Samples for alkalinity are collected with a peristaltic pump and tubing into new, 20 ml HDPE plastic containers with conical caps. The samples are stored refrigerated at 4 degrees Celsius until analysis, which should occur within 2 weeks. The samples are warmed to room temperature and then analyzed with an Orion 720A pH meter and Radiometer combination electrode. The sample is titrated to an endpoint pH of approximately 3.557 by adding 0.05N HCl to 16 mls sample at the Hasler Lab (or 0.01N HCl to 4 mls sample at Trout Lake Station Lab) in 10 microliter increments using a micro-pipette. The pH meter millivolt readings (along with the corresponding the amount of acid added) of the last 10 acid additions prior to the endpoint are recorded.

The detection limit for the gran alkalinity titration is approximately 5 micro-equivalents per liter of CO3 and the analytical range for the method extends to 4000 micro-equivalents per liter of CO3.

Method Log: Prior to 1986 and since 2002, alkalinity titrations were performed as described above. During the period of February 1986 – November 2001, the alkalinity determinations for Trout, Sparkling, Allequash and Big Muskellunge Lakes were made by a Brinkmann 636 Titroprocessor using 0.05N HCl with 16 mls of sample.

LTER Keywords
sampling
ph
chemistry
alkalinity
Protocol Format
Parameter
Protocol ID
param_alkalinity1
Protocol Type
field & laboratory

Samples for ammonium and nitrate/nitrite are collected together with a peristaltic pump and tubing and in-line filtered (through a 0.40 micron polycarbonate filter) into new, 20 ml HDPE plastic containers with conical caps. The samples are stored frozen until analysis, which should occur within 6 months. The samples are analyzed for ammonium (and nitrate/nitrite) simultaneously by automated colorimetric spectrophotometry, using a segmented flow autoanalyzer. Ammonium is determined by utilizing the Berthelot Reaction, producing a blue colored indophenol compound, where the absorption is monitored at 660 nm.

The detection limit for ammonium is approximately 3 ppb and the analytical range for the method extends to 4000 ppb.

Method Log: Prior to January 2006 samples, ammonium was determined on a Technicon segmented flow autoanalyzer. From 2006 to present, ammonium is determined by an Astoria-Pacific Astoria II segmented flow autoanalyzer.

LTER Keywords
sampling
chemistry
ammonium
Protocol Format
Parameter
Protocol ID
param_ammonium1
Protocol Type
field & laboratory

Samples for calcium analysis (as well as dissolved nitrogen and phosphorus, silicon, magnesium, sodium, potassium, iron, and manganese) are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 ml of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

Calcium, as well as magnesium, sodium, potassium, iron, and manganese are analyzed simultaneously on an optical inductively-coupled plasma emission spectrophotometer (ICP-OES). The acidified samples are directly aspirated into the instrument without a digestion. Calcium is analyzed at 317.933 nm and at 315.887 nm and viewed axially for low-level analysis and radially for high level analysis.

The detection limit for calcium is 0.06 ppm with an analytical range of the method extends to 50 ppm.

Method Log: Prior to January 2002, calcium was determined on a Perkin-Elmer model 503 Atomic Absorption Spectrophotometer. Lanthanum at a 0.8% concentration was added as a matrix modifier to suppress chemical interferences. From January 2002 to present, samples are analyzed for calcium on a Perkin-Elmer model 4300 DV ICP.

LTER Keywords
calcium
cations
chemistry
ions
sampling
Protocol Format
Parameter
Protocol ID
param_calcium1
Protocol Type
field & laboratory

Samples for chloride and sulfate are collected together with a peristaltic pump and tubing and in-line filtered (through a 0.40 micron polycarbonate filter) into new, 20 ml HDPE plastic containers with conical caps. The samples are stored refrigerated at 4 degrees Celsius until analysis, which should occur within 6 months. The samples are analyzed for chloride (and sulfate) simultaneously by Ion Chromatography, using a hydroxide eluent.

