

Marshall Steam Station

Surface Water and Seep Monitoring

August and September 2014

Introduction

Flow and water quality measurements were collected at one seep sampling location (S-1) and two locations near the ash basin (Lake Norman-Upstream and Lake Norman-Downstream) associated with Duke Energy's Marshall Steam Station, located in Catawba County, North Carolina. The purpose of the sampling, which was conducted in September 2014, was to measure seepage flows and provide sufficient depth to allow collection of water quality samples for laboratory analysis. Seep location S-2 was not sampled due to an absence of flow during this monitoring event. Samples were collected via a combination of methods as described below. See Figures 1 and 2 for maps of the approximate sampling locations. Descriptions of the seep sampling locations are provided in Table 1.

Seep Flow Measurement Method and Results

A V-notch weir apparatus was utilized at location S-1 to impound the seepage in a channel, which concentrated discharge to a central location that allowed for a flow measurement to be obtained. The weir was constructed from ¼-inch-thick Plexiglas board material. The device was inspected after installation and prior to sampling to confirm sufficient flow and depth for sampling, and to verify that only minimal leakage, if any, was present. Sufficient time was allowed for the impounded seepage flows to reach equilibrium discharge flow before flow measurement and sampling.

The seepage flows at location S-1 were measured using the timed-volumetric method. A volume of water was collected from the discharge of the weir directly into an appropriately sized container. Volumes (in mL) were measured in the field utilizing a graduated container. The amount of time (in seconds) needed to collect the volume of water was recorded and flows (in MGD) were calculated for the timed-volume. The calculated flows (in MGD) at each seep location are presented in Appendix A.

Seep Water Quality Sample Collection Method and Results

Water quality samples were collected at S-1. To minimize potential effects of stormwater runoff, seep samples were collected during a period with minimal preceding rainfall. Samples were collected from the discharge flow at the flow measurement device described above or directly from the seep into sample bottles while minimizing disturbance and entrainment of soil/sediment.

Analytical parameters for analysis were: TSS, TDS, Oil & Grease, Cl, SO₄, F, COD, Al, As, B, Ba, Ca, Cd, Cu, Cr, Fe, Mn, Mo, Mg, Ni, Pb, Sb, Se, Tl, Zn, Hardness, and Hg. Storage and preservation techniques of the samples, after collection and prior to analyses, were followed according to Appendix B. Analyses were conducted by Duke Energy's Huntersville analytical laboratory (NC Wastewater Certification #248) and Pace Analytical Laboratories (NC Wastewater Certification # 12). Laboratory analytical methods used for each parameter are provided in Table 2 and analytical results are presented in Appendix A.

Seep In-situ Measurement Method and Results

In-situ field parameters (temperature, pH, and specific conductance) were measured at location S-1 utilizing calibrated field meters either at the discharge of the seep directly, at the discharge of the flow measurement devices, or in the water pool created behind the device, if sufficient water depth did not exist at the device discharge. The analytical results are presented in Appendix A.

Lake Norman Water Quality and In-situ Sample Collection Method and Results

Water quality samples and in-situ measurements from the Lake Norman were collected at a location upstream (Lake Norman-Upstream) and downstream (Lake Norman-Downstream) of Marshall Steam Station (Figure 2). Additionally, water samples and in-situ measurements were collected from an in-process ash basin location (Figure 1). The grab samples were collected from the river and basin's surface (0.3 m) directly into appropriate sample bottles. Preservation and analyses methods for the river and ash basin samples are provided in Table 2 and Appendix B.

All trace metal results were low, with most values either tabulated as less than the analytical reporting limit (RL) for the method or close to the RL (Table 1). For examples, all samples for arsenic, cadmium, Mercury, lead and selenium were report as less that the RL, whereas only four zinc values were greater than the RL of 1.0 µg/L. All zinc values were below the water quality standard (50 µg/L). Most of the copper values were above the RL, but onle marginally so, all values were also well below the water quality standard (7.0 µg/L). Total dissolved solids (TDS) values were low, and reflective of the low ionic strength and conductivity of waters in Lake Norman and the Catawba River Basin. All values for the nine water quality parameters monitored below NC water quality standards. Duke energy proposes that the in-stream monitoring frequency be reduced for semi-annually to annually.

Recommendations

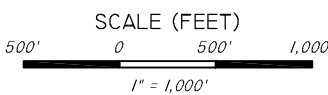
The low volume of flow at each seep location coupled with the relatively low constituent concentrations in the samples, suggests that there is little potential for Marshall Steam Station to influence water quality in the Lake Norman. If reasonable potential analyses demonstrate that there is no potential to exceed water quality standards, then Duke Energy proposes to re-evaluate the seep locations listed in this document annually over the next 5-year permit cycle. These annual evaluations would be documented and would verify the condition of the existing seeps and determine the presence of new seeps.

