WATER QUALITY PROGRAMS DIVISION

Standard Operating Procedure for the Collection of Water Quality Samples in Streams

Revised and Adopted January 2013

Draft Copy

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STANDARD OPERATING PROCEDURE FOR THE COLLECTION OF WATER QUALITY SAMPLES IN STREAMS

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1.0 General Information

Following is a detailed description of sampling procedures. Efficiency is the key, and finding a comfortable sequence of sampling is essential. This will vary from person to person and from sampling team to sampling team. Yet, employing consistent sampling patterns at every site will maximize the number of sites sampled per day and decrease the chance for introduction of sampling error. Sampling sequence is unimportant. Upon reaching the sampling location, site safety determinations should be made before proceeding. These will be different for wadeable and bridge sites. Please refer to the OWRB safety manual for instructions on how to sample both kinds of sites.

2.0 Definitions/Terms

3.0 Safety

Upon reaching the sampling location, site safety determinations should be made before proceeding. These will be different for wadable and bridge sites. Please refer to the OWRB safety manual for instructions on how to sample both kinds of sites. When regulating the flow of traffic is necessary, please refer to the portion of the safety manual outlining “Traffic Safety Protocols”.

4.0 Quality of the Measurement

When sampling for all programs, Quality Assurance/Quality Control (QA/QC) samples will be routinely collected to assure that environmental samples meet the Data Quality Objectives (DQO’s) that are outlined in the controlling Quality Assurance Project Plan (QAPP). QA/QC sampling is designed to control each step of the sampling process. Blanks are collected to ensure that field personnel are properly cleaning the plastics and glassware used in field sampling. Duplicate samples are collected to ensure that composite samples are properly processed. Replicate samples may be collected to ensure that the sampling methodology employed is collecting a representative sample. Spike or known samples may be submitted to test the efficacy of the analytical laboratory.

4.1 Kinds of Samples

Samples are collected to test various DQO’s precision, accuracy, and representativeness. Following is a short description of each kind of sample. Samples are submitted to the analytical laboratory with other “trip” samples.

- **Lab Blank Sample** A laboratory blank sample is collected to ensure that laboratory cleaning methods are adequate and are not contaminating samples. Reagent grade water should always be used to collect these samples. Submitted on a regular schedule.
- **Field Blank Sample** A field blank sample is collected to ensure that field cleaning methods are adequate and are not cross-contaminating samples. Reagent grade water should always be used to collect these samples. Submitted on a regular schedule.

- **Analytical Blank Sample** An analytical blank sample is submitted to control the methods of the analytical laboratory. Reagent grade water should always be used to collect these samples. Submitted on a regular schedule.
- **Duplicate Sample** A duplicate sample is collected to control the sample splitting method. This sample ensures that composite samples are being collected appropriately. Submitted on a regular schedule.
- **Replicate Sample** A replicate sample is collected to control the general sampling methodology that is being employed. This sample ensures that a representative sample is being collected. Submitted on a regular schedule for some types of samples. Replicate samples may also be submitted to verify the accuracy of analytical results.
- **Spike Sample** A spike sample is a known stock solution diluted by environmental sample. - Submit as required.
- **Known Sample** A known sample is a known stock solution diluted in the laboratory with reagent grade water. - Submit as required.

The Oklahoma Department of Environmental Quality (ODEQ) can provide ampule stock solutions for spike or known samples, or under certain circumstances, these samples may be purchased through a laboratory supplier. Consult a supervisor to determine where to acquire stock solutions. When preparing QC spike and known samples, **record everything you do**! It is essential that all steps of the process be adequately documented.

4.2 Preparation of Samples

4.21 Inorganic Chemistry, Metals, Chlorophyll and Field QA/QC Samples

Analytical Blank Sample

- One sample is collected for each "sampling week".
- The sample is collected from reagent grade water provided by the analytical laboratory.
- The sample will include 1-liter bottles for ice preservation, sulfuric acid preservation, metals, chlorophyll, and field parameters (turbidity, hardness, and alkalinity).
- The metals analytical blank should not be pre-filtered.
- Label with a "31" in the QA code.

Laboratory Blank Sample

- One sample is collected for each "sampling week".
- The sample is collected by running reagent grade water through all plasticware that will be used in the field during a particular sampling week.
- The sample will include 1-liter bottles for ice preservation, sulfuric acid preservation, chlorophyll, and field parameters (turbidity, hardness, and alkalinity). A laboratory blank sample is not collected for metals.
- Label with a "32" in the QA code.

Field Blank Sample

- One sample is collected for each "sampling day" or in an arrangement otherwise made by the project manager.

- The sample is collected by running reagent grade water through any plastic ware that is used at more than one station. Normally, this will only be the splitter churn. Water should be aliquotted in a manner that is consistent with normal sampling procedures.
- The sample will include 1-liter bottles for ice preservation, sulfuric acid preservation, chlorophyll, and field parameters (turbidity, hardness, and alkalinity).
- Metals field blanks will be collected daily using filtration devices and digestion cups.
- Label with a “33" in the QA code.

Duplicate Sample

- At least one sample is collected for each “sampling trip”.
- The sample is collected by using a "churn splitter" to divide water from one sample site into two separate samples.
- The sample will include 2 sets of 1-liter bottles for ice preservation, sulfuric acid preservation, chlorophyll, and field parameters (turbidity, hardness, and alkalinity).
- Duplicate samples are collected for dissolved metals by collecting two filtrate samples from one filtering event.
- Label one sample set with an “11” (environmental sample) and the other sample set with a “21" (duplicate sample set) in the QA code.

Replicate Sample

Isokinetic samplers used by the OWRB include the DH-81 (wading), the DH-95 (cable suspension by either hand line or reel), and the D-95 (cable suspension by reel). General characteristics are outlined in Table 1. The supervising F.T.E. will initially demonstrate the use of the instrument. Detailed instructions for use can be found at the following links sponsored by the Federal Interagency Sedimentation Project (FISP) in coordination with a variety of federal agencies:

- At least one sample is collected for each “sampling trip” when noted as needed by the project quality assurance plan.
- The sample is collected by repeating the exact sampling process collecting two independent sample sets.
- The sample will include 2 sets of 1-liter bottles for ice preservation, sulfuric acid preservation, chlorophyll, and field parameters (turbidity, hardness, and alkalinity).
- Total recoverable and dissolved metals replicate samples are made by collecting two grab samples at the same point in the waterbody. Post-processing should proceed as normal.
- Label one sample set with a “12” (environmental sample) and the other sample set with a “22" (replicate sample) in the QA code.
- As a change to general sampling activities for the Summer of 2008, the sample labeled “12” will no longer be collected. Instead, the replicate sample “22” will be collected at the same site as the environmental sample “11”. To ensure comparability of samples, the total volume collected for the replicate sample should match that collected for the environmental/duplicate sample. Furthermore, the replicate “22” and the environmental “11” should both be collected first from the splitter churn.

