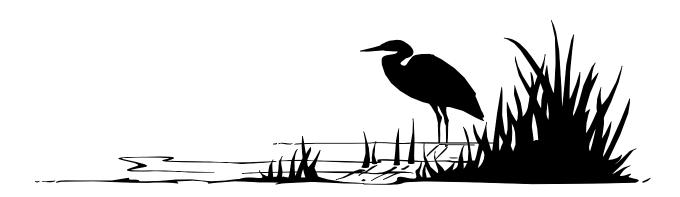
## SURVEYS SECTION FIELD PROCEDURE MANUAL





INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
Office of Water Quality
Assessment Branch
Surveys Section
IDEM 32/02/055/2002
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Surveys Section SOP DRAFT Ver. #: 1 Date: June 2002 Page 1 of 86

## SURVEYS SECTION FIELD PROCEDURE MANUAL

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Revised June 2002

Compilation and development of the final report was the primary responsibility of the Surveys Section Arthur C. Garceau, Surveys Section Chief

INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT
OFFICE OF WATER QUALITY
ASSESSMENT BRANCH
SURVEYS SECTION
IDEM 032/02/055/2001
June 2002

Surveys Section SOP DRAFT Ver. #: 1 Date: June 2002 Page 2 of 86

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## 1.0 OVERVIEW

## 1.1 INTRODUCTION

The Surveys Section is part of the Water Quality Assessment Branch of the Office of Water Quality. The Surveys Section's primary function is to provide water quality and hydrological data to assess Indiana's surface waters. This is accomplished by conducting watershed basin surveys and stream reach surveys throughout the state. As part of the Surface Water Quality Monitoring Strategy, the Surveys Section is conducting a five-year sampling plan of the State's ten major watershed basins. Information collected will be reviewed by the Environmental Toxicology and Chemistry Section and integrated with data from Biological Studies Section to complete a thorough assessment of the State's surface waters.

The Surveys Section is organized into four work groups: Watershed Monitoring, Fixed Station Monitoring, Special Projects, and Data Administration. Staffing charts listing areas of responsibility are presented in Table 1.1 and Table 1.2.

During the summer months, student assistants who are assigned by the Governor's internship program augment the staff. Office, laboratory, and equipment storage space is located at the Shadeland Avenue Office. The Surveys Section is also assisted through the cooperative efforts of the Indiana State Department of Health's Water Quality Laboratory and the U.S. Environmental Protection Agency (U.S. EPA). The Surveys Section also utilizes the contract services of the U.S. Geological Survey and various commercial contract laboratories. A brief discussion of the work group programs follows.

## 1.2 THE WATERSHED MONITORING PROGRAM

This program uses a statistically valid number of randomly selected sites throughout major river basins to assess and characterize the overall water quality and biological integrity. Selection of sites focuses on all streams within the targeted river basin. Sampling is performed during the late Spring to early Fall low-flow time frame (May-October) and is conducted simultaneously with other program areas. Data sets collected and assessed are water and fish tissue samples for laboratory analysis, in-situ water chemistry, channel morphology data, fish community assessments, and habitat assessment. The results of this program further contribute to the Indiana 305(b) Report and the pending Section 303(d) list of impaired streams. The results are applied to the Indiana Fish Consumption Advisory, as well as provide for support of the Environmental Performance Partnership Agreement (EnPPA) and the development of biocriteria for Indiana's surface waters. This program is conducted through the cooperative sampling efforts of the Surveys Section and Biological Studies Section of the Assessment Branch, and through the support of the U.S. EPA Research Laboratory in Corvallis, Oregon.

#### 1.3 THE FIXED STATION MONITORING PROGRAM

The objective of this program is to provide basic information that would reveal recurring surface water quality trends and provide data for the many impacted users of surface water in Indiana.

The program was developed to determine chemical, physical, bacteriological, and biological characteristics of Indiana water under changing conditions. The information has been used in determining background data for certain types of pollutants, such as chlorides and phosphorous; in the development of wasteload allocations and NPDES permits for wastewater treatment plants; for other municipal, industrial, agricultural, and recreational uses; for future pollution abatement activities such as review of non-point pollution sources; and in procuring data to secure public action toward the preservation of streams for all beneficial uses. The fixed station network was established in 1957, and currently samples 160 stations throughout the state on a monthly basis. The Indiana State Department of Health (ISDH) Water Quality Laboratory performs sample analyses.

## 1.4 THE SPECIAL PROJECTS PROGRAM

This group's activities include: the Pesticide Monitoring Program, the *E. coli* Monitoring Program, and other various specialized water quality monitoring projects. These "special projects" include but are not limited to Wasteload Allocation (WLA) studies, stream reach studies, and site specific monitoring in support of the NPDES permitting program.

## 1.5 THE DATA ADMINISTRATION GROUP PROGRAM

The Data Administration group is responsible for the development of reports and dissemination of information collected by the Surveys section. The Data Administration group maintains responsibility for final editing, publication and distribution of documents produced by the Surveys section. Documents produced by the section are published on the Internet. Mapping and graphical representation of data are provided to the Surveys section and the Assessment Branch staff, as well as other Agency personnel by the Data Administration group. Data requests from interested parties outside of the section are received and filled. The Data Administration group has also been assigned responsibility for maintaining the Assessment Branch's database, the Assessment Information Management System or AIMS.

## 2.0 PROCEDURAL GUIDELINES

#### 2.1 SAMPLING PROCEDURES

#### 2.1.1 GENERAL

These procedures apply to all water samples collected by Surveys section personnel for laboratory chemical analysis during surface water monitoring and stream surveys. It is necessary to know how to take a sample that is most representative of the stream, lake, or wastewater. It is obvious that improper sampling will give erroneous results. It is also important to maintain the integrity of that sample by preservation if necessary and to get the sample to the laboratory for analysis within the required holding time.

**Table 1.1 Surveys Section Staffing Table** 

## Art Garceau Surveys Section Chief 308-3381

Watershed Monitoring Group	Fixed Station Monitoring Group	Special Projects Group	Data Administration Group
Larry McFall Senior Environmental Manager 308-3200	Mark Holdeman Senior Environmental Manager 308-3198	Steve Boswell Senior Environmental Manager 308-3201	Chuck Bell Senior Environmental Manager 308-3203
Vacant Environmental Manager	Sam Gibson Environmental Manager 308-3197	Tim Beckman Environmental Manager 308-3195	Joanna Wood Environmental Manager 308-3211
Jim Butler Environmental Scientist 308-3199	Joel Armstrong Environmental Scientist 308-3196	Roseann Hirschinger Environmental Scientist 308-3204	Cindy Martin Environmental Scientist 308-3081
Elizabeth Klicker Environmental Scientist 308-3361	David Arnold Environmental Scientist 308-3398	Ryan McDuffee Environmental Scientist 308-3194	Vacant Laboratory Tech 3

Table 1.2 Surveys Section Contact Persons		
Tim Beckman	ckman Hydrolab, turbidimeter, and pH meter maintenance	
	Calibration standards	
	DI water system maintenance, digital camera	
Steve Boswell	ISCO Automatic Samplers	
	Current Meter maintenance	
Ryan McDuffee	Boat and boat trailer maintenance	
Larry McFall	Flow measurement equipment and automatic sampler maintenance	
	Summer intern coordinator	
Sam Gibson	Vehicle maintenance	
Chuck Bell	Laptop computers	
Mark Holdeman	Health and safety	
Elizabeth Klicker	Cellular phone maintenance	
	-	

#### 2.1.2 SAMPLING LOCATION

If an NPDES permit holder is sampled, generally at least the effluent is taken, and in some instances the influent might also need to be tested. Since no two treatment systems are identical, it is difficult to be precise about the sampling locations until the layout of the plant is known by the sampler. In surveying the effect that the wastewater treatment plant effluent has on the receiving stream, generally one upstream sample is taken and as few as one to several samples are taken downstream, preferable after the effluent is completely mixed with the stream. However, the difficulty comes in knowing exactly where the area of the completely mixed water begins. Quantity and quality of wastewater may change in the discharge. Also, the stream flow may vary within the sampling period. Hence, no specific guidelines are given on the exact location for sampling.

#### 2.1.3 SAMPLING METHODS

Sampling methods are either manual or automatic. The quality of the sample depends directly on the care used in collecting the sample. To get a representative sample with either method, samples generally are collected just under the surface of the water in the main stream of flow. Skimming the surface of the water or dragging the bottom must be avoided.

## 2.1.3.1 Manual Sampling Procedures

Every effort is put forth to collect the sample directly into a sample bottle. A sample collection device containing the sample bottle(s) is lowered from a rope if direct access to the waterbody is not possible. When direct access is possible, hold the bottles in a gloved hand and fill just under the surface of the water while facing upstream to collect a sample. A special sampling device for sample collection provides a way to put the samples directly into certified clean disposable plastic or glass bottles from a bridge or other structure. If a sample collection device is used, clean it thoroughly in the bottle washer at the end of each sampling event. Water taken for field tests (dissolved oxygen, pH, conductivity, etc.) is collected in the above manner, or it can also come from a plastic or stainless steel bucket used to grab samples, after the bucket has been thoroughly rinsed with the sample water. In some cases, temperature of the water changes rapidly and this parameter must be measured as soon as possible after the sample is collected. Also, it is important to keep the sample well mixed in the bucket if turbidity is required because the suspended solids in the sample will settle to the bottom of the bucket in a very short time.

Sampling from the stream bank is permissible providing the collector can reach the main flow. Any deviations from these general procedures must be noted on the field sheet by the collector. The quality of a sample depends directly on the cleanliness and reliability of the sampling method. Preventing the contamination of a sample during the sampling process is the greatest challenge in collecting reliable results and representative samples. Therefore, it is imperative that care be taken to avoid or minimize contamination when collecting samples.

The following step-by-step procedures result in clean and reliable samples.

## 2.1.3.1.1 Bridge Sampling Procedures

Park as close to the sampling site as possible, turn on the vehicle's hazard lights and the strobe light.

- 1. Turn the vehicle off and open the tailgate.
- 2. Put on new, non-talc sampling gloves.
- 3. Remove sampling device from the transportation container and place it in the carrying tote.
- 4. Remove bottles to be used at site. Label if necessary, including date, site number, IDEM sample number (AA number), and parameter abbreviation (G.C., Nx, Metals, Blank, etc.). Place bottles in carrying tote.
- 5. **Note:** Carrying tote should already contain a clean rope and a ziplock bag.
- 6. Take carrying tote with sampler, bottles, and rope to sample collection site.
- 7. At site, attach rope and insert collection bottle(s) into sampling device. **Always** collect metals first.
- 8. Remove caps from the bottles and place caps in a ziplock bag. Secure the bag.
- 9. Retrieve sample from **downstream** side of bridge.
- 10. Replace sampling bottle caps and remove bottles. Place full bottles in carrying tote.
- 11. Insert the remaining sample collection bottles into sampling device.
- 12. Repeat steps 10 through 12 until all samples have been collected.
- 13. Return to vehicle with tote and equipment. **Put on safety glasses.** Preserve all samples and properly dispose of preservative vials
- 14. Rinse sampling equipment with deionized water, including sampling device, bottles, and the rope if it was immersed during sample collection.
- 15. Place sample collection bottles into cooler with ice.
- 16. Detach sampling device from rope. Put rope in storage bag or carrying tote.
- 17. Place clean sampling device into transportation container.
- 18. Discard gloves and close vehicle.
- 19. Proceed to next site.
- 20. At last site, make sure samples are well iced to ensure proper preservation.
- 21. Return to station.

## 2.1.3.1.2 In-Stream Sampling Procedures

- 1. Put on new non-talc sampling gloves.
- 2. Obtain bottles to be used at site. Label bottle, if necessary, with the date, site number, IDEM sample number (AA number), and parameter abbreviation (G.C., Nx., Metals, Blank, etc.). Place bottles in carrying tote.
- 3. Put on Personal Floatation Device per Safety Manual.
- 4. Approach sampling site and wade into stream at a location **downstream of the site** in order to not stir up the stream bed sediments.
- 5. While facing **upstream** remove bottle cap and insert the bottle directly into the stream just below the surface making sure to fill the bottles up to one inch below the opening.
- 6. Re-cap the sample bottle and repeat as necessary until all of the bottles are filled up.
- 7. Return to vehicle with tote and equipment. **Put on safety glasses.** Preserve all samples and properly dispose of preservative vials.
- 8. Rinse the outside of the sample bottles with deionized rinse water and place collection bottles into cooler.
- 9. Make sure the samples are well iced.
- 10. Remove gloves and proceed to next site.

#### 2.1.4 SAMPLE PRESERVATION

**Table 2.3 Sample Preservation Requirements** 

General Chemistry Sample (G.C.)	Ice only. No other preservation required.
Nutrient Sample (Nx):	Preserve with 2 ml of 50% Sulfuric Acid (H <sub>2</sub> SO <sub>4</sub> ).
Metal Sample (Metals)	Preserve with 5 ml of concentrated Nitric Acid (HNO <sub>3</sub> ).
Cyanide Sample (CN):	Preserve with Sodium Hydroxide Crystals (NaOH).
Bacteriological Sample, E. coli (Bug):	Ice only. No other preservation required.

NOTE: Always wear safety glasses and latex gloves when preserving samples.

### 2.1.4.1 Preservative Vials

Preservative vials are provided in the appropriate volumes to preserve the one liter samples typically collected by the Surveys section. The preservative vials come with screw caps and are used for normal field preservation. Simply unscrew the cap and poor the contents into the appropriate sample bottle. When finished, replace the vial cap and dispose of the vial in a sealable plastic container or ziplock bag. Bring this container back to the office for proper disposal.

#### 2.1.5 DELIVERING SAMPLES TO THE LABORATORY

- 1. Never assume that the laboratory personnel know anything about the samples!
- 2. Always wear clean gloves when handling sample bottles.
- 3. Set up bottles in sequential IDEM sample number.
- 4. Make sure all bottles are labeled properly and put laboratory labels with preservative identification on bottles.
- 5. Ensure field sheets, laboratory sheets, and chain of custody (COC) forms are properly filled out and signed.
- 6. Secure a photocopy of the chain of custody form after it is signed.
- 7. Return to station.

#### 2.1.6 Duties to Perform Upon Return to Office

- 1. Unload all equipment from vehicle.
- 2. Remove all trash from vehicle and clean the interior.
- 3. Wipe down the interior with a damp cloth if it is dusty or dirty.
- 4. Wash all equipment that will not fit into washer by hand and allow to air dry. Empty and clean all used coolers immediately upon return.
- 5. Wash smaller equipment in the washer (sampling device, plastic sampling tote, plastic bucket, rope, etc.).
- 6. Place clean and dried sampling rope into ziplock bags and put other clean equipment back on equipment room shelf.
- 7. Report any equipment or vehicle problems to supervisor and to person in charge of that specific equipment. See Page 9 of this manual for the equipment contact person.

#### 2.1.7 CHAIN OF CUSTODY PROCEDURES

## 2.1.7.1 Introduction

The procedures and definitions described below shall be used to maintain data validity and control. This includes the sample numbering system, custody of samples in the field, via certified carrier, and at the laboratory and all associated transfers of custody.

#### 2.1.7.2 Definition

- 1. A sample is in someone's custody if:
- 2. It is in his/her actual possession, or
- 3. It is in his/her view, after being in his/her possession, or

- 4. It is in his/her physical possession and locked up so that no one could tamper with it, or
- 5. It is kept in a secured area restricted to authorized personnel only.

#### 2.1.7.3 Procedures

To assure proper handling and custody of samples collected by field personnel of the Surveys section the following procedures will be followed in the field. These procedures should document the people who have had charge of the sample from the time of the collection until it was delivered to the laboratory. The laboratory also has chain of custody procedures which will be followed after it receives the sample.

The collector will keep field notes. This is to record conditions and activities related to each sample collection. The sample will be placed in a container appropriately marked with the sample site, date of collection, and type of sample. If the sample remains in the custody of the sampler, as described above, no sample seals will be required. However, if the sample will leave the custody of the sampler, a sample seal should be used.

When the sample is delivered to the laboratory for analysis, the person who delivers the sample and the person who receives the sample for the laboratory will sign the chain of custody sheet in the appropriate spaces. At this time the laboratory takes custody of the sample and their chain of custody procedures will be followed. Upon arrival at the laboratory, the sample collector presents to the laboratory personnel laboratory analysis request sheets, chain of custody sheets, and duplicates and/or blanks. The laboratory analysis request sheets can be computer generated.

# 2.2 SAMPLING PROCEDURES FOR THE COLLECTION OF TRACE METALS USING CLEAN SAMPLING TECHNIQUES

### 2.2.1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes methods for the collection of ambient water using EPA method 1669 (USEPA, 1996) for the determination of mercury, methylmercury, trace metals, and hexavalent chromium by EPA test methods 1631, 1630 (Draft), 1638, and 1636, respectively. In 1998, IDEM had undertaken a Trace Metals Pilot Project to develop expertise in collecting ambient water samples using clean sampling techniques and metal analyses by low detect ultra-clean analytical test methods (Ratcliff, B.L. and GhiasUd din, S.M., 1999). This SOP is an extension of this Trace Metals Pilot Project for similar work at IDEM. Adherence to this SOP can be expected to minimize contamination from the sample bottle, any necessary sampling equipment used, and external sources.

This SOP is for the collection of a grab sample directly into the sample bottle with or without the aid of a sampling pump, Teflon® and C-Flex® tubing, capsule filters, and the appropriate Teflon® fittings necessary to join the various types of tubing to the capsule filter.

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## FIGURE 2.1 SAMPLING EQUIPMENT CHECKLIST

	Two (2) sampling devices.*
	Two (2) sections of nylon rope, bagged separately for sampling device.*
	Two (2) clean, plastic stream buckets with rope for field measurements.*
	One (1) Hydrolab with a display unit and one (1) sampling tube.
	Two (2) sampling equipment storage containers (totes).*
	Adequate Tupperware containers for bacterium sample storage.
	Several equipment bags, ziplock bags, rubber bands, extra sample device tubing, trash bags, and tape.
	One (1) pH meter.
	One (1) turbidimeter (if needed).
	One (1) D.O. kit, including two (2) bottles, fresh reagents, extra pipets, extra pipette pump, and Millipore rinse water.
	Two (2) boxes of new, non-talc gloves.
	One (1) bottle of sanitizing gel.
	One (1) eyewash bottle filled with DI water.
	Two (2) carboys with Millipore rinse water and extension tubes.
	Coolers containing labeled sample bottles and bacteria bottles. Always wear gloves when handling sample bottles.
	Two (2) small red coolers, one for blanks and extra reagents, and one for Hydrolab storage, if needed.
	Extra cooler full of ice from equipment room ice machine.
	One (1) large ziplock bag, containing marking pens and bottle labels.
	Waders.
	One (1) set of USGS gage keys.
	Required preservatives for sampling event.
	All field, laboratory, and chain of custody sheets for route with clipboard.
	Route maps, topographic maps, and state map.
	One (1) cell phone for every vehicle.
	Safety Glasses.
	(*indicates non-wadeable/bridge sampling sites only).
1	

#### 2.2.2 SAMPLE BOTTLE AND SAMPLING EQUIPMENT REQUIREMENTS

Only Teflon® sample bottles and any necessary sampling equipment which have been cleaned, tested, and double bagged in a Class-100 clean bench (or equivalent) and certified clean through appropriate testing relative to the relevant EPA test methods can be used. Uncertified Teflon® sample bottles and sampling equipment cannot be used because they may be a source of possible contamination.

Teflon® sample bottles and any necessary sampling equipment are to be obtained from the contract laboratory performing the desired analyses.

#### 2.2.3 SAMPLE COLLECTION

The collection of samples is performed using the "clean hands-dirty hands" technique described in EPA method 1669 (USEPA, 1996). Bottles are sealed tightly and re-bagged using the opposite series of steps as were used to open them. Samples are preserved immediately upon collection and are shipped to the contract laboratory via overnight courier for the desired analyses.

Ideally, at least two people each wearing fresh talc free polyethylene gloves (wrist size) or equivalent are required on a sampling crew. Fresh gloves should be worn at all times when handling samples or sampling equipment.

