

**SUMMARY OF U.S.
EPA-APPROVED METHODS,
STANDARD METHODS, AND
OTHER GUIDANCE FOR 301(h)
MONITORING VARIABLES**

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INTRODUCTION

Monitoring programs for 301(h) dischargers should provide data with which to evaluate the impact of the modified discharge on marine biota, demonstrate compliance with applicable water quality standards, and measure toxic substances in the discharge. Thirty-two biological, sediment, and water quality variables may be included in 301(h) monitoring programs to provide such data. The biological and sediment variables are applicable to samples collected from the receiving environment. The water quality variables are applicable to samples collected from both effluent and receiving water.

Collection of high quality data that are comparable among dischargers requires that analytical methods for each monitoring variable follow established protocols. Available methods for each of the 32 variables are discussed in this synopsis and summarized in Table 1. Methods are divided into three categories: U.S. EPA methods, standard methods, and additional methods available in the scientific literature. U.S. EPA methods are divided further into those that have been approved by the agency, those that have been suggested but not approved, and those for which there is an agency guidance document. Standard methods refer exclusively to American Public Health Association (APHA) standard methods (i.e., APHA 1985). Additional methods are found in a variety of documents.

TABLE 1. U.S. EPA-APPROVED METHODS AND GUIDANCE DOCUMENTS
FOR MEASURING BIOLOGICAL, SEDIMENT, AND WATER QUALITY
VARIABLES IN 301(h) MONITORING PROGRAMS

| Category/Variable | U.S. EPA Methods | | | Standard | Additional |
|--------------------------------------|------------------|----------------|----------------|----------|------------|
| | Approved | Suggested | Guidance | Methods | Methods |
| Water Quality Variables | | | | | |
| pH | X | | X | X | |
| Temperature | X | | | X | |
| Turbidity | X | | | X | |
| Total suspended solids | X | | X | X | |
| Settleable solids | X | | | X | |
| Floating particulates | | | | X | |
| Dissolved oxygen (Winkler) | X | | | X | |
| Dissolved oxygen (Probe) | X | | | X | |
| Biochemical oxygen demand | X | | | X | |
| Oil and grease | X | X | X | | |
| Nitrogen (ammonia) | X | X | X | X | X |
| Nitrogen (total Kjeldahl) | X | | X | X | |
| Nitrogen (nitrate-nitrite) | X | | X | X | X |
| Phosphorus | X | X | X | X | |
| Priority pollutant metals | x ^a | x ^b | X | X | |
| Priority pollutant organic compounds | X | | X | | |
| Total and fecal coliform bacteria | | | X | X | |
| Enterococcus bacteria | | | | X | |
| Sediment Variables | | | | | |
| Grain size | | | X | | X |
| Total volatile solids | | | X | | X |
| Total organic carbon | X | | X | | |
| Oil and grease | X | X | X | X | |
| Priority pollutant metals | | x ^c | X | | |
| Priority pollutant organic compounds | | | x ^d | | |

Biological Variables

| | | | |
|--|----|---|----|
| Phytoplankton | X | X | |
| Chlorophyll <u>a</u> | X | X | X |
| Zooplankton | X | X | X |
| Benthic infauna | X | | X |
| Demersal fishes and megainvertebrates | X | | X |
| Fish histopathology | | | xe |
| Bioaccumulation (priority pollutant metals) | | X | X |
| Bioaccumulation (priority pollutant organic compounds) | xf | | X |

a Except asbestos.

b Cyanide only.

c Mercury only.

d Comprehensive unpublished interim guidelines and further guidance in Plumb (1981).

e Field methods only.

f Laboratory estimation of bioconcentration factors only.

Water Quality Variables

pH--

An approved U.S. EPA method (No. 150.1) for pH analysis is given in U.S. EPA (1979). U.S. EPA guidance for determination of pH is described by Plumb (1981). Standard methods are found in Section 423 of APHA (1985).

Temperature--

An approved U.S. EPA method (No. 170.1) for temperature measurements is given in U.S. EPA (1979). Standard methods are found in Section 212 of APHA (1985).

