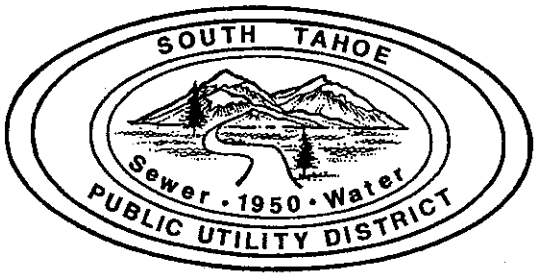

SOUTH TAHOE PUBLIC UTILITY DISTRICT
LABORATORY QUALITY ASSURANCE PROGRAM

by

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PREFACE

The Quality Assurance Program of the South Tahoe Public Utility District (STPUD) is a major part of the daily laboratory routine. Quality assurance is incorporated in sampling procedures, analysis preparation, analysis, maintenance of laboratory equipment and supplies, as well as the reporting of analytical results.

The STPUD Laboratory quality assurance program will never be completely written. Like the Winchester House, it will always have additions, new procedures for better assuring the quality of our data. The STPUD Laboratory is trying its best to circumvent Werner Heisenberg's Uncertainty Principle: that matter is not susceptible to objective measurement. We believe that by continually enhancing our QA program that we can reduce that uncertainty.

The following pages outline the quality assurance program currently practiced by the STPUD Laboratory.

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I. SAMPLE BOTTLE PREPARATION

Bottle preparation is to be done as follows:

Type	Size	Procedure
Bac-t	125 mL	Soap H_2O wash (as needed), hot H_2O rinse, deionize H_2O rinse, add 2 squirts of sodium thiosulfate. Autoclave at 121°C
0.45 µm Filtrates	100 glass	Hot H_2O rinse, HCl rinse, hot H_2O rinse, deionize H_2O rinse
A/E Glass Fiber Filtrate	250 mL plastic	Soap H_2O wash (as needed), hot H_2O rinse, HCl rinse, hot H_2O rinse, deionize H_2O rinse
Metals	500 mL 1,000 mL	Soap water wash, hot H_2O rinse, HNO_3 rinse, hot H_2O rinse, HCl rinse, hot H_2O rinse, deionize H_2O rinse
COD, NH_3, TKN, Total P, PO_4-P	500mL 1,000 mL	Soap H_2O wash (as needed), hot H_2O rinse, HCl rinse, hot H_2O rinse, deionize H_2O rinse
SS, Cl^-, pH, EC, Alk, BOD, NO_3	2,000 mL 1 gallon	Soap H_2O wash (as needed), hot H_2O rinse, deionize H_2O rinse
Oil & Grease	1,000 mL	Freon rinse, soap H_2O wash (as needed), hot H_2O rinse, deionize H_2O rinse.
Blue-Green Algae	250 mL	Soap H_2O wash (as needed), hot H_2O rinse, deionize H_2O rinse
Wastewater Plant Samples	1,000 mL 2,000 mL 2 gallon	Hot H_2O rinse, deionize H_2O rinse. Bottles should be washed with soapy H_2O when grease or algae deposits appear. After soap washing, hot H_2O rinse, HCl rinse, hot H_2O rinse and deionize H_2O rinse.
Organics	various	Supplied by commercial laboratory performing tests. Glassware to be prepared according to EPA specifications
VOC's	40 mL	Supplied by commercial laboratory performing tests. Glassware to be prepared according to EPA specifications. HCl to be pre-added.

Iron deposits can be removed by soaking with 1:1 HNO_3. Fill 1/6 to 1/4 of bottle with acid and dilute to neck (or above deposit) with tap H_2O. Allow to soak for an hour or more or until deposit is dissolved. Rinse with tap water, 1 N HCl, tap water, then deionized water to remove all traces of HNO_3.

Use brushes or nylon scouring pads as necessary to remove dirt or sludge deposits. Make certain threads on both cap and bottle are clean.

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II. SAMPLE CHAIN OF CUSTODY

1. All samples obtained by the South Tahoe Public Utility District (STPUD) for analysis are done so using procedures recommended by the EPA's Methods for Chemical Analysis of Water and Wastes, 1983 edition, Methods for the Determination of Inorganic Substances in Environmental Samples, 1993 edition, and Standard Methods for the Examination of Water and Wastewater, 18th edition.

2. **PLANT SAMPLES** Samples obtained at the STPUD wastewater treatment plant are obtained both automatically and manually.

   A. Automatic Composite Samplers Samples obtained by automatic composite samplers are treated as follows:

      1) Sample bottles are prepared by laboratory personnel. The bottles are rinsed with both tap and deionized water daily. The bottles are detergent washed and HCl rinsed weekly. Refer to Section I.

      2) Sample bottles are placed in the automatic sampler refrigerator by STPUD personnel. The starting date and time of the sample period is recorded on a composite sample log sheet located at the sampler.

      3) Filled sample bottles are picked up the following day by STPUD personnel. The date and time of the end of the sample period is recorded on the composite sample log sheet, as well as the time the sample was picked up. The sample refrigerator temperature, in degrees Celsius, is also recorded.

      4) The technician who picks up the sample records his initials on the composite sample log sheet. The technician who picks up the filled sample bottle installs a cleaned bottle for the next sample period. A copy of the composite sample log sheet is attached.

      5) The filled sample bottles are brought to the laboratory and analyses done on the same day.

      6) On delivery to Laboratory, all samples will be assigned a STPUD Lab ID# in the following format: XXXXYYZZ-AA.

         1. XXXX = the current year
         2. YY = the current month
         3. ZZ = the current day
         4. AA = the next available ID number

3. This Lab ID# is to be placed on the sample monitor sheet, any worksheets, and in the sample log book. A copy of the log book data sheet is attached.

A. Manual Composite Samples This is to be done only when flow-proportioned automatic sampler is not operating. Samples obtained manually are treated as follows.

1) Sample bottles are prepared by laboratory personnel. The bottles are rinsed with both tap and deionized water daily. The bottles are detergent washed and HCl rinsed weekly. Refer to Section I.

2) Samples are obtained in plastic bottles using a plastic dipper. The dipper is rinsed with sample, as is the sample bottle before obtaining sample.

3) 24-hour composite samples are obtained in a 250 mL plastic bottle. Sample are collected in 2-hour intervals. The sampler records his initials on the manual composite sample log sheet at each hour he obtains a sample. The individual composite samples are stored in a 4 °C refrigerator until completion of the sample period. This refrigerator is adjacent to the laboratory.

4) The flows are recorded for each sample hour on the manual composite sample log sheet. Calculations are made for the amount of sample to be used for each sample bottle proportionate to the flow at that hour. 2000 mL of composited sample is prepared. The technician who performs the compositing records his/her initials on the manual composite sample log sheet. A copy of this form is attached. Completed log sheets are filed and stored for a minimum of 5 years.

5) Analyses are performed on composited sample the same day. The sample is referred to on all worksheets by its name and ID#. An example is for the Final effluent:

Final 20070327-04a

B. Manual Grab Samples Samples collected manually and analyzed individually, Grab samples, are treated as follows.

1) Bottles are prepared according to recommended procedures found in the EPA's Methods for Chemical Analysis of Water and Wastes, 1983 edition, and Standard Methods for the Examination of Water and Wastewater, 18th edition. The preparation of the bottle is test dependent. Refer to Section I.

2) The sample is collected by STPUD Laboratory personnel and returned to the laboratory.

3) If the sample is to be analyzed for routine tests such as pH or Cl_2 the sample is referred to on the test data sheets by location, and sample ID#. An example is for the Secondary Effluent:

Sec 20070331-03

4) Bacterial samples are given a sample ID# and are identified on the Coliform worksheet by site, date and time collected, type (whether

routine, repeat or replacement), sample ID#, and sampler.

4. Off Plant Samples

A. Samples obtained off-plant can be any of the following:

1) Indian Creek and Harvey Place Reservoirs
2) Alpine Groundwaters
3) Alpine Surfacewaters
4) Alpine Soils
5) Alpine monitoring wells
6) Emergency Pond monitoring wells
7) Heavenly Valley Creek
8) Sewage spills and possibly contaminated surfacewaters
9) Water system
10) Underground storage tank remediation systems' effluents and soils
11) Suspected sewage and water line leaks

B. All non-plant samples are treated as described below:

1) Bottles are prepared according to recommended procedures found in the EPA's Methods for Chemical Analysis of Water and Wastes, 1983 edition, and Standard Methods for the Examination of Water and Wastewater, 18th edition. The preparation of the bottle is test dependent. Refer to Section I.

