

~~44-01~~ 44-01

Carmen Johnson
Fac/Perm/Co ID # 44-01 Date ~~3/26/12~~ Doc ID#

~~4/4/12~~

**INTERIM REPORT
PRELIMINARY DATA REVIEW
FOR LANDFILL NUMBER 6**

Canton, North Carolina

Volume II

Volume 2

Prepared by:

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5 December 1994

Appendix F

APPENDIX F

PRELIMINARY GROUNDWATER SAMPLING AND ANALYSIS PLAN

1.0 GROUNDWATER LEVEL DATA COLLECTION

Prior to performing groundwater sampling, static fluid level measurements will be obtained from the wells. All measurements will be completed on the same day.

The depth to groundwater will be measured in the well using an electronic water level probe. The depth to water will be measured to the nearest 0.01-foot, referenced to the surveyed top of the PVC well casing. The total depth of each well will also be measured to the nearest 0.01-foot using the water level indicator. The water levels and well depth measurements will be used to calculate the volume of water in each well and the minimum and maximum volume of water that must be purged prior to sampling.

All downhole water level equipment will be decontaminated by washing in a solution of Alconox and potable water, rinsing in potable water, and final rinsing using commercially available distilled water.

2.0 GROUNDWATER SAMPLING PROCEDURES

Upon completion of the liquid level and total depth measurements, the standing water in the well will be purged. A minimum of three calculated well volumes will be purged from the wells prior to sampling (unless the well is purged dry). Purging will be conducted by either pumping with a clean stainless steel electric submersible pump or by repeatedly lowering and retrieving a disposable polyethylene bailer into the well. Bailers will be tethered with new, clean monofilament line. Well purging will be considered complete when pH, conductivity, and temperature are consistent as defined by ± 0.1 pH units, $\pm 10\%$ conductivity, and $\pm 1^\circ$ Celsius (with a minimum of three measurements taken), or when a maximum of five well volumes have been removed. If the well is pumped or bailed dry, purging will be considered to be complete and an appropriate note will be recorded in the field log. During purging activities, the pH, specific conductance, and temperature measurements will be recorded in the field logbook.

After well purging is completed, groundwater samples will be collected from the monitoring well. Groundwater samples will be collected from the wells using a disposable polyethylene bailer. The disposal bailers will not be reused between wells. The bailer will be lowered into and retrieved from the well, and groundwater from the bailer will be immediately poured into appropriate, laboratory-prepared sample containers, including preservatives where appropriate. Each bottle will be properly labeled, placed on ice in a cooler, and sent to the laboratory for analysis. Chain-of-custody documentation will accompany the samples to the laboratory.

Parameters to be sampled for, including bottle type and volume, and preservative (if required) are listed below:

Parameter	Container Size and Type	Preservative
Total Metals*	One liter polyethylene with polyethylene-lined closure	HNO ₃
Dissolved Metals**	One liter polyethylene with polyethylene-lined closure	HNO ₃
Phenol	One liter amber glass with Teflon-lined closure	H ₂ SO ₄
TOX/TOC	One liter amber glass with Teflon-lined closure	H ₂ SO ₄
NO ₂ ,NO ₃	One 500 ml polyethylene with polyethylene-lined closure	H ₂ SO ₄
Cl,Fl,SO ₄ ,TDS,pH, Conductivity	One liter polyethylene with polyethylene-lined closure	None

* Metals include: iron, potassium, lead, magnesium, manganese, nickel, selenium, sodium, zinc, mercury, silver, arsenic, barium, cadmium, calcium, chromium and copper.

** Collected in unpreserved, one liter plastic bottle and filtered into preserved, one liter plastic bottle.

For dissolved metals, it is noted that the sample must be filtered in the field. The sample is first collected in an unpreserved, one-liter plastic bottle, then filtered through a 0.45-micron membrane filter and into a one-liter plastic bottle preserved with HNO₃. Filtering must be initiated within fifteen minutes of sample collection. The pH of the metals sample must be less than two. This is tested by pouring a small amount into a clean jar and testing with litmus paper. This test sample is then disposed of and the jar rinsed.

3.0 SAMPLE DOCUMENTATION IN THE FIELD

Four kinds of documentation will be used in tracking and shipping analytical samples:

- Bound field logbook.
- Standardized data recording forms.
- Sample labels.
- Chain-of-custody records.

At a minimum, the label for each sample container will contain the following information:

- Site name.
- Sample number.
- Date and time of collection.
- Sampler's initials.
- Sample preservation and preservative used.
- Type(s) of analyses to be conducted.

The sample information, as well as the analyses to be performed on the sample, will be entered in the field logbook for each sampling point. If an error is entered into the field log, a single line will be drawn through the error and initialed by the person completing the log. All entries into the sample log, data recording forms, chains-of-custody, sample labels, etc. will be made using an indelible ink pen.

Additionally, the following items will be entered into the field logbook:

- Dates and times of entry.
- Sample depth and number.
- Names of field personnel on-site.
- Names of visitors on-site.
- Field conditions.
- Description of activities.
- Sampling remarks and observations.
- QA/QC samples collected.
- List of photographs taken.
- Sketch of site conditions.

4.0 SAMPLE HANDLING AND TRANSPORTATION

All samples will be kept on ice from the time of collection and forwarded on a daily basis to the laboratory by the on-site geologist. Custody of the samples will be maintained by WESTON personnel from the time of sampling until the time they are forwarded to the analytical laboratory.

Documentation of the sample custody will be maintained on a standard chain-of-custody (COC) record. The following information will be entered on the COC record:

- Project name.
- Signature of sampler.
- WESTON sample number, date, and time of collection, grab or composite sample designation, and sample media.
- Signatures of individuals involved in sample transfer.

WESTON field personnel will complete a COC record to accompany each cooler forwarded from the site to the laboratory. If an error is entered onto a COC record, a line will be drawn through the error and initialed by the person completing the form. The original copy will be placed in a sealable bag and put inside the appropriate cooler, secured to the cooler's lid. The laboratory sample custodian, or his/her representative accepting the sample shipment, whether it is from the commercial carrier or WESTON, will sign and date the COC record upon sample receipt. The original COC record will be returned with the final data report. The laboratory will be responsible for maintaining internal logbooks and records that provide a custody record during sample preparation and analysis.

5.0 DECONTAMINATION PROCEDURES

Purging equipment (i.e, submersible pumps and hoses) will be decontaminated before initial use and after each well to reduce the possibility of cross-contamination occurring between wells. Scrub pump and cord in a solution of Alconox and potable water, pump at least 20 gallons of soapy water through pump, rinse with potable water, pump at least 20 gallons of potable water through pump, and rinse with commercially available distilled water.

A new disposable bailer will be used to sample each well, so no decontamination of bailers is required.

The water level probe and probes for the pH, temperature and conductivity meters will be thoroughly rinsed with distilled water between each use.

6.0 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL

The laboratory will follow the QA/QC requirements in accordance with the State of North Carolina DEHNR guidelines and applicable EPA guidelines. The laboratory will follow the procedures as described in the applicable guidelines in determining laboratory QA/QC requirements, including, but not limited to, laboratory duplicates, reagent and method blanks, and matrix spike duplicate samples. Holding time requirements as required by the appropriate analytical method will be identified and samples will be analyzed within that time frame. Attachment A presents the laboratory QA/QA program for Hydrologic Laboratories, Incorporated (the laboratory providing analytical services for the current monitoring program).

ATTACHMENT A
LABORATORY QA/QC PROGRAM

HYDROLOGIC, INC.

Quality Control/ Quality Assurance
Documentation

of

The Asheville Laboratory Group

and

The Frankfort Laboratory Group

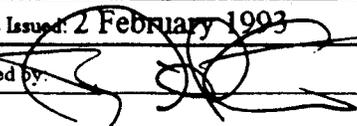
HYDROLOGIC, INC.

Analytical Procedures

ASHEVILLE LABORATORY

HYDROLOGIC, INC.

Analytical Procedure

Title: Alkalinity - Titrametric	
Date Issued: 2 February 1993	Supersedes:
Issued by: 	

Safety:

Lab coat
Safety glasses

Equipment:

pH meter
Stir plate with stir bars
Buret 50 ml
Pipets (various sizes)
Beaker 250 ml

Reagents:

0.1 N Sulfuric acid (H_2SO_4) Ricca cat. #2550-1
0.02 N Sulfuric acid (H_2SO_4) Preparation: To a 500 ml volumetric flask transfer 100.0 ml of 0.1 N H_2SO_4 . Dilute to volume with deionized water. Shake to mix.
0.02 N Sodium carbonate reagent (Na_2CO_3) Preparation: Dry a quantity of anhydrous Na_2CO_3 at 110°C for three hours. Weigh to the nearest 0.1 mg 1.060 g of dried Na_2CO_3 . Quantitatively transfer to a 1000 ml volumetric flask. Add sufficient water to dissolve. Then dilute to volume with deionized water. Shake to mix.
pH 4 Buffer Ricca cat. # 1501
pH 7 Buffer Ricca cat. #1551
pH 9 Buffer Banco cat. #16450
Performance standard of known concentration

Procedure:

Standardize pH meter according to the pH procedure
0.02 N H_2SO_4 Standardization
Standardize 0.02 N H_2SO_4 as described in Total Kjeldhal Procedure (pg 2 of 3). Calculate (see calculation section) and record normality of H_2SO_4 .
Sample Determination:
Transfer 200.0 ml of sample to a 250 ml beaker.
Submerge the pH electrodes in the solution and start agitation.
Record initial pH.
Slowly titrate the solution with standard 0.02 N H_2SO_4 to pH 4.3 to 4.7. Care must be exercised with the addition of H_2SO_4 so as

not to overshoot the target pH range. Allow sufficient time for the pH to equilibrate between H_2SO_4 additions.

When the solution is between pH 4.3 and 4.7 stop titrating and record the volume of 0.02 N H_2SO_4 required. Record the intermediate pH.

Carefully add sufficient standard 0.02 N H_2SO_4 to reduce the pH an additional 0.30 units. Record the volume required and the final pH.

Calculate (see calculation section) the alkalinity of the sample as ppm $CaCO_3$.

Calculation:

Standardization

$$N H_2SO_4 = \frac{(N Na_2CO_3) \times (vol Na_2CO_3)}{vol of H_2SO_4}$$

Sample determination:

$$\text{Total alkalinity (ppm } CaCO_3) = \frac{(2B-C) \times N H_2SO_4 \times 50,000}{\text{Sample vol (ml)}}$$

Quality Control:

Sample for alkalinity should be preserved cooling to 4°C at the time of collection. Samples must be analyzed within 14 days of collection.

The 0.02 N H_2SO_4 is standardized with each analytical session. Duplicate analysis are performed at a rate of not less than 10%.

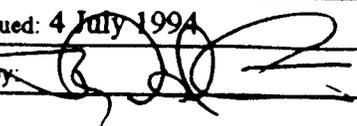
A performance standard of known concentration is run with each analytical session. The value obtained should be within +10% of the vendor's published value. If not, contact the supervisor.

All standardizations and solutions preparations are documented in the benchsheet notebook.

Proper sample preservation is verified at log-in.

HYDROLOGIC, INC.

Analytical Procedure

Title: Ammonia Nitrogen Determination	
Date Issued: 4 July 1994	Supersedes: 21 February 1992
Issued by: 	

Safety:

Lab coat
Safety glasses

Sample Preservation:

Sample requiring ammonia nitrogen determination should be preserved at the time of collection with the addition of H_2SO_4 to pH <2 and chilling to 4°C. When residual chlorine is present, this must also be destroyed at the time of collection.

Reporting limit:

0.1 mg/L N-NH₃

Equipment:

Accumet 925 pH/ISE Meter with gas sensing electrode adaptor
Ammonia Ion Selective Electrode
Magnetic stir plate and stirring bars
Volumetric flasks as required
Beakers
pH paper
Pipets as required

Reagents:

Sodium hydroxide (10N): Prepare by dissolving 100g of reagent grade NaOH in water. Allow solution to cool (Exercise caution in dissolving NaOH in water. The heat of solution is markedly exothermic and severe burns could result.) Transfer quantitatively to a 250 ml volumetric flask. Allow to cool to room temperature and dilute to volume. Label as to contents and date prepared.

Ammonia stock solution (1000 mg/L): Prepare by: Weighing to the nearest 0.1 mg (0.0001g) 1.91 g of NH₄Cl which has been previously dried for 1 (one) hour at 104°C. Dissolve in water and transfer to a 500 ml volumetric flask. Make certain that the solution is at room temperature before diluting to volume; the heat of solution for NH₄Cl is endothermic. Label and date the flask.

Ammonia working solution (100 mg/L): Prepare by: Volumetrically pipeting 50 ml of 1000 mg/L stock solution into a 500 ml volumetric flask and diluting to volume. The working solution should be prepared fresh with each analytical session.

Ammonia standard: Prepare by: Serially diluting successive volumes of ammonia working solution (100 mg/L) to produce standards which are 10.0 mg/L, 5.0 mg/L, 1.0 mg/L, and 0.1 mg/L in ammonia nitrogen respectively.

Procedure:

Instrument standardization:

Transfer the 10 mg/L standard to a 250 ml beaker, immerse the electrode in the solution, and begin moderate agitation by means of a stir bar. Care should be taken to avoid sucking air bubbles into the sample. Should this occur, reduce the stirring speed and repeat the determination (standard or sample which ever is applicable.)

Add 1 ml of 10N NaOH.

Switch the ISE meter to concentration mode by depressing the "MODE" key until concentration appears in the prompt screen. Depress the "STBY/MEAS" key. Depress the "MULTI-POINT CAL" key and at the prompt depress the 3 key and then the "ENTER" key. This manipulation need only be performed prior to standardizing on the first standard.

As prompted enter the concentration of the standard.

Allow the measurement to come to equilibrium (typically 4 or 5 stars) and depress the "ENTER" key. Record the equilibrium millivolts of the standard on the benchsheet.

Repeat this process for the 5.0 mg/L and 1.0 mg/L standard making sure to properly enter the standard concentration.

After completing the standardization the efficiency must be between -0.90 and -1.10. Failure to achieve this efficiency requires restandardization of the instrument. If after the second attempt the instrument efficiency does not meet the requirements, consult the supervisor.

Sample Determination:

Verify the calibration by running a reagent blank, followed by the lower detection limit standard (0.1 mg/L N-NH₃), the linearity check standard (5.0 mg/L N-NH₃) and finally an APG standard. These verifications are determined according to the sample determination procedure given below. Determine the % recovery of each standard as outlined in the Calculation section of this document. Acceptance criteria are outlined in the Quality Control section of this document.

Transfer a 100 ml representative aliquote of sample to a beaker. Immerse the electrode in the sample solution and with agitation add 1 ml of 10N NaOH. Verify pH by means of pH paper. If the pH is above 11 add additional 1 ml aliquotes of NaOH until the

pH is above 11. Typically 1 ml is sufficient.

Depress the "STBY/MEAS" key. When a stable reading is obtained record the concentration on the benchsheet.

Remove the electrode, rinse and blot dry.

Repeat this procedure for each successive sample.

Should a sample produce a concentration greater than 10 mg/L, dilute the sample such that the concentration of the diluted sample is between 1.0 mg/L and 10.0 mg/L N-NH₃. Apply the appropriate dilution factor as given in the calculation. See calculation section.

Calculation:

Sample dilution:

$$\text{mg/L N-NH}_3 = \frac{\text{Obs. conc.} \times \text{final volume}}{\text{Sample aliquote}}$$

Standards Recovery:

$$\% \text{ Recovery} = \frac{\text{Found value}}{\text{True value}} \times 100$$

Spike Recovery:

$$\% \text{ Recovery} = \frac{\text{Conc. of spiked sample} - \text{conc. of unspike sample}}{\text{Spike Concentration}} \times 100$$

Relative percent difference:

$$\text{RPD} = \frac{|\text{(Repl. 1 - Repl. 2)}|}{((\text{Repl. 1} + \text{Repl. 2})/2)} \times 100$$

Quality Control:

Proper sample preservation is verified at log-in. Failure necessitates that the client be notified. If the client persists in having the sample run, NCDEHNR must be notified. Preservation violations are noted on the benchsheet.

Instrument efficiency must be -0.90 to -1.10. Failure requires restandardization.

The standard curve is verified immediately after standardization by running a lower detection limit, LDL, standard (0.1 mg/L), a

linearity check standard, LCS, (5.0 mg/L), and an APG standard. Acceptable recoveries are $\pm 10\%$ or within established control limits, which ever is lowest. Failure requires that the standard be rerun. See calculation section. A second failure necessitates restandardization. If the standards still fail to recover appropriately the supervisor must be consulted.

Samples are duplicated at a minimum rate of 10%. Relative percent difference for the replicates must not exceed 20% or within established control limits which ever is lowest. See calculation section. Failure requires that both replicates be rerun. A second failure requires consultation with the supervisor.

Linearity check standard (LCS) are run at a minimum rate of 10%. Acceptance is as given after initial calibration. See calculation section.

A representative number of samples are selected quarterly and a second representative aliquote distilled prior to ion selective electrode determination. The samples should agree distilled vs undistilled within 20% RPD or within the established control limits which ever is lowest. See calculation section

References:

"Methods for Chemical Analysis of Water and Wastes," Method 350.3, EPA 600-4-79-020.

"Standard Methods for the Examination of Water and Wastewater 17th Ed.," 4500-NH3F, American Public Health Association, *et.al*, 1989.

WASTERWATER SERVICES, INC.
Analytical Procedure

Title: Biological Oxygen Demand

Date issued: 19 Sept 1990

Approved by: 

SAFETY: Use of lab coat, safety glasses and gloves

EQUIPMENT: Incubator capable of maintaining $20 \pm 1^\circ\text{C}$
pH meter Fisher Model 900
Dissolved oxygen probe Orion Model 97-08-00
300 ml BOD bottles
Various pipets
Dilution water carbouy 20 liter

REAGENTS: Phosphate buffer - Prepare by dissolving 8.5g of reagent grade $\text{KH}_2\text{PO}_4 \cdot 7\text{H}_2\text{O}$, 33.4g of $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$, 21.75g K_2HPO_4 and 1.7g NH_4Cl in 1 liter of water.
Magnesium sulfate solution - Prepare by dissolving 22.5g of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in 1 liter of distilled water.
Calcium chloride solution - Prepare by dissolving 27.5g of CaCl_2 in 1 liter of distilled water.
Ferric chloride solution - Prepare by dissolving 0.25 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in 1 liter of distilled water.
Dilution water (20 L)- Prepare by filling a carbouy with distilled water. Add 20 ml (1 ml/L) each of magnesium sulfate solution, calcium chloride solution, and ferric chloride solution. Allow the dilution water to "age" a minimum of 5 days. Just prior to use add 20 ml (1 ml/L) of phosphate buffer.
Glucose-glutamic acid solution - Prepare by weighing to the nearest 0.0001g 0.150g each of glucose and glutamic acid. (These reagents must be dried prior to weighing at 103°C for a minimum for 1 hour.)
Quantitatively transfer to a 1 liter volumetric flask. Dilute to volume. This solution yields a BOD of 200 mg/l.
Sodium sulfite solution - Prepare by weighing to the nearest 0.0001g, 0.1575g of sodium sulfite (Na_2SO_3). Quantitatively transfer to a 100 ml volumetric flask and dilute to volume. This solution must be prepared fresh daily.
Managanous sulfate Reagents Cat. # 7-24305
Alkaline-iodide-azide Ricca Cat # 540
Sulfuric acid Reagents Cat # 5-10260-4

Starch solution - Prepare by dissolving 5g of soluble starch in 900 ml of boiling water. Add 1.25g of salicylic acid and dilute to 1 liter.

Sodium thiosulfate - Prepare by weighing to the nearest 0.0001g, 3.1025 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$. Transfer quantitatively to a 500 ml volumetric flask. Dilute to volume. Standardize against potassium bi-iodate to a clear end point. (Add starch indicator when the $\text{Na}_2\text{S}_2\text{O}_3$ solution becomes straw yellow.) Record normality of $\text{Na}_2\text{S}_2\text{O}_3$ solution. The solution must be $0.020 \text{ N} \pm 0.0005$, if not reprepare solution.

Procedure: Sample Pretreatment: The pH of all samples is adjusted, if necessary, to pH 6.5 - 7.5 with sulfuric acid or sodium hydroxide. The chlorine in chlorinated samples must be neutralized. Using the volume of a sample which will be used in the dilution series, add 10 ml of 1+1 acetic acid and 10 ml of KI solution. Add starch indicator solution and mix. If the solution turns blue titrate to the clear end point with Na_2SO_3 , counting the drops required to reach the end point. If the volume of $\text{Na}_2\text{SO}_3 >$ the volume of sample the sample volume must be reduced such that the volume of Na_2SO_3 required to neutralize the chlorine does not exceed the sample volume.

Sample Dilution: Prior to diluting samples for B.O.D. perform a Winkler titration and calibrate the dissolved oxygen probe. To a 300 ml B.O.D. bottle add the desired volume of sample. Use the following criteria to assist in selecting the proper sample volume.

- the worse a sample looks or smells the lower the dilutions
- the higher the suspended solids contained in the sample, the lower the dilution
- influent samples should always be set very low
- influent samples need not be tested for chlorine or seeded.
- seed all effluent and pretreatment samples with standardized seed.

Seed the samples as required and record the volume of seed used on the bench sheet. Duplicate seed bottles should be prepared for the seed correction factor by adding the selected seed volume to a B.O.D. bottle, adding dilution water to 300 ml, determining the initial dissolved oxygen, and incubating with the

sample series. The final dissolved oxygen is determined as described below. Add sufficient dilution water to bring the final volume of sample and dilution water to 300 ml. Determine the initial dissolved oxygen of the sample using the dissolved oxygen probe. Record this value on the bench sheet. Incubate the sample at $20 \pm 1^\circ\text{C}$ for five (5) days. At the end of the five (5) day incubation period determine the final dissolved oxygen of the sample using the dissolved oxygen probe. The probe should be calibrated against the Winkler titration prior to this determination. Record the final dissolved oxygen on the bench sheet.

Calculations:

① initial dissolved oxygen (IDO) - final dissolved oxygen (FDO) = D.O. loss

loss - seed correction = net loss

dilution factor = 300ml/sample volume (ml)

② seed correction = $\left(\frac{\text{D.O. loss in seed bottle}}{\text{vol. of seed}} \right) \left(\text{vol. of seed used} \right)$

B.O.D.(mg/L) = net loss x dilution factor

Data Reporting: The following criteria are used to select the data reported

<u>F.D.O.</u>	<u>D.O. loss</u>	<u>Report</u>
≥ 1	≥ 2	Calculated BOD ₅ to the nearest 0.1 mg/l.
> 1	$1 < \text{loss} < 2$	Calculate BOD ₅ using the dilution which gives loss closest to 2 mg/l.
< 1		Use lowest ml sample, calculate BOD ₅ and report as $>$ the calculated value

<u>F.D.O.</u>	<u>D.O. loss</u>	<u>Report</u>
	< 1	Calculate BOD ₅ based on 0.1 mg/l D.O. loss; report as < calculated value.

Average when all dilutions have a F.D.O. ≥ 1 mg/l and a D.O. loss ≥ 2 mg/l

Winkler titration: Fill two B.O.D. bottles with distilled water. Place one bottle on the dissolved oxygen probe and let the meter equilibrate. To the other bottle add 2 ml of alkaline-iodide azide solution and 2 ml of manganous sulfate. A brown precipitate will form. Shake the bottle to suspend the precipitate. Allow the precipitate to settle until at least 3/4 of the bottle is clear. Shake the bottle again to suspend the precipitate and allow the suspension to settle out. Repeat this procedure 2 more times. Then add 2 ml of H₂SO₄ and shake again. The resulting solution should be clear and yellow. By means of a 201 ml volumetric flask transfer 201 ml of this solution to a 500 ml Erlenmeyer flask. Titrate with standard Na₂S₂O₃ to a straw yellow color. At this point add starch indicator and continue titrating until the blue color is discharged. The volume of Na₂S₂O₃ required to reach the end point should be equivalent to the dissolved oxygen reading obtained from the bottle filled with distilled water and placed on the meter. If not, adjust the meter such that the D.O. reading corresponds to the volume (in ml) required for the Winkler titration.

Seed Standardization: A source for seeding B.O.D must be standardized prior to use. When a seed is secured, the sample is allowed to settle and syphoned into plastic bottles labelled exclusively for use with B.O.D. seed. To standardize the seed a series of dilutions are prepared according to the chart below. The samples are treated as any other B.O.D. sample. That is, the sample is diluted to 300 ml; the initial D.O. determined, incubated 5 days and the final D.O. determined.

Sample volume (ml)	# of replicates
15	1
10	2
5	2
2	1
1	1
0.5	1

After the 5 day incubation period calculate the loss per ml as follows

$$\text{D.O. loss/ml} = \text{D.O. loss/sample volume}$$

The ideal value for seed correction is within the 0.6-1.0 mg/l range. Under ideal circumstances, the seed volume for the daily "seed check" is determined from the standardization yielding a D.O. depletion of 50% of the initial D.O. The seed volume for sample bottle is that volume which will theoretically produce a D.O. loss of approximately 0.8 mg/l. Often times this requires a bit of experimentation to meet these criteria.

Quality Control: A minimum of two dilutions of each sample are run for each sample. When the sample is unfamiliar three dilutions are prepared. Of the two dilutions prepared, the dilution yielding an oxygen depletion of 2 mg/L with a residual oxygen of at least 1 mg/L is reported. A minimum of one duplicate must be run with each series of samples. If more than one page of samples are set-up a duplicate per page is to be run. The seed volume is sufficient to yield a 0.6-1.0 mg/L oxygen depletion in the seed check bottles.

A standard glucose-glutamic acid solution with B.O.D. = 200 mg/L is run with each series of samples. The B.O.D. found from this solution is recorded.

An independently acquired quality control standard is run monthly. Acceptable results are within 10% of the vendor's stipulated value. All B.O.D. bottles are washed with a soap solution, triple rinsed with water, rinsed with distilled water and allow to air dry before using again.

HYDROLOGIC, INC.

Analytical Procedure

Title: Chloride Determination - Titrametric

Date issued: 9 Sept 1994

Supersedes: 28 Jan. 1993

Issued by:

Safety:

Safety glasses
Lab coat

Preservation:

Sample are to be chilled to 4°C at the time of collection. The samples must be analyzed within 28 days of collection

Reporting limits:

0.50 mg/L Cl⁻

Equipment:

Buret 50 ml
Beakers (various sizes)
Erlenmeyer flask 150 ml
Stir bars
Stir plate
Volumetric flask (various sizes)

Reagents:

Nitric acid (3+997) Preparation: To a 1000 ml volumetric flask pipet 3.0 ml of concentrated nitric acid (HNO₃). Dilute to volume with deionized water. Shake to mix.

Mercuric nitrate 0.0141 N Purchased from a reputable chemical supplier. Do not use if the expiration date has been exceeded.

Mixed indicator (Diphenylcarbazone-borophenol blue)
Purchased from a reputable chemical supplier. Do not use if the expiration date has been exceeded.

Acidifier Reagent: Purchased from a reputable chemical supplier. Do not use if the expiration date has been exceeded.

10% NaOH Preparation: Weigh to the nearest 0.1 g, 10 g of NaOH pellets. Add 90 ml of deionized water. By means of a stir rod agitate to dissolve. This heat of solution is slightly exothermic, use caution to avoid burns.

0.025 N Sodium chloride (NaCl) Preparation: Weigh to the nearest 0.2 mg, 1.4613 g of NaCl which has been previously dried for 3 hours at 110°C. Quantitatively transfer to a 1L volumetric flask. Add enough water to dissolve. Dilute to volume with deionized water. Shake to mix.

Performance standard of known concentration.

Procedure:Standardization:

Transfer 10.0 ml of 0.025N NaCl to a 150 ml Erlenmeyer flask. Record the volume on the benchsheet. Add water to bring total volume in the flask to 50 ml. Add a stir bar. Add 10 drops of mixed indicator and 3 drops of acidification reagent. With agitation add 10% NaOH until the solution is blue violet (a red violet color indicates a slight excess of NaOH and has no effect on the analysis.) Add 3+997 HNO₃ dropwise until the solution is yellow. Add a 1 ml excess of 3+997 HNO₃. Titrate with 0.0141 N Hg(NO₃)₂ to the blue-violet endpoint. Record the volume of Hg(NO₃)₂ required on the benchsheet. Calculate the normality of the titrant (see calculations section) and record the normality on the benchsheet.

Sample determination:

Transfer 100 ml of sample or an aliquote of sample diluted to 100 ml to a 150 ml Erlenmeyer flask. Record the sample volume on the benchsheet.

Add 10 drops of mixed indicator and 3 drops of acidifier reagent. If a blue-violet color develops add 3+997 HNO₃ dropwise with agitation until the solution is yellow. If the color of the solution is initially yellow proceed to the next step.

Then add 10% NaOH dropwise until the solution become blue-violet (red violet color indicates a slight excess of NaOH; this has no effect on the analysis.) Back titrate to a yellow color using 3+997 HNO₃. Add a 1 ml excess of HNO₃.

Titrate with standard Hg(NO₃)₂ to the blue-violet endpoint. Record the titre required on the benchsheet.

Calculate the chloride concentration in the sample and record on the benchsheet. The chloride is in mg/L Cl⁻

A blank should be run and subtracted from the ml Hg(NO₃)₂ required to titrate both the samples and the standardization.

Record the blank value (ml Hg(NO₃)₂) on the benchsheet.

Calculation:Standardization:

$$N \text{ Hg}(\text{NO}_3)_2 = \frac{\text{ml } 0.025 \text{ N NaCl} \times 0.025}{(\text{ml Hg}(\text{NO}_3)_2 - \text{ml blank})}$$

Sample determination:

$$\text{mg/L Cl}^- = \frac{(\text{ml of Hg}(\text{NO}_3)_2 - \text{blank}) \times N \text{ Hg}(\text{NO}_3)_2 \times 35450}{\text{Sample volume (ml)}}$$

Relative percent difference:

$$\text{RPD} = \frac{|\text{Repl. 1} - \text{Repl. 2}|}{((\text{Repl. 1} + \text{Repl. 2})/2)} \times 100$$

Spike Recovery:

$$\% \text{ Spike Recovery} = \frac{\text{mg Cl}^- \text{ spiked sample} - \text{mg Cl}^- \text{ unspiked sample}}{\text{mg Cl}^- \text{ spiked}} \times 100$$

Standard Recovery

$$\% \text{ Recovery} = \frac{\text{Found value}}{\text{True value}} \times 100$$

Quality Control:

Proper sample preservation is verified at log in.

The mercuric nitrate is standardized with each analytical session. A blank is determined with each analytical session and subtracted from the standardization and sample calculation.

Duplicate analysis are performed at a minimum rate of 10%. The replicate determinations must agree within the control limits established by the Precision Control Chart. Failure to meet these acceptance criteria requires the samples to be rerun. If a second failure occurs notification of the supervisor for assistance in locating and correction of the problem is required.

A performance standard of known concentration is analyzed with each analytical session. The value found should be within the control limits established by the Reference Standard Control Chart. If not, contact the supervisor for assistance in identification and alleviation of the problem.

Samples must be spike at a minimum rate of 10%. Spike recovery should be within the control limits established by the Accuracy Control Chart. Failure to meet these acceptance criteria necessitates rerunning the samples. A second failure requires that the supervisor be notified to assist in problem identification and correction.

Solution preparation and standardizations are documented in the benchsheet notebook.

References:

USEPA, *Methods for Chemical Analysis of Water and Wastes*, "Method 325.3 Chloride Titrimetric, Mercuric Nitrate," 1983.

USEPA, *Test Method for Evaluating Solid Waste, Physical/Chemical Methods*, SW 846 3rd Ed., "Method 9252 Chloride Titrimetric, Mercuric Nitrate," 1986.

Clesceri, Lenore S., et. al., Eds., *Standard Methods for the Examination of Water and Wastewater 17th Ed.*, "4500-Cl- C. Mercuric Nitrate Method," p 4-69 ff, 1989.

HYDROLOGIC, INC.

Analytical Procedure

Title: Determination of Cyanide in Solids and Oils	
Date Issued: 12 Sep 94	Supersedes:
Issued by:	

Safety: Lab Coat
Safety glasses
Latex gloves
Other measures as determined by the nature of the sample

Sample Preservation: Sample should be chilled to 4°C at the time of collection. Refer to "Cyanide Determination" procedure for additional guidance.

Report limits: As described in "Cyanide Determination" procedure.

Equipment: TCLP tumbler with extraction vessels (HPDE)
Buchner funnel
Glass wool
Water driven aspirator
Separatory funnel (500 ml)

Reagents: 50% NaOH May be purchased from a reputable supplier. *Alternatively, prepare by mixing equal weight of NaOH pellets and water. Exercise extreme care, the heat of solution is markedly exothermic and severe chemical and/or thermal burns may result. Store in a sealed plastic container labeled as to contents, date of preparation, preparer, and corrosive hazardous nature of contents.*
n Hexane (C₆H₁₄): May be purchased from a reputable supplier.

Procedure: In the presence of a free aqueous phase prepared the Buchner funnel for filtering the sample. First, prepare a 1 cm pad of glass wool cut to fit the funnel. Weigh the pad and record the weight on benchsheet. Place the glass wool filter into the Buchner funnel and wet, under aspiration, the pad with a known amount of water. Turn off the aspiration source and let all the vacuum to dissipate. Decant the liquid portion of the sample in small aliquotes into the Buchner funnel. Then transfer the solid/oily phase to the funnel and rinse the container with known aliquotes of water. When no more

liquid remains in the container, begin aspiration. A small amount of sediment passing into the filtration flask will not affect the analysis. Measure the volume of liquid collected during filtration. Determine the aqueous volume originally present in the sample by subtracting the wash volumes from the total volume obtained.

Should the washings contain an oil layer, this should first be separated from the washings using a separatory funnel. Do not include the volume of the oil in the foregoing calculation.

Transfer the solid/oily phase contained on the glass wool filter pad to a tared weighing dish. Add any oil fraction that may have been contained in the aqueous washings. Calculate the weight of the solid/oily phase by subtracting the tare of the weighing dish and filter pad from the total.

Place 25g of solid/oily phase in the extraction vessel. If the weight of the solid is >25g use a representative aliquote of sample equal to 25g. If the weight of the solid/oily phase is < 25g use all the solids and note the weight on the benchsheet.

In the event that upon mixing a sample suspected of requiring this extraction exhibits uniform suspension of solids, the sample may be analyzed directly by the steps outlined in the "Cyanide Determination" procedure.

Should the sample contain no aqueous or oily phase the procedure commences at this point.

Add to the extraction bottle the weighed solid/oily sample, 500 ml of water, and 5 ml of 50% NaOH. If a heavy grease is present add 50 ml of n hexane.

Shake the extraction bottle for 1 minute. If the pH is < 10 add an additional 5 ml of 50% NaOH. Repeat this procedure until the pH remains 10.

Tumble the mixture for 16 hours.

After 16 hours tumbling, prepare a Buchner funnel as previously described and filter the extract. Entrainment of some sediment will not effect the analysis. It is not necessary to filter all the extract, only a sufficient amount to recombine with the aqueous fraction is necessary. However, the recombination must represent the make-up of the original sample. If the sample contained no aqueous phases filter a sufficient volume to proceed with the analysis as given in the "Cyanide Determination" procedure. Should an oily layer be present after extraction, separate it by means of a separatory funnel. Again, exacting volumes are not necessary. Only sufficient volume to recombine in a representative fashion is required.

Proceed with the analysis as given in the "Cyanide Determination" procedure.

Quality Control:

Those quality control parameters defined in the "Cyanide Determination" procedure apply.

References:

USEPA, *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW 846 3rd Ed.*, "Method 9013 Cyanide Extraction Procedure for Solids and Oils," 1986.

HYDROLOGIC, INC.

Analytical Procedure

Title: Cyanide Determination

Date issued: 11 Sep 1994

Supersedes: 15 Jan 1993

Issued by:

Safety:

Safety glasses

Lab coat

Sample distillation and color development should be carried out under a hood.

Sample Preservation:

In the presence of oxidizing agents such as residual chlorine cyanide is destroyed. To test for the presence of oxidizing agents place a drop of sample on acidified potassium iodide-starch test paper. A blue color is a positive test. Thus, add ascorbic acid a few crystals at a time until no color is produced when a drop of sample is put on the KI-starch test paper. Add an additional 0.6 g of ascorbic acid per liter of sample.

Samples must be preserved to pH > 12 by the addition of 10N NaOH at the time of collection.

Sample should be chilled to 4°C at the time of collection

Sample must be analyzed within 14 days of collection.

Reporting limits:

0.005 mg/L CN⁻

Equipment:

1000 ml Round bottom flask two neck

Condenser (Cold finger)

Absorption tube with frit

Inlet tube

Vacuum source

Water source

Spectrophotometer

250 ml Volumetric flask

100 ml Volumetric flask

Graduated cylinders (various sizes)

Pipets (various sizes)

Buret 50 ml

Stir plate and coated stir bars

Reagents:

1.25 N NaOH Preparation: Weigh to the nearest 0.1g, 50g of NaOH pellets. Transfer to a 1L volumetric flask and slowly dilute to volume. Heat will be generated in this process, use caution to avoid burns. Also allow solution to cool to room temperature before completing the dilution process.

0.025N NaOH: Prepare by weighing to the nearest 0.1 g, 10g of NaOH pellets. Transfer to a 1000 ml volumetric and dilute to volume. Heat will be generated in this process, use caution to avoid burns. Also allow solution to cool to room temperature before completing the dilution process.

Magnesium chloride solution Purchased from a reputable supplier. Do not use if expiration date has been exceeded.

Sulfamic acid Purchased from a reputable supplier. Do not use if expiration date has been exceeded.

Cyanide Standard (1000 mg/L CN⁻) Purchased from a reputable supplier. Do not use if expiration date has been exceeded. This solution should be standardized on a regular basis. *Standardize by* pipetting 25 ml of standard CN⁻ solution into a 125 ml Erlenmeyer flask. Add 50 ml of water. Add 12 drop of Rhodamine indicator. With agitation titrate with 0.0192 N AgNO₃. The end point is the color change from canary yellow to salmon (reddish brown). Titrate a blank. Record data on the benchsheet. Calculate the CN⁻ concentration (See Calculation section) This titration should be performed on a weekly basis to insure accuracy of the standards.

Pyridine Purchased from a reputable supplier. Do not use if expiration date has been exceeded.

Barbituric Acid Purchased from a reputable supplier. Do not use if expiration date has been exceeded

Sodium dihydrogen phosphate (NaH₂PO₄·H₂O) Purchased from a reputable supplier.

Do not use if expiration date has been exceeded

Chloramine T Purchased from a reputable supplier.

Do not use if expiration date has been exceeded

Concentrated Hydrochloric acid (HCl) Purchased from a reputable supplier.

Concentrated Sulfuric acid (H₂SO₄) Purchased from a reputable supplier.

Cyanide standards Preparation: Prepare a series of standards containing approximately 0.5ug, 1.0ug, 2.0 ug, 5.0ug, 10.0ug cyanide by serial dilution of the 1000 ppm cyanide stock solution. These concentrations should be adjusted to accommodate the

cyanide concentration found by the standardization procedure given above. (See calculation Section)

Pyridine-Barbituric Acid Reagent (Color developing reagent)

Preparation: Weigh to the nearest 0.1g, 30g of barbituric acid into a 500 ml volumetric flask. Add just enough water to wash down the sides and wet the barbituric acid. Add 150 ml of pyridine and swirl to mix the flask contents. Slowly add 30 ml of concentrated hydrochloric acid, swirl to mix flask contents and let cool to room temperature. Gradually, add water to dilute to volume. Swirl between each water addition to insure mixing. Note preparation of this reagent should be done under a fume hood. When HCl is added to the pyridine copious noxious vapors will be released. This solution should be stored in the dark at approximately 4°C.

