

Method holding times used by the National Water Quality Laboratory

[Reference to the National Water Quality Laboratory as "NWQL" as a source for a holding time refers to holding times currently (2010) in practice at the laboratory that do not have a specific reference, but may be described in standard operating procedures or other internal documents. Citations for analytical methods and holding times listed may be found online at the NWQL services catalog page on the NWQL USGS-visible intranet web site at URL <http://www.nwql.cr.usgs.gov/USGS/catalog/index.cfm>.

<, less than; °C, degrees Celsius; µS/cm, microsiemens per centimeter at 25°C; ASF, automated segmented flow; C-18, polypropylene column containing porous silica coated with a chemically bonded octadecyl (C-18) phase; cICP-MS, collision/reaction cell inductively coupled plasma-mass spectrometry; ICP-AES, inductively coupled plasma-atomic emission spectrometry; ICP-MS, inductively coupled plasma-mass spectrometry; nm, nanometer; NPDES, National Pollution Discharge and Elimination System; OWQ, U.S. Geological Survey Water Resources Discipline Office of Water Quality; PAH, polycyclic aromatic hydrocarbon(s); PCB, polychlorinated biphenyl; USEPA, U.S. Environmental Protection Agency; UV, ultraviolet; VOC, volatile organic compound(s)].

Benthic Invertebrate populations

Matrix	Parameter	Lab code or schedule ^a	Parameter – method code pair	Description	Container type or label ^b	Holding time ^(c - h)	Reference
biota	benthic Invertebrate populations	2172	L2172	300-count subsample with standard taxonomic assessment	HDPE WM	14 ^d	Moulton & others (2000), NWQL
biota	benthic Invertebrate populations	2174	L2174	100-count subsample with standard taxonomic assessment	HDPE WM	14 ^d	Moulton & others (2000), NWQL
biota	benthic Invertebrate populations	2176	L2176	qualitative visual sort with standard taxonomic assessment	HDPE WM	14 ^d	Moulton & others (2000), NWQL
biota	benthic Invertebrate populations	2892	L2892	field-sorted, 100-count subsample with standard taxonomic assessment	HDPE WM	14 ^d	Moulton & others (2000), NWQL
biota	benthic Invertebrate populations	3113	L3113	200-count subsample with standard taxonomic assessment	HDPE WM	14 ^d	Moulton & others (2000), NWQL

Plant Pigments

biota	chlorophyll a and phaeophytin a in phytoplankton	3152	various ^e	fluorescence, USEPA Method 445.0	CHL	25 ^{cl}	Arar & Collins (1997)
biota	chlorophyll a and phaeophytin a in periphyton	3153, 3154	various ^e	fluorescence, USEPA Method 445.0	CHL	25 ^{cl}	Arar & Collins (1997)

Biomass

biota	biomass, periphyton	603	00573 GRV15	gravimetry, dry weight	CHL	30 ^{cl}	NWQL
biota	biomass, periphyton	603	49954 GRV15	gravimetry, ash-free dry weight	CHL	30 ^{cl}	NWQL
biota	biomass, periphyton	611	00572 GRV15	gravimetry, ash weight	CHL	30 ^{cl}	NWQL
biota	biomass, phytoplankton	2190	81354 GRV06	gravimetry, dry weight	CHL	30 ^{cl}	NWQL
biota	biomass, phytoplankton	2189	81353 GRV05	gravimetry, ash weight	CHL	30 ^{cl}	NWQL

Trace Elements

biota (tissue)	mercury	6050	49258 CV009	cold vapor atomic fluorescence, dry weight	TBI	180	NWQL
biota (tissue)	trace elements	various ^e	various ^e	ICP-AES, ICP-MS, and cICP-MS,, dry weight	TBI	180	NWQL

