discourage little fuzzy critters from considering the box home.

- 6. Check to be sure the field data sheet is completely filled out. Leave this on a clip board in the security box.
- 7. Cover and chain the box.

4.C.6.d. Field Maintenance

No samples collected

- 1. Check the area for signs of vandalism. Determine if the LCD is still flashing 01 and the first time interval. If so, halt the program. Remove the programmer/distributor from the bottle case and lay on the ground beside the box. (Be sure there is enough slack in the lines to allow this.)
- Cap the 24th bottle and carefully dump out the remaining ice 2. and water. (Do not get water in the sample bottles.)
- Clear the sample intake strainer of any accumulated debris. з.
- Test the sampler and activator: restart and activate the Δ. program by submerging the stage activator. Allow the "collected sample" to spill on the ground. Halt the program.
- Replace the ice in the bottle case and remove the cap from the 5. 24th bottle. Replace the programmer/distributor.
- 6.
- Change the battery, if necessary. Check the height of the stage activator above the surface of 7. the water and reposition if necessary. Reset the activator by flipping the toggle switch to reset and then back to latch. Press the start program/reset distributor key. Wait the interval of your first sample to insure the program is not immediately initiated. Place the cover over the programmer/distributor.
- 8. Cover and chain the box.

Collection of Samples

- 1. Upon opening the box record the bottle number and the time interval left to the next sample from the LCD onto the field data sheet. Also record the time and date of the observation.
- 2. Remove the top of the sampler and cap all of the bottles using numbered caps. (It will take two people to lift the full bottle case out of the security box.)
- 3. Determine the exact time each sample was taken and compare these times to your hydrograph and other information you have about the storm to be sure the calculated times are reasonable.
- 4. Line up the bottles in sequence and make notes on any parameters of interest such as turbidity, color or general appearance of the sample. Select which samples you will have analyzed. When you transfer the sample to a lab bottle be sure the sample is shaken well so as to re-suspend settled solids and provide homogeneity to any sub samples.
- 5. Clean the sample bottles before reuse. *The amount of cleaning necessary between events must be determined on an individual basis depending upon the parameters of interest and the amount of time the sample water has been sitting in the bottle. Bottles used to collect nutrients and suspended solids can usually be cleaned sufficiently in the field with two tap water rinses.

4.C.6.e. Quality Assurance

Replicate Samples: when the sampler is operating in a discrete sampling mode, replicate samples can be obtained by hand. This should be done at the same time the automatic sample is collected. Be sure to stand <u>downstream</u> of the automatic sampler intake.

4.C.7. OPERATION OF ISCO MODEL 2870 BUBBLER FLOW METER

4.C.7.a. Preparation

- 1. Check desiccant (2 external and 1 internal containers). The desiccant should be blue. If it is pink replace it.
- Determine if you have enough chart paper. Each roll has 65' and will last about a 120 days at 1/4" per hour.
- 3. Determine if you have a working pen. Check to see if an extra pen is in the meter.
- 4. You will need a nicad battery as a power source.
- 5. It is easier to determine the bubble rate in the lab. A small bucket of water is sufficient to determine the rate. Adjust the **Bubble Rate Adjust** (on the side of the meter). A rate of 1 bubble per second for clean streams and 2 bubbles per second if grease, oil, high sediment or domestic raw sewage is present.

4.C.7.b. Set Up

This set up is for the determination of the water level.

*Unless you have a predetermined stage-discharge relationship from which to determine the rate constant and exponent you cannot determine flow rate. Therefore the **Primary Device Characterization Module** is useful only to define the water depth (ft or M). The meters are normally set for feet and the constant scaling switches have been set to 0.00 +0 to disable the **Total Flow** counter and conserve power. If you need to change these settings be sure the **POWER IS OFF.**

- 1. Connect the battery lead to the 12 VDC outlet and fasten the retaining straps.
- Turn the power on and allow the meter to warm up for 5 to 10 minutes.
- 3. Check the inside surface of the end of the bubble line to be sure it free of deposits which could interfere with the free delivery of air. If necessary cut off 1 to 2 inches. Position the end of the bubble line in the stream below the lowest expected water level. In fast flowing streams (velocity > 5 ft/sec), you may need to isolate the bubble line from the stream flow with a stilling well. (See the instruction manual page 2-68).
- 4. Verify the bubble rate.
- 5. Place the Display Select switch in the Calibrate Recorder Zero position and adjust the zero position of the pen using the Recorder Mechanical Zero Adjust knob.
- 6. Select the desired chart full-scale level span from the six Level span positions of the Recorder Mode/ Span switch. This will usually correspond to the maximum expected level.
- 7. Select the chart recorder speed using the Chart Speed switch.

(Usually 1/4" per hour.)

- 8. Use the **Chart Manual Advance** Thumbwheel to synchronize the pen position on the chart with the real time of day. Record the time and date on the chart.
- 9. Select the time interval between automatic purges of the bubble line on the **Purge** switch. If fouling of the bubble line is anticipated use 5 minute intervals. Otherwise longer intervals will suffice.
- 10. Select the mode of the recorder pen operation on the **Pen** operation switch. (The normal position will save power and smooth the chart record but cannot react as quickly to sudden changes in level.)
- 11. Place the **Display Select** switch in the **Level** position and determine the current level from the liquid crystal display. Use the **Level Adjust** control to adjust the level indicated on the liquid crystal display to match the actual level in the flow stream. Record location, date, time, span, chart speed and starting water level (as determined by staff gage or reference mark) on the chart paper. Close up and secure.

4.C.7.c. Service

- When servicing the meter, open up the case and record: location, date, time and water level as you did for the set up.
- 2. When leaving the meter for an extended period (>1 week) change the desiccant on a regular basis.

4.C.8. REAERATION MEASUREMENTS

4.C.8.a. Tracer Injection

Set-up:

1. Assemble hoses, pumps and electrical cables as in figure 4.C.-4.

Make all electrical connections through a ground fault interrupter.

- 2. Set up ethylene (or propane) cylinder in a safe manner. *If a regulator is attached to the cylinder drain and remove the regulator before you move the cylinder. Always place the cylinder in an upright position and secure with some type of restraining device. The cylinder valve should be open only when the cylinder is in use and supplying gas. Before installing the regulator, slightly open the cylinder valve for an instant and then close it. This will clear the valve seat of particulates. However, do not do this with the ethylene tank when it is near an ignition source. Move the ethylene tank to a safe area before clearing the seat. Introduce pressure to the regulator by slowly opening the cylinder valve. Never put anything on top of the cylinder or regulator that may damage a safety device or interfere with the quick closing of the valve. Ethylene or Propane gas flow should not occur unless the cylinder pressure is reduced by a regulator. Do not drag or roll a cylinder on its side. Always provide ventilation when using Ethylene or Propane. Keep Ethylene and Propane cylinders away from any source of ignition. Shut off ethylene feed line at mixing chamber and pressurize
- 3. Shut off ethylene feed line at mixing chamber and pressurize ethylene feed line up to 60 psi to check for leaks. Fix any leaks.
- 4. Start generator, test ground fault interrupter. Do not proceed without a properly operating ground fault interrupter. Any connection to the generator MUST be made through the ground fault interrupter.
- 5. Test river water pump and dye pump.
- 6. Pour the predetermined amount of dye into dye receptacle and bring to desired volume with river water. Mix thoroughly.



Release:

- 1. Turn off the hose to the River and begin pumping river water through the mixing chamber to bypass hose.
- 2. Adjust ethylene flow to desired rate.
- 3. Begin pumping the dye. Follow progress of dye through plastic tube. Adjust dye pump rate to desired rate.
- Begin ethylene injection: open valve to river and close valve 4. to bypass hose. "The goal is for the ethylene and dye to reach the river at
- the same time. 5. Record start time for injection, dye and gas flow rates,
- volume of pure dye injected.
- 6. Continuously monitor dye and gas delivery rates and keep them constant.
 - Tilt the dye container to draw out the last of the dye solution.
- 7. When dye solution is almost gone fill a pail with river water, approximately 1 gal, and pour it into dye container just as the dye solution is depleted. A leading edge of relatively weak dye solution will now be advancing towards the river. When it reaches the river, turn up the dye pump to full speed in order to flush line quickly. Turn off the gas flow between the rotameter and the mixing
- 8. chamber.
- 9. Note ending time for injection.

Maintenance:

- 1. Continue flushing dye receptacle and dye feed line with clear water.
- 2. Close the cylinder valve, drain the regulator, remove the regulator and replace the cylinder cap.
- з. Clean and put away all equipment.

Reaeration Tracer Study Sampling: Samples can be collected from a bridgeinbyupadingmor Coomecboatl samples with the small sampling can.

- 1. At each sampling station, choose a single point in the This will assure that transect for sample collection. variations in tracer concentrations are due solely to time and not variations across the transect. Determine the sampling point at the location of greatest velocity (usually in the middle). Slack-water locations or side channels should be avoided.
- 2. Place three pre-labeled bottles in the sampling can and put the top on the can. The plastic tubes should go inside the sample bottles.
- 3. Secure the top with two rubber bands.
- 4. Lower the sampling can into the water and fill until no more bubbles emerge from the can.

Perform the next two steps with the sample can kept in an upright position because the headspace created in the sample bottles must be of the correct volume.

- Drain the excess water from the sample can through the drain 5. hole and carefully remove the cap. The volume displaced by the tubes in the bottles will become a headspace (air space) in the sample bottle.
- Carefully cap the three bottles with septum caps. 6. oIf a fluorometer is used in the field to monitor the passage

of the tracer dye slug, a sample can be obtained for this purpose directly into a cuvette from the excess water that is drained from the sampling bucket.

- 7. Record the sample time, temperature and any other pertinent information on the field data sheet.
- 8. Store the bottles upside-down in the dark.

Sampling Frequency: The sampling frequency should be such that the entire ascending and descending portion of the curve is defined by about 20-25 samples. Sampling should be most frequent at the peak portion of the curve, and the ascending portion of the curve. The tail of the curve can have less frequent samples. As a general rule of thumb and in the absence of specific instructions from the survey chief, determine the appropriate sampling interval from the guidance below.

Estimated Travel Time (hours)	Sample Frequen	cy (minutes)
Injection point to sampling point	Ascending and peak portion	Descending portion
0 - 3	3 - 6	4 - 7
3 - 6	5 - 7	7 - 11
6 - 9	8 - 12	12 - 18
>9	13 minimum	20 minimum

4.C.8.b. Quantitative Dye Analysis

Turner Fluorometer (Model 110 or 111)

Instrument Preparation: the fluorometer will need a <u>1 hour</u> warm up time.

- 1. Place the fluorometer on a level, firm base. Place the fluorometer in an area where there will be no abrupt air temperature changes. Shield from direct or strongly reflected sunlight.
- Check the filter placement. Green (primary) on right; orange and blue (secondary) on left with blue facing out.
- 3. Turn all panel switches off (down). Plug the power cord into the receptacle.
- 4. Turn power switch on (up). The pilot light next to dial should come on.
 *Note that this is not an indication that the lamp is on.
- 5. Hold **start** switch up for <u>10 seconds</u>, then release, to turn on lamp. Verify that the lamp is on by observing it through the primary filter. You must re-start the lamp if power should ever be interrupted, even momentarily.
- 6. Allow the fluorometer to warm up for a minimum of one hour.

Calibration:

⇒Fluorometer calibration is <u>not</u> necessary if you do not need to determine the total quantity of dye. (It is usually not necessary to determine the total quantity of dye for time of passage studies but it is always necessary for reaeration studies.)

*If the fluorometer has had sufficient time to warm up, the instrument is not moved, the lamp has not been removed and the room temperature remains constant, the calibration should hold.

1. Prepare six Rhodamine WT dye standards which range from 1 - 100 ug/l.

*Keep these standards at room temperature and protect from the light.

- 2. Adjust the zero: insert the dummy cuvette (black rod), close the door, turn the lever actuated lock counterclockwise to unlock, turn the blank knob to zero and then turn the lever actuated lock clockwise to relock. Remove the dummy cuvette and close the door. *The zero setting need not be readjusted unless later checks show a change of three or more dial divisions.
- 3. Clean the cuvette you will be using and check it carefully for scratches etc. At the top of the cuvette make a single vertical mark with a wax pencil. Always position this mark at the same location when inserting the cuvette. This will keep your error factor due to imperfections in the cuvette constant.
- 4. Rinse cuvette with D.I. water and once with solution to be tested. (This is especially important when testing a low concentration after a high concentration.) Fill to within one-quarter inch of top. Wipe outside of cuvette with tissue. If air bubbles are visible on the inside of the cuvette, dislodge them by gently tapping the cuvette. Handle cuvettes only near the top.
- 5. Insert cuvette in fluorometer, check scale setting and close the door gently but tightly to avoid spillage.
- 6. Observe and record the fluorescence dial reading after the dial has stabilized. *A jittery dial (± 1/2 dial division) is normal for some fluorometer. Once the dial has stabilized, it will tend to creep downward as the sample is heated by the heat from the fluorometer. Make the reading before the heating becomes significant. Sometimes it may be difficult to know when to take the reading. You can obtain a fair degree of consistency by lowering the dial well below the anticipated reading, inserting the cuvette, and taking the reading at the peak of the dial climb; just as it starts downward. *The dial can be lowered by shutting the door each time the cuvette is removed.
- 7. Read and record the results of each standard on every scale. When you insert a sample, partially open the door to shield it from ambient light.
- 8. Record the temperature of each standard at the end of the reading.
- 9. Observe and record the "dummy" reading after the sixth standard.
 *Recalibration is necessary if the change is >3 units.

Sample Testing:

*p***For best results, sample should be allowed to stabilize to** room temperature. Analysis should be completed within 24 hours of collection. Always protect samples from exposure to sunlight.

- 1. Read the samples as you did the dye standards. *For the greatest sensitivity, read each sample on the most sensitive possible and the next most sensitive. (The most sensitive scale is the 30X.) Record the temperature of each sample.
- 2.
- Clean and rinse cuvettes for storage. A thin residue of 3. dye may form inside improperly cleaned bottles and cuvettes which have contained high concentrations.

Quality Control:

- Read and record a reagent blank (the background stream 1. sample which was taken before the dye was injected). There should be a background sample from each station.
- 2. Choose a standard whose concentration corresponds to the range of the sample concentrations and test this standard after every 10 samples. *This periodic standard testing is used to determine if the fluorometer is in or out of control. The fluorometer is in control if the temperature-corrected standard readings do not change by more than ±3%. If the standard readings shift by ≥ 3 %, recalibrate the fluorometer.
- 3. Every tenth sample, record the sample temperature in the cuvette right after reading fluorometer.
- 4. Every tenth sample, observe and record the "dummy" readings. Recalibrate if necessary.
- 5. When you are finished reading the samples, reread every 10th sample as a replicate.
- 6. Observe and record the "dummy" just before turning off the fluorometer.

Determination of Dye Concentrations:

*If possible, use only 1 scale to calculate the dye concentrations for all samples at a particular sampling location. If this is not possible, use the more sensitive (larger) scale as the primary scale. When readings are off this scale, convert the reading from the next smaller scale to a reading for the larger scale. Determine the conversion factor by dividing the readings from the larger scale by the reading for the same sample on the smaller scale. Use the average of several samples to compute the correction factor.

Dye concentration computation:

```
CS = (R_x) (TC) (RS_x)
```

where:

CS = concentration of dye in the sample (ug/l) R_x = sample reading on the fluorometer dial on scale X (fluorometer units) TC = Temperature correction for difference between temperature of standard and the temperature of the sample from the table below [standard temperature sample temperature] (unitless)

 $RS_x = Response$ of instrument to standard on X scale = (ug/L dye) / fluorometer units

°C	°F	TC	°C	°F	тс
5.0	9.0	0.875	-5.0	-9.0	1.140
4.5	8.1	0.885	-4.5	-8.1	1.130
4.0	7.2	0.900	-4.0	-7.2	1.110
3.5	6.3	0.910	-3.5	-6.3	1.095
3.0	5.4	0.925	-3.0	-5.4	1.080
2.5	4.5	0.935	-2.5	-4.5	1.065
2.0	3.6	0.950	-2.0	-3.6	1.050
1.5	2.7	0.960	-1.5	-2.7	1.040
1.0	1.8	0.975	-1.0	-1.8	1.020
0.5	0.9	0.990	-0.5	-0.9	1.010
0.0	0.0	1.000			

Temperature Correction for Dye Analysis:

4.C.8.c. Ethylene Analysis

4.C.8.c.1. Instrument preparation

This should be done 24 hours before analysis.

- 1. Secure the compressed air cylinder to an immovable object before uncapping. Install the proper fitting regulator on the air cylinder.
- 2. Plug the **gas chromatograph** (GC) into a 110 VAC line and connect the air line from the cylinder to the air inlet. Use a snug finger tight pressure on all air fittings. Do not over tighten the fittings.
- 3. With the recorder plugged in and the power off, hook up the leads connecting the GC to the recorder. The 3 lead end attaches to the recorder input. Red is connected to positive at both ends, black to negative at both ends and the gray wire attaches to the chassis ground () of the recorder.
- 4. Turn on the GC. Set the GC range control to 10. Set the recorder input range to 100 mv (0.1v) and the REC/STABY switch to REC. Turn on recorder.
- 5. <u>Slowly</u> open the air cylinder valve and set the secondary pressure to 20 psi. Open the small black (needle valve) knob to allow the air flow to begin. Set both column rotameters at 25 cc/min.
- Set the recorder pen with the recorder zeroing knob to 50% scale. Set the chart speed at 10 cm/hr and let the machine warm up overnight.
- 7. The next morning, set the recorder baseline by using the recorder's zeroing knob. Set the baseline on the chart paper to 3%. This allows the pen room for some negative movement if needed. When ready to analyze samples, set

the chart speed to 1 cm/min.

4.C.8.c.2. Instrument Calibration

- 1. Turn the function switch to Test and depress start switch. The GC will automatically check itself. The display will read: "DIAGNOSTICS RUNNING".
- 2. The ethylene analyzer will automatically check for zeroing error excess drift, proper functioning of electronic circuitry, and range error. If a problem exists in one of these areas the display will read:

1) failed	to zero	3)	ADC/DAC
Error 2) excess error	drift	4)	range

- 3. If one of the above appears on the display: turn on the recorder, set the select switch on test and flip the reset switch up. Adjust the span screw on the back of the meter until the needle it is on the right hand side of the paper. Re-push the start button with the select switch on test. Continue this process until the meter passes the self diagnostics.
- 4. Select a clean gas-tight 1 cc syringe and pump the syringe once to purge it. Insert the needle into the can of ethylene standard. Press the button on the top of the can. The pressure in the can will slowly push back the plunger and fill the syringe with the standard gas. Fill the syringe to 1 cc.
- 5. Push the start button and inject the sample at the sound of the beep.

As quickly as possible, line up the syringe and insert the needle into the injection port of column 1. When certain the needle is properly inserted, fully depress the plunger with a steady, even pressure. The injection should take about 10 seconds. Hold the syringe in place for 5 seconds before removal from the instrument to insure that the entire sample enters the column.

- 6. On the recorder paper, mark the time of injection with a tic mark and identify the ethylene peak with the injection number. Note the time to peak for the ethylene. This time to peak is specific to ethylene and can be used to identify the ethylene peak from other peaks. Let the pen trace return to or near the baseline before making another injection.
- 7. Record the instrument response to the nearest tenth on the worksheet. Each injection should be numbered and logged on the GC worksheet noting injection number, instrument settings, instrument response and the sample of standard identity.
- 8. The instrument response is determined by subtracting the baseline value from the peak height.
- 9. Inject the standard at least 3 times before injecting samples.

The instrument calibration procedure should be followed whenever the GC is switched to a different scale. Only the 1 and the 10 scales should be used. The 100 scale is not linear and should be avoided. The 0.1 scale is too sensitive and methane, carbon monoxide or hydrogen will mask the ethylene peak in this range. *Analyze standards at least once every 10 samples, more frequently if necessary to describe any erratic behavior. (For standard preparation see below.)

4.C.8.c.3. Preparing Water Standards

Standards are prepared from ethylene saturated distilled water, to verify a linear relationship between water concentration and headspace concentration.

- Pour 50 ml distilled water into each of 2 septum bottles. For each bottle, run the aquarium hose with diffuser stone through a hole in the bottle cap. The bottle cap should be loose so that the pressure in the bottle is atmospheric pressure. Push enough hose through the hole so the airstone will rest on the bottom of the bottle. connect the other end of the hose to the gang valve.
- 2. Secure the ethylene tank to an immovable object <u>before</u> removing the cylinder cap.
- 3. Remove the cylinder cap and connect the ethylene regulator to the tank. Attach the reduction fitting to the ethylene regulator outlet and connect a length of aquarium hose to the fitting and the gang valve. Make sure the regulator needle valve and gang valves are closed.
- 4. Slowly (too fast may damage the regulators internally) open the cylinder valve. Set the regulator pressure to 3 psi and open the needle valve. Slightly open the 2 gang valves so that a good rate of bubbling occurs equally in each bottle. Record the water temperature and the ambient air pressure. Periodically check for a good ethylene gas flow.
- 5. After the distilled water has bubbled for 10 minutes, remove the cap, hose and airstone assembly from one bottle while leaving the gas flow on. Immediately cap the bottle. The object is to maintain a pure ethylene atmosphere in the bottle. Cap the other septum bottle and turn off the ethylene flow. Be sure to keep the capped bottles at a constant temperature.
- 6. Pour 10 ml of river water equilibrated to room temperature into each of 10 septum bottles. (Use one saturated bottle of the ethylene saturated distilled water to prepare all of the standards. The other bottle serves as a backup.) *Spike the bottles containing river water with varying amounts of saturated solution to make five different standards, in duplicate, which correspond to the expected range of river samples.
- 7. Stick the needle of a microsyringe about 1/2 inch into an inclined saturated bottle such that the needle point is well below the water surface. Slowly, draw the plunger all the way back several times to wet down the barrel with the water. This water film forms the seal needed. Then slowly draw the plunger back to the desired measure of water (no air bubbles should be present in the syringe). Gently pull the needle out of the septum.
- 8. Remove the cap from a bottle of river water and tilt until the water comes toward the mouth. Place the tip of the needle as far under the water surface as possible and gently inject the saturated water into the river

water. Immediately cap the bottle and then gently swirl the bottle to mix.

9. Analyze these samples with the River samples. See preceding section.

4.C.8.c.4. Sample Processing and Analysis

- 1. Bring the samples to room temperature. Vigorously shake the bottle to be analyzed for at least one minute to drive the ethylene into the headspace.
- 2. Select a clean gas-tight 1 cc syringe. Insert about 2 cm of the needle through the septum by holding the barrel and the needle. (This method will prevent needle damage.)
- 3. Flush the syringe once with air from the headspace and then withdraw the desired volume (usually 1 cc) and remove the syringe by holding the needle hub and lifting it straight out.
- 4. As quickly as possible, make the injection with the same method used to inject the standard. On the recorder paper, make a tic mark corresponding to the time of injection and number the injection.
 The GC is linear up to 10 ppm (v/v) with a 1 cc injection. Therefore, the sample should be diluted if necessary or injection volumes should be reduced so that the GC response is less than the response obtained by a 1 cc injection of 10 ppm (v/v).

4.C.8.c.5. Calculation of Ethylene Concentration

The following equation should be used to calculate relative ethylene concentration:

$$E = R*C$$

$$R_*iv$$

Where:

E= relative ethylene concentration in the water sample (v/v)R= response of GC to sample R_= response of GC to 1 cc of standard C = concentration of standard (v/v)iv= volume of original headspace sample injected into GC (cc)

Temp °C	Solubility mg/l	Temp °C	Solubility mg/l
0	283	16	170
1	273	17	165
2	264	18	161
3	255	19	157
4	247	20	153
5	239	21	149
6	230	22	146
7	223	23	143
8	216	24	140
9	209	25	137
10	203	26	134
11	196	27	131
12	190	28	128
13	185	29	126
14	180	30	123
15	174		

Solubility of Ethylene in Water at 1 Atmosphere

To correct for various air pressures use the following relationships:

$$S_{TP} = S_T * p$$

760

Where:

 S_T = solubility at ambient temperature and 1 atm. (mg/l)

P = ambient pressure (mm Hg)

 S_{TP} = solubility at ambient pressure and temperature (mg/l)

4.C.8.c.6. Troubleshooting and common errors

- 1. The most common problem encountered is a plugged needle. This can cause erratic results. Check needles before each injection by injecting some air into a small capful of water and checking for bubbles. Replace the septum on the GC after every 100 injections.
- 2. Inadequate notes are frequently a problem and prevent the analyst from reconstructing the previous events and evaluating which things may have caused a perceived problem.
- 3. Changed settings can cause the GC to give unexpected results. This can occur if any of the following things are changed: injection volume, air pressure or flow to the GC, room temperature, GC scale, span adjustment on back.

4. Standards analyzed too infrequently. Standards must be analyzed at least once every 10 samples and more frequently if necessary to describe any erratic behavior.

4.C.9. YSI MODEL 56 RECORDING DO METER

4.c.9.a. Instrument preparation

The day before leaving the shop:

- Check the supplies in the compartment within the cover: 1. chart paper, charged desiccant (should be blue), O ring, membranes for probe, electrolyte for probe, D.O. saturation table and instrument QA log.
- Attach probe and stirrer cable. Insert battery pack. 2.
- Turn power on and set the pen input to zero. If the pen з. does not read zero, adjust to read zero on the chart paper using the ZERO ADJUST knob.
- Turn pen input switch to BATTERY CHECK. A reading of 10 4. is fully charged. Recharge batteries if reading is <9.0.

4.C.9.b. Initial Calibration

The day before leaving the shop:

- 1. If the probe has not been stored in a calibration bottle, place a moist piece of tissue paper in the bottom of the small plastic calibration bottle. (The calibration bottle is a small plastic bottle with the bottom cut out. The calibration bottle ensures 100% humidity during calibration). Fasten protective collar to probe and slide the probe into the calibration bottle. The probe membrane must be completely dry but in a humid environment. Place the probe where the temperature will not change rapidly.
- 2. Wait ten minutes for temperature stabilization.
- 3. Set chart speed to 10 cm/hr; set pen input to $O_2 \& C^\circ$; set O_2 range to 0-10 mg/l; set O_2 filter to OFF.
- 4. Wait for the pen to draw a straight line on the paper. This verifies that the probe has stabilized (dashed line is temperature and solid line is D.O.). NOTE: probes with thick membranes will take longer to equilibrate.
- 5.
- Recheck the zero point. Switch to C° and read the temperature. 6.
- 7. Determine the O_2 saturation from the D.O. Saturation Table. Switch pen input to O_2 and adjust the O_2 Calib control until the pen traces at the appropriate reading for that temperature and elevation. Switch Chart Speed control back to 1 cm/hr.
- 8. Leave the meter on overnight to allow stabilization of the probe.

4.C.9.c. Locating the meter

Locate the meter in a secure location well above the likely high water mark. Hide it from view as much as possible to prevent vandalism. An ideal location is upstream of a sewage treatment plant, industry or other private property where the owner can keep an eye on the meter for you. Place the probe outside the chlorine plume of a wastewater treatment plant since chlorine can interfere.

Locate the probe where there is good flow. Secured it to a metal fence post that is pounded into the stream bed or to a bridge abutment or pilings. Secure the probe cable in such a way as to prevent violent oscillations in the current. Ideally, locate the probe in a shaded area to minimize fouling with algae. Locate the probe outside of the influence of the plumes of discharges or tributaries in order to get representative measurements. Be sure probe is well below the potential low water mark.

4.C.9.d. Maintenance

Replace the membrane if you observe any of the following:

- membrane is damaged or wrinkled
- bubbles have formed under the membrane
- erratic readings

- calibration is not stable or will not calibrate Membrane will normally need to be replaced every 2-4 weeks. Always store the probe in a humid environment such as in a plastic whirl-pac. Remove the batteries when you store the instrument. Install a fully charged battery pack each time the meter is serviced; at least every 7 days.

4.C.9.e. Dissolved Oxygen Measurement

4.C.9.e.1. Initial set-up

- 1. With the instrument prepared for use and calibrated, remove protective collar from probe and thread probe onto stirrer.
- 2. Measure and record the D.O. of river water that has been made anaerobic through the addition of excess sodium sulfite (1 gram/liter) and a trace of cobalt chloride.
- 3. Measure and record the D.O. of river water that has been saturated by pouring between two containers. Verify the saturation by demonstrating that the meter reading does not increase after additional aeration of the sample. The measurement of D.O. by the probe should be ± 0.8 mg/l of the theoretical value.
- 3. If not, then <u>re-check all maintenance and calibration</u> procedures. If the error is still greater than ±0.8 mg/l replace membrane. If the error is still greater than ±0.8 mg/l replace probe.
- 4. Collect a Winkler sample. Measure the stream temperature. Note meter D.O. and stream temperature on the lab analysis request form and the field sheet.
- 5. Place the probe in the stream and secure.
- Set chart speed to 1 cm/hr; set pen input to 0₂ & C°; set range to appropriate range (usually 0-20); set the power switch to STIR.
- 7. Mark the chart paper with date, time of day, location, chart speed and O_2 range. Measure the stream temperature and record on the chart.
- 8. Turn on filter.
- 9. Fill out the QA log and the field data sheet.
- 10. Double check all settings.
- 11. Secure the meter in a locked steel drum.

4.C.9.e.2. Field Maintenance

^aService the meter seven days maximum and more frequently at the beginning of a monitoring period.

- Check the probe and the meter for signs of vandalism, damage or other disruption that could affect data quality. Record your observations on the field data sheet.
- 2. Note and record the river D.O. as measured by the continuous meter.
- Check stirrer, membrane condition and battery level. Turn off filter.
- 4. <u>Before</u> making any adjustments, check the meter accuracy by measuring the D.O. of river water that has been saturated and that has been made anaerobic. Also check the reading in saturated air. Record this information in the QA log and on the field data sheet. (If you are removing the meter, take a Winkler.)
- 5. Replace the membrane if necessary.
- 6. Re-calibrate the meter and repeat the steps found in section 4.C.9.e. (Dissolved Oxygen Measurement) above.

4.C.9.e.3. Most Common Sources of Trouble

- The chart is not filled out with date/ time/ speed/ scale/ location.
- 2. Battery runs out before the meter is serviced. Always bring an extra battery so that you can verify the meter readings in the event of battery failure.
- 3. The filter is left on during calibration which causes a very slow response.
- 4. Bubbles or debris under the probe membrane.

4.C.10. PROCEDURES FOR FIXED STATION MONITORING

See GLEAS Procedure # 50

4.D. INLAND LAKE TROPHIC STATUS SAMPLING

4.D.1. INTRODUCTION

The purpose of this section is to outline recommended procedures for sampling activities related to inland lake trophic status determination. The procedures are specifically related to quantifying general water quality conditions of a lake. Some of the techniques are also applicable to other types of sampling in lakes such as point source investigations and Great Lakes monitoring. The objective of this section is to establish standardized field sampling techniques to insure obtainment of representative samples of lake waters with reproducible accuracy.

Lake surveys are typically conducted during spring turnover and/or summer stratification periods. Spring sampling is performed to determine the total phosphorus and other parameter levels of the lake basin(s) while the lake is well mixed. The total phosphorus level is indicative of the productivity potential of the lake. These data are used to identify baseline water quality; to support water quality based effluent limit recommendations for point source phosphorus discharges; and to determine water quality standards violations, water quality trends, and regional water quality characteristics.

Summer sampling is performed to determine profiles of important physical and chemical parameters in the lake basin(s) during maximum water column stratification, as well as to determine aquatic macrophyte distribution and density in the littoral zone of the lake. These data are used to determine lake trophic status as well as water quality standards violations and designated use impairments.

Lakes are generally classified by their physical, chemical and biological characteristics into one of several trophic categories (i.e oligotrophic, mesotrophic, eutrophic, hypereutrophic). The trophic state is a measure of a lake's biological productivity and includes such factors as nutrient levels, organic matter content, dissolved oxygen levels and water transparency. Although there are several methods used to classify lakes, the DNR utilizes the Trophic State Index (TSI) developed by Carlson (1977) and modified to account for aquatic macrophyte populations. The TSI values are derived from calculations based on Secchi disc transparency, chlorophyll <u>a</u>, and summer epilimnetic total phosphorus concentrations.

4.D.2. SAMPLING STRATEGY

Spring lake surveys should be conducted during spring turnover. Generally, lakes in Michigan turnover (e.g. become completely mixed) about one to two weeks after "ice-out". Time of ice-out depends primarily on geographic location and climatic conditions, but generally in Michigan it occurs sometime in April or early May. Complete mixing of a water column is indicated by uniform temperatures from top to bottom.

Summer lake surveys should be conducted during maximum stratification. Lakes in excess of 25 feet deep will begin to stratify after spring turnover and continue to stratify until late September or early October. Shallow lakes less than 25 feet

4.D.-1

deep may not stratify at all, or stratify periodically for short intervals throughout the summer. To ensure that lakes are sampled during maximum stratification periods, samples should be collected during late August or early September in the upper peninsula, and during September in the lower peninsula.

During either spring or summer surveys lakes should be sampled in the major deep basin. Multiple basins should be sampled if the configuration of the lake is such that it has two or more completely isolated basins where there is very little interchange of water between basins. This results in basins which may have very different nutrient characteristics as well as temperature and dissolved oxygen profiles.

The deep areas of a lake can be readily found using DNR hydrographic maps and a fathometer. If the lake has not been mapped, the deep area(s) should be located by making four or five transects (at a minimum) across the lake. Depths and relative locations should be noted while making the transects. The general shape of unmapped lakes should be obtained from U.S.G.S. topographic maps, prior to going into the field. Often the location of the deep basin can be determined by asking lake residents. Any information obtained from the residents, however, should be verified.

There is no standard list of parameters that would enable one to completely assess general water quality conditions in lakes. Under "normal" circumstances, at a minimum, lake surveys should include the following parameters: dissolved oxygen, temperature, pH, alkalinity, conductivity, major cations (Ca, Mg, Na, K), major anions (Cl, SO₄), ortho and total phosphorus, nitrate/nitrite, ammonia, Kjeldahl nitrogen, and chlorophyll <u>a</u>. In addition, water column transparency should be measured at each sampling site.

Measurements or samples are taken at different depths throughout the water column at each site to characterize the stratification profile of the lake basin.

4.D.3. SAMPLING PROCEDURE

All pertinent safety procedures as outlined in the Land and Water Management Division's Safety Program Manual must be followed while sampling lakes. Proper planning and preparation are important for a successful lake sampling trip. Use the equipment checklist when loading the vehicle for the trip and file an itinerary with the office Chief prior to departing on the trip.

State highway maps, county maps, and DNR hydrographic maps will aid in locating the boat launching sites. The launching ramps will range from hard-surfaced to gravel and sand-surfaced ramps. For many lakes, information on launching ramps' surface types are included in the DNR's Public Boat Launch Directory. Before launching the boat, carefully evaluate the condition of the ramp as well as the water depths at the launch site.

All sampling equipment, instruments, bottles, preservatives, anchor, motor, gas tank, and safety equipment should be loaded into the boat prior to launching. The lake sampling notebook which includes laboratory analysis request forms, field data sheets, hydrographic maps, sampling procedures, sample preservation instructions, and instrument operating manuals should be carried into the boat.

All pH measurements are made on-shore in the vehicle after the samples are brought in from the lake. The pH meter should be set up in the back of the vehicle and turned on to standby mode before departing for the sampling site. The temperature/conductivity meter should be calibrated on-shore prior to sampling as described in the conductivity calibration procedures below.

After launching, motor directly to the sampling site using the hydrographic map, fathometer, and shoreline features. Approach the sampling site heading into the wind. Overshoot the site and carefully lower the anchor to minimize sediment disturbance. Let out enough rope to drift back over the sample site. In this process avoid contamination of the site by the boat motor exhaust gases and oil. Once securely anchored, the date, time and weather conditions (i.e. wind velocity and direction, air temperature, cloud cover, and precipitation) should be noted on the field data sheet.

When sampling, try to minimize the possibility of contamination. Instrument and transparency measurements should be taken first. Water samples should be collected next by sampling the various depths starting at the surface and working towards the bottom. Caps should not be removed from the sample bottles until filling the bottles. Bottle caps should always be held in your hand rather than laid down in the boat. If a water sample appears nonhomogeneous (e.g., some large suspended or floating particles) discard and re-sample. If at any time you think the water column has been contaminated you should either move at least 200-300 feet from the current site but still within the deep basin to re-sample or leave the site and return the next day. If sediments are to be sampled, these samples should be the last samples collected from each site. This will prevent sediment contamination of the water samples.

After sampling be sure to clean up all equipment. Rinse sediment off ropes, sampling devices, and the anchors. This will minimize contamination of the next sampling site.

For safety concerns, lake sampling is done by two or more people. It is most efficient if one person determines the Secchi disc transparency and collects the chlorophyll \underline{a} sample while the other person does the temperature/conductivity profiles and locates the thermocline. They then proceed with the other samples to be collected. The specific techniques are outlined in the paragraphs below.

4.D.3.a. Secchi Disc Measurements

A Secchi disc (Figure 4.D.-1) is used to determine the transparency of the water. Transparency is affected by many factors, including phytoplankton, suspended solids, and natural water color. The water column depth equal to twice the Secchi disc depth is considered to represent the euphotic zone. This is the depth that has sufficient light for the growth of aquatic plants (Figure 4.D.-2). Secchi disc readings should be taken between 10:00 a.m. and 4:00 p.m. if possible. Transparency measurements should not be taken when the lake is very rough or when the lake is being used by a large number of motor-powered boats which may agitate bottom sediments and reduce water clarity.









2/94

Use the following steps when measuring transparency.

- -- Anchor the boat before measuring the transparency to ensure that the Secchi disc is observed straight down, instead of on an angle.
- -- Attach a graduated line (one foot increments) to the disc.
- -- Disc measurements should be obtained by lowering the disc into the water on the shaded side of the boat. The observer should lean over the side of the boat, directly over the disc as it is lowered. The depth at which the disc just disappears is noted. It is then raised slowly until it again becomes visible and that depth is noted. The point half-way between the two readings is the Secchi disc measurement. The disc should then be raised several feet and the procedure repeated and an average for the two values calculated. Round results to the nearest one-half foot (e.g., 6, 6½, 7, 7½ feet) and record on the field data sheet. If Secchi disc is visible at the bottom, note this on the field data sheet.

4.D.3.b. Chlorophyll <u>a</u> Samples

After the Secchi disc depth has been determined, measure out twice this length of rope and mark it with a clip or knot. This is the depth of the euphotic zone through which the depth integrated composite water sample for chlorophyll \underline{a} will be collected.

Use the weighted sampling container with two tubes through the cap. The tubes provide escape of air and uniform entry of water while the container sinks and then is retrieved through the water column. See Figure 4.D.-3 for an example of a chlorophyll \underline{a} sampler.

The sampling container should be rinsed with lake water at each site prior to collecting the sample. Lower the sampler to a depth of twice the transparency and then raise it to the surface. The sampler should be lowered and raised at a uniform speed such that the sampling container is slightly less than full as it reaches the surface. If the sampler is full, do not use it for the sample. Empty it and re-sample. The size of the opening in the sampler and/or speed of lowering and raising should be adjusted as necessary. The object of this procedure is to ensure that water is collected from the entire euphotic zone. Figure 4.D.-2 diagrams this procedure.

Extreme care must be taken not to contaminate the sample by resuspending the bottom sediments. Therefore for shallow lakes with depths less than twice the Secchi disc transparency, the sampling container should not be lowered to a depth closer than three feet from the bottom. Invery shallow lakes (ie. <10 feet) the sampling container may be carefully lowered closer to the bottom.

A subsample should be poured into a 250 ml brown bottle, preserved with one drop $MgCO_3$, and handled according to the procedures in Chapter 3, Sample Preservation and Handling.



.



Phytoplankton (Chlorophyll) Sampler Construction Plans

Chlorophyll <u>a</u> samples only have a 48 hour holding time prior to filtering. If these samples can not be delivered to the lab within this time period, they must be field filtered and stored in 90% acetone as per the following procedures:

- -- The chlorophyll <u>a</u> field filtering apparatus consists of a Millipore vacuum pump, filter funnel, and flask.
- -- Connect the vacuum end of the pump to the filter funnel with a high-pressure rubber hose and secure filter funnel to flask.
- -- Place a 0.45 u membrane filter on suction pad and secure funnel cup.
- -- Wet pad with chilled distilled water, turn on vacuum pump, and set vacuum to 12 to 15 in. Hg. Turn off vacuum pump.
- -- Shake MgCO₃ preserved sample vigorously and pour a 50 ml aliquot into a graduated cylinder.
- -- Pour the 50 ml aliquot into the funnel, wash sides of graduated cylinder with chilled distilled water into funnel, and turn on vacuum pump.
- -- When all of the sample has filtered through, wash sides of filter cup with chilled distilled water and allow all water to filter through. Turn off vacuum pump.
- -- Remove the filter cup, then carefully remove the membrane filter from filter pad using forceps.
- -- Fold membrane filter into quarters and place in fluted test tube.
- -- Dispense 10 ml of 90% acetone into test tube and cap with cork stopper. The membrane filter should dissolve in the acetone.
- -- Label test tube and cork stopper with lake name and date, mark a line on outside of test tube at level of acetone, and seal cork stopper with parafilm wrap.
- -- Store test tubes in dark container on ice in ice chest.

4.D.3.c. Temperature and Conductivity

Lake water column profiles of temperature and conductivity are measured using a YSI model 3000 T-L-C meter. Generally, these parameters are measured at ten equally spaced depth intervals between the surface and within three feet of the bottom. If the lake basin is less than 50 feet deep, sample every 5 feet. For lakes designated as coldwater fishery lakes by the DNR, additional measurements should be taken at 212 foot intervals through the thermocline. Record all measurements on the field data sheet.