- 1. One person (designated "dirty hands") removes a bagged bottle from the box or cooler, and opens the outer bag, avoiding touching the inside surface of the bag.
- 2. The other person (designated "clean hands") reaches in, opens the inner bag, and removes the sample bottle. "Clean hands" should not touch anything but the outside surface of the sample bottle and cap. If anything other than the sample bottle, cap, or water is touched, "clean hands" must change gloves.
- 3. "Clean hands" opens the sample bottle and holds the bottle in one hand and the cap in the other. If it is necessary to set the cap down, it should be placed in the inner bag from which the sample bottle was removed. The sample is collected from just below the surface of the water at or near the centroid of flow or transferred from a bigger precleaned bottle used for sample collection. Sampling locations are selected as far away from bridges as possible in order to minimize contamination from road or bridge dust and debris. Due to the potential for contamination, sampling will not occur during a rain event because rain is known to contain mercury (wet atmospheric deposition). The people collecting the sample should be wary of disturbing the flow upstream of the sampling point.
- 4. Rinse the sample bottle and the inside surface of the cap three times with sample water, and fill the bottle to the top with sample while leaving enough void volume to accommodate the appropriate volume of the various preservatives. Replace the cap, and tighten securely.

- 5. Re-bag the bottle in the opposite order it was removed.
- 6. If during sampling a sampling pump, Teflon® and C-Flex® tubing, capsule filters, and the appropriate Teflon® fittings necessary to join the various types of tubing to the capsule filter are utilized, this equipment must be handled in the same manner as the sample bottles are handled using the "clean hands-dirty hands" technique.
- 7. If a sampling pump, Teflon® and C-Flex® tubing, capsule filters, and the appropriate Teflon® fittings necessary to join the various types of tubing to the capsule filter are utilized, this equipment must be purged with sample for one minute before the sample is collected.
- 8. Gloves should be changed between samples and whenever anything not known to be trace metal clean is touched.

#### 2.2.4 COLLECTION OF FIELD BLANKS AND FIELD DUPLICATES

A field blank will be collected with every 10 samples or as described in the sampling plan. A sample bottle for the field blank should be requested from the laboratory. A separate bottle or carboy filled with reagent water is used for the field blank and provided by the contract laboratory.

- 1. To collect the field blank, open an empty sample bottle using the "clean hands-dirty hands" technique described above. Also, open the bottle or carboy containing the reagent water.
- 2. Pour the reagent water into the empty sample bottle. This is now the field blank. If a sampling pump, Teflon® and C-Flex® tubing, capsule filters, and the appropriate Teflon® fittings necessary to join the various types of tubing to the capsule filter are utilized, the reagent water must be pumped through this equipment prior to pouring into the sample bottle. The field blank is collected prior to the collection of a sample. After field blank collection, the Teflon® and C-Flex® tubing, capsule filters, and the appropriate Teflon® fittings necessary to join the various types of tubing to the capsule filter do not have to be changed prior to actual sample collection if this equipment is utilized.
- 3. Re-bag the field blank in the opposite order it was removed.
- 4. A field duplicate will be collected with every 10 samples or as described in the sampling plan. A sample bottle for the field duplicate should be requested from the laboratory. A separate sample bottle is used to collect the field duplicate. The field duplicate is collected in the same manner as an actual water sample. The field duplicate is collected immediately after the original sample without changing the Teflon® and C-Flex® tubing, capsule filters, and the appropriate Teflon® fittings necessary to join the various types of tubing to the capsule filter if this equipment is utilized.

## 2.2.5 Preservation, Packing, Refrigeration, and Shipment of Water Samples

All water samples must be preserved in the field or in the laboratory in accordance with sampling and analysis work plan requirements, capped tightly, and maintained at 4° Celsius

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with ice from the time of collection until receipt by the contract laboratory.

The double-bagged samples are bagged in a large "sampling location bag" which provides protection from the ice and the resultant water from melting ice.

A Chain of Custody sealed in a plastic bag will accompany the samples during shipment to the contract laboratory. The coolers containing the samples will be thoroughly sealed with adhesive tape in order to provide protection from spillage as well as a forming a custody seal. All bottle and preservative lot numbers supplied by the contract laboratory are recorded on the appropriate field sheets.

Samples will be shipped via overnight courier to contract laboratory at the conclusion of each sampling event.

# 2.3 GUIDELINES FOR THE OPERATION OF THE *E. COLI* MOBILE LABORATORY

**NOTE:** Sampling and analysis procedures described in this section are preliminary. Procedures are being refined during the current sampling season. Revisions will be published in the next version of this manual.

## 2.3.1 Sample Collection, Handling, and Preservation

## 2.3.1.1 Sample Collector

The sample collectors shall be trained in aseptic sampling procedures.

#### **2.3.1.2 Sampling**

At least 100 mL of sample must be collected, allowing at least a 1-inch air space to facilitate mixing of the sample by shaking. Immediately after collection, a sample information form should be completed.

## 2.3.1.3 Sample Information Form

After collection, the sampler should enter on a sample information form, or field sheet in indelible ink, the following information:

- Name of site, or identification number
- Sample identification #
- Sample site location
- Sample type

- Date and time of collection
- Analysis required
- Disinfectant residual
- Name of sampler and organization
- Sampler's initials
- Person(s) transporting the samples to the laboratory (if not the sampler)
- Transportation condition (e.g., <10°C, protection from sunlight).
- Any remarks

Source water samples must be representative of the source of supply, collected not too far from the point of intake, but at a reasonable distance from the bank or shore. The sample volume should be sufficient to perform all the tests required.

## 2.3.1.4 Sample Icing

Water samples must be held at <10°C.

## 2.3.1.5 Sample Holding/Travel Time

The time from sample collection to initiation of analysis for total coliforms and fecal coliforms in source water and heterotrophic bacteria in drinking water must not exceed six hours.

## 2.3.1.6 Chain-of-Custody

Sample collectors and laboratories must follow applicable Field Procedure Manual requirements pertaining to chain-of-custody. Even if custody is never transferred, a chain of custody form shall be filled out and filed as part of the data set.

## 2.3.1.7 Laboratory Facilities

Laboratory facilities should be clean, temperature- and humidity-controlled, and have adequate lighting at benches. There will be provisions for disposal of microbiological waste.

Laboratory facilities should have sufficient bench-top area for processing samples; storage space for media, glassware, and portable equipment; and floor space for stationary equipment such as incubators, water baths, refrigerators, etc.

## 2.3.1.8 Temperature Monitoring Devices

Glass, dial, or electronic thermometers must be graduated in 0.5°C increments or less. The fluid column in glass thermometers should not be separated. Dial thermometers that cannot be calibrated should not be used. Calibrations of glass and electronic thermometers should be checked annually and dial thermometers quarterly, at the temperature used, against a

reference National Institute of Standards and Technology (formerly National Bureau of Standards [NBS]) thermometer or one that meets the requirements of NBS Monograph SP 250-23. The calibration factor should be indicated on the thermometer. Also, the laboratory should record the date the thermometer was calibrated and the calibration factor in a QC record book.

If a thermometer differs by more than 1°C from the reference thermometer, it should be discarded. Reference thermometers should be recalibrated every three years.

#### 2.3.1.9 Incubator Unit

Incubator units must have an internal temperature monitoring device and maintain a temperature of  $35 \pm 0.5$ EC. Air-type incubators, especially small ones, may not be able to bring a cold 100 mL water sample(s) to the specified incubation temperature of  $35^{\circ}$ C for several hours. This problem may be further aggravated if several cold water samples are placed in the incubator at the same time. The problem may cause false-negative results with the chromogenic/fluorogenic substrate tests. Therefore, laboratories with air-type incubators should check the time it takes for a 100 mL water sample (or a set of 100 mL water samples, depending on normal use) to reach 35EC, and ensure that the specified incubation period at that temperature is followed. This check should be repeated whenever there is a significant change in the sample load.

Calibration-corrected temperature should be recorded for days in use at least twice per day with readings separated by at least 4 hours.

## 2.3.1.10 Reagent-Grade Water

Only satisfactorily tested reagent water from stills or deionization units may be used to prepare media, reagents, and dilution/rinse water for performing bacteriological analyses. Reagent water quality criteria are listed in Table 2.4.

Table 2.2 Reagent Water Quality Criteria for Bacteriological Analysis

Parameter	Limits	Frequency
Conductivity	<2 micromhos/cm at 25°C	Monthly
Pb, Cd, Cr, Cu, Ni, Zn	<0.05 mg/L per contaminant* (*Collectively, no greater than 0.1mg/L)	Annually
Heterotrophic Plate Count ** (** Pour Plate Method. See Standard Methods 9215B)	< 500/mL	Monthly

## 2.3.1.11 Pipets

To sterilize and maintain sterility of glass pipets, stainless steel or aluminum canisters can be used or individual pipets should be wrapped in char-resistant paper or aluminum foil. Pipets must have legible markings and should not be chipped or etched. Opened packs of disposable sterile pipets should be resealed between use periods. Pipets delivering volumes of 10 mL or less must be accurate within a 2.5% tolerance.

## 2.3.1.12 Laboratory Equipment and Supplies

The laboratory must have the equipment and supplies needed to perform the approved methods.

#### 2.3.2 ANALYTICAL METHODOLOGY FOR COLILERT

Water samples should be shaken vigorously about 25 times before adding media.

#### 2.3.2.1 Use of Colilert Media

Each new lot of dehydrated or prepared commercial medium should be checked before use with positive and negative culture controls. In addition, each batch of laboratory-prepared medium should include positive and negative culture controls. These control organisms can be stock cultures (periodically checked for purity) or commercially available disks impregnated with the organism. Results should be recorded.

These media **must not** be prepared from basic ingredients, but rather purchased from a commercially available source. The media must be protected from light.

Some lots of fluorogenic media have been known to autofluoresce. Therefore, each lot of medium should be checked before use with a 366-nm ultraviolet light with a 6-watt bulb. If the media exhibit faint fluorescence, the laboratory should use another lot that does not fluoresce. If the samples plus a medium exhibit a color change before incubation, it should be discarded and another batch of medium used.

For each lot of medium, a quality control check must be performed by inoculating sterile water containing the medium with a MUG-positive E. coli strain, a MUG-negative coliforms, and a non-coliforms and analyzing them.

## 2.3.2.2 Use of Quanti-tray 2000

Laboratories may also use Quanti-Tray test or Quanti-Tray 2000 test for drinking water and source waters. Both tests use the Colilert medium. If the Quanti-Tray or Quanti-Tray 2000 test is used, the sealer should be checked monthly by adding a dye (e.g., bromcresol purple) to the water. If dye is observed outside the wells, another sealer should be obtained.

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For enumerating total coliforms with the Colilert test, Quanti-Tray or Quanti-Tray 2000 must be used for each sample dilution tested. Dilution water (for the chromogenic/fluorogenic substrate test only), if used, must be sterile dechlorinated tap water, deionized water, or distilled water.

For the Colilert test, samples must be incubated at  $35 \pm 0.5$ °C for 24 hours. A yellow color in the medium equal to or greater than the reference comparator indicates the presence of total coliforms and must be reported as a total coliforms positive. If the sample is yellow, but lighter than the comparator, it must be incubated for another four hours. Do not incubate more than 28 hours total. If the color is still lighter than the reference comparator at 28 hours, the sample should be reported as negative.

Table 2.3 E. coli Equipment Checklist

Expendable Supplies	Capital Supplies
SAMPLE BOTTLES	UV lamp
Quanti-trays	2 Incubators
Colilert Enzyme Substrate	Quanti- tray Sealer
10 mL disposable PIPETS	Thermometers
LATEX GLOVES	CLEAN Ropes in ZIPLOCK bags
Hand sanitizer	4 medium COOLERS
Bench Sanitizer	2 FOR BOTTLE STORAGE
2 CARBOYS FULL OF DI WATER	1 W/ ICE
Heavy Duty Trash Bags	1 for dilution water
EXTRA ZIPLOCK BAGS	2 TOTES
Bio-Hazard Bags	1 To Store CLEAN Sampler
Record-keeping	1 To Take on BRIDGES
E. coli Field Sheets	STAINLESS STEEL BUCKET
SHARPEE MARKERS	PLASTIC BUCKET WITH ROPE
PENS AND PENCILS	SAMPLER AND SPARE SAMPLER
CLIPBOARD	INDIVIDUALS SAFETY GEAR
Laptop computer	HYDROLAB
2 disks	SMALL RED COOLER
PHONE	DO KIT
Work Plan and SOP for van	pH METER
Operations	Turbidity meter
Lab log	
Incubator logs	
MAPS	

## 2.4 ISCO AUTOMATIC SAMPLER PROCEDURES

#### 2.4.1 GENERAL

Currently, four types of ISCO samplers are being used by the Surveys section. They are models 2700, 2900, 3700, and 6000. The primary difference in these models is size and sample capacity. The 2700 and 3700 models are the larger samplers and each contain 24 plastic bottles of 1000 mL capacity. The 2900 model is a smaller sampler with each of its 24 bottles having a 500 mL capacity. The ISCO model 6000 is used for collecting volatile organic compounds (VOCs) and utilizes 40 mL vials for collection of samples. All four of these samplers function by means of peristaltic pumps and can be operated by means of a Nickel-Cadmium battery pack or by an external direct current source. The samplers incorporate sealed control boxes with electronic keypads for easy programming. The programming capabilities of each model allow for numerous sampling possibilities. The operating instructions are listed, in condensed form, on the top of each control box. These samplers are primarily used in a timed-interval mode in which discrete aliquots or samples are collected at fixed intervals. A flow weighted sample is then achieved by proportioning the aliquots according to a continuous flow recording which is made during the sampling period. A further explanation of this method can be found in the U.S. EPA NPDES Compliance Manual (USEPA 1977). All sampler models can be used with the ISCO flow meter models 2870 and 3230 to produce automatically flow-weighted composites. However, it must be known with some accuracy what the total flow for the 24-hour sampling period will be in order for this technique to be used. See page 14 of this Manual in reference to setting up sequential flow sampling. With the exception of the model 6000, the plastic bottles used in these samplers restrict sample collection to general chemistry (BOD, suspended solids, pH, etc.), nutrients (ammonia, phosphorus, TKN, etc.), and metals parameters. Some of the 2700 models have been modified to hold a single 2 1/2 gallon glass container for toxic pollutant sample collection. Samples for toxic pollutants should contact only tetrafluoroethylene (Teflon), glass, and/or medical-grade silicone rubber in the peristaltic pump transport system. Refer to the toxic sample collection section of this SOP for an in-depth explanation of this method.

Important considerations when setting up automatic samplers include water quality and flow rate in a given flow stream. These parameters can vary considerably from one moment to the next. Sampler programming may dictate frequent aliquot collection in some sampling situations to obtain an accurate representation of flow over a given period of time. A flow waste stream may also be highly stratified, nonhomogeneous, and may present a less than ideal medium from which to take a representative sample. Current assessment of sampling methods (USEPA 1977), suggest that strainer placement should be at 60% of the stream depth in an area of maximum turbulence. The selection of 60% depth is based on velocity and sedimentation charts from the same report. Strainer intakes should definitely not rest on the bottom of the flow stream where sediments collect, or against side walls of tanks where solids or bacteriological buildups might artificially enrich the sample.

Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

These samplers are cleaned after each use by rinsing appropriate parts (funnels, bases, etc.) in hot water. If necessary, a detergent will be used. A 50% HCl solution is run through the pumps, followed by several rinses of distilled water. All bottles are cleaned in the Surveys section laboratory bottle washer. After the bottles are cleaned, they are capped until used to prevent contamination. Records concerning the use and maintenance of samplers are found in the sign-in/sign-out log kept in the Surveys section office. Additional information on automatic samplers can be found in the instruction manuals for these instruments.

#### 2.4.2 TIME COMPOSITE SAMPLING

The object of composite time sampling using the Model 2700 is to collect a series of discrete, equal volume samples at a known regular time interval, and to place these samples into a single composite sample container. When programming the sampler in a composite time mode, two quantities must be calculated to allow the sampler to be programmed in a rational manner:

- 1. the total number of samples to be collected, and
- 2. the volume of each individual sample.
- 3. To determine the total number of samples to be collected, two quantities must be known.
- 4. the time increment between samples, and
- 5. the total time over which the composite samples are to be taken.

#### 2.4.3 SEQUENTIAL FLOW SAMPLING

Use of the sequential flow mode (#2) on the ISCO automatic sampler permits flow-proportioning of the composite sample at the time of collection. This eliminates the time consuming tasks of averaging the flow, calculating, measuring, and pouring individual time sequential aliquots.

In order to use this option, a primary flow measuring device, which is compatible with an ISCO flow meter, must be located near the sampling site. It is also recommended that this set-up be employed only where a power source is available. If it is necessary to use battery power, install a battery on both the flow meter and the sampler. When using power packs, the flow meter can be powered by the power pack on the sampler. In addition to the flow meter and sampler, the six (6) prong connector cable is required to complete the set-up. If it is desired to run two samplers off the same flow meter, the splitter cable must be used. The

splitter cable is connected to the flow meter and then the connector cables are fitted to the samplers. Each of the samplers can then be programmed as desired. It is not necessary to program both samplers in the same way.

Prior to programming the samplers, a reliable estimate of the anticipated volume of flow for the survey period is required. This estimate can be obtained from plant Monthly Reports of Operations (MRO) if the facility meter is considered reliable. It is usually advantageous to estimate a little low on the flow volume. This will result in a larger aliquot being taken whereas a high estimate may result in an insufficient volume of the sample.

## 2.4.3.1 Programming Samplers with Flow Meters for Sequential Flow

The object of sequential flow sampling using the model 2700 is to collect a sequential series of discrete equal volume samples in an identifiable series of sample bottles. As previously discussed, the Model 2700 will accept flow proportional inputs of a certain nature from an external flow meter. These electronic flow input signals are transmitted to the sampler at fixed increments of total flow, for example, every 10,000 gallons. That is, each time 10,000 gallons of liquid has flowed past the flow meter, a signal is sent to the sampler, which registers it as a single flow pulse. The Model 2700 Sampler can be programmed to totalize any number of flow pulses from 1 to 9999 before a sampling cycle is initiated. For example, if the sampler were programmed to totalize 5 flow pulses and each flow pulse represented 10,000 gallons of total flow, a sample would be collected each time 50,000 gallons (5 flow pulses of 10,000 gallons each) had flowed past the flow meter.

### 2.4.3.1.1 Programming Sampler with ISCO 2870 Flow Meter

- 1. Detailed programming instructions are provided in the ISCO 2700 sampler manual, pages 2- 43 through 2-46, and are listed in Table 6.
- 2. The volumetric unit/flow pulse switch may be set to 10, 100, or 1000 volumetric units.
- 3. The scaling constant determines the total gallons per volumetric unit. For example:

Scaling Constant 
$$X.XX + 0 = X1$$
  $X.XX + 1 = X10$   $X.XX + 2 = X100$ 

SCALING CONSTANT value establishes multiplying factor for ISCO Flow meter 2870. It is extremely important to note that the setting on the SCALING CONSTANT switches establishes the volumetric units for the flow meter.

For example, if the SCALING CONSTANT switches were set in terms of gallons per second, the flow rate would be measured and transmitted to an external recording device in gallons per second, flow would be totalized in gallons, and an associated sampler

would be paced in a flow proportional mode in gallons. The exponential portion of the setting on the SCALING CONSTANT switches also establishes a multiplying factor which must be applied to the reading on the TOTAL FLOW counter and the settings of the SAMPLER INITIATION SIGNAL switch.

