

**Standard Operating Procedures for  
Room 225A (inside of room 225)  
Laser Controlled Area**

Version 3 (September 23, 2014, Jacob Hoffman)

**Introduction**

This Laser Controlled Area (LCA) comprises the entirety of room 225A. The diagram of the room is given in Appendix A. A pulsed (1kHz), Class IV laser system is housed in the LCA. A YAG/Ti-Sapphire amplified fiber laser is capable of delivering high energy pulses (<1 mJ/pulse) of 775, 387 and 250 nm light. This Standard Operating Procedure describes procedures for daily operation of this system. Inside the LCA, there is one optical table which contains the laser system, optical probe station, non-linear optics (Topas system), and photodetectors. The rest of the data acquisition system (computer, scope, etc.) is placed under the optical table and beside the optical table. A data acquisition computer is placed and atop the desk sitting immediately outside of room 225A, within room 225.

**LCA Supervisor**

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### **Authorized Users**

Authorized users need to have taken the Laser Safety Class provided by UND Risk Management. All potential users are expected to have completed a laser eye exam in accordance with safety procedures. In addition, they must have been trained in the laser-operating procedures of the specific laser by the LCA Supervisor or trained researcher before they are permitted to use the laser. Finally, they must read this SOP and sign the authorized users list located in appendix C.

### **Scientific Collaborators & Spectators**

A *Scientific Collaborator* is a person who is involved in non-laser aspects of the experiment. Such persons are allowed to enter the LCA when laser is powered up, but not when it is lasing (see Warning Light Codes below). See the Eyewear Section below for a description of the proper goggles to wear for the laser for personal safety measures.

A *Spectator* is a visitor who is not involved in the experiment. Such persons should not enter the LCA when the laser is powered up, and, in particular, they should not enter the LCA when the laser beam is exposed. They should be confined to the operator's area outside the LCA. They can be shown the optical equipment inside the LCA only under conditions when the power to the laser is turned off.

### **Laser Start-up/Shut down Procedures**

#### **Start-up:**

**Note:** Reference numbers in parentheses with diagram of room 225A in Appendix A

- 1) Put on goggles: Everyone in the LCA (room 225A) must be wearing goggles when the laser is running
- 2) Check the chiller (2) water level to ensure proper cooling of the system. Do this by uncapping the top and making sure the water-level is at the top. If the water level is low, refill with DI water. Allow the chiller to equilibrate the temperature to 17 °C before using the laser in order to ensure that accurate measurements are taken.

- 3) Open the water valve all of the way (28) by turning it counterclockwise. Then close the valve one-half of a rotation by turning clockwise.
- 4) Turn on the Vibration Generator (if using CaF<sub>2</sub> crystal) (31): Ensure that the CaF<sub>2</sub> crystal is moving side to side. Never turn on the laser without the CaF<sub>2</sub> crystal moving.
- 5) Turn on the HV switch of the DT-505 (14) noting that the main power switch of power supply is to be kept on at all times
- 6) Turn on the oscilloscope (22). (There may be a need to depress the power button several times before the scope turns on.) A green line will appear on screen 2-3 seconds after powering up the oscilloscope.
- 7) Turn on the instrument power supply (16):
  - a. Make sure the power supply is in “standby” mode.
  - b. Turn main power-key clockwise from “off” to “on” position.
  - c. Push “pump start” button, if the instrument is properly mode locked, “MOD. FREQ.” meter should read 1 kHz.
  - d. Turn on the lamp power supply switch, wait about 10 seconds for lamp start button to light up before proceeding.
  - e. Push “lamp start” button, “DC Amperes” meter should go from ~23-28 Aperes. (With aging lamp it may be necessary to adjust the lamp current. Do not exceed above 28 amps)
  - f. Flip shutter switches from closed to open in the following order:
    - i. “INJ” shutter
    - ii. “ORC” shutter
    - iii. “OPEN/MAIN” shutter
- 8) Wait ~40 minutes for the system to thermally stabilize.
- 9) Open Helios Visible from desktop to CPU (10 or 21)
- 10) Turn the chopper (18) on to Low Frequency, the signal should stabilize at 104.8

