Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Autosampler	Agilent	G2614A	CN55237964	2007	NEW
Block Digestor	Bioscience	163-466T		1997	NEW
Block Digestor	Bioscience	2091B1		1997	NEW
BOD auto-analyzer	ManTech	BODAssayPlus			Footnote 1
BOD Incubator	Fisher		00037-090-00		Footnote 1
BOD Incubator	??				Footnote 1
BOD probe	Jenco				Footnote 1
Centrifuge	IEC	--	3634P-14		Footnote 1
Centrifuge	Fisher Scientific	AccuSpin 300	603101639	2003	NEW
Centrifuge	Precision	Durafuge 100	40317924	2003	NEW
Centrifuge	International Centrifuge Co.	HN	98323M-1		Footnote 1
COD Reactor	Bioscience Inc.	2091B1	34613302		Footnote 1
COD Reactor	Bioscience Inc.	163-466T	COD-T349		Footnote 1
Concentrator	O.I. Analytical	4560		1999	NEW
Conductivity Probe	Yellow Springs	32	COD0031		Footnote 1
Conductivity/Dissolved Oxygen Probe	Corning	M90	001253		Footnote 1
Cyanide Distillation Unit	Andrews Glass	MIDI System	MCVA13908221		Footnote 1
Cyanide Distillation Unit	Andrews Glass	MIDI System	33212579		Footnote 1
Digestion Unit	Gerhardt	Kjeldatherm KB	4062216	2007	NEW
Distillation Unit	Gerhardt	Vapodist30	VAP005617	2007	NEW
Drying Oven	Fisher		40200001		Footnote 1
Drying Oven	Fisher	630G	800121		Footnote 1
Drying Oven	Lab Line				Footnote 1
Drying Oven	Scientific Products	DX-61	194002		Footnote 1
Drying Oven	Fisher	630G	801N0001		Footnote 1
Fixed Wavelength Infrared Spectrophotometer	Foxboro	Miran1FF	2592	1997	NEW
Fixed Wavelength Infrared Spectrophotometer	Foxboro	Miran1FF	2733		Footnote 1

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Flashpoint Tester	Koehler	K-162		1992	NEW
Fluoride Probe	Orion	96-09	9609BN		Footnote 1
Gas Chromatograph	Agilent	6890N	US10423014		Footnote 1
Gas Chromatograph	Agilent	6890N	CN10551059	2007	NEW
Gas Chromatograph (Dual ECD)	Hewlett Packard	5890 Series II	3223A43015		Footnote 1
Gas Chromatograph (Dual ECD)	Hewlett Packard	5890 Series II	336A51142		Footnote 1
Gas Chromatograph (Dual ECD)	Hewlett Packard	5890Series II	2750A15311		Footnote 1
Gas Chromatograph (Dual ECD)	Agilent	6890	US10215019		Footnote 1
Gas Chromatograph (Dual ECD)	Agilent	6890N	US10250081		Footnote 1
Gas Chromatograph (Dual ECD)	Hewlett Packard	5890 Series II	3235A45184		Footnote 1
Gas Chromatograph (Dual ECD)	Agilent	6890N	CN10551052		Footnote 1
Gas Chromatograph (Dual FID)	Hewlett Packard	5890 Series II	3126A36534		Footnote 1
Gas Chromatograph (Dual FID)	Hewlett Packard	5890 Series II	3133A37568		Footnote 1
Gas Chromatograph (Dual FID)	Hewlett Packard	5890II	3235A44731		Footnote 1
Gas Chromatograph (Dual FID)	Hewlett Packard	5890 Series II	2950A26022		Footnote 1
Gas Chromatograph (ECD)	Hewlett Packard	5890 Series II	3203A40480		Footnote 1
Gas Chromatograph (FID)	Hewlett Packard	5890 Series II	3126A36955	1997	NEW
Gas Chromatograph (FID)	Hewlett Packard	5890 Series II			Footnote 1

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Gas Chromatograph (FID/PID)	Hewlett Packard	5890 Series II	3203A40477	1993	NEW
Gas Chromatograph (FID/PID)	Hewlett Packard	5890 Series II	3203A41169	1993	NEW
Gas Chromatograph (FID/PID)	Hewlett Packard	5890A	2750A15898	1997	NEW
Gas Chromatograph (FID/PID)	Hewlett Packard	5890 Series II	3223A42733	1993	NEW
Gas Chromatograph (FID/PID)	Hewlett Packard	5890 Series II	3223A60064	1993	NEW
Gas Chromatograph (FID/PID)	Hewlett Packard	5890 Series II	3336A60064	1993	NEW
Gas Chromatograph (FID/PID)	Hewlett Packard	5890 Series II	3033A33301	1998	NEW
Gas Chromatograph (FID/PID)	Hewlett Packard	5890 Series II	3336A60066	1997	NEW
Gas Chromatograph (FID/PID)	Hewlett Packard	5890 Series II			Footnote 1
Gas Chromatograph (FID/PID/ELCD)	Hewlett Packard	5890 Series II	3203A40699	1993	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	6890/5973A	US00007750	2001	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	6890/5973A	US00022931	2000	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6850/5973N	US00001207	2001	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6850/5973	US00001206	2001	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6850/5973N	US01874908	2002	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6850/5973N	US10440793	2002	NEW

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Gas Chromatograph/Mass Spectrometer	Agilent	6850/5973N	US00002860	2003	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6890N/5973	US00034262	2004	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6890N/5973	CN10318006	2004	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6890N/5973	CN10318007	2004	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	6890N/5973		2006	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	6890N/5973		2005	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	5890II/5972		1997	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	6890N/5973		2000	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	6890/5973A	US00020097	1999	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	5890Ser.II/5971	3140A39653	1993	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	5890Ser.II/5972	3235A46723	1995	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	5890Ser.II/5971	3133A37717	1993	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6890/5973	US10130035	2003	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6890N/5973	US10341048	2005	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	5890Ser.II/5971	3033A30488	1993	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	5890Ser.II	3033A32428	1987	NEW

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Gas Chromatograph/Mass Spectrometer	Hewlett Packard				Footnote 1
Gas Chromatograph/Mass Spectrometer	Agilent	6890N/5973	US10206070/A12019	2006	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6890N/5973N	US10222064/A13016	2006	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	5975B/6890N	US62724086/CN10636107	2006	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	6890N/5973		2001	NEW
Gas Chromatograph/Mass Spectrometer	Hewlett Packard	5890IIB/5971A	2921A24077/3188A02848	1992	NEW
Gas Chromatograph/Mass Spectrometer	Agilent	6890/5973	CN10427051/US41720775	2007	NEW
Hot Block	Environmental Express				Footnote 1
Hot Block	Environmental Express				Footnote 1
Hot Block	Environmental Express				Footnote 1
Hot Block	Environmental Express				Footnote 1
Hot Block	Environmental Express				Footnote 1
Hot Block	Environmental Express				Footnote 1
Hot Plate	??				Footnote 1
Hot Plate	??				Footnote 1
Inductively Coupled Plasma Spectrophotometer/MS	Perkin Elmer	ELAN6100E	1650004	2001	NEW
Inductively Coupled Plasma Spectrophotometer/MS	Perkin Elmer	ELAN6100E	G1970008	2004	NEW
Inductively Coupled PlasmaSpectrophotometer	Perkin Elmer	Optima 3000	069N4092201	1997	NEW