Matrix	Parameter	Lab code or schedule ^a	Parameter – method code pair	Description	Container type or label ^b	Holding time ^(c - h)	Reference
Organochlorine Pesticides							
biota (tissue)	organochlorine pesticides and total PCBs (low demand)	2101	various ^c	gas chromatography – electron capture detection	ALF	90	NWQL
Radiochemistry							
unfiltered water	radon-222	1369	82303 LSC01	liquid scintillation; ASTM D5072-98	RURCV	3	NWQL
General inorganic chemistry and physical properties (alphabetically within matrix)							
unfiltered water	acid neutralizing capacity	70	90410 TT040	electrometric titration, laboratory	RU	30	NWQL
unfiltered water	chemical oxygen demand	2144	00340 OX004	colorimetry	COD	30	NWQL
unfiltered water	color	20	00080 CC003	as platinum-cobalt units	RCB	30	NWQL
unfiltered water	pH	68	00403 EL006	electrometric electrode, laboratory	RU	30	NWQL
unfiltered water	specific conductance	69	90095 WHT03	Wheatstone bridge, laboratory	RU	30	NWQL
unfiltered water	total methylene blue active substances	96	38260 SPEC2	spectrophotometry	MBAS	10 ^{cl}	NWQL
unfiltered water	total nonpurgeable organic carbon	3211	00680 COMB9	high temperature combustion	TOC	14	Clesceri & others (1998)
unfiltered water	total petroleum hydrocarbons	2126	45501 00102	solvent extraction and gravimetry; USEPA 1664A and NWQL Technical Memorandum 96.08	OAG	28	NWQL
unfiltered water	total residue	165	00500 ROE12	gravimetry, residue on evaporation at 105°C	RU	180	NWQL
unfiltered water	turbidity	2187	63676 TS098	turbidimeter; Standard Methods, 20th ed., Method 2130	TBY	14	NWQL Technical Memorandum 2004.04
filtered water	alkalinity	2109	29801 TT040	electrometric titration, laboratory	FU	30	NWQL
filtered water	bromide (low level)	1258	71870 IC024	ion chromatography, low level (specific conductance < 100 µS/cm)	FU	180	NWQL
filtered water	bromide	3166	71870 IC027	ion chromatography	FU	180	NWQL
filtered water	chloride (low level)	1259	00940 IC024	ion chromatography, low level (specific conductance < 100 µS/cm)	FU	180	NWQL
filtered water	chloride	1571	00940 IC022	ion chromatography	FU	180	NWQL
filtered water	dissolved organic carbon	2612	00681 OX006	UV-promoted persulfate oxidation and infrared spectrometry, acidified; uses Supor filter	DOC	28	NWQL
filtered water	dissolved organic carbon	2613	00681 OX008	UV-promoted persulfate oxidation and infrared spectrometry, acidified; uses glass fiber filter	DOC	28	NWQL

Matrix	Parameter	Lab code or schedule ^a	Parameter – method code pair	Description	Container type or label ^b	Holding time ^(c - h)	Reference
filtered water	fluoride(low level)	1260	00950 IC024	ion chromatography, low level (specific conductance < 100 µS/cm)	FU	180	NWQL
filtered water	fluoride	31	00950 ISE05	ion-selective electrode, ASF	FU	180	NWQL
filtered water	iodide	1202	71865 CL033	colorimetry, ceric-arsenious,ASF	FU	180	NWQL
filtered water	silica	3121	00955 CL151	colorimetry, molybdate blue, ASF	FU	180	NWQL
filtered water	sulfate (low level)	1263	00945 IC024	ion chromatography, low level (specific conductance < 100 µS/cm)	FU	180	NWQL
filtered water	sulfate	1572	00945 IC022	ion chromatography	FU	180	NWQL
filtered water	total dissolved solids	27	70300 ROE10	gravimetry, residue on evaporation at 180°C	FU	180	NWQL
filtered water	total dissolved solids	159	00515 ROE09	gravimetry, residue on evaporation at 105°C	FU	180	NWQL
filtered water	UV-absorbing organic constituents, 254 nm	2614	50624 UV005	spectrophotometry; uses glass fiber filter; Standard Methods 19th ed., Method 5910	UAS	7 ^{c2}	NWQL
filtered water	UV-absorbing organic constituents, 280 nm	2615	61726 UV007	spectrophotometry; uses glass fiber filter; Standard Methods 19th ed., Method 5910	UAS	7 ^{c2}	NWQL
filtered water	UV-absorbing organic constituents, 254 nm	2616	50624 UV006	spectrophotometry; uses Supor filter; Standard Methods 19th ed., Method 5910	UAS	7 ^{c2}	NWQL
filtered water	UV-absorbing organic constituents, 280 nm	2617	61726 UV002	spectrophotometry; uses Supor filter; Standard Methods 19th ed., Method 5910	UAS	7 ^{c2}	NWQL
solids	inorganic carbon	various ^e	various ^e	coulimetric	CC	100	NWQL
solids	particulate, organic carbon	2611	00689 CAL06	elemental C by combustion and thermal conductance, USEPA 440.0; OWQ Technical Memorandum 2000.08	TPCN	100	Zimmermann & others (1997)
solids	suspended residue	169	00530 SLD04	gravimetry, residue on evaporation at 105°C	SUSO	180	NWQL
solids	total carbon	133	00693 IF002	combustion and thermal conductance	CC	100	NWQL
solids	total particulate carbon	2606	00694 COMB6	combustion and thermal conductance; USEPA 440.0 and OWQ Technical Memorandum 2000.08	TPCN	100	Zimmermann & others (1997)
solids	total particulate inorganic carbon	2608	00688 00127	combustion and thermal conductance; USEPA 440.0 and OWQ Technical Memorandum 2000.08	TPCN	100	Zimmermann & others (1997)
solids	total particulate nitrogen	2607	49570 COMB7	elemental N by combustion and thermal conductivity, USEPA 440.0; OWQ Technical Memorandum 2000.08	TPCN	100	Zimmermann & others (1997)

