

# Instructions for use of Siemens D5000 (Co) Diffractometer

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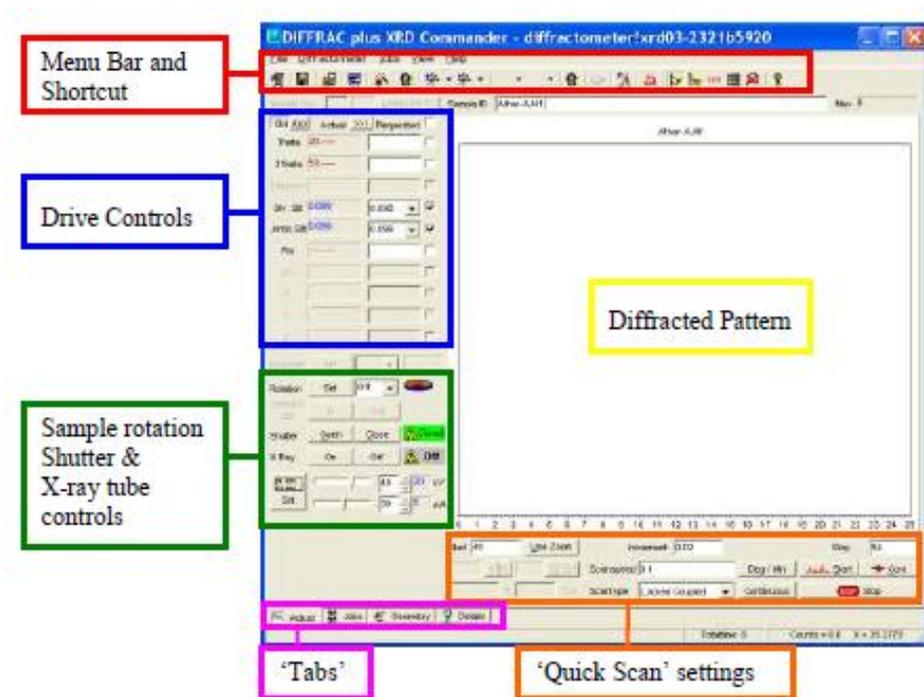
## Section 1: POWER ON?

Check the current status of the generator.

- The machine is already on if:
  - The X-rays On light is lit
  - The digital display on the front of the machine does NOT read 0 kV, 0mA. If the machine is already on, proceed to [Section 2](#). If the machine is not on, then:
- If the machine is not already on:
- Make sure that the water supply is on, and that the power is turned on at the wall-mounted isolator switch.
- Make sure that the PC is on, and that the 'XRD Commander' software is NOT running. If it is running, close it.
- Flick the green 'Mains' switch to on, if not already.
- Turn the Key to the I position, if not already.
- Switch generator to standby mode by pressing the WHITE button. A buzzing alarm will sound if water flow is insufficient.
- Pull out the keypad. Press 'Shift' '6' '1' and then 'Enter'
- Press the GREEN button to start the generator.
- You will probably hear the machine trip – this is normal for this type of generator. Press the WHITE and GREEN buttons again.
- The generator should now be at the minimum settings of 20kV and 5mA.
- Using the black square buttons on the front left panel of the D5000, hold the MODE button and press the ↑ arrow once. Turn the kV up to 40 by holding the EDIT button and repeatedly pressing the ↑ arrow.
- Hold the MODE button and press the ↑ arrow once. Turn the mA up to 30 by holding the EDIT button and repeatedly pressing the ↑ arrow. Hold MODE and press the ↑ arrow twice to return to the main display.
- On the PC, check to see if the 'XRD Commander' software is running already; NEVER HAVE MULTIPLE COPIES OF XRD COMMANDER RUNNING. If it is not already running, start it by double-left clicking on its icon on the desktop.
- Proceed to [Section 2](#).

