Quality Assurance Project Plan

Sediment Budget and Sediment Source Apportionment Study for Plum Creek

Prepared for:

Wisconsin Department of Natural Resources

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SECTION A – PROJECT MANAGEMENT

A.1 Title of Plan and Approval

Quality Assurance Project Plan
Sediment Budget and Sediment Source Apportionment Study for Plum Creek

Prepared by:
U.S. Geological Survey

____Date: 03/21/2017
Faith Fitzpatrick, U.S. Geological Survey, Project Manager/Principal Investigator

____Date: 21 Mar. 2017
Megan O'Shea, Wisconsin DNR Fox River AOC Coordinator

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Donalea Dinsmore, Wisconsin DNR Great Lakes QA Coordinator

____ Date: ___________
Jennifer Conner, GLNPO Grant Manager

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1.0 Project Management

The flow chart of project management and agency roles are shown in figure 1.

1.1 Project Organization

Prinicipal Investigator:

Faith Fitzpatrick, Research Hydrologist: U.S. Geological Survey (USGS), Wisconsin Water Science Center (WIWSC)

Table 1-1. Roles and responsibilities

Individual(s) Assigned	Responsible for:	Authorized to:
Faith Fitzpatrick, Research Hydrologist, USGS WIWSC	• Project management • Sampling design • Provide WDNR and Outagamie County with sediment chemistry and geomorphic data • Coordination with Outagamie County staff • Communicate results to WI Department of Natural Resources AOC Coordinator	• Collect, analyze, summarize, and disseminate information to WI Department of Natural Resources • Coordinate activities with Outagamie County
Megan O'Shea, WI Department of Natural Resources, Fox River AOC Coordinator	• Communication with project investigator	• Receive project updates and preliminary results • Coordinate meetings and calls for dissemination of results
Leah Kammel, Hydrologist, USGS WIWSC	• Sediment fingerprinting data analyses and sediment apportionment interpretation	• Collect, analyze, summarize, and disseminate information to WDNR • Assist with sampling design • Compile and check data • Assist with data analyses and interpretation • Communicate with

		Laboratory
James Blount, Physical Scientist, USGS WIWSC	• Sediment sample collection • Coordination and communication with Outagamie County staff	• Collect sediment samples for fingerprinting • Conduct field assessments • Compile and check data • Assist with data analyses and interpretation
Student, USGS WIWSC	• Assist with data processing and field work	• Compile and check data • Assist with field work
Sarah Francart, Outagamie County Land Conservation Department	• Assist USGS with sediment sampling and stream inventories • Provide landowner communication	• Collect sediment samples for fingerprinting • Conduct field assessments • Compile and check data • Assist with data analyses and interpretation
Jeremy Freund, Outagamie County Land Conservation Department	• Coordinate USGS activities with County schedules • Provide landowner communication	• Collect sediment samples for fingerprinting • Conduct field assessments • Assist with data analyses and interpretation • Coordinate communication with other agencies and landowners
Wisconsin State Laboratory of Hygiene	• Sample analysis	• Understanding analytical requirements in QAPP • Reporting data to USGS and WDNR (through LDES)

		• Communicate with project manager on sample submittals and any questions on sample processing and analyses.
Donalea Dinsmore, Quality Assurance Coordinator, Wisconsin DNR Office of Great Lakes	• Quality Assurance	• Approve QAPP • Arrange for EPA signature on QAPP and place in QA Track

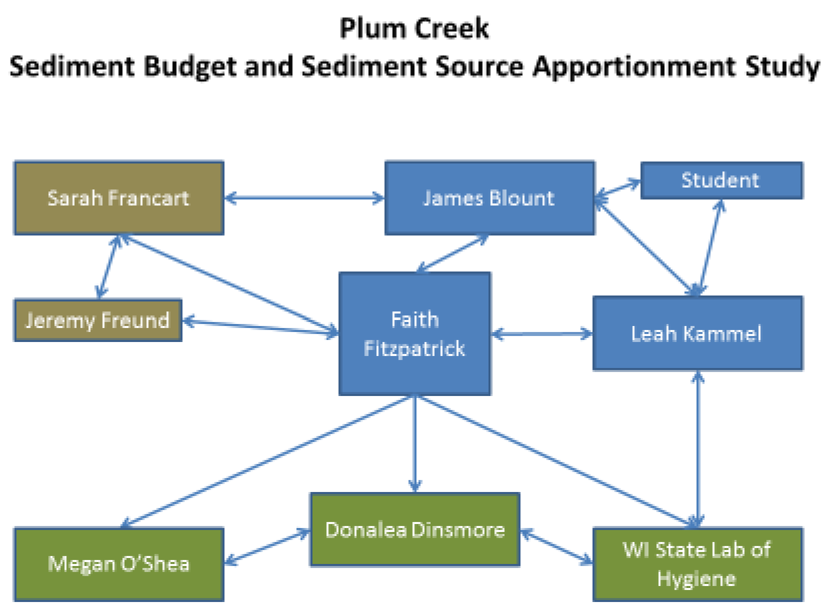

Figure 1-1. Organizational chart

1.2 Distribution List

The Quality Assurance Project Plan (QAPP) will be distributed to USGS WIWSC, Wisconsin Department of Natural Resources (WDNR), and U.S. Environmental Protection Agency (EPA) Great Lakes National Program Office (GLNPO).

A draft report of the study results will be provided to WDNR. Final reports and data files will be kept on WIWSC servers and made available to the public.

1.3 Problem Definition

Plum Creek is part of the Total Maximum Daily Load (TMDL) and watershed management plan for total phosphorus (TP) and total suspended solids (TSS) in the Lower Fox River Basin. The small watershed of

35 mi^2 has almost 20 miles of stream length on the Wisconsin state impaired waters list for TSS. Plum Creek is located about 10 miles upstream of the Green Bay/Fox River Area of Concern (AOC). The AOC has proposed beneficial use impairment (BUI) targets for eutrophication and undesirable algae based on achieving the load reductions identified in the TMDL for 7 subbasins, including Plum Creek. Based on SWAT modeling results, Plum Creek produces an estimated 31,600 lbs/yr of total phosphorus (TP) and 5,500 metric tons/yr of TSS (Cadmus, 2012). Average annual loads at the USGS continuous-recording streamgage on Plum Creek (USGS 04084911) were 28,400 lbs/yr of TP for 2011-14 water years. The TMDL goal is to reduce the TP loading by 77 percent and the TSS load by 70 percent. Based on a SWAT model output, agricultural land in Plum Creek is estimated to contribute 94 and 95 percent of the annual loading of TP and TSS, respectively. Natural areas are estimated to contribute 1 percent of the TP and TSS. TP and TSS from bank erosion sources were not included in the modeling (Cadmus, 2012). However, recent stream inventories of Plum Creek by Outagamie County indicate that 24 of the 43 miles inventoried had actively eroding banks. Preliminary estimates are that these banks could be contributing 45 percent of the TSS annual loading measured at the USGS gage. The percentage of loading of TP from the banks is unknown, but may be similar. If stream processes are producing almost half of the annual loading of TSS, the proposed TMDL goal to reduce TSS by 70 percent will not be achievable through upland soil conservation practices alone.

