techniques.

Completed control charts are filed by the Quality Control Coordinator; current charts are kept at the work station for use in decision-making and updating.

10.1.3 Example Precision and Accuracy Data

The USEPA has compiled historical precision and accuracy data which are presented in Tables 10.1A through 10.2C. Footnotes to each table cite the sources of the data and concentration or concentration range at which the precision and accuracy were determined. When a concentration is not cited, concentration information was not available in the source material. These tables are provided as guidance for precision and accuracy that has been achieved on environmental samples.

The different levels of analytical techniques are described below. (Note: These levels are not the same as the source QC levels described in Section 8.3):

- Level I - Field screening or analysis using portable instruments. Results are often not compound specific and not quantitative but results are available in real-time.

- Level II - Field analysis using more sophisticated portable analytical instruments; in some cases, the instruments may be set up in a mobile or on-site laboratory. There is a wide range in the quality of data that can be generated. Quality depends on the use of suitable calibration standards, reference materials, sample preparation equipment, and the training of the operator. Results are available in real-time or several hours.

- Level III - All analyses performed in an off-site analytical laboratory using standard, documented procedures. The laboratory may or may not be a CLP laboratory.

- Level IV - CLP routine analytical services (RAS). All analyses are performed in an off-site CLP analytical laboratory following CLP protocols.

10.2 DATA VALIDATION

Data validation (review) is the process whereby data are screened and accepted or rejected, based on a set of criteria. Data will have been provided by trained analysts using approved methods and instrument systems in control.

Data validation is a systematic procedure of reviewing a body of data against a set of criteria to verify its validity prior to its intended use. It is applied to a body of data after the fact, systematically and uniformly. It must be applied close to the origin of the data by an independent and objective reviewer.

Data validation begins with the processing of data and continues through review of the data and the reporting of analytical results. Each laboratory will document its data validation procedures in its laboratory-specific attachment to this manual. Data processing can be performed by the analyst who obtained the data or another analyst. Data review starts with an analyst independent of the data acquisition and processing, or the Group Leader, reviewing (validating) that data processing has been correctly performed and continues through verifying that the reported analytical results correspond to the data acquired and processed. Checks are made for internal consistency, proper identification, transmittal errors, calculation errors and transcription errors. Final review of the data to be reported is by the Operations Manager. The procedure is outlined in Figure 10-2.

As stated, the first step in validation is data processing. In general, data

will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, calculations should be done on standard IT calculation paper and attached to the data sheets. The analyst shall sign or initial and date in ink each page of calculations.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number(s) and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst.

If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and response factors, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than). The analyst shall sign and date the resulting output.

10.2.1 Review of Data Processing

Following is a discussion of two methods to be used for reviewing (checking) data processing. At least 20 percent of all data shall be checked using one of these procedures. If, during the checking process, errors are determined in a data set, checking shall be completely (100

percent) performed for the data set. The first procedure is called the "checkprint" procedure:

- The analyst performing the data processing shall give an analyst independent of the work the data package. The package shall include, as appropriate, raw data, data sheets, strip charts, computer input/output, calculations, sources for input parameters such as response factors, etc.

- The independent analyst (checker) will make and review copies of the data for:

  - Appropriateness of equations used

  - Correctness of numerical input

  - Numerical correctness of all calculations; this should be done by reperforming numerical computations

  - Correct interpretation of strip charts, etc.

- All entries and calculations that the checker reviews shall be marked in ink with a check mark or yellow highlighting on the copies. The checking process must be thorough enough to validate that the results are correct. If the checker disagrees with any part of the computations, the checker shall mark through the number with a single line and place the revised number above it.

- Any changes made by the checker shall be back-checked by the originator. If the originator agrees with the change, no action is necessary. If the originator disagrees, the originator and checker must resolve the difference so they agree with the result presented.

- The checker shall sign originals and date in ink all pages of the data package (except for groups of printout such as chromatograms). Signing and dating indicates that the reviewer agrees with the calculations and that any changes made have been agreed to by the originator and entered on the originals.

- If the data have been processed by computer, the

reviewer shall check at least 20 percent of input entries. Agreement should be indicated by a check mark for every line. If the checker disagrees with the input, the number should be marked through with a line and the corrected number indicated above it. Corrections must be backchecked by the originator as discussed above.

If an input error is identified and the data have been processed, it will be necessary to reprocess the data. In this event, the checker shall mark the second set of input to indicate agreement with the input changes. The checker shall sign and date in ink the computer input to indicate agreement.

- Raw data that are automatically acquired and processed do not require any validation at this point beyond that previously discussed.

- The reviewed data are maintained as discussed in Section 12.0.

- Use of the copies of data for checking produces a "checkprint" which should be maintained as a record to document the review performed.

The "original check" procedure is an alternative to the checkprint process; instead of a copy (checkprint) being used, analysts

- Mark on originals for checking in black ink, initialling and dating these entries as above.

- Backcheck changes in black ink using lineouts, initials, and dates as above.

- Summary checklists may be used and attached to the checked data which clearly describes the data checked, what the check was for, who did it and when, and if the data were acceptable.

- Nonconformances and corrective actions such as reprocessing must be documented.

10.2.2 Review of Data Reporting

Review of data reports is required to verify that information reported by an ITAS laboratory corresponds with processed analytical results. Review is only required of the data as it is presented for issuance. Intermediate steps performed after the processed data are checked to prepare the data report (such as data summaries) do not require validation. Referring to Figure 10-1, preparation of the report is the responsibility of the Group Leader.

After the draft data report is prepared (generally in tabular form), the reported results should be checked against the reviewed processed data (not subsequent summaries) so that transcription errors do not occur. The checking process follows:

- Using the draft report, all data entries are checked. The checker can be an Analyst or Group Leader. The checker is not required to be independent of the work because only the transcription from the reviewed data to the data report is being checked.

- The draft data report should be checked so that the items cited for data presentation in Section 11.0 are complete and correct. As the reviewer checks the entries on the draft report, an ink check mark is placed beside each correct entry. Corrected entries are marked through with a single line and the correct entry provided; changes are documented with initials and date. The reviewer will indicate that corrections have been made in the report by placing a second check mark by the correction after comparing the change with the revised copy. The checker shall sign and date every page of the data report in black ink.

- Use of the draft data report results in a checkprint which should be maintained as a record to demonstrate the review.

- If data printouts, such as chromatograms or GC/MS data processing, are included in the data report, review is not required for the data printout.

- If computer output is used directly as the data

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report without further transcription, only the input requires review as discussed in Section 10.2.1.

After checking of the data report is complete, it is given to the Operations Manager for final review. This step is not intended to verify the reported data. This review is intended to determine that the report meets project requirements. From the Operations Manager, the data report is given to the Laboratory Manager for approval to issue.

10.3 VERIFICATION OF SOFTWARE

If computer software is used to acquire, process, or report data, it is necessary to demonstrate that the software correctly performs its intended function.

In general, software is verified by comparing its performance against known results. Verification may be done in several ways:

- If the program has been prepared external to IT and is accepted by ITAS as an "industry standard," independent verification is not required. However, when the program is first used on an ITAS system, available example problems should be processed to demonstrate that the program is fully operational. Example problems must test the capabilities of the software. Industry standard programs are defined as those which are widely used throughout the profession and are brought into ITAS and used without modification.

- For programs that are developed within ITAS, and externally prepared programs that are modified by ITAS, complete checking of performance is required. Checking is dependent upon the function of the software and could include:

  - For software that only performs numerical manipulation, sample sets of numbers for which the results are known should be processed and compared. In this case, known results are

usually generated by performing hand calculations using the same equations and procedures as the software. Verification of the software must test all options of the program. Problems must test both the theory, or basis for computation, and the ability of the software to store and manage files.

- Software that performs as part of instrument operation should be verified by processing reference materials through the instrument system. Processed instrument response should be compared against the standards used.

Whenever a program is modified, reverification is necessary. If the software has had features added, previous test problems should be rerun to demonstrate their function has not been affected. New test problems should be processed as discussed above to verify added performance. If software revision changes the basic operation of the program, complete reverification of the program is required.

Software should be reverified on a semiannual basis. The test problems used to provide initial verification shall be reprocessed and the results compared to demonstrate that performance of the software is unchanged. If software performance has changed, the effect of the change upon intended function and usage since last verification shall be assessed. "Effect" must be determined on a case-by-case basis for the scope and impact of incorrectly reported results. If necessary, the data shall be reprocessed and recipients of affected data reports notified.

Software verification shall be documented by the individual performing the work, signing and dating in ink the computer output, and supporting calculations. If test problems are used, the input shall be marked with check marks to indicate correct usage and the output checked to indicate acceptable comparison. If reference materials are used as the basis for

verifying instrumental related software, the "true" values or certificates for the materials shall be included with the output to demonstrate performance.

All software verifications, whether for initial or subsequent revalidation, shall be maintained in the laboratory operations records management system as discussed in Section 12.0. A separate file shall be maintained for each program. The file shall include the basis for the verification, such as the test problems or hand calculations, results of the software performance, and the results of subsequent reverifications.

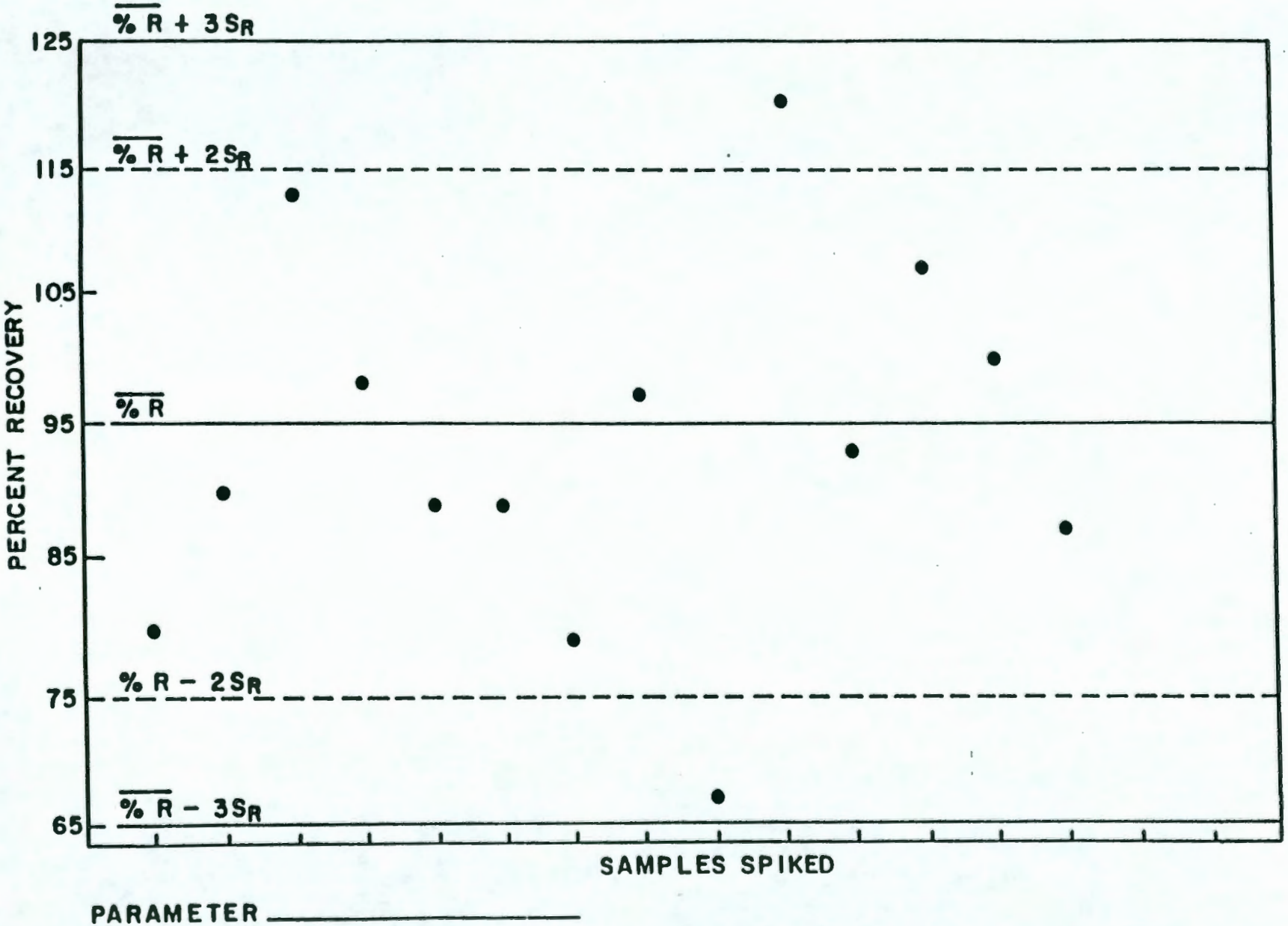

FIGURE 10-1

EXAMPLE ACCURACY QUALITY CONTROL CHART

ACTIVITY	RESPONSIBILITY
Data Processing, Corrections Reviewed, Results Amended	Analyst
Checking of Data Processing; Back check with originator	Independent Analyst or Group Leader
Preparation of Data Summaries and Draft Reports	Group Leader
Checking of Draft Data Report	Group Leader or Analyst
Final Review	Operations Manager
Approval for Issue	Laboratory Manager