Chloramine T solution Preparation: To a 100 ml volumetric flask add 1.0g of Chloramine T and dilute to volume with water. This solution must be prepared fresh for each analytical session.

Sodium dihydrogen phosphate Preparation: In a 1000 ml volumetric flask weigh 138g of sodium dihydrogen phosphate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$). Dilute to volume with water and store at 4°C.

Procedure:

Transfer 500 ml of sample, or an aliquote of sample diluted to 500 ml, to a 1000 ml two neck round bottom flask. Record the sample volume on the benchsheet. Add approximately 10 boiling stones and 2g of sulfamic acid.

To the absorber tube add 50 ml of 1.25 N NaOH.

Assemble apparatus and attach to vacuum source. Turn on vacuum such that the air intake through the inlet tube produces one to two bubbles per second. Turn on water to condenser.

Through the inlet tube add 25 ml of concentrated sulfuric acid in two 10 ml aliquotes and one 5 ml aliquote. Allow time in between each addition to insure complete mixing. Exercise caution to avoid regurgitation of the sulfuric acid out through the inlet tube.

Rinse the inlet tube. After a minimum of three minutes mixing add 20 ml of MgCl_2 solution. Again, exercise care to avoid regurgitation of the flask contents through the inlet tube. Allow flask contents to mix a minimum of three minutes.

Apply heat via the heating mantle and reflux for one hour. Once the solution begins to boil the rheostat setting may be lowered to maintain boiling.

After one hour discontinue heating and allow the solution to cool for a minimum of 15 minutes.

When cooled disassemble the apparatus and quantitatively transfer the contents of the absorber tube to a 250 ml volumetric flask. Dilute to volume with water and mix.

Standard Curve:

Prepare a standard curve by performing the previously described serial dilution of the cyanide standard. The cyanide standards should be prepared in a 100 ml volumetric flask. To each standard add enough 0.025 N NaOH to bring the volume to approximately 50 ml. Add 15 ml of NaH_2PO_4 solution to each flask and mix, then add 2 ml of Chloramine T solution and mix, wait 2 min and 5 ml of pyridine-barbituric acid solution. Dilute to volume with distilled water and mix. Allow color to develop for 8 minutes. Determine absorbance of each solution at 578 nm. The absorbance value must be determined within 15 minutes. Develop a standard curve plotting CN^- concentration in μg against absorbance

Place curve in the benchsheet notebook

Sample Determination:

Pipet 50 ml of sample from the 250 ml volumetric flask to a 100 ml volumetric flask. To each sample add 15 ml of NaH_2PO_4 solution, 2 ml of Chloramine T solution, and 5 ml of pyridine-barbituric acid solution. Dilute to volume and mix. After 8 minutes determine the absorbance of each solution at 578 nm. Record the absorbance on the benchsheet. The absorbance must be determined within 15 minutes.

Determine the CN^- content of each sample in μg by comparing the absorbance of the samples to the standard curve. Record the value on the benchsheet.

Should the absorbance of the sample exceed the absorbance value of the highest standard and appropriately smaller aliquote should be removed from the 250 ml volumetric flask. Dilute the smaller aliquote to 50 ml using 0.25 N NaOH (10g of NaOH to 1L). Then repeat the color developing procedure.

Calculation:

Determination of CN^- :

$$\frac{\text{ug of } \text{CN}^- \text{ found} \times 250}{\text{Aliquote used} \times 500} = \text{ug/ml } \text{CN}^-$$

CN⁻ Standardization:

$$\frac{(\text{ml titer} - \text{blank})}{25.0} = \text{ug/ml CN}^{-}$$

CN⁻ calibration standards Adjustment:

$$\frac{(\text{ug/ml CN}^{-} \text{ from standardization})(\text{aliquote ml})}{\text{final volume (ml)}} = \text{ug/ml CN}^{-}$$

Standard recovery:

$$100 \times \frac{\text{Found value}}{\text{True value}} = \% \text{ Recovery}$$

Relative percent difference:

$$100 \times \frac{|(\text{Repl. 1} - \text{Repl. 2})|}{((\text{Repl. 1} + \text{Repl. 2})/2)} = \text{RPD}$$

Spike recovery:

$$\frac{\text{ug CN}^{-}(\text{spiked sample}) - \text{ug CN}^{-}(\text{unspiked sample})}{\text{ug CN}^{-} \text{ in spike}} \times 100 = \% \text{ Recovery}$$

Quality Control:

Proper sample preservation is verified at log in. Duplicates are run at a rate of not less than 10%. The duplicate must agree within the control limits established by the Precision Control Chart. Failure to meet this criteria requires that the sample be rerun. A second failure requires notification of the supervisor for assistance in locating and correcting the problem. For each analytical session the absorbance of the 0.5 ug (LDL-lower detection limit), 2.0 ug, (LCS-linearity check standard) and 10.0 ug CN⁻ (Hi standard) standards are determined and compared against the standard curve. Recovery of each standard must be within the control limits established for each respective standard.

Failure to recovery within these acceptance limits indicates that a new curve should be prepared.

An independent standard of known CN is run with each analytical session. The recovery of this standard compared against its known value must be within the control limits establish by the Reference Standard Control Chart. Failure to meet the acceptance criteria requires that the standard be rerun prior to reporting any data. A second failure requires notification of the supervisor for assistance in locating and correcting the problem.

Standard curves are prepared by each analyst at least once per quarter. Should the values of a new standard curve exceed those expected when compared against the previous curve the supervisor should be consulted prior to using the curve. The standard curve is archived in the benchsheet notebook. Each standard curve should conspicuously show the analyst name, date of preparation, instrument used, and wavelength.

Sample date and distillation date are documented on the benchsheet to validate analysis within holding time.

Document solution and standards preparation on the appropriate forms in the benchsheet notebook.

References:

Clesceri, Lenore S., *et. al.*, Eds, *Standard Methods for the Examination of Water and Wastewater, 17th Ed.*, "4500-CN E. Colorimetric Method," p 4-31 ff., 1989.

USEPA, *Methods for Chemical Analysis of Water and Wastes*, "Method 335.2, Cyanide, Total, Titrametric, Spectrophotometric," 1983.

USEPA, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW 846, 3rd Ed.*, " Method 9010 Total and Amenable Cyanide (Colorimetric, Manual)," 1986.

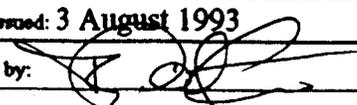
HYDROLOGIC, INC.

Analytical Procedure

Title: **Digestion-Total Metals in aqueous matrices**

Date issued: **3 August 1993**

Supersedes: **13 Jan 1993**

Issued by: 

Safety: Lab Coat
Safety Glasses
Gloves
All evaporation and refluxing process must be carried out in a fume hood

Equipment: Volumetric pipet (various sizes)
Beakers 250 ml
Volumetric flasks 100 ml
Funnels 60°
Filter paper #41 Whatman (or equivalent)
Stir rods
Watch glasses
Hot plates

Reagents: Nanopure water or equivalent
Concentrated Nitric Acid (HNO₃)
Spiking solution

Procedure: This procedure is applicable for all metals digestions for total metal determination except arsenic (As), selenium (Se), and mercury (Hg). By means of a 100 ml volumetric pipet transfer 100.0 ml of sample to a 250 ml beaker.
Add 3.0 ml of concentrated HNO₃. Place a stir rod in the beaker and mix the contents.
Under a hood, heat the samples to evaporate volume to approximately 25 ml. The temperature must not exceed 95°C. Using the stir rod, stir the solution occasionally.
When the sample volume has been reduced remove from the hot plate and allow to cool to room temperature.
Add 2 ml of deionized water and an additional 3.0 ml of concentrated HNO₃. Return the beaker to a hot plate and cover with a watch glass. Reflux the sample for 1 hour with gentle heat. Do not let the temperature exceed 95°C.
At the end of 1 hour remove the sample from the hot plate and cool. When cool add an additional 3.0 ml of HNO₃ and reflux another hour.

Continue this process until the digestion is complete as evidenced by the absence of red NO_x fumes.

When the digestion is complete remove the sample from the hot plate and let cool to room temperature.

Quantitatively filter the sample through Whatman #41 filter paper into a 100 ml volumetric flask. Dilute to volume with deionized water.

Quality control:

Samples should be preserved to $\text{pH} < 2$ with HNO_3 and chilled to 4°C at the time of collection. Sample should be analyzed within 6 months of collection.

Duplicate analysis are performed at a rate of not less than 10%.

Spike samples are run at a rate of not less than 10%. Spike are prepared by taking a second 100 ml aliquote of the selected sample and adding an aliquote of spike solution containing the analyte(s) of interest. The sample is carried through the digestion procedure.

Weekly digestion blanks are run to verify cleanliness of glassware and purity of reagents. A blank is a 100 ml aliquote of nanopure water carried through the digestion procedure.

HYDROLOGIC, INC.

Analytical Procedure

Title: Digestion-Total Metals in aqueous matrices for ICP Determination	
Date issued: 3 August 1993	Supersedes:
Issued by: 	

Safety: Lab Coat
Safety Glasses
Gloves
All evaporation and refluxing process must be carried out in a fume hood

Equipment: Volumetric pipet (various sizes)
Beakers 250 ml Griffin
Volumetric flasks 100 ml
Funnels 60°
Filter paper #40 Whatman (or equivalent)
Stir rods
Watch glasses
Hot plates

Reagents: Nanopure water or equivalent
Concentrated Nitric Acid (HNO₃)
Concentrated Hydrochloric Acid (HCl)
Spiking solution

Procedure: This procedure is applicable for all aqueous metals digestions for total metals determination except mercury (Hg).
By means of a 100 ml volumetric pipet transfer 100.0 ml of sample to a 250 ml beaker.
Add 3.0 ml of concentrated HNO₃. Place a stir rod in the beaker and mix the contents.
Under a hood, heat the samples reducing the sample to a small volume. Do not allow the beaker to go dry. The temperature must not exceed 95°C. Do not allow the contents to boil. Using the stir rod, stir the solution occasionally.
When the sample volume has been reduced remove from the hot plate and allow to cool to room temperature.
Add additional 3.0 ml of concentrated HNO₃. Return the beaker to a hot plate and cover with a watch glass. Reflux the sample for 1 hour with gentle heat. Do not let the temperature exceed 95°C.

At the end of 1 hour remove the sample from the hot plate and cool. When cool add an additional 3.0 ml of HNO_3 and reflux another hour. Continue this process until the digestion is complete as evidenced by the absence of red NO_x fumes or that the solution does not change color with subsequent additions of HNO_3 .

Again evaporate to a low volume avoiding boiling or allowing the beaker to go dry.

Cool the beaker and contents.

Add 10 ml of 1+1 HCL. Recover the beaker with a watch glass and reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from the evaporation steps.

Cool the beaker and contents. In the presence of undissolved silicates or solids which might clog the nebulizer quantitatively filter the sample through Whatman #40 filter paper into a 100 ml volumetric flask. Dilute to volume with deionized water. The final acid concentration is 10%.

Quality control:

Samples should be preserved to $\text{pH} < 2$ with HNO_3 and chilled to 4°C at the time of collection. Sample should be analyzed within 6 months of collection.

Duplicate analysis are performed at a rate of not less than 10%.

Spike samples are run at a rate of not less than 10%. Spike are prepared by taking a second 100 ml aliquote of the selected sample and adding an aliquote of spike solution containing the analyte(s) of interest. The sample is carried through the digestion procedure.

Digestion blanks are run with each digestion batch to verify cleanliness of glassware and purity of reagents. A blank is a 100 ml aliquote of nanopure water carried through the digestion procedure.

HYDROLOGIC, INC.

Analytical Procedure

Title: Total Metals Digestion for Sludges, Sediments, and Soils	
Date issued: 4 July 1994	Supersedes: 5 August 1993
Issued by:	

Safety: Lab Coat
Safety glasses
Gloves
All digestion steps must be carried out under a fume hood

Equipment: Analytical balance capable of weighing to 0.01g
Beakers 250 ml
Thermometer -15°C to 200°C
Watch glass
Glass stir rods
Filter paper Whatman #40
Volumetric flask 100 ml
Funnel 60°
Hot plates
Volumetric pipets various sizes
Drying oven capable of maintaining 104° ± 2°C

Reagents: Concentrated Nitric acid (HNO₃)
Concentrated Hydrochloric acid (HCl)
Hydrogen peroxide 30%
Nanopure water
1+1 Nitric acid Prepare by: Mixing equal portions of concentrated HNO₃ and water. Care should be exercised to add the HNO₃ to the water. The heat of solution is extremely exothermic, due care should be exerted to avoid burns.

Procedure: Sample Digestion:
Thoroughly mix the sample to insure homogeneity.
Into a 250 ml beaker weigh, to the nearest 0.01g, 1.0 to 2.0g of sample. In the event that the sample is low in solids, larger sample sizes may be used. When in doubt contact the supervisor. Record the sample weight on the appropriate benchsheet.
To each sample add 10 ml of 1+1 HNO₃. By means of a glass stir rod, mix the sample and acid solution. Use care, often heat is generated and basic soils will tend to effervesce.

Cover with a watch glass, place on the hot plate, heat the mixture to 95°C and reflux for 10 to 15 minutes.

At the end of 15 minutes let the mixture cool.

Add 5 ml of concentrated HNO₃, recover, and reflux for an additional 30 minutes.

Repeat this step with an additional 5 ml of HNO₃ added after the mixture is cooled.

At the end of the second reflux period, reduce the volume in the beaker to 5 ml. Do not allow any portion of the bottom of the beaker to go dry. Also do not allow the solution to boil. Should either of these events occur, discard the sample and begin again. When the solution has reached approximately 5 ml remove from the hot plate and allow it to cool.

Add 2 ml of nanopure water and 3 ml of 30% H₂O₂.

Recover the beaker and return it to the hot plate. Cautiously warm the beaker and its contents. Exercise care as the effervescence associated with the peroxide reaction proceeds to avoid sample loss or injury. Continue warming until effervescence subsides.

Cool the beaker. Add an additional 1 ml of 30% H₂O₂ and repeat warming. Continue adding 1 ml aliquotes of 30% H₂O₂ to cooled samples followed by rewarming until the effervescence is minimal or the sample generally does not change in appearance. In no case add more than 10 ml total of 30% H₂O₂ to the sample mixture.

If the metals are to be determined by FLAA or ICP, add 5 ml of concentrated HCl and 10 ml of nanopure water.

Cover the beaker, return it to the hot plate, and reflux for 15 minutes.

At the end of the final reflux, cool the beaker, and quantitatively transfer the sample to a 100 ml volumetric flask. At the end of the digestion process, should the sample contain undissolved solids which will clog the nebulizer, the quantitative transfer should be accomplished by filtering the sample quantitatively through Whatman #40 filter paper. Dilute the sample to volume with nanopure water. Record the final volume on the benchsheet.

If the metals are to be determined by GFAA continue to heat the solution resulting from the addition of H₂O₂ reducing the volume to 5 ml.

Remove the solution from the hot plate and allow it to cool.

Quantitatively transfer the sample solution to a 100 ml volumetric flask and dilute to volume with nanopure water. Should the digestate contain solids which will interfere with proper instrument operation, the quantitative transfer should be accomplished by

Cover with a watch glass, place on the hot plate, heat the mixture to 95°C and reflux for 10 to 15 minutes.

Percent Solids Determination:

After beginning the digestion process, weigh to the nearest 0.1 g a minimum 5 g aliquote. Record as initial weight on page 2 of benchsheet.

Place in the drying oven at 104°C for 3 hours.

At the end of the 3 hours reweigh the sample and record as final weight on page 2 of the benchsheet.

Calculate % solids as given in the calculation section.

Calculation:

$$\% \text{ Solids} = \frac{\text{Final weight (b)}}{\text{Initial weight (a)}} \times 100$$

Quality Control:

Samples should be collected in approved properly cleaned containers and chilled as practicable.

Samples should be analyzed as soon after collection as possible.

For each digestion batch samples should be run in duplicate at a rate of not less than 10%. Percent solids should also be determined in duplicate at a rate of not less than 10%.

For each digestion batch randomly chosen samples should be spiked by adding a known volume of spike solution to a replicate sample and carrying the sample plus the spike through the digestion process. Spikes should be performed at a rate of not less than 10%.

Reference samples, as available, should be carried through the digestion process.

All weights and volumes should be recorded on the appropriate benchsheet.

References:

"Test Methods for Evaluating Physical and Chemical Methods, SW-846," 3rd Ed.

Memorandum from North Carolina Department of Environment, Health, and Natural Resources, June 20, 1994.

HYDROLOGIC, INC.

Analytical Procedure

Title: Metals Digestion-Total Recoverable/Dissolved with ICP Determination

Date Issued: 4 August 1993

Supersedes:

Issued by: *R. J. [Signature]*

Safety:

Lab coats
Safety glasses
Gloves
All digestion steps should be carried out under a fume hood

Equipment:

Volumetric pipets (Various sizes)
Beakers 250 ml Griffin
Volumetric flasks 100 ml
Funnels 60°
Glass stir rods
Whatman #40 filter paper or equivalent
Hot Plates

Reagents:

Nanopure water or equivalent
Concentrated nitric acid (HNO₃)
Concentrated hydrochloric acid (HCl)
Spiking solution

Procedure:

Total recoverable metals should be acidified at the time of collection by adding HNO₃ at the rate of 5 ml/L.
Dissolved metals should, at the time of collection, first be filter through a 0.45 μ m filter and then acidified with HNO₃ at the rate of 5 ml/L.
Transfer a well mixed 100 ml aliquote of sample into a 250 ml beaker.
Add 2 ml of HNO₃ and 5 ml of HCl. Mix with glass stir rod.
Place samples on a hot plate and reduce the volume to 15-20 ml. The solution temperature should remain between 90°C and 95°C. In no case should the solution be allowed to boil. Should the solution boil, discard the sample and begin again.
After the volume has been reduced, let the beaker and solution cool to room temperature. Wash the wall of the beaker into the sample solution. If particulates which might clog the nebulizer are present quantitatively filter into a 100 ml volumetric flask through Whatman #40 filter paper. Dilute to volume with nanopure water.

Should filtration not be necessary, quantitatively transfer the sample to a 100 ml volumetric flask and dilute to volume with nanopure water.

The sample is now ready for analysis by ICP.

Quality Control:

Total recoverable metal should be preserved by adding HNO₃ at the time of collection at a rate of 5 ml/L and chilled to 4°C. Dissolved metals should be, at the time of collection, filtered through a 0.45 µm filter, then preserved with HNO₃ at the rate of 5 ml/L and chilled to 4°C.

Both total recoverable metals and dissolved metals should be analyzed within 6 months of collection with the exception of mercury. Mercury must be analyzed within 28 days of collection.

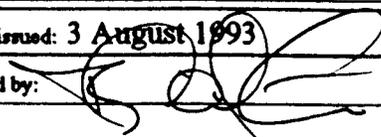
Duplicate samples are run at a rate of not less than 10%

Sample spikes are prepared by taking a second 100 ml of a randomly selected sample and adding a known amount of spike solution. Samples should be spiked at a rate of not less than 10%.

A reagent blank is prepared with each digestion blank by using a 100 ml aliquote of nanopure water and carry it through the digestion procedure.

HYDROLOGIC, INC.

Analytical Procedure

Title: Digestion-Total Arsenic and Selenium in aqueous matrices	
Date issued: 3 August 1993	Supersedes:
Issued by: 	

Safety: Lab coat
Safety glasses
Gloves
All evaporation and refluxing must be carried out in a fume hood

Equipment: Volumetric pipet (Various sizes)
Beakers 250 ml Griffin
Funnels 60°
Filter paper Whatman #41 of equivalent
Glass stir rods
Watch glasses
Hot plates

Reagents: Nanopure water or equivalent
Concentrated Nitric Acid (HNO₃)
Hydrogen peroxide H₂O₂ (30%)
Spiking solution

Procedure: This procedure is applicable determination of aqueous total arsenic (As) and total selenium (Se) by graphite furnace AA.
By means of a 100 ml volumetric pipet transfer 100 ml of well mixed sample to a 250 ml beaker.
Add 2 ml of 30% H₂O₂ to the sample. By means of a glass stir rod mix the H₂O₂ and the sample.
Add 1 ml of concentrated nitric acid.
Gently heat the solution to no greater than 95°C. If the temperature at any time exceeds 95°C or the samples goes to dryness discard the sample and begin the analysis again.
Continue heating until the digestion is complete or the volume is reduced to slightly less than 50 ml.
Discontinue heating and allow the solution to cool.
In the presence of undissolved solids, filter the sample quantitatively through #41 Whatman filter paper into a 100 ml volumetric flask.
Dilute to volume with nanopure water and mix well.

Quality Control: Sample should be preserved to pH < 2 with nitric acid and chilled to 4°C the time of collection. The samples should be analyzed within 6 months of collection.

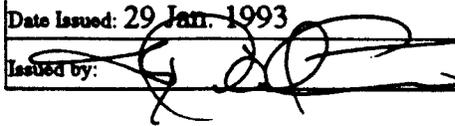
Duplicate samples are performed at a rate of not less than 10%.

Blanks are prepared by using 100 ml of nanopure water carried through the digestion procedure. Blanks should be prepared for each digestion batch.

Spike samples are run at a rate of not less than 10%. Spikes are prepared by taking a second 100 ml aliquote of the selected sample and adding a known aliquote of the analyte of interest. This samples is carried through the entire digestion procedure.

HYDROLOGIC, INC.

Analytical Procedure

Title: Dishwashing	
Date Issued: 29 Jan. 1993	Supersedes:
Issued by: 	

Safety: Lab Coat
Safety Glasses
Gloves

Equipment: Brushes (various sizes)

Reagents: Contrad 70 Detergent CMS Cat. #117-655
1+1 Nitric acid (HNO₃) Preparation: To 250 ml of deionized water slowly add 250 ml of concentrated nitric acid. Use caution, heat will be generated as a result of this addition.
1+1 Hydrochloric acid (HCl) Preparation: To 250 ml of deionized water slowly add 250 ml of concentrated hydrochloric acid. Use caution heat will be generated from this addition.

Procedure: General
Rinse the dirty labware under a stream of running water.
Prepare a soap solution which is very approximately 2% in Contrad 70.
Submerge the dirty labware in the soap solution. For more stubborn residue the labware may require soaking in soap solution. Scrub each piece of labware with the appropriate brush. Thoroughly, clean all surfaces both internal and external. Rinse the labware under a stream of running tap water. Make certain all traces of soap are removed. Typically, this will take four water rinses.
Properly cleaned labware will be free of residue from previous analysis. No water droplets will form on any surface. Return labware to the appropriate storage area.

Metals Glassware
Follow the General cleaning procedure for glassware for metals analysis.
Following the final rinse, further rinse the internal surfaces with 1+1 HCl. Then rinse three times with distilled water. Next rinse the internal glassware surfaces with 1+1 HNO₃. Finally, rinse three times with deionized water.

Total phosphorus/phosphate Glassware

Follow the general cleaning procedure given above.

After the final water rinse, rinse the internal surfaces with 1+1 HNO₃. Follow this with a triple deionized water rinse. Then add 1+1 HCl to each piece and heat to boiling under a fume hood. Let cool to room temperature. Rinse each piece three times with deionized water.

Oil & Grease

Follow the general washing procedure outlined above. This applies to both laboratory ware and sample containers.

Rinse with clean freon. Collect spent freon in another container for disposal.

Allow to air dry.

Fecal/Total Coliform Sample Bottle

Wash according to general procedures given above.

Allow to air dry.

Add appropriate preservatives as dictated by the procedure and autoclave at 121°C and 15 psig for 20 minutes minimum.

Proper sterilization is verified by adding 10 ml of T-soy broth to a total coliform bottle and incubating for 24 hours at 35°C. If the T-soy appears turbid, improper sterilization is indicated.

Pipets

Pipets should be rinsed with deionized water after use.

Place the pipet in the pipet reservoir containing a 2% Contrad 70 solution.

Let the pipets soak for a minimum of eight hours, then rinse for four hours in the pipet washer.

Further cleaning may be required depending on the determination for which the pipet will be used.

Sample Containers

Follow the general washing procedures unless otherwise specified.

Depress the "mode" key repetitively until "CONC" is displayed. Depress the calibrate key and, when prompted enter the number of standards three (3).

When prompted by "Standard 1" pipet 50 ml of the 0.5 mg/L F-standard into a 150 ml beaker. Immerse the electrodes into the solution being agitated without a vortex. Add 5 ml of TISAB. Allow the instrument to stabilize as indicated by an audible "beep" and the prompt "READY CAL." Enter the value of the standard and depress the "Yes" key. Depress the "2nd" key followed by the "mV" key to obtain the millivolts for the standard. Record this value on the benchsheet.

Remove the electrodes from the solution, rinse and blot dry. Avoid rubbing the tips of the electrodes.

At the "Standard 2" prompt repeat the process for the second standard that is 1.0 mg/l F.

Finally, at the "Standard 3" prompt repeat the procedure for the third standard, 2.0 mg/L F.

After entry of the last standard a "percent efficiency" value will be displayed. Record this value on the benchsheet also.

Sample distillation:

Preliminary distillation of the sample removes interferences.

Additionally, samples from each matrix type are analyzed routinely and the results compared to those obtained for undistilled samples to validate results for undistilled samples. (See Quality Control Section)

To the distillation flask, add 400 ml of distilled water. Slowly add 200 ml of concentrated H_2SO_4 . Exercise extreme care in this process, the heat of solution is extremely exothermic. Severe thermal and/or chemical burns can result. All steps in the distillation process must be carried out in a fume hood to minimize the risk of accident.

Ensure that the acid water solution is well mixed and homogeneous. Otherwise, when the distillation starts the flask contents may violently erupt from the flask leading to equipment destruction and possible injury in the form of chemical and/or thermal burns. As with all accidents, should this unfortunate reaction occur notify the supervisor immediately !

Add 25 to 30 glass beads to the distillation flask.

Connect the distillation apparatus making certain that the joints are secure. Begin heating, by means of a heating mantle, the acid/water mixture. Heat to exactly 180°F. To avoid overheating turn the

heating mantle off when the temperature reaches 178°C.

Collect the distillate and discard. This process removes fluoride contamination.

Allow the distillation flask to cool to 80°C. Carefully add a 300 ml sample aliquote to the acid water solution remaining in the flask.

Again, heat to 180°C, turning off the mantle at 178°C to prevent overheating. Collect the distillate for analysis by ISE.

Should chloride be present at levels >5000 mg/L add silver sulfate (Ag_2SO_4) at the rate of 5 mg/mg Cl.

The acid water solution may be repeatedly used until contaminants become entrained in the distillate. This contamination is evidenced when poor recovery of standards is observed. Additionally, if samples containing more than 3 mg/L F are distilled the system is flushed by distilling 300 ml of water. Repeat this process until the fluoride content of the distillate is minimal. Combine successive flushings with the first distillate for analysis.

After periods of inactivity the foregoing flushing procedure is repeated and the distillate discarded.

Sample determination:

Into a 150 ml beaker pipet a 50 ml homogeneous aliquote of sample. Record the volume on the benchsheet. Begin agitation such that no vortex is formed.

Pipet 5 ml of TISAB into the sample and immerse the electrodes.

The instrument will automatically begin the measuring process.

Allow the instrument to come to equilibrium as indicated by an audible beep and a "RDY" prompt. Record the value on the benchsheet. If the F⁻ value exceeds the range of the curve,

>2 mg/L F⁻, an aliquote less than 50 ml is pipetted into a 50 ml volumetric flask and diluted to volume. Record the sample volume on the benchsheet and repeat the sample determination steps. Continue reducing the sample aliquote until the F⁻

concentration falls within the range defined by the standard curve.

Calculate the F⁻ concentration as defined in the Calculation section and record the value reported on the benchsheet.

Calculations:

Concentration (mg/L) F⁻:

$$\text{mg/L F}^- = \frac{\text{mg/L F}^- \text{ observed} \times 50 \text{ ml}}{\text{Sample aliquote}}$$

Relative percent difference (RPD):

$$\text{RPD} = \frac{|\text{Repl. 1} - \text{Repl. 2}|}{((\text{Repl. 1} + \text{Repl. 2})/2)} \times 100$$

Spike Recovery:

$$\% \text{ Recovery} = \frac{\text{mg/L F}^- \text{ spiked} - \text{mg/L F}^- \text{ unspiked}}{\text{Spike conc.}} \times 100$$

Standard recovery:

$$\% \text{ Recovery} = \frac{\text{Found value}}{\text{True value}} \times 100$$

Quality Control:

Proper sample preservation is verified at log in.

Sample duplicates are run at a minimum rate of 10%. The relative percent difference between the replicates are to be within the control limits established by the precision control chart of $\leq 20\%$ RPD which ever is lowest. Failure to meet these acceptance criteria requires that the samples be rerun. On a second failure the supervisor is notified for assistance in identifying the problem and resolving it prior to reporting data.

A minimum of one sample per analytical session is analyzed without distillation and distilled. Agreement between the two runs is as defined in the duplication requirement above.

Samples are spiked at a minimum rate of 10%. Spike recoveries are to be within the control limits defined by the accuracy control chart or $\pm 15\%$ which ever is lowest. Failure to recover within these acceptance criteria necessitates that the samples be rerun. If a second failure occurs notify the supervisor for assistance in locating and correcting the problem before reporting data.

Prior to beginning actual sample determination the standard curve is validated by running a linearity check standard (LCS) and an independent QC standard of known fluoride concentration.

Acceptable recoveries for these standards are within the control

limits established by their respective control charts or $\pm 10\%$, which ever is lowest. Failure to obtain these results requires that the instrument be restandardized and the standards rerun. If a second failure occurs notify the supervisor for assistance in locating the problem and correcting it prior to reporting data.

Instrument stability is demonstrated by running the LCS standard after every tenth sample or fraction thereof. Acceptance criteria are the same described for the LCS standard above. After one failure the LCS standard may be rerun. Failure to obtain acceptable results requires that the instrument be restandardized and the samples run since the last acceptable LCS check be rerun. Report the data obtained after restandardization and successful QC validation.

References:

Clesceri, Lenore S., et.al., Eds., *Standard Methods for the Examination of Water and Wastewater, 17th Ed.*, "4500-F-Fluoride," p 4-84 ff., 1989.

USEPA, *Methods for the Chemical Analysis of Water and Wastes*, "Method 340.2 Fluoride Potentiometric, Ion Selective Electrode," 1983.

USEPA, *Methods for the Chemical Analysis of Water and Wastes*, "Method 340.1 Fluoride, Total Colorimetric , SPANDS with Bellack Distillation," 1983.

HYDROLOGIC, INC.

Analytical Procedure

Title: **Fecal Coliform Determination-Membrane Filter**

Date issued: 13 Jan. 1993

Supersedes

Issued by:

Safety: Lab coat and safety glasses required

Equipment: Magnetic filter and base (Gelman or equivalent)
1000 ml filtering flask
Vacuum or aspirator
Culture dishes with absorbent pads
Presterilized membrane filters
Forceps
Alcohol
Alcohol lamp
Incubator calibrated to maintain $44.5^{\circ} \pm 0.2^{\circ}\text{C}$
Assorted pipets
Presterilized whirlpaks

Reagents: Water (Barnstead Nanopure or equivalent)
M-FC Broth *Preparation:* Weigh into a to the nearest 0.1g, 7.4g of presterilized M-FC broth. Add 2.0 ml of rosalic acid. Add 200 ml of deionized water. Mix thoroughly and heat just to boiling.. Remove from heat source and allow to cool. Determine the pH of the media. The final pH should be 7.4 ± 0.2 standard units. Record value found.
0.2 N NaOH *Preparation:* Weigh to the nearest 0.1mg, 0.8g of NaOH pellets. Dilute to 100 ml
Rosalic Acid *Preparation:* Weigh to the nearest 0.1mg, 0.5g of Rosalic acid. Add just enough of the previously prepared 0.2N NaOH to dissolve the Rosalic acid. Quantitatively transfer the resulting solution to a 50 ml volumetric flask and dilute to volume with 0.2N NaOH.
The rosalic acid should be stored in the dark at room temperature. It has a one week shelf life.
Phosphate buffer: *Preparation:* Weigh to the nearest 0.1g, 10.2g of potassium dihydrogen phosphate (KH_2PO_4). Dissolve in 300 ml of deionized water. Autoclave the resulting solution for 15 minutes at 121°C and 15 psig. The solution should be stored between 2°C and 10°C . Discard if the solution becomes cloudy.
Magnesium chloride solution: *Preparation:* Weigh to the nearest 0.1g, 40.7g of magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$). Dissolve in 500 ml of deionized water. Autoclave for 15 min at 121°C and 15 psig.

Dilution water: Preparation: To 500 ml of deionized water add 2.5 ml of $MgCl_2$ solution by means of a presterilized pipet. Add 0.7 ml of phosphate buffer by means of a presterilized pipet. Autoclave the resulting mixture for 15 min at $121^\circ C$ and 15 psig. Store the mixture in the dark until ready for use.

Procedure:

Prepare the number of culture dishes corresponding to the number of samples by adding enough of the previously prepared media to saturate the pad. (Approximately 2 ml is required) Carefully remove any excess. Using sterile forceps place a sterile membrane filter (grid up) on the base of the Gelman filtering apparatus. Carefully assemble the apparatus.

Seat the filter with a small amount of dilution water.

Transfer by means of a presterilized pipet an aliquote of sample. Record the aliquote on the benchsheet. The aliquote volume is selected based on suspended solids content, odor, and chlorine content. The selected volume should produce between 20 and 60 colonies per filter.

Apply a gentle vacuum and aspirate sample through the filter. Rinse with three successive 20 to 30 ml aliquotes of dilution water.

Immediately after the last final rinse, disassemble the filter apparatus and transfer the filter to a previously prepared culture dish by means of sterilized forceps. While transferring the filter to the culture dish care should be exercised to avoid trapping air between the pad and the filter. Place the culture dish with filter in a presterilized whirlpak. (Two culture dishes will fit in one whirlpak.)

Transfer the whirlpaks containing culture dishes to the wet incubator.

The whirlpaks should be oriented such that the culture plates are upside down. Incubate for 24 ± 2 hrs. at $44.5 \pm 0.2^\circ C$.

At the conclusion of the incubation period remove the whirlpaks containing the culture dishes from the incubator. Count the number of fecal colonies present. Fecal coliform colonies are of various sizes and are bluish in color. Nonfecal colonies are grayish to beige in color. Use of a dissecting microscope can be employed to aid in the accuracy of the count. Record the number of colonies found on the benchsheet.

Calculation:

$$\frac{\# \text{ of fecal coliform colonies found} \times 100}{\text{sample aliquote}} = \text{Fecal coliform/ 100 ml}$$

Quality Control:

Samples must be preserved by chilling to 4°C and analyzed within 6 hours of the time of collection. Sample collection time and analysis time are documented on the benchsheet.

Duplicate analysis are performed at the rate of 10%. The average values for the duplicates are reported.

Each effluent sample is run at two different dilutions to better define fecal coliform values.

One hundred ml of dilution water is analyzed at the beginning and end of each analytical session to verify lack of cross contamination.

The wet incubator temperature is calibrated quarterly against a certified thermometer. Appropriate correction are made.

Use of presterilized bottles for sample collection is encouraged. Bottle sterility is verified by performing a coliform determination on a total coliform bottle sterilized concomitantly with the fecal bottle.

HYDROLOGIC, INC.

Analytical Procedure

Title: Fluoride Determination -ISE with Distillation

Date issued: 8 Sep 1994

Supersedes:

Issued by:

Safety:

Lab coat
Safety glasses
Distillation are to be carried out in a fume hood

Preservation:

Samples are chilled to 4°C at the time of collection. Samples requiring fluoride determination are to be analyzed within 28 days of collection.

Reporting limits:

0.5 mg/L F⁻

Equipment:

ISE meter equipped with a fluoride electrode and appropriated reference electrode.
Stir plate and teflon coated stir bars
Teflon or HDPE beakers (sizes as required)
Pipets (sizes as required)
Volumetric flasks (sizes as required)
Bellack distillation apparatus with heating mantle.

Reagents:

Fluoride Stock Standard 1000 mg/L F⁻ Purchased from a reputable scientific supplier and traceable to NIST standards. Do not use stock standard that exceeds the expiration date.
Intermediate Fluoride Standard 100 mg/L: Prepare by diluting 10 ml of fluoride stock solution to 100 ml in a volumetric flask.
Total Ionic Strength Adjustment Buffer (TISAB): Purchased from a reputable scientific supplier and traceable to NIST standards. Do not use stock standard that exceeds the expiration date.
Sulfuric acid (H₂SO₄) concentrated

Procedure:

Preparation of Standard Curve:

Prepare standards that are 0.5, 1.0 and 2.0 mg/L F⁻ by diluting 0.5 ml, 1.0 ml, and 2.0 ml of intermediate F⁻ standard to 100 ml with water.

Assure that the fluoride electrode and appropriate reference electrode are installed on the ISE meter. Further, select the appropriate channel by depressing "2nd/channel" until the correct channel is displayed.

HYDROLOGIC, INC.

Analytical Procedure

Title: Total Hardness - Titrametric

Date issued: 9 Sep. 1994

Supersedes: 30 Jan. 1993

Issued by:

Safety:

Lab coat

Safety glasses

Preparation of buffer solution should be carried out under a hood.

Preservation:

Sample should be acidified to $\text{pH} < 2$ and chilled to 4°C at the time of collection. Samples for hardness must be analyzed within 6 months of collection.

Reporting limits:

1 mg/L Hardness as CaCO_3

Equipment:

Erlenmeyer flasks (125 ml)

Pipets (various sizes)

Stir plate with stir bars

Buret (50 ml)

Volumetric flask (250 ml)

Reagents:

Buffer solution pH 10 Purchased from a reputable chemical supplier. Do not use if expiration date has been exceeded.

EDTA titrant 0.01M Purchased from a reputable chemical supplier. Do not use if expiration date has been exceeded.

Eriochrome Black T Purchased from a reputable chemical supplier. Do not use if expiration date has been exceeded.

Calcium standard Purchased from a reputable chemical supplier. Do not use if expiration date has been exceeded.

Performance standard of known concentration

Procedure:

EDTA Standardization:

Into a 125 ml Erlenmeyer flask pipet 10.0 ml of calcium standard add sufficient deionized water to bring the volume to 50 ml.

Add 1 ml of buffer solution and 2 drops of Eriochrome Black T indicator.

With agitation titrate with EDTA. The color change will be from reddish to "Sky blue." Record titer of EDTA required on

benchsheet. Calculate the normality of EDTA (see Calculation section) and record on benchsheet.

Sample Determination:

Pipet 100 ml of sample into a 125 ml Erlenmeyer flask

Record sample volume on benchsheet.

Add 2 ml of buffer solution and 2 drops of Eriochrome Black T indicator.

With agitation titrate with standard EDTA to "Sky blue" endpoint.

Record titer of EDTA required on benchsheet.

Analyze a blank of 50 ml of deionized water as though it were a sample. Record the titer of standard EDTA required to reach the endpoint on the benchsheet.

Calculation:

EDTA Standardization:

$$N \text{ EDTA} = \frac{0.2}{\text{ml EDTA titer}}$$

Sample Determination:

$$\text{mg/L Hardness (as CaCO}_3\text{)} = \frac{(\text{ml EDTA}) \times N \text{ EDTA} \times 50,000}{\text{Sample volume (ml)}}$$

Relative percent difference:

$$\text{RPD} = \frac{|(\text{Repl. 1} - \text{Repl. 2})|}{((\text{Repl. 1} + \text{Repl. 2})/2)} \times 100$$

Standard recovery:

$$\% \text{ Recovery} = \frac{\text{Found Value}}{\text{True value}} \times 100$$

Quality Control:

Proper sample preservation is verified at log in.

The standard EDTA titrant is standardized against a calcium standard with each analytical session.

Duplicate analysis are performed at a rate of not less than 10%. The replicate samples must agree within the acceptance criteria established by the Precision Control Chart. Failure to meet this acceptance criteria requires that the samples be rerun. As second failure requires that contacting the supervisor for assistance in locating the problem and correcting it.

