8.0 QUALITY ASSURANCE/QUALITY CONTROL

8.1 QUALITY ASSURANCE:

A set of operating principles that, if strictly followed during sample collection and analysis, will produce data of known and defensible quality. That is, the accuracy of the analytical result can be stated with a high level of confidence.

8.1.1 Chemicals/Reagents/Gases: Ultra Pure chemicals and Ultra High Purity gases or better will be used for all metals analyses. Standards will be supplied with a Certificate of Analysis showing Manufacturer's number, Description, Lot number, Expiration date, Labeled and Measured values and Traceability to NIST SRM's. Individual analytical approved methods may specify additional requirements for the reagents to be used. All reagents will be logged and dated as to the date received and date opened with the analyst's initials, follow all SOP's for chemical receiving. No chemical/reagent will be used past its expiration date. All expired reagents will be disposed of in the proper manner.

8.1.2 Contamination: The detection limits attainable with the MHS are often a function of the amount of contamination present, rather than instrumental capabilities. The following precautions contribute to avoid inorganic contaminants.

General and customary **safety practices as well as those included in instrument manufacturer's manuals and approved methods will be strictly followed. Material Safety Data Sheets will be consulted before using any new or unknown chemical/reagent.

****Glassware preparation**: All glassware used for metals analyses will be separate from all other in the Lab and be specified as such. Only Class A Volumetric glassware will be used. All glassware will be cleaned according to the following procedure:

Between sample transfers:
1) Rinse with 1:1 nitric acid
2) Rinse with RO/DI water
3) Rinse with sample

After use:
1) Wash with detergent (Alconox or Contrad), by hand or in pipet washer, as appropriate.
2) Rinse with tap water
3) Rinse with 1:1 nitric acid
4) Rinse with RO/DI water 2X
5) Place in rack and cover

8.1.3 HNO3 Testing: Analysis of a HNO3 blank will be analyzed with every new lot of HNO3 prior to usage. This will eliminate any possible contamination of HNO3. Record in the HNO3 Logbook

8.1.4 Containers: Auto Sampler Containers: 15 ml Conical Centrifuge Tubes, Polypropylene metal free.

8.1.5 Method Development Study (Internal Quality Control): Before any analytical method is routinely employed, a methods development study will be undertaken to insure compliance with all published criteria. The instrument manufacturer's methods manuals and the selected analytical method source are excellent reference materials.

A.) Sensitivity checked (ABS-vs-CONC..) with mfr's published values +/- 10%.
B.) Linearity check of the concentration range of interest using 3 standards and a blank with a minimum of 3 replicate readings with the mean and standard deviation of the absorbance value calculated. Plot absorbance vs. concentration and calculate the correlation coefficient. The value should be 0.995 or better. If not, correct and repeat.
C.) Detection limits computed for each analyte according to Appendix B to Part 136,40 CFR, Revision 1.11.
D.) Accuracy checked with Certified known concentrations.
E.) Precision checked with duplicates or replicate analyses.
F.) Sample matrix interferences checked with spikes on all new matrices. Recoveries should be within those limits specified in 18th Edition Standard Methods for that analytical method (85-115%).

8.1.6 Method Detection Limit (MDL): MDL's must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquot of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

where: t = students' t value for a 99% confidence level and a standard deviation estimate with n-l degrees of freedom {t=3.14 for seven replicates].

S = Standard deviation of the replicate analyses.

Note: If additional confirmation is desired, reanalyze the seven replicate aliquot on two or more nonconsecutive days and again calculate the MDL values for each day. An average of the three MDL values for each analyte may provide for a more appropriate MDL estimate. See Attachment 8.1

MDL's will be determined **ANNUALLY**, when a new operator begins work or whenever, in the judgement of the analyst, confirmed by the supervisor, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.1.7 Analyst Training: All analysts' will be under strict supervision for the first 6 months upon learning a new instrument procedure. A training class will be provided, at the end of the 6 month training period a in-house PE sample will be issued to the analyst. Also a written exam in reference to operating procedures and chemical hygiene policies will be issued, upon completion of exam and PE the analyst will perform the duties required with limited supervision. See section 13 for complete training program. Training requirements will also apply for analysts' on rotation schedules. Refer to QA Manual Section 14.

