



Indiana State Department of Health

Indiana Drinking Water Laboratory Certification Program (Chemistry)

(Revised April 8, 2009)

1. Introduction

1.1 The Indiana State Department of Health (ISDH) is responsible for surveying and approving laboratories that make chemical examinations of public water. Certificates of approval are issued to the laboratories that comply with the standards of the State Department of Health for such laboratories.

2. Justification

2.1 The Water Pollution Control Board established drinking water standards for public water supplies under 327 Indiana Administrative Code (IAC) 8.

2.2 Rule 327 IAC 8-2-3 establishes the analytical requirements as, "Except as otherwise provided by this rule, the analytical procedures used as methods of analysis to determine the quality of water sampled shall be in accordance with this rule." Laboratories analyzing drinking water for compliance purposes must use methods approved by US Environmental Protection Agency (USEPA).

2.3 Under an agreement with USEPA and the Indiana Department of Environmental Management, the ISDH surveys and approves laboratories that perform the chemical examination of public drinking water.

3. Standards for Approval of Laboratories

3.1 Application Requirements. The petitioning laboratory must:

3.1.1 Submit a formal request for certification by ISDH.

3.1.2 File an administrative questionnaire provided by ISDH.

3.1.3 Analyze successfully two consecutive Water Supply (WS) proficiency testing (PT) samples within the last year and provide the results of the analyses to ISDH, and

3.1.4 Permit an on-site visit by ISDH certification personnel. This visit is to be arranged at the mutual convenience of both parties.

4. Standards for the Laboratory

4.1. Personnel

4.1.1 Laboratory Supervisor

The laboratory supervisor should have at least a bachelor's degree with a major in chemistry or equivalent, and at least one year of experience in the analysis of drinking water. The laboratory supervisor should have at least a working knowledge of quality assurance principles. The laboratory supervisor has the responsibility to ensure that all laboratory

personnel have demonstrated their ability to satisfactorily perform the analyses to which they are assigned and that all data reported by the laboratory meet the required quality assurance and regulatory criteria.

4.1.2 Quality Assurance Manager

The Quality Assurance (QA) manager should be independent from the laboratory management if possible and have direct access to the highest level of management. The QA manager should have a bachelor's degree in science, training in quality assurance principles commensurate with the size and sophistication of the laboratory and at least one year of experience in quality assurance. The QA manager should have at least a working knowledge of the statistics involved in quality control of laboratory analysis and a basic understanding of the methods, which the laboratory employs.

4.1.3 Laboratory Analyst

The laboratory analyst should have at least a bachelor's degree with a major in chemistry or equivalent, and at least one year of experience in the analysis of drinking water. If the analyst is responsible for the operation of analytical instrumentation, he or she should have completed specialized training offered by the manufacturer or another qualified training facility or served a period of apprenticeship under an experienced analyst. The duration of this apprenticeship should be proportional to the sophistication of the instrument. Data produced by analysts and instrument operators while in the process of obtaining the required training or experience are acceptable only when reviewed and validated by a fully qualified analyst or the laboratory supervisor.

Before beginning the analysis of compliance samples, the analyst must demonstrate acceptable results for blanks, precision, accuracy, sensitivity, specificity and satisfactory analysis on unknown samples. This shall be documented according to the laboratory's QA Plan.

4.1.4 Technician

The laboratory technician should have at least a high school diploma or equivalent, complete a method training program under an experienced analyst and have six months bench experience in the analysis of drinking water samples.

Before beginning the analysis of compliance samples, the technician must demonstrate acceptable results for blanks, precision, accuracy, sensitivity, specificity and satisfactory analysis on unknown samples. This shall be documented according to the laboratory's QA Plan.

4.1.5 Sampling Personnel

Personnel who collect samples should be trained in the proper collection technique for all types of samples, which they collect. Experienced sampling or laboratory personnel should review their technique.

4.1.6 Waiver of Academic Training Requirement

The certification officer may waive the need for specified academic training, on a case-by-case basis, for highly experienced analysts.

4.1.7 Training Records

Training records should be maintained for all personnel. These should include all job-related formal education and training taken by the analyst which pertains to any aspect of his/her responsibilities, including but not limited to analytical methodology, laboratory safety, sampling, quality assurance, data analysis, etc.

4.2. Laboratory Facilities

The analysis of compliance samples must be conducted in a laboratory where the security and integrity of the samples and the data can be maintained. The laboratory facilities should be clean, have adequate temperature and humidity control, have adequate lighting at the bench top and must meet applicable OSHA standards. The laboratory must have provisions for the proper storage and disposal of chemical wastes; secondary containment for hazardous waste storage is recommended. The appropriate type of exhaust hood is required where applicable.

There should be sufficient bench space for processing samples. Workbench space should be convenient to sink, water, gas, vacuum and electrical sources free from surges. Instruments should be properly grounded. For safety reasons, inorganic and organic facilities should be in separate rooms; organic analysis and sample extraction should also be separated to prevent cross contamination. The analytical and sample storage areas should be isolated from all potential sources of contamination. There should be sufficient storage space for the safe storage of chemicals, glassware and portable equipment, sufficient floor and bench space for stationary equipment and areas for cleaning materials.

4.3. Laboratory Equipment and Instrumentation

The laboratory must have the instruments and equipment needed to perform the approved methods for which certification has been requested. All instruments must be properly maintained and calibrated.

4.4. General Laboratory Practices

4.4.1 General

4.4.1.1 Chemicals/reagents: Chemicals and reagents used must meet the specifications in the method. If not specified, then Analytical reagent (AR) grade or American Chemical Society (ACS) grade chemicals or better should be used for analyses in certified laboratories. Consult the currently promulgated editions of Standard Methods for the Examination of Water and Wastewater, part 1070 for more detailed information on reagent grades.

4.4.2 Inorganic Contaminants

4.4.2.1 Reagent water: The laboratory must have a source of reagent water having a resistance value of at least 0.5 megohms (conductivity less than 2.0 micromhos/cm) at 25°C. High quality water meeting such specifications may be purchased from commercial suppliers. Quality of reagent water is best maintained by sealing it from the atmosphere. Quality checks to meet specifications above must be made and documented at planned intervals based on use. This planned interval should not exceed

one month. Individual analytical methods may specify additional requirements for the reagent water to be used. Inorganic methods require distilled or deionized water free of the analyte(s) of interest and trace metals methods require ASTM Type 1 water.

4.4.2.2 Glassware preparation: Glassware cleaning requirements specified in the methods must be followed. If no specifications are listed, then glassware should be washed in a warm detergent solution and thoroughly rinsed first with tap water and then with reagent water. This cleaning procedure is sufficient for general analytical needs. It is advantageous to maintain separate sets of suitably prepared glassware for the nitrate and mercury analyses due to the potential for contamination from the laboratory environment.

4.4.3 Organic Contaminants

4.4.3.1 Reagent water: Reagent water for organic analysis must not contain analytes of interest above their respective method detection levels (MDLs). It may be necessary to treat water with activated carbon to eliminate all interferences. Reagent water requirements of individual methods must be followed.

4.4.3.2 Glassware preparation: Glassware cleaning requirements specified in the methods must be followed.

4.4.4 Laboratory safety

While safety criteria are not an aspect of laboratory certification, laboratory personnel should apply general and customary safety practices as a part of good laboratory practices. Each laboratory is encouraged to have a safety plan as part of their standard operating procedure, which includes personnel safety, training and protection. If safety practices are included in an approved method (i.e., 515.1), then they must be followed. See Standard Methods for the Examination of Water and Wastewater, part 1090 for a discussion of laboratory safety.

4.4.5 Quality Assurance

Laboratories should maintain current Quality Assurance Plans. All laboratory activities including, but not limited to, sampling, test methods, instrument operation, data generation, data validation and corrective action procedures should be described in the Plan. All personnel must read plans.

4.5. Analytical Methods

4.5.1 General

A list of promulgated methods for inorganic and organic contaminants can be found in Tables 1 and 2, respectively. Methods manuals must be available to applicable personnel. Allowed modification to the methods must be documented. Other methods cannot be used for compliance samples unless approval has been granted by the Agency by obtaining an Alternate Test Procedure (ATP) approval. Contact the appropriate certifying authority for the ATP process. Tables 1 and 2 list the methods which must be used for the analysis of disinfectant residuals. Recommended methods for Secondary contaminants are listed in Table 5.

4.5.2 Analyses approved by the State

Measurements for turbidity, pH, temperature, disinfectant residual, calcium, orthophosphate, silica, alkalinity, and conductivity need not be made in certified laboratories, but may be performed by any persons acceptable to the State. However, approved methodology must be used (Tables 2, 3 and 5). The State should institute a quality assurance program to assure validity of data from these measurements.

4.5.2.1 Turbidity standards: Sealed liquid secondary turbidity standards purchased from the instrument manufacturer or other sources should be calibrated against properly prepared and diluted formazin or styrene divinylbenzene polymer primary standards and revised values assigned at least every four months in order to monitor for any deterioration. This calibration should be documented. These standards should be replaced when they do not fall within 15% of the initial assigned concentration of the standard. Solid turbidity standards composed of plastic, glass, or other materials are not reliable and should not be used.

4.5.2.2 Residual chlorine standards: If visual comparison devices such as color wheels or sealed ampules are used for determining free chlorine residual, the standards incorporated into such devices should be calibrated at least every six months. These calibrations must be documented. Directions for preparing temporary and permanent type visual standards can be found in Method 4500-Cl-G, of the currently promulgated editions of Standard Methods for the Examination of Water and Wastewater. By comparing standards and plotting such a comparison on graph paper, a correction factor can be derived and applied to future results obtained on the now calibrated apparatus.

4.6. Sample Collection, Handling, and Preservation

The manner in which samples are collected and handled is critical to obtaining valid data. It is important that a written sampling protocol with specific sampling instructions be available to and used by sample collectors and available for inspection by the certification officer. (Appendix A, Chain-of-Custody).

4.6.1 Rejection of Samples

The laboratory's rejection criteria must be documented in writing in the laboratory's QA Plan or in an SOP. The laboratory should reject any sample taken for compliance purposes which does not meet the criteria in 4.6.2 through 4.6.6. The laboratory must notify the authority

requesting the analyses and ask for a resample. If resampling is not possible and the sample is analyzed, the sample data must be clearly identified in the data package as being unusable for its intended purpose. In addition, the inadmissibility of these sample data must be clearly communicated to all end data users.

4.6.2 Sample Containers and Preservation

The type of sample container and the required preservative for each inorganic and organic chemical contaminant are listed in Tables 3 and 4. The laboratory must measure and record the temperature of the sample when it arrives. The use of "blue ice" is discouraged because it generally does not maintain the temperature of the sample at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ or less. If blue ice is used, it should be frozen at the time of sampling, the sample should be chilled before packing, and special notice must be taken at sample receipt to be certain the required temperature (4°C) has been maintained.

4.6.3 Maximum Holding Times

Samples must be analyzed within the maximum holding times required by the method. These are listed in Tables 3 and 4.

4.6.4 Sample Collection and Transport

There must be strict adherence to correct sampling procedures, sample handling, complete identification of the sample, and prompt transfer of the sample to the laboratory. When the laboratory is not responsible for sample collection and transport, it must verify that the paperwork, preservatives, containers and holding times are correct or reject the sample. The rejection criteria must be documented in writing.