## Section 2: INITIALIZATION

The 'XRD Commander' software is more powerful than is present on our older instruments. This also makes it a little more complex, but it's straightforward:



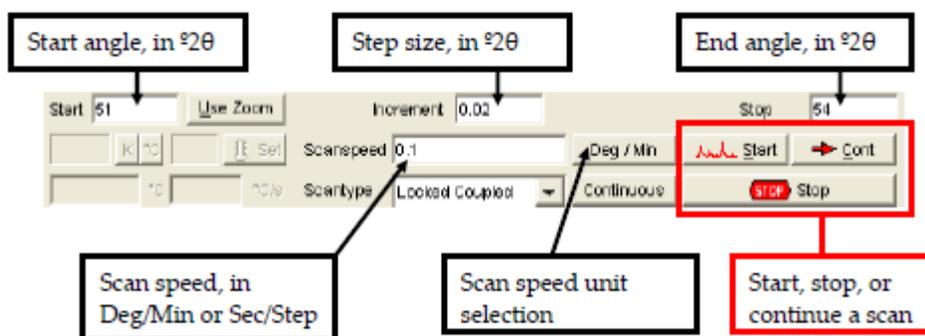
Before we use the D5000, we must first initialize it. To do this:

- In the 'Drive Controls' area, tick the 'Requested ' box to select all drives.
- In the 'Menu Bar', click 'Diffractometer', 'Init Drives'. Untick the 'Requested '.
- The D5000 has variable Divergence and Antiscatter Slits – setting these wider increases the area illuminated by X-rays, at the risk of exposing the sample holder:
- Tick the two  boxes for these slits, as shown above. Enter Slit Size values appropriate for your sample (see [Section 7](#) for suggestions) by typing the desired sizes in the boxes; both slits should be set to the same value.
- In the top 'Menu Bar', click 'Diffractometer' and select 'Move Drives'. Untick the '' boxes for the Div. and Antis. Slits.

You are now ready to start – see [Section 3](#) for a quick scan or [Section 4](#) for a 'Job'

### Section 3: RUNNING A QUICK SCAN

Though the easiest way to run an experiment on the D5000, YOUR DATA WILL NOT BE AUTOMATICALLY SAVED AT THE END OF THE SCAN. If you want to be sure your data is saved at the end of the scan, skip this section and go straight to [Section 4](#). With that in mind, to run a quick scan first make sure you have completed [Section 2](#) to initialize the machine. Next, in the left hand panel of the ‘Adjust’ tab, choose the Rotation speed (if any) and click ‘Set’. Ensure the X-ray generator sliders are at 40kV and 30mA and click ‘Set’. You can enter the settings for a quick scan in the bottom panel of the ‘Adjust’ window in ‘XRD Commander’. This is largely self-explanatory:



Enter appropriate values in each area and press ‘Start’ when ready:

- For phase analysis work, a ‘Start’ angle of ~ 5-20 °2θ, Stopping at 60-80 °2θ, is adequate. ‘Scanspeed’ should be between 0.1 (will take many hours) and 2 (much faster) Deg/Min. For phase analysis work, faster scans should be OK.
- Increment: 0.02 is a good standard, though 0.05 may be adequate for phase analysis work. 0.01 for unit cell refinement or residual stress analysis.

REMEMBER: Save the data at the end of the scan:

- Click File and Save As. Choose the Bruker AT version 2.0 format.

(Other useful definitions:

- Scantype: for regular  $\theta$ :2 $\theta$  work, choose the default ‘Locked Coupled’ We can also alternate between a Continuous or Step Scan at the bottom.)

## Section 4: RUNNING A JOB (not currently working)

It is also possible to set up experiments in advance using the 'XRD Wizard' software program, even if 'XRD Commander' is running.

NOTE: NEVER HAVE MULTIPLES OF XRD COMMANDER RUNNING!

If you have previously created a \*.dql file that you wish to re-use, skip to [Section 4b](#)

### Section 4a: Using XRD Wizard to set up an experimental control (\*.dql) file

From the Desktop, start 'XRD Wizard'

