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Author: <b>Miguel A. Santiago Rivera</b>				

**UPR-NSF Earth X-ray Analysis Center (EXACT!)**  
**Standard Operating Procedure**  
for:

**Operating the Siemens XRF, SRS  
303 Sequential X- Ray Spectrometer**



**UPR-NSF Earth X-ray Analysis Center (EXACT!)**  
X-Ray LABORATORY

DEPARTMENT OF GEOLOGY  
F- 304 Geochemistry Facilities at Physics Building  
University of Puerto Rico – Mayagüez Campus

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### Revision Log

Revision No.	Effective Date	Prepared By	Description of Changes	Affected Pages
R-0	August 17, 2006	Miguel Santiago	New Procedure	All
R-1	March 26, 2007	Miguel Santiago	X-rays Warning for Pregnant and Cancer patient 8.0 - Procedure	One Page - 8

# Operating the Siemens SRS-303

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# Operating the Siemens SRS-303

## 1.0 PURPOSE

This procedure provides instructions for the operation of the Siemens Instruments, incorporated, Model SRS-303, x-ray fluorescence system.

## 2.0 SCOPE

This SOP is a mandatory document and shall be implemented by all Researcher, Faculty member, Technician or students participants when using the XRF SRS-303 for the collection of the XRF data.

## 3.0 TRAINING

3.1 All users of this SOP are trained by reading the procedure, and the training is documented in accordance with the GLP's.

3.2 The Geology Task Leader will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with GLP's.

## 4.0 DEFINITIONS

- 4.1 *SOP* - Standard Operational Procedure
- 4.2 *GLP's* - Good Laboratories Practices
- 4.3 *Machine Custodian* — The Machine Custodian is responsible for Siemens Diffraktometer D5000 maintenance and User instruction. This includes calibration, basic repairs, and software, data taken and electronically-stored system backups, and all instruction and training of Procedure Users.
- 4.4 *XRF* – X ray fluorescence
- 4.5 *SRS 303* — SRS 303 is the model name of the XRF manufactured by Siemens, Incorporated (purchase date month/year, Siemens Service Serial # 001332).
- 4.6 *XRF, Siemens SRS 303 system* — The system includes a chiller to control the temperature, Drifac Plus software and PC to collect the data.

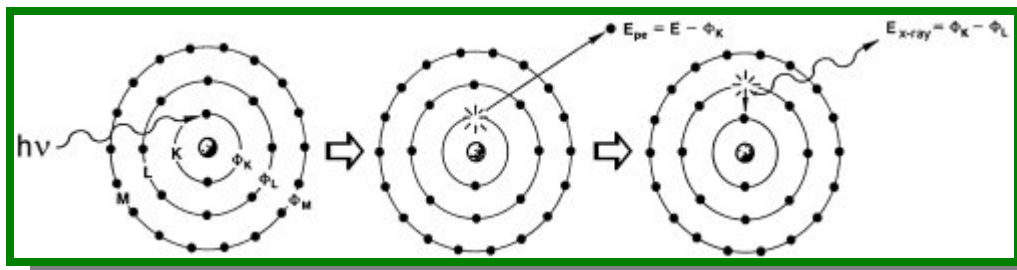
## 5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

- 5.1 Scientific Instrumentation Specialist
- 5.2 Faculty Member
- 5.3 Researcher
- 5.4 Student Participant

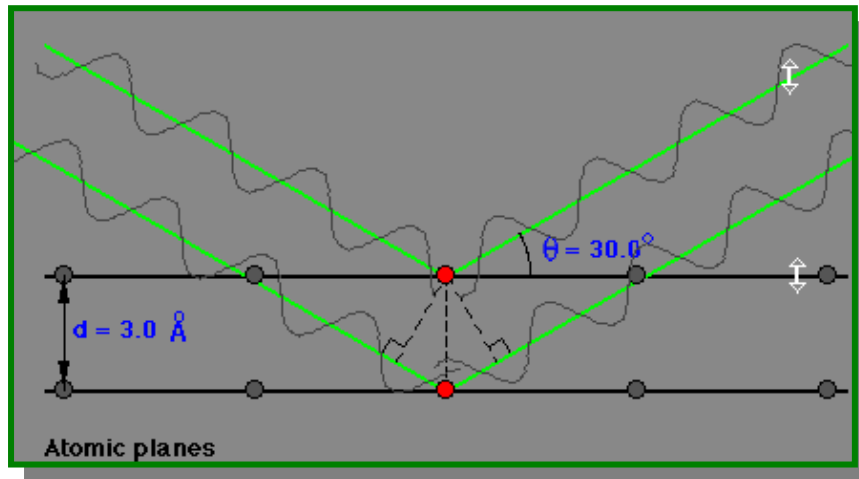
## 6.0 THEORETICAL BACKGROUND

6.1 *XRF Theory* - An electron can be ejected from its atomic orbital by the absorption of a light wave (photon) of sufficient energy. The energy of the photon ( $h\nu$ ) must be greater than the energy with which the electron is bound to the nucleus of the atom. When an inner orbital electron is ejected from an atom, an electron from a higher energy level orbital will transfer into the vacant lower energy orbital. During this transition a photon may be emitted from the atom. This fluorescent light is called the characteristic X-ray of the element. The energy of the emitted photon will be equal to the difference in energies between the two orbitals occupied by the electron making the transition. Due to the fact that the energy difference between two specific orbital shells, in a given element, is always the same (i.e., characteristic of a particular element), the photon emitted when an electron moves between these two levels will always have the same energy.



