

Section No.: 7  
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**MASSACHUSETTS WATER RESOURCES AUTHORITY**  
**INDUSTRIAL PRETREATMENT PROGRAM**  
**SAMPLING PROCEDURES/PROTOCOLS**

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**DISCLAIMER FOR**

**MASSACHUSETTS WATER RESOURCES AUTHORITY**

**INDUSTRIAL PRETREATMENT PROGRAM**

**SAMPLING PROCEDURES/PROTOCOLS**

**The information provided in this document is not official policy of Massachusetts Water Resources Authority (MWRA). It is guidance for MWRA's Toxic Reduction & Control Department (TRAC) sampling staff. TRAC staff's use of the procedures described in the document is subject to the professional judgment of TRAC staff and management. The information contained in this document is not intended to provide any legal rights to any actual or potential sewer user or to any other person.**

**Following the procedures set forth in this document is not a guarantee that sampling will be performed appropriately. Sampling should comply with the requirements of applicable permits, if any, and with the regulatory requirements of the program for which the samples are taken.**

**The information provided in this document is subject to change without notice. Not all of TRAC's sampling procedures are incorporated into this document. This document may not reflect changes to TRAC's procedures that have occurred since the document was last updated.**

***TABLE OF CONTENTS***  
**Chapter Seven**

<b>7.</b>	<b>SAMPLING PROCEDURES</b>	<b>Page #</b>
7.1	<u>Equipment Required</u> .....	2
7.2	<u>Approaching Industries and Municipalities</u> .....	3
7.3	<u>Containers and Preservatives</u> .....	5
7.4	<u>Sampling Location</u> .....	8
7.5	<u>Grab Sampling</u> .....	10
7.6	<u>Composite Sampling</u> .....	15
7.7	<u>pH</u> .....	34
7.8	<u>Temperature</u> .....	37
7.9	<u>Observations</u> .....	37
7.10	<u>Flow</u> .....	37
7.11	<u>Specific Procedures for CSO Monitoring</u> .....	42
7.12	<u>Specific Procedures for POTW Monitoring</u> .....	42
7.13	<u>Sample Delivery/Holding Times</u> .....	46

**List of Figures - Appendix 7A**

**7. SAMPLING PROCEDURES**

## 7.1 Equipment Required

The following sampling equipment must be available, in good working order, and clean:

- Composite samplers and associated equipment (Section 7.6.2)
- Cooler with ice
- pH meter (with pH electrode with built in ATC probe and 4, 7, 10, and 12.45 pH buffers)(Section 7.7)
- De-ionized water rinse bottle for cleaning the pH electrode
- Residual chlorine meter Hach DR/890 Spectrophotometer)
- ISCO or American Sigma portable peristaltic pump
- Appropriate sample bottles for scheduled sampling events
- Extra bottles (to replace broken bottles and enabling sampling of additional sites noted while in the field). Extra bottles may also be required for the actual sampling process. Clean, unpreserved plastic bottles may be cut to fit into small, otherwise inaccessible sampling points). (Section 7.3)
- Funnels (thin and wide neck, enough for each sampled company, plus extras in case one is dirty)
- Bucket and rope
- Pick, shovel and portable gas meter (Biosystems PHD Ultra Gas Meter) for manhole work (see Chapter 10, Section 10.3)
- Gloves (non-powdered polyvinyl for sample collection, cotton work gloves for moving equipment)
- Map Book
- Sample Record Forms (SRFs), industrial permits, and inspection reports (Figure 7.1, 7.2 and 7.3, Appendix 7A) must be consulted when preparing for a monitoring event. The appropriate sample container (including preservative, if applicable) should be packed for each parameter to be sampled (Figure 7.4 and 7.5, Appendix 7A).
- Chain of Custody Forms (COCs) must be completed for each set of samples
- Sample bottle labels

**Note: It is good practice to always have extra equipment and blank sample record forms in the field to handle unexpected situations.**

## 7.2 Approaching Industries and Municipalities

### **7.2.1 Approaching an Industry**

Upon arriving at the industry, the monitoring team should introduce themselves to the receptionist and ask for the contact whose name has been provided on the SRFs. A proper identification includes the use of business cards and identification badges. I.D. badges should be worn at all times during the monitoring activities.

If the contact is unavailable or is no longer with the company, the receptionist is usually helpful in locating a replacement. It is important that monitoring occurs during unannounced visits, so that the sample is representative of an everyday discharge. Only in extreme cases (i.e., infrequent batch discharges) should appointments be made. Under no circumstances is a Sampling Associate to reschedule a visit at the request of industry. If a difficult situation develops, the Sampling Associate should contact the Regional Manager or Sampling Coordinator for instructions. The sampling team should wait no longer than 10 minutes for the contact or his/her designee to meet them. If the waiting time exceeds this limit, then the sampling team should seek justification from the contact. The sampling team should also record this information and forward it to the Industrial Coordinator.

When the contact greets the monitoring team, the lead member of the team should act as a spokesperson and explain the reason for the visit. This may include a brief summary of MWRA's Industrial Wastewater Pretreatment Program and specifics regarding the company. The Sampling Associate should inquire about possible process changes and any other relevant information including hours of operation and discharge along with other pertinent questions mentioned previously in Section 6.1. Any additional areas of concern should be noted on the Monitoring Report and brought to the Industrial Coordinator's attention.

If a contact should question a Sampling Associate's right to enter the business, the Sampling Associate should refer the contact to the MWRA Sewer Use Regulations (360 CMR, Section 10.012, Access to Facilities), or their MWRA Discharge Permit. Both documents describe TRAC's legal authority to enter a facility during normal business hours. Finally, if the contact is still uncooperative, the contact should telephone the MWRA office and speak with the Regional Manager or Sampling Coordinator who will advise the contact of their requirements to cooperate with the program. If all else fails, the monitoring team should specifically ask if the contact is denying access for the monitoring event. If so, leave the company, and contact your supervisor for further instructions.

If the contact states that the company is not discharging process wastewater at the time of the visit, verify this by a visual inspection. The monitoring team can then schedule a return visit when the industry anticipates discharging. This is usually the procedure for batch discharges. If the company is no longer discharging wastewater to the sanitary sewer system, the monitoring team should note this or any process changes on the Monitoring Report. Inform the appropriate

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

Industrial Coordinator using the MWRA - Inspection Communication/Encoding Request Sheet (Figure 7.6, Appendix 7A). This will allow the Industrial Coordinator to verify such changes and have them encoded into TRAC-IS (database).

Any telephone conversations with personnel at a company are recorded using the Telephone Memorandum form (Figure 7.7, Appendix 7A), a copy of which is then stored in the company's main file.

### **7.2.2 Municipal Sewer Facilities**

Before entering or conducting any work in or around municipal manholes, pump stations, etc., contact the community sewer representative for their guidelines to follow when entering into municipal sewer facilities.

### **7.3 Containers and Preservatives**

### 7.3.1 Containers

Some sample containers are supplied to the TRAC monitoring section with preservatives added to the containers. The Central Laboratory supplies bottle labels with the appropriately marked preservative. It is also important to use the correct type of bottle (glass - clear or amber, or polyethylene) when collecting samples. Figure 7.4, Appendix 7A lists the correct EPA required sample bottle type, preservative, holding time and temperature for all parameters currently sampled by TRAC.

### 7.3.2 Bottle Labels

Sample bottle labels (Figure 7.8, Appendix 7A) are supplied by the Central Lab with the sample ID number, the Client name, the Project name, the Location Code, the Trip number, the parameter to be collected, the preservative (if applicable) and the container to use for the sample. Sampling Associates fill in the Date/Time Collected and who collected the sample using permanent felt tip marker (e.g. Sharpie brand). Do not use Dry Erase or water-soluble markers for labeling bottles.

### 7.3.3 Preservative

The preserved samples must be checked for adequate preservative in the field. The pH of all acid preserved (nitric, hydrochloric and sulfuric acid) samples must be equal to or less than <2 s.u. The pH of all base preserved (NaOH) samples must be equal to or greater than >12 s.u. Verify the pH using pH strips by pouring a small amount of the sample into the sample cap and moistening it with the pH strip. The measurement must be recorded on the SRFs and the COCs for the samples. If a sample is not adequately preserved, add additional volumes of the proper preservative. The sample's pH must be immediately adjusted to the proper range for adequate preservation. **The volume/amount of preservative added is noted on the COCs and SRFs for the samples.**

**NOTE: Use the following procedure whenever a sample cannot be adequately preserved:**

**Lowering the pH to <2:**

- **For non-metals samples: Never add more than 10 mL of the acid to the sample. Note on the SRF and COC that the sample could not be adequately preserved with 10 mL of acid.**
- **For metals samples: Never add more than 20 mL of the acid to the sample. Note on the SRF and COC that the sample could not be adequately preserved with 20 mL of acid. The metals sample bottle should be filled to capacity with sample if 20 mL of acid is used.**

**Increasing the pH to >12:**

- **Add enough pellets of NaOH to increase pH to 12 or greater. Note on the SRF and COC the number of NaOH pellets used to preserve the sample. Cyanide samples that are preserved in the field need to be re-checked for pH when the samples are returned to Chelsea for laboratory courier pickup.**

**7.3.4 Bottle Orders**

Sample bottles are supplied from the Central Laboratory. The Sampling Coordinator assigns responsibility for bottle orders along with other maintenance tasks (Chapter 11).

**7.3.4.1** A bottle order form is filled out and the order is faxed to the Central Lab or is picked up by the Central Lab courier.

**7.3.4.2** The Central Laboratory courier delivers bottles to Chelsea. A copy of the drop-off receipt is kept on file.

**7.3.4.3** The Sampling Coordinator is informed by staff responsible for this task when the supply of bottles is less than a one month's supply of a particular type, so more can be ordered in a timely manner. Standing orders can be filled within one week.

**7.3.5 Refrigeration at Chelsea**

**7.3.5.1** All samples stored overnight in the Chelsea location must be stored with a signed custody seal and completed COC in the refrigerator. The COC should be signed as "Relinquished by" with the date and time. In the "Received by" space "SAMPLE STORAGE" is written. When removing samples from the refrigerator, by the "Relinquished by" space fill in "SAMPLE STORAGE" and the date and time and sign your name as "Received By." Sign and date the COC as "Relinquished By" when relinquishing the samples to the lab.

The courier at Chelsea or the sample log-in person at the laboratory who receives the samples will sign as "Received by." By following these procedures, the time of sampling to the time of analysis will be fully documented on the COC.

**7.3.5.2** Sign the "Refrigerator Custody Log" (Figure 7.9, Appendix 7A) that is attached to the refrigerator door when storing and retrieving samples. Also confirm that the temperature is at 4 degrees centigrade, as required.

