BUCK SCIENTIFIC 410 Mercury Analyzer

OPERATOR'S MANUAL February 2008



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Buck Model Buck 410 Mercury Analyzer

INTRODUCTION

The Buck 410 Mercury Analyzer is designed to measure the concentration of elemental Mercury in solution. The instrument incorporates the Hatch and Ott Cold Vapor technique with a long 400mm path length flow cell for trace analysis of Mercury in drinking, surface and saline waters, as well as domestic and industrial wastes. It provides integrated measurements in absorbance intensity, as well as sample concentration in comparison to standard solutions. The absorbance readings are determined using peak area integration.

The Buck 410 Mercury Analyzer can be calibrated using up to 8 concentration values, with units of mg/L, $\mu g/L$, ppm, ppb, mEq/dL, mM/dL, $\mu g/dL$ 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 410VGP 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 intended for 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.

Neither this entire manual nor any part of it may be reproduced without the expressed consent of Buck Scientific, Inc.

Direct all inquiries regarding this manual and/or the Buck 410 Mercury Analyzer to your Buck Scientific Sales Representative or to:

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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 IP28; wide range general purpose, 190-930nm Lenses: Supracil - amorphous silica Optical Filter : 25.4mm Band Pass, 253.7nm center, 26nm FHWM

Operating Modes:

Absorbance: -0.0820 to 3.2000 Concentration: to 5 significant digits Integration Period: 0.5 to 25 seconds Integration Type: Peak Area Screen Refresh : 0.5 to 1.5 seconds Recorder Output: 1V/ABS (-0.08 to 3.2V) Background Correction: In-line Deuterium Arc

Hollow Cathode Lamp:

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.)

Cold Vapor Apparatus:

Flow Cell : 400mm with removable windows Reaction Vessel : 250mL Erlenmeyer Flask Bubbler: Coarse Fritted Dispersion Tube, 6mm septum, #8 stopper Drying Tube Filler: Lint free wipes Filter Tube Filler: Activated Charcoal Tubing: Peroxide cured silicone, 1/8" ID * ¼"OD

Carrier: External Argon, Air or Nitrogen Supply

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: $\leq \pm 5\%$

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)

Argon:

Argon is the preferred carrier gas for use with the Buck 410 Mercury Analyzer. It is often obtained in size 1A cylinders containing about 7000 liters (at STP). It is used with a CGA580 two-stage regulator (part no. BS303-0264). High purity grade (99.995%) Grade Argon is good for most analyses. Argon in the prepurified grade (99.998%) can also be used. Argon cylinders are used at high pressure and should be handled with care at all times.

Nitrogen:

Nitrogen may be used as an alternative to Argon. 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). 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.

Air:

Air is the most readily available carrier gas 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). Medical Grade Air may be used with a CGA-346 regulator set to 50 psi. General Purpose Compressed Air is also suitable instrument use. General purpose air cylindes are operated at 50psi utilizing a CGA590 two-stage regulator (part no. BS303-0264).

As an alternative, an oil-less air compressor may be 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).

SECTION 1: Installation - Preparing the Lab

This section gives details concerning the space and accessories required to set-up the Buck 410 Mercury Analyzer. The instrument is supplied with the following materials:

- 1. Nylon Gas Line for Carrier Gas
- 2. Bubbler Assembly
- 3. 250mL Erlenmeyer reaction flasks
- 4. Drying Tube and Carbon trap installed
- 5. Mercury Hollow Cathode Lamp
- 6. Spare septum and tubing

Equipment to be Provided by the Analyst

The following lists the equipment and materials that you will need to operate the 410 Mercury Analyzer. Many of these materials may have been supplied as options with your 410 Mercury Analyzer. They can also be purchased from your Buck Scientific Sales Representative, and are shown with part numbers for your convenience.

