

**MARYLAND DEPARTMENT OF THE ENVIRONMENT
WATER & SCIENCE ADMINISTRATION**

A. Toxic Pollutant Monitoring Protocol and Reporting Requirements for Toxic Chemical Testing Analytical Data

- It is the responsibility of the permittee to assure that applicable sampling and analytical methodology is used to fulfill permit requirements. The Department strongly suggests that the permittee discuss these requirements with an environmental analytical laboratory familiar with wastewater methods as approved by EPA and listed in 40 CFR Part 136 or other methods approved by EPA for wastewater.
- All testing of wastewater must be performed using sampling and analytical methods for wastewater found in the most recent 40 CFR Part 136 (found at the following website: http://bit.ly/40CFR_Part136).
- To determine compliance with numerical permit limitations, unless otherwise specified in the permit, the analytical methods shall include:
 - a. Any approved method with a Method Detection Level (MDL) adequate to detect concentrations of at least one-tenth the level of the permit limitation and the laboratory's limit of quantitation (LOQ) or Minimum Level must be below the permit limitation or
 - b. If there is no approved method sensitive to at least one-tenth of the permit limitation, then the most sensitive method approved in 40 CFR Part 136 or other method approved by EPA for wastewater is required with an appropriate Minimum Level.
- To fulfill monitoring only requirements or for special studies, the method with the lowest achievable detection level and minimum level must be used unless the Department determines an alternate detection level and minimum level is adequate for the particular purpose of the monitoring event. If it is known that the concentration of the target analyte present is above the most sensitive method, then less sensitive methods that can quantify below the critical value or permit limitation can be utilized. These methods must be approved by 40 CFR Part 136 or are other methods approved by EPA for wastewater analysis. However, if after analysis, the target analyte is not detected, the sample must be reanalyzed using more sensitive 40 CFR Part 136 or other methods approved by EPA for wastewater. The testing laboratory's limit of Quantitation (LOQ) must also be low.
- If the desired LOQ using the above protocol cannot be attained due to matrix interference, current QA/QC protocols to verify that claim will still be in effect.
- The MDL is defined as the minimum concentration that can be measured and reported with 99 percent confidence that the concentration is greater than zero, but the exact concentration cannot be reliably quantified.
- Quantitation Limit or LOQ is defined as the lowest concentration of an analyte that can be reliably measured within specified limits of precision and accuracy for a specific laboratory analytical method and that takes into account analytical adjustments made during sample preparation and analysis.

B. Reporting Requirements For Toxic Chemical Testing Analytical Data

Revised December 2, 2019: RL Table Updated; **Revised September 10, 2019:** RLs Table Updated; **Revised August 14, 2018:** MDL revision 1.11 to revisions 2. 40 CFR link revised; **Revised September 18, 2017:** Added Nonylphenol and lowered LOQ for Cu and Acrolein; **Revised 5/18/2011:** Modified PP list to conform with 40 CFR. 423; **Revised April 12, 2011:** PCB by 1668 removed; **Revised November 11, 2010:** Clarify detection limit with the addition of LOQ, Added PCB by 1668 protocol, lowered LOQs for pollutants to WQ criteria; **Revised April 14, 2009:** Teflon and glass sampling req.'s added for semi-volatiles; **Revised March 17, 2009:** Removed HCA; **Revised July 2, 2007:** Asbestos added, and 2,3,7,8-TCDD footnote added.

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- **Background:** The Maryland Department of the Environment (MDE), Water & Science Administration (WSA) has compiled the following guidelines for reporting analytical data from priority pollutant analyses. These guidelines were formulated in an effort to standardize evaluations of priority pollutant data submitted to the Department. The following reporting requirements were compiled directly from quality control procedures required in the individual analytical methods, information obtained from EPA, and best professional judgment by MDE staff.
- **Toxic Chemical Testing Reporting Requirements:** The results from toxic chemical testing shall be reported in a concise easily understood manner. The report shall include a simple table of contents, sample collection/preservation documentation, a tabular summary of analytical data, and quality assurance data.. The information to be included in the report as described below.

