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EPT Field Report

**WATER QUALITY LABORATORY TRAINING
MANUAL SUMMARY: Sanitary and Epidemiological
Services Laboratory,
Kzyl-Orda, Kazakhstan**

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Environmental Policy and Technology Project

Water Quality Laboratory Training Manual

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INTRODUCTION

INTRODUCTION

The laboratory equipment, materials and training provided are part of the activities included in the Memorandum of Understanding executed on 18 March 1994 between the Government of the United States and the Government of Kazakhstan. These activities are part of the Aral Sea Program financed by the Government of the United States through the U.S. Agency for International Development (USAID) and implemented by the Environmental Policy and Technology Project (EPT).

The Aral Sea Program is intended to provide technical assistance and material for improvements in potable water quality through a series of activities that include water monitoring improvements (laboratory equipment and materials), water quality improvements (chlorination equipment), water delivery improvements (pump station pumps and motors), and educational improvements (public health and sanitation program.) This Training Manual summary has been prepared for the technical training associated with the provision of laboratory equipment and materials. This summary is organized in as follows:

1. Introduction - This section presents an overview of the training and equipment provided and includes a list of the instruments and parameters that can be analyzed.
2. Basic laboratory practices - This section provides a summary of the basic laboratory practices for water quality testing. A discussion is included on the various techniques for verification of the analytical accuracy and precision.
3. Water sampling, storage and pretreatment - The laboratory analysis of water is the final step in the determination of water quality. Appropriate sampling is required to insure the representative analysis of the water source; and storage and pretreatment is necessary to insure inadvertent contamination.
4. Chemical and analytical procedures - The laboratory equipment is provided for the the analysis of specific water quality parameters, and is the provides the basic analyses of water. Analytical procedures are discussed in relation to the importance in water quality.
5. Inventory of equipment and manuals - A detailed inventory of the laboratory equipment is provided including the reference numbers from the manufacturer for all items. A list of the equipment manuals provided is included.
6. International guidelines for water quality - The present standards in use in Kazakhstan are the GOST standards promulgated in 1982, and these standards are under revision. This section includes the World Health Organization guidelines for microbiological and aesthetic quality. Also included is a table of the comparison of selected international drinking water guidelines.

This section includes two tables of the laboratory analytical procedures for the equipment provided - one listed by instrument provided and the other listed by analytical parameter.

Water Quality Laboratory Analytical Procedures (Listed by Analytical Parameter)

Parameter	Range	Analytical Method/Reference Pages	Instrument Method
Alkalinity (*)	10-4000 mg/L as CaCO ₃	Method 8203, pages 1-5 (DREL Lab Manual)	Digital Titrator
Bromine	0-4.5 mg/L as Br ₂	Method 8016, pages 79-80, 83-84 (Supplement Methods)	DR/2000
Carbon Dioxide	10-1000 mg/L as CO ₂	Method 8205, pages 7-9 (DREL Lab Manual)	Digital Titrator
Chloride	10-10,000 mg/L as Cl ⁻	Method 8207, pages 11-14 (DREL Lab Manual)	Digital Titrator
Chlorine, Free (*)	0-2 mg/L as Cl ₂	Method 8021, pages 15-16, 19-20 (DREL Lab Manual)	DR/2000
Chlorine, Free (*)	0-2 mg/L as Cl ₂	Method 8021, pages 19-22, 26-28 (DREL Lab Manual)	Pocket Colorimeter
Chlorine, Total, High Range (*)	0-4.5 mg/L as Cl ₂	Method 8167, pages 37-43 (DREL Lab Manual)	Pocket Colorimeter
Chlorine, Total, Low Range (*)	0-2 mg/L as Cl ₂	Method 8167, pages 21-22, 25-26 (DREL Lab Manual)	DR/2000
Chlorine, Total, Low Range (*)	0-2 mg/L as Cl ₂	Method 8167, pages 29-32, 26-28 (DREL Lab Manual)	Pocket Colorimeter
Chromium, Hexavalent	0-0.60 mg/L as Cr ⁶⁺	Method 8023, pages 27-28, 31 (DREL Lab Manual)	DR/2000
Conductivity (*)	0.2-4.0 mS/cm	Pocket Pal Method, pages 33-36 (DREL Lab Manual)	Pocket Conductivity/TDS Meter
Conductivity (*)	0.2-4.0 mS/cm	Pages 10-13 (Portable Conductivity/TDS Instrument Manual)	Portable Conductivity/TDS Meter
Copper, Complexed & Free	0-5 mg/L as Cu Bicn	Method 8506, pages 3738, 41-42 (DREL Lab Manual)	DR/2000
Dissolved Oxygen	1-10+ mg/L DO	Method 8332, pages 79-82 (DREL Lab Manual)	Digital Titrator
Fluoride	0-2 mg/L as F ⁻	Method 8029, pages 43-44, 47-48 (DREL Lab Manual)	DR/2000
Hardness	10-4000 mg/L as CaCO ₃	Method 8213, pages 49-53 (DREL Lab Manual)	Digital Titrator
Iodine	0-7 mg/L as I ₂	Method 8031, pages 207-208, 211 (Supplemental Methods)	DR/2000
Iron, Total	0-3 mg/L as Fe FV	Method 8008, pages 55-56, 59-60 (DREL Lab Manual)	DR/2000
Nitrogen, Ammonia	0-0.50 mg/L	Method 8155, pages 71-75 (DREL Lab Manual)	DR/2000
Nitrogen, Nitrate (*)	0-30 mg/L as NO ₃ ⁻ -N	Method 8039, pages 61-63, 66-69 (DREL Lab Manual)	DR/2000
pH (*)	0-14 pH	Method 8156, pages 83-85 (DREL Lab Manual)	Hach One Laboratory pH Instrument
pH (*)	0-14 pH	Method 8156, pages 83-85 (DREL Lab Manual)	Hach One Portable pH Instrument
Phosphorus, Reactive	0-2.5 mg/L as PO ₄ ⁻	Method 8048, pages 89-90, 93-96 (DREL Lab Manual)	DR/2000
Phosphorus, Total	0-2.5 mg/L	Method 8180, pages 87-90, 93-96 (DREL Lab Manual)	DR/2000
Sulfate	0-70 mg/L SO ₄ ⁻	Method 8051, pages 97-98, 100-101 (DREL Lab Manual)	DR/2000
Total Dissolved Solids	10-1999 mg/L TDS	Pocket Pal Method, pages 33-36 (DREL Lab Manual)	Pocket Conductivity/TDS Meter
Total Dissolved Solids	10-1999 mg/L TDS	Pages 10-13 (Portable Conductivity/TDS Instrument Manual)	Portable Conductivity/TDS Meter
Turbidity (*)	0-1000 NTU	Pages 4-13 (2100N Instrument Manual)	2100N Turbidimeter Instrument
Turbidity (*)	0-1000 NTU	Pages 103-104 (2100P Instrument Manual)	2100P Turbidimeter Instrument
Total Coliform (*)		Method 8074, pages 19-24, 38-40 (MEL/MF Laboratory Manual)	MEL/MF Laboratory

(*) Denotes Analytical Parameter Being Supplied With Extended Quantity of Reagents

Water Quality Laboratory Analytical Procedures (Listed by Instrument Method)