The North Carolina Department of Environment and Natural Resources – Division of Water Resources (DWR) will be promptly notified if any new seeps are identified or any significant changes are observed for the existing seeps. If any existing or newly identified seeps are determined to reach the Lake Norman, and demonstrate reasonable potential to exceed a water quality standard, Duke Energy will do one of the following: 1) stop the seep, 2) capture and route the seep so that it is discharged through a National Pollutant Discharge Elimination System (NPDES) permitted outfall or 3) address the seep using Best Management Plans approved by DWR.



- LEGEND:**
- DUKE ENERGY PROPERTY BOUNDARY
 - ASH BASIN WASTE BOUNDARY
 - LANDFILL/STRUCTURAL FILL BOUNDARY
 - ASH BASIN COMPLIANCE BOUNDARY
 - - - ASH BASIN COMPLIANCE BOUNDARY COINCIDENT WITH DUKE PROPERTY BOUNDARY
 - - - STREAM
 - - - TOPOGRAPHIC CONTOUR (4-FT INTERVAL)
 - ◆ ASH BASIN COMPLIANCE GROUNDWATER MONITORING WELL
 - SEEP SAMPLE LOCATION
 - ▲ NPDES OUTFALL LOCATION

- NOTES:**
1. PARCEL DATA FOR THE SITE WAS OBTAINED FROM DUKE ENERGY REAL ESTATE AND IS APPROXIMATE.
 2. WASTE BOUNDARY AND ASH STORAGE AREA BOUNDARY ARE APPROXIMATE.
 3. AS-BUILT MONITORING WELL LOCATIONS PROVIDED BY DUKE ENERGY.
 4. COMPLIANCE SHALLOW MONITORING WELLS (S) ARE SCREENED ACROSS THE SURFICIAL WATER TABLE.
 5. COMPLIANCE DEEP MONITORING WELLS (D) ARE SCREENED IN THE TRANSITION ZONE BETWEEN COMPETENT BEDROCK AND THE REGOLITH.
 6. TOPOGRAPHY DATA FOR THE SITE WAS OBTAINED FROM NC DOT GEOGRAPHIC INFORMATION SYSTEM (GIS) WEB SITE (DATED 2007).
 7. AERIAL PHOTOGRAPHY WAS OBTAINED FROM WSP DATED APRIL 2014.
 8. THE COMPLIANCE BOUNDARY IS ESTABLISHED ACCORDING TO THE DEFINITION FOUND IN 15A NCAC 02L .0107 (a).



IDENTIFIED SEEPS AND WATER QUALITY
 SAMPLE LOCATION MAP
 DUKE ENERGY CAROLINAS, LLC
 MARSHALL STEAM STATION ASH BASIN
 CATAWBA COUNTY, NORTH CAROLINA
 NPDES PERMIT #NC0004987