A performance standard is determined with each analytical session. The value must be within the control limits established by the Reference Standard Control Chart. Failure to meet this criteria requires contacting the supervisor for assistance in locating the problem and correcting it.

Standardizations and reagent preparation are documented in the benchsheet notebook.

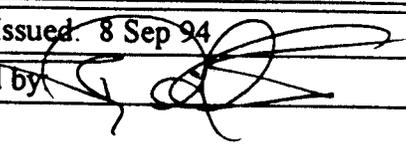
References:

USEPA, *Methods for Chemical Analysis of Water and Wastes*, "Method 130.2 Hardness Total, (mg/l as CaCO₃) Titrametric, EDTA," 1983.

Clesceri, Lenore S., et. al., Eds., *Standard Methods for the Examination of Water and Wastewater 17th Ed.*, "2340 c. EDTA Titrimetric Method," p 2-53 ff., 1989.

HYDROLOGIC, INC.

Analytical Procedure

Title: Hexavalent Chromium Aqueous Matrices	
Date Issued: 8 Sep 94	Supersedes:
Issued by: 	

- Safety:** Safety glasses
Lab coat
- Preservation:** Sample should be chilled to 4°C at the time of collection. Aqueous samples must be analyzed within 48 hours of collection.
- Reporting limits:** 0.20 mg/l Cr⁺⁶
- Equipment:** Spectrophotometer
Beakers (sizes as required)
Pipet (sizes as required)
Volumetric flasks (sizes as required)
- Reagents:**
- Stock Hexavalent chromium (Cr⁺⁶) solution: *Prepare by weighing, to the nearest 0.1 mg, 0.1414 g of primary standard potassium dichromate (K₂Cr₂O₇) which has been previously dried for 3 hours at 105°C. Quantitatively transfer to a 1000 ml volumetric flask. Add sufficient water to completely dissolve the K₂Cr₂O₇. Gently swirl flask to effect dissolution. Dilute to volume. The resulting solution is 50 ug/ml Cr⁺⁶.*
- Working Hexavalent chromium solution: *Prepare by pipeting 10.0 ml of stock Cr⁺⁶ solution into a 100 ml volumetric flask and dilute to volume. The resulting solution is 5.0 ug/ml Cr⁺⁶. Prepare fresh with each analytical session.*
- Acetone
- Diphenylcarbazide solution: *Prepare by weighing, to the nearest 1 mg, 0.250 g of 1,5-diphenylcarbazide. Quantitatively transfer with acetone to a 50 ml volumetric flask. Add sufficient acetone to fully dissolve the solids. Swirl gently to effect dissolution. Dilute to volume with acetone. Store in a labeled amber bottle. Label as to contents, date of preparation, and preparer. Discard and prepare again if the solution becomes discolored.*
- Sulfuric acid solution (0.1 N): *Prepare by diluting 2.8 ml of concentrated sulfuric acid (H₂SO₄) to 1000 ml in a volumetric flask. This solution may, alternatively, be purchased from reputable reagent suppliers.*

Procedure:

Preparation of Standard Curve:

Prepare a series of standards which are 10, 15, 40, 50, and 100 ug Cr⁺⁶ by pipetting 2, 5, 8, 10, and 20 ml of Cr⁺⁶ working solution, respectively, to 100 ml. Dilute to volume with 0.1 N H₂SO₄. (Use of weak H₂SO₄ assures that the solution pH is within the range required for proper color development.)

Transfer each standard to a labeled 250 ml beaker quantitatively. Add 2 ml of diphenylcarbazide solution to each standard. Swirl gently to assure homogeneity.

Allow the color to develop for 10 minutes and record the absorbance of each standard measured at 540 nm. Record the values on the benchsheet.

Plot the standard curve (ug Cr⁺⁶ v absorbance). Label the plot as to method, instrument used, wavelength, and date of preparation. A blank is prepared by carrying 100 ml of water through the analytical process. The resulting absorbance of the blank is subtracted from the standard and sample absorbance by means of zeroing the instrument with the blank.

Sample Determination:

Transfer a homogeneous 50 ml aliquote of sample to a 100 ml volumetric flask. A smaller sample aliquote may be used if the Cr⁺⁶ concentration exceeds the range defined by the standard curve. Record the sample volume on the benchsheet. Dilute to volume with 0.1 N H₂SO₄. (Use of weak H₂SO₄ assures that the solution pH is within the range required for proper color development.)

Quantitatively transfer the diluted sample to a labeled 250 ml beaker. Add 2 ml of diphenylcarbazide solution and swirl gently to insure homogeneity. Let the color develop for 10 minutes. Measure the absorbance at 540 nm of each sample and record the value on the benchsheet.

Calculate the ug of Cr⁺⁶ in the sample by comparing the observed absorbance to the standard curve. The ug Cr⁺⁶ found must be within the limits defined by the curve. Otherwise, a smaller aliquote as discussed above should be used.

Calculate the concentration of Cr⁺⁶ in the sample. (See calculation section)

Calculations:

ug/ml Cr⁺⁶:

$$\text{ug/ml Cr}^{+6} = \frac{\text{ug Cr}^{+6} \text{ from curve}}{\text{Sample volume (ml)}}$$

Relative percent difference:

$$\text{RPD} = \frac{|(\text{Repl 1} - \text{Repl 2})|}{((\text{Repl. 1} + \text{Repl. 2})/2)} \times 100$$

Standard recovery:

$$\% \text{ Recovery} = \frac{\text{Found value}}{\text{True value}} \times 100$$

Spike recovery:

$$\% \text{ Recovery} = \frac{(\text{ug Cr}^{+6} \text{ spike} - \text{ug Cr}^{+6} \text{ unspiked})}{\text{ug Cr}^{+6} \text{ in spike}} \times 100$$

Quality Control:

Proper sample preservation is verified at log in.

Standard curves are prepared on a quarterly basis and documented in the benchsheet notebook. The standard curve shall have the analyst name, instrument, wavelength, and date of preparation conspicuously displayed on the curve.

With each analytical session the lower detection limit standard (LDL), linearity check standard (LCS), and high standard shall be run. The recovery of each standard shall be compared to the true value as defined in the Calculation section. The recovery of each standard shall be within the control limits defined in respective standard control charts or $\pm 10\%$ which ever is less. Failure to meet these criteria requires that the standard curve be prepared again.. Duplicates shall be run at a minimum rate of 10%. The relative percent difference, as calculated as defined above, between the replicates shall be within the control limits establish by the precision control chart or $\leq 20\%$ which ever is lowest. Failure to met these criteria requires that the sample be rerun. On a second failure the

supervisor must be notified for assistance in locating and correcting the problem before reporting the results.

Samples shall be spiked at a minimum rate of 10%. Spike recovery, calculated as given in the Calculation Section, shall be within the control limits defined in the accuracy control chart or $\pm 10\%$ which ever is less. Failure to met these criteria requires that the sample and spike be rerun. A second failure necessitates notification of the supervisor for assistance in identification and resolution of the problem before reporting the results. Very high amounts of molybdenum, vanadium, iron and copper can interfere in the determination of hexavalent chromium. However, when the proper wavelength is used in the determination the interferences are negligible. If it is believed that interfering amounts of these metals are present, contact the supervisor for assistance.

References:

- Clesceri, Lenore S., *et al.*, Eds., *Standard Methods for the Examination of Water and Wastewater, 17th Ed.*, "3500-Cr D Colorimetric Method," p 3-91 ff, 1989.
- USEPA, *Methods for Chemical Analysis of Water and Wastes*, "Method 218.5 Chromium Hexavalent, Dissolved," 1983.
- USEPA, *Test Methods, for Evaluating Solid Waste, Physical/Chemical Methods, SW 846, 3rd Ed.*, "Method 7196A Chromium, Hexavalent (Colorimetric)," 1986.

HYDROLOGIC, INC.

Analytical Procedure

Title: Log-in

Date issued 6 Jan 1992

Supersedes

Issued by:

Safety: Lab coat and safety glasses required.

Equipment: Log-in forms

Computer with Labtrol installed

Procedure: Samples arrive at the laboratory from three sources. First, operators employed by HydroLogic. Second, from clients delivering their own samples. Thirdly, from customers shipping their samples to the laboratory. This procedure applies to all samples regardless of the method by which they arrive.

Verify the samples are accompanied by a chain-of-custody. The chain should contain information regarding who collected the sample, the date/time of the sample, analysis required, and preservation techniques employed. Refer to HydroLogic's Sample Preservation Procedure for correct preservation techniques.

Upon arrival the shipment container (generally a cooler) is emptied. Verify that the samples arrived at 40C. If the samples are in a water/ice mixture this requirement is satisfied.

Verify that the containers received are consistent with the information on the chain-of-custody.

Verify pH of those samples that require pH adjustment as a preservative.

In the case of fecal coliform analysis verify that the samples arrive at the laboratory within 4 hours of collection.

Enter the appropriate information on the proper log sheet.

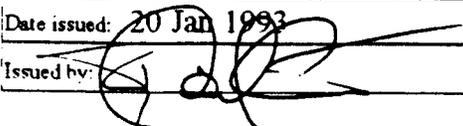
Transfer samples to the appropriate holding area. Any sample which will not be analyzed within 4 hours should be placed in a refrigerator. All coliform samples should be refrigerated until the time of analysis. Those samples receiving immediate analysis should be delivered to the appropriate analytical area and the analyst notified. Those samples with a holding time of 48 hours should be transferred to the appropriate holding area and the analyst or his supervisor notified of the receipt of the sample.

Transfer log-in information to Labtrol. Print labels for each sample. Print log-in information as prompted by Labtrol and submit to Lab Supervisor along with the chain-of-custody. Transfer labels to appropriate samples.

Quality Control: If verification are incorrect notify the Lab. Supervisor immediately. The Lab Supervisor will notify the client of protocol violations. The customer will be encouraged to resample. However, if the customer insists on running the samples the Lab. Supervisor will notify NCDEHNR of the violation as required.

HYDROLOGIC, INC

Analytical Procedure

Title: Metals Determination Flame AA	
Date issued: 20 Jan 1993	Supercedes:
Issued by: 	

Safety:

Exhaust hood
Safety glasses
Lab coat

Equipment:

Atomic absorption spectrophotometer
Hallow cathode lamps/electrodeless discharge lamps
Volumetric glassware (various sizes)
Pipets (various sizes)
10 ml buret

Reagents:

Nitric acid
Nanopure water
Stock standard solution, specific to the element of interest
Performance standard of known concentration
High purity acetylene
USP nitrous oxide
Compressed air

Procedure:

Determine the element of interest and install the proper lamp
Optimize the lamp to provide maximum energy.
Clean the appropriate burner head and install.
Install appropriate aerosol device.
Select proper gas combination and light the burner.
Optimize burner head in light path to give maximum absorbance for the high standard.

Standard curve:

Set the zero by aspirating blank water.
Aspirate each standard starting with the lowest standard and progressing to the highest.
The curve is plotted by the instrument software.
Aspirate the lower detection limit standard.
Aspirate the independent performance standard.

Sample determination:

Aspirate each sample. For total metals aspirate the previously digested sample. (See Digestion-Total Metals Procedure). For

dissolved metals aspirate an aliquote of sample previously filtered through a 0.45 um filter.

Should the sample concentration exceed the highest standard value, dilute the sample so that the observed absorbance falls within the range of the curve and rerun.

Record run on Lotus 1-2-3 spreadsheet. Archive information on floppy disk. The file name should be the symbol of the element determined followed by the date of the determination. (e.g. For a copper run on 1/2/93 the file name would be CU010293.wk1)

Calculations:

No calculation is required if the sample was run without dilution. The instrument software will present the concentration found in a suitable format.

If dilution was required

$$\text{mg/l of metal} = \frac{\text{mg/l found} \times 100}{\text{Aliquote used}}$$

QA/QC calculation:

Standard recoveries:

$$\% \text{ Recovery} = \frac{\text{Value found} \times 100}{\text{Target value}}$$

Spike recoveries:

$$\% \text{ Recovery} = \frac{(\text{mg/l sample} + \text{spike}) - (\text{mg/l sample only}) \times 100}{\text{mg/l of spike}}$$

Quality Control:

Samples should be preserved by the addition of HNO₃ to pH < 2 and cooled to 4°C at the time of collection.

Standard curve should be prepared with each analytical session. The curve should be drawn at the end of the run. A correlation coefficient of 0.995 or greater is required. A curve outside this limit should be rerun using freshly prepared standards. If this criterion is still outside acceptable limits contact the supervisor. The lower detection limit should agree ± 20% of the expected value. Should the value found produce a recovery >20% (see calculations section) the standard maybe rerun one time. If an

Quality control (cont.)

acceptable value is still unattained contact the supervisor.

The performance standard should agree within $\pm 20\%$ of the vendor's published value. Should a value found produce a recovery of $> 20\%$ (see calculations section) the standard may be run one additional time. If an acceptable value is still unattained contact the supervisor.

Duplicates should be performed at a rate of not less than 10%.

Post digestion spikes should be performed at a rate of not less than 5%. Recovery should be within $\pm 25\%$. (see calculation section)

Should more than 25 samples be determined in one analytical session run the performance evaluation as the last sample. The acceptance criteria is the same as previously described. This validates the stability of the instrument and its operating parameters.

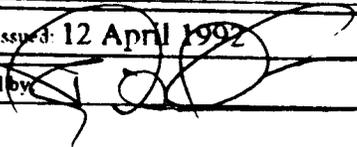
Standards preparation date is documented on the summary spreadsheet.

Proper preservation is verified at log in.

A maintenance contract should be maintained with the instrument vendor.

HYDROLOGIC, INC.

Analytical Procedure

Title: Metals Determination Furnace AA	
Date issued: 12 April 1992	Supersedes:
Issued by: 	

Safety:

Exhaust hood
Safety glasses
Lab coat

Equipment:

Atomic absorption spectrophotometer
Hallow cathode lamps/electrodeless discharge lamps
Volumetric glassware (various sizes)
Pipets (various sizes)
Graphite tubes Perkin Elmer Cat. # B012-1092
L'vov platforms Perkin Elmer Cat. # B012-1091

Reagents:

Nitric acid High Purity Standard Cat # SBHNO₃-500
Nanopure water
Stock standard solution, specific to the element of interest (Use standards purchased from High Purity Standards when available.)
Matrix modifiers appropriate to the element of interest.
Performance standard of known concentration
Argon 99.998%

Procedure:

Determine the element of interest and install the proper lamp.
Assure that the spectrophotometer optics are properly aligned for furnace operation.
Optimize lamp position in optical path for maximum energy.
Verify that the graphite furnace and L'vov platform are properly installed. Clean furnace windows as required.
Verify that the sample pipet is properly aligned so that the sample will be dispensed entirely on the L'vov platform.
Verify that the diluent reservoir and rinse reservoir a full.
Standard curve:
Through serial dilution prepare a standard equivalent to the highest standard defined by the element linearity.
Transfer a standard blank to a 2 ml beaker and place in Position 1 on the sample tray.
Transfer an aliquote of high standard to a 2 ml beaker and place in Position 2. If more than one element will be run place the second standard in Position 3 of the sample tray.

Place the appropriate matrix modifier, if required in the sample tray.

Modify the element program indicating the location of the standards and matrix modifiers as required.

When the element program is initiated the instrument will make appropriate dilutions of the stock standards, diluent, and matrix modifiers. From these dilutions a standard curve will be prepared.

Sample determination:

Transfer 2 ml of sample to a 2 ml beaker. If total metals are to be determined, place 2 ml of previously digested samples (see Digestion-Total Metals Procedure: if the samples are NC monitoring wells the Digestion-Monitoring Wells procedure) in a sample cup. To determine dissolved metals place 2 ml of sample which has been previously filtered through a 0.45 um filter in a 2 ml sample cup. Place the sample consecutively in the sample tray beginning with the lowest numbered sample position. The performance standard of known concentration should be placed as the first sample. (Note that dilution of the performance standard is often required to produce a sample which will fall within the linear range of the element.)

Label each sample in the Element File "Id/Weight Parameter" section. Labelling should include the sample id number and a brief sample description. If the sample is diluted beyond the original 100 ml this factor should also be appropriately entered. In the case of solid matrices the sample weight should be entered in the correct place in the "Id/Weight Parameter" section.

Alter the Element file to indicate the proper location, acceptable limits and run frequency for the performance standard.

Alter the Element file to indicate the samples to be spiked and spike concentration.

Initialize the instrument. If the run is not a multiple element run initialize using the "Run all" command in the element file. If a multiple element run is to be made, save the changes to all element files involved and open the "MultiElement" window. Stipulate the element to be run in the Multielement parameter section and the samples to be determined. Initialize the instrument using the "Start/Stop Auto Run" command.

The instrument will make the run utilizing the programmed delineations including diluting out of range samples. However, the instrument will only make one attempt to dilute an out of range sample. If the attempt remains out of the linear range of the element the sample must be manually diluted and rerun.

Record the run on a Lotus 1-2-3 spreadsheet. Archive the information on floppy disk. The filename should be the symbol of the element determined followed by the date the determination was begun. (E.g. For a lead run on 1/2/93 the filename would be PB010293.wk1.)

Calculations:Sample without dilution

$$\text{mg/L} = \frac{\text{ug/L}}{1.000}$$

Diluted samples

$$\text{mg/L} = \frac{\text{ug/L} \times \text{Dilution factor}}{1,000}$$

QA/QC calculation:Standard recoveries:

$$\% \text{ Recovery} = \frac{\text{Value found} \times 100}{\text{Target value}}$$

Spike recoveries:

$$\% \text{ Recovery} = \frac{(\text{ug/L sample} + \text{ug/L spike}) - (\text{ug/L sample}) \times 100}{\text{ug/L of spike}}$$

Quality Control:

Samples should be preserved by the addition of HNO_3 to $\text{pH} < 2$ and cooled to 4°C at the time of collection.

A standard curve is prepared with each analytical session and the standard should be prepared fresh with each analytical session.

A correlation coefficient of 0.995 or greater is required. If this criterion is not met contact the supervisor.

The performance standard should be run at a rate of not less than 10%. The performance standard should agree within $\pm 20\%$ of the vendor's published value. Should a value found produce a recovery outside these limits (see Calculations section) contact the supervisor.

Spike should be performed at a rate of not less than 10%.

Spike recovery should be within 80 to 120%. If recovery is outside these limits contact the supervisor. In the event that the sample is a NC drinking water sample spiking should be 100%.

Drinking water recovery should be 90 to 110%. If recoveries are outside this limit contact the supervisor.

Duplicates should be performed at a rate of not less than 10%.

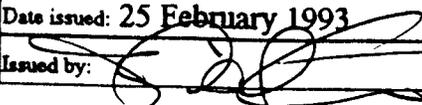
Standards preparation dates should be documented on the summary spreadsheet.

Proper preservation should be verified at log-in.

A maintenance contract should be maintained with the instrument vendor.

HYDROLOGIC, INC.

Analytical Procedure

Title: Metals Digestion - Monitoring Wells	
Date issued: 25 February 1993	Supersedes:
Issued by: 	

Safety:

Lab coat
Safety glasses
Fume hood

Equipment:

Pipets (various sizes)
Glass funnels
Whatman #41 filter paper
Beakers
Glass stir rods
Water bath (Hamilton Beach Automatic Roaster Oven)

Reagents:

1+1 Hydrochloric acid (HCl) Preparation: Mix equal volumes of water and concentrated hydrochloric acid. Agitate to insure complete mixing. Caution: Be certain to add the concentrated HCl to the water. Use caution to avoid chemical and thermal burns.

Procedure:

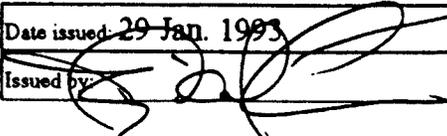
To a 250 ml beaker transfer 100 ml of well mixed sample. Add 5 ml of 1+1HCl. Heat the mixture by means of a steam bath for 15 minutes. Allow to cool and quantitatively filter through Whatman #41 filter paper into a 100 ml volumetric flask. Dilute to volume with reagent water.

Quality Control:

The sample may be collected in glass or plastic. At the time of collection the sample should be preserved with nitric acid at the rate of 5 ml/ 1L of sample. The samples should be digested within 72 hours of collection. Duplicate samples should be run at a rate of not less than 10%. Digestion spikes should be performed at a rate of 20%. Spike recovery should conform to the established recovery for the method. In no case should the recovery be greater than 120% or less than 80%. Failure of spike recovery indicates the need to rerun the sample after cause is identified and corrected. Proper preservation is verified at log-in.

HYDROLOGIC, INC.

Analytical Procedure

Title: Methylene Blue Active Substances (MBAS)	
Date issued: 29 Jan. 1993	Supercedes:
Issued by: 	

Safety:

Lab Coat

Safety Glass

All steps prior to spectrophotometric determination should be carried out under a fume hood.

Equipment:

Volumetric flask (various sizes)

Pipet (Various sizes)

Separatory funnels

Funnels

Glass wool

Spectrophotometer with cuvetts (glass)

Gelman filter apparatus, filtering flask and 0.45 um filters

Reagents:

Linear alkyl sulfonate (LAS) Ricca Cat. #4350

Chloroform

Methylene Blue Reagent Ricca Cat. #4900

Wash Solution Preparation: Fill a 1000 ml volumetric flask about half full with deionized water. Add 6.8 ml of concentrated sulfuric acid. Weigh to the nearest 0.1g, 50g of sodium dihydrogen phosphate monohydrate and quantitatively transfer to the 1000 ml flask. Swirl until dissolved. Dilute to volume with deionized water.

Procedure:**Standard curve:**

Prepare an intermediate standard by pipeting 5.0 ml of LAS stock solution to a 500 ml volumetric flask. Dilute to volume with deionized water and shake to mix.

From the intermediate standard pipet 1, 2, 4, 6, 8, and 10 ml into separatory funnels. Add sufficient water to bring the volume to 100 ml. These standards will be 1, 2, 4, 6, 8, and 10 ug LAS respectively.

Complete the analysis as described in Sample Determination Section. Plot ug LAS against absorbance to form the standard curve.

Sample Determination:

Filter a minimum of 100 ml of sample through a 0.45 um filter. Transfer 100 ml of sample or an aliquote of sample to a separatory funnel. If an aliquote of less than 100 ml of sample is used add sufficient water to bring the volume to 100 ml. Record sample volume on benchsheet.

Add 25 ml of methylene blue reagent and 10 ml of chloroform. Shake the separatory funnel for 30 seconds. Allow layers to separate and drain the chloroform layer into another separatory funnel.

Repeat the chloroform extraction three additional times using 10 ml of chloroform each time. Combine each extraction in the second separatory funnel.

To the second separatory funnel add 50 ml of wash solution. Shake for thirty seconds. Drain the chloroform through glass wool into a 100 ml volumetric flask.

Repeat the chloroform extraction twice more with 10 ml portions of chloroform. After each 10 ml addition shake the separatory funnel, allow the layers to separate, and drain the chloroform layer into a 100 ml volumetric flask.

Dilute to volume with chloroform and determine absorbance at 652 nm. Record on benchsheet.

Calculate the ug LAS in the sample by comparing the observed sample absorbance to the standard curve. Record on benchsheet.

Calculate ppm LAS in the sample (see Calculation section) and record on benchsheet.

A blank is run by using 100 ml of deionized water and carrying it through the analytical procedure. The blank is utilized to set the spectrophotometer baseline.

Calculation:

$$\text{ppm LAS} = \frac{\text{ug LAS}}{\text{Sample aliquote (ml)}}$$

Quality Control:

Samples requiring MBAS must be cooled to 4°C at the time of collection for proper preservation. The sample must be run within 48 hours of collection.

Prior to performing any analysis the analyst must prepare a standard curve. The curve is documented in the benchsheet notebook.

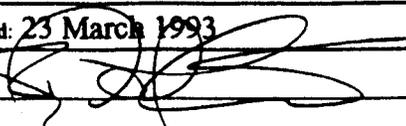
Standard curves should be updated no less frequently than once per quarter. The absorbances of the new standards when compared to the previous standard curve should produce values for ug LAS within $\pm 10\%$ of the expected value. If not, contact the supervisor. Duplicate analysis are performed at a rate of not less than 10%. For each analytical session a 1 ug, 4 ug, and 10 ug LAS standard are determined. The values obtained must be within $\pm 15\%$ of the expected value.

Sample preservation is verified at log-in.

All standards and reagents preparation are documented in the benchsheet notebook.

HYDROLOGIC, INC.

Analytical Procedure

Title: Microbiological Waste Disposal	
Date issued: 23 March 1993	Supersedes:
Issued by: 	

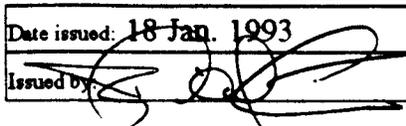
Safety: Lab Coat
Safety Glasses

Equipment: Autoclave bags
Autoclave

Procedure: All disposable contaminated microbiological is collected in the appropriately labeled receptacle. The receptacle should be lined with an autoclave bag.
When full the bag is removed and autoclaved at 121°C and 15 psig for one hour.
Remove from the autoclave, let cool, and dispose of in the usual manner.

HYDROLOGIC, INC.

Analytical Procedure

Title: Nitrate Nitrogen-Brucine Sulfanilic Acid	
Date issued: 18 Jan. 1993	Supersedes:
Issued by: 	

Safety:

Lab Coat
Safety Glasses

Equipment:

Hot water bath
Cold water bath
Spectrophotometer
Test tubes
Pipets (various sizes)
Cuvets

Reagents:

13 N Sulfuric acid (H₂SO₄) Preparation: Place 125 ml of deionized water in a 1000 ml beaker. Slowly add 500 ml of concentrated sulfuric acid. Extreme caution should be exercised, the heat of solution is markedly exothermic.
Nitrate standard Ricca Cat. # 5457
Brucine sulfate reagent Ricca Cat # 1420

Procedure:Standard curve:

Begin to heat hot water bath to 100°C. A good deal of time is required to achieve this temperature. Do not continue the analysis until the hot water bath reaches this temperature.

Dilute the nitrate stock standard ten time to produce an intermediate standard which is 100 ppm N(NO₃). Into separate test tubes pipet 1.0, 2.0, 4.0, 7.0, & 10.0 ml of intermediate standard. Add sufficient water to bring the total volume to 10.0 ml. These test tubes correspond to 1.0 ug, 2.0 ug, 4.0 ug, 7.0 ug & 10.0 ug N(NO₃) respectively.

A blank is run concurrently using 10.0 ml of deionized water. To each standard add 10 ml of 13 N H₂SO₄. Swirl to mix and place immediately in the cold water bath at 20°-25°C.

Allow test tubes to come to thermal equilibrium.

Add 0.50 ml of brucine sulfanilic acid solution to each tube and transfer to the hot water bath at 100°C. Accuracy of the test is incumbent on the hot water bath being 100°C. careful control of this is, therefore, required. Warm the test tubes for exactly 25 minutes.

After 25 minutes remove the tubes to the cold water bath and allow them to come to thermal equilibrium.

Using the blank set the baseline for the determination at 410 nm. Determine the absorbance of each standard and record on the benchsheet. Draw the standard curve and archive in the benchsheet notebook.

Sample Determination:

Place 10.0 ml of sample or an aliquote diluted to 10.0 ml in a test tube. Record the sample volume on the benchsheet.

Add 10.0 ml of 13 N H_2SO_4 , swirl to mix and place in the cold water bath at 20°-25°C. Allow the tube to come to thermal equilibrium.

Add 0.50 ml of brucine-sulfate solution to the sample and transfer to the hot water bath at 100°C. Warm for exactly 25 minutes.

After 25 minutes remove the tubes to the cold water bath and allow to come to thermal equilibrium.

Determine the absorbance of each solution at 410 nm and record the value on the benchsheet.

Determine the $\text{N}(\text{NO}_3)$ content by comparing the sample's absorbance against the standard curve. Record the value found on the benchsheet.

Calculation:

$$\text{ppm N}(\text{NO}_3) = \frac{\text{ug N}(\text{NO}_3)}{\text{Sample Aliquote (ml)}}$$

METHOD REFERENCES

"Methods of Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, EMSL, Cincinnati, OH, March 1979 (EPA-600/4-79-020).

Standard Methods for Examination of Water and Wastewater, 15th Ed., 1981, American Public Health Association, Washington, D.C.

Hach Handbook of Water Analysis, 1979, Hach Chemical Company, PO Box 389, Loveland, CO 80537.

Standard Methods for Examination of Water and Wastewater, 16th Ed., 1985, American Public Health Association, Washington, D.C.

"The Determination of Inorganic Anions in Water by Ion Chromatography", U.S. Environmental Protection Agency, EMSL, Cincinnati, OH 45260, March 1984 (EPA-600/4-84-817).

EPA 600 Series Methods, 40 CFR Part 136, Federal Register, Friday October 26, 1984.

"Test Methods for Evaluating Solid Waste", U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C., Second Edition, Revised, April 1984 (EPA SW-846).

Statement of Work for Organics Analysis, USEPA Contract Laboratory Program, 10/86.

Annual Book of ASTM Standards, American Society for Testing and Materials, 1980.

"Soil Survey of Laboratory Methods and Procedures for Collecting Soil Samples", Soil Conservation Service, Washington, D.C., 1972.

"Leaking Underground Storage Tank and Petroleum; Analytical and Quality Assurance Guidance", Wisconsin Department of Natural Resources, July 1993.

22.0 Standard Traceability

Whenever such materials are available, the lab uses NIST traceable standards for its reference comparisons. Requisitions for standard reference materials are kept on file. Vendor-supplied certificates are maintained on file that certify standard purity, QA information, and Traceability.

23.0 Laboratory Safety Procedures

Lab safety is an integral part of scientific work and is largely a matter of experience and common sense. However, it is the responsibility of anyone working in a technical environment to familiarize themselves with the safety rules, equipment, and hazards of their workplace. Lab technicians and analysts have special responsibilities in this area to promote and maintain a safe working environment.

Lab personnel can prevent most damage and injury by using good judgement based on sound safety information, following the safety guidelines of the lab, and asking questions about any aspect of their work about which they are unsure. Prevention of accidents is paramount.

To ensure proper safety requirements, the Safety Director inspects the premises on a weekly basis. Figure 8 is the Laboratory Safety Inspection Form completed during the weekly inspections.

24.0 General Safety Procedures

The elements of the laboratory safety and chemical hygiene plan are detailed in the Chemical Hygiene Plan/Safety Manual document. This document is currently undergoing revision.

An outline of key elements presented in this document illustrating general safety procedures used by the lab appears below;

1. Special handling instructions for equipment are followed.
2. Safety equipment is located throughout the lab.
3. First aid and fire extinguisher equipment are readily available to lab personnel.
4. Safety practices of the lab are constantly monitored and improved to provide a safe working environment.
5. New employees are familiarized with the safety program, the CHO/SM, and possible hazards of their workplace.

LABORATORY SAFETY INSPECTION FORM

HYDROLOGIC - FRANKFORT, KY

WEEKLY REPORT

INSPECTION BY:

DATE:

FIRE EXTINGUISHERS

- SECURELY MOUNTED
- LABELED CLEARLY
- FULL AND INSPECTED WITHIN LAST 6 MONTHS
- ABC ONLY IN NON-EQUIPMENT AREAS
- ACCESS NOT BLOCKED
- EXTINGUISHERS IDENTIFIED

GC	SVOA	VOA	EXTRACT

SAFETY APPARATUS

- FIRST AID AVAILABLE AND EASILY ACCESSIBLE
- SPILL CLEANUP MATERIALS AVAILABLE
- SAFETY SHOWERS OPERATIONAL
- EYEWASH STATIONS OPERATIONAL
- SHOWERS AND STATIONS UNBLOCKED
- MATERIAL SAFETY DATA SHEETS AVAILABLE
- SPILL LOG FILED AND AVAILABLE
- EMERGENCY LIGHTS FUNCTIONAL
- EXIT SIGNS FUNCTIONAL, UNBLOCKED

PERSONAL SAFETY

- SAFETY GLASSES, FACE SHIELD, GOGGLES WORN
- NO CONTACT LENSES WORN IN LAB
- LAB APRONS / COATS WORN
- ACID APRONS WORN WHERE NECESSARY
- GLOVES SUITED FOR HAZARD
- HAIR BELOW SHOULDER LENGTH TIED BACK
- NO EXCESSIVE JEWELRY
- RESPIRATORS WORN WHERE NECESSARY
- RESPIRATORS MAINTAINED PROPERLY
- NO FOOD OR DRINK IN LAB AREAS OR IN SAMPLE COOLER
- NO SMOKING IN LAB AREAS

FLAMMABLE CHEMICAL STORAGE

CABINETS PROPERLY IDENTIFIED WITH LABELS
ALL LARGE QUANTITIES (> 1 LITER) IN CABINETS
NO FLAMMABLE PRODUCTS NEAR HEAT SOURCES
CONTAINERS LABELED PROPERLY

NON-FLAMMABLE CHEMICAL STORAGE

STORAGE AREAS PROPERLY IDENTIFIED WITH LABELS
CONTAINERS PROPERLY LABELED

ACID STORAGE

CABINETS PROPERLY IDENTIFIED WITH LABELS
ALL LARGE QUANTITIES (> 1 LITER) IN CABINETS
CONTAINERS LABELED PROPERLY
ALL ACIDS STORED IN PROPER CABINETS

SATELLITE ACCUMULATION POINTS

INSPECTED FOR LEAKS
SECURITY INTACT
EXCESS FUMES
DRUMS IN GOOD CONDITION
SEALS IN TACT
LABELS CORRECT AND IN VIEW

REFRIGERATORS

REFRIGERATORS PROPERLY IDENTIFIED WITH LABELS
REFRIGERATORS CLEAN, ITEMS NEATLY STORED
SAMPLE CONTAINERS PROPERLY LABELED
HAZARDOUS SAMPLES PROPERLY LABELED

25.0 Laboratory Notebooks

Lab notebooks will serve as permanent records of tests performed in the lab. Notebooks should contain enough information about a particular analysis that another analyst can clearly understand what occurred during the test.

Ball point pens or pens with permanent ink are to be used for all notebook or logbook entries. Use of a pencil for this documentation is unacceptable. Corrections must be made by single-line strike outs and these authorized corrections must be initialed and dated. Obliteration and use of liquid paper for these documents is unacceptable.

Lab notebooks are used to document routine analyses and any observed irregularities. Unusual observations are to be noted. If the analyst deviates from the standard methodology, this is fully documented. Formulae used for each data set appear in the notebooks.

Computer calculation records are also considered records of analytical testing. These records shall be stored so as to facilitate retrieval and review if necessary. It is not necessary to show each individual calculation.

Lab notebooks are periodically reviewed by the Lab Director, QA/QC Officer, or Section Supervisor. The reviewer initials and dates each page of the notebook.

26.0 Glassware Cleaning

In general, lab glassware is cleaned with an approved laboratory detergent and thoroughly rinsed with deionized water. Containers are rinsed immediately after use with tap water to minimize build up of residues.

Lab personnel are required to properly dispose of any contents in the apparatus to be cleaned immediately after use and prior to washing the apparatus.

Glassware should be cleaned immediately after being used in the digestion, concentration, or analysis of samples.

ORGANIC glassware cleaning includes the following steps:

1. Rinse glassware with extraction solvent soon after use.
2. Scrub all surfaces of the glassware in hot water/lab detergent solution.
3. Rinse three times with tap water.
4. Rinse thoroughly with deionized water.
5. Air dry glassware and store properly.
6. Solvent rinse glassware with extraction solvent prior to use and allow to air dry.
7. Store clean glassware with aluminum foil caps. Aluminum foil should be solvent-rinsed.

Chipped, damaged, or broken glassware is to be immediately discarded and not used for subsequent analysis once such flaws are detected by lab personnel.

27.0 Employee Training Procedures

Personnel employed by the analytical lab must have the necessary attitudes, knowledge, and skills to do their job. This includes all job functions involving data quality from sample collection to data reporting.

Using prior experience or knowledge as the basis of action, an analyst can gain experience and problem solving through basic on-the-job training. This training evolves through various stages listed below:

1. Observation of experienced operators performing a task.
2. Actual performance of the task under direct supervision of an experienced analyst.
3. Continued measurement of analytical performance through Performance Evaluation samples and supervisory observation.

Required certification of personnel follow EPA and state certifying agency's recommendations for tenure and skill levels. Employees are encouraged to improve their professional credentials by attending courses at local colleges and universities. Instrument demonstrations and training courses are other ways that new skills can be acquired and implemented into lab operations. These courses contribute to the general skill level of the lab and assist the lab's effort to produce high quality data for all analytical testing.

The Section Supervisor is responsible for ensuring that an analyst learns a new procedure and completely understands that procedure prior to performing it on real analytical samples. This process includes:

1. Establishing analyst familiarity with the Standard Operating Procedure (SOP). This includes answering questions on the SOP and suggesting further references.
2. Guiding new employees through practice sample analysis.
3. Observation of performance and corrective suggestions.
4. Documentation of the training process.

A Training Documentation Form records this process and documents that an analyst has read and understands each procedure applicable to their jobs. Sample Log Books serve as a permanent and ongoing record of analytical performance. These logs are periodically reviewed as an aid to employee evaluation and further training. The Training Documentation Form (TDF) appears in Figure 9.

28.0 Laboratory Quality Control Procedures

An effective QA/QC Program is vital to the validity of analytical data produced by the lab. Many decisions are made based on quality analytical data. It is the responsibility of the lab to provide that data.

Sample collection, preservation, handling, storage, and analytical methodology are chosen with respect to precision, accuracy, and the specific needs of a given analytical project. Standard approved methods are used for analysis and any additional necessary analytical steps carefully documented.

The lab uses several measures of internal quality control to ensure data quality. These include blank, spikes, and duplicate analysis at required EPA frequencies. Analytical SOPs detail the number and frequency of these checks.

In general, the descriptions of the internal quality checks are as follows:

Method Blank Analysis: A method blank is a "clean" sample (i.e., containing no analyte of concern), most often distilled water, to which all reagents and analytical procedures are performed. Method blanks are analyzed at a rate of one per sample lot, every twenty samples, or daily. A method blank is carried through the entire analytical scheme. The method blank volume should be approximately equal to the sample volumes being processed. A method blank is used in all analyses to verify that the determined concentrations do not reflect contamination. If consistent high blanks are observed, laboratory glassware and reagents are checked for contamination and the analysis halted until the system is brought under control.

Organic Surrogate Spike Analyses: Each sample and blank is spiked with one or more "surrogate" compounds during preparation operations. These surrogate standards are chosen because they have properties similar to sample analytes of the same interest, but are most likely absent from the natural sample. This procedure is used to evaluate the ability of the analytical procedures to recover the true amount of a known compound.

The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis, and the percent recoveries of the surrogate standards are determined. Recoveries should be within the acceptable control limits as specified for each compound. If control limits are exceeded for surrogate standards, calculations are checked for errors. If calculations are correct, internal standards and surrogate spiking solutions are checked for degradation, contamination, or solvent evaporation. The instrument performance is then checked. If all previous items are acceptable, re-purge or re-extraction of the sample maybe necessary depending on the situation and sample availability. If all other measures fail to reveal the reason for low surrogate recovery, check for matrix interference and recalibrate if necessary.

Matrix Spike Matrix Spike Duplicate Analyses: To evaluate the effect of the sample matrix upon analytical methodology, two separate aliquot samples are spiked with a standard mix of compounds appropriate to a given analysis. The matrix spike/matrix

spike duplicate (MS/MSD) are analyzed at a frequency of one set per twenty samples. The percent recovery for the spiking compounds is calculated. The relative difference (RPD) between the MS/MSD is also calculated.

The observed percent recoveries and RPD between the MS/MSD are used to determine the accuracy and the precision of the analytical methods for the sample matrix. If the percent recovery and RPD results exceed the control limits as specified for each spiking compound, calculations are checked for errors. If no errors exist then a re-analysis is required unless a situation of matrix interference exists.