Turbidity--

An approved U.S. EPA method (No. 180.1) for turbidity measurements is given in U.S. EPA (1979). Standard methods are found in Section 214 of APHA (1985).

Total Suspended Solids--

An approved U.S. EPA method (No. 160.2) for total suspended solids analysis is given in U.S. EPA (1979). U.S. EPA guidance for analysis of total solids is provided by Plumb (1981). Standard methods are found in Section 209C of APHA (1985).

Settleable Solids--

An approved U.S. EPA method (No. 160.5) for settleable solids analysis is given in U.S. EPA (1979). Standard methods are found in Section 209E APHA (1985).

Floating Particulates--

No method for the analysis of floating particulates has been approved by the U.S. EPA. A proposed standard method (No. 205A) for the analysis of floating particulates is found in APHA (1985).

Dissolved Oxygen (Winkler Method)--

An approved U.S. EPA method (No. 360.2) for dissolved oxygen analysis using the Winkler Method is given in U.S. EPA (1979). Standard methods are found in Sections 421A-E of APHA (1985).

Dissolved Oxygen (Probe Method)--

An approved U.S. EPA method (No. 360.1) for dissolved oxygen analysis using an oxygen probe is given in U.S. EPA (1979). A standard method is found in Section 421F APHA (1985).

Biochemical Oxygen Demand (BOD)--

An approved U.S. EPA method (No. 405.1) for the analysis of BOD is given in U.S. EPA (1979). Standard methods are found in Section 507 of APHA (1985).

Oil and Grease--

The approved test procedure for oil and grease is U.S. EPA (1979) Method 413.1. An additional procedure (No. 413.2) for low concentrations of oil and grease is also discussed in U.S. EPA (1979). U.S. EPA guidance for analysis of oil and grease is provided by Plumb (1981). Standard methods are described in Section 503 of APHA (1985).

Nitrogen (Ammonia)--

Two U.S. EPA (1979) approved methods (Nos. 350.1 and 350.2) exist for determination of ammonia concentrations. An additional method (No. 350.3)

for potentiometric (ion selective electrode) determination is also discussed by U.S. EPA (1979). Further U.S. EPA guidance is provided by Plumb (1981). Standard methods for ammonia are discussed in Section 416 of APHA (1985). Methods for the determination of ammonia concentrations in marine waters are described in Part II of Strickland and Parsons (1972).

Nitrogen (Total Kjeldahl)--

Approved test procedures for total Kjeldahl nitrogen are U.S. EPA (1979) Methods 351.1, 351.2, 351.3, and 351.4. U.S. EPA guidance is provided by Plumb (1981). Standard methods are described in Section 420 of APHA (1985).

Nitrogen (Nitrate-Nitrite)--

Approved test procedures for analysis of nitrate and nitrite include U.S. EPA (1979) Methods 353.1, 353.2, and 353.3. Further U.S. EPA guidance is provided by Plumb (1981). Standard methods for nitrate and nitrite are discussed in Sections 418 and 419 of APHA (1985). Methods for the determination of nitrate and nitrite concentrations in marine waters are discussed in Part II of Strickland and Parsons (1972).

Phosphorus--

Approved methods (Nos. 365.1, 365.2, and 365.3) for analysis of total phosphorus are described by U.S. EPA (1979). A suggested method (No. 365.4) is also provided in U.S. EPA (1979). Plumb (1981) provides additional U.S. EPA guidance for analysis of phosphates. Standard methods (Nos. 424C, 424F, and 424G) are described in APHA (1985).

Priority Pollutant Metals--

With the exception of asbestos, approved U.S. EPA (1979) test procedures exist for all priority pollutant metals:

- Antimony (Nos. 204.1, 204.2)

- Arsenic (Nos. 206.2, 206.3, 206.4)
- Beryllium (Nos. 210.1, 210.2)
- Cadmium (Nos. 213.1, 213.2)
- Chromium (Nos. 218.1, 218.2, 218.3, 218.4)
- Copper (Nos. 220.1, 220.2)
- Cyanide (Nos. 335.1, 335.2)
- Lead (Nos. 239.1, 239.2)
- Mercury (Nos. 245.1, 245.2)
- Nickel (Nos. 249.1, 249.2)
- Selenium (Nos. 270.2, 270.3)
- Silver (Nos. 272.1, 272.2)
- Thallium (Nos. 279.1, 279.2)
- Zinc (Nos. 289.1, 289.2)

A suggested U.S. EPA (1979) method also exists for total cyanide (No. 335.3). Further U.S. EPA guidance is provided by Plumb (1981). Standard methods for analyses of metals are described in Part 300 of APHA (1985).