**7.3.5.3** The sample refrigerator must be locked at all times even when empty.



Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

- 7.3.5.4** Care must be taken to assure that samples with short holding times are not stored in the refrigerator over extended periods (See section 7.13.5, holding times for sample parameters). Allow at least 8 hours for the lab to start the analysis after dropping off the short holding time samples. If fewer than 8 hours are left before the holding time expires when the sample is received by the lab, notify the Sampling Coordinator or Regional Manager as soon as possible so the laboratory staff can be notified of the situation. No sample should be stored in the refrigerator for more than 3-4 days except under special circumstances.
- 7.3.5.5** The refrigerator is not to be used as a substitute for dropping samples off on a daily basis. Only store samples in the refrigerator when it is not feasible for sample pickup by lab courier by 4:00 PM Monday - Friday. If you must get to the Central Lab after 4:30 PM or on the weekend, please notify the Client Services Coordinator prior to 3:00 PM (or the Friday before in the case of weekend sampling). The Client Services Coordinator will make arrangements with the lab to accept the samples and start the analyses after regular business hours.
- 7.3.5.6** The same person who stored the samples in the refrigerator should deliver them to the laboratory. If this is not feasible, proper sign-off procedures must be followed on the COC for those samples.
- 7.3.5.7** The Senior Sampling Associates check the refrigerator temperature daily and record it on the Refrigerator Temperature Log (required by EPA) on the front of the refrigerator (Figure 7.12, Appendix 7A). The Sampling Coordinator assigns this task along with other maintenance tasks (Chapter 11).

## **7.4     Sampling Location**

- 7.4.1** The first step in the actual sample collection procedure is to carefully evaluate the industrial process of concern, and the path of its discharge into the sewer system via any pretreatment system. In general, if no pretreatment system is present, the sample should be collected at the

point of discharge of the process before the wastewater mixes with other processes or non-process wastewater.

In the case of process discharges which receive pretreatment prior to discharge into the sewer system, samples should be taken at the point of discharge after the pretreatment system and before the treated wastewater mixes with other process or non-process wastewater. Generally, the SRF will provide an adequate description of the appropriate sample point location. If the sample location description is not clear, refer to the inspection report, old monitoring reports or speak the Industrial Coordinator assigned to that facility.

If the sample point location description is not clear, the Industrial Coordinator or the contact for the company should be able to provide the necessary information for clarification. All details and information acquired during monitoring events are noted on SRFs and monitoring reports for future reference.

- 7.4.2** Section 10.008 of the Sewer Use Regulations (360 CMR 10.000) states that the Authority may require an industry to install a suitable site where monitoring can be conducted easily and safely. There is a procedure in place whereby an industry that does not have an accessible and safe monitoring location will be notified and required to install or modify the site to MWRA specifications. If a site is determined to be unsafe, radio or call the office for instructions on how to proceed before conducting any sampling.

If, during a monitoring activity, the Sampling Associate determines that the sampling site is inadequate, the Regional Manager, Industrial Coordinator and the Senior Sampling Associate should be notified using the MWRA - Inspection Communication/Encoding Request Sheet (Figure 7.6, Appendix 7A). Depending on the changes to be made, either the Industrial Coordinator or the Senior Sampling Associate for the region will draft a letter to the specific company requiring them to change or modify the sampling site. Noncompliance with the letter could result in a violation of MWRA Sewer Use Regulations.

**7.4.3 Oil & Water Separators, Grease Traps**

Only sample Oil/Water Separators and Grease Traps after the unit. If a special request is made to sample directly from the separator, the MWRA approved design should be consulted for determining the proper sampling point indicated by an "X" (Figure 7.10, Appendix 7A)

**7.4.4 Sampling at Silver Recovery Units (SRUs)**

The following are guidelines for sampling SRUs. The sampling location will be after the SRU unless otherwise requested.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

**7.4.5** In all cases, when collecting samples it will be necessary to adapt to the conditions found at each site. Although there will be times when it is necessary to deviate from standard procedures, whenever possible the methods described in this QAPP should be followed. Any modifications in procedures must be accurately recorded on the field SRFs and brought to the attention of the Regional Manager for further corrective action if required (Chapter 13).

## **7.5 Grab Sampling**

### **7.5.1 Definition of Grab Sample**

A grab sample is defined in 360 CMR 10.004 as:

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

"Grab Sample shall mean an individual aliquot collected over a period of time not exceeding 15 minutes."

The grab sample is collected from a wastewater discharge on a one-time basis without regard to the flow and consideration of time. The frequency at which grab samples are taken depends on the variability of possible contaminants in the wastewater. If the wastewater is highly variable, take grab samples as often as once every five minutes. If the wastewater is very constant in composition (homogenous), take samples at longer intervals.

Under certain conditions, grab samples are the preferred method of sampling as follows:

- When flow is not continuous.
- When the discharge is a batch process and the tank is periodically dumped, the wastewater shows its true characteristics at the time when the tanks are being discharged.
- When wastewater is collected over time and pretreated as a batch prior to discharge, the frequency of discharge may vary, for example, once per day, once per week, or once per month.
- When is it desirable to check for extreme conditions: Composite methods conceal peak values of wastewater characteristics. For example, pH may appear to be neutral in a composite sample even though it fluctuated over a wide range. The acidic/alkaline conditions encountered may simply neutralize the sample. However, grab samples taken at different times may show variations in pH.
- It should be noted that although grab samples in some cases accurately represent the wastewater discharge, it is MWRA practice in most instances to collect composite samples whenever possible. This includes compositing during the discharge of a batch if the volume and duration of the discharge allow compositing the sample. If the batch discharge occurs in less than one hour, try to take grab samples at the **beginning, middle and end of the discharge and composite them**. Otherwise, only take grab samples if this can't be done.
- Grab samples must be used to sample for the following parameters either due to short holding times, the nature of the analyte, or the requirement of the analytical method:
  - Dissolved Gases
  - Total Chlorine Residual (TCR)
  - Fats, Oil, and Grease (FOG)
  - Petroleum Hydrocarbons (PHC)
  - Bacteria (FCOLI or TCOLI)
  - pH
  - Temperature
  - Formaldehyde

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

**NOTE:** See figure 7.4 and 7.5, Appendix 7A, for additional parameters and holding times, in addition to a list of parameters that are taken as manual composites.

**NOTE:** With every set of paperwork for a monitoring event, there should be one SRF for pH only and one SRF for pH and any other grab parameters. Take the grab parameters on the first day and the pH "only" on the middle or pickup day.

## **7.5.2 Procedure for Grab Sampling**

**7.5.2.1** Use a clean wide mouth glass bottle. If a glass bottle doesn't fit in the sampling location, a small beaker or a plastic bottle cut to fit in the sampling location may be used. Be sure the parameters collected are not for dedicated grab samples and use compatible sampling containers.

Dedicated grab samples are those that can only be collected into the actual sample container that will be submitted for analysis. The sample cannot be collected into a separate container and transferred or collected with a sample pump. There are 2 sample parameters that must be collected in a dedicated sample container: Petroleum Hydrocarbons (PHC), and Fats, Oil, & Grease (FOG).

A PHC sample must be collected into an unpreserved 500-mL amber glass bottle and then preserved to pH <2 with 1:1 HCL. This sample must be collected into the sample bottle that will go to the lab whenever possible. This ensures that the loss of PHC analyte is minimal. A sample transferred or pumped to another container could lose analyte.

Follow EPA Method 1664 to collect FOG samples. An aliquot sample of 500 mL must be simultaneously collected into a graduated beaker (test sample) and a clean 500-mL amber glass sample bottle. The test sample inside the beaker must be acidified (preserved) to a pH <2.0 s.u. with 1:1 HCL. The amount of acid that was used to preserve the test sample must be used to preserve the sample in the sample bottle. Record the amount of acid used on the SRF and the COC. After preserving the FOG sample, neutralize the test sample with NaOH and discard into the wastewater stream.

**7.5.2.2** For sample collection other than dedicated PHC or FOG samples, rinse the collection vessel (if one is used) 3 times in the wastewater stream.

**7.5.2.3** Obtain a grab volume large enough to fill all the sample containers required for the parameters to be analyzed. A Teflon, stainless steel, or glass sample vessel may be useful to facilitate this (**if sampling for metals analysis, do not use stainless steel**). A bucket and a rope are also useful when sampling from manholes.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

**REMEMBER:** Rinse all sample vessels used to collect the sample 3 times in the wastewater stream before collecting the sample. Do **NOT** use a sample vessel when sampling for FOG or PHC. Sample for these directly into the 500-mL amber glass bottles as stated above.

**7.5.2.4** Minimal transferring of sample is desirable. If possible, sample directly from the wastewater stream into the sample bottles. If a bottle contains a preservative, sample using a sample vessel and transfer the contents into the final preserved sample container.

**7.5.2.5** An additional method for grab sample collection is to use a portable sample pump or the composite sampler to pump samples from the waste stream. This method is **NOT** to be used for FOG, PHC, Acrolein or VOA samples, but it can be useful when sampling from drains covered with grates which are difficult to remove or from discharge pipes exiting tanks at a level well below the surface. Intake tubing can be fed into the final discharge pipe.

In all cases, the equipment must be scrupulously clean and rinsed 3 times with the wastewater stream to prevent contamination during the collection and transfer processes. Virgin pump/sample tubing must be used at all times.

**7.5.2.6** The mouth of any bottle or other collection device should be pointed upstream to keep contamination from the outside of the bottle or from the gloves of the person sampling from being washed into the bottle.

**7.5.2.7** Volatile Organics (VOA) sampling: The VOA sample container is a 40-mL borosilicate glass vial sealed with a Teflon/Silicon septum and screw cap.

**NOTE: Dechlorinate VOA samples if TCR is detected. Refer to Sections 7.6.2.20 - 7.6.2.26 for this procedure.**

- The VOA vial is filled with as little aeration as possible until a convex meniscus is formed.
- The septum is placed with the thinner Teflon side towards the sample and the screw cap is tightened in place.
- The vial is then inverted, tapped lightly and checked for any air bubbles. It is of extreme importance that samples analyzed for VOAs are collected without headspace or entrapped air. The above procedure must be repeated until this is achieved. If the sample is warm when collected, entrapped air will often form bubbles upon cooling. There is no way to avoid this problem. If this occurs prior to log-in at the laboratory, note it on the COC.
- Always collect VOA samples in duplicate. In addition, take two trip blanks (VOA vials received from the laboratory with deionized, organic free, water) to each site. At the industry, remove the trip blanks from the bag and place them near the sample collection point. If there are any questions regarding contamination in the VOA sample, analysis of

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

these blanks will provide data confirming that contamination of the sample did not occur during the sampling event or during transport.

- The sample vials and two trip blanks are stored together in a plastic bag for the entire trip from the company to Chelsea and to the laboratory.

**7.5.2.8** Acrolein sampling: The sample container is a 40-mL borosilicate glass vial sealed with a Teflon/Silicon septum and screw cap.