The detection limit for chloride is approximately 0.01 ppm and the analytical range for the method extends to 100 ppm.

Method Log: Prior to January 1998 samples, chloride was determined on a Dionex DX10 Ion Chromatograph, using a chemical fiber suppressor. From 1998 to 2011, chloride was determined by a Dionex model DX500, using an electro-chemical suppressor. From January 2011 until present, chloride is determined by a Dionex model ICS 2100 using an electro-chemical suppressor.

LTER Keywords
chemistry
chloride
sampling
Protocol Format
Parameter
Protocol ID
param_chloride1
Protocol Type
field & laboratory

Samples for silicon analysis (as well as dissolved nitrogen and phosphorus, calcium, magnesium, sodium, potassium, iron, and manganese) are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 ml of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

Dissolved reactive silica is determined by the Heteropoly Blue Method and the absorption is measured at 820 nm.

The detection limit for silicon is 6 ppb and the analytical range is 15000 ppb.

Method Log These determinations were performed manually using a Bausch and Lomb Spectrophotometer from the beginning of the project until April 1984. From 1984 through 2005, dissolved reactive silicon was determined on a Technicon Auto Analyzer II. From January 2006 to present, samples are run on an Astoria-Pacific Astoria II Autoanalyzer.

LTER Keywords
chemistry
sampling
silica
silicon
Protocol Format
Parameter
Protocol ID
param_dissolved_reactive_silicon1
Protocol Type
field & laboratory

Using a peristaltic pump, water is pumped at a modest pace (< 15psi) through in-line filtration cartridges, loaded with 47mm, Millipore glass fiber filters. Filtered water volume is recorded. Cartridges are stored and transported in a cooler with ice.

In a dark, cool environment, filters are removed from the cartridges, folded in half (filtered material on the inside), placed in covered film canisters, and stored in a commercial freezer. The canisters are removed from the freezer, and 25 mL of methanol is added to each. The canisters are capped, shaken lightly, and placed in the refrigerator for 24 hours. The methanol/chlorophyll solution is analyzed using a Turner Designs TD-700 fluorometer.

LTER Keywords
chlorophyll
chlorophyll a
Protocol Format
Parameter
Protocol ID
param_fluoro_chlor1
Protocol Type
field & laboratory

Samples for inorganic and organic carbon are collected together with a peristaltic pump and tubing and in-line filtered, if necessary, (through a 0.40 micron polycarbonate filter) into glass, 24 ml vials (that are compatible with the carbon analyzer autosampler), and capped with septa, leaving no head space. The samples are stored refrigerated at 4 degrees Celsius until analysis, which should occur within 2-3 weeks.

The detection limit for inorganic carbon is 0.15 ppm, and the analytical range for the method is 60 ppm.

Method Log: Prior to May 2006 samples, inorganic carbon was analyzed by phosphoric acid addition on an OI Model 700 Carbon Analyzer. From May 2006 to present, inorganic carbon is still analyzed by phosphoric acid addition, but on a Shimadzu TOC-V-csh Total Organic Carbon Analyzer.

LTER Keywords
carbon
chemistry
dissolved inorganic carbon
inorganic nutrients
sampling
Protocol Format
Parameter
Protocol ID
param_inorganic_carbon1
Protocol Type
field & laboratory

Samples for iron analysis (as well as dissolved nitrogen and phosphorus, silicon, calcium, sodium, potassium, magnesium, and manganese) are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 ml of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

Iron, as well as calcium, sodium, potassium, magnesium, and manganese are analyzed simultaneously on an optical inductively-coupled plasma emission spectrophotometer (ICP-OES). The acidified samples are directly aspirated into the instrument without a digestion. Iron is analyzed at 238.204 nm and at 239.562 nm and viewed axially for low-level analysis and radially for high level analysis.

The detection limit for iron is 0.02 ppm with an analytical range of the method extends to 20 ppm.