Instrument Method	Parameter	Range	Analytical Method/Reference Pages
2100N Turbidimeter Instrument	Turbidity (*)	0-1000 NTU	Pages 4-13 (2100N Instrument Manual)
2100P Turbidimeter Instrument	Turbidity (*)	0-1000 NTU	Pages 103-104 (2100P Instrument Manual)
Digital Titrator	Alkalinity (*)	10-4000 mg/L as CaCO ₃	Method 8203, pages 1-5 (DREL Lab Manual)
Digital Titrator	Carbon Dioxide	10-1000 mg/L as CO ₂	Method 8205, pages 7-9 (DREL Lab Manual)
Digital Titrator	Chloride	10-10,000 mg/L as Cl ⁻	Method 8207, pages 11-14 (DREL Lab Manual)
Digital Titrator	Dissolved Oxygen	1-10+ mg/L DO	Method 8332, pages 79-82 (DREL Lab Manual)
Digital Titrator	Hardness	10-4000 mg/L as CaCO ₃	Method 8213, pages 49-53 (DREL Lab Manual)
DR/2000	Bromine	0-4.5 mg/L as Br ₂	Method 8016, pages 79-80, 83-84 (Supplement Methods)
DR/2000	Chlorine, Free (*)	0-2 mg/L as Cl ₂	Method 8021, pages 15-16, 19-20 (DREL Lab Manual)
DR/2000	Chlorine, Total, Low Range (*)	0-2 mg/L as Cl ₂	Method 8167, pages 21-22, 25-26 (DREL Lab Manual)
DR/2000	Chromium, Hexavalent	0-0.60 mg/L as Cr ⁶⁺	Method 8023, pages 27-28, 31 (DREL Lab Manual)
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DR/2000	Nitrogen, Nitrate (*)	0-30 mg/L as NO ₃ -N	Method 8039, pages 61-63, 66-69 (DREL Lab Manual)
DR/2000	Phosphorus, Reactive	0-2.5 mg/L as PO ₄ ⁻⁻⁻	Method 8048, pages 89-90, 93-96 (DREL Lab Manual)
DR/2000	Phosphorus, Total	0-2.5 mg/L	Method 8180, pages 87-90, 93-96 (DREL Lab Manual)
DR/2000	Sulfate	0-70 mg/L SO ₄ ⁻⁻	Method 8051, pages 97-98, 100-101 (DREL Lab Manual)
Hach One Laboratory pH Instrument	pH (*)	0-14 pH	Method 8156, pages 83-85 (DREL Lab Manual)
Hach One Portable pH Instrument	pH (*)	0-14 pH	Method 8156, pages 83-85 (DREL Lab Manual)
MEL/MF Laboratory	Total Coliform (*)		Method 8074, pages 19-24, 38-40 (MEL/MF Laboratory Manual)
Pocket Colorimeter	Chlorine, Free (*)	0-2 mg/L as Cl ₂	Method 8021, pages 19-22, 26-28 (DREL Lab Manual)
Pocket Colorimeter	Chlorine, Total, High Range (*)	0-4.5 mg/L as Cl ₂	Method 8167, pages 37-43 (DREL Lab Manual)
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Pocket Conductivity/TDS Meter	Conductivity (*)	0.2-4.0 mS/cm	Pocket Pal Method, pages 33-36 (DREL Lab Manual)
Pocket Conductivity/TDS Meter	Total Dissolved Solids	10-1999 mg/L TDS	Pocket Pal Method, pages 33-36 (DREL Lab Manual)
Portable Conductivity/TDS Meter	Conductivity (*)	0.2-4.0 mS/cm	Pages 10-13 (Portable Conductivity/TDS Instrument Manual)
Portable Conductivity/TDS Meter	Total Dissolved Solids	10-1999 mg/L TDS	Pages 10-13 (Portable Conductivity/TDS Instrument Manual)

(*) Denotes Analytical Parameter Being Supplied With Extended Quantity of Reagents

BASIC LABORATORY PRACTICES

BASIC LABORATORY PRACTICE

Boiling Aids

Boiling is included as a necessary step in some procedures. It may be convenient to use a boiling aid such as boiling chips, Cat. No. 14835-31, to reduce bumping. Bumping is caused by the sudden, almost explosive conversion of water to steam as it is heated. Bumping may cause sample loss or a hazardous condition and should be avoided.

All boiling aids used should be checked to verify they will not contaminate the sample. Boiling aids (except glass beads) should not be used again. Loosely covering the sample during boiling will prevent splashing, reduce the possibility of contamination and minimize sample loss.

Filtration of Samples

Filtering is the process of separating particles from the sample by using a medium, generally filter paper, to retain particles but allow the solution to pass through. This is especially helpful when sample turbidity interferes with calorimetric analysis. Two general methods of filtration are gravity and vacuum. Gravity filtration uses the force of gravity to pull the sample through the filter paper. Vacuum filtration uses the pressure difference created by either an aspirator or vacuum pump plus the force of gravity to move the sample through the filter. Vacuum filtration is faster than gravity filtration. Vacuum filter procedure is as follows:

1. Place a filter paper into the filter holder
2. Place the filter holder assembly in the filtering flask and wet the filter with deionized water to ensure adhesion to the holder.
3. Position the funnel housing on the filter holder assembly.
4. While applying a vacuum to the filtering flask, transfer the sample to the filtering apparatus.
5. Slowly release the vacuum from the filtering flask and transfer to another container.

Required Apparatus

Description	Unit	Cat. No.
Filter Discs, glass 47 mm	100/pkg	2530-00
Filter Holder, membrane	each	2340-00
Flask, filter, 1000 mL	each	546-53

Many of the procedures in this manual may be filtered with gravity filtration. The only labware required is filter paper, a conical funnel and a receiving flask. This labware is included under the Optional Apparatus listing for each procedure. Gravity filtration provides better retention of fine particles. For optimum filtering speed, add solution until the filter paper cone is three-fourths filled. Never fill the cone completely. Gravity filter procedure is as follows:

1. Place filter paper into the funnel.
2. Wet the filter with deionized water to ensure adhesion to the funnel.
3. Place the funnel into an Erlenmeyer flask or graduated cylinder.
4. Pour the sample into the funnel.

Required Apparatus

Description	Unit	Cat. No.
Cylinder, graduated, 100 mL	each	508-42
Funnel, poly, 65 mm	each	1083-67
Filter paper, 12.5 cm	each	1894-57
Flask, erlenmeyer, 125 mL	each	505-43

The determination of metals requires acid and heat to pretreat the sample. Because filter paper will disintegrate under these conditions, vacuum filtration with glass fiber filter discs is recommended. Also, glass filter discs do not retain colored species as filter paper would.

Reagent and Standard Stability

Most chemicals and prepared reagents do not deteriorate after manufacture, but storage conditions and packing have a great influence on their stability. Absorption of moisture, carbon dioxide or other gases from the atmosphere, bacterial action, or light (with photosensitive compounds) may affect the reagent shelf life. In some cases reaction with the storage container or interaction of reagent components may occur.

Hach strives continually to prepare stable formulations and devise ways of packaging them to provide maximum protection. Many unique Hach formulations, methods of analysis and forms of packaging have resulted from these challenges. Chemicals supplied with the DR/2000 Spectrophotometer and DREL/2000 Portable Laboratories have indefinitely long shelf life when stored under average room conditions, unless designated otherwise. Notations on product labels specify any special storage conditions required. Otherwise, reagents should be stored in a cool, dry, dark place for maximum life. It is always good practice to date chemicals upon receipt and rotate supplies so the

Warning labels also appear on some of the apparatus used with the test procedures. The protective shields with the COD Reactor and the Degisdahl Digestion Apparatus point out potential hazardous. Be sure appropriate shields are in place during use and observe the precautions recommended.

Wear protective Clothing: Protective clothing should be worn when handling chemicals that cause irritation or burns. When caustic materials are being used, eye protection, in particular, is important to guard against spattering and splashes from accidental spills.

Use tongs or finger cots when transferring hot apparatus.

Use Mechanical Pipettes: Never pipette by mouth. Mouth pipetting could result in accidentally ingesting a dangerous chemical. Make a habit of using mechanical pipetting devices for all pipetting. Mistakes that could cause serious injury will be avoided.

Use Special Care With Dangerous Chemicals and Apparatus : Follow the test procedure steps carefully and observe all precautionary measures. It is good practice to read the entire procedure carefully before beginning the procedure. Use the safety equipment, such as pipette fillers, protective clothing and ventilating appropriate for the test being conducted. Wipe up all spills promptly. Do not smoke or eat in an area where toxic or irritating chemicals are used. Use reagents and apparatus only as they were meant to be used and use them only as directed in the test procedure. Damaged labware and malfunctioning equipment should not be used.

If accidental skin contact with hazardous chemicals occurs, flush the contacted area with water for 15 minutes. Call a physician if necessary. A MSDS (Material Safety Data Sheet) accompanies the first shipment of all products. Please retain the MSDS and refer to it for comprehensive safety data essential for day-to-day operations and safety training.

Sample Cell Matching

Although sample cells furnished with the DR/2000 are essentially distortion-free optically, nicks and scratches acquired during handling can cause a mismatch between two sample cells and introduce error in the test results. This type of error can be minimized by optically matching the sample cells as follows:

1. Turn the instrument ON, select the constant-on mode and wait approximately five minutes for the warm-up period.

2. Enter the stored program for absorbance.

Press: 0 READ/ENTER

The display will show :Abs

older supplies are used first. If in doubt about the reagent shelf life, run a standard to check reagent effectiveness.

Reagent Blank

The term "reagent blank" refers to that portion of the test result contributed solely by the reagent and not the sample. This portion of the result represents a positive error. In several of the tests, the reagent blank is of such magnitude that compensation must be made each time the test is performed. This is done by zeroing the instrument on deionized water and reagents.

In most cases, the reagent blank is so small the instrument is zeroed on either an untreated portion of the original water sample or deionized water. This is done routinely without any significant loss of accuracy except where extremely small amounts of a constituent are sought. In such a case, it is best to determine the reagent blank by performing the test on a sample of high-quality, turbidity-free deionized water. The results is expressed in the concentration units of the test and is subtracted from the test results of subsequent samples using that particular lot of reagent. It is necessary to determine the reagent blank only at first use and at intervals of several months unless subsequent contamination is suspected.