4.6.5 Sample Collector

The sample collector should be trained in sampling procedures and have complete written sampling instructions (SOPs) for each type of sample to be collected. The sampler must be able to demonstrate proper sampling technique.

4.6.6 Sample Report Form

The sample collection report form must contain, at a minimum, the sample identification number, location of collection, date and time of collection, collector's name, preservative(s) added and shipping requirements, container and volume, sample type, analysis required, and any special remarks concerning the sample. Indelible ink must be used.

4.6.7 Sample Compositing

If samples are composited, the compositing must be done in the laboratory. Samples may only be composited if the laboratory detection limit is adequate for the number of samples being composited (up to a maximum of five). For example, for inorganic samples, composite samples from a maximum of five samples are allowed if the detection limit of the method used for analysis is less than one-fifth the MCL. If the concentration of any inorganic chemical in the composite is greater than or equal to one-fifth of the MCL, then a followup sample must be taken within 14 days at each sampling point included in the composite. These samples must be analyzed for the contaminants, which exceeded one-fifth the MCL in the composite sample. [CFR 144.23(a)(4)] Compositing of VOCs is not recommended.

4.7. Quality Control

4.7.1 General Requirements

4.7.1.1 Availability of QA Documents: The laboratory's QA plan and appropriate Standard Operating Procedures (SOPs) must be readily available to the analysts and for inspection by auditors.

4.7.1.2 Availability of QC Information: All quality control information must be readily available for inspection by auditors.

4.7.1.3 Balances and Weights: Balance range must be appropriate for the application for which it is to be used. Drinking water chemistry laboratories should use balances that weigh to at least 0.0001 g. The balances should be calibrated at least annually with ASTM Type I, Class 1 or 2 weights. (ASTM, 1916 Race St., Philadelphia, PA 19103) This may be done by laboratory personnel or under contract by a manufacturer's representative. We strongly recommend that laboratories have a contract to calibrate balances due to the expense of the calibration weights, and to serve as an outside QC check of the weights and balances. Weights meeting ASTM Type I, Class 1 or 2 specifications should be recertified at least every five years or if there is reason to believe damage (corrosion, nicks) has occurred.

Each day the mechanical or digital balance is used, a verification should be performed. The verification consists of a check of a reference mass at approximately the same nominal mass to be determined. Verifications should be done each weighing session unless it can be shown that fluctuations in the environment do not affect the calibration. Weights meeting ASTM Type 1 specifications may be used. These should be calibrated annually against the reference weights at time of balance calibration. The checks and their frequency should be as prescribed in the laboratory's QA Plan. A record of all checks must be kept and be available for inspection.

4.7.1.4 Color Standards: Wavelength settings on spectrophotometers should be verified at least annually with color standards. The specific checks and their frequency must be as prescribed in the laboratory's QA documents. A record of these checks must be kept as prescribed in the laboratory's QA documents and be available for inspection.

4.7.1.5 Temperature Measuring Devices Liquid bearing thermometers such as mercury or alcohol thermometers must be traceable to NIST calibration and verified at least annually and whenever the thermometer has been exposed to temperature extremes. The correction factor should be indicated on the thermometer and the date the thermometer was calibrated and the calibration factor must be kept as prescribed in the laboratory's QA documents and be available for inspection.

Digital thermometers, thermocouples and other similar electronic temperature measuring devices should be calibrated at least quarterly. The date the thermometer was calibrated and the calibration factor must be kept as prescribed in the laboratory's QA documents and be available for inspection.

When an infrared detection device is used to measure the temperature of samples, the device must be verified at least every six months using a NIST certified thermometer over the full temperature range that the IR thermometer will be used. This would include ambient (20°-30°C), iced (4°C) and frozen (0° to -5°C). Each day of use a single check of the IR should be made by checking the temperature of a bottle of water at the temperature of interest that contains a calibrated thermometer. Agreement between the two must be within 0.5°C, or the device must be recalibrated.

4.7.1.6 Traceability of Calibration: Calibrations of all measurement devices must be traceable to national standards whenever applicable.

4.7.2 Specific Requirements: The following are required for each analyte for which a laboratory is certified:

4.7.2.1 Proficiency Testing (PT) Samples: In order to receive and maintain full certification for an analyte, the laboratory must analyze PT samples (if available) acceptable to the Certifying Authority at least once per year for each analyte and by each method used to analyze compliance samples. Results from analysis of the PT sample must be within the acceptable limits established by U.S. EPA. These acceptance limits are listed in the CFR, Primary and Secondary Drinking Water Regulations [§141.23(k)(3)(ii) and 141.24(f)(17) and (19)].” The laboratory should document the corrective actions taken when a PT sample is analyzed unsuccessfully. A copy of this documentation should be forwarded to the certification officer. A make up PT sample must be successfully analyzed. If problems arise, appropriate action must be taken.

Excluding vinyl chloride, the laboratory may be certified for all VOCs if they successfully analyze at least 80% of the regulated VOCs. The intention of this regulation is to allow some flexibility for random misses because the VOC methods include 20 regulated analytes. A laboratory should not be certified for an analyte, which it fails repeatedly. The 80% rule for VOCs has recently been made more difficult to interpret since some PT providers are including THMs in the same vial as the VOCs. The 80% Rule does not apply to the THMs.

The Stage 1 Disinfection By Products (DBP) Rule, which became effective in January 2002, regulates the sum of five haloacetic acids (HAA5): monochloroacetic acid, dichloroacetic acid, trichloroacetic acid, monobromoacetic acid and dibromoacetic acid. Laboratories are certified for HAA5, but successful analyses of the HAA PT samples are based on the results for the individual compounds. The 80% Rule applies to the HAA5s, so if four of the 5 HAA5s are successfully analyzed, the laboratory may be certified for HAA5. As before, a laboratory should not be certified if the same analyte is failed repeatedly.

The DBP Rule also changed the way the trihalomethanes (THMs): chloroform, dichlorobromomethane, chlorodibromomethane and bromoform PTs are evaluated. Laboratories are still certified for total THMs but under the DBP Rule, each THM concentration must be reported, evaluated and passed individually to pass the PT sample. The DBP Rule also states that if a laboratory fails one THM, it cannot be certified for TTHMs, but must analyze another PT sample and pass all four of the THMs in a PT sample to be certified to analyze compliance monitoring samples for total trihalomethanes.

The following table summarizes the 80% Rule.

Analyte(s)	PT Success Requirement
Vinyl Chloride	100%
20 VOCs	80% ¹
4 THMs	100%
5 HAA5s	80% ¹

¹ A laboratory should not maintain certification for analyte(s) which it repeatedly fails.

4.7.2.2 Quality Control Samples: At least once each quarter, the laboratory should analyze a quality control sample for the analytes for which they are certified. The sample should be prepared from a source other than that from which their working standards are prepared. If errors exceed limits specified in the methods, corrective action must be taken and documented, and a follow-up quality control sample analyzed as soon as possible to demonstrate the problem has been corrected.

4.7.2.3 Calibration Curve: Calibration requirements in the methods must be followed. If there are no calibration requirements in the method, the following are guidelines to be used. At the beginning of each day that samples are to be analyzed, a calibration curve covering the sample concentration range and all target analytes should be generated according to the approved SOP. Depending on concentration ranges, the curve should be composed of three or more points. Field measurements (e.g. pH and chlorine residual) must be made on instruments which have been properly calibrated as specified in the method or instrument manual and checked each day of use. The less precise the measurement, the greater the number of concentrations which should be included in the calibration curve.

4.7.2.4 Calibration Check: The calibration for some methods is so time-consuming that 4.7.2.3 is impractical on a daily basis. Where the determinative time is extensive such as Methods 508/508.1, 515.1, 524.2, 525.2, etc. and the instrument is very stable, the calibration curve should be initially developed as specified in 4.7.2.3. Thereafter, each day analyses are performed, this curve should be verified by analysis of at least one standard for each of the target analytes at the expected concentration range. This verification should be done at both the beginning and end of the analyses. All checks must be within the control limits specified in the method or the system must be recalibrated as specified in 4.7.2.3. The concentration of the check standard should vary from day to day across the range of analyte concentrations being measured.

For some methods an initial conditioning injection needs to be made to deactivate active sites that may have developed overnight. Depending on the method, the blank may be appropriate for this.

Specific calibration requirements in the methods must be followed if different than the above.

It is recommended that a calibration standard of one component of a multicomponent analyte (PCBs, toxaphene or chlordane) also be analyzed each day or work shift. By rotating the analyte chosen, continuing calibration data can be obtained on all the multicomponent analytes over a period of one to two weeks. If a positive for a multicomponent analyte is found in a sample, a calibration check for that analyte should be performed as soon as possible.

4.7.2.5 Blanks: Requirements in the methods must be followed. A laboratory reagent blank should be carried through the full analytical procedure with every sample batch. In general, results from laboratory reagent blanks should not exceed the laboratory's MRL.

4.7.2.6 Laboratory Fortified Blanks: Requirements in the methods must be followed. For organic methods, LFBs should be analyzed across the range of analyte concentrations being measured. For some inorganic methods the level at which the LFB must be analyzed is specified in the method, and some methods require that a laboratory fortified blank at ten times the MDL or a mid level concentration be analyzed with each batch of samples. Precision and accuracy data must be documented for this determination. In addition, the analyst should routinely verify the minimum reporting limit (if one is used) for each analyte by analyzing a laboratory fortified blank at the minimum reporting level.

4.7.2.7 Laboratory Fortified Sample Matrix: Laboratory fortified sample matrix requirements in the methods must be met. If there are no laboratory fortified sample matrix requirements in the method, the following are guidelines to be used. The laboratory should add a known quantity of analytes to a percentage (to be described in the approved SOP) of the routine samples to determine sample matrix interference. The fortified concentration should not be less than the concentration of the sample selected for fortification unless specified by the method. If the sample concentration is unknown or less than detectable, the analyst should choose an appropriate concentration (e.g., a percentage of the MCL or mid point in the calibration range). Over time, samples from all routine sample sources should be fortified. The procedure should be described in the SOP. If any of these checks are not within the criteria specified in the method or control limits specified in 4.7.2.7, and the laboratory performance is in control, the result for that sample must be flagged to inform the data user that the results are suspect due to matrix effects.

4.7.2.8 Control Charts: Control charts for accuracy and precision, generated from laboratory fortified blanks (LFBs) should be maintained and used by the laboratory. Until sufficient data are available from the laboratory, usually a minimum of 20 to 30 test results on a specific analysis, the laboratory should use the control limits specified in the methods. If there are no control limits specified in the method, the limits may be statistically calibrated using the procedure below.

When sufficient data become available, the laboratory should develop LFB control charts from the mean percent recovery (\bar{x}) and the standard deviation (S) of the percent recovery for the QC checks specified above (see Standard Methods for the Examination of Water and Wastewater, part 1020B, or similar QC reference texts for

further information). These data are used to establish upper and lower control limits as follows:

$$\text{upper control limit} = \bar{x} + 3S \quad (\text{upper warning limit} + 2S)$$

$$\text{lower control limit} = \bar{x} - 3S \quad (\text{lower warning limit} - 2S)$$

After each five to ten new recovery measurements, new control limits should be calculated using the most recent 20-30 data points. These calculated control limits should not exceed those established in the method. If any of these control limits are tighter than the method specifications, the laboratory should use the tighter criteria.