### **Shut Down**

- 1) Turn off instrument power (16) supply
  - a. Flip shutter switches from “open” to “close in the following order.
    - i. “OPEN” or “MAIN” shutter
    - ii. “ORC” shutter
    - iii. “INJ” shutter
  - b. Turn lamp power supply switch to “OFF” position
  - c. Turn main power key counter clockwise from “ON” to “OFF” position
  - d. Make sure the power supply is in standby mode
  - e. Standby Settings
    - i. All shutters are closed
    - ii. Power supply switch “off”
    - iii. Main power key “off”
    - iv. Modulation control knob set to “external”
    - v. Modulation control power switch is flipped up
- 2) Turn off Oscilloscope (22)
- 3) Turn-off the Vibration generator (if using CaF<sub>2</sub> crystal) (31)
- 4) Turn off HV on DT-505 (14)

*IMPORTANT. DO NOT turn off “power switch on DT-505*

- 5) Close water valve completely (28)
- 6) Make sure chopper (18) is turned off
- 7) Before leaving Room 225A make sure to return everything to their original positions including tools, optics, samples and goggles.

### **Target Area (Sample) Preparation**

When preparing samples for analysis it is of high importance to consider their absorption in the UV and Visible regions. An optimal range of absorbance is approximately between the values of 0.3 and 0.4, this absorbance magnitude will still allow for the transmission of the white light probe which is essential when using this system. When analyzing materials in the liquid

phase, it is important to store them in a sealed container so that no contamination can occur in the LCA. If any spills should occur, and are not manageable by the user, please see the list of contacts above for assistance in taking the proper action.

### **Normal Laser Operation**

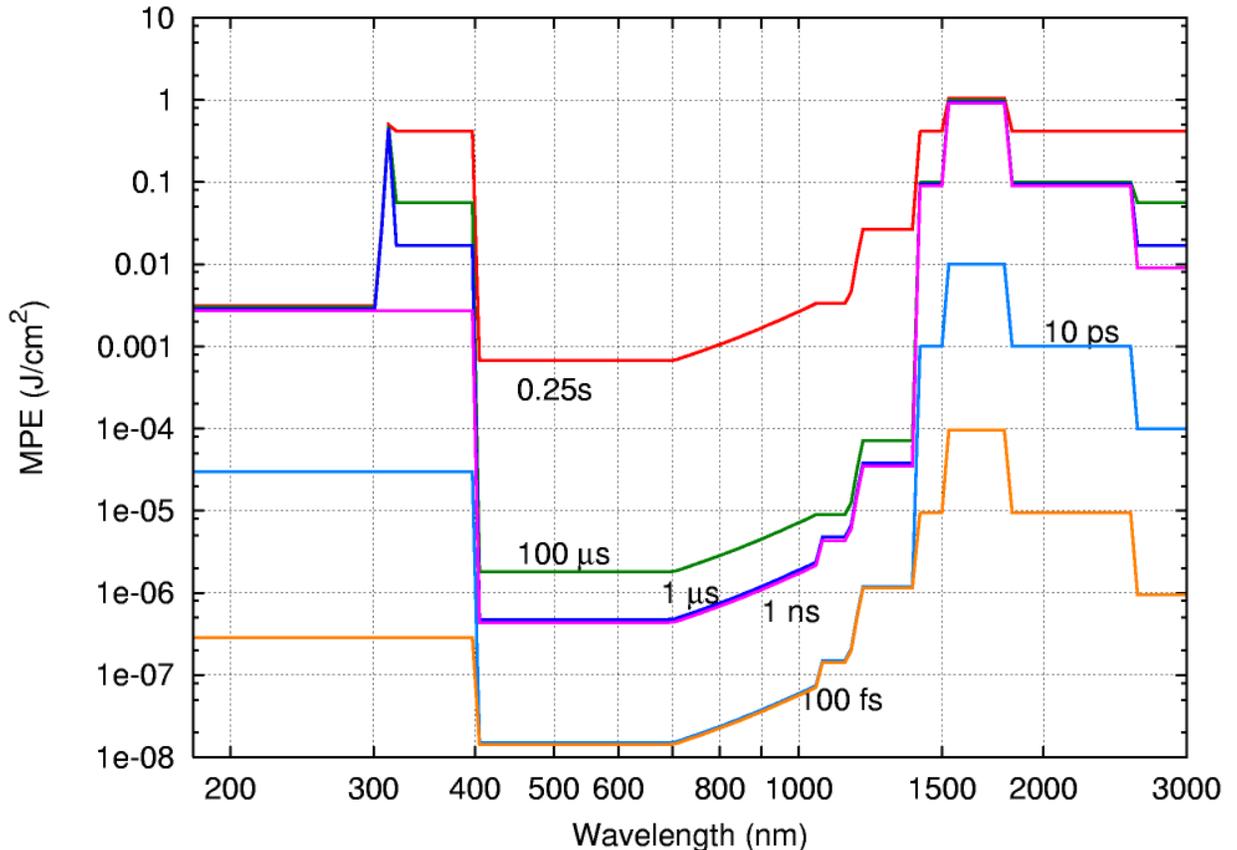
Beam exposure only occurs when two conditions are met simultaneously. First, the laser system needs to be powered up properly and all internal shutters need to be opened. Second, all conditions are met to allow the opening of the external shutter. These conditions include: the door to room 225A must be completely closed and its interlock engaged; both of the emergency beam stop triggers must be disengaged; and finally, the external shutter control switch must be set to the “open” position. Appendix A contains a diagram of the external shutter safety components specifically colored in red. Once the laser passes through the external shutter, it interacts with numerous optics, all of which are located on the optics table. The usual alignment for these optics is given in appendix B.

