New Mexico Environment Department
Surface Water Quality Bureau

Standard Operating Procedure

for

# NPDES WASTEWATER SAMPLING

Approval Signatures

Shelly Lemon
Subject Matter Expert — Date: 8-8-2014

Jodey Kougioulis
Quality Assurance Officer — Date: 8/8/14

Bruce Yurdin
Program Manager — Date: 8/8/2014

## 1.0 Purpose and Scope

The purpose of this procedure is to document both general and specific procedures, methods and considerations to be used and observed when collecting wastewater samples for field screening or laboratory analysis during NPDES Compliance Sampling Inspections.

For procedures describing ambient monitoring, see SOP 8.2.

## 2.0 Personnel Responsibilities

Field personnel who collect wastewater samples for NPDES Compliance Sampling Inspection activities or who supervise those who do are responsible for implementing this procedure. On the occasion that SWQB field personnel determine that any of the procedures described in this SOP are inappropriate, inadequate or impractical and that an alternative field procedure must be used to obtain a wastewater sample, the variant procedure will be approved by the Program Manager, if practical, and documented in the field log book along with the circumstances requiring its use.

## 3.0 Background and Precautions

**Background**
Prior to mobilizing, the sampler must decide what kind of samples to collect, what analytes are to be analyzed, and where to collect the samples. An NPDES permit should specify this information along with the type of sample to collect: grab sample, time composite or flow-proportional composite. *The inspector must always use the method(s) required by the permit.*

The sampler must take care to ensure that the sampling location is correct with respect to the discharge location and NPDES permit requirements (if applicable) and that the samples are representative (e.g., flow is turbulent and well-mixed). Wastewater samples collected by the PSRS in conjunction with an NPDES permit compliance sampling inspection are collected from the facility outfall sampling location if practical and appropriate. The sampling location(s) must be in accordance with NPDES permit requirements and

downstream of all treatment units. If pre-existing stations are not available, then the Lead Inspector will select stations from a location that is representative of the wastewater discharge quality. An accessible station will be selected far enough upstream from the discharge point to eliminate any possibility of influence from the discharge. A downstream station will be selected at a point where the effluent is completely mixed in the receiving water. This point can be determined by checking the specific conductance of a transect taken completely across the receiving water. When the readings are consistent (±10 µmhos/cm), the effluent is considered to be completely mixed for sampling purposes. Overall, the Lead Inspector should evaluate sampling locations to ensure their usefulness in determining compliance with the water quality standards.

**Table 1** provides a general summary of the parameters commonly sampled during NPDES compliance sampling inspections. Sampling frequency is once per compliance sampling inspection event and the number of samples taken is one for all parameters except when noted or as required by the NPDES permit. Any deviations from the generalized sampling plan set forth in **Table 1** is documented as part of the NPDES permit compliance sampling inspection plan and report. The sampling process design for NPDES compliance monitoring for *enforcement purposes* will be developed on a case by case basis to determine sampling locations, frequency of data collection, and parameters to be sampled.

**Safety Precautions**
Proper safety precautions must be observed when collecting wastewater samples. Refer to the SWQB Job Hazard Analysis (JHA) and any pertinent site-specific Health and Safety Plans for guidelines on safety precautions. Consult the SWQB Chemical Hygiene Plan (CHP) and/or the SWQB Lab Safety Officer regarding acceptable practices for transferring concentrated acids from the Runnels Building Laboratory. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant safety requirements such as eye protection, hard hats, and steel-toed boots, as appropriate.

**Procedural Precautions**
The following precautions should be considered when collecting wastewater samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample.
- Collected samples are in the custody of the Lead Inspector or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the Lead Inspector and stored in a secure place.

**Special Precautions for Wastewater Sampling**

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised. It may be easiest to put on two pairs of gloves initially so that if the outer glove is compromised it can be simply removed and replaced.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.
- Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly contaminated media

must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background/control samples.

- If possible, one member of the field sampling team should take all the notes and photographs, fill out labels/tags, etc., while the other members collect the samples.
- Field investigators must use new, verified certified-clean disposable or non- disposable equipment cleaned according to procedures contained in SWQB Standard Operating Procedure for Equipment Cleaning Procedures (SOP 8.1).

**Table 1. Parameters Commonly Sampled during NPDES Compliance Sampling Inspections**

| *Analytical Suite* | *Parameters* | *Notes, if applicable* |
|---|---|---|
| Field Parameters | pH | |
| | Temperature | |
| | Specific Conductance | |
| | Dissolved Oxygen (DO) | |
| | Turbidity | |
| | Flow (Discharge) | Inspection team evaluates permittee's flow-measuring equipment and uses the flow obtained if the equipment is found to be acceptable |
| Metals | Dissolved Metals | List of metals analyzed is determined on a permit-specific basis |
| | Total Metals | List of metals analyzed is determined on a permit-specific basis |
| Anions and Cations | Alkalinity | |
| | Bicarbonate | |
| | Calcium | |
| | Carbonate | |
| | Chloride | |
| | Fluoride | |
| | Hardness | Must be collected concurrently with metals for which the WQS criterion is "hardness dependent" |
| | Magnesium | |
| | Potassium | |
| | Sodium | |
| | Sulfate | |
| | Total Dissolved Solids | |
| | Total Suspended Solids | |
| Nutrients | Ammonia | |
| | Nitrate plus Nitrite | |
| | Phosphorus, Total | Orthophosphate is analyzed only when specifically requested |
| | Total Kjeldahl Nitrogen | |
| | Total Organic Carbon | Sampled only if required in NPDES permit |
| | Chlorophyll a | Sampled only if required in NPDES permit |
| Bacteria | Fecal Coliform | Duplicate samples required |
| | Escherichia coli | |
| Other Parameters | Cyanide | Sampled only if required in NPDES permit; WQS criterion is for dissolved and weak acid dissociable cyanide |
| | Biochemical Oxygen Demand (5-day) | |
| | Chemical Oxygen Demand | Sampled only if required in NPDES permit |
| | PCBs | Sampled only if required in NPDES permit |
| | Total Residual Chlorine | Sampled only if required in NPDES permit |

**Wastewater Sample Handling and Preservation Requirements**

- During Compliance Sampling Inspections for NPDES compliance determination and/or enforcement activities, all sample collection and preservation procedures will comply with those listed below and approved under 40 CFR Part 136, specified in the permit, or approved by the Regional Administrator, as appropriate.

