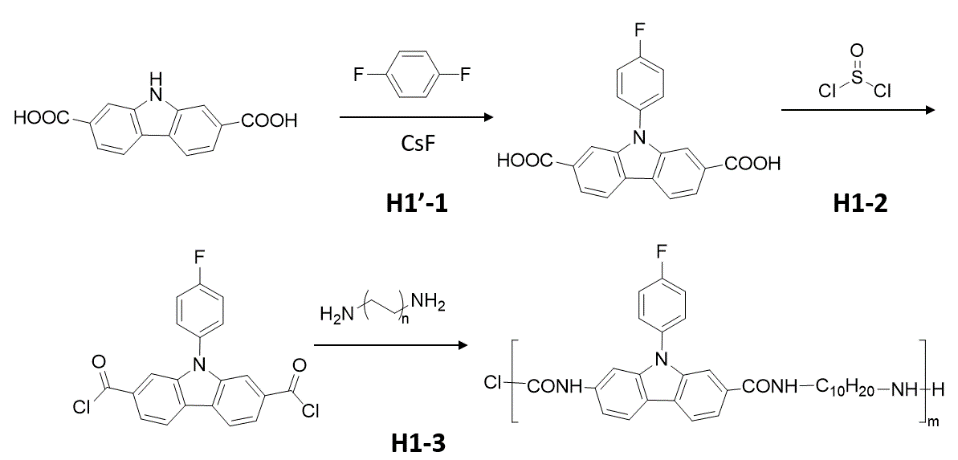
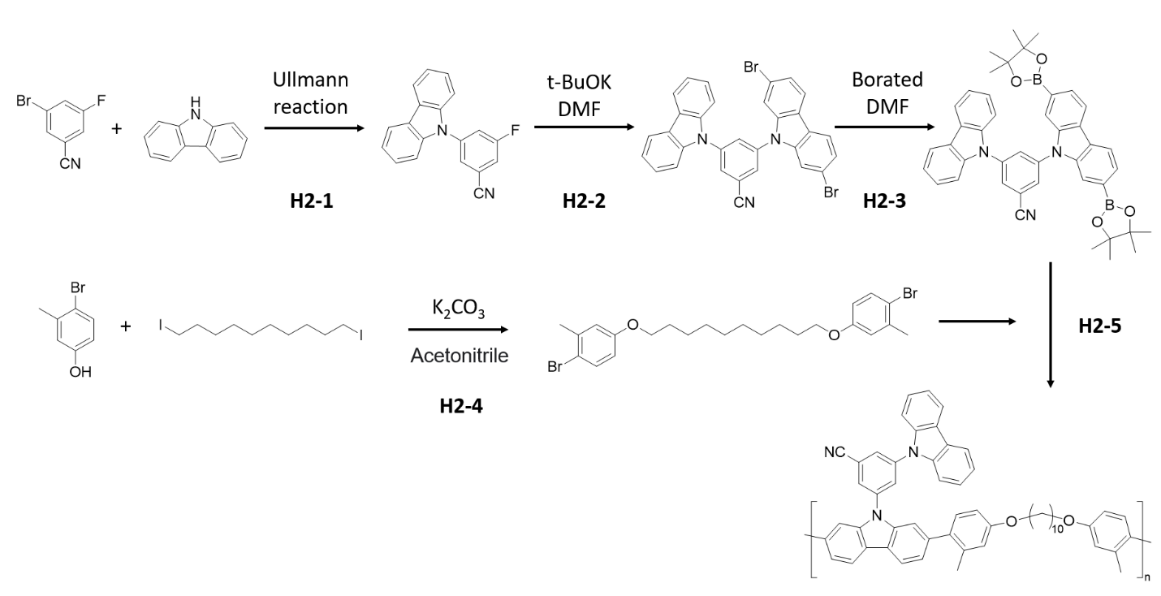
Routes

