

PARAMETER HANDBOOK

FOR THE

NATIONWIDE URBAN RUNOFF PROGRAM



October 1979

Water Planning Division

U.S. Environmental Protection Agency

Washington, D.C. 20460

PARAMETER HANDBOOK

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AREAWIDE ASSESSMENT PROCEDURES MANUAL

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for the

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APPENDIX D, PART II

PARAMETER HANDBOOK

This Parameter Handbook has been written as a part of the Areawide Assessment Procedures Manual to aid 208 planning agencies in the establishment and conduct of water quality monitoring programs. The material presented summarizes existing work rather than representing new research results. The intent is to present, on one sheet of paper, enough salient information about a particular water quality parameter to allow decisions to be made as to the likelihood of the constituent's presence in a particular stream or discharge, its effects upon water quality or use, and factors pertaining to sampling and analysis of the constituent that should be considered in determining the ramifications of including the parameter in a water quality monitoring program. The information presented on analytical methodology, including sample quantity and preservation and handling considerations, was largely taken from one of three widely available sources:

- Methods for Chemical Analysis of Water and Wastes, 1974 (commonly called "EPA Methods Manual"). Available from USEPA Environmental Research Information Center, Cincinnati, OH 45268.
- Standard Methods for the Examination of Water and Waste Water, 14th Edition, 1976 (commonly called "Standard Methods"). Available from the American Public Health Association, 1015 18th Street, N.W., Washington, D.C. 20036.
- Annual Book of Standards, Part 31, Water, 1975 (commonly called "ASTM Methods"). Available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

The information presented herein was taken in the order indicated above, i.e., if the parameter is covered in the EPA Methods Manual, that reference was used as the primary source; if the parameter is not in the EPA Methods Manual but is covered by Standard Methods, the latter was used as the primary source; and so on. For parameters not treated by any of the above sources, other publications, especially those of the U.S. Geological Survey, were used.

It must be emphasized that this Parameter Handbook is not a specification; the information presented herein is illustrative, not exhaustive, and carries no legal authority. In this latter

regard, the USEPA has published, pursuant to section 304(g) of the Water Pollution Control Act Amendments (PL 92-500), "Guidelines Establishing Test Procedures for the Analysis of Pollutants" in the Wednesday, December 1, 1976 issue of the Federal Register (40 CFR 136), a copy of which is attached at the end of this Parameter Handbook. Some minor corrections to these Guidelines were published in the Federal Register on Tuesday, January 18, 1977.

The synoptic presentations in this Parameter Handbook are written for a reader without extensive training or experience in water and wastewater analysis. A common format, depicted in Figure 1, has been used for each parameter. The entries will be discussed in turn.

Parameter Name: This is the most common name by which the parameter is most frequently known, not necessarily its proper chemical name. Where other names are commonly used or the chemical formula might be helpful, they are indicated in the general discussion.

A number of parameters are part of the USEPA water quality data storage and retrieval system (STORET) at the present time, and more will be added in the future. Where a parameter is a part of the STORET System as of March 1977, the following two entries are filled in; otherwise they are left blank.

Parameter Group: Each parameter is assigned to a designated group (e.g., metals, general organic, pesticides) in the STORET System, and this entry indicates the group to which the parameter belongs.

STORET Units: The units that must be used for entry of the concentration of the parameter into the STORET System are given here; e.g., micrograms per liter ($\mu\text{g}/\ell$).

General: This is a brief summary of salient parameter characteristics. Typically covered are such things as what the parameter is; any common alternate name or chemical formula where possibly helpful; natural sources; uses of the substance and possible sources related thereto; indications of the persistence of the parameter in water, including its solubility where appropriate; effects of the parameter on water use, including toxicity data where appropriate; and, since many of these parameters are actually toxic substances or surrogate measures for toxic substances, the level of regulation that has been imposed upon them (such as the two technologically feasible control oriented standards of "best practicable control technology currently available" (BPT) or "best available control technology economically achievable" (BAT) or "toxicity effluent limitations") has been noted in accordance with the regulation (40 CFR 136), BAT parameters under the Consent Decree, or toxicity guidelines under the Consent Decree or other regulatory mandates.

PARAMETER NAME	
<u>Parameter Group:</u>	<u>STORET Units:</u>
<u>General:</u>	
<u>Criterion:</u>	
<u>Preservation Method:</u>	
<u>Maximum Holding Time:</u>	
<u>Container Type:</u>	
<u>Sample Volume Required:</u>	
<u>Measurement:</u>	
<u>Precision and Accuracy:</u>	
<u>Cost of Analysis:</u>	

Figure 1. Format for Parameter Information

Criteria: If the USEPA has issued water quality criteria for the parameter, they are given here along with the beneficial use to be protected by the criterion established.

Preservation Method: Physical and chemical preservatives to be used to help maintain sample integrity are indicated here along with any special sample handling considerations, e.g., keep sealed until analyzed.

Maximum Holding Time: The maximum holding time between gathering and preserving the sample and its analysis in the laboratory is given here. It is a function not only of the physical and chemical characteristics of the substance involved but also of the other constituents in the sample. The holding times given are conservative in some instances and, if data indicate that longer holding times do not significantly affect analytical results, they may be used.

Container Type: Acceptable sample container materials are indicated here. Although not addressed here, sample equipment cleaning is very important, and special cleaning protocols will be required for some parameters, e.g., pesticides, as will other special considerations such as the use of TFE fluorocarbon cap liners, etc.

Sample Volume Required: An estimate of the quantity of sample necessary to allow analysis for the parameter is given here. No allowance for replication, sample splitting, spillage, etc., is made. The exact sample quantity required will depend upon the strength of the constituent, the need for concentration or dilution, removal of interferences, etc., and is best established after preliminary laboratory work, but the given volumes can be used as a first cut.

Measurement: Descriptions of common methods for making the determination are given here, primarily to indicate any special laboratory equipment that might be required (e.g., AA, GC). Applicable concentration ranges are given, and possible interferences and precautions are indicated in many cases. Where a measurement is mandated by regulation, the regulation is cited.

Precision and Accuracy: Method sensitivity and detection limits are provided where generally agreed to. Precision and accuracy data are given where known.

Cost of Analysis: The information provided here is intended to give an appreciation of the relative magnitude of cost for performing the analysis. The data are typically presented as a range that represents differences in cost among laboratories and (sometimes) methods for a given parameter. Cost information was obtained from various laboratories across the United States and

representative values were selected for the range. Extremely low or high costs for a particular parameter from a given laboratory were discarded as atypical. Costs are also influenced by sample preparation procedures necessary to remove interferences. This is especially true for pesticide analyses and is represented by large ranges in many instances, with the lower end of the range being more typical in most cases. Finally, although not indicated in the cost data presented herein, many laboratories offer quantity discounts that may be quite substantial, and local laboratories should be consulted if firm budget numbers are desired.

There are many water quality parameters that might be of interest to some 208 agencies. Those that were selected for inclusion in this first edition of the Parameter Handbook are the ones that were considered to have the broadest appeal. They represent a compilation of those found in the newly issued EPA Water Quality Criteria, the EPA Methods Manual, and the majority of the substances listed in the Consent Decree. It is contemplated that the parameter coverage will be increased in future editions of this handbook. For many parameters, preferred analytical methods, preservation techniques, maximum holding times, etc., have not been established or are tentative. Considerable advancement is expected in the near term time frame, and future editions will be updated to disseminate this information.

To assist the reader in locating parameters of interest, a number of tables are provided. In Table 1, all the parameters in the handbook are listed in alphabetical order. The parameters are alphabetically listed within each STORET parameter group in Table 2. Those parameters for which the USEPA has issued water quality criteria are alphabetically listed in Table 3. Finally, those parameters designated by the Consent Decree are listed alphabetically in Table 4. The parameter sheets in the handbook are in alphabetical order. A copy of the 1 December 1976 Federal Register is attached at the end.

TABLE 1. ALPHABETICAL LISTING OF PARAMETERS

Acidity	2, 4-D
Acrolein	DD
Acrylonitrile	DDE
Aldrin	DDT
Alkalinity	Demeton
Aluminum	Diazinon
Antimony	Dichlorobenzenes
Arsenic	Dichlorobenzidine
Asbestos	Dichloroethylenes
Atrazine	2, 4-Dichlorophenol
	Dichloropropane
Barium	Dichloropropene
* Benzene	Dieldrin
Benzene Hexachloride (BHC)	2, 4-Dimethylphenol
Benzidine	Dissolved Oxygen
Beryllium	Disyston
Biochemical Oxygen Demand (BOD)	Diuron
Boron	
Bromide	Endosulfan
	Endrin
Cadmium	* Ethylbenzene
Calcium	
Captan	Fecal Coliform
Carbaryl	Fecal Streptococci
Carbon Tetrachloride	Fluoride
Chemical Oxygen Demand	
Chlordane	Guthion
Chloride	
Chlorinated Benzenes	Haloethers
Chlorinated Ethanes	* Halomethanes
Chlorinated Naphthalene	Hardness, Total
Chlorinated Phenols (Other)	Heptachlor
Chlorine Demand	
Chlorine Dioxide	Iodine
Chlorine, Residual	Iron
Chloroalkyl Ethers	
* Chloroform	Lead
2-Chlorophenol	Lindane
Chromium	Lithium
Cobalt	
Color	Magnesium
Copper	Malathion
Cyanide	Manganese

TABLE 1. ALPHABETICAL LISTING OF PARAMETERS (Cont'd)

** methylene chloride*

Mercury	Residue, Total
Methane	Residue, Total Filterable
Methoxychlor	Residue, Total Nonfilterable
Methyl Parathion	Residue, Volatile
Methylene Blue Active Substances (MBAS)	Selenium
Mirex	Silica
Molybdenum	Silicon
	Silver
Naphthalene	Silvex (2, 4, 5-TP)
Nickel	Sodium
Nitrilotriacetic Acid (NTA)	Specific Conductance
Nitrobenzene	Strontium
Nitrogen-Ammonia	Sulfate
Nitrogen, Kjeldahl	Sulfide
Nitrogen, Nitrate	Sulfite
Nitrogen, Nitrate-Nitrite	
Nitrogen, Nitrite	2, 4, 5-T
Nitrophenols	Temperature
	Thallium
Oil and Grease	Threshold Odor
Organic Carbon	Tin
	Titanium
Parathion	*Toluene
PCNB	Total Coliform
Pentachlorophenol	Toxaphene
pH	Trichloroethylene
Phenolics	Turbidity
Phosphorous (all forms)	
Phthalate Esters	Uranium
Polychlorinated Biphenyls	
Polynuclear Aromatic Hydrocarbons	Vandium
Potassium	Vinyl Chloride
Radioactivity (alpha and beta)	Xylene
Radium	
Residue, Settleable	Zinc

** tetrachloroethylene*

TABLE 2. LISTING OF PARAMETERS ACCORDING TO
STORET GROUP

<u>Bacteriologic</u>	<u>Metals</u>
Fecal Coliform	Aluminum
Fecal Streptococci	Antimony
Total Coliform	Arsenic
	Barium
	Beryllium
	Boron
<u>Dissolved Oxygen</u>	Cadmium
Dissolved Oxygen	Calcium
	Chromium
	Cobalt
<u>General Organic</u>	Copper
Benzene	Iron
Methylene Blue Active	Lead
Substances (MBAS)	Lithium
Nitrilotriacetic Acid (NTA)	Magnesium
Oil and Grease	Manganese
Organic Carbon	Mercury
Phenolics	Molybdenum
Phthalate Esters	Nickel
Polychlorinated Biphenyls	Potassium
Toluene	Selenium
Xylene	Silver
	Sodium
	Thallium
	Tin
<u>General Inorganic</u>	Titanium
Acidity	Uranium
Alkalinity	Vanadium
Asbestos	Zinc
Bromide	
Chloride	<u>Miscellaneous</u>
Chlorine Demand	Chlorine, Residual
Cyanide	
Fluoride	
Hardness, Total	
Iodide	
Sulfate	
Sulfide	
Sulfite	

TABLE 2. LISTING OF PARAMETERS ACCORDING TO
STORET GROUP (Cont'd)

Nitrogen

Nitrogen-Ammonia
Nitrogen, Kjeldahl
Nitrogen, Nitrate
Nitrogen, Nitrate-Nitrite
Nitrogen, Nitrite

Mirex
Parathion
Pentachlorophenol
Silvex (2, 4, 5-TP)
2, 4, 5-T
Toxaphene

Oxygen Demand

Biochemical Oxygen Demand
(BOD)
Chemical Oxygen Demand

Phosphorous

Phosphorus (all forms)

Pesticides

Aldrin
Atrazine
Benzene Hexachloride (BHC)
Benzidine
Captan
Carbaryl
Carbon Tetrachloride
Chlordane
Chloroform
2, 4-D
DDD
DDE
DDT
Demeton
Diazinon
Dieldrin
Disyston
Diuron
Endosulfan
Endrin
Guthion
Heptachlor
Lindane
Malathion
Methoxychlor
Methyl Parathion

Physical

Color
PH
Specific Conductance
Threshold Odor
Turbidity

Radiological

Radioactivity (alpha and beta)
Strontium

Solids

Residue, Settleable
Residue, Total
Residue, Total Filterable
Residue, Total Nonfilterable
Residue, Volatile
Silica
Silicon

Temperature

Temperature

TABLE 3. PARAMETERS FOR WHICH THE USEPA
HAS ISSUED WATER QUALITY CRITERIA

Aldrin	Lead
Alkalinity	Lindane
Arsenic	Malathion
Barium	Manganese
Beryllium	Mercury
Boron	Methoxychlor
Cadmium	Mirex
Chlordane	Nickel
Chlorine, Residual	Oil and Grease
Chromium	Parathion
Color	pH
Copper	Phenolics
Cyanide	Phosphorus (all forms)
2,4-D	Phthalate Esters
DDT	Polychlorinated Biphenyls
Demeton	Residue, Total Filterable
Dieldrin	Residue, Total Nonfilterable
Dissolved Oxygen	Selenium
Endosulfan	Silver
Endrin	Silvex
Guthion	Temperature
Heptachlor	Toxaphene
Iron	Zinc

TABLE 4. CHEMICAL CLASSES AND COMPOUNDS DESIGNATED
AS PRIORITY POLLUTANTS IN THE TOXICS SETTLEMENT AGREEMENT

Acenaphthene	2, 4-Dimethylphenol
Acrolein	2, 4-Dinitrophenol
Acrylonitrile	Dinitrotoluene
Aldrin	1, 2-Diphenylhydrazine
Antimony (total)	Endosulfan and Metabolites
Arsenic (total)	Endrin and Metabolites
Asbestos	Ethylbenzene
Benzene	Fluoroanthene
Benzidine	Haloethers (other than those listed elsewhere)
Beryllium (total)	Halomethanes (other than those listed elsewhere)
Cadmium (total)	Heptachlor and Metabolites
Carbon Tetrachloride (tetrachloromethane)	Hexachlorobutadiene
Chlordane (technical mixture and metabolites)	Hexachlorocyclohexane (all isomers)
Chlorinated Benzenes (other than dichlorobenzenes)	Hexachlorocyclopentadiene
Chlorinated Ethanes (including 1, 2-trichloroethane and hexachloroethane)	Isophorone
Chlorinated Naphthalene	Lead (total)
Chlorinated Phenols, (other than those listed else- where; includes trichloro- phenols and chlorinated cresols)	Mercury
Chloroalkyl Ethers (chloro- methyl, chloroethyl, and mixed ethers)	Naphthalene
Chloroform (trichloromethane)	Nickel (total)
2-Chlorophenol	Nitrobenzene
Chromium (total)	Nitrophenols (including 2, 4-dinitrophenol and dinitro- cresol)
Copper (total)	Nitrosamines
Cyanide (total)	Pentachlorophenol
DDT and Metabolites	Phenol
Dichlorobenzenes	Phthalate Esters
Dichlorobenzidine	Polychlorinated Biphenyls (PCB's)
Dichloroethylenes (1, 1-dichloroethylene and 1, 2-dichloroethylene)	Polynuclear Aromatic Hydrocarbons
2, 4-Dichlorophenol	Selenium (total)
Dichloropropane and Dichloro- propene	Silver (total)
	2, 3, 7, 8-Tetrachlorodibenzo- P-Dioxin (TCDD)
	Tetrachloroethylene
	Thallium (total)
	Toluene
	Toxaphene
	Trichloroethylene
	Vinyl Chloride (chloroethylene)
	Zinc (total)

ACIDITY

Parameter Group: General
Inorganic

STORET Units: mg/ℓ as CaCO₃

General: Acidity is a measure of a gross property of water, its quantitative ability to neutralize a strong base to a designated pH. It can be interpreted in terms of specific substances only when the chemical composition of the sample is known. Acids contribute to corrosiveness and influence certain chemical and biological processes; therefore, the acidity of water is important. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as practicable. Fill sample bottles completely and cap tightly. The sample should not be agitated or exposed to air for a prolonged period of time. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The pH of the sample is determined and a measured amount of standard acid is added, as needed, to lower the pH to 4 or less. Hydrogen peroxide is added, the solution boiled for several minutes, cooled, and titrated electrometrically with standard alkali to pH 8.2. Suspended matter present in the sample, or precipitates formed during the titration may cause a sluggish electrode response. This may be offset by allowing a 15-20 second pause between additions of titrant or by slow dropping addition of titrant as the endpoint pH is approached. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: On a round robin conducted by ASTM on 4 acid mine waters, including concentrations up to 2,000 mg/ℓ, the precision was found to be ±10 mg/ℓ.

Cost of Analysis: \$4 - \$5

ACROLEIN

Parameter Group:

STORET Units:

General: Acrolein (also known as acrylic aldehyde or 2-propenal) is a clear, colorless liquid at ordinary temperatures with a pungent irritating odor. It is extremely irritating to the skin and mucous membranes and is readily soluble in water. Its main use is as an aquatic weed killer. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: No standard procedures for acrolein have been developed. It may require special treatment to extract from water prior to gas chromatographic analysis. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Precision and accuracy data are not available.

Cost of Analysis: Expensive; must be quoted based on sample composition.

ACRYLONITRILE

Parameter Group:

STORET Units:

General: Acrylonitrile is a flammable liquid used in the manufacture of synthetic rubber and plastics and as a pesticide fumigant for stored grain. It is moderately soluble in water and does not disassociate markedly. Upon disassociation it can form HCN, the toxic cyanide principle. Concentrations of 20 mg/l are deleterious for many fish. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: No preferred method has been established. Acrylonitrile has been determined in wastewater by azeotropic distillation with methanol followed by measurement of NH_3 liberated by alkali saponification, but the method may not be practicable for many wastewaters. Detection limits are around 2,000 $\mu\text{g/l}$. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Precision and accuracy data are not available.

Cost of Analysis: Expensive; must be quoted based on sample composition.

ALDRIN

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Aldrin, the common name of an organochlorine insecticide, is metabolically converted to dieldrin by aquatic organisms. Because of this metabolic conversion and because of evidence that dieldrin is as toxic or slightly more toxic than aldrin to aquatic organisms, an acceptable water concentration is based on the presence of either aldrin or dieldrin or the sum of both. Aldrin is used agriculturally at rates varying from 2 oz to 6 lb per acre, usually as a dust or emulsifiable concentrate; it is virtually insoluble in water. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criteria

- 003 $\mu\text{g}/\ell$ for freshwater and marine aquatic life
- The persistence, bioaccumulation potential, and carcinogenicity of aldrin cautions human exposure to a minimum.

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for aldrin. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration. For example, at the 0.015 and 0.110 $\mu\text{g}/\ell$ concentrations, recoveries were around 69% and 72% and precisions were 47% and 41%, respectively.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

ALKALINITY

Parameter Group: General
Inorganic

STORET Units: mg/ℓ as CaCO₃

General: Alkalinity is a measure of a gross property of water, its quantitative ability to neutralize a strong acid to a designated pH. It can be interpreted in terms of specific substances only when the chemical composition of the sample is known. Alkalinity, therefore, is a measure of the buffering capacity of the water, and since pH has a direct effect on organisms as well as an indirect effect on the toxicity of certain other pollutants in the water, the buffering capacity is important to water quality. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 20 mg/ℓ or more as CaCO₃ for freshwater aquatic life except where natural concentrations are less.

Preservation Method: Analyze as soon as practicable. Fill sample bottles completely and cap tightly. The sample should not be agitated or exposed to air for a prolonged period of time. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or otherwise altered in any way. Substances such as salts of weak organic and inorganic acids present in large amounts may cause interference in the electrometric pH measurements. Oil and grease may interfere by coating the electrode, thereby causing sluggish response. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: No general statement can be made about precision due to the great variation in sample characteristics. Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate equivalent to around 9 and 116 mg/ℓ CaCO₃. The bias was approximately +16% and -8% and relative standard deviation was approximately 14% and 5%, respectively.

Cost of Analysis: \$4 - \$5

ALUMINUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Al

General: Aluminum, being the third most abundant element in the earth's crust, occurs in minerals, rocks, and clays. Aluminum is found as a soluble salt, a colloid, or an insoluble compound in natural waters. Aluminum in wastewaters occurs from primary aluminum production and from secondary aluminum processes such as ingot cooling and shot quenching, scrubbing of furnace fumes during demagging, and wet milling of residues. Washwater from water treatment plants is another likely source, as are discharges from dyeing and cloth printing operation, paper mills, disinfectant operation, tanneries, viscose rayon plants, and many other industrial operations. Very little ingested aluminum is absorbed in the alimentary canal, so its presence does not normally pose a public health problem. Conflicting literature abounds on crop effects. An average daily dose of 2 mg aluminum has not harmed rats. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 309.2 nm. Aluminum is partially ionized in the nitrous oxide-acetylene flame. This problem may be controlled by the addition of an alkali metal (potassium, 1,000 $\mu\text{g}/\text{mL}$) to both sample and standard solutions. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 1,000 $\mu\text{g}/\ell$; its detection limit is 100 $\mu\text{g}/\ell$. The optimum concentration range is 5,000-100,000 $\mu\text{g}/\ell$. At a concentration of 300 $\mu\text{g}/\ell$, the relative standard deviation is 22.2%, and the relative error is 0.7%. Precision and accuracy decrease markedly for decreasing concentrations. For example, in an interlaboratory study on trace metals analysis, at true values of 35 and 15 $\mu\text{g}/\ell$, the relative standard deviations were 309% and 1,120%, respectively, while the relative errors were 175% and 627%, respectively.

Cost of Analysis: \$10 - \$20

ANTIMONY

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Sb

General: Natural antimony occurs chiefly as the sulfide or in oxide forms. Antimony is used in various industrial operations, especially in alloying as, for example, with lead for storage battery plates, with lead and tin in type metals, and with tin and copper as a bearing or antifriction material, and may be introduced into wastewaters from such sources, as well as the rubber, textile, explosives, paint, ceramic, and glass industries. Antimony has been reported to cause dermatitis and gastrointestinal disturbances in humans (it has long been used as an emetic) and has been found to shorten the life span of rats. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 217.6 nm. In the presence of lead (1,000 mg/L), a spectral interference may occur at the 217.6 nm resonance line. In this case, the 231.1 nm antimony line should be used. Increasing acid concentrations decrease antimony absorption. To avoid this effect, the acid concentration in the samples and in the standards should be matched. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 500 $\mu\text{g}/\ell$; its detection limit is 200 $\mu\text{g}/\ell$. The optimum concentration range is 1,000-40,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 5,000 μg and 15,000 μg Sb/L, the relative standard deviations were 1.6% and .66%, respectively. Recoveries at these levels were 96% and 97%, respectively.

Cost of Analysis: \$10 - \$20

ARSENIC

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as As

General: Mineral dissolution, industrial discharges, or the application of pesticides may lead to the occurrence of arsenic in water. Though most forms of arsenic are toxic to humans, arsenicals have been used in the medical treatment of spirochaetal infections, blood dyscrasias and dermatitis. Arsenic and arsenicals have many diversified industrial uses such as hardening of copper and lead alloys, tannery operations, pigmentation in paints and fireworks, and the manufacture of glass and ceramics, cloth, electrical semiconductors, and petroleum products. Arsenicals are used in the formulation of herbicides for forest management and agriculture. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 50 $\mu\text{g}/\ell$ for domestic water supplies (health)
- 100 $\mu\text{g}/\ell$ for irrigation of crops

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric gaseous hydride method is recommended for determining total arsenic, using a wavelength of 193.7 nm. The method is applicable to most fresh and saline waters in the absence of high concentrations of chromium, copper, cobalt, mercury, molybdenum, nickel, and silver. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is approximately 2.5 $\mu\text{g}/\ell$; its detection limit is 2.0 $\mu\text{g}/\ell$. The working range of the method is 2.0-20 $\mu\text{g}/\ell$. At a concentration of 10 $\mu\text{g}/\ell$, the relative standard deviation is 6% and the relative error is 1%. Ten replicate solutions of o-arsenilic acid at the 5, 10, and 20 $\mu\text{g}/\ell$ level were analyzed by a single laboratory. Relative standard deviations were 6%, 9%, and 5.5% with recoveries of 94, 93, and 85%, respectively.

Cost of Analysis: \$15 - \$20

ASBESTOS

<u>Parameter Group:</u>	General	<u>STORET Units:</u>	Count/liter
	Inorganic		with length/ width >1

General: Asbestos is primarily an air pollutant which has been shown to produce asbestosis, lung cancer, and mesothelioma in asbestos workers. However, the problems of asbestos in water have been recognized. It was discovered in 1973 that the drinking water of Duluth, Minnesota, and other cities on Lake Superior was heavily contaminated with asbestos. Sources of asbestos contamination include: asbestos mining, pulpmills, asbestos products, installation of asbestos construction material, spray-on steel fireproofing, and insulating cement application. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible

Maximum Holding Time: Unknown

Container Type: Glass or plastic

Sample Volume Required: Approximately 1,000 ml

Measurement: The present procedure is the microscopic counting of fibers in water. Asbestos probably cannot be routinely determined in effluents in the absence of gross contamination. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Precision and accuracy data are not available at this time.

Cost of Analysis: No standard pricing due to impracticability of analysis.

ATRAZINE

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Atrazine, 2-chloro-4-ethylamino-6-isopropylamino-S-triazine, is a triazine pesticide. It is used as a selective herbicide. It has an oral LD_{50} in rats of 3.08 g/kg. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 1,000 mL

Measurement: The recommended method covers the determination of various symmetrical triazine pesticides. It involves an efficient sample extraction procedure and provides, through use of column chromatography, a method for the elimination of non-pesticide interferences and the pre-separation of pesticide mixtures. Identification is made by selective gas chromatographic separation, and measurement is accomplished by the use of an electrolytic conductivity detector (CCD). Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The interferences in industrial effluents are high and varied. Nitrogen containing compounds other than the triazines may interfere. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Atrazine can be determined by this method with a sensitivity of 1 $\mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 to \$150, depending upon preparation required.

BARIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Ba

General: Barium compounds are used in a variety of industrial applications including the metallurgical, paint and dye, glass, ceramic, and electronics industries, as well as for medicinal purposes, the vulcanizing of rubber, and explosives manufacturing. Barium naturally occurs only in trace amounts in water. Therefore, appreciable amounts of barium indicates undesirable industrial discharges. A barium dose of 550,000 to 600,000 μg is considered fatal to human beings. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 1 mg/ℓ for domestic water supply (health)

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 553.6 nm. The use of a nitrous oxide-acetylene flame virtually eliminates chemical interference; however, barium is easily ionized in this flame and potassium must be added (1,000,000 $\mu\text{g}/\ell$) to standards and samples alike to control this effect. If the nitrous oxide flame is not available and acetylene-air is used, phosphate, silicon and aluminum will severely depress the barium absorbance. This may be overcome by the addition of 2,000,000 $\mu\text{g}/\ell$ lanthanum. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 400 $\mu\text{g}/\ell$; its detection limit is 30 $\mu\text{g}/\ell$. The optimum concentration range is 1000-20,000 $\mu\text{g}/\ell$. At a concentration of 500 $\mu\text{g}/\ell$, the relative standard deviation is 10%, and the relative error is 8.6%. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 400 and 2,000 $\mu\text{g Ba}/\ell$, the relative standard deviations were 10.8% and 6.5%, respectively. Recoveries at these levels were 94% and 113%, respectively.

Cost of Analysis: \$10 - \$15

BENZENE

Parameter Group: General
Organic

STORET Units: $\mu\text{g}/\ell$

General: Benzene (C_6H_6) is the simplest of the aromatic compounds and is used extensively as a commercial solvent and for the synthesis of other organic substances. At normal temperatures it is a volatile, flammable, colorless liquid with an ethereal odor. It is moderately soluble in water, $820 \text{ mg}/\ell$ at 22°C . It occurs in wastes from chemical plants, dyeing and other textile operations, and many other industrial processes. The oral LD_{50} for rats is around $5,600 \text{ mg}/\text{kg}$ of body weight. The toxicity of benzene toward fish has been reported from $5,000 \mu\text{g}/\ell$ up to $395,000 \mu\text{g}/\ell$ depending upon age and species. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: Hexadecane extraction followed by a gas chromatographic and mass spectrometric analysis is often used. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits should be around $2\text{-}10 \mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$15 - \$30

BENZENE HEXACHLORIDE (BHC)

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Benzene hexachloride (BHC), the common name of hexachlorocyclohexane, is an organochlorine pesticide. It has five known stereoisomers, the gamma isomer (lindane) being the most powerful insecticidal principle. BHC has a residual life in soil approaching that of DDT. Elevated concentrations of BHC reduce treatment plant efficiency, cause stream organisms to disappear, and produce disagreeable odors. It can impart a musty odor and taste to crops. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for BHC. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

BENZIDINE

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Benzidine (4, 4'-diaminobiphenyl, $\text{C}_{12}\text{H}_{12}\text{N}_2$) is a polynuclear organic pesticide. A crystalline substance, it is only slightly soluble in water. It possesses carcinogenic properties and must be handled with great care. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C .