I. Sample Collection & Preservation Documentation

1. Chain of custody forms, which shall include the following information:
 - sample source (e.g. name of facility)
 - sample collection location (e.g. Outfall 001A), date & time
 - sampling method (e.g. composite or grab)
 - signatures of persons collecting, transporting, and receiving samples
 - a description of sample condition upon arrival at the laboratory
2. A description of sample preservation and treatment techniques (i.e., All samples must be refrigerated during holding and transport to the testing laboratory and chemical preservation must be performed at the time of collection. To ensure that preservation required under 40 CFR is followed, the collector and the laboratory must track and document the temperature and chemical preservation of the samples. The laboratory must verify and document all chemical preservation such as pH adjustment and the temperature of the samples upon arrival at the lab).
3. All semi-volatile composite sampling must be collected using Teflon tubing and composited into glass containers.

II. Analytical Results

- Analytical results and quantitation levels achieved, not method detection levels for each parameter or analyte. The reporting limit shall not be lower than the lowest calibration standard analyzed with the run. If the target analyte is detected at a concentration or level between the MDL and the LOQ/ Reporting limit, the estimated results must be reported with a qualifier.
- Report date and time of extraction of primary sample, QC samples and other samples associated with primary sample analytical run, i.e. surrogates. (for organic tests)
- Report date and time of sample analysis

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- Report analytical methods used (the method number and text reference including edition is sufficient)
- Report any deviations taken from analytical methods
- Report all steps and target sample manipulations taken to overcome matrix interferences e.g. dilution, and amount, chemical additions, manual integrations

III. Quality Assurance Data:

- Quality assurance data is collected to document the accuracy and precision of the analytical method. Quality assurance data, which are required by the analytical methods, are collected by the analytical laboratory at method or regulatory specified frequencies, whichever is more frequent. (e.g. usually with each set of samples analyzed or every 10% of samples analyzed).
- Annual method detection limit (MDL) studies must be conducted to establish the laboratory's ability to detect analytes at low levels. The laboratory must determine the MDL in accordance with the procedure in 40 CFR 136, Appendix B (Revision 2) using the apparatus, reagents, and standards that will be used in the practice of this method.
- The laboratory must have documents verifying an initial demonstration of capability for each reported analyte and records documenting the proficiency of each analyst conducting the testing.
- The analysis of duplicates, matrix spikes (MS) and matrix spike duplicates (MSD) are required to demonstrate accuracy and precision and to monitor matrix interferences.
- The analyses of blanks are required to demonstrate acceptable levels of contamination.
- The laboratory must demonstrate on an ongoing basis that the analytical system is in control through the analysis of reagent blanks (LRB), laboratory fortified blanks (LFB) and quality control samples (QCS). The laboratory must analyze at least one LFB with each batch of samples and calculate accuracy as percent recovery. The laboratory must use LFB analyses data to assess the performance of the laboratory against established control limits of preferably 90-110%. The control limits will vary by analyte and test method but should fall within the acceptance range specified in the specific test method and or assigned value of the QC samples. QCS show the ability of the laboratory to report analytical results of known documented quality. QCS and LFB results falling outside of the acceptance criteria is sufficient reason to reject sample results from that run. Samples must then be reanalyzed.

IV. Organic Tests

The following information is required for each organic analysis (e.g. volatiles, semi-volatiles, pesticides / Aroclors PCBs)

- Trip, Field and Method blank results¹ (i.e. Method blank: An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The method blank is used to determine if target analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

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Method Blanks must be analyzed at the original concentration with no dilution factors. If method blanks show detectable results of the target analyte, then the sample values from the runs should be flagged or qualified. Field blank is an aliquot of reagent water or other reference matrix that is placed in a sample container in the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample. A trip blank is a sample of analyte-free reagent water or media collected in the same type of container that is required for the analytical testing of volatile organic compounds and taken from the laboratory or the beginning of the sampler's route to the sampling site and returned to the laboratory unopened.)