  - "Guidelines establishing test procedures for the analysis of pollutants under the Clean Water Act; analysis and sampling procedures" 40 CFR Part 136 or any test procedure approved or accepted by EPA using procedures provided in 40 CFR Parts 136.3(d), 136.4 and 136.5, Most Recent Version; and
  - State of New Mexico Standards for Interstate and Intrastate Surface Waters (20.6.4 NMAC), Most Current Amendments.

- Wastewater samples will typically be collected either by directly filling the sample container or by using an automatic sampler or other device.
- During sample collection, if transferring the sample from a collection device, make sure that the device does not come in contact with the sample containers.
- Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 6.1.8.2 Volatile Organic Compounds). For all other samples, field personnel should allow for some vacant space in the sample container.
- All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. Samples requiring filtration must be filtered within 15 minutes of collection.
- All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.
- Samples collected for compliance monitoring or enforcement purposes shall be custody sealed during storage and/or shipment to prevent tampering or contamination. Samples taken during CSIs shall follow chain of custody procedures provided by the contracted analytical laboratory.

## 4.0 Definitions

***Sample*** *means the medium, the sample bottle and preservative or growth reagents. It includes those materials that cannot be readily separated from the medium that is sampled.*

***Sampling equipment*** *is anything the medium contacts before becoming a sample.*

***Grab sample*** *is an individual sample collected over a period of time not to exceed 15 minutes and is representative of conditions at the time the sample is collected.*

***Composite sample*** *is collected over time, either by continuous sampling or by mixing discrete samples, and represents the average characteristics of the stream during the compositing period.*

***24-hour composite sample*** *consists of a minimum of 12 effluent portions collected at equal time intervals over the 24-hour period and combined proportional to flow or a sample collected at frequent intervals proportional to flow over the 24-hour period.*

***12-hour composite sample*** *consists of 12 effluent portions collected no closer together than one hour and composited according to flow. The daily sampling intervals shall include the highest flow periods.*

***6-hour composite sample*** *consists of six effluent portions collected no closer together than one hour (with the first portion collected no earlier than 10:00 a.m.) and composited according to flow.*

***3-hour composite sample*** *consists of three effluent portions collected no closer together than one hour (with the first portion collected no earlier than 10:00 a.m.) and composited according to flow.*

## 5.0 Equipment and Tools

Select sampling equipment based on the parameters of interest, the specific equipment use and the available equipment. Clean all sampling equipment and obtain clean sample containers using the appropriate protocols specified in SOP 8.1**.**

a. MANUAL SAMPLING: Manual sampling is used for collecting grab samples and/or for immediate in-situ field analyses. However, it can be used in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to evaluate unusual conditions.

b. AUTOMATIC SAMPLERS: A wide variety of automatic samplers are commercially available. For unattended surface water monitoring, the SWQB uses a Teledyne ISCO 3700 portable automated sampler, often referred to simply as “an ISCO,” or “the sampler,” programmed to collect samples at preset intervals, and ideally linked to a model 4230 flow meter, “the bubbler.” Field staff should confirm that the bubbler is functional and properly linked to the sampler. If equipped, flow data are stored electronically on the flow meter RAM and are available for retrieval through a model 581 rapid transfer device (RTD).

See **Appendix A** for a list of equipment that may be necessary for wastewater sampling.

## 6.0 Wastewater Sample Collection and Processing

There are two basic types of sampling techniques: grab and composite. The NPDES permit specifies the appropriate sample type. *The sample type collected must be in accordance with NPDES permit requirements and/or a project-specific sampling plan*. A complete description of all NPDES sampling procedures and techniques is presented in the *NPDES Compliance Inspection Manual* (USEPA 2004).

Use a waterproof, permanent marker to label sample containers with the date/time (if composite sample then date/time of first and last aliquot), sample site description, analysis required (i.e., parameter to be tested), collector’s name, and preservative, if used. Place a pre-printed SLD reference ID (RID) sticker on the lid of each sample container. Write the RID number on the container as well. Use self-adhering labels for organic bottles. Label all containers before sampling to avoid having to write on wet surfaces.

In most cases, the sample container should never be rinsed with the sample prior to collection. Pre-rinsing may allow some contaminants to stick to the sides of the container or settle to the bottom prior to the final sample collection. For instance, pre-rinsing with an influent sample may cause "extra" oil and grease to stick to the sides or "extra" settleable solids to remain in the bottom of the bottle. This would cause a high bias to the sample answer. Samples collected for bacteria (*E.* coli, total or fecal coliforms) should never be rinsed.

For composite samples, limit compositing period to 24 hours and start the hold time limits when the last aliquot is collected. See **Appendix B** for a summary of the container types, sample preservatives, and holding times for various analyses.

### *6.1 Grab Samples*

Grab samples are either single, discreet samples or individual samples collected over a period of time not to exceed 15 minutes. Grab samples provide information about pollutant concentrations only at the moment they are collected; therefore they should be representative of the wastewater conditions at the time of sample collection. Grab samples are usually taken manually, but can be collected using an automatic sampler. Grab samples for oil and grease or volatile organics should be collected manually, not by an automatic sampler. Oil and grease may adhere to automatic sampler tubing while volatile organics in an automatic sampler will volatilize.