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Inductively Coupled PlasmaSpectrophotometer	Perkin Elmer	Optima 4300	077N1100901	2002	NEW
Inductively Coupled PlasmaSpectrophotometer	Perkin Elmer	Optima 5300DV	077N5112802	2006	NEW
Injector	Agilent	7683 series	CN55130059	2007	NEW
Injector Tower	Hewlett Packard	7673			Footnote 1
Ion Chromatograph	Dionex	DX 500	98060923	1996	NEW
Ion Chromatograph	Dionex	DX 100	40452	1997	NEW
Ion Chromatograph	Dionex	DX 600	139082221	2002	NEW
Ion Chromatograph	Dionex	ICS-1000	03110585	2002	NEW
Ion Chromatograph	Dionex	CD25A	01060463	2005	NEW
Ion Chromatograph	Dionex	AD25	01050864	2007	NEW
Ion Chromatograph	Dionex	CD25-1	00070432	2002	NEW
Ion Chromatograph	Dionex	LC20	94010215	2007	NEW
Ion Chromatograph (with UV/VIS)	Dionex	DX 500	94120366	2000	NEW
Ion Chromatograph/Mass spectrometer	Metrohm/Agilent /	LC30-1/LC110/IC800		2005	NEW
Kiln	Cress	E2418	0503DD	2005	NEW
Mercury Analyzer	Perkin Elmer	FIMS 400	4109	1995	NEW
Mercury Analyzer	Perkin Elmer	FIMS 400	4167	1995	NEW
Orbital shaker	Lab-Line	--			Footnote 1
pH Meter	Beckman	Phi - 40			Footnote 1
pH Meter	Beckman	Phi - 40			Footnote 1
pH Meter	Beckman	Phi - 32			Footnote 1
pH Meter	Mettler Toledo	SevenEasy	1227116127		
pH Probe	Orion	91-56	9156000		Footnote 1
pH Probe	Orion	91-56			Footnote 1

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Purge & Trap Concentrator	O.I. Analytical	4460A		1992	NEW
Purge & Trap Concentrator	O.I. Analytical	4460A		1993	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		1993	NEW
Purge & Trap Concentrator	O.I. Analytical	4460A		1997	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		1993	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		1992	NEW
Purge & Trap Concentrator	O.I. Analytical	4460A		1993	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		1998	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2001	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2000	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2001	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2001	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2002	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2002	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2003	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2004	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2004	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2004	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2006	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2005	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2000	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		1997	NEW
Purge & Trap Concentrator	O.I. Analytical	4460A			Footnote 1
Purge & Trap Concentrator	O.I. Analytical	4560	H351460339	2006	NEW
Purge & Trap Concentrator	O.I. Analytical				Footnote 1

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Purge & Trap Concentrator	O.I. Analytical	4560	E324406	2006	NEW
Purge & Trap Concentrator	O.I. Analytical	4560		2001	NEW
Purge and Trap Water/Soil AutoSampler	O.I. Analytical	4552		1993	NEW
Purge and Trap Water/Soil AutoSampler	EST	8100		2006	NEW
Rapid Vap	Labconco		266435		Footnote 1
Rapid Vap	Labconco		705319		Footnote 1
Rapid Vap	Labconco		21098412F		Footnote 1
Rapid Vap	Labconco		010194458E		Footnote 1
Rapid Vap	Labconco	7910000	040824527		Footnote 1
Rotator	N/A				Footnote 1
Rotator	N/A				Footnote 1
Rotator	N/A				Footnote 1
Rotator	N/A				Footnote 1
SPE-Controller	Horizon Technology	SPE-DEX	020357		Footnote 1
SPE-Extractor	Horizon Technology	SPE-DEX 4790	030359		Footnote 1
SPE-Extractor	Horizon Technology	SPE-DEX 4790	030360		Footnote 1
TOC Analyzer	Shimadzu	TOC-5000A	33N01036A	1998	NEW
TOC Analyzer w/AS	Tekmar-Dohrmann	Phoenix 8000	US02106006	2002	NEW
TOC Autosampler	Shimadzu	ASI-500A-H-P	33212579	1998	NEW
TOC Soil Sampler Module	Shimadzu	SSM-5000A	34613302	1998	NEW
Turbidity Meter	HF Instruments	DRT-100B	24942		Footnote 1
Turbidity Meter	Orbeco-Hellige	965-10A	4389	2007	NEW
Turbidity Meter	Orbeco-Hellige	965	5078	2007	NEW
Turbo Vap	Zymark		04053		Footnote 1
Turbo Vap	Zymark	--			Footnote 1
Turbo Vap II	Zymark		04516		Footnote 1

Instrument/ Equipment	Manufacturer	Model Number	Serial Number	Year Put into Service	Condition When Received
Turbo Vap II	Zymark		04272		Footnote 1
Turbo Vap II	Zymark		TV0239N11193		Footnote 1
Turbo Vap LV	Caliper LifeSciences	103200/2	TV0429N12434		Footnote 1
Turbo Vap LV	Caliper LifeSciences	103200/2	TV0429N12435		Footnote 1
UV/VS Spectrometer	Thermospectronic	Genesys20		2002	NEW

[1]Although equipment is operational and calibration maintained, this information is not available.

Table 21-2. Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Graphite Furnace (GFAA)	Inspect graphite tube Inspect contact rings Clean windows Align lamp	Daily Daily Daily Daily
Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Fill reductant bottle with 10% Stannous Chloride	Daily Daily Daily
ICP	Check/replace pump tubing Check liquid argon supply Check fluid level in waste container Check/clean/replace filters Check torch Clean torch and nebulizer	Daily/as needed Daily Daily Daily/as needed Daily As needed
ICP MS	Check/replace pump tubing Inspect torch and injector cones Clean/replace ion lens Replace torch o-rings Check/replace gas filters Change rough pump oil Check chiller water level	Daily/as needed Daily As needed As needed As needed As needed Weekly
UV-Vis Spectrophotometer	Clean sample holder Precision check/alignment of flow cell Wavelength verification check	As required As required Semi-annually
Gas Chromatograph/Mass Spectometer (GCMS)	Bake trap (VOC only) Clean source Check/change vacuum pump oil Clean injectors; replace liners (SVOC only) Replace column Clean cooling fan grills	Daily As needed Annually, as needed Daily As needed Semiannually

Instrument	Procedure	Frequency
Gas Chromatograph (GC)	Change septum Check gases Replace or clip column Clean injectors; replace liners Clean cooling fan grills	As needed Daily As needed As needed Semiannually
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually Sent out, as needed
Flame Ionization Detector (FID)	Detector cleaning	As required
Flame Photoionization Detector (FPD)	Clean and/or Replace Lamp	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
Ion Chromatograph (IC)	Replace column disks Change guard columns Check pump seals Replace tubing Replace suppressor Check fluid level in waste container Clean cooling fan grills	As required As required As required As required As required Daily Semiannually
Balances	Class “S” traceable weight check Clean pan and check if level Outside calibration service	Daily, when used Daily At least Annually
Conductivity Meter	0.01M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb Clean sample holder	Daily, when used Daily, when used
Deionized/Distilled Water	Daily conductivity check Check deionizer light Monitor for VOA’s System cleaning Replace cartridge & large mixed bed resins	Daily Daily As required As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	When used As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required
BOD Incubator	Temperature monitoring Incubator cleaning	Daily As required
Centrifuge	Check brushes and bearings	As needed
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed
Automated Solvent Extraction units (ASE)	Check solvent reservoirs Check tubing	Daily Daily

Instrument	Procedure	Frequency
TurboVaps	Check gas lines Check water level Calibrate temperature	Daily Daily Annually
Total Organic Carbon Analyzer	Check gas flow Check reagent reservoir levels Replace o-rings Check autosampler needle Replace scrubbers Replace catalyst	Daily Daily As needed Daily Annually As needed
Automated Analyzer	Clean sampler Check all tubing Clean detector Clean optics and cells	Daily Daily Daily Daily
Infrared Spectrophotometer (IR)	Clean lens/optimize	As needed
Flashpoint Apparatus	Check gas line for leaks Check stirrer speed	Daily Annually
Rotators	Verify rotation speed	Annually

Table 21-3. Periodic Calibration

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using weights calibrated against ASTM Class 1 NIST-traceable weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by an accredited vendor annually.	Daily	± 3 digits at smallest (rightmost) display	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Top Loading Balance	Accuracy determined using weights calibrated against ASTM Class 1 NIST-traceable weights. Minimum of 2 standards bracketing the weight of interest. Inspected and calibrated by an accredited vendor.	Daily	± 3 digits at smallest (rightmost) display	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
Reference ASTM Class 1 NIST-traceable Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per ASTM Class 1 specifications	Replace.

