BUCK SCIENTIFIC

210 / 211VGP ATOMIC ABSORPTION SPECTROPHOTOMETERS 220GF GRAPHITE FURNACE & 220AS AUTOSAMPLER

OPERATOR'S MANUAL
February 2005 VER 3.94 C

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Buck *Model 210 VGP* Atomic Absorption Spectrophotometer

INTRODUCTION

The Buck 210VGP atomic absorption spectrophotometer is designed to measure the concentration of elemental metals in solution. It provides integrated measurements in absorbance or emission intensity, as well as sample concentration in comparison to standard solutions. The readings can be integrated over a period from 0.5 to 10 seconds. For the transient signals obtained using flame-less techniques (cold vapor, hydride, and graphite furnace), absorbance is determined using peak area integration.

The Buck 210VGP can be calibrated using up to 8 concentration values, with units of *mg/L, µg/L, ppm, ppb, mEq/dL, mM/dL,* µg/dL and *%*. Calculations can be performed using linear regression, 2nd, 3rd, and 4th order curves. Optional accessories allow the user to print absorbance data, background absorbance and concentrations in real time, and to collect absorbance values using a laboratory recorder. The Buck 210VGP interfaces with the *Analyze* software package for sophisticated data processing, documentation and reporting. An external computer may also process the data by using a communications program to download the data into a spreadsheet program.

SAFETY

The methods and analytical procedures described in this manual are designed to be carried out by properly trained personnel in a suitably equipped laboratory. In common with many laboratory procedures, the methods described may involve hazardous materials or substances of unknown toxicity. For the correct and safe execution of the methods, it is essential that laboratory personnel follow standard safety procedures for the handling of hazardous materials.

While the greatest care has been exercised in the preparation of this information, Buck Scientific, Inc. expressly disclaims any liability to users of the procedures for consequential damages of any kind arising out of or connected with the use of these procedures.

For specific safety information, refer to the OSHA documentation on hazardous materials handling and procedures, and consult the Material Safety Data Sheet (MSDS) for the chemicals with which you are working. By law, MSDS sheets must be made available by the company which manufactures the chemicals you are using.

Direct all inquiries regarding this manual and/or the 210VGP Atomic Absorption Spectrophotometer to your Buck Scientific Sales Representative or to:

Buck Scientific, Inc.
58 Fort Point St.
East Norwalk, Ct. 06855

Tel: 203-853-9444 or 1-800-562-5566
Fax: 203-853-0569
E-mail: quality@bucksci.com www.bucksci.com

SECTION 1: Installation

GENERAL SPECIFICATIONS

Electrical:

110V AC nominal (+10%), 50/60 Hz
220, 240V AC, 50/60 Hz
Power Consumption: 50W

Optics:

Detector: model 928; wide range general purpose, 190-930nm
Optional Detectors: model 955; UV enhanced, wide-range, 190-930nm
model EMI9783B; narrow range furnace/hydride application, 165-600nm
Lenses: Supracil - amorphous silica
Monochromator: 0.25m Ebert mount
Grating: 32nm x 27nm; 600 grooves/mm
Wavelength adjustment: 3 digit mechanical, 0 to 1000nm +1 nm
Reproducibility: +0.2 nm
Resolution: variable slit - 2Å, 7Å, and 20Å

Operating Modes:

Absorbance/Emission: -0.0820 to 3.2000
Concentration: to 5 significant digits
Integration Period: 0.5 to 10 seconds
Screen Refresh : 0.5 to 1.5 seconds
Recorder Output: 1V/ABS (-0.08 to 3.2V)
Background Correction: In-line Deuterium Arc
Giant Pulse (Self-reversal)

Hollow Cathode Lamps:

Dimension: 1.5" OD
Striking Voltage: 500V
Lamp Current: 0 to 18 mA average current
Duty Cycle: 25%
Modulation Frequency: variable; 33 to 200 Hz (142 Hz Norm.)

Burner Assembly:

Design: Polyethylene Pre-mix chamber, glass impact bead dispersion
Burner Head: Titanium; air-acetylene head - 4" x 0.026" single slot
(nitrous oxide head - 2" x 0.019" single slot)
Adjustments: Horizontal g

Performance:

Average Noise (at 3σ): 0.0018 ABS (Cu at 324.7nm, 7Å slit, 5 sec. int.)
Sensitivity: see specific element chart (Sect. 4)
Reproducibility: <+5%

SECTION 1: Installation - Gas Supplies

GAS SUPPLIES: *(This material was extracted from uncopyrighted information provided by the Scientific Apparatus Makers Association****, No. Al 2.1)***

Acetylene:
For the majority of analysis, commonly available welder's grade acetylene is the required fuel for use with the model 210VGP. Acetylene is usually obtained in size 1B cylinders containing about 9000 liters (at STP) of gas dissolved in acetone. An air-acetylene flame consumes about 5 liters per minute, whereas a nitrous oxide-acetylene flame consumes about 15 liters per minute. Consequently, a single 1B cylinder will give from 10 to 30 hours of operation, depending on the amount of N_2O to air used. Acetylene cylinders utilize a CGA510 two-stage regulator (part no. BS303-0106) with a flash arrestor (part no. 6103A).

Acetylene is dissolved in acetone in order to prevent explosive decomposition when compressed to greater than about 30 psi. To provide a margin of safety, acetylene should not be used above 15 psi line pressure. As acetylene is removed from the cylinder, acetone vapors accompany it in increasing proportion as tank pressure falls. Since acetylene is in solution, the pressure drop is not linear, and a pressure of 75 psi indicates a nearly empty tank. For some elements, absorption sensitivity will change as the amount of acetone increases, so it is a good practice to replace the cylinder when the pressure falls to between 75 and 100 psi. Running the instrument with the pressure below 75 psi may result in acetone getting into the instrument and damaging flow meters or gas controls.

Special Cautions: Acetylene forms unstable acetylides if it comes in contact with copper, silver or mercury. Wet acetylene forms explosive acetylides with copper. The rate of acetylide formation increases in the presence of air or carbon dioxide. ***Pure copper should never be used for acetylene plumbing***. Acetylides are formed much less rapidly on brass than copper.

Hydrogen:
In certain applications hydrogen is used as the fuel, usually obtained in 1A cylinders containing about 5500 liters (at STP). The extra dry grade (99.9+% purity) is suitable for atomic absorption work. An air-hydrogen flame requires about 15 liters per minute, which represents about 6 hours of operation for a 1A cylinder. The air-hydrogen flame is invisible under normal circumstances, and extra caution should be excersized when using this flame. Do not place hand on or lean over a burner where hydrogen is being used in case the flame is actually lit. Always test first by placing a shiny surface above the region of combustion; the formation of water vapor indicates that the flame is lit. Hydrogen cylinders are used at high pressure and should be handled with care at all times. They are operated at 40psi using a CGA350 two-stage regulator (part no. BS303-0265).

Air:
Air is the most common oxidant and can be obtained from either a compressed air cylinder or from a compressor unit. A standard 1A air cylinder contains about 6200 liters (at STP). The 210VGP premix burner-nebulizer will use about 20 liters per minute, and therefore one cylinder will last about five hours. Occasionally cylinder air has gone through a liquefication stage during which the oxygen to nitrogen ratio can change, and it is not uncommon to find other than 20% oxygen in air cylinders. This can be a potential safety hazard, and cause variable burner performance and analytical results. Medical grade or Breathing Air is often compounded from Nitrogen and Oxygen to contain greater than the normal Oxygen content of Air and are not recommended. General Purpose Compressed Air is suitable instrument use. Air cylinders are operated at 50psi utilizing a CGA590 two-stage regulator (part no. BS303-0264).

Because of the limitations inherent in using compressed air cylinders, an oil-less air compressor is usually used. The compressor should provide at least 30 liters per minute at 50 psi, with a water and oil trap installed between the compressor and the 210VGP. Buck Scientific supplies a suitable compressor (part no. BS303-0313) and filter assembly (part no. BS303-0229).

Nitrous Oxide:

Nitrous Oxide is usually obtained in 1A cylinders containing about 15,000 liters (at STP). The N_2O is in the liquid state, at an initial pressure of about 750 psi. Because of this, the pressure gauge does not indicate how much liquid remains until the pressure starts to fall off rapidly as residual gas is withdrawn. A nitrous oxide-acetylene flame consumes about 20 liters per minute of N_2O at 50 psi; therefore a single 1A cylinder will last about 12 hours.

When N_2O is removed from the cylinder at this rate the expanding gas cools the diaphragm of the regulator so that sometimes it freezes, causing loss of regulation. It is therefore advisable to use a Ambient Air Heated Regulator (part no. BS303-0204). All lines carrying N_2O should be free of grease, oil or other organic material, as it is possible for spontaneous combustion to occur. Cylinders of N_2O should be considered high pressure cylinders and handled with care at all times.

Argon:

Argon is usually obtained in size 1A cylinders containing about 7000 liters (at STP). It is used with a CGA580 two-stage regulator (part no. BS303-0264). Argon is generally used in conjunction with hydrogen as a flame diluent to provide a cool flame, as a purge gas in the analysis of hydride-forming metals, and also as a sheathing gas in graphite furnace work. Consequently the consumption rate varies widely, depending on the particular application. High purity grade (99.995%) Grade Argon is good for most analyses. Argon in the pre-purified grade (99.998%) can also be used but is not recommended for the Graphite Furnace since the grade often contains a small amount of oxygen which may shorten tube life. Argon cylinders are used at high pressure and should be handled with care at all times.

Nitrogen:

Nitrogen is usually obtained in size 1A cylinders containing about 6500 liters (at STP). It is used with a CGA580 two-stage regulator (part no. BS303-02640). Nitrogen is used similarly to argon, and therefore the consumption rates vary widely with the application. The high purity grade (99.9%) or the extra dry grade (99.7%) is suitable for atomic absorption work. Nitrogen cylinders are used at high pressure and should be handled with care at all times.

SECTION 1: Installation - Preparing the Lab

This section gives details concerning the space and accessories required to set-up the Buck 210VGP atomic absorption spectrophotometer. The spectrophotometer is supplied with the following materials:

1. Nylon Gas Lines for Fuel, Air and Nitrous Oxide
2. Tygon Drain Tubing
3. 0.023" OD Nebulizer Tubing
4. Igniter
5. Maintenance Tools

Equipment to be Provided by the Analyst

The following lists the equipment and materials that you will need to operate the 210VGP. Many of these materials may have been supplied as options with your 210VGP. They can also be purchased from your Buck Scientific Sales Representative, and are shown with part numbers for your convenience.

1. Exhaust Vent (part # BS303-0407)
2. Standards, matrix modifiers & acids for the elements to be determined
3. One (110/120 v-15 amp) outlet for instrument. Add one outlet each if you will be using a printer, computer and autosampler.
4. Distilled or deionized water.
5. 3' X 5' lab bench area.
6. Drain Vessels for Waste fluids (no glass)
7. Hollow Cathode Lamps for elements to be determined (*see catalogue for part # 's)*

For general flame analysis...

1. Size 1A compressed air cylinder (General Purpose) & regulator CGA-590 (part # BS303-0264) **or** oil-less air compressor (part # BS303-0313) & filter assy (part # BS303-0229)
2. Size 1B acetylene cylinder (welding grade) & two stage regulator CGA-510 (part # BS303-0106)
3. Flash arrestor for acetylene tank (part # 6103A)
4. Size 1A nitrous oxide cylinder (if you will be doing N20 determinations) & Ambient-Air heated regulator (part # BS303-0204).

For As & Se flame analysis...

1. Size 1A hydrogen cylinder & regulator CGA-350 (part # BS303-0265).
2. Size 1A argon cylinder & regulator CGA-580 (part # BS303-0264).

For graphite furnace analysis...

1. Size 1A argon or nitrogen cylinder & regulator CGA-580 (part # BS303-0264) (argon recommended)
2. One (208/240v-30amp-3 wire) single phase outlet.
3. Cooling water supply & drain or a temperature controlled recirculating unit (chiller). The water source should should be regulated to supply 10-15 psi and a flow of 1½ - 2 Liters per minute. Chiller Temperatures to be 18-20 C.

Note: All regulators & water supplies must be ready to accept a 1/4" swagelock nut for installation.

Note: If performing all 3 types of analysis, you only need one tank of any one gas.

SECTION 1: Installation - Basic Instrument and Flame

Suitable Work Area

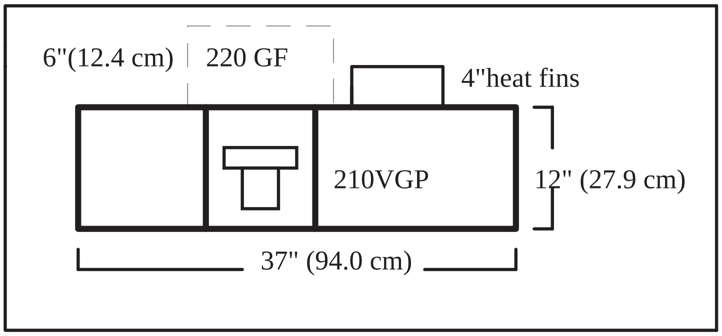
For best performance, the 210VGP should be located in a well-ventilated room, free of dust, draughts, and corrosive fumes and vapors. Because it must be vented through an exhaust duct, it is best to locate the instrument near an external wall, or close to other duct lines that are used for similar purposes. It is sometimes possible to tie the exhaust vent into a laboratory fume hood exhaust line. A backflow preventer should be installed between the 210VGP exhaust line and the fume hood line to prevent cross-contamination.

Locate the spectrophotometer on a workbench or table large enough to accommodate the instrument, samples and all accessories. The recommended dimensions are shown in the diagram below. The workstation should be flat, sturdy and free of vibration.

Model 210 VGP Dimensions as shown
Minimum Rear Clearance: 4"

Recommended Table Dimensions:
48"W x 24"D x 30"H (28" deep with model 220AS autosampler)

NOTE: model 220 GF graphite furnace:
add 4" to minimum rear clearance (8" total)
Dimensions: 12"W x 6"D x 18"H



The laboratory environment should be regulated to provide stable temperature and humidity. The 210VGP should be maintained at temperatures from 10° to 32°C (50° - 90°F), and relative humidity of 30% to 80%.

In many applications data handling may be accomplished using an external printer, the ***Aanalyze*** software package from an IBM style PC or laptop computer, a laboratory recorder, or all three. These accessories should be located near the instrument for easy access. It is advisable to place the computer and printer on a separate workbench or table on the right hand side of the 210VGP, since the external connections are made on that side of the instrument. This will prevent the cables from having to cross over the gas lines, and make them more accessible.

SECTION 1: Installation *Continued*

Ventilation

The combustion of metallic and organic compounds can produce toxic vapors, as well as extreme heat. In order to protect the analyst and maintain a safe, clean laboratory environment, a permanent vent should be installed. The ventilation system must meet the specifications listed in Table 1 below.

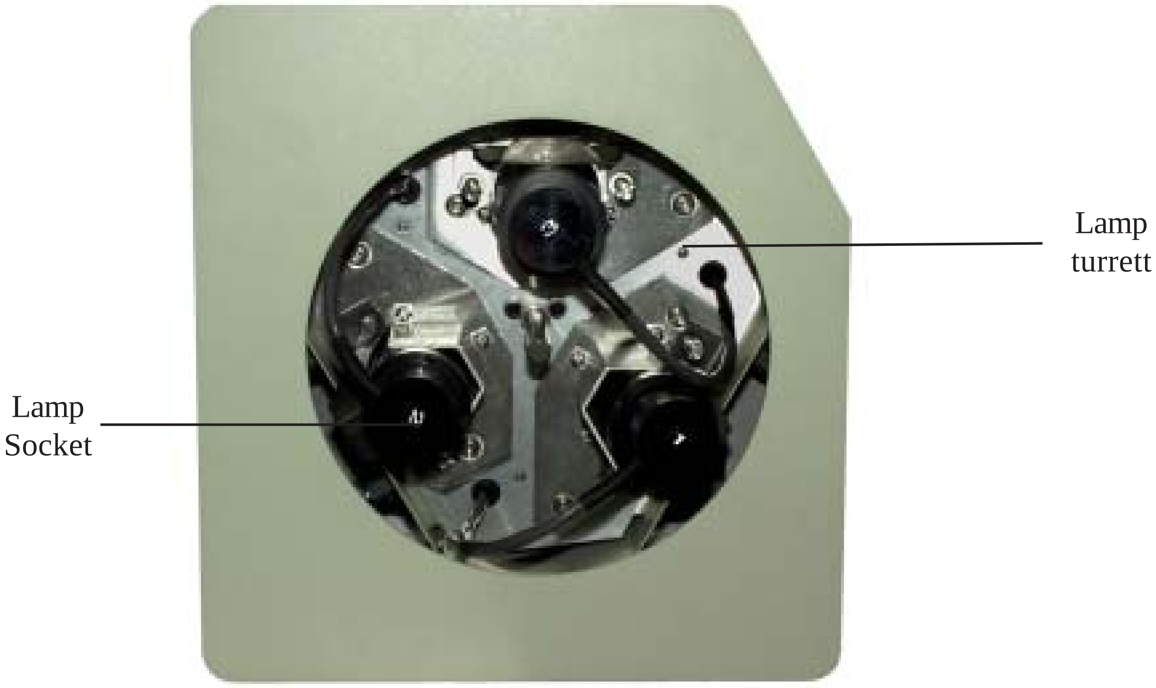
Table 1: Exhaust Ventilation System Specifications

Exhaust Manifold:	Stainless Steel Cone: 8” x 12” opening tapering to 4”collar, 12” overall length
Manifold Installation:	directly above combustion chamberat a height of 18” to 22” above the bench
Primary Exhaust Duct:	4” diameter flexible stainless steel
Secondary Exhaust Duct:	6” diameter minimum dimension
Blower Capacity:	300cfm
Blower Installation:	minimum 10 feet from manifold

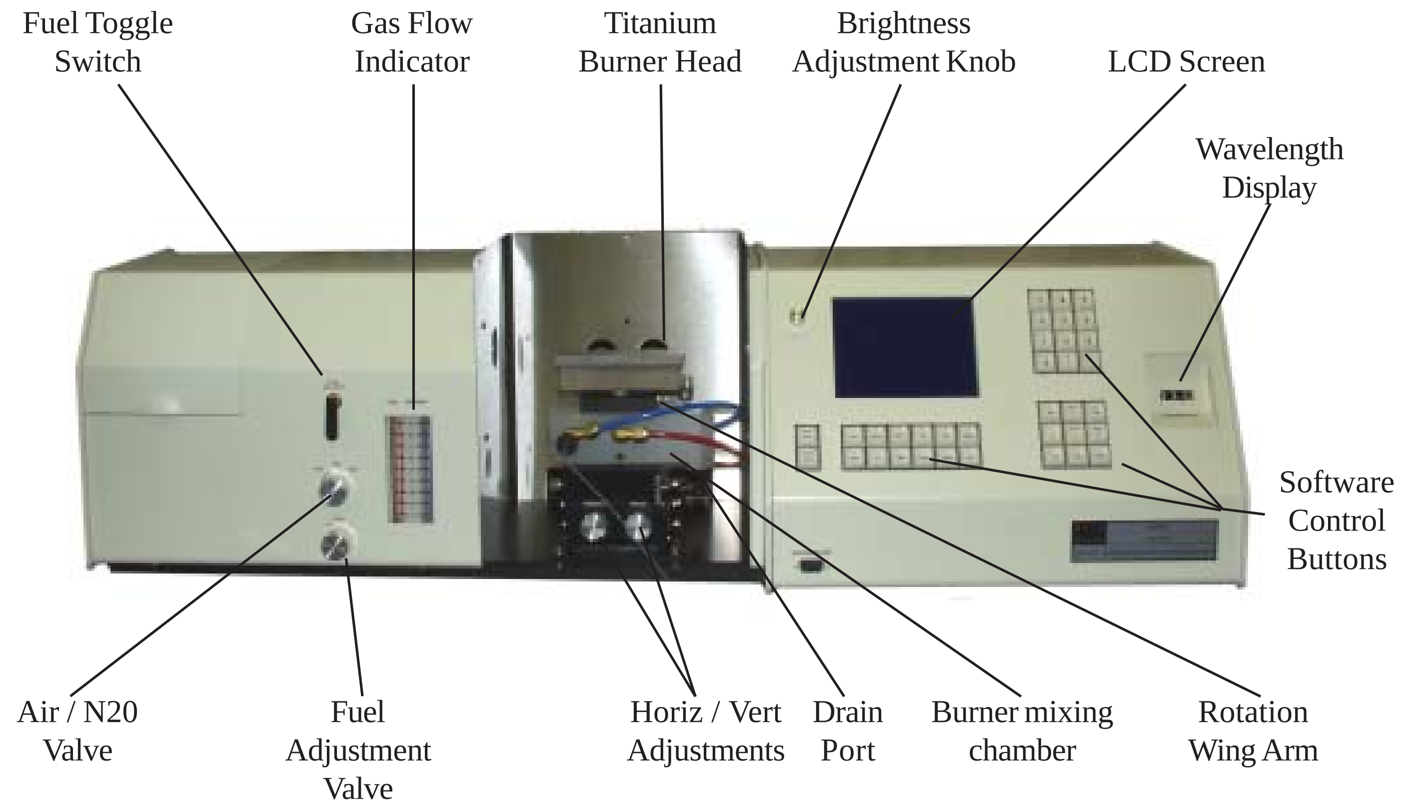
Locate the exhaust intake manifold directly above the combustion chamber of the 210 VGP as shown below.

Right Side

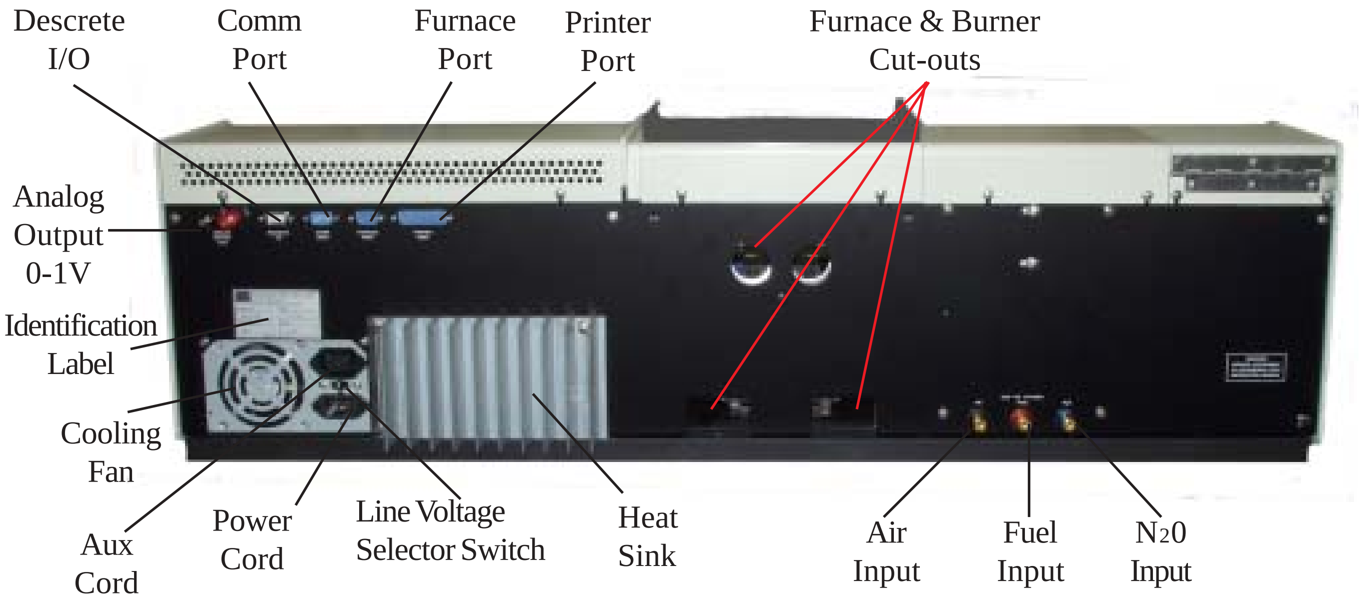
Left Side



Front View



Rear View



SECTION 1: Installation *Continued*

INSTALLATION

Gas Connections

Using the 1/4" brass fittings, connect the BLACK nylon hose from the Air supply to the AIR port (black fitting) on the back of the 210-VGP. Connect the BLUE hose from the Nitrous Oxide tank to the N_2O port (blue fitting). Connect the RED hose from the Acetylene tank to the C_2H_2 port (red fitting). Tighten all fittings 1/2 turn past finger tight to insure a good seal.

Electrical Connections

Plug the power cord into a standard outlet and connect the other end to the power adapter on the back of the 210-VGP unit.

International clients MUST check to see that the small voltage selection switch is set appropriately to 220 Volts.

SECTION 1: Installation *Continued*

Drain Line:

Connect the 3/8" clear Tygon tubing to the black plastic Drain port on the 210 burner assembly. Form a 8" loop in the tubing just below the level of the instrument, and secure it with cable tie or tape. Fill the loop with water using a wash bottle before you have connected it to the drain block, or more conveniently with the drain tube connected, turn on the air only at the instrument & aspirate water through the burner for a while (see *Setting up the Burner*). Place the other end in a large (1 gallon) ***Plastic*** jug, *do not use glass*, making sure the tubing is ABOVE the level of the waste liquid, and secure in place with tape or twist ties.

The water loop acts as a vapor trap to prevent combustible gas mixtures from entering the waste container. If this should happen a potentially hazardous condition would exist. Flash backs can occur from the burner head if the combustion mixture is made too lean. This is especially true when using nitrous oxide. If the loop is empty a flash back can explode into the waste container, causing severe damage to equipment and personnel.

NOTE: When using organic solvents (i.e., MEK, MIBK) for concentration/extraction purposes always flush the drain line with water after analyses are completed. Otherwise a flashback can explode in the loop itself.

SECTION 1: Installation - Graphite Furnace

Installing the furnace accessory:

Remove the drain tube from the spray chamber. Detach the flame assembly by removing the 2 front screws in the base plate with the ball allen driver tools provided. Pull the flame assy forward and out then set aside. Set the furnace assy in the sample compartment and push back making sure to line up the fittings to the bulkhead connectors for water and argon at the rear. Bolt down the furnace power cables to the terminals at the rear of the sample compartment. Screw the baseplate down using the same screws removed from the flame assy.

Proceed to section 4 to install the tube and align the furnace. Then if you have an autosampler read the next section to install it.

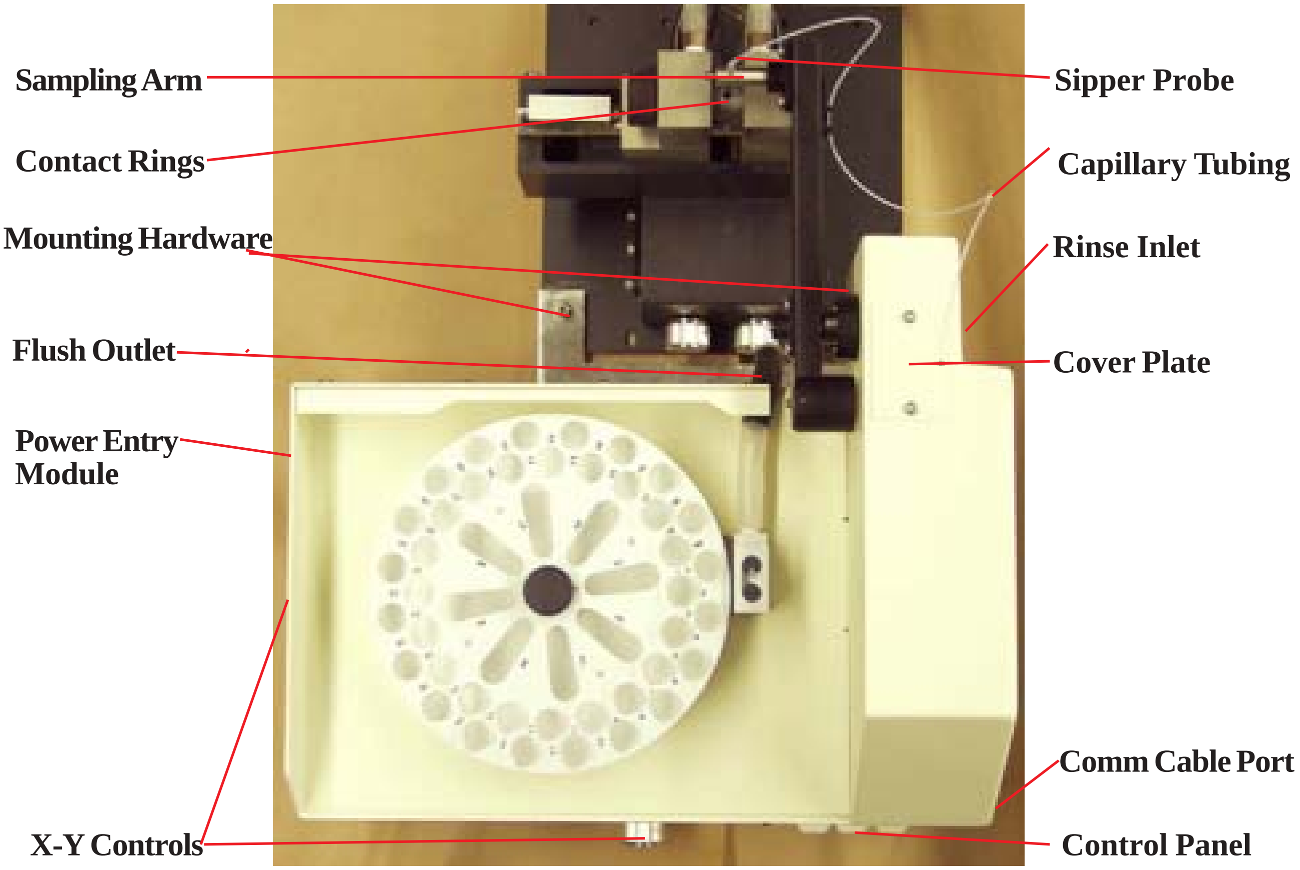
Gas Connections:
Using the 1/4" brass fittings, connect the black nylon hose from the argon regulator on the tank to the argon fitting on the left side of 220GF's box (see diagram to the right). If you are using an alternate gas, connect another black hose from it's regulator to the alternate gas fitting next to the Argon fitting.

Water Connections:
Using the 1/4" brass fittings provided, connect a black hose from your water source to the fitting marked "H2O IN" on the left side of the furnace box (shown above). The water pressure at the inlet should be at least 10psi but no more than 15psi. The flow rate should be at least 1 liter per minute. If your water pressure is too high, you should obtain a needle valve as well as a shut off valve from a plumbing supply house to reduce the pressure or you could burst the tubing on the furnace. Using the 1/4" brass fittings, connect the fitting marked "H_2O OUT" to a drain, unless your state does not allow you to do so. If necessary, you can use a recirculating pump setup as long as it can supply at least 10psi of pressure at the water inlet and a flow rate of at least 1½ - 2 lpm. However, a chiller is recommended for this type of cooling since the water in your water bath needs to remain at a fairly constant temperature or the furnace may overheat and you will lose sample upon injection if the furnace does not cool properly after each burn. If using a chiller, the pressure should be 10 psi and a minimum flow rate of 1½ - 2 lpm. The temperature should be set for 18-20 C to ensure proper cooling without condensation. If you use a chiller, larger tubing (½" OD) should be used and stepped down to ¼" at the instrument to ensure the necessary flow rate.

Electrical Connections:
Connect the 30 Amp 240 volt power plug supplied by your electrician to the power cord for the furnace. The furnace can run on 208 or 240 volt power. This setting may be preset at the factory according to your specifications. If you need to change the setting, have a qualified technician follow the instructions in Section 10.5 for instructions for changing the high voltage transformer taps to best suit your electrical system.