4.22 Bacteria QA/QC Samples

Bacteria collections require all QA/QC samples described in 4.21 of this document with the exception of the analytical blank sample (qa code “31”). The analytical blank sample is not collected because no plastic ware is reused.

4.23 Organic Chemistry QA/QC Samples

Organic chemistry collections require all QA/QC samples described in 4.21 of this document with the exception of the field blank sample (qa code “33”). The field blank sample is not collected because no glass or plastic ware is reused during the sampling process.

5.0 Personnel and Equipment

Principle investigators for the OWRB are required to have degrees and/or experience with biological or other applicable sciences. Principle investigators are defined as crew leaders, and this designation may be made upon the leader of a multi- or a one-person crew. Training is required for all SOPs dealing with water quality and quantity collections and measurements as well as habitat assessments and biological collections. In-house training will be conducted for the use of all meters and digital titrators used for water quality or quantity measurements. Investigators must be familiar with OWRB SOP document and all training will follow the methods outlined in that document. Extra training will be provided when new SOPs are developed. Training of field crews will be done through dry run exercises in the laboratory to familiarize field crews with sample collection, sample preservation, instrument operation, calibration, and maintenance. In addition, when new personnel are hired or new methods developed, qualified staff will train on sample collection, measurement, and field analysis methods through side-by-side field trips. These trips will familiarize staff with SOP requirements. When training is considered adequate, a qualified staff member will check field staff for adherence to SOPs.

In most instances, the collection of water quality samples requires only one field person. However, depending on the safety requirements of a particular station, additional crewmembers may be necessary to ensure a safe work zone. Equipment used to collect the chlorophyll-a sample is described in a separate document (OWRB, 2006).

5.1 Sampling Equipment

Sampling equipment in flowing waters fall into two general categories—isokinetic and non-isokinetic samplers. Sampling conditions and flow characteristics will largely determine the type of sampler to be used. Those used by the OWRB are outlined in Table 1. Complete instructions for the use of each sampler are available through the websites linked in the table and further referenced in the bibliography. The types of samples collected by each equipment group are discussed in greater detail in Section 6.0 of this document.

Isokinetic, depth-integrating samplers are considered the most representative because they collect continuously through the water column and water enters the sampler at the velocity at which it is flowing through the stream. These samplers can use rigid bottles or bags for collection and current designs allowing for wading or using a hand line or cable/reel to collect from a bridge. However, utility of the samplers is limited. Because of physical limitations in design, samplers have maximum and minimum depths and velocities at which they can be used (Table 1).

Non-isokinetic samplers are less representative than their counterparts, but are commonly used because of limitations to isokinetic equipment or the objectives of certain programs (Section 6.0). Open-mouth, or grab, samplers are commonly used because of applicability across a number of sampling scenarios, but they are generally considered unrepresentative. These sampler types may be hand-held or suspended (weighted bottle sampler). Open mouth samplers are the most common type of alternative sampler used by the OWRB stream/river programs. Other non-isokinetic samplers include thief samplers (e.g., Kimmerer or van Dorn), single stage samplers, automatic samplers, and pumps. Although these samplers are not currently employed by the OWRB in stream/river sampling, they may have some usefulness in the future. In that event, this SOP will be revised to address their use.

Table 1. Types and characteristics of samplers used to collect in streams/rivers.

Sampler Name	Sampler Type	OWRB Applications	Sampler dimensions (Length-inches, Width-inches, Weight-pounds)	Distance of nozzle from bottom, in inches	Suspension method	Minimum/maximum calibrated velocity, in feet per second	Sampler container, in liters	Nozzle intake size, in inches (max transit rate ratio R_t/V_m)	Maximum sampling depth, in feet
US DH-81	Isokinetic/Depth Integrated	GC, Metals, Organics	6.5, 3.2, 0.5	4	Wading rod	1.5/7.6	1 plastic or teflon with mason jar threads	3/16 (0.2) 1/4 (0.3). 5/16 (0.4)	15 (14 when using 5/16 nozzle)
US DH-95	Isokinetic/Depth Integrated	GC, Metals, Organics	22.0, 4.0, 29	4.8	Hand-line cable with pulley; reel and cable	1.5/7.4	1 plastic or teflon with mason jar threads	3/16 (0.2) 1/4 (0.3). 5/16 (0.4)	15 (14 when using 5/16 nozzle)
US D-95	Isokinetic/Depth Integrated	GC, Metals, Organics	28.5, 6.0, 65	4.5	Reel and cable	1.5/6.7	1 plastic or teflon with mason jar threads	3/16 (0.2) 1/4 (0.3). 5/16 (0.4)	15 (14 when using 5/16 nozzle)
Weighted Bottle Sampler	Open Mouth Sampler	GC, Metals, Organics, Bacteria	varied	varied	Hand-line cable	Undetermined	varied	not applicable	undetermined
Bottle	Open Mouth Sampler	GC, Metals, Organics, Bacteria	varied	varied	by hand	Undetermined	varied	not applicable	undetermined

5.11 Isokinetic Samplers

Isokinetic samplers used by the OWRB include the DH-81 (wading), the DH-95 (cable suspension by either hand line or reel), and the D-95 (cable suspension by reel). General characteristics are outlined in Table 1. The supervising F.T.E. will initially demonstrate the use of the instrument. Detailed instructions for use can be found at the following links sponsored by the Federal Interagency Sedimentation Project (FISP) in coordination with a variety of federal agencies:

- DH-81 Depth-Integrated Sampler
- DH-95 Depth-Integrated Sampler
- D-95 Depth-Integrated Sampler

5.12 Non Isokinetic Samplers

Non-Isokinetic samplers used by the OWRB include the hand held bottle sampler (wading), the weighted bottle sampler (cable suspension by hand line), and the US D-77 without a nozzle (cable suspension by reel or hand line). The supervising F.T.E. will initially demonstrate the use of the instrument.