Therefore, by determining the energy (wavelength) of the X-ray light (photons) emitted by a particular element, it is possible to determine the identity of that element. For a particular energy (wavelength) of fluorescent light emitted by an element, the number of photons per unit time (generally referred to as peak intensity or count rate) is related to the amount of that analyzed in the sample. The counting rates for all detectable elements within a sample are usually calculated by counting, for a set time, the number of photons that are detected for the various analytes "characteristic" X-ray energy lines. Therefore, by determining the energy of the X-ray peaks in a sample's spectrum, and by calculating the count rate of the various elemental peaks, it is possible to qualitatively establish the elemental composition of the sample and to quantitatively measure the concentration of these elements.

6.2 *Bragg's Law* - In 1914 Bragg developed a law, which defines a diffraction relationship between the wavelength of an incoming ray and the d-spacing of a diffracting crystal.



Bragg's Law:

$$n \lambda = 2d \sin\theta$$

where;  $n$  = the order of reflection

$\lambda$  = wavelength of incident ray

$d$  = interplanar spacing of the crystal

$\theta$  = angle of incidence and reflection of incident ray

This is the principle under which the wavelength-dispersive detectors operate.

## 7.0 EQUIPMENT

A description of equipment constituting the XRF SRS Wavelength Dispersive X-ray Spectrometer is providing below.

7.1 *XRF Siemens SRS 303 system* — The Siemens SRS-303, Wavelength Dispersive X ray Spectrometer instrument has capable of running solid (pellets) and liquid samples in Helium mode, Includes computer, software for data collection, which is performing under computer control using the "Spectra AT/3000" software application and options: Includes Sample Changer to fit 10 positions to samples. , Filters (4 positions), 1) copper, 2) aluminum, 3) aluminum, 4) open, Collimators ( 2 positions): 1) 0.15 degrees, 2) 0.46 degrees; Masks (3 positions): 1) open, 2) 30 mm, 4) 23 mm; Crystals ( 6 positions): 1) LiF 110, 2) LiF 100, 3) PET, 4) open, 5) open, 6) open; K730 generator (computer controlled), Rhodium X ray tube, 2 vacuum pump system, Air stabilizer.)

7.2 *Principle of Functioning* - When the matter is bombarded with appropriate energetic X-ray photons, characteristics radiations of same nature are emitted by its chemical elements. This is X-ray fluorescence phenomena. Emitted X-rays are first collimated and then selectively separated on the analysing crystal by diffraction (application of Bragg's relationship). Number of emerging X-ray photons are measured with a proportional detector. Corrected intensities from inter-element effects are linearly correlated with certified concentrations of reference materials.

## 8.0 PROCEDURE

**Note:** All personnel performing work under the UPR-NSF Earth X-ray Analysis Center (EXACT!) quality program may follow this standard operating procedure (SOP) for XRF analysis. May use their own procedure(s) as long as the substitute, only if, meets the minimum requirements prescribed by the UPR-NSF Earth X-ray Analysis Center (EXACT!) Plan or are better in the performing, and have been approved by the Director of the Department of Geology, UPR-Mayagüez Campus, before the commencement of the activitie(s).

**Note:** UPR-NSF Earth X-ray Analysis Center (EXACT!) personnel may produce paper copies of this procedure printed from the controlled-document electronic file. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear. Deviations from SOPs are made in accordance with Department Director approval and documented in accordance with the GLP's.

**WARNING:** X-rays equipments may produce radiation out of the equipment. Pregnant may risk in injury to the unborn child and/or may interfere with the treatment for cancer patient. If you are pregnant or cancer patient, consult your physician before to use and/or be exposed to x-rays equipments.

8.1 *Introduction* — The XRF Siemens SRS-303 instrument (Purchase Date: month/year, Siemens Service Serial Number: 001332 and UPRM-Property Number: 059925) allows elemental qualitative and quantitative chemical analyses of solid down to ppm level. Large sample cups are used to obtain the highest accuracy and lowest detection level. The operation of the S-303 is completely computer controlled, and samples can be analyzed either by using known standards or by using the theory of fundamental properties. Wavelength X-ray fluorescence spectrometry (WD-XRF) is a powerful multi-elemental and non-destructive analytical technique. This method allows to determine with accuracy the global chemical composition of a solid sample, in major elements and in trace elements at the  $\mu\text{g/g}^{-1}$  level. Its domain of investigation lies from Boron (N=5) to Uranium (N=92). It presents following advantages, simple sample preparation, rapid analysis, good reproducibility and low cost in use.