8.1.8 Flame Standards Logbook: All standards will be recorded in the Flame Standards Logbook. The standard book will provide a method for preparation of standards which will include the stock standard and vendor, the amount of mls that will be used, and the total volume that will be prepared (CIVI=CFVF).

The analysts will record the **DATE PREP**, **ANALYST INITIALS**, **LOT NO.**, **EXPIRATION DATE**, and **REFERENCE NO** in the Logbook. The reference number will correspond to the page of the specific standard-date analyzed (For example 22-031595, 22 is the page of number 031595 is the date prepared).

The reference number, expiration date and analyst initials will be added to the container of the specific standard. This reference number will also be added to ID/WT files on all instruments for all batches analyzed. This will allow the analyst to cross reference any standard in the laboratory.

Note: The AA 800 software is not capable of adding the reference # to the ID/WT file.

8.1.9 Corrective Actions Logbook: Each instrument will be provided with a logbook for recording all corrective actions that take place with instrument control and/or sample analysis. Samples that don't meet the acceptable criteria will also be texted, using standard language, in SQLLIMS. The logbook will also be used to document any problems that may occur prior to analysis. The purpose of this logbook is to monitor trends in instrument and sample analysis.

8.1.10 Equipment Preventive Maintenance Procedure: There are two sections to the Maintenance and Repair Logbook, section one is the Maintenance Log with will be the responsibility of the analyst, running the instrument in question, to perform the specified duties on a daily routine. The second section will be the Repair Log, it will also be the

responsibility of the analyst to have any Perkin-Elmer Technician fill and complete at time of service, also to fill out Maintenance Log in reference to PM visits by Perkin-Elmer. A second Instrument Maintenance Log Book will be maintained by the Spectroscopy Supervisor. This log book will include all service reports generated by Perkin-Elmer. It will be the responsibility of the supervisor to order the service reports form Norwalk on a quarterly basis.

8.1.11 Performance Audits: EPA PE and in-house PE samples will be provided to the metals quarterly by the QA Manager. Two Water Pollution Studies (WP) and two Water Supplies Studies(WS) will be performed by all analysts known to be trained and proficient in the procedure involved. Performance of EPA samples will be evaluated and reported by the suppler of the proficiency samples, all in-house proficiency samples will be evaluated by the Quality Manager at WQL. See QA Manual Section 15.

8.1.12 Internal Quality Audits: The Quality Manager at WQL will conduct internal quality audits of the analytical procedures being performed by the staff. The internal quality audit will include observation of standard operating procedures, evaluation of the procedure for adequacy and precision, and evaluation of correct safety procedures. Proficiency samples are frequently submitted as a part of the audit process. Internal audits will be conducted yearly for all active SOP's. See QA Manual Section 8.

8.1.13 Prioritization of Sample Type: Due to the diversity of samples, the metals lab will employ a priority plan to run sample types in the following order Water, Environmental Health, Urban Waters, Pretreatment, and Sludge. These procedures will help to eliminate cross contamination. RULE: RUN THE CLEANEST SAMPLE TO THE DIRTIEST.

8.1.14 Instrument Quality Control:

1) Maintain a full 500 ml beaker of 1% HNO3 for aspiration when flame is lit.
2) A rinse sample will be run between samples to eliminate cross contamination.
3) Follow all maintenance procedures to eliminate contamination.

8.1.15 Data Assessment Procedure for bias and precision:

A. Laboratory Performance:

LRB-data is used to assess contamination from the laboratory environment. LRB values that exceed the MDL indicate laboratory or reagent contamination should be suspected. When LRB values constitute 10% or more of the analyte level determined for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the

source of contamination has been corrected and acceptable LRB values have been obtained.

LCS-Calculate accuracy as percent recovery using the following equation:

$$R = \frac{LCS-LRB}{s} \times 100$$

R =percent recovery
LCS=Laboratory Control Sample/Laboratory Fortified Blank
LRB=Laboratory Reagent (Method) Blk
s =concentration equivalent of analyte added to fortify the LRB solution

The laboratory must use LCS analyses data to assess laboratory performance against the required control limits of 85-115%. If the recovery of any analyte falls outside the required control limits of 85-115%, that analyte is judge out of control, and the source of the problem should be identified and resolved before continuing analyses.