**NOTE: Dechlorinate Acrolein samples if TCR is detected as described in Section 7.6.2.22 and in the Acrolein/Acrylonitrile SOP with associated flow chart.**

- The adjusted Acrolein vial is filled with as little aeration as possible until a convex meniscus is formed.
- The septum is placed with the thinner Teflon side towards the sample and the screw cap is tightened in place.
- The vial is then inverted, tapped lightly and checked for any air bubbles. It is of extreme importance that samples analyzed for Acrolein are collected without headspace or entrapped air. The above procedure must be repeated until this is achieved. If the sample is warm when collected, entrapped air will often form bubbles upon cooling. There is no way to avoid this problem. If this occurs prior to pick up by the courier or at sample log-in at the laboratory, note it on the COC.
- Always collect Acrolein samples in duplicate. Take two trip blanks (vials received from the laboratory with deionized, organic free, water) to each site. If there are any questions regarding contamination in the Acrolein sample, analysis of these blanks will provide data confirming that contamination of the sample did not occur during the sampling event or during transport. Remove the trip blanks from the plastic bag and place them near the sample collection point.
- The sample vials and two trip blanks are stored together in a plastic bag for the entire trip from the company to Chelsea and to the laboratory.

**7.5.2.9** Bacteria samples should also be collected in a dedicated manner whenever possible. Collect these samples into a 250-mL sterile bottle. Sodium thiosulfate should be added whenever the presence of TCR is suspected. Otherwise, the residual chlorine will kill the bacteria

**7.5.2.10** Use a minimal amount of duct tape for sample collection (preferably, use no tape), as the adhesives can contaminate the organics samples. Do not custody seal the sample containers with duct tape.

**7.5.2.11** Wheaton Grab Samplers or Swing Samplers can be used with a 1-liter non-preserved amber glass jar to assist in obtaining grab samples.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

- 7.5.2.12** Some industrial permits require analysis of pH only without composite parameters. In these cases, use one of the small opaque plastic screw-top bottles purchased expressly for collection of pH samples. Although it is not absolutely necessary to use these bottles, they are less expensive than other bottles. Measure the pH of these samples immediately in the field, and then discard the sample. Write pH only in the comment section of the COC. Submit the COC to the Central Laboratory with the pH reading on it.
- 7.5.2.13** When collecting samples from spigots, other devices or straight runs of pipe where solids may settle, purge the line before collecting grab samples or attaching suction tubing for composite samples.

## **7.6     Composite Sampling**

### **7.6.1   Definition of Composite Sample**

Composite samples are defined in 360 CMR 10.004 as:



Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

"**Composite Sample** shall mean a combination of a series of aliquots taken on either a time or flow proportional basis over a period of time."

A representative composite sample is always collected over the entire process day (or as close to it as possible). The use of times to stop and restart the automatic samplers and the use of the delay programs in the autosampler meet this requirement. Samples taken over a time period of 2 hours for an industry that discharges for 8-24 hours is not representative or acceptable. The composite must cover the entire process day and not overlap calendar days (unless the wastewater batch discharge continues after midnight). If the company does not have a 24-hour discharge, set the autosampler to run during the hours of discharge within the same calendar day. However, there are exceptions. For example, if the company discharges from 7 am until 2 am, try to sample all of the discharge for a representative sample for that time period. Twenty-four hour discharges can overlap calendar days. If an autosampler is set up to run for the calendar day, the starting time and ending time is 0001 to 2359 hours, respectively. When in doubt about unusual discharges, see the Regional Manager or the Sampling Coordinator.

The daily average limit, the highest allowable concentration for any pollutant in a waste stream discharged during one day by an industrial user, is based upon a representative composite sample. Ideally, the composite sample should reflect the variation in flow rate as well as the characteristics of the wastewater.

- 7.6.1.1 Equal sample volumes over regular time intervals are sometimes collected, but these do not give an accurate profile of the wastewater if the wastewater is highly variable. However, this method of time-pacing can be used in cases where flowmeters are not available or flow cannot be measured.
- 7.6.1.2 Composites of up to 24 hours may be collected, although the time period should never exceed 24 hours in the interest of sample preservation. The composite sample container must be packed in ice throughout the compositing period following EPA protocol for sample preservation. The sampling time period is determined according to the hours of discharge at the industry.

## 7.6.2 **Procedure for Composite Sampling**

Before attempting to use any of the sampling equipment, Sampling Associates must read all the literature supplied by the manufacturer with the unit. This ensures familiarity with the general procedures necessary to operate the unit. Each type of unit has a different set of procedures, and the Sampling Associate must be ready to set up any unit at any time. This preparation by the Sampling Associate is indicated on the Sampling Associate Training/ Field Audit Checklist.

Although the operation of each type of autosampler is different, there are basic steps, which must be followed to ensure a successful sampling event.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

- 7.6.2.1** Make sure the unit is clean. Break down the unit completely, checking all parts for contamination. Discrete autosamplers should be cleaned under the head to ensure free rotation of the sample distribution arm.
- 7.6.2.2** Change the silicone pump tubing and desiccant before taking any unit into the field. Use **NEW** tubing for each sampling event. The pump tubing is changed as follows:
- Make sure the unit is off and the power supply is disconnected.
  - Remove the screws on the pump housing and gently pull on the housing until it comes free of the shaft.
  - Rotate the pump while pulling on the tubing.
  - Install a clean, **NEW** section of tubing of the same length as the old tubing by reversing the removal procedures.
  - The desiccant is located either in a tube on top of the unit or in a bag under the head. Change the desiccant when necessary as noted on the indicator reading.
  - Change the fluid sensor and the composite tube if present on the model being used.
- 7.6.2.3** Place all necessary equipment with the unit. Strainers, hose clamps, batteries, power cords, and composite bottles should be placed in the unit so they will be ready when they are needed.
- 7.6.2.4** Check the unit's functions beforehand to save time trouble-shooting in the field.
- 7.6.2.5** Prepare an extra autosampler to avoid wasting time if there is a problem with an automatic sampler. If there is a problem with a unit, note the serial number of the unit and notify the staff responsible for the equipment or the Sampling Coordinator of the problem. Mark the autosampler as "out of order" and set aside in the Chelsea TRAC work area.
- 7.6.2.6** At the industry, locate the correct sampling point. Under most circumstances, use the sampling location identified in the facility's Sewer Use Discharge Permit. The sampling point should be easily accessible and allow placement of the strainer or the connection of the intake tubing to a sample port. A proper sampling point would be located after all pretreatment and prior to any mixing with sanitary wastes or other sources of dilution.
- 7.6.2.7** Use an AC power cord whenever possible to avoid losing samples due to low batteries. Inform the Regional Manager and note in the Monitoring Report any company that does not have access to AC power.
- 7.6.2.8** Autosampler placement: Place the autosampler as close to the sampling point as possible without interfering with the normal operations of the industry. This will allow the suction tube to be short and straight. Folds and/or bends in the suction tube may lead to inconsistent sample volumes.

**7.6.2.9** Place the autosampler in a location for ease of programming and setup.

**7.6.2.10** The strainer should be placed in an area of the wastewater discharge where it will be submerged during the entire sampling event. Place the strainer into the discharge pipe whenever possible.

Avoid placing the strainer in a cascading water situation; this will result in collection of unequal sample volumes. Avoid placing the strainer in standing effluent; this will give a non-representative sample (except in the case of a flow proportional sample). Keep the strainer in an area of flowing effluent, as close to the final discharge point as possible. This will ensure an accurate volume and a representative sample. If there is a problem with the flow being too small, try using a "low flow" strainer.

The strainers filter debris out of the waste stream before it can block the suction tubing (especially when sampling from sanitary wastewater). A large strainer should always be used where evidence of solids exists.

**7.6.2.11** Calculate the volume of sample to be taken at a given interval taking into account the number of samples needed to complete the sampling event, and the total volume of the composite. To calculate the volume of each interval sample, divide the number of samples into the total volume of the composite bottle.

**Example:** An autosampler is programmed to sample every 15 minutes over an 8-hour period. The volume of the composite bottle is 10 L.

$$\begin{aligned}\text{\# of samples} &= 4 \times 8 = 32 \\ \text{volume of sample} &= 10,000/32 = 312.5 = 320 \text{ mL}\end{aligned}$$

Always give extra volume to account for missed samples and inconsistencies inherent in the pumping system of the autosamplers. Regardless of the total number of parameters, try to get a full bottle (pickle jar) at all times.

**7.6.2.12** Pack ice around the sample bottle with sufficient ice to last from sampler setup to sample retrieval. According to EPA protocol, the composite sample temperature must be maintained at 4°C (40°F) or less throughout the sampling event for sample preservation. If on the second day of a 3-day setup, re-ice the sampler if necessary.

**7.6.2.13** Lock the cover of the autosampler to the base with 2 padlocks to prevent tampering with the sample. The tubing should be taped to prevent the strainer from being displaced. However, use care and make certain that the tape does not contact the wastewater. Custody tape (Chapter 8) should be used on all screw clamps, strainer tubing, and at the base of the autosampler (taped from the autosampler to the floor or adjacent wall) to prevent tampering. If tampering is suspected, never accuse a contact. Report the situation to the Regional Manager. Corrective action procedures will be followed (Chapter 13).

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

If automatic samplers must be left in outdoor locations, chains or cables should be used if possible to secure the autosampler to the nearest tree, pole, etc. Additionally, for rain protection, large trash bags can be used to cover the sampler. Trash bags can also be used for unusually foul environments within industries or manholes.

- 7.6.2.14** Sampling for Solids: Flat areas with sluggish flow are poor sampling locations because solids will tend to settle there. Under these circumstances, the collected sample may not be representative of the actual discharge. Solids may also settle upstream of weirs, therefore place the strainer downstream of weirs to collect samples.

A sample for total solids (TS), total suspended solids (TSS), and total dissolved solids (TDS) or any combination of these can be collected for analysis in one plastic, unpreserved, 1 liter bottle. The test for Settleable Solids (SS) requires the use of at least one liter of liquid (dedicated sample).

- 7.6.2.15** When retrieving composites, it is important that each individual parameter bottle accurately represent the composite. Therefore, use the following procedure:

- Note the presence of solids on the SRFs and COC.
- Swirl the composite to create a homogeneous mixture.
- Do not place gloved (or ungloved) hands inside the pickle jar.
- Fill each parameter bottle 1/3 of the way and then repeat until all parameter bottles are full.
- Use clean funnels if it is difficult to pour from the pickle jar to the parameter bottles.
- Place the caps of the parameter bottles with the inside (Teflon side) facing up on a clean surface (at many companies, the inside of the top of the autosampler will probably be the cleanest location).

- 7.6.2.16** Record all information about the composite sampling event on the Composite SRF. Sampling Associates are not required to indicate the specifics of the programming used to collect composite samples to the contact. Inform anyone who requests this information or any additional information, that you are not obliged to release it to him or her. If a company contact is insistent, suggest that they call the Regional Manager. Enforcement cases warrant that Sampling Associates take extreme care in what information is released.

