FLORIDA STATE UNIVERSITY

Powder X-Ray Diffraction Alignment Procedure

Procedure for aligning Rigaku Ultima III w/ Mercury CCD using Scintillation Detector for calibration

11/18/2014

This document describes the alignment procedure for Florida State University Chemical Sciences Lab's Rigaku Ultima III powder x-ray diffractometer with Mercury CCD. The procedure involves swapping the CCD with Scintillation Counter for alignment and back again for nano material powder x-ray diffraction. © 2013-14 Thayumanasamy Somasundaram.

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Version 0.9.6 dated November 18, 2014

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ULTIMA III ALIGNMENT PROCEDURE V0.9.6 (Beta)

VER 0.9.5; DATED NOVEMBER18, 2014

SWITCHING FROM CCD TO SCINTILLATION COUNTER AND BACK

Written by Dr *Thayumanasamy Somasundaram* on November 17, 2004

The following write-up with photographs will show the alignment procedure to be done on Rigaku Ultima III Powder X-Ray Diffractometer (ru-pxrd) once in couple of months. Thanks to David Carnevale for help in demonstrating the procedure.

Note: *This write-up is a beta version and some refinement is needed on some steps. Steps done at the PC (computer) will be shown in* Times New Roman *font; Sections done at the instrument r-pxrd will be shown in* Georgia *font.*

START THE SHUTDOWN

Rigaku Ultima III Powder X-Ray Diffractometer (shown on the right hand panel) with Mercury CCD and Scintillation Detector is located in CSL 1011 in FSU's Chemical Sciences Laboratory.

The generator has a Copper fixed anode tube and is cooled by Haskris chiller (originally a refrigerated version; now water-to-water chiller). The Mercury CCD is cooled by Julabo MD F25 chiller.

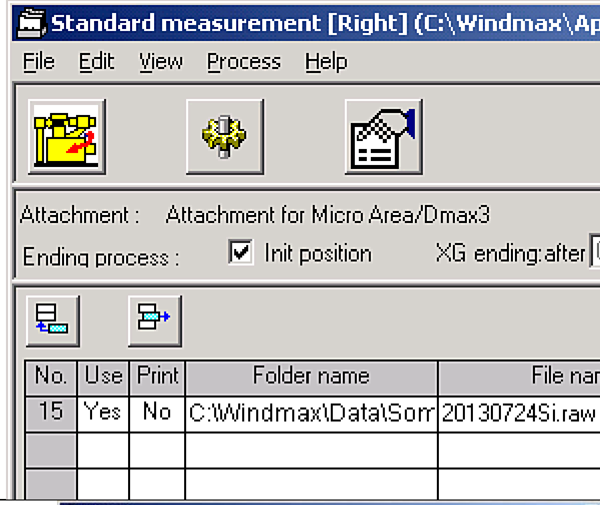
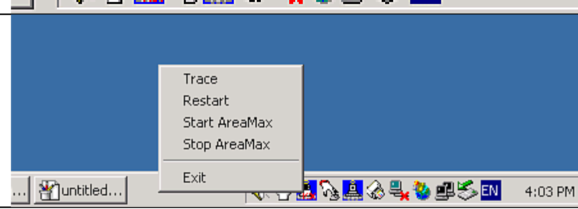
Haskris chiller for X-Ray tube

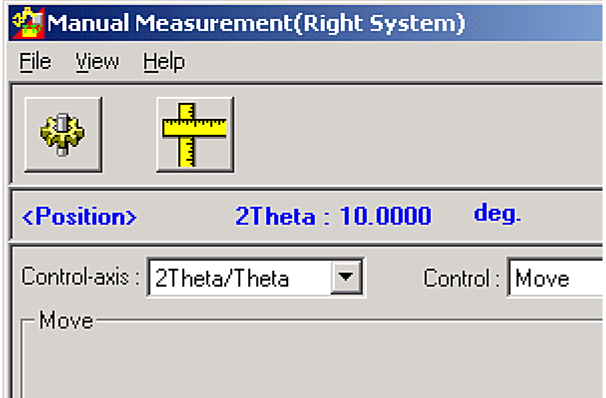
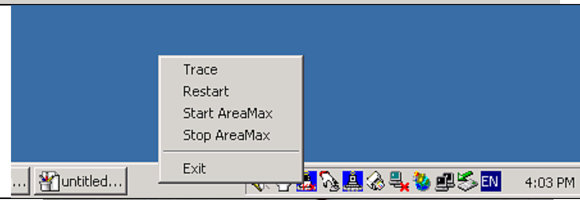

Julabo MD F25chiller for CCD

Two computers control data collection and processing: 1) Control PC on the right and 2) Frame Grabber PC on the left. However, there is only ONE monitor and key board. So you access each computer using a KVM switch.

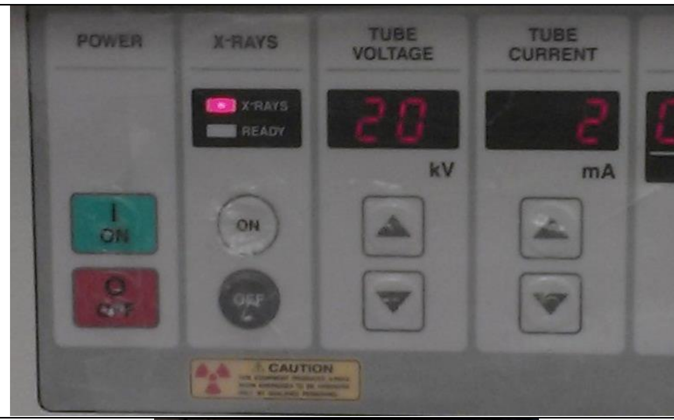
In the keyboard press, "Scroll Lock" key twice quickly to switch between the two PCs. Almost all of the time the monitor outputs and keyboard inputs are from and to Control PC.

Log in to control PC. It is likely that it is already running.

In the ‘Standard Measurement Window’, select • File > Save • Confirm This allows the Standard Measurement values to be remembered when we restart the computer.	
Close RaxVideo window	
Left Click on RGKExpand on the left side of System Tray, then select • Stop AreaMax This closes the AreaMax window (better way to close it)	 
Close Standard Measurement Window	

<table>
<tr>
<td>Close Manual Measurement Window</td>
<td>
</td>
</tr>
<tr>
<td>Left Click on RGKExpand on the left side of System Tray, then select
Exit</td>
<td>
</td>
</tr>
<tr>
<td>In the Ultima III Generator’s Left Top Panel, under the SAFETY RELEASE panel, press the ‘DOOR’ button. While the DOOR’ button is blinking, open the sliding door all the way to left and right.</td>
<td>
</td>
</tr>
<tr>
<td>Inside the x-ray enclosure, locate the DALSA power supply (usually located on the right hand side and sits on the base plate) for the CCD detector. Power it down by switching it OFF (the power switch is located in the front of the unit, however, only the unit’s back is visible) by locating the switch on the far right hand side.</td>
<td>
</td>
</tr>
<tr>
<td>Now, in the computer using the single keyboard press, “Scroll Lock” key twice quickly to switch to Frame Grabber PC.
Log in using username and password
Wait for the computer to display a DOS window
Using the Start Button, shutdown the computer</td>
<td></td>
</tr>
</table>

<table>
<tr>
<td>Now, in the Ultima III Generator’s Left Top Panel,

Under X-RAYS
Press the Grey OFF button, then
Under POWER
Press Red OFF button
</td>
<td>
</td>
</tr>
<tr>
<td>Then, in the Ultima III Generator’s Left Bottom Panel, open the door and locate the SERVO AMP UNIT,

Turn it OFF by pressing the black power switch to off position.

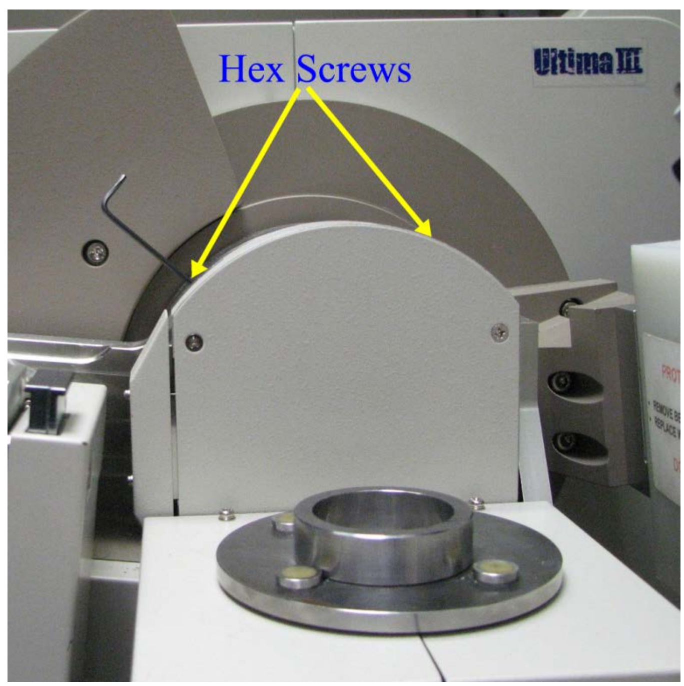
Turn-off the Control PC now.</td>
<td>
</td>
</tr>
</table>

SWAP THE EQUIPMENT

SWAP CCD W/ SCINTILLATION COUNTER AND MICRO AREA W/ STANDARD SAMPLE HOLDER

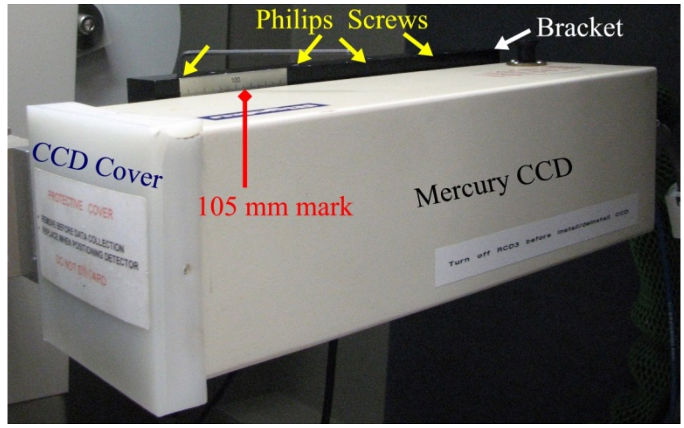
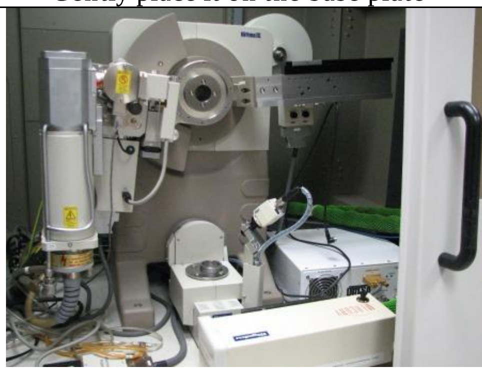
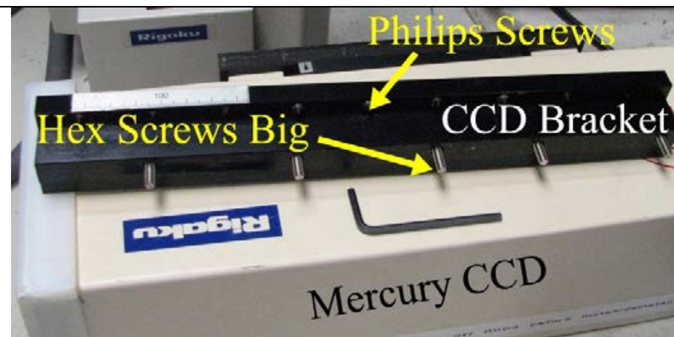
<table>
<tr>
<td>With Ultima III generator door open, locate the CN ATT2 cable (this is plugged underneath the stage) and unplug it</td>
<td>
</td>
</tr>
</table>

<table>
<tr>
<td>Attach the CN ATT2 cable to the ATTACHMENT ADAPTER sitting on the base plate.