One of the first steps in the sediment TMDL process along with identifying targets is to identify the major sources of sediment (U.S. Environmental Protection Agency, 1999). A stream corridor-based sediment budget and source apportionment study is needed to quantify the proportion of the TP and TSS loading originating from in-stream sources of bank and channel erosion compared to soil erosion. Gullying associated with headward extension of stream networks also needs to be quantified as a possible TP and TSS source. This proposal describes a combined sediment budget and sediment fingerprinting approach that will help identify the proportion of annual loading of TP and TSS originating from stream corridor sources. The results from this study will be compared to expected field contributions based on RUSLE2 calculations and measured TP and TSS loadings from the USGS water quality monitoring stations. The source assessment results can be used in all subsequent steps of the TMDL process, including monitoring and targeted implementation of the plan.

1.4 Project Description

This study will build off of previous stream corridor inventories of sediment sources conducted by Outagamie County and additional sampling activities by others that are being conducted in the watershed. The study approach will follow the newly released EPA manual on identification of sediment sources and sinks (Gellis et al., 2016). Data on annual TP and TSS loading calculated at the USGS streamgage on Plum Creek (USGS 04084911) will be used for the watershed export and transport. The study will also be linked with the 2016 USGS GLRI study of characterizing stream functional zones of high potential nutrient cycling (William Richardson, 2015, USGS, oral commun.). The study will take two years to complete, the first year for writing a QAPP, compilation of existing data, watershed reconnaissance of geomorphic setting, collection of sediment samples, and conducting field assessments needed for estimating a sediment and phosphorus (P) budget for the stream network (table 1-2). The second year is needed for laboratory analyses, data workup and interpretation, and publishing results.

Table 1-2. Timeline for proposed investigation.

	2016			2017												2018								
Task	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep
QAPP writing	X																							
Compilation of existing data	X	X																						
Site selection	X	X	X																					
Riverwalk/stream inventory		X	X			X	X																	
Rapid geomorphic assessment						X	X	X																
Bed and bank sampling		X	X			X																		
Insitu suspended sediment sampling	X	X	X			X	X	X	X	X	X	X												
Upland soil sampling	X	X	X																					
Sediment sample prep and laboratory analyses		X	X	X	X	X	X	X	X	X	X	X	X	X	X									
Data entry	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X									
GIS analyses	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X						
Constructing sediment and TP budget										X	X	X	X	X	X	X	X	X						
Sediment fingerprinting mixing model										X	X	X						X	X	X				
Results/writeup and journal submission																			X	X	X	X	X	X
Project update meetings with partners			X			X							X				X							
Presentation of results at meeting																				X	X			

As part of the sediment budget approach, the USGS will first become familiar with the extensive bank erosion inventory conducted by Outagamie County (S. Francart, Outagamie County LCD, written commun., 2016). In close coordination with Outagamie County staff, the USGS will conduct an inventory of sediment sources and sinks using a river walk approach and aerial photo analyses (Gellis et al., 2016; Fitzpatrick et al., 1999; 2015; 2016; Miller, 1986), where additional information is needed beyond what was done in the County inventory. The river walk approach is used to inventory and map predetermined features such as bank erosion and is usually done for multiple miles of stream channel of interest. Channel sources of sediment will include bank erosion, valley side and terrace erosion, gully erosion and channel incision. For bank erosion rates, estimates of lateral recession rates will be employed (Natural Resources Conservation Service, 2003) and sediment and P sinks will be measured and mapped. These include measurements of the volume of soft, fine-grained sediment deposited in the channel, depositional bars, as well as floodplain deposition. Channel derived sediment sources, transport, and sinks will be described in terms of a longitudinal continuum along the drainage system and mapped in a geographic information system (GIS).

Rapid geomorphic assessments of channel characteristics will be conducted at 30 sites in the Plum Creek watershed. Sediment samples will be collected from eroding banks and soft sediment deposition at the 30 sites and analyzed for TP, particle size, density, and a suite of trace elements used in sediment fingerprinting. Samples will be analyzed at the Wisconsin State Laboratory of Hygiene (WSLH).

The USGS streamgage on Plum Creek will be augmented with an in situ suspended sediment sampler (Phillips et al., 2000). The sampler will be used to collect suspended sediment for fingerprinting and TP laboratory analyses. Samples will be collected monthly for approximately one year starting in November 2016 to characterize potential difference in sources over a range of hydrologic events and seasons.

Sediment fingerprinting and source apportionment will be conducted for agricultural upland, hillslope processes, and channel sources such as bank erosion upstream of the Plum Creek streamgage using techniques previously developed by Gellis et al. (2016) and used in the Pecatonica watershed (Lamba et al., 2015a, b). Approximately 75 sediment samples will be collected from cropland, road ditches, forested slopes, gullies, and eroding banks. The targets will include suspended sediment and soft streambed sediment samples. Since the Plum watershed is naturally rich in fine-grained sediment, the study will help toward distinguishing anthropogenic vs. natural sediment loading and distinguish reaches along the stream network with gullying, bank, and channel erosion.

The laboratory analyses of 57 tracers conducted on the sediment samples for source apportionment includes TP. The analytical method is different than that used by agronomists and farmers to measure soil P or TP in water samples. A portion of the samples collected for this study will be retained for additional analyses of TP used for nutrient tests in agricultural fields or for studies interested in the more biologically active oxalate extractable portion.

Throughout the study the USGS will continue close coordination and communication with Outagamie County, the WDNR AOC coordinator, the WDNR Fox River TMDL coordinator, and the Lower Fox River TMDL Agriculture Committee.

Products from this study will include GIS maps and spreadsheets of stream corridor sources and sinks of sediment and TP. A presentation will be given and made available to project partners and local watershed groups. A journal article will be published with the results, with submittal to a journal (to be selected with input from partners) at the end of year 2.

1.5 Site Description

Plum Creek is an eastern tributary of the lower Fox River in Outagamie County. Soils are generally clayey. Topography is steep and the valley of Plum Creek is deeply incised (fig. 1-2). The river enters the lower Fox River upstream of the community of Wrightstown and below Rapide Croche Dam. There are two USGS continuously operating streamgages on Plum Creek, the drainage area upstream of the main gage on Plum Creek (04084911) is 21.2 mi^2. The gage has been operating from October 2010 through the present in cooperation with the University of Wisconsin-Green Bay and Fox Wolf Watershed Alliance. A second streamgage is located on West Plum Creek (04084927) which drains 9.4 mi^2 and joins Plum Creek near its mouth. This newer gage has been operating since October 2014.

Land cover in Plum Creek is mainly agricultural cropland, with smaller percentages of forested land and grassland/pasture (Homer et al., 2015) (fig. 1-1). Much of the grassland is made up of right-of-ways along roads and grassy areas adjacent to subdivision or rural residential lots. There are few pastures in the watershed. Much of the forested land is located adjacent to Plum Creek and its tributaries, along steeply sloping valley sides.

Based on the land cover distribution in Plum Creek, the land-cover categories that will be used for upland soil sampling will likely include cropland, grassy rights-of-way along road ditches, and forested land (fig. 1-1).

