Illinois State Water Survey
Health and Environmental Applications Laboratory

Standard Operating Procedure
For
Determination of Organic and Inorganic Carbon by High Temperature Combustion

(Based on SM 5310 B 2000, 21st ed)

SOP Number: AN.PSL.IN.C.3.3

Revision 3.3, 26 November 2024

Illinois State Water Survey
2204 Griffith Drive
Champaign, IL 61820-7495

NOTE THE HEALTH & SAFETY WARNINGS IN SECTION 4.0

Prepared by: Rachel Van Allen _____ Date: 12/18/2024
Rachel Van Allen, Analytical Chemist

Approved by: _____ Date: 12/18/2024
Evan Rea, HEAL Director

The following individuals (in addition to those listed on the cover) have reviewed this SOP.

Name (printed)	Signature	Date
Margarita Bargon	Margarita Bargon	12/19/2024

Beginning Revision	Ending Revision	Revision Date	Changes
2.1	3.0	1/19/24	Added information for inorganic carbon analysis.
3.0	3.2	7/26/24	Updated calibration ranges and LFB/LFM recipe. Added Appendix B for method parameters. Updated QC scheme.
3.2	3.3	11/26/24	Addressed comments and added method clarification, especially regarding inorganic carbon analysis.

Table of Contents

1.0 Scope and Application

1.1 This method is suitable for the determination of non-purgeable organically bound carbon (NPOC) in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, and reagent waters. In many surface and ground waters the POC/VOC contribution to TOC is negligible. Therefore, in practice, the NPOC determination is substituted for TOC.

1.2 This method may also be used to measure inorganic carbon (IC). A similar procedure is followed, with the key difference being the analysis of unacidified samples.

1.3 For NPOC, the Method Detection Limit (MDL) is 0.20 mg/L. There are two calibration ranges from 0.5 to 3.0 mg/L and from 3.0 to 50.0 mg/L. For IC, the MDL is 0.22 mg/L. There are two calibration ranges from 1.0 to 5.0 and from 5.0 to 100 mg/L. The range of analysis can be extended by dilution of the samples with matrix blank.

2.0 Summary of Method

2.1 For NPOC determination, organic carbon in a sample is converted to carbon dioxide (CO_2) by catalytic combustion. The CO_2 formed is measured directly by a nondispersive infrared detector. The amount of CO_2 is directly proportional to the concentration of carbonaceous material in the sample.

2.2 The instrument utilizes combustion at 680°C plus a platinum catalyst to oxidize organic material. The CO_2 produced is carried in the gas stream to an infrared detector that is specifically tuned to the absorptive wavelength of CO_2.

2.3 Sample preservation with acid eliminates the IC fraction of total carbon. Subsequent purging with gas also removes the purgeable/volatile organic carbon (POC or VOC) thus making the determination in this method NPOC. VOC is assumed to contribute a negligible amount of carbon to the samples routinely analyzed by the HEAL.

2.4 For IC analysis, the non-acidified sample reacts with an acid reagent in the instrument to convert all inorganic carbon to dissolved CO_2, which is then sparged and measured on the same infrared detector.

3.0 Definitions

3.1 Total organic carbon (TOC) – The sum of the purgeable organic carbon (POC) and the non-purgeable organic carbon (NPOC) as defined in 3.2 and 3.3. TOC is all carbon atoms covalently bonded in organic molecules.

3.2 Purgeable/Volatile organic carbon (POC/ VOC) – The organic carbon matter that is transferred to the gas phase when the sample is purged with air.

3.3 Non-purgeable/ non-volatile organic carbon (NPOC/ NVOC) – The organic carbon that remains after removal of the purgeable fraction from the sample.

3.4 Inorganic carbon (IC) – The carbonate, bicarbonate, and dissolved CO_2 in a sample. In most water samples, the IC fraction is many times greater than the TOC fraction.

3.5 Dissolved organic carbon (DOC) -- The fraction of TOC that passes through a 0.45 µm filter.

3.6 Dissolved inorganic carbon (DIC) -- The fraction of inorganic carbon that passes through a 0.45 µm filter.

HEAL Health and Environmental Applications Laboratory
QA Quality Assurance
QC Quality Control

4.0 Safety Warnings and Waste Management

4.1 Always wear eye protection in the laboratory.

4.2 Food, drinks, and smoking are not allowed in the laboratory.

4.3 Safety Data Sheets (SDS) applicable to this SOP can be found online by using the University of Illinois Division of Research Safety (DRS) website: https://www.drs.illinois.edu/Programs/SafetyDataSheets.

4.4 The Illinois State Water Survey Chemical Hygiene Plan covers the ISWS laboratory safety program, including, but not limited to, personal protective equipment used, control equipment inventory and operations (such as vented hoods), employee training programs, medical programs, and safety. The ISWS Chemical Hygiene Plan is available at https://go.illinois.edu/ISWS-Chemical-Hygiene-Plan.

4.5 The University of Illinois DRS has a laboratory safety plan available at https://www.drs.illinois.edu/site-documents/LaboratorySafetyGuide.pdf. The ISWS has their own laboratory safety manual, available at https://go.illinois.edu/ISWS-Laboratory-Safety-Manual.

4.6 The HEAL practices pollution prevention, which encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. The quantity of chemicals purchased should be based on the expected usage during its shelf life and disposal cost of unused material.

4.7 Laboratory waste management practices must be consistent with all applicable rules and regulations. Excess reagents and samples and method process wastes should be characterized and disposed of by DRS. It is the responsibility of the user of this method to comply with relevant disposal and waste regulations.

4.8 The HEAL has listed known health and safety warnings for this SOP, but this list should not be assumed to comprise all health and safety issues.

5.0 Cautions

The door of the instrument must be closed while in use.

6.0 Interferences

6.1 Removal of carbonate, bicarbonate, and dissolved CO_2 (inorganic carbon) by acidification and purging with purified gas results in the loss of volatile organic substances.

6.2 Large organic particles, or very large or complex organic molecules such as tannins, lignins, and humic acids require pretreatment (homogenization, filtration, dilution and sample stirring) to break up the particulate matter. A particulate kit (larger-diameter [0.8 mm] injection line) may also be used to accommodate suspended material.

6.3 Samples with high salt content have the effect of devitrifying the quartz combustion tube. Strategies used to prolong the life of the combustion tube and catalyst include dilution and decreased injection volume.