The laser is a Clark-MXR model 2010. The output wavelength is 775 nm, pulsed at 1 kHz. The pulse is approximately 130 fs (full width at half max). The output energy is between 0.6 and 0.9 mJ/pulse. The cross-section of the beam is circular, and its unfocused cross-sectional area is about between 0.25 cm<sup>2</sup>. Assuming a maximum of 0.9 mJ/pulse and a laser cross section of 0.25 cm<sup>2</sup>, the laser operates at an energy density of  $3.6 \times 10^{-3}$  J/pulse\*cm<sup>2</sup>. This is a Class IV laser.

### **Eyewear Section**

The proper goggles to wear for this system depend heavily on what wavelengths and energy densities the user may be exposed to during the course of the experiment. The relevant wavelengths that need to be considered are 775 nm (the fundamental), 387 nm (second harmonic), and 259 nm (third harmonic). Additional wavelengths to consider are 532 nm (the YAG inside the Clark-MXR accessed when maximizing fundamental power) and 590 nm (the most common wavelength accessible from the TOPAS). The variety of laser goggles available in 225A cover these regions.

The maximum permissible exposure per pulse (the exposure where no eye damage will occur) for different pulse widths and wavelength is presented in figure 1 (found in ANSI Z136.1). The MPE per pulse is very low for 100 fs pulses due to the large amount of power delivered in a short timeframe. Due to this the required OD is often high.