FIGURE 10-2

DATA VALIDATION PROCESS

9 1 1 2 3 6 4 0 2 4 4

TABLE 10.1A HISTORICAL PRECISION AND ACCURACY DATA/WATER [a]

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP RAS METHODS

ANALYTES	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % BIAS
BENZENE	624 (GC/MS)	11 ug/l	16	0
		480 ug/l	21	-16
	8240 (GC/MS)	5-100 ug/l	21	12
BROMODICHLOROMETHANE	624 (GC/MS)	8 ug/l	26	-8.8
		480 ug/l	16	-6.7
BASE SYSTEM	501.1 (PURGE & TRAP GC)	0.9 ug/l	66	0
		550 ug/l	34	-3.8
	501.2 (EXTRACTION GC)	1.8 ug/l	61	33
		170 ug/l	23	-19
BROMOFORM	624 (GC/MS)	9 ug/l	32	-23
		400 ug/l	30	10
	501.1 (PURGE & TRAP GC)	4.8 ug/l	44	-27
		550 ug/l	41	7.5
	501.2 (EXTRACTION GC)	8 ug/l	14	-23
		170 ug/l	15	1.8

TABLE 10.1A: HISTORICAL PRECISION AND ACCURACY DATA/WATER [a]
(continued)

LEVEL III ANALYTICAL TECHNIQUES - METHODS OTHER THAN CLP BAS METHODS

ANALYTES	METHOD (TECHNIQUE)	CONCENTRATION RANGE	PRECISION % RSD	ACCURANCY % BIAS
METHYLENE CHLORIDE	624 (GC/MS)	7.2 ug/l	78	-17
		480 ug/l	52	-25
TOLUENE	624 (GC/MS)	13.5 ug/l	19	15
		600 ug/l	31	-14
	8240 (GC/MS)	25 ug/l	19	-10
		75 ug/l		
TRICHLOROETHENE	624 (GC/MS)	5.4 ug/l	48	44
		360 ug/l	39	-2.3
	8240 (GC/MS)	25 ug/l	24	5
		75 ug/l		
LEAD	200.7 (ICP)	42 ug/l	34	31
		47.7 ug/l	5	4.4
	239.1 (FLAME AA)	12 ug/l	5.9	17
		105 ug/l	6.7	-1.9
	239.2 (FURNACE AA)	10 ug/l	53	-.22
		234 ug/l	19	-3.1

a. Source: Draft Compendium of Information and Performance Data on Routinely Used Measurement Methods (RUMM) - Pilot Phase, RTI/3087/03, prepared for EPA Quality Assurance Management Staff, January 1986. This document should be consulted for more information on individual analytes.

TABLE 10.1B: HISTORICAL PRECISION AND ACCURACY DATA/WATER

LEVEL III ANALYTICAL TECHNIQUES - SW-846 METHODS

Method Number	Method Name	Data Source	Range of Recovery (%)	Precision (%)	MDL (mg/l)
8310	Polynuclear Aromatic Hydrocarbons (HPLC) (Capillary)	SW 846	78 - 116	7.3 - 12.9	0.03 - 2.3
INORGANICS:	Metals (ICAP)	EPA 200.7	NA	3 - 21.9 (RSD)	1.3 - 75 Mg/l
	Metals (FLAME) 7000 Series	EPA 200	NA	NA	0.01 - 5
7000 Series	Metals (FLAME LESS/GF)	EPA 200	NA	NA	0.001 - 0.2 Mg/l
7470	Metals (MERCURY)	EPA 245.2	87 - 125	0.9 - 4.0	0.0002
9010	Cyanides	EPA 335.2	85 - 102	0.2 - 15.2	0.02 Mg/l
9030	Sulfides	EPA 376.1	NA	NA	1 Mg/l

a. For water only

b. NA Not Available

NOTES: Method Detection Limit (MDL) as listed on this table is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

Accuracy, presented as an average percent recovery, was determined from replicate (10-25) analyses of water and wastewater samples fortified with known concentrations of the analyte of interest at or near the detection limit. In most cases this was less than 10 times the MDL.

Precision data are used to measure the variability of these repetitive analyses reported as a single standard deviation or, as a percentage of the recovery measurements. For presentation purposes accuracy, precision and MDL information is presented as an average range of individual values for every analyte covered by the procedure. If specific information on a particular compound is required, the specific analytical method cited should be consulted.

TABLE 10.1C: HISTORICAL PRECISION AND ACCURACY DATA/WATER

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % Bias
Semivolatiles[d]	GC/MS	N.A.[c]		
4-Methylphenol			33	-36
N-Nitroso-di-n-propylamine			31	+0.3
Nitrobenzene			32	-23
Isophorone			23	-0
2-Nitrophenol			30	-21
bis(2-Chloroethoxy)methane			34	-2.6
2,4-Dichlorophenol			29	-20
1,2,4-Trichlorobenzene			30	-47
Naphthalene			44	-30
4-Chloro-3-methylphenol			26	-32
2,4,6-Trichlorophenol			25	-17
2-Chloronapthalene			24	+3.4
Acenapthene			28	-12
2,4-Dinitrophenol			24	-23
2,4-Dinitrotoluene			34	-33
2,6-Dinitrotoluene			25	-40
4-Chlorophenyl-phenylether			34	+12
Fluorene			25	-24
4,6-Dinitro-2-methylphenol			30	-13
4-Bromophenyl-phenylether			32	-0.1
Hexachlorobenzene			36	-42
Pentachlorophenol			31	-24
Phenanthrene			21	-28
Fluoranthene			42	-15
Benzo(b)fluoranthene			39	-10
Benzo(a)pyrene			42	-29

TABLE 10.1D: HISTORICAL PRECISION AND ACCURACY DATA/SOILS[a]

LEVEL IV ANALYTICAL TECHNIQUES - CLP RAS METHODS

ANALYTES	TECHNIQUE	CONCENTRATION RANGE	PRECISION % RSD	ACCURACY % Bias
Volatiles[b]	Purge & Trap GC/MS	N.A.[c]		
Chloroform			8.0	-0.1
1,2-Dichloroethane			13.1	+11.1
Dibromochloromethane			35.0	-12.0
Benzene			32.1	-10.3
Bromoform			16.6	-12.1
2-Hexanone			16.6	-45.5
Toluene			13.8	+13.7
Chlorobenzene			21.2	+13.2
Semivolatiles[d]	GC/MS	N.A.[c]		
1,4-Dichlorobenzene			27	-51
Nitrobenzene			21	-48
Isophorone			24	-47
2-Nitrophenol			35	-36
2,4-Dichlorophenol			31	-59
1,2,4-Trichlorobenzene			28	-43
Penta Chlorophenol			17	-40
Pyrene			25	-15
2-Methylnaphthalene			26	-42
bis-(2-Ethylhexyl)phthalate			33	-2
Phenol			38	-27
Acenaphthylene			26	-27
Diethyphthalate			16	-20
Dioxin[e]				
2,3,7,8-TCCD		1-10 ug/kg	15	-11.5

TABLE 10.2A: HISTORICAL PRECISION AND ACCURACY DATA/SOILS

LEVEL I FIELD SCREENING TECHNIQUES

MEASUREMENT	INSTRUMENT (TECHNIQUE)	INSTRUMENT RANGE	INSTRUMENT PRECISION [b]	INSTRUMENT ACCURACY [c]
RESISTIVITY	Bison 2390 T/R (Resistivity meter)	0-1999 millivolts	at 1% range setting, 0-5% of full scale	2% of measured value
TERRAIN CONDUCTANCE	EM 31 (conductivity)	0-1000 millimhos/meter	2% of full scale	5% at 20 millimhos/meter
TERRAIN CONDUCTANCE	EM 34-3 (conductivity)	0-300 millimhos/meter	2% of full scale	5% at 20 millimhos/meter
Magnetic Field Intensity	EDA - Omni IV (Magnetometer)	18000-110000 gammas	0.02 gamma	1 gamma at 50000 gammas at 23oC
Subsurface Lithology Changes	SIR-8 (Ground Penetrating Radar)	1-81 dielectric constant	N/A[d]	N/A[d]
Subsurface Lithology Changes	EG+G 1225 (Seismograph)	0-2000 milliseconds	N/A[d]	0.01%

TABLE 10.2B: HISTORICAL PRECISION AND ACCURACY DATA/AIR[a]

LEVEL I FIELD SCREENING TECHNIQUES[b]

ANALYTES	INSTRUMENT (TECHNIQUE)	INSTRUMENT RANGE	INSTRUMENT SENSITIVITY[c]	INSTRUMENT PRECISION[c]
Organics	Century OVA-128 (Flame Ionization)	0.1 - 1000 ppm Methane	0.1 ppm Methane	N.A.[d]
Organics	HNu PI-101 (Photoionization)	0.1 - 2000 ppm Benzene	0.1 ppm Benzene	± 1% of full scale deflection
Organics	AID - 710 (Flame Ionization)	0.1 - 2000 ppm Methane	0.1 ppm Methane	N.A.[d]
Organics	PhotoVac (GC-Photoionization)	N.A.	0.001 ppm Benzene	N.A.[d]

a. Source: Manufacturers' manuals unless otherwise cited. Mention of specific models does not constitute an endorsement of these instruments.

b. It is difficult to differentiate between Level I and Level II techniques and instrumentation. Several instruments may be used at both levels.

c. Sensitivity and precision refer to instrument specifications.

d. N.A. = Not Available.

TABLE 10.2C: HISTORICAL PRECISION AND ACCURACY DATA/SOIL[a]

LEVEL II FIELD TECHNIQUES

ANALYTES	INSTRUMENT (TECHNIQUE)	FIELD RESULTS IN ppm (x)	CLP RESULTS IN ppm (y)	ACCURACY[b] % BIAS
PCBs	HNu 301 (GC/ELECTRON CAPTURE)	6.0	22.0	−72.7
		6.0	6.1	−1.6
		6.0	510.0	−98.8
		9.0	3.9	+56.7
		13.0	3.0	+333.3
		14.0	3.1	+351.6
		14.0	23.5	−40.4
		21.0	8.1	+159.3
		35.0	7.7	354.5
		41.0	2.1	+1,852
		48.0	11.0	+336.3
		50.0	480.0	−89.1
		65.0	23.1	+181.4
		87.0	18.7	+256.3
		92.0	75.0	22.7
		95.0	30.0	+216.7
		11	12.3	−10.6
		202	99.0	+104.0
		269	370.0	−27.3
		286	80.5	+255.3
		1215	640.0	+90.0
		1647	1040.0	+58.4
		3054	9,300	−67.2.

a. Source: CDM Project files.

b. Source: Accuracy calculated by assuming that CLP results on the same samples were completely accurate. % Bias = 100 $\frac{(x-y)}{y}$

11.0 DATA REPORTS

The format and content of a data report are dependent upon project needs, such as: whether or not explanatory text is required, client or contract requirements, and government agency reporting formats. The ITAS Quality Assurance Program does not specify a report format; however, the following are applicable to data presentation:

- The final data presentation shall be checked in accordance with data verification requirements of Section 10.2.2 and approved by the Laboratory Manager.

- Data are presented in a tabular format whenever possible.

- Data are generally formatted as a Certificate of Analysis such as shown on Figure 11-1, or formatted as an IT Corporation memorandum if issued within IT. Figure 11-2 is an example of a computer-generated report from the ITAS Sample Management System (SAM).

- Each page of data is identified with the project number and name; date of issue; and, if appropriate, client name.

- Data presentation includes:

  - Sample identification number used by the ITAS laboratory and/or the sample identification provided to the laboratory, if different than identification used in the laboratory

  - Chemical parameters analyzed, reported values, units of measurement, and analytical method used for the types of analysis specified

  - Detection limit of the analytical procedure if less than the detection limit is reported (e.g., <DL (10) where 10 is the detection limit)

  - Data for a chemical parameter reported with consistent significant figures for all samples (See Guidance, ASTM E29-67(80) and Standard Methods 103)

  - Results of Quality Control samples analysis if appropriate (See Section 8.3, Quality Levels)

9 1 1 2 7 6 4 0 2 5 3

- Achieved accuracy, precision, and completeness of data if appropriate

- Footnotes referenced to specific data if required to explain reported values.

- Data should be released from the laboratory only by the Laboratory Manager or Operations Manager.

If explanatory text is not issued with the analytical results, it is recommended that:

- A letter of transmittal be included for external clients

- A memorandum be included for work performed within IT.

As necessary, the letter of transmittal/memorandum should include:

- Person(s) receiving the data

- Person transmitting the data

- Document if the chain of custody was not provided or correct, if any samples were damaged in shipment, if sample containers were inappropriate for analysis, or if volume provided was inadequate for proper analysis

- Brief discussion of samples analyzed and the analytical program

- Discussion of any apparent data anomalies

- Discussion of any analytical difficulties.