- 7.6.2.17** When collecting samples from spigots, other devices or straight runs of pipe where solids may settle, purge the line before collecting grab samples or attaching suction tubing for composite samples.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

**7.6.2.18 Procedure for Collecting Cr<sup>+6</sup>, CN<sup>-</sup> (Total and Amenable), VOA, Acrolein and Pharmaceutical samples as Manual Composites**

Refer to the industrial file and make copies of the permit and the last monitoring report, as usual. The monitoring team must plan the most effective way to collect the parameters for each particular industry:

- If any of these parameters, Cr<sup>+6</sup>, CN<sup>-</sup>, VOA, pharmaceuticals or Acrolein samples are required, then use the manual composite method for collection. The manual composite method consists of 4 aliquots collected at least 15 minutes apart. In some cases, it may not be possible to collect all 4 sets due to low or intermittent flows. Under these circumstances, collect as many aliquots as possible.

**Some constraints to keep in mind:**

- Individual VOA, Acrolein and pharmaceutical samples are always collected as grab samples.
- All VOA samples must be collected on the same day that the composite sample is being collected for ABNs whenever results from both analyses are used to calculate Total Toxic Organics.
- A pH adjust test sample must always be collected for Acrolein for each set collected to determine the amount of hydrochloric acid needed to add to the sample for preservation (Refer to Acrolein SOP and/or flow chart).
- Cr<sup>+6</sup> samples and CN<sup>-</sup> samples that contain sulfides must be delivered to the lab on the same calendar day that the first aliquot was collected. Be sure to record the time that the first aliquot was taken in the space for composite start, so that the lab will know when the holding time started.

**MISCELLANEOUS NOTES**

- At Federal Category 414 industries remember that a flow figure is necessary for the exact period that the composite sample is collected. This requires you to record totalizer readings at the composite start and end times, or set up flow measuring equipment. Category 414 regulates VOA, ABN, CN<sup>-</sup>, Pb, and Zn. Collect your manual VOA composite within the time period of your flow measurement while the composite sampler is sampling for ABNs.

**7.6.2.19 CN<sup>-</sup> (Total and Amenable) Manual Composite Method**

Use the following procedure for collecting CN<sup>-</sup> samples:

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

- Affix a pre-printed, bar-coded label to a 1-L, unpreserved plastic bottle. Complete the label; write in the date and the sampler's initials.
- Collect all 250 mL sample aliquots in a clean sample vessel or directly into a 1-liter plastic sample bottle marked with graduation points every 250 mL.
- Collect the first 250-mL aliquot. If collected into a sample vessel: Pour the first 250-mL sample into the clean 1-liter plastic sample bottle. Record the time of collection, color, and odor, on the manual composite/grab SRF in the tabled section. Record the collection start time in the composite section on the SRF. Wait at least 15 minutes to collect a second 250-mL aliquot, and pour it into the 1-L bottle. Record the time of collection, color, and odor on the SRF in the tabled section.
- Collect a third aliquot as above, and record the time of collection, color and odor on the SRF in the tabled section. Wait 15 minutes to collect a fourth aliquot as above. Record time of collection, color, and odor in the tabled section and record the stop time on the SRF in the appropriate locations. Record the composite end time in the composite picked up line of the SRF. Measure the pH and temperature of the sample and record this information on the SRF at the bottom of the sheet along with the final color, odor, and composite end time on the SRF.

#### **7.6.2.20 Procedure for Cyanide Preservation**

- After collection of the cyanide manual composite sample, pour a small amount of sample (about 10 mL) from the 1-liter plastic bottle into a small plastic beaker.
- Test for sulfides with lead acetate (PbAc) paper by moistening the paper with pH 4 buffer solution and dipping the paper into the small plastic beaker. The paper will change color to a brownish appearance if sulfides are present. Note the presence or absence of sulfides in large letters on the COC.
- **Do not preserve with NaOH or treat for oxidizers if sulfides are present.**
- Samples that contain sulfides will need to be analyzed within 24 hours. Samples that do not contain sulfides must be checked for oxidizers (see below) and will have the full 14-day holding time. The laboratory must be informed that there are samples that contain sulfides ASAP, especially when collected on a Friday or on a day before a holiday.
- If sulfides are not present, test for oxidizers using KI paper moistened with pH 4 buffer in the same method that sulfides are tested. The paper will change to a brownish appearance if oxidizers are present. If oxidizers are present, add 1 powder pillow of ascorbic acid (0.03 g) to the sample. Retest the sample using a new strip of KI paper. Repeat addition of ascorbic acid pillows and testing with the KI strips until oxidizers are no longer present. Add 2 additional pillows of ascorbic acid to the sample. Note on the COC the quantity of ascorbic acid added to the sample to eliminate oxidizers.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

- Once oxidizers are eliminated and not detected, preserve the sample with NaOH and note it on the COC (see next bullet for NaOH preservation).
- Add the appropriate amount of NaOH to the sample in the 1-liter plastic sample bottle. Cap the sample bottle and invert it a few times to ensure the sample is well mixed in order to increase pH to 12 s.u. (or greater). It is best to start with 6-8 NaOH pellets by placing them into the NaOH container cap and then transferring the pellets to the sample bottle. Use additional NaOH pellets until the required pH is reached. Note the amount of NaOH pellets used on the COC.
- **NOTE: Recheck the pH for cyanide samples in Chelsea before sample storage and/or giving the samples to the lab courier. Ensure that the pH is equal to or >12 s.u.**

#### **7.6.2.21 Manual Grab/Composite Procedure for VOA Sampling**

Affix the pre-printed, bar-coded labels to the two trip blanks and eight VOA vials. Complete the labels; write in the date, time and sampler's initials on the appropriate labels. Use these same two trip blanks for all four sets of grab samples. **Refer to sections 7.6.2.20 - 7.6.2.26 for dechlorination and preservation procedures.**

1. While keeping the two trip blanks nearby, fill two of the VOA vials from the wastewater stream or sample vessel. Note the color and odor of the wastewater. Record the color, odor, and time of collection on the manual composite/grab sample record form in the VOA section.
2. Wait at least fifteen minutes to fill a set of two more VOA vials following the same procedure used to collect the first set.
3. Wait at least fifteen minutes to fill a set of two more VOA vials following the same procedure you used to collect the first and second set.
4. Wait at least fifteen minutes to collect a fourth and final set of grab samples following the same procedures used for the first three sets. Place all eight VOA vials and the two Trip Blanks back into the plastic bag and seal the bag with custody tape. Put the bag on ice in a cooler and complete the COC. Fill in "N/A" in the spaces on SRF for pH, temperature, color and odor (if CN<sup>-</sup> or Cr+6 are not collected). The composite start time will be the time of collection of the first aliquot. The composite end time and the composite pick up time will be the time of collection of the last aliquot. Also, use the time of collection of the last aliquot for the trip blanks.

**NOTES: Test for TCR and neutralize it each time VOAs are collected.**

Be sure to use the date and time of the last aliquot for the composite end time. Always try to collect all four VOA grab samples to make up the composite. However, if you

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

can't collect all four samples, submit what was collected. Note what prevented collection of all four sets on the SRF. For example, a batch discharge may not have not have discharged long enough to collect all 4 grab VOA sample sets.

#### **7.6.2.22 Manual Grab/Composite Procedure for Acrolein Sampling**

Affix the pre-printed, bar-coded labels to the two trip blanks and eight Acrolein vials. Complete the labels; write in the date, time and sampler's initials on the appropriate labels. Use these same two trip blanks for all four sets of grab samples. **See sections 7.6.2.21 and 7.6.2.25 for dechlorination and preservation procedures.**

**Note: See Appendix 7-A for reference to a more detailed Acrolein/Acrylonitrile SOP and sampling/preservation flow chart.**

1. While keeping the two trip blanks nearby, fill two 250 mL graduated beakers from the wastewater stream or sample vessel (main sample) with equal volumes to at least the 100-mL mark. Record the color and odor of the water and time of collection on the manual composite/grab sample record form in the VOA/Acrolein section.
2. Use one beaker as the test sample and the other as the sample for analysis.
3. Check the pH of the test sample with pH paper. Adjust the pH to 4-5 s.u. with HCL. Note the amount of acid used to preserve the test sample. Add the same amount of acid to the beaker containing the sample for analysis, but do not check the pH of this sample.
4. In conjunction with the pH adjustment, perform the TCR testing in accordance with section 7.6.2.25 on the main sample. Add the appropriate amount of sodium thiosulfate whenever warranted to each vial.
5. Fill each of the two vials from the beaker that contains the sample for analysis (the one that was not tested with pH paper). Be sure to fill the vials completely with a convex meniscus on top. It is important that air bubbles are not trapped in the vials.
6. Collect other manual composite samples as required.
7. Wait at least fifteen minutes and fill a second set of two more vials from the wastestream or the sample vessel following procedures 1-5 above. Record all of the data on the manual composite/grab sample record form and the vial labels in the same manner.
8. Wait at least fifteen minutes to collect a third set of grab samples following the same procedure you used to collect the first and second set.
9. Wait at least fifteen minutes and collect a fourth and final set of grab samples following the same procedures you used for the first three. Place all eight Acrolein vials and the two Trip Blanks back into the plastic bag and seal the bag with custody tape. Put the bag on ice in a cooler and complete the COC. Fill in "N/A" in the spaces for pH, temperature, color and odor (if  $\text{CN}^-$  or  $\text{Cr}^{+6}$  are not collected). The composite start time will be the time of collection the first aliquot. The composite end time and the composite pick up



Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

time will be the time of collection of the last aliquot. Also, use the time of collection of the last aliquot for the trip blanks.

**NOTES: Test for TCR and neutralize each time Acrolein samples are collected. The results can be used for VOAs and Acrolein if they are taken from the same main sample.**

Be sure to use the date and time of the last aliquot for the composite end time. Always try to collect all four VOA grab sample sets to make up the composite. However, if you can't collect all four, submit what was collected. Note what happened on the SRF the reason for not collecting four sets.

#### **7.6.2.23 Manual Composite Procedure for Pharmaceutical Samples**

The collection of pharmaceutical samples is virtually the same as that for VOAs. The parameters and EPA test Methods 1671, 1666 and 524 are mandated and found in the Federal Register, 40 CFR Part 439 for pharmaceutical manufacturers.

**For proper sampling, dechlorination, and preservation methods, refer to figure Appendix 7-B, for the flow chart describing these procedures.**

#### **7.6.2.24 Manual Composite Procedure for Cr<sup>+6</sup> (Hexavalent Chromium) Sampling**

Keep in mind that the Cr<sup>+6</sup> composite sample must be delivered to the laboratory on the same day that the composite started because of the 24 hour holding time. This will usually mean that the Cr<sup>+6</sup> composite sample is collected before the company's process day is finished.

