

# Flint, Michigan Sampling Support Project

## Drinking Water Lead and Water Quality Sampling Quality Assurance Project Plan (QAPP)

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**U.S. Environmental Protection Agency**  
**Conducted by: Region 5 Ground Water and Drinking Water Branch**  
**Chicago, Illinois**

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## List of Acronyms

ATSDR	Agency for Toxic Substances and Disease Registry
CCT	Corrosion Control Treatment
CRL	Chicago Regional Laboratory
DBP	Disinfection Byproducts
<i>E. coli</i>	<i>Escherichia coli</i>
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
GPS	Global Positioning System
GWDWB	Ground Water and Drinking Water Branch
HAA9	Nine Haloacetic Acids
HDPE	high density polyethylene
H <sub>2</sub> SO <sub>4</sub>	sulfuric acid
HNO <sub>3</sub>	nitric acid
KWA	Karegnondi Water Authority
LDPE	low density polyethylene
LIMS	Laboratory Information Management System
LSL	Lead Service Line
MDEQ	Michigan Department of Environmental Quality
MDHHS	Michigan Department of Health and Human Services
µg/L	micrograms per liter
mL	milliliter
MF	minute flush
MS	minute stagnation
NPDWR	National Primary Drinking Water Regulations
NSF	NSF International
ORD	Office of Research and Development
PVC	polyvinyl chloride
RL	Reporting Limit
SOP	Standard Operating Procedure
SU	Standard Unit
Technical Support Team	Flint Drinking Water Technical Support Team
TDS	total dissolved solids
TTHM	Total Trihalomethanes
QA	Quality Assurance
QAPP	Quality Assurance Project Plan

## 2. Distribution List

On October 16, 2015, U.S. Environmental Protection Agency (EPA) established the Flint Safe Drinking Water Task Force, which is now known as the Flint Drinking Water Technical Support Team (Technical Support Team) to provide the Agency’s technical expertise through regular dialogue with designated officials from Michigan Department of Environmental Quality and the City of Flint. Key Points of Contact at EPA, City of Flint, and Michigan Department of Environmental Quality (MDEQ) are listed below. In consultation with the Technical Support Team, EPA’s Water Division and EPA’s Office of Research and Development (ORD) may identify additional contacts for inclusion in this distribution list.

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### 3. Project Organization

The Technical Support Team is responsible for overseeing the provision of technical assistance to the City of Flint.

The EPA Water Division is responsible for project development, oversight, and implementation in coordination with the Technical Support Team, MDEQ, City of Flint, EPA and contract laboratories, and EPA ORD. This includes oversight of all sampling projects, selection of sampling locations, training for water sampling activities, coordination of sampling containers and return shipment, and preparation of the sampling summary reports and final project reports. The EPA Water Division is also responsible for maintaining the documents listed under Project Officer in Section 14 and any follow-up documentation, as well as distributing copies of the final EPA sampling results to all relevant parties (see Section 2) and posting results on EPA's Flint Drinking Water Response website.

The Chicago Regional Laboratory (CRL) Director is responsible for overseeing the provision of sampling supplies and coordinating laboratory analyses to be performed at CRL and in other EPA regional laboratories. This includes oversight of all EPA CRL quality assurance requirements, including data review and qualification of the data. The CRL Director is also responsible for providing the Laboratory Data Reports (analytical results) to the Technical Contacts in the EPA Water Division and maintaining the documents listed under EPA CRL in Section 14.

Other EPA Laboratories and contract laboratories may be identified for support laboratory analytical services. It is currently anticipated that Region 1, Region 4, and Region 10 as well as the PHILIS Contract Laboratory (e.g., TestAmerica Laboratories, Inc.), will provide analytical services under this Quality Assurance Project Plan (QAPP). In addition, Region 7 was involved in the laboratory analytical services prior to March 2016. Each laboratory contract program manager is responsible for overseeing the provision of sampling supplies and coordinating laboratory analyses to be performed in EPA laboratories. Each EPA laboratory director or laboratory contract program manager will be responsible for oversight of the applicable laboratory quality assurance requirements, including data review and qualification of the data. Each EPA laboratory director or laboratory contract program manager is also responsible for providing the Laboratory Data Report (analytical results) to the Technical Contacts in the EPA Water Division and maintaining the documents listed under Other Laboratories in Section 14.

The EPA Water Team Field Leads are responsible for coordinating with and providing technical assistance and training to field sampling coordinators for all sampling activities, including consumable supplies and calibration procedures.

Field sampling coordinators, either EPA personnel or other personnel trained in sampling techniques and under the oversight of EPA, are responsible for field implementation of water sampling activities, including coordination with residents involved in sampling activities, completing applicable sampling activities, conducting field measurements and field preservation, verifying the completeness of field sampling forms and chains-of-custody, and shipping samples for laboratory analysis, as described in this QAPP.

The EPA Water Division Technical Contact(s), in coordination with the Technical Support Team, are responsible for amending the QAPP and sampling instructions, as required based on analytical results and field observations. The EPA Water Division Technical Contact(s) are responsible for assisting with data management, interpreting the sampling results, and evaluating the need for further action.

#### **4. Background**

Lead is a toxic metal that can be harmful to human health when ingested. Young children are particularly sensitive to the effects of lead because their bodies are still undergoing development. Lead can be released into drinking water primarily from interaction of the water with plumbing materials containing lead through corrosion, as well as physical plumbing disturbances and changes to water chemistry. Common sources of lead in drinking water include lead service lines (LSL), as well as lead solder, fluxes, other pipes and pipe fittings, fixtures, and sediment within the water distribution network.

Drinking water treatment and distribution systems can be complex, involving many different chemical, physical, and microbial processes that can directly affect water quality. Drinking water rules and regulations are based on contaminant levels found in the source water as well as contaminants that can enter the water distribution system or increase within the distribution system. Monitoring in a public water system is conducted at the source, within the treatment plant, and across the distribution system. Treatment processes must be adjusted based on water quality changes in the source water, which can affect the performance of various treatment processes. Water quality changes in the distribution system can directly impact microbial activity; corrosion of metals such as lead, copper, and iron; formation of disinfection byproducts; taste and odor of the water; and physical appearance of the water. In many cases, water utilities add chemicals to their water, such as corrosion inhibitors and disinfectants, the fate and subsequent effectiveness of which can change in the distribution system. Optimal Corrosion Control Treatment (CCT) is defined as treatment that minimizes lead and copper levels at users' taps while ensuring that the treatment does not cause the water system to violate any National Primary Drinking Water Regulations (NPDWR).

Scales, deposits, and sediment develop on and in all water distribution pipes. These materials may have widely different compositions depending on the type of metal that is corroding, the water chemistry, and the inhibitors that are added to the drinking water. Scales may have detrimental impacts on water distribution systems in numerous ways. For example, scales can provide sinks for toxic metals (e.g., copper and lead), microbes, or aesthetically problematic constituents (e.g., manganese, iron, calcium, and zinc) via sorption, precipitation, or corrosion product growth. These metals or constituents can be released because of subsequent water quality changes or physical/hydraulic disturbances. However, scales in the distribution system can also protect water by reducing the rate of corrosion or metal release in drinking water. Scales also enable reductions in exposure to regulated metals, such as copper and lead.



## 5. Project Description

The EPA has offered, and the City of Flint has accepted, the assistance of EPA experts on corrosion and corrosion control treatment under current water quality conditions as well as during and after the upcoming transition to Flint's selected long-term water source(s).

EPA will work, in cooperation with local partners and voluntary residents/owners, to conduct drinking water testing in Flint including faucet filter evaluation, distribution system monitoring for disinfectant and disinfection byproducts (DBP), lead source/release diagnostic evaluation, health and direct contact exposure evaluation, aerator particulate evaluation, distribution system monitoring for treatment assessment, distribution system sampling for Coliform bacteria, resident-requested sampling and hot water evaluation, special purpose sampling, and lead service line (LSL) extraction and pipe scale evaluation, as discussed in the following sections. EPA sampling activities were completed during calendar year 2016, with the exception of the LSL Detection Study that is currently being conducted as described in Section 5.11 below.

Details regarding sampling are provided in Appendices A through H.

### 5.1 Faucet Filter Evaluation

Water samples will be collected to assess concentrations of lead in filtered and unfiltered drinking water at selected sampling sites, including homes where existing analytical data indicates elevated lead in water concentrations above 150 micrograms per liter ( $\mu\text{g/L}$ ). In general, three grab water samples will be collected at the kitchen faucet at each selected sampling site, as discussed below.

- Filtered Water, Existing Faucet Filter - One grab sample will be collected through the existing water filter (if present). The field sampling coordinators will note the type (brand) of the filter, status of the filter indicator, and available information from the resident regarding the time since the filter or cartridge was installed.
- Unfiltered Water - The filter will be removed, and an unfiltered water sample will be collected as the first grab sample following removal of the filter and/or aerator. No cleaning or flushing will be conducted prior to the unfiltered water grab sampling.
- Filtered Water, New Faucet Filter - Following the collection of the unfiltered sample, a new filter or new filter cartridge will be installed, and the water will be allowed to run through the new filter for approximately two minutes. Following installation and flushing of the new filter or replacement filter cartridge, a grab sample will be collected through the newly installed filter.

If no faucet filter is currently in place at the location, or if a faucet filter is unable to be installed, the field sampling coordinators will collect fewer samples and will document reasoning in the field notes. Samples will be collected in accordance with Section 8.1 and analyzed for total metals in accordance with Section 9.1. Specific procedures for faucet filter evaluation sampling are provided in Appendix A. Water samples will be sent to EPA or contract laboratories for analysis of total metals including lead.

Beginning in late March 2016, a modified procedure was implemented to include the collection of unfiltered samples following a minimum of 6 hours stagnation. When stagnation can be confirmed, two water samples will be collected as described below at each selected sampling site, generally at the kitchen faucet.

- Unfiltered Water - The filter will be removed (or bypassed), and an unfiltered water sample will be collected as a first flush sample following removal of the filter and/or aerator and a minimum of 6 hours stagnation. No cleaning or flushing will be conducted prior to the unfiltered water grab sampling.
- Filtered Water, Existing (or New) Faucet Filter - One grab sample will be collected through the existing water filter (if present). If no existing filter is present, field sampling coordinators will install and flush a new filter, as described above, then collect the filtered water sample through the newly-installed water filter. The field sampling coordinators will note the type (brand) of the filter, status of the filter indicator, and available information from the resident regarding the time since the filter or cartridge was installed.

The results of the filtered and unfiltered grab samples will be evaluated to assess whether the NSF International (NSF)-certified household filters being distributed are effective for removal of lead above the NSF-designated criteria of 150 µg/L. The total number of sampling sites for this task will be determined based on evaluation of available data and field conditions.

## **5.2 Distribution System Monitoring for Disinfectant and Disinfectant Byproducts**

Chlorine, pH, and temperature will be measured weekly or biweekly at up to 35 representative sites in the distribution system in accordance with the sampling plan developed by EPA ORD (see Appendix B).

Total trihalomethanes (TTHM) and nine haloacetic acids (HAA9) will be measured biweekly or monthly at up to 35 sites within the distribution system in accordance with the sampling plan developed by EPA ORD (see Appendix B).

Samples will be collected in accordance with Section 8.1 and analyzed for TTHMs and HAA9 in accordance with Section 9.1. Specific procedures for distribution system water quality monitoring for disinfectant and DBPs are provided in Appendix B. Field water quality parameters, including pH, temperature, and chlorine residual, will be measured and recorded in the field. All TTHM and HAA9 samples will be collected into certified clean, pre-preserved vials provided by the PHILIS Contract Laboratory, and field sampling coordinators will ensure all vials are collected with zero headspace. The TTHM and HAA9 samples will be sent to and analyzed by the PHILIS Contract Laboratory.

Distribution system water quality monitoring results will be used evaluate DBP formation and potential microbial contamination, including chlorine residual, TTHM, and HAA9 levels. The number and location of sites and the frequency of sampling may be adjusted based on the previous round(s) of sampling.

### **5.3 Lead Source/Release Diagnostic Evaluation**

Sequential sampling was conducted in January through March 2016 at more than 100 ‘candidate’ sampling sites selected based on available information including previous high lead concentration results, indicating the potential presence of LSLs. The results of the initial sampling from the candidate sampling sites, along with available plumbing information, has been used to identify and select approximately 45 single-family residences with a variety of plumbing materials, including sites which may have LSLs and sites which may not have LSLs, for evaluating differences in lead release from different potential sources of lead. Additional sequential sampling, plumbing investigations, and other work may be conducted to focus on specific issues, such as homes which likely do not have LSLs with results indicating have high lead sources potentially within the home plumbing.

Beginning in May 2016, the selected single-family residences with LSLs or significant interior sources of lead will be sampled approximately every two months for subsequent evaluation of corrosion control treatment and effectiveness. Procedures for subsequent rounds of sequential sampling may be tailored for each single-family residence, based on available information including analytical results and plumbing information. In addition, smaller bottle sizes (e.g., 500-mL) may be used to further evaluate potential sources of lead in interior plumbing.

Following at least 6 hours of stagnation, sequential samples will be collected in accordance with Section 8.1 and analyzed for total metals in accordance with Section 9.1. Specific procedures for lead source/release diagnostic evaluation sampling are provided in Appendix C. Water samples will be sent to EPA laboratories for analysis of total metals including lead. Tin will be added to the list of parameters to be analyzed starting with second round of sequential sampling in May 2016.

In November 2016, to assist with the effort to develop a sampling protocol to identify LSLs, additional fully-flushed distribution system water samples will be collected at selected locations known to have a full LSL or suspected of having a partial (public-side) LSL. These additional distribution samples will be collected at the end of the standard sequential sampling procedure, which is conducted with the faucet fully open (i.e., the faucet’s maximum flow rate). Two additional distribution system water samples will be collected, targeting two-thirds of the maximum flow and one-third of the maximum flow, to evaluate whether flow rate might be a factor to consider in the sampling framework for LSL identification. Flow rates will be measured and recorded. Additional details are provided in Appendix C-4.

Lead source/release diagnostic evaluation sampling results will be evaluated by the Technical Support Team so that any necessary corrosion control adjustments can be implemented. The total number of sampling events for this task will be determined based on evaluation of available data.

### **5.4 Health and Direct Contact Exposure Evaluation**

Water samples will be collected to assess concentrations of potential health concerns including direct contact exposure from identified potential irritants that may be present in drinking water. EPA will coordinate with Michigan Department of Health and Human Services (MDHHS) and Agency for Toxic Substances and Disease Registry (ATSDR) to collect grab water samples from

bathroom faucet(s), bathtub tap(s), shower head(s), and kitchen faucets at homes selected by MDHHS and ATSDR. The results of the grab water samples will be provided to MDHHS and ATSDR for evaluation of any health concerns or source(s) of skin irritants present in drinking water that is used for washing and bathing. The request letter from MDHHS is included in Appendix D; MDHHS subsequently clarified that mercury analysis is not needed.

Grab water samples will be analyzed for potential skin irritants and water quality parameters identified by MDHHS and ATSDR, including laboratory analysis for total metals (long list), total dissolved solids (TDS), alkalinity, sulfate, chloride, fluoride, and hardness and field measurement including pH, chlorine residual, turbidity, and conductivity. The following samples will be collected at each location at the discretion of the field sampling coordinators:

- Kitchen sink faucet – one set of cold water samples analyzed for total metals and water quality parameters including TDS, alkalinity, sulfate, chloride, fluoride, hardness, pH, chlorine residual, turbidity, and conductivity
- Bathroom sink faucet – one cold water sample analyzed for total metals
- Bathtub faucet – one cold water sample analyzed for total metals
- Shower faucet – one cold water sample analyzed for total metals
- Bathtub or Shower faucet (whichever is used more) – one set of hot water samples analyzed for total metals and water quality parameters including TDS, alkalinity, sulfate, chloride, fluoride, and hardness, pH, chlorine residual, turbidity, and conductivity

At the direction of MDHHS and ATSDR, turbidity, conductivity, and TDS were discontinued at the end of March 2016.

Specific procedures for sampling for health and direct contact exposure evaluation are provided in Appendix D-1. Grab samples should generally be collected after removing any aerator or faucet filter and bypassing existing whole house filter(s) and/or water softeners. If a faucet filter is currently in use at the sampling site, field sampling coordinators may collect additional filtered water sample(s) and document the sample collection methods in the field records. Field sampling coordinators will also record additional field observations including the presence and descriptions of any color, odor, and sediment or debris in the water from each tap sampled.

At the request of MDHHS and ATSDR, additional evaluation is being conducted in April 2016 at a limited number of homes (e.g., 5 homes with reported health issues and 5 “control” homes without health concerns). Water samples will be evaluated for an extended list of organic compounds including volatile organic compounds (VOC), semi-volatile organic compounds (SVOC), and DBPs to compare water quality at homes with reported health concerns in comparison to homes without health concerns. Cold water samples will be collected from bathtub faucets, following a minimum of 6 hours stagnation, and then hot water samples will be collected after running the hot water at the bathtub’s maximum setting for approximately one minute. Specific procedures for sampling for the limited “pilot” evaluation are provided in Appendix D-2.

Samples will be collected and analyzed in accordance with Sections 8.1 and Section 9.1, respectively. Specific procedures for sampling for health and direct contact exposure evaluation

are provided in Appendices D-1 and D-2. Field water quality parameters, including pH, turbidity, conductivity, and chlorine residual, will be measured and recorded in the field, and water samples will be sent to EPA or contract laboratories for analysis of the remaining parameters.

## **5.5 Aerator Particulate Evaluation**

During scheduled sampling events at selected locations with previously reported high lead results, field sampling coordinators may collect scale and debris that has accumulated on aerators and water filter screens. Solids samples will be collected at the discretion of field sampling coordinators and only if sufficient solid material is present behind the aerator and/or water filter screen at a given sampling location. Photographs will be taken onsite of the scale/debris on the aerators and screens, and field observations will be recorded. Aerator particulate sampling will generally be conducted while at a sampling site for other sampling activities under this QAPP.

Samples will be collected in accordance with Section 8.1 and analyzed for total metals in accordance with Section 9.1. Specific procedures for aerator particulate evaluation sampling are provided in Appendix E. Solid samples will be sent to the PHILIS Contract Laboratory for analysis of total metals including lead.

Aerator particulate analytical results will be evaluated and used to produce educational material to reinforce the need for residents to periodically clean their aerators. The total number of sampling locations for this task will be determined based on evaluation of available data. This sampling effort will continue until sufficient information has been obtained to produce educational material.

## **5.6 Distribution System Monitoring for Treatment Assessment**

Information regarding the water quality in the distribution system will be collected to evaluate the stability of the water quality throughout the distribution system, and to detect locations that may have the highest risk of lead release or the presence of microbial contamination.

Distribution system water quality assessment sampling may be conducted at sampling sites after the completion of other sampling activities as specified below.

- Field water quality parameters, including pH and chlorine residual, will be measured following grab sampling, including sampling faucet filter evaluation (Section 5.1), health and direct contact exposure evaluation (Section 5.4), and resident-requested sampling (Section 5.8) and following sequential sampling for lead source/release diagnostic evaluation (Section 5.3).
- Water samples for water quality parameters including total phosphorous, alkalinity, chloride, sulfate, and fluoride will be collected following sequential sampling for lead source/release diagnostic evaluation (Section 5.3).

Distribution system water quality sampling will be conducted following at least 5 minutes of flushing, depending on the size and length of plumbing between the sampling location and the water distribution main. Field water quality parameters, including pH and chlorine residual, will be measured and recorded in the field, and water samples will be sent to EPA CRL for laboratory

analysis of the remaining water quality parameters. Samples will be collected and analyzed in accordance with Section 8.1 and Section 9.1, respectively. Specific procedures for distribution system water quality monitoring for treatment assessment are provided in Appendix F.

Distribution system water quality characterization assessment results will be used to evaluate:

- corrosion control, including range of pH, alkalinity, total phosphorus, and anions (sulfate, chloride, fluoride) and
- potential for microbial contamination, including chlorine residual.

Data collected on water quality parameters that affect corrosion control can be used along with metals data for lead and copper to set “optimal water quality control parameters”, as required by 141.82(f). The total number of sampling sites for this task will be determined based on evaluation of available data and field conditions.

### **5.7 Distribution System Sampling for Coliform Bacteria**

Samples may be collected to assess the presence of Coliform bacteria in drinking water at locations where field monitoring conducted under this QAPP (see Section 5.2 and Section 5.6) indicates low chlorine residual. Approximately 0.05 mg/L to 0.2 mg/L has been used as a “low chlorine residual” threshold for collecting water samples for analysis of Total Coliforms and *E. Coli*.

EPA will coordinate with Genesee County Health Department or MDHHS to obtain sterile sampling containers, conduct this sampling, and analyze the samples for bacterial indicators including Total Coliform and *Escherichia coli* (*E. coli*). Samples will be couriered to a partner laboratory such as Genesee County Health Department or MDHHS to analyze these samples using Colilert analytical methods. Specific procedures for sampling for Coliform bacteria are provided in Appendix G.

### **5.8 Resident-Requested Sampling and Hot Water Evaluation**

Water samples may be collected at selected sampling sites at the request of residents. In general, two grab water samples will be collected at the kitchen faucet at each selected sampling site, as discussed below.

- Unfiltered Cold Water – After at least 6 hours of stagnation, an unfiltered first-draw cold water sample will be collected. Any faucet filter or aerator will be removed prior to sampling, and any whole house water treatment will be bypassed.
- Unfiltered Hot Water – After the cold water sample is collected, the hot water tap will run until the water is hot and then an unfiltered hot water sample will be collected.

While on-site, the field sampling coordinators will collect information regarding the hot water tank and the resident’s willingness to participate in hot water evaluation sampling from their water tank. At selected locations, field sampling coordinators may return to collect hot water samples from the hot water tank drain, to assess the lead levels in hot water tanks.

Samples will be collected in accordance with Section 8.1 and analyzed in accordance with Section 9.1. Specific procedures for resident-requested sampling and hot water evaluation sampling are provided in Appendix H. Water samples will be sent to EPA laboratories for analysis of total metals, including lead.

Hot water sampling results will be evaluated and used to produce educational material to reinforce the need for residents not to use hot water for human consumption (e.g., making baby formula and cooking). The total number of sampling locations for this task will be determined based on evaluation of available data. This sampling effort will continue until sufficient information has been obtained to produce educational material.

## **5.9 Special Purpose Sampling**

At the discretion of the field sampling team, additional field measurements for chlorine, and possibly sampling for Total Coliform and *E. coli*, will be conducted, based on requests from the community regarding concerns over the water quality in their area. Depending on the nature of the request, sampling may target water within the premise plumbing or water representative of that delivered by the water utility (water main sampling).

The difference between premise plumbing and water main sampling is flushing time. If flushing is conducted for water main sampling, times will be adjusted based on site-specific characteristics (i.e., service line pipe diameter, distance from sample tap to the water main, tap flowrate, etc.) to ensure that measurement captures water from within the water main. The field sampling team will note whether or not flushing occurred, how long, and if determined, the approximate distances and pipe sizes as well as other pertinent information used to determine the flushing time(s) for the samples.

Beginning in late June 2016, following the completion of the faucet filter evaluation study, a limited number of resident-requested samples have been collected; these have been grab water samples collected from one or more faucets, generally limited to unfiltered water. These 1,000-mL resident-requested water samples are labeled with the prefix “W”, field preserved (see Section 8.1), and analyzed for total metals including lead.

In late August 2016, a limited number of filtered and unfiltered distribution system water samples were collected to evaluate whether orthophosphate (as total phosphorus) is removed by faucet (point-of use) filters. Sample locations were selected in cooperation with MDEQ and EPA ORD. These distribution system water samples were collected following flushing for a site-specific volume approximately equal to the volume of premise and service line plumbing between the sampled faucet and the distribution main. Following flushing, an unfiltered water sample was collected with no filter or aerator in place, then the filter was replaced and a filtered water sample was collected. These 125-mL water samples were labeled with the prefix “FP”, field preserved (see Section 8.1), and are analyzed for total phosphorus.

## **5.10 Lead Service Line Extraction and Pipe Scale Evaluation**

In order to inform corrosion control treatment optimization decisions, segments from four LSLs will be extracted and used for assessing the current condition and composition of the scales

within the LSLs. Pipe scale analyses on segments of these pipes will be used to determine the type of scales that have formed inside the lead lines under different chlorine levels and water quality conditions.

Four full LSLs will be identified by EPA ORD and the Technical Support Team for initial extraction, including two sites in low chlorine areas and two sites in high chlorine areas. Two 4-foot segments from each LSL will be carefully extracted, packed and shipped to EPA ORD for scale analyses. If possible, the extracted segments should be driven back to EPA ORD to avoid physical disturbances during shipping. Depending on the suitability and condition of the extracted LSLs, additional LSLs may need to be extracted using the procedures outlined in this section. Specific procedures for LSL extraction and pipe scale evaluation, including a copy of the EPA ORD Pipe Scale Analysis and Evaluation: Quality Management Plan for Research at the Advanced Photon Source of the Argonne National Laboratory, are provided in Appendix I.

Sequential sampling may be conducted at selected LSL extraction locations, both prior to and following LSL extraction, for evaluation of the effects of LSL removal on drinking water quality. Sequential sampling procedures are provided in Appendix C.

### **5.11 Lead Service Line Detection Protocol**

The Technical Support Team has identified that the development of a protocol for LSL identification is necessary to reduce lead release and unhealthy exposure from the piping. A protocol for LSL identification will be developed for the Flint water distribution system using home drinking water sampling results from two cohorts of residential settings: (1) 10 to 15 homes with known full or partial LSLs, and (2) 10 to 15 homes that have never had a LSL. A previous study<sup>1</sup> was developed and validated such a protocol for a district in Montreal, Canada. However, that protocol is valid only for the unique distribution of plumbing configurations and signature lead concentrations specific to Montreal. This task will develop a protocol for direct application for LSL identification in the Flint drinking water system and methods will be identified to customize the protocol for application in other drinking water systems after the gathering of distribution-specific data.

The distribution system-specific water sampling will evaluate a series of flushed samples collected using various flow rates (high, medium, and low) and sequential samples from the kitchen tap of homes in each cohort. Samples will be collected in accordance with Section 8.1 and Appendix C-5 (Lead Service Line Detection Protocol). All samples will be collected by passing existing point of use filters. Field measurements will include pH, free chlorine, and temperature. Samples will be analyzed for lead, copper, and zinc; additional analytes (e.g., anions, iron) or field measurements may be identified based on initial sampling results. It is anticipated that up to 900 individual samples will be collected. Field and laboratory data for the LSL Detection Study will be collected, reviewed, and stored by ORD and its contractor (Battelle) to ensure data completeness and quality consistent with Sections 12 through 14.

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<sup>1</sup> Cartier, C., A. Bannier, M. Pirog, S. Nour and M. Prevost (2012). "A Rapid Method for Lead Service Line Detection." Journal - American Water Works Association: 596-607.



Sampling profiles will then be developed to determine drinking water lead concentration cutoff levels that can be used to identify the presence of LSLs under conditions of full LSL, partial LSL, and possible presence of interior plumbing lead sources that may contribute to lead levels in drinking water at the tap.

As part of this project, the protocol may be used at one or more additional cities (e.g., Galesburg, IL) to assess transferability and variability in LSL detection threshold(s).

## **6. Quality Objectives**

### **6.1 Precision**

EPA and contract laboratories will perform replicate analysis of known samples to assess method precision. The acceptance criterion for replicate analysis is a maximum of 20 percent (%) Relative Percent Difference, unless otherwise specified by EPA or contract laboratory standard operating procedures (SOP).

### **6.2 Bias**

EPA and contract laboratories will perform analysis of Laboratory Fortified Blanks to assess accuracy/bias. The acceptance criterion for accuracy for the results is to be within plus or minus 10% recovery of the known value, unless otherwise specified by EPA or contract laboratory SOPs.

### **6.3 Representativeness**

The selection of sample locations, analyses, and sample sizes is designed to identify potential sources of lead throughout the plumbing network at each sampling site, and to collect samples that are representative of concentrations of lead and other water quality parameters in City of Flint drinking water.

### **6.4 Comparability**

The analytical methodology for this project is standard analytical methodology used to assess concentrations of lead and other water quality parameters, as described in Section 9.1, in City of Flint drinking water.

Sampling results for sampling activities in this QAPP (see Section 5) are intended to identify appropriate sampling sites, characterize the water quality throughout the distribution system, assess potential risk associated with various water quality and plumbing materials, and to monitor the effectiveness of corrosion control treatment. The sampling results for these sampling activities are not intended to be used to assess compliance with the lead and copper action levels in EPA's Lead and Copper Rule.

## 6.5 Completeness

Samples will be collected from specified sampling locations, with the goal that one hundred percent (100%) of samples will be analyzed and reported. However, some samples may not generate valid data, and additional sampling may be conducted as needed to meet overall objectives.

## 6.6 Sensitivity

EPA and contract laboratory reporting limits (RL) for the determination of metals and water quality parameters (see Section 9.1 and Appendix J) meet the objectives of this project.

## 7. Non-Direct Measurement (Secondary Data)

There are no non-direct measurements associated with this project. However, secondary data previously collected and reported may be used to identify sampling sites.

## 8. Field Monitoring Requirements

This section includes general procedures for water sampling methods, field water quality testing, and field quality control. Details regarding sampling and field water quality testing are described in Appendices A through H.

### 8.1 Water Sampling Methods

The following equipment and supplies will be needed to perform field sampling activities:

- HDPE or LDPE pre-certified clean wide-mouth 125-milliliter (mL), 500-mL, and 1,000-mL single use rigid plastic bottles for all metals analysis (preserved to pH<2 with nitric acid HNO<sub>3</sub>),
- Pre-certified clean single use sampling containers for other laboratory analyses as determined and provided by EPA CRL or the contract laboratory (method-specific)
  - 125-mL HDPE or LDPE bottles for analysis of total phosphorus (field preserved to pH<2 with sulfuric acid H<sub>2</sub>SO<sub>4</sub>)
  - 125-mL HDPE or LDPE bottles for analysis of alkalinity and anions (chloride, sulfate, and fluoride)
  - 125-mL HDPE or LDPE bottles for analysis of TDS
  - 40-mL glass VOA vials pre-preserved with sodium thiosulfate for analysis of TTHMs (three vials per sample)
  - 250-mL amber glass bottles pre-preserved with ammonium chloride for analysis of HAA9
  - 40-mL glass VOA vials, Teflon-capped, ascorbic acid pre-dosed for analysis of VOCs including THMs (three vials per sample, field-preserved with hydrochloric acid and zero headspace)

- 60-mL glass vials, Teflon-capped, ammonium chloride and phosphate buffer pre-dosed for analysis of DBPs General List (three vials per sample, field-preserved with zero headspace)
- 60-mL glass vials, Teflon-capped, sodium sulfite and phosphate buffer pre-dosed for analysis of Chloral Hydrate (two vials per sample, field-preserved with zero headspace)
- 1000-mL amber glass bottles, sodium sulfite crystals pre-dosed for analysis of SVOCs (two per sample)
- Trip blanks for analysis of TTHMs and VOCs
- pH meter including calibration solutions and maintenance equipment,
- Conductivity meter including calibration solutions and maintenance equipment,
- Turbidimeter including calibration solutions and maintenance equipment,
- Thermometer,
- Colorimetric kits for chlorine residual measurement,
- Weatherproof labels,
- Chain-of-custody forms (see Appendix J),
- Sturdy coolers and packing materials (e.g., absorbent material, bubble wrap, trash bags, zip-type plastic bags, shipping tape, and shipping labels),
- Bound field logbook or electronic field note recorder (e.g., iPad with iForms software),
- Photograph labels (e.g., dry erasable board or sheet of paper),
- Global Positioning System (GPS) hand-held locator (optional),
- Indelible ink pen/marker, and
- Camera.

Field sampling coordinators, either EPA personnel or other personnel trained in sampling techniques and under the oversight of EPA, will conduct sampling as summarized below. The field sampling coordinators will prepare field records, chains-of-custody, and sampling labels, conduct any field measurements or field preservation, and ask the residents to verify any stagnation or pre-flushing procedures were followed.

- Each water sample is to be collected from the sample tap outlet directly into the sampling container provided by the laboratory.
- The sampler will follow the protocols provided in Appendices A through H, ensuring that samples are collected in accordance with sampling instructions and training.
- If allowed by the resident, a label with the sample ID written in indelible marker may be placed on the underside of the sampling fixture, in the event the local partner has to re-visit the sampling site. If possible, a photograph may be taken of the sampling fixture (i.e., kitchen faucet); the unique sample ID, date, and sampler should be written on a dry-erase board or sheet of paper and placed in the view for identification of any photograph(s).
- A chain-of-custody form will be filled out to include all samples to be shipped together (i.e., one form per cooler), including sample times and unique sample IDs (nomenclature as described in Appendices A through H).

- Samples will be packaged and shipped overnight or couriered to the applicable EPA or contract laboratory. Whenever possible, overnight shipment will be on the same day as sampling is completed.

Field sampling coordinators will preserve metals samples with nitric acid (HNO<sub>3</sub>) to a pH of 2 standard units (SU) or less in the field, prior to shipment to the EPA laboratory. EPA or contract laboratories will check and add additional HNO<sub>3</sub>, if needed, within 48 hours of sample receipt. The analytical method allows samples to be preserved within 14 days from sample collection.

Additional details on specific sampling methods are provided in Appendices A through H. A summary of sampling activities is provided in the table below.

Sample Objective	Sample ID Type	Laboratory Analyses and Approximate Number of Samples per Site Visit										Field Analyses				
		Total Metals (standard list -see note 1)	Total Metals (full list -see note 2)	Total Phosphorus	Total Alkalinity	Anions (see note 3)	TTHMs	HAA9	TDS	Hardness	Total Coliform / E. coli	Chlorine Residual	pH	Temperature	Turbidity	Conductivity
Faucet Filter Evaluation	"FG--", "FGW--", "FGC" and "FH--"	2 to 3	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Distribution System Monitoring for Disinfectant and DBPs	"T--" and "H--"	NA	NA	NA	NA	NA	1	1	NA	NA	5 to 1*	1	1	1	NA	NA
Lead Source/Release Diagnostic Evaluation	"S--" and "DS--"	12 to 30	NA	1	1	1	NA	NA	NA	NA	5 to 1*	1*	1*	NA	NA	NA
Health and Direct Contact Exposure Evaluation	"R--"	NA	3 to 6	NA	1	1	NA	NA	1**	1	5 to 1*	1	1	NA	1**	1**
Aerator Particulate Evaluation	"--AP"	1	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Distribution System Monitoring for Treatment Assessment	Field and "DS--"	NA	NA	1	1	1	NA	NA	NA	NA	5 to 1*	1	1	NA	NA	NA
Distribution System Sampling for Coliform Bacteria	Address	NA	NA	NA	NA	NA	NA	NA	NA	NA	1	NA	NA	NA	NA	NA
Resident-Requested Sampling and Hot Water Evaluation	"G--"	2 to 3	NA	NA	NA	NA	NA	NA	NA	NA	5 to 1*	1*	1*	NA	NA	NA
Special Purpose Sampling	TBD															

Notes:

\* Field measurements for chlorine residual and pH will generally be collected at each sampling location following other sampling objectives and additional flushing, as needed. Samples for Coliform bacteria may be collected if low chlorine residual is observed in distribution system (fully flushed) samples collected at individual sites.

\*\* At the direction of MDHHS and ATSDR, turbidity, conductivity, and total dissolved solids were discontinued at the end of March 2016.

1. "Standard list" for total metals includes Lead, Copper, Zinc, Aluminum, Iron, Calcium, Cadmium, Potassium, Magnesium, Manganese, Sodium, Nickel, Chromium, and Tin.
2. "Full list" for total metals includes Lead, Copper, Zinc, Aluminum, Iron, Calcium, Cadmium, Potassium, Magnesium, Manganese, Sodium, Nickel, Chromium, and Tin, as well as Antimony, Arsenic, Barium, Beryllium, Boron, Molybdenum, Selenium, Silver, Thallium, and Vanadium.
3. Anions include chloride, fluoride, and sulfate (as SO<sub>4</sub>).

Field sampling coordinators will preserve all total phosphate samples with sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) to a pH below 2 SU immediately after sample collection (i.e., within approximately 15 minutes). Sulfuric acid, nitric acid (if applicable), and narrow-range pH paper will be provided by EPA CRL.

## 8.2 Field Water Quality Testing

Field instruments such as pH meters, conductivity meters, turbidimeters, and colorimetric testing kits (e.g., chlorine residual) will be inspected, maintained, and calibrated according to manufacturer recommendations. Field sampling coordinators will record all field water quality measurements, along with maintenance calibration records, in the field records.

## 8.3 Field Quality Control

Field blanks will not be collected for this project.

Field replicates will be analyzed at a frequency of approximately 20% (one replicate per 5 samples) for field measurements such as colorimetric analysis (e.g., chlorine residual), turbidimeter, conductivity meter, and pH meter.

Field replicates will be collected and analyzed at a frequency of approximately 5% (one replicate per 20 samples) for TTHM and HAA9 analyses (see Section 5.2). Because metals analytical results in drinking water are highly dependent on the plumbing materials, as well as sequence and volumes of sampling, field replicates will not be collected for metals analysis.

Trip blanks containing analyte-free water will be provided by the PHILIS Contract Laboratory prior to sampling for TTHM and VOC analysis (see Section 5.2 and Section 5.4). Trip blanks will be kept in the cooler(s) and will not be opened in the field. Trip blank analytical results will be used to assess potential contamination sources during sample transportation.

## 9. Analytical Requirements

### 9.1 Analytical Methods

Water samples submitted to the appropriate EPA or contract laboratory shall be analyzed in accordance with analytical methods, detection limits, and reporting limits listed below.

Water samples for metals analysis will be digested in accordance with EPA or contract laboratory SOPs. Water samples for other analyses will be prepared in accordance with EPA or contract laboratory SOPs.