6.4 With any organic carbon measurement, contamination during sample handling and treatment can be a source of interference.

7.0 Personnel Qualifications

Analysts in training must complete at least five days of training or a manufacturer's training course, and a satisfactory demonstration of capability before analyzing routine samples.

8.0 Equipment and Supplies

8.1 Equipment

8.1.1 Total organic carbon analyzer – Shimadzu TOC-L combustion catalytic oxidation/ NDIR detector (model TOC-LCPH)

8.1.2 Computer with sufficient specifications to run Shimadzu's TOC-Control L software.

8.1.3 Autosampler – ASI-L

8.1.4 TOC vials and screw-on tops with septa (disposable): Pre-Cleaned 40 mL amber type-1 borosilicate glass vial (Environmental Sampling Supply part number 0040-0400-QC), or equivalent.

8.1.5 CO_2 absorber, Shimadzu part number 630-00999

8.1.6 Platinum catalyst, Shimadzu part number 638-60116

8.2 Chemicals and Solutions

8.2.1 Reagent water (DI): Deionized water, free of organic compounds. The ISWS uses 0.2 µm filters on all deionized water during the treatment process. In addition, the laboratory uses a separate polishing unit for final water purification. Reagent water should have TOC values less than twice the MDL.

8.2.2 Standard Solutions – Prepare calibration standards and quality control solutions by combining appropriate volumes of the stock solutions with 2.5 mL of concentrated H_3PO_4 in 500 mL volumetric flasks (0.5% H_3PO_4) and dilute to volume with DI water (see tables 8-1 and 8-2). Fresh standards should be prepared every 6 months, or as needed, with the realization that concentrations can change with age. Calibration standards not prepared

from primary standards must be initially verified using a certified reference solution. Store standards at 4°C. The amount of stock solution to use is calculated as follows:

$$\text{Amount to add} = \frac{\text{Total volume x target concentration}}{\text{stock concentration}}$$

8.2.3 Phosphoric acid (CASRN 7664-38-2), H_3PO_4, 85% (HPLC Grade): Used for sample and standard preservation and pH adjustment.

8.2.4 Air: compressed air, ultra-zero grade, hydrocarbon and CO_2 free.

8.2.5 Organic carbon (OC) stock standard, 1000 mg/L: A commercially prepared 1000 mg/L Ultra TOC stock standard is purchased, such as Ultra Scientific catalog # IQC-106TOC or equivalent.

NOTE: To make a 2000 mg/L OC stock standard in the laboratory, weigh 425 mg of reagent grade potassium hydrogen phthalate ($C_8H_5O_4K$, CASRN 877-24-7) dried at 104°C to a constant weight. Transfer quantitatively with reagent water to a 100 mL volumetric flask and bring to volume with DI. Preserve with H_3PO_4 to pH ≤ 2 and store in dark glass at 4°C.

8.2.6 Inorganic Carbon (IC) stock standard, 1000 mg C/L: weigh 0.874 g of $NaHCO_3$ and 1.10 g Na_2CO_3, then quantitatively transfer into a 250 mL volumetric flask and bring to volume with deionized water.

8.2.7 IC reagent – Prepare the IC reagent by diluting 50 mL of 85% phosphoric acid with DI water to a final volume of 250 mL. Pour the prepared IC reagent into the 250 mL container provided with the instrument. Secure the tubing and replace the cap on the container. Right-click on the instrument icon and select Maintenance – Regeneration of the IC solution. Click Start. Repeat the process until IC reagent level reaches the lower branch of the IC reagent drain tube.

8.2.8 Continuing Calibration Blank (CCB) – Prepare the calibration blank by adding 10 mL concentrated H_3PO_4 to a 1000 mL volumetric flask and diluting to volume with DI. This should be prepared at the same time as the mixed calibration standards. Store at 4°C.

8.2.9 Initial calibration verification (ICV) check standards –The ICV must be obtained from a source different from the standard stock solutions and prepared with the same acid concentration as the calibration standards. For the concentration of the analytes in the ICV solutions, see Table 8-1.

8.2.10 Continuing calibration verification (CCV) check standard –The CCV should contain the elements of interest at less than 50% of the high concentration calibration standard. Typical TOC standard concentrations used in the laboratory are listed in Table 8-1.

8.2.11 The laboratory fortified blank (LFB) is prepared by fortifying an aliquot of DI with the analytes of interest. The source of the analytes in the LFB must be from a source separate from that of the calibration standards. The LFB should be carried through the same preparation steps as the samples.

To prepare the LFB, add 0.3 mL of IC stock standard, 0.15 mL H_3PO_4, and 0.3 mL TOC

standard to 29.25 mL of DI water. The LFB true value is 10 mg/L for NPOC and IC.

To prepare the LFB for IC, add 0.3 mL of IC spike to 29.7 mL of DI water.

8.2.12 Laboratory Reagent Blank (LRB) – combine 29.55 mL DI water with 0.3 mL IC stock and 0.15 mL H_3PO_4. The LRB is used to assess efficiency of IC removal.
DI water is used for the LRB and CCB QC checks for the IC analyses. Fill LRB and CCB vials to the top with fresh DI water, with no headspace, to prevent the water from equilibrating with air to form bicarbonate.

8.2.13 Laboratory fortified matrix (LFM) – TOC spike - add 0.7 mL of 1000 µg/L TOC from KHP stock (Absolute Standards Inc. #59179, or equivalent) to acidified 34.3 mL sample (see section 11.1). LFM true value is 20 mg/L for NPOC and IC.

To prepare the IC spike - add 0.7 mL of 1000 mg/mL Inorganic Carbon stock purchased from LabChem, catalog # LC129007 to 34.3 mL of sample.

8.2.14 Method Detection Limit (MDL) NPOC verification sample – 1.0 mg/L, prepare from the same stock as the CCV. Analyze once per month.

8.2.15 Method Detection Limit (MDL) IC verification sample – 1.0 mg/L, prepare from the same stock as the CCV. Prepare a fresh solution for each analysis and fill the vial to the top with no headspace. Analyze once per month.

Table 8-1. Typical TOC concentrations in standards and quality control samples. All solutions are made in 0.5% H_3PO_4 (see 8.2.5).

Solution	TOC (mg/L)	Stock Concentration (mg/L)	mL of stock to add (to 500mL volumetric flask)
STD 1 (blank)	0.0	-	-
STD 2	0.5	1000	0.25
STD 3	1	1000	0.50
STD 4	1.5	1000	0.75
STD 5	2	1000	1.00
STD 6	3	1000	1.50
STD 7	5	1000	2.50
STD 8	10	1000	5.00
STD 9	25	1000	12.5
STD 10	50	1000	25.0
MDL verification	1	1000	0.50
CCV -Low	2	1000	1
CCV-High	20	1000	10.0
ICV-Low	2.5	1000	1.25
ICV-High	10	1000	5.0

Table 8-2. Typical IC concentrations in standards and quality control samples.

Solution	TOC (mg/L)	Stock Concentration (mg/L)	Volumetric flask size (mL)	mL of stock to add (to 250mL volumetric flask)
STD 0 (blank)	0.0	-	-	-
Stock STD 1	50	1000	250	12.5
Stock STD 2	100	1000	250	25.0
MDL verification	1	1000	50	0.05
CCV-Low	3	1000	500	1.5
CCV-High	40	1000	500	20.0
ICV-Low	4	1000	500	2
ICV-High	10	1000	500	5.0

9.0 Instrument Setup and Calibration

9.1 Prepare a series of 5 non-zero standards for NPOC, covering the desired range, a blank and an ICV as described in section 8. Label sample vials and fill about three-quarters with solution.

TOC-L prepares a series of 5 non-zero standards for each curve for IC by dilution from a standard solution (Low curve: 1, 1.5, 2, 3, 5 mg/L; High curve: 5, 10, 25, 50, 100 mg/L).

9.2 Turn the Shimadzu TOC-L ‘on’ by pressing the power button on the front of the machine. Turn on and log into the connected computer and start up the TOC-Control software. Click File, New, Sample Table to open a new NPOC or Inorganic Carbon sample table. Click ‘connect’ in the upper righthand corner to connect the TOC-L with the computer software. For NPOC analysis, select the option for “TOC-L 2”. For IC analysis, select “TOC-L IC”.

9.3 The TOC needs time to warm up, which can take about 1 hour. Click the ‘Monitor’ button to see the status of the various components of the machine, and to view the baseline. Once all the boxes turn from red (unready) to green (ready), the instrument will display a green ‘Ready’ button in the upper righthand corner.

9.3.1 Ensure the furnace temperature is set to 680°C and the gas turned on. Check the gas flow rate to the furnace, which should be around 150 cc/min.

9.4 While the machine is warming up, the rinse vessel, humidifier and DI vessel can be refilled with fresh DI water. Also, the LFM and LFB samples can be made (see section 8), and the sample table can be filled out.

9.4.1 To fill out the sample table for standards, go to File>New>calibration curve. This will insert one row that will have all calibration standards associated with it. Select the options: ‘normal’, NPOC or IC, remove zero-shift and select “Enable multiple injections”, and add a file name as the analysis date. For each calibration standard, the options for acid addition (none), injection volume (100 µL), and concentration (see table 8-1) will need to be set. Save the calibration curve, using the analysis date as the file name.

9.4.2 To insert the calibration curve into the sample table, select a row before the samples, and select insert calibration curve. The vial position can now be set for each calibration standard by selecting 'set vials>view vial settings'.

9.4.3 For samples: click on a line after the calibration curve and select 'add multiple samples'. Choose the method file being used and number of samples. Add extra lines in case of dilutions. Prepare a Preparation and Analytical batch in LIMs. Copy the exported Excel file with the sample numbers to the instrument software Sample name column.

9.5 Pour about 20 mL of calibration standard or QC sample into each sample vial (about half full). Place each standard, sample, and a quality control sample in the corresponding vial location in the autosampler- double check the sample table to ensure the proper location.

9.5.1 It is a good idea to stabilize the instrument before calibration by running 3 'wash' samples, which are the same as the calibration blank.

9.6 When all calibration standards, quality control solutions, and samples are added to the sample table, click 'Start', and select whether the machine should keep running once the queue is finished, or shut down.

9.7 Using the software, prepare a standard curve by plotting instrument response against concentration values. The r^2 value must be ≥0.995.

9.8 If a calibration curve is used over several days, it must be verified on each working day by the analysis of appropriate QC samples.

10.0 Sample Collection, Handling and Preservation

10.1 Samples should be collected in glass bottles with TFE-fluorocarbon (TFE) lined caps. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to ensure a representative sample, allow for replicate and spiked analysis, if required, and minimize waste disposal.

10.2 Sample containers should not be shaken or opened more than is necessary to create a sample aliquot for analysis. When pouring samples for carbon analyses, care should be taken not to aerate the sample. Additionally, the aliquot for carbon analysis should be capped immediately.

10.3 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between sample collection and the start of analysis should be kept to a minimum. Samples should be stored at 4°C and protected from sunlight and atmospheric oxygen.

10.4 When analysis cannot be performed immediately, the sample should be acidified to pH 2 with H_3PO_4. Acidification at the time of collection is especially desirable for unstable samples and may be used on all samples. Acid preservation results in the invalidation of inorganic carbon determination.

10.5 Holding time for preserved samples is 28 days from the time of collection.

10.6 Each set of glass sample bottles is checked for contamination after cleaning by measuring

UV absorbance at 254 nm. 40 mL TOC vials are purchased pre-cleaned and quality controlled by the vendor.

11.0 Sample Preparation and Analysis

11.1 To prepare samples for NPOC analysis, pour 35 mL of sample into a glass sample vial and add 0.175 mL of H_3PO_4,. Add a clean stir bar to TOC samples; samples for dissolved organic carbon analysis do not need a stir bar. For samples that are already acid-preserved in a larger bottle, simply pour about 30 mL of acidified sample into each vial. Label and cap each vial. For vials with 35 mL sample volume, just prior to analysis, poke a vent hole in the vial cap with a needle to prevent sample from bubbling out during sparging.

11.2 To prepare samples for DIC analysis, pour 35 mL of sample into a glass sample vial. Do NOT add any acid.

11.2 Follow the steps in section 9 for instrument setup, including filling out the sample table. Make note of vial locations and load the autosampler accordingly.

12.0 Troubleshooting

12.1 If issues with blanks are encountered, the autosampler rinse chamber should be emptied with a pipet and refilled with fresh DI water.

13.0 Data and Record Management

13.1 Report only those values that fall between the MDL and the highest calibration standard. Samples exceeding the highest standard should be diluted and reanalyzed.

