



Albuquerque Bernalillo County

Water Utility Department

WATER RECLAMATION DIVISION
4201 2ND STREET SW, ALBUQUERQUE, NEW MEXICO 87105

**WATER QUALITY LABORATORY
STANDARD OPERATING PROCEDURE APPROVAL FORM**

WQL SOP 503 Flame/Furnace AAS

CURRENT REVISION # 02 DATE May 2006

ORIGINAL ISSUE DATE January 2002

MODIFICATIONS AND REASONS FOR REVISION

*New WQL Management Staff
*New SOP Numbering System
*New QA/QC Filing System

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Out of Service By: _____ Date: / /

Reason: _____

Revision Date: March 2005

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PUBLIC WORKS DEPARTMENT
WASTEWATER UTILITY DIVISION
ENVIRONMENTAL SERVICES SECTION
TECHNICAL PROGRAMS UNIT

WATER QUALITY LABORATORY

STANDARD OPERATING PROCEDURE APPROVAL FORM

WQLSOP

SP/INST/ANALYST 800

PERKIN-ELMER ATOMIC ABSORPTION SPECTROMETER

CURRENT REVISION # 01 DATE 02/00

MODIFICATIONS AND REASONS

- **Replacement of PE5000 AA Spectrometer
- **Detailed Instrument Operation and Corrective Actions
- **Detailed QA/QC Requirements
- **Detailed Safety and Analyst Training
- **Detailed Maintenance and Troubleshooting

LABORATORY QUALITY MANAGER Jessy Moore DATE 4-11-00

CHEMICAL HYGIENE OFFICER J. Stegik DATE 1/11/2000

LABORATORY MANAGER Michael Dan DATE 4-11-2000

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CITY OF ALBUQUERQUE
WATER QUALITY LABORATORY
STANDARD OPERATING PROCEDURES
PERKIN-ELMER ANALYST 800 FLAME &
GRAPHITE FURNACE ATOMIC ABSORPTION
SPECTROMETER

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WQLSOP SP/INST/ANALYST 800
ORIGINAL ISSUE DATE JAN 2000

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1.0 SCOPE & APPLICATION

1.1 This procedure is applicable to the determination of silver, cadmium, chromium, copper, nickel, lead, zinc by flame atomic absorption. Arsenic, selenium by graphite furnace atomic absorption.

1.2 Metals in solution may be readily determined by atomic absorption spectroscopy. Their procedures are applicable to a large number of metals in drinking waters, industrial waste, treatment process control, urban stormwater runoff, and environmental health monitoring samples.

1.3 Metals analytical technique and method detection limits of this procedure are shown in Attachment 1.1.

1.4 For reference where this method is approved for use in compliance monitoring programs consult both the appropriate sections of the Code of Federal Regulation (40 CFR Part 136 Table 1B for NPDES, and Part 141 section 141.23 for drinking water).

1.5 All flame analyses will be conducted according to Method 3111B--Direct Flame Method Standard Methods for the Examination of Water and Wastewater 18th Edition.

1.6 All furnace analyses will be conducted according to Method 3113B-- Furnace Standard Methods for the Examination of Water and Wastewater 18th Edition. Reference EPA Method 200.9 Determination of Trace.