Maximum Holding Time: 1 week

Container Type: Borosilicate glass

Sample Volume Required: 1,000-4,000 mL, depending on concentration and instrument used.

Measurement: Benzidines are separated and concentrated by multiple extractions and then oxidized by chloramine T. The oxidation product is extracted and measured spectrophotometrically. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is approximately $0.2 \mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$20 - \$40; because of its carcinogenic properties, special facilities may be required at greatly increased cost.

BERYLLIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Be

General: Beryllium is not likely to occur at significantly toxic levels in ambient natural waters. Beryllium could enter waters in effluents from certain metallurgical plants and discharges from industries dealing with atomic reactors, X-ray diffraction tubes, neon signs, aircraft and rockets, and missile fuel. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 11 $\mu\text{g}/\ell$ for the protection of aquatic life in soft fresh water
- 1,100 $\mu\text{g}/\ell$ for the protection of aquatic life in hard fresh water
- 100 $\mu\text{g}/\ell$ for continuous irrigation on all soils; except
- 500 $\mu\text{g}/\ell$ for irrigation on neutral to alkaline fine-textured soils

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric or aluminum colorimetric methods are suitable. The latter requires either a spectrophotometer for use at 515 nm or a filter photometer equipped with a green filter having maximum transmittance near 515 nm; either must provide a light path of 5 cm. Sodium and silicon at concentrations in excess of 1,000,000 $\mu\text{g}/\ell$ have been found to severely depress the beryllium absorbance. Bicarbonate ion is reported to interfere; however, its effect is eliminated when samples are acidified to a pH of 1.5. Aluminum at concentrations of >500 $\mu\text{g}/\ell$ is reported to depress the sensitivity of beryllium. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 25 $\mu\text{g}/\ell$; its detection limit is 5 $\mu\text{g}/\ell$. The optimum concentration range is 50-2,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 10, 50, and 250 $\mu\text{g}/\ell$, the relative standard deviations were 10%, 2%, and 1%, respectively. Recoveries at these levels were 100%, 98%, and 97%, respectively. In 32 laboratories using a known sample containing 250 $\mu\text{g}/\ell$, the beryllium was determined colorimetrically with a relative standard deviation of 7% and a relative error of 12%.

Cost of Analysis: \$10 - \$20

BIOCHEMICAL OXYGEN DEMAND (BOD)

Parameter Group: Oxygen Demand STORET Units: mg/ℓ

General: The biochemical oxygen demand (BOD) determination is an empirical test used to obtain a measure of the relative oxygen demand of water, especially treatment plant loadings and removal efficiencies. It is important to realize that BOD results cannot be compared unless the results have been obtained under identical test conditions and that the test is of limited value in determining the actual oxygen demand of surface waters. Complete stabilization of a given sample may require a period of incubation too long for practicable purposes, so the 5-day test is most commonly reported. As an indicator parameter, BOD is not a pollutant and exercises no direct harm. Its indirect effect is to depress dissolved oxygen levels. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 6 hours

Container Type: Plastic or glass

Sample Volume Required: 1000 ml

Measurement: The recommended method is an empirical bioassay type procedure which measures the dissolved oxygen consumed by microbial life while assimilating and oxidizing the organic matter present. The standard test conditions include dark incubation at 20°C for 5 days. The determination of dissolved oxygen may be made by use of either the modified Winkler or the electrode method. Many samples will require seeding due to low microbial populations. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Eighty-six analysts in fifty-eight laboratories analyzed natural water samples plus an exact increment of biodegradable organic compounds. At mean values of 2.1 and 175 mg/ℓ BOD, the standard deviations were ± 0.7 and ± 26 mg/ℓ, respectively. There is no acceptable procedure for determining the accuracy of the BOD test.

Cost of Analysis: \$10 - \$17

BORON

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as B

General: Boron is usually found in nature as a sodium or calcium borate salt. A major source of boron in domestic wastewater is sodium perborate, used as a bleach in household washing powders. Fluoroborate solutions are used for plating of cadmium, copper, lead, nickel, tin, and zinc. Boron salts are used in fire retardants, the production of glass, leather tanning and finishing industries, cosmetics, photographic materials, metallurgy, and for high energy rocket fuels. The ingestion of excessive doses of borates may cause nausea, cramps, convulsions, coma, or other symptoms of distress. It appears to pose a greater hazard to plants than humans, however. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 750 $\mu\text{g}/\ell$ for long-term irrigation on sensitive crops

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Polyethylene bottles or alkali-resistant, boron-free glassware.

Sample Volume Required: 50-200 mL

Measurement: The curcumin method using colorimetric equipment is recommended for concentrations in the 100 to 1,000 $\mu\text{g}/\ell$ range. When a sample of water containing boron is acidified and evaporated in the presence of curcumin, a red-colored product called rosocyanine is formed. The rosocyanine is taken up in a suitable solvent, and the red color is compared with standards either visually or photometrically. One of the following equipments will be required: (a) spectrophotometer for use at 540 nm with a light path of 1 cm, or (b) a filter photometer equipped with a green filter having a maximum transmittance near 540 nm with a minimum light path of 1 cm. Nitrate nitrogen concentrations above 20,000 $\mu\text{g}/\ell$ interfere. Significantly high results are possible when the total of calcium and magnesium hardness exceeds 100,000 $\mu\text{g}/\ell$ as CaCO_3 . Passing the sample through a cation exchange resin eliminates this problem. Close control of such variables as volumes and concentrations of reagents, as well as time and temperature of drying, must be exercised for maximum accuracy. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The minimum detectable quantity is 0.2 $\mu\text{g}/\ell$ B. A synthetic sample, containing 240 $\mu\text{g}/\ell$ B, 40 $\mu\text{g}/\ell$ As, 250 $\mu\text{g}/\ell$ Be, 20 $\mu\text{g}/\ell$ Se, and 6 $\mu\text{g}/\ell$ V in distilled water, was analyzed by the curcumin method in 30 laboratories with a relative standard deviation of 22.8% and a relative error of 0%.

Cost of Analysis: \$5 - \$20

BROMIDE

Parameter Group: General
Inorganic

STORET Units: mg/l as Br

General: Naturally occurring bromide in water is negligible, outside of coastal areas, the major sources being chemical industry and saltworks effluents. It is used for medicinal compounds, dyestuffs, gasoline additives, and swimming pool water sterilization. Like other halogens it is antiseptic and disinfectant and, hence, may possibly interfere with bacterial and other natural purification processes. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The titrimetric method is recommended. The concentration range for this method is 2-20 mg bromide/l. After pretreatment to remove interferences, the sample is divided into two aliquots. One aliquot is analyzed for iodide. The other aliquot is analyzed for iodide plus bromide. Bromide is then calculated by difference. Iron manganese and organic matter can interfere; however, the calcium oxide pretreatment removes or reduces these to insignificant concentrations. Color interferes with the observation of indicator and bromine - water color changes. This interference is eliminated by the use of a pH meter instead of a pH indicator and the use of standardized amounts of oxidant and oxidant quencher. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: In a single laboratory, using a mixed domestic and industrial waste effluent, at concentrations of 0.3, 2.8, 5.3, 10.3, and 20.3 mg/l of bromide, the relative standard deviations were 43%, 13%, 7.2%, 4.3%, and 2.1%, respectively. At concentrations of 2.8, 5.3, 10.3, and 20.3 mg/l of bromide, recoveries were 96%, 83%, 97%, and 99%, respectively.

Cost of Analysis: \$15 - \$20

CADMIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Cd

General: Cadmium occurs in nature chiefly as a sulfide salt, frequently in association with zinc and lead ores. The salts of the metal also may occur in wastes from electroplating plants, pigment works, textile and chemical industries. Cadmium is also used in everyday items such as paint, some pottery pigments, plastics, and automobile tires. Cadmium is present as an impurity in the more common galvanized coatings. Biologically, cadmium is a nonessential, nonbeneficial element recognized to be of high toxic potential. The concentration and not the absolute amount determines the acute toxicity of cadmium. Cadmium and cadmium compounds produce acute or chronic symptoms varying in intensity from irritations to extensive disturbances resulting in death. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 10 $\mu\text{g}/\ell$ for domestic water supply (health)
- Aquatic Life:

Fresh Water

Soft Water

Hard Water

0.4 $\mu\text{g}/\ell$

1.2 $\mu\text{g}/\ell$

for cladocerans
and salmonid
fishes

4.0 $\mu\text{g}/\ell$

12.0 $\mu\text{g}/\ell$

for other, less
sensitive, aquatic
life

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 228.8 nm. For levels of cadmium below 20 µg/ℓ, the extraction procedure is recommended. The dithizone procedure may also be used. It requires either a spectrophotometer for use at 518 nm or a filter photometer equipped with a green filter having a maximum light transmittance near 518 nm; either must provide a light path of at least 1 cm. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 250 µg/ℓ; its detection limit is 2 µg/ℓ. The optimum concentration range is 50-2,000 µg/ℓ. At a concentration of 50 µg/ℓ, for the AA method the relative standard deviation is 21.6% and the relative error is 8.2%, while for the dithizone method they are 24.6% and 6.0%, respectively.

Cost of Analysis: \$10 - \$15

CALCIUM

Parameter Group: Metals

STORET Units: mg/ℓ as Ca

General: Calcium enters water supplies through passage over deposits of limestone, dolomite, gypsum, and gypsiferous shale. Calcium salts and ions are among the most commonly encountered substances in water. Calcium salts breakdown on heating to form scale in boilers, pipes, and cooking utensils. Calcium adds to the total hardness of water. Calcium salts used on unpaved roadways and in innumerable industrial discharges represent other sources. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 422.7 nm. Phosphate, sulfate and aluminum interfere but are masked by the addition of lanthanum. The nitrous oxide-acetylene flame will provide two to five times greater sensitivity and freedom from chemical interferences. Ionization interferences should be controlled by adding a large amount of alkali to the sample and standards. For general use, the EDTA titrimetric method is the method of choice due to its simplicity and rapidity. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 0.08 mg/ℓ; its detection limit is 0.003 mg/ℓ. The optimum concentration range is 0.2-20 mg/ℓ. In a single laboratory, using distilled water at concentrations of 9.0 and 36 mg/ℓ, the relative standard deviations were 3.3% and 1.6%, respectively. Recoveries at both these levels were 99%. In a 44-laboratory test, synthetic unknown samples containing 108 mg/ℓ Ca (with other metals) were analyzed with a relative standard deviation of 9.2% and a relative error of 1.9%.

Cost of Analysis: \$5 - \$15

CAPTAN

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Captan is an approved name for the organochlorine fungicide $\text{C}_9\text{H}_8\text{Cl}_3\text{NO}_2\text{S}$. It is the active ingredient in the proprietary product Captan 50-W and was also known as SR-406, Vancide 89, and Orthocide. It is insoluble in water but partially soluble in some organic solvents. Captan has a very low toxicity to mammals (e.g., the LD_{50} for rats is over 9 g/kg of body weight) and is readily hydrolyzed, the effective residual life being on the order of two weeks. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for captan. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors but usually falls in the 0.001 to $1 \mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

CARBARYL

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Carbaryl, commonly known as Sevin, is an O-ARYL carbamate insecticide. It is commonly used on lawns as well as for other purposes. It is slightly soluble in water, sparingly soluble in most organic solvents, but freely soluble in amides. It has low mammalian toxicity, the acute oral LD_{50} to rats being reported from 500,000 to 2,190,000 $\mu\text{g}/\text{kg}$ of body weight. Although persistent, its toxicity to aquatic life appears to be low also. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 1,000 mL

Measurement: In the recommended method, a measured volume of water is extracted with methylene chloride, and the concentrated extract is cleaned up with a Florisil column. Appropriate fractions from the column are concentrated and portions are separated by thin-layer chromatography. The carbamates are hydrolyzed on the layer and the hydrolysis products are reacted to yield specific colored products. Quantitative measurement is achieved by visually comparing the responses of sample extracts to the responses of standards on the same thin layer. Identifications are confirmed by changing the pH of the layer and observing color changes of the reaction products. Phenols interfere directly, and indirect interferences may be encountered from naturally colored materials whose presence masks the carbamate reaction. The method is recommended for use only by an experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Carbaryl can be determined with a sensitivity of $1 \mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 - \$60

CARBON TETRACHLORIDE

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Carbon tetrachloride, CCl_4 , is used in industry as an organic solvent, fire extinguisher, and for dry cleaning of clothing. In human and veterinary medicine, it is used as an anti-helminthic. Carbon tetrachloride is colorless nonflammable liquid with a strong odor. Death has occurred from ingestion of 5 mL, about 8 grams. Repeated skin contact will result in dermatitis. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect the sample from phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: The recommended method for carbon tetrachloride is a direct aqueous-injection procedure for the determination of gas chromatographable chlorinated hydrocarbons. A 3-10 $\mu\ell$ aliquot of the sample is injected into the gas chromatograph equipped with a halogen specific detector. Compounds containing bromine or iodine will interfere with the determination. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 $\mu\text{g}/\ell$. Detection limits of 0.2-3 $\mu\text{g}/\ell$ may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60.

CHEMICAL OXYGEN DEMAND

Parameter Group: Oxygen Demand STORET Units: mg/ℓ

General: The chemical oxygen demand (COD) test determines the quantity of oxygen required to oxidize a portion of organic matter in a waste sample, under specific conditions of oxidizing agent, temperature, and time. It is an important parameter for stream and industrial waste studies and control of waste treatment plants and can be rapidly determined. Exactly the same technique must be used each time, since the results depend upon the chemical oxidant used, the structure of the organic compounds, and the manipulative procedures. Although empirical correlations with other oxygen demand indicators may be made for a given waste stream, there is no uniform theoretical basis for association. COD is not a pollutant in and of itself and exercises no direct harm. Its indirect effect is to depress dissolved oxygen levels. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add H_2SO_4 to pH <2.

Maximum Holding Time: 7 days

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The dichromate reflux method is recommended. The method is applicable to domestic and industrial waste samples having an organic carbon concentration greater than 15 mg/ℓ. For lower concentrations of carbon such as in surface water samples, the low level modification should be used. When the chloride concentration of the sample exceeds 2,000 mg/ℓ, the modification for saline waters is required. To reduce loss of volatile organics, the flask should be cooled during addition of the sulfuric acid solution. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Eighty-six analysts in fifty-eight laboratories analyzed a distilled water solution containing oxidizable organic material equivalent to 270 mg/ℓ COD. The relative standard deviation was 6.6% and relative error was 4.7%. A set of synthetic unknowns analyzed by 74 laboratories resulted in a relative standard deviation of 6.5% at the 200 mg/ℓ COD level. At 160 mg/ℓ COD and 100 mg/ℓ chloride, the relative standard deviation was 10.8%.

Cost of Analysis: \$10 - \$17

CHLORDANE

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Chlordane, the common name of an organochlorine insecticide, is a highly persistent chemical which bioaccumulates in aquatic organisms used for human food. Technical grade chlordane is a mixture of toxic compounds that have not been separated in manufacture. There is an extremely wide range for the acute toxicity of chlordane to various species of freshwater fishes. Fishes can concentrate chlordane directly from water by a factor of 1,000 to 3,000 times, and invertebrates may concentrate to twice this magnitude. Chlordane is stable in the soil and is fungicidal. It could be contained in irrigation return flows. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 0.01 $\mu\text{g}/\ell$ for freshwater aquatic life
- 0.004 $\mu\text{g}/\ell$ for marine aquatic life
- The persistence, bioaccumulation potential, and carcinogenicity of chlordane cautions human exposure to a minimum.

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for chlordane under favorable conditions. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

CHLORIDE

Parameter Group: General
Inorganic

STORET Units: mg/ℓ as Cl

General: Chloride is one of the major inorganic anions in water arising from natural mineral origin, seawater intrusion, salts used for agricultural purposes, sewage, industrial effluents (including paperworks, galvanizing plants, water softening plants, oil wells, and petroleum refineries), roadway deicing, and other sources. Chlorides in drinking water are not normally harmful at palatable concentrations. It is generally the cation associated with the chloride that produces a harmful effect. Chloride ions exert a significant effect on the corrosion rate of metals (e.g., steel and aluminum) and are considered to be among the most troublesome anions in irrigation water. Injury to livestock seldom occurs below the 4,000 mg/ℓ level, but injury to fish has been reported at 400 mg/ℓ. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: None required

Maximum Holding Time: 7 days

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The mercuric nitrate method is recommended wherein a dilute mercuric nitrate solution is added to an acidified sample in the presence of mixed diphenylcarbazone-bromophenol blue indicator. The method is suitable for all concentration ranges, but to avoid large titration volumes, the sample aliquot should not contain more than 10 to 20 mg Cl per 50 mL. Sulfites interfere and, if their presence is suspected, oxidize by treating 50 mL of sample with 0.5 to 1.0 mL of H_2O_2 . Bromide and iodide are titrated in the same manner as chloride. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: A synthetic unknown sample containing 241 mg/ℓ Cl was analyzed in 10 laboratories with a relative standard deviation of 3.3% and a relative error of 2.9%.

Cost of Analysis: \$3 - \$4

CHLORINATED BENZENES

Parameter Group:

STORET Units:

General: Chlorinated benzenes (other than dichlorobenzenes) include chlorobenzene, 1, 2, 4-trichlorobenzene, and hexachlorobenzene. They are heavy liquids and settle to the bottom in quiescent water unless emulsified. Their chief use is as aquatic herbicides to control weeds in lakes and ditches. They have pungent odors and therefore are unlikely to cause serious harm to humans through direct ingestion. Mild symptoms of poisoning of sheep and cattle have been reported at concentrations in excess of 2,700 mg/ℓ. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: No standard procedures have been developed. The methodology generally requires extraction, concentration, and gas chromatographic analysis. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits of 0.1 to 10 µg/ℓ should be achievable. Precision and accuracy data are not available at this time.

Cost of Analysis: \$25 - \$40

CHLORINATED ETHANES

Parameter Group:

STORET Units:

General: Chlorinated ethanes are volatile halocompounds including: 1, 2-trichloroethane; hexachloroethane; 1, 2-dichloroethane; 1, 1, 1-trichloroethane; hexachloroethane; 1, 1-dichloroethane; 1, 1, 2-trichloroethane; 1, 1, 2, 2-tetrachloroethane; and chloroethane. Widely used in various industries and processes, their characteristics vary from compound to compound. For example, 1, 2-dichloroethane (also called ethylene dichloride, $C_2H_4Cl_2$) is a heavy liquid with a pleasant odor and sweet taste and is highly soluble in water. It is used as an industrial solvent and in the manufacture of tobacco extract. Its oral LD_{50} for rats is 770 mg/kg of body weight. By contrast, 1, 1, 1-trichloroethane (also called methyl chloroform) is insoluble in water. It is used as a solvent for fats, waxes, resins, and alkaloids, and for cleaning metal and plastic molds. Its toxicity towards the marine pinperch is twice that of 1, 1, 2-trichloroethane (i.e., 75-100 mg/l versus 150-175 mg/l). This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: In the recommended Bellar procedure the sample is stripped with an inert gas; volatiles are captured on an adsorbent trap and desorbed into a modified gas chromatograph equipped with a halogen-specific detector. Methodology should be checked for interferences, e.g., from bromine or iodine. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 $\mu\text{g}/\text{L}$. Detection limits of 0.2-3 $\mu\text{g}/\text{L}$ may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60

CHLORINATED NAPHTHALENE

Parameter Group:

STORET Units:

General: 2-chloronaphthalene ($C_{10}H_7Cl$) is a solid (at normal temperatures) polynuclear organic compound. It is insoluble in water, but moderately soluble in other media such as alcohol, benzene, and ether. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly. Cool to 4°C.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mL

Measurement: The general procedure involves extraction and measurement with a gas chromatograph. Various cleanup techniques to remove interferences may be required depending upon other constituents in the sample. A skilled chemist or specialist will be required. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits in the 1-10 $\mu g/l$ range should be achievable. Precision and accuracy data are not available at this time.

Cost of Analysis: \$40 - \$60

CHLORINATED PHENOLS (OTHER)

Parameter Group:

STORET Units:

General: Other chlorinated phenols ($C_6H_3Cl_3O$) include the tri- . chlorophenols and chlorinated cresols (C_7H_7ClO). Although their specific properties vary from compound to compound, they are generally only slightly soluble in water but fairly soluble in other media such as alcohol, benzene, and ethers. Their main aesthetic problem stems from their organoleptic properties in water and fish. For example, the threshold odor level in water for 2, 4, 6-trichlorophenol is 3 $\mu g/l$. They tend to be persistent in water and are capable of being transported long distances. They are not efficiently removed by conventional water treatment processes and can cause odor problems in distribution systems. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Acidify to a pH of 4 with H_3PO_4 . Add 1.0g $CuSO_4 \cdot 5H_2O/l$ to inhibit biodegradation of phenols. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mg/l or more depending upon initial concentration.

Measurement: The recommended method involves direct aqueous injection for the gas-liquid chromatographic determination of concentrates containing more than 1 mg/l phenolic compounds. A flame-ionization detector is used for their individual measurement. Suspended matter may interfere by plugging the microsyringe. Interfering nonphenolic organic compounds may be removed by distillation. Steps should be taken to minimize or eliminate ghosting. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Few precision and accuracy data are available. Precision is very operator dependent. For example, total precision may be 2 to 5 times single operator precision values.

Cost of Analysis: \$40 - \$60

CHLORINE DEMAND

Parameter Group: General Inorganic STORET Units: mg/l

General: The chlorine demand of water is caused by inorganic reductants and others and varies with the amount of chlorine applied, contact time, pH, and temperature. It is the difference between the amount of chlorine applied at the amount of free, combined, or total available chlorine remaining at the end of the contact period. The usual purpose of a chlorine demand test is to determine the amount of chlorine that must be applied to produce a specific residual after a selected period of contact, rather than as an indicator of pollution. It should not be confused with chlorine requirement.

Criterion: Not designated

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 200-500 mL; 5,000 mL may be required to develop a breakpoint curve.

Measurement: A laboratory method is recommended which involves the addition of chlorine to the sample until the "breakpoint" is reached. At the end of the contact period the free and/or combined available residual chlorine is determined by a suitable technique, e.g., the amperometric titration method.

Precision and Accuracy: Precision and accuracy will depend upon the method chosen to measure free and/or combined available residual chlorine.

Cost of Analysis: \$50 - \$80 with breakpoint curve

CHLORINE DIOXIDE

Parameter Group:

STORET Units:

General: Chlorine dioxide is added to water supplies to combat tastes and odors due to phenolic-type wastes, actinomycetes, and algae as well as to oxidize soluble iron and manganese to a more easily removable form. Chlorine dioxide acts as a disinfectant. See also the residual chlorine discussion.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Avoid exposing the sample to sunlight or agitation that aerates the sample excessively.

Maximum Holding Time: No holding. Analyze on site if possible.

Container Type: Plastic or glass

Sample Volume Required: 200 ml

Measurement: The amperometric titration method is recommended. It is an extension of the method for residual chlorine.

Precision and Accuracy: Precision and accuracy data are not available.

Cost of Analysis: \$30 - \$40

CHLORINE, RESIDUAL

Parameter Group: Miscellaneous STORET Units: mg/ℓ

General: The toxicity of chlorine to aquatic life will depend upon the concentration of total residual chlorine, which is the amount of free chlorine plus chloramines. The persistence of chloramines is dependent on the availability of material with a lower oxidation-reduction potential. Free available chlorine (HOCl and OCl^-) and combined available chlorine (mono- and di-chloramines) appear transiently in surface or ground waters as a result of disinfection of domestic sewage or from industrial processes that use chlorine for bleaching operations or to control organisms that grow in cooling water systems. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria: Total residual chlorine:

- 2.0 µg/ℓ for salmonid fish
- 10.0 µg/ℓ for other freshwater and marine organisms

Preservation Method: Analyze as soon as possible. Cool to 4°C. Chlorine determinations should be started immediately after sampling, avoiding excessive light and agitation.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The amperometric titration method is recommended. It is applicable to all types of waters and wastes that do not contain a substantial amount of organic matter. This method cannot be used for samples containing above 5 mg/ℓ total residual chlorine. Samples containing significant amounts of organic matter interfere with the amperometric titration and the iodometric method must be used. The amperometric titration is not subject to interference from color, turbidity, iron, manganese, or nitrite nitrogen. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: A sample containing a concentration of .8 mg/ℓ was analyzed by 23 laboratories using the amperometric method. The relative standard deviation was 42.3% with a relative error of 25.0%.

Cost of Analysis: \$30 - \$40

CHLOROALKYL ETHERS

Parameter Group:

STORET Units:

General: Chloroalkyl ethers are volatile halocompounds including bis (chloromethyl) ether, bis (2-chloroethyl) ether, and 2-chloroethyl vinyl ether (mixed). This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: No standard procedure has been established. Methodology may require extraction, concentration, gas chromatography, and mass spectrometry. Detection limits of 60 µg/L or less should be achievable if procedure is optimized for sample composition. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Precision and accuracy data are not available.

Cost of Analysis: \$20 - \$30 each

CHLOROFORM

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Chloroform is used as an anesthetic, counterirritant, solvent, cleansing agent, and antiseptic. It is a colorless and volatile liquid with an ethereal odor and sweetish taste. Stickleback will avoid solutions of 100,000 to 200,000 $\mu\text{g}/\ell$ of chloroform in tap water. At 500,000 $\mu\text{g}/\ell$, they become anesthetized. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect the sample from phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: The recommended method for chloroform is a direct aqueous-injection procedure for the determination of gas chromatographable chlorinated hydrocarbons. A 3-10 $\mu\ell$ aliquot of the sample is injected into the gas chromatograph equipped with a halogen specific detector. Compounds containing bromine or iodine will interfere with the determination. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 $\mu\text{g}/\ell$. Detection limits of 0.2-3 $\mu\text{g}/\ell$ may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60.

2-CHLOROPHENOL

Parameter Group:

STORET Units:

General: 2-chlorophenol (C_6H_5ClO) is a liquid only slightly soluble in water but fairly soluble in other media such as alcohol. Its major aesthetic problem stems from its organoleptic properties in water and fish. Threshold odor levels for 2-chlorophenol are around $2 \mu g/\ell$. It is a persistent substance, capable of being transported long distances in water and is not removed efficiently by conventional water treatment. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Acidify to a pH of 4 with H_3PO_4 . Add $1.0g CuSO_4 \cdot 5H_2O/\ell$ to inhibit biodegradation of phenols. Cool to $4^\circ C$.

Maximum Holding Time: 24 hours

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mg/ ℓ or more depending upon initial concentration.

Measurement: The recommended method involves direct aqueous injection for the gas-liquid chromatographic determination of concentrates containing more than $1 mg/\ell$ phenolic compounds. A flame-ionization detector is used for their individual measurement. Suspended matter may interfere by plugging the microsyringe. Interfering nonphenolic organic compounds may be removed by distillation. Steps should be taken to minimize or eliminate ghosting. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Few precision and accuracy data are available. Precision is very operator dependent. For example, total precision may be 2 to 5 times single operator precision values.

Cost of Analysis: \$40 - \$60

CHROMIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Cr

General: The primary source of chromium is industrial discharges. Chromium compounds are used in cooling water to inhibit corrosion and are employed in the manufacture of paint pigments, in chrome tanning, aluminum anodizing, and other metal cleaning, plating, and electroplating operations. Chromium in industrial wastes occurs predominately as the hexavalent form, but the trivalent form is also present, either as a result of partial wastewater treatment or from its direct use. Industries that use trivalent chromium directly in manufacturing processes include glass, ceramics, photography, and textile dyeing. It is not clear if chromium is an essential element to man. Hexavalent chromium has been considered a toxic metal for years. Trivalent chromium is less toxic, no reports of oral toxicity are known. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 50 $\mu\text{g}/\ell$ for domestic water supply (health)
- 100 $\mu\text{g}/\ell$ for freshwater aquatic life

Preservation Method: Acidify all samples at the time of collection to keep the metal in solution and prevent plating out on the container wall; therefore, analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended for the determination of total chromium in water and wastewater samples. The colorimetric method may be used for the determination of hexavalent chromium in potable water. Use a wavelength of 357.9 nm with the AA spectrophotometric method. The absorption of chromium is suppressed by iron and nickel. If the analysis is performed in a lean flame the interference can be lessened but the sensitivity will also be reduced. The interference does not exist in nitrous oxide-acetylene flame. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 100 $\mu\text{g}/\ell$; its detection limit is 20 $\mu\text{g}/\ell$. The optimum concentration range is 200-10,000 $\mu\text{g}/\ell$. At a concentration of 50 $\mu\text{g}/\ell$, the relative standard deviation is 26.4%, and the relative error is 2.3%. These decrease with concentration; at 15.0 $\mu\text{g}/\ell$ they are 60% and 6.8% respectively, while at 7.4 $\mu\text{g}/\ell$ they are 105% and 38%, respectively.