The CN-ATT2 cable may fit loosely but make sure it secured as firmly as practical.</td>
<td>
</td>
</tr>
<tr>
<td>Locate the plastic lid for the CCD and cover the CCD with it</td>
<td>
</td>
</tr>
<tr>
<td>Remove the micro-area stage (used for powder samples)
Use the small hex wrench and undo the two screws until they are flush (on-top) with surface [2 or 3 mm hex wrench]
Gently pull the stage toward the user
Sit it down on the base plate of the instrument</td>
<td rowspan="2">
</td>
</tr>
<tr>
<td>
Stage sitting on base plate</td>
</tr>
</table>

<table>
<tr>
<td>Remove the CCD Camera (with lid on it)

Hold the CCD in one hand
Note down the value on the back side (~105mm)
Use Philips screw driver and undo all the four screws flush to the top
Slowly wiggle the CCD and remove it
Gently place it on the base plate

</td>
<td rowspan="2">
</td>
</tr>
<tr>
<td>
Stage and CCD sitting on base plate</td>
</tr>
<tr>
<td>Using larger hex wrench undo all the larger screws flush to the top and remove the black bracket that was holding the CCD and remove it completely (with the screws) and leave it on top of the CCD inside the enclosure.</td>
<td>
</td>
</tr>
<tr>
<td>Now, remove the two black brackets from the cabinet above the Control PC (they are marked #1 and #2)

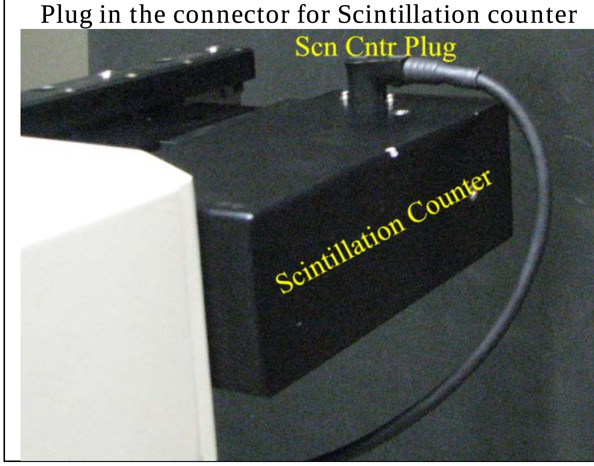
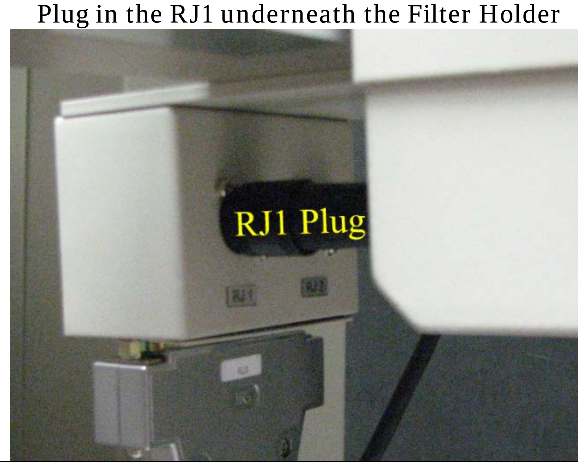
Put the #1 bracket close to the sample
Put the #2 bracket away from the sample
Use the hex wrench and secure them to the detector-arm of the instrument (the same place where the long black bracket was secured)

</td>
<td>
</td>
</tr>
<tr>
<td>Now, we to put the Filter Holder (Part #53D-0466) in front and Scintillation Counter (Part # 57 38E401) and at the back with very little gap between them.</td>
<td></td>
</tr>
</table>

<table>
<tr>
<td>First, put the Filter Holder (FH) in front

Lighlty secure it in the detector-arm with 3 small hex screws. DO NOT fully tighten it
Look underneath the FH and locate a “Red Line”
Match this “Red Line” with the “Red Line” underneath the detector-arm
Now tighten the Filter Holder fully
</td>
<td>
View from the bottom of the Filter Holder</td>
</tr>
<tr>
<td>Filter Holder in position now</td>
<td>
</td>
</tr>
<tr>
<td>Now, put the Scintillation Counter behind the Filter Holder with very little space between them

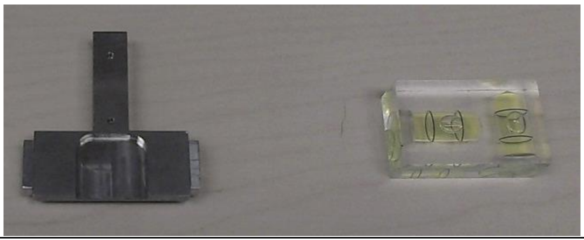
Secure it with 2 hex screws

Now we have both Filter Holder and Scintillation counter in place</td>
<td>
</td>
</tr>
<tr>
<td>Plug in the connector for Scintillation counter

</td>
<td>Plug in the RJ1 underneath the Filter Holder

</td>
</tr>
</table>

Now, put the new stage Standard Sample Holder (stored inside the cabinet above the PC)

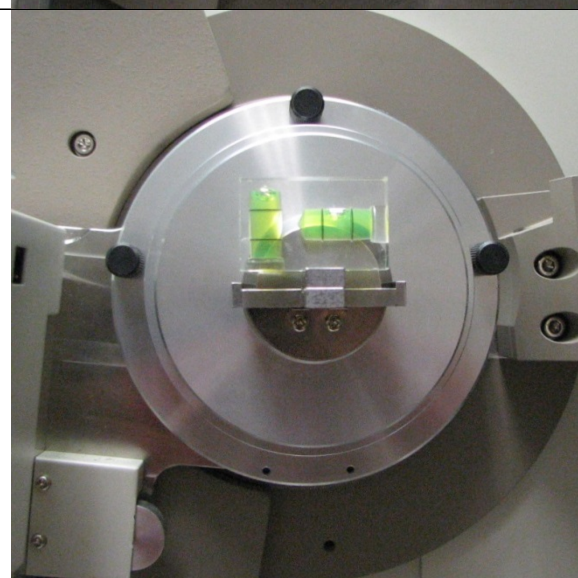
- Push the stage into the slot where the Micro-Area stage was installed
- The correct position of stage is guaraunteed if the three holes in the stage are aligned with three holes on the instrument back panel
- Put the thumb screws and gently tighten
- Insert center slit

Center Slit and bubble level



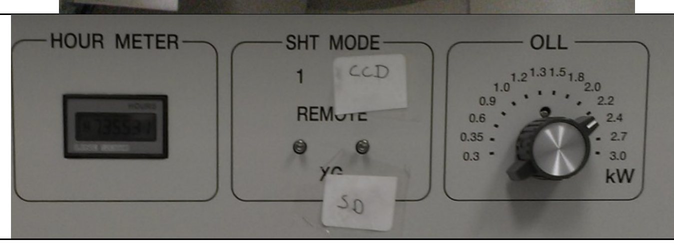
With the center slit and bubble level in place now gently rotate the stage until the bubble is level both horizontally and vertically.
Then hand tighten the thumb screws. The standard sample holder is now set.

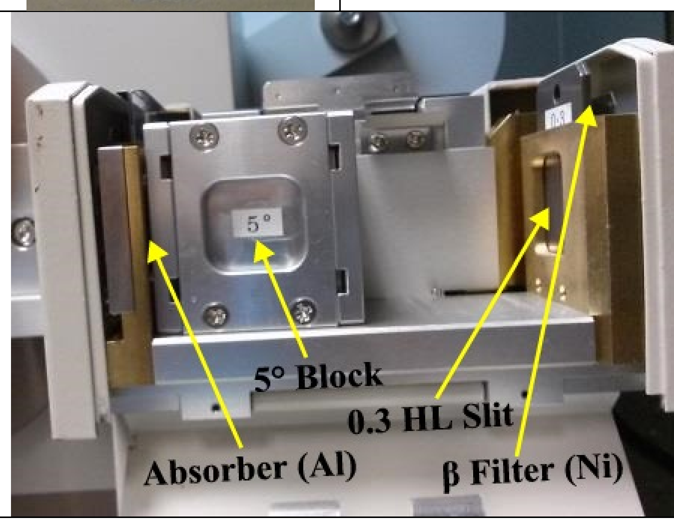
- Remove the level and the center slit



Now open the Right Hand Side Bottom panel on the generator

- Change the SHT MODE switch from CCD to SD (flit it down)



Now open the Left Hand Side Bottom panel on the generator • Press the first switch from bottom on the left-most PC card once. • Briefly the blinking "green" LED light will stop blinking confirming your reset • You will also see all the front panel LEDs light up and go away	
• In CBO mirror box remove the H2 slit and put H10 slit. The lable should be facing the sample. • Leave the PB in the holder	 
In the back filter holder • Open the box • Put Al foil in the front • (0.3) slit in the back • Put in 5 deg block (letter facing the user) • Ni foil at the back • All labels facing the sample Close the box	

RESTART

RESTART W/ SCINTILLATION DETECTOR FOR ALIGNMENT

Restart in this sequence

1. Switch ON the SERVO AMP UNIT
2. Press GREEN button in POWER Panel
3. Press White button on X-RAY panel
4. **Confirm Shutter 2 is in EXT mode**

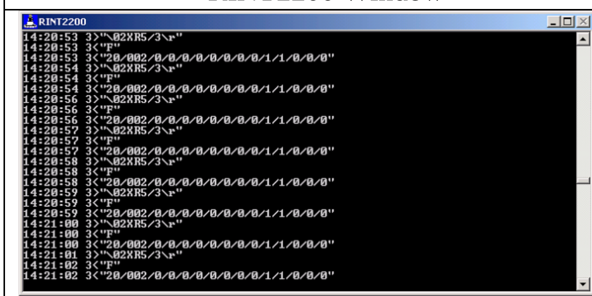
Turn on the Control PC

Hit ‘F1’ to continue

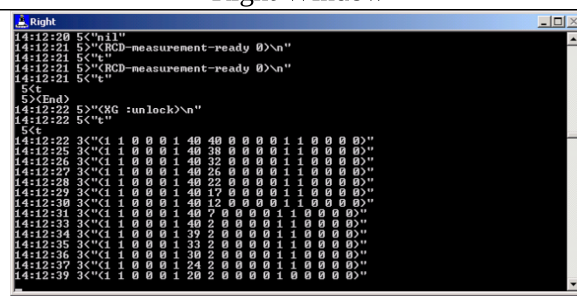
Log in

- Administrator
- Password
- Right Click on Blue Rigaku Measurement server in Toolbar Menu
- Select RIN2200 and select to ‘ShowWindow’
- Select Right and select to ‘ShowWindow’

