IBIL setup operation manual
for SynerJY® software version 1.8.5.0

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Author: Carlos Marques

Equipment Managers: Carlos Marques, +351219946084, cmarques@itn.pt
Luís Alves, +351219946112, lcalves@itn.pt

Instituto Tecnológico e Nuclear
Unidade de Física e de Aceleradores
General description

The IBIL setup is comprised of a monochromator (TRIAX® 190), to which a Peltier cooled CCD detector (Symphony® 1024×256 pixel) is coupled, and a cooling unit, as shown in figure 1. From the monochromator to the experimental chamber goes an optical fiber and a coupling and focusing device, detailed in fig. 2. The connections are numbered, each pair cable – socket is univocally identified with the same number, from 1 to 7.

The available range of measurement is from 200 nm to 1100 nm, with 0.3 nm resolution in the optimum combination of diffraction grating and entrance slit width (software controlled, from 0.002 mm to 2 mm). There are manually switchable filters (cutting up to 385 nm – top filter - and up to 630 nm – bottom filter, with the central position empty) at the entrance of the monochromator.

Figure 1 – Image of the IBIL setup, with the optical fiber attached to the experimental chamber via mirror based coupling device.
Figure 2 – Photography of the coupling device attached to the experimental chamber.

**Operation:**

The operation of the equipment can be divided in three phases:

1. Check connections, power on equipment and start the software (running on Windows XP®);

2. Calibrate the system (zero calibration and wavelength calibration, with fluorescent ambient light) and adjust the coupling device and move the sample in order to focus on the beam spot;

3. Measurements and shut down.

**Phase 1:**

1. Remove black blankets; check all connections: between CCD and monochromator (shutter control, BNC cable 4), between CCD and cooling unit (cables 5 and 6) and between equipment and computer (ethernet RJ45 cable 1 and RS232 cable 2);

2. Power on monochromator (just plug the socket cable 3) and cooling unit (cable 7); turn cooling unit on (red and green light stable for 30 seconds and then blinking at 1 Hz); start computer and enter Windows XP® partition; logon;

3. Insert USB disk to allow SynerJY Origin 7.5® based software to run (usually located in the right hand drawer);
4 – Double click on SynerJY® icon located at the computer desktop;

5 – Click on the icon shown in fig. 3 to initialize the equipment. The screen of fig. 4 will appear, followed by screens of fig. 5 and fig. 6;

Figure 3 – Initial screen of SynerJY® acquisition ORIGIN 7.5® based software.
Figure 4 – Hardware configuration selection.

Figure 5 – Hardware initialization screen.
6 – Check temperature, selecting ADVANCED on the screen of fig. 6. Fig. 7 screen will show up; wait for T < 204 K to start acquisition (about 10 minutes) and click OK;

Figure 7 – Temperature check. This menu also allows cosmic ray removal (removes intense and narrow peaks) and background correction (removes baseline noise, which is 1240 ± 10 counts, check it with the shutter closed on the PREVIEW screen, fig. 11).
If you got up to this screen and T is about 202 K – 204 K then proceed to phase 2. Lights on the cooling unit should both be green and stable.

**Phase 2**

7 – From the acquisition menu shown in fig. 8 choose EXPOSURE TIME (longer times are prone to cosmic ray appearance) and TYPE of scan: CCD RANGE, where the selection is the initial and final wavelength to acquire, or CCD POSITION, where the central wavelength is chosen and side bands are also acquired (around ± 50 nm for the 1200 l/mm grating and ± 200 nm for the 300 l/mm grating);

![Figure 8 – Acquisition screen in the default DETECTORS mode.](image)

8 – From the screen of fig. 8 click MONOS on the GENERAL tab and screen of fig. 9 will appear; choose diffraction GRATING (low wavelengths with 1200 l/mm; high wavelengths with 300 l/mm), and front entry slit width (from 0.002 mm to 2 mm, the wider the poorer resolution attainable); the exit slit is not installed;
Figure 9 – Acquisition screen in the MONOS mode, allowing choosing the grating and slit width.

Figure 10 – Photography of the optical fiber in the test position to calibrate the system and monitor its sensitivity; also in the picture is the beam current meter.

9 – With the optical fiber in the test position (fig. 10) perform an acquisition of light from the fluorescent lamp and check peak positions; from screens of fig. 8 or fig. 9, click PREVIEW, screen in figure 11 appears;
Figure 11 – Preview menu, in DETECTORS default, with a 0 order preview acquisition.

10 - Zero order calibration: position at 0 nm and acquire (choosing 0.002 mm entrance slit width and appropriate exposure time in order not to saturate the CCD, which happens over 65536 – or 16 bit - counts) by clicking RUN; a Gaussian shaped band should appear centered at 0 nm; calibrate if peak position is different (step 11); adjust CCD and the entrance of the optical fiber if band is not Gaussian (refer to Jobin Yvon manual); repeat for the other grating;

11 – If the peak is not centered at 0 nm, click MONOS on screen of fig. 11 and the screen of fig. 12 will appear. Next, click CALIBRATE and fig. 13 shows the expected screen. To the CURRENT POSITION wavelength value subtract the measured peak center (if this is negative… add) and input this value on the CALIBRATED POSITION field. Click OK and acquire new spectra; Repeat this step if zero order is not centered at 0 nm. Repeat for the other grating;
Figure 12 – Preview screen in the MONOS mode.

Figure 13 – Zero calibration; to the current position subtract the desired peak value.
12 – Wavelength calibration: acquire a spectrum centered at 550 nm (to monitor Hg line at 546 nm, FWHM about 0.4 nm), again for both gratings, (fig. 14) and confirm that the bands in table 1 are found in their expected positions. Choose exposure times and entrance slit width in order not to saturate the CCD; use top filter to avoid 2nd order diffractions;

Figure 14 – Typical emission lines present in common fluorescent lamps.