Cost of Analysis: \$10 - \$15

COBALT

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Co

General: Cobalt naturally occurs primarily as arsenide and sulfide, generally associated with iron, nickel, copper, and silver minerals. Cobalt is used in alloys for magnets, high hardness steels, cutting tools, heat resistant jet engine parts, etc., and may appear in discharges from these and other industrial sources, including nuclear technology, china and glass, ink, galvanoplasting, and as a feed supplement in salt licks. Ingestion of cobalt salts may cause nausea or vomiting due to irritation, but it has a relatively low toxicity to man. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 240.7 nm. For levels of cobalt below 50 mg/ℓ , the extraction procedure is recommended. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 200 $\mu\text{g}/\ell$; its detection limit is 30 $\mu\text{g}/\ell$. The optimum concentration range is 500-10,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 200, 1,000 and 5,000 $\mu\text{g Co}/\ell$, the relative standard deviations were 6.5%, 1.0%, and 1.0%, respectively. Recoveries at these levels were 98%, 98%, and 97%, respectively.

Cost of Analysis: \$10 - \$15

COLOR

Parameter Group: Physical

STORET Units: Platinum-
Cobalt Units

General: The most common causes of color in natural water are minerals and complex organic compounds originating from the decomposition of naturally-occurring organic matter. Sources of organic material include humic materials from the soil such as tannins, humic acid and humates; decaying plankton; and other decaying aquatic plants. Virtually all industrial discharges and irrigation return flows also contain color to varying extents. The effects of color on public water supplies are aesthetic. The effects of color in water on aquatic life are to reduce light penetration, and thereby generally reduce photosynthesis by phytoplankton and to restrict the zone for aquatic vascular plant growth. Color is undesirable in waters for a number of industrial uses also. Color values are extremely pH dependent. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria:

- Waters shall be virtually free from substances producing objectionable color for aesthetic purposes;
- The source of supply should not exceed 75 color units on the platinum-cobalt scale for domestic water supplies;
- Increased color (in combination with turbidity) should not reduce the depth of the compensation point for photosynthetic activity by more than 10 percent from the seasonally established norm for aquatic life.

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The platinum-cobalt visual comparison method is acceptable for measuring the color of potable water. The method is not applicable to color measurement on waters containing highly colored industrial wastes, in which case the spectrophotometric or tristimulus methods are useful. In the platinum-cobalt method, color is measured by visual comparison of the sample with platinum-cobalt standards. One unit of color is that produced by

1 mg/l platinum in the form of the chloroplatinate ion. Slight amounts of turbidity interfere with the determination; therefore, samples showing visible turbidity should be clarified by centrifugation. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Precision and accuracy data are not available at this time.

Cost of Analysis: \$3 - \$5 for visual
 \$10 - \$15 for tristimulus
 \$30 - \$40 for spectrophotometric (10 ordinates)
 \$70 - \$80 for spectrophotometric (30 ordinates)

COPPER

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Cu

General: Oxides and sulfates of copper are used for pesticides, algicides, and fungicides. Copper is frequently incorporated into paints and wood preservatives to inhibit growth of algae and invertebrate organisms. Copper salts are used in water supply systems for controlling biological growths and for catalyzing the oxidation of manganese. Primary sources of copper in industrial wastewater are metal process pickling and plating baths; other sources involve mine drainage, pulp and paper mills, fertilizer manufacturing, petroleum refining, and certain rayon processes. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 1.0 mg/ ℓ for domestic water supplies (welfare).
- For freshwater and marine aquatic life, 0.1 times a 96-hour LC_{50} as determined through nonaerated bio-assay using a sensitive aquatic resident species.

Preservation Method: Copper ion tends to be adsorbed on the surface of the sample container; therefore, analyze as soon as possible. If storage is necessary, use 0.5 mL 1 + 1 HCl per 100 mL of sample to prevent plating out. Alternatively, add HNO_3 to pH < 2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 50 to 200 mL

Measurement: The AA spectrophotometric and neocupreine methods are recommended because of their high degree of freedom from interferences. The latter requires either a spectrophotometer for use at 457 nm or a filter photometer equipped with a narrow-band violet filter having maximum transmittance in the 450- to 460-nm range; either must provide a light path of at least 1 cm. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 100 $\mu\text{g}/\ell$; its detection limit is 100 $\mu\text{g}/\ell$. Precision and accuracy decrease with concentration. At 1,000 $\mu\text{g}/\ell$, the relative standard deviation is around 11% and the relative error, 3%. At 300 $\mu\text{g}/\ell$, the relative standard deviation has increased to nearly 18%, at 70 $\mu\text{g}/\ell$ it is over 30%, and approaching 10 $\mu\text{g}/\ell$ it exceeds 80%. Relative error has increased to nearly 16% at the last concentration.

Cost of Analysis: \$5 - \$10

CYANIDE

Parameter Group: General
Inorganic

STORET Units: mg/ℓ as CN

General: All of the CN groups in cyanide compounds that can be determined as the cyanide ion, CN^- , whether in simple, e.g., $\text{A}(\text{CN})_x$, or complex, $\text{AyM}(\text{CN})_x$, form. In the first expression, A may be an alkali or a metal; in the second, A is an alkali and M a heavy metal. In such latter alkali-metallic cyanides, the anion is not the CN group but the radical $\text{M}(\text{CN})_x$. Sources of cyanide in waste streams include ore mining and extracting, photographic processing, coke furnaces, synthetic manufacturing, case hardening and pickling of steel, electroplating, and industrial gas scrubbing. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 5.0 $\mu\text{g}/\ell$ for freshwater and marine aquatic life and wildlife.

Preservation Method: Most cyanides are very reactive and unstable. Analyze as soon as possible. If oxidizing agents are present, reduce with ascorbic acid. Add NaOH to raise sample pH to 12 or above and cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 500 mL

Measurement: For total cyanides, both dissociable and nondissociable forms of cyanide are being measured. Cyanides amenable to chlorination represent only the former. Standard methods for determination of total cyanide make use of a reflux-distillation procedure for concentrating and removing cyanides. The liberated hydrogen cyanide is collected in sodium hydroxide, and its concentration determined by using a titration method (above 1 mg/ℓ), a colorimetric method (below 1 mg/ℓ), or an ion selective electrode method (0.05 to 10 mg/ℓ). Although the distillation procedure eliminates or reduces many interferences, sulfides will distill over and adversely affect the colorimetric and titrimetric procedures, fatty acids will distill and form soaps under the alkaline titration procedures obscuring the end point, thiocyanates may interfere when distillation is carried out with the cuprous chloride reagent, and aldehydes will convert cyanide to nitrile under the distillation conditions. Special precautions are required when any of these are present. The colorimetric method requires either a spectrophotometer for use at 578 nm or a filter photometer equipped with a red filter having maximum transmittance

in the 570 to 580 nm range; either must provide a light path of 1 cm. The ion selective electrode method requires a suitable meter, a cyanide-ion selective electrode, and a double junction reference electrode. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The titrimetric method yields a relative standard deviation of 2% for samples containing more than 1 mg/l CN without significant interferences, increasing with decreasing concentration down to the limit of sensitivity, which is around 0.1 mg/l, e.g., at 0.4 mg/l the relative standard deviation is 8%. The colorimetric method is sensitive to about 0.02 mg/l. Within its designated range, its overall precision is given as $0.115X + 0.031$, where X is the CN concentration in mg/l. Using mixed domestic and industrial waste samples at concentrations of 0.28 and 0.62 mg/l CN, relative standard deviations of 11% and 15% and recoveries of 85% and 102%, respectively, were observed in a single laboratory. The overall precision of the ion selective electrode method is given as $0.113X + 0.024$, where X is the concentration in mg/l CN.

Cost of Analysis: \$10 - \$30

CAUTION! Exercise care in the manipulation of cyanide samples because of their toxicity. Avoid contact, inhalation, or ingestion.

2, 4-D

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: 2, 4-D (2, 4-dichlorophenoxyacetic acid) is the widely used chlorophenoxy herbicide $\text{C}_8\text{H}_6\text{Cl}_2\text{O}_3$. This compound is formulated in a variety of salts and esters that may have a marked difference in herbicidal properties, but all are hydrolyzed rapidly to the corresponding acid in the body. 2, 4-D herbicide is used for weed control on land, and as an aquatic herbicide in lakes, streams, and irrigation canals. It is a plant hormone that stimulates excessive growth, causing the plant to destroy itself. 2, 4-D is of low toxicity to mammals, the acute oral LD_{50} for rats being 500,000 $\mu\text{g}/\text{kg}$ of body weight, but may give water an unpleasant taste. Fish toxicity levels are in the hundreds of mg/ℓ . This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 100 $\mu\text{g}/\ell$ for domestic water supply (health).

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mL, depending upon measurement method used.

Measurement: In the recommended method, chlorinated phenoxy acids and their esters are extracted from the acidified water sample with ethyl ether. The esters are hydrolyzed to acids and extraneous organic material is removed by a solvent wash. The acids are converted to methyl esters which are extracted from the aqueous phase. The extract is cleaned up by passing it through a micro-adsorption column. Detection and measurement are accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography. Interferences may be high and varied and often pose great difficulty in obtaining accurate and precise measurement of chlorinated phenoxy acid herbicides. Organic acids, especially chlorinated acids, cause the most direct interference with the determination. Phenols including chlorophenols will also interfere with this procedure. The method is recommended for use only by an experienced pesticide analyst (or under the close supervision of such a person): For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is 1 $\mu\text{g}/\ell$. Detection limits of 0.05 $\mu\text{g}/\ell$ or so may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: \$45 - \$150, depending upon preparation required.

DDD

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: DDD, a metabolite of DDT, is an organochlorine insecticide. It is the same as TDE and is also known as Rhothane. DDD has much the same properties and is used similarly to DDT. Its insecticidal activity approaches that of DDT, but its mammalian toxicity is only about 20% of that of DDT. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for DDD. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

DDE

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: DDE, a metabolite of DDT, is an organochlorine insecticide. It is the same as DDX. DDE has much the same properties and is used similarly to DDT. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for DDE. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

DDT

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: DDT (1, 1, 1-trichloro -2, 2-bis (p-chlorophenyl) ethane) is an organochlorine insecticide. Acute toxicity to mammals generally is low. DDT is a highly persistent chemical which bioaccumulates in aquatic organisms used for human food and also is considered a potential human carcinogen. DDT will accumulate in the food chain. A residue accumulation of up to two million times for fish can occur. Application of DDT in agriculture and forest areas contributes to the presence of this toxic material in surface and ground waters. Practically insoluble in water, dilute acids, and alkalies, it is readily soluble in many organic solvents. The vehicle is very important in determining the toxicity of DDT. It has been found in river waters at concentrations to $20 \mu\text{g}/\ell$. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criterion:

- $0.001 \mu\text{g}/\ell$ for freshwater and marine aquatic life
- The persistence, bioaccumulation potential, and carcinogenicity of DDT cautions human exposure to a minimum.

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for DDT. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration. For example, at the 0.040 and 0.200 $\mu\text{g}/\ell$ concentrations, recoveries were around 101% and 77% and precisions were 40% and 19%, respectively.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

DEMETON

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Demeton (also known as Systox) is the organophosphorus insecticide $\text{C}_8\text{H}_{19}\text{O}_3\text{PS}_2$. Commercial demeton is a mixture of isomers of varying toxicities. It is insoluble in water but soluble in alcohol. The estimated fatal dose to a 70-kg man is 0.1 gram. The acute oral LD_{50} for stock and wildlife is reported between 2,500 to 40,000 $\mu\text{g}/\text{kg}$ of body weight. Toxicity to aquatic life varies widely with age and species. Demeton is unique in that the persistence of its ACHE enzyme inhibiting ability is greater than that of ten other common organophosphates, even though its acute toxicity is apparently less. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 0.1 $\mu\text{g}/\ell$ for freshwater and marine aquatic life.

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100 mL or more

Measurement: The use of co-solvent extraction, column chromatography, and liquid-liquid partition, and detection and measurement accomplished by flame photometric gas chromatography using a phosphorus specific filter is recommended for demeton. Great care must be exercised in the selection and use of methods to minimize interferences, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors but is usually 0.010 $\mu\text{g}/\ell$ or higher. Sensitivity is typically 1 $\mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

DIAZINON

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Diazinon is the registered trade name of an organophosphorus insecticide. It is a liquid with a faint ester-like odor and is miscible with a number of hydrocarbon solvents. Diazinon has very high insecticidal and acaricidal properties. The estimated fatal dose for a 70-kg man is 25 grams. The oral LD_{50} to rats ranges from 100,000 to 435,000 $\mu\text{g}/\text{kg}$ of body weight. Toxicity data for aquatic life are limited. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100 mL or more

Measurement: The use of co-solvent extraction, column chromatography, and liquid-liquid partition, and detection and measurement accomplished by flame photometric gas chromatography using a phosphorus specific filter is recommended for diazinon. Great care must be exercised in the selection and use of methods to minimize interferences, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but is usually $0.010 \mu\text{g}/\ell$ or higher. Sensitivity is typically $1 \mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

DICHLOROBENZENES

Parameter Group:

STORET Units:

General: Dichlorobenzenes ($C_6H_4Cl_2$) include 1, 2-dichlorobenzene, 1, 3-dichlorobenzene, and 1, 4-dichlorobenzene. Metadichlorobenzene is a colorless liquid, insoluble in water, and seldom used commercially. Othodichlorobenzene is also a liquid and insoluble in water and is used as a solvent for waxes, for preserving plants, and for destroying insects such as termites. Paradichlorobenzene is a white crystalline solid with a characteristic odor used chiefly for killing moths, their larvae, and other insects. It is slightly soluble in water, 70 mg/l at 25°C. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: No standard procedures have been developed. The methodology generally requires extraction, concentration, and gas chromatographic analysis. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits of 0.1 to 10 µg/l should be achievable. Precision and accuracy data are not available at this time.

Cost of Analysis: \$25 - \$40

DICHLOROBENZIDINE

Parameter Group:

STORET Units:

General: Dichlorobenzidine (3, 3'-dichlorobenzidine) is a polynuclear organic compound. Due to its suspected carcinogenic properties, it must be handled with great care. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 1 week

Container Type: Borosilicate glass

Sample Volume Required: 1,000-4,000 mL depending on concentration and instrument used.

Measurement: Dichlorobenzidine is separated and concentrated by multiple extractions and then oxidized by chloramine T. The oxidation product is extracted and measured spectrophotometrically. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is approximately 0.2 µg/L. Precision and accuracy data are not available at this time.

Cost of Analysis: \$20 - \$40; because of its carcinogenic properties, special facilities may be required at greatly increased cost.

DICHLOROETHYLENES

Parameter Group:

STORET Units:

General: Dichloroethylenes are volatile halocompounds including 1, 1-dichloroethylene and 1, 2-dichloroethylene. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: In the recommended Bellar procedure the sample is stripped with an inert gas; volatiles are captured on an adsorbent trap and desorbed into a modified gas chromatograph equipped with a halogen-specific detector. Methodology should be checked for interferences, e.g., from bromine and iodine. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 µg/L. Detection limits of 0.2-3 µg/L may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60

2, 4-DICHLOROPHENOL

Parameter Group:

STORET Units:

General: 2, 4-dichlorophenol ($C_6H_4Cl_2O$) is a colorless, crystalline substance only slightly soluble in water but fairly soluble in other media such as alcohol. It is used in the manufacture of the herbicide 2, 4-D as well as for other purposes. It is persistent and, since it is not efficiently removed by conventional water treatment processes, can cause odor problems in distribution systems. Fish flesh tainting concentrations range from 1 to 5 $\mu g/\ell$, levels that do not appear to adversely affect the fish. The threshold odor level in water is as low as 1 $\mu g/\ell$. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Acidify to a pH of 4 with H_3PO_4 . Add 1.0g $CuSO_4 \cdot 5H_2O/\ell$ to inhibit biodegradation of phenols. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mg/ ℓ or more depending upon initial concentration.

Measurement: The recommended method involves direct aqueous injection for the gas-liquid chromatographic determination of concentrates containing more than 1 mg/ ℓ phenolic compounds. A flame-ionization detector is used for their individual measurement. Suspended matter may interfere by plugging the microsyringe. Interfering nonphenolic organic compounds may be removed by distillation. Steps should be taken to minimize or eliminate ghosting. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Few precision and accuracy data are available. Precision is very operator dependent. For example, total precision may be 2 to 5 times single operator precision values.

Cost of Analysis: \$40 - \$60

DICHLOROPROPANE

Parameter Group:

STORET Units:

General: 1, 2-dichloropropane (also called propylene chloride, $C_3H_6Cl_2$) is a heavy liquid that is slightly soluble in water.

This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: In the recommended Bellar procedure the sample is stripped with an inert gas; volatiles are captured on an adsorbent trap and desorbed into a modified gas chromatograph equipped with a halogen-specific detector. Methodology should be checked for interferences, e.g., from bromine or iodine. Confirmation should be made for dichloropropane. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 $\mu\text{g}/\ell$. Detection limits of 0.2-3 $\mu\text{g}/\ell$ may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60

DICHLOROPROPENE

Parameter Group:

STORET Units:

General: 1, 3-dichloropropene ($C_3H_4Cl_2$) is a heavy liquid, insoluble in water, and with a chloroform-like odor. It is used as a soil fumigant for the control of nematodes. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: In the recommended Bellar procedure the sample is stripped with an inert gas; volatiles are captured on an adsorbent trap and desorbed into a modified gas chromatograph equipped with a halogen-specific detector. Methodology should be checked for interferences, e.g., from bromine or iodine. Confirmation should be made for dichloropropene. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 $\mu g/L$. Detection limits of 0.2-3 $\mu g/L$ may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60

DIELDRIN

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Dieldrin, the common name for an organochlorine insecticide, is a highly persistent chemical which bioaccumulates in aquatic organisms used for human food and is also considered a potential human carcinogen. The USEPA has suspended the production and use of dieldrin. This should result in a gradual decrease in concentration in the environment. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criteria:

- .003 $\mu\text{g}/\ell$ for freshwater and marine aquatic life
- The persistence, bioaccumulation potential, and carcinogenicity of dieldrin cautions human exposure to a minimum.

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for dieldrin. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration. For example, at the 0.02 and 0.125 $\mu\text{g}/\ell$ concentration, recoveries were around 108% and 85% and precisions were 91% and 24%, respectively.

Cost of Analysis: \$30 - \$150 depending upon preparation required.

2, 4-DIMETHYLPHENOL

Parameter Group:

STORET Units:

General: 2, 4-dimethylphenol (2, 4-dimethyl-1-hydroxybenzene) is only slightly soluble in water but highly soluble in other media such as alcohol. It has a higher odor threshold concentration than many other phenolic compounds, up to 75 $\mu\text{g}/\ell$. It is a persistent substance, capable of being transported long distances in water and is not removed efficiently by conventional water treatment processes. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Acidify to a pH of 4 with H_3PO_4 . Add 1.0g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}/\ell$ to inhibit biodegradation of phenols.

Maximum Holding Time: 24 hours

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mg/ ℓ or more depending upon initial concentration.

Measurement: The recommended method involves direct aqueous injection for the gas-liquid chromatographic determination of concentrates containing more than 1 mg/ ℓ phenolic compounds. A flame-ionization detector is used for their individual measurement. Suspended matter may interfere by plugging the microsyringe. Interfering nonphenolic organic compounds may be removed by distillation. Steps should be taken to minimize or eliminate ghosting. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Few precision and accuracy data are available. Precision is very operator dependent. For example, total precision may be 2 to 5 times single operator precision values.

Cost of Analysis: \$40 - \$60

DISSOLVED OXYGEN

Parameter Group: Dissolved Oxygen STORET Units: mg/ℓ

General: Dissolved oxygen (DO) levels in water, an important gage of its overall quality, depend upon its physical, chemical, and biological activities. Although excessive DO may be detrimental to certain uses (e.g., it increases metallic corrosion), the main concern is with DO deficiencies. Insufficient DO in the water column may be detrimental to aquatic fauna, causes anaerobic decomposition of any organic materials present, and generally degrades the aesthetic quality of the water body. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria:

- Aesthetics: Water should contain sufficient dissolved oxygen to maintain aerobic conditions in the water column and, except as affected by natural phenomena, at the sediment-water interface.
- Freshwater aquatic life: A minimum concentration of dissolved oxygen to maintain good fish populations is 5.0 mg/ℓ. The criterion for salmonid spawning beds is a minimum of 5.0 mg/ℓ in the interstitial water of the gravel.

Preservation Method: Electrode: determine on site; Winkler: fix on site.

Maximum Holding Time: No holding

Container Type: Glass only

Sample Volume Required: 300 mL

Measurement: The electrode method is recommended for a variety of reasons, including freedom from interferences and, when used in situ, from sampling effects that are otherwise difficult to account for. Modified Winkler methods may be used, but great care in sampling and accounting for interferences must be exercised. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: With most commercially available membrane electrode systems an accuracy of ± 0.1 mg/ℓ and a precision of ± 0.05 mg/ℓ should be obtainable. No meaningful precision and accuracy data are available for the modified Winkler method.

Cost of Analysis: \$3 - \$6

DISYSTON

Parameter Group: Pesticides STORET Units: $\mu\text{g}/\ell$

General: Disyston, an organophosphorus insecticide, is a clear, oily liquid that is slightly soluble in water and quite soluble in most organic solvents. Its acute oral LD_{50} to rats has been reported from 2,600 to 12,500 $\mu\text{g}/\text{kg}$ of body weight. Toxicity data for aquatic life are sparse but indicate a wide variability with age and species. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100 mL or more

Measurement: The use of co-solvent extraction, column chromatography, and liquid-liquid partition, and detection and measurement accomplished by flame photometric gas chromatography using a phosphorus specific filter is recommended for disyston. Great care must be exercised in the selection and use of methods to minimize interferences, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but is usually $0.010 \mu\text{g}/\ell$ or higher. Sensitivity is typically $1 \mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

DIURON

Parameter Group: Pesticides STORET Units: $\mu\text{g}/\ell$

General: Diuron is the urea pesticide $\text{C}_9\text{H}_{10}\text{Cl}_2\text{N}_2\text{O}$. It is a crystalline compound only slightly soluble in water and oils. Its acute oral LD_{50} to rats is 3,400 $\mu\text{g}/\ell$ of body weight. Its toxicity to fish varies widely with age and species, but lethal doses are measured in mg/ℓ concentrations. Apparently, aeration slightly reduces the toxicity of diuron to aquatic life. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 1,000 mL

Measurement: The recommended method involves an extraction process with methylene chloride and the concentrated extract is cleaned up with a Florisil column. A thin-layer chromatography process is then used. The layer is sprayed with 1-naphthol and the products appear as colored spots. The measurement is achieved visually. Direct interferences may be encountered from aromatic amines that may be present in the sample. Indirect interferences may be encountered from naturally colored materials whose presence masks the chromogenic reaction. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The determination can be made with a sensitivity of 1 $\mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 - \$60

ENDOSULFAN

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: The acute toxicity of endosulfan, an organochlorine insecticide, to different fish species varies widely. No data are available on the levels to which endosulfan could be expected to accumulate in tissues of aquatic organisms at various water concentrations. Residues in fish are not anticipated to pose a hazard to fish-eating predators because of endosulfan's low oral toxicity to birds and mammals. Application of endosulfan in agriculture and forest areas contributes to the presence of this toxic material in surface and ground waters. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 0.003 $\mu\text{g}/\ell$ for freshwater aquatic life
- 0.001 $\mu\text{g}/\ell$ for marine aquatic life

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for endosulfan. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

ENDRIN

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Application of endrin, an organochlorine insecticide, in agriculture and forest areas contributes to the presence of this toxic material in surface and ground waters. It is possible that some fish would accumulate endrin to 30,000 times water concentration. Although it has strong residual toxicity as does its closely related compound dieldrin, endrin has been found to be eliminated quickly after termination of exposure and to disappear relatively quickly. Thus, it does not appear to cause an accumulation problem. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criteria:

- 0.2 $\mu\text{g}/\ell$ for domestic water supply (health)
- 0.004 $\mu\text{g}/\ell$ for freshwater and marine aquatic life

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for endrin. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

ETHYLBENZENE

Parameter Group:

STORET Units:

General: Ethylbenzene (C_8H_{10}) is a volatile, flammable liquid with an ethereal odor. It is insoluble in water at normal temperatures. It is used commercially as a solvent and in the synthesis of other organic compounds. Its toxicity to fish varies with water temperature, age, and species. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: Hexadecane extraction followed by gas chromatographic and mass spectrometric analysis is often used. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits should be around 2-10 $\mu\text{g}/\text{L}$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$15 - \$30

FECAL COLIFORM

Parameter Group: Bacteriologic STORET Units: MPN

General: Pollution of aquatic systems by the excreta of warm-blooded animals creates public health problems for man and animals and potential disease problems for aquatic life. It is known that enteric microbial pathogens may inhabit the gut of most warmblooded animals and are shed in feces. The presence of bacterial, viral, protozoan, and possibly fungal species is indicated by the presence of the fecal coliform group of bacteria. The number of fecal coliforms present is indicative of the degree of health risk associated with using the water for drinking, swimming, or shellfish harvesting. The fecal coliform bacteria, which comprise a portion of the total coliform group, are able to grow at 44.5°C and ferment lactose, producing acid and gas. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria:

Bathing Waters

Based on a minimum of not less than five samples taken over a 30-day period, the fecal coliform bacterial level should not exceed a log mean of 200 per 100 mL, nor should more than 10 percent of the total samples taken during any 30 day period exceed 400 per 100 mL.

Shellfish Harvesting Waters

Not to exceed a median fecal coliform bacterial concentration of 14 MPN per 100 mL with not more than 10 percent of samples exceeding 43 MPN per 100 mL for the taking of shellfish.

Preservation Method: Cool to 4°C. Add a dechlorinating agent (e.g., sodium thiosulfate) if residual chlorine is present. Samples high in heavy metals should have a chelating agent (e.g., EDTA) added to reduce metal toxicity.

Maximum Holding Time: 6 hours (30 hours absolute maximum for potable water samples).

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The multiple tube fermentation technique may be used if a determination of the total coliform group is also being made. Otherwise, the simpler membrane filter technique is

recommended. Results of the former are expressed statistically as the Most Probable Number (MPN), while the latter are expressed as number of colonies per 100 mL. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The accuracy of the membrane filter technique for differentiating between coliforms from warm-blooded animals and coliforms from other sources is approximately 93%.

Cost of Analysis: \$10 - \$12 MFT
\$15 - \$20 MPN

FECAL STREPTOCOCCI

Parameter Group: Bacteriologic STORET Units: Unspecified

General: The normal habitat of the fecal streptococcus group of bacteria is the intestines of man and other warm-blooded animals and, thus, these organisms are indicators of fecal pollution. Because of their survival characteristics, it is not recommended that fecal streptococci be used as the sole fecal indicator. Since certain fecal streptococci are host-specific, they may provide valuable additional information about the source of pollution; e.g., a predominance of *S. bovis* and *S. equinus* would indicate excrement from nonhuman, warm-blooded animals as, for example, from feedlot and farmland runoff, dairy wastes, and meat processing plants. *S. faecalis* var *liquefaciens* is not restricted to the intestines of warm-blooded animals, being also associated with vegetation, insects, and certain types of soils. Biochemical characterization is required to eliminate the possibility of a preponderance of this latter type, thus avoiding misinterpretation of results. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C. Add a dechlorinating agent (e.g., sodium thiosulfate) if residual chlorine is present. Samples high in heavy metals should have a chelating agent (e.g., EDTA) added to reduce metal toxicity.

Maximum Holding Time: 6 hours (30 hours absolute maximum for potable water samples).

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The multiple tube fermentation technique and the simpler membrane filter technique are both recommended, especially for nondrinking water tests. Results of the former are expressed statistically as the Most Probable Number (MPN), while the latter are expressed as number of colonies per 100 mL. The fecal streptococcal plate count method may also be used. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Not applicable

Cost of Analysis: \$10 - \$12 MFT
\$15 - \$20 MPN

FLUORIDE

Parameter Group: General
Inorganic

STORET Units: mg/ℓ as F

General: The most reactive nonmetal, fluorine is never found free in nature, but it is a constituent of a number of minerals. Fluorides in high concentrations are not common in natural surface waters. They are used as insecticides, as disinfectants, as a flux in steelmaking, for preserving wood and mucilages, for the manufacture of glass and enamels, in chemical industries, tooth-paste manufacture, for water treatment, and a host of minor applications. They are not normally found in industrial wastes (other than traces) except as a result of spillage. In sufficient quantities (over 200 mg), fluorides can be toxic to humans. Up to 5 mg/ℓ the only bad effect seems to be tooth discoloration. Under 100 mg/ℓ produces little adverse effects on plants. Toxic effects on aquatic life are observed starting at concentrations above 2 mg/ℓ. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 7 days

Container Type: Plastic or glass

Sample Volume Required: 300 mL

Measurement: The SPADNS method with Bellack distillation is recommended. A spectrophotometer for use at 570 nm or a filter photometer equipped with a greenish yellow filter having maximum transmittance at 550-580 nm is required; either must have a light path of at least 1 cm. The method covers the range from 0.1 to about 2.5 mg/ℓ F. Following distillation to remove interferences, the sample is treated with the SPADNS reagent. The loss of color resulting from the reaction of fluoride with the zirconyl-SPADNS dye is a function of the fluoride concentration. The SPADNS reagent is more tolerant of interfering materials than other accepted fluoride reagents. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: On a sample containing 0.83 mg/ℓ F with no interferences, the results of 53 analysts using the SPADNS method had a relative standard deviation of 8% and a relative error of 1.2%. After direct distillation, the relative standard deviation was 11.0% and the relative error 2.4%. On a sample containing 0.57 mg/ℓ F (with 200 mg/ℓ SO₄ and 10 mg/ℓ Al as

interferences) results from the 53 analysts had relative standard deviations and errors of 16.2% and 7.0% without distillation and 17.2 and 5.3 with distillation.