Analyte	Analytical Method	Sample Matrix	MDL <sup>1</sup>	Reporting Limit	Hold Time	Bottle Type	Preservative
Total Lead	Based on EPA 200.7/200.8	Drinking Water	0.014 µg/L	0.5 µg/L	6 months	HDPE – 125mL or 1,000-mL	HNO <sub>3</sub> to pH<2
Total Metals - Copper, Zinc, Aluminum, Iron, Calcium, Cadmium, Potassium, Magnesium, Manganese, Sodium, Nickel, Chromium, Tin	Based on EPA 200.7/200.8	Drinking Water	In accordance with EPA or contract laboratory SOPs.				
Additional Total Metals - Antimony, Arsenic, Barium, Beryllium, Boron, Molybdenum, Selenium, Silver, Thallium, Vanadium	Based on EPA 200.7/200.8	Drinking Water	In accordance with EPA or contract laboratory SOPs.				
Total Phosphorus	Based on EPA 365.4	Drinking Water	In accordance with EPA CRL SOPs.		28 days	HDPE – 125mL	H <sub>2</sub> SO <sub>4</sub> to pH<2, Cool <6C
Total Alkalinity	Based on EPA 310.1	Drinking Water	In accordance with EPA or contract laboratory SOPs.		14 days	HDPE – 125mL	Cool <6C
Water Quality Anions – Sulfate, Chloride, and Fluoride	Based on EPA 300.0	Drinking Water	In accordance with EPA or contract laboratory SOPs.		28 days		Cool <6C
VOCs including TTHMs	EPA 524.2	Drinking Water	In accordance with contract laboratory SOPs.		14 days	Three 40-mL VOAs	Cool <4C, Sodium Thiosulfate or Ascorbic Acid, no headspace
HAA9	EPA 552.3	Drinking Water	In accordance with contract laboratory SOPs.		14 days (21 days for extract)	One 250-mL amber glass	Cool <10C, Ammonium Chloride
TDS	Based on Standard Method 2540C	Drinking Water	In accordance with EPA CRL or contract laboratory SOPs.		7 days	HDPE – 125-mL	Cool <6C
Hardness, Calculated	Based on Standard Method 2340B	Drinking Water	In accordance with EPA CRL SOPs.		6 months	N/A – metals bottle(s)	HNO <sub>3</sub> to pH<2
DBPs General List <sup>3</sup>	EPA 551.1	Drinking Water	In accordance with contract laboratory SOPs.		14 days	Three 60-mL vials	Cool <4C, ammonium chloride and phosphate buffer, no headspace

Analyte	Analytical Method	Sample Matrix	MDL <sup>1</sup> / Reporting Limit	Hold Time	Bottle Type	Preservative
Chloral Hydrate <sup>3</sup>	EPA 551.1	Drinking Water	In accordance with contract laboratory SOPs.	14 days	Two 60-mL vials	Cool <4C, sodium sulfite and phosphate buffer, no headspace
SVOCs <sup>3</sup>	EPA 525.2	Drinking Water	In accordance with contract laboratory SOPs.	14 days	Two 1,000-mL amber glass	Cool <4C, sodium sulfite

<sup>1</sup> Method Detection Limits - These MDLs will be re-determined before the samples are analyzed per the EPA or contract laboratory's Quality Management Plan which requires MDL studies to be performed yearly.

<sup>2</sup> Method Detection Limits and Reporting Limits for CRL, Region 4, Region 7, Region 10, and PHILIS Contract Laboratory TestAmerica are included in Appendix J-4.

<sup>3</sup> Analytical methods, analyte lists, and detection limits were selected by MDHHS and ATSDR.

## 9.2 Analytical Quality Control

EPA and contract laboratories have established protocols for the analysis of Quality Control samples with each analytical batch of samples, generally defined as a maximum of twenty samples. Laboratory blank and laboratory fortified blank pairs will be run at a frequency of 1 pair per 20 samples, unless otherwise specified by EPA or contract laboratory SOPs. Laboratory fortified sample and duplicate sample pairs will also be run at a frequency of 1 pair per 10 samples unless otherwise specified by EPA or contract laboratory SOPs. All Quality Control results must be assessed and evaluated on an on-going basis and Quality Control acceptance criteria must be used to determine the validity of the data.

Laboratory blanks run with the samples will not have any detections of lead above the reporting limit, and laboratory fortified blanks and laboratory fortified samples will have recovery limits specified in EPA or contract laboratory SOPs.

Specific information regarding the frequency, composition, acceptance criteria and corrective actions is documented in the specific SOP for a target analyte or procedure, in accordance with applicable EPA or contract laboratory SOPs.

## 10. Sample Handling and Custody Requirements

### 10.1 Sample Custody

Distribution and return transportation for water samples analyzed by the laboratory are discussed below.

- EPA CRL or contract laboratories, in coordination with the EPA Water Division, will distribute the sampling bottles and supplies to field sampling coordinators.

- Field sampling coordinators will coordinate sampling schedules with volunteer residents or owners and will provide instructions for any preparation (stagnation or pre-flushing) required prior to collecting water samples.
- Upon arrival at a sampling site, field sampling coordinators will document field observations and resident information including plumbing, physical disturbances, stagnation time, and home water treatment (e.g., whole house filtration, faucet filters, water softener).
- Field sampling coordinators will collect the water samples and complete field sampling records (hard copy field forms or electronic field forms such as iForms software).
- Field sampling coordinators will conduct any field measurements or field preservation.
- Field sampling coordinators will prepare and review sampling labels and chains of custody, including documenting sample time for each sample.
- Field sampling coordinators will ship the water samples overnight to the applicable EPA or contract laboratory, using coolers and shipping labels provided by EPA or the contract laboratory. Whenever possible, overnight shipment will be on the same day as sampling is completed. Field sampling forms and chain-of-custody forms will be placed in sealed Ziploc bags inside each cooler. A hand-written or typed chain-of-custody form (see Appendix J-1) will be used for this project, and standard EPA chain-of-custody procedures will be followed.
- Shipments will be tracked and accounted for between the field staging area and the laboratories. In general, this will include pre-shipment notification to the applicable laboratory's sample coordinator.
- The laboratory will provide copies of the chains of custody forms following receipt and log-in, and notify the EPA Water Division Technical Contacts and field sampling coordinators regarding any issues noted upon receipt.
- The applicable EPA or contract laboratory will analyze the samples and provide the results to the EPA Water Division.

## **10.2 Sample Archive/Disposal**

The samples received the applicable EPA or contract laboratories, including any digestates, will be archived unless a written request for disposal is provided by EPA Water Division to the applicable EPA or contract laboratory.

## **11. Instrument/Equipment Testing, Inspection, Maintenance, & Calibration Requirements**

### **11.1 Instrument/Equipment Testing, Inspection, and Maintenance**

All laboratory instrumentation will be inspected and maintained according to EPA or contract laboratory maintenance protocols.

Field instruments such as pH meters, turbidimeters, conductivity meters, and colorimetric testing kits will be inspected and maintained by field personnel according to manufacturer recommendations.



## **11.2 Instrument/Equipment Calibration and Frequency**

All laboratory instrumentation will be calibrated according to EPA or contract laboratory SOPs.

Field instruments, such as pH meters, turbidimeters, and conductivity meters will be calibrated according to manufacturer recommendations.

## **11.3 Inspection/Acceptance of Supplies and Consumables**

Sample containers provided by EPA CRL or contract laboratories will be single use and obtained as certified clean from the supplier.

Laboratory reagents are evaluated through the use of laboratory blanks and spikes for purity.

## **12. Data Management**

A diagram presenting data management workflow is presented below. Field and laboratory data for the LSL Detection Study will be collected, reviewed, and stored by ORD and its contractor (Battelle) to ensure data completeness and quality consistent with Sections 12 through 14.

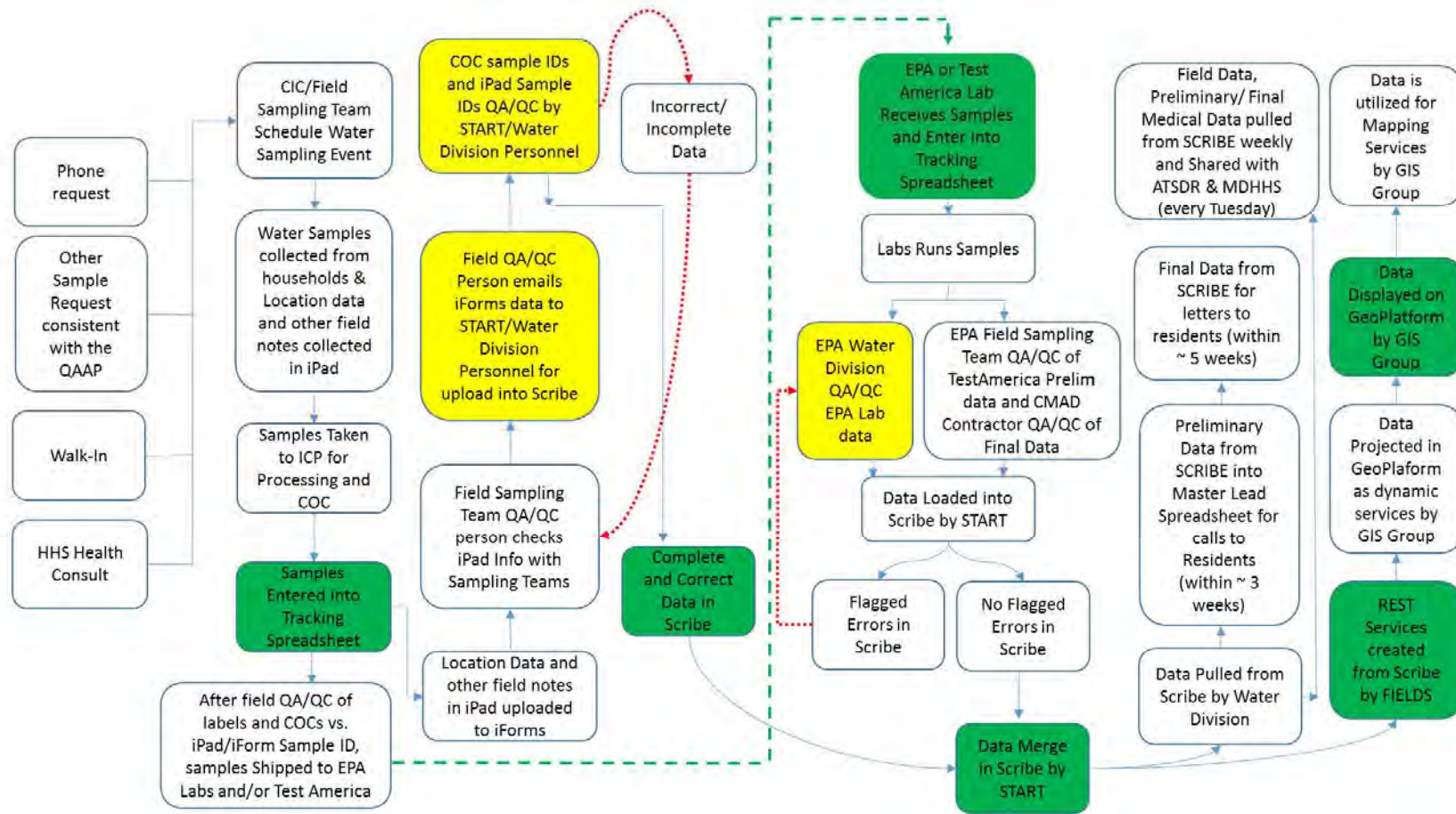
The sampling team will generate field records (either hard copy or electronic records) which will be utilized to document sampling activities and relevant observations. An electronic field record management system (using iPads and iForm software) has been developed for the project and should be used to document all field observations whenever possible. Electronic field records will be reviewed by the field sampling team, generally on the same day as sampling occurred, prior to uploading the field notes into the project Scribe database.

All laboratory data and analytical results will be stored in EPA laboratory information management systems (LIMS). The applicable EPA or contract laboratory will store this data in accordance with its standard data storage procedures. In addition, electronic data deliverables (EDD) compatible with the Scribe database will be provided for preliminary and final analytical results. Analytical results will be provided in .pdf electronic document read-only format and as an EDD. EDDs will be reviewed by the Water Division Technical Contacts (or designee such as Water Division QA personnel) prior to uploading preliminary and final analytical results into the project Scribe database. Data deliverables will also be provided in Excel format.

Preliminary analytical results will be provided by the EPA or contract laboratory to the EPA Water Division Technical Contacts as soon as feasible, within 6 business days after receipt of each shipment of samples. Final peer-reviewed analytical results will be forwarded from the EPA or contract laboratory to the EPA Water Division Technical Contacts and the Ground Water and Drinking Water Branch (GWDWB) Quality Assurance (QA) Coordinator, as soon as feasible, within 10 business days after preliminary analysis of each shipment of samples. The EPA Water Division Technical Contacts will arrange for archival of the final analytical reports in accordance with Federal Record retention schedules, and updating all data providers of changes to the distribution list for data deliverables.

All data will be validated as meeting laboratory and QAPP-defined quality control requirements by EPA and contract laboratories according to their procedures. In addition, the preliminary and final data files and associated lab reports will be compared for discrepancies and reviewed for missing samples or information by a team of Water Division Technical Contacts.

**Data Flow Diagram – Flint Drinking Water Response**



### **13. Data Assessment and Response Actions**

Formal field audits by quality assurance personnel are not anticipated for this project. Identification of problems related to technical performance will be the responsibility of the technical staff working on this project.

The EPA Project Officer will assess any problems that arise in the field. If necessary, modifications to technical procedures may be considered. Any changes in technical procedures will be documented in the field notebook, evaluated to determine if there will be any impact to the data and then highlighted in the final project report.

Laboratory personnel will perform self-audits and institute corrective actions in accordance with their respective written procedures. Data review of all EPA or contract laboratory generated data is performed by a second analyst not associated with the actual measurement operations for the given analytical batch, but knowledgeable in the analytical processes employed. It is the responsibility of the reviewer to ensure that all data generated are correct and of known and documented quality. Once the review is completed, the reviewer will sign and date the appropriate Data Review checklist according to the EPA or contract laboratory SOPs. Any limitations on the use of data, e.g., data qualifiers, will be included in the final report for the project.

The Water Division Technical Contacts (or designee such as Water Division QA personnel) will perform a data quality assessment on the results of each batch of water samples, based on laboratory blanks, laboratory fortified blanks, and laboratory-fortified samples (i.e., matrix spikes). The Water Division Technical Contacts will review the analytical report and determine any limitations on the use of the data (see Section 6) and include these limitations in the final project report. If any data is assessed to be unusable by the Water Division Technical Contacts or Project Officer, re-sampling may be required.

## 14. Reporting, Documents, and Records

Original documents will be stored as detailed below.

	EPA Project Officer	EPA CRL	Other Laboratories
Analytical Request Form		XX	XX
QAPP	XX		
Field Records (Electronic or Hard Copy)	XX		
Certification of Sample Containers	XX	XX	
Chain-of-Custody	XX	XX	XX
Photos	XX		
Analytical Results	XX	XX	XX
Final Project Report	XX		

Project files will be kept for as long as Technical Assistance to City of Flint continues. The EPA Water Division Technical Contacts will arrange for archival of the final analytical reports in accordance with Federal Record retention schedules. Final analytical results will also be distributed to all relevant parties, provided to the applicable resident in accordance with Consumer Notification requirements, and posted on EPA’s Flint Drinking Water Response website.

Photos will be stored as per the August 3, 2006 “Digital Camera Guidance for EPA Civil Inspections and Investigations”. Photos will be downloaded onto a CD or DVD and stored with the project file at the EPA Region 5 Office.

## 15. Dispute Resolution

Quality assurance disputes regarding this QAPP will be brought to the attention of the GWDWB QA Coordinator, respective laboratory director, and the Water Division QA Manager. If a solution cannot be reached or agreed upon within three working days, the issue will be raised by either the GWDWB QA Coordinator, Water Division QA Manager, or the respective laboratory director to the Deputy Water Division Director, and if necessary, to the Water Division Director for final resolution.

## Appendices

Appendix A – Faucet Filter Evaluation

Appendix B – Distribution System Monitoring for Disinfectant and DBPs

Appendix B-1 – EPA ORD Sampling Plan

Appendix B-2 – TTHM and HAA9 Sampling Instructions

Appendix C – Lead Source/Release Diagnostic Evaluation

Appendix C-1 Sequential Sampling Instructions to Residents

Appendix C-2 Residential Sampling Volunteer Survey

Appendix C-3 Plumbing Inspections for Corrosion Control Treatment Assessment

Appendix C-4 Additional Distribution Samples for Flow Rate Assessment

Appendix C-5 Lead Service Line Detection Protocol

Appendix D – Health and Direct Contact Exposure Evaluation

Appendix D-1 – Health and Direct Contact Exposure Evaluation, General Procedures

Appendix D-2 – Limited “Pilot” Evaluation

Appendix E – Aerator Particulate Evaluation

Appendix F - Distribution System Monitoring for Treatment Assessment

Appendix G – Distribution System Sampling for Coliform Bacteria

Appendix H – Resident-Requested Sampling and Hot Water Evaluation

Appendix I – Lead Service Line Extraction and Pipe Scale Analyses

Appendix J – Sample Custody, Packaging, and Shipment

Appendix J-1 – Chain-of-Custody Form

Appendix J-2 – Sample Packing Instructions

Appendix J-3 – Sample Shipment Instructions

Appendix J-4 – Reporting Limits and Detection Limits for EPA and Contract Laboratories

## Appendix A – Faucet Filter Evaluation Sampling

Water samples will be collected to assess concentrations of lead in filtered and unfiltered drinking water at selected sampling sites, including homes where existing analytical data indicates elevated lead in water concentrations above 150 µg/L. Field sampling coordinators will confirm the schedule with the applicable resident(s) in advance of sampling activities. In general, three grab water samples will be collected at the kitchen faucet at each selected sampling site:

- Filtered Water, Existing Faucet Filter - One grab sample will be collected through the existing water filter (if present). The field sampling coordinators will note the type (brand) of the filter, status of the filter indicator, and available information from the resident regarding the time since the filter or cartridge was installed.
- Unfiltered Water - The filter will be removed, and an unfiltered water sample will be collected as the first grab sample following removal of the filter and/or aerator. No cleaning or flushing will be conducted prior to the unfiltered water grab sampling.
- Filtered Water, New Faucet Filter - Following the collection of the unfiltered sample, a new filter or new filter cartridge will be installed, and the water will be allowed to run through the new filter for approximately two minutes. Following installation and flushing of the new filter or replacement filter cartridge, a grab sample will be collected through the newly installed filter.

No stagnation or flushing is generally required prior to grab sampling. If no faucet filter is currently in place at the site, or if a faucet filter is unable to be installed, the field sampling coordinators will collect fewer samples and will document reasoning in the field notes. All grab samples should be collected bypassing existing whole house filter(s) and/or water softeners. The sampling location (e.g., kitchen sink faucet) must be clearly documented in the field records.

Beginning in late March 2016, a modified procedure was implemented to include the collection of unfiltered samples following a minimum of 6 hours stagnation. The resident will be asked to record the time when water was last used in the home (i.e., no faucets, toilets, shower, bath tub, washing machine, dishwasher, and hose), and field sampling coordinators will record the total stagnation time in the field records. When stagnation can be confirmed, two water samples will be collected as described below at each selected sampling site, generally at the kitchen faucet.

- Unfiltered Water - The filter will be removed (or bypassed), and an unfiltered water sample will be collected as a first flush sample following removal of the filter and/or aerator and a minimum of 6 hours stagnation. No cleaning or flushing will be conducted prior to the unfiltered water grab sampling.
- Filtered Water, Existing (or New) Faucet Filter - One grab sample will be collected through the existing water filter (if present). If no existing filter is present, field sampling coordinators will install and flush a new filter, as described above, then collect the filtered water sample through the newly-installed water filter. The field sampling coordinators will note the type (brand) of the filter, status of the filter indicator, and available information from the resident regarding the time since the filter or cartridge was installed.

Beginning in May 2016, a similar procedure was implemented to collect filtered and unfiltered samples at residences selected and referred by ATSDR and partner health agencies. At each sampling site, two water samples will generally be collected as described below, generally at the kitchen faucet.

- Unfiltered Water - The filter will be bypassed (or removed), and an unfiltered water sample will be collected. No cleaning or flushing will be conducted prior to the unfiltered water grab sampling.
- Filtered Water, Existing Faucet Filter - One grab sample will be collected through the existing water filter (if present). If no existing filter is present, field sampling coordinators will install and flush a new filter, as described above, then collect the filtered water sample through the newly-installed water filter. The field sampling coordinators will note the type (brand) of the filter, status of the filter indicator, and available information from the resident regarding the time since the filter or cartridge was installed.

If field sampling coordinators observe an expired filter, based on filter indicator light or other indicators, they will install a new filter and flush the new filter per field procedures described above. In those cases, a third sample would be collected as a filtered sample through the new filter.

For each grab water sample, one 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH} < 2$ ) for analysis of total metals including lead. Field sampling coordinators will be responsible to pack water samples and ship to the selected EPA laboratory or the PHILIS Contract Laboratory, as described in Appendix J.

Water sample identification will use the nomenclature below.

- Prefix of “FG” will indicate the sampling site was selected based on a previously reported lead in water concentration above 150  $\mu\text{g/L}$ .
- Prefix of “FGW” will indicate the sampling site was selected based on the request of the resident or home owner.
- Prefix of “FH” will indicate the sampling site was selected by ATSDR and partner health agencies.
- Prefix of “FGC” will indicate the unfiltered sample is a first-flush sample collected following removal of the filter and/or aerator and a minimum of 6 hours stagnation.
- Numbers will be used to indicate unique samples.
- House letter unique to the address and sample type will be included following numbers.
- Suffix of “U” will indicate the sample is filtered water collected through an existing faucet filter.
- No suffix will indicate the sample is unfiltered water.
- Suffix of “N” will indicate the sample is filtered water collected through a newly installed faucet filter.



Unique sample identification will be required. Deviations from this sampling nomenclature must be clearly documented in the field records including hard copy field forms, electronic field forms, and/or chains of custody.

Field records should also include the following, to the extent information is provided by the resident or observed in the field:

- Interior plumbing including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Service line including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Estimated distance (or pipe length) between the sample tap and the distribution water main
- Filter(s) in use at the home, including any that were being used at the time of sampling
- Known physical disturbances such as recent road work or utility work that could disturb the service line near the sampling location
- Other relevant field observations such as activities completed at the home (e.g., flushing, aerator cleaning) and color, odor, or debris in the water
- Photographs of the sample tap(s) and underlying fixtures and components

Following the completion of grab sampling, field measurements for pH and chlorine residual may be collected as discussed in Section 5.6.

Before leaving the site, the field sampling coordinator should confirm the faucet filter is properly installed and returned to “Filter” mode (not “Bypass” mode). If time allows, the field sampling coordinator will work with residents to check and clean aerators on all other faucets in home.

## **Appendix B – Distribution System Water Quality Characterization**

Chlorine, pH, and temperature will be measured weekly or biweekly at up to 35 representative sites in the distribution system in accordance with the sampling plan developed by EPA ORD (see Appendix B-1).

Total trihalomethanes (TTHM) and nine haloacetic acids (HAA9) will be measured biweekly or monthly at up to 35 sites within the distribution system in accordance with sampling instructions provided by the contract laboratory (see Appendix B-2). The sample identification for all TTHM and HAA9 samples will use a unique station number (assigned to a specific sampling location, along with a prefix of “T” or “H” for TTHMs and HAA9 analysis, respectively). The sample identification will also include the station location as the sample date and the sample address in the following format: MMDDYYNNNNStreet, where MM is the calendar month, DD is the calendar date, YY is the calendar year, and NNNN is the house number for the identified street address. Trip blanks for TTHM analysis will be identified on the chains of custody using a prefix of “B”. Field replicates will be identified on the chains of custody using a suffix of “D” (e.g., T12D would be a field replicate of sample T12).

Field sampling coordinators will confirm the schedule with the applicable resident(s) in advance of sampling activities. Field sampling coordinators will measure field water quality parameters including temperature, chlorine residual, and pH, immediately after sample collection, within 15 minutes. Field sampling coordinators will be responsible for obtaining and maintaining field water quality equipment (e.g., pH meter and colorimetric test kits for chlorine residual) and following manufacturer guidance to ensure accurate field measurements are obtained. Field sampling coordinators will record all equipment maintenance (including calibration) and field measurements for each sample in the field records.

Water samples for water quality parameters including TTHMs and HAA9 will be placed on in ice-packed coolers as soon as possible after collection. Field sampling coordinators will be responsible to pack distribution system samples for TTHMs and HAA9 and ship to the PHILIS Contract Laboratory, as described in Appendix J.

## **Appendix B-1 – EPA ORD Sampling Plan**

# Flint Distribution System Chlorine Residual Sampling Plan

## Introduction

In January 2016, a team of engineers with extensive experience in chlorine disinfection was requested to assess chlorine disinfection in the Flint, MI water distribution system to ensure best practices were being used, lowering risks from waterborne disease and ensuring proper health protection was being carried out through the use of disinfection in the public water system. The Flint water system aligns pretty closely to city boundaries, as shown in Figure 1, with only a few hundred water customers outside these boundaries.

Through meetings with U.S. EPA staff already in Flint, meetings with Flint Water Utility Staff, and driving sampling sites throughout the city, it was quickly determined that additional chlorine monitoring was required in Flint. Through Legacy agreements between all the systems that receive Detroit water, the Michigan Department of Environmental Quality (MDEQ) has been implementing a modified monitoring plan for the Total Coliform Rule (TCR) (which includes chlorine monitoring) for the Detroit metropolitan area (which includes Flint, MI) in accordance with Section 141.29 (Monitoring of Consecutive Public Water Systems), since Michigan was granted primacy in 1978. While the Flint MI water system has been monitoring the full number of prescribed samples (100) per month, the number of monitoring sites is small (10), which is a result of the legacy agreements going back many years.

In light of the limited chlorine monitoring locations and some preliminary chlorine sampling conducted by both U.S. EPA and Flint community advocates that resulted in low chlorine concentrations, an organized repetitive chlorine sampling program was deemed necessary to establish the success of the current chlorination objectives being carried out in Flint, MI, and to work with the water utility to improve disinfectant residual maintenance and reduce risks of waterborne disease if low chlorine residuals are encountered in the distribution system.

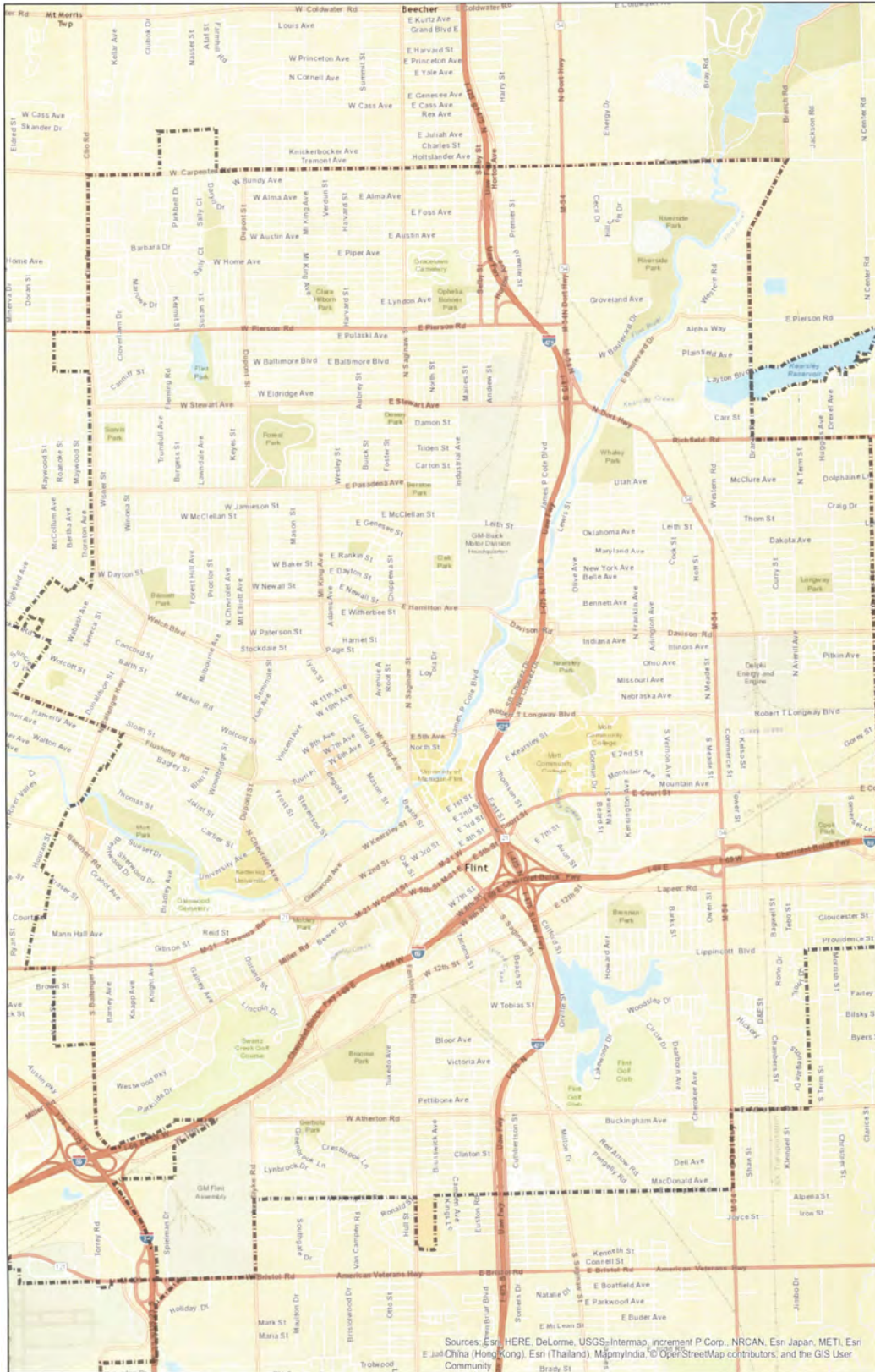


Figure 1 - City of Flint Map (dotted line is city boundary)

## Sample Site Number Selection Methodology

Because of its service population of approximately 100,000 people, the City of Flint is required to collect 100 total coliform (TCR) samples each month (USEPA, 1989). Currently, the City of Flint accomplishes this by sampling the 10 sites listed in Table 1. In light of all that has been learned with regards to disinfection and distribution systems as chemical reactor systems since the late 1970s, monitoring 10 distribution sites in a system the size of Flint is not a current best practice for monitoring the maintenance of a residual disinfectant as a barrier to microbial contamination.

**Table 1 – Existing City of Flint Total Coliform (TCR) Sample Site Locations**

Location	Street	City	State	Zip Code
Arby's	3742 Davison Rd	Flint	MI	48506
Cedar St. Reservoir	1100 Cedar St	Flint	MI	48503
Liquor Palace	3302 S Dort Hwy	Flint	MI	48507
North Flint Automotive	6204 N Saginaw St	Flint	MI	48505
Rite-Aid Pharmacy	2838 E Court St	Flint	MI	48506
Rite-Aid Pharmacy	5018 Clio Rd	Flint	MI	48504
Salem Housing	3216 MLK Blvd	Flint	MI	48505
Taco Bell	3606 Corunna Rd	Flint	MI	48532
University Market	2501 Flushing Rd	Flint	MI	48504
West Side Reservoir	1416 Dupont St	Flint	MI	48504

Current best practice for a water utility the size of Flint (approximately 100,000 people) to ensure distribution system sampling represents the entire distribution system would be to organize the TCR sampling program to include 50 total sample sites, representing one-half of the total number of monthly required TCR samples. These 50 sampling sites would be used on a geographical rotating basis such that each sample location would be monitored twice monthly, resulting in the required 100 TCR samples. To this end, the City of Flint also currently monitors an additional 15 sites on a quarterly basis (Table 2), resulting in a total of 25 sample site locations that are currently monitored by the City of Flint on some interval.

Based on a desired target of 50 total sample site locations and the existence of 25 sample site locations, a target for this sampling site plan was to add an additional 20-25 sample site locations, bringing to the total number of sample site locations in the City of Flint to approximately the desired number of 50.

**Table 2 - Existing City of Flint Quarterly Sample Site Locations**

Location	Street	City	State	Zip Code
Arby's	3559 S Dort	Flint	MI	48507
Clio Road Pharmacy	4902 Clio Rd	Flint	MI	48504
Cummings Elementary School	G 2200 Walton Ave	Flint	MI	48532
Flint City Hall	1101 S Saginaw	Flint	MI	48502
Flint Public Library	1026 E Kearsley St	Flint	MI	48503
Grandma's Recipes	3538 Richfield Rd	Flint	MI	48506
Quizzno's	3818 E Court	Flint	MI	48506
Rite-Aid Pharmacy	3521 Corunna Rd	Flint	MI	48503
Rite-Aid Pharmacy	3717 Fenton Rd	Flint	MI	485041
Russell Collection Agency	3285 Van Slyke Rd	Flint	MI	48507
Southwestern Classical Academy	1420 West 12th St	Flint	MI	48507
Taco Bell	1740 S Dort	Flint	MI	48503
Tim Hortons	1274 N Ballenger Hwy	Flint	MI	48504
White Horst Tavern	621 W Court St	Flint	MI	48503
Yaya's	519 S Dort	Flint	MI	48503

### Sample Site Location Selection Methodology

Individual sample site locations were selected to assess chlorine residuals and potential microbial contamination at locations where existing sampling is not currently conducted by the City of Flint (i.e., sample locations presented in Table 1 and Table 2). Furthermore, the sampling site location selection focused on locations (1) serviced by smaller distribution mains (i.e., 8 inches or less in diameter) and (2) that are publicly accessible (e.g., schools, places of worship, community centers) throughout the City of Flint service area to allow for repeated sampling (e.g., weekly). Sampling locations were selected based on available distribution system layout records and known City of Flint total coliform rule (TCR) sampling locations. During sampling site selection, the site owner/manager was asked for their willingness to participate in the regular sampling effort, and sampling will be scheduled based on the location's availability to the extent feasible.

To assess the locations of smaller distribution mains, the best available record was a City of Flint water distribution system map from 1980 (Figure 2) that indicated all mains that were 8" or greater. A working sample site selection map (Figure 3) was then generated by combining (1) the distribution system map, (2) the City of Flint provided TCR sample site locations, and (3) a web-based search to determine publicly accessible sampling locations throughout the City of Flint. The working sample site selection map was then used to evaluate approximate 25 locations that provided a representative coverage of the City of Flint, and the extent possible, would be located on 8" or smaller distribution system mains where the City of Flint does not currently monitor water quality.



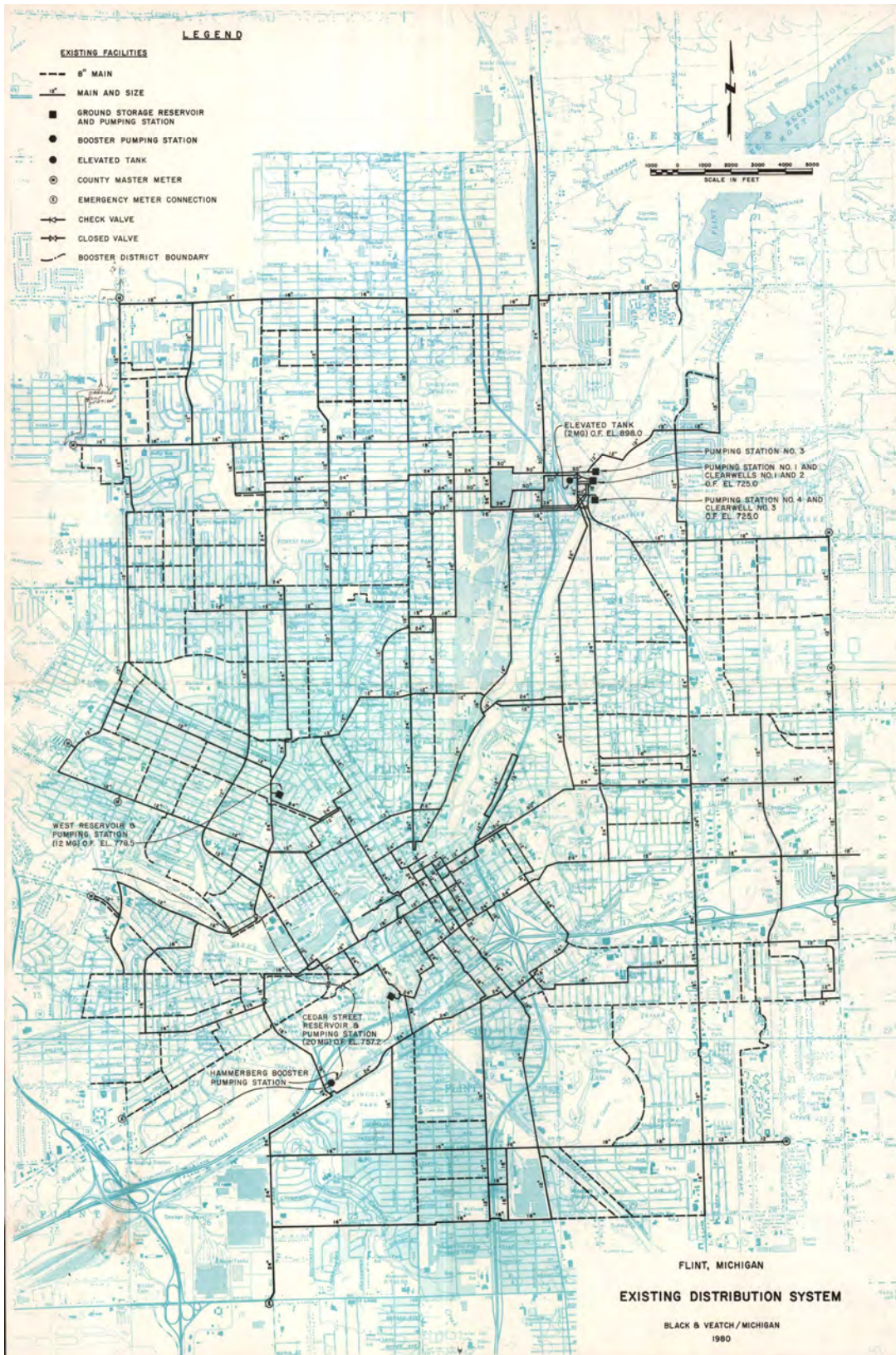
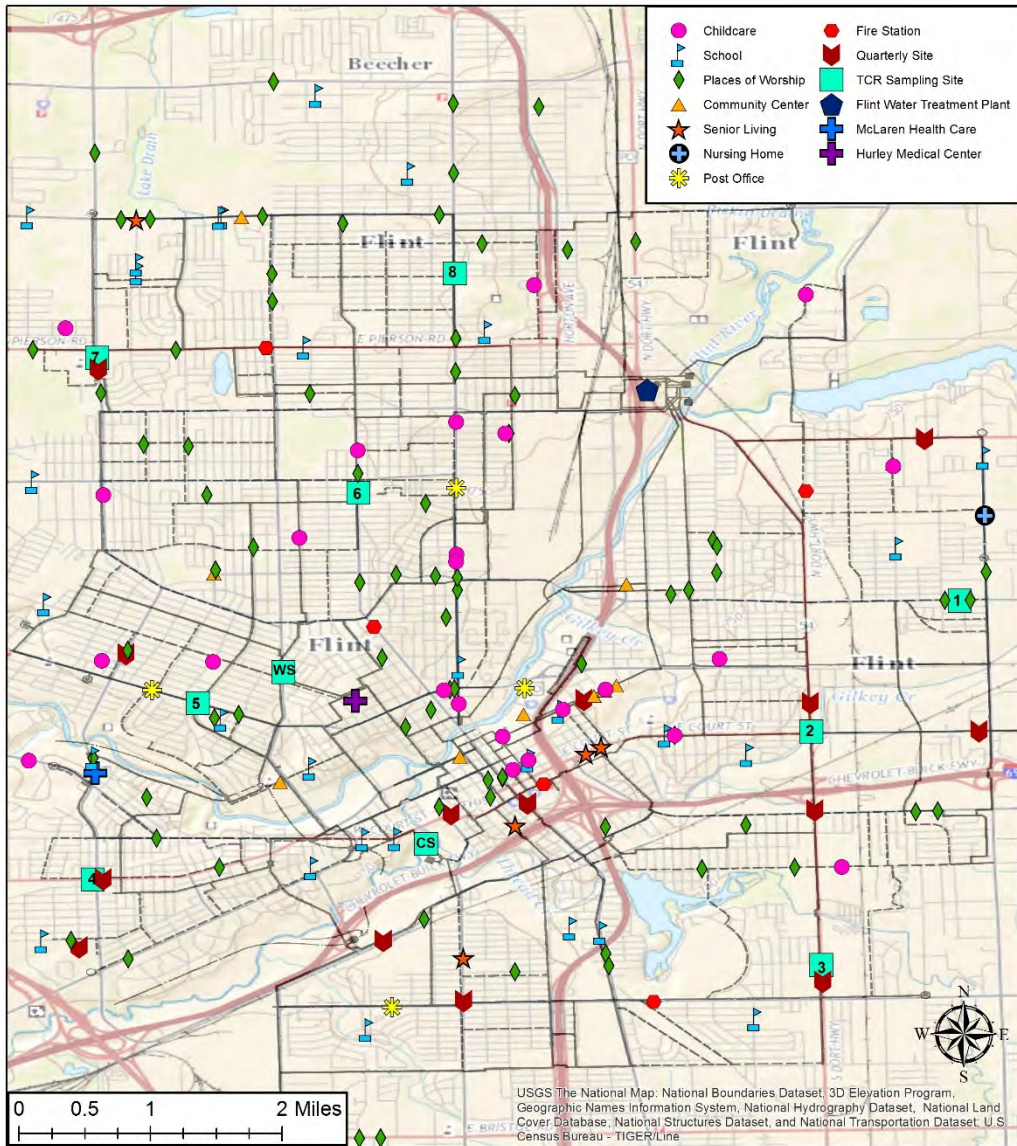


Figure 2 – City of Flint Water Distribution System Map from 1980

## Potential Free Chlorine Sampling Sites in Flint, MI



**Figure 3 - Working Sampling Plan Site Selection Map**

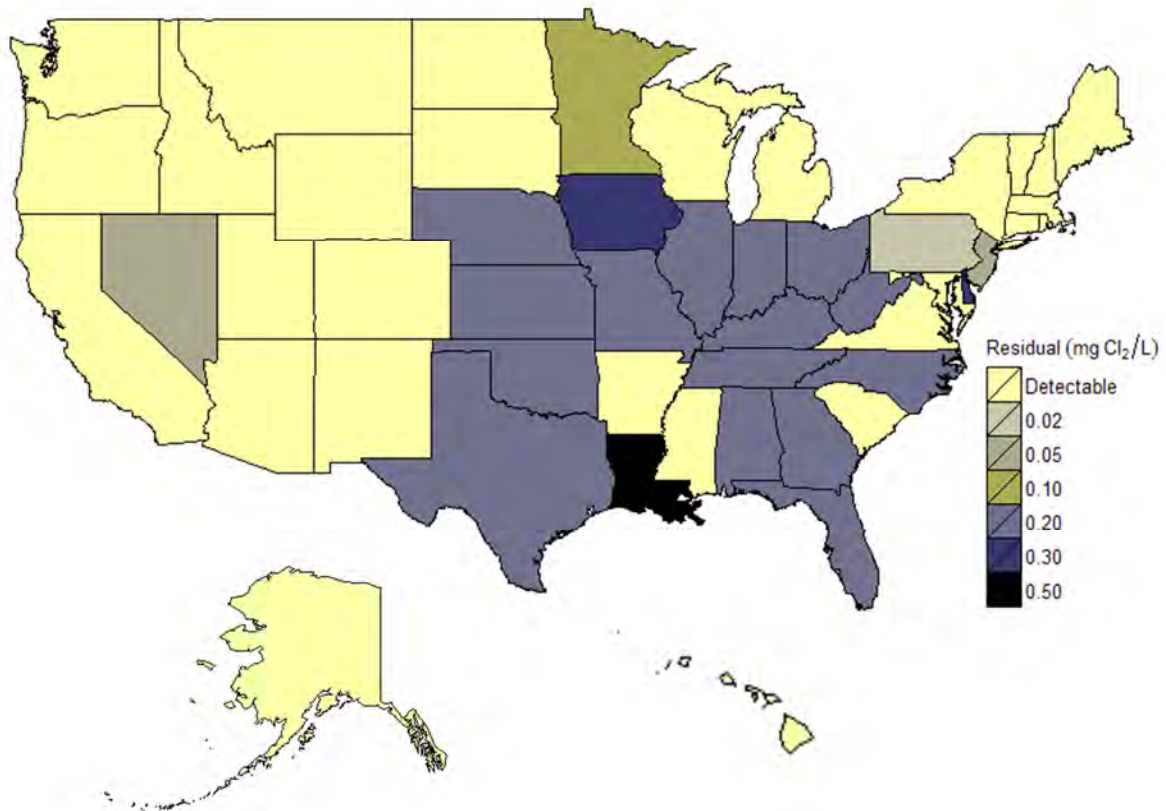
### Sample Site Analysis Methodology

To assess the presence of a disinfectant residual in the main distribution system mains, residual chlorine, pH, and temperature will be immediately measured after sample collection (i.e., within 15 minutes), according to manufacturer guidance of the field instruments to ensure accurate field measurements are obtained. If a chlorine residual is measured less than 0.20 mg Cl<sub>2</sub>/L, a microbial sample will also be collected. Specific procedures for microbial sampling are

provided in Appendix E, except that the flushing time may be greater than 5 minutes if this is required to guarantee sampling from the targeted distribution system main. The specific flushing time required for each sample location was determined as described in Flushing Methodology. The 0.20 mg Cl<sub>2</sub>/L trigger for microbial sampling was selected because it represents (1) the performance goal for the Area-Wide Optimization Program's (AWOP) approach to maintaining distribution system water quality in free chlorine systems and (2) a common minimum distribution system level used across the country to ensure that a measurable residual is present (Figure 4), and (3) the minimum free chlorine residual in a water distribution system recommended in the Ten States Standards (Great Lakes – Upper Mississippi River Board of State and Provincial Public Health and Environmental Managers, 2012). Furthermore, it is the goal of this sampling program to have chlorine residuals greater than 0.2 mg Cl<sub>2</sub>/L throughout the distribution system.