8.2 *Sample Preparation* - Samples to be examined with the XRF machine must be in a pellet form or size that can be inserted into, or attached to, an SRS-303 stage mount. Quantitative microanalysis routines assume that all samples for analysis will be relatively flat at the top of the surface and that analysis sites will be normal to beam incidence. To prepare the pellet sample, find the materials in the glass window cabinet at the UPR-NSF Earth X-ray Analysis Center (EXACT), F-304-C and use it at the press in the Preparation Area in F-305.





8.3 *Selection of Standards* - The Procedure User may employ standards traceable to National Institute of Standards and Technology (NIST, formerly National Bureau of Standards, NBS) or well-characterized materials published in credible technical journals and widely used by microanalysts or any "standard" that the Researchers brings for their own purposes for calibration of quantitative X-Ray analysis routines. Procedure Users may use standards from other sources for specific applications but must document the basis for usage of these standards in their notebooks. Specially, these one, should not be touched by naked hands.

8.4 *Sample Control* – Sample identification will be based on the unique identifier marked on the sample.

8.5 *Safety Considerations* - Normal operating conditions as performed by trained Procedure Users present no safety hazards.

8.5.1 *Electrical Power* - The SRS 303 system requires two separates electrical lines, one for the spectrometer and one for the generator. The spectrometer feed may range from 208V to 230V and requires a 20 amp fuse. The generator/deionizer feed from 208V to 240V through a 40 amp fuse. An isolated 20 amp, 110-120 VAC line services the computer and related peripherals

In addition, connect the system chassis ground directly to the grounding terminal at the service entrance. To limit noise on the conductors, only the spectrometer system may use this ground. The ground resistance must measure less than 0.5 ohms. Install the connection within 3.5m (12ft) of the rear of the spectrometer module.

Connect the power from the building mains switch box near the system. The power connections are made at the rear of the cabinet on the right side when viewed from the front.

Note: wire the room Air Conditioning or any external pumps and compressors to a different power line than is used for the instrument. High starting currents from their motors could adversely affect line voltage stability.

8.5.2 *Plumbing Requirements* – Provide a source of filtered cold water and a drain. The open drain should be installed close the system because a long hose length reduces the effectiveness of the aspirator.

The cooling water should be virtually free of suspended matter. If the water is badly polluted, install two water filters in a parallel arrangement so that the system can operate while one filter is cleaned.

8.5.3 *Temperature Controller Drain* - The spectrometer contains a built in temperature controller. Provide a drain or evaporator tray to eliminate the water condensation run-off. The drain must be at floor level; the drain hose must have a downward gradient.

8.5.4 *Compressed Air Supply* - Must install a compressed air connection free of shocks, water and oil and matching the following specifications:

- 20 liters/min (0.7ft<sup>3</sup>/min)
- 4.5-10 bar pressure (approx.)

If compressed air supply is not available, use a compressor of sufficient capacity (60 liters (2ft<sup>3</sup>) / min) at an operating pressure of 6 bar. Install the compressor in a separate room from the spectrometer.

8.5.5 *Vacuum Device* - The vacuum pumps sit within the spectrometer and are equipped with mist eliminators. An ideal installation would include venting the port to the outside, but this is generally not required.

8.5.6 *Counter Tube Gas Supply* – Install a bottle of a gas for the flow counter, mounted with two-stage regulator. Stabilize the gas flow through the system by using a fine regulator capable of setting pressure of 500 mbar (7 psi). Make sure the bottle is securely mounted to avoid tipping. Observe all safety regulation for flammable gases. Also, keep a second bottle in reserve. Keep the supply line for the gas bottle to the spectrometer relatively short and only use viton tubing. For longer gas supply line, use refrigerant grade copper or stainless steel tubing. The lines must be clean, free of water, solvents and particulate matter. Avoid running the gas lines near a heat source or subjecting them to any excessive temperature.

8.5.7 *Helium Supply* – For equipment using Helium flush, install a helium bottle with a double pressure 1.6 bar (20-psi) regulator. Secure the bottle with mounting support to avoid accidental tipping.

## **8.6 Performing the Standard Operation Procedure for XRF Siemens SRS 303 Sequential X-Ray Spectrometer**

8.6.1 *Air Compressor* – A 60-gal and 5-hp Craftsman (Sears Brand), which is located in front of F-305 at the balcony / hall of the third floor of Physics building.