Grab samples are collected by filling the sample containers by holding them just beneath the surface of the waste stream, or under the flow at an outfall pipe. The mouth of the container should be faced into the

current while keeping the hands, sampler and any other equipment downstream to minimize the chance of contamination. Where it is impractical or unsafe to sample by hand, a sampling rod or bucket and rope can be used. Sampling rods are polycarbonate or stainless steel poles with a large clamp or cage on one end designed to securely hold various sizes of sample container. Containers are placed in the cage while sampling to provide extra reach or to prevent the hands from contaminating the sample or contacting wastes. The container should be gently but quickly lowered into the water to minimize the contribution of surface films to the sample. When using an intermediate container or when sampling with a bucket and rope, a sub-sample is immediately poured off using the techniques described below for sampling directly from the waste stream.

**The following procedures are for grab samples:**

*6.1.1 Anions, Alkalinity, Total Suspended Solids (TSS) and Total Dissolved Solids (TDS)*

Fill a 1-quart cubitainer with wastewater sample and keep sample on ice, at 6°C or less. A 500 mL high density polyethylene (HDPE) bottle can be used if only TSS and/or TDS samples are needed.

*6.1.2 Nutrients*

Fill a 1-quart cubitainer with wastewater sample and acidify with approximately 2 mL concentrated sulfuric acid or enough to adjust the pH to < 2. Samples may also be collected in 500 mL HDPE bottles and acidified with 1 mL concentrated sulfuric acid. Keep samples on ice, at 6°C or less.

*6.1.3 Total Metals*

Fill a 1-quart cubitainer with wastewater sample and acidify with 5 mL of concentrated nitric acid. Use two, 2.5 mL aliquots of acid to minimize exposure of metal pipette parts to the acid. Keep samples at ambient temperature.

6.1.4 *Total Recoverable Aluminum*

If turbidity is 30 NTUs or less, follow the instructions for total metals samples (6.1.3). If turbidity is greater than 30 NTUs, follow the instructions for dissolved metals samples (6.1.5), but use a 10-μm filter in place of the 0.45-μm filter. If there are equipment problems prohibiting the measurement of turbidity in the field and the wastewater sample has any cloudiness as determined by visual inspection, then the total recoverable Al sample should be filtered using a 10-μm filter. Acidify with two, 2.5 mL aliquots of concentrated nitric acid. Keep samples at ambient temperature.

6.1.5 *Hardness/Dissolved Metals*

Fill a 1-quart or 1-gallon cubitainer with wastewater sample. Set up the Geo Pump by connecting a power cord to a cigarette lighter, a 110 VAC outlet or a 12 V battery and clamping a piece of acid-washed, 0.19 inch internal diameter silicone tubing in to the pump head. Avoid touching the ends of the tubing even with gloved hands and do not let the ends of the tubing contact metal. Attach a 0.45-μm filter to one end of the tubing by pulling the plastic bag containing the filter over the barbed fittings until they rupture the bag and pushing the tubing onto the fitting on the upstream end of the filter. Insert the opposite end of the tubing into the cubitainer containing the wastewater sample. Pump approximately 50 mL of sample water through the filter and discard. Pump the remaining sample water through the filter and into a second, new, 1-quart cubitainer. Keep the filter to the side of the neck of the receiving cubitainer to prevent dirt and dust from falling into the sample. Reduce the pumping rate when sample is turbid. Acidify with two, 2.5 mL aliquots of concentrated nitric acid. Keep samples at ambient temperature.

*6.1.5 Radionuclides*

Fill two, 1-gallon cubitainers with wastewater sample. Preserve samples with sufficient nitric acid to adjust the pH to below 2. Typically, 7-10 mL per gallon is adequate. Check pH with pH paper on an aliquot of the acidified sample if unsure. If pH paper is not available, use at least 10 mL of nitric acid to preserve. Keep samples at ambient temperature.

*6.1.6 Cyanide*

Fill a 1-quart cubitainer with wastewater sample. Evaluate the sample water for the presence of chlorine. If chlorine is suspected to be present, samples can be checked using potassium iodide (KI)-starch test paper. A blue color indicates the presence of chlorine. If chlorine is present, it must be removed using ascorbic acid. To do this, add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the starch test paper then add one additional crystal. After adding ascorbic acid (if necessary), preserve samples for cyanide analysis with sodium hydroxide (NaOH). Determine the pH of the sample on an aliquot (after adding ascorbic acid) using pH test paper. Preserve the sample with sufficient NaOH to produce a pH of 12 or greater. Most samples will require about 0.5 g solid NaOH per quart, or approximately five pellets. Handle the pellets with wooden or plastic utensils, or pour directly from the container. Keep samples on ice, at 6°C or less.

*6.1.7 Biochemical Oxygen Demand (BOD)*

Samples for BOD analysis may degrade significantly during storage between collection and analysis, resulting in low BOD values.

If grab samples are required by the NPDES permit, samples analyzed immediately (less than 15 minutes of collection) do not need to be refrigerated. If analysis is not started within 15 minutes of sample collection, keep sample in the dark at or below 6°C from time of collection. Begin analysis within 6 hours of collection; when this is not possible because the sampling site is distant from the laboratory, store at or below 6°C and report length and temperature of storage with results. In no case should BOD analysis start more than 24 hours after sample collection. When samples are to be used for regulatory purposes, every effort should be made to deliver samples to lab within 6 hours of collection.

*6.1.8 Organic Chemicals (GENERAL)*

Facility Inspectors are responsible for obtaining organics sample containers and VOC preservative (10% HCl) from the SLD Organics Section (505-383-9031) and should contact SLD at least 1 week prior to sample collection to ensure extractions and analyses can be carried out within holding times. When collecting samples for organic compounds, wear nitrile gloves. Gloves are worn to prevent organic compounds that may be present on skin from contaminating samples. Avoid the false sense of security gloves may give; a gloved hand can still spread contamination. Put gloves on as close to the time of sample collection as possible. If glove cleanliness is compromised, they should be replaced. It may be easiest to put on two pairs of gloves initially so that if the outer glove is compromised it can be simply removed and replaced.