Cost of Analysis: \$3 - \$5 without distillation
 \$15 - \$20 with distillation

GUTHION

Parameter Group: Pesticides STORET Units: $\mu\text{g}/\ell$

General: Guthion is the organophosphorus insecticide $\text{C}_{10}\text{H}_{12}\text{N}_3\text{O}_3\text{PS}_2$. It is a brown waxy solid that is insoluble in water but soluble in most organic solvents. The half-life of guthion spray and dust on cotton leaves has been reported as 2-4 days and 1-2 days for pondwater. An investigation of the persistence of guthion in fish revealed that 50% of the chemical was lost in less than one week. The estimated fatal dose for a 70-kg man is 0.2 grams. The acute oral LD_{50} to rats ranges from 11,000 to 80,000 $\mu\text{g}/\text{kg}$ of body weight. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 0.01 $\mu\text{g}/\ell$ for freshwater and marine aquatic life.

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100 mL or more

Measurement: The use of co-solvent extraction, column chromatography, and liquid-liquid partition, and detection and measurement accomplished by flame photometric gas chromatography using a phosphorus specific filter is recommended for guthion. Great care must be exercised in the selection and use of methods to minimize interferences, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but is usually 0.010 $\mu\text{g}/\ell$ or higher. Sensitivity is typically 1 $\mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

HALOETHERS

Parameter Group:

STORET Units:

General: Haloethers as used here comprise 4-chlorophenyl phenyl ether; 4-bromophenyl phenyl ether, bis (2-chloroisopropyl) ether; and bis (2-chloroethoxy) methane. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: No standard procedures have been developed. The methodology generally requires extraction, concentration, and gas chromatographic analysis. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits of 1 to 10 $\mu\text{g}/\ell$ should be achievable. Precision and accuracy data are not available at this time.

Cost of Analysis: \$40 - \$60

HALOMETHANES

Parameter Group:

STORET Units:

General: The halomethanes include dichloromethane (methylene chloride), chloromethane (methyl chloride), bromomethane (methyl bromide), tribromomethane (bromoform), dichlorobromomethane, trichlorofluoromethane (Freon 11), dichlorodifluoromethane (Freon 12), and chlorodibromomethane. These volatile halocompounds are mostly gaseous at surface water temperatures and atmospheric pressure. They range from soluble to insoluble in water; e.g., methyl chloride is soluble to about 4,000 mg/l at 20°C. Chief uses are as refrigerants, aerosol propellents, and certain industrial operations. Taste of water containing halomethanes in appreciable concentrations is sharp, sickening, and sweetish when first taken into the mouth, followed by a burning sensation. It is unlikely that humans would voluntarily drink such water. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 ml

Measurement: In the recommended Bellar procedure the sample is stripped with an inert gas; volatiles are captured on an adsorbent trap and desorbed into a modified gas chromatograph equipped with a halogen specific detector. Methodology should be checked for interferences, e.g., from bromine or iodine. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 µg/l. Detection limits of 0.2-3 µg/l may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60

HARDNESS, TOTAL

Parameter Group: General
Inorganic

STORET Units: mg/ℓ as CaCO₃

General: Water hardness is caused by the polyvalent metallic ions dissolved in water. Principally, these are calcium and magnesium. Other metals such as iron, strontium, and manganese contribute to the extent that appreciable concentrations are present. Natural sources of hardness are soil and geological formations (e.g., limestone) with which the water may have come in contact. Industrial sources include the inorganic chemical industry and discharges from operating and abandoned mines. Irrigation return flows also increase hardness. The detrimental effects of hardness include excessive soap consumption, the formation of scums and curds in laundries and textile mills, the toughening of vegetables cooked in hard water, and the formation of scabs in boilers, hot water heaters, pipes, and utensils. Hence, they are principally economic in nature. The hardness of "good" water should not exceed 250 mg/ℓ. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 7 days

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The EDTA method, recommended when a complete mineral analysis is not performed, is applicable to drinking, surface, and saline waters, domestic and industrial wastes. Calcium and magnesium ions in the sample are sequestered upon the addition of disodium ethylenediamine tetraacetate (Na₂EDTA). The end point of the reaction is detected by means of Calmagite Indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered. Excessive amounts of heavy metals can interfere. This is usually overcome by complexing the metals with cyanide. Routine addition of sodium cyanide solution to prevent potential metallic interference is recommended. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: A synthetic unknown containing 610 mg/ℓ total hardness as CaCO₃ was analyzed in 56 laboratories with a relative standard deviation of 2.9% and a relative error of 0.8%.

Cost of Analysis: \$5 - \$15

HEPTACHLOR

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: The acute toxicity of heptachlor, a refined ingredient of the well-known organochlorine insecticide chlordane, is generally low to mammals; however, aquatic organisms exhibit sensitivity to this pesticide at microgram-per-liter levels. Heptachlor will accumulate in the food chain. Heptachlor is a highly persistent chemical which bioaccumulates in aquatic organisms used for human food and also is considered a potential human carcinogen. In July 1975, the USEPA suspended the production and use of heptachlor. This should result in a gradual decrease in concentrations in the environment. Any addition of heptachlor to water should be considered potentially hazardous to humans. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion:

- 001 $\mu\text{g}/\ell$ for freshwater and marine aquatic life
- The persistence, bioaccumulation potential, and carcinogenicity of heptachlor cautions human exposure to a minimum.

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for heptachlor. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

IODIDE

Parameter Group: General
Inorganic

STORET Units: mg/l as I

General: Only trace concentrations of iodides are found in natural fresh water; seawater is somewhat higher. Higher concentrations may also be found in natural brines, waters treated with iodine as the disinfectant, and a limited number of industrial wastes. It is used sparingly in industry, e.g., for medicines, germicides, analytical chemistry, and as a table salt additive. All waterborne pathogens are destroyed by 8 mg/l of iodine; no adverse effects were reported when water containing over twice this concentration was consumed in the tropics. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 100 ml

Measurement: The titrimetric method is recommended. After pretreatment to remove interferences, the sample is analyzed for iodide by converting the iodide to iodate with bromine water and titrating with phenylarsine oxide (PAU) or sodium thiosulfate. Iron, manganese and organic matter can interfere; however, the calcium oxide pretreatment removes or reduces these to insignificant concentrations. Color interferes with the observation of indicator and bromine-water color changes. This interference can be eliminated by the use of a pH meter instead of a pH indicator and the use of standardized amounts of bromine water and sodium formate solution instead of observing the light yellow color changes. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: In a single laboratory, using a mixed domestic and industrial waste effluent, at concentrations of 1.6, 4.1, 6.6, 11.6, and 21.6 mg/l of iodide, the relative standard deviations were 14.4%, 4.1%, 1.4%, .5%, and 2.3%, respectively. At concentrations of 4.1, 6.6, 11.6 and 21.6 mg/l of iodide, recoveries were 80%, 97%, 97%, and 92%, respectively.

Cost of Analysis: \$15 - \$20

IRON

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Fe

General: Iron is an essential trace element required by both plants and animals. The ferrous, or bivalent (Fe^{++}), and the ferric, or trivalent (Fe^{+++}) irons, are the primary forms of concern in the aquatic environment. The ferrous (Fe^{++}) form can persist in waters void of dissolved oxygen and originates from groundwaters or mines when these are pumped or drained. The ferric (Fe^{++}) form is insoluble. Potential sources of dissolved iron species include discharges from mining operations, ore milling, chemical industries (organic, inorganic, petrochemical), dye industries, metal processing industries, textile mills, food canneries, tanneries, titanium dioxide production, petroleum refining, and fertilizers. Limitations in drinking water arise primarily from taste consideration. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria:

- 0.3 mg/ ℓ for domestic water supplies (welfare)
- 1.0 mg/ ℓ for freshwater aquatic life

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2. For precise determinations of total iron, use a separate container for sample collection and treat with acid immediately to place the iron in solution and prevent adsorption or desposition on the container walls.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 248.3 nm. The orthophenanthroline method may be used for natural and treated waters. It requires either a spectrophotometer for use at 510 nm or a filter photometer equipped with a green filter having maximum transmittance near 510 nm; either must have a light path of at least 1 cm. In the presence of excessive amounts of organic constituents, the sample should first be digested to ensure complete dissolution of the iron. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 120 $\mu\text{g}/\ell$; its detection limit is 20 $\mu\text{g}/\ell$. The optimum concentration range is 300-10,000 $\mu\text{g}/\ell$. At a concentration of 300 $\mu\text{g}/\ell$, the relative standard deviation is 16.5%, and the relative error is 0.6%. For the colorimetric method at 300 $\mu\text{g}/\ell$ Fe, the values were 25.5% and 13.3%, respectively, from a 44-laboratory test. Serious divergences have been found in reports of different laboratories because of variations in methods of collecting and treating samples.

Cost of Analysis: \$3 - \$15

LEAD

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Pb

General: Natural lead concentrations in surface waters may range up to $40 \mu\text{g}/\ell$. Lead and its compounds may also enter water at any stage during mining, smelting, and processing. Lead is used in the manufacture of storage batteries, television tubes, printing, pigments, fuels, photographic materials, pesticides, and explosives. The dissolution of lead plumbing is another source. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- $50 \mu\text{g}/\ell$ for domestic water supply (health)
- 0.01 times the 96-hour LC_{50} value, using the receiving or comparable water as the diluent and soluble lead measurements (nonfilterable lead using a 0.45 micron filter), for sensitive freshwater resident species.

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time. 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 283.3 nm. The analysis of this metal is exceptionally sensitive to turbulence and absorption bands in the flame. Therefore, care should be taken to position the light beam in the most stable, center portion of the flame. The dithizone colorimetric method may also be used. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is $500 \mu\text{g}/\ell$; its detection limit is $50 \mu\text{g}/\ell$. The optimum concentration range is 1,000-20,000 $\mu\text{g}/\ell$. At a concentration of $50 \mu\text{g}/\ell$, the relative standard deviation is 23.5%, and the relative error is 19.0%. At $25 \mu\text{g}/\ell$, the relative error was 25.7% in a 60-laboratory test.

Cost of Analysis: \$10 - \$15

LINDANE

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Lindane, the common name of the gamma isomer of benzene hexachloride (BHC), is an organochlorine insecticide. Application of lindane in agriculture and forest areas contributes to the presence of this toxic material in surface and ground waters. The highest level of lindane found to have minimal or no long-term effects in the most sensitive mammal tested, the dog, is 15.0 mg/kg in the diet or 0.3 mg/kg of body weight/day. An increased resistance to lindane toxicity among fish and invertebrates experiencing previous exposure to the chemical has been observed. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 4.0 $\mu\text{g}/\ell$ for domestic water supply (health)
- 0.01 $\mu\text{g}/\ell$ for freshwater aquatic life
- 0.004 $\mu\text{g}/\ell$ for marine aquatic life

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for lindane. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration. For

example, at the 0.010 and 0.100 $\mu\text{g}/\ell$ concentrations, recoveries were around 97% and 73% and precisions were 53% and 26%, respectively.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

LITHIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Li

General: Lithium is present in fresh waters in concentrations below 10,000 $\mu\text{g}/\ell$; brines and thermal waters may be higher. Lithium and its salts are used in dehumidifying units, as a deoxidizer and degasser for nonferrous castings, to form a protective atmosphere in furnaces, in medicinal waters, in metallurgical processes, in the manufacture of some types of glass and storage batteries, and as the hydride for many controlled organic reductions. In addition to these sources, lithium hypochlorite is used as a source of chlorine in some swimming pools. Lithium may have a toxic effect on plants and some forms of aquatic life, but little data exist documenting toxicity to man.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Pyrex bottle

Sample Volume Required: 100-200 mL

Measurement: The flame photometric method is often used, using a wavelength of 671 nm. Interferences in the photometric determination include barium, strontium, and calcium. These can be removed by the addition of a sodium sulfate-sodium carbonate solution. Digestion will be necessary if considerable organic matter is present.

Precision and Accuracy: The minimum detectable lithium concentration is approximately 100 $\mu\text{g}/\ell$. In a lithium range of 700 to 1,200 $\mu\text{g}/\ell$, an accuracy of ± 0.1 to 200 $\mu\text{g}/\ell$ can be obtained in the determination of the lithium concentration.

Cost of Analysis: \$12 - \$18

MAGNESIUM

Parameter Group: Metals

STORET Units: mg/l as Mg

General: Magnesium salts are important contributors of hardness to water. Sources of magnesium include mining and ore processing, oxide production, metallurgy, refractories, iron and steel production, and its use in flash and incendiary products, signal flares, as a deoxidizer in the casting of metals, as a reagent in organic chemistry, and a host of other applications. Magnesium is an essential element to humans, the daily requirement being about 700 mg. Taste considerations, rather than toxicity, are paramount for magnesium in drinking water. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 ml

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 285.2 nm. The interference caused by aluminum at concentrations greater than 2 mg/l is masked by the addition of lanthanum. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 0.007 mg/l; its detection limit is 0.0005 mg/l. The optimum concentration range is 0.02-2 mg/l. At a concentration of .2 mg/l, the relative standard deviation is 10.5%, and the relative error is 6.3%. In a single laboratory, using a distilled water sample at concentrations of 2.1 and 8.2 mg/l, the relative standard deviations were 4.7% and 2.4%, respectively. Recoveries at both of these levels were 100%.

Cost of Analysis: \$10

MALATHION

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Malathion, the organophosphorus pesticide $\text{C}_{10}\text{H}_{19}\text{O}_6\text{PS}_2$, enters the aquatic environment primarily as a result of its application as an insecticide. Because it degrades quite rapidly in most waters, depending on pH, its occurrence is sporadic rather than continuous. It is soluble in water to 145,000 $\mu\text{g}/\ell$. The freshwater fish most sensitive to malathion appear to be the salmonids and centrarchids. Many aquatic invertebrates appear to be more sensitive than fish to malathion. It appears to be about 100 times less toxic to warm-blooded animals than parathion, but only 2 to 4 times less toxic to insects. The estimated fatal dose for a 70-kg man is 60 grams. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 0.1 $\mu\text{g}/\ell$ for freshwater and marine aquatic life.

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100 mL or more

Measurement: The use of co-solvent extraction, column chromatography and liquid-liquid partition, and detection and measurement accomplished by flame photometric gas chromatography using a phosphorus specific filter is recommended for malathion. Great care must be exercised in the selection and use of methods to minimize interferences, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but is usually 0.010 $\mu\text{g}/\ell$ or higher. Sensitivity is typically 1 $\mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

MANGANESE

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Mn

General: Manganese and its salts are used in manufacturing steel alloys, dry cell batteries, glass and ceramics, paint and varnish, ink and dye, and matches and fireworks. Manganese is normally ingested as a trace nutrient in food. Very large doses of ingested manganese can cause some diseases and liver damage. Inadequate quantities of manganese in domestic animal food results in reduced reproductive capabilities and deformed or poorly maturing young. Manganese imparts objectionable and stubborn stains to laundry and plumbing fixtures. Low limits on domestic water supplies stem from these, rather than toxicological, considerations. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria:

- 50 $\mu\text{g}/\ell$ for domestic water supplies (welfare)
- 100 $\mu\text{g}/\ell$ for protection of consumers of marine mollusks

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 279.5 nm. For levels of manganese below 25 $\mu\text{g}/\ell$, the extraction procedure is recommended. Analytical sensitivity is dependent on lamp current. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 50 $\mu\text{g}/\ell$; its detection limit is 10 $\mu\text{g}/\ell$. The optimum concentration range is 100-10,000 $\mu\text{g}/\ell$. At a concentration of 50 $\mu\text{g}/\ell$, the relative standard deviation is 13.5%, and the relative error is 6.0%. These increase at decreasing concentrations. In a 55-laboratory test, at concentrations of 17 and 11 $\mu\text{g}/\ell$ the relative standard deviations were 118% and 245%, respectively, and the relative errors were 22% and 93%, respectively.

Cost of Analysis: \$10

MERCURY

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Hg

General: Mercury is widely distributed in the environment, and biologically is a nonessential or nonbeneficial element. Discharged mercury does not remain localized. Mercury can enter the environment by seeping up through layers of earth to the surface, outgassing of mercury from rock and soil, and transport by natural cycles. Most industrial mercury is eventually lost as waste into streams or the atmosphere. Uses of mercury include the electrical industry, chlor-alkali industry, industrial control equipment, paints, agriculture, dental preparations, pulp and paper industry, catalysts in chemical manufacturing processes, and general laboratory uses. The toxicity of mercury is attributed to its high affinity for sulfur-containing compounds. Toxic effects vary with the form of mercury and its mode of entry into the organism. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 2.0 $\mu\text{g}/\ell$ for domestic water supply (health)
- 0.05 $\mu\text{g}/\ell$ for freshwater aquatic life and wildlife
- 0.10 $\mu\text{g}/\ell$ for marine aquatic life

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 38 days (glass), 13 days (hard plastic)

Container Type: Glass or hard plastic

Sample Volume Required: 100 mL

Measurement: The flameless AA spectrophotometric method is recommended. It is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentrations. Possible interference from sulfide is eliminated by the addition of potassium permanganate. Copper has also been reported to interfere. Interference from certain volatile organic materials which will absorb at this wavelength is also possible. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: At a concentration of $0.4 \mu\text{g}/\ell$, the relative standard deviation is 21.2%, and the relative error is 2.4%. In a single laboratory, using an Ohio River composite sample with a background mercury concentration of $0.35 \mu\text{g}/\ell$, spiked with concentrations of 1, 3, and $4 \mu\text{g}/\ell$, the standard deviations were ± 0.14 , ± 0.10 , and ± 0.08 , respectively. Standard deviation at the 0.35 level was ± 0.16 . Percent recoveries at the three levels were 89%, 87%, and 87%, respectively.

Cost of Analysis: \$15 - \$25

METHANE

Parameter Group:

STORET Units:

General: Methane is a gaseous saturated (paraffin) hydrocarbon. It is colorless, odorless, tasteless, and flammable. Methane sources include the anaerobic decomposition of organic matter (e.g., some marshes, mines, treatment plants, etc.) and natural gas and petroleum plants. Concern about methane arises from its explosion hazard rather than its negligible toxicity. For example, an explosive limit of methane in air could be reached in a poorly ventilated space sprayed with hot (68°C) water having a methane concentration of only 0.7 mg/l.

Criterion: Not established

Preservation Method: Analyze as soon as possible. When collecting the sample, ensure that the sample is under sufficient pressure to ensure that no gas escapes from the water.

Maximum Holding Time: Unknown, but short

Container Type: Glass

Sample Volume Required: 3,000 mL

Measurement: The combustible-gas indicator method is often used. The procedure is based on the catalytic oxidation of a combustible gas or a heated platinum filament that is made a part of a wheat-stone bridge. Small amounts of ethane, hydrogen gas, and hydrogen sulfide may interfere. For greater accuracy, a gas chromatograph should be used.

Precision and Accuracy: The sensitivity of the method is approximately 0.2 mg/l. The accuracy of the determination is limited by the accuracy of the instrument employed; errors of around 10% may be expected.

Cost of Analysis: \$15 - \$20

METHOXYCHLOR

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Application of methoxychlor, an organochlorine insecticide, in agriculture and forest areas contributes to the presence of this material in surface and ground waters. It is slightly soluble in water but very soluble in alcohol. Sodium and dimethylamine salts are freely soluble in water. The concentration of methoxychlor has been found to be degraded in a few weeks or less in natural waters. The highest level of methoxychlor found to have minimal or no long-term effects in man is 2.0 mg/kg of body weight/day. Few data are available on acute and chronic effects of methoxychlor on freshwater fish. Methoxychlor appears to be considerably less bioaccumulative in aquatic organisms than some of the other organochlorine pesticides. Methoxychlor has a very low accumulation rate in birds and mammals. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria:

- 100 $\mu\text{g}/\ell$ for domestic water supply (health)
- 0.03 $\mu\text{g}/\ell$ for freshwater and marine aquatic life

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for methoxychlor. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

METHYL PARATHION

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Methyl parathion is an organophosphorus insecticide similar in action to parathion, Phosdrin, and TEPP. Its toxicity is also similar. The half-life of methyl parathion on cotton leaves is less than one hour. The estimated fatal dose for a 70-kg man is 0.15 gram. The acute oral LD_{50} for rats ranges from 9,000 to 25,000 $\mu\text{g}/\text{kg}$ of body weight. Toxicity data for aquatic life are sparse but appear to range widely with age and species. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100 mL or more

Measurement: The use of co-solvent extraction, column chromatography and liquid-liquid partition, and detection and measurement accomplished by flame photometric gas chromatography using a phosphorus specific filter is recommended for methyl parathion. Great care must be exercised in the selection and use of methods to minimize interferences, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but is usually $0.010 \mu\text{g}/\ell$ or higher. Sensitivity is typically $1 \mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

METHYLENE BLUE ACTIVE SUBSTANCES (MBAS)

Parameter Group: General
Organic

STORET Units: mg/ℓ

General: Certain solutes, even at low concentrations, have the property of lowering the surface tension or other interfacial properties of their solvents. Such solutes are known as surfactants or surface-active agents. They are found in soaps, detergents, emulsifiers, wetting agents, and penetrants, with the most common use, by far, being in synthetic detergents where they may account for 20% - 40% of the product in active form alone. The specific surfactant most widely used until recently is the group of alkyl benzene sulfonates (ABS), which persist in sewage and streams in biologically active solution without appreciable decomposition from either treatment processes or natural purification, being largely immune to biological degradation. Today, the more biodegradable linear alkyl sulfonate (LAS) has essentially replaced ABS on the surfactant market so that measurable surface-active agents will probably be LAS type materials. In addition to foaming problems, anionic surfactants may enhance slime growth, inhibit the growth of nitrifying bacteria, delay gas exchange with the atmosphere, and interfere with the uptake of oxygen. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 250 mL

Measurement: Anionic-type surfactants react with methylene blue dye in aqueous solution to form a blue colored salt which is extractable with chloroform, its color intensity being proportional to the concentration of MBAS. The more complicated, time consuming, and expensive tests for specific substances (e.g., LAS) are not usually warranted. The method is recommended for determination in drinking waters, surface waters, domestic and industrial wastes. It is not applicable to measurement of surfactant-type materials in saline waters. Chlorides at concentration of about 1,000 mg/ℓ show a positive interference, but the degree of interference has not been quantified. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: A sample of filtered river water, spiked with 2.94 mg LAS/ℓ was analyzed in 110 laboratories with a relative standard deviation of 9.1% and a relative error of 1.4%. In similar analyses with a sample of tap water spiked with 0.48 mg LAS/ℓ, relative standard deviations and errors of 9.9% and 1.3% were obtained, and for a sample of distilled water spiked with 0.27 mg LAS/ℓ, the respective values were 14.8% and 10.6%.

Cost of Analysis: \$10 - \$15

MIREX

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Mirex, an organochlorine insecticide, is largely used to control the imported fire ant in the southeastern United States. Crayfish and channel catfish survival is affected by mirex in the water or by ingestion of the bait particles. Bioaccumulation is well established for a wide variety of organisms but the effect of this bioaccumulation on the aquatic ecosystem is unknown. There is evidence that mirex is very persistent in bird tissue. Considering the extreme toxicity and potential for bioaccumulation, every effort should be made to keep mirex bait particles out of water containing aquatic organisms. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: $0.001 \mu\text{g}/\ell$ for freshwater and marine aquatic life.

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50 to 100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for mirex. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to $1 \mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

MOLYBDENUM

Parameter Group: Metals

STORET Unit: $\mu\text{g}/\ell$ as Mo

General: Molybdenum occurs naturally as molybdenum sulfide and lead molybdate. Its chief use is in the production of alloy steels (especially corrosion-resistant stainless steels) where advantage is made of its marked passivity. Other possible sources include mining and ore processing operations, chemical production, some fertilizers, and metallurgical operations. Molybdenum has a relatively low order of toxicity. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 313.3 nm. With the nitrous oxide-acetylene flame, interferences of calcium and other ions may be controlled by adding 1,000,000 $\mu\text{g}/\ell$ of a refractory metal such as aluminum. This should be done to both the samples and standards. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 300 $\mu\text{g}/\ell$; its detection limit is 100 $\mu\text{g}/\ell$. The optimum concentration range is 500-20,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 300, 1,500, and 7,500 $\mu\text{g Mo}/\ell$, the relative standard deviations were 2.3%, 1.3%, and .93%, respectively. Recoveries at these levels were 100%, 96%, and 95%, respectively.

Cost of Analysis: \$10 - \$15

NAPHTHALENE

Parameter Group:

STORET Units:

General: Naphthalene ($C_{10}H_8$) is the most abundant single constituent of coal tar. It is a white solid with the odor of moth balls. It is soluble in water at 20°C to the extent of about 30 mg/ℓ. The use of naphthalene in organic syntheses and dye manufacture is extensive, and hence it may occur in wastes from refineries, coal-tar plants, textile mills, and chemical industries. Lethal concentrations to fish are around 5-20 mg/ℓ. Fish tainting can occur at 1 mg/ℓ. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly. Cool to 4°C.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mL

Measurement: The general procedure involves extraction and measurement with a gas chromatograph. Various cleanup techniques to remove interferences may be required depending upon other constituents in the sample. A skilled chemist or specialist will be required. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits in the 1-10 µg/ℓ range should be achievable. Precision and accuracy data are not available at this time.

Cost of Analysis: \$40 - \$60

NICKEL

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Ni

General: Nickel principally occurs in nature as sulfide. Its main industrial use is in electroplating, alloying, coin making, and in alkaline storage batteries. Other potential sources include silver refineries, basic steel works and foundaries, motor vehicle and aircraft industries, and printing operations. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permit program. It is one of the Consent Decree pollutants.

Criterion: 0.01 of the 96-hour LC_{50} for freshwater and marine aquatic life.

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 232.0 nm. The 352.4 nm wavelength is less susceptible to nonatomic absorbance and may also be used. The calibration curve is more linear at this wavelength; however, there is some loss of sensitivity. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is $150 \mu\text{g}/\ell$; its detection limit is $20 \mu\text{g}/\ell$. The optimum concentration range is 300-10,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 200, 1,000, and 5,000 $\mu\text{g Ni}/\ell$, the standard deviations were ± 0.011 , ± 0.02 , and ± 0.04 , respectively. Recoveries at these levels were 100%, 97%, and 93%, respectively.

Cost of Analysis: \$10 - \$15

NITRILOTRIACETIC ACID (NTA)

Parameter Group: General
Organic

STORET Units: mg/ℓ

General: Nitrilotriacetic acid (NTA) is insoluble in water, but its tribasic salt is quite soluble. NTA has a strong affinity for iron, calcium, magnesium, and zinc, but its relative affinity for toxic metals such as cadmium and mercury is not known, nor have its chelating properties in complex ionic solutions been characterized. It has a potential large-scale use as a substitute for phosphates in detergents. No cases of acute human poisoning by NTA have been reported. It is biodegraded in the natural environment within 4 to 5 days; degradation is accelerated by biological waste treatment.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The zinc-zircon method is often used. In this method, NTA refers to the tri-sodium salt of nitrilotriacetic acid. It is applicable to surface waters in the range of 0.5-10.0 mg/ℓ NTA. Cations, such as calcium, magnesium, zinc, copper, iron, and manganese, complex with NTA and give a negative interference. These ions are removed by batch treating samples with ion-exchange resin. At concentrations higher than expected in typical river waters, only zinc, copper, and iron were not completely removed with ion-exchange treatment.

Precision and Accuracy: In a single laboratory, using spiked surface water samples at concentrations of 0.5, 2, 6, and 10 mg/ℓ NTA, relative standard deviations were 3.4%, 7%, 1.7%, and 1.6%, respectively. In a single laboratory, using spiked surface water samples at concentrations of 1.0 and 7.5 mg/ℓ NTA, recoveries were 120% and 103%, respectively.

Cost of Analysis: \$10 - \$12

NITROBENZENE

Parameter Group:

STORET Units:

General: Nitrobenzene ($C_6H_5NO_2$) is moderately soluble in water.

It is used in the manufacture of analine, soaps, and shoe polishes. Nitrobenzene is an oily liquid and has an almond odor. A concentration of 0.03 mg/l in water will produce a faint odor. The oral LD₅₀ for rabbits is 700 mg/kg of body weight. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: No standard procedures have been developed. The methodology generally requires extraction, concentration, and gas chromatographic analysis. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits of 1 to 10 µg/l should be achievable. Precision and accuracy data are not available at this time.

Cost of Analysis: \$40 - \$60

NITROGEN-AMMONIA

Parameter Group: Nitrogen

STORET Units: mg/ℓ as N

General: Ammonia, one of the chemically interconvertible components of the nitrogen cycle, is naturally present in surface and ground water in concentrations from less than 0.01 to around 0.2 mg/ℓ as N in the absence of pollution. It is produced largely by the deamination of nitrogenous organic matter and the hydrolysis of urea. It may also result from the reduction of nitrate under anaerobic conditions. Other sources include the discharge of industrial wastes from chemical and gas plants, from ice plants, and where it is used in scouring and cleaning operations. There appears little physiological risk in palatable concentrations, the odor threshold being 0.037 mg/ℓ. Because it changes rapidly to nitrites and nitrates, ammonia is actually a fertilizer for most crops; ammonium salts constitute a major source of nitrogen fertilization. The toxicity of ammonia to fish is highly pH dependent. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Add 2 mL of concentrated H_2SO_4 or 40 mg HgCl_2 /ℓ and store at 4°C. The use of mercuric chloride is discouraged whenever possible, however.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 400 mL

Measurement: The distillation procedure is recommended for the determination of ammonia-nitrogen. The method covers the range from about 0.05 to 1.0 mg/ℓ $\text{NH}_3\text{-N}$ /ℓ for the colorimetric procedures, from 1.0 to 25 mg/ℓ for the titrimetric procedure, and from 0.05 to 1,400 mg/ℓ for the electrode method. A number of aromatic and aliphatic amines will cause turbidity upon the addition of Nessler reagent. Cyanate will hydrolyze to some extent. Volatile alkaline compounds may cause an off-color upon Nesslerization. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Precision and accuracy are highly dependent upon concentration, other constituents present, and the finish method. Relative standard deviations may range from around 4% to 40% and relative errors from under 1% to over 15%.