### Sample Site Flushing Methodology

Required sample flush times were determined individually at each sample site location. The flushing objective was to displace the volume of water between the sample location and water distribution system main, ensuring that water samples were representative of water quality within the distribution system main. Required flush volumes were determined by measuring the length and diameter of individual segments of water line (i.e., water service line and premise plumbing). The total flush volume was determined by adding the calculated flush volume of each segment. The service line length was measured from the water main to the meter connection inside the building. The diameter of the service line was determined by inspecting the size of line entering the building prior to the meter connection. The premise plumbing length was estimated between the meter connection and the sampling location. In general, the sample site location nearest to the meter connection was selected. The diameter of premise plumbing was assumed to be ¾" when actual diameter was unknown. All line lengths were measured using a rolling measuring wheel. A conservative flowrate of 2 gpm was assumed at the majority of sample locations, although actual flowrates were greater. The conservative estimate was intended to provide a "factor of safety" to ensure that a sufficient volume of water was flushed prior to sample collection. Sample flush times were then calculated based on the estimated flush volume and assumed flowrate. In a few cases where calculated flush times were excessive (e.g., greater than 30 minutes), the actual flowrate was measured and flush times were adjusted. A minimum sample flush time of five minutes was conducted at all locations.



Cl<sub>2</sub>—chlorine

- Colorado has proposed requiring a 0.2 mg Cl<sub>2</sub>/L minimum residual throughout the distribution system.
- Iowa provides an exception for dead ends or areas that represent very low use.
- Louisiana requires a “detectable” residual but currently has an emergency rule requiring a minimum 0.5 mg Cl<sub>2</sub>/L free chlorine residual; therefore, 0.5 mg Cl<sub>2</sub>/L is used here.
- Minnesota requires a “detectable” residual but also requires measurement to the nearest 0.1 mg Cl<sub>2</sub>/L in the range below 0.5 mg Cl<sub>2</sub>/L; therefore, 0.1 mg Cl<sub>2</sub>/L is used here.
- Nebraska allows a minimum 0.1 mg Cl<sub>2</sub>/L free chlorine residual if additional conditions are met.
- Nevada requires a “detectable” residual but also requires measurement to the nearest 0.05 mg Cl<sub>2</sub>/L; therefore, 0.05 mg Cl<sub>2</sub>/L is used here.
- North Carolina’s minimum residual included here is for Total Coliform Rule compliance samples. North Carolina requires a “detectable” residual at maximum residence times.
- Pennsylvania has proposed requiring a 0.3 mg Cl<sub>2</sub>/L free chlorine residual.
- Vermont requires a “detectable” residual but further states that the residual “should be 0.1” mg Cl<sub>2</sub>/L.

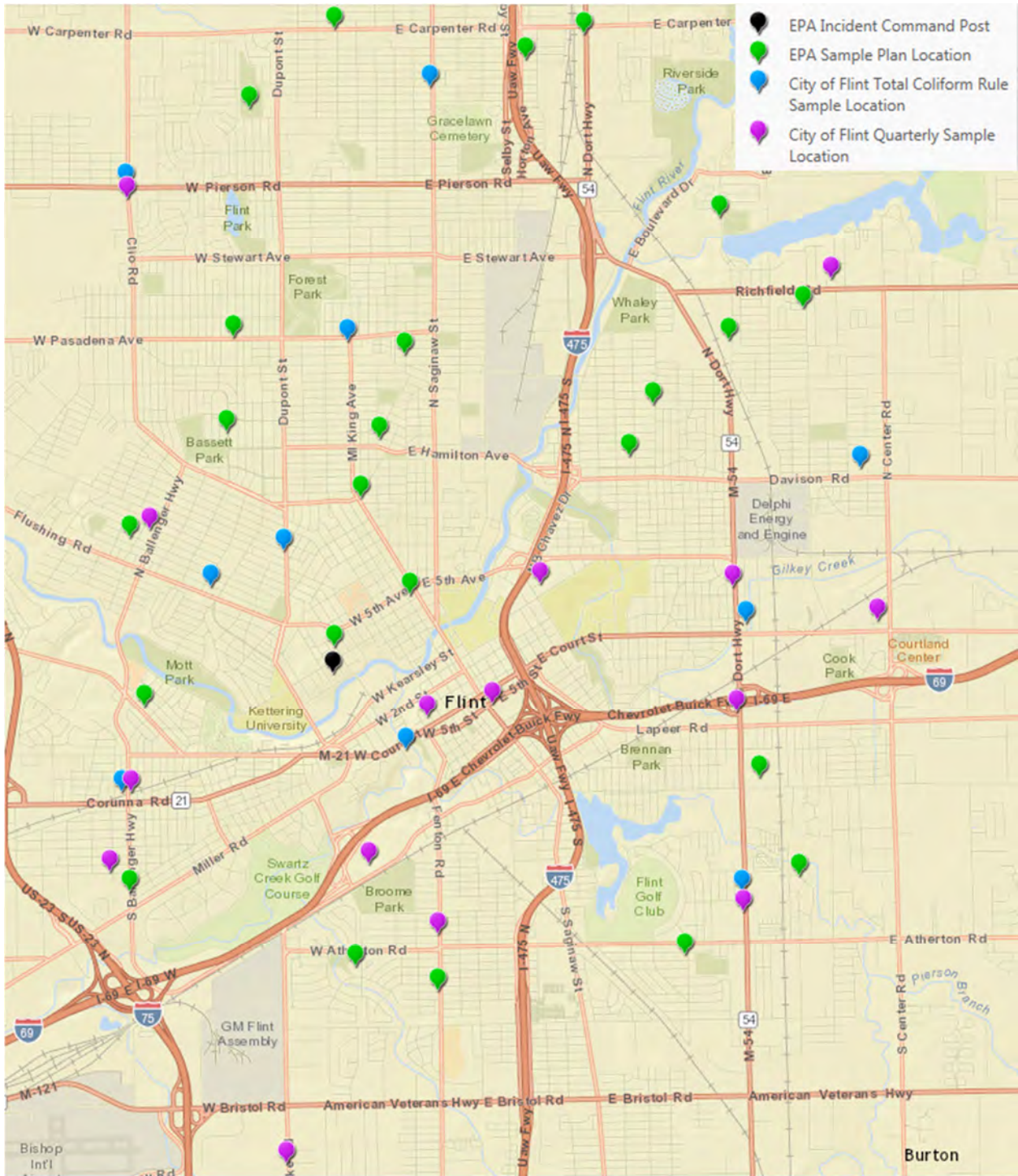
**Figure 4 – United States minimum free chlorine residuals in the distribution system (not entering the distribution system) for Subpart H Surface Water Treatment Rule systems based on review of existing states’ rules and regulations as of January 2015 (Wahman & Pressman, 2015)**

## Results of Site Selection Evaluation

Beginning with all the potential sites developed and mapped (Figure 3), a smaller subset of sites was chosen based on spreading the geographic location of sites throughout the city and on interest in capturing samples from embedded locations near homes which are most representative of where the water is consumed. The team canvassed the city over a one week period, evaluating sites for the repetitive sampling plan. Some sites were deemed unfeasible based on factors such as distance from the distribution main, size of service line, or ability to access the facility. For example, some locations with 4 or 6 inch service lines that are hundreds of feet from the street would require more than an hour of flush time, which is not feasible for this type of sampling program. Therefore, these sites were eliminated. By the end of the week, 24 sites were selected for the repetitive sampling program, as summarized in Table 3 and shown on Figure 5. These sites represent, when combined with the 10 TCR and 15 quarterly sites monitored by the city, good geographic distribution and representation from smaller distribution pipes not previously evaluated in the City’s TCR and quarterly locations.

**Table 3 - Selected Sample Site Locations**

Location	Street	City	State	Zip Code
True Light Ministry Baptist Church	6917 Martin Luther King Ave	Flint	MI	48505
Hasselbring Senior Center	1002 W. Home Ave	Flint	MI	48505
St. Luke's New Life Center	3115 Lawndale Ave	Flint	MI	48504
Fire Station #3	1525 Martin Luther King Ave	Flint	MI	48503
Triumph Church	1657 Broadway Blvd	Flint	MI	48506
Fire Station #5	3402 Western Rd	Flint	MI	48506
Boys and Girls Club of Flint	3701 N. Averill Ave	Flint	MI	48506
Sam's Rollingwood Market	4612 Western Rd	Flint	MI	48506
Freeman School	4001 Ogema Ave	Flint	MI	48507
Gloria's Little Angels Childcare	2440 Lippincott Blvd	Flint	MI	48507
Genesee Community Health Center	3109 Kleinpell St	Flint	MI	48507
Neithercut Elementary	2010 Crestbrook Ln	Flint	MI	48507
Domino's Pizza (Fenton)	4402 Fenton Rd	Flint	MI	48507
Domino's Pizza (Ballinger)	2113 S. Ballenger Hwy	Flint	MI	48503
Palace Liquor Store	3609 Beecher Rd	Flint	MI	48503
Greater Flint Outreach Center	1114 University Ave	Flint	MI	48504
Carriage Town Ministries	605 Garland St	Flint	MI	48503
Christ Enrichment Center	322 E. Hamilton Ave	Flint	MI	48505
Sunny Patch Learning Center	1160 Donaldson St	Flint	MI	48504
Saints of God Church	2200 Forest Hill Ave	Flint	MI	48504
Shiloh Missionary Baptist Church of Flint	502 Leith St	Flint	MI	48505
St Mary Church	2500 N. Franklin Ave	Flint	MI	48506
Foss Avenue Baptist Church	1159 E. Foss Ave	Flint	MI	48505
Greater Holy Temple Ministries Church of God in Christ	6702 N. Dort Hwy	Flint	MI	48505



**Figure 5 - Summary of Selected Sample Site Locations and Existing Total Coliform (TCR) Rule Sampling Site Locations**

### Data Collection, Analysis, and Operational Responses

The chlorine residual data collected for each time period will be shown on a map of the city and posted to the EPA Flint website. Methods for further data analysis as related to enhanced

visual communication of the information both inside and outside EPA are currently being evaluated. U.S. EPA is also working on obtaining and using an already developed distribution system network model, which when completed, will allow further advanced analysis of the measured chlorine residuals. Should low chlorine residual areas exist within the distribution system, U.S. EPA will work with the Flint Water Utility to implement appropriate measures to increase the chlorine residuals. Such measures may include, for example, low velocity flushing (to improve residual with minimal disturbances to scale) and adjusting chlorine additions at the City's chlorination points.

## Sampling Plan Implementation

The chlorine sampling of the identified locations should be conducted initially on a weekly basis until the extent of any distribution system residual issues are identified and the initial remediation steps are initiated by working with the water utility. If availability of any sites becomes a problem, they will be replaced with new sites in nearby locations. Furthermore, additional sites may be added as necessary to fully assess chlorine residuals in the distribution system. Depending on whether problems are identified, this initial phase is to be a minimum of 4 weeks. Upon review of the data and changes to distribution system operations made in response by the water utility, an evaluation can be made after 4 weeks to determine if sampling can be extended to once every two weeks. This may be possible during later winter and early spring while the water is still cold.

Once spring and summer arrive and bring warmer water temperatures, the chlorine decay in the distribution system will tend to increase and could possibly result in more low chlorine residual problems in the distribution system. As this process begins to occur with warmer weather, sampling will likely need to be ramped back to a weekly basis. Through this process of sampling and evaluating time series data from representative locations throughout the city, the success of disinfection within the distribution system (and resulting risk reduction) of having a measurable chlorine residual throughout, can be evaluated. Ensuring proper health protection through chlorine disinfection is an immediate and acute need. However, there are other more chronic unintended consequences of modifying chlorination practices. The most direct consequence of changing chlorination practices is the resulting change in disinfection byproducts (DBPs). Therefore, DBPs, specifically the regulated trihalomethanes and haloacetic acids, will be added to the sampling routine as needed to characterize the concentrations of DBPs through the system and assess whether additional remediation activities are required to keep DBPs below regulated thresholds. Finally, large changes in water chemistry, such as adding significantly higher concentrations of chlorine, can affect pipe scale and inorganic metals concentrations; therefore, prior to making significant changes to current practices, the EPA Task Force experts will be consulted to ensure that everyone agrees on proposed changes to treatment and distribution.

## References

- Great Lakes – Upper Mississippi River Board of State and Provincial Public Health and Environmental Managers, 2012. *Recommended Standards for Water Works*. Health Research Inc., Albany, NY.
- USEPA, 1989. Drinking Water; National Primary Drinking Water Regulations; Total Coliforms (Including Fecal Coliforms and E. Coli); Final Rule. *Federal Register*, 54:124:27544.
- Wahman, D.G. & Pressman, J.G., 2015. Distribution System Residuals-Is "Detectable" Still Acceptable for Chloramines? *Journal American Water Works Association*, 107:8:53.

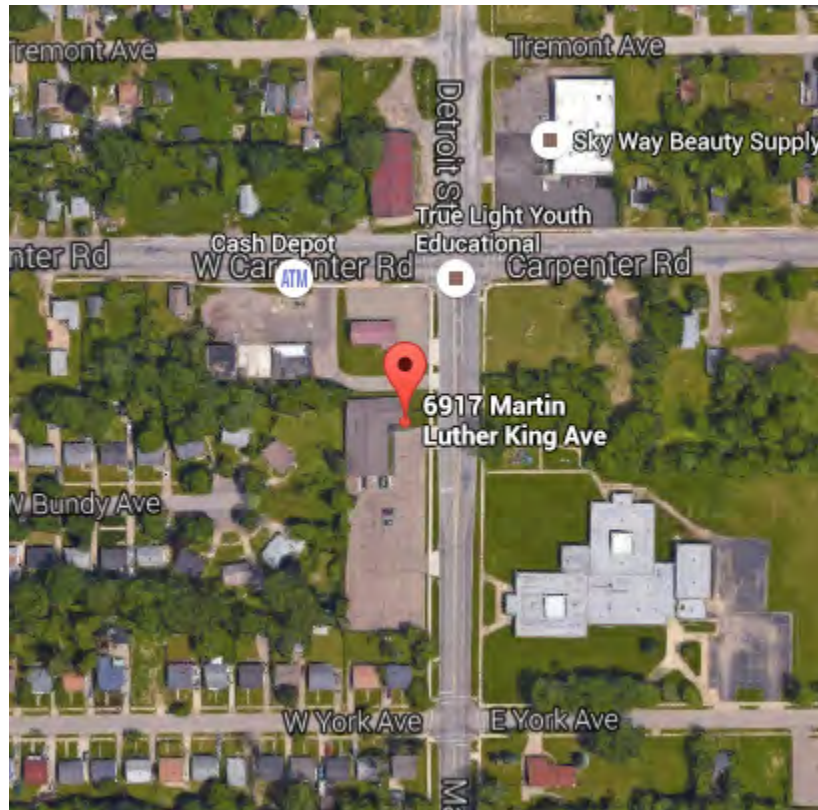


## SAMPLE SITE INFORMATION

### *Sample Site Location #MI (Monday 1<sup>st</sup> Stop)*

Property Name: True Light Missionary Baptist Church

Physical Address: 6917 Martin Luther King Ave, Flint, MI 48505



Contact: Reverend Robert Rushing

Contact Phone: 810-686-3707 (home) 810-624-5048 (mobile)

Site Availability: Monday 9:00am- 1:00pm and Thursday 11:00am-12:30pm

Special Instructions: None

Sample Location: Sink in Dining Hall Kitchen



Flush Time Estimation: 155 feet of 1" service line to meter connection inside the building. 50 feet of 3/4" line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm was 4 minutes. A flowrate of 3.4 gpm was measured and the flush time was adjusted to 2.4 minutes.

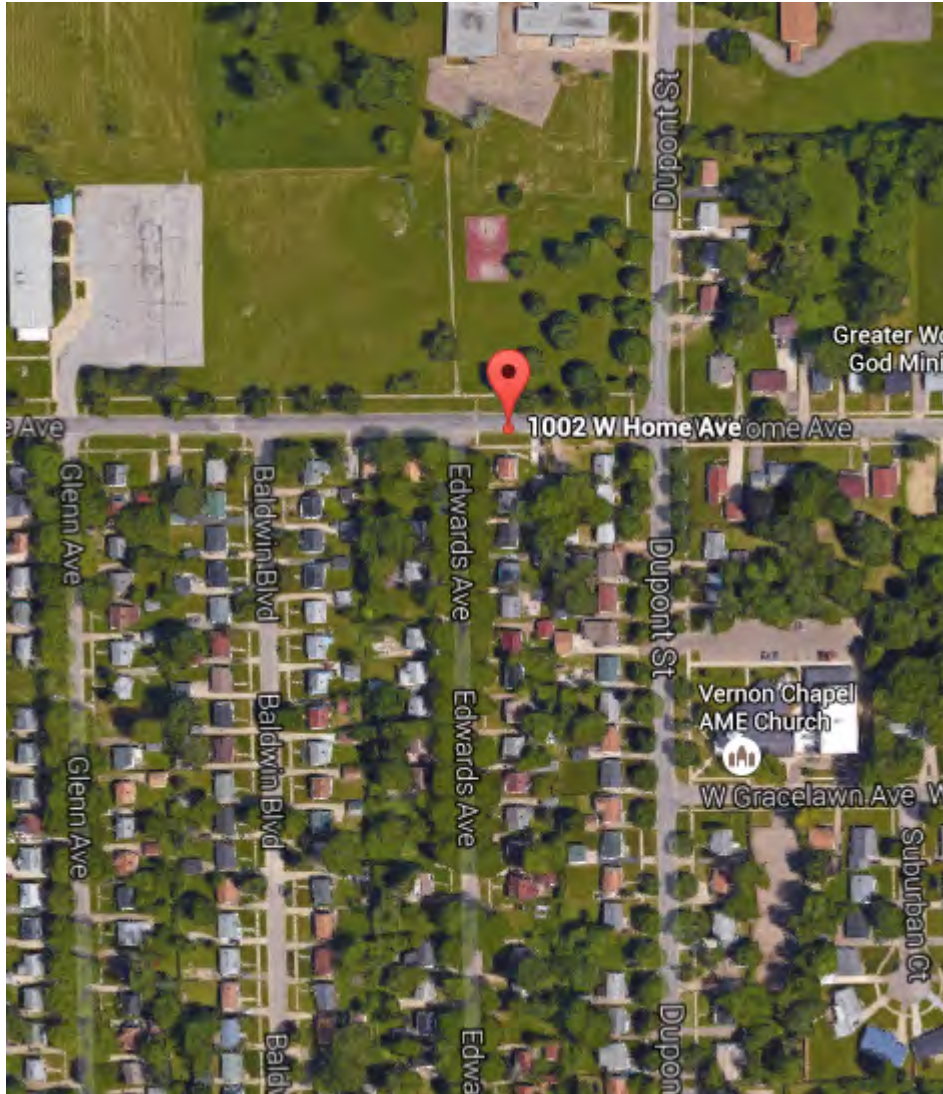
Estimated Flush Flowrate: 3.4 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #M2 (Monday 2<sup>nd</sup> Stop)***

Property: Hasselbring Senior Center

Physical Address: 1002 W. Home Ave, Flint, MI 48505



Contact: Latoya Jenkins

Contact Phone: 810-766-9516

Site Availability: Monday – Friday 8:00am- 5:00pm

Special Instructions: Call Vince at St. Luke's New Life Center (next stop) to give 30 minute notice of arrival

Sample Location: Kitchen Sink



Flushing Time Estimation: 190 feet of 2" service line to meter connection inside the building. 70 feet of ¾" line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm is 16.4 minutes. The measured flowrate was 3 gpm, so the flush time was adjusted to 11 minutes.

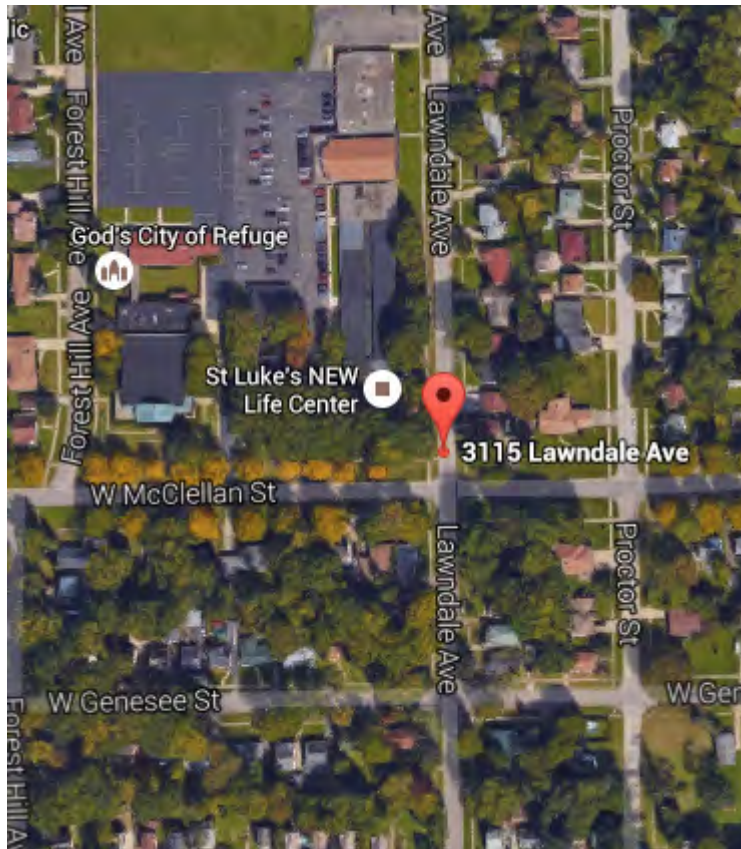
Estimated Flush Flowrate: 3 gpm

Required Sample Flush Time: 11 minutes

***Sample Site Location #M3 (Monday 3<sup>rd</sup> Stop)***

Property Name: St. Luke's New Life Center

Physical Address: 3115 Lawndale Ave Flint, MI 48504



Contact Name: Vince Vigil

Contact Phone: 810-239-8710

Site Availability: Monday – Friday 7:00am- 5:00pm

Special Instructions: Call Vince 30 minutes before arrival. Alternate contacts: Sister Julie or Sister Carol, 810-247-5414(m)

Sample Location: Bathroom Sink in Adult Literacy Program Office, Room 302



Flush Time Estimation: 30 feet of 2" service line to meter connection inside the building. 50 feet of ¾" line from the meter connection to the bathroom sink. Estimated time for flushing at 2 gpm flowrate was 10 minutes. The measured flowrate was 7.8 gpm, so the flush time was adjusted to 2.3 minutes.

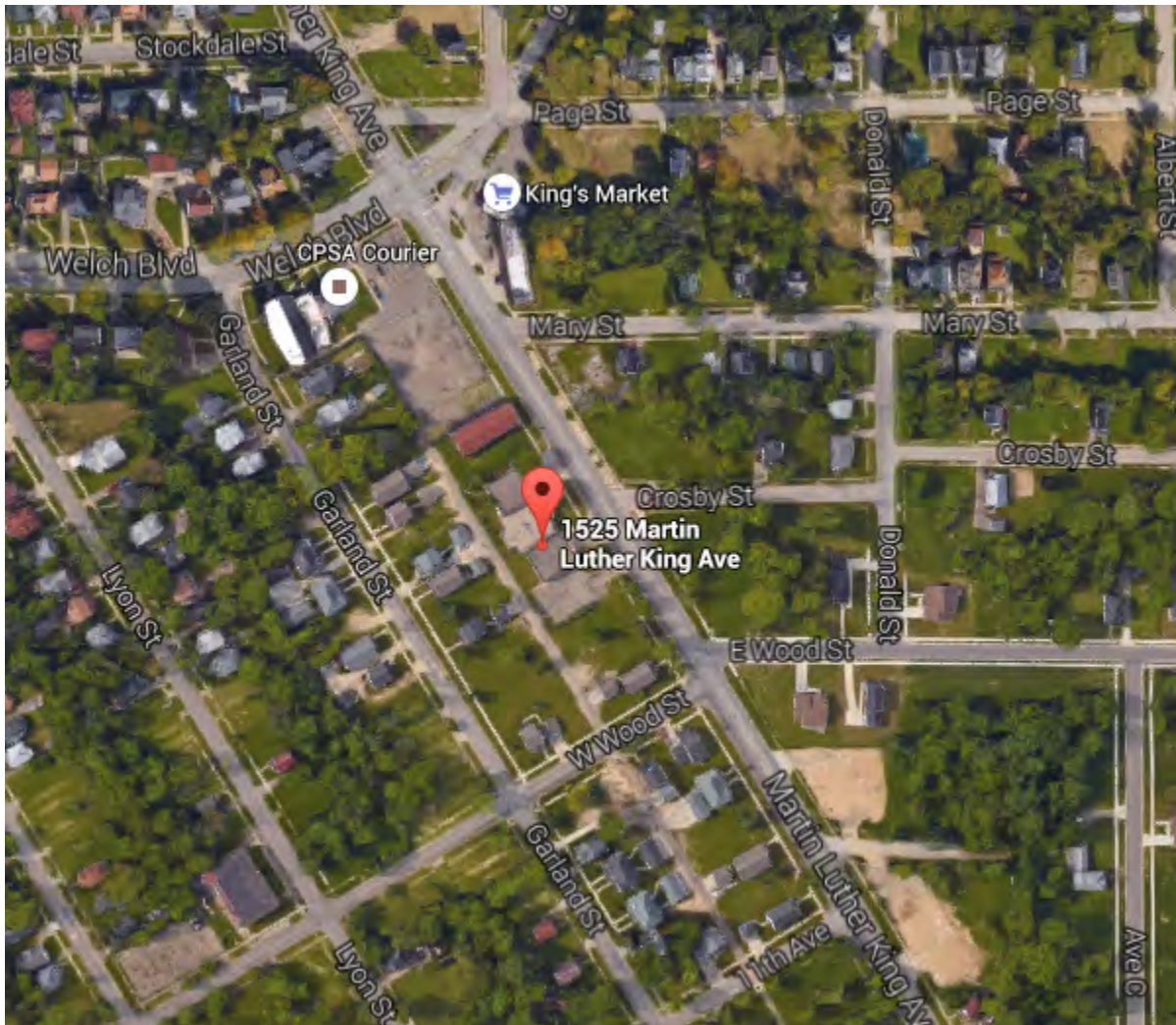
Estimated Flush Flowrate: 7.8 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #M4 (Monday 4<sup>th</sup> Stop)***

Property Name: Fire Station #3

Physical Address: 1525 Martin Luther King Avenue, Flint, MI 48503



Contact: Ask for “Officer of the Station”

Contact Phone Number: 810-762-7355

Site Availability: Daytime Hours

Special Instructions: Permission granted by District Commander Mark Kovach; access should be permitted as long as that is not rescinded

Sample Location: Public Bathroom Sink



Flush Time Estimation: 75 feet of 2" service line to meter connection inside the building. 45 feet of 1" line from the meter connection to the bathroom sink. Estimated time for flushing at 2 gpm is 7 minutes. The measured flowrate was 2.3 gpm, so the flush time was adjusted to 6 minutes.

Estimated Flush Flowrate: 2.3 gpm

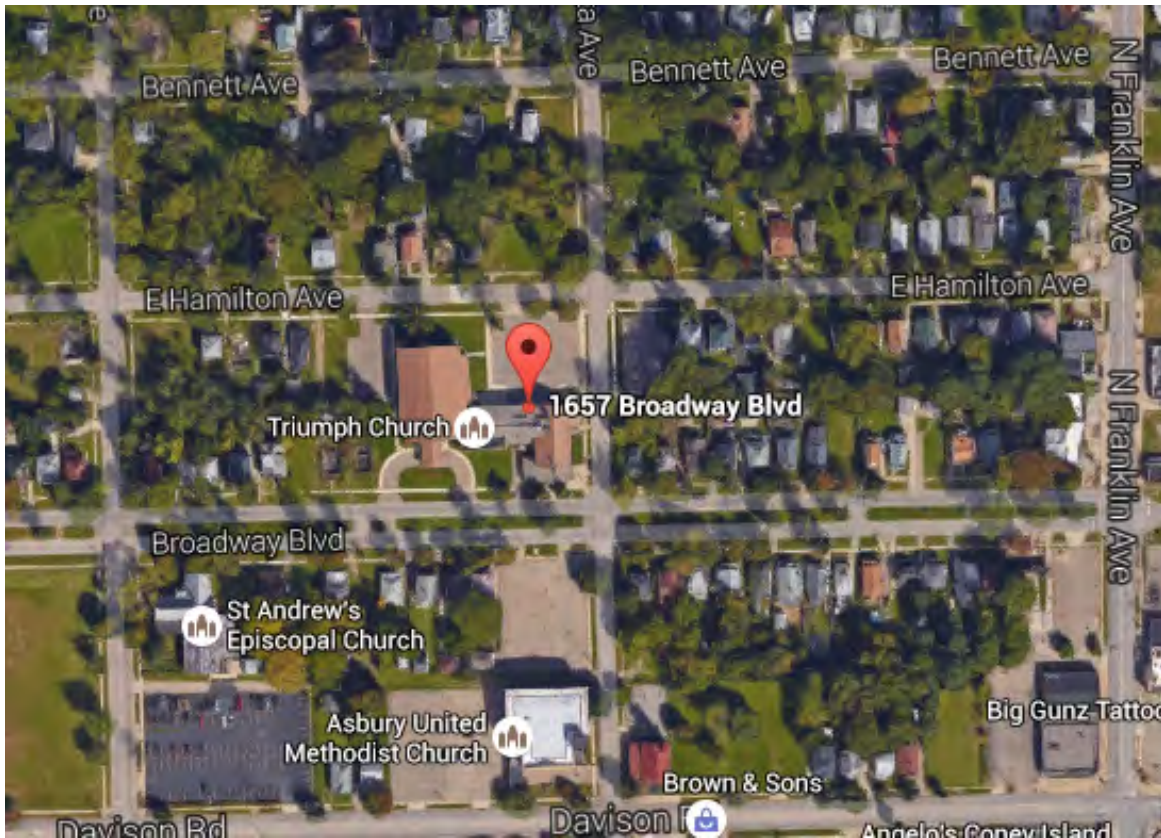
Required Sample Flush Time: 6 minutes



***Sample Site Location #M5 (Monday 5<sup>th</sup> Stop)***

Property Name: Triumph Church

Physical Address: 1657 Broadway Boulevard, Flint, MI 48506



Contact: Hettie Collins, Secretary

Contact Phone Number: 810-250-1892

Site Availability: Daytime Hours

Special Instructions: Alternate Contact(s)—Elder James Davis (810-210-2037)

Sample Location: Downstairs Kitchen Sink



Flush Time Estimation: 170 feet of 1" line, includes service line and premise plumbing from meter connection to downstairs kitchen sink. Estimated time for flushing at 2 gpm is 3.5 minutes. The measured flowrate was 2.6 gpm, so the flush time was adjusted to 2.7 minutes.

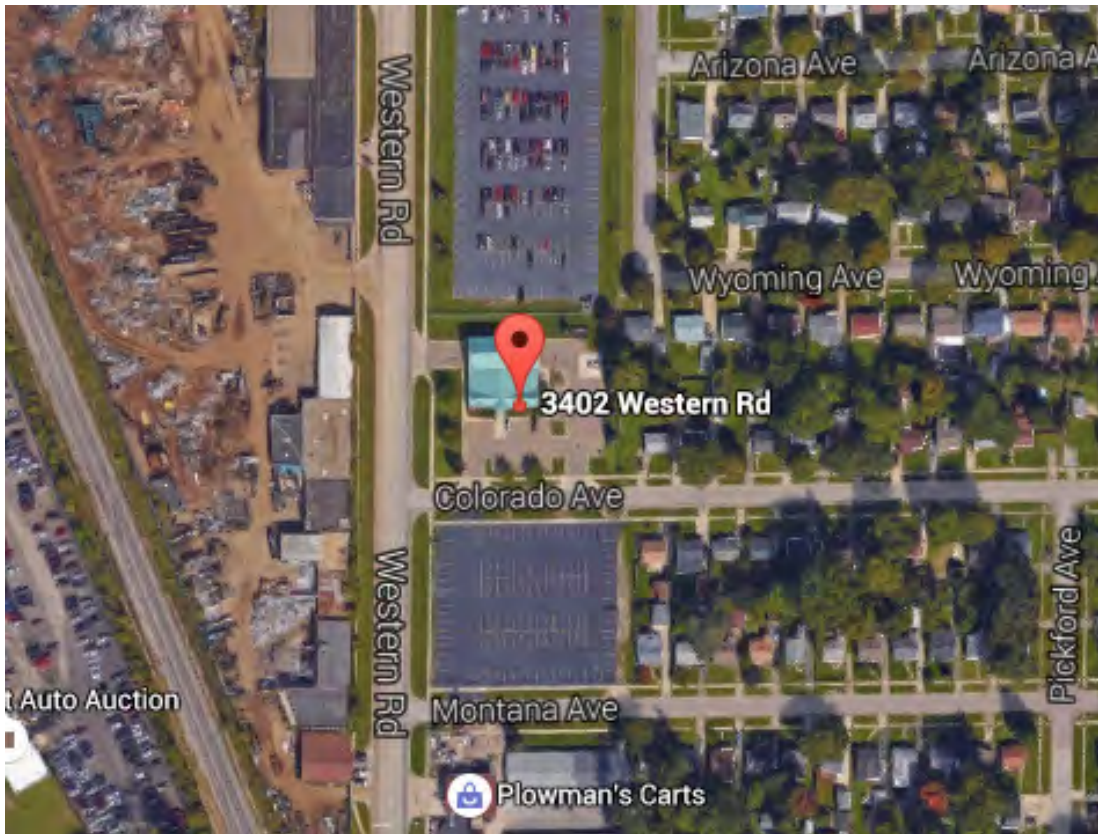
Estimated Flush Flowrate: 2.6 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #M6 (Monday 6<sup>th</sup> Stop)***

Property Name: Fire Station #5

Physical Address: 3402 Western Road, Flint, MI 48506



Contact: Ask for “Officer of the Station”

Contact Phone Number: 810-766-7331

Site Availability: Daytime Hours

Special Instructions: Lt. Logan helped us and is in charge of the station

Sample Location: Kitchen Sink



Flush Time Estimation: 130 feet of 2" service line to meter connection inside the building. 80 feet of 3/4" line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm is 12 minutes. The measured flowrate was 2.3 gpm, so the flush time was adjusted to 11 minutes.