4.7.2.9 Initial Demonstration of Capability: Before beginning the analysis of compliance samples, an initial demonstration of capability (IDC) must be performed for each method. The IDC includes a demonstration of the ability to achieve a low background, the precision and accuracy required by the method, and determination of the method detection limit (MDL)(see below). It is recommended that an IDC be performed for each instrument. It is also recommended that an IDC be performed by each analyst. In addition, it is recommended that the IDC also address the variability introduced if more than one sample preparation technician is used. Precision, accuracy and MDL should be similar for each technician. The analyst should recalculate IDCs when a change in the method, analyst or instrument is made which could affect the precision or accuracy. Minor changes should prompt a check to ascertain that the precision, accuracy and sensitivity have been maintained.

4.7.2.10 Quantitation of Multicomponent Organic Analytes (toxaphene, chlordane and PCBs) The quantitation of multicomponent analytes requires professional judgment on the part of the analyst. This is required due to the complex nature of the chromatography involved, sample weathering, degradation and interferences that may be present in the samples. The pattern of peaks found in the sample should be examined carefully and compared to a standard. The peaks in the sample that match the peak ratios in the standard can be used in quantitation. Peaks that have obvious interferences (such as pesticides or phthalates or peaks exhibiting poor peak shape) or appear to have been degraded or weathered should not be used for quantitation. A representative number (5-9) of peaks is suggested. Peak area should be used for quantitation and the analyst should ensure that the samples and standards have been integrated in the same manner. Quantitation can be done by using the total peak area or height (comparing the area of the 5-9 peaks used for quantitation of the sample to the area of the standard) or by calculating each peak separately (using area) and taking the average concentration of the 5-9 peaks. Because of factors such as peak shape and baseline rise, the most accurate quantitation is obtained when the concentration of the sample closely matches that of the standard (e.g., within 20% of the standard). See EPA Method 8081, Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique, (EPA SW 846 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, Third Edition) for a more detailed discussion of quantitation of multicomponent analytes.

Note: PCBs are qualitatively identified as Aroclors and measured for compliance purposes as decachlorobiphenyl. Chlordane is regulated as technical chlordane, a mixture of at least 11 major components and 30 minor ones.

4.7.2.11 MDL Calculation: Most methods require initial MDL calculations for all analytes and certification officers should require the laboratories to calculate their detection limits for all regulated contaminants. If there is no procedure to determine the detection limits in the method, it should be determined in accordance with the procedure given in 40 CFR 136, Appendix B. The CFR, at 141.24(f)(17)(ii)(C) requires an MDL of 0.0005 mg/L be attained for VOCs, and 141.89(a)(1)(iii) requires an MDL of 0.001 mg/L be attained for lead if the lab will be processing source water composite samples. For inorganics and SOCs, a method detection limit of 1/5 of the MCL must be attained for compositing [CFR 141.23(a)(4)] and [CFR 141.24(f)(10)]. VOCs should not be composited. The SOC detection limits listed at CFR (141.24(h)(18) are required to reduce monitoring (CFR 141.24(f)(11)(iv).

Sample preparation and analyses for the MDL calculation should be made over a period of at least three days to include day-to-day variation as an additional source of error. The analyst should determine MDLs initially, when any change is made which could affect the MDLs, or more frequently if required by the method. (Inorganic methods may require MDLs to be determined differently, and in all cases the methods must be followed.) In addition, the analyst must demonstrate low level capability on an ongoing basis through an MDL determination or repeated low level analyses (MRL).

The calculation of MDLs by the CFR procedure may not be adequate for toxaphene and chlordane because they require pattern or peak profile recognition for identification. Presently, no standard procedure exists, so it is recommended that the MDL be defined as the lowest concentration for which pattern recognition is possible. Pattern recognition is used for qualitative identification of PCBs as Aroclors. Quantitation of PCBs is achieved by conversion of PCBs to decachlorobiphenyl (DCB).

4.7.2.12 Low Level Quantitation: The laboratory's minimum reporting limits (MRL) should be reported to the client along with the data. The reporting limit must be below the MCL. Laboratories should run a LFB at or below their MRL every analysis day and should not report contaminants at levels less than the level at which they routinely analyze their lowest standard. While this is a scientifically sound practice, whether it is an acceptable practice will depend on State and Federal reporting requirements. It is important for users of data to understand the statistical and qualitative significance of the data. Laboratories may be required by the States to achieve a specific MDL or quantitation limit more stringent than that required by EPA.

4.8. Records and Data Reporting

4.8.1 Legal Defensibility: Compliance monitoring data should be made legally defensible by keeping thorough and accurate records. The QA plan and/or SOPs must describe the policies and procedures used by the facility for record integrity, retention and storage. If samples are expected to become part of a legal action, chain of custody procedures should be used (See Appendix A).

4.8.2 Maintenance of Records: The laboratory should maintain easily accessible records for five years or until the next certification data audit is complete, whichever is longer. This includes sampling information, all raw data, calculations, and quality control data. These data files may be either hard copy, microfiche or electronic. Electronic data should always be backed up by protected tape or disk or hard copy. If the laboratory changes its computer hardware or software, it should make provisions for transferring old data to the new system so that it remains retrievable within the time frames specified above. Changes in ownership, mergers, or closures of laboratories do not eliminate these requirements. The client water system should be provided with the laboratory's records retention policies. Data, which is expected to become part of a legal action, may need to be maintained for a longer period of time.

4.8.3 Sampling Records: Data should be recorded in ink with any changes lined through such that the original entry is visible. Data may also be kept electronically. Changes must be initialed and dated. The following information should be readily available:

4.8.3.1 Date, location [including name of utility and Public Water Supply (PWS) ID number], site within the system, time of sampling, name, organization and phone number of the sampler, and analyses required;

4.8.3.2 Identification of the sample as to whether it is a routine distribution system sample, check sample, raw or finished water sample, repeat or confirmation sample or other special purpose sample;

4.8.3.3 Date of receipt of the sample;

4.8.3.4 Sample volume/weight, container type, preservation and holding time and condition on receipt;

4.8.3.5 pH and disinfectant residual at time of sampling (if required) (from plant records);

4.8.3.6 Transportation and delivery of the sample (person/carrier, conditions).

4.8.4 Analytical Records Data should be recorded in ink with any changes lined through such that original entry is visible. Changes must be initialed and dated. The following information should be readily available:

4.8.4.1 Laboratory and persons responsible for performing analysis;

4.8.4.2 Analytical techniques/methods used;

4.8.4.3 Date and time of analysis;

4.8.4.4 Results of sample and quality control analyses;

4.8.4.5 Calibration and standards information.

4.8.4.6 Analyst and technician Initial Demonstration of Capability documentation should be kept on file as well as results of proficiency testing.

4.8.5 Reconstruction of Data: Adequate information should be available to allow the auditor to reconstruct the final results for compliance samples and PT samples.

4.8.6 Computer programs: Computer programs should be verified initially and periodically by manual calculations and the calculations should be available for inspection. Access to computer programs and electronic data must be limited to appropriate personnel.

4.9. Action in Response to Noncompliant Laboratory Results

When a laboratory is responsible, either by contract or State policy, to report sample results, which would indicate a system is out of compliance, the laboratory must promptly notify the proper authority. The authority, in turn, must request the water utility to resample from the same sampling location(s) immediately.

5.0 Requirements for Maintaining Certification

5.1 Periodic PT Proficiency Testing (PT) Samples. Certified drinking water laboratories must satisfactorily analyze WS PT samples on an annual basis for each chemical contaminant for which certification has been granted and for each method used to analyze compliance monitoring samples. Results must be within the acceptance limits established by USEPA for each analysis. Laboratories must use the same PT sample supplier for at least one calendar year before changing suppliers. PT samples must be analyzed in the same manner as routine samples. The laboratory should provide documentation that the person(s) analyzing any PT sample is a laboratory employee that routinely analyzes drinking water compliance samples.

5.2 Methodology. Laboratories must use methodologies listed in Tables 2, 3 and 6 or otherwise approved by USEPA for compliance with the Safe Drinking Water Act.

5.3 Notification of major changes. Certified laboratories should notify the certification officer, in writing, within 30 days of major changes in personnel, equipment, or laboratory location. A major change in personnel is defined as the loss or replacement of the laboratory supervisor or a situation in which a trained and experienced analyst is no longer available to analyze a particular parameter for which certification has been granted. The certification officer should discuss the situation with the laboratory supervisor and establish a schedule for the laboratory to address major changes. If the certification officer determines that the laboratory can no longer produce valid data, the certification officer should follow the

procedure for revocation of certification.

5.4 On-Site Evaluation. In order to be satisfied that the laboratory is maintaining the required standards of quality for certification, the laboratory must pass an on-site evaluation, which will be conducted at least every three years.

6. Annual Review of Certification Status.

In February of each year, the certification status of the certified laboratories will be reviewed. The laboratory's performance on the WS PT studies will be evaluated. Based on this review, a laboratory's certification status may be changed. Laboratories and Drinking Water Branch at IDEM will be informed of any changes in status.

7. Downgrading or Revoking Certification Status

7.1 Downgrading Certification Status. A laboratory may be downgraded to a Provisionally Certified status for:

7.1.1 Failure to analyze a WS PT sample within the acceptance limits,

7.1.2 Failure to notify ISDH within 30 days of a major change which could impair the results of the laboratory, or

7.1.3 Failure to maintain required standards based on the on-site evaluation.

7.2 Revoking Certification Status. A laboratory may be downgraded to a Not Certified status for:

7.2.1 Failure to analyze a WS PT samples for a particular contaminant within the acceptance limits,

7.2.2 Failure to correct identified deficiencies by the time specified by the certifying authority, or

7.2.3 Falsification of data or other deceptive practices.

7.3 Reinstatement of Certification. Certification may be reinstated when and if the laboratory can demonstrate to the certifying authority that the deficiencies causing downgrading or revoking certification have been corrected. This may include an on-site evaluation, analysis of additional WS PT samples or other measures deemed appropriate by the certifying authority.

8. Reciprocity: The State of Indiana may elect to enter into agreements with the governments of other states or agencies of the federal government for recognition of their environmental laboratory inspections and certifications if such certification programs are judged to be equivalent to those in Indiana. Laboratories in states not having reciprocal agreements with Indiana may request certification from ISDH. These laboratories will be evaluated by review of the most recent home state on-site evaluation report and results of the two most recent WS PT evaluation sample sets by the certification officer.

9. Requirements for Administrative Certification.

9.1 In order to allow laboratories to improve their certification status between on-site evaluations, laboratories may request administrative certification for analytes which become regulated as the result of changing regulations and for which the laboratory has developed analytical capability.