- 1. Standards, reagents & acids for the Cold Vapor Technique
- 2. One (110/120 v-15 amp) duplex outlet for instrument and printer if necessary.
- 3. Distilled or deionized water.
 - 4. 3' X 5' lab bench area.
 - 5. Drain Vessels for Waste fluids
- 6. Carrier gas supply

SECTION 1: Installation

Suitable Work Area

For best performance, the Buck 410 Mercury Analyzer should be located in a wellventilated room, free of dust, draughts, and corrosive fumes and vapors. The Buck 410 has a carbon trap to remove Mercury from the exhaust gas before exiting the instrument. If deemed necessary, a nipple is provided so the gases may be vented into a fume hood or outside. The exit tube should be kept as short as possible to avoid back pressure problems on the flow cell.

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 410 Mercury Analyzer Dimensions:

37"(94cm) W x 12" (28cm) D x 12" (28cm) H Minimum Rear Clearance: 4"

Recommended Table Dimensions: 48"W x 24"D x 30"H

The laboratory environment should be regulated to provide stable temperature and humidity. The 410 Mercury Analyzer should be maintained at temperatures from 10° to 32° C (50° - 90° F), and relative humidity of 30% to 80%.

SECTION 1: Installation *Continued*

In many applications data handling may be accomplished using an external printer, a 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 410 Mercury Analyzer, 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.

Ventilation

The Buck 410 Mercury Analyzer includes a Carbon Trap to catch most remaining Mercury Vapor and other potentially harmful substances so venting into the lab may be acceptable. However we cannot guarantee efficiency nor the life of the carbon trap. If you determine that the vent tube should be run outside or to a ventilation system, a nipple fitting has been provided to make the connection. The instrument should be located near the vent to minimize the effect of the vent tubing on your analysis.

Gas Connections

Using the 1/4" brass fittings, connect the BLACK nylon hose from the Argon, Nitrogen or Air supply to the AIR port on the back of the 410 Mercury Analyzer. Input pressure should be set for 30-100psi.

Electrical Connections

Plug the power cord into a standard outlet and connect the other end to the power adapter on the back of the 410 Mercury Analyzer unit.

International clients MUST check to see that the small voltage selector switch is set appropriately to 230 Volts if required.



Data Output Connections

There are optional connections which may be made to output data to a Printer, Computer or Chart Recorder.

- 1.) The analog output is a 0-1volt output for the absorbance data which may be connected to a strip chart recorder.
- 2.) The comm port is a 9 pin female RS-232 connection for a computer. The cable used should use the following configuration:



3.) A printer may be connected to the printer port using a standard PC printer cable. All dot matrix printers will work with the Buck 410 Mercury Analyzer. Some Ink Jet Printers will also work.

Hollow Cathode Lamp

For a new installation, gently insert the Mercury Hollow-Cathode Lamp into the Turret opening on the left-side of the Buck 410 Mercury Analyzer, and push in till the collar hits the turret mount. Rotate carefully, line up the Slot in the socket with the Key on the lamp and place the Power Plug on the end.

Bubbler Assembly

- 1.) Attach the Carrier Gas **Outlet** Tube to the **longer Glass Bubbler Tube** on the reaction flask Stopper assembly.
- 2.) Attach the **Sample Return** Tube to the shorter Glass Tube on the Stopper.

NOTE: Wet the Glass Tube if necessary to slide the Silicone Tubing into place.

Section 2: Setting Analysis Parameters--[Cntls] key

- 1) Pressing the [cntls] button displays the following menu:
- Note: Use the [left/right] arrows to change the selection then press [enter].
- **Display mode:** The options are **[absorbance] or [concentration].** This will automatically display concentration after running a calibration curve.
- **<u>Read-key Sampling:</u>** The options are **[on] or [off]**. Set to on allows you to print out sample identifiers from the **[smpls]** page for each sample read. 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 will advance the sample number by one each time. Pressing **[esc]** will abort the analysis.
- **<u>Read-Key Sampling Auto-Increment:</u>** 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.
- **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.
- <u>Measurement Controls</u>: Displays file information including Lamp Currents and Integration Time.
- **<u>Remote Communication Controls:</u>** 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.

<u>Report and Display Controls</u>: 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.

<u>Conc Display Precision:</u> 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.

<u>Read-Key Reporting</u>: The options are **[on] or [off].** Produces a printout of results when the read, Autozero or Reslope key is pressed.

