

Appendix F: Environmental Sampling Protocols

XRF Sampling for Environmental Assessment
SOP 100
ATSDR Exposure Investigation
Jasper and Newton Counties, MO

1. **Purpose:** The purpose of this Standard Operating Procedure (SOP) is to establish uniform procedures for the collection of information for the completion of the indoor environmental assessment and XRF sampling to determine the presence of lead-based paint.
2. **Application:** The procedures outlined in this SOP are applicable to all personnel collecting environmental samples for the Oronogo-Duenweg Mining Belt and Newton County Mine Tailings ATSDR Lead Exposure Investigation in Jasper and Newton Counties, MO.
3. **General Guidelines:** Direct reading XRF measurements will be made on selected interior and exterior surfaces that are painted or varnished. The condition of the painted surfaces will be recorded. The sample collection process is based on the U.S. Department of Housing and Urban Development (HUD) guidelines. A worst-case scenario evaluation will determine sample locations within each room. Areas that have the most potential to be a hazard (i.e., deteriorated paint) will be sampled.
4. **Selection of Sample Locations:** The Home Schematic Form (FORM 100) will be completed.
 - 4.1. XRF measurements will be obtained by taking one reading from each unique test combination of the child's bedroom, kitchen, child's main play area, two exterior walls, and porch.
 - 4.2. Components to be sampled include window components, door components, walls, cabinets, and floors that are painted or coated.
5. **Sampling Equipment:** Sampling equipment will at minimum consist of:
 - 5.1. Portable XRF unit
 - 5.2. Non-alcohol wipes
 - 5.3. XRF calibration source(s)
6. **Method of Sampling:**
 - 6.1. Complete FORM 100
 - 6.1.1. Place pre-prepared ID sticker on top left corner and add date.
 - 6.1.2. Include a room plan sketch on the back of FORM 110 used for each room.

- 6.1.2.1. All schematic diagrams will be labeled using the convention of Main address exterior wall labeled 'A' with sequential lettering (B, C, and D) in a clockwise direction. The room number will be '1' for the child's bedroom, '2' for the kitchen, and '3' for the child's main play area. On the sketch clearly indicate the direction for North.
- 6.1.3. Complete the general information questions for the home.
 - 6.1.3.1. Inspector and location type information.
 - 6.1.3.2. Exterior covering type and water source. (See key at bottom of FORM 100).
- 6.1.4. Complete information for each room to be sampled.
 - 6.1.4.1. Floor is the floor of the house. The front entry floor area is floor '1'. If there is a basement of lower floor than it is indicated as '0'.
 - 6.1.4.2. Indicate floor type from the key at the bottom of the data collection FORM 100.
 - 6.1.4.3. If not wall-to-wall carpet, indicate if piece carpet is present. A 'N' circled indicates no piece carpet present.
 - 6.1.4.4. If the child's bedroom or kitchen is also the child's main play area than indicate here as 'Y'. If not indicate 'N'.
 - 6.1.4.5. Indicate the general condition of neatness of the room on a rating scale (See key at bottom of FORM 100).
- 6.2. XRF measurements are obtained in interior rooms, two exterior walls, and one exterior porch (this should be the MAIN PORCH). One sample is taken from each unique test combination. A test combination is determined by component type and substrate material.
 - 6.2.1.1. **Interior** sampling within each of the child's bedroom, kitchen, and child's main play area (FORM 110).
 - 6.2.1.1.1. One reading representative of the most accessible interior window area. Take the reading on the sash.
 - 6.2.1.1.2. One reading representative of the most accessible outer window area. Take the reading on the sill/stool (where the child has access).
 - 6.2.1.1.3. One reading representative of the most accessible interior door. **Note:** If no door is present, this sample is not taken.
 - 6.2.1.1.4. One reading representative of the most accessible door jamb.
 - 6.2.1.1.5. One representative floor reading, unless carpeted.
 - 6.2.1.1.6. One reading of the most accessible wall.
 - 6.2.1.1.7. One reading of the most accessible baseboard, if present.
 - 6.2.1.1.8. One reading of the most accessible radiator, if present.
 - 6.2.1.1.9. One representative reading of cabinets and/or shelves.

6.2.1.2. **Exterior** sampling (FORM 120).

6.2.1.2.1. Readings are taken from only two exterior walls. The first wall will be the side with the MAIN PORCH, or if no porch then WALL A. The second wall is at the discretion of the Risk Assessor. If there is an obvious difference among the walls, the second wall should be selected to represent this.

6.2.1.2.1.1. From each of the two walls, take one reading representative of each test combination of: wall, window sash, window trough (if available), door, and door jamb.

6.2.1.2.2. Main porch. Only one exterior porch is tested. If more than one porch is present, the Risk Assessor must decide which porch is most representative in usage.

6.2.1.2.2.1. One reading representative of each porch component: floor, banister, column. If doors and windows are present, they should be included as part of 'wall' form.

6.3. Obtaining XRF Measurements.

6.3.1. Perform XRF calibration check prior to use, at the end of each sampling day or every four hours, and if the instrument is knocked, dropped, or other impact, turned off for more than two hours, or has been exposed to extreme temperature changes for more than an hour. Using the 1.02 mg/cm² source (or other as recommended by the PCS). Take three consecutive measurements. If any single measurement is off by more than 0.4 mg/cm², or the average of each of the three measurements is off by more than 0.2 mg/cm², then turn the instrument off, then on again, and repeat. If this occurs again, contact the manufacturer concerning how to correct this.

6.3.2. If surface is visibly soiled or dusty, wipe surface with a non-alcohol wipe as necessary and/or place a piece of plastic or paper (such a tissue) between the instrument and surface. Use a clean piece of paper or plastic that has previously been checked for possible interference. This is to ensure that the XRF window is not contaminated, and sample results are from the paint and not surface deposited material. If this surface will be used for a wipe sample, perform the wipe sample first (See SOP 200).

6.3.3. On FORM 110 for each area tested enter all the following information on a new form:

6.3.3.1. Place pre-prepared ID sticker and add date.

6.3.3.2. Indicate inspector and XRF instrument.

6.3.3.3. For indoor samples indicate room number (1 – child's bedroom, 2 – kitchen, 3 – child's main play area).

- 6.3.3.4. Indicate number of doors and windows in sample area for rooms and walls.
- 6.3.3.5. For each XRF sample taken for the specific components indicated on the form:
 - 6.3.3.5.1. If condition intact or deteriorated:
 - 6.3.3.5.1.1. Intact indicates no obvious visible deterioration.
 - 6.3.3.5.1.2. Deteriorated includes any paint coating on a damaged or deteriorated surface or fixture, or any interior or exterior lead-based paint that is peeling, chipping, blistering, flaking, worn, chalking, alligating, cracking, or otherwise becoming separated from the substrate.
 - 6.3.3.5.2. Estimated percent of total damage area represented by this sample.
 - 6.3.3.5.3. XRF result (mg/cm²) reported by instrument.
- 6.3.4. On FORM 120 for the two exterior/outdoor walls tested enter all the following information.
 - 6.3.4.1. Place pre-prepared sticker and add date.
 - 6.3.4.2. Indicate inspector and XRF instrument.
 - 6.3.4.3. Indicate location letters for Wall 1 and Wall 2. Wall 1 should either contain the MAIN PORCH and/or be Wall A.
 - 6.3.4.4. Indicate number of doors and windows. This is the combined number for the two walls selected and includes those within a porch area.
 - 6.3.4.5. For each XRF reading taken for the specific components indicated on the form:
 - 6.3.4.5.1. If condition intact or deteriorating:
 - 6.3.4.5.1.1. Intact indicates no obvious visible deterioration.
 - 6.3.4.5.1.2. Deteriorated includes any paint coating on a damaged or deteriorated surface or fixture, or any interior or exterior lead-based paint that is peeling, chipping, blistering, flaking, worn, chalking, alligating, cracking, or otherwise becoming separated from the substrate.
 - 6.3.4.5.2. Estimated percent of total damage area represented by this sample.
 - 6.3.4.5.3. XRF result (mg/cm²) reported by instrument.
- 6.3.5. On FORM 120 for the MAIN PORCH enter all the following information.
 - 6.3.5.1. Place pre-prepared ID sticker and add date.
 - 6.3.5.2. Indicate inspector and XRF instrument.
 - 6.3.5.3. Indicate wall letter the MAIN PORCH is located.
 - 6.3.5.4. For each XRF sample taken for the specific components indicated on the form:
 - 6.3.5.4.1. If condition intact or deteriorating:

- 6.3.5.4.1.1. Intact indicates no obvious visible deterioration.
- 6.3.5.4.1.2. Deteriorated includes any paint coating on a damaged or deteriorated surface or fixture, or any interior or exterior lead-based paint that is peeling, chipping, blistering, flaking, worn, chalking, alligating, cracking, or otherwise becoming separated from the substrate.
- 6.3.5.4.2. Estimated percent of total damage area represented by this sample.
- 6.3.5.4.3. XRF result (mg/cm²) reported by instrument.

**Dust Wipe Sampling
SOP 200
ATSDR Exposure Investigation
Jasper and Newton Counties, MO**

1. **Purpose:** The purpose of this SOP is to establish uniform procedures for the collection of interior dust wipe samples.
2. **Application:** The procedures outlined in this SOP are applicable to all personnel collecting environmental samples for the Oronogo-Duenweg Mining Belt and Newton County Mine Tailings ATSDR Blood Exposure Investigation in Jasper and Newton Counties, MO.
3. **General Guidelines:** Samples will be collected from each location type. Wipe sample site selection will be performed after FORM 100 is complete. The sample collection process is based on the U.S. Department of Housing and Urban Development (HUD) guidelines. A worst-case scenario evaluation will determine sample locations within each room. Areas that have the most potential to be a hazard (i.e., near deteriorated paint or lead-paint hazards) will be sampled.
4. **Selection of Sample Locations:** Wipe samples will be obtained from the entryway, primary living area, kitchen, child's bedroom, and child's main play area. Nine samples plus one blank will be collected per household.
 - 4.1. Entryway: A sample will be collected from just inside the entryway on the floor.
 - 4.2. Primary living area: Two samples will be collected, one from the floor and one from the windowsill.
 - 4.3. Kitchen: Two samples will be collected, one from the floor and one from the windowsill.
 - 4.4. Child's bedroom: Two samples will be collected, one from the floor and one from the windowsill.
 - 4.5. Interior play area: Two samples will be collected, one from the floor and one from the windowsill.
5. **Sampling Equipment:** Sampling equipment will consist of a minimum of:
 - 5.1. Disposable gloves
 - 5.2. Individual wrapped sampling wipes
 - 5.3. Measuring tape
 - 5.4. Masking or painter's tape
 - 5.5. Moistened towelettes or baby wipes

- 5.6. Sample tubes
- 5.7. Reusable floor template (optional)

