LSASD Project ID: 20-0018

Sample and Analysis Plan

Characterization of Ambient PFAS in the Chattooga River Watershed

Project Location: Chattooga River Watershed (Georgia and Alabama)

Project Date(s): November 4th – 8th, 2019

Final SAP Approval Date: October 25, 2019

Project Leader: Greg White
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Athens, Georgia 30605-2720

The activities depicted in this Sampling and Analysis Plan (SAP) are accredited under the US EPA Region 4 Laboratory Services & Applied Science Division ISO/IEC 17025 accreditation issued by the ANSI-ASQ National Accreditation Board. Refer to certificate and scope of accreditation AT-1644.

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This Sample and Analysis Plan (SAP) is designed to be used in conjunction with the *Applied Science Branch Quality Assurance Project Plan* (USEPA, 2019*a*).

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SECTION A: Project Planning Elements		
A1. Distribution List		
Recipient	**Organization**	**Address/Email**
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A2. Project Personnel		
Team Members[1,2]	**Organization**	**Responsibilities**
Greg White	EPA/R4/LSASD	Project Leader
Nate Barlet	EPA/R4/LSASD	Safety Officer/Sampler
Jerry Ackerman	EPA/R4/LSASD	Discharge Task Lead/Sampler
Mel Parsons	EPA/R4/LSASD	Sampler
John Ruiz	EPA/R4/LSASD	Sampler
Bill Simpson	EPA/R4/LSASD	Sampler

[1] Project team members subject to change due to scheduling conflicts.

[2] Project Leader and all Task Leaders assisting with this project have been deemed competent by LSASD management, under ISO 17025 accreditation, to conduct the tasks required to fulfill the prescribed goals.

A3. Site Description and Background Information

The headwaters of the Chattooga River begin in Walker County north of LaFayette Georgia. The Chattooga River flows south across the Alabama-Georgia state line and feeds into Weiss Lake near Gaylesville Alabama. Per- and polyfluoroalkyl substances (PFAS), specifically perfluorooctanesulfonate (PFOS), were detected in the surface water at several sites along the Chattooga River during a 2018 study performed by U.S. EPA Region 4's Laboratory Services & Applied Science Division (LSASD) (USEPA, 2018). PFAS have also historically been detected downstream at public drinking water intakes in Centre Alabama and Gadsden Alabama (USEPA, 2019*b*).

PFAS are man-made chemicals that do not occur in nature and have been found to be persistent and accumulate in both the environment and the human body via exposure pathways such as consumption of contaminated food and drinking water. PFAS have been extensively used in industry, manufacturing of commercial products, and most notoriously as a component in aqueous film forming foams (AFFF) used for firefighting. There is evidence that suggests exposure to PFAS can lead to adverse health effects and are an emerging concern to public health. PFAS is a generic nomenclature encompassing a broader array of chemicals, with the most studied being perfluorooctanoic acid (PFOA) and perfluorooctanesulfonate (PFOS). The U.S. EPA has issued a Recommended Health Advisory for drinking water of 70 ng/L (ppt) for combined concentrations of PFOA and PFOS compounds. Extensive information regarding PFASs can be found at http://www.epa.gov/pfas.

A4. Problem Definition

The 2018 Phase 1 Study by LSASD observed positive detections of PFOS in surface water in the upper Chattooga River Watershed. PFOA was not detected in the 2018 study conducted by LSASD. The 2018 sampling of the Chattooga River by LSASD was conducted during an extreme high flow event thus dilution effects may have been a factor (USEPA, 2018). Major findings of the 2019 Phase 2 Study by LSASD with respect to the Chattooga River include: 1) the highest total PFAS concentrations of all streams sampled; 2) the highest diversity of distinct PFAS compounds detected; 3) a contribution of about 25% of total PFAS loading to Weiss Lake at a flowrate 7 times lower than the Coosa River during the study period (USEPA, 2019*b*). As guided by findings of the 2018 Chattooga River Study (Phase 1), the 2019 Weiss Lake Study (Phase 2), and directives of the R4 Water Division, this study will target near-base flow conditions as background concentrations of PFAS for the Chattooga River Watershed are largely unknown.

This study will observe background concentrations of PFAS in surface water and co-located sediment samples to characterize the relative distribution of PFAS in the Chattooga River Watershed along key segments determined by the Water Division. Ambient surface water concentrations of PFASs will be coupled with flow measurements to calculate instantaneous mass loading rates at near-base flow conditions. Sampling locations bracket off the following key segments of the Chattooga River based on the following municipalities in Georgia: Lyerly, Summerville, Trion, and LaFayette.

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A5. Project Description, Goals, and Study Boundaries

Study Goal:

Characterize the distribution and instantaneous mass loading of PFAS in the Chattooga River Watershed at near-base flow conditions along key segments determined by the R4 Water Division.

Study Objectives:

1. Collect surface water samples coupled with discharge measurements to compute instantaneous mass loading rates of PFAS along key segments of the Chattooga River Watershed.
2. Collect sediment samples collocated with surface water sample locations to determine the relative distribution and the potential for migration of PFAS contaminated sediments to the receiving waters of Weiss Lake.

Study Area:

The study area for this project includes the main stem of the Chattooga River and several inflowing tributaries. Proposed sampling locations range from the lower Chattooga River near Gaylesville Alabama where the river terminates into Weiss Lake, to the headwaters of the Chattooga River north of LaFayette Georgia (Appendix A: Site Maps). A total of 13 sites will be assessed which includes 8 stations on the main stem of the Chattooga River, 1 station on Town Creek which forms the headwaters of the Chattooga River, 3 tributary stations in watersheds with active biosolids application sites, and 1 station on Mill Creek, a significant tributary in Alabama. See Table 1 for a description of all sampling sites.

Study Design/Approach:

Standard Operating Procedures for all sampling and field measurement activities outlined in this study plan are referenced in Section B5: Sampling and Measurement Procedures.