<u>Read-Key Report Header:</u> The options are **[on] or [off].** Prints the file information at the top of the report.

<u>Calibration Reporting</u>: The options are **[on] or [off].** Prints calibration information during the calibration procedure as well as a summary of results at the end.

Printer: The options are **[on] or [off].** Activates the printer.

<u>Printer Page Eject:</u> 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 3.0: Setting 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 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 98 labeled as Std1-blank, position 99-100 labeled as Std2 STD3. These can be changed to any position you wish. You **must** identify a blank and standards in order to run a calibration. 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:** If you put a space in the first character or leave an entire blank line between your last sample ID and the start of the blank & standards on the samples table, the program end the report after all the samples are run.
- **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.
- **<u>Rename all Samples to Default:</u>** 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. In the *loc* column put in a sample # of the qc check standard or blank you wish to check. 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 & read*. 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. The average is printed after the next sample is read once.

Section 4: Reagents for Cold Vapor Technique

The following required Reagents must be prepared as noted below to meet the requirements for Federal and State guidelines for the Standard Methods for Mercury:

- Reducing Agent = 10% Stannous Chloride in 20% Hydrochloric Acid [10% SnCl2 in 20% HCl]. To prepare Place 50 grams fresh Stannous Chloride in a 250 ml beaker, add 100 ml. conc. HCl and ~5 grams mossy Tin or foil; swirl to dissolve SnCl2 crystals, warming gently if necessary. In a HDPE put approximately 300 ml of deionized/distilled water. Add the tin/acid solution to the water, being careful not to splash as the mossy tin is added to the bottle. Dilute this solution to a total volume of 500 ml. This "preserved" solution is STABLE for ~60 days.
- 2.) Acid Diluent = 60% Hydrochloric Acid + 10% Sulfuric Acid [6% HCl-1% H2SO4]. To prepare Measure ~250ml. DI water in a clean 1L HDPE bottle, and add 600ml. conc. Hydrochloric Acid; swirl to mix and chill to room temperature in refrigerator. Add 100ml. conc. Sulfuric Acid slowly with stirring (to avoid bumping); and dilute to 1L (top of bottle neck) when cool. Use a double-distilled grade of water for all dilutions, or a brand certified for trace environmental analyses. Use 10ml. for every 100-150ml. reaction solution used. The H2SO4 improves the reproducibility of the Mercury signal.
- 3.) Stock Standard = 10 PPB (ug/L) Mercury in 10% Hydrochloric Acid [10PPB Hg in 10% HCl]. Prepare FRESH 0.01mg/L Hg STANDARD in 10% HCl from 1000 mg/l stock (use 0.01ml/L or make TWO suitable Dilutions to reach this level, as desired).

Section 5 : Operation:

SETUP

- 1.) Press the POWER button on the right-side of the Buck 410 Mercury Analyzer to turn it ON. It is recommended to turn the system on ~30 minutes before any tests. Put in a lab area, no cooler than 60°F to avoid Hg vapor condensation.
- 2.) The drying tube must have fresh Kimwipes to block aerosol droplets, change monthly (~100 hours).
- 3.) Start any Printer or P-C, if used.
- 4.) Turn the carrier gas tank or compressor ON and set delivery pressure to 30psi .

NOTE: The peak window is set for approximately 60 seconds. If this window is too small or large to effectively catch the Mercury peak for your samples, you can adjust the timing using the following guidelines.

- A) Timing Parameters are on the measurement controls menu. Press <cntls> then highlight "Measurement Controls" and press <enter>.
- B) Change the "Data" parameter and the "Period" parameter will change to an appropriate value automatically.
- C) A change in the data parameter will increase the time for peak integration almost 10X (1sec. Increase for data parameter will increase time approximately 10sec.)
- D) Always reset Integration time to 1.5 sec. After changing parameters or the Autozero function will take too long.

CALIBRATION

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

Cup 98 99 100 0 0 0 0	Name Std1-Blank Std2-Max Std3	Cor 0.000 0.0000 0.0000	nc Abs-sec: 00 0.000000 0.000000 0.000000
0 0 0			

Vapor Calibration Form

1) Press the **[cal]** button. The screen will display the calibration form with cup 98 as std1- blank and 99-100 as std's 2&3.