- The hand held bottle sampler is the simplest form of sampler. A collection bottle is dipped in the river to collect the sample. This is used when the velocity and/or depth is less than the minimum for a depth integrating sampler.

- The weighted bottle sampler is used to sample rivers/streams where the velocity of the water is less than the minimum required for an Isokinetic depth-integrating sampler to be used and where the water is too deep to safely wade. The sampler consists of a weighted device that can securely hold a sample bottle, and that can be attached to a rope for lowering off of a bridge. The device should have sufficient weight to easily draw the bottle under the surface of the water. Care should be taken to not disturb the bottom sediment with the sampler. If the bottom is contacted with the sampler the water should be discard and taken again.

- The US D-77 can be used in the US D-95/95H as a non-Isokinetic sampler without a nozzle attached. It should be used in the same fashion as described in the Isokinetic sampler protocol. The benefit of using it without the nozzle is that you can use it in waters where the velocities are less than the minimum for using an Isokinetic depth-integrating sampler, but faster then is practical to use a weighted bottle sampler.

5.13 Weighted Bacteria Sampler

The weighted bacteria sampler is a weighted PVC pipe with a bacteria bottle holder at the top. It is only used when waterbodies are unwadeable. A supervising F.T.E. will initially demonstrate the use of the sampler.

6.0 Collection of Water Quality Samples

6.1 General Sampling Methodology

A variety of sampling methods are available. These methods include depth-integrated composites (horizontal), composite grab, point grab, and near shore sampling. Depending on various factors, any of the preceding methods can be employed. However, data quality objectives (DQO's) are the most important consideration. Representativeness and accuracy are affected by methodology. Table 1 outlines how each of the sampling methods effects these DQO's based. It is important to note, however, that site conditions may exclude methods with higher representativeness and accuracy, and conversely, may increase DQO level. Unless otherwise noted, all sampling programs will use depth-integrated composites.

Table 1. Relationship of sampling methodology to level of representativeness and accuracy. (* = denotes that level is dependent on site conditions)

Sampling Method	General Level of Representativeness	General Level of Accuracy
Depth-Integrated Composites	High	High
Composite Grab	Medium to High*	Medium to High*
Point Grab	Low to Medium*	Low to Medium*
Near Shore Sampling	Low	Low

The method most suited for a site is dependent upon several things—project data quality objectives (DQO's) and environmental factors. The DQO's will change from project to project and should be clearly defined in the projects workplan or QAPP (Table 2). In addition, several environmental factors will determine what sampling method will be used. The method to use is not the same from month to month and will be affected by current site conditions. The most obvious are the accessibility, depth, and flow of the stream. Depth-integrating samplers require a minimum sampling depth (MSD). The MSD is roughly defined as “the minimum depth at which the mouth of the nozzle can be fully immersed into the water column while remaining perpendicular to the flow of the water”. Samplers used from bridges require an ~ 10 inch MSD to 8 inch MSD while wadeable samplers require an ~ 4 inch MSD. If a site does not meet the MSD for bridge sampling, it must be sampled by wading the stream. A site can be considered wadeable if the site is accessible by road or safely by foot and if the sampling personnel feels comfortable entering the stream. Therefore, if the site is not accessible for wading, a composite or point grab sample must be taken. During extreme flows, depth integration may be impossible. During these situations, a surface grab may be the only possible method. Conversely, during minimal flows depth-integration may be impractical. Even though depths may meet the MSD for depth-integrating samplers, the samplers may stir up bottom sediments, which would bias the sample. In these cases some form of grab sample is appropriate. Figures 1&2 give a guideline on which technique to use for a given situation.

Table 2. General Rule for Sampling for Certain Programs

General Program Types	Depth-Integrated Composites	Composite Grab	Point Grab	Near Shore Sampling
Ambient Trend Program (BUMP)	preferred	if necessary by protocol (document)	if necessary by protocol (document)	not recommended
Probabilistic Monitoring	not necessary	preferred--multiple or braided channel; unevenly mixed	preferred--single channel; well-mixed	not recommended
Use Support Assessment for Grants and Contracts	preferred	if necessary by protocol (document)	if necessary by protocol (document)	not recommended
State Revolving Fund	preferred	if necessary by protocol (document)	if necessary by protocol (document)	if necessary by protocol (document)
Screening Programs	Not necessary but may be desired (document)	preferred	if necessary by protocol (document)	if necessary by protocol (document)

Figure 1. Conditions for various sampling techniques from a bridge.