In-Situ Water Quality

Surface water quality measurements of temperature, dissolved oxygen, specific conductance, turbidity, and pH will be collected *in-situ* via multi-parameter data sondes at each site. See Table 2 for a detailed list of *in-situ* water quality parameters and measurement uncertainties. All multi-parameter data sondes will be maintained and calibrated in accordance with LSASD Standard Operating Procedure for Equipment Inventory and Management (SESDPROC-1009-R0) and those selected in Section B.5. All equipment calibrations will be verified in accordance with LSASD Calibration and End-Check Acceptance Criteria (SESDFORM-060-R0).

PFAS Loading Rates (Surface Water Sampling and Discharge Measurements)

Surface water samples will be collected at each site and transported to the EPA R4 Laboratory at LSASD in Athens Georgia to be analyzed for the 23 PFAS analytes listed in Table 4. PFAS sample collection, preservation, and holding times are listed in Table 3. Surface water sample collection methods will vary based on site conditions (e.g. direct fill method where possible or grab method via stainless-steel bucket or

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scoop). A corresponding discharge will be either directly measured via handheld or remotely-operated flowmeters or retrieved from USGS gaging stations for each sampling location to compute an instantaneous mass loading rate of detected PFAS compounds. This study will target near-base flow conditions. Approximate base flow conditions will be defined as a discharge below the monthly mean for November as recorded by historical streamflow data collected at the USGS gage located on the lower Chattooga River (USGS 02398300) over a 30-year period. The threshold value is 450 ft^3/s. A discharge above this threshold will be considered non-base flow conditions and the sampling event will be postponed until water levels recede to appropriate levels. Best professional judgement will be used in consultation with the R4 Water Division to determine how to proceed if these environmental conditions are not met.

Sediment Sampling

Sediment samples will be collected at each site and transported to the EPA R4 Laboratory at LSASD in Athens Georgia to be analyzed for the 23 PFAS analytes listed in Table 4. PFAS sample collection, preservation, and holding times are listed in Table 3. Sediment sample collection methods will vary based on site conditions (e.g. grab method via stainless-steel scoop where possible or an alternate grab method via stainless-steel petite Ponar). Each sediment sample will consist of a composite of 3 sediment aliquots collected across a transect perpendicular to the stream flow to account for streambed heterogeneity.

Quality Control Samples

Multiple control samples will be collected in accordance with LSASD Standard Operating Procedures and accepted trace-level contaminant sampling practices. Control samples will include trip blanks, field blanks, field equipment rinse blanks, field duplicate samples, and matrix spike/matrix spike duplicate (MS/MSD) field samples. Surface water and sediment samples collected for PFAS analysis will be sampled via a trace level sampling technique to avoid cross-contamination of PFAS samples due to sample collection and handling. This process will require two field personnel for PFAS sample collection. A designated sampler will handle the sample media and sample container only. A second designee will operate sampling equipment and assist with sample container packaging and labeling. An outline of all quality control samples is listed in Section B3: Quality Control.

Project Timeline:

All field activities for this study are planned for the week of November 4th, 2019. Laboratory turn-around time is 35 days from the time samples are received. The draft final report for this study is expected to be provided to the Water Division on January 22th, 2020.

A6. Applicable Regulatory Information

The U.S. EPA has established a life-time exposure recommended health advisory level for drinking water of 70 parts per trillion for PFOA and PFOS individually or combined. There are currently no Maximum Contaminant Levels (MCLs) or enforceable standards for PFOA, PFOS, or other PFAS related compounds in any media (e.g. drinking water, surface water, soils and sediments) set by the U.S. EPA or the states of Georgia and Alabama.

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A7. Decision(s) to be made based on data

This study will provide relative mass loadings of PFAS compounds in surface water along key segments of the Chattooga River Watershed and insight into the relative distribution and the potential for migration of PFAS compounds in sediments to the receiving waters of Weiss Lake; as well as provide a comparison of PFAS concentrations and compositions in sediment and surface water along the Chattooga River Watershed. All further decisions, recommendations, and/or actions will be made at the discretion of the U.S. EPA's R4 Water Division.

SECTION B: Data Generation, Acquisition, and Reporting

Will samples or physical evidence be collected:	☒ **Yes –** *If yes, complete all subsections in Section B.*
	☐ **No** – *If no, no action needed for B1, B2, B3 or B4, proceed to B5.*

B1. Sampling Design/Information Inputs

Sample Media	Total Number of Samples	Analyses
Surface Water	13 samples + duplicate + 4 QC + MS/MSD	PFAS (See Tables 3 & 4)
Sediment	13 samples + duplicate + MS/MSD	PFAS (See Tables 3 & 4)

B2. Sampling Handling and Custody

As outlined in the *Applied Science Branch Quality Assurance Project Plan* (USEPA, 2019*a*), all samples will be handled and custody maintained in accordance with the LSASD Laboratory Services Branch Laboratory Operations and Quality Assurance Manual, LSASD Operating Procedure for Sample and Evidence Management, SESDPROC-005, and LSASD Operating Procedure for Packing, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209.

Will a Chain-of-Custody be produced?	☒ **Yes**
	☐ **No**

During the duration of the event, have preparations been made to ensure that custody is maintained? *Custody of a sample or physical evidence is defined as:* • *It is in the actual possession of an investigator* • *It is in the view of an investigator, after being in their physical possession* • *It was in the physical possession of an investigator and then they secured it to prevent tampering* • *It is placed in a designated secure area*	☒ **Yes** ☐ **No**

B3. Quality Control

Field quality control measures will be performed in accordance with the LSASD Operating Procedure for Field Sampling Quality Control, SESDPROC-011.

Field quality control (QC) samples include the following:

- Each batch of samples will contain a duplicate quality control sample for each analysis. The duplicate samples will be collected at CHR01.
- Each batch of surface water and sediment samples being analyzed for PFAS will also contain an additional sample volume for matrix spike/matrix spike duplicates (MS/MSD). MS/MSD volumes will be collected at TOC01 at the headwaters of the Chattooga River to characterize background PFAS concentrations in surface water and sediment.
- Temperature blanks will be placed in all sample coolers.

