Electronic Supplementary Material (ESI)

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Supporting Information

Base-promoted synthesis of diarylsulfones from sulfonyl hydrazines and diaryliodonium salts

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1. General

All commercial reagents were used directly without further purification, unless otherwise stated. CDCl₃ was purchased from Shanghai aladdin Biochemical Technology Co., Ltd. All Schlenk tubes and sealed vessels (50 mL) were purchased from Beijing Synthware Glass. The following abbreviations were used to describe NMR signals: s = singlet, d = doublet, t = triplet, m = mulitplet, dd = doublet of doublets, q = quartet.

2. Experimental sections

2.1 The synthesis of sulfonyl hydrazides¹

Hydrazine monohydrochloride (6.0 mmol) and NaOH (6.6 mmol) was added to water (30 mL) and was cooled to 0 °C. A solution of arylsulfonyl chloride (3.0 mmol) in THF (10 mL) was added by dropwise at 0 °C. The mixture was further stirred at 0 °C for 30 min, followed by addition of EtOAc (20 mL). The mixture was extracted with water (3×10 mL). The combined organic extract was concentrated and concentrated by silica gel column chromatography to provide the desired product.

2.2 General procedure for diphenyliodonium triflate²

Various symmetrical and unsymmetrical diphenyliodonium triflates were synthesized by us according to published methods.

2.3 Optimization of reaction conditions

 Table S1. Base and Solvents Screening^a



Entry	Base	Solvent	Yield(%) ^b
1°	Na_2CO_3 (1.0 eq)	CH ₃ CN	trace
2	Na_2CO_3 (1.0 eq)	CH ₃ CN	37

3	K_2CO_3 (1.0 eq)	CH ₃ CN	30
4	NaHCO ₃ (1.0 eq)	CH ₃ CN	55
5	DABCO (1.0 eq)	CH ₃ CN	31
6	$Et_{3}N(1.0 eq)$	CH ₃ CN	35
7	-	CH ₃ CN	20
8	NaHCO ₃ (0.5 eq)	CH ₃ CN	43
9	$NaHCO_3$ (1.5 eq)	CH ₃ CN	37
10	NaHCO ₃ (1.0 eq)	DMSO	51
11	NaHCO ₃ (1.0 eq)	DMF	39
12	NaHCO ₃ (1.0 eq)	Dioxane	56
13	NaHCO ₃ (1.0 eq)	DCE	23
14	NaHCO ₃ (1.0 eq)	H_2O	48
15	NaHCO ₃ (1.0 eq)	EtOH	64
16	NaHCO ₃ (1.0 eq)	<i>i</i> -PrOH	40
17	NaHCO ₃ (1.0 eq)	<i>n</i> -PrOH	65
18	NaHCO ₃ (1.0 eq)	glycol	20
19	NaHCO ₃ (1.0 eq)	glycerol	35
20	NaHCO ₃ (1.0 eq)	TFE	39
21	NaHCO ₃ (1.0 eq)	HFIP	35

^{*a*} Reaction conditions: **1a** (0.2 mmol), **2a** (0.2 mmol, 1.0 equiv.), solvent (2 mL), 100 °C under an atmosphere of N_2 for 24 h. ^{*b*} Isolated yield. ^{*c*} CuI (10 mol%).

Table S2. Variation of temperature, time and molar ratio in optimization of reaction conditions^a

	O, O S NHN	VH ₂ +	NaHCO n-PrOI		o C
	1a	2a		3a	l
Entry	1a/mmol	2a /mmol	T/ºC	Time/h	Yield (%) ^{b}
1	0.2	0.2	100	6	56
2	0.2	0.2	100	12	70
3	0.2	0.2	100	18	75
4	0.2	0.2	80	18	66
5	0.2	0.2	90	18	73
6	0.2	0.2	110	18	64
7	0.2	0.2	120	18	64
8	0.2	0.3	100	18	90
9	0.2	0.4	100	18	83
10	0.3	0.2	100	18	74

11 ^c	0.2	0.3	100	18	67
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^{*a*} Reaction conditions: NaHCO₃ (1.0 equiv.), *n*-PrOH (2 mL), under N₂. ^{*b*} Isolated yield. ^{*c*} Under air.