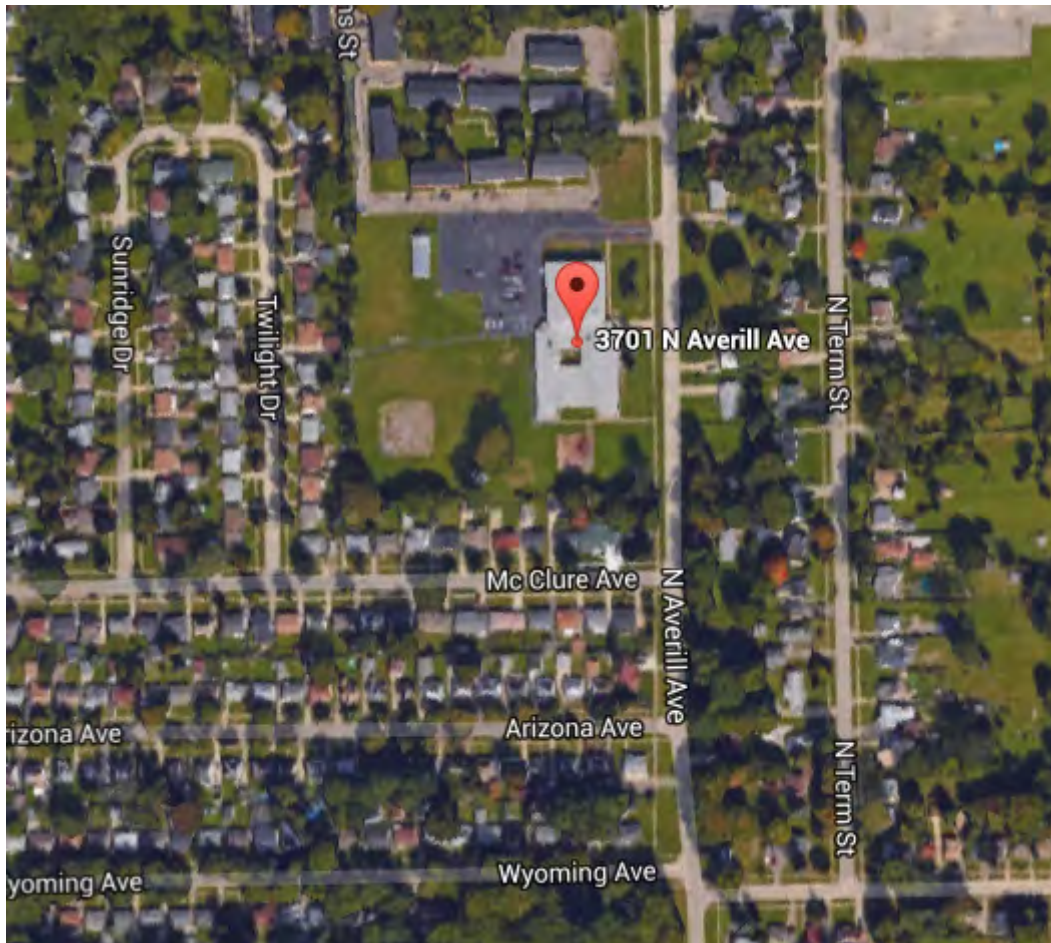
Estimated Flush Flowrate: 2.3 gpm

Required Sample Flush Time: 11 minutes

***Sample Site Location #M7 (Monday 7<sup>th</sup> Stop)***

Property Name: Boys and Girls Club of Flint

Physical Address: 3701 N. Averill Ave 48506



Contact: Shirley Hunter

Contact Phone: 810-249-3413

Site Availability: Monday – Friday generally okay after 9:00am

Special Instructions: Alternate contact is Amber Miller

Sample Location: Kitchen Sink



Flush Time Estimation: 150 feet of 3” service line to meter connection inside the building.

Estimated time for flushing at 2 gpm flowrate is 27.3 minutes. A flowrate of 3.5 gpm was measured and the flush time was adjusted to 16 minutes. Based on further testing and looking at the temperature profiles during flushing, the required flush time was adjusted to 30 minutes.

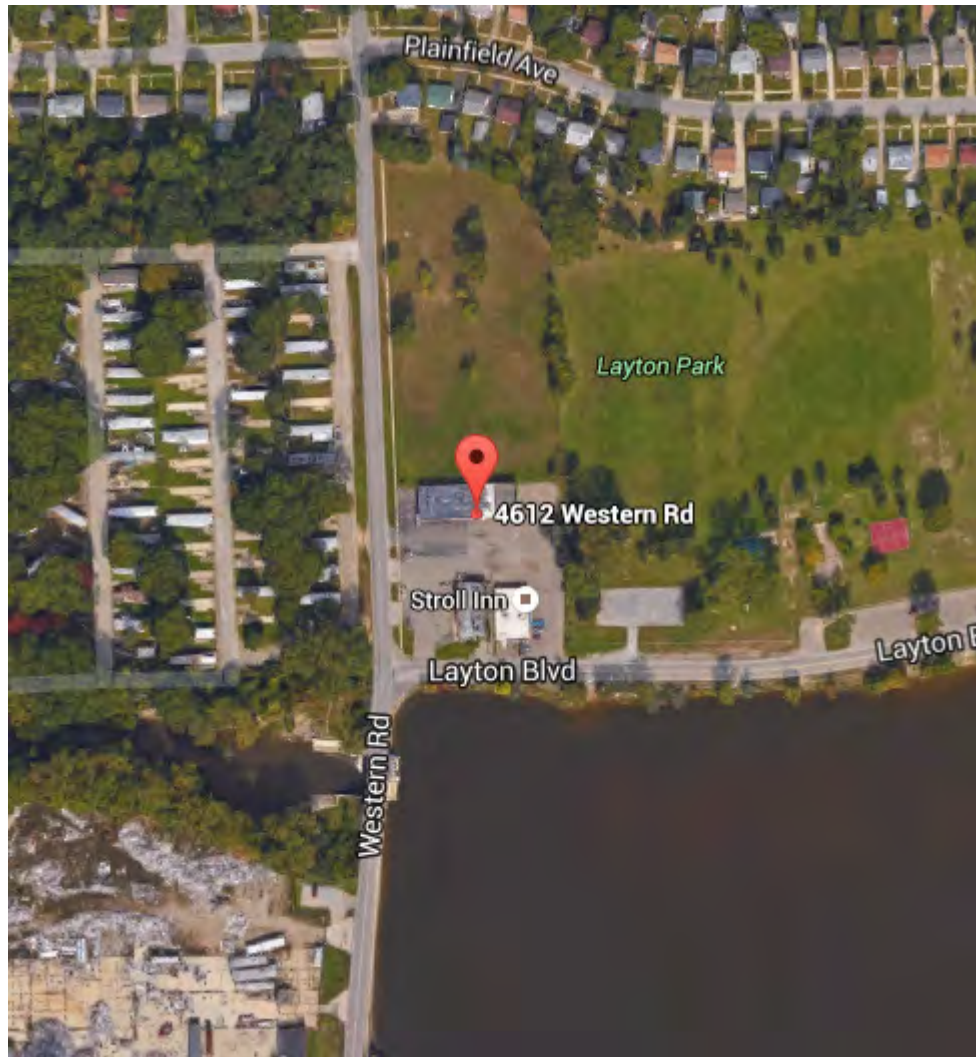
Estimated Flush Flowrate: 3.5 gpm

Required Sample Flush Time: 30 minutes

***Sample Site Location #M8 (Monday 8<sup>th</sup> Stop)***

Property Name: Sam's Rollingwood Market

Physical Address: 4612 Western Rd, Flint, MI 48506



Contact: Sam Mohamed

Contact phone: 810-736-6618, 810-265-6605 (cell)

Site Availability: 6am- 10pm daily

Special Instructions: None

Sample Location: Kitchen Sink



Flush Time Estimation: 45 feet of 4" service line to meter connection inside the building. 30 feet of ¾" line from the meter connection the kitchen sink. Estimated flush time at 2 gpm was 15 minutes. A flowrate of 5.2 gpm was measured and the flush time was adjusted to 6 minutes.

Estimated Flush Flowrate: 5.2 gpm

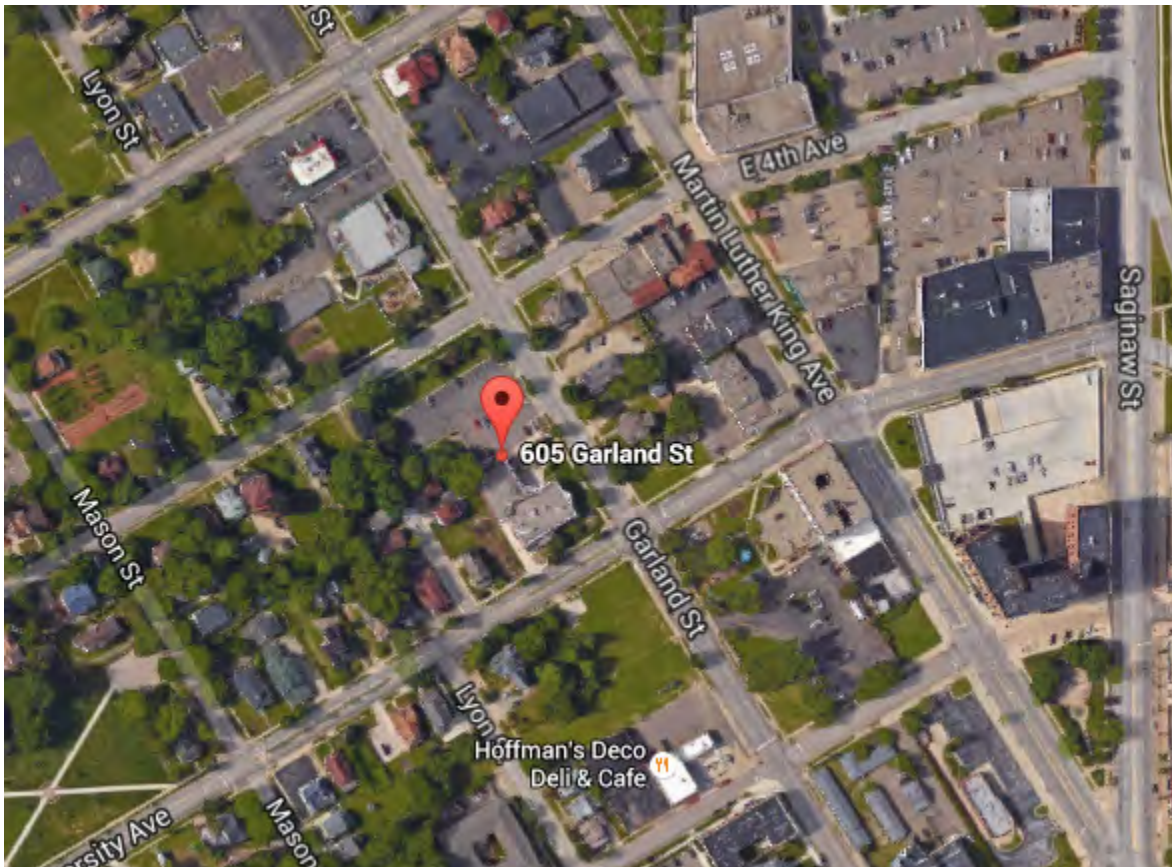
Required Sample Flush Time: 6 minutes



***Sample Site Location #M9 (Monday 9<sup>th</sup> Stop)***

Property Name: Carriage Town Ministries

Physical Address: 605 Garland Street, Flint, MI 48503



Contact: Nate Oliver (Food Service Director)

Contact Phone Number: 810-233-8787

Site Availability: Daytime hours

Special Instructions: None

Alternate Contact(s)—Marilyn Mayfield, Kitchen; Nick Gatlin, Director; Dallas Gatlin, Director

Sample Location: Kitchen Sink



Flush Time Estimation: 40 feet of 2" service line to meter connection inside the building. 30 feet of 1" line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm is 4 minutes. A flowrate of 7.9 gpm was measured and the flush time was adjusted to 1 minutes.

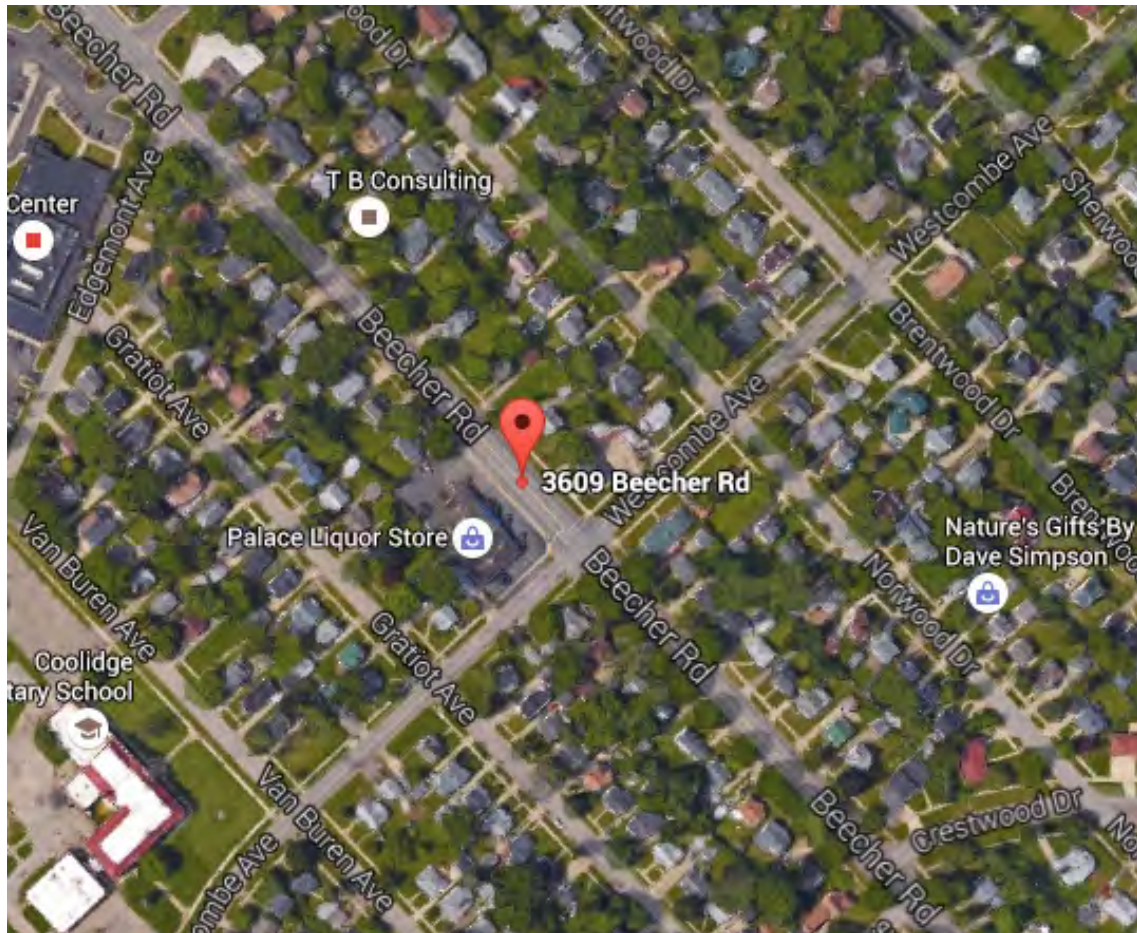
Estimated Flush Flowrate: 7.9 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #M10 (Monday 10<sup>th</sup> Stop)***

Property Name: Palace Liquor Store

Physical Address: 3609 Beecher Road, Flint, MI 48503



Contact: Sue and Dave Najor

Contact Phone Number: 810-235-1935

Site Availability: Weekdays 11 a.m. to Midnight

Special Instructions: Alternate Contact(s)—Chris Gonzales helped us

Sample Location: Kitchen Sink



Flush Time Estimation: 100 feet of 2" service line to meter connection inside the building. 50 feet of 1" line (assumed) from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm is 10 minutes. A flowrate of 2.9 gpm was measured and the flush time was adjusted to 7 minutes.

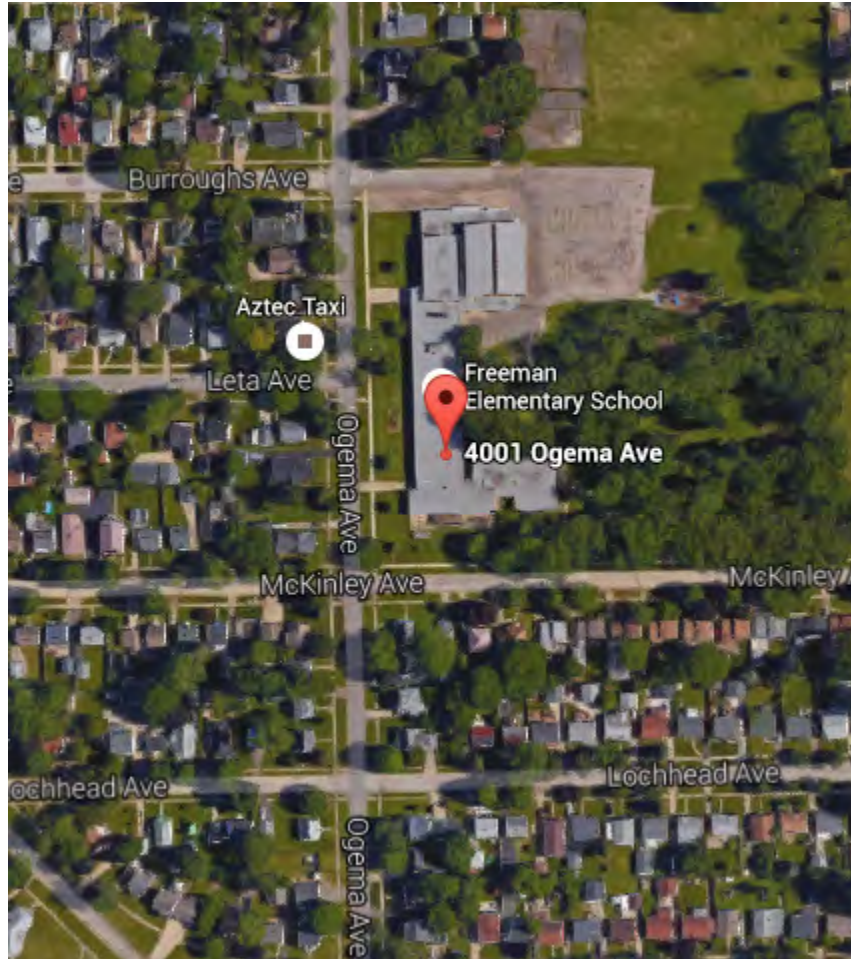
Estimated Flush Flowrate: 2.9 gpm

Required Sample Flush Time: 7 minutes

***Sample Site Location #T1 (Tuesday 1<sup>st</sup> Stop)***

Property Name: Freeman School

Physical Address: 4001 Ogema Ave, Flint, MI 48507



Contact Name: Tony Sitko

Contact Phone: 810-767-6030

Site Availability: Day must be arranged. Additional contacts for scheduling are Scott Rowell (master plumber) 989-413-0110 and Mike Loll (maintenance supervisor) 810-293-9249.

Special Instructions: Pre-arrange day with Tony. Use hose during flushing to floor drain and remove hose prior to sampling. Need access to room.

Sample Location: Tap on service line in boiler room.



Flush time estimation: 125 feet of 4" service line to meter connection inside the building.

Estimated time for flushing at 2 gpm was 40 minutes. The measured flowrate was 16.2 gpm, so the flush time was adjusted to 5 minutes.

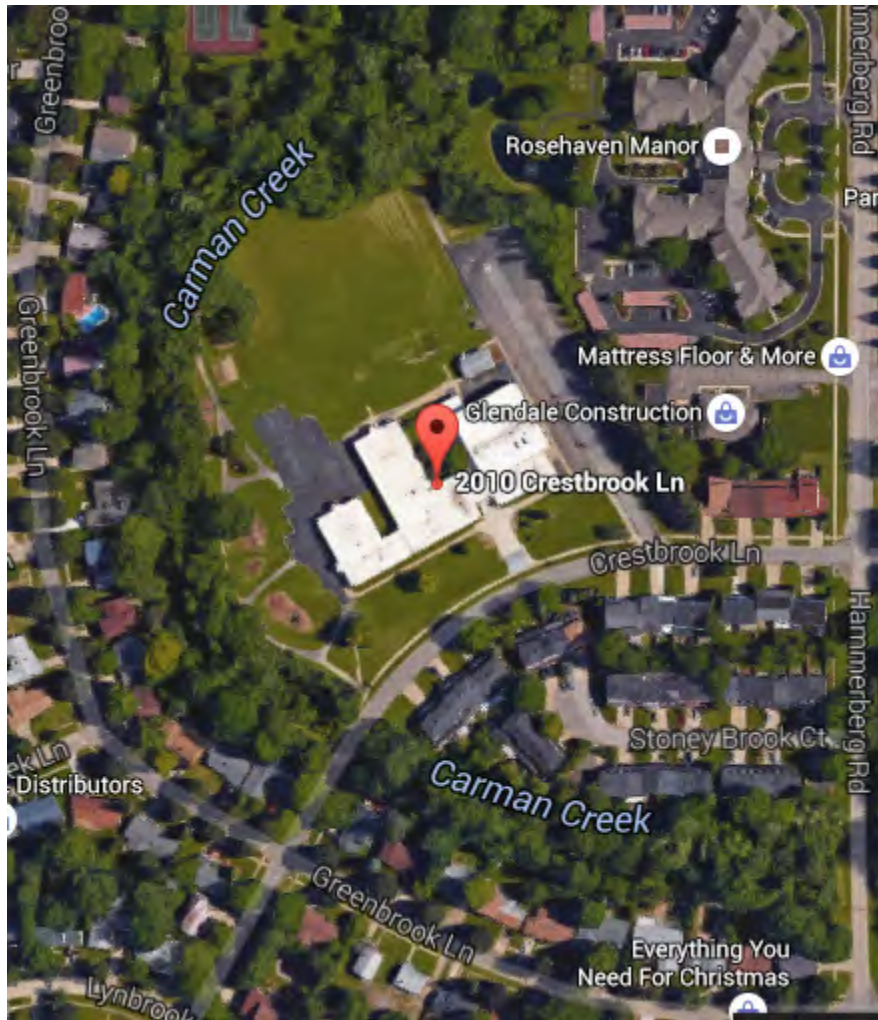
Estimated Flush Flowrate: 16.2 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #T2 (Tuesday 2<sup>nd</sup> Stop)***

Property Name: Neithercut Elementary

Physical Address: 2010 Crestbrook Ln, Flint, MI 48507



Contact Name: Tony Sitko

Contact Number: 810-767-6030

Site Availability: Day must be arranged. Additional contacts for scheduling are Scott Rowell

(master plumber) 989-413-0110 and Mike Loll (maintenance supervisor) 810-293-9249.

Special Instructions: Pre-arrange day with Tony Sitko

Sample Location: Janitor Sink next to Main Entrance (disconnect hose prior to flushing and sampling)



Flush Time Estimation: 200 feet of 4" line, includes service line to meter connection and length of line inside building. 10 feet of 3/4" line from 4" line to janitor sink. Estimated time for flushing at 2 gpm was 65 minutes. The measured flowrate was 5 gpm, so the flush time was adjusted to 25 minutes.

Estimated Flush Flowrate: 5 gpm

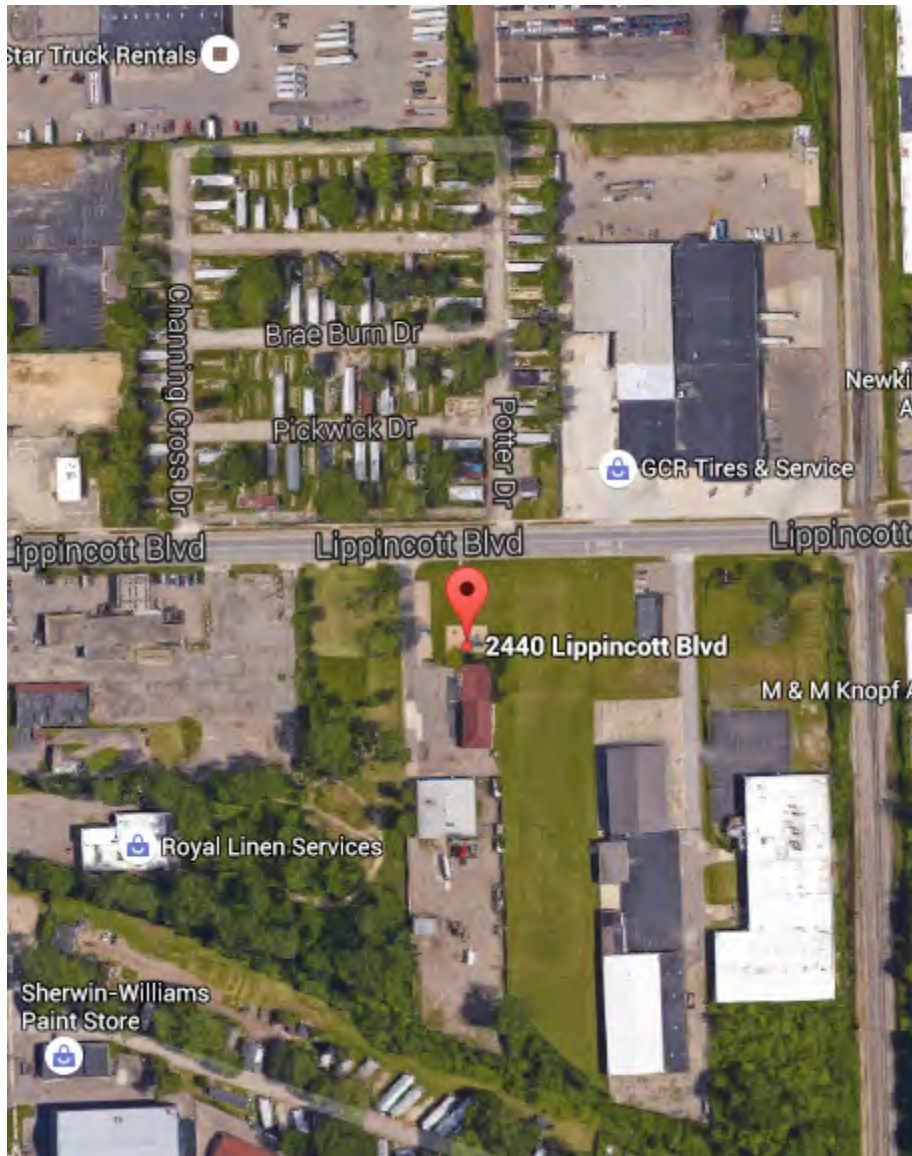
Required Sample Flush Time: 25 minutes



***Sample Site Location #T3 (Tuesday 3<sup>rd</sup> Stop)***

Property Name: Gloria's Little Angels Children

Physical Address: 2440 Lippincott Blvd, Flint, MI 48507



Contact: Venita Hiler

Contact Phone: 810-424-9649

Site Availability: Any time before 6:30pm

Special Instructions: None

Sample location: Kitchen Sink



Flush Time Estimate: 190 feet of 4" service line to meter connection inside the building. 25 feet of 1/2" line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm flowrate is 62 minutes. Because of the long flush time required, both sinks in the kitchen were turned on to reduce the flush time. The measured flowrate combined from both sinks was 4 gpm, so the flush time was adjusted to 30 minutes.

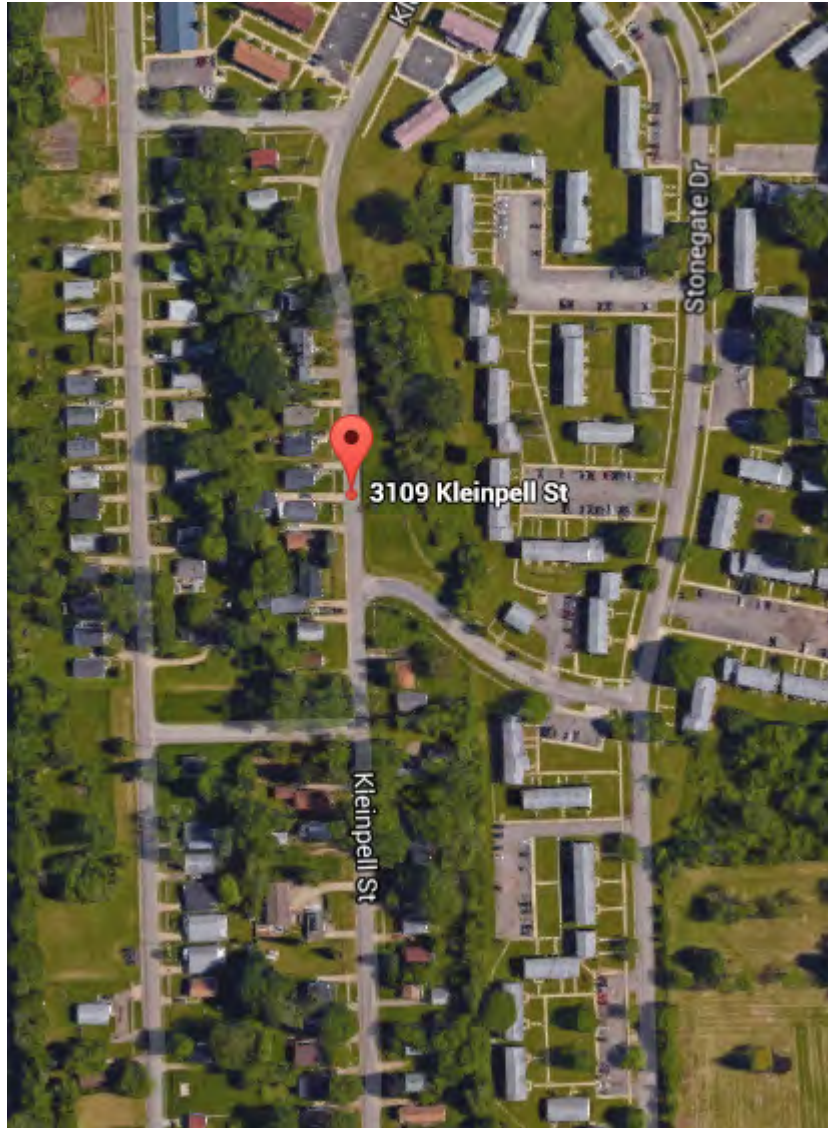
Estimated Flush Flowrate: 4 gpm with both sinks running

Required Sample Flush Time: 30 minutes

***Sample Site Location #T4 (Tuesday 4<sup>th</sup> Stop)***

Property Name: Genesee Community Health Center- Atherton

Physical Address: 3109 Kleinpell Flint, MI 48507



Contact: Tammy Kenebrew

Contact Phone Number: 810-496-5542 (office) 810-845-8259 (cell)

Site Availability: 8:00am- 4:30pm daily (end of day is better)

Special Instructions: Alternate contact: Patty Wagonhauser 810-496-5542

Sample Location: Laboratory Sink (go left down the hallway, last door on right)



Flush time Estimation: 35 feet of 2" service line to meter connection inside the building. 25 feet of ½" line from the meter connection to the laboratory sink. Estimated time for flushing at 2 gpm is 3 minutes. The measured flowrate was 1.9 gpm, so the flush time was adjusted to 3.1 minutes.

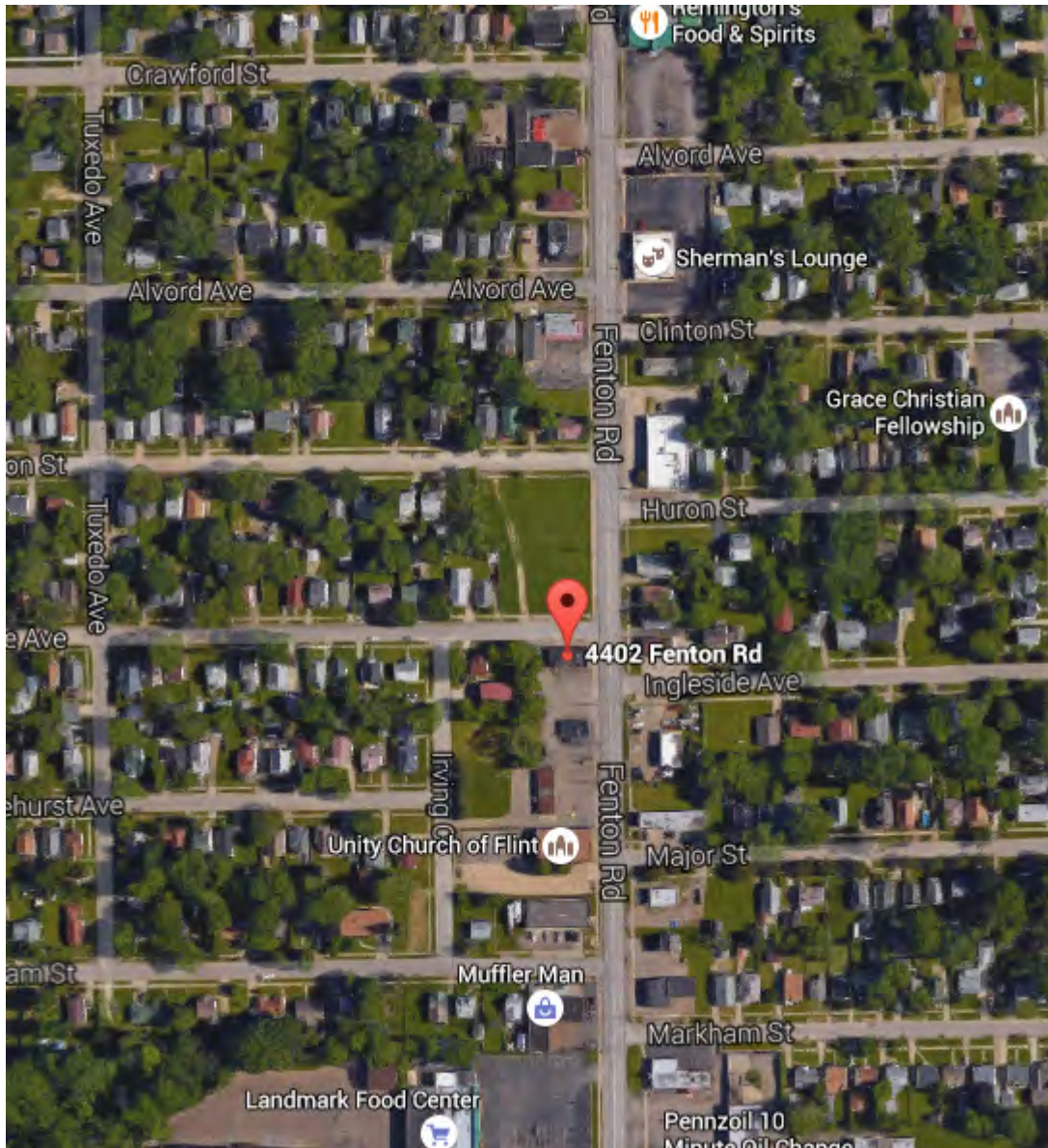
Estimated Flush Flowrate: 1.9 gpm

Required Sample Flush: 5 minutes

***Sample Site Location #T5 (Tuesday 5<sup>th</sup> Stop)***

Property Name: Domino's Pizza (Fenton Road)

Physical Address: 4402 Fenton Rd, Flint, MI 48507



Contact: Rob VanBoren

Contact Phone: 810-234-5333

Site Availability: 10:30am – 12:00am Daily

Special Instructions: Use connected hose during flushing and then remove hose for sampling

Sample Location: Mop Sink in Utility Area



Flush Time Estimation: 110 feet of  $\frac{3}{4}$ " line, including distance of service line to meter connection and line to mop sink inside building. Estimated time for flushing at 2 gpm flowrate was 1.2 minutes. The measured flowrate was 5.3 gpm, so the flush time was adjusted to 0.5 minutes.

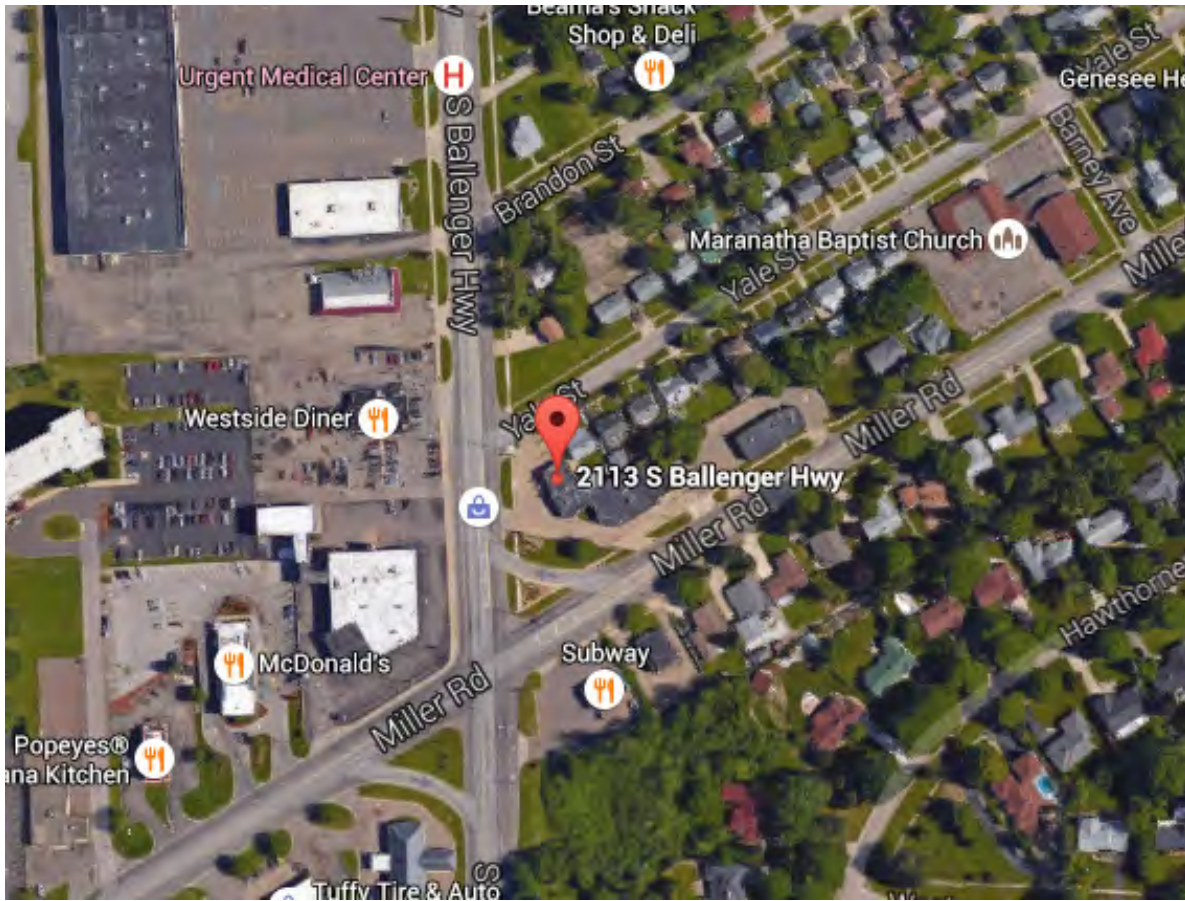
Estimated Flush Flowrate: 5.3 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #T6 (Tuesday 6<sup>th</sup> Stop)***

Property Name: Domino's Pizza (Ballenger Highway)

Physical Address: 2113 S. Ballenger Highway, Flint, MI 48503



Contact: Will Dieke, General Manager

Contact Phone Number: 810-341-1111

Site Availability: Weekdays 10:30 a.m. to Midnight

Special Instructions: Alternate Contact(s)—Robert Kasgorgis, Owner (248-701-8074)

Sample Location: Hand Wash Sink



Flush Time Estimation: 70 feet of 1.5" service line to meter connection inside the building. 20 feet of 3/4" line from the meter connection to the hand wash sink. Estimated time for flushing at 2 gpm is 3.5 minutes. The measured flowrate was 2.2 gpm, so the flush time was adjusted to 3.2 minutes.

