Acquiring Luminescence Spectra using the ARC – NCL Spectral Measurement System + Boxcar Integrator
Disclaimer

- Safety –the first !!! This presentation is not manual. It is just brief set of rule to remind procedure for simple measurements. You should read manual first.

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Luminescence

is a process in which an excited material emits light (electromagnetic radiation)

For example, the following types of luminescence caused by different excitation processes could be classified as:

**Chemoluminescence**, is the emission of light as the result of a chemical reaction

*Light production in fireflies is due to a type of chemical reaction called bioluminescence*

**Photoluminescence** (PL) is a process in which a material absorbs photons (electromagnetic radiation) and then re-radiates photons

*Banknote photoluminescence after excitation by UV light from flash lamp*

**Electroluminescence** (EL) is an process in which a material emits light in response to an electric current passed through it, or to a strong electric field.

*Infrared electroluminescence of photodiode in the remote is stimulated by electrical current*
Parameters of the Luminescence

- Luminescence **spectra** shows how intensity of the luminescence depends on wavelength.

- Luminescence **lifetime** refers to the average time the molecule/ion stays in its excited state before emitting a photon (or how long luminescence could be observed).

- The luminescence **quantum yield** gives the efficiency of the luminescence. It is defined as the ratio of the number of photons emitted to the number of photons absorbed.

**Goal of the Lab:** Measuring of the Luminescence spectra using ARC – NCL Spectral System.
Experiment Background

1. First of all, we need excitation source to transfer our sample into excited (high energy) state. Here we will consider only optical excitation. It means that we will use optical radiation to excite the sample.

2. Second, we need an optical system to collect the luminescence and direct it to the detector. Also we need to select luminescence from the excitation radiation. For these purposes we can use optical filter or monochromator which can help select radiation only at required wavelength.

3. We need to detect optical signal (convert intensity of the optical radiation into electrical signals) and then convert signal into digital format.
Experimental setup (Principal Scheme)

**Triggering signal**

**Pulse signal**

**Sample**

**Grating turret**

**Detector**

**Registration system**

**DC signal**

**To Analog-Digital Converter**

**Spectra Pro 300i**

**SpectraPro-300i Control Unit (NCL)**
Requirements to optical excitation

A wavelength of the optical excitation pulses should be within absorption band of the studied samples!!!

Available commercial tunable solid-state lasers

30ps

OPO Ekspla

7ns

OPO Spectra Physics

60-100ns

Typical curve of the Alexandrite laser
Optical Setup

1) Avoid reflection of the excitation radiation to the measurement system!!!

2) Acceptance angle of Spectra Pro300i is $\Theta=1/4$. Therefore lens diameter should be $D>L\Theta$ (for $L2=L1=2F$ configuration $D>F/2$)
**How to Choose a Detector?**

A **optical detector** should convert luminescence of the sample into an electrical signal.

Therefore the **major requirement to the optical detector**: to be sensitive at the wavelength of the luminescence photons.

**Operating Ranges for Judson Technologies detectors**

**Attention, these detectors should be cooled by liquid Nitrogen!!!**
Operating Ranges for ARC detectors

<table>
<thead>
<tr>
<th>Device</th>
<th>Wavelength in Nanometers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Model P1 PMT</td>
<td>0 - 200</td>
</tr>
<tr>
<td>Model P2 PMT</td>
<td>200 - 400</td>
</tr>
<tr>
<td>Model SI-440-UV Silicon Photodiode</td>
<td>400 - 800</td>
</tr>
<tr>
<td>Model SI-440 Silicon Photodiode</td>
<td>800 - 1000</td>
</tr>
<tr>
<td>Model P3 PMT</td>
<td>1000 - 1200</td>
</tr>
<tr>
<td>Model ID-441 and ID-441-C InGaAs</td>
<td>1200 - 2000</td>
</tr>
<tr>
<td>Model ID-442 PbS</td>
<td>2000 - 3000</td>
</tr>
<tr>
<td>Model ID-443 InSb</td>
<td>3000 - 4000</td>
</tr>
<tr>
<td>Model ID-444 M-C-T</td>
<td>4000 - 6000</td>
</tr>
</tbody>
</table>

The graph shows the operating ranges of different ARC detectors in terms of wavelength in nanometers.
Two type of the electronic registration system

depends on the ratio of the signal duration (Ts) and period between pulses (Tr); duty cycle

- Lock-in Amplifier for signals when \( T_s \sim T_r \)
- Boxcar Integrator for signals when \( T_s \ll T_r \)
Experiments with Boxcar Integrator

A gated integrator (also called a boxcar integrator or averager) integrates an analytical signal over a fixed time window. In pulsed experiments the integrator gate is synchronized with the analytical signal by a trigger. This method increases the signal-to-noise ratio by recording the voltage only when the signal is present, and ignoring time periods when there is no signal and only noise.
Spectra Pro 300i

Optical design: Czerny – Turner

Focal length: 300 mm
Aperture ratio: f / 4

Grating size: 68 x 68 mm
Grating mount: triple-grating turret
Grating #1 300gr/mm ($\lambda_{\text{blaze}}$) ($\lambda_{\text{max}}$ = 5.6)
Grating #2 600 gr/mm ($\lambda_{\text{blaze}}$) ($\lambda_{\text{max}}$ = 2.8)
Grating #3 150 gr/mm ($\lambda_{\text{blaze}}$) ($\lambda_{\text{max}}$ = 11.2)

Standard slits: adjustable from 10 μm to 3 mm wide;

Linear dispersion (nm/mm)@500 nm:
11- Grating #1 300gr/mm
5- Grating #2 600 gr/mm
21- Grating #3 150 gr/mm

Output slits
Input slits
Spectra Pro 300i Connections

To Power Supply unit

To Control Unit (NCL)

From Lock-in- Amplifier

Power
RS-232
Mono 1

To Power Supply unit

To Computer

To Spectrometer
Experimental Setup and Wiring

Spectra Pro 300i

Thorlabs Si Photo Detector can be used. Detector should be set to “see” scattered light, NOT under direct beam.