13.2 Report results in mg C/L.

13.3 The ISWS laboratory precision and bias data for NPOC are shown in Appendix 1.

13.4 Make any notes in the TOC laboratory notebook and/or in electronic files, as necessary.

14.0 Quality Control and Quality Assurance

14.1 Initial Calibration Verification (ICV) – Two ICV samples for the low and high calibration ranges shall be analyzed after initial calibration. If the ICV check standard results do not meet the acceptance criteria of ±10% of the true value, the laboratory will reanalyze the ICV. If the determined concentrations are still not within limits, performance is unacceptable. The source of the problem should be identified and corrected.

14.2 Continuing Calibration Verification (CCV) – For all determinations the laboratory should analyze a low and a high CCV following calibration, after every tenth sample and at the end of the sample run. Analysis of the CCV solution must verify that the instrument is within ±10% of calibration. If the analysis of the CCV solution falls outside the limits, sample analysis should be discontinued, and the CCV rerun. If the CCV fails a second time, sample analysis will stop, and the cause will be determined. In the case of drift, the instrument will be recalibrated. All samples following the last acceptable CCV solution should be reanalyzed.

14.3 Continuing Calibration Blank (CCB) - For all determinations the laboratory should analyze a CCB (along with a CCV) following calibration and at the end of the sample run. Data produced are used to assess contamination from the instrument runs (carryover), standard storage, and laboratory environment. CCB results should be less than the MDL. If the CCB fails, cease analysis immediately and investigate the cause. Rerun the CCB, if that also fails, recalibrate and reanalyze all samples since the previous acceptable CCB/CCV.

14.4 Laboratory Reagent Blank (LRB) - A laboratory reagent blank shall be analyzed with each preparation batch. LRB data are used to assess contamination from the laboratory environment. LRB results should be less than the MDL. When the LRB exceeds the MDL, rerun the LRB. If the reanalyzed value is still too high, then corrective action should be taken and sample analysis discontinued.

14.5 Laboratory Fortified Blank (LFB) – The laboratory must analyze at least one LFB with each batch of prepared samples. If the recovery of any analyte falls outside the control limits of 85 to 115%, that analyte is judged out of control. The source of the problem should be identified, documented, and corrected. The LFB may be reanalyzed. If the reanalyzed LFB is still out of control, then the affected batch of samples shall be reanalyzed. An exception to this procedure would be for cases where the LFB result was biased high and the corresponding sample data yielded values less than the MDL. In these instances, the values may be reported as valid. If additional sample is not available for reanalysis, the data must be flagged with a caveat documenting the unacceptable accuracy.

14.6 Laboratory Fortified Sample Matrix (LFM) – The laboratory must add a known amount of analyte to at least one sample in a preparation batch. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The LFM recovery range is 70 to 130%. Recovery calculations are not required if the concentration added is less than 30% of the sample background concentration.

14.6.1 Over time, samples representing all routine sample sources and types should be fortified. Because of the wide variety of projects, choice of a specific sample for spiking is at the discretion of the analyst.

14.7 Duplicate (DUP) sample analyses will be performed once for every ten samples in a preparation batch, with a minimum of one per preparation batch, matrix type or preparation procedure. Precision for duplicate samples shall be deemed acceptable if the RPD is ≤20% when the analyte concentrations are at least ten times the MDL. If the precision is unacceptable, the samples, if available, associated with the batch shall be reanalyzed.

14.7.1 Over time, samples representing all routine sample sources and types should be analyzed in duplicate. Because of the wide variety of projects, choice of a specific sample for duplication is at the discretion of the analyst.

14.8 Check filters, when used, for DOC contribution by analyzing a filtered blank.

14.9 Check bottle blanks with each set of sample bottles to determine effectiveness or necessity of cleaning. Note that if pre-cleaned glass bottles and vials are used that come with a cleaning certificate, such testing is not necessary.

14.10 Check efficiency of inorganic carbon removal for each sample matrix by splitting a sample

into two portions and adding to one portion an inorganic carbon level similar to that of the sample. The TOC value for the spiked and unspiked samples should agree. If they do not, then adjust sample volume, pH, purge gas flow rate, and purge time to obtain complete removal of inorganic carbon.

NOTE: The LRB and LFB are spiked with IC solution to check removal efficiency of clean matrices. However, each time a new sample matrix is analyzed, an efficiency test should be performed.

14.11 Consecutive sample/standard injections must be reproducible to within ±10%.

14.12 Method Detection Limit (MDL) sample -- Analyze at least two MDL verification samples per quarter, made from separate batches.

Table 14-1. Quality control summary.

Type	Frequency	Target NPOC Value (mg/L)	Target IC Value (mg/L)	Criteria
ICV-low	At the start of each analytical batch	2.5	4	±10%
ICV-high	At the start of each analytical batch	10	10	±10%
CCV-low	Every 10 Samples	2	3	±10%
CCV-high	Every 10 Samples	20	40	±10%
CCB	At the start and end of each analytical batch	0	0	<MDL
LRB	One per prep batch	0	0	<MDL
LFB	One per prep batch	10	10	±15%
LFM	One per prep batch	20	20	±30%
DUP	One per 10 samples per prep batch			RPD ≤20% when analyte concentration is ≥10*MDL

14.12 Corrective Action

14.12.1 Further corrective actions for nonconforming quality control samples are given in the Corrective Action Policy (AD.HEAL.0.CorrectiveAction).

15.0 References

American Public Health Association, American Water Works Association, Water

Environment Federation. (2005). 5310 Total Organic Carbon and 5310B High Temperature Combustion Method (Approved by Standard Methods Committee, 2000). In A. Eaton, L. Clesceri, E. Rice, A. Greenberg & M. Franson (Eds.), *Standard Methods for the Examination of Water and Wastewater*, (21st ed.). Washington, DC: American Public Health Association.

Definition and Procedure for the Determination of the Method Detection Limit – Revision 1.11, In *Code of Federal Regulations 40*, Chapter 1, Part 136, Appendix B.

Rea, E. 2023. Health and Environmental Applications Laboratory Quality Management Plan. Illinois State Water Survey, Champaign, IL.