Method Log: Prior to January 2002, iron was determined on a Perkin-Elmer model 503 Atomic Absorption Spectrophotometer. From January 2002 to present, samples are analyzed for iron on a Perkin-Elmer model 4300 DV ICP.

LTER Keywords
cations
chemistry
ions
iron
sampling
Protocol Format
Parameter
Protocol ID
param_iron1
Protocol Type
field & laboratory

Samples for magnesium analysis (as well as dissolved nitrogen and phosphorus, silicon, calcium, sodium, potassium, iron, and manganese) are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 ml of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

Magnesium, as well as calcium, sodium, potassium, iron, and manganese are analyzed simultaneously on an optical inductively-coupled plasma emission spectrophotometer (ICP-OES). The acidified samples are directly aspirated into the instrument without a digestion. Magnesium is analyzed at 285.213 nm and at 280.271 nm and viewed axially for low-level analysis and radially for high level analysis.

The detection limit for magnesium is 0.03 ppm with an analytical range of the method extends to 50 ppm.

Method Log: Prior to January 2002, magnesium was determined on a Perkin-Elmer model 503 Atomic Absorption Spectrophotometer. Lanthanum at a 0.8% concentration was added as a matrix modifier to suppress chemical interferences. From January 2002 to present, samples are analyzed for magnesium on a Perkin-Elmer model 4300 DV ICP.

LTER Keywords
cations
chemistry
ions
magnesium
sampling
Protocol Format
Parameter
Protocol ID
param_magnesium1
Protocol Type
field & laboratory

Samples for manganese analysis (as well as dissolved nitrogen and phosphorus, silicon, calcium, sodium, potassium, magnesium, and iron) are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 ml of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

Manganese, as well as calcium, sodium, potassium, magnesium, and iron are analyzed simultaneously on an optical inductively-coupled plasma emission spectrophotometer (ICP-OES). The acidified samples are directly aspirated into the instrument without a digestion. Manganese is analyzed at 257.610 nm and at 259.372 nm and viewed axially for low-level analysis and radially for high level analysis.

The detection limit for manganese is 0.01 ppm with an analytical range of the method extends to 2 ppm.

Method Log: Prior to January 2002, manganese was determined on a Perkin-Elmer model 503 Atomic Absorption Spectrophotometer. From January 2002 to present, samples are analyzed for manganese on a Perkin-Elmer model 4300 DV ICP.

LTER Keywords
cations
chemistry
ions
manganese
sampling
Protocol Format
Parameter
Protocol ID
param_manganese1
Protocol Type
field & laboratory

Samples for nitrate/nitrite and ammonium are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into new, 20 ml HDPE plastic containers with conical caps. The samples are stored frozen until analysis, which should occur within 6 months. The samples are analyzed for nitrate/nitrite (and ammonium) simultaneously by automated colorimetric spectrophotometry, using a segmented flow autoanalyzer. Nitrate/nitrite is determined by utilizing the automated cadmium reduction method, as described in Standard Methods, where the absorption is monitored at 520 nm.

The detection limit for nitrate/nitrite is approximately 2 ppb and the analytical range for the method extends to 4000 ppb.

Method Log: Prior to January 2006 samples, nitrate/nitrite was determined on a Technicon segmented flow autoanalyzer. From 2006 to present, nitrate/nitrite is determined by an Astoria-Pacific Astoria II segmented flow autoanalyzer.

LTER Keywords
sampling
nitrite
nitrate
chemistry
Protocol Format
Parameter
Protocol ID
param_nitrate_nitrite1
Protocol Type
field & laboratory

Samples for organic and inorganic carbon are collected together with a peristaltic pump and tubing and in-line filtered, if necessary, (through a 0.40 micron polycarbonate filter) into glass 24 ml vials (that are compatible with the carbon analyzer autosampler), and capped with septa, leaving no head space. The samples are stored refrigerated at 4 degrees Celsius until analysis, which should occur within 2-3 weeks.