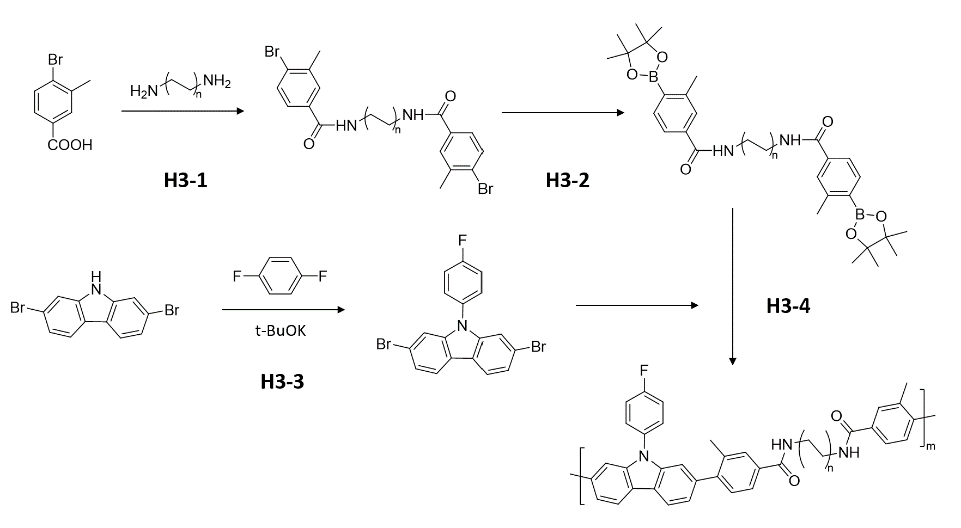
Host1:



Host2 (H2):



Host3 (H3):



2022.02.22

Reaction H1-1:



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 – 1 mmol = 255.23 mg

B – 3 mmol = 342.27 mg

C - ~~1mmol = 112.21 mg~~ 3.5 mmol = 392.74 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 mg, 1 mmol~~ 392.74 mg, 3.5 mmol) was added and stirred for 12 h. The reaction was quenched with water (20 mL), and precipitated in ~~methanol~~ dilute HCl, and washed with DI water three times to remove DMF. Use column chromatography to get the pure product.

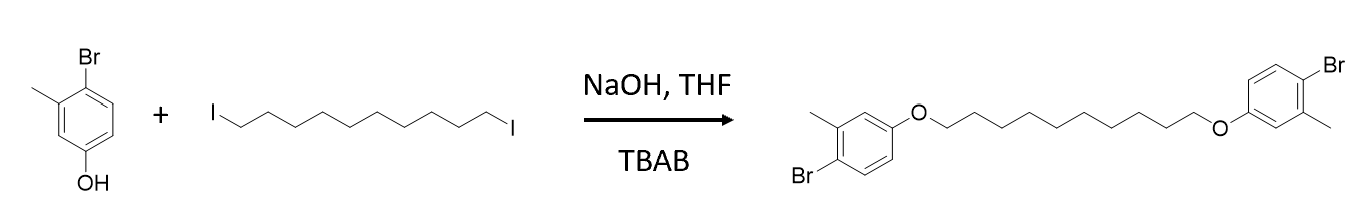
Yield = \_\_\_\_ %

2022.02.22 (tiral1) - low yield – C should be at least 3 times higher than A.

2022.02.25 (trial 2) - low yield – A wasn’t deprotonated well.

2022.02.03

Reaction H2-4



A – CAS 14472-14-1; Mn = 187.03;

B – CAS 16355-92-3; Mn = 394.08;

C – CAS 584-08-7; Mn = 138.21;

Solution – Acetonitrile

Product – Mn = 510.08;

A – 15 mmol = 2.805 g

B – 5 mmol = 1.970 g

C – 15 mmol = 2.073 g

Solution - 45 mL

A solution of A, B and C in 45 mL Acetonitrile under nitrogen at 95 for 12h. After cooling to room temperature, the solution was poured into DI water. The precipitate was collected by filtration and washed with water (light yellow). Finally, it was purified by column chromatography on silica gel using hexane/DCM (v/v = 4:1) as eluent to give white solid products.