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DETECTION LIMITS BY METHOD

<u>ELEMENT</u>	<u>FLAME</u>	<u>FURNACE</u>	<u>ICP</u>	<u>FIMS</u>
(Al) Aluminum			< 10 ug/l	
(Sb) Antimony		< 2 ug/l		
(As) Arsenic		< 2 ug/l		
(Ba) Barium			< 1 ug/l	
(Be) Beryllium			< 1 ug/l	
(B) Boron			< 40 ug/l	
(Cd) Cadmium	< 20 ug/l	< 0.1 ug/l		
(Ca) Calcium			< 0.05 mg/l	
(Cr) Chromium	< 10 ug/l	< 1 ug/l		
(Co) Cobalt			< 5 ug/l	
(Cu) Copper	< 5 ug/l		< 5 ug/l	
(Fe) Iron			< 5 ug/l	
(Pb) Lead	< 20 ug/l	< 2 ug/l		
(Li) Lithium			< 0.05 mg/l	
(Mg) Magnesium			< 0.05 mg/l	
(Mn) Manganese			< 1 ug/l	
(Hg) Mercury				< 0.5 ug/l
(Mo) Molybdenum			< 25 ug/l	
(Ni) Nickel	< 20 ug/l		< 5 ug/l	
(K) Potassium			< 1 mg/l	
(Se) Selenium		< 2 ug/l		
(Si) Silicon SiO ₂			< 0.5 ug/l	
(Ag) Silver	< 5 ug/l	< 0.05 ug/l		
(Na) Sodium			< 0.10 mg/l	
(Sr) Strontium			< 1 ug/l	
(Tl) Thallium		< 2 ug/l		
(V) Vanadium			< 5 ug/l	
(Zn) Zinc	< 5 ug/l		< 5 ug/l	

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2.0 SUMMARY OF PRINCIPLES & PROCEDURES

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2.1 Atomic absorption is the process that occurs when a ground state atom absorbs energy in the form of light of a specific wavelength and is elevated to an excited state. The amount of light energy absorbed at this wavelength will increase as the number of atoms of the selected element in the light path increases. The relationship between the amount of light absorbed and the concentration of analyte present in known standards can be used to determine unknown concentrations by measuring the amount of light they absorb.

2.2 There are five basic components of an atomic absorption instrument (Fig 2.1):

1. The light source (hollow cathode lamp or electrodeless discharge lamp) that emits the spectrum of the element of interest
2. An "absorption cell" in which atoms of the sample are produced (flame, graphite furnace, FIMS cell)
3. A monochromator for light dispersion, isolating the specific wavelength of light to be used
4. A detector, which measures the light intensity and amplifies the signal
5. A display that shows the reading after it has been processed by the instrument electronics

The light from the source lamp is divided into a sample beam, which is focused through the sample cell, and a reference beam, which is directed around the sample cell. In a double-beam system, the readout represents the ratio of the sample and reference beams.

2.3 Since atoms absorb light at very specific wavelengths, it is necessary to use a narrow-line source which emits the narrow-line spectra of the element of interest. The main sources used for atomic absorption are the hollow cathode lamp (HCL) and the electrodeless discharge lamp (EDL). The hollow cathode lamp is an excellent, bright, stable line source for most elements. However, for some volatile elements, where low intensity and short lamp lifetime are a problem, EDL's are available. EDL's are typically more intense than HCL's, therefore, may offer better precision and lower detection limits for some elements.