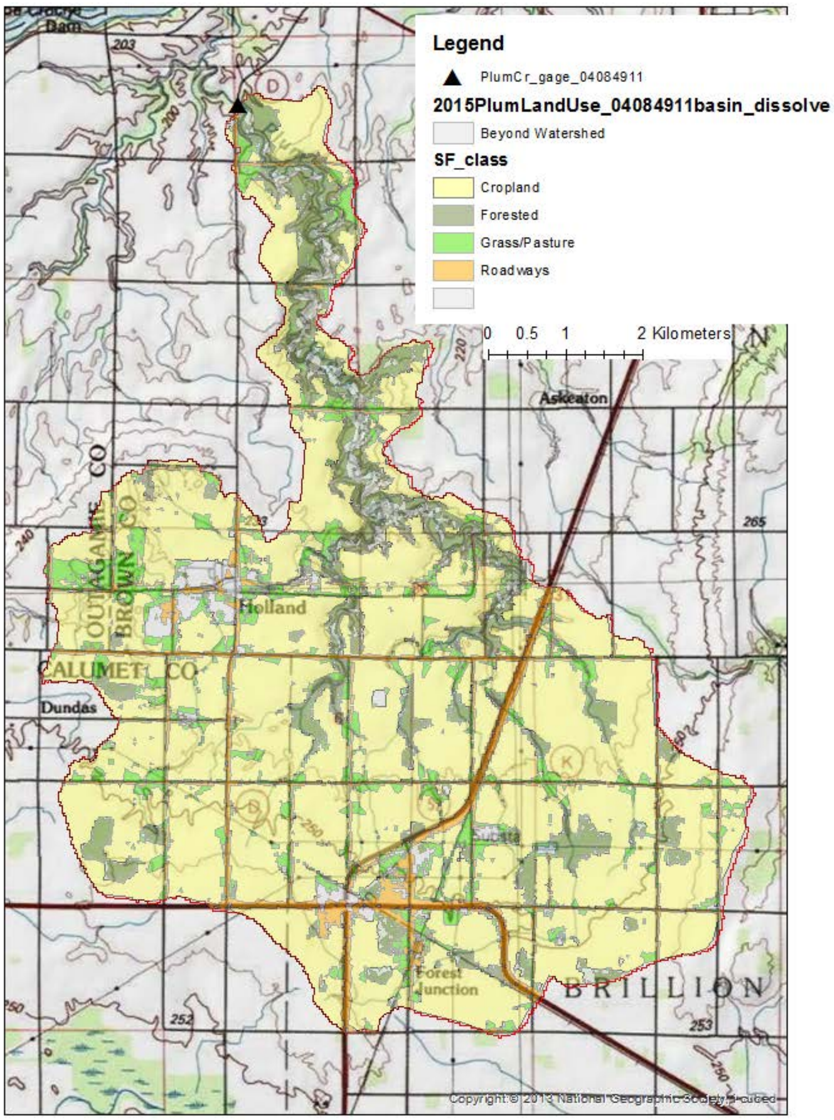

Figure 1-1. Plum Creek study area with major land-cover categories (Homer et al., 2015).

Baseline monitoring data from 2011-15 at the USGS monitoring station on Plum Creek indicate annual suspended sediment loads have ranged from less than 4,000 tons/yr in 2012 and 2015 to almost 13,000 tons/yr in 2014 (fig. 1-2). Sediment loads were lower in 2011 than in 2014 even though streamflow (Q) was higher, illustrating likely differences in the timing and intensity of the precipitation and resulting runoff. Total phosphorus (TP) loads ranged from 13,500 lbs in 2012 to over 40,000 lbs in 2014.

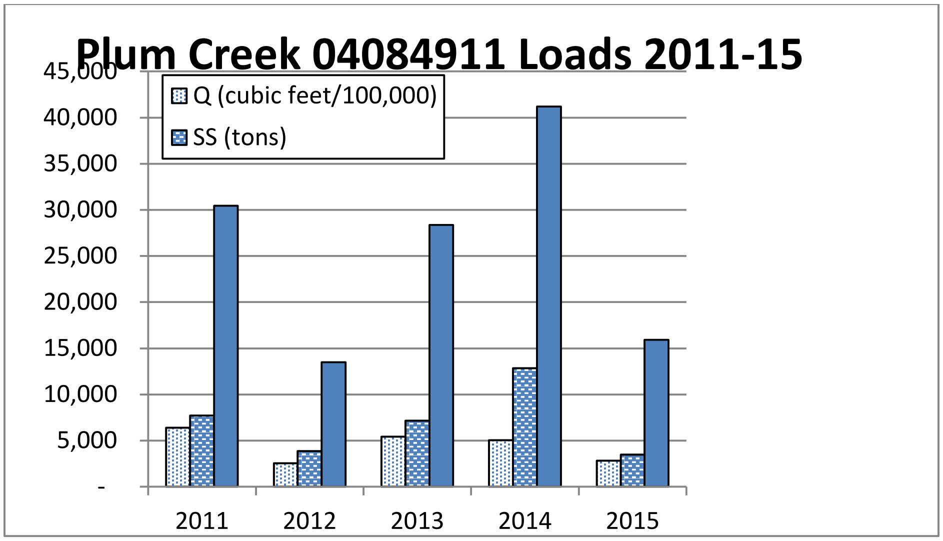

Figure 1-2. Plum Creek annual discharge and suspended sediment and total phosphorus loads at U.S. Geological Survey streamgage (identification number 04084911), 2011-15.

Plum Creek and West Plum Creek currently have one year's worth of overlapping data from the 2015 water year. These preliminary data indicate that, on a unit-area basis, water yields are higher from West Plum Creek than Plum Creek, whereas Plum Creek sediment and total phosphorus yields are higher than West Plum Creek (fig. 1-3).

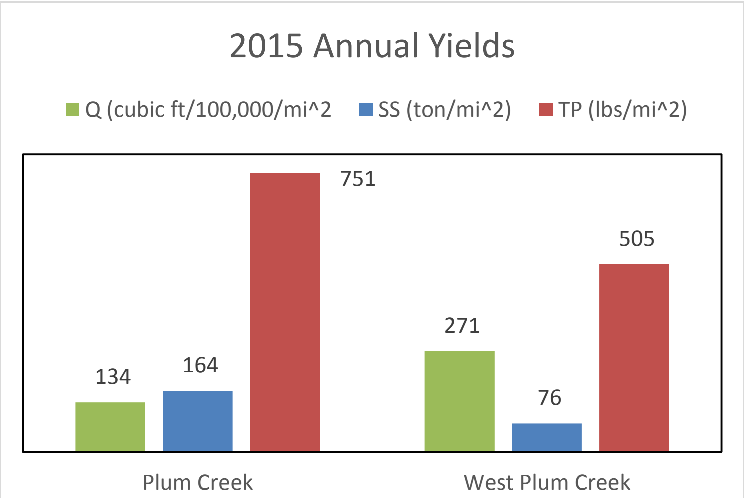

Figure 1-3. Comparison of Plum Creek and West Plum Creek annual discharge, suspended sediment, and total phosphorus yields, 2015.

1.6 Quality Objectives and Criteria for Measured Data

The overall QA objective for analytical data is to ensure that data of known, acceptable, and defensible quality are generated. To achieve this goal, data must be reviewed for 1) precision, 2) accuracy or bias, 3) representativeness, 4) comparability, and 5) completeness. Data Quality Objectives (DQOs) that cannot be expressed in terms of accuracy, precision, or completeness will be reported by fully describing the specified method; all other QA requirements will be fulfilled. Measurement performance criteria for data to be collected during this project are discussed in the following subsections.