Estimated Flush Flowrate: 2.2 gpm

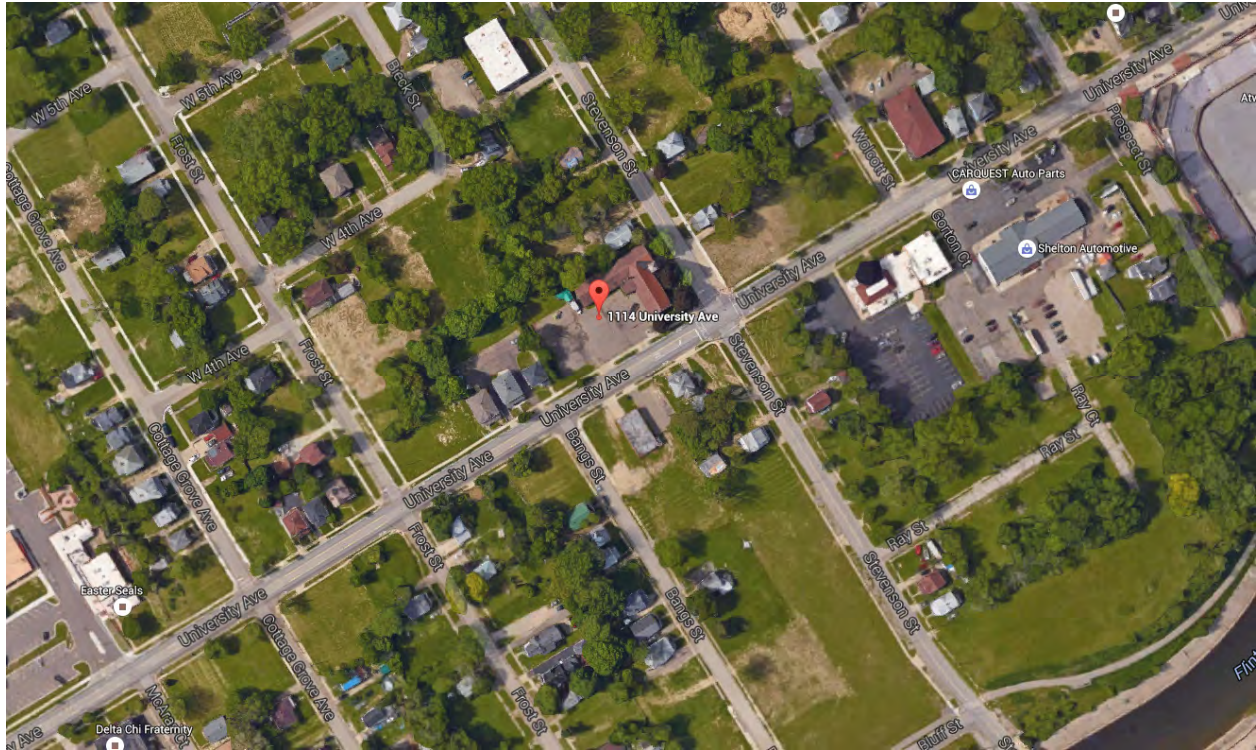
Required Sample Flush Time: 5 minutes



***Sample Site Location #T7 (Tuesday 7<sup>th</sup> Stop)***

Property Name: Greater Flint Outreach Center

Physical Address: 1114 University Avenue, Flint, MI 48504



Contact: Phyllis Ott

Contact Phone Number: 810-767-4064

Site Availability: Monday, Tuesday, Thursday, and Friday 9:15 AM – 3:00 PM

Special Instructions: If door is locked, call Phyllis.

Alternate contact: Pastor Rottiers, 810-240-5324

Sample Location: Kitchen Sink in Basement



Flush Time Estimation: 25 feet of 2" service line was reduced to a 5/8" meter connection inside the building. 50 feet of 1" line from the meter connection to the kitchen sink in the basement. Estimated time for flushing at 2 gpm is 3 minutes. The measured flowrate was 7.9 gpm, so the flush time was adjusted to 0.3 minutes.

Estimated Flush Flowrate: 7.9 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #T8 (Tuesday 8<sup>th</sup> Stop)***

Property Name: Christ Enrichment Center

Physical Address: 322 E. Hamilton Avenue, Flint, MI 48505



Contact: Shan Buckley

Contact Phone Number: 810-233-8787

Site Availability: M, T, W, Th; 9 a.m. to 3 p.m.

Sample Location: Upstairs Men's Room



Flush Time Estimation: 100 feet of 2" service line to meter connection inside the building. 30 feet of 1" line from the meter connection to the upstairs men's restroom. Estimated time for flushing at 2 gpm is 9.5 minutes. The measured flowrate was 1.4 gpm, so the flush time was adjusted to 15 minutes.

Estimated Flush Flowrate: 1.4 gpm

Required Sample Flush Time: 15 minutes

***Sample Site Location #T9 (Tuesday 9<sup>th</sup> Stop)***

Property Name: Sunny Patch Learning Center

Physical Address: 1160 Donaldson Street, Flint, MI 48504



Contact: Annette Hughes, Director

Contact Phone Number: 810-235-2444

Site Availability: Weekdays 8 a.m. to 3 p.m.

Special Instructions: Alternate Contact(s)—Fadrika Nash, Asst. Director; Teresa Vanover, Staff

Member

Sample Location: Kitchen Sink



Flush Time Estimation: 65 feet of  $\frac{3}{4}$ " service line to meter connection inside the building. 20 feet of  $\frac{3}{4}$ " line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm is 1 minute. The measured flowrate was 1.4 gpm, so the flush time was adjusted to 1.5 minutes.

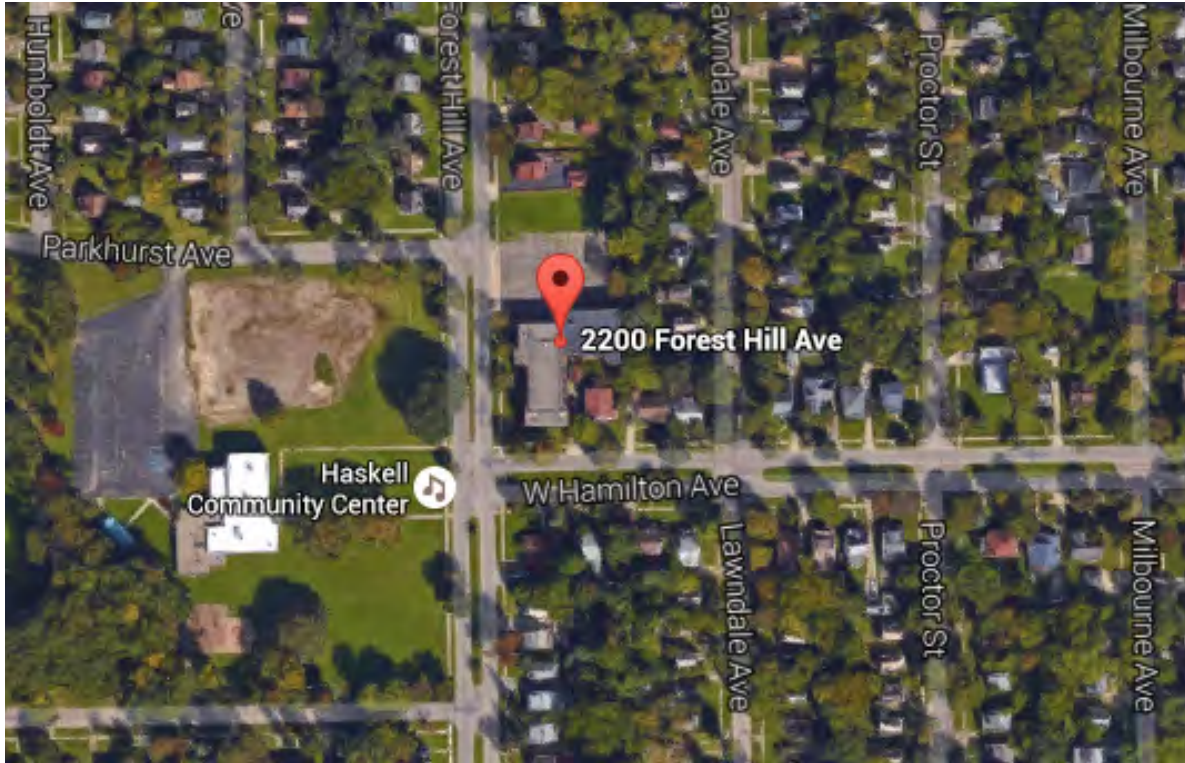
Estimated Flush Flowrate: 1.4 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #W1 (Wednesday 1<sup>st</sup> Stop)***

Property Name: Saints of God Church

Physical Address: 2200 Forest Hill Avenue, Flint, MI 48504



Contact: Pastor Harris

Contact Phone Number: 810-394-6787

Site Availability: W 9 a.m. to 11:30 a.m. (best) or 6:30 p.m. to 8 p.m.

Special Instructions: None

Sample Location: Kitchen Sink



Flush Time Estimation: 90 feet of 2" service line to meter connection inside the building. 10 feet of 1" line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm is 8 minutes. The measured flowrate was 3 gpm, so the flush time was adjusted to 6 minutes.

Estimated Flush Flowrate: 3 gpm

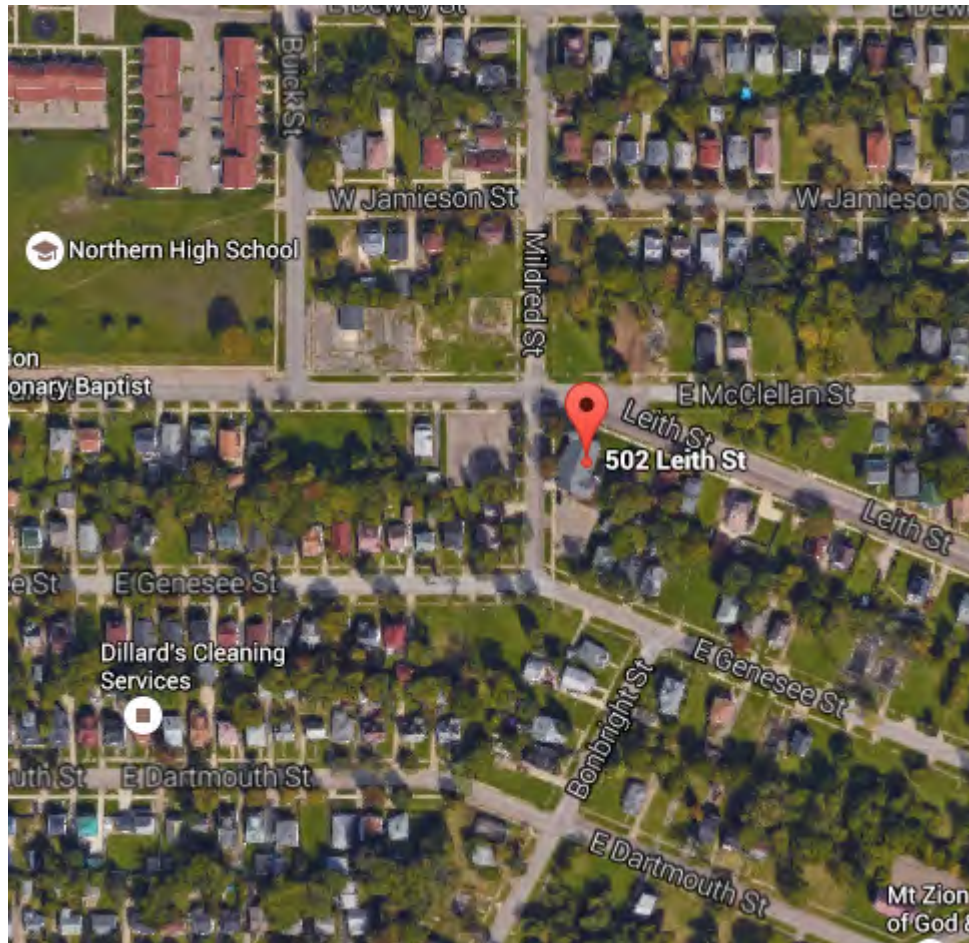
Required Sample Flush Time: 6 minutes



***Sample Site Location #W2 (Wednesday 2<sup>nd</sup> Stop)***

Property Name: Shiloh Missionary Baptist Church of Flint

Physical Address: 502 Leith St, Flint, MI 48505



Contact: Pam Gadson

Contact Phone: 810-813-3256 (cell) 810-785-6271 (church)

Site Availability: Monday and Wednesday 9:00amd – 12:00pm

Special Instructions: Pastor is Daniel Moore 810-471-2116.

Sample Location: Left Faucet in Kitchen Sink



Flush Time Estimation: 85 feet of  $\frac{3}{4}$ " service line to meter connection inside the building. 40 feet of  $\frac{1}{2}$ " line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm was 1.2 minutes. The measured flowrate was 6.3 gpm, so the flush time was adjusted to 0.5 minutes.

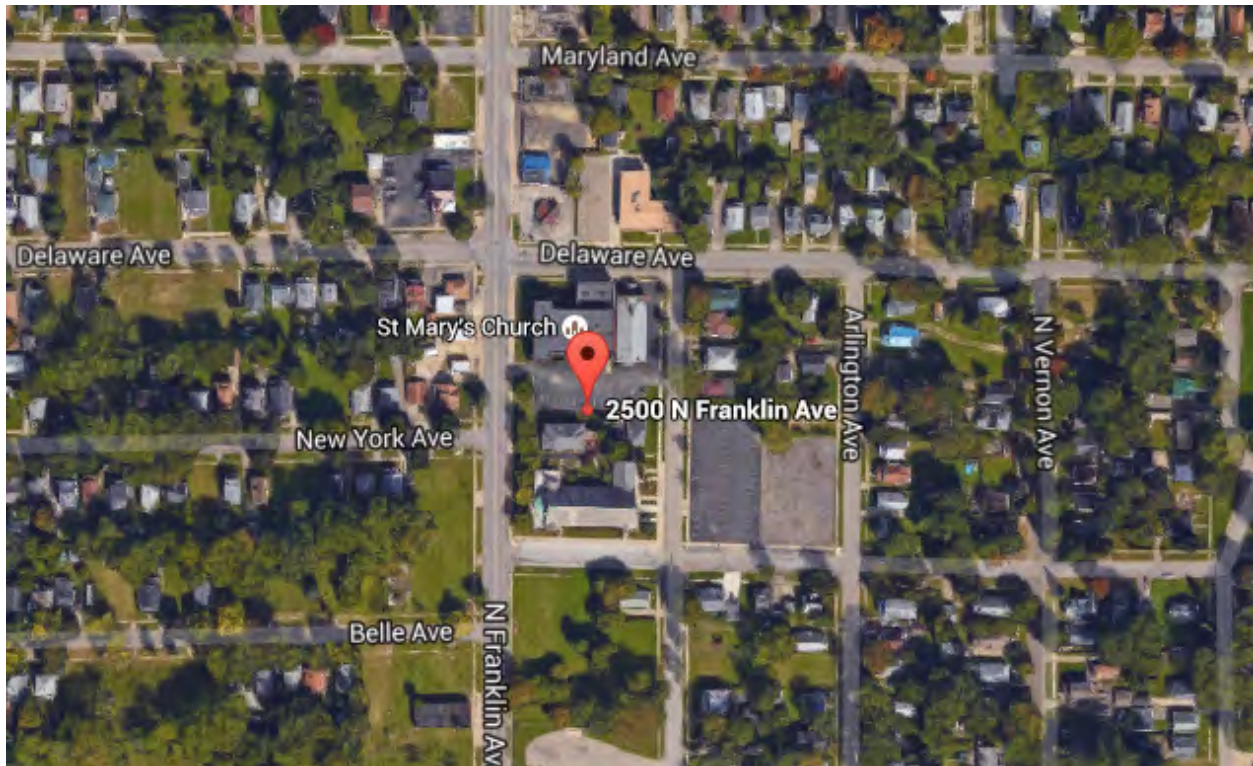
Estimated Flush Flowrate: 6.3 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #W3 (Wednesday 3<sup>rd</sup> Stop)***

Property Name: St. Mary's Church

Physical Address: 2500 N. Franklin Avenue, Flint, MI 48506



Contact: Kathy Tomczyk

Contact Phone Number: 810-232-4012

Site Availability: Daytime M, W, Th, F

Special Instructions: None

Sample Location: Kitchen Sink



Flush Time Estimation: 140 feet of 1" service line to meter connection inside the building. 20 feet of 3/4" line from the meter connection to the kitchen sink. Estimated time for flushing at 2 gpm is 3 minutes. The measured flowrate was 1.9 gpm, so the flush time was adjusted to 3.2 minutes.

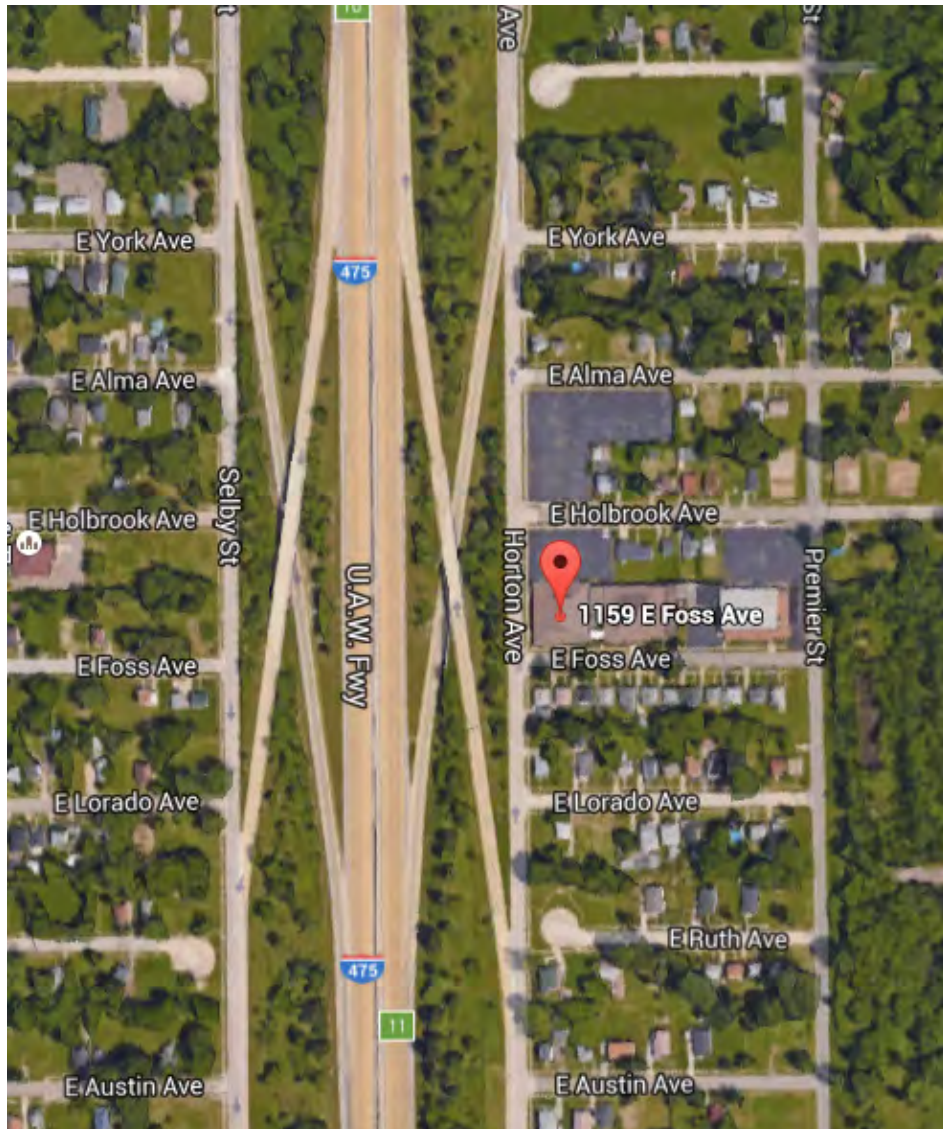
Estimated Flush Flowrate: 1.9 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #W4 (Wednesday 4<sup>th</sup> Stop)***

Property Name: Foss Avenue Baptist Church

Physical Address: 1159 E. Foss Ave, Flint, MI 48505



Contact: Alby Winfrey

Contact Phone: 810-787-9019

Site Availability: Tuesday- Friday 9:00am-5:00pm

Special Instructions: Jackie Scott is the secretary

Sample Location: Woman's Restroom (near E. Foss Avenue entrance, fourth sink from entrance)



Flush Time Estimation: 30 feet of 4" service line to meter connection inside the building. 30 feet of 3/4" line from them meter connection to the fourth sink from the entrance in the women's restroom. Estimated time for flushing at 2 gpm was 10 minutes. The measured flowrate was 4 gpm, so the flush time was adjusted to 5 minutes.

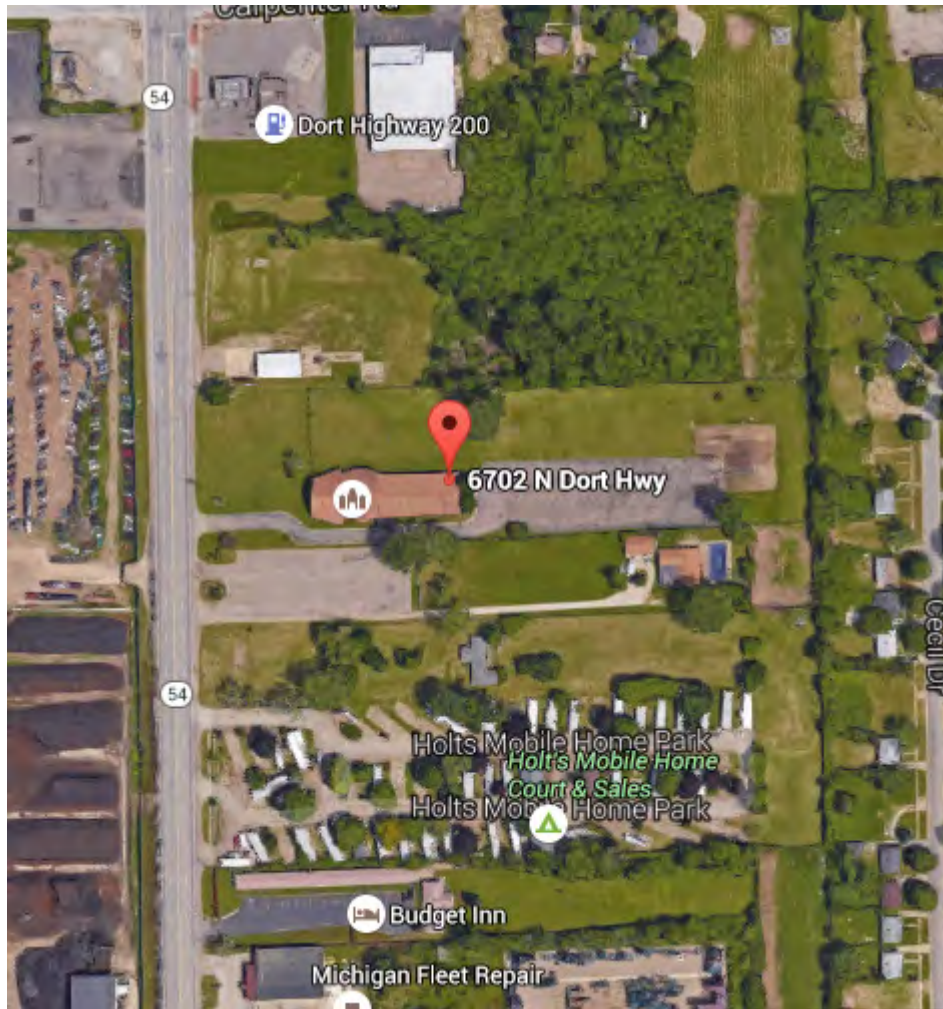
Estimated Flush Flowrate: 4 gpm

Required Sample Flush Time: 5 minutes

***Sample Site Location #W5 (Wednesday 5<sup>th</sup> Stop)***

Property Name: Greater Holy Temple Ministries Church of God in Christ

Physical Address: 6702 N. Dort Hwy, Flint, MI 48505



Contact: Sandra Jones

Contact Phone: 832-428-4887

Site Availability: Tuesday, Wednesday, or Thursday 11:00am to 3:00pm

Special Instructions: Bishop's name is Roger Jones

Sample Location: sink on side of pulpit



Flush Time Estimation: 200 feet of 2" service line to meter connection inside the building. 100 feet of ¾" line from the meter connection to the laboratory sink. Estimated time for flushing at 2 gpm is 17.5 minutes. A flowrate of 4.5 gpm was measured and the flush time was adjusted to 8 minutes.

Estimated Flush Flowrate: 4.5 gpm

Required Sample Flush: 8 minutes



## **Appendix B-2 – TTHM and HAA9 Sampling Instructions**

## **SAMPLING INSTRUCTIONS**

**for**

### **TRIHALOMETHANES and HALOACETIC ACIDS**

#### TRIHALOMETHANES (THMs)

1. When sampling from a water tap, open the tap and allow the water to flow until the water temperature has stabilized (Usually about 3-10 minutes).
2. Adjust the flow to about 500 mL/minute and collect three vials of sample.
3. Each vial contains a Sodium Thiosulfate preservative, so do not rinse the vial. Fill the vials to overflowing but take care that the preservative is not rinsed out.
4. No air bubbles should pass through the sample as the vial is being filled or be trapped in the samples when the vial is sealed.
5. After sealing, the vial should be inverted a few times to make sure no bubbles have been trapped and to mix the preservative with the sample.
6. Samples must be chilled during storage and shipment to about 4°C and maintained at that temperature until analysis. Crushed ice should be used as blue ice typically cannot maintain a temperature of 4° C for an extended period of time.

#### HALOACETIC ACIDS (HAAs)

1. Fill sample bottles but take care not to flush out the ammonium chloride which, for a 250 mL bottle, is 25 mg of Ammonium Chloride dissolved in a small amount of water. Because the target analytes of this method are not volatile, it is not necessary to ensure that the sample bottles are completely headspace free.
2. When sampling from a water tap, remove the aerator. Open the tap and allow the system to flush until the water temperature has stabilized (usually about 3-10 minutes). Collect samples from the flowing system.
3. When sampling from an open body of water, fill a 1-quart wide-mouth bottle or 1 liter beaker with sample from a representative area, and carefully fill sample vials from the container.
4. After collecting the sample, seal the bottle and agitate by hand for 15 seconds.
5. Samples must be chilled to 10° C during storage and shipment. Crushed ice should be used as blue ice typically cannot maintain a temperature of 10° C for an extended period of time.

## Appendix C – Lead Source/Release Diagnostic Evaluation

As discussed in Section 5.3, sequential water sampling will be conducted initially at approximately 100 sampling sites, then on an ongoing basis at approximately 45 single-family residences. Procedures for subsequent rounds of sequential sampling may be tailored for each single-family residence, based on available information including analytical results and plumbing information. In addition, smaller bottle sizes (e.g., 500-mL) may be used to further evaluate potential sources of lead in interior plumbing.

Field sampling coordinators will confirm the schedule with the applicable resident(s) in advance of sampling activities. At each sampling site, the kitchen sink faucet will be identified, where water is drawn for human consumption (drinking, cooking, formula preparation, etc.). For the first round of sequential sampling, any existing filter must be removed from the faucet prior to flushing or sampling. For the second round of sequential sampling, existing filters will be bypassed, rather than removed, to assess whether high results are from lead sources in specific locations or from sporadically released particulate from unknown locations. At least 6 hours prior to sampling (i.e., the night before), the resident will flush the sample tap for 5 minutes and then assure that water is not used overnight. The resident will be asked to record the time at which flushing was completed and when water was last used in the home (i.e., no faucets, toilets, shower, bath tub, washing machine, dishwasher, and hose), and field sampling coordinators will record the total stagnation time in the field records. Appendices C-1 and C-2 provide example forms for communication with residents during scheduling and upon mobilization to the sampling site.

After at least 6 hours of stagnation time, two 125-mL water samples and then approximately 15 sequential 1,000-mL water samples will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals including lead [tin will be added to the list of parameters to be analyzed starting with second round of sequential sampling in May 2016]. The total number of 125-mL, 500-mL, and 1,000-mL water samples will be selected by the field sampling coordinators, depending on the lengths and diameters of plumbing materials to the water main. Field sampling coordinators should also determine whether any water outlet (whether or not they are used for human consumption) is leaking. Repairs should be made to the leaking outlets prior to beginning sampling. All sequential samples should be collected after removing any existing kitchen faucet filter and/or aerator and bypassing existing whole house filter(s) and/or water softener(s). The sampling location (e.g., kitchen sink faucet) must be clearly documented in the field records.

The procedures below will be followed for sequential sampling, and any deviations will be noted in the field records.

- Place the open bottles in order by sample number (S01 125-mL HDPE bottle, S02 125-mL HDPE bottle, subsequent 500-mL HDPE bottles for interior plumbing volumes, and/or subsequent 1,000-mL HDPE bottles for plumbing and/or service line volume) and remove the caps from all bottles so that all of the bottles are ready to fill.
- Record the beginning sample date/time.
- Begin by placing the Sample 1 bottle under the faucet and open the cold water slowly until the faucet is fully open. Beginning with the second round of sequential

sampling, place the Sample 1 bottle under the filter bypass and open the water slowly until the faucet is fully open. While one bottle is filling, grab the next bottle so that you are ready to move it under the faucet quickly. Collect the samples without shutting off the water in between samples, and try not to let any water spill in between samples.

- Once the bottle is filled, quickly place the Sample 2 bottle under the faucet, and continue until you have filled all sample bottles.
- Once all sample bottles have been filled, place the caps tightly on all sample bottles.
- Record the final sample date/time.
- Fill in the highlighted areas of the Chain-of-Custody form and sign/date the form when the samples are relinquished for shipping.

Following the completion of sequential sampling, field measurements for pH and chlorine residual and water sampling for laboratory analysis of total metals, total phosphorous, alkalinity, chloride, sulfate, and fluoride may be collected as discussed in Section 5.6 and listed below.

- DS-01 - One 125-mL HDPE bottle will be collected for analysis of total phosphorus, field-preserved ( $H_2SO_4$  to  $pH < 2$ ) within 15 minutes of sampling.
- DS-02 - One 125-mL, unpreserved HDPE bottle will be collected for analysis of alkalinity and anions (sulfate, chloride, fluoride).
- DS-03 - one 1,000-mL HDPE bottle will be collected and field preserved ( $HNO_3$  to  $pH < 2$ ) for analysis of total metals including lead.

Field sampling coordinators will be responsible to pack water samples and ship to the selected EPA laboratory, as described in Appendix J. Samples for total phosphorus (DS-01) and anions/alkalinity (DS-02) will be placed in an ice-filled cooler following sampling and shipped on ice.

Water sample identification will include the nomenclature below.

- A prefix of “S” will indicate sequential samples with the subsequent numbers indicating the order of sampling.
- The prefix of “DS” will indicate distribution system samples, generally collected after approximately 5 minutes of flushing, with subsequent numbers indicating total phosphorus analysis (01), alkalinity and anions (02), and total metals (03).
- House letter unique to the address and sample type will be included following numbers.
- The date (or a number indicating the sampling round) may be appended as a suffix for subsequent resampling at a given sampling location. For example, “S2” and “DS2” will be used to indicate second-round sequential samples.

Unique sample identification will be required. Deviations from this sampling nomenclature must be clearly documented in the field records including hard copy field forms, electronic field forms, and/or chains of custody.

Field records should also include the following, to the extent information is provided by the resident or observed in the field:

- Water meter reading
- Interior plumbing including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Service line including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Estimated distance (or pipe length) between the sample tap and the distribution water main, including distances to the curb, shut-off, and/or water meter
- Filter(s) in use at the home, including any that were being used at the time of sampling
- Known physical disturbances such as recent road work or utility work that could disturb the service line near the sampling site
- Other relevant field observations such as activities completed at the home (e.g., flushing, aerator cleaning) and color, odor, or debris in the water
- Photographs of the sample tap(s) and underlying fixtures and components

Before leaving the site, the field sampling coordinator should confirm the faucet filter is properly installed and returned to “Filter” mode (not “Bypass” mode). If time allows, the field sampling coordinator will work with residents to check and clean aerators on all other faucets in home.

Following the review of initial sequential sampling results, EPA may return with licensed plumbers to initial sequential sampling sites to collect additional plumbing information for further evaluation of analytical results and identification of LSL sites. Plumbing information will be recorded in the field records, using electronic field forms when possible. Appendix C-3 includes an example form for plumbing inspection.

## Appendix C-1 Sequential Sampling Instructions to Residents

### Sequential Sampling Instructions

Please read all instructions at least one day prior to scheduled sampling.

- Use only the kitchen faucet for all of these samples. **Remove any point-of-use filter from the faucet prior to flushing or sampling.**
- **If there is a filter** - The night before sampling (right before everyone goes to bed), remove any point-of-use filter from the faucet.
- The night before sampling (right before everyone goes to bed) run the water from the kitchen faucet for at least 5 minutes, so that sequential sampling provides representative information on the sources of lead for each specific volume of water.\* **Write down the date/time you finished running the water.**
- **Do not use ANY water from the home plumbing for at least 6 hours prior to samples being collected the following morning.** Specifically, do not shower, flush toilets, wash laundry, or use other water taps. Any automatic lawn sprinkler systems should also be turned off. It may help to tape a sign in the kitchen and bathrooms with a reminder not to use the water, in case people forget. If water is accidentally used, please notify EPA so that resampling can be considered.

\*NOTE: As EPA and others have reported, the practice of “pre-flushing” can result in underestimation of lead in water exposure when using a single 1,000-mL “first-draw” sample for Lead and Copper Rule compliance monitoring. Because relatively clean water from the water main is flushed through the service line and premise (interior) piping, a single 1,000-mL “first-draw” sample will only capture lead contributed by the portions of the premise (interior) piping closest to the faucet. For homes with lead service lines, significant lead exposure may be present but not captured by the single 1,000-mL “first-draw” sample, especially if “pre-flushing” is used.

By comparison, this study uses sequential water sampling, which collects volumes of water that capture lead contribution from sequential segments of the premise and service line piping (i.e., first bottle captures water that has been sitting overnight in piping nearest the faucet, and subsequent bottles capture water farther away towards the water main). By flushing the lines prior to sequential sampling, the lead contribution from individual pipe segments can be more easily distinguished.



## Appendix C-3 Plumbing Inspections for Corrosion Control Treatment Assessment

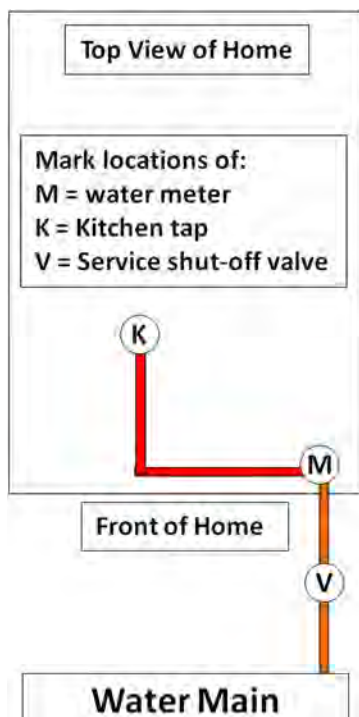
EPA has completed sequential sampling at approximately 100 homes that have previously tested high for lead in the water. The objective of the plumbing inspections is to identify homes which have lead service lines and a variety of plumbing materials commonly found in homes for use in assessing the progress of corrosion control treatment.

In order to properly assess the effectiveness of the corrosion control treatment for the variety of plumbing materials commonly found in homes, the goal is to identify 15 homes in each of three plumbing categories (galvanized iron pipe, copper pipe and plastic pipe). Based on the results of the plumbing inspections and other information, EPA will select 45 homes for conducting sequential sampling every two months to assess lead levels.

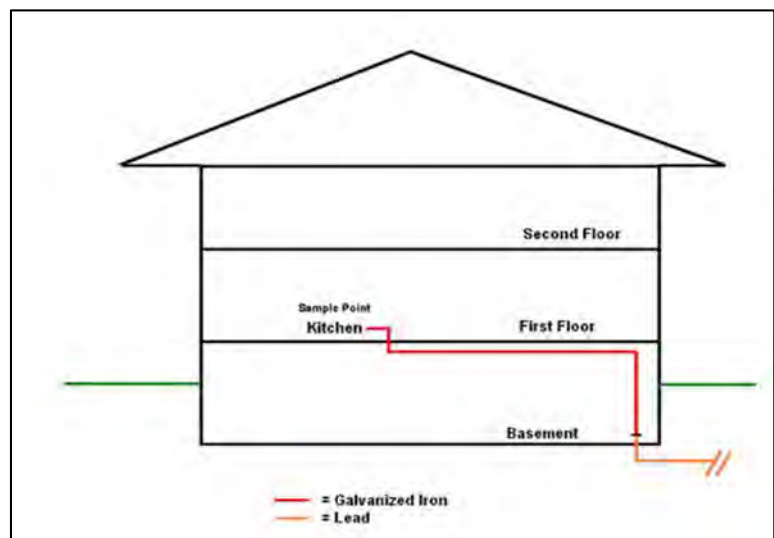
Information to be collected includes the water main location, service line information, and internal (home) plumbing information.

Beginning with the service line entering the home, note the material and pipe diameters and measure the length of pipe for each pipe segment from the service line inlet to the kitchen tap. Note any connectors, valves, and fittings at the end of each segment. Also note the location, make and model of any water treatment units connected to the plumbing from the service line inlet to the kitchen tap.