6. Method of Sampling:

- 6.1. Place pre-prepared ID sticker and add date (FORM 200).
- 6.2. Prepare sample collection tube with complete sample number and date. The sample number consists of the ID# and assigned sample number (e.g., D-E-1 for an entryway sample, D-L-F-1 for a primary living area floor sample, etc.). Sample numbers for each type are indicated on FORM 200.
- 6.3. Record all information on FORM 200.
 - 6.3.1. Dimensions of the area wiped should be recorded to the closest quarter inch. For a floor use a clean sampling template or tape to mark out a 12" x 24" sample area. For a windowsill tape a rectangular area adjacent to the window sash, this area should not include edges along the side of the vertical window casing, and should be at least 4" x 4", larger if possible.
 - 6.3.2. If surface being wiped is deteriorated, such as chipping and flaking paint, delaminating, and so on, indicate the condition (Y/N) on FORM 200.
 - 6.3.3. If loose soil/dust is seen in the sample, indicate (Y/N) on FORM 200.
 - 6.3.4. If paint chips are seen in the sample, indicate (Y/N) on FORM 200.
 - 6.3.5. Only comments concerning conditions or sampling procedure that would affect interpretations of results should be recorded.
- 6.4. Put on new disposable gloves for each sample.
- 6.5. When a reusable floor template is used, wipe clean between samples and tape to the floor to keep it from moving while wiping.
- 6.6. To sample floors, remove a sampling wipe from package, carefully unwrap, do not touch other objects.
 - 6.6.1. Place wipe down firmly at an upper corner of the sample area, excessive pressure will cause the wipe to curl and too little pressure will result in poor collection. Make as many "S"-like motions as needed to wipe the entire sample area moving side-to-side. Do not cross the outer border of the template or tape.
 - 6.6.2. Fold the wipe in half with the contaminated side facing inward, take care not to spill dust when folding. Once folded, place the wipe in the upper corner of the sample area and repeat wiping with "S"-like motions to wipe the entire sampling area, this time moving from top-to-bottom. Do not cross the outer border of the template or tape. Fold the wipe in half again with the dust collection side facing inward and make a third pass around the perimeter of the sample area, concentrating on any remaining dust in the corners of the wiping area. If visible dust remains use a second wipe to collect the remaining dust and clearly note on the form the need to composite the wipes for analysis.

- 6.6.3. Place the wipe(s) into the labeled collection tube.
- 6.7. To sample windowsills, remove a sampling wipe from package, carefully unwrap, do not touch other objects.
 - 6.7.1. If the surface is a narrow rectangle, two side-to-side passes must be made over the sample area, the second pass should be made with the wipe folded so that the contaminated side faces inward.
 - 6.7.1.1. Do not attempt to wipe the irregular edges presented by the contour of the window trough or the rounded inside edge of the sill.
 - 6.7.2. If there are paint chips or debris in the sample area of the trough, it should be collected as part of the dust sample.
 - 6.7.3. Fold the wipe with the contaminated side facing inward again.
 - 6.7.4. Place the wipe into the labeled collection tube.
- 6.8. Continue until all wipes of each type have been collected, all waste should be collected and disposed of off-site.
- 6.9. Field sample blanks
 - 6.9.1. A field sample blank for each home is required by the State Public Health Laboratory (SPHL).
 - 6.9.2. Before leaving the dwelling, remove a wipe from the package with a new glove, shake the wipe open, refold it in a manner like the above procedures, and place into a labeled collection tube, clearly labeled "blank".

Soil Sampling
SOP 300
ATSDR Exposure Investigation
Jasper and Newton Counties, MO