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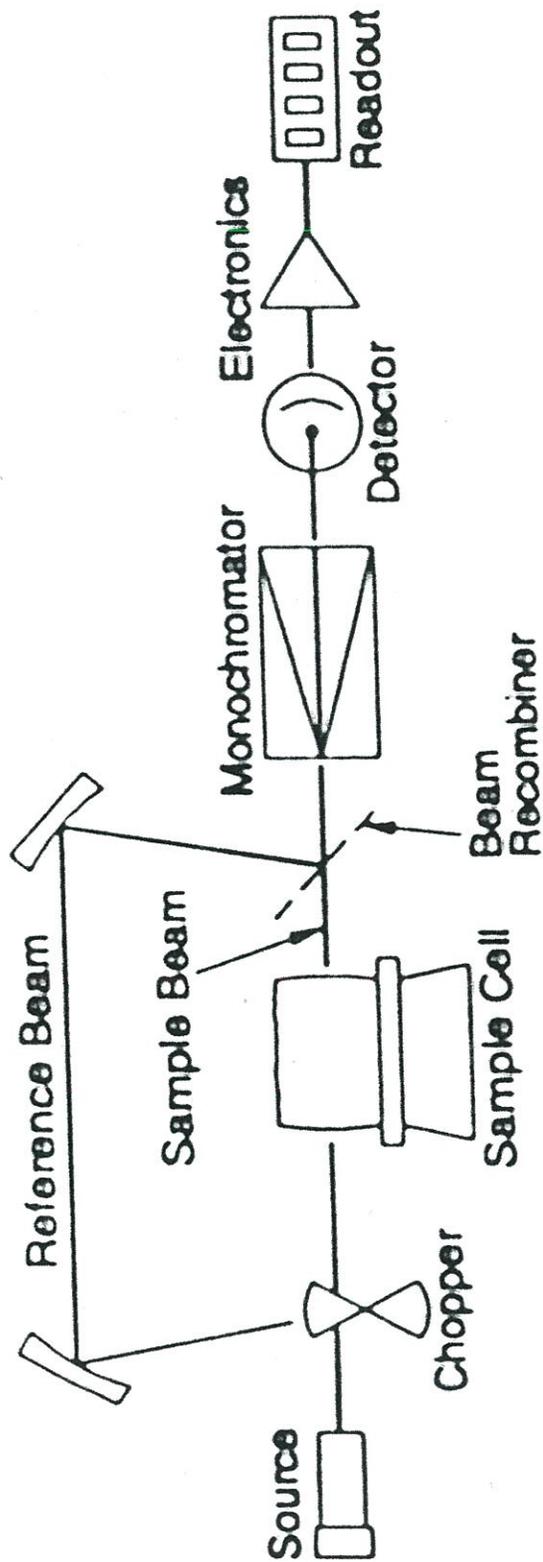


Figure 2.1 Double-Beam Atomic Absorption Spectrometer

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3.0 DEFINITION OF TERMS

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3.1 Accuracy: Combination of bias and precision of an analytical procedure, which reflects the closeness of a measured value to a true value.

3.2 Analytical Batch: Samples which are analyzed together with the same method sequence and the same lots of reagents and with the same manipulations common to each sample within the same time period. The samples are of the same TYPE and the batch will be designated by sample type, protocol, revision#, year, the actual number of batches since the beginning of the current year, and sample collection beginning and ending dates. **A batch consist of 20 samples or less of the same WQL protocol.**

3.3 Calibration Blank (Cal Blk): A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to auto-zero the instrument.

3.4 Continuing Calibration Verification Standard (CCVS): A mid-range ICAL standard used to verify continuing calibration. CCVS standards are used to verify instrument performance on a daily basis.

3.5 Control Charts: Charts used to maintain a current awareness of the accuracy/precision of each method and that the default acceptance criteria in the methods be updated to properly reflect the capability of the laboratory.

3.6 Correlation Coefficient (CC): Is a number in the range -1 through +1 that measures how closely the calculated line fits the data. All calibration curves must have a calculated CC of greater than or equal to 0.995.

3.7 Dissolved Analyte: The concentration of analyte in an aqueous sample that will pass through a 0.45 μ m membrane filter.

3.8 Field Duplicate (FD): A field duplicate sample that is exposed to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FD is to determine if method analytes or other interferences are present in the field.

3.9 Field Reagent Blank (FRB): An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to the sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

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3.10 Initial Calibration Standard (ICAL): A solution prepared from the dilution of stock standard solutions. The ICAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.11 Initial Calibration Verification Standard (ICVS): A standard of known concentration obtained from an second source vendor, the same source but different lots, the same lot prepared by a second analyst, or prep on a different day. ICVS standards are used to verify the calibration standards.

3.12 Instrument Detection Limit (IDL): The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the same wavelength.

3.13 Instrument Performance Check (IPC): A specific test to evaluate the performance of the instrument.

3.14 Laboratory Fortified Blank (LFB)=Laboratory Control Sample (LCS): A reagent water blank to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

3.15 Laboratory Duplicates (DUP): Two aliquot of the same sample taken in the laboratory and analyzed separately with identical procedures. The DUP indicates precision associated with laboratory procedures, but not with samples collection, preservation, or storage procedures.

3.16 Laboratory Fortified Sample Matrix (LFM)=Matrix Spike (MS): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes positive or negative bias to the analytical results.

3.17 Laboratory Fortified Sample Matrix Duplicate (LFM)=Matrix Spike Dup (MSD): Two aliquot of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of MS and MSD indicates precision associated with laboratory procedure.

3.18 Laboratory Reagent (Method) Blank (LRB): A reagent water blank carried through the entire analytical sample preparation procedure and is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and apparatus. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.19 Linear Range: The concentration range over which the analytical curve remains linear. Determined in the initial method development and performance demonstration.

3.20 Method Detection Limit (MDL): 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 an analysis of a sample in a given matrix containing the analyte.

3.21 Precision: A measure of the degree of agreement among replicate analyses of a single sample or duplicate usually expressed as the standard deviation or % difference.

3.22 Proficiency Evaluation Samples (PE): EPA water studies and in house quality control using secondary reference materials provided by QA Manger. The purpose of PE sample is to evaluate the laboratory performance and analyst performance.

3.23 Quality Assurance: A definite plan for laboratory operation that specifies the measures used to produce data of known precision.

3.24 Quality Control: Set of measures within a sample analysis methodology to assure that the process is in control.

3.25 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using standards purchased from a commercial source.

3.26 Replicate: Repeated operation occurring within an analytical procedure. Minimum of three data points for each method of analysis is required.

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4.0 INTERFERENCES

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Atomic absorption is a very specific technique with few interferences. The interferences that do exist fall into six categories: chemical interferences, ionization interferences, matrix interferences, emission interferences, spectral interferences, and background absorption.

4.1 Chemical Interferences:

The most common interferences in atomic absorption are chemical interferences. If the sample being analyzed contains a thermally stable compound with the analyte that is not totally decomposed by the energy of the flame a chemical interference exists. As a result, the number of atoms in the flame capable of absorbing light is reduced.

Chemical interferences can normally be overcome or controlled in two ways: the use of a high temperature flame or the addition of a releasing agent to the sample or standard solution. A releasing agent, or competing cation, when added to the sample solution will preferentially react with the interferant releasing the analyte and thus removing the interference. A higher temperature flame will provide additional energy to break down a compound which is stable in a lower temperature flame.

4.2 Ionization Interferences:

Ionization interferences occur when the flame temperature has enough energy to cause the removal of an electron from the atom, creating an ion. As these electronic rearrangements deplete the number of ground state atoms, atomic absorption is reduced.

Ionization interferences can be controlled by the addition of an excess of an easily ionized element to the blank, standards, and samples.

4.3 Matrix Interference:

Matrix interferences can cause either a suppression or enhancement of the analyte signal. Matrix interferences occur when the physical characteristics (viscosity, burning characteristics, surface tension) of the sample and standard differ considerably. This can happen when the sample solution contains a high concentration of dissolved salts or acid, when different solvents are used for sample and standard solutions, or when the sample and standard solutions are at radically different temperatures.

To compensate for matrix interferences, try to match as closely as possible the matrix components in the sample, standard, and blank. Any reagent added to the samples during preparation should also be added to the standards and the blank.

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4.4 Emission Interference:

At high analyte concentrations, the atomic absorption analysis for highly emissive elements sometimes exhibits poor analytical precision, if the emission signal falls within the spectral bandpass being used.

There are several ways to compensate for this interference, including decreasing the slit width, increasing the lamp current, diluting the sample, and using a cooler flame.

4.5 Spectral Interference:

A spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the bandwidth of the absorption line of the element of interest. The results of the determination will then be erroneously high, due to the contribution of the interfering element to the atomic absorption signal.

This can be overcome by using a smaller slit or selecting an alternate wavelength.

4.6 Background Interferences:

There are two causes of background absorption: light scattering by particles in the flame and molecular absorption of light from the lamp by molecules in the flame.