**Figure 1: Maximum Permissible Exposure for beams of a particular wavelength and pulse width.**

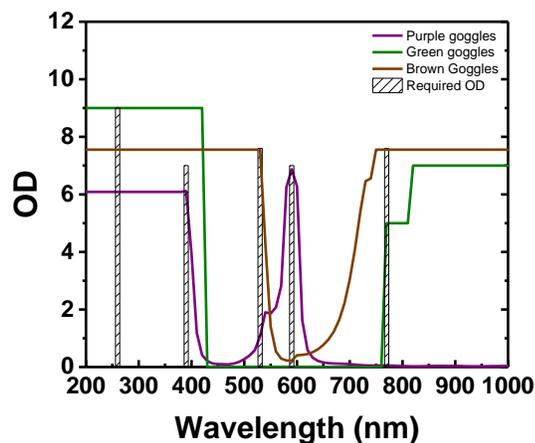
Exposure times for visible laser radiation are limited to the blink reflex (0.25 s) and for non-visible radiation 10 s. Thus, from the laser repetition frequency and the wavelength we can find the number of pulses per exposure to be 250 for the visible regions (775 nm, 590 nm, 532 nm, 387 nm) and 10,000 for 259 nm. According to ANSI Z136.1 the final MPE for pulsed systems operating under 55 kHz can be found by multiplying the MPE for a single pulse by  $n^{-1/4}$  (where  $n$  is the number of pulses). The MPE calculated this way for each laser wavelength is listed in table 1.

The required OD is the log of the attenuation factor (Energy exposed/MPE). The laser energy the eye is exposed to is simply the maximum energy density per pulse ( $3.6 \times 10^{-3}$  J/pulse\* $\text{cm}^2$ ) multiplied by the number of pulses during exposure. The calculate values are found in table 1.

**Table 1: Calculated OD parameters**

Wavelength (nm)	MPE ( $\text{J}/\text{cm}^2$ )	Number of pulses	Exposure Power ( $\text{J}/\text{cm}^2$ )	Attenuation Factor	Required OD
775	$7.5 \text{ e-}8$	250	0.9	$3.6 \text{ e}7$	7.5
387	$7.5 \text{ e-}8$	250	0.9	$1.2 \text{ e}7$	7
255	$3.0 \text{ e-}8$	10000	36	$1.2 \text{ e}9$	9
532	$3 \text{ e-}9$	250	0.9	$3.6 \text{ e}7$	7.5
590	$3 \text{ e-}9$	250	0.04	$1.3 \text{ e}7$	7

Figure 2 outlines the available goggles and the protection required for each wavelength accessible. When using 259 nm excitation the only adequate protection are the green goggles with OD 9+ in that region. For 387 nm both the green and brown goggles are good. For 532 and 775 nm the brown goggles should be used. Finally for 590 nm excitation (from the TOPAS) the best protection is provided by the purple goggles. These are acceptable because the max power output is significantly lower once the fundamental goes through the TOPAS (lowering the required OD).



**Figure 2: Available goggles and required protection**

## **Alignment Hazard Control**

The laser (inside of the box) was factory aligned and should not require any additional internal adjustments. If and when additional alignments are needed, permission is required from the LCA supervisor, Prashant Kamat.

**(1) Daily alignment of the laser.** The options available for excitation of the cell on the detection table are quite limited, however, adjustments of the final mirror and the lens just before the cell must be checked daily by the user-of-the-day.

This adjustment should be done by monitoring the beam with the luminescence visible when the laser is absorbed by a paper business card. **Warning: proper laser goggles must be worn during this operation** (check the Eyewear Section). If there is any doubt where the beam is going, it should be checked first at the optics which are closest to the laser itself.

**To align the laser beam in the daily routine**, at the sample holder there is both a pump (single wavelength) and probe (white light spectrum, focused) beam focused on the sample. Daily operations necessitate the following procedures:

- (a) With the pump beam blocked, the white light profile should be examined for both spectral features and overall intensity via the CCD camera within the Helios software. The user can adjust the probe beam intensity by rotating the neutral density filter wheel immediately before the sample. Adjusting the spectral features of the probe beam is more complicated, and involves tuning one or all of the following: the optical density of the sample, the half-waveplate, the polarizer, the oscillation setting on the vibration generator (if using CaF<sub>2</sub> crystal), the physical position of the crystal, and the intensity of the 775 nm beam incident on the crystal. There are three crystals available a sapphire crystal, an IR crystal and a CaF<sub>2</sub> Crystal. For a more complete description of creating white light with specific crystals see appendix D. Once the proper white profile has been achieved, one should confirm that this profile is consistent through the entirety of the delay-track positions. This can be checked by moving the delay track electronically (in the “Single Spectrum” portion of the software) to 5

positions spread across the delay track—such as 0, 400, 800, 1200, 1600—and checking the white light spectrum (in the “CCD Scope” portion of the software) at every position. If the white light profile varies significantly between positions, contact the LCA supervisor, Prashant Kamat, as probe beam alignment is necessary.