Priority Pollutant Organic Compounds--

Approved U.S. EPA (1982) methods exist for all organic priority pollutants (summarized herein by compound class):

- Purgeable halocarbons (No. 601)

- Purgeable aromatics (No. 602)
- Acrolein and acrylonitrile (No. 603)
- Phenols (No. 604)
- Benzidines (No. 605)
- Phthalate esters (No. 606)
- Nitrosamines (No. 607)
- Pesticides and PCBs (No. 608)
- Nitroaromatics and isophorone (No. 609)
- Polynuclear aromatic hydrocarbons (No. 610)
- Haloethers (No. 611)
- Chlorinated hydrocarbons (No. 612)
- 2,3,7,8-Tetrachlorodibenzo-p-dioxin (No. 613)
- Purgeables (No. 624)
- Base/Neutrals, Acids, and Pesticides (No. 625).

Additional U.S. EPA guidance is provided by Plumb (1981).

Total and Fecal Coliform Bacteria--

No method for the analysis of total and fecal coliform bacteria concentrations has been approved by the U.S. EPA. U.S. EPA guidance is available in Part III of Bordner and Winter (1978). Standard methods for the most

probable number (MPN) method are presented in Section 908 of APHA (1985). Standard methods for the membrane filter method are presented in Section 909 of APHA (1985).

Enterococcus Bacteria--

No method for the analysis of enterococcus bacteria concentrations has been approved by the U.S. EPA. U.S. EPA guidance is available in Part III of Borner and Winter (1978). Standard methods for the most probable number (MPN) method are presented in Section 908 of APHA (1985). Standard methods for the membrane filter method are presented in Section 909 of APHA (1985).

Sediment Analyses

Grain Size--

No method for the analysis of sediment grain size has been approved by the U.S. EPA. Particles having a minimum diameter of 0.63 um (i.e., very fine sand) are analyzed using the dry sieve method. Procedures for the dry sieve analysis are found in Folk (1968), Buchanan (1984), and Plumb (1981). Analyses for particles <63 um diameter (i.e., silts and clays) may be conducted by pipet, hydrometer, or Coulter counter. Tetra Tech (1985b) recommends the pipet method, details of which are found in Plumb (1981).

Total Volatile Solids--

No method for the analysis of total volatile solids has been approved by the U.S. EPA. Guidance for estimating total volatile solids by combustion is given in Buchanan (1984) and Plumb (1981).

Total Organic Content (TOC)--

An approved U.S. EPA method (No. 415.1) for TOC is given in U.S. EPA (1979). Additional U.S. EPA guidance is found in Plumb (1981).

Oil and Grease--

An approved U.S. EPA method (No. 413.1) for the analysis of oil and grease is given in U.S. EPA (1979). A suggested method (No. 413.2) is also given in U.S. EPA (1979). U.S. EPA guidance is provided by Plumb (1981). Standard methods for analyses of oil and grease are found in Section 503 of APHA (1985).

Priority Pollutant Metals--

There are no U.S. EPA approved methods for analysis of priority pollutant metals in sediments. A suggested test procedure (No. 245.5) for the analysis of mercury in sediments is described by U.S. EPA (1979). The U.S. EPA (1979) also indicates that the approved method (atomic absorption spectrophotometry) most commonly used in analysis of effluent and receiving waters may be applied to metals in solids that are solubilized by some form of sample processing. U.S. EPA guidance for analysis of metals in sediments is provided by Plumb (1981).