RINT2200 Window

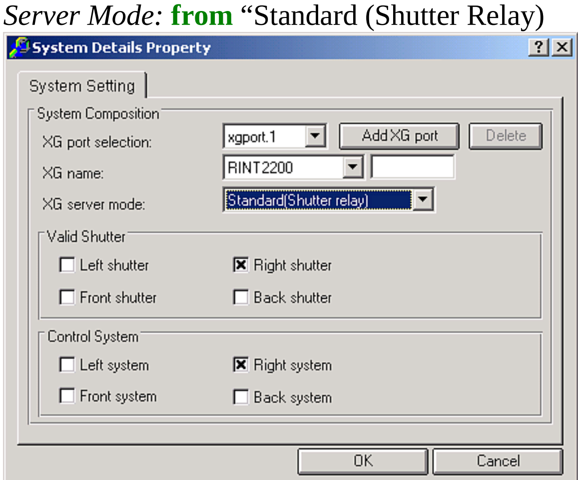


Right Window

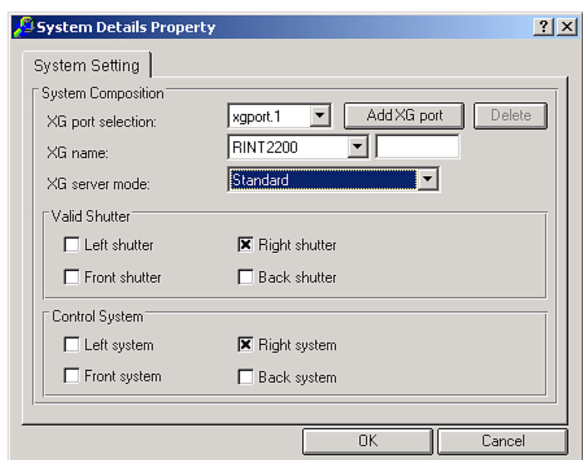


From Desktop open the Rigaku Control Panel Folder. In Control Panel open “System Details Button”

System Details Property Window change the *XG Server Mode:* **from** “Standard (Shutter Relay)



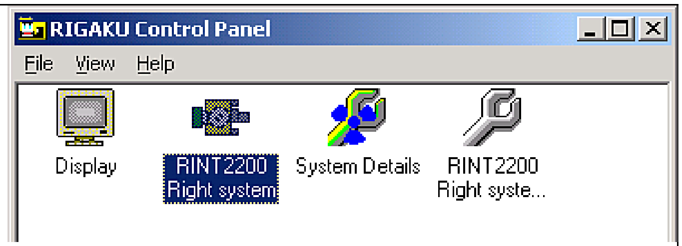
To “Standard”



Click ‘Ok’ and ‘Confirm’ the change. Right window will update.

Now click on the RINT2200 Right System (but not the ~~RINT2200 Right System Add/Delete button~~). This should open a new dialog box. In the following three tabs RINT2200 Right System Property need to be changed.

- System Construction
- X-Ray Beam Type
- Geometry System

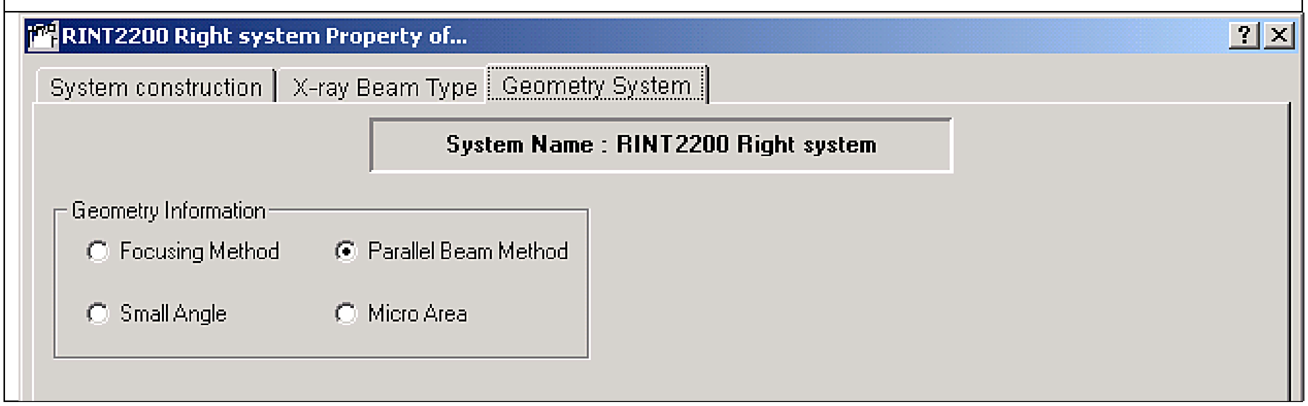


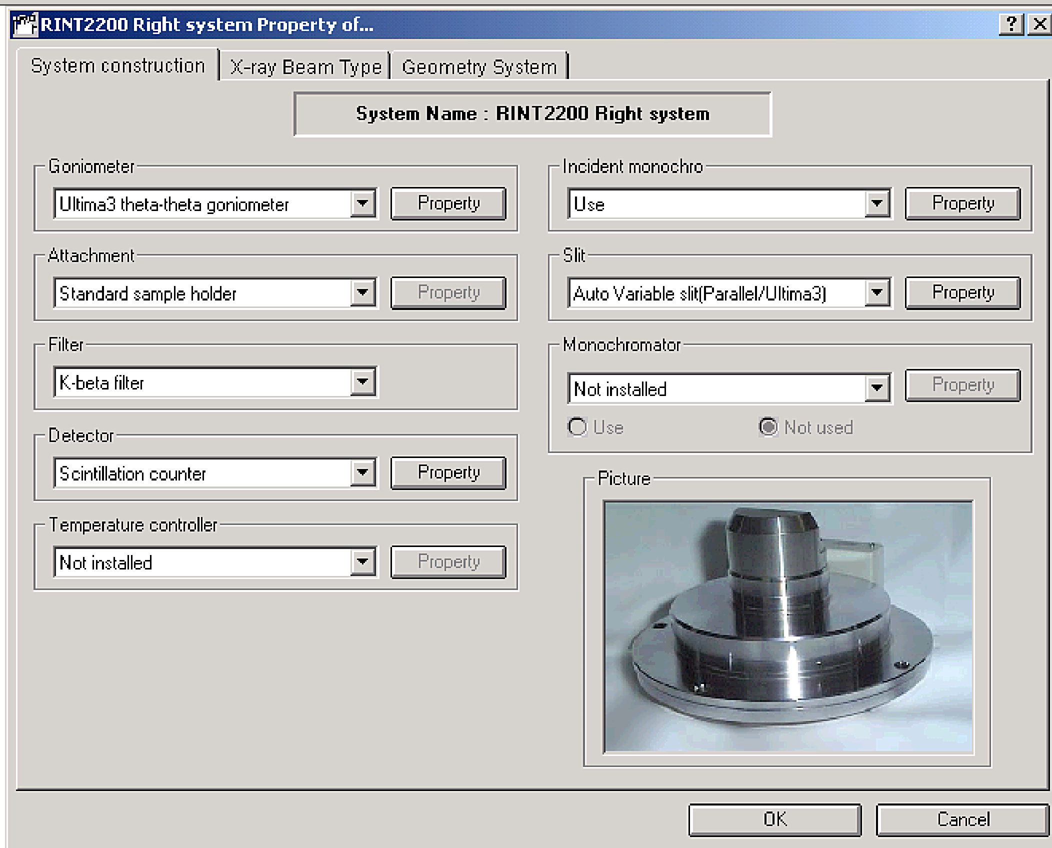
System Construction	
CHANGE FROM	CHANGE TO
Goniometer: Ultima 3 theta-theta goniometer+Mercury CCD	*Goniometer*: Ultima 3 theta-theta goniometer
Attachement:Micro area	*Attachment*:Standard Sample Holder
Filter: Not installed	*Filter*:K-beta filter
Detector:Mercury CCD	*Detector*: Scintillation Counter
Temp Contoller:Not installed	*Temp Contoller*:Not installed
Incident Monochromateor: use	*Incident Monochromateor*: use
Slit: Auto Variable (Mercury CCD/general)	*Slit*:Auto Variable Slit (Parallel/Ultima3)
Monochromator: Not installed	*Monochromator*: Not installed

X-Ray Beam Type	
CHANGE FROM	CHANGE TO
No change. *Focus Selection*: Line; *Wave length selection*: K-alpha	

Geometry System	
CHANGE FROM	CHANGE TO
Geometry Information: Micro Area	*Geometry Information:* Parallel Beam