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Daily laboratory weights	Verified against laboratory's ASTM Class 1 reference set	1 year	±0.1mg of expected or less than lowest weight the balance can read	Replace
NIST-Traceable Thermometer	Accuracy determined by accredited measurement laboratory.	5 years	As per certificate.	Replace.
Thermometer, glass	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	Correction factor of ± 2°C	Replace
Thermometer, digital	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	Correction factor of ± 2°C	Replace
InfraRed Temperature Guns	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	Correction factor of ± 2°C	Repair/replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again a few hours later and document	>0 to 6°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Freezer	Temperature checked using NIST-traceable thermometer	range, check again a few hours later and document	-10 to -20°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	104 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Incubator	Temperature checked using NIST-traceable thermometer.	When in use.	BOD: 20 ± 1.0°C	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°C	Adjust. Replace.

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or dispensing devices)	One delivery by weight. Using DI water, dispense into tared vessel. Record weight with device ID number.	Monthly	± 2% Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.
Glass Microliter Syringes	None	Accuracy must be initially de-monstrated if syringe was not received with a certifi-cate attesting to established accuracy.	± 1%	Not applicable.
Conductivity Meter	Cell impedance calibrated with two KCl standards.	Each use.	2nd source verfied within vendor-specified limits	Recalibrate.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Weekly	<1 µmhos/cm^2	Record on log. Report discrepancies to QA Director.

Table 21-4. Preventive Maintenance Procedures For Field Equipment

Instrument/ Equipment Type	Activity	Frequency	Maintenance
Automatic Sampler – ISCO 3710/3910	Check tubing and connections through pump head	Before and after use	Replace tubing when necessary
	Check battery power and program	Before and after use	Replace battery when necessary
	Clean tubing in pump head	After each use	Replace pump head tubing when necessary
	Clean tubing for sample collection	After each use	---
	Check functionality – manual sample; program sample	Prior to use	---
	Check sample container for breakage, etc.	Prior to use	Replace if needed
YSI 3000 – Depth Meter, Temperature, and Conductivity	Check battery	Before and after use	Replace batteries when necessary
	Check cable	Before and after use	Send for repair
	Check probe	Before and after use	Send for repair
	Check LCD	Before and after use	Send for repair
Bailers – Miscellaneous sizes	Check ball valve for overall condition	Prior to use	Clean/replace accordingly

Instrument/ Equipment Type	Activity	Frequency	Maintenance
	Check rope	Before, during and after use	Retie or replace as necessary
	Clean inside and out	Before and after use	---
Residual Chlorine – HACH Kit	Check battery	Before and after use	Replace batteries when necessary
	Inspect glass cells	Before and after use	Replace as necessary
	Clean glass cells	Prior to use	---
Residual Chlorine – HACH Kit	Inspect cell holder	Before and after use	Remove obstructions, if present
	Check expiration dates of reagents	Prior to use	Remove and reorder as necessary
	Inspect ampules for cracks	Before and after use	Replace as necessary
Dissolved Oxygen – HACH Kit	Check battery	Before and after use	Replace batteries when necessary
	Inspect cell holder	Before and after use	Remove obstructions, if present
	Inspect rubber ampule cover	Before and after use	Replace as necessary

SECTION 22

MEASUREMENT TRACEABILITY
(NELAC 5.5.6)

22.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

"Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties." There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as "determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional 'true' value of the measurand."

Uncertainty is defined as "a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand." Measurement of Uncertainty is discussed is Section 20 of this QA Manual.

22.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory. Refer to Section 21 for calibration of weights and thermometers.

22.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. (Refer to Section 9 for additional information on purchasing). The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to Table 9-1 in Section 9 for general storage requirements and Table 22-1 for additional storage information. Please refer to method SOPs "Standards and Reagents" section for additional details. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

22.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and

acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained by each laboratory department. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to the laboratory's SOP IR-QA-STD (Reagent and Standard Preparation, Control, and Documentation) as well as method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

22.4.1 All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS:

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically (with the exception of metals working standards which are prepared daily and documented in a controlled logbook) for standard and reference material

preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

22.4.2 All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID Code (from LIMS or logbook)
- Special Health/Safety warnings if applicable

22.4.3 In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1)with the manufacturer's recommendations; 2) with requirements in the specific analytical methods; and 3) according to Table 22-1.

Table 22-1.

Standard Sources and Preparation

Method Group	Source*	How Received	Stock Storage	Preparation	Intermediate & Working Standard Storage	Frequency
Metals	SPEX; Environmental Express	1000 ppm Solutions	Room Temperature	Working standards from stock	Room Temperature	Daily
Wet Chemistry	Ricca; Spectrum:ERA	Solutions	Refrigerate	As received	Refrigerate	Various
Volatile Organics	Absolute; Restek	Ampoule/ Solutions	Freezer (-10°C)	Working standards from stock	Refrigerate	Monthly; Gas, weekly
Semi-Volatile Organics	Absolute; Restek	Ampoule/ Solutions	Refrigerate or Room temp.	Working standards from stock	Refrigerate	Monthly
Infrared Spec-trophotometry	Aldrich; Sigma	Pure Reagent	Room Temperature	Working standards from stock	Refrigerate	Six months

*Or equivalent

SECTION 23.0

SAMPLING
(NELAC 5.5.7)

23.1 OVERVIEW

TestAmerica Irvine provides sampling services. Sampling procedures are described in the SOP FIELD.SOP (Field Sampling).

23.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory. Additionally, TestAmerica Irvine lot tests all 40-millilter VOA vials for volatile organics by GCMS and all polyethylene bottles for common anions and trace-level metals.

23.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Instra-Analyzed or equivalent
- Sodium Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

23.2.2 Preparing Container Orders

When new containers arrive at the laboratory, the lot numbers for VOA vials are checked against the list of laboratory-approved lots. For polyethylene bottles, the date of receipt is recorded on the box(es) and randomly selected bottles of each unpreserved size and each nitric-preserved size are submitted for metals and anion analysis. Upon request, the containers are then sent to clients for use in collecting samples. The shipping date, type and number of containers are maintained on file by the lab. Shipping personnel insure that container stock is rotated so that “first in” is “first out.” When a client requests containers, a client services representative creates a container request in LIMS; it is then stored permanently in LIMS with a unique container order number. Copies of the container request are printed for the shipping department. One copy goes to the client with the containers; one copy is filed in the shipping department. See the laboratory’s SOP LOGIN.SOP (Sample Control) and LOTTEST.SOP (Container and Reagent Verification by Lot Testing) for more details.

The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

23.3 FIELD QUALITY CONTROL (QC)

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample containers for all volatile organic analyses. Blanks generated in the field will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

23.3.1 Equipment Blank / Rinsate Blank - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected.

23.3.2 Field Blank - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank should be when the blank is prepared in the field.

23.3.3 Trip Blank - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the “Trip” ends).

23.3.4 Field Duplicates - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

23.4 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in “days” (e.g 14 days, 28 days), the holding time is based on calendar day measured. Holding

times expressed in "hours" (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis.

23.4.1 Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date until the day solvent contacts the sample. Holding times for analysis are measured from the date of initiation of extraction to the time of injection into the gas chromatograph.

23.4.2 Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph. The time of initiation of purging is considered the injection time, but data systems record the start of the chromatographic run rather than the start of purging. Hence, if a sample is so near expiration that the start-of-purging time rather than the chromatographic run time is needed to document the integrity of the sample; the analyst must observe and record the start-of-purging time in the instrument log. Extractions, e.g. for high level soils, must be completed in time to allow for analysis to be initiated within the maximum allowable holding time.