Table 2.4 Set Up Example for ISCO 2870 Flow Meter

Ц	Set Up Example for ISCO 2870 Flow Meter Primary device: 90 degree v-notch weir
	Scaling constant (1 ft): 18.7 GPS = 1.87 +1
	Estimated flow: .450 MGD
	Total sample volume: 15.0 liters
	Number of samples: 45 (@ 300 mL/sample)
	450,000 divided by 45 samples + 10,000 gallons/sample
	10,000 gallons = interval between samples
	Set volumetric units/flow pulse switch at 100 (100 x scaling constant = $x.xx + 1 = 100 x 10 = 0$
	1,000 gallons/flow pulse)
	Set sampler for 10 flow pulses sample interval.
	Set sampler to collect 300 mL/sample
	Three samples/bottle

For example, consider a SCALING CONSTANT setting of 6.42 + 1 gallons per second. The exponential portion of this setting is:

$$+1 = 10^1 = 10$$

This multiplying factor of x10 must be applied to the items listed above. The reading on the TOTAL FLOW counter should be multiplied by 10. The possible settings of the SAMPLER INITIATION SIGNAL switch should also be multiplied by 10, resulting in possible settings of 100, 1,000, and 10,000.

## 2.4.3.1.2 Programming Sampler with ISCO 3230 Flow Meter

The volumetric flow pulse can be set to any volume. For example, the flow meter/sampler setup would be as follows:

- estimated flow: 0.450 MGD
- total sample volume: 15 liters (15 sampler bottles)
- number of sampling periods: 45 (15 bottles x 3 samples per bottle)
- 450,000 (0.450 MGD) divided by 45 samples = 10,000
- sample every 10,000 gallons
- ISCO 3230 flow meter flow pulse interval: 1,000 gallons
- set sampler initiation interval for 10 flow pulses

• set sampler to collect 300 ml per sample (three samples per bottle)

## 2.4.3.2 Automatic Sampler Sequential Flow Set-up Example

To program the sampler for automatic operation in a sequential flow mode, the use of the following set of step-by-step instructions is recommended. These instructions assume that the sampler is off and that the sample bottle tub has been properly prepared with 24 plastic or glass sample bottles.

Table 2.5 Program Instructions for ISCO MODEL 2700 Automatic Sampler

Table 2.5 Program Instructions for ISCO MODEL 2700 Automatic Sampler				
	INDICATOR			
STEP NO.	LIGHT ON	DISPLAY		
1. Press ON key. This places the sampler	1. None	1. Depending upon mode previously programmed,		
into the <b>standby</b> state.		rotates between four dashes ("") and the bottle		
		number ("-01-"*) or shows the dashes ("") only. If		
		sampling program previously completed, rotates		
		between four dashes ("") and "FULL".		
2. Press PROGRAM/STEP PROGRAM	2. MODE	2. Previously programmed mode ("3"*).		
key. This places the sampler into the	2	2. Tro nouszy programmou moue ( c ).		
program state.				
3. Enter on the numeric keypad Mode 2	3. MODE	3. "2".		
(SEQ. FLOW).				
4. Press ENTER VALUE key.	MODE	4. "2".		
5. Press PROGRAM/STEP PROGRAM	5. INTERVAL	1. Previously programmed interval in flow pulses		
key.	BETWEEN	("15"*).		
	SAMPLES			
6. Enter on the numeric keypad the desired	6. INTERVAL	6. "5".		
interval between samples in flow pulses (up	BETWEEN			
to 9999 flow pulses). For example, assume it	SAMPLES			
is desired to collect samples at 100,000				
gallon intervals, and each flow pulse				
represents 20,000 gallons-enter 5				
(100,000/20,000=5) on the numeric keypad.		0 1171		
7. Press ENTER VALUE key.	7. INTERVAL	2. "5".		
	BETWEEN SAMPLES			
See attached addendum sheet regarding delay to first sample.				
8. Press PROGRAM/STEP PROGRAM	8. NOMINAL	8. Previously programmed nominal sample volume		
key.	SAMPLE	in 10's of ml ("50"*).		
	VOLUME	0 117511		
9. Enter on the numeric keypad the desired	9. NOMINAL	9. "75".		
nominal sample volume in 10's of milliliters	SAMPLE			
(to a maximum of 99). For example, assume	VOLUME			
that it is desired to collect samples with a volume of 750 ml-enter 75 on the numeric				
keypad.				
10. Press ENTER VALUE key.	10. NOMINAL	10. "75".		
10. Press ENTER VALUE Key.	IU. NUMINAL	10. /5 .		

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CEED NO	INDICATOR	DICDI AN
STEP NO.	LIGHT ON	DISPLAY
	SAMPLE	
11 D DDOCD AM/CTED DDOCD AM	VOLUME	11. Por in all annual to the first time.
11. Press PROGRAM/STEP PROGRAM	11. Type of	11. Previously programmed type of suction line
key.	SUCTION LINE	("3"*) 12. "2".
12. Enter on the numeric keypad the	12. TYPE OF	12. "2".
number corresponding to the desired type of	SUCTION LINE	
suction line. For example, assume that a <sup>1</sup> / <sub>4</sub> "		
ID by 25' long suction line is being used-		
enter type 2 (1/4" X 25') on the numeric		
keypad.	13. TYPE OF	13. "2".
13. Press ENTER VALUE key.		13. 2.
14 P PROCEAN/GTOP PROCEAN	SUCTION LINE	14 P : 1 1: 6 :
14. Press PROGRAM/STOP PROGRAM	14. SUCTION	14. Previously programmed suction head in feet
key.	HEAD	("10"). 15. "12".
15. Enter on the numeric keypad the suction	15. SUCTION	15. "12".
head in feet, to a maximum of 20 ft. The suction head is the vertical distance from the	HEAD	
surface of the liquid source to the pump		
inlet. For example, assume that the suction head is 12 ft-enter 12 on the numeric		
keypad.		
16. Press ENTER VALUE key.	16. SUCTION	16. "12".
10. Fless ENTER VALUE key.	HEAD	10. 12.
17. Press PROGRAM/STOP PROGRAM	17.	17. Previously programmed multiplex mode ("2"*).
key.	MULTIPLEX	17. The viousity programmed multiplex mode (2 ).
icy.	MODE	
18. Enter the number corresponding to the	18.	18. "1".
desired mode of multiplex operation on the	MULTIPLEX	10. 1.
numeric keypad. For example, assume that	MODE	
no multiplex operation is desired-enter	MODE	
MODE 1 on the numeric keypad		
19. Press ENTER VALUE key.	19.	"1".
	MULTIPLEX	
	MODE	
20. Press PROGRAM/ STEP PROGRAM	20. NONE	20. Rotates between four dashes ("") and the
key. This returns the sampler to the standby		bottle number ("-01"*) of "FULL", if sampling
state.		previously completed.
21. Press the START PROGRAM/RESET	21. None	21. Rotates between the interval in flow pulses until
DISTRIBUTOR key. This places the		the first/next sample is collected ("0042"*) and the
sampler in the RUN state. If the display		bottle number into which this sample will be placed
previously had read "FULL", the distributor	l	<u> </u>
		("-01"*).
will automatically be repositioned to the		(-01 *).
		(-01 *).

This completes the programming of the Model 2700 Sampler in a sequential flow mode. Following the example, the sampler will collect the first 750 mL sample in the first sample bottle after 5 flow pulses (100,000 gallons) have been received from the external flow

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meter. The display will continue to rotate between the interval in flow pulses remaining until the next sample is collected (for example, "0004") and the bottle number into which the sample will be placed (for example, "-02-"). The flow pulse interval to the next sample is reset to the programmed value of 5 when the START PROGRAM/RESET DISTRIBUTOR key is pressed in step 21. As flow pulses are received from the external flow meter, the flow pulse interval to the next sample shown in the display will decrement to zero, at which point a sample will be collected, the display will be reset to the programmed value of 5 and the decrementing process will begin again as flow pulses are received. The 750 mL samples will continue to be collected at 5 flow pulse (100,000 gallon) intervals until the 24<sup>th</sup> and last sample bottle has been filled, at which time the sampler will shut off, and the display will read "FULL".

#### **2.4.4 MODEL 2700 ADDENDUM**

The Model 2700 software has been updated resulting in operational changes to the controller. These changes are present on units beginning with the serial number of 5349-001 or on all units with software revision 7.23 or greater.

The following controller operations will be affected:

- 1. While programming modes 2, 4, and 6, the sampler will now accept a value for "DELAY TO FIRST/NEXT SAMPLE", this will cause the sampler to delay a fixed amount of time before accepting flow pulses. If it is desired that the unit start sampling immediately after the "START PROGRAM/RESET DISTRIBUTOR" key is depressed, a zero should be entered at the 'DELAY TO FIRST/NEXT SAMPLE" prompt. If a time delay was entered for the "DELAY TO FIRST/NEXT SAMPLE", when the program is started, the four digit display will alternate between the time delay left and "HHHHH" until the time expires.
- 2. In all modes that accept a value for "DELAY TO FIRST/NEXT SAMPLE", the unit will now accept a zero. This is useful in the case of modes 2, 4, and 6 where it is desired that the sampler start sampling immediately. In the other modes, entering a zero will cause the delay to the first sample to be set at the number entered at the "INTERVAL BETWEEN SAMPLES" prompt.
- 3. When ordering a replacement eprom be sure to order part number 60-2703-152 to receive the 2700 EPROM (Rev. 7.25). Do not order part number 60-2703-134 as shown in the replacement parts list or the 2700 EPROM (Rev. 4) will be sent.

#### 2.4.5 CHURN USAGE FOR MIXING AND SPLITTING SAMPLES

The Surveys section uses a fifteen liter polyethylene churns for mixing, homogenizing, and splitting samples for most parameters except for organic toxic pollutants. Aliquots are proportioned, according to flow measurement, into the churn at the end of the sampling period. Calculations for total sample volume should allow for an excess of 3 liters of sample. The excess volume barely clears the outlet spigot located on the side of the churn and should be discarded after the actual sample has been mixed and drawn into the sample bottles. To

insure thorough mixing of sample, at least 20 strokes of the churn handle should be effected before the first sample is drawn. Agitation during sample removal is also necessary to prevent settling and keep the sample homogenous. All churns should be cleaned with hot water and detergent and thoroughly rinsed after each survey.

#### 2.4.6 ISCO SAMPLER EQUIPMENT MAINTENANCE

In order to insure adequate confidence levels in the survey activities, the maintenance of all of the ISCO Samplers are of prime importance. A logbook has been set up for these samplers. Maintenance and calibration instructions are contained in the manufacturer supplied manuals. The following duties always apply to all personnel using the ISCO automatic samplers:

- 1. Use all sign in/out log books. The personnel in charge of the automatic samplers will have the responsibility to make sure that you do. Relate verbally any equipment problems you might have had to the person in charge of that equipment and notate this problem in the log.
- 2. Make sure desiccant is dry when checking out or in. Change if pink. Clean equipment promptly. DO NOT LEAVE IT SITTING AROUND. Acid rinse pump tubing with 50% HCl. Then follow with a distilled water rinse. Wash sampler bottom tub and any other parts which came into contact with sampled water. If necessary, wash with detergent and wipe down the outside of the sampler top to bottom. Wash all dirty bottles, and/or churns, in the laboratory bottle washer.

## 2.5 SAMPLING PROCEDURES FOR TOXIC SUBSTANCES

#### 2.5.1 Introduction

A priority pollutant toxic organic chemical survey is a sampling for those contaminants for which the U.S. EPA has developed water quality and human health criteria recommendations. Generally referred to as a "toxic scan" survey, it is a particular sampling of those designated 126 "priority pollutant compounds." These parameters have been separated into eight categories based on the different specific laboratory analysis, yet each dictating various means of field collection and handling. These categories are metals, halogenated volatile organics, nonhalogenated volatile organics, aromatic organics, base/neutral fraction, organochlorine pesticides, phenols, and polychlorinated biphenyls (PCBs).

#### 2.5.2 METHODS AND FORMS OF TOXIC SAMPLING

The terms "manual sampling", "automatic sampling", "grab sample" and "composite sample", all retain the same meaning with respect to toxic parameters as they do with the more traditional parameters. Each of the eight categories of priority pollutants mentioned in the introduction may be sampled in the manner defined by the above terms. "Sampling location" is likewise dictated by the same criteria that govern the more traditional parameters.

#### 2.5.3 EQUIPMENT PREPARATION

## 2.5.3.1 Automatic Sampler Preparation for Composite Sampling Collection

Currently, the ISCO Sampler, Model 2700, is used to collect composite samples of these toxic parameters: Base/Neutral Fraction, Organochlorine Pesticides, Phenols, and PCB's. The sample is collected in a single glass container at equal time intervals. Proper sampler tubing and strainer preparation are extremely important aspects in preventing interference with and contamination of samples. Only 3/8-inch I.D. medical grade silastic tubing manufactured by Dow Corning is the standard approved tubing used in the peristaltic sampler pumps. Teflon tubing is the approved sampling line-tubing used, but medical grade silastic tubing can also be used for the sampling line-tubing. The strainer is made of stainless steel. All surfaces which have contact with the sample must be rinsed with acetone to remove any moisture or water followed by a petroleum ether rinse to make any residual organic toxics soluble for removal. These solvents must be passed through all sampler tubing and over the strainer. The ends of the tubing are then sealed with aluminum foil and secured by taping for protection against contamination until usage at the sampling site. The strainer is similarly protected by wrapping in aluminum foil after solvent rinsing. Because of hazardous fumes, this solvent-rinsing procedure should be conducted under ventilation hoods. Presently this rinsing is carried out by the ISDH Laboratory.

## 2.5.3.2 Sample Container Preparation

The base/neutral fractions, pesticides, phenols and PCBs are collected with the ISCO samplers. The present method is to pump the sample into a 2½gallon glass jug and then divide as needed into solvent rinsed, one liter glass containers. Good quality control requires that acetone, petroleum ether, hexane, or methylene chloride be used to solvent rinse sampling containers for collection of the aforementioned parameter groups. If a chlorinated sample is to be collected, sodium thiosulfate crystals equal to ~.008 percent, approximately one crystal/liter of sample, should be added to the jug to deactivate chlorine. Once sampling is completed, the sample is shaken up thoroughly and poured into the one-liter glass solvent rinsed bottles and then sealed with Teflon lined caps. If split samples are requested by the inspected facility, then additional bottles are poured according to facility analytical needs.

Halogenated volatile organics, nonhalogenated volatile organics, and aromatic volatile organics are all collected in 40 mL purgeable vials with a Teflon lined septum cap. These containers are prepared by laboratory staff along with two field blanks that must accompany all samples for quality control checks. The chemical instability of these parameters prohibits composite sampling. Thus, individual discrete samples are collected at times and intervals dictated by facility operation changes or other survey conditions. Volatile samples are collected in two 40 mL vials at each time and sampling location to

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permit quality assurance by duplication of analysis in those instances where parameters are found just above detection levels. It is important to remember to account for these extra vials when projecting the number required for a survey. As with the glass composite jugs, sodium thiosulfate crystals must be added to the vials when sampling chlorinated effluents.

Sludge samples for all toxic parameters except the volatile organics must be collected in one pint glass solvent rinsed jars with aluminum foil lined caps. Two jars per sludge sampling site will assure sufficient sample. Sludge sample collection for VOCs is collected in the 40 mL purgeable vials simply by pushing the vial directly into the sludge forcing out all air and bubbles in the process. One vial per sampling site is sufficient for analyses of all VOCs. If the sludge is in a liquid state, more sample might be needed.

All heavy metal parameters and mercury are composited into one two-liter plastic bottle. A one-liter plastic bottle is used for each cyanide composite sample to be collected. Preservatives are added on site. Standard blanks should also be prepared, using the same preservatives.

## 2.5.3.3 Manual Sampling Preparation

Solvent rinsed stainless steel buckets should be used in these instances where manual sampling is required. The buckets should be covered by aluminum foil immediately after solvent rinsing in the laboratory to prevent contamination during transport to the sampling site.

#### 2.5.4 ON-SITE SAMPLE COLLECTION

## 2.5.4.1 Automatic Sampler Set Up for Compositing Samples

Refer to the ISCO sampler, Model 2700, operation manual for basic information on set up. Currently, only equal volume composites for the base/neutral fraction, organochlorine pesticides, phenols and PCBs are collected in the single glass jug using an automatic samplers. When assembling a sampler for toxic sampling, the tubing should be extended two (2) inches into the amber jug. If tubing is more than two (2) inches, it is possible that when the jug is nearly full, sample will be withdrawn during the postpurge cycle. The sampler tubing and strainer should be unwrapped just prior to set up, with care being given to handling to minimize contamination. One hour sequences for aliquot collection is the most commonly used programming interval. The staff person should evaluate the circumstances at a particular sampling site and determine the intervals needed to make the most representative sample. Upon completion of sampler programming, ice should be added to the sampler tub to preserve the sample at 4°C during collection.

## 2.5.4.2 Collection of Volatile Organic Samples

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Ideally, VOCs should be sampled below the surface of a liquid source to minimize volatilization, which would occur by turbulence of the liquid entering the vials. In practical sampling situations this is difficult to achieve without some aeration while transferring the sample from the manual collection apparatus to the sample vial. If at all possible, the sample should not be skimmed from the surface of the sampling site or poured as a means of transferring the sample. A good approach is to use a solvent rinsed glass, stainless steel, Teflon, or silastic pump tube and, by placing one end into the liquid source, seal the other end with your thumb or clamp, place the sampling end into the bottom of the vial, slowly relieve the pressure on the other end and allow the sample to fill the vial. Slowly withdraw the tube while allowing the tube to drain, so that the vial is filled and a convex meniscus develops over the top. The septum inside the cap is rather loose fitting and can be easily lost or contaminated by handling or dropping. The Teflon side of the cap must be placed inward. After the cap is secure, the vial should be inverted and tapped to determine if the sample is completely free of air bubbles. The collector should practice this sampling technique numerous times so that proficiency is developed in avoiding entrapment of air bubbles. The ISCO sampler model 6000 can also be used in collection of VOCs.

When a 24-hour composite sampling survey is warranted, VOCs are commonly collected once per eight hour shift. Again this interval may vary depending on the collectors evaluation of survey conditions.

## 2.5.4.3 Collection of Sludge and Sediment Samples

Surveys staff are sometimes required to sample sewage and industrial sludge, and bottom sediments of waterways. Collection and analysis of these materials can give important information about the present and past status of the overlying water column with regard to its chemical constituents, especially those that may persist for longer periods of time.

Sewage and industrial sludge samples as well as stream sediment samples for all toxic parameters except the volatile organics must be collected in one pint glass solvent rinsed jars with aluminum foil lined caps. Sludge and sediment sample collection for VOCs is in 40 mL purgeable vials, simply by pushing the vial directly into the sludge/sediment forcing out all air and bubbles in the process. No preservatives are needed for sludge/sediment samples. Samples should be placed on ice or put into a refrigerator and stored at 4°C until delivery to the laboratory.

When sludge sampling, consider where the sample should be collected. In most cases, this will be the oldest or final dry state just before ultimate disposal. Solid or dry sludge should be packed into the purgeable vials leaving as little air space as possible. A solvent rinsed stainless steel utensil is useful for this purpose. A few situations may warrant collecting VOCs at a liquid sludge source before dewatering. The liquid source would give analysis of levels in the ripe sludge since VOCs disperse easily in the drying process. All other priority pollutant sludge samples are collected in one pint solvent rinsed jars either by

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scooping of a dry sample or collection at a liquid source. Sludge collection is an obvious situation where protective gloves should be worn.

Collection of stream sediment samples for all analyses, with the exception of VOCs, are in glass pint jars rinsed with either hexane or acetone solvent. Jars should be sealed with either aluminum foil or Teflon lined screw-on lids. A suitable site for sediment collection is one that does not contain large areas of sand or gravel. In most cases, sediment samples can be collected by hand using a solvent rinsed stainless steel scoop and mixing pan. Sediment is scooped up from several areas across the stream bottom, put into the mixing pan, and thoroughly stirred in order to acquire a homogenous sample. A portion of this mixture is then put in a solvent-rinsed glass jar and as much of the water as possible is poured from the jar, taking care not to lose any of the sediment.