9.2 The following documentation must be provided:

9.2.1 the method to be used for drinking water analysis;

9.2.2 a summary of initial performance data including: method detection limit per 40 CFR Part 136, appendix B, initial demonstration of capabilities (i.e. precision and accuracy) and calibration data (such as, initial calibration results, including a raw chromatogram to show separation of all compounds for GC, GC/MS or HPLC methods, if applicable); and,

9.2.3 results for two (2) successfully analyzed WS PT samples.

9.3. In order to be administratively certified, the laboratory must:

9.3.1 meet the requirements for acceptable performance outlined in the method and

9.3.2 pass two (2) WS PT samples within the last year for each analyte to be certified.

9.4. This certification will be valid until the laboratory's next scheduled on-site evaluation unless personnel changes occur or the laboratory's performance on the WS PT samples causes the laboratory's certification to be downgraded.

TABLE 1

APPROVED METHODOLOGY FOR INORGANIC CONTAMINANTS¹⁵

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ²⁷	<u>Other</u>
Alkalinity	Titrimetric Elec. titration		D1067-92, 02 B	2320B	2320 B-97	I-1030-85 ⁵
Antimony	Inductively coupled plasma mass spectrometry	200.8 ²				
	Atomic absorption; gaseous hydride platform furnace	200.9 ²	D3697-92, 02	3113B	3113 B-99	
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Arsenic ¹⁷	Inductively coupled plasma mass spectrometry	200.8 ²				
	Atomic absorption; platform furnace gaseous hydride	200.9 ²	D2972-97, 03 C D2972-97, 02 B	3113B 3114B	3113 B-99 3114 B-97	
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Asbestos	Transmission electron microscopy	100.1 ⁹				
	Transmission electron microscopy	100.2 ¹⁰				
Barium	Inductively coupled plasma emission spectrometry	200.7 ²		3120B	3120 B-99	

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ²⁷	<u>Other</u>
Barium	Inductively coupled plasma mass spectrometry	200.8 ²				
	Atomic absorption; direct aspiration			3111D	3111 D-99	
	Furnace			3113B	3113 B-99	
Beryllium	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
	Inductively coupled plasma emission spectrometry	200.7 ²		3120B	3120B-99	
	Inductively coupled plasma mass spectrometry	200.8 ²				
	Atomic absorption platform furnace	200.9 ²		D3645-97, 03 B	3113B	3113B-99
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Bromate	Ion chromatography	300.1	D6581-00			
	Ion chromatography & post column reaction	317.0, Rev. 2.0				
	Ion chromatography & post column reaction	326.0				
	IC/ICP-MS	321.8				
Bromide	Ion chromatography	300.0	D6581-00			
	Ion chromatography	300.1				
	Ion chromatography & post column reaction	317.0, Rev. 2.0				
	Ion chromatography & post column reaction	326.0				

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ²⁷	<u>Other</u>
Cadmium	Inductively coupled plasma emission spectrometry	200.7 ²				
	Inductively coupled plasma mass spectrometry	200.8 ²				
	Atomic absorption platform furnace	200.9 ²		3113B	3113B-99	
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Calcium	EDTA titrimetric		D511-93, 03 A	3500-Ca D (3500-Ca B, 20 th and 21 st)	3500-Ca B-97	
	Atomic absorption; Direct aspiration		D511-93, 03 B	3111B	3111B-99	
	Inductively coupled plasma Emission spectrometry	200.7 ²		3120B	3120B-99	
	Ion chromatography Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸		D6919-03		
Chlorite	Ion chromatography	300.0				
	Ion chromatography	300.1				
	Ion chromatography	317.0 Rev 1.1				
	Ion chromatography	326.0				
Chlorite	Ion chromatography	327.0 Rev 1.1				
	Amperometric titration Amperometric titration			4500-ClO ₂ E 4500-ClO ₂ E-00		

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ²⁷	<u>Other</u>
Chromium	Inductively coupled plasma Emission spectrometry	200.7 ²		3120B	3120B-99	
	Inductively coupled plasma Mass spectrometry	200.8 ²				
	Atomic absorption Platform Furnace	200.9 ²		3113B	3113B-99	
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Conductivity	Conductance		D1125-95(99)A	2510B	2510B-99	
Copper	Atomic absorption furnace		D1688-95, 02C	3113B	3113B-99	
	direct aspiration		D1668-95, 02 A	3111B	3111B-99	
	Inductively coupled plasma emission spectrometry	200.7 ²		3120B	3120B-99	
	mass spectrometry	200.8 ²				
	Atomic absorption platform	200.9 ²				
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Cyanide	Manual distillation: followed by Spectrophotometric, amenable		D2036-98A D2036-98B	4500-CN C 4500-CN G	4500-CN G-99	
	Spectrophotometric, manual Semi-automated	335.4 ⁶	D2036-98A	4500-CN E	4500-CN E-99	I-3300-85 ⁵
	Ion selective electrode method			4500-CN F	4500-CN G-99	
	UV/distillation/spectrophotometri c					Kelada 01 ²²
	Distillation/spectrophotometric					QuikChem 10-204-00-1-X ²³

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ²⁷	<u>Other</u>
Cyanide	Ligand exchange-amperometry					OIA-1677, DW ²⁴
Fluoride	Ion chromatography	300.0 ⁶	D4327-97, 03	4110B	4110B-00	
	Colorimetric SPADNS, with distillation			4500-F B,D	4500-F B, D-97	
	Manual electrode		D1179-93, 99B	4500-F C	4500-F C-97	380-75WE ¹¹
	Automated electrode Automated alizarin fluoride blue with distillation			4500-F E	4500-F C-97	129-71W ¹¹
Lead ¹⁹	Atomic absorption; furnace		D3559-96, 03 D	3113B	3113B-99	
	Inductively coupled plasma mass spectrometry	200.8 ²				
	Atomic absorption platform	200.9 ²				
	Differential pulse anodic stripping voltametry					Method 1001 ¹⁸
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Magnesium	Atomic absorption		D-511-93, 03 B	3111B		3111B-99
	Inductively coupled plasma emission spectrometry	200.7 ²		3120B		3120B-99
	Complexation titrimetric methods		D-511-93, 03 A	3500-Mg E (3500-Mg B, 20 th and 21 st)	3500-Mg B-97	
	Ion chromatography Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸		D6919-03		

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ²⁷	<u>Other</u>
Mercury	Manual cold vapor	245.1 ²	D3223-97, 02	3112B	3112B-99	
	Automated cold vapor	245.2 ¹				
	Inductively coupled plasma mass spectrometry	200.8 ²				
Nickel	Inductively coupled plasma emission spectrometry	200.7 ²		3120B	3120B-99	
	mass spectrometry	200.8 ²				
	Atomic absorption platform direct furnace	200.9 ²		3111B 3113B	3111B-99 3113B-99	
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Nitrate-N	Ion chromatography	300.0 ⁶	D4327-97, 03	4110B	4110B-00	B-1011 ⁸
	Ion chromatography	300.1 ²⁶				
	Automated cadmium reduction	353.2 ⁶	D3867-90A	4500-NO ₃ F	4500-NO ₃ F-00	
	Ion selective electrode			4500-NO ₃ D	4500-NO ₃ D-00	601 ⁷
	Manual cadmium reduction		D3867-90B	4500-NO ₃ E	4500-NO ₃ E-00	
	Capillary ion electrophoresis					D6508, Rev. 2 ²⁵
Nitrite-N	Ion chromatography	300.0 ⁶	D4327-97, 03	4110B	4110B-00	B-1011 ⁸
	Ion chromatography	300.1 ²⁶				
	Cadmium reduction: automated	353.2 ⁶	D3867-90A	4500-NO ₃ F	4500-NO ₃ F-00	
	manual		D3867-90B	4500-NO ₃ E	4500-NO ₃ E-00	
	Spectrophotometric			4500-NO ₂ B	4500-NO ₃ B-00	
	Capillary ion electrophoresis					D6508, Rev. 2 ²⁵
Ortho-phosphate ¹²	Colorimetric: ascorbic acid manual: single reagent		D515-88A	4500-P E		
	automated	365.1 ⁶		4500-P F		
	Colorimetric: phosphomolybdate					I-1601-85 ⁵

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ²⁷	<u>Other</u>
Ortho-phosphate ¹²	automated segmented flow automated discrete flow Ion chromatography	300.0 ⁶	D-4327-97, 03	4110 B		I-2601-90 ⁵ I-2598-85 ⁵
Perchlorate	Ion chromatography	314.0 ²⁰				
pH	Electrometric	150.1 ¹	D1293-95, 99	4500-H B	4500-H B-00	
	Electrometric	150.2 ¹				
Residual disinfectant						
Free chlorine	Amperometric titration		D1253-03	4500-Cl D	4500-Cl D-00	
	DPD titrimetric			4500-Cl F	4500-Cl F-00	
	DPD colorimetric method			4500-Cl G	4500-Cl G-00	
	Syringaldazine (FACTS)			4500-Cl H	4500-Cl-H-00	
Combined chlorine	Amperometric titration		D1253-03	4500-Cl D	4500-Cl D-00	
	DPD titrimetric			4500-Cl F	4500-Cl F-00	
	DPD colorimetric method			4500-Cl G	4500-Cl G-00	
Total chlorine	Amperometric titration		D1253-03	4500-Cl D	4500-Cl D-00	
	Amperometric titration-low level			4500-Cl E	4500-Cl E-00	
Total chlorine	DPD titrimetric			4500-Cl F	4500-Cl F-00	
	DPD colorimetric method			4500-Cl G	4500-Cl I-00	
	Iodometric electrode			4500-Cl I	4500-Cl I-00	
	DPD colorimetric method			4500-ClO ₂ D		
Chlorine dioxide	Amperometric Method II			4500-ClO ₂ E	4500-ClO ₂ E-00	
	Lissamine Green, spectrophotometric	327.0 Rev 1.1				
	Indigo method			4500-O ₃ B	4500-O ₃ B-97	
Selenium	Atomic absorption: gaseous hydride		D-3859-98, 03A	3114B	3114B-97	
	Inductively coupled plasma mass spectrometry	200.8 ²				

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ²⁷	<u>Other</u>
Selenium	Atomic absorption: platform furnace	200.9 ²				
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸	D-3859-98, 03	3113B	3113B-99	
Silica	Colorimetric, molybdate blue manual					I-1700-85 ⁵
	Automated-segmented flow Colorimetric Molybdosilicate		D-859-94, 00			I-2700-85 ⁵
	Heteropoly blue			4500-Si D [4500-Si O ₂ C, 20 th and 21 st]	4500-Si O ₂ C-97	
				4500-Si E {4500-SiO ₂ D, 20 th and 21 st }	4500-Si O ₂ D-97	
	Automated method for molybdate-reactive silica			4500-Si F [4500-SiO ₂ E, 20 th and 21 st]	4500-Si O ₂ E-97	
	Inductively coupled plasma emission spectrometry	200.7 ²			3120B	3120B-99
Sodium	Inductively coupled plasma emission spectrometry	200.7 ²				
	Atomic absorption: direct aspiration					
	Ion chromatography		D6919-03			
Specific ultraviolet absorbance (SUVA)	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
				5910B (UV ₂₅₄)		

<u>Contaminant</u>	<u>Methodology</u> ¹⁶	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴ 2550B	<u>SM Online</u> ²⁷ 2550-00	<u>Other</u>
Temperature	Thermometric					
Thallium	Inductively coupled plasma mass spectrometry	200.8 ²				
	Atomic absorption: platform	200.9 ²				
	Axially viewed inductively coupled plasma-atomic emission spectrometry (AVICP-AES)	200.5 Revision 4.2 ²⁸				
Total and dissolved organic carbon (TOC)	High-temperature combustion			5310 B	5310 B-00	
	Persulfate-ultraviolet or heated-persulfate oxidation			5310 C	5310 C-00	
	Wet-oxidation			5310D	5310 D-00	
	Persulfate-ultraviolet or heated-persulfate oxidation	415.3 Rev 1.1				
Turbidity	Nephelometric			2130B		
	Nephelometric	180.1 ¹³				
	Great Lakes Instruments					Method 2 ¹⁴
	Hach Filter Trak					10133 ²⁴

NOTES:

¹ Methods 150.1, 150.2 and 245.2 are available from USEPA, NERL, Cincinnati, OH 45268. The identical methods were formerly in "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, March 1983, which is available at NTIS, PB84-28677.