The following additional quality control (QC) samples will be collected and analyzed for PFAS contamination:

- A field blank will be collected by the sampling team at the onset and completion of field activities.
- Trip blank(s) will be stored and transported with collected samples through the duration of the study.
- If a field equipment decon is needed, a separate field equipment rinse blank will be collected for PFAS sediment sampling equipment (e.g. stainless-steel petite Ponar sediment grabs, spoons, scoops, and bowls), and PFAS surface water sampling equipment (e.g. buckets and/or scoops).
- All blank quality control (QC) samples will be prepared utilizing PFAS-free water supplied by the U.S. EPA LSASD laboratory in Athens, GA.

PFAS sampling protocol:

- A two-person trace-level sampling protocol will be used for all PFAS sample collection. One member of the sampling team will handle the sample media and sample container only. A second team member will be designated to handle sampling equipment and assist with sample packaging and labeling.
- All sampling equipment will be cleaned using Luminox® and warm tap-water, then rinsed in PFAS-free water before being air-dried and sealed in clean plastic sheets in preparation for field activities.
- Sampling equipment known to contain PFAS will be avoided during sampling activities.

Laboratory quality control measures are specified in the *LSASD Laboratory Services Branch Laboratory Operations and Quality Assurance Manual* (USEPA, 2019*c*).

B4. Analytical Methods and Support

Samples will be analyzed by the EPA/LSASD laboratory in Athens, GA in accordance with the LSASD Laboratory Services Laboratory Operations and Quality Assurance Manual (USEPA, 2019*c*). Specific analytical methods are listed in Table 4.

Samples submitted to a Contract Laboratory Program (CLP) laboratory will be analyzed in accordance to the current statement of work.

Laboratory Turn-Around-Time Requested: 35 Days

Reporting Levels:	☒ Non-Routine Reporting Levels **ARE NOT** Required, No Further Action. ☐ Non-Routine Reporting Levels **ARE** Required, List Below.
Non-Routine Reporting Levels:	*n/a*

Waste Samples Anticipated:	☐ **Yes** ☒ **No** ☐ **Unknown**

If answer is yes, specify laboratory to receive samples: *n/a*

B5. Sampling and Measurement Procedures

Sampling and measurement activities will be in accordance with the LSASD operating procedures. The following field procedures will be followed during this study, check all that apply. The most recent version of LSASD operating procedures can be found at https://www.epa.gov/quality/quality-system-and-technical-procedures-sesd-field-branches *(Last Update: 4/05/18)*

	Field Measurement Procedures*	**SESDPROC-**	**Revision**
☒	Field pH Measurement	100	R4
☒	Field Specific Conductance Measurement	101	R6
☒	Field Temperature Measurement	102	R5
☒	Field Turbidity Measurement	103	R4
☐	Groundwater Level and Well Depth Measurement	105	R3
☒	Field Measurement of Dissolved Oxygen	106	R4
☐	Field X-Ray Fluorescence (XRF) Measurement	107	R4
☐	Wastewater Flow Measurement	109	R4
☒	Global Positioning System	110	R4
☒	In-Situ Water Quality Monitoring	111	R4
☐	Field Measurement of Total Residual Chlorine	112	R5
☐	Field Measurement of Oxidation-Reduction Potential (ORP)	113	R2
	Field Sampling Procedures*	**SESDPROC-**	**Revision**
☒	Sediment Sampling	200	R3
☒	Surface Water Sampling	201	R4
☐	Soil Sampling	300	R3
☐	Groundwater Sampling	301	R4
☐	Waste Sampling	302	R3
☐	Ambient Air Sampling	303	R5
☐	Potable Water Supply Sampling	305	R3
☐	Wastewater Sampling	306	R4
☐	Soil Gas Sampling	307	R3
	Ecology Section Field Sampling Procedures*	**SESDPROC-**	**Revision**
☒	Hydrological Studies	501	R4
☐	Water Column Oxygen Metabolism	504	R4
☐	Reaeration Measurement by Diffusion Dome	505	R4
☐	Sediment Oxygen Demand	507	R4
☐	Multi-Habitat Macroinvertebrate Sampling in Wadeable Freshwater Streams	508	R4
☐	Marine Macroinvertebrate Field Sampling	511	R4
☐	Fish Field Sampling	512	R4
☐	Pore Water Sampling	513	R3
☐	Dye Tracer Measurements	514	R2
☐	Bottom Water Sampling for Sulfide	515	R0

***If procedures allow for different sampling and measurement methods, the utilized method(s) must be identified in the project description section. Additionally, verify procedure revision numbers before issuance of SAP.**

Section C: Reporting	
C1. Reporting	
Estimated Report Completion Date: 01/22/2020	
Is a Provisional Data Release Anticipated?	☒ **Yes**
	☐ **No**
Provisional data refers to final analytical and field measurement results that may be subject to further interpretation and/or data assessment by the project leader prior to the issuance of a final field investigation report. Provisional data may be provided prior to the completion of the LSASD final report only if LSASD management approves the release of the information and the analytical data have been released as final from the LSASD Laboratory Services Branch, for LSASD generated data, and/or the LSASD Quality Assurance Section, for non-LSASD generated data. Release of provisional data will be transmitted by electronic or hard copy with official correspondence from the Section Chief in accordance with the LSASD Operating Procedure for Report Preparation and Distribution (SESDPROC-003).	
Additional Comments: *Provisional data may be released to EPA R4 Water Division pending issuance of final report for the purpose of planning regional priorities related to PFAS.*	

References

SESDFORM-060-R0 (2018). SESD Calibration and End-Check Acceptance Criteria. U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.

SESDPROC-1009-R0 (2017). Standard Operating Procedure for Equipment Inventory and Management. U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.

USEPA (2018). Phase 1: Study of PFAS Compounds on the Chattooga River (Project ID 18-0142). U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.

USEPA (2019*a*). Applied Science Branch Quality Assurance Project Plan. U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.