- Recheck compressor wiring. Make sure wires are secure at all terminal connection. Free all contacts of loose wire cuttings, etc.
- Position the fuse disconnect or circuit breaker to the ON position and, if equipped, turn the ON/Auto-OFF switch on the pressure switch to the ON position.
- Open the air outlet valve fully to permit air to escape and prevent air pressure build up in the tank during the break in period.
- Run the compressor for 30 minutes. Make sure the air outlet, or globe valve, is open and there is no tank pressure build up.
- Check all air line fittings and connections / piping for air leaks by applying a soap solution. Correct as necessary. Even minor leaks can cause the air compressor overwork, resulting in premature breakdown or inadequate performance.
- Check for excessive vibration and noise. Adjust air compressor belt guard as necessary to eliminate chatter. Re-adjust or shim the air compressor feet, if necessary for proper level.
- Close the outlet valve and let air compressor pump up to “cut-out pressure”. Turn the air compressor off and check oil level. Add oil if necessary. Connect air hose to air outlet adapter.
- Now, the compressor is ready for use.

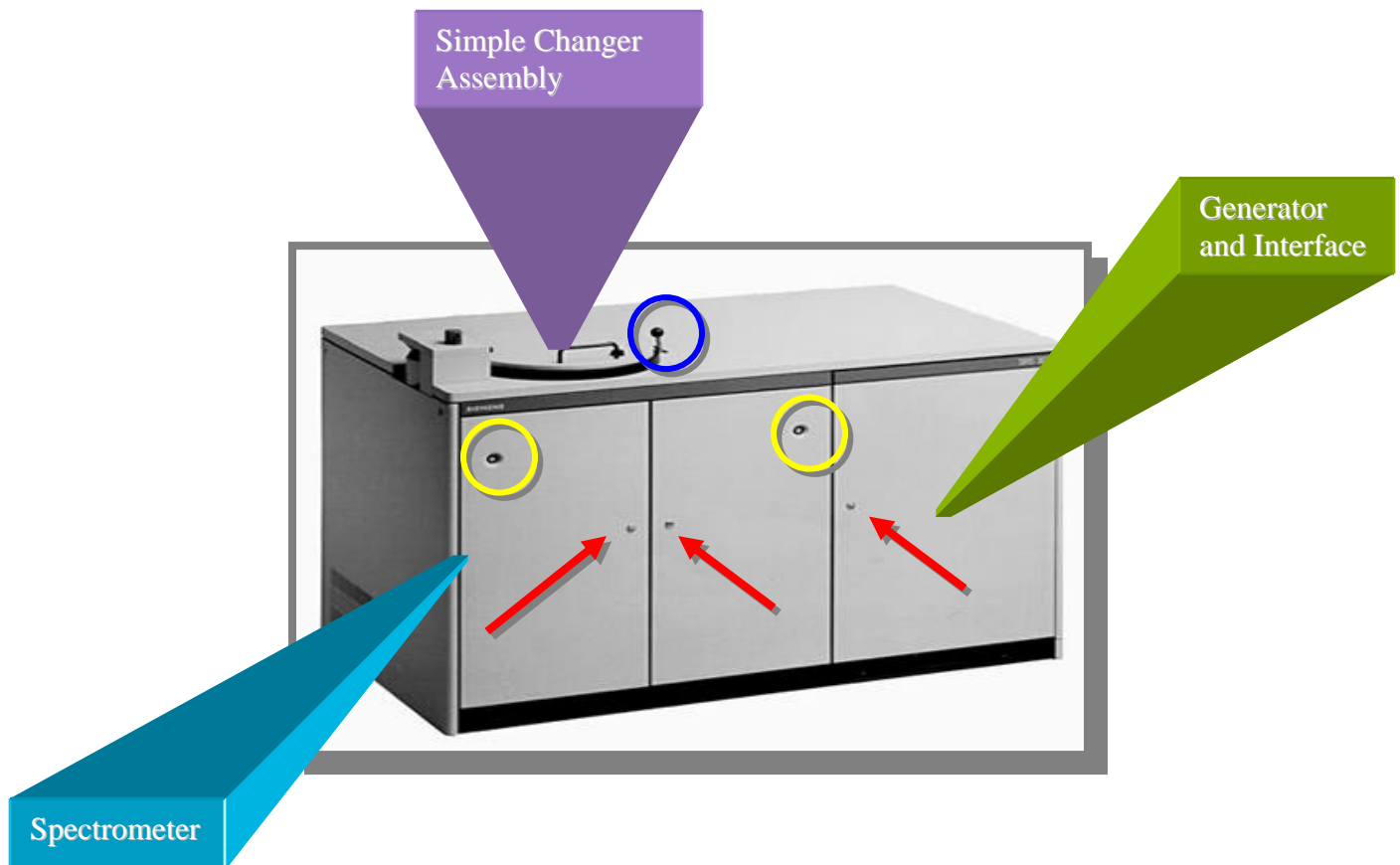


8.6.2 *Environmental Considerations* – Maintain the room temperature between 17°C (62°F) and 26°C (78°F), at a gradient less than 0.5°C/hr and a fluctuation of no greater than 2°C. Relative humidity should not exceed 80% and should be no less than 20%. Ensure that the heat from the generator, spectrometer, computer and vacuum pump is adequately dissipated by ventilation or air conditioning and located so that the airflow is not directed at the instrument. A clean, dust-free environment is also necessary. Avoid the direct sunlight to XRF machine.