A communications cable is provided for the furnace. You'll need to connect it to the 9-pin female connector labeled "furnace port" on the 210VGP and the only 9-pin female connector on the back of the furnace.

SECTION 1: Installation - Graphite Furnace Autosampler



1) Before attaching the autosampler make sure the furnace has been aligned according to section 4.0 and 4.1.
2) Attach the Adjustable feet to the bottom of the Autosampler using the holes best suited for your benchspace.
3) Attach the autosampler to the instrument with the 2 hex screws and washers provided (do not tighten yet).
4) Remove the small cover plate of the autosampler located on the top right rear to expose the needle of the syringe mechanism. Attach the end of the sipper probe capillary to the syringe needle and reinstall the cover plate.
5) Insert the probe into the sampling arm of the autosampler by loosening the small screw and inserting the probe up to the flange of the probe then tightening the screw finger tight.
6) Connect the larger tubing provided to the flush outlet at the bottom rear of the autosampler and place the other end of the drain line in some type of acid resistant container.
7) Connect the Rinse inlet line provided to the right rear of the autosampler and put the long plastic probe into the rinse reservoir bottle. The bottle can be filled with de-ionized or distilled water.
8) Connect the power cord and communication cable.
9) Make sure the autosampler is relatively level (this can be adjusted with the 2 front feet of the autosampler). With power off to the autosampler move the probe arm to the graphite tube and roughly center it so that the probe tip enters the dosing hole of the contact rings, now finish tightening the screws.
10) Use the X-Y controls (the 2 knobs on the base of the autosampler) to align the autosamplers’ probe with the hole in the graphite tube. Move the arm up and down by hand to be sure the probe enters the tube without hitting the side of its’ hole. Once aligned, you may turn the power on and allow the autosampler to go through it’s power up sequence.
11) The remainder of the alignment procedure is covered in section 4.2.

Configuring the Hardware: Before powering up for the first time perform this procedure.

Press and hold the **[upper case]** button and turn power on by pressing the red button on the right side of the 210. When text appears on the screen the **[upper case]** button can be released. Make any necessary changes to the hardware configuration for:
Graphite Furnace : "YES" if installed
Graphite Furnace Autosampler : "48 Cup" for new systems
Flame Autosampler : "YES" if installed
Printer : "YES" if installed.
Clock : 12 or 24 hour, US or European Date.

. Use the **[left/right]** arrows to make changes in each category and always press **[enter]** to initiate the change. Press **[esc]** when finished. This screen never needs to be accessed again.

SECTION 2: Wavelength, lamp & burner alignment

Notes: Throughout the software most screen have directions to help you, please read them. The help key is functional only for the calibration screen. Use the [left/right] arrows to change the options within a menu item and always press [enter] afterwards or the change will not be recognized. Use the [up/down] arrows to scroll to the next menu item.

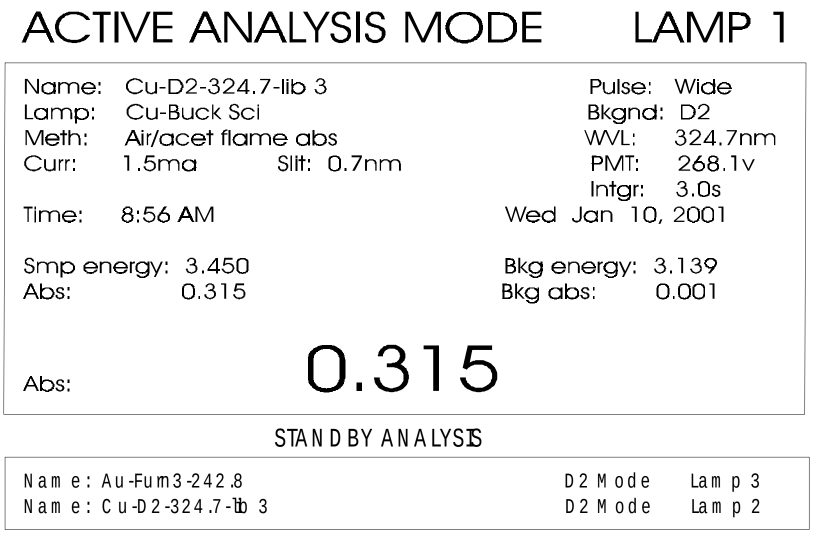
Align the wavelength

1) Install desired lamp in the upper-most position of the lamp turret, this is the operating position. Plug the lamp connector for this lamp position (as designated by the number on the cap) onto the lamp.

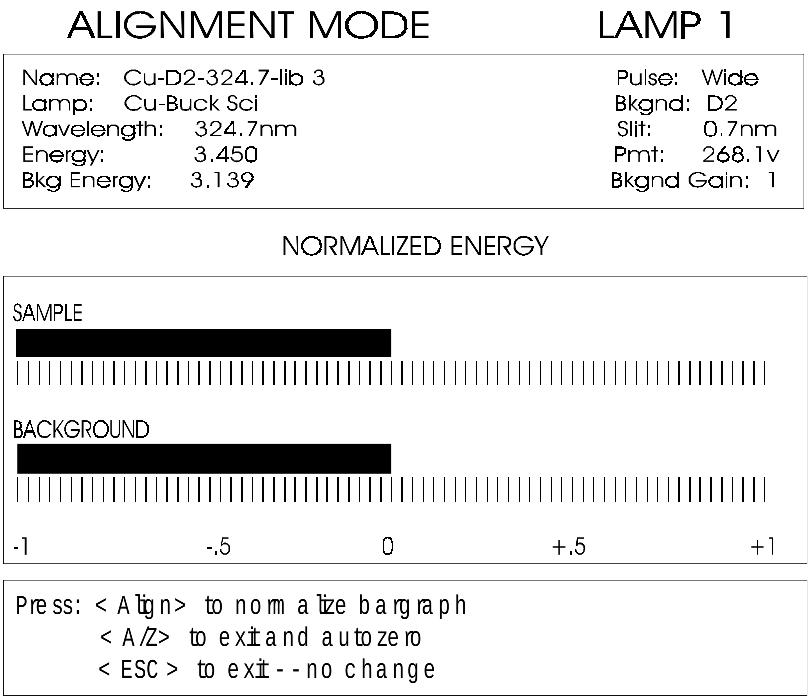
2) Press the **[lib]** button to enter the library. Press **[sel]** button until the lamp number (top of screen) matches the turret position you are using. Press the **[up/down]** arrows until the desired metal and method are shown in the library window. Make sure you have selected an absorbance/flame file (flame files are designated as Xx-D2-wavelength-lib3). Press **[2]** to load the method then **[enter]** then **[esc]**. The file you selected will now appear in the top active analysis window. Alternatively you can press **[1]** for ***enter a library name*** then enter the first letter or two of the atomic symbol of the element you wish to run (you can access the letters on the keyboard by pressing and holding either the **[upper case]** or **[lower case]** button. Press **[enter]** then either the **[up/down]** arrow to select the exact file you wish to run then press **[esc]**. Turn the slit selector knob to the position specified by the library. Turn wavelength knob to the correct wavelength. Press **[align]** to display the bargraphs.

```
                 ACTIVE ANALYSIS
Name: Cu-D2-324.7-Lib 3             Pulse: Wide
Lamp: Cu buck sci                   Bknd: On

                    LIBRARY
Name: Cs-D2-852.1-Lib 3             Pulse: Wide
Lamp: Cs buck sci                   Bkgnd: Off

Press:
<SELECT> Select next lamp
<ESC>       Exit
<up  arrow> or <down arrow> Browse library
     <1> Enter a library name
     <2> Load analysis from library
     <3> Enter a password


  Password required to modify library
```

```
Wide
Lamp: Cu buck sci                         Bkgnd:
D2
Wavelength: 324.7                          Slit:
0.7nm

Set the lamp turret to position 1
Set wavelength and slit as specified
Then:

(1)  Press <ALIGN>
(2)  Fine adjust lamp position and
     Wavelength for maximum energy
(3)  Press <AUTOZERO> when done
     Or
     Press any other function key
```



```
ACTIVE ANALYSIS MODE        LAMP 1

Name:   Cu-D2-324.7-lib 3                    Pulse:   Wide
Lamp:   Cu-Buck Sci                          Bkgnd:  D2
Meth:   Air/acet flame abs                   WVL:     324.7nm
Curr:   1.5ma          Slit:  0.7nm          PMT:     268.1v
                                             Intgr:   3.0s
Time:   8:56 AM                          Wed  Jan  10, 2001

Smp energy:  3.450                       Bkg energy:  3.139
Abs:         0.315                       Bkg abs:     0.001

Abs:                 0.315

                  STANDBY ANALYSIS

Name: Au-Furn3-242.8                     D2 Mode     Lamp 3
Name: Cu-D2-324.7-lib 3                  D2 Mode     Lamp 2
```



```
ALIGNMENT MODE                    LAMP 1

Name:   Cu-D2-324.7-lib 3                    Pulse:   Wide
Lamp:   Cu-Buck Sci                          Bkgnd:  D2
Wavelength:    324.7nm                       Slit:      0.7nm
Energy:        3.450                         Pmt:      268.1v
Bkg Energy:    3.139                         Bkgnd Gain:  1

                  NORMALIZED ENERGY

SAMPLE

BACKGROUND

-1            -.5            0            +.5            +1

Press: <Align> to normalize bargraph
       <A/Z> to exit and autozero
       <ESC> to exit--no change
```

3) The bargraph indicates the amount of energy from the lamp reaching the detector. “0” on the scale represents the amount of energy you had after pressing align. Very slowly turn the wavelength knob in any direction. If the bar graph goes off screen to the right press **[align]** to center it. If the bar graph goes off scale to the left turn the wavelength knob in the other direction. Repeat this procedure until you get the HIGHEST reading (bar graph farthest to the right) you can. Pay attention to the numeric value for ***energy.*** This runs on a scale from 0 to 6 and you want to get it as high as possible. The bargraph will follow the energy reading (ie: as the bargraph moves to the right the energy value will increase. Most lamps will give an energy between 2.5 and 4.5. If you have trouble aligning the lamp, press **[Bkgnd]** to turn the background lamp off and try again. Press **[Align]** when finished. The wavelength is now set.

Align the lamp

4) On the top left of the instrument are 2 knobs for lamp alignment (front knob for horizontal, rear knob for vertical). Rotate either of the two knobs in any direction watching the sample bargraph, again trying to get the most energy you can in a manner similar to the wavelength alignment.

5) When the energy has been maximized, move to the other knob and repeat the procedure. Go back and forth between the two knobs until you can't get any more energy. (NOTE: There will not be nearly the change in energy as there was with the wavelength). The lamp is set when you can't get any more energy. Press **[A/Z]** to zero and exit to the analysis screen.

Align the burner (vertical)

vertical adjustment —> right knob (burner moves up & down)
vertical adjustment —> left knob (burner moves front to back)
rotation adjustment —> middle knob on older instruments or burner bar on newer

6) With the flame and gasses off place a business card or similar surface on top of the burner so that you can see the lamp image on the card. Rotate the vertical adjust knob so that the bottom edge of the light at the focal point is approximately 4mm from the top of the burner (best position for most analysis). Adjust the horizontal if necessary to get the image over the burner head slot, this is only a rough adjustment for the horizontal. (NOTE: Some elements may require different height settings especially when using nitrous oxide, consult the standard conditions section for these instances). Another way to set the vertical position is to lower the burner head until it is clearly not blocking the beam. Perform an autozero by pressing the **[a/z]** key. Slowly raise the burner head while watching the absorbance display. When the burner head intersects the beam the number will suddenly go positive. As soon as the reaction is noticed, stop and lower the burner about 2 turns of the dial.

Align the burner (horizontal)

FLASHBACK HAZARD: ALWAYS turn on the air first, and shut it off last. Make sure the drain tube has a loop approximately 8 inches in diameter for proper drainage and to prevent acetylene from flowing into the waste vessel. **NEVER** use glass or something that can shatter as the waste vessel.

7) With the air and acetylene tanks on and the D2 background lamp off, press **[a/z]** to zero the display, turn on the air knob at the instrument while aspirating water so that you have a mist coming from the burner head. Turn the horizontal adjust knob in any direction while noticing the absorbance display. The object is to locate the burner head directly under the lamp beam to get the highest absorbance reading you can.

8) Repeat the procedure with the rotation adjust (either the burner head bar or rotation adjust knob, depending on which instrument you have). If you have the burner bar you must physically rotate the head to get the highest absorbance reading. Generally, having the burner head so it looks square in the burner compartment is adequate.

Adjust the nebulizer

10) While still aspirating the water turn the nebulizer adjust counterclockwise until air bubbles out through the capillary into the beaker. Slowly turn it clockwise until the nebulizer starts aspirating the water and watch the absorbance readings. Adjust the nebulizer to get the highest and most quiet absorbance that you can (this will probably be within a turn or two from where the nebulizer started to aspirate the standard. The nebulizer is now set and should not need adjustment for many months. (NOTE: The nebulizer comes factory adjusted and should not need any initial adjustment).

11) When you are finished turn the background corrector back on (if your method requires it) by pressing **[bkgnd]**. The background corrector is useful below 450 nanometer to reduce or eliminate false absorbance due to dissolved solids or organic material in the sample.

Remember: If you have not changed anything you only need to check these alignments if you are having problems or need to change the burner head height for special analyses (see section 3.2). When you change lamps you only need to align the wavelength and the lamp.

Igniting the Flame

1) Open the acetylene tank valve and set to 12-13psi.
2) Turn the air compressor on or open air tank valve and set to 50-60psi.
3) Turn on the air valve at the instrument.
4) Turn on the fuel valve at the instrument and adjust flow for 4 on the flowmeter.
5) Light the burner with a long-handled igniter or striker.
6) You are now ready to set up for running your samples.

Shutdown

While the flame is lit, turn off the fuel flow making sure the flame shuts down. Aspirate distilled water for a few minutes with the air still on to cool the burner and flush it out. The flame is now shutdown.

Shutdown for the night

With the flame off, turn off the acetylene at the tank. Turn on the fuel at the instrument, when the flow meter has dropped to 0 the fuel line has been bled. Now turn off the fuel knob and any other gasses at the tanks then turn off the power.

SECTION 2.1: Running Nitrous Oxide

1) Remove the air/acetylene burner head by removing the 3 hex nuts on the top of the burner assembly just below the burn er head. Remove the head and install the 5 cm nitrous oxide head making sure that you re-install the 3 nuts, the o-ring is in place, and the interlock pin is inserted. Do not over-tighten the nuts, finger tight plus a 1/4 turn is good enough.

2) Align the burner head exactly the same way as you would with the air/acetylene head. Keep in mind that your absorbances will be 1/2 of that which you got for the air acetylene head because the burner slot is 1/2 as long. Generally, the burner height will be 1 turn lower than with Air/Acetylene and the horizontal control always needs adjustment after switching burner heads.

3) Make sure that the nitrous oxide is hooked up to the rear of the instrument, that the regulator is set to 50-60 psi and that you are using a ambient air/heated regulator.

4) Ignite the flame as usual running air/acetylene. **Never ignite or shutdown the flame in nitrous mode**. After the flame is lit, adjust the fuel flow so that the flame is a rich yellow and is smoky at the top. This might require a number of turns of the fuel knob past the point where the flow meter ball hits the top of the scale.

5) In one motion turn the oxidant selector switch to the N20 position, **do not stop in the middle**. At this point the flame should have a red cone at the top of the burner head about
1-2 inches high. Adjust the fuel so that the red cone is about 1/2 an inch high.

6) Proceed with your analysis.

7) To shutdown Turn the oxidant selector switch, **again all in one motion back to the air position**, adjust the fuel flow back to the normal position of approximately 4.

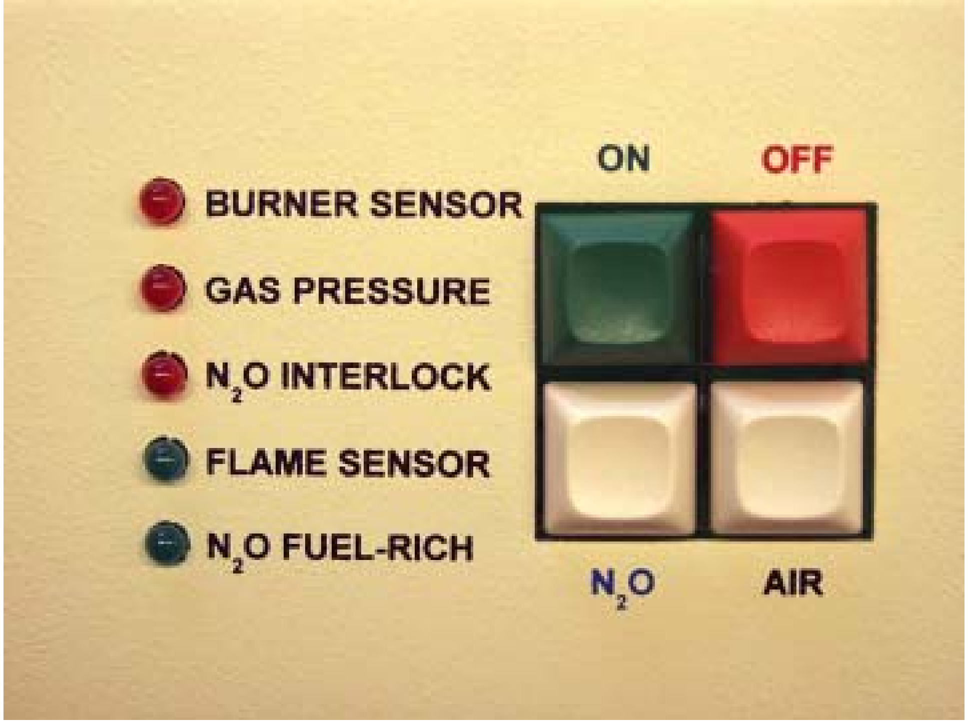
Important note: Buffers for Nitrous Oxide

Spectrographic buffering is essential to minimize the effects of ionization in either a nitrous oxide flame or a rich air-acetylene flame (for doing chromium, tin or barium by air). Analysis without buffering may produce erroneously high readings.

For Calcium, Strontium and Barium: Use Lithium buffer & Lanthanum release agent
Lanthanum is generally used for the alkaline earths to minimize the chemical interferences from phosphate and occassionally sulfate. Dilute all samples, blanks and standards so that they will contain 1000ppm (0.1%) of both the buffering and releasing elements by adding 10ml of each solution to 100ml of prepared final solution.

For all General nitrous oxide work: Use Lithium & Potassium buffers
Dilute all samples, blanks and standards so that they will contain 1000ppm (0.1%) of the buffering element by adding 10ml of buffer to each 100ml of prepared final solution. Lithium may give better results than Potassium in certain circumstances, depending on the sample matrix and flame conditions.

SECTION 2.2: Operation of the 211 Automated Gas box



Safety Features:

The automated gas box has been designed for pushbutton flame operation. The gas box also has safety features built in to prevent improper operation.

1) A flame sensor, which shuts down the gas if no flame is detected.
2) A drain sensor, which prevents ignition unless the trap is filled with water.
3) Blow out plug sensor, which prevents ignition unless the plug is installed.
4) Burner head sensor, which insures the proper burner is installed for the gasses used.
5) Power failure detection, which shuts the flame down safely should a power failure occur.
6) Keyboard detection, which shuts down the flame should an improper key selection be made.

Igniting the Air / Acetylene Flame:

The auto gas box has a special drain sensor that is in line with the drain tube. Fill the sensor with water by unscrewing the top until water is seen coming out the bottom hose. If this is not filled with water the flame will not light. Make sure the drain sensor is hanging vertically. Connect the drain line to the spray chamber and make the electrical connection. The other end of the drain line should go to a plastic acid resistant waste container.

1) Set tank air pressure to 60psi and acetylene tank pressure to 13psi.
2) Press and hold the **[air]** button on the left panel and check to see you have at least a flow of 5 for both the air and acetylene gases. Adjust the acetylene flow up if necessary or the flame will not ignite then release the **[air]** button.
3) The interlock lights should all be off except for the **[N20 interlock]** light. If the burner light stays on then either water needs to be added to the drain sensor or the burner head or blowout plug are not properly in place.

4) Press and hold the **[on]** button for 5-8 seconds until the flame ignites then release the button. If the flame does not ignite, recheck the gas flows and try again.
5) After ignition adjust fuel down so that it is 1-2 units less then the air unless your analysis requires a higher flow. At this point the **[on], [air]** and **[N20]** keys are disabled.
6) To shut flame down press the **[off]** button and gases will shut down and extinguish the flame

Igniting the Nitrous Oxide Flame:

1) Remove the 3 nuts holding the air burner head in place then remove the burner head
2) Make sure the o-ring is in place then install the nitrous burner, tighten the 3 nuts and connect the interlock pin. Be careful not to over-tighten the nuts.
3) Press the **[air]** button and check for proper ignition flows.
4) Press and hold the **[ON]** button for 5-8 seconds until the flame lights.
5) Press the **[N20]** button and release. The flame will 1st switch to a bright yellow flame then to a bluish, tall nitrous oxide flame. The flame should have a red feather on top of the burner about ½" high, if not, adjust the fuel flow for this condition. Do not lower the fuel so much that the red disappears and try to avoid raising so much that the flame becomes white. The more fuel that is introduced the quicker carbon will build up on the burner slot and cause readings to drift. Some elements may require a higher fuel flow for optimum sensitivity. The carbon can be scraped off from time to time with a long handled screwdriver while the Air/Acetylene flame is lit. See section 3.2 Optimizing the Flame for information on adjusting the flame conditions for maximum absorbance.
6) To shut off the nitrous flame **(normal)**, press the **[air]** button to switch back to an air based flame, then after 10 seconds press the **[off]** button.
7) To shut off the nitrous flame **(emergency)**, just press **[off]** and the system will immediately purge all the flammable gases from the system with a puff of compressed air to extinguish the nitrous flame with a gentle "popping" sound. This is not a flashback but a safe "forced shutdown" of the flame.

ALL OTHER INSTRUMENT OPERATION & SET UP IS THE SAME AS THE MODEL 210. PLEASE REVIEW THOSE SECTIONS OF THE MANUAL.

SECTION 3: Setting Flame Analysis Parameters—[Cntls] key

1) If you have not already selected a library method do it now by pressing **[lib]** and selecting the proper lamp number by using the **[sel]** button. When your lamp is selected use the **[up/down]** arrows to select the desired method, press **[2]** to load the library and then press **[enter]** and **[esc]**. Align the lamp and wavelength then **[esc]** to the analysis screen.

2) Pressing the **[cntls]** button displays the following menu:

Note: Use the **[lef/right]** arrows to change the selection then press **[enter]**.

Display mode: The options are **[absorb/emiss] or [concentration].** This will automatically display concentration after running a calibration curve.
Read-key Sampling: The options are **[on] or [off]**. Set to on allows for single sample analysis with or without the autosampler. In this manner you can print out sample identifiers from the **[smpls]** page which will be explained later. When the **[read]** key is pressed the instrument will ask you to enter a starting sample number and this identifier will be printed on the screen and also sent to the printer if the print functions have been turned on. Pressing the **[up]** arrow then the **[read]** key from here on out will advance the sample position by one each time. Pressing **[esc]** will abort the analysis.
Read-Key Sampling Auto-Increment: Automatically increments sample ID after each sample is read when using Read-Key Sampling
D2 Staywarm: The options are [yes] or [no]. This keeps the D2 lamp warmed up while not in use. This should be left on.
HCL Staywarm: The options are **[yes] or [no]**. Keeps the unused hollow cathode lamps warmed up when not in use. This should be left on.
Autosampler: The options are **[off] or [on]**. Set to on if you have an autosampler and will be using it.
Recorder Response: The options are **[0] or [tim1-tim8]**. For using a strip chart recorder which is connected to the rear of the instrument. Higher timing speeds provide for more dampening of the signal to the recorder.
Measurement Controls: Displays file information including Lamp Currents and Integration Time.
Remote Communication Controls: The options are **[off], [library/report] or [remote program]**. Set to off unless you will be using a computer for data collection in which case it will be set to library/report.
Set Clock: To change clock settings.
Report and Display Controls: Displays the following sub menu:

Decimal Digits (raw data): Selects how many decimal digits will be displayed for absorbance. This should be set to 3.
Conc Display Precision: Selects how many integer and decimal digits will be displayed for the high standard when in concentration mode.
Display Re-Write Interval (in seconds): Selects how often the main display number will update itself. A higher time value is recommended.
Read-Key Reporting: The options are **[on] or [off].** Produces a printout of results when the read, Autozero or Reslope key is pressed.
Read-Key Report Header: The options are **[on] or [off].** Prints the file information at the top of the report.

Calibration Reporting: The options are **[on] or [off].** Prints calibration information during the calibration procedure as well as the calibration summary of results at the end.
Printer: The options are **[on] or [off].** Activates the printer.
Printer Page Eject: The options are **[on] or [off].** Ejects the last page when the analysis is completed. This keeps the next report header at the top of the following page.

SECTION 3.1: Setting Flame Analysis Parameters—[Smpls] Key

Note: The [smpls] form does not have to be filled out to calibrate or run an analysis, it is only useful if you have an autosampler or you need samples identified on the printout.

Edit Sample Identifiers: Allows you to print sample identifiers and/or set up the protocol for running the autosampler. Fill in the table by entering your sample ID's starting from position 1. When the **[start]** button is pressed when starting your analysis the autosampler gives you the option to start at any position. By default the software comes with position 151 labeled as Std1-blank, position 152-156 labeled as Std2 – STD6 and positions 157-161 as QC1 – QC5. These can be changed to any position you wish. You **must** identify a blank and standards in order to run a calibration with or without an autosampler. Position 151 for blank and 152-154 for standards should automatically appear in the calibration form without doing anything to this table. After entering the identifiers press **[esc]** twice to exit.

Note 1: If you press the **[up arrow/down arrow]** while holding the **[lower case]** button in the form will advance a page at a time. If you do the same with the **[upper case]** button the form will advance to the very beginning or end.
Note 2: Make sure you put a space in the first character or leave an entire blank line between your sample ID's and the start of the blank & standards. This tells the program to stop the analysis (and the autosampler if applicable), otherwise the program will run the blank and standards again after your samples are finished.
Note 3: A dash (-) in the first character space of a line tells the software to skip that sample.
Note 4: Pressing **[esc]** will abort a run.
Note 5: Your standards can be entered low to high or high to low, just make sure the blank goes first.

Rename all Samples to Default: Will restore the default sample ID page if changes have been made. This is also accomplished by pressing **[Align]** from the Cal Form.
Blank Std Cup: The Blank standard cup number and description as defined on the Cal Form which changes automatically when the curve is calculated.
Max Std Cup: The Max standard cup number and description as defined on the Cal Form which changes automatically when the curve is calculated.
Sample Order: Allows you to run up to 4 qc checks or check standards during the course of an analysis. You can also perform automatic autozero's and reslope's. In the ***loc*** column put in the tray location of the standard, qc check, reslope standard or blank you wish to run. The columns ***before***, ***interspersed***, and ***after*** indicate that the functions you select will be carried out before, interspersed during, or after your regular samples are run. The functions available are ***none, rslp, autozero & read***. This means that with any of the standards/samples that you enter into the ***loc*** column you can choose to do nothing with it, reslope on it, autozero on it, or simply read it as a sample/standard/qc check. In this screen you can also specify how often to run this group of standard/samples when interspersed is selected.

Sample Replicates: Designates how many times each sample is to be read.
Stabilization time: Designates the length of the read delay before the sample is analyzed.
Rinse time: Designates how long the autosampler will rinse between samples.

SECTION 3.2: Sensitivity Check / Optimizing the Flame

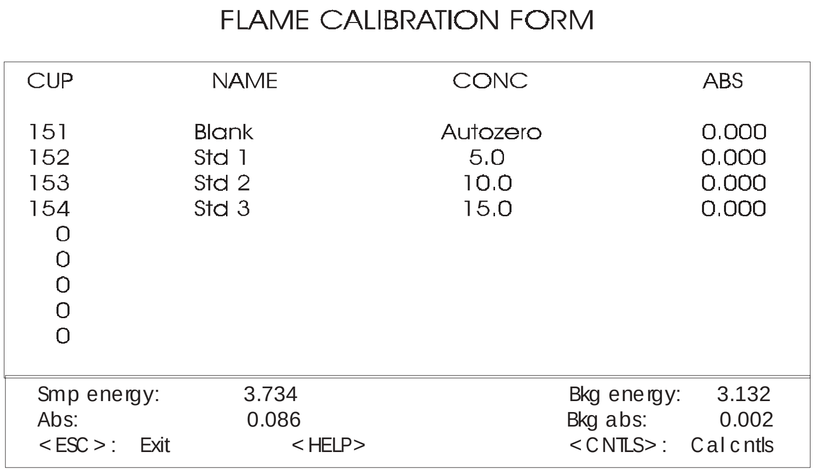
Sensitivity check

You may wish to perform a sensitivity check before calibrating to verify that the burner system is working well and adjusted properly. Light the flame and aspirate your high standard. Make note of your absorbance reading. Look up in **Table 1 : Flame Atomic Absorption Concentration Ranges** in the back of this manual for the average absorbance obtained for “CAL MAX” standard under the “Sensitivity Check” column. If you are unable to obtain absorbance readings similar to those on the table, you may need to go through the burner alignment procedure again, clean the nebulizer and burner head (see Section 10 & 11 Troubleshooting and Maintenance) and/or optimize the flame.

Optimizing the Flame

Most elements run well with a lean blue flame. As a result, setting the fuel at 4 on the flow-meter and adjusting the burner height with a business card as described in Section 2 is sufficient for most elements. However, for elements requiring richer flames (including those requiring nitrous oxide), or if you are having trouble achieving the sensitivity check, optimizing the flame may improve your results. Starting with the burner head 4mm below the beam, light the flame and let the burner warm up a few minutes while aspirating de-ionized water. Zero the instrument then aspirate your high standard. Slowly increase the fuel (turn the fuel adjust counter clockwise) while watching the absorbance reading until you reach the best absorbance. If increasing the fuel does not improve it, try decreasing instead (If you are running nitrous oxide, be careful not to decrease below the ½” cone). Since increasing fuel will change the height of the flame, you should then adjust the vertical burner adjustment in the same manner.

SECTION 3.3: Calibrating Standards and Analyzing Samples — [Cal] Key



```
                FLAME CALIBRATION FORM

CUP        NAME          CONC          ABS

151        Blank         Autozero      0.000
152        Std 1          5.0          0.000
153        Std 2         10.0          0.000
154        Std 3         15.0          0.000
  0
  0
  0
  0
  0

Smp energy:     3.734            Bkg energy:   3.132
Abs:            0.086            Bkg abs:      0.002
<ESC>:  Exit       <HELP>        <CNTLS>:   Cal cntls
```

Note: You should check the [help] menu for a list of special key functions available to you.

1) Press the **[cal]** button. The screen will display the calibration form with cup 151 as std1- blank and 152-154 as std's 2 to 4.

Note 1: The first standard (at the top of the table) must always be the blank.
Note 2: The default settings define Std 2 as the "max" standard, which is necessary to define for emission analysis but may be ignored for other calibrations.
Note 3: If the Calibration Form had previously been edited, other data may be present. Pressing **[align]** here restores the above default sample data.
Note 4: You may wish to set up your own cup numbers and identifiers for use on the Cal Form by editing the sample identifiers under the **[smpls]** menu (see section 3.1)
Note 5: A cup number, name and concentration value must be present otherwise an error will result.

