Appendix II – Sample Lab Report

Lab # 9
Cis-Trans Isomerization of 1,2-dibenzoylethylene

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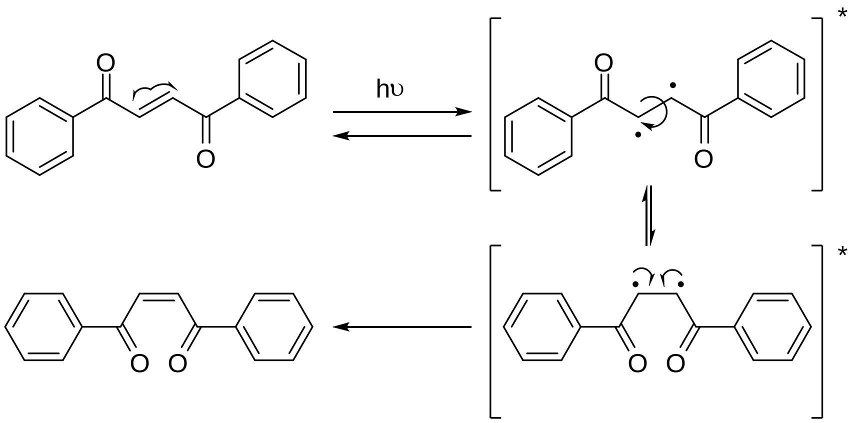
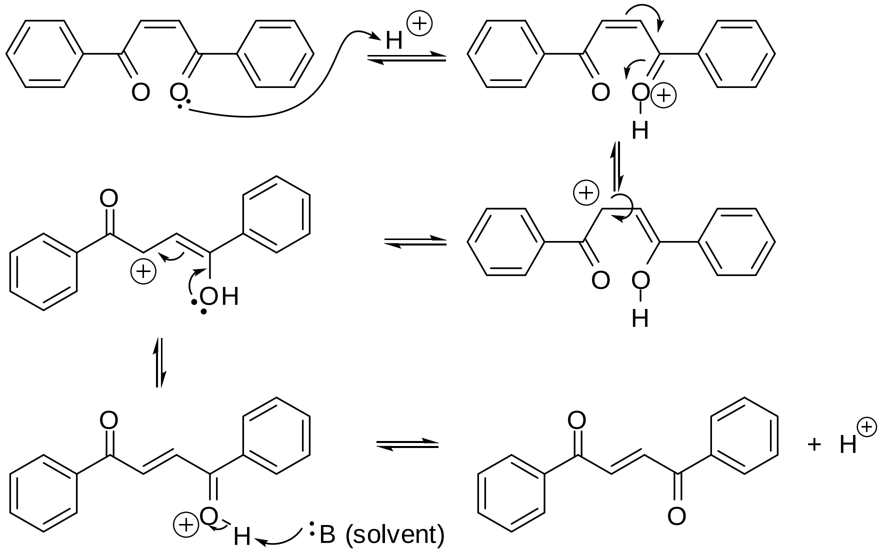
Note: Refer to the section entitled "Reports" at the start of the lab manual for complete instructions for completing a lab report. There are serious consequences for plagiarized lab reports. See the University policy for details.[1]

Mechanism for the *trans* to *cis* isomerization:



After isolating the *cis* compound, it will be converted back into the *trans* form by brief exposure to acid according to the process shown. Acid is a catalyst for this process.

The mechanism is shown below:



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[1] Plagiarism: www.uottawa.ca/plagiarism.pdf. Regulation on academic fraud: web5.uottawa.ca/mcs-smc/academicintegrity/regulation.php

Procedure and observations:

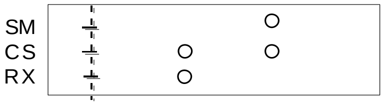
Part A: Isomerization of *trans* 1,2-dibenzoylethylene to *cis* 1,2-dibenzoylethylene.