² "Methods for the Determination of Metals in Environmental Samples-Supplement I", EPA-600/R-94-111, May 1994. Available at NTIS, PB94-184942.

³ The procedures shall be accordance with the *Annual Book of ASTM Standards*, 1994, 1996 and 1999, Vols. 11.01 and 11.02, American Society for Testing and Materials; any year containing the cited version of the method may be used. The previous versions of D1688-95A, D1668-95C (copper), D3559-95D (lead), D1293-95 (pH), D1125-91A (conductivity) and D859-94 (silica) are also approved. These previous versions of D1668-90A, D3559-90D, D1293-84, D1125-91A AND D859-88 respectively are located in the *Annual Book of ATSM Standards, 1994* or any year containing the cited version of the method,, *Vols. 11.01*. Copies may be obtained from the American Society of Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

⁴ 18th, 19th, 20th and 21st editions and the Supplement to the 19th edition of *Standard Methods for the Examination of Water and Wastewater*, 1992, 1995 and 1998, respectively, American Public Health Association, any version may be used, except that the versions of methods 3111B, 3111D, 3113B and 3114B in the 20th edition may not be used. Methods for bromate, chlorite and residual disinfectant are from the 19th, 20th and 21st editions only. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.

⁵ Method I-2601-90, *Methods for Analysis by the U.S. Geological Survey National Water Quality Laboratory-Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments*, Open File Report 93-125, 1993; For Methods I-1030-85, I-1601-85, I-2598-85, I-2700-85, and I-330-85 See *Techniques of Water Resources Investigation of the U.S. Geological Survey*, Book 5, Chapter A-1, 3rd ed., 1989. Available from Information Services, U.S. Geological Survey, Federal Center, Box 25286, Denver, CO 80225-0425.

⁶ “Methods for the Determination of Inorganic Substances in Environmental Samples”, EPA-600/R-93-100, August 1993. Available at NTIS, PB94-121811.

⁷ This procedure shall be done in accordance with Technical Bulletin 601, *Standard Method of Test for Nitrate in Drinking Water*, July 1994, PN 221890-001, Analytical Technology, Inc. Copies may be obtained from ATI Orion, 529 Main Street, Boston, MA 02129.

⁸ Method B-1011, *Waters Test Method for Determination of Nitrite/Nitrate in Water Using Single Ion Chromatography*, Millipore Corporation, Waters Chromatography Division, 34 Maple Street, Milford, MA 01757.

⁹ Method 100.1, “Analytical Method for Determination of Asbestos Fibers in Water”, EPA-600/4-83-043, EPA, September 1983. Available at NTIS, PB83-260471.

¹⁰ Method 100.2, “Determination of Asbestos Structure Over 10- μ m in Length in Drinking Water”, EPA-600/R-94-134, EPA, June 1994. Available at NTIS, PB94-201902.

¹¹ Industrial Method No. 129-71W, *Fluoride in Water and Wastewater*, December 1972, and Method No. 380-75WE, “Fluoride in Water and Wastewater”, February 1976, Technicon Industrial Systems. Copies may be obtained from Bran & Luebbe, 1025 Busch Parkway, Buffalo Grove, IL 60089.

¹² Unfiltered, no digestion or hydrolysis.

¹³ “Methods for the Determination of Inorganic Substances in Environmental Samples”, EPA-600/R-93-100, August 1993. Available at NTIS, PB94-121811.

¹⁴ GLI Method 2, *Turbidity*, November 2, 1992, Great Lakes Instruments, Inc., 8855 North 55th Street, Milwaukee, WI 53223.

¹⁵ Criteria for analyzing arsenic, barium, beryllium, cadmium, chromium, copper, lead, nickel, selenium, sodium and thallium are contained in *Technical Notes on Drinking Water Methods*, EPA-600/R-94-173, October 1994. This document is available from NTIS, PB95-104766, US Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll-free number is 800/553-6847.

¹⁶ Because MDLs reported in EPA Methods 200.7 and 200.9 were determined using a 2X preconcentration step during sample digestion, MDLs determined when samples are analyzed by direct analysis (i.e., no sample digestion) will be higher. For direct analysis of cadmium and arsenic by Method 200.7, and arsenic by Method 3120B sample preconcentration by pneumatic nebulization may be required to achieve lower detection limits. Preconcentration may also be required for direct analysis of antimony, lead, and thallium by Method 200.9; antimony and lead by Method 3113B; and lead by Method 3559-90D unless multiple in-furnace depositions are made.

¹⁷ If ultrasonic nebulization is used for the determination of arsenic by Methods 200.7, 200.8, or SM 3120B, the arsenic must be in the pentavalent state to provide uniform signal response. For methods 200.7 and 3120B, both samples and standards must be diluted in the same mixed acid

matrix concentration of nitric and hydrochloric acid with the addition of 100 µL of 30% hydrogen peroxide per 100 mL of solution. For direct analysis of arsenic with method 200.8 using ultrasonic nebulization, samples and standards must contain one mg/L of sodium hypochlorite.

¹⁸ The description for Method Number 1001 for lead is available from Palintest LTD, 21 Kenton Lands Road, P.O. Box 18395, Erlanger, KY 41018. Or from the Hach Company, P.O. Box 389, Loveland, CO 80539.

¹⁹ If a laboratory is analyzing composite samples for lead, it must achieve a method detection limit of 0.001 mg/L using the procedures in 40 CFR, part 136, appendix B.

²⁰ This method is available from the Safe Drinking Water Hotline (800/426-4791) or electronically at www.epa.gov/safewater/methods/sourcalt.html. Information for laboratories seeking approval to analyze samples for perchlorate can find this information at www.epa.gov/OGWDW/libcint.html.

²¹ The description of the Keleda 01 method, “Keleda Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate”, Revision 1.2, August 2001, EPA#821-B-01-009 is available for the National Technical Information Service (NTIS), PB 2001-108275, 5285 Port Royal Road, Springfield, VA 22161. The toll free telephone number is 800/553-6847.

²² The description of the QuikChem Method 10-204-00-1-X, “Digestion and distillation of total cyanide in drinking and wastewaters using MICRO DIST and the determination of cyanide by flow injection analysis”, Revision 2.1, November 30, 2000, for cyanide is available from Lachat Instruments, 6645 W. Mill Rd., Milwaukee, WI 53218, USA. Phone: 414/358-4200.

²³ A description of the Hach Filter Trak Method 10133, “Determination of Turbidity by Laser Nephelometry”, January 2000, Revision 2.0, can be obtained from Hach Co., P.O. Box 389, Loveland, CO 80539-0389. Phone: 800/227-4224.

²⁴ Method OIA-1677, DW “Available Cyanide by Flow Injection , Ligand Exchange and Amperometry”, January 2004, EPA-821-R-04-001. Available from ALPKEM, A Division of OI Analytical, P.O. Box 9010, College Station, TX 77842-9010.

²⁵ Method D6508, Rev.2, “Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte”, available from Waters Corp. 34 Maple Street, Milford, MA 01757, Telephone:508/482-2131, Fax 508/482-3625.

²⁶ “Methods for the Determination of Organic and Inorganic Compounds in Drinking Water”, Vol. 1, EPA 805-R-00-014, August 2000, Available from NTIS, PB2000-106891.

²⁷ The Standard Methods Online version that is approved is indicated by the last two digits in the method number which is the year of approval by the Standard Methods Committee. Standard Methods On-line are available at <http://www.standardmethods.org>. The methods listed are the only online methods that may be used.

²⁸ EPA method 200.5, Revision 4.2, “Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry”, 2003. EPA/R-06/115. (Available at <http://epa.gov/nerlcwww/ordmeth.htm>)

TABLE 2
APPROVED METHODOLOGY FOR ORGANIC CONTAMINANTS

<u>Contaminant</u>	<u>Methodology</u> ¹	<u>Method No.</u>
<u>Non-volatile Synthetic Organic Compounds</u>		
Adipate [Di(2-ethylhexyl)adipate]	Liquid/Solid Extraction-Gas Chromatography- Photoionization Detector	506
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2
Alachlor ²	Microextraction-Gas Chromatography-Electron Capture Detector	505
	Gas Chromatography-Nitrogen/Phosphorus Detector	507
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2
	Liquid/Liquid Extraction-Gas Chromatography- Electron Capture Detector	551.1
Atrazine	Microextraction-Gas Chromatography- Electron Capture Detector	505
	Gas Chromatography-Nitrogen/Phosphorus Detector	507
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2
	Liquid/Liquid Extraction-Gas Chromatography- Electron Capture Detector	551.1
	Immunoassay	Syngenta AG-625
Carbofuran	High Performance Liquid Chromatography- Post Column Reactor	531.1
	High Performance Liquid Chromatography- Post Column Reactor	531.2
	High Performance Liquid Chromatography- Post Column Reactor	6610 B
Chlordane	Microextraction-Gas Chromatography- Electron Capture Detector	505
	Gas Chromatography-Electron Capture Detector	508
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2

TABLE 2 (CONTINUED)

<u>Contaminant</u>	<u>Methodology</u>	<u>Method No.</u>
Dalapon	Gas Chromatography-Electron Capture Detector	515.1
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	515.3
	Liquid/Liquid Microextraction-Fast Gas Chromatography-Electron Capture Detector	515.4
	Ion Exchange Liquid/Solid Extraction-Gas Chromatography-Electron Capture Detector	552.1
	Liquid/Solid Extraction-Gas Chromatography- Electron Capture Detector	552.2
	Dibromochloropropane (DBCP)	Microextraction-Gas Chromatography-Electro Capture Detector
2,4-D ⁴ (as acid, salts and esters)	Liquid/Liquid Extraction-Gas Chromatography- Electron Capture Detector	551.1
	Gas Chromatography-Electron Capture Detector	515.1
	Liquid/Solid Extraction-Gas Chromatography- Electron Capture Detector	515.2
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	515.3
	Liquid/Liquid Microextraction-Fast Gas Chromatography-Electron Capture Detector	515.4
	High Performance Liquid Chromatography-Photo Diode Array Ultraviolet Detector	555
	Gas Chromatography-Electron Capture Detector	D5317-93, 98 (03)
Dinoseb ⁴	Gas Chromatography-Electron Capture Detector	515.1
	Liquid/Solid Extraction-Gas Chromatography- Electron Capture Detector	515.2
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	515.3
	Liquid/Liquid Microextraction-Fast Gas Chromatography-Electron Capture Detector	515.4
	High Performance Liquid Chromatography-Photo Diode Array Ultraviolet Detector	555
	Diquat	Liquid/Solid Extraction-High Performance Liquid Chromatography-Ultraviolet Detector
Endothall	Liquid/Solid Extraction-Gas Chromatography- Electron Capture Detector	548.1
Endrin	Microextraction-Gas Chromatography- Electron Capture Detector	505
	Gas Chromatography-Electron Capture Detector	508
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2