Matrix	Parameter	Lab code or schedule ^a	Parameter – method code pair	Description	Container type or label ^b	Holding time ^(c - h)	Reference
solids	volatile suspended residue	49	00535 SLD05	gravimetry, volatile on ignition at 550°C, suspended solids	SUSO	180	NWQL
Nutrients I — Nitrogen (alphabetically by parameter)							
filtered water	ammonia	1991	00608 CL036	acidified, salicylate-hypochlorite, colorimetry, ASF	FCA	30	Patton & Gilroy (1999)
unfiltered water	ammonia	2188	00610 CL017	acidified, colorimetry, ASF	WCA	30	Patton & Gilroy (1999)
filtered water	ammonia	3116	00608 00048	salicylate-hypochlorite, colorimetry, discrete analyzer	FCC	30	Patton & Gilroy (1999)
filtered water	ammonia + organic nitrogen	1985	00623 KJ002	microkjeldahl digestion, colorimetry, ASF	FCC	30	Patton & Gilroy (1999)
unfiltered water	ammonia + organic nitrogen	1986	00625 KJ008	acidified, microkjeldahl digestion, colorimetry, ASF	WCA	30	Patton & Gilroy (1999)
filtered water	ammonia + organic nitrogen	1994	00623 KJ003	acidified, microkjeldahl digestion, colorimetry, ASF	FCA	30	Patton & Gilroy (1999)
filtered water	nitrite	3117	00613 00049	diazotization, colorimetry, discrete analyzer	FCC	30	Patton & Gilroy (1999)
filtered water	nitrate + nitrite (low level)	1979	00631 CL050	cadmium reduction-diazotization, colorimetry, ASF	FCC	30	Patton & Gilroy (1999)
filtered water	nitrate + nitrite	1975	00631 CL048	cadmium reduction-diazotization, colorimetry, ASF	FCC	30	Patton & Gilroy (1999)
filtered water	nitrate + nitrite	1990	00631 CL047	acidified, cadmium reduction-diazotization, colorimetry, ASF	FCA	30	Patton & Gilroy (1999)
filtered water	total dissolved nitrogen	2754	62854 CL063	alkaline persulfate digestion, colorimetry, ASF	FCC	30	Patton and Kryskalla, 2003
filtered water	total dissolved nitrogen	2755	62854 CL062	acidified alkaline persulfate digestion, colorimetry, ASF	FCA	30	Patton and Kryskalla, 2003
unfiltered water	total nitrogen	2756	62855 AKP01	acidified alkaline persulfate digestion, colorimetry, ASF	WCA	30	Patton and Kryskalla, 2003
Nutrients II — Phosphorus (alphabetically by parameter)							
filtered water	orthophosphate	3118	00671 00048	phosphomolybdate, colorimetry, discrete analyzer	FCC	30	Patton & Gilroy (1999)
filtered water	total dissolved phosphorus (low level)	2331	00666 CL020	acid persulfate digestion; colorimetry, ASF; USEPA 365.1	FCC	30	U.S. Environmental Protection Agency (2005a), table II
filtered water	total dissolved phosphorus (low level)	2332	00666 CL019	acidified, acid persulfate digestion, colorimetry, ASF; USEPA 365.1	FCA	30	U.S. Environmental Protection Agency (2005a), table II
filtered water	total dissolved phosphorus	1983	00666 KJ005	microkjeldahl digestion, colorimetry, ASF	FCC	30	Patton & Gilroy (1999)