USEPA (2019*b*). Phase 2: Prioritization of PFAS Contributions to Weiss Lake (Project ID 19-0253). U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.

USEPA (2019*c*). Laboratory Services Branch Laboratory Operations and Quality Assurance Manual. U.S. Environmental Protection Agency, Region 4, Laboratory Services & Applied Science Division, Athens, GA.

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Table 1: **Sampling Site Locations and Descriptions**

Station ID	Water Body	Approximate Coordinates (DD.ddddd)		Site Description
		Latitude	Longitude	
CHR01	Chattooga River	34.26362	-85.56017	Chattooga River at Hwy 35 in Gaylesville AL
MIC01	Mill Creek	34.29581	-85.50949	Mill Creek at Hwy 68 near Gaylesville AL
CHR02	Chattooga River	34.33585	-85.44564	Chattooga River at Rte 323 in Chattoogaville GA
HIC01	Hinton Creek	34.33456	-85.43668	Hinton Creek at Rte 323 in Chattoogaville GA
CHR03	Chattooga River	34.40220	-85.39595	Chattooga River at Lyerly Dam Rd in Lyerly GA
CHR04	Chattooga River	34.44476	-85.36263	Chattooga River at Hwy 100 near Summerville GA
RAC01	Raccoon Creek	Redacted	Redacted	Raccoon Creek upstream of Summerville public drinking water intake facility GA
CHR05	Chattooga River	34.51955	-85.30120	Chattooga River at Penn Bridge Rd near Trion GA
CHR06	Chattooga River	34.54532	-85.31792	Chattooga River upstream of low-head dam near Trion GA
TEC01	Teloga Creek	34.54353	-85.38531	Teloga Creek at Hwy 327 in Broomtown Valley GA
CHR07	Chattooga River	34.66671	-85.30005	Chattooga River at Foster Mill Dr near LaFayette GA
CHR08	Chattooga River	34.70723	-85.28696	Chattooga River near Culberson Ave in LaFayette GA
TOC01	Town Creek	34.71414	-85.26769	Town Creek at Round Pond Rd near LaFayette GA

Table 2: ***In-Situ*** **Water Quality Parameters**

In-Situ **Water Quality Parameter Measurement Uncertainty**			
Parameter	**Units**	**Measurement Technology**	**Measurement Uncertainty**
pH	SU	Glass electrode	± 0.2 SU
Dissolved Oxygen	mg/L	Luminescent DO probe	± 0.2 mg/L
Temperature	°C	LDO Thermistor	± 0.2 °C
Specific Conductance	µS/cm	Nickel electrode cell	± 0.5% of reading
Turbidity	FNU	Optical Probe	± 5% of reading

Table 3: **Sample Collection, Preservation and Holding Times**

Analyses	Media	Container	Preservation	Holding Time
PFAS	Surface Water	2 x 15mL Polypropylene Vial	Ice (≤ 4°C)	42 days
	Sediment	50mL Polypropylene	Ice (≤ 4°C)	42 days

Table 4: PFAS Target Analyte List

Region IV Laboratory Per - and Polyfluoroalkyl Substances (PFAS) Target Analyte List Method Detection Limits (MDLs) & Minimum Reporting Limits (MRLs)				
Analyte[1]	**Water[2] µg/L (ppb)**		**Soil/Sediment[3] µg/kg (ppb)**	
	MDL	**MRL**	**MDL**	**MRL**
Perfluorotridecanoic acid (PFTrDA)	0.039	0.040	0.040	0.100
Perfluorododecanoic acid (PFDoA)	0.029	0.040	0.040	0.100
Perfluoroundecanoic acid (PFUDA)	0.021	0.040	0.040	0.100
Perfluorodecanoic acid (PFDA)	0.096	0.160	0.040	0.100
Perfluorononanoic acid (PFNA)	0.016	0.040	0.040	0.100
Perfluorooctanoic acid (PFOA)	0.026	0.040	0.040	0.100
Perfluoroheptanoic acid (PFHpA)	0.014	0.040	0.040	0.100
Perfluorohexanoic acid (PFHxA)	0.031	0.040	0.040	0.100
Perfluoropentanoic acid (PFPeA)	0.018	0.040	0.040	0.100
Perfluorobutyric acid (PFBA)	0.022	0.040	0.040	0.100
Perfluorodecanesulfonate (PFDS)	0.032	0.039	0.040	0.096
Perfluorononanesulfonate (PFNS)	0.015	0.038	0.040	0.096
Perfluorooctanesulfonate (PFOS)	0.017	0.037	0.040	0.092
Perfluoroheptanesulfonate (PFHpS)	0.017	0.038	0.040	0.095
Perfluorohexanesulfonate (PFHxS)	0.017	0.036	0.040	0.091
Perfluoropentanesulfonate (PFPeS)	0.013	0.038	0.040	0.094
Perfluorobutanesulfonate (PFBS)	0.023	0.035	0.040	0.088
Perfluorooctanesulfonamide (FOSA)	0.031	0.040	0.040	0.100
Fluorotelomer sulfonate 8:02 (8:2 FTS)	0.034	0.038	0.040	0.096
Fluorotelomer sulfonate 6:02 (6:2 FTS)	0.029	0.038	0.040	0.095
Fluorotelomer sulfonate 4:02 (4:2 FTS)	0.021	0.037	0.040	0.094
N-(Heptadecafluorooctylsulfonyl)-N-methylglycine (N-MeFOSAA)	0.110	0.160	0.040	0.100
Hexafluoropropylene oxide–dimer acid (HFPO-DA)	0.026	0.040	0.040	0.100

[1]PFAS analytes for both surface water and sediment/soil matrices are analyzed via the method outlined in LSBPROC-800-R1.

[2]PFAS analytes in surface water are analyzed using ASTM standard D7979-17.

[3]PFAS analytes in solids (e.g. soil, sediment, and waste) are analyzed using ASTM standard D7968-17a.