2) The sample is collected by STPUD Laboratory personnel. A field worksheet is filled out at the site of sample collection. Items that must be tested for immediately, such as pH, Cl_2, temperature, and DO, are done so at the time of collection. This data is recorded on the field worksheet.

3) The location, date and time of sample collection, depth (if applicable) and type of preservative are also recorded at sample site. The sampler's initials are also recorded as well as any comments or observations.

4) All samples and sub-samples are preserved on site, except from Alpine County groundwater (ranch) sites (to prevent accidents to homeowners and school children from presence of concentrated acids). If a sample will be analyzed for several tests whose preservatives are mutually exclusive the sample is subdivided. Each sub-sample is obtained in its own specially prepared bottle and preserved using the recommendations found in the Methods of Chemical Analysis of Water and Waste, 1983, pages xv-xx. All sub-samples share the same ID# except for the subscript which identifies the bottle-type and preservative used. Refer to Section III, **Table 3**. A copy of a typical field worksheet is attached.

5) Samples requiring filtration are filtered in the field. Use the portable suction pump and Nalgene filter apparat. Special labeled bottles are to be used for each filtrate type (Gelman A/E or Gelman 0.45 µm GN-6)

6) The sample is transported in a freezer chest and returned to the laboratory as soon as possible. The samples are entered into the Laboratory's sample log, given an ID#, and the sub-samples recorded.

7) The samples are then stored in a refrigerator kept at 4 EC, if applicable.

8) All non-plant samples are referred to by station #, or site description, and sample ID#. The subscript is also listed for each test performed. This is done by both STPUD laboratory personnel and outside commercial laboratories.

5. Commercial Laboratories Any samples that are to be sent to an outside laboratory for analysis must be given a STPUD sample identification number. The sample is labeled with the STPUD ID number. A letter describing the sample(s), preservative, if any, and test(s) requested is to accompany the sample. The form *Samples Sent To Commercial Laboratories* (found in Appendix B) can be used as an aid. The following information is to be entered in the *Sample Tracking Log*:

1) STPUD ID numbers
2) Sample type
3) Laboratory sample(s) sent to
4) Date sample(s) sent
5) Date sample results returned to STPUD

B. If the samples are to be used for court purposes, contractual agreements, etc. a **SAMPLE CHAIN OF CUSTODY RECORD** must accompany the sample(s).

6. Sample Storage

A. Water Samples

1) All samples except soils are stored in the laboratory's sample refrigerator.

2) Adhere to holding times specified in the most current Federal Register for Water and NPDES compliance. When storing samples enter information on *Repeat and Unfinished Analyses Sample Log* attached to sample refrigerator glass door. This information includes:

1. Site
2. ID#
3. Sample date
4. Analyses (check off required tests)

B. Soil Samples

1) Soils are air dried for 2-4 weeks. Prior to analysis they are ground to pass a 2 mm sieve (No 10) and homogenized.

7. Sample Disposal

A. Water samples

1) Most water samples are non-hazardous and are disposed down the sewer. These samples are usually obtained from streams, reservoirs, lakes, ranch soils, potable water wells, potable water distribution systems and storage tanks.

2) Sludge samples are disposed of through the sludge treatment system. Incineration is the ultimate fate.

3) Any hazardous samples will be discarded though a hazardous waste facility.

B. Soils

1) Soils are kept for one year after analysis in case repeat Analyses are required.

2) Tested soils are disposed by using them to fill the bore hole (of the same sample location) for new samples. This is usually done in the winter months, when the ground is frozen and it is difficult to obtain loose soil to fill the bore hole. The soil samples are taken from pasture lands and unfilled bore holes are a danger to cattle and horses.

C. Ash

1) Ash samples are returned to the Ash Bin or dropped into a Primary clarifier.

III. SAMPLING PROTOCOLS

1. Bacteriological Samples

A. Sample bottles are to be made of polypropylene. Use 125-250 mL bottles with threaded caps. Bottles should have 4 drops of 10% sodium thiosulfate added prior to autoclaving. All bottles should have sterile indicating tape on the caps. Bottles should be autoclaved for at least 30 minutes. The caps should be tightened as soon as bottles are cooled to room temperature. No bottle should be used which does not have black stripes appearing on the sterile indicating tape, nor which has caps loosely threaded.

1) Alternatively, commercially sterilized plastic bacteriological sample bottles can be used. These must:

1. Contain sodium thiosulfate for neutralizing chlorinated waters.
2. Have a secure seal indicating sterility.
3. Be constructed of clear plastic so it can be used as the culture flask for the Colilert method of coliform analysis.
4. Be clearly marked at the 100 mL volume.

B. At the sample site, turn water on and allow to flush, at a rate of 1 gpm, for at least three minutes. When sampling from dedicated sampling stations, allow to flush for at least 30 seconds before obtaining sample. Make certain sample faucet is not leaking nor dripping from threads or valve handle.

C. Obtain temperature and Free Cl_2 residual.

D. Obtain coliform sample. **DO NOT TOUCH INSIDE OF BACTERIAL SAMPLE BOTTLE NOR INSIDE OF CAP.** If sample bottle cap is loose, use another bottle. Make certain sterile bottle cap is facing with threads toward ground when un-capped. Do not pre-rinse bottle but fill to just-below neck as soon as possible (Fill Colilert bottles to the 100 mL line.).

E. Secure bottle cap and shake to mix sample with dechlorinating agent (sodium thiosulfate).

F. Record sample time, water temperature, and Cl_2 residual on sample monitor sheet. Include on the sample field sheet and in the sample logbook each sample's type: routine, repeat, replacement, process control, or customer complaint.

G. Place bacteriological sample bottle in ice chest. Transport samples to lab within 6 hours of collection.

2. Physical and Chemical Samples

A. When sampling wells make certain chlorine feed is turned off prior to collection of samples. The possibility of backflow of chlorine into sample taps located upstream of the chlorine feed is a distinct possibility.

B. Perform the following tests at the time of sample collection (if tests are required):

1) Temperature
2) Cl_2 residual, Total
3) EC (can also be done in the Lab)
4) Dissolved Oxygen
5) pH

C. All sample bottles, except for Oil & Grease, should be pre-rinsed with sample before taking aliquot. Bottles should be obtained from the Laboratory where they are properly prepared (see page 3).

D. All samples should be preserved on site according to the tests to be performed. A Lab ID# subscript should be assigned as described in Table 3.

1. **TABLE 3**

Sample Type	Preservative	Laboratory ID# Subscript
Physical	4 °C	a
Mineral	4 °C	a
Nutrients, next day analysis	4 °C	a
Nutrients, 28 day holding time	H_2SO_4, 4 °C	c
Metals	HNO_3	e
Radionucliides	HNO_3, 5 mL/L	e
Organics, TPH_D, Pesticides, Herbicides,	4 °C	d
Volatile organics, BTEX, TPH_G	4 °C, VOA vials	g
THM	4 °C, VOA vials	g
Radon	4 °C, VOA vials	g
TOC	H_2SO_4, 4 °C	c
CN	pH ≥12 , NaOH	h
\		

3. Radionuclides

A. Uranium and Gross Alpha

1) Obtain sample in a plastic I liter plastic bottle.

B. Radon

1) Use 2 VOA vials for Radon. This sample needs to be sent to the analytical laboratory within 24 hours.

2) Fill bucket with sample using a hose. The discharging end of the hose should be placed in the bottom of the bucket. When filled, remove hose, tap bucket to remove air bubbles and allow sediment to settle for 2-3 minutes.

3) Place radon vials in top third of sample and fill. Cap vials while they are immersed in the sample. Check for air bubbles and repeat filling if necessary.

4) Record exact time for filling the vials.

4. VOC's and Trihalomethanes (THM)

A. To prepare for sampling obtain the following:
1) VOA vials with HCl and ascorbic acid provided by commercial lab performing Analyses
2) Ice chest with blue ice
3) Travel Blank, provided by commercial lab. If one is not available, prepare one by boiling deionized water for ten (10) minutes, cool and pour into a THM vial provided by commercial lab.
4) Field Sheet and pen
5) HACH Chlorine test kit, for Free Cl_2
6) Thermometer
7) Beaker

B. THM sampling procedures:

1) If there is no Cl_2 residual, do not sample at this site. Choose another site in the area that does have a Cl_2 residual.

2) At the time of sampling, the following tests should be performed:

1. Temperature
2. Cl_2 residual, Free

3) Use VOA vials provided by laboratory performing the Analyses. These must be borosilicate glass vials of at least 40 mL capacity with screw caps (Pierce #13075 or equivalent). The screw caps shall have a Teflon-faced silicon septum (Pierce #12722 or equivalent). HCl, as a preservative must be added to the vial prior to sample collection. If Cl_2 is present, ascorbic acid for dechlorinating can be added to the vial after it is partially filled with sample.