Samples to be analyzed for purgeable organics (VOCs) are collected in 40 mL screw cap vials fitted with a Teflon lined silicon septum. Usually the sediment sample can be collected in the manner described above, but there are some additional procedures to be followed. The vials are filled as full as possible with the sediment, and then the cap, with the Teflon face of the septum down, is placed on the vial. Care must be taken to prevent air being trapped in the vial. Staff members have found that holding the vial beneath the surface of the water while screwing the cap on usually prevents air from being trapped in the vial. If this is not possible, water should be added to the vial to form a convex meniscus at the vial mouth, and the cap carefully screwed on the vial. After sealing, the vial should be checked for air bubbles by inverting it. If air bubbles are present, the vial should be resealed by removing the cap, adding more water to form the convex meniscus and replacing the cap as before.

If sampling of the sediment cannot be done by hand, some type of mechanical sampler such as a Ponar is used. The sampler is cleaned and solvent rinsed before each sample is taken. Care must be taken to lower the sampler slowly to the bottom so as to minimize the disturbance of the surface layers of the sediment. The sample is then brought to the surface and the sediment either collected as described above, or the sediment is pushed into the sample jar using a clean, solvent rinsed piece of aluminum foil.

If a core sampler is used, the sampler is cleaned and solvent rinsed before each use. Samples are placed in solvent rinsed pint glass jars with aluminum foil or Teflon lined lids. If samples from different layers of the sediment core are required, the core is wrapped in solvent rinsed aluminum foil. Care must be taken to preserve the vertical integrity of the core sample in this case.

### 2.5.4.4 Collection of Metals, Mercury and Cyanide Samples

Metals and mercury are collected together by means of an ISCO Automatic Sampler and flow weighted by measuring the individual discrete samples in a glass graduated cylinder in proportion to the flow measurements. The sample is composited into a two-liter plastic bottle, and preserved with 10 milliliters of concentrated nitric acid. Samples are preserved at the end of the 24-hour collection period rather than each individual sampler bottle at the time of set-up. This is due to the corrosive affect of the nitric fumes on metal parts inside the sampler. Automatic samplers are also used in cyanide sampling, but each individual one-liter sampler bottle should be preserved at the time of set-up with one milliliter of 50 percent sodium hydroxide (pH 12). For chlorinated samples, ascorbic acid (0.6g) should also be added during set-up to nullify the destructive affect of chlorine on the cyanide. The flow-weighted cyanide sample aliquots are composited into a one liter plastic bottle at the end of the 24-hour sampling period.

#### 2.5.5 SPLIT SAMPLING METHODS

Sometimes a facility will request a "split sample." Splitting volatile organic parameters is done by simply collecting additional purgeable vials during each sampling event. Metals, mercury, and cyanide samples can be flow weighted and split by first measuring the samples in proportion to flow and then pouring them into a medium density polyethylene churn for thorough mixing. Then while churning, draw off individual samples. Due to the cyanide sample being preserved at set-up, churning and splitting must be done separately from the metals and mercury sample. All other "toxic" parameters can be split by means of pouring the composited sample from the glass jug.

#### 2.5.6 TRANSPORT AND STORAGE OF SAMPLES

All samples should be iced while being transported to the laboratory. Care should be taken to not submerge the purgeable vials in the ice, because it is possible for water to move through the septum cap.

## 2.6 FLOW MEASUREMENT

#### 2.6.1 Introduction

For the most part, the following are summaries of the methods employed in the field and are not intended to replace the documents mentioned above. Each of the instruments and/or methods utilized by the Surveys section personnel are discussed in some detail below.

# 2.6.2 PRIMARY FLOW DEVICES COMMONLY USED FOR MEASURING FLOW IN WASTEWATER TREATMENT FACILITIES

#### 2.6.2.1 Weirs

- 1. The standard contracted rectangular weir
- 2. The standard suppressed rectangular weir

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- 3. The standard Cipolletti weir
- 4. The V-notch weir

## 2.6.2.2 Weir Design Requirements

To assure accurate discharge measurement, there are certain general weir design requirements that apply to all types:

- 1. The weir should consist of a thin plate 1/8 to 1/4 inch thick with a straight edge or a thicker plate with a downstream chamfered edge. The upstream sharp edge prevents the nappe from adhering to the crest. Knife edges should be avoided because they are difficult to maintain. However, the upstream edge of the weir must be sharp with right angle corners, since rounded edges will decrease the head for a given flow rate.
- 2. The upstream face of the weir should be smooth and perpendicular to the axis of the channel in both horizontal and vertical directions. The crest of the weir should also be exactly level to insure a uniform depth of flow.
- 3. The connection of the weir to the channel should be waterproof. Therefore, the joint between the weir plate and channel should be packed with chemically inert cement or asphalt type roofing compound.
- 4. The length of the weir crest or the notch angle must be accurately determined, because the percentage error in measured flow rate will be proportional to the error in determining these dimensions.
- 5. The weir should be ventilated, if necessary, to prevent a vacuum from forming on the underside of the nappe.
- 6. The height of the weir from the bottom of the channel to the crest should be at least 2 times the maximum expected head of liquid above the crest. This is necessary to lower the velocity of approach. The weir height should never be less than 1 foot.
- 7. The approach section should be straight upstream from the weir for a distance of at least 20 times the maximum expected head of liquid, and should have little or no slope.
- 8. The crest must be set higher than the maximum downstream elevation of the water surface, otherwise a submerged flow condition will occur instead of the free flow condition required for reliable flow measurement.
- 9. The device for measuring the head (flow meter) should be placed upstream at a distance of at least 3 times the maximum expected head on the weir and should be located in a quiet section of the channel away from all disturbances, preferable in a stilling well. Also, the zero point of the head measuring device must be set exactly level with the weir crest.
- 10. The crest of the weir must be kept clean. Fibers, stringy materials, and larger particles tend to cling to the crest and should be removed periodically. The

upstream side of the weir should also be periodically purged of accumulated silt and solids.

- 11. The weir size should be selected only after preliminary studies have determined the expected flow rates in the channel in question. The Manning formula can sometimes be used to estimate the flow rate in open channels.
- 12. The cross-sectional area of the approach channel should be at least 8 times that of the nappe at the crest for a distance upstream of 15 to 20 times the head of the crest. This is necessary to minimize the velocity of approach. The approach channel should also permit the liquid to approach the weir in a smooth stream free from turbulence, and the velocity should be uniformly distributed over the channel; this may be accomplished through the use of baffle plates if necessary.
- 13. If the weir pool is smaller than defined by the above criteria, the velocity of approach may be too high and the head reading too low. Weirs should be installed and maintained to make the velocity of approach negligible, but where this is not possible, appropriate corrections should be made.

#### 2.6.2.3 Flumes

- 1. The Palmer-Bowlus flume
- 2. The Leopold-Lagco flume
- 3. The Parshall flume

A Parshall flume is a specially-shaped open-channel flow section which may be installed in a channel, lateral, or ditch to measure the rate of flow of water. The constricted throat of the flume produces a differential head that can be related to discharge. The crest followed by the downwardly sloping floor, gives the Parshall flumes its ability to withstand relatively high degrees of submergence without affecting the rate of flow. The converging upstream portion of the flume accelerates the entering flow, thereby essentially eliminating the deposition of sediment which would otherwise reduce measurement accuracy. Velocity of approach, which often is a detrimental factor in the operation of weirs, usually has little effect on the rate of discharge of the flume. The approaching flow should, however, be well distributed across the channel and should be relatively free of turbulence, eddies, and waves. Flumes should not be located where they are subjected to high velocity due to pump cycles or force mains.

Discharge through a Parshall flume can occur for two conditions of flow. The first, free flow, occurs when there is insufficient backwater depth to reduce the discharge rate. The second, submerged flow, occurs when the water surface downstream from the flume is far enough above the elevation of the flume crest to reduce the discharge. For free flow, only the head (two-thirds the distance back on the converging section) at the upstream gage location (Ha) is needed to determine the discharge. The free-flow range includes some of the range which might ordinarily be considered submerged flow because Parshall flumes tolerate 50 to 80 percent submergence before the free-flow rate is measurably reduced. For

submerged flows, that is, when submergence is greater than 50 to 80 percent, both the upstream head (Ha) and downstream head (Hb) are needed to determine the discharge. Hb is the measurement 1" to 2" from the downstream side of the throat at a depth the same as the floor of the converging section.

Presently, there is no standard design plan for either the Palmer-Bowlus or Leopold Lagco flumes. Each manufacturer has developed their own design and formula for each flume for its specific use. To measure flow in such a flume, it is necessary to obtain design criteria for that flume.

## 2.6.2.3.1 Flume Design Requirements

To assure accurate discharge measurement, there are certain general requirements for the installation of flumes that apply to all types and size of flumes:

- 1. A flume should be located in a straight section of the open channel, without bends immediately upstream.
- 2. The approaching flow should be well distributed across the channel, and relatively free of turbulence and waves.
- 3. Generally, a site with high velocity of approach should not be selected for a flume installation. However, if the water surface just upstream is smooth with no surface boils, waves, or high velocity current concentrations, accuracy may not be greatly affected by velocity of approach.
- 4. Consideration should be given to the height of upstream banks with regard to their ability to sustain the increased depth caused by the flume installation.
- 5. Although less head is lost through flumes than over weirs, it should be noted that significant losses may occur with large installations.
- 6. The possibility of submergence of the flume due to backwater from downstream should also be considered, although the effect of submergence upon the accuracy of most flumes is much less than is the case with weirs.

#### 2.6.3 ISCO FLOW METER

The Model 2870 and 3230 Flow Meters are a compact, easily transported devices intended to measure and record flow rate or level in an open channel. These flow meters are normally used in conjunction with some type of primary measuring device. This could be a weir, flume, or any other open channel flow situation for which a known relationship exists between level and flow rate. These two meters measure the liquid level in the primary device using a bubbler system, and electronically converts the level into a corresponding flow rate. Flow rate is permanently recorded on an integral strip chart recorder and total flow is continuously displayed on a totalizer. Alternately, liquid level only may be recorded.

#### 2.6.3.1 Instructions for ISCO Flow Rate Measurement Model 2870

(Refer to the ISCO Instruction Manual - Model 2870 for additional operating instructions)

- 1. Turn the POWER switch off and remove the Primary Device characterization Module from the flow meter.
- 2. Select Device No. 1 on the module's DEVICE NO. switch. This corresponds to the device cell containing level flow rate information for the primary device being used, a 6-inch Palmer-Bowlus flume. Then, set a value of 5.16 -1 on the module SCALING CONSTANT switches. This is the scientific notation of the flow rate (0.516 cubic feet per second) through a 6-inch Palmer-Bowlus flume at a 0.469 ft. head, expressed in the desired volumetric units of cubic feet. The setting establishes the volumetric units of the system as cubic feet, and also establish a multiplying factor of 0.1 (10-1) for the TOTAL FLOW counter and the SAMPLER INITIATION SIGNAL switch.
- 3. Reinstall the module in the flow meter.
- 4. Place the POWER switch in the ON position. For highest accuracy, allow the flow meter to warm-up for 5 to 10 minutes after being turned on, providing the level measurement system time to stabilize.
- 5. Place the DISPLAY SELECT switch in the SCALING CONSTANT setting. A setting of 5.16-1 should be shown on the display. If desired, the maximum head programmed into the module may also be verified by placing the DISPLAY SELECT switch in the MAXIMUM LEVEL position. A level of 0.469 should be shown on the display.
- 6. Adjust the BUBBLE RATE valve to obtain a bubble rate of approximately 1 to 2 bubbles per second.
- 7. Place the DISPLAY SELECT switch in the CALIBRATE RECORDER ZERO position and confirm (or set) the zero position of the recorder pen.
- 8. Place the DISPLAY SELECT switch in the RECORDER FULL-SCALE position.
- 9. Select the desired flow rate span on the RECORDER MODE/SPAN switch. Since it is desired to obtain precise flow rate information from the chart, the AUTOMATIC SCALING method of selecting the flow rate span should probably be used. This method results in "even" chart divisions making subsequent reading of the chart much easier. The value set on the module's SCALING CONSTANT switches is 5.16 -1 (5.16 X 10-1 = 0.516) cubic feet per second. For the AUTOMATIC SCALING method of selecting the flow rate span, the three available RECORDER MODE/SPAN switch settings are:
- 10. NORMAL = 1.0 CFS
- 11. EXPAND 1 = 0.50 CFS
- 12. EXPAND 2 = 0.20 CFS
- 13. With the DISPLAY SELECT switch in the RECORDER FULL-SCALE position, the full-scale values associated with the three AUTOMATIC SCALING positions of the

RECORDER MODE/ SPAN switch may be observed on the display as the switch is rotated through the positions. The NORMAL and EXPAND 1 positions of the switch would assure that the chart cannot go off-scale, since the full-scale spans associated with these positions, 1.0 CFS and 0.5 CFS, respectively, are greater than the maximum expected flow rate of 0.38 CFS. However, since it is known that the average flow rate for the majority of the day is only 0.15 CFS, a full-scale span of 1.0 CFS would result in a chart record for the majority of the day at only 15 percent of full-scale limiting the resolution of the chart record. To increase the resolution of the flow rate record for the majority of the day, one of the two lower full-scale spans could be selected. In the EXPAND 1 position, the full-scale span of 0.5 CFS would assure that the maximum flow rate of 0.38 CFS would remain on-scale, and still maintain adequate resolution at the average flow rate of 0.15 CFS. In the EXPAND 2 position, the full-scale span of 0.2 CFS would maximize the resolution for the average flow rate of 0.15 CFS, and allow the automatic over-ranging feature of the flow meter to record the daily peak flow of 0.38 CFS. In this case, either the EXPAND 1 or EXPAND 2 positions of the switch would be suitable; to illustrate the automatic over-ranging feature of the flow meter, the EXPAND 2 position will be chosen for this example. Thus, place the RECORDER MODE/SPAN switch in the EXPAND 2 position, establishing the recorder full-scale as 0.2 CFS.

- 14. Place the CHART SPEED switch in the 1 INCH PER HOUR position.
- 15. Using the Chart Manual Advance thumb wheel, synchronize the pen position on the chart with the real time of day.
- 16. Assuming the a moderate amount of sediment will accumulate upstream of the flume at the level measuring point, select a moderate automatic purge rate by placing the PURGE switch in the 15 MINUTES position.
- 17. To conserve power, place the PEN OPERATION switch in the NORMAL position.
- 18. Place the DISPLAY SELECT switch in the LEVEL position to allow the level currently being measured by the flow meter to be shown on the display.
- 19. Using the LEVEL ADJUST control, adjust the level indicated on the display to match the actual level at the measuring point in the flume. Note that with a Palmer-Bowlus flume, the zero reference level is not the invert of the sewer, but rather is the floor of the flume.
- 20. Since there is no sampler in the system, the SAMPLER INITIATION SIGNAL switch may be left in any position.
- 21. Depress the TOTAL FLOW counter to reset the counter to zero. The volumetric units of cubic feet may be written in the VOLUMETRIC UNITS box to the side of the counter. The multiplying factor of 0.1 may also be written in the space provided.

#### 2.6.3.2 Instructions for ISCO Flow Rate Measurement Model 3230

(Refer to the ISCO Instruction Manual - Model 3230 for additional operating instructions)

The ISCO model 3230 flow meter allows for programming of multiple primary flow devices without the need of the user to program in specific flow equations or scaling constants. Programming is very "user friendly" with on-line menus. Programming involves "stepping through" 12 different program steps.

## 2.6.3.3 Programming Flow Equations With the ISCO Model 3230 Flow Meter

#### 2.6.3.3.1 Weirs

Rectangular weirs without end contractions are linear. Rectangular weirs with end contractions are not linear.

All rectangular weirs without end contractions have the following flow equation:

CFS = 3.330LH1.5

WHERE: L = Weir Crest Length

H = Maximum Head

## 2.6.3.3.2 Example

To calculate the flow equation for a 12 foot crest length rectangular weir without end contractions, take the flow equation for the 1 foot crest length rectangular weir without contractions, and multiply 3.330 by 12 which equals 39.96. This is the flow equation for a 12 foot rectangular weir without end contractions provided the maximum head is 1 foot. The display on the 3230 ISCO flowmeter will look similar to this:

$$CFS = 1.000 \text{ H} ^2.00 + 001.000 \text{ H} ^2.00$$

Then punch in 39.96, and then punch in 1.5 for the exponent of the H (maximum head). The part of the equation after the "+" sign is not used for these purposes because it does not pertain to weirs and flumes. Since the part of the equation after the "+" sign is not important to this use, make this part of the equation equal zero. This is done by setting the whole integer to zero and setting the power to one. The display should then look like this:

$$CFS = 39.96 \text{ H} ^ 1.5 + 0.000 \text{ H} ^ 1.00$$

After this equation is set up, the next program step will be to select the value for the maximum head. Punch in the maximum head and the flow meter will automatically adjust the previously entered equation for that given head.

Setting up a flow equation for a rectangular weir with end contractions is a little different. This type of weir is not linear, but it is not difficult to determine. All

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rectangular weirs with end contractions have the following flow equation:

CFS = 3.330 (L-0.2H)H1.5

WHERE: L = Crest Length of Weir.

H = Maximum Head

Once the flow equation is determined, plug in the result and follow the same procedures as previously mentioned above making sure that the part of the equation after the "+" sign equals zero.

V-notch weirs have different flow equations for each particular type. Once the flow equation is determined for the particular v-notch weir in question, just plug in the result and follow the same procedures as previously mentioned above, again making sure that the part of the equation after the "+" sign equals zero.

#### 2.6.3.3.3 Flumes:

All flumes have different equations for each particular type. The Parshall flume is the most common type of flume you will encounter. Once you determine the flow equation for the particular flume you are working on, just plug in the result and follow the same procedures as previously mentioned above, again making sure that the part of the equation after the "+" sign equals zero.

#### 2.6.3.4 Conclusion:

Most of the problems that are experienced will be with "odd" sized rectangular weirs, both with and without end contractions. Since the ISCO 3230 flow meter standard program menu will not allow programming for over a 10 foot weir crest length, the flow equation must be entered manually in order to set up a weir with a crest length greater than 10 foot. All other "normal" sized primary flow devices (v-notch weirs, Parshall flumes, etc.) should be able to be programmed using the standard program menu.

#### 2.6.4 MAINTENANCE AND CALIBRATION OF ISCO FLOW METER EQUIPMENT

#### 2.6.4.1 General

In order to insure adequate confidence levels in the survey activities, the maintenance of all of the ISCO Flow Meters are of prime importance. A log book has been set up for these Flow Meters. Maintenance and calibration instructions are contained in the manufacturer supplied manuals.

The following duties always apply to all personnel using the ISCO Flow Meters.

1. Use all sign in/out log books. The personnel in charge of the ISCO Flow Meters will

have the responsibility to make sure that you do. Relate verbally any equipment problems you might have had to the person in charge of that equipment and notate this problem in the log.

- 2. **Check desiccant for dryness**. Do not forget their intakes; change if necessary; check bubbler rod and line to make sure they are clear; check pen and paper.
- 3. **Check all bubbler rods**; make sure they are clear and free of cracks.

## 2.6.4.2 Calibration of Isco Flow Meters

The calibrations of the ISCO flow meters are performed in the field and in the laboratory. Surveys section staff can check the calibration in the field by timing with a stop watch the totalizer counter which is measured in gallons per second. Also, the electronic readings can be checked for accuracy by comparing the readings to a discharge table. When a discrepancy occurs, the flow meter is then set-up in the laboratory and run under controlled conditions.

The method of calibration in the laboratory is to set the flow meter in a constant amount of water with the meter set for a certain primary device. The meter is allowed to run for 24 hours and then the totalizer is compared to the discharge table for the level and primary device programmed into the meter. If the percent difference is greater than one or two percent the flow meter is sent back to the manufacturer.

#### 2.6.5 Measuring Stream Flow Using Current Velocity Meters

Personnel of the Office of Water Quality also measure stream flow using four types of current meters. These are the Price AA, and the Marsh-McBirney Models 201, 201D, and 2000 meters. The proper way to measure flow in a stream and the operation of each of these meters is discussed below. Please consult the appropriate flow meter manuals for an in depth discussion of meter operation.