DATE
 SEPTEMBER 30, 2014

FIGURE
 1

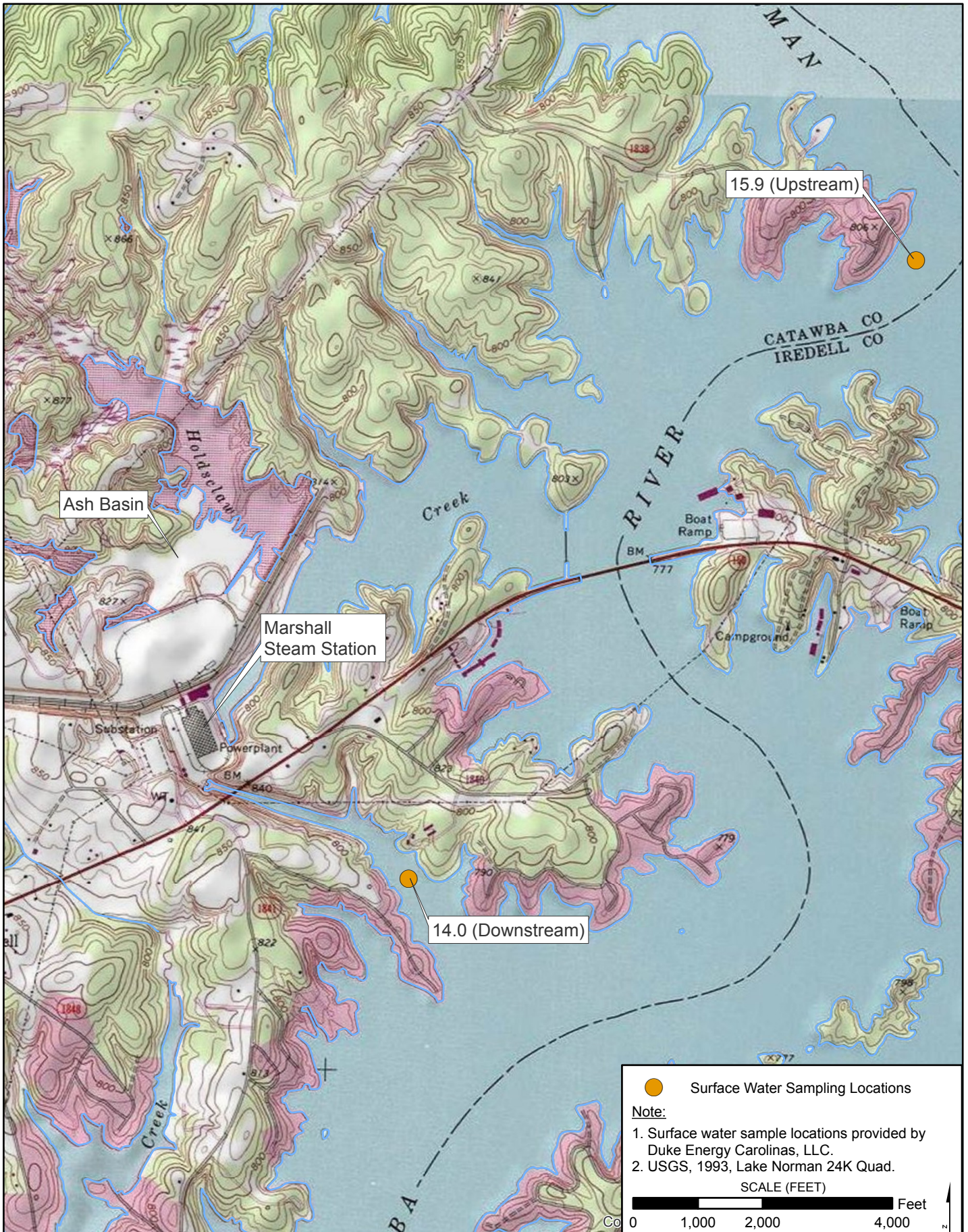


Table 1 – Marshall Steam Station Surface Water/Seep Locations and Descriptions

Seep ID	Location Coordinates ¹		Flow Description ²	Seep Description
	Latitude	Longitude		
S-1	35°36.712'	80°57.621'	Continuous	Located northeast of Active Ash Basin. Tributary toward Lake Norman. Well-defined stream approximately 2.5-ft wide.
S-2	35°42.560'	80°21.566'	No Flow	Located east of Active Ash Basin. Sheet flow toward Lake Norman. No visible flow was evident during the site visit.

Notes:

1. Location coordinates (degrees) for seep sampling locations are approximate, and are in NAD 83 datum.
2. Flow description for each seep sample location is based on observation during site visits performed by HDR Engineering, Inc. (HDR) on August 26, 2014. Flow measurements and analytical samples were collected on September 4, 2014.

Table 2 – Laboratory Analytical Methods

Parameter	Method	Reporting Limit	Units	Laboratory
Chemical Oxygen Demand (COD)	HACH 8000	20	mg/L	Duke Energy
Chloride (Cl)	EPA 300.0	1	mg/L	Duke Energy
Fluoride (F)	EPA 300.0	1	mg/L	Duke Energy
Sulfate (SO₄)	EPA 300.0	1	mg/L	Duke Energy
Oil and Grease	EPA 1664B	5	ug/L	Pace Analytical
Mercury (Hg)	EPA 245.1	0.05	ug/L	Duke Energy
Aluminum (Al)	EPA 200.7	0.005	mg/L	Duke Energy
Barium (Ba)	EPA 200.7	0.005	mg/L	Duke Energy
Boron (B)	EPA 200.7	0.05	mg/L	Duke Energy
Calcium (Ca)	EPA 200.7	0.01	mg/L	Duke Energy
Hardness	EPA 200.7	0.19	mg/L (CaCO ₃)	Duke Energy
Iron (Fe)	EPA 200.7	0.01	mg/L	Duke Energy
Magnesium (Mg)	EPA 200.7	0.005	mg/L	Duke Energy
Manganese (Mn)	EPA 200.7	0.005	mg/L	Duke Energy
Zinc (Zn)	EPA 200.7	0.005	mg/L	Duke Energy
Antimony (Sb)	EPA 200.8	1	ug/L	Duke Energy
Arsenic (As)	EPA 200.8	1	ug/L	Duke Energy
Cadmium (Cd)	EPA 200.8	1	ug/L	Duke Energy
Chromium (Cr)	EPA 200.8	1	ug/L	Duke Energy
Copper (Cu)	EPA 200.8	1	mg/L	Duke Energy
Lead (Pb)	EPA 200.8	1	ug/L	Duke Energy
Molybdenum (Mo)	EPA 200.8	1	ug/L	Duke Energy
Nickel (Ni)	EPA 200.8	1	ug/L	Duke Energy
Selenium (Se)	EPA 200.8	1	ug/L	Duke Energy
Thallium (Tl) Low Level	EPA 200.8	0.2	ug/L	Duke Energy
TDS	SM2540C	25	mg/L	Duke Energy
TSS	SM2540D	5	mg/L	Duke Energy