To compensate for this problem, the background absorption must be measured and subtracted from the total absorption measured to determine the atomic absorption signal. Background absorption is to use a background corrector, which utilizes a continuum source (a deuterium arc lamp in the ultraviolet wavelength). A continuum source emits light over a broad spectrum of wavelengths instead of at specific lines. With background correction, simultaneous compensation is obtained at the same wavelengths used to measure atomic absorption. With this system, light from the primary source and the continuum source are passed alternately through the flame. The element being determined effectively absorbs light only from the primary source, while background absorption affects both beams equally. Therefore, when the ratio of the two beams is measured electronically, the effect of the background absorption is eliminated (see Figure 4.1). Background correction may be used for Nickel and Lead analysis, due to chloride interference at that wavelength for those samples suspected of containing high chlorides.

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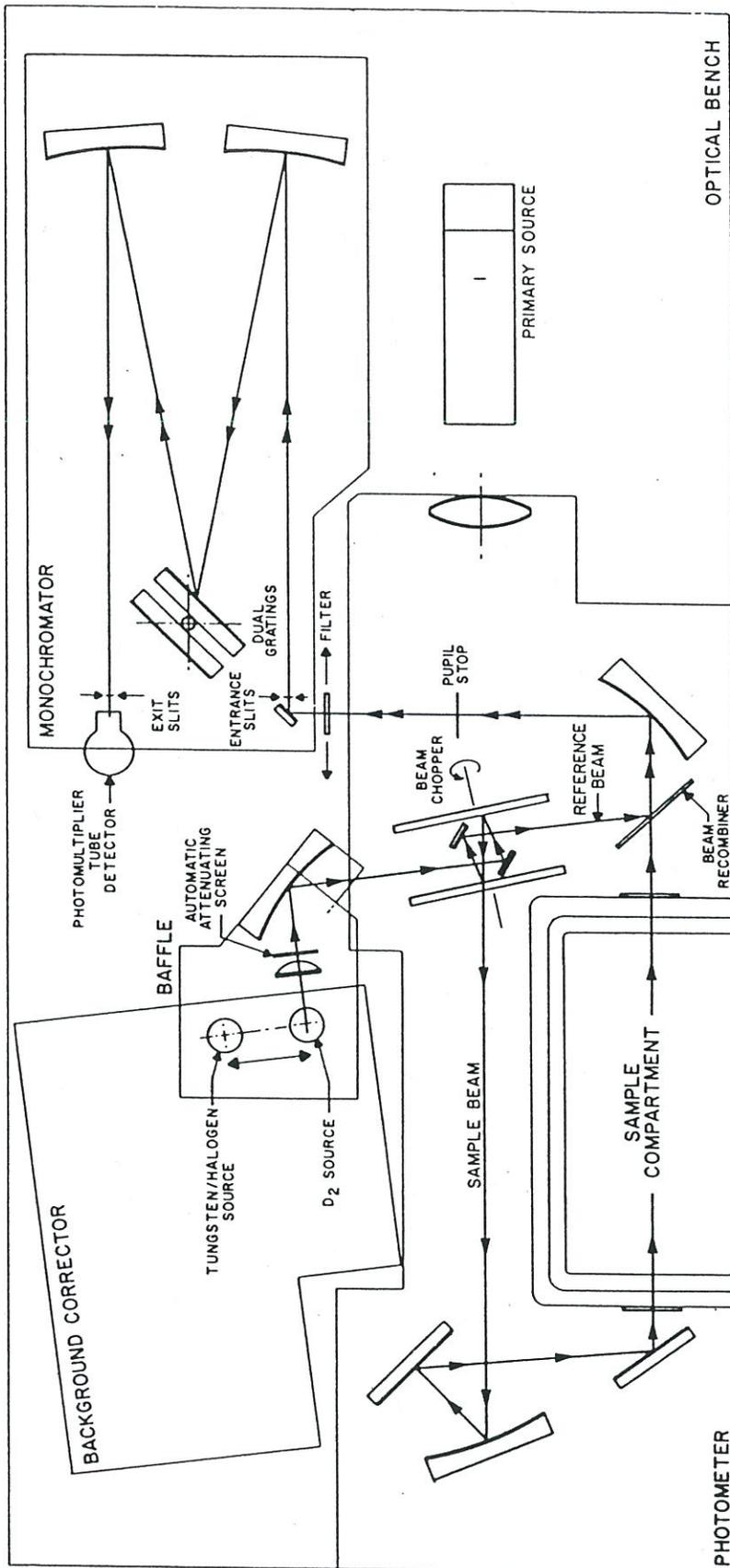


Figure 4-2 - Model 5000 Optical Schematic, BG Mode.