Matrix	Parameter	Lab code or schedule ^a	Parameter – method code pair	Description	Container type or label ^b	Holding time ^(c - h)	Reference
filtered water	total dissolved phosphorus	1992	00666 KJ004	acidified microkjeldahl digestion, colorimetry, ASF	FCA	30	Patton & Gilroy (1999)
filtered water	total dissolved phosphorus	2757	00666 CL063	alkaline persulfate digestion	FCC	30	Patton and Kryskalla, 2003
filtered water	total dissolved phosphorus	2758	00666 CL062	acidified, alkaline persulfate digestion, colorimetry, ASF	FCA	30	Patton and Kryskalla, 2003
unfiltered water	total phosphorus (low level)	2333	00665 CL021	acidified, acid persulfate digestion, colorimetry, ASF; USEPA 365.1	WCA	30	U.S. Environmental Protection Agency (2005a), table II
unfiltered water	total phosphorus	1984	00665 KJ009	acidified, microkjeldahl digestion, colorimetry, ASF	WCA	30	Patton & Gilroy (1999)
unfiltered water	total phosphorus	2759	00665 AKP01	acidified, alkaline persulfate digestion, colorimetry, ASF; (Patton and Kryskalla, 2003)	WCA	30	Patton & Gilroy (1999)

Trace elements, mercury, and arsenic species

filtered water	(arsenite, arsenate, monomethyl arsonate, or dimethylarsinate)	various ^c	various ^c	high performance–liquid chromatography ICP–MS or cICP–MS	SAS	90	U.S. Geological Survey (variouslydated); Garbarino & others (2002); Garbarino & others (2006)
filtered water	dissolved mercury	2707	71890 CV014	cold vapor–atomic fluorescence spectrometry	FAM	120 ^h	Garbarino and Damrau (2001)
unfiltered water	total mercury	2708	71900 CV018	cold vapor–atomic fluorescence spectrometry	RAM	120 ^h	Garbarino and Damrau (2001)
solids	total mercury	8512	71921 00026	cold vapor–atomic fluorescence spectrometry	CU	180	NWQL
unfiltered water	trace elements	various ^c	various ^c	acidified, ICP-AES, ICP-MS, or cICP-MS	RA	180	U.S. Environmental Protection Agency (2005b)
filtered water	trace elements	various ^c	various ^c	ICP-AES, ICP-MS, or cICP-MS, acidified	FA	180	U.S. Environmental Protection Agency (2005b)
solids	trace elements	various ^c	various ^c	ICP-AES, ICP-MS, or cICP-MS, digestate	CU	180	U.S. Environmental Protection Agency (2005b)

Organic methods I

Volatile organic compounds (VOCs), gas chromatography

unfiltered water	1,2-Dibromoethane and 1,2-Dibromo-3-chloropropane	1306	various ^c	microextraction electron capture detection	GCV	14 ^{c1}	Munch (1995)
unfiltered water	selected VOCs	1307	various ^c	acidified, purge-and-trap, full-scan mass spectrometry	GCV	14 ^{c2}	Connor & others (1998)
unfiltered water	benzene, toluene, ethylbenzene, xylenes, and methyl tertiary-butyl ether	1378	various ^c	purge-and-trap, full-scan mass spectrometry	GCV	14 ^{c2}	Connor & others (1998)