4) Allow the sample tap to flow freely until the temperature is stable. Adjust the flow to about 500 mL/minute (½ quart/minute). Fill and seal

the VOA vials as follows:

5) Slowly fill each vial ¾ full with sample. If Cl_2 is present, ascorbic acid for dechlorinating can be added now.

6) Screw on vial cap and invert 3-4 times to mix sample with preservative (and dechlorinating agent if Cl_2 is present).

7) Remove cap and continue to fill to overflowing, allowing the sample within the vial to form an inverted meniscus.

8) Set the container on a level surface (or hold level) and carefully screw on cap, making certain the septum's Teflon side is facing the sample. Avoid entrapping air (filling an upside-down cap with sample before screwing on cap aids in keeping air bubbles out of the vial). Make certain cap is screwed on tight.

9) **NO BUBBLES SHOULD BE IN VIAL AFTER COLLECTING SAMPLE.**

    1. To ensure the sample has been properly sealed, invert and gently tap the lid on a solid surface. The absence of entrapped air bubbles indicates a properly obtained sample. If air bubbles are present, open the bottle, fill again to overflowing and reseal lid in the same manner as stated above.

10) Place sample in ice chest for transport to laboratory. The sample must remain tightly sealed and maintained at 4 °C until it is analyzed.

11) **EACH SAMPLE MUST BE TAKEN IN TRIPLICATE**. This is done to provide backup samples in case of breakage during shipment and for confirmation analyses if VOC's are detected.

12) Ship by next-day air. Care should be taken to not ship on Fridays, since the sample will not arrive till the following Monday. The sample may warm to above 5 EC if the blue-ice melts. If the sample needs a 24-hour turn-around time, make arrangements with the laboratory to receive the sample on Saturday.

C. Volatile Organic Chemicals (VOC's, TPH_{-Gas} and BTEX sampling procedures:

1) At the time of sampling, the following tests should be performed:

    1. Temperature
    2. Cl_2 residual, Free
    3. pH and EC if sampling for BTEX / TPH_{-Gas}

2) Use VOA vials provided by laboratory performing the Analyses. These must be borosilicate glass vials of at least 40 mL capacity with screw caps (Pierce #13075 or equivalent). The screw caps shall have a Teflon-faced silicon septum (Pierce #12722 or equivalent). HCl, as a preservative must be added to the vial prior to sample collection. If Cl_2 is present, ascorbic acid for dechlorinating can be added to the vial after it is partially filled with sample.

3) Allow the sample tap to flow freely until the temperature is stable. Adjust the flow to about 500 mL/minute (½ quart/minute). Fill and seal the VOA vials as follows:

4) Slowly fill each vial ¾ full with sample. If Cl_2 is present, ascorbic acid for dechlorinating can be added now.

5) Screw on vial cap and invert 3-4 times to mix sample with preservative (and dechlorinating agent if Cl_2 is present).

6) Remove cap and continue to fill to overflowing, allowing the sample within the vial to form an inverted meniscus.

7) Set the container on a level surface (or hold level) and carefully screw on cap, making certain the septum's Teflon side is facing the sample. Avoid entrapping air (filling an upside-down cap with sample aids in keeping air bubbles out of the vial). Make certain cap is screwed on tight.

8) **NO BUBBLES SHOULD BE IN VIAL AFTER COLLECTING SAMPLE.**.

    1. To ensure the sample has been properly sealed, invert and gently tap the lid on a solid surface. The absence of entrapped air bubbles indicates a properly obtained sample. If air bubbles are present, open the bottle, fill again to overflowing and reseal lid in the same manner as stated above.

9) Place sample in ice chest for transport to laboratory. The sample must remain tightly sealed and maintained at 4 °C until it is analyzed.

10) **EACH SAMPLE MUST BE TAKEN IN TRIPLICATE**. This is done to provide backup samples in case of breakage during shipment and for confirmation analyses if VOC's are detected.

11) Ship by next-day air. Care should be taken to not ship on Fridays, since the sample will not arrive till the following Monday. The sample may warm to above 5 EC if the blue-ice melts. If the sample needs a 24-hour turn-around time, make arrangements with the laboratory to receive the sample on Saturday.

5. Non-Purgeable Organic Chemicals (all organic chemicals that are not VOC's)

A. For Non-Purgeable Organic Chemicals (i.e., Base/Neutral and Acid Extractables, Organochlorine Pesticides and PCB's, etc.) use one (1) liter amber glass containers with caps threaded to screw onto the container. Caps shall be lined with Teflon. Foil may be substituted for Teflon if the water sample is not corrosive.

B. Allow the sample tap to flow freely until the temperature is stable. Adjust the flow to about 500 mL/minute and collect sample in a one (1) liter amber glass bottle (Section 5). Fill bottle so that head space is no greater than the threaded portion of the neck. Cap bottle with lined Teflon cap. Sample must be refrigerated at 4° C from the time of collection until analysis.

IV. REAGENTS

1. Grades

A. Reagents used in all Analyses shall be at least ***Analytical Reagent Grade***. The term ***Analytical Reagent Grade*** is synonymous with ***ACS Analytical Reagent Grade*** and ***Reagent Grade***. In all cases, the method's specified reagent grades will be used. In methods where no reagent grade is specified, ***Analytical Reagent Grade*** will be used.

B. Special grades of reagents are required for specific Analyses:

1) Metal Analyses
    1. ***Ultrex, Ultrex II***, or ***Trace Metal Grade***

2) Organic Analyses
    1. ***Ultra Resi-Analyzed Reagent, HPLC*** or ***Pesticide Grade***

C. Reagents will be purchased from reliable sources, ie: JT Baker, Fisher Scientific, Aldridch, GFS and Sigma.

2. Preparation

A. Prepared solutions will be logged into the Laboratory Log Book with the following information:

1) Date of preparation.
2) Initials of preparer.
3) Solution's name (in red).
4) Solutions code, if applicable.
5) Names of each reagent used in solutions's preparation.
6) Reagents' manufacturer, manufacturer's catalog number, and lot number.
7) Weight or volume of each reagent used in preparation.
8) Special instructions such as: *filtered through 0.45 µm filter*
9) Purpose of prepared solution.
10) Concentration of solution, if a standard (ie: 10 ppm COD)

B. Solutions will be prepared with STPUD deionized water. The resistance of the deionized water shall be at least 10 megohms.

C. All prepared reagents will be labeled with date of preparation and preparer's initials. Solutions will be checked regularly for indications of deterioration: discoloration, precipitates, bacterial and algal growths.

D. Primary standards will be obtained from a reliable source, dried, accurately prepared using Class A calibrated volumetric pipets, graduate cylinders and flasks. Prepared primary standards will be stored in containers that will not alter the reagent.

3. Standardization

A. All standardizations will be conducted using a minimum of three titrations. Prepared standards will be labeled with the date of preparation, preparer, and standard code. Percent recoveries of the standards will be checked regularly and the standard discarded when the recovery falls outside the established

control limits.

4. Storage

A. Use borosilicate glass bottles with ground glass stoppers for storage of most prepared reagents. Use plastic (polyethylene, polypropylene, etc.) containers for alkaline solutions such as sodium hydroxide (NaOH). Do not use plastic containers for organic reagents. Amber glass bottles should be used to store light sensitive reagents. Light sensitive reagents should be stored in a dark, cool place.

B. When not in use reagent bottles should be kept stoppered to prevent evaporation and resultant change in concentration.

C. Do not store incompatible chemicals together (ie: oxidizers and flammable liquids).

D. Purchase chemicals in safety containers (plastic-coated bottles) whenever possible.

V. WEIGHING

1. General Instructions:
   A. Weighing of samples and chemicals should be conducted in an area free of dust, thermal convection currents and drafts. Use the appropriate balance for the item to be weighed. Large masses requiring a sensitivity of ≥ 0.01 grams should be weighed on the top loading balance. Masses requiring a sensitivity ≥ 0.01 milligrams should be weighed on the analytical balance.

      1) Level the balance

      2) Make sure the balance is calibrated. Balances should be calibrated annually by a certified technician.
      3) Keep balance and area around balance clean and free from dust.

      4) Handle all masses with forceps, never with fingers. Place all masses as close as possible to the center of the pan

      5) Make certain the mass has cooled to room temperature. Place all hot masses in a desiccator to cool to room temperature prior to weighing. Check to make certain desiccator has fresh desiccant. Chemicals used for standards should be dried at 104 °C for at least 2 hours and cooled in a desiccator to room temperature before weighing.