Appendix 1. ISWS Precision and Bias of NPOC

ANALYTE	KNOWN CONC. (mg/L)	NUMBER OF REPLICATES	MEAN RECOVERY mg/L	MEAN RECOVERY %	STANDARD DEVIATION mg/L
NPOC	5.00	20	5.21	104.3	0.127

Appendix 2. Instrument Method Parameters

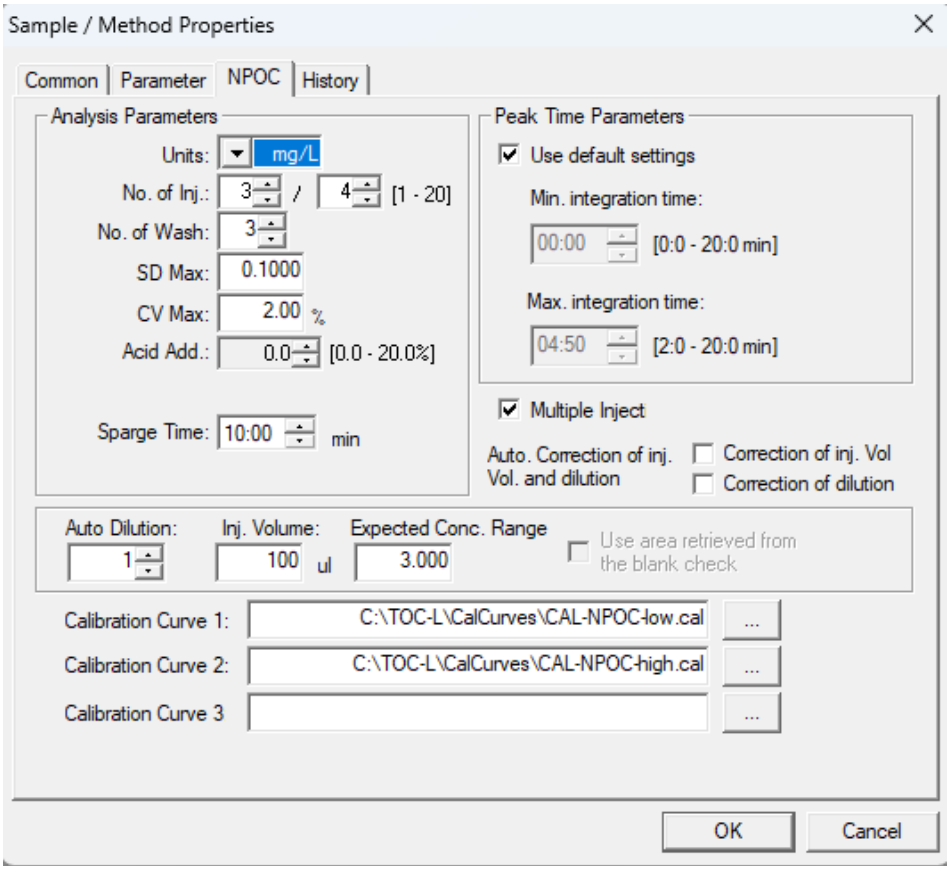


Figure 1. Method parameters for NPOC from method file ISWS-NPOC-081924.met.

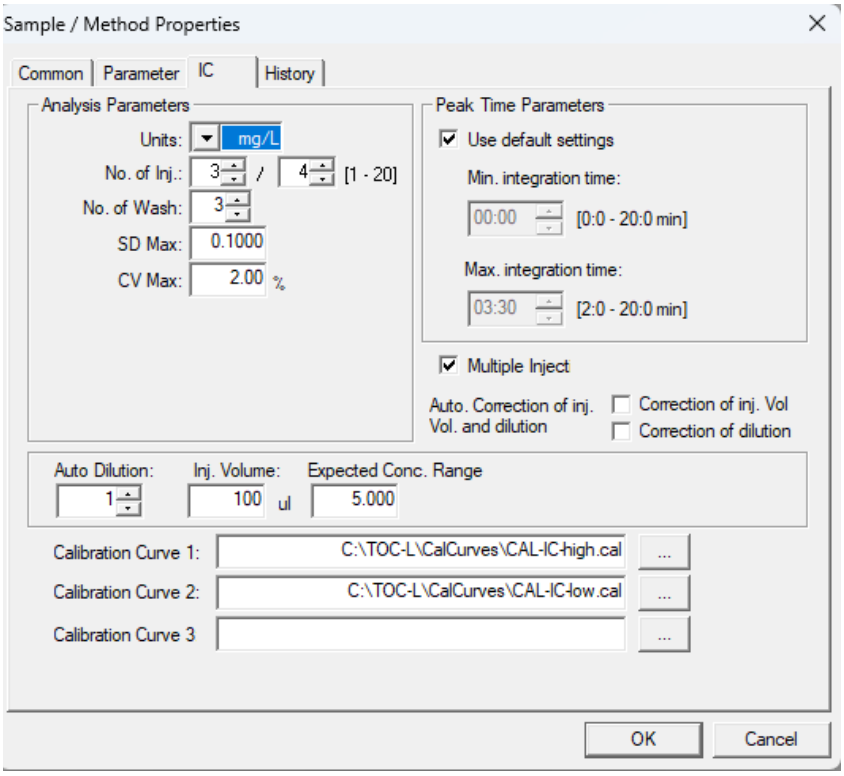


Figure 2. Method parameters for IC from method file ISWS-IC-081924.met

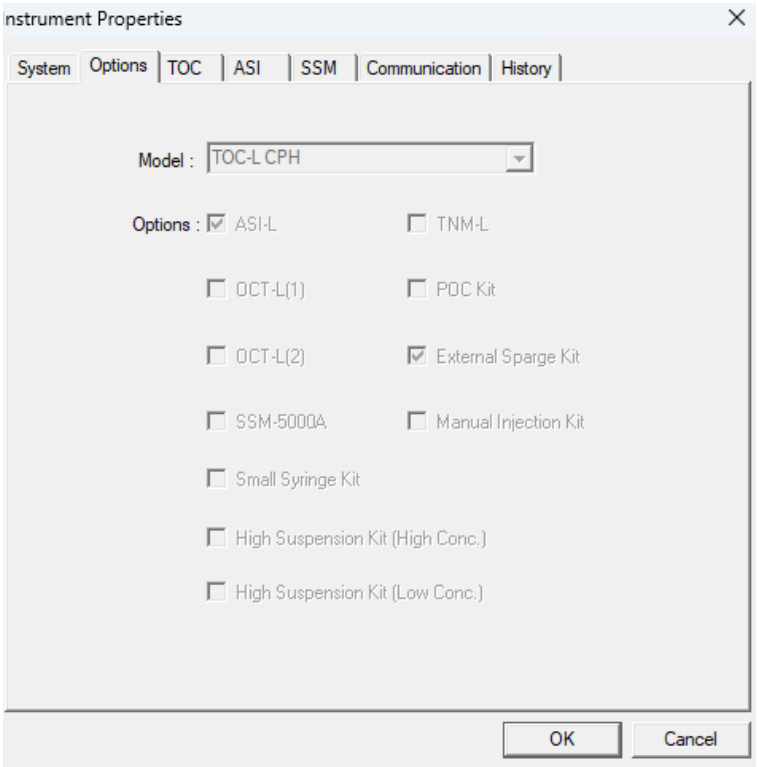


Figure 3. Instrument parameters, called H/W Settings in the TOC Control software, for the “TOC-L 2” configuration, Options tab. IC configuration has the External Sparge Kit option de-selected.

