

# Short instructions for the JEOL JSM-5800

## WARNINGS

- Move the joystick if you want to stop the movement!
- Read the manufacturer's instructions!
- Use washers to raise the sample.

## Initial settings

- Keyboard lights: **X-Y, SL 1 and TONE**.
- **Monitors are off (first in the day)** and dimmed.
- LCD monitors off (imaging and EDS).

## Sample Preparation

- Put your prepared samples in the holder (sleds), tighten the screws. All the samples are on the same height!
- Check that it doesn't fall out.
- **If you have offset – be careful with lifting the table!**

## Inserting the sample

- Hold »VENT« for 2s (gently).
- When it stops blinking and you hear a click open the door.
- Insert the sleds (straight edge forward).
- Clean the O-ring.
- Close the door and hold 2s "EVAC".

## Start of work

### The parameter settings (PRE VAC or EVAC)

- **First in the day: turn on the monitor.**
- Illuminate the monitor (potentiometer B and C).
- Click the tab »STAGE F5«:
  - Choose your holder (e.g.  $\Phi$  51).
  - **Click »INIT POZ« unless you have offset!!!**
- Click »MENU F2«:
  - »SETUP«, where you select »Data print«.
  - »ACCV« select voltage (e.g. 20 kV).
  - »IMS« choose »SEI«.
- Click »SPOT SIZE« (standardly set to 9 (8+1), if necessary, change it).
- Click »TONE«.

### Secondary electrons imaging (»HT READY«)

- Turn ON and **OFF** the vacuum gauge.
- Turn down to a minimum magnification.
- Wait for the pressure to be  $< 5 \cdot 10^{-4}$  mbar.
- Turn on HT.
- Slowly (50s / 1 section) move the filament potentiometer to the saturation, which is written on the post-it.
- Click TONE (TONE 2x =ACB)
- Set focus: first the coarse then the fine focus (use D-mag or 2xSL1).

## BEAM ALIGNMENT

- Magnification (MAG) to ~20.000X
- Set the focus
- Correct astigmatism
- Set the focus
- Correct the aperture (»WOBB«)
- Set the focus

For optimal image change WD, accelerating voltage, current and aperture. Do not forget to focus and align the beam.

## Lifting the table/sample

- **Set WD:**
  - **click F1**
  - **Write: cmd>stage zXX (XX never under 10 mm!)**
  - **click enter**

## Backscattered electrons imaging

- Capture »SEI« (previous steps!).
- Click »MENU F2«:
  - »IMS« choose »BEI (Compo)«.
- Chose the aperture and the suitable current (e.g. #3 =SZ 8, #2 =SZ 12).
- Click TONE and correct the contrast.
- »BEIW« – opens a window BEIW CONTROL – select FINE, you can fix the SHADOW – ON
- »LUT« tab – make a binary image (if you have two phases).

## Capturing images - ORION

- On the left LCD open the program ORION
- Click image capturing »foto«.
- You choose the quality (norm or HR) - set it also on SEM (Menu - Setup – norm or HR)
- When you acquire a photo the light »PHOTO« turns on (»slow scan«)
- Indicate to save the picture.
- Unfreeze the picture (FREEZE button – L in R)

## End of the work and removal of the sample

- Turn down to a minimum magnification.
- Turn down the potentiometer filament (slowly).
- Turn off HT.
- Dim the left of the screen and delete tags in the »SETUP-Data print«.
- In the »STAGE« click »Exchange position«
- Dim the right of the screen (leave the tab »stage F5«!)
- **Last in the day: turn off the monitor.**
- Set the initial settings!
- Hold »VENT« for 2s when you hear a click open the door.
- Remove the sleds.
- Close the door and hold »EVAC« for 2s.
- Clean up (remove your sample).
- Write your name and notes in the book.

# Short instructions for the JEOL JSM-5800

## EDXS analyze

The Oxford Isis system consists of specialized programs that are selected from the control and database program called "Labbook". The programs are:

*X-Ray Analysis*

*Autobeam* (digital image acquisition)

*Detector Control* (turning the detector on or off)

*Autostage* (automated stage control)

*IMQuant* (digital image analysis)

*Link Opal*

*Disk Inspector*

*Notebook* (writing reports)

- WD has to be **10 mm**, accelerating voltage 20 kV
  - take an image with Orion SEI/BEI and open in »paint« (or with Link ISIS – Autobeam, but at the end the pictures have to be converted to TIFF!)
  - aperture #3
  - Right LCD is for EDS
  - activate program »link ISIS« 
    - a window opens »Welcome to Link ISIS«
    - choose your own name or default user
    - Job: default
    - The »Labbook« opens 
  - you choose »X-Ray Analysis«
  - Set the conditions:
    - Edit→Acquisition setup:
    - acquisition t = life time 60s
    - More: Process time (from 1 to 6)
    - conditions Δ(#3, spot size, PC, ACCV)
  - MENU→BCX and you select the area (point or square) and click SET (Save Image in paint and the area that you covered) or move the stage to the area of interest and zoom in.
  - in Link ISIS click record (round button). Check dead time and counts.
  - Magnifier glass serves to enlarge the spectrum. With the mouse you move.
  - »?« IDENTIFY PEAKS you find and mark the peaks (the trick - if you click with the mouse to the top, it shows possibilities)
  - Mark peaks - label or paint
  - **Quantification:** »%« SEM quant (ZAF)
    - Click select elements - check or uncheck them
    - Click quantify → All elements and keep a note: file - copy text, and you've made in the text document \*
- quantification consider before you do what you've got (If the stoichiometry is known) then it makes sense to quantify at the stoichiometric ratio (for example, a particular element. Oxygen)

- **Speedmap** (before you launch it you have to color the window) (trick: shift and mouse).

### Exporting Spectra:

- As a graphic plot: Click the Print button, pull down "File" menu and select "Select as TIFF (line spectrum)" or click TIFF export under the File Menu
- As an ASCII file: pull down the File menu and select "Spectrum Conversion" select "Conversion Type ..." click the EMSA / MAS button and click OK. Pull down the File menu again and click "Export". Choose your destination and name and click OK.
- Note that you get comma separated text with the header. There are 1024 data points in 5 columns and can be read with Excel, Origin or Desktop Spectrum Analyzer from NIST.

Standards: Under "Options" click "Quant calibration..." and be sure you have a suitable standard!

Read the Oxford manuals that are in the cabinet for more information!

	ACCV (kV)	WD	Spot Size	
<b>High MAG</b> (>10 000x)	<b>20-30</b>	<b>8-12 mm</b>	<b>5-8</b>	<b>#1</b>
<b>Low MAG</b> (min 18x)	<b>5-15</b>	<b>48 mm</b>	<b>8-16</b>	
<b>Quantitative EDS</b>	<b>20</b>	<b>10-12 mm</b>	<b>8-16</b>	<b>#3</b>