      6) Press all display-function buttons smoothly with finger. This will prevent jarring of balance. **Do not use pointed objects to press display-function buttons**.

      7) Do not overload balance

      8) Never place moist objects or chemicals directly on the balance pans.

      9) Close balance doors (if part of balance) when weighing.

      10) Record mass on worksheet. Never try to remember mass for future recording.

      11) Make certain balance has stabilized before recording mass. Even though the electronic balances have a stabilization indicator, wait until reading is consistent for at least 10 seconds before recording mass.

   B. Errors in Weighing: Changes in moisture or CO_2 content can cause problems. Some materials take up H_2O or CO_2 from the air during weighing. Warm objects will cause convection currents than may cause the pan to be buoyed up, causing the mass to weigh less than the true mass. Static electricity may cause problems to the balance.

VI. QUALITY CONTROL SAMPLES

1. Source Routine quality control samples (QCS) are prepared by STPUD laboratory personnel, obtained from the EPA or purchased from laboratory supply vendors. QCS are used to determine the accuracy of laboratory methods of chemical, physical and biological Analyses and to ensure the instrument is operating properly.

   A. STPUD QCS samples are prepared as described in the section titled **REAGENTS**.

   B. Bacterial QCS samples are obtained commercially.

2. Quality Control for Routine and Non-routine Samples

   A. All routine and non-routine samples reported for NPDES and State Health requirements will follow QCS guidelines. Sample analysis will be repeated if QCS fall outside established limits. All samples whose tests results are to be used for planning purposes, contractual agreements, customer complaints, etc., will also have their Analyses repeated until the QCS fall within acceptable limits.

   B. Routine and non-routine samples whose analytical results are used in-house by STPUD personnel, only, are not required to follow QCS guidelines, though it should always be attempted. These types of samples include Primary, Secondary, Pond effluents, centrifuge feed, centrate, and cake sludges, aeration basin liquors, etc. Such samples are not required to have the Analyses repeated if the QCS fall outside acceptable limits. It is up to the analyst to determine if such data is acceptable to be reported to STPUD personnel. Such data should always be reported with the explanation that there may be error in the result.

3. Frequency of Analysis of QCS

   A. Every analysis will be run with some or all types of QCS. The following schedule will be followed for the analysis of QCS.

      1) Standards For each method, which specifies the preparation of standards, at least two standards shall be run with each batch of samples (BOD will be run with one standard). The standards used should cover both the low and high end of the sample concentration range. If any of the standards fall outside the established control limits the samples will be re-analyzed until the standards are within limits.

         1. For methods requiring a graph at the time of analysis, a curve with at least three (3) standards will be run for each batch of samples.

         2. When an automatic instrument is used for analysis, a calibration check standard will be run immediately after the calibration, and after every 10 samples for drinking water compliance samples and after every 20 samples for wastewater compliance samples and at the end of the analysis. The results of each sample set (every 10 or 20 samples) will be approved if both the beginning check standard and ending check standard for each sample set is no more than ±10 per cent of the true value of the check

standards.

2) External QCS standards Standards from a source not used to develop the calibration curve will be analyzed immediately after the calibration. Analysis will proceed only if the result of the External QCS is less than ±10 percent of the true value.

3) Duplicates At least one set of duplicates will be run with each batch of samples, or every 10 samples, for every analytical method. The range between duplicate analyses allows the analyst to determine the reproducibility of the test method.

    1. For spectrophotometric Analyses, the range between duplicate absorbencies will be used in place of the calculated result. In gravimetric Analyses (Suspended Solids and Total Dissolved Solids) the net weight difference, in milligrams, will be used. This will negate the effect of increased error due to sample dilution. If the range between duplicate samples falls above the upper control limit the samples will be re-analyzed until the control limits are met.

4) Performance Evaluation Samples (PE) PE samples, from either commercial suppliers or from U. S. EPA PE studies, will be run semi-annually for both water and wastewater methods. These will aid in determining whether internal laboratory error has developed.

5) Spikes Spiked samples (samples with a measured amount of standard added) will be run with each batch of samples, or every 10 samples. The recovery of the standard is determined after the amount of analyte contributed by the sample is subtracted from the final result. This enables the analyst to determine if the sample has constituents (matrix effects) that may interfere with the test method.

    1. The percent recovery of the added standard should fall within established limits. The samples should be repeated if the limits are exceeded. The formula for spike recovery is:

$$\%\text{Recovery} = 100\% \times \left[\frac{(\text{Spike } \mu\text{g/mL} \times \text{Total mL in Spike}) - (\text{Sample } \mu\text{g/mL} \times \text{Total mL Sample in Spike})}{(\text{Standard } \mu\text{g/mL} \times \text{Total mL Standard in Spike})} \right]$$

6) Method Blanks A method blank will be run with each batch of samples. For calorimetric methods the spectrophotometer will be zeroed against the method blank and all subsequent readings compared to the zeroed method blank. The absorbency of the method blank should be compared to a deionized water blank and recorded. Instrument malfunction, reagent deterioration and improper methodology can be detected by uncharacteristic method blank absorbencies. Blanks will be run with suspended solids and total dissolved solids tests. The results will be subtracted/added to the sample results. Filter blanks usually have a negative value due to filter loss.

    1. Reagent Blanks When an automatic instrument is used for

analysis, blanks will be run immediately after the calibration, and after every 10 samples for drinking water compliance samples and after every 20 samples for wastewater compliance samples and at the end of the analysis. The results of each sample set (every 10 or 20 samples) will be approved if both the beginning blank and ending blank have no detectable analyte. If Analyte is detected in the either of the blanks, the reporting limit will be adjusted to the amount detected in the highest blank.

7) Travel Blanks Travel blanks are sample bottles filled at the Laboratory with deionized water. The travel blanks' bottles and preservative will be the same as those of the field samples. Travel blanks are useful in determining if sampling methods, sample bottle preparation and sample preservation and storage are properly conducted.

1. A travel blank will be analyzed with each batch of field samples for every constituent the field samples are tested for.

4. Performance Evaluation Testing

A. The STPUD Laboratory participates in several performance evaluation testing programs:

1) EPA Wastewater Laboratory Performance Evaluation(Studies (DMRQA). (Annual)

2) EPA Water Supply Laboratory Performance Evaluation Studies. (Semi-Annual)

3) EPA Water Pollution Laboratory Performance Evaluation Studies. (Semi-Annual)

4) Analytical Products Group Performance Evaluation Tests (PET). (Semi-annual)

B. Samples are received twice a year from the Analytical Products Group, Inc. and run along with routine samples. In addition, the EPA annually sends the STPUD Laboratory performance evaluation samples for those parameters listed on the STPUD's NPDES permit. The EPA also sends two samples per year for both its PE studies: Water Pollution and Water Study. The results of semi-annual PET samples and the EPA samples are analyzed by STPUD Laboratory staff to determine the following:

1) Methods are being properly performed.
2) Reagent quality is satisfactory.
3) Laboratory equipment is operating properly.

1. If the results of STPUD Laboratory test method fall outside acceptable limits, the method is closely scrutinized to determine the source of error.

VII. STANDARD CURVE DEVELOPMENT

1. The following are the criteria that must be used when evaluating whether a standard curve is acceptable. These criteria do not mean the curve must be accepted. If standard *determined values* are unacceptable to the technician, the graph does not have to be accepted.

   A. The standards used must bracket the concentrations of the samples. No samples can be reported for compliance purposes that are outside the range of the standard curve. A sample can be diluted so that the concentration of the diluted sample is within the working range of the curve.

   B. Standards must be in the detection limit for reporting (DLR) to the maximum contaminant level (MCL) range for the samples.

   C. The standard curve must be constructed from a method blank and a minimum of three standards. It is good laboratory practice to use at least five (5) standards when using automated analytical equipment, or when performing Analyses whose standard curves are not linear over the entire range of concentrations.

   D. The standard curve must have correlation coefficient (r) ε 0.995. This is a measure of the linearity of the curve.

      1) Standards may be deleted from the curve to achieve an r ε 0.995. This is to be performed only as a last resort and only under the following conditions.

         1. The standard to be removed is outside the range of sample concentrations. The deleted standard may not be one that makes up either the upper or lower limit of the range of the sample's concentrations. For example, if the samples have analyte concentrations # 10 mg/L and the curve is composed of 6 standards (0, 0.5, 1.0, 5.0, 10, 20, 50), the 50 mg/L, and even the 20 mg/L standard may be deleted to achieve an r ε 0.995.

         2. A standard within the range of sample concentrations may be deleted only if it is a duplicate point. These are usually found in segmented curves where only two standards are used for a segment. Both points of a standard can not be deleted.