- Click on to the 'Quick Edit' tab.
- Enter the Sample ID, and any Comment.
- Check the Scantype (for regular  $\theta:2\theta$  work, choose 'Locked Coupled')
- Check Scan Mode (Continuous or Step Scan)
  - There is not much difference between the two modes. 'Continuous' is kinder to the machine's gearing. Step scan is better for high quality work, i.e. data for Rietveld analysis, lattice parameter calculation, stress analysis.
- Enter the desired 'Scan Definition':
  - Enter start angle, in  $^{\circ}2\theta$ .
  - Enter stop angle, in  $^{\circ}2\theta$ .
  - Enter step size (0.02 is a good standard, though 0.05 is often adequate for quick, good quality data for phase analysis work. Use the minimum possible value, 0.01, for unit cell refinement or residual stress analysis).
  - Enter the desired scan rate – this has to be in seconds/step (see [Section 8](#) for help on converting between degrees/minute and seconds/step if necessary). Make sure you will have enough time to complete your experiment in your allotted time.
- Set appropriate values for the 'Motorized Slits':
  - The D5000 has variable Divergence and Antiscatter Slits – the wider these are set, the larger the area illuminated by X-rays. See [Section 7](#) for slit sizes appropriate to your sample / sample holder size, and enter your chosen values.
- Set 'Sample Rotation' On or Off as desired.
  - If set to On, enter the rotation speed (0 – 15 rpm)

- Click 'File', then 'Save As' to save your \*.dql file. This is the control file that tells the diffractometer how to perform the experiment – it is NOT specific to the sample; i.e. you could set up one \*.dql file for all phase analysis scans, another for stress measurements, etc.

If you wish to run a multi-segment scan, click 'Add' in the 'Ranges' area, and repeat the steps above as appropriate before clicking 'File', then 'Save As'.

NOTE: These \*.dql files have nothing to do with your sample! They merely tell the machine how to perform the experiment. You can re-use the same \*.dql files over and over again!

### **Section 4b:** Setting up a Job in XRD Commander

In 'XRD Commander':

- Complete [Section 1](#) and [Section 2](#) to ensure the diffractometer is on, and initialised.
- Click the 'Jobs' tab.
- Click 'Create Jobs' (button with four small blobs on, or from 'Jobs' menu)
- Leave 'Pos' blank
- Enter a Sample ID, for reference.
- In the Parameters column, click the '...' button and navigate to your experiment's \*.dql file, created in 'XRD Wizard'.
- In the 'Raw Data' column, click the '...' button and navigate to where you wish to save your data. Type in your desired File Name, and click 'Open'.
- Click 'Create' to add your Job to the list.

To begin the scan, highlight your Job and click 'Start' on the 'Jobs' tab. If the drives need to be initialized, the software will prompt you straight away – simply press OK to confirm and the experiment should start.

The diffraction pattern will then be displayed as it is collected on the 'Adjust' tab.\

## Section 5: TURNING THE DIFFRACTOMETER OFF

The diffractometer is in use 24/7, and accordingly should always be left running with the 'XRD Commander' program running and ready for the next user. The D5000 has automated power control systems which will turn the generator down to lower settings to save power and preserve the X-ray tube.

However, if the machine does need to be switched off for any reason:

- In 'XRD Commander', stop any scans which may be running (either by clicking 'Stop' on the Adjust tab or if this is greyed out by clicking to the Jobs tab, selecting the Active job and pressing the stop button).
- Using the black square buttons on the front left panel of the D5000, hold the MODE button and press the ↑ arrow twice. Turn the mA down to 5 by holding the EDIT button and repeatedly pressing the □ arrow.
- Hold the MODE button and press the ↓ arrow once. Turn the kV down to 20 by holding the EDIT button and repeatedly pressing the ↓ arrow. Hold MODE and press the ↓ arrow to return to the main display; the generator should now be at the minimum settings of 20kV and 5mA.
- On the front of the diffractometer, press the square yellow button.
- On the front of the diffractometer, turn the key to the 'O' position.
- On the front of the diffractometer, toggle the green Netz/Mains switch to the 'O' position.

If necessary, also switch off the power completely at the isolator switch to the right behind the machine. After allowing 5 minutes for the X-ray tube to cool, you can also turn off the water supply (unless there is suspected to be a serious problem with the water cooling system, i.e. a significant leak).

## Section 6: D5000 FILE FORMATS

It should be noted that the XRD Commander software will save data in a variety of Siemens \*.raw formats – NOTE: these are NOT the same as the STOE \*.raw format.