- Surrogate spike results and acceptable surrogate spike recovery range² (i.e. analysis of surrogate compounds added to each environmental sample for documentation of instrument response and extraction efficiency).
- Matrix spike & matrix spike duplicate results² to access acceptable recovery range³ (i.e. Matrix spike/matrix spike duplicate (MS/MSD))—An aliquot of an environmental sample to which a known quantity of the method analyte is added in the laboratory and then analyzed in the same manner as the sample. The purpose of the MS/MSD is to determine whether the sample matrix contributes bias to the analytical results.).
- Duplicate analysis of the environmental sample to access the precision of the analytical system.
- Quality control check standard results³ (i.e. EPA/NELAC approved or QCS traceable to NIST standards obtained from a source that is different from the instrument calibration standards).
- Laboratories must minimize manual integration by properly maintaining the instrument, updating retention times, and configuring peak integration parameters, etc.,. On occasion manual integration may be necessary due to a variety of matrix effects so each laboratory must have a documented policy or guidelines, which **defines proper methods for performing manual integrations when they are necessary**. The policy must follow analytically and **scientifically sound manual integration practices and detail or specify proper methods for performing manual integrations when they are necessary**.

V. Inorganic Tests

The following information is required for each separate inorganic analysis:

- Equipment, Field and Method blank results²
- MS and MSD results² and acceptable recovery range³ (i.e. Matrix spike/matrix spike duplicate (MS/MSD) — An aliquot of an environmental sample to which a known quantity of the method analyte is added in the laboratory and then analyzed in the same manner as the sample. The purpose of the MS/MSD is to determine whether the sample matrix contributes bias to the analytical results).
- Duplicate analysis of the environmental sample to access the precision of the analytical system.
- Quality control check standard results³ (i.e. EPA/NELAC approved or QCS traceable to NIST standards obtained from a source that is different from the instrument calibration standards).

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Note:

In recognition of the ongoing advances occurring in analytical technology, and the methods update rule (MUR) promulgated by EPA to allow the analyst to overcome sample matrix interferences, the laboratory or analyst is permitted certain options or modifications to improve analysis or lower the costs of testing. These modifications include alternate extraction, concentration, cleanup procedures, and changes in columns, instrumentation and detectors. However, alternate analytical or determinative techniques and method procedural changes that degrade method performance are **not** allowed.

If method modifications are used, those techniques must have an analytical accuracy equal to or better than the accuracy of the techniques in the established methods for the target analytes of interest. Documentation of the equivalency of the modifications as specified by the MUR must be kept on file by the testing laboratory for review by MDE auditors or inspectors.

Footnotes to Sections IV and V:

- ¹ *Method blanks, duplicates, matrix spikes (MS), and matrix spike duplicates (MSD) shall be performed at or above the minimum frequency specified in the test method (e.g. usually 5% -10% of samples analyzed or once per each batch of samples analyzed, whichever is more frequent). If matrix interferences are suspected, it is recommended that recovery rates for the target analytes of these samples are determined with the use of MS and MSD (Sample results shall be qualified by the laboratory based upon these results or reanalysis may be necessary).*
- ² *The source of acceptable matrix spike and surrogate spike recovery ranges shall also be stated (e.g. from EPA method specifications or from laboratory control charts).*
- ³ *Frequency of analysis of QCS will vary depending on recoveries of matrix and surrogate spikes. The results of QCS that fall outside of the acceptance range may indicate that the laboratory's analytical system is out of control or that the target analytes have been estimated (Sample results shall be qualified by the laboratory based upon these results or reanalysis may be necessary).*