1. **Purpose:** The purpose of the SOP is to establish uniform procedures for the collection of soil samples.
2. **Application:** The procedure outlined in this SOP is applicable to all personnel collecting environmental samples for the Oronogo-Duenweg Mining Belt and Newton County Mine Tailings ATSDR Lead Exposure Investigation in Jasper and Newton Counties, MO.
3. **General Guidelines:** A rough sketch of the aerial view of the yard will be made which includes the division and indication of the yard areas into sample site categories for: dripline, yard non-play area, and play area/s. A composite soil sample will be collected from each category. Disposable gloves will be worn for the collection of all samples.
4. **Selection of Sample Locations:**
 - 4.1. Soil sampling will include a composite collected from the general yard non-play area within approximately 100 feet of the structure, dripline within three feet of structure walls, and primary play areas of the child.
 - 4.2. An aerial view diagram of the residence and property will be sketched on the reverse side of the Soil Collection Form (FORM 300). The dripline will include the areas contiguous with and extending three feet from the house walls. The general yard non-play area will extend from the drip line to the yard outer boundaries not to exceed 100 feet or a distance that is reasonably considered to include areas where a child may frequent. Play areas will extend three feet beyond a play area boundary or play equipment.
 - 4.3. Dripline
 - 4.3.1. The drip-line soil composite sampling sites (9) will be located 1 ½ feet away from the wall and any water discharge locations (i.e., see diagram for approximate locations). Adjustments may be made based on field conditions.
 - 4.4. General Yard Non-Play Area
 - 4.4.1. Sampling sites for the yard will be determined by superimposing a “+” using the mid-point of the structure as the center. Sample sites (9) will be taken from each of the four quadrants and combined into one composite (36) sample. Adjustments may be made based on field conditions.
 - 4.5. High Contact/Play Area

- 4.5.1. Play area samples (9) will be taken in a similar manner as the general yard non-play area. Up to two primary play or high contact areas (i.e., gardens) will be sampled.

5. Sample Collection:

- 5.1. Label sample storage container with pre-prepared ID sticker, sample number, and date. Sample numbers will be for general yard non-play area (Y-1), play area (P-1), garden area (G-1), and dripline (D-1). Sequential numbers may be used for additional samples of the same sample type (e.g., P-2).
- 5.2. Each sample location will be recorded using a global positioning system (GPS) unit. The GPS coordinates will be recorded in the field logbook.
- 5.3. Complete FORM 300 for composite sample to be obtained. This will entail:
 - 5.3.1. Place pre-prepared ID sticker and indicate date. EPA may use additional stickers for laboratory use.
 - 5.3.2. Determine the percent of bare ground (exposed soil) to covered ground in the region sampled. Covered ground is considered vegetation and hard surfaces (concrete, asphalt, etc.).
 - 5.3.3. Following sample collection, indicate number of samples used for composite and note any adjustments made based on field conditions.
- 5.4. Use a new pair of disposable gloves for each composite type.
- 5.5. Insert collection instrument ½ to 1 inch into the soil and remove soil.
- 5.6. Remove any vegetation from top of soil sample and add to collection container.
- 5.7. Dispose of any remaining soil and wipe residual soil from sample probe.
- 5.8. Continue the process at each sample site placing each new composite into the sample container until all samples have been collected. Repeat for all composite types.
- 5.9. Unless dedicated equipment was utilized, de-contaminate sample probe by wiping off all visible soil with gloved hand and paper towels. Dispose of all waste off-site.

