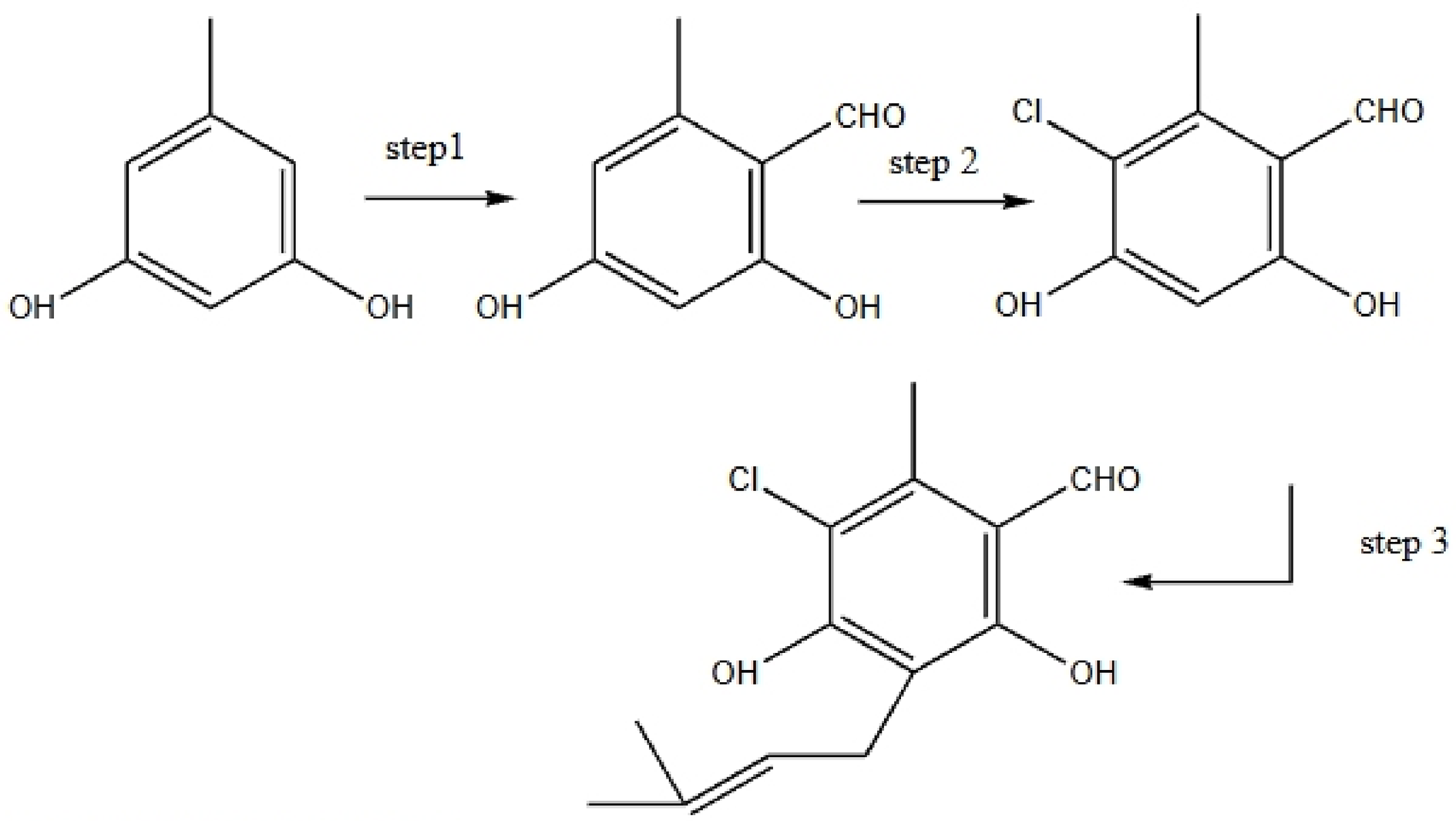
Honors Cup Synthetic Proposal

Section: 231
Group Members: Andrew Lewandoski, Brian Meredith, Phil Yun

Title: A Three Step Synthesis of Colletochlorin D

Introduction: (what makes your target interesting?)
Colletochlorin D is one of 4 molecules found in a fungus that causes a disease of the tobbacco plant. We liken this process to the synthesis of a poison or disease-causing agent, but one not dangerous to us.