Press the **[down arrow]** to highlight 152 then press **[sel]** button to move the cursor to the conc column and enter in the concentration of the standards you will be analyzing. Type in the value of the standard and press **[enter]** or **[down arrow]** to enter the value. If you have less than 3 standards to run you can highlight the cup you want to remove then press and hold the [upper case] button then press [del] and [enter] to remove the line. If you want to add a 5th or 6th standard type in the cup number of 155 or 156 under 154 and press [enter]. The sample identifier will be brought over from the [smpls] page automatically. To enter more standards you can add appropriate identifiers on the [smpls] page.

2) Press the **[cntls]** button.
a.) Select the ***units*** to report results in using the arrow keys and press enter when complete.
b.) Select the curve type under ***degree. Linear*** requires a minimum of 1 standard, ***2nd*** requires 2 standards, etc. For best results, use at least 1 more standard than required. You should be able to obtain a good calibration curve with 2 or more standards using 2nd order fit. Use the option for 3rd or 4th order curves for more standards only if necessary.
c.) Select how many ***standard replicates*** to run for each standard.
The rest of this form contains data from the last calibration for reference only. Press **[esc]** to return to calibration form.

3) Before starting make sure the instrument and flame have been on for at least 5 minutes. The background corrector should always be on for any analysis below 450 nanometer. Press the **[start]** button and follow the directions at the bottom of the screen. Aspirate the blank and press the **[enter]** key. Always allow 5 seconds between aspirating a blank/standard/sample, and reading a sample to let the sample equilibrate in the flame. An absorbance reading of 0.00 will fill in. Aspirate the 1st standard and press **[enter]**. If you selected to run more than 1 replicate the software will prompt you read the standard that number of times then it will report the average under the ***abs*** column. Continue for all standards.

4) When finished press the **[cal]** button to calculate the curve. If you have made any errors either by selecting the wrong degree or the standards were improperly prepared resulting in a curve that could not be fit, it will be reported here. If there are no errors press the [yes] button to calculate the curve then press the [enter] button to look at the curve. Examine the graph. If the calibration curve appears to be "falling off " the data points, press **[esc]**, then **[cntrls]** and change the ***Polynomial Degree*** to a lesser value. Then press **[esc]** then **[cal]** and recalculate the curve and look at the plot. If your absorbances are higher than about 0.600, you will most likely require at least a 2nd order curve with 3 standards for a good fit as most elements are only linear to this value. If for some reason an improper read was made while calibrating you don't have to perform the whole calibration over. You can highlight the cup position and press either the [a/z] or [read] button to update the absorbance reading then recalculate.

5) After you've viewed a satisfactory curve, press **[esc]** twice to return to ***active analysis*** where you can now read your samples. The instrument display will read continuously as you aspirate your samples. For best results, press the **[read]** button to have the instrument perform an integration of the signal for the time specified in the library. This will provide a more accurate and reproducible reading as the software is taking an integration over several seconds then reporting an average of the result.

Note 1: It is always a good idea to check a blank and a standard before starting your run and every 5 or 10 sample to insure accuracy. It is possible for any atomic absorption instrument to drift over a period of time due to changing conditions. You can reset your curve if drift occurs by aspirating the blank and pressing **[a/z]** then aspirating your high standard and pressing **[rslp]** at any time then continuing on with your samples.

Note 2: Your high standard should always be slightly higher than your expected results. If you try to run a sample that is significantly higher than your highest standard you will receive a ***concentration over-range*** error message.

Note 3: When a file has been loaded into a turret position it will stay there until a new file is selected in the ***lib*** screen. When a calibration curve is applied, it to will stay there until a new curve is run. Therefore, if you use the same lamp, turret position and standards and have not loaded a new file to the turret position it is possible to recalibrate the instrument from day to day without even going to the calibration form. Simply let the the instrument and flame warm-up for 5 minutes then from the ***active analysis*** screen aspirate the blank and press **[a/z]** then the high standard and press **[rslp]**. As long as the absorbance values are close to what they were previously the instrument will recalibrate by adjusting the previous curve that was saved and you can run your samples.

Automated Analysis

If you will be using an autosampler and you wish to run fully automated press the **[start]** key and your samples will run according to the protocol you set in the ***Smpls*** page starting at the position you specify. You can set up the ***Sample Order*** table under the **[smpls]** key for the autosampler to read, autozero, or reslope any of your standards before, during or after the run (see section 3.1) You can run manually with an autosampler if you turn ***read key sampling*** on. In this mode, after pressing **[read], [A/Z], or [Rslp]** the screen will ask you to enter a cup location. Enter a number then press **[enter]** twice. After this sample is read you can press the **[up arrow]** to select the next sample.

Manual Analysis

1.) If you do not have an autosampler you can still use the **[start]** key. The screen will prompt you for a sample position and that identifier will appear according to what you entered in the s***mpls*** page and the ***sample order*** table as in the Automated Analysis. This allows you to set up a protocol for checking QC standards during the run so the operator is prompted to check them at certain intervals. After setting up the options under the **[smpls]** key as described in section 3.1, press the **[start]** key, enter the starting sample #, press **[enter]** then simply aspirate your sample and press the **[enter]** key again. If the ***Auto-increment*** option on the **[cntls]** menu is ON, the sample number will automatically increment for the next reading. Pressing the **[up arrow]** after the read is complete will advance the cup position to the next position in the ***smpls*** page.

2.) If you do not need to set up a protocol for sampling, you can start your run using Read-Key Sampling which gives you the option to press **[read], [A/Z] or [rslp]** at any time during the run while still using sample identifiers.

3.) If you do not need to set up sample ID's or you only have a couple of samples to run you can skip the s***mpls*** page (make sure that read key sampling is off if you do this). Just run your standard curve as usual then go to the active analysis screen, aspirate your samples and press **[read]**. If you do not want to press **[read]** for each sample you can press **[enter]** when you are in the ***active analysis*** screen and the instrument will read continuously. Be aware that if you read continuously the result will not be integrated, it will only be a "snapshot" of the concentration at that instant. You must use the **[read]** key if you want the result integrated at the time you specified.

SECTION 3.5: Emission mode

1) Set up as you would for absorbance except choose an emission file from the library and do not change the mode to concentration.

2) Remove the lamp from the top turret position or move the turret to a position that does not have a lamp.

3) Turn the wavelength to the approximate correct position. Press **[align]**, turn on the flame and aspirate your high standard. If a low energy error occurs you will need to press **[align]** again. Optimize the wavelength for maximum energy using the wavelength knob. Press **[Rslp]** when you are finished to set emission range to 100 and enter the active analysis screen.

4) Aspirate your blank and press **[a/z]**. Aspirate your high standard and press **[rslp].** Aspirate the blank again and press **[a/z].** This will set the pmt gain to the proper level. The emission counts will show about 100.0 for your high Standard.

5) If you want sample identifiers on the printout press **[smpls]** and fill in the table as usual, if not you can skip this step.

1) From the ***active analysis*** screen press the **[cal]** key. Set up the calibration information just as you would for absorption then press the **[start]** key and follow the instructions to calibrate. The software will prompt you to run your high standard, then perform a reslope automatically. Then the calibration will proceed as in Absorbance mode. Calculate as usual then return to active analysis and run your samples.

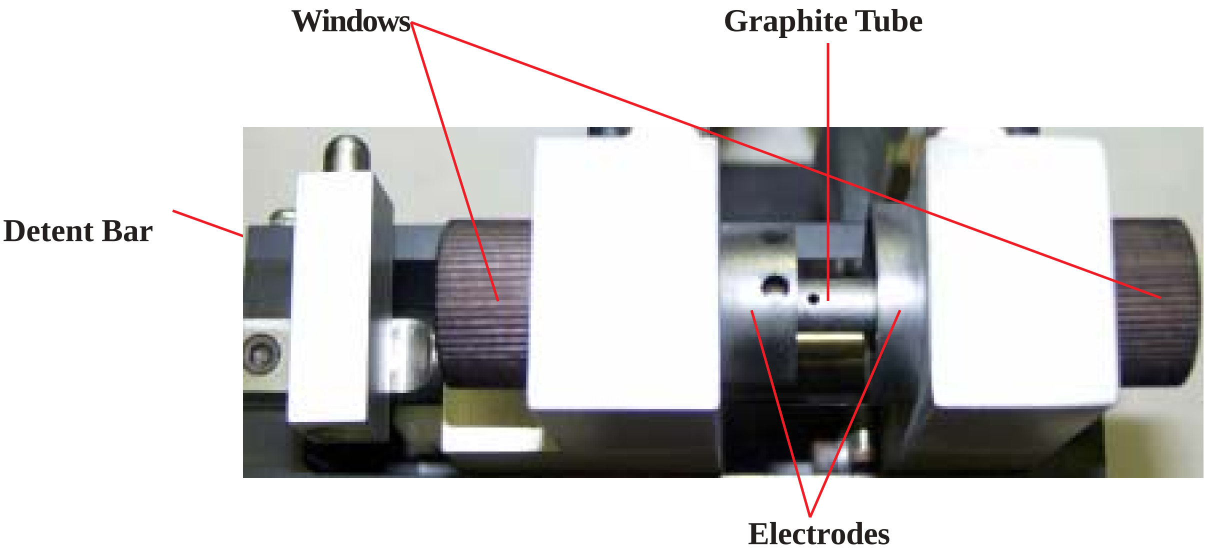
SECTION 4: Basic Operating Procedures - Graphite Furnace Technique

THE M-220 GRAPHITE FURNACE SYSTEM & AUTOSAMPLER

The M220 graphite furnace utilizes a rapid, frequency-induced heating technique, coupled with accessible multi-step microprocessor control. The M220 graphite furnace uses 50 µL capacity graphite cuvettes (p/n BS007-0699), pyrolitically coated furnace cuvettes (p/n BS009-1504), or L'Vov platform cuvettes (p/n BS-220-2697).

Each element in the 210VGP library has a preconfigured furnace method, which is designed for use when analyzing dilute aqueous samples. The use of matrix modifiers is strongly recommended in all graphite furnace work. Consult the standard conditions listing Section 6 for the elements of interest when selecting the appropriate matrix modifiers. The furnace methods should be adjusted when analyzing other complex samples, for instance biological fluids, where the matrix contains very high levels of dissolved or suspended materials (see Section 2: *Editing a Method*).

SECTION 4.0: Installing the Graphite Tube



1) Turn the furnace ***detent bar*** (located below and to the left of the left hand quartz window) either forward or back to release the pressure on the furnace assy.

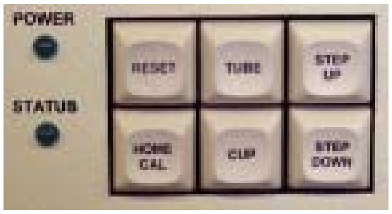
2) By hand spread the furnace cooling towers apart and insert the graphite tube. It is easiest to start the tube into the right hand contact ring first then level it off and squeeze the cooling towers together. Line the hole in the graphite tube up with the top hole in the contact ring as best you can then insert a pipette tip through the contact ring hole and into the hole of the graphite tube to center the tube hole with the contact ring hole. When this is set push the ***detent bar*** back into position. You should check the tube position with a small flashlight when finished to make sure it is in the proper position.

SECTION 4.1: Aligning the Furnace

1) Load the desired library into a lamp position as described in section 2. Furnace files use the designation ***furn3*** in the file, make sure these are the files that are loaded.
2) Hold a business card in front of the left hand furnace window to see the lamp image. If it is not centered through the window use the ***horizontal and vertical adjustments*** at the front of the furnace assembly to center it.
3) To fine tune the adjustment , press align and watch the bargraph as you adjust the same knobs to get the most energy through the furnace.

SECTION 4.2: Aligning the Autosampler

1) You'll first need to familiarize yourself with each key switch function on the 220AS Autosampler's front panel. The following is a list of all key switch names, descriptions and locations.



A. **RESET**:

(1) If pressed and released, acts the same as the power-up sequence. Each motor is initialized using an optical-sensor and the positions stored in memory. A rinse cycle is performed and the arm is placed in the SAFE position.
(2) If held for 4 seconds, resets position relative to motor phase (all motors). Otherwise same as (1). This is used if both lights blink during operation which means a
"Motor Error" has occurred.

B. **HOME/CAL**:

(1) Toggles between HOME and CALIBRATED arm position modes. Initially in CAL mode, arm positions are as previously calibrated (retained during power-off). Toggling to HOME (as indicated by "Status" led) provides higher arm positions for TUBE and CUP commands (for when the probe has been disturbed). Toggling between HOME and CAL does not affect the calibration.
(2) If held during power-up: Suppresses power-up reset
to allow testing of motors individually.

C. **TUBE**:

Moves arm to the furnace HOME or CAL position.

D. **CUP**:

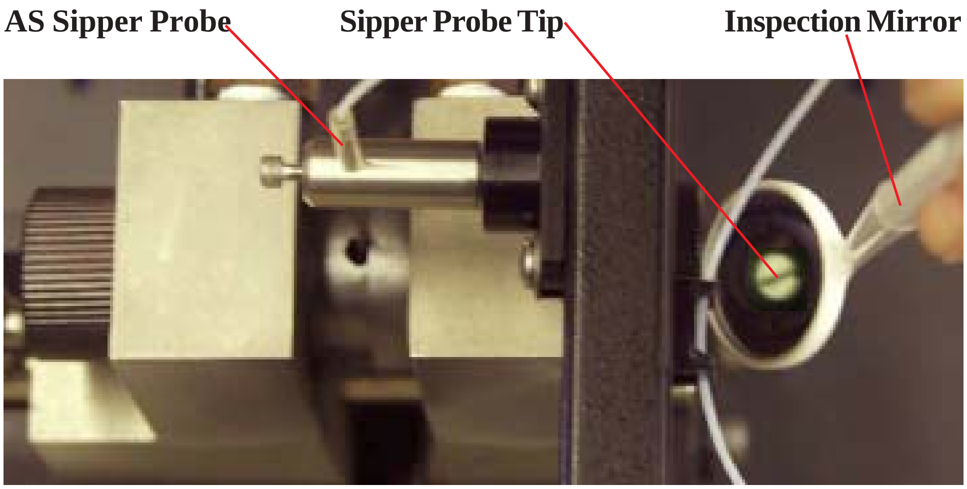
(1) Moves arm to cup HOME or CAL position.
(2) Holding this button down will first move the arm to a cup position, then lift the arm to the SAFE position, move and/or rotate the tray to position 1 and move the arm into the cup.
The CUP and TUBE commands do not affect the calibration.

E. **STEP UP**:

If arm has been put in either TUBE or CUP position, moves arm one step up (or repetitively if held). The new arm position is stored as the CALIBRATED position.

F. **STEP DOWN**:

If arm has been put in either TUBE or CUP position, Moves arm one step down (or repetitively if held). The new arm position is stored as the CALIBRATED position.



1) Press the **[HOME/CAL]** button. The POWER light will go off and the STATUS will come on to show that you're operating in HOME mode.
2) Put one of the sample cups into position 1. Press and hold the CUP button for 4 seconds. The arm will immediately move to the cup's home position, then, after a few seconds, the autosampler arm will move to the SAFE position, the tray will rotate and/or move to line up position 1, then lower the arm back to the cup's HOME position.
3) Using the **[STEP DOWN]** button, lower the arm 'step by step' into the cup until the Sipper Probe Tip is approximately 1/16 of an inch or more off the bottom of the cup. If you go too far, use the **[STEP UP]** button to move the arm back up a step. This CALIBRATED position is automatically stored into the autosampler memory. The STATUS light will blink until either the other arm position is calibrated or another motion command is given.
4) Press the **[TUBE]** button to move the arm to the 'Home' position on the Graphite Furnace side. Using the **[STEP DOWN]** button, lower the Sipper Probe 'step by step' into the Graphite Tube until it's tip is approximately 1mm of the bottom of the tube. With the D2 background lamp off, use the included inspection mirror to look in the right-hand side Furnace Window to check the probe's depth.
5) During operation, the 'POWER' and 'STATUS' lights will indicate the state of the autosampler. The following table may be used as a guide for autosampler states:

POWER	STATUS	STATE
off	off	power off
on	off	ready
blinking(fast)	—	Not communicating
blinking(slow)	off	Performing a command
off	on	Home Mode
off	blinking	Arm partly calibrated
blinking	blinking	Motor Error (press and hold "RESET" to clear)

SECTION 4.3: Setting Furnace Analysis Parameters—[cntls] key

These methods are designed for general purpose analyses of dilute aqueous samples. The use of matrix modifiers is strongly recommended for most graphite furnace work (see section 13). The furnace methods will have to be adjusted when analyzing other types of samples, for instance biological fluids, where the matrix contains very high levels of dissolved or suspended materials.
Many of the parameters for furnace work will be very similar to those of flame so they will not be repeated here, only the differences will be explained.

In the **[cntls]** page the item ***furnace/vapor controls*** has been added as described below:

Peak Timing: The options are ***auto, armed and fixed***.

Auto: The software will automatically determine the integration window for the peak during calibration.

Armed: The software will set the window for the next reading. For best results, this setting should never be used for calibration.

Fixed: The operator sets the peak delay and duration manually by observing the largest peak. Fixed settings are preferred for calibration when maximum sensitivity is needed.

Set Peak Delay (in seconds): The time from the Y-axis at which the integration begins.

Set peak Duration (in seconds): The length of time the integration will last.

Note: If you want to set your own values for ***delay*** and ***duration*** peak timing must be set to ***fixed***

Replot Last Peak: Plots the peak of the last analysis. A White bar at the top of the plot shows the area used in integration.

Furnace Program: Displays the entire furnace program. No changes should need to be made for ordinary aqueous analysis. However if you are working with organic or complex sample matrices you may need to adjust ash time and/or temperatures. You may need to adjust the Dry and cool time and/or temperatures to accommodate your cooling apparatus. To edit a furnace program, highlight the step to change and fill in the appropriate information.

Furnace Clean-up Cycle: Runs a furnace program with no sample introduction to clean out the graphite tube.

FURNACE PROGRAM

#	Phase Name	Temp	time
0	Initialize	80	10.0
1	Dry-ramp	250	10.0
2	Dry	250	5.0
3	Ash-ramp	850	10.0
4	Ash	850	5.0
5	Int-flow-off	850	0.0
6	Autozero	850	3.0
7	Data-start	850	1.0
8	Atomize-ramp	1950	1.5
9	Atomize	1950	3.5
10	Int-flow-on	1950	0.0
11	Burn	1950	1.5
12	Data-stop	1950	0.0
13	cool	80	5.0

<ENTER> to edit <ESC> to exit

EDIT FURNACE PHASE

```
Current furnace phase:

9       atomize         2400          5.0

Select phase type:     atomize
Set (END) temperature      2400
Set time                              5.0
Insert/add phase
Delete phase

<ESC> to exit        <ENTER> to save
```

To ***Delete*** the selected phase, highlight ***Delete Phase***, and press **[enter]**.

To ***Insert*** a phase *Before* the selected phase; highlight ***Insert/Add Phase***, and press **[enter]**. To ***Modify*** the existing phase, use the **[up/down arrow]** keys to scroll to ***Select Phase Type***. Use the **right arrow** key to select the type of phase desired (listed below), and press **[enter]**. To modify a ***Time*** or ***Temperature*** setting, highlight the corresponding line and enter the new value. Press **[enter]** after each modification or the system will remain unchanged.

Initiallize	*defaults general operation of gas, internal and external* ***only,*** *sets initial temperature*
Init-Miniflow	*defaults gas flow for internal, external* ***and*** *mini-internal flow****,*** *sets initial temperature*
Dry	*sets time at temperature for dry stage*
Dry-Ramp	*sets ramp time and final temperature for dry stage*
Ash	*sets time at temperature for ash stage*
Ash-Ramp	*sets ramp time and final temperature for ash stage*
Atomize	*sets time at temperature for atomize stage*
Atomize-Ramp	*sets ramp time and final temperature for atomize stage*
Burn	*sets time at temperature for burn-up stage*
Burn-Ramp	*sets ramp time and final temperature for burn-up stage*
Cool	*sets cool time and temperature*
*A**utozero***	*time-based event (usually 3 seconds before atomization)*
Data-Start	*time-based event (begin just before atomization, time delays atomize step)*
Data-Stop	*time-based event (ends data collection)*
Int-Flow-On	*turns internal internal gas on (mini-flow stays on)*
Int-Flow-Off	*stops internal gas flow (mini-flow stays on)*
Alt-Flow On	*turns alternate gas flow on (mini-flow stays on)*
Alt-Flow-Off	*stops alternate gas flow (mini-flow stays on)*
End	*default command to stop furnace program*

To exit from any screen to the previous screen after changes have been made, press **[esc]**. The new settings will be active the next time you start the furnace. After all changes have been made, press **[esc]** from the main controls menu to return to the Active Analysis Screen.

SECTION 4.4: Setting Furnace Analysis Parameters—[smpls] key

Additions to the flame menu are as follows:

Extra Sample/Dry Cycles: Normally set to 0. If the samples you are running are below the detection limits of the instrument you can concentrate the sample by making numerous injections. The furnace will dry each injection without performing an atomization. After the sample has been loaded the number of times specified the rest of the furnace program will run as usual.
Dry Cycle Cool Time: Used in conjunction with ***extra sample/dry cycle.*** This is the time in seconds from the end of the dry cycle to the introduction of the next injection.
Rinse Time: The amount of time the autosampler rinses after an injection.
Sample Volume: Amount of sample to be injected. 20uL is the standard volume but up to 50uL can be used. Be aware that if you increase the volume you will have to lengthen the dry step in the furnace program.
Matmod Spec: (optional) The name of the matrix modifier can be entered.
Matmod1 Cup: Enter the tray position that the modifier is in. 47 is default.
Matmod1 Volume: Enter the amount of modifier to add to each standard/blank/sample. 5uL is the normal volume. Enter 0 if no modifiers are being used or you have pre-mixed the modifier into you standards, blank and samples.
Matmod2 Cup: Enter the tray position that the 2nd modifier is in if you are using one.
Matmod2 Volume: Enter the amount of the 2nd modifier to add.
Set Matmod Cup Defaults: Sets modifier cup positions back to default .

After all changes have been made, press **[esc]** to return to the Active Analysis Screen.

SECTION 4.5: Calibration and Analyzing Samples

Matrix modifiers for furnace work

When running many elements by graphite furnace, it is necessary to add matrix modifiers to all standards including the blank as well as your samples. Their purpose is to normalize the desired element into a form that is not easily volatilized (from halogens in the ashing step) or into a form suitable for atomic absorption (as in preventing carbides due to organic carbon). The following procedures are general recommendations for using matrix modifiers for improving performance in graphite furnace applications. These formulations will also serve to stabilize the metals in some inert form so a clean sharp atomization is seen. They are suggested starting points and the actual conditions for optimal analyses with your samples may vary. Try adjusting the levels of matrix modifiers added by 20% and see if stability or sensitivity of the signal improves, if so change it another 20% till you see no change or a decrease in quality of the data.

500 ppm Mg-NO_3 + 2000ppm NH_4-PO_4: Universal matrix modifier for:
Transition metals: Cr, Mn, Fe, Co, Ni, Zn
Heavy metal (TCLP): Cd, Pb, Tl, Sn

1000ppm Mg NO_3 + 200ppm Pd NO_3 OR 2000ppm Ni-NO_3 for all hydride forming metals for:
As, Se, Sb, Te rarely

For samples with high levels of salt (NaCl): Seawaters, high saline sol, physiologicals (serum, blood, urine):

Add 1500ppm NH_4-NO_3 matrix modifier solution to eliminate halogen losses, can use in conjunction with other modifier solutions. Can use up to 4000ppm for very high levels of salt.

For samples with high levels of biological/organic carbon: Serum, urine, blood, high algae water, foodstuffs:
Make prepared solutions to contain 2-5% HNO_3 to help oxidize the organic carbon and reduce carbide losses, can use in conjunction with other matrix modifier solutions.

General recommendations:

Refractory metals (Ti, V, Cr, Si, Al, Mo, W, Ba) will form carbides unless nitrate (NO3 salt or acid) is present. 0.2% Nitric acid should be added in addition to matrix modifiers. For the hydride metals, the preferred modifier is 1000ppm Mg-NO_3 + 200ppm Pd-NO_3 since palladium will have the same basic affect as Ni-NO_3 and not contaminate the graphite tube with nickel for future analyses. Most of the heavy metals form volatile halides, so be sure that there is no salt left after the ashing step (you can see the smoke coming out the top of the graphite tube).

Graphite Tubes:

There are three basic types of tubes used in the graphite furnace:

Pyro-Coated Tubes are used for most elements. The pyrolytic graphite coating improves the sensitivity of carbide-forming metals

Un-coated Tubes are more economical and provide greater reproducibility for certain elements including Lead and Cadmium.

Pyro-Platform Tubes provide a flat surface for sample injection. Although these tubes are generally more expensive, they provide greater longevity and possibly better stability.

Before calibration, it is best to make sure the best tube for your analysis is installed and that it is in good condition. See Section 11.2 for instructions on changing tubes.

Gas Flow Settings

Note: The furnace must be activated using the **Read or Start** key before flows may be adjusted.

The Buck 220GF Power Unit has 4 flow meters on the front:

1) *Sheath Gas* is Argon flow outside the tube. It's purpose is to prevent the Oxygen in the atmosphere from causing the rapid combustion of the Graphite Tube. The Sheath Gas should be set to 1.25 mL/min, or about half way up the scale.
2) *Internal Flow* is Argon flow inside the tube. The purpose is to remove unwanted compounds from the furnace tube as they are vaporized or ashed. The Internal Flow should be set to 200 uL/min. The internal flow is turned off during Atomize to allow data collection.
3) *Mini-Flow* is also Argon gas flow in the tube, but the flow remains ON during atomize. Mini-Flow is used to reduce the sensitivity of the Analyte and/or peak duration. The flow is normally set to 50 uL/min to start, then increased to obtain the desired peak.
4) *Alternate Internal Flow* is the optional flow of another gas (possibly Oxygen or Air) used during the analysis of special types of samples.

Calibration

1) Make sure the argon regulator is set to 40 psi and the valve is open. Check that the cooling water is flowing (about 1.5 liter/min is sufficient).

2) If you would like to use the *Auto-peak Timing*, make sure that ***Peak Timing*** is set to ***Auto*** in the ***Furnace/ Vapor Controls*** menu, then skip to step 4.

3) If you are setting the peak timing manually using the fixed timing mode, make sure that ***Peak Timing*** is set to ***Fixed*** in the ***Furnace/Vapor Controls*** menu. Read your high standard using the **[read]** button. After the furnace program ends, Press **[esc]**, then **[cntls]**, then highlight ***Furnace/Vapor Controls*** and press **[enter]**. Use the ***Replot Last Peak*** function to examine the peak and select the proper peak delay and duration so that the entire peak is integrated including the tail. Press **[esc]** then enter the values in for peak delay and duration. You may return to replot last peak to check to see if the white bar, which denotes the area of the curve which will be integrated, covers the entire peak.

4) Press the **[cal]** button. By default the blank is in cup 41 and std's 1-3 are in cup's 42-44 but you can specify any location. The blank must be in the top location on the calibration form. Enter in the concentration values and curve parameters just as with flame (see section 3.3). For best results, run at least 3 replicates for your blank and standard when running a graphite furnace analysis. The absorbance for your blank will be taken into account when the calibration curve is calculated.

5) Press the **[start]** button. If you have an Autosampler, it will first inject the high standard and run the furnace program. If you are using *Auto-Peak Timing*, the software will determine the best integration window for the peak then continue on with the blank and all other standards.
If you don't have an autosampler , the software will give directions at the bottom of the screen to instruct you when and which standard to pipette by hand.

6) When the standards are done, you can examine the data for inconsistencies. If one of the standards is not consistent, you can read that standard again by using the arrow keys to highlight the standard and press read. The software will automatically run the same # of replicates for the standard. After you are satisfied with the data, press **[cal]** to calculate then **[enter]** to view the plot. If the curve is satisfactory, press **[esc]** 3 times to return to the ***active analysis screen***.

Automated Analysis

If you will be using an autosampler and you wish to run fully automated press the **[start]** key, enter the cup number you would like to start with and your samples will run according to the protocol you set in the ***Smpls*** page (see Section 3.1). You can run manually with an autosampler if you turn ***read key sampling*** on. In this mode, after pressing **[read]** the screen will ask you to enter a tray location. Enter a number then press **[enter]**.

Manual Analysis

1) If you do not have an autosampler you can still use the **[start]** key. The screen will prompt you to inject samples according to what you entered in the ***Smpls*** page. Using the **[start]** key, the system will instruct you to run the specified number of replicates for all samples. Simply inject your sample when prompted and press the **[enter]** key. The software will automatically turn off the internal flow for better manual injections.
2) If you do not need to set up sample ID's or you only have one test sample to run you can skip the ***Smpls*** page. Just run your standard curve as usual then go to the Active Analysis screen. With ***read-key sampling*** OFF, inject your sample and then press **[read]**. If you would like a printout turn ***read key printing*** on. The software will not turn the flow off for a second injection.
3) If you run samples using the **[read]** key with ***read-key sampling*** ON, you can run any number of replicates for each sample and the software will automatically turn off flow before each injection. Sample Ids will be used, but if they are not necessary tey will default to S1-S40. Turn ***read key printing*** on to print the results.

SECTION 5: Running Cold Vapor and Hydride

Cold vapor for mercury and hydride for arsenic, and selenium uses very much the same graphics technique as graphite furnace in that a peak of a fixed duration will be obtained as apposed to flame where there is a steady state signal as long as you are aspirating something. The difference is that a chemical reaction in the reaction vessel will cause the peak as opposed to using the furnace or furnace program.

1) Setup the cold vapor or hydride system as explained in the respective manuals.
2) Select the (Hg-CV) file from the 210 library if running cold vapor for mercury. Select (As-hydride) or (Se-hydride) if running arsenic or selenium by hydride. Peak up the lamp and wavelength as normal and allow the system to warm up.
3) Setup the ***cntls*** and ***smpls*** page as you would for flame. The peak timing is normally preset for the maximum range allowed in the library since Cold Vapor and Hydride Peaks are very broad
4) Press the **[cal]** key to enter the calibration screen and enter your concentration values. Press **[a/z]** to zero the absorbance.
   Note: The cold vapor and hydride techniques are very dependent on timing for proper quantitation. Try to use the same timing between injecting and pressing the **[enter]** key.
   Note: When running mercury cold vapor it's always a good idea to press **[a/z]** before reading standards, blanks or samples because due to the nature of mercury the lamp will most certainly drift during the run.
5) Press **[start]** and the software will instruct you to inject the reagent (as specified in the accessory's manual). Inject the reagent into the flask with your high standard and press **[enter]** immediately following injection. A graphics screen 28 seconds in length will appear and as the reaction takes place the peak will appear. The integration window (bar on the top of the screen over the peak) should span most of the way across this screen.
6) Wait for the absorbance reading to come back down near zero then press **[a/z]** again to zero the absorbance. Inject the reagent into your blank and immediately press **[enter].**
7) Continue this procedure for all remaining standards running them from low to high.
8) Calculate and view the plot just as with the flame or furnace.
9) When running samples you have the same options for manual analysis as with the flame (see section 3.3). The software will instruct you to inject the reagent for each sample. The reslope function only works for the flame analyses since this is the only method using a "forced zero" calibration curve.

SECTION 6: Editing the Library

Passwords

There are two different levels of passwords that can be used on the Buck 210 AA. Both Level 1 and level 2 can be used to make changes in the file libraries as well as other parameters. The two passwords and their differences are as follows:

- Level 1 (**pw1**)

A "pw1" password can make any changes, save, and delete element libraries that have been saved by a level 1 password only. "pw" **must** be typed using the lowercase key.