Every effort is made to produce reagents with the lowest possible blank. In most cases, it is less than 0.009 absorbance units. In some instances, it is either impossible or not practical to produce reagents with such a low blank. In these cases, it is best to determine the reagent blank as explained above and subtract it from each determination. A note is included in the appropriate procedures describing when this is necessary.

Safety

Safety is the responsibility of each individual when performing analysis procedures, and the analyst must develop and maintain good safety habits, because many of the procedures in this methods manual require the use of potentially hazardous chemicals and apparatus. It is important for the individual conducting them to minimize chances for accidents by practicing good laboratory techniques. Several rules applying to water analysis in the laboratory and in the field follow. They are not all inclusive, of course, General in nature, they emphasize that often are key factors in personal injury incidents.

Read Lables Carefully: Each reagent lable should be read carefully with particular attention paid to the precautionary information. Never remove the label from a reagent container while it contains reagent. Do not put a different reagent into a labeled container without changing the label. When preparing a reagent or standard solution, be sure to label the container clearly.

3. Rotate the wavelength dial until the small display shows: 510 nm OR another wavelength of interest.
4. Pour at least 25 mL of deionized water into each of two sample cells.
5. Place one sample cell into the cell holder. Face the 25-mL mark to your right. Close the light shield.
6. Press: Zero
The display should show: WAIT then: 0.000 Abs
7. Place the other sample cell into the cell holder. Close the light shield.
8. Wait approximately three seconds for the value to stabilize and read the absorbance. Record the resulting absorbance.
9. Try to achieve a match of ± 0.002 Abs. Rotate the cells 180 degrees (25-mL mark facing to your left) and recheck the match.

If the sample cells cannot be matched within 0.002 absorbance units, they still can be used by compensating for the difference. For instance, if the second sample cell reads 0.0002 absorbance units higher than the reference cell, future readings using these same cells at this wavelength can be corrected by subtracting 0.002 absorbance units from the reading. Likewise, if the sample cell had a negative absorbance value relative to the reference cell, that value should be added to the reading.

Or, match the cells using the stored program of interest and its wavelength. Use the most transparent sample cell as the blank. The concentration of the other sample cell should be subtracted from future readings. Instructions for matching cells on other instruments can be found in the instrument manual.

Sample Dilution Techniques

Twenty-five milliliters (mL) is the specified volume for most calorimetric tests in Section II. However, in some tests, the color developed in the sample may be too intense to be measured. Unexpected colors may develop in other tests. In both cases, it is necessary to dilute the sample or determine if interfering substances are present.

For example, when performing the chromium tests, the spectrophotometer may read above 0.6 mg/L. This results in the display showing OVER-RANGE. A sample dilution is necessary. The test must be repeated, but with a 25-mL graduated cylinder filled to the sample and then to the 25-mL mark with the sample and then to the 25 mL mark with deionized water. Because the sample was diluted to twice its original volume (12.5 to 25 mL), the test result should be multiplied by 2 to give the correct concentration of chromium.

To accomplish the sample dilution conveniently, pipet the chosen sample portion into a clean graduated cylinder (or clean volumetric flask for more accurate work) and fill the cylinder (or flask) to the desired volume with deionized water. Mix well. Use the diluted sample when running the test.

As an aid, the following sample dilution table shows the amount of sample taken, the amount of deionized water used to bring the volume up to 25 mL and the multiplication factor.

Sample Dilutions

Sample Volume (mL)	Deionized Water Used to Bring the Volume to 25 mL (mL)	Multiplication Factor
25.0	0.0	1
12.5	12.5	2
10.0*	15.0	2.5
5.0*	20.0	5
2.5*	22.5	10
1.0*	24.0	25
0.250*	24.75	100

* For sample size of 10 mL or less, a pipet should be used to measure the sample into the graduated cylinder or volumetric flask.

The concentration of the sample is equal to the diluted sample reading times the multiplication factor.

An example: A 2.5 mL sample was diluted with 22 mL of deionized water. The results was 0.35 mg/L. What is the concentration of the sample?

$$0.35 \times 10 = 3.5 \text{ mg/L}$$

More accurate dilutions can be done with a pipet and a 100-mL volumetric flask. Pipet the sample and dilute to volume with deionized water. Invert several times to mix.

**Multiplication Factors to be Used
When Sample is Diluted to 100 mL**

Sample Volume (mL)	Multiplication Factor
1	100
2	50
5	20
10	10
25	4
50	2

Sample dilution also influences the level at which a substance may interfere. The effect of the interference decreases as the sample size decrease. Therefore, the effect of the interference described in the procedure notes will decrease as the sample size decrease. In other words, higher levels of an interfering substance can be present if the sample is diluted.

An example: Copper does not interfere at or below 100 mg/l for a 25.00 mL sample in a procedure. If the sample volume is diluted with an equal volume of water, what is the level at which copper will not interfere?

$$\frac{\text{Total volume}}{\text{Sample volume}} = \text{Dilution factor}$$

$$\frac{25}{12.5} = 2$$

12.5

$$\text{Interference level} \times \text{Dilution factor} = \text{Interference level in sample}$$

$$100 \times 2 = 200 \text{ mg/L}$$

The level at which copper will not interfere in the sample is at or below 200 mg/L.

Temperature Considerations

For best results, most tests described in this manual should be performed with sample temperatures between 20 degrees C (68 degrees F) and 25 degrees C (77 degrees F). If certain tests require closer temperature control, that requirement will be indicated in notes following procedures.

Use of Pipets and Graduated Cylinders.

When small sample quantities are used, the accuracy of measurements is important. Rinse the pipet or cylinder two or three times with the sample to be tested before filling. Use a pipet filler bulb to draw the sample into the pipet. Never pipet chemical reagent solutions or samples by mouth. When filling a pipet, keep the tip of the pipet below the surface of the sample as the sample is drawn into the pipet.

Serological pipets are long tubes with a series of calibrated marks to indicate the volume of liquid delivered by the pipet. The calibrated marks may extend to tip of the pipet or may be only on the straight portion of the tube. Fill serological pipets to the zero mark and discharge the sample by allowing the sample to drain until the meniscus is level with the desired mark. If the serological pipet has calibrated marks extended to the tip of the pipet, the sample must be blown out of the tip for accurate sample measurements.

Volumetric (transfer) pipets are long tubes with a bulb in the middle and a single ring above the bulb to indicate the volume of liquid to be delivered when it is filled to the mark. To discharge the sample from a volumetric pipet, hold the tip of the pipet at a slight angle against the container wall and drain. Do not attempt to discharge sample or reagent remaining in the tip of the pipet after draining. Volumetric pipets are designed to always retain a small reproducible amount of sample in the tip of the pipet.

Use of Reagent Powder Pillows.

Dry powdered reagents are used when possible to minimize problems of leakage and deterioration. Powders are packaged in individual, pre-measured, polyethylene "powder pillows". Each pillow contains enough reagent for one test and is opened easily with nail clippers or scissors.

Using PermaChem Pillows.

For the best results, lightly tap the pillow on a hard surface to collect powdered reagent in the bottom. Then:

1. Tear across, from side to side, holding the pillow away from your face.
2. Using two hands, push both sides toward each other to form a spout.
3. Pour the pillow contents into the sample cell and continue your procedure according to the instructions.

Mixing Water Samples.

The following two methods may be helpful in tests that require mixing sample with chemicals (usually indicated by "swirl to mix" instructions).

1. When mixing sample in a squire sample cell, the swirling motion is attained by a simple twisting motion. Grasp the neck of the cell with thumb and index finger of one hand while resting the concave bottom of the cell on the tip of the index finger of other hand. Rotate the cell quickly, first one way and then the other, to mix the sample.

2. A swirling motion is recommended when mixing sample in a graduated cylinder or a titration flask. In this case, grip the cylinder (or flask) firmly with the tips of three fingers. Hold the cylinder at a 45-degree angle and twist the wrist. This motion will move the cylinder in an approximately 12-inch circle, giving the liquids an intense rotation to accomplish complete mixing in a few turns. This swirling procedure is the most gentle and offers the least interference from the atmosphere when testing for carbon dioxide and other gases. Both methods are simple but take a bit of practice in order to obtain the best results.

Comparability Testing

For application of a given test to your sample type for the purpose of reporting USEPA-required data, it may be necessary to compare one chemical method to another. The following outline is acceptable to the U.S. Environmental Protection Agency (EPA) for reporting such data. This procedure is designed to provide data on the comparability (equivalency) of two dissimilar analytical methods for measurement of the same property or constituent. In making the comparison, the analyst assumes one method is satisfactory (standard) and the second or alternate method is compared for equivalency. To provide sufficient data to apply statistical measurements of significance, the following determinations are required:

1. Use a sample representative of normal operating processes, well-mixed between aliquot withdrawals, to run four replicate determinations by each method. Record all values as shown in Table below.