Data Quality Objectives (DQOs) for the ongoing basin studies include:

1. Identify and collect sediment from potential source areas in the basin. Sampling locations are identified using a GIS. Source areas of sediment include upland fields, forested slopes, ditches along roadways, streambanks, and gullies.
2. Fluvial sediment is collected during major events and for an entire year to ensure that seasonal changes in sediment sources are identified. Fluvial sediment includes suspended and streambed.
3. The objective of the fluvial and sediment source data collection is to provide accurate and precise elemental analysis that can be used to determine the sources of fine-grained sediment and associated TP
4. Apportion the loading of stream TSS and TP to various source areas listed in (1) during runoff events and seasons.

The statistical analyses (Helsel and Hirsch, 2002; Gellis et al., 2016; Gorman-Sanisaca et al., 2017) will be computed by the USGS PM and will be checked by the USGS QAO.

Measurement performance criteria for data to be collected during this project are discussed in the following subsections.

Precision and Accuracy

Precision is the degree of agreement among repeated measurements of the same characteristic, or parameter, and gives information about the consistency of methods. Accuracy is a measure of confidence that describes how close a measurement is to its “true” value. Field replicates will be collected for sediment source and fluvial samples.

To control variance in field samples, sources and targets of sediment are composite samples covering a representative areal extent. Upland soil samples are a composite of 30 point samples spaced 10 m apart along a grid. Streambank samples are a composite of 15-30 point samples spaced approximately 0.3 m apart depending on the areal extent of erosion. In situ suspended sediment samples are composited over a time period of a month. Gully and soft streambed sediment samples are a composite of 15-30 point samples spaced approximately 1 m along a 150-m reach.

Field replicate samples are collected by side-by-side sampling of the same grid. In situ suspended sediment samples are compared to an additional sampler set near the main sampler, a composite cross section sample, and a suspended sediment sample collected from the automated water sampler at the USGS streamgage.

Field analytical precision will be evaluated by the relative percent differences (RPD) between field replicate samples using the following formula:

RPD = [(R1 - R2)/ ((R1 + R2)/2)] x 100

Where: R1 = the larger of the two replicate values

R2 = the smaller of the two replicate values

The values of RPD are intended to provide information on the variability of field samples. Differences that are greater than 10 percent are flagged but not discarded. Replicate analyses for the two samples are averaged for the final sediment sourcing analysis.

Quality assurance procedures are described in the quality assurance/quality control manual Techniques of Water-Resources Investigations of the USGS (http://water.usgs.gov/pubs/twri/, Book 4, Chapter A3: Hydrologic Analysis and Interpretation).

Data Representativeness

Representativeness is the extent to which measurements actually represent the true environmental condition. It is the degree to which data from the sampling accurately represent a particular characteristic of the watershed that is being tested. Representativeness of samples is ensured by adherence to standard field sampling, measurement, and laboratory protocols. The design of the sampling scheme and number of samples for this plan provide representativeness of the part of the watershed being monitored (see section 2.1 on design for more details). The representativeness of the data is dependent on 1) the sampling locations, 2) the number of samples collected, and 3) the sampling procedures. Site selection and use of only approved analytical methods will ensure that the measurement data represents the conditions at the site. The goal for meeting total representation of the targeted drainage area is tempered by the availability of time and funding. Representativeness will be measured with the completion of sample collection in accordance with the approved QAPP.

Based on previous studies and guidance in Gellis et al. (2016) a minimum of 15 samples should be collected from each source area.

The level of completeness from the in situ suspended sediment samples is that they represent runoff events and low flows from all non-ice seasons. The majority of TP loads happen during events, making it critical to capture the runoff events with the in situ samplers. The sampler will be taken out during the winter, but placed back on its mounts above the ice if need to catch a winter rain on snow event.

Data Comparability

Comparability is the degree to which data can be compared directly to similar studies. The apportionment results from this study can be compared with other watershed studies that use fingerprinting and budget methods to determine the major sources of sediment. However, the results from the Plum Creek study should not be directly applied to other watersheds. Caution must be used when applying the results with consideration for differences in physiography, soils, geomorphology, and land use, and channel alteration history. The comparability of the data produced is predetermined by the commitment of the staff to use only approved procedures as described in this QAPP. Comparability is also guaranteed by reporting data in standard units, by using accepted rules for rounding figures, and by reporting data in a standard format as specified in this QAPP. The methods used to determine sediment sources are methods that are established in the literature.

Comparability will be checked by collecting replicate samples (about 10 percent of sediment source samples). Replicate samples are collected by sequentially taking two field samples for each analysis from the same source. Comparability goals for field replicate samples are to have results within 10 percent of each other. Samples that exceed this are flagged. Replicate analyses for the two samples are averaged for the final sediment sourcing analysis. The project manager has the option of discarding the sample if the percent difference between the two replicates is greater than expected for that source type.

Data Completeness

Completeness is the comparison between the amounts of usable data collected versus the amount of data called for in the sampling plan. For the purposes of this project, it will be measured as the percentage of valid results obtained compared to the total number of samples taken for a parameter. Ideally, 100% of the data should be available. However, the possibility of a small percentage of unavailable data due to accidents, insufficient sample volume, or lost samples, is to be expected. Therefore, it will be a general goal of the project that 90% data availability is achieved. Percent completeness is calculated using the following formula:

% Completeness (per parameter) = (# of valid results/# of samples taken) x 100

The primary objectives of the QA process are to assure that the data collected and analyzed are representative of the site, and can be used to support the needed decision. For each soil or sediment sample, the compliment of metals included in the analysis needs to be consistent to develop fingerprint signatures. Missing elements throughout the data set may affect the usability of the data. At least 15} metals are necessary to establish a fingerprint that can be attributed to a sediment source.

1.7. Special Training and Certification

No highly specialized training or certification of personnel is required for this project. Faith Fitzpatrick is an author on the EPA manual for sediment source identification (Gellis et al., 2016). As part of the manual development, Leah Kammel worked with authors, specifically on the sediment apportionment technique (Gorman-Sanisaca et al., 2017; Gellis et al., 2016) on a test run for a similar project for Otter Creek (Sheboygan River).

1.8 Documents and Records

All data will be stored at the USGS WIWSC. Data that do not reside in an existing federal or state data base will be published in USGS ScienceBase (https://www.sciencebase.gov/catalog/). Field data gathered during this project are recorded on site at the time sampling occurs using a datasheet or field book. Field related data are entered into spreadsheets when returning from the field. Photographs of sampling sites are taken during each field excursion.

If any changes to this QAPP are required during the project, a memorandum will be sent to each person on the distribution list describing the change(s), pending approval by appropriate parties. Any such memoranda will be attached as addenda to the QAPP.

The USGS will be responsible for maintaining original documents pertaining to field monitoring and sediment analysis data. This will include a summary of the types of data collected, sampling dates, data values, and any problems observed during sample collection and QC checks performed during sample collection.

Any amendment or modification of the QAPP will be reviewed by the project QA officers, before implementation of changes. Any such changes will require QA review.