The detection limit for organic carbon is 0.30 ppm and the analytical range for the method is 30 ppm.

Method Log: Prior to May 2006 samples, organic carbon was analyzed by heated persulfate digestion on an OI Model 700 Carbon Analyzer. From May 2006 to present, Organic carbon is analyzed by combustion, on a Shimadzu TOC-V-csh Total Organic Carbon Analyzer.

LTER Keywords
carbon
dissolved nutrients
dissolved organic carbon
organic carbon
sampling
total organic carbon
Protocol Format
Parameter
Protocol ID
param_organic_carbon1
Protocol Type
field & laboratory

Samples for orthophosphate, nitrate/nitrite and ammonium are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into new, 20 ml HDPE plastic containers with conical caps. The samples are stored frozen until analysis, which should occur within 6 months of freezing. The samples are analyzed for orthophosphate (nitrate/nitrite, and ammonium simultaneously) by automated colorimetric spectrophotometry, using a segmented flow autoanalyzer. Orthophosphate is determined by forming a phosphoantimonylmolybdenum complex and the absorption is monitored at 880 nm.

The detection limit for orthophosphate is approximately 3 ppb and the analytical range for the method extends to 400 ppb.

Method Log: LTER began running orthophosphate during the summer of 2007 following the method described above. Orthophosphate is determined by an Astoria-Pacific Astoria II segmented flow autoanalyzer.

LTER Keywords
sampling
chemistry
Protocol Format
Parameter
Protocol ID
param_orthophosphate1
Protocol Type
field & laboratory

Samples for potassium analysis (as well as dissolved nitrogen and phosphorus, silicon, calcium, magnesium, sodium, iron, and manganese) are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 ml of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

Potassium, as well as calcium, magnesium, sodium, iron, and manganese are analyzed simultaneously on an optical inductively-coupled plasma emission spectrophotometer (ICP-OES). The acidified samples are directly aspirated into the instrument without a digestion. Potassium is analyzed at 766.490 nm and is viewed axially for low-level analysis and radially for high level analysis.

The detection limit for potassium is 0.06 ppm with an analytical range of the method extends to 10 ppm.

Method Log: Prior to January 2002, potassium was determined on a Perkin-Elmer model 503 Atomic Absorption Spectrophotometer. Lithium at an 1800-2000 ppm concentration was added as a matrix modifier to suppress ionization. From January 2002 to present, samples are analyzed for potassium on a Perkin-Elmer model 4300 DV ICP.

LTER Keywords
cations
chemistry
ions
potassium
sampling
Protocol Format
Parameter
Protocol ID
param_potassium1
Protocol Type
field & laboratory

Samples for sodium analysis (as well as dissolved nitrogen and phosphorus, silicon, calcium, magnesium, potassium, iron, and manganese) are collected together with a peristaltic pump and tubing and in-line filtered (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 ml of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

Sodium, as well as calcium, magnesium, potassium, iron, and manganese are analyzed simultaneously on an optical inductively-coupled plasma emission spectrophotometer (ICP-OES). The acidified samples are directly aspirated into the instrument without a digestion. Sodium is analyzed at 589.592 nm and is viewed axially for low-level analysis and radially for high level analysis.

The detection limit for sodium is 0.06 ppm with an analytical range of the method extends to 50 ppm.

Method Log: Prior to January 2002, sodium was determined on a Perkin-Elmer model 503 Atomic Absorption Spectrophotometer. Lithium at an 1800-2000 ppm concentration was added as a matrix modifier to suppress ionization. From January 2002 to present, samples are analyzed for sodium on a Perkin-Elmer model 4300 DV ICP.

