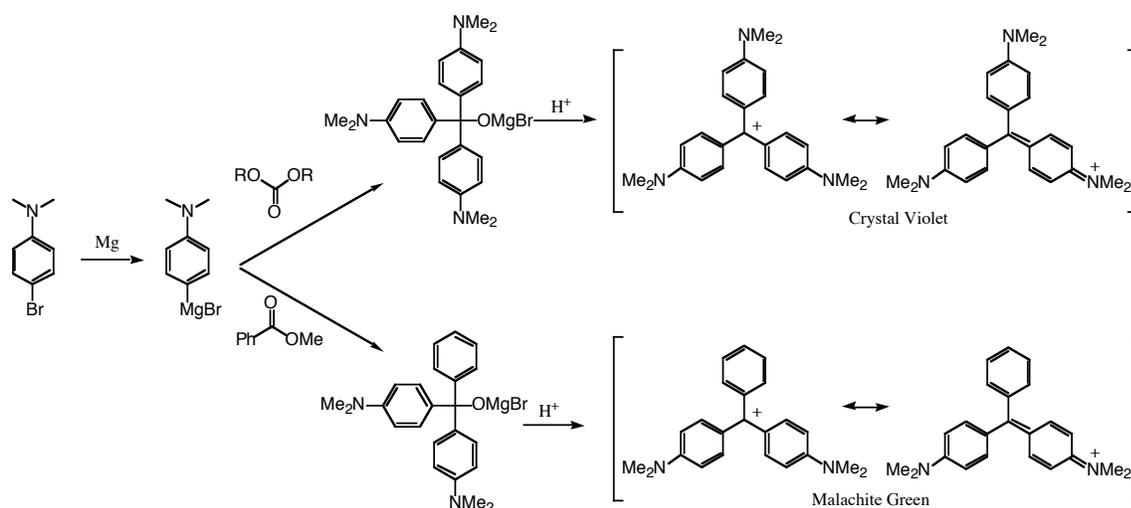


CHEM-333: EXPERIMENT 10: GRIGNARD REAGENT PREPARATION AND REACTION:

READ THIS HANDOUT CAREFULLY! This **MUST** be read before entering the laboratory in addition to Chapter 19.

OVERVIEW:

This experiment concerns the Grignard synthesis of a dye. Analysis will be by inspection of the final product as applied to a cloth sample.



PROCEDURE:

For the success of the Grignard reaction, it is essential that all glassware that is clean and dry. Fit a 100 mL one neck round bottom flask with a condenser. The bottom fitting of the condenser should be connected to a hose that supplies water, and the top fitting on the condenser should be outletted to a drain hose. The apparatus should be held in place by two clamps affixed to a ring stand, and a heating mantle should be placed under the round bottomed flask. Obtain the following from your laboratory instructor:

0.5 g of *p*-bromo-*N,N*-dimethylaniline

0.8 g of Mg turnings

50 mL of anhydrous tetrahydrofuran (THF)

a catalytic amount (enough to cover 1/2 of the tip of a microspatula) of iodine.

YOU HAVE A CHOICE OF ESTERS, PICK ONE:

a) 0.59 g of diethyl carbonate in a small test tube, to make Crystal Violet, or

b) 0.85 g of methyl benzoate in a small test tube, to make Malachite Green

To dry the glassware, rinse the condenser and flask with a few mL of anhydrous THF (discard into waste solvent). With the condenser removed, add the bromide to the flask, and use the remaining THF to ensure that all of the material is transferred to the round bottom. Sequentially add the Mg and the iodine. Replace the condenser and swirl the mixture. Make sure that the water is flowing through the condenser **gently**. Begin heating the flask (use the 1-2 setting), and allow the mixture to reflux gently. (**If reflux becomes too vigorous, remove the heating mantle immediately!**). Allow reflux to continue for 30 minutes with swirling. The Mg will be consumed and the dark color will eventually disappear (approx. 20 minutes) and be replaced by a solution that looks like 'dirty dishwater' – as is typical of Grignard reagents. Allow the system **to cool to room temperature**, using a cold water bath.

With a pipette, dropwise add the methyl benzoate or diethyl carbonate to the Grignard reagent at room temperature. Swirl the solution as you do this, as the reaction will be exothermic. Add 1 mL of THF to the test tube that contained the benzoate or carbonate, so that the last traces can be transferred to the reaction vessel. Warm the solution to reflux temperature, and swirl the mixture for 5 min. Cool the solution to room temperature, and then transfer the contents to a beaker (250mL or 400 mL). Obtain 10 mL of 5% aqueous HCl and **slowly** add this to the beaker. **Residual Mg will be consumed, and the solution will foam- if this happens too rapidly, it will foam out of the flask!** Obtain a cloth sample and dip it into the dye solution using forceps. Contact time should be at least 1 min for best results. Remove cloth, rinse with tap water and blot dry. If performed correctly you will notice an intense color.

If time permits there are variations that can be incorporated into the process. For example, one can "paint" the dyes onto fabric in varying patterns, but use your own imagination. PLEASE NOTE: the purity of the dye is NOT suitable for clothing or any other routine application. Treat it like any other laboratory chemical - **MINIMAL CONTACT**.

Your report should include observations of color changes the reaction goes through as well as the cloth sample. You will be graded on the quality of your sample rather than yield. The Grignard is water sensitive so once the glassware has been rinsed with THF it is to be loaded and assembled directly to minimize exposure to the air. These dyes are potent! Wear old clothes to lab, avoid contact with the dye and wash promptly. As

always, wash glassware before returning it to drawer. Any chemicals spilled on self must be washed off immediately!

Excess dye must be disposed of in the chemical waste container