23.4.3 Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable).

23.5 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times (refer to Tables 23-1 to 23-7) or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

23.6 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis. In that regard the following guidelines apply to analysts:

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

23.6.1 For water samples, before taking each aliquot for analysis, invert the sample container end-over-end three times and immediately pour off the aliquot. Especially when suspended solids are present, adequate mixing of the sample is extremely important.

23.6.2 For solid samples, when volatile organics are not requested, if the solid can be mixed, stir before removing the aliquot. Mix more than is needed for the analysis to be performed (e.g. if 30 g are needed, mix 50-100 g, if 1 g is needed, mix 20 g, etc…).

- If the solid cannot be easily mixed: After thoroughly mixing the sample within the sample container or, for non-organic methods, the sample can be transferred to a wip bag (or other suitable plastic bag) for manual mixing, a sub-sample from various quadrants and depths of the sample are taken to acquire the required sample weight.
- For soil samples, avoid debris in the subsample aliquot as much as possible (e.g. gravel, sticks, roots and grass); note this information in the sample preparation record.
- If the solid is extremely heterogeneous, and the client has given no instructions, utilize the following technique: separate the like materials into groups on a clean surface and take portions of masses from each group, proportional to their contribution to the original sample, to make a composite. Record in detail exactly how the composite was created. For very unusual samples, consult with the QA department or Department Manager.

NOTE: Subsampling is addressed in greater detail in SUBSAMP.SOP (Subsampling).

23.6.3 For solid samples, when volatile organics analysis is requested, the sample should be manipulated as little as possible. In most cases, the sample will arrive already preserved or in an EnCore™ sampler of the correct mass (requiring quick preservation of the entire amount). If the client requests volatiles on a solid sample which has been collected in a jar and is in a common container from which aliquots for other test methods must be taken, login should deliver the container to the volatiles department for preparing a proper aliquot prior to any other aliquots being taken out.

23.6.4 For multiphasic samples, the client should instruct the laboratory as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst should record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results are combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume.

Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. ***Please note****: the holding times are program specific and different programs may have different holding times for equivalent methods (e.g., there are difference in Holding times for many Organic analytes between SDWA and NPDES. RCRA methods may also be different.)*

Table 23-1.
Holding Times, Preservation and Container Requirements: Drinking Water (SDWA)

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[23]	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Asbestos	Plastic/Glass	4ºC	None	48 hours[5]	1 L
Coliforms (Total and Fecal)	Plastic/Glass[20]	10ºC	$Na_2S_2O_3$	30 hours[21]	120 mL
Cyanide	Plastic/Glass	4ºC	NaOH to pH >12	14 days	500 mL
Fluoride	Plastic/Glass	None	None	None	250 mL
Heterotrophic Plate Count	Plastic/Glass[20]	10ºC	$Na_2S_2O_3$	8 hours (24 hours[22])	120 mL
Mercury	Plastic/Glass	None	HNO_3 to pH<2	28 days	250 mL
Metals[4]	Plastic/Glass	None	HNO_3 to pH<2	6 months	250 mL
Nitrate	Plastic/Glass	4ºC	None	48 hours[6]	250 mL
Nitrate-Nitrite	Plastic/Glass	None	H_2SO_4 to pH<2	28 days	250 mL
Nitrite	Plastic/Glass	4ºC	None	48 hours	250 mL
THMs Only	Glass[8]	4ºC	$Na_2S_2O_3$	14 days	3 X 40 mL
Volatile Organic Compounds	Glass[8]	4ºC	HCl to pH <2 $Na_2S_2O_3$ or Ascorbic acid[9]	14 days	3 X 40 mL
EDB, DBCP, 1,2,3-TCP (EPA 504.1)	Glass[8]	4ºC	$Na_2S_2O_3$	14 days	3 X 40 mL
Organochlorine Pesticides/PCBs (EPA 505)[10]	Glass[8]	4ºC	$Na_2S_2O_3$	14 days[11]	3 X 40 mL
Nitrogen and Phos. Pesticides (EPA 507)	Glass-Amber[8]	4ºC	$Na_2S_2O_3$	14 days[12]	1 L
Total PCBs (EPA 508A)	Glass-Amber[8]	4ºC	None	14 days[13]	1 L
Pesticides and PCBs (EPA 508.1)[14]	Glass-Amber[8]	4ºC	HCl to pH <2 $Na_2S_2O_3$[9]	14 days[13]	1 L
Chlorinated Acids (EPA 515.1)	Glass-Amber[8]	4ºC	$Na_2S_2O_3$	14 days[12]	1 L
Semivolatiles (EPA 525.2)	Glass-Amber[8]	4ºC	HCl to pH <2 $Na_2S_2O_3$[9]	14 days[13]	1 L

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[23]	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
N-Methylcarbamoyloxamines and N-Methcarbamates (EPA 531.1)	Glass[8]	4ºC	$Na_2S_2O_3$, Monochloroacetic Acid buffer to pH<3	28 days	3 X 60 mL
Glyphosate (EPA 547)	Glass[8]	4ºC	$Na_2S_2O_3$	14 days	3 X 60 mL
Endothall (EPA 548)	$Na_2S_2O_3$	4ºC	None	7 days[15]	1 L
Diquat/Parquat (EPA 549.1)	Glass-Amber[8] (Silanized or PVC amber)	4ºC	H_2SO_4 to PH <2 $Na_2S_2O_3$[9]	7 days[16]	1 L
Chlorinated Disinfection Byproducts, Chlorinated Solvents, and Halogenated Pesticides/Herbicides (EPA 551)	Glass[8]	4ºC	Phosphate Buffer and Ammonium Chloride[19]	14 days[17]	3 X 60 mL
Haloacetic Acids (EPA 552.1)	Glass-Amber[8]	4ºC	Ammonium Chloride	28 days[18]	250 mL

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, 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 compliance. For the preservation requirements of Table 6-8, 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 (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) 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).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. All metals except Hg.
5. Instructions for containers, preservation procedures and holding times as specified in Method 100.2 must bed adhered to for all compliance analysis including those conducted with Method 100.1.
6. If the sample is chlorinated, the holding time for an un-acidified sample kept at 4°C is extended to 14 days.
7. Nitrate-Nitrite refers to a measurement of total nitrite.
8. With Teflon lined septum.
9. If chlorinated add $Na_2S_2O_3$ prior to acidification.
10. Heptaclor has a 7 day hold time
11. 14 days until extraction. 24 hours after extraction.
12. 14 days until extraction. 28 days after extraction.
13. 14 days until extraction. 30 days after extraction.
14. For cyanazine, cool to 4°C only.

Key to Table

15. 7 days until derivitation. 1 day after derivatation.
16. 7 days until extraction. 21 days after extraction.
17. 14 days until extraction. 14 days after extraction.
18. 28 days until extraction. 48 hours after extraction.
19. Sodium Sulfite may be used as a dechlorinating agent in some instances. Verify with laboratory prior to sampling.
20. Sterilized. Plastic must be Polypropylene.
21. 40 CFR part 141.74 regulations to avoid filtration or disinfection state 8 hours (DW compliance testing). Most facilities are using either disinfection or filtration so the 8 would not apply in most cases.
22. 40 CFR part 141.74 regulations for Disinfection By-Product rule state 8 hours (DW compliance testing) where SM 9215 allows up to 24 hours if sample is stored between > 0 and $\leq 4^{o}$ C
23. For samples with a temperature requirement of 4^{o}C, a sample temperature of just above the water freezing temperature to $\leq 6^{o}$C is acceptable.