## 2.6.5.1 Price Current Velocity Meter

This instrument consists of a cup-type bucket wheel which rotates in flowing water and a device for determining the number of revolutions. As the bucket wheel rotates, an electrical contact is closed on either a single-contact cam or a penta gear. If a headset or counter is attached along with a battery series, a signal is produced each time the bucket wheel completes a revolution. If the headset is connected to the penta-contact post, a signal is produced once every five revolutions. The penta-contact is very useful in fast water.

The velocity at the point of the current meter is measured by counting the number of signals (revolutions) in a specified time interval. Thus, a standard piece of equipment accompanying the use of a current meter is a stopwatch. Each meter is calibrated by the supplier and an equation for the relationship between velocity and revolutions per unit time

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derived. For most Price meters, the meter is supplied with a rating table, which shows the velocity for a given number of revolutions in a given time interval. Forty seconds is the smallest time interval listed on the rating table. This time interval is required to obtain a time-average velocity at the point. The user would be well advised to memorize the "stop counts" in the columns of the rating table as stopping the count in some intermediate number of revolutions (27, for example) negates the use of the table and requires the use of an equation to calculate the velocity.

In order to ensure consistent accuracy with a current meter, good preventive maintenance is a must. For all vane-type meters such as the Price, a most important maintenance item is the protection of the pivot and the pivot bearing. The pivot assembly provides a low-friction surface on which the bucket wheel is supported. If the pivot becomes blunted, or the pivot bearing damaged, the resistance increases and the meter will give low velocity readings. The greatest potential for damage occurs when a meter is transported with the pivot bearing and pivot in contact. On the Price meters, a raising nut is provided. When screwed down, the raising nut lifts the pivot bearing off the pivot and prevents contact. Whenever a Price meter is transported, if only across the river, the raising nut should be screwed down. This pivot may be replaced by the operational pivot by loosening the set screw at the front of the yoke and slipping one pivot out and the other in. Do not attempt to measure velocities with the travelling pivot in. Likewise, don't transport one of these meters with the operational pivot in.

Prior to and immediately following each use, the components of the meter should be cleaned and lubricated. A light, water-resistant oil should be used for a lubricant. Key oil for clarinets has been found to be a good, cheap lubricant. Oil should be applied to the pivot and pivot bearing, the penta gear and penta gear bushings, and the bearing lug. If measurements are made in silty or turbid water, the meter should be oiled frequently during its use.

The condition of the bearings should be checked prior to each use by a "spin test". With the shaft in a vertical position and cups protected from wind currents, the cups are given a quick spin. If the meter is in good condition, the cups should not stop spinning for at least three minutes. If the duration of spin is more than 1 minute, the meter may be used for all but very low velocities (less than one foot per second). A spin of less than one minute indicates that the instrument should be reconditioned.

For fairly deep or fast water, the Price type meter is usually the most practical instrument. For depths of less than about 0.5 feet (15 cm) the Marsh McBirney meter is more appropriate.

Current meters are suspended by a sounding system which allows concurrent measurement of depth and velocity. For shallow, wadeable rivers, the most convenient system is a top-setting wading rod. The top-set rod has a main column, 1/2 inch, hexagonal stock

which is graduated in 0.1 foot increments for measuring the depth. Interval markings follow the convention of a single mark every 0.1 foot, a double mark for each 0.5 foot increment, and a triple mark at each whole foot increment.

For most suspension systems, the 0.2, 0.6, and 0.8 depths must be calculated from the total depth as determined by sounding, i.e., the meter (or sensor) will be positioned on the wading rod (or other suspension) at either 0.2, 0.6, or 0.8 of the depth as measured from the surface. However, the top-set wading rod has a feature which allows the current meter or sensor to automatically be set at the 0.6 level. The depth of the vertical is read on the hexagonal sounding rod. Then the meter or sensor is placed in the 0.6 depth position appropriate for the measured depth by the meter positioning rod. Example: if the depth is 1.4 feet, the "1" mark of the meter positioning rod is set even with the "4" mark on the grip of the wading rod. To move the meter positioning rod, the brake must be released by pushing the brake in toward the grip with the thumb. When releasing the brake, hold on to the positioning rod so that the meter does not slam into the ground. When the positioning rod is in this position, the meter will be suspended exactly 0.84 feet below the surface, which is 0.6 times 1.4, the depth of the vertical.

## 2.6.5.2 Marsh-McBirney Model 201, 201D, and 2000 Current Meters

These meters consist of a sensor probe with attached cables and an electronic processor with a panel meter readout. The probe consists of an electromagnet inside a molded plastic housing with a pair of electrodes spaced 180° apart on the sensor surface. Water flowing around the sensor probe interacts with the electromagnetic field to produce a small voltage in the water near the probe which is sensed by the electrodes. This extremely small voltage is amplified, demodulated, filtered, and displayed on the panel meter.

#### 2.6.5.2.1 Operation

- Open carrying case and set selector switch to the CAL position. The meter should read in the calibration sector of the meter scale on model 201 meters.
   On the model 201D meters the digital readout should fall between 9.8-10.2.
   This indicates the batteries are good and the instrument is operating properly. If the meter needle does not come within the CAL range, replace the batteries and check CAL again. If the meter fails to attain the CAL position, then it should be sent in for servicing by the manufacturer.
- 2. Place the rubber current probe on either a wading rod or cable suspension unit. Set selector switch to the 2.5, 5.0 or 10.0 range (represents foot per second gradation up to that level) for velocity conditions present. Remember to wait at least 20 seconds after positioning the probe before reading the panel meter.
- 3. Read the panel meter directly. This reading is in feet per second.

#### 2.6.5.2.2 Maintenance and Calibration

The Marsh-McBirney Meter is maintained by simply cleaning the sensor with mild soap and water to keep the carbon electrodes free of non-conductive grease or oils. The calibration of the Marsh-McBirney Meter is to check the electronic readings in calibration mode and if the meter readings do not calibrate on the calibration setting the meter is sent in for repair. In order to check the accuracy of the velocity readings of the meter, OWM personnel check measured stream velocities against each meter in the same stream and location. Also the other current meters are checked in this same manner. When a meter comes back from the manufacturer, a certificate of calibration accompanies the meter. This newly calibrated meter is then used to compare velocity readings with other meters.

## 2.6.5.3 Measuring Flow From a Bridge with a USGS Type-A Crane

Flow measurements from bridges are made by the Surveys section when wading the stream to obtain flow data is not possible. A USGS Type-A Crane with a three-wheel base equipped with a Model #3100, A-55 reel with an 80 foot, 0.100" diameter cable is used. A Price AA meter is mounted on the end of the cable. A 15 lb. or 50 lb. C type weight, depending on the velocity of the current, is used with the meter in order to hold it as vertical as possible. A battery, headphone and stopwatch are used to monitor the revolutions of the meter.

To determine points of measurement from a bridge, the same criteria as those used when wading are applied.

#### 3.6.5.3.1 **Procedures:**

- 1. Inspect bridge; excessive debris in the channel will lessen accuracy of measurement.
- 2. Clear path for crane.
- 3. Measure and mark bridge (usually on railing) where vertical measurements will be made.
- 4. Assemble crane and attach meter, weight, headphone and battery. Make spin test. When attaching meter and weight, see procedure #6 to help determine distance between the two.
- 5. Begin measurement by leaning crane against bridge railing at first vertical.
- 6. Zero the counter (computing depth indicator) on reel. This is accomplished by lowering the AA meter until the horizontal tailpiece touches the water. Then pull out on small crank handle on the counter and adjust to "0" starting point. Push in handle to set.
- 7. Continue to lower the meter until the C type weight touches bottom. The value

on the reel counter is the actual depth not counting the distance between the bottom of the C-weight and the mid-point (horizontal tailpiece) of the meter. The hanger bar on which the C-weight and AA meter are suspended has various holes in order to adjust the distance between the two, depending on the size of the weight. The distance between the weight and mid-point must be taken into account when calculating the actual measuring depths along the verticals. Since a depth of 1.25' is the minimum depth recommended for a reading with the AA meter, then a distance of at least .50' is needed between the bottom of the weight and the mid-point of the meter in this case in order to accomplish a velocity measurement at .6 of the 1.25' depth.

- 8. At depths greater than 2.5', the two point method is used the same as when wading. In order to set the .8 measurement easily the reel counter gage has a spiral scale which reads .8 of the depth registered directly on the scale around the outside edge.
- 9. **Example:** If 3.0' is measured as the vertical depth (including the distance between the weight and the AA meter), then raise the AA meter from the stream bed until 3.0' is shown on the spiral scale. The AA meter will then be suspended at .8 of 3.0' depth or 2.4' on the outside scale.
- 10. Proceed across the bridge making all necessary measurements. Make the total calculation in the same manner as when wading. Make notes of all obstructions and anomalies which might affect the total flow. When counting clicks from the headphone, count the first one as "0", i.e., 0, 1, 2, 3 etc. Initiate the stop watch on the first count.

## 2.6.5.3.2 Problems Affecting Flow Measurement From Bridges

One of the most common problems to be resolved when measuring flow from bridges is direction of flow. If the direction of the flow is not perpendicular to the cross section, a correction must be made. The most convenient method is to use "USGS Form 9-275, Field Note Sheet". Around the outer margins are printed the cosine values of the possible angles of flow. By aligning the point of origin "0" on the left side of the Field Sheet with the edge of the bridge rail and turning the sheet until the right edge aligns with the direction of flow, the cosine of the angle of flow can be read. The cosine is then multiplied times the measured velocity for the correct speed.

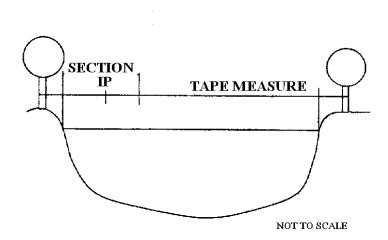
## 2.6.5.4 Measuring Stream Flow

#### 2.6.5.4.1 General

In-stream flow measurements are taken by the Surveys Section in wadeable streams where there are no USGS stream-gaging stations. This method of measurement requires the stream to be divided into at least 20 sections with equal amounts of flow and volume. The average velocity and area is then measured for each section. The product of

each sections width, depth, and velocity is the "flow" for the section, which is measured in feet per second (fps). The sum of flow for all the sections of a stream is equal to the flow of the stream in cubic feet per second (cfs).

Figure 2.2 Stylized Cross Section of Stream Demonstrating Flow Measurement



# 2.6.5.4.2 Selecting the Best Cross-Section of a Stream for In-stream Flow Measurement

- 1. Examine the stream reach for cross sections and select the one that is most suitable based on the following:
  - (i) The stream channel is straight and the cross section is perpendicular to the direction of flow.
  - (ii) The stream bed and banks are as uniform as possible.
  - (iii) The minimum velocity is greater than 0.5 fps.
  - (iv) The stream is safely wadeable.
- 2. After selecting the best cross section, stretch a tape measure across the stream, with the tape measure at right angles (perpendicular) to the direction of the current and secure the tape. Determine the proper widths of the sections to be measured by observing the total width of the stream and making sure you take at least 20-25 sections to be measured. No one section is to make up more than 5% (10% in very small streams) of the total flow of the stream, so be sure to shorten the section width if there is more flow in a particular area of the stream.
- 3. The number of vertical cross-sections varies with depth and width of the stream. No fixed rules can be made for number of vertical, other than they should be spaced so as to disclose the real shape of the stream bed and the true mean velocity. This usually means one (1) foot or smaller spacing increments on smaller streams and

two (2) foot spacing on larger, uniform streams. Note: Uneven spacing of verticals may be necessary when the stream bed is irregular.

4. Establish the initial point (IP) from which you will begin to measure sections of flow. This IP should be one half the predetermined section width from the stream bank.

## 2.6.5.4.3 General Operation of Current Velocity Meter with Top Setting Rod

- 1. Turn the meter on and set it to read in "feet per second".
- 2. With flow rod facing the tape measure (upstream), stand 1-3 inches downstream from the tape and 18 inches or more from meter rod.
- 3. Keep flow rod vertical and meter parallel to the direction of flow.
- 4. Read the depth of the cross section by viewing the flow rod from side, then adjust the depth of the meter to the appropriate depth (read to half-tenths if possible). The depth can be measured with the depth gage rod. Each single mark represents 0.10 of a foot, each double mark represents 0.50 of a foot, and each triple mark represents 1.00 foot.
- 5. After meter is in place, allow a few moments for it to become stable in the current before the velocity measurement is recorded. Allow extra time for velocities less than one foot per second.
- 6. If the depth of the vertical is less than 1.5 feet, measure the velocity at 0.6 (60%) of the depth. To set up the sensor at 0.6 of the depth, line up the foot scale on the sliding rod with the tenth scale on the top of the depth gauge rod. If, for example, the total depth is 1.3 feet, line up the 1 on the foot scale with the 3 on the tenth scale. If the depth of the vertical is 1.5 feet or greater, use the two-point method. Measure the velocity at 0.2 (20%) and 0.8 (80%) of the depth and average the readings. To set the sensor at 0.2 depth, multiply water depth by 2 and set this value as described above. To set the sensor at 0.8 depth, divide the depth by 2 and set this value as described above.

## 2.7 MEASUREMENT OF TIME-OF-TRAVEL (T.O.T.)

#### 2.7.1 Introduction

Time-of-travel can be defined as the calculation of the average velocity of a flowing waterbody over a given reach or distance of its length.

The Surveys section conducts time-of-travel (T.O.T.) measurements primarily in order to provide data to the Office of Water Quality Modeling Section for wasteload allocation calculations that are ultimately the basis of the NPDES permits. When the streams that are being modeled vary widely in topography so as to affect the flow with pools and riffles, it becomes especially beneficial to supplement the hydrologic and geometric data with T.O.T. data using dye tracer techniques.

Rhodamine WT (20%) is injected as the tracer. Measurements based on time are made using a fluorometer to identify concentrations at various points along the studied stream reach.

#### 2.7.2 T.O.T. MEASUREMENT PROCEDURES

In preparing and executing a T.O.T. measurement, the steps below should be followed:

- 1. Obtain maps and facility information of the area under study. Perform a reconnaissance and determine "when, where, how" and how much dye will be injected. "When, where, and how" will be determined by the resources available in terms of manpower, equipment, and the physical size of the waterbody segment under study. "How much" dye to be used is calculated from the "Computation For Time-of-Travel Worksheet." Sometimes more than one injection site may be used. This is done in order to save time when stream velocities are quite slow. Great care must be taken in this case, though, in order to avoid confusion in overlapping the dye clouds. For purposes of modeling, the water body flow should not exceed 10X the Q7-10 at the time of the T.O.T study.
- 2. Determine the sites where waterbody sampling will be done and whether it will be manual or by automatic sampler in order to track the dye. The waterbody stream reach must be physically inspected as much as possible for dams, water intakes, outfalls, pools, riffles, i.e. anything which could affect flow so that allowances can be made. How often samples will be collected should be determined at this point in your planning. Usually 20 to 30 samples are taken to define the time-concentration curve at each sampling location. Usually 3 to 4 sites are used downstream for sampling over a distance of approximately 5 miles.Perform preliminary flow measurements. This must be accomplished just prior to the T.O.T study in order to calculate the dye required and to estimate the T.O.T. to aid in sampling see the "Time-of-Travel Approximation" in Table 6 below. Markers can be set at this time to help monitor the stage of flow. This can be accomplished with something as simple as wooden stakes with increments. The study should be done at a steady state period.
- 3. Initiate study. Dye should be injected as a slug at the outfall point. This should be done as rapidly as possible since this represents a dye "peak" at this point. Automatic samplers or personnel should be in place down stream in order to start sampling at the estimated times necessary to capture the leading edge and peak of the dye cloud. It is also desirable to capture the trailing edge, though sometimes not practical. It is common practice to define the trailing edge as the time when the dye concentration drops to 10% of the peak value. Samples should be collected every 15 minutes until the dye arrives. Then sample at 10% of the travel time. Example: If the dye takes 30 minutes to arrive, then sample at 3 to 5 minute intervals.

**Table 2.6 Time-of-Travel Approximation** 

		<u>,</u>	Stream Miles			
Velocity	1	2	3	4	5	6
Feet/Sec.	HRS MIN	HRS MIN	HRS MIN	HRS MIN	HRS MIN	HRS MIN
0.1	14 42	29 24	44 06	58 48	73 30	88 12
0.2	7 21	14 42	22 03	29 24	36 45	44 06
0.3	4 54	9 48	14 40	19 36	24 30	29 24
0.4	3 40	7 21	11 01	14 42	18 22	22 02
0.5	2 56	5 52	8 48	11 44	14 40	17 36
0.6	2 27	4 54	7 21	9 48	12 15	14 42
0.7	2 06	4 12	6 18	8 24	10 30	12 36
0.8	1 50	3 40	5 30	7 20	9 10	11 00
0.9	1 38	3 16	4 34	6 32	7 50	9 08
1.0	1 28	2 56	4 24	5 52	7 20	8 48

- 4. Collect and read dye samples with a fluorometer. The Surveys section uses a Turner Model 10 fluorometer for this purpose. The operator of this fluorometer should be familiar with the operating and service manual of this instrument. See the Table 7, "Fluorometer Settings", below. Ideally the samples should be read using the fluorometer in the field and again in the lab after the sampling is finished. Remember that temperature will affect the readings. Avoid letting the samples warm in the sun or in the fluorometer. Samples for dye should be collected from just a few inches under the water surface at the centroid of the flow. Consistency in sample collection location is required. Clean glass cuvettes with caps are used for sample collection. Label each cuvette. Blanks made with DI water should be used to standardize the fluorometer, along with dye standards, which are prepared as serial dilutions (2, 4, 10, 20 ppb, etc). The collector should be sure to collect a background water sample prior to the arrival of the dye. The Form "TIME-OF-TRAVEL FIELD SHEET", Figure 3, should be used to log sampling information at each site. The data can be presented directly as is on the form or it may be reduced further.
- 5. Equipment and supplies: fluorometer, power supply (auto battery), gloves, dye, measuring container, waders, and cuvettes.

	2000		
	Min. Sensitivity	Top Scale	0-10 ppb
100	x3.16	Bottom Scale	0-3 ppb
100x	x10	Top Scale	0-1 ppb
	x31.6	Bottom Scale	0-0.316 ppb
	x31.6	Bottom Scale	0-31.6 ppb
1,,	x10	Top Scale	0-100 ppb
1x	x3.16	Bottom Scale	0-316 ppb
	Min. Sensitivity	Top Scale	0-1000 ppb

## 2.7.2.1 Preparation of Standards for Fluorometer

The stock dye solution is 20% product which translates to 200,000 parts per million (ppm). Serial dilutions must be made in order to calibrate the instrument for field use. All dilutions are to be made using de-ionized water.

Dilution #1-- 1 mL stock dye solution to 1000 mL DI water=200 ppm.

Dilution #2-- 1 mL of 200 ppm solution to 1000 mL = 200 ppb.

Using the 200 ppb stock solution, standards are prepared for ranges of 2, 4, 10, and 20 ppb.

Dilution #3--1 mL of stock to 100 mL water = 2 ppb

Dilution #4--2 mL of stock to 100 mL water = 4 ppb

Dilution #5--5 mL of stock to 100 mL water = 10 ppb

Dilution #6--10 mL of stock to 100 mL water = 20 ppb

Partially fill the volumetric flask with de-ionized water. Add the designated dose of dye stock and then finish filling the flask to the required amount. Mix thoroughly.

Use the volumetric pipets when possible, especially with the lower dose quantities. Pipets of most size ranges should be located in the standards tray in the Surveys section laboratory.

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Figure 2.3 Example of Time of Travel Worksheet

<b>Computations For Time of Travel Worksheet</b>	
Stream Reach	<del>-</del>
L = distance, in miles. from drop to most downstream picku Point	ıp
Q = maximum discharge in reach, in cubic feet per second	
V = estimated average velocity of flow in reach in feet per s	econd
Cp = peak concentration desired at most downstream pickup Parts per billion	p points in
Vd = volume of Rhod. Wt 20% dye solution, in liters, needs (equation to solve Vd is below. 0.000227 is the know	•
Computation for Amount of Dye	
0.000227 x(L) x(Q) x	
X(V)	
Vd = liters	
Time of Drop	
Dropped liters at	(Location)
At (Time) on	(Date)
Estimated Time of Pickup	
Leading edge ETAat	
Peak ETAat	(location)
Leading edge ETA at	(location)
	(location)

**Figure 2.4 Time of Travel Field Sheet** 

Time-of-Travel Study on \_\_\_\_\_

Sampling Section \_\_\_\_\_

## TIME-OF-TRAVEL FIELD SHEET

## INDIANA DEPARTMENT OF ENVIRONMENTAL MANAGEMENT

Dye I	njected	at cted					Time _						
Amou	ınt Inje	cted			Тур	e of Dy	/e		_ Con	c. In %			
Samp	ling Se	ction D	ischarg	;e		cfs	Width		Me	ean Der	oth		
Bottle No.	Sample Point	Sample Time	Fluoro	meter R	Readings	(ppb)	Bottle No.	Sample Time	Fluorometer Readings (ppb)				
			M.S.	x3.16	x10	x31.6			M.S.	x3.16	x10	x31.6	
(Ci	ircle Ran	ıge)	1x	1x	1x	1x	(Circle Range)			1x 100x	1x	1x	
			100x	100x	100x	100x	—		100x		100x	100x	
							<u> </u>				<u>                                     </u>		
									<del></del>				
		<del>                                     </del>						<del>                                     </del>					
		<del>  </del>	<b> </b>			<del>                                     </del>		<del>  </del>					
		<del>                                     </del>		<u> </u>		<u> </u>		<del>  </del>					
								<u> </u>	<del>                                     </del>		<sup> </sup>		
		<u> </u>	<u> </u>		<u> </u>	<u> </u>		<u> </u>	<u> </u>		<u> </u>		
											<u> </u>		
					!				l				

- Column: 1 1 Number on Sample Bottle.
  - 2 When more than one point in section is sampled, indicate as "A", "B", "C", etc., from left to right bank.
  - 3 Actual military time.
  - 4 Fluorometer dial reading on scales used.

## 2.8 STREAM REACH STUDY PROCEDURES

#### 2.8.1 Introduction

Stream reach studies are in essence "mini" basin surveys, but are designed more towards the acquisition of specific types of data for designated uses. Wasteload allocations, stream model calibration, permit limit qualifications, and permit compliance are some of the activities to which acquired data may be applied.

The stream survey usually covers approximately a 6 mile reach, within which is a permitted point source discharge or a specific site where a new discharge is proposed. Data must be collected upstream of the existing or proposed discharge point and then for at least five miles downstream, preferably at intervals of one mile or less. Any existing discharges must also be included.

#### 2.8.2 DATA REQUIREMENTS

The following table lists minimum data requirements for certain projects and recommended flow stages. In some low-gradient, slow-moving streams, sediment oxygen demand and reaeration data may be necessary. By combining the table criteria, many data needs can be supplied with one good survey done under proper flow conditions. Make sure that all field and lab data sheets required by the type of survey conducted are filled in completely.

**Table 2.8 Stream Reach Study Minimum Data Requirements** 

Data end use	Flow (stream)	*Slope	тот	WQ	Visual	Facility wq/flow
Wasteload Allocation (WLA) low flow	Y	Y	Y	Y	Y	?
WLA renewal - low-ave flow	Y	*	Y	Y	Y	Y
Model Calibration / verification At least one @ low-ave flow	Y	*	Y	Y	Y	Y
Permit limit qualification low flow	Y	*		Y	Y	Y
Compliance Sampling	***			Y**	Y	Y

<sup>\*</sup>Slope should be measured at least one time.

#### 2.8.3 SUMMARY OF SURVEY

Standardized report forms are available for raw data compilation. In addition to these, a brief summary regarding each data set is very helpful in putting the data in proper perspective.

4. Sampling station descriptions must be as detailed as possible and include calculated miles upstream or downstream from the central point of activity, i.e., the existing or proposed

<sup>\*\*</sup> Sample upstream and downstream of outfall if physical observations indicate problems and flow conditions are right.

<sup>\*\*\*</sup>Flow measurements helpful but not absolutely necessary. Estimate may be OK.

discharge point. Latitude and longitude are necessary for entry into data base. Provide map of area with all sites of interest plainly discernible.

- 5. Flow measurements should be discussed in relation to the estimated  $Q_{7-10}$  which is of particular significance to stream modeling. Cross sections and velocities are listed on the individual flow sheets but should be averaged at the bottom. Further evaluation of flow data from each site can be made by the user if necessary.
- 6. Provide physical descriptions of each site and the stream in general. General topography can sometimes be determined by slope and time of travel comparisons in conjunction with the actual observations made at each sample site. For example, very slow time of travel in an area of fairly high gradient would indicate the long pool-riffle-pool genre of stream. Mention land use and geography of reach under study.
- 7. Briefly discuss field data and laboratory data in relation to water quality standards. If a facility was sampled, compare data with permit limits.
- 8. Document anything else that is thought to be pertinent; such as riparian activities which may threaten stream quality, i. e., livestock in stream, bank erosion, etc.

## 2.9 MEASUREMENT OF SEDIMENT (BENTHIC) OXYGEN DEMAND

#### 2.9.1 GENERAL

Benthic oxygen demand may be broadly defined as the oxygen consumed from the overlaying water by the biochemical oxygen demands of the assemblage of micro- and macro-organisms associated with bottom sediments and substrates. Inorganic chemical oxidation reactions also contribute to the demand but are usually minor when compared with the biological demands. The on-site measurements of benthic oxygen demand are made by using a chamber respirometer, with a means of internally circulating the water. This chamber is then sunk a predetermined depth into the bottom sediments. The operation consists essentially of containing a known volume of water over a given bottom area with a chamber and measuring the dissolved oxygen drop with a galvanic oxygen probe implanted in the chamber.

#### 2.9.2 SAMPLING METHODOLOGIES AND PROCEDURES

The procedures for setting up and operating the benthic oxygen demand respirometer are relatively simple:

- 9. After the galvanic dissolved oxygen probe and meter have been calibrated the DO probe with stirrer is secured inside the respirometer. See Section in this manual titled "Measurement of Dissolved Oxygen" for a discussion on methods for measuring dissolved oxygen.
- 10. The electrical cables are taped to the inside and outside of the respirometer so that the sharp sides of the respirometer do not cut loose cables. Attach the DO probe and stirrer cables to the meter and the respirometer is now ready to use.
- 11. Determine the consistency of the bottom deposits. If the bottom is not clearly visible, a Ponar dredge sample should be taken and examined. If the sediments are mucky and watery,

extension flanges may be added to prevent sinking of the unit beyond the desired depth. Rocky or compacted bottoms may not allow the respirometer to sink in far enough to seal itself. A successful run is made when the respirometer does not allow water to flow in or out of the chamber, and there is a continual dissolved oxygen drop.

- 12. When a site looks favorable, the respirometer is inverted underwater so that all air is expelled. Flip the respirometer right side up (underwater) and check the cables once more for secure attachments. Turn on the stirrer and probe switches and follow the procedure of the dissolved oxygen meter to check operation.
- 13. Note the surface DO and lower the respirometer to just above the bottom and note the DO there.
- 14. Seat the respirometer and allow the DO to stabilize before recording the dissolved oxygen. This stabilization usually takes two to five minutes. A bad seal will be noticeable if within 15 to 30 minutes there is not a DO drop, or there is a DO increase along with a surface DO increase.
- 15. Dissolved oxygen concentrations are recorded manually either at 5 to 10 minute intervals or noting the time when the DO changes every 0.1 ppm. A DO recorder should also be used to obtain a permanent record.
- 16. Readings should continue until the dissolved oxygen drop rate is the same or levels off but should not continue over two hours. It can then be assumed that the DO will be used at this rate until it is completely consumed.
- 17. All readings are placed on the appropriate form, the points plotted on the graph, and the rate calculated.
- 18. A Winkler DO titration is taken at the bottom and at the surface. The respirometer is pulled from the bottom and DO readings taken at the bottom and surface and compared to the Winkler titration's to check accuracy of the probe. In some instances where a stream does not flow sufficiently to flush out the respirometer when it is lifted from the bottom, it may be necessary to pull the respirometer to the surface and flush it out before lowering it again to the bottom.
- 19. The respirometer is cleaned of any mud or sediment and the DO meter turned off. The probe is detached from the stirrer and the membrane inspected for bubbles or leaks. Place the protective cover on the DO probe.

## 2.10 FIELD DATA COLLECTION PROCEDURES

#### 2.10.1 TURBIDITY

The Surveys section currently has the capability of analyzing turbidity either by a Hydrolab multiprobe, or a separate portable turbidimeter. The instructions for operating a Hydrolab multiprobe are discussed later in this document. The operation of a Hach turbidimeter is discussed below.

#### 2.10.1.1 Instructions for Use of Hach Portable Turbidimeter Model 2100P

The Hach Portable Turbidimeter allows for real time reading of turbidity in Nephelometric Turbidity Units (NTU).

#### 2.10.1.1.1 Turbidimeter Operation

- 1. Collect a well mixed and representative sample into a sample vial and fill to the line.
- 2. Wipe the cell with a soft, lint free cloth to remove water spots and fingerprints. Apply a thin film of silicon oil on outside of vial, if necessary.
- 3. Press: I/O. The instrument will turn on. Place the instrument on a flat, sturdy surface. Do not hold the instrument while making measurements.
- 4. Put the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment. Close the cover.
- 5. Press: Read. The display will show "----NTU" then the turbidity in NTU. Record the turbidity after the lamp symbol turns off.

#### 2.10.1.1.2 Turbidimeter Calibration Checks

Before each sampling route or event, the turbidimeter should be checked against a Gelex Secondary Standard. These standards can be found inside each carrying case and are only valid with that particular unit. All calibration checks should be recorded on the log sheet. Choose a Gelex Standard vial that will closely resemble the actual sample value i.e., if the water is relatively clear choose the 0-10 NTU vial. Be sure the Gelex standard vial is aligned correctly when inserting them into the unit (diamond aligns with orientation mark). If the reading is not within 5% of the previously established value (the value in pencil in the white diamond area), make note of the difference on the field sheet and the log sheet and inform the personnel in charge of turbidimeters of the discrepancy.

#### 2.10.2 pH

The Surveys section currently has the capability of analyzing pH either by a Hydrolab multiprobe, or a separate portable pH meter. The instructions for operating a Hydrolab multiprobe are discussed later in this document. The operation of two different type of pH meters are discussed below.

All permanent staff members are responsible for maintaining and calibrating individually assigned pH meters. A pH METER LOG must be kept with the pH meter at all times. These logs should be folded and kept in zip locked bags inside the pH meter case. A copy of the pH meter operating instructions should also be kept in a zip locked bag inside the meter case. The pH meter should be calibrated before every sampling period (route) using a 7.00 Standard Units (su) buffer and 10.00 su slope buffer solution. Buffers should be replaced at least

weekly and should be replaced more often in heavy use periods. All calibration dates and buffer replacements should be recorded in the log. If the log is full, consult the personnel who is responsible for the pH meter maintenance for a new blank log and to file the old log sheet.

# 2.10.2.1 Calibration of Cole-Parmer Model 5985-80 Digi-Sense pH Meter 2.10.2.1.1 General

When calibrating field pH meters, it is best to use a slope buffer that will closely resemble the pH of the waters that you are sampling. For Indiana surface waters a slope buffer of 10.00 su should be used (Indiana surface waters tend to be more alkaline). With this in mind, each pH meter case should contain pH buffer 7.00 and 10.00 su. A rinse bottle full of distilled water should be used in place of the pH buffer 4.00. This rinse water should be replaced every time the buffers are replaced. When using the (ATC) probe, the meter automatically compensates for changes in buffer temperature. When not using the ATC probe, make sure that the temperature is set manually to reflect the temperature of the solution by using a separate thermometer. To do this, push the RANGE button until the degree Celsius symbol is displayed. Set the temperature from the thermometer by using the arrow keys.

- 1. Connect the pH electrode and the ATC probe (if ATC probe is used).
- 2. Take plastic tip off of pH sensor end. Inspect bulb to make sure it is clean. If the bulb has dried out, it will have to be rehydrated before calibration. Rehydrate the bulb by immersing in tap water for 30 minutes.
- 3. Place the electrode in a pH 7.00 buffer solution along with the ATC (or thermometer). Wait approximately 30 seconds for the sensors to stabilize and write down reading under "Meter Rd." column, and the press CAL.
- 4. If the electrode recognizes the pH 7.00 solution, the exact value will appear on the display in accordance with the pH and temperature charts. If not, the symbol "E4" will be displayed-see "error code guide" in the pH manual.
- 5. Wait 30 seconds and then push CON again to accept the buffer.
- 6. The first calibration is now finished. "E5" will appear on the display at this point, indicating that the instrument has entered the slope calibration mode.
- 7. Take the electrode and ATC (or thermometer) out of the 7.00 buffer, and rinse (dip) them with distilled water and dip it into the slope buffer (most likely 10.00). "E5" will disappear when the electrode is placed in the slope buffer, and the value of the buffer will then appear on the display. Wait 30 seconds for the sensors to stabilize and write down the reading under the "Meter Rd." column, and press CON. The pH meter is now calibrated.
- 8. Rinse electrode with distilled water and place black plastic tip with KCl (Potassium Chloride) solution back onto sensor end.

## 2.10.2.1.2 Operation

- 1. Connect the pH electrode and the ATC probe (if ATC probe is used).
- 2. Take plastic tip off of pH sensor end. Inspect bulb to make sure it is clean.
- 3. Push the ON/OFF button to turn unit on.
- 4. Then push Range until the display indicates the desired mode. The unit should already be in the pH mode when it is turned on. For the temperature measurement press the RANGE key until the degrees Celsius symbol appears. Press the RANGE key again to get millivolts displayed as "mv". This parameter is not recorded so ignore this. Press the Range key again to display the pH mode.
- 5. Rinse electrode with distilled water and place black plastic tip with KCl solution back onto sensor end.

**Note:** When using the instrument, press the keys firmly and hold for a half second. Please consult the operating manual for further operating instructions.

#### 2.10.2.1.3 Additional Notes

- 1. Disconnect pH sensor cable and the ATC cable from unit before closing pH meter case. This will prevent damage to the senior cables.
- 2. Always keep sensor bulb moist with black storage tip. If KCl storage solution is not available, use tap water or pH 4.0 buffer. **Do not use Distilled water**.
- 3. If readings start to drift and never really stabilize, it might be time for a new electrode. Please instruct the personnel in charge of pH meter maintenance.

## 2.10.2.2 Calibration of Hach Model EC20 pH Meter

When calibrating field pH meters, it is best to use a slope buffer that will closely resemble the pH of the waters that you are sampling. For Indiana surface waters a slope buffer of 10.00 su should be used (Indiana surface waters tend to be more alkaline). With this in mind, each pH meter case should contain pH buffer 7.00 and 10.00 su. A rinse bottle full of distilled water should be used in place of the pH buffer 4.00. This rinse water should be replaced every time the buffers are replaced.

- 1. Connect the pH electrode to the meter.
- 2. Take plastic tip off of pH sensor end. Inspect bulb to make sure it is clean. If the bulb has dried out, it will have to be rehydrated before calibration. Rehydrate the bulb by immersing in tap water for 30 minutes.
- 3. Press the **I/O** key to turn on meter.
- 4. Press the **MODE** key until the pH mode indicator is displayed.
- 5. Place the electrode into the 7.0 Buffer.

- 6. Wait 30 seconds and then push CON again to accept the buffer.
- 7. Press the **CAL** key. Calibrate will be displayed above the main field. After the current calibration slope is displayed, P1 will be displayed in the lower field. P1 indicates the meter is ready to accept the first buffer point. Make sure the electrode is in the correct buffer.
- 8. When the electrode is stable, the meter will beep and READY will be displayed along with the temperature-corrected value for the buffer (Flashing). Press the **YES** key to accept this point.
- 9. The display will remain fixed momentarily, then P2 will be displayed in the lower field. The meter is now ready for the second buffer.
- 10. Rinse the electrode and place it in the second buffer (10.0). Wait for a stable pH reading (the meter will beep) and for READY to be displayed. Press the **YES** key to accept the second point. The screen will display P3 in the lower field.
- 11. Press the **MODE** key to end the calibration.

#### 2.10.2.2.1 Operation

- 1. Connect the pH electrode to the meter.
- 2. Take plastic tip off of pH sensor end. Inspect bulb to make sure it is clean.
- 3. Press **I/O** key to turn meter on.
- 4. Place the electrode in the sample and wait for a stable pH reading (the meter will beep) and for READY to be displayed.
- 5. Record the concentration directly from the upper meter display. Temperature is displayed in the lower field.

#### 2.10.2.2.2 Additional Notes

- 1. Disconnect pH electrode cable from the meter before closing the case. This will prevent damage to the electrode cable.
- 2. Always keep sensor bulb moist with black storage tip. If KCl storage solution is not available, use tap water or pH 4.0 buffer. **Do not use Distilled water**.
- 3. Remove 9-volt battery from back of unit after each use. This will extend battery life. A few extra batteries will be in each case for backups.

## 2.10.2.3 Calibration of Oakton pH 6 Acorn Series Meter

When calibrating field pH meters, it is best to use a slope buffer that will closely resemble the pH of the waters that you are sampling. For Indiana surface waters a slope buffer of 10.00 su should be used (Indiana surface waters tend to be more alkaline). With this in mind, each pH meter case should contain pH buffer 7.00 and 10.00 su. A rinse bottle full of distilled water should be used in place of the pH buffer 4.00. This rinse water should be

replaced every time the buffers are replaced.

- 1. Connect the pH electrode to the meter.
- 2. Take plastic tip off of pH sensor end. Inspect bulb to make sure it is clean. If the bulb has dried out, it will have to be rehydrated before calibration. Rehydrate the bulb by immersing in tap water for 30 minutes.
- 3. Immerse probe tip into pH buffer 7.00 calibration standard and allow reading to stabilize.
- 4. Press CAL key.
- 5. Press HOLD/ENTER key to confirm value.
- 6. Repeat with the slope pH buffer 10.0 calibration standard (or 4.00 for acid waters).

#### 2.10.2.3.1 Operation

- 1. Connect the pH electrode and the temperature probe to the meter.
- 2. Take plastic tip off of pH sensor end. Inspect bulb to make sure it is clean and hydrated.
- 3. Press the ON/OFF key to power up the meter.
- 4. Place the electrode and temperature probe in the sample and wait for a stable pH reading.
- 5. Record the pH and temperature value from the meter display. Pressing the MODE/INC Key will toggle between the pH/mV/Temperature readings.

#### 2.10.2.3.2 Additional Notes

- 1. Disconnect the pH sensor and temperature electrode from meter after use. This will prevent damage to the cables when closing the carrying case.
- 2. Always keep sensor bulb moist with KCL. After use, always put the pH sensor tip back into the storage cap filled with KCL. If KCL storage solution is not available, use tap water or pH 4.0 buffer. **Do not use Distilled water**.
- 3. Unstable pH readings usually indicate that the pH electrode should be replaced.

## 2.10.3 MEASUREMENT OF DISSOLVED OXYGEN (DO)

Personnel of the Surveys section often make measurements of the dissolved oxygen concentration in bodies of water they are surveying or studying. These measurements are made either using the Winkler iodometric titration test method, or a Hydrolab multiprobe. The Winkler Method is discussed in detail below. The instructions for operating a Hydrolab multiprobe are discussed later in this document.

#### 2.10.3.1 Winkler Method

The field test for dissolved oxygen is a very important aspect of a water pollution survey. It is therefore necessary that each member of the survey team uses proper and consistent techniques in performing this test. Reagents, procedures, equipment, and sampling method are all important and will be explained in the following guidelines.

## 2.10.3.1.1 Field Equipment for Winkler Method

D.O. field equipment consists of the following items:

- 1. Chemical reagents
- 2. A 500 mL Erlenmeyer flask or equivalent
- 3. A 10 ml titration pump
- 4. Tray of standard size calibrated 272 mL (+3 ml) DO bottles
- 5. A thermometer
- 6. A calibrated 10 mL pipette graduated in 0.1 ml increments
- 7. Sampling equipment

The reagents used for the Winkler method are manganous sulfate (MnSO<sub>4</sub>), alkaline iodine-azide solution (KI), 18N (50 %) sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), starch solution, and 0.0335N thiosulfate solution. These reagents are prepared by personnel of the ISDH Laboratories and are available upon request. All Surveys Section staff required to maintain their respective D.O. kits.

When reagents are taken from the ISDH Laboratories, it is very important to mark the date on the bottle. This is necessary in order to insure that fresh reagents are always on hand for the field tests. Starch and thiosulfate will breakdown readily in the heat and sun, so it is important to replace these reagents at least weekly during the summer, and monthly during the winter. The thiosulfate should be kept in opaque bottles in the field D.O. kits. Starch and thiosulfate can be kept in significant quantities (2 to 3 liters) in the equipment room refrigerator to insure a fresh supply is always available.

Except for thiosulfate and starch, the reagents are dispensed from 250 mL plastic (Nalgene) squeeze bottles which deliver a measured 2 mL amount of liquid. The thiosulfate solution is dispensed using the 10 mL pipette and titration pump.

## 2.10.3.1.2 Sampling Methods for Winkler Method

The D.O. sample can be collected from the stream by using a plastic bucket that has first been rinsed with sample water. After the sample is collected, the D.O. bottle should be rinsed with sample water. Then, either **gently** pour the sample from the bucket into the D.O. bottle or submerge the D.O. bottle directly into the bucket to allow it to slowly fill up. After the bottle is filled, the cap must be replaced carefully on the D.O. bottle to avoid entrapment of air bubbles. This can be done easily by recapping the D.O. bottle

while it is still submerged in the bucket or by gently tapping on the side of the bottle until all of the bubbles have escaped. After the bottle is capped, follow the instructions in the Winkler Method Sample Preparation and Analysis section below.

When insufficient water depth or other factors prohibit the use of a bucket, collection of the sample must be done by hand. This is accomplished by holding the D.O. bottle slightly under the water surface and allowing the water to run slowly down the inside of the bottle. When the bottle is full, submerge the bottle and replace the cap, again avoiding air entrapment.

When dissolved oxygen sampling is required on municipal or industrial effluents, it is sometimes necessary to collect the sample from an outfall where excessive turbulence or other conditions make both the above methods impractical. In this situation, fill a bucket with the effluent and allow several minutes for quiescence. Then collect the D.O. sample from the bucket using the hand technique described above.

samples should always be collected from a point in the stream where maximum mixing has occurred. Very turbulent riffles and extremely static pools should be avoided.

Whichever collection methods are used, the water temperature must be recorded as soon as possible. This is a very necessary factor in data interpretation.

#### 2.10.3.1.3 Winkler Method Sample Preparation and Analysis

- 1. Obtain and wear Safety Glasses.
- 2. Remove the stopper and add 2 mL of KI and 2 mL MnSO<sub>4</sub> from squeeze bottles. Make sure the squeeze bottle is pointed away from all personnel.
- 3. Place stopper in bottle, drain excess water from around stopper and rinse bottle in sample water.
- 4. Mix sample by inverting D.O. bottle several times. Allow bottle to set until the solids settle to within approximately one inch of the bottom of the bottle.
- 5. Repeat step four (4).
- 6. After the solids have settled, remove stopper and add 2 mL of 50% H<sub>2</sub>SO<sub>4</sub>.
- 7. Rinse bottle in sample water.
- 8. Invert D.O. bottle several times. If solid precipitate persists, add an additional 2 mL 50% H<sub>2</sub>SO<sub>4</sub>, replace the stopper, rinse the bottle as before, and invert several more times.
- 9. Pour contents of D.O. bottle into titration flask.
- 10. Titrate sample using a burette, with 0.0344 N sodium thiosulfate solution, to a pale straw yellow color.
- 11. Add starch solution from squeeze bottle until the sample water turns dark blue.

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- 12. Continue titrating until the sample becomes completely clear. Check sample against something white to be sure.
- 13. On the field sheet, record the amount of sodium thiosulfate used as the D.O. measurement.
- 14. Rinse all glassware when finished, and put back into kit.

#### 2.10.3.1.4 General Notes

A general description of the analytical methods, reagents, and equipment needed to run the azide modification of the Winkler Method can be found in Standard Methods, 20th Edition. This method is modified slightly when used by Surveys section personnel. These modifications involve changes in the volume of solution titrated and the normality of the sodium thiosulfate titrant. The normality of the thiosulfate titrant has been changed from the 0.025N solution described in Standard Methods to 0.0335N sodium thiosulfate. This more concentrated solution is used to titrate 272 mL (+3 mL) of the water solution prepared for dissolved oxygen analysis. The 272 mL (+3 mL) volume is the volume of the D.O. bottle used to collect the sample. Thus, the field investigator does not have to measure the 200 mL volume called for in the procedure in Standard Methods. The bottles used by field personnel have been selected for this volume (272+3 mL) by water laboratory personnel. Studies conducted by water laboratory personnel show that no significant differences were found in values when dissolved oxygen analyses were done in this manner, compared to the procedure outlined in Standard Methods.

#### 2.10.4 TOTAL RESIDUAL CHLORINE

## 2.10.4.1 Operation of HACH Pocket Colorimeter Test (0-2 MG/I)

- 1. Fill a 10 mL cell to the 10 mL line with sample and cap. Note: Samples must be analyzed immediately and cannot be preserved for later analysis.
- 2. **Note:** Be sure the instrument is in the low range mode. See the instruction manual, page 18.
- 3. Add the continents of one DPD Total Chlorine Powder Pillow to the sample cell (the prepared sample). Cap the cell and gently shake for 20 seconds.
- 4. **Note:** A pink color will develop if chlorine is present.
- 5. **Note:** Accuracy is not affected by undissolved powder.
- 6. **Note:** Gently shaking the cell dissipates bubbles which may form in samples containing dissolved gases.
- 7. Wait at least 3 minutes, but no longer than 6 minutes. During this period, proceed with step 8 12.
- 8. Fill a 10 mL cell to the 10 mL line with sample (the blank) and cap.

- 9. Remove the instrument cap.
- 10. **Note:** For best results, zero the instrument and read the sample under the same lighting conditions.
- 11. Place the blank in the cell holder, making sure the diamond mark faces the front of the instrument. Cover the cell with instrument cap. Flat side should face the back of the instrument. Be sure it fits tightly against the instrument.
- 12. Press: ZERO. The instrument will turn on and the display will show --- followed by 0.00.
- 13. **Note:** The instrument automatically shuts off after 1 minute. If this occurs, the last zero is stored in memory. Press READ to turn the instrument on and complete sample analysis.
- 14. Remove the cell from the cell holder.
- 15. **Within 3** minutes after the 3-minute period, place the prepared sample in the cell holder.
- 16. Cover the cell with the instrument cap.
- 17. Press: READ. The instrument will show --- followed by the results in mg/L total chlorine (Cl<sub>2</sub>).

**Note**: If the sample temporarily turns yellow after reagent addition, or shows over range (flashing 2.20 in display), dilute a fresh sample and repeat the test. A slight loss of chlorine may occur because of the dilution. Multiply the result by the appropriate dilution factor.

## 2.11 THE HYDROLAB<sup>a</sup> H20<sup>a</sup> MULTIPROBE SYSTEM

#### 2.11.1 Introduction

The Hydrolab H20 multiparameter sensor unit allows for a real time readout of dissolved oxygen, pH, temperature, specific conductance, depth, total dissolved solids, and turbidity. The unit is compact in size and helps eliminate the need to carry multiple pieces of equipment and reagent chemicals.

## 2.11.1.1 Components of the Hydrolab H20 System

There are 6 different components that should be utilized in most deployments of the Hydrolab H20 unit.

- 1. The "H20" Transmitter sonde
- 2. The "Scout 2" display unit
- 3. Rechargeable battery pack
- 4. Stirrer Unit
- 5. Cable (various lengths)
- 6. PVC tube

## 2.11.1.2 Assembly of the H20 Unit

- 1. At the first site, take the H20 sonde out of the carrying case and remove the storage cup.
- 2. Note: The assembly might take place in the equipment room before departure.
- 3. Screw the stirrer unit clockwise onto the sonde until tight.
- 4. Note: on units # 4, 5, 8, 9, 10, and 11 pay special attention to the web of the sensor guard to make sure the turbidity sensor has a "clear line of sight". That is, make sure that one of the sensor guard webs lines up just below the U-shaped turbidity sensor as the sonde is held horizontally.
- 5. Remove pin from the V-shaped metal handle from top of sonde and line up eye hook from the connector cables and reinsert pin.
- 6. Connect the cable to the end of the sonde (six pin) making sure to line up the round notch on the outside of the connector with the largest pin on the sonde. When making this connection, make sure to remove any trapped air in the connector by squeezing connector until you feel or hear the air escape.
- 7. Connect stirrer cable (2 pin) into 2 pin connector on cable. Again, remove any trapped air in connector, as in step 4.
- 8. Connect the round six pin screw connector cable into the left side of the Scout 2 display unit. Line up notches, push in, and turn clockwise until pins latch securely
- 9. Connect the rechargeable battery cable to the right side of the Scout 2 display unit. Again, line up the notches, push in, and turn clockwise until pins latch securely.

## 2.11.1.3 Scout 2<sup>a</sup> Display Functions

Push the "On/Off" button on the display unit to turn on the display unit and to "power-up" the sonde. The "Main Screen" should be displayed first, if not, push the "Screen Escape" button.

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Table 2.9 Main Screen Parameters of the Scout 2 Display Unit

Temperature(degrees Celsius)	Dissolved Oxygen(mg/l)	<b>Depth</b> (for units #4 and #5 (Ft)
		only, otherwise blank)
Conductivity (us/cm)	<b>pH</b> (su)	Blank

Push the "Screen Escape" button for the "Alt Screen" parameters which should be displayed in the following arrangements and units:

<b>Total Dissolved Solids</b> (g/l) (Units #8-#12 only, otherwise, blank)	<b>Turbidity</b> (Units #8-#12 only, otherwise, blank )(ntu)	Battery(Volts)
<b>Turbidity</b> (*For units #4 and #5 only)(ntu)		Time (amount of time unit has been turned on)
or		
<b>Dissolved Oxygen % Sat.</b> (Units		
#1-#3, and #8-#12 only)		

Note: The "Calibrate", "Variables", and "Enter" buttons are not used during ordinary field operation.

## 2.11.1.4 Field Calibration Checks of the Hydrolab H20 Unit

Record the Hydrolab Unit number being used in the appropriate space on the field sheet. D.O. and pH Hydrolab readings should be checked against a Winkler titration, and a separate pH meter, respectively. In addition, temperature and turbidity Hydrolab readings should also be checked and compared with separate meters. The Hydrolab temperature reading should be compared to the temperature reading from the pH meter and the Hydrolab turbidity reading should be compared to a separate field turbidimeter. All of these QC check analyses should be collected from the leftover sample water remaining in the plastic bucket after the PVC tube is filled. All field calibration checks should be collected at the first sample site of the day and recorded in the appropriate space on the field sheet. Make sure to record the Hydrolab unit number on the sheet. If the D.O. difference between the Winkler titration and the Hydrolab is 0.6 mg/L or greater, then another Winkler check should be run at the next sampling site. If this discrepancy still exists, continue to collect/record D.O. readings using the Hydrolab, but be sure to report this problem to the assigned Hydrolab maintenance personnel upon return to office. Collect the pH calibration check reading at the same site as the D.O. check. If the pH difference between the portable meter and the Hydrolab is 0.6 su or greater, check the portable pH meter calibration and run QC check again at the next sampling site. If discrepancy still exist, continue to collect/record pH readings using the Hydrolab, but report problem to the assigned pH and Hydrolab maintenance personnel upon return.

#### 2.11.2 How to Record Field Data Using a Field Sheet

(See Figure 2.5, Stream Sampling Field Data Sheet)

Prior to the sampling event, The Surveys Section AIMS database will automatically generate field sheets for each project or sampling event. These preprinted field sheets should already

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contain all of the site identification information as well as the sample numbers. When recording field data, make sure to fill in the following areas on the field sheet: Date Sampled, Hydrolab #, Water Depth/Gage Ht (if applicable), Water Flow (if applicable), Flow Estimated (if applicable), and all of the stream physical description checks boxes. In addition, record all the field data readings that are applicable to the type of sampling being conducted, in the appropriate area. Please write legibly as this is the only place that field data is recorded. Also, do not forget to record the bottle lot numbers and the preservative lot numbers, if applicable.

Figure 2.5 Stream Sampling Field Data Sheet

Samp	de#	Site#		Sample Medium Water						200	Samp	le Type		Duplicat	e Sample #			
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Stream Na	me: Daus	man Ditch	-					8	iver Mi	le:		Country	Kosc	fusko				
ite Descrip	ntion: SR 1	9																
Survey Cr	- International	Sample Col	-	2000	ite	2010100			Depth/		ter Flow	Flow		450000	Aquatic			
Chief	RJM	2	3	7	pled 2000	HydroLi 1	IID #	Gauge	Ht (ft)	1	5.11	Estimat	ed?	Algael	Life?			
	ample Take			Aliquots	2000	0.75	ter Flow	Year		- 11				~	V			
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ield Da	ita:																	
Date	24-hr Tim		77020	Water		ond Tur				blorine	Chloride	Chiero		Weat	her Codes			
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Sald Co	Managia.				k Rej	ected [See Ca	manufact)	_	Sky Ca	aditions b bus		Direction (Pilogram)	_	and Strengt	h Air Tem			
Date Date	libration Time	Calibrator	-	Calibr	otion		113		le sitored.	9 Seaw 30 Short	9 Hast C	O Degrees (18) Degrees	10	Light	2 33-45			
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/27/2000	8:20	TJB	DO		8.4	mg/L		1 15	Shower		1		6	Gale	1 0.00			
		Calibration Type	pit pio Todddis			1	d											
reserva	tives/Bo	ttle Lots:	I Mare State I	70														
Troup: Pre	servative	Preservati	ve Lot #	Bottle T	ype	Bott	le Lot∜	77.0										
General Chemistry:				1000P	_	00004030			_		Preservativ			Bottle T				
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									Mends Musler HNO3 Cs Cyankin NsOH O&C O&& Grence H2SO4 Textor Textor for		Cyaside NaOH O# & Grosse H25O4 Teston foe		5007 2507 1000G 500G 250G	07 900ml. Plastic, Namer Meet 07 250ml. Plastic, Namer Meet 000 1001ml. Glass, Namer Meet 000 500ml. Glass, Wide Mouth				
									VOA	Basteriologic Ice Valuele Organico (ICEA 7) Praticides Ice		Valuate Organica: HCL& Translater   1256			1256 40GV	G #25 m.L. Gleen, Wide Mouts		

### 2.11.3 OPERATING THE HYDROLAB H20 SYSTEM FOR FIELD DATA COLLECTION

Collect the field data sample using a separate rope and a 5 gallon plastic bucket. Rinse bucket once with sample water, dump so as not to disturb stream, and then retrieve sample water again. Take bucket full of sample water back to vehicle and rinse PVC tube and Hydrolab unit thoroughly with sample water. Rinse the PVC tube by turning it so all rinse water comes in contact with the whole inside surface area of the tube. Refill the PVC tube half way with the sample water. Pour sample water slowly into PVC tube, so as to minimize any artificial aeration that might occur. Turn on the scout display unit and slowly submerge Hydrolab unit, with stirrer attached, into tube. Rotate Hydrolab unit while in tube 1/2 turn in a back and forth agitation motion a couple of times. This will allow any trapped air bubbles to escape from around the turbidity sensor. Hydrolab turbidity readings might drift plus or minus 2-3 ntu and might never really "stabilize". Use Best Professional Judgement (BPJ) when recording this reading. Allow sufficient time for readings to stabilize. Sensors need to equilibrate to sample temperature, and a longer than normal stabilization period should be expected at the first site, maybe as long as 5 to 10 minutes, especially if the sonde was left in a very hot vehicle. The sonde unit should be left submerged in the PVC tube with the previous sample site water during transit between sampling sites. A small red cooler should be used to hold the PVC tube in a somewhat upright position between sampling sites. Current sampling site water should be used to thoroughly rinse the Hydrolab unit and the PVC tube before the final current sample water is poured into the PVC tube for analysis.

#### 2.11.4 ADDITIONAL OPERATING NOTES FOR THE HYDROLAB UNIT

- 1. **Do not let the Hydrolab sensors dry-out**. <u>Clean</u> sample water or tap water should be used in the screw on cup for storage after sampling run is complete. **Do not use DI water in the storage cup under any circumstances.**
- 2. **Never let the Hydrolab unit freeze**. Units should not be kept in vehicles overnight if the temperature is expected to drop below freezing.
- 3. Keep an eye on the battery voltage. If it drops below 10 volts, the battery should be recharged. **If the battery volts drop below 9, the sonde will stop operating**. A recharging pack adapter should be in the battery pack. Simply connect the battery pack cable to the charging adapter and plug into electrical outlet. An overnight recharge time is probably sufficient.
- 4. **Do not sharply bend or kink the cables. Watch the cable when shutting the tailgate of the vehicle**. Do not get in a hurry and slam it in the door. These cables are expensive to replace. Keep the cables rolled up as best as possible.
- 5. Try not to drop the plastic PVC tubes. The tubes will crack on the bottom and start leaking.

## 2.11.5 OPERATING RANGE, RESPONSE TIME, AND DRIFTS FOR THE H20 UNIT

If the Hydrolab readings fall outside any of these ranges, response times, or drift, please

consult the Hydrolab Troubleshooting section for in-field diagnostics. Stability time refers to the expected time needed for the reading to become stable. The drift refers to the drift of the Hydrolab reading after appropriate stability time has been allowed for sensor equilibrium. Note: Even under "ideal" sampling conditions some of the Hydrolab readings will not totally stabilize.

**Table 2.10 Hydrolab Operational Parameters** 

Parameter	Range	Stability Time	Drift
Dissolved Oxygen	2-14 mg/L	1-5 minutes	0.1 mg/L
PH	6-9 su	1 minute	0.1 su
Conductivity	100-3,000 us/cm	1 minute	1 us/cm
Turbidity	1-800 NTU	< 1 minute	5 NTU
Temperature	Temperature 0-40 degrees Celsius < 1 min		0.1 degrees Celsius

### 2.11.6 HYDROLAB TROUBLESHOOTING

**Note:** Any problems or concerns with Hydrolab equipment should be related to the Hydrolab maintenance personnel (page 9) upon return to the office.

## 2.11.6.1 Dissolved Oxygen

- 1. Is there enough water in sampling tube to fully submerge D.O. sensor?
- 2. Is the stirrer operating properly?
- 3. Lift up sonde while unit is turned on. Is the stirrer rotating?
- 4. Move around stirrer cable. Does stirrer go on and off indicating a short in cable?
- 5. Unplug and plug back in stirrer connector cable from sonde cable. Is this connection tight and free of air pockets?
- 6. Inspect D.O. membrane for any noticeable tears or deformities. If holes or tears are observed, stop recording the Hydrolab D.O. and use the Winkler method.
- 7. If still in doubt, re-sample with fresh sample water. If problems still exist, record the Winkler method for comparison purposes. If the difference between the Hydrolab and the Winkler is greater than 0.6 mg/L, record the Winkler concentration on the field sheet, but make note of problem on the field sheet and check the Hydrolab D.O. value against the Winkler value at the very next sampling site.

## 2.11.6.2 pH

- 1. Is there enough water in sampling tube to fully submerge the two pH sensors?
- 2. Is there debris covering the pH bulb or the reference sleeve?

3. If still in doubt, re-sample with fresh sample water. If problems still exist, use the portable pH meter for comparison purposes. If the difference is greater than 0.6 su, record the portable pH meter reading on the field sheet, but make note of problem on the field sheet and check the Hydrolab pH reading against the portable pH meter at the very next sampling site.