5.0 SAFETY

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5.1 The Chemical Hygiene Policy (CHP) establishes procedures to protect City of Albuquerque laboratory employees from the health hazards presented by chemicals used at the Water Quality Laboratory. The CHP has also been developed to comply with the Occupational Safety and Health Administration (OSHA) regulation. It is required for all analysts to read and study all aspects of the CHP. proficiency and exams will be issued by the Chemical Hygiene Committee annually to ensure the CHP procedures are followed.

5.2 Material Safety Data Sheets (MSDS) for all standards and chemical reagents are available in the metals laboratory office. It is mandatory that all analysts read all MSDS sheets before beginning analysis.

5.3 Venting system is required to remove the fumes and vapors from the flame for atomic absorption instruments. Vents are checked monthly by Chemical Hygiene Officers. Exhaust Venting is important for three reasons:

- A. It will protect laboratory personnel from toxic vapors which may be produced.
- B. It will improve the stability of the flame and tend to remove the effects of room drafts and the laboratory atmosphere.
- C. It will help to protect the instrument from corrosive vapors.

Caution: The escape of fuel gas mixture from the drain tube, the burner system, the gas control, or the gas connections result in a serious explosion hazard. Make sure there are no breaks or leaks in any of these systems. If a leak is suspected lockout/tagout procedures will be followed and a Perkin-Elmer representative will be called.

5.4 Safe Handling of Chemicals:

- Wear appropriate eye protection at all times while handling chemicals.
- Wear suitable protective clothing, including gloves.
- Perform sample/standard preparation away from the instrument to minimize corrosion and contamination.
- Clean up spills immediately.
- Do not put open containers of solvent near the instrument.
- Store solvents in an approved cabinet away from the instrument.

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5.5 High Temperatures:

Burner System

The flame can generate temperatures of up to 2800 C.

- Do not touch the burner head until it has cooled to room temperature.

Graphite Furnace

The THGA graphite furnace can generate temperatures of up to 2600 C

- Do not touch any part of the graphite furnace until it has cooled to room temperature.
- Do not attempt to inject a sample into the graphite tube when the furnace is hot. You may be subjected to fumes from the sudden vaporization of the sample, and may damage the pipet.

5.6 Storing Cylinders:

- Acetylene is dissolved in acetone because it cannot be compressed as a dry gas to greater than about 30 psi. Tanks will be replaced when the cylinder drops to 100 psig. Failure to change the acetylene cylinder below 100 may cause damage to valves or tubing within the burner gas control system due to high acetone carryover.
- Gas cylinders are stored in the gas storage room, make sure that ventilation is adequate to prevent toxic or explosive accumulations of gas.
- Do not allow ignition sources in the storage area and keep cylinders away from readily ignitable substances such as gasoline or waste, or combustibles.
- Store all gas cylinders only in a vertical position, with the valve cap in place, and fastened securely to an immovable bulkhead.

5.7 Safe Operation of the Flame:

- Before you ignite the flame make sure that:
 - ▶ The laboratory fume ventilation system is operating
 - ▶ The burner head is installed correctly
 - ▶ The burner end cap is secured firmly
 - ▶ The fuel and oxidant tubing fittings are properly connected
 - ▶ The atomizer compartment door is closed
- Do not leave the flame unattended. Always make sure that there is a fire extinguisher near the instrument.
- Never change the gas pressure or shut a gas valve while the flame is burning.
- Do not allow the burner head slot to become blocked. This can cause a flashback of the flame.
- Do not place open containers of flammable liquids and solvents near to the flame. Be especially careful with samples that contain highly volatile solvents.
- When you have analyzed samples containing organic solvents, flush all traces of solvent from the burner system.