Table 23-2
Holding Times, Preservation and Container Requirements: NPDES – Bacteria, Protozoa, Toxicity Tests

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.	PRESERVATION[2,3] Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Total, Fecal, and E.coli Coliforms	Plastic/Glass	10ºC	0.0008 % $Na_2S_2O_3$[5]	6 hours	100 mL
Fecal Streptococci	Plastic/Glass	10ºC	0.0008 % $Na_2S_2O_3$[5]	6 hours	100 mL
Enterococci	Plastic/Glass	10ºC	0.0008 % $Na_2S_2O_3$[5]	6 hours	100 mL
Cryptosporidium	LPDE Plastic	0-8ºC	None	96 Hours	500 mL
Giardia	LPDE Plastic	0-8ºC	None	96 Hours	500 mL
Toxicity – Acute/Chronic	Plastic/Glass	$\leq$ 6ºC[5]	None	36 Hours	2 L

Key to Table

1. Plastic should be Polypropylene or other sterilizable plastic.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, 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 compliance. For the preservation requirements of Table 6-8, 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 (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) 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 analysis and still be considered valid.
5. Samples must not be frozen. Sufficient ice should be placed 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 samples arrive, it is necessary to measure the temperature of the samples and confirm that the $\leq$ 6°C temperature has not been exceeded.
6. Should only be used in the presence of residual chlorine.

Table 23-3
Holding Times, Preservation and Container Requirements: NPDES - Inorganic

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp[14].	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Acidity	Plastic/Glass	≤ 6ºC	None	14 days	100 mL
Alkalinity	Plastic/Glass	≤ 6ºC	None	14 days	100 mL
Ammonia	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH<2	28 days	400 mL
BOD 5 Day	Plastic/Glass	≤ 6ºC	None	48 hours	1000 mL
Boron	Plastic[5]	None	HNO_3 to pH<2	6 months	200 mL
Bromide	Plastic/Glass	None	None	28 days	100 mL
CBOD 5 Day	Plastic/Glass	≤ 6ºC	None	48 hours	1000 mL
COD	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH<2	28 days	100 mL
Chloride	Plastic/Glass	None	None	28 days	50 mL
Chlorine, Residual	Plastic/Glass	None	None	15 min.[6]	200 mL
Color	Plastic/Glass	≤ 6ºC	None	48 hours	50 mL
Cyanide -Total	Plastic/Glass	≤ 6ºC	NaOH to pH >12, 0.6 g ascorbic Acid[7]	14 days	100 mL
Cyanide -Amenable	Plastic/Glass	≤ 6ºC	NaOH to pH >12, 0.6 g ascorbic Acid[7]	14 days	100 mL
Fluoride	Plastic	None	None	28 days	300 mL
Hardness	Plastic/Glass	None	HNO_3 to pH<2[8]	6 months	100 mL
Hexavalent, Chromium	Plastic/Glass	≤ 6ºC	Ammonium sulfate buffer pH = 9.3 - 9.7	28 dys / 24 hrs[15]	200 mL
Hydrogen Ion (pH)	Plastic/Glass	None	None	15 min.[6]	200 mL
Kjeldahl and organic Nitrogen	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	500 mL
Mercury[11]	Plastic/Glass	None	HNO_3 to pH<2	28 days	200 mL
Metals[9,10]	Plastic/Glass	None	HNO_3 to pH<2	6 months	200 mL
Nitrate	Plastic/Glass	≤ 6ºC	None	48 hours	100 mL
Nitrate-Nitrite	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	100 mL
Nitrite	Plastic/Glass	≤ 6ºC	None	48 hours	100 mL
Oil and Grease	Glass	≤ 6ºC	H_2SO_4 or HCl to pH <2	28 days	1 L

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp[14].	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Organic Carbon (TOC)	Plastic/Glass	≤ 6ºC	H_2SO_4 or HCl to pH <2[12]	28 days	250 mL
Orthophosphate	Plastic/Glass	≤ 6ºC	Filter within 15 min.	48 hours	250 mL
Oxygen, Dissolved Probe	Glass[13]	None	None	15 min.[6]	200 mL
Oxygen, Winkler	Glass[13]	None	Fix on site and store in dark.	8 hours	300 mL
Phenols	Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	500 mL
Phosphorus, Elemental	Glass	≤ 6ºC	None	48 hours	250 mL
Phosphorus, Total	Plastic/Glass	≤ 6ºC	H_2SO_4 to pH <2	28 days	250 mL
Residue, Total	Plastic/Glass	≤ 6ºC	None	7 days	1 L
Residue, Filterable	Plastic/Glass	≤ 6ºC	None	7 days	1 L
Residue, Non-Filterable	Plastic/Glass	≤ 6ºC	None	7 days	1 L
Residue, Settleable	Plastic/Glass	≤ 6ºC	None	48 hours	1 L
Residue, Volatile	Plastic/Glass	≤ 6ºC	None	7 days	1 L
Silica	Plastic[5]	≤ 6ºC	None	28 days	250 mL
Specific Conductance	Plastic/Glass	≤ 6ºC	None	28 days	250 mL
Sulfate	Plastic/Glass	≤ 6ºC	None	28 days	250 mL
Sulfide	Plastic/Glass	≤ 6ºC	Zinc acetate plus NaOH to pH>9	7 days	500 mL
Sulfite	Plastic/Glass	None	None	15 min.[6]	200 mL
Surfactants	Plastic/Glass	≤ 6ºC	None	48 hours	1 L
Temperature	Plastic/Glass	None	None	N/A	100 mL
Turbidity	Plastic/Glass	≤ 6ºC	None	48 hours	1 L

Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at ≤ 6ºC until compositing and sample splitting is completed.

Key to Table

3. When any sample is to be shipped by common carrier or sent through the United States mails, 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 compliance. For the preservation requirements of Table 6-8, 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 (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) 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 analysis and still be considered valid.
5. May also be collected in quartz or PFTE Plastic.
6. For compliance testing, the analysis must be performed in the field at the time of analysis. If transported to the laboratory for analysis, the analysis will be performed as soon as practical and reported qualified.
7. Should only be used in the presence of residual chlorine.
8. H_2SO_4 to a pH <2 is also acceptable.
9. Except Mercury and Hexavalent Chromium.
10. Samples should be filtered on site before adding HNO_3 preservative for dissolved metals.
11. Samples collected for determination of trace level mercury (100 ng/L) using EPA 1631 must be collected in tightly capped fluoropolymer or glad 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. Samples collected for dissolved trace level mercury should be filtered in the laboratory. However, if circumstances prevent overnight shipping, samples should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. Samples that been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.
12. Phosphoric acid (H_3PO_4) may also be used.
13. Should have glass lid or top.
14. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, 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 ‘‘≤ °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).

15. Holding time is 24 hours if pH adjustment is not performed.

Table 23-4
Holding Times, Preservation and Container Requirements: NPDES - Organic