Table of reagents

Compound	Mol. Wt (g/mol)	Amount	density (g/mL)	mmol
trans 1,2-dibenzoylethylene	236.27	1.01 g		4.275
EtOH	46.07	43 mL	0.790	

Procedure:

- the *trans* 1,2-dibenzoylethylene was dissolved in the ethanol. A bright yellow solution was produced.
- the resulting solution was placed in the photochemical apparatus and irradiated at 280 nm for 2 ½ hours. The solution became a very pale yellow (almost colorless) but remained clear. TLC showed that all of the starting material had been consumed, and a single spot was observed.



CCl_4 : EtOAc
9 : 1
UV + I_2

- The solution was cooled to 0 °C using an ice bath. Crystals formed after 30 minutes. These were filtered and washed with cold ethanol. Drying under vacuum gave 0.49 g of white needles (49 %). Melting point = 130-131 °C. Literature value (Lab manual) = 132 °C

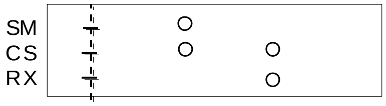
Part B: Isomerization of *cis* 1,2-dibenzoylethylene to *trans* 1,2-dibenzoylethylene.

Table of reagents

Compound	Mol. Wt (g/mol)	Amount	density (g/mL)	mmol
cis 1,2-dibenzoylethylene	236.27	0.29 g		1.23
HCl (12 N)	36.5	1 drop		
EtOH	46.07	21 mL	0.790	

Procedure:

- the *cis* 1,2-dibenzoylethylene was suspended in the ethanol giving a colorless solution.
- while stirring, the solution was heated to reflux. The HCl was then added through the condenser. This initiated the isomerization as shown in the mechanism above
- the solution immediately turned bright yellow. TLC taken immediately showed no starting material remained



CCl_4 : EtOAc
9 : 1
UV + I_2

- flask removed from heat and allowed to cool to room temperature
- no crystals formed, cool in an ice bath 20 minutes, yellow crystals formed. Filtered and washed with cold ethanol. Gave 0.22 g bright yellow needles (76 %). Melting point 107 °C. Literature value (lab manual) = 109 °C.

Discussion:

Part A. In photoisomerization, the starting alkene absorbs UV light and undergoes a π to π* transition. The π bond is no longer present and the molecule is free to rotate around the remaining σ bond. When the molecule relaxes, the π bond re-forms. Depending on the conformation of the molecule at the time that relaxation occurs, the resulting product will have either a *cis* or *trans* configuration.

The wavelength of the light used to excite the alkene is chosen so that only the *trans* isomer absorbs. This is selected by examining the absorption spectra of the *cis* and *trans* forms. The *trans* form shows a λ_{max} of 280 nm whereas the *cis* form has a λ_{max} of 240 nm. By irradiating at 280 nm, only the *trans* form will be excited. Any *cis* product that forms will be unaffected as no π to π* conversion takes place from this compound. As the reaction proceeds, the excited species relax to produce both *cis* and *trans* products. The *trans* molecules produced will be re-excited while the *cis* forms remain unaffected. Over time, all of the *trans* molecules will be converted to *cis*.

Part B. Reconversion to the *trans* form is done by treatment with acid. This process occurs by the mechanism shown in the introduction above. This is an equilibrium process. Because the *trans* form is more stable than the *cis* form, the *trans* compound forms preferentially.

The stability of the *trans* compound arises from a lack of crowding. The *cis* isomer is more sterically crowded, and this increases the energy of the *cis* form relative to the *trans*. The crowding in the *cis* form also forces the carbonyls out of alignment with the alkene thus decreasing conjugation since the overlap of the π orbitals will not be complete. This decreases the extent of conjugation for the *cis* form relative to the *trans*, thus further destabilizing the cis form relative to the *trans*.

Questions.

1) The *cis* isomer has a higher melting point because it has a larger net dipole moment. The trans compound has no net dipole because the individual dipoles of the carbonyl groups cancel each other out. The *cis* isomer has a larger dipole moment, and therefore the molecules in the solid will be held together by stronger dipole-dipole interactions than the *trans* isomer is resulting in a higher melting point for the *cis* compound.