

PROCEDURES OF THE SIMAA 6000 (Perkin Elmer Verison 2.5 AA WinLab) SIMULTANEOUS MULTI-ELEMENT GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROPHOTOMETER(AA)

PREPARING THE AA INSTRUMENT FOR GSR ANALYSIS.

Check Argon Gas cylinder for adequate amount of gas to run samples.

Next, dump waste water container(right container, as you face machine). Refill De-ionized water container (left bottle, as you face machine).

Power up Printer, Computer and AA. Start the Calibration Log sheet for AA.

Removing previous Data files

Go to the **SIMAA 6000** window, double click on **File Manager Icon** → **File Manager** window opens
Click on “C” drive

Find and access the directory by clicking on the directory → **AAUSER** → clicking on the directory
→ **SAMPLEINFO (C:\AAUSER\SAMPLE INFO*.*)**

Then Select the following type files “PBBAGSR1/PBBAGSR.SIF , SBGSR1/SBGSR.SIF ie any file with the extension of *.sif”

After Selecting the file(s) → Delete these files Selected by clicking on the → **Delete**, In the **File** menu → or use the keyboard **Delete** button. After the completion of this process, **Close** → **File Manager**

Starting the AA Software and Selecting the Method

Go to the **SIMAA 6000** window → double click on **AAWinlab Analyst Icon** →
AAWinlab Analyst window opens → Click on **Use a Custom Design Workspace** button → Open window
(C:\aausers\labprocs) opens → Select the **CFM.frn** → click on **OK**

SIMAA 6000 Work Screen opens with the following sub-windows: **Automated Analysis, Calibrations, Peaks and Results.**

Note: All computer work done from this point on is under the SIMAA 6000 Work Screen.

Clearing and Re-Setting the Library Files

In the **File** menu, click on → **Utilities**

In the **Utilities** sub-menu, click on → **Library Manager** → **Winlab Library** Window opens

Select all files as individuals or as a group to removed → Click on the **Delete Icon** → **Winlab** window opens →
Click on → **OK**

Click on → **File** menu in the Winlab Library Window

Click on → **Pack Library** → Winlab window opens → click on **OK**

Winlab Library Window's Tool bar click on → **Check Icon** → Winlab window opens → click on **OK**

Close → **Winlab Library** Window

Setting up the AA Data Files to run Antimony in GSR

On the Tool bar for AAWinlab Analyst window → Click on **Sample Information** icon → **Sample Information Editor** window opens

Click into the **Description field** → Enter the following information → SBGSR1 or SBGSR

Click into the **Batch ID field** → enter the following information → SB1

Click into the **Analyst Field** → enter the following information → your name or initials

Click into the **Sample ID at Position #1** (use arrows beside number pad. This is to setup auto-sampler location enter the subjects name and sample information(Control-C), such as → “Smith” C

proceed to the #2 location and follow the same procedure for sample location #1

“Smith” RB, “Smith” LB, “Smith” RP, “Smith” LP.

At the completion of the naming of the sample locations (#1- #70 or #75)

(Note: Use the queue sheet prepared during kit preparation that parrots the above data description and location.)

Go to → **File** menu

Click on → **Save AS** → Select → **Sample Info File** → type in the name the file “SBGSR1” or “SBGSR”(do not need to add extension) → Click on → **OK** (C:\aauser\sampleinfo)

Setting up the AA Data Files to run Barium/Lead in GSR

(Note :You must change Description, Batch ID, & Dilution.)

Click into the **Description field** → Enter the following information → “PBBAGSR1 or PBBAGSR”

Click into the **Batch ID field** → Enter the following information → PBBA1

Double Click into **User Dilution Field** in sample location #1 in the →

Dilution Column Fill Window opens

Set **Dilution Vale** → 20.00

Under Location Range

Enter the **Start location** → 1 or first sample to be run

Enter the **End location** → 70 or last sample to be run(up to 75)

Click on → **OK**

note: make sure dilution setting is correct for all sample locations.

Go to → **File** menu

Click on → **Save AS** → Select **Sample Info File** → Enter the name the file PBBAGSR or PBBAGSR1 (do not need to add extension) → Click on → **OK**

Close → **Sample Information** window

Note: Does not matter if you activate the Lamps or Furnace first.

Activating Lamps

On the Tool bar for SIMAA 6000 window → Click on → **Lamp** Icon → Lamp window opens

Click on → drop down menu for the **Beam Combiner** →

Select → **2 Lamp Mode**(loc. 2,3) for Ba/Pb Lamp

Click on → **ON/OFF** Icon for **Each Lamp**

Visually check Beam → Circular beam of light (Note: You should see a circular red light for Pb in the Hollow Cathode Lamp for Lead. You should see a circular blue light for Ba in the Hollow Cathode Lamp for Barium.&

Click on → **Activate** button → listen for the beam combiner to activate and set-up

The Activate button will un hi-light → Click on → **Activate** button → Close → **Lamp** window

Note: You should allow a minium of ½ hour of warm up after activating the lamps before starting a AA run.

Activating Furnace

On the Tool bar for SIMAA 6000 window → Click on → **Furnace Icon** → **Furnace Control** window opens
Click on → **Open\Close** button → WinLab window opens

Now to **Change the Graphite Tube by Manually** by loosening lock screw near bottles → allows the auto-sampler to swing free.

[Note: tube - Transverse Heated Graphite Atomizer(T.H.G.A.)-Part # for P E is BO 504033(BO 508884)].

Slide the auto-sampler out, swing support lever out of the way, and lower the front contact housing.

Use the PE clothes pin to remove old graphite tube.