Appendix A
Seep Flows and Analytical Results

Marshall Steam Station
Surface Water/Seep Monitoring Analytical Results – August 2013 and September 2014

Parameter	Units	Seep Monitoring Location ^{1,2}		Lake Norman 15.9 Upstream ^{3,4}	Lake Norman 14.0 Downstream ^{3,4}
		S-1	S-2		
Oil & Grease	mg/l	<5	--	N/A	N/A
COD - Chemical Oxygen Demand	mg/l	<20	--	N/A	N/A
Cl - Chloride (00940)	mg/l	5.1	--	N/A	N/A
Fl - Fluoride	mg/l	<0.5	--	N/A	N/A
SO4 - Sulfate (00945)	mg/l	290	--	N/A	N/A
Hg - Mercury (71900)	µg/l	<0.05	--	<0.05	<0.05
Al - Aluminum (01105)	mg/l	0.278	--	N/A	N/A
Ba - Barium (01007)	mg/l	0.098	--	N/A	N/A
B - Boron (01022)	mg/l	<3.99	--	N/A	N/A
Ca - Calcium	mg/l	63.3	--	N/A	N/A
Hardness	mg/l (CaCO ₃)	291	--	N/A	N/A
Fe - Iron (01045)	mg/l	4.41	--	N/A	N/A
Mg - Magnesium	mg/l	32.3	--	N/A	N/A
Mn - Manganese (01055)	mg/l	0.936	--	N/A	N/A
Zn - Zinc (01092)	mg/l	<0.005	--	<1	2.23
Sb - Antimony (01097)	µg/l	<1	--	N/A	N/A
As - Arsenic (01002)	µg/l	<1	--	<1	<1
Cd - Cadmium (01027)	µg/l	<1	--	<1	<1
Cr - Chromium (01034)	µg/l	<1.47	--	<1	<1
Cu - Copper (01042)	µg/l	1	--	2.46	5.06
Pb - Lead (01051)	µg/l	<1	--	<1	<1
Mo - Molybdenum	µg/l	<1	--	N/A	N/A
Ni - Nickel (01067)	µg/l	<1.93	--	N/A	N/A
Se - Selenium (01147)	µg/l	<4.58	--	<1	<1
Tl - Thallium (01059)	µg/l	<0.2	--	N/A	N/A
TDS - Total Dissolved Solids (70300)	mg/l	530	--	40	60
TSS - Total Suspended Solids	mg/l	18	--	N/A	N/A
pH	s.u.	6.55	--	N/A	N/A
Temperature	°C	21.5	--	N/A	N/A
Specific conductance	µS/cm	2700	--	N/A	N/A
Flow	MGD	0.0019	--	N/A	N/A

Notes:

1. Flow measurements at S1 was collected on September 4, 2014.
2. Seep S2 was not sampled due to absence of flow during this monitoring event.