Priority Pollutant Organic Compounds--

There are no U.S. EPA approved methods for analysis of organic priority pollutants in sediments. Draft interim guidelines for analysis of organic contaminants in sediments are being developed by personnel with the U.S. EPA Environmental Monitoring Laboratory in Cincinnati, OH, but have yet to be published (Longbottom, J., 22 April 1985, personal communication). Additional U.S. EPA guidance for analysis of organic contaminants is described by Plumb (1981).

Biological Variables

Phytoplankton--

Methods for the collection and analysis of phytoplankton samples have not been approved by the U.S. EPA. U.S. EPA guidance for sampling phytoplankton

Stofan and Grant (1978). This document includes an extensive papers and reports that are pertinent to the collection of phytoplankton samples. Standard methods for phytoplankton and in Section 1002 of APHA (1985).

on the collection and analysis of chlorophyll a samples have been provided by the U.S. EPA. Tetra Tech (1985b) recommends that concentrations be determined using the fluorometric method or spectrophotometric method because the former technique (i.e., requires less sample, and can be used for in vivo measurements). U.S. EPA guidance for fluorometric determinations of chlorophyll a is found in Stofan and Grant (1978). Chlorophyll a determination methods using the fluorometric method are described in Sections IV and V and in Parsons (1972) and as Standard Method 100262 in APHA (1985).

on collection and analysis of zooplankton samples have not been provided by the U.S. EPA. U.S. EPA guidance for quantitative sampling in baseline and monitoring programs is described by Jacobs (1984). Standard methods for sampling zooplankton populations are found in Section 1002 of APHA (1985). Additional methods for sampling zooplankton are provided by UNESCO (1974, 1976).

on the collection and analysis of benthic infauna samples have not been provided by the U.S. EPA. U.S. EPA guidance for the collection of subtidal benthic samples is given in Swartz (1978). Guidance is provided by McIntyre et al. (1984), Gamble (1984), Holme (1984), and Tetra Tech (1985b). U.S. EPA guidance on collection and processing of intertidal benthic samples is found in Section 1002 of APHA (1985).

in Gordon and Kemp (1978). Sampling and transecting techniques for coral reef habitats are described by Reed (1980) and Dodge et al. (1982).

Demersal Fishes and Megainvertebrates--

There are no U.S. EPA approved methods for analysis of demersal fish and epibenthic invertebrate communities. U.S. EPA guidance for sampling demersal fishes and epibenthic megainvertebrates is provided by Mearns and Allen (1978). Additional procedures describing appropriate sampling methods for demersal fishes are provided by Saville (1977) and Hayes (1983). Methods for visual censusing of reef-dwelling fish communities are discussed by Goldman and Talbot (1976), Talbot et al. (1978), Brock et al. (1979), Bohnsack and Talbot (1980), Sale (1980), and Ogden and Ebersole (1981).

Fish Histopathology--

Methods for measuring histopathological conditions in fishes have not been approved by the U.S. EPA. Field methods for examination of fish and for collection of blood and tissue samples are found in Strange (1983). However, this work is a general discussion emphasizing parasitism and grossly visible abnormalities. It provides no guidance on microscopic analyses of blood or tissues for pathological conditions, nor does it discuss in detail the pathological conditions that occur in fishes. At present, no detailed guidance is available on laboratory methods for histopathological determinations in fishes. Furthermore, the terminology for histopathological conditions has not been standardized.

Bioaccumulation (Priority Pollutant Metals)--

Methods to assess bioaccumulation of priority pollutant metals have not been approved by the U.S. EPA. Quality control guidelines for measuring tissue concentrations of metals are provided by Tetra Tech (1985b). Standard methods for extraction, digestion, and measurement of metals in tissue samples are described in Part 300 of APHA (1985).

Bioaccumulation (Priority Pollutant Organic Compounds)--

Methods to assess bioaccumulation of priority pollutant organic compounds have not been approved by the U.S. EPA. Quality control guidelines for measuring tissue concentrations of organic contaminants are provided by Tetra Tech (1985b). U.S. EPA laboratory methods for measuring bioconcentration factors are described by Veith et al. (1979a,b; 1980) and are evaluated by Kosian et al. (1981). These studies were developed using freshwater fishes and may not be applicable to marine organisms (Tetra Tech 1985a).

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