

Synthesis of ethyl 4-(butylamino)benzoate

93.9g (0.48 mol) p-nitrobenzoate and 138.2 g (4.32 moles) methanol were added to a 1L four-necked flask equipped with a stirrer, a spherical condenser, a thermometer and a constant pressure dropping funnel. And then 38.1g (0.53 mol) n-butyraldehyde and 124.8g (1.92 mol) zinc powder were added there too. After cooling to 0-5, 230.4g (3.84 mol) acetic acid was added to the system with 0.5h. And then room temperature reaction and TLC tracking reaction started, and this process spent about 2h. After the reaction, light brown yellow transparent liquid can be obtained by direct pumping, and the filter cake was washed with water (50 mL × 3). In addition, the generated salt was removed and the zinc powder was recovered meanwhile, the mother liquor obtained by suction filtration was evaporated under reduced pressure and the methanol was used for recovery. The remaining part was dissolved using 200mL ethyl acetate, and the salt and acetic acid were removed by washing. And then the organic phase was dried. 100.8g ethyl p-butylamino benzoate crude can be obtained with 94.7% yield. The target product of nuclear magnetic resonance spectrum was shown in Fig. 2.