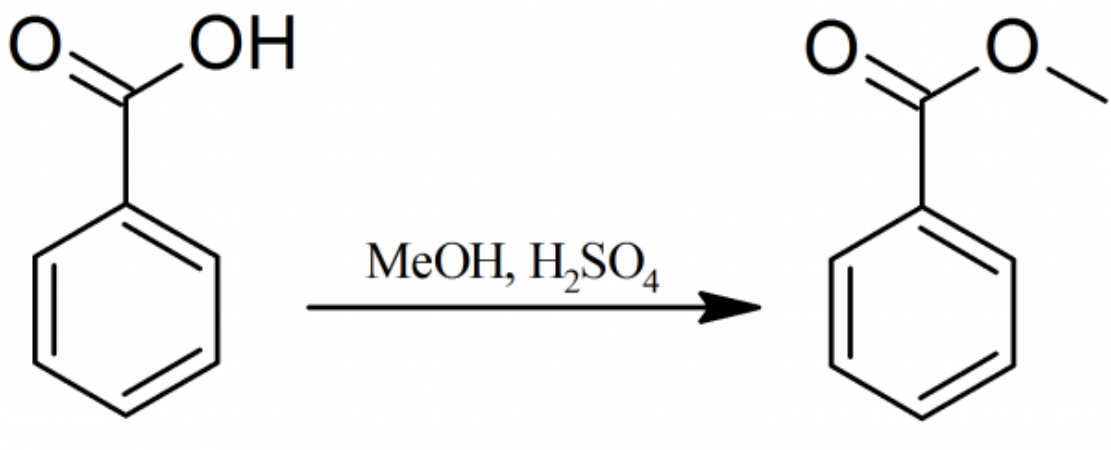

In a 500 ml round-bottomed flask, a mixture of 24.4 g. of benzoic acid (0.2 mole), 70 ml of methyl alcohol, and 7 ml of concentrated sulfuric acid are is placed. A boiling chip is added, a reflux condenser is attached, and the mixture is boiled gently on the steam bath for one hour. The condenser is arranged for downward distillation and the excess alcohol is distilled as long as it comes over easily (the residue should be about one-third to one-half the original volume). The flask is then cooled well under the tap and the residue is poured into a separatory funnel and treated with 70 ml of ether, using a part of this to rinse the flask. The ethereal solution is then washed thoroughly with two 50 ml portions of water in order to remove most of the unreacted alcohol and the sulfuric acid. The mixture is then shaken with 50 ml of 10% sodium bicarbonate solution in order to remove the last traces of sulfuric acid and to separate the ester from any unreacted benzoic acid. The washing with bicarbonate is repeated until no precipitate forms on acidification of the aqueous layer. The ethereal solution is then separated carefully, poured into a dry flask, and dried with about 5 grams of anhydrous potassium carbonate for at least one-half hour. The dried solution is filtered into a distilling flask, the vessel being rinsed with a little ether. The ether is removed by distillation and the distillation of the ester itself is carried out over a free flame (no gauze). The boiling point of methyl benzoate is 199° C, so a water-cooled condenser is not only unnecessary but inadvisable, on account of the danger of cracking the condenser. An air condenser tube is employed for the distillation procedure. A small fore-run should be collected separately and the main fraction boiling at 190-196° C is collected yielding 20 grams of methyl benzoate.

Experiments in Organic Chemistry, L. F. Fieser, 62-64, 1941