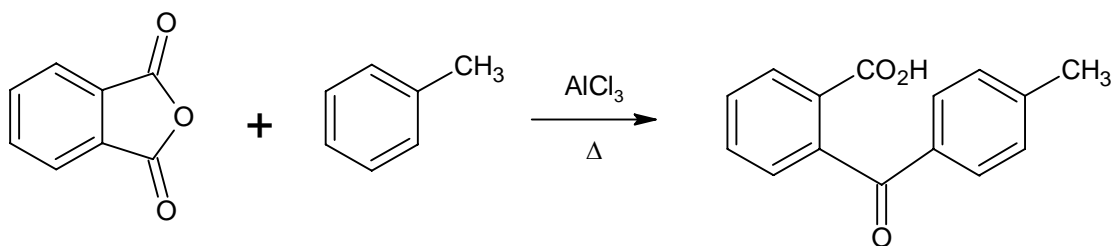


# Synthesis of *p*-Toluylo-benzoic Acid

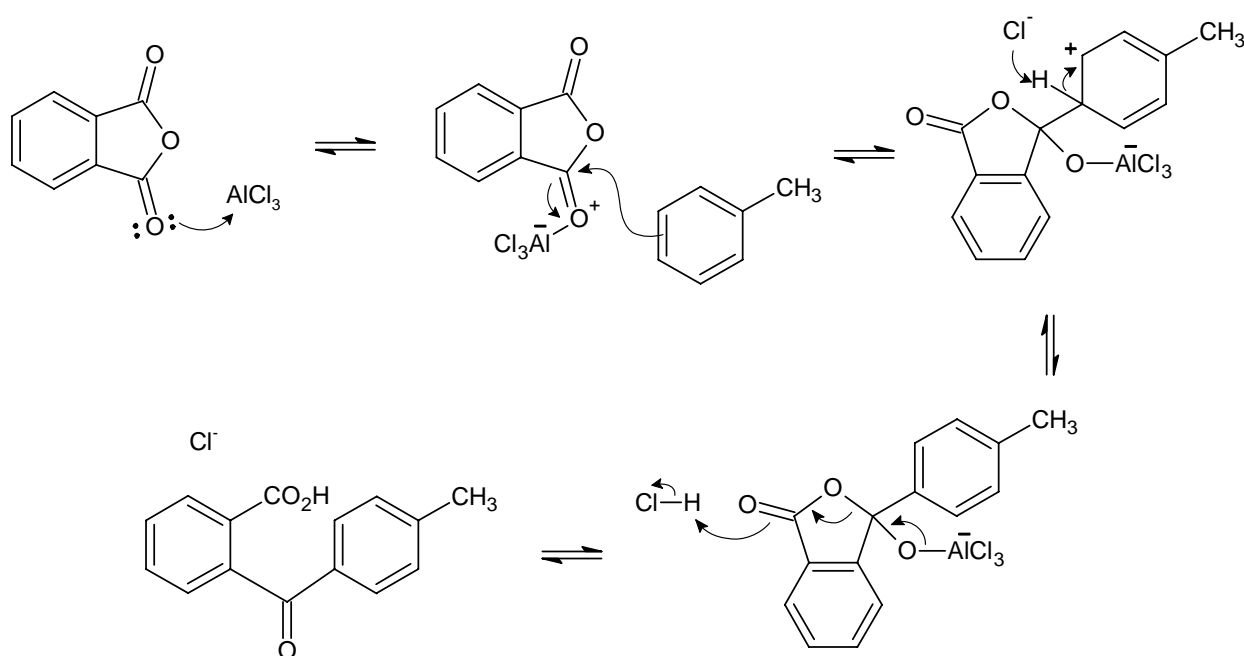
Matt Koutroulis  
October 17, 2008  
Chemistry 231

Title: Synthesis of *p*-toluyl-*o*-benzoic acid

Chemical Equation:



Reaction Mechanism:



Discussion and Results

2.01 grams (13.5 mmol) of phthalic anhydride is combined with 10.08 grams (109.4 mmol) of toluene in a 25-mL round-bottom flask. The solution remains clear and colorless. After cooling the solution in an ice bath for 10 minutes, 5.05 grams (37.9 mmol) of anhydrous aluminum chloride is added to the flask rapidly and with stirring. The mixture immediately takes on a dark black color. The flask is fitted with a condenser topped with a thermometer adapter which is attached to rubber tubing which terminates in a glass funnel suspended over a beaker of water, serving as a trap for the hydrogen chloride gas evolved in the reaction. After stirring for 15 minutes, the ice bath is removed and replaced with a water bath. The temperature of the bath is adjusted to 90. °C and the reaction is allowed to continue for 90 minutes. Hydrogen chloride gas is evolved rapidly for the first 30 minutes of the

reaction, after which the amount produced diminishes considerably. Thin-layer chromatography on silica plates using a 1:1 eluent of hexanes and ethyl acetate is used to confirm that the reactions has completed ( $R_{f[\text{phthalic anhydride}]} = 0.48$ ,  $R_{f[\text{product}]} = 0.78$ ).

The mixture is allowed to cool to room temperature. The round bottom flask is carefully removed from the rest of the apparatus and carried to the fume hood, where the contents of the flask are poured into a beaker containing approximately 50 milliliters of ice water. The mixture is carefully stirred with a glass rod until all the ice has melted. Fifteen milliliters of concentrated hydrochloric acid is added to the flask in 3 milliliter portions over ten minutes, during which time the solution becomes clear. The flask is capped and placed in a locker drawer.

After two days, the contents of the round bottom flask remain clear but have developed a faint amber color. Etc.

*At this point in the report, the remainder of the experimental procedure and observations would be described in the same style as above.*

The compound is purified by recrystallization from toluene, producing white plates which melt at 137.5-139.0 °C (the published value is 138-139 °C). 2.53 grams of purified product is ultimately collected, which corresponds to a percent yield of 78.0%. While this recovery is lower than the amount predicted in the literature (96%) it is not unreasonable for the scale at which this experiment was performed. A significant amount of product was lost in the collection of the crude product as well as in the isolation of the recrystallized compound.

*Notice that the experimenter (i.e. the student) never mentions themselves or their emotions ("I was happy...", "It was sad to see..." etc.) anywhere in the text of the discussion! Also note the pervasive use of the passive voice.*

*Finally, observe that the correct number of significant digits have been reported for all measurements, although this may not be obvious without reference to the laboratory pages*

### Analysis of Spectra

An infrared spectrum of the final product was obtained employing the ATR apparatus. The broad signal observed on the left side of the spectrum is the characteristic stretch of the O-H bond of a carboxylic

acid. The sharp, forked signals at 1670-1700  $\text{cm}^{-1}$  correspond to the distinct stretching signals of the carbonyl carbon and oxygen atoms in the ketone and carboxylic acid functional groups.

A proton NMR spectrum of the product dissolved in  $\text{CDCl}_3$  was provided by the instructor. Two distinct singlets are observed: one at 11.3 ppm corresponding to the acid proton, and one at 2.4 ppm corresponding to the methyl protons. Two distinct groups of overlapping multiplets are observed in the region extending from 7.0 ppm to 8.2 ppm; however, at this level of resolution (90 MHz) it is not possible to identify which of the aromatic protons are giving rise to each signal. A carbon-13 spectrum was not provided.

### References

Fieser, L.F. "*p*-toluyl-*o*-benzoic acid". *Organic Synthesis*, Collective Vol. 1, p. 517 (1941).

Koutroulis, M.R. *Organic Chemistry Laboratory Notebook*, p.32-36.

### Post-Lab Questions

1. The text of the question would go here. Suppose this question does not require the use of mathematical and/or chemical symbols.

*The response to the question would go here.*

4. The text of the question would go here (assuming questions 2 and 3 were not assigned). Suppose that this question does require the use of mathematical and/or chemical symbols.

*The response to the question could go here if you are able to generate the required figures using your computer. Otherwise, enter the statement "See attached pages" and attach the responses on a piece of paper immediately following the end of this section. Be sure to number the question on the attached page.*

*Copies of pages from the lab notebook (in the correct order!) should follow this section. Finally, any (preferably annotated) spectra obtained or given to you by the instructor would be attached at the end of the report.*