- (b) One should ensure that there is proper pump-probe overlap. In order for reliable data to be collected, the entirety of the probe-sample interaction must overlap spatially with the pump-sample interaction. The pump may irradiate portions of the sample which are not irradiated by the probe, but it is important that the probe does not irradiate portions of the sample which are not irradiated by the pump. When making adjustments to achieve proper pump-probe alignment, the user should adjust only the alignment of the pump beam. If probe beam alignment becomes necessary, the user should first contact the LCA supervisor, Prashant Kamat.
- (c) Using a sample with known transient features (usually CdSe quantum dots), the user should collect data with resolution of  $\Delta t=0.05$  ps close to the zero point (the point at which the pump and probe pulses arrive at the sample at the same time) of the delay track so that he or she can setup his or her experiment relative to the zero point.
- (d) Once the zero point has been determined, one should fine-tune the pump probe overlap. This can be done by monitoring the transient signal in real time using the Single Spectrum software feature, white delay track set to  $\sim 1$  ps after the zero time. Making slight adjustments to the pump beam mirror located immediately before the sample while monitoring the transient signal in real time allows one to maximize transient signal.

During the daily laser alignment, Scientific Collaborators and Spectators should never be in the LCA. Only approved users are allowed to be present in the LCA. If the user-of-the-day requires assistance with the alignment, an approved user or the LCA Supervisor should be called. Often a second person is recommended for safety considerations, such as when it is necessary to

open shutters some distance from where a detailed adjustment is being made by the user-of-the-day.

### **Laser Hazard Control**

**Borders of the LCA.** The LCA is enclosed on all sides by permanent walls (the entire room 225A is designated as the LCA). The entrance/exit to the LCA is through the sliding door connecting room 225 with room 225A. The external shutter is designed such that the beam will only enter into room 225A if this sliding door is completely closed.

**Warning Light System.** There is a Green-Yellow-Red light stack located on the wall immediately outside of room 225A. This is intended to make potential users and visitors aware of the current system status.

(1) Green means no power to the laser, but it provides a sign to personnel that the warning light system is functioning. When the Green lights are on, it is safe for anyone to enter the LCA without laser goggles. Note that because the green light is turned on most of the time, its bulb often burns out. If one notices that all three lights (Green-Yellow-Red) in the light stack are turned off, they should immediately contact the LCA supervisor, Prashant Kamat.

(2) Yellow means there is power going to the laser, but the external laser shutter is blocking laser exposure to the LCA. On Yellow, the proper laser goggles must be worn (see Eyewear Section). Only the user-of-the-day and their Scientific Collaborators can enter the LCA when the Yellow light is ON. Spectators are not permitted in the LCA when Yellow is ON.

(3) Red means there is lasing action and the external laser shutter is open. Only the users-of-the-day and other authorized users should be inside the LCA under these conditions, and then only for the purpose of aligning the laser optics. Scientific Collaborators and Spectators are not allowed inside the LCA when the Red light is ON. If help is needed to align the lasers, then other approved users should be called.

**Normal Operation.** When data is being collected, sliding door into room 225A should be closed and the user, collaborators, and spectators should not be inside of the LCA. The computer

which controls data collection simultaneously displays on the monitor inside of 225A and the monitor sitting on the desk immediately outside of 225A. That computer is also controlled simultaneously by keyboards and mice located both inside and outside of 225A. Once the laser and the system optics have been properly aligned, experimental parameters are to be set, and data is to be collected and analyzed, by the computer controls located outside of the LCA.