Use the following procedure to collect a Cr<sup>+6</sup> manually:

- Affix the appropriate pre-printed, bar-coded label to a 500-mL unpreserved plastic bottle. Complete the label; write in the date and the sampler's initials.
- Collect the first 125-mL aliquot of sample from the wastewater stream with a sample vessel and pour it into the 500-mL plastic bottle.
- Record the initial time of collection, and date onto the composite SRF in the composite section and the time, color and odor of the wastewater stream in the VOA section if VOAs are also collected.
- Wait at least fifteen minutes to collect a second 125- mL aliquot. Record the time of collection and the color and odor of the waste stream in the VOA section of the SRF, if warranted.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

- Wait at least fifteen minutes to collect a third aliquot the same way you collected the first and second and record the collection time, color, and odor in the VOA section again, if warranted.
- Wait at least fifteen minutes to collect a fourth aliquot the same way you collected the first three and record the time, color and odor in the VOA section, if warranted. Record the manual composite end time and date in the composite section of the SRF. Measure the pH and temperature of the manual composite sample. Record the measurements onto the SRF at the bottom of the sheet. Record the final color and odor at the bottom of the SRF also.
- Seal the Cr<sup>+6</sup> sample bottle with custody tape, label it with the sample end time and place the sample on ice. Complete the COC and write N/A in the space for pH and temperature. Use the time of collection of the last aliquot for the composite end time and the pick up time.

**NOTES ON MANUAL COMPOSITES:**

- The holding time for Cr<sup>+6</sup> is 24 hours, measured from the time of the first grab aliquot. Deliver the sample to the laboratory courier on the same day of sample collection.
- Whenever VOA or Acrolein samples are collected with either CN or Cr<sup>+6</sup>, collect 1000 mL of sample into a clean beaker and pour the appropriate amount of sample into each parameter bottle for each sample interval set. However, TCR must be checked for each set of VOA or Acrolein samples.
- Always try to collect all four aliquots of the composite sample. However, if it is not feasible to collect all four samples, submit the sample(s) that were collected and note what happened on the sample record form.

**7.6.2.25 Priorities for Low Volume Composites**

If an autosampler has taken a representative sample but the sample volume is too low for all the composite parameters, use the following guidelines for parameter priority:

<u>Priority</u>	<u>Parameter</u>	<u>Minimum Volume</u>
1	Metals	100 mL
2	ABNs	1000 mL
3	MBAS	250 mL
4	TSS	100 mL
5	BOD	1000 mL
6	Pesticide/PCBs	500 mL

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

The above is a guideline only. Use your best judgment in cases where a particular parameter may have less priority based on the requirements of the project. When in doubt, ask the Regional Manager or the Sampling Coordinator.

**Note: See Fig. 7.4, Appendix 7A for a more comprehensive list of parameters and minimum volumes.**

#### **7.6.2.26 Dechlorination of Organics Samples**

EPA regulations, 40 CFR Part 136, state that samples analyzed for organics must be tested for chlorine and, if present, the sample must be dechlorinated prior to conducting the analysis for organic compounds.

The majority of organics samples collected by the TRAC sampling staff are Method 624 (purgeables), Method 625 acid and base/neutrals (ABN extractables), Method 608 (PCB/Pesticides), and Method 604 (Phenols).

Guidance for sample dechlorination for the different organics samples collected by Monitoring follows:

- **Group A** - organics include Methods 601 (purgeable halocarbons), 602 (purgeable aromatics), 603 (Acrolein and acrylonitrile) and 624 (purgeables).

If the sample contains TCR, 10 mg/40 mL of sodium thiosulfate is sufficient to dechlorinate up to 5 ppm Cl<sub>2</sub>. The Central Laboratory will supply VOA vials containing 10 mg of sodium thiosulfate.

- **Group B** - organics include Methods 604 (phenols), 605 (benzidines), 607 (nitrosamines), 610 (polynuclear aromatic hydrocarbons), 611 (haloethers) 613 (2,3,7,8-tetrachlorodibenzo-p-dioxin), and 625 (acids and base/neutrals).

If the sample contains TCR, 80 mg/L of sodium thiosulfate is required to dechlorinate this group of parameters up to 5 ppm Cl<sub>2</sub>. The Central Laboratory will supply 1-Liter amber bottles with 80 mg of sodium thiosulfate. After pouring the sample into the pre-preserved bottle, mix well.

- **Group C** - organics for Method 608 (Organochlorine PCBs and pesticides) only.

If the sample contains TCR, 80 mg/L of sodium thiosulfate is required to dechlorinate up to 5 ppm Cl<sub>2</sub>. The Central Laboratory will supply 1-Liter amber bottles with 80 mg of sodium thiosulfate. After pouring the sample into the pre-preserved bottle, mix well.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

### **7.6.2.27 Procedure for Dechlorinating Organics**

**GROUP A:** Methods 601, 602, 603, 624

**GROUP B:** Methods 604, 605, 607, 610, 611, 613, 625

**GROUP C:** Method 608

#### **GROUP A (VOA-624 and Acrolein-603) SAMPLES:**

A test for TCR must be performed **BEFORE EACH SET** of VOA or Acrolein vials is filled (i.e. VOA or Acrolein "A", VOA or Acrolein "B", VOA or Acrolein "C" VOA or Acrolein "D"). A single measurement for TCR can be used for each set of both VOA and Acrolein samples.

1. Collect a sample volume greater than 500 mL (1000 mL if other parameters are needed) for the test aliquot from the waste stream into a clean, glass beaker. Pour off 100 mL of this sample into a smaller beaker. Use this smaller sample to test for TCR. For VOA samples, the pH of the sample in the small glass beaker must be adjusted between pH 5.0 and 9.0 following the procedure in "C" of the Notes listed in section 7.6.2.25 before checking for chlorine residuals. The pH can be checked with pH test strips.
2. After checking and adjusting the pH, conduct the chlorine test as specified in section 7.6.2.25 and then go to step 3.
3. If TCR is not present, fill two unpreserved VOA vials with sample from the larger beaker and process as usual. Fill the two Acrolein vials according to section 7.6.2.21. Also, if it is necessary to collect  $\text{CN}^-$  and/or  $\text{Cr}^{+6}$ , pour off 125 mL into the  $\text{Cr}^{+6}$  container and 250 mL into the  $\text{CN}^-$  container for the first aliquot from the large beaker. Use the same collection time on the composite/grab SRF. Note that chlorine measured 0 mg/L on the SRF and the COC for the VOA and/or Acrolein samples.
4. If TCR is present, go to step #5.
5. If the chlorine reading is above 0.2 but below 5.0 mg/L measured with the DR/890, fill two VOA and/or Acrolein vials pre-preserved with 10 mg of sodium thiosulfate from the larger beaker. Also collect the  $\text{Cr}^{+6}$  and  $\text{CN}^-$  samples, if applicable, using the same collection time on the composite/grab SRF. Record dechlorination of the VOA and/or Acrolein samples with 10 mg of sodium thiosulfate on the SRF and COC forms.
6. If the chlorine test indicates that chlorine is greater than 5.0 mg/L measured with the DR/890, add 10 mg of sodium thiosulfate to a clean, glass beaker. Fill the beaker with 40 mL of sample from the original test aliquot (the large beaker) taken in step #1 and mix gently (obtain 10 mg of sodium thiosulfate from a pre-preserved VOA vial). Reuse the VOA vial again if rinsed three times in the wastewater stream. Use this 40-mL sample as the test aliquot to conduct the chlorine test as specified in section 7.6.2.25.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

**NOTE: - The same glass beaker can be used for all four sets of VOA and/or Acrolein samples: rinse it three times in the wastewater stream before each 40 mL test aliquot is taken, if possible. If it is not possible, rinse the beaker with DI water.**

7. If the chlorine reading is 0 mg/L after this test, fill two pre-preserved VOA and/or Acrolein vials with sample from the large beaker after adjusting the pH. A TCR reading of 0 mg/L indicates the chlorine concentration was equal to or less than 5 ppm when the 10 mg of sodium thiosulfate was added to the 40 mL sample in step #6). Also, collect the Cr<sup>+6</sup> and CN<sup>-</sup> samples, if applicable, using the same collection time on the composite/grab SRF. Note the amount of sodium thiosulfate used to dechlorinate the VOA and/or Acrolein samples on the SRF and the COC.
8. If the TCR reading is between zero and 3.5 mg/L measured with the DR/100 (5.0 mg/L measured with the DR/890) after adding the 10 mg of sodium thiosulfate in step #6, fill two pre-preserved VOA and/or Acrolein vials with 10 mg of sodium thiosulfate for a total of 20 mg of sodium thiosulfate in each vial and fill with sample from the large beaker. To prevent the sodium thiosulfate from being flushed out of the pre-preserved vials, do not submerge them into the wastewater stream.
9. If chlorine is greater than 5.0 mg/L measured with the DR/890, repeat the dechlorination procedure until it is between 0 and 5.0 mg/L measured with the DR/890 (at this point, add an additional 10 mg of sodium thiosulfate to fully dechlorinate the sample). Record the amount of preservative used on the SRF and the COC. If applicable, collect the Cr<sup>+6</sup> and CN<sup>-</sup> samples at the same time.
10. Since the chlorine test using the HACH meter is a colorimetric test, it can only be used on clear samples. Therefore, if the sample contains any color to it, use pre-preserved bottles for both VOA and/or Acrolein samples (10 mg). Note this information on the SRF and the COC.
11. After adding the DPD powder pillow contents to the 10 mL test aliquot sample for the chlorine test, if the color of the sample is reddish initially and clears up after the container has been shaken for 20 seconds, this indicates that the amount of chlorine in the sample is excessive and has overwhelmed the DPD powder. In this case, add 10 mg of sodium thiosulfate to a 40-mL sample from the original test aliquot and retest for chlorine. Follow this procedure as many times as necessary to fully dechlorinate the sample to zero chlorine.
12. Acrolein vials must be filled from the pH-adjusted sample in the beaker set as the sample for analysis.

**GROUP B (ABN-625) & GROUP C (PCB/PEST-608) SAMPLES:**

A test for TCR must be performed on Group B (most commonly, ABN) and Group C (most commonly, PCB/PEST) parameters as follows:

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

1. Pour off a 1000-mL volume of test aliquot into a clean, unpreserved 1-L amber bottle from the composite sample.
2. **For Group B and Group C (PCB/Pest-608), the following procedures apply with the exception of adjusting the pH for Method 608 (the pH of the sample must be adjusted between pH 5 and 9, following the procedure in "C" of the Notes listed in sections 7.6.2.24 and 7.6.2.25.**

The pH can be checked with pH test strips.