Any analytical results verbally communicated are considered preliminary until data are presented in hard copy, with a formal letter of transmittal or memo accompanying the results.

9 1 1 2 7 6 4 0 2 5 5

FIGURE 11-1

EXAMPLE - CERTIFICATE OF ANALYSIS

IT Santa Clara ANALYTICAL REPORT

COMPANY
FACILITY

REPORT
TO

ATTEN

PREPARED BY INTERNATIONAL TECHNOLOGY CORPORATION
397 Mathew Street
Santa Clara, CA 95050
PHONE (408)727-4277
CONTACT JIM

WORK ID
LABORATORY WORK ORDER # S7-06-157
LABORATORY JOB #
RECEIVED 06/19/87 REPORTED 06/26/87

SAMPLES 5 Waters
P.O. #
CERTIFIED BY

Duplicate of report of 06/25/87.

TABLE OF CONTENTS FOR ANALYTICAL RESULTS

PAGES	LABORATORY #	SAMPLE IDENTIFICATION
	S7-06-157-01	D4316 TRIP BLANK(6/15)6/18
2	S7-06-157-02	D4661 VINION DOM
2	S7-06-157-03	D4662 ITM7C
2	S7-06-157-04	D4314 ITM9C
2	S7-06-157-05	D4315 VINION DOM

FIGURE 11-2

EXAMPLE - COMPUTER GENERATED REPORT

&FOOOBH.PRN
06/26/87

IT Santa Clara ANALYTICAL RESULTS

COMPANY
FACILITY

WORK ID
LABORATORY WORK ORDER # S7-06-157
LABORATORY JOB #
RECEIVED 06/19/87 REPORTED 06/26/87

SAMPLE / TEST	D4661 VINION DOM S7-06-157-02	D4662 ITM7C S7-06-157-03	D4314 ITM9C S7-06-157-04	D4315 VINION DOM S7-06-157-05	UNITS	NORMAL METHOD LIMIT
1,1,-dichloro-ethane	nd @ 0.2	nd @ 0.5	nd @ 0.2	nd @ 0.2	ug/L	
1,1,-dichloro-ethylene	nd @ 0.1	nd @ 0.2	nd @ 0.5	nd @ 0.1	ug/L	
1,2,-dichloro-ethane	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Benzene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Ethyl Benzene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Tetrachloro-ethylene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
1,1,1-Tri-chloroethane	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Trichloroeth-ylene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Toluene	nd @ 0.2	nd @ 0.5	nd @ 0.5	nd @ 0.2	ug/L	
Total Xylene	nd @ 1.0	nd @ 1.5	nd @ 1.5	nd @ 1.0	ug/L	

FIGURE 11-2 continued

EXAMPLE - COMPUTER GENERATED REPORT

12.0 RECORDS MANAGEMENT

The ITAS Quality Assurance Program has been developed to provide analytical results of known quality. To demonstrate that quality has been achieved, each ITAS laboratory maintains a records management system that includes documents which demonstrate the analytical performance of the laboratory.

Laboratory records are maintained in two broad categories:

- Documents which are specific to a project or a group of samples within an ongoing project, such as chain of custody and raw analytical data.
- Documents which demonstrate overall laboratory operation, such as instrument log books and control charts. These records will directly affect the data for a specific project, but in general their applicability is not limited to one project.

Following is a description of the records system used as the model within ITAS Laboratories. If it is necessary for a specific laboratory to deviate from this system, the laboratory shall maintain a description of their system as a Laboratory-Specific Attachment to the Quality Assurance Manual. Also, ITAS recognizes that specific regulatory or contractual demands may require deviation from the system described herein. In these instances, records shall be maintained as externally required.

The criteria for an acceptable records management system are that the data must be secure, retrievable, and complete. All laboratory records from time of sample receipt through reporting and disposal must be available and stored in a manner that safeguards their integrity from tampering or physical damage and loss. Any documentation that bears on the reported results must be available if requested by the client or an authorized regulatory agency or court. This includes operational and project-specific data. Data may be stored in "real-time" as it is

9 1 1 2 7 4 0 2 5 8

produced, or filed in a manner to allow prompt retrieval and assembly into a complete project file as described in the following section. Laboratory-Specific Attachments to this Quality Assurance Manual must describe therein (or by reference to SOPs provide the details) how the complete set of documentation is compiled, including the flow of data forms, locations, responsibilities, and checks on the records management system implemented.

12.1 PROJECT RECORDS

Separate record packages are maintained for each project or are retrievable so that packages can be assembled properly. Filing of records for a specific project shall be by the unique project identification number assigned by the laboratory for that project. Within a project file, categories of information are filed separately.

Table 12.1 presents the categories used within a project file. It is not expected that all categories will be applicable for every project. However, in all project files, specific information will be filed in accordance with the category designation provided. Following is a brief discussion of each category:

Project Index

Each project file shall have an index that lists each record unit within the project file.

Figure 12-1 presents an example of a project index page. An individual index file page should be used for each category applicable to a project. Within a specific category, individual files should be given a unique file number and be listed on the file index page. For example, if a testing program involves distinct groups of samples over a period of time, file number B1 should contain the chain of custody from the first group, B2 the chain of custody for the second group, etc. A brief description of the file contents should be made. Such as for the chain

of custody records for a group of samples, the description could be the dates the samples were collected.

A - Correspondence

All correspondence pertinent to the analytical program shall be maintained. This includes letters to and from clients and internal memorandums. Correspondence should be filed chronologically. Data reports and analysis requests are maintained in separate categories. The correspondence file should be updated as necessary. Each item of correspondence should not be entered in the project index. Rather, the index should list blocks of dates, such as for file A1, the description could be July 30, 1982 to January 10, 1983; for file A2, the description could be January 11, 1983 to September 17, 1983, etc.

B - Chain of Custody

Chain of custody records shall be maintained by the laboratory. The chain of custody forms should be filed for groups of samples as received and should be placed in the project file immediately after they are signed by the Quality Control Coordinator. When the samples are disposed of, this entry shall be made on the appropriate chain of custody form. Chain of custody records for samples sent to other laboratories must be maintained so the custody chain is unbroken.

C - Field Records

All field records supplied by field personnel pertinent to the analytical program shall be maintained. This could include the results of field tests or logs of sample collection. It is the responsibility of field personnel to provide records to the laboratory.

D - Request for Analysis

Analysis requests provided by field personnel are maintained in this file. Also, any changes or additions to the analytical program should be documented in this file.

E - Calibration Records

In general, calibration records are maintained with laboratory operation records. However, if an analytical program requires calibration which is performed solely for a project, the records shall be maintained in this file. If calibration is performed as an integral part of the analytical process; the calibration records should be maintained with the analytical data.

F - Analytical Data

Analytical data files should be complete for a group of samples in accordance with the needs of the project. The file should contain raw analytical data, processing of the data and/or data reduction, and data validation. It should be possible to use a data file to completely demonstrate that the data have been adequately obtained, processed, and reviewed. It is recommended that data be filed in accordance with the type of analysis performed. Suggested subcategories for data are presented in Table 12.1.

G - Quality Control Samples

If Quality Control samples, such as field blanks, are processed for a specific project, the data shall be maintained with the project file. The results of Quality Control samples processed on a general basis are included in the laboratory operations files. Statistical evaluation of Quality Control sample data for a project shall also be maintained in this file.

If Quality Control samples are processed as an integral part of a group of samples such that the data cannot be readily separated, the Quality Control sample data can be stored with the analytical data.

H - Data Reports

Complete copies of all reports issued by the laboratory shall be kept in the project file. With the reports shall be the review copy checked to

verify the data. If the report provides copies of data packages which are stored in Category F - Analytical Data, a memorandum should be attached to the report copy stating which specific files were included.

I - Project-Specific Requirements

If a project requires analytical procedures other than these adopted in the ITAS Quality Assurance Program, the requirements shall be included in this file. Specific requirements may be due to government regulation, specific contracts, or project need. Changes from stated practice can be, for example, frequency of QC sample analysis, test method, statistical data evaluation, and reporting format.

J - Nonconformances

Nonconformances and subsequent corrective actions which are specific to a project are included in this file. The record should be in the form of a memorandum (or copy of other records discussed in this manual) with the nonconformance stated, how it was corrected, and the approval for the correction. A separate file for each incidence is not required, the file should be maintained chronologically. A copy should be printed for the master nonconformance record (Section 12.2).

K - QA Plans

If specific Quality Assurance Plans, and revisions, are prepared for a project, they shall be stored in this file.

L - Miscellaneous

The miscellaneous file includes all records not applicable to the previous categories. Each distinct record(s) in this file should be entered in the project index.

M - Method Description

A written statement identifying the method(s) used for the project sample analyses, including the reference to approved methods and any approved modifications to the methods.

9 1 1 2 7 6 4 0 2 6 2

If it is necessary to adopt a new analytical procedure, a procedure different than conventionally used, or alter an existing procedure, the method used for the project must be documented. If the analytical procedure is developed by an ITAS laboratory as part of the analytical program, the procedure shall be documented and included. If an existing procedure is altered, the Analyst or Group Leader shall prepare a memorandum to the project file stating what the changes were and the justification for change.

N - Work Order Records

Internal tracking documents used by the laboratory (e.g., computer-generated daily status reports, etc.) must be included as part of the traceability of the project samples through the laboratory. If a duplicate manual log is maintained, traceability of the systems is required to show the same information is contained and accessible in both.

O - Subcontractor Records

Correspondence and reports and raw data (samples, calibration, and QC) must be received from the subcontractor following analysis reporting. Arrangements to retrieve these data are required in the ITAS analytical subcontracting policy, and must be followed to achieve complete project files.

12.2 GENERAL LABORATORY OPERATIONS RECORDS

General laboratory records document overall laboratory performance and operation. These records are filed separately from project records and will be maintained so they can be referenced to project records if necessary. Examples of general records pertinent to project records are instrument log books and computer software verifications.

9 1 1 2 7 5 4 0 2 6 3

There are two types of general laboratory records:

- Documents which demonstrate laboratory performance
- Reference documents for laboratory operations.

Records which demonstrate laboratory performance shall be filed in categories in a manner similar to project files. Reference documents are not indexed and their usage is not controlled.

Table 12.2 recommends the laboratory performance records to be maintained and the category system for their maintenance. Also shown in this table is the manner in which the files can be correlated to project files, if required. Indexing of files should be as described in Section 12.1.

Many of the laboratory operations records are in daily use, such as the Master Sample Log Book, instrument calibration logs, and control charts. It is not intended that the records be stored daily while they are in use. However, when individual log books, etc., are filled, they shall be placed in the files. For a book that is in use, it should be designated by a file index code and entered into the index. For example, considering the Master Sample Log books, the book for the period August 30, 1983 to April 4, 1984 is designated A1. Because it is complete, it is stored. Book A2 starts on April 5, 1984 and is still in use; therefore, the index would list A2, April 5, 1984 to _____. The completion date would be entered when book A3 is started.
Following is a brief discussion of the categories:

Index File

The index should be maintained as discussed in Section 12.1.

A - Master Sample Log Book

The master sample log books chronologically record all samples entering the laboratory, independent of project designation.

B - Instrument Calibration Logs

All calibration performed independent of a specific project shall be recorded by instrument. A separate file should be maintained for each instrument subject to calibration.

C - Instrument Maintenance Logs

Separate maintenance files should be kept for each instrument incorporated in the preventive maintenance program. The file shall include records of maintenance performed in-house or by outside groups. Service contracts should be included in the file for the applicable instrument.

D - Computer Software Verification

Copies of the data used to verify performance of software shall be maintained. For software that is periodically reverified with standard problems, the results of the performance shall be maintained. A separate file for each software package shall be indexed.

E - Performance Evaluation Records

Laboratory participation in Performance Evaluation Programs shall be documented in this category. If performance standards are analyzed as part of the overall Quality Control sample program, the results should be included in Category G.

F - Certification Program Records

If the laboratory participates in Certification programs, such as the AIHA program, the results shall be maintained in this category. Records should include all correspondence, analytical data, agency results, and certificates of performance.

9 1 1 2 7 6 0 2 6 5

G - QC Sample Analysis

This file should include the results of all QC samples inserted into the sample stream by the QC Coordinator. It is recommended that the files be maintained chronologically for each parameter included in the QC program.

H - Control Charts

Control charts should be filed chronologically for each parameter monitored.

I - Purchased Material Certificates

All information which verifies that purchased materials meet the requirements of the laboratory should be maintained. Certification may be supplied by a vendor or from in-house verification analysis. Separate files should be kept for chemicals, gases, water, glassware, etc.

J - QC Coordinator Reports

This file includes the monthly reports prepared by the QC Coordinator. Required signatures must be included. The file should be maintained chronologically.

Table 12.3 lists reference documents which should be available within the laboratory. These documents need not be included in the laboratory record system and may be kept where used. Reference documents shall be revised and updated as necessary to maintain them as "currently applicable documents."

K - Audit Records

Formal audit reports of internal system and performance audits are to be filed in this category, by date and topic (items audited). Where the audit is project-specific, it should be cross-referenced to the project audited (see Project Records Filing Category I). If the audit was performed by an external agent, a copy of the audit report should be

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included in this category; if the audit was project-specific, it should be filed in the Project Records Filing Category I by date and project number and/or client.