The T-L-C meter should be calibrated before and after each

sampling season using the calibration test probe (YSI model 3040). The calibration of the meter should be verified daily while in use by using the 147 umho and 291 umho conductivity standards prepared by the Lab. Notes on the calibration should be recorded on the field data sheets. Refer to the instructions manual for the YSI model 3000 T-L-C meter for specific operating information. For other temperature and conductivity meters, see Introduction and General Procedures, Section 4.A. of this chapter for procedures.

4.D.3.d. Dissolved Oxygen

Water column dissolved oxygen will often vary from 10 to 0 mg/l between the surface and bottom of a lake. Although D.O. meters are convenient to use, they cannot be calibrated to accurately measure oxygen over this extended range. Additionally, hydrogen sulfide which is often present in the D.O. depleted hypolimnetic waters of lakes will interfere with the measurements and may ruin the probe electrode. Therefore, it is recommended that the Winkler method for D.O. analysis be used for lakes (See Introduction and General Procedures, Section 4.A. of this chapter for specific information on the Winkler method).

Dissolved oxygen samples should be collected at the surface, midwater column, and three feet off the bottom of the lake during the spring sampling season. For lakes that do not turnover every spring, additional D.O. samples should be collected at 10 foot intervals and at additional depths through the thermocline. For summer stratification, D.O. samples should be collected at the same depths that temperature and conductivity were measured. For lakes designated as coldwater fishery lakes by the DNR, additional D.O. samples should be collected at 21/2 foot intervals through the thermocline. For shallow lakes that do not stratify during the summer, D.O. samples should be collected only at the surface, midwater column, and three feet off the bottom of the lake. Dissolved oxygen samples should be collected in standard glass BOD bottles using a Kemmerer sampling container as described below. Dissolved oxygen samples should be titrated in the field within eight hours of collection using the procedures outlined in the Introduction and General Procedures, Section 4.A. of this Chapter. All results should be recorded on the field data sheet.

Water samples at specific depths are taken using a Kemmerer, VanDorn or other similar depth type sampler. This document discusses the use of the Kemmerer sampler. When using other equipment, obtain field procedure guidance from manufacturers.

The Kemmerer sampler is a long, cylindrical container, approximately one and one half to two feet in length, open from both ends which can be remotely closed and sealed to collect a discrete sample at various depths. A Wildco brass 3.2 L Kemmerer sampler is used for all inland lake sampling.

Procedures for using the Kemmerer sampler are as follows:

- -- Use a rope or line marked in one foot intervals with waterproof paint, ink, nail polish, or tape. Mark with different colors at 5 or 10 foot increments.
- -- A seven or eight inch plastic tube should be connected to the

3/94

sampler outlet mechanism to eliminate air bubbles in the dissolved oxygen samples.

- -- Open both ends of sampler.
- -- Lower to desired depth, being careful not to touch bottom and disturb sediments. Start by sampling the depth closest to the surface and proceed down to the next lower depth.
- -- Drop messenger down to trigger closure of sampler at the desired depth. After closing the sampler at the bottom depth, lower the sampler to verify that you are within three feet of the bottom.
- -- Retrieve and fill sample bottles.

The dissolved oxygen bottle should be the first bottle filled from each Kemmerer grab sample. Fill the D.O. bottle by running water down the inside of the bottle with no agitation. Overfill the bottle by 2 to 3 times and insure that any air bubbles are removed before capping.

4.D.3.e. pH

Water column samples for pH measurements are taken from the Kemmerer grab samples at surface, mid-water column, and three feet off the bottom depths for spring turnover sampling. For summer stratification sampling, these samples are taken from the surface, mid-thermocline, and three feet off the bottom depths. All pH samples should be kept in plastic 250 ml bottles and stored in a styrofoam cooler until analyzed. The samples are analyzed onshore using a Sargent Welch model 2000 pH meter. The pH meter should be calibrated against pH 7 and pH 10 buffers according to the following procedures:

- -- Adjust center outer (clear) dial to 100% on the white slope line.
- -- Adjust center inner (black) dial to temperature of the buffer.
- -- Insert probe in pH 7 buffer; adjust intercept knob so meter reads "7".
- -- Rinse probe and insert into pH 10 buffer; adjust center inner (black) dial so that the meter reads "10".
- -- Adjust center outer (clear) dial to correct temperature. This determines the efficiency of the probe.
- -- The center inner (black) dial should be adjusted to the temperature of the individual samples.
- -- Check calibration at each lake.

For other pH meters, see Introduction and General Procedures, Section 4.A. of this Chapter for calibration procedures.

Refer to the instructions manual for the Sargent Welch model 2000 pH meter for specific operating information and glass electrode

3/94

storage and care instructions. Batteries should be replaced prior to spring turnover sampling each year and, if needed, prior to summer stratification sampling.

4.D.3.f. Alkalinity, Major Cations and Anions

Water column samples for alkalinity and major cations (Ca, Mg, Na, K) and anions (Cl, SO₄) are taken from the Kemmerer grab samples at surface, mid-water column, and three feet off the bottom depths during spring turnover sampling. These samples are kept in plastic 250 ml bottles and they should be preserved and handled in accordance with the procedures in Chapter 3 of this manual. Generally, alkalinity and the major cations and anions are only measured once per lake for background levels.

4.D.3.g. Nutrients

Water column samples for ortho and total phosphorus, nitrate/nitrite, ammonia, and Kjeldahl nitrogen are also taken from the Kemmerer grab samples at discrete depths for each lake. For spring turnover sampling, these samples are collected at the surface, mid-water column, and three feet off the bottom of the lake. For summer stratification sampling, these samples are taken at the surface and at three feet off the bottom of the lake as well as at the mid-thermocline depth. Nutrient samples are kept in plastic 250 ml bottles and they should be preserved and handled in accordance with the procedures in Chapter 3 of this manual.

4.D.3.h. Sediment Samples

Sediment samples should be collected by slowly lowering an Eckman dredge to the bottom being careful not to dissipate the sediment upon impact or flush it through the top of the dredge. Sediment should be skimmed off the top with a teflon spoon or scoop to obtain the most recently deposited material. For paleolimnological analyses, sediment cores should be taken, however for general sediment analyses, the surficial sediment layer is most representative of current lake conditions. When both inorganic and organic parameters are to be analyzed, a single sediment sample should be prepared, thoroughly mixed and then subsampled. This will insure that each sample is approximately the same percent of total solids. Both percent total solids and percent organics should be run on each sample to allow for inter sample comparisons.

4.D.3.i. Macrophytes Survey

A qualitative macrophyte survey is conducted for each lake during the summer sampling period to determine general aquatic plant distribution and density around the lake. For most lakes, 300 to 500 foot sections of the littoral zone along each orientation of the lake (usually four or more areas) are surveyed for macrophyte types and density. The back of the field data sheet is used to tally the species present and location as well as their density in general terms (i.e. found, sparse, moderate, dense, impaired use). A garden rake is used in shallow water and a weighted multi-tined grabbling hook sampler is used for deeper water to bring the aquatic plants to the surface for identification.

4.D.4. EQUIPMENT INVENTORY CHECKLIST

Boat, motor, gas tank, oars, anchor, and safety equipment Fathometer: meter, transducer, battery pH Meter, extra glass electrode, standard buffers

T-L-C Meter, conductivity standards, pocket thermometer Kemmerer sampler Secchi disc Composite chlorophyll <u>a</u> sampler Filter pump and apparatus for chlorophyll <u>a</u> samples Rope or line graduated in one foot increments Appropriate sample bottles and preservatives Hydrographic maps, county maps, state highway map Field notebook, field data sheets, and lab sheets

4.D.5. FIELD NOTES

Frequently not enough notes are taken while in the field. The more information that is obtained, the more meaningful the analytical data will be. At a minimum, field notes should include the following:

Name of sampler Lake name and location (county and township) Date and time of sample collection Precise description of sampling location on the lake (include basin depth) Weather conditions at time of sampling Depths sampled Location and characteristics of the thermocline Percent of shoreline development Apparent water color Description of bottom material type Macrophytes -- Species composition -- Relative abundance (sparse, moderate, dense, etc.)

4.E. AQUATIC SEDIMENTS SAMPLING

4.E.1. INTRODUCTION -THE SEDIMENTARY PROCESSES

Sediments of freshwater systems can act as reservoirs for hazardous substances in the aquatic environment. Freshwater ecosystems contain dissolved and suspended particles of organic and inorganic substances originating from municipal, industrial and agricultural wastes, decomposing organic matter, and soil erosion. Sediments accumulate when these materials are deposited on stream or lake beds. Table 4.E.-1 shows the size range of various sedimentary materials, as well as the current velocities required to move particles of various sizes along a stream bed.

Metal and organic contaminants are primarily associated with particulate matter in water and are conveyed to underlying deposits bound to finegrained materials such as organic debris, hydroxides of iron, and manganese or clay minerals.

The primary mechanism for this association is thought to be adsorption - a poorly understood process involving both physical factors (typified by van der Waals forces) and chemical factors. Adsorption of pesticides on charged clay mineral surfaces can occur through various forms of ionic bonding. Several hydrogen bonds utilizing oxygen atoms on the clay surface or edge hydroxyls have been suggested as possibilities for bonding organic compounds to clay minerals. Cationic pesticides such as diquat are adsorbed on clay minerals by ion exchange reactions. Acidic pesticides such as 2,4-D are only weakly adsorbed by particulate matter via anionic bonding. Basic pesticides such as Triazine herbicides are physically adsorbed to particulate matter in neutral solutions, but they become protonated and ionically adsorbed in acid systems or on acid surfaces. The adsorption of polar pesticides to organic matter may also be considered of this nature due to the high cationic exchange capacity and high surface area of soil humus.

Adsorption of non-polar pesticides such as chlorinated hydrocarbons (e.g. DDT, dieldrin) cannot be adequately explained in terms of ionic bonding. Rather, the process might be explained by hydrophobic bonding similar to that in living systems where lipid tissues are primary concentration sites for chlorinated hydrocarbons such as DDT and polychlorinated biphenyls. Chlorinated hydrocarbons have low water solubilities. Since these compounds do not readily dissolve and they do not form hydrogen bonds with water, they are readily lost from: the solution phase and adhere to lipophilic substances present in soil organic matter.

The above mechanisms describing the binding of organic compounds to clay and organic matter have been studied in detail. Although there is a lack of information on hydrated sedimentary systems, some of these same processes may occur in sediments. For instance, behavior of pesticides in sediments may be inferred from data describing reactions in moist soil. In general, moisture tends to affect pesticides with polarizable moieties more than non-polar chlorinated hydrocarbons.

Table 4. E. - 1

Relationships between stream velocity and sediment particle size

	Critical mean current velocity cm./sec.			
Type of bed	Clear water	Muddy water		
Fine-grained clay	30	50		
Sandy clay	30	50		
Hard clay	60	100		
Fine sand	20	30		
Coarse sand	30-50	45-70		
Fine gravel	60	80		
Medium gravel	6 080	80-100		
Coarse gravel	100-140	140-100		
Angular stones	170	180		

TABLE A The mean current velocity of clear and muddy water required to initiate movement along a stream bed of various types of bottom deposit. Modified from Schmitz (1961).

<u>^.</u>	
Speed of flow cm./sec.	Dianicter of mineral particles moved, em.
10	0.3
25	1.3
50	5.0
75	11.0
100	20.0
150	45.0
200	80.0
300	180.0

TABLE B. The speeds of flow required to move mineral particles of different sizes. From Nielsen (1950)).

		-			
		Mesh size and methods of measurement			
Range of size in mm.	phi- scale	mm.	Approx. in. equivalent	U.S. sieve no.	Tyler sieve no.
>250	-3	Direct me:	surement		
64-256	-6-7	Individual wire square			
32-64	-5	Individual wire square			
16-32	-4	16	0.625 (15.9	mm.)	
8-16	-3	S	0.312 (7.93 mm.)		
4-3	-2	4	0.137	5	5
2-4	— I	2	0.0282	10	9
1-2	0	I	0.0394	18	15
0·5-I	I.	0.2	0.0192	35	32
0.23-0.3	2	0.25	0.0008	60	60
0.125-0.25	3	0.125	0.0049	120	115
0.0625-0.125	4	0.0625	0.0054	230	250
0.0039-0.0025	5, 6, 7, 8	Separated by settling			
<0.0030	9	Separated	by centrifuge		
	Range of size in mm. > 256 64-256 32-64 16-32 8-16 4-3 2-4 1-2 0.5-1 0.25-0.5 0.125-0.25 0.0625-0.125 0.0039-0.0625 < 0.0039	Range of size in mm. phi-scale > 256 -3 $64-256$ $-6-7$ $32-64$ -5 $16-32$ -4 $8-16$ -3 $4-3$ -2 $2-4$ -1 $1-2$ 0 $0.5-1$ 1 $0.25-0.5$ 2 $0.125-0.25$ 3 $0.0525-0.125$ 4 $0.039-0.0625$ $5, 6, 7, 8$	Mesh size a of measurem Range of size in mm. phi-scale > 256 -3 Direct measurem 64-256 $-6-7$ Individual 32-64 -5 Individual 16-32 -4 16 8-16 -3 8 4-3 -2 4 2-4 -1 2 1-2 0 I 0.5-1 I 0.5 0.5-25-0.5 2 0.25 0.525-0.55 3 0.125 0.525-0.125 4 0.5625 0.5039-0.0625 5, 6, 7, 8 Separated <0.039	Mesh size and methods of measurementRange of size in mm.phi- scaleApprox. in. mm. > 256 -3 Direct measurement $64-256$ $-6-7$ Individual wire square $32-64$ -5 Individual wire square $16-32$ -4 16 0.52 -4 16 0.52 -4 16 0.52 -4 16 0.52 -4 16 0.52 -4 16 0.52 -2 4 $0.5-1$ 1 0.577 $0.5-1$ 1 0.5 $0.5-1$ 1 0.5 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 2 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 1 0.52 $0.5-1$ 2 0.52 $0.5-1$ 2 0.52 $0.5-2$ 0.52 $0.5-2$ 0.52 $0.5-2$ 0.52 $0.5-2$ 0.52 $0.5-2$ 0.52 $0.5-2$ 0.52 $0.5-2$ 0.52 $0.5-2$ 0.52 $0.5-2$ 0.52 $0.5-2$ <td< td=""><td>Mesh size and methods of measurement Range of size in mm. phi-scale Mm. Approx. in. U.S. >256 -3 Direct measurement sieve no. >256 -6-7 Individual wire square 32-64 -5 Individual wire square 16-32 -4 16 o.625 (15.9 mm.) 8-16 -3 S o.312 (7.93 mm.) 4-3 -2 4 o.157 5 2-4 -1 2 o.0787 10 1-2 0 I o.0394 18 o.5-1 I 0.5 o.0197 35 o.25-0.5 2 o.25 o.0098 60 o.125-0.25 3 o.125 c.0049 120 o.0625-0.125 4 o.0625 o.0024 230 o.039-0.0025 5, 6, 7, 8 Separated by settling <0.039</td> 9 Separated by centrifuge</td<>	Mesh size and methods of measurement Range of size in mm. phi-scale Mm. Approx. in. U.S. >256 -3 Direct measurement sieve no. >256 -6-7 Individual wire square 32-64 -5 Individual wire square 16-32 -4 16 o.625 (15.9 mm.) 8-16 -3 S o.312 (7.93 mm.) 4-3 -2 4 o.157 5 2-4 -1 2 o.0787 10 1-2 0 I o.0394 18 o.5-1 I 0.5 o.0197 35 o.25-0.5 2 o.25 o.0098 60 o.125-0.25 3 o.125 c.0049 120 o.0625-0.125 4 o.0625 o.0024 230 o.039-0.0025 5, 6, 7, 8 Separated by settling <0.039

TABLE C Suggested terminology, categories, and methods for particle-size analysis. Modified from Cummins (1962).

The organic matter in soil has been found to form bonds with pesticides that are resistant to aqueous leaching, while clay mineral soils have been shown to reversibly hold and release pesticides in dry and moist conditions respectively.

The inter-action of metals with the sediments is usually more straightforward. Most metallic salts completely dissociate to ionic forms in water. Sedimentary deposits will therefore harbor these salts as a mix of ions. Some metallic salts such as mercuric chloride (HgCl₂) and cadmium sulfate (CdSO₄) only weakly dissociate in water and are found intact in sediments.

The process of adsorption can retard bio-degradation. Cationic pesticides and nonionic pesticides with low water solubilities such as DDT and dieldrin are immobile in soil systems; that is, their biological activity is greatly reduced or nonexistent in the adsorbed state. In contrast, the weakly adsorbed acidic pesticides are mobile in soil and aquatic systems, while the biological activity of basic pesticides are greater under alkaline or neutral conditions than under acid conditions. However, pesticides and metals immobilized in sediments by adsorption may enter the food chain through ingestion by mobile life forms, absorption by roots or leaves of plants, or chemical leaching into the surrounding medium. Thus, these contaminants can pose a very real threat to the aquatic environment.

4.E.2. SEDIMENT SAMPLING PROCEDURES

Proper collection, preservation and storage of sediment samples is vital to the process of contaminant level determination. A sampling program must be planned to satisfy the objectives of the study, yet remain within the limitations of available manpower, time, and money. The survey should use the minimum number of sediment samples that adequately represent effluent sites or the body of water from which they were taken. A composite sample (composed of three or more individual samples) from an area with a similar sediment type, should be used unless the objective of the study is to determine the variability of contaminants in the area.

Whenever possible, sample collectors should be very familiar with the location of sampling sites before beginning collection procedures. Since contaminants adsorb to fine particulate material including organic debris, the collector should look for depositional zones of fine silts and avoid sand or gravel. When the study involves a lake, the sampler should use a hydrographic map of the lake bottom: to locate such depositional zones. The following sites should be considered for inclusion in any sampling program; inlets and outlets, known or potential point and nonpoint sources of pollution, across the long axis of the water body via grids or transects, and recreational areas such as bathing beaches. Potential sampling sites on a lake are shown in Figure 4.E.-1.

Small streams should be sampled at background stations upstream from the source of contamination, at the suspected source, and downstream of the source at various distances to delineate zones of pollution. Depositional zones of fine sediments are usually located along and just below the concave bank of a curve in the stream, or among bars and eddies formed when the stream widens. A meandering stream has deposits of fine sediments downstream of the midpoint of the curve on the convex bank. Larger streams are usually not well mixed laterally for long distances downstream from the pollution sources. Thus quarter-point sampling below the pollution source will reveal channeling. Sampling sites on a large stream are shown in Figure 4.E.-2.

Certain conditions may be more conducive to sediment sampling. For instance, better results may be obtained if sediments at a source of groundwater contamination or near agricultural runoff are sampled after a heavy rain. Also, prevailing winds may favor the build-up of contaminants on the leeward side of a lake. One study suggests that fall overturn in a stratified lake resuspends littoral deposits of fine sediment particles and redistributes them across the entire basin.

Sediment samples must be representative; that is, pertinent components must be the same in the sample as in the material being sampled. To meet this criteria, sampling equipment and handling procedures should be designed so that no significant changes occur in the sample before tests are performed.

Samples for organic and metal contaminant analysis should be collected and transferred immediately into clean glass containers with screw caps lined with Teflon. Clean bottles should be provided by the laboratory, however, if such containers are not available bottles and caps should be thoroughly cleansed with detergent and hot water, followed by a hot water rinse to remove all traces of detergent. Finally, bottles and caps should be rinsed three times with laboratory pure water. To prevent adsorption of organic constituents to the container, rubber, cork and most plastics should be avoided in sampling equipment and storage containers. Some plastic containers such as the sterile whirl-pak plastic bags can be used as the convenience of the container outweighs the minimal adsorption and phthalate leaching that can occur. Also, a field notebook should be completed, with a description of each sampling station and the physical characteristics of each sample. Other pertinent information such as weather conditions, air-water temperatures, stream flow, turbidity, and presence of aquatic vegetation should be included.

For sampling sediments in the shallows, a 250 ml wide mouth glass bottle can be used to carefully scoop out fine sediments of the appropriate depositional zone by hand. The sampler should take care not to alter the sample by stirring up the bottom. After collection, let the sediment settle in the bottle for a few minutes, decant any excess water, then seal with a Teflon-lined cap. Attach a sample label to the bottle as described in Chapter 6, Chain of Custody.

4.E.3. SAMPLING GEAR

The Van Donsel-Geldreich sediment sampler shown in Figure 4.E.-3 collects sediment in sterile whirl-pak plastic bags down to 60 foot depth. This device operates in the following way. The bag mouth is attached to a nosepiece, and the bag is kept shut during descent by a bag clamp bar. As the mud plate base contacts the bottom, the nosepiece is driven into the sediment by the weight of the sampler. As the nosepiece moves downward, the bag slides through the bag clamp bar, opens, and fills with sediment. The bag is sealed when the double noose tied to the bottom of the bag is pulled, before the apparatus is returned to the surface. Grab samplers can also be used to collect sediments, and each type has definite advantages for sampling particular substrates. The Petersen grab (Figure 4.E.-4a) is used for sampling hard bottoms such as sand, gravel, marl and clay in swift currents in deep water. It is fairly heavy (30 lb - 70 lb with auxiliary weights) and is_manufacture in_sizes that will sample an area of 0.06 to 0.09 m (.6 to 1.0 ft). To operate, set the jaws open and lower it slowly to the bottom to avoid disturbing lighter bottom materials. Rope tension is then eased to release the catch. A lever closes the jaws as the grab is raised.

The Ponar grab (Figure 4.E.-4d) is used in deep lakes and is capable of sampling the widest variety of substrates. Its size, weight and lever system are similar to those of the Petersen grab, although the sample compartment has side plates and a screen on the top to prevent loss of a sample during closure.

The Ekman grab (Figure 4.E.-4e) is useful for sampling softer substrates such as silt, mud, and sludge in waters with little current. This lightweight (7 lb) device cannot be used on rocky or sandy bottoms as small pebbles or grit may prevent proper closure of the spring-operated jaws. These jaws on the bottom of the sample compartment must be manually locked before the device is lowered into position The top of the grab has two hinged overlapping lids. These lids are held partially open by water passing through the sample compartment for an easy, straight descent. Water pressure keeps these lids tightly closed when the sampler is being retrieved. The sampler is made in three sizes, although the smallest (15 cm) is usually the most desirable for taking replicate samples.

The 100 lb Smith-McIntyre grab (Figure 4.E.-5c) can only be operated from a large boat equipped with a winch. However, its bulk and weight make it very stable and easy to control in rough water. This device can sample an area of 20 m (2.15 ft).

The Shipek grab (Figure 4.E.-5a) is used primarily on large lakes and marine waters. The sample compartment is composed of two concentric half cylinders. When the grab touches bottom, inertia from a self-contained weight releases a catch and helical springs rotate the inner half cylinder 180 degrees. The sample bucket may be disengaged from the upper semicylinder by releasing the two remaining latches. This device is designed to take a sample 0.04 m (1 in x 8 in) in surface area, and approximately 10 cm (4 in) deep at the center.

The orange peel grab (Figure 4.E.-5b) is used to sample sandy substrates in deep water. This multijawed round grab has a canvas closure at the top serving as part of the sample compartment. The 1600 cm (100 in) size is generally used.

Core samplers are used to sample sediments in depth. Such samples can be used to document historic contamination. The area collected at the mud-water interface is small (2 to 4 in). The widely used Phlegler core

(Figure 4.E.-4b) operates on the gravity principle. Designs vary among manufacturers--some have interchangeable weights that allow variations between 17 and 77 lbs., while other fixed weight designs weigh 90 lbs or more. The Wilding or stovepipe sampler (Figure 4.E.-4c) is especially useful in sampling sediments among dense vascular plant growth. In a study of Southern Lake Huron sediments, the EPA used an improved core sampler manufactured by Benthos, Inc., of N. Falmouth, Massachusetts. The plastic core liners were outfitted with a butterfly valve which decreased resistance to water flow through the core during descent through the water column and upon impact with the sediments. This alteration, plus the procedure of lowering the sampler to a position 5-10 meters above the sediments before allowing it to fall freely, kept disturbance of surface sediments to a minimum.

Sediment samples obtained using dredges and corers should be processed as soon as possible to ensure that the sample is truly representative. After emptying the dredge carefully into a pan, the operator should use a clean 250 ml wide mouth glass bottle to scoop out the sample from the center of the material. The bottle should be sealed with a Teflon-lined cap, and labeled as discussed above.

Cores are removed from the coring device (smaller hand corers usually have a plunger, while longer cores may need to be removed by a hydraulic extruding device) and sectioned. In a coarse sectioning scheme, the core is cut into the following lengths: 1 cm intervals to 14 cm, 2 cm intervals to 30 cm, and 5 cm intervals to 60 cm depth or to the bottom of the core. In a fine sectioning scheme, cuts are made at these points: .5 cm intervals to 10 cm, 1 cm intervals to 20 cm, 2 cm intervals to 30 cm, and 5 cm intervals to 60 cm depth or to the bottom of the core.

The sections are placed in whirl-pak plastic bags and labeled. The label should include core length along with the other information mentioned above.

All sediment samples, whether collected by hand, dredge, or coring device, must be preserved if the sample is to maintain its integrity. Freezing is the best method. This can be done by placing the samples in an insulated container with dry ice. If freezing is not feasible, the samples should be kept at 4°C in insulated containers filled with ice. Sediment samples for certain toxic material analyses must not be held longer than two days prior to laboratory testing. To avoid photodegradation, samples should always be held in the dark when possible.

The field collector is responsible for the samples collected until they are properly dispatched to the receiving laboratory. This responsibility extends to making sure that samples are assigned laboratory I.D. numbers and that these numbers accurately correspond to the samples described on the laboratory sheet. Laboratory receipts for samples submitted should be maintained by the sample collector until analytical results are received.

For samples requiring expedited (priority) analysis, Chapter 2 - Laboratory Priorities and Scheduling, should be consulted.



FIGURE 4.E.-I

Sampling a Lake or Impoundment. (A) inlets, (B) potential source of pollution, (B^1) village, (B^2) agricultural run-off, (B^3) home septic tank, (C) multi-point transect, (D) bathing beach and (E) outlet above and below dam.


FIGURE 4.E.-2

Sampling a Large Stream. (A) control station, (B) agricultural pollution, (C) industrial discharge, (D) quarterpoint transect (E) tributary, and (F) downstream monitoring.



FIGURE 4.E.-3

Van Donsel-Geldreich Sediment Sampler. (A) sterile
"Whirl-Pak" plastic bag, (B) nose piece, (C) weight,
(D) mud plate, (E) slide bar, (F) part of the double
noose, (G) attachment for the suspension line and
(H) bag clamp bar.



Figure 4. E-4a. Petersen grab.

Figure 4. F-4b. Phleger core sampler.



Figure 4 E -4c. Wilding or stovepipe sampler.



Figure 4 -4d. Ponar grab.





Figure 4 F -4e. Ekman grab.





FIGURE 4. E-5

4.F. QUALITATIVE BIOLOGICAL AND HABITAT SURVEYS IN WADABLE STREAMS See GLEAS Procedure #51

4.G. FISH TASTE AND ODOR STUDIES
 See GLEAS PROCEDURE #55)

4.H. CAGED FISH CHEMICAL BIOCONCENTRATION STUDY PROCEDURE See GLEAS PROCEDURE #62

4.I. FISH CONTAMINANT MONITORING
 See GLEAS Procedure #31

4.F.-1

CHAPTER 5

FIELD QUALITY CONTROL PROCEDURES

Prepared by State of Michigan Department of Natural Resources September 1994

Table of Contents

5.A.	INTRODUCTION		•	•	•	•	•	•	•	•	•	•	•	•	•	•	٠	•	•	•	•	•	•	•	1
5.B.	FIELD BLANKS		•	•	•	•	•	•	•	•	•	٠	•	•	•	•	•	•	•	•	•	•	•	•	1
5.C.	FIELD REPLICATES		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	o	•	•	•	3
5.D.	BLIND DUPLICATES		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	3
5.E.	SPLIT SAMPLES		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	3
5.F.	FIELD SPIKES	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	4
5.G.	LAB MATRIX SPIKES	5	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	4

CHAPTER 5. FIELD QUALITY CONTROL PROCEDURES

5.A. INTRODUCTION

An integral part of any quality assurance program includes field QC samples. These are particularly valuable for evaluating the effectiveness of sampling strategies and estimating the precision and accuracy of results. This section describes the general guidance and frequency for collecting field replicates and blanks. Specific details on the methods used can be found in chapter 4.

5.B. FIELD BLANKS

Field blanks are samples of distilled water that demonstrate the cleanliness of sample bottles, preservatives, equipment, and the sample handling procedures. Deionized water is taken in the field and poured into sample bottles at the same time as when field samples are being preserved. When preparing the field blank any preservative added to investigative samples should also be added to blank samples. When a sample is collected with sampling equipment such as automatic samplers the blank should be an equipment rinsate. For soil or sediment samples the blank should be a sample collected from a background location or a site that is known to be uncontaminated.

Ideally, each batch of samples collected should have a set of appropriate field blanks. However, this is not always practical for sampling programs that typically collect only a few samples at a time. For these programs the number of blanks collected should be sufficient to allow an annual assessment of whether contamination of samples is occurring from equipment washing or sample handling procedures. For all samples except volatile organic samples, as an absolute minimum, one blanks should be collected for every 20 investigative samples. However for some programs a frequency of one blank for every 10 investigative samples may be more appropriate.

For volatile organic samples, trip blanks will be collected at a frequency of one (consists of 2 vials) per cooler that contains volatile organic samples. This is to check for possible cross contamination during shipping and storage. Trip blanks will be prepared in the laboratory by filling sample bottles with organic-free reagent water, capped and shipped along with empty sample bottles to the field. The trip blanks will remain unopened throughout the whole sampling trip, and will be shipped along with filled sample bottles back to the lab for analysis. Field blanks are applicable to most of the parameters analyzed from the following sample containers:

Sample		
Container	Code	Parameter Groups
GA		Gen. Chem./Acidic (COD, TOC, phenolics, ammonia, tot.
		phos., Kjel. N.)
GB		Gen. Chem. Basic (Cyanides)
S		Sulfide
GN		Gen. Chem./Neutral (nitrate, nitrite, orthophosphate,
_		BOD, turbidity, residues and silicates)
MA		Metals, Total Acidic
GG		General Chem Phenolics
GP		General Chem Phenolics- Dechlorinated
MD		Metals, Dissolved (Lab Filtration and Preservation)
MAD		Metals, Acidic Dissolved (Field Filtration and
		Preservation) +6
MN		Minerals/Neutral (pH, cond., Cr , anions, cations)
MB		Minerals/Brine (anions and cations)
PO-1		Purgeable Organics, Scan 1
PO-2		Purgeable Organics, Scan 2
POV		Purgeable Organics, soil
ON		Org./Neutral Extract, Scan 3
OB		Org./Basic Ext. GC/MS, Scan 9
OA		Organics, Acid Extractables (Scan 8)
OG		Oil and Grease
CA		Chlorophyll
MX		Metals EP Toxicity
OX		TCLP Volatiles

A field blank should contain all the same preservatives as the investigative samples. However, dechlorinating agents are used to remove chlorine from some water samples. Because field blanks will not contain chlorine, dechlorinating agents should not be added to field blanks.

Sample containers used for field blanks should come from the same supply as used for routine samples. The field blank should be entered on the sample sheet(s) in the same manner as other samples and identified as a "field blank". The time of chemical addition should be entered as the time of collection on the sample sheet(s). Field blanks should be stored and transported in the same manner as the other containers of the same container code.

Field blanks are not applicable to non-water matrices and some water samples. These include:

Sample <u>Container</u>	Code	Parameter Groups
DO		Dissolved Oxygen
GS		Gen. Chem., SedSoils (Tot-P, Kjel-N., CN, COD, Phenolics)
MS		Metals, Soil, Sediment, Sludge
м		Metals, Tissue
MO		Metals/Oil, Liquid and Solid Waste
OS		Organics, Sediment, Sludge, Soil
OL		Organics, Liquid Waste
OT		Organics, Tissue
BC		Bacteria
POV		Purgable volatiles/soil

5.C. FIELD REPLICATES

Field replicates are two independent samples taken from one sampling point. Depending on the sampling program and proposed analyses, these may be side-by-side grab samples, composite samplers mounted in parallel, or samples collected with two different but equally acceptable methods. The main purposes of field replicates are to measure the total variability of samples including analytical variability and field variability. The laboratory quality control data generated from duplicate analyses of a single sample measures analytical variability only. Analytical variability may be only a small portion of the total variability. Field staff must evaluate their own replicate results and investigate possible sources of error.

The frequency of field replicates should be the same as field blanks, or as a minimum about one field replicate for every twenty samples. A higher frequency may be necessary for special projects or to help resolve special sampling problems. Each sampling program will keep track of all blank and replicate results and annually summarize results. The laboratory does not summarize or analyze field replicate or blank results other than to possibly flag obvious discrepancies.

Overall, goals for field blanks and field replicates are for no deterioration over time. For each program beginning with the 1994 calendar year the annual average, median and 10% exceedance value for replicate range and blank results will be calculated from the field blank and replicate data.

5.D. BLIND DUPLICATES

Blind duplicates are generally splits of a single field sample which has been submitted without notifying laboratory personnel. Their major purpose is as an external audit on the quality of laboratory data. If an adequate quality assurance program is in operation, the additional information provided by blind duplicates is very limited. They are of no value if routine formal evaluation of the results is not practiced. The laboratory should be promptly notified of <u>all</u> blind duplicate results after the person submitting them has received the results. The results may indicate a problem, but it will generally be too late to investigate and correct the cause of the problem if prompt notification of the lab is not done. Blind duplicates should be submitted only to address special problems or needs. Therefore there is no recommended frequency for these samples.

5.E. SPLIT SAMPLES

If samples are to be analyzed by two or more different laboratories as a check of analytical performance, it is imperative that each laboratory is analyzing the same sample to start with. A representative sub-sample of a homogenized sample should be submitted to each lab. It should be absolutely clear to all participants which parameters are to be analyzed and how they are to be reported.

If there are only two participants, the results should be checked for overlapping confidence intervals based on the expected precision of the two laboratories. It may be difficult to locate the source of any significant differences identified. Some of the most frequent causes of error are:

- -- the use of an improper method,
- -- the improper use of a correct method,
- -- an inadequate quality control program,
- -- poorly trained laboratory staff, or
- -- insufficient attention to preservation techniques and holding times.

When more than two laboratories perform split sample analyses, it may be easier to identify poorly performing laboratories. Quite often, individual performance is measured against a median or mean value. Outliers should not be excluded from final statistical evaluation for establishing adequate performance. It is possible that a significant number of participants may erroneously influence the results through common use of biased methods. Different studies may require different methods of evaluation depending on individual circumstance.

5.F. FIELD SPIKES

Field spikes of standards into actual samples or laboratory supplied deionized-distilled water are of interest only for special studies. They are not practical for routine use because of the special precautions which must be taken. Field spikes are particularly valuable for determining the suitability of sample containers for certain constituents, the accuracy of methods, and the applicability of holding times. The components of a study using field spikes should be evaluated on an individual basis.

5.G. LAB MATRIX SPIKES

A matrix spike is a sample which has had added to it a volume of a standard of known concentration. It is used to measure the recovery of a process or as a measure of precision when analyzed in duplicate. The use of a sample matrix is intended to include matrix interference effects in the precision or accuracy measurement.

The MDNR Environmental Lab analyzes organic sample matrix spikes at a frequency of 1/20. The field staff should periodically submit twice as much sample (for organic parameters) to the lab to provide the necessary volume of sample for the matrix spikes.

CHAPTER 6

CHAIN OF CUSTODY PROCEDURE

Prepared by State of Michigan Department of Natural Resources September, 1994

TABLE OF CONTENTS

6.A.	INTRODUCTION	•	1
6.B.	DEFINITIONS	•	1
6.C.	GENERAL CONSIDERATION	•	1
6.D.	SAMPLE COLLECTION, HANDLING AND IDENTIFICATION	•	2
6.E.	TRANSFER OF CUSTODY AND SHIPMENT	•	3
6.F.	LABORATORY CUSTODY PROCEDURES	• •	4
6.G.	GAINING LEGAL ACCESS	•	4

APPENDICES

6.F1	Sample	Handl	ing P	rocedur	es	f	or	Cri	.min	al	Co	urt	: C	as	es	•	•	6-5
6.G1	Consent	to Se	earch	Form	•	•	•••	•	• •	•	•			•	•	•	•	6-6

CHAPTER 6 - CHAIN OF CUSTODY PROCEDURES

6.A. INTRODUCTION

Physical evidence in the form of samples, pictures or other documents, may be used to support litigation. Thus, the regulatory agency must be able to provide the chain of possession and custody of any samples which are offered for evidence or which form the basis of analytical test results introduced into evidence. Therefore, it is imperative that these written procedures be available and followed whenever samples are collected, transferred, stored, analyzed, or destroyed. The primary objective of these procedures is to create an accurate written record which can be used to trace the possession and handling of the sample from the moment of its collection through analysis and its introduction as evidence in a court of law.

6.B. DEFINITIONS

-- Custody: A sample is in someone's custody if

- It is in one's actual physical possession; or
- It is in one's view, after being in one's physical possession; or
- 3. It is in one's physical possession and then locked up so that no one can tamper with it; or
- 4. It is kept in a secured area, restricted to authorized personnel only.

-- Preservation Blank: A sample container filled with distilled and deionized water and preserved with the same chemical preservative and in the same manner as samples collected in the field.

-- Laboratory Sample Custodian: A laboratory employee who has been designated to take charge of receiving, storing, and destroying samples.

-- Sample Storage Area: A room or area which is either locked or access is limited to authorized personnel only.

-- Laboratory Report Sheet: The laboratory report sheet will record sample location, collection time and date, sampler's signature or written initials, custody transfers, laboratory log number, date and time received at the laboratory, examiner's name, and is the means of conveying laboratory results.

-- Sample Preservation: Treatment after collection of the sample to stabilize one or more constituents of the sample prior to transport to the laboratory for analysis. Procedures used to preserve samples include refrigeration, pH adjustment, and chemical treatment.

-- Sample Identification: A tag or label affixed to the sample container used to identify the sample.

6.C. GENERAL CONSIDERATION

-- Confirm the legality of the search; (i.e., consent search, search warrant, etc. to be obtained). Trespassing for the purpose of evidence collection is an illegal act.

- -- The number of people handling the samples should be minimized.
- -- All records must be legible and filled out in waterproof ink.

3/94

Changes are made by crossing out, not erasing, and initialing.

- -- Each sample must be accurately and completely identified.
- -- Proper sampling methods must be utilized. It is necessary to use proper sample collection techniques, tools, bottles, preservatives, and observe holding times so the samples can be used for evidence at a later date. Not following any one of the necessary chemical, biological or legal rules could result in the evidence not being admitted into the court. It is also very important to be sure that cross-contamination between samples does not occur.

6.D. SAMPLE COLLECTION, HANDLING AND IDENTIFICATION

- -- Procedures for sample collection and preservation as addressed in previous sections are to be followed.
- -- Each sample is to be identified by affixing a pressure sensitive gummed label or standard tag on the container(s) (See Chapter 3, Section 3.C.1, Sample Bottle Labeling and Codes). Using a waterproof pen, the label should contain, as a minimum, the sample field identification number corresponding to the laboratory sheet, source name; e.g., corporation, river, landfill (initials may be used), and location of sampling (process or cooling water discharge) or STORET number, date sample was collected, bottle code (analysis required), and preservative techniques used. Where a label is not available, the same information should be affixed to the sample container with indelible, water proof marking pen. The samples collected for pending or existing litigation may require additional labeling requirements. Law Enforcement Division should be contacted in these instances.

-- All samples collected shall, if possible, be maintained in custody during transportation to the laboratory with the laboratory reporting sheet.

-- Whenever the samples cannot be maintained in one's physical possession, whether in the field or laboratory, the samples must either be locked inside the vehicles, or placed in a secured sample storage area.

-- Field records should be completed at the time the sample is collected and should be signed or initialed, including the date and time, by the sample collector. Field records should contain the following:

- -- Sample or log number
- -- Date and time
- -- Source of sample (including name, location, and sample type)
- -- Preservative used
- -- Analyses required
- -- Name of collector
- -- Pertinent field data (Temp., pH, Do, Cl, residual, etc.)
- -- Serial numbers on seals and transportation cases
- -- Sampling equipment used
- -- Maps or drawings of each sampling site

This information should be organized in a bound field notebook with numbered pages, and should contain any other pertinent information necessary to freshen the sampler's memory if asked to testify regarding collection of the sample. For example, if the sample is from a 55gallon drum, the name on the drum, and any other information obtained from the drum label should be recorded. Each drum must be positively identified by spraying or etching a number on the drum exterior and recording that same number on the sample label and sample log. Or if from a private well or monitoring well, the duration the well was purged should be recorded. When a specific investigation may involve an extended period of time requiring several contacts, a separate notebook should be maintained to record the field notes, follow-up and clean-up program.

- -- Color slides or photographs taken of the sample location and of any visible pollution are recommended to facilitate identification and later recollection by the inspector. A photograph log should be made at the time the photo is taken. After the photo or slide has been developed, record the signature of the photographer, time, date, site location, and view on the photo or slide. Do not describe what the photo illustrates on the back of the photo. Photographs and written records which may be used as evidence should be handled in such a way that chain of custody can be established.
- -- A blank should be provided for each type of sample collected as a check on preservative or container contamination. At least one of every 20 samples should have a blank (see Chapter 5 and specific sampling procedures in Chapter 4 for more details on how to collect banks). Additional blanks may be used at the discretion of the sampler. If a preservative has not been used recently, or if samples are collected for pending or existing litigation, additional preservative blanks should be prepared.