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.[15]	PRESERVATION[1,2] Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Purgeable Halocarbons	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5]	14 days	40 mL
Purgeable Aromatic Hydrocarbons	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5], HCl to pH<2	14 days[6]	40 mL
Acrolein and Acrylonitrile	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5], adjust pH to 4-5[7]	14 days	40 mL
Phenols[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[8]	1 L
Benzidines[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[8, 11]	1 L
Phthalate esters[9]	Glass[4]	≤ 6ºC	None	7 days[8]	1 L
Nitosamines[9,12]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
PCBs[9]	Glass[4]	≤ 6ºC	None	1 year[8]	1 L
Nitroaromatics and Isophorone[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
Polynuclear Aromatic Hydrocarbons[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5,13]	7 days[8]	1 L
Haloethers[9]	Glass[4]	≤ 6ºC	0.0008 % $Na_2S_2O_3$[5]	7 days[8]	1 L
Chlorinated Hydrocarbons[9]	Glass[4]	≤ 6ºC	None	7 days[8]	1 L
CDD/CDFs[9] – Aqueous: Field/Lab Preservation	Glass	≤ 6ºC	pH <9, 0.0008 % $Na_2S_2O_3$[5]	1 year	1 L
CDD/CDFs[9] – Solids/Mixed Phase/ - Field Preservation	Glass	≤ 6ºC	None	7 days	1 L
CDD/CDFs[9] – Tissue – Field Preservation	Glass	≤ 6ºC	None	24 hours	
CDD/CDFs[9] – Solids/Mixed Phase/Tissue - Lab Preservation	Glass	< -10ºC	None	1 year	1 L
Pesticides[9]	Glass	≤ 6ºC	pH 5-9 [14]	7 days[8]	1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at $\leq 6^{o}C$ until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, 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 compliance. For the preservation requirements of Table 6-8, 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 (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) 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).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
4. With Teflon lined septum.
5. Should only be used in the presence of residual chlorine.
6. Samples receiving no pH adjustments must be analyzed within 7 days. If 2-chlorovinylethylether is a target analyte, the sample should not be acidified.
7. The pH adjustment is not required if acrolein is not being measured. Samples for acrolein receiving no pH adjustment must be analyze within three days of sampling.
8. 7 days until extraction, 40 days after extraction. (PCB only – 1 year after extraction)
9. 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. When the analytes of concern fall within two or more categories, the sample may be preserved by cooling to $\leq 6^{o}C$ reducing residual chlorine with 0.0008 % sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine) and footnotes 10 and 11(re the analysis of Benzidine).
10. If 1,2-diphenylhydrazine is likely to be present, adjust pH to of the sample to 4.0 $\pm$ 0.2 to prevent rearrangement to benzidine.
11. Extracts may be stored up to 30 days before analysis if storage temperature is $< 0^{o}C$.
12. For the analysis of diphenylnitrosamine, add 0.008 % $Na_2S_2O_3$ and ajust pH to 7-10 with NaOH within 24 hours of sampling.
13. Store in dark.
14. The pH adjustment may be performed upon receipt in the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin , add 0.0008 % $Na_2S_2O_3$.
15. Aqueous samples must be preserved at ≤6 °C unless otherwise indicated, 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 ‘‘≤ °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).

Table 23-5.
Holding Times, Preservation and Container Requirements: NPDES - Radiological

PARAMETER	CONTAINER	PRESERVATION[1,2] Temp.	Chemical	HOLDING TIME[3]	SAMPLE VOLUME
Alpha, Beta, Radium	Plastic/Glass	None	HNO_3 to pH<2	6 months	1 L

Key to Table

1. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
2. When any sample is to be shipped by common carrier or sent through the United States mails, 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 compliance. For the preservation requirements of Table 6-8, 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: Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater).
3. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Table 23-6.
Holding Times, Preservation and Container Requirements: RCRA - Aqueous

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.[12]	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Chloride	Plastic/Glass	4ºC	None	28 days	100 mL
Cyanide -Total	Plastic/Glass	4ºC	NaOH to pH >12[5]	14 days	250 mL
Cyanide -Amenable	Plastic/Glass	4ºC	NaOH to pH >12[5]	14 days	250 mL
Hydrogen Ion (pH)	Plastic/Glass	4ºC	None	24 hours[11]	100 mL
Nitrate	Plastic/Glass	4ºC	None	48 hours	28 days
Oil and Grease	Glass	4ºC	HCl	28 days	1 L
Organic carbon (TOC)	Plastic/Glass	4ºC	pH to <2[6] Store in dark	28 days	28 days
Sulfate	Plastic/Glass	4ºC	None	28 days	400 mL
Sulfide	Plastic/Glass	4ºC	Add Zn Acetate	7 days	400 mL
Chromium VI	Plastic/Glass	4ºC	None	24 hours	250 mL
Mercury	Plastic/Glass	None	HNO_3 to pH<2	28 days	250 mL
Other Metals	Plastic/Glass	None	HNO_3 to pH<2	6 months	250 mL
Acrolein and Acrylonitrile	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7], Adjust pH to 4-5[13]	14 days	1 L
Benzidines	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Chlorinated Hydrocarbons	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Dioxins and Furans	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Haloethers	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L
Nitroaromatics and cyclic ketones	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Nitrosomines	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Organochlorine Pesticides	Glass[10]	4ºC	None	7 days[8]	1 L
Organophosphorus Pesticides	Glass[10]	4ºC	Adjust pH[9]	7 days[8]	1 L
PCBs	Glass[10]	4ºC	None	7 days[8]	1 L
Phenols	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	7 days[8]	1 L

PARAMETER	CONTAINER [1]	PRESERVATION[2,3] Temp.[12]	Chemical	HOLDING TIME[4]	SAMPLE VOLUME
Phthalate Esters	Glass[10]	4ºC	None	7 days[8]	1 L
Polynuclear Aromatic Hydrocarbons	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7], store in dark	7 days[8]	1 L
Purgeable Hydrocarbons	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7] Adjust pH <2[2]	14 days	40 mL
Purgeable Halocarbons	Glass[10]	4ºC	0.0008 % $Na_2S_2O_3$[7]	14 days	40 mL
Total Organic Halides (TOX)	Glass[10]	4ºC	Adjust pH to <2 with H_2SO_4	28 days	1 L
Radiological Tests (Alpha, Beta, Radium)	Plastic/Glass	None	HNO_3 to pH<2	6 months	250 mL

Key to Table

1. Plastic should be Polyethylene.
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States mails, 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 compliance. For the preservation requirements of Table 6-8, 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 (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) 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 analysis and still be considered valid.
5. If oxidizing agents are present, add 5 mL 0.1 N $NaAsO_2$ or 0.06 g of ascorbic acid per L. See Cyanide SOP for additional information about other interferences.
6. Adjust pH to <2 with H_2SO_4, HCl, or solid $NaHSO_4$. Free Chlorine must be removed prior to adjustment.
7. Free Chlorine must be removed by the appropriate addition of $Na_2S_2O_3$.
8. 7 days until extraction. 40 days after extraction.
9. Adjust pH to 5-8 using NaOH or H_2SO_4.
10. With Teflon lined septum.
11. Holding Time is listed as “As Soon as Possible” in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
12. For samples with a temperature requirement of 4°C, a sample temperature of just above the water freezing temperature to $\leq$ 6°C is acceptable.
13. Based on guidance from EPA MICE, if samples are received without pH adjustment, the holding time is 7 days.

Table 23-7.
Holding Times, Preservation and Container Requirements: RCRA – Non-Aqueous

PARAMETER	CONTAINER [1]	PRESERVATION Temp.[7]	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Chloride	Glass	4ºC	None	28 days	50 g
Cyanide -Total	Glass	4ºC	None	14 days	50 g
Cyanide -Amenable	Glass	4ºC	None	14 days	50 g
Hydrogen Ion (pH)	Glass	4ºC	None	7 days[6]	50 g
Nitrate	Glass	4ºC	None	N/A	50 g
Oil and Grease	Glass	4ºC	None	28 days	50 g
Sulfide	Glass	4ºC	Add Zn Acetate, zero headspace	7 days	50 g
Chromium VI	Glass	4ºC	None	24 hours	50 g
Mercury	Plastic/Glass	None	None	28 days	50 g
Other Metals	Plastic/Glass	None	None	6 months	50 g
Acrolein and Acrylonitrile	Glass[4]	4ºC	None	14 days	50 g
Benzidines	Glass[4]	4ºC	None	14 days[3]	50 g
Chlorinated Hydrocarbons	Glass[4]	4ºC	None	14 days[3]	50 g
Dioxins and Furans	Glass[4]	4ºC	None	14 days[3]	50 g
Haloethers	Glass[4]	4ºC	None	14 days[3]	50 g
Nitroaromatics and cyclic ketones	Glass[4]	4ºC	None	14 days[3]	50 g
Nitrosomines	Glass[4]	4ºC	None	14 days[3]	50 g
Organochlorine Pesticides	Glass[4]	4ºC	None	14 days[3]	50 g
Organophosphorus Pesticides	Glass[4]	4ºC	None	14 days[3]	50 g
PCBs	Glass[4]	4ºC	None	14 days[3]	50 g
Phenols	Glass[4]	4ºC	None	14 days[3]	50 g
Phthalate Esters	Glass[4]	4ºC	None	14 days[3]	50 g
Polynuclear Aromatic Hydrocarbons	Glass[4]	4ºC	None	14 days[3]	50 g

PARAMETER	CONTAINER [1]	PRESERVATION Temp.[7]	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Purgeable Hydrocarbons	Glass[4]	4ºC	None	14 days[5]	50 g
Purgeable Halocarbons	Glass[4]	4ºC	None	14 days[5]	50 g
Total Organic Halides (TOX)	Glass[4]	4ºC	None	28 days	50 g

Key to Table

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. 14 days until extraction. 40 days after extraction.
4. With Teflon Lined Septum
5. See Volatile SOP for more detailed preservation requirements.
6. Holding Time is listed as “As Soon as Possible” in SW 846. Per EPA MICE, the recommended maximum holding time for pH in water is 24 hours and pH in soil is 7 days. There are no mandated regulatory requirements.
7. For samples with a temperature requirement of 4^{o}C, a sample temperature of just above the water freezing temperature to $\leq$ 6^{o}C is acceptable.