2.0 Data Acquisition

2.1 Sampling Process Design

Recent advances have been made in developing field-based approaches to identify sediment sources using the sediment fingerprinting and sediment budget approaches, which provide a direct method for quantifying watershed sources of fine-grained sediment, including determination of upland soil erosion compared to channel and bank erosion (Gellis et al., 2016). The proportion of specific sources of sediment is determined by establishing a minimal set of physical and/or chemical properties, i.e., tracers, which uniquely define each source. Suspended sediment collected under different flow conditions exhibits a composite, or fingerprint, of properties that allows them to be traced back to their respective sources.

Areas designated for upland sediment source sampling will be identified for the Plum Creek through GIS analyses of land cover and the expertise of Outagamie County Land Conservation Department staff. Once source area categories are identified, a stratified random sampling approach is used with a GIS to select 15 sample locations for upland soils by land-cover type. The procedure is run at least twice, in case alternative sites are needed. It is common to need alternative sites during the site selection process, due to two major reasons: denial of access to a particular site on private land, and land-cover changes that occurred after mapping. The upland source categories for Plum Creek include cropland, forested, and grassed ditches and rights-of-way along roadways (table 2-1).

Table 2-1. Approximate number of sediment samples collected for fingerprinting for the Plum Creek study.

Type of sample	# samples	# QA samples	#total samples
Upland -- cropland	15	1	16
Upland roadways, grassy right-of-ways	15	1	16
Upland/slopes forested	15	1	16
Gullies	15	1	16
Eroding banks	15	1	16
In situ suspended sediment	10	3	13
Bed samples	10	1	11
	Total # of samples:		104

For channel sources of sediment eroding bank samples will be collected from about 15 representative eroding banks along Plum Creek. Site selection will be based on the previously conducted eroding bank inventories and include a range of slope and stream orders. Sediment will also be collected from eroding gullies. In terms of landscape position, the gullies may show a different elemental signature from forested slopes and channel banks because the gullies will likely be incised into unweathered glacial deposits, potentially below the zone of soil development. Eroding channel banks sampled will be composed of legacy alluvial sediment. Priority will be given to sampling locations for eroding banks and gullies that can be paired spatially with nearby forested slopes, especially if accessibility is an issue.

Target samples include 10 suspended sediment samples collected with an in situ suspended sediment sampler located at the Plum Creek streamgage. The sampler will be emptied on a monthly basis and operated for a year (during unfrozen conditions) to capture seasonal variability in sources. Three additional suspended sediment samples will be collected for comparison to the in situ sampler. A cross-section composite sample will be collected during one event to provide additional information on the representativeness of the location of the in situ suspended sediment sampler relative to the cross section. Two additional in situ suspended sediment samples will be collected – one from a co-located sampler next to the main sampler and another from the automated sampler at the West Plum Creek gage.

Full details on site selection can be found in:

SOP.Sediment.fingerprinting_Site.selection_20170310.pdf

2.2 Sampling Methods

Sampling for this study includes collection of sediment from terrestrial and fluvial settings. The total mass needed for trace elements, phosphorus, and particle size analyses is about 3 g dry weight of < 63 micron-sized sediment. The standard operating procedure for collection of sediment is in:

SOP.Sediment.fingerprinting_Sediment sampling_20170310.pdf

Locational information is stored with a where in the grid – Grid size and location, with handheld GPS with accuracy of ± 20 ft.

Field IDs and Station numbers will be recorded for each sampling site.

In situ suspended sediment samples are compared to an additional sampler set near the main sampler, a composite cross section sample, and a suspended sediment sample collected from the automated water sampler at the USGS streamgage.

Details specific to sampling sediment in Plum Creek for target and source samples follow.

Target Fluvial Suspended Sediment Samples

Placement – A passive in situ suspended sediment sampler will be deployed in November 2016 and operated through September 2017 except when the channel is iced over. Samples will be retrieved on a monthly basis. The passive sampler is secured to two posts that are firmly hammered into the channel bed. The passive sampler can be moved up and down on the posts using hose clamps or other fasteners that can be removed. The passive sampler can be placed anywhere in the channel cross-section where the sampler will obtain a representative sample. Backwater areas, or placing the intake too close to the bed or near obstructions (boulders, bridge piers, trees, etc.), should be avoided.

Figure 2-2. In situ suspended sediment sampler tube setup prior to lowering the sampler below water column. Photograph by James Blount, USGS, October 2016.

Sample retrieval -- To retrieve samples from the passive sampler, the technician will use gloved hands to place a plastic stopper in the inflow and outflow tubes prior to lifting the sampler out of the water. After the sampler is removed from its brackets, the capped end of the sampler will be carefully unscrewed and the contents of the tube will be emptied into a 5-gallon clean plastic or Teflon lidded bucket. In general, about 0.5 L of the "settled" wet sediment will be collected for analysis. The sampler will be scrubbed with plastic brushes and native water and replaced on the brackets, and thoroughly checked to make sure the intake and outlet tubing are free from debris and algae. Weather and precipitation will be tracked at the gage and nearby climate stations to record timing of storm events.

Additional samples -- Three additional suspended sediment samples will be collected for comparison to the in situ sampler. A cross-section composite sample will be collected during one event to provide additional information on the representativeness of the location of the in situ suspended sediment sampler relative to the cross section. Two additional in situ suspended sediment samples will be collected – one from a co-located sampler next to the main sampler and another from the automated sampler at the West Plum Creek gage.

Target Fluvial Soft Sediment Samples

Soft streambed sediment will be collected once during the river inventories and/or rapid channel assessments using approximately 4-cm diameter plastic or Teflon tubing approximately 30 cm long. The tube is inserted into the top 2 cm of soft sediment. A plastic spatula or scraper is placed under the tube and the tube is brought up through the water column. Clear water is poured off the top of the tube and the sediment/water slurry is poured into a wide-mouthed 1 L plastic jar. Approximately 15 points are sampled from a depositional area and composited into a single jar, usually within a 150-m long reach coincident with the rapid geomorphic assessment. The jar will be about half to three quarters filled with composited wet sediment. A field replicate of soft sediment will be collected by side-by-side sampling of the same points.

Source Upland, Bank and Gully Samples

Soil samples for source analysis from upland areas are taken from the top 1-2 cm of the soil surface with a plastic hand shovel. To account for variability in the tracer properties at upland sites, sediment is collected across transects and composited into one sample. Samples are collected at three transects with each transect 100 m in length and spaced 10 m apart for a total of 30 point samples composited into a gallon-sized plastic bag. The transect length and number may be adjusted to fit the sampled land cover. Shovels are washed with phosphate-free liquid detergent and rinsed with deionized water between sampling locations.

To obtain a representative sample of sediment from eroding banks, the surficial 1-2 cm of exposed sediment will be sampled with a plastic hand shovel from the bottom to the top of the bank face. Three to five points along each of three to six transects are sampled, depending on the height and length of the eroding bank, with a total of 15-30 point samples composited into one gallon-sized plastic bag. If streambanks are eroding on both sides of the channel, then samples will be collected on both sides of the river and composited into one bag. Plastic shovels are well-rinsed between locations with native stream water.