**Control of Additional LCA Hazards.** The laser system located inside of the LCA has additional hazards associated with it.

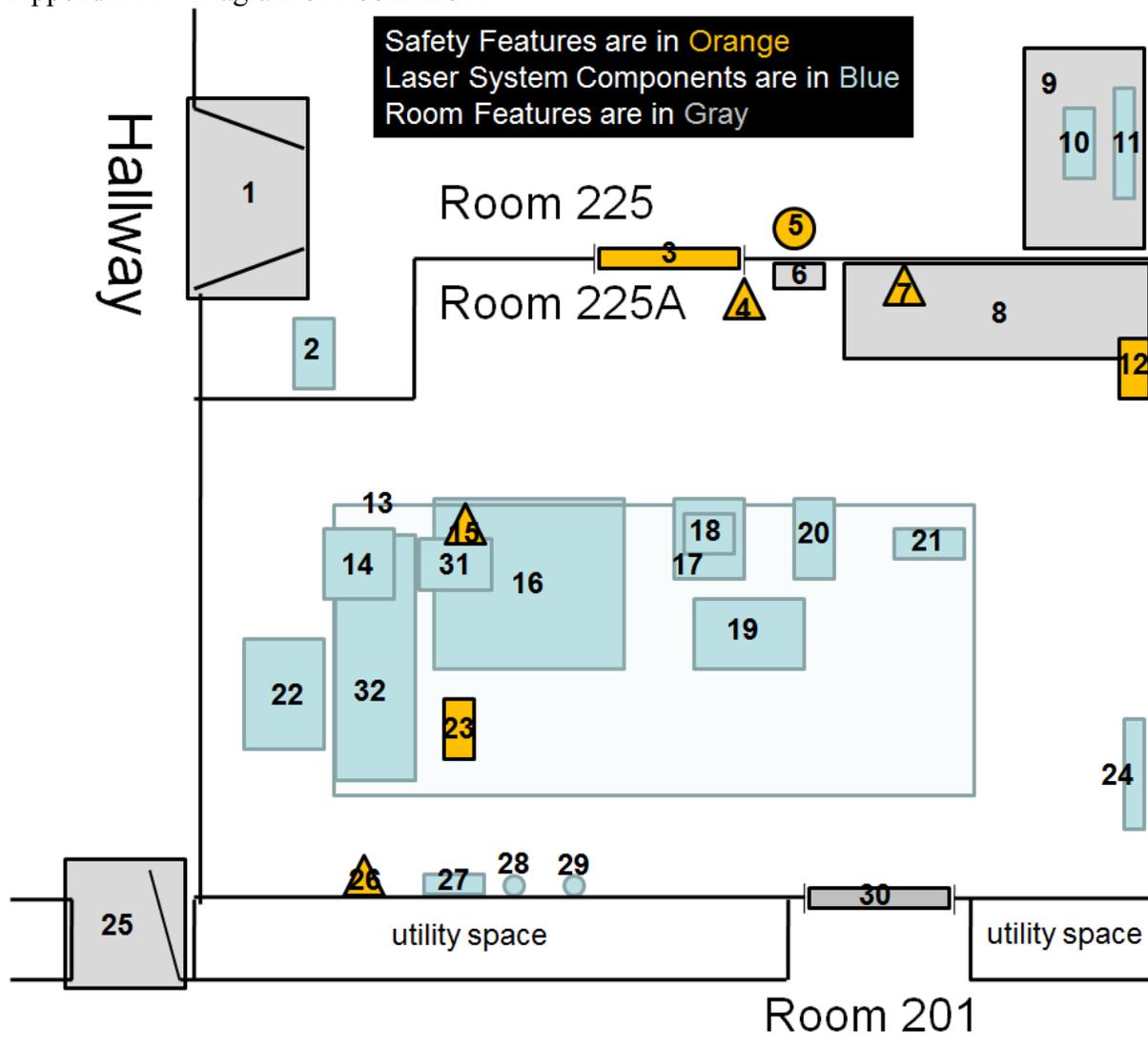
The laser utilizes high voltage circuitry that is contained in the laser power supply (located on the floor, under the optical table). There are two water sources (one line directly coming out of the wall and a second coming from the chiller) in the laser cavity that are used for cooling the system. It is important to monitor these water hoses to make sure that there are no leaks in them. Leaking hoses could cause anything from a fall to electric shock if the water manages to reach the high voltage source.