TABLE 2 (CONTINUED)

<u>Contaminant</u>	<u>Methodology</u>	<u>Method No.</u>
Ethylene Dibromide (EDB)	Microextraction-Gas Chromatography-Elector Capture Detector	504.1
	Liquid/Liquid Extraction-Gas Chromatography- Election Capture Detector	551.1
Glyphosate	High Performance Liquid Chromatography- Post Column Reactor-Fluorescence Detector	547
	High Performance Liquid Chromatography- Post Column Reactor-Fluorescence Detector	6651
Haloacetic acids 5 (HAA5)	Liquid/Solid Extraction-Gas Chromatography- Electron Capture Detector	552.1
	Liquid/Liquid Extraction-Gas Chromatography- Electron Capture Detector	552.2
	Liquid/Liquid Extraction-Gas Chromatography- Electron Capture Detector	552.3
	Liquid/Solid Extraction-Gas Chromatography- Electron Capture Detector	6251B
	Liquid/Solid Extraction-Gas Chromatography- Electron Capture Detector	6251B-94
	Microextraction-Gas Chromatography- Electron Capture Detector	505
Heptachlor	Gas Chromatography-Electron Capture Detector	508
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2
	Liquid/Liquid Extraction-Gas Chromatography- Election Capture Detector	551.1
	Microextraction-Gas Chromatography- Electron Capture Detector	505
	Gas Chromatography-Electron Capture Detector	508
Heptachlor epoxide	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2
	Liquid/Liquid Extraction-Gas Chromatography- Election Capture Detector	551.1
	Microextraction-Gas Chromatography- Electron Capture Detector	505
	Gas Chromatography-Electron Capture Detector	508
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	508.1
Hexachlorobenzene	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2
	Liquid/Liquid Extraction-Gas Chromatography- Election Capture Detector	551.1
	Microextraction-Gas Chromatography- Electron Capture Detector	505
	Gas Chromatography-Electron Capture Detector	508
	Liquid/Solid Extraction- Gas Chromatography- Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2

TABLE 2 (CONTINUED)

<u>Contaminant</u>	<u>Methodology</u>	<u>Method No.</u>
Hexachlorocyclopentadiene	Microextraction-Gas Chromatography-Electron Capture Detector	505
	Gas Chromatography-Electron Capture Detector	508
	Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2
	Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector	551.1
	Lindane	Microextraction-Gas Chromatography-Electron Capture Detector
Gas Chromatography-Electron Capture Detector		508
Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector		508.1
Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry		525.2
Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector		551.1
Methoxychlor		Microextraction-Gas Chromatography-Electron Capture Detector
	Gas Chromatography-Electron Capture Detector	508
	Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector	508.1
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2
	Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector	551.1
	Oxamyl (vydate)	High Performance Liquid Chromatography-Post Column Reactor
High Performance Liquid Chromatography-Post Column Reactor		531.2
High Performance Liquid Chromatography-Post Column Reactor		6610 B
Polyaromatic Hydrocarbons Benzo(a)pyrene		Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry
	Liquid/Liquid Extraction-High Performance Liquid Chromatography-Ultraviolet and Fluorescence Detectors	550
	Liquid/Liquid Extraction-High Performance Liquid Chromatography-Ultraviolet and Fluorescence Detectors	550.1
Pentachlorophenol	Gas Chromatography-Electron Capture Detector	515.1
	Liquid/Solid Extraction-Gas Chromatography-Electron Capture Detector	515.2
	Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector	515.3
	Liquid/Liquid Microextraction-Fast Gas Chromatography-Electron Capture Detector	515.4

TABLE 2 (CONTINUED)

<u>Contaminant</u>	<u>Methodology</u>	<u>Method No.</u>	
Pentachlorophenol	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2	
	High Performance Liquid Chromatography-Photo Diode Array Ultraviolet Detector	555	
	Gas Chromatography-Electron Capture Detector	D5317-93, 98 (03)	
Phthalate [Di(2-ethylhexyl)phthalate]	Liquid/Solid Extraction-Gas Chromatography-Photoionization Detector	506	
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2	
Picloram ⁴	Gas Chromatography-Electron Capture Detector	515.1	
	Liquid/Solid Extraction-Gas Chromatography-Electron Capture Detector	515.2	
	Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector	515.3	
	Liquid/Liquid Microextraction-Fast Gas Chromatography-Electron Capture Detector	515.4	
	High Performance Liquid Chromatography-Photo Diode Array Ultraviolet Detector	555	
	Gas Chromatography-Electron Capture Detector	D5317-93 , 98 (03)	
	Polychlorinated Biphenyls ³ (screen)	Microextraction-Gas Chromatography-Electron Capture Detector	505
		Gas Chromatography-Electron Capture Detector	508
Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector		508.1	
Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry		525.2	
Packed column-Gas Chromatography		508A	
(as decachlorobiphenyl) Simazine ²	Microextraction-Gas Chromatography-Electron Capture Detector	505	
	Gas Chromatography-Nitrogen/Phosphorus Detector	507	
	Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector	508.1	
	Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry	525.2	
	Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector	551.1	
	Toxaphene	Microextraction-Gas Chromatography-Electron Capture Detector	505
		Gas Chromatography-Electron Capture Detector	508
Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector		508.1	
Liquid/Solid Extraction-Capillary Column-Gas Chromatography/Mass Spectrometry		525.2	

TABLE 2 (CONTINUED)

<u>Contaminant</u>	<u>Methodology</u>	<u>Method No.</u>
2,3,7,8 TCDD (Dioxin)	Capillary Column-High Resolution Gas Chromatography-High Resolutions Mass Spectrometry	1613
2,4,5-TP ⁴ (Silvex)	Gas Chromatography-Electron Capture Detector	515.1
	Liquid/Solid Extraction-Gas Chromatography-Electron Capture Detector	515.2
	Liquid/Solid Extraction- Gas Chromatography-Electron Capture Detector	515.3
	Liquid/Liquid Microextraction-Fast Gas Chromatography-Electron Capture Detector	515.4
	High Performance Liquid Chromatography-Photo Diode Array Ultraviolet Detector	555
	Gas Chromatography-Electron Capture Detector	D5317-93, 98 (03)
Total Trihalomethanes (TTHM)	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
	Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector	551.1
<u>Volatile Organic Compounds.</u>		
<u>(VOC)</u>		
Benzene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
Carbon Tetrachloride	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
	Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector	551.1
Chlorobenzene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
1,4-Dichorobenzene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2

TABLE 2 (CONTINUED)

<u>Contaminant</u>	<u>Methodology</u>	<u>Method No.</u>
1,2-Dichlorobenzene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
1,1-Dichloroethylene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
cis-1,2-Dichloroethylene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
trans-1,2-Dichloroethylene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
Dichloromethane	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
1,2-Dichloropropane	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
Ethylbenzene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
Styrene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
Tetrachloroethylene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
	Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector	551.1

TABLE 2 (CONTINUED)

<u>Contaminant</u>	<u>Methodology</u>	<u>Method No.</u>
Toluene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
1,2,4-Trichlorobenzene	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
1,1,1-Trichloroethane	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
1,1,2-Trichloroethane	Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector	551.1
	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
Trichloroethylene	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
	Liquid/Liquid Extraction-Gas Chromatography-Election Capture Detector	551.1
Vinyl Chloride	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2
Xylene (Total)	Purge and Trap-Capillary Column-Gas Chromatography-Photoionization Detector-Electrolytic Conductivity Detector	502.2
	Purge and Trap-Capillary Column-Gas Chromatography/Mass Spectroscopy	524.2

NOTES:

1. Methods 508A and 515.1 are in "Methods for the Determination of Organic Compounds in Drinking Water", EPA-600/4-88-039, December 1988, Revised July 1991. This document is available from NTIS, PB91-231480, US Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll-free number is 800/552-6847.

2. Methods 547, 550 and 550.1 are in “Methods for the Determination of Organic Compounds in Drinking Water-Supplement I”, EPA-600/4-90-020, July 1990. This document is available from NTIS, PB91-146027, US Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll-free number is 800/552-6847.
3. Methods 548.1, 552.1 and 555 are in “Methods for the Determination of Organic Compounds in Drinking Water-Supplement II”, EPA-600/R-92-129, August 1992. . This document is available from NTIS, 92-207703, US Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll-free number is 800/552-6847.
4. Methods 502.2, 504.1, 505, 506, 507, 508, 508.1, 515.2, 524.2, 525.2, 531.1, 551.1, and 552.2 are in “Methods for the Determination of Organic Compounds in Drinking Water-Supplement III, EPA/600/R-95-131, August 1995. This document is available from NTIS, 95-261616, US Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll-free number is 800/552-6847.
5. Method 1613 is titled, “Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope-Dilution HRGC/HRMS”, EPA-821-B-94-005, October 1994. This document is available from NTIS, 95-104774, US Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161. The toll-free number is 800/552-6847.
6. Method 6651 shall be followed in accordance with *Standard Methods for the Examination of Water and Wastewater*, 18th edition, 1992, 19th edition, 1995 and 20th edition, 1998, American Public Health Association. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.
7. Method 6610 B shall be followed in accordance with the Supplement to the 18th edition of *Standard Methods for the Examination of Water and Wastewater*, 1994, with the 19th edition of *Standard Methods for the Examination of Water and Wastewater*, 1995, American Public Health Association, with the 20th edition of *Standard Methods for the Examination of Water and Wastewater*, 1998, American Public Health Association or with the 21st edition of *Standard Methods for the Examination of Water and Wastewater*, 2005. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.
8. Method 6251 B shall be followed in accordance with the 19th edition of *Standard Methods for the Examination of Water and Wastewater*, 1995, American Public Health Association or with the 20th edition of *Standard Methods for the Examination of Water and Wastewater*, 1998, American Public Health Association. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.
9. Methods 507, 508 and 515.1 for UCMR analytes are in “Methods for the Determination of Organic Compounds in Drinking Water”, EPA-600/4-88-039, December 1988, revised July 1991.
10. Methods 515.2 and 524.2 for UCMR analytes are contained in “Methods for the Determination of Organic Compounds in Drinking Water-Supplement II”, EPA-600/R-92-129, August 1992.
11. Method D5812-96 is found in the Annual Book of ASTM Standards, 1998, Vol. 11.02. Methods 5790-95, D5475-93 and D5317-93 are found in the Annual Book of ASTM Standards, 1996 and 1998, Vol. 11.02.
12. AOAC methods are found in the Official Methods of Analysis of the AOAC (Association of Official Analytical Chemist) International, 16th edition, 4th revision, 1998, Volume I. Copies may be obtained from, AOAC International. First Union National Bank Lockbox, P.O. Box 75198, Baltimore, MD 21275-5198.
13. Method 6200B is found in the 20th edition of *Standard Methods for the Examination of Water and Wastewater*, 1998, American Public Health Association. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.
14. Other analytical test procedures are contained in “Technical Notes on Drinking Water Methods”, EPA-600/R-94-173, October 1994, NTIS, B95-104766.

