2022.01.25

Reaction 1-1:



t-BuOK

A - CAS 1409971-87-4; Mn = 255.23; Density = 1.583 g/cm3;

B - CAS 540-36-3; Mn = 114.09; Density = 1.1725 g/cm3;

C - CAS 865-47-4; Mn = 112.21;

Solution - N,N-dimethylformamide

A - 1mmol = 255.23 mg

B - 3mmol = 342.27 mg

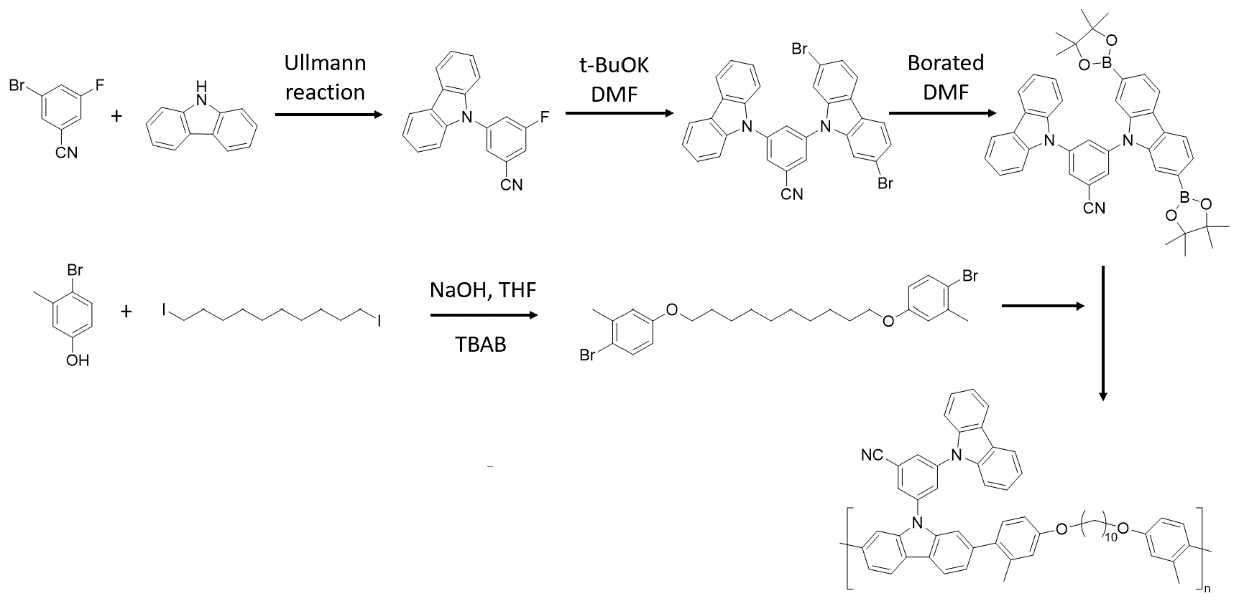
C - 1mmol = 112.21 mg

Solution – 15mL

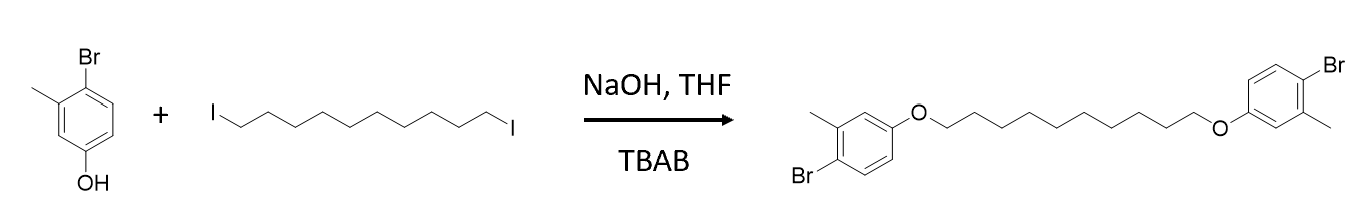
A mixture of A (255.23 mg, 1 mmol) and B (342.27 mg, 3 mmol) in N,N-dimethylformamide (15 mL) was stirred for 15 min under argon at room temperature, and then the reaction mixture was heated up to 110 °C and potassium tert-butoxide (112.21 g, 1 mmol) was added and stirred for 12 h. The reaction was quenched with water (20 mL), and precipitated in methanol, filtered by vacuum, and washed with methanol three times to obtain the crude product

Yield = \_\_\_\_ %

Route 2



2022.02.03



A – CAS 14472-14-1; Mn = 187.03;

B – CAS 16355-92-3; Mn = 394.08;

C – CAS 1310-73-2; Mn = 39.997;

D – CAS 1643-19-2; Mn = 322.37;

Solution - THF

A – 15mmol = 2.805 g

B – 6mmol = 2.364 g

C – 15mmol = 0.6g

D – 0.15mmol = 48.356 mg

Solution - 12+3 mL

A solution of A, B and D in 12 mL THF were stirred for 30 min under nitrogen at 65. C in 3 mL THF was added and stirred for 12h. After cooling to room temperature, the solution was poured into methanol. The precipitate was collected by filtration and washed with water/methanol. Finally, it was purified by column chromatography on silica gel using hexane/DCM (v/v = 4:1) as eluent to give white solid products.