

INDIAN INSTITUTE OF TECHNOLOGY KANPUR

DEPARTMENT OF CHEMICAL ENGINEERING

PG Research lab

STANDARD OPERATING PROCEDURE

XPRT POWDER (XRD)

START UP PROCEDURE

- 1 3 Phase supply to be on.
- 2 Compressor and chiller switch to be on. Open Compressor outlet valve
- 3 XRD machine power to be on.
- 4 Power switch to be on at XRD panel.
- 5 After default value ($2\theta=5^\circ$) display, key to be moved in clockwise at 90 degree ($\frac{1}{4}$ circle) in right side.
- 6 Wait for HT LED on and wait for initial default readings (30 kV, 10 mA)
- 7 Keep sample ready (in proper sample holder)
- 8 Open Door by pressing Door open button
- 9 Load sample in sample port and close the door.

- 10 Computer to be switched on
- 11 Data collector software to be open.
- 12 User name (User-1) and password (galaxy) to be login.
- 13 Instrument to connect by instrument connect option.
- 14 Start Breeding Procedure –
Normal Breeding – If instrument was switched off for >2 Days (takes 45 mins)
Fast Breeding –If instrument was in ran within last 2 days (takes 15-20 mins)

BREEDING PROCEDURE

- 14.1 From left side bar under X-Ray > Double click on Generator MPPC
- 14.2 Instrument settings dialogue box opens > Click on X-Ray
- 14.3 Select Breed > Tube Breeding window opens > Select Normal of Fast as per 1.14 above
- 14.4 Click ok.
- 14.5 After Completion of breeding instrument is ready for switch on

- 15 Click on measure and click on switch on procedure.
- 16 After completion of switch on procedure display should be 45 kV and 40 mA
- 17 Wait for Measurement status to be No Program Executing. Close the dialogue box
- 18 Ready for analysis.

ANALYSIS PROCEDURE

From Menu bar

File > Open Program>Select 'Power Scan FDS'

Change A. Start Angle

B. End Angle

C. Scan Speed in Degree/min

Close the window after completion of entering above parameters

From Menu bar

Measure> Program>Select Program 'Power Scan FDS'

Change file name and path (C:\XRD\Data\Panalytical)

Click Ok – Execution under progress status should come

SHUT DOWN PROCEDURE

1. Unlock doors and take out samples.
2. From menu bar > Measure > Program > Switch off procedure
3. Click open
4. After completion steps > Instrument disconnect > exit software.
5. Move key quarter anticlockwise.
6. Power off panel.
7. Switch off XRD (by pressing power off button)
8. Switch off chiller.