Use sampling equipment with fluorocarbon polymer, glass, or metal components if components will directly contact samples to be analyzed for organic compounds. Do not use plastics other than fluorocarbon polymers. Organic compounds tend to concentrate on the surface of sampling devices or containers, therefore do not rinse the sampling device and sample container with sample water before collecting. Store and transport all organic samples on ice at 6°C or less.

Free chlorine will oxidize organic compounds in the water sample even after it is collected. If residual chlorine is suspected, measure the residual chlorine using a separate water subsample (see *Section 6.1.9* below). If residual chlorine is above a detectable level (i.e., the pink color is observed upon adding the reagents), immediately add 100 milligrams (mg) of sodium thiosulfate to the pesticides, herbicides, semivolatiles, and volatile organic samples; invert until the sodium thiosulfate is dissolved. Record the residual chlorine concentration in the field book. If residual chlorine is below detectable levels, no additional sample treatment is necessary.

*6.1.8.1 Semivolatile Organics Compounds (SVOCs), Herbicides, Pesticides*

For each sample planned, obtain two 1-L glass, amber colored bottles with Teflon lined caps and bubble-wrap sleeves from the SLD Organics Section. Fill both bottles by submersion and recap underwater if possible. If the effluent stream is not deep enough to submerse the amber bottles, use a third 1-L glass amber bottle or other glass container to fill the two sample bottles. A headspace is

acceptable with SVOC samples. Keep jars in bubble-wrap sleeves before and after filling to avoid breakage. Store samples in the dark and on ice, at 6°C or less.

*6.1.8.2 Volatile Organic Compounds (VOCs)*

For each sample planned, obtain two 40-mL vials in a whirl pack from the SLD Organics Section. Fill the vials by submersion. Add 5 drops of 10% HCl (also obtained from the SLD Organics Section and less than 1 month old) to each vial. Fill the caps with sample water and cap the vials without leaving a headspace – you should not see any air bubbles in the sample container. Keep the vials together in a closed Whirl-Pack, on ice, at 6°C or less. Store VOC preservative (10% HCl) at 20°C and use within 1 month.

*6.1.9 Total Residual Chlorine (TRC)*

Fill the 10-mL sample cell bottles provided in the HACH Chlorine Pocket Colorimeter kit to the line either directly from effluent stream or from an aliquot of sample water. Wipe off excess water from the outside of sample bottles with lens paper or a KimWipe and ensure that the glass is dry and clean (no fingerprints or smudges). Add the contents of one DPD Total Chlorine Powder Pillow to one 10-mL sample cell and gently shake for 20 seconds. Let it stand for 3 to 6 minutes before reading. During this period, place the other 10-mL cell bottle (the blank) into the colorimeter cell holder, with the diamond mark facing you. Tightly cover the cell with the instrument cap and press zero. Remove the blank sample cell bottle from colorimeter cell holder. Place the cell bottle with reagent into the colorimeter cell holder, again with the diamond mark facing you, and cover tightly with instrument cap. Between 3 and 6 minutes after adding the reagent (Total Chlorine Powder Pillow) to the sample press READ and record the total residual chlorine value (in mg/L) in the comment section of field sheet.

*6.1.10 Bacteria (E. coli and Total Coliforms)*

Wear disposable gloves. Collect samples in sterile bottles. Do not rinse sample bottle before collection and do not remove the caps or shrink wrap until immediately before sampling. When practical, collect the sample directly into the sample bottle. Where this is not practical (such as when the waste stream is not within reaching distance) an intermediate container and/or sampling rod (see Section 6.1) may be used. If it is necessary to transfer the sample water from an intermediate container to the sample bottle, note this in the field log book and/or field form and also when entering the sampling event into SWQB's database (SQUID).

Verify that the bottle is properly labeled and remove and dispose of the shrink band or sealing tape. Uncap the sample bottle. Place the lid over the mouth leaving a small opening on one side. Place one hand around the bottle with a finger holding the lid in place. Quickly submerge the bottle (mouth facing up) through the surface layer with the top of the bottle tilted forward and the opening facing upstream. Keep the bottle submerged long enough for the bottle to fill up to the 100-mL line near the shoulder of the bottle. Alternatively, the sample bottle (or intermediate container) can be attached to a sampling rod to collect the sample (see Section 6.1).

Try to avoid overfilling the bottle. If the bottle is filled above the 100-mL line, immediately decant excess sample water. Each IDEXX bottle has enough sodium thiosulfate to neutralize 10 mg/L chlorine (IDEXX 2004), so decanting a small amount should not significantly reduce the amount of sodium thiosulfate needed for neutralization in most situations. However, if chlorine concentration is known to be high (i.e., ≥10 mg/L) then an intermediate container (e.g., Whirl-Pak® bag) and/or sampling rod should be used to collect the sample to avoid overfilling and ensure proper neutralization.

To collect the sample in a Whirl-Pak® bag, tear off the top of the bag along the perforation above the wire tab just prior to sampling. Avoid touching the inside of the bag. If you accidentally touch the inside then use another bag. Hold the two white pull tabs in each hand and lower the bag into the waste stream with the opening facing upstream. Open the bag under the surface of the water by pulling the white pull tabs apart. The bag should fill with sample water. Fill the bag no more than ¾ full. Lift the bag out of the water and pour out any excess water. Transfer 100 mL of the sample water to the labeled sample bottle containing sodium thiosulfate.

After collecting the sample, carefully replace the lid and store sample on ice or refrigerate it at a temperature less than 10°C until ready to process. Use insulated containers to assure proper maintenance of storage temperature. Ensure that sample vessels are not totally immersed in water during transit.