Effluent Sample Representative of Normal Operating Conditions.

Aliquot	Standard Methods*	Alternate methods
1		
2		
3		
4		

*Cite Method References

2. If variations occur in the concentration of the measured constituent in the plant effluent, repeat the above testing on two more samples, one collected at the highest level of constituent normally encountered in the samples examined by the laboratory and one having a concentration at or near the lowest level usually examined. If these are not readily available, it is permissible to dilute the average sample to obtain a low value sample and to add a "spike" of a standard solution to obtain a high value sample. If the average sample usually has a negligible amount of the measured constituent, the

test should be run on a sample to which three different levels of standard solution have been added. One should be in the low end of the test range, one in the middle and one at the high end. Record all values as shown in Table below.

**Effluent Samples
of Varying Concentrations**

Low Level		High Level		
Aliquot	Std.	Alt.	Std.	Alt.
1				
2				
3				
4				

3. The above data can be used to gain permission on use an alternate test methods for reporting purposes as required by discharge permits or drinking water regulations. Data, with information identifying the kind of testing the variance is for, permit numbers (if any) and a description of the test should be mailed to the appropriate state or regional EPA office. EPA personnel will evaluate the data and generally must reply within 90 days.

WATER SAMPLING, STORAGE AND PRETREATMENT

2. WATER SAMPLING STORAGE AND PRETREATMENT

Sampling and Storage

Correct sampling and storage are critical to the accuracy of each test. For greatest accuracy minimize contamination from the sampling device, remove residues of previous samples from sample container and preserve the sample properly, if necessary.

Taking Water Samples

Samples for analysis should be collected carefully to make sure the most representative sample possible is obtained, they should be taken near the center of the vessel or duct and below the surface. Use only clean containers (bottles, beakers) for collecting samples. Rinse the container several times first with the water to be sampled.

Samples should be taken as closely as possible to the source of the supply to minimize the effects of a distribution system. The water should be allowed to run for sufficient time to flush the system, and the sample container should be filled slowly with a gentle stream to avoid turbulence and air bubbles. Water samples from wells should be collected after the pump has run long enough to deliver water representative of the ground water feeding the well.

It is difficult to obtain a truly representative sample when collecting surface water samples. Best results can be obtained by running a series of tests with samples taken from several locations and depth at different times. Results that can be used to establish patterns applicable to that particular body of water.

Generally, analyze samples as soon as possible after collection.

Depending on the nature of the test, special precautions in handling the sample also may be necessary to prevent natural interferences such as organic growth or loss or gain of dissolved gases. Sample preservatives and storage techniques are described in each procedure for sample held for later testing.

Acid Washing Bottles

A procedure may suggest the sample bottles be acid-washed to minimize the effect of interferences. This is accomplished by using a detergent to clean the glassware or plasticware, rinsing with tap water, rinsing with a 1:1 Hydrochloric Acid Solution or 1:1 Nitric Acid Solution, rinsing with deionized water. This may require successive rinses, up to 12-15 may be necessary if chromium is being determined. Air dry. The nitric acid rinse also is important if lead is being determined.

Chromic acid or chromium-free substitutes may be used to remove organic deposits from glass containers, but rinse containers thoroughly with water to remove traces of chromium.

Glassware for phosphate determinations should be washed with phosphate-free detergents and acid-washed with 1:1 HCL. This glassware must be rinsed thoroughly with distilled water. For ammonia and Kjeldahi nitrogen, the glassware must be rinsed with ammonia-free water.

Storage and Preservation

The most cost-effective sample containers are made of polypropylene or polyethylene. The best and most expensive containers are made of quartz or TFE (tetrafluoroethylene, Teflon). Avoid soft glass containers for metals in the microgram-per-liter range. Store samples for silver determination in light-absorbing containers.

Avoid introducing contaminating metals from containers, distilled water or membrane filters. Thoroughly clean sample containers as described under Acid Washing Bottles.

Preservation techniques can retard the chemical and biological changes continuing after sample is taken. These changes may change the amount of a chemical species available for analysis. As a general rule, it is best to analyze the samples as soon as possible after collection. This is especially true when the concentration is expected to be low. Analyzing immediately reduces the potential for error and minimizes labor.

Preservation methods are limited generally to pH control, chemical addition, refrigeration and freezing. The recommended preservation for various constituents is given in Table 15. Other information provided in the table is the suggested type of container and the maximum recommended holding times for properly preserved samples.

Aluminum, cadmium, chromium, cobalt, copper, iron, lead, nickel, phosphorus, potassium, silver and zinc samples can be preserved for at least 24 hours by the addition of one Nitric Acid, 1:1 Solution Pillow (Cat. No. 2540-98) per liter of sample. Check the pH with pH indicator paper or a pH meter to assure the pH is 2 or less. Add additional pillows if necessary. Alternative, adjust the pH to 2 or less with Nitric Acid, ACS, or 1:1 Nitric Acid. Adjust the sample pH prior to analysis by adding an equal number of Sodium Carbonate Anhydrous Powder Pillows (Cat. No. 179-98). Or, raise the pH to 4-5 with Sodium Hydroxide Standard Solution, 1 N or 5 N.

Table 1. Required Containers, Preservation Techniques and Holding Times¹

Parameter No./Name	Container ²	Preservation ^{3,4}	Maximum Holding Time ⁵
Table 1A—Bacterial Tests:			
1-4. Coliform, fecal and total	P, G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁶	8 hours.
5. Fecal streptococci	P, G	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁶	8 hours.
Table 1B—Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	Cool, 4°C	14 days.
4. Ammonia	P, G	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days.
16. Chloride	P, G	None required	28 days.
17. Chlorine, total residual	P, G	None required	Analyze immediately.
21. Color	P, G	Cool, 4°C	48 hours.
24-24. Cyanide, total and amenable to chlorination	P, G	Cool, 4°C, NaOH to pH >12, 0.6g ascorbic acid ⁶	14 days ⁷ .
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO ₃ to pH <2, H ₂ SO ₄ to pH <2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days.
Metals:⁸			
18. Chromium VI	P, G	Cool, 4°C	24 hours.
35. Mercury	P, G	HNO ₃ to pH <2	28 days.
3, 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except chromium VI and mercury.	P, G	HNO ₃ to pH <2	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite ⁹	P, G	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days.
42. Organic carbon	P, G	Cool, 4°C, HCl or H ₂ SO ₄ to pH <2	28 days.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top	None required	Analyze immediately.
47. Winkler	G Bottle and top	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days.
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H ₂ SO ₄ to pH <2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	Cool, 4°C	48 hours.
55. Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days.
56. Residue, Settleable	P, G	Cool, 4°C	48 hours.
57. Residue, volatile	P, G	Cool, 4°C	7 days.
61. Silica	P	Cool, 4°C	28 days.
64. Specific conductance	P, G	Cool, 4°C	28 days.
65. Sulfate	P, G	Cool, 4°C	28 days.
68. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH >9.	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P, G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze immediately.
73. Turbidity	P, G	Cool, 4°C	48 hours.

¹This table was taken from Table II published in the *Federal Register*, Friday, October 26, 1984, Vol. 49, No. 209, pages 28 to 29.

²Polyethylene (P) or Glass (G).

³Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

⁴When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (about pH 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

⁵Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details.

⁶Should only be used in the presence of residual chlorine.