Matrix	Parameter	Lab code or schedule ^a	Parameter – method code pair	Description	Container type or label ^b	Holding time ^(c - h)	Reference
unfiltered water	61 VOCs	1380	various ^e	acidified, purge-and-trap, full-scan mass spectrometry, reported at < 0.2 reporting level	GCV	14 ^{c2}	Connor & others (1998)
unfiltered water	85 VOCs with nontargets (tentatively identified compounds)	2020	various ^e	acidified, purge-and-trap, full-scan mass spectrometry	GCV	14 ^{c2}	Connor & others (1998)
unfiltered water	85 VOCs	2021	various ^e	acidified, purge-and-trap, full-scan mass spectrometry	GCV	14 ^{c2}	Connor & others (1998)
unfiltered water	gasoline oxygenates, degradates, and benzene, toluene, ethylbenzene, xylenes	4024	various ^e	purge-and-trap, full-scan mass spectrometry	GCV	14 ^{c2}	Rose & Sandstrom (2003)
unfiltered water	gasoline oxygenates, degradates, and benzene, toluene, ethylbenzene, xylenes	4025	various ^e	acidified, purge-and-trap, full-scan mass spectrometry	GCV	14 ^{c2}	Rose & Sandstrom (2003)
unfiltered water	85 VOCs at minimum reporting levels with non-targets (tentatively identified compounds)	4054	various ^e	acidified, purge-and-trap, full-scan mass spectrometry	GCV	14 ^{c2}	Connor & others (1998)

Organic methods II
Continuous liquid-liquid extraction, gas chromatography

unfiltered water	semivolatile organic compounds— chlorinated hydrocarbons, haloethers, nitroaromatics and isophorone, nitrosoamines, phthalate esters, polynuclear aromatics, and priority pollutant phenols	1383	various ^e	full-scan mass spectrometry	GCC	14 ^f	NWQL
unfiltered water	organophosphorus pesticides	1403	various ^e	flame photometric detection	GCC	7 ^g	NWQL
unfiltered water	semivolatile organic compounds	1494	various ^e	full-scan mass spectrometry	GCC	14 ^f	NWQL
unfiltered water	anthropogenic waste indicator compounds	4433	various ^e	full-scan mass spectrometry	GCC	14 ^f	Zaugg & others (2006b)

Organic methods III
Solid-phase extraction, gas chromatography

filtered water	organonitrogen herbicides (Triazine)	1379	various ^e	selected-ion monitoring mass spectrometry	GCC	7 ^f	NWQL
filtered water	organophosphorus pesticides	1402	various ^e	flame photometric detection	GCC	7 ^g	NWQL
filtered water	anthropogenic waste indicator compounds	1433	various ^e	full-scan mass spectrometry	GCC	14 ^f	NWQL
filtered water	high-use pesticides and metabolites	2001	various ^e	selected-ion monitoring mass spectrometry	GCC	7 ^g	NWQL
filtered water	high-use pesticides and metabolites	2010	various ^e	selected-ion monitoring mass spectrometry	C-18	7 ^{c1}	NWQL
filtered water	selected pesticides and degradation compounds	2003	various ^e	selected-ion monitoring mass spectrometry	GCC	7 ^g	NWQL
	selected pesticides and degradation compounds	2033	various ^e	selected-ion monitoring mass spectrometry	GCC	7 ^g	