Cost of Analysis: \$10 - \$12

NITROGEN, KJELDAHL

Parameter Group: Nitrogen

STORET Units: mg/l as N

General: Kjeldahl nitrogen is defined as the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(\text{NH}_4)_2\text{SO}_4$ under conditions of digestion. Organic nitrogen includes natural materials such as proteins and peptides, nucleic acids and urea, and numerous synthetic organic substances. The organic nitrogen concentrations of water and wastewater may vary from less than 0.01 mg/l for the former to over 10 mg/l for the latter. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Add 2 ml of concentrated H_2SO_4 to pH <2 or 40 mg HgCl_2/l and store at 4°C.

The use of mercuric chloride is discouraged whenever possible, however.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 500 ml

Measurement: In the Kjeldahl nitrogen determination the sample is heated in the presence of concentrated sulfuric acid, K_2SO_4 , and HgSO_4 and evaporated until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and is treated and made alkaline with a hydroxide-thiosulfate solution. The ammonia is distilled and determined by either the titrimetric method, the Nesslerization method, or the potentiometric method. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Thirty-one analysts in twenty laboratories analyzed natural water samples containing exact increments of organic nitrogen. At the 0.2-0.3 mg/l as N concentration level the relative standard deviation and error were around 90% and 10%, respectively. At around 4 mg/l as N they were about 25% and 1%, respectively.

Cost of Analysis: \$15 - \$20

NITROGEN, NITRATE

Parameter Group: Nitrogen

STORET Units: mg/ℓ as N

General: Nitrate, one of the chemically interconvertible compounds of the nitrogen cycle, occurs in trace quantities in surface water and in small amounts in fresh domestic wastewater. It is seldom abundant, since it serves as an essential nutrient for all types of plants. Some ground water may contain high levels of nitrate (as a result of leachings from cesspools or excess applications of fertilizers, etc.) due to the lack of photosynthetic action. There has been no reporting of physiological harm at concentrations of less than 10 mg/ℓ as N. Nitrates are injurious for certain industrial uses (e.g., fabric dyeing, fermentative processes). High nitrate concentrations stimulate the growth of plankton and aquatic weeds and accelerate eutrophication. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Add 2 mL concentrated H_2SO_4 /ℓ to pH <2 or 40 mg HgCl_2 /ℓ and store at 4°C.

The use of mercuric chloride is discouraged whenever possible, however.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The brucine method is recommended in the range from 0.1 to 2 mg $\text{NO}_3\text{-N}$ /ℓ for determination in drinking, surface, and saline waters, domestic and industrial wastes. Dissolved organic matter will cause an off color. The effect of salinity is eliminated by the addition of sodium chloride to the blanks, standards, and samples. All strong oxidizing or reducing agents interfere. Residual chlorine interference is eliminated by the addition of sodium arsenite. Ferrous and ferric iron and quadrivalent manganese give slight positive interferences. Uneven heating of the samples and standards during the reaction time will result in erratic values. The cadmium reduction method may also be used; see discussion under Nitrogen, Nitrate-Nitrite. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Five synthetic samples containing nitrate and other constituents were analyzed in 50 laboratories at concentrations of 0.05, 0.5, and 5 mg/l as N; relative standard deviations were 66.7%, 14.4%, and 15.4% and relative errors were 7.6%, 0.6%, and 4.5%, respectively.

Cost of Analysis: \$10 - \$12

NITROGEN, NITRATE-NITRITE

Parameter Group: Nitrogen

STORET Units: mg/ℓ as N

General: See discussions under nitrate and nitrite. The combined test is less expensive than making individual determinations and provides a determination of total oxidized nitrogen. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Store at 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The cadmium reduction method is recommended for the determination of nitrite and nitrate combined in drinking, surface, and saline waters, domestic and industrial wastes. Buildup of suspended matter in the reduction column will restrict sample flow. Low results might be obtained for samples that contain high concentrations of iron, copper, or other metals. EDTA is added to the samples to eliminate this interference. Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The applicable range of this method is 0.01 to 1.0 mg/ℓ nitrate-nitrite nitrogen. In a single laboratory, using sewage samples at concentrations of 0.04, 0.24, 0.55, and 1.04 mg NO₃ + NO₂-N/ℓ, the relative standard deviations were 12.5%, 1.6%, .9%, and .9%, respectively, while recoveries were 100%, 102%, and 100%, respectively.

Cost of Analysis: \$10 - \$15

NITROGEN, NITRITE

Parameter Group: Nitrogen

STORET Units: mg/ℓ as N

General: Nitrite, one of the chemically interconvertible compounds of the nitrogen cycle, occurs in the oxidation of ammonia to nitrate and in the reduction of nitrate. This oxidation and reduction may occur in wastewater treatment plants, water distribution systems, and natural waters. In conjunction with ammonia and nitrate, nitrites are often indicative of water pollution. They exhibit the same deleterious effects as nitrates except at lower concentrations, e.g., no physiological harm under 2 mg/ℓ as N. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Store at 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The diazotization method is recommended for the determination of nitrite nitrogen in the range from 0.01 to 1.0 mg NO₂-N/ℓ. The diazonium compound formed by diazotation of sulfanilamide by nitrite in water under acid conditions is coupled with N-(1-naphthyl)-ethylenediamine to produce a reddish-purple color which is read in a spectrophotometer at 540 nm. The presence of strong oxidants or reductants to the samples will affect the nitrite concentrations. High alkalinity (>600 mg/ℓ) will give low results due to a shift in pH. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Precision and accuracy data are not available.

Cost of Analysis: \$5 - \$12

NITROPHENOLS

Parameter Group:

STORET Units:

General: Nitrophenols include 2, 4-dinitrophenol; dinitrocresol, 2-nitrophenol, 4-nitrophenol, and 4, 6-dinitro-o-cresol. Metanitrophenol is highly soluble in cold water, p-nitrophenol moderately so, and o-nitrophenol only sparingly soluble. The ortho isomer is used in chemical manufacturing. Minimum lethal doses to fish vary with isomer, species, and other water constituents (e.g., hard water concentrations may be 10 times greater than those of distilled water). This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: No standard procedures have been developed. The methodology generally requires extraction, concentration, and gas chromatographic analysis. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits of 1 to 10 µg/L should be achievable. Precision and accuracy data are not available at this time.

Cost of Analysis: \$40 - \$60

OIL AND GREASE

Parameter Group: General
Organic

STORET Units: mg/ℓ

General: Oils and grease are not definitive chemical categories but include thousands of organic compounds with varying physical, chemical, and toxicological properties. Grease and oil include hydrocarbons, fatty acids, soaps, fats, waxes, and oils. The three major industrial sources of oily waste are the petroleum industry, metals manufacture and machining, and food processors. Field and laboratory evidence have demonstrated both acute lethal toxicity and long-term sublethal toxicity of oils to aquatic organisms. Bioaccumulation of petroleum products presents two especially important public health problems: (1) the tainting of edible, aquatic species, and (2) the possibility of edible marine organisms incorporating the high boiling, carcinogenic polycyclic aromatics in their tissues. The direct effects of aquatic oil pollution on man are minimal. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria: For domestic water supply: Virtually free from oil and grease, particularly from the tastes and odors that emanate from petroleum products.

For aquatic life:

- 0.01 of the lowest continuous flow 96-hour LC_{50} to several important freshwater and marine species, each having a demonstrated high susceptibility to oils and petrochemicals.
- Levels of oils or petrochemicals in the sediment which cause deleterious effects to the biota should not be allowed.
- Surface waters shall be virtually free from floating non-petroleum oils of vegetable or animal origin, as well as petroleum-derived oils.

Preservation Method: Analyze as soon as possible. If storage is required, cool to 4°C, add H_2SO_4 to pH <2.

Maximum Holding Time: 24 hours

Container Type: Glass

Sample Volume Required: 1,000 mL

Measurement: The Soxhlet extraction method is recommended when relatively polar, heavy petroleum fractions are present. The method is applicable to the determination of relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, and greases. The separatory funnel extraction method can also be used. The infrared method is applicable for measurement of most light petroleum fuels. The Soxhlet extraction and separatory funnel extraction methods are not applicable to the light hydrocarbons that volatilize at temperatures below 70°C. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The three oil and grease methods were tested by a single laboratory on a sewage. The Soxhlet extraction method determined the oil and grease level in the sewage to be 14.8 mg/ℓ. When 1-liter portions of the sewage were dosed with 14.0 mg of a mixture of #2 fuel oil and Wesson oil, the recovery was 88% with a standard deviation of 1.1 mg. The separatory funnel extraction method determined the oil and grease level in the sewage to be 12.6 mg/ℓ. When 1-liter portions of the sewage were dosed with 14.0 mg of a mixture of #2 fuel oil and Wesson oil, the recovery was 93% with a standard deviation of 0.9 mg. The infrared method determined the oil and grease level in the sewage to be 17.5 mg/ℓ. When 1-liter portions of the sewage were dosed with 14.0 mg of a mixture of #2 fuel oil and Wesson oil, the recovery was 99% with a standard deviation of 1.4 mg.

Cost of Analysis: \$15 - \$30

ORGANIC CARBON

Parameter Group: General
Organic

STORET Units: mg/l as C

General: Organic carbon is the carbon oxidized by dichromate or other strong oxidizing agents, the most common measurement being total organic carbon (TOC). As in the case of BOD, TOC is a measure of a significant aspect of the strength of a discharge but is not a pollutant per se. The value of TOC usually falls below the true concentration of organic contaminants because other constituent elements are excluded. TOC is a more direct expression of the organic chemical content of water than either the BOD or COD tests and is faster and more convenient. It is often used, after an empirical relationship has been established, to estimate BOD or COD. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C. Add H_2SO_4 to pH <2.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass (brown glass preferred)

Sample Volume Required: 25 mL

Measurement: The combustion-infrared method is recommended. The method is applicable to measurement of organic carbon above 1 mg/l. Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be removed or accounted for in the final calculation. Instrument manufacturer's directions must be followed. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The difficulty of sampling particulates limits the precision to approximately 5 to 10% or higher. On clear or filtered samples, the precision may approach 1 to 2%. A distilled water solution containing 107 mg/l of oxidizable organic compounds was analyzed by 28 analysts in 21 laboratories with a relative standard deviation of 7.6% and a relative error of 1.01%.

Cost of Analysis: \$12 - \$15

PARATHION

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Parathion is the organophosphorus insecticide $\text{C}_{10}\text{H}_{14}\text{O}_5\text{NPS}$.

It is a yellow liquid that is insoluble in water or kerosene but freely soluble in alcohols and aromatic hydrocarbons. It is most commonly applied to row and orchard crops. Few chronic exposure data are available for aquatic organisms. At high concentrations of parathion, deformities, tremors, convulsions, hypersensitivity, hemorrhages were evident in bullheads. Inhibition of cholinesterase enzymes is the established mode of physiological action of parathion. Parathion has been found acutely toxic to aquatic invertebrates. The half-life of parathion in river water (pH 7.3-8.0) is one week. The estimated fatal dose for a 70-kg man is 0.1 gram. The acute oral LC_{50} for rats ranges from 3,000 to 15,000 $\mu\text{g}/\text{kg}$ of body weight. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 0.04 $\mu\text{g}/\ell$ for freshwater and marine aquatic life.

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100 mL or more

Measurement: The use of co-solvent extraction, column chromatography and liquid-liquid partition, and detection and measurement accomplished by flame photometric gas chromatography using a phosphorus specific filter is recommended for parathion. Great care must be exercised in the selection and use of methods to minimize interferences, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but is usually 0.010 $\mu\text{g}/\ell$ or higher. Sensitivity is typically 1 $\mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$.30 - \$150, depending upon preparation required.

PCNB

Parameter Group:

STORET Units:

General: Pentachloronitrobenzene (PCNB) is an organochlorine insecticide. It is used as an agricultural fungicide. It is soluble in carbon disulfide, benzene, and chloroform. It is practically insoluble in water and cold alcohol.

Criterion: Not established

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for PCNB. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person).

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 µg/L range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required

PENTACHLOROPHENOL

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Pentachlorophenol ($\text{C}_6\text{HCl}_5\text{O}$) is a crystalline material only slightly soluble in water but freely soluble in alcohol, ether, and benzene. However, its sodium salt is highly soluble in water. Pentachlorophenol possesses bactericidal, herbicidal, insecticidal, fungicidal, and molluscicidal properties. In concentrated doses, it causes lung, liver, and kidney damage to humans. In sea water, a concentration of $1.0 \text{ mg}/\ell$ of sodium pentachlorophenol prevents the attachment of marine fouling organisms in pipe and conduit. Its toxicity is highly dependent upon the vehicle in which it is administered. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Acidify to a pH of 4 with H_3PO_4 . Add $1.0 \text{ g CuSO}_4 \cdot 5\text{H}_2\text{O}/\ell$ to inhibit biodegradation of phenols. Cool to 4°C .

Maximum Holding Time: 24 hours

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mg/ℓ or more depending upon initial concentration.

Measurement: The recommended method involves direct aqueous injection for the gas-liquid chromatographic determination of concentrates containing more than $1 \text{ mg}/\ell$ phenolic compounds. A flame-ionization detector is used for their individual measurement. Suspended matter may interfere by plugging the microsyringe. Interfering nonphenolic organic compounds may be removed by distillation. Steps should be taken to minimize or eliminate ghosting. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Few precision and accuracy data are available. Precision is very operator dependent. For example, total precision may be 2 to 5 times single operator precision values.

Cost of Analysis: \$40 - \$60

pH

Parameter Group: Physical

STORET Units: Standard Units

General: The pH of a solution is expressed as the logarithm of the reciprocal of the hydrogen ion activity in moles per liter at a given temperature. The practical scale extends from 0 (very acidic) to 14 (very alkaline) with 7 corresponding to exact neutrality at 25°C. Whereas alkalinity and acidity are measures of the total resistance to pH change or buffering capacity of a sample, pH represents the free hydrogen ion activity not bound by carbonate or other bases. The pH of most natural waters falls in the range of 4 to 9 with the majority being slightly basic. Changes in the normal pH for a given water may indicate the discharge of alkaline or acidic wastes. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria:

- 5-9, domestic water supplies (welfare).
- 6.5 - 9.0, freshwater aquatic life.
- 6.5 - 8.5, marine aquatic life (within this range, pH should not be more than 0.2 unit outside the normally occurring range).

Preservation Methods: Analyze on site if at all possible. Otherwise, seal the sample container and cool to 4°C. Sample bottle should not be opened before analysis.

Maximum Holding Time: Any holding time beyond 6 hours should be reported with the measurement.

Container Type: Plastic or glass

Sample Volume Required: 25 to 100 mL

Measurement: Although pH can be measured colorimetrically, the method suffers from numerous interferences, deterioration of indicators and color standards, and limited indicator range. The glass electrode method is the standard technique, employing either a glass electrode in conjunction with a separate reference (constant potential) electrode, e.g., calomel, silver-silver chloride, or a combination electrode (glass and reference). The measurement is temperature-dependent. Oil and grease may coat the pH electrode and cause a sluggish response. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The precision and accuracy attainable will depend upon the type and condition of the water and the care used in standardization and operation. Precisions of ± 0.02 pH and accuracies of ± 0.05 pH are achievable, but ± 0.1 pH represents the accuracy limit under normal conditions. Typical standard deviations are from 0.1 to 0.2 pH.

Cost of Analysis: \$3

PHENOLICS

Parameter Group: General
Organic

STORET Units: $\mu\text{g}/\ell$

General: Phenols are hydroxy derivatives of benzene and its condensed nuclei. Phenolic compounds include a wide variety of organic chemicals and may arise from the distillation of coal and wood; from oil refineries; chemical plants; livestock dips; human and other organic wastes; hydrolysis, chemical oxidation, and microbial degradation of pesticides; and from naturally occurring sources and substances. Despite the fact that it is used as a bactericide, weak phenol solutions are decomposed by bacteria and biological action, rates typically exceeding $2,000 \mu\text{g}/\ell$ per day in natural streams. Chlorination of water containing phenolic compounds produces odoriferous and objectionable tasting chlorophenols. The ingestion of concentrated solutions of phenol will result in severe pain, renal irritation, shock, and possibly death. A 1.5-gram dose may be fatal. Many of the phenolic compounds are more toxic than pure phenol, especially to lower life forms. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: $1 \mu\text{g}/\ell$ for domestic water supply (welfare), and to protect against fish flesh tainting.

Preservation Method: Analyze as soon as possible. Cool to 4°C . Add H_3PO_4 to $\text{pH} < 4$ and $1.0\text{g CuSO}_4/\ell$.

Maximum Holding Time: 24 hours

Container Type: Glass only

Sample Volume Required: 500 mL

Measurement: The 4-aminoantipyrine (4-AAP) method with distillation is recommended and is applicable to the analysis of drinking, surface, and saline waters, domestic and industrial wastes. Phenolic materials react with 4-aminoantipyrine in the presence of potassium ferricyanide at a pH of 10 to form a stable reddish-brown colored antipyrine dye. The amount of color produced is a function of the concentration of the phenolic material. For most samples, a preliminary distillation is required to remove interfering materials. Gas chromatograph tests can be used to isolate specific compounds. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Using the extraction procedure for concentration of color, six laboratories analyzed samples at concentrations of 9.6, 48.3, and 93.5 $\mu\text{g}/\ell$. Relative standard deviations were 10.3%, 6.4%, and 4.5%, respectively. The method must be regarded as an approximation representing the minimum amount of phenols present because the phenolic value varies with the types of phenols within a given sample. It is therefore impossible to express the accuracy of the method.

Cost of Analysis: \$15 - \$25.

PHOSPHORUS (ALL FORMS)

Parameter Group: Phosphorus

STORET Units: mg/l P

General: Phosphorus in its elemental form (yellow phosphorus) does not occur free in nature and is particularly toxic to animal life, being subject to bioaccumulation in much the same way as mercury. Phosphorus as phosphate is abundant in nature and also from the activities of man. Phosphates occur as a result of leaching from minerals and ores in natural processes of degradation, from agricultural drainage as one of the stabilized products of decomposition of organic matter, as a result of innumerable industrial discharges, from some treated cooling waters, and as a major element of municipal sewage. It is an essential nutrient for plant and animal growth. Major uses include fertilizers, detergents, and industrial chemicals. Organic phosphates are used extensively in pesticides. The chief deleterious effect of high concentrations is accelerated eutrophication. They also interfere with coagulation and removal of turbidity. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. Definitions of the various phosphorus forms are given below.

Total Phosphorus - all of the phosphorus present in the sample, regardless of form, as measured by the persulfate digestion procedure.

Total Orthophosphate - inorganic phosphorus in the sample as measured by the direct colorimetric analysis procedure.

Total Hydrolyzable Phosphorus - phosphorus in the sample as measured by the sulfuric acid hydrolysis procedure, and minus pre-determined orthophosphates. This hydrolyzable phosphorus includes polyphosphorus plus some organic phosphorus.

Total Organic Phosphorus - phosphorus (inorganic + oxidizable organic) in the sample measured by the persulfate digestion procedure, and minus hydrolyzable phosphorus and orthophosphate.

Dissolved Phosphorus - all of the phosphorus present in the filtrate of a sample filtered through a phosphorus-free filter of 0.45 micron pore size and measured by the persulfate digestion procedure.

Dissolved Orthophosphate - as measured by the direct colorimetric analysis procedure.

Dissolved Hydrolyzable Phosphorus - as measured by the sulfuric acid hydrolysis procedure and minus pre-determined dissolved orthophosphates.

Dissolved Organic Phosphorus - as measured by the persulfate digestion procedure, and minus dissolved hydrolyzable phosphorus and orthophosphate.

When sufficient amounts of phosphorus are present in the sample to warrant such consideration, the insoluble forms may be calculated as the total minus the dissolved fraction and reported as Insoluble Phosphorus, Insoluble Orthophosphate, Insoluble Hydrolyzable Phosphorus, or Insoluble Organic Phosphorus.

Criterion: 0.10 µg/ℓ yellow (elemental) phosphorus for marine or estuarine waters.

Preservation Method: Filter on site if dissolved determination is desired. Cool to 4°C. Analyze as soon as possible. Add 40 mg/ℓ mercuric chloride as a preservative if absolutely necessary, but its use is discouraged whenever possible.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The persulfate digestion method is recommended. After digestion, determine the total orthophosphate in the sample by the direct colorimetric analysis procedure. High iron concentrations can cause precipitation of and subsequent loss of phosphorus. Mercury chloride interferes when the chloride level of the sample is low, <50 mg Cl/ℓ. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Natural water samples with an exact increment of organic phosphate were analyzed by 33 analysts in 19 laboratories. At around 0.1 and 0.8 mg/ℓ P, relative standard deviations were around 35% and 15%, respectively, and relative error ranges were 3-12% and 1-3%, respectively. Natural water samples with an exact increment of orthophosphate were analyzed by 26 analysts in 16 laboratories. At around 0.01 and 0.02 mg/ℓ P, relative standard deviations were around 28% and 6% respectively, and relative errors were around 5.5% and 2.3%, respectively.

Cost of Analysis: \$10 - \$15

PHTHALATE ESTERS

Parameter Group: General
Organic

STORET Units: $\mu\text{g}/\ell$

General: Phthalate esters include bis (2-ethylhexyl) phthalate, butyl benzyl phthalate, di-n-butyl phthalate, diethyl phthalate, and dimethyl phthalate and are organic compounds used as plasticizers, particularly in polyvinyl chloride plastics. The di-2-ethylhexyl and di-n-butyl phthalates are used as an orchard acaricide and insect repellent. Phthalate esters can be detrimental to aquatic organisms at low water concentrations. Ability to concentrate high levels from water and reproductive impairment in certain species are suggestive of potential environmental damage. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: 3 $\mu\text{g}/\ell$ for freshwater aquatic life.

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: No standard procedures have been developed. The methodology generally requires extraction, concentration, and gas chromatographic analysis. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits of 0.1 to 10 $\mu\text{g}/\ell$ should be achievable. Precision and accuracy data are not available at this time.

Cost of Analysis: \$25 - \$40

POLYCHLORINATED BIPHENYLS

Parameter Group: General
Organic

STORET Units: $\mu\text{g}/\ell$

General: Polychlorinated biphenyls (PCB's) are a class of compounds produced by the chlorination of biphenyls and are registered in the United States under the trade name Aroclor^(R). PCB compounds are slightly soluble in water (25 to 200 $\mu\text{g}/\ell$), soluble in lipids, oils, and organic solvents, and resistant to both heat and biological degradation. PCB's are relatively nonflammable, have useful exchange and dielectric properties, and were used principally in the electrical industry in capacitors and transformers. The acute and chronic effects of PCB's have been determined on a number of aquatic organisms. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criterion:

- .001 $\mu\text{g}/\ell$ for freshwater and marine aquatic life and for consumers thereof
- Every reasonable effort should be made to minimize human exposure.

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mL

Measurement: The recommended gas chromatograph method covers the following PCB mixtures: Aroclors 1221, 1232, 1242, 1248, 1254, 1260, and 1016. It is an extension of the method for organochlorine pesticides - both the PCB's and the organochlorine pesticides may be determined on the same sample. They are co-extracted by liquid-liquid extraction and separated from one another prior to gas chromatographic determination. A combination of the standard Florisil column cleanup procedure and a silica gel microcolumn separation procedure are employed. Identification is made from gas chromatographic patterns obtained through the use of two or more unlike columns. Detection and measurement is accomplished using an electron capture, microcoulometric, or electrolytic conductivity

detector. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Analysis: The detection limit is approximately 1 µg/ℓ. Precision and accuracy data are not available at this time.

Cost of Analysis: \$45 to \$50 for a scan and one compound
\$95 to \$100 for total PCB's

POLYNUCLEAR AROMATIC HYDROCARBONS

Parameter Group:

STORET Units:

General: Polynuclear aromatic hydrocarbons include 1, 2-benzanthracene, 3, 4-benzopyrene, 3, 4-benzofluoranthene, 11, 12-benzofluoranthene, chrysene, acenaphthylene, anthracene, 1, 12-benzoperylene, fluoroethane, phenanthrene, 1, 2:5, 6-dibenanthracene, indeno (1, 2, 3-C, D) pyrene, and pyrene. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly. Cool to 4°C.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 1,000-4,000 mL

Measurement: Procedures for determination of polynuclear aromatic hydrocarbons involve extraction, thin layer chromatography, and fluorescence or UV absorption spectra. They require confirmation on wastewater. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits range from around 0.2 to 20 µg/L. Precision and accuracy data are not available at this time.

Cost of Analysis: \$300 - \$600

POTASSIUM

Parameter Group: Metals

STORET Units: mg/ℓ as K

General: Potassium occurs in nature as chloride or sulfate in certain salt deposits, in common rocks (average of the solid earth shell is 2.6%) and minerals (e.g., feldspar, greensand, alunite, leucite), and is present in vegetation. It is one of the most active metals and, hence, is only found in the ionized or molecular form. Its salts are indispensable for fertilizers, some varieties of glass, and certain other purposes. All are highly soluble and uses include baking powders, effervescent antacids, as a flux for silver solders, treating coal to prevent slag formation, tanning, soap manufacturing, in matches and explosives, pesticides, in wood industries, dyeing and bleaching cotton, paint and varnish removers, electroplating, photoengraving, lithography, photographic emulsions, table salt, disinfectants, and a host of other uses. Potassium is an essential nutritional element, but in excessive doses it acts as a cathartic. Its level of toxicity to fish and other aquatic life depends upon its form and the age and species involved. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or Pyrex

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 766.5 nm. Sodium may interfere if present at much higher levels than the potassium. This effect can be compensated by approximately matching the sodium content of the potassium standards with that of the sample. The flame photometric method is rapid, sensitive, and accurate but requires a special instrument and much preliminary work before samples can be run routinely. The colorimetric method is usually inadvisable for potassium levels below 10 mg/ℓ. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 0.04 mg/ℓ; its detection limit is 0.005 mg/ℓ. The optimum concentration range is 0.1-2 mg/ℓ. In a single laboratory, using distilled

water samples at concentrations of 1.6 and 6.3 mg/ℓ, the relative standard deviations were 13% and 8%, respectively. Recoveries at these levels were 103% and 102%. In a 33-laboratory test using a synthetic unknown at 3.1 mg/ℓ K, results from the flame photometric method yielded a relative standard deviation of 15.5% and a relative error of 2.3%.

Cost of Analysis: \$5 - \$10

RADIOACTIVITY (ALPHA AND BETA)

Parameter Group: Radiological

STORET Units: Unspecified

General: Naturally occurring radioactivity in water is due to contact with mineral deposits; many springs and deep wells have high levels of radioactivity. Uranium, thorium, and radium and their long series of daughter products are the chief naturally occurring emitters of alpha and beta radiation. With the advent of nuclear science, man has produced a long series of radioactive products, including almost all of the elements in the periodic table. In addition to mining and separation operations, other manmade sources include the manufacture of nuclear weapons, nuclear reactors, the production of isotopes, and their use in medical therapy, research, and industrial processes and instrumentation. Radioactivity may be considered as an indestructable property from the viewpoint of man's inability to cancel or neutralize it by chemical or physical means. Gross alpha and beta activity measurements represent the best overall indicator of the presence of radioactive contamination in waters and the need for more specific determinations of the more hazardous radionuclides. The radioactivity of natural waters is usually in the 1 to 1,000 pCi/l range but may reach 100,000 pCi/l, and the radon (short-lived) content of some mineral springs has been found to be as high as 750,000 pCi/l. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Preservatives may alter the distribution of radioactivity in a sample and should not be used until the sample is separated into suspended and dissolved fractions.

Maximum Holding Time: Unstated. Adsorption onto container surfaces represents the greatest problem.

Container Type: Plastic or glass

Sample Volume Required: 1,000 mL

Measurement: The internal proportional counter is the recommended instrument for counting gross beta radioactivity. With a Geiger counter, the alpha activity cannot be determined separately. Alpha counting efficiency in end-window counters may be very low. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: In a study of two sets of paired water samples containing known additions of radionuclides, 15 laboratories determined the gross alpha activity and 16 analyzed the gross beta activity. The average recoveries of added gross alpha activity were 86%, 87%, 84%, and 82%. The average recoveries of added gross beta activity were 99%, 100%, 100%, and 100%.

Cost of Analysis: \$9 - \$15

RADIUM

Parameter Group:

STORET Units:

General: There are four naturally occurring radium isotopes: radium 223, radium 224, radium 226, and radium 228. Radium 226 has a half life of 1600 years. Ra-228 is a beta emitter; the others are alpha emitters. Although alpha particles cannot penetrate the skin, they are particularly dangerous when ingested and deposited within the body. The determination of radium by precipitation is a screening technique applicable in particular to drinking water. It includes all alpha emitting isotopes, and as long as concentrations are within standards for Ra-226, the need for examination by a more specific method is minimal. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: None

Maximum Holding Time: Unknown, but prompt analysis is recommended.