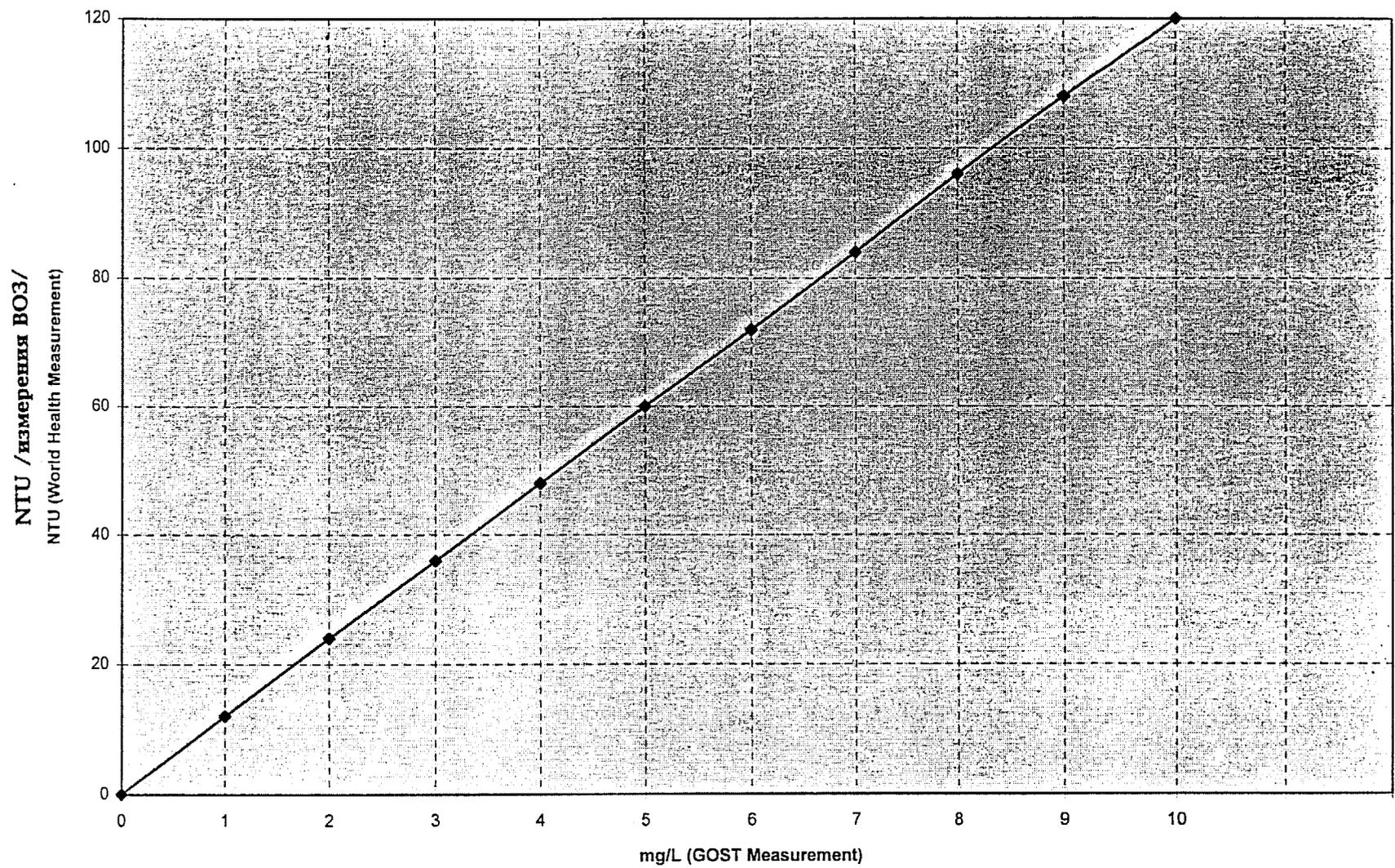
⁷Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

⁸Samples should be filtered immediately on-site before adding preservative for dissolved metals.

CHEMICAL AND ANALYTICAL PROCEDURES

СОПОСТАВЛЕНИЕ МУТНОСТИ

TURBIDITY COMPARISON



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мг/л /измерения в ГОСТ/

OPERATION

Operating Controls and Indicators

Figure 1 shows the Portable Incubator controls and indicators.

Key	Description
I O	Power switch to turn instrument on and off. The switch must be on before any systems are operational, including the control circuitry.
^	The up arrow key. Press and hold the button down until the display begins to blink, displaying the temperature set point. Press the key to decrease the incubation temperature set point.
v	The down arrow key. Press and hold the button down until the display begins to blink, displaying the temperature set point. Press the key to decrease the incubation temperature set point.

Collecting and Preserving Samples

Proper sampling technique ensures that results are representative of the sample source. Avoid sample contamination during collection.

Sample Size

Collect a sufficient volume of water for analysis, at least 100 mL of sample. World Health Organization guidelines suggest 200 mL per sample, while Standard methods for the Examination of Water and Wastewater guidelines suggest 100 mL per sample. Collect at least 100 mL of sample in presterilized plastic bags or bottles or in sterile glass or plastic sample bottles. Sample containers should not be filled completely. Maintain at least 2.5 cm (approximately 1") of air space to allow adequate space for mixing the sample prior to analysis.

Potable Water

Potable water should contain no coliforms per 100 mL, so testing should be done on undiluted samples. Use the Membrane Filtration test, 5-tube or 10-tube, most probable Numbers test, or Presence/Absence test for potable water. All these procedures except the 5-tube are USEPA-approved for reporting drinking water results.

Table 4

Suggested Sample Volumes for MF Fecal Coliform Test*

	VOLUME TO BE FILTERED (mL)						
	100	50	10	1	0.1	0.01	0.001
Water resource							
Lakes, reservoirs	X	X					
Wells, springs	X	X					
Water supply intake		X	X	X			
Natural bathing waters		X	X	X			
Sewage treatment plant secondary effluent		X	X	X			
Farm ponds, rivers				X	X	X	
Storm water run-off				X	X	X	
Raw municipal sewage					X	X	X
Feedlot run-off					X	X	X

*Standard Methods for the Examination of Water and Wastewater, 18th ed., page 9-56

Diluting Sampling

As indicated by Table 3 and 4, very small sample volumes may be required for testing water samples high in turbidity or coliform number. Because it is almost impossible to measure these small volumes accurately, a series of dilutions should be made. The following procedure describes one method of preparing a series of dilutions.

Dilution Technique

1. Wash hands.
2. Open a bottle of sterile Buffered Dilution Water.
3. Shake the sample collection container vigorously, approximately 25 times.
4. Use a sterile transfere pipet to pipit the required amount of sample into the sterile Buffered Dilution Water.
5. Recap the buffered dilution water bottle and shake vigorously 25 times.
6. If more dilutions are needed, repeat Steps 3-5 using clean, sterile pipets and additional bottles of sterile Buffered Dilution Water.

Dilution Series

A. If 10-mL sample is required:
Transfer 11 mL of sample into 99 mL of sterile buffered dilution water. Filter 100 mL of this dilution to obtain the 10 mL sample.

B. If 1-mL sample is required:

Transfer 11 mL of the 10 mL dilution from A into 99 mL of sterile dilution water. Filter 100 mL of this dilution to obtain the 1-mL sample.

C. If 0.1-mL sample is required:

Transfer 11 mL of the 1 mL dilution from B into 99 mL of sterile dilution water. Filter 100 mL of this dilution to obtain the 0.1-mL sample.

D. If 0.01-mL sample is required:

Transfer 11 mL of the 0.1 mL dilution from C into 99 mL of sterile dilution water. Filter 100 mL of this dilution to obtain the 0.01-mL sample.

E. If 0.001 mL sample is required:

Transfer 11 mL of the 0.01 mL dilution from D into 99 mL of sterile dilution water. Filter 100 mL of this dilution to obtain the 0.001-mL sample.

F. If 0.0001-mL sample is required:

Transfer 11 mL of the 0.001 mL dilution from E into 99 mL of sterile dilution water. Filter 100 mL of this dilution to obtain the 0.0001-mL sample.

Potable Water Procedures

Total Coliform procedure
Method 8074

* USEPA-approved for drinking water

* Presumptive total coliform test using m-Endo Broth.

* The procedure can also be used for nonpotable water, if the sample has been appropriately diluted.

1. Place a sterile absorbent pad in sterile petri dish (use sterilized forceps). Replace petri dish lid.
2. Open an ampule of m-Endo Broth. Pour the contents evenly over the absorbent pad. Replace petri dish lid.
3. Set up the Membrane Filter Apparatus. With sterile forceps, place a membrane filter, grid side up, into the assembly.
4. Shake the sample vigorously to mix. Pour 100 mL of Sample into the funnel. Apply vacuum and filter the sample. Rinse the funnel walls 3 times with 20 to 30 mL of sterile buffered dilution water.
5. Turn off the vacuum and lift off the funnel top. Using sterile forceps, transfer the filter to the previously prepared petri dish.

6. With a slight rolling motion, place the filter, grid side up, on the absorbent pad. Check for trapped air under the filter and make sure the filter touches the entire pad. Replace petri dish lid.

7. Invert the petri dish and incubate at 35 ± 0.5 degrees C for 24 hours.

8. After incubation count the greenish-gold metallic sheen colonies by using illuminated. magnifier or a 10-15X microscope.

Turbidity Measurement Procedure

1. Collect a representative sample in a clean container. Fill a sample cell to the line (about 15 mL), taking care to handle the sample cell by the top. Cap the cell. (See Section on Measurement Techniques for more information about collecting a representative sample).
2. Wipe the cell with a soft, lint-free cloth to remove water spots and fingerprints.
3. Apply a thin film of silicone oil. Wipe with a soft cloth to obtain an even film over the entire surface.
4. Press I/O. The instrument will turn on. Place the instrument on a flat, sturdy surface. Do not hold the instrument while making measurements.
5. Put the sample cell in the instrument cell compartment so the diamond or orientation mark aligns with the raised orientation mark in front of the cell compartment. **Close the cover.**
6. Select manual or automatic range selection by pressing the RANGE key. The display will show "AUTO RNG" when the instrument is in automatic range selection.
7. Select signal averaging mode by pressing the SIGNAL AVERAGE key. The display will show "SIG AVG" when the instrument is using signal averaging. Use signal average mode if the sample causes a noisy signal (display changes constantly).
8. Press READ. The display will show "-----NTU" then the turbidity in NTU. Record the turbidity after the lamp symbol turns off.

