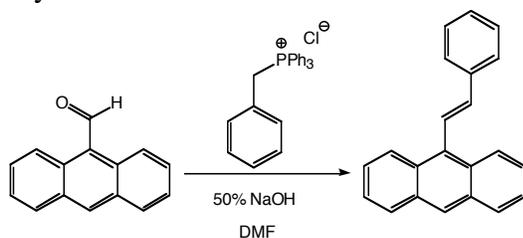


# CHEM333 LAB EXPERIMENT 9

## THE WITTIG REACTION

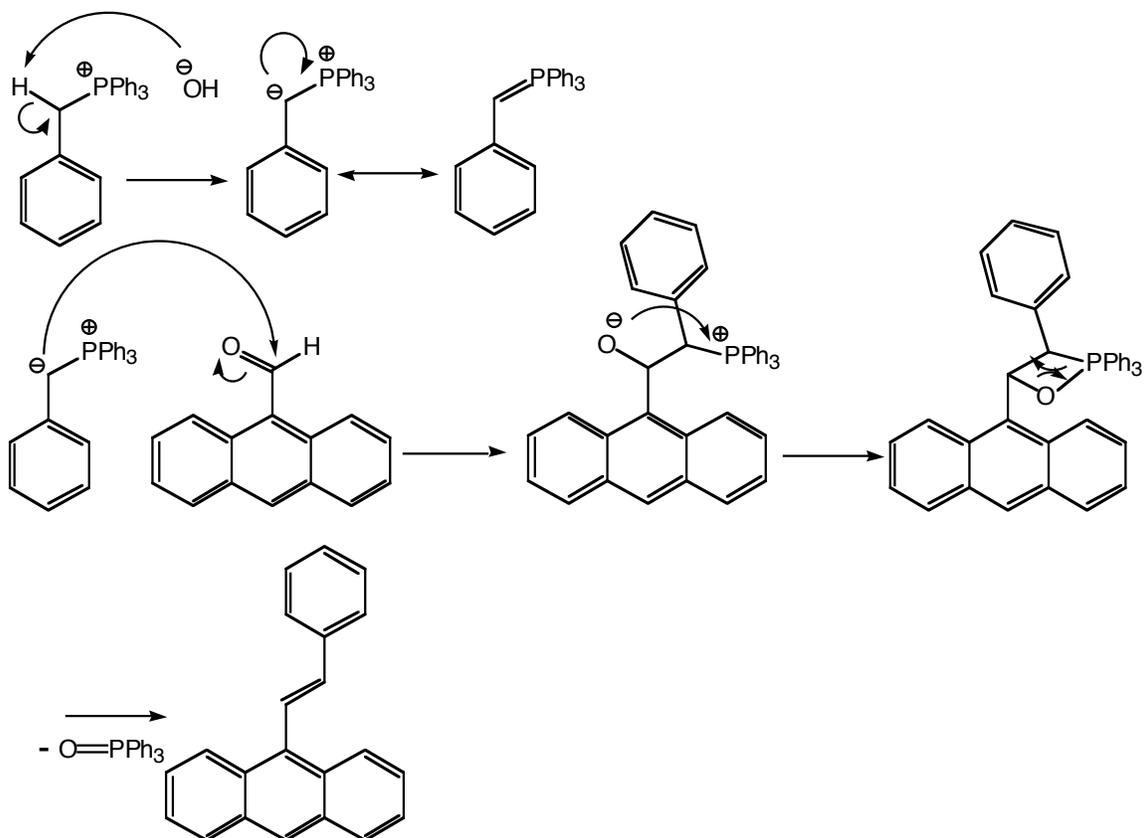
### WITH CHEMILUMINESCENCE!

In the Wittig reaction, an aldehyde or ketone is treated with a phosphorous ylide (also called a phosphorane) to give an olefin. Phosphorus ylides are usually prepared by treatment of a phosphonium salt with a base, and phosphonium salts are usually prepared from the phosphine and an alkyl halide.



The key step of the mechanism is the formation of the oxaphosphetane, the cyclic intermediate. Wittig reactions give primarily Z olefins but often some of the E, and others give a mixture.

