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|  | SAFE WORK PROCEDURE: | Doc No. | | SWP-COE-12 | | |
| | | Last Rev/Date | | 00 | 31 Mar 2010 | |
| | X-ray Diffraction system-MRD- XRD | Current Rev/Date | | 00 | 31 Mar 2010 | |
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1. Application to:

All X-ray scans using the PanAlytical MRD X-Ray Diffractometer.

2. Pre-requisite for Operation of XRD system:

- 2.1. Must have attended Safety Orientation.
- 2.2. Must have read and understood the operation procedure
- 2.3. Must possess the NEA L5 license
- 2.4. Must be qualified user before using the system.

3. Hazards that may be present:

- 3.1. Risk of X-ray exposure.
- 3.2. Risk of Electrocutation due to high tension.

4. Personal Protection Required:

- 4.1. Gloves for handling samples
- 4.2. NEA's TLD badge for registering any X-ray radiation

5. Procedures:

5.1. Operating the XRD

- 5.1.1. On the sample stage, paste the sample at the centre of the stage.
- 5.1.2. On the computer screen, click on 'X-pert Data Collector'.
- 5.1.3. Type in the user name and password
- 5.1.4. Click on 'Instrument'
- 5.1.5. Click 'connect'
- 5.1.6. Select 'Rocking curve'
- 5.1.7. Click 'OK'
- 5.1.8. Select 'Yes' to retain the saved offset values
- 5.1.9. Click on any of the parameters.
- 5.1.10. Set all the dimensional parameters to '0'
- 5.1.11. On the X-ray button, turn on the X-ray.
- 5.1.12. Conduct a quick goni scan around the '0' position
- 5.1.13. Zero the scan when completed if the peak is not at the '0' position

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- 5.1.14. When the scan is zeroed, increase the 'z' position to a position such that it is half of the counts of the peak value
- 5.1.15. Set the 2θ to the desired position.
- 5.1.16. Increase the Tension and Current slowly in steps of 10 to 40 KV and 40 mA respectively.
- 5.1.17. Conduct a 2θ scan , align the green line to the highest point of the peak.
- 5.1.18. Repeat for Omega Scan
- 5.1.19. Repeat for Phi Scan
- 5.1.20. Repeat for Psi scan
- 5.1.21. With all the parameters optimized, conduct a fine scan for the desired rocking curve
- 5.1.22. When the scan is completed, save the file to the D drive

5.2 Maintenance of the XRD system

- 5.2.1 Check that the water flow of the chiller is above 3.5 litres per min
- 5.2.2 Ensure that the fan for the machine CPU is working
- 5.2.3 Ensure that the 'X-rays on' light is on when the machine is running
- 5.2.4 Ensure that the XRD crater moving arm is not obstructed when in operation

5.3 Specific Processes

N.A.

6. Communication Frequency:

The Lab Supervisor shall arrange for this Safe Work Procedure to be communicated every once a year and as & when required.

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