8.6.3 *Chiller* - must be turn on 5 to 10 minutes before of the operating the XRF machine at pressure of 60 – 80 psi and a temperature of 65 F.

8.6.4 In front of the machine, unlock the three front doors with their own key to access the panels, see in the figure below with the red arrows ( → ).

8.6.5 In the left side of the XRF machine, turn on the switch of the Spectrometer at 23 psi, see in the figure.



8.6.6 In the right side of the XRF machine, the Generator and the Interface, see in the figure.

- Turn on the **green** switch of ON and OFF.
- Turn the two switches of the cards SC and FC
- Turn the key, in the X-Ray section.
- Press the **yellow** button, in the X-Ray section.
- Wait for a while a **red** indicator switch to **yellow**, in the Water Level Section. The indicator of water Level must be less than 50% after turned the compressor on. **Note:** This step take a some time to reach the level desired, after d the compressor was on.
- Then, press the **green** button, in the X-Ray section and a Warning X-Ray indicator will turn on.

8.6.7 To open the Sample Changer, in the Simple Changer Assembly.

- Move the handle to unlock the Sample Changer, see in the figure above the blue circle (○).
- At the same time, press the yellow circles (○) in the front panel of the XRF machine, see in the previous figure above.

8.6.8 Turn on the XRF computer. **SIEMENS Spectra-3000** software window will appear. Choose from the toolbars menu among the followings options: **Measurement, Quantitative, Qualitative** and **System Maintenance**.

**8.6.9 Qualitative Scan - For Qualitative Analysis option to Define/Modify Qualitative Measurement Program.**

**NOTE:** This part of the Use and Management of the software should be use in parallel with the **Spectra 3000 – Training Manual**, because it will help you with the specific graphics screen, for each subjects or topics.

- Select '**qualitative**', then '**Edit Scan Ranges**'
- For a Full Scan; type **FULLSCAN.QAL** in the Program File.
- Select '**Select Analytical Program**'.
- Type the name for this Qualitative program (**\*.QAN**), or hit **<Enter>** when blank and use **<Down Arrow>** to choose an existing name.
- Select '**Edit Program parameters**'.

- Type a title for this program, and make sure the '**Measuring mode**' is correct. If desired external programs can be linked in on the '**User task**' line.
- Select '**edit Measuring ranges**'.
- Press **<Enter>** to define the first scan range.
- Fill in the required scan conditions. Starting and ending angles can be given as discrete values, or as element symbols for the K-alpha or L-alpha lines. Press **<Esc>** to go back to the list of ranges.
- Use **<Down Arrow>** to move to range 2, then press **<Enter>** to define this range.
- Fill in the required scan conditions. Press **<Esc>** to go back to the list ranges.
- Use **<Down Arrow>** to move to range 3, then press **<Enter>** to define this range.
- Fill in the required scan conditions. Press **< Esc>** to go back to the list of ranges.
- Press **< Esc>** after the ranges have been defined.
- Select '**Return to SPECTRA/DOS**' to exit.

#### 8.6.10 Automatic of Qualitative Data (\*.SSD)

- Select '**qualitative**', and then '**Automatic Evaluation**'
- Enter the name of the input file (\*.SSD) – this is normally the same as the '**Sample Identification**' used when the sample was measured. Accept the '**Option File**', or give a different name if desired. The default plotting characteristics will be read from this file, and later can be saved to this file.
- The first screen provides a summary of the data, and allows setting the full scale value for the intensity axis (**Full\_Ys**), and the size of the graphical plot (**X\_mode**). Once these have been set press **<F10>** to continue.
- Select the plotting characteristics. Use '**Save display Option**' to save the characteristics in a defaults file. Use **<F10>** to begin the plot.

#### 8.6.11 Interactive Evaluation of Qualitative Data (\*.SSD)

- Select '**qualitative**' and then '**Interact. Evaluation**'.
- Do '**select data File**'.
- Select the file to be viewed from the list. This displays the number of ranges the 1<sup>st</sup> time, and goes to the next screen the 2<sup>nd</sup> time the file is selected.
- Highlight the range to be viewed and press **<Enter>**.
- The data displayed on the screen. Intensity and 2-theta readings can be made with the mouse pointer.