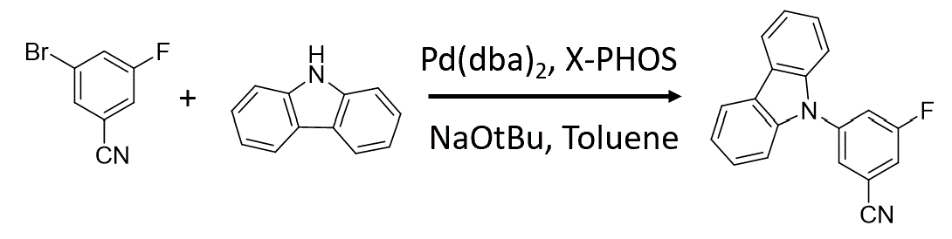
Notes:

1. The filtration is slow. Needs to switch filter paper.
2. Use Hexane to wash the final product.

Yield = \_\_\_/2.5504 = ~ 43 %

2022-02-21

Reaction H2-1



A – CAS 179898-34-1; Mn = 200.01

B – CAS 86-74-8; Mn = 167.21

C – CAS 32005-36-0; Mn = 575.00

D – CAS 564483-18-7; Mn = 476.72

E – CAS 865-48-5; Mn = 97.11

Solution – Toluene

Product – Mn = 286.09

A – 16 mmol = 3.200 g

B – ~~8 mmol = 1.338 g~~ 12 mmol = 2.007 g

C – 0.3 mmol = 0.1725 g

D – 0.9 mmol = 0.429 g

E – 20 mmol = 1.942 g

Solution – ~~35mL~~ 40mL

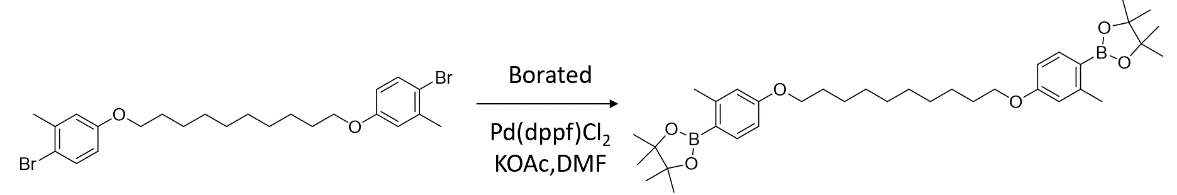
A solution of A,B,C,D and E in 35 mL toluene under N2 at 85 for 12h.

2022.02.24 (trial 1) – low yield – F was replaced by carbazole, reacted for too long time.

2022.02.28 (trial 2) -

2022.02.14

Reaction H2-s1



A – CAS NA; Mn = 510.08;

B – CAS 73183-34-3; Mn = 253.94;

C – CAS 72287-26-4; Mn = 731.72;

D – CAS 127-08-2; Mn = 98.15;

Solution – ~~DMF~~ 1,4-dioxane

Product – Mn = 606.46

A – 2mmol = 1.020 g

B – 6mmol = 1.524 g

C – 0.1mmol = 0.073 g

D – 6mmol = 0.5889 g

Solution – 15 mL

A solution of A,B,C,D and E in 15 mL degassed DMF under N2 at 100 for 12h. After cooling to room temperature, the solution was poured into 150 mL saturated sodium chloride solution and filtered by vacuum to obtain the crude product, purified by column chromatography on silica gel (short ~7cm) using (DCM/Hex= 3:1), then DCM as eluent (don’t use THF). give the product (bright yellow powder, 0.58g, yield 36.0%).

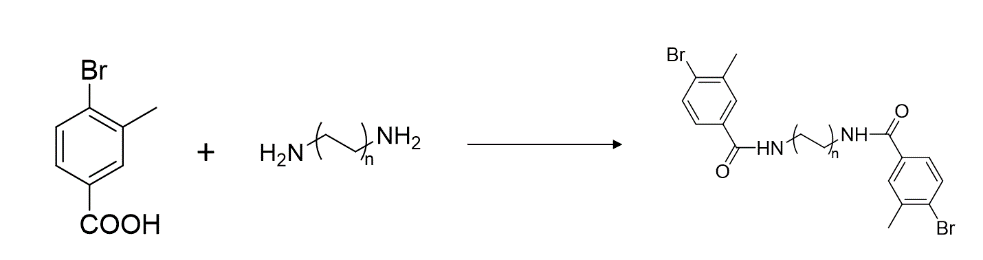
Yield = Low!