L - Personnel Files

Training files of individuals are to be maintained current in this filing category. Records of SOP training received, proficiency tests passed, external training obtained, etc. are documented in this category chronologically by individual and by topic.

M - SOPs

A listing of the current laboratory Standard Operating Procedures (SOP) is maintained as an index, together with a complete collection of approved SOPs, their issue dates, and individuals authorized to perform the SOPs (see category L above); if SOP manuals are issued, the distribution records are maintained therein, by number and topic.

N - Methods

Those methods used by the laboratory are summarized, indexed, and compiled to provide a complete reference collection of the analytical procedures used. Method references are listed by reference method name and number and source, within the laboratory groups or classes of instruments tested (GC, GC/MS, AA, ICP, Wet Chemistry, etc.).

O - Subcontractor Records

Data received from subcontractors from analysis of audit (PE) samples, routine QC samples, blind samples submitted to them, and other information establishing the laboratory's performance levels are maintained in this category by subcontractor and project number.

P - Master Nonconformance Record

As described in Section 12.1, a copy of project-specific (and other) nonconformances and corrective actions is to be supplied to the Quality Control Coordinator. This Master Record will be periodically examined and reports issued describing systematic trends which in turn require investigation and correction by responsible Group Leaders, the Technical Director, the Operations Manager, or the Laboratory Manager. Group Leaders are encouraged to maintain Group Records to perform such evaluations regularly.

12.3 RECORD CONTROL

The Laboratory Manager shall designate an individual responsible for the records management system. This person shall:

- Initiate new project files including the project index
- Add new records to existing files, initiate new files within a category, and update the index
- Assist laboratory personnel in withdrawing and returning records.

To maintain control of records within the laboratory, a master sign-out sheet should be maintained. The sign-out sheet should indicate:

- Project from which file is borrowed
- File designation, such as E13, etc.
- Date and person borrowing record
- Date returned to the record system.

12.4 RECORD RETENTION

ITAS will maintain records associated with specific projects for the following time periods after completion of the project:

- Analysis performed as part of site mitigation activities - 10 years

9 1 1 2 7 6 4 0 2 6 3

- Records associated with facilities governed by the Resource Conservation and Recovery Act (RCRA) - 5 years after closure, if the analysis was performed prior to closure or for the 30-year monitoring period following closure, if the analysis was performed for the purpose of closure monitoring

- Conventional analysis - 7 years.

If necessary, pertinent supplemental records in the laboratory operations record system will be added to the affected project file.

If a specific contractual requirement, project demand, or government regulation requires that records be maintained for a longer period of time, project files will be kept as required. For projects that must be kept beyond the periods stated above, the project index shall be marked to indicate the required retention period.

Prior to scheduled record destruction, records shall be reviewed to determine if the holding period should be extended.

12.5 SAMPLE STORAGE

Analytical samples will be routinely stored (regardless of reason for analysis) by an ITAS laboratory, after submittal of a data report, for six weeks prior to disposal or return to client.

Samples will be stored for different periods if specified by project or contractual requirements.

Table 12.1
EXAMPLE PROJECT RECORDS
FILING CATEGORIES

CATEGORY	RECORD DESCRIPTION
-	Project Index
A	Correspondence
B	Chain of Custody
C	Field Records
D	Request for Analysis
E	Calibration Records
F	Analytical Data
Example: F-1	- GC Data
Example: F-2	- GC/MS Data
Example: F-3	- Metals
Example: F-4	- General Chemistry
Example: F-5	- Soils Chemistry
Example: F-6	- Other
G	QC Samples
H	Data Reports
I	Project-Specific Requirements
J	Nonconformances
K	QA Plans
L	Miscellaneous
M	Methods Descriptions
N	Work Order Records
O	Subcontractor Records

Table 12.2
EXAMPLE LABORATORY PERFORMANCE RECORDS
FILING CATEGORIES

CATEGORY	RECORD DESCRIPTION	CORRELATION TO PROJECT FILES
-	Index File	--
A	Master Sample Log Books	By date of arrival and sample number
B	Instrument Calibration Logs	By date, instrument serial number, and analyst
C	Instrument Maintenance Logs	By date and instrument serial number
D	Computer Software Verification	By date and software designation
E	Performance Evaluation Records	By date and sample designation
F	Certification Program Records	By name of program, date, and sample designation
G	QC Sample Analysis	By date and sample number
H	Control Charts	By chemical parameter, date, and sample number
I	Purchased Material Certificates	By date and parameter
J	QC Coordinator Reports	By date
K	Audit Records	By date and topic
L	Personnel Files	By individual, chronologically
M	SOPs	By numbered topic
N	Methods	By method and group
O	Subcontractor Records	By subcontractor and project
P	Matter Nonconformance Record	By group, chronologically

Table 12.3
REFERENCE DOCUMENTS

- Instrument Manuals
- Computer/Software Instruction Manuals
- Analytical Procedures
- QA Manual/Laboratory-Specific Attachments
- Manuals of Practice

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PROJECT INDEX

PROJECT NUMBER _____
PROJECT NAME _____

FILE CATEGORY _____

FILE DESIGNATION	DESCRIPTION

FIGURE 12-1
EXAMPLE
PROJECT INDEX FILE

9112160273

13.0 NONCONFORMANCES AND CORRECTIVE ACTION

A deficiency is an unauthorized deviation from documented procedures, practices, or standards, or a defect in an item, that could lead to degradation of quality.

A nonconformance is a deficiency in characterization documentation, or procedure sufficient to render the quality of an item unacceptable or indeterminate or any event which is beyond the limits documented and established for laboratory operation. Non-conformances may be caused by non-Laboratory operations (e.g., field collection paperwork not complete) and be identified at the Laboratory.

Nonconformances may include (but are not limited to) the following:

- Failure of an instrument to work properly
- Sample receiving documentation not correct
- Sample condition on receipt not acceptable
- Sample holding time exceeded
- Sample storage conditions outside criteria
- Incorrect sample preparation/analysis procedures used
- QC sample data (blank, spike, duplicate, surrogates, etc.) outside limits
- Calibration requirements not met
- Data recording errors, transcription errors, or failure to document
- Data validation errors
- A recovery or RPD result that is out of control (e.g., more than three standard deviations from the weighted mean for that month)
- Relative standard deviation for response factors greater than accepted limits

9 1 1 2 3 4 0 2 7 4

- Positive blanks
- Any situation or result which might affect the quality of data.

A nonconformance memorandum is formal documentation of a nonconformance that includes a description of the problem, the corrective action taken, the individual recognizing the problem and the date discovered, the sample analyses affected (if any) and the initials of appropriate reviewers and the Quality Control Coordinator (QCC) (see examples in Figure 13-1 through 13-3).

A corrective action is an appropriate measure applied to correct a deficiency and minimize the possibility of recurrence.

Corrective action will include, but not necessarily be limited to:

- Recalibration of instruments, using freshly prepared calibration standards
- Reanalysis of samples
- Replacement of lots of solvent or other reagents that give unacceptable blank values
- Additional training of laboratory personnel in correct implementation of sample preparation and analytical methods
- Reassignment of personnel, if necessary, to improve the overlap between operator skills and method requirements
- Communication with the clients to determine the appropriate action (e.g., insufficient sample remaining for reanalysis).

13.1 RESPONSIBILITIES

All employees of the Laboratory staff are responsible for reporting nonconformance they observe/identify.

Employees identifying problems that might affect quality are responsible for reporting them to the appropriate supervisor and signing nonconformance memos.

9 1 1 2 7 4 1 0 2 7 5

Each laboratory group leader is responsible for documenting and correcting problems that might affect quality in accordance with the requirements of this procedure. The supervisor is responsible for preparing and signing nonconformance memos, issuing nonconforming item tags, stopping work in the event of out-of-control situations, and notifying the QCC of nonconforming items. With the cognizant QCC, the supervisor is responsible for determining whether reported problems are nonconformances, concurring with proposed corrective action, and notifying the QCC that corrective action has been completed.

The QCC is responsible for reviewing nonconformance memos, recommending or approving proposed corrective actions, maintaining an up-to-date nonconformance log, verifying that corrective action has been completed, releasing nonconforming item tags, distributing and filing nonconformance memos, and assisting in resolving disagreements. With the lab management and group leaders, the QCC also is responsible for determining whether reported problems are nonconformances, whether operations need to be stopped, and establishing schedules for completion of corrective action. The QCC is responsible for assisting in resolving disagreements and quality problems, and performing audits of all affected organizations for compliance with the requirements of this procedure.

13.2 GENERAL PROCEDURE

The corrective action process is considered internal when the deficiency is identified by a laboratory employee, whether that employee works in the responsible group or is independent of it (the QCC, for example). External corrective action results when a deficiency is identified by an external organization, particularly through quality assurance functions such as audits.

13.3 INTERNAL NONCONFORMANCE CORRECTIVE ACTION PROCEDURE

Any laboratory employee noticing a deficiency suspected of being a nonconformance shall report the deficiency to the responsible supervisor and to the QCC on a nonconformance memo form.

If the QCC and the responsible supervisor determine that quality is affected, the deficiency becomes a nonconformance and the remainder of this procedure shall be implemented.

If the nonconformance is hardware/equipment related, the item shall be labeled with a nonconforming item tag (Figure 13-4) and segregated if possible so that it is not used until corrective action has been taken. Either the responsible supervisor or the QCC may issue a nonconforming item tag.

If analytical data (chromatograms, spectra, calculation sheets, etc.) that have not been completely processed or reported to other organizations are determined to be in error because of equipment malfunction, defective material, failure to follow procedures, anomalies in the data, process errors, quality control sample failure, or other errors, and the non-conforming data are to be maintained for historical purposes, a nonconformance memo shall be initiated describing the error, listing and attached to the affected data sheets. After confirmation of the problem as a nonconformance by the responsible supervisor and/or the QCC, each page of affected data is lined through, marked as rejected data, initialed and dated to prevent subsequent use in processing. A copy of the completed nonconformance memo is filed with the rejected data in the appropriate analytical data category of the project file. If there is justification for disposition of rejected data (the responsible supervisor and/or the QCC shall make this decision), a record of rejected data disposal and brief explanation shall be documented in the analytical run log.

Examples of corrective actions are the modification of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of unqualified personnel, and reanalysis of any affected samples.

The Quality Control Coordinator shall maintain a log of nonconformances that includes a description of the problem and corrective action, and lists the responsible manager, the affected projects and sample numbers, and initial and closeout dates.

Once the corrective action has been taken, the QCC shall verify, through a special surveillance or audit, that the problem has been corrected. The QCC shall document that the corrective action has been completed satisfactorily (closed out) by signing the nonconformance memo and making an entry in the nonconformance log (Figure 13-5) for filing in the Master Nonconformance Record. The QCC can then remove the nonconforming item tag from out-of-service equipment.

The QCC shall file a copy of all records pertaining to the nonconformance with the nonconforming item records (i.e., specific project files or procurement files). The nonconformance log maintained by the QCC shall contain a reference to the file location.

13.4 EXTERNAL NONCONFORMANCE CORRECTIVE ACTION PROCEDURE

Reports of quality assurance functions by external organizations and reports of compulsory proficiency tests are considered to be externally generated.

External reports should be sent to the affected group supervisors and the QCC.

If the report identifies deficiencies that require corrective action, the QCC shall generate a nonconformance memo describing the particular deficiency and make an entry in the nonconformance log. The QCC shall verify that action is taken and that a response or notification of unavoidable delay is generated in time to meet the due date to the client.

The QCC shall verify that the problem has been corrected. With the responsible supervisor, the QCC shall prepare a formal written response to the external organization, if required. The Laboratory Manager shall transmit the

response to the external organization, with copies to other cognizant managers, as deemed necessary.

Upon notification by the external organization of acceptance of the corrective action, the QCC shall notify the responsible supervisor, the Laboratory Manager, and other cognizant managers. If corrective action is not acceptable, the QCC shall start the process over until the problem is satisfactorily resolved, at which time the nonconformance closeout is documented and dated in the nonconformance log.

The QCC will file the records pertaining to the nonconformance with the external audit documents and reference the file location in the nonconformance log.

Nonconformances and required corrective action can also result from the ongoing laboratory review of the QCC and audits performed by the ITAS Quality Assurance/Control Director. These activities are discussed in Section 14.0.

Written laboratory nonconformances shall be reported monthly to the ITAS Quality Assurance/Control Director by the Quality Control Coordinator. Corrective actions for nonconformances that are detected after data have been reported must also be reported to the Director. Figure 13-1 through 13-3 are example forms used to document nonconformance and corrective actions taken. The form is filed in the associated project file(s) and in the QCC's master nonconformance record.