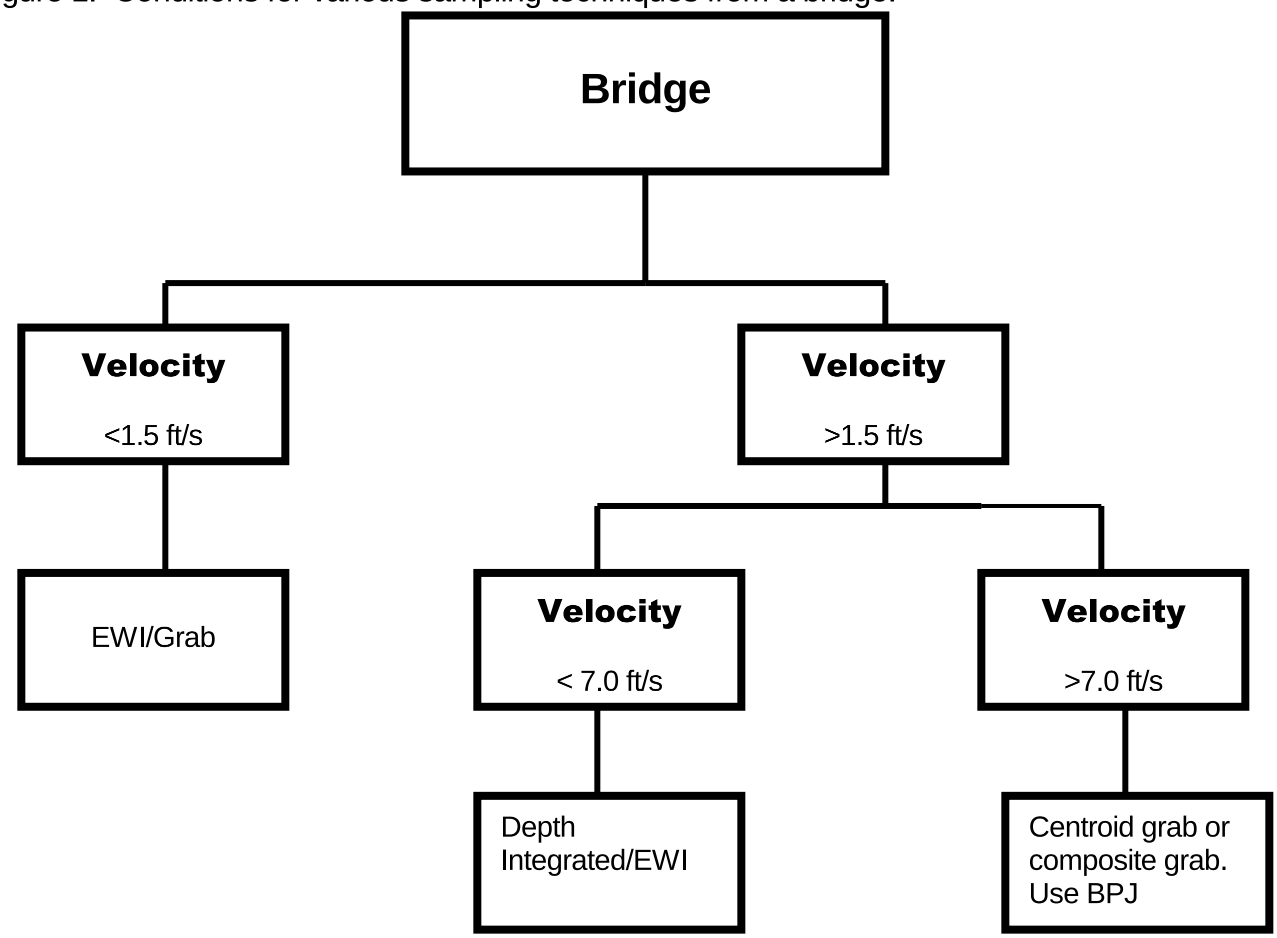
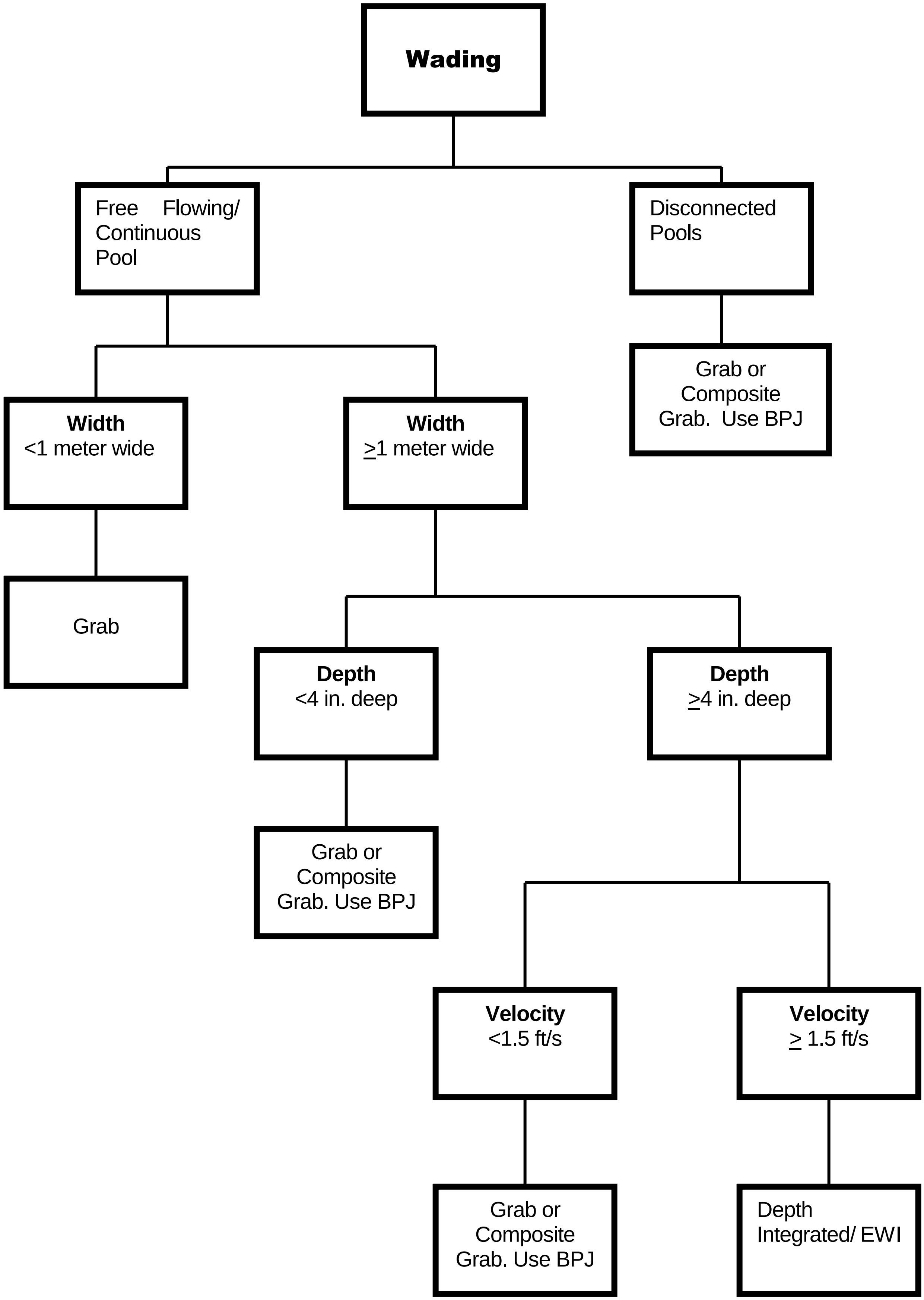


Figure 2. Conditions for various sampling techniques while wading.