Appendix A: Site Maps

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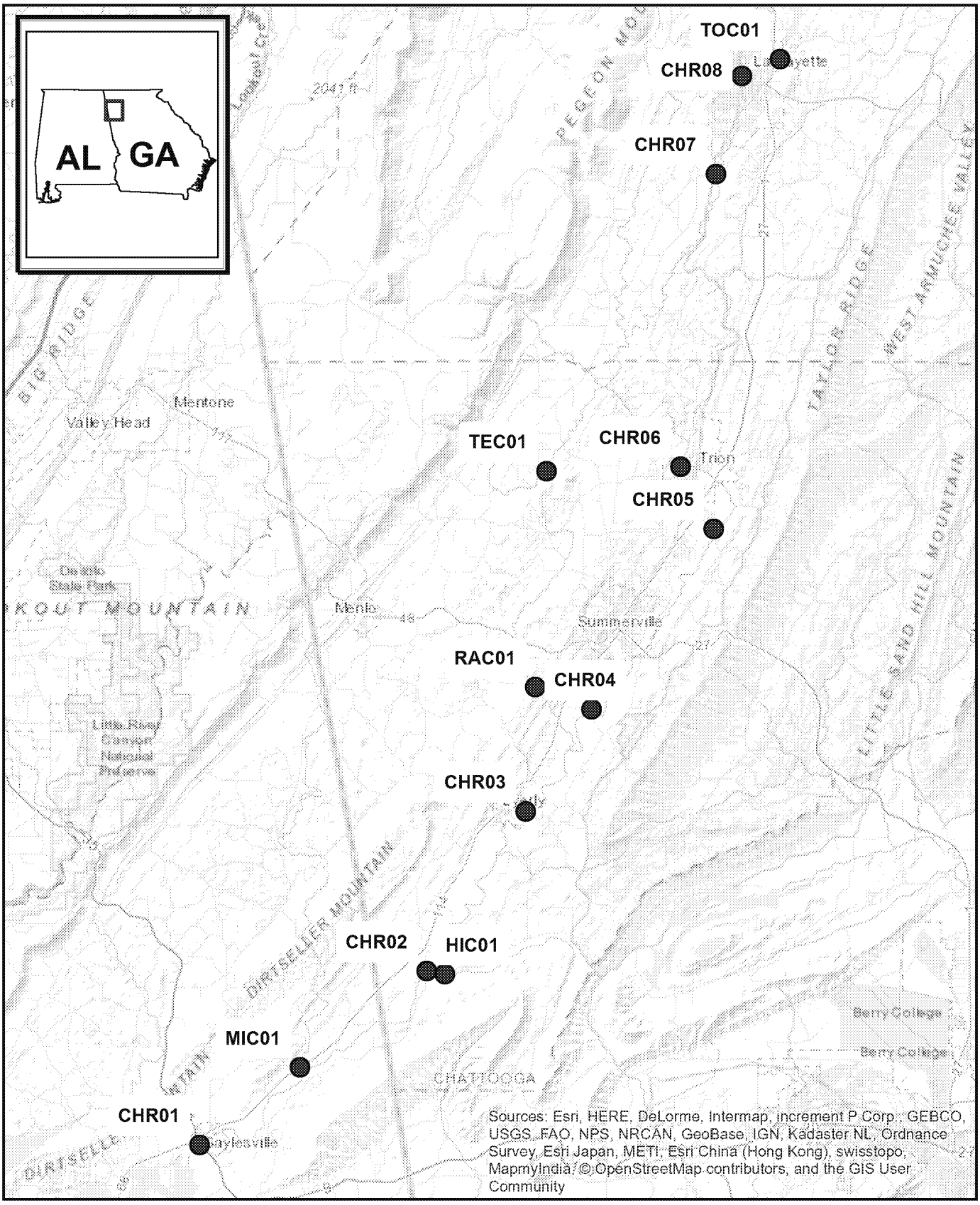
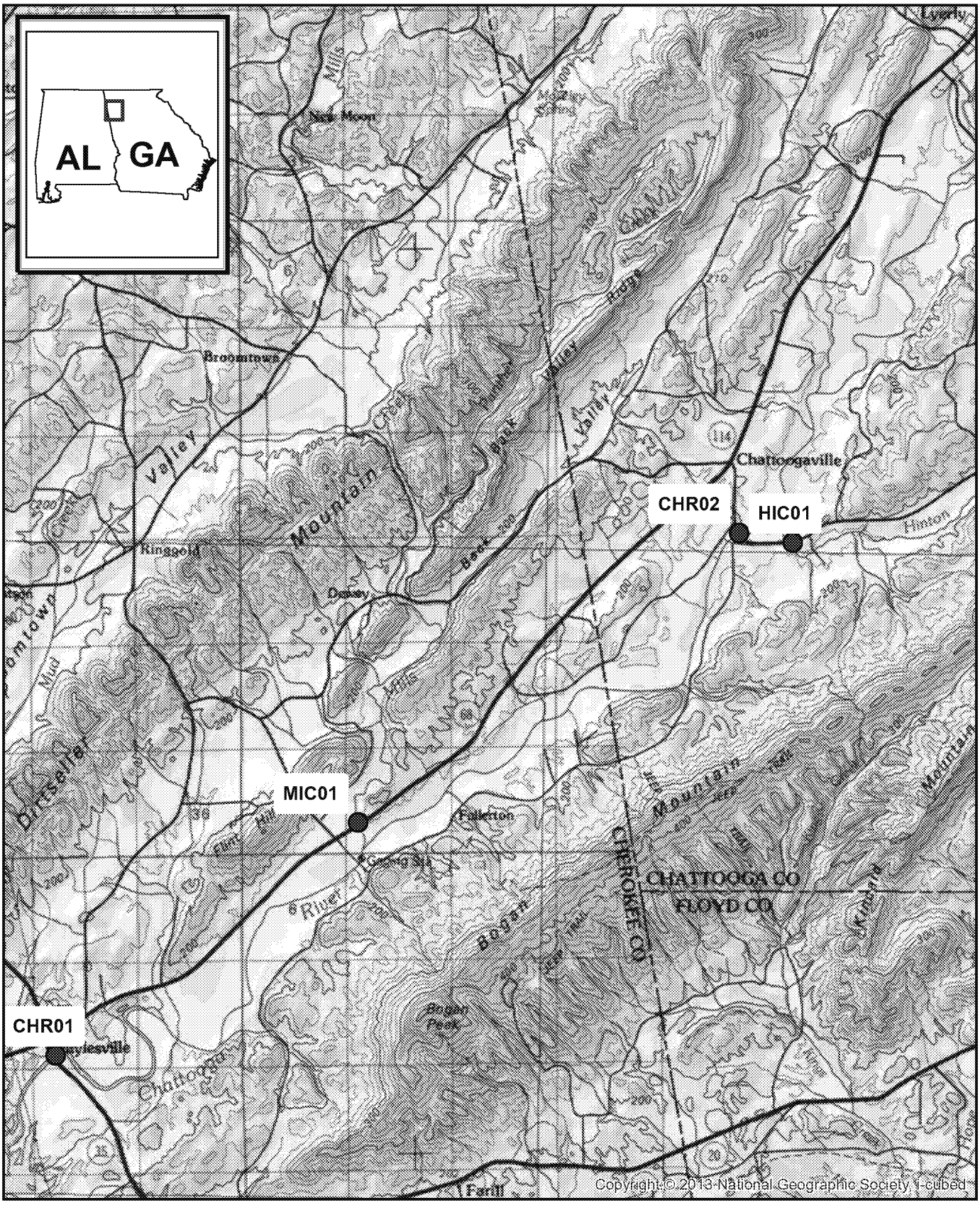