Container Type: Plastic or glass

Sample Volume Required: 1,000 mL

Measurement: The recommended method is the determination of radium by precipitation. It involves the alpha counting of a barium-radium sulfate precipitate that has been isolated from the sample and purified. The method is also applicable to sewage and industrial wastes, provided that steps are taken to destroy organic matter and eliminate other interfering ions. A counting instrument is required. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: In a 20-laboratory study involving the analysis of four samples for total radium, all four results from two laboratories and two results from a third had to be rejected as outliers. Of the remainder, recoveries averaged higher than 95%. At the 95% confidence level, the precision was around 30%.

Cost of Analysis: \$40 - \$50

RESIDUE, SETTLEABLE

Parameter Group: Solids

STORET Units: mL/L

General: Settleable residue (solids) which blankets the bottom of water bodies damage the invertebrate populations, block gravel spawning beds, and if organic, remove dissolved oxygen from overlying waters. They can interfere with recreation, navigation, fish and shellfish production, and destroy aesthetic values of water. They may decompose to produce putrefactive odors and may exude products of decomposition to overlying waters. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 1000 mL

Measurement: Settleable matter is measured volumetrically with an Imhoff cone. The practical lower limit of the determination is about 1 mL/L/hr. For some samples, a separation of settleable and floating materials will occur. In such cases, the floating materials are not measured. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Precision and accuracy data are not available at this time.

Cost of Analysis: \$3 - \$5

RESIDUE, TOTAL

Parameter Group: Solids

STORET Units: mg/ℓ

General: Total residue (total solids) refers to all the solid matter (suspended and dissolved) in water or wastewater and is the material left in a vessel after evaporation of a sample and its subsequent drying in an oven. Thus it is the sum of filterable and nonfilterable residue. Waters with high residue are generally of inferior palatability and may induce adverse reaction to transient consumers. Also see discussions of filterable and nonfilterable residue. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 7 days

Container Type: Plastic or resistant glass

Sample Volume Required: 100 mL

Measurement: A well mixed aliquot of the test sample is quantitatively transferred to a pre-weighed evaporating dish and evaporated to dryness at 103-105°C. The practical range of the determination is from 10 mg/ℓ to 20,000 mg/ℓ. Large, floating particles or submerged agglomerates (non-homogeneous materials) should be excluded from the test sample. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The practical upper limit for this determination is 20,000 mg/ℓ. Precision and accuracy data are not available at this time.

Cost of Analysis: \$3 - \$15

RESIDUE, TOTAL FILTERABLE

Parameter Group: Solids

STORET Units: mg/ℓ

General: Total filterable residue (total dissolved solids) consists of inorganic salts, small amounts of organic matter, and dissolved materials. The principal inorganic anions dissolved in water include the carbonates, chlorides, sulfates and nitrates (principally in ground waters); the principal cations are sodium, potassium, calcium, and magnesium. Excess dissolved solids are objectionable in drinking water because of possible physiological effects, unpalatable mineral tastes, and higher costs because of corrosion or the necessity for additional treatment. Some communities use water containing up to 4,000 mg/ℓ for drinking purposes. There is no proof of beneficial or therapeutic value to mineral waters. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 250 mg/ℓ for chlorides and sulfates in domestic water supplies (welfare).

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 7 days

Container Type: Plastic or resistant glass

Sample Volume Required: 100 mL

Measurement: The recommended method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. A well mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C. Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing. Samples containing high concentrations of bicarbonate will require careful and prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate. Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The practical range of the determination is 10 mg/ℓ to 20,000 mg/ℓ. Precision and accuracy data are not available at this time.

Cost of Analysis: \$3 - \$15

RESIDUE, TOTAL NONFILTERABLE

Parameter Group: Solids

STORET Units: mg/ℓ

General: Total nonfilterable residue (suspended solids) is the material retained on a standard glass fiber filter disk after filtration of a well mixed sample. In natural waters it consists of erosion silt, organic detritus, and plankton. The discharge of wastewater presents virtually unlimited possibilities. Total nonfilterable residue includes all settleable solids. It has varying effects upon water uses (apart from individual effects of the substances constituting the suspended solids). It is the most difficult parameter in terms of obtaining a representative sample from the bulk source and is used as a measure of treatment plant efficiency. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Should not reduce the depth of the compensation point for photosynthetic activity by more than 10% from the established norm - for aquatic life.

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 7 days

Container Type: Plastic or resistant glass

Sample Volume Required: 100 mℓ

Measurement: The recommended method involves filtering a well mixed sample through a standard glass fiber filter, and the residue retained on the filter is dried to constant weight at 103-105°C. Too much residue on the filter will entrap water and may require prolonged drying. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The practical range of the determination is 10 mg/ℓ to 20,000 mg/ℓ. Precision and accuracy data are not available at this time.

Cost of Analysis: \$3 - \$15

RESIDUE, VOLATILE

Parameter Group: Solids

STORET Units: mg/ℓ

General: The volatile components in the residue represent a rough indication of the amount of organic matter present. Since the result may reflect loss of water of crystallization, loss of volatile organic matter before combustion, incomplete oxidation of certain complex organics, and decomposition of mineral salts during combustion, it may not yield an accurate measure of organic carbon. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 7 days

Container Type: Plastic or resistant glass

Sample Volume Required: 100 mL

Measurement: The recommended method determines the weight of solid material combustible at 550°C. The residue obtained from the determination of total, filterable, or nonfilterable residue is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as mg/ℓ volatile residue. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: A collaborative study involving three laboratories examining four samples by means of ten replicates showed a standard deviation of ±11 mg/ℓ at 170 mg/ℓ volatile residue concentration.

Cost of Analysis: \$10 - \$15

SELENIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Se

General: Selenium appears in the soil as basic ferric selenite, calcium selenate, and as elemental selenium. Selenium salts are used in many industries, including paint, pigment and dye producers, electronics, glass manufacture, insecticide sprays, electrical apparatus (rectifiers, semiconductors, photoelectric cells, etc.), rubber, and alloying. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 10 $\mu\text{g}/\ell$ for domestic water supply (health)
- For marine and freshwater aquatic life; 0.01 of the 96-hour LC_{50} as determined through bioassay using a sensitive resident species

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The AA spectrophotometric gaseous hydride method is recommended using a wavelength of 196.0 nm. The method is applicable to most fresh and saline waters, in the absence of high concentrations of chromium, cobalt, copper, mercury, molybdenum, nickel and silver. The diaminobenzidine colorimetric method may also be used. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is approximately 2.5 $\mu\text{g}/\ell$; its detection limit is 2 $\mu\text{g}/\ell$. The working range of the method is 2-20 $\mu\text{g}/\ell$. At a concentration of 10 $\mu\text{g}/\ell$, the relative standard deviation is 11% and the relative error is 0.0%. Ten replicate solutions of selenium oxide at the 5, 10 and 15 $\mu\text{g}/\ell$ level were analyzed by a single laboratory. Relative standard deviations were 12%, 11%, and 19% with recoveries of 100%, 100%, and 101%.

Cost of Analysis: \$15 - \$40

SILICA

Parameter Group: Solids

STORET Units: mg/l as SiO₂

General: Silica is abundant in the earth's crust. It appears as an oxide in many rocks. The degradation of the rocks results in the presence of silica in natural waters. Silica is also widely used in industry and in water treatment. Silica in water forms silica and silicate scales in various equipments, particularly on high pressure steam turbine blades. In normally occurring concentrations it does not appear to cause adverse physiological effects. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 7 days

Container Type: Plastic or hard rubber

Sample Volume Required: 50-1000 mL

Measurement: Total silica is determined by a gravimetric method wherein silica acids are formed and precipitated, ignited, and the final determination made as the loss on volatilization. The recommended method for dissolved silica involves filtering a well-mixed sample through a 0.45μ membrane filter. The filtrate, upon the addition of molybdate ion in acidic solution, forms a greenish-yellow color complex proportional to the dissolved silica in the sample. The color complex is then measured spectrophotometrically. Excessive color and/or turbidity interfere. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Precision of the gravimetric method is approximately ±0.2 mg SiO₂. Photometric evaluations by the aminonaphthal-sulfuric acid procedure have an estimated precision of ±0.10 mg/l in the range from 0 to 2 mg/l. Photometric evaluations of the silica-molybdate color in the range from 2 to 50 mg/l have an estimated precision of approximately 4% of the quantity of silica measured.

Cost of Analysis: \$5 - \$15

SILICON

Parameter Group: Solids

STORET Units: $\mu\text{g}/\ell$ as Si

General: Silicon, the second most abundant element making up 26% of the earth's crust, is not found free in nature but occurs chiefly as the oxide (silica) in sand, quartz, agate, opal, etc., and as silicates in granite, feldspar, kaolinite, and other minerals. Silicon is one of man's most useful elements, with applications ranging from metallurgy to solid state electronics and the production of silicones, polymeric products ranging from liquids to hard glass-like solids with many beneficial properties. Silicon is also important in plant and animal life.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is often used, with a wavelength of 251.6 nm. Avoid any prolonged contact with glass.

Precision and Accuracy: The AA method sensitivity is $2,000 \mu\text{g}/\ell$; its detection limit is $300 \mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$10 - \$20

SILVER

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Ag

General: Silver ions cannot be expected to occur in significant concentrations in natural waters. As a solid metal, silver is used in the jewelry, silverware, metal alloy, and food processing industries. The solid metal produces very little soluble waste. Silver nitrate, which is soluble, is used in the porcelain, photographic, electroplating and ink manufacturing industries, and as an antiseptic. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criteria:

- 50 $\mu\text{g}/\ell$ for domestic water supply (health)
- For marine and freshwater aquatic life, 0.01 of the 96-hour LC_{50} as determined through bioassay using a sensitive resident species

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 328.1 nm. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 60 $\mu\text{g}/\ell$; its detection limit is 10 $\mu\text{g}/\ell$. The optimum concentration range is 100-4,000 $\mu\text{g}/\ell$. At a concentration of 550 $\mu\text{g}/\ell$, the relative standard deviation is 17.5%, and the relative error is 10.6%.

Cost of Analysis: \$10 - \$15

SILVEX (2, 4, 5-TP)

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Silvex, 2 (2, 4, 5-trichlorophenoxy) propionic acid, is a chlorinated phenoxy acid herbicide. It is used for weed control on land, and its esters and salts have been used as an aquatic herbicide in lakes, streams, and irrigation canals. It is slightly soluble in water and freely soluble in acetone and methyl alcohol. Silvex is reported to be slightly less toxic than 2, 4-D and 2, 4, 5-T type materials. Its acute oral LD_{50} to rats is 650,000 mg/kg of body weight. At a level of 2,000 $\mu\text{g}/\ell$ it has temporarily (e.g., 2 weeks) reduced the number of plankton in lakes, but fish are unaffected. Apparently the threshold of toxicity for fish is around 5,000 $\mu\text{g}/\ell$. In some instances, however, fish have acquired an unpleasant, oily taste following exposure to Silvex. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: 10 $\mu\text{g}/\ell$ for domestic water supply (health).

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100 - 1,000 mL, depending on measurement method used.

Measurement: In the recommended method, chlorinated phenoxy acids and their esters are extracted from the acidified water sample with ethyl ether. The esters are hydrolyzed to acids, and extraneous organic material is removed by a solvent wash. The acids are converted to methyl esters which are extracted from the aqueous phase. The extract is cleaned up by passing it through a micro-adsorption column. Detection and measurement are accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography. Interferences may be high and varied and often pose great difficulty in obtaining accurate and precise measurement of chlorinated phenoxy acid herbicides. Organic acids, especially chlorinated acids, cause the most direct interference with the determination. Phenols including chlorophenols will also interfere with this procedure. The method is recommended for use only by an experienced pesticide analyst (or under the close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is 1 $\mu\text{g}/\ell$. Detection limits of 0.01 $\mu\text{g}/\ell$ or so may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: \$45 - \$150, depending upon preparation required.

SODIUM

Parameter Group: Metals

STORET Units: mg/ℓ as Na

General: Sodium is present in most natural waters and, as the cation of many salts used in industry, is one of the most common ions in industrial waters. A high sodium ratio has harmed soil permeability. Humans with certain diseases (cardiac, renal, and circulatory) require water with a low sodium concentration. Otherwise, taste considerations prevail as far as human ingestion is concerned. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Add HNO_3 to pH of 2.

Maximum Holding Time: 6 months

Container Type: Polyethylene bottles

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 589.6 nm. Low-temperature flames increase sensitivity by reducing the extent of ionization of this easily ionized metal. Ionization may also be controlled by adding potassium (1,000 mg/ℓ) to both standards and samples. The flame photometric method may also be used. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 0.015 mg/ℓ; its detection limit is 0.002 mg/ℓ. The optimum concentration range is 0.03-1.0 mg/ℓ. In a single laboratory, using distilled water samples at levels of 8.2 and 52 mg/ℓ, the relative standard deviations were 1.2% and 1.5%, respectively. Recoveries at these levels were 102% and 100%. In a 35-laboratory test using the flame photometric method on a synthetic unknown at 19.9 mg/ℓ Na, a relative standard deviation of 17.3% and a relative error of 4.0% were reported.

Cost of Analysis: \$5 - \$10

SPECIFIC CONDUCTANCE

Parameter Group: Physical

STORET Units: $\mu\text{mhos/cm}$ @ 25°C

General: The determination of conductivity (specific electrical conductance) is a quick method for determining the ion concentration of water. The mobility of each of the various ions, their valences, and their actual and relative concentrations affect conductivity. The specific conductance of potable waters generally ranges from 50 to 1,500 $\mu\text{mhos/cm}$; for wastewaters it is highly variable and may be well in excess of 10,000 $\mu\text{mhos/cm}$. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The specific conductance of a sample is measured by use of a self-contained conductivity meter, Wheatstone bridge-type or equivalent. Samples are preferably analyzed at 25°C. If not, temperature corrections are made and results reported at 25°C. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Typically, relative standard deviations of around 7 to 9% and relative errors from 2 to 5% are experienced.

Cost of Analysis: \$3 - \$5

STRONTIUM

Parameter Group: Radiological STORET Units: Unspecified

General: The radioactive nuclides of strontium produced in nuclear fission are Sr-89 and Sr-90. Strontium 90 is one of the most hazardous of all fission products. It has a half-life of 28 years. Strontium is concentrated in the bones if it is ingested. Ten percent of the occupational maximum concentration for Sr-90 in water is 100 pCi/l. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established.

Preservation Method: None

Maximum Holding Time: Unknown, but prompt analysis is recommended.

Container Type: Plastic or glass

Sample Volume Required: Not determined

Measurement: The recommended method involves the use of a "carrier" which is inactive strontium ions in the form of strontium nitrate. Precipitation is used to obtain strontium carbonate from the strontium carrier and the radionuclide of strontium. It is dried to determine recovery of the carrier and then measured for radioactivity. Radioactive barium interferes in the determination of radioactive strontium. A counting instrument is required. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: In a study of two sets of paired water samples containing known additions of radionuclides, 12 laboratories determined the total radiostrontium and 10 laboratories determined Sr-90. The average recoveries of total radiostrontium from the four samples were 99%, 99%, 96%, and 93%. The average recoveries of added Sr-90 from the four samples were 90%, 96%, 80%, and 94%.

Cost of Analysis: \$40 - \$50

SULFATE

Parameter Group: General
Inorganic

STORET Units: mg/ℓ as SO₄

General: Sulfates occur naturally in waters as a result of leachings from gypsum and other common minerals or as the final oxidized stage of sulfides, sulfites, and thiosulfates having both mineral and organic origins. They may also be found in the wastes from numerous industries, including tanneries, sulfate pulp mills, textile mills, and other plants using sulfates or sulfuric acid. Excessive sulfates may exert a laxative action toward new users and cause taste problems, but such effects are not observed below 500 mg/ℓ. Limits for industrial users (especially sugar making) are much lower. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 7 days

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The turbidimetric method using a nephelometer is normally acceptable. The method is suitable for all concentration ranges of sulfate; however, in order to obtain reliable readings, use a sample aliquot containing not more than 40 mg/ℓ SO₄. Suspended matter and color interfere. Correct by running blanks from which the barium chloride has been omitted. The gravimetric method is recommended when results of the greatest accuracy are required. It is most accurate for sulfate concentrations above 10 mg/ℓ. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: A synthetic unknown sample containing 259 mg/ℓ sulfate, 108 mg/ℓ Ca, 82 mg/ℓ Mg, 3.1 mg/ℓ K, 19.9 mg/ℓ Na, 241 mg/ℓ chloride, 250 µg/ℓ nitrite N, 1.1 mg/ℓ nitrate N and 42.5 mg/ℓ total alkalinity (contributed by NaHCO₃) was analyzed by the gravimetric method, with a relative standard deviation of 4.7% and a relative error of 1.9% in 32 laboratories. Using the turbidimetric method in 19 laboratories, the relative standard deviation was 9.1% and the relative error, 1.2%.

Cost of Analysis: \$4 - \$12

SULFIDE

Parameter Group: General
Inorganic

STORET Units: mg/l as S

General: Sulfide is often present in groundwater and is common in some natural waters and sewage, coming in part from the anaerobic decomposition of organic matter. Sulfides are constituents of many industrial wastes, e.g., tanneries, paper mills, chemical plants, and gas works. It also occurs due to bacterial reduction of sulfates. The highly unpleasant taste and odor that results when sulfides occur in water make it unlikely that humans or animals will consume a harmful dose. Small traces of sulfide may be detrimental to some industrial uses. Sulfides are of little importance in irrigation waters. The sulfide ion readily reacts with free hydrogen ions in water to form hydrogen sulfide, which is very toxic, attacks metals directly, and indirectly causes serious corrosion to concrete sewers. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Add 2 mL zinc acetate, fill bottle completely, and stopper.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The titrimetric iodine method is recommended. It is applicable to the measurement of total and dissolved sulfides. Acid insoluble sulfides are not measured by this test. Reduced sulfur compounds, such as sulfite, thiosulfate and hydrosulfite, which decompose in acid may yield erratic results. Volatile iodine consuming substances will give high results. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Precision and accuracy data have not been determined.

Cost of Analysis: \$5 - \$12

SULFITE

Parameter Group: General
Inorganic

STORET Units: mg/l as SO_3

General: Sulfite may occur in certain industrial wastes but is most commonly found in boilers and boiler feedwater to which sodium sulfite has been added to reduce dissolved oxygen to a minimum and prevent corrosion. It is thought that a high concentration of sulfite in water may cause exema. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The recommended method has a minimum detectable limit of 2-3 mg/l SO_3 . An acidified sample containing an indicator is titrated with a standard potassium iodide-iodate titrant to a faint permanent blue end point. The temperature of the sample must be below 50°C. Oxidizable substances, such as organic compounds, ferrous, iron and sulfide are positive interferences. Nitrite gives a negative interference by oxidizing sulfite when the sample is acidified. Copper and possibly other metals catalyze the oxidation of sulfite. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Precision and accuracy data are not available.

Cost of Analysis: \$5 - \$12

2, 4, 5-T

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: 2, 4, 5-T (2, 4, 5-trichlorophenoxyacetic acid) is the chlorinated phenoxy acid herbicide $\text{C}_8\text{H}_5\text{Cl}_3\text{O}_3$. It is a crystalline substance, almost insoluble in water, but soluble in alcohol. It is a plant hormone. The estimated lethal dose for a 90-kg man is 54 grams. The acute oral LD_{50} to rats is 300,000 mg/kg of body weight. 2, 4, 5-T forms phenol as a breakdown product. Toxicity data for aquatic life are sparse. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C ; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 100-1,000 mL, depending on measurement method used.

Measurement: In the recommended method, chlorinated phenoxy acids and their esters are extracted from the acidified water sample with ethyl ether. The esters are hydrolyzed to acids and extraneous organic material is removed by a solvent wash. The acids are converted to methyl esters which are extracted from the aqueous phase. The extract is cleaned up by passing it through a micro-adsorption column. Detection and measurement are accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography. Interferences may be high and varied and often pose great difficulty in obtaining accurate and precise measurement of chlorinated phenoxy acid herbicides. Organic acids, especially chlorinated acids, cause the most direct interference with the determination. Phenols including chlorophenols will also interfere with this procedure. The method is recommended for use only by an experienced pesticide analyst (or under the close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is $1 \mu\text{g}/\ell$. Detection limits of $0.002 \mu\text{g}/\ell$ or so may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: \$45 - \$150, depending upon preparation required.

TEMPERATURE

Parameter Group: Temperature

STORET Units: °C

General: Temperature changes in waters are due to natural climatic phenomena or the discharge of irrigation return flows and wastes, such as distilling effluents and cooling waters. The elevation of stream temperatures may contribute to decreased oxygen capacity, increased oxygen demand, anaerobic zones, and putrefaction of sludge deposits. Temperature is a significant factor for water treatment and many industrial uses, e.g., pulp and paper. Temperature also affects the value of numerous other water quality parameters. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criteria:

Freshwater Aquatic Life

For any time of year, there are two upper limiting temperatures for a location (based on the important sensitive species found there at that time):

1. One limit consists of a maximum temperature for short exposures that is time dependent and is given by the species-specific equation:

$$\text{Temperature}_{(C^{\circ})} = (1/b) \left(\log_{10} [\text{time}_{(\text{min})}] - a \right) - 2^{\circ}\text{C}$$

where: \log_{10} = logarithm to base 10 (common logarithm)

a = intercept on the "y" or logarithmic axis of the line fitted to experimental data and which is available from Appendix II-C, NAS, 1974 for some species.

b = slope of the line fitted to experimental data and available from Appendix II-C, NAS, 1974 for some species.

and

2. The second value is a limit on the weekly average temperature that:

a. In the cooler months (mid-October to mid-April in the north and December to February in the south) will protect against mortality of important species

if the elevated plume temperature is suddenly dropped to the ambient temperature, with the limit being the acclimation temperature minus 2°C when the lower lethal threshold temperature equals the ambient water temperature (in some regions this limitation may also be applicable in summer).

or

- b. In the warmer months (April through October in the north and March through November in the south) is determined by adding to the physiological optimum temperature (usually for growth) a factor calculated as one-third of the difference between the ultimate upper incipient lethal temperature and the optimum temperature for the most sensitive important species (and appropriate life state) that normally is found at that location and time.

or

- c. During reproductive seasons (generally April through June and September through October in the north and March through May and October through November in the south) the limit is that temperature that meets site-specific requirements for successful migration, spawning, egg incubation, fry rearing, and other reproductive functions of important species. These local requirements should supersede all other requirements when they are applicable.

or

- d. There is a site-specific limit that is found necessary to preserve normal species diversity or prevent appearance of nuisance organisms.

Marine Aquatic Life

In order to ensure protection of the characteristic indigenous marine community of a water body segment from adverse thermal effects:

- a. The maximum acceptable increase in the weekly average temperature due to artificial sources is 1°C (1.8°F) during all seasons of the year, providing the summer maxima are not exceeded; and

- b. Daily temperature cycles characteristic of the water body segment should not be altered in either amplitude or frequency.

Summer thermal maxima, which define the upper thermal limits for the communities of the discharge area, should be established on a site-specific basis. Existing studies suggest the following regional limits:

	<u>Short-term Maximum</u>	<u>Maximum True Daily Mean*</u>
Sub-tropical Regions (south of Cape Canaveral and Tampa Bay, Florida, and Hawaii)	32.2°C (90°F)	29.4°C (85°F)
Cape Hatteras, N.C., to Cape Canaveral, Florida	32.2°C (90°F)	29.4°C (85°F)
Long Island (south shore) to Cape Hatteras, N.C.	30.6°C (87°F)	27.8°C (82°F)

* (True Daily Mean = average of 24 hourly temperature readings.)

Baseline thermal conditions should be measured at a site where there is no unnatural thermal addition from any source, which is in reasonable proximity to the thermal discharge (within 5 miles) and which has similar hydrography to that of the receiving waters at the discharge.

Preservation Method: Determination on site

Maximum Holding Time: No holding

Container Type: Plastic or glass

Sample Volume Required: 1000 mL

Measurement: Temperature measurements may be made with any good grade of mercury-filled or dial type Celsius thermometer, or a thermistor. The measurement device should be checked against a precision thermometer certified by the National Bureau of Standards. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Precision and accuracy will depend upon instrument used.

Cost of Analysis: Not immediately determinable.

THALLIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Tl

General: Thallium salts are used as rodenticides and ant bait, dyes and pigments in fireworks, in optical glass, and as a dipilatory. They are highly soluble in water and discharges are not likely to form precipitates. It is a cumulative poison, four times as toxic as arsenious oxide and affects the nervous system, causes muscular pain, endocrine disorders, and loss of hair. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 276.8 nm. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is $500 \mu\text{g}/\ell$; its detection limit is $100 \mu\text{g}/\ell$. The optimum concentration range is 1,000-20,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 600, 3,000, and 15,000 $\mu\text{g}/\ell$ Tl, the relative standard deviations were 3%, 1.7%, and 1.3%, respectively. Recoveries at these levels were 100%, 98%, and 98%, respectively.

Cost of Analysis: \$15 - \$20

THRESHOLD ODOR

Parameter Group: Physical

STORET Units: Threshold
Number

General: Odor is a quality factor that affects water in several ways including the acceptability of drinking water, tainting of fish, and the aesthetics of recreational waters. Odor can originate from industrial and municipal waste discharges and from natural sources such as decomposition of vegetable matter and living microscopic organisms. Odorous substances in water must be vaporizable in order to be smelled.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Glass

Sample Volume Required: 200 to 500 ml

Measurement: The consistent series method, in which the sample is divided to the point of the least definitely perceptible odor to each tester, is often used. Highly odorous samples are reduced in concentration proportionately before being tested. The method is applicable to samples ranging from nearly odorless natural waters to industrial wastes with threshold odor numbers in the thousands. Most tap waters and some waste waters are chlorinated. Dechlorination is achieved using sodium thiosulfate in exact stoichiometric quantity.

Precision and Accuracy: Precision and accuracy data are not available at this time.

Cost of Analysis: \$5 - \$10

TIN

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Sn

General: Tin does not occur in natural waters. . It is used in dyeing of fabrics, decorating porcelain, glassworks, fingernail polishes, some lacquers and varnishes, fungicides, insecticides, antihelminthics, antifoulant marine coatings and, of course, the tinning of vessels, especially foodstuff containers. Other sources include iron and steel production and power plant and industrial boilers. Tin is not believed to be toxic to man or other life forms. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 286.3 nm. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 4 mg/ℓ ; its detection limit is 800 $\mu\text{g}/\ell$. The optimum concentration range is 16,000-200,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 4,000, 20,000, and 60,000 $\mu\text{g}/\ell$ Sn, the relative standard deviations were 6.2%, 2.5%, and .8%, respectively. Recoveries at these levels were 96%, 101%, and 101%, respectively.

Cost of Analysis: \$15 - \$20

TITANIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Ti

General: Titanium ores and salts are abundantly distributed in the earth's crust, constituting from 0.5% to 10% of soils. The metal is used chiefly in alloying, and its salts are used in paint, paper, and dyeing industries, in the manufacture of electronic components, and in glass and ceramic production. There is little evidence of harm to life forms from titanium. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 365.3 nm. A number of elements increase the sensitivity of titanium. To control this problem, potassium (1,000 mg/L) must be added to the standards and samples. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 2,000 $\mu\text{g}/\ell$; its detection limit is 300 $\mu\text{g}/\ell$. The optimum concentration range is 5,000-100,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 2,000, 10,000, and 50,000 $\mu\text{g}/\ell$ Ti, the relative standard deviations were 3.5%, 1.0%, and .8%, respectively. Recoveries at these levels were 97%, 91%, and 88%, respectively.

Cost of Analysis: \$10 - \$20

TOLUENE

Parameter Group: General
Organic

STORET Units: $\mu\text{g}/\ell$

General: Toluene ($\text{C}_6\text{H}_5\text{CH}_3$), a flammable liquid with an odor of benzene, is a constituent of coal tar. It is used in the manufacture of organic substances and as a solvent in the extraction of various principles from plants. Toluene is modestly soluble in water at normal temperatures. Its LD_{50} for rats is 7,000 mg/kg of body weight. Lethal concentrations to fish in clean water range from 10,000 to over 90,000 $\mu\text{g}/\ell$ depending upon temperature and species. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: Hexadecane extraction followed by gas chromatographic and mass spectrometric analysis is often used. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Detection limits should be around 2-10 $\mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$15 - \$30

TOTAL COLIFORM

Parameter Group: Bacteriologic STORET Units: See below

General: The coliform bacteria group as defined by the tests described herein includes organisms of diverse origins, including intermediate and Aerobacter aerogenes strains, which are usually of soil, vegetable, or other non-fecal origin; E. coli, which is usually but not always of fecal origin; and fecal coliform, which is a positive indication of the excrement of warm-blooded animals. The direct examination for the presence of a specific pathogen in water is not usually practicable for control purposes, and total coliform has been widely used as a microbiological indicator organism. The more specific fecal coliform indicator is gaining in popularity, however. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Cool to 4°C. Add a dechlorinating agent (e.g., sodium thiosulfate) if residual chlorine is present. Samples high in heavy metals should have a chelating agent (e.g., EDTA) added to reduce metal toxicity.

Maximum Holding Time: 6 hours (30 hours absolute maximum for potable water sample).