FIGURE 13-1

EXAMPLE NONCONFORMANCE MEMO

SAMPLE RECEIVING

DATE _____
LIMS CODE _____
FILED BY _____

SAMPLE NO.(s) _____
_____

NONCONFORMANCE: [Check applicable item(s)]:

_____ (1) Not enough ample sent for proper analysis.
_____ (2) Sample bottle received broken and/or cap not intact.
_____ (3) Paperwork went to one lab; sample sent to another.
_____ (4) Samples received without proper refrigeration, when it has been deemed necessary.
_____ (5) Illegible sample numbers or label missing from bottle.
_____ (6) Numbers on sample not the same as numbers on paperwork.
_____ (7) Incomplete instructions received with sample(s), i.e., no Request for Analysis, no Chain-of-Custody, incomplete billing instructions, no due dates, etc.
_____ (8) Samples received in improper container.
_____ (9) Physical characteristics different than those on sampling sheets, i.e., two phases.
_____ (10) Rush samples put on hold because of incomplete paperwork.
_____ (11) Standard operating procedure not adhered to (specify) _____
_____
_____ (12) Other (specify) _____
_____

CORRECTIVE ACTION TAKEN: [Check applicable item(s)]:

_____ (1) Sample processed "as is."
_____ (2) Resampling requested.
_____ (3) Sample "on hold" until further notice.
_____ (4) Other (specify) _____
_____

ROUTING

Title	Initials	Date	Check if Corrected
Coding Specialist	_____	_____	_____
QC Coordinator	_____	_____	_____
Lab Supervisor	_____	_____	_____

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FIGURE 13-2

EXAMPLE NONCONFORMANCE MEMO

EXTRACTIONS

DATE _____
LIMS CODE _____
FILED BY _____

SAMPLE NO.(s) _____

NONCONFORMANCE: [Check applicable item(s)]:

_____ (1) Method development or modification, to include any extraction or cleanup sequence not currently used on a regular basis in the extractions lab. (Requires QA approval.) PLEASE SPECIFY _____

_____ (2) Sample matrix not as described on paperwork, i.e., supposed to be organic but is actually aqueous, nonhomogenous, etc. PLEASE SPECIFY _____

_____ (3) Reprep caused by: _____
(a) Error in spiking or surrogating
(b) Lost extract
(c) Lost sample
(d) Contamination
(e) Low recovery
(f) Other _____

_____ (4) Standard operating procedure not adhered to (SPECIFY) _____

_____ (5) Other (PLEASE SPECIFY) _____

CORRECTIVE ACTION TAKEN: [Check applicable item(s)]:

_____ (1) Error corrected by analyst.
_____ (2) Situation noted on sample tracking sheet and appropriate lab personnel notified.
_____ (3) Sample processed "as is."
_____ (4) Reextraction or resampling requested/performed.
_____ (5) Sample put "on hold" until further notice.
_____ (6) Client informed verbally.
_____ (7) Client informed by memo/letter.
_____ (8) Other (specify) _____

ROUTING

Title	Initials	Date	Check if Corrected
Analyst			
Group Supervisor			
QC Coordinator			
Lab Manager			

FIGURE 13-3

EXAMPLE NONCONFORMANCE MEMO

DATA REVIEW - GC/MS

DATE _____
LIMS CODE _____
FILED BY _____

SAMPLE NO.(s) _____
_____

NONCONFORMANCE: [Check applicable item(s)]:

_____ (1) Data missing.
_____ (2) Data improperly filled out.
_____ (3) Analysis incomplete or not meeting client request.
_____ (4) Daily tune nonconformance.
_____ (5) Initial calibration nonconformance.
_____ (6) Continuing calibration nonconformance.
_____ (7) Method blank nonconformance.
_____ (8) Sample nonconformance.
_____ (9) Additional QC nonconformance.
_____ (10) Other (specify) _____

Explanation of Above (if needed): _____

CORRECTIVE ACTION TAKEN: [Check applicable item(s)]:

_____ (1) Project returned to analyst(s) for rework
_____ (2) Reanalysis requested
_____ (3) Reprep and reanalysis requested
_____ (4) Data accepted "as is"
_____ (5) Client informed verbally
_____ (6) Client informed by memo/letter
_____ (7) Project "on hold" until further notice
_____ (8) Other (specify) _____

ROUTING

Title	Initials	Date	Check if Corrected
Analyst			
Lab Supervisor			
QC Coordinator			
EPA Project Manager (if necessary)			

9 1 1 2 3 6 4 0 2 8 2

IT CORPORATION

CAUTION

NONCONFORMING ITEM

Tag No _____ Affected Item _____

_____

_____

Problem _____

_____

_____

_____

☐ Work may continue on this item subject to the following limitations

_____

_____

_____

☐ Work may not proceed on this item until this nonconformance has been dispositioned.

_____ _____

QC Coordinator Date

FIGURE 13-4

EXAMPLE - NONCONFORMING ITEM TAG

9112764023

EXAMPLE NONCONFORMANCE LOG

Date Issued	Responsible Manager	Description	Corrective Action Completion Date		Verified Date	Reference File Index
			Target	Date		

14.0 QUALITY ASSURANCE/QUALITY CONTROL AUDITS

Audits of an analytical laboratory are described as:

- Performance audits conducted on an ongoing basis within the laboratory by the Quality Control Coordinator. These audits are reported to the Laboratory Manager and the ITAS QA/C Director. They include: monthly surveillances, and semiannual internal audits.

- System audits performed on a scheduled, periodic (semi-annual) basis by the ITAS Quality Assurance/Control Director. These audits are external to the laboratory and are reported to the Vice President, Analytical Services and the Director, Quality Assurance and Discipline Management, Environmental Projects Group.

- Data audits performed on respresentative projects from a laboratory. These audits are conducted on a project-specific basis by the ITAS QA/C Director or his designee.

Audits of the laboratories are performed for the following reasons:

- To determine that contractual and regulatory obligations are fulfilled

- To determine that IT procedures and standards are being followed. These audits may include the ITAS QA Manual, Laboratory-Specific Attachments, project-specific QA Project Plans, Chain-of-Custody forms, Sample Collection Logs, etc.

- To establish that Quality Assurance objectives are met, including holding times, reporting turnaround times, use of approved analytical methods, and stated objectives for precision, accuracy, representativeness, completeness, and comparability (PARCCs)

- To serve as a management tool to evaluate appropriateness of Quality Assurance policies

- To identify potential or actual deficiencies for the purposes of evaluating compliance with requirements and providing the means for correction.

- To determine that records are prepared/maintained as required.

Audits are <u>not</u> conducted to assign blame.

The content and conduct of the audits are discussed below.

14.1 PERFORMANCE AUDITS

As stated in Section 2.1, the Quality Control Coordinator is responsible for the preparation of Quality Control samples, insertion into the sample stream, and analysis of the results. The samples are analyzed on a daily, ongoing basis and provide the means for demonstrating data quality by statistical analyses.

To complete internal laboratory reviews, the Quality Control Coordinator shall provide ongoing monitoring of laboratory operations. The review is conducted on behalf of the Technical Director and the Laboratory Manager to verify that the laboratory Quality Assurance Program is implemented and functioning on a daily basis.

Reviews may be of two kinds: surveillances or semiannual internal audits. Surveillances are detailed inspections of specific areas of a laboratory and its QA program, performed on a monthly basis by the QCC. Surveillances do not require as extensive planning and preparation as do audits, and prior communication with the surveyed group or personnel is not necessary. The QCC shall observe the activity of interest while it is in process and/or review objective evidence. A predesigned checklist based on the applicable documents and criteria will be used for this review. The semiannual internal audit covers the same criteria that the semiannual ITAS Quality Assurance Systems Audit does, namely the laboratory's QA Program. The audit is intended to be a spot check and should include:

- Sample maintenance
  - Are stated temperatures for sample storage provided?
  - Are samples processed and tested within prescribed holding times?
  - Are samples properly logged in?

- Calibration
  - Are calibrations performed as required?
  - Are they properly documented in instrument log books, or as part of project data if required?
  - Do calibration results indicate a trend in instrument performance?
- Preventive maintenance
  - Are adequate spare parts available?
  - Do specific instruments have repeated maintenance problems?
  - Is preventive maintenance performed and properly documented?
- Receipt and storage of standards, chemicals, and gases
  - Are all reagents, chemicals, and gases purchased for use in the laboratory of adequate grade for the intended use?
  - Are certifications of material compositions provided when required?
  - Are materials adequately stored to prevent degradation?
  - Are materials kept beyond stated shelf life?
  - Are internal standards properly prepared and stored?
  - Are internal standards kept beyond stated shelf life?
- Analytical Methods
  - Are the methods used appropriate for project requirements?
  - Are alternate methods approved for use?
- Data verification
  - Are data processed and validated as prescribed?
- Records management
  - Are the records of analyses complete and properly identified?

9 1 1 2 7 4 1 0 2 3 7

- Are documents submitted to the record system in a timely manner and are they properly maintained?

Nonconformances observed by the QCC shall be reported to the Group Leaders and Operations Manager for corrective action, or the Technical Director, Laboratory Manager, or the ITAS Quality Assurance/Control Director, if necessary, for corrective action to be taken. The QCC shall keep a log of nonconformances. The log shall document the nonconformance, date of occurrence, reason for occurrence if known, date of corrective action, and corrective action taken.

14.2 SYSTEM AUDITS

System audits shall be conducted by the ITAS QA/C Director or designee. These audits shall be on a semi-annual basis and provide a thorough overview of implementation of the Quality Assurance Program within the laboratory. If project-specific audits are performed because of project requirements, the audit shall focus only on the performance of the laboratory for the project.

System audits will review operation of the laboratory and resulting documentation, including all items reviewed by the Quality Control Coordinator. Particular emphasis will be placed upon implementation of the Quality Control sample program and nonconformance log. Review of these aspects of the laboratory Quality Assurance Program should indicate trends adverse to data quality.

Audits by the ITAS QA/C Director shall be performed in the following manner:

- An audit plan shall be prepared which considers the activities of the specific laboratory. The Quality Assurance Manual and Manuals of Practice are the sources of generic requirements for the audit. Laboratory-Specific Attachments are the source documents for specific laboratory activities. The audit plan should be reviewed and updated for every semiannual audit, with information gained during previous audits considered. The audit plan shall be the basis for the audit and should define: participating auditors, applicable documents, schedule, scope of laboratory activities. (See Figure 14-1 for an example form for an Audit Plan.)

A lead auditor will be responsible for the audit planning and performance. As necessary, technical specialists will assist the audit team in preparing and conducting of the audit. All persons participating in the audit team will be independent of the laboratory audited.

- Based on the audit plan, detailed checklists of questions to be asked during the audit will be prepared. The checklists will provide adequate means for indicating whether the question is satisfactorily answered, or if it is not applicable, and for comments.

- The audit team will meet at the beginning of the audit with the Laboratory Manager to discuss the laboratory operations to be audited. The QCC should be available to the audit team throughout the audit.

- At the close of the audit, the audit team will meet with the Laboratory Manager to discuss the audit findings. As necessary, the Operations Manager, Group Leaders, Technical Director, and Quality Control Coordinator should attend the audit closure to discuss the findings. The lead auditor can close a finding during this discussion if the laboratory staff can satisfactorily demonstrate that the finding is inappropriate. Also during the audit-closure meeting, the means for corrective action and verifying correction will be discussed. If corrective action can be initiated immediately after the audit closure, the action should be taken.

- An audit report will be prepared by the lead auditor and will discuss the following:

  - Date and location of audit

  - Audit team members and persons contacted in the laboratory

  - Laboratory operations audited

  - Description of items requiring corrective action and, if possible, the means for correction

  - Due date for completion of corrective actions

  - Means for verifying completion of corrective action

  - Review of the Quality Assurance Program.

  The audit report shall be issued as soon as possible after completion of the audit (required within 30 days).

- The Laboratory Manager is responsible for responding to the audit report. Response shall be in writing to the lead auditor and shall

state the corrective action taken or the action underway. If correction can be verified through correspondence, the Laboratory Manager shall attach documentation of corrective action to the audit response.

- Upon receipt of the audit response, the lead auditor must verify completion of the corrective action.
- After verification of corrective action, the lead auditor will issue a closure statement stating that all corrective action has been completed and the audit is closed. All audits must be closed.

During the course of the semiannual system audits, the ITAS Quality Assurance/Control Director and/or lead auditor should be cognizant of recurring nonconformances in a laboratory or trends which will affect quality. Recurring nonconformances and trends should be addressed in the audit report. Correction for such events may require a review of the adequacy of the Quality Assurance Program. If the inherent problem lies within the Program, the Program shall be amended through appropriate revision of Quality Assurance documents.

14.3 DATA QUALITY AUDITS

Data quality audits are designed to address the precision, bias (accuracy), representativeness, and completeness of the data. Representative projects performed by the Laboratory are evaluated periodically by the ITAS QA/C Director or his designee. USEPA guidelines and project-specific checklists are used as criteria during the audits. Results are reported to the laboratory QC Coordinator, Laboratory Manager, and the Project Manager.