3. After checking and, if necessary, adjusting the pH, conduct the chlorine test as specified in section 7.6.2.25.
4. If the sample is free of chlorine (colorimeter reads 0 mg/L), process the ABNs and/or PCB/PEST parameter bottle(s) as usual. Collect the sample into sample bottles that do not contain sodium thiosulfate.
5. If the sample contains TCR above zero or below 5.0 mg/L measured with the DR/890, pour the samples into amber bottles pre-preserved with 80 mg of sodium thiosulfate and mix gently. Note the amount of sodium thiosulfate added in the space provided on the sample record form and the chain-of-custody.
6. If the chlorine test indicates chlorine reading in excess of 5.0 mg/L measured with the DR/890, add 80 mg of sodium thiosulfate to the original 1-L test aliquot sample and mix gently. The sodium thiosulfate can be taken from pre-preserved 1-L amber bottles or measured by using the spatula provided by the Central Laboratory. Filling the cupped portion of the spatula level approximates 80 mg.
7. Conduct another TCR test as specified in section 7.6.2.25 on a small sample poured off from the same 1-L sample. Keep following this procedure until the chlorine reading is between 0 and 5.0 mg/L measured with the DR/890. At this point, add an additional 80 mg of sodium thiosulfate to fully dechlorinate the sample. Note the amount and type of dechlorinating agent added in the space provided on both the SRF and the COC.
8. After the amount of sodium thiosulfate needed for dechlorination has been determined, add the same amount to the other 1-L ABN sample.
9. Top off the original 1-L sample (the one used to pour off the small chlorine test aliquots) with composite sample from the pickle jar.
10. Since the chlorine test using the HACH meter is a colorimetric test, it can only be used on clear samples. Therefore, if the sample has any color to it, use pre-preserved bottles for ABNs and PCB/Pest samples (80 mg in each 1-L bottle). Note this information on the SRF and the COC.
11. After adding the DPD pillow powder to the mixing vessel for the chlorine test, if the color of the sample is reddish initially and clears up after the container has been shaken for 20 seconds, this indicates that the amount of chlorine in the sample is excessive and has overwhelmed the DPD powder. In this case, add 80 mg/L of sodium thiosulfate to the 1000 mL sample and retest for chlorine.

**NOTES:**

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

- Parameters not requiring a test for TCR should be poured off from the composite sample first.
- If there is not enough volume for all parameters, take only one for the ABN sample and pour it into a pre-preserved amber bottle.
- Use the "Minimum Volume Required" list for all parameters if the sample shortage does not allow enough test aliquots for sample chlorination test procedures. In this case, pour all organics samples into pre-preserved bottles as required.

#### 7.6.2.28

##### **NOTES: Total Chlorine**

- A. It is not necessary for all the particles to dissolve to obtain an accurate reading.
- B. If the sample temporarily turns yellow when adding the DPD Total Chlorine Reagent or reads above the highest scale division, the TCR concentration is too high for the meter scale and a sample dilution is needed. A slight loss of chlorine may result from the dilution.
- C. Samples should have a pH between 6 and 7. If necessary, an appropriate amount of an acid or base that does not contain ammonium ions can be used (for example: 1N Sulfuric Acid Standard Solution or 1N Sodium Hydroxide). Test the sample immediately after adding the acid or base. The pH can be checked with pH test strips.

#### 7.6.2.29 Hach DR/890 Colorimeter

A Hach DR/890 Colorimeter can be used for checking residual chlorine in the field. The EPA approved this field test method. It can also be used for testing free chlorine in the field. For the organics methods listed above, the field test is for residual or total chlorine in the 0-2 mg/L for the low range and 0-5 mg/L for the high range using the following procedure:

##### **Total Chlorine Residual:** Program #9, Low Range 0-2 mg/L

1. Turn the unit on by pressing the **[ENTER]** button.
2. Enter the stored program number for total chlorine residual low range by pressing the **[PRGM (7)]** button. The display will show: **PRGM ?**.
3. Press the **[9 (PRINT)]** button, then press the **[ENTER]** button. The display will show **mg/L, CL<sub>2</sub>**, and the **ZERO** icons.
4. Fill a DR/890 sample cell with 10 ml of sample (this is the sample blank in which you will zero the instrument with).
5. Place the sample cell with the blank into the cell holder and tightly cover the sample cell with the instrument cap.
6. Press the **[ZERO (0)]** button. The display will show **0.00 mg/L CL<sub>2</sub>**.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

7. Fill a second sample cell to the 10 mL mark with the sample, and add the contents of 1 DPD Total Chlorine Powder Pillow (**LOW** range: M00110F10) to the sample cell. Cap the cell and swirl it vigorously to dissolve the powder (it is not necessary to dissolve the entire reagent).
8. Press the [TIMER(CE)] button and then the [ENTER] button. Wait for the timer to elapse for the 3 minute reaction.
9. After the unit beeps (the 3 minute reaction has elapsed), place the prepared sample cell into the cell holder and cap tightly with the instrument cap.
10. Press the [READ] button and the result will be shown in the liquid crystal display.

**Total Chlorine Residual:** Program #8, High Range 0-5 mg/L

1. Turn the unit on by pressing the [ENTER] button.
2. Enter the stored program number for total chlorine residual high range by pressing the [PRGM (7)] button. The display will show: **PRGM ?**
3. Press the [8(SETUP)] button, then press the [ENTER] button. The display will show **mg/L, CL<sub>2</sub>**, and the **ZERO** icons.
4. Fill two DR/890 sample cells with 10 mL of sample.
5. Add the contents of one DPD Total Chlorine Powder Pillow (**HIGH** range: M00110F25) to one sample cell (the prepared sample), cap the cell and swirl vigorously to dissolve the reagent.
6. Press the [TIMER (CE)] button and then the [ENTER] button. Wait for the timer to elapse for the 2 minute reaction.
7. After the unit beeps, add deionized water to each cell to the 25-mL mark. Cap each sample cell and invert twice to mix.
8. Place the diluted sample cell with no reagent (the blank) into the cell holder and tightly cover the sample cell with the instrument cap.
9. Press the [ZERO (0)] button. The display will show **0.00 mg/L CL<sub>2</sub>**.
10. After the unit is zeroed, place the prepared sample into the cell holder and cap tightly with the unit's cover. Press the [READ] button and the result will be shown in the liquid crystal display.

**Notes:**

1. A blank must be run for each sample collected.
2. Before re-using the sample cells, they and the caps must be rinsed 3 times in the effluent waste stream. Ensure the caps are not switched between cells, and ensure the one used for the blank is not used for the sample.

**7.6.3 Automatic Sampler Calibration Frequency**

The sample volume must be calibrated at each set up.



**7.6.4 Automatic Sampler Calibration Procedure**

- 7.6.4.1 Measure the actual volume of sample pumped for one sample using a 500 or 1000 mL graduated cylinder.
- 7.6.4.2 Adjust the pump time per sample to account for any differences in volume actually pumped from the volume entered into the sampler program.
- 7.6.4.3 Select auto-calibrate mode and check calibration with a graduated cylinder.

**7.6.5 Cleaning Automatic Samplers**

Clean the units only with soapy warm water. Never use a solvent to clean sampling equipment to eliminate the potential of contaminating samples. Avoid getting water into the electronics of the unit, which could cause mechanical failures. Discard pump tubing and sample tubing after each sampling event. Send strainers and sample bottles (pickle jars) for cleaning to the Central Laboratory.

**7.6.6 Troubleshooting Automatic Samplers**

- 7.6.6.1 Each unit's manual has a troubleshooting section with solutions to common problems. Become familiar with these sections so you can recognize a problem and correct it as quickly as possible.
- 7.6.6.2 Avoid opening the electronics in the field. Always take an extra autosampler as a contingency.
- 7.6.6.3 If there is no power getting to the unit, first check all connections along the power cord. If DC power is being used, try another fully charged battery. If the power source is fine, check the fuse. The fuse will be located either on top of the unit or inside the unit. If inside the unit, the fuse is accessed via the desiccant plug. Only if these steps fail should you open the electronics and check for loose wires or connections.
- 7.6.6.4 If there is poor suction or erratic volumes are collected, check to see if the strainer is clogged. Make sure the vertical lift is not over 26 ft. (this will burn out the pump). Tighten all hose clamps along the suction line. Check for cracks in the pump tubing.
- 7.6.6.5 The best tool in troubleshooting is information. Keep a manual for each unit on hand and know where to find the information in it. Each manufacturer provides a telephone number for technical services. If you cannot identify a problem, they will help you.

### **7.6.7 ISCO Automatic Samplers**

The Monitoring section uses several types of ISCO autosamplers. Make sure the unit being used is suitable for the sampling desired. When in doubt, consult the manufacturers' manuals located on the bookshelves in the Chelsea work area. In general, check the following for fieldwork:

- Check inside the unit to see that you have the correct positioning insert.
- Calibrate the volume each time you change the intake tubing length.
- Check the desiccant indicator before each use. If you need to change it, follow the manufacturer recommendations and instructions located in the manuals.
- Know the parameters for the sampling event before you start programming the unit (# of samples, start time, stop time, sample volume).

### **7.6.8 List of Tools and Equipment for Composite Sampling**

#### **7.6.8.1 Sampler Set-up**

- Pliers
- Knife
- Screwdriver
- Hose clamps
- Nut driver
- Tubing cutter

#### **7.6.8.2 Sampler Preparation**

- Pliers
- Knife
- Screwdriver (Phillips and regular)
- Allen wrenches
- Nut driver

#### **7.6.8.3 Troubleshooting**

- Pliers
- Multimeter (volt/amp meter)
- Wrenches
- Screwdriver (Phillips and regular)
- Soldering iron
- Crimper
- Operations Manual

#### **7.6.8.4 Equipment for Composite Sampling**

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

- Strainer (stored in a plastic bag)
- Teflon tubing (stored in plastic bag)
- Hose clamps
- Extension cords and adapters
- Power cord/Power supply
- Sample containers
- Pump tubing
- Ice
- Nut driver
- Screwdriver
- Extra batteries

## 7.7 pH

### 7.7.1 pH Calibration Frequency

Calibrate the pH meter using pH 4, 7, and 10 buffers in the TRAC Chelsea work area before going in the field and at each industry. Three point calibrations should record (time, date, and analyst initials) in the pH meter logbook stored with each meter. If a pH violation is noted ( $\text{pH} < 5.5$  or  $\text{pH} > 12.0$  s.u.) then the pH buffer closest to the measured pH should be read to verify the reading and recorded in the pH record log book. For pH violations greater than 12, each van has pH 12.45 buffer, which should be used to verify calibration and the violation. The violation must also be recorded in the pH record logbook.

When calibrating the pH meter, monitor the electrode response slope reading in the pH logbook. A properly working pH electrode should give a slope reading of 98% to 99.5%. A slope of 94% or less possibly indicates a failing electrode. At this point, the electrode should be monitored closely for further deterioration. If possible, replace or recondition the electrode. **Do not use an electrode with a pH slope of 90% or less.**

### 7.7.2 pH Calibration Procedures

**pH Beckman 200/255 Series pH meters:**

To perform a three-point calibration:

- 7.7.2.1 Turn the meter ON by depressing the "Φ" key.
- 7.7.2.2 Depress the "CAL" key to enter the calibration mode.
- 7.7.2.3 Depress the "CLEAR" key to remove the existing calibration.
- 7.7.2.4 To confirm the removal of the calibration, depress "CLEAR" key again.
- 7.7.2.5 Rinse the pH electrode with deionized water and blot gently with a Kimwipe.
- 7.7.2.6 Immerse the pH electrode into pH 4 buffer solution, stir briefly, and depress the "READ" key.
- 7.7.2.7 After the "Auto-Eye" stops flashing, the meter will display the pH of the first standard buffer.
- 7.7.2.8 Rinse the pH electrode with deionized water and blot gently with a Kimwipe.
- 7.7.2.9 Immerse the pH electrode into pH 7 buffer solution, stir briefly, and depress the "READ" key.
- 7.7.2.10 After the "Auto-Eye" stops flashing, the meter will display the pH of the second standard buffer.
- 7.7.2.11 Rinse the pH electrode with deionized water and blot gently with a Kimwipe.
- 7.7.2.12 Immerse the pH electrode into pH 10 buffer solution, stir briefly, and depress the "READ" key.
- 7.7.2.13 Depress the "EXIT" key to complete the calibration and to exit the calibration mode.
- 7.7.2.14 Record all pH readings and temperatures along with time, date, and analyst's initials in the pH meter logbook (Figure 7.11, Appendix 7A) stored with the meter.