PFAS Screening:
Chattooga River (Base-flow)

Proposed Sampling Sites, November 2019

0 1.75 3.5 7 Miles

Sampling Locations



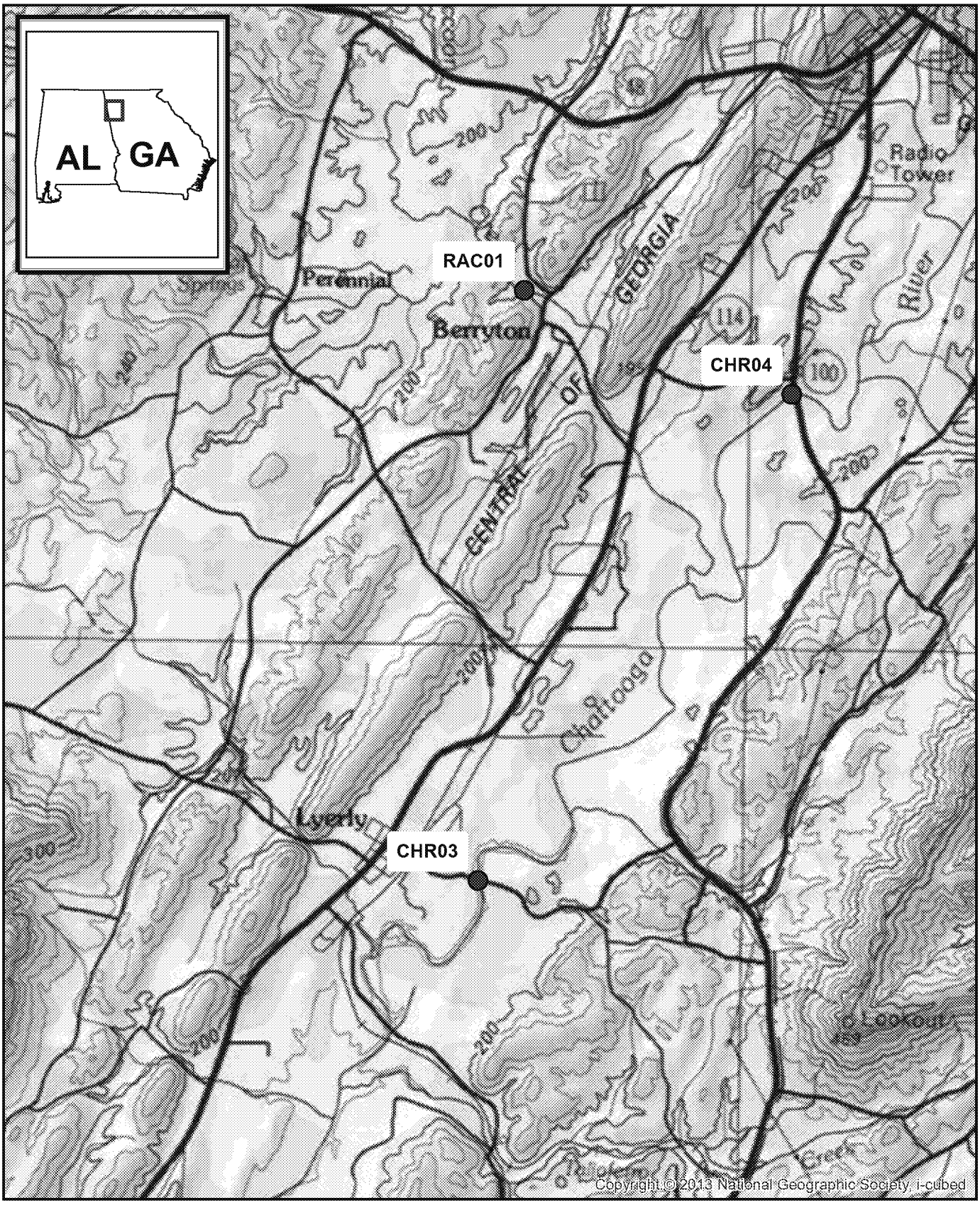
EPA

PFAS Screening:
Chattooga River (Base-flow)