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Table: Required Toxic Chemical Analytes				
Number	Parameter	Cas Number	Minimum Reporting Limit (RL) µg/L, except as noted ^c	
			Discharges to Fresh Water	Discharges to Salt Water
1	Aluminum	7429-90-5	5	5
2	Antimony	7440360	0.4	0.4
3	Arsenic	7440382	1	1
4	Asbestos ^a	1332214	0.2 MFL (million fibers per liter)	0.2 MFL (million fibers per liter)
5	Barium	7440393	10	10
6	Beryllium	7440417	2	2
7	Cadmium	7440439	0.2	0.2
8	Chromium (total)	7440473	1	1
9	Chromium VI	18540299	0.1	0.1
10	Cobalt	7440-48-4	5	5
11	Copper	7440508	2	2
12	Cyanide (total)	57125	5	5
13	Cyanide (free or available)	57125	5	1
14	Iron	7439-89-6	5	5
15	Lead	7439921	1	1
16	Mercury	7439976	0.2	0.2
17	Nickel	7440020	10	10
18	Selenium	7782492	5	5
19	Silver	7440224	1	1
20	Thallium	7440280	0.4	0.4
21	Zinc	7440666	5	5
22	1,1 Dichloroethylene (DCE)	75354	0.7	0.7
23	1,1,1-Trichloroethane (TCA)	71556	0.5	0.5
24	1,1,2,2-Tetrachloroethane	79345	0.3	0.3
25	1,1,2-Trichloroethane	79005	0.5	0.5
26	1,2,4-Trichlorobenzene	120821	0.3	0.3
27	1,2-Dichlorobenzene	95501	0.2	0.2
28	1,2-Dichloroethane	107062	1	1
29	1,2-Dichloropropane	78875	0.5	0.5
30	1,2-Diphenylhydrazine	122667	1	1
31	1,2-Trans-Dichloroethylene	156605	0.5	0.5
32	1,3-Dichlorobenzene	541731	0.5	0.5

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			Discharges to Fresh Water	Discharges to Salt Water
33	1,3-Dichloropropene (1,2-Dichloropropylene)	542756	0.5	0.5
34	1,4-Dichlorobenzene	106467	0.2	0.2
35	2,4,6-Trichlorophenol	88062	3	3
36	2,4-Dichlorophenol	120832	1	1
37	2,4-Dimethylphenol	105679	1	1
38	2,4-Dinitrophenol	51285	3	3
39	2,4-Dinitrotoluene	121142	1	1
40	2,6-Dinitrotoluene	606-20-2	1	1
41	2-Chloronaphthalene	91587	1	1
42	2-Chlorophenol	95578	1	1
43	<i>Phenol</i>	108952	5	5
44	4,6-dinitro-o-cresol (2-Methyl-4,6-dinitrophenol)	534521	5	5
45	Acrylonitrile	107131	5	5
46	Benzene	71432	0.5	0.5
47	Benzidine	92875	5	5
48	Bis(2-Chloroethyl) Ether	111444	1	1
49	Bis2(Chloroisopropyl) Ether	108601	1	1
50	4-chlorophenyl phenyl ether	7005723	1	1
51	4-bromophenyl phenyl ether	101553	1	1
52	Bis(2-chloroethoxy) methane	111911	1	1
53	Bromoform (tribromomethane)	75252	0.5	0.5
54	Carbon tetrachloride	56235	0.5	0.5
55	Chlorobenzene	108907	0.5	0.5
56	Chlorodibromomethane	124481	1	1
57	Chloroethane	75003	0.5	0.5
58	Chloroform	67663	1	1
59	Dichlorobromomethane	75274	0.5	0.5
60	Ethylbenzene	100414	0.5	0.5
61	Hexachlorobenzene	118741	5	5
62	Hexachlorobutadiene	87683	5	5
63	Hexachlorocyclopenta-diene	77474	5	5
64	Hexachloroethane	67721	5	5