6.E. TRANSFER OF CUSTODY AND SHIPMENT

-- When transferring possession of the samples to the laboratory, the laboratory sample custodian will sign the laboratory report sheet and assume custody. The time, date, and custodian's signature must appear on the laboratory report sheet.

-- When an intermediate is required to deliver samples to the laboratory, they must assume custody of the sample. The identification tag, if required, and laboratory report sheet must contain their signature, date and time of day they assumed custody of the samples.

<u>Note</u> In transferring custody of the samples to either the laboratory or intermediate custodian in the field, an account of the number of samples should be made. If samples are transferred using a sealed storage chest, only the number of chests transferred should be noted.

-- When an intermediate custodian transfers samples to the laboratory, the laboratory sample custodian must follow the same procedure as indicated in the first item above.

Transfer of custody samples by commercial carrier is to be discouraged. Whenever possible, department vehicles or planes should be used. If transfer by commercial carrier is necessary, Department of Transportation (D.O.T.) regulation on labeling, packaging and shipment must be followed, particularly, where hazardous wastes may have been sampled, (see Chapter 3). Note, the individual who ships samples contrary to D.O.T. regulations as well as the department may be held liable for violations.

If packages are sent by common carrier, receipts must be retained

9/94

as part of the permanent chain of custody documentation.

6.F. LABORATORY CUSTODY PROCEDURES

-- A specific person shall be designated laboratory sample custodian and an alternate(s) designated to act as custodian in their absence. All incoming samples shall be received by the custodian who shall indicate receipt by signing the accompanying laboratory report form.

- -- After arrival at the laboratory, each batch is assigned a unique log number and each sample in the batch is assigned a unique number by the lab computer. This number will also be entered into a permanent laboratory log book. The Sample Receiving staff will also record the person delivering the sample, the person receiving the sample, date and time received, source of same, and the condition the sample was received in.
- -- The Sample Receiving staff will then alert the appropriate analyst of samples ready for analyses.
- -- A clean, dry dark isolated room, building and/or refrigerated space that can be securely locked from the outside shall be designated as the "Sample Storage Area."
- -- The custodian shall ensure that heat-sensitive, light-sensitive samples, radioactive, or other sample materials having unusual physical characteristics, or requiring special handling, are properly stored and maintained prior to analysis.
- -- The laboratory area shall be maintained as a secured area, restricted to authorized personnel only.

-- Once the sample analyses are completed, the unused portion of the sample together with all identifying labels, are returned to the sample storage area. The returned samples are retained until permission to destroy the sample is received by the custodian or the recommended maximum holding time has elapsed.

-- Samples to be retained for physical evidence should be so designated and should not be destroyed. Only upon the order of the Laboratory Director, and in consultation with a previously designated enforcement officials shall the sample be destroyed.

-- See Appendix 6.F.-1 for sample handling procedure for criminal court cases.

6.G. GAINING LEGAL ACCESS

When collecting samples on private property, it is important to have legal access to the site. There are four ways to gain legal access:

- -- Voluntary verbal access
- -- Voluntary written access (consent to search form)
- -- Administrative Inspection warrant
- -- Full criminal search warrant

Voluntary written access permission is best if you expect to go to court. However, overstating the importance of this to the person who is to sign could make him reluctant to allow access. Quite often search

9/94

warrants are the first thing attacked in pre-trial motions, and may represent the weakest link in the case. Trespassing or committing other illegal acts to gain evidence is inadmissible in court. Appendix 6.G.-1 is an example of a consent to search form.

CONSENT TO SEARCH*

	(Date)
	(Location)
Ι,	, an authorized
representative of	
having been informed of my co	institutional right not to have a search
made of the premises and/or s	ites hereinafter mentioned without a
search warrant and of my righ	it to refuse to consent to such a search,
hereby authorize	
n)	ame of agencies & officers/agents)
to conduct a complete search	of the premises and/or sites located at
These (officers or agents) as	re authorized by me to take from the above
inese (officers of agencs) ar	e authorized by me to take from the above
mentioned premises or sites _	······································
This is a voluntary, wri	itten permission given by me, an authorize
representative of	to the above named
person and/or agencies.	
Witnesses:	Signed:
	Date:

* From State of Michigan, Department of Natural Resources, Environmental Enforcement Division.

.

6-6

· · ·

NO.: PD-18

ENVIRONMENTAL LABORATORY

PROCEDURE

DATE REV. 10-22-92

SUBJECT: Chain of Custody Procedure for Criminal Court Cases

FORMS: Transmittal of Evidence and Laboratory Analysis (R9103, Rev. 6/81) Request for Priority I Laboratory Analysis (R4422, Rev. 8/85)

EFFECTIVE DATE: October 22, 1992

- 1. Definition
 - 1.1 A sample is in custody if it is:

1.1.1 In actual physical possession, or

1.1.2 In view after being in physical possession, or

1.1.3 Locked in a cabinet after being in physical possession.

- 1.2 Sample custody must be maintained from sampling to disposal.
- 1.3 Only samples meeting the following criteria will be kept in criminal case custody (all others will be kept in routine custody as outlined in Quality Assurance for Water and Sediment Sampling; Pub. 3730-0028):
 - 1.3.1 MUST have an environmental investigation section case number provided by the ECO in charge of the case, and
 - 1.3.2 The case MUST be (or have the potential of becoming) a criminal (not civil) case, and
 - 1.3.3 Each bottle MUST be identified with a green enforcement label (proper container and preservation technique must be used), and
 - 1.3.4 A sample custody form MUST accompany the samples.
- 2. Sample Receiving
 - 2.1 Criminal case samples will be treated as ordinary samples with respect to scheduling, lab capacity and priority assignment. Assignment of higher priority will require completion and proper approval of Form R4422 (Request for Priority I Laboratory Analysis) for each batch of samples submitted.
 - 2.2 The sample receiving employee will contact the supervisor or his/her designee of the Laboratory unit which has the most analyses to perform on the sample or which has previously worked on this particular case. The supervisor or a lead worker from that unit will be designated as the case coordinator (see 3.2). He/she will be responsible for sample receipt and maintenance of custody.
 - 2.3 The case coordinator must initial and date:

- 2.3.1 The Laboratory log book.
- 2.3.2 The analysis request sheet.
- 2.3.3 The sample custody form.
- 2.4 Sample receiving personnel must:
 - 2.4.1 Count and check the sample bottles for correspondence to the analysis request sheet.
 - 2.4.2 Fill in information in the sample log book (except for the signature of the person receiving the samples). Put "E" in the column to the right of the order number.
 - 2.4.3 Fill in the information at the top of the analysis request sheet (except for the signature of the person receiving the samples). Stamp "ENFORCEMENT" on the top of the sheet and write "E" to the right of the order number.
 - 2.4.4 After the case coordinator signs the analysis request sheet, make a copy for each Laboratory unit, one for the sample submitter and one for the SAM operator.
 - 2.4.5 Write the Lab order number(s) on the custody form and give the canary copy (second page) to the submitter.
 - 2.4.6 Give the remainder of the custody form to the person in charge of custody records.
 - 2.4.7 Attach the bottle labels after the computer prints them out.
 - 2.4.8 Lock the samples in one of the custody lockers.
- 2.5 Computer Log-In
 - 2.5.1 The sample information is logged into the Laboratory computer (SAM) the same as other samples, except that the case number is also entered.

3. Sample Storage

- 3.1 After proper receipt, the sample receiver will lock the samples in one of the custody lockers.
- 3.2 If the case coordinator is not present, the sample submitter will lock the samples in the limited access file cabinet. When the case coordinator returns, the sample receiving employee will have him/her sign the necessary papers, complete the sample receiving procedure (see #2) and transfer the samples to the custody lockers.
- 3.3 The sample lockers will be arranged sequentially with a separate section assigned to each order number.

- 3.4 Keys to the sample lockers are assigned to lab analysts and supervisors. Keys to the limited access file cabinet are assigned only to case coordinators.
- 4. Analysis
 - 4.1 Authorized analysts will unlock the custody locker, remove desired sample bottles, analyze and return the samples to the same section of the locker when finished.
 - 4.2 The sample must be "in custody" at all times when out of the locked active sample locker.
 - 4.3 If an analyst takes samples from one DNR Lab location to another DNR Lab location (e.g., from the main Lab to the Filley Street Lab) for preparation or analysis, he/she will sign the white copy of the custody form and enter the date and time. The same custody procedures will be followed at the Filley Street Lab as at the main Lab, including keeping samples in a locked cabinet when not in use. The analyst will return the samples to the main Lab to its assigned location in the custody locker.
- 5. Reporting
 - 5.1 The analytical results will be printed out after all data is entered into the Lab computer (SAM). One copy is sent to the appropriate agency and another is kept on file at the Laboratory.
 - 5.2 A separate file with all pertinent information will be maintained for each Lab order number which the samples have been designated as criminal custody.
- 6. Sample Release
 - 6.1 After completion of analyses, a form letter will be sent to the investigator stating that the samples are no longer needed by the Laboratory and that they should be picked up within one month.
 - 6.2 If no response to the letter is received within one month, LOCKED CUSTODY WILL NO LONGER BE GUARANTEED due to space limitations.
 - 6.3 The Lab has additional locked storage space at the Filley Street warehouse which enforcement staff may use if they wish to retain samples for a period of time after analyses have been completed. It is, however, their responsibility to pick the samples up from the Lab and take them to the warehouse.

Approved by: 10/22 Lab Director

CHAPTER 7 DATA HANDLING PROCEDURES

Prepared by State of Michigan Department of Natural Resources Bureau of Environmental Protection June 1985

COMMENTS ON CHAPTER 7

.

DATA HANDLING PROCEDURES

Quality Assurance for Water & Sediment Sampling

I have reviewed this chapter of the Quality Assurance Manual and recommend the following improvements:

(attach additional pages if necessary)
Submitted by (Name):
(Division):
(Unit):
(Unit):
(Telephone):
(Date):

Your comments will be considered in revising the manual to benefit all users. Please send this form to:

Quality Assurance Coordinator Environmental Services Division Department of Natural Resources P.O. Box 30028 Lansing, Michigan 48909

CONTENTS

•

7.A.	Intro	oduction	<u>Page</u> 7-1
7.B.	Gener	cal Data Collection and Handling Guidelines	7-1
	1.	Field Procedures	7-2
	2.	Laboratory and Office Procedures	7-2
	3.	General Information	7-4
7.C.	Rout	ine Analysis Request and Data Reporting Procedures	7-4
	1.	Analysis Request Sheets	7-4
	2.	Result Reporting	7-4
7.D.	STOR	ET Analysis Request and Data Reporting Procedures	7-4
	1.	Field and Laboratory Data Sheets	7-4
		a. Field Staff Entry of Information on Data Sheets	7-4
		b. Laboratory Entry of Information on Data Sheets	7-14
	2.	Monitoring Site Documentation and STORET Station Numbering	7-14
	3.	Data Handling Procedures for STORET Storage	7-17
		a. STORET Station Storage	7-17
		b. Data Storage in STORET	7-29
	4.	Retrieval and Analysis	7-42
		a. STORET Water Quality Data System	7-42
		b. STORET Retrieval Requests and Data Analysis	7-48
7.E.	Data	Analysis Guidelines	7-48
	1.	Field Data Review	7-51
	2.	Formulas - Conversion Factors	7-51
		a. Conversion Factors	7-51
		b. Formulas and Procedures for Data Analysis	7-51
		1. Heat Load Addition	7-51
		2. Percent Effluent via Specific Conductance - Estimated	7-52
		3. Geometric Means	7-52
		4. Mass Loadings	7-52
		5. Percent Unionized Ammonia	7-52
		6. Degree of Waste Dilution in Receiving Streams	7-52
	3.	Significant Figures	7-52
		a. Significant Digits	7-52
		b. Multiplying Significant Digits	7-53
		c. Flow Data	7-53
		d. Rounding-Off Numbers	7-53
7.F.	Reco	rd Maintenance and Quality Assurance	7-54

TABLES

7.D1	Field Parameter List	<u>Page</u> 7-10
7.D2	County Code List	7-15
7.D3	Minor River Basin Names and Codes	7-18
7.D4	Michigan Fish List by Family	7-32
7.D5	STORET Tissue Codes	7-37

FIGURES

7.C1	Routine Analysis Request Sheet (water)	7-3
7.C2	Organic Unit Data Report Format	7-5
7.C3	Inorganic Unit Data Report Format	7-6
7.D1	Blank Field and Laboratory Data Sheet	7-7
7.D2	Completed Data Sheet	7-19
7.D3	Stream Sheet Front	7-21
7.D4	Stream Sheet Back	7-23
7.D5	Inland Lake Sheet Front	7-25
7.D6	Inland Lake Sheet Back	7-26
7.D7	Great Lake Sheet Front	7-27
7.D8	Great Lake Sheet Back	7-28
7.D9	Data Storage Process	7-30
7.D10	Edited Data Sheet	7-39
7.D11	Coded Tissue Sheet	7-41
7.D12	Descriptive Paragraph Page	7-43
7.D13	Example of Ret Format	7-44
7.D14	Example of APARM Format	7-45
7.D15	Example of INV 120 Format	7-47
7.D16	Retrieval Request Form - Station Inventory	7-49
7.D17	Retrieval Request Form - Inhouse	7-50

APPENDICES

7.E.-1 Units of Measurement, Conversion Factors and Special Tables

.

CHAPTER 7 - DATA HANDLING PROCEDURES

7.A. INTRODUCTION

The purpose of this chapter is to outline recommended procedures for water quality data collection and handling. Routine and STORET procedures are described in Sections 7.C. and 7.D, respectively. Data quality can be affected during the field collection effort, laboratory procedures, computer system storage, data analysis and reporting. If quality assurance steps are not followed in these areas, the integrity of the data will be lost, and the data will misrepresent the situation being monitored. In the process of collection and handling, data can be incompletely described, incorrectly transcribed, improperly transposed, or even lost if the proper procedures are not followed.

This chapter also provides data analysis guidelines which include the use of significant figures, conversion factors, statistical analysis guidelines, and record maintenance. Data handling within the laboratory is covered in Chapter 1 of this manual.

7.B. GENERAL DATA COLLECTION AND HANDLING GUIDELINES

7.B.1. FIELD PROCEDURES

A meaningful data handling procedure begins with proper and adequate preparation for the field collection effort. The following steps, as a minimum, should be performed: 1) sampling objectives established, 2) sample sites researched and selected, 3) selection of parameters to be sampled, 4) data recording documents prepared, and 5) staff briefed. If these steps are properly done, the field staff should be able to obtain all of the necessary information in the field with minimal effort and avoid errors in the process.

It is very important that the field staff record all of the field information when they are performing the work rather than relying upon their memories. Sampling sites as well as dates and times should be specifically and accurately recorded. All of this relevant information should be recorded on the data sheets in the spaces provided prior to delivery of samples to the lab. This information will then be available to the lab personnel for their use during analysis as well as to the user upon receipt of the completed analysis from the lab.

7.B.2. LABORATORY AND OFFICE PROCEDURES

Within the laboratory and data user's office, care should be exercised to avoid error in data handling. Persons responsible for data analysis and handling should be properly trained in the procedures required. Supervisors should check results by periodically verifying a percentage of the calculations by employees. When data is transposed from one record to another, all data should be proofed by someone other than the transposer. In general, accuracy should be continually stressed by supervisors at all levels of data handling. The laboratory data management system is in a transition period. As various components of this system are implemented, procedures and forms for submitting samples and reporting data will be revised. These changes will be implemented with as much advance notice and with as little confusion as possible.

There are presently two separate procedures for submitting samples to the laboratory. These are described as Routine procedures and STORET procedures in Sections 7.C and 7.D., respectively.

7.B.3. GENERAL INFORMATION

Data sheets are used to record field information and to request analyses from the laboratory. Two sets or types of data sheets may be used. Analysis Request Sheets are for routine use by a variety of programs. STORET sheets are for programs which store their data in the STORET system.

Analysis Requst Sheets are available from the laboratory in the receiving room. STORET sheets are available from the STORET Coordinator, Water Quality Surveillance Section. The data sheets are subject to change as lab methods and organization change. Each data sheet has a date and number in the lower right hand corner and a title in the upper right hand corner. The most recent data sheet revision should always be used.

Users should fill out as much information on the data sheets as possible before going into the field. This will minimize errors on the sheets and is essential to good field work organization.

7.C. ROUTINE ANALYSIS REQUEST AND DATA REPORTING PROCEDURES

7.C.1. ANALYSIS REQUEST SHEETS

An Analysis Request Sheet for water samples is shown as Figure 7.C.-1 Use of this form eliminates the need for completing a separate analysis request sheet for each laboratory unit. As with STORET Analysis Request Sheets, a separate sheet is required for each matrix. A listing of the most current analysis request sheets is provided below:

<u>No.</u>	Matrix	Color	Revision Date
EL068	Sediment/Soil	goldenrod	11/84
EL069	Tissue	pink	5/84
EL070	Water	white	11/84
EL085	Organics/Oil ASTM Leachate/E.P. Tox.	canary blue	5/85 6/85

EL070	Figure 7.C-1.	outine Analysis Request Sheet	
11/84 Matrix = Water	NICHIGAN DEPT OF	NATURAL RESOURCES ENVIRONMENTAL LABORA ANALYSIS REQUEST SHEET	TORY YES / NO - INFO ON BACK ***** SAFETY WARNING *****
LAB PROJ LOG# CODE	COST	RECEIVED DAT	E AM F/ PM
		COLLECTED	TRANSFERED
SAMPLE REMARKS	*****	SEND RESULTS TO	
	IN OR DESCRIPTION		
! !011			1 I I I
! !02!			
! !03!		· !	i
! !04!		·!	
 ! !05!		!	
! !06!		!	
! !07!		!	
! !08!	****************	!	·····
! !09!		!	!!!!
! !10!			! ! !
 ORGANIC	12222222222222222222222222222222222222	**************************************	GENERAL CHEMISTRY
PO1 #1 HALOCARBONS1 2	3 4 5 6 7 8 9 10 MAL	FIELD FILTER+PRES1 2 3 4 5 6 7 8 9 10	DO DISS OXYGEN1 2 3 4 5 6 7 8 9 10
P02 #2 AROMATIC HC1 2 OBN #3 CL HC&PEST &PCB.1 2 #6 PHTHALATES1 2 #7 PNA1 2 1 2 1 2	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	LAB FILIKATIUN1 2 3 4 5 6 7 8 9 10 A CA MG NA K1 2 3 4 5 6 7 8 9 10 CD CR CU NI PB ZN1 2 3 4 5 6 7 8 9 10 H6 MERCURY1 2 3 4 5 6 7 8 9 10 AS ARSENIC1 2 3 4 5 6 7 8 9 10 SE SELENIUM1 2 3 4 5 6 7 8 9 10 SB ANTIMONY1 2 3 4 5 6 7 8 9 10	GN 0-PHOS NO21 2 3 4 5 6 7 8 9 10 RES-NF (SS)1 2 3 4 5 6 7 8 9 10 RES-TF (TDS)1 2 3 4 5 6 7 8 9 10 1 2 3 4 5 6 7 8 9 10 BOD TOT 5 DAY.1 2 3 4 5 6 7 8 9 10 BOD CARB 5 DAY.1 2 3 4 5 6 7 8 9 10 1 2 3 4 5 6 7 8 9 10
1 2 OA #8 PHENOLS1 2 OB OIL & GREASE-IR1 2 OIL & GREASE-GRAV1 2	3 4 5 6 7 8 9 10 3 4 5 6 7 8 9 10	DTHERS (CIRCLE) AG AL BA BE CO FE1 2 3 4 5 6 7 8 9 10 LI MN MO TI V1 2 3 4 5 6 7 8 9 10 FURNACE- CD1 2 3 4 5 6 7 8 9 10 • CR CU NI PB1 2 3 4 5 6 7 8 9 10 • PH, CONDUCTANCE1 2 3 4 5 6 7 8 9 10	SA COD1 2 3 4 5 6 7 8 9 10 TOC1 2 3 4 5 6 7 8 9 10 NO3+NO2 NH31 2 3 4 5 6 7 8 9 10 KJEL N, TOT P1 2 3 4 5 6 7 8 9 10 PHENOLICS1 2 3 4 5 6 7 8 9 10 1 2 3 4 5 6 7 8 9 10
1 2 1 2 	2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10 2 3 4 5 6 7 8 9 10	CL SO4 TOTAL ALK1 2 3 4 5 6 7 8 9 10 FLUORIDE1 2 3 4 5 6 7 8 9 10 CR+61 2 3 4 5 6 7 8 9 10 HC03- CO3=1 2 3 4 5 6 7 8 9 10 CA M6 NA K -DISS. 1 2 3 4 5 6 7 8 9 10 B BRINE (CIRCLE) * CL SO4 BR 1 2 3 4 5 6 7 8 9 10 * ALK HC03- CO3=1 2 3 4 5 6 7 8 9 10 * ALK HC03- CO3=1 2 3 4 5 6 7 8 9 10 * CA M6 NA K1 2 3 4 5 6 7 8 9 10	GB TOTAL CN1 2 3 4 5 6 7 8 9 14 FREE CN1 2 3 4 5 6 7 8 9 14 BC FECAL COLI1 2 3 4 5 6 7 8 9 14 TOTAL CDLI1 2 3 4 5 6 7 8 9 14 1 2 3 4 5 6 7 8 9 14 CA CHLOROPHYLL1 2 3 4 5 6 7 8 9 1 1 2 3 4 5 6 7 8 9 1 1 2 3 4 5 6 7 8 9 1 1 2 3 4 5 6 7 8 9 1 1 2 3 4 5 6 7 8 9 1

.

Field staff are responsible for providing the appropriate Project Code, Cost Center Number, Location Sampled, Collected By, Transferred To, Sample Remarks and Send Results To portions of the form. Field staff are also responsible for completing the Field I.D. or Description, Sample Information, and Date and Time portions for each sample. As per the number of empty spaces, the form can accommodate up to ten samples. The analyses requested for each sample are indicated by circling the sample number following that sample. For example, if sulfide analysis was requested on sample 4, the numeral 4 following sulfide would be circled. However, if all samples were to be analyzed for sulfide, then the letter "A" signifying all samples should be circled.

The Laboratory is responsible for assigning the laboratory log number and the sample number to the analysis request sheet. The laboratory also completes the Received At Lab, the Date/Time, and the sample priority assignment (PR) according to procedures described in Chapter 2.

7.C.2. RESULT REPORTING

Each Laboratory Unit has a different result report format due to differences in data management hardware. Organic and Inorganic Unit results are reported to laboratory users by copy of a computer printout. The General Chemistry Unit presently reports results on STORET forms as described in Section 7.D.l.a.

The Organic Unit report provides results of those organic compounds detected in each requested scan(s). Undetected compounds are not formally reported. However, the compounds looked for in each scan are reported with sample results by providing an up-to-date scan list with each set of results. An example of Organic Unit reporting is provided in Figure 7.C.2.

The Inorganic Unit provides results for each requested metal as per the example in Figure 7.C.-3.

7.D. STORET ANALYSIS REQUEST AND DATA REPORTING PROCEDURES

7.D.1 FIELD AND LABORATORY DATA SHEETS

7.D.1.a. FIELD STAFF ENTRY OF INFORMATION ON DATA SHEETS

STORET Analysis Request Sheets are maintained by the Water Quality Surveillance Section, Surface Water Quality Division. Users requiring sheets different from those currently available should contact this Section.

Specific information to be entered on the data sheets by the field staff is indicated by letter code in Figure 7.D.-l and is as follows:

B) Project Code: Each project is assigned a code, which is used to keep track of work being done for each project within the lab. This code must be completed by field staff.

MICH DNR ENVIRONMENTAL LAB ORGANIC RESULTS FOR LAB LOG #4227

LAB#	39259	us/L	SCAN 1	СС)MI	IEŀ	ITS
		1.00	Detection limit this run				
LAB#	39259	ue/L	SCAN 2	CC	1111	ΙEŀ	ITS
		420.00 510.00 400.00	BENZENE Toluene Xylenes	* * *	* * *	* * *	* *
		5.00	Detection limit this run				

LA6#	39260	ue/L	SCAN 1	COMMENTS
		1.00	Detection limit this run	
LAB#	39260 .	ua/L	SCAN 2	COMHENTS
		270.00 250.00 330.00	BENZENE Toluene Xylenes	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$

5.00 Detection limit this run

Unless noted above under COMMENTS, analyses were performed for the compounds on attatched scan list. Concentrations are rounded to 2 significant figures.

Approved _____

.

				INORG	ANIC UNIT	EXAMINER	1 Volou	
			LOG	ND: 4159			- 	
06 #	LAB # =====	ELEMENT	RES	ULTS	UNITS	ANALYSIS	MATRIX	RUN #
159	38 9 06	ALUMINUM	K	1000.0	UG / L	TOTAL	WATER	00358
159	38906	ARSENIC		1.8	UG / L	TOTAL	WATER	102
159	38 9 06	BARIUN	K	1000.0	UG / L	TOTAL	WATER	00358
159	38906	CADMIUM	K	20.0	UG / L	TOTAL	WATER	0035 8
159	38906	COBALT	K	50.0	UG / L	TOTAL	WATER	00358
159	3 89 06	CHROMIUM	K	50.0	UG / L	TOTAL	WATER	00358
159	38906	COPPER		85.0	UG / L	TOTAL	WATER	00358
159	38906	IRON		1340.0	UG / L	TOTAL	WATER	00358
159	38 9 05	MERCURY	K	0.5	UG / L	TOTAL	WATER	103
159	38906	NICKEL	K	50.0	UG / L	TOTAL	WATER	00358
159	38906	LEAD	к	50.0	UG / L	TOTAL	WATER	00358
1159	38906	SELENIUM	K	0.5	UG / L	TOTAL	WATER	101
159	38906	ZINC		85.0	UG / L	TOTAL	WATER	00358
¥159	38906F	CADMIUM		0.4	UG / L	TOTAL	WATER	199
159 159 159 159	38908 38908 38908 38908 38908	HEX CHROMIUM CADMIUM COBALT CHROMIUM	К К К	2.0 20.0 50.0 50.0	UG / L UG / L UG / L UG / L	TOTAL TOTAL TOTAL TOTAL	WATER Water Water Water	18 00358 00358 00358
4159	38 9 08	COPPER		6 0. 0	UG / L	TOTAL	WATER	00358
4159	38908	IRON		1600.0	UG / L	TOTAL	WATER	0035B
4159	38908	NICKEL	К	50.0	UG / L	TOTAL	WATER	0 0 35 8
4159	38908	LEAD		50.0	UG / L	TOTAL	WATER	00358
4159	3 8 90 8	ZINC		95.0	UG / L	TOTAL	WATER	00358
3	8908							
4159	38909	ALUMINUM	K	1000.0	UG / L	TOTAL	WATER	003 58
4159	38909	BARIUM	K	1000.0	UG / L	TOTAL	WATER	00358
4159	38909	HEX CHROMIUM	K	2.0	UG / L	TOTAL	WATER	18
4159	38909	CADMIUN	К	20.0	UG / L	TOTAL	WATER	00358
4159	3 8 90 9	COBALT	K	50.0	UG / L	TOTAL	WATER	00358
4159	38909	CHRONIUM	K	50.0	UG / L	TOTAL	NATER	00358
4159	38909	COPPER	K	20.0	UG / L	TOTAL	WATER	00358
4159	38909	IRON		340.0	UG / L	IOTAL	WATER	00358
4159	38909	NICKEL	K	50.0	UG / L	IOTAL	NATER	00358
4159	38909	LEAD	K	50.0	06 / L	IUTAL	AATER	00358
	10000	/ ENC	K	50.0	UG / Ł	TUTAL	WALER	90.358

FIGURE 7.D.-1 Field and Laboratory Data Sheet

I MIC	HIGAN D	EPT. OF	NATURA	AL RESOL	IRCES,	ENVIRO	NMENT	AL LA	BORATO	RY ANA	ALYSI	<u>s</u>	ENV.	QUALITY	-WATER -	POINT	SOURCE	STUDIES	SEC.	
LAB	A	PROJ	0	OST ENTER -	©	PR 🔘	BY	ECTED	E			TRAN	SFERED	Ē	A1	CEIVED	<u> </u>	EXAMIN	er 🕧	
LOC	- ATION PLÉD -	1						RE	MPLE MARKS	٩					SEND (NAME	RESULTS	TO 10N)	ĸ		
C		-"DO NO	T PUNCH	SAMPLIN	> 1G	REF IS	TORET	STAR DATE YYMM	T ITIM MIL DDITTT	EIS,T IOR TIB	NUM SAM- PLES			ME DEP- TH TT FEET	LAB NO.	TEMP. DEGREE	PH STAND.	CL2 T. RES	DIS. MG/L	IC.O.D. HIGH MG/L
	- <u>s</u>	ITE OR	SAMPLE			P -	<u></u>					<u> </u>		<u>></u>	00008	100010	00400	50060	00300	00340
	- <u>(</u>)						<u>()</u>			<u> </u>	<u> </u>	<u> </u>) <u>(</u>	<u> </u>						\rightarrow (f)
						C06														
	-					- C07														
	-					cos -				-										
						C09														
						610														
REF NO.	T.O.C. AMPUL MG/L 00630	PHENOL T. REC UG/L 32730	CN TOTAL MG/L 00720	CN FREE MG/L 00722	S= TOTAL MG/L 00745	BOD- TŪTA 5 MG/ 0031	5 IND3 L ITOT L MG7 0 1004	8N02 AL (L N I (30 0	NH3 TOTAL MG/L N D0610	KJEL TOTAL MG7L 00625	N OR TO N MG	TH.P TAL /L P 507	PHOS. TOTAL MG/L F 00665	CR6+ TOTAL UG/L 01032	CL TOTAL MG/L 00940	SO4= TOTAL MG/L 00945	ALK. TOTAL MG/L 00410			
01	1																			- (T)
02																				
03																				
04																				
05										•										
-06																				
07																				
09											-				-					
10				<u> </u>				!				!		1		:		1 1		
1																ESD-003	303 REV	. 05/80		0

٠

6/85

7-7

- C) Cost Center: This code is used by the laboratory to track time expended on each set of samples. This code must be completed by field staff to assure that lab staff time is charged against the proper grant.
- E) Collected By: The person(s) collecting the samples should print their name(s), and initial here.
- F) Transferred To: This is used in the chain of custody procedure to document the transferring of samples from the collector to another person who delivers the samples to the lab. The person accepting the samples should sign or initial here and indicate the date and time of the transfer.
- Location Sampled: This space is used by the collector to indicate the general location sampled, e.g. facility name, river study, or primary monitoring run number.
- J) Sample Remarks: This space can be used to alert laboratory personnel about anything unusual concerning the samples, or that the samples are routine, e.g. suspected contamination source, contaminant, sample hazardous to lab staff or routine monitoring.
- K) Send Results to (Name & Section): This is used to indicate where the lab should send the sample results upon completion.
- L) Field I.D.: This column can be used by the collector to indicate identification codes used on the sample bottles other than assigned STORET stations numbers. This space is normally used in the absence of, or in addition to a STORET station number for the sampling site.
- M) Description of Sampling Site or Sample: This space is used to accurately describe the specific sampling site or source. It should be completely detailed to avoid confusion as to the source of the sample. PERMANENT LANDMARKS SHOULD BE USED TO LOCATE SAMPLE SITES. Collectors should avoid references to information about landmarks which are subject to change, such as the color of a house, light poles, trees, etc. Use street names, addresses, distance upstream or downstream from specific landmarks, etc. to describe sampling sites. Attention to these details is important.
- N) STORET Number: Preassigned STORET numbers specific to the sample site are entered in this space. New STORET numbers are assigned through the WISER and STORET GROUPS. Please see Section 7.D.2. of this chapter on station location and numbers for the sources of STORET station numbers. NOTE: Either a STORET number or a field I.D. code is required for all samples.
- 0) Start Date: This space is used for the date of the sample for grab samples, or for the beginning date for composite samples.

- P) Start Time: This space is used for the time of sampling for grab samples, or for the beginning time for composite samples. The time is indicated by using the 24-hour clock system. Fill in leading zeros and <u>do not</u> include a colon (:). NOTE: Time is required for all samples in order to determine proper holding times, and is required for storage of data in STORET.
- Q) Composite Information (these fields do not appear on grab blanks).
 - a) Space, Time, or Both Field: This field is used to indicate whether the sample is a space composite only (code an S), a time composite only (code a T), or if it is both a time and space composite (code a B). Enter only one code letter as appropriate.
 - Number of grabs composited or continuous composite: Enter either the number of individual grabs composited, or C for continuous composite.
 - c) End Date: In this space the ending date of a composite sample is entered.
 - d) End Time: The ending time of a composite sample is entered here.

NOTE: Leave these four fields blank or cross them out if you collect a grab sample when using a composite type data sheet.

- R) Depth: This field is used when samples are taken at depth (in feet). Leave this field blank if the sample is collected at, or just below the surface. For vertically integrated samples the depth indicated should be the bottom of the column sampled. Indicate the vertical integration by following the depth with a V.
- T) Parameter Fields: These fields are used to describe the physical, chemical, or biological parameters analyzed. Some parameter values are determined in the field. A list of some of the field parameters is located in Table 7.D.-1. Field values should be recorded in the appropriate column and line by the field staff. Appropriate detection limits and remark codes should be used for field values. Remark codes are listed in Chapter 1, Appendix 1.C.-9., of this manual.

Parameters which are not pre-printed on the data sheet should be entered by the collector using the appropriate description, units, and STORET parameter number. Consult the appropriate lab unit or the STORET Group for the appropriate parameter number.

Parameters for which analysis is desired can be indicated as follows:

	TABLE 7.D1	Field Parameter List
REF NUM	DATA SHEET RMK HEADER CODE	PARAMETER DESCRIPTION
001	STREAM WIDTH FEET 00004	STREAM WIDTH (FEET)
002	VERLOC % TOT. DEPTH 00005	X-SEC. LOC., VERTICAL (PERCENT OF TOTAL DEPTH)
003	X-LOC FT FRM L.BANK 00009	X-SEC. LOC., (FT FROM LEFT BANK LOOKING DOWNSTREAM)
004	TEMP. DEGREE CENT. 00010	TEMPERATURE IN DEGREES CENTIGRADE
005	TEMP. AIR CENT. 00020	TEMPERATURE OF AIR IN DEGREES CENTIGRADE
006	WEIGHT IN POUNDS 00023	SAMPLE WEIGHT IN POUNDS
007	LENGTH IN INCHES 00024	SAMPLE LENGTH IN INCHES
008	BARMTR PRESS. MM HG 00025	BAROMETRIC PRESSURE (MM OF HG)
009	FIELD IDENT. NUMBER 00029	FIELD IDENTIFICATION NUMBER - GREAT LAKES GRID NUMBER .
010	CLOUD COVER PERCNT 00032	CLOUD COVER, FERCENT AT TIME OF SAMPLE
011	WIND SPEED MPH OOC35	WIND VELOCITY (MILES PER HOUR)
012	WIND DEGREE FROM N 00036	WIND DIRECTION IN DEGREES FROM TRUE N (CLOCKWISE)
013	ALTUDE FEET AB MSL 60042	ALTITUDE IN FEET ABOVE MEAN SEA LEVEL
014	PRECIP TOTDAY INCHES 00045	PRECIPITATION, TOTAL (INCHES PER DAY)
015	STREAM VLOCTY FTZSEC 00095	STREAM VELOCITY (FEET MER SECOND)

.

-
.

REF NUM	DATA SHEET RMK HEADER CODE	PARAMETER DESCRIPTION	
016	STREAM FLOW MD CFS 00060	FLOW, STREAM, MEAN DAILY (CUBIC FEET PER SECOND)	
017	STREAM FLOW INSCFS 00061	FLOW, STREAM, INSTANTANEOUS (CUBIC FEET PER SECOND)	
018	NO.SAM POINTS X-SEC 00063	SAMPLING POINTS, NUMBER OF IN A CRUSS SECTION	
019	STREAM DEPTH MEANFT 00064	DEPTH OF STREAM, MEAN (FT)	
020	SECCHI DISC INCHES 00077	SECCHI DISC OBSERVATION IN INCHES	
021	COND. FLD25C US/CM 00094	SPECIFIC CONDUCTANCE, FIELD (UMHOS/CM @ 250)	
022	INTSVE Survey ID.NO. 00114	INTENSIVE SURVEY IDENTIFICATION NUMBER YY26NN	
023	D.O. Probe Mg/L 00299	OXYGEN, DISSOLVED (ELECTRODE) (MG/L)	
024	D.O. SATUR PERCNT 00301	OXYGEN, DISSOLVED (PERCENT OF SATURATION)	
025	PH SU 00400	PH (STANDARD UNITS)	
026	STREAM FLOW SEVRTY 01351	FLOW SEVERITY: 1=DRY, 2=LOW, 3=NORMAL, 4=FLOOD, 5=ADOVE NORMAL	
027	CNDUIT FLOW MGD SOOSO	FLOW IN CONDUIT OR THRU A TREATMENT PLANT (MILLION GALLONS PER	DAY :
028	CL2 T. RES MG/L 50040	CHLORINE, TOTAL RESIDUAL (MG/L AS CL)	
029	LNDSRF DATUM FEET 72000	ELEVATION OF LAND SURFACE DATUM (FEET ADOVE MEAN SEA LEVEL)	
030	DEPTH HOLE FRET 72001	BEPTH, TOTAL OF HOLE (FT BELOW LAND SURFACE DATUM)	-11

6/85

	TABLE 7.D1	Continued
REF NUM	DATA SHEET RMK HEADER CODE	PARAMETER DESCRIPTION
031	DEPTH TOP WB ZONEFT 72002	DEPTH, TOTAL TO TOP OF WATER-BEARING ZONE SAMPLED (FEET)
032	DEPTH BOT WB ZONEFT 72003	DEPTH, TOTAL TO BOTTOM OF WATER-BEARING ZONE SAMPLED (FEET)
033	FLOW - BEFORE S MIN 72004	PUMP OR FLOW PERIOD PRIOR TO SAMPLING (MINUTES)
034	DEPTH WELL FEET 72003	DEPTH, TOTAL OF WELL (FEET BELOW LAND SURFACE DATUM)
035	DEPTH TOPSAM FEET 72015	DEPTH TO TOP OF SAMPLE INTERVAL (FEET BELOW LSD)
036	DEPTH BOTSAM FEET 72016	DEPTH TO BOTTOM OF SAMPLE INTERVAL (FT BELOW LSD)
037	DEPTH WATER FT LS 72019	DEPTH TO WATER LEVEL (FEET BELOW LAND SURFACE)
038	SPECIE F&W NUMBER 74990	FISH SPECIES NUMERIC CODE, F & W SERVICE (SEE SPECIES CODE LIST)
039	NUMERC TISSUE CODE* 74995	NUMERIC ANATOMY TISSUE CODE (SEE TISSUE CODE LIST)
040	GENINF PURP. CODE S4002	CODE, GENERAL INFORMATION, ALPHA-NUMERIC CODE (SEE PURPOSE CODE LIST)
041	SPECIE F&W Alpha 84005	FISH SPECIES CODE, ALPHA, F & W SERVICE (SEE SPECIES CODE LIST)
042	ALPHA TISSUE CODE* 84007	ALPHA ANATOMY-TISSUE CODE (SEE TISSUE CODE LIST)
043	FIELD IDCODE ALPNUM 84029	FIELD IDENTIFICATION ALPHA-NUMERIC CODE
044	CLOUD ANT ALPHA 84030	CLOUD ANOUNT ALPHA WEATHER CODE (SEE LIST)
045	PHYCAL WEACHIK CODE 84031	FHYSICAL WEATHER ALPHA WEATHER CODES (SEE LIST)

. **. .** . .

6/05

REF DATA SHEET RMK PARAMETER DESCRIPTION NUM HEADER CODE

046 STREAM STREAM CONDITION ALPHA WEATHER CODES (SEE LIST) CONDN ALPHA 84032

•

-

.

•

For specific parameter columns where all samples require analysis you can either a) leave the columns untouched which require analysis and cross out parameter headers for those not required, or b) place a check above the parameter header to be analyzed and leave the remaining columns untouched.

For specific parameter columns where only some of the samples require analysis, place a small check in the upper right hand corner of the cells. Leave all others blank. See Figure 7.D.-2 for an example of this coding.

The methods delineated above indicate to the lab the analysis required. Specific cells should not be crossed out so that the lab can record values if they obtain results for the parameter. In some testing procedures, results are obtained for several parameters at a time and thus, may be reportable even if not requested.

7.D.1.b. LABORATORY ENTRY OF INFORMATION ON DATA SHEETS

The following items on the data sheets are completed by the laboratory.

- A) Lab Log #: This number is assigned at the laboratory and is used to track the set of samples throughout the laboratory procedures.
- D) Priority (PR): The priority assigned to the set of samples is written here. Priorities are assigned according to the procedures outlined in Chapter 2, Laboratory Priority and Scheduling.
- G) Received at Lab: The person receiving the samples at the lab initials here.
- H) Examiner: The person at the lab reviewing and entering the results on the sheet initials this space. This is normally done by the lab unit chiefs.
- S) Lab Number: Unique numbers for each specific sample are assigned by the lab and are used to track the sample within the lab.
- T) Parameter Values: Most Organic and Inorganic Unit results are reported on a computerized data output. In these cases, it is the responsibility of the data user to transfer parameter values to the appropriate STORET data sheet. General Chemistry Unit laboratory results are reported on STORET sheets since data reporting by this unit is not yet computerized.