After the change the three tabs should like the following



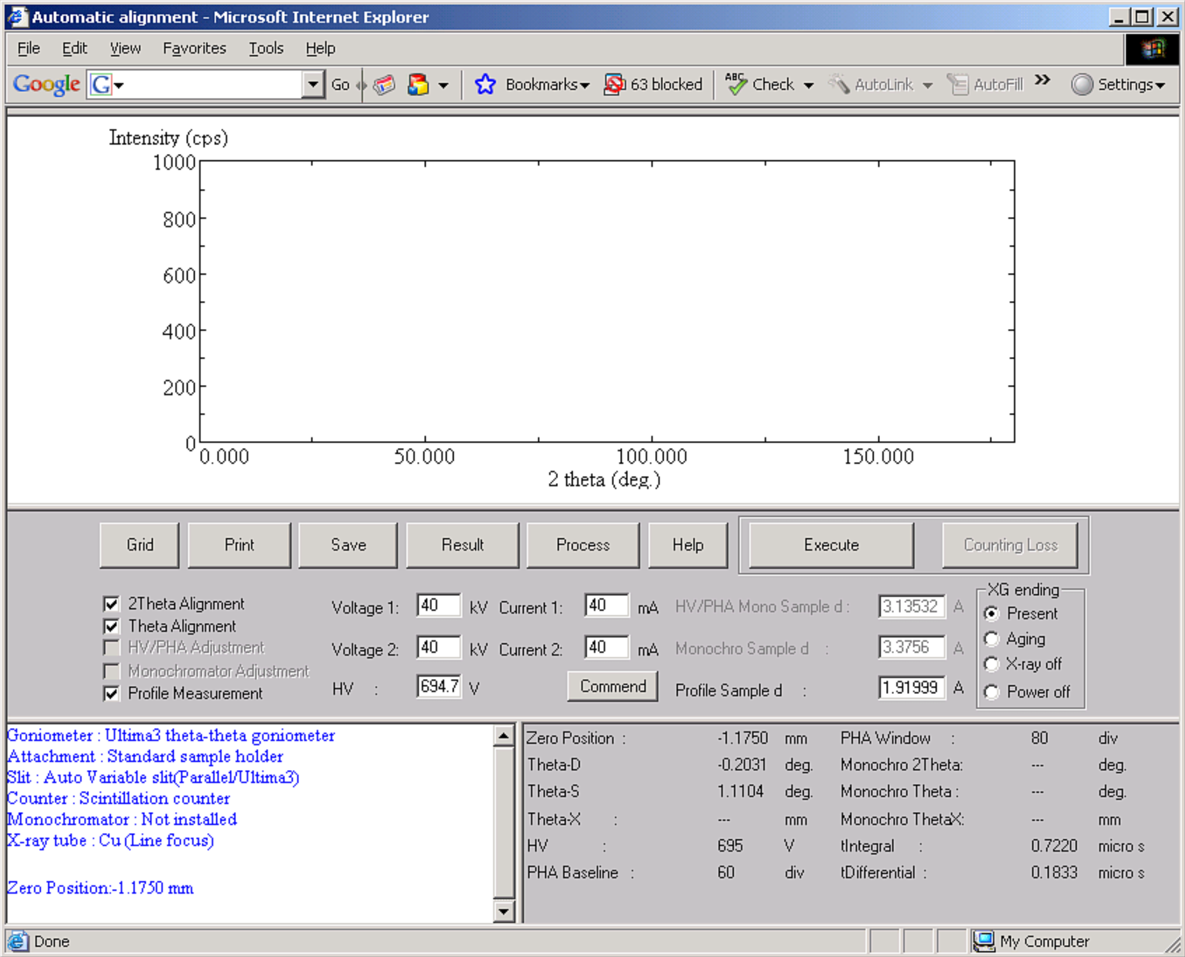


START THE ALIGNMENT PROCEDURE

ALIGNMENT PROCEDURE USING. HTM FILE

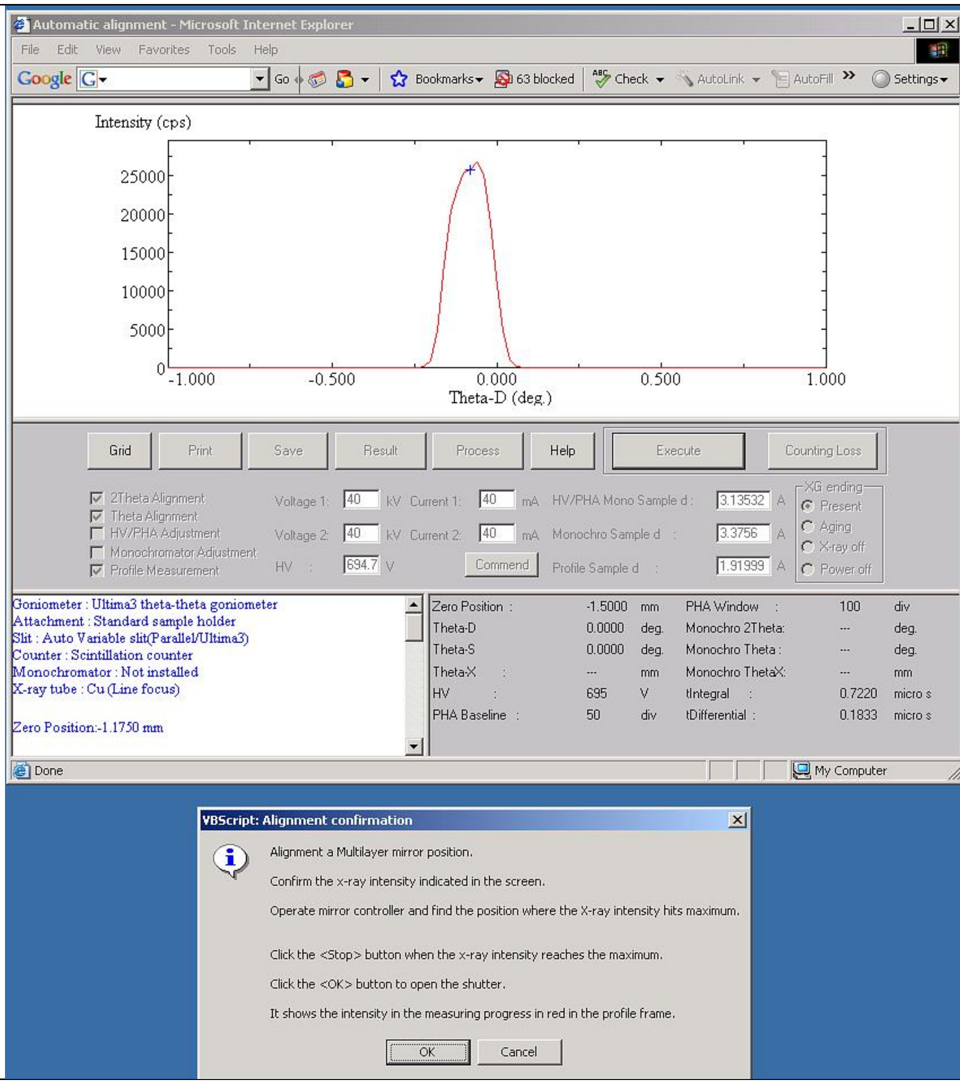
Now open the Auto-align(Right system).htm link in the Desktop

- It will open an Internet Exploer window and will load lots of stuff
- In the window, select the following
  - 2-Theta Alignment
  - Theta Alignment
  - Profile Measurement



Now click the button “Execute” in the .htm file (center area) and follow the directions

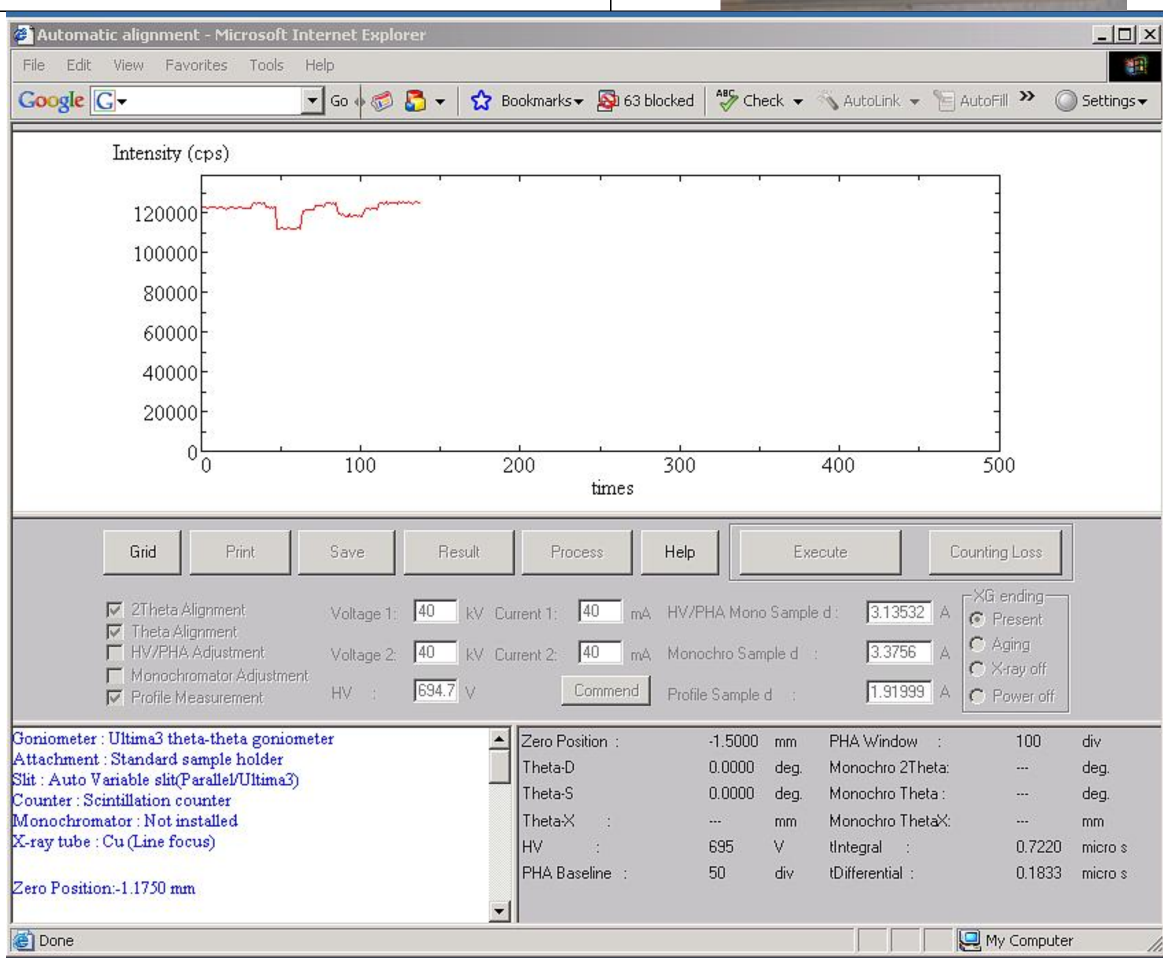
- Confirm everything
- Initializing … … (takes sometime)
- X-Ray tube (left side of the instrument) will move
- 2 Theta arm (right side of the instrument) will move
- CBO box will move up and down
- 20 kv and 2mA will go to 40 kv and 40 mA
- Tube and 2 Theta will move to zero positions
- A scan of -1.0° to +1.0°Theta-D will be executed and the results will be displayed on the top portion of the htm file and bottom left hand side will also update information constantly



Now Press "OK" to confirm. The htm window will update and will now proceed to next step.

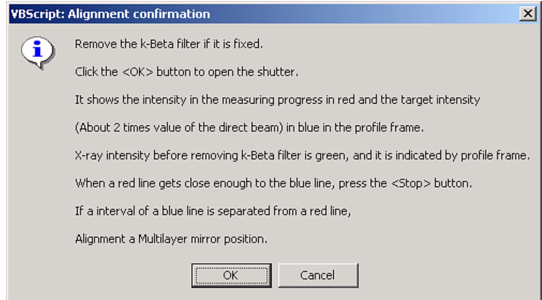
Now pick-up the DC Motor Controller (usually found on top of the instrument cabinet,on the Julabo side)

- Put two 9V batteries on the side opening
- Swith the Power ON (top of the controller)
- Now toggle the joystick left or/and right while watching either the .htm window or the right window
- The goal is to maximize the intensity (the joystick has some hystersis and will occasionally reduce the intensity precipitously even for a small L or R toggle). So be patient and careful!
- Once successful, hit “Break” stop measurement and Confirm “Stop”
- The results are shown below.