5.8 The Drain System:

- Always place the waste container in a well ventilated place underneath the spectrometer, in full view while you are working with the spectrometer. This prevents the build-up of potentially hazardous gases, and allows you to see the liquid level.
- Never use a glass waste container.
- Do not kink, fold, or loop the drain tube.
- Never place the drain tube directly into a laboratory sink.
- Empty the waste container frequently; especially do not allow the waste container to fill with organic solvent.
- Thoroughly flush the drain system when you shut down the system.
- Check the condition of the drain tube regularly. Replace the drain tube when it first shows signs of cracking or discoloration.

5.9 Flashbacks:

A flashback is an explosion of the premixed gases in the spray chamber. The safety interlocks normally prevent conditions that could cause a flashback, but if a flashback should ever occur:

- Check that the burner head slot is clean.
- Make sure that you are using compressed air and not oxygen-enriched air.
- Check the flow spoiler or impact bead for damage, and replace any damaged parts.
- Carefully check and tighten all connections to, and seals on, the burner unit, and check the gas flow settings before you attempt to ignite the flame. Seepage of fuel and oxidant from the spray chamber may cause a flashback.
- Check that the burner head is seated correctly.

5.10 Employees will attend bi-weekly safety meetings.

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Rules for Handling Compressed Gases

1. Always use a hand truck for transport. Chain cylinder to hand truck.
2. Do not transport in closed vehicles.
3. Cylinders should be chained in place or otherwise secured at all times.
4. Do not drop cylinders, or otherwise permit them to strike each other.
5. Leave valve cap on cylinder until secured and ready for use.
6. Ground all cylinders containing flammable gases.
7. Use only in an upright position.
8. All valves should be closed when not in actual use.
9. Use the proper regulator for the particular gas.
10. Carefully open all valves and adjust gas flow rates.
11. Always consider cylinders to be full and handle accordingly.
12. Discontinue using a high pressure cylinder when the pressure approaches 30 psig, and clearly mark EMPTY, then remove for return to vendor.

Handling

Rules for handling of compressed gases are given at the left. The rules for handling employ a good deal of common sense, a useful tool in safe work habits. Follow the rules. Sloppy technique in handling hazardous gases could result in serious injury, death, and severe property damage.

6.0 APPARATUS & EQUIPMENT

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6.1 Atomic absorption spectrometer:

Features of the AAnalyst 800 System:

- Two fully intergrated atomizers: a burner system for flame atomization and a graphite furnace for electrothermal atomization. Atomizer changeover is automatic at the click of a mouse button.
- Transversely-heated graphite furnace featuring full Stabilized Temperature Platform Furnace (STPF) technology.
- Longitudinal Zeeman-effect background correction for electrothermal atomization.
- Deuterium background correction for flame atomization.
- Automated self-optimization of the burner system.
- High capacity furnace autosampler.
- Easy-to-use AA WinLab software.

System compents:

- The spectrometer, containing the optical system, the electronics, and the atomizer compartment.
- The burner system, mounted on a carriage that can be driven into and out of the atomizer compartment by software command (see 'Burner system design' in the 'Burner System User's Guide' for more information).
- The graphite furnace, mounted on a carriage that can de driven into and out of the atomizer compartment by software command (see 'Furnace design' in the 'Graphite Furnace User's Guide' for more information).
- The furnace autosampler, mounted on a swing arm in front of the atomizer compartment.
- The furnace cooling system.
- The computer and the associated AA WinLab software.

6.2 Burner System: The burner system includes a flow spoiler molded of polypropylene and held in position by three support arms which press-fit to the walls of the mixing chambers.

6.3 Furnace Gases: The graphite furnace requires a supply of inert gas to prevent the tube and the analyte atoms from being oxidized when the tube is heated. Argon is the inert gas used.