- Level 2 (**PW2**)

A "PW2" password can make any changes, save, and delete element libraries that have been saved under a level 1 or a level 2 password. "PW" **must** be typed using the uppercase key.

Once a password is entered, the Buck 210VGP remains in "password mode" until the 210 is turned off so you'll only have to enter a password once while you're optimizing your library file. You enter a password by pressing the **[LIB]** key while on the active analysis screen. Select option **<*3*>** ***Enter A Password***, then type the password and press enter. Options for saving and deleting library files will then appear on the menu.

Changing Measurement Controls

Certain settings may be stored in the library so they are automatically loaded every time you run that analysis. If you change one of these settings and then load a new element library in the same lamp position without saving it you will lose the changes. The majority of the settings that can be saved are in on the Measurement Controls menu.

1) ***Library Name;*** Many operators who run only a few elements like to keep them grouped together in the library so they are easier to find. The library name is limited to 16 characters. You may, for example, start each library name with the initials for your company. You MUST change the library name before you can save changes. The Library name can also be changed on the ***LIB*** menu.
2) ***Method;*** If a element does not have a library with the method you wish to run, you can create a new library with the desired method. For example, If you load the library for Ca_D2_422.7 (Air/Acet Flame Meth), you can change the method to *Graphite Furnace* on the Measurement Controls menu. Simply highlight the existing method and toggle through the method options using the arrow keys and press **[enter]** when you get to the method. Not all elements can be run using all methods so you should check a applications resource before proceeding. Graphite Furnace programs and other settings unique to the new method may have to be entered before you can obtain accurate results.
3) ***Lamp Currents;*** If a lamp from another manufacturer is being used which requires a different lamp current setting or your Buck lamp needs to have the current adjusted, you can change lamp current on the ***measurement controls*** screen. Change the ***Peak pulse*** value so that the ***ave current*** is the value desired. (NOTE: ave current = 25% of peak pulse). For example, if a 5ma ave current is desired type **[020]** then **[enter]** for peak pulse. The ave current will change to 5 when **[enter]** is pressed.
4) ***PMT Volts;*** This is the initial PMT voltage setting when the library is loaded. You will not normally change this setting since the PMT voltage is set automatically when you Autozero.
5) ***DC Suppress;*** Preset for best results, no need to change this setting.
6) ***Data, period;*** Preset timing variables, normally no need to change this setting
7) ***Integration Time;*** The time the software integrates when you press **[read]** during a Flame Analysis. You can increase the integration time to improve reproducibility.

8) ***Maximum Peak Timing;*** Sets the length of the peak window for Furnace and Vapor methods. Select 14 or 28 second windows using the arrow keys.
9) ***Background Gain;*** Preset for best results. Automatically set with Autozero from the align screen.
10) ***D2 Background;*** Preset ON for elements which can make use of it.
11) ***Wavelength, Slit;*** Designations you can change for libraries using alternate wavelengths.
12) ***Lamp;*** Designation you can change for library using a different lamp.

Calibration Curves

Calibration parameters (options under calibration controls except replicates) can be saved for the active analysis. The points for the calibration curve will not be saved. However, the curve without the points can be viewed for future reference.

Saving Libraries

You cannot overwrite the master library files that are preprogrammed into the 210VGP. However, you can save this information by creating a new library name and saving the current analysis information under this new name.

After making your changes, use the **[esc]** key to get back to the active analysis screen, then press the **[lib]** key. Enter the desired password by selecting **<3>**.If you have not already changed the name under *Measurement Controls* , select **<1>** ***Enter a library name***. The current library name will then come up (Example : *Pb-Furn3-283.2*). You can then use the right arrow key to move the cursor to the part of the name you'd like to change. Then, using the **[del]** key, you can erase some or all of the name. After typing the new name, it may look like this example: *Pb-Blood-283.2* (in this case the new name describes a library for running lead in blood). To save the changes, select **<4>** ***Save Active Analysis***, and respond by pressing the **[yes]** key when prompted. You can delete a Library you've created by selecting **<5> Delete Active Analysis.** You cannot alter or delete the master library files.

SECTION 7: Method of Standard Additions

If the sample matrix is unknown and interferences are likely, the method of standard additions may be used instead of the standard calibration technique used by the Buck 210's internal software. The Buck 210 cannot read out the concentration of a sample using this method. However, the results may be tabulated manually from the Buck 210's absorbance reading using one of the following procedures.

SA.1 The standard addition techniques involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots of the sample solution, each of volume Vx, are taken. To the first (labeled A) is added a small volume Va of a standard analyte solution of concentration cs. To the second (labeled B) is added the same volume Vs, of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration cx is calculated:

$$c(x) = \frac{S(B)\ V(s)\ c(s)}{(S(A) - S(B))Vx}$$

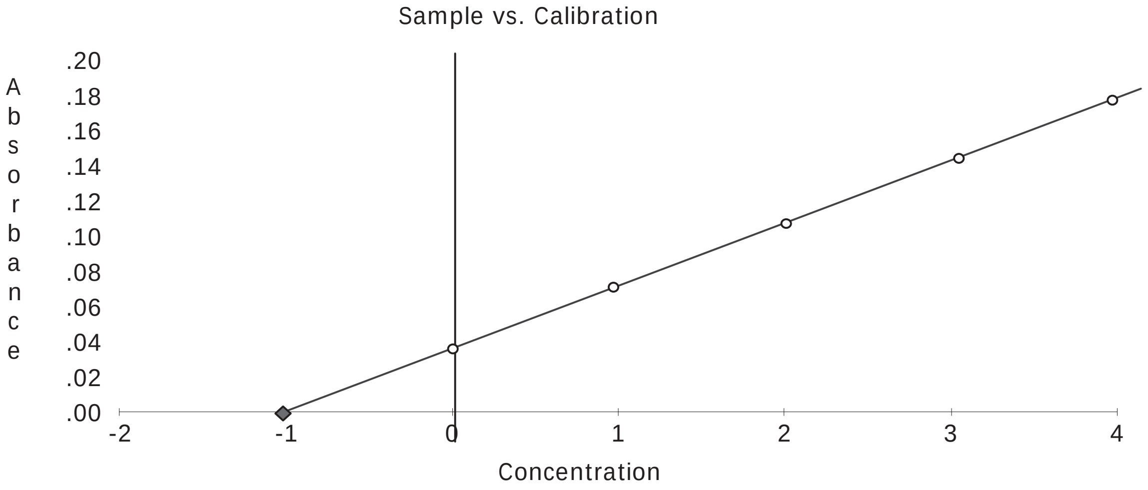
where S(A) and S(B), are the analytical absorbance readings (corrected for the blank) of solutions A and B, respectively.

Vs, and cs, should be chosen so that S(A) is roughly twice S(B) on the average. It is best if Vs, is made much less than Vx, and thus cs, is much greater than Cx, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. The absorbance data must be corrected for any additive interference.

When greater accuracy is required, the method of standard addition **SA.2** found on the following page is recommended:

SA.2 Add equal volumes of deionized water and three standards containing different amounts of the test element to 4 aliquots of the sample. The aliquots must also be of equal volume. Read the absorbance of each solution and plot as shown below. The concentration of the standards is taken as the *X* value, with the sample assigned the value *X=0.* When the resulting line is extrapolated back to zero absorbance, the point of intersection with the horizontal axis is the concentration of the unknown.



(*courtesy of BUCK SCIENTIFIC, Inc. Applications Department, Norwalk, CT*).

The method of standard addition is subject to certain limitations, which must be taken into consideration when examining the results. The curve must be within the liner range of the analysis. For the best results, the slope of the curve should be nearly the same as that of the standards alone. The diagram above shows a typical relationship between the sample analysis (upper curve) and the curve of the standard solutions.

If the slope of the standard addition curve differs by more than 20% of the standard curve, the results are suspect. In addition, the effect of interferences should not vary with concentration of the analyte or other components in solution. Spectral interferences are not corrected for by this method; use suitable background correction (i.e., deuterium, giant pulse, etc.).

Graphing the results enables the analyst to visually determine the validity of the results by checking for linearity, and by comparison with a curve of the standard solutions; however it leads to some uncertainty in determining the concentration of the unknown. For the highest precision, the unknown should be determined by calculation from:

$$[u] = 0.25\{(\Sigma x) - [\{4\Sigma x^2 - (\Sigma x)^2\}/\{4\Sigma x\Sigma xy - \Sigma x\Sigma y\}]\}$$

where: [u] is concentration of unknown
[y] is an absorbance value for each corresponding concentration, x

SECTION 8: Flame Techniques

OVERVIEW

This section describes standard conditions for Flame Atomic Absorption Spectroscopy (FAAS) techniques. These ttechniques utilize combustion mixtures of either air-acetylene (A-A), nitrous oxide-acetylene (N-A) or argon-hydrogen (Ar-H). While nearly all elements can be determined in an A-A flame to some extent, this is often not the best type of flame to use. The flame mixtures given in this section are those which provide the greatest sensitivity for each element. In order to provide good sensitivity, an optimal combustion mixture will have the following characteristics:

1. reaches an appropriate temperature for excitation of the analyte.
2. supplies chemical agents necessary to convert or stabilize the analyte in the atomic form.
3. reduces or eliminates spectral and/or chemical interferences.

The working temperature and ranges of the various flame types are given below, with the oxidizing gas ratio having the hottest temperature in each range. Both chemistry and temperature are influenced by the oxidant-to-fuel ratio. A fuel rich acetylene flame provides a highly reducing environment due to the excess amount of carbon radicals. This suppresses the ionization of easily oxidized elements and results in greater sensitivity for elements such as chromium and tin.

An oxidizing flame burns hotter than a reducing flame and creates less spectral interference in the near UV for elements such as nickel and zinc, which are not so easily ionized. The hotter temperature provides a greater proportion of excited atoms to the analysis, thereby increasing the sensitivity for these elements.

Characteristics of Different Combustion Mixtures

Oxidant	***Fuel***	***Average Temperature***	***Temperature Range***
Air	Acetylene	2300	2120 to 2400
Nitrous Oxide	Acetylene	2750	2650 to 2800
Argon/Air	Hydrogen	400	350 to 1000

There are about 13 elements that form refractory oxides and cannot be dissociated in even the hottest air-acetylene flame. It is necessary then to use a nitrous oxide-acetylene flame for these elements. The N-A flame has the advantage of being able to decompose refractory compounds, but suffers from relatively higher noise caused by emission radiation from combustion by-products (CN, CH and NH). These by-products can also cause specific interferences with some elements where the emission spectrum overlaps an absorbing line. Sometimes this type of interference cannot be removed by background correction, making analysis virtually impossible. In the very hot N-A flame, an ionization suppressant must be added to the sample (usually a Potassium or Lanthanum salt) to prevent the analyte from being lost to the analysis through ionization.

Elements with characteristic wavelengths near the start of the vacuum UV range show considerable improvement in sensitivity with an argon-hydrogen flame. It is not yet certain what atomization mechanisms occur, however, it is generally agreed that hydrogen has an active role in the process. Because of it’s very high transparency the Ar-H flame gives particularly good sensitivity for Arsenic and Selenium.

A more sensitive technique, hydride generation, doesn’t require any modification of the combustion mixture. In this technique Arsenic and Selenium are converted to the Arsine or Selenine gas and swept into a quartz cell heated by the flame. The hydride technique is used when highest sensitivity for these elements is required. In

A more sensitive technique, hydride generation, doesn’t require any modification of the combustion mixture. In this technique Arsenic and Selenium are converted to the Arsine or Selenine gas and swept into a quartz cell heated by the flame. The hydride technique is used when highest sensitivity for these elements is required. In this case the A-A flame is used but only serves as a convenient source of heat. The technique uses sodium borohydride to reduce the As or Se to free metal then delivers it to a Quartz “Tee Tube” which is warmed by the flame in beam of the Atomic Absorption Spectrophotometer. Buck Scientific offers the Model 1018 Hydride/ Cold Vaporaccessory which provides gas controls, glassware and mounts for this analysis. This system makes use of the Buck 210VGP’s “peak-picker” software to analyze the data. Buck Scientific also offer the Model 420 Continuous Flow Hydride system which allows you to analyze data using flame integration.

Since elemental Mercury has a significant vapor pressure at room temperatures, and is extremely volatile at 500 C, it is best performed using a flame-less technique. The most common is the Cold Vapor technique which reduces the Mercury to free metal in an acid solution using stannous chloride or sodium borohydride then delivered to an absorption cell mounted in the beam of the Atomic Absorption Spectrophotometer. Buck Scientific offers the Model 1018 Hydride/Cold Vapor Accessory for Mercury analysis using the Buck 210VGP’s “peak-picker” software.

SECTION 9: Interferences

There are basically three categories of interferences that can occur in flame atomic absorption work, termed ***physical***, ***chemical***, and ***spectral***. Chemical interference is most often encountered and is caused by lack of absorption of atoms bound in molecular combination in the flame. This occurs when the flame is not hot enough to dissociate the molecule. Phosphates interfere with Magnesium, Calcium and Barium, and are overcome by adding Lanthanum to the solution. Similarly, Silica interferes in the determination of Manganese and can be eliminated by the addition of Calcium.

Forming other compounds with the interfering materials may also eliminate chemical interferences. Although matrix modifiers are primarily added to increase the sensitivity of the analysis, they may also be used to eliminate or reduce interferences.

Highly refractory metal oxides, especially those of the rare earth metals, do not dissociate at the temperature of an air-acetylene flame. Other metals dissociated into the atomic state often recombine with oxygen in the flame so rapidly that further atomization is not possible. In these cases an alternate combustion mixture is used, most often a nitrous oxide-acetylene flame, to provide greater heat for decomposition.

If an element in the atomic state becomes ionized in the flame it’s absorption spectra will change, effectively removing it from the analysis. The fraction of ionized atoms in the flame increases with increased temperature, and at the heat of a nitrous oxide-acetylene flame nearly all elements are significantly ionized. This type of interference is most pronounced for elements such as Barium, which is readily ionized but requires high temperature excitation for analysis at the usual concentration range. Ionization can generally be controlled by the addition of a large excess (>1,000 mg/L) of an easily ionized element such as K, Na, Li or Cs to the sample.

All metals are not equally stable in a digested solution, especially if it contains only nitric acid, and not nitric and hydrochloric acids together. The digestate should be analyzed as soon as possible, with preference given to Antimony, Barium, Molybdenum, Silver and Tin. Precious metals are less likely to plate on to plastic containers than glass when making dilutions.

High concentrations of dissolved solids in the sample may result in interferences from physical (non-atomic) absorbance such as light scattering. If background correction is not used, the sample can be re-analyzed at a nearby, non-specific wavelength*. If an absorbance is found at this wavelength, it is due to a physical effect and the sample should be diluted and/or treated by a filtration, digestion or extraction procedure to remove the interference.

* All hollow cathode lamps emit not only the line spectra of the element comprising the cathode, but also that of the fill gas and other incidental impurities; therefore, it is always possible to find an energetic line somewhere near the resonant wavelength of the element of interest which will not respond to the element, but will respond to physical interferences.

Physical Interference can occur with some sample matrices when the instrument is calibrated using aqueous standards. Samples and standards should be monitored for viscosity differences that may alter the aspiration rate. These interferences may be eliminated by diluting the samples or by mixing standards in a similar matrix.

Spectral interference can occur when an absorbing wavelength of an element present in the sample but not being determined falls within the width 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. Interference can also occur when resonant energy from another element in a multi-element lamp, or from a metal impurity in the lamp cathode, falls within the band-pass of the slit setting when that other metal is present in the sample. Narrowing the slit width may sometimes reduce this type of interference.

Molecular spectra of certain common compounds have broad absorption profiles and can produce a positive interference; that is, the measured absorbance is greater than the actual absorbance of the analyte. The table below illustrates some common molecular absorbance bands:

Table 3: Overlapping Spectra of Some Common Analytes *(Source: Norris & West;* ***Analytical Chemistry****; 1974, V46, p. 1423).*

Analyte	**Wavelength**	**Overlapping Element**	**Wavelength**
Aluminum	396.15	Fe	396.11
Bismuth	206.17	I	206.16
Calcium	422.67	Ge	422.66
Cadmium	228.80	As	228.81
Chromium	359.35	Hg	359.35
		Ne	359.35
Copper	217.89	Sb	217.92
	324.75	Fe	324.73
	324.75	Eu	324.75
	327.40	Fe	327.45
Cobalt	253.65	Hg	253.65
	241.16	Pb	241.17
Iron	213.86	Zn	213.86
Lead	217.00	Sb	217.02
Lithium	323.26	Sb	323.25
Magnesium	285.21	Fe	285.21
	285.21	Hg	285.24
Manganese	279.48	Fe	279.47
	403.31	Ga	403.30
Mercury	253.65	Co	253.65
Nickel	231.10	Sb	213.15
	352.45	Fe	352.43
Palladium	247.64	Pb	247.64
Platinum	271.90	Fe	271.90
Silver	338.29	Fe	338.24
Strontium	460.73	Fe	460.77
Vanadium	250.69	Si	250.69
	308.21	Al	308.22
Zinc	213.86	Fe	213.86

SECTION 10 - TROUBLESHOOTING, TIPS & TECHNIQUES

Section 10.1: Low Absorbance / Stability

About 95% of problems are related to the burner system or the lamps. The instrument's electronics and optics rarely fail.

PROBLEM: LOW ABSORBANCEPOSSIBLE CAUSES:

1) Wavelength is not tuned in correctly or is peaked on the wrong spectral line. Some elements have many spectral lines in the same region, lines other than the primary line may give you much less absorbance than the primary line. This is common with nickel. Any element that calls for a 0.2nm slit will have more than one spectral line in the region.
2) Nebulizer either blocked, not tuned correctly or needs replacement due to extended use. Check for blockage in the sample capillary (this usually occurs where the plastic capillary meets the nebulizer). The uptake rate for the nebulizer is typically between 8 - 10 ml/min. With a burner head that is cold turn on the air (no fuel) and aspirate water. On a well peaked nebulizer you should see a good mist coming from the burner head slot. As a nebulizer degrades you may notice that the flow rate required for peak sensitivity increases.
3) Burner system out of alignment. For maximum sensitivity the path of the burner slot must be directly underneath the path of the light beam. Refer to Section 2 of this manual for alignment instructions.
4) Fuel / air ratio not correct. Most elements work well with a lean blue flame , however some elements may give better sensitivity with more or less fuel. Any element that specifies a rich yellow flame condition needs higher fuel settings to achieve the sensitivity stated in the standard conditions section of the manual. Refer to this section for suggested flame conditions. If you have problems meeting the sensitivity spec, experiment with flame condition for best results (see section 3.2).
5) Burner height not correct. Certain elements may also work better if the burner head is lowered. If you increase fuel flow chances are you will need to lower the burner head as well for peak sensitivity.
6) Acetylene tank low. As the acetylene pressure drops you may encounter a decrease in absorbance and an increase in background due to acetone. Since the amount of acetone in the acetylene varies, we recommend you change the tank when the pressure drops below 75psi. Failure to do so may also result in damage to the flame controls in the instrument.
7) Impact bead not adjusted correctly. This should not normally need adjustment unless the nebulizer has been removed and replaced or the bead has broken. The impact bead is located at the rear of the spray chamber directly across from the nebulizer. To adjust the bead peak up on any lamp and appropriate wavelength, make sure the background corrector is off then autozero the instrument. With a cold burner head turn the air on at the front of the instrument and aspirate water (DO NOT TURN ON THE FUEL AND LIGHT THE FLAME). You should see a mist coming from the burner head. If not adjust the nebulizer for best absorbance on the main display then using a ½" wrench adjust the impact bead for best absorbance. (NOTE: DO NOT ADJUST THE BEAD TO FAR CLOCKWISE OR IT MAY RUN INTO THE NEBULIZER AND BREAK). If you are unsure as to the proximity of the bead to the nebulizer you can remove the spray chamber and remove the blow-out plug on the left side. The bead should be a couple of millimeters from the end of the nebulizer.

PROBLEM: DRIFT OR FLUCTUATION IN READINGS
POSSIBLE CAUSES:

1) Lamp. To determine if it is the lamp turn off the flame and all gasses. Zero the absorbance and watch the display. After warmup drift should be less than 0.001 per minute and noise should be less than +/- 0.002. Most lamps perform much better than this. If this is stable the problem is probably with the burner system.
If a lamp drifts or is noisy, selecting a different operating current may help. Lamp energy values for the 210VGP should be 3.000 or greater in a 0.7nm slit and in the upper 2's to low 3's for a 0.2nm slit.
2) Burner system. If drift or noise only occurs during your run then the burner system is in doubt. Check that the drain is working properly. There should be a steady drip or flow. If there is any water buildup in the drain block you will most certainly get a decrease in absorbance. A gurgling sound from the burner is a good indication of this. Make sure the small clear tubing is in the drain elbow to eliminate air bubbles in the elbow. Make sure the end of the drain tube is not submerged in the waste water. Try readjusting the nebulizer. A new nebulizer may be needed. Make sure there are no leaks in the burner, check the o-rings.
3) Thermal drift. If the lab is subject to temperature changes the optical bench of the instrument may shift causing a slight change in energy. To determine if this is the problem peak up the wavelength for maximum energy and zero the absorbance. If after a period of time the absorbance drifts go back and repeak the wavelength. If you can adjust it for more energy then the wavelength has drifted off peak resulting in an absorbance change.
4) Vibrations in the lab can also cause wavelength drift. Make sure any vibrating equipment including air compressors are not near the instrument.
5) Unstable supply gas pressure. Although the instrument has internal regulation, supply pressure change can cause fluctuation of absorbance for any element that is flame sensitive, particularly iron. Air supply: Many failures of the pneumatics or excessive noise in results can be attributed to contaminated air or acetylene. An air filter is a must when using an air compressor for your supply to filter out oil, water & particulates. It should be cleaned on a regular basis. The inside of the plastic bowl should be cleaned with water and soap and the filter element with ethyl alcohol or similar solvent. Refer to the manufacturers instructions for complete information.

PROBLEM: YELLOW / ORANGE FLAME
POSSIBLE CAUSES:

1) Acetylene: If you notice your flame becoming orange in color and it is not due to your samples there is acetone coming from the tank, you should shut down when this is noticed. A new tank should sit for at least several hours undisturbed before use to let the acetone settle. Eventually liquid acetone will appear in the flow tube of the acetylene. For this reason do not let tank pressure drop below 75psi. As your tank pressure drops more acetone will be introduced resulting in decreased absorbance signal and increased background levels.MISCELANEOUS:

1) A 0.500 absorbance screen is supplied with the instrument. It should result in .450 to .550 absorbance when inserted into the light path. This indicates the electronics are working properly.**2)** Your standards, samples and blank should be prepared in the same matrix as your samples so
as to avoid erroneous results.

SECTION 10.2.1: Error messages

NOTE: If the instrument has been set up correctly pressing **[a/z]** in “Active Analysis” screen should clear most error messages.

LAMP CURRENT ERROR

1) The Hollow Cathode (element) Lamp is not working
2) The working lamp position does not match the lamp selected in the software (ie; the lamp number on the top of the active analysis screen should be the lamp position in use as well as the number on the plug.
3) If the message goes away when the D2 Lamp is turned off, check to see if the background corrector lamp is not functioning.
4) If the lamp is flickering or unclear there is a good chance the lamp is bad.
5) If the lamp is flickering on and off when you touch the connector, the lamp connector (#210-0550) may need to be replaced. If possible, test another lamp in the same lamp position to verify the problem.
6) If the lamps are lit and are not flickering or unclear, there may be a problem with the Power Control PCB (#210-2006). If possible, test another lamp in the same lamp position to verify the problem.

UNDER RANGE ERROR

1) If the error occurs while aspirating a sample, a sample of too high a concentration has been aspirated which has absorbed all the energy from the lamp.
2) If the error occurs with the flame off, something is most likely blocking the beam from the lamp. You may use a white business card to check the beam from the lamp.

CONCENTRATION OVER RANGE

After calibration has been performed a sample has been aspirated which is greater than the highest standard.

OVER RANGE ERROR

Lamp energy has increased for some reason in the **[active analysis]** mode from what it was in the **[align]** mode. Pressing **[a/z]** should re-set the gain circuitry and clear the error.

RESLOPE ERROR

The standard that you are trying to reslope on is either incorrect (not the high standard) or the absorbance has drifted dramatically from what it was when the calibration was performed.

AUTOZERO ERROR

The blank has drifted dramatically from what it was or the signal is fluctuating too much. The error may be caused by a bad lamp or a contaminated blank if the error only occurs when aspirating the blank.

BACKGROUND ERROR

The signal from the D2 lamp has changed. Pressing **[a/z]** should reset the gain circuitry and clear the error.

CAN’T DIRECT REPORT

The user is trying to output data but the printer or remote computer has not been turned on in the software. A printer or remote computer must be connected to use the **[START]** function from the “Active Analysis” screen.

PRINTER ERROR

The instrument is trying to print but the printer is not responding.

FURNACE NOT INSTALLED: Furnace Accessory “OFF” on the initialization screen. Turn the Buck 210 off, then hold the uppercase key down and turn it back on to get to the initialization screen. After turning the option “ON” with the left or tight arrow key, you must press <enter> to lock in the setting. Press <esc> to return to Active Analysis

VERY LOW ENERGY or NO ENERGY

1) Check that the lamp turret # and socket # being used match the lamp number at the top of the screen. Only one lamp at a time is electrically modulated so if the incorrect position is selected you will receive a “low energy error” or “lamp current error”. The lamp # is changed by pressing the “sel” button in either the “active analysis” or “lib” mode.
2) Pull the lamp out of the turret and reconnect then look at the lamp. The image should be a bright dot in the center, the image should not be fuzzy, diffused or arcing to the glass.
3) While looking at the lamp press the “sel” key continually from the “active analysis” screen. When the lamp number at the top of the screen matches the lamp you are looking at you should see a change in brightness of the lamp. This indicates that the lamp is being modulated and that the lamp circuitry is probably functioning correctly.
4) Check that the wavelength and slits are set correctly.
5) Make sure the proper element lamp is installed.
6) Check all lamp positions. It is possible that only 1 position is not working correctly.
7) If the problem only occurs when the background corrector is on then there may be an energy mismatch between the hollow cathode lamp and background lamp. Autozero from the Align screen should correct the gain to offset this mismatch.
8) Turn the wavelength to read 000. At or very near this wavelength the grating is parallel to the slits and the energy should be very high (5.0 or greater).
9) Remove the top right cover of the instrument and check that the wavelength mechanism appears to be operating as you turn the wavelength knob. The Grating arm should be perpendicular to the wavelength micrometer such that the stainless steel ball lines up with it.

SECTION 10.2.2: Software Reset

Should the text on the screen become corrupted or missing characters are noted you may be able to restore the original library by using the following procedure:

1) Turn power off.
2) Press and hold the **[upper case]** key and turn on power.
3) When the "Initialization Menu" appears on the screen release the **[Upper Case]** key.
4) Scroll to (enter password) and press **[enter]**.
5) Type in "PW2" holding **[upper case]** for the "PW" and releasing for "2".
6) Press **[esc]**.
7) Scroll to "Restore Default Settings" and press **[enter].**
8) Press **[ESC]** to get to the Active Analysis screen.

This will load default libraries into each lamp position. You should now try to load a library into each lamp position while looking for signs of memory corruption such as strange characters. If the problem persists, contact Buck Scientific for replacement software chips (#210-0616) to correct the problem.

1) SECTION 10.3 Replacement Part Assembly Instructions

SECTION 10.3.1: D2 lamp installation and alignment

1) Remove the top left cover of the 210 be removing the 2 screws at the back.
2) Before removing the old D2 lamp, peak up the energy and wavelength and lamp alignment on any hollow cathode lamp. Note the energy value from the align screen on a piece of paper.
3) Turn off the power to the 210.
4) Disconnect the cord to the D2 and remove it from it’s holder.
5) Install the new D2 lamp in the holder and connect the cord.
6) Turn power on to the 210.
7) Press <ALIGN>
8) Align the D2 lamp by sliding the lamp back and forth in the holder and/or loosening the bracket and moving it up and down. . Roughly center the lamp so the image from the hollow cathode lamp is centered through the hole of the D2 lamp. Use the bar graph as you would to align the hollow cathode lamp alone except in this case you need to maximize energy for both the D2 lamp and hollow cathode lamp signals.
9) Fine tune the hollow cathode lamp adjustment. You should not have to make any large adjustment to maximize the HCL. The value should be very close to when the old D2 lamp was there.
10) Make sure everything is tight and re-install the cover.

SECTION 10.3.2: Impact bead alignment

NOTE: This procedure is only to be performed when a new nebulizer is installed or when nebulizer and burner alignment fail to correct the low sensitivity or air flow problem.

1) If the impact bead is in an unknown position or the nebulizer has been changed, the initial position of the bead should be checked. Remove the spray chamber from the mount and remove the blow out plug. The impact bead should be approximately 1 mm from the end of the nebulizer. Adjust as necessary and replace the burner system.
2) The burner and nebulizer should be peaked up according to the manual as best you can before performing the following procedure.
3) Make sure the burner head is cold (room temp).
4) Turn on the air only then zero the absorbance. Aspirate water and at this point you should see mist coming from the burner slot.
5) The impact bead adjustment nut is located at the rear of the spray chamber.
6) Using a half inch open end wrench (or if it loose enough you can turn it by hand) turn the nut slowly in one direction or another and note the absorbance value. Adjust the nut for the highest absorbance value you can get. Make sure not to turn the nut to far clockwise as this will force the bead into the nebulizer and break the bead.

SECTION 10.3.3: Micrometer alignment / Wavelength calibration

1) With the micrometer out of the instrument turn it to the second hash-mark from zero.
2) Dial the wavelength counter to 000 and have the slit at 7 angstrom. The right front and right top panels should be off the instrument. The right front panel should be set up in front so the keyboard can be accessed. Power should be off at this point.
3) Install the micrometer onto the fork and through the bracket on the left. Do not tighten the setscrew on the bracket so the micrometer can be adjusted.
4) Power on the 210.
5) Install a lamp, preferably with wavelength between 300-400nm, and call up the file for that lamp. Dim the lights in the room.
6) Press <CNTLS>, then go to measurement controls and set the PMT volts to –100 (to protect the PMT from excess light). Remove the long black monochromator cover.
7) At the back left of the monochrometer is the exit slit located on the slit wheel that turns. The detector is in a small black housing behind it.
8) The image of the lamp can be seen near the slit as a vertical line. The image will move as the micrometer is moved left and right.
9) Try as best to align the micrometer (left or right) so that the image is going through the exit slit.
10) Tighten the set screw on the micrometer shaft bracket so that it does not move. Do not over-tighten or the micrometer will not turn.
11) Replace the monochrometer cover and proceed to the “wavelength calibration” section.

Wavelength calibration

1) Press the (align) button and slowly move the wavelength back and forth around 000. The highest energy should occur at 000 wavelength, the numerical value for the energy should be above 5.0. If this is not so follow the procedures below:
2) Pull the wavelength knob out to disengage the gears then rotate the digits (holding the gear in place) so that 000 coincides with the highest energy reading then tighten the gear. If the highest energy appears to be below zero than the gear will have to be loosened first so that the wavelength can be turned negative to find the peak.
3) Check the upper end by tuning to the wavelength of the lamp in use and see that the highest energy occurs at the specified wavelength. Make any further adjustments as necessary.
4) Turn off power and replace covers.

SECTION 10.3.4: Removing the electronics modules

Make sure the instruments power is turned off before removing any electronic modules since High Voltage may be Present!