When operating in the LCA it is required to wear the correct laser goggles, gloves, and a lab coat. Additionally, wearing watches and jewelry in the LCA is prohibited due to the reflective nature of these items.

**Control of Emergencies & Abnormal Situations.** (a) In the case of electrical shock from high voltage, the voltage should be turned off if possible without danger to the non-injured users, and 911 should be called. (b) Exposures to laser beams should also be treated as other medical emergencies and reported to the Safety Committee.

All emergency situations and injuries should be reported to the management team after first aid issues have been dealt with. The names of the management team are listed on the yellow posters in the hallways.

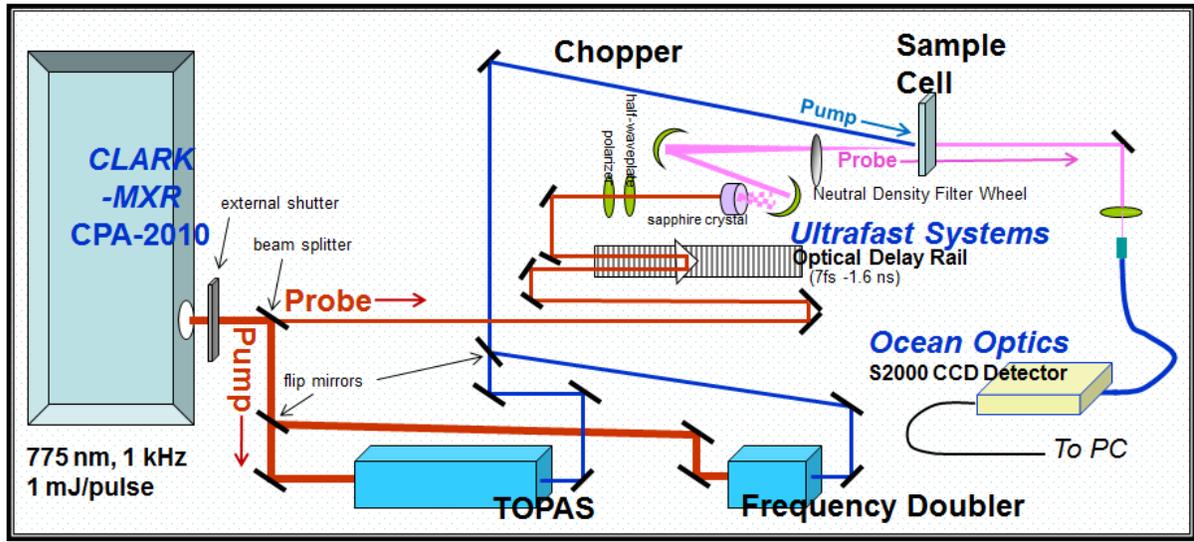
Appendix A – Diagram of Room 225A



1. Double door connecting Hallway with Room 225A
2. Chiller to maintain laser cavity temperature
3. Sliding door connecting Room 225 with Room 225A
4. Triggering mechanism which monitors whether sliding door (3) is open/closed (near top of door)
5. Green-Yellow-Red light stack indicating laser status
6. Light switch for Room 225A
7. Emergency beam stop switch (located above lab bench (8))