3. GC-MS analysis of reaction mixture.



Figure S1

4. Data for the sulfone products



1-methyl-4-(phenylsulfonyl)benzene (3a)³

White solid; mp: 117.7-119.2 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.93 (d, *J* = 7.7 Hz, 2H), 7.83 (d, *J* = 8.3 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 2H), 2.39 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 144.17, 141.92, 138.58, 133.00, 129.92, 129.22, 127.70, 127.48, 21.57.



Sulfonyldibenzene (3b)⁴

White solid (41 mg, 94%); mp: 123.0-123.7 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.98 – 7.92 (m, 4H), 7.57 (t, *J* = 7.4 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 4H). ¹³C NMR (125 MHz, CDCl₃) δ 141.54, 133.20, 129.28, 127.64.



1-ethyl-4-(phenylsulfonyl)benzene (3c)⁵

White solid (41 mg, 83%); mp:120.7-122.1 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.94 (d, *J* = 7.5 Hz, 2H), 7.86 (d, *J* = 8.4 Hz, 2H), 7.54 (t, *J* = 7.3 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 2.68 (q, *J* = 7.6 Hz, 2H), 1.22 (t, *J* = 7.6 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 149.24, 140.88, 137.71, 131.98, 128.19, 127.75, 126.75, 126.47, 27.78, 14.05.



4-(phenylsulfonyl)-1,1'-biphenyl (3d)³

White solid (55.3 mg, 94%); mp: 144.7-145.6 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.00 (t, J = 8.5 Hz, 4H), 7.70 (d, J = 8.5 Hz, 2H), 7.57 (dd, J = 6.5, 5.1 Hz, 3H), 7.52 (t, J = 7.4 Hz, 2H), 7.46 (t, J = 7.4 Hz, 2H), 7.40 (t, J = 7.3 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃) δ 146.16, 141.69, 140.07, 139.13, 133.19, 129.32, 129.04, 128.58, 128.19, 127.93, 127.63, 127.33.



1-(tert-butyl)-4-(phenylsulfonyl)benzene (3e)⁶

White solid (47.8 mg, 87%); mp: 156.8-159.1 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.95 (d, *J* = 7.4 Hz, 2H), 7.87 (d, *J* = 8.7 Hz, 2H), 7.59 – 7.47 (m, 5H), 1.30 (s, 9H). ¹³C NMR (125 MHz, CDCl₃) δ 157.06, 141.88, 138.48, 133.01, 129.21, 127.57, 127.50, 126.30, 35.16, 31.01.



1-nitro-4-(phenylsulfonyl)benzene (3f)³

White solid (36.4 mg, 69%); mp: 143.6- 145.0°C; ¹H NMR (500 MHz, CDCl₃) δ 8.35 (d, J = 8.9 Hz, 2H), 8.14 (d, J = 8.9 Hz, 2H), 7.98 (d, J = 7.7 Hz, 2H), 7.65 (t, J = 7.4 Hz, 1H), 7.57 (t, J = 7.7 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 150.35, 147.36, 140.00, 134.19, 129.75, 129.02, 128.06, 124.58.



4-(phenylsulfonyl)benzonitrile (3g)³

White solid (29.6 mg, 61%); mp: 124.9- 125.7 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.06 (d, J = 8.4 Hz, 2H), 7.96 (d, J = 7.6 Hz, 2H), 7.81 (d, J = 8.4 Hz, 2H), 7.64 (t, J = 7.4 Hz, 1H), 7.56 (t, J = 7.7 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 145.86, 140.11, 134.08, 133.13, 129.70, 128.31, 128.01, 117.19, 116.94.