Matrix	Parameter	Lab code or schedule ^a	Parameter – method code pair	Description	Container type or label ^b	Holding time ^(c - h)	Reference
Organic methods IV							
Solid-phase extraction, high performance liquid chromatography (HPLC)							
filtered water	polar pesticides and metabolites	2060	various ^e	selected-ion monitoring mass spectrometry	GCC	7 ^{c1}	NWQL
filtered water	human-health pharmaceuticals	2080	various ^e	selected-ion monitoring mass spectrometry	GCC	7 ^{c1}	NWQL
Organic methods V							
Liquid-liquid (separatory funnel) extraction, gas chromatography							
unfiltered water	chlorophenoxy-acid herbicides, Dicamba, and Picloram	79	various ^e	electron capture detection	GCC	7 ^f	NWQL
unfiltered water	chlorophenoxy-acid herbicides	1304	various ^e	electron capture detection	GCC	7 ^f	NWQL
unfiltered water	organophosphate insecticides	1319	various ^e	flame photometric detection	GCC	7 ^f	NWQL
unfiltered water	organochlorine insecticides with gross PCBs	1324	various ^e	electron capture detection	GCC	7 ^f	NWQL
unfiltered water	PCBs and aroclors (low demand)	1364	various ^e	electron capture detection	GCC	7 ^f	NWQL
unfiltered water	organonitrogen herbicides (Triazine)	1389	various ^e	nitrogen-phosphorus detection	GCC	7 ^f	NWQL
unfiltered water	organochlorine insecticides with gross PCBs (low level)	1398	various ^e	electron capture detection	GCC	7 ^f	NWQL
unfiltered water	organochlorine pesticides and specific PCB aroclors	1608	various ^e	electron capture detection (NPDES-USEPA Method 608)	GCC	7 ^f	U.S. Environmental Protection Agency (2005a), pt. 136, app.A
Organic methods VI							
Solids, gas chromatography							
solids	organophosphorus pesticides	1404	various ^e	Soxhlet extraction, flame photometric detection	BGC	180 ^{c1}	NWQL
solids	organochlorine pesticides and total PCBs	2501	various ^e	Soxhlet extraction, electron capture detection	BGC	365 ^{c1} before extraction if kept at -20°C	Furlong & others (1996)
solids	semivolatile organic compounds	2502	various ^e	Soxhlet extraction, full-scan mass spectrometry	BGC	365 ^{c1} before extraction if kept at -20°C	Furlong & others (1996)
solids	selected PAH and total PCBs	2504	various ^e	Soxhlet extraction, full-scan mass spectrometry (PAH); electron capture detection (PCB)	BGC	365 ^{c1} before extraction if kept at -20°C	Noriega & others (2004)
solids	selected PAH	2505	various ^e	Soxhlet extraction, full-scan mass spectrometry	BGC	365 ^{c1} before extraction if kept at -20°C	Noriega & others (2004)
solids	selected PAH with alkylated homologs	2506	various ^e	Soxhlet extraction, full-scan mass spectrometry	BGC	365 ^{c1} before extraction if kept at -20°C	Noriega & others (2004)

Matrix	Parameter	Lab code or schedule ^a	Parameter – method code pair	Description	Container type or label ^b	Holding time ^(c - h)	Reference
solids	anthropogenic waste indicator compounds	5433	various ^c	pressurized solvent extraction, full-scan mass spectrometry	BGC	365 ^{c1} before extraction if kept at -20°C	Burkhardt & others (2006)
solids	organochlorine pesticides and PCB aroclors	5504	various ^c	Soxhlet extraction, electron capture detection	BGC	365 ^{c1} before extraction if kept at -20°C	Noriega & others (2004)
solids	PAHs and semivolatiles	5506	various ^c	pressurized solvent extraction, full-scan mass spectrometry	BGC	365 ^{c1} before extraction if kept at -20°C	Zaugg & others (2006a)
solids	PAHs, semivolatiles, and PAH homologs	5507	various ^c	pressurized solvent extraction, full-scan mass spectrometry	BGC	365 ^{c1} before extraction if kept at -20°C	Zaugg & others (2006a)

a
Lab codes are included for benthic invertebrate populations, plant pigments, biomass, trace elements, mercury, arsenic species, radiochemistry, and nutrients regardless of matrix. Schedules are included regardless of matrix for organic analyses and methods.

b
The abbreviations used for sample container types and labels are described in table below and includes the name, description, other pertinent information about sampling, and temperature and shipping requirements for samples.

c
The holding time is the maximum time in calendar days that a sample may be held prior to analysis and its analytical results still be considered valid.

^{c1} - The holding time is the maximum calendar days from sample collection to sample extraction or isolation.

^{c2} - The holding time is the maximum calendar days from sample collection to injection on the analytical instrument.

d
The holding time for benthic invertebrate samples reflects the maximum time in calendar days from arrival at the NWQL to stabilization with ethanol.

e
Information on individual analytes may be found in the NWQL services catalog, accessible on the USGS-visible intranet page at URL <http://www.nwql.cr.usgs.gov/USGS/catalog/index.cfm>.

f
The holding time is based upon the arrival of the sample at the NWQL. Samples may be stabilized with a preservative upon arrival.

g
Samples received for some methods are extracted within 2 working days of receipt at the laboratory, or 7 days from sample collection. NWQL SOPs ORGP0053.x, Automated preparation by method 2001: NWQL schedules 2001, 2003, 2010, and 2033, and ORGF0462.x, Pesticide Analysis by SIM-GC/MS, and may be useful references.

h
Samples must be preserved with BrCl within 5 days from arrival at the NWQL in order to extend the holding time to 120 days.