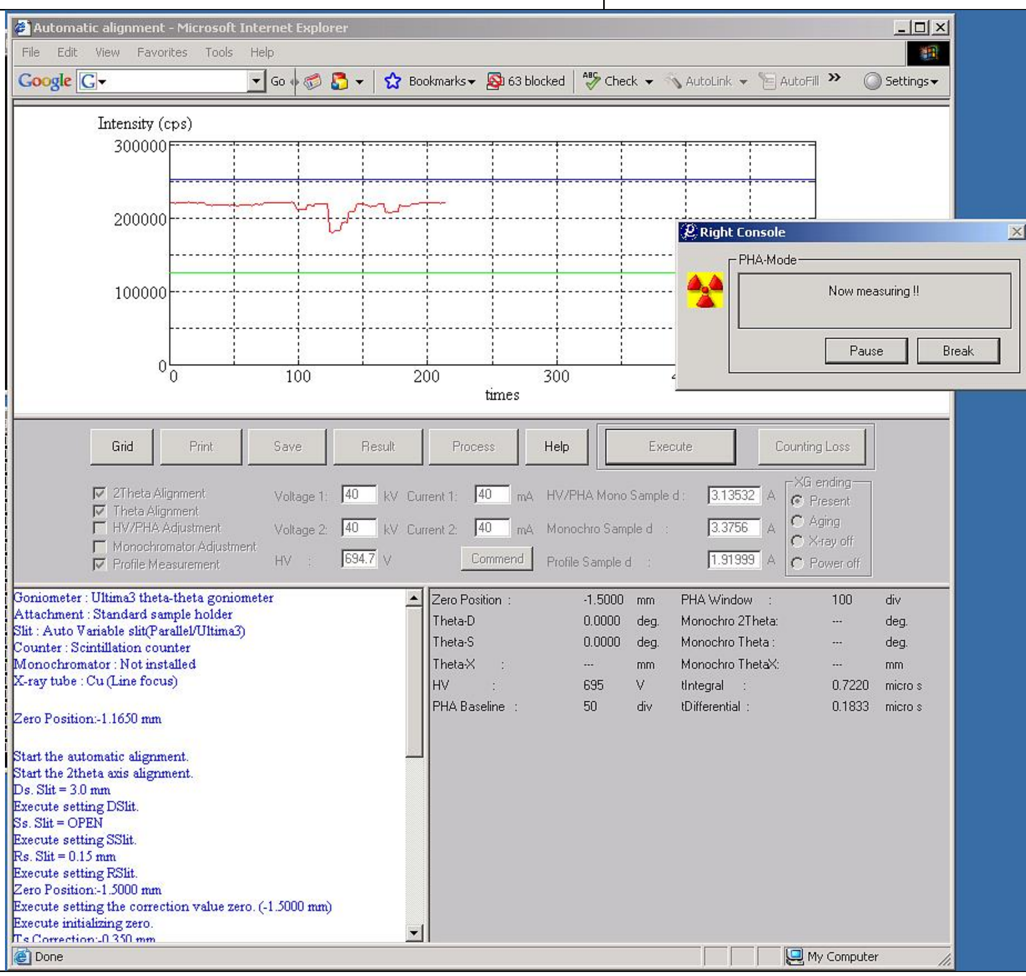


Now follow the on screen instructions and do the following:

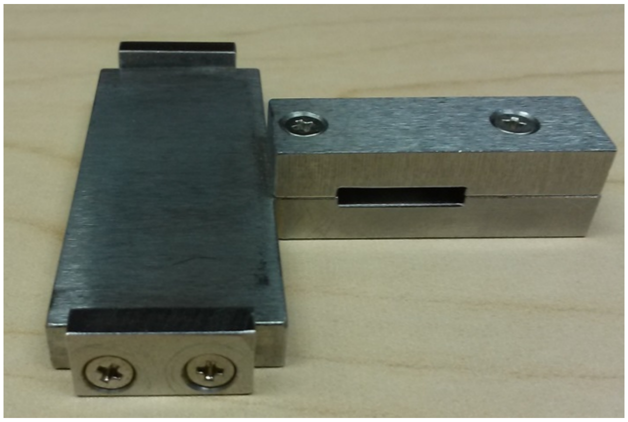
- Remove the Ni foil (K-beta filter); Open the door, remove filter, **close door of the filter box too!**
- Hit “Okay” on the pop-up window
- New htm window will open and three lines will be shown:
    - Red: Current Measurement (w/o K-beta filter!)
    - Green: Value with K-beta filter
    - Blue: Target intensity
- Once again the toggle the joystick to maximize the intensity. As close as possible to the “Blue line”.



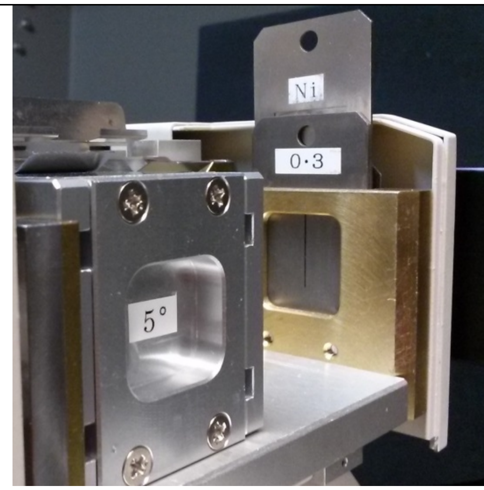
Once maximum is achieved, click the “Break” button which stop the optimization procedure. Confirm to stop measurement. Typical result is shown below



Since no more optimization is possible, select “**No More alignment**” button when presented. Now swith “OFF” the DC Motor Controller, remove the two 9-VDC batteries and store the batteries in the cabinet box and place the controller back on top of the X-Ray Safety Cabinet (on the Julabo chiller side)