Organic Internal Standards: An Internal Standard is used to correct for variations due to sample injection techniques and some matrix interferences. Internal standards are added to the sample by the analyst, after extraction and before analysis at a frequency consistent with the method being used. The area counts of the Internal Standard are compared to the daily standard used to calibrate the instrument. If the Internal standard recovery falls outside of the acceptable control limits, the internal standard is checked for any degradation, contamination, or solvent evaporation. The operation of the system and its calibration is evaluated if the internal standard is deemed acceptable.

Duplicate Sample Analyses: Duplicate analyses are performed only for those parameters which do not permit matrix spike recovery determination to evaluate the reproducibility of the method. Results of the duplicate analyses are used to determine the relative percent difference (RPD) between replicate samples. For each parameter analyzed at least one duplicate sample is run per group of 20 samples. If possible, the duplicate analysis should be performed on a sample for which original result is above the detection limit.

A file of quality control samples of known concentration is maintained in the lab. This check standard bank is a valuable source of performance test samples, instrument performance check standards, and training sample spikes, duplicates, etc. A detailed listing of available quality control samples, their identity, and concentration is maintained in the file. Results of quality control samples document the validity of data and help control data quality within predetermined acceptance limits.

The following sections detail the types of quality control samples analyzed by the lab to validate the accuracy and precision of its measurements.

29.0 Performance Evaluation Standards

Performance Evaluation Standards provide interlaboratory comparison and are a valuable performance indicator for the lab. The lab routinely participates in the U.S. EPA Water Pollution (WP) audit program. Twice yearly results from these samples are used as a requirement for maintaining state certifications. Results are reviewed by the Lab Director, QA/QC Officer, and Section Supervisors with their assigned personnel. These standards are a valuable gauge of individual analyst performance under normal lab operating conditions.

If the lab fails in its ability to analyze samples of this type, the entire analytical measurement process shall be immediately examined to determine the cause of these errors. This process shall be documented and maintained as a part of these performance standard records.

30.0 Performance and System Audits

The Lab QA/QC Officer carries out two basic types of audits in order to ensure the production of high quality data by the lab. These are Systems Audits and Performance Audits. Each is explained below.

SYSTEM AUDITS are qualitative evaluations of all components of field and laboratory quality control measurement systems. They determine the effectiveness of the execution of these systems as designed. Such audits evaluate executed versus scheduled QA/QC activities for a given project. An example is the data management system audit in which only data collection and management activities are addressed.

PERFORMANCE AUDITS are a quantitative evaluation of the measurement systems of a program. Accuracy and precision are determined on samples of known composition in order to test a specific measurement system. This action may be overtly or covertly implemented by the QA Officer.

PRECISION is a term denoting the scatter of results around a target data point. Precision improves as scatter becomes smaller. Precision is measured as standard deviation and reported as relative percent difference.

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ACCURACY is the degree of agreement of an analytical result with the true value. Systematic and random errors can influence accuracy. Accuracy of a given data set is determined from recovery of spiked samples and surrogates. Although spiked samples are an estimate due to matrix or method interference effects, they are used to determine accuracy acceptance limits. The overall accuracy for each compound is the arithmetic mean over all of the spiked samples.

The ACCURACY for each compound is calculated using the formula below:

$$A = (R-X)/T \times 100$$

where: A= recovery of the added spike

R= analytical result of the spiked sample in ug/l (ppb)

X= mean concentration of duplicate results

T= true value of the added spike

31.0 Evaluation of QA/QC Data

When the analysis of a sample set is completed, the results are reviewed and evaluated to assess the validity of the data set. The Lab Director completes and documents this review process prior to the release of analytical data from the lab. The Lab Director's signature authorizes release to the client on each data report. A Data Evaluation Checklist documents this procedure as illustrated in Figure 10.

The Lab Director considers many factors in this evaluation. Some of these have been previously discussed in this document and are summarized in the following section:

1. Review of Accuracy and Precision measurements associated with this sample set.
2. Specific instrument checks.
3. Calibration data.
4. Operational parameters such as GC or GC/MS conditions.
5. Data history of site if available.
6. Internal logic of results.
7. Sampling data directly affecting results.

All laboratory data are routinely assessed for precision, accuracy, and completeness. Initially these evaluations are applied over a broad concentration range. As more data points are accumulated, precision and accuracy acceptance limits are developed to define accuracy and precision over specific concentration ranges.

A minimum of ten measurements of accuracy and precision are required to establish initial control limits. Twenty or more measurements shall be used thereafter to establish these limits.

In general, control limits of two standard deviations, a 95% Confidence Interval, shall be the Warning Limit and three standard deviations, a 99% Confidence Interval, shall be the Upper and Lower Control Limits for the test. Control Limits are updated as additional accuracy and precision data become available.

Control charts, if required by a state agency or requested by a specific client, are used to maintain a permanent record of the accuracy and precision of their data.

These charts make it easy for analysts to spot outliers or out of control events that generate non-acceptable data (data that does not meet established acceptance criteria). These charts also are valuable for illustrating trends in analytical data.

An OUTLIER is an extreme value, either high or low, which has questionable validity as a member of a measurement set. Outliers may be rejected from a data set in which a known experimental aberration occurred, or the data fails a statistical criterion called the "t" value.

Failure of this test occurs when the datum exceeds the tabulated two tailed students' t for $t = 0.05$ at $n-1$ degrees of freedom. The t value is calculated using the formula below:

$$t = (X - X_1) / S$$

where: X_1 = the extreme value being statistically treated
 X = the mean of the measurement set for n observations
 S = the standard deviation associated with X

If the value is found to be an outlier and rejected, the mean and standard deviation are recalculated using the remaining values in the data set. This procedure is reiterated until no outliers remain.

HYDROLOGIC LABORATORY EQUIPMENT
 UPDATED FEBRUARY 4, 1994

ITEM	MODEL	AGE(YR)
Gas Chromatograph/ Mass Spectrometer Column: DB5-MS 30m, 0.32mm ID Film Thickness: 1.0 microns Linear Velocity = 40 cm/sec Temperature Program = 45 deg. C for 5 min; 300 deg. C at a rate of 5 deg/min Autosampler: CTC A200S; 105 positions	Incos XL Finnigan	0
Gas Chromatograph/ Mass Spectrometer Column: Resteck RTX 502.2 105 m, 0.52mm ID Film Thickness: 1.0 microns Carrier Flow = 8 ml/sec Temperature Program = 75 deg. C for 4 min; 220 deg. C at a rate of 8 deg/min Autosampler: Tekmar LSC200/2016	4500 Finnigan	9
Gas Chromatograph/ Mass Spectrometer with Tremetrics 9000 series Gas Chromatograph Column: JW DB-5 MS 30m, 0.32mm ID Film Thickness: 1.0 microns Linear Velocity = 40 cm/sec Temperature Program = 45 deg. C for 5 min; 300 deg. C at a rate of 8 deg/min Autosampler: Tremetrics 774; 42 positions	1050 Finnigan	8
Gas Chromatograph/ Mass Spectrometer Column: Resteck RTX-1 60m, 0.53mm ID Film Thickness: 3.0 microns Carrier Flow = 8 ml/min Temperature Program = 45 deg. C for 5 min; 180 deg. C at a rate of 8 deg/min Autosampler: Tekmar LSC/ALS; 10 positions	3400 Varian	

Gas Chromatograph/
 ECD/Flame Ionization 9000 Tremetrics 1
 Column: 3% SP-2100 on 100/120 Supleco Port
 Carrier Flow = 30 ml/min
 Temperature Program (FID) = 50 deg. C for 7 min; increase at
 a rate of 20 deg/min; 260 deg. C
 for 5.5 min.
 Temperature Program (ECD) = 200 deg C for 40 min
 Autosampler: Tremetrics 774; 42 positions

Gas Chromatograph/
 Photo Ionization & 9000 Tremetrics 3
 HECD & Flame Ionization
 Column: J&W DB-VRX 75m, 0.45mm ID
 FID: Vocal 30m, 0.52mm ID
 Film Thickness: 1.0 microns
 Carrier Flow = 8 ml/min
 Temperature Program = 35 deg. C for 5 min; 180 deg. C at a
 rate of 5 deg/min, hold for 1 min.; 200
 deg C at a rate of 10 deg/min. hold for
 12 min.
 Autosampler: Dynatech Dynasoil; 30 positions

Gas Chromatograph/
 Electron Capture & 5890 Hewlett Packard
 Flame Ionization
 Column: Supleco Volcol 30m, 0.53mm ID
 Film Thickness: 3.0 microns
 Column: J&W DB-608 30m, 0.53mm ID
 Film Thickness: 0.83 microns
 Carrier Flow = 8 ml/min
 Temperature Program (FID) = 60 deg. for 7 min.; 200 deg. C at
 a rate of 15 deg/min, hold for 2.5
 min.
 Temperature Program (ECD) = 140 deg. C for 1 min; 240 deg. C
 at a rate of 15 deg/min, hold for
 7 min.; 265 deg C at a rate of 6
 deg/min. hold for 13 min.
 Autosampler: Hewlett Packard 7673a; 100 positions &
 Dynatech PTA30NS; 30 positions

Gas Chromatograph/
Photo Ionization & 3400 Varian
HECD
Column: J&W DB-624 75m, 0.53mm ID
Film Thickness: 0.83 microns
Carrier Flow = 8 ml/min
Temperature Program = 20 deg. C for 5 min; 180 deg. C at a
rate of 10 deg/min, hold for 10 min.
Autosampler: Dynatech Dynasoil; 30 positions

Gas Chromatograph/
Photo Ionization & 3400 Varian
Flame Ionization
Column: J&W DB-624 75m, 0.53mm ID
Film Thickness: 1.0 micron
Carrier Flow = 8 ml/min
Temperature Program = 70 deg. C for 5 min; 200 deg. C at a
rate of 15 deg/min, hold for 2 min.
Autosampler: Dynatech PT30WS; 30 positions

Gas Chromatograph/
Flame Ionization & 3400 Varian
Flame Ionization
Columns: 3% SP-2100 on 100/120 Supleco Port
Carrier Flow = 30 ml/min
Temperature Program = 50 deg. C for 7 min; 250 deg. C at a
rate of 20 deg/min, hold for 10 min.
Autosampler: Tremetrics 774; 42 positions

Certification:

HydroLogic-Frankfort is certified in the following states and areas:

<u>State</u>	<u>Category</u>	<u>Certification Number</u>
Georgia	Wastewater/UST	N/A
Indiana	Drinking Water - Organics	C-KY-03
Kentucky	Drinking Waster - Organics	00065
New Jersey	Wastewater	58957
North Carolina	Wastewater/Groundwater	399
Oklahoma	Wastewater/UST	9430
South Carolina	Wastewater	70002
Tennessee	UST Parameters	N/A
West Virginia	Wastewater/UST	093

Methodology:

HydroLogic-Frankfort strictly adheres to methodology for the 500 and 600 Series published by the Environmental Protection Agency. HydroLogic-Frankfort also performs work according to SW846 procedures.

Quality Control:

HydroLogic-Frankfort has a Quality Assurance Manual and Standard Operating Procedures Manual. These procedures and guidelines are strictly adhered to for the assurance of accurate data. Both of these manuals are available upon request.

Containers and Shipping:

HydroLogic-Frankfort supplies all containers, chain of custody documents, labels, preservatives, coolers, and bubble wrap to clients who request these items. All containers are shipped new to avoid any contamination problems. A temperature blank is included in each cooler sent out and is requested to be returned with each cooler in order to assure proper preservation of all samples. Containers received at the laboratory are never reused but disposed of according to Federal and State Hazardous Wastes regulations.

HydroLogic-Frankfort ships and receives samples through transportation services of Federal Express and United Parcel Services. All shipments are on a priority overnight basis. HydroLogic assumes the responsibility of cost for all shipping cost to and from our facilities. After receipt a fax will be sent confirming the receipt and expected completion date of the project. The project manager or the sample custodian will phone to eliminate and discrepancies if needed.

Invoices:

Invoices will be sent in the same envelope with the hard copy of each project upon completion.

FIGURE 1. QUALITY ASSURANCE PROGRAM

OVERVIEW

OPERATION	PROCEDURE	AUDIT	FREQUENCY	
Sample Preparation-----		- Method Blank	1	
		- Spike	2	
		- Duplicate	2	
		- Reagents	5	
Sample Analysis	GC-----	- Standard Mix	3	
	GC/MS-----	- Mass Calibration	3	
		- Response Calibration	3	
		- Standards	3	
Data Reduction-	GC----	Qualitative----	- GC Retention Times	4
			- Verification By Second Analysis	4
		Quantitative----	- Peak Measurement	4
			- Calculations	4
	GC/MS	Qualitative----	- Computer Match	4
			- Reference Spectra	4
			- GC Retention Times	4
		Quantitative----	- Peak Measurement	4
			- Calculations	4
Data Reporting-----		- Completeness	1	
		- Accuracy	1	

FREQUENCY CODES:

- 1 once per project
- 2 once per 20 samples
- 3 daily
- 4 in every case
- 5 as new reagents are received

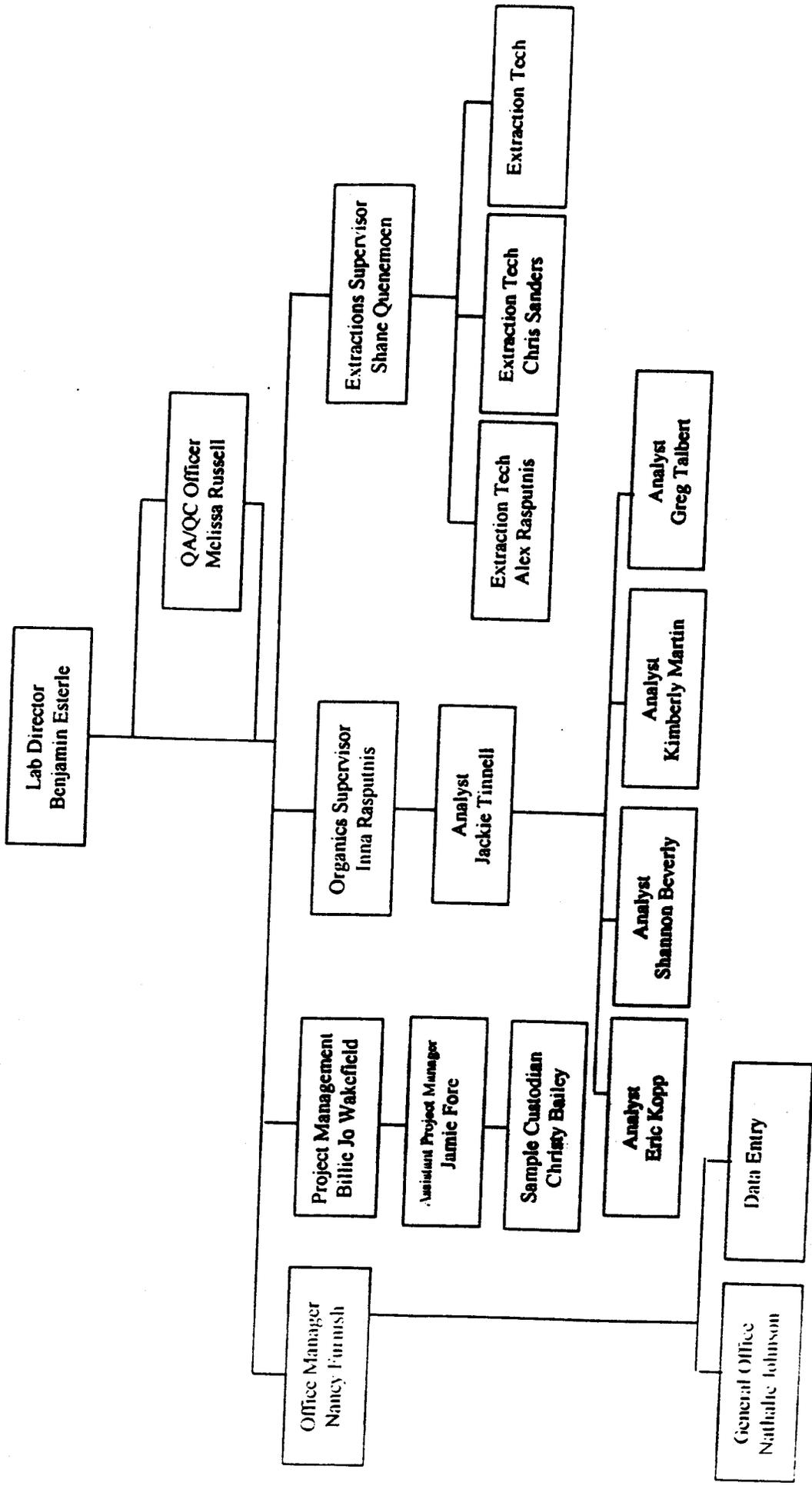


Figure 2

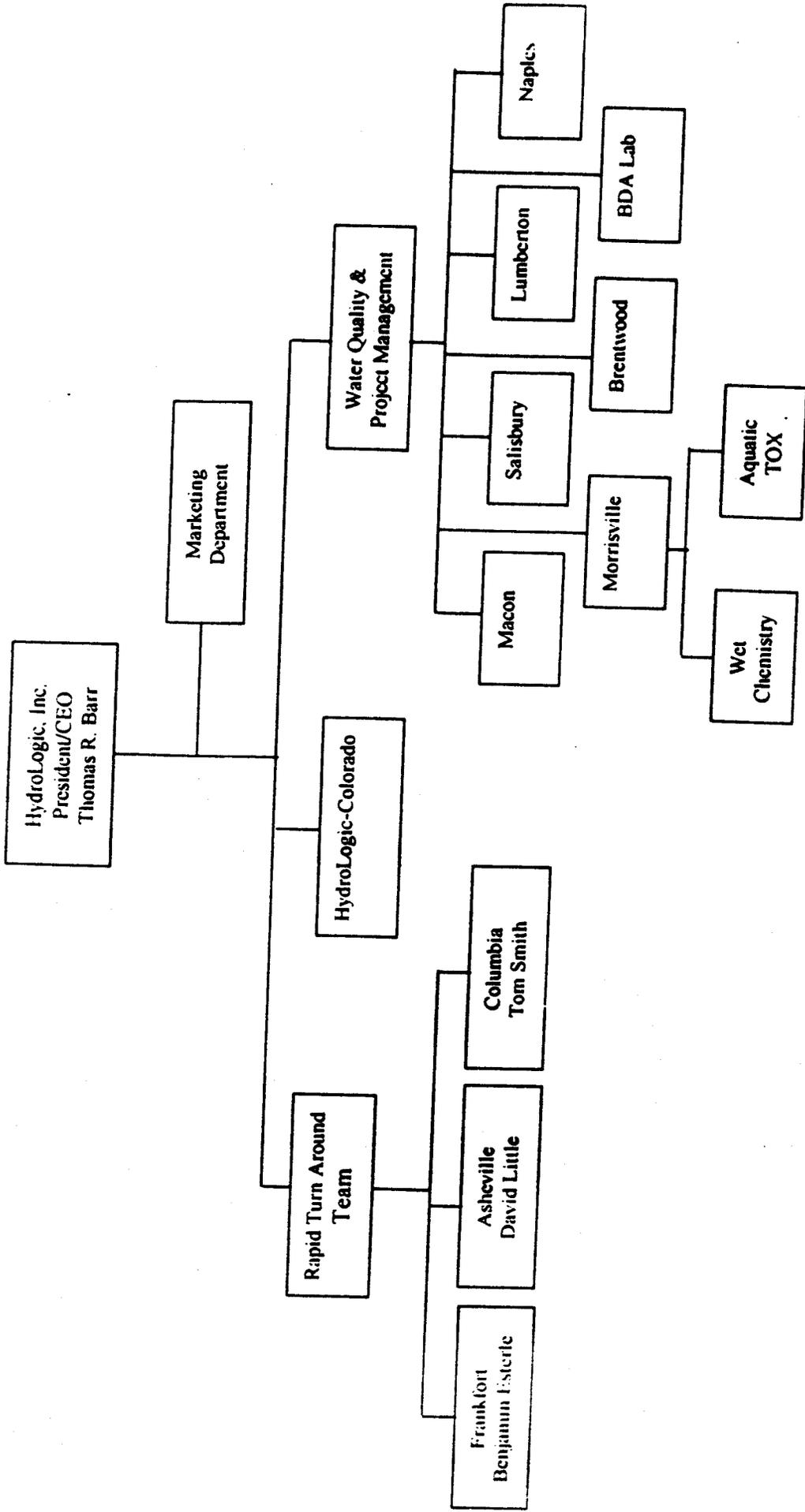


Figure 3

3.0 LABORATORY FACILITIES/EQUIPMENT

The Chemistry Section of the lab occupies roughly 4500 sq. ft. The lab is equipped with advanced Gas Chromatograph instrumentation and support instrumentation essential to a modern environmental facility. Figures 4 and 5 represents a floor plan of the lab detailing office and lab space.

Access to the lab is limited to authorized personnel. During normal working hours, access to the lab is through the monitored reception area at the lab entrance. Visitors are required to be accompanied by a lab employee while in the building.

The lab is equipped with advanced instrumentality for the fast, accurate and precise analyses of environmental samples. A listing of equipment appears in Figure 6.

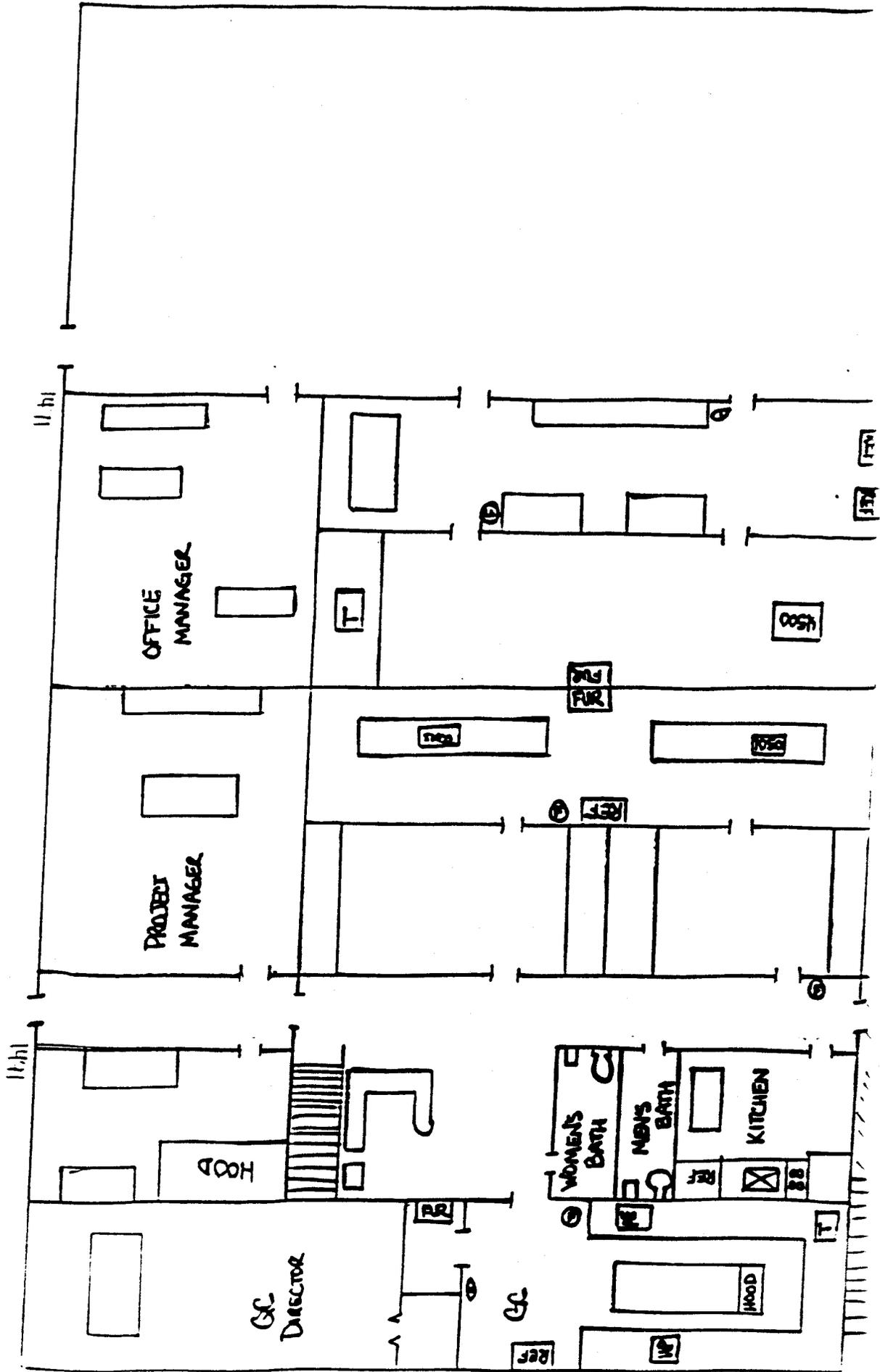
Figure 6. Major Laboratory Equipment

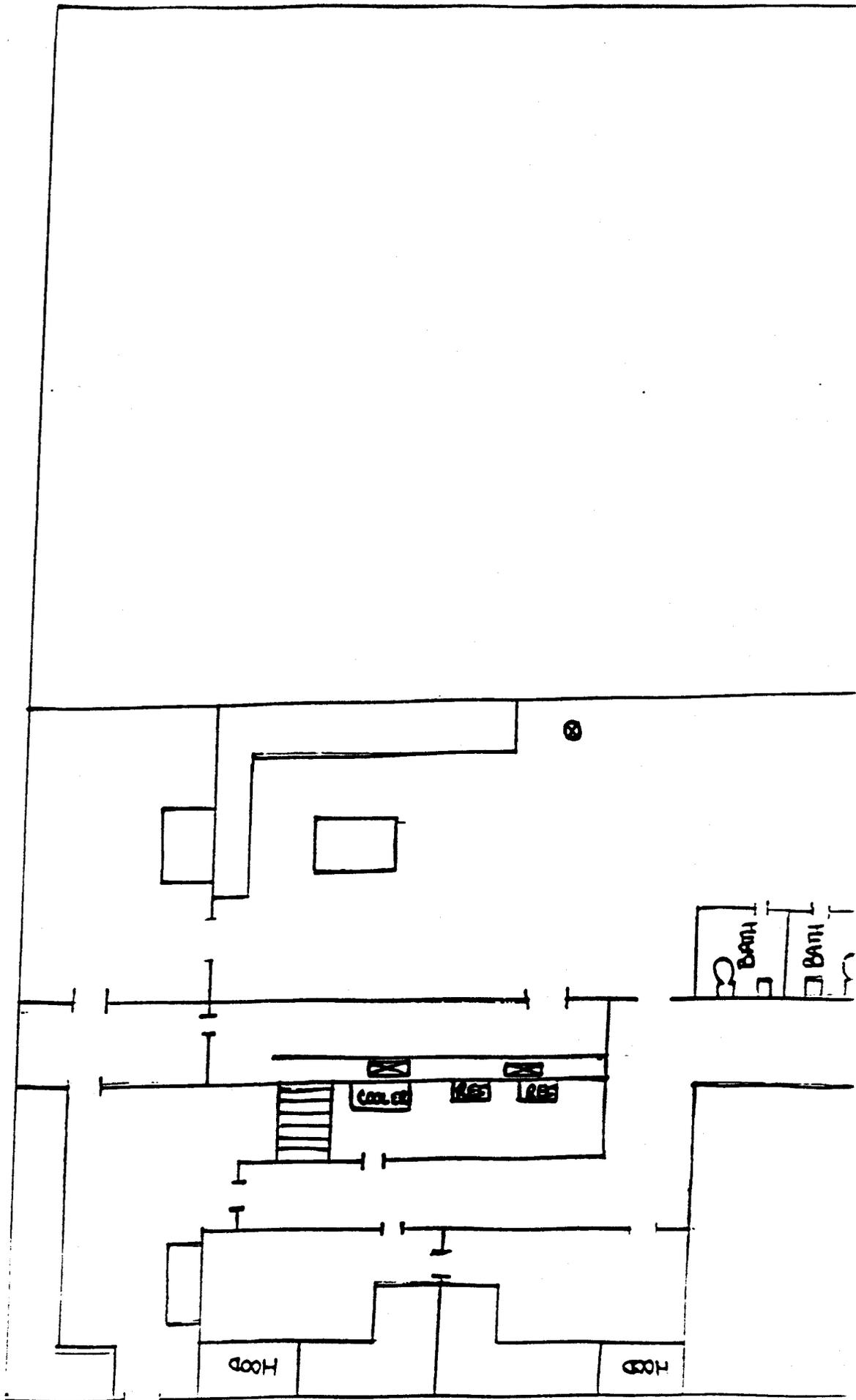
Item	Model	Age(yr)
Gas Chromatograph/ Mass Spectrometer	Incos XL Finnigan	0
Gas Chromatograph/ Mass Spectrometer	4500 Finnigan	9
Gas Chromatograph/ Mass Spectrometer	1050 Finnigan	8
Gas Chromatograph/ Mass Spectrometer	3400 Varian	
Gas Chromatograph/ Flame Ionization Auto Sampler	9000 Tremetrics	1
Gas Chromatograph/ Electron Capture	8300 Perkin Elmer	8
Gas Chromatograph/ Photo Ionization & HECD & Flame Ionization	9000 Tremetrics	2.5
Gas Chromatograph/ Flame Ionization	5890 Hewlett Packard	0
Gas Chromatograph/ Photo Ionization & HECD & Flame Ionization	Varian 3400	
Gas Chromatograph/ Photo Ionization HECD & Flame Ionization	Varian 3400	

4.0 SAMPLE COLLECTION/CUSTODY/TRACKING

Special consideration is given to the procurement, storage, and transportation of environmental samples to be analyzed. Hydrologic-Frankfort does not supply services for sampling, however, often supplies containers and preservatives for clients. U.S. EPA protocols are followed for containers, amounts, types,

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methodology, and sample holding times. Procedures ensure that analytes originally present in the sample matrix do not undergo degradation or concentration, and that contaminants which might interfere with the analysis have not been added. The following are examples of these procedures.

1. Plastic containers are avoided for samples which will be analyzed by Electron Capture Detectors due to possible phthalate contamination.
2. Metal containers are avoided since they may contain trace impurities such as oil films, lacquers, or rosin from soldered joints which can interfere with GC testing.
3. Glass bottles/jars with teflon-lined lids or aluminum foil are used as the most generally suitable sample containers.
4. Special preservatives are added during sample collection as necessary to counter effects such as analyte instability and/or possible matrix interactions.
5. Preservatives are added to samples upon collection whenever possible. Addition upon receipt is documented.
6. Trip blanks of laboratory pure water are prepared and sent out with prepared sample containers as an indicator of possible contamination.
7. Surrogates such as a,a,a-Trichlorofluorotoluene are added to UST samples upon analysis in order to confirm proper functioning of the analysis system.

Appendix A summarizes sample container requirements, preservatives, and holding times. Chain of Custody forms are completed for all samples. An example of this form appears in Figure 7.

The Lab Director or his representatives are responsible for the following items on samples:

1. Ensure that appropriate sampling equipment is used.
2. Ensure that appropriate sample containers are used.
3. Ensure that samples are collected, preserved, and transported in a correct and timely manner.
4. Ensure that all sample tracking documentation is correct.

5.0 Sample Receipt/Log-In

Samples submitted to the lab are received by the sample custodian(s) in the sample receiving area. Samples are checked against the Chain of Custody for discrepancies. The Lab Director is contacted to clarify noted discrepancies, clients are contacted directly as necessary, and comments that effect the sample in question are noted on the Chain of Custody remarks section. If no problems are noted with the samples, the custodian(s) signs the Chain of Custody record and logs in the samples.



COMPANY NAME

CONTACT NAME

TELEPHONE NO.

Company's Print Name

Signature

Project No

ANALYSIS

SAMPLE ID

SAMPLE LOCATION

Composts

Glb

Date

Time

Water

Liquid

Oil

Solid

No. of Containers

REMARKS

COMMENTS:

Requested by (Signature)

Requested by (Signature)

Requested by (Signature)

Received by: (Signature)

Received by: (Signature)

Received for laboratory by:

Date

Time

Date

Time

Date

Time

Samples are recorded upon receipt in the Hydrologic LIMS system and identified by assignment of unique sample ID numbers. A DAILY printout of this log stands as a hardcopy record of sample receipt activity of the lab. Samples are processed through the lab using this number.s

A summary of the Laboratory Information Management (LIMS) system follows this section. Completed analytical sample records and associated QA/QC are maintained on computer storage medium such as discs and electronic data files of raw chromatographic runs. Logging functions, sample labels, multiple and custom report formatting, sample status and tracking, and bench sheet generation are some of this system's capabilities. The system is called LABTROL and was developed as a customized LIMS for the Hydrologic group of laboratories. Detailed information on each sample also appears on the Chain of Custody form. This information includes:

1. Sample ID
2. Sample Location (Client ID)
3. Sample Type, Date and Time Collected
4. Number of Containers
5. Analyses Requested
6. Remarks

6.0 Custody in the Laboratory

Once received in the lab, samples are placed in a secure storage area accessed only by lab personnel. Custody is defined by the following criteria. A sample is in the custody of the lab if:

1. It is in the possession of an analyst.
2. It is in the analyst's view after physical possession.
3. The sample was in an analyst's physical possession and was sealed or locked to prevent tampering.
4. If the sample is in a secured area of the lab.

In order to satisfy these custody provisions, the following procedures are implemented by the lab.

1. Samples are stored in a secure area of the lab.
2. Lab access is only through a monitored reception area.
3. Visitors are escorted by lab personnel while in the lab.
4. Samples remain in the secure storage area until removed for sample preparation and subsequent analysis.
5. Custody is the responsibility of an analyst removing a sample from storage.
6. Each analyst must return samples to the secure storage area before the end of the working day.

7.0 Sample Tracking

Samples are tracked through the lab using the unique sample ID number assigned during log-in. This number is affixed to the unique samples.

Associated sample preparation, analysis records, and associated QA/QC documentation is keyed to this ID number.

In summary, this system consists of Chain of Custody information, sample log-in information, computer generated analysis and QA/QC information, instrument operations notebooks, instrument printouts, and final analytical reports. This tracking system ensures valid, defensible, data that is well documented in all stages of generation.

Samples shall be returned to the client for final disposition, unless alternative arrangements (and charges) are made.

8.0 Document Control Procedures

Accountable documents used by the lab include logbooks, Chain of Custody records, bench sheets, data system printouts, and other documents relating to the sample or its analysis. The lab has established the following document control procedures to ensure that documentation is thorough and retrievable.

1. All observations and surrogate recovery results recorded by the lab are entered into permanent lab logbooks organized by parameter.
2. The responsible analyst shall sign and date each page of entry. Entries shall be maintained in chronological order.
3. Instrument run logs are maintained to provide data on run sequences of individual instruments.
4. Entries in logbooks are to be made in black ink.
5. Corrections are made by crossing a single line through the error. All changes are dated and initialed.

Before analytical results are released to the client, the lab assembles and cross-checks information on relevant sample documentation to ensure consistency and data validity. No copies of data, other than confidential file copies, will be generated without approval of the client.

9.0 Data Files

Raw chromatographic data analysis files are archived and stored. LIMS information is also continually updated with new project configurations/definitions. Older sample information is stored on disc or tape cartridge backup.

No sample files or associated documentation are to be destroyed without the direct approval of the Lab Director. Wastewater data is also maintained for a minimum of ten years. Data is annually archived in storage boxes and electronic files packed for long-term storage to facilitate re-access and retrieval if necessary.

10.0 Detection Limits

A detection limit is the smallest concentration for which there is at least a 95% chance that an analyte will be detected as a positive value. Thus, at this level, there is only a 5% chance of obtaining a false negative. The Minimum Detection Limit (MDL) is statistically defined per U.S. EPA analysis methodology.

Frankfort protocols call for a compound reporting limit to be based on the Practical Quantitation Limit (PQL) of a given compound. This level signifies the actual attainable detection/quantitation level achievable by a given analyst using specific instrumentation.

The lab reports are based on determined PQL values for specific compounds. Low end report limits are based on specific equipment operating under approved conditions per methodology. Actual reported detection limits are also matrix and type (water, soil) dependent. These limits will be altered to reflect necessary dilution factors. A table of typical reported PQLs (Table 1) for water samples is included after this section.