*Refer to SOP 9.1 for in-house IDEXX processing and analysis of bacteria samples.*

**Samples for Submittal to SLD**

Use 125-mL, sterile polypropylene sample bottles containing sodium thiosulfate (available from SLD). *SLD will not accept samples in IDEXX bottles.* Use the same procedures described above to collect a wastewater sample for submittal to SLD. Contact SLD in advance of delivery of the samples to verify that the samples can be processed within the required time. Note the temperature of the samples when they are submitted to SLD receiving. Fill out one submittal form for each sample and apply "Do Not Tamper" seals to bottles before submitting. Maximum transport time is 6 hours from time of collection until time of delivery at SLD, and 8 hours from collection to the start of incubation.

#### *6.1.11 Supplementary Data Collection*

While conducting wastewater sampling, the following information will also be obtained (if applicable):

- Flows associated with the samples collected – continuous and instantaneous flows with composite samples and instantaneous flows with grab samples.
- Diagrams and/or written descriptions of the wastewater treatment systems (if available).
- Photographs of pertinent wastewater associated equipment, such as flow measuring devices, treatment units, etc.
- Completion of applicable forms required during specific investigations.

All observations, measurements, diagrams, etc., will be entered in bound field logbooks.

### *6.2 Composite Samples*

Composite samples are collected over time, either by continuous sampling or by mixing discreet samples. They reflect the average characteristics of the wastewater during the sampling period. If composite samples are taken, keep samples at or below 6°C during compositing (See Section 6.2.3). Limit compositing period to 24 hours. Use the same criteria as grab samples for processing, handling, and storage (Sections 6.1.1 – 6.1.8), starting the hold time at the end of collection period (i.e., after the last aliquot is collected).

Various methods for compositing samples are available and are based on either time or flow proportioning. The compositing methods are described below (USEPA 2004):

- Time Composite Sample: composed of discreet sample aliquots collected in one container at constant time intervals. This method provides representative samples when the flow of the sampled stream is constant (±10 percent of the average flow rate).
- Flow-Proportional Composite Sample: there are two methods used to collect this type of sample. One method collects a constant sample volume per stream flow (e.g. 200 mL sample collected for every 5000 gallons of stream flow) at time intervals proportional to stream flow. This method provides representative samples when the flow is measured accurately. In the other method, the sample is collected by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots.
- Sequential Composite Sample: composed of discreet samples composited in individual containers at constant time intervals or constant discharge increments. For example, samples collected every 15 minutes are composited each hour. The 24-hour composite is made up from the individual 1-hour composites. A variation of this method is to collect a constant volume of sample taken at constant discharge increments.
- Continuous Composite Sample: collected continuously from the sample stream. The sample may be a constant volume which is similar to the time composite, or the volume may vary in proportion to the flow rate of the stream, in which case the sample is similar to the flow-proportioned composite.

For wastewater samples, if the flow rate does not vary by more than ± 10 percent of the average flow rate, it is assumed that a time-intervaled composite will provide a representative measurement of the wastewater characteristics and load discharged over the sampling period (USEPA 2004).

Composite samples may be collected using the permittee's equipment or with SWQB's ISCO 3700 composite sampler. If collected with the permittee's equipment, the Lead Inspector will coordinate with the permittee to collect the composite sample (refer to Sections 6.2.3 – 6.2.4, as needed). If collected using SWQB's equipment, refer to Sections 6.2.1 – 6.2.4 for setting up and programming the ISCO sampler, collecting the sample, and manually flow-proportioning the sample.

*6.2.1 ISCO 3700 Composite Sampler*

For unattended sampling, the SWQB uses a Teledyne ISCO 3700 portable automated sampler. The 3700 Sampler is principally designed to collect sequential (discrete) samples; however, you can program it to collect a number of other types of samples. The extended programming mode is used for more complex sampling routines.

In general, when using an automatic sampler, the sampler should collect samples where the water is well mixed. Typically, a good location is near the center of the flow channel at 40% to 60% of the depth of the channel. The volume of sample obtained should be sufficient to perform all required analyses plus any additional amount for quality control. Individual portions of a composite sample should be at least 100 milliliters in order to minimize sampler solids bias. For automatic samplers which use a peristaltic pump, the sampler must obtain adequate flow rates in the tubing to effectively transport the suspended solids. To avoid solids bias, the velocity of the wastewater in sample tubing should be at least 2 feet per second (fps) and the tubing diameter should be at least ¼ inch Internal Diameter.

*6.2.2 Programming*

*General ISCO 3700 Set-up*

1. The ISCO 3700 carousel can accommodate up to 24 bottles or disposable bags (polypropylene, polyethylene, and/or glass depending on analyte).
2. Power the sampler from a 12v deep cycle battery or other power source. Then connect the ISCO to the flow meter, if using, with the 6-pin cable provided by the manufacturer.
3. Install the ISCO sampler, flow meter, and battery on relatively level ground above the flood plain, if applicable, and within 26 vertical feet of the channel (the maximum lift of the ISCO pump at optimal conditions). Often large tool/storage boxes are used for equipment protection in the field.
4. Attach a length of 3/8-inch diameter Teflon suction line to the ISCO intake and anchor the other end to the channel bottom. The proper placement of the suction line assures the collection of representative samples. Slope the line from the sampler to the sample point in a continuous downhill fashion allowing water to drain between sample intervals. Orient the line intake facing upstream and anchor it to the channel bottom in the main flow of the stream, not in an eddy or edge of the flow. Intake at the channel bottom may result in excess heavy solids, or bed load. Professional judgment determines the intake height above the channel bottom. For example, wide, low-flowing channels may require locating the intake within 2 inches above the bottom, while a narrow, high-flowing channel may allow placement of the intake higher in the water column.
5. (Optional) Attach a length of ¼-inch diameter vinyl tubing to the flow meter, at the barbed fitting below the cable connectors, and anchor the end of the hose to the channel bottom near the ISCO sampler intake. Professional judgment is required in locating the ISCO tube ends at a secure and appropriate location in the sample stream.
6. Program the ISCO sampler first (i.e., ISCO configure sequence and ISCO program sequence), place it in standby, and then program the flow meter, if applicable.
    a. For a time composite sample, program the sampler to collect a minimum of 100 mL for each sample interval. Adjust the volume collected according to the duration of the sampling event, the sampling interval, and the size of the container.

b. For a flow-proportional composite sample, program the sampler to collect a minimum of 100 mL for each sample interval, with the interval predetermined based on the flow of the sample stream.