Actively eroding gullies are sampled in a similar fashion to streambanks, with the top 1-2 cm of sediment sampled with a plastic hand shovel. Sampling points include the actively eroding bottom and sides of the gully. The sample will be a composite of about 15-30 points samples composited into one gallon-sized plastic bag. Similar to the other source samples, the transect or grid size will be fit to the gully dimensions.

For each of the three types of source samples, the composited sediment will fill about one-quarter to one-half of the gallon-sized plastic bag.

Field replicates of source samples will be collected by side-by-side sampling of the same grid.

2.3 Sample Handling and Custody

All samples are assigned a unique identification number in the field, which is then used to link the samples with the appropriate laboratory forms. After collection, samples are transported to the preparatory laboratory at the USGS WIWSC in a plastic cooler and stored on ice during the warm season. According to EPA's METHODs for determining trace metals (e.g. 200.8 (https://www.epa.gov/sites/production/files/2015-06/documents/epa-200.8.pdf) (Creed et al., 1994), "Solid samples require no preservation prior to analysis other than storage at 4°C. There is no established holding time limitation for solid samples." Once the samples arrive at the USGS, fluvial samples are allowed to settle for a minimum of 24-72 hours until the clear water can be carefully poured off the sediment slurry. The fluvial slurry, soil, and sediment samples are frozen prior to sample processing to keep the samples from becoming moldy or experiencing unwanted organic-matter decomposition during storage.

GBF1603_PlumCreek-Lab slip.xlsx

Standard procedures for storing, processing, sieving, and labeling samples are in:

SOP.Sediment.fingerprinting_Sample.processing.sieving_20170320.pdf

After thawing, all fluvial and source samples are wet-sieved through a 63-micron polyester sieve using de-ionized water. ASTM D3977-97 (2002) Method C, wet-sieving filtration, is used. The <63-micron sample of water-sediment mixture is captured in a glass bowl(s) or jars and dried at 60 degrees Celsius for 24-48 hours. After drying, the sediment in the bowl is weighed, lightly ground if needed with a ceramic mortar and pestle, and split into two plastic vials for transfer to WSLH. The vials will contain on optimum of 1 g of dried sediment each for trace elements analyses, particle size determination, and phosphorus (ICP) analyses. The remaining portion of <63-micron fraction will be bagged and refrozen, to be potentially used for laboratory analyses at a later date for additional phosphorus concentration analyses using different methods and organic matter content. The >63-micron fraction will be dry-sieved for sand-sized particle size determinations at the WIWSC.

The dried and <63-micron samples for tracers, particle-size, and P determinations will be hand-delivered to the WSLH for laboratory analyses. An example Test Request Form is attached to the sample processing SOP as well as to the end of this document.

Upon arrival at the WSLH the samples are unpacked and assigned unique internal control numbers. Samples are properly stored within the laboratory until analysis (refer to sections 3.0). To ensure that the samples are not contaminated and to provide personal protection for the sampling team, the technicians will wear nitrile gloves during sample collection. A new pair of gloves will be used for processing each sample.

2.4 Analytical Methods

Table 2-2 lists the inorganic and trace elements that are recommended for use in fingerprinting studies (Gellis et al., 2016). Additional secondary tracers include Dy, Eu, Gd, Hf, Ho, Lu, Nd, Pd, Pr, Pt, Rh, S, Sm, Yb, and Zr. These additional elements include the platinum group elements (PGM) useful for identifying anthropogenic (vehicle impact) sources, sulfur (anthropogenic sourcing and redox conditions), rare earth elements (helpful in identifying anthropogenic sources and unique signatures). Additional conservative elements are Hf and Zr, which may help with normalizing concentrations of non-conservative elements. Sediment concentrations are determined by inductively coupled plasma-emission spectrometry- high resolution mass spectroscopy (WSLH SOP 420.0 Revision 1, 2016 (comparable to EPA Method 200.8, SW846-Method 200.8 ; Creed et al., 1994; Martin et al., 1994) using microwave digestion methods ESS Method 550.0 Revision 6 (WSLH, 2015).

Table 2-2. List of 41 core elements typically used for fingerprinting sediment (Gellis et al., 2016)

Aluminum (Al)	Cadmium (Cd)	Gallium (Ga)	Mercury (Hg)	Scandium (Sc)	Tin (Sn)
Antimony (Sb)	Calcium (Ca)	Iron (Fe)	Molybdenum (Mo)	Selenium (Se)	Titanium (Ti)
Arsenic (As)	Cerium (Ce)	Lanthanum (La)	Nickel (Ni)	Silver (Ag)	Uranium (U)
Barium (Ba)	Cesium (Cs)	Lead (Pb)	Niobium (Nb)	Sodium (Na)	Vanadium (V)
Beryllium (Be)	Chromium (Cr)	Lithium (Li)	Phosphorus (P)	Strontium (Sr)	Yttrium (Y)
Bismuth (Bi)	Cobalt (Co)	Magnesium (Mg)	Potassium (K)	Thalium (Tl)	Zinc (Zn)
Boron (B)	Copper (Cu)	Manganese (Mn)	Rubidium (Rb)	Thorium (Th)	

The trace elements analyses will be conducted at WSLH EHD Metals Department. Data will be reported in milligrams per kilogram (mg/kg) for the soil/sediment matrix. Particle-size determinations for the <63 micron fraction will be done at WSLH Inorganics Chemistry Department using a Beckman Coulter Multi-sizer Particle Size Counter (ESS INO Method 355.3; WSLH, 2011a, b).

2.5 Quality Control

Quality Control Procedures for determinations of elemental analysis are described below. These procedures apply to all the analyses WSLH.

Sample collection, handling, analysis, and data handling procedures have been established to ensure data accuracy, precision, and comparability. Analyses of field replicate samples will be evaluated to assess environmental variability. Laboratory duplicate and spike samples will be evaluated to assess analytical procedures and laboratory equipment. Field replicates are submitted on 10 percent of all water-quality samples submitted to the laboratory (see section 1.6). Field replicates are used to assess overall precision, including the reproducibility of (combined) sampling procedures (as distinguished from the precision of analysis of laboratory duplicates) and environmental variability.

All samples for laboratory analyses will be delivered by hand to WSLH by USGS technicians. Records shall be kept that describe the number and type of samples included in the delivery, sampling sites information, and technician's initials.

Samples received for laboratory analysis will be checked in by a WSLH analyst. If data or samples are lost, duplicated, overdue, or otherwise mishandled, appropriate corrective action shall be taken and documented with initials of analysts checking in the samples.

Equipment Calibration/Maintenance

Laboratory calibration and maintenance will be done according the quality assurance manual and relevant SOPs. Analytical instrumentation shall be calibrated prior to use. Calibration shall be checked periodically during use. Any Analytical instrumentation calibration, calibration verification, and calibration checks that do not meet the criteria established in the SOP shall be documented in the QC report that accompanies the results.

Field equipment is minimal and includes sampling tubes, hand trowels, 60-m measuring tapes, and meter sticks. Sampling equipment will be washed, rinsed with deionized water, dried, and stored in plastic bags and bins between sampling outings. Stream measurement gear will be washed with detergent and rinsed thoroughly between outings. Locations identified with hand-held global positioning systems will be checked against 2015 aerial photography.