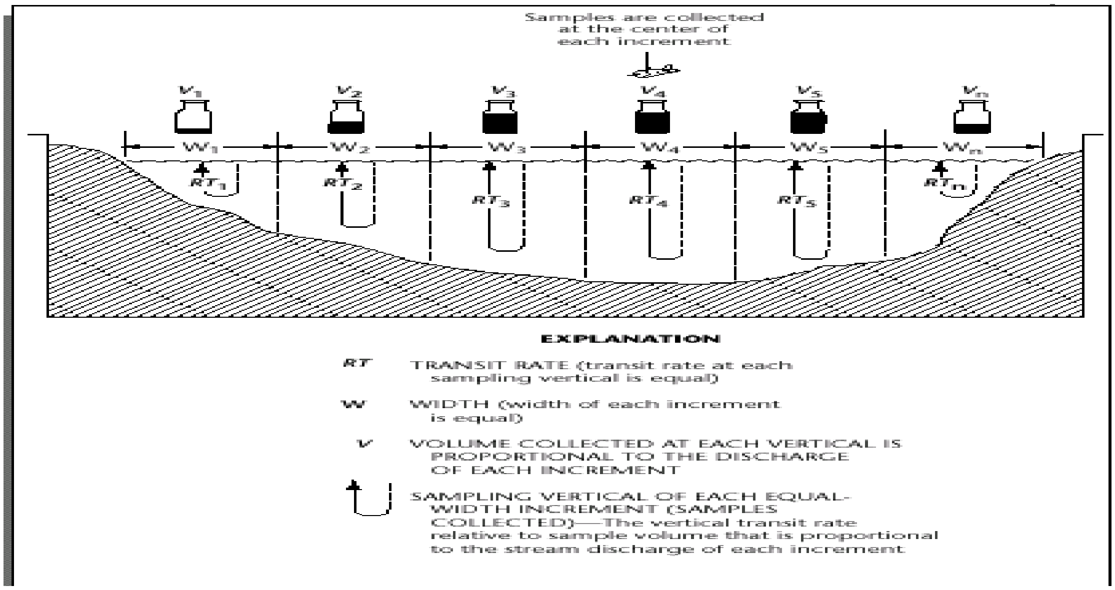
6.11 Depth Integrated Composite Sampling [Depth-Integration/Equal Width Increments (DI-EWI)]

NOTE: This method is time and equipment intensive, and may not be necessary to meet the data quality objectives of all programs.

Depth-integration (D-I) allows a flow weighted sediment sample to be collected by continuously collecting from the surface of the water through the water column. The Equal Width Increments (EWI) collects water at intervals, or verticals, over the cross-section of the entire river or stream. By coupling the methods and compositing the water collected at each vertical, a sample may be collected which represents both the horizontal and vertical profile of the waterbody.

Why is it necessary to go to so much trouble to collect a representative sample from both profiles? The method allows for a representative sample to be collected at all velocities and of all sediment sizes. Most chemicals (nutrients, minerals, toxicants, etc.) in streams and rivers may be attached to sediment. Unfortunately, sediment does not travel at a consistent rate because of two inherent properties—one related to sediment and the other related to the waterbody. The property inherent to sediment is weight. Because sediment particles are not uniform in size and density, they will settle at varying rates in flowing water. Therefore, because a larger or denser particle may contain a larger amount of certain chemical, it is important to collect a representative sample of each particle size. The property inherent to the waterbody is velocity of flow. Streams and rivers do not flow through symmetrical concrete weirs and because of this do not have symmetrical velocities. They flow through channels that are uneven due to deposition and scouring, that meander and move, and have numerous physical obstructions. Therefore, if a stream is viewed at a single point, velocities differ through (vertically) and across (horizontally) the water column. This has a tremendous effect on how sediment flows. Therefore, because these varying velocities may contain differing amounts and types of sediments, it is important to collect a representative sample at all velocities. The entire method is described graphically in Figure 3. Please refer to the graphic throughout this subsection.

Figure 3. Graphical representation of DI-EWI



6.11a Establishing EWI

Following is a detailed, step-by-step process of how to establish EWI.

1. **Determine the inaccessible and accessible portions of the cross-section**. Many things can make a portion of the cross-section inaccessible. These include:
   - Immovable upstream obstructions such as bridge piers (bridge sampling) and brush piles (bridge and wadeable sampling).   Bridge piers are permanently marked on the bridge railing in this way " E| |P ".
   - Immovable instream obstructions such as brush piles (bridge and wadeable), rocks (bridge and wadeable), and illegally disposed of objects (bridge and wadeable).
   - Dangerous flow (bridge and wadeable). If bridge sampling is not feasible and 80 percent of the cross section can be safely waded, the sample can be taken in-stream. If this is done, make every reasonable attempt to collect a representative vertical in the area of high flow.
   - Minimal flow (bridge and wadeable). These areas only become a problem when sample collection cannot be done without inadvertently collecting substrate that is suspended by the act of sampling.
   - Unwadeable depth (wadeable). If bridge sampling is not feasible and 80 percent of the cross section can be safely waded, the sample can be taken in-stream. If this is done, make every reasonable attempt to collect a representative vertical in the area of unwadeable depth.
   - Minimal MSD not met (bridge and wadeable). Refer to previous section on sampling equipment.
   - **Exposed substrate is always excluded.**

2. **Determine the width of the accessible portion of the cross-section.** Because EWI involves taking measurements at incremental points along the cross-section, a tagline is used to accommodate measurements. On bridges, the tagline is painted hashmarks along the bridge wingwall, and in certain instances, along the bridge decking. The areas of the bridge railing that are outside of the normal banks but are in the bank full area of the river are not delineated to 5 and/or 10 feet but only have the 100 and 50 foot marks. A calibrated tagline or tape may also be laid on the bridge deck. All bridges should be marked with the following hashmarks (all OWRB hashes are marked in blue):

   100-foot line—full, triple mark with value beside it (e.g., 1 = 100, 2 = 200, etc.);
   50-foot line—full, double mark;
   10-foot line—full, single mark;
   5-foot line—half, single mark (not done on all bridges);
   2-foot line—half, single mark (not done on all bridges).

   In stream, a calibrated tagline is stretched across the water. Many things can be used as a tagline including measuring tape and calibrated twine. However, because Oklahoma is windy , it can be extremely difficult to set some things, and because measuring tapes and string can stretch over time, taglines may go out of calibration. Therefore, the OWRB uses 1/16 or 3/16 inch coated Kevlar. The taglines are extremely durable, stretch very little over time, and hold steady under most winds. The tagline is calibrated as follows:

   100-foot line—double, red mark;
   50-foot line—single, green mark;
   10-foot line—single, red mark;
   5-foot line—single, black mark (not done on all bridges).