TABLE 1 TYPICAL PQLs

Test Name: bna-62	Matrix: water	Method: 625	
Department: ms	Components: 56	Hold Time To Extract: 7d	
Component	DAS	Units	AMDL
Phenol	108-95-2	ug/l	5.0
bis(2-Chloroethyl) ether	111-44-4	ug/l	5.0
2-Chlorophenol	95-57-8	ug/l	5.0
1,3-Dichlorobenzene	541-73-1	ug/l	5.0
1,4-Dichlorobenzene	106-46-7	ug/l	5.0
1,2-Dichlorobenzene	95-50-1	ug/l	5.0
bis(2-Chloroisopropyl) ether	39638-32-9	ug/l	5.0
N-Nitroso-di-n-propylamine	621-64-7	ug/l	5.0
Hexachloroethane	67-72-1	ug/l	5.0
Nitrobenzene	98-95-3	ug/l	5.0
Isophorone	78-59-1	ug/l	5.0
2-Nitrophenol	88-75-5	ug/l	5.0
2,4-Dimethylphenol	105-67-9	ug/l	5.0
bis(2-Chloroethoxy) methane	111-91-1	ug/l	5.0
2,4-Dichlorophenol	120-83-2	ug/l	5.0
1,2,4-Trichlorobenzene	120-82-1	ug/l	5.0
Naphthalene	91-20-3	ug/l	5.0
Hexachlorobutadiene	87-68-3	ug/l	5.0
4-Chloro-3-methylphenol	59-50-7	ug/l	5.0
2,4,6-Trichlorophenol	88-06-2	ug/l	5.0
2-Chloronaphthalene	91-58-7	ug/l	5.0
Dimethylphthalate	131-11-3	ug/l	5.0
Acenaphthylene	208-96-8	ug/l	5.0
Acenaphthene	83-32-9	ug/l	5.0
2,4-Dinitrophenol	51-28-5	ug/l	25.0
4-Nitrophenol	100-02-7	ug/l	25.0
2,4-Dinitrotoluene	121-14-2	ug/l	5.0
2,6-Dinitrotoluene	606-20-2	ug/l	5.0
Diethylphthalate	84-66-2	ug/l	5.0
4-Chlorophenyl-phenylether	7005-72-3	ug/l	5.0
Fluorene	86-73-7	ug/l	5.0
4,6-Dinitro-2-methylphenol	534-52-1	ug/l	25.0
4-Bromophenyl-phenylether	101-55-3	ug/l	5.0
Hexachlorobenzene	118-74-1	ug/l	5.0
Pentachlorophenol	87-86-5	ug/l	25.0
Phenanthrene	85-01-8	ug/l	5.0
Anthracene	120-12-7	ug/l	5.0
Di-n-butylphthalate	84-74-2	ug/l	5.0
Fluoranthene	206-44-0	ug/l	5.0
Pyrene	129-00-0	ug/l	5.0
Butylbenzylphthalate	85-68-7	ug/l	5.0
3,3-Dichlorobenzidine	91-94-1	ug/l	10.0
Benzo(a)anthracene	56-55-3	ug/l	5.0
bis(2-Ethylhexyl)phthalate	117-81-7	ug/l	5.0
Chrysene	218-01-9	ug/l	5.0

Di-n-octylphthalate	117-84-0	ug/l	5.0
Benzo(b)fluoranthene	205-99-2	ug/l	5.0
Benzo(k)fluoranthene	207-08-9	ug/l	5.0
Benzo(a)pyrene	50-32-8	ug/l	5.0
Indeno(1,2,3-cd)pyrene	193-39-5	ug/l	5.0
Dibenz(a,h)anthracene	53-70-3	ug/l	5.0
Benzo(g,h,i)perylene	191-24-2	ug/l	5.0
Hexachlorocyclopentadiene	77-47-4	ug/l	10.0
N-Nitrosodiphenylamine	86-30-6	ug/l	10.0
Benzidine	92-87-5	ug/l	80.0
N-Nitrosodimethylamine	62-75-9	ug/l	10.0

Component	CAS	Units	AMDL
Chloromethane	74-87-3	ug/l	5.0
Bromomethane	74-83-9	ug/l	5.0
Vinyl Chloride	75-01-4	ug/l	5.0
Chloroethane	75-00-3	ug/l	5.0
Methylene Chloride	75-09-2	ug/l	5.0
Trichlorofluoromethane	75-69-4	ug/l	5.0
1,1-Dichloroethene	75-35-4	ug/l	1.0
1,1-Dichloroethane	75-34-3	ug/l	1.0
trans-1,2-Dichloroethene	156-60-5	ug/l	2.0
Chloroform	67-66-3	ug/l	1.0
1,2-Dichloroethane	107-06-2	ug/l	1.0
1,1,1-Trichloroethane	71-55-6	ug/l	1.0
Carbon Tetrachloride	56-23-5	ug/l	1.0
Bromodichloromethane	75-27-4	ug/l	1.0
1,2-Dichloropropane	78-87-5	ug/l	1.0
cis-1,3-dichloropropene	10061-01-5	ug/l	5.0
Trichloroethene	79-01-6	ug/l	1.0
Dibromochloromethane	124-48-1	ug/l	1.0
1,1,2-Trichloroethane	79-00-5	ug/l	1.0
Benzene	71-43-2	ug/l	1.0
trans-1,3-Dichloropropene	10061-02-6	ug/l	5.0
2-Chloroethyl vinyl ether	110-75-8	ug/l	5.0
Bromoform	75-25-2	ug/l	1.0
Tetrachloroethene	127-18-4	ug/l	1.0
1,1,2,2-Tetrachloroethane	79-34-5	ug/l	1.0
Toluene	108-88-3	ug/l	1.0
Chlorobenzene	108-90-7	ug/l	1.0
Ethylbenzene	100-41-4	ug/l	1.0
1,2-Dichlorobenzene	95-50-1	ug/l	5.0
1,3-Dichlorobenzene	541-73-1	ug/l	5.0
1,4-Dichlorobenzene	106-46-7	ug/l	5.0

Component	CAS	Units	AMDL
alpha-BHC	319-84-6	ug/l	0.05
beta-BHC	319-85-7	ug/l	0.05
delta-BHC	319-86-8	ug/l	0.05
gamma-BHC (Lindane)	58-89-9	ug/l	0.05
Heptachlor	76-44-8	ug/l	0.05
Aldrin	309-00-2	ug/l	0.05
Heptachlor epoxide	1024-57-3	ug/l	0.05
Endosulfan I	959-98-8	ug/l	0.05
Dieldrin	60-57-1	ug/l	0.1
4,4'-DDE	72-55-9	ug/l	0.1
Endrin	72-20-8	ug/l	0.1
Endosulfan II	33213-65-9	ug/l	0.1
4,4'-DDD	72-54-8	ug/l	0.1
Endosulfan sulfate	1031-07-8	ug/l	0.1
4,4'-DDT	50-29-3	ug/l	0.1
Endrin Ketone	53494-70-5	ug/l	0.1
Chlordane	57-74-9	ug/l	0.5
Toxaphene	8001-35-2	ug/l	1.0
Endrin aldehyde	7421-93-4	ug/l	0.1
Aroclor-1016	12674-11-2	ug/l	0.5
Aroclor-1221	11104-28-2	ug/l	0.5
Aroclor-1232	11141-16-5	ug/l	0.5
Aroclor-1242	53469-21-9	ug/l	0.5
Aroclor-1248	12672-29-6	ug/l	0.5
Aroclor-1254	11097-69-1	ug/l	1.0
Aroclor-1260	11096-82-5	ug/l	1.0

Component	CAS	Units	AMDL
2,4-D	94-75-7	ug/l	10.0
2,4,5-TP (Silvex)	93-72-1	ug/l	2.0
2,4,5-T	93-76-5	ug/l	2.0

Component	CAS	Units	AMDL
Bromodichloromethane	75-27-4	ug/l	1.0
Bromoform	75-25-2	ug/l	1.0
Bromomethane	74-83-9	ug/l	1.0
Carbon Tetrachloride	56-23-5	ug/l	1.0
Chlorobenzene	108-90-7	ug/l	1.0
Chloroethane	75-00-3	ug/l	1.0
2-Chloro Ethyl Vinyl Ether	110-75-8	ug/l	1.0
Chloroform	67-66-3	ug/l	1.0
Chloromethane	74-87-3	ug/l	1.0

Dibromochloromethane	124-48-1	ug/l	1.0
1,2-Dichlorobenzene	95-50-1	ug/l	1.0
1,3-Dichlorobenzene	541-73-1	ug/l	1.0
1,4-Dichlorobenzene	106-46-7	ug/l	1.0
Dichlorofluoromethane	75-43-4	ug/l	1.0
1,1-Dichloroethane	75-34-3	ug/l	1.0
1,2-Dichloroethane	107-06-2	ug/l	1.0
1,1-Dichloroethene	75-35-4	ug/l	1.0
trans-1,2-Dichloroethene	156-60-5	ug/l	1.0
1,2-Dichloropropane	78-87-5	ug/l	1.0
cis-1,3-Dichloropropene	10061-01-5	ug/l	1.0
trans-1,3-Dichloropropene	10061-02-6	ug/l	1.0
Methylene Chloride	75-09-2	ug/l	1.0
1,1,2,2-Tetrachloroethane	79-34-5	ug/l	1.0
Tetrachloroethene	127-18-4	ug/l	1.0
1,1,1-Trichloroethane	71-55-6	ug/l	1.0
1,1,2-Trichloroethane	79-00-5	ug/l	1.0
Trichloroethene	79-01-6	ug/l	1.0
Trichlorofluoromethane	75-69-4	ug/l	1.0
Vinyl Chloride	75-01-4	ug/l	1.0

Test Name: 602-full	Matrix: water	Method: EPA 602	
Department: gc	Components: 8	Hold Time: 14d	
<u>Component</u>	<u>CAS</u>	<u>Units</u>	<u>AMDL</u>
Benzene	71-43-2	ug/l	1.0
Chlorobenzene	108-90-7	ug/l	1.0
1,2-Dichlorobenzene	95-50-1	ug/l	1.0
1,3-Dichlorobenzene	541-73-1	ug/l	1.0
1,4-Dichlorobenzene	106-46-7	ug/l	1.0
Ethylbenzene	100-41-4	ug/l	1.0
Toluene	108-88-3	ug/l	1.0
Xylene (Total)	1330-20-7	ug/l	1.0

11.0 Significant Figures

A uniform method of rounding and reporting numbers has been established by EPA. These criteria appear in the EPA HANDBOOK OF ANALYTICAL QUALITY CONTROL IN WATER AND WASTEWATER LABORATORIES, EPA-600/4-79-019. An excerpt from this document appears in Appendix B of this manual. This method shall be utilized unless otherwise specified in sample documentation.

12.0 General Laboratory Practice

Many analytical procedures performed by the lab are specific to a particular analysis type or instrument. Whenever possible the analytical methods used by the lab are from agencies such as the U.S. Environmental Protection Agency (USEPA), American Public Health Association (APHA), American Society for Testing and Materials (ASTM), the National Institute for Occupational Safety and Health (NIOSH), and Standard Methods.

Only approved methodology shall be used for the generation of regulatory data as listed in section 40 CFR of the Federal Register and its updates.

Analysts shall implement new procedures only after thorough testing in the lab using standards, spikes, and duplicates as controls. Once the procedure is adequately defined and statistically in control, the method may be actually used for sample analysis in the lab.

The following sections will describe general procedures such as calibration, preventive maintenance, supply control, safety, and training.

13.0 Calibration Standards

Internal and Surrogate standards are obtained from reputable chemical suppliers with certification of purity and concentration. These commercial materials are used as stock standards. Working concentrations are made from stocks to cover the linear range of specific calibration curves. Calibration check standards from different vendor sources are used as a QA measure.

All standard preparation documentation is kept in a Standards Notebook. The following information is defined for each compound or mixture of compounds:

1. Standard Number/Source/Lot Number
2. Preparation date
3. Name of each compound and aliquot used
4. Standard Concentration
5. Final Concentration
6. Solvent used
7. Final volume
8. Preparer

Standard containers are labeled with the standard number. All stock standards are checked for expiration dates and response compared to that attained on previous materials as a QA measure.

14.0 Calibration Procedures

The following general calibration procedures are routinely performed by the lab. Specific calibration procedures for a particular instrument are detailed in each analytical method. Periodic calibrations are performed on equipment such as balances, thermometers, and waterbaths as detailed below. All calibration measurements are recorded in logbooks.

1. Analytical balances are checked daily by use of Class S weights and calibrations recorded.
2. The balance is serviced and calibrated under service contract on a yearly basis.
3. The temperature of water baths, ovens, and refrigerators is checked daily and recorded.
4. Thermometers are calibrated yearly against a certified NBS thermometer.
5. pH meters are calibrated daily using manufacture's instructions and readings of buffers recorded.
6. Analytical Balance is calibration-checked daily using a Class S or equivalent certified weight.

15.0 Preventive Maintenance Procedures

The manufacture's instrument manual serves as guidance of preventive maintenance such as lubrication, detector and source cleaning, and their frequency. Chromatographic injector septa, carrier gas purification traps, and injector liners are replaced on a regular basis to maintain proper instrument response. Trends in sample and QA data are used to spot instrument response reduction or malfunction. Maintenance must be performed by qualified personnel when instrument performance indicates degradation of the analytical system.

Symptoms of this degradation include the following:

1. Degradation of peak resolution
2. Degradation of compound response
3. Shift in calibration curves
4. Decreased sensitivity
5. Failure to meet defined QC criteria

Preventive Maintenance Logbooks are maintained on each analytical instrument. These logbooks contain detailed schedules for routine service functions. They also serve as a service history of the instrument.

Instrument downtime is minimized by maintaining adequate supplies of expendable items such as gas tanks, line filters, septa, syringes, GC columns, ferrules, computer paper, printer heads/ribbons, etc.

16.0 Hood Air Flow

The minimum allowable air face velocity for hoods is 100 cfm. Each hood is marked for the allowable sash opening. Adequacy of this flow is determined at time of installation and verified by flagging the air flow constantly. This ensures proper air movement at each hood and ensures proper personnel protection from fume exposure. The air face velocity is monitored once per month.

17.0 Equipment Maintenance

Maintenance and repair for lab instrumentality is documented in a looseleaf Equipment Maintenance Logbook. This book establishes records of equipment problems and aids in the recognition of consistent problems. It also gives a general indication of expected analytical performance.

The following items must be completed on the form in the Equipment/Maintenance Logbook:

1. Manufacturer: Enter the name of the manufacturer of the particular equipment item
2. Model Number: Enter specific model number of the item
3. Serial Number: Enter the serial number of the item.
4. Date: Enter the date of the problem or action.
5. Type of Service: Enter a brief description of the problem along with your initials.
6. Service Action/Report: Detail the corrective action required to restore the item to normal operating conditions.
7. Initials: Analysts should initialize the repair as completed by the lab or service personnel.

The records of the Equipment Maintenance Logbook will provide valuable information on the durability of the equipment, lab personnel capabilities, trends in equipment operation, and the evaluation of the manufacturer and the effectiveness of their service personnel. The equipment maintenance records are maintained in the logbook concurrently with the preventive maintenance records in the same log book.

18.0 Balance Calibration

An analytical balance is used to prepare reference solutions for stock or pure chemicals. All analytical results are based on the accuracy of the prepared reference solutions. Therefore the analytical balance must be properly maintained and operated. The following procedures are used to ensure that this is achieved:

1. Accuracy, Precision, and Sensitivity are checked yearly by a qualified balance specialist.
2. The balance is used on a heavy shock-proof table or counter.
3. Balance is located away from heavy lab traffic and drafts.
4. The inside and outside of the balance is kept clean.
5. Class S Weights or their equivalents are used to verify performance on a daily basis.

The manufacture's literature should be consulted by all first-time users of the balance. If deviations from accepted values occurs on Class S Weights, notify the Lab Director immediately. A visit by a qualified balance specialist may be required.

19.0 Thermometer Calibration

All thermometers used in the lab are calibrated when purchased and yearly thereafter. The thermometers are calibrated against NBS reference thermometer in the operational range of the device. Temperature calibration tags are placed on the thermometer at the time of calibration.

20.0 Supply Quality Control

The production of quality analytical data is based on many factors. Essential to this is the specification of the chemical standards, equipment, and reagents used by the lab. Materials or equipment not meeting requirements of approved analytical methodology are not considered for use by the lab. Purchase decisions must be approved by the Lab Director or his representative to ensure this.

Lab reagents are poured from stock bottles in measured amounts for immediate use. Remaining or excess solution is not to be poured back into stock containers due to possible contamination.

All lab supplies shall be of required quality to ensure proper instrument calibration and performance. Many approved analytical methods detail the type and grade of acceptable materials, and these guidelines must be followed.

All reagents used by the lab are prepared from Analytical Reagent Grade (AR) chemicals or higher. The preparation of all reagents is documented for source, weight, and dilution. Reagents are clearly labeled with the composition, concentration, date prepared, preparer's initials, expiration date, any special storage requirements, date received, and date opened for use by the lab.

Reagents are stored in appropriate containers under conditions necessary to maintain their chemical integrity. Reagents are discarded after they have exceeded their shelf life.

Dry reagents such as glass wool, sodium sulfate, alumina, and silica gel are either heated to 400 degrees C or solvent extracted prior to use for organic chemical analysis.

Organic free water for the lab is purchased commercially. Analysis of blanks provides continuing quality checks.

Pesticide Grade organic solvents are used by the lab for organic testing. Solvents and reagents are checked for contamination using reagent blanks before they are used for actual samples.

All Quality Control Reference Materials (QCRMs) are acquired from reputable vendor sources or sources commonly used by the U.S. EPA. All organic standard materials and working standards are detailed in the Standards Logbook. These preparations are identified by unique ID numbers, and labeled for reference. A single page in this logbook should be used for single standards that are a mixture of other components. Several working standards may appear on a single page if they result from serial dilutions of only one standard.

Gases used by the laboratory are to be equal or superior in quality to those described in approved analytical methodology. Extra tanks of primarily used gases shall be maintained in storage by the lab.

Gas dryers are used to prevent moisture contamination of GC and GC/MS equipment. Drying chemicals must be routinely replaced. Glassware used for analytical testing shall be Class A.

21.0 Analytical Procedures

The lab uses approved methods from the U.S. EPA, APHA, SM, ASTM, and NIOSH as appropriate to the analysis of environmental samples. Prior to lab implementation, new procedures are tested using blanks, spikes, and duplicates as controls. Once properly understood by the analyst and quality data production is achieved, the method is documented and approved for use in the lab by the Lab Director. These analytical procedures are reviewed and updated periodically as needed. The methods routinely used by the lab are selected from the publications listed in Table 6.

TABLE 2

ROUTINELY USED METHODS

1. Annual Book of ASTM Standards Part 31-Water. ASTM. 1981. American Society for Testing and Materials. Philadelphia, PA 19103.
2. Collection and Analysis of Purgeable Organics Emitted from Wastewater Treatment Plants. U.S. EPA, Cincinnati, Ohio, 1980. Publication No. EPA-600/2-80-017.
3. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; U.S. EPA, 1984, Federal Register, 40CFR Part 136.
4. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, U.S. EPA 1977. Publication No. EPA-600/4-79-019.
5. Handbook for Sampling and Sample Preservation of Water and Wastewater. U.S. EPA, Cincinnati, Ohio 1982. Publication No. EPA-600/4-82-029.
6. Laboratory Protocols for Evaluating the Fate of Organic Chemicals in Air and Water, U.S. EPA, Athens, GA, 1982, Publication No. EPA-600/3-82-022.
7. Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, U.S. EPA, Research Triangle Park, N.C., 1980, Publication No. EPA-600/8-80-038.
8. Manual of Analytical Quality Control for Pesticides and Related Compounds, U.S. EPA, Research Triangle Park, N.C., 1979, Publication No. EPA-600/1-79-008.
9. Manual of Chemical Methods for Pesticides and Devices, U.S. EPA, Office of Pesticide Programs, Beltsville, Maryland, 1982.
10. Methods of Analysis of the Association of Official Analytical Chemist, 14th Edition, 1985.
11. Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Cincinnati, Ohio, 1979, Publication No. EPA-600/4-79-020.
12. Methods for Organic Chemical Analysis for Municipal and Industrial Wastewater. U.S. EPA, Cincinnati, Ohio, 1982. Publication No. EPA-600/4-82-057.
13. NIOSH Manuals of Analytical Methods, Vols. 1-7, National Institute for Occupational Safety and Health, Cincinnati, Ohio 1981.

TABLE (Continued)

14. Procedures for Handling and Chemical Analysis of Sediment and Water Samples, U.S. EPA, 1981, Technical Report EPA/CE-81-1.9

15. Standard Methods for the Examination of Water and Wastewater, Water Pollution Control Federation, 16th Edition, 1985.

16. Test Methods for Evaluating Solid Waste-Physical/Chemical Methods. Office of Solid Waste, U.S. EPA, Washington, DC, SW-846, 3rd Edition, 1986.

17. The Analysis of Polychlorinated Biphenyls in Transformer Fluid and Waste Oil, U.S. EPA/EMSL, Cincinnati, Ohio.

HYDROLOGIC

DATA EVALUATION CHECKLIST

CLIENT: _____

SAMPLE ID NUMBERS: _____

CONFIRMATION OF CHAIN-OF-CUSTODY YES NO INITIALS _____

ASSIGNED LOG NUMBERS CHECK YES NO INITIALS _____

NUMERICAL TRANSCRIPTION/UNITS _____

HISTORICAL DATA CHECK _____

INTERNAL REPORT PARAMETER LOGIC: _____

PERMIT LIMITS: AVAILABLE NOT AVAILABLE CHECK OF LIMITS

INITIALS _____

REFERRED TO: _____

REASON: _____

SIGNATURE _____

32.0 Data Reports

Format and content of final data reports are dependent upon project requirements. These requirements determine the need for explanatory text, or agency required reporting formats.

PQLs are reported if the detected value is less than the detection limit established by the lab. Data are generally reported in a tabular format available in the LIMS. Footnotes are referenced to specific data if explanations of reported data are required.

All reports are signed by the Laboratory Director, QA/QC Officer, or their designated representative.

Reported data from subcontracted labs is reported on laboratory reports along with the Frankfort Lab ID Number of the lab completing the testing. Only reputable labs are used for these services.

33.0 Quality Assurance/Quality Control Officer

The QA/QC Officer provides routine reports to management regarding lab operations, performance of measurement systems, and data quality. Associated document copies or raw data are incorporated into these records as necessary.

Reports should cover results of interlaboratory and intralaboratory precision/accuracy studies, results of system and performance audits, and data quality assessments.

Any significant problems affecting data quality must immediately be brought to the attention of the Lab Director. Appropriate corrective action shall be implemented and evaluated for its effectiveness in solving the problem.

The QA/QC Officer must ensure that any problem requiring corrective action is fully resolved. This is documented using a corrective action form (Figure 11). This form indicates the problem, the cause, and the corrective action necessary to solve the problem. The following procedures are noted in this evaluation system:

1. Identify the problem.
2. Assign investigative responsibility.
3. Determine the cause of the problem.
4. Determine necessary corrective action.
5. Implement the corrective action.
6. Verify elimination of the problem.

Documentation of this action is maintained on file in the lab.

34.0 Manual Review

This QA/QC Manual and other SOP Manuals are periodically reviewed by upper management and the QA/QC Officer. Manuals are updated as necessary.

Revisions are distributed to all affected personnel and documentation is secured to show that revisions are understood and implemented.

HYDROLOGIC-KENTUCKY
LABORATORY DEVIATION FORM
HYDROLOGIC-KENTUCKY ID
DATE COLLECTED
CLIENT
SAMPLE MATRIX
FORM INITIATED BY

PROBLEM DESCRIPTION/SUSPECT PARAMETERS:

SUMMARY OF DATA REVIEW/ANALYST INQUIRY

FINAL DATA STATUS (A/R, REASONS)

RESULTING ACTION

APPROVED BY:
DATE:

APPENDIX A



Containers, Preservation, Hold Times

Parameters	Recommended Volume (ml)	Containers	Preservative	Holding Time
Physical Properties				
Color	125	P, G	Cool, 4° C	48 hours
Conductance	125	P, G	Cool, 4° C	28 days
Hardness	125	P, G	HNO ₃ to pH < 2	6 months
Odor	200	G only	Cool, 4° C	24 hours
pH	50	P, G	None	Immediately
Residue, Filterable	250	P, G	Cool, 4° C	7 days
Residue, Nonfilterable	250	P, G	Cool, 4° C	7 days
Residue, Total	250	P, G	Cool, 4° C	7 days
Residue, Volatile	250	P, G	Cool, 4° C	7 days
Settleable Matter	1000	P, G	Cool, 4° C	48 hours
Temperature	1000	P, G	None	Immediately
Turbidity	100	P, G	Cool, 4° C	48 hours
Metals				
Dissolved	250	P, G	Filter on site HNO ₃ to pH < 2	6 months
Total	500	P, G	HNO ₃ to pH < 2	6 months
Hexavalent Chromium	250	P, G	Cool, 4° C	24 hours
Mercury, Dissolved	250	P, G	Filter on site	28 days
Mercury, Total	250	P, G	HNO ₃ to pH < 2 HNO ₃ to pH < 2	28 days



Parameters	Recommended Volume (ml)	Containers	Preservative	Holding Time
Inorganic Non-metallic				
Acidity	250	P, G	Cool, 4° C	14 days
Alkalinity	250	P, G	Cool, 4° C	14 days
Bicarbonate	100	P, G	Cool, 4° C	14 days
Bromide	250	P, G	None	28 days
Carbonate	100	P, G	Cool, 4° C	14 days
Chloride	125	P, G	None	28 days
Residual Chlorine	500	P, G	None	Immediately
Coliform, Fecal	100	Ster. P, G	Ster. P, G	6 hours
Coliform, Total	100	Ster. P, G	Ster. P, G	6 hours
Corrosivity (Langlier Index)	350	P, G	Cool, 4° C	Immediately
Cyanides (5,6)	1000	P, G	Cool 4° C, NaOH to pH > 12, 0.6g ascorbic acid	14 days
Fluoride	500	P only	None	28 days
Iodide	125	P, G	Cool, 4° C	24 hours
Nitrogen, Ammonia or Ammonia (as N)	1000	P, G	Cool, 4° C H ₂ SO ₄ to pH < 2	28 days
Nitrogen, Total Kjeldahl	1000	P, G	Cool, 4° C H ₂ SO ₄ to pH < 2	28 days
Nitrate plus Nitrite	250	P, G	Cool, 4° C H ₂ SO ₄ to pH < 2	28 days
Nitrate (as N)	250	P, G	Cool, 4° C	48 hours
Nitrite (as N)	125	P, G	Cool, 4° C	48 hours
Dissolved Oxygen	300	G only	None	Immediately
Phosphorus Ortho-phosphate	125	P, G	Filter on site Cool, 4° C	48 hours
Phosphorus, Total	125	P, G	Cool, 4° C 2 H ₂ SO ₄ to pH < 2	28 days
Phosphorus, Total, Dissolved	125	P, G	Filter on site H ₂ SO ₄ to pH < 2	28 days
Resistivity	100	P, G	Cool, 4° C	28 days
Silica (Silicon Dioxide)	125	P only	Cool, 4° C	28 days
Sulfate	125	P, G	Cool, 4° C	28 days
Sulfide	1000	P, G	Cool, 4° C, 2ml Zn Acetate, NaOH to > 9	7 days
Sulfite	125	P, G	None	Immediately



Parameters	Recommended Volume (ml)	Container	Preservative	Hold
Organics				
BOD	1000	P, G	Cool, 4° C	48 hours
COD	125	P, G	Cool, 4° C	28 days
Oil and Grease	2 x 1000	G only	H ₂ SO ₄ to pH < 2 Cool, 4° C	28 days
Phenols (Phenolics)	250	G only	H ₂ SO ₄ to pH < 2 Cool, 4° C	28 days
MBAS (Surfactants)	500	P, G	Cool, 4° C	48 hours
Total Organic Carbon	125	G only	H ₂ SO ₄ to pH < 2	28 days
Total Recoverable Pet. Hydrocarbons	2 x 1000	G only	H ₂ SO ₄ to pH 2	14 days
Volatiles, (Purgeable)	2 x 40	G only	Cool, 4° C	14 days
Semi-Volatiles	2500	G only	Cool, 4° C	7 days for ex 40 days for a
Pesticides & PCBs	2500	G only	Cool, 4° C	7 days for ex 40 days for a
Total Organic Halides	250	G (Amber)	Cool, 4° C	7 days
Purgeable Halocarbons	2 x 40	G, Teflon lined septum	H ₂ SO ₄ to pH < 2 Cool, 4° C 0.008%	14 days
Purgeable Aromatics	2 x 40	G, Teflon lined septum	Na ₂ S ₂ O ₃ ⁶ Cool, 4° C 0.008%	14 days
Acrolein and Acrylonitrile	2 x 40	G, Teflon lined septum	Na ₂ S ₂ O ₃ ⁶ HCl to pH < 2 Cool, 4° C 0.008%	14 days
Phthalate Esters	2500	G, Teflon lined cap	Adjust pH to 4-5 Cool, 4° C	7 days for extr 40 days to ana
PCBs	2500	G, Teflon lined cap	Cool, 4° C	7 days for extr 40 days to ana

APPENDIX B



Parameters	Recommended Volume (ml)	Container	Preservative	Holding Time
Organics (continued)				
Chlorinated Herbicides	2500	G, Teflon lined cap	Cool, 4° C	7 days for extr 40 days for a
Chlorinated Hydrocarbons	2500	G, Teflon lined cap	Cool, 4° C	7 days for extr. 40 days for an
TCDD	2500	G, Teflon lined cap	Cool, 4° C 0.008% Na ₂ S ₂ O ₃	7 days for extra 40 days for an
Radiological Tests				
Alpha, beta and radium	2500	P, G	HNO ₃ to pH < 2	6 months

DATA HANDLING AND REPORTING

Introduction

To obtain meaningful data on water quality, the sample collector must obtain representative sample and then deliver it unchanged for analysis. The analyst must perform the proper analysis in the prescribed fashion, complete calculations, and convert results to final form for permanent recording of the analytical data in meaningful, exact terms. These results are transferred to a storage facility for future interpretation and use.

The following sections discuss processing of actual values, recording and reporting of data in the proper way, some means of quality control of data, and the storage and retrieval of data.

The Analytical Value

Significant Figures

The term "significant figure" is used, sometimes rather loosely, to describe a judgment of the reportable digits in a result. When the judgment is not soundly based, meaningful digits are lost or meaningless digits are reported. On the other hand, proper use of significant figures gives an indication of the reliability of the analytical method used.

The following discussion describes the process of retention of significant figures.

A number is an expression of quantity. A figure or digit is any of the characters 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, which, alone or in combination, serve to express a number. A significant figure is a digit that denotes the amount of the quantity in the particular decimal place in which it stands. Reported analytical values should contain only significant figures. A value is made up of significant figures when it contains all digits known to be true and one last digit in doubt. For example, if a value is reported as 18.8 mg/l, the 18 must be firm while the 0.8 is somewhat uncertain, but presumably better than one of the values 0.7 or 0.9 would be.

The number zero may or may not be a significant figure depending on the situation.

Final zeros after a decimal point are always meant to be significant figures. For example, to the nearest milligram, 9.8 g is reported as 9.800 g.

Zeros before a decimal point with nonzero digits preceding them are significant. With no preceding nonzero digit, a zero before the decimal point is not significant.

If there are no nonzero digits preceding a decimal point, the zeros after the decimal point but preceding other nonzero digits are not significant. These zeros only indicate the position of the decimal point.

Final zeros in a whole number may or may not be significant. In a conductivity measurement of 1,000 $\mu\text{mho/cm}$, there is no implication by convention that the conductivity is $1,000 = 1 \mu\text{mho}$. Rather, the zeros only indicate the magnitude of the number.

When one number is subtracted from another, rounding off should be completed after the subtraction operation, to avoid possible invalidation of the operation.

When two numbers are to be multiplied, all digits are carried through the operation, then the product is rounded off to the number of significant digits of the multiplier with the fewer significant digits.

When two numbers are to be divided, the division is carried out on the two numbers using all digits. Then the quotient is rounded off to the number of significant digits of the divisor or dividend, whichever has the fewer.

When a number contains n significant digits, its root can be relied on for n digits, but its power can rarely be relied on for n digits.

: Rounding Off the Results of a Series of Arithmetic Operations

The preceding rules for rounding off are reasonable for most calculations; however, when dealing with two nearly equal numbers, there is a danger of loss of all significance when applied to a series of computations that rely on a relatively small difference in two values. Examples are calculation of variance and standard deviation. The recommended procedure is to carry several extra figures through the calculations and then to round off the final answer to the proper number of significant figures.

Glossary of Statistical Terms

To clarify the meanings of statistical reports and evaluations of water quality data, the following statistical terms are introduced. They are derived in part from usage (1.2) of the American Society for Quality Control.

Accuracy—The difference between an average value and the true value when the latter is known or assumed.

Arithmetic mean—The arithmetic mean (or average) of a set of n values is the sum of the values divided by n :

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n}$$

Bias—A systematic error due to the experimental method that causes the measured values to deviate from the true value.

Confidence limit, 95 percent—The limits of the range of analytical values within which a single analysis will be included 95 percent of the time.

$$95 \text{ percent CL} = \bar{X} \pm 1.96S$$

where CL is the confidence level and S is the estimate of the standard deviation.

A good measure of the significance of one or more zeros interspersed in a number is to determine whether the zeros can be dropped by expressing the number in exponential form. If they can, the zeros may not be significant. For example, no zeros can be dropped when expressing a weight of 100.08 g in exponential form; therefore the zeros are significant. However, a weight of 0.0008 g can be expressed in exponential form as 8×10^{-4} g, so the zeros are not significant. Significant figures reflect the limits in accuracy of the particular method of analysis. It must be decided whether the number of significant digits obtained for resulting values is sufficient for interpretation purposes. If not, there is little that can be done within the limits of the given laboratory operations to improve these values. If more significant figures are needed, a further improvement in method or selection of another method will be required.

Once the number of significant figures obtainable from a type of analysis is established, data resulting from such analyses are reduced according to set rules for rounding off.

Rounding Off Numbers

Rounding off of numbers is a necessary operation in all analytical areas. It is automatically applied by the limits of measurement of every instrument and all glassware. However, when it is applied in chemical calculations incorrectly or prematurely, it can adversely affect the final results. Rounding off should be applied only as described in the following sections.

Rounding-Off Rules

If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example, 11.443 is rounded off to 11.44.

If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45.

If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42.

Rounding Off Arithmetic Operations

When a series of numbers is added, the sum should be rounded off to the same number of decimal places as the addend with the smallest number of places. However, the operation is completed with all decimal places intact, and rounding off is done afterward. As an example,

$$\begin{array}{r} 11.1 \\ 11.12 \\ +11.13 \\ \hline 33.35 \end{array}$$

The sum must be rounded off to 33.4.



Series—A number of test results with common properties that identify them uniquely.

Skewness—A measure of the asymmetry of a frequency distribution.

$$K = \frac{\sum (X_i - \bar{X})^3}{n\sigma^3}$$

This measure is a pure signed number. If the data are perfectly symmetrical, the skewness is zero. If K is negative, the long tail of the distribution is to the left. If K is positive, the long tail extends to the right.

Standard deviation—The square root of the variance of the universe.

$$\sigma = \sqrt{\frac{\sum_{i=1}^N X_i^2 - \left(\sum_{i=1}^N X_i\right)^2 / N}{N}}$$

Standard deviation estimate—The most widely used measure to describe the dispersion of a set of data. Normally $\bar{X} \pm S$ will include 68 percent, and $\bar{X} \pm 2S$ will include about 95 percent of the data from a study:

$$S = \sqrt{\frac{\sum_{i=1}^n X_i^2 - \left(\sum_{i=1}^n X_i\right)^2 / n}{n - 1}}$$

Standard deviation, single analyst—A measure of dispersion for data from a single analyst that is calculated here using an equation developed by Youden for his nonreplicate study design (3).

$$S_r = \sqrt{\frac{\sum_{i=1}^n (D_i - \bar{D})^2}{2(n - 1)}}$$

where D = the difference in paired values obtained from a single analyst.

Universe—The total set of items or measurements.

Variable—An experimentally determined estimate denoted X for a particular quality or trait of the population.

Quality control:

Those samples for wastewater nitrate only should be chilled to 4°C at the time of collection as preservation and analyzed within 48 hours of collection.

All other analysis of which nitrate is a part should be preserved by chilling to 4°C and adding H₂SO₄ to pH < 2 at the time of collection. The sample must be analyzed within 28 days of collection.

Duplicate analysis must be run at a rate of not less than 10%.

For each analytical session the 1.0 ug, 4.0 ug and 10.0 ug standard must be run as a minimum requirement. Upon comparison to the standard curve agreement must be within ± 20% of the standard's value.

A standard curve must be prepared by each analyst prior to running samples. The curves must be updated no less than once per quarter.

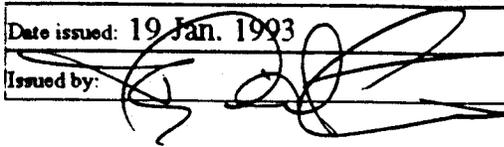
A performance standard of known concentration is analyzed during each analytical session. The value determined should be within ±20% of the vendor's published value.

Sample preservation is verified at log in.

Standards and reagents preparation are documented in the benchsheet notebook.

HYDROLOGIC, INC.

Analytical Procedure

Title: Nitrite Nitrogen	
Date issued: 19 Jan. 1993	Supersedes:
Issued by: 	

Safety: Safety glasses
Lab coat

Equipment: Spectrophotometer
Pipets (various sizes)
Tripour beakers
Cuvets
Volumetric flasks 50 ml

Reagents: Nitrite standard Ricca Cat.# 5460
Sulfanilamide Reagent Ricca Cat.# 8103
Naphthylethylenediamine dihydrochloride Ricca Cat.# 5230

Procedure: Standard Curve
Dilute stock nitrite standard by pipeting 2.0 ml to a 500 ml volumetric flask and diluting to volume with distilled water. The solution is 1 ug/ml N(NO₂).
Prepare working standards by pipeting appropriate aliquotes of the 1 ug/ml N(NO₂) standard into a 50 ml volumetric flask and diluting to volume. The resulting standards should be 0.5, 1.0, 1.5, 2.0, 3.0, 4.0, 5.0, and 10.0 ug N(NO₂).
Transfer each standard to a 150 ml beaker and add 1 ml of sulfanilamide to each standard, mix, and let stand for 8 minutes. Then add 1 ml of naphthylethylenediamine dihydrochloride reagent to each solution, mix and let stand a minimum of 8 minutes.
Determine the absorbance of each standard at 540 nm and record the standard concentration and absorbance on the benchsheet. The absorbance determination must be performed within 2 hours of the addition of the last reagent. A blank is prepared using 50 ml of deionized water and carried through the above outlined reagent additions. The blank should be used to set the baseline for the analysis.
Prepare a standard curve by plotting ug N(NO₂) against absorbance. Store curve in appropriate benchsheet notebook.

Sample determination:

Place 50.0 ml of sample or an aliquote diluted to 50 ml into a 150 ml beaker. Record sample volume on the benchsheet.

To each sample add 1 ml of sulfanilamide reagent, mix and let stand for 8 minutes.

Then add 1 ml of naphthylethylenediamine dihydrochloride to each sample, mix and let stand a minimum of 8 minutes.

Determine the absorbance of each sample at 540nm. The absorbance determination must be performed within 2 hours of the addition of the final reagent. Record absorbance on the benchsheet.

Determine the $N(NO_2)$ content in the sample by comparing the absorbance found against the standard curve. Record value found on the benchsheet in micrograms $N(NO_2)$.

Calculation:

$$\text{ppm } N(NO_2) = \frac{\text{ug } N(NO_2) \text{ found}}{\text{sample vol. (ml)}}$$

Quality Control:

Those sample for nitrite only should be chilled to 4°C at the time of collection and analyzed within 48 hours of collection.

All other analysis of which nitrite is a part should be preserved by chilling to 4°C and adding H_2SO_4 to pH < 2 at the time of collection. The sample must be analyzed within 28 days of collection.

Duplicate analysis must be run at a rate of not less than 10%.

Each analyst must prepare a standard curve prior to running any analysis. The curve must be updated quarterly as a minimum requirement.

For each analytical session a 0.5, 2.0, and 10.0 ug $N(NO_2)$ standard must be determined. The absorbance observed must be within $\pm 10\%$ of the value expected when compared to the standard curve. If not, a new standard curve must be prepared.

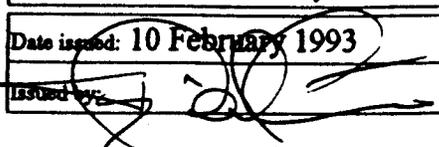
A performance standard of known concentration is analyzed during each analytical session. Agreement within $\pm 10\%$ of the vendor's published value is required. If such a recovery is not found consult the supervisor

Sample preservation is verified at log-in

Standards and reagents preparation are documented in the benchsheet notebook.

HYDROLOGIC, INC.

Analytical Procedure

Title: Oil and Grease - Hydrocarbon	
Date issued: 10 February 1993	Supersedes:
Issued by: 	

Safety:

Lab coat

Safety glasses

Extraction should be carried out under a fume hood to avoid inspiration of vapors.

Freon 113 is flammable and should not be exposed to an ignition source.

Equipment:

1000 ml separatory funnel

250 ml Erlenmeyer flask with 24/40 standard taper

12.5 cm Whatman #40 filter paper

Water bath capable of maintaining $70 \pm 1^\circ\text{C}$

Distilling head

West condenser

1000 ml graduated cylinder

Stir plate with stir bars

Reagents:

Freon 113 (1,1,2-trichloro-1,2,2-trifluoroethane)

Silica gel 60 to 200 mesh

Procedure:

Follow "Oil and Grease" Procedure to initially separate Freon soluble oils and greases.

If hydrocarbon oil and grease is to be determined in addition to determining total oil and grease, proceed as follows.

- Extract the sample as described in the Oil & Grease - Total procedure.
- After weighing the evaporated total oil and grease residue, redissolve the extract with 100 ml of Freon. The total oil and grease should contain less than 100 mg of extract.
- Add 3.0 g of silica gel, stopper and stir the mixture for 5 minutes
- Weigh to the nearest 0.1 mg a 250 ml Erlenmeyer flask and record on the benchsheet.
- Filter into a tared Erlenmeyer flask with 24/40 taper through Whatman #40 filter paper to remove silica gel. Rinse the original flask and funnel with 10 ml of freon.
- Distill off the freon at $70^\circ \pm 1^\circ\text{C}$. Record the water bath temperature on the benchsheet.

- Place in a desiccator for a minimum of 30 minutes.
 - Weigh to the nearest 0.1 mg the resulting residue and calculate (see Calculation section) the mg/L Hydrocarbon Oil and Grease
- If the hydrocarbon oil and grease is to be determined without regard to the total oil and grease concentration proceed as follows:
- Extract the sample as described in the Oil & Grease - Total method and then:
 - Add 3.0 g of silica gel to the Freon extract, stopper, and let stir for 5 minutes.
 - Weigh to the nearest 0.1 mg a 250 ml Erlenmeyer flask with 24/40 standard taper. Record weight on benchsheet.
 - Into a tare 250 ml Erlenmeyer flask with a 24/40 taper filter the mixture through Whatman #40 filter paper. Rinse the original flask and funnel with 10 ml of Freon.
 - Distill of the Freon at $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Record water bath temperature on the benchsheet.
 - Reweigh to the nearest 0.1 mg the flask and calculate (see Calculation Section) the mg/L of Hydrocarbon Oil and Grease.
- A blank should be run substituting 500 ml of distilled water for the sample. Any residue weight found in the blank must be subtracted from the sample residue weight as defined by the calculation.