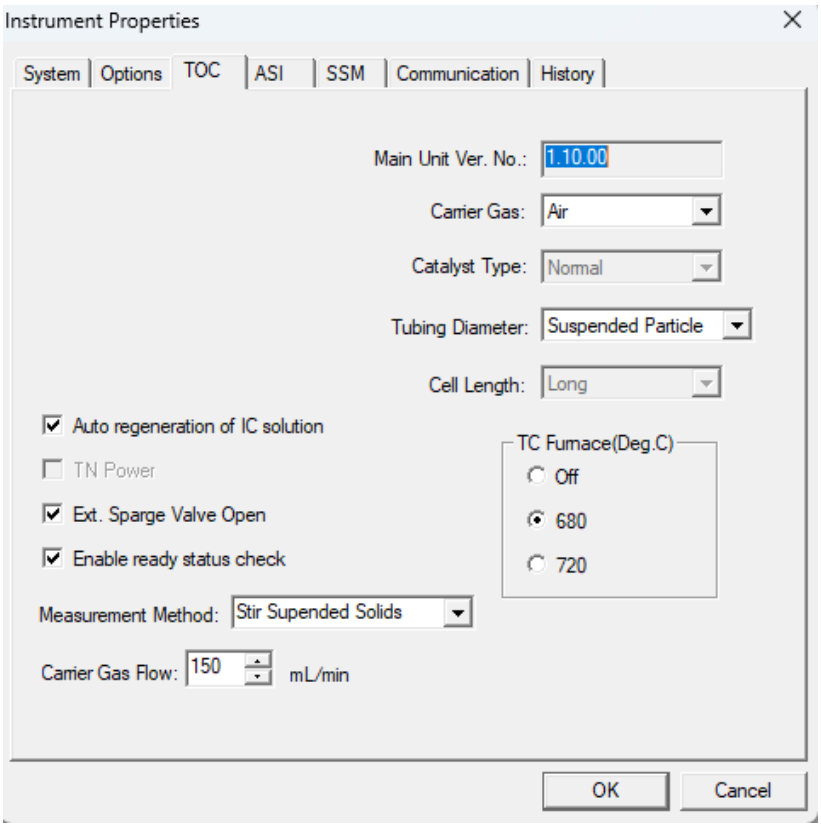


Figure 4. Instrument parameters, called H/W Settings in the TOC Control software, for the “TOC-L 2” configuration, TOC tab. IC configuration is the same.

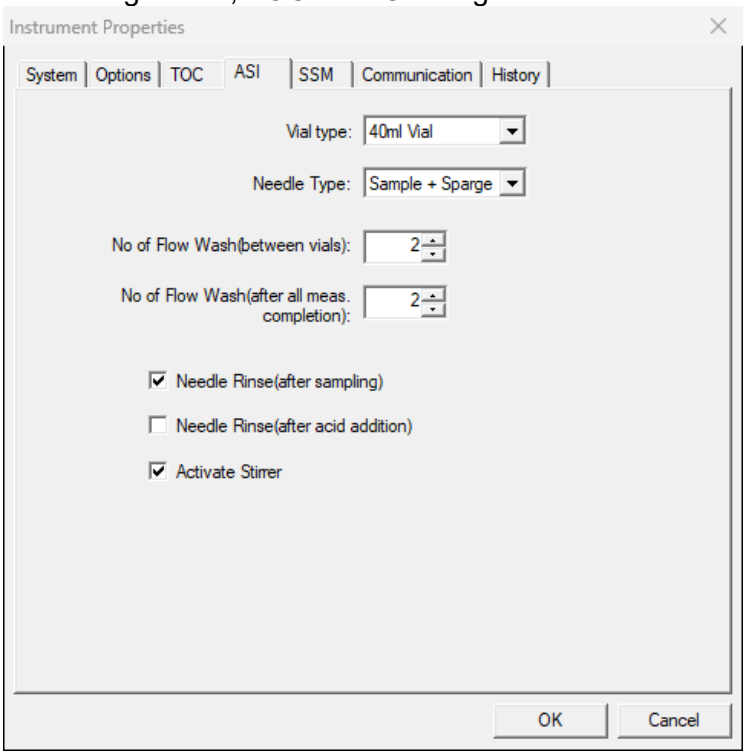


Figure 5. Instrument parameters, called H/W Settings in the TOC Control software, for the “TOC-L 2” configuration, ASI tab. IC configuration has the Needle Rinse (after acid addition)

option selected.