When sufficient internal performance data become available (usually a minimum of twenty to thirty analyses), optional control limits can be developed from the mean percent recovery (x) and the standard deviation (S) of the mean percent recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S
LOWER CONTROL LIMIT = x + 3S

The control limits must be equal to or better than the required control limits of 85-115%. After each five to ten new recovery measurements, new control limits can e calculated using only the most recent twenty to thirty data points. Also, the standard deviation (S) data should be used to established an on-going precision statement for the level of concentrations included in the LCS. These data must be kept on file and be available for review.

ICVS: When beginning the use of a method verify the calibration standards and acceptable instrument performance with the preparation and analyses of a second source standard (ICVS). If the determined concentrations are not within +/-10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before continuing with on-going analyses.

CCVS: The laboratory must analyze the CCVS and a calibration blank immediately following each calibration, after every tenth sample and at the end of the sample run. Subsequent analyses of the CCVS must be within +/-10%, if it cannot be verified within specific limits, reanalyze either or both the CCVS and the calibration blank. If the second analysis of the CCVS or calibration blank confirm the calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. ALL SAMPLES FOLLOWING THE LAST ACCEPTABLE CCVS SOLUTION MUST BE REANALYZED.

B. Assessing Analyte Recovery and Data Quality:

MS/MSD: Sample homogeneity and the chemical nature of the sample matrix can affect analyte recovery and the quality of the data. Taking separate aliquot from the sample for replicate and fortified analyses can in some cases assess these effects. The analyte interference effects are operative in selected samples.

The laboratory must run a MS for every batch. In each case the MS aliquot must be a duplicate of the aliquot used for sample analysis and for total recoverable determination added prior to sample preparation. The added analyte concentration must be the same as that used in the laboratory fortified blank. Over time, samples from all routine sample source should be fortified.

Calculate the percent recovery for each analyte, corrected for concentration measured in the unfortified sample, and compare these values to the designated MS recovery range of 85-115%. RECOVERY CALCULATIONS ARE NOT REQUIRED IF THE CONCENTRATION ADDED IS LESS THAN 25% OF THE UNFORTIFIED SAMPLE CONCENTRATION. Percent recovery may be calculated using the following equation:

$$R = \frac{Cs - C}{s} \times 100$$

R = percent recovery
Cs= fortified sample concentration
C = concentration equivalent of analyte
s = concentration equivalent of analyte added to fortify the sample

If the recovery of any analyte falls outside the designated MS recovery range (but is still within the range of calibration) and the laboratory performance for that analyte is shown to be in control, the recovery problem encountered with the MS is judged to either matrix or solution related, not system related. All actions will be recorded in Corrective Action Logbook and the sample will be texted in LIMS as possible matrix interference.

8.1.16 Sample Control and Documentation:

A. Control Charts: Two control charts will be generated for each analyte per instrument.

MEASURE ACCURACY CONTROL CHART 1:
Percent Recovery= LCS-LRB/s x100

MEASURE PRECISION CONTROL CHART 2:
Percent Difference= [LCS-LCSD]2/LCS+LCSD X100

Note: All quality control data will be documented and files will be kept in the Control Data Logbook. Disk copies of all Control Charts generated in the computer will be kept on file.

B. Corrective Actions: All corrective actions will be documented in the Corrective Action Logbook.

8.1.17 Data Reduction/Reporting/Validation

A. Data Reduction:

Instrument Criteria: All instrument control data will be reported +/- percent difference from expected values. This will include ICVS and CCVS values.

$$\text{Calculation: } \frac{\text{Value Read - Expected Value}}{\text{Expected Value}} \times 100$$

Note: Reports will read + or - of Expected Value

Sample Criteria: All sample control data will be reported as percent difference or percent recovery of absolute value.

LCS $\quad \text{Calculation: } \frac{\text{LCS-LRB}}{\text{s}} \times 100 \quad$ %Recovery

LCSD $\quad \text{Calculation: } \frac{[\text{LCS-LCSD}]2}{\text{LCS+LCSD}} \times 100 \quad$ %Difference

MS	Calculation: $\frac{Cs-C}{s} \times 100$	%Recovery
MSD	Calculation: $\frac{[MS\ MSD]2}{MS+MSD} \times 100$	%Difference

Note: MSD %Difference must be absolute value of 10% or better.
LCS %Recovery within 85-115%
MS %Recovery within 85-115%
***Control Chart LCS and LCSD

B. Reporting: All laboratory data is entered via SQLLIMS system. Laboratory analysts are responsible for the data and result entry of the data they produce each day. All data entries will be performed at the TASK LEVEL ONLY. See QA Manual Section 4.