- Define a “Zoom Window” where a single peak can be located. The data within this zoomed window will be used to calculate the gross peak intensity and to determine the background intensity.
- Click on '**Element**' name and enter the name of this element. Click on '**Concentration**' and enter the concentration for this element. NOTE: The concentration units must be given when entering value in “ppm” (25-ppm). The LLD for a counting time of 100 seconds will be displayed.
- Use '**Range operations**' to subtract one spectra from another or to adjust a spectra by some constant value.
- To set or check background measurement position in a Quantitative program: Select '**Quantification**' followed by '**select Quantitative program**'. Enter the name of the Quantitative program if it is not displayed (**.QAN.file**).
- Select the element from the list of elements.

#### 8.6.12 Plotter Manager

- Select '**quaLitative**' and then '**Plotter Management**'
- Select the files to be printed by setting these to '**Plot**'. Make sure the files you want to keep in the queue are set to '**Keep**'. Set the mode (BW.C4, C8 and C16). Select the transparency (TP) or normal print out (NP). Select high (**H**), medium (**M**), or low (**L**) print density. Press **<Enter>** when finished.
- Select '**Execute**' to begin making the printouts.

#### 8.6.13 Quantitative Scan - For Quantitative Analysis option to Modify Measuring Conditions (Measuring Parameter Sets)

- Select '**Quantitative**' and then '**Edit / calibrate**'.
- From the Main Menu window select '**Measurement parameters edition**'.
- Select '**add/remove Measurement parameters sets**'.
- Add: On blank line enter Element, Element + Line or Element + Line + \ Text View/ Change: Highlight desired MPS set **<Enter>**
- Make needed changes to conditions (Crystal, collimator, etc.). Move past last field and press **<Enter>** to view adjacent lines. **<Esc>** to exit.
- To go back **<Esc>** to exit.
- Return to main menu.
- Return to SPECTRA/DOS.

#### 8.6.14 Optimize Measuring Conditions for MPS sets

- Select Measurement and then Interactive.
- Interactive Adjust Screen
- Spectrometer control (left side of screen).  
To change item: 'click' on line (turns green),  
Special keys (yellow boxes):  
<F2> = Change MPS set (line)  
<F3> = Change sample position  
Other special keys:  
<Arrows> = Move mouse pointer  
<F1> = Toggle speed  
<Enter> = Left mouse button  
<Esc> = Right mouse button
- Program Control / Display (right side screen)  
To change item:  
'click' on line (turns green)  
Special keys (yellow boxes):  
<F4> = Select measuring mode  
<F5> = Send direct command to SRS  
<F6> = Call interactive evaluation program  
<F7> = Call qual program editor  
<F8> = Clear graphics area  
<F9> = Exit program.  
<F10> = Start/Stop measurements

Other special keys same for spectrometer control side of screen.

- If the Flow detector is being used, check the discriminator settings for it by:  
Press <F4> and set Mode to "PHA (F.C.)"  
If Data Collection is "Stopped" press <F10> to switch to "Running". After data is displayed on screen the discriminator (blue line below graphics area) can be adjusted by:  
  
Click on "LLD – F.C." under detection (lower left)  
Use <Arrows> or mouse to change lower level  
  
Click on "ULD – F.C." under detection (lower left)  
Use <Arrows> or mouse to change upper level
- If the Scint detector is being used, check the discriminator settings for it by:  
Press <F4> and set Mode to "PHA (S.C.)"



If Data Collection is “Stopped” press <F10> to switch to “Running”. After data is displayed on screen the discriminator (blue line below graphics area) can be adjusted by:

Click on “LLD – S.C.” under detection (lower left)  
Use <Arrows> or mouse to change lower level

Click on “ULD – S.C.” under detection (lower left)  
Use <Arrows> or mouse to change upper level

- Check the location of the peak, where to measure backgrounds, and interference from adjacent lines by:

Press <F4> and set Mode to “Peak Search”

If Data Collection is “Stopped” press <F10> to switch to “Running”. After data is displayed on screen the white vertical line in the graphics area indicates the peak angle which is currently stored, and the green vertical line represents where the actual peak location is.

- To identify any peak:  
Click on the peak in the graphics area. A table of 1<sup>st</sup> and 2<sup>nd</sup> order lines within a fixed window (the green line below the graphics area) will be displayed below the right side of the graphics area.  
Press any key to continue.
- To set the peak angle to the calculated angle :  
'click' on the “Stored 2 theta” field in the Peak Box. Adjust peak position with mouse, or press <F1> to save the calculated angle.
- To save the data in a file, and run the “Interactive evaluation” program press <F6> and enter the name for the data file.
- If a file already exists with the same name, the select:  
Change Name to enter a different file name  
Append data to add this data to the end of the existing file  
Overwrite to delete the existing file  
Quit to ADJ to cancel
- All the normal functions of the “Interactive Evaluation” program are available.