   Under certain conditions, increments can be established without using a tagline. These include extremely wide cross-sections, inaccessible banks or substrates, and unmarked bridges. Increments can be established by pacing cross-section or using consistently spaced objects along the wingwall of the bridge (e.g., vertical I-beams on trestle bridges). Remember, in each situation, care should be taken to use consistent increments of measurement.

3. **Establish the points of collection for each vertical.** The number of verticals needed may vary depending on site conditions. In general, follow this rule of thumb for number to determine verticals needed;

   - Use 5 verticals when accessible cross-section is >1 but < 30 meters wide.
   - Use 10 verticals when accessible cross-section is > 30 meters wide.
   - Grab samples should be collected when accessible cross-section is < 1 meter wide.

NOTE: Site conditions that may change the preceding rule of thumb could be multiple channels, backwater flows, eddies, etc.

To establish the points do the following:

- Beginning at the near end of the accessible cross-section, mark the inaccessible portions while measuring the width of the accessible of the cross-section. (Two widths should be noted on the field sheet: 'total bank to bank width/sampled width'.)
- Divide the width of the accessible cross-section by 6 (5 increments) or 11 (10 increments) to determine the width of each increment. This excludes both near shore areas from the sampling cross-section.
- Starting at the far end of the accessible cross-section, measure out one full increment and place a mark to establish the first vertical.
- Establish each vertical in the same way until the near end of the accessible cross-section is reached (remember to exclude the inaccessible portions).

6.11b Depth-Integration
To use EWI D-I, follow these steps:

1. **Approximate the velocity (none, minimal, light, moderate, heavy, or storm water) at each vertical.** This can be done by visually assessing the displacement of the sampler or wading before sampling. Make notes on amount of weight and/or nozzle size to be used at each vertical.
2. **Approximate the transit rate to be used.** Collect water for the native rinse at the vertical with the greatest estimated flow (highest velocity and deepest depth). Establish the transit rate as the "time it takes to obtain approximately 1-liter of water at the thalweg." This will ensure that an adequate amount of water is collected.
3. **Choose appropriate sized sampler and attach appropriate nozzle.** Remember to maintain an equal rate of transit throughout the water column. Detach the nozzle cap before pouring sample into churn splitter. The general rule of thumb for nozzle size is:

    - No nozzle in minimal velocities (will be using a weighted bottle sampler anyway);
    - 5/16 nozzle for low to medium (narrower) velocities;
    - ¼ nozzle for medium to high (narrower) velocity;
    - 3/16 nozzle for high (wider) to storm water velocities.

4. **At the approximated transit rate, lower and raise the unit through the water column.**
5. **Repeat steps 4 until cross-section is completed.** When sampling only 5 verticals it may be necessary to sample each vertical more than once so that enough water is collected. **<u>If this is necessary, each vertical must be sampled the same number of times</u>**.
6. **By using a churn or cone splitter, split the composite sample into the appropriate number of sampling bottles.**

NOTE: Inevitably, there will be occasions when the velocity cannot be compensated for and a representative sample cannot be collected. On such occasions, collect as far into the water column a possible, and note that "sampling through the water column was not possible due to..." on the 'sampler's comments' field sheet.

NOTE: When samplers are hand-lined, it is difficult to maintain a consistent transit rate. To be as consistent as possible, establish a count for each overhand pass of the hand-line and always use the same hand-line motion during the entire sampling event.

6.12 Composite Grab

A composite grab is made under several circumstances:

1. **The MSD for the wadeable or bridge sampler is not met.** As was stated before, samplers require a 4-10 inch MSD to work. At certain times of the year, this can't be met at any point across the stream. Occasionally, in sandy-bottomed waters, a space can be dug out to allow the MSD to be met. However, care should be taken not disturb bottom sediments into the sampler.
2. **The waterbody is at minimal flows.** When a stream or river is at minimal flow, attempting to take a depth-integrated sample may disturb bottom sediments, which would potentially bias the sample.
3. **The waterbody is at extreme flows.** When a stream or river is at extreme storm water flow, it may be impossible to lower the sampler through the water column. In these cases, by continuing to use the sampler, a grab sample can be taken. It is a grab sample because it does not sample completely through the vertical profile.
4. **The waterbody is a series of disconnected pools.** In these cases, take a representative grab sample from several of the pools and add to the composite.
5. **Used for Bacteria samples.**

To take a composite grab, follow these steps:

1. **Using EWI, establish verticals through the cross-section (refer to above).**
2. **When wading the waterbody, use a wide-mouthed bottle to collect water at each vertical and add to the composite.**
3. **When sampling from a bridge, use a weighted-bottle sampler to collect at each vertical and add to the composite.**
4. **Note the use of the composite grab technique on the field sheet.**
5. **When collecting bacteria in the stream, invert the bottle before dipping in the current.**

6.13 Point Grab

A point grab is made under several circumstances:

1. **The stream is less than one meter wide.**
2. **The stream is less than three meters wide and is at minimal flow.**
3. **The waterbody is one large pool surrounded by dry streambed.** Take a point grab at the center of the pool.

To take a point grab, follow these steps:

1. **Establish the thalweg (deepest, fastest moving part) or center of the waterbody.**
2. **When wading the waterbody, use a wide-mouthed bottle to collect water at the point and fill the necessary amount of sample bottles.**
3. **When sampling from a bridge, use a weighted-bottle sampler to collect water at the point and fill the necessary amount of sample bottles.**
4. **Note the use of the point grab technique on the field sheet.**

6.14 Near Shore Sampling

When a stream or river is inaccessible by bridge, boat, or wading, it may be necessary to collect a near shore sample. Extended samplers are made and should be used to extend the point of sampling as far away from the shoreline as possible. If an extended sampler is unavailable, every effort should be made to extend as far as possible away from the shore.

To take a point grab, follow these steps:

1. **Use a wide-mouthed bottle to collect water at the point and fill the necessary amount of sample bottles.**
2. **Note the use of the point grab technique on the field sheet.**

6.15 Sampling Pools and Documentation of Dry Sites

Please, adhere to the following general guidance when sampling pooled sites. <u>Written and photo-documentation are required</u>. Dry sites (no visible water) should be documented through the same process.