FRONT PANEL

1) Remove top right cover by loosening the two screws holding it in back. Lift the back of the cover enough to clear the screws, then pull the cover backward to remove.
2) Remove the 2 screws from underneath the front panel.
3) Remove the front most screw from the right side panel of the 210 with a 3/32" allen wrench (supplied in 210 packing kit)..
4) Remove the screw from the left side of the front panel. To do this the screw can be accessed through a hole in the burner shield. On older 210's the shield may have to be removed by taking out the 3 screws around the perimeter of the shield, once screws are out the shield can be removed.
5) The front panel can now be removed. If the cables need to be disconnected, label the cables before removal to make re-installation easier.

210-0616 SOFTWARE CHIP SET

1) Remove the front panel. You should not have to disconnect any cables to change the software.
2) Look for the EPROM's located in sockets U224 and U225 in the bottom left hand corner of the circuit board. These chips are rectangular and have a small white label reading "V3.XX, C&D" and "V3.XX, E&F". You can remove the chips by carefully prying them up using a flat head screwdriver, as long as it does not have a magnetic tip.
3) Install the new EPROM's in the proper location taking care not to bend any pins in the process. Keep in mind that the notch in the top of the chip should line up with the notch on top of the socket. The socket number on each chip should correspond with the number below the socket on the board. Take care to make sure you have grounded any static charge before installing the chips since they are static sensitive devices.
4) If you need to install a new 210 library chip as well, you'll find the library SRAM in socket # U222 near the EPROM's. You can carefully pry it up with a screwdriver like the others. When installing the new library chip, there is a recessed dot in the corner of the chip that should end up on the end of the socket where the notch is. Be careful not to bend any pins and double check that all pins are in the socket. Double check that the top of the chip (the end marked with the recessed dot) is on the notched side of the socket or the library chip will be permanently damaged when you power the instrument up.
5) Turn on the instrument while holding down the "upper case" key. keep holding the upper case key until the initialization menu appears on the screen. If screen is blank, double check that the chips are installed in the right location, are oriented properly and have no bend pins.
6) On the initialization screen, use the left and right arrow keys to change the furnace, furnace autosampler, flame autosampler, printer and/or clock options to "yes" and press enter after each change. Change only the options for the accessories you have.

210-2000 SYSTEM PCB

1) Remove Front panel
2) Remove the intensity knob from the front panel.
3) Disconnect the Display Ribbon Cable.

1) Remove all screws holding the board to the panel.
2) When installing the new system PCB, be careful to align the connector for the keyboard before screwing down.

210-2002 KEYBOARD

1) Remove System PCB
2) Remove screws keyboard to the front panel
3) Remove the standoffs holding down the keyboard while making note of their location.

998-1046 LIQUID CRYSTAL DISPLAY

1) Remove the System PCB
2) Remove screws holding the display to the front panel
3) Transfer the backlight power cable extension to the new display and run past the bottom of the system PCB before re-assembly.

210-1006 POWER SUPPLY

1) Remove front panel but you do not have to disconnect any ribbon cables.
2) Unplug the power connectors from the Power Control PCB.
3) Remove the screw from the bracket on the front of the power supply
4) Remove 2-4 screws holding the power supply to the 210 chassis in the rear

210-2006 POWER CONTROL PCB ASSEMBY (LAMP POWER SUPPLY)

1) Remove the Front Panel. Only the large 36pin ribbon cable going to the Power Control PCB needs to be disconnected.
2) Unplug the 3 power supply plugs from the right side of the Power Control PCB.
3) Remove the 3 or 4 screws holding the finned heatsink to the rear of the 210.
4) Slowly pull the supply outward to expose the aluminum cover but do not pull all the way out. Remove the screws holding this cover on. Again, label all cables before removal of supply to make things easier. Once all cables are removed the supply can be pulled out.

DETECTOR (PMT OR PHOTOMULTIPLIER TUBE)

1) Remove the top right cover
2) At the rear left of the optical base is a small black cover held in place with 2 screws, remove this cover.
3) “CAREFULLY”!!! remove the detector from the socket by wiggling back and forth while at the same time pulling up (try to grab the detector by its base so as not to break the glass envelope). Once removed, put in a safe place. The detector is very expensive so exercise extreme caution.

210-2004 PREAMP ASSEMBLY

1) Remove the Front Panel Assembly.
2) Remove the PMT
3) Remove the 2 screws on either side of the hole where the detector was. You will need to hold the nuts underneath the optical base to prevent them from turning. The preamp is enclosed in the small metal case. Once the screws are removed the pre-amp assembly can be taken out.

211-0621 FLAME CONTROL PCB

1) Remove left side top cover by loosening the two screws holding it down in the back. Then lift the back up to clear the screw heads, slide the panel back then lift the front up to expose the PC Board.
2) Disconnect the 4 cables to the board making note of there location.
3) Unscrew the transistor from the cover first, then the 4 screws holding down the board.

211-0623 FLAME SENSOR PCB

1) Remove the burner assembly including the X-Y Table by removing the two screw in the front of the burner compartment.
2) Remove the 5 screws securing the burner shield and slide it out far enough to access the flame sensor PCB
3) Be careful with the white wire, which is the low current 500V feed for the flame sensor which does not go down immediately after power shutdown due to capacitors on the line.
4) Disconnect the two plugs to the board and unscrew the two screws to remove the board.
5) When installing the new board, make sure the small hole in the black flame sensor cover lines up with the hole in the shield. If not, slide the sensor up or down in it's holder to align.

220GF REPLACEMENT PARTS IGBT PCB ASSEMBLY, PCB MAIN

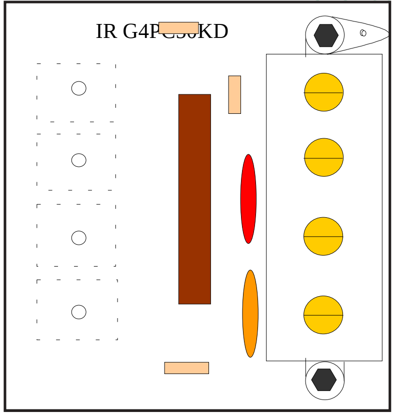
1) To find out if the IGBT assembly is bad we first need to check they're drivers. When the IGBTs burn out they almost always burn out they're drivers and/or regulators as well as possibly damaging some lands on the Printed Circuit Board due to excessive current drawn through these circuits. As a result, we recommended that the PCB be replaced along with the IGBTs. With the IGBTs (plugs P8 & P9) disconnected you can power up the bad board to see what other components are bad. **DO NOT TAKE MEASUREMENTS ON THE CIRCUIT BOARD WITH THE IGBTS CONNECTED BECAUSE THERE COULD BE LETHAL VOLTAGES ON THE BOARD WHEN THE IGBTS ARE CONNECTED.** Also and accidental shorts on the IGBT driver section of the board could blow the IGBTs if they are connected. With the IGBTs (P8 & P9) disconnected you can power up the furnace board and make the following measurements with a DC voltmeter to determine what components are blown:
    a. Measure the output voltage of each LM78L15 Regulator (U216, U221, U218) to see if they are putting out 15 volts. If any are putting out the proper voltage then that regulator and it's associated driver chip are probably both O.K.
    b. If any are not, remove the associated TC1426CPA driver chip and check the voltage again. If the output of the regulator is O.K. now, just the driver chip is blown. If the output of the regulator is still not approximately 15volts, then both the regulator and driver chip are bad.
    c. If any of the regulator or driver chips are blown then the IGBT PCB assembly (part # 220-1090) must be replaced. The main PCB (# 220-0021) must be repaired, however we recommend replacement since the board cannot be tested under similar load without risking another set of IGBTs.

The IGBT are high speed, high voltage switching devices that control the duty cycle controlling the temperature of the furnace. It is often impossible to explain their demise since the driver circuitry is damaged when they blow. Good grounding is important since the circuit breaker will not trip if all circuits are not properly grounded. Before connecting a new IGBT PCB Assembly to a repaired main PCB, compete the 'Furnace Printed Circuit Board Test' as outlined on the next page.

2.Disconnect 220VAC Furnace Power Cord.

3. To make it easier to work on the furnace, rotate the instrument so you can access the Furnace Control Box from the rear. To accomplish a complete electronics test you will need to access the instrument’s software so rotating the instrument clockwise 90 degrees and supporting the right side with a tall stool or cart is a good way to accomplish this without disconnecting water or gas lines.
4. Remove the 6 screws on the top part of the back cover on the Furnace Control Box and fold the top half down, exposing the Circuit Board. Remove the screw holding the regulator (U222) to the heat sink shield, then remove the 4 screws holding down the shield. Clip the wire tie holding the IGBT wires down to the back cover if applicable. Carefully disconnect the other 3 or 4 plugs across the top of the circuit board, then lift up the top half of the back cover and remove the 6 screws on the bottom half of the back cover to remove the back cover with the circuit board attached.
5. Disconnect the incoming water line from the outside of the IGBT heat sink and the outgoing from the inside. Remove the 4 IGBT mounting screws on its heat sink and remove the IGBT PCB assembly with the sink from the power unit box such that you can access the terminal block.
6. Loosen the screws on the terminal block and remove the wires one by one, making note of their location for re-assembly. Check all connections to the IGBTs for corrosion and make sure there are no stray strands that could short to another terminal. A bad or erroneously wired connection on one of the IGBT terminals can cause them to blow again. Reconnect the transformer and capacitor according to the following wiring diagram:

220-1090 IGBT PCB



5 Green/Yellow Wire, Grounding Screw

4 Black Wire, –170 V from Capacitor

3 Black Wire, Transformer Lead

2 Black Wire, Transformer Lead

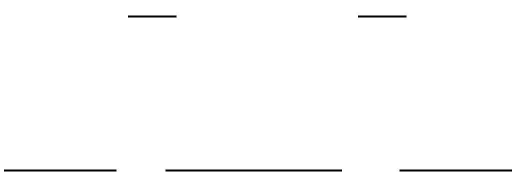
1 Red Wire, +170 V from Capacitor

If your unit has a long ground wire running behind the transformer up to a ground terminal, disconnect it. Use the short ground wire on the new assembly instead and use the included hardware to tie it to the existing hole in the shelf.

7. Feed the control cables through the hole and mount the assembly. Connect the water connections to the heat sink
8. Attach the back panel with the **NEW** PCB attached. Carefully connect the 4 plugs to the new circuit board making sure all pins are inside the receptacles. **DO NOT RECONNECT IGBTS (P8 & P9) AND TRY TO RUN THE FURNACE WITH THE OLD CIRCUIT BOARD OR YOU WILL BURN THEM OUT AGAIN.** If and plugs are misplaced the IGBTs may blow again.
9. Attach the Shield, Heat Sink to the standoffs and secure the Regulator to the shield. Be sure to connect the ground wire to the shield. Close the Furnace box up and test with low power first.

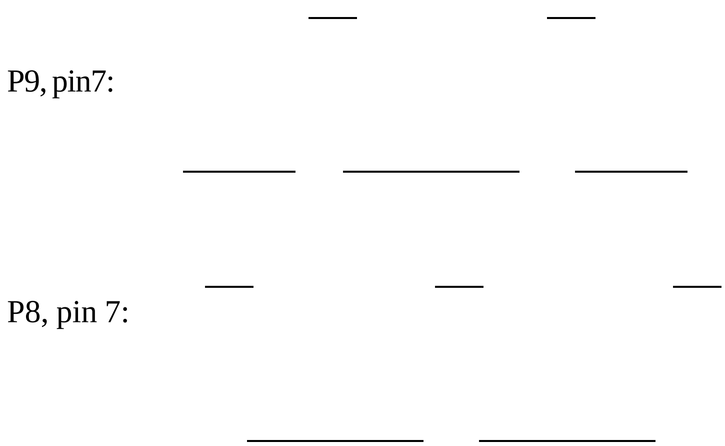
The following test procedure is used to make sure the main PCB is working properly to avoid damage to the IGBTs:

1. Physically inspect the circuit board with a magnifier looking for bad solder connections, short circuits or burnt lands especially if there has been a problem with the power part of the circuitry previously. The IGBTs are sensitive to short circuits and poor contacts even if they are only momentary.
2. The printed circuit board tests **MUST** be performed with the IGBTs disconnected since there is **LETHAL VOLTAGES** on the board with IGBTs connected and the power on as well as the potential to damage the IGBTs while taking measurements on the board. With the power off, make sure the IGBTs (P8 & P9) are disconnected, then power up the furnace.
3. Using an oscilloscope with a trigger lead, connect the trigger to the top (closest to driver chip U217) of 10k resistor R230, and its ground to the bottom of C217. Set the time/div to .1 ms. Connect a 10x probe to pin 1 on the P8 header (right most pin) and its ground to the bottom of C217. Set the volts/div to .5v. You will see the waveform on the scope until you activate the furnace from the 210VGP.
4. On the 210VGP, load a furnace library into one of the lamp positions. Under ‘Controls’ select ‘furnace cleanup cycle’. Set the ‘cleanup time’ for 99sec. Highlight Run Cleanup Cycle’ and press enter. If the furnace responds with ‘Furnace Not Responding’ or repeats the message ‘Furnace Initializing’ for more than one minute, then there is a communication problem. Turn the furnace off, wait a few seconds, then turn it on and try again. If it is still not communicating, check that the furnace communications cable is properly installed, and check that the outputs of regulators U214, U215, and U222 are +12vdc, -12vdc, and 5vdc respectively. You can also check the handshaking signals from the 210 on J1, using the schematic as a guide.
5. When the furnace responds, on the scope you’ll first see the baseline with a line of small dots above it representing the small duty cycle for furnace initialization. Then you should see the still wave form of the burn duty cycle which looks something like this:



If you get this waveform, then this IGBT Driver is operational. Try the same test on P9, pin 1 (leftmost pin), but you’ll have to move the trigger to R231 with ground C220 since the IGBT drivers are completely Isolated. Both P8 pin 1 and P9 pin 1 must have the same waveform to properly fire the IGBTs.

6. The center driver on the PCB drives one gate in each IGBT package. To test, hook up the trigger to R233 and ground to C223 like above. Compare the waveforms on P8, pin 7 and P9, pin 7. The waveforms should be the same as the other driver outputs but the two should be ‘out of phase’ as shown in the diagram below:



Both these Waveforms need to be present and out of phase to ensure the IGBTs are driven properly. Keep in mind that you may need to restart the furnace cleanup cycle on the 210VGP if you need more time to complete the tests. This test does not ensure the IGBTs are 100% safe to connect since the test cannot be made under a proper load. However, if you complete this test, inspect the wiring to ensure it agrees with the wiring diagram after servicing and check for corroded contacts or bad solder connections you can be confident that you should not have problems

7. Press <esc> on the 210 to end the cleanup cycle when you are done. **TURN THE POWER TO THE FURNACE OFF** before connecting the IGBTs to P8 & P9 and closing the back of the furnace.

PROCEDURE FOR INSTALLING SOFTWARE IN THE BUCK 220GF

1.)Make sure the power is shut off and unplug the furnace box. Remove the top seven screws on the back of the 220GF furnace box and let the top half of the back down exposing the circuit board. Remove metal shield covering the circuit board if applicable. DO NOT disconnect any wires and be careful not to damage any of the wiring.

2.) Look for the socket labeled “U110” (this number will be upside down as you are looking on the board). The chip in U110 is a 512K EPROM in a slightly larger socket so that we can expand to a bigger capacity EPROM for future upgrades if necessary. Before removing this chip, note its orientation in the socket. The left side of the chip (the side with no notch) is all the way in the left side of the socket. the right side of the chip(notched) falls two pins short of filling the socket in each row. The notch in the chip is still on the same side as the notch in the socket. The new chip is the same size and is therefore installed in the same manner.

3.) After you’ve examined the position of the chip, you can remove it by carefully prying it up with a flathead screw driver. Please do not discard the chip because it can be reprogrammed for future updates. Please put it in the packaging the new chip came in and return it to us.

4.) Install the chip marked “FU110, V1.20” into the socket marked “U110” toward the left side of the socket as described in step 2. Make sure the notch on the chip is on the same side as the notch on the socket and be careful not to bend any pins.

5.) In order to test this new software, you must have V3.54 software installed in the Buck 210VGP, have a furnace library loaded (Pb Furn3 for example) for a lamp, and have your water and argon gas connected to the furnace. If you’ve never run the Buck 220GF furnace before, read the instruction manual before running.

6.) With the “read key sampling” and “read key printing” options set to “off” under the controls menu, press the “read” button to start the furnace. If ,after a delay , the instrument responds with “furnace not responding”, check to make sure the chip as well as the furnace cable are properly installed. Then press esc twice on the 210VGP, turn the furnace power off, wait a few seconds and turn the furnace back on before trying again. If the furnace responds with a “furnace ready” message, you know the software is working .

7.) Press ESC to stop the furnace program.

SECTION 10.4 : Troubleshooting The 211 Auto-Gas Control System

PROBLEM: BURNER SENSOR LIGHT ON:

1.) The burner head is not installed and screwed down to make contact.
2.) The "blowout plug" on the left side of the block is not installed.
3.) The drain sensor is not filled with water, not plugged in or not making contact.

PROBLEM: GAS PRESSURE LIGHT ON:

1.) There is not 13-15psi Acetylene at the Fuel Inlet.
2.) There is not 50-60psi Air at the Air Inlet.
3.) If you have a long run of tubing between the tank and the instrument, you may need to turn the tank regulator higher to overcome losses.

PROBLEM: WILL NOT SWITCH TO NITROUS OXIDE / N_2O LIGHT ON:

1.) There is not 50-60psi N_2O at the N_2O Inlet.
2.) The N_2O Burner Head and/or interlock is not installed.
3.) You have a long run of tubing between the tank and the instrument and you need to turn the tank regulator higher to overcome losses.
4.) Any of the other sensors are triggered.

PROBLEM: FLAME KEEPS SHUTTING DOWN:

1.) The Burner's vertical adjustment is too high or low for the Flame Sensor to work properly.
2.) There is some other obstruction in between the flame sensor and the burner head.
3.) Tubing for Air or Fuel supply damaged, restricting flow

SECTION 10.5 – Graphite furnace tips/troubleshooting

The windows on either side of the furnace cooling towers should be cleaned regularly (they can be removed to clean). Dirty windows will reduce energy throughput.

When the system is first set up the furnace program should be checked. During the dry step use the mirror to check the sample drying process. The mirror can be held to the right of the right side furnace window and using the hollow cathode lamp as a backlight, the sample can be seen as a shiny surface on the bottom of the tube. It may take a little practice to get the angle of the mirror correct. It's helpful to turn the background corrector off to avoid getting a blurry image. The mirror must be inserted after the message "setting pmt voltage" disappears otherwise an error message will result and the furnace program will not run. The sample should be completely dry before the ash step is reached but should not boil or spatter in the tube. It should look like it slowly dissolves into the bottom of the tube. Boiling, spattering or incomplete drying can produce erratic results or double peaks.

Using the default furnace program should be good enough for most samples however you can fine tune the program for your samples

To optimize sensitivity start with the default program and make 50 degree changes in the atomize step both up and down and note which temperature gives the highest absorbance and good peak shape (change both the ramp & atomize step each time). Try to use as low a temperature as possible to preserve tube life.

When aligning the autosampler the pipette tip should be aligned so that the tip delivers the sample about 1 to 2 mm from the bottom of the tube. The tip should be touching the sample as it pulls out of the tube.

The inside of the contact rings where the tube touches should be kept clean of deposits. This can be done with a q-tip and some methanol, or a small burnishing tool for more stubborn deposits. Poor contact will cause the furnace not to heat even though it runs through the program.

Tube life depending on sample matrix should be about 100-500 cycles. If tube burning is noticed then either the argon is contaminated with air, there is a leak, or there is a severe draft near the instrument (a draft can displace the argon in the furnace area).

If poor precision is noticed using the sampler then pipette by hand and see if it clears up.

If you get inconsistent results from the same solution, the temperatures may be set too low, or you may need some matrix modifier to stabilize the species in the tube.

If your baseline does not return to zero after each run, you are getting a build up of residue in the tube. This should be burned up using the burn out feature, or by doing some blank runs with 10 % nitric acid to oxidize any residual organics or 10% hydrochloric acid to volatilize any metals.
Common elements like Na, K, Mg, Ca, Zn and Cu represent contamination and may show poor reproducibility on graphite furnace. Also since they are very sensitive elements by flame there is not much need to use the furnace.

Here are some solutions for problems you may encounter while running the 220GF Graphite Furnace:

PROBLEM: 220GF WILL NOT TURN ON

1.) If the Circuit Breaker stays on, but the power light is off, check your 208 or 240 VAC power source.
2.) If the circuit breaker “trips” when you turn it on, check to see if there is something shorting the electrodes on the furnace head. If the breaker continues to trip, contact a qualified service engineer to find the short circuit in the high voltage system.

PROBLEM: 220GF WILL NOT RESPOND TO BUCK 210VGP

1.) If the “Furnace Not Installed” message appears at the bottom of the screen, see page 15 “Configuring the Hardware” to initialize the Buck 210VGP for Graphite Furnace use.
2.) If the “Furnace Not Responding” message appears at the bottom of the screen, or if the “Waiting for Furnace Reply” message remains for more than 30 seconds without a response, check to see that the 220GF power is on and that the communications cable is connected.
3.) If the “Furnace Error” message appears, turn the 220GF power off, wait a few seconds then turn it back on. Wait a minute for the 220GF to initialize, then try to start the furnace again.

PROBLEM: FURNACE WILL NOT HEAT UP AS BUCK 210 RUNS THE PROGRAM

1.) If the furnace sounds as if it would when heating up, the tube is not making good contact with the electrode. If the tube is old, replace it and twist the tube around a few times in each electrode before clamping the electrodes together. If necessary, tighten the set screw on the detent a quarter turn to increase tension on the tube.
2.) After a couple of years you may need to replace the electrodes in the furnace head to ensure proper contact. A extra pair of electrodes are included with the instrument.
3.) If the furnace does not make any sound while going through the furnace cycle, a qualified service technician to assess the power circuitry as described in Section 10.3.4.

PROBLEM: SHORT TUBE LIFE

1.) The presence of oxygen in the tube will cause it to deteriorate rapidly. If there is any amount of oxygen in the Argon gas, or if air from the building’s ventilation system is blowing on the furnace the graphite tubes will not last long.
2.) The tube will deteriorate more rapidly at higher temperatures. Since some elements require Atomization temps as high as 2900 degrees, tubes won’t last as long when running these elements. You can, however, reduce the time at this temperature without sacrificing a lot of sensitivity by using 2-4 Atomize steps where the second is 2900 degrees for 1 sec, for example, followed by more short steps at decreasing temperatures.
3.) The chemical composition of your samples can cause the graphite tubes to deteriorate faster. The simplest way to reduce this effect is by making use of the low detection limits of the system as best you can and dilute your samples as much as you can.

Changing Furnace Transformer Taps

There are two transformers in the 220GF Power Unit Box, both with taps for 208 and 240 VAC. A qualified electrical technician can easily change the taps if necessary using the following instructions.

1. Unplug the Power Unit from the 208 or 240 VAC outlet.
2. Remove the 6 screws on the upper half of the power unit box and fold down the top half of the back panel. Be careful not to disturb any of the wiring to the circuit board.
3. The small transformer is located on the bottom left-hand corner of the top compartment. Move the red wire to the appropriate quick disconnect tab.
4. Install the screws back in to the top half of the back panel then remove the six on the bottom.
5. The large transformer provides the large step up in amperage needed to heat the graphite tube. Connect the black transformer wire to the appropriate tab using the 10-32 pan head screw.

SECTION 10.6: Troubleshooting The 220AS Graphite Furnace Autosampler

Motor Errors

While working with the Buck 220AS you may encounter motor errors from time to time. This is when the Autosampler stops and both lights are blinking. Normally this condition may be cleared up with a “hard reset” (performed by pressing and holding the reset button for 5 seconds). This resets the motor position with respect to motor phase. Just pressing and releasing the <reset> button will not clear the error but merely set the mechanisms back to home position. If the motor errors occur repeatedly, you’ll need to assess which mechanism is at fault by watching to see what the last motion was before the error [*arm* movement, tray moving left/right (*translation*), tray *rotation* or *syringe*]. Repeated motor errors may also be caused by inconsistent line voltage. If the line voltage is consistantly higher or lower the normal (120 or 240 VAC), you may change the line voltage selector on the power module using the following procedure:

Changing Line Voltage:

1.) Unplug the power cord,
2.) Remove the fuse holder with a small blade screwdriver
3.) Using needle nose pliers carefully pull out the small circuit board.
4.) Rotate the board and plastic indicator for the best voltage (preferably + or – 7 volts from your actual line voltage) and re-insert the board.
5.) Re-install the fuse holder.

In the following troubleshooting guide, we will address each mechanism (as described above) separately:

PROBLEM : ARM MOTOR ERROR

1.) Make sure the Autosampler is properly aligned (see section 4.2) such that the probe does not come in contact with the cup or the furnace.
2.) Inspect the mechanism by removing the necessary covers to see if the motion is restricted, or if any belts or pulleys are loose. You must not be able to move the arm with respect to it’s pulley and chopper or the set screws must be tightened.

PROBLEM : SYRINGE MOTOR ERROR

1.) Check the coupler between the syringe and motor. Make sure the drive screw is screwed into the coupler as far as it goes and that it’s set screw is tight.
2.) Make sure the syringe plunger is into the coupler all the way and that it’s set screw is secured

PROBLEM : TRANSLATION MOTOR ERROR

1.) Make sure the tray assembly slides smoothly back and forth when the power is turned off.

PROBLEM : ROTATION MOTOR ERROR

1.) Make sure the tray screw is tight, securing the tray

Here are some other situations you may encounter with simple solutions:

PROBLEM: NO SENSITIVITY, NO INJECTION

1.) Autosampler is not aligned properly to pick up sample in the cup.
2.) The rinse water bottle is empty. You will have to press <RESET> several times to purge the system after re-filling the bottle.

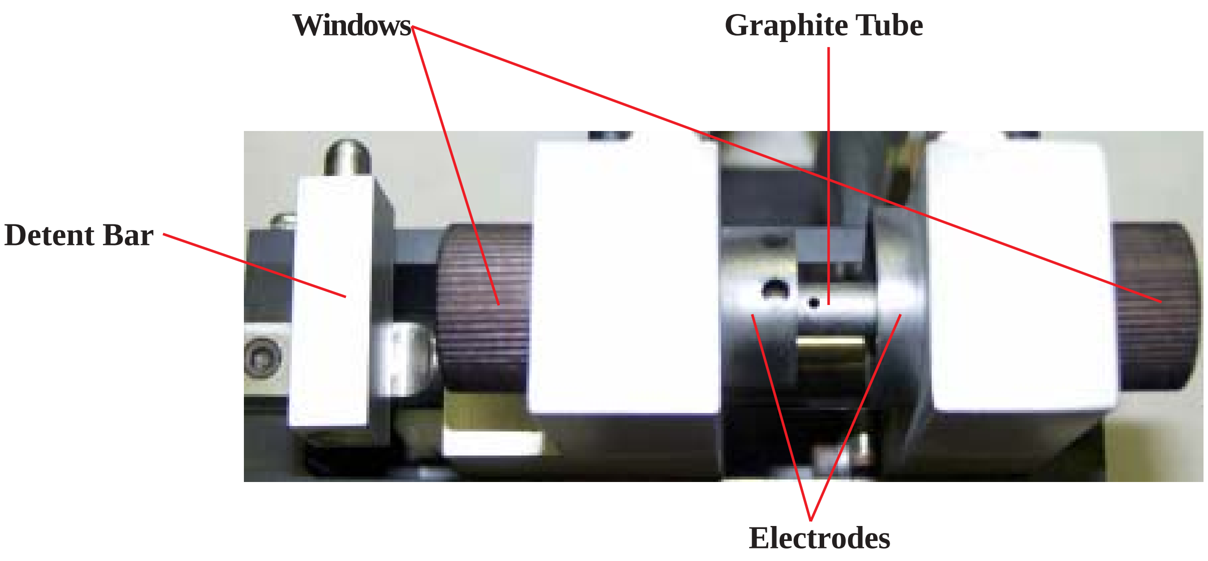
PROBLEM: SIPPER PROBE TIP DAMAGED

The teflon sipper tube can be pulled through the probe, then cut off at a 45 degree angle with about 3/8 inch (9.5mm) of teflon tube protruding from the end.

SECTION 11.1 : Maintenance Buck 210VGP

1) Twice a year the o-rings should be checked in the burner system. Remove the 3 cap nut screws holding on the burner head then remove. Check the integrity of the o-ring underneath and replace if necessary. Coating the o-ring with a thin layer of teflon grease is a good idea.
2) Remove the screw on the front of the drain block underneath the red acetylene line and pull the drain block out. Pry out the blow out plug on the left side with a screwdriver and check that o-ring.
3) If you see an irregular shaped flame use the cleaning tool provided to clean the burner slot.
4) The entire burner assy can be put in an ultrasonic tank for a thorough cleaning.
5) Keep the lenses on either side of the burner compartment clean for maximum energy.
6) The instrument does not need to be sent in for calibration. If your standards read back correctly during the analysis then it is functioning ok.

SECTION 11.2 : Maintenance Buck 220GF

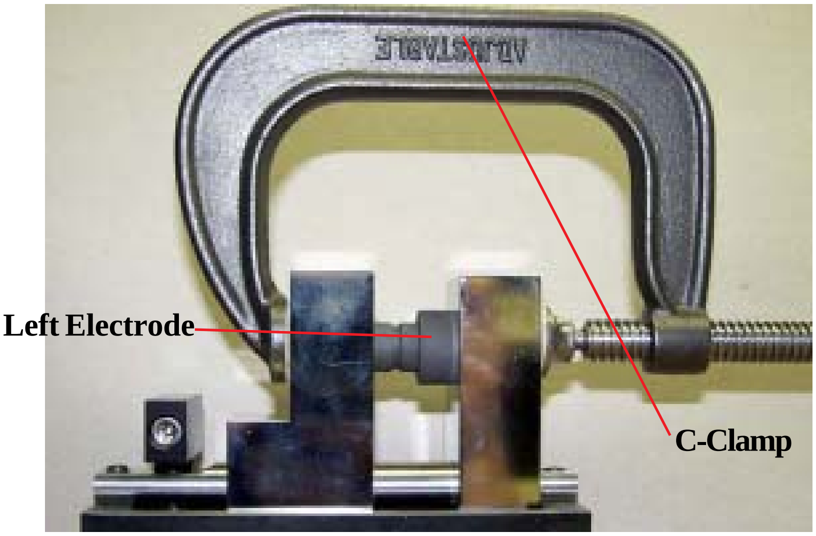


GRAPHITE TUBE REPLACEMENT

The Graphite Tubes are a consumable part of the Graphite Furnace. They last between 100-500 furnace cycles in normal operation depending on the furnace program and the sample matrix. To change the tubes, Twist the electrode detent to release tension on the tube. Slide the left electrode away from the right and remove the old tube left side first using tweezers. Be sure to remove all remnants of the old tube. When installing the new tube, it is best to twist it into the ends of the electrodes to ensure good contact. Twist the detent back into place.

CLEANING WINDOWS

If you experience a loss of lamp energy or excess background absorbance, the quartz windows may be dirty. You can remove the windows by twisting and turning the housing to remove it from the furnace. Then clean the windows with a cotton swab wet with alcohol.