#### Mechanism:



## PART I. Synthesis of trans-9-(2-Phenylethenyl)anthracene

In a 25-mL Erlenmeyer flask place a small stir bar, 0.50g (0.00242 moles) of 9-anthraldehyde, 0.87g (0.00223 moles) of benzyltriphenylphosphonium chloride. Dissolve the mixture in 6 mL of DMF. Vigorously stir the mixture for at least 5 minutes. Carefully added 0.200 uL (10 drops) of 50% sodium hydroxide (50% NaOH w/w) solution to the rapidly stirred reaction mixture. Use the DMF in the flask to wash any solid off the walls of the Erlenmeyer flask. Note: The reaction changes color during this period, starting out dark yellowish and ending reddish-orange after 30 minutes.

After stirring vigorously for 30 minutes, added 4 mL of a 1:1 of 1-propanol/distilled water to precipitate the product (yellow solid). Collect the crude product by vacuum filtration.

Recrystallize the crude product with as little 1-propanol as possible (ca. 4 mL). After recrystallization, a yellowish crystalline solid is obtained 73.5% (0.50g).

TLC conditions: (eluent: hexanes/ethyl acetate 5:1);

$R_f$  product = 0.75, blue spot and

$R_f$  anthraldehyde = 0.50, yellow spot.

DCM = Dichloromethane

DMF = N,N'-Dimethylformamide

NaOH = Sodium Hydroxide

Note:

Use a Pasteur pipette with bulb to measure out the 0.200 uL (10 drops) of 50% NaOH. The pipette must be held exactly vertically straight for the desired amount.

## Part II. Procedure for Chemiluminescence

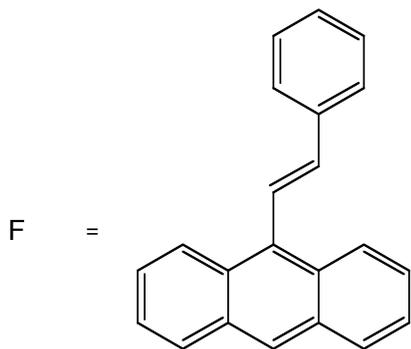
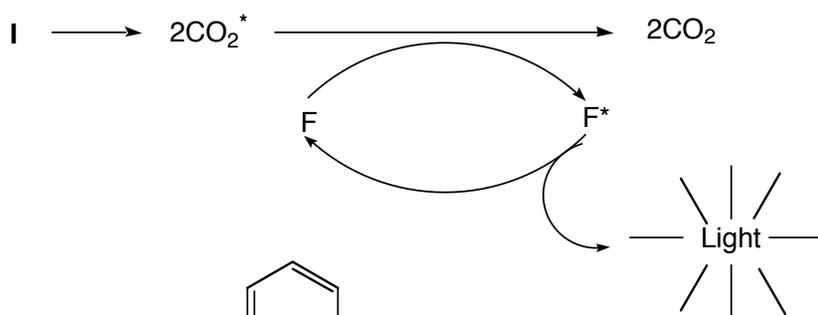
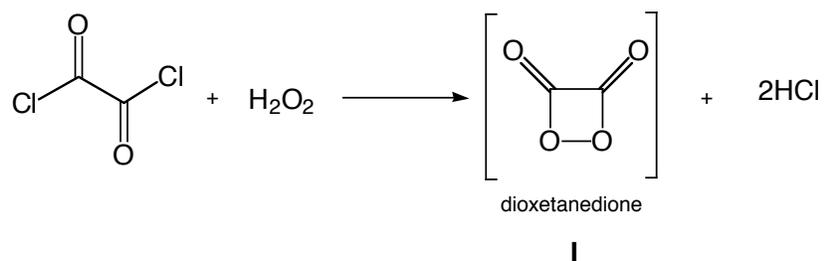
In a 13-100mm test tube, placed 2 mL of 10% oxalyl chloride/dichloromethane solution, 2 mL of DCM, 1 mg of the fluororescer, and 2 mL of 30% hydrogen peroxide. Take the test tube to a dark area with a Pasteur pipette and bulb mix the solution thoroughly. Record observations.

Color: Light-blue color lasts for 10 seconds and turns purple color for an additional 15 to 30 seconds.

SAFETY NOTE:

Caution should be use when handling the following reagents due to their corrosive properties: dichloromethane, sodium hydroxide, oxalyl chloride, and hydrogen peroxide. Wear gloves at all times.

## Mechanism for Chemiluminescence



Note for Lab Services:

The 10% oxalyl chloride in dichloromethane should be made Fresh. In addition, each lab section should have its own bottle. If the bottle is left uncapped, results will differ from section to section therefore it is best for each lab section to have their own.