- If a pool(s) exist, pick a representative pool and take a point grab sample. Do not composite across pools.
- If water is not in view from the normal site(s), do not attempt to find pooled or running water elsewhere. Exceptions to this are sites with alternative sampling locations (e.g., Kiamichi River near Tuskahoma or Canadian River near Taloga). <u>DO NOT STIR UP BOTTOM SEDIMENTS.</u>
- Sample from the bank, and, if needed, use a collection bottle to fill the sample bottle.
- Collect grab samples before *in situ* measurements.
- When collecting *in situ* measurements, it may be necessary to walk out deeper in order to obtain a representative sample. However, be aware that bottom sediments may change certain parameters such as conductivity or pH. To recognize this, always take a measurement from the bank before proceeding into the pool.
- **<u>DOCUMENT DOCUMENT DOCUMENT DOCUMENT</u>**. Describe the size of the pools and how many are visible. Note which pool was sampled if more than one exists. Take numerous pictures to photodocument the described conditions.

6.2 Methods for Splitting Composite Samples

6.21 Splitter Churn

A splitter churn is used to aliquot a composite sample into various bottles for further analytical analysis. It can be made of polyethylene or Teflon and is composed of the following parts—the bucket with a spline used for guiding the churn and a spigot for dispensing the sample, a churn, a hose, and a top cap. The following guidelines must be followed when using a splitter churn:

- The churn should always mate properly to the spline when inserted into the bucket. By doing this, the vertical lip of the churn is centered over the spigot. This orientation allows for the churned composite to be properly dispensed.
- Churning of the sample should always been done at a slow, consistent pace. Churning the sample at a high rate of speed can agitate the sample and bias it.
- The spigot should always be kept free of obstructions so that a continuous even flow exits the bucket into the sample containers.
- A hose should be attached to the spigot when dispensing the sample to avoid cross contamination of the sample.
- Flow through the hose should be maintained in a continuous stream when aliquotting the sample.
- The churn cap should always be in place during both sample collection and dispensing.
- The churn should be kept in a clean plastic bag during both sample collection and dispensing.

6.22 Sample Inversion

Sample inversion is used to aliquot a composite sample into various bottles for further analytical analysis. The method is normally used when a plastic churn cannot be used, a Teflon churn is not available, and for bacteria sample. The method involves collection of the composite sample to a larger collection bottle usually made of glass or a 1-quart plastic bottle for bacteria. The sample is alliquotted by inverting the collection bottle until adequately mixed and dispensing the mixed composite to the sample bottles. The following guidelines must be followed when using the sample inversion method:

- The collection bottle should always be capped between verticals.
- To adequately mix the composite, the collection bottle should be inverted at least ten times.
- To avoid sample aeration or over agitation, the inversions of the bottle should be done at a slow, consistent pace.
- Only one sample bottle should be filled for each inversion cycle. If two bottles are required for a complete sample, the collection bottle should go through an inversion cycle for each bottle.

6.23 Whirl-Paks

Whirl-Paks are used for compositing bacteria samples. Before sample is aliquotted to bacteria sample bottles, ensure that sample is completely mixed.

6.3 Types of Water Quality Samples

6.31 Inorganic Panel

The inorganic panel is processed through an analytical laboratory for a nitrogen series, phosphorus series, and certain minerals. A solids series may also be included in this panel. These samples are almost always split using a splitter churn. Unless otherwise described in a project quality assurance plan, these samples are collected by splitting the composite to two 1-liter polyethylene sample bottles. Completed samples should be void of air. One sample bottle is preserved on ice at 4°C, and the other sample bottle is preserved with concentrated sulfuric acid on ice at 4°C. Because these samples are normally processed for both a nitrogen and phosphorus series, they normally have a 48 hour hold time and must be returned to the laboratory within that holding time. In the event that ortho-phosphorus and ammonia nitrogen ars analyzed using field methods (OWRB, 2010) and nitrate/nitrite are to be analyzed as a single parameter, the holding time for inorganic samples increases to 7 days.

6.32 Field Panel

The field panel is processed by field staff for turbidity, hardness, and alkalinity. These samples are almost always split using a splitter churn. Unless otherwise described in a project quality assurance plan, these samples are collected and processed at the site but may be split by a small polyethylene sample bottle or the 1 liter collection bottle used during sampling. Completed samples should be void of air. If samples are collected for processing at a later time, the sample is preserved on ice at 4°C. Because alkalinity and turbidity have a 24-hour hold time, samples must be processed within a 24 hour time period and must be brought to ambient temperature before analyzed. Processing of these samples is described in other SOP's (OWRB, 2005a; OWRB, 2005b).

Additionally, if ortho-phosphorus and ammonia nitrogen are analyzed using Hach® field methods (OWRB, 2010), special care should be taken in processing and/or preserving these samples. Preferably, these parameters should be processed while at the site, but they can be held for up to 24 hours on ice. Processing of these samples is described in other SOP's, which are referenced in Section 9.0 of this document.

6.33 Metals Panel

The metals panel can include both dissolved and total recoverable metals or one or the other depending on project needs. Samples are processed through an analytical laboratory for metals included in the Oklahoma Water Quality Standards (OWQS), Appendix G, Table 2 (OWRB, 2007) and various other metals depending on the project. In the past, these samples were always collected by the specific methodology used to collect the inorganics panel. However, because of concerns over cross-contamination and the OWRB is now filtering dissolved metals samples in the field, metals samples will from this point forward (3/1/2010) be collected using only the point grab methodology (USEPA, 2000).

Total recoverable metals will be collected by grab sampling technique, using a wide-mouth , screw-cap 250-ml polyethylene collection bottle(provided by ODEQ). When sampling for dissolved metals, a grab sample will be collected using a wide-mouth, screw-cap125-mL polyethylene collection bottle (preferably, the SC490 digestion cup from Environmental Express®). To avoid contamination, samplers should follow the clean hands/dirty hands methodology (CH/DH) described in Section 2.2 of USEPA methodology (USEPA, 2000). Because the OWRB will not be using peristaltic pumps during collection, the specific tasks related to pumps and tubing are not applicable. However, all tasks related to collection, handling, and filtration of the sample should be followed.

After the sample is collected, post-processing steps differ for total recoverable and dissolved samples. Total recoverable samples will be immediately preserved with 20 drops concentrated nitric acid (HNO_3) upon returning to the vehicle using CH/DH. To ensure sample efficacy, dissolved samples will be filtered within 6 hours of collection. Preferably, sample filtration should occur on site, but if environmental conditions could increase the possibility of cross-contamination (e.g., heavy rain, wind, etc.), the sample may be placed on ice and filtered at a different location within the 6-hour holding time.