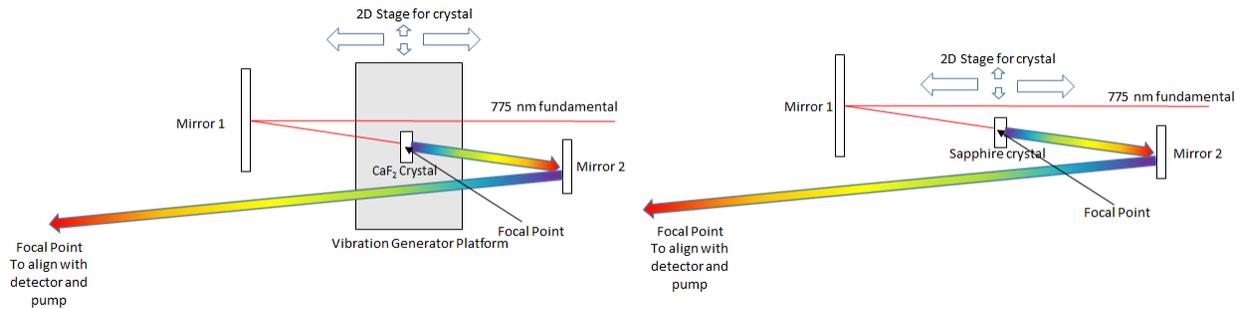
8. Lab bench
9. Computer Desk
10. Keyboard/Mouse for laser operation and data collection/analysis from Room 225.
11. Computer monitor for laser operation and data collection/analysis from Room 225.
12. Circuitry controlling external shutter.
13. Optics Table
14. DT-505 power supply for laser system (sitting under optics table)
15. External shutter switch, “open”/“closed” (sitting on top of power supply (16))
16. Main system power supply and control panel (sitting under optics table)
17. Delay stage power supply (sitting under optics table)
18. Chopper frequency control (sitting on top of chopper power supply, under optics table)
19. Data acquisition hardware (sitting under optics table)
20. PC (sitting under optics table)
21. Keyboard/Mouse for laser alignment (sitting on optics table)
22. Oscilloscope
23. External shutter (sitting on optics table)
24. Computer monitor for laser alignment
25. Door connecting Hallway with Room 201
26. Emergency beam stop switch
27. High voltage power supply for laser (emergency shutoff switch)
28. City water supply for cooling of reservoir inside of main system power supply (16)
29. Nitrogen source for reducing humidity inside of laser cavity
30. Door connecting Room 225A with Room 201 (bolted shut)
31. Vibration Generator control box
32. Clark-MRX 2010

Appendix B – Optical table setup





## Appendix D- White light crystals



There are three probe generating crystals: two sapphire crystals that generate spectral profiles of 430-800 nm, and 900-1200 nm respectively, and a CaF<sub>2</sub> crystal that generates a spectral profile of 390-700 nm.

The procedure to generate white light varies between the sapphire and CaF<sub>2</sub> crystals. **sapphire crystals.** The vibration generator is not necessary. The crystal should be mounted and put into the 2D stage. The crystal should be adjusted so that the 775nm fundamental is hitting in the middle of the crystal. To produce white light the 2D stage should be adjusted so that the focal point between the two mirrors is in the middle of the crystal. After white light production is maximized, the white light should be aligned to the detector by adjusting the knobs on the second mirror. The focal point of the white light outside the optics box should be able to overlap with the pump. If the focal point of the white light cannot overlap with the pump the position of the mirrors should be adjusted. **THIS SHOULD BE DONE ONLY WITH THE LCA SUPERVISOR PRESENT. .**

**CaF<sub>2</sub> crystal.** White light generation using this crystal is generally the same. However, special care should be taken with this crystal. The crystal should be mounted on the vibration generation platform, and should be moving anytime the laser is on (otherwise it will destroy the crystal). Care should also be taken so that the generated white light is not being impacted by the moving

crystal on the way to the detector. Additionally, for this crystal to produce stable white light the 775 nm laser hitting it should not exceed 2.5 mW in power. The power should be checked before using this crystal and if the 775 nm laser is too powerful a neutral density filter of sufficient thickness should be put in the optics box.