CHANGING ELECTRODES

After a year or two of regular use, the electrodes may wear to the point where the furnace not longer heats up reliably. A spare set of electrodes is included with the Buck 220GF Graphite Furnace System. Here aresome tips for changing electrodes:

1) Remove the Quartz windows
2) Remove the Graphite Tube.
3) The old electrodes may be remove using a piece of ½ inch wooden dowel or equivalent to knock the electrode out with a hammer.
4) Install the left electrode with the injection holes first. Make sure one hole is pointing straight up and the other back towards the tubing side. You can use a 4 inch “C-clamp” to easily press the electrode into the cooling block. Slide the moving block up to the electrode (you must press the electrodes in one at a time) and apply pressure to the outside of the blocks with the C-clamp (you may wish to use a piece of cardboard to protect the furnace head).
5) After the left electrode is installed, you can install the more fragile right electrode in the same manner. If you do not have a C-clamp available, you can turn the electrodes down with some abrasive pad or very-fine sand paper so you can press the electrodes in by hand. Make sure you do not turn them down too much since they should fit tightly to ensure good contact.

SECTION 12: REPLACEMENT PART LIST

BURNER SECTION

PART #	**DESCRIPTION**
999-2202	Burner head slot cleaner
990-1536	“Burner head O-ring [2]
990-1333	Burner head retaining nuts [3]
990-1531	O-ring for X-Y Table [2]
210-5087	Spray Chamber only
210-6070	“Spray Chamber Only, 211”
210-0552	Spray chamber [complete] - except burner head
210-0542	“Burner head Assembly [air/acetylene], 210”
210-0541	“Burner head Assembly [nitrous oxide], 210”
210-0632	“Burner head Assembly [air/acetylene], 211”
210-0631	“Burner head Assembly [nitrous oxide], 211”
210-0538	“Collar, Burner head”
210-0618	“Collar, Burner head, 211”
200-0088	Impact bead
999-1549	Spray chamber drain elbow
200-1090	Blow out plug + O-ring
999-3083	O-ring for blow out plug [2]
200-0056	Nebulizer - complete [stainless steel]
200-0080	Nebulizer - needle assy only
210-0551	Nebulizer - body only
200-0060	Nebulizer - complete [corrosion resistant]
210-0554	X-Y Table Assembly
210-0606	Chimeny Assembly
210-6007	Sample Tray
210-0051	“Glass shield, burner compartment, for old style 210”
BS186-0450	0.5 Absorbance screen
BS30040	“Spares kit - includes nebulizer, impact bead,” “ blow out plug & O-ring, capillary tubing [10 feet],” “ nebulizer cleaning wire [10 pieces], “ “ burner slot cleaner, retaining nuts [3]”

ELECTRONICS SECTION

210-0540	Front panel complete and tested
210-1006	Computer style main power supply
210-2006	Power control assembly (includes chassis)
210-2000	System pcb

PART #	**DESCRIPTION**
210-2002	Keyboard
990-1072	Power Cord
998-1038	Fuses, 100mA for HCL [3]

998-1039	Fuses, 200mA for D2 [3]
210-0616	Software with library chip
210-0617	Software with library chip for 210 with Furnace
210-0553	Keycap Set (complete)
998-1046	Liquid crystal display
998-1026	Photomultiplier -R928
210-0550	Lamp socket end piece
200-1120	Lamp socket cap

PNEUMATICS

999-1532	Fuel toggle switch
999-1534	Fuel adjustment valve
999-1555	Regulator [internal air]
210-1086	Solenoid [air/nitrous oxide]
210-0607	Flowmeter [new]-for 210 sn 606 or greater

OPTICAL

210-1052	D2 lamp
200-1053	Grating
200-1052	Monochromator mirror
210-5026	Lenses
210-1116	“Counter, Wavelength”
999-2032	Micrometer for wavelength
999-3419	Monocover strap

TOOLS

999-3127	Hex wrench 3/16 for burner head
999-3123	Hex wrench 5/32 for burner block
999-1635	“Hex Driver, 1/4 for Furnace Power Cable”
999-3124	1/16 Allen Wrench

GRAPHITE FURNACE SUPPLIES

220-0021	Furnace PCB
220-1090	IGBT PCB Assembly
BS290-1820	Non-pyro coated graphite tubes [10 pack]
BS007-0699	Non-pyro coated graphite tubes [50 pack]
BS013-5653	Pyro coated graphite tubes [10 pack]
BS009-1504	Pyro coated graphite tubes [50 pack]
BS220-2697	Platform graphite tubes [10 pack]
204-1044	Left Contact Ring
204-1045	Right Contact Ring
990-1537	O-ring for water connections [2]
990-1532	O-ring for windows [2]
220-0014	Furnace Communucations Cable
991-2004	Variable Pipettor 10-100uL
220-0015	“Cooling Water install kit (ball valve, needle valve ,tube) “

FURNACE AUTOSAMPLER

PART #	DESCRIPTION
BS008-7056	Polyethylene sample cups for furnace autosampler
BS011-9079	Polystyrene sample cups for furnace autosampler
220-3056	Polystyrene 8.5mL Sample Cups (pkg 50)
990-1636	3/32 Allen Wrench
990-1072	Power Cord
220-0339	Communication cable
220-0340	Adjustable Feet (2)
220-0341	Mounting Hadware Kit
220-0342	Sipper probe
220-3117	4' of rinse tubing
220-3118	Stiff tube for rinse bottle
220-3089	Rinse bottle
220-3076	Inspection mirror
F	4.0% Phosphate (as ammonium)
G	1.0% Magnesium nitrate
H	2.0% Nickel nitrate
K	0.2% Palladium (as nitrate)

ACCESSORIES

BS303-0106	Acetylene Pressure Regulator
BS303-0263	Air Pressure Regulator
BS303-0264	Argon Pressure Regulator
BS303-0265	Hydrogen Pressure Regulator
BS303-0204	Nitrous Oxide Pressure Regulator
R06-221	Pressure Regulator with Gauge
BS303-0407	“Hood and Vent Kit, Stainless Steel “
BS303-0417	“Hood and Vent Kit, Stainless Steel (220V)”
BS303-0313	Oil-less Air Compressor
BS303-0314	Oil-less Air Compressor (220V)
BS303-0229	Air Filter
BS057-2471	Replacement Cartridge for Filter
6103A	Flash Arrestor
BS30055	Atomic Absorption Spectrometry by S.J. Haswell
999-1514	“Fitting, 1/4 Tube to 1/4 NPT Male”
999-1594	“Fitting, 1/4 Tube to 1/4 NPT Female”
990-1855	Black Flexible Tubing (per foot) with nuts & Ferrules
990-1857	Blue Flexible Tubing (per foot) with nuts & Ferrules
990-1856	Red Flexible Tubing (per foot) with nuts & Ferrules
BS990-8265	Nebulizer Capillary Tubing
BS303-0135	Nebulizer Cleaning Wire
991-1047	Flame Ignitor

SECTION 13: Standard Conditions

Quick Overview

Table 1 serves as a quick reference guide to the sensitivity and performance using flame techniques. The detection limits are determined as the lowest concentration given an absorbance detectable above the noise range. These values were determined empirically under Buck Scientific standard test conditions (see Appendix A).

Sensitivity is a measure of the instrument response to the analyte, and by convention, shows the concentration of each element required to absorb 1% of the incident light energy. This corresponds to an absorbance value of 0.0044. Elements with greater sensitivity will have the lowest concentration values in that category. The values for “sens. check” in table 1 are the amounts in mg/l required to give an absorbance reading of 0.200 abs.

The “linear range” is the amount of analyte in mg/l which will produce an absorbance of approximately 0.300 and safely keep the analysis in the linear part of the calibration curve. This area of the curve requires only one standard to be run but an additional standard run as a check is good practice. Above this area a multi-point calibration must be used.

Table 2 lists alternate wavelengths you can use in order to increase the linear range of your analysis or to reduce interferences from other elements. RS stands for “relative sensitivity”. This describes how sensitive this wavelength is compared to the primary wl which will always have a relative sensitivity of 1.0. For example, if a secondary line has an RS of 2 it will give you an absorbance 1/2 of the primary wavelength.

TABLE 1: Flame Atomic Absorption Concentration Ranges

TABLE 1: Flame Atomic Absorption Concentration Ranges

Metal	Wl (Nm)	Slit (Nm)	Detec Limit (mg/L)	Sens Check (mg/L)	Linear Range (mg/L)	Flame Type Color
Aluminum (Al)	309.3	0.7	2.00	25	50.00	N-A, rich/red
Aluminum (Al)	394.4	0.7	5.0	55	100.00	N-A, rich/red
Antimony (Sb)	217.6	0.2	0.30	11.5	20.00	A-A, lean/blue
Arsenic (As)	193.7	0.7	0.25	22.5	25.00	A-A, lean/blue
Barium (Ba)	553.6	0.7	0.50	10	25.00	N-A, rich/red
Beryllium (Be)	234.9	0.7	0.04	0.75	4.00	N-A, rich/red
Bismuth (Bi)	222.8	0.7	0.10	10	25.00	A-A, lean/blue
Cadmium (Cd)	228.9	0.7	0.01	0.75	2.00	A-A, lean/blue
Calcium (Ca)	422.7	0.7	0.01	1	3.00	N-A, rich/red
Calcium (Ca)	422.7	0.7	0.05	3.5	6.00	A-A rich/yellow
Cesium (Cs)	852.1	0.2	—	5	7.50	A-A, lean/blue
Chromium (Cr)	357.9	0.7	0.04	2	5.00	A-A, rich/yellow
Chromium (Cr)	357.9	0.7	0.04	5	10.00	N-A, rich/yellow
Chromium (Cr)	425.5	0.7	0.04	10	25.00	A-A, rich/yellow
Chromium (Cr)	425.5	0.7	0.04	25	50.00	N-A, rich/yellow
Cobalt (Co)	240.7	0.2	0.05	3.5	5.00	A-A, lean/blue
Cobalt (Co)	352.7	0.2	0.5	45	75.00	A-A, lean/blue
Copper (Cu)	324.8	0.7	0.005	2	5.00	A-A, lean/blue
Copper (Cu)	327.4	0.7	0.010	4	10.00	A-A, lean/blue
Dysprosium (Dy)	421.2	0.2	—	22.5	33.75	N-A, rich/red
Erbium (Er)	400.8	0.2	—	15	22.50	N-A, rich/red
Europium (Eu)	459.4	0.2	—	15	22.50	N-A, rich/red
Gadolinium (Gd)	368.4	0.2	—	425	637.5	N-A, rich/red
Gallium (Ga)	287.4	0.7	—	30	45.00	A-A, lean/blue
Germanium (Ge)	265.1	0.2	—	50	75.00	N-A, rich/red
Gold (Au)	242.8	0.7	0.20	5	10.00	A-A, lean/blue
Gold (Au)	267.6	0.7	0.40	8.5	15.00	A-A, lean/blue

TABLE 1: *Continued*

Metal	Wl (Nm)	Slit (Nm)	Detec Limit (mg/L)	Sens Check (mg/L)	Linear Range (mg/L)	Flame Type Color
Hafnium (Hf)	286.6	0.2	—	225	337.5	N-A, rich/red
Holmium (Ho)	410.4	0.2	—	20.0	30.00	N-A, rich/red
Indium (In)	303.9	0.7	—	17.5	26.25	A-A, lean/blue
Iridium (Ir)	264.0	0.2	—	250	375	A-A, rich/yellow
Iron (Fe)	248.3	0.2	0.05	2.5	5.00	A-A, lean/blue
Iron (Fe)	372.0	0.2	0.50	25	50.00	A-A, lean/blue
Lanthanum (La)	550.1	0.2	—	1250	1875	N-A, rich/red
Lead (Pb)	217.0	0.7	0.04	5	10.00	A-A, lean/blue
Lead (Pb)	283.3	0.7	0.08	10	20.00	A-A, lean/blue
Lithium (Li)	670.8	0.7	—	1	1.50	A-A, lean/blue
Lutetium (Lu)	336.0	0.2	—	125	187.5	N-A, rich/red
Magnesium (Mg)	285.2	0.7	0.005	0.75	1.50	A-A, lean/blue
Magnesium (Mg)	202.6	0.7	0.25	40	80.0	A-A, lean/blue
Manganese (Mn)	279.5	0.7	0.03	1.25	2.50	A-A, lean/blue
Mercury (Hg)	253.7	0.7	~ 5.0			A-A, lean/blue
Molybdenum (Mo)	313.3	0.7	0.80	15	20.00	N-A, rich/red
Neodymium (Nd)	492.4	0.2	—	175	262.5	N-A, rich/red
Nickel (Ni)	232.0	0.2	0.05	3.5	4.00	A-A, lean/blue
Nickel (Ni)	341.5	0.2	0.10	7.0	8.00	A-A, lean/blue
Nickel (Ni)	339.1	0.2	0.50	40	45.00	A-A, lean/blue
Niobium (Nb)	334.4	0.2	—	350	525	N-A, rich/red
Osmium (Os)	290.9	0.2	—	22.5	33.75	N-A, rich/red
Palladium (Pd)	244.8	0.2	0.15	5	10.00	A-A, lean/blue
Platinum (Pt)	265.9	0.2	0.80	50	20.00	A-A, lean/blue
Platinum (Pt)	262.8	0.2	2.40	150	100.00	A-A, lean/blue
Potassium (K)	766.5	0.7	0.01	0.75	3.00	A-A, lean/blue
Potassium (K)	769.9	0.7	0.025	2	5.00	A-A, lean/blue
Potassium (K)	404.4	0.7	2.50	200	300	A-A, lean/blue
Rhenium (Re)	346.0	0.2	—	325	487.5	N-A, rich/red
Rhodium (Rh)	343.5	0.2	—	4.5	6.75	A-A, lean/blue
Rubidium (Rb)	780.0	0.7	—	25	37.5	A-A, lean/blue
Ruthenium (Ru)	349.9	0.2	—	15	22.5	A-A, lean/blue
Scandium (Sc)	391.2	0.2	—	7.5	11.25	N-A, rich/red
Selenium (Se)	196.0	0.2	0.50	15	25.00	Ar-H
Silicon (Si)	251.6	0.2	1.00	50	50.00	N-A, rich/red
Silver (Ag)	328.1	0.7	0.02	1.25	3.00	A-A, lean/blue
Silver (Ag)	338.3	0.7	0.05	3.25	3.50	A-A, lean/blue
Sodium (Na)	589.0	0.2	0.005	0.25	2.00	A-A, lean/blue
Sodium (Na)	330.3	0.2	1.2	60.00	100.00	A-A, lean/blue
Strontium (Sr)	460.7	0.2	—	2.5	3.75	N-A, rich/red
Tantalum (Ta)	271.5	0.2	—	275	412.5	N-A, rich/red
Technetium (Tc)	261.4	0.2	—	50	75	A-A, rich yellow
Tellurium (Te)	214.3	0.7	—	10	15	A-A, lean/blue
Terbium (Tb)	432.6	0.2	—	150	225	N-A, rich/red
Thallium (Tl)	276.8	0.7	0.40	15	25.00	A-A, lean/blue
Thulium (Tm)	371.8	0.2	—	10	15	N-A, rich/red
Tin (Sn)	286.3	0.7	1.10	90	120.0	A-A, rich/yel
Tin (Sn)	286.3	0.7	1.00	70	100.0	N-A, rich/red
Tin (Sn)	224.6	0.7	0.5	35	50.00	A-A, rich/yel
Tin (Sn)	224.6	0.7	0.5	37	50.00	N-A, rich/red
Titanium (Ti)	364.3	0.2	1.00	40	25.00	N-A, rich/red
Tungsten (W)	400.9	0.2	0.5	225	337.5	N-A, rich/red

TABLE 1: *Continued*

Metal	Wl (Nm)	Slit (Nm)	Detec Limit (mg/L)	Sens Check (mg/L)	Linear Range (mg/L)	Flame Type Color
Uranium (U)	351.5	0.2	—	2750	4125	N-A, rich/red
Vanadium (V)	318.4	0.2	0.40	45	75.00	N-A, rich/red
Ytterbium (Yb)	398.8	0.2	—	2.5	3.75	N-A, rich/red
Yttrium (Y)	410.2	0.2	—	40	60	N-A, rich/red
Zinc (Zn)	213.9	0.7	0.005	0.50	2.50	A-A, lean/blue
Zirconium (Zr)	360.1	0.2	—	150	225	N-A, rich/red

NOTE: The notations refer to preferred technique where FAAS is generally not suitable:
hg means hydride technique; *cv* means cold vapor technique

TABLE 2: Alternate Wavelengths

	Wl	Slit	Rs		Wl	Slit	Rs
Aluminum	396.2	0.7	1.1	Gadolinium	407.9	0.2	1.0
	308.2	0.7	1.6		378.3	0.2	1.1
Antimony	206.8	0.2	1.5		405.8	0.2	1.2
	231.2	0.2	2.1		405.4	0.2	1.3
Arsenic	189.0	0.7	0.8		371.4	0.2	1.7
	197.2	0.7	2.0		419.1	0.2	2.7
Barium	350.1	0.2	16.0		367.4	0.2	2.9
Beryllium	none				404.5	0.2	3.2
Bismuth	222.8	0.2	2.4		394.6	0.2	6.5
	306.8	0.7	3.7	Gallium	294.4	0.7	1.0
	206.2	0.2	8.6		417.2	0.7	1.4
	227.7	0.2	14.0		250.0	0.7	9.0
Boron	none				245.0	0.7	9.6
Cadmium	326.1	0.7	435		272.0	0.7	20
Calcium	239.9	0.7	120	Germanium	259.2	0.2	2.2
Cesium	455.5	2.0	85		271.0	0.2	2.4
Chromium	359.4	0.7	1.7		275.5	0.2	2.6
	360.5	0.7	2.2		269.1	0.2	3.8
	425.4	0.7	3.0	Gold	267.6	0.7	1.8
	427.5	0.7	3.8		312.3	0.7	900
	429.0	0.7	4.5	Hafnium	307.3	0.2	
Cobalt	242.5	0.2	1.2		289.8	0.2	
	241.2	0.2	1.8		296.5	0.2	
	252.1	0.2	2.0	Holmium	405.4	0.2	1.3
	243.6	0.2	2.9		416.3	0.2	1.7
	304.4	0.2	12		417.3	0.2	4.2
	352.7	0.2	22		404.1	0.2	5.2
	346.6	0.2	30		410.9	0.2	9.8
Copper	327.4	0.7	2.0		412.7	0.2	11
	216.5	0.2	6.0		422.7	0.2	24
	222.6	0.2	15	Indium	325.6	0.2	1.0
	249.2	0.7	72		410.5	0.7	2.9
	224.4	0.2	157		451.1	0.7	3.1
Dysprosium	404.6	0.2	1.1		256.0	0.7	12
	418.7	0.2	1.2	Iridium	208.9	0.2	0.3
	419.5	0.2	1.6		266.5	0.2	1.2
	416.8	0.2	6.8		237.3	0.2	1.3
Erbium	386.3	0.2	2.7		285.0	0.2	1.4
	415.1	0.2	2.7		250.3	0.2	1.7
	389.3	0.2	5.0		254.4	0.2	2.1
	408.8	0.2	7.0		351.4	0.2	8.6

TABLE 2: *Continued*

	Wl	Slit	Rs
	381.0	0.2	8.4
	390.5	0.2	20
Europium	462.7	0.2	1.3
	466.2	0.2	1.5
	321.1	0.2	12
	321.3	0.2	15
	311.1	0.2	15
Lanthanum	418.7	0.2	1.6
	495.0	0.2	1.7
	357.4	0.2	4.0
	365.0	0.2	4.0
	392.8	0.2	4.0
Lead	217.0	0.7	0.4
	261.4	0.7	10
	368.4	0.7	25
Lithium	323.3	0.7	235
Lutetium	331.2	0.2	1.8
	337.7	0.2	2.0
	356.8	0.2	2.1
	298.9	0.2	9.2
	451.9	0.2	11
Magnesium	202.6	0.7	24
Manganese	279.8	0.2	1.3
	280.1	0.2	1.9
	403.1	0.2	9.5
Molybdenum	317.0	0.7	1.6
	379.8	0.7	1.8
	319.4	0.7	2.0
	386.4	0.7	2.5
	390.3	0.7	3.3
	315.8	0.7	4.0
	320.9	0.2	8.7
Neodymium	?		
	?		
	?		
Nickel	231.1	0.2	1.5
	352.5	0.2	3.3
	341.5	0.2	3.5
	305.1	0.2	4.5
	346.2	0.2	6.6
Niobium	358.0	0.2	1.1
	334.9	0.2	1.2
	408.0	0.2	1.4
	335.8	0.2	1.5
	412.4	0.2	1.9
	357.6	0.2	2.5
Osmium	305.9	0.2	1.6
	263.7	0.2	1.8
	301.8	0.2	3.2
	330.2	0.2	3.6
Palladium	247.6	0.2	1.0
	276.3	0.2	2.7
	340.5	0.2	3.0
Phosphorus	214.9	0.2	2.0

	Wl	Slit	Rs
Iron	248.8	0.2	1.7
	302.1	0.2	3.7
	252.7	0.2	4.6
	372.0	0.2	5.7
	373.7	0.2	10
Platinum	306.5	0.7	2.1
	283.0	0.2	3.4
	293.0	0.7	3.7
	273.4	0.2	4.1
Potassium	769.9	0.7	2.3
Praseodymium	513.3	0.2	1.4
	473.7	0.2	2.2
	492.5	0.2	2.2
	502.7	0.2	2.5
Rhenium	346.5	0.2	1.7
	345.2	0.2	2.4
Rhodium	369.2	0.2	1.7
	339.7	0.2	2.5
	350.2	0.2	3.7
	365.8	0.2	6.0
Rubidium	794.8	2.0	2.1
	420.2	0.7	120
Ruthenium	372.8	0.2	1.4
	379.9	0.2	2.2
	392.6	0.2	11
Samarium	476.0	0.2	1.4
	511.7	0.2	1.4
	520.1	0.2	1.6
	472.8	0.2	2.0
Scandium	390.8	0.2	1.0
	402.4	0.2	1.4
	402.0	0.2	1.8
	405.5	0.2	2.7
	327.0	0.2	3.2
	408.2	0.2	7.0
	327.4	0.2	12
Selenium	204.0	0.2	3.0
	206.3	0.2	11
	207.5	0.2	35
Silicon	250.7	0.7	2.8
	252.8	0.2	3.2
	252.4	0.2	3.7
	221.7	0.2	4.3
	221.1	0.2	8.0
Silver	338.3	0.7	1.9
Sodium	589.6	0.2	1.0
	330.2	2.0	185
Strontium	none		

TABLE 2: ***Continued***

	Wl	Slit	Rs		Wl	Slit	Rs
Tantalum	260.8	0.2	2.1	Tungsten	255.1	0.2	0.5
	265.7	0.2	2.5		294.4	0.2	0.7
	293.4	0.2	2.5		268.1	0.2	0.7
	255.9	0.2	2.5		272.4	0.2	0.7
	265.3	0.2	2.7		294.7	0.7	0.7
	269.8	0.2	2.7		283.1	0.2	1.0
	275.8	0.2	3.1		289.6	0.2	1.4
Technetium	260.9	0.2	4.1		287.9	0.2	2.4
	429.7	0.2	6.5		430.2	0.2	7.2
	426.2	0.2	8.1	Uranium	358.5	0.2	0.3
	318.2	0.2	10		356.7	0.2	0.5
	423.8	0.2	11	Vanadium	306.6	0.2	2.4
	363.6	0.2	11		306.0	0.2	2.4
	317.3	0.2	100		305.6	0.2	3.0
Tellurium	225.9	0.7	15		320.2	0.2	6.4
	238.6	0.7	50		390.2	0.2	6.5
Terbium	431.9	0.2	1.2	Ytterbium	346.4	0.2	3.5
	390.1	0.2	1.6		246.4	0.2	7.5
	406.2	0.2	1.8		267.2	0.2	40
	433.8	0.2	2.0	Yttrium	407.7	0.2	1.1
	410.5	0.2	3.6		412.8	0.2	1.2
Thallium	377.6	0.7	2.7		414.3	0.2	1.4
	238.0	0.2	6.7		362.1	0.2	2.0
	258.0	0.2	24	Zinc	307.6	0.7	4700
Thulium	410.6	0.2	1.4	Zirconium	354.8	0.2	1.5
	374.4	0.2	1.6		303.0	0.2	1.5
	409.4	0.2	1.7		301.2	0.2	1.7
	418.8	0.2	1.9		298.5	0.2	1.7
	420.4	0.2	3.0		362.4	0.2	1.9
	375.2	0.2	5.7				
	436.0	0.	9.3				
	341.0	0.2	14				
Tin	224.6	0.2	0.5	Titanium	365.4	0.2	1.0
	235.5	0.7	0.8		320.0	0.2	1.2
	270.6	0.7	2.0		363.6	0.2	1.2
	303.4	0.2	2.8		335.5	0.2	1.4
	254.7	0.7	4.4		375.3	0.2	1.6
	219.9	0.2	4.7		334.2	1.2	1.6
	300.9	0.7	5.9		399.9	0.2	1.6

AA3001:
Sample Preparation of Glass and Ceramic Materials for Atomic Absorption Analysis

The analysis of silicate-based glass and ceramics has always been a moderately difficult job, since the components that comprise the glass are usually not stable together in solution. For example, most typical acidic digestions that solubilize silicon will cause calcium fluoride to precipitate. If no fluoride is present, the silicon will not be fully decomposed, and if the solution is heated, boron can be lost, etc.

There are several industry standard procedures that are used for specific elements, thereby requiring at least two (2) preparations to be used for the complete, reproducible analysis of each sample. If only certain components are important, then perhaps a single preparation can be employed. Listed below are some methods developed at Buck Scientific for use on the Model 210VGP Atomic Absorption Spectrophotometers.

(1) Fuse 0.2 – 1.0 grams of powdered glass with 5x the amount of a mixed lithium carbonate/zinc oxide flux (2:1) for 10-15 minutes at 900 – 950°C in an Ni or Pt crucible. Cool and dissolve the melt in hydrochloric acid (1:1) with warming. The resultant solution is quantitatively diluted, filtered to remove insoluble silica, and analyzed for Ca, Mg, Al, B, Na, K, Fe, Cu, and Mn. If Cr, V, Ti or Mo are to be determined, the flux should contain ~1/2 part sodium peroxide, or the “melt” can be dissolved in 25% hydrochloric acid and 10% hydrogen peroxide with satisfactory results.

(2) Fuse 0.2 – 1.0 grams of powdered glass with ~7x the amount of either sodium hydroxide or potassium hydroxide for 10 – 30 minutes at 400 – 500°C in an Ni or Pt crucible. The melt is cooled, dissolved in water, and promptly analyzed for Si, B, Mo, Ti, V, Zn, halogens, P and Al.

(3) Moisten 0.1 – 0.5 grams of powdered glass with 1ml of alcohol in a Pt or PtFe dish, add ~5ml water, 1ml perchloric acid, 1ml sulfuric acid, and ~10ml hydrofluoric acid. Digest on a steam bath or hot plate until fumes are evolved. Add 1ml sulfuric acid, 10ml hydrofluoric acid, and 2–10ml methyl alcohol. Take to near dryness (sulfuric acid minimizes undesirable reactions from perchloric acid). Add 5–20ml of hydrochloric acid, dilute, and analyze. This procedure allows the accurate determination of almost all elements except Si and B. Adding 0.1 – 0.2% lanthanum or strontium will improve the response for Al, Ca and Mg.

The Buck 210VGP series can accurately determine these major and minor elements in ranges from 0.005% to over 20%, depending on the dilution used, with typical precision of 1.5% or better if using bracketed standards. The entire procedure, from preparation of sample to final analysis, can be standardized to a simple format so that a novice lab technician can perform the test easily.

Analyst: Gerald J. De Menna

SIC: 321, 322, 3252, 326

Analytical Results for Standard Glass Samples

Samples: Standard Reference Materials from NIST / NBS, ASTM and BCS
Preparation: Method #2 for Si, Al, Zn & B; #3 for the others
Calibration: Buck Certified Atomic Absorption Standards
Instrument: The Buck 210VGP Atomic Absorption Spectrophotometer
Conditions: Air / Acetylene flame, N_2O / Acetylene flame, Integrate mode, Normal parameters
[Potassium (K) and Sodium (Na) are done in **emission** mode].

NOTE: All values listed below are in **Weight Percent** (%wt) in the original glass sample; [**K**] = known assay, [**M**] = measured data.

	Soda-Lime (green)		**Borosilicate** (hard, Hi-B)		**Lead "Crystal"** (tinted)	
Element	**[K]**	**[M]**	**[K]**	**[M]**	**[K]**	**[M]**
[#2 prep]						
Si	34.2	34.0	37.8	37.2	30.6	30.9
Al	0.17	0.15	1.21	1.24	0.09	0.09*
Zn	0.18	0.19	.011	.009	1.11	1.09#
B	1.42	1.38	3.93	3.90	0.05	0.04
[#3 prep]						
Na	10.0	10.3	2.97	3.08	4.22	4.25
K	0.49	0.48	.012	.011	6.97	7.03
Zn	0.16	0.18	.006	.007	1.07	1.04#
Ca	6.15	5.97	.007	.009	0.28	0.30
Mg	2.35	2.40	.008	.008	0.05	0.04
Mn	.015	.016	.004	.005	0.11	0.12
Ba	0.17	0.15	4.77	4.61	1.38	1.42
Al	0.15	0.14	1.22	1.20	0.08	0.07*
Pb	<0.01	<0.01	<0.01	<0.01	16.3	16.1
Cu	0.08	0.09	.002	.002	0.16	0.15
Fe	4.08	3.95	.024	.022	0.41	0.42

The above data shows the powerful flexibility and stability of the Buck 210VGP system for the wide-ranging requirements of the glass and ceramics industry. The excellent correlation between the known and measured values ranges from 0.6% to 3.0% (average RSD = 1.6%) demonstrates the precision of the instrument. The fact that two different preparations gave superb reproducibility on two different elements, Zn (#) and Al (*), reveals the high accuracy of the chemistry.

Analytical Results for Standard Glass Samples

Samples:	Standard Reference Materials from NIST / NBS, ASTM and BCS
Preparation:	Method #2 for Si, Al, Zn & B; #3 for the others
Calibration:	Buck Certified Atomic Absorption Standards
Instrument:	The Buck 210VGP Atomic Absorption Spectrophotometer
Conditions:	Air / Acetylene flame, N_2O / Acetylene flame, Integrate mode, Normal parameters [Potassium (K) and Sodium (Na) are done in **emission** mode].

NOTE: All values listed below are in **Weight Percent** (%wt) in the original glass sample; [**K**] = known assay, [**M**] = measured data.

	Soda-Lime (green)		**Borosilicate** (hard, Hi-B)		**Lead "Crystal"** (tinted)	
Element	**[K]**	**[M]**	**[K]**	**[M]**	**[K]**	**[M]**
[#2 prep]						
Si	34.2	34.0	37.8	37.2	30.6	30.9
Al	0.17	0.15	1.21	1.24	0.09	0.09*
Zn	0.18	0.19	.011	.009	1.11	1.09#
B	1.42	1.38	3.93	3.90	0.05	0.04
[#3 prep]						
Na	10.0	10.3	2.97	3.08	4.22	4.25
K	0.49	0.48	.012	.011	6.97	7.03
Zn	0.16	0.18	.006	.007	1.07	1.04#
Ca	6.15	5.97	.007	.009	0.28	0.30
Mg	2.35	2.40	.008	.008	0.05	0.04
Mn	.015	.016	.004	.005	0.11	0.12
Ba	0.17	0.15	4.77	4.61	1.38	1.4
Al	0.15	0.14	1.22	1.20	0.08	0.07*
Pb	<0.01	<0.01	<0.01	<0.01	16.3	16.1
Cu	0.08	0.09	.002	.002	0.16	0.15
Fe	4.08	3.95	.024	.022	0.41	0.42

The above data shows the powerful flexibility and stability of the Buck 210VGP system for the wide-ranging requirements of the glass and ceramics industry. The excellent correlation between the known and measured values ranges from 0.6% to 3.0% (average RSD = 1.6%) demonstrates the precision of the instrument. The fact that two different preparations gave superb reproducibility on two different elements, Zn (#) and Al (*), reveals the high accuracy of the chemistry.