   E. An analysis must be stopped if any of the check standards exceed 10%. Those samples following the last acceptable check standard need to be re-analyzed with a new standard curve. Alternately, the run may killed and the check standard analyzed. If this check standard passes, the samples following the last acceptable check standard in the initial run may be analyzed with the original curve.

   F. If an analytical run is unattended, and on completion it was found that a group of samples was bracketed by two acceptable check standards but had one unacceptable check standard in the middle (ie. Chk Std Pass - samples - Chk Std fail - samples - Chk Std pass - samples...), those samples on both sides of the failed check standard must be re-analyzed.

   G. Spikes and control standards should be set at between 5 and 50 times the MDL or 1-10 times the ambient level, whichever is higher.

H. QC limits should be set at:

Analysis	Recovery of Known Additions %	Precision of Low-level Duplicates Δ %	Precision of High-level Duplicates Δ %
Nutrients	80-120	25	10
Anions	80-120	25	10
Metals	80-120	25	10
Other Inorganics	80-120	25	10

1) Additions calculated as % of the known addition recovered, duplicates calculated as the difference as a percentage of the mean $[100*(x_1-x_2)/0]$.

2) Low-level refers to concentrations less than 20 times the MDL. High-level refers to concentrations greater than 20 times the MDL.

VIII. CONTROL LIMITS

1. Quality Control (QC) limits are established for every tests for accuracy and precision. QC charts are developed for standard recovery, spike recovery (both accuracy) and range between duplicate samples (precision).

   A. Data is collect on each method for at least thirty (3), but usually sixty, Analyses. Separate charts are developed for spike recovery, standard recovery and precision.

   B. Accuracy Standard and spike recovery charts are developed by calculating the arithmetic mean and standard deviation of the test, after discarding outliers. Warning and Control limits are established at $\pm$ 2 and $\pm$ 3 standard deviations from the mean, respectively.

   C. Precision Precision charts are developed by calculating the arithmetic mean (R) of the range between duplicate Analyses and multiplying it by Shewhart's factors of D_3 and D_4 (2.51 and 3.27) to determine the warning and control limits, respectively.

      1) Warning Limit = $(D_3 \times R) = 2.51R$

      2) Control Limit = $(D_4 \times R) = 3.27R$

      3) To avoid problems of decreased precision due to sample dilutions, the ranges between duplicate absorbencies will be used for calorimetric Analyses and the range between duplicate net weights will be used for gravimetric Analyses. Titrimetric Analyses will use the range between duplicates' titrant volume.

2. Analyst Review

   A. The analyst will check the data for compliance with established control limits, that results were within the range of the standard curve, method blank quality, current trends and both state and federal requirements. Samples will be subjected to repeat analysis (with dilution if necessary) if any of the above items are not met.

   B. If a test is repeated because the initial result fell outside the current trend or exceeded the state or federal requirement, but was within control limits, both the initial and repeat test results will be averaged and reported.

   C. If the test was repeated because the QCS were outside the control limits, only repeat analytical results whose QCS were within control limits will be reported.

   D. Analysts will record results on summary forms. All worksheets will then be reviewed and corrected by lab personnel. All manual calculations will be repeated by the reviewer. The recorded results will be checked for proper accuracy and transcription mistakes. Once data has been reviewed, the worksheets will be signed and dated by the reviewer in red ink. Recorded results on the summary sheets will be marked with a red dot in the upper right-hand corner of the entry box. A red dot signifies that the data is ready for reporting and entry into the computer.

      1) After the data has been entered into the computer a printout will be

generated. The data on the red-dotted summaries will be cross-checked against the computer printout. All errors on the printout will be corrected in the computer.

3. Supervisor Review

A. All analytical results will be reviewed by the Laboratory Director before dissemination to outside agencies. The data will be checked for compliance with established control limits, that results were within the range of the standard curve, method blank quality, current trends and both state and federal requirements. Samples may be subjected to repeat analysis (with dilution if necessary) if any of the above items are not met.

B. If a test is repeated because the initial result fell outside the current trend or exceeded the state or federal requirement, but was within control limits, both the initial and repeat test results will be averaged and reported.

C. If the test was repeated because the QCS were outside the control limits, only repeat analytical results whose QCS were within control limits will be reported.

IV. ANALYTICAL METHODS - June 30, 2010

The following methods are employed for all reported Analyses. Exceptions will be stated where a different procedure was used.

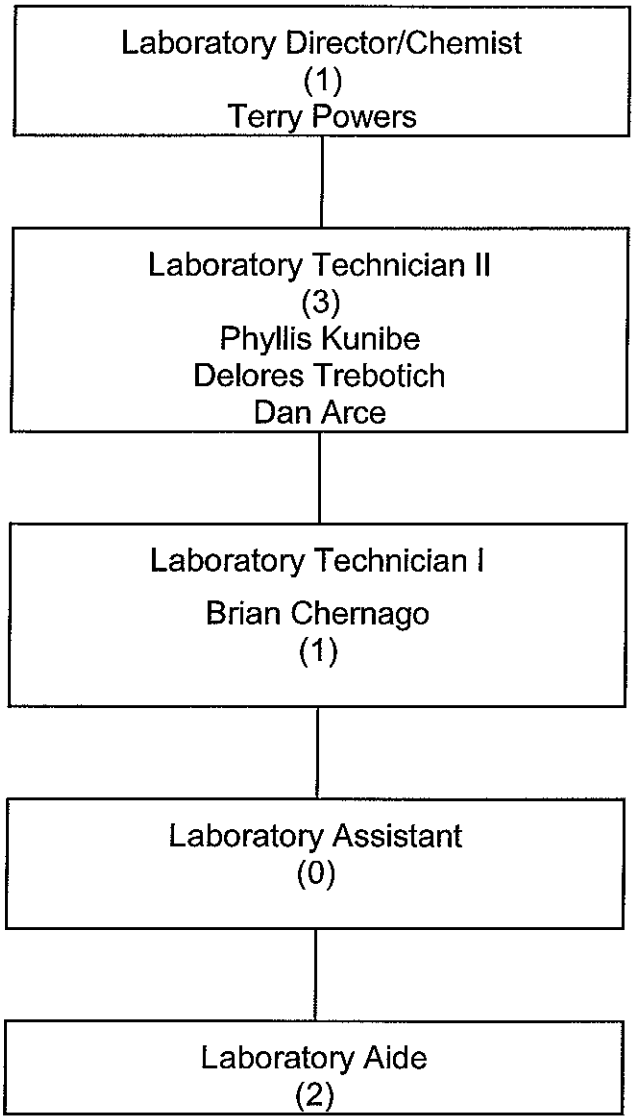
TEST	METHODS
Alkalinity	EPA 310.1
Ammonia-Nitrogen	EPA 350.1 (Automated Phenate)
BOD	Standard Methods, 18th ed., sec 5210 B
Boron	Standard Methods, 18th ed. sec 4500-B
Calcium	EPA 215.2 / EPA 215.1
CaO	STPUD Method
Chloride	EPA 325.3 / EPA 325.2 (Automated Ferricyanide)
Chlorine	EPA 330.1
COD, low range	HACH Chemical, Spectrophotometric
COD, high range	HACH Chemical, Spectrophotometric
Conductivity	EPA 120.1
Color	Standard Methods, 18th ed. sec. 2120 B
Total Dissolved Solids	EPA 160.1
Hardness	EPA 130.2
MBAS	Standard Methods, 18th ed. sec. 5540 C
Nitrate-Nitrogen	EPA 352.1, Brucine, wastewater, high levels
Nitrate-Nitrogen	EPA 353.2, Cadmium Reduction, low level
Nitrite-Nitrogen	EPA 353.2 / EPA 354.1
Oil & Grease	Standard Methods, 18th ed. sec. 5520 B
Organic-Nitrogen	EPA 351.2, Semi-Automated Block Digestion
pH, water	EPA 150.1
Phosphorous, total	EPA 365.1 / EPA 365.2
Phosphorous, ortho	EPA 365.1 / EPA 365.2
Sulfate, Turbidimetric	EPA 375.2 / EPA 375.4
Sulfides	Standard Methods, 18th ed. sec. 4500-S^{2-} D
Suspended Solids	EPA 160.2
Total Kjeldahl Nitrogen	EPA 351.2
Turbidity	EPA 180.1
CEC	UC Davis Handbook, method S-19.0
pH, soil	UC Davis Handbook, method S-3.0
Saturated Soil Paste	UC Davis Handbook, method S-1.0
Soil Saturation %	UC Davis Handbook, method S-2.0
Saturation Extract	UC Davis Handbook, method S-5.0
Soil Conductivity	UC Davis Handbook, method S-6.0
Soil Nitrate-Nitrogen	STPUD Method
Soil Ammonia-Nitrogen	STPUD Method
Soil Total Nitrogen	UC Davis Handbook, method S-30.0
Extractable Phosphorus	STPUD/Davis: Ascorbic acid detection
Soil Sulfate	Methods of Soil Analysis, sec. 79-4.2

EPA = Agency *Methods for Chemical Analysis of Waters and Wastewaters, March 1983*, U.S. Environmental Protection

Methods of Soil Analysis, American Society of Agronomy, 1965

Water, Soil and Plant Tissues, Tentative Methods of Analysis for Diagnostic Purposes, by J. Quick, University of California, Davis, Agricultural Extension Laboratories.