When available the result entry using ARE will be used for instrument generated data. When this system is not available manual data entry will be used.

C. Validation: Result approval is performed at the TASK level only. A second analyst other than the analyst entering the data will verify and approve all data entry. Two signatures will be signed on all reports, the first will be the signature of the analyst who performed the test and enter the data. The second signature will be initialed by a second analyst who will validate the first analysts data.

Out of Spec Data:
A third validation will be initialed by the supervisor if QA/QC limits are out of spec. If the QA/QC requirements are not within reporting limits; data will be reviewed by supervisor. At this time samples may be re-prepped or corrective actioned. This process is to insure proper sample prep, proper entry and calculations of all data in the metals lab.

Changes In Data: Changes in data will be performed at the TASK level only. All changes in LIMS must be approved by supervisor.

8.2 Quality Control:

8.2.1 Quality Control Requirements: The minimum requirements of this QC program consist of an initial demonstration of laboratory capability, and periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data thus generated.

***Calibration**
*Calibration Curve - Blank & 3 Standards
*Calibration Verification - Blank & Standard

***Sample Analysis**

*Laboratory Reagent Blank - < MDL
*Laboratory Fortified Blank - Minimum one per batch
*Laboratory Fortified Sample Matrix - Minimum one per batch
*Laboratory Fortified Matrix Duplicate Sample - Minimum one per batch

***Periodic Requirements**

*EPA PE Samples - Quarterly
*Method Detection Limit - Annually
*In-house PE Samples - Quarterly

8.2.2 Instrument Performance: Determination of linear dynamic ranges and analysis of quality control samples.

***Calibration Blank (Cal Blk)**

*Acid Blank Matrix
*Auto -zero instrument
*Control Limits - Ongoing Analysis < MDL
*Frequency - Before calibration, prior to sample analysis and after every CCVS
*Corrective Action - If > MDL terminate run and correct before proceeding
*Check - 1% HNO3 contamination
*Record - Corrective actions in logbook

***Initial Calibration Standards (ICAL)**

*Minimum of 3 pt calibration S1/S2/S3
* 1st source vendor
*Control Limits - Correlation coefficient equal or greater than 0.995 for curve
*Frequency - Prior to analysis of each analyte and if CCVS criteria not meet
*Corrective Action - If criteria does not meet recalibrate
*Check - Expiration date of standards, methodology applied and calculations
*Record - Corrective actions in logbook

***Initial Calibration Verification Standard (ICVS)**

*Mid-range of calibration curve
*2nd source vendor
*Control Limits - +/- 10% expected value
*Frequency - One per each calibration curve produced
*Corrective Action - If criteria not meet rerun 2nd time, if still not corrected recalibrate
*Check -Expiration date of standard, methodology applied and calculations

*Control Chart - Verification of instrument control done daily for each analyte
*Record - Corrective actions in logbook

***Continuing Calibration Verification Standard (CCVS)
*Mid-range ICAL = S2
*Control Limits - Initial +/- 5% expected value
Ongoing +/- 10% stated value
*Frequency - After calibration, every 10th sample and end of run
*Corrective Action - If criteria not met rerun 2nd time, if still not corrected recal and all samples following the last acceptable CCVS must be reanalyzed
*Check - Expiration date of standard, methodology applied and calculations
*Record - Corrective actions in logbook

8.2.3 Laboratory Performance: Determination of method detection limits.

***Laboratory Reagent (Method) Blank (LRB)
*reagent water
*Control Limits - Analyze conc < MDL
*Frequency - Each batch of 20 or fewer samples
*Corrective Action - When LRB values constitute 10% or more of the analyte level determination for a sample or is 2.2 times the analyte MDL whichever is greater, fresh aliquot of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained.
*Check - Laboratory or reagent contamination should be suspected
*Record - Corrective actions in logbook