AA3002:
Determination of Trace Lead, Poly-Phenols and Tannins in Wines Using a Single Analytical Instrument

The manufacturing of wine and other fermented beverages has typically been a fairly simple process. Historically, the expertise of the sage wine-master was used to gauge the quality of the product. Today, many environmental concerns, health factors, and government rules, prompt the producers of fine wines to monitor several important components in the grape, the fermentation vats, and in the final wine. Typically, several different analytical instruments have been used to perform these tests including Flame Atomic Absorption Spectrophotometers (**FAAS**), UV Spectrophotometers (**UV**), and Colorimeters (**VIS**).

The 210VGP Atomic Absorption system from Buck Scientific, Inc. employs a high performance monochromator and a unique cell-holding attachment that allows it to be used as three analytical tools in one: FAAS, UV and VIS. This means that three of the more critical tests can be performed with a single device. The FAAS system normally uses a Lead Hollow Cathode Lamp (HCL) to measure the **Lead** signal at 283nm, while the UV test for **Poly-Phenols** at 280nm is best done with a D_2 (Deuterium) HCL. The VIS determination of **Tannins** at 520nm uses a Cr (Chromium) HCL and the Buck #5711 cell holder. Tannins can also be measured at 760nm using a Cs (Cesium) HCL. Other test procedures for measurements at 420nm and 620nm can be done with a Rb (Rubidium) HCL. Examples of actual samples are listed on the back.

Analyst: Gerald J. DeMenna

SIC: 2084

Basic Conditions: (for best linearity and precision)

Lead Calibration:	5 ppm Standard and an Acetic Acid Blank
Phenol Calibration:	2.0, 5.0, and 10 ppm in 5% Acetic Acid; 10 cm Quartz
Tannin Calibration:	25, 100, and 500 ppm in Water: Ethanol; 5 cm Glass

Sample ID	**Pb (ppm)**	**Phenols (ppm)**	**Tannins (ppm)**
Napa Varietal			
Must	8.7	768	2,570
Napa Varietal			
First Ferment	6.3	227	982
Medocino County			
Sauvignon Blanc	0.93	169	64
LaCour Pavillion			
Bordeaux	1.4	403	1,328

AA3003: Determination of Major Components and Trace Contaminants in Assorted Plating Baths by Atomic Absorption Analysis

Precious metal, non-ferrous, and ferrous plating operations cover almost every facet of modern life: automotive parts, kitchen & bathroom fixtures, electrical parts, electronic components, furniture, tools, plumbing, construction supplies, die castings, jewelry, etc.

Good manufacturing practices, and lately, government regulations for environmental safety, have prompted many plating operations to perform more stringent tests on their products. This determines the composition of the major elements in the bath, and also verifies the "cleanliness" of the bath (i.e. no toxic or hazardous metals). Checking the effluent and wastewater from a production operation is also required under most state guidelines. In the past, simple titrimetric or colorimetric tests were sufficient for the determination of major components, but lacked the speed for good quality control methodology, and sensitivity for trace analysis.

Atomic Absorption Spectrophotometry has been an accurate and precise analytical tool for many laboratory environments for over 50 years. The general purpose system lacks the spectral interferences of emission spectroscopy techniques and is significantly more sensitive and accurate than methods such as X-ray fluorescence. The Buck 210VGP AA system is substantially less expensive than almost any other analytical tool, while keeping its unique flexibility and versatility.

Lab Requirements:

5.0ml & 10.0ml Class-A Pipets, 100ml & 500ml Class-A volumetric flasks, distilled water, 110V AC, and air & acetylene tanks.

Procedure:

(1) Pipet 5.0ml of bath into a 100ml flask and dilute with water. Dilution = **1:20** / Use to determine trace elements (~200 ppb)

(2) Pipet 10.0ml of **1:20** into a 100ml flask and dilute with water. Dilution = **1:200** / Use to analyze minor elements (0.1% - 0.0005%)

(3) Pipet 10.0ml of **1:200** into a 500ml flask and dilute with water. Dilution = **1:10,000** / Use to analyze major elements (10% - 0.1%)

Calibration:

A 1.0 ppm standard and matrix blank for trace elements. A 10 to 50 ppm standard and a water blank for major / minor elements.

Analysis:

Insert element lamp, peak in wavelength, optimize energy, set blank zero, calibrate on standard, aspirate sample and read concentration.

The Buck 210VGP Series can accurately determine most elements from 100 ppb to over 10%, depending on the dilution, with typical precision levels of 1.5% or better if using bracketed standards. The entire procedure can be standardized to a simple, easily followed format for a lab technician.

SIC: 3471

Analytical Results for Various Plating Baths

Samples: Three (3) baths from an electronics application, a die-cast factory and a chrome-finishing fixtures plant.
Preparation: Three serial dilutions, as previously described.
Calibration: Buck Certified Atomic Absorption Standards.
Instrument: The Buck 210VGP Atomic Absorption Spectrophotometer.
Conditions: Air / Acetylene flame, Integrate mode, Normal parameters.

NOTE – All values listed below are in Volume Percent (%v) in the original bath sample unless otherwise noted.

AVG = Average of 3 preps, RSD = Relative Standard Deviation of 3 analyses.

	Silver-Palladium-Copper in HBF_4		Electroless Nickel / Tin		Hot Dichromate in H_2SO_4	
Element	**AVG**	**RSD**	**AVG**	**RSD**	**AVG**	**RSD**
(Majors & Minors in %v)						
Ag	1.37%	0.9%	0.09%	2.3%	<0.01%	-
Pd	0.77%	1.3%	<0.01%	-	<0.01%	-
Cu	2.75%	1.1%	0.73%	0.7%	0.20%	0.5%
Ni	0.23%	1.6%	5.51%	0.9%	1.08%	1.4%
Sn	0.10%	2.4%	2.74%	1.6%	0.63%	2.2%
Cr	<0.01%	-	0.26%	1.8%	7.59%	0.8%
Fe	0.02%	2.9%	0.42%	1.5%	1.16%	1.1%
Au	0.18%	3.2%	~0.01%	4.1%	<0.01%	-
Zn	0.34%	1.7%	0.69%	1.3%	1.03%	0.9%
(Traces in PPM, mg/L)						
Cd	79 ppm	2.8%	130 ppm	1.9%	27 ppm	5.3%
Pb	228 ppm	1.7%	472 ppm	0.7%	115 ppm	2.2%
Mn	24 ppm	3.9%	326 ppm	2.1%	471 ppm	1.8%
Co	<5 ppm	-	554 ppm	1.5%	83 ppm	2.3%

The above data shows the powerful flexibility and stability of the Buck 210VGP system for the wide-ranging requirements of the plating industry. The excellent correlation between major / minor values ranges from 0.7% to 3.2% (average RSD = 1.3%), exemplifying the high precision of the instrument. The overall accuracy of the various trace metals supports the interference-free quality of the data. Together, the combinations provide for an unmatched pair in economy and performance.

AA3004: Analysis of Whole Blood for Trace Lead (Pb) by Graphite Furnace AAS

Toxic effects from heavy metals (Pb, As, Se, Cd, Bi, Sn, Tl, and Hg) have been characterized in medical literature for decades. Originally, complex extractions were performed to isolate the poisonous components from the complex biological matrices: blood, serum, urine and tissues. Recently, optical emission and atomic absorption spectroscopy techniques were developed to determine these toxic elements. As analytical instrumentation performance improved, real limits of detection were also improved. The advent of Inductively-Coupled Plasma (ICP) and Graphite Furnace AAS (GFAAS) brought the limits of analysis into the parts-per-billion (PPB) range for many matrices. This capability has been invaluable in screening blood and urine samples from pediatric and chronically ill patients to track the exposure and elimination of lead.

The determination of trace lead in blood samples is very important, particularly in “third world” and urban environments, where environmental pollution, lead-based paints, gasolines, and poor plumbing systems increase the exposure of lead in various forms. The standard clinical procedure for the measurement of lead in blood is very simple and is based on the use of highly sensitive GFAAS instrumentation. The procedures listed below and the data were developed on and obtained from the Buck 210VGP Atomic Absorption System with the Model 220 Graphite Furnace option.

Sample / Standard Preparation:

There are several procedures for the quantitative and reproducible preparation of Whole Blood samples prior to atomic analysis. Below is the most popular technique:

(1) Collect Blood and preserve with Heparin, EDTA, or Oxalate.
(2) Dilute 100µL of sample with 400µL of “Diluent” (contains 0.25% Triton X-100, 2000 ppm ammonium dihydrogen phosphate, and 750 ppm magnesium nitrate). These “matrix modifiers” serve to aid hemolysis, sequester the Pb, and increase atomization efficiency.
(3) Make 0, 0.01, 0.05, 0.1, 0.25, and 0.5 mg/L lead standards in diluent.
(4) Analyze 20µL loads with the following GFAAS conditions: 283.3nm wavelength, 7A slit, D2 background correction, Peak Height, Argon purge (~50ml/min), Auto-Zero off, grooved furnace tube. Dry: Ambient to 125°C in 15 second ramp, 5 second hold. Ash: 125°C to 600°C in 45 second ramp, 20 second hold. Atomize: 600°C to 2400°C in fast ramp or step, 5 second hold.

This technique provides a well-matched calibration equivalent to 40, 200, 400, 1000, and 2000µg/L (ppb) lead in the original blood sample. The precision ranges from ~3% relative at the upper limit to ~8% at the low end, with a method detection limit of 25µg/L in blood calculated from the blank variance taken at 2-sigma.

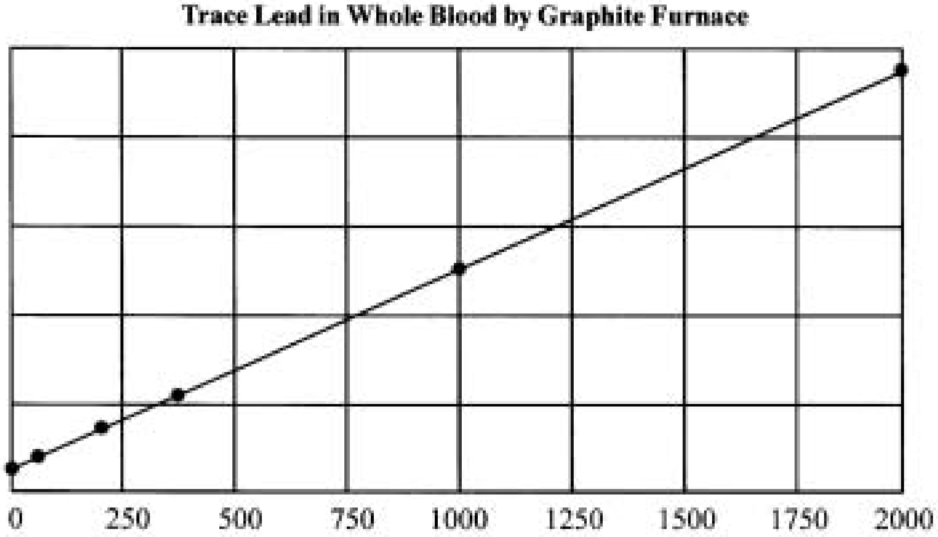
SIC: 801, 8062, 8071

Analysis of Whole Blood for Trace Lead (Pb)

Samples:	“blank” pooled Blood sample (Red Cross), no Lead. “real” pediatric Blood collection (Newark Children’s Hospital) “spiked” reference Blood (S-K Bioscience, asssay = 50.6 PPB)
Results:	Values are µg/L (PPB) in the original diluted samples:

Sample ID	**Trip. Readings**	**Average**	**R.S.D.**
“blank”	23, 27, 26	25.3	6.7%
PED-113	169, 181, 166	172	3.8%
Ref-50	53, 47, 49	49.7	5.0%

Standard Calibration Curve: (linear regression)



Correlation Coefficient
of Calibration = 0.996

Correlation of SKB
Reference to Standards = 1.77%

The superb precision and accuracy shown by the above data are typical of the quality optical components and engineering design of the Buck AA systems. The industrially rugged nature of this design also allows the instrument to be used in a wide range of environments, from Hospital R&D Centers, to Clinical Testing Laboratories with much ease and confidence.

AA3005: Determination of Wear Metals and Additives (Soaps) in Lubricating Oils by Atomic Absorption

Lubricating & hydraulic fluids are crucial to the proper functioning of many types of machines: automobiles, farm equipment, heavy-duty motors, airplanes, and military hardware. Most “lubes” are simple hydro-carbon based oils or greases that have certain additives in them to prevent wear and abrasion of components (piston rings, bearings, etc.) or oxidation degradation of the oil. These additives are typically “soaps” made from an organic acid and a metal (usually barium, zinc, calcium, or magnesium). As the oil ages, these materials are continually lost and need to be monitored so that they can be replaced to provide the proper level of protection.

Wear metals are formed in lube oils under the harsh conditions of temperature and pressure that occur in heavy machinery. Under continuous heat and pressure, the surface of the metal piece becomes slightly oxidized, forms salts with the degradation products of the oil, and becomes soluble in the oil. The friction of motion in machinery also causes micro-fine particles to actually shear off the surface and become suspended in the oil. Determining what these “wear metals” are and their respective levels is very important to the proper maintenance and operation of the equipment. Copper & tin can come from bearing wear, lead & antimony come from Babbit metal bushings, chromium & molybdenum from piston rings and seals, nickel & iron from crankshaft wear and engine body damage, aluminum & silicon from catalyst residues, and sodium & silicon from bad air filters and from water / coolant leaks. Careful monitoring of these materials can be used in preventative maintenance of large, expensive engines, minimizing potential repair costs.

One of the best technologies for this type of determination is Atomic Absorption Spectrometry. There are several techniques for the analysis of metals in petroleum matrices by AAS. The simplest is dilution of the oil with a solvent like MIBK or kerosene, and analysis using organo-metallic standards. This solution burns well in an air / acetylene flame and gives very good detection limits. Another calls for the dry ashing of the sample, where the organics are burned off to leave an inorganic residue (ash). The ash is dissolved in acids and analyzed as an aqueous solution. However, significant errors can occur in this method because many metals are easily volatilized (sublimation, distillation), giving low assays. Recently, the advent of microwave digestion technology enables the chemist to decompose the oil with acids under pressure. This results in a clear, aqueous solution that can be analyzed using simple water-based standards. For any of these methods, the Buck 210VGP system and an optional MEGA-1200 Microwave Digestion system are ideally suited for rapid and economical determinations of wear metals and additives in all types of hydrocarbons and lubricant media.

SIC: 2992, 35, 55, 753

Analysis of Wear Metals and Additives in Lubricating Oils

Sample: 30W Engine Oil; abused & oxidized, 0.1 – 10 micron particulates.

Preparation:
(1) 10 grams diluted with 3:1 MIBK / Xylene solvent to 100ml
(2) 10 grams dry ashed @ 650°C, dissolved in acids to 100ml
(3) 10 grams microwave digested in HNO_3 / $HClO_4$ / H_2O_2 to 100ml

Calibration: Buck Certified AA Standards; Aqueous and Organo-Metallic, at 0.5 ppm and 5 ppm levels in appropriate matrices.

Instrument: Buck 210VGP Atomic Absorption Spectrophotometer.

Conditions: Air or N_2O / Acetylene flame, normal parameters.

Values are mg / kg (ppm) in the original sample unless otherwise noted.
AVG / sd = Average & deviation of 3 preps; D.L. = 3-sigma detectability.

		Method (1)		**Method (2)**		**Method (3)**	
Element	**Wavelength (nm)**	**AVG / sd**	**D.L.**	**AVG / sd**	**D.L.**	**AVG / sd**	**D.L.**
Ag	328	2.7 / 0.4	0.25	1.9 / 0.8	0.08	2.5 / 0.1	0.01
Ca	240	115 / 8.5	0.65	103 / 23	0.25	122 / 6.5	0.20
Cr	357	32 / 2.1	0.50	24 / 4.7	0.12	30 / 1.6	0.03
Cu	324	12 / 0.5	0.22	11 / 1.3	0.03	12 / 0.1	0.01
Fe	248	47 / 0.9	0.43	44 / 2.4	0.05	45 / 1.1	0.02
Na	589	4.9 / 0.8	0.46	3.1 / 1.1	0.31	5.5 / 0.4	0.17
Ni	232	8.4 / 0.7	0.55	7.9 / 1.4	0.17	8.1 / 0.5	0.12
Pb	283	18 / 0.9	0.65	11 / 3.7	0.27	16 / 0.4	0.15
Si	251	5.4 / 1.1	0.77	3.6 / 1.5	0.45	5.8 / 0.7	0.31
Sn	224	28 / 2.1	0.75	19 / 5.8	0.34	26 / 1.0	0.25
Zn	214	205 / 4.2	0.51	188 / 6.8	0.22	196 / 3.10	0.13

The above data shows the powerful flexibility and stability of the Buck 210VGP system for the wide-ranging requirements of the “wear metals” industry. The high precision of the major and trace data exemplifies the accuracy of the instrument. The overall high sensitivity of the various trace metals supports the interference-free quality of the data. Together, the combination provides for an unmatched pair in economy and performance.

AA3006: Determination of Major Electrolytes, Minor Minerals, and Trace Heavy Metals in Physiological Fluids by Flame and Graphite Furnace Atomic Absorption Spectroscopy

Accurate measurements of specific metals in various biological systems is extremely important. These metals can be classified into several groups: the high level major electrolytes (Na, K, Ca, Mg), which are crucial to normal primary physiological processes like cellular activity and metabolism; the minor minerals (Fe, Mn, Zn, Co, Cu), which are present in lower levels to act as metabolic agents and enzyme catalysts; and the trace heavy metals, or toxics, that should not be present in a healthy organism (Ag, Al, As, Se, Pb, Cr, Cd, Hg, Ni, TI, Sr, Ba and V). These last elements are either known to be detrimental to the body, or have a currently unknown activity that is purported to be harmful.

Such a listing of elements is prone to rapid changes. Some vitamin metabolic processes and enzymatic reactions appear to need very small amounts of “poisonous” metals like Se and Cr. Metals like Al and Sn do not have any well-documented harmful effects, but are believed to be related to Alzheimer’s Disease and kidney dysfunction, respectively. Even certain “odd” metals like Mo and Au, not normally found in the food system, are found to disturb digestion and skeletal processes. Other elements like Li, Pt, and La are being used in drug products and require monitoring in the body. Obviously, there is a need for an analytical system to be able to differentiate and quantitate these 20+ elements easily and reproducibly.

The most economical instrument suitable for this work is the Buck 210VGP Atomic Absorption Spectrophotometer. The Buck 210, based on a unique “Stable-Beam” technology that gives superior sensitivity and stability over older double-beam systems, is extremely simple to install and operate. The various excitation “sources” (air, nitrous oxide, hydride, and graphite furnace) allows analyses to be made in a multitude of matrices (tissue digests, serum, plasma, whole blood, packed RBC’s, urine, amnion, CSF, etc.) for metals from the parts-per-billion to the percentage level. Most clinical environments prefer to mg% or mEq/L for the major electrolytes and in mg/dL (or µg/dL) for the toxic elements.

Chemical considerations are quite simple for handling physiological matrices, particularly if employing the Buck 210AA with its perfectly balanced “in-line” deuterium lamp background corrector. This feature permits the analyst to use a single, simple aqueous standard for calibration of many different sample matrices, which would have required individual matrix-matched standards for each type of sample.

The enclosed data shows a simplified, standardized technique for the preparation of various body fluids, and the conditions for the precise determination of a range of metals.

SIC: 8071

Analysis of Electrolytes, Minerals, and Trace Heavy Metals …

Samples: (1) Whole blood, collected in EDTA or Citrate & chilled
(2) Serum, centrifuged to separate all solids
(3) Reconstituted Packed Cells, in isotonic saline
(4) Urine, collected, and refrigerated

Preparation: A “Physio-Sol” diluent developed at Buck Scientific is used for cell hemolysis (TX-100), stabilization (EDTA & Tartrate), and spectrographic buffering (Lanthanum) of the sample.

High concentration electrolytes are measured in a 1:50 dilution:

(1) 1.0ml sample in 50ml centrifuge tube, add 10.0ml of Physio-Sol™, vortex for 60 seconds, dilute to 50ml with DI Water, re-vortex to mix.

Low level minerals and toxics are determined in a 1:5 dilution:

(2) 2.0ml sample in 10ml centrifuge tube, add 2.0ml of “Physio-Sol”™, vortex for 60 seconds, dilute to 10ml final volume and re-vortex.

Calibration: A 20% “Physio-Sol”™ Blank (0) is used with a High-Std of 1.0ppm (mg/L) made up in a 20% “Physio-Sol”™ matrix. Use for all analyses.

Instrument: Buck Model 210VGP AAS with attachments as described below:

Conditions: Air Flame for Na, Mg, Cu, Zn, Fe // Nitrous Flame for Ca // Furnace for Pb, Cr, Al //
Hydride for As // Cold Vapor for Hg //
Values are mg/L (ppm) in the original sample unless otherwise noted.

Element	**Wavelength (nm)**	**Detection Limit**	**SMP-1**	**SMP-2**	**SMP-3**	**SMP-4**
Na	589	N/A	1890	3220	255	750
Mg	285	N/A	44	26	79	7.8
Ca	422	N/A	83	96	6.1	56
Cu	324	0.2	0.94	1.2	0.66	N/D
Zn	214	0.1	7.9	1.3	5.7	~0.1
Fe	248	0.05	1.85	1.2	2.2	0.07
Cr	357	0.01	0.02	0.05	N/D	N/D
Al	309	0.07	0.13	0.19	N/D	0.08
Pb	283	0.02	0.48	0.04	0.40	N/D
As	193	0.01	0.27	0.12	N/D	0.05
Hg	253	0.005	N/D	N/D	N/D	0.02

The above data shows the powerful flexibility and stability of the Buck 210VGP systems for the wide-ranging requirements of the Clinical Laboratory. The high precision of the Major and Trace data exemplifies the stability of the instrument. The overall high sensitivity of the various Trace metals supports the interference-free quality of the data. The result is an unmatched system in economy and performance.

AA3007: Determination of the Environmental TCLP Metals in Waste-Waters, Solid Wastes, and Soils by Flame Atomic Absorption Spectrophotometry

Hazardous wastes can originate from many sources – industrial processes, product formulations, synthetic operations, virtually any manufacturing business. The nature and composition of the "wastes" can vary so much that one industry's discharge can serve as another's raw materials. It is the tremendous transfer of these materials – their storage, transportation, spillage and leakage into the environment – which has caused great concern. Regulation of these discharged solid and liquid materials is a crucial part of the U.S. EPA and the State-level Departments of Environmental Protection programs for defining the "degree of hazard" of a particular material. The basic methods describe procedures to "leach" any soluble heavy metals from samples using acid(s) (to simulate acid rain, for example). While there is a lot of printed material available from these Government agencies, the basic protocols for the collection and preparation of this type of sample is referenced in the Standard Methods of SW-846 as part of the Resource Conservation and Recovery Act (RCRA).

Principle

The Toxic Characteristic Leach Procedure (TCLP) is a specific protocol to extract and decompose any complex or bound metals in a waste-water, solid waste or soil sample. This makes a solution suitable for atomic analysis by Flame Atomic Absorption Spectroscopy (FAAS) or Inductively Coupled Plasma Optical Emission Spectrocopy (ICP-OES). Flame AAS is an inexpensive, efficient method that has been time tested for many decades. ICP-OES is a newer technique that is faster, but significantly more expensive than FAAS. The sample preparation range from boiling water samples with acid, to shaking a solid sample in an acid slurry for 18 hours, to digesting at controlled temperatures in a fluoro-polymer microwave oven pressure vessel. The protocols for microwave oven assisted leachings are defined in the U.S. EPA Methods 3015 (liquids) and 3051 (solids).

Practice

An assortment of samples is prepared according to the appropriate methodology, and the resulting solutions are ready for instrumental analysis. The Buck Model 210VGP Flame AA system with the Nitrous Oxide assembly and the Model-420 Hydride unit are set up for running the metals listed in the TCLP program. Accuracy is ensured and detection limits are maximized by using the unique "in-line" Deuterium Background Corrector and the wavelength-independent Variable Giant Pulse Corrector (standard parts of the 210VGP) to eliminate any interferences in the sample measurements.

Analyst: Gerald J. DeMenna

SIC: 071, 8734, 951

Determination of the Environmental TCLP Metals

Samples:	A composite wastewater sample, a 30' deep drilling core soil sample, and a settling pond sludge samples were analyzed for 12 metals.
Preparation:	(Modified from the EPA guideline methods for TCLP & RCRA) [1] 100ml wastewater boiled for 5 minutes with 5ml of nitric acid. [2] 50 grams soil, leached with 100ml of 10% nitric acid / 5% acetic acid. [3] 10 grams sludge, digested with 25ml of 25% nitric acid / 10% acetic acid.
Calibration:	Certified EPA-type standards, containing the 12 analytes at levels from 0.1 to 5 ppm (mg/L), were used for a linear calibration curve.
Instrument:	Buck 210VGP Atomic Absorption Spectrophotometer, D_2 Corrector, Nitrous Oxide burner, Hydride generator with Argon / Hydrogen.
Conditions:	Air / Acetylene flame (Ag, Cd, Pb, Ni, Cr) N_2O / Acetylene flame (Al, Ba, Be) Hydride Generator (As, Se, Sn) Cold Vapor Generator, Quartz tube (Hg)

NOTE – Values are mg/L (ppm) in the original sample // D.L. = 3-sigma detectability

Element	**Wavelength (nm)**	**Waste-D.L.**	**Water**	**Soil**	**Sludge**
Ag	328	0.04	0.09	0.36	6.97
Cd	228	0.02	0.23	0.69	1.21
Pb	283	0.15	0.72	1.82	8.03
Ni	232	0.10	2.48	2.17	5.85
Cr	357	0.08	1.34	0.61	3.53
Al	394	0.65	5.63	(100)	(100)
Ba	553	0.35	4.52	2.43	0.54
Be	234	0.02	0.03	<0.02	0.39
As	193	0.001	0.28	0.13	0.74
Se	196	0.002	0.16	0.05	0.83
Sn	286	0.005	0.08	0.17	1.49
Hg	253	0.00001	0.0008	0.0012	0.0185

The above data shows the powerful flexibility and stability of the Buck 210VGP systems for the wide-ranging requirements of the Environmental industry. The overall high sensitivity of the various Trace metals supports the interference-free quality of the data. The combination of components provides for an unmatched system in economy and performance.

AA3008: Indirect Determination of Gold Purity by Measurement of Impurities with Flame Atomic Absorption Spectroscopy

The determination of the purity of scrap and refined gold is a very important procedure for the financial markets, the jewelry business and the electronics industry.

Over the years, the classical fire assay technique has been used for gold determination but this is a very labor-intensive procedure, and prone to many operator errors. Hundreds of books have been written with special steps and secret protocols to follow for accurate and reproducible fire assays; but it still is a subjective method and is sometimes used more for semi-quantitative analyses for those labs without experienced asssayers.

Trying to determine the exact concentration of gold instrumentally can be done directly by measuring the gold itself, but preparing an accurate dilution of a sample is very difficult unless the volumetrics are calibrated, the water is thermostatted and the balance is certified. Slight changes in temperature, or vessel cleanliness or calibration can result in percentage errors in the final data. The error of the measuring instrument (Gravimetric balance, Arc / Spark, Flame-AA, Plasma Emission, etc) can also affect the final value by defining a limiting precision; for example: D-C Plasma has a systematic error of 1.5%; so the best data for gold off a DCP instrument will have a built-in error, or variability, of 1.5%. A better, and more useful procedure, is determining the total impurities in the sample and subracting this from 100% to get

the gold by difference. Many large companies (Engelhard, Texas Instruments, Krementz) use this procedure to assay their incoming gold. Reporting errors can be reduced to less than 0.1% in some case for a more accurate purity determination of the gold material.

Atomic Absorption Spectroscopy is a very acceptable technique for gold assays, and is a very simple technique for technicians and chemical operators to learn. Sample preparation and calibration are easily performed according to a standardized procedure, and the operation of Buck AA instruments takes only a few hours to master. Performing these analyses is actually simple on the Buck Model 210VGP Atomic Absorption system. The high optical throughput and stability of our “Stable-Beam” design provide excellent sensitivity, superb precision and, with the proprietary Background Correction modes on the 210, complete freedom from interferences. The unique single-mirror monochromator used in the optical system is the key to the system’s small size and high performance. It is the incorporation of the “in-line” Deuterium Background Correction that will minimize or eliminate most types of spectral or spectrochemical interferences without any loss in data quality. The data shown on this appnote is a clear statement to the stability and energy of the Buck AA instruments.

Analyst: Gerald J. DeMenna

SIC: 1041, 367, 3911, 6011

Analysis of Gold Purity by Measurement of Impurities

Samples:	[1] Smelted scrap (~75% Au) [2] Refined ore (~99% Au)
Preparation:	1.0gm in 25ml Aq. Regia, fumed with HCl, dilute to 100gm.
Standards:	A Hydrochloric Acid Blank (0), and a 1 ppm (µg / gm) Standard.
Results:	Values are ppm (µg / gm) in the original solid sample, with RSD calculations on triplicate runs (using 1:100 dilution factor).

Element	**Wavelength (nm)**	**Flame Type**	**Sample #1 / RSD**	**Sample #2 / RSD**
Silver	328	Air – even	5220 ppm / 1.3%	3.8 ppm / 2.5%
Cadmium	228	Air – lean	1740 ppm / 2.1%	<0.9 ppm / -
Chromium	357	Air – rich	120 ppm / 2.6%	<2.5 ppm / -
Copper	324	Air – even	51850 ppm / 0.8%	15 ppm / 1.8%
Iron	248	Air – lean	690 ppm / 3.0%	<3.0 ppm / -
Nickel	232	Air – lean	82700 ppm / 1.2%	33 ppm / 1.7%
Lead	283	Air – even	21500 ppm / 1.6%	12 ppm / 2.4%
Tin	286	N_2O – rich	60700 ppm / 3.2%	265 ppm / 4.7%
Zinc	214	Air – even	10350 ppm / 0.9%	21 ppm / 1.3%
Total Impurities PPM (wt%) =			234,870 (23.49%)	365 (0.036%)
Gold Purity (by difference) =			76.51% Au	99.964% Au

AA3009: Analytical Methodology for the Characterization of Steels and Iron Alloys by Atomic Absorption Analysis

The analysis of irons and steels has always been a very important job in the industrial and commercial environment. The grading of steels and iron alloys is critical to characterizing their respective strengths to corrosion and stress. There are also many protocols for the several hundred types of steels available. Fe is always the primary component, with elements such as Ni and Cr as major consstituents, and Mn, Zn, B, P, Cu, V, Ti, Co, Pb, Mo, Si, and Sn as essential minor elements. Much of the standard methods of analyses have been documented and issued by agencies like the ASTM (American Society of Testing and Materials), and the ISO (International Standards Organization). Some of the procedures listed here are taken from the ASTM Standards, volume 03.05 (Chemical Analysis of Metals); methods E350 (Low Alloy Steels), E351 (Cast Irons), E352 (Tool and High Alloy Steels, and E353 (Stainless Steels). All these standards employ Flame Atomic Absorption Spectrophotometry (FAAS).

Preparation of the samples and calibration of the instrumentation is actually quite easy using the Buck Scientific Model 210VGP AA system. The unique components allow interference-free measurements at high levels of sensitivity and precision. Aspiration of the sample by the high efficiency nebulizer provides tremendous sample throughput, and the proprietary "in-line" Deuterium (D_2) Lamp Background Corrector gives the highest energy of any commercially available system. These features combined with the single-mirror, high-resolution monochromator and low noise electronics create an integrated analytical tool of remarkable power.