INTERNATIONAL TECHNOLOGY CORPORATION

QUALITY ASSURANCE AUDIT PLAN

PROJECT NAME	DEVELOPED BY
PROJECT NO.	DATE

AUDIT TITLE

AUDIT LOCATION	AUDIT SCHEDULE	CHECKLISTS TO BE USED
	FROM _____	GENERIC • • • • • ☐
	TO _____	AUDIT SPECIFIC • • ☐

AUDIT PERSONNEL

LEAD AUDITOR _____

QA STAFF _____

TECHNICAL SPECIALISTS _____

GROUP OR ORGANIZATION TO BE AUDITED

IT GROUP _____

SUBCONTRACTOR _____

OTHER EXTERNAL ORGANIZATION _____

AUDIT TYPE

- PREQUALIFICATION • • • • • • • • ☐
- FIELD OPERATIONS • • • • • • • • ☐
- LABORATORY • • • • • • • • • ☐
- IN-PROCESS PROJECT • • • • • • • ☐
- PROJECT AND REPORT • • • • • • • ☐
- PROJECT FILE • • • • • • • • • ☐
- SOURCE • • • • • • • • • • • ☐
- OTHER • • • • • • • • • • • ☐

APPLICABLE DOCUMENTS

- IT QA MANUALS • • • • • • • • • ☐
- PROJECT QA PLAN • • • • • • • • ☐
- CONTRACT/PROCUREMENT DOCUMENT • ☐
- SPECIFIC/GENERIC PROCEDURES • • • ☐

ACTIVITIES AND MATERIALS TO BE AUDITED

- SUBCONTRACTOR CAPABILITIES • • • • • ☐
- QA/QC PROGRAMS • • • • • • • • • ☐
- FIELD ACTIVITIES & IN-PROCESS RECORDS • • ☐
- IDENTIFICATION & CONTROL OF SAMPLES • • ☐
- LABORATORY ACTIVITIES & IN-PROCESS RECORDS • ☐
- EQUIPMENT CALIBRATION & RECORDS • • • ☐
- PRODUCTION ACTIVITIES & IN-PROCESS RECORDS • ☐
- IN-PROCESS NUMERICAL ANALYSES & DESIGN • ☐
- COMPUTER PROGRAM VERIFICATION & RECORDS • ☐
- COMPLETED RECORDS AND CHECKPRINTS.
  - SUBCONTRACTOR • • • • • • • • • ☐
  - FIELD • • • • • • • • • • • • ☐
  - LABORATORY • • • • • • • • • • ☐
  - NUMERICAL ANALYSES & DESIGN • • • • ☐

TRANSMITTAL OF INFORMATION TO IT • • ☐

TRANSMITTAL OF INFORMATION FROM IT:

- DATA & RECORDS • • • ☐
- COMPUTER ANALYSES • • ☐
- SAR SECTIONS • • • • ☐
- LICENSE/PERMIT APPLICATION • • • • ☐
- RI/FS • • ☐
- REPORT • • ☐
- OTHER • • ☐

_____

_____

RECORD CONTROL & RETENTION • • • • ☐

OTHER • • • • • • • • • • • • ☐

_____

_____

FIGURE 14-1

QA AUDIT PLAN FORM

15.0 QUALITY REPORTS TO MANAGEMENT

Both the Quality Control Coordinator and the ITAS Quality Assurance/ Control Director are responsible for preparing reports to management indicating effectiveness of the Quality Assurance Program.

15.1 PERFORMANCE AUDIT REPORTING

The internal laboratory review activities of the Quality Control Coordinator shall be summarized in a monthly surveillance report to the ITAS QA/C Director. The report can be in the form of a checklist, with brief narratives as required, describing the activities reviewed. A copy of the nonconformance log for the month shall be attached. The monthly report shall emphasize ongoing or recurring problems. To demonstrate review, the Laboratory Manager shall sign and date the report. The signed copy shall be maintained by the Quality Control Coordinator for a period of two years after the date of the audit.

15.2 SYSTEM AUDIT REPORTING

Audit reports prepared from the semiannual system audits shall be distributed to the Vice President, Analytical Services; Laboratory Manager; Regional Director Analytical Operations; and the ITAS QA/C Director (if not the lead auditor); and the Environmental Projects Group Director, Quality Assurance and Discipline Management. The ITAS QA/C Director shall sign and date the reports and shall maintain copies for two years after the date of the audit.

15.3 NONCONFORMANCE/CORRECTIVE ACTION RESOLUTION FROM SYSTEM AUDITS

The Laboratory Manager and ITAS Quality Assurance/ Control Director shall seek to resolve directly all differences concerning cited nonconformances, requests for corrective action, or the completion of corrective action. If the difference cannot be resolved, the Vice President, Analytical Services shall be the arbiter. The decision of the Vice President is final.

15.4 MANAGEMENT REVIEW OF THE QUALITY ASSURANCE PROGRAM

Review of the appropriateness and adequacy of the Quality Assurance Program is ongoing. At any time, the Laboratory Manager should present recommended changes to the ITAS QA/C Director.

During system audits, the Quality Assurance Program should be discussed. The audit report will document recommendations made by either the Laboratory Manager or the audit team for revision.

In addition to these reviews, the Vice President, Analytical Services shall conduct an annual review of the Quality Assurance Program considering:

- Results of system audit reports, such as, are undesirable trends occurring?
- Is the present status of Quality Assurance documents adequate? Should manuals be revised; are new manuals needed?
- Are audits fulfilling their purpose?

The Vice President will consult with the Laboratory Managers and the ITAS QA/C Director, and the Environmental Projects Group Quality Assurance Director during the review as deemed appropriate. To document the review, the Vice President will issue a memorandum to the ITAS Quality Assurance/Control Director, the Environmental Projects Group Director, Quality Assurance and Discipline Management, and Laboratory Managers stating the extent of the review and recommendations.

16.0 TRAINING

All quality related activities performed by ITAS shall be accomplished by personnel qualified on the basis of education, experience, and training.

The following definitions are relevant to the discussion of training in this section:

- Training - In-depth instruction to develop proficiency in the application of requirements, methods, and procedures. Such instruction may be internal or external classroom sessions, courses, or informal on-the-job assignments.
- Indoctrination - To instruct in fundamentals so as to provide understanding of principles involved.
- Qualification (Personnel) - The characteristics or abilities gained through training or experience or both, that enable an individual to perform a required function.
- Certification - The action of determining, verifying and attesting, in writing, to the qualifications of personnel or material.
- Orientation - The act or process of acquainting individuals with the existing situation, environment or condition.

16.1 QUALIFICATIONS

Each laboratory shall have job descriptions for all positions affecting data quality. These job descriptions shall provide the minimum qualifications in terms of education and experience, knowledge, and skills necessary for an analyst to carry out work in the laboratory. During an annual performance review, the laboratory supervisors shall compare each analyst's performance with the qualifications established in his/her job description.

ITAS normally expects necessary knowledge and fundamental chemical laboratory skills to have been demonstrated by formal academic training to include course work in general chemistry, qualitative analysis, quantitative analysis, and instrumental analysis.

16.2 PROFESSIONAL STAFF, TRAINING, AND QUALIFICATIONS

Laboratory staffs are composed primarily of professional personnel who are scientists. Such personnel shall be assigned duties within the capabilities of their education and experience by the Laboratory Manager or the Operations Manager. Qualifications of all professional personnel shall be documented by resumes which include academic credentials, employment history, experience, and professional registrations. Technicians and support personnel performing a technical function are qualified through experience and this will be indicated in their resumes and training files. These personnel shall also be assigned by the Laboratory Manager or the Operations Manager based on their capabilities. Technicians and support personnel shall be supervised in their activities by experienced personnel until in the opinion of the Laboratory Manager or Operations Manager, or the responsible Group Leader they are capable of independently performing their duties. This authorization to perform independently shall be documented in the training files.

16.2.1. Technical Training and Qualifications

An analyst hired to perform sample preparation procedures and/or analytical procedures shall receive direct instruction from a professional staff member. To become qualified on a particular procedure, the analyst must pass a specified qualifying step under an instructor's supervision. The qualifying is described in Laboratory-specific Attachments to this Manual or SOPs. Only upon acceptable completion of the appropriate qualifying step shall the analyst perform the procedure without assistance or supervision.

Some of the basic operations that should be periodically reviewed with laboratory personnel are: sample logging, sample handling, measuring, weighing, glassware preparation, use of instrumentation, data handling and reporting, quality control and safety.

Technicians or analysts who have not performed an analysis within a period of

one year shall be required to re-qualify before being allowed to perform the procedure again. Laboratory-specific criteria shall be set for maintaining qualifications.

The Laboratory Manager shall perform an annual review and, as appropriate, re-qualify each technician for performing specific analytical procedures. The QC Coordinator shall monitor the qualification status of each technician based on spike results and length of time since the analysis has been performed by each technician. A quarterly status report shall be provided to the Laboratory Manager.

Written procedures shall detail the qualification process and criteria to be used.

16.2.2 Quality Assurance Training and Qualifications

General training in the requirements of the ITAS Quality Assurance Program is required of all laboratory personnel. Formal training sessions and examinations will be conducted and documented by QA personnel. The training program shall address regulatory requirements as appropriate, basic quality control practices, responsibilities of the technical staff, responsibilities of each the QCC, the reporting of non-conformances, and the performance of audits. In addition, each laboratory analyst shall become familiar with the laboratory quality assurance program by reading quality control procedures and sections of the laboratory-specific attachment and the ITAS QA Manual appropriate to his/her position.

For initial quality assurance qualification, each new employee shall be given a quality assurance examination that will test the trainee's knowledge and use of the laboratory's quality assurance program. Passing this examination will demonstrate sufficient knowledge of the quality assurance program to perform work in the laboratory upon training in a specific technical area. Acceptance criteria for the QA examination are given in the laboratory-specific training procedures, which also denotes qualification of current employees. For

maintenance of QA qualification and retraining, the laboratory staff shall conduct periodic quality assurance training sessions for the laboratory analysts. Training shall be documented on a standard form (Figure 16-1) and maintained in a training file for each individual.

Training in the nature and goals of the Quality Assurance Program shall be provided at least once a year to all laboratory personnel by the Quality Control Coordinator.

16.3 QUALITY CONTROL COORDINATOR'S TRAINING AND QUALIFICATIONS

All Quality Control Coordinators shall receive training from the ITAS QA/C Director so that the QC Coordinators are proficient in the requirements of the IT Analytical Services Quality Assurance Program and its application. Training will emphasize the performance of audits and required reporting. This will include audit fundamentals, objectives, characteristics, organization, performance and results of quality auditing, and reporting and follow-up actions involved in conducting audits. Quality Control Coordinators must be capable of communicating effectively, in writing and orally, with all levels of the organization. Additional training may be required by on-the-job experience for practice and evaluation, or by classroom sessions such as workshops, speech-making, or management courses which stress public speaking. Related training should include statistical quality control, the writing of Standard Operating Procedures (SOPs), and the review of analytical data and QA Project Plans using standardized guidelines.

An examination of QC Coordinators will be conducted at the end of the training course. The examination may be either oral, written, practical, or a combination thereof to verify the QCC's comprehension of and ability to apply the prescribed body of knowledge and auditing skills. The examination will be documented, thus providing certification of the QCC's qualifications, and signed by the ITAS QA/C Director. The certification must include the QC Coordinator's name, date of certification, and date for recertification; the bases of certification, such as education, experience, skills, training, and

examination results; and the signature of the authorized certifying person.

The examination should cover applicable aspects of the IT Quality Assurance Program, regulatory requirements, and Quality Assurance auditing. The ITAS Quality Assurance/Control Director is responsible for preparing and administering the examination and evaluating the results.

Continued proficiency of Quality Control Coordinators shall be maintained through active participation in Quality Assurance audits and the preparation and the review of Quality Assurance documents. Evaluation of Quality Control Coordinator proficiency shall be conducted on a yearly basis by the Director, ITAS QA/C. If a previously certified Quality Control Coordinator fails to maintain proficiency through active participation as above, formal requalification by the ITAS QA/C Director shall be required. This requalification will consist of:

- Re-examination
- Satisfactory participation in at least one Quality Assurance audit of an IT laboratory or project
- Additional training and/or experience as deemed necessary by the ITAS QA/C Director.

Formal certification and the annual evaluation of QC Coordinators shall be documented and maintained by the Director, ITAS QA/C.

16.4 QUALIFICATION AND TRAINING RECORDS

Each laboratory employee shall have a Personnel Qualifications Record as shown in Figure 16-2. The record shall be documented with technical and quality assurance procedures for which the employee is qualified, the dates of qualification and renewal, and the approval signature. Other qualification and training documents include:

- Personnel resumes
- Record of observation on a sample preperation procedure

- Results of qualifying sample sets
- Quality assurance examinations
- Attendance records of training sessions
- Professional certificates
- Internal training certificates of qualifications.

The QC Coordinator is responsible for maintaining the personnel qualifications and training records as quality documents in the Quality/Operations record system Category N. The ITAS QA/C Director maintains the training file for all ITAS QC Coordinators.