- To exit the “Interactive Evaluation” program and to return to the “Interactive Adjust” program:  
‘click’ on “Files / Plot / system” on the menu line at the bottom of the screen.  
‘click’ on “Quit-> DOS/Spectra”.

- The sample was continuously measured while the “Interactive Evaluation” program was run. The data from these measurements is read, and the graphics display will be updated.

- To create a qualitative program for measuring peak scans and pha scans at a future data without using the “Interactive Adjust” program:

Press <F7> and enter the name for the qualitative program (\*.QAL).

- If this is a new program, the measuring conditions are automatically transferred from the “Interactive Program” to the first range.
- If this is an existing program, transfer the measuring conditions by positioning the cursor on the range (line) typing “A” (copy ADJ measuring parameters).
- The sample was continuously measured while the “Interactive Evaluation” program was run. The data from these measurements is read, and the graphics display will be updated.
- To exit the “Interactive Adjust” program press <F9> or <Esc>  
Select “Return save updates” to update measuring parameter sets (MPS) in MEASPARM.DAT

#### 8.6.15 Create/Modify Quantitative Program (\*.QAN)

- Select the **Quantitative** option and then select for **Edit/calibrate**
- **Program file selection**
- Enter a name for this program
- Enter **Y** to create.
- **Edit analytical program**
- **Edit Program parameters**
- Enter mode, mask, data files, defaults etc.  
**<Esc>** to exit
- **Add / remove standards**

- Enter the names of calibration standards (file names\*.STD)  
<Esc> to exit
- **Add/remove compounds**
- Enter reporting names for compounds (elements) in print order.  
Follow each with <Down-Arrow>.  
Highlight compound and press <Enter> to view details.
- Enter info for this compound (units, decimals, etc.) Highlight the MPS field and press <Enter> to check it.
- <Esc> to exit.
- Move past last field to pop-up additional menu. Select appropriate type, normally “**Fixed order**” (measuring seq)  
Press <Enter>
- If “Fixed order” was selected from previous screen, enter the sequence number for measuring this line and press <Enter>
- <Esc> to exit.
- <Esc> to exit.
- Return to main menu
- Return to SPECTRA/DOS

#### 8.6.16 Enter/Modify Concentrations for Standards (\*.STD)

- Select the **Quantitative** option and then select for **Edit/calibrate**
- **Program file selection**
- Enter program name and press <Enter>
- **Standard data edition**
- **Load concentrations**
- **Input concentrations**  
<Esc> to exit.
- To calculate total or balance element click Auxiliary menu
- Adjust sum/compliment <Enter> (looks like it does nothing).
- Return to prev. menu
- Input concentrations (To look at calculated sum/complement)
- Enter the concentrations values
- <Esc> to exit.
- Write concentrations
- Return to main menu
- Return to SPECTRA/DOS

#### 8.6.17 Measure Intensities for Calibration Standards (\*RSI)

- Select the **Quantitative** option and then select for **Edit/calibrate**
- **Programs file selection** (if program is not already selected).
- Enter program name and press **<Enter>**
- Make standard measurement file
- Creates a sample set file (\*.SSF)  
Press any key to continue.
- Return to SPECTRA/DOS
- **Measurements**
- **Routine**
- **<Esc>**
- **Select sample set file**
- Enter program name and press **<Enter>**
- Load standards according to screen **<F10>** starts measurement.
- Return to DOS or SPECTRA

#### 8.6.18 Calculate Calibrations Coefficients (Regression)

- Select the **Quantitative** option and then select for **Edit/calibrate**
- **Programs file selection** (if program is not already selected).
- Enter program name and press **<Enter>**
- Load all standards measured  
(NOT Load all standards stored)
- **Calibration routines**
- **X = Automatic calibration**
- **Graphical Evaluation**
- Select options from scrolling menu at bottom.
- **Adjust\_coef** = calculate line
- **Disp\_tables** = show calibration tables
- Use **<Up/Down Arrows>** to select
  - to disable
  - + to enable
  - <Esc>** to exit
  - <Enter>** recalculate when removed
- **Compound** = select the next element (if more than one element)
- Use **<Up/Down Arrows>** or mouse to select new compound
- **Return** = Leaves the graph (plot)
- Return / update program
- Return Spectra / DOS
- Append changed data