Before replacing tube, clean the contacts with Q-tips + de-ionized water. Use one end of q-tips that has been moistened to clean and the other end to dry. Manually place new Graphite tube with use PE clothes pin to handle the new tube. The flange that is present in the area of the L'vov platform is to your left as you place the new tube into the AA with the hole up in the tube. Reset the front contact housing and support lever.

Reset auto-sampler and re-tighten lock screw.

Click on → **OK** → Click on → **Open\Close** button(listen for Click) → Click on **Condition tube** button (9 step takes 10-15 minutes).

Close → **Furnace Control** window

Sample Preparation

Prepare samples and load them into auto-sampler tray marked Pb/Ba and separate tray marked Sb.

Calibration Standard

Fill AA large sample cup labeled zero with 5% nitric acid to fill line. AA large sample cup labeled # 77 (location in auto-sampler) is filled 3/4 to volume with the prepared 100 % GSR calibration standard. AA large sample cup Labeled #80 is a 50% dilution of GSR calibration standard. Place the AA large sample cups 77 and 80 into auto-sample tray in corresponding locations on the tray.

Running Zero Blank and Verification of Pipette Function

Maximize the Automated Analysis window

Click on → **Analyze** tab

Click on → **Select Location** button → enter Zero(0)

Click on → **Analyze Sample** button with green dot → **Stopping an Analytical Sequence** window opens

Click on → **Complete Current Replicate**(1 run of Selected location) or **Complete all Replicates for Current Sample** (2 runs of Selected location)

Click on → **OK** → Check on pipette function by doing the following steps: When the computer screen reads → **AGC (Automatic Gain Control)** → get mirror ready to verify pipette's function correctly.

As soon as the pipette arm drops → drop the mirror → you should see pink circle with white tip entering the pink circle and dispensing the liquid. Remove mirror as soon as arm starts to re-set. The liquid should bubble off!

Setting up AA for Pb/Ba-GSR to Start Analysis

Automated Analysis window still Maximized

Click on → **Setup** tab

Click on → **Browse** button for **Sample Information File** → Open window opens

Select PBBAGSR(1).SIF → Click on → **OK**

Click on → **Browse** button for **Results Data Set Name** → **Choose Results Data Set** window opens

Click into → **Field Result Name** → Enter the file name, such as 010103pb or 010103ba for Jan. 1, 2003 ie XXXXXXpb or XXXXXXba → Click OK (which will save an electronic copy of the data)

Select → **Lamp** box for Off After Analysis

Select → **Print Log** box for During Analysis

Select → **Use entire sample into file**

Load auto-sampler tray marked Pb/Ba + 77 & 80 cup.

Click on → **Analyze** tab

Click on → **Analysis All** button (note: lamps to auto shut off & print data)

Check log energy counts after about 1 hour into run. Record calibration information in AA log book for Standards 1,2,3 & QC.

Setting up AA for Sb-GSR to Start Analysis

Activating Sb Lamp

On the Tool bar for SIMAA 6000 window → Click on → **Lamp** Icon → **Lamp** window opens

Click on → drop down menu for the **Beam Combiner** →

Select → **1 lamp mode**(loc. 1) for Sb Lamp

Click on → **ON/OFF** Icon for each lamp

Visually check Beam → Circular beam of light (Note: You should see a circular red light for Sb in the Hollow Cathode Lamp for Antimony.)

Click on → **Activate** button → listen for the beam combiner to activate and set-up

The Activate button will un hi-light → Click on → → button → Close → **Lamp** window

Note: The Energy counts will be low during the warming up period. You should allow a minium of ½ hour of warm up after activating the lamps before starting a AA run.

Automated Analysis window still maximized

Click on → **Setup** tab

Double Clicking on → **PBBAGSR** in the Method box → Open Method (C:\aausers\methods\) window opens
→ Select **SBGSR** → Click on → **OK**

Click on → **Browse** button for **Sample Information File** → Open window opens

Select → **SBGSR(1).SIF** → Click on → **OK**

Click on → **Browse** button for **Results Data Set Name** → Choose Results Data Set window opens

Click into → **Field Result Name** → Enter the file name, such as 010103sb for Jan. 1, 2003 ie XXXXXXsb
→ Click OK (which will save an electronic copy of the data)

Select → **Lamp** box for Off After Analysis

Select → **Print** Log box for During Analysis

Select → **Use Entire Sample into File**

Replace auto-sample tray Pb/Ba for Sb tray (switch out cup 77/80); Record energy count in AA log book.

Saving data to Excel File Format

To Save files to diskette(A:*.*) for data entry(excel formatted files will be →*.csv)

SIMAA 6000 window →Click on → **File** in the menu → Select → **Utilities** →

Select → **Reformat** →Winlab Reformat Data window opens

On the tool bar Click on → **Open Design** button →Open Reformat Design window opens → Select **Test**

→Click on → **OK** →Click on → **Browse** button for **Data Set Name**→Select one file at a time (either pb/ba or sb)→Click on → **OK** → Click into → field for **Reformatted File Name** → enter the file name to be saved as

→xxxxxxba\xxxxxxpb or xxxxxxsb →On the tool bar for **Winlab Reformat Data** →Click on → **Save Results** button →Winlab window opens →Click on → **OK**

Repeat process for all files needed to be converted to Excel files.

Note: Saving the file should begin with date first & then corresponding element IE Jan. 1, 2003 on PbBa run.....

“010103pb” or “010103ba”→ PbBa run. Choose either Pb or Ba at the end of the file. Since, these two elements are always run together. Repeat the same process for Sb run..... “010103sb”

Close → **Winlab Reformat Data** window → Winlab window opens → Click on → **OK**

Close → **SIMAA 6000** window

Close → **Program Manager**

Power down → Computer and AA.

Keys:

Control = C

Right Back = RB

Left Back = LB

Right Palm = RP

Left Palm = LP