Proposed Sampling Sites, November 2019

0 0.5 1 2 Miles

Sampling Locations

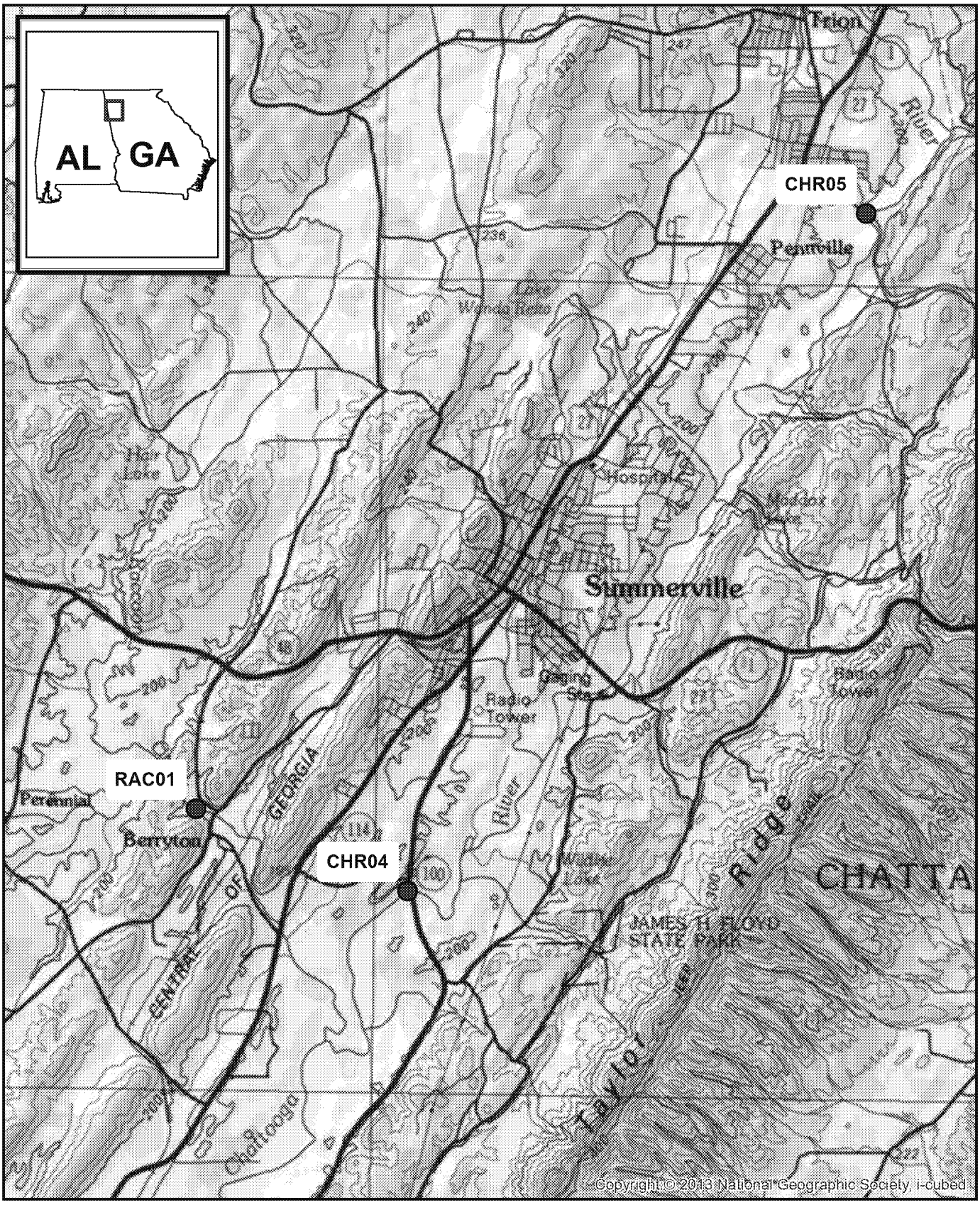


PFAS Screening:
Chattooga River (Base-flow)