### 7.7.3 pH Measurement

- 7.7.3.1 All Sampling Associates are responsible for reading the manufacturer's operations manual that accompanies the pH meters. This is documented on the Sampling Associate Training/Field Audit Checklist.
- 7.7.3.2 The pH measurement is performed on an aliquot of sample in a clean, disposable screw-top bottle or disposable plastic beaker.
- 7.7.3.3 The pH electrode is rinsed with deionized water and gently blotted with a KimWipe after each pH measurement.
- 7.7.3.4 pH measurements are determined for unpreserved grab samples, manual composite samples (no measurement if VOAs are the only parameter) and the composite samples (only record composite pH on the SRF and not COC).
- 7.7.3.5 A pH violation is indicated by a pH < 5.5 or > 12 s.u. of the unpreserved grab sample.
- 7.7.3.6 The pH of preserved samples must be measured with pH test strips and this measurement must be recorded on the SRFs and the COCs forms indicating proper preservation according to Figure 7.4, Appendix 7A. Any reactions that occur as a result of preservation (color change, precipitate

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

formation, fuming etc.) must be noted on the SRF and the COC. The amount of preservative must also be recorded on both documents.

7.7.3.7 The pH of the unpreserved grab sample is recorded on the Grab SRF and the COC for the sample.

7.7.3.8 After performing the 3-point calibration and the pH of the unpreserved grab sample shows a violation of the local limits (pH < 5.5 or pH > 12 s.u.), check the meter with a pH standard buffer closest to the pH violation. If the measurement of the buffer is not within range, recalibrate the instrument and recheck the pH of the sample and the buffer closest to the violation. Record all readings in the pH logbook and on the SRF.

7.7.3.9 Violations are recorded in the pH logbook using the Industry permit #, date, pH violation, and calibration information.

#### 7.7.4 **pH Troubleshooting**

7.7.4.1 If the response time for a pH reading is delayed or if the reading is drifting or unstable, then the junction of the probe may be clogged or the bulb may be coated with a particular substance that will not allow ions to pass readily (i.e. oil, soap). The bulb should be immersed in an enzyme cleaning solution for 5 minutes, rinsed with deionized water, and immersed in the pH 4.0 buffer solution for 5-10 minutes to rejuvenate the electrode. Recalibrate the meter after cleaning the electrode.

7.7.4.2 The bulb must be rinsed with deionized water between readings and stored in storage solution at all times when not in use. Do not store pH electrodes with the tip immersed in tap water, deionized water, cleaning solution, or other seemingly 'obvious' solutions. These solutions all have properties, which tend to clog the reference junction and contaminate filling solution. If there is no storage solution available, pH 4.0 buffer solution may be used for short periods of time.

7.7.4.3 If the meter will not calibrate (i.e. meter shows pH 7 for all buffers), then the problem may be electrical. Alternatively, the probe may have a cracked bulb or body. The probe connecting wire may need to be replaced or the meter itself may need service.

7.7.4.6 An error message of ?/PROBE SYMBOL on the meter display usually indicates that the buffer(s) have been contaminated. **Buffer solutions must be changed in the morning on a daily basis.**

7.7.4.7 Note: **Do not use an electrode with a pH slope of 90% or less as noted in section 7.7.1, above.**

#### 7.7.5 **Continuous pH Monitoring**

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

Continuous pH meter modules are attached to ISCO Model 6700 samplers and can be set up at the request of the Industrial Coordinator for compliance and permit issues. On the initial visit, and each subsequent visit, a pH strip chart is downloaded from the pH data logger and submitted with the monitoring report for the industry.

**7.8 Temperature**

**7.8.1** Take temperature measurements with the pH electrode and built in ATC probe attached to the pH meter.

**7.8.2** The temperature of the composite sample is the same as the storage temperature of the composite sample container. Therefore, it is important to take this measurement immediately by pouring a small amount out to take the temperature.

**7.9 Observations**

Any identifying characteristics, such as color, odor and solids content, must be noted on the SRF and COC.

**7.10 Flow**

**7.10.1 Introduction**

Measure flow with flow meters at all industries that require a primary measuring device. These devices are required to be installed at industries that are Significant Industrial Users (no flow minimum) and most non-Significant Industrial Users with flows  $\geq 300$  gpd. If a flow meter

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

cannot be set up, it will be determined jointly by the Regional Manager, Industrial Coordinator and the Compliance Coordinator whether the discharge is significant enough to require the industry to modify the sampling site to facilitate flow measurement.

Collection of this data will enhance the database as well as verify existing information, such as daily flow and time of peak flow. It will also allow flow proportional samples to be collected, which provide a more accurate representation of the industrial discharge.

**NOTE: Use this section as an outline in setting up the meters and handling different situations. The Flow Meter Manual and ISCO Open Channel Flow Measurement Handbook should be consulted for more specific information. The reading of these two manuals is documented on the Training Checklist.**

#### **7.10.2 General Inspection of Flowmeters**

Use the following guidelines prior to using the flowmeter equipment in the field:

- 7.10.2.1** Visual Inspection. Look at the front of the flowmeter and check the casing for damage.
- 7.10.2.2** On the ISCO models, check the strip chart printer roll and make certain there is sufficient chart paper and ink ribbon for the length of the planned monitoring event.
- 7.10.2.3** Change the external and internal desiccant cartridge(s) when required.
- 7.10.2.4** Inspect the receptacles on the flow meter. Make certain they are intact so that any necessary connections can be properly secured.
- 7.10.2.5** Choose the power source; a DC 12-volt battery or if AC power is desired, a 120 VAC power pack is available. Inspect the ends of the power cords for breakage. If broken, it will be difficult to secure a connection to the receptacle.
- 7.10.2.6** Battery Connection. To connect the DC or AC power source to the flow meter, screw on the two-pin connector in a clockwise direction. Check all batteries for a full charge before using in the field.

#### **7.10.3 Bubble Tubes and Rods**

The flowmeter is provided with a 25' flexible PVC bubble tube and either a 2' or 4' foot flexible stainless steel bubble rod. The bubble tube connects to the barbed fitting on the flow meter casing and is routed to the measurement point. The bubble tube should be positioned securely at the primary measuring device. It is usually much easier to secure after it has been fit on the stainless steel bubble rod.

#### **7.10.4 Connection to Sampler**

The flowmeter will transmit a signal to an automatic sampler to collect samples in proportion to flow rate. To interface with the autosampler, connect the flow meter to the autosampler using the six-pin cable and secure each end with a clockwise turn.

#### **7.10.5 Programming the ISCO Flowmeter**

The bubble and ultrasonic type ISCO flow meters have LCDs that prompt the user to program the flowmeter in a step by step method. The keypad is used to enter the program data as requested by the prompts. The ISCO flowmeters print out reports based on the data requirements programmed in by the user. Refer to the ISCO manufacturer manual for programming examples and report generation. Additional flow meter programming information can be found in the ISCO Flowmeter Handbook Manual.

**7.10.5.1** Maintenance for flowmeters is minimal. Clean the exterior case with soapy water. Ensure that the cover is latched and all connectors are tightly capped.

**7.10.5.2** The case seal should be kept clean from dirt and grease by periodically inspecting and cleaning the seal with a damp cloth.

**7.10.5.3** Replace desiccant as needed. For the ISCO models, there is an internal desiccant case (inside the front door) that is attached with two thumbscrews. The canister has an inspection window to allow the user to see the condition of the desiccant. If the desiccant is blue, it is in good condition. If the desiccant is pink, it needs to be regenerated. Place the canister in the drying oven until the desiccant turns blue.

**7.10.5.4** The bubbler type flow meters also have exterior desiccant tubes that dry the incoming air drawn into the flow meter. This air is used to generate the air bubbles. The same principle applies with the desiccant as described above (the desiccant needs to be regenerated when it is pink). However, since the desiccant container is plastic, do not place it in the drying oven. Empty the desiccant into a glass beaker, which can then be placed into the drying oven.

**7.10.5.5** Periodically replace the cotton balls in the desiccant tubes. The cotton balls prevent desiccant dust from getting into the air pump system and causing operational problems.

**7.10.5.6** For the ISCO units, information on changing plotter paper and the ink ribbon is detailed in the ISCO manual. The user should become familiar with this procedure.

#### **7.10.6 Primary Measuring Devices**



Both the setting of the flowmeter and the positioning of the bubble tube are dependent on the primary measuring device being employed. The specific procedures for each device are discussed in the following sections.

**7.10.6.1 Parshall Flume:** This is the most common type of flume and it is relatively easy to utilize.

- Parshall flume sizes are designated by the throat width. This can be measured by laying a flat edge ruler across the top of the flume at the throat and measuring distance between the inner walls.
- To measure the liquid level (H) a flat edged ruler may be used. The measurement should be taken in tenths of ft. The measurement point is at "H" (Figure 7.13, Appendix 7A). (Some manufacturers have a permanent scale marked on the inside wall of the flume at the point where the bubble should be positioned.)
- The bubble rod connected to the bubble tube is positioned at the measurement point either inside the flume or inside the stilling well if there is one fixed to the flume. The rod is positioned in such a way that the air bubbles are allowed to flow freely downstream if it's inserted into the flume. If the rod is inserted into the stilling well, the rod is inserted below the opening between the stilling well and the flume so that the rod end is submerged, even under zero flow
- The rod can be secured to the wall of the flume by using a C-clamp, duct tape, wire ties, etc.
- The approaching flow should be laminar and relatively free of turbulence, eddies, and waves for accurate measurements.

#### **7.10.7 Weirs**

**7.10.7.1 V-Notch Sharp Crested Weir** (Figure 7.15, Appendix 7A)

- This design is particularly suited for low flows.
- The angle of the notch most commonly used is 90°, although V-notch weirs with angles of 60°, 45°, 30°, and 22 1/2° are also used.
- The best location for the bubble rod is a little below the base of the V-notch, but located upstream from the V-notch in the center of the weir box at a distance approximately 3 - 4 times the maximum expected head of flow. This is also the best location for measuring the height of head flowing through the V-notch. The rod should be positioned vertically and perpendicular to the flow, with the end submerged below the V-notch base.
- The easiest way to secure the bubble tube is by placing a piece of wood, etc. across the walls of the tank and suspending the bubble tube or rod from that material. Duct tape, C-clamps, or wire ties may be used to accomplish this.
- To measure the liquid level: measure the distance from the bottom of the weir box to the base of the V-notch in tenths of feet (x). Then measure the distance from the bottom of

the weir box to the liquid level inside the weir box (y) at the bubble rod location in tenths of feet. Subtract x from y to calculate the liquid level of water (h) flowing through the V-notch weir. The best location to measure is in the center of the weir box.