Table 23-8.
Holding Times, Preservation and Container Requirements: Air Samples

PARAMETER	CONTAINER [1]	PRESERVATION Temp.	PRESERVATION Chemical	HOLDING TIME[2]	SAMPLE WEIGHT
Volatile Organics	Summa Cannister	None	None	30 days	6L or 1L
Volatile Organics	Tedlar Bag	None	None	72 hrs[3,4]	1 L

Key to Table

1. Plastic should be Polyethylene.
2. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.
3. Holding Time is based on SW 846 Method 0040 “SAMPLING OF PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS FROM COMBUSTION SOURCES USING TEDLAR® BAGS”. Some states specifically enforce this holding time (e.g. Florida, New Jersey) and others have not specified this information in their regulatory requirements.
4. The holding time is 72 hours unless the laboratory has a documented validation study that indicates a longer HT is acceptable for the analytes of interest.

SECTION 24

HANDLING OF SAMPLES
(NELAC 5.5.8)

Sample management procedures at TestAmerica Irvine ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

24.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and can be initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

24.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

Note: Independent couriers (e.g. FedEx) are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is attached to the COC and kept with the entire project file.

24.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal (Figure 24-2), retain the shipping record with the COC, and initiate an internal COC (Figure 24-3) for laboratory use by analysts and a sample disposal record (Figure 24-4).

24.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

24.2.1 Laboratory Receipt

(See LOGIN.SOP (Sample Control) for more details on sample receipt procedures)

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. On a client-specific basis, a Project Receipt Checklist may be filled out to document custody seals, cooler temperatures, preservation, and notifications of discrepancy. See Figure 24-6. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Notification of Discrepancy Form (NOD). See Figure 24-7. Discrepancies are forwarded to the Project Manager and are brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

24.2.1.1 Inspection of samples include a check for:

- Complete documentation to include sample identification, location, date and time of collection, collector's name, preservation type, sample type and any additional comments concerning the samples.
- Complete sample labels to include unique identification in indelible ink.
- Use of appropriate sample containers (see Section 23)

- Adherence to holding times as specified in the test method and/or summarized in Section 23.
- Adequate sample volume for required analyses (see Section 23).
- Damage or signs of contamination to sample container. Volatile vials are also inspected for headspace

24.2.1.2 Check and record the temperature of the samples, temperature blanks, that require thermal preservation.

- Samples shall be deemed acceptable if arrival temperature is just above freezing and less than or equal to 6.0° C. Samples that are hand-delivered immediately after collection may not be at the required temperatures; however, if there is evidence that the chilling process has begun, such as the arrival on ice, the samples shall be considered acceptable. This will be documented on the COC **.**
- If the samples were shipped in ice and solid ice is still present and in direct contact with samples, report the samples as "received on ice." Direct contact means samples must be surrounded by ice cubes or crushed ice. Ice present in a plastic bottle or other container does not constitute direct contact. Samples shipped with only “blue ice” may not be reported as “received on ice”.

24.2.1.3 Verify sample preservation as specified in the test method by inspection of the preservation listed on the container. Actual pH is verified by the laboratory at the time of analysis and documented on a benchsheet or runlog. Chlorine is checked at the time of analysis on samples requiring extractable organics, BOD, TOX, cyanide, fluoride, ammonia, TKN, CBOD and Nitrate; presence or absence is recorded.

24.2.1.4 After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

24.2.1.5 If samples are received without a COC, TestAmerica will provide a generic COC form to be completed by the client when the samples are brought to the laboratory. The client is always provided with a copy of the completed COC form for their records.

24.2.1.6 If analyses with short holding times are requested, the dates and times are inspected to ensure that holding times have not already expired.

24.2.1.7 Samples received after normal working hours are left in their coolers and placed in the walk-in refrigerator**.** The person receiving the samples must record the date and time received, the presence or absence of ice and custody seals, the temperature of samples, presence and type of packing material, and initials.

24.2.1.8 Any deviations from the checks described in Section 24.2.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance criteria (Section 24.3) are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples , or

- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

24.2.2 Sample Log-in

All samples that are received by the laboratory are logged into the LIMS to allow the laboratory to track and evaluate sample progress. Each group of samples that are logged in together (typically one project from a given client/sampling event) is assigned a unique job number. Within each job, each sampling point (or sample) receives a unique number. Sample numbers are generated sequentially over time, and are not re-assigned. A sample may be composed of more than one bottle since different preservatives may be required to perform all analyses requested. Even if multiple containers are received for a single sample, each container is uniquely identified with an alphabetic letter added to the sample number. The LIMS generates sample labels that are attached to each bottle for a given sample.

Each job/set of samples is logged into LIMS with a minimum of the following information:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information (most of this information is “default information” that is stored in the LIMS).
- Date and time sampled;
- Date and time received;
- Job and/or project description, sample description;
- Sample matrix, special sample remarks;
- Reporting requirements (i.e., QC level, report format, invoicing format);
- Turn-around-time requirements;
- Parameters (methods and reporting limits or MDLs are default information for a given parameter)

24.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 24-5) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method;
- sample holding times must be adhered to;
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

24.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. (Exception: preserved metals samples are stored at room temperature.) Samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks. See REFBLANK.SOP (Refrigerator Storage Blank) for more details.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for three weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are stored for an additional three weeks before they are disposed of. This six week holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, a Hazardous Sample Notice must be completed by the analyst. This form may be completed by Sample Control, Project Managers, or analysts and must be attached to the report. The sample itself is clearly marked with a red stamp, stamped on the sample label reading “HAZARDOUS” or “FOREIGN SOIL” and placed in a colored and/or marked bag to easily identify the sample. The date, log number, lab sample number, and the result or brief description of the hazard are all written on the Hazardous & Foreign Soil Sample Notice. A copy of the form must be included with the original COC and Work Order and the original must be given to the Sample Control Custodian. Analysts will notify Sample Control of any sample determined to be hazardous after completion of analysis by completing a

Hazardous Sample Notice. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm that lab-packs all hazardous samples and removes them from the laboratory. Foreign soil samples are sent out for incineration by a USDA-approved waste disposal facility.