- **Inten\_model** = Enable / disable background subtraction  
Line-overlap factors  
Ratio to internal standard
- ? in overlap corrections column and **<Enter>** to display adjacent lines.
- **<Esc>** to exit
- Enter overlap compound if needed. **<Esc>** to exit.
- Read RSI files
- **Formula\_cnc** = Fix/calculate offset  
Inter-element corrections  
Polynomials
- Change Model, weighting, offset.  
In absorption column ?**<Enter>** calculates relative absorptions.
- Enter major element (matrix) or **X** if concentrations sum to 100%. **<Enter>**
- Displays relative absorption of analyte line by all elements  
**<Esc>** to exit.
- Enter absorber and press **<Enter>** to calculate correction factors **<Esc>** to exit.
- **Point\_samp.** = enable mouse pointer to select samples.
- Select sample with mouse pointer and click **LEFT (or <Del>)**
- Name for selected standard is displayed
- Enter **C** to display concentrations
- Enter **I** to display intensities
- Enter **U** to disable
- Enter **E** to enable
- **<ESC>** to exit
- **Zoom\_int.** = zoom using intensity scale
- **Zoom\_dev.** = zoom using deviation scale
- **fix\_Scale** = set explicit values for intensity, concentrations and deviation scales.
- **reset\_Scale** = reset the intensity, concentrations and deviation factors.
- **Presentation** = select what gets plotted and displayed.
- Print mode selection
- **Expanded**
- **X = Automatic calibration**

8.6.19 *Recalibration (Drift Correction)* – Preliminary setup if Reference Samples were not declared when the Quantitative Program was first created.

Note that reference standards can only be added to a calibrated quantitative program if they are part of the original calibration standards. If they are different all standards must be re-measured.

- At the SPECTRA 3000 screen, select 'Quantitative'.
- Select 'Edit / calibrate'.
- If program is not already selected do "Program file selection".
- Enter the quantitative program name <Enter>.
- Edit Analytical program.
- Add / remove compounds
- Highlight compound and press <Enter>.
- Make sure that Reference Samples are named for this compound. <Esc> to exit.
- <Esc> to exit, once again.
- This return to main menu.

If Reference sample are being added to a calibrated quantitative program, the initial intensities (those measured when the calibration standard were ran) must be manually loaded by:

Calibration routines

- Maintenance Operations
- Load initial (All)
- Return to main menu
- Return / update program
- Return to SPECTRA / DOS
- To make future measurements of the Reference sample simpler, a special sample set file can be created by:  
Measurement  
Routine
- <Esc> to exit.
- Select sample set file
- Enter the name for this sample set file (R + program name).
- Enter 'Y' to create the sample set file.
- Edit sample set file
- Enter reference sample names, quant program, mode RE onto form. If desired, the T.S. (time scale) can be increased to improve precision (e.g. 2 or 3 X the counting time). <Esc> to leave.

- Return to DOS Spectra
- Measurement
- Routine
- <Esc> to exit
- Select sample set file.
- Enter the name for the sample set file (R + program name).
- Edit sample set file. Load Reference sample in the order shown. <F10> to start the measurement
- Return to DOS Spectra


8.7 *Data Acquisition* - Data may consist of the graph information and/or elemental information of a Qualitative or Quantitative process. Graph and Elemental information will be processed after the procedure instructions in the SRS-303, automatically. The graph and Elemental information will be printed.

#### 8.8 *Turn-OFF the XRF, Once Finished*

8.8.1 In the right side of the XRF machine, the Generator and the Interface, see in the figure.

- Press the **yellow** button, in the X-Ray section.
- Then, press the **red** button, in the X-Ray section and a Warning X-Ray indicator will turn Off.
- Turn the key, in the X-Ray section.
- Turn OFF the two switches of the cards SC and FC
- Turn OFF the **green** switch of ON and OFF.

8.8.2 In the left side of the XRF machine, turn on the switch of the Spectrometer at 23 psi, see in the figure.

8.8.3 In front of the machine, lock the three front doors with their own key to access the panels, see in the figure 8.6 with the red arrows (  ).

8.8.4 *Chiller* - must be Turn OFF 10 to 15 minutes after of the operating the XRF machine.

8.8.5 *Air Compressor* – A 60-gal and 5-hp Craftsman (Sears Brand), which is located in front of F-305 at the balcony / hall of the third floor of Physics building, must be Turn OFF 15 to 30 minutes after of the operating the XRF machine.

## 9.0 RECORDS

The **Procedure User** is responsible for submitting the following records to the Machine Custodian/Lab. Instrumentation specialist to be storage at the UPR-NSF Earth X-ray Analysis Center (EXACT!), F-304-C.

- 9.1 Log-Book or Notebook records of the sample handling and results of analysis relevant to Production of XRF data.
- 9.2 Data submittals will be storage in the electronic database.
- 9.3 It is obligation of the user to backup the data obtained.
- 9.4 Data will be held by the laboratory for thirty (30) days, after which it may be eliminated from departmental files.

## 10.0 REFERENCES

The following documents have been cited within this procedure by:

- <http://geology.uprm.edu/facilities/exact.html>
- <http://geology.uprm.edu/facilities/labrules/exact.doc>
- SIEMENS SRS 303 and SRS 303 AS Sequential X-Ray Spectrometer – Training Course; SIEMENS Analytical X-Ray Instruments, Inc.
- Spectra 3000 – Training Manual; SIEMENS SPECTRA – 3000, v3.1  
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