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The multiple tube fermentation technique, which defines the coliform group as all aerobic and facultative anaerobic, gram negative, rod-shaped, nonspore-forming bacteria that ferment lactose with gas formations within 48 hours at 35°C, is recommended. The simpler membrane filter technique, which defines the coliform group as the above bacteria that produce a dark colony with a metallic sheen within 24 hours on an Endo-type medium containing lactose, is also recommended, especially for nondrinking water tests. Results of the former are expressed statistically as the Most Probable Number (MPN), while the latter are expressed as number of colonies per 100 mL. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: Not applicable

Cost of Analysis: \$10 - \$12 MFT
\$15 - \$20 MPN

TOXAPHENE

Parameter Group: Pesticides

STORET Units: $\mu\text{g}/\ell$

General: Toxaphene is a chlorinated camphene insecticide. It is insoluble in water but highly soluble in organic solvents and oils. It has been reported that lakes treated with toxaphene concentrations ranging from 40 to 150 $\mu\text{g}/\ell$ remained toxic to fish for periods of a few months to five years. Bioconcentration accumulations of toxaphene of 5,000 to 21,000 times water concentrations have been observed in brook trout exposed only through water. Accumulation factors of 3,400 to 17,000 from aqueous solution have been reported for bacteria, algae, and fungi. Owing to the turpentine odor, it is not likely that toxic concentrations will be consumed by man or animals. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants. A toxic effluent limitation has been prescribed for this parameter by the NPDES permits program.

Criteria:

- 5 $\mu\text{g}/\ell$ for domestic supply (health)
- 0.005 $\mu\text{g}/\ell$ for freshwater and marine aquatic life

Preservation Method: Cool to 4°C; analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 50-100 mL or more

Measurement: The use of co-solvent extraction and detection and measurement accomplished by electron capture, microcoulometric or electrolytic conductivity gas chromatography is recommended for toxaphene under favorable conditions. Many interferences exist, especially PCB's, phthalate esters, and organophosphorus pesticides, and the method is only recommended for use by a skilled, experienced pesticide analyst (or under close supervision of such a person). For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The detection limit is affected by many factors, but usually falls in the 0.001 to 1 $\mu\text{g}/\ell$ range. Increased sensitivity is likely to increase interference. Typically, the percent recovery decreases with increasing concentration.

Cost of Analysis: \$30 - \$150, depending upon preparation required.

TRICHLOROETHYLENE

Parameter Group:

STORET Units:

General: Trichloroethylene, a nonflammable liquid with a chloroform-like odor, is practically insoluble in water. It is used as a solvent and in solvent extraction by several industries, in degreasing, in the manufacture of chemicals and pharmaceuticals, and in dry cleaning. The oral LD₅₀ for dogs is 5.86 g/kg of body weight. Concentrations of 55 mg/ℓ will stupify fish within 10 minutes. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: In the recommended Bellar procedure the sample is stripped with an inert gas; volatiles are captured on an adsorbent trap and desorbed into a modified gas chromatograph equipped with a halogen-specific detector. Methodology should be checked for interferences, e.g., from bromine or iodine. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 µg/ℓ. Detection limits of 0.2-3 µg/ℓ may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60

TURBIDITY

Parameter Group: Physical

STORET Units: Formazin
Turbidity
Units

General: Turbidity is an optical property of water, reflecting its propensity for scattering light. From chlorination considerations, finished drinking waters typically have a maximum limit of 1 turbidity unit where the water enters the distribution system. Turbid water interferes with recreational use and aesthetic enjoyment of water. The less turbid the water, the more desirable it becomes for swimming and other water contact sports. See discussion for suspended solids. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Cool to 4°C.

Maximum Holding Time: 24 hours

Container Type: Plastic or glass

Sample Volume Required: 100 mL

Measurement: The recommended method is applicable to drinking, surface, and saline waters in the range of turbidity from 0 to 40 nephelometric turbidity units (NTU). The method is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles will affect the results in a positive manner. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: In a single laboratory, using surface water samples at levels of 26, 41, 75 and 180 NTU, the relative standard deviations were 2.3%, 2.3%, 1.6%, and 2.6%, respectively.

Cost of Analysis: \$3 - \$5

URANIUM

Parameter Group: Metals

STORET Units: mg/l

General: In addition to atomic energy applications, uranium is used in photography, glazing and painting porcelain, and in chemical processes. Many uranium salts are soluble in water. It has been reported that uranium and many of its salts are toxic; however, limited studies indicate that natural uranium, absorbed by people through the water and foodstuffs grown on land, may be a limiting factor in the incidence of leukemia. There is generally greater concern about the radiological hazards of uranium than about its chemical effects, however.

Criterion: Not established

Preservation Method: Analyze as soon as possible. Add HNO_3 to $\text{pH} \approx 2$ and cool to 4°C .

Maximum Holding Time: Unknown

Container Type: Plastic or glass

Sample Volume Required: 50 mL

Measurement: The direct fluorometric method is often used. The concentration range is from 0.005 to 2.0 mg/l. For higher concentrations, the extraction method may be used. The method involves the measurement of the fluorescence of a fused disk of sodium fluoride, lithium fluoride, and uranium compound exposed to ultraviolet light. The intensity of the fluorescence is proportional to the uranium concentration. Small quantities of cadmium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, lead, platinum, silicon, thorium, and zinc interfere by quenching the uranium fluorescence and a purification or spiking method must be used. The AA method may also be used.

Precision and Accuracy: The single operator precision (S) at a uranium concentration of X mg/l may be estimated from

$$\log (S-0.0016) = \log 0.129 + 120 \log X.$$

Cost of Analysis: \$30 - \$40

VANADIUM

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as V

General: Minerals containing vanadium are widespread in nature. In addition to its metallurgical uses, principally in steel alloying, its salts are used in the manufacture of glass, ceramics, ink, in photography, and in the dyeing and printing of fabrics. It is not considered toxic and, in fact, may play a beneficial role in reducing cholesterol, preventing heart disease and dental caries, and lowering the phospholipid content of the liver. Small quantities of vanadium may stimulate plant growth. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program.

Criterion: Not established

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 318.4 nm. High concentrations of aluminum and titanium increase the sensitivity of vanadium. This interference can be controlled by adding excess aluminum (1,000 mg/L) to both samples and standards. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 800 $\mu\text{g}/\ell$; its detection limit is 200 $\mu\text{g}/\ell$. The optimum concentration range is 1,000-100,000 $\mu\text{g}/\ell$. In a single laboratory, using a mixed industrial-domestic waste effluent at concentrations of 2,000, 10,000, and 50,000 $\mu\text{g}/\ell$ V, the relative standard deviations were 5%, 1%, and .4%, respectively. Recoveries at these levels were 100%, 95%, and 97%, respectively.

Cost of Analysis: \$10 - \$20

VINYL CHLORIDE

Parameter Group:

STORET Units:

General: Vinyl chloride (chloroethene, $\text{CH}_2:\text{CHCl}$), a flammable gas with an ethereal odor, is only slightly soluble in water. It is prepared by catalytic addition of hydrogen chloride to acetylene or by pyrolysis of ethylene dichloride and is used chiefly for making vinyl resins. U.S. production exceeds 3 billion pounds annually. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Criterion: Not established

Preservation Method: Sample history must be known before any chemical or physical preservation steps can be applied to protect against phase separation. Fill the sample bottle completely and seal until analysis is performed. Do not refrigerate.

Maximum Holding Time: Unknown; preferably analyze within 1 hour.

Container Type: Borosilicate glass

Sample Volume Required: In excess of 200 mL

Measurement: In the recommended Bellar procedure the sample is stripped with an inert gas; volatiles are captured on an adsorbent trap and desorbed into a modified gas chromatograph equipped with a halogen-specific detector. Methodology should be checked for interferences, e.g., from bromine or iodine. A BAT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: Sensitivity of the method is approximately 1,000 $\mu\text{g}/\ell$. Detection limits of 0.2-3 $\mu\text{g}/\ell$ may be achieved. Precision and accuracy data are not available at this time.

Cost of Analysis: Around \$60

XYLENE

Parameter Group: General
Organic

STORET Units: $\mu\text{g}/\ell$

General: Xylene ($\text{C}_6\text{H}_4(\text{CH}_3)_2$), a flammable liquid, is a constituent of coal tar. It is used in the manufacture of dyes and organic substances, as a solvent, and as a cleaning agent. Xylene is insoluble in water. Its LD_{50} for white rats is 4.3g/kg of body weight. Lethal concentrations to fish range from 10,000 to 90,000 $\mu\text{g}/\ell$ depending upon temperature and species.

Criterion: Not established

Preservation Method: Not determined. Analyze promptly.

Maximum Holding Time: Unknown

Container Type: Borosilicate glass

Sample Volume Required: 200-1,000 mL

Measurement: Hexadecane extraction followed by gas chromatographic and mass spectrometric analysis is often used.

Precision and Accuracy: Detection limits should be around 2-10 $\mu\text{g}/\ell$. Precision and accuracy data are not available at this time.

Cost of Analysis: \$15 - \$30

ZINC

Parameter Group: Metals

STORET Units: $\mu\text{g}/\ell$ as Zn

General: Zinc is usually found in nature as the sulfide. Zinc is used in galvanizing and in the preparation of alloys for dye casting. Zinc is also used in brass and bronze alloys, slush castings, photoengraving, printing plates, silver and stainless steel tableware, viscose rayon yarn, wood pulp, and newsprint paper. Other sources include mining areas, paint pigments, cosmetics, pharmaceuticals, insecticides, and many more. Zinc is an essential and beneficial element in human metabolism. Excessive amounts of zinc affect growth rates and decrease both the weight and fat content of the liver. This is a parameter which is regulated by BPT guidelines prescribed by the NPDES permits program. This parameter will be regulated by BAT guidelines prescribed by the NPDES permits program. It is one of the Consent Decree pollutants.

Zinc is a

Criteria:

- 5,000 $\mu\text{g}/\ell$ for domestic water supplies (welfare).
- For freshwater aquatic life, 0.01 of the 96-hour LC_{50} as determined through bioassay using a sensitive resident species.

Preservation Method: Analyze as soon as possible. If storage is necessary, add HNO_3 to pH <2.

Maximum Holding Time: 6 months

Container Type: Plastic or glass

Sample Volume Required: 100-200 mL

Measurement: The AA spectrophotometric method is recommended, using a wavelength of 213.9 nm. The air-acetylene flame absorbs about 25% of the energy at the 213.9-nm line. The sensitivity may be increased by the use of low-temperature flames. For BPT NPDES purposes the measurement of this parameter is prescribed by 40 CFR 136. A BPT NPDES method will be prescribed for this parameter in 40 CFR 136.

Precision and Accuracy: The AA method sensitivity is 20 $\mu\text{g}/\ell$; its detection limit is 5 $\mu\text{g}/\ell$. The optimum concentration range is 50-2,000 $\mu\text{g}/\ell$. At a concentration of 500 $\mu\text{g}/\ell$, the relative standard deviation is 8.2%, and the relative error is 0.4%.

Cost of Analysis: \$10 - \$15

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PART II



ENVIRONMENTAL PROTECTION AGENCY

■

WATER PROGRAMS

**Guidelines Establishing Test Procedures
for Analysis of Pollutants**

Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL
PROTECTION AGENCY

SUBCHAPTER D—WATER PROGRAMS

PART 136—GUIDELINES ESTABLISHING
TEST PROCEDURES FOR THE ANALY-
SIS OF POLLUTANTS

Notice was published in the *FEDERAL REGISTER* issue of June 29, 1973 (38 FR 17318) at 40 CFR 130, that the Environmental Protection Agency (EPA) was giving consideration to the testing procedures required pursuant to section 304(g) of the Federal Water Pollution Control Act Amendments of 1972 (86 Stat. 816, et seq., Pub. L. 92-500 (1972)) hereinafter referred to as the Act. These considerations were given in the form of proposed guidelines establishing test procedures.

Section 304(g) of the Act requires that the Administrator shall promulgate guidelines establishing test procedures for the analysis of pollutants that shall include factors which must be provided in: 1, any certification pursuant to section 401 of the Act, or 2, any permit application pursuant to section 402 of the Act. Such test procedures are to be used by permit applicants to demonstrate that effluent discharges meet applicable pollutant discharge limitations, and by the States and other enforcement activities in routine or random monitoring of effluents to verify effectiveness of pollution control measures.

These guidelines require that discharge measurements, including but not limited to the pollutants and parameters listed in Table I, be performed by the test procedures indicated; or under certain circumstances by other test procedures for analysis that may be more advantageous to use, when such other test procedures have the approval of the Regional Administrator of the Region where such discharge will occur, and when the Director of an approved State National Pollutant Discharge Elimination System (NPDES) Program (hereinafter referred to as the Director) for the State in which such discharge will occur has no objection to such approval.

The list of test procedures in Table I is published herein as final rulemaking and represents major departures from the list of proposed test procedures which was published in 38 FR 17318, dated June 29, 1973. These revisions were made after carefully considering all written comments which were received pertaining to the proposed test procedures. All written comments are on file and available for public review with the Quality Assurance Division, Office of Research and Development, EPA, Washington, D.C.

The principal revisions to the proposed test procedures are as follows:

1. Where several reliable test procedures for analysis are available from the given references for a given pollutant or parameter, each such test procedure has been approved for use for making the measurements required by sections 401 and 402 and related sections of the Act. Approved test procedures have been

selected to assure an acceptable level of intercomparability of pollutants discharge data. For several pollutants and parameters it has still been necessary to approve only a single test procedure to assure this level of acceptability. This is a major departure from the proposed test procedures which would have required the use of a single reference method for each pollutant or parameter.

2. Under certain circumstances a test procedure not shown on the approved list may be considered by an applicant to be more advantageous to use. Under guidelines in §§ 136.4 and 136.5 it may be approved by the Regional Administrator of the Region where the discharge will occur, providing the Director has no objections. Inasmuch as there is no longer a single approved reference method against which a comparison can be made, the procedures for establishing such comparisons that were required by the proposed test procedures in § 130.4(b) have been deleted from this final guideline for test procedures for the analysis of pollutants.

3. A mechanism is also provided to assure national uniformity of such approvals of alternate test procedures for the analysis of pollutants. This is achieved through a centralized, internal review within the EPA of all applications for the use of alternate testing procedures. These will be reviewed and approved or disapproved on the basis of submitted information and other available information and laboratory tests which may be required by the Regional Administrator.

As deemed necessary, the Administrator will expand or revise these guidelines to provide the most responsive and appropriate list of test procedures to meet the requirements of sections 304(g), 401 and 402 of the Act, as amended.

These final guidelines establishing test procedures for the analysis of pollutants supersede the interim list of test procedures published in the *FEDERAL REGISTER* on April 19, 1973 (38 FR 9740) at 40 CFR Part 126 and subsequent procedures published on July 24, 1973 (38 FR 19894) at 40 CFR Part 124. Those regulations established interim test procedures for the submittal of applications under section 402 of the Act. Because of the importance of these guidelines for test procedures for the analysis of pollutants to the National Pollution Discharge Elimination System (NPDES), the Administrator finds good cause to declare that these guidelines shall be effective October 16, 1973.

JOHN QUARLES,
Acting Administrator.

OCTOBER 3, 1973.

PART 136—TEST PROCEDURES FOR THE
ANALYSIS OF POLLUTANTS

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| Sec. | |
| 136.1 | Applicability. |
| 136.2 | Definitions. |
| 136.3 | Identification of test procedures. |
| 136.4 | Application for alternate test procedures. |
| 136.5 | Approval of alternate test procedures. |

AUTHORITY: Sec. 304(g) of Federal Water Pollution Control Act Amendments of 1972 (86 Stat. 816, et seq., Pub. L. 92-500).

§ 136.1 Applicability.

The procedures prescribed herein shall, except as noted in § 136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(a) An application submitted to the Administrator, or to a State having an approved NPDES program, for a permit under section 402 of the Federal Water Pollution Control Act as amended (FWPCA), and,

(b) Reports required to be submitted by dischargers under the NPDES established by Parts 124 and 125 of this chapter, and,

(c) Certifications issued by States pursuant to section 401 of the FWPCA, as amended.

§ 136.2 Definitions.

As used in this part, the term:

(a) "Act" means the Federal Water Pollution Control Act, as amended, 33 U.S.C. 1314, et seq.

(b) "Administrator" means the Administrator of the U.S. Environmental Protection Agency.

(c) "Regional Administrator" means one of the EPA Regional Administrators.

(d) "Director" means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) "National Pollutant Discharge Elimination System (NPDES)" means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) "Standard Methods" means *Standard Methods for the Examination of Water and Waste Water*, 13th Edition, 1971. This publication is available from the American Public Health Association, 1015 18th St. NW., Washington, D.C. 20036.

(g) "ASTM" means *Annual Book of Standards, Part 23, Water, Atmospheric Analysis*, 1972. This publication is available from the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pennsylvania 19103.

(h) "EPA Methods" means *Methods for Chemical Analysis of Water and Wastes*, 1971, Environmental Protection Agency, Analytical Quality Control Laboratory, Cincinnati, Ohio. This publication is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402 (Stock Number 5501-0067).

§ 136.3 Identification of test procedures.

Every parameter or pollutant for which an effluent limitation is now specified pursuant to sections 401 and 402 of the Act is named together with test descriptions and references in Table I. The discharge parameter values for which reports are required must be de-

terminated by one of the standard analytical methods cited and described in Table I, or under certain circumstances by other methods that may be more advantageous to use when such other methods have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing that the Director of the State in which such discharge will occur does not object to the use of such alternate test procedures.

Under certain circumstances the Re-

gional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator or Director upon the recommendation of the Director of the Methods Development and Quality Assurance Research Laboratory.

TABLE I—LIST OF APPROVED TEST PROCEDURES

Parameter and units	Method	References		
		Standard methods	ASTM	EPA methods
General analytical methods:				
1. Alkalinity as CaCO ₃ mg CaCO ₃ /liter.	Titration: electrometric, manual or automated method—methyl orange.	p. 370	p. 143	p. 6. p. 8.
2. B.O.D. five day mg/liter.	Modified winkler or probe method.	p. 489		
3. Chemical oxygen demand (C.O.D.) mg/liter.	Dichromate reflux.	p. 498	p. 219	p. 17.
4. Total solids mg/liter.	Gravimetric 103-105° C.	p. 635		p. 280.
5. Total dissolved (filterable) solids mg/liter.	Glass fiber filtration 180° C.			p. 276.
6. Total suspended (non-filterable) solids mg/liter.	Glass fiber filtration 103-105° C.	p. 637		p. 278.
7. Total volatile solids mg/liter.	Gravimetric 650° C.	p. 636		p. 282.
8. Ammonia (as N) mg/liter.	Distillation—nesslerization or titration automated phenolate.			p. 134. p. 141.
9. Kjeldahl nitrogen (as N) mg/liter.	Digestion + distillation—nesslerization or titration automated digestion phenolate.	p. 469		p. 149. p. 167.
10. Nitrate (as N) mg/liter.	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction.	p. 458 p. 461	p. 124	p. 170. p. 175. p. 186.
11. Total phosphorus (as P) mg/liter.	Persulfate digestion and single reagent (ascorbic acid), or manual digestion, and automated single reagent or stannous chloride.	p. 626 p. 632	p. 42	p. 235. p. 246. p. 259.
12. Acidity mg CaCO ₃ /liter.	Electrometric end point or phenolphthalein end point.		p. 148	
13. Total organic carbon (TOC) mg/liter.	Combustion—Infrared method ¹ .	p. 257	p. 702	p. 221.
14. Hardness—total mg CaCO ₃ /liter.	EDTA titration; automated colorimetric atomic absorption.	p. 179	p. 170	p. 76. p. 78.
15. Nitrite (as N) mg/liter.	Manual or automated colorimetric diazotization.			p. 185. p. 196.
Analytical methods for trace metals:				
16. Aluminum—total ¹ mg/liter.	Atomic absorption.	p. 210		p. 98.
17. Antimony—total ¹ mg/liter.	Atomic absorption ¹ .			
18. Arsenic—total mg/liter.	Digestion plus silver diethyldithiocarbamate; atomic absorption. ¹	p. 65 p. 62		p. 13.
19. Barium—total ¹ mg/liter.	Atomic absorption ¹ .	p. 210		
20. Beryllium—total ¹ mg/liter.	Aluminum; atomic absorption.	p. 67 p. 210		
21. Boron—total mg/liter.	Curcumin.	p. 69		
22. Cadmium—total ¹ mg/liter.	Atomic absorption; colorimetric.	p. 210 p. 422	p. 692	p. 101.
23. Calcium—total ¹ mg/liter.	EDTA titration; atomic absorption.	p. 84	p. 692	p. 102.
24. Chromium VI mg/liter.	Extraction and atomic absorption; colorimetric.	p. 429		p. 94.

Parameter and units	Method	References		
		Standard methods	ASTM	EPA methods
25. Chromium—total mg/liter.	Atomic absorption; colorimetric.	p. 210	p. 692	p. 104.
26. Cobalt—total mg/liter.	Atomic absorption.	p. 428	p. 403	
27. Copper—total mg/liter.	Atomic absorption; colorimetric.	p. 210	p. 692	p. 106.
28. Iron—total mg/liter.	do.	p. 430	p. 410	
29. Lead—total mg/liter.	do.	p. 210	p. 692	p. 108.
30. Magnesium—total mg/liter.	Atomic absorption; Gravimetric.	p. 433	p. 182	
31. Manganese—total mg/liter.	Atomic absorption.	p. 210	p. 692	p. 110.
32. Mercury—total mg/liter.	Flameless atomic absorption.	p. 435		
33. Molybdenum—total mg/liter.	Atomic absorption.	p. 418	p. 692	p. 112.
34. Nickel—total mg/liter.	Atomic absorption; colorimetric.	p. 201		
35. Potassium—total mg/liter.	Atomic absorption; colorimetric; flame photometric.	p. 210	p. 692	p. 114.
36. Selenium—total mg/liter.	Atomic absorption.	p. 443	p. 692	
37. Silver—total mg/liter.	Atomic absorption.	p. 283	p. 326	p. 116.
38. Sodium—total mg/liter.	Flame photometric; atomic absorption.	p. 285		
39. Thallium—total mg/liter.	Atomic absorption.	p. 317	p. 326	p. 118.
40. Tin—total mg/liter.	do.			
41. Titanium—total mg/liter.	do.			
42. Vanadium—total mg/liter.	Atomic Absorption; Colorimetric.	p. 157		
43. Zinc—total mg/liter.	Atomic Absorption; Colorimetric.	p. 210	p. 692	p. 120.
Analytical methods for nutrients, anions, and organics:				
44. Organic nitrogen (as N) mg/liter.	Kjeldahl nitrogen minus ammonia nitrogen.	p. 468		p. 149.
45. Ortho-phosphate (as P) mg/liter.	Direct single reagent; automated single reagent or stannous chloride.	p. 632	p. 42	p. 235.
46. Sulfate (as SO ₄) mg/liter.	Gravimetric; turbidimetric; automated colorimetric—barium chloranilate.	p. 331	p. 61	p. 248.
47. Sulfide (as S) mg/liter.	Titrimetric—iodine.	p. 334	p. 62	p. 269.
48. Sulfite (as SO ₃) mg/liter.	Titrimetric; iodide-iodate.	p. 551		p. 286.
49. Bromide mg/liter.	do.	p. 337	p. 261	p. 288.
50. Chloride mg/liter.	Silver nitrate; mercuric nitrate; automated colorimetric—ferricyanide.	p. 96	p. 23	p. 294.
51. Cyanide—total mg/liter.	Distillation—silver nitrate titration or pyridine pyrazolone colorimetric.	p. 97	p. 21	p. 29.
52. Fluoride mg/liter.	Distillation—SPADNS.	p. 397	p. 656	p. 31.
53. Chlorine—total residual mg/liter.	Colorimetric; amperometric titration.	p. 171	p. 191	p. 41.
54. Oil and grease mg/liter.	Liquid-Liquid extraction with trichlorotrifluoroethane.	p. 174	p. 223	p. 64.
55. Phenols mg/liter.	Colorimetric, 4 A.A.P.	p. 502	p. 445	p. 232.
56. Surfactants mg/liter.	Methylene blue colorimetric.	p. 339	p. 619	p. 181.
57. Aldehydes mg/liter.	Gas chromatography.			
58. Benzidine mg/liter.	Diazotization—colorimetric.			
59. Chlorinated organic compounds (except pesticides) mg/liter.	Gas chromatography.			
60. Pesticides mg/liter.	Gas chromatography.			
Analytical methods for physical and biological parameters:				
61. Color platinum-cobalt units or dominant wave-length, hue, luminance, purity.	Colorimetric; spectrophotometric.	p. 160		p. 38.
62. Specific conductance mho/cm at 25° C.	Wheatstone bridge.	p. 323	p. 163	p. 284.
63. Turbidity Jackson units.	Turbidimeter.	p. 350	p. 467	p. 308.

See Note at end of Table I

Parameter and units	Method	References		
		Standard methods	ASTM	EPA methods
64. Fecal streptococci bacteria number/100 ml.	MPN; membrane filter; plate count.....	p. 689.....		
65. Coliform bacteria (fecal) number/100 ml.	MPN; Membrane filter.....	p. 690.....		
66. Coliform bacteria (total) number/100 ml.do.....	p. 691.....		
Radiological parameters:		p. 664.....		
67. Alpha—total pCi/liter..	Proportional counter; scintillation counter	p. 598.....	p. 509.....	
68. Alpha—counting errordo.....	p. 599.....	p. 512.....	
69. Beta—total pCi/liter..	Proportional counter.....	p. 598.....	p. 478.....	
70. Beta—counting error..do.....	p. 598.....	p. 478.....	
71. Radium—total pCi/liter.	Proportional counter; scintillation counter..	p. 611.....	p. 674.....	
		p. 617.....		

¹ A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on Combustion-Methane Detection, is also acceptable.

² For the determination of total metals the sample is not filtered before processing. Choose a volume of sample appropriate for the expected level of metals. If much suspended material is present, as little as 50-100 ml of well-mixed sample will most probably be sufficient. (The sample volume required may also vary proportionally with the number of metals to be determined.)

³ Transfer a representative aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated distilled HNO₃. Place the beaker on a hotplate and evaporate to dryness making certain that the sample does not boil. Cool the beaker and add another 3 ml portion of distilled concentrated HNO₃. Cover the beaker with a watch glass and return to the hotplate. Increase the temperature of the hotplate so that a gentle reflux action occurs. Continue heating, adding additional acid as necessary until the digestion is complete, generally indicated by a light colored residue. Add (1:1 with distilled water) distilled concentrated HCl in an amount sufficient to dissolve the residue upon warming. Wash down the beaker walls and the watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentrations. The sample is now ready for analysis. Concentrations so determined shall be reported as "total".

⁴ See D. C. Manning, "Technical Notes", Atomic Absorption Newsletter, Vol. 10, No. 6 p. 123, 1971. Available from Perkin-Elmer Corporation, Main Avenue, Norwalk, Connecticut 06852.

⁵ Atomic absorption method available from Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, USEPA, Cincinnati, Ohio 45268.

⁶ For updated method, see: Journal of the American Water Works Association 64, No. 1, pp. 20-25 (Jan. 1972) or ASTM Method D 3223-73, American Society for Testing and Materials Headquarters, 1916 Race St., Philadelphia, Pa. 19103.

⁷ Interim procedures for algicides, chlorinated organic compounds, and pesticides can be obtained from the Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, USEPA, Cincinnati, Ohio 45268.

⁸ Benzidine may be estimated by the method of M.A. El-Dib, "Colorimetric Determination of Aniline Derivatives in Natural Waters", El-Dib, M.A., Journal of the Association of Official Analytical Chemists, Vol. 54, No. 6, Nov., 1971, pp. 1383-1387.

⁹ As a prescreening measurement.

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an appli-

cation for an alternate test procedure may be made by letter in triplicate. Any application for an alternate test procedure under this subchapter shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will occur has final responsibility for approval of any alternate test procedure.

(b) Within thirty days of receipt of an application, the Director will forward such application, together with his recommendations, to the Regional Administrator. Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application, and shall forward a copy of the rejected application and his decision to the Director of the State Permit Program and to the Director of the Methods Development and Quality Assurance Research Laboratory.

(c) Before approving any application for an alternate test procedure, the Regional Administrator shall forward a copy of the application to the Director of the Methods Development and Quality Assurance Laboratory for review and recommendation.

(d) Within ninety days of receipt by the Regional Administrator of an application for an alternate test procedure, the Regional Administrator shall notify the applicant and the appropriate State agency of approval or rejection, or shall specify the additional information which is required to determine whether to approve the proposed test procedure. Prior to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Director of the Methods Development and Quality Assurance Research Laboratory. A copy of all approval and rejection notifications will be forwarded to the Director, Methods Development and Quality Assurance Research Laboratory, for the purposes of national coordination.

[FR Doc.73-21466 Filed 10-15-73; 8:45 am]

WEDNESDAY, DECEMBER 1, 1976



PART II:

**ENVIRONMENTAL
PROTECTION
AGENCY**



WATER PROGRAMS

**Guidelines Establishing Test Procedures
for the Analysis of Pollutants**

Amendments

Title 40—Protection of Environment
CHAPTER I—ENVIRONMENTAL
PROTECTION AGENCY
SUBCHAPTER D—WATER PROGRAMS
[FRL 630-4]

PART 136—GUIDELINES ESTABLISHING
TEST PROCEDURES FOR THE ANALYSIS
OF POLLUTANTS

Amendment of Regulations

On June 9, 1975, proposed amendments to the Guidelines Establishing Test Procedures for the Analysis of Pollutants (40 CFR 136) were published in the *FEDERAL REGISTER* (40 FR 24535) as required by section 304(g) of the Federal Water Pollution Control Act Amendments of 1972 (86 Stat. 816, et seq., Pub. L. 92-500, 1972) hereinafter referred to as the Act.