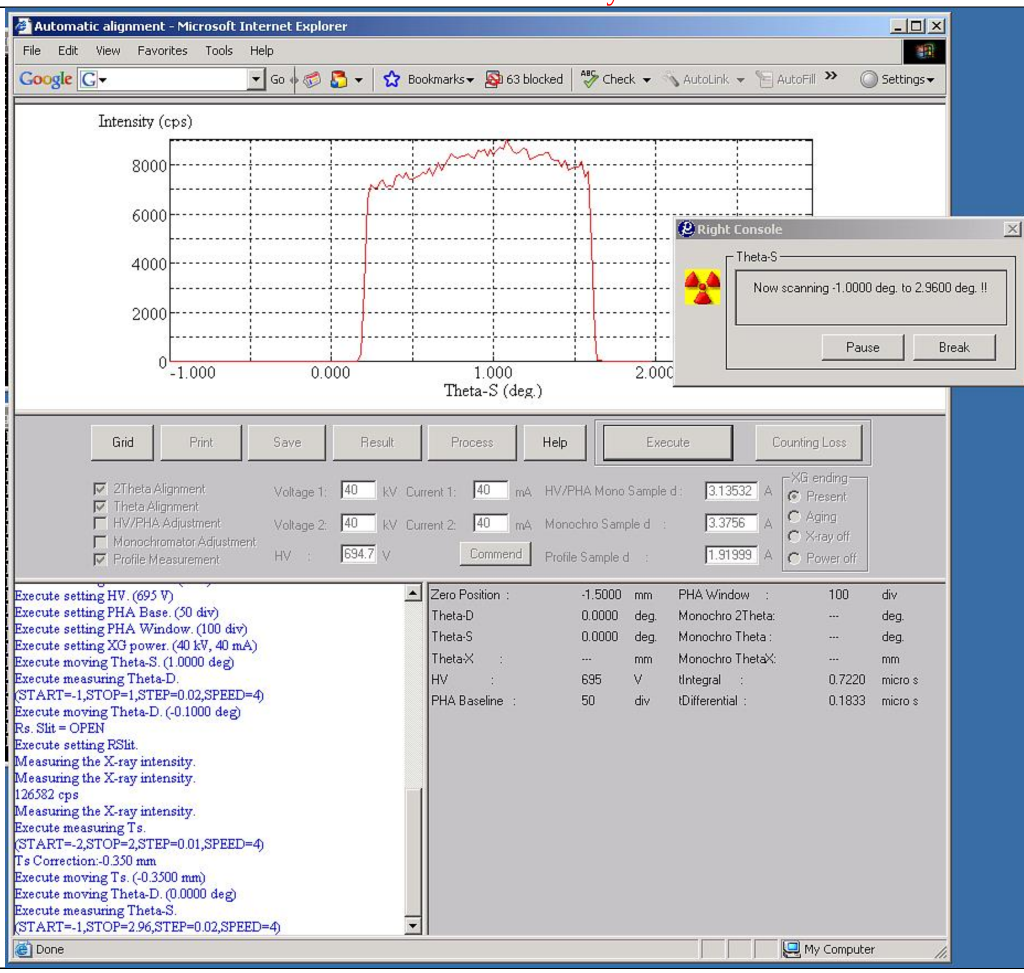


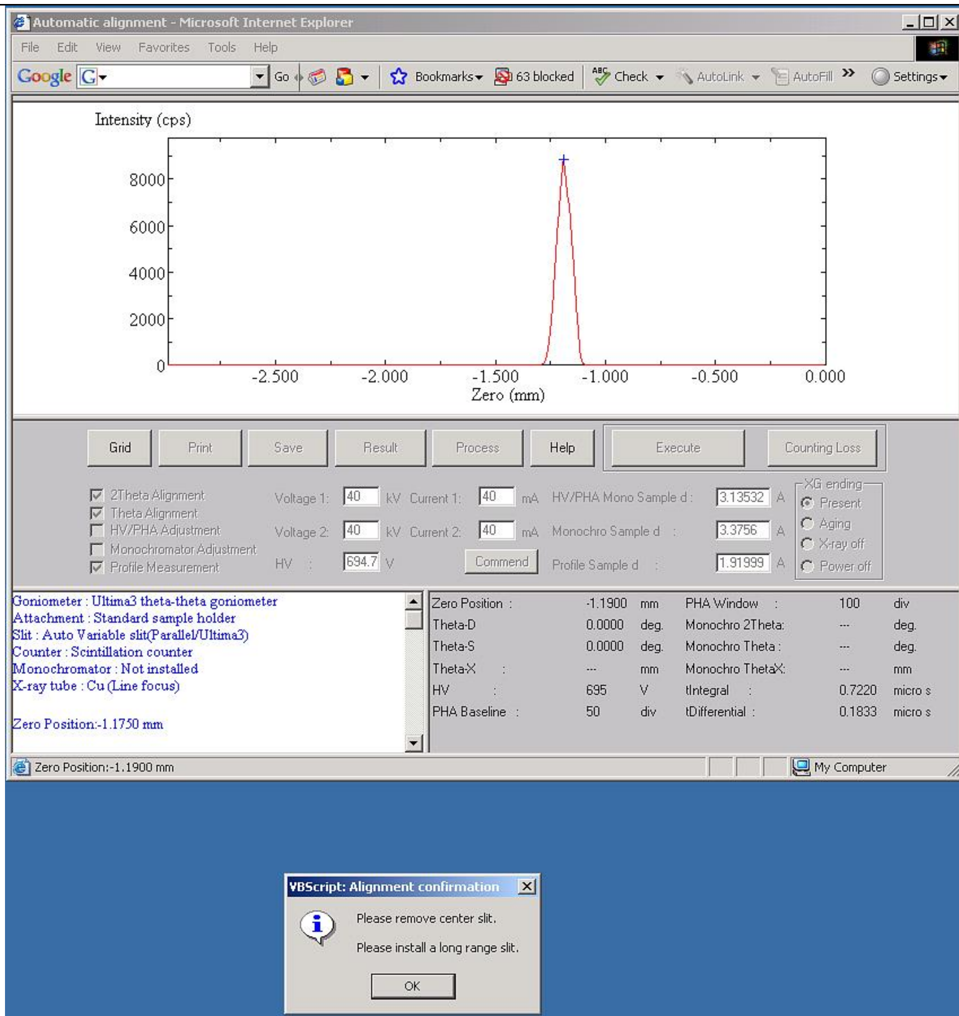
Center slit



Ni filter in the filter box

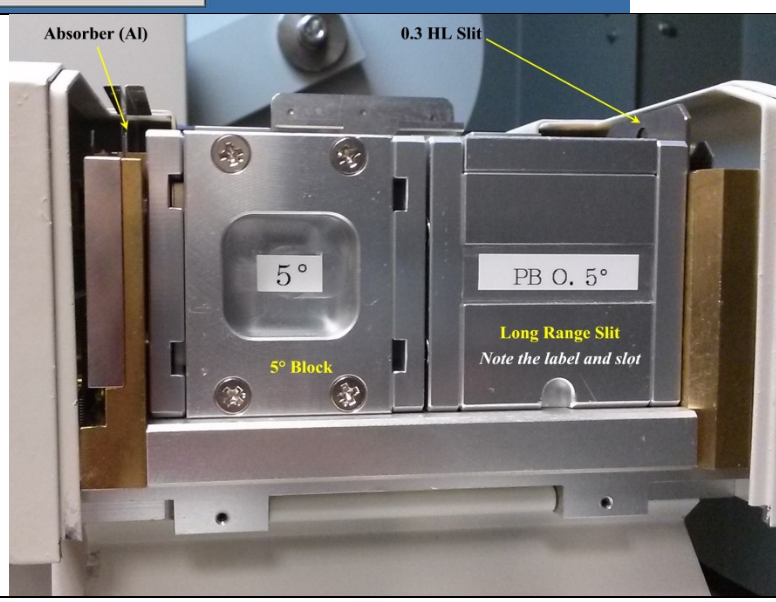
Now put the center slit in the standard sample holder and put the Ni-filter (aka K-beta filter) in the filter holder box. Click “OK” on the pop-up window to allow the measurment to continue. The first meaurement result will be on Zero Position and is shown below. There may be a Theta S Measurement after this!!!

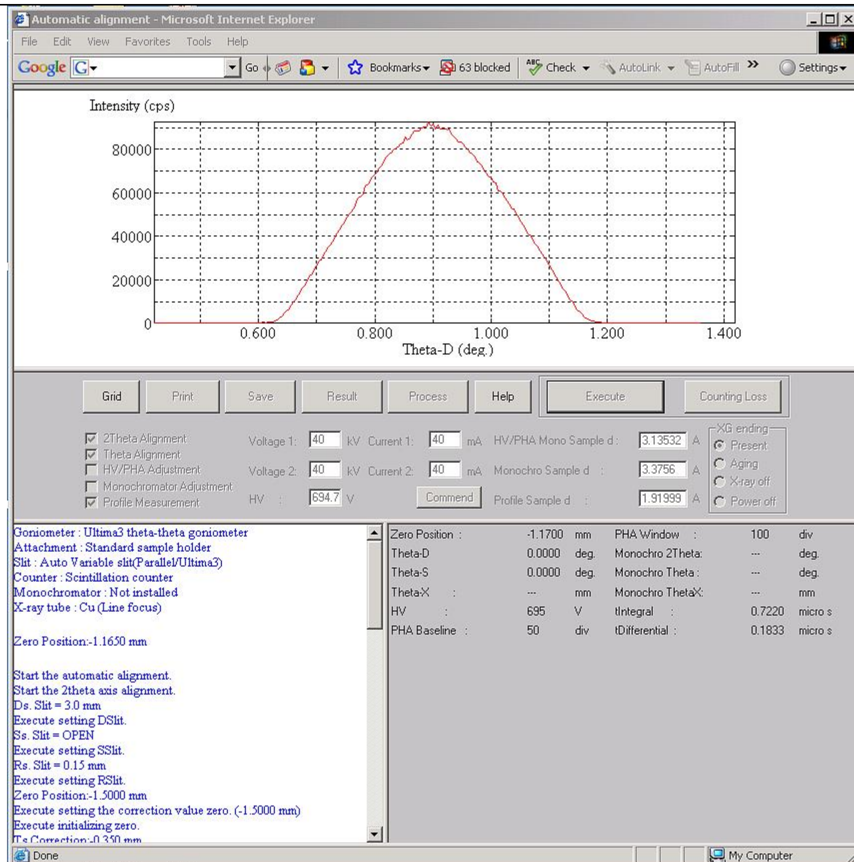
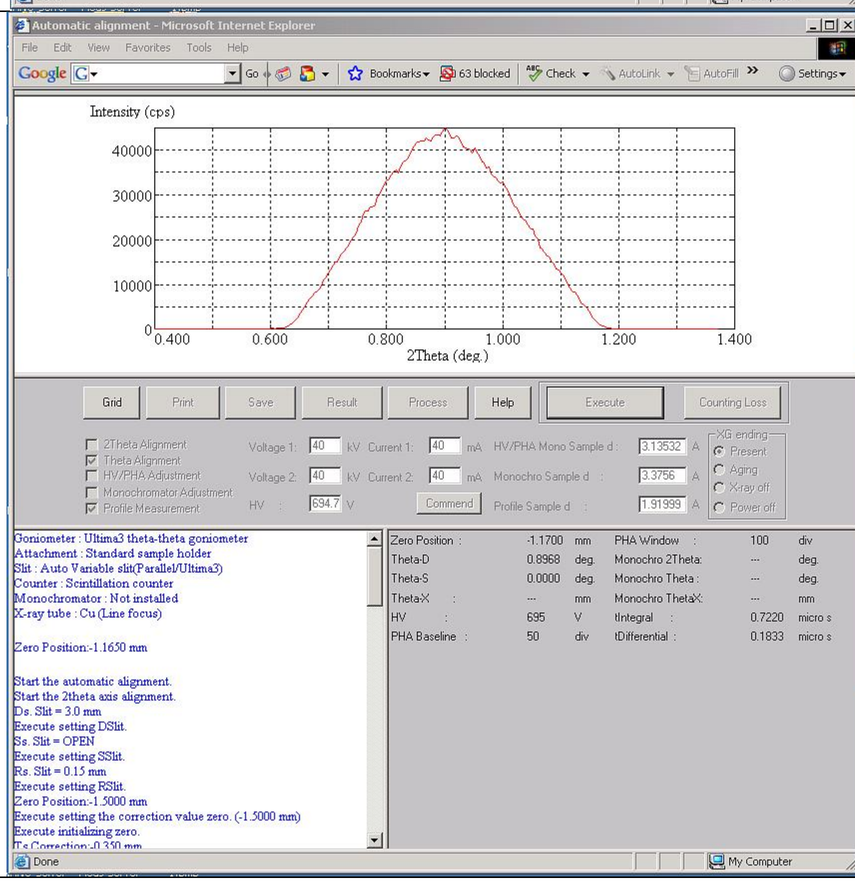




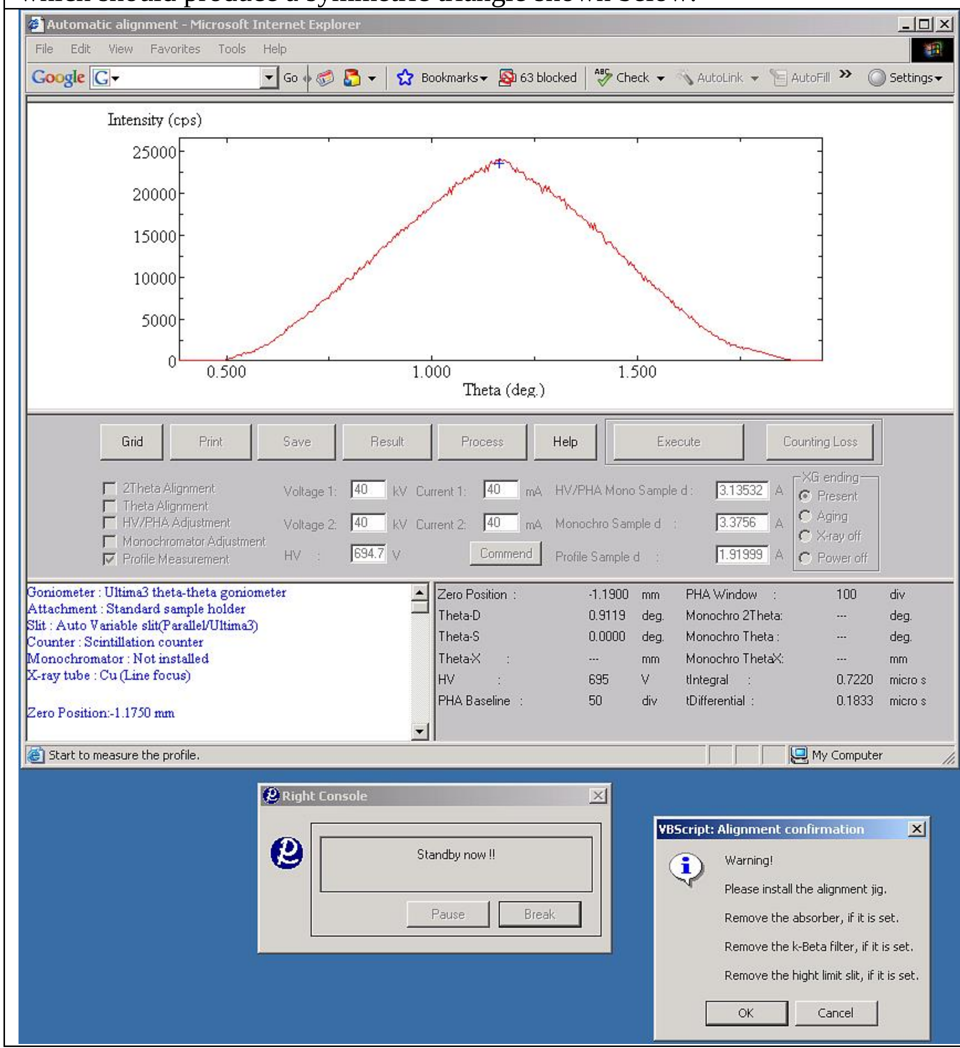
Now remove the center slit and put the long range slit in filter box.