1-fluoro-4-(phenylsulfonyl)benzene (3h)⁴

White solid (43.5 mg, 92%); mp: 110.9- 113.1 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.00 – 7.91 (m, 4H), 7.52 (s, 3H), 7.18 (t, *J* = 8.6 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 165.42(d, *J* = 255.78 Hz), 141.42, 137.64(d, *J* = 3.78 Hz), 133.34, 130.48(d, *J* = 10.08 Hz), 129.39, 127.56, 116.61(d, *J* = 22.68 Hz).



1-chloro-4-(phenylsulfonyl)benzene (3i)⁴

White solid (44.1 mg, 87%); mp: 90.3- 92.2 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.96 – 7.91 (m, 2H), 7.91 – 7.86 (m, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.55 – 7.45 (m, 4H). ¹³C NMR (125 MHz, CDCl₃) δ 141.16, 140.09, 139.90, 133.45, 129.62, 129.42, 129.12, 127.63.



1-bromo-4-(phenylsulfonyl)benzene (3j)⁴

White solid (47 mg, 79%); mp: 105.9 – 106.6 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.93 (dd, J = 8.3, 1.3 Hz, 2H), 7.81 (d, J = 8.6 Hz, 2H), 7.64 (d, J = 8.5 Hz, 2H), 7.59 (t, J = 7.4 Hz, 1H), 7.52 (t, J = 7.6 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 141.10, 140.63, 133.47, 132.60, 129.43, 129.19, 128.46, 127.64.



1-(phenylsulfonyl)-4-(trifluoromethyl)benzene (3k)⁶

White solid (50.6 mg, 88%); mp: 89.7-90.5 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.08 (d, J = 8.2 Hz, 2H), 8.00 – 7.94 (m, 2H), 7.77 (d, J = 8.3 Hz, 2H), 7.58 (dt, J = 36.2, 7.3

Hz, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 145.18, 140.54, 134.84 (q, *J*= 32.76 Hz), 133.81, 129.56, 128.21, 127.90, 123.46 (q, *J*= 3.78 Hz), 123.09 (q, *J*= 273.42 Hz).



1-(phenylsulfonyl)-4-(trifluoromethoxy)benzene (31)⁷

White solid (53.9 mg, 89%); mp:59.3-60.7 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.00 (d, *J* = 8.9 Hz, 2H), 7.98 – 7.93 (m, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 2H), 7.33 (d, *J* = 8.7 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 152.55, 141.04, 139.92, 133.57, 129.90, 129.49, 127.74, 123.26, 121.10, 120.16(q, J= 260.82 Hz).



1,4-dimethyl-2-(phenylsulfonyl)benzene (3m)⁸

White solid (41.9 mg, 85%); mp: 117.1-119.3°C; ¹H NMR (500 MHz, CDCl₃) δ 8.05 (s, 1H), 7.88 – 7.84 (m, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.29 (d, *J* = 7.7 Hz, 1H), 7.11 (d, *J* = 7.7 Hz, 1H), 2.42 (s, 3H), 2.37 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 141.41, 138.32, 136.49, 134.82, 134.40, 132.97, 132.64, 129.75, 129.02, 127.59, 20.94, 19.73.



1,2-dimethoxy-4-(phenylsulfonyl)benzene (3n)⁸

White solid (49.9 mg, 89%); mp: 112.3-113.3 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.91 (dd, J = 7.1, 1.5 Hz, 2H), 7.58 – 7.45 (m, 4H), 7.37 (d, J = 2.1 Hz, 1H), 6.92 (d, J = 8.5 Hz, 1H), 3.89 (d, J = 3.0 Hz, 6H). ¹³C NMR (125 MHz, CDCl₃) δ 153.05, 149.26, 142.24, 133.05, 132.94, 129.25, 127.26, 121.94, 110.87, 109.86, 56.28, 56.23.



2-(phenylsulfonyl)naphthalene (30)³

White solid (49.2 mg, 91%); mp: 113.5 – 115.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.59 (s, 1H), 8.04 – 7.96 (m, 3H), 7.93 (d, *J* = 8.7 Hz, 1H), 7.90 – 7.83 (m, 2H), 7.66 – 7.58 (m, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.50 (t, *J* = 7.4 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 141.59, 138.35, 134.99, 133.19, 132.19, 129.66, 129.40, 129.29, 129.16, 129.09, 127.92, 127.70, 127.64, 122.67.



