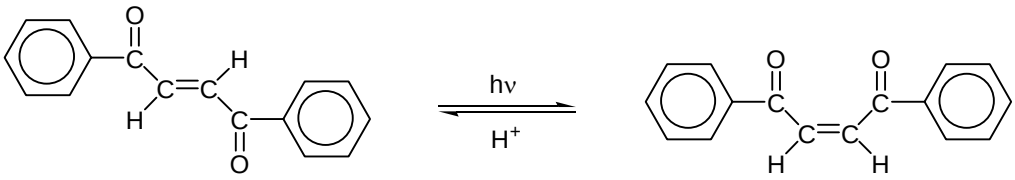
ISOMERIZATION OF 1,2-DIBENZOYLETHYLENE



trans-1,2-dibenzoylethylene
mp 109°

cis-1,2-dibenzoylethylene
mp 132°

READING

Zubrick, Ch. 31
Instrument handout *Using the High Performance Liquid Chromatograph (HPLC)*

PROCEDURE

A. *Trans*-to-*cis* Isomerization

Dissolve 1.0 g of *trans*-1,2-dibenzoylethylene in 50 mL of 95% ethanol in a 125-mL Erlenmeyer flask with steam bath heating (do not let the ethanol boil away). Transfer two drops of this solution to a vial and dilute it with 10.0 mL of HPLC-grade methanol (using a graduated pipet). Immediately obtain the HPLC of this *trans* solution.

Stopper the flask and place it in bright light for one week. At the start of the reaction period, the mixture contains predominantly yellow *trans* crystals, but as the reaction proceeds, the *trans* isomer is slowly converted to the colorless *cis* isomer.

B. *Cis*-to-*trans* Isomerization

After several days exposure to light, chill the flask in an ice bath and remove the crystals by vacuum filtration. Recrystallize the colorless *cis* product from ethanol, dry the product over vacuum, obtain its mass, and reserve a small amount for a melting point.

Dissolve 100 mg of your purified *cis* product in 3.8 mL of 95% ethanol with heating if necessary (do not let the ethanol boil away). When dissolved, transfer two drops of this solution to a vial and dilute it with 10.0 mL of HPLC-grade methanol. Obtain the HPLC of this *cis* product solution.

To the remaining warm ethanolic solution, add 3 drops of HCl (*conc*) and continue warming the solution on a steam bath for 5 minutes. (Again, do not allow ethanol to boil dry!) Transfer two drops of this solution to a vial and dilute it with 10.0 mL of methanol. Obtain the HPLC of this diluted "after HCl" solution. Chill the remaining solution and isolate the solid by suction filtration, drying over vacuum for 15-20 min. Take the mass and melting point.

Include all HPLC traces, properly labeled and pasted, in your notebook. Calculate retention times and areas for all peaks, and identify all peaks as much as possible (justify how you know). Use the areas to calculate the % *cis* and *trans* isomer in each solution. Report percent yields for each of your isomerization reactions.