15. Methods 515.3 and 549.2 are available from U.S. Environmental Protection Agency, National Exposure Research Laboratory (NERL) - Cincinnati, 26 West Martin Luther King Drive, Cincinnati, OH 45268.
16. ASTM Method 5317-93 is available in the *Annual Book of ASTM Standards*, 1996, Vol. 11.02, American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428, or in any edition published after 1993 that contains the cited version.
17. EPA Method 515.4, "Determination of Chlorinated Acids in Drinking Water by Liquid-Liquid Microextraction, Derivatization and Fast Gas Chromatography with Electron Capture Detection", Revision 1.0, April 2000, EPA/815/B-00/001, can be accessed and downloaded directly on-line at www.epa.gov/safewater/methods/sourcalt.html.
18. The Syngenta AG-635, "Atrazine in Drinking Water by Immunoassay", February 2001 is available from Syngenta Crop Protection, Inc., 410 Swing Road, Post Office Box 18300, Greensboro, NC 27419. Phone number 336/632-6000. This method may not be used in any system where chlorine dioxide is used for drinking water treatment. In samples for all other systems, any result for atrazine generated by Method AG-635 that is greater than one-half the MCL must be confirmed using another approved method for this contaminant and should use additional volume of the original sample collected for compliance monitoring. In instances where a result for Method AG-635 triggers such confirmatory testing, the confirmatory result is to be used to determine compliance.
19. Method 531.2, "Measurement of N-methylcarbamoyloximes and N-methylcarbamates in Water by Direct Aqueous Injection HPLC with Postcolumn Derivatization:", Revision 1.0, September 2001, EPA 815/B/01/002 can be accessed and downloaded directly on-line at www.epa.gov/safewater/methods/sourcalt.html.
20. The Standard Methods Online version that is approved is indicated by the last two digits in the method number which is the year of approval by the Standard Methods Committee. Standard Methods On-line are available at <http://www.standardmethods.org>. The methods listed are the only online methods that may be used.

FOOTNOTES:

¹ Substitution of the detector specified in Method 505, 507, 508, or 508.1 for the purpose of achieving lower detection limits is allowed as follows. Either an electron capture or nitrogen phosphorus detector may be used provided all regulatory requirements and quality control criteria are met.

² PCBs are qualitatively identified as Aroclors and measured for compliance purposes as decachlorobiphenyl. Users of Method 505 may have more difficulty in achieving the required detection limits than users of Methods 508, 508.1 or 525.2.

³ Accurate determination of the chlorinated esters requires hydrolysis of the sample as described in EPA Methods 515.1, 515.2, 515.3, 515.4 and 555, and ASTM Method D5317-93.

TABLE 3

SAMPLE COLLECTION, HANDLING AND PRESERVATION FOR INORGANIC CONTAMINANTS¹

<u>Contaminant</u>	<u>Preservative</u> ²	<u>Container</u> ³	<u>Maximum Holding Time</u> ⁴
Alkalinity	Cool, 4°C	P or G	14 days
Antimony	Conc. HNO ₃ to pH<2	P or G	6 months
Arsenic	Conc. HNO ₃ to pH<2	P or G	6 months
Asbestos	Cool, 4°C	P or G	48 hours ⁵
Barium	Conc. HNO ₃ to pH<2	P or G	6 months
Beryllium	Conc. HNO ₃ to pH<2	P or G	6 months
Cadmium	Conc. HNO ₃ to pH<2	P or G	6 months
Calcium	Conc. HNO ₃ to pH<2	P or G	6 months
Chloride	None	P or G	28 days
Chromium	Conc. HNO ₃ to pH<2	P or G	6 months
Copper	Conc. HNO ₃ to pH<2	P or G	6 months
Cyanide	NaOH to pH>12, cool, 4°C	P or G	14 days
Fluoride	None	P or G	28 days
Free Chlorine Residual	None	P or G	Analyze immediately ⁶
Lead	Conc. HNO ₃ to pH<2	P or G	6 months
Mercury	Conc. HNO ₃ to pH<2	P or G	28 days
Nickel	Conc. HNO ₃ to pH<2	P or G	6 months
Nitrate-N	Cool, 4°C	P or G	48 hours ⁷
Total Nitrate/Nitrite	H ₂ SO ₄ to pH<2	P or G	14 days
Nitrite-N	Cool, 4°C	P or G	48 hours
Ortho-Phosphate	Filter immediately, cool, 4°C	P or G	48 hours
pH	None	P or G	Analyze immediately ⁶
Selenium	Conc. HNO ₃ to pH<2	P or G	6 months
Silica	Cool, 4°C	P	28 days
Sodium	Conc. HNO ₃ to pH<2	P or G	6 months
Temperature	None	P or G	Analyze immediately ⁶

TABLE 3 (CONTINUED)

<u>Contaminant</u>	<u>Preservative</u> ²	<u>Container</u> ³	<u>Maximum Holding Time</u> ⁴
Silver	Conc. HNO ₃ to pH<2	P or G	6 months
Thallium	Conc. HNO ₃ to pH<2	P or G	6 months
Total Filterable Residue (TDS)	Cool, 4°C	P or G	7 days
Turbidity	Cool, 4°C	P or G	48 hours

NOTES:

¹ The laboratory director must reject any samples taken for compliance purposes not meeting these criteria and notify the authority requesting these analyses.

² When indicated, samples must be acidified at the time of collection to pH<2 with concentrated acid or adjusted with sodium hydroxide to pH>12. When chilling is indicated the sample must be shipped and stored at 4°C or less.

³ P = plastic, hard or soft; G = glass, hard or soft.

⁴ In all cases, samples should be analyzed as soon after collection as possible. Follow additional (if any) information on preservation, containers or holding times that is specified in the method.

⁵ These samples should never be frozen. Instructions for containers, preservation procedures and holding times specified in Method 100.2 must be adhered to for compliance analyses including those conducted with Method 100.1.

⁶ "Analyze immediately" generally means within 15 minutes of sample collection.

⁷ If the sample is chlorinated, the holding time for unacidified sample kept at 4°C is extended to 14 days.

TABLE 4

SAMPLE COLLECTION, CONTAINERS, AND PRESERVATION FOR ORGANIC CONTAMINANTS

<u>Contaminant</u>	<u>Method</u>	<u>Preservative</u>	<u>Container</u>	<u>Holding Time to Extraction</u>	<u>Holding Time After Extraction</u>
Non-volatile SOC	504.1	Sodium thiosulfate, cool, 4°C	Glass Teflon lined septum	14 days	4°C, 24 hours
	505	Sodium thiosulfate, cool, 4°C	Glass Teflon lined septum	14 days*	4°C, 24 hours
	506	Sodium thiosulfate, cool, 4°C, dark	Glass (amber) Teflon cap liners	14 days	4°C, dark, 14 days
	507	Sodium thiosulfate, cool, 4°C, dark	Glass (amber) Teflon cap liners	14 days***	4°C, dark, 14 days
	508	Sodium thiosulfate, cool, 4°C, dark	Glass Teflon cap liners	7 days***	4°C, dark, 14 days
	508A	Cool, 4°C	Glass Teflon cap liners	14 days	30 days
	508.1	Sodium sulfite, HCl to pH<2, cool, 4°C	Glass Teflon cap liners	14 days***	30 days
	515.1	Sodium thiosulfate, cool, 4°C, dark	Glass (amber) Teflon cap liners	14 days	4°C, dark, 28 days
	515.2	Sodium thiosulfate, HCl to pH<2, cool, 4°C, dark	Glass (amber) Teflon cap liners	14 days	≤4°C, dark, 14 days
	515.3	Sodium thiosulfate, cool, 4°C, dark	Glass (amber) Teflon cap liners	14 days	14 days
	515.4	Sodium sulfite, dark, cool ≤10°C for first 48 hrs., ≤6°C thereafter	Glass (amber) Teflon cap liners	14 days	≤0°C 21 days
	525.2	Sodium sulfite, dark, HCl to pH<2, cool, 4°C	Glass (amber) Teflon cap liners	14 days***	30 days
	531.1	Sodium thiosulfate, monochloroacetic acid to pH<3, cool, 4°C	Glass Teflon cap liners	28 days	No extract

TABLE 4 (CONTINUED)

<u>Contaminant</u>	<u>Method</u>	<u>Preservative</u>	<u>Container</u>	<u>Holding Time to Extraction</u>	<u>Holding Time After Extraction</u>
	531.2	Sodium thiosulfate, potassium dihydrogen citrate buffer to pH 4, dark, $\leq 10^{\circ}\text{C}$ for first 48 hrs, $\leq 6^{\circ}\text{C}$ thereafter	Glass Teflon cap liners	28 days	No extract
	547	Sodium thiosulfate, cool, 4°C	Glass (amber) Teflon cap liners	14 days (18 months frozen)	No extract
	548.1	Sodium thiosulfate, (HCl pH 1.5-2 if high biological activity), cool, dark, 4°C	Glass (dark) Teflon cap liners	7 days	14 days, $\leq 4^{\circ}\text{C}$
	549.2	Sodium thiosulfate, cool, 4°C , dark	High density PVC or silanized amber glass	7 days	21 days
	550	Sodium thiosulfate, 6N HCl to pH<2, cool, 4°C	Glass (amber) Teflon cap liners	7 days	30 days
	550.1	Sodium thiosulfate, 6N HCl to pH<2, cool, 4°C	Glass (amber) Teflon cap liners	7 days	40 days, dark, 4°C
	551.1	Ammonium chloride, sodium sulfite, cool, 4°C	Glass	4°C , 14 days	$< -10^{\circ}\text{C}$, 14 days
	552.1	Ammonium chloride, cool, 4°C	Glass (amber) Teflon cap liners	28 days	48 hours
	552.2	Ammonium chloride, cool, 4°C	Glass (amber) Teflon cap liners	14 days	$\leq 4^{\circ}\text{C}$ 7 days $\leq 10^{\circ}\text{C}$ 14 days
	555	Sodium sulfite, HCl to pH<2, dark, cool, 4°C	Glass Teflon cap liners	14 days	No extract
	1613B	80 mg/L sodium thiosulfate, cool, 4°C	Glass (amber) Teflon cap liners	---	40 days
	6610	Sodium thiosulfate, monochloroacetic acid to pH<3, cool, 4°C	Glass Teflon cap liners	28 days	No extract

TABLE 4 (CONTINUED)

<u>Contaminant</u>	<u>Method</u>	<u>Preservative</u>	<u>Container</u>	<u>Holding Time to Extraction</u>	<u>Holding Time After Extraction</u>
	6651	Sodium thiosulfate	Plastic Teflon cap liners	Two weeks, 4°C	No extract
	D5317-93	Sodium thiosulfate, cool, 4°C, dark	Glass (amber) Teflon cap liners	14 days	4°C, dark, 28 days
TTHM	502.2	Sodium thiosulfate or ascorbic acid, 1:1 HCl to pH<2, cool, 4°C	Glass Teflon lined septum	14 days	No extract
	524.2	Ascorbic acid, 1:1 HCl to pH<2, cool, 4°C	Glass Teflon lined septum	14 days	No extract
	551.1	Ammonium chloride, sodium sulfite, cool, 4°C	Glass	4°C, 14 days	No extract
VOC**	502.2	Sodium thiosulfate or ascorbic acid, 1:1 HCl to pH<2, cool, 4°C	Glass Teflon lined septum	14 days	No extract
	524.2	Ascorbic acid, 1:1 HCl to pH<2, cool, 4°C	Glass Teflon lined septum	14 days	No extract
	551.1	Ammonium chloride, sodium sulfite, cool, 4°C	Glass	4°C, 14 days	No extract

NOTES:

* The holding time for heptachlor under this method is 7 days.