For this reason, it may be appropriate to have 1 folder for your raw data, and 1 for your imported data – otherwise things will get pretty complicated!

Siemens \*.raw files can still be imported into the STOE WinXPOW software in the usual way:

- Go to Raw Data / Raw Data Handling
- Click File / Import
- Change to 'Files of Type' = Diffrac Raw Files
- Select your Siemens \*.raw file
- Save, WITH A DIFFERENT FILE NAME, as a STOE \*.raw file
- Click Parameter / Wavelength, and change the wavelength to Cobalt.
- Click File / Save As, and overwrite the STOE \*.raw file.

The STOE Win XPOW software is not available for users to install on their own PCs.

## Section 7: TROUBLESHOOTING

\* If an alarm is sounding and the detector drive lost its reference position, or has hit a limit switch (i.e. if the **DETECTOR** appears to be at a very high or low (sub-zero) angle):

- On the machine keypad, press 'Shift', 'Circle', '1', ':', '3' and then 'Enter'. If the machine beeps on keystrokes, try turning off ([Section 5](#)) and back on first ([Section 1](#)).
- Press and hold the central TUNING key; then press several times and hold either the '□' key or the '◻' key to move the drive clockwise or anticlockwise respectively.
- Once the reference point is passed, perform the initialization procedure in [Section 2](#).

\* If an alarm is sounding and the detector drive lost its reference position, or has hit a limit switch (i.e. if the **TUBE** appears to be at a very high or low (sub-zero) angle):

- On the machine keypad, press 'Shift', 'Circle', '1', ':', '1' and then 'Enter'. If the machine beeps on keystrokes, try turning off ([Section 5](#)) and back on first ([Section 1](#)).
- Press and hold the central TUNING key; then press several times and hold either the '□' key or the '◻' key to move the drive clockwise or anticlockwise respectively.
- Once the reference point is passed, perform the initialization procedure in [Section 2](#).

\* If the machine attempted to start a new Job, has moved to the start angle, but now appears to be frozen:

- Check the adjust tab: has an intensity scale (i.e. numbers) appeared on the left hand side of where the plot should appear. Are the figures in the Drive Controls pane changing at all? If no, then:
  - Close XRD Commander. Ignore any error messages.
  - Switch off the diffractometer ([Section 5](#), skipping any instructions that relate to XRD Commander)
- Switch the diffractometer back on ([Section 1](#)).

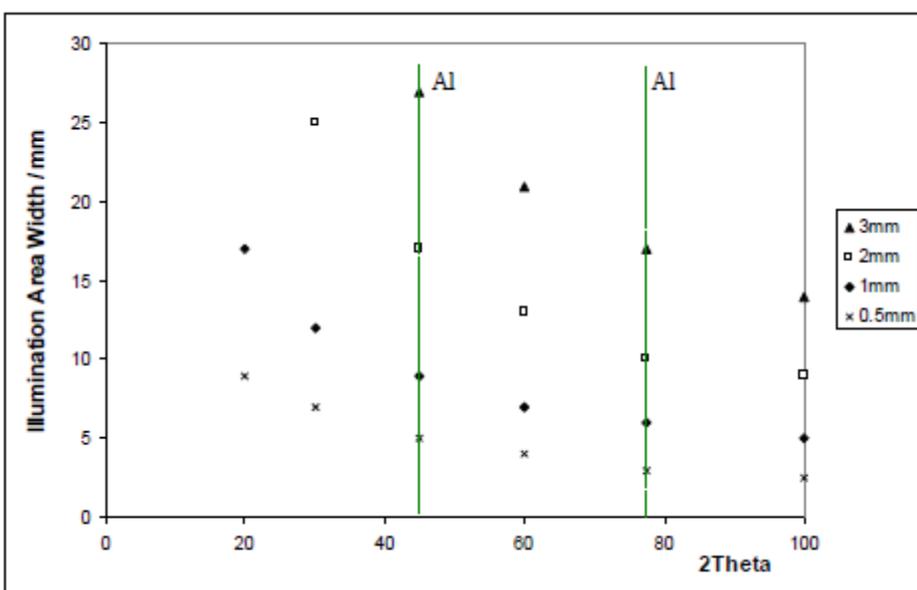
\* When starting a new run, the machine returns the Error message "Unable to send the kV to the Generator!" with Errorcode 400:

- Switch the diffractometer back on ([Section 1](#)), starting at the part where instructed to press the white button.
- Start your scan again.