Measurement Notes

- Always cap the sample cell to prevent spillage of sample into the instrument.
- When taking a reading, place the instrument on a level, stationary surface. It should not be held in the hand during measurement.
- Always close the sample compartment lid during measurement and storage.
- Do not leave a sample cell in the cell compartment for extended periods of time. This may compress the spring in the cell holder.
- Remove sample cell and batteries from instrument if the instrument is stored for extended time periods (more than 2 months).

Nonpotable Water

Nonpotable water testing generally requires dilution of the original sample, based on probable coliform concentration. For MPN testing, three different dilutions should be tested, using 5 tubes for each dilution, for a total of 15 tubes.

Membrane Filtration (MF) Procedures

The MF methods is a fast, simple way of estimating bacterial population in water. In the initial step, an appropriate sample volume is passed through a membrane filter with a pore size small enough (0.45 microns) to retain the bacteria present. The filter is placed on an absorbent pad (in a petri dish) saturated with a culture medium that is selective for coliform. The petri dish containing the filter and pad is incubated, upside down, for 24 hours at the appropriate temperature. After incubation, the colonies that have grown are identified and counted by using illuminated magnifier or 10-15X microscope.

The MF Methods is especially useful for drinking water because large numbers of samples can be analyzed in a short time.

Preparing materials.

Start the incubator while preparing other materials. Adjust the incubator temperature setting 35 degrees C for total coliforms or 44.5 degrees C for fecal coliforms.

Using Presterilized Equipment and Media.

You will need sterile materials, a disinfected work and proper handling techniques, or contamination may give false results. To simplify technique and minimize the possibility of contamination, use presterilized equipment and media. Hach offers presterilized and disposable membrane filters, pipets, petri dishes, absorbent pads, inoculating loops, filter pads, buffered dilution water in 99-mL bottles, sampling bags, and prepared growth media. MELs include presterilized consumable and field filtration assembly.

If you are using a conventional filter funnel assembly, it will require sterilization. Sanitize the funnel by immersing it in boiling water for 5 minutes prior to use. You will also need to sterilize the forceps included with an portable lab. Just dip the forceps in alcohol and flame.

Using Field Filtration Apparatus

1. Flame sterilize the top surface of the stainless steel Field Vacuum Support.
2. Attach the luer tip of syringe to the tubing attached to the vacuum support.

3. Using sterile forceps, place a membrane filter, grid side up, onto the center of the vacuum support.

Note: To sterilize forceps, dip forceps in alcohol and flame in an alcohol or Bunsen burner. Let forceps cool before use.

4. Open a package of funnels (start at the bottom of the package). remove a funnel (base first) from the package.

5. Place the funnel onto the vacuum support. Do not touch the inside of the funnel. Push evenly on the upper rim of the funnel to snap it onto vacuum support.

6. Pour the sample into the funnel.

Note: See specific procedures for the sample volume required.

7. Pull on the syringe plunger to draw the sample through the filter apparatus.

8. Remove the funnel.

9. Press the lever on the vacuum support stem to lift the membrane filter from the surface of the vacuum support.

10. Use sterile forceps to remove the membrane filter.

11. Place the membrane filter into a prepared petri dish and incubate according to the appropriate procedure.

12. Disconnect the luer tip of the syringe from the tubing attached to the vacuum support. Dispose of the liquid in the syringe.

13. Follow steps 1-12 to filter remaining samples.

Using Autoclavable Equipment.

When numerous samples must be run on a routine basis, you may prefer to use an autoclave for nondisposable materials.

1. Wash all sample bottles, pipets, petri dishes, filter holder with stopper and graduated cylinder (if needed) with hot water and detergent.

2. Rinse several times with tap water and then with demineralized water and dry thoroughly.

3. Prepare all equipment for autoclaving.

* Loosely thread caps on sample bottles and cover caps and bottles necks with metal foil or paper.

* Cover the opening of graduated cylinders with metal foil of paper.

* Insert the base of the filter funnel into an autoclavable rubber stopper that will fit the filter flask.

* Wrap the two parts of the filter funnel assembly separately in heavy wrapping paper and seal with masking tape.

* Wrap petri dishes (borosilicate_ in paper or place in aluminum or stainless cans.

4. Sterilize equipment in an autoclave at 121 degrees C for 15 minutes. Borosilicate glass items may be sterilized with dry heat at 170 degrees C for a minimum of 1 hour.

Preparing Autoclavable Filter Assembly.

Disinfect the work bench or work area with a germicidal cloth, dilute bleach solution or dilute iodine solution. Wash hands thoroughly with soap and water.

1. After sterilization remove the filter funnel assembly from the wrapping paper.

2. Do not contaminate the funnel by touching the inner surfaces that will be exposed to the sample.

3. Insert the funnel with rubber stopper into the filtering flask or filter funnel manifold and connect to the water trap and aspirator with rubber tubing.

4. Using sterile forceps, place a sterile membrane filter on the filter base and attach the filter funnel top.

5. Filter a small quantity of sterile buffered dilution water through the funnel to assure a good seal on the filter and connections before running the sample through.

Sample Size.

Sample size is governed by bacterial density as well as turbidity.

* Ideal sample volume for total coliform testing yields approximately 20 to 80 coliform colonies and not more than 200 colonies for all types per filter.

* Ideal sample volume for fecal coliform testing yields approximately 20 to 60 coliform colonies and not more than 200 colonies for all types per filter.

To accomplish these ideal situations, three different volumes should be filtered for samples where the coliform number is uncertain. Tables 3 and 4 list recommended volumes for various types of samples,

When the sample is less than 20 mL (diluted or undiluted), 10 mL of sterile dilution water should be added to the filter funnel before vacuum is applied. This aids in the uniform distribution of the bacteria over the entire membrane filter.

Table 3.

Suggested Sample Volumes for MF Total Coliform Test*

Water resource	VOLUME TO BE FILTERED (mL)							
	100	50	10	1	0.1	0.01	0.001	0.0001
Drinking Water	X							
Swimming pools	X							
Wells, Springs	X	X	X					
Lakes, reservoirs	X	X	X					
Water supply intake			X	X	X			
Bathing beaches			X	X	X			
River water				X	X	X	X	
Chlorinated sewage				X	X	X		
Raw sewage					X	X	X	X

* Standard Methods for the Examination of Water and Wastewater, 18th ed., page 9-56

-Always use clean sample cells in good condition. Dirty, scratched, or damaged cells can cause inaccurate readings.

-Avoid operating in direct sunlight.

-Make certain cold samples do not "fog" the sample cell.

-Avoid settling of sample prior to measurement.

-Keep sample compartment lid closed to prevent dust and dirt entering.

Measurement Techniques

Proper measurement techniques are important in minimizing the effects of instrument variation, stray light and air bubbles. Regardless of the instrument used, measurements are more accurate, precise and repeatable if the analyst pays close attention to the proper measurement techniques.

Measure samples immediately to prevent temperature changes and settling. Avoid sample dilution when possible. Particles suspended in the original sample may dissolve or otherwise change characteristics when the sample temperature changes or when the sample is diluted, resulting in a non-representative sample measurement.

Cleaning Sample Cells

Cells must be extremely clean and free from significant scratches. The glass used to make cells is easily scratched- manufacturing cells free of minor scratches and other imperfections is difficult. However, minor imperfections are effectively masked by applying silicone oil as outlined in the section on Applying Silicone Oil.

Clean the inside and outside of the cells by washing with laboratory detergent. Follow with multiple rinses of distilled or demineralized water. Allow cells to air dry. Handle cells only by the top to minimize dirt, scratches and fingerprints in the light path.

Applying Silicone Oil

Applying a thin coat of silicone oil will mask minor imperfections and scratches which may contribute to turbidity or stray light. Use silicone oil equivalent to Hach Cat No. 1269-36. This silicone oil has the same refractive index as glass. When applied in a thin, uniform coat, the oil fills in and masks minor scratches and other imperfections in the glass. Apply the oil uniformly by wiping with a soft, lint-free cloth. Avoid application of excess oil. Applying excess oil may retain dirt and contaminate the instrument's cell compartment.

1. Thoroughly clean the sample cell.

2. Apply a small bead of silicone oil from the top to the bottom of the cell - just enough to coat the cell with a thin layer of oil.
3. Using a soft, lint-free cloth, spread the oil uniformly, then wipe off the excess so that only a thin coat of oil is left. The cell should appear nearly dry with little or no visible oil.