** At the time of collection, VOC samples must be tested for the presence of chlorine residual and dechlorination must be done by the addition of ascorbic acid.

*** See method for exceptions.

TABLE 5

RECOMMENDED METHODS FOR SECONDARY CONTAMINANTS⁶

<u>Contaminant</u>	<u>EPA</u>	<u>ASTM</u> ³	<u>SM</u> ⁴	<u>SM Online</u> ⁸	<u>Other</u>
Aluminum	200.7 ²		3120B	3120B-99	
	200.8 ²		3113B	3113B-99	
	200.9 ²		3111D	3111D-99	
	200.5 ⁹				
Chloride	300.0 ¹	D-4327-97, 03	4110B	4110B-00	
			4500-CI D	4500-CI D-97	
		D512-89 (99)BB	4500-CI B		
Color			2120B	2120B-01	D6508, Rev. 2 ⁷
Foaming Agents			5540C	5540C-00	
Iron	200.7 ²		3120B	3120B-99	
	200.9 ²		3111B	3111B-99	
			3113B	3113B-99	
Manganese	200.5 ⁹				
	200.7 ²		3120B	3120B-99	
	200.8 ²		3113B	3113B-99	
	200.9 ²		3111B	3111B-99	
	200.5 ⁹				
Odor			2150B	2150B-97	
Silver	200.7 ²		3120B	3120B-99	I-3720-85 ⁵
	200.8 ²		3113B	3113B-99	
	200.9 ²		3111B	3111B-99	
	200.5 ⁹				
Sulfate	300.0 ¹	D4327-97, 03	4110B	4110B-00	
	375.2		4500-SO ₄ F		
			4500-SO ₄ C,D		
		D516-90, 02	4500-SO ₄ E		
TDS			2540C	2540C-97	D6508, Rev. 2 ⁷
Zinc	200.7 ²		3120B	3120B-99	
	200.8 ²		3111B	3111B-99	

NOTES:

¹ “Methods for the Determination of Inorganic Substances in Environmental Samples”, EPA-600/R-93-100, August 1993. Available at NTIS, PB94-121811.

² “Methods for the Determination of Metals in Environmental Samples Supplement I”, EPA-900/R-94-111, May 1994. Available at NTIS, PB95-125472.

³ The procedures shall be accordance with the *Annual Book of ASTM Standards*, 1994, 1996 and 1999, Vols. 11.01 and 11.02, American Society for Testing and Materials; any year containing the cited version of the method may be used. The previous versions of D1688-95A, D1668-95C (copper), D3559-95D (lead), D1293-95 (pH), D1125-91A (conductivity) and D859-94 (silica) are also approved. These previous versions of D1668-90A, D3559-90D, D1293-84, D1125-91A AND D859-88 respectively are located in the *Annual Book of ATSM Standards, 1994, Vols. 11.01*. Copies may be obtained from the American Society of Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Copies may be inspected at EPA’s Drinking Water Docket, 401 M Street SW, Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street NW, Suite 700, Washington, DC.

⁴ 18th, 19th, 20th and 21st editions and the Supplement to the 19th edition of *Standard Methods for the Examination of Water and Wastewater*, 1992, 1995 and 1998, respectively, American Public Health Association, any version may be used, except that the versions of 3111B, 3111D, 3113B and 3114B in the 20th edition may not be used. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 USC 552(a) and 1 CFR Part 51. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005. Copies may be inspected at EPA’s Drinking Water Docket, 401 M Street SW, Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street NW, Suite 700, Washington, DC.

⁵ Method I-3720-85, *Techniques of Water Resource Investigation of the U.S. Geological Survey*, Book 5, Chapter A-1, 3rd ed., 1989. Available from Information Services, U.S. Geological Survey, Federal Center, Box 25286, Denver, CO 80225-0425.

⁶ Criteria for analyzing aluminum, iron, manganese, silver and zinc with digestion and directly without digestion, and other analytical test procedures are contained in *Technical Notes on Drinking Water Methods*, EPA-600/R-94-173, October 1994, which is available at NTIS, PB95-104766.

⁷ Method D6508, Rev. 2, “Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte”, available from Waters Corp., 34 Maple Street, Milford, MA 01757, Telephone 508/482-2131, Fax: 805/482-3625.

⁸ Standard Methods Online available at <http://www.standardmethods.org>. The year in which each method was approved by the Standard Methods Committee is designated by the last two digits in the method number. The methods listed are the only online methods that may be used.

⁹ EPA Method 200.5, Revision 4.2, “Determination of Trace Elements in Drinking Water by Axially Viewed Inductively Coupled Plasma-Atomic Emission Spectrometry”, 2003. EPA/600/R-06/115. (Available at <http://www.epa.gov/nerlcwww/ordmeth/htm>)

Appendix A

Chain-of-Custody Evaluations

A. Introduction

Written procedures for sample handling should be available and followed whenever samples are collected, transferred, stored, analyzed or destroyed. For the purposes of litigation, it is necessary to have an accurate written record to trace the possession and handling of samples from collection through reporting. The procedures defined here represent a means to satisfy this requirement.

A sample is in someone's "custody" if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is one's physical possession and then locked up so that no one can tamper with it;
4. It is kept in a secured area, restricted to authorized personnel only.

B. Sample Collection, Handling and Identification

1. It is important that a minimum number of persons be involved in sample collection and handling. Guidelines established in standard manuals for sample collection preservation and handling should be used (e.g., EPA NPDES Compliance Sampling Inspection Manual, MCD 51, *Standard Methods for Examination of Water and Wastewater*). Field records should be completed at the time the sample is collected and should be signed or initialed, including the date and time, by the sample collector(s). Field records should contain the following information:
 - a. Unique sample or log number;
 - b. Date and time;
 - c. Source of sample (including name, location and sample type);
 - d. Preservative used;
 - e. Analyses required;
 - f. Name of collector(s);
 - g. Pertinent field data (pH, DO, Cl₂ residual, etc.);
 - h. Serial number on seals and transportation cases;
 - i. Comments.
2. Each sample is identified by affixing a pressure sensitive gummed label or standardized tag on the container(s). This label should contain the sample number, source of sample, preservative used, and the collector(s') initials. The analysis required should be identified. Where a label is

not available, the sample information should be written on the sample container with an indelible marking pen.

3. The closed sample container should then be placed in a transportation case along with the chain-of-custody record form, pertinent field records, and analysis request form. The transportation case should then be sealed and labeled. All records should be filled out legibly in waterproof pen. The use of locked or sealed chests will eliminate the need for close control of individual sample containers. However, there will undoubtedly be occasions when the use of a chest will be inconvenient. On these occasions, the sampler should place a seal around the cap of the individual sample container which would indicate tampering if removed.

C. Transfer of Custody and Shipment

1. When transferring the possession of the samples, the transferee must sign and record the date and time on the chain-of-custody record. Custody transfers, if made to a sample custodian in the field, should account for each individual sample, although samples may be transferred as a group. Every person who takes custody must fill in the appropriate section of the chain-of-custody record.
2. The field custodian (or field sampler if a custodian has not been assigned) is responsible for properly packaging and dispatching samples to the appropriate laboratory for analysis. This responsibility includes filling out, dating, and signing the appropriate portion of the chain-of-custody record. A recommended chain-of-custody format is illustrated in Figure A-2.
3. All packages sent to the laboratory should be accompanied by the chain-of-custody record and other pertinent forms. A copy of these forms should be retained by the field custodian (either carbon or photocopy).
4. Mailed packages can be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent chain-of-custody documentation.
5. Samples to be transported must be packed to prevent breakage. If samples are shipped by mail or by other common carrier, the shipper must comply with any applicable Department of Transportation regulations. (Most water samples are exempt unless quantities of preservatives used are greater than certain levels.) The package must be sealed or locked to prevent tampering. Any evidence of tampering should be readily detected if adequate sealing devices are used.
6. If the field sampler delivers samples to the laboratory, custody may be relinquished to laboratory personnel. If appropriate personnel are not present to receive the samples, they should be locked in a designated area of the laboratory to prevent tampering. The person delivering the samples should make a log entry stating where and how the samples were delivered and secured. Laboratory personnel may then receive custody by noting in a logbook, the absence of evidence of tampering, unlocking the secured area, and signing the custody sheet.

D. Laboratory Sample Control Procedures

Sample control procedures are necessary in the laboratory from the time of sample receipt to the time the sample is discarded. The following procedures are recommended for the laboratory:

1. A specific person must be designated as custodian and an alternate designated to act as custodian in the custodian's absence. All incoming samples must be received by the custodian, who must indicate receipt by signing the accompanying custody/control forms and who must retain the signed forms as permanent records.
2. The custodian must maintain a permanent logbook to record, for each sample, the person delivering the sample, the person receiving the sample, date and time received, source of sample, date the sample was taken, sample identification log number, how transmitted to the laboratory, and condition received (sealed, unsealed, broken container, or other pertinent remarks). This log should also show the movement of each sample within the laboratory; i.e., who removed the sample from the custody area, when it was removed, when it was returned, and when it was destroyed. A standardized format should be established for logbook entries.
3. A clean, dry, isolated room, building, and/or refrigerated space that can be securely locked from the outside must be designated as a "custody room."
4. The custodian must ensure that heat-sensitive samples, light-sensitive samples, radioactive samples, or other sample materials having unusual physical characteristics, or requiring special handling, are properly stored and maintained prior to analysis.
5. Distribution of samples to the analyst performing the analysis must be made by the custodian.
6. The laboratory area must be maintained as a secured area, restricted to authorized personnel only.
7. Laboratory personnel are responsible for the care and custody of the sample once it is received by them and must be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses are completed.
8. Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample must be retained in the custody room until permission to destroy the sample is received by the custodian.
9. Samples will be destroyed only upon the order of the responsible laboratory official when it is certain that the information is no longer required or the samples have deteriorated. (For example, standard procedures should include discarding samples after the maximum holding time has elapsed.) The same procedure is true for sample tags. The logbook should show when each sample was discarded or if any sample tag was destroyed.
10. Procedures should be established for internal audits of sample control information. Records should be examined to determine traceability, completeness, and accuracy.