Notes:

1. Use 1,4-dioxane as the solvent.

2022.02.28

Reaction H3-1

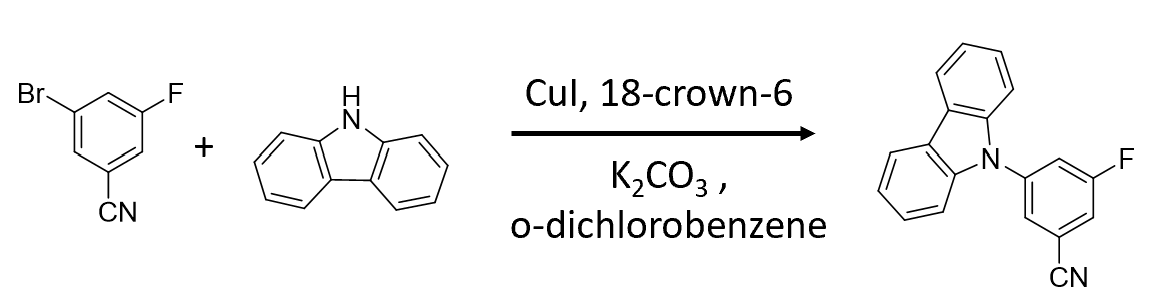


A – CAS 7697-28-1; Mn = 215.04;

B – CAS 646-25-3; Mn = 172.31;

2022.03.04

Reaction h2-s1



A – CAS 179898-34-1; Mn = 200.01

B – CAS 86-74-8; Mn = 167.21

C – CAS 7681-65-4; Mn = 190.45

D – CAS 17455-13-9; Mn = 264.122

E – CAS 584-08-7; Mn = 138.2055

Solvent o-dichlorobenzene

Product – Mn = 286.09

A – 12 mmol = 2.4 g

B – 10 mmol = 1.6721 g

C – 1 mmol = 0.1905 g

D – 1.5 mmol = 0.3962 g

E – 30 mmol = 4.1462 g

Solvent = 40 mL

A, B, C, D and E were added in the degassed E. Reacted at 180 for 48 h. After cooling to R.T., the mixture was quenched with saturated (NH4)2CO3 solution and extracted with dichloromethane, the combined organic layer was washed with distilled water three times, separated and dried over anhydrous magnesium sulfate. Then the solvent was evaporated in vacuum to give the crude product, which was purified by column chromatography on silica gel using (petroleum ether: DCM = 20:1) as eluent.

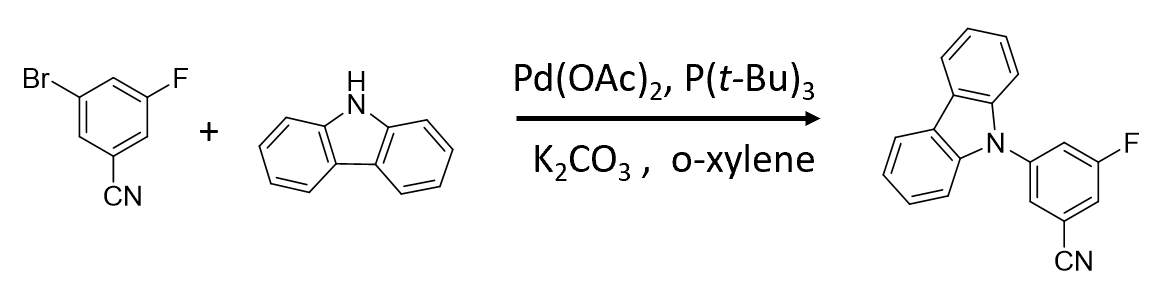
Yield = /2.861 g = % Low!

Notes:

1. Many side reactions leads to low yield and hard to purify.

22.03.08

Reaction h2-s1



A – CAS 179898-34-1; Mn = 200.01

B – CAS 86-74-8; Mn = 167.21

C – CAS 3375-31-3; Mn = 224.51

D – CAS 13716-12-6; Mn = 202.32

E – CAS 584-08-7; Mn = 138.21

Solvent – Toluene

Product – Mn = 286.09

A – 4.14 mmol = 828.234 mg

B – 3.7647 mmol = 629.496 mg

C – 0.2 mmol = 44.3871 mg

D – 0.6 mmol = 120 mg

E – 11.28 mmol = 1.561 g

Solvent – 30 mL

A, B, C, D and E were added to 30 mL toluene. 100 for 14 hours.