Orienting Sample Cells

Precise measurements for very low turbidity samples require using a single cell for all measurements or optically matching the cells. Using one cell provides the best precision and repeatability. When one cell is used, an orientation mark (other than the factory-placed diamond) can be placed on the cell so it's inserted into the instrument with the same orientation each time.

Orienting a Single Cell

When using a single cell, make an index or orientation mark on the cell as follows:

1. Fill the clean sample cell to the line with high quality water (<0.5 NTU). Cap and wipe with lint-free cloth. Apply silicone oil. See section on Correcting for Turbidity of Dilution Water for more information about high quality water.
2. Press: I/O to turn the instrument on.
3. Insert the sample cell into the sample compartment. Close the cover.
4. Press READ. Record the cell's position in the cell compartment and the displayed reading.

Remove the cell, rotate it slightly and reinsert it into the cell compartment. Close the cover, then press READ. Record the cell's position and the displayed reading.

6. Repeat Step 5 until the lowest reading is displayed. Place an orientation mark on the cell's marking band near the top of the cell so the cell can be consistently inserted in the position that yields the lowest reading. When using the cell, always place it in the instrument so the orientation mark aligns with the raised mark on the instrument.

Matching Multiple Sample Cells

Precise measurements of very low turbidity samples require the cells be optically matched or a single cell be used for all measurements. If more than one cell is used, follow this procedure to match (index) the cells:

1. Clean and oil the sample cell as instructed in Cleaning Sample Cells and in Applying Silicone Oil.

2. Fill the clean sample cells to the line with the same sample.
3. Press: I/O to turn the instrument on.
4. Insert the first sample cell into the sample compartment and close the cover.
5. Press: READ. Record the cell's position in the cell compartment and the displayed reading. Place an orientation mark on the cell's marking band.
6. Insert the second sample cell into the cell compartment and close the cover.
7. Press: READ. Record the cell's position in the cell compartment and the displayed reading.
8. Remove the cell, rotate it slightly and reinsert into the cell compartment. Close the cover, then press READ again. Record the cell's position and the displayed reading.
9. Repeat Step 8 until the value displayed for the second cell is within 0.01 NTU (or 1%) of the value obtained for the first cell. Place an orientation mark on the second cell's marking band so it is consistently inserted in this position.
10. Repeat Steps 6-9 if matching other sample cells.

Calibration

Calibration of the 2100P Turbidimeter is based on formazin, the primary standard for turbidity. The instrument's electronic and optical design provide long-term stability and minimize the need for frequent calibration. The two-detector ratioing system compensates for most fluctuations in lamp output. However, **a formazin recalibration should be done at least once every three months**, more often if experience indicates the need. When calibration is necessary, use formazin primary standards to calibrate.

Preparation of Formazin Stock Solution

Dilute formazin standard solutions from a 4000 NTU stock solution equivalent to Hach Cat. No. 2461-11. The prepared stock solution is stable for up to one year when properly prepared. An alternative to purchasing the 4000 NTU stock solution is preparing a stock solution as follows:

1. Dissolve 5.000 grams of reagent grade hydrazine sulfate ($N_2H_4 \cdot H_2SO_4$) in 400 mL of distilled water.
2. Dissolve 50.000 grams of pure hexamethylenetetramine in 400 mL of distilled water.

3. Pour the two solutions into a 1000-mL volumetric flask and dilute to the mark with distilled water.
4. Let the solution stand for 48 hours and 25 degrees C (77 degrees F). During this time a suspension will develop. This is the 4000 NTU stock solution. The standing temperature is critical for correct formation of formazin polymers.
5. Mix the 4000 NTU stock solution for at least ten minutes before use.

Correcting for Turbidity of Dilution Water

The 2100 Turbidimeter automatically compensates for turbidity contributed by dilution water when calculating the true value of the lowest formazin standard. High quality distilled or deionized water is usually sufficient if less than 0.5 NTU. If the turbidity is greater than 0.5 NTU, prepare the water as directed below. The 2100P will display E 1 after calibration if the dilution water turbidity is greater than 0.5 NTU.

The value of the dilution water can be arbitrarily forced to zero (see calibration procedure). This is not recommended for most applications and, if done, should be done only if the dilution water turbidity is less than 0.2 NTU.

Preparing the Dilution Water

Collect at least 1000 mL of high quality dilution water (e.g., distilled, demineralized or deionized water). Check the turbidity of the dilution water before use. When the 2100P is received, it may be used to check the dilution water turbidity because the instrument is precalibrated at the factory. If the turbidity is greater than 0.5 NTU, the water may be filtered using the Sample Filtration and Degassing Kit (Cat. No. 43975-10) or the equivalent. When measuring low range turbidity, all glassware should be cleaned with 1:1 hydrochloric acid and rinsed several times with dilution water. If the glassware is not used immediately, use stoppers to prevent small particles from contaminating it.

Dilution Water Preparation

1. Attach the syringe to the 3-way valve by gently twisting the square end into the syringe tip. Attach the connector, tubing and a 0.2 micron filter (clear part faces syringe) as shown. Be sure the connections are tight.
2. Fill a beaker or container with the water to be filtered. Insert the tubing into the container. Slowly draw the water into the syringe by pulling up on the syringe plunger.
3. Draw about 50 mL of sample into the syringe. Slowly push on the plunger to force the water through the filter and into a graduated cylinder or volumetric flask. Repeat Steps 2 and 3 until the desired amount of water is obtained.

Preparing Formazin Dilutions (Factory Recommended)

Hach Company recommends using 20, 100 and 800 NTU formazin standards for calibrating the 2100P Turbidimeter. Other dilutions can be prepared and used. If problems occur when using alternate solutions, use the dilutions specified in this section.

Prepare all formazin dilutions immediately before use and discard the solutions after calibration. The 4000 NTU solution is stable for up to a year, but dilutions deteriorate more rapidly. Prepare the 20, 100 and 800 NTU dilutions following the directions below. Use the same high quality water (turbidity <0.5 NTU) for the dilutions and the blank.

Preparing the 20, 100 and 800 Standards

Formazin Standard Preparation

20 NTU

1. Add 100 mL of dilution water to a clean 200-mL class A volumetric flask.
2. With a TenSette pipet add 1.0 mL of well-mixed 4000 NTU Formazin stock solution to the 200-mL flask.
3. Dilute to the mark with dilution water. Stopper and mix.

100 NTU

1. Add 100 mL of dilution water to a clean 200-mL class A volumetric flask.
2. With a TenSette pipet add 5.0 mL of well-mixed 4000 NTU Formazin stock solution to the 200-mL flask.
3. Dilute to the mark with dilution water. Stopper and mix.

800 NTU

1. Add 50 mL of dilution water to a clean 100-mL class A volumetric flask.
2. With a TenSette pipet add 20.0 mL of well-mixed 4000 NTU Formazin stock solution to the 100-mL flask.
3. Dilute to the mark with dilution water. Stopper and mix.

Calibrating the Turbidimeter

1. Rinse a clean sample cell with dilution water several times. Then fill the cell to the line (about 15 mL) with dilution water.

2. Insert the sample cell in the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid. Press I/O.
3. Press CAL. The "CAL" and "SO" icons will be displayed (the "O" will flash). The 4 digit display will show the value of the SO standard for the previous calibration. If the blank value was forced to 0.0, the display will be blank. Press the right arrow key to get a numerical display.
4. Press READ. The instrument will count from 60 to 0, (67 to 0 if signal average is on), read the blank and use it to calculate a correction factor for the 20 NTU standard measurement. If the dilution water is equal to or greater than 0.5 NTU, E 1 will appear when the calibration is calculated (See section on Preparing the Dilution Water for more dilution water information). The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.
5. The display will show the "S1" (with the 1 flashing) and "20 NTU" or the value of the S1 standard for the previous calibration. If the value is incorrect, edit the value by pressing the right arrow key until the number that needs editing flashes. Use the up arrow key to scroll to the correct number. After editing, fill a clean sample cell to the line with well mixed 20 NTU standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.
6. Press: READ. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. The display will automatically increment to the next standard. Remove the sample cell from the cell compartment.
7. The display will show the "S2" (with the 2 flashing) and "100 NTU" or the value of the S2 standard for the previous calibration. If the value is incorrect, edit the value by pressing the right arrow key until the number that needs editing flashes. Use the up arrow key to scroll to the correct number. After editing, fill a clean sample cell to the line with well mixed 100 NTU standard. Insert the sample cell into the cell compartment by aligning the orientation mark on the cell with the mark on the form of the cell compartment. Close the lid.
8. Press: READ. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Then, the display will automatically increment to the next standard. Remove the sample cell from the cell compartment.
9. The display will show the "S3" (with the 3 flashing) and "800 NTU" or the value of the S3 standard for the previous calibration. If the value is incorrect, edit the value by pressing the right arrow key until the number that needs editing flashes. Use the up arrow key to scroll to the correct number. After editing, fill a clean sample cell to the line with well mixed 800 NTU standard. Insert the sample

cell into the cell compartment by aligning the orientation mark on the cell with the mark on the front of the cell compartment. Close the lid.