Chapter 4 of the *Teledyne-ISCO, Inc. Model 3700 Portable Sampler Instruction Manual* has a detailed description of each step in programming the ISCO (ISCO 2013). Alternatively, Section 2.3 "Programming Procedures" of the *Teledyne-ISCO, Inc. 3700 Portable Sampler Pocket Guide* offers an abbreviated version (ISCO 2004). The sampler's programming process is self-prompting. Prompts displayed on the LCD step you through the programming sequence in a logical order, indicating the needed value or option. Once in a step, the arrow keys allow you to scroll between choices, while the Enter/Program key selects your choice. The sampler accepts only appropriate values for the program settings and rejects any unacceptable values. If you turn off the unit or the power is disconnected, the lithium battery retains settings in the sampler's memory.

After programming the automatic sampler, installing the equipment in the field, recovering samples, or reloading bottles for a future sampling, press the start sampling key. SAMPLER INHIBITED will then be displayed on the screen. This display is vital in determining whether the ISCO has been properly programmed.

Remember, an NPDES permit may specify which composite sample type to use, either time composite or flow-proportional composite. *The inspector must always use the method required by the permit.*

*6.2.3 Composite Sample Collection*

The sample is usually transported from the sample intake to the collection bottle by a plastic tube called the "sample transport subsystem." Make sure the composite sampler is placed well above the sample stream to ensure the tubing runs in a taut, straight line to prevent pooling of liquid. Take care to avoid sharp bends and twists in the transport line. The sample storage subsystem can accommodate either a single large collection bottle or a number of smaller collection bottles. Samplers with individual bottles for discrete collection are usually equipped with a cassette that rotates to fill the bottle during sampling. The total sample volume storage capability should be at least 2 gallons (7.6 liters). Some samplers have a capacity of up to 5 gallons.

If the preservation requirements for a particular analysis specify that a sample be thermally preserved, use a refrigerated autosampler or cool the sampler by placing cubed or crushed ice in the center of the base section so samples are kept at 6°C or less during the entire sampling period. Field staff can either dump ice into the center of the base section or place the ice in a plastic bag. For the most uniform cooling, it is best to let the melt water from the ice run between the sample bottles, creating an ice bath. The quantity of ice used will be dependent upon the ambient temperature of the sampling site and type of bottles used. For maximum cooling, fill the base (with bottles in place, as well as the sampler's retaining ring) with water and freeze the base and contents. Both the center section and the base section have double-walled construction with polyurethane foam insulation. The insulation has a standard thermal resistance factor of R-11 (ISCO 2013). For a composite sample collected with an automated sampler, filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited in place of a composite sample collected automatically.

Sample collection vessels, either large composite or discrete sample containers, must be constructed of materials appropriate for the tests to be performed. If samples for extractable organics are to be collected, *all* parts of the sampler that come in contact with the wastewater stream must be constructed of materials appropriate for extractable organics collection and analysis. See **Appendix B** for a list of approved container types for various analyses.

**Special Considerations for Metals**
If the automatic sampler is attached to a metal conduit pipe, the intake tubing should be carefully installed upstream and away from the conduit to prevent metals contamination. This can be accomplished by clamping the tubing upstream of the conduit using laboratory clamps and wrapping the submerged portion of the conduit pipe with a protective barrier (e.g., duct tape).

**Special Considerations for Extractable Organics, Pesticides, and PCBs**
When an automatic sampler is used for collecting samples for the analyses of extractable organic compounds, pesticides and/or PCBs, the installation procedures include cutting the proper length of new Teflon® tubing, rinsing of the entire sampler collection system with analyte-free water and collection of appropriate equipment blanks for organic compounds analysis. For the analyte-free water rinse, approximately one-half gallons is initially pumped into the composite sample container and discarded. An additional one and one-half gallons (approximate) are then pumped into the composite sample container for distribution into the appropriate blank container(s).

### *6.2.4 Manual Flow-Proportioned Composite Samples*

If the required composite sample is flow-proportional and a “bubbler” was not used to apportion samples, then at the end of the sampling period Field Inspectors will need to manually combine individual samples proportional to flow. This section presents a calculation you can use to determine the volume of each grab sample at a given time to produce a composite sample manually.

The volume of sample collected at any given time depends on the volume of flow at that time, the total flow for the sampling period, and the total composite sample volume required for all analyses. The following equation can be used to calculate a grab sample's volume for compositing:

$$\text{Grab sample volume, mL} = \frac{(\text{Flow rate at sample time}) \times (\text{Composite sample volume needed})}{(\text{Total flow during compositing period})}$$

For example, the total flow during the compositing period is 600,000 gal/day and you need 2,000 mL of sample water to conduct analyses. At the time you take your first sample, the plant's flow is 40,000 gal/day, so you can calculate the volume of the 1st aliquot as follows:

$$\text{Aliquot sample volume, mL} = \frac{40{,}000\ gpd \times 2{,}000\ mL}{600{,}000\ gpd}$$

$$\text{Aliquot sample volume, mL} = 133\ mL$$

So, 133 mL of sample water from bottle #1 should be added to the composite jug for analysis. Once you calculate the grab sample volumes necessary to create the flow-proportional composite sample from the remaining samples, ensure that the contents of each individual grab sample container are thoroughly mixed before pouring the appropriate sample volume into the compositing jug. Then, stir the contents of the compositing jug before siphoning its contents (poured if no visible solids) into the respective sample containers for analysis. Immediately filter and/or preserve the samples, if required, then cap, seal, and ensure sample containers are properly labeled. Refer to **Appendix B** for appropriate sample containers and preservation procedures.