Section 304(g) of the Act requires that the Administrator shall promulgate guidelines establishing test procedures for the analysis of pollutants that shall include factors which must be provided in: (1) any certification pursuant to section 401 of the Act, or (2) any permit application pursuant to section 402 of the Act. Such test procedures are to be used by permit applicants to demonstrate that effluent discharges meet applicable pollutant discharge limitations and by the States and other enforcement activities in routine or random monitoring of effluents to verify compliance with pollution control measures.

Interested persons were requested to submit written comments, suggestions, or objections to the proposed amendments by September 7, 1975. One hundred and thirty-five letters were received from commenters. The following categories of organizations were represented by the commenters: Federal agencies accounted for twenty-four responses; State agencies accounted for twenty-six responses; local agencies accounted for seventeen responses; regulated major dischargers accounted for forty-seven responses; trade and professional organizations accounted for eight responses; analytical instrument manufacturers and vendors accounted for seven responses; and analytical service laboratories accounted for six responses.

All comments were carefully evaluated by a technical review committee. Based upon the review of comments, the following principal changes to the proposed amendments were made:

(A) *Definitions.* Section 136.2 has been amended to update references: Twenty commenters, representing the entire spectrum of responding groups pointed out that the references cited in §§ 136.2(f), 136.2(g), and 136.2(h) were out-of-date; §§ 136.2(f), 136.2(g), and 136.2(h), respectively, have been amended to show the following editions of the standard references: "14th Edition of Standard Methods for the Examination of Water and Waste Water;" "1974 EPA Manual of Methods for the Analysis of Water and Waste;" and "Part 31, 1975 Annual Book of ASTM Standards."

(B) *Identification of Test Procedures.* Both the content and format of § 136.3, "Table I, List of Approved Test Proce-

dures" have been revised in response to twenty-one comments received from State and local governments, major regulated dischargers, professional and trade associations, and analytical laboratories.

Table I has been revised by:

(1) The addition of a fourth column of references which includes procedures of the United States Geological Survey which are equivalent to previously approved methods.

(2) The addition of a fifth column of miscellaneous references to procedures which are equivalent to previously approved methods.

(3) Listing generically related parameters alphabetically within four subcategories: bacteria, metals, radiological and residue, and by listing these subcategory headings in alphabetic sequence relative to the remaining parameters.

(4) Deleting the parameter "Algicides" and by entering the single relevant algicide, "Pentachlorophenol" by its chemical name.

(C) *Clarification of Test Parameters.* The conditions for analysis of several parameters have been more specifically defined as a result of comments received by the Agency:

(1) In response to five commenters representing State or local governments, major dischargers, or analytical instrument manufacturers, the end-point for the alkalinity determination is specifically designated as pH 4.5.

(2) Manual digestion and distillation are still required as necessary preliminary steps for the Kjeldahl nitrogen procedure. Analysis after such distillation may be by Nessler color comparison, titration, electrode, or automated phenolate procedures.

(3) In response to eight commenters representative of Federal and State governments, major dischargers, and analytical instrument manufacturers, manual distillation at pH 9.5 is now specified for ammonia measurement.

(D) *New Parameters and Analytical Procedures.* Forty-four new parameters have been added to Table I. In addition to the designation of analytical procedures for these new parameters, the following modifications have been made in analytical procedures designated in response to comments.

(1) The ortho-tolidine procedure was not approved for the measurement of residual chlorine because of its poor accuracy and precision. Its approval had been requested by seven commenters representing major dischargers, State, or local governments, and analytical instrument manufacturers. Instead, the N,N-diethyl-p-phenylenediamine (DPD) method is approved as an interim procedure pending more intensive laboratory testing. It has many of the advantages of the ortho-tolidine procedure such as low cost, ease of operation, and also is of acceptable precision and accuracy.

(2) The Environmental Protection Agency concurred with the American Dye Manufacturers' request to approve its procedure for measurement of color, and copies of the procedure are now available at the Environmental Monitoring and

Support Laboratory, Cincinnati (EMSL-CI).

(3) In response to three requests from Federal, State governments, and dischargers, "hardness," may be measured as the sum of calcium and magnesium analyzed by atomic absorption and expressed as their carbonates.

(4) The proposal to limit measurement of fecal coliform bacteria in the presence of chlorine to only the "Most Probable Number" (MPN) procedure has been withdrawn in response to requests from forty-five commenters including State pollution control agencies, permit holders, analysts, treatment plant operators, and a manufacturer of analytical supplies. The membrane filter (MF) procedure will continue to be an approved technique for the routine measurement of fecal coliform in the presence of chlorine. However, the MPN procedure must be used to resolve controversial situations. The technique selected by the analyst must be reported with the data.

(5) A total of fifteen objections, representing the entire spectrum of commenters, addressed the drying temperatures used for measurement of residues. The use of different temperatures in drying of total residue, dissolved residue and suspended residue was cited as not allowing direct intercomparability between these measurements. Because the intent of designating the three separate residue parameters is to measure separate waste characteristics (low drying temperatures to measure volatile substances, high drying temperatures to measure anhydrous inorganic substances), the difference in drying temperatures for these residue parameters must be preserved.

(E) *Deletion of Measurement Techniques.* Some measurement techniques that had been proposed have been deleted in response to objections raised during the public comment period.

(1) The proposed infrared spectrophotometric analysis for oil and grease has been withdrawn. Eleven commenters representing Federal or State agencies and major dischargers claimed that this parameter is defined by the measurement procedure. Any alteration in the procedure would change the definition of the parameter. The Environmental Protection Agency agreed.

(2) The proposed separate parameter for sulfide at concentrations below 1 mg/l, has been withdrawn. Methylene blue spectrophotometry is now included in Table I as an approved procedure for sulfide analysis. The titrimetric iodine procedure for sulfide analysis may only be used for analysis of sulfide at concentrations in excess of one milligram per liter.

(F) *Sample Preservation and Holding Times.* Criteria for sample preservation and sample holding times were requested by several commenters. The reference for sample preservation and holding time criteria applicable to the Table I parameters is given in footnote (1) of Table I.

(G) *Alternate Test Procedures.* Comments pertaining to § 136.4, Application for Alternate Test Procedures, included objections to various obstacles within

these procedures for expeditious approval of alternate test procedures. Four analytical instrument manufacturers commented that by limiting of application for review and/or approval of alternate test procedures to NPDES permit holders, § 136.4 became an impediment to the commercial development of new or improved measurement devices based on new measurement principles. Applications for such review and/or approval will now be accepted from any person. The intent of the alternate test procedure is to allow the use of measurement systems which are known to be equivalent to the approved test procedures in waste water discharges.

Applications for approval of alternate test procedures applicable to specific discharges will continue to be made only by NPDES permit holders, and approval of such applications will be made on a case-by-case basis by the Regional Administrator in whose Region the discharge is made.

Applications for approval of alternate test procedures which are intended for nationwide use can now be submitted by any person directly to the Director of the Environmental Monitoring and Support Laboratory in Cincinnati. Such applications should include a complete methods write-up, any literature references, comparability data between the proposed alternate test procedure and those already approved by the Administrator. The application should include precision and accuracy data of the proposed alternate test procedure and data confirming the general applicability of the test procedure to the industrial categories of waste water for which it is intended. The Director of the Environmental Monitoring and Support Laboratory, after review of submitted information, will recommend approval or rejection of the application to the Administrator, or he will return the application to the applicant for more information. Approval or rejection of applications for test procedures intended for nationwide use will be made by the Administrator, after considering the recommendation made by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati. Since the Agency considers these procedures for approval of alternate test procedures for nationwide use to be interim procedures, we will welcome suggestions for criteria for approval of alternate test procedures for nationwide use. Interested persons should submit their written comments in triplicate on or before June 1, 1977 to: Dr. Robert B. Medz, Environmental Protection Technologist, Monitoring Quality Assurance Standardization, Office of Monitoring and Technical Support (RD-680), Environmental Protection Agency, Washington, D.C. 20460.

(H) *Freedom of Information.* A copy of all public comments, an analysis by parameter of those comments, and documents providing further information on the rationale for the changes made in the final regulation are available for inspection and copying at the Environmental Protection Agency Public Information Reference Unit, Room 2922,

Waterside Mall, 401 M Street, SW., Washington, D.C. 20460, during normal business hours. The EPA information regulation 40 CFR 2 provides that a reasonable fee may be charged for copying such documents.

Effective date: These amendments become effective on April 1, 1977.

Dated: November 19, 1976.

JOHN QUARLES,
Acting Administrator,
Environmental Protection Agency.

Chapter I, Subchapter D, of Title 40, Code of Federal Regulations is amended as follows:

1. In § 136.2, paragraphs (f), (g), and (h) are amended to read as follows:

§ 136.2 Definitions.

(f) "Standard Methods" means *Standard Methods for the Examination of Water and Waste Water*, 14th Edition, 1976. This publication is available from the American Public Health Association, 1015 18th Street, N.W., Washington, D.C. 20036.

(g) "ASTM" means *Annual Book of Standards, Part 31, Water*, 1975. This publication is available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

(h) "EPA Methods" means *Methods for Chemical Analysis of Water and Waste*, 1974. Methods Development and Quality Assurance Research Laboratory,

National Environmental Research Center, Cincinnati, Ohio 45268; U.S. Environmental Protection Agency, Office of Technology Transfer, Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268. This publication is available from the Office of Technology Transfer.

2. In § 136.3, the second sentence of paragraph (b) is amended, and a new paragraph (c) is added to read as follows:

§ 136.3 Identification of test procedures.

(b) . . . Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator or the Director upon the recommendation of the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(c) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring and Support Laboratory, Cincinnati, additional alternate test procedures for nationwide use.

3. Table I of § 136.3 is revised by listing the parameters alphabetically; by adding 44 new parameters; by adding a fourth column under references listing equivalent United States Geological Survey methods; by adding a fifth column under references listing miscellaneous equivalent methods; by deleting footnotes 1 through 7 and adding 24 new footnotes, to read as follows:

TABLE I.—List of approved test procedures¹

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS 1975 methods ²	
1. Acidity, as CaCO ₃ , milligrams per liter.	Electrometric end point (pH of 8.2) or phenolphthalein end point.	1	273(41)	116	40	³ (607)
2. Alkalinity, as CaCO ₃ , milligrams per liter.	Electrometric titration (only to pH 4.5) manual or automated, or equivalent automated methods.	3	278	111	41	⁴ (607)
3. Ammonia (as N), milligrams per liter.	Manual distillation ⁵ (at pH 9.5) followed by nesslerization, titration, electrode. Automated phenolate.	159	410	237	116	³ (614)
		165	412			
		168	616			
BACTERIA						
4. Coliform (fecal) ⁶ , number per 100 ml.	MPN; ⁸ membrane filter.		922			
5. Coliform (fecal) ⁶ in presence of chlorine, number per 100 ml.	do. ⁸		937		⁷ (45)	
6. Coliform (total), ⁴ number per 100 ml.	do. ⁸		922			
7. Coliform (total) ⁴ in presence of chlorine, number per 100 ml.	MPN; ⁸ membrane filter with enrichment.		928, 937		⁷ (35)	
8. Fecal streptococci, ⁴ number per 100 ml.	MPN; ⁸ membrane filter; plate count.		916			
9. Benzidine, milligrams per liter.	Oxidation—colorimetric ⁹ .		944		⁷ (50)	
10. Biochemical oxygen demand, 5-d (BOD ₅), milligrams per liter.	Winkler (Azide modification) or electrode method.		947			
11. Bromide, milligrams per liter.	Titrimetric, iodine-iodate.	14	543	823	58	
12. Chemical oxygen demand (COD), milligrams per liter.	Dichromate reflux.	20	550	872	124	⁸ (610)
13. Chloride, milligrams per liter.	Silver nitrate; mercuric nitrate; or automated colorimetric-ferriyanide.		303	267		⁸ (17)
		29	304	265		⁸ (615)
		31	613		¹¹ (46)	

See footnotes at end of table.

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Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods ¹	
14. Chlorinated organic compounds (except pesticides), milligrams per liter.	Gas chromatography ¹¹					
15. Chlorine—total residual, milligrams per liter.	Iodometric titration, amperometric or starch-iodine end-point; DPD colorimetric or titrimetric methods (these last 2 are interim methods pending laboratory testing).	35	318 322 332 320	278		
16. Color, platinum cobalt units or dominant wave length, hue, luminance, purity.	Colorimetric; spectrophotometric; or ADMI procedure. ¹²	36 39	64 66		82	
17. Cyanide, total, ¹⁴ milligrams per liter.	Distillation followed by silver nitrate titration or pyridine pyrazolone (or barbituric acid) colorimetric.	40	361	503	85	¹⁴ (22)
18. Cyanide amenable to chlorination, milligrams per liter.	do.....	49	376	505		
19. Dissolved oxygen, milligrams per liter.	Winkler (Azide modification) or electrode method.	51 56	443 450	368	126	¹ (609)
20. Fluoride, milligrams per liter.	Distillation ¹ followed by ion electrode; SPADNS; or automated complexone.	65 59 61 68 70	391 393 306 306	307 306	93	
21. Hardness—Total, as CaCO ₃ , milligrams per liter.	EDTA titration; automated colorimetric; or atomic absorption (sum of Ca and Mg as their respective carbonates).	68 70	202	161	94	¹ (617)
22. Hydrogen ion (pH), pH units.	Electrometric measurement.	239	460	178	129	¹ (606)
23. Kjeldahl nitrogen (as N), milligrams per liter.	Digestion and distillation followed by nesslerization, titration, or electrode; automated digestion automated phenolate.	175 165 182	437		122	¹ (612)
METALS						
24. Aluminum—Total, milligrams per liter.	Digestion ¹¹ followed by atomic absorption ¹⁴ or by colorimetric (Eriochrome Cyanine R).	92	152 171		¹¹ (19)	
25. Aluminum—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced methods for total aluminum.					
26. Antimony—Total, milligrams per liter.	Digestion ¹¹ followed by atomic absorption. ¹⁴	94				
27. Antimony—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total antimony.					
28. Arsenic—Total, milligrams per liter.	Digestion followed by silver diethyldithiocarbamate; or atomic absorption. ^{14 18}	9 96	285 283 150		¹¹ (31) ¹¹ (37)	
29. Arsenic—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total arsenic.					
30. Barium—Total, milligrams per liter.	Digestion ¹¹ followed by atomic absorption. ¹⁴	97	152		52	
31. Barium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total barium.					
32. Beryllium—Total, milligrams per liter.	Digestion ¹¹ followed by atomic absorption ¹⁴ or by colorimetric (Aluminon).	99	152 177		53	
33. Beryllium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total beryllium.					
34. Boron—Total, milligrams per liter.	Colorimetric (Curcumin)....	13	287			
35. Boron—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total boron.					
36. Cadmium—Total, milligrams per liter.	Digestion ¹¹ followed by atomic absorption ¹⁴ or by colorimetric (Dithizone).	101	148 182	345	62 ¹ (619) ¹⁸ (27)	
37. Cadmium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total cadmium.					
38. Calcium—Total, milligrams per liter.	Digestion ¹¹ followed by atomic absorption; or EDTA titration.	103	148 189	345	66	
39. Calcium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total calcium.					
40. Chromium VI, milligrams per liter.	Extraction and atomic absorption; colorimetric (Diphenylcarbazide).	89, 105	192		76 75	
41. Chromium VI—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for chromium VI.					
42. Chromium—Total, milligrams per liter.	Digestion ¹¹ followed by atomic absorption ¹⁴ or by colorimetric (Diphenylcarbazide).	105	148 192	345 286	76 77	¹ (619)
43. Chromium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total chromium.					

See footnotes at end of table.

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Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods ²	
44. Cobalt—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁴	107	148	345	80	¹⁵ (37)
45. Cobalt—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total cobalt.					
46. Copper—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Neocuproine).	108	148 196	345 293	83 ³ (619) ¹⁶	(37)
47. Copper—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total copper.					
48. Gold—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁹					
49. Iridium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁹					
50. Iron—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Phenanthroline).	110	148 208	345 326	102	⁴ (619)
51. Iron—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total iron.					
52. Lead—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Dithizone).	112	148 215	345	105	⁴ (619)
53. Lead—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total lead.					
54. Magnesium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption; or gravimetric.	114	148 221	345	109	⁴ (619)
55. Magnesium—Dissolved milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total magnesium.					
56. Manganese—Total milligrams per liter.	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Persulfate or periodate).	116	148 225, 227	345	111	⁴ (619)
57. Manganese—Dissolved milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total manganese.					
58. Mercury—Total, milligrams per liter.	Flameless atomic absorption.	118	156	338	¹¹ (51)	
59. Mercury—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total mercury.					
60. Molybdenum—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁴	139		350		
61. Molybdenum—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total molybdenum.					
62. Nickel—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Heptoxime).	141	148	345	115	
63. Nickel—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total nickel.					
64. Osmium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁹					
65. Palladium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁹					
66. Platinum—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁹					
67. Potassium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption, colorimetric (Cobaltinitrite), or by flame photometric.	143	235 234	403	134	⁴ (620)
68. Potassium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total potassium.					
69. Rhodium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁹					
70. Ruthenium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁹					
71. Selenium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption. ¹⁹	145	159			
72. Selenium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total selenium.					
73. Silica—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by colorimetric (Molybdosilicate).	274	487	398	139	
74. Silver—Total, ²⁰ milligrams per liter.	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Dithizone).	146	148 243		142 ⁴ (619) ¹⁹ (37)	
75. Silver—Dissolved, ²⁰ milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total silver.					
76. Sodium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption or by flame photometric.	147	250	403	143	⁴ (621)
77. Sodium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total sodium.					

See footnotes at end of table.

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Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods ¹	
78. Thallium—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption. ¹⁴	149				
79. Thallium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total thallium.					
80. Tin—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption. ¹⁴	150			¹¹ (65)	
81. Tin—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total tin.					
82. Titanium—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption. ¹⁴	151				
83. Titanium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total titanium.					
84. Vanadium—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption ¹⁴ or by colorimetric (Gallic acid).	153	152	441	¹¹ (67)	
85. Vanadium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total vanadium.					
86. Zinc—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption ¹⁴ or by colorimetric (Dithizone).	155	148 265	345	159	³ (619) ¹⁰ (37)
87. Zinc—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total zinc.					
88. Nitrate (as N), milligrams per liter.	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction. ²¹	201 197 207	423 427 620	358	119	³ (614) ¹⁰ (28)
89. Nitrate (as N), milligrams per liter.	Manual or automated colorimetric (Diazotization).	215	434		121	
90. Oil and grease, milligrams per liter.	Liquid-liquid extraction with trichloro-trifluoroethane-gravimetric.	229	515			
91. Organic carbon; total (TOC), milligrams per liter.	Combustion—Infrared method. ²²	236	532	467	²² (4)	
92. Organic nitrogen (as N), milligrams per liter.	Kjeldahl nitrogen minus ammonia nitrogen.	175, 159	437		122	³ (612, 614)
93. Orthophosphate (as P), milligrams per liter.	Manual or automated ascorbic acid reduction.	249	481	384	131	³ (621)
94. Pentachlorophenol, milligrams per liter.	Gas chromatography ²³	256	621			
95. Pesticides, milligrams per liter.	do. ²²		555	529	²² (24)	
96. Phenols, milligrams per liter.	Colorimetric, (AAAP)	241	582	545		
97. Phosphorus (elemental), milligrams per liter.	Gas chromatography ²³					
98. Phosphorus; total (as P), milligrams per liter.	Persulfate digestion followed by manual or automated ascorbic acid reduction.	249 256	476, 481 624	384	133	³ (621)
RADIOLOGICAL						
99. Alpha—Total, pCi per liter.	Proportional or scintillation counter.		648	591 ¹¹ ²⁵ (75+78)		
100. Alpha—Counting error, pCi per liter.	do.		648	594	¹¹ (79)	
101. Beta—Total, pCi per liter.	Proportional counter.		648	601 ¹¹ ²⁵ (75+78)		
102. Beta—Counting error, pCi per liter.	do.		648	606	¹¹ (79)	
103. (a) Radium—Total, pCi per liter.	do.		661	661		
(b) ²²⁶ Ra, pCi per liter.	Scintillation counter.		607		¹¹ (81)	
RESIDUE						
104. Total, milligrams per liter.	Gravimetric, 103 to 105° C.	270	91			
105. Total dissolved (filterable), milligrams per liter.	Glass fiber filtration, 180° C.	266	92			
106. Total suspended (nonfilterable), milligrams per liter.	Glass fiber filtration, 103 to 105° C.	268	94			
107. Settleable, milliliters per liter or milligrams per liter.	Volumetric or gravimetric.		95			
108. Total volatile, milligrams per liter.	Gravimetric, 550° C.	272	95			
109. Specific conductance, micro-mhos per centimeter at 25° C.	Wheatstone bridge conductivity.	275	71	120	148	³ (606)
110. Sulfate (as SO ₄), milligrams per liter.	Gravimetric; turbidimetric; or automated colorimetric (barium chloranilate).	277 279	493 496	424 425		³ (624) ³ (623)
111. Sulfide (as S), milligrams per liter.	Titrimetric—iodine for levels greater than 1 mg per liter; Methylene blue photometric.	284	505		154	
112. Sulfite (as SO ₃), milligrams per liter.	Titrimetric, iodine-iodate.	285	508	435		
113. Surfactants, milligrams per liter.	Colorimetric (Methylene blue).	157	600	444	¹¹ (11)	
114. Temperature, degrees C.	Calibrated glass or electrometric thermometer.	286	125		³ (31)	
115. Turbidity, NTU.	Nephelometric.	295	132	223	156	

¹ Recommendations for sampling and preservation of samples according to parameter measured may be found in "Methods for Chemical Analysis of Water and Wastes, 1974" U.S. Environmental Protection Agency, table 2, pp. viii-iii.

² All page references for USGS methods, unless otherwise noted, are to Brown, E., Skougstad, M. W., and Fishman, M. J., "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A1, (1970).

³ EPA comparable method may be found on indicated page of "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 12th ed. (1975).

⁴ Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

⁵ The method used must be specified.

⁶ The 5 tube MPN is used.

⁷ Slack, K. V. and others, "Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples: U.S. Geological Survey Techniques of Water-Resources Inv. book 5, ch. A4 (1973)."

⁸ Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.

⁹ Adequately tested methods for benzidine are not available. Until approved methods are available, the following interim method can be used for the estimation of benzidine: (1) "Method for Benzidine and Its Salts in Wastewaters," available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

¹⁰ American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, N.Y. 10018.

¹¹ Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey for Analysis of Wastewaters," (1976) open-file report 76-177.

¹² Procedures for pentachlorophenol, chlorinated organic compounds, and pesticides can be obtained from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

¹³ Color method (ADMI procedure) available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

¹⁴ For samples suspected of having thiocyanate interference, magnesium chloride is used as the digestion catalyst. In the approved test procedure for cyanides, the recommended catalysts are replaced with 20 ml of a solution of 500 g/l magnesium chloride (MgCl₂·6H₂O). This substitution will eliminate thiocyanate interference for both total cyanide and cyanide amenable to chlorination measurements.

¹⁵ For the determination of total metals the sample is not filtered before processing. Because vigorous digestion procedures may result in a loss of certain metals through precipitation, a less vigorous treatment is recommended as given on p. 83 (4.1.4) of "Methods for Chemical Analysis of Water and Wastes" (1974). In those instances where a more vigorous digestion is desired the procedure on p. 82 (4.1.3) should be followed. For the measurement of the noble metal series (gold, iridium, osmium, palladium, platinum, rhodium and ruthenium), an aqua regia digestion is to be substituted as follows: Transfer a representative aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated redistilled HNO₃. Place the beaker on a steam bath and evaporate to dryness. Cool the beaker and cautiously add a 5 ml portion of aqua regia. (Aqua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO₃.) Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 50 min. Remove cover and evaporate to dryness. Cool and take up the residue in a small quantity of 1:1 HCl. Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

¹⁶ As the various furnace devices (flameless AA) are essentially atomic absorption techniques, they are considered to be approved test methods. Methods of standard addition are to be followed as noted in p. 78 of "Methods for Chemical Analysis of Water and Wastes," 1974.

¹⁷ Dissolved metals are defined as those constituents which will pass through a 0.45 µm membrane filter. A pre-filtration is permissible to free the sample from larger suspended solids. Filter the sample as soon as practical after collection using the first 50 to 100 ml to rinse the filter flask. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Discard the portion used to rinse the flask and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO₃ to a pH of 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the samples.

¹⁸ See "Atomic Absorption Newsletter," vol. 13, 75 (1974). Available from Perkin-Elmer Corp., Main Ave., Norwalk, Conn. 06852.

¹⁹ Method available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

²⁰ Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/l and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/l 20 ml of sample should be diluted to 100 ml by adding 40 ml each of 2M Na₂S₂O₃ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/l the recommended method is satisfactory.

²¹ An automated hydrazine reduction method is available from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

²² A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on combustion-methane detection is also acceptable.

²³ Goertitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A3 (1972).

²⁴ R. F. Addison and R. G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," "Journal of Chromatography," vol. 47, No. 3, pp. 421-426, 1970.

²⁵ The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only suspended. Therefore, the 2 results must be added together to obtain "total."

²⁶ Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors. Field Measurement and Data Presentation: U.S. Geological Survey Techniques of Water Resources Inv., book 1 (1975)."

4. In § 136.4, the second sentence of paragraph (c) is amended by deleting the word "subchapter" immediately following the phrase "procedure under this" and immediately preceding the word "shall" and replaced with the phrase "paragraph c;" and § 136.4 is amended by adding a new paragraph (d) to read as follows:

§ 136.4 Application for alternate test procedures.

(c) * * * Any application for an alternate test procedure under this paragraph (c) shall: * * *

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. Any application for an alter-

nate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

§ 136.5 [Amended]

5. In § 136.5, paragraph (a) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "test procedure" and before the period that ends the paragraph.

6. In § 136.5, paragraph (b) is amended by inserting in the first sentence the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "such application" and immediately before the comma. The second sentence of paragraph (b) is amended by deleting the phrase "Methods Development and Quality Assurance Research Laboratory" immediately after the phrase "State Permit Program and to the Director of the" at the end of the sentence, and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

7. In § 136.5, paragraph (c) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the phrase "application for an alternate test procedure" and immediately before the comma; and by deleting the phrase "Methods Development and Quality Assurance Research Laboratory" immediately after the phrase "application to the Director of the" and immediately before the phrase "for review and recommendation" and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

8. In § 136.5, the first sentence of paragraph (d) is amended by inserting the phrase, "proposed by the responsible person or firm making the discharge," immediately after the phrase, "application for an alternate test procedure," and immediately before the comma.

The second sentence of paragraph (d) is amended by deleting the phrase, "Methods Development and Quality Assurance Research Laboratory," immediately after the phrase, "to the Regional Administrator by the Director of the," and immediately preceding the period ending the sentence and inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

The third sentence of paragraph (d) is amended by deleting the phrase, "Methods Development and Quality Assurance Research Laboratory," immediately after the phrase, "forwarded to the Director," and immediately before the second comma and by inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

9. Section 136.5 is amended by the addition of a new paragraph (e) to read as follows:

RULES AND REGULATIONS

§ 136.5 Approval of alternate test procedures.

(e) Within ninety days of the receipt by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati of an application for an alternate test procedure for nationwide use, the Director of the Environmental Monitoring and Support Laboratory, Cincinnati shall notify the applicant of his recommendation to the Administrator to approve or reject the application, or shall specify additional information which is required to determine whether to approve the proposed test procedure. After such notification, an alternate method determined by the Administrator to satisfy the applicable requirements of this part shall be approved for nationwide use to satisfy the requirements of this subchapter; alternate test procedures determined by the Administrator not to meet the applicable requirements of this part shall be rejected. Notice of these determinations shall be submitted for publication in the FEDERAL REGISTER not later than 15 days after such notification and determination is made.

[FR Doc.76-35032 Filed 11-30-76;8:45 am]

Title 40—Protection of Environment**CHAPTER I—ENVIRONMENTAL
PROTECTION AGENCY****SUBCHAPTER D—WATER PROGRAMS****PART 136—GUIDELINES ESTABLISHING
TEST PROCEDURES FOR THE ANAL-
YSIS OF POLLUTANTS****Amendment of Regulations; Corrections**

In FR Doc. 76-35032 appearing at pages 52780 to 52786 in the FEDERAL REGISTER of Wednesday, December 1, 1976, the following changes should be made:

§ 135.3 [Amended]

1. On Page 52783, for parameter number 62, Nickel—Total, add "232" to the page references in the column under the 14th edition of Standard Methods opposite the colorimetric method designation.

2. On page 52784, for parameter number 89, change the parameter designation from "Nitrate" to "Nitrite."

3. On page 52784, for parameter number 96, Phenols, delete the present method designation, "Colorimetric, (4AAP)," and replace it with the method designation, "Distillation followed by colorimetric, (4AAP)"; delete the page reference in the column under the 14th edition of Standard Methods, "582," and replace it with page number "574".

Dated, January 10, 1977.

WILSON K. TALLEY,
*Assistant Administrator for
Research and Development.*

[FR Doc.77-1463 Filed 1-17-77; 8:45 am]