3. Lake Norman Samples were collect on August 5, 2013.
4. N/A indicates not applicable

Appendix B
Sample Preservation and Hold Times

Parameter Name	Container ¹	Preservation ^{2, 3}	Maximum Holding Time ⁴
Table IB—Inorganic Tests			
1. Acidity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days
2. Alkalinity	P, FP, G	Cool, ≤6 °C ¹⁸	14 days
4. Ammonia	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
10. Boron	P, FP, or Quartz	HNO ₃ to pH <2	6 months
11. Bromide	P, FP, G	None required	28 days
14. Biochemical oxygen demand, carbonaceous	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes
21. Color	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
23-24. Cyanide, total or available (or CATC) and free	P, FP, G	Cool, ≤6 °C ¹⁸ , NaOH to pH >10 ^{5, 6} , reducing agent if oxidizer present	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO ₃ or H ₂ SO ₄ to pH <2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days
Table IB—Metals⁷			
18. Chromium VI	P, FP, G	Cool, ≤6 °C ¹⁸ , pH = 9.3-9.7 ²⁰	28 days
35. Mercury (CVAA)	P, FP, G	HNO ₃ to pH <2	28 days
35. Mercury (CVAFS)	FP, G; and FP-lined cap ¹⁷	5 mL/L 12N HCl or 5 mL/L BrCl ¹⁷	90 days ¹⁷
3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except boron, chromium VI, and mercury	P, FP, G	HNO ₃ to pH <2, or at least 24 hours prior to analysis ¹⁹	6 months
38. Nitrate	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days
40. Nitrite	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
41. Oil and grease	G	Cool to ≤6 °C ¹⁸ , HCl or H ₂ SO ₄ to pH <2	28 days

Parameter Name	Container ¹	Preservation ^{2, 3}	Maximum Holding Time ⁴
42. Organic Carbon	P, FP, G	Cool to ≤6 °C ¹⁸ , HCl, H ₂ SO ₄ , or H ₃ PO ₄ to pH <2	28 days
44. Orthophosphate	P, FP, G	Cool, to ≤6 °C ^{18, 24}	Filter within 15 minutes; analyze within 48 hours
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours
48. Phenols	G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days
49. Phosphorous (elemental)	G	Cool, ≤6 °C ¹⁸	48 hours
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C ¹⁸ , H ₂ SO ₄ to pH <2	28 days
53. Residue, total	P, FP, G	Cool, ≤6 °C ¹⁸	7 days
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C ¹⁸	7 days
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C ¹⁸	7 days
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C ¹⁸	7 days
61. Silica	P or Quartz	Cool, ≤6 °C ¹⁸	28 days
64. Specific conductance	P, FP, G	Cool, ≤6 °C ¹⁸	28 days
65. Sulfate	P, FP, G	Cool, ≤6 °C ¹⁸	28 days
66. Sulfide	P, FP, G	Cool, ≤6 °C ¹⁸ , add zinc acetate plus sodium hydroxide to pH >9	7 days
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes
68. Surfactants	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours
69. Temperature	P, FP, G	None required	Analyze
73. Turbidity	P, FP, G	Cool, ≤6 °C ¹⁸	48 hours

Notes:

1. "P" is for polyethylene; "FP" is fluoropolymer (polytetrafluoroethylene (PTFE); Teflon®), or other fluoropolymer, unless stated otherwise in this table; and "G" is glass.
2. Except where noted in this table and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sample (e.g., using a 24-hour composite sample; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this table or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this table or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of results of analysis of individual grab samples. For examples of laboratory compositing

procedures, see EPA Method 1664 Rev. A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

3. When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirement of this table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater; Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid. Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under Sec. 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14-15. For static-renewal toxicity tests, each grab or composite sample may also be used to prepare test solutions for renewal at 24 h, 48 h, and/or 72 h after first use, if stored at 0-6 °C, with minimum head space.
5. ASTM D7365-09a specifies treatment options for samples containing oxidants (e.g., chlorine). Also, Section 9060A of Standard Methods for the Examination of Water and Wastewater (20th and 21st editions) addresses dechlorination procedures.
6. Sampling, preservation and mitigating interferences in water samples for analysis of cyanide are described in ASTM D7365-09a. There may be interferences that are not mitigated by the analytical test methods or D7365-09a. Any technique for removal or suppression of interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide through quality control measures described in the analytical test method. Any removal or suppression technique not described in D7365-09a or the analytical test method must be documented along with supporting data.
7. For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.
8. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
9. If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.
10. The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
11. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for

- thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).
12. If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.
 13. Extracts may be stored up to 30 days at <0 °C.
 14. For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
 15. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.
 16. Place sufficient ice with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature. Aqueous samples must not be frozen. Hand-delivered samples used on the day of collection do not need to be cooled to 0 to 6 °C prior to test initiation.
 17. Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly- capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
 18. Aqueous samples must be preserved at ≤ 6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of " ≤ 6 °C" is used in place of the " 4 °C" and " <4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤ 6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).
 19. An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.
 20. To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.
 21. Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.
 22. Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.
 23. For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB-EC) or 1681 (A-1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.
 24. The immediate filtration requirement in orthophosphate measurement is to assess the dissolved or bio-available form of orthophosphorus (i.e., that which passes through a 0.45-micron filter), hence the requirement to filter the sample immediately upon collection (i.e., within 15 minutes of collection). [38 FR 28758, Oct. 16, 1973].