X. LABORATORY ORGANIZATION
SOUTH TAHOE PUBLIC UTILITY DISTRICT



XI. CALIBRATION AND MAINTENANCE OF INSTRUMENTS

1. All Laboratory equipment will be maintained on a routine schedule. A maintenance log book will be kept with records of calibration and maintenance. Instruments requiring daily calibration will have monthly log sheets attached to the instrument. These log sheets will be kept on file for a minimum of five years.

2. Equipment operation manuals will be filed in the Laboratory office file cabinet for easy access to all analysts and instrumentation techs.

3. Following are the procedures for calibration of balances, D.O. meters, spectrophotometers, pH meters, and automatic samplers

LABORATORY WATER SYSTEM

1. The laboratory has three types of water: tap or community (#1), Type II (single-stage deionized water) and deionized water (DW or multi-stage deionized water).

   A. Tap water is groundwater that has been treated with chlorine, corrosion inhibitors and sequestering agents. Tap water, both cold and hot, is used for preliminary cleaning of laboratory equipment. Detergents are used with tap water. Tap water can also be used for the preliminary rinse of laboratory equipment. Tap water cannot be used for the final rinsing of lab equipment, to prepare reagents, nor bacterial culture media.

   B. Type II water is ε1 megohm water. It is tap water that has had the chlorine removed with an activated carbon filter, minerals removed with single-stage deionization. Type II water is used to fill the water baths and rinse cleaned glassware after tap water rinsing. It can be used to make chemical solutions used for on-line instruments, such as chlorine analyzers. It is not to be used for lab reagent preparation nor for final rinsing of lab equipment used in metals or organic sampling and analysis.

      1) The electrical conductivity of the Type II water is measured daily. The Type II water unit should be serviced when the EC is ≤ 1.0 µmhos for two successive days.

   C. DW water is ε 10 Megohm water. It is RO water that has gone through organic removal, demineralizer, and sub-micron (0.2 µm) filters. It is used for reagent preparation and final rinsing of equipment used in metal and organic sampling and analysis.

      1) The electrical resistance of the DW is measured daily. The complete set of cartridges should be replaced when the resistance is +10 Megohms. The 0.2 µm filter should be replaced when the HPC is ≥ 10 CFU.

2. **Type II Water and Deionizer Cartridge Replacement**
   A. The following instructions are for the Siemens Type II and Barnstead E-Pure units.

3. **<u>Siemens SDI-01 Type II Water</u>**

      1) Unplug Inlet Influent line solenoid valve.
      2) Turn off Influent and Effluent water lines.
      3) Open needle drain valves to drain water. Close when lines are drained
      4) Remove Tank #1 (used tank) and place next to door for pickup.
      5) Move DI Tank #2 to Tank #1 position. Make sure to switch In-line 200 K ohm light to Tank #1 Effluent side (position between Tank #1 and Tank #2).

6) Place new tank in Tank #2 position.
7) Secure all connections. Tighten ONLY HAND TIGHT.
8) Make certain needle drain valves are closed.
9) Turn on both Influent and Effluent Water lines.
10) Plug in Inlet Influent line solenoid valve electric plug.
11) Log gallons meter reading and tank change
12) Call for new tankMembrane Installation

4. **E Pure**

A. Replacement of full set of cartridges: Instructions can be found in each replacement kit of cartridges (contains all 4 cartridges), not in single cartridge boxes. When removing old cartridges, drain canisters of water and rinse prior to cartridge replacement.

1) Remove 1st cartridge (left-most canister)
2) Replace first cartridge (left-most canister).
3) Remove final filter (0.2 µm)
4) Leave remaining old cartridges in place.
5) Turn system on.
6) Run to drain for 15 minutes.
7) Shut system off.
8) De-pressurize system
9) Replace cartridges in last three canisters.
10) Turn system on.
11) Run to drain for 5 minutes
12) Replace final filter (0.2 µm filter).
13) Flush 2 liters prior to use.

STANDARDIZING SPECTROPHOTOMETER TEST PROCEDURES

Using the HACH DR4000 and Spectronic Spec Standards

A. 0% Transmittance TesT

1. Install the Single-Cell module. Turn on the UV lamp.

2. Select the Single λ (wavelength) mode.
3. Select View: %T
4. Insert the Control Standard (the one with no glass) in the instrument.
5. Set the wavelength to 400nm.
6. Set the meter to read 100%T by Zeroing.
7. Turn the control standard 90E to block light path.
8. Record the result, which is the 0%T reading.
9. If the 0%T reading exceeds tolerances, correct it by repeating steps 1 through 4 before proceeding.

B. Stray Radiant Energy Test

1. Select the Single λ (wavelength) mode.
2. Select View: %T
3. Insert the control standard (the one with no glass) in the instrument.
4. Set the wavelength to 400nm.
5. Set the meter to read 100%T by Zeroing
6. Replace the control standard with the SRE (Stray Radiant Energy) 400nm standard. Observe the %T reading and record it on the sheet.
7. Insert the control standard again.
8. Set the wavelength to 340nm.
9. Set the meter to read 100%T by Zeroing
10. Replace the control standard with the SRE 340nm standard. Observe the %T reading and record it on the sheet.
11. Insert the control standard again.
12. Set the wavelength to 220nm. The UV light must be turned on for at least 10 minutes.
13. Set the meter to read 100%T by Zeroing
14. Replace the control standard with the SRE 220nm standard. Observe the %T reading and record it on the sheet.

C. Wavelength Accuracy Test

1. Only those peaks within the wavelength range of the spectrophotometer will be detected by the wavelength accuracy test. The spectrophotometer can detect the three first-order peaks transmitted by the wavelength evaluation standard and automatically block the second-order peak. (On the diagram in the certificate provided with your standards, the first-order peaks are shown as solid lines; the second-order peak is shown by a dashed line.)
2. Select the Scan λ (Scan mode).
3. Select View: ABS

4. Set the Options for λ Min, λ Max and λ Step.
5. Set the λ interval from 395 - 405 nm in 0.1 nm steps.
6. Insert the Control Standard and perform a Baseline by pressing the Baseline soft-key.
7. Insert the 525.3nm wavelength evaluation standard in the cuvette holder.
8. Press the Start Scan soft-key to scan the wavelengths.
9. After scan is completed, press the CURSOR soft-key, then the · ¹ soft-keys to select the λ of maximum absorbance.
10. Record the wavelength (not the absorbance) where this peak is found. This wavelength should match the certified wavelength within the tolerances given in table 1.
11. Repeat steps 3-8 using the following parameters:

    a) λ interval from 520 - 530 nm in 0.1 nm steps (for 525.3 nm peak)
    b) λ interval from 777 - 787 nm in 0.1 nm steps (for 782.4 nm peak)

D. **Phometric Accuracy/Linearity Test**

1. Select the Single λ (wavelength) mode.
2. Select View: %T
3. Insert the control standard (the one with no glass) in the instrument.
4. Set λ to 590nm.
5. Using the Control Standard, adjust the instrument to read exactly 100%T by Zeroing.
6. Insert the 45.9 %T590 photometric performance standard. Record the instrument reading. The reading should conform to the tolerances in table 1.
7. Insert the 48.1 %T590 standard. Record the instrument reading. The reading should conform to the tolerances in table 1.
8. Insert the 9.7 %T590 standard. Record the instrument reading. The reading should conform to the tolerances in table 1.
9. Insert the 9.9 %T590 standard. Record the instrument reading. The reading should conform to the tolerances in table 1.

TABLE 1

HACH DR4000

Test	Maximum Acceptable Deviations
Stray Radiant Energy	0 - 0.05%T
Wavelength Accuracy	+/- 1 nm
0 %T	0.0
Photometric Accuracy	+/- 0.5%T

SPECTROPHOTOMETERS

1. This is a description of the procedure for starting up and using a UV/Visible spectrophotometer.

A. STARTUP

1) Remove the dust covers from the spectrophotometer and sample pump.