EXAMPLE TOP VIEW



EXAMPLE FRONT VIEW





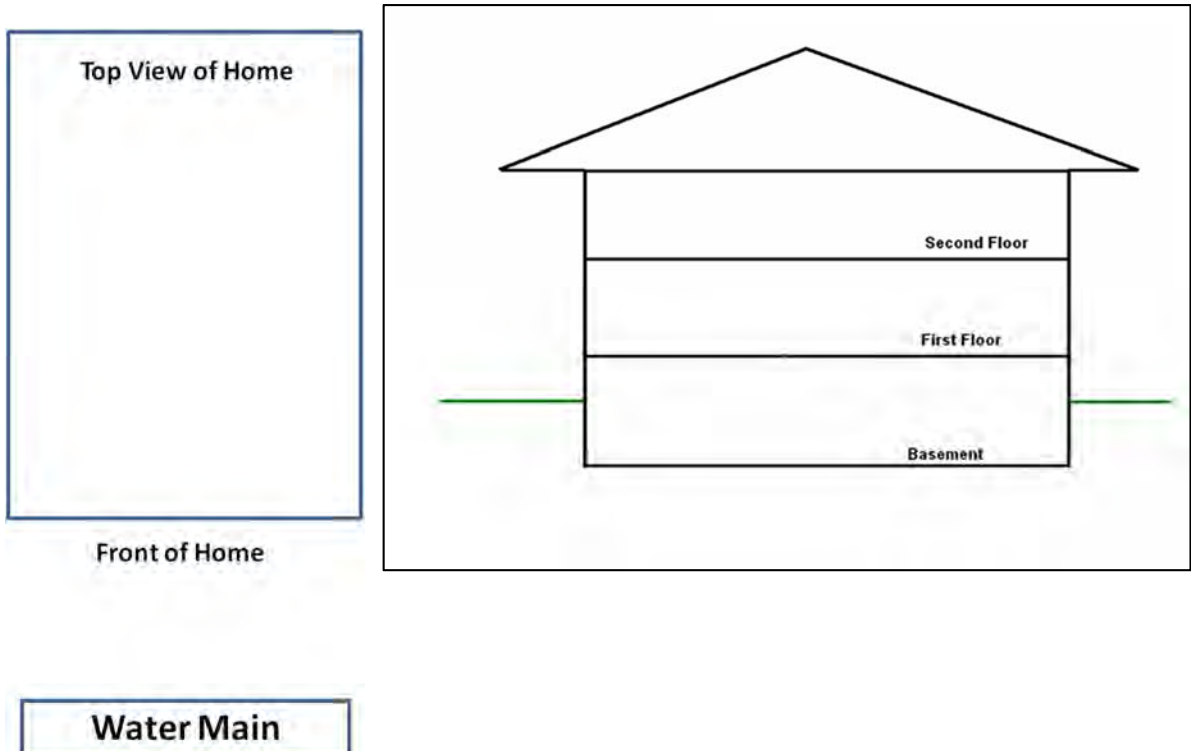
**EXAMPLE COMPLETED FORM**

<b>Resident Information</b>				
Name: Samantha Smith		Address: 1415 W. Main St Flint, MI xxxxx		Phone: (810) xxx-xxxx
Plumber(s): Sam Stone			Phone #(s): (810) xxx-xxxx	
<b>Water Main Location</b>				
Note whether the water main is located on the same (near) side as the home (N) or on the far side across the street (F)		N <u>X</u> F ___		
<b>Service Line Information</b>				
*Please use the following abbreviations for all plumbing information: L = Lead, C = Copper, G = Galvanized Iron, P = Plastic, B = Brass				
For the information below, list the source of information used, such as City of Flint or other plumbing records, visual inspection, etc.				
Service line material, pipe diameter and length of pipe from water main to service shut-off. Information Source: <u>City of Flint records</u>		*Material Unknown	Pipe Diameter (inches) Unknown	Length (inches) 224
Service line material, pipe diameter and length of pipe from service shut-off into home Information Source: <u>Visual inspection</u>		L	1 inch	630
<b>Internal (Home) Plumbing Information</b>				
*Please use the following abbreviations for all plumbing information: L = Lead, C = Copper, G = Galvanized Iron, P = Plastic, B = Brass, S = stainless steel				
Pipe Segment	*Material	Pipe Diameter (inches)	Length (inches)	Note all connectors, valves, and other plumbing components
1	L	1 inch	6 inches	Lead service line comes into home and runs 6 inches to brass shut-off valve.
2	C	1 inch	12 inches	Copper pipe from shut-off valve to water meter.
3	C	1 inch	65 inches	Copper pipe from water meter to brass shut-off valve.
4	C	½ inch	200 inches	Copper pipe to brass shut-off valve under sink.
5	SS	¼ inch	16 inches	Stainless steel tube from shut-off valve to faucet connection.

## Plumbing Inspection Form

### General Location of Sampling Tap

Using the home diagrams at the bottom of the page, draw the plumbing path from the service line inlet to the kitchen tap as shown in the two example diagrams (top view and front view) above.



Information to be collected using the form below includes the water main location, service line information and internal (home) plumbing information. Beginning with the service line entering the home, note the material and pipe diameters and measure the length of pipe for each pipe segment from the service line inlet to the kitchen tap. Note any connectors, valves, and fittings at the end of each segment. Also note the location, make and model of any water treatment units connected to the plumbing from the service line inlet to the kitchen tap.

Should you have any questions, please contact \_\_\_\_\_

## Plumbing Inspection Form, continued

<b>Resident Information</b>				
Name:	Address:	Phone:		
Plumber(s):		Phone #(s):		
<b>Water Main Location</b>				
Note whether the water main is located on the same (near) side as the home (N) or on the far side across the street (F)		N ____ F ____		
<b>Service Line Information</b>				
*Please use the following abbreviations for all plumbing information: L = Lead, C = Copper, G = Galvanized Iron, P = Plastic, B = Brass				
For the information below, list the source of information used, such as City of Flint or other plumbing records, visual inspection, etc.		*Material	Pipe Diameter (inches)	Length (inches)
Service line material, pipe diameter and length of pipe from water main to service shut-off.  Information Source: _____				
Service line material, pipe diameter and length of pipe from service shut-off into home  Information Source: _____				
<b>Internal (Home) Plumbing Information</b>				
Beginning with the service line entering the home, note the material and pipe diameters and measure the length of pipe for each pipe segment from the service line inlet to the kitchen tap. Note any connectors, valves, and fittings at the end of each segment. Also note the location, make and model of any water treatment units connected to the plumbing from the service line inlet to the kitchen tap.				
*Please use the following abbreviations for all plumbing information: L = Lead, C = Copper, G = Galvanized Iron, P = Plastic, B = Brass, S = stainless steel				
Pipe Segment	*Material	Pipe Diameter (inches)	Length (inches)	Note all connectors, valves, and other plumbing components
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				

## **Appendix C-4 Additional Distribution System Samples for Flow Rate Assessment**

In order to assist with the effort to develop a sampling protocol to identify LSLs, two additional DS samples will be collected at an appropriate subset of sequential sampling sites known to have a full LSL or suspected of having a partial (public-side) LSL to evaluate whether flow rate might be a factor to consider in the sampling framework for LSL identification.

Following the collection of sequential samples with the faucet fully open (i.e., maximum flow rate), as described in the standard sequential sampling procedures (see Appendix C above), the following additional sampling and flow rate measurement activities will be conducted:

- After the DS-03 sample has been collected, the water will be allowed to continue flowing uninterrupted and the fully-open flow rate will be measured by placing a marked pitcher under the faucet and capturing 2 liters of water and recording the amount of time it takes to capture the 2 liters of water. The flow rate will be calculated in liters per minute by dividing 2 liters by the time in seconds and multiplying the resultant value by 60 to convert seconds to minutes:  $(2L/\text{time (sec)}) \times (60 \text{ sec}/1 \text{ min})$ . The flow rate will be recorded in L/min in the field records for DS-03.
- The flow will then be reduced, targeting approximately two-thirds of the fully-open flow rate. The second flow rate will then be measured and calculated as described above. Following this second flow rate measurement, the water will be allowed to continue flowing uninterrupted at this second flow rate and sample DS-04 (1,000 mL) will be collected. The flow rate will be recorded in L/min in the field records for DS-04.
- The flow will be further reduced, targeting approximately one-third of the full flow rate. The third flow rate will be measured and calculated as described above. Following the flow rate measurement, the water will be allowed to continue flowing uninterrupted at this third flow rate and sample DS-05 (1,000 mL) will be collected. The flow rate will be recorded in L/min in the field records for DS-05.

DS-04 and DS-05 will be field-preserved and analyzed for total metals, including lead, as described in Section 8.1.

## Appendix C-5 Lead Service Line Detection Protocol

This task will develop a protocol for direct application for LSL identification in the Flint drinking water system and methods will be identified to customize the protocol for application in other drinking water systems after the gathering of distribution-specific data. As part of this project, the protocol may be used at one or more additional cities (e.g., Galesburg, IL) to assess the variability of LSL Detection, including the LSL threshold(s).

### *Phase 1 – Preliminary Sample Collection*

Phase 1 of preliminary sample collection will take place in early 2017. Battelle will perform sampling and may be accompanied or supported by other parties (e.g., EPA, City of Flint, MDEQ). In coordination with City of Flint and/or MDEQ, EPA ORD will select 10 to 15 houses that are very likely to have lead service lines (LSL) and are high priority for LSL replacement and 10 to 15 houses with that have never had LSL (i.e., copper to copper). Homes within each category will be selected to include a mix of interior plumbing materials (e.g., plastic, galvanized), as available information allows. Homes for which the existing lead service line (private and/or public) is replaced will then be resampled approximately 3 to 4 weeks after replacement. A consistent flushing protocol will be performed by contractors and residents after LSL replacement (usually 15 minutes flushing at each faucet) that is distinct from the pre-sampling flushing described in the LSL detection protocol.

Before sample collection, the following field data will be collected at each home sampled and documented in the field notebook with ultimate entry into an Excel® project database:

- The location of the water main (e.g. same side of street or across the street)
- Known physical disturbances such as recent road or utility work that could disturb the service line near the home being sampled
- Verification the resident completed the 5 minute pre-stagnation flush (with POU removed) at the faucet and followed the 6 hour stagnation time
- Type of material (e.g., polyvinyl chloride [PVC], galvanized iron, copper, and/or lead) for interior pipes and connectors (basement and under the fixtures) where accessible. A magnet will be used to check for galvanized pipe. For suspected lead pipe, the tip of a pen will be carefully pushed into the pipe surface to check the softness of the material.
- Observations of plumbing lengths and diameters, where possible, for volumes estimation
- Water meter material (e.g. plastic or brass) and the water meter readin.
- Photographs of the sample tap and underlying fixtures and components

If the 5 minute pre-stagnation flush or 6 hour stagnation time were not followed, the sampling will be rescheduled.

For each applicable service line, the material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter will be confirmed after LSL removal for those LSLs that were identified.

The same sample collection procedures and ICP/MS metals analyses will be performed for all homes. Samples will be analyzed for lead, copper, and zinc. Field measurements will include pH, free chlorine, and temperature. Additional analytes (e.g., anions, iron) may be identified based

on initial sampling results. Sampling will use bottles identified for Methods Analysis in Section 8.1.

The sampling collection procedure will include:

- Plumbing observations and estimates.
- Sequential sampling of up to eighteen samples (Two [2] 125 mL bottles at the start followed by up to sixteen [16] 1 L bottles) representing the pipe length extending from the kitchen tap to the water main in the street. Fifteen samples will be collected if the water main is on the same side of the street and 18 samples will be collected if the water main is across the street. This will facilitate generation of a lead concentration profile that will indicate LSL presence and/or verify the results from the flushed sample.
- Temperature will be measured for at least 4 of the sequential samples to allow for the possibility of temperature measurements to estimate location of basement wall/service line.
- Flush kitchen tap for 5 minutes (5 MF) at approximately 10 L/min (maximum flow) and collect 1L.
- Measure pH and free chlorine.
- Flush kitchen tap for 5 minutes (5 MF) at approximately 5.5 L/min (half maximum flow) and collect 1L
- Flush kitchen tap for 5 minutes (5 MF) at approximately 2L/min and collect 1L

Unique sample identification will be required and deviations must be clearly documented in field records, including hard copy field forms, electronic field forms, and/or chains of custody. Water sample identification will include the nomenclature below.

- For sequential samples, a prefix of “B6” will be used to indicate Battelle and the round of sampling (beginning with 6 in April 2017), subsequent numbers will reflect the order of sampling.
- For Flush samples, a prefix of “BF6” will be used with a value of 2, 5.5, or 10 will indicate flush samples with the flow rates of approximately 2 L/minute, 5.5 L/minute, and 10 L/minute, respectively
- A prefix of S will indicate sequential stagnation samples with the subsequent numbers reflecting the order of sampling.
- House letter (starting with CCa, [“CC” for copper to copper]) unique to the address and sampling type will be included following the numbers.
- The date will be appended as a suffix.

The procedure listed below will be used for sequential sample collection and temperature measurement, and deviations will be noted in the field records.

- Verify with the homeowner which valve is for cold water at the kitchen sink.
- Place paper towels on the kitchen counter.
- On the paper towel, place the pre-labeled open bottles in order by sample number (the sample number will also be written on the outside of each bottle with a Sharpie® for better visibility) and remove caps so bottles are ready to fill.
- Record the beginning sample date/time.

- Begin by placing the B6-1 bottle (125 mL) under the faucet and open the cold water slowly with as high flow as possible with no water spills. Collect sample. While one bottle is filling, grab the next bottle so that it can be moved rapidly under the faucet for the next sample. When the third sample (the 1<sup>st</sup> 1 L sample) has started collection, increase the flow until the faucet is fully open (if not already). Collect the samples without shutting off the water between samples, and try not to let any water spill between samples.
- Continue until all sample bottles have been filled.
- Record the presence of color, odor, or debris in samples.
- Keep the faucet running for flush sample collection.
- Once the sequential sample bottles have been filled, place the caps tightly.
- Measure the temperature in at least 4 sequential sample bottles. Clean the temperature probe with DI water after each measurement.

The procedure listed below will be used for flush sampling sample collection and water quality parameter measurement, and deviations will be noted in the field records.

- Place the pre-labeled open bottles in order by sample number and remove caps so bottles are ready to fill.
- Measure the fully-open flowrate of the fully open tap using a beaker. Record the time it takes for water to reach the 2 L mark on the beaker.
- Perform flushing for 5 minutes.
- Measure the pH and free chlorine.
- After flushing, record the sample time. Collect the 1 L sample (10 L/min).
- After sample collection, reduce the cold water flow by about half. Measure and record the flowrate. Perform flushing for 5 minutes. Collect the 1 L sample (5.5 L/min).
- After sample collection, reduce the cold water flow by about half again. Measure and record the flowrate. Perform flushing for 5 minutes. Record the sample time. Collect the 1 L sample (2 L/min).
- Once all sample bottles have been filled, place the caps tightly on all sample bottles.
- Record the presence of color, odor, or debris in samples.
- Provide the homeowner with a handout with Battelle contact information and the pH and free chlorine measurements.

After sequential and flush sample collection, Battelle will prepare the samples for shipment.

- Write the sample time and date on the label.
- Add the nitric acid preservative to each bottle. Use a pH strip to verify that the pH is less than 2.
- Complete the Chain-of-Custody form and sign/date the form when the samples are relinquished for shipping.

Sample handing and custody requirements will be performed in accordance with Section 10 and Appendix J. Field sampling coordinators will be responsible for packing and shipping samples to the selected EPA laboratory in accordance with Appendix J.

If samples are sent for analysis at regional or contract laboratories other than EPA CRL, analyses will be performed with the lowest MDL and reporting limits available through the program to allow for comparability of all analytical data gathered. Analytical quality control will be performed in accordance with Section 9.2. Laboratory/equipment testing, inspection, maintenance, and calibration requirements will be performed in accordance with Section 11. All laboratory data and analytical results will be handled consistently with Section 12. Field data for the LSL Detection Study will be collected, reviewed, and stored by ORD and its contractor (Battelle) to ensure data completeness and quality consistent with Sections 12 through 14.

The Phase 1 data will then be analyzed and LSL threshold(s) will be identified in the following manner:

- Analyze the collected sampling data. Determine if there is a difference between 5MF sample concentrations collected at the different flow rates. Then, compare the average concentration and spread for the 5MF of chosen flow rate samples across the homes with and without LSL. Verify 5MF sample with the 30MS sequential sampling profile.
- Determine the concentration below which samples only came from non-LSL sites. This will be the threshold concentration for LSLs. Verify 5MF sample with the 30MS sequential sampling profile.
- Determine the lowest concentration above which samples only came from LSL sites. This will be the screening concentration for LSLs; if a sample concentration is above this threshold, there should be high certainty that an LSL is present. Verify 5MF sample with the 30MS sequential sampling profile. For samples below this concentration, develop a threshold concentration for LSLs.

### *Phase 2 - System-Wide LSL Screening*

Phase 2 may be performed during the third quarter of calendar year 2017. The protocol used to gather data will be used in Phase 2 (with any identified modifications in place after implementation and review of data). During Phase 2, the resulting flushed concentrations will be compared to the thresholds identified determined during Phase I conducted earlier in 2017 to assess the presence of LSL using the protocol.



## **Appendix D – Health and Direct Contact Exposure Evaluation**

EPA will coordinate with Michigan Department of Health and Human Services (MDHHS) and Agency for Toxic Substances and Disease Registry (ATSDR) to collect grab water samples from selected locations including bathroom faucet(s), bathtub tap(s), shower head(s), and kitchen faucets at homes selected by MDHHS and ATSDR.

## Appendix D-1 – Health and Direct Contact Exposure Evaluation, General Procedures

EPA will coordinate with Michigan Department of Health and Human Services (MDHHS) and Agency for Toxic Substances and Disease Registry (ATSDR) to collect grab water samples from selected locations including bathroom faucet(s), bathtub tap(s), shower head(s), and kitchen faucets at homes selected by MDHHS and ATSDR.

Field sampling coordinators will confirm the schedule with the applicable resident(s) in advance of sampling activities. Several samples will be collected at each location at the discretion of the field sampling coordinators, generally following the nomenclature and bottle types listed below. No stagnation or flushing is required prior to grab sampling. Field sampling coordinators will also record additional field observations including the presence and descriptions of any color, odor, and sediment or debris in the water from each tap sampled. Grab samples should generally be collected after removing any aerator or faucet filter and bypassing existing whole house filter(s) and/or water softeners. If a faucet filter is currently in use at the sampling site, field sampling coordinators may collect additional filtered water sample(s) and document the sample collection methods in the field records. The sampling location (e.g., kitchen sink faucet) must be clearly documented in the field records. A house letter unique to the address and sample type will be included following numbers.

- Kitchen sink faucet (cold water samples)
  - R01 - one 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals (long list – see Section 9.1) and hardness
  - R02 - one 125-mL, unpreserved HDPE bottle will be collected for analysis of alkalinity and anions (sulfate, chloride, fluoride)
  - R03 - one 125-mL, unpreserved HDPE bottle will be collected for analysis of TDS
- Bathroom sink faucet (cold water sample)
  - R04 - 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals (long list – see Section 9.1) and hardness
- Bathtub faucet (cold water sample)
  - R05 - 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals (long list – see Section 9.1) and hardness
- Shower faucet (cold water sample)
  - R06 - 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals (long list – see Section 9.1) and hardness
- Bathtub or Shower faucet, whichever is used more (hot water samples)
  - R07 - one 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals (long list – see Section 9.1) and hardness
  - R08 - one 125-mL, unpreserved HDPE bottle will be collected for analysis of alkalinity and anions (sulfate, chloride, fluoride)
  - R09 - one 125-mL, unpreserved HDPE bottle will be collected for analysis of TDS

Unique sample identification will be required, and sample times should be recorded for each sample bottle. Deviations from this sampling nomenclature must be clearly documented in the field records including hard copy field forms, electronic field forms, and/or chains of custody.

Field measurements for pH, chlorine residual, turbidity, and conductivity will be collected from cold water at the kitchen faucet after grab sampling and flushing for an additional 3 more minutes. If no kitchen faucet is currently in use at the sampling site, field sampling coordinators may collect additional field measurements and document the sample collection and location in the field records. Field sampling coordinators will measure field water quality parameters including chlorine residual, pH, conductivity, immediately after sample collection, within 15 minutes. Field sampling coordinators will also measure turbidity within 48 hours. If a turbidimeter is not available for field measurement, an additional unpreserved 125-mL sample may be collected and shipped for laboratory analysis within the 48 hour hold time. Field sampling coordinators will be responsible for obtaining and maintaining field water quality equipment (e.g., pH meter, conductivity meter, turbidimeter, and colorimetric test kits for chlorine residual) and following manufacturer guidance to ensure accurate field measurements are obtained. Field sampling coordinators will record all equipment maintenance (including calibration) and field measurements for each sample in the field records.

At the direction of MDHHS and ATSDR, turbidity, conductivity, and total dissolved solids were discontinued at the end of March 2016.

Field records should also include the following, to the extent information is provided by the resident or observed in the field:

- Interior plumbing including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Service line including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Estimated distance (or pipe length) between the sample tap and the distribution water main
- Filter(s) in use at the home, including any that were being used at the time of sampling
- Known physical disturbances such as recent road work or utility work that could disturb the service line near the sampling site
- Other relevant field observations such as activities completed at the home (e.g., flushing, aerator cleaning) and color, odor, or debris in the water
- Photographs of the sample tap(s) and underlying fixtures and components

Water samples for the assessment of health and direct contact exposure evaluation, including for analysis of TDS (and turbidity, if applicable), will be placed in ice-packed coolers as soon as possible after collection. Field sampling coordinators will be responsible to pack samples and ship to the applicable EPA or contract laboratory, as described in Appendix J. Due to short hold times, any turbidity samples must be shipped overnight on the same date as collection.

Before leaving the site, the field sampling coordinator should confirm the faucet filter is properly installed and returned to “Filter” mode (not “Bypass” mode). If time allows, the field sampling coordinator will work with residents to check and clean aerators on all other faucets in home.

## **Request Letter from MDHHS**



RICK SNYDER  
GOVERNOR

STATE OF MICHIGAN  
DEPARTMENT OF HEALTH AND HUMAN SERVICES  
LANSING

NICK LYON  
DIRECTOR

January 28, 2016

Brian Kelly, On-Scene Coordinator  
Jeffery Lippert, On-Scene Coordinator  
U.S. Environmental Protection Agency  
Emergency Response Branch  
9311 Groh Road, Grosse Ile, MI 48138

**RE: Pilot public health response for rash complaints received from city of Flint water users (i.e., Pilot Rash Response)**

Dear Mr. Kelly and Mr. Lippert,

This letter is a request for technical assistance in collecting water samples from homes in the Flint area that are on city of Flint water users where residents are reporting rashes. The details of this request are stated below. The US EPA technical assistance will be in collaboration with MDHHS pilot public health response to residents with reported rashes. Information from this effort will be used in collaboration with the US EPA, CDC, ATSDR and MDHHS as they continue to develop a more comprehensive epidemiologic-based public health response.

As discussed earlier today, here is the intended process:

**Objectives**

1. MDHHS will interview resident to gather additional information on their complaint.
2. US EPA will conduct water sampling to get initial characterization of water as described below.

**Scheduling Visits**

MDHHS-DEH will schedule visits with residents. Currently, we have three appointments scheduled for Friday, January 29, 2016. The exact times of the appointments are still to be coordinated with the US EPA sampling team. MDHHS-DEH staff will be present with the EPA during sampling and will interview the resident.

**Sampling Locations**

MDHHS will rely on EPA OSC expertise to provide a uniform grab sample collection process. Grab samples will not necessary represent worst case metals concentrations, as the water will not necessary represent 6-hours of stagnant water prior to collection. Location of grab samples from indoor water sources may include, but are not limited to, shower heads, bath tub faucets, bathroom faucets, and kitchen faucets. Consultation with the resident will assist in identifying faucets to sample. Each water sample must be given a unique identification code and information recorded on the bottle needs to include sample location within the home, type of water tap, the

sample volume, with or without filter, order of sample collection, hot or cold water, date, time, and address.

### Field - Direct Read Measurements

MDHHS request the following, to the best of EPA's immediately available resources:

- pH
- free chlorine
- color
- turbidity (if available)
- conductivity/TDS (if available)

Record any odor characteristics and visual observations (e.g. sediment), if relevant.

If possible, collect sample for (either direct read or for lab analysis)

- Hardness

### Laboratory Metal Analysis

- Please confirm with US EPA laboratory, that the appropriate method is US EPA Method 200.8 for inorganics
  - *Method for Determination of Metals in Environmental Samples, Supplement I*, U.S. Environmental Protection Agency, Method 200.8, 1999 Revision 5.5.
  - MDHHS is most interested the metals: Cr, Cu, Fe, Pb, Ni, Zn
  - MDHHS would like the full list metals analyzed, if possible:

Analyte	Chemical Abstract Services Registry Number
Aluminum	7429-90-5
Antimony	7440-36-0
Arsenic	7440-38-2
Barium	7440-39-3
Beryllium	7440-41-7
Boron	7440-42-8
Cadmium	7440-43-9
Chromium	7740-47-3
Copper	7440-50-8
Iron	7439-89-6
Lead	7439-92-1
Manganese	7439-96-5
Mercury	7439-97-6
Molybdenum	7439-98-7
Nickel	7440-02-0
Selenium	7782-49-2
Silver	7440-22-4
Tin	7697-37-2
Thallium	7440-28-0
Uranium	7440-61-1
Vanadium	7440-62-2
Zinc	7440-66-6

## **Sampling Processing and Handling**

MDHHS requests US EPA handle sample collection, processing, and shipment of samples to US EPA Region V Laboratory for analysis.

## **Data Management, Evaluation, and Reporting**

MDHHS will work with EPA, CDC and ATSDR to determine data management, evaluation, and reporting. MDHHS will report results and interpretation back to the resident.

Thank you for your consideration of this requests. For questions, please contact either Kory Groetsch (517-335-9935) or Jennifer Gray (517-373-7672).

Sincerely,



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## Appendix D-2 – Limited “Pilot” Evaluation

EPA will coordinate with MDHHS and ATSDR to collect grab water samples from homes selected by MDHHS and ATSDR to compare water quality at homes with reported health concerns in comparison to homes without health concerns. These water samples will be evaluated for an extended list of organic compounds including volatile organic compounds (VOC), semi-volatile organic compounds (SVOC), and disinfection byproducts (DBP). Sampling procedures were approved by MDHHS and ATSDR, and analytical methods, analyte lists, and detection limits were selected by MDHHS and ATSDR.

Field sampling coordinators will confirm the schedule with the applicable resident(s) in advance of sampling activities. At least 6 hours prior to sampling (i.e., the night before), the resident will assure that water is not used overnight. The resident will be asked to record the time at which water was last used in the home (i.e., no faucets, toilets, shower, bath tub, washing machine, dishwasher, and hose), and field sampling coordinators will record the total stagnation time in the field records.

At each selected sampling site, water samples will be collected from a bathtub faucet at two temperatures: cold water and hot water (maximum temperature after running the hot water for approximately one minute). For each water temperature, the following water samples will be collected and analyzed by a contract laboratory:

- Total Metals (one 1,000-mL wide mouth bottle, field-preserved with nitric acid) for analysis using EPA Method 200.7/200.8 (long list – see Section 9.1)
- VOCs including THMs (three 40-mL vials, Teflon-capped, ascorbic acid pre-dosed and field-preserved with hydrochloric acid, zero headspace, and ice), for analysis using EPA Method 524.2
- DBPs General List (three 60-ml vials, Teflon-capped, ammonium chloride and phosphate buffer pre-dosed and field-preserved with zero headspace and ice), for analysis using EPA Method 551.1
- Chloral Hydrate (two 60-mL vials, Teflon-capped, sodium sulfite and phosphate buffer pre-dosed and field-preserved with zero headspace and ice), for analysis using EPA Method 551.1
- HAAs (one 250-mL amber glass bottle, ammonium chloride pre-dosed and field-preserved with ice), for analysis using EPA Method 552.3
- SVOCs (two 1,000-mL amber glass bottles, sodium sulfite crystals pre-dosed and field-preserved with ice), for analysis using EPA Method 525.2

In addition, field measurement of temperature will be collected and recorded (for both cold water and hot water) following filling all bottles.

Samples for VOC analysis will be filled slowly to reduce loss of volatiles and to prevent overfilling. Field sampling coordinators will wear gloves during sampling to protect from acid preservative, and any droplets that fall while closing the zero-headspace bottles will be rinsed down the drain as a safety measure.

Unique sample identification will be required, and sample times should be recorded for each sample bottle. Deviations from the sampling nomenclature indicated below must be clearly documented in the field records including hard copy field forms, electronic field forms, and/or chains of custody:

- Suffix of “RP” will indicate the sampling site was selected by MDHHS and ATSDR, based on health concern information provided by the resident.
- Suffix of “RPC” will indicate the sampling site was selected as a control site by MDHHS and ATSDR; available information indicates there are no health concerns at these sites.
- House letter unique to the address and sample type will be included following numbers.
- Numbers will be used to indicate unique samples:
  - RP01 (or RPC01) – Cold water (first flush) sample for total metals (long list – see Section 9.1)
  - RP02 (or RPC02) – Cold water (first flush) sample for VOCs including TTHMs
  - RP03 (or RPC03) – Cold water (first flush) sample for DBPs general list
  - RP04 (or RPC04) – Cold water (first flush) sample for Chloral hydrate
  - RP05 (or RPC05) - Cold water (first flush) sample for HAAs
  - RP06 (or RPC06) - Cold water (first flush) sample for SVOCs
  - RP07 (or RPC07) – Hot water sample for total metals (long list – see Section 9.1)
  - RP08 (or RPC08) – Hot water sample for VOCs including TTHMs
  - RP09 (or RPC09) – Hot water sample for DBPs general list
  - RP10 (or RPC10) – Hot water sample for Chloral hydrate
  - RP11 (or RPC11) - Hot water sample for HAAs
  - RP12 (or RPC12) - Hot water sample for SVOCs
- Trip blanks for VOC and TTHM analysis will be included in each shipment and identified on the chains of custody (e.g. “Blank”).

Field records should also include the following, to the extent information is provided by the resident or observed in the field:

- Interior plumbing including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Service line including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Estimated distance (or pipe length) between the sample tap and the distribution water main
- Filter(s) in use at the home, including any that were being used at the time of sampling
- Known physical disturbances such as recent road work or utility work that could disturb the service line near the sampling site
- Other relevant field observations such as activities completed at the home (e.g., flushing, aerator cleaning) and color, odor, or debris in the water
- Photographs of the sample tap(s) and underlying fixtures and components

Water samples, including for analysis of VOCs, SVOCs, and DBPs, will be placed in ice-packed coolers as soon as possible after collection. Field sampling coordinators will be responsible to pack samples and ship to the applicable contract laboratory, as described in Appendix J.

Following the completion of grab sampling, field measurements for pH and chlorine residual may be collected as discussed in Section 5.6.

If time allows, the field sampling coordinators will work with residents to check and clean aerators on all other faucets in home.

## **Appendix E – Aerator Particulate Evaluation**

During scheduled sampling events at selected locations with previously reported high lead results, field sampling coordinators may collect scale and debris that has accumulated on aerators and water filter screens.

Solids samples will be collected at the discretion of field sampling coordinators and only if sufficient solid material is present behind the aerator and/or water filter screen at a given sampling location. Photographs will be taken onsite of the scale/debris on the aerators and screens, and field observations will be recorded.

Because aerator particulate sampling will generally be conducted while at a sampling site for other sampling activities under this QAPP, the sample nomenclature and field notes will be as discussed in those respective sampling instructions (Appendices A through H). Aerator particulate samples will include a sample identification suffix of “AP”. Unique sample identification will be required.

For each solid sample, one unpreserved glass or HDPE bottle will be used to collect as much particulate as possible for analysis of total metals including lead. Field sampling coordinators will be responsible to pack solid samples and ship to the PHILIS Contract Laboratory, as described in Appendix J.

## Appendix F - Distribution System Water Quality Assessment for Treatment Assessment

Distribution system water quality assessment sampling may be conducted at sampling sites after the completion of other sampling activities including sampling faucet filter evaluation (Section 5.1), health and direct contact exposure evaluation (Section 5.4), and resident-requested sampling (Section 5.8) and following sequential sampling for lead source/release diagnostic evaluation (Section 5.3), as specified below.

- Field water quality measurements for pH and chlorine residual will be collected and recorded following other grab sampling and sequential sampling activities.
- Water samples for laboratory analysis of total phosphorous, alkalinity, chloride, sulfate, fluoride, and total metals will be collected following sequential sampling, using the nomenclature in Appendix C (copied below). Unique sample identification will be required.
  - DS-01 - One 125-mL HDPE bottle will be collected for analysis of total phosphorus, field-preserved ( $\text{H}_2\text{SO}_4$  to  $\text{pH}<2$ ) within 15 minutes of sampling.
  - DS-02 - One 125-mL, unpreserved HDPE bottle will be collected for analysis of alkalinity and anions (sulfate, chloride, fluoride).
  - DS-03 - one 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals including lead.

Distribution system water quality sampling will be conducted following at least 5 minutes of flushing, depending on the size and length of plumbing between the sampling location and the water distribution main. All grab samples should be collected bypassing existing whole house filter(s) and/or water softeners. The sampling location (e.g., kitchen sink faucet) must be clearly documented in the field records.

Field sampling coordinators will measure chlorine residual and pH immediately after sample collection, within 15 minutes. Field sampling coordinators will be responsible for obtaining and maintaining field water quality equipment (i.e., pH meter and colorimetric test kits for chlorine residual) and following manufacturer guidance to ensure accurate field measurements are obtained. Field sampling coordinators will record all equipment maintenance (including calibration) and field measurements for each sample in the field records.

Field sampling coordinators will preserve all total phosphorus samples with one or two drops concentrated sulfuric acid to a pH below 2 SU immediately after sample collection (i.e., within approximately 15 minutes). Sulfuric acid, nitric acid, and narrow-range pH paper will be provided by EPA CRL.

Water samples for water quality parameters including total phosphorous, alkalinity, chloride, sulfate, and fluoride will be placed on in ice-packed coolers as soon as possible after collection. Field sampling coordinators will be responsible to pack distribution system water quality characterization samples and ship to the EPA CRL, as described in Appendix J.

Before leaving the site, the field sampling coordinator should confirm the faucet filter is properly installed and returned to “Filter” mode (not “Bypass” mode).

## Appendix G – Distribution System Sampling for Coliform Bacteria

Distribution system sampling for Total Coliforms and *E. Coli* will be conducted at single-family residences with low chlorine residual measured during other sampling activities (see Section 5.2 and Section 5.6). Between February and August 2016, approximately 0.05 mg/L was used as a “low chlorine residual” threshold for collecting water samples for analysis of Total Coliforms and *E. Coli*. Beginning in August 2016, 0.2 mg/L has been used as a “low chlorine residual” threshold for collecting water samples for analysis of Total Coliforms and *E. Coli*. Water samples will be preserved and transported as soon as possible (due to short hold time) after collection for analysis by a partner laboratory such as Genesee County.

Field sampling coordinators will confirm the schedule with the applicable resident(s) in advance of sampling activities. At each selected sampling site with previously measured low chlorine residual, field sampling coordinators will collect water samples from the kitchen sink cold water tap after flushing water for approximately 5 minutes. After flushing water from the sample tap for approximately 5 minutes, Coliform bacteria sample containers will be collected to assess water quality in the distribution main at each sampling site. No stagnation period is needed prior to flushing or water quality characterization sampling.

Samples will be collected in sterile 125-mL plastic bottles, placed in an ice-filled cooler, and transported directly (on the same day as sampling occurs) to the laboratory for analysis of Coliform bacteria. Samples will be preserved in the field or at the laboratory using sodium thiosulfate. Samples will be cooled to <10 °C (<50 °F) but not allowed to freeze.

### Sampling Instructions

Wear gloves when collecting samples. Do not rinse the bottles. The bottles are sterile, so care must be taken not to contaminate the bottle or cap. Once the distribution line is flushed and the flow reduced, quickly open the bottle (but do not set the cap down), hold the cap by its outside edges only, and fill the sample bottle to just above the 100-mL line, leaving approximately one inch headspace. Cap the bottle immediately and place it into a cooler with ice for delivery or overnight shipment to the laboratory.

### Holding Times

Maximum holding time for samples is 30 hours. Deliver samples to the lab the day of collection if possible or ship via overnight delivery.

Trip blanks may analyzed per the standard operating procedure of the laboratory.

When choosing sampling locations, field sampling coordinators will avoid these sampling sites for Total Coliform, if possible:

- Outdoor faucets
- Faucets connected to cisterns, softeners, pumps, pressure tanks or hot water heaters
- New plumbing & fixtures or those repaired recently
- Faucets that hot & cold water come through

- Threaded taps
- Swing spouts
- Faucets positioned close to sink or ground
- Leaky faucets

Additional tips on collecting samples for Coliform bacteria:

- remove any attachments on the faucet
- allow water to flow for 5 or 6 minutes before sampling
- do not rinse or overfill container
- always collect cold water; never sample hot water
- do not touch the inside of the sample bottle or its cap



## Appendix H – Resident-Requested Sampling and Hot Water Evaluation

Water samples may be collected at selected sampling sites at the request of residents. At least 6 hours prior to sampling (i.e., the night before), the resident will record the time at which water was last used in the home (i.e., no faucets, toilets, shower, bath tub, washing machine, dishwasher, and hose), and field sampling coordinators will record the total stagnation time in the field records.

In general, two grab water samples will be collected at the kitchen faucet at each selected resident-requested sampling site, using the sample identification nomenclature listed below. A house letter unique to the address and sample type will be included following numbers.

- G01 (unfiltered cold water sample) – After at least 6 hours of stagnation, one 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals, including lead, in first-draw unfiltered cold water.
- G02H (unfiltered hot water sample) – After the cold water sample is collected, the hot water tap will run until the water is hot. Then, one 1,000-mL HDPE bottle will be collected and field preserved ( $\text{HNO}_3$  to  $\text{pH}<2$ ) for analysis of total metals, including lead, in unfiltered hot water.

Field sampling coordinators will confirm the schedule with the applicable resident(s) in advance of sampling activities. Any faucet filter or aerator will be removed prior to sampling, and all samples should be collected bypassing existing whole house filter(s) and/or water softeners. The sampling location (e.g., kitchen sink faucet) must be clearly documented in the field records. Unique sample identification will be required. Deviations from this sampling nomenclature must be clearly documented in the field records including hard copy field forms, electronic field forms, and/or chains of custody.

While on-site, the field sampling coordinators will collect information regarding the hot water tank and the resident's willingness to participate in hot water evaluation sampling from their water tank.

- Determine if homeowner would allow a return visit and assist in sampling tank;
- Take picture of hot water tank including information on the water tank tag and condition/configuration/location of drain; and
- Ask owner for estimated age of hot water tank, identify any periodic maintenance, and record information in field record.

At selected locations, field sampling coordinators may return to collect hot water samples from the hot water tank drain, as discussed below. Hot water tank sampling sites will be selected based on the following criteria: (1) homeowner would like a sample of the hot water tank water, (2) if homeowner is willing to open valve, and (3) the tank appears to be in good condition.

- G03HWT (unfiltered hot water sample from hot water tank drain) – Homeowners will voluntarily open the hot water tank valve and fill one HDPE bottle (125-mL or 1,000-mL, depending on clearance between the valve and the floor) will be collected. Field sampling

coordinators will field preserve ( $\text{HNO}_3$  to  $\text{pH}<2$ ) the hot water tank sample for analysis of total metals, including lead.

