

How do I react phthalic acid with an alkanol?

The following procedure has been recommended by an experienced user. The chosen alcohol is methanol:

1. Weigh out phthalic acid and dissolve it in acetone (e.g., 50 mg phthalic acid in 50 mL acetone for a 1 mg/mL solution).
2. Transfer the needed volume into a culture tube for esterification (e.g., 500 μ L).
3. Dry completely under nitrogen on a warm hotplate or place into a 70 °C oven until the sample is free of water. There will be a layer of white phthalic acid adhering to the bottom.
4. Add a sufficient amount of boron trifluoride in methanol (BF_3/MeOH).
5. Heat at 80 °C for 1 hour, then let cool.
6. Add 2 mL of hexane and shake vigorously to dissolve the dimethyl phthalate into hexane.
7. Add 3 mL of a 5 wt. % aqueous solution of NaCl and shake vigorously to transfer boron trifluoride into the water/MeOH phase, then allow phases to separate from each other.
8. Pipette the topmost phase (hexane) into a glass vial.
9. Extract the leftover water/MeOH phase two more times with hexane and combine all hexane extracts into one glass vial.
10. Add some anhydrous sodium sulfate to dry the hexane extract, then separate the supernatant hexane from the drying agent and transfer to a different vial.
11. Condense the dry hexane extract to few milliliters. Calculate the final concentration of dimethyl phthalate from the originally used phthalic acid to determine how much you will need for a GC-FID measurement.
12. Use an appropriate amount for testing via GC-FID.

The typical yield for this procedure is ~70 %, which is close to the maximum yield cited in textbooks for this type of reaction. A low yield of this reaction (e.g., 45 %) will shift the $\delta^2\text{H}$ by a few permil to more negative values.