2) Turn on power to spectrophotometer and sample pump. Power should be turned on at least 10 minutes before readings are to be taken to obtain a stable light source.

3) Secure sample sipper to peristaltic pump.

4) Place sample tubing in a beaker of deionized water and sip up water.

B. OPERATION

1) Set spectrophotometer to analytical wavelength.

2) Make certain instrument is set to ***Absorbency***

3) Set wavelength to desired reading.

4) Place sample tubing in a beaker of deionized water and introduce DI water into sample cell.

5) Set Absorbency to **0.000**.

6) Place sample tubing in reagent blank and depress sipper lever.

7) Read absorbency of reagent blank (compared to deionized water) and record in parentheses.

8) Set absorbency of reagent blank to **0.000**.

9) Wash cell by either sipping deionized water or air (you can also use the next sample to wash cell by sipping two aliquots of sample and take reading of second aliquot.)

10) Place sample tubing in either standard or sample and sipper sample.

11) Wait till absorbency readings stabilize and record result. These results have now been corrected against a reagent blank. Stabilization time is dependant on type of sample but usually takes no more than 10-15 seconds. Some tests, such as Brucine nitrates, can require up to 60 seconds before stabilization is complete.

C. SHUTDOWN

1) Place sample tubing in beaker of deionized water and wash with ten aliquots of water.

2) Disconnect pump tubing from pump.

3) Shut off spectrophotometer and sample pump.

4) Cover pump, spectrophotometer and power unit with dust covers.

ORION DISSOLVED OXYGEN PROBE CALIBRATION

1. The following is a description of the calibration of the Orion 97-08 DO electrode using the Orion 920 Ion Analyzer:

2. Determine barometric reading in mm mercury and divide answer by 100 to obtain mg/L oxygen.

3. Connect Orion DO electrode to electrode 2 on the Orion EA 920 pH meter.

4. Select the **O_2** mode on EA 920 pH meter, using the **mode** key.

5. Select **SAMPLE** function using the **display** key. Press **enter**. Pressing **enter** automatically zeroes the EA 920 and sets slope at 100%.

6. Choose the display resolution, **.1, .01, or .001,** by pressing and holding the **electrode** key while pressing the **x10** key.

7. Turn **mode** switch on electrode to **BT CK**. Good battery operation is indicated by a reading of **13.00** or greater on meter.

8. Turn **mode** switch on electrode to ZERO. Use zero calibration control to set meter to read **0.00**.

9. Insert funnel into a BOD sample bottle containing enough water to just cover bottom of bottle. Insert electrode, making sure that electrode tip is not immersed in the water and does not have water droplets clinging to outside of membrane. Let stand for 30 minutes to ensure water saturation of air in BOD bottle (if probe has been stored in this bottle there is no need to wait 30 minutes). Also use this bottle for storage between measurements..

10. Turn electrode mode switch to AIR position. Use AIR calibration control to set the EA 920 reading to the prevailing barometric pressure (divided by 100).

11. Turn electrode mode switch to **H_2O** for sample measurements.

SIRCO AUTOMATIC SAMPLER CLEANING

1. This is a description of the procedures to routinely clean SIRCO automatic samplers.

   A. Equipment

      1) Deionized water in 1 liter spray bottle
      2) Clean measuring chambers, if required
      3) Test tube brush
      4) Bottle brush (large diameter)
      5) Rags or paper towels (2 or 3 rags or 10 paper towels)
      6) Rubber gloves. A face shield is not a bad idea, either.
      7) Bucket

   B. It is a good idea to remove anything that might fall out of your lab coat, such as pens, forceps, etc. This is because the floor by the sampler is a grating.

   C. Procedure

      1) Open refrigerated sample compartment and remove sample bottle. Place bucket under sample outlet hose.

      2) Open door of sampler control panel. Turn off sampler with master **POWER** switch. Remove the two (2) wing-nuts at the top of the measuring chamber. Carefully set nuts to one side of compartment.

      3) Lift up chamber top and remove measuring chamber from below. Clean inside of chamber with brush and rags (or replace with clean measuring chamber). Use spray bottle to rinse scum into bucket.

      4) Wipe off stainless steel tubes on bottom of measuring chamber lid with a paper towel or rag. Use tube brush to clean inside of discharge tube and sample lines.

      5) Re-assemble unit, making sure chamber rests correctly on O-ring and replace wing nuts.

      6) Make certain obstructions (rags) are removed from intake hose in sample stream.

      7) Turn **POWER** switch ON and hit **RESET** and then the **MANUAL** sample button. Watch sample cycle (about 1-2 minutes). Make sure O-ring does not leak and final sample volume is less between 50 and 75 mL. Repeat this one more time. This will clean the sample outlet hose.

8) If sampler is set for ***Proportional/Flow***, adjust counts as follows:

Desired action	Count Control
Increase sample jug volume	Decrease counts
Decrease sample jug volume	Increase counts

9) Remove bucket from refrigerated sample compartment and replace the sample jug.

10) Record cleaning on sample composite log and initial.

GENERIC AUTOMATIC SAMPLER CLEANING

1. This is a description of the procedures to routinely clean automatic samplers.

   A. Equipment

      1) Deionized water in 1 liter spray bottle
      2) Test tube brush
      3) 600 mL beaker
      4) Bottle brush (large diameter)
      5) Rags or paper towels (2 or 3 rags or 10 paper towels)
      6) Rubber gloves. A face shield is not a bad idea, either.
      7) Bucket

   B. It is a good idea to remove anything that might fall out of your lab coat, such as pens, forceps, etc. This is because the floor by the sampler is a grating.

   C. Procedure

      1) Open refrigerated sample compartment and remove sample bottle. Place bucket or beaker under sample outlet hose.

      2) Wipe all surfaces with damp rag.

      3) Clean all dippers, tubing, funnels and discharge outlets with brush.

      4) Rinse all dippers, tubing, funnels and discharge outlets with deionized water.

      5) If present, remove plastic dams and allow sample flow to scour flow channel clean.

      6) Replace dams, if present.

      7) Make certain obstructions (rags) are removed from any intake hoses in sample stream.

      8) Make certain mode switch is set for proper setting (***Proportional*** or ***Flow*** for Raw and Final samplers, ***Time*** or ***Constant*** for Primary and Secondary effluent samplers).

      9) Adjust flow of sample stream, if it is controlled by valve. Make certain flow through models have flow stream high enough to fill dipper during sample episode.

10) If sampler is set for ***Proportional/Flow***, adjust counts as follows:

Desired action	Count Control
Increase sample jug volume	Decrease counts
Decrease sample jug volume	Increase counts

11) Remove bucket from refrigerated sample compartment and replace the sample jug.

12) Record cleaning on sample composite log and initial.

pH METERS

1. STANDARDIZATION

   A. All standardizations should use two calibration standards which bracket the sample pH.

   B. Make sure pH meter is set for pH mode and the correct electrode is selected.

   C. Make certain the pH probe has both the correct electrolyte and sufficient electrolyte. The electrolyte level should be close to the filling hole. This will prevent backflow of sample into the pH probe. If too many crystals are clogging the junction, empty probe of internal filling solution, rinse several times with DI water, the two times with electrolyte. Consult electrode manual for further information of electrolyte maintenance. Fill probe with electrolyte.

   D. Rinse electrodes with deionized H_2O and place in pH 7.00 buffer. When reading indicates a stable reading press enter.

   E. Rinse electrodes with deionized H_2O and place in pH 10.00 or 4.00 buffer. When reading indicates a stable reading press enter.

   F. Record standards used and slope on pH standardization QA form.

2. SAMPLE MEASUREMENT

   A. Rinse electrodes with deionized H_2O and place in sample. Make certain mode is **pH** and meter is on.

   B. When making pH measurements in the laboratory, place a Teflon stir bar in the sample container (usually a beaker) and place sample on magnetic stir plate. Set stir speed so that sample does not splash out of beaker.

   C. Press the button to measure pH (this may be ***pH***, =, or **Read**)

   D. Once the meter and electrode has reached a stable reading (indicated by a prompt such as ***Ready*** or some other symbol) record the reading.

   E. To make next measurement repeat steps 1-3.

3. These meters never go wrong. If there is a problem, 99.9999999 % of the time it is a faulty electrode. However, check that the battery is good.

   A. Check that the filling solution inside the electrode is higher than the level of the sample.

B. Determine if there are too many crystals forming inside the electrode. Use pipet to remove overly-saturated KCl solution, rinse with DI water. Rinse with fresh fill solution, then refill with fill solution.