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 not necessary to follow.

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 (section 3) **Note 5:** A cup number, name and concentration value must be present otherwise an error will result.

Press the **[down arrow]** to highlight 99 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 2 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 another standard type in a cup number less than 100 under Std 3 and press **[enter]**. The sample identifier will be brought over from the **[smpls]** page automatically.

2) Press the [cntls] button and select the *units* to report results in. 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. Select how many *standard replicates* to run. The rest of this form contains data from the last calibration for reference only. Press [esc] to return to calibration form.

- **3)** Have the prepared Standards and Reagent Blank and reducing agent ready before continuing.
- 4) Press [A/Z] to zero the absorbance reading.

Section 5 : Operation/Calibration Continued:

- 5) Fill the Flask with 100ml. of your High standard and add 10ml. of Acid Diluent.
- 6) Place Bubbler / Stopper Assembly on Flask.
- 7) Highlight the Cup 99 line for the High standard, press the [READ] key and follow the directions on screen by injecting 5ml of the SnCl₂ Reducing Agent into the Flask. Swirl for a ~15-20 seconds to initiate the reaction and press the Green [START] button on the 410 gas control panel (on the left side of the unit). The Integration Screen will appear and show you the Mercury Peak.

NOTE1: Do not press [ENTER] after injecting the reducing agent as the screen prompt states since the enter function is preformed by the gas control panel to ensure consistent timing between the gas flow coming on and the peak integration commencing.

NOTE2: Adjust Carrier GAS Flow to MAXIMIZE the evolution of the Peak.

- 8) After the Integration is complete, remove the Bubbler Stopper from the Reaction Flask, rinse with DI Water from a wash-bottle and place into a CLEAN, EMPTY Flask. Press the White [PURGE] button to rapidly flush out any residual Mercury vapor until ABS reading on the screen drops to near zero again.
- 9) Press the Red [STOP] button on the gas control panel to shut off the gas.
- **10)** Repeat Steps 3-9 using the Sample Prep Reagent BLANK, highlighting the Cup 98 line for the Blank.
- 11) After all standards and the blank have been run, 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 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 the **[read]** button to update the absorbance reading then recalculate.

- **12)** After you've viewed a satisfactory curve, press [esc] twice to return to *active analysis* where you can now read your samples.
- **Note:** 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.

Section 5 : Operation/Analyzing Samples

- 1) Press **[A/Z]** to zero the absorbance reading.
- 2) Fill the Flask with 100ml. of your prepared sample and add 10ml. of Acid Diluent.
- 3) Place Bubbler / Stopper Assembly on Flask.
- 4) Press the [READ] key and follow the directions on screen (Enter sample identifier if you are using "read key sampling") by injecting 5ml of the SnCl₂ Reducing Agent into the Flask. Swirl for a ~15-20 seconds to initiate the reaction and press the Green [START] button on the 410 gas control panel (on the left side of the unit). The Integration Screen will appear and show you the Mercury Peak.
- 5) After the Integration is complete, remove the Bubbler Stopper from the Reaction Flask, rinse with DI Water from a wash-bottle and place into a CLEAN, EMPTY Flask. Press the White [PURGE] button to rapidly flush out any residual Mercury vapor until ABS reading on the screen drops to near zero again.
- 6) Press the Red [STOP] button on the gas control panel to shut off the gas.
- 7) If you are using sample IDs, press the **[UP]** arrow key to advance the sample ID and repeat Steps 1-7 for the rest of your samples.
- 8) Press **[ESC]** to end the report.