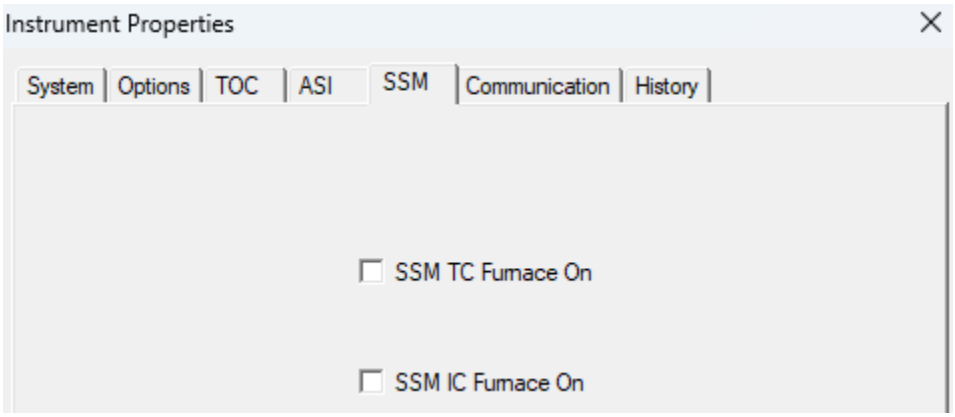
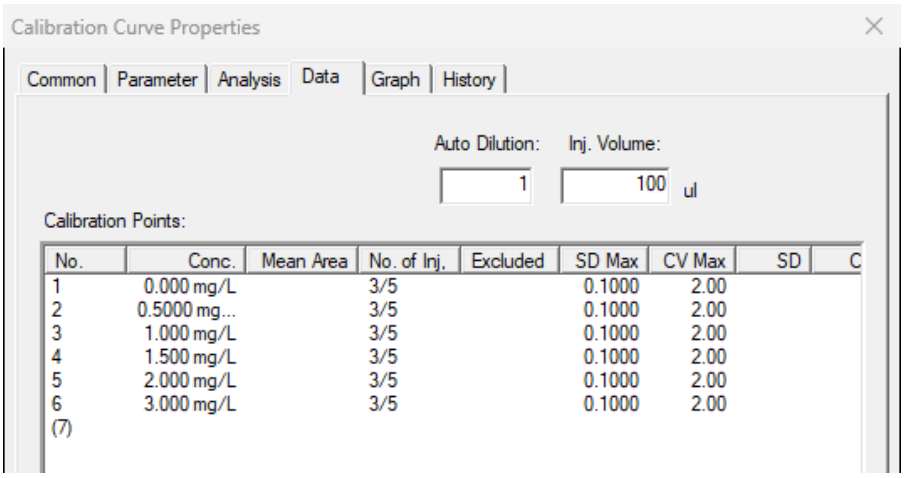
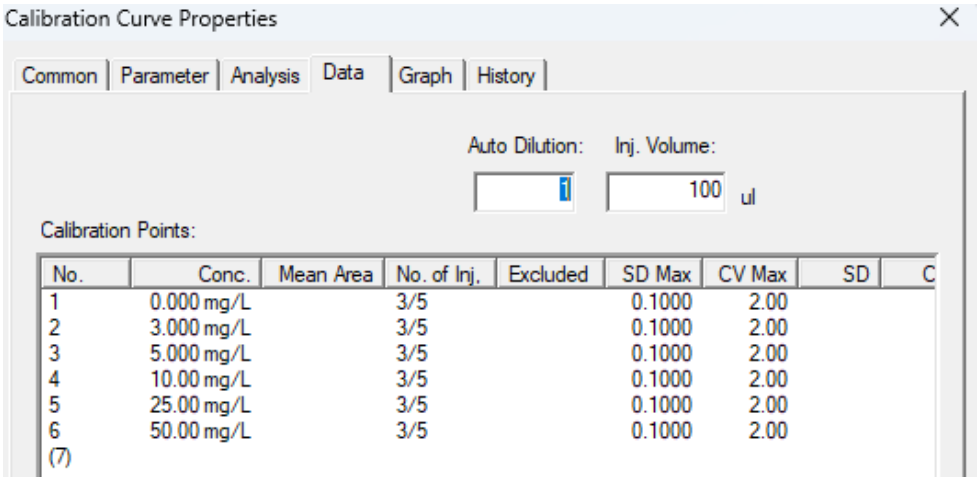


Figure 6. Instrument parameters, called H/W Settings in the TOC Control software, for the "TOC-L 2" configuration, SSM tab. IC configuration is the same.



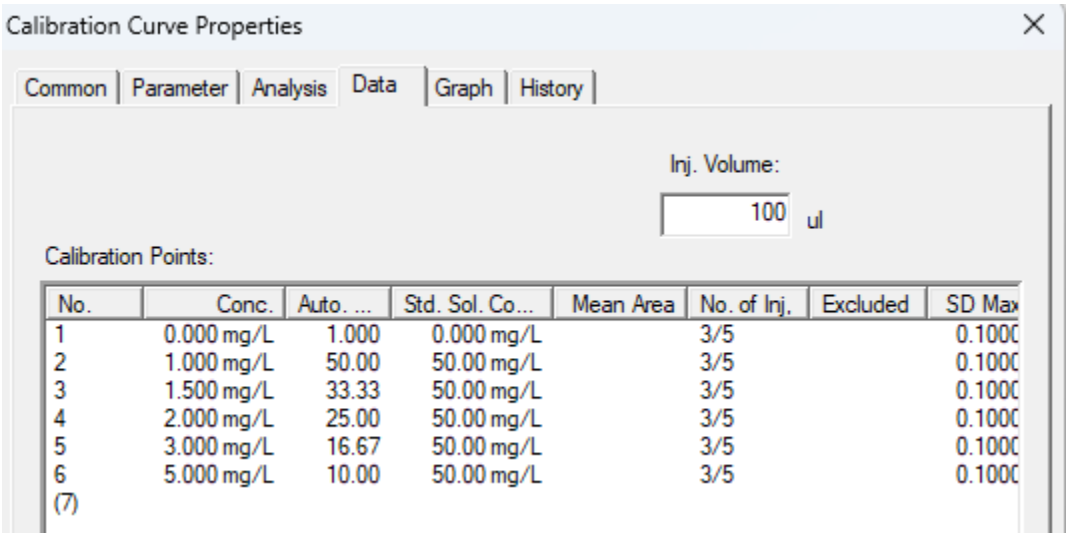
No.	Conc.	Mean Area	No. of Inj.	Excluded	SD Max	CV Max	SD	C
1	0.000 mg/L		3/5		0.1000	2.00		
2	0.5000 mg...		3/5		0.1000	2.00		
3	1.000 mg/L		3/5		0.1000	2.00		
4	1.500 mg/L		3/5		0.1000	2.00		
5	2.000 mg/L		3/5		0.1000	2.00		
6	3.000 mg/L		3/5		0.1000	2.00		
(7)								

Figure 7. Calibration curve properties for low-range NPOC calibration, found in file CAL-NPOC-low.cal.