***Laboratory Fortified Blank (LFB)
*Laboratory Control Sample (LCS)
*Control Limits - Recovery 85-115%
*Frequency - One LCS per batch
*Corrective Action - Source of the problem should be identified and resolved
*Check - Source of the problem should be identified & resolved before continuing
*Record - Corrective Actions must be recorded in laboratory logbook
*Control Chart - Verification of laboratory performance done daily for each analyte
*Record - Corrective actions in logbook

8.2.4 Data Verification: Defines the quality of data generated.

***Laboratory Fortified Matrix (MS)**

*Analyte concentration must be the same as that used in the LFB
*Spiked sample will be carried through the same analytical procedure as samples
*Control Limits - Percent recovery for each analyte of 85-115%
*Frequency - One per batch
*Corrective Action - Source of the problem should be identified & resolved
*Check - Spike solution, methodology applied and sample prep technique
*Caution - Addition of a volume of spiking solution grater than 1-2% of the sample volume may result in significant dilution error. If the volume of the spiking solution is within 2% of the sample volume, correction for dilution is not needed.
*Record - Corrective actions in logbook

***Laboratory Fortified Matrix Duplicate (MSD)**

*Analyte concentration must be the same as that used in the LFB
*Spiked sample will be carried through same analytical procedures as MS
*Control Limits - Percent difference of +/-10% between MS & MSD
*Frequency - One per batch of 20 samples or less
*Corrective Action - Source of the problem should be identified & resolved
*Check - Spike solution and sample prep technique
*Record - Corrective actions in logbook

***Field Duplicates**

*At discretion of sampling organization

***Field Blanks**

*A field blank should be prepared and analyzed as required by the data user.

*Use the same container and acid as used in sample collection.

code of federal regulations

Protection of Environment

40

PARTS 100 TO 149

Revised as of July 1, 1993

APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11

Definition

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

Procedure

1. Make an estimate of the detection limit using one of the following:

(a) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

(d) Instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

(1) Obtain another sample with a lower level of analyte in the same matrix if possible.

(2) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each

9.0 PROCEDURE-FLAME

9.1 General: Before operating the instrument for the first time, it is required that the analyst familiarize him- or herself with the operating controls and other aspects of the analysis. Standard Operating Procedure (SOP) and the PE800 Operation Manual should be studied. Proficiency will be mandated of all analysts performing analyses.

9.2 Sample Handling:

A. Collection: All samples received and handled by this section will meet the requirements of the Policy and Procedure Manual of the Water Quality Lab pertaining to handling and preservation criteria.

Samples will be logged by the Sample Custodian and assigned a unique log number. Pertinent information, i.e. location, date and time of collection, collector's name, sample type, date received by this section, person's name receiving the sample, preservation used, and any special remarks concerning the sample will be entered into the computer.

A batch number will be assigned by this section. All batches will be numbered with reference to protocol and year, for example pretreatment batches will be assigned PRE2001 beginning with 01 for batch one of the 2000 year.

B. Preservation and Preparation: All samples will be prepared following Spectroscopy's Standard Operating Procedures Sample Preparation Manual. Samples will be preserved with 2.5 ml HNO3 prior to admitting to lab. It will be the responsibility of sample custodian to check for pH. See QA Manual Section 1.0.

C. Storage: Samples that may have legal ramifications due to not being within regulatory compliance limits are to be stored in a secure area but for no longer than their allowed holding times. Confer with the supervisor before discarding any samples.

D. Holding time: All samples will be stored in the spectroscopy refrigerator until samples are prepped. Analysis will be performed within the time specified in the protocol, not to be held longer than 3 months for metal analyses.

9.3 Switching on the system:

1. Make sure fume ventilation system is operating.
2. Turn on the gas supplies.
3. For furnace systems, make sure that the cooling system is filled to the MAX mark and the switch on the rear of the cooling system is in the ON position.
4. Switch on the computer

5. Switch on the spectrometer.
6. Start AA WinLab:
   On the **Taskbar**, click on **Start** then on **Programs>{Spectrometer_name}>AA WinLab Analyst.**

9.4 Preliminary Preparation:

9.4.1 Setting up the flame system:

1. **Switch on the instruments.**
2. On the **Toolbar**, click on **Technique** and select the flame technique.
3. **Create or open a method.**
4. **Install and align the lamps.**
5. Set up the burner system.
6. **Ignite the flame**.
7. **Set up the nebulizer** record Cu Absorption
8. Set the burner reference position -use Ag method as the reference positon to start the machine and to shut it down.
9. Optimize the burner if necessary.