## Section 8: CHOOSING SLIT SIZES

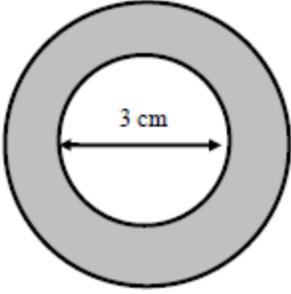
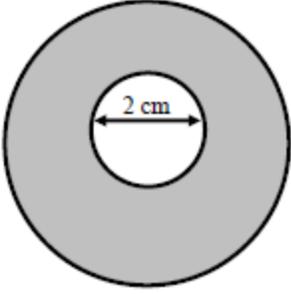
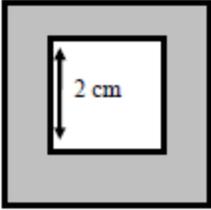
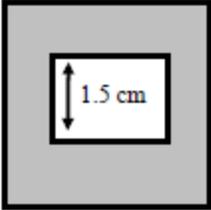
The D5000 has variable Divergence and Antiscatter Slits – setting these wider increases the area illuminated by X-rays, meaning that more of your sample can diffract and that the intensities in your diffracted pattern will be increased. However, this comes at the risk of exposing the sample holder.

The area illuminated by X-rays is always around 14-15 mm deep (i.e. front to back as you look at the machine), but the width (i.e. distance side to side) covered varies dramatically with both  $2\theta$  and slit size, as shown in this graph:



One wants to avoid getting peaks from the sample holder (Some aluminum peak positions are marked with green dotted lines in the graph above). You should also avoid overspill at lower angles – this will lead to lower than expected intensities, which may make analyses difficult.

We have four standard Aluminum holders in the XRD Lab suitable for the D5000. Assuming the sample is placed centrally, you should not observe any peaks from the aluminum holder when using the following slit sizes:

| Sample Holder<br>Used   | Maximum slit size permissible |                     |
|---|-------------------------------|---------------------|
|   | Rotating sample               | Non-rotating sample |
|  <p>3 cm</p>     | Up to 2.5mm                   | Up to 2.5mm         |
|  <p>2 cm</p>     | Up to 1.7mm                   | Up to 1.7mm         |
|  <p>2 cm</p>    | Up to 2mm                     | Up to 2mm           |
|  <p>1.5 cm</p> | Up to 1.25mm                  | Up to 2mm           |

## Section 9: SCAN RATE CONVERSIONS

One of the 'enforced' changes brought about by our use of the XRD Commander software on the new Siemens D5000 is that more often we have to enter our scan rate in seconds/step, rather than degrees/minute.

It's very easy to convert between the two, however, using the equation:

$$t = 60s/r$$

Where:  $t$  = scan rate, in seconds/step

$s$  = step size, in degrees

$r$  = scan rate, in degrees/minute

So, for example:

| STEP SIZE /<br>Degrees 2 $\theta$ | SCAN RATE   |              |
|-----------------------------------|-------------|--------------|
|                                   | Degrees/min | Seconds/Step |
| 0.01                              | 0.1         | 6            |
|                                   | 0.5         | 1.2          |
|                                   | 1           | 0.6          |
|                                   | 2           | 0.3          |
| 0.02                              | 0.1         | 12           |
|                                   | 0.5         | 2.4          |
|                                   | 1           | 1.2          |
|                                   | 2           | 0.6          |
| 0.05                              | 0.1         | 30           |
|                                   | 0.5         | 6            |
|                                   | 1           | 3            |
|                                   | 2           | 1.5          |

## References

BASED ON:

Instructions for use of Siemens D5000 (Co) Diffractometer

Taken from:

<http://uspace.shef.ac.uk/servlet/JiveServlet/previewBody/35614-102-2-91268/Siemens%20D5000%20Instructions.pdf>

Modified by: Blake Gaspar