Table 1 – Most intense Hg\(^+\) emission lines (CRC Handbook).

<table>
<thead>
<tr>
<th>Emission lines of Hg(^+), present in typical fluorescent lamps</th>
<th>wavelength (nm)</th>
<th>relative intensity</th>
<th>wavelength (nm)</th>
<th>relative intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td>184.95</td>
<td>1000</td>
<td>407.78</td>
<td>150</td>
<td></td>
</tr>
<tr>
<td>253.65</td>
<td>15000</td>
<td>433.92</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>265.20</td>
<td>250</td>
<td>434.75</td>
<td>400</td>
<td></td>
</tr>
<tr>
<td>265.37</td>
<td>400</td>
<td>435.83</td>
<td>4000</td>
<td></td>
</tr>
<tr>
<td>289.36</td>
<td>150</td>
<td>546.07</td>
<td>1100</td>
<td></td>
</tr>
<tr>
<td>296.73</td>
<td>1200</td>
<td>567.59</td>
<td>160</td>
<td></td>
</tr>
<tr>
<td>302.15</td>
<td>300</td>
<td>576.92</td>
<td>240</td>
<td></td>
</tr>
<tr>
<td>312.57</td>
<td>400</td>
<td>578.97</td>
<td>100</td>
<td></td>
</tr>
<tr>
<td>313.16</td>
<td>320</td>
<td>579.07</td>
<td>280</td>
<td></td>
</tr>
<tr>
<td>313.18</td>
<td>320</td>
<td>580.38</td>
<td>140</td>
<td></td>
</tr>
<tr>
<td>365.02</td>
<td>2800</td>
<td>690.75</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>365.48</td>
<td>300</td>
<td>708.19</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>366.33</td>
<td>240</td>
<td>709.19</td>
<td>250</td>
<td></td>
</tr>
<tr>
<td>404.66</td>
<td>1800</td>
<td>1013.97</td>
<td>2000</td>
<td></td>
</tr>
</tbody>
</table>

13 – Focus on the beam spot: input the coupling mirror based device with a laser beam; from the binoculars check the laser spot on the sample and make it coincide with the beam spot by adjusting the sample position and the optical coupling (cf. fig. 1);

14 – Reconnect the optical fiber to the coupling device; cover the binoculars and the equipment with the black blanket, but allowing for CCD ventilation;
Phase 3

15 – From the data preview menu optimize wavelength range, exposure time, entrance slit width with several RUNs; then click TRANSFER to send all these parameters to the acquisition menu (otherwise it will keep the last ones), shown in figures 9 and 11. Slide the desired filter, fig. 15;

Figure 15 – Photography of the filter slide (bottom filter on) close to the optical fiber entrance.

16 – Click RUN; before displaying the data the software will ask for a project name (for example: TEST), which will correspond a dedicated ORIGIN 7.5® project in the program folder (H:\Program Files\Jobin Yvon\SynerJY Data\TEST.opj; each spectra will be an ORIGIN 7.5® graph named sequentially DefaultdataX. Use acquisition sheets were all parameters can be written; don’t forget to annotate the logbook with the information requested;

17 – Click the same icon as in step 5 and make a new measurement;

18 – To turn the equipment off, simply close the SynerJY® software, turn off cooling unit and unplug cables 3 and 7 from mains.

Practical tips:

Top filter cuts up to 385 nm and is thus recommended to measure in the 385 nm – 770 nm range;

Bottom filter cuts up to 630 nm and is thus suitable to measure from 630 nm to 1260 nm;

Central filter is vacant and thus choose this position to measure from 200 nm to 400 nm;

The above limits are chosen to avoid second-order diffractions. Figure 16 illustrates the filter concept.
Figure 16 – Luminescence spectra of a fluorescent lamp acquired with the 385 nm or the 630 nm filter. The use of the filter prevents some 2\textsuperscript{nd} order diffractions and while the 385 nm inhibits these diffractions up to 770 nm (and thus allow the 404 nm 2\textsuperscript{nd} diffraction at 808 nm, cf. table 1) the 630 nm filter extends this effect up to 1260 nm.

Use black blankets to avoid stray light, covering essentially the monochromator and filter region, as well as the CCD connection (do not cover the entire CCD or the temperature will not drop to 203 K). Don’t forget to cover also the binoculars.

Take one spectra without beam in the experimental conditions used to ascertain the absence of any system related features.

Take one spectra with the minimum of ambient light possible to exclude the appearance of external light features on the spectra.

Considering the efficiency curves of the 1200 l/mm and 300 l/mm gratings (fig. 17) the first should be used for the lower wavelengths while the latter used for higher ones. Each grating has a maximum or blaze wavelength, 250 nm for the 1200 l/mm and 1000 nm for the 300 l/mm.
Figure 17 – Theoretical spectral efficiency curves for a) 300 grooves/mm (transversal electric, TE, and transversal magnetic, TM) and b) 1200 grooves/mm, used in our system.

Step 12 should be repeated for every sample or spot analysed.

The usable wavelength range is dictated by the efficiency of the CCD, fig. 18.

Figure 18 – CCD spectral sensitivity at RT.
Troubleshooting…

You don’t get the screen shown in figure 6: check connections between PC and equipment (cables 1 and 2);

The temperature doesn’t reach 203 K: check if CCD ventilation is not obstructed;

The noise level is higher than 1240 counts: check CCD temperature and block stray light.

User Notes: