Photoreduction of Benzophenone

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The objective of this lab is to introduce students to the Nd:YAG laser and its application in photochemistry. The photoreduction of benzophenone is a well known photochemical reaction from which students can learn a lot. The apparatus consists of the following:

1. Surelite/Continuum Nd:YAG pulsed laser with installed 355 nm third harmonic crystal.
2. Surelite Separation Package (SSP) with installed 355 nm dichroic optics.
3. Newport lens holders.
4. Newport iris.
5. Quartz test tubes.
6. Test tube holder.
7. Benzophenone.
8. Isopropyl Alcohol (2-propanol).
9. Glacial Acetic Acid.

Refer to Figure 1. Photoreduction of Benzophenone for schematic diagram.

Reaction(s):

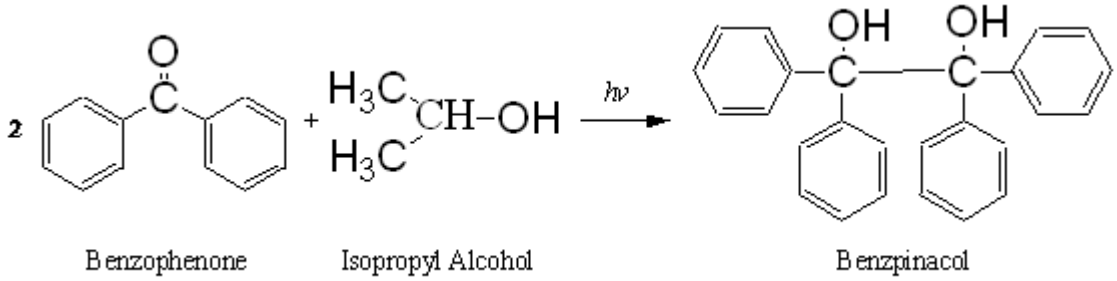

Fig. 1. Photoreduction of benzophenone.

Safety Precautions:

The Nd:YAG laser is a very high-powered laser capable of burning through wood, plastic, some metals, and human skin. Take every precaution to be clear of the beam when the laser is on, even on low power settings.
Also, as always when working with lasers, wear the provided Nd:YAG laser safety goggles. These will protect your eyes from the specific wavelengths given off by the laser.

Glacial acetic acid can cause burns to the skin so handle this chemical carefully. Benzophenone is an irritant. Take precautions to not allow contact with any part of your body.

Laser Procedures:

First a detailed look into the operation of the Nd:YAG laser is essential. There are certain startup and shutdown procedures to follow to allow proper operation of the laser.

Startup:
Check the toggle switch labeled **AC POWER** on the front, upper left corner of the **POWER COOLING UNIT (PCU).** It should be switched on and left on. (If it is not, switch it on. Because of interlocks built into the system if the power is completely cut to the laser it will not lase for up to half an hour, this is normal. Follow the startup procedure and allow the laser head to flash for a half an hour or until the head starts to lase.)
At this time the display should read OFF. Next, find the keyed power switch that is also on the front of the **POWER COOLING UNIT (PCU).** Turn this key counter-clockwise to the ON position. The LED display will flash some numbers that correspond to the number of times the flashlamp has gone off. After the numbers flash the **LASER ON** light will come on and the **PCU** will begin circulating the water. You will be able to hear the pump start.
Now we will focus on the display on the front of the **PCU** and the three buttons below it: up, down, and select. Before you can start lasing you must check the parameters. These are set by using the select button to cycle through the modes and the up and down buttons to change the selected values. There are six modes to verify accuracy of the settings:

Rep Rate	Displayed as **F10**, this is the laser's frequency of pulses in hertz. Leave at **F10**. (Ex. F10=10 Hz)
Q-Switch	Gives a number in μs. A light to the right of the display will light that says **Q-SW DELAY**. Normal value is 188 μs.
Pulse Division	Displayed as **P01**. Flashlamp discharge divider for laser output. Take the frequency of the flashlamp and divide it by the **P##** to get the frequency of the laser output. (Ex. 10hz/01=10hz) Normal value is **P01**.
Pump Voltage	Displayed as 1.57. A light to the right of the display will light up that says **VOLTS (KV).** This is the flashlamp discharge voltage in kilovolts. Normal value is 1.57 kV.
	The next two we have no concern for in this lab. They are displayed as **SoF** and **EoF**, respectively. Leave these off.

Now we are ready to start up the laser head. On the front of the **PCU** push the button under **START/STOP**. The laser head will now flash at the designated frequency. You can also use this button to stop the laser head. Allow the head to lase for 15-20 minutes to thermally stabilize the YAG head (this most likely will be done for you). Now you should put on your safety goggles. Next, open the shutter on the front of the laser bench. Now press the **SHUTTER** button on the front of the **PCU**. An audible click can be heard as the intercavity shutter withdraws from the oscillator. The system should now lase. If harmonic crystals are used in the system the laser output should be allowed to pass through the crystals for five minutes and then optimize the crystals for maximum energy (this most likely will be done for you beforehand).

Shutdown:
First, press the **START/STOP** button on the front of the **PCU**. This will close the shutter and stop the laser head from flashing. Next, close the shutter on the front of the laser bench. Finally, turn the power key clockwise to shutdown the laser. The **LASER ON** light should go out. IMPORTANT!!! Do not shut off the main **AC POWER** toggle switch. This is to be left on at all times, unless maintenance is to be done on the laser or if the laser is not going to be used for an extended amount of time. Remove key and place on the tool board.

Optics Cleaning:

Clean optics are essential during the use of the Nd:YAG laser. If there are any particles or residues left on the optics the laser will burn the particles or residue into the optic rendering it useless. Examine the optics of the SSP and the diverging lenses, carefully. If it is found that the optics are dirty follow this procedure to clean them:

1. Obtain lens paper, gloves, and reagent-grade methanol. These should be available in the lab.
2. Wash your hands to remove oils and residue.
2. Put on gloves and select clean area to work on.
3. Dismount the optics and place on lens paper.
4. Handle optics only on the sides.
5. Put some methanol on a piece of folded over lens paper.
6. Swipe lens paper over the optic face once only. If any residue is left over or it needs further cleaning, use less methanol and a new piece of lens paper and repeat one swipe until clean.
7. Remount optic carefully. Be sure that the optic is mounted facing the same way as before. An arrow on the side will serve as an indicator.

Procedure:

Obtain a 13x100 quartz test tube with a rubber stopper. Rubber stoppers can be obtained from the chemistry stockroom. The quartz test tubes are located in one of the Laser Lab cabinets. Obtain 0.50 grams of benzophenone, 10 ml of isopropyl alcohol (2-propanol), and a drop of glacial acetic acid (Pasteur pipet) from the chemistry stockroom. Warm up

some water on a hot plate (does not need to be boiling). Add 0.50 grams of benzophenone to the test tube. Next, add only 2 ml of isopropyl alcohol to the test tube. Heat this in the warm water bath until the solids dissolve. When the solids have dissolved remove from the water bath and add one drop of glacial acetic acid (Pasteur pipet). Fill the test tube nearly to the top with more isopropyl alcohol. Stopper the test tube and shake well. Place the tube in the test tube holder so it is in the diverged laser beam. Next, follow the laser start-up procedures. Check all parameters to be sure they are on default. Begin lasing and check the tube every 15 minutes to mix the tube and rotate to fully optimize the exposed area of the solution.

Write-up:

After carrying out the foregoing procedure, please document your experiment in the write-up format illustrated by the attachment to this handout.

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