Proposed Sampling Sites, November 2019

0 0.4 0.8 1.6 Miles

Sampling Locations

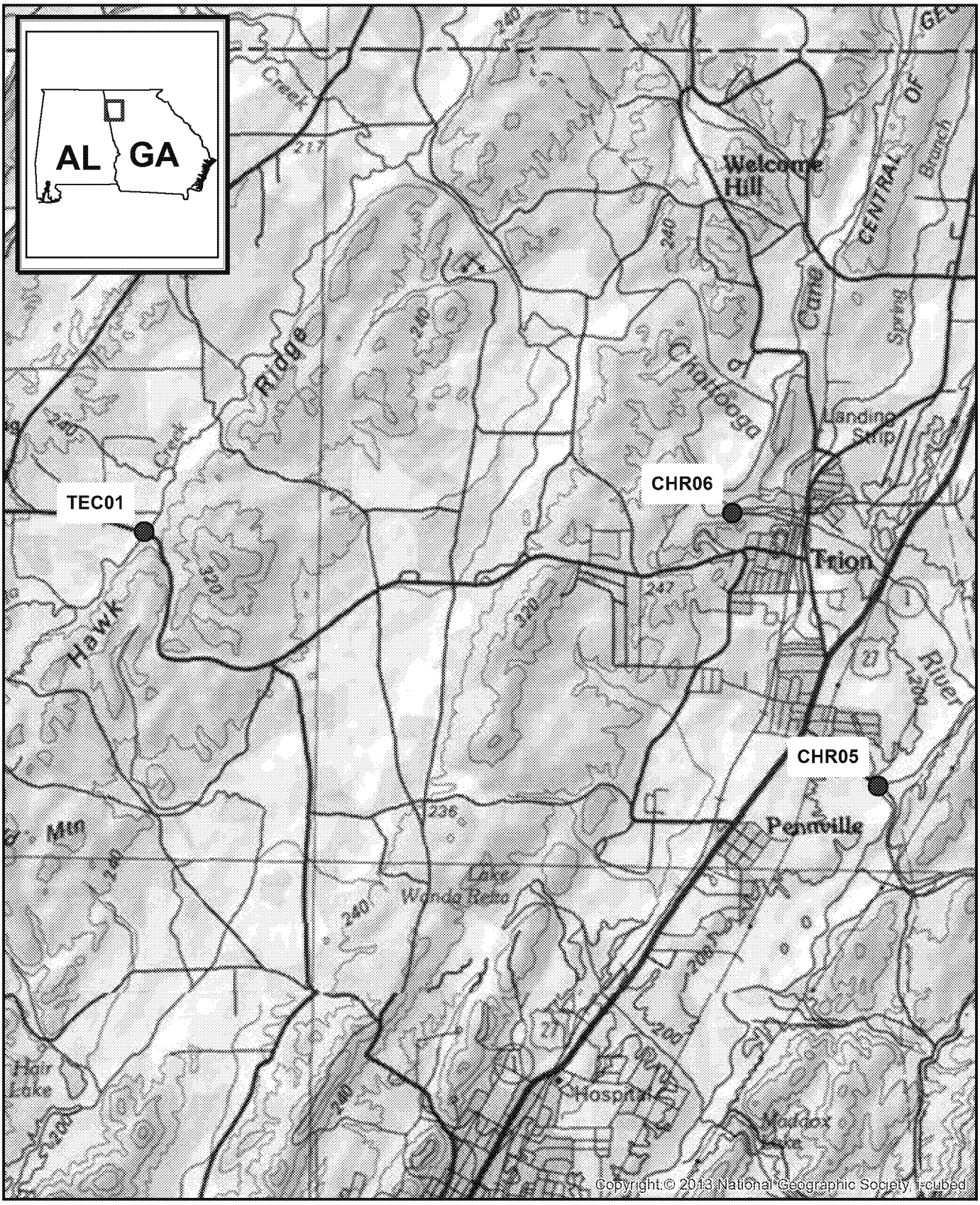


PFAS Screening:
Chattooga River (Base-flow)

Proposed Sampling Sites, November 2019

0 0.5 1 2 Miles

Sampling Locations

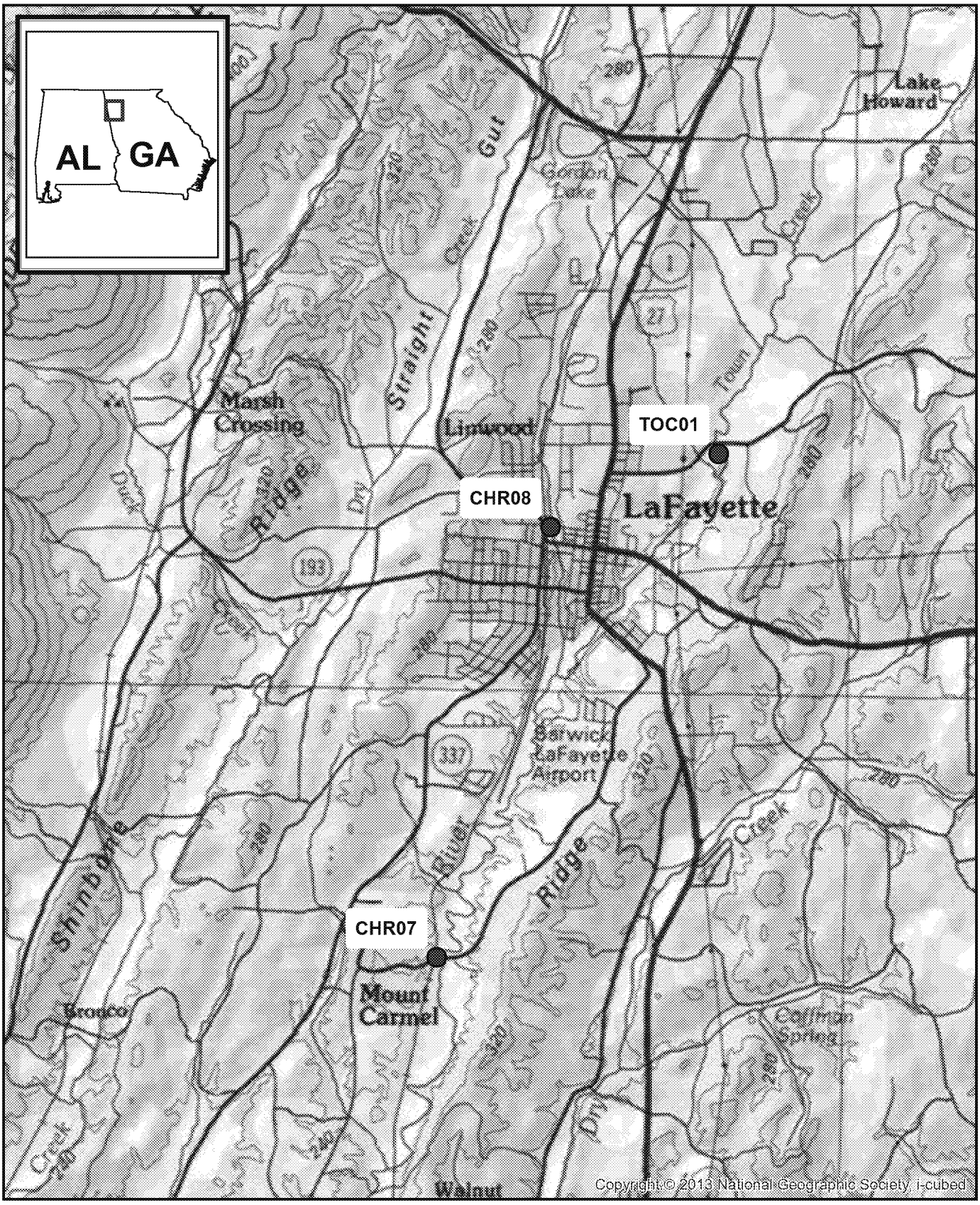


PFAS Screening:
Chattooga River (Base-flow)

Proposed Sampling Sites, November 2019

0 0.3 0.6 1.2 Miles

Sampling Locations



PFAS Screening:
Chattooga River (Base-flow)

Proposed Sampling Sites, November 2019

0 0.3 0.6 1.2 Miles

Sampling Locations

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