Figure 7.D.-2 is an example of a completed data sheet.

7.D.2. MONITORING SITE DOCUMENTATION AND STORET STATION NUMBERING

STORET station numbers are assigned by the DNR, Management Information Division, WISER group, and by the STORET group. The WISER group handles industrial and municipal NPDES permit monitoring site station number

MICH	EPA	COUNTY	MICH	EPA	COUNTY
CODE	CODE	NAME	CODE	CODE	NAME
01	001	ALCONA	46	091	LENAWEE
02	003	ALGER	47	093	LIVINGSTON
03	005	ALLEGAN	48	095	LUCE
04	007	ALPENA	49	097	MACKINAC
05	009	ANTRIM	50	099	MACOMB
06	011	ARENAC	51	101	MANISTEE
07	013	BARAGA	52	103	MARQUETTE
08	015	BARRY	53	105	MASON
09	017	BAY	54	107	MECOSTA
10	019	BENZIE	55	109	MENOMINEE
11	021	BERRIEN	56	111	MIDLAND
12	023	BRANCH	57	113	MISSAUKEE
13	025	CALHOUN	58	115	MONROE
14	027	CASS	59	117	MONTCALM
15	029	CHARLEVOIX	60	119	MONTMORENCY
16	031	CHEBOYGAN	61	121	MUSKEGON
17	033	CHIPPEWA	62	123	NEWAYGO
18	035	CLARE	63	125	OAKLAND
19	037	CLINTON	64	127	OCEANA
20	039	CRAWFORD	65	129	OGEMAW
21	041	DELTA	66	131	ONTONAGON
22	043	DICKINSON	67	133	OSCEOLA
23	045	EATON	68	135	OSCODA
24	047	EMMET	69	137	OTSEGO
25	049	GENESEE	70	139	OTTAWA
26	051	GLADWIN	71	141	PRESQUE ISLE
27	053	GOGEBIC	72	143	ROSCOMMON
28	055	GRAND TRAVERSE	73	145	SAGINAW
29	057	GRATIOT	74	147	ST CLAIR
30	059	HILLSDALE	75	149	ST JOSEPH
31	061	HOUGHTON	76	151	SANILAC
32	063	HURON	77	153	SCHOOLCRAFT
33	065	INGHAM	78	155	SHIAWASSEE
34	067	ICNIA	79	157	TUSCOLA
35	069	IOSCO	80	159	VAN BUREN
36 37 38 39 40	071 073 075 077 079	IRON ISABELLA JACKSON KALAMAZOO KALKASKA	81 82 83	161 163 165 994 996	WASHTENAW WAYNE WEXFORD LAKE ERIE LAKE SUPERIOR
41 42 43 44 45	081 083 085 087 089	KENT KEWEENAW LAKE LAPEER LEELANAU	84 85 86 00	997 998 999	LAKE HURON LAKE MICHIGAN OHIO INDIANA WISCONSIN CANADA

.

assignments and documentation. The STORET group oversees the ambient monitoring location station number assignments and documentation. Each STORET station number is composed of a two digit numeric county code followed by a four digit sequence number. The sequence number has no specific meaning as each station is assigned the next number in the sequence as needed. The station numbers are assigned to monitoring sites from a set of station set-up books located in the Management Information Division. The set-up books contain information pertaining to each station number, along with a brief description of each station.

All ambient monitoring sites, discharger outfalls, and intakes that have been assigned STORET station numbers are mapped on a set of U.S. Geological Survey quadrangle maps. These maps are located in the Water Quality Surveillance Section and maintained by the STORET group. The maps are used for many purposes. Most frequently they are used for locating monitoring sites and point source discharges for permit reviews and waste load allocations, and in planning field work.

Descriptions of all active industrial and municipal monitoring sites are maintained in the WISER computer system. This system is used to track the compliance status of monitoring sites, and can be used as a source for STORET station numbers which have been assigned to industrial and municipal monitoring sites.

Descriptions of ambient monitoring sites are maintained by the STORET group. The publication WATER QUALITY STATION INVENTORY lists all ambient monitoring sites for which data has been stored in STORET. This annual publication is the best source of ambient monitoring site station numbers. Some sites have been assigned station numbers which have not been established in STORET. The USGS maps maintained by Water Quality Surveillance Section are the only available source for these station numbers.

Users are cautioned when using STORET station numbers to carefully verify the number. In several areas of the state, streams may be crossed by a particular road several times in the same section, township, or county. In other instances, several names may apply to the same road or street. It is recommended that station numbers be verified from the maps in Water Quality Surveillance Section before using them. A misassignment can lead to storage of data in STORET under an incorrect station number.

Ambient monitoring sites which do not have a STORET station number should be assigned a number using the following procedure.

First, from the Water Quality Surveillance Section USGS quadrangle maps verify that the site does not have a number. If it does not, gather information necessary to describe the station from the maps and other sources. Then, assign the site the next number in the appropriate county using the STORET station set-up books in the DNR Data Center. DO NOT WRITE ON TYPEWRITTEN PAGES IN THE BOOKS. Use the handwritten sheets at the end of the county or start a new page. The description in the books should include all of the following information.

- -- description of the source, e.g., Grand River;
- -- landmark description of the location, e.g., at M-21 Bridge;
- -- township, section, town-range and/or city, e.g., Ada Township, Section 35, T7N, R1OW. (Use the downstream section number in cases where the station is on the section line between two sections);
- -- county name, e.g., Kent County;

-- map number as assigned to the USGS maps in Water Quality Surveillance Section.

This description should be complete enough to precisely locate the station on a map. Give as many details as necessary. Latitude and longitude should be given for stations in large lakes. Following the assignment of a station number, immediately proceed to the maps and map it. Use an open arrow away from the monitoring site and put the station number near the end of the arrow. When both 15 minute and $7\frac{1}{2}$ minute quads are present for the same area, map the station on the $7\frac{1}{2}$ minute quad.

7.D.3. DATA HANDLING PROCEDURES FOR STORET STORAGE

7.D.3.a. STORET STATION STORAGE

Ambient stations are set-up in STORET for data storage by completion of station set-up sheets. One sheet should be completed for each new station to be entered into the STORET system. Figures 7.D.-3 through 7.D.-8 are examples of set-ups for various types of stations. On these figures the hand written information must be coded. The following key describes the information to be coded:

- AGENCY CARD (A Card). Skip this card. It is used by STORET group to identify the agency and data being entered.
- STATION TYPE CARD (T Card). Enter either lake or stream after AMBNT/. The word "STREAM" is coded in Figure 7.D.-3.
- STATION CARD (S Card). Enter the following on this card:
 - station number (6-digits) as assigned from set-up books, left justified.
 - optional secondary station numbers (see STORET group for information on secondary numbers and their use).
 - 3-digit EPA (not Michigan) county code number (see Table 7.D.-2).
- LATITUDE/LONGITUDE CARD (HEADER CARD 0). Enter the following on this card:
 - latitude and longitude, zero filling blank spaces (see the STORET group for specific techniques for determining lat/longs).
 - in column 28 code

TABLE 7.D.-3, MINOR RIVER BASIN NAMES IN MICHIGAN

Lake Erie Basin

Lake Michigan Basin

0601 Maumee 0611 Raisin 0612 Huron 0613 Detroit 0614 Clinton 0615 St. Clair

Lake Huron Basin

2101	Cherry Creek
2102	Pigeon
2103	Wiscoggin
2104	Saginaw
2105	Kawkawlin
2106	Rifle
2 107	AuGres
2108	AuSable
2109	Black (at Black River)
2110	Thunder Bay
2111	Swan Creek
2112	Ocqueoc
2113	Cheboygan
2114	Carp
2 115	Pine
2116	Munuscong
2117	Charlotte

2118 Waiska

Lake Superior Basin

2201 Pendills 2202 Tahquamenon 2203 Two Hearted 2204 Autrain 2205 Chocolay 2206 Carp 2207 Salmon Trout 2208 Sturgeon (Houghton County) 2209 Tobacco 2210 Portage Lake 2211 Misery 2212 Ontonagon 2213 Iron 2214 Presque Isle 2215 Montreal

0803 Menominee 0804 Cedar 0805 Ford 0806 Escanaba 0807 Rapid 0808 Whitefish 0809 Sturgeon 0810 Fishdam 0811 Manistique 0812 Millecoquins 0813 Bear 0814 Pine 0815 Elk 0816 Boardman 0817 Platte 0818 Betsie · 0819 Manistee 0820 Big Sable 0821 Pere Marquette 0822 Pentwater 0823 White 0827 Muskegon 0828 Grand 0829 Black River (at Lake Macatawa) 0830 Kalamazoo 0831 Black River (at South Haven) 0832 St. Joseph 0833 Galien

FIGURE 7.D.-2 Example of a Completed Data Sheet

.

: MICI	IGAN	DEPT. OF	NATURA	L RESOU	RCES, I	ENVIR	ONMENT	AL LAB	DRATOF	RY ANA	ALYSI	S EI	v. au	ALITY-	WATER -	POINT	SOURCE	STUDIES	SEC.	
LAB	36	27 PROJ	<u>9</u> Z c	OST 9	73901	PR 3	COLL	ECTED	A. Sa	mple		TRANSFI	RED	4. Go/	For RE	CEIVED	JR	EXAMIN	er _]/	<i>o</i>
	TION LED	ABC	Сом	PANY,	Some	Cit	Y	Rem	PLE ARKS -	Routi	'n e	Comp	iunce	×/15/30	SEND (NAME		TO P	P. Man sint So	unce S	urveys
<		"DO NO	T PUNCH	SAMPLIN	> G	REF	STORET	ISTART DATE YYMMDI		S,T OR B	NUM SAM- FLES	END DATE YYMMDI		IDEP- TH FEET	LAB NO.	ITEMP. DEGREE	PH STAND.	CL2 T. RES	DIS.	IC.O.B. HIGH MG/L
110.		SITE OR	SAMPLE			P 									00003		00400	50060	00300	00340
\$501-1	ABC	Co, Trea	ted Proc	ess 002 T			850001	800810	1300		76	800817	1300		73 99					
8501-1	3		{·		{		850001	800817	1339	<i></i>	76	300818	1335		1400		6.7			
8501-						C03	850001	300817	08/0						1401	25	7.0	F0.2	6.8	
850/-7					V		850001	80031	1335						1402		6.7	F0.2	6.5	
8502-	A ARC	Co Treate	d Proces	ss 008 to		205	850002	8008/0	1240	T = T	192	80081	1230		1403					
9502-1							8 50002	30081	- 1235		172	800818	12 +0		1404		7.2			
850Z-1			<u> </u>		<u> </u>		850002	800317	2/49						1405		7.1	0.45	5.6	
<u>SC-1</u>	<u>6</u> d	K. C. KK	Br abo	ve HBC	Co	277	850159	300817	031					3	7406	22.5	8.0		6.8	
56-2	6d 1	(. @ P)	NEST	below HE	sc Co	C091	850/60	800817	0340					<u> </u>	7401	23.5	<u>_/.9</u>		5.6	
									·	i I 		i 				i 	i 	i 		
NŨ.	AMPUL MG7L 00680	. TPHENUL T. REC UG7L 32730	TOTAL MG/L 00720	FREE MSXL 0072X	TOTAL MG/L 00745	1800 1707 81 MG 1003	-5 INU. AL ITO 7L IMG. 10 1007	3NU2: N FAL TO /L N MO 530 60	1H3 1TAL 57L N 1610	KJEL TOTAL MG/L 00625	N 10R 1 T01 N 1MG 1 705	TAL TO TAL TO 71 PIME 507 100	103. TAL /L P 665	UR6+ TOTAL UG/L 01032	TOTAL MG/L 00940	SU4≃ TOTAL MG/L 00945	ALK. TOTAL MG/L 00410	100-20 CARB M6/L 20087	FLOW CONDUIT A16P S0050	
01	8.7	6				1	4 0	.49 0	0.02	0.27	k').01 0	1.02		38	140		20	13800	
02	9.7	8				1	7 0	16 0	2.01	0.73	0	01 0	0.06		33	130		21	16200	
03						10	6 0.	53 0	.06	0.32	K	0.01 0	1.03						14000	
04						2	4 0	.78 ().04	0.66	KI	0.01 0	0.02						15000	
05						6	9 0.	57 4	4.0	8.5	10	.02 0),/7		68	450		72		
						6	7 0.	70 3	2.0	35.0	0.	19 0	.23		60	490		70		
07						6	7									530				
08		15	K0.01			3.	9 0.	16 1	2.39	1.86	0.	10 0	5.28	8	41	50	204	16		
09		14	K0.01	·		8.	5 0.	19 0	.49	2.19	10.	33 0	.116	1/	44	66	202	20		
10			 																	1
																ESD-003	303 REV.	05/80		

.

1

6/85

"2" for lat/long with 1 second precision or

"3" for lat/long with 10 second precision

- total depth in feet at the station, enter "000" if unknown
- RMI CARDS (HEADER CARDS 1 and 2). Skip these cards. River mile index information is not available at this time.
- MAJOR/MINOR BASIN CARD (HEADER CARD 3). Enter the following:
 - Great Lake name in the major basin spaces as appropriate for the station
 - minor basin name (from Table 7.D.-3) if applicable, or other descriptive information about the station
 - 4-digit map number (from maps in Comp. Studies)
 - major/minor basin code (from Table 7.D.-3). NOTE: For Great Lakes stations use the major basin code and "75" for the minor basin code, e.g., 0875 for Lake Michigan stations.
 - sub-basin code, if defined; "00", if undefined.
- LOCATION DESCRIPTION CARD (HEADER CARD 4). Enter the stream or lake name, landmark to locate station, township name, and section number. Separate the landmark and the township name by ";". If the station is within a city, enter "city of _____", or "in _____" instead of the township and section number. In large lakes the township and/or section number may not be applicable. In these instances, use the space to describe the location from two landmarks or use a bearing and distance from a landmark.

On the HEADER CARD 4 the following abbreviations can be used:

Q	at	MICH	Michigan
ABV	above	N	North
AVE	Avenue	NE	Northeast
BLW	below	NW	Northwest
ЗR	bridge	R	river
BR	branch	S	South
CENT	central	SE	Southeast
CR.	creek	SW	Southwest
DNST	downstream	SEC	section
DRN	drain	STR	stream
E	East	TWP	township
FT	feet	TRIB	tributary
L	little	W	West
L or LK	lake	YDS	yards
MI	miles		

FIGURE 7.D.-3. Station Set-up Sheet (Front) Completed for a Stream Station

STORET WATER QUALITY FILE - STATION LOCATION STORAGE	
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 2 122 23 24 25 26 27 28 29 30 31 3 2 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 56 59 60 61 62 63 64 65 66 67 68 69 70 7 172 73 74 7	75 75 77 78 79 3
AGENCY CODE FILE UNLOCK UNLOCKING CODE INDIVIDUAL STORING DATA NAME, LOCATION, AGENCY (Bigne)	K AFTERICCN-
(Left Just./y) (cops) (Left Just./y) (cops) (Left Just./y) (Optional Comments will not be stored) (Left Just./y) (cops) (Left Just./y) (Left Just./y) (Left Just./y) (cops) (Left Just./y) (<u>R MO (CCDE 75 76 77 78 73 8</u>
	9 9 9 9 1
A STRING OF VALID COMBINATIONS OF STATION TYPE CODES SEPARATED BY SLASHES	
AMBINIT/STREAM	78/79/5
SORT PRIMARY STATION CODE	
NO (Left Justity) (Bigne) (Bigne) (I)SECONDARY STATION CODE (2)SECONDARY STATION CODE STATE COUNTY (Bigne) (Left Justity) (Left Justity) (Left Justity) (Left Justity) 58 59 (Left Justity) 67 68 69 73 72 73	77 76 73 6
390058 26077	NSS
SCRT LATITUDE LONGITUDE (Bionk) (Bionk) (Bionk) (Bionk) (Bionk) (Bionk)	77.
$\frac{1}{4} \frac{1}{2} \frac{1}{2} \frac{1}{2} \frac{1}{3} \frac{1}{4} \frac{1}{2} \frac{1}{0} \frac{1}{3} \frac{1}{4} \frac{1}{4} \frac{1}{2} \frac{1}{0} \frac{1}{2} \frac{1}{5} \frac{1}{5} \frac{1}{5} \frac{1}{3} \frac{1}$	
RMI CARD (HEADER CARD I)	
SORT UP A MAN LEVEL I LEVEL 2 LEVEL 3 LEVEL 3 LEVEL 4 LEVEL 5 LEVEL 5 LEVEL 6 LEVEL 6 LEVEL 7	7 LE.E. ES LSED 76 77 78 79 L
SURT (REALL WA MIN STEAM LEVEL 8 LEVEL 8 LEVEL 9 LEVEL 9 LEVEL 10 LEVEL 10 LEVEL 11 LEVEL 11 LEVEL 12 LEVEL 12	
NO $\frac{1}{1}$ $\frac{3}{2}$ $\frac{4}{1}$ $\frac{6}{7}$ $\frac{6}{12}$ $\frac{10}{11}$ $\frac{13}{14}$ $\frac{17}{10}$ $\frac{10}{21}$ $\frac{21}{22}$ $\frac{2526}{2930}$ $\frac{2930}{33}$ $\frac{33}{34}$ $\frac{3730}{33}$ $\frac{4142}{456}$ $\frac{4550}{50}$ $\frac{5354}{53}$	12 4
SCRT STATION LOCATION MAJOR BASIN NAME STATION LOCATION MINOR BASIN NAME OR OTHER LOCATION INFORMATION MAP NO BASIN CODES	(Blank)
NO 3 4 Major Vince Sub , 3 4 63 64 67 69 67 70 71 72 73 74	79-1
LOCATION OF SITE, TOWNSHIP AND SECTION (Bionk)	
KALAWAZOO RAT "D" AVE BR; COOPER TWP, SEC. 22	7510

6/85

Use entire names where possible and good punctuation. Abbreviate as necessary.

 DESCRIPTIVE PARAGRAPH CARDS (HEADER CARD 5). These lines are used to give a full description of the station location, monitoring justification, and other relevant information for the station. Do not abbreviate on these cards. Indent paragraphs and use punctuation. Different formats are used for streams, inland lakes and Great Lakes as follows:

STREAMS (See Figure 7.D.-4). Enter the following for stream stations in the order given (do not abbreviate):

- First paragraph.

-	stream name and location in respect to fixed landmarks	
-	"SECTION ",	
-	" TOWNSHIP", or "City of	11
-	"T_N, R_E", (change E, W, N, S as appropriate)	
-	COUNTY, MICHIGAN."	

- Second paragraph.

Station justification. Enter a justification or purpose for the station similar to one of the following:

- This station is used to determine background water quality upstream from ______ (enter city, discharger, tributary or other impact on the stream).
- Water quality within ______ is determined at this station (enter a city name).
- Water quality downstream from is determined at this station (enter city, discharger, tributary or other impact on the stream).

- Additional paragraphs (optional information).

Enter additional information relevant to the station. Examples of these are:

- River Mile Point. For mainstream stations enter:

This station is _____ miles (or kilometers) from the mouth.

For tributary stations enter:

FIGURE 7.D.-4. Station Set-up Sheet (Back) Completed for a Stream Station

.

1 2 3 4 5 6	7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 66 69 70 7 172 73 74 75 176 77 78 175 50										
}	DESCRIPTION (7-78)										
U.M. I.D (BIONK)	78.75.3										
	KALAMAZOO RIVER AT THE "D" AVENUE BRIDGE; SECTION 22, COOPER 105										
SCAT NO (BIONX)	7 7 / / / / / / / / / / / / / / / / / /										
2.fit 1.2 (Blonk)	TO[WNS]H[I]P], [T]15], [R]1 1 V , [K]A L A M A Z 0 0 [C 0 U M TY], [M]1 C H I G A N . []] []] []] []] []] []] []] []] []] []										
1 3 4 6	WATER QUALITY DOWNSTREAM FROM KALAMAZOO IS DETERMINED AT THIS 25										
107 1.7 (BIOAR)	7 97 67										
LIFT IL IEIONNI	STATION.										
1 3 4 6	THE MOUTH. STATION IS 57.7 MILLES FROM THE MOUTH.										
ART 112 (213AK)	78 79 60.										
	515										
1 3 4 6	<u>7</u> 										
SCRT 1:0 (BIONK)											
S BL NUL (2) CAN	7.5										
1 3 4 6	75 e7187 [] [] [] [] [] [] [] [] [] [] [] [] [] [
CRT NO (BIONK)	7 78 75 6C										
	9,5										
	78 ⁷ 73 £C										
SCAT NO (BIONK)											
SCHT NO (BIOAR)	78 TB 50										
	7. [2] 70 71 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7										
SURT NO (BIDNE)											

.

6/85

This station is _____ miles (or kilometers, feet, or meters) from the confluence with the ______ river. The confluence is ______ miles from the mouth.

- Flow information. Enter the following:
 - USGS Gage No.
 - Drainage area ratio
 - Drought flow and date of determination

INLAND LAKES (See Figure 7.D.-6). Enter in paragraph format the following for inland lake stations in the order given (do not abbreviate):

- lake name
- depth and location of basin (if basin oriented lake station)
- location of basin by section, town, range, township, county, and state
- location of sampling site from at least two landmarks
- inlets and outlets of the lake
- other relevant information

<u>GREAT LAKES</u> (See Figures 7.D.-7 and 7.D.-8). Enter in paragraph format the following for Great Lakes stations in the order given (do not abbreviate):

- lake name and bay, harbor, strait, or inlet, etc. if pertinent
- distance and direction, or magnetic bearing (give date of bearing) from at least one, and preferably two pertinent landmarks
- information relevant for locating the station from a boat, such as bearings to onshore landmarks, buoys, Loran-C coordinates (including chains), etc.

Other formats may be necessary for stations which are different from the examples above. The important point is to accurately describe the location of the station and include other important information about the station. Documentation of routine samples should be avoided in the paragraph. Unusual conditions or sampling projects (e.g., storm surveys) however, should be documented by giving the date(s) and the conditions. Any other information relevant to data interpretation of the specific station can be entered in the remaining spaces.

Corrections can be made to existing stations by completion of the line(s) of the setup sheet in error and the "S" card. The word "CORRECTION" should be written at the top of the sheet. Note the lines on the back of the sheet each represent one line in the output and in STORET.

When set-up sheets are completed, forward them to the STORET group for entry into STORET. The STORET group reviews the set-up sheets for accuracy and completeness, and then sends them to the Data Entry Section of the Michigan Department of Transportation. This Section keys the information to disk, verifys the keying, and dumps the keyed information to a computer tape. The tape is then sent to the EPA Computer Center FIGURE 7.D.-5. Station Set-up Sheet (Front) Completed for an Inland Lake Station

,

STORET WATER QUALITY FILE - STATION LOCATION STORAGE
1 2 3 4 5 6 7 8 9 10 1 1 12 13 14 1 5 1 6 17 18 19 20 2 1 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 4 9 50 51 52 53 54 55 56 57 56 59 60 61 62 63 64 65 66 67 68 69 70 7 172 73 74 75 76 77 78 79 30
AGENCY CARD (A CARD)
AGENCY CODE FILE UNLOCKING CODE INDIVIDUAL STORING DATA NAME, LOCATION, AGENCY (Bionk) (Left Justify) (Left Justify) (Left Justify) (Deficinal Comments will not be stored) (Bionk) (Bionk) (Bionk) (Bionk) (Bionk) (Bionk) (Bionk) (Bionk) (CCE (Bionk)
2 I M I C H L A N S I N G J J J J J J J J J J J J J J J J J J
A STRING OF VALID COMBINATIONS OF STATION TYPE CODES SEPARATED BY SLASHES
AMBNT/LAKE STATION CARD (S CARD)
SORT PRIMARY STATION CODE FIPS CODES t:0 (Left Justify) (I)SECONDARY STATION CODE (2)SECONDARY STATION CODE (3)SECONDARY STATION CODE 1 3 4 (Left Justify) 56 59 (Left Justify)
1/2/0/1/3/7/1/1/1/1/1/1/1/1/1/1/1/1/1/1/1/1/1
LATITUDE / LONGITUDE LATITUDE / LONGITUDE CARD (HEADER CARD O)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$
HI CARD (HEADER CARD I)
SORT TERM TERM LEVEL 2 LEVEL 2 LEVEL 3 LEVEL 3 LEVEL 4 LEVEL 5 LEVEL 5 LEVEL 6 LEVEL 6 LEVEL 7
NO (BIONA) MAININ STREAM MILES INDEX MILES
INNI CARD (NEADER CARD 2)
NO (BIONK) NA MIN STREAM INDEX MILES INDEX MILES INDEX MILES INDEX MILES (BIONK)
MAJOR/MINOR BASIN CARD (HEADER CARD 3)
SORT STATION LOCATION MAJOR BASIN NAME STATION LOCATION MINOR BASIN NAME OR OTHER LOCATION INFORMATION MAP NO BASIN NAME (BASIN COES) (Bight) (BASIN COES)
MAJ BASINI LAKE MICHIGANMIN BASINI ST. JOSEPH RIVER 0093083200 13
LOCATION DESCRIPTION CARD (HEADER CARD 4)
LOCATION OF SITE, TOWNSHIP AND SECTION . (Bionk) 1 3 4 SI 52 79-62
CRAIG LK N-CENT BASIN; GIRARD TWP., SEC. 29

٠

6/85

FIGURE 7.D.-6. Station Set-up Sheet (Back) Completed for an Inland Lake Station

123456	7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 39 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 73 30
	DESCRIPTIVE PARAGRAPH CARDS (HEADER CARD S)
SORT NO (BIONA)	78 72 83
	CRAIG LAKE IN A 25 FOOT DEEP BASIN IN THE NORTH-CENTRAL PART OF THEUS
GORT NC (BIONK)	767980
SET LOUBLOOK	LAKE. THE BASIM IS LOCATED IN SECTION 29, 155, RGM, GIRARD TOWNSHIP, 115
	7 PPANCU COUNTRY I MUCHUGAN THE COMPULUE SUTE US APPROXUMATER Y 750 FEFT 215
SURT NO IBIONAL	
	NORTHWEST OF AN ISLAND NEAR THE EAST SHORE. INLET: COLDWATER RIVER FROM 35
SORT NG (BIONK)	79 72
SET NO (Blank)	MORRISON LAKE AND UNNAMED STREAM ON EAST SHORE. OUTLET: COLDUATER 1
1 3 4 6	7 PIVER 55
SORT NG (diana)	7
	65
SCRT NO (BIONA)	
SURT NG (Bione)	
STRT NO (Brank)	
	35
1 3 4 6	
SORT NO (Blenk)	$\left[\begin{array}{c} 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 $
	78 173 σ2 Γ Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι Ι
SORT NO (Biank)	78 79 80
SURT NO (BIONA)	78 78 78 78 78 78 78 78 78 78 78 78 78 7
SCRT NO (BIONK)	
	7 7
Quarkan kan dan dan dan dan dan dan dan dan dan d	

∞ FIGURE 7.D7. Station Set-up Sheet (Front) Completed for a Great Lakes Station)n
STORET WATER QUALITY FILE - STATION LOCATION STORAGE	
1 2 3 4 5 6 7 8 9 10 11 12 13 14 5 16 17 1 0 19 20 21 22 23 24 25 26 27 28 29 30 31 3 2 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 4 50 51 52 53 54 55 56 57 58 59 60 61	626364656667686970717273747576777875
AGENCY CARD (A CARD)	
AGENCY CODE FILE UNLOCKING CODE INDIVIDUAL STORING DATA NAME, LOCATION, AGENCY (left Justify) (left Justify) (left Justify) (Optional Comments will not be stored)	(Bionk) (DXX AFTER UN- (Bionk) YR MO CCDE 62 73 74 75 75 77 76 79 80
	A @ @ @ @
	T
(Leff Justify) A STRING OF VALID COMBINATIONS OF STATION TYPE CODES SEPARATED BY SLASHES	78 99 60
STATION CARD (S CARD)	Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ Τ
SORT PRIMARY STATION CODE	FIPS CODES
(Blank) (Blank) (I)SECONDARY STATION CODE (2)SECONDARY STATION CODE (3)SECONDARY (Left Justify) (Left Justify) (Left Justify) (Left Justify)	TY STATION CODE STATE COUNTY (BIONE)
LATITUDE /LONGITUDE CARD (HEADER CARD O)	
SCRT LATITUDE LONGITUDE (Blank) NO (Blank) dd mm sss ddd mm sss ddd mm sss (Blank) C D STATION (Blank) Ad mm sss ddd mm sss ddd mm sss (Blank) C D STATION (Blank)	72 00
4 5 4 10 2 5 0 0 8 4 2 19 5 5 0 1 1 3 F 0 0 3	
RMI CARD (HEADER CARD I)	
SGRT (BIGNA) TERM TERM LEVEL I LEVEL 2 LEVEL 2 LEVEL 3 LEVEL 3 LEVEL 4 LEVEL 4 LEVEL 5	EL 6 LEVEL 6 LEVEL 7 LEVEL 7 LAST DEX MILES INDEX MILES USED
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	
SORT (BION) TERM LEVEL 8 LEVEL 8 LEVEL 9 LEVEL 9 LEVEL 10 LEVEL 10 LEVEL 11 LEVEL 11 LEVEL 12 LEVEL 1 2 NO (BION) MA VII STREAM INDEX MILES INDEX	(Bian #)
	2
MAJOR/MINOR BASIN CARD (HEADER CARD 3)	····· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲···· ▲····
SORT STATION LOCATION MAJOR BASIN NAME STATION LOCATION MINOR BASIN NAME OR OTHER LOCATION INFORMATION	MAP NO BASIN CODES (Blank)
$\frac{1}{2}$	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$
$\frac{1}{1} \frac{1}{1} \frac{1}$	
LOCATION DESCRIPTION CARD (TRADER CARD 4)	
LOCATION OF SITE, TOWNSHIP AND SECTION	(81ank)
I LI HIURION 2000 FIT WINE STOWY POINT & HEADING 34 DEG.	
	en de la

FIGURE 7,D.-7. Station Set-up Sheet (Front) Completed for a Great Lakes Station

1 2 3 4 5 6	7 8 9 10 11 12 13 14 15 16 17 18 9 20 21 22 23 24 25 26 27 29 29 30 31 32 33 34 35 36 37 32 39 40 41 42 43 44 45 46 47 46 44 7 46 49 50 51 52 53 (54 55 56 57 55) 55 (56 61 62 63 64 65 66 66 7 (56 66) 70 71 72 73 74 75 76 77 75 79 5
}	DESCRIPTIVE PARAGRAPH CARDS (HEADER CARD S)
CAT IN (BIONE)	7 72 72 72
	LAKE HURON, 200 FEET NORTH-NORTHEAST OF STONY POINT ON A MAGNETICI OF
50RT NO (BIONK)	7 79.79 6
2011 13 13/2 n k)	COMPAISS BEARING OF 034 DEGREES (AUGUSIT, 11980). THIS STATION IS LOCATED 115
1 34 6	$\frac{1}{21}$
CONT 1.0 (3:0AX)	
	WATER QUALITY RELATED TO A CLADOPHORA PROBLEM OFFSHORE OF THE MOUTH 35
577 NO 13 3 AL	
SCAT NO (BIONK)	OF THE LITTILE BLACK RIVER IS DETERMIMED AT THUS STATION.
SONT 1.0 (3: CAR) 3 4 6	7
3 4 6	
5261 15 (Blonk)	
SCATINO (BIONE)	
5.4.1 N. (2/364)	79 ¹ /5 cc
ECRT NO (BIORN)	
LORI NO (BIONE)	
SET NO (3/20x)	75 ⁷ 3 d
SCRT NO (BILAN)	
3 4 6	

6/85

by the STORET group for entry into STORET. At the Computer Center the data is put onto disk, edited by means of the STORET storage procedure, and then stored.

Errors from the edit are resolved and corrected by the STORET group by using the set-up sheets, maps, and by contacting the user group responsible for the set-ups. After storage, the station is retrieved by the STORET group. The resulting computer printout of the stations is returned to the user along with the station set-up sheets. The retrieval is then proofed by the user against the set-up sheets for accuracy and completeness. Any corrections required should be submitted to the STORET group. These will be submitted to STORET, retrieved and returned to the user for additional proofing.

7.D.3.b. DATA STORAGE IN STORET

7.D.3.b.1. Water Column and Sediment Data Preparation

All ambient water quality data should be entered into STORET so that it will be available for agency usage. The data storage process is diagrammed in Figure 7.D.-9. After the sheets are returned from the lab and reviewed by the program manager and the collector, the data sheets should be prepared for STORET entry as follows:

- Verify that all monitoring sites have been established in STORET. Those not yet established should be coded for input per instructions in Section 7.D.2.
- On the data sheets check data for legibility, accuracy, and completeness. In particular, check station numbers, dates and times. Multiple sheets for the same sample should be cross referenced for consistency of these items.
- Make sure supplemental parameters (i.e., those not preprinted) are coded with the correct parameter numbers, and that precoded parameter numbers have not changed since the data sheet was printed.
- Check that each cell has only one valid STORET remark code preceding the value. Eliminate the two digit clarifying remark codes following the data values by marking through them in red. Values which are deemed incorrect because of the remarks should not be stored.
- Any parameter column not to be entered (i.e., was not analyzed or the parameter number is not available) should be crossed out in red and a vertical line drawn through the column. In parameter columns where a particular cell has information which is not to be stored, place a red X in that cell. Any complete sample on the sheet which is not to be entered (i.e., non-STORET station or invalid results) should be crossed off in red.

FIGURE 7.D.-9. Process of Data Storage in STORET Water Quality File



- Field parameters should be coded with the correct remarks codes and detection limits.
- If any data for a study is to be stored at depth, then all data for the study should have a depth indicated. For surface samples in these cases indicate 0 for the depth. This will result in a more consistant STORET retrieval.

Figure 7.D.-10. is an example of a data sheet which has been edited for keypunch.

7.D.3.b.2. Tissue Data Preparation

The storage of tissue data in STORET requires unique coding from other data. Special parameters are to be coded where applicable as follows:

84005	Alpha Species Code
74990	F + W Species Numeric Code
74995	Numeric Tissue Code
84007	Alpha Tissue Code
81614	Number of Individuals in sample
81615	Number of Species in sample
00024	Length in Inches
00023	Weight in Pounds
00029	Field ID. No. (Great Lakes Grid No. Only)

Species and tissue codes are listed in Tables 7.D.-4 and 7.D.-5.

When coding tissue data, station number, year, month, and day are indicated as usual (see Section 7.D.3.a.l.). The time of sampling however, is used to indicate the type of tissue that was analyzed. Table 7.D.-5 lists the time code for each tissue type. The appropriate tissue time code must be coded in the time field. Note that the code placed in the time field is not always the same as that coded under parameter 74995, numeric tissue code. This is due to the limitation of 0-59 minutes in the time field.

The depth field is used to identify species and sample sequence. The value coded in this field is a combination of the numeric species code plus a one digit sequence code appended to the right of the species code. Up to ten samples of the same species per date can be coded using this method. Start the sequence coding with a 1 and proceed through 0, with 0 representing the tenth sample. If more than 10 samples of the same species are collected, the date should be increased by one day and the sequence started over with 1. Continue increasing the date of sampling until all samples for that species are coded.

If a composite sample is collected, do not code as a regular composite. Instead code as a grab with the appropriate numbers under the parameters for number of individuals and number of species in sample. Where more than one species is included in the sample the species code of "MIX" is appropriate.

TABLE 7.D.-4. MICHIGAN FISH LIST BY FAMILY

COMMON NA	AME	SCIENTIFIC NAME	STORET ALPHA CODE (84005)	STORET NUMERIC CODE (74990)	BIOSTGRET CODE
	LAMPREYS (PETROMYZ	ONTIDAE)			
SILVER LA NORTHERN CHESTNUT SEA LAMPE	AMPREY BROOK LAMPREY LAMPREY REY BROOK LAMPREY	ICHTHYPZYZON UNICUSPI: ICHTHYOMYZON FOSSOR ICHTHYOMYZON CASTANEU: PETROMYZON MARINUS LAMPETRA LAMOTTEI	5 SVL SBL 5 CHL NA 4BI	445 331 330 NA	32040201002007 32040201002004 32040201002002
	PADDLEFISHES (POLY	ODONTIDAE)	F12.12		010101000001
PADDLEFIS	зн	POLYODON SPATHULA	PF	106	32060102001001
	STURGEONS (ACIPENS	ERIDAE)			
LAKE STUP	RGEON	ACIPENSER FULVEOSCENS	LST	099	
	GARS (LEPISOSTEIDA	E)			
SPOTTED (LONGNOSE	GAR GAR	LEPISOSTEUS OCULATUS LEPISOSTEUS OSSEUS	SG LG	050 032	32060201001001 32060201001002
	BOWFIN (AMIIDAE)				
BOWFIN		AMIA CALVA	BF	068	32060301001001
	TROUTS, WHITEFISHE	S (SALMONIDAE)			
ATLANTIC BROWN TR RAINBOW BROOK TR LAKE TRO CISCO (L SISKOWIT IVES LAK SHORTNOS SHORTJAW LONGJAW BLOATER KIYI BLACKFIN DEEPWATE LAKE WHI PYGMY WH ROUND WH ARCTIC G PINK SAL	SALMON OUT TROUT OUT UT AKE HERRING) LAKE CISCO E CISCO CISCO CISCO CISCO CISCO CISCO TEFISH ITEFISH ITEFISH ITEFISH RAYLING MON	SALMO SALAR SALMO TRUTTA SALMO GAIRDNERI SALVELINUS FONTINALIS SALVELINUS NAMAYCUSH COREGONUS ARTEDII COREGONUS BARTLETTI COREGONUS BARTLETTI COREGONUS REIGHARDI COREGONUS REIGHARDI COREGONUS ALPENAE COREGONUS ALPENAE COREGONUS NIGRIPINNIS COREGONUS NIGRIPINNIS COREGONUS NIGRIPINNIS COREGONUS CLUPEAFORMI PROSOPIUM COLLTERI PROSOPIUM CYLINDRACEU THYMALLUS ARCTICUS ONCORHYNCHUS GORBUSCH	ASL BT RBT BKT LT CIS NA NA NA NA BLO KIY NA S LWH NA NA S LWH NA A A CR A	167 011 039 096 029 085 NA NA NA NA 006 098 NA NA 030 NA 030 NA 044 002 NA	32060901004005 32060901004006 32060901005007 32060901005004 32060901005004 32060901001002 32060901001005 32060901001007 32060901001004 32060901003003 32060901003003
COHO SAL SOCKEYE CNINDOK	MON Salmon Salmon	ONCORHYNCHUS KISUTCH ONCORHYNCHUS NERKA ONCORHYNCHIS TSHAWYTS	CHO SOS CHA CHN	081 481 083	32060901002003 32060901002004 32060901002005

SMELTS (OSMERIDAE) RAINBOW SMELT OSMERUS MORDAX RSM 080 32060902003001 HERRINGS (CLUPEIDAE) ALEWIFE ALOSA PSEUDOHARENGUS AW 075 32060701001005 GIZZARD SHAD DOROSOMA CEPEDIANUM 6SH 021 32060701004001 MOONEYES (HIODONTIDAE) MOONEYE HIDDON TERGISUS MO 034 32060801001002 MUDMINNOWS (UMBRIDAE) CENTRAL MUDMINOW UMBRA LIMI CMM 334 32060906003001 PIKES (ESOCIDAE) GRASS PICKEREL ESOX AMERICANUS GPK 091 32060907001001 NORTHERN PIKE 32060907001002 ESOX LUCIUS NP 036 MUSKELLUNGE ESOX MASQUINONGY ML. 084 32060907001003 SUCKERS (CATOSTOMIDAE) 32061103007003 BLACK BUFFALD ICTIOBUS NIGER BL B 105 SMALLMOUTH BUFFALO ICTIOBUS BUBALUS SBU 048 32061103007001 OHTHE BACK CARPIODES CYPRINUS QUB 074 32061103001002 MOXOSTOMA DUQUESNEI BLACK REDHORSE BRH 339 32061103010007 GOLDEN REDHORSE MOXOSTOMA ERYTHRURUM RSU 041 32061103010008 SILVER REDHORSE 170 32061103010001 MOXOSTOMA ANISURUM SRH SHORTHEAD (NORTHERN) REDHORSE MOXOSTOMA MACROLEPIDOTUM SRD 101 32061103010012 MOXOSTOMA VALLHCIENNESI GREATER REDHORSE NA NΑ RIVER REDHORSE MOXOSTOMA CARINATUM RRH 388 32061103010004 NORTHERN HOG SUCKER HYPENTELIUM NIGRICANS NHO 094 32061103006002 WHITE SUCKER CATOSTOMUS COMMERSONI 32061103002005 WSÚ 061 LONGNOSE SUCKER CATOSTOMUS CATOSTOMUS LSU 033 32061103002002 SPOTTED SUCKER MINYTREMA MELANOPS SSU 051 32061103009001 LAKE CHUBSUCKER ERIMYZON SUCETTA LC 028 32061103005002 CREEK CHUBSUCKER ERIMYZON OBLONGUS CCS 387 32061103005001 MINNOWS CARP CYPRINUS CARPIO С 32061102007001 012 GOLDFISH (1) CARASSIUS AURATUS GF 024 32061102004001 GOLDEN SHINER NOTEMIGONUS CRYSOLEUCAS GSN 022 32061102026001 CREEK CHUB SEMOTILUS ATROMACULATUS CKC 090 32061102039001 PEARL DACE SEMOTILUS MARGARITA PD 329 32061102039003 REDSIDE DACE GILA ELONGATA NA NA PUGNOSE MINNOW NOTROPIS(OPSOPOEODUS) EMILIAE PNM 358 32061102027041 PHOXINUS NEGOGAEUS FINESCALE DACE NA NA SOUTHERN REDBELLY DACE PHOXINUS ERYTHROGASTER RBD 381 32061102030002 NORTHER REDBELLY DACE PHOXINUS EOS NA NA LAKE CHUB COUESIUS PLUMBEUS NA NA HORNYHEAD CHUB 349 NOCOMIS BIGUTTATUS HHC 32061102025002

NOCOMIS MICROPOGON

HYBOPSIS STORERIANA

RC

SVC

073

346

RIVER CHUB SILVER CHUB

7-33

32061102025005

32061102016015

BIGEYE CHUB	HYBOPSIS AMBLOPS	BEC	343	32061102016002
BLACKNOSE DACE	RHINICHTHYS ALTRATULUS	BUD	554	32061102035001
LONGNOSE DACE	RHINICHTHYS CATARACTAE	LNE	108	32061102035002
EMERALD SHINER	NOTROPIS ATHERINOIDES	ESH	077	32061102027012
STLVER SHINER	NOTROPIS PHOTOGENIS	NA	NA	
ROSVEACE SHINER	NOTROPIS FUBELLUS	BES	370	32061102027075
REDEIN SHINER	NOTROPIS HMESATILIS	RSR	376	32061102027092
COMMON OUTNER	NOTROPIS CORNETES	reu	000	32061102027037
TONICOLOD CUINED	NOTROFIC CUALVEACHE	100	254	22001102027000
LIFER CUINED	NOTROPIC TEYANUS	100	275	32061102027027
READER OUTNER	NOTBODIC RETEDOTION	NO	07-0 NA	52061102027057
CONTAIN CUINCO	NOTROFIS REFERENCES	NH		
DICHOUTU CUINER	NOTROFIS RODONIUS	NH	NH	
DIGNOOTH SHINER	NOTROFIS DOROHLIS	000	070	000/1100007005
SPUTFIN SHINER	NOTROPIS SPILOPIERUS	375	3/3	32061102027085
PUGNUSE SHINER	NUTROFIS ANUGENUS	NA		000/1100007007
SAND SHINER	NOTROPIS STRAMINEUS	505	432	32061102027087
MIMIC SHINER	NUTROPIS VOLUCELLOS	MCS	3/8	32061102027095
BLACKNUSE SHINER	NUTRUPIS HETERULEPIS	NA	NA	000/1100010001
SILVERJAW MINNUW	ERICYMBA BUULATA	SUM	443	32081102010001
BRASSY MINNOW	HYBOGNATHUS HANKINSONI	NA	NA	
BLUNTNOSE MINNOW	PIMEPHALES NOTATUS	BLM	087	32061102031001
FATHEAD MINNOW	PIMEPHALES PROMELAS	FHM	382	32061102031002
STONEROLLER	CAMPOSTOMA ANOMALUM	SRL	335	32061102003001
LOACHES (COBITIDA	E)			
ORIENTAL WEATHERFISH	MISGURNUS ANGUILLICAUDATUS	NA	NA	
CATFISHES (ICTALU	RIDAE)			
PLACK BULLHEAD	ICTALURUS MELAS	BKB	004	32061201001005
BROWN BULLHEAD	ICTALURUS NEBULOSUS	BRB	010	32061201001007
YELLOW BULLHEAD	ICTALURUS NATALIS	YEB	062	32061201001006
CHANNEL CATFISH	ICTALURUS PUNCTATUS	CHC	016	32061201001010
FLATHEAD CATEISH	PYLODICTIS OF TVARIS	FHC	019	32061201003001
TADPOLE MADITOM	NOTHRUS GYRINUS	TEM	397	32061201002012
STONE CAT	NOTURUS ELAVUS	SCT	396	32061201002008
BRINDLED MADTOM	NOTURUS MIURUS	BRM	399	32061201002017
ELLS (ANGUILLIDAE)			
AMERICAN EEL	ANGUILLA ROSTRATA	AME	076	32060501001001
TOPMINNOWS (CYPRI	NODIONTIDAE)			
BANDED KILLIFISH	FUNDULUS DIAPHANUS	BKF	209	32061804006006
STARHEAD TOPMINNOW	FUNDULUS NOTTI	STM	405	32061804006015
BLACKSTRIPE TOPMINNOW	FUNDULUS NOTATUS	BTM	404	32061804006014
LIVEBEARERS (POEC	ILIIDAE)			
MOSQUITOFISH	GAMBUSIO AFFINIS	MQF	407	32061805002001
CODFISHES (GADIDA	E)			
BURBOT	LOTA LOTA	BUR	082	32061702007001

æ

TROUT-PERCH (PERCOPSIDAE)

· · . .