### *6.3 Quality Control Activities*

Prepare and submit blanks (**Table 2**) at a frequency of once per sampling run and collect equipment blanks each time the automatic sampler is set up, unless specifically indicated by the NPDES permit(s), or as described in a project-specific work plan.

#### *6.3.1 Nutrients Field Blank*

Fill a 1-quart cubitainer with analyte-free water in the field. Add 2 mL of concentrated sulfuric acid when acidifying the wastewater samples. Keep blank on ice, at 6°C or less.

#### *6.3.2 Bacteria Field Blank*

Fill a 125-mL sterile polypropylene/IDEXX bottle containing sodium thiosulfate to the 100-mL line with analyte-free water in the field. Keep blank on ice, below 10°C. In most cases, collect one set of quality control samples with each group of samples collected on consecutive days within one week. Generally, no less than 10% of the samples should be quality control samples. Process the blank sample at the same time as wastewater samples. Incubation must begin no later than 8 hours from time of collection.

#### *6.3.3 Cyanide Field Blank*

Fill a 1-quart cubitainer with analyte-free water in the field. Add the same number of sodium hydroxide (NaOH) pellets to the blank as were added to the wastewater samples. Keep blank on ice, at 6°C or less.

#### *6.3.4 Dissolved Metals Equipment Blank*

Fill a 1-quart cubitainer with analyte-free water in the field. Follow the filtering procedure for collecting a dissolved metals sample (6.1.5) but instead of inserting the tubing into the cubitainer containing the wastewater sample, insert it into a cubitainer containing the analyte-free water. Rinse the tubing and filter with approximately 50 mL of analyte-free water and discard. Pump the remaining analyte-free water through the filter and into a second, new, 1-quart cubitainer. Acidify with two, 2.5 mL aliquots of concentrated nitric acid when acidifying the wastewater samples. Keep blank at ambient temperature. After the blank has been prepared, transfer the tubing to the 1-quart cubitainer containing the wastewater sample and proceed with method 6.1.5.

#### *6.3.5 Volatile Organic Compounds (VOC) Trip Blanks*

The SLD Organics Section prepares VOC Trip Blanks. They should be included with any request for VOC sample vials. They should be labeled by the Inspector, kept in a Whirl-Pack and exposed to the same conditions as environmental VOC samples, except that the vial lids should never be removed.

#### *6.3.6 Other Blanks*

Collect blanks for ions, organics or total metals only if required by the NPDES permit(s) or study-specific work plan. For other types of equipment blanks, pass analyte-free water into, over, or through the equipment (e.g., churn splitter, automatic samplers, etc.) that is exposed to the sample. Collect composite sampler container blanks by adding analyte-free water to the cleaned sample container, mix the water thoroughly within the container, and then pour off an aliquot for analysis. Equipment blanks should be collected when an automatic sampler is used for collecting samples for metals analyses, if equipment is field cleaned and re-used on-site, or if necessary to document that low-level contaminants were not introduced by the sampling equipment. Collect the blank in the same type of container as the wastewater sample. Preserve and transport in the same manner and analyze for the same parameters.

#### *6.3.7 Replicates and Duplicates*

Collect replicate or duplicate samples only if required by the NPDES permit(s) or study-specific work plan.

**Table 2.** Blank types and suggested frequencies for submittal.

| ***Analytical Suite*** | ***21* Field Blank*** | ***26* Trip Blank*** | ***27* Reagent Blank*** | ***28* Equipment Blank*** | ***Submittal Frequency (per run**)*** |
|---|---|---|---|---|---|
| TSS/TDS/Alkalinity/Ions | | | | | 0 |
| Nutrients | X | | | | 1 |
| Bacteria | X | | | | 1 |
| Biological Oxygen Demand – 5 day | X | | | | 2*** |
| Total Metals/Total Recoverable Aluminum | | | | | 0 |
| Dissolved Metals/Total Recoverable Aluminum (if filtered) | | | | X | 1 |
| Cyanide | X | | | | 1 |
| SVOCs/Herbicides/ Pesticides | X | | | | 1 |
| VOCs | | X | | | 1 |
| Radionuclides | | | | | 0 |

* Numbers refer to Water Quality Exchange (WQX) activity type. ** Run is usually equivalent to a sampling inspection trip. Some inspection trips are composed of multiple facilities on multiple days in which case blanks should be collected at a rate of 10% of the total number of samples collected for each parameter. ***When preparing samples for 5-day BOD analysis, completely fill two bottles with dilution water to be incubated as blanks.

### *6.4 Handling, Packaging, and Transporting Samples*

After collection, preserve each sample within 15 minutes of collection if necessary. Place samples with temperature storage requirements in coolers. Ensure that all samples required to be kept cool are surrounded and in contact with enough ice to cool to 6°C or less. It is important that containers are in an ice bath; i.e. in contact with water that is in contact with ice, especially in warm weather, to ensure adequate cooling. Make sure that all glass sampling containers are placed in bubble-wrap sleeves to protect from breaking. Bubble-wrap may insulate samples from cooling and it may be necessary to place additional ice in coolers. Check to see that samples are adequately labeled and that container lids are secure. Handling procedures and holding times are summarized in **Appendix B**.

*6.4.1 Sample Custody*

Two levels of sample custody are practiced at the SWQB: a sample tracking procedure used for ambient/assessment water quality monitoring samples and a formal chain of custody procedure used for compliance sampling inspections.

The SWQB uses a sample tracking procedure that requires that the receiving laboratory acknowledges receipt of the samples by date stamping the submittal forms and providing copies of the stamped forms to the person delivering the samples. Compliance samples also require an additional handling step of sealing all samples collected for compliance monitoring purposes with evidence tape to prevent tampering or contamination. Samples taken during compliance sampling inspections shall follow chain of custody procedures provided by the contracted analytical laboratory.