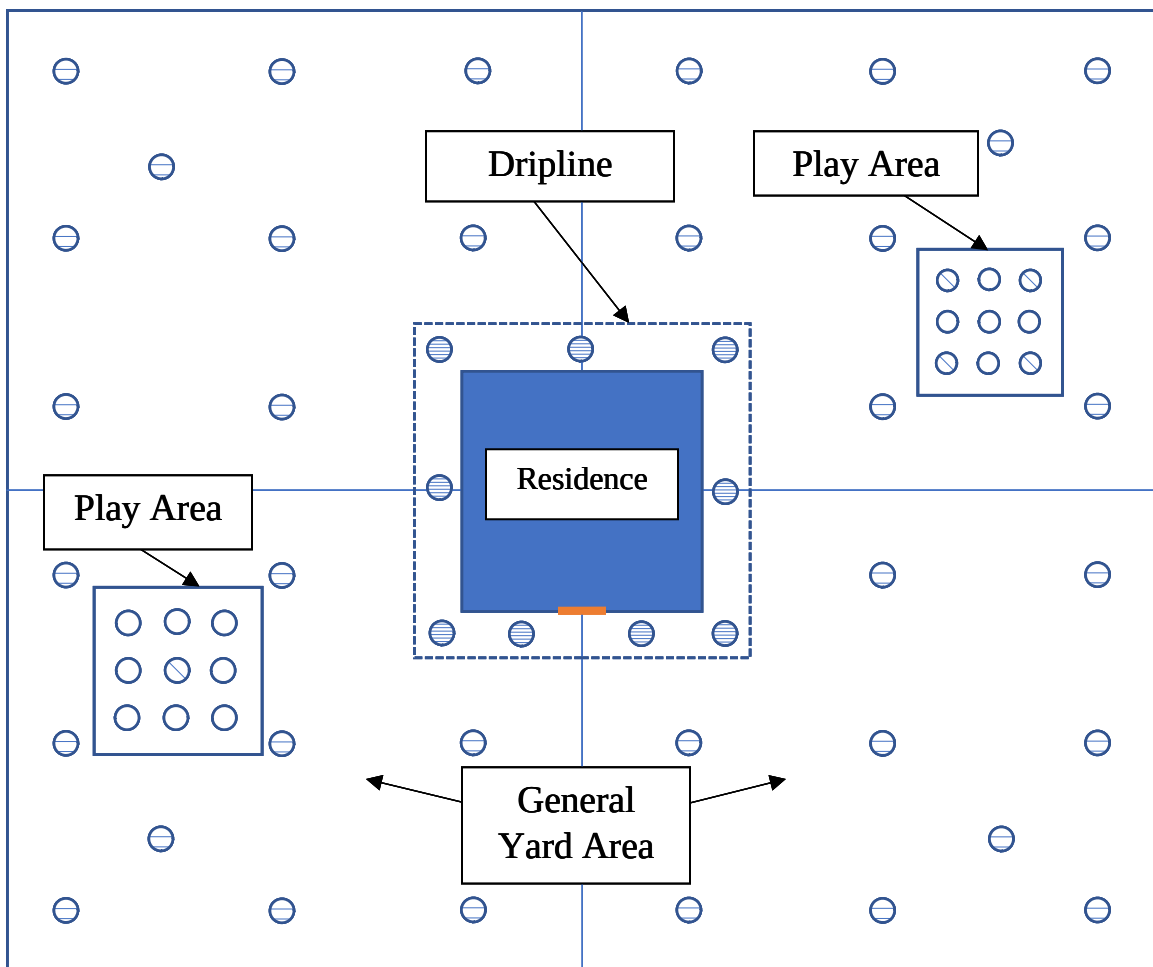
6. Sample Analysis:

- 6.1. Soil samples for each area will be homogenized in a clean, dedicated aluminum pan or plastic bag. Debris, such as sticks and larger stones, will be removed.
- 6.2. Each sample will be taken from the disposable pan or bag, dried, sieved by No. 100 (150 micrometers) sieve, homogenized, and packed into sample containers provided by the laboratory.
- 6.3. The soil samples will be analyzed using a combination of XRF screening and fixed-laboratory confirmation analyses.
- 6.4. Ten percent of the soil samples analyzed using ex-situ XRF will be sent as confirmation samples to the laboratory for lead analysis.

- 6.4.1. The confirmation samples will be selected from the lower, middle, and upper range of concentrations measured by the XRF [EPA, 2018].
- 6.5. The EPA Region 7 Generic Quality Assurance Project Plan (QAPP) for Region 7's Superfund Lead-Contaminated Sites and QAPP Addendum discuss EPA-specific sample documentation and handling.

7. Laboratory Quality Control:

- 7.1. Appropriate quality assurance/quality control (QA/QC) samples also will be prepared and collected including duplicate and matrix spike (MS)/matrix spike duplicate (MSD) samples.
 - 7.1.1. Duplicate samples will be collected at a rate of 10 percent of the total number of soil samples.
 - 7.1.2. All QC samples will be uniquely identified and will be documented in EPA-specific field logbooks and field sheets.
 - 7.1.3. All QC samples of the confirmation samples will be sent to the laboratory for analysis.
 - 7.1.4. Precision for the fieldwork is evaluated by using the relative percent difference (RPD) between the results for the field duplicate samples.
 - 7.1.4.1. An RPD goal of +/- 25% will be used for both field and lab analyses.
- 7.2. The EPA Region 7 Generic QAPP for Region 7's Superfund Lead-Contaminated Sites and QAPP Addendum discuss EPA-specific sample documentation and handling.