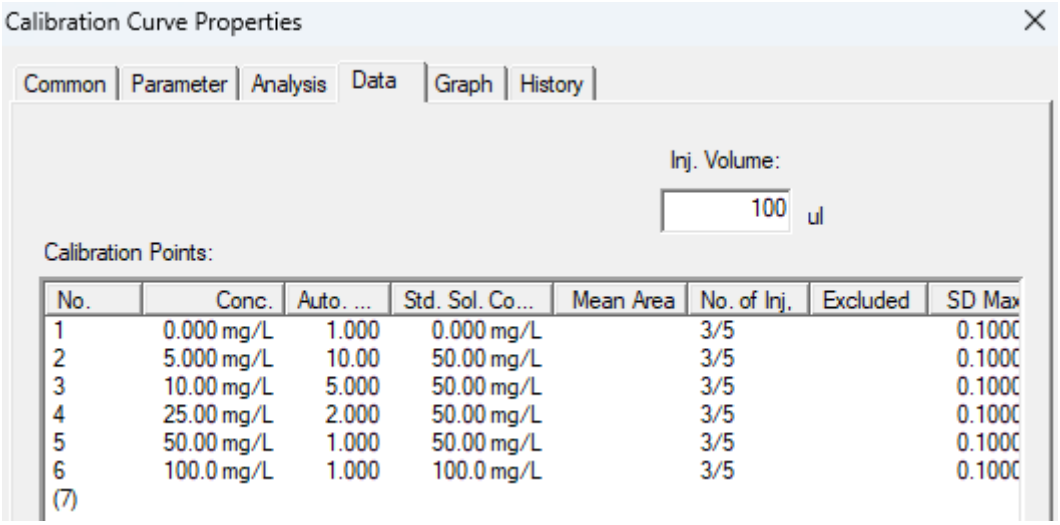
No.	Conc.	Mean Area	No. of Inj.	Excluded	SD Max	CV Max	SD	C
1	0.000 mg/L		3/5		0.1000	2.00		
2	3.000 mg/L		3/5		0.1000	2.00		
3	5.000 mg/L		3/5		0.1000	2.00		
4	10.00 mg/L		3/5		0.1000	2.00		
5	25.00 mg/L		3/5		0.1000	2.00		
6	50.00 mg/L		3/5		0.1000	2.00		
(7)								

Figure 8. Calibration curve properties for high-range NPOC calibration, found in file CAL-NPOC-high.cal.



No.	Conc.	Auto. ...	Std. Sol. Co...	Mean Area	No. of Inj.	Excluded	SD Max
1	0.000 mg/L	1.000	0.000 mg/L		3/5		0.1000
2	1.000 mg/L	50.00	50.00 mg/L		3/5		0.1000
3	1.500 mg/L	33.33	50.00 mg/L		3/5		0.1000
4	2.000 mg/L	25.00	50.00 mg/L		3/5		0.1000
5	3.000 mg/L	16.67	50.00 mg/L		3/5		0.1000
6	5.000 mg/L	10.00	50.00 mg/L		3/5		0.1000
(7)							

Figure 9. Calibration curve properties for low-range IC calibration, found in file CAL-IC-low.cal. CV Max, which is out of view, is set to 2.



No.	Conc.	Auto. ...	Std. Sol. Co...	Mean Area	No. of Inj.	Excluded	SD Max
1	0.000 mg/L	1.000	0.000 mg/L		3/5		0.1000
2	5.000 mg/L	10.00	50.00 mg/L		3/5		0.1000
3	10.00 mg/L	5.000	50.00 mg/L		3/5		0.1000
4	25.00 mg/L	2.000	50.00 mg/L		3/5		0.1000
5	50.00 mg/L	1.000	50.00 mg/L		3/5		0.1000
6	100.0 mg/L	1.000	100.0 mg/L		3/5		0.1000
(7)							

Figure 10. Calibration curve properties for high-range IC calibration, found in file CAL-IC-high.cal. CV Max, which is out of view, is set to 2.

AN.PSL.IN.TOC.3.3

Final Audit Report 2024-12-19

Created:	2024-12-17 (Central Standard Time)
By:	Evan Rea (erea@illinois.edu)
Status:	Signed
Transaction ID:	CBJCHBCAABAA3X2LlWqXu_MY6CtTvAe3M9uLjwT3eiDQ
Number of Documents:	1
Document page count:	18
Number of supporting files:	0
Supporting files page count:	0

"AN.PSL.IN.TOC.3.3" History

Document created by Evan Rea (erea@illinois.edu)
2024-12-17 - 5:54:42 PM CST- IP address: 130.126.105.85

Document emailed to Rachel Van Allen (vanallen@illinois.edu) for signature
2024-12-17 - 5:56:55 PM CST

Email viewed by Rachel Van Allen (vanallen@illinois.edu)
2024-12-18 - 9:29:30 AM CST- IP address: 130.126.105.137

Agreement viewed by Rachel Van Allen (vanallen@illinois.edu)
2024-12-18 - 9:31:41 AM CST- IP address: 130.126.105.137

Rachel Van Allen (vanallen@illinois.edu) authenticated with Adobe Acrobat Sign.
2024-12-18 - 9:36:13 AM CST

Document e-signed by Rachel Van Allen (vanallen@illinois.edu)
Signature Date: 2024-12-18 - 9:36:13 AM CST - Time Source: server- IP address: 130.126.105.137

Document emailed to Evan Rea (erea@illinois.edu) for signature
2024-12-18 - 9:36:15 AM CST

Email viewed by Evan Rea (erea@illinois.edu)
2024-12-18 - 9:36:48 AM CST- IP address: 130.126.105.85

Agreement viewed by Evan Rea (erea@illinois.edu)
2024-12-18 - 9:37:29 AM CST- IP address: 130.126.105.85

Evan Rea (erea@illinois.edu) authenticated with Adobe Acrobat Sign.
2024-12-18 - 9:37:33 AM CST

Document e-signed by Evan Rea (erea@illinois.edu)
Signature Date: 2024-12-18 - 9:37:33 AM CST - Time Source: server- IP address: 130.126.105.85

Document emailed to Margarita Bargon (rbargon@illinois.edu) for signature
2024-12-18 - 9:37:34 AM CST

Email viewed by Margarita Bargon (rbargon@illinois.edu)
2024-12-19 - 1:49:49 PM CST- IP address: 130.126.105.134

Agreement viewed by Margarita Bargon (rbargon@illinois.edu)
2024-12-19 - 1:50:02 PM CST- IP address: 130.126.105.134

Margarita Bargon (rbargon@illinois.edu) authenticated with Adobe Acrobat Sign.
2024-12-19 - 1:52:20 PM CST

Document e-signed by Margarita Bargon (rbargon@illinois.edu)
Signature Date: 2024-12-19 - 1:52:20 PM CST - Time Source: server- IP address: 130.126.105.134

Agreement completed.
2024-12-19 - 1:52:20 PM CST