6-(phenylsulfonyl)-2,3-dihydrobenzofuran (3p)

White solid(41.7 mg, 80%), R_f: 0.51(PE/EtOAc=6/1); mp: 121.1 – 122.0 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.94 – 7.89 (m, 2H), 7.75 (d, *J* = 7.0 Hz, 2H), 7.54 (t, *J* = 7.3 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 2H), 6.83 (d, *J* = 9.1 Hz, 1H), 4.65 (t, *J* = 8.8 Hz, 2H), 3.24 (t, *J* = 8.8 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 164.33, 142.51, 133.02, 132.76, 129.47, 129.19, 128.67, 127.24, 124.93, 109.78, 72.37, 28.97. HRMS (ESI) calcd for C₁₄H₁₂O₃S (M + H)⁺ 261.0507, found 261.0576.



8-(phenylsulfonyl)quinoline (3q)⁴

White solid (34.3 mg, 64%); mp: 117.7 – 119.2 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.96 (d, *J* = 2.6 Hz, 1H), 8.72 (d, *J* = 7.4 Hz, 1H), 8.24 (d, *J* = 7.4 Hz, 2H), 8.17 (d, *J* = 8.3 Hz, 1H), 8.07 (d, *J* = 8.2 Hz, 1H), 7.69 (t, *J* = 7.8 Hz, 1H), 7.52 (t, *J* = 7.3 Hz, 1H), 7.49 – 7.42 (m, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 151.19, 143.71, 141.78, 137.80, 136.33, 134.66, 132.92, 131.82, 129.19, 128.93, 128.31, 125.51, 122.11.



2-(phenylsulfonyl)thiophene (3r)³

White solid (12.5 mg, 28%); mp:120.5- 122.1 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.03 – 7.98 (m, 2H), 7.70 (dd, J = 3.8, 1.3 Hz, 1H), 7.65 (dd, J = 5.0, 1.3 Hz, 1H), 7.55 (dt, J = 31.8, 7.3 Hz, 3H), 7.09 (dd, J = 4.9, 3.8 Hz, 1H). ¹³C NMR (125 MHz, CDCl₃) δ 142.99, 142.02, 133.91, 133.39, 133.32, 129.32, 127.86, 127.31.



(benzylsulfonyl)benzene (3s)9

White solid (20.1 mg, 43%); mp: 142.7 – 143.9 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.65 – 7.58 (m, 3H), 7.48 – 7.42 (m, 2H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.28 – 7.23 (m, 2H), 7.08 (d, *J* = 7.2 Hz, 2H), 4.31 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 137.79, 133.76, 130.84, 128.92, 128.80, 128.65, 128.61, 128.10, 62.89.



4,4'-sulfonylbis(methylbenzene) (4a)¹⁰

White solid (35.2 mg, 72%); mp: 145.5 - 145.8 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.81 (d, J = 8.3 Hz, 4H), 7.28 (d, J = 8.1 Hz, 4H), 2.39 (s, 6H). ¹³C NMR (125 MHz, CDCl₃) δ 143.96, 139.02, 129.88, 127.57, 21.58.



1-(tert-butyl)-4-tosylbenzene (4b)¹¹

White solid (55.3 mg, 96%); mp: 142.2-143.1 °C ¹H NMR (500 MHz, CDCl₃) δ 7.84 (dd, *J* = 8.4, 5.2 Hz, 4H), 7.49 (d, *J* = 8.6 Hz, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 2.39 (s, 3H), 1.30 (s, 9H). ¹³C NMR (125 MHz, CDCl₃) δ 156.86, 143.96, 138.98, 138.90, 129.88, 127.67, 127.37, 126.28, 35.17, 31.06, 21.59.