Calculation:

$$\text{mg/L Hydrocarbon Oil and Grease} = \frac{(\text{D} - \text{Blank}) \times 1000}{\text{A}}$$

Where: A = Sample volume in liters
B = Final flask weight
C = Initial flask weight
D = B - C

Quality control:

Samples for hydrocarbon oil and grease determination should be preserved by lowering pH to < 2 with H_2SO_4 at the time of collection and cooled to $4^\circ C$. All samples must be collected in glass and be at least 850 ml in sample volume. The samples should be analyzed within 28 days of collection.

A blank determination is run with each analytical session.

A performance evaluation is run with each total oil and grease analytical session and carried through to the hydrocarbon oil and grease determination. No hydrocarbon oil and grease should be found.

A blank determination is made with each analytical session and the residue weight in the blank subtracted from the sample residue weight in the calculation.

The balance is calibrated daily and a 100 g Class S weight is weighed before each weighing session dictated by the methodology. The observed weight of the Class S weight is recorded on the benchsheet.

The water bath temperature is verified and recorded on the benchsheet with each analytical session.

Proper preservation is verified at log-in

HYDROLOGIC, INC.

Analytical Procedure

Title: Oil and Grease - Total	
Date issued: 12 February 1993	Supersedes: 30 May 1990
Issued by:	

Safety:

Lab coat

Safety glasses

Extraction should be carried out under a fume hood to avoid inspiration of vapors.

Freon 113 is flammable and should not be exposed to an ignition source.

Equipment:

1000 ml Separatory funned

250 ml Erlenmeyer flasks with 24/40 standard tapers

Whatman #40 filter paper 12.5 cm

Water bath capable of maintaining $70^{\circ}\text{C} \pm 1^{\circ}\text{C}$

Distilling head

West condenser

Graduated cylinders (various sizes)

Reagents:

Freon 113 (1,1,2-trichloro-1,2,2trifluoroethane)

Sodium sulfate, anhydrous (Na_2SO_4)

Procedure:

Weigh to the nearest 0.1 mg a 250 ml Erlenmeyer flask with 24/40 standard taper. Record the weight on the benchsheet.

Mark the meniscus of the sample in the sample bottle

Transfer the entire contents of the sample to a 1000 ml separatory funnel.

Add 30 ml of Freon 113 to the sample bottle and swirl to remove in sample remaining in the sample container. Transfer the rinse to the separatory funnel containing the sample.

Shake the separatory funnel vigorously for 2 minutes. Allow the aqueous and organic layer to separate.

Drain the freon layer through Whatman #40 filter paper into a tared 250 ml Erlenmeyer flask with 24/40 standard taper. Should an emulsion develop add 1 g of Na_2SO_4 to the funnel in which the filter paper is place. Additional Na_2SO_4 may be added in 1 g aliquotes to insure a dry aliquote is collected.

Shake the sample with 30 ml portions of Freon 113 an additional two times.

Rinse the tip of the separatory funnel and the filter with 10 ml of Freon 113.

Connect the Erlenmeyer flask with standard taper to the West condenser via the distilling head. Submerge the set up in the water bath at $70^{\circ} \pm 1^{\circ}\text{C}$. Record water bath temperature on the benchsheet. Distill until all the Freon is out of the Erlenmeyer flask.

Remove from the water bath and disconnect the distillation apparatus. Remove solvent vapors by sweeping the flask with air. Place the flask in a desiccator for a minimum of 30 minutes. Determine the sample volume by filling the sample container to the mark previously made. Transfer the water to a 1000 ml graduated cylinder and record the volume on the benchsheet in liters.

Reweigh the Erlenmeyer flask to the nearest 0.1 mg and record weight on the benchsheet. Calculate (see Calculations Section) the oil & grease content of the sample and record on the benchsheet. If the oil and grease content is less than 1 mg/L report as <1.0 mg/L.

Determine a blank by following the analytical procedure substituting 500 ml of water for the sample. Any residue weight found in the blank must be subtracted from the sample residue weight as described in the calculation.

Calculation:

$$\text{mg/L oil \& Grease} = \frac{(\text{D} - \text{Blank}) \times 1000}{\text{A}}$$

Where: A = Sample volume in liters

B = Final flask weight (g)

C = Initial flask weight (g)

D = B - C

Quality Control:

Samples for oil and grease determination should be preserved by adding H_2SO_4 to pH <2 and chilling to 4°C at the time of collection. Oil & Grease samples must be grab samples collected in glass with a volume of no less than 850 ml. The samples must be analyzed within 28 days of collection. (Concentrated HCl may be substituted as preserving acid for H_2SO_4 .)

A blank determination using H_2O is run with each analytical session. Any determinable residue weight in the blank is subtracted from the sample residue weight.

A performance standard of known concentration is run with each analytical batch.

The balance is calibrated daily and a 100 g Class S weight weighed to verify calibration. The Class S weight is weighed at the start of each weighing step and the observed value recorded on the benchsheet.

The water bath temperature is verified with each analytical session and documented on the benchsheet.

Proper sample preservation is verified at log-in.

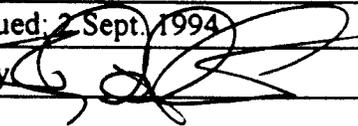
HYDROLOGIC, INC.

Analytical Procedure

Title: pH Determination Aqueous Samples

Date Issued: 2 Sept. 1994

Supersedes: 7 May 1990

Issued by: 

Safety:

Lab coat
Safety glasses

Preservation:

Sample should be chilled to 4°C at the time of collection. The samples should be analyzed as soon after collection as practicable.

Reporting limits:

There are *per se* no detection limits for pH. Thus, the pH value observed should be reported. The temperature at which the determination is made as well as the temperature of the buffer solutions must be documented on the benchsheet. The temperature of the buffer solutions and samples should be within $\pm 2^\circ\text{C}$ of each other.

Equipment:

pH Meter equipped with a temperature compensation probe, reference electrode, and low sodium error electrode.
Glass beakers (one for each sample and buffer solution)
Stir plate and teflon coated stir bars.

Reagents:

Buffer solution pH 4.0
Buffer solution pH 7.0
Buffer solution pH 9.0
Buffer solution pH 12.0
Note all buffer solutions must be traceable to NIST primary standards.

Procedure:

Calibration: Select a pair of buffers which will bracket the expected pH of the sample being measured. Complete the standardization procedure for the specific meter available at the Asheville Laboratory as given below.
Fisher 925 MP: Remove the electrodes from the storage buffer, rinse and blot dry. Avoid rubbing the pH electrode tip directly as this will impart a charge to the glass and protract the calibration process. Immerse the electrode in the selected buffer with the lowest pH which is being agitated such that no vortex is formed. Exercise care when immersing the electrodes to avoid striking the electrode with the stir bar.
Depress the "Stby/Meas" key followed by the "Two Point Cal" key. Enter the value of the standard. When the instrument has come to equilibrium as indicated by the display of five (5) stars (*), depress the "Enter" key and record the millivolt reading and the temperature of the buffer solution on

the benchsheet

Remove the electrodes from the buffer, rinse and blot dry.

Immerse the electrode into the higher buffer solution which is being agitated such that no vortex is formed.

Depress the "Stby/Meas" key followed by the value of the buffer. Allow the instrument to come to equilibrium as indicated by the display of five (5) stars (*), depress the "Enter" key and record the millivolt reading and the temperature of the buffer solution on the benchsheet.

Enter the efficiency on the benchsheet.

Orion 720 A: Select the channel which has the pH and reference electrodes installed. This can be accomplished by depressing 2nd/channel until the correct channel is selected. Depress the "Mode" key until the pH mode indicator is displayed.

Depress the "Calibrate" key and after a few seconds enter the number of buffers to be used for the standardization. Typically, this is two (2).

When the "Buffer 1" prompt appears Remove the electrodes from the storage buffer, rinse and blot dry. Avoid rubbing the pH electrode tip directly as this will impart a charge to the glass and protract the calibration process. Immerse the electrode in the selected buffer with the lowest pH which is being agitated such that no vortex is formed. Exercise care when immersing the electrodes to avoid striking the electrode with the stir bar.

Allow the instrument to stabilize as indicated by the audible beep and appearance of the "Ready Cal" prompt. Via the numeric key pad enter the value of the buffer and depress the "Yes" key. Depress the "2nd" key and millivolts. Record the value and temperature on the benchsheet.

At the "Buffer 2" prompt immerse the electrodes in the second buffer and wait for the instrument to stabilize as previously described. Enter the value of the second buffer solution and depress yes. Depress the "2nd" key and millivolts. Record the value and temperature on the benchsheet. Also, record the "Slope" of the calibration in the on the benchsheet.

Sample Determination:

Place an aliquote of sample (approximately 100 ml) into a 150 ml beaker. After beginning agitation which does not produce a vortex immerse the electrodes in the solution.

Observe the stable pH value. Add an additional 25 ml aliquote and again observe the stable pH reading. If the pH values agree within 0.1 pH units record the value and temperature of the sample on the benchsheet. Should the initial and subsequent pH value differ by more than 0.1 pH units continue adding successive aliquotes until agreement within 0.1 pH units is obtained. Record the pH and temperature on the benchsheet.

Calculations:

$$\% \text{ Standard recovery} = \frac{\text{Observed value}}{\text{True Value}} \times 100$$

$$\text{RPD} = \frac{(| \text{Repl. 1} - \text{Repl. 2} |)}{((\text{Repl 1} + \text{Repl 2})/2)} \times 100$$

Quality Control:

Proper sample preservation is verified at log in.

Duplicate samples are analyzed at a minimum rate of 10%. The replicates must agree within the upper control limit as defined by the precision control chart. Failure to achieve acceptable replication requires that the sample be rerun. Should on the second attempt acceptable replication not be obtained notify the supervisor for assistance in identification and correction of the problem.

Prior to beginning the sample run the calibration must be verified by analyzing a buffer solution with a value covered by the calibration curve. The observed value must recover within the upper and lower control limits of the buffer control chart. Failure to recover within this range requires that the calibration be redone and the verification reattempted. Failure on the second attempt requires notification of the supervisor for identification and remedy of the cause.

Prior to beginning the sample run an independent set point standard must be run. The observed value must recover within the upper and lower control limits of the buffer control chart. Failure to recover within this range requires that the calibration be redone and the verification reattempted. Failure on the second attempt requires notification of the supervisor for identification and remedy of the cause.

Should one (1) hour or more lapse between the initial calibration and sample determination, the integrity of the calibration must be demonstrated by running the verification buffer as described above. Failure to recover within the control limits necessitates recalibration of the instrument.

References:

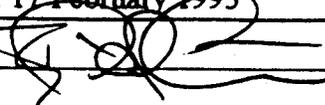
Clesceri, Lenore S., *et. al.*, *Standard Methods for the Examination of Water and Wastewater, 17th Ed.*, " 4500-H+ B Electrode Method," p 4-95 ff, 1989.

US EPA, *Methods for Chemical Analysis of Water and Wastes*, "Method 150.1 pH Electrometric," 1983.

US EPA *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW 846, 3rd Ed.*, " Method 9040A pH Electrometric Measurement," 1986.

HYDROLOGIC, INC.

Analytical Procedure

Title: Phenol Determination 4-AAP with Distillation	
Date issued: 17 February 1993	Supersedes:
Issued by: 	

Safety:

Lab Coat

Safety glasses

Chloroform is flammable, do not expose to an ignition source
Chloroform extractions should be performed under a fume hood to avoid inhalation of vapors.

Caution should be exercised in handling concentrated ammonium hydroxide, it is extremely corrosive and severe burns can result if contacted with skin.

Preparation of the buffer solution should be carried out under a fume hood to avoid inhalation of ammonia fumes.

Equipment:

Separatory funnels

Graduated cylinders (various sizes)

Pipets (various sizes)

Erlenmeyer flask (500 ml)

pH meter (Standardized according to pH procedure)

Whatman #40 filter paper

Kjeltec 1026 distillation unit with 750 ml distillation tube.

Volumetric flasks (various sizes)

Reagents:

Phenol standard 1000 ppm Ricca Cat. #5740

Intermediate phenol standard 10 ppm Preparation: Into a 500 ml volumetric flask pipet 5.0 ml of phenol standard. Dilute to volume with deionized water. Shake to mix. Prepare fresh with each analytical session.

Working phenol standard 1 ppm Preparation: Into a 500 ml volumetric flask pipet 50.0 ml of intermediate phenol standard. Dilute to volume with deionized water. Shake to mix. Prepare fresh with each analytical session.

Cupric sulfate (CuSO₄) Preparation: To the nearest 0.1 g weigh 100 g of CuSO₄·5H₂O. Quantitatively transfer to a 1000 ml volumetric flask. Add sufficient water to dissolve. When CuSO₄ is in solution dilute to volume with deionized water. Shake to mix.

Ferrous Ammonium Sulfate (Fe(NH₄)₂(SO₄)₂) Preparation: Weight to the nearest 0.1 mg, 1.1 g of Fe(NH₄)₂(SO₄)₂·6H₂O.

Quantitatively transfer to a 1 L volumetric flask containing 500 ml of deionized water and 1 ml of concentrated sulfuric acid. Swirl to mix. When $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ is dissolved dilute to volume with deionized water. Shake to mix.

Buffer solution Preparation: To the nearest 0.1 mg weigh 33.8 g of ammonium chloride (NH_4Cl). Quantitatively transfer to a 500 ml volumetric flask and add 286 ml of concentrated ammonium hydroxide (NH_4OH). Swirl to dissolve NH_4Cl . When dissolved dilute to volume with deionized water. Shake to mix. The pH of this solution at 25°C should be 10. Verify using pH procedure. Additionally, the pH should be verified prior to each analytical session when not prepared fresh. If the pH of the solution is not 10 @ 25°C, reprepare.

Preparation of this solution should be carried out under a fume hood to avoid inhalation of ammonia vapor.

4-aminoantipyrine Preparation: To the nearest 0.1 mg weigh 2.0g of 4-aminoantipyrine. Quantitatively transfer to a 100 ml volumetric flask. Add sufficient deionized water to dissolve. Then dilute to volume with deionized water. This solution should be prepared fresh with each analytical session.

Potassium ferricyanide ($\text{K}_3\text{Fe}(\text{CN})_6$) Preparation: To the nearest 0.1 mg weigh 8.0 g of $\text{K}_3\text{Fe}(\text{CN})_6$. Quantitatively transfer to a 100 ml volumetric flask. Add sufficient to dissolve. Then dilute to volume and mix. This solution should be prepared fresh with each analytical session.

Sodium sulfate anhydrous (Na_2SO_4)

Reference standard of known concentration

Procedure:

Standard curve:

Prepare a standard curve by pipeting 3.0, 5.0, 10.0, 20.0, and 25.0 mls of working standard into individual separatory funnels. Add sufficient water to make the total volume 500 ml. The solutions are respectively 6.0, 10.0, 20.0, 40.0, and 50.0 ug/L in phenol.

To each solution add 10 ml of buffer solution and mix. Then add 3 ml of 4-aminoantipyrine and mix. Finally add 3 ml of $\text{K}_3\text{Fe}(\text{CN})_6$ and mix. Allow the solutions to stand for 3 minutes. After 3 minutes add 25 ml of chloroform and shake a minimum of 10 times. Allow the aqueous and organic layers to separate. Shake the solution again a minimum of 10 times and allow the layers to separate.

Drain the chloroform layer through a Whatman #40 filter paper containing approximately 5 g of Na_2SO_4 .

Read the absorbance of each standard solution at 460 nm and record observed absorbance on the benchsheet.

A blank should be carried through the analytical procedure using 500 ml of deionized water. Set the instrument baseline using the blank extract.

Prepare a standard curve plotting standard concentration against absorbance. Archive in benchsheet notebook.

Sample Determination:

Into a 750 ml distillation tube place a 500 ml aliquote or other suitable aliquote of sample add 5 ml of CuSO_4 and 5 ml of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$. Record the sample aliquote on the benchsheet. Attach the distillation tube to the distillation unit and distill until a volume equal to the sample aliquote is collected.

Transfer the distillate to a separatory funnel. If less than 500 ml of sample is used add sufficient water to bring the volume in the separatory funnel to 500ml.

Add 10 ml of buffer solution and mix. Follow this with 3 ml of 4-aminoantipyrine and mix. Next add 3 ml of $\text{K}_3\text{Fe}(\text{CN})_6$ and mix. Finally, allow the color to develop for 3 minutes.

After 3 minutes add 25 ml of chloroform and shake the mixture at least 10 times. Allow the layer to separate and repeat the shaking procedure.

Drain the chloroform layer through Whatman #40 filter paper containing approximately 5 g of NaSO_4 .

Determine the absorbance of the solution at 460 nm and record the value on the benchsheet.

Determine the phenol concentration in the sample by comparing the observed sample absorbance to the standard curve. Record this value on the benchsheet.

Calculate the phenol concentration as outline in the calculation section. Record on benchsheet.

Calculation:

$$\text{ug/L Phenol} = \frac{\text{ug/L phenol from curve} \times 500}{\text{Sample vol. (ml)}}$$

Quality control:

Samples requiring phenol analysis must be collected in glass and preserved by lowering the pH to <2 with H_2SO_4 at the time of collection. Phenol samples should be grab samples.

A standard curve must be prepared by each analyst prior to running any samples. Validity of this curve should be verified by the supervisor.

With each analytical session a blank and a 6.0, 10.0, and 50.0 ug/L standard should be determined. The values obtained should be within $\pm 10\%$ of the values predicted by the standard curve. If not, preparation of a new standard curve is indicated.

Duplicate analysis are performed at a rate of not less than 10%.

A performance standard of known concentration is determined with each analytical session. The value found should be within $\pm 10\%$ of the vendor's published value. If not, contact the supervisor for corrective action

Solution and standards preparation are verified in the benchsheet notebook.

Proper sample preservation is verified at log-in.

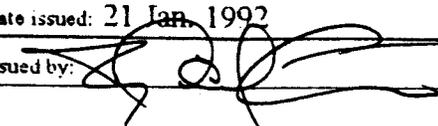
HYDROLOGIC, INC.

Analytical Procedure

Title: Phosphorus - Ortho

Date issued: 21 Jan, 1992

Supercedes:

Issued by: 

Safety:

Safety glasses
Lab coat

Equipment:

Beakers (various sizes) precleaned with boiling 1+1 HCl
Pipets (various sizes)
Volumetric flasks (various sizes)
Spectrophotometer
Cuvets

Reagents:

Nanopure water

Sulfuric acid 5N Preparation: Fill a 500 ml volumetric flask approximately half full with deionized water. Slowly add 70 ml of concentrated sulfuric acid (H_2SO_4). Allow to cool to room temperature. Dilute to volume with deionized water.

Ascorbic acid solution Preparation: To the nearest 0.1mg weigh 1.76g of ascorbic acid. Quantitatively transfer to a 100 ml volumetric flask and dilute to volume with deionized water. This solution is prepared fresh with each analytical session.

Antimony potassium tartrate solution Preparation: Weigh 1.3715 g of antimony potassium tartrate hemihydrate and quantitatively transfer to a 500 ml volumetric flask. Add enough water to dissolve. Then dilute to volume with deionized water. Store refrigerated in a dark bottle.

Ammonium molybdate solution Preparation: Weigh to the nearest 0.1 mg 20.0 g of ammonium molybdate tetrahydrate and quantitatively transfer to a 500 ml volumetric flask. Swirl to dissolve and dilute to volume. Store at 4°C in the flask in which the reagent is prepared..

Combined reagent Preparation: In exactly the order given mix the following: 50 ml of 5N H_2SO_4 , 5 ml of antimony potassium tartrate solution, 15 ml of ammonium molybdate solution and 30 ml of ascorbic acid solution. Swirl after the addition of each reagent. The resulting solution must be turbidity free before proceeding. Should any of the reagents be turbid prior to use discard the solution and prepare a fresh solution.

Phosphate standard 50 ppm PO₄, Ricca Cat. # 5830

Intermediate phosphate standard Preparation: Pipet 5 ml of phosphate standard into a 500 ml volumetric flask and dilute to volume. Mix well. The intermediate standard is 0.5 ppm PO₄.

Performance sample of known concentration

Procedure:

Standard Curve

Into 50 ml volumetric flask serially dilute the intermediate PO₄ standard to produce a set of working standards which are 0.01, 0.03, 0.05, 0.10, 0.20, 0.30, 0.40, and 0.50 ppm PO₄ respectively. Transfer each to a beaker. Prepare a blank by placing 50 ml of deionized water in a beaker.

To each standard add 8 ml of combined reagent and swirl to mix. Allow the color to develop for 10 minutes

Measure the absorbance of each standard at 880 nm and record on the benchsheet. Use the blank to set the baseline. Prepare a standard curve by plotting concentration PO₄ (mg/l) against concentration.

Sample determination

Pipet 50.0 ml of sample or an aliquote diluted to 50 ml into a 150 ml beaker. Record sample volume on benchsheet.

Add 8 ml of combined reagent and allow the color to develop for 10 minutes.

Within 30 minutes determine the absorbance of sample at 880 nm and record on benchsheet.

Determine the concentration of total phosphorus in the sample by comparing the sample absorbance to the standard curve. Record the value on the benchsheet. Calculate ppm total phosphorus in the sample and record on the benchsheet.

Calculation:

$$\text{mg/L total phosphorus} = \frac{\text{mg/L found} \times 100}{\text{Sample aliquote}}$$

Quality Control:

Samples for ortho phosphorus analysis should be filtered on-site and cooled to 4°C. The analysis must be performed within 48 hours of collection.

Duplicate analysis are performed at a rate of not less than 10%. For each analytical session a 0.01, 0.10, and 0.50 mg/L ppm PO_4 standard must be run. The values obtained must be within $\pm 15\%$ of the value expected from the standard curve. Failure of this criterion indicates the necessity of a new standard curve.

A standard curve must be prepared and documented in the benchsheet notebook by each analyst prior to performing any total phosphorus analysis. The curve must be updated at least one time per quarter. Upon updating the new curve must produce values within $\pm 15\%$ of the value expected when compared to the previous curve.

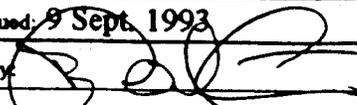
A performance standard of known concentration is analyzed during each analytical session. The value obtained must be within $\pm 20\%$ of the vendor's published value.

Sample preservation is verified at log-in.

Standards and reagent preparation is documented in the benchsheet notebook.

HYDROLOGIC, INC.

Analytical Procedure

Title: Total Phosphorus	
Date issued: 9 Sept. 1993	Supersedes: 20 Jan. 1992
Issued by: 	

Safety:

Safety glasses
Lab coat

Equipment:

Autoclave capable of maintaining 121°C @ 15-20 psig for 30 min.
125 ml Erlenmeyer flask precleaned with boiling 1+1 HCl
Beakers (various sizes) precleaned with boiling 1+1 HCl
Pipets (various sizes)
Volumetric flasks (various sizes)
Spectrophotometer
Cuvets

Reagents:

Nanopure water

Sulfuric acid 11N Preparation: To 600 ml of deionized water slowly add 310 ml of concentrated sulfuric acid (H_2SO_4).

Exercise care in preparation of this reagent, the heat of solution is extremely exothermic and the possibility of thermal and chemical burns is great.

Ammonium Persulfate

Sulfuric acid 5N Preparation: Fill a 500 ml volumetric flask approximately half full with deionized water. Slowly add 70 ml of concentrated sulfuric acid (H_2SO_4). Allow to cool to room temperature. Dilute to volume with deionized water.

Ascorbic acid solution Preparation: To the nearest 0.1mg weigh 1.76g of ascorbic acid. Quantitatively transfer to a 100 ml volumetric flask and dilute to volume with deionized water. This solution is prepared fresh with each analytical session.

Antimony potassium tartrate solution Preparation: Weigh 1.3715 g of antimony potassium tartrate hemihydrate and quantitatively transfer to a 500 ml volumetric flask. Add enough water to dissolve. Then dilute to volume with deionized water. Store refrigerated in a dark bottle.

Ammonium molybdate solution Preparation: Weigh to the nearest 0.1 mg 20.0 g of ammonium molybdate tetrahydrate and quantitatively transfer to a 500 ml volumetric flask. Swirl to dissolve and dilute to volume. Store at 4°C in the flask in which the reagent is prepared..

Combined reagent Preparation: In exactly the order given mix the following: 50 ml of 5N H_2SO_4 , 5 ml of antimony potassium tartrate solution, 15 ml of ammonium molybdate solution and 30 ml of ascorbic acid solution. Swirl after the addition of each reagent. The resulting solution must be turbidity free before proceeding. Should any of the reagents be turbid prior to use discard the solution and prepare a fresh solution.

Phosphate standard 50 ppm PO_4 Ricca Cat. # 5830

Intermediate phosphate standard Preparation: Pipet 20 ml of phosphate standard into a 500 ml volumetric flask and dilute to volume. Mix well. The intermediate standard is 2.0 ppm PO_4 .

Performance sample of known concentration

Procedure:

Standard Curve

Into 50 ml volumetric flask serially dilute the intermediate PO_4 standard to produce a set of working standards which are 0.04, 0.12, 0.20, 0.40, 0.80, 1.20, 1.60, and 2.00 ppm PO_4 respectively. Transfer each to a beaker. Prepare a blank by placing 50 ml of deionized water in a beaker.

To each standard and blank add 1.0 ml of 11 N H_2SO_4 and 0.4 g of ammonium persulfate.

Place in an autoclave and heat for 30 min at 121°C and 15-20 psig. Note that heating does not begin until the temperature and pressure requirements are met. Thus, the total time in the autoclave will be about 30 minutes longer.

After heating let the standards and blank cool to room temperature. Pipet 25 ml of each solution into a 100 ml volumetric flask and dilute to volume. The standards concentrations will be 0.01, 0.03, 0.05, 0.10, 0.20, 0.30, 0.40, and 0.50 ppm respectively.

To each standard and blank add 8 ml of combined reagent and swirl to mix.

Allow the color to develop for 10 minutes

Measure the absorbance of each standard at 880 nm and record on the benchsheet. Use the blank to set the baseline. Prepare a standard curve by plotting concentration PO_4 (mg/l) against concentration.

Sample determination

Pipet into a 125 ml Erlenmeyer flask 50.0 ml of sample.

Add 1.0 ml of 11N H_2SO_4 and 0.4g of ammonium persulfate.

Place in an autoclave and heat for 30 min at 121°C and 15-20 psig. Note that heating does not begin until the temperature and pressure

requirements are met. Thus, total time in the autoclave will be longer than 30 minutes.

After heating, depressurize the autoclave slowly, remove samples and let cool to room temperature.

Transfer the sample or an aliquote of the sample to a 100 ml volumetric flask and dilute to volume with deionized water. Shake the flask to mix contents. Record the sample volume on the benchsheet.

Transfer the sample to a 150 ml beaker and add 8 ml of combined reagent.

Allow the color to develop for ten minutes.

Within 30 minutes determine the absorbance of sample at 880 nm and record on benchsheet.

Determine the concentration of total phosphorus in the sample by comparing the sample absorbance to the standard curve. Record the value on the benchsheet. Calculate ppm total phosphorus in the sample and record on the benchsheet.

Calculation:

$$\text{mg/L total phosphorus} = \frac{\text{mg/L found} \times 100}{\text{Sample aliquote}}$$

Quality Control:

Samples for total phosphorus should be preserved with the addition of H_2SO_4 to pH <2 and chilling to 4°C at the time of collection. The analysis must be performed within 28 days of the time of collection.

Duplicate analysis are performed at a rate of not less than 10%. Duplicates should agree within the upper confidence limit established by the precision control chart.

Sample spikes are performed at a rate of not less than 10%. Spike recovery should be within 15% of the expected value.

For each analytical session a 0.01, 0.10, and 0.50 mg/L ppm PO_4 standard must be run. The values obtained must be within $\pm 10\%$ of the value expected from the standard curve. Failure of this criterion indicates the necessity of a new standard curve.

A standard curve must be prepared and documented in the benchsheet notebook by each analyst prior to performing any total phosphorus analysis. The curve must be updated at least one time per quarter. Upon updating the new curve must produce values within $\pm 10\%$ of the value expected when compared to the previous curve.

A performance standard of known concentration is analyzed during each analytical session. The value obtained must be within $\pm 12\%$ of the vendor's published value.

Sample preservation is verified at log-in.

Standards and reagent preparation is documented in the benchsheet notebook.

Quality Control:

Samples requiring sulfate analysis should be preserved by cooling to 4°C at the time of collection. The sample must be analyzed within 28 days of collection.

A standard curve must be prepared by each analyst prior to performing any analysis. The curve must be updated at least one time per quarter. Upon updating the curve the value produced by the new standards should be within $\pm 10\%$ of the expected values predicted by the previous curve.

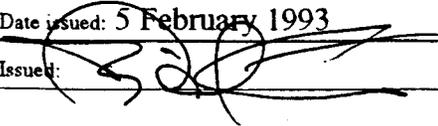
Duplicate analysis are performed at a rate of not less than 10%. For each analytical session the 1.0, 5.0, and 10.0 ppm SO_4^- standards must be run. The values found when compared to the curve should be within $\pm 15\%$ of the expected concentration. Failure to produce this recovery indicates the need for a new curve.

A performance standard of known concentration must be run during each analytical session. The value obtained must be within $\pm 15\%$ of the vendor's published value. If not, contact the supervisor.

Meter standardization is repeated after each sample determination. Solution preparation is documented in the benchsheet notebook. Proper sample preservation is verified at log in.

HYDROLOGIC, INC.

Analytical Procedure

Title: Sulfide Determination - Titrametric	
Date issued: 5 February 1993	Supercedes:
Issued: 	

Safety:

Lab Coat
Safety glasses

Equipment:

Volumetric flask (various sizes)
Pipets (various sizes)
Buret (50 ml)
Stir plate with stir bars
Erlenmeyer flasks (500 ml)

Reagents:

6 N HCl Preparation: Into a 500 ml volumetric flask which is partially filled with water, slowly add 250 ml of concentrated HCl. Swirl to mix, let cool, and dilute to volume with deionized water. Exercise caution the heat of solution is markedly exothermic and burns could result.

Starch indicator Preparation: See B. O. D. procedure
0.025 N Iodine solution Ricca Cat. #3975

0.025 N Sodium thiosulfate (Na₂S₂O₃) Reagents Cat. #7-32805

Procedure:

To a 500 ml Erlenmeyer flask pipet 20 ml of 0.025 N Iodine. Record volume of iodine added on the benchsheet.
Add 2 ml of 6N HCl.
With the pipet tip below the liquid surface in the Erlenmeyer flask, pipet 200 ml of sample. Record sample volume.
If the iodine color disappears add an additional 20 ml of 0.025 N Iodine. Record the volume of additional Iodine required on the benchsheet. Continue to add 0.025 N Iodine until the color persists.
Add starch indicator.
Titrate with 0.025 N Na₂S₂O₃ until the blue starch color is discharged. Record the volume on the benchsheet.
Calculate the sulfide concentration (see Calculation section) and record on the benchsheet.

Calculation:

$$\text{mg/L sulfide} = \frac{400(A - B)}{\text{Sample vol(ml)}}$$

A = Volume of 0.025 N Iodine (ml)

B = Volume of 0.025 N NaS_2O_3

Quality Control:

Sample requiring sulfide determination should be cooled to 4°C at the time of collection. Two ml of $\text{Zn}(\text{Ac})_2$ should also be added and the pH raised to pH >9 with NaOH at the time of collection. Duplicate analysis are performed at the rate of not less than 10%. All solution preparation is documented in the benchsheet notebook.

Sample preservation is verified at log-in.

HYDROLOGIC, INC.

Analytical Procedure

Title: Sub-contracted analysis

Date issued: 7 Jan 1992

Supersedes:

Issued by:

Safety: Safety glasses and lab. coat

Equipment: Chain of custody

Procedure: All subcontracted analysis should be shipped within 24 hours of receipt.

Toxicity samples are the exception in that they must be shipped immediately and by a carrier which will insure that the sample arrives at the laboratory performing the analysis within 24 hours of sampling.

Complete the normal log-in process as described in "Log-in Procedure."

Split samples according to the minimum volumes as stipulated in the "Sample Preservation Procedure."

Verify that the split samples are properly preserved.

Label each bottle with the appropriate sample label as generated by Labtrol.

Complete chain-of-custody for the samples being subcontracted.

Complete subcontracted analysis shipping log. Use one log sheet per day.

Typical subcontracted analysis are summarized in the chart below

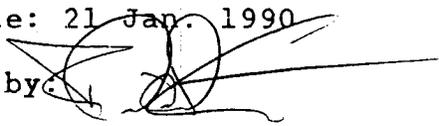
Parameter	Matrix	Subcontract lab
All organic analysis	All types	HydroLogic Frankfort, KY
Toxicities	All types	HydroLogic Morrisville
Arsenic	Drinking water	Oxford
Barium	Drinking water	Oxford
Selenium	Drinking water	Oxford
Arsenic	Waste water/soil	HydroLogic Morrisville
Selenium	Waste water/soil	HydroLogic Morrisville
Beryllium	All types	HydroLogic Morrisville
Tin	All types	Oxford
Mercury	Minette Mills	Oxford
Antimony	All types	Oxford
VOC	Drinking water	Webb Tech
COD	All types	HydroLogic Lumberton
MLVSS	Waste water	HydroLogic Lumberton
Volatile suspended solid	All types	HydroLogic Lumberton
TCLP	All types	HydroLogic Morrisville
Fluoroide	Waste water	Oxford

WASTEWATER SERVICES, INC.

Analytical Procedure

Title: Sample Preservation

Issue date: 21 Jan. 1990

Approved by: 

Safety precautions: Eye, skin protection required

Purpose: Although degradation of a sample cannot be altogether eliminated, the effects of such degradation can be retarded. Thus, sample preservation is designed to retain the integrity of the sample.

Reagents: Hydrochloric acid (HCl)
Nitric acid (HNO₃)
Sulfuric acid (H₂SO₄)
Sodium hydroxide (NaOH)
Zinc acetate (Zn(Ac)₂)
Ascorbic acid

Equipment: Dropping pipets
Scoopula
pH paper (pHydrion papers range pH 1 to 12)
Stirring rods

Procedure: For those samples requiring a pH adjustment to achieve proper preservation proceed as follows:

- Select the appropriate pH adjusting reagent as given in Table 1.
- Remove approximately 3 cm of pHydrion paper from the dispenser. By means of a wash bottle moisten the pH paper.
- Immerse a clean stirring rod in the sample and agitate the sample.
- Remove the stirring rod from the sample and touch the end of the stirring rod to the pHydrion pH paper. Match the color of the paper to the approximate pH of the sample.
- Carefully add the appropriate pH adjusting reagent (see Table 1) to the sample. Take care not to grossly overshoot the desired pH, but use sufficient reagent to achieve the desired pH. The pH of the sample should be frequently checked by moistening a 3 cm strip of pHydrion pH paper, immersing a clean stir rod in the sample, stirring, and touching the end of the stir rod to the paper.

Those samples requiring a preservation technique other than simple pH adjustment should be preserved according to the instructions given in Table 1

TABLE 1

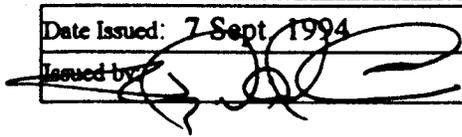
Parameter	Container	Volume required	Preservation method
Color	P,G	50 ml	Cool to 4°C
Conductance	P,G	100 ml	Cool to 4°C
Hardness	P,G	100 ml	HNO ₃ to pH <2
Odor	G	200 ml	None required
Residue			
Filterable	P,G	100 ml	Cool to 4°C
Non-filterable	P,G	100 ml	Cool to 4°C
Total	P,G	100 ml	Cool to 4°C
Volatile	P,G	100 ml	Cool to 4°C
Setteable	P,G	1000 ml	Cool to 4°C
Metals	P,G	200 ml	HNO ₃ to pH <2
Chromium +6	P,G	200 ml	Cool to 4°C
Acidity	P,G	100 ml	Cool to 4°C
Alkalinity	P,G	100 ml	Cool to 4°C
Chloride	P,G	100 ml	None required
Cyanides	P,G	500 ml	NaOH to pH >12 0.6g ascorbic acid
Fluoride	P,G	300 ml	None required
Nitrogen			
Ammonia	P,G	400 ml	H ₂ SO ₄ to pH <2
TKN	P,G	500 ml	H ₂ SO ₄ to pH <2
Nitrate/	P,G	100 ml	H ₂ SO ₄ to pH <2
Nitrite			
Nitrate	P,G	100 ml	Cool to 4°C
Nitrite	P,G	100 ml	Cool to 4°C
Phosphorus			
Ortho	P,G	50 ml	Cool to 4°C
Total	P,G	100 ml	H ₂ SO ₄ to pH <2
Sulfate	P,G	50 ml	Cool to 4°C
Sulfide	P,G	500 ml	2 ml Zn(Ac) ₂ Plus NaOH to pH >9

TABLE 1
(cont.)

Parameter	Container	Volume Required	Preservation method
BOD	P,G	1000 ml	Cool to 4°C
COD	P,G	100 ml	H ₂ SO ₄ to pH <2 Cool to 4°C
Oil & Grease	G	1000 ml	H ₂ SO ₄ to pH <2
Phenolics	G	500 ml	H ₂ SO ₄ to pH <2
MBAS	P,G	250 ml	Cool to 4°C

HYDROLOGIC, INC.

Analytical Procedure

Title: Specific Conductance	
Date Issued: 7 Sept 1994	Supersedes:
Issued by: 	

Safety: Lab coat
Safety Glasses

Preservation: Sample should be chilled to 4°C at the time of collection. The samples should be analyzed as soon after collection as practicable.

Reporting limits: There are *per se* no detection limits for specific conductance. The specific conductance measurement should be made at 25°C. If the temperature is not 25°C a temperature correct should be made and the value obtained reported. (See Calculation Section)

Equipment: Conductivity Meter equipped with conductivity cell
Thermometer capable of being read to the nearest 0.1°C.

Reagents: Water with conductivity of less than 1 μ mho/cm (18 megaohm)
Potassium chloride (KCl) anhydrous
Standard KCl solution (0.0100 N): Prepare by weighing to the nearest 0.1 mg 0.7456 g of KCl which has been previous dried for 3 hours at 105°C. Quantitatively transfer to a 1000 ml volumetric flask. Add sufficient water to fully dissolve KCl. Swirl to affect dissolving. Dilute to volume with water. The specific conductance of this solution is 1413 μ mho/cm at 25°C.

Procedure: Determination of cell constant:
Rinse the conductivity cell with three aliquotes of standard KCl solution. Immerse the conductivity cell in a fourth aliquote of standard KCl and record conductivity and temperature of the solution. The temperature of this solution should be as near 25°C as possible. Calculate the cell constant as given in the calculation section. Record on the benchsheet..

Sample Determination:

Rinse the conductivity cell with several aliquotes of sample
Adjust the temperature of the final aliquote to 25°C and record the specific conductance and temperature on the benchsheet. It is important to make

the determination as near 25°C as possible. As the temperature deviates from 25°C the less uncertain the temperature correction becomes. If the temperature is not 25°C apply the temperature correction calculation as given in the calculation section.