Figure 16 - 1

EXAMPLE - INDIVIDUAL TRAINING RECORD

FOR: _____ HIRE DATE: _____

TITLE: _____ SUPERVISOR: _____

ACADEMIC TRAINING (to include College, Business, Trade or Technical School)

Name of Institution (including current enrollment)	City and State	Curriculum Major	Attendance	Title of Degree(s) Received or Expected	Date Degree Received or Expected

PROFESSIONAL CERTIFICATIONS

Certification	From	Valid from, to

Figure 1f continued

SPECIAL SCHOOLING/TRAINING

Year	Course	Sponsored by/Location

ON THE JOB TRAINING

	Dates BASIC	Dates QUALIFIED	Action & Signature
Quality Assurance Manual			
Standard Operating Procedures			
Laboratory Documentation			
Receiving Inspection			
Specific Job Function			
New Employee Orientation			
Safety Orientation			

Documented On _____

By _____

QC Concurrence _____

Figure 16 - 2

EXAMPLE - PERSONNEL QUALIFICATION RECORD

NAME: _____ HIRE DATE: _____

TITLE: _____ SUPERVISOR: _____

PROCEDURE NAME, NUMBER	QUALIFY DATE	RENEW BY	APPROVAL

TRAINING SESSION	DATE	APPROVAL

9112364030 2

ITAS QA MANUAL

GLOSSARY

This glossary is intended to provide standard definitions for terms in common use in ITAS Laboratories.

Absolute Method

Method in which characterization is based entirely on physical (absolute) defined standards.

Accuracy

The degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X - T, or the difference as a percentage of the reference or true value, 100(X - T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias inherent in the system.

Aliquot

A measured portion of a sample taken for analysis.

Analysis Matrix Spike

A sample created by spiking target analytes into a prepared portion of a sample just prior to analysis.

Arithmetic Mean (Cm for populations, X for samples)

The most commonly used measure of central tendency, commonly called the "average". The sum of all the values of a set divided by the number of values in the set.

Audit

A documented activity performed in accordance with written procedures or checklists to verify, by examination and evaluation of objective evidence, that applicable elements of the quality assurance program have been developed, documented and effectively implemented in accordance with specified requirements. An audit should not be confused with surveillance or inspection.

Bias

A systematic (consistent) error in test results. The difference between the population mean and the true or reference value, or as estimated from sample statistics, the difference between the sample average and the

reference value.

Blind Replicate

A blind replicate sample is a duplicate sample which has been introduced as a separate sample by the Quality Control Coordinator during the log-in process or prior to analysis; it is not known to be a replicate sample when the analyst receives it (blind).

Bulk Sampling

Sampling of a material that does not consist of discrete, identifiable, constant units, but rather of arbitrary, irregular units (i.e., insulation material, asbestos tiles).

Calibration

Establishment of a relationship between various calibration standards and the measurements of them obtained by a measurement system, or portions thereof. The levels of the calibration standard should bracket the range of levels of which actual measurements are to be made; or the act of making a scheduled comparison of instrument performance against national standards for instruments which measure physical parameters, such as mass, time, and temperature. This type of calibration is independent of use in specific analyses and projects.

Calibration Check Compounds (CCC)

Target compounds used to evaluate the calibration stability (precision) of the GC/MS system. Maximum percent deviations of the CCCs are defined in the USEPA's Contract Laboratory Program (CLP) protocol.

Calibration Standard

A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to Standard Reference Materials or primary standard.

Certified Reference Material (CRM)

A reference material accompanied by a certificate issued by an organization generally accepted as technically capable to do so.

Chain-Of-Custody

A system of documentation to demonstrate physical custody and traceability of samples to provide defensible, scientifically sound data.

Certificate of Analysis

An ITAS form for reporting analytical results.

Checkprint

Copy of raw data or data report on which data is reviewed (checked), initialed or signed, and dated.

Check Standard Analyses

A midpoint standard or check standard analyzed with each group of samples to verify the standard curve.

Chemical Calibration

Relation of response of a measurement system to the concentration or amount of analyte of concern.

Coefficient of Variation (Relative Standard Deviation)

A measure of precision (relative dispersion). It is equal to the standard deviation divided by the mean and multiplied by 100 to give a percentage value.

$$CV\ (RSD) = \frac{s}{\bar{X}} \times 100 \text{ for a sample group}$$

Collaborative Tests (or Studies)

The evaluation of a new analytical method under actual working conditions accomplish by the participation of a number of typical or representative laboratories in analyzing portions of carefully prepared homogeneous samples.

Collocated Samples

Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. The results will include sampling as well as anlaytical variability.

Comparability

A measure of the confidence with which one data set can be compared to another.

Comparative Method

Method in which characterization is based on chemical standards (i.e., comparison with such standards).

Completeness

The amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal operations, to fully evaluate and understand the system under optimum conditions; usually expressed as a percentage.

Composite

A sample composed of two or more increments.

Confidence Coefficient

The chance or probability, usually expressed as a percentage, that a confidence interval has of including the population value.

Confidence Interval

A value interval that has a designated probability (the confidence coefficient) of including some defined parameter of the population.

Confidence Limits

The outer boundaries of a confidence interval.

Continuing Calibration

Analytical standard run every 12 hours to verify the calibration of the GC/MS system.

Contract Laboratory Program CCLP

USEPA Superfund contract-required analytical methods and QC criteria.

Corrective Action

A measure taken to correct a deficiency and to minimize the possibility of recurrence.

Correlation Coefficient

A number between -1 and 1 that indicates the degree of linear relationship between two sets of numbers.

Data Quality Objectives

The precision, accuracy, representativeness, completeness and comparability goals to be achieved for a particular data set or project.

Data Validation

A systematic effort to identify any outliers and/or errors, and thereby cause deletion or flagging of suspect values for investigation. To provide valid data to the user and to demonstrate that numerical computation of data is correct and that it is correctly reported.

Data Verification

The steps taken within an analytical laboratory so that reported results correctly represent the analyses performed.

Decafluorotriphenylphosphine (DFTPP)

Compound chosen to establish mass spectral tuning performance for semi-volatile analysis by GC/MS.

Deficiency

An unauthorized deviation from accepted procedures, practices, or standards; or a defect in an item that could lead to degradation of quality.

Degrees of Freedom

The number of independent deviations used in calculating an estimate of the standard deviation.

Duplicate Sample Analyses

Duplicate analyses (different aliquots of the same sample) are performed to evaluate the precision of an analysis.

Error

The difference between an observed or measured value and its true value, or the probability interval that contains the systematic and random error with 1-Cs confidence.

External Reference Material (ERM)

A reference material provided by someone other than the end-user laboratory.

Field Blank

A blank that is prepared, handled, and analyzed in the same manner as normal carrying agents except that it is not exposed to the material to be selectively captured during sample collection.

Field Matrix Spike

A sample created by spiking target analytes into a portion of a sample in the field at the point of sample acquisition.

Geometric Mean

The nth root of the product of all values in a set of n values or the antilogarithm of the arithmetic mean of the logarithms of all the values of a set of n values. The geometric mean is generally used when the logarithms of a set of values are nearly normally (Gaussian) distributed, such as is the case of much pollution data.

Grade A Standard

Atomic weight standard.

Grade B Standard

Ultimate standard - a substance which can be purified to virtually Grade A.

Grade C Standard

Primary standard - commercially purified to a purity of 100 C= 0.02%.

Grade D Standard

Working standard - commercially available, purity of 100 C= 0.05%.

Grade E Standard

Secondary standard - of lower purity, standardized against Grade C material.

Gross Sample (also called Bulk Sample, Lot Sample)

One or more increments of material taken from a larger quantity (lot) of material for assay or record purposes.

Homogeneity

The degree to which a property or substance is randomly distributed throughout a material. Homogeneity depends on the size of the units

9 1 1 2 2 4 0 7 0 3

under consideration. Thus a mixture of two minerals may be inhomogeneous at the molecular or atomic level, but homogeneous at the particulate level.

Hazardous Substance List

A USEPA Superfund list of chemicals whose presence is to be tested in each sample received for CLP analysis. It includes the Priority Pollutants plus other commonly-occurring chemicals found at hazardous waste sites.

IDL

Instrument Detection Limit - the smallest signal an instrument can reliably detect.

Increment

An individual portion of material collected by a single operation of a sampling device from parts of a lot separated in time or space. Increments may be either tested individually or combined (composited) and tested as a unit.

Individuals

Conceivable constituent parts of the population.

Initial Calibration

Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of an instrument to the target compounds.

Internal Reference Material (IRM)

A reference material developed by a laboratory for its own internal use.

Internal Standards

Compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for VOAs), and sample extract (for semi-volatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds by GC/MS.

Laboratory Matrix Spike

A sample created by spiking target analytes into a portion of a sample when it is received in the laboratory.

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Laboratory Sample

A sample, intended for testing or analysis, prepared from a gross sample or otherwise obtained. The laboratory sample must retain the composition of the gross sample. Often reduction in particle size is necessary in the course of reducing the quantity.

Laboratory-Specific Attachment

Documents supplementary or alternative implementing practices and services of a specific laboratory; for the specific laboratory, becomes incorporated with ITAS Quality Assurance Manual. Laboratory-Specific Attachments to the Quality Assurance Manual are prepared for three purposes:

- If an individual ITAS laboratory provides services that are unique to that laboratory, the service and necessary Quality Assurance requirements will be described in an attachment.

- Certain aspects of laboratory operations discussed in the Quality Assurance Manual are dependent upon laboratory facilities. Quality Assurance activities much as preventive maintenance, calibration, and data verification are functions of the equipment in a specific laboratory. Attachments will be prepared by each laboratory to document their specific operations. Laboratory-Specific Attachments are adopted as part of the Quality Assurance Manual for the preparing laboratory.

- A specific laboratory may perform certain actions in accordance with an alternate, approved technique to that presented in the ITAS QA Manual.

LOD

Limit of Detection - smallest concentration/amount of analyte that can be reliably reported as found/detected in a material/sample.

Lot

A quantity of bulk material of similar composition whose properties are under study.

Lot Size (N)

The number of units in a particular lot.

Lot, Batch

A definite quantity of samples collected under conditions that are considered uniform.

Macro Method

Method requiring more than milligram amounts of sample.

Manuals of Practice

Detailed discussions of specific technical subjects. Provides detailed information related to technical topics discussed in the Quality Assurance Manual. For example, a Manual of Practice for the field collection, preservation, and shipment of samples to ITAS laboratories provides specific uniform direction to IT personnel.

Matrix

The predominant material in which the analyte(s) of primary interest is found in the sample to be analyzed. A sample matrix is usually either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

Matrix Effect

Materials in a sample cause interferences in measurement of analytes and QC parameters which prevent the acquisition of acceptable results.

Matrix Spike

Aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix. The percent recovery for the respective compound(s) will then be calculated.

Matrix Spike Duplicate

A second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

MDL

Method Detection Limit - smallest concentration/amount of analyte a method can reliably detect, wherever located; when the sample containing the analyte is processed through the entire preparation and analysis procedure. The minimum concentration of an analyte that can be reported with 99% confidence to have a value that is above zero. The MDL is operationally defined as:

$$MDL = st_{(n-1,\ 1-\alpha\ =\ 0.99)}$$

Where s = the standard deviation of a minimum of 7 measurements of a solution containing the analyte at a concentration near the lowest standard recommended in the method and

$t_{(n-1,1-\alpha = 0.99)}$ = the student's t value for a one-tailed test at the 99% confidence level and n-1 degrees of freedom.

Mean

The arithmetic mean is a measure of control tendency; it is equal to the sum of the observed values divided by the number of observations. Also call "average".

Measurement

The process or operation of ascertaining the extent, degree, quantity, dimensions, or capability with respect to a standard.

Median

The middle value of a set of data when the set of data are ranked in increasing or decreasing order. If there are an even number of values in the set, the median is the arithmetic average of the two middle values.

Method

An assemblage of techniques.

Method Blank

An analytical control consisting of all reagents, which may include internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background contamination. A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples.

Micro Method

Method requiring milligram or smaller amounts of sample.

Mode

The value or values occurring most frequently in a set or sample of data.

Modified Method

A standard or reference method which has been changed to meet project or matrix requirements.

Nonconformance

Any event which is beyond the limits established for laboratory operation. A deficiency in characteristic, documentation, or procedure sufficient to render quality of an item unacceptable or indeterminate.

Observed value, Observation, or Variate (X)

The particular designated value of a characteristic; for example, X444C1555, X444C2555, X444C3555, and so on.

Operational Calibration

Routinely performed as part of instrument usage, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operational calibration is generally performed for instrument systems.

Outlier

An extreme value that statistically does not belong to the group of values with which it is associated.

Parameter

A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). Also, a chemical being measured, i.e., an analyte.

Percent Difference

If two independent measurements of the same characteristics are available, it is convenient to use the percent difference instead of the coefficient of variation.

$$\text{Percent Difference} = \frac{|X - X|}{(X + X)/2} \times 100 = \frac{\text{Range}}{\bar{X}} \times 100$$

Percent Moisture

An approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at 105°C, including water.