When filtering samples, all applicable CH/DH tasks should be employed to minimize contamination of the sample. The OWRB uses several types of filtration devices. Regardless of the filtration method used, 30 to 45-mL of filtrate should be collected for processing by the laboratory. The primary filtration device is the FlipMate 100, which has a 0.45 micron polyethersulfone filter preceded by 2.0 micron glass fiber pre-filter. The FlipMate accepts two threaded SC490 digestion cups—one containing the sample to be filtered and an empty cup to receive the filtrate. The assembly is then "flipped" over, and a vacuum is applied using a hand held or electric pump. The sample is pulled through the filter into the empty cup, and the filtrate is preserved with 4 drops of concentrated HNO_3, capped, and stored for later analysis. A secondary filtration device is the FilterMate™, which has a 0.45 micron Teflon ® filter. The plunge-type filter fits directly into a 50-mL digestion vessel filled with sample water. Using tortuous path methodology, the filter is pushed through the sample to the bottom of the cup with a detachable plunger. After filtration is complete, the plunger is removed and discarded while the filter assembly remains in the bottom of the cup. The filtrate is then poured off into an SC490 digestion cup, preserved, and stored for subsequent analysis. A 2.0 micron Teflon ® FilterMate™ pre-filter can be used for turbid samples, but because an additional handling step is added to the filtration process, the separate pre-filter should be used sparingly.

Several quality assurance steps are employed for metals sampling over and above the traditional blank and replicate samples. Currently, collection cups and filters are not pre-certified to sub-part per billion levels. Based on hardness-dependent metals criteria in the OWQS (OWRB, 2007), the OWRB and our analytical laboratory (ODEQ-SEL) have developed two sets of reporting limits depending on average hardness (Table 3). For sites with average hardness > 150 ppm, a 1.0 ppb reporting limit is used for all metals in the OWQS, except mercury (0.500 ppb). For sites with average hardness < 150 ppm, sub-part per billion reporting limits have been developed for cadmium, copper, lead, and silver so that reporting limits are not 1.5 times greater than calculated criteria. To ensure that samples are collected in vessels and processed through media that do not artificially raise sample concentrations, the OWRB and ODEQ-SEL pre-certify production batches of filters and digestion cups received from Environmental Express® or other future vendors. Additionally, for sample sites with average or anticipated hardness values < 30 ppm, filters and digestion cups are pre-conditioned with concentrated nitric acid or pre-certified laboratory grade deionized water before use.

6.34 Organics Panel

The organics panel is processed through an analytical laboratory for various organics listed in the OWQS. Depending on the organics of interest, the analytical methods may include 515.3, 608, 614, 8260, and 8270. These samples are almost always split using the bottle inversion method. Unless otherwise described in a project quality assurance plan, these samples are collected by splitting the composite from a 1-gallon glass jar to various quart or pint glass containers. Completed samples should be void of air. Bottles are preserved on ice at 4^oC.

6.35 Bacteria Panel

The Bacteria panel is processed through an analytical laboratory for various bacteria listed in the OWQS. These samples are almost always split using the bottle (1-quart plastic) inversion method. Samples are always collected using the composite grab method. Bottles are placed in doubled ziplock Baggies and are preserved on ice at 4^{o}C. Samples should not go below 4^{o}C or above 10 ^{o}C.

Table 3. ODEQ-SEL reporting limits and criteria for metals listed in OWQS, Appendix G, Table 2. (OWRB, 2007)

Metal	Reporting Limit	Low Hardness Reporting Limit	5ppm Hardness-Dependent Criteria	10ppm Hardness-Dependent Criteria
Arsenic	1.000	1.000	40.000	40.000
Barium	1.000	1.000	1000.000	1000.000
Cadmium*	1.000	0.180	0.115	0.194
Chromium	1.000	1.000	50.000	50.000
Copper*	1.000	0.080	0.950	1.717
Lead*	1.000	0.120	0.086	0.191
Mercury	0.500	0.500	0.500	0.500
Nickel*	1.000	1.000	12.479	22.432
Silver*	1.000	0.040	0.020	0.066
Selenium	1.000	1.000	5.000	5.000
Thallium	1.000	1.000	1.700	1.700
Zinc*	1.000	1.000	8.189	14.734

* = Criteria based on a dissolved conversion factor

7.0 Forms

7.1 Field Notes

Field notes are documents used to annotate and record information that is gathered at the project site. They are a data sheet and should be treated as such. Therefore, they should be written, legible, and complete. To avoid confusion and loss of data, a new sheet should be used at each new project site. Field notes should be initialed and dated by the collecting personnel and data entry personnel. For guidance on proper procedure to complete the field notes, refer to your supervisor and or FTE. Field notes can be found at S:\Monitoring\STREAMS\forms\Field Notes.doc.

7.2 Laboratory Log-in Sheets

Login sheets are documents turned into the analytical laboratory for each sample collected. These forms are used to denote the parameters that should be analyzed. They are a data sheet and should be treated as such. Therefore, they should include the date and time of sample collection and be legible and complete. To avoid confusion and loss of data, a new sheet should be used at each new project site. For guidance on proper procedure to complete the login sheets, refer to your supervisor and or FTE. Login sheets can be found at S:\Monitoring\STREAMS\forms\.

7.3 Chains of Custody

Chains of custody are documents turned into the analytical laboratory for each group of samples collected. These forms are used for several purposes. They act as a legal document to show proper delivery of samples occurred and they make a general list of the parameters that should be analyzed. Chains of custody are available for inorganic, metals, and organics panels. They are a data sheet and should be treated as such. Therefore, they should include the date and time for each sample collected and be legible and complete. They should also be signed and dated by field and laboratory receiving personnel at the time of delivery. To avoid confusion and loss of data, a new chain of custody should be used for each group of samples. For guidance on proper procedure to complete the chains of custody, refer to your supervisor and or FTE. Chains of custody can be found at S:\Monitoring\STREAMS\forms\.

8.0 Data Storage

All completed paper copies of forms and data sheets should be maintained with the appropriate station notebook. The data from the field notes and laboratory data sheets should be either entered into or uploaded to the Water Quality Database. Each sample should be maintained electronically in the database under a unique sample number.

9.0 References

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