Standard Operation Procedure (SOPs)

General procedures for field methods are documented in Gellis et al. (2016) (https://cfpub.epa.gov/si/si_public_record_report.cfm?dirEntryId=335394). Specific standard operating procedures for site selection and sediment sampling are described in:

SOP.Sediment.fingerprinting_Site.selection_20170310.pdf

SOP.Sediment.fingerprinting_Sediment sampling_20170310.pdf

Laboratory SOPs document analytical procedures that are performed routinely according to EPA standard operating procedures development guidelines. The WSLH SOPs for this study include:

WSLH ESS INO IOP 550.0 rev. 6).

ESS INO Method 420.0 rev. 1

ESS INO METHOD 355.1, Rev. 2, Particle Size

ESS INO METHOD 355.1, Rev. 2, March 2011

ESS INO IOP 550.0 rev 6_Milestone Micrc

ESS INO METHOD 420.0 rev 1_High Res

QA/QC Checks

QA/QC checks shall be performed as defined in applicable SOPs and the laboratory's quality manual. Standard reference materials (SRMS) from EPA or other commercial sources are used to the extent feasible to evaluate method bias and variability. For each analysis, the analyst shall review results for QC checks performed to determine compliance with acceptance criteria specified in the SOPs (table 2-2). If prescribed acceptance criteria are not met, the analysis is discontinued, and the analyst shall perform corrective action as required by SOPs. Definitions of QC duplicate (or replicate) sample types include the following:

If both concentrations are below the detection limit, the duplicate range will not be recorded on the control chart. The duplicate range will be determined on samples that have a concentration above the detection limit of the procedure. If only one of the concentrations is below the detection limit, then the duplicate range will be calculated by using half the value of the detection limit as the concentration of the sample below detection limit. This value will be subtracted from the concentration value which is above the detection limit to obtain the range. The difference in concentration between the two readings of the same sample will be recorded on the QC report for each parameter.

Detection limits for each element as well as acceptance criteria for precision and accuracy are:

UW-HRICPMS_Element_QCmetricsDEC062

Samples affected by quality control samples with results outside of the laboratory established limits will be qualified. The investigator will consider the performance criteria for duplicate measurements listed in table 2-3 in usability determinations.

Table 2-3. Duplicate or replicate measurement performance criteria.

Concentration range	Acceptable comparability in environmental duplicate or replicate samples
Greater than two orders of magnitude above the method detection level	± 10 percent
One to two orders of magnitude above the method detection level	± 25 percent
Less than one order of magnitude above the method detection level	± 50 percent

Laboratory Audits

The WSLH analyzes performance testing samples semi-annually based upon unknown reference samples obtained from the USGS Branch of Quality Assurance. Internal reference samples area analyzed quarterly. The laboratory's quality system is audited. A complete system audit of the WSLH may be conducted by the USGS Branch of Quality Assurance.

2.6 Inspection / Acceptance of Supplies and Consumables

USGS personnel are responsible for assuring that adequate supplies are on site during the sampling. The USGS will ensure that all sample containers and equipment are inspected by the sampling team leader to ensure that the quality of the samples is not compromised during any part of the sample collection, preparation, or delivery process.

2.7 Non-Direct Measurements

Non-direct measurement data gathered during preliminary investigations include government maps, files, and databases; geographic information systems; historical information such as old newspaper articles, journal articles, city directories, and interviews with local citizens. All non-measurement data considered useful to the project will be collected by the USGS. Information may include published digital data sets describing land cover, soils, topographic data, and watershed boundaries for the sites identified in section 1.5.

2.8 Data Management

Original data and lab reports shall be maintained by the lab in accordance with established record retention practices (at least 10 years). Electronic copies are stored on multiple media (compact disk, lab server, LIMs). All analytical results initially are stored in the Laboratory Information Management system

(LIMs) data base that will be transferred to the WDNR's SWIMS system according to established procedures. If the results are not transferred through the LDES system, at a minimum, the reports will be attached as documents to the project record. Reasonable efforts will be made to upload the data into SWIMS by arrangement with DNR's laboratory liaison. Data that do not fit into USGS or WDNR's databases will be published in the USGS Sciencebase.

2.9 Assessments and Response Actions

Prior to delivery, USGS field sampling coordinator will verify COC paperwork and ensure that samples are labeled and packed properly. The WSLH lab analyst will verify that samples were inspected immediately upon receipt by the WSLH analyst to identify any problems that may exist with sample collection, handling. If any blanks or other QA/QC samples are found to be contaminated, re-runs will be requested to check for errors in laboratory data reporting, analysis, or other causes of the problem. Any field or laboratory problems that cause erroneous data to be generated will be identified and corrected as soon as possible. The lab analyst will verify QC sample results against SOP criteria, and take corrective action as defined in the SOP/method for out of control results.

3.0 Data Validation and Usability

3.1 Data Reduction and Reporting

The USGS is responsible for reporting all data, including a description of the field investigation, experimental methods, any observations of note, and the analytical data resulting from all of the analyses described in this QAPP. The analytical data will be presented in tabular form unless otherwise noted.

The final report will be a narrative prepared in draft and then in final after submittal to the USGS review and approval process. The report and other products from the project also will be reviewed by WDNR prior to publication or public presentation of results. Project updates and data overview reports will be provided at least quarterly. The data reports may include all data tabulated in Microsoft® Excel or Word formats, and will be provided in print and electronic formats. Interpretive figures also may be provided. The data reports will not include narratives of the methods and results, which will be provided in the final report only. Statistical analysis may be provided using SAS® software.

3.2 Data Review, Verification, and Validation

The USGS Project Manager will inspect all data, including project field replicates, as it is returned from the WSLH.

3.3 Verification and Validation Methods

Procedures to evaluate field data for this project primarily include checking for transcription errors and review of data sheets and data entry.

Personnel at WSLH will verify that all data have correct units and the correct number of significant figures. Quality control checks for each analysis will be reviewed by WSLH prior to data release. Any data that fails to meet internal laboratory criteria may cause the need for samples to be reanalyzed after any appropriate remediation in sample handling, equipment calibration, or equipment operation is made. Data associated with quality control failures will be qualified in accordance with current laboratory practices.

Results of field QC data will be reported along with other sample analysis data. Data will be flagged and qualified according to table 2-3. If warranted, retained samples may be submitted for additional analyses.

3.4 Reconciliation with User Requirements

Several statistical procedures are used to identify the optimum set of fingerprint properties that will be used in the final composite fingerprint to distinguish the potential sources and establish their relative contribution to the sediment flux at the watershed outlet (Gellis et al., 2016). The aim is to identify those properties that clearly discriminate the potential sources and to select a small subset of these properties that optimizes the discrimination provided by the composite fingerprint. These statistical procedures are outlined in figure 2-1.