9.4.2 Aligning Lamps:

- Install the lamps in the lamp compartment
- On the **Toolbar**, click on **Lamps**. The Align Lamps window appears
- Enter the required information for the lamps:
  For coded lamps, you do not need to enter any information since the system sets the recommended values for the lamp parameters.

For uncoded lamps:

1. Type the element symbol, or for a multi-element lamp, the list of symbols separated by commas.
2. Select the lamp type: EDL or HCL
   The system sets the recommended values for the other parameters.

- In the **Set Up** column, click on **Lamp #** for the lamp that you want to align
  Allow an EDL to warm up for 10 to 20 minutes before you align it
- Align each lamp to maximize the enery reaching the detector. The energy is shown by the bar graph and the **Energy** value
- Close the align lamps window
- Close the lamp compartment cover

9.5 Summary: Performing analyses:

- Create or open a method
- Make sure that you set up the calibration pages correctly for the calibration technique that you intend to use
- Allow the lamps to warm up for a least 10 minutes
- Optimize the analytical conditons if necessary
- Prepare the autosample with samples, QC and check samples and calibration solutions
- **Create a sample information file**
  The entries in the sample information file depend on calibration technique that you intend to use
- **Perform an automated analysis**

9.5.1 Setting up the Automatic Analysis window

- On the **Toolbar**, click on **Auto**
- Double-click on an entry field in the **Method** column and select the methods that you require
- Set a delay time between methods or run one Method at a time
- Select the samples you want to analyze
  Either select a sample information file, and then select all or some of the locations defined in this file, or , if you do not want to use a sample information file, just select the sample tray locations.
- Select the data saving and printing options

9.5.2 Automated Analysis: Flame

Either
Analyze all the solutions

1. In the **Automated Analysis** window, click on **Analyze All**
   The system analyzes the calibration solutions first, immediately followed by the samples and any other solutions, for example, the QC and check samples

---or---

Analyze the calibration solutions, check the calibration curve, then analyze the samples

1. In the **Automated Analysis** window, click on **Calibrate**
   The system analyzes the calibration solutions and creates a calibration curve
2. If necessary, **edit the calibration curve**
3. When you are satisfied with the calibration, analyze the samples:
   In the **Automated Analysis** window, click on **Analyze Samples**

9.5.3 Stopping an analysis:

- In the Automated Analysis click on the button you used to start the analysis, for example, **Analyze Blank, Calibrate, or Analyze Samples**

  The Stopping an Analytical Sequence dialog appears

- Use this dialog to tell the system exactly which solutions to analyze before it stops

---or---

Click on **Cancel** to continue the analysis where you interrupted it.

9.5.4 Restarting an analysis:

To restart the analysis from the beginning

- Stop the analysis
- Use the dialog to tell the system exactly which solutions to analyze before it stops
- In the **Automated Analysis** window, click on **Reset**.....
- **Restart the analysis**

To continue the analysis where you interrupted it or to continue with a specific sample

- Stop the analysis
- The Continuing an Analytical Sequence dialog appears
- Use this dialog to tell the system exactly which solution to start the analysis with

9.5.5 Recalibration and reslope:

You define the type and frequency of automatic recalibrations and reslopes on the **Checks page** of the Method Editor

To perform a recalibration:

- Stop the analysis
- In the **Automated Analysis** window, click on **Reset...**
- In the **Automated Analysis** window, click on **Calibrate**

To perform a reslope:

- Stop the analysis
- In the **Automated Analysis** window, click on **Reset...**
- In the **Automated Analysis** window, click on **Reslope**

9.6 Shutting down:

9.6.1 The flame system:

- With the flame still burning, aspirate the 1% nitric acid rinsing solutions (acetone can also be used) to rinse the nebulizer and burner.
- **Extinuish the flame and bleed the gas lines**
- Exit AA WinLab: in the **File** menu, click on **Exit**
- Switch off the spectrometer and any accessories
- Shut down the computer and printer

9.7 Expression of Data:

A. Generating a Report:

1.) After all the data has been collected a report will be generated.
2.) Go into **Reporter**.