C. Is the fill-hole covered (it shouldn't be).

D. Try alternately soaking in dilute (0.1 N) HCl and NaOH in one minute intervals.

E. Clean or replace ceramic junction.

F. Soak in 80EC water for 5 minutes.

OHAUS ELECTRONIC BALANCE
MODEL G 160D

1. **WEIGHING**

   A. Press the **ON/OFF** switch and the display will show the following for about 3 seconds:

      1. $\pm$ 8.8.8.8.8.8.8.$_g$

      2) This indicates that all display segments are working properly. The balance will then do a self diagnostic check. If all functions are operating properly the balance will then display zero.

   B. **TARE** the balance by pressing the **RE-ZERO** switch. Before doing so, make certain both side doors and the top one are closed. Make certain the balance is in the 160 gram range; there should be only 4 zeroes to the right of the decimal. If there are 5 zeroes to the right of the decimal press the **RANGE** switch. When tared the balance display should look like:

      0.000 g

      1) It may take several seconds to tare the balance. The busy light, a $_o$ on the lower left side of the display, will appear until taring is complete.

   C. Place the filter on the center of the pan. Again, close all three doors.

   D. When the **g** appears to the right of the last decimal record the reading (the **g** indicates the reading is stable).

   E. To make next measurement repeat steps 2-4.

   F. To turn balance off press the **ON/OFF** switch.

Notes:

1. Make certain that the window is closed when using the balance. Drafts will cause the balance to give erratic readings.

2. Never lean on the balance table, especially when you are weighing. Pressing against the table will give erratic results.

3. The knob on the lower left side of the balance is for calibrating in both the 30 gram and 160 gram ranges. Please do not use this knob. Lab staff will perform calibrations on a routine basis.

YSI MODEL 57, ANALOG DISSOLVED OXYGEN METER

1. **STANDARDIZATION**

   A. Use plastic bottle calibration chamber. Insert Kimwipe into bottom of bottle and wet with DI water. Push DO probe through bottle opening until the bottle fits snug on the electrode. Make certain no water droplets are on the membrane.

   B. Set function switch to ***RED LINE*** and adjust needle to the red line. This adjusts the power output. Use the mirror backing to optimize adjusting the indicator needle. You should not see a mirror image of the needle.

   C. Set function switch to **ZERO**. Adjust to 0.0 with **ZERO** control knob.

   D. Set function switch to ***TEMP*** and allow to equilibrate for 15 minutes.

   E. Determine the mg/L D.O. for the indicated temperature and the correction factor for the present pressure or elevation. There is a table on the back of the meter for each correction factor. Lake Tahoe's elevation is 6200" (0.80). Woodfords is 5600" high (0.81). The field sheet clip boards have a table of temperature adjusted D.O. readings corrected for the Woodfords area.

      1) If determining the pressure / temperature adjusted DO, multiply the appropriate pressure correction factor by the temperature adjusted D.O. value. For example, if the temperature of the electrode was 21°C and you were at Woodfords:

         (0.81 H 9.0) = 7.29 mg/L

   F. Set function switch to the **0-10** or **0-20** range and adjust the needle to the appropriate calibration value from step E. Wait two minutes to verify calibration stability. Readjust if necessary.

   G. The probe is now calibrated and ready for measurements.

2. MEASUREMENT

   A. Standardize meter as described above.

   B. Turn function switch to desired range.

   C. Place probe in sample, aeration basin, stream or reservoir. No fly casting is allowed.

   D. Water movement of 1 foot per second is required for accurate measurement.

You should lift probe up or down or swing sideways though water to achieve this. Do not allow probe to sit on bottom of basin, stream or lake; this will give false low readings.

E. When reading has stabilized record displayed value.

3. PROBLEMS

A. Most problems are a result of faulty membrane, low batteries or a break in the DO cable sheath.

B. A low battery.

C. The membrane should be replaced every two weeks of operation or whenever a bubble forms in the electrolyte or when scum has fouled the membrane surface. Replace membranes as per instructions in attached leaflet.

YSI MODEL 58, DIGITAL DISSOLVED OXYGEN METER

1. **STANDARDIZATION**

A. Use stainless steel calibration chamber. Insert solid stopper into bottom of chamber. Push DO probe through hollow stopper until the small end of stopper is at the top of the notch where the pressure compensation unit is located (7). Place probe in calibration ring (3) and place assembly in bucket of fresh water for 5 minutes; this permits probe to come to the same temperature as sample. Wet the inside of chamber with fresh water. Drain excess water from the chamber, shake any droplets from the probe membrane and promptly insert probe into the calibration chamber. Make certain a chamber is sealed.

B. Place chamber in bucket of water for additional 5 minutes for final temperature equilibration.

C. Set function switch to **ZERO**. Adjust **O_2 ZERO** until display reads **0.00**.

D. Set function switch to **%**.

E. Allow 15 minutes for probe to stabilize in moist air in chamber. Unlock and adjust **O_2 CALIB** control until meter reads **80.0**; relock **O_2 CALIB** control. Unit is now standardized. Moist air at this altitude will hold 80.0% of the oxygen air at sea level will.

2. **MEASUREMENT**

A. Standardize meter as described above.

B. Turn function switch to **0.1 mg/L** or **0.01 mg/L**

C. Place probe in sample, aeration basin, stream or reservoir. No fly casting is allowed.

D. Water movement of 1 foot per second is required for accurate measurement. You should lift probe up or down or swing sideways though water to achieve this. Do not allow probe to sit on bottom of basin, stream or lake; this will give false low readings.

E. When reading has stabilized record displayed value.

3. **PROBLEMS**

A. Most problems are a result of faulty membrane, low batteries or a break in the DO cable sheath.

B. **LOBAT** will appear automatically whenever the batteries need replacing (when 5 hours life, or less, remain). Four D cell batteries are required. The BATT CHK on the stirrer knob does **not** indicate meter battery status.

C. The membrane should be replaced every two weeks of operation or whenever a bubble forms in the electrolyte or when scum has fouled the membrane surface. Replace membranes as per instructions in attached leaflet.

APPENDIX A
Sample Preservation

from EPA: *Methods of Chemical Analysis of Water and Wastewater*, 1983

APPENDIX B

FORMS

Sampling Forms

1. *Field Monitoring Record*
2. *Composite Sample Data*
3. *Composite (manual) Sampling Record*
4. *Laboratory Sample Log*
5. *Samples Tracking Log*
6. *Sample Chain of Custody Record*
7. *Samples sent to Commercial Laboratories Preparation Guide*
8. *Repeat Analysis or Unfinished Analyses*

Equipment Calibration Forms

1. *Bacterial Media Preparation & Sterilization (Autoclave)*
2. *Sterilization Indicator*
3. *Sterile Sample Bottle Sterilization Record*
4. *Bacterial Pipet Sterilization Record*
5. *Temperature Monitoring (Coliform)*
6. *Autoclave Temperature Check*
7. *Used Media Sterilization Record*
8. *RO & Deionizer Water Quality Record*
9. *Balance Calibration Quality Control Record*
10. *Spectronic Standards Test Log*
11. *pH Meter Calibration Recor*
12. *Turbidimeter Calibration Quality Control Record*
13. *Weekday Cleanup Schedule*
14. *Weekend Cleanup Schedule*

Test Worksheets

1. *Alkalinity*
2. *Ammonia Nitrogen*
3. *BOD*
4. *Boron*
5. *Chloride*
6. *Chloride, Hardness and Calcium (EDTA)*
7. *Chlorine*
8. *COD, Spectrophotometric (HACH)*
9. *COD, Oven - Reflux*
10. *Color*
11. *Daily Physical Tests: pH, Turbidity, Settleable Solids, Alkalinity and Chlorine*
12. *Fluoride by electrode*
13. *MBAS*
14. *Nitrite - Manual Spectrophotometric*
15. *Oil & Grease*
16. *pH, Solids, Soils and Wastes*
17. *Phosphorus, Total*
18. *Phosphorus, Ortho*
19. *Sulfate, Water - Turbidimetric*
20. *Sulfate, Soils - Turbidimetric*
21. *Suspended Solids*

22. *TDS*
23. *Turbidity*

Bacterial Worksheets

1. *Bacteriological Sampling Instructions*
2. *Sewer Coliforms*
3. *Drinking Water Weekly Coliform Field / Test Worksheet*
4. *Coliform Worksheet*
5. *Drinking Water Repeat Coliform Worksheet*
6. *Colilert Worksheet*
7. *Completed Test Worksheet*
8. *Heterotrophic Plate Count Worksheet*
9. *Quality Assurance Worksheet*
10. *API Bacterrial Identification Worksheet*
11. *Feelab Drinking Water Colilert Worksheet*