Yield = /1.077 g = % low

Notes:

1. Degas the solvent should be necessary.

A – 1 mmol = 207.0585 mg B – 0.94 mmol = 157.374 mg

C – 0.05 mmol = 11.097 mg D – 0.15 mmol = 30 mg

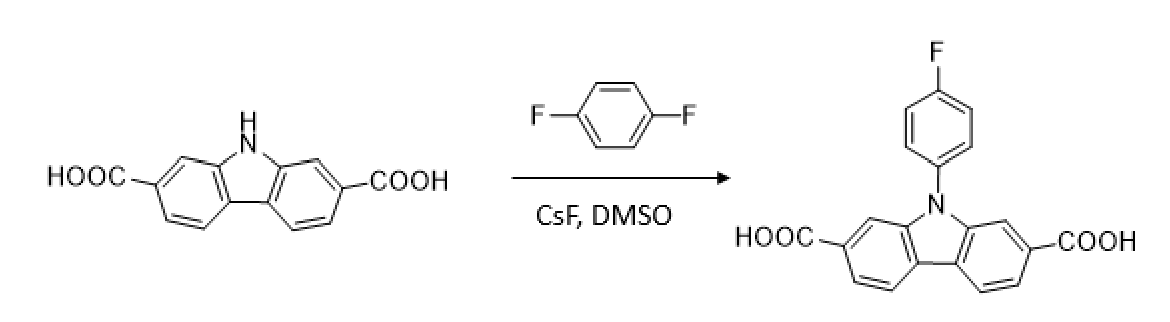
E – 2.82 mmol = 390.25 mg Solvent – 7 mL

Notes:

1. Low yield.

2022.03.09

Reaction h1-1



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 13400-13-0; Mn = 151.903;

Solvent – DMSO

Product – Mn = 349.32

A – 1 mmol = 255.23 mg

B – 3 mmol = 342.27 mg

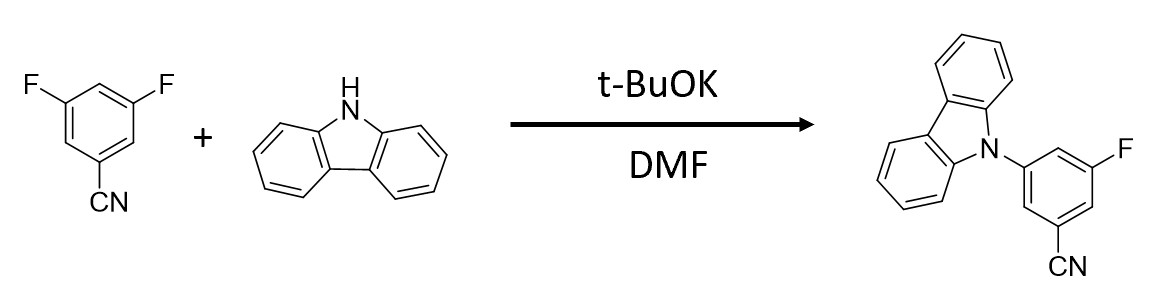
C – 6 mmol = 911.418 mg

Solvent – 20 mL

C in 20 mL solvent was stirred at R.T. To the solution, A and B were added in sequence. The mixture was heated with stirring at 150 for 12h and then precipitated into 80 mL Methanol.

2022.03.14

Reaction h2-1s



A – CAS 64248-63-1; Mn = 139.10

B – CAS 86-74-8; Mn = 167.21

C – CAS 865-47-4; Mn = 112.21;

Solvent – N,N-dimethylformamide

A – 7 mmol = 0.9737 g

B – 6 mmol = 1.0033 g

C – 6 mmol = 0.6733 g

Solvent – 50 mL (dehydrated DMF)

B in 20 mL DMF was added to C in 20 mL DMF. After 3h, A in 10 mL DMF was added. 8h, 80.