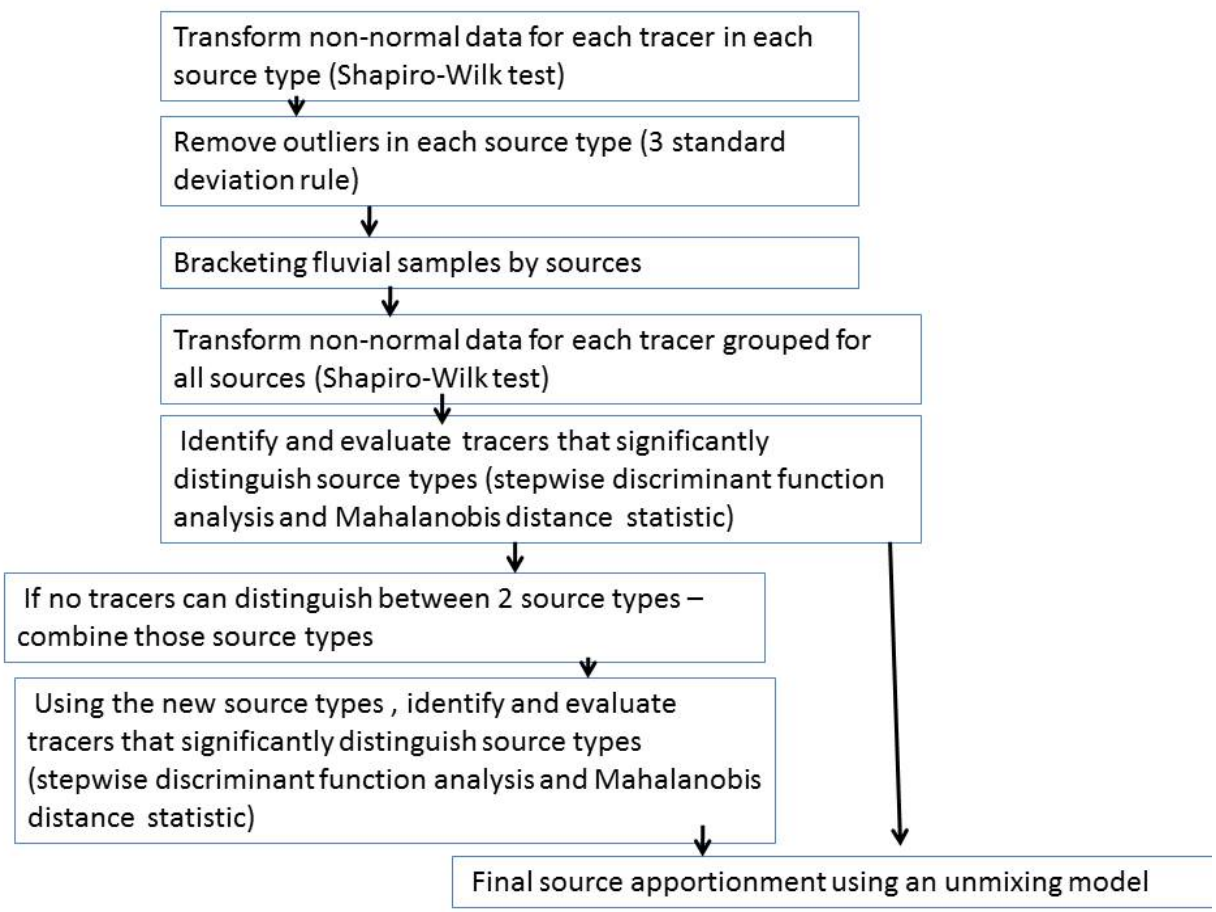

Figure 2-1. Statistical steps used in sediment-source analysis (Gellis et al., 2016).

The sediment-fingerprinting approach provides a quantification of the sources of sediment but it does not provide quantification of the rate of erosion or target 'hotspots' of erosion. For example, sediment fingerprinting may indicate that the streambanks are a major source of sediment in a watershed, but areas with the highest rates of bank erosion cannot be identified in the sediment fingerprinting approach. To determine specific erosion rates of given sediment sources and locations, a sediment budget approach is used (Reid and Dunne, 1996). A sediment budget is an accounting of the sources, storage, transport, and delivery of sediment in a watershed and can be presented as an equation:

$$I \pm \Delta S = O$$

Where I is the sediment input, ΔS is the change in sediment storage and O is the sediment output.

For the sediment budget approach, a variety of techniques used in other Wisconsin watersheds will be applied (Fitzpatrick et al., 1999; 2015). For Plum Creek, upland (land cover) erosion rates have been estimated through a SWAT model for the TMDL process (Cadmus, 2012). Bank erosion will be based on the eroding bank inventories completed along the main stem of Plum Creek by Outagamie County. Actively eroding gullies and ravines will be identified in this study through river walks and additional field inventories of headwater channels. The river walks and inventories will also include estimates of channel incision and soft sediment deposition in channels. Approximately 30 reaches along the Plum Creek stream network will have rapid channel assessments (Fitzpatrick et al., 2006; 2016). Channel corridor elements of bank erosion and gullying are budgeted by multiplying the field-measured areas of erosion by an estimated rate based on key indicators (Natural Resources Conservation Service, 2003; Natural Resources Conservation Service, 2002). Combining the sediment-fingerprinting and sediment-budget approaches provides a valuable and defensible tool to identify the significant sources of watershed derived fine-grained sediment. The 'hot spots' that result from the sediment budget approach are useful for targeting management actions to reduce erosion and can be displayed as maps.

Products from this study will include GIS maps and spreadsheets of stream corridor sources and sinks of sediment and TP. GIS maps and other data that does not fit into a data base will be published in USGS Sciencebase (https://www.sciencebase.gov/catalog/). A powerpoint presentation will be given and made available to project partners and local watershed groups. A journal article will be published with the results (journal to be selected with input from partners). The peer review process of a journal article includes a USGS selected reviewer and USGS approval prior to approval by the journal.

Data and associated analysis results generated by this project will directly support the WDNR's effort to identify sediment sources in Plum Creek. The results from this study are specific to Plum Creek. Caution must be used when applying the results to other nearby watersheds in the lower Fox River Basin with different physiography, soils, geomorphology, and land use.

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State of Wisconsin
Laboratory of Hygiene

Test Request

Billing and Reporting

Account Number **GL048**	Field Number (Bottle Label ID)	Report to Address (Non-DNR only) **USGS**		
DNR User ID	Report To Name **Faith Fitzpatrick**	City Middleton	State WI	ZIP
Date Results Needed (mm/dd/yyyy)		Report to Email (Non-DNR only)		

Date and Time of Sample Collection

Date (mm/dd/yyyy)	Time (24-hr clock)	End Date (mm/dd/yyyy)	End Time

Sample Type

SU Surface Water	NP Storm Water	EF Effluent (Treated Wastewater)	IF Influent (Untreated wastewater)
D Public Drinking Water SL Sludge	MW Monitoring Well SO Soil	PO Private Well TI Tissue	SE Sediment

Sample Type:
(select one)

Who collected the sample

Collected By Name	Telephone	Email

Where the sample was collected

Station ID (STORET #)	Sample Address or Location Description	
County **45**-Outagamie	Waterbody ID (WBIC)	Point / Outfall (or SWIMS Fieldwork Seq No)

Sample Details

Sample Description / Device Description

Enforcement? **No** If yes, include chain of custody form.	If Field QC Sample (select one): Duplicate Blank	Depth of Sample: ft m in cm Or Top and Bottom of Sample Interval: - ft m in cm
Is Sample Disinfected? **No** If yes, how?	Grant or Project Number **GBF1603_PLUMCREEK**	

Analyses Requested

A (No A Code) SF-ICPMS Multiple Elements
B IC49602 Particle Size/Sieve
C IC52009 Phosphorus, Solid ICP

Note that sample has been sieved to <63 um.