TROUT-PERCH	PERCOPSIS OMISCOMAYOUS	NA	NA	
PIRATE PERCH (APHR	REDODERIDAE)			-
FIRATE PERCH	APHREDODERUS SAYANUS	PP	410	32061302001001
SILVERSIDES (ATHER	(INIDAE)			
BROOK SILVERSIDE	LABIDESTHES SICCULUS	BSS	033	32061806005001
TEMPERATE BASSES	(PERCICHTHYIDAE)			
WHITE BASS	MORONE CHRYSOPS	WHB	057	32062 302001002
SUNFISHES (CENTRAF	RCHIDAE)			
SMALLMOUTH BASS LARGEMOUTH BASS WARMOUTH GREEN SUNFISH PUMPKINSEED REDEAR SUNFISH BLUEGILL SUNFISH LONGEAR SUNFISH ROCK BASS WHITE CRAPPIE BLACK CRAPPIE	MICROPTERUS DOLOMIEUI MICROPTERUS SALMOIDES LEPOMIS GULOSUS LEPOMIS CYANELLUS LEPOMIS GIBBOSUS LEPOMIS MICROLOPHUS LEPOMIS MACROCHIRUS LEPOMIS MEGALOTIS AMBLOPLITES RUPESTRIS POMOXIS ANNULARIS POMOXIS NIGROMACULATIS	SMB LMB W GSF SF RES BGS LSF RKB WHS BKS	047 031 056 025 038 040 008 072 043 059 005	32062305008002 32062305008005 32062305007004 32062305007002 32062305007003 32062305007009 32062305007008 32062305007008 32062305002002 32062305009001 32062305009002
PERCHES (PERCIDAE)			
WALLEYE BLUE PIKE SAUGER YELLOW PERCH BLACKSIDE DARTER LOGPERCH RIVER DARTER CHANNEL DARTER NORTHERN SAND DARTER JOHNNY DARTER GREENSIDE DARTER IOWA DARTER RAINBOW DARTER ORANGETHROAT DARTER FANTAIL DARTER LEAST DARTER	STIZOSTEDION VITREUM STIZOSTEDION VITREUM GALUCUM STIZOSTEDION CANADENSE PERCA FLAVESCENS PERCINA MACULATA PERCINA CAPRODES FERCINA COPELANDI AMMOCRYPTA PELLUCIDA ETHEOSTOMA NIGRUM ETHEOSTOMA BLENNIOIDES ETHEOSTOMA BLENNIOIDES ETHEOSTOMA CAERULEUM ETHEOSTOMA SPECTABILE ETHEOSTOMA FLABELLARE ETHEOSTOMA MICROPERCA	WE NA SAG YP BSD LP RD CHD NA GRD NA GRD NA GRD NA D GRD D TD LD	055 NA 046 063 436 433 441 434 NA 109 545 NA 546 429 550 422	32062306005002 32062306005001 32062306003001 32062306004011 32062306004004 32062306004002 32062306002044 32062306002005 32062306002007 32062306002065 32062306002041
DRUMS (SCIAENIDAE)			
FRESHWATER DRUM	APLODINOTUS GRUNNIENS	FWD	020	32062322001001
SCULPINS (COTTIDA	Ε)			
FOURHORN SCULPIN MOTTLED SCULPIN SLIMEY SCULPIN SPOONHEAD SCULPIN	MYOXOCEPHALUS QUADRICORNIS COTTUS BAIRDI COTTUS CO®NATUS COTTUS RICEI	NA MDS SLS NA	NA 443 079 NA	32062371009005 32062371009008

-

.

STICKLEBACKS (GASTEROSTEIDAE)

BROOK STICKLEBACK NINESPINE STICKLEBACK	CULAEA INCONSTANS Pungitius pungitius	NA NA	NA NA	-
MISCELLANEOUS CODES ++++++++	+++++++++++++++++++++++++++++++++++++++	*+++++++++	*****	*****
MIXED SPECIES		MIX	64	
UNKNUWN FISH Daco (UNKNOUN CDECIEC)		UNK	65 05	22042205002
CRAPPIE (UNKNOWN SPECIES)	POMOYIS (GENUS)	CPY	97	32062303003
MINNOWS (UNKNOWN SPECIES)	CYPRINIDAE (FAMILY)	MIN	100	32061102
SUCKER (UNKN SPECIES)	CATESTOMUS SPP.	SUK	104	32061103002
SMELT (SPECIES UNKNOWN)	(FAMILY) OSMERIDAE	SMT	152	32060902
REDHORSE	MOXOSTOMA SPP.	RHS	192	
COMMON CHUB	GILA SP.	CCB	480	32061102012
CUTHROAT/RAINBOW TROUT	HYBRID	CRT	482	
HYBRID BASS (WHITE/STRIPED)	MORONE SP.	HYS	198	
SHINER (COMPOSITE)	NOTROPIS SP.	SHI	497	
CARP/GOLDFISH (HYBRID)	N/A	CGH	500	
CATFISH (UNKNOWN SPECIES)	ICTALURUS (GENUS)	CFH	519	32051201001
SUNFISH (UNKNOWN SPECIES)	LEPOMIS (GENUS)	CSN	521	3204230 5007
COMMON SCULPIN	COTTUS SP.	SCU	534	
STICKLEBACK	GASTEROSTEUS SP.	STK	536	
GRUNT	HAEMULON SP.	GRU	537	

NA = CODE NOT PRESENTLY AVAILABLE

.

DESCRIPTION	STORET NUMERIC TISSUE CODE 74995	STORET TIME CODE	STORET ALPHA TISSUE CODE 84007
ALIMENTRY	01	$\bigcirc \bigcirc \bigcirc 1$	AL 1 M
MOUTH	02	0002	MOUTH
TEETH	03	8000	TEETH
ESCENACIO	04	0004	FSOPH
STOMACH	0.57	0005	STOM
I TVFR	04	0004	
TATESTINE	077	0007	1817 GH
CALL DIAGNED	107 700		1. (M. E. C. F. 1. (M. E. C. F.
ANNS	199 Alto		
MNCO CARDIO HACCHIAC	U 2 4 A		HNUD
LARDIU-VASUULAR	10		LHHK LL Y
	1. 1.		
HEARIZVENIRILLE	1 2	0012	門を出合い
HEARI/BULB ART	13	0013	HBART
HEART/AURICLE	14	0014	HAUR
HEART/CONUS ART	15	0015	CONUS
ARTERIES	$1 \oplus$	0014	ARTS
VEINS	1.77	00i.7	a new analysis and a second
ENDOCRINE	18	0018	ENDON
CYCLIC CHANGE	19	0019	CYCCH
PITUITARY	20	0020	PITY
RENAL BODY	21	0021	RNEY
ADRENAL	22	0022	ARNI
SIPBARENAI		0073	C P P N
HUTIMARGAN RODY			
DOCTIONDORAN DODT	and a sub-	0004	
CODUCORHNOR Composition Andrea	21. (1) 		raon Tata
LUNH UF SIAN Thistopia	uli / mom		Luiusige mais as viewere
			INTRU TANG
FANCREAS	27		t data. Tana ar
SAC VASCULE	30	00:30	SACVS
EXURETURY	131	0031	EXCRI
KIDNEY	32	0062	KIDNY
KIDNY/GLOM	33	0033	KGLM
KIDNEY/AGLOM	34	0034	ce <u>e</u> r
KIDNEY/URIN TUB		0035	KDETB
KIDNEY/COLL TUB	36	0036	KCCLL
BLADDER	37	0037	BLADR
URETERS	28	0038	
URINARY FORE		0020	이구 아파 그
HEMOPOIETIC	40	0040	
HEAD KIDNEY	41	5554 t	HE CO
THVMLE		0042	지 않는 것 같은 것 같
CO FEN	1. 1.	0040	· · · · · · · · · · · · · · · · · · ·
- VMDHADVIIC	1 A	and the second sec	nan saise Si taina tai u
		side side and the side of the	
		Sign Sign ## sign strateging	a sana a sana ang sa
1 MROUNDOLT (ES	Ange (2)	ಸ್ಥೆಂಗ್ಯತಿ ಕೊಳ್ಳಲ್ಲಿ ಮುಂದು ವಿಶ್ವ ಗಳಗ	
EUSIMURHILES	4		Carlor and A
AUTEROFHILES	4	0048	
URANULOCYTE8	<u>a</u> tăr	이가 있 <mark>다</mark> 같은 것이다.	
MUSCO-SKEL		그는 뜻은	The Brazilian Street Stre
MUSCLE/SOMATIC	tin≣ na anti-sa anti-sa	0051	and a star for a star for the star and a star for the st
MUSCLE/VISCERAL	and and and	지수 동물	
		1×0 131 B	an an she
RUME/ACCLLIGA		12 A 11 A	
n an	and the		

JONN TISSUE	56	0034	白いて夢し
le mais	100	CC57	
BKIN -		00580	ININ
WHOLE ORGANISM		0059	WHCRO
NERVOUS	60	0100	s di installa E 112 d'Alexandre
BRAIN	61	0101	BRAIN
SPINAL CORD	62	0102	SPINE
GANGLIONS	63	0103	C:ANC
NEURONS	6.4	0104	NEURN
NERVE FIBERS		0105	NVEIB
REPRODUCTIVE	36	0106	PEARO
REFRO CYC CHAN	67	0107	THE C
MALE	<u>43</u>	0108	
FEMALE	69	0109	FMALE
OVARY	70	O11O	OVARY
RESPIRATORY	71	0111	REGP
GILLS	72	0112	GILLS
RESP EPITHELIUM	73	0113	REPI
CHLORIDE CELLS	74	0114	CHLOR
SECRETORY CELLS	75	0115	SECCL
GILL RAKERS	76	0.1.1.6	GLEKE
SENSORY	77	0117	
LATERAL LINE	78	0118	LATLN
NASAL PASSAGES	79	0119	NASAL
TENTACLES	SO	0120	TENT
EYES	81	0121	EYES
EARS	82	0122	EARC
NEUROEPITHELIUM	83	0123	MRCEP
SWIM BLADDER	(⊒:4	0124	SWBLD
LYMPH SYSTEM		0125	LYMEH
FILET	御仏	0126	FILET
EDIBLE FORTION	87	0127	EAT
HEADLS WHL FISH		01128	ribLS
EVISCERATD WH F	89	O 1.2%	EVICO
VISCERA FISH	(2) (2)	0130	VIEC
LIPID TISSUE	91	0131	LIFID
FRY	92	0132	FRY.
EGGS	93	0133	ESCS.
UNKNOWN	74	0134	4.846
NO HEAD OR VISC	95	0135	NaCher√
NO SIN,HD,VISC	$\Im \Delta$	0136	as ve
EXOSKELETON	$\odot 7$		

FIGURE 7.D.-10.Example of a Data Sheet Edited for Keypunch

1 MICE	HIGAN DEPT. OF NATURAL RESO	URCES, ENVIR		AL LABOR	ATORY	ANALYS	IS	ENIVR	DINMEN FAL	OUAL I T	Y - h	ATER	- 681	NERAL U	BAGE	
LÀB	6070 PROJ 24 COST	93007 FR	7 COLLE	CTED B	ob W	'ood	TRAN	SFERED			EEIVE I AR	ті "Я		EXAMIN	ER Jos	mleck
LOCA SAMP	TION Monthly Monitori	ng Run 7	<i>‡5</i>	SAMPL REMAR	E K	Contin	. M	on.to	ing	SEND - (NAME	RESUL & SE	TS TO CTION	, <u>J.</u>	Vihter	lic, C	Studies
	DESCRIPTION OF SAMPLE	> REF NO.	STORET NUMBER	START DATE YYMMDD	TIME MIL TITT	DEP-INI TH FEET 2	AB	ŢX	IOXYGEN DIS. MG/L	10.0.D. HIGH MG7L	AMIX		ENOL REC 57L 730	CN TOTAL MG7L 00720		1800-5 TOTAL S MG/L
10.	STIE OR SHIPLE		190162	800826	1125	14	208		75	25						ACC
	Flint R P Elmi Rel	102	7 50033	800826	0925	14	209		6.7	41	1	0	.8	0.0029	r	ACC
	Flint R. Q Carponter Rd		250098	800826	1005	14	210		6.4	41		0	.6	0.0015		ACC
	Pinnebog R. C. US-25	<u></u> 104	320100	800826	1325	14	211		10.7							ACC
	Clinton R.C. N. Bad Grath	ot Ave 105	500233	800827	1005	11	2/2		6.5	33						ACC
	Clinton R. C. Humlin Rd	104	630252	800827	1105	14	2/3		9.0	22		/	0	0.013		Acc
	Clinton R. @ HI-59	107	630 529	800827	1145	14	2/14		7.2	22	1	0	.6	0.0018		ACC
	Black R. Q Wadhams	<i>RL</i> 108	740153	800826	1630	1-4	215		9.2							Acc
	Pine R. C S. St. Clair Bo	at DK. 109	740166	800827	0835	1-7	2/6		6.8							ACC
	Belle R. C. Meisner Rd.	110	740167:	800827	09001	:/4	217	<u> </u>	: 8.5		<u></u>					ACC
	<pre>/</pre>			<u>/ </u>	/				~	<u> </u>	XIII					
REF NO.	NOSNO2 NOS NOZ NH3 TOTAL TOTAL TOTAL TOTAL MG/L NIMG/L NIMG/L NIMG/L 00430 10420 00419 00410	086 N KJE TOTAL TOT N MG7L N MG7 00605 006	L N:081 AL TOT L N:MG7 25 705	H.P.PHU AL TOT L P.MG7 07 004	5. AL L P 65 3	MBAS TO MG/L TO SX60 OI	R6+ ITAL IG7L 032	TOTAL MG L 00951	TOTAL MG/L 00940	91 REACT. MG/L 00958	1014 1014 116 10074	≕ AL L TOT L MO S OO4	K. FAL 57L 110	HCO3- MG/L 100440	COS= 10/L 00445	DEGREE CENT. 00010
01	0.65 0 0.32	01.5 /	.8 0.0	533 0.1	51			/	98	1.13				c /	C	24
02	0.95 0 0.022	C 1.8 1	9 0.0	056 0.1	23		/	/	51	2.7				c /	c	23
03	0.006 0 0.002	01.6 1.	6 0.0	010 0.1	04		\Box		27	2.7			\sum	1	ſ.	23.5
04	0.015 C 0.000	0.87 0.9	38 0.0	060 0.1	46		X		67	1.2			}	<u>6</u>	<u>c</u>	23
-05	3 .0 C 0.16	3 CO.96 1.	1 0.	102 0.1	80		\square		93	3.0				7	C	23.5
05	4.0 0 0.014	4 0 1.0 1.0	0.	030 0.0	263	/		\	103	1.42		/-		<u> </u>	C	13.5
07	0.04/ C 0.018	3 10.62 0.6	4 0.0	03 0.0	20	/		<u> </u>	50	1.95		/		<u> </u>	C 	24.5
08	0.003 0 0.004	¢ 0 1.2 1.	2 0.	015 0.0	075			<u>\</u>	51	1.82		<u> </u>		r	Ş	25.5
09 	0./32 0 0.06	7 0.74 0.	B1 0.0	X7 0.0	63	·/		·\	85	2.6				C	<u></u>	23
10	0.017 10 (1 1 0.018	3 + 0 / 1 + /.	1 : 0.0	35:0.1	14 1/			· \	14	3.5	<u> </u>		1	0 +	<u> </u>	23.5
i											_ESD+	03201	REV.	05/80		

The use of the time and depth fields in the above manner allows for selection of specific tissues and/or species during the retrieval and analysis procedures using STORET.

Figure 7.D.-11 is an example of a coded tissue sample sheet.

7.D.3.b.3. User Data Entry, Review, Editing and Proofing

Users are responsible for tabulating laboratory data on appropriate STORET data sheets. Edited STORET data sheets are then entered into an IBM-PC or equivalent microcomputer via KEYDATA COM. The user then reviews the microcomputer master file for completeness and errors. The Master File is then sent to the STORET Group which transmits the data to EPA Computer Center for storage in STORET.

At the Computer Center the STORET group transfers the data to disk, and edits it. The edit is performed using a set of programs called SIDES (STORET Input Data Editing System). SIDES was developed by EPA Region IV to provide for better editing than the STORET system provides. The SIDES system provides the following editing:

- Station number check from an independent list of stations for the batch being edited.
- Parameter number check from an independent list of parameter numbers for the batch being edited.
- Date check from a date specification card.
- Checks for valid information in each field.

Errors found by the SIDES edit are listed on an error report. The errors are corrected, and the SIDES edit is rerun until a good edit is accomplished. An echo printout of the original data sheets is then produced for the edited data.

Once a successful edit is made on the data, the STORET group returns the data sheets and the echo printout of the data to the user. The user proofs the printout against the original data sheets and notifies the STORET group of errors. The STORET group corrects any errors, and then stores the data in STORET.

After storage in STORET, the data is retrieved. The resulting printout is used to verify data storage and the corrections. The user is then notified that his data is stored and available for STORET retrieval and analysis.

For ongoing programs in which stations are periodically sampled, newly stored data is compared against previous stored data for inconsistencies. This review is performed by the STORET group. Users and/or the laboratory staff are contacted for verification of inconsistencies. Any errors which are found are then corrected in STORET. FIGURE 7.D.-11. Completed Tissue Data Sheet Edited for Keypunch

.

MIC	HIGAN	DEPT. OF	NAT	TURA	L RESOU	RCES,	E	VIRO	MENT	AL LAB	ÓRATO	RY	ANAL	YSIS	s	ORG	ANIC	с <u>я - т</u>)	ISSUE -	WET	WT.	BAS	15				
LAB	. 185	C PROL	GL	0 0	UST P	5230	PF	₹ 7	COLLI BY	ECTED	J. Ha	1-1	<i>¹1</i>	1	RAN 0	SFER	ED _		A1		/ED	JR		EXA	MIN	ER JE	
LOC	" ATION FLED	Lake Si	pe	riot	<u> </u>	: M.	irg	mett	e	्म SAM REM	PLE ARKS								SEND	RESU		TO ION))ر ري_	oe i omf	/; h	telic Studie	٤
FIEL	D	"DO NO DESCRIPT SITE OR	IT FL ION SAMP	INCH OF LE	SAMPLIN	 G	-> ! F	REFIS	INBER	START DATE YYMMD		1E T	DEP- TH FEET		EV.	SPE F & NŬ 749	CIE W 70	SPECIE CODE ALPHA 84005	NUMERO TISSUE CODE+ 74995	ALF TIS CO 840	PHA SUE DE*	INUMI IND IDU 816	BER' IV- ALS 14	NUM SPE 816	BER F CIE 15	LENGTH IN INCHES 00024	WEIGHT IN FOUNDS 00023
1334-	8 LK	Syperio	- M	15-4	-GRID 13	3f TH	E .	101 5	20189	740627	012	6	0298	25	51	2	9	LT	86	FI	LET			1		22.8	3.9
1334-	9		Ĩ				1	102					0299	25	52	1					1	1				23.0	5.5
1334-	10							103		V			0 2 9 0	25	53									_		21.7	3.5
1334-	<u>'</u>						1	04		740628		!	0291	25	54											2.7.0	11.1
1334-1	2							105					0292	25	55											25.9	8.5
1334-1	3						11	106					0293	25	6											25.3	7.3
1334-	4						-11	107			. i	i.	0194	25	<u>\$7</u>						<u> </u>		i			29. Z	8.0
1334-	15						<u>i</u>	08					0295	25	58											27.0	6.1
1331-1	4						1	09				_	0276	25	59						L					27.6	3.8
1334-	17		$\underline{\mathbf{V}}$				<u>!</u>]	10!	$\underline{\mathbf{V}}$		<u>i</u> V	<u>_ i</u>	0297	25	50	<u> </u>	<u>/ i</u>	V	· V	! \		<u>i</u> 1	/ !	7		14.5	1.3
İ		<hr/>			·····			<u> </u>	<u> </u>	<u> </u>	1																
REF I	ALPHA	L FAT	1A X	23/2	A-1254	A-1	6				opr_	¦D	ILDRN	1.CHP	FIIN	GLE	CS		-	;		:	;		1		
i i	10 84023	139105	M57	Ki I	MG/KG	MyZI	§ !	MGZKG	ing,	KG IN	57KG	in.	67KG 9404	MOZ	R	TO	201			i		i					
ii	MOA	10.7	<u></u>	<u>t</u> -	1	/1	i	2 16		70		1-	 0 07		F=-	000	47		·	i		!	į				
$\frac{1}{02}$	1	10.2	!	+	7.72		i	1 69		27	112	1-	0.05			133				1			[
03		30.6	!		2 0		!	A 74		15	201	-	0.06				į						į		i		
 04	·	FA 0	!	t	17 2		}	2 2 4		78 78	070	1-	209						İ			!			i		
 05		37.0	i	+	5.66		<u>+-</u> !	17		13	107	-	0.08				į										
06		30.4	i	1	575		t-i	1.00		36	· 03		005			! <i>†</i>			i	i			· į				
07		101	i	t i	2 4 4		<u>+-</u>]	101		70	1 10	·	0 03				i		İ	i			· į				
08		5.9		ti	6.16		<u>+-</u> !	1.36		41 1	17	<u> -</u>	2.03						İ								
09		94		<u>├</u>	2.09		<u></u> †−!	100		11 1	· 7 -	1-1	- <u>07</u>	i			į		!				· į		i		
10		N DEPT. OF NATURAL RESOURCES. ENVIRONMENTAL LABORATION ANALYSIS ORGANICS - TISSUE - UET WT. BASIS 256 FOOD CL_CORE TABLE 75230 PR 7 CLLCTED J. Ma-413 TRANSFERED RECEIVED JR EXAMINER J8 N Lake Superior, aff Marguette Received the section of the																									
	* SEE	CODE LI	iJ st	L									0.05		L		·			ESD	-029	502 F	FV.	10/	/79		

6/85

7.D.4. RETRIEVAL AND ANALYSIS

7.D.4.a. STORET WATER QUALITY DATA SYSTEM

Ambient water quality data collected by the Michigan Department of Natural Resources is stored in the U.S. Environmental Protection Agency's "STORET" computer system. STORET retrievals consist of an optional descriptive paragraph page and data pages. All pages have a brief description at the top describing the location of the sampling station. An example of the descriptive paragraph page is given in Figure 7.D.-12. Data can be obtained from STORET in several different formats. Three formats are described as follows. The first format, raw data RET, produces tabular listings with ten parameters per page. An example of the RET format is given in Figure 7.D.-13. The second format, raw data APARM, gives listings in parameter number order with fewer samples per page. Figure 7.D.-14 is an example of APARM. The third option, INV 120, produces statistical summaries for each station. An example of INV 120 is given in Figure 7.D.-15. Other formats and data analysis are available through the STORET group of the Water Quality Surveillance Section. The STORET group should be consulted for your special needs. For routine requests the user should indicate the retrieval format he/she desires on the request form (see the following section).

In the figures the STORET retrieval codes, abbreviations, and data are identified by letter code and explained as follows.

A - STORET station number
B - latitude
C - longitude
D - degree of precision associated with the indicated latitude and longitude values as follows:

- indicates one tenth (0.1) second
- indicates one (1) second
- indicates ten (10) seconds
- indicates thirty (30) seconds
- indicates one (1) minute
- E brief description of the station location
- F EPA state-county code
- G state and county name
- H major and minor basins or further location description
- I major, minor, and sub-basin codes for station
- J code for the agency responsible for the station
- K date station established in STORET
- L total depth of water at sample site, if available -- otherwise 0000 or 999 is indicated



101% CCF1104

- PINE RIVER (STANDISH) AT STATE ROAD SRIEGE, ARLAG CHENTY, TIBA,

R5E, STANDISH TOWNSHIP.

THIS STATION IS LOCATED MEAN THE MOUTH OF THE PANE RIVER OF

STANDISH. WATER CURLITY AND RIVER LORDINGS ENTERING LUCE ADADA AND DETERMINED AT THIS STATION.

STATISTICAL MEAN FLOW AT THIS CONTINUE S3 CFS. TOTAL RIVER DEPTH: 2 FT.

FITORET RE	ETRIEVAL DOT.	79/09/1				•					
			_ ;				2 Bor Course Million Affantie Million Affantie	1000 1000 80 - 1000 1001 1000 80 - 1000 1000 1000 1000 7 - 1000 1000 1000	.1 ARUENS .1.1	107, 820	34
/TYPA/AMI	BNTZLACE			C)	ŢĨ.	1.1967 17.114 12.114 (1 14.17)	lander fan de fan Neiser fei Sand Grei Gelffer fan San	E BURKA ENWINIYER MAI KENES	210400 00	0279	
DATE FROM TQ	TIME DEFINE OF DAY FEET	32109 DHERPHYE A UGZE	SEELI CHERCHYL A UGZI CORRECTS			00400 176 8 d	e staast Philitia- (to) RGZE IP	- 2007 Protect GR/RG PG/L P	04110 NG2: 10 NHTCE MG72	Norto Noral Tutal Norl	006003 636 - 19 74 8676
65/09/23	[[<u>86]]</u> - 6015 6015	R	·	17.0 8.6 7.0	515 116 010	21. 72 77. 433 77. 443	0,100 0,000 0,200	0.000 0.500 0.100		0.000 0.000 0.800	0,8.3 6.30 7.460
<u>27704705</u> [7 <u>3709730</u>]	0002 09 00 0000 09 00 0010			0.0 15.0 15.0	5. 5. 7. 5. 7.	4.27 4.20 5.10 9.10	0.000	0.000 0.010K	ðu stulik	0.020	0,200 s
V	69 66 6626 69 66 6636 69 66 6640 69 66 6655			10.0 11.0 7.0 5.0		n an an an Calain San An Calain An An San an Calain An		0.0103		01010	Qe izv
Y	09 60 6040 69 60 6076 02 60 6075 09 60 6688			3.0 5.0 4.5 4.5	1.0 0.8 0.5	61,90 01,20 81,20 84,30	0.120	0.110	0.0200	0.390	0.376
CP(S)-01 74/09/30	07 07 07 01	12.8		1 X D			0.014			ð 11 12	
72704723	17 62 0600 17 62 0620 17 64 0676 17 66			7.0 7.0 4.5	10.0 10.0 12.9	>++09 >+00 2+00	0.022 0.022 0.039	01001 01001 01001	an an an an an an an an an an an an an a	$0.002 \\ 0.002$	0,0000 0,5400
CP(S)-01 79704723	17 07	5.1									

ξ.,

FIGURE 7.D.-13, Example of the STORET Raw Data RET Format

6/85

·

		/	FI GURI	E 7.D.14, H	Example of	the STORE	T Raw Dat	a APARM F	ormat	
STORET	DATE: 72/	09/20								
25024	2	ncuo545.	1	······						
42 47	30.0 053	49 58.0 4			0					
LOBICL	L LARE-WE	ST BASIN;	ARGENTINE T	WP, SEC SS	and the second se					
25049	GENESCE C	0., MI								
HAU LA	EIN: LAKE	HURGN	210400							
HIN BE	ISIN: SAGI	NAN RIVER	0.	279		M G				
2114161	ł	021114	02							
		DEPTH	78			1				
/TYPA/	AMBNTZLAK	E				1				
				/						
T	5175 Y (51 - 1.7)			<u>E </u>	31 742673	- চলা চাই সন্থৰ	i tan kitu kata	- yes yes in some	70000000	74/2012/0
1	NITIAL DE	iste Sente distance de la composition de la composition de la composition de la composition de la composition de la	an an an a sua	hoooloo	<u>9</u> 74792232 78 6065777	- Prezenade Binara	1 11204120	an an trainn an trainn Ar Ar An Ar An Ar An Ar An Ar An Ar An Ar An Ar An Ar An Ar An Ar An Ar An Ar An Ar An Ar	n na standar ann an sa	- 7 7 8 7 7 4 - 1 77 C.A
1	181111961 - 14 18651 - 160257	- ner ber unfe	.04.1.0h	0,00,00		174709720	1,00,000	· 1/06 000		79/64/2
I-	THATE NATE	- - 60.00000000 - 600	1 6 6 6 6 6 6 6		i	10901 01	i			1757 01
г с	11461. (1166 19.00/07/07/07/07	-ранноса на 10 темперта	- 694000666 TTGENDAL 51 1			10P				. 2-S
	HATER	TEMP	EFIST	a 17	2.57	L	1 (4.0)	7.5	4.15	
Conta d	MATER.	THE M	E ARM	AG 1	ALL R		57.2	46. 6	43.0	
00011	TERS	TESTMIC	BACH ETH	·2 8						
1007Q	TRAJES	RECORT	Tables				72			
0080	Côi GR	PT-CO	6:115	15						
30025	CNEUCTVY	AT 250	MICROMHO	410						
00200	$D\bar{D}$		PIG71.	Ö.7	0.2		12.3	12.8	12.9	
00301	D 0	SATUR	PERCENT	5.2	2.0		118.3	107.6	14513	
00400	PiH		SU	8.30	3.30		8.80	9,00	OL: N	
00410	T ALK	CACOS	1567L	177			161	163	<u> </u>	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~
QQÇQQ	RESTINCT	TOTAL	25074L	lic t						/
oondo.	RESIDUE	TOT NELT	15-37 L	E.					/	
00665	ORG N	11	1467 L	0.370			0.446 0	0.570 0	<u>U</u>	
oodd o	iaH3~N	TOTAL	2406.4L	C. 420			0.04	0.002	0.001 8 (87	
00630	NG26/NG3	N-TOTAL	1867L	0.020 K	17		0.0.0	0.004	0.003	
00545	PHOS-TOT		MG/L P	0.120			0.016	0.022	0.018	
00900	TOT BARU	CARUS	FREE L							
00216	CALCIUM	UAHIUI Momor	おいりど (二) オン・アイ							
00727 - 2700700	TENSION -	- 1909 1991 - NA 19107	1957 E. 1177 A	111.9 General						
いいごうび	SUDIUN DISCINS	NHA DOL DOT	1 PHZ L. PACT A.	2.00						
unn an Castance	n noostan Instruction Sig	- 10 1 00 1 (7)	「「「」」	15						
0094A	SHEEKTE.	564-0195	110/1	24.0						
00258	SHIDGE	UNE REAT	MOZE SI	4. 00						
51045	18954	FE, TOT	1467E	$1 \ge 0$						
02269	CHERRYL	À	EG/E			$1 \ge 1 \le 1$				$\odot.1$
76504	0193 306	S.44	1670.	253						
76567	PHOS-T	<u>ortho</u>	PIG T. P	0.120			0.001	0.001	Q.GO.	
1	1		ý							
	1									
Ś	÷		L L							
~	•		0							

3

- M archive classification
- N type code
- 0 river index information
- P date of data retrieval
- Q on data pages -- repeat of A through M above
- R depth at which sample was taken -- blank or zero indicates just below surface; on fish tissue samples indicates fish species code
- S STORET parameter number
- T parameter name -- may include analytical method or chemical unit of expression
- U units in which values are expressed
- V date and time the sample was collected (time may not appear; time for tissue samples indicates tissue analysed; date is not repeated for multiple samples in the same day)
- W parameter value
- X letter codes follow some of the parameter values and indicate the following:
 - A Value reported is mean of two or more determinations.
 - B Results based upon colony counts outside the acceptable range.
 - C Calculated.
 - D Indicates field measurement.
 - E Indicates extra samples taken at composite stations.
 - F In the case of species, F indicates female sex.
 - G Value reported is maximum of two or more determinations.
 - H Value based on field kit determination; results may not be accurate.
 - J Estimated value; value may not be accurate.
 - K Actual value is less than the value given.
 - L Actual value is greater than the value given.
 - M Presence of material verified but not quantified. In the case of temperature or oxygen reduction potential, M indicates a negative value. In the case of species, M indicates male sex.
 - N Presumptive evidence of material.
 - 0 Sampled, but analysis lost.
 - S Laboratory test.
 - T Value reported is less than criteria of detection.
 - U Indicates material was analyzed for but not detected. In the case of species, U indicates undetermined sex.
 - W Value observed is less than lowest value reportable under "T" code.
- Y composite samples dates and times

CP(T) - 12 indicates a composite by time, consisting of 12 portions. The beginning and end date and time of sample collection are also given. CP(S) - 12 indicates a space composite of 12 samples.
FIGURE 7.D1	5. Example	of the ST()RET Statis	tical Su	ummary IN	V 120 Fo	rmat	Q 	,	
STORET DUTE 797	09729							- -		
The second second second second second second second second second second second second second second second s					,	محمد منصوبي المهر معان أما التي المان	· · · ·	79.011		
					× 1	5 42 COLO	033-292	·		
						diletta and	al-Galla e.	Sinta Abb	ionina I	ur, sze csj.
r				-,	14	.042 601FS	NEE COLL P	11		
				4		ಂತ <u>ಪ್ರಾ</u> ರ್ಥಿಕರ್ ಸ್ವಾಹಿಸಿಗಳು	4 fills and	81 - 21C	4,014	Sec. 9.
					10-	국 공간원합니다.	SHOIN W F	(1V=:(0414)
ZTYPAZAMBNTZLAKE	-			1	173	PD R.Fr.	5			
				i	· · · ·	are the state of	967 - 1775 A. 1967 - 1975 A.			1
				i						
DARKNETEG			L.B. P. P. COD	in a start and a start and a start a start a start a start a start a start a start a start a start a start a st				Professional Ref.	1. V. V.	
COOTO LOTER	710140	CONT	L <u>ochiech</u>	<u> </u>	<u></u>	<u></u>	17	11 · · · · · · · · · · · · · · · · · ·		
00000 WATEN 00074 TUER	16115 1012 0 0 0 0 0 0 0 0	10010 10001 5001	17	1 9500	1991 - 1992 - 1992 - 1993 -	- 1990-1997 - 1992-197	7.20000	4.140.150		5 797077 <u>10</u> 5 79707700
00070 TRANS	SECONT	TRUETS	 		1.201.102	43.75.5	156.650	55.0000	- 65 7 - 27 - 33	5 7970-723
	ET-CG	00173		143,000.4	6.36050	2.36676	15.0000	10,0000	- 7 17 Percia	75 097 10
00025 ENDLOTVY	AT 250	M LOUGHHO	- -	572.550	1757.50	41.2803	420,040	- S11, 500	1212-11276	111109/00
60.300 BB		ho/L	16	3.03700	25.2484	6132309	12.50.0	10000 (NGC)	100002 30	3 1177047113
00335 000	LOWLEVEL	hG/L	з	16.3667	16.3382	4.04152	21.00 8	13,0000	- 21 / 27 CF	1.60109708
60400 PH		SU	17	8.44117	1206263	.50-323	9.200CO	7,50000	65709702	. 25764723
00410 T ALE	CACOS	MG7L	9	172.778	1232.20	65.524S	250.000	104.000	- ASV - 9723) 72704723
OOSOG RESIDUE	TUTAL	1407L	3	237.667	550. SZS	23.5027	261.000	314,000	7.7 6 2. 2.3	1 747 09750
OOFRO RESIDUE	TOT NELT	NG7L	t.	3.0000/	10.00.07	_≎ . (∛ 147)	ရ ရေပါက္က မ		- 68 / 67 / R.	a da presidente de la compañía de la compañía de la compañía de la compañía de la compañía de la compañía de la
QODDS RESIDE	VOL MELT	N#374_	2	.000360	, 04. s. 20	100000	,00000		- 김희 사람은 가슴이	e southed filed -
00605 URG N	N	NG/L	2			1400-0		(دريال و روي <u>،</u>		S. Alexandri Carriera
Q0510 NH3~N	THE	19637E.	9	14 (Cont) 1 (Cont)		a 1997un 1944 / Normanie Inc. S		a Graffaran (- 60797734 - 60797677	
OUSLO NUSTR	TUTAL N. KOTAL	11071. MC /I		.037500	- 10030410 	10000 VI2	-10000000 555555		- Maria I. S. C. and - Maria - Control	- 37794793 - - 76 / 1799 -
00030 PULONGS 664 -5 986032007	N-TOTHE		0	- 1911 AUA HOL - AAA D 200	1999 - 1999 - 1999 - 1999 1997 - 1997 - 1997	200208 2000208		1000000	- 不能不能に 不能を) 70704720 > 70704709
00200 1100 111	CACOB	- HG71	7	1831.57	970	• • • • • • • • • • • • • • • • • • •	710.000	1010000	- 440 - 175 - 126 - 71 - 165 - 175	(10709740) (10709740)
60716 EALE108	CA-TOT	no/L	3	4-1-6657	4513332	6.00700	52,000	27.0001	7.708725	1 74767730
60227 N6HSI04	MG, TOT	Piů/L	S	21,0000	,000000	.000000	21.00.00	21.0000	74/09/00	14.202720
00929 SCDIUM	NA, TOT	11674.	3	9.50606	.003340	.009498	9.60000	S. 50000	14.102736	173739730
00087 PTSSIUM	К,ТОТ	相位之间	. 3	1.65687	.173335	.414355	2,00790	1.20000	74708768	174709730
00940 CHLORIDE	CL.	NG/L	3	17.6457	.335984	.577914	10,0000	17.0000	742097620	25/09/30
00245 SULFATE	S04-14SS	E67L	3	23.3333	1.350/4	15405	24,0000	22.0000	7.202730	2 747997 80
00958 SILICATE	UNE REAT	867L SI	3	<u></u>	1.56000	1.24970	4,00000	1.40000	74705730	/ 74709700
01045 IR04	FE TOT	U571	3	20.0000	1800.00	್ರಾ ಕಿಂದರೆ	1101 890	-89,96693	- 2 - 1 2 5 7 3 6	74,402,750
	LLVLN	SEVENDAY	3	اليكنية ميكن المركزية الم المركز المركز المركزية الم	ا في اليادية الي الي الي الي الي الد الدين ال	ارکانی، پارتان کی ہے۔ مراجع ارتباط کی ج	الي المراجع وي المراجع وي المراجع المراجع المراجع المراجع المراجع المراجع المراجع المراجع المراجع المراجع الم المراجع المراجع	i desdete Socia	ليتربين كالمحافظ كم المراجع ال	- 4 Q∀7123 - ***
BEEDZ CHERCHYL Robot Fice Cou	- Fi - E 1 9 2		4	- 81 YG K 9 - 84 9 - 84 9	17.0301	- C. 44473 - 67 - 67 - 64	an an an taon an taon an taon an taon an taon an taon an taon an taon an taon an taon an taon an taon an taon a Taon an taon ,10000	- 2 - 2 - 2 - 2 - 2 - 2 - 2 - 2 - 2 - 2	- 1779 - 1720) 	
[20507] [9405-1]	08030	ING / ST	3 10	-200-200 000000	454.373	21.0424 73660	- 999 - 2000 - 200 - 202 - 202 - 202	230,000	- アッインプアント - いちアカウアウン	- 74702700 56763700
	00000		10	•00-1000	.004700	.000221	الكالية ليراد والراقية ويتبينا الها			12.04720
		1 Í								
		1								
5		U								

1

6/85

7-47

CP(B) - 12 indicates a composite over space and time, consisting of 12 samples.
CP(S) - C indicates a space composite collected continuously.
Z - on INV 120 pages, the following appear and are defined as follows:
NUMBER - number of parameter values
MEAN - mean value for all parameter values for the period of record retrieved
VARIANCE - variance of the parameter values retrieved
STAN DEV - standard deviation of the parameter values retrieved
MAXIMUM - maximum value of all the parameter values retrieved
MINIMUM - minimum value of all the parameter values retrieved
BEG DATE - date of the last parameter value retrieved

7.D.4.b. STORET RETRIEVAL REQUESTS AND DATA ANALYSIS

STORET water quality data is available upon request from the STORET group of the Water Quality Surveillance Section, Surface Water Quality Division. Requests must include the following:

- user's name and address,
- reason for the data request,
- station number(s),
- parameter(s) or parameter group(s) desired,
- period of record in which the user is interested, and
- retrieval format desired.