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Number	Parameter	Cas Number	Minimum Reporting Limit (RL) µg/L, except as noted ^c	
			Discharges to Fresh Water	Discharges to Salt Water
65	Isophorone	78591	3	3
66	Naphthalene	91203	5	5
67	Methyl bromide (bromomethane)	74839	0.5	0.5
68	Methylene chloride (dichloromethane)	75092	0.5	0.5
69	Methyl chloride (dichloromethane)	74873	0.5	0.5
70	Nitrobenzene	98953	1	1
71	2-nitrophenol	88755	1	1
72	4-nitrophenol	100027	1	1
73	N-Nitrosodimethylamine	62759	3	3
74	N-Nitrosodi-n-Propylamine	621647	1	1
75	N-Nitrosodiphenylamine	86306	1	1
76	Nonylphenol	84852153	6.6	1.7
77	Phenol	108952	5	5
78	Tetrachloroethylene	127184	3	3
79	Toluene	108883	0.5	0.5
80	Trichloroethylene (TCE)	79016	0.5	0.5
81	Vinyl chloride (chloroethylene)	75014	0.5	0.5
82	Trihalomethanes (THM) ^b	<i>See individual compounds</i>	<i>See individual compounds</i>	<i>See individual compounds</i>
83	<i>Acenaphthene</i>	<i>83329</i>	1	1
84	<i>Anthracene</i>	<i>120127</i>	1	1
85	<i>Benzo(a)Anthracene</i> (1,2-benzanthracene)	<i>56553</i>	1	1
86	3,3'-Dichlorobenzidine	91941	5	5
87	Parachlorometa cresol	59507	1	1
88	3,4-Benzofluoranthene (Benzo(b)fluoranthene)	205992	1	1
89	<i>Chrysene</i>	<i>218019</i>	1	1
90	<i>Benzo(k)Fluoranthene</i> (11,12-benzofluoranthene)	<i>207089</i>	1	1
91	<i>Dibenzo(a,h)Anthracene</i> (1,2,5,6-Dibenzanthracene)	<i>53703</i>	1	1

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			Discharges to Fresh Water	Discharges to Salt Water
92	1,12-benzoperylene (Benzo(ghi) perylene)	191242	1	1
93	<i>Fluoranthene</i>	206440	1	1
94	<i>Fluorene</i>	86737	1	1
95	Phenanthrene	85018	1	1
96	<i>Ideno(1,2,3-cd)Pyrene</i> (2,3-o-pheynylene pyrene)	193395	1	1
97	<i>Pyrene</i>	129000	1	1
98	<i>Bis(2-Ethylhexyl) Phthalate</i>	117817	3	3
99	<i>Butylbenzyl Phthalate</i>	85687	3	3
100	<i>Diethyl Phthalate</i>	84662	1	1
101	<i>Dimethyl Phthalate</i>	131113	1	1
102	<i>Di-n-Butyl Phthalate</i>	84742	3	3
103	Di-n-octyl phthalate	117840	3	3
104	<i>4,4'-DDD</i>	72548	0.02	0.02
105	<i>4,4'-DDE</i>	72559	0.02	0.02
106	<i>4,4'-DDT</i>	50293	0.001	0.001
107	<i>Aldrin</i>	309002	3	1.3
108	<i>alpha-BHC</i>	319846	0.02	0.02
109	<i>alpha-Endosulfan</i>	959988	0.056	0.0087
110	<i>Atrazine</i>	319857	3	3
111	<i>beta-BHC</i>	319857	0.02	0.02
112	<i>beta-Endosulfan</i>	33213659	0.056	0.0087
113	<i>Delta BHC</i>	319868	0.02	0.02
114	<i>Carbaryl</i>	63252	2.1	1.6
115	<i>Chlordane</i> (technical mixture and metabolites)	57749	0.0043	0.004
116	<i>Chlorpyrifos</i>	2921882	0.041	0.0056
117	<i>Diazinon</i>	333415	0.17	0.82
118	<i>Dieldrin</i>	60571	0.056	0.0019
119	<i>Endosulfan Sulfate</i>	1031078	0.02	0.02
120	<i>Endrin</i>	72208	0.036	0.0023
121	<i>Endrin Aldehyde</i>	7421934	0.02	0.02
122	<i>gamma-BHC (Lindane)</i>	58899	0.95	0.16