24.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses, a trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

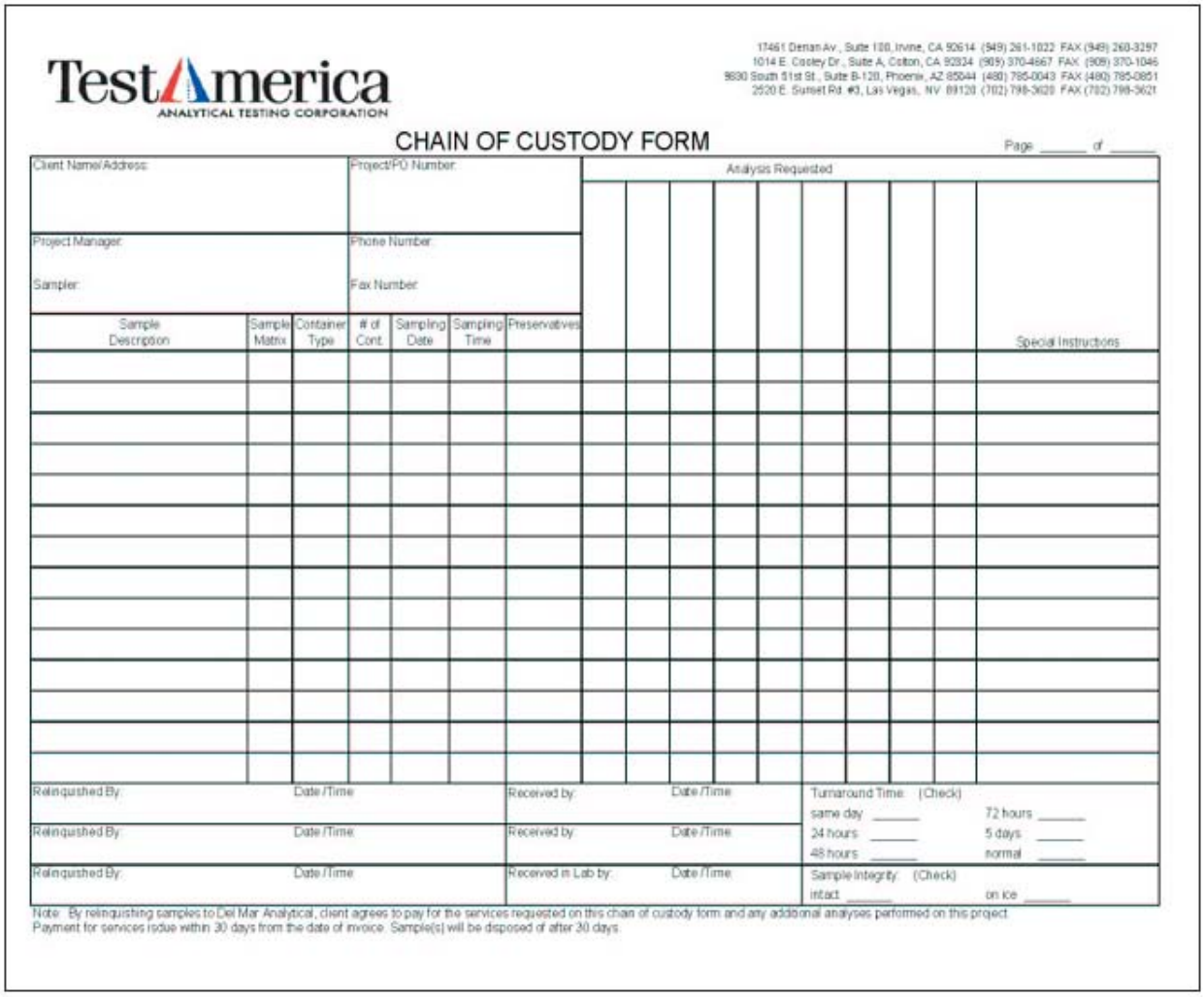
24.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures as documented in the laboratory's Chemical Hygiene Plan. All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than six weeks from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record (Figure 24-4) should be completed.

Figure 24-1.

Example: Chain of Custody (COC)



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2620 E. Sunset Rd. #3, Las Vegas, NV 89120 (702) 798-3620 FAX (702) 798-3621

CHAIN OF CUSTODY FORM

Page _____ of _____

Client Name/Address:	Project/PO Number:	Analysis Requested
Project Manager:	Phone Number:	
Sampler:	Fax Number:	

Sample Description	Sample Matrix	Container Type	# of Cont.	Sampling Date	Sampling Time	Preservatives									Special Instructions

Relinquished By:	Date /Time:	Received by:	Date /Time:	Turnaround Time: (Check)	
Relinquished By:	Date /Time:	Received by:	Date /Time:	same day _____	72 hours _____
Relinquished By:	Date /Time:	Received in Lab by:	Date /Time:	24 hours _____	5 days _____
				48 hours _____	normal _____
				Sample Integrity: (Check)	
				intact _____	on ice _____

Note: By relinquishing samples to Del Mar Analytical, client agrees to pay for the services requested on this chain of custody form and any additional analyses performed on this project. Payment for services is due within 30 days from the date of invoice. Sample(s) will be disposed of after 30 days.

Figure 24-2.

Example: Custody Seal

Figure 24-3.

Example: Internal Chain of Custody (COC)

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WORK ORDER
IPL2715

Client:
Client Code:
Project Manager:

Project Name:
Project Number:
Printed:

Internal Sample Custody

Refrigerator ID:

Sample	In	Out	In	Out	In	Out	In	Out	Archived	Disposed
IFL2715-01G										
IFL2715-01H										
IFL2715-01I										
IFL2715-01L										
IFL2715-01M										
IFL2715-01P										
IFL2715-01Q										
IFL2715-01R										
IFL2715-01S										
IFL2715-01T										

Reviewed By _____ Date _____ Time _____

12/28/2006 10:08:48AM
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Figure 24-4.

Example: Sample Disposal Record

SAMPLE ARCHIVE/DISPOSAL TRACKING FORM

Month/Year: ____________________

Matrix: ____________________

Sample Numbers		Date Removed	Initials	Date Disposed	Initials	Comments
Starting	Ending					

\\Dmai\Office\Depts\QUALITY\Archive-disposal form.doc

Figure 24-5a.

Example: Sample Acceptance Policy, page 1

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TestAmerica Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
   - Client name, address, phone number and fax number (if available)
   - Project name and/or number
   - The sample identification
   - Date, time and location of sampling
   - The collectors name
   - The matrix description
   - The container description
   - The total number of each type of container
   - Preservatives used
   - Analysis requested
   - Requested turnaround time (TAT)
   - Any special instructions
   - Purchase Order number or billing information (e.g. quote number) if available
   - The date and time that each person received or relinquished the sample(s), including their signed name.
   - The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.
   - **Information must be legible**

2) Samples must be properly labeled.
   - Use durable labels (labels provided by TestAmerica are preferred)
   - Include a unique identification number
   - Include sampling date and time & sampler ID
   - Include preservative used.
   - Use indelible ink
   - **Information must be legible**

3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. See TA Sample Container Guide.

4) Samples must be preserved according to the requirements of the requested analytical method (See TA Sample Container Guide). Most analytical methods require chilling samples to 4^{0} C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6^{0} C and above freezing (0^{0}C). For methods with other temperature criteria (e.g. some bacteriological methods require $\leq$ 10 ^{0}C), the samples must arrive within $\pm$ 2^{0} C of the required temperature or within the method specified range. **Note:** Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

Continued on other side.

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Figure 24-5b.

Example: Sample Acceptance Policy, page 2

- Chemical preservation (pH) will be verified prior to analysis and the project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.

- For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
  - 1. Test for residual chlorine in the field prior to sampling.
    - If no chlorine is present, the samples are to be preserved using HCl as usual.
    - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCl.
  - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCl after filling the VOA vial with the sample.

5) Sample Holding Times
   - TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (working days) remaining on the holding time for us to ensure analysis.

   - Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab. However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. The actual times of all "field" sample analyses are noted on the "Short Hold Time Detail Report" in the final report. Only samples analyzed outside of these criteria will be qualified on the final report with an 'H' to indicate holding time exceedance.

6) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply a blank with the bottle order.

7) The project manager will be notified if any sample is received in damaged condition TestAmerica will request that a sample be resubmitted for analysis.

8) Recommendations for packing samples for shipment.

   - Pack samples in Ice rather than "Blue" ice packs.

   - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.

   - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.

   - Fill extra cooler space with bubble wrap.

G:\Depts\QUALITY\FORMS\SMPACPT6.DOC

Updated January 31, 2008