9.7.1 Order of reports:

1.) The raw data collected will be stapled with a copy of the ID/WT file followed by raw data of Daily, Lead, CMP in that order.
2.) The report will be stapled separate.
3.) Once calculations are done place report and raw data in corresponding files.

B. Data Analysis and Calculations:

All samples will be reported in ug/l, except for sludge and solid samples which require units of mg/kg. The ID/WT file will have the appropriate dilution and weight factors.

For dilution and concentration factors use the following equation:

$$\text{Result} \times \frac{\text{Final Sample Volume}}{\text{Original sample Volume}}$$

EXAMPLES:
Dilution factor:

$$\text{Final Sample Volume } \frac{\text{(10 ml sample + 40 ml DI/RO water)}}{\text{Original sample volume } 10 \text{ ml}} = 50X$$

Concentration factor:

$$\text{Final Sample Volume } \frac{25 \text{ ml}}{\text{Original Sample Volume } 250 \text{ ml}} = 0.10X$$

MG/KG Conversation:

$$\frac{\text{(mg/L)(Final Volume ml)}}{} \times \text{Dilution Factor (2)} = \text{mg/kg}$$

(wt grams)

--- or---

(ug/L)(Final Volume ml)1000 x Dilution Factor (2) = mg/kg (wt grams)

C. Reporting Data:

9.7.2 Reporting MDL's- See Attachment 1.1 for all current metals MDLS's.

9.7.3 Result Approval and Verification: The analyst running the specified test will enter all data in the SQLLIMS. This analyst will also be responsible in acquiring a second analyst to validate the data entered.

Making Dilutions

It is often necessary to make a dilution of your chemicals in order to analyze them. Dilutions can be made in one of three manners: weight/weight, weight/volume and volume/volume. The most common of these is weight/volume. The units of measurement used are commonly expressed in parts per million (ppm) or parts per billion (ppb). For weight/weight, it is expressed as 1 ppm = 1μg/gm, for weight/volume, 1 ppm = 1μg/ml, and for volume/volume, 1 ppm = 1μl/L. Chem Service supplies most of their solutions at a concentration of 0.1 mg/ml (or 100 ppm). In order to make a 100 ppm concentration starting with a pure chemical, you need to know your final volume. Then, if the material is a solid, the calculations are:

0.1mg/ml (final conc.) x 100 ml (final volume) = weight of chemical needed (10 mg)

$$FC \times FV = W$$

If the material is a liquid, it is usually easier to convert to a liquid measurement (i.e. μl) for handling purposes. Therefore, the density is needed and the calculations are:

$$\frac{\text{0.1mg/ml (final conc.) x 100 ml (final volume)}}{\text{density (mg/}\mu\text{l)}} = \text{volume (\# }\mu\text{l) of chemical needed}$$

$$\frac{FC \times FV}{d} = V$$

If you need to make a further dilution from a prepared solution, the calculations would be basically the same format:

For example, to dilute 100 ppm to 1 ppm, take 1 ml of 0.1 mg/ml solution, add to your solvent and dilute up to your final volume (100 ml).

$$\frac{\text{0.1mg/ml (initial conc.) x 1 ml (vol. injected)}}{\text{100 ml (final vol.)}} \times \frac{\text{1000 }\mu\text{g (conversion factor)}}{\text{1 mg}} = \text{1}\mu\text{g/ml (final conc.)}$$

