Phosphorus Chemistry - Experimental Procedures

Inorganic Chemistry I (CHE-258), Spring 2004

Week 1

1. Reaction of Phosphorus Trichloride with Methanol

Apparatus

Place a stirring bar in a 50 mL 19/22 round bottom flask, then put a Claisen adapter on the flask. Stopper the center opening with a serum stopper, and attach a reflux condenser to the side arm. Insert a medicine dropper point-first into a thermometer adapter, remove the bulb from the medicine dropper; put this assembly into the joint at the top of the condenser.

Place a rubber tube on the medicine dropper, then attach the other end of the tube to the stem of a funnel. Clamp the funnel above water in a beaker so as to have the funnel mouth close to the surface of the water (note: the funnel should **not** be beneath the surface).

Reaction

Place 20 mL of dry methylene chloride (dichloromethane) in the flask, then add 3.0 ml of anhydrous methanol, by syringe, through the serum stopper (the methanol will be dispensed from a Sure-Seal bottle fitted with a calcium chloride drying tube as the air inlet). Place an ice bath around the reaction flask.

Turn on the cooling water for the condenser, then, **in the hood**, measure out 2.2 mL of phosphorus trichloride. Insert the syringe through the serum stopper and start the magnetic stirrer. Over a period of ca. 10 minutes, add the phosphorus trichloride dropwise to the reaction mixture. Remove the ice bath and leave the reaction mixture to stir for ca. one hour. Give the reaction mixture to your instructor for product isolation.

Thoroughly rinse the syringe and needle that were used for dispensing phosphorus trichloride with methylene chloride.

2. Reaction of the Product from Procedure 1 with Benzylamine

Place 20 mL of methylene chloride in a 50 mL Erlenmeyer flask. **In the hood**, add, by syringe, 0.92 mL of the product from Procedure 1, 2.25 mL of benzylamine, and 1.2 mL of carbon tetrachloride.

Stopper the flask tightly with a **cork** (**not** a rubber stopper), seal the flask with Parafilm, label it with your name, and leave it on the shelf under the laboratory windows until your next session.

Week 2

In the hood, filter off any solid from Procedure 2 and allow it to dry. Evaporate the solvent and volatile product to obtain a second solid product.

Your instructors will obtain IR and NMR spectral data for your products.