An example of a "universal" sample preparation technique is listed below. This procedure gives complete recovery of all components with no losses or chemical interferences, and can be used for Certified Standards from ASTM, NIST / NBS, BCS, IUPAC, or other regulatory agencies. Two basic dilutions cover the range of concentrations from 0.001% up to 25.0%.

Preparation of Irons and Steels for Flame AAS Analyses: Aqua Nasty ™

[1] Weigh 1.0gm sample chips into a tared 4oz HDPE Nalgene bottle.

[2] Add 10ml HCl, 2ml HNO_3, 1ml HF, and 1gm $K_2S_2O_8$. Heat in water bath or in microwave oven for 5 – 10 minutes.

[3] Dilute to 100grams final weight, or cool and dilute to 100ml volume. This is the 1% solution (1:100 dilution factor) for minor elements.

[4] Weigh or pipet 1gm or 1ml of this to 100ml to make 100 ppm solution (1:10,000 dilution factor) for Major components.

The Buck 210VGP AA can accurately determine these major and minor elements, depending on the dilution used, with typical precision levels of 1% or better if using bracketted standards. The entire procedure can determine over 40 elements per hour for high turnover and high quality results.

SIC: 101, 331, 332, 3462

Analysis of Irons and Steels by Atomic Absorption Analysis

Samples:	Standard Reference Materials from NIST / NBS, ASTM, and BCS
Preparation:	“Aqua Nasty” method previously listed
Calibration:	Buck Certified Atomic Absorption Standards
Instrument:	The Buck 210VGP Atomic Absorption Spectrophotometer
Conditions:	Air / Acetylene flame, Integrate mode, Normal parameters N_2O flame for Sn, Si, Ti, V, and Mo

NOTE – All values listed below are in Weight Percent (%wt) in the original Steel sample; [K] = Known assay, [M] = measured data.

Element	**Cast And Pig Irons**		**Stainless #316S**		**Tool Steel No. 3**	
	[K]	**[M]**	**[K]**	**[M]**	**[K]**	**[M]**
Cr	2.45	2.48	18.1	18.0	0.69	0.70
Ni	1.17	1.19	12.9	13.0	0.21	0.20
Mn	0.79	0.80	1.77	1.75	0.18	0.19
Co	0.54	0.56	0.27	0.25	4.82	4.80
Al	0.35	0.33	0.88	0.89	0.22	0.24
Cu	0.67	0.67	0.24	0.23	.079	.080
Ag	.011	.010	.009	.008	-	-
Mo	0.48	0.50	2.11	2.10	0.48	0.47
Ti	0.17	0.15	0.44	0.45	-	-
Si	2.86	2.83	0.66	0.68	0.22	0.21
V	0.15	0.16	.021	0.22	0.13	0.12
Pb	0.17	0.16	0.28	.029	.059	.060
Zn	0.88	.090	-	-	.028	.029
Sn	.013	.011	.035	.036	.029	.030
Mg	.097	.098	-	-	.040	.039
Bi	.046	.045	-	-	-	-
As	0.12	0.11	-	-	-	-

The above data shows the powerful flexibility and stability of the Buck 210 system for the wide-ranging requirements of the Iron and Steel industry. The excellent correlation between the Known and Measured values ranges from 0.6% to 3.0% (average RSD = 1.6%), exemplifying the high precision of the instrument. The fact that a single preparation gives superb reproducibility on three (3) widely varying metals, with a range of difficult elements, reveals the accuracy of the chemistry. The above components provide for an unmatched system in economy and performance.

AA3011: Evaluation of Mineral Supplements for Content and Purity by Flame / Graphite Furnace AAS

Doctors, dieticians and health officials recommend a balanced diet that provides essential minerals for proper nutrition in the human body. The accurate analysis of metals in vitamin / mineral preparations and food supplements is very important. These metals can be classified into several groups – the major electrolytes (Na, K, Ca, Mg), crucial to normal primary physiological processes like cellular activity and metabolism; the minor minerals (Fe, Mn, Zn, Cu), present in lower levels to act as metabolic agents and enzyme catalysts; and the micro (trace) minerals, in low levels (Se, Cr, Mo), for less defined reactions. There is a need for an analytical tool to quantitate these elements easily and reproducibly. Ferrous gluconate is a source of iron, but can be contaminated with manganese, titanium and vanadium. Dolomite is an excellent source of calcium and magnesium, but is often contaminated with lead and silver. Zinc oxide provides an essential form of easily absorbed zinc, though the amount of cadmium and arsenic found can be a problem.

Examining some of the common over-the-counter and prescription formulations, one can see that the potential contaminants present in some of these higher concentration minerals is as important as the accurate determination of these essential minerals themselves. For this type of low-level analysis in such a complex matrix (high organics and alkali elements), graphite furnace atomic absorption spectroscopy (GFAAS) with deuterium background correction (D2) is one of the few acceptable techniques for error-free determination of trace metals. GFAAS is also the preferred technique for measuring the desired micro-minerals. The electrolyte metals and minor minerals are best done by simple flame AA (FAAS).

Both flame and furnace techniques can suffer from tremendous interferences when low level elements are determined in "dirty" or complex matrices. The alkali elements (Na, K, Mg, Ca) and the refractory metals (Al, Si) can create background effects that will add signficant inaccuracies to an analysis. The presence of organic materials in the sample matrix can cause the formation of smoke particulates in a graphite furnace analysis and cyanogen bands in a flame method; which also contribute to analytical errors. The nature of these interferences can be compensated for by the use of an efficient D2 Background Correction system; and the unique design of the Buck 210VGP AA system provides the maximum energy throughput for maximum sensitivity and accuracy.

Analyst: Gerald J. DeMenna

SIC: 074, 20, 283, 512

Evaluation of Mineral Supplements for Content and Purity by Flame / Graphite Furnace AAS

Samples:	1) An enriched Wheat Flour; 2) A Multi-Vitamin & Mineral Capsule; 3) A Dolomitic Limestone [a natural Calcium and Magnesium source]; and 4) A Mineral Supplement Concentrate
Preparation:	Dissolve 1gram of sample or 1 capsule in 100ml 5% Nitric Acid. Filter and dilute to 50ml to make a 1:50 dilution, or a 2% solution to be used for **trace** elements. Dilute 1ml of this to 100ml for a 1:5,000 sample dilution for the **electrolytes** and **major** metals.
Calibration:	Buck Certified standards at 5 ppm [Flame] and 0.1 ppm with modifiers for [Furnace].
Instrument:	Buck 210VGP AA with the 220 Graphite Furnace and 420 Hydride accessories as noted.
Conditions:	Flame for Na, K, Mg, Ca, Cu, Zn, Fe, Mn Hydride for Se and As Furnace for Cr, Mo, Ag, Pb, and Cd Values are noted as Percent, ppm, or ppb in the **original** sample; or mcg in the capsule.

Element	**Wavelength**	**Detection Limit**	**Flour**	**Capsule**	**Lime**	**Conc**
Sodium	589 nm	0.5 ppm	2,575 ppm	<10 mg	0.83%	255 ppm
Potassium	766	2.0 ppm	4,150 ppm	15 mg	0.24%	810 ppm
Magnesium	285	0.05 ppm	375 ppm	20 mg	9.82%	175 ppm
Calcium	422	1.0 ppm	1,500 ppm	100 mg	17.2%	75 ppm
Copper	324 nm	0.05 ppm	21 ppm	120 mcg	0.09%	550 ppb
Iron	248	0.10 ppm	315 ppm	100 mg	0.35%	1070 ppm
Manganese	257	0.10 ppm	46 ppm	1.5 mg	0.18%	58 ppm
Zinc	214	0.05 ppm	185 ppm	5 mg	1.04%	245 ppm
* Arsenic	194 nm	10 ppb	<10 ppb	<5 mcg	47.5 ppm	<10 ppb
Selenium	196	25 ppb	69 ppb	7.9 mcg	16.3 ppm	78 ppb
Chromium	357 nm	50 ppb	3.8 ppm	12.5 mcg	0.33%	155 ppb
* Lead	283	25 ppb	<25 ppb	<10 mcg	0.29%	<25 ppb
* Silver	328	10 ppb	1.7 ppm	<5 mcg	0.12%	<10 ppb
* Cadmium	228	5.0 ppb	<5 ppb	<2 mcg	665 ppm	<5 ppb
Molybdenum	313	50 ppb	2.4 ppm	9.3 mcg	0.09%	165 ppb

Sample and Standard Preparation procedures for Trace Elements in Lead

[1] Preparation of Lead (Pb) Samples:

Drill Pb metal to make small pieces. Weigh 2.0 grams of these pieces on a balance. Place the sample in a 400ml Pyrex breaker and add 10ml H_2O, 2.5 grams of tartaric acid and 7ml HNO_3. Warm on a hot-plate until the sample dissolves. Dilute to 100ml in a volumetric flask with water (H_2O). This prepares a 2% sample solution, with a dilution factor of 50.

[2] Preparation of Sulfuric Acid (H_2SO_4) Samples:

Using a graduated cylinder, place 75ml of water (H_2O) into a 100ml volumetric flask. Slowly add sulfuric acid (H_2SO_4) to the water, swirling to mix. **Solution will bubble up and get hot, do NOT spill it on yourself – wear gloves!** Add $H2SO_4$ to bring the final volume to 100ml. This prepares a 25% sample solution, with a dilution factor of 4.

[3] Preparation of Pure / Waste Water (H_20) Samples:

Using a digital pipettor or glass pipet, add 1ml nitric acid (HNO_3) to 100ml volumetric flask. Add water sample to 100ml mark. There is basically no dilution and the energy relates directly to the sample.

[4] Preparation of Multi-element Standards for Calibration:

Using a pipet or pipettor, add 100ml of each of the 1000ppm stock concentrate standards (Buck standards) to a 1 Liter volumetric flask containing 250ml water (H_2O) and 50ml nitric acid (HNO_3). The following groupings will prepare stable 100ppm Stock Standards . (Add water to bring final volume to 1 Liter):

A) Bi, Ni, Ag, Zn, Cu, Cd
B) Fe, Sb, Sn, As, Al, Ca

Dilute the 100ppm Stock Standards into the following Working Standards:

25ml	Stock + 1ml HNO_3 to 100ml in Volumetric Flask =		25ppm
10ml	“	“	10ppm
5ml	“	“	5ppm
2ml	“	“	2ppm
1ml	“	“	1ppm
0.5ml	“	“	0.5ppm

Use these standards for Cu, Ag, Fe, Ca:
0, 0.5, 2, 5ppm.

Use these standards for Ni, Zn, Cd:
0, 0.5, 1, 2ppm.

Use these standards for Bi, Sb, Sn, As, Al, Pb:
0, 5, 10, 25ppm.

Analyst: Gerald J. DeMenna

SIC: 173, 351, 353, 361, 362, 366, 3691, 3692, 3714, 3724, 5013

Determination of Trace Element in Lead

Samples:	Lead Sample #19, Lead Sample #21, Sulfuric Acid (~98%)
Sample Prep:	2% solutions of lead in 5% HNO_3 / 2.5% tartric acid; 10% solution of sulfuric acid (1:10 dilution)
Calibration:	0.5 and 2.5 µg / ml (ppm) analyte metal standard in 2% high-purity lead matrix, 2% high-purity lead matrix blank; for lead samples. 1.0 µg / ml (ppm) analyte metal standard in pure (distilled / deionized) water, pure water blank; for sulfuric acid sample.
Instrument:	Buck 210VGP Atomic Absorption Spectrophotometer, Giant Pulse and In-Line D_2 Correction, and Model 420 Hydride Generation system.
Conditions:	Standard operating conditions for 210 unit; analytical parameters and correction modes as listed per element; air / acetylene flame for Ni, Ag, Zn, Cu, Fe, Cd; nitrous oxide / acetylene for Al, Ca; argon hydrogen for As, Sb, Sn, Bi.
Results:	Values are weight percent (% w/w) in original sample: Data based on 1:50 Pb dilution and 1:10 H_2SO_4 dilution: D.L. [detectability] based on 2-sigma statistics for Pb samples.

Element	**Wavelength**	**D.L.**	**Lead # 19**	**Lead # 21**	**H_2SO_4**
Ni	232nm	0.0015%	<0.0015%	0.0018%	<0.0015%
Ag	328	0.0003	0.0017	0.0018	0.0005
Zn	213	0.0003	0.0009	0.0004	0.0027
Cu	324	0.0005	0.0150	0.0093	0.0021
Bi	223	0.0008	0.0167	0.0184	<0.0008
Fe	248	0.001	<0.001	0.002	0.005
Sb	217	0.0005	<0.0005	0.0012	<0.0005
Sn	224	0.0007	<0.0007	0.0009	<0.0007
As	193	0.0001	<0.0001	0.0011	<0.0001
Cd	228	0.0005	0.0006	0.0009	0.0008
Al	309	0.005	<0.005	<0.005	0.006
Ca	422	0.0005	0.0006	0.0022	0.0472

These data show the powerful flexibility and stability of the Buck 210VGP system for the wide-ranging requirements of the manufacturing industry. The overall high sensitivity of the various trace metals supports the interference-free quality of the data. The combination of unique components provides an un-matched system in performance and economy.

AA3015: Evaluation of Drinking Water Quality by EPA-Certified Graphite Furnace AAS

With the development of residential homes in all parts of the World, there is a need for accurate, high-volume measurements of various Metals in the Drinking Water that people in these residences will live on. Accurate measurements of specific metals will determine the types of pipes used, the lifetime of the plumbing, the potential for mineral stains on porcelain, and more importantly, the overall health of the individuals. It is important to get a meaningful idea of the essential "electrolyte" elements, the "minor minerals"; and the "trace" or "micro-minerals" to verify the quality of the water.

In addition to these essential metals, all States require all determination of Priority Pollutant metals in all Potable Drinking Water; as defined in the guidelines of USEPA Standard Methods 200.6 / 200.9 for the Analysis of Heavy Metals in Drinking Water by GFAAS. These metals include Lead (Pb), the most highly analyzed metal on Earth; Mercury (Hg), Arsenic (As), Antimony (Sb), Cadmium (Cd), Nickel (Ni), Silver (Ag), Tin (Sn), Selenium (Se), Barium (Ba), Chromium (Cr) and Thallium (Tl).

The presence of metals in Drinking Water can affect all biological organisms; from human beings to pets to plants and agricultural systems. These metals can be classified into several groups: the high level major electrolytes (Na, K, Ca, Mg), which are crucial to normal primary physiological processes like cellular activity and metabolism. The minor minerals (Fe, Mn, Zn, Co, Cu), which are present in lower levels to act as metabolic agents and enzyme catalysts; and the trace heavy metals, or toxics, that should not be present at significant levels in a healthy organism (Ag, Al, As, Se, Pb, Cr, Cd, Hg, Ni, Hl, Sr, Ba, and V). These last elements are either known to be detrimental to the body, or have a currently unknown activity that is purported to be harmful.

Some biological processes and reactions appear to need very small amounts of "poisonous" metals like Se and Cr. Metals like Al and Sn don't have any well-documented harmful effects, but are believed to be related to Alzheimer's Disease and kidney dysfunction. Certain "odd" metals like Mo and Au, not normally found in the food system, are found to disturb digestion and skeletal processes. Obviously, there is a need for an analytical system to be able to quantitate these elements easily and reproducibly.

The most economical instrument suitable for this work is the Buck 210/211 Atomic Absorption Spectrophotometer. The Buck 210/211 is based on a unique "Stable-Beam" technology that gives superior sensitivity and stability over older double-beam systems. It is also extremely simple to install and operate. The various excitation sources (air, nitrous, hydride and graphite furnace) allows Potable Water analyses to be made with the highest level of sensitivity and precision; even at sub-PPB levels for some metals! This unsurpassed stability stems from the unique high-frequency power supply of the compact, back-mounted assembly and the very short electrical cables (<6") used to transfer "heat" to the Graphite. Other commercial systems employ long power cables, resulting in energy losses and poor reproducibility.

Chemical considerations are quite simple for "clean" water matrices when following EPA and most State guidelines for a Standardized Matrix Modifier. All potential interferences can be fully compensated for using either the high-energy "In-Line" Deuterium (D2) Correction, or the Zeeman-like Variable Giant Pulse (VGP) mode. This feature permits the analyst to use a single, simple aqueous standard for calibration of many different sample matrices.

Analysis of Potable Drinking Water by EPA-Certified GFAAS …..

Samples:
(1) New York City Drinking Water
(2) Los Angeles City Drinking Water
(3) Independent QC "Check Standard"

Preparation: This represents a "composite" protocol based on several State DEP and USEPA Standard Methods for the Collection, Preparation and Storage of Potable Water].

(1) Take a 100.0ml of the Sample, add 20ml concentrate (~65%) Nitric Acid; transfer to a plastic vessel (beaker, wide-mouth bottle); heat in a lab-guide microwave oven at low-medium power for ~10 - 15 minutes or until the volume is reduced back down to ~100ml. Cool and transfer to a Volumetric Flask, re-dilute to 100.0ml final volume.
(2) Buffer a portion of the Sodium for Flame-AAS with 1,000ppm Lanthanum (La) and Lithium (Li) for Ca, Mg, and Ba work; Matrix Modify another portion of GFAAS work with 1,000ppm Magnesium Nitrate and 500ppm Palladium Nitrate (per USEPA).

Calibration: A linear, 2-point Calibration was made using the appropriate Matrix Blank for the Flame or Furnace and the Standards set within the CAL MAX range for each metal.

Instrument: Buck Model 210/211 AAS with attachments as described below:

Conditions:
* Air Flame for Na, Mg, Ca, Fe, Cu, Cr
* Furnace for Pb, Cr, Ba, Al, As, Fe
* Cold Vapor for Hg.
* Nitrous Flame for Ba, Al
* Hydride for As

Values are µg/L (ppb) unless otherwise noted, with [%RSD} on triplicate measurements.

Element	Detection Limit	CAL MAX	METHOD	SMP-1 / NY	SMP-2 / CA	SMP-3 / NJ
Na	0.005ppm	2.0ppm	AIR-Flame	34ppm [2.1]	11ppm [1.8]	73ppm [1.5]
Mg	0.005ppm	3.0ppm	AIR-Flame	1.3ppm [1.9]	2.6ppm [2.3]	1.9ppm [1.3]
Ca	0.025ppm	5.0ppm	AIR-Flame	0.6ppm [2.2]	1.2ppm [2.1]	1.7ppm [1.8]
Fe	0.05ppm / 2ppb	5ppm / 250ppb	AIR / GF	0.48ppm [3.1]	0.29ppm [2.5]	0.18ppm [2.2]
Cu	0.005ppm / 1ppb	5ppm / 250ppb	AIR / GF	0.23ppm [1.6]	0.09ppm [1.4]	0.36ppm [1.5]
Ba	0.5ppm / 5ppb	25ppm / 500ppb	N2O / GF	375ppb [4.1]	625ppb [3.5]	170ppb [4.4]
Al	2ppm / 25ppb	50ppm / 1000ppb	N2O / GF	160ppb [2.9]	215ppb [3.1]	97ppb [2.7]
Cr	0.02ppm / 3ppb	5ppm / 250ppb	AIR / GF	745ppb [2.3]	460ppb [2.1]	255ppb [1.7]
Pb	0.8ppb	250ppb	GFAAS	2.1ppb [2.0]	3.4ppb [2.2]	1.1ppb [1.6]
As	0.001ppm / 1ppb	1.0ppm / 250ppb	HYD / GF	N/D	N/D	8.4ppb [3.4]
Hg	0.02ppb / (100ml)	2.0ppb	1018-CV	N/D	0.04 [6.7]	N/D

The above data shows the powerful flexibility and stability of the Buck 210/211 AA systems for the wide-ranging requirements of the water laboratory. The high precision of the Major and Trace data exemplifies the stability of the 210GF. The overall high sensitivity of the various Trace metals supports the interference-free quality of the data. The result is an unmatched system in economy and performance.

SECTION 15.0: Analyze/Buck Data Package V2.1

Installation

1) Place the Analyze/Buck data package CD-ROM into your computer's drive. If the setup program does not come up automatically, Click on **[start]** the **[run]** then "**setup.exe**".

2) Click on "**Install ODBC 2.1**", then **[Continue]**. Click on "**Microsoft FoxPro Driver (*.dbf)**", then click **[OK].**

3) Click **[Add]**, highlight "**Microsoft FoxPro Driver (*.dbf)**", then click **[OK].** In the "Data Source Name" field type "LABAUDIT" in all capital letters. Under version select "**FoxPro 2.5**". Then, you can either click the "Use Current Directory" box or select a new directory. Click **[OK]** when finished. Click **[Close]** then **[OK]** to return to the installation menu.

4) Click "**Install Analyze/Buck**". Follow the "Install Wizard" prompts to install the software package.

5) After the installation of Analyze/Buck is complete, you may wish to veiw the instruction manual for the software. If you do not already have Adobe Acrobat Reader installed on your computer, you must load it before you can veiw the manuals. Click on Adobe Acrobat Reader 5.0 and follow the install wizard to install. You may now click on the Analyze/Buck Manuals to select and view different parts of the manual.

Configuring the software

1) Double click on the icon to enter the Analyze software. The first screen that appears asks you to register the software. You may use the software for 30 days without registering but you will not be able to enter the program afterword without the code. You'll need to get a new code every time you load the software. To register, you must send the serial number you find in the CD-ROM cover, the reset number from the software, how many instruments you have connected and your companies name to Labtronics, Inc. A fax back form is included in the cover. After you receive the registration code, click the **[register]** button to enter the information and register.

2) If you wish to register later and finish the setup now, click **[OK]**.Click on "**System**" located in the top left corner of the menu bar. Then click "**Import Methods**" to load the Buck 210VGP methods from the Analyze CD-ROM. Select your CD-ROM drive then open the "**Methods**" directory. There are seven methods in the directory which may be used with your instrument. There are two types for each mode of operation:

> ***Absorbance Methods*** : Collect Absorbance data only. Calibrations are performed by the Analyze software which provides the capability to print the calibration curve as well as correlation coefficients. These methods are designated by _ABS file names.
>
> ***Concentration Methods*** : Collect Concentration and absorbance data. Allows Buck 210VGP to perform calibration. This method is preferred when using the 220AS Graphite Furnace Autosampler since the calibration may be used to set the peak timing automatically. These methods are designated by _CON file names.

The three modes of operation used on the Buck 210 are **Flame Absorption, Flame Emission** and **Graphite Furnace.** The Graphite Furnace methods may also be used for **Cold Vapor / Hydride** operation. The flam_215.exp file is for use on older instruments which use V2.15 firmware in the Buck 210. Load each method file individually by clicking on it's file name then clicking **[OK]**. In the field labeled "New Method Name", enter a name for the method (i.e. Flame Absorbance Method) to identify it in the Analyze Program, Then click **[OK]**. Repeat the process for any other methods you will be needing.

3) Click on **[system]** then **[configure methods]**. Click on the new method name then click **[OK].** Click on the **[Instrument]** button then the **[rs-232]** button and enter your computer ***serial port*** information and a ***baud rate*** (the maximum baud rate on the Buck 210 is 38400). Click on the **[OK]** button 3 times then click on **[system]** again. Repeat this step for all methods loaded above.

Running the software

1) Load the analysis you wish to run into a lamp position on the 210VGP. Leave the instrument in absorbance mode since all concentration values will now be calculated with Analyze/Buck.

2) Turn on your computer and click on the **[Analyze]** icon with your mouse to start the program.

3) Click on the **[run]** button (the leftmost button on the tool bar). Select the method for the Buck 210VGP by highlighting it's method name. If you wish to work on a worksheet that has already been created, highlight the worksheet number or description to bring this worksheet up, then click on the **[OK]** button. If you wish to create a new worksheet, click on the **[new]** button at the bottom of the window.

2) Type a description and operator name for the worksheet. Although this information is not required, it will allow you to identify the worksheet so you can look at it later. The time, date and worksheet numbers are set by the software and cannot be changed by the user. When you're done, click on the **[OK]** button. The software will setup the header on the worksheet automatically when data is first sent from the Buck 210VGP.

3) If you are running an Absorbance method, you will need to setup sample identifiers for your standards. If you are running a Concentration method, you may skip this step. Click on "**options**" then "**Analyze2**" then "**setup**" to get to the standard curve setup screen. Click on the "AutoCalc" on button so the software automatically calculates a concentration value for each absorbance reading. Click on the **[setup]** button next to Blank Correction. You'll see the default sample identifier for the blank is "Blank". Do not change the cell location, but you may change the ID. Click **[OK]** when finished. Click on the **[setup]** button for the Standard Curve. Here you'll see a table with default sample ID s S1-S20. You'll need to type in values into the concentration column for each of the standards you'll be using and make note of the ID. You may change the sample ID to make it easier to differentiate between standards. There is also options to set up concentration ranges, QC standard Checks, etc. on these screens. Consult the Analyze instruction manual for information on these options. When finished click **[OK].**

4) With the communications cable connected from the Buck 210VGP's "Comm Port" to the computer's serial port, Click on the **[start]** button (the one with a green arrowhead on it) below the Aanalyze software's menu bar. This function will initialize communication with the instrument, but will not start the analysis on the Buck 210VGP. To start the instrument, press the **[cntls]** key on the Buck 210VGP, highlight **[remote communication]** then press **[enter]**. Use the right arrow key to move the cursor to the **"remote mode"** option, then use the **[right]** arrow key to change to **[library/report]** then **[enter],** select proper baud rate using **[left/right]** arrows then press **[enter]**, then **[esc]** twice to return to the main screen. Press **[start]** on the 210 to run your standards & samples.

5) As you read your standards and samples in absorbance mode, a value for absorbance will appear in the Analyze worksheet a few moments after the absorbance value appears on the Buck 210VGP's active analysis screen. When you autozero the Autozero delta (representing the change in absorbance after autozero) will appear. For best results, Autozero on your blank first, read your blank and then your other standards in the order of increasing concentration so it will be easy to identify them when your ready to calibrate. For best results, enter the appropriate **sample ID** for each blank and standard as they are read so you don't count erroneous readings as standards. Use the value for reading the blank in your calculation and not the autozero delta. You **must** enter **sample IDs** exactly as they are written in the set up table (they are case sensitive) and all blanks and standards must have an ID or they will not be considered in the calibration. You may enter the sample IDs at any time. You may also wish to enter sample ID s for you sample as well, just be sure you don't use any sample ID s from the standard curve setup table.

SECTION 15.0: Analyze/Buck Data Package V2.1 *continued*

6) If you are not using AutoCalc, you'll need to calculate concentration values by clicking on **[options]** on the menu bar. Select **[Analyze2]**, then **[Calculate]**. Click on **[yes]** when you are prompted to update the worksheet. Click on **[OK].** If you wish to print the calibration curve, click on the **[File]** option on the menu bar. Select **[print]**, then **[exit to print the calibration curve]**. When you return to the worksheet, concentration values for each standard and sample will be displayed on the worksheet. To print out the worksheet, Click on the **[print]** button (the one with a picture of a printer on it) below the menu bar. When you're done, Click on the **[stop]** button (marked with a red square on the tool bar).

7) If you have more work to do on this worksheet, Click on the **[close]** button before exiting the program or opening another worksheet. If you're done working with this particular worksheet, you should save it to the hard drive using the **[archive]** function. While the worksheet is open, Click **[Samples]** on the menu bar, then select **[Archive]**. Enter a label for the worksheet so that you can identify it for future reference. You may also enter an optional operator name and description for the archive file. Click on the **[OK]** button. The worksheet will automatically be deleted from the method and stored in the archive file. If you do not archive the worksheets the program will use more memory since the worksheets are loaded into RAM with the program.

8) To retrieve a worksheet that has been archived, follow the procedure for starting a new worksheet. After the worksheet has been created, click on **[Samples]** on the menu bar, then select **[Unarchive]**. Select the worksheet you wish to retrieve and then click on the **[OK]** button.

9) If you are done with the worksheet but do not want to save it, you should delete it from memory by clicking on **[Worksheet]** on the menu bar, then select **[Delete worksheet]**. If the worksheet you wish to delete is not currently running, you'll have to bring it back up to delete it. It is important to either archive or delete worksheets once they are complete because building up too many worksheets may cause the software to exceed the memory requirements of your computer.

SECTION 15.1: Data Collection Without Aanalyze Package

If you don't need to generate a fancy report but only need to store the information on disk you can use any one of a number of shareware communication packages including Hyperterminal, Procomm, Telix, Crosstalk, etc. The data can then be imported into Microsoft Excel or the like. The procedure for Procomm will be explained here.

1) Connect the comm port at the back of the 210 to the serial port of the computer using a double female 9 pin serial cable wired as shown:

210 pin	**P-C pin**
5	5
2	3
3	2
7	8
8	7

2) Start the communications software. The default settings to match the 210 are:

4800 baud
8 bits
no parity
1 stop bit
rts/cts flow control (handshake routine)

SECTION 15.1: Data Collection Without Aanalyze Package *continued*

5) From the Procomm main page press the ***page down*** button on the pc the select option ***7*** for ***ascii download***. Enter a filename that you wish to save the report as. Usually a date or time serves as an identifier, such as 090499.prn.

6) Set up the 210 as normal except press the **[cntls]** button, then highlight ***remote communication controls*** and press **[enter]**. Change ***remote mode*** to ***library/report*** and make sure the settings are the same as mentioned in step 4 above then press **[esc].**

7) From the ***cntls*** menu highlight ***report and display controls*** and press **[enter]**. Turn ***read key reporting, read key report header*** and ***calibration reporting*** to on. The ***printer*** can be on or off depending if you wish an immediate hard copy or not. Under the ***cntls*** menu you can turn ***read key sampling*** on if you wish to use the sample identifiers page.

8) Calibrate your standards and run your samples as usual. The information will appear on the computer in the same format as on the printer.

9) When finished go back into the ***cntls*** screen and change ***remote communication controls*** back to off and return to ***active analysis***.

10) To process the ascii file in Excel, close the Procomm program by pressing ***esc*** and ***alt x*** and start the Excel program.

11) Select ***file*** then ***open*** and set ***files of type*** to ***text files/.prn.*** Browse thru the Procomm directory and select the data file you named above.

12) Excel will prompt you with the ***text import wizard*** then follow these steps. Highlight ***delimited*** and press ***next***, highlight ***tabs*** and press ***next*** then select ***general*** and ***finish***.

13) Edit the information as needed and save.

NOTE: Once the ***comm*** is active, you cannot change any analytical parameters until you finish/exit.

SECTION 15.2: Data Collection using Buck Analyst

The set-up program on the CD-ROM should automatically run, however it can be run using either the explorer or by using the Run command from the start menu. Please note carefully that the program was written to be backward compatible with Windows 95 and later. If Set-Up offers to replace a file that is newer than the corresponding file included with the software, you should click on Keep the Current File.

Press the **[cntls]** key on the Buck 210VGP, highlight **[remote communication]** then press **[enter]**. Use the right arrow key to move the cursor to the **"remote mode"** option, then use the **[right]** arrow key to change to **[library/report]** then **[enter],** select 9600 Baud and RTS-CTS Handshaking using **[left/right]** arrows then press **[enter]**, then **[esc]** twice to return to the main screen. Use the Instrument / Setup Comm Menu to select the correct communications port for your computer and the same Baud Rate setting as above.

The program will capture data from the Active Analysis Screen or from the Calibrate Screen. To get the software to capture data from the instrument click on the Capture menu selection in the Instrument menu then press START on the instrument. After the runs are finished click on Stop Capture in the Instrument Menu.

A description of the Menus is available in the Help File included with the software.

END OF MANUAL