Percent Recovery (%R)

The results of spiked samples are used to calculate this quality control parameter for accuracy evaluation.

$$\%R = \frac{\text{observed concentration spiked sample - sample concentration}}{\text{true spike concentration}} \times 100\%$$

Performance Audit

Planned independent sample checks of actual output made on a random basis to arrive at a quantitative measure of the quality of the output. Conducted on an ongoing basis within the laboratory by the Quality Control Coordinator. These audits are reported to the Laboratory Manager.

Periodic Calibration

Performed at prescribed intervals for equipment, such as balances and ovens. In general, equipment which can be calibrated periodically is a distinct, singular purpose unit and is relatively stable in performance.

Population

A generic term denoting any finite or infinite collection of individual things, objects, or events in the broadest concept an aggregate determined by some property that distinguishes things that do and do not belong.

Pre-Award Survey

On-site inspection, review, and discussions with prospective contractors at their facilities. Discussions would normally include, but not be limited to, the proposed project plan, personnel, procedures, schedule, and facilities.

Precision

A measure of mutual agreement (or variability) among individual measurements of the same property, usually under prescribed similar conditions. Precision is most desirably expressed in terms of the standard deviation but can be expressed in terms of the variance, range, or other statistic.

Preventive Maintenance

An organized program, within ITAS laboratories, of actions (such as equipment cleaning, lubricating, reconditioning, adjustment and/or testing) taken to maintain proper instrument and equipment performance

and to prevent instruments and equipment from failing during use.

Primary Standard

A material having a known property that is stable, that can be accurately measured or derived from established physical or chemical constants, that is readily reproducible and which can be accepted (within stated limits) and used without question to establish the same value of another substance or item.

Procedure

Detailed instructions to permit replication of a method.

Proficiency Testing

Special series of planned tests to determine the ability of field technicians or laboratory analysts who normally perform routine analyses. The results may be used for comparison against established criteria, or for relative comparisons among the data from a group of technicians or analysts.

Project-Specific Manuals

A manual that describes analytical and/or QA procedures required by a regulatory agency or by contract. It may supplement or change Quality Assurance and/or Quality Control practices for specific project.

Protocol

Methodology specified in regulatory, authoritative, or contractual situations.

Purge and Trap (Device)

Analytical techniques (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

Qualification (Personnel)

The characteristics of abilities gained through education, training or experience, as measured against established requirements, such as standards or tests, that qualify an individual to perform a required function.

Quality

The totality of feature and characteristics of a product or service that bears on its ability to satisfy a given purpose. Absence of defects.

Quality Assurance

The overall controls imposed upon laboratory operations. The name given to procedures by which one ascertains that individual measurements are good enough for their intended purpose. Included are all actions taken by personnel, and the documentation of laboratory performance as specified in the Quality Assurance Program. A system for integrating the quality planning, quality assessment, and quality improvement efforts of various groups in an organization to enable operations to meet user requirements at an economic level.

Quality Assurance Manual

An orderly assembly of management policies, objectives, principles, and general procedures by which ITAS outlines how it intends to produce quality data. The Quality Assurance Manual defines the ITAS Quality Assurance Program. The Manual discusses all aspects for Quality Assurance and Quality Control, both administrative and technical. However, it is not intended that the Manual provide in-depth technical discussion. The Quality Assurance Manual has precedence in policy matters over all other ITAS quality-related documents. The Manual is applicable to and provides direction for all ITAS analytical laboratories.

Quality Assurance Project Plan

An orderly assembly of detailed and specific procedures by which an agency or laboratory delineates how it produces quality data for a specific project or measurement method. A USEPA-specified 16-element format is used to present the information for client and/or regulatory agency approval, prior to beginning field/laboratory work.

Quality Control

The daily, specific actions taken within the laboratory to verify sample integrity, performance of analyses, data processing, and record maintenance. A system of inspections, testing, and remedial actions applied to a process or operation so that, by inspecting a small portion (a sample) of the product currently produced, an estimate can be made of its quality and whether or not, or what if any, changes need to be made to achieve or maintain a predetermined or required level of quality.

QC Check Sample

A reference matrix containing known concentrations of parameters of

interest. If prepared in the laboratory, it is made using stock standard solutions independent of those used for calibration. If the results of these parameters do not meet acceptance criteria concentrations are taken.

Random Error

Variations of repeated measurements that are random in nature and individually not predictable.

Random Samples

Samples obtained in such a manner that all items or members of the lot, or population, have an equal chance of being selected in the sample.

Range (R)

The difference between the largest and smallest numbers in a set of numbers.

Range, Relative (RR)

The range divided by the mean of a particular set of numbers.

Raw Data

All documentation associated with the original recording of anlaytical results pertinent to a specific sample or set of samples. This may include laboratory worksheets, calculation forms, instrument-generated output, analyst notes, etc., from sample receipt through final reporting.

Reagent Blank

Sample composed of materials (water, etc.) which will be added to client samples and analyzed. If contaminants are found in the reagents at levels affecting these sample results, corrective actions must be taken.

Reagent Water

Water in which an interferent is not observed at or above the minimum quantitation limit of the parameters of interest. D1193 ASTM Type II reagent water specifications are:

Grade of Water	Maximum Total Matter (mg/l)	Maximum Electrical Conductivity at 25°C (mho/cm)	Minimum Electrical Resistivity at 25°C (M ohms/cm)	Minimum Color Retention Time of KMnO (min)
Type II	0.1	1.0	1.0	60

Reconstructed Ion Chromatogram (RIC)

A mass spectral graphical representation of the separation achieved by a gas chromatograph; a plot of total ion current versus retention time.

Recovery

A determination of the accuracy of the analytical procedure made by comparing measured values for a fortified (spiked) sample against the known spike values. Recovery is determined by the following equation:

$$\%Rec = \frac{\text{measured spiked value - unspiked value}}{\text{known spiked value}} \times 100\%$$

Reference Material (RM)

Substance in which one or more properties are established for use to calibrate or verify a measurement.

Reference Method

A method of known and demonstrated accuracy.

Regression Coefficients

The quantities describing the slope and intercept of a regression line. Intercept (β_o for populations, b_o for samples), Slope (β_1 for populations, b_1 for samples).

Regression Line or Equation

The function that indicates the regression relationship. For example; $X = + T$ for populations, and $X = b + b T$ for samples.

Relative Error

An error expressed as a percentage of the true value or accepted reference value.

Relative Percent Different (RPD)

Statistic for evaluating the precision of a replicate set. For replicate results D_1 and D_2:

$$RPD\% = \frac{D_1 - D_2}{\frac{D_1 + D_2}{2}} \times 100\%$$

Relative Response Factor (RRF)

A measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where A = area of the characteristic ion measured
C = concentration
is = internal standard
x = analyte of interest

Relative Standard Deviation

See Coefficient of variation.

Repeatability

The precision, usually expressed as a standard deviation, measuring the variability among results of measurements at different times of the same sample at the same laboratory. The unit of time should be specified, since within-day repeatability would be expected to be smaller than between-day repeatability.

Replicability

The precision, usually expressed as a standard deviation, measuring the variability among replicates. It could also be expressed as an upper confidence limit for the difference between two replicates as discussed above.

Replicated Sample

A sample that has been divided into two or more portions, at some step in the measurement process. Each portion is then carried through the remaining steps in the measurement process.

Replicates

Repeated but independent determinations of the same sample, by the same analyst, at essentially the same time and same conditions.

Representative Sample

A sample taken to represent a lot or population as accurately and precisely as possible.

Representativeness

The degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition.

Reproducibility

The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

Resolution (aka Separation)

The separation between peaks on a chromatogram; calculated by dividing the height of the valley between the peaks by the peak height of the smaller peak being resolved, multiplied by 100.

Routine Method

Procedure used in measurement of an analyte. It must be qualified by other adjectives since no degree of reliability is implied.

Sample (Statistical)

A subset or group of objects or things selected from a larger set, called the "lot", or "population". It may consist of an individual or groups of individuals. A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

Sample Average

Same as arithmetic mean.

Sample Size (n)

The number of units in a sample.

Secondary Standard

A material having a property that is calibrated against a primary standard.

Segment

A specifically demarked portion of a lot, either actual or hypothetical.

Semi-volatile Compounds

Compounds amenable to analysis by extraction of the sample with an organic solvent. Used synonymously with Base/Neutral/Acid (BNA) compounds.

Spiked Sample

A normal sample of material (gas, solid, or liquid) to which is added a known amount of some substance of interest. The extent of the spiking is unknown to those analyzing the sample.

Split Sample

A sample divided into two portions, one of which is sent to a different organization or laboratory and subjected to the same environmental conditions and steps in the measurement process as the one retained in-house.

Standard Analysis

An analytical determination made with known quantities of target compounds; used to determine response factors.

Standard Deviation

The square root of the variance of a set of values:

$$\sigma = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n}}$$

A measure of the dispersion about the mean of the elements in a population.

Standard deviation(s)

A measure of the dispersion about the average of the elements in a sample. An estimate of the standard deviation of a population.

Standard Method

A method of known and demonstrated precision issued by an organization generally recognized as competent to do so.

Standard Operating Procedures

Written, detailed documents describing the performance of routine laboratory tasks. They specify what is done, whose responsibility it is to perform tasks and whose to verify their correctness; they are sufficiently detailed to provide data of known quality and integrity, with a minimum loss of data due to out-of-control situations. They also provide for documentation to record the performance of all tasks and their results, and demonstrate the validation of the data each time the data are recorded, calculated, or transcribed. SOPs address the following major elements upon which analytical quality depends:

Organization and personnel
Facilities and equipment
Analytical methodology
Sample custody procedures
Quality control
Data handling.

Standard Reference Material (SRM)

A material produced in quantity, of which certain properties have been certified by the National Bureau of Standards (NBS) or other agencies to the extent possible to satisfy its intended use.

Standardization

The establishment of the value of a potential standard with respect to an established or known standard.

Statistic

A constant or coefficient that describes some characteristic of a sample. Statistics are used to estimate parameters of populations.

Statistical Control Chart

A graphical chart with statistical control limits and plotted values (usually in chronological order) of some measured parameter for a series of samples.

Strata

Segments of a lot that may vary with respect to the property under study.

Stratified Sample

A sample consisting of various portions that have been obtained from identified subparts or subcategories (strata) of the total lot, or population. Within each category or strata, the samples are taken

randomly to obtain a more representative sample than that which might otherwise be obtained by a completely random sampling.

Subsample

A portion taken from a sample. A laboratory sample may be a subsample of a gross sample; similarly, a test portion may be a subsample of a laboratory sample.

Surrogate Standard Analyses

A sample is fortified with surrogate spiking compounds before purging or extraction to monitor preparation and analysis of samples.

Surrogates (Surrogate Standard)

Compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labelled compounds not expected to be detected in environmental media.

Surveillance

A review (monitoring) or observation of ongoing work for the purpose of verifying that an action has been accomplished as specified.

System Audit

A systematic on-site qualitative review of facilities, equipment, training, procedures, recordkeeping, validation, and reporting aspects of total (quality assurance system to arrive at a measure of the capability and ability of the system. Performed on a scheduled, periodic basis by the Quality Control Coordinator or the ITAS Quality Assurances/Control Director.

System Performance Check Compounds (SPCC)

Target compounds designated to monitor chromatographic performance, sensitivity and compound instability or degradation on active sites. Minimum response factor criteria for the SPCCs are defined in the USEPA's CLP GC/MS protocols.

Systematic Error

The condition of a consistent deviation of the results of a measurement process from the reference or known level.

Target Analyte Spiking

Spiking with the analyte that is of basic interest in the environmental sample.

Technique

Physical or chemical principle for characterizing materials of chemical systems.

Tentatively Identified Compounds (TIC)

Compounds detected in GC/MS analysis of samples that are not target compounds, internal standards or surrogate standards. Up to 30 peaks (those greater than 10% of peak areas or heights of nearest internal standards) are subjected to mass spectral library searches for tentative identification.

Traceability of Data

The entire chain of acquired data from the original acquisition effort through to the final tabulation, synthesis, reduction and storage activity will be clearly documented in a fashion which will allow complete reconstruction.

Traceability of Instrumentation

All equipment used in the derivation of data during the conduct of project activities either in the field or during laboratory efforts shall be designated by a unique identification number. This number will in turn be indexed into a system which specifically designates all devices and instrumentation employed by a particular measurement unit and will in turn specify usage and performance periods associated with the identified instrument or device.

Traceability of Samples

During all environmental monitoring field efforts, acquired samples will be assigned specific and unique identification numbers, and such numbers shall be accompanied by documentation which clearly identifies all parameters associated with sample acquisition.

Traceability of Standards

All measurement activities which utilize standards for calibration purposes must have reliable and documented evidence as to the verifiability of standards used.

In this context, all calibration standards must be validated against primary standards such as those available from the National Bureau of

Standards (NBS) or from other primary sources.

Trip Blank Analyses

Trip blanks will be prepared by filling two VOA vials with organic-free water and shipping the blanks with each field kit (group of samples). Trip blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. If the trip blanks indicate possible contamination of the samples, depending upon the nature and extent of the contamination, the samples may either be corrected for the trip blank concentration or the sources re-sampled.

Verification

An act of confirming and substantiating that an activity or condition has been implemented in conformance with the specified requirements.