$$\frac{IC \times VI \times CF}{FV} = FC$$

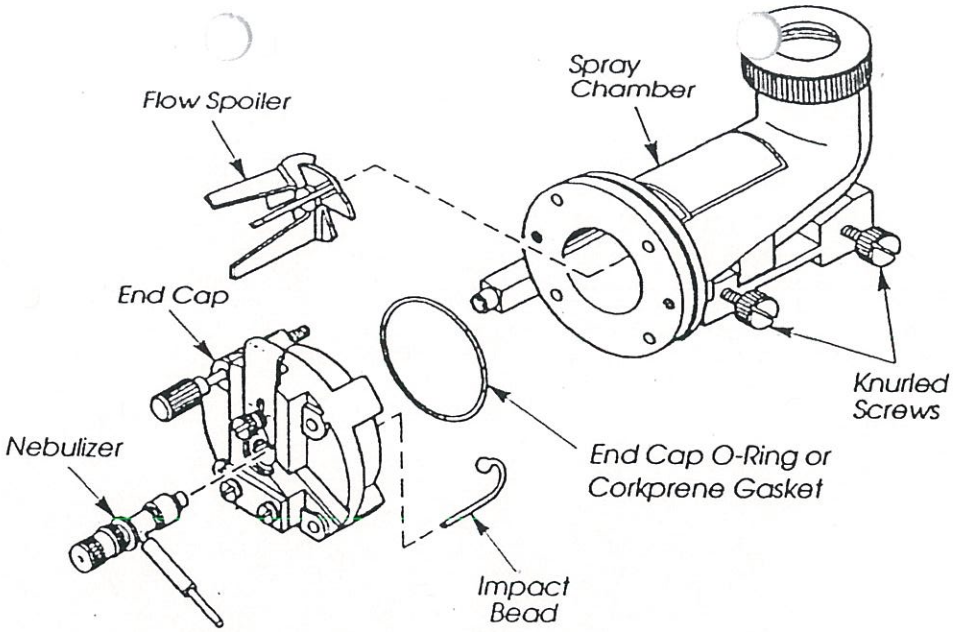
The above formula gives the final concentration of your solution. The formula to obtain the volume to inject in order to make this dilution for this final volume is:

$$\frac{FC \times FV \times CF}{IC} = VI$$

As one can see, these calculations can be manipulated to allow you to achieve any final volume or concentration you need. They can also be used to calculate to other types of concentrations (weight/weight or volume/volume).

Legend

FC	=	final concentration	IC	=	initial concentration
FV	=	final volume	VI	=	volume injected
W	=	weight	CF	=	conversion factor
d	=	density			



Spray chamber and associated parts.

Figure 9.1

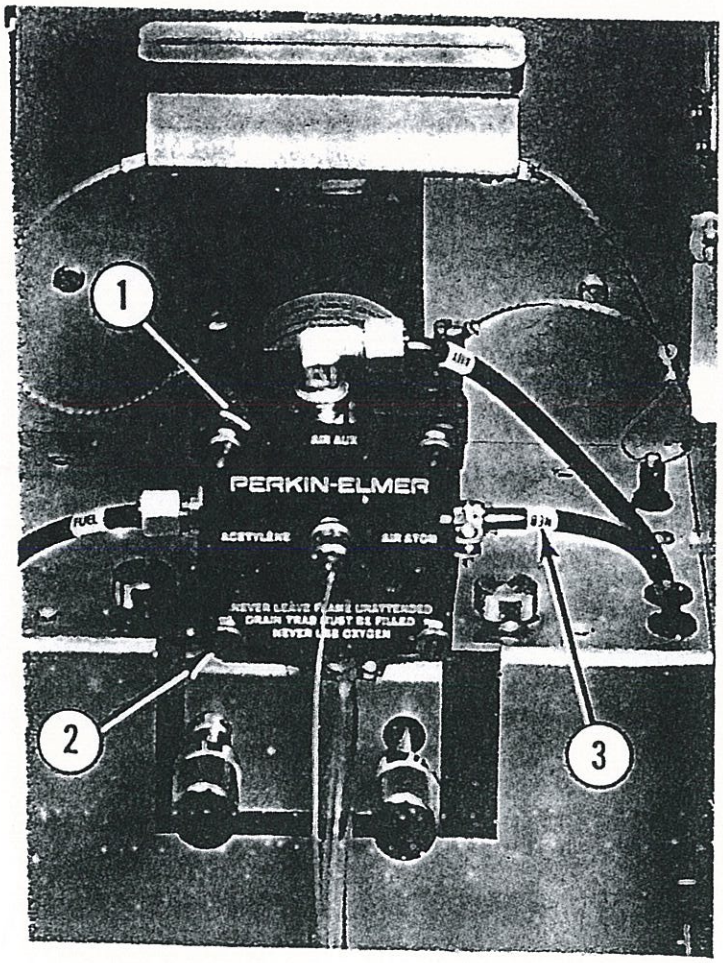


Figure 9.2