LTER Keywords
cations
chemistry
ions
sampling
sodium
Protocol Format
Parameter
Protocol ID
param_sodium1
Protocol Type
field & laboratory

Samples for sulfate and chloride are collected together with a peristaltic pump and tubing and in-line filtered (through a 0.40 micron polycarbonate filter) into new, 20 ml HDPE plastic containers with conical caps. They are refrigerated at 4 degrees Celsius until analysis, which should occur within 6 months. The samples are analyzed for sulfate (and chloride) simultaneously by Ion Chromatography, using a hydroxide eluent.

The detection limit for sulfate is approximately 0.01 ppm and the analytical range for the method extends to 60 ppm.

Method Log: Prior to January 1998 samples, sulfate was determined on a Dionex DX10 Ion Chromatograph, using a chemical fiber suppressor. From 1998 to 2011, sulfate was determined by a Dionex model DX500, using an electro-chemical suppressor. From January 2011 until present, sulfate is determined by a Dionex model ICS2100, using an elctro-chemical suppressor.

LTER Keywords
sulfur
sulfate
sampling
chemistry
Protocol Format
Parameter
Protocol ID
param_sulfate1
Protocol Type
field & laboratory

Samples for total and dissolved nitrogen and phosphorus analysis (as well as dissolved silicon, calcium, magnesium, sodium, potassium, iron, and manganese) are collected together with a peristaltic pump and tubing and in-line filtered, when necessary, (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 mL of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

The samples must first be prepared for analysis by adding an NaOH–Persulfate digestion reagent and heated for an hour at 120 degrees C and 18-20 psi in an autoclave.
The samples are analyzed for total nitrogen and total phosphorus simultaneously by automated colorimetric spectrophotometry, using a segmented flow autoanalyzer. Total nitrogen is determined by utilizing the automated cadmium reduction method, as described in Standard Methods, where the absorption is monitored at 520 nm.

The detection limit for total and dissolved nitrogen is approximately 21 ppb and the analytical range for the method extends to 2500 ppb.

Method Log: Prior to January 2006 samples, total nitrogen was determined on a Technicon segmented flow autoanalyzer. From 2006 to present, total nitrogen is determined by an Astoria-Pacific Astoria II segmented flow autoanalyzer.

LTER Keywords
chemistry
nitrogen
nutrients
sampling
total nitrogen
Protocol Format
Parameter
Protocol ID
param_total_nitrogen1
Protocol Type
field & laboratory

Samples for total and dissolved phosphorus and nitrogen analysis (as well as dissolved silicon, calcium, magnesium, sodium, potassium, iron, and manganese) are collected together with a peristaltic pump and tubing and in-line filtered, when necessary, (through a 40 micron polycarbonate filter) into 120 ml LDPE bottles and acidified to a 1% HCl matrix by adding 1 mL of ultra pure concentrated HCl to 100 mls of sample. For every sample acidification event, three acid blanks are created by adding the same acid used on the samples to 100 mls of ultra pure water supplied from the lab. Once acidified, the samples are stable at room temperature until analysis, which should occur within one year. Until acidification, the samples should be refrigerated at 4 degrees Celsius.

The samples must first be prepared for analysis by adding an NaOH–Persulfate digestion reagent and heated for an hour at 120 degrees C and 18-20 psi in an autoclave.

The samples are analyzed for total nitrogen and total phosphorus simultaneously by automated colorimetric spectrophotometry, using a segmented flow autoanalyzer. Total phosphorus is determined by forming a phosphoantimonylmolybdenum complex and the absorption is monitored at 880 nm.

The detection limit for total phosphorus is approximately 3 ppb and the analytical range for the method extends to 800 ppb.

Method Log: Prior to January 2006 samples, total phosphorus was determined on a Technicon segmented flow autoanalyzer. From 2006 to present, total phosphorus is determined by an Astoria-Pacific Astoria II segmented flow autoanalyzer.

LTER Keywords
chemistry
nutrients
phosphorus
sampling
total phosphorus
Protocol Format
Parameter
Protocol ID
param_total_phosphorus1
Protocol Type
field & laboratory