- Note the correct orientation (as shown in the figure on the right) for the long range slit.
- Confirm the pop-up dialog to start new measurement.
- The htm program will perform Theata D correction
- The htm program will perform 2 Theata correction



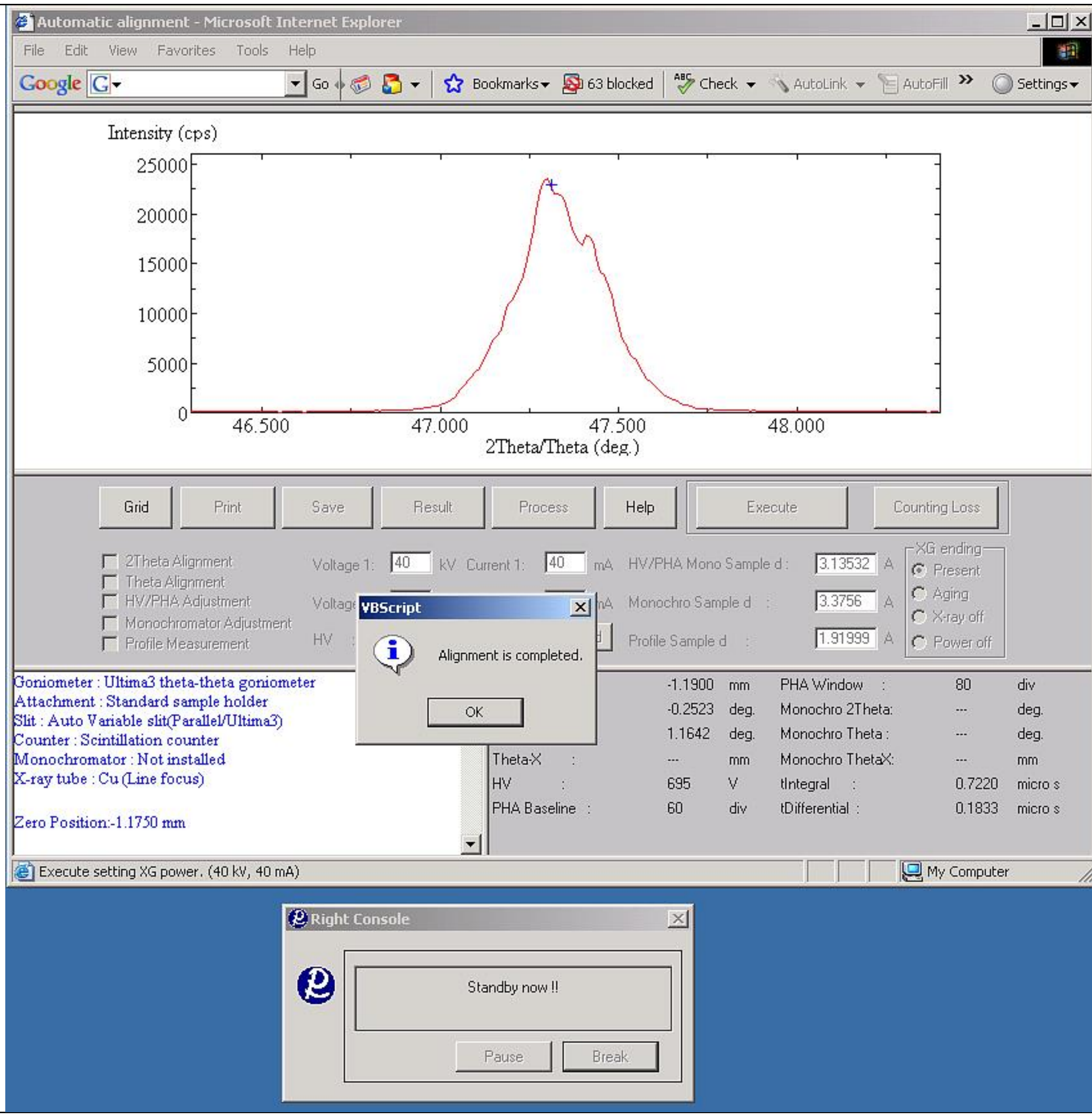
<table>
<tr>
<td>Theta D alignment
(correction)</td>
<td>
</td>
</tr>
<tr>
<td>2 Theta alignment
(correction)</td>
<td>
</td>
</tr>
</table>

Now install the alignment jig (actually it is labelled as Setting jig). A metal plate with standard compressed silicon (Si) powder in the standard sample holder. Confirm OK to proceed with next measurement of Theta which should produce a symmetric triangle shown below.



Now,

- Keep the setting jig
- Remove the Al foil, if present (Absorber)
- Remove the Ni foil, if present (Beta filter)
- Remove the hight liimiting jig (03. HL)
- Confirm OK to proceed to next measurement
- That will be final alignment



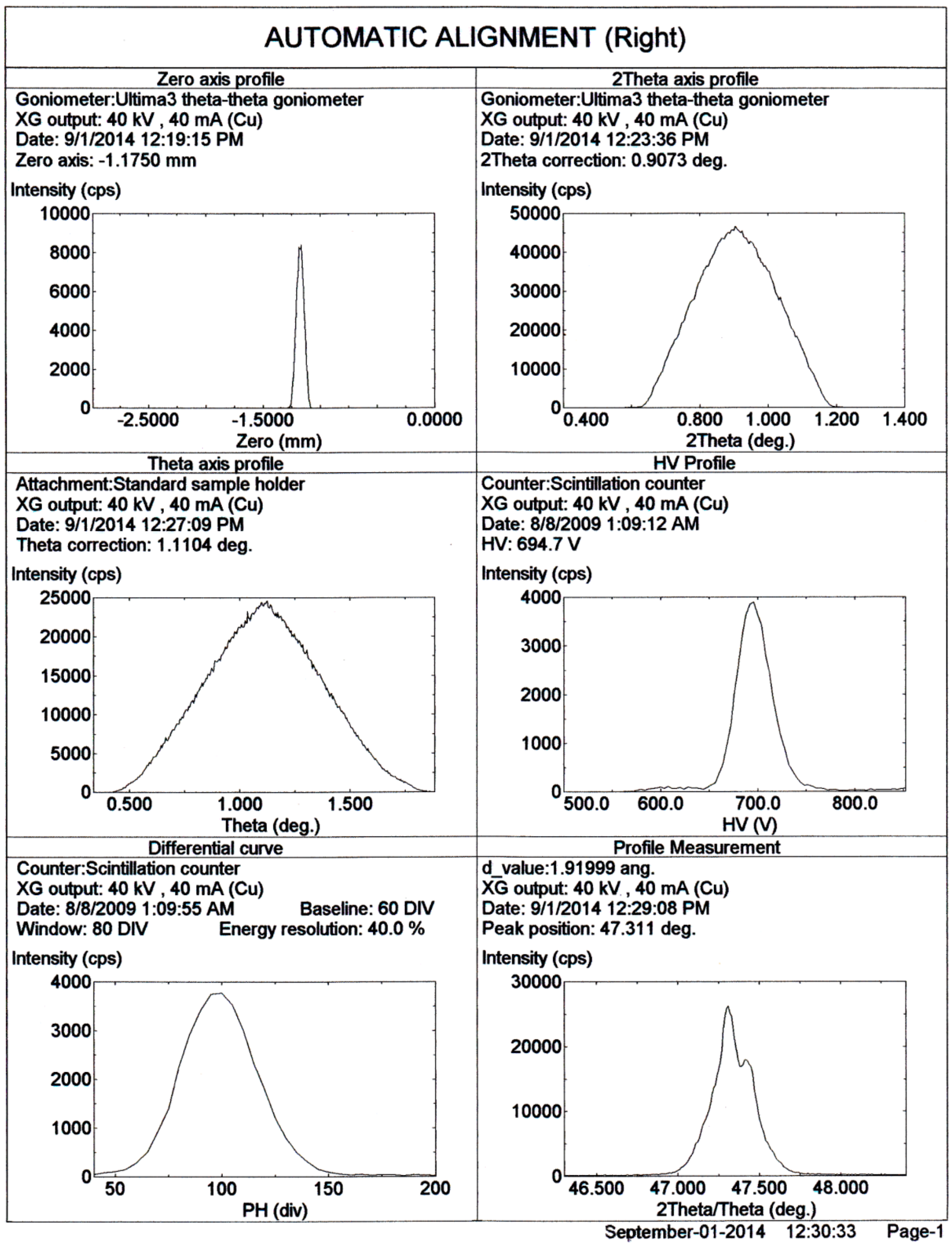
COMPLETE ALIGNMENT, SAVE & PRINT RESULTS

At his point the alignment is complete. However, <u>it is very important to "Save" the results</u>. Now the "Save" and "Print" buttons will become active. First, click on "Save" button. This saves the current alignment values.

Now scroll down the .htm file's left hand section and note the following values:

- **Ts**
- **Zero Position**
- **Theta D Correction**
- **Theta S Correction**

Now click on the "Print" button and select the "Registered Alignment Results". The printer on top of the cabinet will print a page similar to the one shown on next page. Write down the four values we got above on the sheet of paper and save it in the drawer.



Results obtained during the alignment procedure

SWAP THE EQUIPMENT BACK TO ORIGINAL CONDITION

Now having completed the alignment using the Scintillation Detector, we need to switch back to the original set-up with Mercury CCCD.

- First, we need to bring the XG operation back to 2o kV and 2 mA. This can be done using the XG Operation Program
- Now we need shut-down the X-Ray Generator by doing the following:
    - Press the grey "OFF" button below X-RAYS in the front panel of the generator
    - Next press the red "OFF" button below POWER in the front panel of the generator
    - Switch "OFF" Servo AMP Unit after opening the bottom left panel
- Shut-down the Control PC
- Remove the Scintillation Detector by doing the following:
    - Un-plug the Scintillation Detector cable
    - Un-plug the RJ1 cable
    - While holding the Scintillation Detector, undo the screws that hold it and gently remove it
- While holding the Filter Holder, undo the screws that hold it and gently remove it and remove all the components inside it. Store the components inside the cabinet above the Control PC.
- Now, remove the two (2) black pieces that were holding the Filter Holder and the Scintillation Detector and place them along with their screws inside the cabinet above the Control PC.
- Now, remove the Standard Sample Holder by doing the following:
    - Remove the three thumb screws that hold the Sample Holder
    - Gently wiggle the Holder and pull toward you until comes out
    - Put the Holder in its holding-base and store it inside the cabinet above the Control PC
- Next, attach the black piece that holds the CCD on 2-Theta arm and secure all six screws
- Next, while carefully holding the CCD place it in the black stripe's dove-tail and move the CCD until the mark on the top CCD matches the original distance before removing the CCD (in our case it was 105 mm)
- Now using the four Philips screws, while still holding the CCD with your one hand, fully secure the CCD the black stripe.
- Next, put the original MicroArea holder back in the middle of the goniometer (from where we just removed the Standard Sample Holder) by gently wiggling it inside the center hole
- Using the two hex screws secure the MicroArea holder ensuring the video camera is not pushed back or forward
- Swap the H10 slit with the original H2 in the CBO box
- Remove the CCD cover
- Close the X-Ray Cabinet door
- Switch the SERVO AMP unit power
- Now we need start the X-Ray Generator by doing the following:
    - Press the green "ON" button below POWER in the front panel of the generator
    - Wait for the READY green LED to light below the X-RAYS panel
    - Next, press the white "ON" button below X-RAYS in the front panel of the generator
    - Make sure, the EXT button on Shutter 2 is ON