#### **7.10.7.2 Rectangular Weir with End Contractions**

(Figure 7.15, Appendix 7A)

- This weir is able to measure much higher flows than the V-notch weir. The discharge equation is more complicated than for other types of weirs.
- The liquid level (H) is measured in the same way as in the V-notch weir.
- The positioning of the bubble tube is the same as that of the V-notch weir.

#### **7.10.7.3 Rectangular Weir without End Contractions**

(Figure 7.16, Appendix 7A)

- Able to measure same range of flows as contracted rectangular weir, but has simpler discharge equation.
- The liquid level (H) is measured in the same way as in the V-notch weir.
- The positioning and securing of the bubble tube is similar to that of the V-notch weir.

**7.10.7.4** Other types of primary flow measuring devices such as Palmer-Bowlus flumes, H Flumes and trapezoidal flumes may be encountered in the field. Check the flow meter manuals and/or the ISCO Open Channel Flow Measurement Handbook for details on programming.

### **7.10.8 Troubleshooting Flow Meters**

**7.10.8.1** Flowmeter will not zero. This may be caused by a kink in the bubble tube restricting airflow or buildup of solids at the end of the bubble tube. Check the entire length of tubing and clear away any solids around end of tubing. Also it is a good idea to stick a pin in the end of the bubble tube to make sure it is free of debris. If the flow meter still does not zero, replace it.

**7.10.8.2** If bubble tube cannot be secured. This problem may arise quite often. For a Parshall Flume, a C-clamp might be best for securing the bubble tube; for a V-notch Weir, a small 2x4 and some duct tape might work.

**7.10.8.3** If a flowmeter cannot be utilized. At many industries it is impossible to use a flowmeter due to the set up of the treatment area. In this case assess the situation and note what changes can be made so that a flowmeter may be used in future visits. This must be noted on the Monitoring Report submitted to the Regional Manager.

**7.10.8.4** Power failure. A blown fuse, poor battery connection, or a dead battery may cause this. Unscrew fuse knob and inspect the fuse. Check battery cable connection to the flowmeter DC receptacle and make sure it is secure. If none of the above corrects the problem, disconnect the battery and replace it.

**7.10.9 Instantaneous Flow Measurement using ISCO Flow Poke**

The instruction manual for the ISCO Flow Poke flow meter is located on the bookshelves in the TRAC Chelsea work area. Training in this procedure is documented on the Training Checklist. Chapter 3 of the Flow Poke manual provides detailed step-by-step operating procedures for this equipment.

**7.11 Specific Procedures for CSO Monitoring**

Refer to latest Revised CSO SOP located on the "G" drive "Chelsea1(G)/CSO Sampling/SOP CSO NPDES Rev.##". The CSO SOP changes frequently, so make sure you have the latest version and are familiar with the most recent changes.

**7.12 Specific Procedures for POTW Monitoring**

**7.12.1 Clinton POTW NPDES Sampling Schedule**

**7.12.1.1** POTW sampling is required by the NPDES permit to take place four times per year for the Clinton POTW. The EPA permitted monitoring schedule for the Clinton POTW includes wet chemistry and biotoxicity samples as follows:

- Sunday:** 24-hr. composite is set up.
- Monday:** Composites picked up, grabs collected. Composites reset Tuesday - Wednesday
- Wednesday:** Composites picked up, grabs collected, Composites reset Thursday - Friday
- Friday:** Composites picked up, grabs collected.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

For Clinton POTW: Biototoxicity and Wet Chemistry are sampled quarterly (March, June, September and December).

**NOTE: The Central Laboratory staff collects all Deer Island NPDES samples.**

**7.12.1.2** If the composite sampler does not collect enough sample volume for NPDES POTW sampling for Clinton, reset and use the following schedules:

1. First day sample fails, reset to run:  
**Monday - Tuesday**  
**Tuesday - Wednesday**  
**Wednesday - Friday**
  2. Second day sample fails, reset to run:  
**Wednesday - Thursday**  
**Thursday - Friday**
  3. Third day sample fails, reset to run:  
**Friday - Saturday**
- Notification procedure for weekday NPDES sample failures - Sampling Associates must notify the Sampling Coordinator or the Regional Manager, who in turn, will notify ENQUAD (NPDES group) for further instructions.
  - Notification for weekend NPDES sample failures - Sampling Associates must page the on-call ENQUAD manager at (617) 403-5803 and the on-call Central Laboratory manager (617) 403-5788 to notify them of the sample failure. On Monday morning, the Sampling Associates must notify the Sampling Coordinator or Regional Manager.
  - Minimum volumes required:  
**Day 1 & 2** - 3.5 L (each day) for Wet Chemistry, minimum of 4 gallons for Biototoxicity;  
**Day 3** - 3.5 L for Wet Chemistry, minimum of 4 gallons for Biototoxicity.

The sample for Metals (only) can be taken from the Biototoxicity sample if not enough sample is in the wet chemistry sampler. Samples for Organics must be taken from a glass compositing container. Sample from the Local Limits glass container or NPDES chemistry can also be used to make up volume for the Biototoxicity sample. Under no

circumstances shall Biototoxicity sample be substituted for or added to the Chemistry sample to get enough volume for organics.

**7.12.1.3** The Clinton POTW has recently undergone an intense monitoring program to evaluate the local limits as set forth in 360 CMR 10.024. Local Limits sampling will continue to be done one day every other month at 4 sites:

1. headworks (influent)
2. after primary treatment
3. after secondary treatment
4. isolated source of domestic wastewater.

Clinton Local Limits:

Unless otherwise instructed, TRAC staff samples all Local Limits locations every other month.

**7.12.1.4 Clinton Weekend Sampling**

The Clinton POTW used to have a lock box that contained the key for accessing the facility after normal work hours and on weekends. However, they now have someone staffing the facility on weekends. Dial the main number (978) 368-6144 to contact the operator on duty whenever you are assigned to setup on a Sunday for NPDES.

**7.12.1.5 Clinton Landfill and Leachate Sampling**

TRAC staff samples 7 monitoring wells and 2 landfill leachate discharge sites twice a year, once in December and once in May.

**7.12.2 Sampling Locations**

The monitoring staff is responsible for sampling at one facility for NPDES requirements:

- 1) Clinton - The 5 sites for the bi-monthly local limits and the quarterly NPDES sites are described in detail in Figure 7.19, Appendix 7A.

**7.12.3 Sample Parameters for Clinton POTW**

**7.12.3.1 Clinton**

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

The parameters for the Clinton sampling are listed in Figure 7.20, Appendix 7A.

#### **7.12.4 Equipment**

**7.12.4.1 Bottles (NPDES)** The list of required bottles and preservatives for Clinton sampling is given in Figure 7.20, Appendix 7A.

**7.12.4.2 Clinton Equipment** The following equipment is needed for Clinton NPDES monitoring:

- sample strainers
- extension cords are required.
- 5 Pre-printed Chains of Custody forms
- 3 automatic samplers
- 6 pickle jars
- 3 5-gallon buckets
- 2 ISCO power packs
- 3 extension cords
- 3 sets of bottles (note size of bottles used for routine monitoring)
- 40 ft. Teflon tubing
- 3 strainers
- Misc. equipment (funnels, tape, tools, etc)
- Ice and cooler

**7.12.4.3** Extra equipment (batteries for backup, buckets, etc.) should be brought to the POTW on pick-up days. If the battery fails, both the toxicity and the chemistry samplers need to be reset and the extra equipment must be available.

#### **7.12.5 Background and Residential Local Limits Sampling**

The Monitoring section also samples four metro residential sites, four metro background/commercial sites, and three I&I sites for pollutant loading calculations at the Deer Island POTW. The background sites are a combination of residential discharges and commercial discharges. Central Lab staff samples the Deer Island POTW.

The sample locations for the residential and background sites are listed in Figure 7.23, Appendix 7A. The local limits parameters are listed in Figure 7.24, Appendix 7A.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

### **7.13 Sample Delivery/Holding Times**

#### **7.13.1 Industrial and non-Industrial samples**

Industrial samples will be picked up on a daily basis at Chelsea by the Deer Island courier between 3 p.m. and 4 p.m. If a sampling team cannot return to Chelsea prior to 4 p.m., contact the Deer Island courier by radio about samples arriving late.

#### **7.13.2** Contact the Central Lab if samples with short-holding times will be collected after normal business hours. Use the following procedure for delivering any samples to the Central Lab after normal business hours:

1. Notify the Deer Island Security Main Gate at (617) 539-4292 that samples will be delivered to the lab.
2. Enter the laboratory building through the main doors and sign in.
3. Walk through the door, past by the elevator to get to the sample drop-off area.
4. In the sample drop-off area if no one from the lab is there, proceed to the TRAC drop-off refrigerator, which is located in the wash down area located to the right of the stainless steel counters (refrigerator is behind the wall).
5. Place samples and COCs in the refrigerator.
6. Locate the key in the envelope and lock the refrigerator door.
7. Place the key back inside the envelope and slide the envelope under the door labeled Room 145.
8. Sign out when leaving the facility.

Section No.: 7  
Revision No.: 3  
Date: January 16, 2007

**7.13.3** If samples are to be left in the refrigerator at Chelsea, use the COC to relinquish (sign COC) the samples over to "sample storage" and retrieve them (sign COC) from storage. See section 7.3.5.1 for complete details on sample storage at Chelsea.

**7.13.4** All NPDES biotoxicity and wet chemistry samples are delivered to the Central Lab.

**7.13.5 Holding Times**

Note the following holding times when signing samples over to sample storage:

<u>PARAMETER</u>	<u>HOLDING TIME</u>
Solids (TDS, TSS, TS)	7 Days
Metals	6 Months
Hexavalent Chromium ***	24 Hours
Mercury	28 Days
BOD ***	48 Hours
Coliform ***	6 Hours
Cyanide, Total (no sulfides)	14 Days
Cyanide, Total (with sulfides)***	24 Hours
Formaldehyde	72 Hours
MBAS (Surfactants) ***	48 Hours
Nitrate/Nitrite	48 Hours
Nitrogen, Ammonia/TKN	28 Days
FOG	28 Days
PHC	14 Days
Total Phenolics	28 Days
Phosphorus, Total	28 Days
Sulfate	28 Days
Sulfide	7 Days
ABNs	7 Days
Pesticides/PCBs	7 Days
VOAs (Preserved)	14 Days
VOAs (Unpreserved)	7 Days

**NOTE:** \*\*\* Do not store these samples overnight in the refrigerator.

For a more complete list of parameters and holding times, see Figure 7.4 and 7.5, Appendix 7A.