Section 6: 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;* The library name is limited to 16 characters. You MUST change the library name before you can save changes. The Library name can also be changed on the *LIB* menu.
- 2) *Method;* Only the Cold Vapor/Hydride Method may be used with the Buck 410.
- 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.
- *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;* May be increased to expand peak window. (see guidelines in section 5)
- 7) *Period;* May be increased to expand peak window. (see guidelines in section 5)
- 8) Integration Time; Effects only Autozero. Set to 1.5s for best Results.
- 9) *Maximum Peak Timing;* Sets the length of the peak window. Set to 28 seconds for Cold Vapor Analyses

- 10) *Background Gain;* Preset for best results. Automatically set with Autozero from the align screen.
- 11) D2 Background; Preset ON for elements which can make use of it.
- 12) *Wavelength, Slit;* Not Applicable to Buck 410 Mercury Analyzer.
- 13) *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 : Hg_CV_253.7). 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: 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

Something is blocking the energy from the lamp. This could be caused by dirty windows on the flow cell and/or dirty lenses

VERY LOW ENERGY or NO ENERGY

- 1) 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.
- 2) 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.

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.

Section 6: Error Messages, Continued

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.

SECTION 7: 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.
- 9) This will load default libraries into each lamp position. You should now load the Hg_CV_253.7 library into the 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.

SECTION 8 Replacement Part Assembly Instructions

SECTION 8.1: Hg Hollow Cathode Lamp Replacement Instructions

- 1) Unplug lamp socket and slide the lamp out from the left side of the Buck 410 Mercury analyzer,
- 2) Install the new Mercury lamp (Buck Part # 4134) in the holder and slide it in until the metal collar contacts the instrument's side plate.
- 3) Line up the bump on the lamp with the notch in the Lamp connector of the Buck 410. Plug in the lamp connector.
- **4)** Press [align] to display the bargraphs.

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

NORMALIZED ENERGY



5) 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. On the left side of the instrument are 2 knobs for lamp alignment (rear knob for horizontal, bottom 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. If the bar graph goes off screen to the right press [align] to center it. Repeat this procedure with the other knob 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. The lamp should give you a energy of at least 4.0. If you have trouble aligning the lamp, press [Bkgnd] to turn the background lamp off and try again. Press [A/Z] when finished.

SECTION 8.2: 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 410.
- 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 8.3: 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.

SECTION 8.3: Removing the electronics modules, Continued 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.
- 4) Remove all screws holding the board to the panel.
- 5) 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.

SECTION 8.3: Removing the electronics modules, Continued

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 preamp assembly can be taken out.

Part Number	Description	
AO-HG	Mercury Standard Solution	
400-0100	Aerator (bubbler) Assembly	
400-0200	Flask 250ml, pkg 12	
410-0006	Carbon Trap	
400-0400	Drying Tube	
400-0500	Silicone Tubing 1/4" OD (7ft.)	
410-1002	400mm Absorption Tube	
410-1003	Cell End Caps	
BS006-6549	Quartz Windows	
990-1543	O-rings (2)	
220-1052	Window Holding Ring	
410-1004	Support, Cell	
4134	Mercury Lamp	
BS290-1180	Photo Detector Hamamatsu IP-28	
400-1200	10cc Plastic Syringe (pkg. of 10)	
410-1027	PCB, Gas Control	
	Aerator Replacement Parts	
400-0101	Filter Tube (3-1/8")	
400-0103	Pyrex Lube (3")	
400-0105	Septum Socket (2-1/4")	
400-0106	Septum Pack of 5	
400-0107	3-Hole Silicone Stopper	

Section 10: Maintainance

Flow Cell

The flow cell is designed for easy cleaning. To remove the cell disconnect the tubing and pull the cell upwards by the end caps. On the bottom of the cell support there is two screws you can loosen to allow the end caps to be removed by sliding the outward making it easy to clean the cell and end caps. You may clean both the cell and windows with a mild soapy water solution. Be sure to wipe the windows dry so no residue is leftover. To remove buildup, you may use a 2% HCL solution to soak the tube and end caps.

Drying Tube

The Kim Wipes in the drying tube should be replaced on a monthly basis or as necessary.

Carbon Trap

The carbon trap should be replaced as necessary. Since the amount of Mercury built up in the trap will vary depending on the concentration of Mercury in your samples as well as hours of use, It is up to the user to determine how often it should be replaced. The trap should be disposed of properly as **Hazardous Waste**.

Bubbler

The septum should be replaced when it becomes to perforated to provide a good seal. The gas dispersion tube should be replaced if ot is broken, clogged or contaminated.