10. Press: READ. The instrument will count from 60 to 0 (67 to 0 if signal average is on), measure the turbidity and store the value. Then the display will increment back to the SO display. Remove the sample cell from the cell compartment.

11. Press: CAL to accept the calibration. The instrument will return to measurement mode automatically.

INVENTORY OF EQUIPMENT AND MANUALS

**Inventory of Laboratory Equipment
(Kzyl-Orda, SES)**

44800-60	DR/200 Spectrophotometer
30331-00	Container HC-7
20950-00	Portable Turbidimeter
14034-46	NitrVer 5 Powder Pillows
14070-49	DPD Free Chlorine Powder Pillows
46647-00	Lamp Assembly

LIST OF MANUALS

Water Analysis Handbook
DR/200 Spectrophotometer
Manual

INTERNATIONAL GUIDELINES FOR WATER QUALITY

Drinking water standards. GOST 2674-82

Microbiological indicators

Indicator	Standard	Method of testing
Number of microorganism in 1mm ³ of water	100	GOST 18963-73
Number of bacteria of E.coli in 1 l of water (coli-index)	3	GOST 18963-73

Chemical indicators

Chemical component	Standard	Method of testing
Aluminum residual (Al)	0.5	GOST 18165-81
Beryllium (Be)	0.0002	GOST 18291-81
Molybdenum (Mo)	0.25	GOST 18308-72
Arsenic (As)	0.05	GOST 4152-81
Nitrate (NO ₃)	45.0	GOST 18826-73
Polyacrilamide, residual	2.0	GOST 19353-74
Lead (Pb)	0.03	GOST 19413-81
Selenium (Se)	0.001	GOST 19413-81
Strontium (Sr)	7.0	GOST 23950
Fluoride (F) not exceed for climatic regions		
I & II	1.5	
II	1.2	
III	0.7	

Esthetic indicators

Chemical component	Standard	Method of testing
pH	6.0-9.0	pH meter
Iron (Fe)	0.3	GOST 4011-72
Total hardness mg x eqv/l	7.0	GOST 4151-72
Manganese (Mn)	1.0	GOST 4388-72
Polyphosphate residual (PO ₄ - - -)	3.5	GOST 18309-72
Sulfate (SO ₄ - -)	500	GOST 4389-72
Dried precipitates	1000	GOST 18164-72
Chloride (Cl-)	350	GOST 4245-72
Cincum (Zn ²⁺)	5.0	GOST 18289-72
Odor 20oC rate	2	GOST 3351-74
Taste 20oC	2	GOST 3351-74
Color degree	200	GOST 3351-74
Turbidity mg/l	1.5	GOST 3351-74

Note: Standards are not to exceed values expressed in mg/l

World Health Organization Guidelines for Microbiological Quality

Organism	Unit	Guide-line value	Remarks
A. Piped Water Supplies			
<i>A.1 Treated water entering the distribution system</i>			
Fecal coliforms	number/100 mL	0	Turbidity < 1 NTU; for disinfection with chlorine, pH preferably < 8.0; free chlorine residual 0.2-0.5 mg/L following 30 min (minimum) contact
Coliform organisms	number/100 mL	0	
<i>A.2 Untreated water entering the distribution system</i>			
Fecal coliforms	number/100 mL	0	In 98% of samples examined throughout the year
Coliform organisms	number/100 mL	0	
Coliform organisms	number/100 mL	3	
<i>A.3 Water in the distribution system</i>			
Fecal coliforms	number/100 mL	0	In 95% of samples examined throughout the year
Coliform organisms	number/100 mL	0	
Coliform organisms	number/100 mL	3	
B. Unpipied Water Supplies			
Fecal coliforms	number/100 mL	0	Should not occur repeatedly
Coliform organisms	number/100 mL	10	
C. Bottled Drinking Water			
Fecal coliforms	number/100 mL	0	Source should be free from fecal contamination
Coliform organisms	number/100 mL	0	
D. Emergency Water Supplies			
Fecal coliforms	number/100 mL	0	Advise public to boil water in case of failure to meet guideline value
Coliform organisms	number/100 mL	0	

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World Health Organization Guidelines for Aesthetic Quality

Constituent	Guideline value*	Remarks
Aluminum	0.2	
Chloride	250	
Chlorobenzenes and chlorophenols	No guideline value set	These compounds may affect taste and odor
Color	15 TCU†	
Copper	1.0	
Detergents	No guideline value set	There should not be any foaming, taste, or odor problem
Hardness	500 (as CaCO ₃)	
Hydrogen sulfide	Not detectable by consumers	
Iron	0.3	
Manganese	0.1	
Oxygen, dissolved	No guideline value set	
pH	6.5-8.5	
Sodium	200	
Solids, total dissolved	1000	
Sulfate	400	
Taste and odor	Inoffensive to most consumers	
Temperature	No guideline value set	
Turbidity	5 NTU‡	Preferably < 1 for disinfection efficiency
Zinc	5.0	

*Unless otherwise specified, all units are mg/L.

†TCU—true color unit.

‡NTU—nephelometric turbidity unit.

Comparison of International Drinking Water Guidelines

Parameter	USEPA	EEC		WHO	Canada
	Max. Contamination Level	Guide Line	Max. Admissible Conc.	Guide Line	Max. Admissible Conc.
Aluminum		0.05 mg/L	0.2 mg/L	0.2 mg/L	
Ammonium		0.05mg/L	0.5 mg/L		
Antimony			10 µg/L		
Arsenic	0.05 mg/L		0.05 mg/L	0.05 mg/L	0.05 mg/L
Barium	1.0 mg/L	0.1 mg/L	0.1 mg/L	NS*	1.0 mg/L
Boron		1000 µg/L	NS*		5.0 mg/L
Cadmium	0.01 mg/L		0.005 mg/L	0.005 mg/L	0.005 mg/L
Calcium		100 mg/L			
Chloride	250 mg/L	25 mg/L	NS*	250 mg/L	250 mg/L
Chromium	0.05 mg/L		0.05 mg/L	0.05 mg/L	0.05 mg/L
Chloride	250 mg/L	25 mg/L	NS*	250 mg/L	250 mg/L
Coliforms (organisms/100 mL)	1		10	0	0
Color	15 cu	1 mg Pt-Co/L	20 mg Pt-Co/L	15 cu	15 cu
Copper	1 mg/L**	100 µg at treatment plant	NS*	1 mg/L	1.0 mg/L
Cyanides			50 µg/L		
Fluoride	4.0 mg/L	varies w/temperature In area	NS*	1.5 mg/L	1.5 mg/L
Iron	0.3 mg/L**	50 µg/L	300 µg/L	0.3 mg/L	0.3 mg/L
Lead	0.05 mg/L		0.05 mg/L	0.05 mg/L	0.05 mg/L
Magnesium		30 mg/L	50 mg/L		
Manganese	0.05 mg/L**	20 µg/L	50 µg/L	0.1 mg/L	0.05 mg/L
Mercury	0.002 mg/L		0.001 mg/L	0.001 mg/L	0.001 mg/L
Molybdenum					
Nickel			50 µg/L		
Nitrates	10.0 mg/L (as N)	25 mg/L (as NO ₃)	50 mg/L	10.0 mg/L (as N)	10.0 mg/L (as N)
Nitrites			0.1 mg/L		1.0 mg/L
Odor	3 TON	0 dilution number	2 Dilution number @ 12 °C; 3 dil. No. @ 25 °C		
pH	6.5-8.6**	6.5-8.5	NS*	6.5-8.5	6.5-8.5
Phosphorus		400 µg/L	5000 µg/L		
Phenols			0.5 µg/L C ₆ H ₅ OH		0.002 mg/L
Potassium		10 mg/L	12 mg/L		
Selenium	0.01 mg/L		0.01 mg/L	0.01 mg/L	0.01 mg/L
Silica dioxide		1 mg/L	10 mg/L		
Silver	0.05 mg/L		0.01 mg/L	NS*	0.05 mg/L
Solids, Total dissolved	500 mg/L**	NS*	NS*	1000 mg/L	500 mg/L
Sodium		30 mg/L			
Sulfate	250 mg/L**	25 mg/L	NS*	400 mg/L	500 mg/L
Turbidity (non microbial)		0-4 JTU	4 JTU	5 NTU	5 NTU
Zinc	5 mg/L**	100 µg at treatment plant	NS*	5.0 mg/L	5 mg/L

*NS = No standard
**U.S. Secondary MCL