6.4 Cooling the Spectrometer System:

- **Furnace cooling:** The graphite furnace uses water cooling to allow the graphite tube to return to its equilibrium temperature quickly between measurements.
- **Spectrometer cooling:** Cooling coils are incorporated in the spectrometer's power supply to maintain the electronic components at a comfortable working temperature.

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- **Cooling system:** PE offers a recirculating cooling system; it provides coolant at a constant temperature and flow, to allow the most reproducible analytical conditions.

6.5 Lamps: Single element hollow cathode lamps (HCL) will be used. All lamps will be ordered from Perkin-Elmer. For Lead analysis an electrodeless discharge lamp (EDL) will be used, which offers better precision and lower detection limits.

6.6 Air Filter Assembly: A filter system specifically designed to remove water, oil, and dirt particles from compressed airlines.

6.7 Vent: Placed 15-30 cm above the burner to remove fumes and vapors from the flame. This precaution protects laboratory personnel from toxic vapors, protects the instrument from corrosive vapors, and prevents flame stability from being affected by room drafts. All ventilation systems in the Water Quality Lab are checked monthly by Chemical Hygiene Officers.

6.8 Labware: All reusable labware should be sufficiently clean (See Section 8.1.2 for proper instructions) for the task objectives.

- Glassware Volumetric flasks Class A
- Calibrated Pipettes Class A
- 15ml Conical Centrifuge Tubes Polypropylene Metal Free

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7.0 REAGENTS AND STANDARDS

7.1 Air: House air, which is dried and filtered, is delivered from a compressor located in the Mechanical Room-WQL. An air filter assembly is also added to the system before delivery to the Analyst 800. It must be cleaned and dried through a suitable filter to remove oil, water, and other foreign substances. Filter refills must be replaced yearly. Part No. PE N930-2196.

7.2 Acetylene: Grade 5, Ultrapure acetylene will be used. All replacement cylinders must be ordered through the plant warehouse. Product No. 9377.

7.3 1% HNO₃: Use 1% HNO₃ for the preparation of all reagents, calibration standards and as dilution water. Also use for rinse samples during analysis, Auto Zero blank samples, and rinse vessel.

7.4 Initial Calibration Standards (ICAL): Prepare a series of standard metal solutions in concentration ranges listed below. Follow method procedures in Flame Standards Logbook, record date prepared, lot number, analyst, expiration date, and reference # in logbook. Due to the variability of linear ranges of the element analyzed the following standards will be prepared:

A.)	Ag	
	S1	1000ppb
	S2	2000ppb
	S3	4000ppb
B.)	ICAL/S1	Cd/Cr/Ni/Zn: 500ppb
	ICAL/S2	Cd/Cr/Cu/Ni/Zn 1000ppb
	ICAL/S3	Cd/Cr/Cu/Ni/Zn: 2000ppb
	ICAL/S4	Cu 5000ppb
C.)	Pb	
	S1	5ppm
	S2	10ppm
	S3	20ppm

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Note: All standards will be prepared every two weeks. Standards may need to be prepared weekly if 3 pt calibration is not achievable.

7.5 Initial Calibration Verification Standard (ICVS): The ICVS sample should be midrange of the calibration curve. Also, the ICVS samples should be prepared using a supply from a second vendor, same vendor different lot, prepared by a different analyst, or same analyst different day. Follow all method procedures, record date prepared, lot number, analyst, expiration date and reference # in the Flame Standards Logbook. Must be prepared every two weeks.

- | | | |
|-----|-------------|--------------|
| A.) | Ag | ICVS 2000ppb |
| B.) | Cu | ICVS 2000ppb |
| | Cd/Cr/Ni/Zn | ICVS 1000ppb |
| C.) | Pb | ICVS 10.0ppm |

7.6 Continuing Calibration Verification Standard (CCVS): All S2 standards will be used to verify instrument performance.

COPY NO. 01
DATE 1/9/09
QA *[Signature]*

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