## 2.11.6.3 Conductivity

- 1. Is there enough water in sampling tube to fully submerge the conductivity block?
- 2. Is the conductivity block free of debris? Is there any ice forming on the pins?
- 3. Is the stirrer operating properly? (see the dissolved oxygen troubleshooting section.)
- 4. If still in doubt, re-sample with fresh sample water. If problems still exist, record the value, but make note of problem on the field sheet.

## **2.11.6.4 Turbidity**

- 1. Is there enough water in the sampling tube to fully submerge the u-shaped turbidity sensor?
- 2. If the concentration is greater than 800 NTU, rotate sensor unit ½turn back and forth a couple of times while in sampling tube. This will release trapped bubbles possibly caught inside of sensor. Also, observe clarity of sample water. If water appears very muddy, it is possible in some circumstances that the reading is accurate.
- 3. If turbidity is less than 1 NTU, agitate sample water in tube to re-suspend any solids that might have settled to the bottom of the tube.
- 4. If still in doubt, re-sample with fresh sample water. If problems still exist, record the value, but make note of problem on the field sheet.

#### 2.11.7 HYDROLAB H20 SYSTEM CALIBRATION PROCEDURES

**Note:** Calibration procedures are listed in the order that they should be conducted.

## 2.11.7.1 Dissolved Oxygen (Air Calibration)

**Note:** It easier to calibrate the D.O. using air calibration, than a Winkler. It has also been found that the calibration results are the same no matter which procedure is used. Better repeatability is achieved with the air calibration method. It is recommended that air calibration be used for most calibration events.

- 1. With the transmitter oriented so that the sensors are pointed toward the ceiling, pull the white D.O. sensor guard off of the sensor housing and gently remove any water droplets from the membrane.
- 2. Screw on the calibration cup and fill with tap water until the water is just level with

the small black o-ring that is used to secure the membrane and making sure not to get the membrane wet. Any water that gets on the membrane can be removed with the corner of a paper towel.

- 3. Turn the blue or black rubber calibration cover upside down (concave up) and lay it over the top of the calibration cup.
- 4. Turn on the "Scout 2" display unit and wait until the D.O. reading stabilizes.
- 5. Get the current barometric pressure from a calibrated barometer or the local weather service (or call the weather line at 635-5959). First you must convert this pressure reading from inches of mercury to millimeters of mercury. Look at the chart on the calibration room wall for the conversion. Next you must convert this number to an "un-corrected" atmospheric pressure. For the Shadeland office, you need to subtract 21.2 millimeters of mercury from the previously converted millimeters of mercury pressure. (Note: If you are out in the field and you do not know your elevation and/or your barometric pressure, you can use the default setting of 760, but this might induce a little bit of error. In this situation, it might be best to calibrate against a Winkler providing the Winkler reagents are fresh).
- 6. After the reading has stabilized, note the value. Chose "calibrate" on the display unit and choose "%" (D.O. % sat) by using the right or left arrow keys. Now use the arrow keys to enter the millimeters of mercury value you obtained in step 5. Press the "enter" key and select "Y" to save new calibration.
- 7. Enter the drift value in the "Hydrolab Log Book". This drift value is the difference between the initial D.O. value in mg/L before calibration and the D.O. value in mg/L after calibration. If the post-calibration value was higher indicate with a "-" before the value and if it was lower indicate with a "+" before the value. The D.O. air calibration is complete.

## 2.11.7.2 pH

- With the transmitter oriented so that the sensors are pointed toward the ceiling, and with the calibration cup screwed on, rinse with DI water and then with once used "pH 7 Rinse." Discard the rinse and unscrew calibration cup and dry with paper towel. Also dry the sensors as best you can.
- 2. Fill calibration cup with fresh pH 7.00 buffer making sure to fill calibration cup so buffer is just over the D.O. sensor.
- 3. Wait for the pH reading to stabilize and make note of the value.
- 4. Select "Calibrate" from display unit and then use arrow keys to select "P" and select "enter." Enter the value of your zero buffer (7.00) by using the arrow keys, and then select "enter." Select "Y" to save calibration.
- 5. Enter the drift value in the "Hydrolab Log Book". This drift value is the difference between the initial pH value before calibration and the pH value after calibration. If the post calibration value was higher, indicate with a "-" before the value, and if it was

lower indicate with a "+" before the value.

- 6. Dump "used" pH 7 buffer in extra cup marked "pH 7 Rinse" and use this for next "pH 7 buffer calibration.
- 7. Rinse sensors with DI water and then with once used "pH 10 Rinse" and discard. Wipe calibration cup dry with a paper towel and dry all sensors as best you can.
- 8. Fill calibration cup with fresh pH 10.00 (slope) buffer making sure to fill calibration cup so buffer is just over the D.O. sensor.
- 9. Select "Calibrate" from display unit and then "P" (pH) and select "enter." Enter the value of your slope buffer (10.00) by using the arrow keys, and then select "enter." Select "Y" to save calibration.
- 10. Dump "used" pH 10 buffer in extra cup marked "pH 10 Rinse." This can be used for future calibration. The pH calibration is complete.

## 2.11.7.3 Specific Conductance

- 1. With the transmitter oriented so that the sensors are pointed toward the ceiling, and with the calibration cup screwed on, rinse with DI water and then with once used "conductivity rinse." Discard the rinse and unscrew calibration cup and dry with paper towel. Also dry the sensors as best you can.
- 2. Fill calibration cup with fresh conductivity solution making sure to fill calibration cup so solution is just over the D.O. sensor.
- 3. Wait for the conductivity reading to stabilize and note the value.
- 4. Select "Calibrate" from display unit and then "C" (Sp Cond) and select "enter." Enter the value of your conductivity solution (most likely 718 μs/cm) by using the arrow keys, and then select "enter." Select "Y" to save calibration.
- 5. Enter the drift value in the "Hydrolab Log Book". This drift value is the difference between the initial conductivity value before calibration and the conductivity value after calibration. If the post calibration value was higher, indicate with a "-" before the value, and if it was lower indicate with a "+" before the value.
- 6. Dump "used" conductivity solution in extra cup marked "Cond. Rinse." This can be used for future specific conductance calibration. The conductivity calibration is complete.

## 2.11.7.4 Turbidity

- 1. With the transmitter oriented so that the sensors are pointed toward the ceiling, and with the calibration cup screwed on, rinse with DI water and then with "Turbidity Free Water". After this rinse, discard and unscrew calibration cup and dry with paper towel. Also dry the sensors as best you can.
- 2. Fill calibration cup with unused "Turbidity Free Water" (0 ntu) solution making sure

not to trap any air bubbles in the U-shaped turbidity sensor, and fill calibration cup so solution is just over the D.O. sensor.

- 3. Wait for the turbidity reading to stabilize and make note of the value.
- 4. Select "Calibrate" from display unit and then "P". Enter the value of the "Turbidity Free Water" (0.0 ntu) by using the arrow keys, and then select "enter". Select "Y" to save calibration.
- 5. Enter the drift value in the "Hydrolab Log Book". This drift value is the difference between the initial turbidity value before calibration and the turbidity value after calibration. If the post calibration value was higher, indicate with a "-" before the value, and if it was lower indicate with a "+" before the value.
- 6. Rinse sensors with once used "Turbidity Rinse" (typically 40 ntu for slope) and discard. Wipe calibration cup dry with a paper towel and dry all sensors as best you can.
- 7. Fill calibration cup with fresh slope (40 ntu) solution. Shake this solution rigorously before filling calibration cup. Again, make sure not to trap any air bubbles in the U-shaped turbidity sensor, and fill calibration cup so solution is just over the D.O. sensor.
- 8. Select "Calibrate" from display unit and then "Y". Enter the value of your slope solution (40 ntu) by using the arrow keys, and then select "enter". Select "Y" to save calibration.
- 9. Dump "used" turbidity 40 ntu solution in extra cup marked "40 ntu Rinse." This can be used for future calibration. The turbidity calibration is complete.

#### 2.11.8 HYDROLAB MAINTENANCE PROCEDURES

All sensors should be continuously checked for objectionable deposits and kept clean. All maintenance and calibration for each unit should be recorded in the Hydrolab Log Book. During all calibration procedures, the drift (difference between the standard solution value and the reading before calibration is set) should be noted and recorded in the log book. The drift is important because it can aid in determining an undetectable fouling of sensors, a shift in the calibration of the system, or a slowly failing sensor.

The dissolved oxygen sensor membrane and electrolyte should be changed on a regular basis. The manufacturer, however, does not specify how often this should be done. From experience, it is felt that changing the membrane and electrolyte every two months is probably sufficient if no fouling has occurred. In heavy use periods i.e. summer, the membrane and electrolyte might need to be changed more often. The sonde should be allowed to sit overnight to allow the dissolved oxygen membrane to "relax" before calibration.

The pH measurement system consist of a pH glass electrode and a pH reference electrode. The pH glass electrode requires maintenance only when a coating of oil, dirt, or biological growth

is observed. The pH glass electrode should be cleaned with a cotton ball wetted with rubbing alcohol. The pH reference electrolyte should be replaced every two months.

Maintenance on the pH reference electrode should definitely be conducted if the pH readings seem to drift, or if the porous junction is observed to be dirty or coated with biological growth. Maintenance on the specific conductance sensor, consisting of six pin-shaped nickel electrodes located under the white cell block, should be cleaned at least once a year, or sooner if fouling is observed.

# 2.12 THE HYDROLAB<sup>a</sup> MINISONDE<sup>a</sup> MULTIPROBE SYSTEM

#### 2.12.1 Introduction

The Hydrolab Minisonde multiparameter sensor unit allows for a real time readout of dissolved oxygen, pH, temperature, specific conductance, depth, total dissolved solids, and turbidity. The unit is compact in size and helps eliminate the need to carry multiple pieces of equipment and reagent chemicals.

## 2.12.1.1 Components of the Hydrolab Minisonde System

There are 5 different components that should be utilized in most deployments of the Hydrolab H20 unit. Currently, Surveys has two Minisonde units available, Units # 6 and # 7.

- 1. The Minisonde Transmitter sonde
- 2. The Surveyor 4 display unit
- 3. The weighted sensor guard
- 4. Minisonde Cable
- 5. PVC tube

## 2.12.1.2 Assembly of the Minisonde Unit

- At the first site, take the sonde out of the carrying case and remove the storage cup.
   Note: The assembly might take place in the Surveys Section equipment room before departure.
- 2. Screw the weighted sensor guard onto the sonde until tight. Pay special attention to the web of the sensor guard to make sure the turbidity sensor, if installed, has a "clear line of sight" between the webs of the sensor guard.
- 3. Connect the cable to the end of the sonde (the six pin connector) making sure to line up the round notch on the outside of the connector with the largest pin on the sonde. When making this connection, make sure to remove any trapped air in the connector by squeezing connector until you feel or hear the air escape. Secure the bulkhead cable guard to the sonde by turning clockwise until snug.
- 4. Connect the square nine pin connector to the Surveyor 4 display unit and secure by

rotating the two screws clockwise.

## 2.12.1.3 The Surveyor 4<sup>a</sup> Display Functions

Push the "On/Off" key on the display unit (the key located in the top right corner of the unit) to turn on the display unit and to "power-up" the sonde. After a short moment, all of the parameters will be displayed line by line on the screen in the following order:

**D/T:** Date and Time (MMDDYY,HHMMSS)

**DO:** Dissolved Oxygen (mg/L) **Tem:** Temperature (degrees C) **SPC:** Specific Conductance (us/cm)

**DO%:** Dissolved Oxygen (percent saturation)

IBV: Internal Battery Voltage

**IB%:** Internal Battery percent of full charge

The turbidity sensor is currently not installed on either Unit # 6 or # 7.

#### 2.12.2 HYDROLAB MINISONDE SYSTEM CALIBRATION PROCEDURES

To calibrate the Minisonde using the Surveyor 4 display unit, a password will have to be entered. A password is needed for calibration or to make any changes to the sonde set-up or the Surveyor 4 display set-up. The password for both Surveyor 4 units # 6 and # 7 is "ABC" (without the quotes). The fresh flow circulator (the stirrer) is an integrated sensor that is situated next to the dissolved oxygen sensor, and as such, should be shut off during calibration. The general calibration procedure as described in the Hydrolab H20 System Calibration Procedures section should be followed. To calibrate the Hydrolab Minisonde utilizing the Surveyor 4 display unit, press the key "Calibrate" and then "Sonde." Next choose each individual parameter for calibration as follows:

**DO%: Sat -** for dissolved oxygen calibration.

**SpCond: us/cm** - for specific conductance calibration.

Turb: NTUs - for turbidity calibration (if turbidity sensors are installed).

**pH:** Units - for pH calibration.

# 2.13 THE HYDROLAB<sup>a</sup> DATASONDE<sup>a</sup>

#### 2.13.1 Introduction

The Hydrolab Datasonde 3 and Datasonde 4 loggers are off-line units that are completely submersible. They can be deployed and left for unattended monitoring until recovered at the end of their deployment period. These datasondes can monitor dissolved oxygen, pH, temperature and conductivity. Each Datasonde unit should be calibrated before and after each deployment period. The post calibration procedures should be conducted on sensors that

have not been cleaned of any biological fouling. This is especially important for the dissolved oxygen sensor, because this sensor is the mostly likely to become fouled during deployment. The post calibration of datasondes is very important in order to account for any changes that have occurred during the deployment period. Calibration, programming, and downloading of these units require the use of a personal computer with a communication software package. All calibration procedures are the same as for the H20 units. Please consult that section for details.

#### 2.13.2 Procedures for Deployment of Datasonde

- 1. If you are not the person calibrating and programming the Datasonde give at least 72 hours notice to the individual doing the set-up.
- 2. Always use a weighted sensor guard or a stirrer to protect the probes.
- 3. Datasondes must be secured and locked when deployed. Use the protective aluminum casings along with cables and locks to secure and weight the sondes. Usual anchoring locations are tank rails or trees on the river banks.
- 4. Place loggers in a flowing area of the stream, not a stagnant area. Also, be sure probes will not be inhibited from working by sedimentation or biological growth from the stream. It might be necessary to suspend the weighted datasonde from a buoy to keep it off of the bottom of a stream.
- 5. After recovering the logger, the probes should be stored in tap water or clean sample water. Do not store sensors in distilled water.
- 6. Return sonde to laboratory for post-calibration and downloading of data.

## 3.0 QUALITY CONTROL AND QUALITY ASSURANCE

## 3.1 COLLECTION OF BLANKS

A field blank sample should be collected for each sample event (route). The Assessment Branch AIMS database automatically assigns field blanks when each specific project is scheduled and at the frequency designated by the project leader, but never less than one per sample event. The blank may be collected in the Surveys Section laboratory prior to the sample event using ultra pure blank water or in the field using the same ultra pure water. The field blank serves both as a trip blank and as a preservative blank. A trip blank shows that contamination was not introduced into the sample during handling and transport. A preservative blank shows that contamination was not introduced into the sample by the preservative.

## 3.2 COLLECTION OF DUPLICATES

Field precision is assessed through the collection of field duplicates at a rate of one duplicate for

every ten samples collected. The Assessment Branch AIMS database automatically assigns field duplicates when each specific project is scheduled and at the frequency designated by the project leader, but never less than one duplicate for every ten samples collected. All original and duplicate samples should be collected at the same time and side by side if possible for each parameter (original nutrient sample with duplicate nutrient sample, and original metal sample with duplicate metal sample, etc.)

# 3.3 COLLECTION OF MATRIX SPIKES/MATRIX SPIKE DUPLICATES (MS/MSD)

A Matrix Spike/Matrix Spike Duplicate (MS/MSD) sample is collected for a laboratory analysis control at the rate of one MS/MSD sample for every ten samples collected. The Assessment Branch AIMS database automatically assigns MS/MSD samples when each specific project is scheduled and at the frequency designated by the project leader, but never less than one MS/MSD sample for every ten samples collected. The MS/MSD sampling should follow the duplicate sampling protocol in order to collect a representative MS/MSD sample.

## 3.4 DATA QUALITY ASSESSMENT (DQA) LEVELS FOR FIELD DATA

The following table contains DQA levels, calibration QA/QC Requirements, and acceptance criteria for field data parameters collected by Surveys Section personnel.

#### 3.4.1 CONTROL LIMITS FOR FIELD DATA QUALITY ASSURANCE/QUALITY CONTROL CHECKS

The following table list control limits for dissolved oxygen and pH as promulgated from the Surveys Section field data QA/QC checks for the years 1999 through 2001.

Table 3.1 Acceptance Criteria for Surveys Section Field Data QA/QC Checks

Field Parameter	Control Limit (Relative Percent Difference [RPD])	
Dissolved Oxygen	20	
pH	11	

If the Relative Percent Difference (RPD) calculated from any field result for the above listed parameter and its respective field QA/QC check result is less than or equal to the Control Limit for the listed parameter, then all the field results obtained for the indicated parameter during a given sampling event will be considered "in control." If the Relative Percent Difference (RPD) calculated from any field result for the above listed parameter and its respective field QA/QC check is greater than the Control Limit for the listed parameter, then all the field results obtained for the indicated parameter during a given sampling event will be considered "out of control." Out of control field results will be considered as estimated results and assigned the data qualifier (data flag) "QJ."

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The Relative Percent Difference (RPD) is calculated in the following manner:

 $RPD = \{(|Field\ Result - QA/QC\ Check\ Result|) \div (Field\ Result + QA/QC\ Check\ Result)\} \times 200$ 

The QA/QC field check control limits for the various field parameters were calculated in the following manner:

Control Limit = (Mean RPD) +  $(3 \times Standard Deviation of RPDs [n = > 200] for each parameter)$ 

Note: The Control Limits represent a 99.7% Confidence Limit.

Table 3.2 Quality Assessment (DQA) Levels for Field Data

DQA Level	Parameter	Analytical Test Method	Description of Analytical Test Method	Calibration and QA/QC Requirements	QA/QC Acceptance Criteria
1	Chloride	YSI	Not an EPA Approved Analytical     Test Method     Ion Selective Electrode (ISE)	1 point calibration performed weekly	Not enough confidence in data; lacks QA/QC data
	Chlorophyll A (Uncalibrated)	EPA 445.0 (Modified)	Modification of an EPA Approved     Analytical Test Method     Fluorescence	Baseline is zeroed weekly and YSI default calibration used	Not enough confidence in data; lacks QA/QC data
2	Specific Conductance	EPA 120.1	EPA Approved Analytical Test     Method     Conductivity Bridge	Calibrated once per year unless out of control	Check performed weekly (+/- 5%)
	Chlorophyll A (Calibrated)	EPA 445.0 (Modified)	Modification of an EPA Approved     Analytical Test Method     Fluorescence	Baseline is zeroed weekly and YSI default calibration used.     Actual calibration is from contract laboratory results	QA/QC is performed by the contract laboratory in accordance with EPA 445.0.
3	РН	EPA 150.1	EPA Approved Analytical Test Method     Electrometric	Calibration performed weekly	Checked using secondary stand-alone instrumentation during each sampling event     Frequency is section specific (# Control Limit).
	Dissolved Oxygen (DO)	EPA 360.1	EPA Approved Analytical Test Method     Membrane Electrode	Calibration performed weekly	Checked using a different analytical test method during each sampling event     Frequency is section specific     (# Control Limit).
	Turbidity	EPA 180.1 (Modified)	Modification of an EPA Approved Analytical Test Method     Nephelometric     Light Emitting Diode	Calibration performed weekly     Calibration performed yearly if     checked by a Check Standard from a     second source prior to each sampling     event	Checked using secondary stand-alone instrumentation or check by a standard from a second source prior to or during each sampling event     Frequency is section specific (#Control Limit).
	Temperature	EPA 170.1	EPA Approved Analytical Test Method     Thermometric Thermistor	Calibration is checked against NIST Traceable Thermometer once per year unless out of control.	Checked using secondary stand-alone instrumentation during each sampling event     Frequency is section specific (#Control Limit).

## 3.5 MAINTENANCE AND CALIBRATION LOG BOOKS

Log books should be maintained for all field equipment used by the Surveys Section. All calibration and maintenance activities should be recorded in their respective equipment logs. Please be sure to initial and date each entry of the log book.

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