Two different types of retrieval request forms are found in Figures 7.D.-16 and 7.D.-17. Figure 7.D.-16 is the request form supplied to users within the publication WATER QUALITY STATION INVENTORY. The other form is used within the Department for retrieval of specific parameters.

The STORET group reviews all requests for completeness and appropriate use of the data being requested. Any problems are clarified with the requestor. The STORET retrieval is then executed and the output verified by the STORET group. Any limitations of the data or other information relevant to the use of the data are communicated to the requestor by the STORET group.

7.E. DATA ANALYSIS GUIDELINES

Data analysis, reporting and conclusions are often based on mathematical and statistical techniques. Care should be exercised in this process to ensure that the data are properly handled. The analyst performing these operations should be familiar with the assumptions and limitations of the various techniques. Reviews and comments should be performed by qualified staff.

FIGURE 7.1. 16.STORET Retrieval Request Form From the WATER QU. _ITY STATION INVENTORY Publication

ת ת	PARAMETER : GROUPS (CHECK THOSE REQUESTED - SEE PAGE FOR DETAILS)
DATE OF REQUEST:	
	WATER FISH
REQUESTED BY	Flow Heavy Metals
	Biochemical Oxygen Demand (BOD)
	Dissolved Oxygen
NAME	Nutrients SEDIMENTS
	🗌 Solids 👘 🗍 General t Nutrients
	Chlorides Events
ADDRESS	Bacteriological Organic Contaminants
	Major Ions
	Pesticides and Organic Contaminants
CITY STATE ZIF	П РСВ'S
	Other Toxics
REPRESENTING	Radioactivity
NET NESENTING	Chlorophyll A
TELEPHONE NUMBER	PARAMETERS NOT COVERED IN ABOVE GROUPS:
PERIOD OF RECORD REQUESTED	
	STORET NUMBERS REQUESTED
FROM TO	
WHAT WILL DATA BE USED FOR P	
RETRIEVAL FORMAT DESIRED:	
RAW DATA RET FORMAT	
RAW DATA APARM FORMAT	
STATISTICAL SUMMARY INV 120 FORMAT	

Figure

FE V 05 V1 E1

STORET DATA RETRIEVAL REQUEST FORM

DATE REDUESTED:	SEBCRIPTIVE RARAGRAPH DESCRED (+ 38 N°	GATE FETFIEVED:
REQUESTED &::	PERIOD OF RECORD RECUENTED.	CLOS NUMBERS.
LATE REGULPED;	PURFOSE OF RETRIE (HL:	- 047E FECEICED;
STATION NUMBERE REQUESTED:		SEIRIEVAL FORMAT DEBIRED)
		 CO RAN DATA RES FORMAT LEINITED 10 30 FARAMETERS RETA CO RAN DATA ARARM FORMAT CO STATIENTEAL BETMARA, IN 1110 FORMAT CO STATIENTEAL BETMARA, IN 1110 FORMAT CO STATIENTEAL BETMARA FOR PORTA CO STATIENTEAL BETMARA FOR PORTA CO STATIENTEAL FORMATA

-				÷	31		••		-	- 2	•	 100	-	-	11	•	~	C 11		
		1.1	- 1	÷.,					-		х.	-			*	~	-+	-	15	•
	. en 1	-	9.3	-		-	2		2		2		-	-	1	-	÷.	-	44	

CD GENERAL RARAMSTERE CD METALS PARAMETERS CD RESILCISES & GILS CD DIMER IPGARICS CD PARIOR(NIMITA) CD RESIMENT METALS & JENERAL CD SEDIMENT GROANICS CD TISELS

FARAMETERE REQUESTED (PLEASE CIRCLE)	UMBER) OR INDICATE GROUPS ON	51347	COMER FRAMENERS HEITER MURAFER PERING,
<pre>9 00010 TEMPERATURE, WATER CENT. 9 00060 FLOW, STEEAM MEAN CAIL 9 00060 FLOW, STEEAM MEAN CAIL 9 00075 FLOW, STEEAM INSTANTANEOUS 9 00075 TURBIDITY, JOU 9 00075 SECONI, INCHES 9 00075 CONDUCTANCE, SPECIFIC 9 00075 CONTENT OF CONTENT 9 00075 CONDUCTANCE, TOT 9 00075 CONDUCTANCE, TOT, SPECIFIC 9 00075 SUIDER, TOT, SPECIFIC 9 0005 SUIDER, TOT, SPECIFIC 9 0005 SUIDER, TOT, SPECIFIC 9 0005 SUIDER, TOT, SPECIFIC 9</pre>	 3 00905 MAGNESIUM, DISS. 3 00907 SODUM, TOT. 5 00909 SODUM, TOT. 5 00909 SODUM, TOT. 5 00905 POTASSIUM, DISS. 5 00905 POTASSIUM, TOT. 5 00945 SULFATE, TOT. 6 00945 SULFATE, TOT. 6 00945 SULFATE, DISS. 5 00951 FLUGRIDE, DISS. 6 00951 FLUGRIDE, DISS. 6 00951 FLUGRIDE, TOT. 3 00951 FLUGRIDE, TOT. 3 00951 FLUGRIDE, TOT. 3 00953 SULFATE, DISS. 5 00954 SULFATE, TOT. 3 00955 SULFATE, DISS. 5 00955 SULFATE, DISS. 5 00955 SULFATE, DISS. 6 01012 SEF.LLIEM, TOT. 6 01012 SEF.LLIEM, TOT. 6 01012 SEF.LLIEM, TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01012 SUFF.M. TOT. 7 01013 SUFF.M. TOT. 7 01014 SUFF.M. TOT. 7 01014 SUFF.M. TOT. 7 01015 SUFF.M. TOT. 7 01015 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 7 01017 SUFF.M. TOT. 8 00017 SUFF.M. TOT. 9 01017 SUFF.M.	 M 01040 MOLVEDENUM. 707. M 01045 MICKEL, 0105. M 01075 MICKEL, 0105. M 01075 MICKEL, 0105. M 01075 MICKEL, 707. M 01096 MICKEL, 707. M 01097 VANADIUM, 707. M 01097 VANADIUM, 707. M 01097 MICK, 0108. M 01100 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 01120 LIPHION, 027. M 0120 LIPHION, 027. <	WEIRIC MEIRIC Frons Fronz Fron
		2010-2010-2010-2010-2010-2010-2010-2010	
2 du Ta I la Bala ann a shr	- 11111 - FYGRHEII, 11114	11	· · ·

7.E.1. FIELD AND LABORATORY REPORT DATA REVIEW

The program manager should review all reported data as soon as possible upon receipt from the lab. This review should verify the field and laboratory work to the greatest extent possible using the information the program manager has concerning the planned work, knowledge of the sampling sites, and expected data values. Any inconsistencies or errors should be discussed with field and/or laboratory staff as appropriate. Errors should be corrected on the original data sheets or noted on attached sheets. Information concerning unusual values should be noted on the data sheets.

Once the program manager has completed his review, the person collecting the samples should also review the data sheets for errors. After the reviews have been completed, the data can be certified for use in reports and/or entry into STORET.

7.E.2. FORMULAS - CONVERSION FACTORS

The following are conversion factors and formulas used frequently in data analysis and reporting. Appendix 7.E.-1, Units of Measurement - Conversion Factors and Special Tables, is a useful reference for other conversion factors.

7.E.2.a. Conversion Factors

<u>To Obtain</u>	Multiply	By
Cubic Meters/day (M ³ /d) Kilograms/day (kg/d) MGD lb/d	Million Gallons/day (MGD) pqunds/day (lb/d) M /d kg/d	3785 0.4536 0.0002642 2.205
M [°] /day	cubic feet/second (cfs)	2447
cts	M⁻/d	0.0004088

7.E.2.b. Formulas and Procedures for Data Analysis

7.E.2.b.1. Heat Load Addition

- -- Determine change in temperature: temperature of effluent (°C) - temperature of influent (°C) = Change in temperature (°C)
- -- Convert change in temperature [°]C to [°]F: change in temperature ([°]C) x 1.8 = change in temperature ([°]F)
- -- Determine number of pounds of water per day: flow (M³/day) x 1000 (kg) x 2.205 (1b) = flow (1b) $\frac{M^3}{M^3}$ kg day
- -- Determine number of pounds of water per hour: $flow (1b) \div 24 = flow (1b)$ day hr

the first non-zero digit on the left of the decimal point, or beginning with the first non-zero digit after the decimal point if there is no <u>non-zero</u> digit to the left of the decimal point, and ending, in both cases with the last digit to the right. Final zeros in a whole number may or may not be significant. Final zeros in a whole number may or may not be significant. Final zeros after a decimal point are always meant to be significant digits. Examples

Figure	Significant Digits
8	1
80	1 or 2
800	1, 2, or 3
800.	3
80.	2
8.	1
8.0	2
80.0	3
800.0	4
0.8 or .8	1
0.08 or .08	1
0.080 or .080	2
0.008 or .008	1
8.080	4

7.E.3.b. Multiplying Significant Digits

When multiplying figures, the answer may not have more significant digits than the multiplier with the smallest number of significant digits.

Examples

 $.08 \times 80 = 6 \text{ not } 6.4 \qquad .04 \times .08 = .003 \text{ not } .0032 \\ (1) \quad (1) \quad (1) \qquad (1) \qquad (1) \qquad (1)$

7.E.3.c. Flow Data

Flow cannot be reported to more than three significant figures. In no case will discharger flow rates be reported in quantities less than 1 m /day.

7.E.3.d. Rounding-off Numbers

1. If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged.

i.e., 23.443 is rounded off to 23.44

2. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1.

i.e., 23.446 is rounded off to 23.45

3. If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number.

i.e., 23.435 rounds off to 23.44 23.425 rounds off to 23.42

7.F. RECORD MAINTENANCE FOR QUALITY ASSURANCE

All field and laboratory notes, data sheets, and other pertinent records should be maintained <u>indefinitely</u>. In some cases, specific statutes require that specific records be kept. In other cases, they may be needed for litigation, or for historical purposes. The sheets for data stored in STORET are referenced periodically for verification and/or clarification of the data. These sheets frequently reflect more information than can be stored in STORET. Other records which are not needed should be sent to the State Records Center, Department of Management and Budget. They should not be destroyed. Appendix 7.E.-1.



UNITS OF MEASUREMENT Conversion Factors and Special Tables

October, 1972

REPRINTED FROM

Units of Weight and Measure International (Metric) and U.S. Customary

NBS Miscellaneous Publication 286 May, 1967

WITH THE COURTESY OF : TECHNICAL INFORMATION OFFICE NATIONAL ENVIRONMENTAL RESEARCH CENTER, CINCINNATI U.S. ENVIRONMENTAL PROTECTION AGENCY

Units of Weight and Measure

International (Metric) and U.S. Customary

L. J. Chisholm

The primary purpose of this publication is to make available the most often needed weights and measures conversion tables—conversions between the U.S. Customary System and International (Metric. System. A secondary purpose is to present a brief historical outline of the International (Metric) System following it from its country of origin, France, through its progress in the United States.

Key Words: Conversion tables, International System (SI), Metric System, U. S. Customary System, weights and measures, weights and measures abbreviations, weights and measures systems, weights and measures units.

Introduction

Two systems of weights and measures exist side by side in the United States today, with roughly equal but separate legislative sanction: the U.S. Customary System and the International (Metric) System. Throughout U.S. history, the Customary System (inherited from, but now different from, the British Imperial System) has been, as its name implies, customarily used; a plethora of Federal and State legislation has given it, through implication, standing as our primary weights and measures system. However, the Metric System (incorporated in the scientists' new SI or Système International d'Unités) is the only system that has ever received specific legislative sanction by Congress. The "Law of 1866" reads:.

It shall be lawful throughout the United States of America to employ the weights and measures of the metric system; and no contract or dealing, or pleading in any court, shall be deemed invalid or liable to objection because the weights or measures expressed or referred to therein are weights or measures of the metric system.¹

Over the last 100 years, the Metric System has seen slow, steadily increasing use in the United States and, today, is of importance nearly equal to the Customary System.

The International System *

* For up-to-date information on the international metric system, see current edition of The International System of Units (SI), Editors: Chester Page and Paul Vigoureux (NBS Special Publication 330). For sale by Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402. Price 30 cents. For NBS policy on the usage of SI, see NBS Technical News Bulletin Vol. 55 No. 1, pp. 18-20, January 1971.

Act of 28 July 1866 (14 Stat. 339)-An Act to authorize the use of the Metric System of Weights and Measures.

(Reprint includes only those items asterisked)	P
*INTRODUCTION	· · · · · · · · · · · · · · · · · · ·
THE INTERNATIONAL SYSTEM	
Prefixes	
HISTORICAL OUTLINE	
France The United States	
WEIGHTS AND MEASURES IN THE WORLD'S INDEPENDENT STAT	ES
Metric Nonmetric	· · · · · · · · · · · · ·
IMPORTANT DATES IN U. S. METRIC HISTORY	
SELECTED BIBLIOGRAPHY	
*DEFINITIONS	
* Definitions of Units	
*SPELLING AND SYMBOLS FOR UNITS	
* Some Units and Their Symbols	
* UNITS OF MEASUREMENT—CONVERSION FACTORS	
*Length *Mass *Capacity, or Volume	
* CDECIAL TADIES	
*Equivalents of Decimal and Binary Fractions of an Inch in Millimeters *International Nautical Miles and Kilometers	
UNITS OF MEASUREMENT—TABLES OF EQUIVALENTS	
Length Mass Capacity, or Volume Area	
Chisholm, L.J., Units of Weight and Measure. Interr (Metric) and U.S. Customary. (NBS Miscellaneous Pub 286) For cale by Superintendent of Documents. U.S.	national plicatio Governm

Six units have been adopted to serve as the base for the International System: *

Length	meter
Mass	kilogram
Time	second
Electric current	ampere
Thermodynamic temperature	kelvin
Light intensity	candela

Some of the other more frequently used units of the SI and their symbols and, where applicable, their derivations are listed below.

SUPPLEMENTARY UNITS							
Quantity	Unit	Symbol	Derivation				
Plane angle	radian	rad					
Solid angle	steradian	sr_					
	DERIVED UNITS						
1.000	aquara matar						
Volumo	square meter	m ¹					
Frequences	borts	u.	(a-1)				
Prequency	hertz	liz	(5)				
Velocity	motor nor second	kg/m ²					
A moule a velocitar	ndian par second	m/s					
Angular velocity	radian per second	rau/s					
Acceleration	meter per second squared	$111/S^2$					
Angular acceleration	radian per second squared	rau/s ²	(l_{1}, m, l_{2})				
r orce	newton	N/m^2	(kg ·m/s·)				
Pressure King and the second state	newton per square meter	N/m^2					
Rinematic viscosity	square meter per second	M^{-}/S					
Dynamic viscosity	newton-second per square meter	T S/ III-	(\mathbf{N},\mathbf{m})				
work, energy, quantity of neat	joule	\mathbf{J}	$(\mathbf{I} \cdot \mathbf{m})$				
Power	wall	C	(J/S)				
Electric charge		v	$(\mathbf{H}^{*}\mathbf{S})$				
voltage, potential difference,	Voit	v	(\mathbf{W}/\mathbf{A})				
Electromotive force	welt non motor	V/m					
Electric field strength	ohm	•/m	(\mathbf{V}/\mathbf{A})				
Electric resistance	forad	л Т	(\mathbf{V}/\mathbf{A}) $(\mathbf{A},\mathbf{z}/\mathbf{V})$				
Magnetic dur	weber	Wh	$(\mathbf{X} \cdot \mathbf{s})$				
Inductoria	henme	ਸ	$(\mathbf{V},\mathbf{s}/\mathbf{A})$				
Mognetie Aux density	tosla		$(\mathbf{W}\mathbf{h}/\mathbf{m}^2)$				
Magnetic flux density	ampere per meter	1 1/m	(110/111)				
Magnetic neid Strength	ampere per meter	11/ III					
Flux of light	lumen	l m	(cd.sr)				
Luminan <i>a</i> a	andela per square mater	d/m^2	(cu st)				
Illumination	lux	lv	(lm/m^2)				
mummation	IUX	1.4					

* Recent (1971) addition of the mole as the unit for amount of substance brings the total to seven units. See asterisked footnote on page 1.

Definitions

In its original conception, the meter was the fundamental unit of the Metric System, and all units of length and capacity were to be derived directly from the meter which was intended to be equal to one ten-millionth of the earth's quadrant. Furthermore, it was originally planned that the unit of mass, the kilogram, should be identical with the mass of a cubic decimeter of water at its maximum density. The units of length and mass are now defined independently of these conceptions.

In October 1960 the Eleventh General (International) Conference on Weights and Measures redefined the meter as equal to 1 650 763.73 wavelengths of the orange-red radiation in vacuum of krypton 86 corresponding to the unperturbed transition between the $2p_{10}$ and $5d_{4}$ levels.

The kilogram is independently defined as the mass of a particular platinum-iridium standard, the International Prototype Kilogram, which is kept at the International Bureau of Weights and Measures in Sèvres, France.

The liter has been defined, since October 1964, as being equal to a cubic decimeter. The meter is thus a unit on which is based all metric standards and measurements of length, area, and volume.

Definitions of Units

Length

A meter is a unit of length equal to 1 650 763.73 wavelengths in a vacuum of the orangered radiation of krypton 86.

A yard is a unit of length equal to 0.914 4 meter.

Mass

A kilogram is a unit of mass equal to the mass of the International Prototype Kilogram.

An avoirdupois pound is a unit of mass equal to 0.453 592 37 kilogram.

Capacity, or Volume

A cubic meter is a unit of volume equal to a cube the edges of which are 1 meter.

A liter is a unit of volume equal to a cubic decimeter.

A cubic yard is a unit of volume equal to a cube the edges of which are 1 yard.

A gallon is a unit of volume equal to 231 cubic inches. It is used for measuring liquids only.

A bushel is a unit of volume equal to 2 150.42 cubic inches. It is used for measuring dry commodities only.

Area

A square meter is a unit of area equal to the area of a square the sides of which are 1 meter.

A square yard is a unit of area equal to the area of a square the sides of which are 1 yard.

Spelling and Symbols for Units

The spelling of the names of units as adopted by the National Bureau of Standards is that given in the list below. The spelling of the metric units is in accordance with that given in the law of July 28, 1866, legalizing the Metric System in the United States.

Following the name of each unit in the list below is given the symbol that the Bureau has adopted. Attention is particularly called to the following principles:

1. No period is used with symbols for units. Whenever "in" for inch might be confused with the preposition "in", "inch" should be spelled out.

2. The exponents "2" and "3" are used to signify "square" and "cubic," respectively, instead of the symbols "sq" or "cu," which are, however, frequently used in technical literature for the U. S. Customary units.

3. The same symbol is used for both singular and plural.

Unit	Symbol	Unit	Symbol	Unit	Symbol
acre	acre	fathom	fath	millimeter	mm
are	8,	foot	ft	minim	minim
harrel	bbl	furlong -	furlong	ounce	0 2
board foot	fbm	gallon	gal	ounce, avoirdupois	oz avdp
bushel	bu	grain	grain	ounce, liquid	liq oz
carat	с	gram	g	ounce, troy	oz tr
Celsius, degree	°C	hectare	ha	peck	peck
centare	Ca	hectogram	hg	pennyweight	dwt
centigram	cg	hectoliter	hÌ	pint, liquid	liq pt
centiliter	el	hectometer	hm	pound	lb
centimeter	cm	hogshead	hhd	pound, avoirdupois	lb avdp
chain	ch	hundredweight	cwt	pound, troy	lb tr
cubic centimeter	cm ³	inch	in	quart, liquid	lig gt
cubic decimeter	dm³	International		rod	rod
cubic dekameter	dam ³	Nautical Mile	INM	second	9
cubic foot	ft ³	kelvin	К	square centimeter	cm²
cubic hectometer	hms	kilogram	kø	square decimeter	dm ²
cubic inch	in ³	kiloliter	kÎ	souare dekameter	dam ²
cubic kilometer	km ³	kilometer	km	square foot	ft ²
cubic meter	m ³	link	link	square hectometer	hm²
cubic mile	mi ³	liquid	lia	square inch	in ²
cubic millimeter	mm³	liter	liter	square kilometer	km²
cubic vard	vd ³	meter	m	souare meter	m²
decigram	der	microgram	ug	souare mile	mi²
deciliter	dÎ	microinch	μin	square millimeter	mm²
decimeter	dm	microliter	щl	square vard	vd²
dekagram	dag		- -	stere	stere
dekaliter	dal	mile	mi	ton, long	long ton
dekameter	dam	milligram	mg	ton, metric	t
dram avoirdupois	dr avdn	milliliter	ml	ton, short	short tor
dram, arondapous	ar arap			vard	vd

Some Units and Their Symbols

Units of Measurement-Conversion Factors*

To Convert from Centimeters	L .	To Convert fro Meters	m
To	Multiply by	To	Multiply by
Inches	0.393 700 8	Inches	39.370 08
Yards	. 0.032 808 40 . 0.010 936 13	Feet Yards	3.280 840 1.093 613
Meters	0.01	Miles	0.000 621 37
		Millimeters	1 000
		Centimeters	100
		To Convert fro	m

Units of Length

	ı	To Con F	vert from eet
To Convert from Inches		То	Multiply by
To Mu	ltiply by	Inches	
Feet	33 333 33	Miles	0.000 189 39
Yards	27 777 78	Centimeters	
Centimeters	L III	Meters	0.304 8
Meters	25 4	Kilometers	0.000 304 8

* All boldface figures are exact; the others generally are given to seven significant figures.

In using conversion factors, it is possible to perform division as well as the multiplication process shown here. Division may be particularly advantageous where more than the significant figures published here are required. Division may be performed in lieu of multiplication by using the reciprocal of any indicated multiplier as divisor. For example, to convert from centimeters to inches by division, refer to the table headed "To Convert from Inches" and use the factor listed at "centimeters" (2.54) as divisor.

To C	onvert from Yards
Το	Multiply by
Inches	
Feet	3
Miles	0.000 568 18
Centimeters	
Meters	0.914 4

To	Convert from Miles		
To	2	M	ultiply by
Inches		5	360
Feet		5	280
Yards	1	l	760
Centimeters	160)	934.4
Meters		Ł	609.344
Kilometers			1.609 344

Units of Mass

To Convert from Grams			
Το	Multiply by		
Grains	15.432 36		
Avoirdupois Drams. 0.564.383 4			
Avoirdupois Ounces	0.035 273 96		
Troy Ounces	0.032 150 75		
Troy Pounds	0.002 679 23		
Avoirdupois Pounds	0.002 204 62		
Milligrams	1 000		
Kilograms.	0.001		

To Convert from Metric Tons		
То	Multiply by	
Avoirdupois Pounds Short Hundredweights Short Tons Long Tons Kilograms	2 204.623 22.046 23 1.102 311 3 0.984 206 5 1 000	

To Convert from Grains				
Τυ	·Mu	ltiply	y by	
Avoirdupois Drams	0.036	571	43	
Avoirdupois Ounces	0.002	285	71	
Troy Ounces	0.002	083	33	
Troy Pounds	0.000	173	61	
Avoirdupois Pounds	0.000	142	8 6	
Milligrams	64.798	91		
Grams	0.064	798	91	
Kilograms	0.000	064	798	91

To Convert from Avoirdupois Pounds To Multiply by	
Grains	7 000
Avoirdupois Drams	256
Avoirdupois Ounces	16
Troy Ounces	14.583 33
Troy Pounds	1.215 278
Grams	453.592 37
Kilograms	0.453 592 37
Short Hundredweights	0.01
Short Tons	0.000 5
Long Tons	0.000 446 428 6
Metric Tons	0.000 453 592 37

To Convert from Kilograms			
To	Multiply by		
Grains	15 432.36		
Avoirdupois Drams	564.383 4		
Avoirdupois Ounces	35.273 96		
Troy Ounces	32.150 75		
Troy Pounds	2.679 229		
Avoirdupois Pounds	2.204 623		
Grams	1 000		
Short Hundredweights	0.022 046 23		
Short Tons	0.001 102 31		
Long Tons	0.000 984 2		
Metric Tons	0.001		

To Convert from Avoirdupois Ounces		
То	Multiply by	
Grains	437.5	
Avoirdupois Drams	16	
Troy Ounces	0.911 458 3	
Troy Pounds	0.075 954 86	
Avoirdupois Pounds	0.062 5	
Grams	28.349 523 125	
Kilograms	0.028 349 523 125	

.

To Convert from Short Hundredweights		
To Multiply by		
Avoirdupois Pounds	100	
Short Tons	0.05	
Long Tons	0.044 642 86	
Kilograms	45.359 237	
Metric Tons	0.045 359 237	

To Convert from Short Tons		
Το	Multiply by	
Avoirdupois Pounds	2 000	
Short Hundredweights	20	
Long Tons	0.892 857 1	L
Kilograms	907.184 74	
Metric Tons	0.907 184 7	74

To Convert from Long Tons		
То	Multiply by	
Avoirdupois Ounces Avoirdupois Pounds Short Hundredweights Short Tons Kilograms Metric Tons	35 840 2 240 22.4 1.12 1 016.046 908 8 1.016 046 908 8	

To Convert from Troy Ounces			
То	Multip	oly b	у
Grains	480		
Avoirdupois Drams	17.554	29	
Avoirdupois Ounces	1.097	143	
Troy Pounds	0.083	333	3
Avoirdupois Pounds	0.068	571	43
Grams	31.103	476	8

To Convert from Troy Pounds				
То	Multiply by			
Grains	760			
Avoirdupois Drams	210.651 4			
Avoirdupois Ounces	13.165 71			
Troy Ounces	12			
Avoirdupois Pounds	0.822 857 1			
Grams	373.241 721 6			

Units of Capacity, or Volume, Liquid Measure

To Convert from Milliliters			
Το	Multi	ply b	рy
Minims	16.230	73	
Liquid Ounces	0.033	814	02
Gills	0.008	453	5
Liquid Pints	0.002	113	4
Liquid Quarts	0.001	056	7
Gailons	0.000	264	17
Cubic Inches	0.061	023	74
Liters	0.001		

To Convert from Cubic Meters			
To	Multiply by		
Gallons Cubic Inches Cubic Feet	264.172 05 61 023.74 35.314 67 1 000		
Cubic Yards	1.307 950 6		

To Convert from Liters			
To	Multiply by		
Liquid Ounces	33.814 02		
Gills	8.453 506		
Liquid Pints	2.113 376		
Liquid Quarts	1.056 688		
Gallons	0.264 172 05		
Cubic Inches	61.023 74		
Cubic Feet	0.035 314 67		
Milliliters	1 000		
Cubic Meters	0.001		
Cubic Yards	0.001 307 95		

To Convert from Minims			
To	Mult	iply	by
Liquid Ounces Gills Cubic Inches Milliliters	0.002 0.000 0.003 0.061	083 520 759 611	33 83 77 52

To Convert from Gills		
To	Multiply by	
Minims	1 920	
Liquid Ounces	- 4	
Liquid Pints	_ 0.25	
Liquid Quarts	. 0.125	
Gallons	0.031 25	
Cubic Inches	7.218 75	
Cubic Feet	0.004 177 517	
Milliliters	118.294 118 25	
Liters.	0.118 294 118 25	

To Convert from Liquid Pints		
То	Multiply	by
Minims	7 680	
Liquid Ounces	16	
Gills	4	
Liquid Quarts	0.5	
Gallons	0.125	
Cubic Inches	28.875	
Cubic Feet	0.016 7	10 07
Milliliters	473.176 43	73
Liters	0.473 12	76 473

To Convert from Liquid Ounces To	Multir	oly b	y
Minims 4 Gills 1 Liquid Pints 1 Liquid Quarts 1 Gallons 1	180 0.25 0.062 0.031 0.007	5 25 812	5
Cubic Inches Cubic Feet Milliliters Liters	1.804 0.001 29.573 0.029	687 044 53 573	5 38 53

To Conve Cubic 1 To	rt from Feet Multipl	y by	
Liquid Ounces Gills Liquid Pints Liquid Quarts Gallons	957.506 5 239.376 6 59.844 16 29.922 08 7.480 51	9	
Cubic Inches 1 Liters Cubic Meters Cubic Yards	728 28.316 84 0.028 31 0.037 03	6 592 6 846 7 04	592

To Convert from Cubic Inches To Multiply by				
Minime Liquid Ounces Gills Liquid Pints Liquid Quarts Gallons	265.974 0.554 0.138 0.034 0.017 0.004	0 112 528 632 316 329	6 1 03 02 0	
Cubic Feet Milliliters Liters Cubic Meters Cubic Yards	0.000 16.387 0.016 0.000 0.000	578 064 387 016 021	7 064 387 43	064

To Convert from Cubic Yards			
То	Multiply by		
Gallons Cubic Inches Cubic Feet Liters Cubic Meters	201.974 0 46 656 27 764.554 857 984 0.764 554 857 984		

To Convert from Liquid Quarts To Multiply by	To Convert from Gallons To Multiply by	
Minims 15 360 Liquid Ounces 32 Gills 8 Liquid Pints 2 Gallons 0.25 Cubic Inches 57 75	Minims 61 440 Liquid Ounces 128 Gills 32 Liquid Pints 8 Liquid Quarts 4 Cubic Inches 231	
Cubic Feet 0.033 420 14 Milliliters 946.352 946 Liters 0.946 352 946	Cubic Feet 0.133 680 6 Milliliters 3 785.411 784 Liters 3.785 411 784 Cubic Meters 0.003 785 411 78 Cubic Yards 0.004 951 13	

Units of Capacity, or Volume, Dry Measure

.

To Conve Liter	rt from
То	Multiply by
Dry Pints	1.816 166
Dry Quarts	0.908 082 98
Pecks	0.113 510 4
Bushels	0.028 377 59
Dekaliters	0.1
Derauters	

To Convert from Dekaliters	
То	Multiply by
Dry Pints	18.161 66
Dry Quarts	9.080 829 8
Pecks	1.135 104
Bushels	0.283 775 9
Cubic Inches	610.237 4
Cubic Feet	0.353 146 7
Liters	10

To Convert from Cubic Meters		
To	Multiply by	
Pecks	113.510 4	
Bushels	28.377 59	

To Convert from Dry Pints			
То	Multi	ply t	у
Dry Quarts	0.5		
Pecks	0.062	5	
Bushels	0.015	625	
Cubic Inches	33.600	312	5
Cubic Feet	0.019	444	63
Liters	0.550	610	47
Dekaliters	0.055	061	05

Dry Qua	irts
Το	Multiply by
Dry Pints	2
Pecks	0.125
Bushels	0.031 25
Cubic Inches	67.200 625
Cubic Feet	0.038 889 25
Liters	1.101 221
Dekaliters	0.110 122 1

To Convert from Pecks			
To	Multiply by		
Dry Pints	16		
Dry Quarts	8		
Bushels	0.25		
Cubic Inches	537.605		
Cubic Feet	0.311 114		
Liters	8.809 767 5		
Dekaliters	0.880 976 75	5	
Cubic Meters	0.008 809 77	7	
Cubic Yards	0.011 522 74	ł	

To Convert from Bushels		
То	Multipl	y by
Dry Pints	64	
Dry Quarts	32	
Pecks	4	
Cubic Inches	2 150.42	
Cubic Feet	1.244	456
Liters	35.239	07
Dekaliters	3.523	907
Cubic Meters	0.035	239 07
Cubic Yards	0.046	090 96

To Convert from Cubic Feet		
To	Multiply by	
Drv Pints	51.428 09	
Dry Quarts	25.714 05	
Pecks	3.214 256	
Bushels	0.803 563 95	

To Convert from Cubic Inches			
Το	Multiply by		
Dry Pints Dry Quarts Pecks Bushels	0.029 761 6 0.014 880 8 0.001 860 10 0.000 465 025		

	To Convert from Cubic Yards
Το	Multiply by
Pecks Bushels	86.784 91 21.696 227

Units of A	lrea
------------	------

To Convert from Square Centimeters									
To Multiply by									
Square Inches	0.155	000	3						
Square Feet	0.001	076	39						
Square Yards	0.000	119	599						
Square Meters	0.000	1							

.

To Convert from Square Meters								
То	Multiply by							
Square Inches Square Feet	1 550.003 10.763 91							
Square Yards	1.195 990 0.000 247 105							
Square Centimeters Hectares	10 000 0.000 1							

To Conver Hectar	t from res
То	Multiply by
Square Feet	. 107 639.1
Square Yards	. 11 959.90
Acres	. 2.471 054
Square Miles	0.003 861 02
Square Meters	. 10 000

To Convert from Square Inches	n
То	Multiply by
Square Feet Square Yards Square Centimeters Square Meters	0.006 944 44 0.000 771 603 6.451 6 0.000 645 16

To Convert from Square Feet								
To .	Multiply by							
Square Inches	144							
Square Yards	0.111 0.000	111 022	1 957					
Square Centimeters	929.030	4						
Square Meters	0.092	903	04					

To Convert from Acres								
To Multiply by								
Square Feet 4	3 560							
Square Yards	4 840							
Square Miles	0.001 562 5							
Square Meters	4 046.856 422 4							
Hectares	0.404 685 642 24							

To Convert from Square Yards									
To Multiply by									
Square Inches	1	296			<u></u>				
Square Feet		9							
Acres		0.000	206	611	6				
Square Miles		0.000	000	322	830	6			
Square Centimeters.	8	361.273	6						
Square Meters		0.836	127	36					
Hectares		0.000	083	612	736				

To Convert from Square Miles								
To Multiply by								
Square Feet27Square Yards3Acres3Square Meters2Hectares2	878 400 097 600 640 589 988.110 336 258.998 811 033 6							

Special Tables

Length—Inches and Millimeters—Equivalents of Decimal and Binary Fractions of an Inch in Millimeters

From	1/64	to 1	Inch	
------	------	------	------	--

½'s	¥'s	8ths	16ths	32ds	64ths	Milli- meters	Decimais of an inch	Inch	!∕2's	1 4 's	8ths	16ths	32ds	64ths	Milli- meters	Decimals of an inch
			1	1	1 2 3 4	= 0.397 = .794 = 1.191 = 1.588	0.015625 .03125 .046875 .0625						17	33 34 35	= 13.097 = 13.494 = 13.891	0.515625 .53125 .546875
				3	5 6 7	= 1.984 = 2.381 = 2.778	.078125						18	37 38 30	= 14.884 = 15.081 = 15.478	.578125
		1	2	4	8	= 3.175	. 1250				5	10	20	40	= 15.475 = 15.875	.625
			3	5 6	9 10 11 12	= 3.572 = 3.969 = 4.366 = 4.762	.140625 .15625 .171875 .1875					11	21	41 42 43 44	= 16.272 = 16.669 = 17.066 = 17.462	.640625 .65625 .671875 .6875
	1	2	4	7 8	13 14 15 16	= 5.159 = 5.556 = 5.953 = 6.350	.203125 .21875 .234375 .2500			3	6	12	23 24	45 46 47 48	= 17.859 = 18.256 = 18.653 = 19.050	.703125 .71875 .734375 .75
			5	9 10	17 18 19 20	= 6.747 = 7.144 = 7.541 = 7.938	.265625 .28125 .296875 .3125					13	25	· 49 50 51	= 19.447 = 19.844 = 20.241 = 20.638	.765625 .78125 .796875
		3	6	11 12	21 22 23 24	= 8.334 = 8.731 = 9.128 = 9.525	.328125 .34375 .359375 .3750				7	14	20 27 28	53 54 55 56	= 20.033 = 21.034 = 21.431 = 21.828 = 22.925	.828125 .84375 .859375 .875
			7	13 14	25 26 27 28	= 9.922 = 10.319 = 10.716 = 11.112	.390625 .40625 .421875 .4375					15	29	57 58 59	= 22.622 $= 23.019$ $= 23.416$ $= 22.812$. 890625 . 90625 . 921875
				15	29 30 31	= 11.509 = 11.906 = 12.303	. 453125 . 46875 . 484375					10	31	61 62 63	= 23.812 = 24.209 = 24.606 = 25.003	.9375 .953125 .96875 .984375
1	2	4	8	16	32	= 12.700	.5	1	2	4	8	16	32	64	= 25.400	1.000

Int. nautical miles	Kilometers	Int. nautical miles	Kilometers	Kilometers	Int. nautical miles	Kilometers	Int. nautical miles
0		50	92 600	0	;		
1	1.852	1	04 450	0		50	26.9978
2	3 704	1	94.402		0.5400	1	27.5378
2	5 5 5 6	4	90.304	2	1.0799	2	28.0778
5	5.000	3	98.156	3	1.6199	3	28.6177
4	7.408	4	100.008	4	2.1598	4	29.1577
5	9.260	5	101.860	5	2.6998	5	29.6976
0	11.112	6	103.712	6	3.2397	6	30.2376
1	12.964	7	105.564	7	3.7797	7	30 7775
8	14.816	8	107.416	8	4.3197	8	31 3175
9	16.668	9	109.268	9	4.8596	9	31.8575
10	18.520	60	111.120	10	5,3996	60	32 3074
1	20.372	1	112.972	1	5 9395	1	22.0374
2	22.224	2	114.824	2	6 4795	2	22.9014
3	24.076	3	116.676	3	7 0104	2	33.4/73
4	25.928	4	118 528	1	7 5504	3	34.0173
_		-	110.020	*	1.5594	4	34.5572
5	27.780	5	120.380	5	8.0994	5	35 0972
6	29.632	6	122.232	6	8,6393	6	35 6371
7	31.484	7	124.084	7	9 1793	7	26 1771
8	33.336	8	125.936	8	9 7192		26 7171
9	35.188	9	127.788	9	10.2592	9	37.2570
20	37.040	70	129 640	20	10,7001	-	
1	38,892	1	131 402	20	10.7991	10	37.7970
2	40 744	2	122 244		11.3391	1	38.3369
3	12 596	2	125 106	2	11.8790	2	38.8769
4	44 448	3	135.190	3	12.4190	3	39.4168
Ŧ	17.110	+	137.048	4	12.9590	4	39.9568
5	46.300	5	138.900	5	13 4080	5	10 1069
6	48.152	6	140.752	6	14 0380	5	40.4908
7	50,004	7	142,604	7	14.5799	~	41.0307
8	51.856	8	144 456	8	15 1199	1	+1.5/6/
9	53,708	à	146 309	0	15.1100	8	42.1166
_		5	140.000	9	13.0387	9	42.6566
30	55.560	80	148.160	30	16.1987	80	43, 1965
1	57.412	1	150.012	1	16.7387	1	43 7365
2	59.264	2	151.864	2	17.2786	2	44 2765
3	61.116	3	153.716	3	17.8186	3	11.2160
4	62.968	4	155.568	4	18.3585	4	45.3564
5	64.820	5	157.420	5	18,8985	5	15 2062
6	66.672	6	159.272	ĥ	10 1784	5	16 4262
7	68.524	7	161 124	7	10.0794	-	40.4000
8	70.376	8	162 976		20 5194		+0.9/02
9	72.228	à	164 898	0	20.0104	8	47.5162
·		5	104.328	9	21.0583	9	48.0562
4 0	74.080	90	166.680	40	21.5983	90	48 5961
1	75.932	1	168.532	1	22,1382	1	19 1361
2	77.784	2	170.384	2	22.6782	2	49 6760
3	79.636	3	172.236	3	23 2181	3	50 2160
4	81.488	4	174.088	4	23.7581	4	50.7559
5	83.340	5	175 040	=	24 2001	-	51 00 5 5
ถื	85,192	R	177 709	6	47.4981 94 6960	ð	51.2959
7	87 044	7	170 644	0 -	24.8380	6	51.5359
2	99 00C	.0	1/9.044		25.3780	7	52.3758
0	00.090	0	151.490	8	25.9179	8	52.9158
3	50.740	100	183.348	9	26.4579	9	53.4557
		100	185.200			100	53.9957
		1	4	1	1		

Length—International Nautical Miles and Kilometers Basic relation: International Nautical Mile = 1.852 kilometers.