Pulsed Laser

Sample Luminescence signal

Triggering signal

Averaged Signal

Gate

Power switch

Ch1
Description of Processes Above

1) Pulsed laser excites the sample
2) Si detector detects excitation pulse and triggers both Boxcar and Oscilloscope
3) Monochromator splits the PL signal from the sample and directs the desirable wavelength onto the detector
4) Triggered oscilloscope reads and displays the signal from the detector
5) Triggered Boxcar outputs a Gate Signal.
6) This Gate Signal needs to be adjusted and shifted onto the Luminescence signal using the controller knobs (see next slide for detailed description of Boxcar operation)
7) Boxcar receives the Luminescence signal coinciding with the gate and averages it increasing the amplitude and quality of the signal
8) Averaged signal is sent to PC through the NCL and Luminescence Spectrum is obtained

NOTE: Before proceeding with the steps described in the next slide bring the monochromator to the expected Luminescence Wavelength, ex: for Cr:ZnSe set monochromator to ~2600nm (Look at page 21)
Operation of Boxcar

**Triggering** - After wiring every component as shown above and firing the laser power on the Boxcar. Triggering is set using the RATE and RATE ADJUST knobs until green LED (TRIG) starts blinking. The Trigger Signal is used by Boxcar to initiate Gate Signal.

**Delay** – The **Gate Signal** needs to be **positioned onto the Luminescence Signal** (both of these signals should be displayed on the oscilloscope display, you are just trying to visually overlap the signal like shown on the Oscilloscope’s display above). This is done using SCALE and MULTIPLIER knobs (in delay section). Read the time scale on the Oscilloscope this is roughly the scale you should set on the SCALE knob. Rotating MULTIPLIER knob you should be able to shift the Gate left and right on the screen.

**Width** - The width of the Gate Signal can be adjusted using the SCALE and MULTIPLIER knobs (in width section).

**Signal** - There are many sources of noise in this set up. These noises can be eliminated by off-setting the noise signal. To do this connect LAST SAMPLE (from OUTPUTS) to either ANALOG METER or DIGITAL METER port (to the left of the POWER Switch) the signal will be displayed on the analog or LED read-outs above. Cover the crystal with cardboard blocking Luminescence from crystal. Using the SENSITIVITY and INPUT OFFSET bring the signal to a small positive reading (in other words you just zeroed the background noise). Now remove the cardboard and check if the sensitivity is appropriate, if signal is too low rotate the SENSITIVITY knob to a lower voltage.

**Averaging** – Using the SAMPLES knob you can set how many signals are taken to output 1 averaged signal. The integration time in the “SpectraSense” software (next slide) needs to be set to integer multiples of \( \frac{1}{\text{laser pulse freq.}} \) \times \text{(number of samples)} to prevent integration of signals corresponding to later wavelengths. For example, if laser has 10 pulses per second and SAMPLES is set to 3 Integration time can be 0.3, 0.6, 0.9...etc.

**NOTE**: Some of our detectors produce and negative signal, in this case there are two switches on the back of Boxcar that need to be switched to “inverted” position.
Operation Procedure

1. Switch on:
   - Boxcar Integrator
   - Detector Power Supply (see manual)

2. Switch on Spectra Pro 300i:
   - A-Switch on PC
   - B-Switch on Spectrometer power supply
   - C-Switch on Controller Unit (NCL)

Do not switch Laser on !!!
Operation Procedure

3. Run “SepctraSense” Program
Operation Procedure

4. Select Hardware Status Menu

Or Here
Operation Procedure

4. Select grating (#1, #2, or #3) – A

5. To Select required wavelength: input wavelength, in nm and press “GOTO” – B

6. Switch laser on according to the laser manual (see safety instruction !!!)
Operation Procedure

6. For Spectra Measuring:
   6.A- Select Accusation mode
6. For Spectra Measuring:

6.B- Select Scan of the Monochromator #1
Operation Procedure

6. For Spectra Measuring:
   6.D. Select Acquisition Mode – Chanel #1
Operation Procedure

7. For Spectra Measuring:
   7.E- Select Scan Parameters

- E1 - Initial Wavelength
- E2 - Final Wavelength
- E3 - Increment
- E4 - Integration Time at each wavelength
- E5 - Number of scanning cycles

8. Press Acquire to start measurements
Results will be shown in the “Live Data’ window
9. To save Spectra in the ASCII code (Text File)
9.1 Select File>Save
9.2 Select “Experiment Data”
9.3 Press “OK”
9.4 Select Directory and File Name
Operation Procedure

10. After work done, shutdown:

- Laser (according manual)
- Detector Power Supply Unit
- Boxcar Integrator
- NCL controller
- Monochromator power supply unite