Field sampling coordinators will be responsible to pack water samples and ship to the selected EPA laboratory, as described in Appendix J.

Field records should also include the following, to the extent information is provided by the resident or observed in the field:

- Interior plumbing including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Service line including material (e.g., PVC, galvanized iron, copper, and/or lead), length, and diameter
- Estimated distance (or pipe length) between the sample tap and the distribution water main
- Filter(s) in use at the home, including any that were being used at the time of sampling
- Known physical disturbances such as recent road work or utility work that could disturb the service line near the sampling site
- Other relevant field observations such as activities completed at the home (e.g., flushing, aerator cleaning) and color, odor, or debris in the water
- Photographs of the sample tap(s) and underlying fixtures and components

Following the completion of sampling, field measurements for pH and chlorine residual may be collected as discussed in Section 5.6.

Before leaving the site, the field sampling coordinator should confirm the faucet filter is properly installed and returned to “Filter” mode (not “Bypass” mode). If time allows, the field sampling coordinator will work with residents to check and clean aerators on all other faucets in home.

## **Appendix I – Lead Service Line Extraction and Pipe Scale Analyses**

In order to inform corrosion control treatment optimization decisions, segments from four LSLs will be extracted and used for assessing the current condition and composition of the scales within the LSLs. Pipe scale analyses on segments of these pipes will be used to determine the type of scales have formed inside the lead lines under different chlorine levels and water quality conditions.

Four full LSLs will be identified by EPA ORD and the Technical Support Team for initial extraction, including two sites in low chlorine areas and two sites in high chlorine areas. Based on ORD's assessment of the 5 full LSL addresses provided by EPA and the 37 full LSL addresses from MDEQ, evaluations are being conducted on LSL locations in comparison to the following chlorine data sources:

- 4 years of Flint water Utility Total Coliform Rule chlorine data from 10 sites
- Chlorine data from the 24 EPA chlorine monitoring locations (see Section 5.4)
- All chlorine screening data collected by the lead sampling teams (see Section 5.6)

Two 4-foot segments from each LSL will be carefully extracted, packed and shipped to EPA ORD for scale analyses. If possible, the extracted segments should be driven back to EPA ORD to avoid physical disturbances during shipping. Depending on the suitability and condition of the extracted LSLs, additional LSLs may need to be extracted using the procedures outlined in this appendix. The sections below discuss EPA and City of Flint activities associated with LSL extraction and pipe scale analysis.

### **EPA Activities**

Prior to extraction of the LSL, sequential sampling and WQP monitoring should be conducted at each of the four identified LSL sites in accordance with Appendix C.

Following the extraction of each LSL and the subsequent installation of new service lines at each site, EPA will conduct thorough whole-home flushing at all taps for at least 5 minutes, beginning with the tap closest to the point at which the service line comes into the home. Aerators should be removed and cleaned while the taps are being flushed.

Residents should also be provided with a water filter and replacement cartridge.

Following the replacement of the LSL and whole-home flushing, a final round of sequential sampling will be conducted in accordance with Appendix C.

Once post-replacement sample results are received, the residents will be provided with test results and explanation of the results.

## City of Flint Activities

Two 4-foot segments would be extracted from each site identified in the above summary, then packed and delivered to EPA ORD (Cincinnati) for pipe scale analyses as specified below.

### *Documentation/Sample Tracking*

Each pipe segment should be clearly labeled with the address of the site, the removal date, the installation date or year (if known) and a unique sample identification number (e.g., 314-1, 314-2). The addresses and other personally identifiable information will not be shared outside EPA except under an appropriate data sharing agreement.

### *Pipe Extraction Procedures*

The location of each extracted pipe segment should be recorded in terms of distance from the water main.

The orientation of each pipe segment must be maintained during extraction so that the direction of water flow (arrow pointing from the water main into the home) can be clearly marked on each pipe segment.

It is important to maintain the physical integrity of the scales within the LSLs to be extracted. Pipes should not be extracted using cable tools. Proper equipment and procedures should be utilized in the excavation and extraction of the LSLs to avoid damage to the scales within the LSLs. Where possible, surface material (asphalt, cement, etc.) should be cut rather than jackhammered to avoid vibration and jarring of LSLs which can dislodge the scale within the LSLs. Excavation of subsurface down to the LSLs should be done carefully to avoid the same.

For each extracted pipe, two 4-foot long sections should be carefully cut using a rotary cutting tool. Electric saws or other tools that would vibrate the pipe should not be used.

- Pipes should not be bent during extraction, or in preparation for transport or to fit any shipping container.
- Pipes should be physically cut, not crimped on the ends.
- Short moist sponges should be inserted in the ends of the pipes before capping ends of each pipe segment to retain moisture.
- Each end of the pipe segments should be sealed using plastic inserts or plastic caps and taped securely in place on each end.
- Pipe segments should be packed such that the pipe sections do not shift or move inside the container during shipment (e.g., stiff thick-walled cardboard mailing tubes with bubble-pack)
- Where the LSL is connected to different components or connections, the LSL on either end of the component(s)/connection(s) or other pipe junctions of dissimilar metals should be preserved intact for shipment to EPA ORD with the pipe segments for scale analyses. These connection points are analyzed to determine if the water chemistry produces evidence of actual galvanic corrosion. Similar to the LSL segments, these pipes should be physically cut, not crimped on the ends. Short moist sponges should be inserted in the ends of the pipes

before capping ends of each pipe segment to retain moisture and each end of the pipe segments should be sealed using plastic inserts or plastic caps and taped securely in place on each end.

The extracted LSL segments will be sent to Kirk Sheckel at EPA ORD (Cincinnati) for sample analysis by various elemental and spectroscopic techniques in accordance with the Quality Management Plan for Research at the Advanced Photon Source of the Argonne National Laboratory, included below.

**Pipe Scale Analysis and Evaluation:  
Quality Management Plan for Research at the Advanced Photon Source of the  
Argonne National Laboratory**

# **Quality Management Plan for Research at the Advanced Photon Source of the Argonne National Laboratory**

# NRMRL

## Quality Management Plan (QMP)

<b>Title:</b>	Quality Management plan for Research at the Advanced Photon Source of the Argonne National Laboratory		
<b>Division/Staff:</b>	LRPCD		
<b>QLOG Number:</b>	L-18735-QM-1-0		
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## Acronyms

ANL:	Argonne National Laboratory
APS:	Advanced Photon Source
MRCAT:	Materials Research and Collaborative Access Team
GUP:	General User Proposal
IAG:	Interagency Agreement
EXAFS:	Extended X-ray Absorption Fine Structure
HASP:	Health and Safety Plan
QAPP:	Quality Assurance Project Plan
QAM:	Quality Assurance Manager
NRMRL:	National Risk Management Research Laboratory
LRPCD:	Land Remediation and Pollution Control Division
WSWRD:	Water Supply and Water resource Division
GWERD:	Ground Water and Ecosystem Resources Division
RAP:	Research Action Plan

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## Executive Summary

The Quality Management System implemented at the Materials Research Collaborative Access Team (MRCAT) and other Advanced Photon Source (APS) general user research stations is a description of the procedures and practices implemented for EPA research activities at the Advanced Photon Source (APS) of the Argonne National Laboratory. The EPA National Risk Management Research Laboratory (NRMRL) provides funding to the MRCAT collaborative team to gain access to the APS facility for environmental research on the fate and transformation of metal and metalloids in the environment. Access to MRCAT is accomplished through an Interagency Agreement (Primarily funded by LRPCD and GWERD with occasional assistance from WSWRD), which affords NRMRL and collaborative EPA entities access when working with NRMRL researchers such as other ORD National Labs, EPA Program Offices, and EPA Regional researchers. EPA research also gains access time to the Advanced Photon Source through general user proposals (GUPs) to conduct independent and external collaborative research studies at a variety of other beamlines with different capabilities.

This Quality Management Plan (QMP) establishes quality assurance practices and procedures that apply to the research conducted by EPA at the APS through the MRCAT vehicle and GUPs. The NRMRL APS QMP defines the structure of the Quality Management System implemented by EPA at the APS, and assigns important roles and responsibilities. This QMP is written to be consistent with the policies and procedures which are documented in the NRMRL QMP which is an addendum to the EPA Office of Research and Development (ORD) QMP.

It is the EPA policy that all intramural and extramural research projects comply with Agency requirements with specific adaptations as determined by NRMRL and collaborative EPA entities, to be consistent with the Agency graded approach to Quality Assurance. More specifically, it is the EPA policy that all research projects be planned, managed, and assessed in a manner that ensures results are of the quality needed for the known or anticipated data use, and that each study be thoroughly and correctly documented to ensure that those data and conclusions drawn from them are defensible. The primary responsibility for planning and implementing research studies to produce defensible data is assigned to the Project Leader, Project Officer, or Principal Investigator.

## INTRODUCTION

The Materials Research and Collaborative Access Team (MRCAT) is an external collaboration of multiple organizations formed to utilize the equipment at the Argonne National Laboratory Advanced Photon Source facility. MRCAT is one of 32 active research sectors at the APS. The Advanced Photon Source is a synchrotron light source that produces high-energy, high-brilliance x-ray beams. The source is optimized to put large quantities of high-energy photons into a very small area in a very short time

The facility operates for 3 three-month runs (or cycles) each year, with a one-month shutdown between runs for maintenance. The runs typically span February to April, June to August, and October to December.

In general, the APS makes beam time available to the international scientific community in two ways: General User access and Partner User (collaborative access teams such as MRCAT) access. Specific requirements govern both modes of access.

General Users are those who require less than 10% of the beam time on a beamline in a given cycle. Proposals from General Users are considered through a web-based process three times a year and reviewed by a panel of experts with beam time awarded based on merit.

Partner Users are those whose work involves a greater scope and greater commitment by both the user and the APS.

One kind of Partner User is a Collaborative Access Team (CAT). A user whose home institution is a member of a CAT may be eligible to request beam time through the CAT. Options for time-sensitive access are available. As member of the MRCAT, EPA has access to the two beamlines located at the MRCAT sector and to other beamlines as a general user.

For research that will be published in the open literature, there is no charge for beam time. Users are expected to acknowledge and notify the APS of publications resulting from work conducted here. For proprietary work, an hourly fee is charged to recover facility costs.

Because Argonne National Laboratory is a controlled-access site operated by the U.S. Department of Energy (DOE), APS users must follow DOE requirements regarding permission to enter the site (especially for users who are not U.S. citizens), training, safety practices for experiments, shipping of samples and equipment, etc. However, APS and beamline staff work very closely with users to help them fulfill these requirements.

The need to decrease uncertainties in risk assessment and develop viable remediation techniques for metal-contaminated soils/sediments has led to the realization that solutions require knowledge of material properties and processes at the molecular level. This realization has resulted in an explosion of research activity in a field that has become known as molecular environmental science (MES). The aim of MES is to identify the metal species that are of concern in terms of toxicity, mobility, and bioavailability. The research involves determinations of chemical speciation, reactions at surfaces that modify speciation, the inherent mobility and bioavailability of contaminants, and physical processes that control transport.

Synchrotron radiation plays a crucial role in solving environmental science problems. The advent of high-brilliance synchrotron sources has led to x-ray-based analytical techniques to accomplish what is impractical by other means. Foremost among these techniques are (1) x-ray absorption fine structure (XAFS), which provides atomic-level information on oxidation state, coordination, bond lengths, and the identity of nearest neighbors, and (2) surface scattering, which provides information on the arrangement of atoms at and near interfaces between gases, liquids, and solids. Microtomography is also an increasingly used analytical method; it enables nondestructive imaging of the internal structure and composition of fragile objects (e.g., soil aggregates), as well as pseudo-real-time studies of fluid flow through porous media.

Synchrotron radiation techniques are important on a national level in the area of MES. The national effort has been formalized with the formation of EnviroSync, a national organization representing the growing community of MES synchrotron radiation users in the United States. EnviroSync is modeled

after two existing organizations, GeoSync and BioSync, which represent U.S. synchrotron radiation users in the geologic and biological sciences, respectively.

A survey by the EnviroSync committee (<http://envirosync.org>) indicated that 45 experiment stations at the 4 synchrotron facilities operated by DOE (APS, SSRL, National Synchrotron Light Source at Brookhaven, and Advanced Light Source at Lawrence Berkeley) are being used for MES research. However, only 2 of the 45 experiment stations are dedicated to MES research. The MES community is continuing to expand the demand for beam time for MES experiments.

Increasing number of new users are entering the field for the first time with exciting new research, and a substantial number of experienced researchers have developed established research programs that need regular and extended access to synchrotron-based analytical facilities. Yet the experiment stations currently used for MES are either fully subscribed or oversubscribed in virtually all cases. To address the shortage of experimental MES facilities, a Collaborative Access Team (CAT) at the APS (EnviroCAT) has been established. EnviroCAT works to provide the dedicated state-of-the-art facilities that are critically needed for research on a broad range of environmental science problems. EnviroCAT focuses on developing multifaceted microanalysis facilities with insertion device and bending magnet beamlines. EnviroCAT works to provide the dedicated state-of-the-art facilities that are critically needed for research on a broad range of environmental science problems. EnviroCAT focuses on developing multifaceted microanalysis facilities with insertion device and bending magnet beamlines.

Research based on these facilities involves a wide range of issues of central importance for worldwide human welfare. The needs span problems in both pure and applied science and engineering. For example, scientists will use the EnviroCAT facilities to study the following:

- Behavior of toxic and radioactive species in the environment, including sorption and reactions on mineral and biological surfaces, migration through the biosphere and crust, stabilization mechanisms, and remediation processes
- Development and evaluation of remediation technologies
- Preventive measures for fungal attack on agricultural crops and wood products
- Dynamics of fluid transport through Earth's crust, relevant to groundwater contamination
- Bioavailability of toxic species in the environment
- Microscale analytical techniques, such as x-ray fluorescence, XAFS spectroscopy,
- Micro-tomography, and surface studies using crystal truncation rod diffraction are crucial in this research for providing atomic- and molecular-level information on structures and reactions.

EnviroCAT has an approved Letter of Intent from the APS, has established residence at the MRCAT where synchrotron-based environmental studies are active, and design and development activities for the beamlines and associated instrumentation continues to expand the analytical options for researchers.

The scientific justification for the merger of the EnviroCAT with MRCAT has been approved by the APS scientific Advisory Committee.

# 1 MANAGEMENT AND ORGANIZATION

## 1.1 Mission

National Risk Management Research Laboratory's participation in MRCAT and other beamlines of the APS advances the mission of providing state of the art x-ray analytical capability in support of environmental science and remediation technology-related research. These activities benefit Agency research activities by:

- Providing state-of-the-art materials analysis capability.
- Establishing a stimulating and collegial environment for conducting cutting edge research that will benefit continued progress toward global sustainability, materials management, pollution prevention, and public health
- Fostering multi-disciplinary, internal and external research collaborations
- Assisting the Agency, ORD laboratories, and other Agency laboratories in their mission to conduct research on ways to prevent pollution and protect human health
- Developing innovative analysis and preparation techniques as well as maintaining proficiency with state-of-the-art practice techniques
- Earn national recognition for its unique capabilities, as well as its research quality and productivity

## 1.2 Quality Policy

It is the policy of NRMRL that systematic planning shall occur for all NRMRL research products. Depending on the scope of research, as well as on any contractual or Agency requirements, planning documents (including QAPPs) appropriate to the scope are developed. The NRMRL Division QAMs participate in the planning process by providing QA expertise and guidance on the QA requirements for research activities.

## 1.3 Applicability of this Document

### 1.3.1 Programmatic Scope

A user agreement is signed by the home institution and is a prerequisite for experimental work at any of Argonne's user facilities. The Department of Energy has formulated master agreements that cover liability, intellectual property, and financial issues. NRMRL has a non-proprietary user agreement currently in place through the APS. As a non-proprietary user, the following conditions are applicable to the research activities conducted at the APS by NRMRL researchers:

- Access to the User Facility is based on a peer-reviewed general user proposal process or as a MRCAT member institution.
- User may access the User Facility with or without collaboration of Argonne researchers.
- Users provide sufficient information to ensure that each planned experiment can be performed safely.
- Argonne personnel provide appropriate safety training and oversight.

- Users are not charged for "machine time" or potential agreed upon collaborative effort of Argonne researchers; users may be charged for ancillary materials, supplies, and services obtained from the User Facility.
- Users must intend to publish their research results in the open scientific literature. Research results are available to the public.
- Users may retain title to their researchers' inventions, and Argonne retains title to its researchers' inventions; the Government retains a royalty-free license and some oversight of utilization efforts.
- The experiment is not directed towards producing a specific commercial end result (e.g., marketable product).

The Land Remediation and Pollution Control Division (LRPCD) leads the NRMRL collaborative team for EPA participation at MRCAT, with Dr. Kirk Scheckel serving as the technical lead and facilitates access to the MRCAT facilities for agency researchers. Dr. Scheckel provides technical expertise and serves as the primary point of contact for those intra agency and external collaborators who wish to access the APS facilities using the agency collaborative agreement or via general user proposals. As an expert in the area of environmental application of synchrotron radiation, Dr. Scheckel provides training to other users, as well as offers technical assistance. The NRMRL research mission is focused on providing research support at the APS in the following key areas:

- Materials management research studies providing innovative solutions for managing municipal, industrial, biological, and emerging wastes and materials, such as nanomaterials
- Land management research studies that reduce and control risks associated with prior and future uses
- Land systems decision analysis research to develop multi-attribute datasets needed to make sustainable land use and materials management decisions

The equipment the APS is an important aspect of the NRMRL advanced materials analysis capabilities and supports the goals described in EPA's strategic plan, particularly the Agency's aim to provide researchers with the technologies they need to ensure the safety and protection of public drinking water, land, and air. In addition, the analytical equipment can benefit researchers in all ORD laboratories as well as potentially other EPA research centers across the nation.

The MRCAT is located at the Advanced Photon Source (APS) at Argonne National Laboratory in Illinois. MRCAT is an informally recognized compilation of shared tools, staff, and activities from NRMRL and collaborators. Supervision of the APS laboratory space, equipment and staff ultimately falls to the APS staff. Formal requests to initiate research activities and use instruments within MRCAT are coordinated through Dr. Scheckel. Beamline access is ultimately the responsibility of the APS administration. Online scheduling for research access is implemented and utilized by all researchers. This ensures that the requested activity occurs in a time-frame compatible with other beamline users, and a formal management mechanism is in place to oversee activities within MRCAT.

A description of the beamlines, equipment and support apparatus to which NRMRL has access through the MRCAT and other general user access beamlines is provided as an appendix to this document.



NRMRL requests to use MRCAT can be split into two types: “routine” sample analysis and major research activities. Time, sample condition, number of samples, and other factors will be considered. Cost of expendable supplies, should be agreed to prior to project start. Research should occur only after health and safety plan (HASP) and quality assurance project plan (QAPP) are approved.

NRMRL users include EPA Principal Investigators (PI) and research staff that use or rely on the instruments to complete their research. Specifically, EPA research staff including engineers, chemists, and technicians. Contract staff, students, and post doc researchers also constitute EPA users at the APS.

## 1.4 Quality System Roles and Responsibilities

As a collaborative access association, the NRMRL MRCAT and general user collaboration at the APS serves to provide a user group with diverse and wide ranging research goals to pool financial and technical resources to achieve research objectives.

The nature of the individual research project at the APS will determine the nature of the QA planning and review process. EPA researchers may participate in a collaboration at MRCAT or general user proposal where the primary responsibility for QA planning is with the collaborating organization(s). Nevertheless, it is incumbent on the ORD and NRMRL researchers to determine that the QA planning is appropriate to the intended research objective, and that this planning is adequately documented.

### 1.4.1 QA Responsibilities

NRMRL has established minimum QAPP requirements for various research types. As described in the NRMRL QMP, a graded approach to QA is applicable for both intramural and extramural activities.

Effective quality planning requires clear identification of project goals and intended use of data. The EPA PI for the project determines the appropriate QAPP requirements to be used, with the assistance of the division Quality Assurance manager (QAM). The PI will consult all with project participants to ensure that the planning documentation is appropriate to the nature of the research.

The process for identifying the QA requirements for extramural projects are described in the NRMRL QMP. The management controls defined in the NRMRL QMP require the generation of a project tracking list by each Division, including both intramural and extramural projects. A similar practice is suggested for EPA facilitated projects at the APS. This tracking list should be reviewed and updated regularly to ensure that all projects are identified. This practice helps to ensure that all projects have appropriate QA documentation. For intramural projects, the Quality Assurance Review Form is also used to document the QA requirements.

It is the PI's responsibility to identify and involve any and all appropriate sponsoring organizations, responsible official(s), project personnel, stakeholders, scientific experts, etc. (e.g., all customers and suppliers) in the planning the project activities conducted through the MRCAT and general user proposals. This is true for both intramural and extramural projects. Once the planning is complete, project documentation should include (at a minimum) a complete description of the following:

- the project's goals, objectives, questions, and issues to be addressed,
- the project's schedule, resources (including budget), milestones, and any applicable requirements (e.g., regulatory requirements, contractual requirements),

- the type of data needed and how the data will be used to support the project's objectives,
- the quantity of data needed and the specification of performance criteria for measuring data quality,
- how, when, and where the data will be obtained (including existing data) and identification of any constraints on data collection,
- specifications of needed QA/QC activities to assess the quality performance criteria (e.g., QC samples for both the field and laboratory, audits, technical assessments, performance evaluations, etc.), and
- How the acquired samples will be analyzed (either in the field or the laboratory), evaluated (i.e., QA review, validation, verification), and assessed against the quality performance criteria and for its intended use.

Once project planning is complete and documented, it is the PI's responsibility to submit the QAPP to the QA Manager. The QA manager performs a review against QAPP requirements according to the QA category to determine whether planning requirements have been met and project goals can be met, consistent with the QA requirements.

The QA manager review report shall indicate deficiencies if any and recommendations for corrective actions as appropriate. Detailed review comments shall be provided to the PI identifying any deficiencies, and resolution of all findings shall be accomplished and documented before any research is started. If there are no findings then the QAPP will be circulated for concurrence by NRMRL line management, indicated by the manager's signature on the QAPP title page. Additionally, the QAPP will be circulated to all participants for review and concurrence. Over the course of a research activity, NRMRL line management, EPA RAP project leads, and the QA Manager will communicate to identify QA issues and progress updates. This communication will serve to coordinate the QA effort and ensure all personnel are aware of their responsibilities for the EPA QA program.

Each NRMRL research project utilizing APS instrumentation through the MRCAT or general user proposals shall have written Standard Operating Procedures (SOPs) or protocols for routinely used procedures, methods, and techniques. SOPs should be written in the current NRMRL format for SOPs where the procedure is specific to NRMRL research activities. The SOPs should document all aspects of the data collection process including sample preparation, calibration, data reduction to final reported results, and definition of QA elements of the data collection procedure. The SOP facilitates the consistent application of analytical techniques and provides a reference for other intramural or extramural scientists to verify the quality of the data.

A central file of all SOPs will be maintained on the NRMRL SOP intranet webpage. A copy of the approved SOP must be sent to the QA Manager and should follow the procedures given by "Standard Operating Procedure (SOP) for the Creation of Standard Operating Procedures in the National Risk Management Research Laboratory", March 2013. SOPs are reviewed bi-annually and tracked by the QAM as part of the review process.

The use of APS instrumentation will be monitored and recorded using a system of login forms and log books, consistent with the applicable EPA policy PPM 13.2, "Paper Laboratory records".

### 1.4.2 Quality Management

NRMRL and EPA\ORD division QA managers work with agency researchers to prepare QA planning documentation, perform QA assessment of data, and QA review of research products. The QA oversight responsibilities and functions are defined in the NRMRL QMP.

### 1.4.3 Staff Responsibilities

Research staff have the primary responsibility of ensuring that QA planning is completed before data collection begins, that procedures defined in the QAPP are followed in the data collection process, and that data quality is verified to meet the requirements of the QAPP, and validated to support the successful achievement of research objectives.

### 1.5 Dispute Resolution

Disputes related to the EPA Quality system are addressed through line management and/or EPA RAP project managers. Within EPA line management, disputes will be initially addressed at the branch level, with elevation to the division management if a resolution cannot be reached initially. If the dispute cannot be resolved at the division level, then NRMRL IO will then be involved, with ultimate responsibility for dispute resolution residing with the NRMRL laboratory director.

Disputes related to the MRCAT non-proprietary user agreement are handled according to the guidelines in Article XIV of that agreement. Such disputes may be related to the assignment of authorship and rights to the technical information produced by research at the APS, and are addressed by the terms of the ANL non-proprietary user agreement.

## 2 QUALITY SYSTEM COMPONENTS

### 2.1 Overview of Quality System

The unique properties of synchrotron radiation are its continuous spectrum, high flux and brightness, and high coherence, which make it an indispensable tool in the exploration of matter. The wavelengths of the emitted photons span a range of dimensions from the atomic level to biological cells, thereby providing incisive probes for advanced research in materials science, physical and chemical sciences, metrology, geosciences, environmental sciences, biosciences, medical sciences, and pharmaceutical sciences. The features of synchrotron radiation are especially well matched to the needs of nanoscience.

The fundamental parameters that we use to perceive the physical world (energy, momentum, position, and time) correspond to three broad categories of synchrotron experimental measurement techniques: spectroscopy, scattering, and imaging. By exploiting the short pulse lengths of synchrotron radiation, each technique can be performed in a timing fashion.

- Spectroscopy is used to study the energies of particles that are emitted or absorbed by samples that are exposed to the light-source beam and is commonly used to determine the characteristics of chemical bonding and electron motion.
- Scattering makes use of the patterns of light produced when x-rays are deflected by the closely spaced lattice of atoms in solids and is commonly used to determine the structures of crystals and large molecules such as proteins.

- Imaging techniques use the light-source beam to obtain pictures with fine spatial resolution of the samples under study and are used in diverse research areas such as cell biology, lithography, infrared microscopy, radiology, and x-ray tomography.

Quality Controls provide the means to verify the data such that the intended use of the data to achieve project specific research may be validated. The necessary quality controls will be specific to the analytical technique, and to the nature of the research. QA aspects related to the specific analysis technology will be defined in this document. QA aspects specific to the research project objectives will be further defined in addenda to this document for each project.

## 2.2 Analytical Calibration Procedures

All analytical procedures, as well as QA/QC procedures, are defined within the APS specific SOPs and Quality Assurance Projects Plans (QAPP).

### 2.2.1 Calibration Procedures and Frequency

Established calibration procedures and calibration verification procedures are followed as defined in the APS Synchrotron SOP.

Specific instructions on the calibration procedure and frequency of calibrations are defined in the Synchrotron SOP. In general, instruments are either calibrated daily or have calibrations verified daily through the analysis of one or more calibration standards. As a check for each sample, the calibration standard is typically collected simultaneously with a reference detector with each sample scan to verify instrument calibration. The SOP and/or QAPP establishes the standards required, the linearity requirements, and continuing calibration acceptance criteria.

- All calibrations are verified initially by the analysis of an independent check standard.
- Calibration verification or check standards are analyzed during the daily analytical runs at a predetermined frequency, with correction actions initiated in the event that one of these check standards fail acceptance criteria. Recalibration is required in the following cases:
  - Failure of passing method acceptance criteria upon initial calibration.
  - Failure of daily calibration checks.
  - Major maintenance performed on analytical system.
  - Other specific requirements are found in the instrument or method specific documentation and/or project QAPP.

### Energy calibration of the beam.

The beam is calibrated by scanning a metal foil or reference compound, particularly for the metal of interest. For example, if one is examining lead (Pb) (all beamlines have reference foils) the Pb foil is placed where a sample may go or an additional detector (dedicated to collecting the reference foil spectrum congruently during sample scans) may be used. Metal foils are often utilized as they tend to be stable and data tables for K- and L-edge absorption energies are readily available: <http://xdb.lbl.gov/>. Once a scan of the reference foil is collected, the derivative of the scan can be displayed on the computer. The first inflection point of the metal foil or compound is calibrated to the specific binding energy of the material used (See examples at:

<https://www.synchrotron.org.au/images/beamlines/XrayAbsorptionSpectroscopy/foils.pdf>). For Pb metal foil the LIII energy is 13,035 eV. If the measured value is off, the monochromator can be realigned via software to the correct energy position. This can also be accomplished later in the Athena program. However, for redox sensitive material, such as arsenic (As), it is important that the beam is carefully calibrated. Since elemental As foil is not possible, typically a reference metal foil that is close to the As K-edge energy (11867 eV) is used, usually gold (Au) (11919 eV LIII-edge); likewise, for As, a sodium arsenate salt has a verified absorption edge of 11874 eV and can be utilized for calibration purposes. This ensures the beam is consistently calibrated and eliminates this step in the data reduction when using Athena. Since data collection can be viewed real time, simultaneous collection of the calibration standard with an additional downstream detector for each scan provides additional assurance the monochromator is properly calibrated or signals that a calibration is needed.

Data resulting from the analyses of samples are reduced according to protocols described in the laboratory standard operating procedures (SOPs). The analyst is responsible for reducing data from analytical testing to a format that allows for easy review of the data. This data report is subsequently validated as described in Section 2.3. All information used in the calculations (e.g., raw data, calibration files, and background-correction protocols) is recorded in order to enable reconstruction to the final result at a later date. Information on the preparation of the sample (e.g., weight or volume of sample used, percent dry weight for solids, extract volume, dilution factor used) is also maintained in order to enable reconstruction of the final result at a later date. An explanation of how results from blanks are treated in calculations is clearly described.

Data reduction is accomplished in one of the following ways:

1. Manual computation of results
2. Manual input of raw data for computer processing
3. Direct acquisition and processing of raw data by a computer

If the analyst uses manual computation methods, all steps in the computation process are shown including the equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. These calculations are maintained with original raw data (e.g., chromatograms). If appropriate, a reference to a method or SOP is used.

If the analyst enters raw data for computer processing, a copy of the input is kept and uniquely identified with the analytical batch number and other information as needed. The laboratory sample ID's, the project sample ID's, and their corresponding raw data for input are easily identifiable. The computer algorithm is documented to show the exact computational operations used. The computer software used for the data processing must meet the requirements of section 6, Computer Hardware and Software.

If data are directly acquired from the instrumentation and processed, the analyst verifies that the following are correct:

- Laboratory sample ID
- Project sample ID
- Calibration factors and/or response factors
- Output parameters such as units

- Numerical values used for detection limits when an analyte is reported as undetected

## 2.3 Quality System Tools

### 2.3.1 Planning Tools

This QMP, the NRMRL QMP, and the ORD QMP are the guiding documents for defining the QA planning requirements for all research conducted by EPA researchers at the APS.

The QAPP serves as the primary planning tool. A QAPP is developed, or an existing QAPP may be revised as appropriate to address the specific QA requirements of a research project.

Formal Data Quality Objectives (DQO) will developed if the project is assigned EPA QA category "A". The requirements for the DQO process may be found in [EPA QA-G4 Guidance on Systematic Planning using the Data Quality Objectives Process](#).

### 2.3.2 Implementation Tools

QA is implemented through the EPA formal QA training requirements for new hires, QMP revisions, and as implemented at the division level. Core QA training will include the EPA policy and procedure manual (PPM) sections relevant to QA for laboratory research. This includes the following final PPM sections:

13.1, "Development of QA/QC Policies and Procedures for ORD Research Activities"

13.2, "Paper Laboratory Records"

13.3, "implementing QA under the ORD RAP"

13.4, "QA/QC Practices for ORD Laboratories Conducting Research"

13.7, "Use of the Graded Approach for QA of Research"

SOPs are developed for all routine research activities at APS (See "Synchrotron SOP", others)

### 2.3.3 Assessment Tools

The following assessment tools may be used as appropriate to ensure that research follows the approved QAPP, research data meets data quality acceptance criteria as defined in the QAPP, procedures are followed as defined in the applicable SOPs, and that the data collected is of sufficient type and quality to achieve the research project objectives:

- Data Quality Audits
- Technical System Audits
- Surveillances
- Peer Review
- Performance Evaluations

Specific elements and procedures related to each of the above tools may be found in the NRMRL QMP and/or ORD QMP.

The analyst who generates the analytical data has the primary responsibility for the correctness and completeness of the data. Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This

application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently. General validation procedures are described below

## 2.4 Data Reporting

Data is submitted in both electronic and/or hard copies as appropriate. Contents of Final Data Report. The report or research product should include readily identifiable QA elements which are consistent with the direction of this QMP, the QAPP, and the SOP. The QA section of the research product should include discussion of deviations or special circumstances which may impact the data quality. The report indicates the units for each measurement and each matrix, and whether data has been reported on a wet or dry weight basis.

### Data Collection Information

This section lists the data collection parameters that should be reported in a manuscript where a table of EXAFS results are included.

The synchrotron should be listed with the following instrumentation parameters for the synchrotron:

- The beamline should be given including a reference for the standard operating procedures used for data collection.
- The mechanism for x-ray energy selection, such as monochromator type (double crystal Si(111) monochromator with pre-monochromator slits of 0.6 mm).
- The mechanism for harmonic x-ray energy rejection (for example, detune the second monochromator crystal by X%, Rh coated harmonic rejection mirror).
- Detectors used to monitor I0 and It, including fill gasses for ionization chambers and filters.
- The cross section of the incident x-rays on the sample.
- Method used to calibrate the monochromator energy
- Reference sample.
- Scattered x-rays used to monitor reference sample.
- Sample type (powder, paste, aqueous, solid).
- For sample type powder, define the particle size, how mounted (tape mount, pellet). For sample type paste, define the concentration and components of the sample if known) as well as the sample holder size.
- For sample type aqueous, define the solution concentration and components (if known).
- For sample type solid, define the concentration and composition, as well as the size of the sample (weight, and dimensional size for monolithic materials).

XAFS analysis results reports should include the following specific information:

- The data range in k, and the fit range in R
- A method for accounting for each EXAFS parameter for each path included in the model (S02, CN, R, sigma2, E0).
- A description of the software and data reduction procedures used to perform the analysis.
- Results should be presented with the data overlaid with the model fit (in k or R) and a table of EXAFS results with uncertainties for each parameters.
- Variations for specific analysis procedures including PCA and LCF should be included in the report.

An example of the information to be reported for for calibrations and quality control for X-ray absorption near-edge structure spectroscopy follows:

*“Pb LIII-edge (13, 035 eV) X-ray absorption spectra (XAS) were collected at the Materials Research Collaborative Access Team’s (MRCAT) beamline 10-ID, Sector 10 at the Advanced Photon Source at the Argonne National Laboratory, Argonne, IL. The electron storage ring was operated in top-up mode at 7 GeV with a ring current of 101 mA. The pre-monochromator slit width was set to 0.5 mm and the liquid N<sub>2</sub> cooled Si(III) double crystal monochromator utilized a platinum-coated mirror to reject higher-order harmonics. The monochromator beam energy position was calibrated by assigning the first inflection of the absorption edge of a Pb metal foil (13035 eV) and the Pb foil was scan congruently with each sample for calibration verification. Spectra were collected in fluorescence mode with either a Lytle (purged with argon) or a solid-state silicon drift detector at room temperature with a beam size of 1 mm by 1 mm. Ionization chambers for I<sub>o</sub> and I<sub>t</sub> were purged with nitrogen and argon, respectively. The samples were prepared as thin pellets using an IR pellet press and samples were secured to sample holders using Kapton tape. For each sample, a total of fifteen to seventeen scans were collected using the Lytle detector or 4 to 5 scans using the solid state detector and averaged. Data were analyzed using the Athena software program (Ravel and Newville, 2005). Sample spectra were compared with synthesized minerals and mineral specimens acquired from the Smithsonian National Museum of Natural History (USA). All minerals were verified with XRD before use as reference materials.*

*Soil Pb speciation was determined by comparison of Pb standards to the field samples via Linear Combination Fitting (LCF). Linear Combination Fitting refers to the process of selecting a multiple component fitting function with a least-squares algorithm that minimizes the sum of the squares of residuals. A fit range of -20 to 50 eV was utilized for the X-ray absorption near-edge structure (XANES) portion of the XAS spectra and up to four variables. The best fitting scenarios are determined by the smallest residual error ( $\chi^2$ ) and the sum of all component fractions being close to 1. Detailed descriptions of the fitting procedure are described elsewhere (Isaure et al., 2002; Roberts et al., 2002, Scheinost et al., 2002). The reference samples used in the LCF model were plumboferrite (PbFe<sub>4</sub>O<sub>7</sub>), plumbonacrite (Pb<sub>5</sub>O(OH)<sub>2</sub>(CO<sub>3</sub>)<sub>3</sub>), chloropyromorphite (Pb<sub>5</sub>(PO<sub>4</sub>)<sub>3</sub>Cl), Pb-sorbed to hydroxyapatite complex (Ca<sub>5</sub>Pb<sub>5</sub>(PO<sub>4</sub>)<sub>6</sub>(OH)<sub>2</sub>), galena (PbS), hydrocerussite (Pb<sub>3</sub>(CO<sub>3</sub>)<sub>2</sub>(OH)<sub>2</sub>), anglesite (PbSO<sub>4</sub>), plumbomagnetite (PbFe<sub>2</sub>O<sub>4</sub>), litharge (PbO), lead hydroxide (Pb(OH)<sub>2</sub>), and Pb sorbed to humic and fulvic acids, goethite, gibbsite, kaolinite, bentonite, and calcite. “*

See NRMRL-LRPCD SOP-01, “Preparation of Reports to External Data Users” for further guidance on reporting.

## 2.5 Graded Approach

The EPA graded approach to QA of research is implemented for all ORD research activities. The applicable PPM 13.7 provides the specific roles and responsibilities for EPA staff under the graded approach.



## 3 PERSONNEL QUALIFICATIONS AND TRAINING

### 3.1 Training Policy

It is ORD policy that all persons performing, managing, or directing projects have appropriate training for their assigned work. The greatest challenge of a research laboratory in meeting its obligations and commitments, both in the short and long term, is its management of human resources. Maintaining a highly qualified and well trained staff is essential in meeting organizational goals and continuous quality improvement. Therefore, an effective training program will result in increased productivity, continued expansion of laboratory skills and capabilities. Requirements for research at the APS are augmented by the specific training provided by the APS, and by the EPA MRCAT technical lead.

### 3.2 Training Requirements

The NRMRL technical lead provides training according to the following objectives:

- Increase productivity to maximize utilization of limited beamline time.
- Develop and ensure adequate training for scientists so that highly qualified staff is available to meet current and future NRMRL research needs.

Principal investigators and managers conducting research at the APS are required to have either a Ph.D. or an M.S. or a B.S. degree with demonstrated ability to independently plan, conduct, and publish scientific research. The NRMRL APS technical lead trains researchers in QA/QC related to their tasks.

Investigators receive formal QA/QC training from the QAM according to the requirements of the NRMRL QMP. Training in QA/QC to meet the goals of the NRMRL and APS QMP will be done primarily through the EPA QAM. Specific training needs will be assessed by the QAM appropriate to the research objectives and EPA\ORD QA policies. All EPA research staff are required to complete NRMRL QMP training, and to understand the requirements of EPA ORD PPM 13.2, "Paper Laboratory Records", and PPM 13.4, "Minimum QA/QC Practices for Conducting Research in ORD Laboratories". The QA manager is responsible for documenting QA training.