CHAPTER 8

STAFF TRAINING

Prepared by

State of Michigan Department of Natural Resources Bureau of Environmental Protection

October, 1983

COMMENTS ON CHAPTER 8

Staff Training

Quality Assurance for Water & Sediment Sampling

I have reviewed this chapter of the Quality Assurance Manual and recommend the following improvements:

----____ -----(attach additional pages if necessary) (Name): Submitted by (Division): (Unit): (Telephone): (Date):

Your comments will be considered in revising the manual to benefit all users. Please send this form to:

Quality Assurance Coordinator Environmental Services Division Department of Natural Resources Box 30028 Lansing, Michigan 48909

CONTENTS

- 8.A. Introduction
- 8.B. General Sample Collection and Preservation Training
- 8.C. Specialized Sample Collection and Preservation Training

TABLE

8.C.1. Example Employee Activity Checkoff List

8.A. INTRODUCTION

Obtaining a representative sample and maintaining its integrity until analysis is an integral part of all data acquisition programs. Field methods have been developed for sampling and sample preservation procedures and incorporated into this manual. Likewise, significant progress has been made towards standardizing analytical techniques and confirming the accuracy and precision of these techniques through laboratory quality control practices. However, analytical results can only be as good as the sampling and preservation methods employed. The purpose of sample collection and preservation training is to assure that field staff are familiar with and use appropriate field collection methods. Another intent of sample collection and preservation training is to provide an opportunity for presenting newly developed methods to field staff along with periodic review of existing methods.

Sample collection and preservation training is divided into two parts: a) general sample collection and preservation procedures applicable to all field staff, and b) specialized types of sample collection applicable to certain field staff.

8.B. GENERAL SAMPLE COLLECTION AND PRESERVATION TRAINING

General sample collection and preservation training is the responsibility of each program division. Laboratory staff and the Bureau Quality Assurance Coordinator provide training assistance through Laboratory User Meetings, Laboratory Newsletters and the EPB Quality Assurance Manual. Training is summarized by category as follows:

On The Job Training: Trainees receive verbal and written instructions from experienced staff. Experienced staff then demonstrate the operation and repeat instructions. Lastly, the trainee performs the operations under the supervision of experienced staff. When experienced staff are satisfied that the trainee has mastered the operation, the trainee is allowed to perform the operation independently.

Documentation of Standard Operating Procedures: Standard operating procedures are documented in the EPB Quality Assurance Manual. Such documentation provides sufficient detail to enable most sampling or field analytical activities to be performed with very little additional tormal training or oversight. Laboratory Newsletter: The laboratory distributes a newsletter to laboratory users at approximately quarterly intervals. The newsletter contains information concerning field quality control, analytical procedures, sample preservation and other information pertaining to collection and submittal of samples to the laboratory.

Laboratory User Meetings: One day laboratory user meetings are held annually or semi-annually to convey information to laboratory users. Topics are similar to items covered in the Laboratory Newsletter except that the information is conveyed in a speaker audience format which allows for more complete coverage of complex topics as well as the opportunity for direct feedback from laboratory users.

Division Training Sessions: Three day division training sessions are held annually by each program division of the Environmental Protection Bureau. The topics presented at the training sessions depend on the needs of each division at the time of the training session. Information presented may include such topics as laboratory use, sampling and preservation procedures, quality control data, safety and legal issues, federal and state regulations, as well as overviews of other DNR programs.

General sample collection and preservation training includes the following topics. Special topics may be added as appropriate.

- I Introduction
- II Sampling Objectives
 - A. General
 - B. Planning Objectives
 - C. Facility Design and Operation Objectives
 - D. Regulatory, Compliance and Enforcement Objectives
- III The Sample Plan
 - A. General
 - B. Collection Methods
 - C. Parameter Selection and Detection Limits
 - 0. Sampling Locations and Frequency
 - E. Safety

- IV Sample Collection, Preservation and Laboratory Use
 - A. Sample Handling Procedures
 - 1. Sample Priority and Scheduling
 - 2. Sample Containers, Preservation and Holding Times
 - 3. Sample Labeling Requirements
 - 4. Chain of Custody Procedures
 - B. Collection Procedures
 - 1. Special Sampling Notes
 - 2. Field Notes
 - 3. Point Source Sampling
 - 4. Sampling Through Ice
 - 5. Sediment Sampling
 - 6. Soil Sampling
- V Analytical Methods
 - A. Laboratory Measurements
 - B. Field Measurements
- Vi Quality Control
- VII Interpretation and Reporting
- VIII Solution or Recommendation

Sample collection and preservation activities of each division must be appropriate for the monitoring programs of each division. Sample collection and preservation training includes items I-VIII. Item IV, Sample Collection, Preservation and Laboratory Use, is applicable to most divisions and programs and is covered by the Quality Assurance Nanual, the Laboratory Newsletter, and Laboratory User Meeting. Other items, such as sampling objectives and sample planning are program specific and must be addressed through specific training sessions and on-thejob training. Although it is not necessary for all staff to possess a working knowledge of items I-VIII, all staff should thoroughly understand their role and the way that role complements other roles.

8.C. SPECIALIZED SAMPLE COLLECTION AND PRESERVATION TRAINING

Contain units or sections are involved in specialized types of sampling or monitoring and have specialized training needs. Since these needs are diverse, training programs need to be designed to address these specific needs. Examples of such specialized sampling programs include:

- 1. Surveys of industrial and municipal discharges.
- 2. Intensive stream surveys (DO-BOD modeling).
- 3. Intensive lake surveys.
- 4. Groundwater surveys.
- 5. Hazardous waste surveys.

As with safety, proper and adequate job training of employees is a responsibility of management. Program managers have an obligation to assure that their individual organizational units receive and participate in training programs. Training needs for these units vary tremendously due to the complexity of the sampling procedures, the number of people involved, the physical location of the persons within the organizational units, the complexity of the sampling equipment, and the hazards involved. Existing training programs range from on-the-job training conducted by more experienced staff to formal scheduled training sessions. The role of the Bureau Quality Assurance Coordinator in these specialized training programs is to: a) assure that procedures are consistent when two or more units are involved in the same type of specialized sampling (e.g., groundwater sampling and hazardous waste sampling); b) to assure that the training programs adequately meet the needs of the staff involved in sampling; and c) to assure that instruction is consistent with procedures detailed in this manual.

To monitor and record the training of each employee engaged in these specialized sampling activities, each division must develop and update where necessary, activity checkoff lists for the various classifications or positions within organizational units involved in sampling. The activity checkoff list is a listing of operations or activities that each employee must know to perform the duties and responsibilities of his or her position. The activities checkoff list is maintained by the Supervisor of the organizational unit and submitted to the Division Chief and Bureau Quality Assurance Coordinator annually each October. The objective of maintaining activity checkoff lists is to provide the Division Chief, Unit Supervisor, and Quality Assurance Coordinator with a mechanism for monitoring employee training and to plan training opportunities for individuals in mod of further training. An example of an activity checkoff list is attached.

TABLE 8.C.1. EXAMPLE EMPLOYEE ACTIVITY CHECKOFF LIST - BIOLOGY SECTION

ACTIVITIES	Darwin	Pennak	Prescott	Fasset	Hynes	Cousteau
Qualitative Benthos Sampling	Р	Р	N	N	Р	NA
Artificial Substrate Sampling	Р	P	N	N	Р	NA
Ponar Sampling	NA	Р	N	N	Р	NA
Benthic Sample Analysis	Р	Р	N	N	Р	NA
Macrophyte Sampling	Р	N	Ъ	р	Р	NA
Lake Sampling	P	P	Р	р	Р	Р
Paytopianton Sampling	Р	N	Р	р	Р	Р
Periphyton Sampling	P	N	Р	Р	Р	2
Phyroplankton-Periphyton Analysis	2	N	Р	P	Р	NA
Gill Net Sampling	NA	Р	N	N	Ъ	Р
Boom Shocker Sampling	NA	Р	N	N	Р	Р
Back Pack Shocker Sampling	NA	Р	N	N	Р	Р
Fish Identification	Р	Р	N	N	Р	Р

Status: 2 - employee is capable of performing this activity.

N ~ employee needs training for this activity.

NA this activity is not applicable to the employee's duties.

CHAPTER 9

QUALITY ASSURANCE AND INTERNAL AUDITS

Prepared by State of Michigan Department of Natural Resources September, 1994

CONTENTS

9.A.	INTRODUCTION
9.B.	U.S. EPA QUALITY ASSURANCE EVALUATIONS
9.C.	REFERENCE SAMPLE EVALUATIONS
9.D.	LABORATORY INTERNAL AUDIT POLICY
	1. INTRODUCTION
	a. Quantitative Results
	b. Semi-Quantitative Results
	c. Qualitative Results
9.E.	LABORATORY INTERNAL AUDIT PROCEDURES
	1. AUDIT DEFINITIONS
	2. INORGANIC UNIT ROUTINE QUALITY CONTROL AUDITS 9
	4. ORGANIC UNIT ROUTINE QUALITY CONTROL AUDITS
9.F.	QUALITY CONTROL DATA EVALUATION
	1. INTRODUCTION
	2. GLOSSARY
	3. ESTABLISHMENT OF CONTROL LIMITS
	a. Scope and Application
	b. Summary of Method
	c. Collection of Data
	d. Test for Outliers
	e. Calculation of Statistics
	1. Mean
	2 Variance 23
	3 Standard Deviation 25
	6 Lincon Democracion
	6. Linear Regression
	7. Correlation Coefficient and Coefficient of
	8. Pooling Estimates of Standard Deviation 26
	4. EXAMPLES
	a. Introduction \ldots \ldots \ldots \ldots \ldots 27
	b. Data Sets
	c. Example 1: Paired Replicate Values
	1. Mean Range
	2. Segmenting the Sample
	3. Line of Regression
	d. Example 2: Univalued Audit
	1. Mean and Standard Deviation
	2. Moving Average and Range
	5. CONTROL CHARTS
	a. Introduction
	b. Control Chart Development
	C. Interpretation of Control Charts

List of Tables

9.C1	Summary of Reference Sample Evaluation Procedures 9-3
9.D1	Method and Analyses Categories 9-5
9.E1	Environmental Laboratory Quality Control Samples Used to Monitor Precision, Accuracy and Calibration Within Each Laboratory Unit
9.E2	Inorganic Unit Routine Quality Control Audits and Frequencies
9.E3a	Inorganic Unit Routine Quality Control Audits, Frequencies and Control Limits for Water Samples 9-10
9.E3b	Inorganic Unit Routine Quality Control Audits, Frequencies and Control Limits for Sediment Samples 9-11
9.E4a	Organic Unit Routine Quality Control Audits and Frequencies for Water Samples
9.E4b	Organic Unit Routine Quality Control Audits and Frequencies for Sediment and Tissue Samples 9-16
9.E5	Factors for Calculating Control Limits 9-21
9.F1	t Distribution Values
9.F2	Ordered Paired Replicate Values
9.F3	Large Univalued Data Set

FIGURES

9.F1	Quality Contr	ol Charts		•	•	•	•	•	•	•		•	9-3	8

Page

CHAPTER 9. QUALITY ASSURANCE AND INTERNAL AUDITS

9.A. INTRODUCTION

Successful monitoring of the environment requires identification of contaminants, accurate measurement of the amounts present and pinpointing the sources. Because of the increase in the number of environmental contaminants requiring sophisticated trace level analyses, the involvement of an increasingly large number of people and laboratory capabilities are required.

Performance enhancement of the Environmental Laboratory is the primary goal of the laboratory audits described in this chapter. Their purpose is to assess the extent to which the laboratory follows sound scientific and analytical procedures, operates under the guidance of good management and professional supervision, utilizes proper equipment, and maintains and uses accurate records. The audit provides the laboratory with an opportunity for improvement by identifying weaknesses in its organization or performance and to obtain information and assistance for overall improvement. In this sense, the audit serves to assure laboratory competence.

9.B. U.S. EPA QUALITY ASSURANCE EVALUATIONS

The U.S. Environmental Protection Agency (EPA), Quality Assurance Office, Region V, annually conducts an evaluation of the Environmental Laboratory and the quality assurance program in general. The evaluation includes eight major functional areas. These areas are as follows:

- 1. Organization.
- 2. Personnel.
- 3. Laboratory facilities.
- 4. Laboratory equipment and instrumentation.
- 5. Methodology.
- 6. Sample collection, holding times and preservation.
- 7. Quality control.
- 8. Data handling.

The EPA evaluates the eight major functional areas according to specific EPA criteria designed to assure the quality of data in federally funded monitoring programs. The EPA summarizes their evaluation in a system evaluation report designed to outline deficiencies noted during the evaluation of the laboratory and the quality assurance program. The state responds to the system evaluation and outlines plans to resolve the identified deficiencies.

The EPA also administers an Annual Water Pollution Performance Evaluation Program. The program consists of prepared samples for a large number of parameters applicable to water pollution programs. Reference samples are shipped to participating laboratories in sealed vials. The participating laboratory then makes up test solutions from these vials, preserves the samples according to standard practices, and conducts the analyses. The EPA evaluates the analytical results and reports back to the participating laboratory. The entire process from sample shipment to final report involves approximately four months. Results reported outside acceptable ranges serve as notice to participants that review of analytical techniques or equipment may be necessary.

9.C. REFERENCE SAMPLE EVALUATIONS

Reference samples are special samples developed by an outside source for internal evaluation of analytical performance. The Environmental Laboratory presently analyzes reference sample sets provided by the U.S.

Environmental Protection Agency (EPA). The Quality Assurance Coordinator is responsible for scheduling and distributing the reference sample sets to the laboratory units. Sets are scheduled for analysis every four months and are distributed without "true values". Reference samples are also available upon request from the Laboratory Director.

Analysts perform the analyses along with other routine samples using normal procedures. If more than one method of analysis is routinely employed or more than one analyst commonly performs certain analyses, each method and analyst should be evaluated separately. The analysts transmit sample results to unit supervisors who then transmit results to the Quality Assurance Coordinator. The Quality Assurance Coordinator notifies the appropriate laboratory unit in writing when reference sample results are overdue, records the true value (or mean value), calculates the percent bias and distributes these results to the Unit Supervisor and the reference sample result file. The reference sample result file is available for review by the Quality Assurance Team and other laboratory staff. The Quality Assurance Coordinator is responsible for on-going compilation, review and evaluation of reference sample results. When reference sample results deviate from the true value by more than acceptable limits, the appropriate unit supervisor is notified in writing. The Unit Supervisor must then investigate the cause of the bias and notify Quality Assurance Coordinator of his evaluation and/or proposed actions. This procedure is summarized in Table 9.C.-1.

EPA reference sample acceptance limits (if established) are used to evaluate performance. In those cases where EPA reference sample acceptance limits have not been established, acceptance limits will be as follows. The Inorganic Unit acceptance limits are plus or minus twice the reported detection limit plus 20% of the "true value". Acceptance limits for the Organic Unit are plus or minus 50% of the "true value" or mean recovery derived from the method validation study.

9.D. LABORATORY INTERNAL AUDIT POLICY

9.D.1. INTRODUCTION

The Laboratory is charged with providing analytical services to the programs it supports compatible with the needs of those programs. The amount of effort devoted to a particular sample type or group of analyses may vary according to program needs. Low level analysis of contaminants in drinking water or fish tissue may require more attention to sample
Table 9.C.-1 Summary of Reference Sample Evaluation Procedures

Who	Does What
Quality Assurance Coordinator	Schedules and distributes reference sample sets 3 times per year.
Analyst	Performs analysis with routine samples.
Analyst	Transmits reference sample results to the Unit Supervisor.
Unit Supervisor	Reviews and reports reference sample results to the Quality Assurance Coordinator.
Quality Assurance Coordinator	Notifies Unit Supervisor in writing when reference sample sets are overdue.
Quality Assurance Coordinator	Records the true value, and/or mean calculates the percent bias, distributes these results to Unit Supervisors.
Quality Assurance Coordinator	Notifies the Unit Supervisor and Lab Director in writing when results deviate from the true value by more than accept- able limits.
Unit Supervisor	Investigates the cause of the bias and notifies the Quality Assurance Coordina- tor findings, conclusions or proposed
Quality Assurance Coordinator	actions. Follows-up on any analytical problems being investigated and corrected by administering another reference sample
Quality Assurance Coordinator	Continuously compiles, reviews and evaluates reference sample results.

collection, preservation, preparation and analysis details than analysis of a liquid waste or sludge.

Data users must be aware of the quality of their data to properly interpret and apply it. To assist laboratory users with interpretation of their data, the quality of laboratory results is categorized as either quantitative, semi-quantitative, or qualitative. These categories are described in Sections 9.D.1.a through 9.D.1.c. and in Table 9.D.-1.

9.D.1.a. Quantitative Results

Quantitative results are achieved through the use of procedures approved by the Laboratory Director which possess routine quality control audits (in-control) specific to the matrix and working range of the method. In addition, the analysis is performed on a properly collected and preserved sample, analyzed within the prescribed holding time. Results from a quantitative analysis are reported without qualifier codes and should be accurate within the limits specified in the method.

9.D.1.b. Semi-Quantitative Results

Semi-quantitative results may result from the use of either an approved method (approved by the Laboratory Director) or a published method (Standard Methods, Federal Register, ASTM, EPA Reference or other reference.) When an approved method is used, one of the following conditions may limit the quality of data and cause results to be reported as semiquantitative:

- 1. The analysis was conducted without a quality control audit applicable to the matrix and concentration range.
- 2. The analytical system was out of control or control status was unknown.
- 3. The quality of the sample was compromised through the use of inadequate collection, preservation or holding time procedures.

Results will also be considered semi-quantitative when a published method is used which has not previously been verified by the laboratory. Semi-quantitative analyses are reported with qualifier codes listed in Appendix 1.C.-9.

9.D.1.c. Qualitative Results

A qualitative result may result from any of the following causes:

- 1. The use of an unapproved laboratory method.
- 2. The use of a procedure which does not have a routine quality control audit for the matrix or applicable concentration range.
- 3. Results obtained during a period or sample batch when existing quality control audits indicated a potential gross bias.
- 4. Results from samples improperly collected, preserved or held so that accuracy is severely affected.

Results from a qualitative analysis are reported in the form of a narrative statement or a concentration range.

Table 9.D.-1. Method and Analyses Categories

Category	Test Procedure	Quality Control	Results
Type I Quantitative Analysis	Laboratory Approved Methods	Routine QC audits for the matrix and applicable concentration range, Analytical system in control, <u>and</u> Properly collected and preserved sample and analyzed within maximum holding time.	Numerical
Type II Semi-quantitative	Laboratory Approved Methods or methods validated for	Insufficient or no routine QC audit for the matrix and applicable concentration range, <u>or</u> System out-of-control or unknown status, result <u>or</u>	Numerical with appropriate remark code
Analysis	environmental analysis	Sample collection, preservation or holding time are different from the recommended procedure so that the accuracy of the results may be affected.	
		No routine QC audits,	
Type III	May not have a Laboratory Approved	<u>or</u> OC audits indicate a potential gross bias and source	Statement or concentration
Qualitative Analysis	Method or a validated method	of bias unknown, <u>or</u> Sample collection, preservation or holding time are significantly different from the recommended procedures so that the accuracy of the results may be severely affected.	range

9.E. LABORATORY INTERNAL AUDIT PROCEDURES

All quantitative analyses have quality control audits and established control limits for calibration, precision and accuracy, if appropriate. These audits are designed so that an accurate determination of an in- or out-of-control situation can be made. Control limits are three standard deviations or less and are based on an estimate of the in-control population standard deviation.

Table 9.E.-1 identifies and compares the audits used by each laboratory unit. These audits are described in Section 9.E.1. Each laboratory unit has procedures for evaluating and correcting out-of-control situations. These procedures are as follows:

In general, when a control limit is exceeded, the analyst, lead worker or supervisor evaluates the situation to determine whether the system or sample is out-of-control. If it is determined that the sample is out of control, the analysis is repeated if sufficient sample is available. If there is not sufficient sample, results are reported with an appropriate qualifier code. If it is determined that the system is out-of-control, the analysis must be stopped and the problem corrected. Quantitative analyses may not be reported from an out-of-control analytical system. Appropriately qualified semi-quantitative or qualitative results may be reported.

Analytical precision (determined from lab duplicates and matrix spike duplicates) and accuracy are periodically reviewed to determine if control limits need to be changed or if the precision and accuracy statement of the analytical method needs to be changed.

9.E.1. AUDIT DEFINITIONS

<u>Within-run duplicates</u> are two aliquots of a single sample analyzed within a single batch of samples. Results are usually used to measure within-run precision.

<u>Between-run duplicates</u> are two aliquots of a single sample analyzed in two (usually consecutive) batches of samples. Results are used informally to confirm positive samples. Measures precision of between run duplicates.

<u>Control Sample or Mixed Control Sample</u> is a standard, stable natural sample, or a natural sample fortified with a standard which is analyzed in each analytical batch to check calibration, accuracy, or between-run precision.

<u>Sample spike</u> is a sample aliquot which has had added to it a known volume of a standard. It is generally used to measure the recovery of an extraction or digestion step. The accuracy of a method can be estimated from recovery data.

Table 9.E.-1. Environmental Laboratory Quality Samples Used to Monitor Precision, Accuracy, and Calibration Within Each Laboratory Unit. W(water), S (sediments) and T (tissue) indicate the quality control sample matrix.

	GENERAL CHEM	INORGANIC	ORGANIC
PRECISION Within-run duplicates	W, S	W, S,T	W,S,T
Mixed control sample (dup.)		W, S	
Control sample Matrix spike (dup.)	W, S	W,S	W,S,T
ACCURACY 1 Sample spike	W	W,S,T	s, w
Blank spike Surrogate	W, S	W,S,T	W, S,T
Control sample	W, S		
Matrix sample			W, S,T
CALIBRATION			
Checked standard	W	W	W,S
Reagent blank	W	W,S	W,S
Sample blank	W	W	
Titrant check	W		
Blank spike			W

¹ Accuracy is monitored in the Organic Unit sediment and tissue samples by comparing duplicate matrix spike samples to an unspiked matrix control.

<u>Blank spike</u> is a reagent blank which has had added to it a volume of a standard of known concentration. It is generally used to measure the recovery of a process such as an extraction or digestion step or as a check on calibration.

<u>Matrix spike</u> is similar to a blank spike except that the spike is added to a sample matrix. It is used to measure the recovery of a process or as a measure of precision when analyzed in duplicate. The use of a sample matrix is intended to include matrix interference effects in the precision or accuracy measurement.

<u>Internal standard</u> is a compound not naturally found in routine samples spiked into samples and blanks before analysis. Its recovery is used to correct sample concentrations or variations in instrument response, injection volume and etc.

<u>Surrogate standard</u> is a compound not naturally found in routine samples spiked into samples and blanks before analysis to monitor the total recovery of sample preparation and analysis procedures.

<u>Reagent blank</u> is an aliquot of distilled water and sample reagents taken through an entire analytical procedure to check the zero-intercept of a calibration procedure or to determine that the analytical system is interference free.

<u>Check standard</u> is a standard which is analyzed separate from the calibration procedure as a check on it.

<u>Sample blank</u> is a sample aliquot to which all chemical reagents except one key ingredient have been added. It is used in colorimetric procedures to define background absorbance on samples due to sources other than the constituent being quantified.

<u>Titrant check</u> is a check on the normality of the titrant. It is used in those instances where a standard curve is not practical (dissolved oxygen and alkalinity).

<u>Gas Chromatograph (GC) Monitoring Compound</u> is a compound of known concentration added to a GC extract prior to injection to monitor retention times and detector response.

Routine quality control audits and control limits used by Inorganic Unit are documented in each method. These audits and their frequency are identified in Table 9.E.-2. Generally three standard deviation control limits are statistically derived and used to identify an out-of-control situation. In addition, most precision audits (generally within-run duplicates) also have two standard deviation warning limits. If an audit exceeds a warning limit on three consecutive batches, the system is also considered out-of-control. The various routine audits are evaluated and used as described in 9.E.2.a.-g. below.

a. <u>Within-run duplicates</u>. Within-run duplicates are used to evaluate precision. To reflect increasing precision over the analytical operating range of the procedure, a line of regression is usually defined from the mean difference of the duplicate segmented values versus the mean of their values in the data segment. Warning and control limits are established from this line of regression. This method of data analysis is explained in 9.F.4.c.2.-3. of this Chapter. b. <u>Sample spikes</u> Mixtures of samples and standards are routinely used in the Inorganic Unit to determine the accuracy of the analytical process. Control limits are determined from the mean recoveries and the variability of the sample/standard mixture spike over the operating range of the test. Recovery calculations are explained in 9.F.3.e.5. of this Chapter.

c. <u>Control sample</u>. For those parameters that are determined from a sediment matrix, the Inorganic Unit analyzes special sediment samples in each batch for which expected values have been derived from past analyses. From the results of these control samples, between-run precision is calculated and recovery is estimated. Three standard deviation control limits are used.

d.<u>Check standard</u>. The Inorganic Unit frequently uses check standards, particularly if the instrument calibration technique is not very sophisticated (e.g. an intercept and slope adjustment only). Three standard deviation control limits are derived from evaluation of past performance.

e. <u>Reagent blank</u>. A reagent blank is an aliquot of distilled water taken through the entire analytical procedure to check the zero-intercept of the calibration procedure. The frequency of reagent blanks depends upon whether a baseline shift is routinely experienced on a particular analytical system. Three standard deviation control limits are established from statistical evaluation of past audits.

f. <u>Sample blank</u>. As used in the Inorganic Unit, a sample blank is a separate sample aliquot to which all chemical reagents have been added except one key ingredient. For colorimetric procedures, this defines background absorbance from samples due to sources other than the constituent being quantified. The background absorbance is subtracted from regular absorbance before the concentration is calculated. If sample blank absorbance is very high, calculated results are then coded (PA) to indicate that the precision of results may have been affected.

g. <u>Titrant check</u>. For titrimetric procedures, a standard curve is usually defined for each analytical batch so that the normality of the titrant is not particularly critical. A check on the normality of the titrant is used if a standard curve is not practical (i.e. dissolved oxygen). Three standard deviation control limits are derived from statistical evaluation of past performance.

9.E.2. INORGANIC UNIT ROUTINE QUALITY CONTROL AUDITS

Routine quality control audits used by the Inorganic Unit are documented in each method. These quality control audits, frequencies and control limits are summarized in Table 9.E.-3a for water samples and in Table 9.E.-3b for sediment samples. Table 9.E.3a. Inorganic Unit Routine Quality Control Audits, Frequencies and Control Limits for Water Samples.

With each batch of analyses a set of duplicate analysis, a midpoint standard, a spike to determine accuracy (recoveries), and reagent and water blanks are analyzed one for each twenty (20) analyses.

Standard curves are run with each batch of samples.

Interlaboratory QC consists of running a known certified sample with each analytical batch and participation in performance audits from EPA on a quarterly basis.

Parameters	Accuracy	Precision
	Range of %	% Difference
	Recovery	(between
		duplicates)
Alkalinity	97.6 +/- 5	5
Aluminum	101.3 + /-10	10
Arsenic	97.7 + / - 10	10
Antimony	94.0 + / - 10	10
Barium	99.1 + / - 10	10
Beryllium	96.0 +/- 10	10
Cadmium	103.4 + / - 10	10
Calcium	99.3 +/- 5	10
Chromium	98.0 +/- 10	10
Chloride	99.7 +/- 5	5
Cobalt	100.4 + / - 10	10
Copper	96.5 +/- 10	10
Hexavalent Chromium	103.6 + / - 10	5
Iron	97.7 +/- 15	10
Lead	96.7 +/- 10	10
Lithium	96.2 +/- 10	10
Magnesium	98.8 +/- 5	5
Manganese	100.5 +/- 10	10
Mercury	99.9 +/- 15	10
Molybdenum	98.5 +/- 10	10
Nickel	97.9 +/- 10	10
Potassium	100.4 +/- 5	5
Selenium	95.0 +/- 10	10
Silver	97.1 +/- 10	10
Sodium	100.1 + - 5	5
Thallium	93.8 +/- 15	15
Titanium	96.7 +/- 10	10
Vanadium	101.9 + / - 10	10
Zinc	100.4 +/- 15	10

Table 9.E.3b. Inorganic Unit Routine Quality Control Audits, Frequencies and Control Limits for Sediment Samples.

With each batch of analyses a set of duplicate analysis, a midpoint standard, a spike to determine accuracy (recoveries), and reagent and water blanks are analyzed one for each twenty (20) analyses.

Standard curves are run with each batch of samples.

Interlaboratory QC consists of running a known certified sample with each analytical batch and participation in performance audits from EPA on a quarterly basis.

Parameters	Accuracy	Precision
	Range of %	<pre>% Difference</pre>
	Recovery	(between
		duplicates)
Aluminum	101.5 +/- 15	15
Arsenic	102.2 +/- 15	15
Barium	104.7 +/- 15	15
Beryllium	99.3 +/- 15	15
Cadmium	104.2 +/- 10	10
Chromium	104.7 +/- 15	15
Cobalt	107.2 +/- 15	15
Copper	107.2 +/- 10	10
Iron	110.3 +/- 15	15
Lithium	102.2 +/- 15	15
Lead	105 + / - 15	15
Mercury	99.9 +/- 15	15
Manganese	106.9 + / - 15	15
Molybdenum	98.4 +/- 15	15
Nickel	108.4 + / - 15	15
Selenium	95.3 +/- 15	15
Titanium	101.6 +/- 15	15
Vanadium	99.9 +/- 15	15
Zinc	105 +/- 15	15

The analyst evaluates the control data from each run to determine outof-control situations and identifies invalid data for review by the Unit Supervisor. The analyst then initials the results and transmits them to the unit supervisor with a recommendation to repeat the samples or accept the data. In most cases the samples can be repeated if necessary. The analyst also periodically evaluates the trend of each audit to determine if the procedure is performing adequately and notifies the unit supervisor when corrections or modifications are necessary.

The unit supervisor makes the final decision on the acceptability of the data by accepting or rejecting runs partially or totally, sets control limits for each type of audit and periodically evaluates the performance of the system.

۰.

Table 9.E.-4a Organic Unit Routine Quality Control Audits' for Water Samples. he minimum frequencies are: Juplicate Matrix Spikes - every batch for extractables, ever 12 hours for 624. Reagent Blank - every batch for extractables, ever 12 hours for 624. Sample Surrogates - all samples, blanks, and spikes. Continuing Calibration Standard - every 12 hours or after 10 samples. (Maximum percent difference is calculated from the original calibration).

	Accuracy	Precision	<u>Calibration</u>
Parameters	Range of 8	8 Difference	(all compounds)
	Recovery	(of duplicates)	max.% Difference
Method 624 Control Limits			25% of RRF
1.1-DICHLOROETHENE	98 +/- 46	12	(internal std.)
BENZENE	102 + / - 16	8.2	(,
CHLOROBENZENE	100 + / - 16	8.8	
TOLUENE	106 + / - 20	9.8	
TRICHLOROETHENE	99 +/- 27	8.8	
Method 624 Surrogate Contro	l Timita		
1 2 DICHLOROFTHANE-d4	$\frac{1}{101} \frac{1}{101} \frac{1}{101} \frac{1}{101} \frac{1}{100} \frac{1}$		
BROMOFLUOROBENZENE	102 + 7 = 26		
TOLUENE-d8	102 + - 19		
	/		
Method 625 (Base-Neutral) C	ontrol Limits		25% of RRF
1,2,4-TRICHLOROBENZENE	56 +/- 24	21	(internal std.)
1,2-DICHLOROBENZENE	61 + 7 - 22	20	· · · ·
,2-DIPHENYLHYDRAZINE	83 +/- 42	25	
., 3-DICHLOROBENZENE	56 +/- 20	18	
1,4-DICHLOROBENZENE	63 +/- 23	21	
2,4-DINITROTOLUENE	71 + / - 41	20	
2,6-DINITROTOLUENE	76 +/- 46	28	
2-CHLORONAPHTALENE	71 + 7 - 27	29	
4-BROMODIPHENYLETHER	95 + / - 44	29	
4-CHLORODIPHENYLETHER	96 + 7 - 65	31	
BIS (2-CHLOROETHOXY) METHANE	98 + / - 22	23	
BIS(2-CHLOROETHYL)ETHER	95 +/ - 28	26	
BIS (2-CHLOROISOPROPYL) ETHER	98 +/- 24	34	
BIS(2-ETHYLHEXYL)PHTHALATE	78 +/- 66	72	
BUTYL BENZYL PHTHALATE	91 +/- 67	36	
DI-N-BUTYL PHTHALATE	104 + 7 - 44	29	
DI-N-OCTYL PHTHALATE	63 + 7 - 52	51	
DIETHYL PHTHALATE	80 + / - 29	31	
DIMETHYL PHTHALATE	74 + / - 47	38	
HEXACHLOROBENZENE	72 + 7 - 40	16	
HEXACHLOROBUTADIENE	47 + 7 - 30	31	
HEXACHLOROCYCLOPENTADIENE	38 + 7 - 68	46	
HEXACHLOROETHANE	$50 \pm 7 = 23$	21	
ISOPHORONE	79 + 1 - 55	20	
N-NITROSO-DI-N-PROPVIAMINE	110 + 46	34	
N-NITROSO DI N FROTILAMINE	127 + / - 51	24	
NTTROBENZENE	74 + / - 38	20	
	, <u>, ,</u> , , , , , , , , , , , , , , , ,	20	

Table 9.E.-4a continued.

<u>Parameters</u>	Accuracy Range of % Recovery	Precision% Difference(of duplicates)	Calibration(all compounds)% Difference
Method 625 Surrogate Control	LImits		
NITROBENZENE-d5	77 + 7 - 31		
2-FLUOROBIPHENYL	75 + 7 - 31		
TERPHENYL-d4	82 +/- 45		
	,		
Method 625 Phenols			
2-CHLOROPHENOL	54 +/- 24	20	25% of RRF
4-CHLORO-m-CRESOL	59 +/- 34	33	(internal std.)
4-NITROPHENOL	19 +/- 22	35	
PENTACHLOROPHENOL	78 +/- 56	45	
PHENOL	23 +/- 10	10	
Method 625 (phenols) Surrog	ate Control L	imits	
2,4,6-TRIBROMOPHENOL	67 +/- 47		
2-FLUOROPHENOL	32 +/- 22		
PHENOL-D5	23 +/- 30		
Method 608/612/8121 (Scan 3)		0.0 a
1, 3-DICHLOROBENZENE	49 +/- 26	18	20%
1,4-DICHLOROBENZENE	53 +/- 33	27	(external std.)
1, 2-DICHLOROBENZENE	53 +/- 2/	22	
1 2 E TRICULOROETHANE	52 +/- 29	20	
1, 3, 5-TRICHLOROBENZENE	54 +/- 2/	19	
1,2,4-IRICHLOROBENZENE	57 + 7 = 29 61 + 7 = 27	17	
HEYACHI ODORUTADIENE	51 + 7 = 27	17	
	50 + 7 - 33	22	
HEXACHLOROCYCLOPENTADIENE	45 + / = 44	24	
2-CHLOBONAPHTHALENE	82 +/- 27	17	
1.2.3.4-TETRACHLOROBENZENE	67 + 7 - 29	21	
PENTACHLOROBENZENE	75 + / - 20	18	
A-BHC	84 + / - 29	15	
HEXACHLOROBENZENE	76 +/- 26	16	
B-BHC	91 +/- 22	16	
G-BHC (LINDANE)	87 + / - 30	17	
PENTACHLORONITROBENZENE	74 +/- 28	15	
D-BHC	83 +/- 40	14	
HEPTACHLOR	84 +/- 39	25	
ALDRIN	78 +/- 32	27	
HEPTACHLOR-EPOXIDE	89 +/- 27	19	
G-CHLORDANE	87 +/- 25	17	
ENDOSULFAN 1 ²	20 +/- 20	4	
A-CHLORDANE	92 +/- 33	19	
DIELDRIN	83 +/- 43	18	
4,4'-DDE	86 +/- 30	18	
ENDRIN	95 +/- 47	22	
4,4'-DDD	90 +/- 34	24	
4,4'-DDT	96 +/- 46	19	
HEXABROMOBENZENE	65 +/- 27	14	
METHOXYCHLOR	101 +/- 44	20	
MIREX	93 +/- 56	33	
PPB (BP-6)	69 +/- 49	14	
AROCLOR 1242	86 +/- 24	17	
AROCLOR 1260	93 +/- 24	16	

"able 9.E.-4a continued.

<u>Parameters</u>	<u>Accuracy</u> Range of % <u>Recovery</u>	Precision <u>Precision</u> <u>Difference</u> (of duplicates)	Calibration (all compounds) % Difference
Method 608/612 Surrogate 2,4-DICHLOROTOLUENE 4,4-DIBROMOBIPHENYL	<u>Control Limits</u> 68 +/- 33 80 +/- 22		

¹Accuracy and precision data are derived from 1993 Duplicate Matrix Spikes and are updated quarterly or when sufficient data is collected. Surrogate accuracy data was derived from 1993 data and is updated quarterly or when sufficient data is collected.

²Endosulfan 1 is reported semi-quantitatively.

Table 9.E.-4b Organic Unit Routine Quality Control Audits¹ for Sediment Samples. The minimum frequencies are: Duplicate Matrix Spikes - every batch for extractables, ever 12 hours for 8260. Reagent Blank - every batch for extractables, ever 12 hours for 8260. Sample Surrogates - all samples, blanks, and spikes. Continuing Calibration Standard - every 12 hours or after 10 samples. (Maximum percent difference is calculated from the original calibration).

	Accuracy	Precision	<u>Calibration</u>
Parameters I	Range of %	<u>% Difference</u>	(all compounds)
	Recovery	(of duplicates)	max. %Difference
Method 8260 (Volatile) Cont	trol_Limits		25% of RRF
1,1-DICHLOROETHENE	104 +/- 31	15	(internal std.)
BENZENE	96 +/- 38	14	
CHLOROBENZENE	100 +/- 20	11	
TOLUENE	98 + /- 26	16	
TRICHLOROETHENE	111 +/- 50	12	
Method 8260 Surrogate Cont:	rol Limits		
1,2-DICHLOROETHANE-d4	102 + / - 45		
BROMOFLUOROBENZENE	96 +/- 39		
TOLUENE-d8	107 +/- 55		
Method 8270 (Base-Neutral)	Control Limits		25% of RRF
1,4-DICHLOROBENZENE	60 +/- 18	21	(internal std.)
N-NITROSO-DI-N-PROPYLAMINE	88 +/- 63	27	
1,2,4-TRICHLOROBENZENE	66 +/- 26	24	
ACENAPTHENE	80 +/- 32	24	
2,4-DINITROTOLUENE	66 +/- 30	27	
PYRENE	106 +/- 76	42	
Method 8270 Surrogate Cont	rol Limits		
NITROBENZENE-d5	69 +/- 18		
2-FLUOROBIPHENYL	79 + / - 19		
TERPHENYL-d14	117 + / - 33		

Table 9.E.-4b continued.

1ethod 8121/8081 (Scan 3)	Control Limits		20%
1,3-DICHLOROBENZENE	51 +/- 32	20	(external std.)
1,4-DICHLOROBENZENE	51 +/- 34	20	
1,2-DICHLOROBENZENE	55 +/- 36	20	
HEXACHLOROETHANE	66 +/- 40	22	
1,2,4-TRICHLOROBENZENE	68 +/- 31	24	
HEXACHLOROBUTADIENE	71 +/- 36	25	
2-CHLORONAPHTHALENE	77 +/- 40	22	
HEXACHLOROBENZENE	77 +/- 35	20	
PENTACHLORONITROBENZENE	87 +/- 42	29	
HEPTACHLOR	87 +/- 39	30	
ALDRIN	90 +/- 32	21	
4,4'-DDE	102 +/- 41	30	
HEXABROMOBENZENE	104 +/- 60	27	
MIREX	100 +/- 40	22	
g-BHC (LINDANE)	92 +/- 38	39	
HEPTACHLOR-EPOXIDE	96 +/- 37	32	

	Accuracy	<u>Precisi</u>	<u>on Calibration</u>
Parameters	Range of % Recovery	<pre>% Difference (of duplicates)</pre>	(all compounds) max. %Difference
		(·····································	

Method 8121/8081	(Scan 3) Control Limits continued.	
g-CHLORDANE	106 +/- 43	34
a-CHLORDANE	102 +/- 42	33
4,4'-DDD	102 +/- 42	39
4,4'-DDT	97 +/- 41	32
P-6 (PBB)	92 +/- 45	31
AROCLOR 1016	96 +/- 37	24

AROCLOF	1260		98 +/- 37 98 +/- 33	
Method	8081/8121	Surrogate	Control Limits	

2,4-DICHLOROTOLUENE	70	+/-	27
DECACHLOROBIPHENYL	82	+/-	56

¹Accuracy and precision data are derived from 1993 Duplicate Matrix Spikes and are updated quarterly or when sufficient data is collected. Surrogate accuracy data was derived from 1993 data and is updated quarterly or when sufficient data is collected.