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			Discharges to Fresh Water	Discharges to Salt Water
123	Heptachlor	76448	0.0038	0.0036
124	Heptachlor Epoxide (BHC-hexachlorocyclohexane)	1024573	0.0038	0.0036
125	Total Polychlorinated Biphenyls PCBs ^d	various	0.014	0.02
126	Toxaphene	8001352	0.002	0.002
127	Tributyltin (TBT)		0.072	0.0074
128	Pentachlorophenol (PCP)	87865	8	8
129	2-chloroethyl vinyl ether	110-75-8	2.5	2.5
130	Acrolein	107028	3	3
131	PCB-1242 (Aroclor 1242)	53469219	0.4	0.4
132	PCB-1254 (Aroclor 1254)	11097691	0.4	0.4
133	PCB-1221 (Aroclor 1221)	11104282.	0.4	0.4
134	PCB-1232 (Aroclor 1232)	11141165	0.4	0.4
135	PCB-1248 (Aroclor 1248)	12672296	0.4	0.4
136	PCB-1260 (Aroclor 1260)	11096825	0.4	0.4
137	PCB-1016 (Aroclor 1016)	12674112	0.4	0.4
138	2, 3, 7, 8-TCDD (Dioxin)	1746016	5.0 pg/L	5.0 pg/L

^a Analysis by EPA Method 100.2 - Only asbestos structures greater than 10 µm are counted in this method. Therefore, analytical laboratories testing wastewater from facilities that discharge to surface waters that are used for public drinking water supply must use a 0.1 µm pore size polycarbonate or MCE filter membrane to prevent loss of small asbestos fibers during filtration at the time of analysis or use **EPA Method 100.1**.

^b Four compounds (bromoform, chlorodibromomethane, chloroform, and dichlorodibromomethane) are found in combination and comprise a category of contaminants called "trihalomethanes". The concentration results of these compounds should be summed and the individual concentrations reported.

^c In some instances the Water Quality Criteria may be lower than the most sensitive methodology or practically achievable limits using any of the available approved methods. In such instances, the Department will accept qualified estimate values (J qualified values) where possible. The approval for the use of an estimated value for each contaminant should be requested with the Toxic Chemical Testing Study Plan. There may also be instances where the Water Quality Criteria may be lower than the method detection limit (MDL) for the contaminant using the most sensitive approved analytical method. In this case, explain in the Toxic Chemical Testing Study Plan and provide the name of the contaminant, test method, MDL and RL and the Department will determine how to proceed.

Revised December 2, 2019: RL Table Updated; **Revised September 10, 2019:** RLs Table Updated; **Revised August 14, 2018:** MDL revision 1.11 to revisions 2. 40 CFR link revised; **Revised September 18, 2017:** Added Nonylphenol and lowered LOQ for Cu and Acrolein; **Revised 5/18/2011:** Modified PP list to conform with 40 CFR. 423; **Revised April 12, 2011:** PCB by 1668 removed; **Revised November 11, 2010:** Clarify detection limit with the addition of LOQ, Added PCB by 1668 protocol, lowered LOQs for pollutants to WQ criteria; **Revised April 14, 2009:** Teflon and glass sampling req.'s added for semi-volatiles; **Revised March 17, 2009:** Removed HCA; **Revised July 2, 2007:** Asbestos added, and 2,3,7,8-TCDD footnote added.

**MARYLAND DEPARTMENT OF THE ENVIRONMENT
WATER & SCIENCE ADMINISTRATION**

Table: Required Toxic Chemical Analytes			
Number	Parameter	Cas Number	Minimum Reporting Limit (RL) µg/L, except as noted ^c
			Discharges to Fresh Water

^dTotal PCBs is defined as the sum of all congener or all isomer or homolog or Aroclor analyses.

Revised December 2, 2019: RL Table Updated; **Revised September 10, 2019:** RLs Table Updated; **Revised August 14, 2018:** MDL revision 1.11 to revisions 2. 40 CFR link revised; **Revised September 18, 2017:** Added Nonylphenol and lowered LOQ for Cu and Acrolein; **Revised 5/18/2011:** Modified PP list to conform with 40 CFR. 423; **Revised April 12, 2011:** PCB by 1668 removed; **Revised November 11, 2010:** Clarify detection limit with the addition of LOQ, Added PCB by 1668 protocol, lowered LOQs for pollutants to WQ criteria; **Revised April 14, 2009:** Teflon and glass sampling req.'s added for semi-volatiles; **Revised March 17, 2009:** Removed HCA; **Revised July 2, 2007:** Asbestos added, and 2,3,7,8-TCDD footnote added.