Nitration of Methyl Benzoate TUD Department of Chemistry

When methyl benzoate is treated with a mixture of H_2SO_4 and HNO_3 the aromatic ring undergoes electrophilic aromatic substitution and one or more of the three possible mono-nitrated products shown below may be formed. In this experiment we will carry out this nitration reaction and determine which of the possible products is formed.



Add 6 mL of concentrated $H_2SO_4^1$ to a 50 mL Erlenmeyer flask and cool to near 0° C in an ice bath. Add 3.0 g of methyl benzoate to the flask and again cool to near 0° C. Over a period of 5–10 minutes, add a cooled mixture of 2 mL H_2SO_4 plus 2 mL HNO₃ to the flask. Add the acid mixture drop by drop with frequent swirling and keep the reaction mixture between 5 and 10° C by returning to the ice bath as necessary. After all of the acid mixture has been added allow the flask to return to room temperature and stand for 20 minutes. Pour the mixture onto 25 g of ice and stir until the ice has melted. Collect the solid on a filter and wash with several small portions of aqueous NaHCO₃, several small portions of ice-water, and two 5 mL portions of ice cold methanol. Recrystallize the product from methanol and place the product in the desiccator to dry. When the product has dried determine the %yield and the melting point and obtain an IR spectrum. Deduce which of the possible products was produced

¹ You will be working with corrosive and toxic materials in this experiment. Wear gloves and an apron and if any of the acids or products contact your skin wash well with soap and water.