Calculations:Cell Constant:

$$C = (0.001413)(R_{KCl}) + 0.0191(t-25)$$

Where:

R_{KCl} is the measured resistance in ohms

t is the observed temperature

Temperature correction:

$$K = \frac{(K_m)(C)}{1 + 0.0191(t - 25)}$$

Where:

K = Conductivity in umho/cm

C = Cell constant

K_m = measured conductivity in umho/cm at the observed temperature

t = the observed temperature

Relative percent difference (RPD):

$$RPD = \frac{|(\text{Repl. 1} - \text{Repl. 2})|}{((\text{Repl. 1} + \text{Repl. 2})/2)} \times 100$$

Standard recovery:

$$\% \text{ Recovery} = \frac{\text{Found value}}{\text{True value}} \times 100$$

Quality Control:

Proper sample preservation is verified at log in.

Duplicate samples are run at a minimum rate of 10%. Duplicates should agree within the UCL of the Precision control chart or less than 20% RPD which ever is lower. Failure to meet these criteria requires that the sample be rerun. With a second failure notify the supervisor for assistance in identification and correction of the problem.

A standard of known specific conductance is run with each analytical batch. The found value must be within the upper and lower control limit defined by the control chart or $\pm 10\%$ of the true value, whichever is lowest. Failure to achieve results within these criteria necessitates notification of the supervisor for assistance in identification and correction of the problem.

The specific conductance of the standard KCl solution is determined with each analytical batch. The observed specific conductance, corrected for temperature, must be within $\pm 5\%$ of 1413 or within the limits established by the control chart. Failure to produce a recovery within these criteria requires that the standard be reprepared and rerun. A second failure necessitates contacting the supervisor for identification and correction of the problem.

References:

USEPA, *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW 846*, "Method 9050 Specific Conductance," 1986.
USEPA, *Methods for Chemical Analysis of Water and Wastes*, "Method 120.1 Conductance," 1983.

HYDROLOGIC, INC.

Analytical Procedure

Title: Sulfate Determination	
Date Issued: 29 Jan, 1993	Supersedes:
Issued by: 	

Safety:

Safety glasses
Lab Coat

Equipment:

Volumetric pipets (various sizes)
Mohr pipets (various sizes)
Volumetric flasks (various sizes)
Stir plate and stir bars
Stop watch
Turbidimeter Hach 2100A
Filtering flasks
Whatman Type A/E glass fiber filters
Gelman filter apparatus

Reagents:

Nanopure water or equivalent
Sulfate standard (100 ppm SO₄²⁻) Ricca Cat. #8110
Barium chloride crystals (BaCl₂) Reagents Cat. #1-14700
Conditioning reagent Ricca Cat. # 2235
Turbidity standard 10 NTU
Performance standard of known concentration

Procedure:**Standard curve:**

Prepare a standard curve by serially diluting the 100 ppm sulfate standard such that working standards which are 1.0, 2.0, 5.0, 8.0, and 10.0 ppm in sulfate are produced. Note to prepare the 8.0 ppm working standard prepare an intermediate standard by pipeting 20.0 ml of 100 ppm stock solution to a 50 ml volumetric flask and dilute to volume. From this well mixed intermediate standard pipet 20.0 ml of intermediate standard to a 100 ml volumetric flask. Dilute to volume. This standard is 8.0 ppm in SO₄²⁻. Standardize the turbidimeter using a 10 NTU standard. Transfer the standards to a 250 ml beaker. While vigorously agitating the sample add 5 ml of conditioning reagent and a mid-size scoop of BaCl₂. (BaCl₂ should be in excess to insure complete reaction with the SO₄²⁻ present.) Allow the mixture to continue to stir for exactly 1 minute after the BaCl₂ addition.

Immediately transfer to a clean, dry turbidity cuvet. Place the cuvet in the turbidimeter and measure the turbidity at half minute increments for 4 minutes. The highest turbidity reading observed is taken as the turbidity of the standard.

Plot the SO_4^{2-} concentration against turbidity in NTU's to form the standard curve.

Sample determination:

Filter 300 ml of sample through Whatman Type A/E glass fiber filters to remove interfering turbidity.

Transfer 100 ml of sample, or an aliquote of sample diluted to 100 ml to a 250 ml beaker. Record on the benchsheet. With vigorous agitation add 5 ml of conditioning reagent and a mid size scoop of BaCl_2 crystals. (Sufficient BaCl_2 should be added to insure complete reaction with SO_4^{2-} present in the sample.)

Immediately transfer an aliquote of sample to a clean, dry turbidity cuvet. Record the turbidity of the sample in half minute increments for four minutes. The highest turbidity observed should be recorded as the turbidity of the sample.

Determine the SO_4^{2-} content of the sample by comparing the observed turbidity against the standard curve. Record the concentration found. Calculate SO_4^{2-} concentration and record on the benchsheet. (See calculation section)

Calculation:

$$\text{mg/L SO}_4^{2-} = \frac{\text{mg/L SO}_4^{2-} \text{ found} \times 100}{\text{Sample aliquote (ml)}}$$

HYDROLOGIC, INC.

Analytical Procedure

Title: Total Dissolved Residue

Date issued: 21 Oct 1991

Supersedes:

Issued by:

Safety Precautions: Lab coat and safety glasses required.
Use caution handling evaporating dishes after drying, they are hot and can cause severe burns.

Equipment: Evaporating dishes
100 ml Graduated cylinders
Drying oven capable of maintaining 103-105°C
Drying oven capable of maintaining 178-182°C
Analytical balance
Crucible tongs
Dessicator
Membrane filter funnel
Suction flask
Aspiration source
Crucible holder
Crucibles
Whatman 934 AH microfibre filters of equivalent

Procedure: Crucibles, evaporating dishes, and filtering apparatus should be thoroughly cleaned with laboratory soap and water.
Shake sample to insure a homogeneous sample is obtained.
Filter a minimum of 200 ml of sample through crucible with microfibre filter
Weigh an evaporating dish which has been previously dried by placing in an oven at 104°C for a minimum of 2 hours, to the nearest 0.1 mg. Record weight as initial weight (W1)
By means of a graduated cylinder transfer 100 ml SAMPLE TO EVAPORATING DISH

HYDROLOGIC, INC

Analytical Procedure

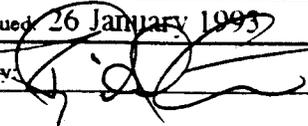
Title: Total Dissolved Solids

Pg 3 of 3

Accuracy of the balance is verified by weighing a Class S weight at the beginning of each weighing step. The observed weight of the Class S weight is recorded on the benchsheet. A distilled water blank is run with each batch of analysis to insure that the water is free of total residue. Blank values are subtracted from the $W1 - W2$ weight difference.

HYDROLOGIC, INC.

Analytical Procedure

Title: Total Kjeldhal Nitrogen (TKN)	
Date Issued: 26 January 1993	Supercedes:
Issued by: 	

Safety:

Lab Coat
Safety glasses
Fume hood

Equipment:

Vacuum source
Tekator 1026 Distillation Unit
Tekator 1007 Block Digestor
Digestion tubes (100 ml) with boiling rods
Pipets various sizes
Beakers various sizes
Buret 50 ml

Reagents:

Digestion Reagent Ricca Cat.# 2550-1
Sodium hydroxide 40% with 2.5% sodium thiosulfate
Ricca Cat.# 7490-2.5
0.1N Sulfuric Acid (H₂SO₄) Reagents Cat.# 7-30550
0.02N Sulfuric acid (H₂SO₄) Preparation: Transferring 100 ml to a 500 ml volumetric flask. Dilute to volume with deionized water.
Boric acid 2% Preparation: Weight to the nearest 0.1g 40 g of boric acid. Quantitatively transfer to a 2000 ml volumetric flask. Add enough water to dissolve. Then dilute to volume.
Mixed indicator: Preparation Weigh into a 100 ml volumetric flask 0.20 g of methyl red. Dilute to volume with ethanol and mix to dissolve. In another 100 ml volumetric flask weight 0.20 g of methylene blue indicator. Dilute to volume with ethanol and mix to dissolve. To prepare the indicator mix two part of the methyl red indicator and 1 part methylene blue. This indicator should be prepared fresh with each analytical session.
0.02N Sodium carbonate reagent (Na₂CO₃) Preparation: Dry a quantity of Na₂CO₃ at 110°C for three hours. Weigh to the nearest 0.1 mg 1.060 g of dried Na₂CO₃. Quantitatively transfer to a 1000 ml volumetric flask. Add sufficient water to dissolve. Then dilute to volume with water.
TKN standard Preparation: Weigh to the nearest 0.1 mg 2.1006 g of L (+) glutamic acid. Quantitatively transfer to a 2000 ml

volumetric flask. Add 2 ml of concentrated H_2SO_4 . Add enough water to dissolve. Dilute to volume with distilled water. Dilute this solution ten times to prepare the 10 PPM TKN working standard.

Procedure:

Transfer 100 ml of sample or an aliquote diluted to 100 ml to a digestion tube. Record sample volume on benchsheet.

Add 20 ml of digestion reagent. Place 2 boiling rods in each tube such that the concave end of the rod in down.

Assemble the digestion apparatus. To capture the SO_2 fumes evolved place the aspiration discharge line under water. Exercise care not to turn the aspiration on too strongly, otherwise sample may be aspirated once boiling begins.

Heat the tubes at $385^\circ F$ until the generation of SO_2 fumes is minimal.

Disassemble the digestion apparatus. After a 5 minute cooling period remove the boiling rods. This should be done before the sample residue solidifies.

To a 250 ml beaker add 25 ml of 2% boric acid solution.

Place the digestion tube in the distillation unit. Verify unit settings as: 2 Alkali pump strokes, 2 count delay, and 4 minute distillation. Initialize distillation by depressing "Auto/Man" button. The distillate should be collect below the level of the boric acid.

Disassemble the distillation apparatus and titrate with 0.02 N H_2SO_4 to the purple mixed indicator end point. Record volume of 0.02N H_2SO_4 on the benchsheet.

A blank consisting of 100 ml of water should be carried through the analytical process. The volume of 0.02N H_2SO_4 required to titrate it to the proper endpoint should be noted on the benchsheet. Calculate TKN concentration in the sample. Record on benchsheet.

Standardization:

Pipet 25 ml of 0.02N Na_2CO_3 in to a 250 ml beaker. Record volume on benchsheet.

Titrate with 0.02N H_2SO_4 to the purple mixed indicator record the volume required on the benchsheet. (See calculation section for calculation.)

Calculation:

$$N H_2SO_4 = \frac{(N Na_2CO_3) \times (Vol Na_2CO_3)}{Vol \text{ of } H_2SO_4}$$

$$mg/L \text{ TKN} = \frac{(ml H_2SO_4 - \text{blank})(N H_2SO_4)(14,000)}{\text{Sample aliquote}}$$

Quality Control:

Sample should be preserved by chilling to 4°C and adding H₂SO₄ to pH < 2 at the time of collection.

A blank is run with each analytical session and the volume of H₂SO₄ required to titrate it subtracted from the volume of H₂SO₄ required to titrate each sample.

A 10 ppm TKN standard is run with each analytical session. The value found must be within ± 10% of the anticipated value.

A performance standard of known concentration is run with each analytical session. The value obtained must be within ±15% of the vendor's published value.

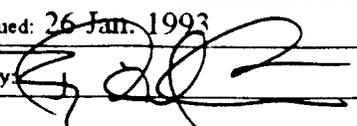
The 0.02N H₂SO₄ is standardized during each analytical session.

Proper sample preservation is verified at log-in.

All solution preparation and standardizations are documented in the benchsheet notebook.

HYDROLOGIC, INC.

Analytical Procedure

Title: Total Nitrogen	
Date issued: 26 Jan. 1993	Supersedes:
Issued by: 	

Equipment: Calculator

Reagents: None required

Procedure: Total nitrogen is a calculation combining the results of the TKN, N(NO₃), and N(NO₂) determinations. All data are reported in mg/L.

Calculation: Total Nitrogen = mg/l TKN + mg/l N(NO₃) + mg/l N(NO₂)

Quality Control: As required by the individual procedures.

HYDROLOGIC, INC.

Analytical Procedure

Title: Total suspended solids	
Date issued: 27 February 1992	Supersedes: 10/21/91
Issued by: 	

Equipment: Aluminum Foil Weighing Dishes
100 ml Graduated cylinder
Drying oven (capable of maintaining 103-105°C)
Gelman glass fiber filters Type A/E 47 mm
Gelman filtering apparatus
Filtering flasks
Dessicator
Analytical balance
Crucible tongs
Vacuum source

Procedure: Weigh each filter to the nearest 0.1 mg and record as initial weight.
Place filter in prenumbered aluminum weighing dish.
Shake samples to insure homogeneity
Transfer 100 ml of sample or an aliquot diluted to 100 ml to the graduated cylinder. Record sample volume used.
Place filter corresponding to the sample being determined in the filter apparatus and begin aspiration.
Pour sample through filtering apparatus.
Rinse graduated cylinder with a minimum of 10 ml of water.
Disassemble filter apparatus and return filter to the original numbered weigh dish.
Place the aluminum weighing dish, and filter, in the drying oven at 104°C for 1 (one) hour.
(Begin timing after the oven returns to 104°C.)
At the end of 1 (one) hour, place the filters along with the aluminum weighing dishes in the dessicator to cool to room temperature. (Minimum 30 minutes)
Weigh dried filter to the nearest 0.1 mg and record weight as final weight.
Care should be taken determining the initial and final weights that the balance is stable.

HYDROLOGIC, INC.

Analytical Procedure

Title: Toxicity Characteristic Leaching Procedure (TCLP)	
Date Issued: 6 September 1993	Supersedes:
Issued by:	

Safety: Lab coat
Safety glasses
Gloves
All sample manipulations should be carried out under a fume hood as practicable.

Equipment: Balance capable of weighing 0.01g
Magnetic stir plate and stir bars
Hot plate
Pressure filtration system
Rotary agitator capable of maintaining 30 ± 2 rpm for 18 ± 2 hrs.
Glass fiber filters 0.6 to 0.8 μ m
pH meter Standardized according to "pH Procedure"
Extraction jars
Sieve 9.5 mm
400 ml Griffin beakers
1000 ml graduated cylinder

Reagents: 1.0 N HCl Ricca Cat. #3700
1.0 N NaOH Ricca Cat. #7450
Glacial Acetic acid Reagents Cat. #5-10060
Extraction Fluid #1 Ricca Cat. #8377
Extraction Fluid #2 Ricca Cat. #8378

Procedure: This procedure applies only to metals determinations. For those samples which contain no significant amount of solids (<0.5% dry solids), the liquid after filtration through 0.6 to 0.8 μ m glass fiber filter paper is considered the TCLP extract. This extract should be acidified to pH < 2 with HNO₃ immediately and digested according to the procedure "Digestion- Total Metals in aqueous matrices." Those waste containing a significant amount of solids (>0.5% dry solids) should be carried through the following procedure.

Determination of Extraction Fluid

Weigh to the nearest 0.01g a 5g subsample into a 400 ml beaker. Add 96.5 ml of Nanopure water.

Cover the mixture and vigorously agitate for 5 minutes. Determine the pH of the mixture and record on the benchsheet.

If the pH is >5.0 add 3.5 ml of 1.0N HCl. Heat the mixture to 50°C for 10 minutes. Let cool to room temperature and determine the pH again.

Record the value on the benchsheet.

Extraction

Prior to beginning the actual extraction evaluate the results of the Preliminary Evaluations based on the following criteria.

1. Will the sample obviously yield no liquid when subjected to pressure filtration? If so, proceed with the extraction after carrying out the necessary size reduction and extraction fluid determination.
2. Are the percent solids $\geq 0.5\%$? If so, proceed with the extraction using the solid phase, after carrying out the necessary size reduction and extraction fluid determination. The result liquid phase will be recombined with the TCLP extract at the end of the procedure.
3. Did the sample contain no solids? If so, the filtrate is considered the TCLP extract. This extract should be appropriately preserved and digested as prescribed in the "Digestion-Total Metals in Aqueous Matrices," procedure.
4. Was there a filtrate produced as a result of the pressure filtration? If so, store at 4°C. This filtrate will be combined with the liquid fraction resulting from the TCLP extraction of the solid phase. The resulting liquid will become the TCLP extract. Should the two liquids be incompatible, that is form layers or form a precipitate upon recombination, they will be analyzed separately and the results combined mathematically.
5. Was particle size reduction necessary? If so, reduce the particle size of the solid phase and proceed with the extraction using the appropriate extraction fluid.
6. What was the initial pH of the sample? Was it <5.0 ? If so, use Extraction fluid #1 (pH 4.93) and proceed with the extraction after completing all other preliminary evaluations.
7. Was the pH >5.0 initially? What was the pH after the addition of 1.0N HCl? If the second pH was <5.0 use extraction fluid #1 (pH 4.93). If the second was still >5.0 use Extraction fluid #2 (pH 2.88).

When the Preliminary Evaluations are complete quantitatively transfer the solid phase to the extraction vessel. Record the weight of solid phase used on the benchsheet. Determine the amount of extraction fluid required and record on the benchsheet. (See Calculation Section)

Slowly add the proper extraction fluid. Tightly close the extraction vessel. By means of the rotary tumbler, tumble the mixture for 18 ± 2 hrs. at 30 ± 2 rpm. Record the beginning and ending times on the benchsheet. Some wastes may produce a gas as the extraction proceeds. Any excess pressure may be vented, under a hood, at 15 minute intervals as required. At the conclusion of the extraction period, turn the agitator off and separate the solid and liquid phases by filtering through acid washed 0.6 to 0.8 μ m glass fiber filter paper. Determine the pH of the liquid phase and record on the benchsheet.

If there was no initial liquid phase in the waste then the filtered liquid is the TCLP extract. Otherwise, the filtered liquid phase should be recombined with the filtrate as outlined above.

Calculations:*Determination of Percent Solids:*

$$\% \text{ Solids} = \frac{(\text{Weight of solid + filter}) - (\text{Weight of filter})}{(\text{Weight of waste + vessel}) - (\text{Weight of vessel})} \times 100$$

Determination of Weight of Extraction Fluid:

$$\text{Weight of extraction} = \frac{20 \times \% \text{ Solids} \times \text{Weight of waste}}{100}$$

Quality Control:

TCLP extraction should be carried out as soon as practicable after collection. No preservatives should be added prior to TCLP extraction. If refrigeration to 4°C does not adversely affect the sample the samples may be cooled to 4°C . Should precipitation occur on refrigeration this should be avoided.

Sufficient quantity of sample should be collected to allow completion of the Preliminary Evaluations.

TCLP extracts should be preserved to $\text{pH} < 2$ with HNO_3 , if doing so does not produce precipitation. If precipitation occurs the extraction must be repeated, eliminating the final acid preservation.

Duplicate extractions should be carried out at a rate of not less than 10%.

KENTUCKY LABORATORY

QUALITY ASSURANCE / QUALITY CONTROL MANUAL

for
Hydrologic, Inc.
1491 Twilight Trail
Frankfort, Kentucky 40601
(502) 223-0251

Prepared by
Hydrologic, Inc.
1491 Twilight Trail
Frankfort, Kentucky 40601
(502) 223-0251



[Laboratory Director]



[QA/QC Officer]

8/11/94
[Date]

8/11/94
[Date]

Quality Assurance / Quality Control Manual
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QUALITY ASSURANCE/QUALITY CONTROL
STATEMENT OF COMMITMENT

The purpose of this document is to detail the Quality Control/Quality Assurance program of the laboratory. The program is designed to meet the requirements of modern Good Laboratory Practices (GLP).

This program is followed to insure that our clients receive analytical data meeting their requirements for precision, accuracy, completeness, and legal and scientific validity.

Various employees of the lab have differing responsibilities in the implementation of this plan, but the primary responsibility for setting company policy and committing company funds is vested with the Lab Director. The Quality Assurance Officer in coordination with the Lab Director is responsible for the commitment to and approval for the Quality Control Program.

This lab has adopted this Quality Control Plan to establish and maintain the quality and performance of analytical services for all clients.

Frankfort-Hydrologic
Melissa J. Russell
QA/QC Officer

1.0 INTRODUCTION

This lab is committed to producing high quality analytical data from the analysis of environmental samples. We have developed this QA/QC Plan to ensure that standardized protocols are followed and documented for each sample analyzed.

Several tasks must be accomplished using this program.

1. Samples must be representative of sampled conditions.
2. Proper analytical procedures must be followed.
3. Analytical equipment must be in good working order.
4. Raw data must be reduced to usable client formats.
5. All above items must be well documented.

With these goals in mind, this lab has developed a comprehensive QA/QC Program that is documented in this manual and in various standard operating procedures (SOP) manuals. The key elements of this program are detailed in Figure 1. A combination of elements is used to satisfy the requirements of a specific analysis and/or project.

2.0 LAB ORGANIZATION/PERSONNEL

Lab staff is designed to allow individuals to specialize in key areas of responsibility. An Organizational Chart of the lab is shown in Figure 2. An Hydrologic Corporate organizational chart is shown in Figure 3. Given the current small size of the lab, duties will be assigned as necessary to cover these areas of responsibility.

Brief descriptions of responsibilities for the individuals involved in lab management and operation are listed below:

1. Laboratory Manager-serves as daily operations and technical manager, responsible for final review of data and reports, responsible for coordination of all lab projects.
2. QA/QC Officer-serves as an advisor of all QA/QC aspects, administrator of quality control procedures and techniques, responsible for evaluating data quality and maintaining records of QC charts and quality assurance programs, reviews performance evaluation results.
3. Project Manager-responsible for client liaison activities and coordination of lab projects with the Lab Manager, advisor of waste management and chemical hygiene/safety programs.
4. Section Supervisors-responsible for daily technical operation of their related department, responsible for review of analytical data for clarity and validity, responsible for verifying that QC and analytical procedures are being followed as specified

in this manual and Standard Operating Procedures, advise Laboratory Manager of progress, needs and potential problems in their department.

3. Sample Custodians-responsible for completing and filing all documents pertinent to the analyses of each set of samples, responsible for issuing designated serialized documents and accountability for said documents.

4. Analysts-perform analytical procedures, data manipulations, and recording per established Standard Operating Procedures including calibration and preventive maintenance of instrumentation, responsible for reporting out-of-control situations to their supervisors.

H Y D R O L O G I C , I N C

HYDROLOGIC-FRANKFORT STAFF

Name: **Benjamin Carl Esterle**
Position: **Laboratory Director**

Education: **B.S Chemistry University of Louisville 1984**
Minor Math

Brief Description: The Laboratory Director serves as the Senior Scientist/Consultant on all aspects of technical lab operations, both to clients and to lab personnel. The Laboratory Director retains responsibility for the efficient execution and overall performance of the laboratory. Mr. Esterle has been HydroLogic-Frankfort's Laboratory Director since July 1993. Before becoming Laboratory Director, he served as a GC/MS chemist at HydroLogic after eight years of environmental experience gathered throughout the United States.

Name: **Melissa J. Russell**
Position: **QA/QC Coordinator**

Education: **M.Eng. Chemical Engineering,**
University of Louisville, 1988
B.S. Applied Science and Mathematics,
University of Louisville, 1987

Brief Description: The QA/QC Officer is responsible for execution of performance according to the Quality Assurance/Quality Control Manual and Standard Operating Procedures. The QA/QC Officer review all reports prior to release to a client for proper QA/QC performance. The QA/QC Officer is also responsible for all communications with state certification offices.

Name: **Jamie Fore**
Position: **Project Management**

Education: **One Year at Kentucky State**

Brief Description: **The Project Manager manages all aspects of an environmental project including technical consulting, defining test, and furnishing sample kits/containers, directing shipping and receipt schedule of samples, monitoring project timelines, and insuring appropriate deliverables (data) in client-specified formats.**

Name: **Billie Wakefield**
Position: **Sample Custodian**

Education: **One Year at Kentucky State**

Brief Description: **The Sample Custodian receives all samples, checks for any discrepancies between the chain of custody and the samples received, and logs the samples into the system for analysis. The sample custodian may contact the client to confirm the proper analysis for each sample. The sample custodian is also responsible for shipping supplies to clients.**

Chemist: Each chemist is responsible for a particular set of analysis. They are as follows:

Name: **Inna Rasputnis**
Position: Organics Supervisor

Education: Ph.D. Chemistry, Leningrad University, 1971
Analysis responsibility: GC/MS Volatile

Name: **Jackie Tinnell**
Position: Organic Chemist

Education: B.S Chemistry/Forensic Science,
Eastern Kentucky University, 1991
Analysis responsibility: GC/MS Semi-Volatile

Name: **Kimberly Martin**
Position: Organic Chemist

Education: B.S. Biology, Georgetown College, 1991
Analysis responsibility: Diesel range Organics/Pesticides/
Herbicides/PCBs

Name: **Greg Talbert**
Position: Organic Chemist

Education: B.S. Chemistry/Forensic Science,
Eastern Kentucky University, 1991
Analysis responsibility: GC Volatiles

Name: **Eric Kopp**
Position: Organic Chemist

Education: B.S. Biology, Purdue University, 1992
Analysis responsibility: Gasoline Range Organics/ GC Volatile

The extraction department members are as follows:

Name: **Alexander Rasputnis**
Position: **Extraction Technician**

Education: **B.S. Civil Engineering, Leningrad University, 1978**

Name: **Chris Sanders**
Position: **Extraction Technician**

Education: **One year at ICS Penn State**

Name: **Jake Maynard**
Position: **Extraction Technician**

Education: **One semester at University of Kentucky**

Name: **Phillip Hall**
Position: **Extraction Technician**

Education: **Western Hills High School**

Name: Nancy Furnish
Position: Office Manager

Education: B.S. Accounting, Kentucky State University, 1975

Brief Description: The Office Manager is responsible for all financial activities of the specific laboratory including all invoicing to clientele.

Name: Nathalie Johnson
Position: General Office

Education: B.T.S. Tourism, Tour Operator Travel Agent, 1991

Name: Position not filed at this time
Position: Data Entry

Education:

Statement of Qualifications:



**Statement of
Qualifications
1994**

Statement of Qualifications





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HydroLogic's Corporate Mission

For our clients, it is our commitment:

- ◆ To provide courteous, responsive and personalized service.
- ◆ To provide high quality, legally defensible data - ultimately converting clients to advocates.
- ◆ To provide advice and solutions capable of meeting our clients' challenges.
- ◆ To perform our services in a timely and efficient manner - providing our clients with the required data on the due date.
- ◆ To treat all clients with respect, remembering they are the reason we are here.

For our employees, it is our responsibility:

- ◆ To provide a motivating and gratifying work environment enabling our employees to realize their full potential.
- ◆ To maintain a climate of trust and integrity in relationships inside and outside the corporation.
- ◆ To support our communities with cooperative and responsible corporate citizenship.
- ◆ To help our people share in the company's success, which they make possible.

For our shareholders, it is our promise:

- ◆ To ensure a competitive rate of return on their long-term investment.
- ◆ To continuously strengthen existing client relationships while aggressively pursuing new business.
- ◆ To anticipate the changes in our industry, giving ourselves and our clients a competitive edge.



Introduction

HydroLogic Laboratories, Inc., is a dynamic and rapidly growing analytical chemistry laboratory network system. This system currently features eleven testing facilities that are located throughout the United States. HydroLogic offers a full range of environmental organic/inorganic analyses and aquatic toxicity testing. With over sixty years of experience, HydroLogic has established a national reputation for delivering accurate, timely and cost effective data.

HydroLogic provides testing for commercial and government clients including water and wastewater treatment facilities; utilities; landfills; hazardous waste management companies; engineering, geo-technical and legal firms; electronics, manufacturing, textile, paper, metal finishing, brewing and agricultural industries.

Benefits of the HydroLogic Network

The many benefits to using HydroLogic's laboratory network are summarized as follows:

- ◆ By offering a broad base of experience and a variety of analytical services throughout the network, HydroLogic alleviates the difficulty in searching for multiple laboratories to complete a client's project.
- ◆ By maintaining comprehensive quality assurance/quality control (QA/QC) programs, HydroLogic is able to provide high quality services throughout the network system. As a result, the client can depend on HydroLogic's quality regardless of the laboratory selected to perform the analysis.



- ◆ Several of the individual laboratories offer complementary services to ensure that, when necessary, overflow work can be quickly shuttled to another location where qualified professionals are available to perform the analyses within the required turn-around time.

Analytical Services

The objective of HydroLogic Laboratories, Inc., is to provide quality analytical work at a reasonable cost in the format and time frame required by the clients. HydroLogic provides both routine and specialized analyses including:

- ◆ Aquatic toxicity testing
- ◆ Polychlorinated biphenyl (PCB), volatile, semi-volatile, pesticides (organophosphorus, carbamates, triazine, chlorinated), herbicide, trace metal, hydrazines and nutrient analyses
- ◆ Waste characterization and method development

HydroLogic's Laboratories

Currently, there are ten laboratories in the network strategically located in the eastern, midwestern and western regions of the United States. All laboratories are equipped with sophisticated instrumentation to provide quality analytical services locally, regionally and nationwide. The laboratories are located in:

Asheville, NC
Lumberton, NC
Raleigh, NC
Salisbury, NC
Brentwood, TN

Columbia, SC
Frankfort, KY
Denver, CO
Naples, FL
Macon, GA



Nationwide Locations

HydroLogic Asheville Lab

122 Lyman St
Asheville, NC 28801
CONTACT: Melissa Shook
704-254-5169 / FAX: 704-252-9711
800-231-8889

HydroLogic Frankfort Lab

1491 Twilight Trail
Frankfort, KY 40604
CONTACT: Billie Jo Wakefield
502-223-0251 / FAX: 502-875-8016
800-728-2251

HydroLogic Salisbury Lab

P O Box 1389
122 Mahaley Avenue
Salisbury, NC 28145
CONTACT: Eric Bolin
704-633-8089 / FAX: 704-633-6359

HydroLogic Lumberton Lab

2003 North Pine Street, Suite 2
Lumberton, NC 28358
CONTACT: Pamela Hester
910-738-6190 / FAX: 910-671-8837

HydroLogic Raleigh Lab

2500 Gateway Centre Blvd., Suite 900
Morrisville, NC 27560
CONTACT: Pomeroy Smith, II
919-380-9699 / FAX: 919-380-9717
800-241-4174

Aquatic Toxicity Lab

Raleigh NC
CONTACT: Linda McKenzie
800-241-4174

HydroLogic Columbia Lab

100 Ashland Park Lane, Suite E
Columbia, SC 29210
CONTACT: Shari Baker
803-750-0913 / FAX: 803-750-9505
800-243-0913

HydroLogic Naples Lab

2082 Trade Center Way, #303
Naples, FL 33942
CONTACT: Laura Eldridge
813-597-6059 / FAX: 813-597-7056
800-331-5340

HydroLogic Laboratories, Inc., Brighton, CO

695 N. 7th Avenue
Brighton, CO 80601
CONTACT: Bob Hart
303-659-0497 / FAX: 303-659-5064

HydroLogic Brentwood Lab

201 Summit View Drive
Brentwood, TN 37024
CONTACT: Teresa McClanahan
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HydroLogic Macon Lab

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Future Considerations

HydroLogic Laboratories, Inc., constantly evaluates the ever changing environmental market. As analytical needs change, HydroLogic is committed to providing services that will keep pace with new regulations and new client requirements.

As HydroLogic continues to expand its laboratory locations, facilities will be selected with particular attention to quality, convenience and efficient operations.

What's Covered In This Document

The information presented in this document describes the various components of the HydroLogic network of laboratories. This information should give the reader a thorough understanding of how the components fit together to form HydroLogic's quality-based organization that is committed to providing the client the best possible service:

Chapter 1, Introduction, gives an overview of HydroLogic Laboratories, Inc., by describing the benefits of using the laboratory network, discussing the laboratory locations, listing HydroLogic's analytical services, and taking a look at future considerations at HydroLogic.

Chapter 2, Professional Services, presents the diverse capabilities of HydroLogic's network system, provides a complete list of HydroLogic's analytical services, furnishes details concerning specific regulatory packages used in sample analyses, and describes the "value added" services that HydroLogic offers to its clients.

Chapter 3, Personnel, Instruments and Facilities, describes the laboratory personnel, provides information about HydroLogic's instruments and equipment, describes HydroLogic's facilities and outlines the profiles of HydroLogic's key personnel.



Chapter 4, The HydroLogic QA/QC Summary, discusses organizational responsibility for the QA/QC program, describes the procedures HydroLogic uses to produce quality data, and tracks a sample through the HydroLogic system, from the time it arrives at a HydroLogic laboratory through completion of the analysis.

Chapter 5, Certifications and Accreditation, outlines the state certifications and accreditation held by HydroLogic.

Chapter 6, Project Experience, illustrates the experience that HydroLogic has in performing analyses for commercial and government clients.

Appendix A, HydroLogic Laboratory Backgrounds, gives a brief history of each HydroLogic location.



Professional Services

In the environmental industry, there are a variety of methods, analytical services, and data packages from which to choose. HydroLogic's unique network approach to analytical services makes the decision simple. This network system offers clients the flexibility and responsiveness of a local laboratory while providing the extensive resources of a large laboratory.

HydroLogic's laboratories perform high-quality analyses that meet the criteria of regulatory requirements. In addition, HydroLogic is able to handle large projects, method development and project management. These functions are routinely offered throughout the laboratory network.

This chapter presents the following information:

- ◆ The diverse capabilities of the HydroLogic network
- ◆ A complete list of HydroLogic's analytical services and details concerning specific regulatory packages
- ◆ The "value added" services HydroLogic offers

Laboratory Capabilities

HydroLogic's laboratories have the ability to offer a full range of analytical services to clients who must comply with regulatory requirements. The majority of methodologies used in our environmental analyses meet the requirements of the Code of Federal Regulations (CFR), SW846 (8000 series), and the EPA's 100 through 600 series of organic/inorganic methods for drinking water and wastewater. HydroLogic also has biomonitoring capabilities.



Scope of Services

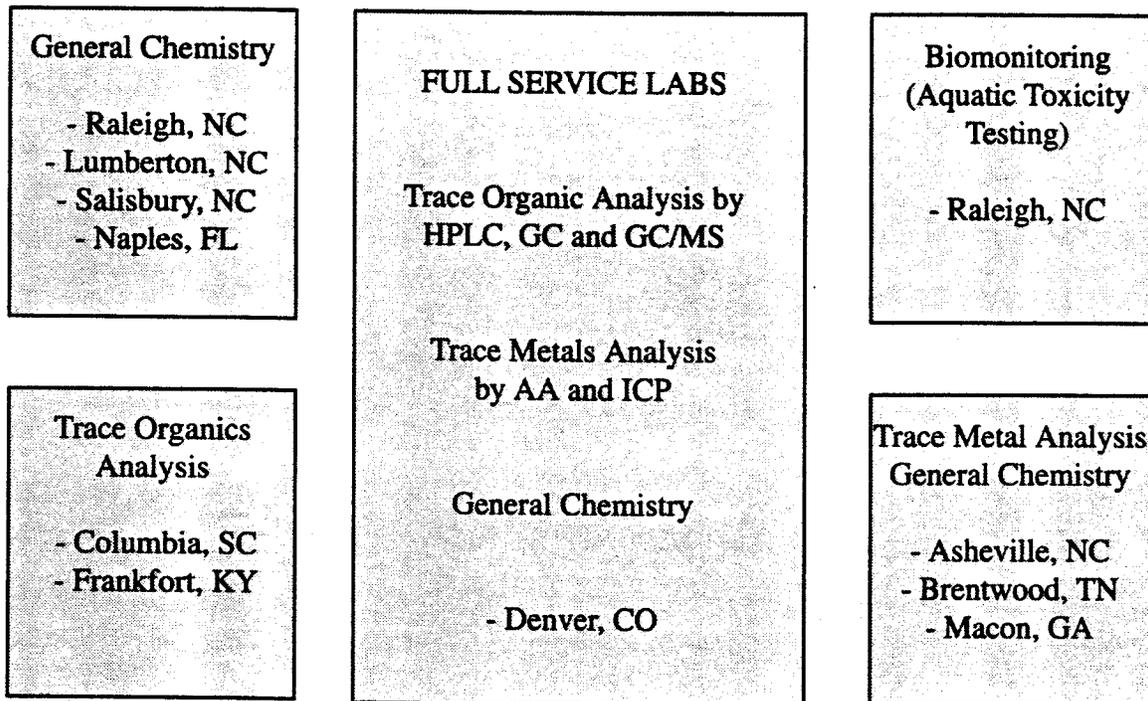
The following is a summary of the analyses that are performed at HydroLogic Laboratories, Inc.:

Packages	Analyses
(RCRA) Resource Conservation and Recovery Act	Ignitability, corrosivity, reactivity, TCLP, Appendix IX, ground water quality, drinking water suitability, underground storage tanks (USTs), Hazardous Substances List, waste characterizations
(CERCLA/SARA) Comprehensive Environmental Response, Compensation and Liability Act	CLP Target Compound List (TCL) for organics, CLP Target Analyte List (TAL) for inorganics, CLP deliverables
(SDWA) Safe Drinking Water Act	Volatiles, semi-volatiles, pesticides, herbicides, trihalomethanes, primary and secondary drinking water parameters
(CWA) Clean Water Act	NPDES monitoring, priority pollutants, biomonitoring, storm water analysis
(FIFRA) Federal Insecticide, Fungicide, and Rodenticide Act	Pesticides/herbicides analysis, biomonitoring for acute toxicity
(FDA) Food and Drug Administration	Aquatic biomonitoring for acute/chronic toxicity
(TSCA) Toxic Substances Control Act	PCBs, biomonitoring for acute/chronic toxicity
(NPDES) National Pollutant Discharge Elimination System	Biomonitoring (routine and non-routine) - Toxicity Identification Evaluation (Phase I & II) - Toxicity Reduction Evaluation (Phase I & II)
	Classical Chemistry
	Trace Metals Analyses
	Water Quality Testing



Illustration of Services Within the Network

The following illustration shows the diversity of HydroLogic's capabilities throughout the network laboratory system.





“Value Added” Services

In addition to satisfying regulatory, analytical and data management requirements, “value added” services are provided to all clients.

Sampling Container Kits

Sample containers are provided to the client free of charge for non-priority delivery. The client is responsible for shipping costs associated with overnight emergency deliveries, and for transporting filled sample bottles to HydroLogic for testing.

A typical sampling kit may include:

- ◆ Sample bottles cleaned according to the required EPA certification level and procedure
- ◆ Preservatives already added to the bottles (Preservatives may be sent separately when a client wishes to do preservation in the field.)
- ◆ Bottle labels
- ◆ Return shipping labels
- ◆ Chain of custody forms
- ◆ Custody seals
- ◆ Coolers and ice packs, as appropriate

Data Management

HydroLogic's data management system is designed to ensure confidentiality and supplies a quick retrieval method for checking the status of a sample. The system uses both electronic media and hard-copy records to ensure the safety and integrity of the data.

Samples are tracked, scheduled, and reported utilizing HydroLogic's information management systems. These resources are periodically updated to accommodate the ever changing analytical requirements of the industry.



Data Reporting

To meet the needs of clients, HydroLogic offers a variety of data package formats. Four standard data packages are available that vary in the degree of supporting data supplied to the client. The most sophisticated data package provides standard CLP deliverable forms. Upon request, computer diskette deliverables and customized reports are available for an additional fee.

Client Service

HydroLogic is committed to providing excellent client service and support. Each HydroLogic laboratory assigns a project manager or a member of the lab management staff to serve as the primary client contact for a given project. These individuals are responsible for tracking the status of the project and ensuring that it is completed according to the client's specifications.

Each laboratory maintains a staff of qualified personnel who are available to provide pricing and methodology information as needed. These professionals can quickly answer any client's questions by accessing HydroLogic's network of professional experts.

Turnaround Time

HydroLogic Laboratories, Inc., recognizes the critical importance of timely completion of the clients' analytical projects. Turnaround time at HydroLogic begins when a sample is received and accepted and ends when a report is issued (including a data package, if requested by the client.) Delivery of information by telefax or verbal communication is available for clients requiring immediate analytical results.



Rush services are available for those instances when a client needs quick turnaround times. All projects are tracked through the lab with constant attention to the required due date.