#### 3.2.1 Technical Training

Personnel who have been trained and qualified conduct all quality-related activities performed at the APS. Training and qualification documentation should be maintained in personnel training files. These files include the following information on each employee:

- Internal/external training
- QA training
- Health and safety training
- Hazardous Waste Management

General requirements for technical training are presented in this section. Laboratory-specific procedures are discussed in the NRMRL Synchrotron SOP.

Experienced NRMRL research staff will train new research staff using the APS specific SOPs, as well as their experience and knowledge. Qualification is typically accomplished by performing the task with the guidance of the EPA MRCAT technical lead and meeting established performance and quality assurance acceptance criteria. The training activities include reading the method or SOP, performing the method or SOP under direct supervision of the MRCAT technical lead, and acceptable performance on a

proficiency sample(s). The APS technical lead is responsible for documenting technical training and/or qualification and maintaining a copy of all training documents for review by the QA manager.

All research staff are required comply with the SHEM Cincinnati laboratory Chemical Hygiene Plan (CHP), and any additional requirements specific to the APS.

### 3.2.2 General Training Requirements for APS Users

A series of core user training courses are required for all users of the APS facilities. All core courses can be taken via the Remote Training web site which is accessed through the ANL website, [http://www.aps.anl.gov/Safety\\_and\\_Training/Training/](http://www.aps.anl.gov/Safety_and_Training/Training/). See the user training requirements summary for general information. Special training requirements may apply in certain situations. This will be identified in the user account in the User Portal and in emails from the User Office.

How to use the Remote Training system:

- Go to Remote Training.
- Enter your user badge number. You will be shown a page with core courses listed.
- To take any course, click on the link for the course.
- You can print a PDF version of the course as a study guide, take the course and test, or take a challenge exam.
- If you elect either of the latter two options, you will need to re-enter your badge number and web password.
- Successful completion of the course will be automatically recorded in Argonne's Training Management System within an hour.
- Users may be required to take additional web-based training courses for certain situations. These courses are available in the "Additional Courses" section on the On-site User Facility Training page. These courses require your Argonne domain account and password (only available inside Argonne firewall).
- Sector orientation is given at the beamline.

### 3.2.3 QA Training

APS users are expected to complete required EPA QA training before any data collection activities at the APS. This includes the QA orientation training for new employees on the ORD QMP, and the applicable EPA policies for laboratory research (PPM 13.4) and recordkeeping (PPM 13.2). Additional requirements include familiarization with this QMP, and the NRMRL Synchrotron SOP. QA orientation training is documented by the EPA Quality Assurance Manager. The APS MRCAT technical lead is responsible for ensuring that all required APS training is completed before data collection begins, including that the APS QMP and synchrotron SOP are reviewed and that the QA requirements understood and implemented at the beamline.

## 4 EXTRAMURAL AGREEMENTS

EPA has specific requirements for extramural agreements such as are implemented for research through the MRCAT at the APS. These requirements are detailed or are incorporated by reference to the ORD and NRMRL QMPs. All requirements of the QMP are applicable to extramural agreements entered into by EPA for the purpose of conducting research at the APS.

#### 4.1 MRCAT Extramural Agreements

The nature of the MRCAT at APS means that much of the research is collaborative involving one or more other organizations by means of an IAG. The QA requirements for the MRCAT IAG are as described in section 4.3 of the NRMRL QMP for an IAG. The responsibility for QA must be negotiated within the agreement. The EPA PI/technical leads in consultation with the EPA QAM must agree on and document (on a QARF and in the agreement) which organization will take the lead for QA, the specific person from the organization who will be responsible, and the QA requirements that will be adhered to during the agreement.

## 5 DOCUMENTS AND RECORDS

### 5.1 ORD Records Management Policy

NRMRL LRPCD follows the guidance given in the Agency's Information Resources Management Policy Manual Chapter 10, Records Management. The Office of Information Resources Management (OIRM) in EPA's Office of Administration and Resources Management (OARM) is responsible for the management of EPA's records. The legal requirements for EPA's record management is contained in the Federal Records Act of 1950, as amended (44 U.S.C.), and the Paperwork Reduction Act of 1980 (44 U.S.C.) (5).

Administrative records management of research activities at the APS is with the EPA APS MRCAT technical and administrative lead, Dr. Kirk Scheckel. This person is responsible for records inventory, classification, document control, vital records, records life cycle and disposition, and information security. These records include: budget information, records for managing contracts and subcontracts, property inventory, and other organizations receiving funds from EPA. Records of Interagency Agreements and Cooperative Agreements are also maintained by agency Project Officer.

Research data management is the responsibility of the individual researcher and/or project leader. Data are recorded in laboratory notebooks following the EPA ORD Laboratory Research Notebook Policy (PPM 13.2). These records will be managed according to the applicable EPA records retention requirements.

### 5.2 Document Control and Storage

#### 5.2.1 APS Data Management Practices

Users of the APS are responsible for meeting their Data Management obligations to their home institutions. The APS does not provide any long-term data archiving or management service. Once data have been provided to each APS experimental group, the user is responsible for managing the long-term retention of his/her data, and should not rely on the APS for this service.

The data management practices at individual beamlines/sectors at the APS vary significantly, with some beamlines providing specific resources that could help users meet some of their data management needs and obligations. Users should consult a local beamline representative for a more complete description of how and how long their data will be made accessible.

Therefore, NRMRL users will implement a document control program to ensure that all necessary documents are prepared and completed during the course of a project, and that the documents can be tracked from initiation to disposal. The ORD policy for paper records. PPM 13.2 "Paper Laboratory Records" sets forth the requirements for research notebook recordkeeping by EPA users at the APS. EPA draft policy PPM 13.6 for electronic records defines requirements for electronic data management

and retention. NRMRL users may utilize the existing NRMRL SOPs for electronic data management and results reporting:

- NRMRL-NRMRL SOP-13, “Laboratory Instrument Analytical Data Backup”
- NRMRL-LRPCD SOP-01, “Preparation for Reports for External Data Users”.

#### 5.2.2 Project Files

Project related files are records that are specific to a project. The following minimum records are filed together as one unit that is called a Project file:

- Original work order/ COC and other pertinent documents received with samples.
- Communications between the laboratory and the USEPA
- Corrective Action Reports
- Laboratory data packages
- Finalized data report
- Electronic data
  - Raw data collection at APS has a permanent record on APS servers
  - EPA personnel access APS servers to secure a copy of data either by direct download from an APS computer or via FTP software connection to APS servers
  - EPA personnel are responsible for collected data which includes requirements for QA as well as integrity of data interpretation.

#### 5.2.3 Quality Assurance Files

### 5.3 QA Documents and Records

Quality Assurance files are records that affect reported data, but are not limited to a specific project. They cover all aspects of laboratory operation from sample receipt to sample disposition. Following are examples of what are classified as QA records (but not limited to):

- Laboratory Notebooks
- SOPs
- Certification and training records
- Quality certificates
- Laboratory audits
- Personnel training files
- Nonconformance records

## 6 COMPUTER HARDWARE AND SOFTWARE

Guidance published by the Office of Environmental Information (OEI) ensures that computer hardware and software used in environmental programs meets technical requirements and quality expectations. Specific guidance includes EPA Directive 2100, Information Resources Management Policy Manual, and EPA Order 2165.1, Software Management and Piracy Policy.

These comprehensive guidance documents address many issues regarding the use of computer systems, including purchase of computers, purchase or development of software, design of databases, records management, security, and data standards. OEI has specified Agency-wide standards for software, and OEI approval is required for nonstandard software for Division-wide or broader implementation.

Project-specific requirements for hardware configurations and for configuration control are specified by the PI in specific project QAPPs. Project-specific requirements and plans for software testing, validation, and documentation, whether the software is developed or purchased from an outside source, are also described in the QAPPs for those projects.

To assist in the implementation of software and hardware standards, each NRMRL location employs a designated Information Technology (IT) Coordinator who advises and assists NRMRL researchers in planning and implementing projects involving software development, database design and development, and other computer-based projects. Funding packages and plans (work or research/QA plans) for all NRMRL projects involving software development or purchase or computer purchase must undergo review and be approved by the IT Coordinator to ensure that this guidance is followed and quality is ensured. Any project that plans to collect significant amounts of data should be reviewed by the IT Coordinator and the NRMRL Information Manager to ensure there is sufficient lead time, capacity, and budget to accommodate the planned data acquisition, storage, and analysis.

#### Computer Software Quality Assurance

Electronic data systems for data collection, manipulation, and generation are maintained by the APS beamlines. Responsibilities include installation, validation, modifications, and maintenance of all software used in beamline operations, whether it is automated data collection systems.

The following documentation is maintained:

Documentation of Manufacturer Operational and Maintenance Manuals. A library of operating procedure is established, maintained and controlled. For systems created by the laboratory, SOPs are developed and implemented. Master tapes/CD ROMs are kept under locked storage with limited access.

Documentation of Validation. Software verification procedures are enforced and documented for all software before use. Verification of software tests all options of the program, such as the theory, basis of computation, and its ability to store and manage files. Before use, the software is proven that it functions for its intended purpose.

Documentation of Software Modification. Software configuration control procedures are enforced and documented for requesting, testing, approving, and issuing software modifications. Whenever a modification has been performed, re-verification is necessary for the software program as a whole, not only for the modification performed

Documentation of Maintenance. Current software usage is audited once a year. This is to ensure the approved version of each package/spreadsheet is being used, and that no unapproved modifications have been made.

Historical Records. A historical record of all software used within the laboratory is documented, maintained, controlled and retained according to the appropriate EPA retention schedule.

## 7 PLANNING

Access of NRMRL researchers to the APS can be accomplished by two means: 1) General Users Proposals for competitive access to any of the APS's beamlines or 2) granted access time to MRCAT based on IA

contributions from one's respective Division. Access to MRCAT is a pay-to-play process since contributions are typically provided from individual PI research budgets and Divisional support.

At three points in the year when GUPs are due for review, beamtime requests (BTRs) for MRCAT members are requested from each member for each beamline (10-BM and 10-ID). MRCAT members generally have first choice of dates during the experimental cycle. Kirk Scheckel works with NRMRL contributors for selection of time and information in BTRs. For LRPCD time, Dr. Scheckel generally completes the BTRs, while Rick Wilkin (GWERD) will make requests for GWERD participants. When contributing, Chris Impellitteri or Mike Schock submit BTRs for WSWRD.

Once BTRs are submitted with our preferred dates for each beamline, approximately two weeks later the requestor will receive electronic confirmation from the MRCAT scheduler. If particular dates are in conflict, the MRCAT scheduler communicates with the requestor to find a resolution. If EPA personnel submit GUPs requesting beamtime, those proposals are reviewed by a proposal review panel to determine merit and recommend amount of time. GUPs awarded time will be communicated directly to the EPA requestor.

Once the BTR or GUP is approved, the EPA requestor must then submit an experimental safety assessment form (ESAF) that documents the type of experiment and detectors needed, number of samples, chemical safety information for each chemical planned for analysis, and other safety considerations (nanomaterial protocols, electrical issues (if for example we bring our own equipment), off gassing measures, etc.)

### 7.1 Strategic Planning

NRMRL involvement at the APS for synchrotron data collection should be in line with a RAP specific task so that the effort supports and advances the activities and needs of the EPA.

### 7.2 Program Planning

NRMRL researchers should be intimately involved with RAP planning to envision the application of APS technology to support research efforts. This is a good opportunity to seek funding support for the MRCAT IA to provide guaranteed access to MRCAT rather than relying on competitive general user proposals.

### 7.3 Project Planning

During project planning as part of the RAP procedure, individuals will need to develop project specific descriptions of the planned effort. This document should address the need of APS services and can serve as a basis for QAPP development as a requirement for access to the APS.

## 8 IMPLEMENTATION OF WORK PROCESSES

### 8.1 Site Access

Argonne National Laboratory is a controlled-access facility. A visitor's pass or a user badge is required to enter the Argonne campus.

Advance notification of a site visit is required; see the steps below. When you arrive, you will be asked to present your identification documents, and then you will be given your pass or badge. You can then come and go freely during the period for which your pass or badge is valid. You must wear your pass or

badge visibly at all times while on site. Important: Please ensure that you have received all necessary confirmations from the ANL User Office before you travel to Argonne.

STEPS FOR USERS WORKING ON SITE: These steps apply to individuals who will conduct hands-on work at the APS.

- Register at the user registration login to receive a badge number
- Notify APS of arrival/departure dates via the User Portal ("check-in" tab, APS badge number required)
- Bring the appropriate identification documents
- Verify you are covered by a user agreement between your institution and APS
- Complete training (APS badge number required)
- Report to the visitor center to get your user badge (or after hours to main gate for gate pass)
- Proceed to APS User Office (Building 401, Room B1100) to complete your check-in

It is essential that sample integrity be maintained from the time of collection to disposal. Requirements for sample container preparation, sample receipt and login at the laboratory, shipment of samples to other laboratories, sample storage and holding time requirements should be established in the project specific QAPP and or the SOP.

## 8.2 Project Sample Container Preparation

Sample containers must meet project requirements as defined in the QAPP or in the applicable SOP. Sample containers may be reused, but are pre-cleaned prior to reuse. Appropriate DOT rules and regulations must be observed. Additionally, USDA permitted soils must be accounted for and approved for access to the APS. Cleaning requirements for all sample containers are summarized below.

- Each sample container is thoroughly washed with a non-phosphate detergent.
- Each sample container is thoroughly rinsed with deionized water to completely remove the detergent.
- Each sample container is triple rinsed with a volume equal to one-tenth the containers capacity; of a 10% (v/v) acid solution (the acid used is dependent upon the analyses for which the sample is collected).
- Each sample container is triple rinsed with a volume equal to one-tenth the containers capacity, reagent grade water and air dried.

Depending on the analytes to be sampled for, the acid wash may be omitted from the above summary or additional steps may be added to the above summary to achieve conditions that will have the least impact on sample integrity.

### 8.2.1 Sample Shipments to Laboratory

Samples may be transported to the APS by technicians collecting samples in the field, shipping samples by courier or commercial carrier. Shipping requirements for samples to the APS can be found at: [http://www.aps.anl.gov/Safety\\_and\\_Training/shipping\\_intro.html](http://www.aps.anl.gov/Safety_and_Training/shipping_intro.html). Requirements are summarized below.

Each sample container will be identified with a sample label that adheres to the container and that is able to withstand being soaked with water without losing its integrity. It is suggested that a piece of clear tape be placed over the label to protect it. When needed, a sample tag is attached to the same

container by a string. The information on the label and/or the tag for each sample will be identical and will include project identification, sample source, date of sampling, preservative, and analytes requested. Each label or tag will be given a unique ID number during sample login.

A Chain of Custody (COC) will accompany each sample shipment. At a minimum, the COC will include the project identification, the sample source, a sample type, the date and time of sampling, the analyses requested for each sample, the sampler's signature and any comments or remarks required to ensure proper QA/QC procedures are followed in processing or analyzing the samples. The person who ships the samples to the laboratory will sign off on each COC to release the samples to the lab. If sample login procedures are implemented at the laboratory, the ID number assigned at login must be entered on the COC for each sample, and must also be marked on the sample container.

- Samples will ship in sturdy, insulated coolers or other secure containers as appropriate to the required sample preservation. Samples must be packaged with packing material to protect sample containers from damage during transit. Coolers will be chilled with bagged ice or blue ice.
- COC forms and other documents will be placed inside sealable plastic bags and taped securely to the underside of the cooler lid.
- Samples anticipated to contain high contaminant concentration will be shipped in separate coolers from samples of low or unknown concentrations and notice of the suspected high contaminant concentration will be recorded in the comments section of the COC.
- Outside sources shipping samples by courier must seal coolers with straps and/or tape.
- Coolers may be shipped by commercial or government carrier and will be received by the research staff as soon as practical from the time the samples were collected.
- All labels, tags, CoC forms, field notebook entries must be made with waterproof non-erasable ink. If an error is found, it will be crossed out with one line, corrected, and initialed by the person correcting the error.
- Sample shipment to the APS must conform to DOT shipping regulations.

#### 8.2.2 Sample Receipt, Log-In, and Storage Procedures

Samples associated documentation, including COC forms, are reviewed for completeness and consistency. Sample storage conditions must be adequate to maintain the integrity of the sample. These procedures are summarized below.

- Samples delivered to APS are received by a beamline staff member. The coolers/samples are taken to the sample login area. The shipping containers are unpacked and the contents are verified against the CoC records.
- Each sample is thoroughly examined to verify that proper sampling, preservation, packaging, and labeling techniques have been employed. This verification of sample integrity is documented on the CoC.
- The researcher is responsible for assessment of the condition samples upon arrival.
- A binder containing a manual log of all samples and/or an MS Excel spreadsheet is used to track sample information. Information recorded includes the date and time of receipt, the assigned unique identification (ID) number, analyses requested, preservative, etc.
- The samples are stored in limited-access refrigerated storage. The requirements for storage temperature and holding times are included in the project-specific QAPP. Samples that have been analyzed and are awaiting data validation or disposal are considered archived samples. The laboratory should maintain a storage system and a dedicated area for archived samples separate from "active" samples, to the extent that this is possible at the APS beamline. See the



NRMRL LRPCD SOP-02 “Sample Management Procedures” for general guidance on sample storage and management.

### 8.2.3 Health and Safety and Environmental Compliance

While synchrotron research relies on X-ray radiation for its operation, shielding and safety precautions at these facilities essentially eliminate this concern. Users at synchrotron facilities are required to take extensive training and refresher testing to qualify to operate at beamlines. The list of training requirements at the APS include:

- APS User Orientation
- Argonne National Laboratory User Facility Orientation
- General Employee Radiation Training (GERT)
- Cyber Security
- Electrical Safety Awareness Training
- Sector-specific Orientation
- Other topic-specific safety training (e.g., laser safety, cryogenic safety, etc.)

All laboratory and field projects conducted at US EPA facilities are required to have a current, project-specific Health and Safety Plan (HASP). For research conducted at the APS, the EPA requestor must submit an experimental safety assessment form (ESAF) that documents the type of experiment and detectors needed, number of samples, chemical safety information for each chemical planned for analysis, and other safety considerations (nanomaterial protocols, electrical issues (if for example we bring our own equipment), off gassing measures, etc.) It is expected EPA research staff will follow safety best practices as would be required when working in EPA ORD laboratories. These include the proper personal protective equipment for handling hazardous materials, clear labelling of samples, reagents, and standards used in research at the APS, and using best practices in the storage of samples, standards, and reagents.

## 9 ASSESSMENT AND RESPONSE

Quality assurance assessment and response for research at the APS follows the guidance and definitions found in section 9 of the NRMRL QMP. Section 2.2.3 of the NRMRL QMP lists the types of audits and assessments used to evaluate programs and projects within NRMRL. All of these assessment types may be utilized for QA activities related to EPA research at the APS.

Tracking and resolution of corrective actions identified by audits or assessments is a basic management responsibility in NRMRL, starting at the level of TLP or COR, and flowing up to the branch chief, Division Director, and Laboratory Director. Section 9.6 of the NRMRL QMP provides procedures relating to performing corrective actions for audits and assessments.

Reports from the QA Manager to line management and RAP project leads are provided to evaluate effectiveness of quality improvement activities. Recommendations from the research scientists, RAP project leads, and line management may also be included in reports to NRMRL management. These reports will be the documentation for assessing the effectiveness of quality improvement activities and will help provide continuous quality improvement.

## 10 QUALITY IMPROVEMENT

The overall goal of the NRMRL quality system as implemented at the APS is to maintain quality research practices as are implemented in EPA ORD laboratories. This will ensure that high-quality science is generated from research activities and collaborations at the APS. The following steps are incorporated to meet this goal, as defined in the NRMRL QMP:

**(1) Preventive action**

Preventive action is implemented through QA training activities and by the interaction of QA staff and the research staff on a regular basis, to reinforce understanding and implementation of QA practices and requirements. The goal of preventive action is to minimize problems with the quality of data and products through participation in training and communications with QA staff.

**(2) Problem identification and analysis**

Problem identification and analysis is accomplished through QA audits, assessments, and reviews of planning documents and research products.

**(3) Corrective action**

Corrective actions identified as the result of reviews and assessments are resolved by the interaction of the researcher, line management and EPA QA manager to implement corrective actions identified during assessments.

**(4) Improvement of practices and processes**

Best practices should be identified in the assessments and documented in the review and reports generated from assessments. Research management best practices identified and implemented within EPA ORD laboratories should be adapted at the APS wherever possible. This is accomplished by adapting and/or developing innovative QA procedures in collaboration with the EPA researchers working at the APS.

**(5) Assessment of quality improvement.**

Assessment of quality improvement focuses on the scientific and engineering research products generated using research data from the APS. Quality assurance goals are defined in the QA planning documents and are integral to the RAP planning process. These products must advance the goals of EPA research as defined in the RAP planning process and in the collaborative agreements EPA utilizes to produce these products at the APS through the facility of the MRCAT.

## 11 REFERENCES

- 11.1 ORD Quality Management Plan (QMP), June 2012,  
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- 11.2 NRMRL Quality Management Plan (QMP), December 2013,  
<http://v26265ncay514.rtord.epa.gov/p2/nrmrl/quality-management-plan>
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### Appendices:

#### A: MRCAT Instrumentation

#### Attachments

#### 1: APS Beamlines and instrumentation used by MRCAT researchers

## Appendix A: MRCAT Instrumentation

The MRCAT Insertion Device line is fully operational and is used primarily for spectroscopy and fluorescence mapping experiments. The Bending Magnet line is fully operational and is available for spectroscopy, photochemistry, and high energy tomography.

### Insertion Device Line

#### Wide Energy Range Monochromator

We currently have a Si (111) monochromator consisting of a cryo-cooled first crystal designed by the IIT Center for Synchrotron Radiation Research and Instrumentation (CSRRI) and a 250mm long second crystal which provides an energy range of 4.8keV to 30keV from the fundamental reflection. Both crystals have been chemically polished and have been measured to deliver the theoretical photon flux to the experimental station. The second crystal has a piezoelectric tuning actuator with a.c. feedback and a Bragg-normal motion which permits some degree of fixed-offset operation. In combination with the multichannel scaler electronics described below, we are able to take complete EXAFS scans of 1000eV in under 1 min.

#### Harmonic Rejection and Steering Mirrors

The MRCAT beamline has a 60cm long flat harmonic rejection mirror with Pt and Rh coatings. This mirror resides in the experimental station and may be moved vertically in or out of the beam as the experiment requires.

#### Microfocusing

Basic microfocusing capability is now available. our system consists of a separate kinematically mounted table of our own design with a vibration isolation breadboard and positioning systems for sample, microscope and Kirkpatrick-Baez mirrors. The minimum spot size for the current mirrors is approximately  $5\mu\times 5\mu$ . The system has been used for fluorescence mapping and spectroscopy experiments. A new microprobe with x-ray emission spectroscopy capability is being developed.

### Bending Magnet Line

#### Wide Energy Range Monochromator

We currently have an Oxford Si (111) monochromator consisting of a water-cooled first crystal designed by the IIT Center for Synchrotron Radiation Research and Instrumentation (CSRRI) and a 50mm long second crystal which provides an energy range of 4.0keV to 32keV from the fundamental reflection. Both crystals have been chemically polished and have been measured to deliver the theoretical photon flux to the experimental station. The second crystal has Bragg parallel and normal motions and is run at 50% detuning for removal of harmonics. The experimental station is 25m from the source which provides a relatively high photon flux density without the need of focusing optics.

The monochromator is exceptionally easy to set through the use of a GUI which allows the user to select an element to be measured.

### High Energy Tomography and Photochemistry

The Bending Magnet first optical enclosure contains a 1 m long Pt-coated mirror which can be used to tailor the incident spectrum for photochemistry or photolithography. A three axis (horizontal, vertical, rotational) stage can be used to position the samples for irradiation or for high energy (> 100 keV) tomography.

### Detector Systems

The MRCAT sector is currently equipped with 22cm long aluminum spectroscopy ion chambers for use in transmission mode x-ray absorption spectroscopy and 6 smaller Cornell-type ion chambers which may be mounted on the spectroscopy rail for special purposes. Three Lytle-type fluorescence detectors are also available. Data collection is through a standard instrument chain of Keithley electrometers, V-F converters and a 32 channel multichannel scaler. The multichannel scaler permits continuous of the energy and the goniometer detector motors.

The MRCAT instrumentation also includes a single-element YAP scintillation detector and a 19-element solid state detector for dilute XAFS and Fluorescence measurements. At the present time, we are limited to approximately collection times of greater than 6s per data point with the Ge detector. A 4-element Vortex detector is available primarily for use at the Bending Magnet line and additional 4-element Vortex detectors from the APS Detector Pool can be easily integrated into the beamline as needed. Bent Laue fluorescence analyzers are also available upon request.

### Data Acquisition

Data acquisition is handled by the MX system, written by William Lavender. A preliminary manual as well as source code is available at <http://mx.iit.edu>.

## **Appendix J – Sample Custody, Packaging, Shipment, and Analysis**

**Proper packing and shipping is critical to ensuring samples reach the laboratory in good condition.** If samples are damaged or the shipping company will not deliver the samples (such as if the water/ice leaks), resampling may be requested.

Whenever possible, overnight shipment will be on the same day as sampling is completed. All samples **MUST** be shipped overnight to arrive Monday through Friday or hand-delivered. No deliveries are accepted at EPA laboratories on weekends or Federal holidays. Exceptions may be made on a case by case basis dependent on sampling priority/emergency status.

An example Chain of Custody is provided in Appendix J-1. Sample packing instructions are provided in Appendix J-2. Laboratory addresses and sample coordinator contacts are provided in Appendix J-3 for shipment of samples.

Reporting limits and detection limits for EPA and contract laboratory analyses are summarized in Appendix J-4.



## Appendix J-2 – Sample Packing Instructions

### Supply list:

- Sturdy cooler(s)
- Heavy-duty, 30-gallon trash bags (2 per cooler)
- Absorbent material, suggested
- Bubble-wrap style materials, suggested (required if any glass sample bottles are to be shipped)
- **For shipment of sample bottles for analysis of organics (including VOCs/TTHM, HAA9, DBPs, and SVOCs), total phosphorus, TDS, and anions/alkalinity (see Sections 5.2, 5.4, and 5.6):** Fresh ice, approximately 15-25 pounds of ice per large cooler – at least 1/3 of the airspace of the cooler should be ice-filled during moderate weather; more ice should be used if weather is hot (80 F or hotter). Sample containers should be stored on ice as soon as possible after collection. If samples must be held more than a few hours before shipping, fresh ice may be needed for shipping.) – **not required for metals sample bottles**
- Filled and labeled sample bottles, with all lids tightly closed
- Sampling form(s) and chain-of-custody form(s)
- Zip-type plastic bags (Freezer style is best) such as Ziploc (1 per cooler)
- Shipping tape
- Completed shipping labels, with pouches if needed (1 per cooler)
- Paper towels

### Cooler Packing Instructions:

1. Begin with a dry, sturdy cooler. If the cooler has a drain, the drain should be closed and taped shut.
2. If available, place the dry absorbent material in the bottom of the cooler.
3. If available, place dry bubble wrap in the bottom and along the sides of the cooler.



4. Open one of the trash bags. Place the trash bag inside the cooler as a liner.
5. **If the cooler will contain sample bottles for analysis of organics, total phosphorus, TDS, and anions/alkalinity:** Place 1-2 inches of ice inside the trash bag along the



bottom of the cooler. Take care to avoid getting any ice outside the trash bag. Ice may be omitted for coolers containing only metals samples.

6. Open the second trash bag. Place the trash bag inside the first trash bag (lining the cooler again, but with any ice between the two bags).
7. Place sample bottles upright in the bottom of the cooler (inside both trash bags). If needed, dry off the sample bottles before placing them inside the dry trash bag. Depending on the number of sample bottles, additional cooler(s) may be needed.



8. Double check the chain-of-custody form against the labels and number sample bottles in the specific cooler. If more than one cooler is used, the chain-of-custody form(s) should match the contents of the individual cooler.
9. Place the chain-of-custody form(s) and sampling form(s) corresponding to the samples in the cooler inside a sealed Zip-type plastic bag.
10. After confirming the contents of the cooler, tie the second (inside) trash bag shut, taking care to press out excess air from the bag. Do not use drawstrings, use the sides of the trash bag to tie the bag shut securely. Check that the cooler lid is able to close (rearrange samples or remove excess air as needed).
11. **If the cooler will contain sample bottles for analysis of organics, total phosphorus, TDS, and anions/alkalinity:** Place 2-3 inches of ice on top of the closed second (inside) trash bag, inside the first (outside) trash bag. Take care to avoid getting any ice in the bottom of the cooler outside the trash bags.
12. Tie the first (outside) trash bag shut, taking care to press out excess air from the bag. Do not use drawstrings, use the sides of the trash bag to tie the bag shut securely. Check that the cooler lid is able to close (rearrange samples, shake ice to fill space between samples, and/or remove excess air as needed).
13. If the cooler is not full, use additional packing materials (i.e., bubble wrap) to fill remaining airspace.



14. Tape the sealed Zip-type plastic bag to the top inside of the cooler. This bag should contain the chain-of-custody form(s) and sampling form(s) corresponding to the samples in that cooler.
15. Shut the cooler lid (there should be no gap) and use packing tape to bind the cooler shut. Packing tape should make two to three full passes around the cooler at two different places.



16. Affix the shipping form to the outside of the cooler.
17. Drop off the cooler at the shipping vendor (e.g., FedEx) before the last pick-up for the day.

**If the cooler will contain sample bottles for analysis of organics, total phosphorus, TDS, and anions/alkalinity:** If the last pick-up is missed, the cooler should be repacked with fresh ice.

Photo credit: <https://www.youtube.com/watch?v=iSO7gjD5KF0> (Pace Analytical Services: How to Pack a Cooler)

## Appendix J-3 – Sample Shipment Instructions

In general, send all samples to:

Robert Snyder  
US EPA Region 5  
Chicago Regional Laboratory  
536 S. Clark Street, 10th Floor  
Chicago, IL 60605

Before shipping samples, please notify the CRL Sample Coordinator (312-353-9078, Thompson.robert@epa.gov) to arrange for sample receipt.

If faster turn-around time is needed, an alternative qualified laboratory may be used. Field personnel should notify the Water Division Technical Contacts if this is needed. The alternative laboratories listed below have been identified for potential analysis of certain project samples; sample shipment instructions are provided below.

- Region 1 (metals analysis, as needed)

Samples Receiving / Dan Boudreau  
US EPA New England Regional Laboratory  
Office of Environmental Measurement & Evaluation  
11 Technology Drive  
Chelmsford, MA 01863-2431

Before shipping samples to Region 1, please notify the Region 1 Sample Coordinator (Doris Guzman, 617-918-8618, guzman.doris@epa.gov) to arrange for sample receipt.

- Region 4 (metals analysis, as needed)

Samples Receiving / Jeffrey R. Hendel  
U.S. EPA - Region 4 Laboratory  
980 College Station Road  
Athens, GA 30605

Before shipping samples to Region 4, please notify the Region 4 Sample Coordinator (Direct: 706-355-8839, Cell: 706-340-2145, hendel.jeffrey@epa.gov) to arrange for sample receipt.

- Region 7 (metals analysis, prior to March 2016)

Samples Receiving / Nicole Roblez  
U.S. EPA - Region 7 ENST/LTAB  
300 Minnesota Ave.

Kansas City, KS 66101

Before shipping samples to Region 7, please notify the Region 7 Sample Coordinator (913-551-5130, [roblez.nicole@epa.gov](mailto:roblez.nicole@epa.gov)) to arrange for sample receipt.

- Region 10 (metals analysis, as needed)

Samples Receiving / Gerald Dodo  
U.S. EPA Region 10 Laboratory  
7411 Beach Dr. East  
Port Orchard, WA 98366

Before shipping samples to Region 10, please notify the Region 10 Sample Coordinator (360-871-8728, [Dodo.Gerald@epa.gov](mailto:Dodo.Gerald@epa.gov)) to arrange for sample receipt.

- PHILIS Contract Laboratory (metals analysis for selected Faucet Filter Evaluation samples [see Section 5.1], metals analysis for Aerator Particulate samples [see Section 5.5], TTHM and HAA9 analysis for Distribution System samples [see Section 5.2], and various analyses for Health and Direct Contact Exposure Evaluation samples [see Section 5.4])

PHILIS CSS-Dynamac  
c/o EPA Region 2  
2890 Woodbridge Ave, Bldg 238  
Edison, NJ 08837

Before shipping samples to the PHILIS Contract Laboratory, please notify the sample coordinator contact (Sang Chung, 219-477-8860) to arrange for sample receipt.

## **Appendix J-4 – Reporting Limits and Detection Limits for EPA and Contract Laboratories**

Analyte	Analytical Method	Sample Matrix	MDL - CRL	Reporting Limit - CRL	MDL - R1	Reporting Limit - R1	MDL - R4	Reporting Limit - R4	MDL - R7	Reporting Limit - R7	MDL - R10	Reporting Limit - R10	MDL - Test America	Reporting Limit - TestAmerica	Units	Hold Time	Bottle Type	Preservative					
<b>Metals - Standard List</b>																							
Total Lead	Based on EPA 200.7/200.8	Drinking Water	0.014	0.500		0.20	0.038	0.50	0.35	1.0	0.019	0.50	0.110	0.50	µg/L	6 months	HDPE – 125mL or 1,000-mL	HNO <sub>3</sub> to pH<2					
Total Copper			0.140	1.00		0.20	4.1	10	0.66	2.0	0.16	1.0	0.750	5.00	µg/L								
Total Zinc			1.20	10.0		5.0	1.1	10		2.0	0.54	5.0	7.30	50.0	µg/L								
Total Aluminum			0.0270	0.200		0.110	0.017	0.10		0.050	0.0087	0.20	0.00900	0.200	mg/L								
Total Iron			0.0160	0.0800		0.040	0.010	0.10		0.050	0.0030	0.080	0.0160	0.100	mg/L								
Total Calcium			0.0210	0.200		0.100	0.025	0.25	0.07	2.0	0.0053	0.20	0.240	5.00	mg/L								
Total Cadmium			0.000210	0.00200			0.000066	0.00050		0.0010	0.00018	0.0020	0.0000610	0.00200	mg/L								
Total Potassium			0.0720	0.800		1.00	0.083	1.0	0.12	2.0	0.023	0.80	0.0300	5.00	mg/L								
Total Magnesium			0.0220	0.200		0.100	0.025	0.25	0.05	2.0	0.0038	0.20	0.0480	5.00	mg/L								
Total Manganese			0.00160	0.00800		0.020	0.00050	0.0050		0.005	0.00025	0.0080	0.00110	0.0150	mg/L								
Total Sodium			0.0360	0.400		1.00	0.10	1.0	0.02	5.0	0.0063	0.40	0.0680	5.00	mg/L								
Total Nickel			0.00180	0.00600		0.020	0.000046	0.0010		0.0010	0.00036	0.0060	0.00023	0.0400	mg/L								
Total Tin			0.00410	0.0200		0.020	0.0015	0.015	NA	NA	0.0016	0.020	0.0013	0.10	mg/L								
Total Chromium			0.000860	0.00500		0.020	0.0011	0.0050		0.0020	0.00048	0.0050	0.000200	0.00500	mg/L								
<b>Additional Metals - Full List</b>																							
Total Antimony					0.000069	0.0010	NA	NA	0.000033	0.0010	NA	NA	NA	NA	0.00016				0.010	mg/L			
Total Arsenic					0.00015	0.0010	NA	NA	0.00064	0.0010	NA	NA	NA	NA	0.00018				0.010	mg/L			
Total Barium					0.00042	0.00300	NA	NA	0.00083	0.0050	NA	NA	NA	NA	0.0011				0.20	mg/L			
Total Beryllium					0.000210	0.00200	NA	NA	0.00018	0.0030	NA	NA	NA	NA	0.000053				0.0050	mg/L			
Total Boron					0.00470	0.0500	NA	NA	0.0050	0.050	NA	NA	NA	NA	0.011				0.20	mg/L			
Total Molybdenum			0.00160	0.0120	NA	NA	0.00041	0.010	NA	NA	NA	NA	0.00023	0.040	mg/L								
Total Selenium			0.0370	0.0500	NA	NA	0.00046	0.0020	NA	NA	NA	NA	0.00025	0.0050	mg/L								
Total Silver			0.00097	0.0100	NA	NA	0.00060	0.0050	NA	NA	NA	NA	0.000020	0.0050	mg/L								
Total Thallium			0.000050	0.00050	NA	NA	0.000091	0.0010	NA	NA	NA	NA	0.000074	0.0100	mg/L								
Total Vanadium			0.00025	0.0010	NA	NA	0.0019	0.0050	NA	NA	NA	NA	0.00023	0.0070	mg/L								
<b>Total Phosphorus</b>	Based on EPA 365.4	Drinking Water	0.06	0.15	Not applicable		Not applicable		Not applicable		Not applicable		Not applicable		mg/L	28 days	HDPE – 125mL	H2SO4 to pH<2, Cool <6C					
<b>Total Alkalinity (as CaCO<sub>3</sub>)</b>	Based on EPA 310.1	Drinking Water	5.0	20	Not applicable		Not applicable		Not applicable		Not applicable		1.9	5.0	mg/L	14 days	HDPE – 125mL	Cool <6C					
<b>Water Quality Anions</b>	Based on EPA 300.0		Sulfate (as SO <sub>4</sub> )	0.02	0.12	Not applicable		Not applicable		Not applicable		Not applicable		0.13	1.0	mg/L		28 days	Cool <6C				
Chloride			0.03	0.12	0.41									1.0	mg/L								
Fluoride		0.01	0.02	0.009	1.0									mg/L									
<b>TTHMs</b>	EPA 524.2	Drinking Water	In accordance with PHILIS Contract Laboratory SOPs.													14 days	Three 40-mL VOAs	Cool <6C, Sodium Thiosulfate, no headspace					
<b>HAA9</b>	EPA 552.3	Drinking Water	In accordance with PHILIS Contract Laboratory SOPs.													14 days (21 days for extract)	One 250-mL amber glass	Cool <6C, Ammonium Chloride					
<b>TDS</b>	Based on Standard Method 2540C	Drinking Water	20.0	20.0	Not applicable		Not applicable		Not applicable		Not applicable		7.4	10	mg/L	7 days	HDPE – 125mL	Cool <6C					
<b>Turbidity</b>	Based on EPA 180.1	Drinking Water	0.12	0.5	Not applicable		Not applicable		Not applicable		Not applicable		Not applicable		NTU	48 hours	HDPE – 125mL	Cool, <6C					
<b>Hardness, Calculated (as CaCO<sub>3</sub>)</b>	Based on Standard Method 2340B	Drinking Water	0.11	1.32	Not applicable		Not applicable		Not applicable		Not applicable		Not applicable		mg/L	6 months	N/A – metals bottle(s)	HNO <sub>3</sub> to pH<2					

Notes:

Region 7 (R7) reports estimated detections of lead, copper, calcium, magnesium, potassium, and sodium down to the MDL (with J qualifiers) to allow quantification to similar levels as CRL.

TestAmerica reports estimated detections of lead and other metals down to the MDL (with J qualifiers) to allow quantification to similar levels as CRL.

Method Detection Limits - The MDLs will be re-determined before the samples are analyzed per the EPA or contract laboratory's Quality Management Plan which requires MDL studies to be performed yearly.