20

9.E.4. ORGANIC UNIT ROUTINE QUALITY CONTROL AUDITS

Routine quality control audits and control limits used by the Organic Unit are documented in each method. Audits for aqueous samples are summarized in Table 9.E.-4a. Audits for sediment samples are summarized in Table 9.E.-4b.

Duplicate matrix spike samples are used to evaluate the precision of samples. Control samples, spiked samples and surrogate standards are used to monitor accuracy of aqueous and sediment samples. Check standards and reagent blanks are used as a check on calibration. These audits are described in further detail in the following paragraphs.

<u>Contamination checks</u> - Each day before processing any samples, an organic free water or reagent blank is analyzed. The system is considered interference free if the concentration of all compounds is less than one-half the reported detection limit. If the reagent blank value is less than 1/2 the detection limit, the blank value is disregarded. If the reagent blank value is greater than 1/2 the detection limit but less than 1/2 the sample value, the value is reported for reagent blank contamination and coded RB.

<u>Calibration Checks</u> - Standards are used to verify instrument calibration. The frequency, type, and acceptance limits are defined in tables 9.E.-4a and 9.E.-4b. Either an internal or external standard calibration is used.

<u>Internal Standards</u>- A known amount of compounds that are similar in analytical behavior to compounds of interest are selected and added to all standards, samples, and QC audits analyzed. It must be demonstrated that the measurement of these internal standards are not affected by method or matrix interferences. A response factor generated from analyzing calibration standards is used to calculate the amounts of compounds of interest.

<u>External Standards</u> - Compounds of known concentrations are analyzed at multiple levels. The response or area of each peak corresponding to a compound of interest is determined. This calibration is then used to determine the amounts of compounds of interest in standards, QC audits, and samples.

<u>Sample Audits</u> - The precision and accuracy of each sample batch (20 samples or less) is monitored through the use of two spiked and one unspiked matrix sample. An investigative sample will be used as the matrix sample to be spiked. Precision is evaluated by comparison of duplicate spiked matrix samples. Accuracy (recovery) is evaluated by comparing the average of the two spiked matrix samples to the unspiked matrix sample.

<u>Surrogate</u> - Surrogate compounds are added at the initial stage of sample preparation for most organic analysis to monitor the efficiency of the method. Many critical steps and the sample matrix can affect recovery. Control limits are established for the surrogate compounds in each method. If recovery of the surrogate standard exceeds the control limits, the sample is repeated or if the sample cannot be repeated, sample results are coded to indicate matrix interference or possible low or high recovery. See table 9.E.-4a and 9.E.-4b for compounds and control limits.

<u>Control Limit Calculations</u> - Control limits for Organic Unit scans are calculated as outlined in section 9.F. (Quality Control Data Evaluation) of this chapter. The number of values used to calculate each control limit will be specified in each method. Where possible, the number of values used to set control limits follows the guidelines of 9.F.3.c. (20 values for limits on a mean and 50 values for limits on a range). Some control limits have been established with fewer values and will be revised as more values are collected. The data is tested for outliers as specified in 9.F.3.d. and outliers are removed from the data set. Control limits for low level calibration standards, medium level calibration standards and recovery of the matrix spike accuracy audit are expressed as either 2 or 3 standard deviations from the mean and correspond to 95% and 99.7% confidence limits, respectively. Precision of samples is evaluated through the use of duplicate matrix spike samples. Control limits developed from less than 50 values are considered preliminary and will be revised as additional data is collected. The number of pairs used to establish each control limit should be identified in each method along with the time period over which the data were accumulated. The mean range is calculated according to procedures for paired replicate values (9.F.4.c.1.). Control limits on the mean range are calculated by multiplying the mean range value by 3.267 (the appropriate D4 factor for paired replicates from Table 9.E.-5). Duplicate matrix spike samples used to monitor water sample precision are spiked with a constant amount of standard making the audit specific to the standard concentration.

Method validation - Before a new method can be adopted or an existing method can be changed, the method must be validated as a part of the method approval process described in Appendix 1.C-1. All alternate methods must be approved by US EPA. Method validation is an initial demonstration of capability and consists of spiking a representative concentration of each compound to be measured into reagent water and/or representative sample matrices. Low level (5% of the operating range) and medium to high level replicates (spiked at concentrations representative of samples to be analyzed) should be analyzed. If natural matrices are used, the background levels in these matrices should be determined by analyzing unspiked aliquots. The average percent recovery and the standard deviation of the percent recovery is determined from spiked results according to procedures identified in 9.F.3.e.5 and 9.F.3.e.3, respectively. These recovery and standard deviation values are then compared to performance of the appropriate EPA method, EPA performance criteria or other applicable standard. Method performance is reviewed by the Unit Supervisor and the Laboratory Director for equivalency to the appropriate EPA method and ability to meet program needs.

9.F. QUALITY CONTROL DATA EVALUATION 9.F.1. INTRODUCTION

Valid data are those results obtained under controlled conditions. Statistical criteria must have been developed by which it is possible to judge when controlled conditions exist. Without this, the interpretation of the data and the conclusions reached may be misleading or of little value.

Experimental measurements are random variables. The variability of routine analyses such as differences between samples, analysis, and instrument response constitutes the analytical "error". These experimental errors are inherent characteristics of every measurement process and cannot be eliminated. The source of error affects the precision and

accuracy of all results and consequently influences their validity. All data will be reported and appropriate qualifier codes used when control limits are exceeded on outliers.

It is frequently necessary to decide whether the difference between two quantities is due to a bias or is caused purely because of the variability. If there is only a small chance that the difference is due to random error, then it is reasonable to conclude with a given degree of confidence that there is a bias. Such conclusions are based on probabilities. It is understood that a decision based on such results has a calculated chance of being wrong. The purpose of establishing control limits for a given variable is to contain the experimental error within a narrow zone and thereby minimize its effect.

TABLE 9. E.-5

FACTORS FOR CALCULATING CONTROL LIMITS

	for \overline{X}	for \overline{X}	for S	for R	for X _i	for X _i
	from R	from S	from S	from \overline{R}	from R	from S
Observations						
in Sample, n	^A 2	^A 3	^B 4	D ₄	^E 2	E3
2	1.880	2.659	3.267	3.267	2.660	3.760
3	1.023	1.954	2.568	2.574	1.772	3.385
4	0.729	1.628	2.266	2.282	1.457	3.256
5	0.577	1.427	2.089	2.114	1.290	3.191
6	0.483	1.287	1.970	2.004	1.184	3.153
7	0.419	1.182	1.882	1.924	1.109	3.129
8	0.373	1.099	1.815	1.864	1.054	3.109
9	0.337	1.032	1.761	1.816	1.010	3.095
10	0.308	0.975	1.716	1.777	0.975	3.084

C

.

ľ

9.F.2. GLOSSARY Population The set of all possible observations of a random variable. Sample Any subset of the population. Parameter A descriptive characteristic of a population, such as population mean, п. Statistic A descriptive characteristic of a sample, such as sample mean, \bar{X} . Precision The ability to replicate a value within which random deviations can usually be expected to fit. Accuracy The ability to measure a sample constituent, compared to a standard or true value, without bias or error. Control Limits The boundaries within which only the normal chance variations are present in the system. The system is then "in control". 9.F.3. ESTABLISHMENT OF CONTROL LIMITS 9.F.3.a. Scope and Application This method may be used to establish statistical control limits for most quality control audits. It is applicable to any random variable which approximates a normal distribution. Because 3s control limits are used, all control limits established by this method have a type I error of = 0.003. In other words, when a control limit is exceeded,, there is a 99.7 percent probability that the situation is not solely due to random variability. 9.F.3.b. Summary of Method Applicable audits are chosen for a given analytical procedure. Generally, QC audits are needed to control or verify calibration, accuracy, and precision. Additionally, system audits may be used to monitor and control other variables in an analytical process. For each audit, the

population to be controlled by the audit is defined. A random sample of the population is collected to establish initial control limits for an audit. Control limits may be re-evaluated as necessary to reflect changes in the population as more data are accumulated. However, change in an audit indicating a lower level of performance in a system should be investigated and not be automatically accepted.

Any result falling outside the control limits indicates an out-ofcontrol situation with respect to the population subset used to define the control limit. If the population is normally distributed, this conclusion can be made with a 99.7 percent level of confidence.

9.F.3.c. Collection of Data

Sufficient data from a controlled system must be collected from the population before statistical limits can be established. Large samples, N, are desirable. For limits on a mean, at least 20 values should be used. To establish control limits for a standard deviation or a range, at least 50 values should be obtained.

9.F.3.d. Test for Outliers

When evaluating a set of data, it may initially be necessary to test whether all values should be included in the evaluation. Wild or unusual observations may exist in the data set. Table 9.F.-1, t Distribution Values, can be used to identify outliers with a probability of 0.995 (= 0.0025). Data points acquired that can be attributed to identifiable and correctable problems should NOT be used as a valid data point.

For paired replicate values (Example 1, 9.F.4.c.), first calculate the mean range. Divide the mean range by 1.128 to obtain the standard deviation. Multiply the standard deviation times the appropriate t value for the degree of freedom used. Discard replicate values if their range exceeds the obtained value. Recalculate the mean range and again check for any outliers. Continue this process until no values are discarded.

For an univalued audit (Example 2, 9.F.4.d.), calculate the mean and standard deviation. Multiply the standard deviation times the appropriate t value. Use this number to set a confidence interval around the mean. If any values are outside the confidence interval, discard the <u>one</u> farthest from the mean. Then recalculate the mean and standard deviation and repeat the process until no more outliers are found.

Note: Again, we have assumed that the population approximates a normal distribution. If it does not, particularly if it is highly skewed, the above test is inappropriate.

9.F.3.e. Calculation of Statistics

9.F.3.e.1. Mean

The best estimate of the true population mean, u, is the overall sample mean (or average), X, where:

$$X = \frac{X_1 + X_2 + \dots + X_n}{N} = \frac{\Sigma X}{N}$$

or, the sample mean is equal to the sum of the individual values divided by the number of values, N.

9.F.3.e.2. Variance

A common measure of variability is variance. The population variance, 0-2, can be estimated from the sample variance, s^2 , where:

$$s^{2} = \frac{(X - X)^{2}}{N-1}$$

or, the sample variance is equal to the sum of individual values minus the sample mean squared divided by one less than the number of values.

TABLE 9.F.-1

t DISTRIBUTION VALUES

Degrees of Freedom (N-1)

Value at 0.995 Probability

1	127.32
2	14.089
3	7.453
4	5.598
5	4.773
6	4.317
7	4.029
8	3.833
9	3.690
10	3.581
11	3.497
12	3.428
13	3.372
14	3.326
15	-3.286
16	3.252
17	3.222
18	3.197
19	3.174
20	3.153
22	3.119
24	3.091
26	3.067
28	3.047
30	3.030
40	2.971
60	2.915
120	2.860
\sim	2.807

-

2/94

:

9.F.3.e.3. Standard Deviation

Because variance is a sum of squares, its unit is the square of the unit in which the measurement was made. A standard deviation, or the square root of variance, is used to obtain a measure of variability in the same unit as the data. The population standard deviation, , can be estimated by the sample standard deviation, s, where:

$$s = \sqrt{s^2} = \sqrt{\frac{(X - X)^2}{N-1}^2}$$

9.F.3.e.4. Range

The range of values, R, in a sample may be required, where:

R = X max - X min

or, the range is equal to the largest value minus the smallest value.

9.F.3.e.5. Recovery and Bias

Usually when accuracy or calibration audits are evaluated, recovery and/or bias are determined, either as an absolute or relative to a known or design value (a percentage is calculated).

When a QC audit with a known (or design) value is analyzed, such as a check standard, primary standard, or reference sample, it can be evaluated using any (or all) of following techniques:

-- bias = result obtained - true value

-- % bias = <u>bias</u> x 100% true value

% recovery = <u>value obtained</u> x 100% true value

When spiking techniques are used, the concentration of the solution being spiked, the concentration of the spiking standard, and the volumes of the solution and standard may need to be known. If the volumes are unimportant due to the spike volume being an insignificant part of the total volume (less than two percent) or volumes are changed by an extraction or digestion process, use the following techniques:

theor. spiked sample value = sample conc. + std. conc. x (standard volume sample volume

bias = analyzed spiked sample value - theoretical spiked sample value

% bias = _____bias x 100%
theoretical value of spike

% recovery = <u>analyzed value of spike</u> x 100% theoretical value of spike

When spike and sample volumes are critical, calculate the theoretical spiked sample value as:

Bias, % bias, and % recovery would be calculated as above.

For the special case when equal volumes of sample and standard are mixed and analyzed, the theoretical spiked sample value is simply:

theoretical spiked sample value = <u>sample value + standard value</u>

When blank spikes are used, portions of the above equations relating to "sample" concentration (which would be "zero") can be deleted.

9.F.3.e.6. Linear Regression

Frequently it is desirable to evaluate the linearity of the relationship of two variables. Fortunately, this is a simple process for most statistical calculators. The basic notations to manually calculate a

least squares regression line of $\hat{y} = \hat{\alpha} + \hat{B} X$ are:

$$s_{x}^{2} = \Sigma X^{2} - N X^{2}$$

$$s_{xy}^{2} = \Sigma X Y - N X Y$$

$$\hat{B} = S_{xy}^{X} / S^{2}$$

$$\hat{\alpha} = Y^{xy} - B(X)^{x}$$

where S_{v^2} is the sum of squared deviations from the means of the X

values, S is the sum of the cross products of deviations, and B and α xy are the least square estimates of the slope (b) and y-intercept (a), respectively for the regression line. An example is included in Section

9.F.3.e.7. Correlation Coefficient and Coefficient of Determination

The sample correlation coefficient, r, is a useful statistic to define how well a linear regression fits a straight line. The correlation coefficient is always a value between -1 and +1. The two extreme values of -1 and +1 are approached as the relationship between the two variables approaches a straight line with a negative or positive slope, respectively. If r = 0, then there is no <u>linear</u> relationship between the two variables. The correlation coefficient is easily determined on most statistical calculators, or the following equation can be used to determine r for variables X and Y:

$$r = \underbrace{\Sigma XY - N XY}_{\sqrt{(\Sigma x^2 - \Sigma NX^2)(Y^2 - \Sigma NY^2)}}$$

An example can be found in Section 9.F.4.

The coefficient of determination, r^2 , is usually preferred to explain the strength of association between X and Y when using regression analysis.

9.F.3.e.8. Pooling Estimates of Standard Deviation

As new data are collected, it may be possible to improve existing estimates of the standard deviation by pooling (combining) the existing data set and the new data set. However, before such data can be pooled, it

9/94

is first necessary to verify that there has not been a significant change in variability between the two data sets. This can be done statistically by comparing the ratio of the two variance estimates (si^2/s_2) to appropriate values of the F distribution. If data sets are of similar size and variability, the two data sets can be pooled. However, if the variability of a procedure has changed, the procedure should be carefully reviewed to determine the cause. When it appears that variability has not changed, a pooled estimate of variability may be calculated.

The pooling method consists of attaching the proper amount of statistical "weight" to the two variance estimates based on the degrees of freedom of the respective data sets, summing the weighted variance estimates, and dividing the sum by the sum of the degrees of freedom associated with the two estimates. The quotient which results is the pooled estimate, s², from which the new pooled estimate of the standard deviation, s, is obtained. The following equation is used to calculate pooled standard deviation.

$$s^{2}(pooled) = \frac{(n_{1}-1) s_{1}^{2} + (n_{2}-1) s_{2}^{2}}{(n_{1} + n_{2} - 2)}$$

9.F.4. EXAMPLES

9.F.4.a. Introduction

Usually in the laboratory, unknown population parameters are used. Because of this, the factors used in the following examples will be for unknown populations. Table 9.E.-5, Factors for Calculating Control Limits, contains a summary of necessary factors for up to n = 10 where n is the number of observations per subgroup. Factors for larger values of n and for known populations are available in other references.

Also, when working with ranges and standard deviations, lower control limits may also be used. For tests with significant variability, a low (or zero) range or standard deviation may be significantly different from the expected performance. Lower control limits for ranges and standard deviations are deleted from the following examples to facilitate QC application.

9.F.4.b. Data Sets

Standard data sets have been designed and established to be used for examples with the various application techniques. Although real data sets are sometimes desirable, they can frequently be limited in their applicability.

Table 9.F.-2, Ordered Paired Replicate Values, contains 100 pairs of computer generated values. They have been arranged by increasing mean

TABLE 9.F.-2

ORDERED PAIRED REPLICATE VALUES

Order <u>No.</u>	Initial Value	Replicate Value	Mean	Range	Order No.	Initial Value	Replicate Value	Mean	Range
,	0.00	,	0 005						
1	-0.96	1.3/	0.205	2.33	51	26.65	33.74	30.195	7.09
2	1.25	0.78	1.015	0.4/	52	28.67	32.05	30.36	3.38
3	2.50	2.65	2.575	0.15	53	29.37	31.60	30.485	2.23
4	2.88	3.60	3.24	0.72	54	34.19	27.59	30.89	6.60
2	/.85	6.06	6.96	1.80	55	32.14	30.22	31.18	1.92
0 7	5.48	9.//	7.625	4.29	56	31.31	31.06	31.185	0.25
/	10.37	10.84	10.605	0.47	57	30.43	32.57	31.50	2.14
8	12.40	12.69	12.545	0.29	58	29.37	34.12	31.745	4.75
9	13.94	11.83	12.885	2.11	59	34.91	28.60	31.755	6.31
10	13.33	12.64	12.985	0.69	60	34.81	28.73	31.77	6.08
11	13.70	13.17	13.435	0.53	61	32.31	31.75	32.03	0.56
12	13.99	13.14	13.565	0.85	62	35.82	28.77	32.295	7.05
13	16.59	13.51	15.05	3.08	63	36.35	28.51	32.43	7.84
14	13.81	20.36	17.085	6.55	64	32.09	32.98	32.535	0.89
15	16.53	17.75	17.14	1.22	65	34.11	31.35	32.73	2.76
16	16.84	18.26	17.55	1.42	66	29.56	36.29	32.925	6.73
17	16.24	20.54	18.39	4.30	67	33.27	32.96	33.115	0.31
18	16.98	20.02	18.50	3.04	68	35.44	30.90	33.17	4.54
19	17.72	21.35	19.535	3.63	69	33.61	33.43	33.52	0.18
20	17.30	22.32	19.81	5.02	70	32.73	34.55	33.64	1.82
21	19.22	22.04	20.63	2.82	71	36.02	31.76	33.89	4.26
22	21.27	23.13	22.20	1.86	72	33.43	34.61	34.02	1.18
23	21.02	23.72	22.37	2.70	73	34.67	34.11	34.39	0.56
24	23.07	22.10	22.585	0.97	74	31.76	39.35	35.555	7.59
25	20.13	25.41	22.77	5.28	75	35.82	35.79	35.805	0.03
26	24.72	21.76	23.24	2.96	76	31.81	41.06	36.435	9.25
27	23.32	23.93	23.625	0.61	77	32.46	41.08	36.77	8.62
28	23.29	24.55	23.92	1.26	78	37.14	36.86	37.00	0.28
29	23.46	25.04	24.25	1.58	79	37.46	38.22	37.84	0.76
30	23.92	25.72	24.82	1.80	80	36.67	39.25	37.96	2.58
31	25.11	24.69	24.90	0.42	81	39.26	37.08	38.17	2.18
32	26.98	22.92	24.95	4.06	82	41.52	35.18	38.35	6.34
33	25.66	25.02	25.34	0.64	83	37.11	40.95	39.03	3.84
34	28.82	22.85	25.835	5.97	84	40.96	37.19	39.075	3.77
35	29.14	22.79	25.965	6.35	85	42.66	38.33	40.495	4.33
36	26.27	27.22	26.745	0.95	86	37.37	44.21	40.79	6.84
37	26.81	28.00	27.405	1.19	87	46.94	35.50	41.22	11.44
38	25.06	29.94	27.50	4.88	88	39.79	44.32	42.055	4.53
39	26.50	29.04	27.77	2.54	89	40.58	44.07	42.325	3.49
40	27.80	29.57	28.685	1.77	90	39.81	46.27	43.04	6.46
41	35.11	23.19	29.15	11.92	91	43.35	44.15	43.75	0.80
42	28.37	29.96	29.165	1.59	92	49.41	44.59	47.00	4.82
43	26.66	31.68	29.17	5.02	93	48.46	47.50	47.98	0.96
44	25.88	32.48	29.18	6.60	94	44.41	52.12	48.265	7.71
45 ·	28.71	29.79	29.25	1.08	95	48.93	48.92	48,925	0.01
46	29.09	29.41	29.25	0.32	96	49.32	51.29	50.305	1,97
47	33.00	25.84	29.42	7.16	97	54.87	46.19	50.53	8.68
48	30.79	28.08	29.435	2.71	98	52.75	50.33	51.54	2.42
49	33.26	26.00	29.63	7.26	99	51.07	54.25	52.66	3,18
50	24.78	34.52	29.65	9.74	100	49.94	56.19	53.065	6.25

2/94

1

values. Design criteria for the set stipulated that range increased with increasing values.

Table 9.F.-3, Large Univalued Data Set, contains 200 computer generated values with a design mean of 55, a design standard deviation of 2.0, and a normal distribution of values. The values were then adjusted to simulate two potential data problems. The variate used to establish values 116 through 140 was increased by 2.0 to simulate a step error of two standard deviation. The variate used to establish values 171 through 200 was gradually increased to simulate a gradual shift in data (variate used = design variate + 0.1 (sample value no. - 170)).

9.F.4.c. Example 1: Paired Replicate Values

Our first example will be working with the data found in Table 9.F.-2, Ordered Paired Replicate Values. The data is typical of duplicate values generated to determine precision. As previously mentioned, it is desirable to have 50 or more data pairs before an evaluation can be made by the following technique. Other values for factor D₄ should be used if n is less than 50.

9.F.4.c.1. Mean Range

The mean range, $R^{\ }$, is simply the sum of the ranges, R, divided by N, or:

$$R = \frac{R}{N} = \frac{345.53}{100} = 3.4553$$

The upper control limit (UCL) for the range can be determined from using the appropriate D₄ factor from Table 9.E.-5. Because we are determining ranges from duplicate values (n = 2) D₄ = 3.267. The upper control limit for the range is:

$$UCL_{R} = D_{4} R = 3.267 x 3.4553 = 11.29$$

Review of the data pairs in our sample (Table 9.F.-2) would indicate that the control limit was exceeded with set no. 41 (R = 11.92) and set no. 87 (R = 11.44). If these values are discharged as outliers, the new R = 3.2874 and the new UCL = $3.267 \times 3.2874 = 10.74$. The recalculated control limit is slightly tighter (about 5 percent) than the original.

9.F.4.c.2. Segmenting the Sample

If there is sufficient data (at least 100 sets), it may be feasible to group the pairs into data segments. The pairs in Table 9.F.-2 have been ordered sequentially by increasing means to facilitate observation. With many analytical systems, the range of replicate analyses tends to increase as the level of the analyte increases. Segmenting data will show whether this is the case for a data set.

Segments can be established by using an established frequency, n, per segment or by setting an interval for the means. Enough segments to differentiate significant increases in range are desirable without decreasing the frequency, N, too much per segment. We will evaluate the data in Table 9.F.-2 using five segments.

TABLE 9.F.-3

LARGE UNIVALUED DATA SET

<u>No.</u>	Value	<u>No.</u>	Value	<u>No.</u>	Value	No.	Value
1	55.4	51	58.3	101	53.3	151	55.5
2	55.7	52	55.9	102	54.9	152	56.7
3	55.7	53	55.8	103	53.0	153	53.7
4	54.3	54	57.4	104	56.4	154	54.9
5	54.5	55	55.7	105	55.2	155	60.0
6	53.7	56	53.6	106	53.6	156	52.7
7	56.6	57	53.7	107	57.3	157	52.0
8	51.7	58	54.9	108	57.2	158	57.8
9	52.2	59	58.3	109	56.0	159	53.7
10	53.6	60	54.3	110	55.2	160	56.3
11	60.2	61	52.3	111	57.8	161	53.6
12	53.5	62	55.7	112	53.1	162	55.5
13	54.9	63	56.2	113	56.2	163	57.0
14	55.5	64	54.9	114	56.5	164	54.5
15	55.2	65	55.8	115	54.7	165	54.1
16	52.1	66	54.7	116	57.7	166	54.8
17	56.6	67	52.4	117	60.1	167	53.3
18	54.0	68	53.8	118	58.3	168	56.6
19	56.3	69	56.3	119	58.7	169	51.4
20	55.6	70	51.6	120	57.6	170	57.6
21	56.7	71	57.2	121	56.8	171	54 9
22	55.2	72	54.8	122	57.7	172	56 1
23	54.8	73	58.9	123	58.4	173	52 1
24	55.0	74	53.4	124	60.4	174	55.6
25	54.7	75	52.0	125	60.9	175	54.5
26	52.4	76	58.0	126	59.7	176	50 5
27	55.3	77	55.0	127	56.2	177	60.4
28	51.1	78	58.9	128	58.5	178	58 4
29	55.1	79	58.5	129	61.3	179	59.4
30	52.7	80	51.5	130	60.8	180	59.2
31	59.0	81	52.7	131	61.4	181	58.4
32	55.4	82	55.4	132	57.6	182	60.0
33	56.3	83	52.3	133	61.4	183	63.1
34	51.6	84	53.1	134	58.9	184	61 1
35	56.1	85	54.7	135	54.7	185	60.3
36	52.8	86	56.0	136	59.7	186	57.9
37	59.7	87	56.1	137	59.2	187	58.8
38	56.5	88	55.5	138	57.1	188	60.4
39	54.1	89	54.9	139	57.6	189	59 2
40	52.6	90	57.5	140	58.4	190	59 6
41	56.9	91	54.8	141	54.3	191	59.1
42	53.2	92	55.3	142	57.6	192	57.7
43	55.2	93	54.2	143	56.3	193	59.6
44	54.7	94	54.2	144	58.1	194	64.9
45	53.8	95	53.0	145	55.9	195	58.5
46	56.6	96	55.2	146	53.4	196	60.4
47	52.3	97	54.3	147	53.9	197	57.2
48	57.5	98	56.2	148	52.5	198	62.4
49	56.8	99	51.9	149	58.3	199	60.7
50	56.2	100	57.8	150	53.9	200	63.0

٠.

If the data is separated into five segments with N = 20, the following summary is obtained:

Segment	Set Nos.	<u>Grand Mean (X)</u>	<u>Mean Range (R)</u>
1	1- 20	12.035	2.148
2	21- 40	24.775	2.530
3	41- 60	30.218	4.708
4	61- 80	34.403	3.390
5	81-100	44.928	4.5010

There does appear to be an increase in range as analyte level increases. However, the mean range for segment 3 is a little high compared to other segments.

If the data is segmented into five mean intervals of about ten units, the following summary is obtained:

Segment	<u>Set Nos.</u>	<u>Mean Interval</u>	<u>Grand Mean (X)</u>	<u>Mean Range (R)</u>	UCLR
1	1- 6	10.00	3.603	1.627	5.32
2	7- 20	10.00 to 19.99	9 15.648	2.371	7.75
3	21- 50	20.00 to 29.99	9 26.294	3.467	11.33
4	51- 84	30.00 to 39.99	9 33.934	3.667	11.98
5	85-100	40.00	46.497	4.618	15.09

The mean ranges in the above summary do increase with each increasing segment, but the number of sets (N = 6, 14, and 16, respectively) is quite small for segments 1, 2, and 5. Using the range control limits from the above example for segmented data would allow for better quality control than using a single control limit for range of all data. Also, with the new segmented control limits, set no. 87 is no longer out-of-control (set no. 41 is still out-of-control).

9.F.4.c.3. Line of Regression

Because the above evaluation indicates an increase in range with increasing mean values, it is worthwhile to try to define the line of regression associated with their relationship. This also provides an opportunity to demonstrate the equations given in Sections 9.F.3.e.6., Linear Regression, and 9.F.3.e.7., Correlation Coefficient and Coefficient of Determination.

In linear regression analysis, the two variables must be defined first. Let's compare the mean range (variable Y) to the grand mean (variable X) of each segment. From our last example:

Seqment	<u>X (grand mean)</u>	<u>Y (mean range)</u>	X 2	XY
1	3.603	1.627	12.981609	5.862081
2	15.648	2.371	244.859904	37.101408
3	26.294	3.467	691.374436	91.161298
4	33.934	3.667	1151.516356	124.435978
5	46.497	4.618	2161.971009	214.723146
	125.976	15.750	4262.703314	473.283911
	X 125.976/5 = 25	.1952		
	Y = 15.750/5 = 3.	150		

s ² = ΣX^2 - n X^2 = 4262.703314 - 5(25.1952)² = 4262.703314 - 3173.9905152 = 1088.7127988

 $s_{xy} = \Sigma xy - n \ xy = 473.283911 \ 5(25.1952)(3.150) = 473.283911$ - 396.8244 = 76.459511 $B = s_{xy}/s_{x}^{2} = 76.459511/1088.7127988 = 0.070229$ $\hat{\alpha} = Y - B(X) = 3.150 - 0.070229(25.1952) = 1.3806$

The same least square estimates for the slope and y-intercept are obtained from a statistical calculator (with considerably less effort).

The line of regression from above is: $R = 1.38 + 0.0702 \text{ x}^{-1}$. A control limit for the range based on the above line (multiply by 3.267) would be UCL = $4.51 + 0.229 \text{ X}^{-1}$. Again, only data set no. 41 is out-of-control ($R \stackrel{B}{=} 11.92$ versus UCL = 4.51 + .229(29.15) = 11.19).

The correlation coefficient is next calculated to determine how well the data fits a straight line. First, calculate the sum of Y':

Segment	Y (mean range)	<u> </u>
1	1.627	2.647129
2	2.371	5.621641
3	3.467	12.020089
4	3.667	13.446889
5	4.618	21.325924
_		55,061672

Now, calculate the correlation coefficient as:

 $r = \underbrace{\sum XY - N XY}_{r}$ $r = \underbrace{- \\ \sqrt{(x^2 - N X^2)(Y^2 - N Y^2)}}_{\frac{473.283911 - (5)(25.1952)(3.150)}{\sqrt{(4262.703314 - 5(25.1952)^2)(55.061672 - 5(3.150)^2)}}_{\frac{76.459511}{5932.583}} = \underbrace{\frac{76.569511}{77.023265}}_{\frac{77.023265}{2}}$

The coefficient of determination, r^2 , is 0.9854 indicating that the points defining the line of regression fit the line well.

Least squares lines of regression can also be calculated from the previous example with five segments of N = 20 and from individually comparing each range to its mean value. Results from all three techniques are:

Technique		<u>y - int. (α)</u>	<u>slope (B)</u>	<u> </u>
5 segments from intervals	of means	1.38	0.0702	0.9927
5 segments of $N = 20$		1.28	0.0745	0.7907
individual range versus me	an, $N = 100$	1.37	0.0713	0.2920

Although the correlation coefficient is very low when all values are entered individually (r = 0.2920), the estimated line of regression agrees well with the other two methods. This may not always be the case. Because the line of regression attempts to fit the most points, the line will not adequately define the whole range unless the points are fairly equally distributed over the full range of the test.

9.F.4.d. Example 2: Univalued Audit

Frequently, an audit may be analyzed once with each batch of samples. If the audit values are not expected to change with time, a reproducible sample mean and standard deviation should be obtained.

9.F.4.d.1. Mean and Standard Deviation

To establish control limits for an univalued audit, first calculate the mean and standard deviations for the first 50 values in Table 9.F.-3 (X = 54.952 and s = 2.0016). The control limits are then X \pm 3s_or from 48.95 to 60.96. Warning limits, if they are to be used, are X \pm 2s, or from 50.95 to 58.96.

If an out-of-control situation is defined by a single excursion outside the control limits or three consecutive excursions beyond the warning limits, the first out-of-control situation occurs with values 124-126. These all exceed the warning limits (remember that the ordinate values for 116-140 have been raised by 2.0).

9.F.4.d.2. Moving Average and Range

Precision can be maintained on a single value by pairing consecutive results. If the first twenty values in Table 9.F.-3 are paired, the following results are obtained:

<u>Pair</u>	x ₁	x ₂	Х	<u>R</u>
1	55.4	55.7	55.55	0.3
2	55.7	55.7	55.7	0.0
3	55.7	54.3	55.0	1.4
4	54.3	54.5	54.4	0.2
5	54.5	53.7	54.1	0.8
6	53.7	56.6	55.15	2.9
7	56.6	51.7	54.15	4.9
8	51.7	52.2	51.95	0.5
9	52.2	53.6	52.9	1.4
10	53.6	60.2	56.9	6.6
11	60.2	53.5	56.85	6.7
12	53.5	54.9	54.2	1.4
13	54.9	55.5	55.2	0.6
14	55.5	55.2	55.35	0.3
15	55.2	52.1	53.65	3.1
16	52.1	56.6	54.35	4.5
17	56.6	54.0	55.3	2.6
18	54.0	56.3	55.15	2.3
19	56.3	55.6	55.96	0.7

X = 54.8316 R = 2.1684

The control limit on the moving average is:

= $x^{=} \pm A_2 R$ = 54.8316 ± 1.880(2.1684) = 54.8316 ± 4.0766 or 50.755 X 58.908 The control limit on individual values is: = $x^{=} \pm E_2 R$ = 54.8316 ± 2.660(2.1684) = 54.8316 ± 5.7679 or 49.064 < X < 60.600

The control limit on the moving range is:

9.F.5. CONTROL CHARTS

9.F.5.a. Introduction

A useful tool in a quality assurance program is control charting. It is best applied when a laboratory is running the same analysis on many samples over a period of time. Basically, the technique indicates when a measurement process in a state of statistical control has moved out of statistical control.

A control chart is a graphical chart with the vertical scale plotted in units of the test result and the horizontal scale in units of time or sequence of results. A central horizontal line represents the average or sequence of results. A central horizontal line represents the average or the standard value of the control sample value being plotted. Upper and lower control lines (limits) are drawn on the chart above and below the central horizontal line as criteria for action or for judging the significance of variations from the central line. Routine precision and accuracy data are plotted on the control chart to monitor, evaluate and document data quality trends. Examples of charts used to monitor precision and accuracy are included in Figure 9.F.1.

9.F.5.b. Control Chart Development

To set up a control chart, measurements must be gathered when the process is in control. This means that the analyst must have explored the possible sources of error and done sufficient validation to be reasonably sure that results are acceptable. After the method has been validated and is ready for use on actual samples, control measurements should be collected. The recommended number of data points is described in 9.F.3.c. Testing for outliers is described in 9.F.3.d. Section 9. \underline{F} .4 provides examples of control limit calculations for sample means (x) and the mean difference between paired replicate values (R).

9.F.5.c. Interpretation of Control Charts

Control charts and control limits are set up to detect analytical errors including random error, systematic shifts, systematic drifts and mixtures of these three. The ability or probability for error detection is dependent on the number of control observations and the choice of control rules. Commonly used control rules include:

- One observation outside three standard deviations (1:3s)
- One observation outside two standard deviations (1:2s)
- Two consecutive observations outside two standard deviations (2:2s)
- Four consecutive observations outside one standard deviation (4:1g)
- Ten consecutive control observations on one side of the mean (10x)

No single rule is best for detecting all errors, combinations of rules are desirable.

For detection of random analytical error, control rules based on a single control observation exceeding a specified limit (1:3s) generally have a higher probability for error detection than those which require more than one consecutive observation exceeding a specific limit. For detecting systematic errors, rules which require a series of consecutive observation's exceeding the same control limit (such as 2:2s, 4:1s and 10x rules) have a high probability of correctly signaling an out of control situation.

Many internal quality control systems in operation today use 1:3s and 1:2s rules, the first as an action (control) limit and the second as a warning limit. When a control observation exceeds an action limit, the

process is stopped, the problem is investigated and corrected and then the run is repeated. When a control observation exceeds a warning limit, it is not always clear what should be done. Many laboratories automatically repeat those runs or at least the control samples. however, since this may require repeating 10% of all routine work (assuming 2 control samples per run) this becomes an expensive way to respond to a warning limit. An alternative way of interpreting the warning limit is to suspend data reporting until a more intensive inspection can be made of the control data. For example, data reporting can be stopped until a second control observation is obtained. If the second observation exceeds the same 2s limit, then the 2:2s and 1:3s rules can be used to determine rejection. Another valuable combination is to use the 1:2s rule to suspend data reporting, followed by inspection of past data to see if rejection is justified by the 4:1s or the 10x rules, then inspection of future data to determine rejection by either of these rules, or by the 2:2s and 1:3s.

There are many factors that could lead to problems in an analysis which would show up on the control chart. Most factors are specific to instrumentation and methodology. The basic symptoms are either shifts or trends. When a system is judged out of control, the analyst is responsible for assessing and correcting the problem.

Figure 9.F.-1. Quality Control Chart Examples. UCL and LCL refer to Upper and Lower Control Limits, UWL and LWL refer to Upper and Lower Warning Limits, CL and WL refer to Control and Warning Limits.



a. Accuracy chart based on differeneces from the mean (\overline{x}) .



b. Precision chart based on the mean range (\overline{R}) or mean difference between duplicates.

2/94

E

a,

PARAMETERS	PRECISION	ACCURACY			CALIBRATION	
Sample	Within-Run Titrant	Sample/Standard		Check	Reagent	
Water Matrix Check	Duplicate	Mixture	Control Sample	Standard	Blank	Blank
_	······································					
Chlorophyll <u>a</u>	1 per 10 samples	footnote 2				
Dissolved oxygen Ea.batch	l per 15 samples 1 per 15 samples	footnote 2			,	
Chemical oxygen demand-color Chemical oxygen demand-titr. Total organic carbon	1 per 15 samples 1 per 15 samples 1 per 15 samples	1 per 15 samples 1 per 15 samples 1 per 15 samples		1	Each batch ₁ Each batch ₁ Each batch ₁	
Nitrate plus nitrite Nitrite	1 per 15 samples 1 per 15 samples	1 per 15 samples 1 per 15 samples 1 per 15 samples		Each batch ₁ Each batch ₁ Each batch ₁	Each batch ₁ Each batch ₁ Each batch ₁	
Kjeldahl nitrogen Orthophosphate-P	1 per 15 samples 1 per 15 samples 1 per 15 samples	1 per 15 samples 1 per 15 samples 1 per 15 samples		Each batch ₁ Each batch ₁ Each batch	Each batch ₁ Each batch ₁ Each batch	
Total Phosphorus Reactive Silicates	1 per 15 samples 1 per 15 samples	1 per 15 samples 1 per 15 samples 1 per 15 samples		Each batch Each batch	Each batch ₁ Each batch ₁	
Sulfide sample	1 per 15 samples	1 per 15 samples		1	Each batch	Each
Phenolics-automated Phenolics-manual	1 per 15 samples 1 per 18 samples	l per 15 samples 1 per 18 samples		Each batch <mark>1</mark> Each batch1	Each batch1 Each batch1	
Cyanide-automated Cyanide-manual	l per 15 samples 1 per 15 samples	1 per 15 samples 1 per 15 samples		Each batch1 Each batch	Each batch1 Each batch	
Cyanide (RCRA) Res. Non Filterable (SS)	1 per 10 samples 1 per 10 samples	footnote 2				
Res. Non-Filt. Vol (SVS) Res. Total Settleable (Set.S)	l per 10 samples 1 per 10 samples	footnote 2 footnote 2				
Res. Total Filterable (TDS) Res. Total (TS) Fecal Coliform	1 per 18 samples 1 per 10 samples 1 per 10 samples	1 per 18 samples 1 per 10 samples footnote 2		footnote 3		
Total Coliform Fecal Streptococci	1 per 10 samples 1 per 10 samples 1 per 10 samples	footnote 2 footnote 2 footnote 2		footnote 3 footnote 3		
Turbidity Biochemical Oxygen Demand	l per 10 samples 1 per 15 samples	footnote 2 footnote 2	.'	3 per batch ¹	2 per batch ¹	

Table 9.E.-2 General Chemistry Unit Routine Quality Control Audits and Frequencies. (Unless otherwise stated, inclusion of an audit frequency means that control limits are included in the method.)

2404

5276

9/q4

.
PARAMETERS	PRECISION	ACCURACY			CALIBRATION	
Sample	Within-Run	Sample/Standard		Check	Reagent	
Water Matrix Check	Duplicate	Mixture	Control Sample	Standard	Blank	Blank
SEDIMENT MATRIX						
Chemical oxygen demand	1 per 15 samples		Each batch		Each batch ₁	
Kjeldahl nitrogen	1 per 15 samples		Each batch ₁		Each batch	
Total phosphorus	l per 15 samples		Each batch	1	Each batch	
Phenolics	l per 15 samples		Each batch1	Each batch	Each batch	
Cyanide	1 per 15 samples		Each batch	Each batch	Each batch	
Cyanide (RCRA)	1 per 10 samples					
Solids, Total (% TS)	1 per 10 samples	footnote 2				
Solids, Volatile (% TVS)	l per 10 samples	footnote 2				

A batch is one or more samples analyzed at a given time or on a given day, depending on the system. Calibration frequency is 1/10 sample measured or 1/batch, whichever is more frequent.

 $^{2}\,$ A accuracy audit is not practical due to the absence of an acceptable standard.

³ A blank filter is analyzed at the beginning of each series to check the sterility of the media, rinse water and apparatus.

9/94

1