Field Services

HydroLogic provides its clients with a full range of in-field services. HydroLogic's OSHA-trained sampling teams are equipped with state-of-the-art sampling equipment and are capable of sampling monitoring wells, sanitary and storm sewers, lakes and streams, as well as a wide variety of industrial monitoring settings. Automated equipment is available for time or flow weighted composite sampling.

Sample Return

HydroLogic limits the client's exposure to liability by returning any remaining samples to the client 30 days after completion of the requested analyses.



Personnel, Instruments, & Facilities

HydroLogic Laboratories, Inc., maintains strict standards of quality by selecting exceptional personnel, high-caliber instrumentation and conveniently located laboratory facilities.

HydroLogic's carefully selected team of professionals ensures that clients receive scientific expertise and outstanding client service. Instrumentation is selected to provide the optimum combination of high-quality data and economical operations. The HydroLogic facilities provide direct access to clients throughout the United States.

This chapter presents the following information:

- ◆ A summary of HydroLogic's personnel
- ◆ HydroLogic's organization chart
- ◆ Information about HydroLogic's instruments and equipment
- ◆ A brief description of the laboratory facilities
- ◆ The profiles of HydroLogic's key personnel

Personnel

HydroLogic's laboratory personnel are highly qualified professionals dedicated to providing quality, value and service for a full scope of analytical services. The members of each laboratory staff meet or exceed the standard in the environmental laboratory industry. For their professional development, HydroLogic continually trains all laboratory personnel in QA/QC, analytical methodologies and new technologies. Continuing education and cross-training are strongly encouraged at all levels of the organization.



Personnel Summary

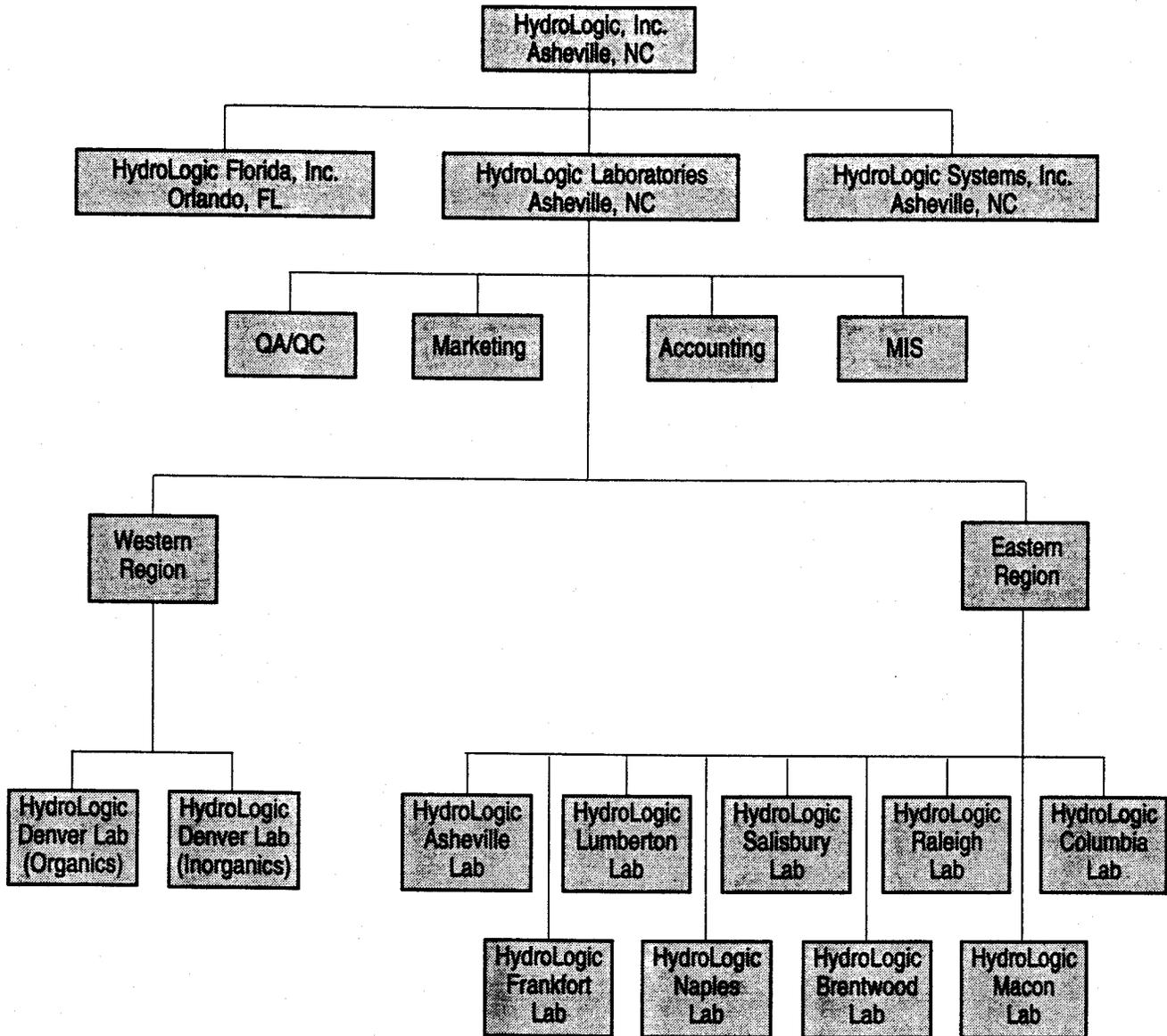
The following is a summary of HydroLogic's personnel presented by functional classification.

Funtional Category	Total Number of Employees	Percentage of Total (%)
Senior Chemist	11	12
Senior Biologist	2	2
Chemist	23	26
Biologist	6	7
Technician	26	29
Client Support	13	14
Administration	9	10
TOTAL	90	100



The HydroLogic Organization

The following organization chart outlines the HydroLogic network.





Instrumentation

Instrumentation includes gas chromatographs with a wide range of detectors, gas chromatography/mass spectroscopy, for the determination of trace organic parameters, and high performance liquid chromatography. Inductively coupled plasma and atomic absorption spectroscopy instruments provide trace metal analysis. HydroLogic also has a wide range of instrumentation for classical chemistry and general chemistry parameters.

HydroLogic's instrumentation receives continual preventative maintenance to ensure that they are capable of meeting the stringent requirements of this industry. Whenever possible, automation is utilized to reduce operating costs, and provide cost efficient analyses and high-quality data.

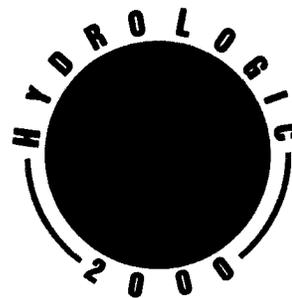
Maintenance and repair of the instrumentation are completed by in-house staff as well as by authorized service personnel from instrument distributors. For critical pieces of equipment, service contracts are maintained to ensure instrument reliability.



Instrument Summary

The following is a summary of the instrumentation utilized throughout the HydroLogic network.

Instrument Type	No. of Instruments
Gas Chromatograph	30
Gas Chromatograph/Mass Spectrometer	15
High Performance Liquid Chromatograph	2
Inductively Coupled Plasma Spectrophotometer	4
Atomic Absorption Spectrophotometer	7
TOC Carbon Analyzer	4
TOX Analyzer	3
Gel Permeation Chromatograph	2
UV/VIS Spectrophotometer	8
Infrared Spectrophotometer	3
TCLP Extractor	40
Zero Headspace Extractor	16
Ion Chromatograph	2
Incubator	15
Wastewater Samplers	26
Automatic Titration System	4
Autoclave	7
Environmental Chamber	1
Auto Analyzer	4



Facilities

All locations have been designed to optimize the use of space and create an efficient environment for the performance of analyses, data management, sample receiving and tracking, and safety. Each laboratory is also designed with features allowing flexibility to meet the demands of the environmental testing market.

These features include:

- ◆ Refrigerated sample storage
- ◆ Adequate sample preparation areas
- ◆ Well-designed ventilation systems to minimize contamination
- ◆ Adequate Hood Space
- ◆ Separate instrumentation areas

Safety

To provide a safe working environment for all HydroLogic personnel, each laboratory has established a safety program that is administered by both laboratory and management personnel. HydroLogic maintains its excellent safety record and adheres to all federal and state laws concerning hazardous waste disposal of samples and extracts.

Profiles of Key Personnel

The following are profiles of selected key personnel at HydroLogic Laboratories, Inc.

Kirby Oblachinski

Vice President of Sales and Marketing, HydroLogic, Inc.

Kirby Oblachinski is responsible for managing, coordinating and directing all sales and marketing activities in the company. He has over twenty years of sales and marketing experience and has focused on managing environmental sales activities



for major environmental firms over the past several years. He has established client based customer service programs for environmental labs and assists HydroLogic in developing and coordinating our customer service program.

Alan Kerschen

Director of Inorganic Laboratories

Mr. Kerschen reports directly to the President and CEO of HydroLogic, Inc. He is responsible for the company's seven inorganic laboratories, special projects and assists in corporate customer service. He has over twenty years environmental laboratory experience, including twelve years of laboratory management. He possesses a broad range of experience including technical expertise, customer service, sales and marketing, and management.

Benjamin Carl Esterle

General Manager, Frankfort, KY Laboratory

Ben Esterle coordinates and directs laboratory operations at the Frankfort facility and is in charge of GC, GC/MS and organic sample preparation. His experience includes: 9 years as an analytical chemist with extensive GC/MS experience; and specialized GC/MS course work throughout his career.

Pamela S. Hester

General Manager, Lumberton, NC Laboratory

Pam Hester coordinates and directs the laboratory at the Lumberton facility. She is North Carolina Grade IV Wastewater Certified and North Carolina Grade B Water Certified. Pam's experience includes extensive treatment plant operations and

lab supervision for a major municipal wastewater/water laboratory. This provided her with considerable experience in Industrial Pretreatment Monitoring and Regulation.



B. David Little

***General Manager, Laboratory Services, Asheville, NC Laboratory
Technical Director, Inorganic Laboratories***

David Little provides technical advice to the management of HydroLogic Laboratories, Inc. Additionally, he manages the operations of the Asheville laboratory which has trace metals and general chemistry capabilities. He has gained considerable experience in several industrial settings as well as in contract laboratory environments. Mr. Little specializes in atomic absorption spectrophotometry.

Linda McKenzie

Aquatic Biomonitoring Supervisor, Raleigh, NC Laboratory

Linda McKenzie is responsible for the coordination of all technical aspects of the Aquatic Biomonitoring Division including: client relations; project management; test scheduling; data analysis and review; and administrative organization of the laboratory. Her experience is based in aquaculture and mariculture research.

Pomeroy Smith, II

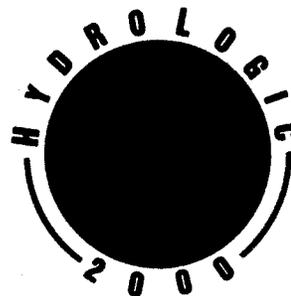
General Manager, Raleigh, NC Laboratory

Pomeroy Smith is responsible for the administrative and technical operations of the Raleigh laboratory. This includes client services, project management, supervision of laboratory personnel and administrative organization of the facility.

Eric Bolin

General Manager, Salisbury, NC Laboratory

Eric Bolin manages the administrative and technical operations for the drinking water and wastewater division of the Salisbury laboratory. He is responsible for: QA/QC management; and technical assistance and review of biological analyses for drinking water, BOD, ammonia nitrogen, total suspended solids, total dissolved solids and fecal coliform.



Dinah H. Trammel

Senior Project Manager, Corporate Office, Asheville, NC

Dinah Trammel is responsible for managing major projects from the proposal stage through final reporting. She brings to this position extensive expertise from her prior experiences with HydroLogic. Her twenty years of experience enables her to provide practical, valuable advice to our clients. Dinah's main responsibility is to keep our clients aware of their project's progress in the laboratory and be the interface between our clients and our laboratories.

Thomas D. Smith, Jr.

Laboratory Manager, Columbia, SC Laboratory

Thomas Smith coordinates and directs the Columbia laboratory. He is in charge of GC, GC/MS and organic sample preparation and is responsible for specialized support analyses for underground storage tank and site remediation projects utilizing gas chromatography and mass spectrometer analyses. Mr. Smith has over nine years experience in environmental organic chemistry and has extensive technical expertise in GC and GC/MS instrumentation.

Vernice Thomas

Laboratory Manager, Macon, GA Laboratory

Vernice Thomas coordinates and directs the laboratory at the Macon, Georgia facility. Her experience includes analytical chemistry, spectrophotometry, wet chemistry techniques, quality assurance and quality control. Vernice is certified for wastewater and drinking water analysis and has over eight years of experience in analytical laboratory work.

Teresa McClanahan

Laboratory Manager, Brentwood, TN Laboratory

QA/QC Coordinator, Inorganic Laboratories

Teresa McClanahan is an analytical chemist with three years experience in quality assurance and control, technical personnel training, and multimedia analytical testing. Ms. McClanahan has six years of experience utilizing atomic absorption spec



troscopy, automated colorimetric analysis, UV/Vis spectrophotometry and wet chemistry techniques. She has been involved in state laboratory certification programs and has a thorough knowledge of EPA protocols for sampling and analysis.

Melissa J. Russell
QA/QC Officer, Frankfort, KY Laboratory

Melissa Russell coordinates the development, implementation, and management of the Quality Assurance/Quality Control (QA/QC) program. She performs internal audits and reports her findings and recommendations to HydroLogic's management. Melissa is responsible for the QA/QC manual and SOP documentation as well as HydroLogic's state certification program. She also coordinates industrial on-site audits.

Randy Greaves, Ph.D.
Vice President, (HydroLogic Laboratories, Inc.) Denver, CO
Laboratory

Randy Greaves is responsible for directing all laboratory operations at our Denver laboratory. He has over fifteen years of environmental laboratory experience including project management, technical and operational laboratory management. His expertise encompasses CLP, USATHAMA, COE, NAVY CLEAN, AFCAA, USACE, DOD and commercial specified guidelines and standard protocols. Mr. Greaves has considerable experience defining, communicating and assessing program and project specifications to match laboratory services to the client's needs.

Tyler H. Garber
Laboratory Manager (HydroLogic Laboratories, Inc.) Denver, CO
Laboratory

Ty Garber supervises the administrative and technical operations of the wet chemistry, ion chromatography, and metal analyses division of the Denver laboratory. He is responsible for the management and productivity of this section. Mr. Garber's nine years of analytical experience has encompassed technical, operational, and administrative responsibilities in both research and environmental production facilities.



HydroLogic QA/QC Summary

The purpose of HydroLogic's Quality Assurance/Quality Control (QA/QC) program is to ensure that all clients receive analytical data that meets their need for completeness, precision, accuracy, representativeness, comparability, legal defensibility, and scientific validity. This Quality Assurance program is an integrated system that combines planning, assessment, and continual improvement. Quality Control is a daily process of verification and documentation to ensure required levels of quality. This program is available for review in HydroLogic's QA/QC manuals.

This chapter presents the following information:

- ◆ Organizational responsibility for the QA/QC program
- ◆ The quality practices HydroLogic uses to produce quality data
- ◆ HydroLogic's laboratory functions and a general flow diagram of sample handling procedures

Organizational Responsibility

The Board of Directors issues directives in regard to company policies, in particular, HydroLogic's quality assurance program. Implementation of the program is the responsibility of all employees.

To ensure consistency, completeness, and accuracy of data generated at HydroLogic Laboratories, Inc., the QA/QC Officer conducts both systems and operations audits. The results from the audits are submitted to management in order to summarize the areas where planning, assessment and improvement are necessary.



Quality Practices

Material Procurement and Control of Quality-Related Materials

The quality of materials procured for use in the laboratories must meet specified requirements that may necessitate testing prior to acceptance. Once acceptance testing is completed, control measures are taken to ensure that the quality of the material is maintained.

Chain of Custody

With the use of a chain of custody form, HydroLogic ensures the security and integrity of samples submitted for analysis. When samples are received, they are checked for damage, completeness and proper preservation. The Sample Custodian signs the chain of custody form and logs the samples into the laboratory analysis system.

The chain of custody record is the initial step in the HydroLogic system of tracking samples through preparation and analysis. Chain of custody reaches its conclusion at the disposal site. Sample retention and disposal complies with all applicable federal, state and local laws and regulations.

Sample Identification

Integrity of the sample is maintained throughout the entire analytical process by assigning sample identification numbers to the client's samples upon receipt. A list of sample identification numbers, cross-referenced to the client's internal identification numbers, is then generated for the project.



HydroLogic Laboratories, Inc., has adopted a sample tracking system that ensures data can be used as valid evidence should it become subject to litigation. This documentation encompasses chain of custody and sample notification forms, project logbooks, laboratory notebooks, instrument operation logbooks, instrument printouts and final analytical reports.

Sample Notification Forms

Sample notification forms are generated at HydroLogic to alert laboratory personnel that samples have been received. This notification, which is generated in sample receiving, details the specific analytical requirements of each sample. Once submitted to the laboratory, analyses are scheduled based on allowable holding times. The notification form also ensures that the sample will be analyzed using the appropriate methodology and correct reporting format.

Calibration Procedures

HydroLogic has a formal calibration program that utilizes calibration standards obtained from chemical suppliers with certification of purity and concentration. These commercial standards are used to prepare working standards. Working standards are made at appropriate concentrations to include the linear range of the calibration curve. Calibration check samples, obtained from a dissimilar source, are used to verify concentrations and ensure precision and accuracy. These procedures verify that instrumentation has the proper range, accuracy, and precision as specified in relevant analytical methods.



Preventative Maintenance

Preventative maintenance procedures are performed with the same or greater frequency suggested by the manufacturer. Written records are kept to document maintenance operations by analysts trained in equipment trouble-shooting. Performance criteria checks are utilized to establish a need for operating adjustments, maintenance, or repair. Preventative maintenance schedules are in place to prevent instrument failure and ensure instrument/personnel efficiency.

Quality Control Samples

Quality control samples (blanks, spikes, duplicates) are used to monitor laboratory performance throughout the network and to ensure operations are within prescribed requirements for accuracy and precision. Analysis of quality control samples is carried out using the same procedures that are used for field samples. The number and frequency of these samples are determined by method specifications. Analytical results of quality control samples are used to document the validity of data and to control data quality within predetermined acceptance limits.

Standard Operating Procedures

In order to standardize and document laboratory operations and routine analyses, detailed Standard Operating Procedures (SOPs) are maintained. These documents are designed to be bench manuals for daily use. The SOP includes pertinent details of each procedure to support the utility of the data generated by HydroLogic. After review, each SOP is approved with the signatures of the Laboratory Director, the Laboratory Supervisor and the QA/QC Officer.



Documents are controlled by an SOP numbering system, an SOP distribution list, and an archive file for outdated documents. SOPs are located where users can easily access them, and revision dates are prominently displayed to ensure that the current version is being referenced.

Biomonitoring Practices

The aquatic biomonitoring laboratories provide toxicity testing and biomonitoring support for municipalities, industries and commercial testing laboratories. Good Laboratory Practice (GLP) procedures are utilized by all personnel involved in test organism culture, generation and analysis of data, and general laboratory maintenance.

The aquatic biomonitoring laboratories maintain active cultures of all species used for on-site toxicity testing. *Ceriodaphnia dubia*, *Pimephales promelas*, *Daphnia pulex*, and *Selenastrum capricornutum* are cultured according to standard EPA protocols and recommendations. If it is necessary to supplement the genetic diversity of the cultures on hand, organisms are obtained from EPA Cincinnati or North Carolina Department of Environment, Health, and Natural Resources (NCDEM).

Sample Collection Procedures for Biomonitoring

The aquatic biomonitoring laboratories will generally rely on their clients to collect and ship effluent samples for toxicity tests. The laboratory staff will provide all necessary sample and shipping containers to the client along with sampling guidelines and chain of custody documentation.

Effluent sample collection by HydroLogic is available upon request and is collected at the point of discharge to the receiving stream or at the sampling location specified in the NPDES permit. The type of sample collected will depend on the type of toxicity test being conducted and the requirements of the discharge permit.



Grab samples are collected using appropriate containers that have been chemically cleaned prior to use. All samples are maintained at 1 to 4° C during storage and shipment. Composite samples are collected in a commercial sampling device such as a HydroLogic 100A Sampler.

Data Validation and Reporting

Data validation is performed from analysis to the final report stage. It begins with the analyst and continues with independent reviewers including the Laboratory Supervisor, the QA/QC Officer and the Laboratory Manager. The reviewer verifies that the reported results correspond to the data acquired and ensures that the data processing and calculation were done correctly. This process also serves as a monitor for sample traceability and as an audit for quality control samples. This review system ensures that the data is valid for the intended use prior to reporting.

If requested, the final report includes a summary of QC data to meet the needs of the project. Final data presentation is reviewed and approved by the Laboratory Director before release to confirm that the objectives of the project have been met.

Records Management

To ensure that all documents are accessible and traceable, HydroLogic Laboratories, Inc., maintains a records management system. Documentation is initiated at sample log-in with the chain of custody form and continues with all log books, graphs, and notebooks. Project records (raw data, chain of custody) and quality/operations data (log books, etc.) are kept separately.



All analytical data files and associated documentation are maintained in a secured central repository that safeguards them from tampering and physical loss or damage.

SOP and QA/QC Manual revisions and distribution are controlled by the QA/QC Officer. Documents are retained in accordance with applicable laws and regulations.

Nonconformance and Corrective Action

The QA/QC plan defines nonconformance according to standard operating procedures and methods. HydroLogic employees are required to report all observed or potential nonconformances to a supervisor. Upon reporting the nonconformance, the individual will prepare and sign a corrective action form and notify the QA/QC Officer. In uncontrollable situations, work will be stopped until the nonconformance situation can be resolved.

HydroLogic's analysts are trained to recognize, document, and correct problems that might affect quality. This system of review and corrective action ensures nonconformance events are resolved and recurrences avoided. This system is maintained through a cooperative effort between the analyst, section supervisor, laboratory supervisor, QA/QC officer and laboratory manager.

All corrective action forms are filed in project files.

Personnel Qualifications and Training

All employees are trained to efficiently execute the procedures which they perform in the laboratory. This training occurs through apprenticeship as experienced analysts and section supervisors answer questions and provide direct supervision. Once the analyst understands and can perform a procedure, it



is documented on a Training Check List for Lab Personnel. This form is unique for each employee, and is continually updated to reflect the entire spectrum of skills that the employee has acquired.

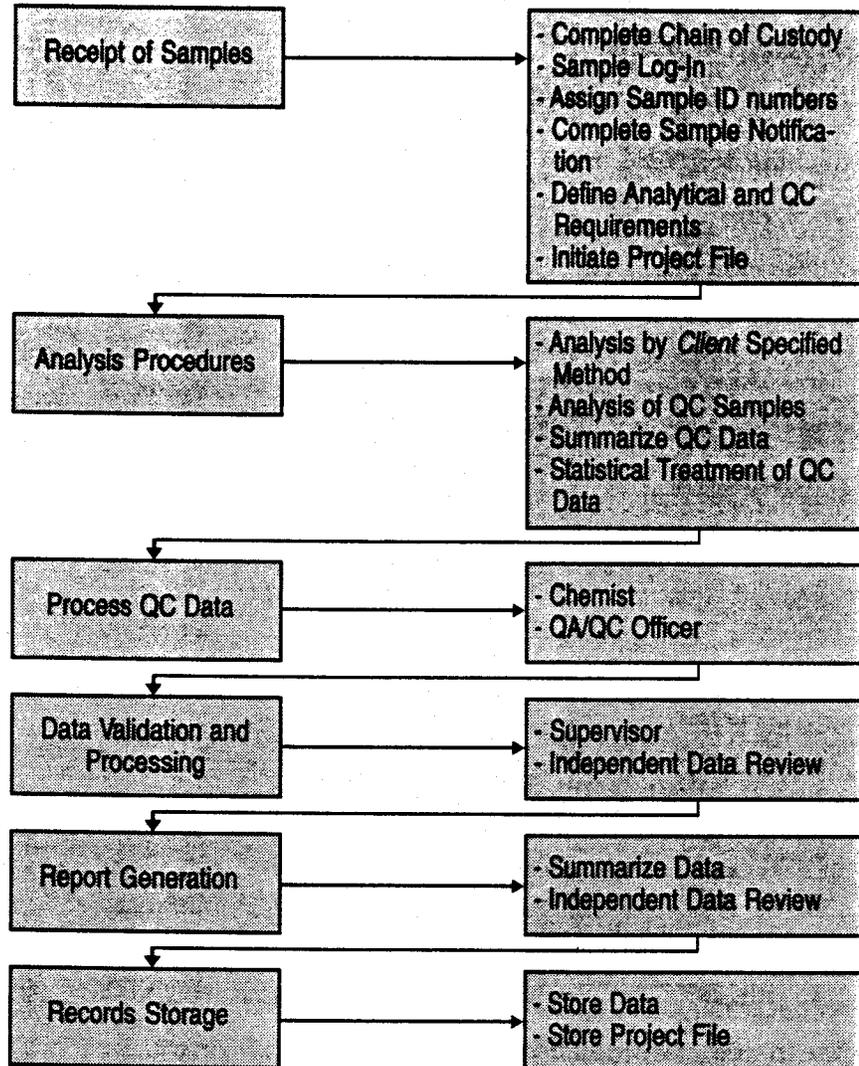
Sample Tracking

Based on HydroLogic's quality control procedures, the following chart shows the various laboratory functions and illustrates how a sample is tracked through the HydroLogic system from the time it arrives at the laboratory through completion of the analysis.



Laboratory Functions

Activities





Industrial Hygiene Plan

HydroLogic has established a complete Industrial Hygiene Plan that is used to train all personnel in the following areas:

- ◆ Proper use of safety materials
- ◆ Proper handling of chemicals, gas cylinders, and hazardous waste
- ◆ Proper response to all emergencies including fire, chemical spills, and medical crises
- ◆ Proper disposal of all hazardous waste, ensuring that HydroLogic follows both federal and state guidelines for hazardous waste handling

The Industrial Hygiene Plan is also used to guarantee that laboratory safety equipment is in good condition. Routine monitoring ensures that hoods, sprinkler systems, alarms, eyewash/shower stations and safety supplies are up to standards.

The Industrial Hygiene Plan is also used to protect the health and safety of all personnel. All personnel are regularly checked for personal exposure to chemicals in the laboratory, and routine physicals are scheduled with a specialized toxicologist.

Waste Minimization Plan

It is the policy of all HydroLogic companies to access and implement strategies of waste minimization in all aspects of operations. A continual effort will be made to identify opportunities for waste reduction in our laboratories and field operations.

All levels of management and personnel will be involved in these programs in order to achieve Pollution Prevention goals while maintaining environmental regulatory compliance. Specific goals will be defined for individual facilities in the HydroLogic group of companies.



**HydroLogic endorses the concept of Sustainable Development-
Economic Growth sustained by a Protected Environment.
Through this program, HydroLogic will set responsible stan-
dards of waste reduction for its firms.**



Certifications and Accreditation

This chapter outlines the state certifications and accreditation held by HydroLogic Laboratories. HydroLogic is currently certified to conduct analyses in a large number of states. In addition, all HydroLogic locations are striving to expand their certifications and accreditation.

The quality of data produced by HydroLogic is verified not only by a rigorous QA/QC program, but also through participation in external studies sponsored by independent third-party sources. All locations are involved in various performance evaluation (PE) programs. These programs are administered by government agencies and state certifying organizations as proof of analytical criteria compliance.

The following is a list of important environmental associations that HydroLogic supports to promote understanding and communication within the industry:

- ◆ American Chemical Society (ACS)
- ◆ American Society for Quality Control
- ◆ Colorado Association of Commerce and Industry (CACI)
- ◆ Colorado Hazardous Waste Management Society (CHWMS)
- ◆ International Association of Environmental Testing Laboratories (IAETL)



State Certifications and Approvals

The following chart lists the states in which HydroLogic is certified to conduct specific environmental analyses.

Lab Location	State Certification
Asheville, North Carolina	NC / SC / WV / TN
Lumberton, North Carolina	NC / SC
Raleigh, North Carolina	NC / SC
Salisbury, North Carolina	NC / SC
Columbia, South Carolina	SC / NC / TN
Frankfort, Kentucky	NC / KY / NJ / SC / WV / TN / IN
Denver, Colorado	CO / ND / KS / KY / MT / NM / NC / SC / SD / UT / VA / WA / WY
Naples, Florida	FL
Brentwood, Tennessee	NC / TN / SC
Macon, Georgia	GA



Project Experience

HydroLogic Laboratories, Inc., is a dynamic and rapidly growing corporation. As a result of this growth, HydroLogic is constantly increasing the diversity of its clients.

HydroLogic provides testing for commercial and government clients including: water and wastewater treatment facilities; utilities; landfills; hazardous waste management companies; engineering, geo-technical and legal firms; electronics, manufacturing, textile, paper, metal finishing, brewing and agricultural industries.

This chapter illustrates the breadth of experience that HydroLogic has in performing analyses for commercial and government clients.

Selected Commercial Projects

Cogentrix, Inc. - Lumberton, NC
1989 - Present

Perform routine and non-routine NPDES wastewater analyses for six major privately owned co-generation power plants. Analyses that are shared between the network of laboratories include: 126 priority pollutants; stormwater; biomonitoring; and a diverse list of inorganics.

Highway 36 Land Development Company - Last Chance, CO
1989 - Present

Highway 36 is the newest operating hazardous waste landfill in the country. HydroLogic's Cenref Laboratory has been involved with analytical testing at the facility for the past three years. Initially, the laboratory performed background monitoring of soils and groundwater prior to site operation.

Currently, Cenref is analyzing surface waters, leak detection system samples, and groundwaters during the operational phase of the facility. Over 40 wells are sampled and analyzed quarterly for the complete range of analytical parameters.



In addition to the environmental analytical assessments for the facility, Cenref also performs waste characterizations for Highway 36 to assess the applicability of the waste for possible treatment and disposal at their waste facility.

Non-Profit research organization (name withheld to preserve confidentiality)
1992

Determined toxicity of a compound according to TSCA regulations under GLP protocols. Methods used included 797.1300, a *Daphnia* acute toxicity test, and 797.1400, a fish acute toxicity test found in 40CFR 797.

Textile manufacturer (name withheld to preserve confidentiality)
1992

Identified and confirmed the causative agent of toxicity in an effluent. HydroLogic is presently working with the client to address operational changes necessary to remove the toxicant. Methods used included Phase I Acute Toxicity Identification Evaluation as specified by EPA 600/3.88/034.

Viar & Company - Washington, DC
1988 - 1990

Participated in a research and development project designed to evaluate tuning guidelines for GC/MS work. Awarded a contract for the analysis of pesticides, volatiles, and semi-volatiles in high level samples.

Westinghouse - Hanford, WA
1988 - 1989

Performed waste characterizations on 250 drums stored at a DOE site. The purpose of the testing was to evaluate each drum sample to assess proper disposal options. The testing involved a complex analytical scheme which encompassed both qualitative and quantitative techniques for inorganic and organic evaluations.



*Whirlpool Corporation - Columbia, SC
May 1992 - Present*

Sampled and analyzed fuel sources to identify hydraulic fluids containing PCBs and to determine proper disposal decisions as enforced by the EPA using RCRA methods. HydroLogic used EPA SW-846 guidelines for PCBs. Over 120 samples were analyzed.

Selected Government Projects

*Sanibel Wastewater Treatment Facilities - Sanibel, Florida
1993 - Present*

HydroLogic developed a unique partnership with the city of Sanibel by designing a program of flexible cost savings, cost sharing and liability control. The agreement called for HydroLogic to implement a full range of wastewater treatment services: construction and repair, plant operation, supervision of outside/contract service vendors and analytic laboratory testing. Strict professional standards were required due to the environmental sensitivity of the area.

*City and County of Denver - Denver, CO
1989 - Present*

Analyzed groundwater samples taken from monitoring wells at the Lowry Landfill near Denver. Typical analytes included volatiles, semi-volatiles, pesticides, PCBs, and inorganics by SW-846 methods.

*Denver Water Department - Denver, CO
1989 - 1990*

A pilot plant with one million gallons per day capacity was constructed to demonstrate the feasibility of converting wastewater to drinking water. Cenref was selected to analyze the effluent for all organics normally looked for in drinking water but at lower detection limits due to the high visibility of the project. The quality of the data was a significant factor since numerous agencies would be reviewing the project.



Environmental Protection Agency - Denver, CO
1985 - 1990

Performed Routine Analytical Services (RAS) for organics under the Contract Laboratory Program (CLP), which was established to provide consistent analytical services for Superfund. To support their investigations, Cenref provided data that was of documented quality. This was accomplished by operating a comprehensive Quality Assurance program involving analytical standards, performance evaluation samples and blind check samples, as well as chain of custody procedures, and document control.

North Carolina Department of Corrections - Asheville, NC
1991 - Present

Provide testing of water samples from nine correctional facilities. Composite sampling is performed bi-monthly for bacteriological testing. HydroLogic is required to conform to Standard Methods for Wastewater and Water (APHA) by a North Carolina state certified laboratory.

T.C. Analytics Inc. - Norfolk, VA
1991 - Present

This two-year contract requires analyses of soil and water samples taken from military bases. Typical analytes include volatiles, semi-volatiles, pesticides, herbicides, organics, and PAHs. This work requires that CLP QA/QC be provided with the report.



*U.S. Forest Service - Asheville, NC
1991 - Present*

Perform bacteriological testing of wastewater samples for five treatment plants and provide operations and maintenance of sites. HydroLogic is required to conform to Standard Methods for Wastewater and Water (APHA) by a New York state certified laboratory.

Representative Clients

The following is a brief list which is representative of HydroLogic's clients.

Amoco Production Company	Hitachi
Anheiser Busch Company	Holiday Inn Corp.
BASF	Kayser Roth
Burlington Knitted Fabric	Pepsico Bottling
Campbell Soup	Phillips Pipeline Company
Chen Northern	Public Service Co. of Colorado
Colorado Dept. of Health	Reynolds Metals Corp.
Conoco, Inc.	Rockwell International
Converse, Inc.	Seneca Foods Corp.
Days Inn	Shell Oil Company
Ecology & Environment	Sonoco Products
EPA	Texas Eastern
Ethan Allen	TranscoGas Pipeline
Georgia Pacific	Unocal Chemicals
Gerber Company	Western Area Power Admin.
Hewlett-Packard	Westinghouse



HydroLogic Laboratory Backgrounds

The laboratories within the HydroLogic network have a common history of providing high quality analytical data and ensuring client satisfaction. This appendix provides an abbreviated history of each HydroLogic location and describes how a commitment to quality has made the organization what it is today.

HydroLogic Asheville Laboratory Asheville, NC

The HydroLogic Asheville Laboratory is the corporate headquarters of HydroLogic Laboratories, Inc. Originally formed as Wastewater Services in 1973, the company offered comprehensive wastewater treatment plant operations and performed general chemistry services mandated by North Carolina regulations.

In 1986, Wastewater Services was acquired from its founder and became the cornerstone of the group of companies known as HydroLogic. The corporate name remained Wastewater Services until 1990. The basic commitment to providing quality analytical services has been a common theme throughout the company's metamorphosis.

The Asheville laboratory continued to expand its client base to include treatment plants, industrial clients, municipalities, and private individuals. With this expansion, the variety of the analyses offered to clients increased, necessitating relocation in late 1990 to a larger facility.

Currently, the Asheville laboratory offers a full spectrum of inorganic chemical analyses. Additionally, the Asheville facility provides wet chemistry and spectrographic analysis.



HydroLogic Lumberton Laboratory Lumberton, NC

The HydroLogic Lumberton Laboratory has been in existence for 17 years and was previously operated under the name of Professional Environmental Laboratory of Lumberton. In October, 1989, it was acquired by Wastewater Services (HydroLogic). The name was then changed to Professional Lab and Environmental Services, Inc., and it became part of the growing HydroLogic network.

The laboratory officially changed its name to HydroLogic Laboratories, Inc., in June, 1991. Highly regarded by state certification and regulatory officials, the Lumberton facility provides services to municipalities, industrial clients, and individual clients in North Carolina and South Carolina.

HydroLogic Raleigh Laboratory Raleigh, NC

The HydroLogic Raleigh Laboratory opened in 1985 under the name of Carolina Environmental Inc., and specialized in NPDES Aquatic Biomonitoring. Last year, the lab increased its certifications to include acute and chronic Ceriodaphnia and Fathead Minnow testing for North Carolina.

The staff has grown to four biologists. The Raleigh team has successfully completed toxicity identification evaluations and toxicity reduction evaluations, and has extensive research capabilities for non-routine testing on aquatic species.

In September of 1992, HydroLogic purchased Harden Environmental Management of Durham, North Carolina. This facility was merged into the Raleigh facility, which added NPDES wet chemistry capabilities.



HydroLogic Salisbury Laboratory Salisbury, NC

The HydroLogic Salisbury Laboratory was purchased by HydroLogic Laboratories, Inc., in the last quarter of 1989. Originally named Hamilton Labs, it was strictly a certified drinking water laboratory.

After becoming part of the HydroLogic network, the lab added wastewater capabilities and became certified in North Carolina for NPDES parameters including biological and inorganics. The lab now has extensive contracts to test public and private drinking water in the south central section of North Carolina.

HydroLogic Columbia Laboratory Columbia, SC

The HydroLogic Columbia Laboratory began operations as an aquatic toxicity lab in 1989, under the name of Toxicity Testing Laboratory.

Following HydroLogic's purchase of the lab in 1991, additional staff and GC instrumentation were added to address the UST market in South Carolina. Following certification in 1992, growth in the organic section of the laboratory has been remarkable. Today, the HydroLogic Columbia Laboratory is one of the leading UST laboratories in the region.



HydroLogic Frankfort Laboratory Frankfort, KY

The HydroLogic Frankfort Laboratory began operations in 1985 as Mass Spec Services, offering GC/MS analyses. Within a year, additional GC instrumentation was purchased for pesticide and PCB analyses. Later that year, the lab moved to its present location and added more personnel and GC/MS instrumentation.

HydroLogic purchased the lab in 1990. An increasing work load from the UST markets required the purchase of more instrumentation and the addition of both technical and administrative staff. By October, 1991, the facility doubled its size to contend with growing sales attracted by the lab's reputation. Today it is one of the leading organic labs in the Ohio Valley and provides analytical services to clients in a six-state area.

HydroLogic Laboratories, Inc. Denver, CO

HydroLogic's Denver Laboratory has been performing analytical chemistry testing since the early 1930's. Initially servicing the oil and gas industry, their capabilities have grown to encompass a full scope of analytical services. Their clients include governmental agencies, engineering and consulting firms, electronics, manufacturing, brewing and agricultural industries.

This lab was owned by Panhandle Eastern prior to its acquisition by HydroLogic in January. Under the leadership of the HydroLogic parent company, the Denver location continues its planned growth with major investments in new instruments and the hiring of additional qualified staff. The Denver lab specializes in environmental analyses including groundwater monitoring, priority pollutant testing, RCRA characterizations, NPDES wastewater monitoring and drinking water analysis.



HydroLogic's Denver lab served as a Contract Laboratory for the Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) for organic analysis from 1985 to 1990. They participate in numerous state certification programs and are certified by several state agencies for environmental monitoring.

With highly experienced personnel and state-of-the-art analytical instrumentation, the Denver lab meets today's most demanding environmental problems in a reliable, fast and cost-efficient manner.

HydroLogic Brentwood Laboratory Brentwood, TN (Nashville)

The HydroLogic Brentwood Laboratory opened in 1990 as a laboratory for the ADVENT Group, Inc., an environmental engineering firm. The laboratory was purchased by HydroLogic in June of 1993. The laboratory specializes in trace metals analysis and can also perform a large variety of other inorganic parameters. The lab has recently increased its capabilities to include toxicity characteristic leaching procedure (TCLP) extractions.

The laboratory holds drinking water certification in Tennessee and wastewater certification in North Carolina. The lab's clients consist of national and international industries as well as several Superfund sites.