***6.5 Documents and Records***

Compliance/Inspection documents include field notebooks, calibration records, instantaneous probe data, validation and verification records, sample collection data, records of analytical data in hard copy or in electronic form and QC records. Documents will be maintained in accordance with the requirements of the Bureau Quality Assurance Project Plan (QAPP).

| Organizational Unit | Reporting Documents Produced | Description |
|---|---|---|
| Point Source Regulation Section (PSRS) | Inspection Reports | Inspection reports are partially based on a review and evaluation of records maintained by the facility and PSRS. Records reviews vary by the type of facility (different facilities/permits have different record keeping requirements) and may include: monitoring reports, previous inspection reports, permit applications, permits, and past or pending EPA enforcement actions, laboratory records, site self-inspection records, employee training records, nutrient management records, etc. Results from inspections are documented on EPA form 3560-3 and are discussed in the Inspection Report. Inspection Reports include details on all findings made during an inspection and may include photographs taken during the inspection. Inspection reports are submitted to both the facility operator(s) and EPA. Inspection reports may be used to determine compliance with the federal CWA. |

**NPDES-Specific Records**
Information generated or obtained by PSRS personnel during a Compliance Sampling Inspection will be organized and accounted for in field forms, bound field logbooks, and/or chain-of-custody documentation.

## 7.0 Related Forms

Field Data Form/Field Log Book
Wastewater Sampling Equipment Checklist
ISCO Deployment Form
Total Residual Chlorine Data Upload Form
Bacteria Data Upload Form
SLD Submittal and Chain-of-Custody Forms
EPA Form 3560-3

## 8.0 Revision History

Original – 08/08/2014

## 9.0 References


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## APPENDIX A

Wastewater sampling equipment checklist.

| | |
|---|---|
| **SAMPLING SUPPLIES**<br>Field sheets<br>Submittal forms/Chain of Custody Forms<br>RID stickers<br>Tape for custody sealing samples<br>Cubitainers (quarts, gallons)<br>Whirl-Packs<br>Organics sample bottles (vials/bottles)<br>VOC trip blanks<br>Analyte-free water for preparing blanks<br>Cartridge filters (0.45 µm and 10 µm) – for metals filtering<br>Acid washed silicon tubing – for metals filtering<br>Geo pump, with appropriate power cord(s)<br>Kimwipes<br>Acid kit with pipettes, tips, and concentrated sulfuric and nitric acid<br>Pipette repair kit<br>Sodium hydroxide pellets<br>10% HCl for acidifying VOC samples<br>pH paper<br>Potassium iodide (KI) starch Test Paper<br>Ascorbic acid<br>Sodium thiosulfate<br>Nitrile gloves<br>Pencils<br>Sharpies (fat and fine point)<br>Pole sampler<br>Nylon rope (optional – depends on sampling locations)<br>Metal bucket<br>Plastic bucket<br>Ice<br>Coolers | **GENERAL**<br>Access authorization documents<br>Defensive driving certificate<br>Camera<br>Cell phone<br>First aid kit<br>Flashlight<br>GPS unit<br>Maps<br>NMED business cards<br>NPDES Inspector Credentials<br>Toolbox<br>Calculator<br>Hat<br>Sun screen<br>Rain gear (optional)<br>Sunglasses/polarized glasses/safety glasses<br>Clipboard<br>Boots/waders/sandals/steel-toed boots<br>Hard hat/safety vest<br>Garbage bags<br>Apron<br><br>**COMPOSITE SAMPLING (ISCO)**<br>ISCO 3700 Composite Sampler<br>collection bottles with caps<br>compositing jug<br>pump tubing and suction lines<br>strainers<br>flow meter (“bubbler”)<br>battery/power pack |

## APPENDIX B

Sample handling procedures and holding times.

| Sample Type | Sample Container | Processing, Preservation and Storage | Maximum Holding Time |
|---|---|---|---|
| **Bacteria** | | | |
| Total Coliform and *E. coli* | 125-mL sterile polypropylene or glass container | Cool on ice, less than 10°C, 0.0008% $Na_2S_2O_3$ | 8 hours |
| **Inorganics** | | | |
| TSS/TDS/Ions/Alkalinity | 1-quart polyethylene cubitainer or 250 mL HDPE bottle if sample is only for TSS/TDS | Cool on ice, ≤ 6°C | 7 days if TSS included; otherwise 14 days. |
| Total Nutrients | 1-quart polyethylene cubitainer | 2.0 mL concentrated sulfuric acid, cool on ice, ≤ 6°C | 28 days |
| Cyanide | 1-quart polyethylene cubitainer | 5-7 pellets NaOH, 0.6g ascorbic acid if chlorine present, cool on ice, ≤ 6°C | 14 days |
| Biochemical Oxygen Demand | 1-quart polyethylene cubitainer or 1-L glass bottle | Cool on ice, ≤ 6°C | 48 hours |
| **Metals** | | | |
| Total Metals | 1-quart polyethylene cubitainer | 5.0 mL concentrated nitric acid | 28 days |
| Total Recoverable Aluminum | 1-quart polyethylene cubitainer | Filter (10 μm) within 15 min of sample collection; 5.0 mL concentrated nitric acid. | 6 months |
| Dissolved Metals / Hardness | 1-quart polyethylene cubitainer | Filter (0.45 μm) within 15 min; 5.0 mL concentrated nitric acid | 28 days if mercury included in analysis; otherwise 6 months |
| **Organics** | | | |
| Semivolatile Organic Compounds, Herbicides, Pesticides | Two 1-liter glass amber bottles (lab) | Cool on ice, ≤ 6°C | 7 days |
| Volatile Organic Compounds | Two 40-mL glass vials (lab) in a Whirl-Pack | 5 drops 10% HCl per bottle (HCl provided by lab), cool on ice, ≤ 6°C | 14 days |
| 10% HCl for VOC preservation | 10 mL dropper bottle | Cool on ice, ≤ 6°C or in refrigerator | 30 days |
| **Radionuclides** | | | |
| Radionuclides | Two 1-gallon polyethylene cubitainers | 7-10 mL concentrated nitric acid per gallon | 6 months |