- General Yard Area
- ◌ Play Area
- ◌ Dripline

*This diagram provides an example of sample locations and should not be considered prescriptive. Adjustments to sample locations may be necessary depending upon field conditions, home layout, etc., however the number of samples collected per area (36 – general yard, 9 – play area, 9 – dripline) are required.

Private Well Drinking Water Sampling
SOP 400
ATSDR Exposure Investigation
Jasper and Newton Counties, MO

1. **Purpose:** The purpose of this SOP is to establish uniform procedures for the collection of private drinking water samples.
2. **Application:** The procedure outlined in this SOP is applicable to all personnel collecting environmental samples for the Oronogo-Duenweg Mining Belt and Newton County Mine Tailings ATSDR Blood Lead Exposure Investigation in Jasper and Newton Counties, MO.
3. **General Guidelines:** Water samples are to be collected for participants on a private water supply (i.e., private well) from the kitchen faucet. At least 500 milliliters (mL) of water should be collected. Water samples for lead analysis are acidified upon receipt in the laboratory or upon sample collection with the use of pre-acidified containers.
4. **Sampling Equipment:** Sampling equipment will consist at minimum of:
 - 4.1. Disposable gloves
 - 4.2. One-quart laboratory supplied sampling containers
 - 4.3. Masking tape
 - 4.4. Large sealable plastic bag
5. **Method of Sampling:**
 - 5.1. Place pre-prepared ID sticker and add date on FORM 400.
 - 5.2. Label sample container with pre-prepared sticker and sample number W-1.
 - 5.3. Flush water line by letting the water run for at least 5 minutes before collecting sample.
 - 5.4. Place on fresh disposable gloves.
 - 5.5. Rinse container three times with water to be collected.
 - 5.6. Fill with at least 500 mL of water.
 - 5.7. Secure lid, tape with masking tape, and place into plastic bag.
 - 5.8. Please note if there is a water filtration system in the home.
 - 5.9. Affix any EPA-specific laboratory-provided sample sticker and package samples according to laboratory requirements.

**Public Drinking Water Sampling
SOP 500
ATSDR Exposure Investigation
Jasper and Newton Counties, MO**

1. **Purpose:** The purpose of the SOP is to establish uniform procedures for the collection of public drinking water samples.
2. **Application:** The procedure outlined in the SOP is applicable to participants collecting samples for the Oronogo-Duenweg Mining Belt and Newton County Mine Tailings ATSDR Blood Lead Exposure Investigation in Jasper and Newton Counties, MO.
3. **General Guidelines:** Water samples are to be collected by participants who are served by a public or rural water district. At least 250 mL will be collected to evaluate the potential for exposure to lead in pipes and/or kitchen tap fixtures.
4. **Sampling Equipment:**
 - 4.1. MDHSS supplied sampling container.
 - 4.2. Pen or permanent marker.
5. **Method of sampling:**
 - 5.1. Sample should be collected after water has been stagnant in pipes for an 8- to-18-hour period, this is typically first thing in the morning. Please collect this water sample as closely as possible to the day MDHSS will be sampling the paint and dust in your house.
 - 5.2. Fill the container immediately after turning on the faucet or opening the water valve with 250 mL of water.
 - 5.3. Secure the lid and mark the label with the date and time the sample was collected.
 - 5.4. Please note if there is a water filtration system in your home.
 - 5.5. MDHSS will pick up the sample the day they come to your home for dust and paint sampling.