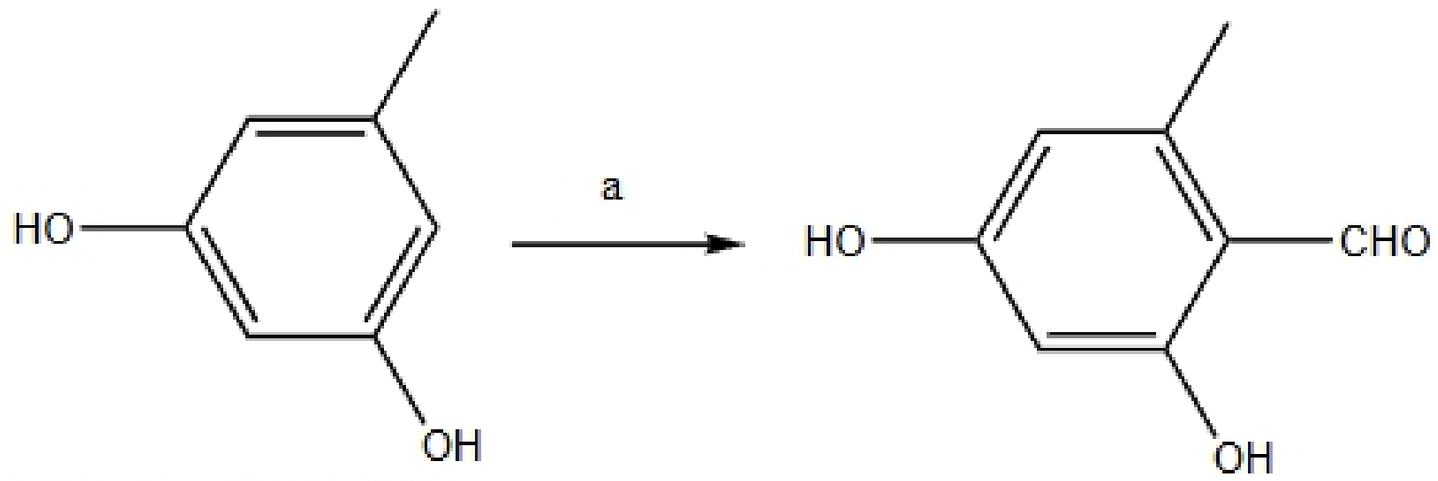
Overall synthetic reaction scheme: (a Chemdraw or similar drawing of all three steps)



Step 1: $Zn(CN)_2$, Et_2O, HCl
Step 2: SO_2Cl_2, Et_2O, 0degreesC
Step 3: 1-bromo-3-methylpenta-2-ene, KOH, H2O

Step 1

Synthetic transformation 1: (Chemdraw picture of first transformation)



a: $Zn(CN)_2$, Et_20, HCl

Experimental 1 (notes if this transformation is not exactly the one reported in literature (e.g. on a different scale) and how it was modified):

Add 5g orcinol to solution of ether (125ml) in round bottom flask. Then add 1 mol equivalent of zinc cyanide (3.66g) and stir for .5 hours. Dry HCl added and the product then separates into oil. After 10-30 min, oil turns to solid. Add water to product to decompose HCl. Let sit overnight (or more) and then filter product. (this entire process is known as the Gattermann process, and can be found online.)

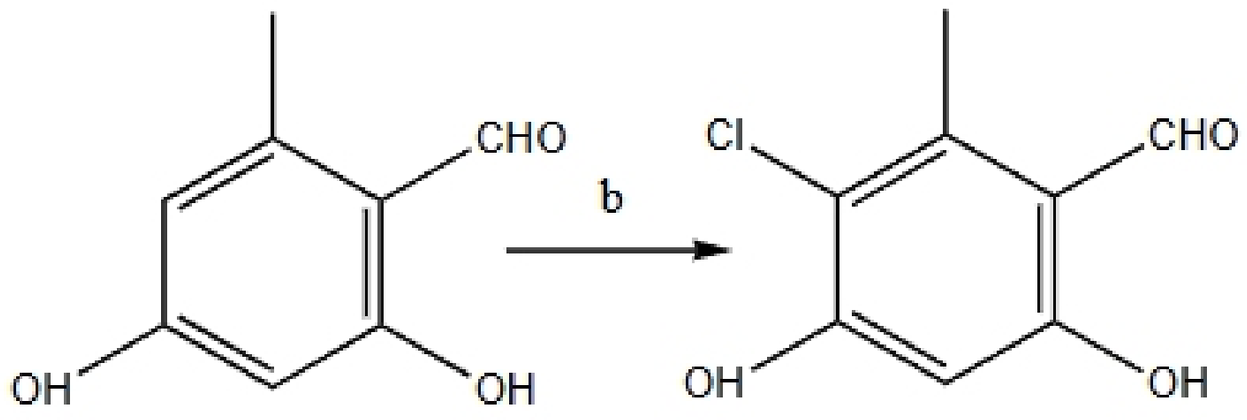
Expected yield: 76-85 % 3.36g

Safety, disposal and green issues 1:

Safety issues for this involve the use of dry HCL. Be sure to use this in the hood. Disposal of the products should be done very carefully and into correct containers. In addition to this, the students involved in synthesis should be properly dressed and very careful during the lab.

Step 2

Synthetic transformation 2: (Chemdraw picture of second transformation)



b: SO_2Cl_2, Et_2O, 0°C

Experimental 2 (notes if this transformation is not exactly the one reported in literature (e.g. on a different scale) and how it was modified):
In a round-bottomed flask, the aromatic aldehyde (1.11g, 7.3mmol) formed in experimental 1 is stirred into solution in dry ether (125mL) in a ice water bath. Sulfuryl chloride (1.08g, 8mmol) is dissolved in dry ether (25mL) and added dropwise into the stirred solution. The reaction is stirred for 1.5 hours in the ice water bath, and then at room temperature for 3 hours. A TLC (hexane:EtOAc, 6:1) is then run to assess the disappearance of the starting material. The solution is then washed with NaHCO3, saturated NaCl solution and water. It is dried over MgSO4, filtered, and concentrated in vacuo. The product is then separated via flash column chromatography with 15% ethyl acetate in hexane as the eluant.
*Altered the time of stirring in room temperature from 8 to 3 hours. This may result in a slightly smaller yield than expected.

Expected yield: 76.5 % 1.14 g

Safety, disposal and green issues 2:
Make sure gloves, goggles, and close-toed shoes are worn at all times while working with chemicals.
Dispose of gloves and other solids in the solid waste container.
All reactions should be carried out under a hood.