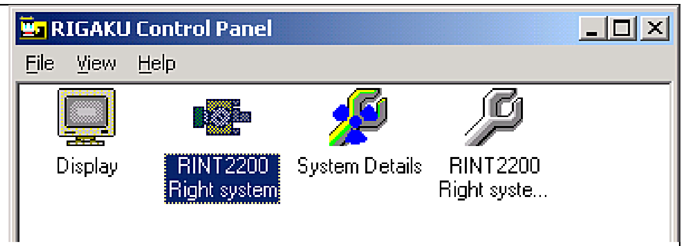
- Now restart the Control PC
    - Log-in to Control PC
- Start the Frame Grabber PC
    - Log-in to Frame Grabber PC

- Wait for the DOS window to come on
- Switch back to Control PC

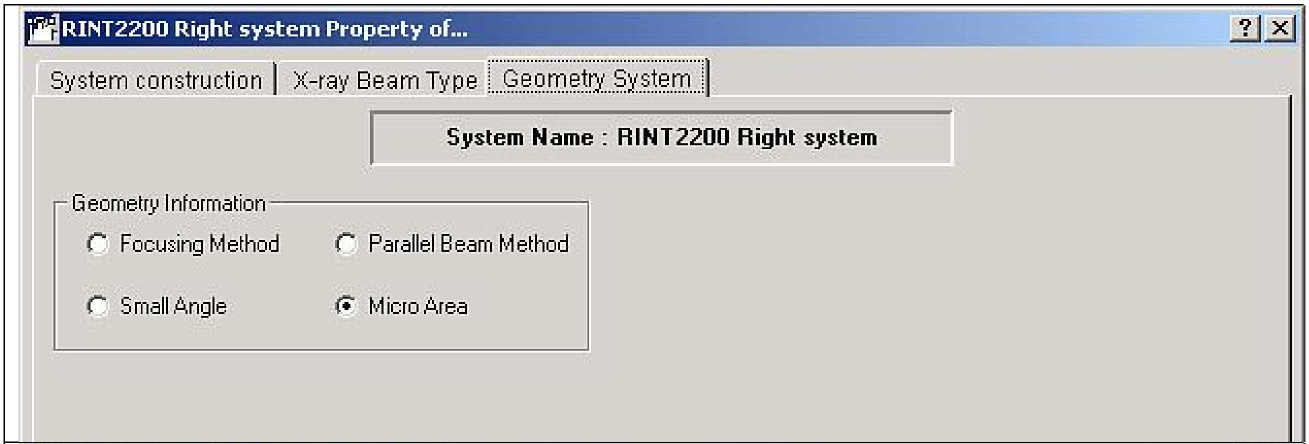
- In Control PC do the following:
  - Right Click RGHExpand
  - Under RINT2200, select, “Show Window”
    - This will open a RINT2200 control window
  - Under Right, select, “Show Window”
    - This will open a Right control window
  - Click open Rigaku Control Panel Directory
  - In System Details do the following:
    - Switch from XG Server Mode: Standard to Std (Shutter relay)
    - Click OK to confirm it
    - The Right Control window will update
  - Now click open the RINT2200 button in the Rigaku Control Panel Directory
  - Now do the following switch backs:

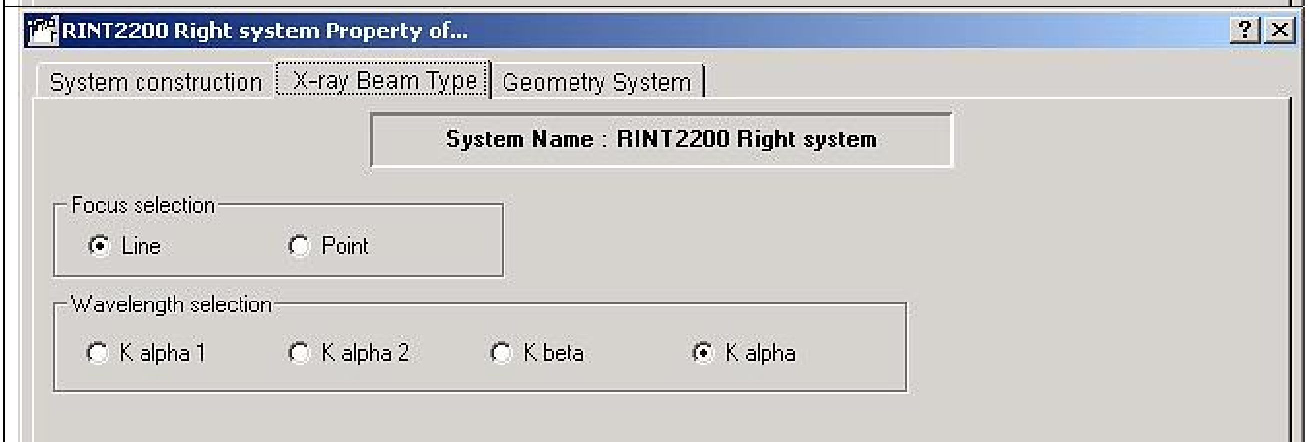
Now click on the RINT2200 Right System (but not the ~~RINT2200 Right System Add/Delete button~~). This should open a new dialog box. In the following three tabs RINT2200 Right System Property need to be changed.

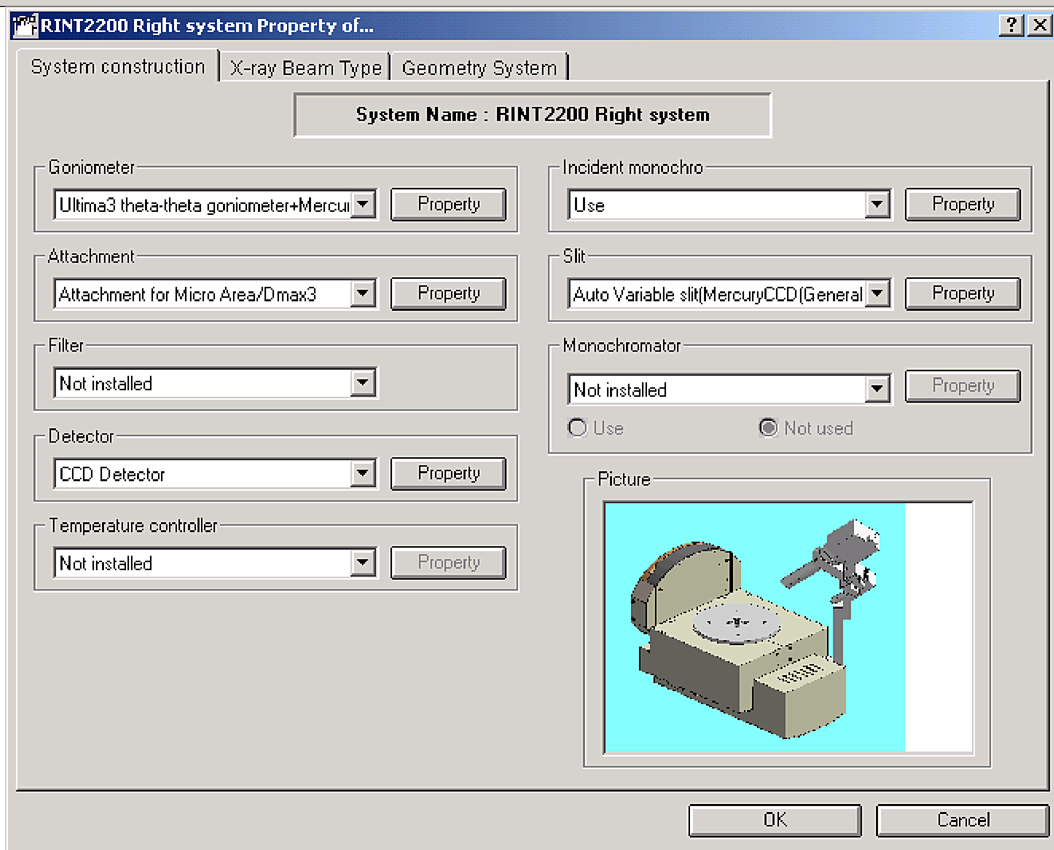
- System Construction
- X-Ray Beam Type
- Geometry System



System Construction	
CHANGE FROM	CHANGE TO
Goniometer: Ultima 3 theta-theta goniometer	*Goniometer*: Ultima 3 theta-theta goniometer+Mercury CCD
Attachement: Standard Sample Holder	*Attachment*: Micro area
Filter: K-beta filter	*Filter*: Not installed
Detector: Scintillation Counter	*Detector*: Mercury CCD
Temp Contoller:Not installed	*Temp Contoller*:Not installed
Incident Monochromateor: use	*Incident Monochromateor*: use
Slit: Auto Variable Slit (Parallel/Ultima3)	*Slit*: Auto Variable (Mercury CCD/general)
Monochromator: Not installed	*Monochromator*: Not installed
X-Ray Beam Type	
CHANGE FROM	CHANGE TO
No change. *Focus Selection*: Line; *Wave length selection*: K-alpha	
Geometry System	
CHANGE FROM	CHANGE TO
Geometry Information: Parallel Beam	*Geometry Information:* Micro Area
After the change the three tabs should like the following	


RINT2200 Right system Property of...
System construction
X-ray Beam Type
Geometry System
System Name : RINT2200 Right system
Geometry Information
Focusing Method
Parallel Beam Method
Small Angle
Micro Area


RINT2200 Right system Property of...
System construction
X-ray Beam Type
Geometry System
System Name : RINT2200 Right system
Focus selection
Line
Point
Wavelength selection
K alpha 1
K alpha 2
K beta
K alpha


RINT2200 Right system Property of...
System construction
X-ray Beam Type
Geometry System
System Name : RINT2200 Right system
Goniometer
Ultima3 theta-theta goniometer+Mercu
Property
Incident monochro
Use
Property
Attachment
Attachment for Micro Area/Dmax3
Property
Slit
Auto Variable slit(MercuryCCD(General
Property
Filter
Not installed
Monochromator
Not installed
Property
Use
Not used
Detector
CCD Detector
Property
Picture
Temperature controller
Not installed
Property
OK
Cancel

- Click OK
- Click Yes to confirm it

SUMMARY

This procedure is what is being followed at Florida State University Chemical Sciences Laboratory Powder X-Ray Diffraction Facility's Rigaku Ultima III w/ Mercury CCD. It is likely that your procedure is slightly different from mine. If you have questions, please contact me at tsomasundaram@fsu.edu or at 1-850-644-6448.

P.S. The procedure is still beta version 0.9.6 and needs minor tweaks especially toward the last stage of restarting the CCD. I will update this procedure in early 2015 after redoing the alignment couple of more times and taking precise notes.

Version 0.9.6 dated November 18, 2014

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