1-fluoro-4-tosylbenzene (4c)¹⁰

White solid (30.7 mg, 76%); mp: 106.1-107.2 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.95 (dd, J = 8.9, 5.1 Hz, 2H), 7.81 (d, J = 8.3 Hz, 2H), 7.31 (d, J = 8.4 Hz, 2H), 7.16 (t, J = 8.6 Hz, 2H), 2.40 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 166.35, 164.31, 144.39, 138.49, 138.07(d, J = 2.52 Hz), 130.32(d, J = 10.08 Hz), 130.04, 127.64, 116.54(d, J = 22.68 Hz), 21.61.



1-chloro-4-tosylbenzene (4d)¹⁰

White solid (39.3 mg, 67%); mp: 130.5-131.6 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.86 (d, *J* = 8.7 Hz, 2H), 7.81 (d, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 8.7 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 2.40 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 144.55, 140.52, 139.68, 138.22, 130.08, 129.58, 128.99, 127.72, 21.63.



1-bromo-4-tosylbenzene (4e)¹⁰

White solid (47.5 mg, 76%); mp: 133.7-134.2 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.80 (dd, *J* = 10.9, 8.5 Hz, 4H), 7.63 (d, *J* = 8.5 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 2.40 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 144.57, 141.06, 138.16, 132.56, 130.09, 129.07, 128.25, 127.73, 21.64.



1-chloro-3-tosylbenzene (4f)¹⁰

White solid (41.6 mg, 79%); mp: 128.3-129.4 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.91 (s, 1H), 7.82 (t, *J* = 8.0 Hz, 3H), 7.51 (d, *J* = 8.0 Hz, 1H), 7.43 (t, *J* = 7.9 Hz, 1H), 7.32 (d, *J* = 8.3 Hz, 2H), 2.41 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 144.75, 143.75, 137.89, 135.41, 133.19, 130.60, 130.14, 127.88, 127.56, 125.64, 21.65.



1-methyl-3-tosylbenzene (4g)¹⁰

White solid (42 mg, 85%); mp: 114.2-115.7 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.83 (d, *J* = 8.3 Hz, 2H), 7.73 (d, *J* = 6.5 Hz, 2H), 7.40 – 7.32 (m, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 2.39 (s, 6H). ¹³C NMR (125 MHz, CDCl₃) δ 144.10, 141.76, 139.50, 138.76, 133.86, 129.91, 129.11, 127.80, 127.69, 124.68, 21.60, 21.37.



1,4-dimethyl-2-tosylbenzene (4h)⁵

White solid (51.6 mg, 97%); mp: 104.9-106.7 °C; ¹H NMR (500 MHz, CDCl₃) δ 8.03 (s, 1H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.30 – 7.26 (m, 3H), 7.09 (d, *J* = 7.7 Hz, 1H), 2.41 (s, 3H), 2.40 (s, 3H), 2.37 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 143.87, 138.68, 138.45, 136.40, 134.69, 134.22, 132.58, 129.63, 129.60, 127.68, 21.60, 20.93, 19.73.



1,3,5-trimethyl-2-tosylbenzene (4i)¹⁰

White solid (39.6 mg, 72%); mp: 118.3 - 118.8 °C; ¹H NMR (500 MHz, CDCl₃) δ 7.67 (d, *J* = 8.3 Hz, 2H), 7.25 (s, 1H), 6.93 (s, 2H), 2.59 (s, 6H), 2.39 (s, 3H), 2.29 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 143.40, 143.21, 140.61, 139.97, 134.13, 132.19, 129.49, 126.33, 22.87, 21.57, 21.04.



1,3,5-triisopropyl-2-tosylbenzene (4j)

Colourless liquid (34.6 mg, 48%), R_f: 0.53(PE/EtOAc=9/1); ¹H NMR (500 MHz, CDCl₃) δ 7.64 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 7.16 (s, 2H), 4.19 (hept, *J* = 6.7 Hz, 2H), 2.90 (hept, *J* = 6.9 Hz, 1H), 2.39 (s, 3H), 1.25 (d, *J* = 6.9 Hz, 6H), 1.13 (d, *J* = 6.8 Hz, 12H). ¹³C NMR (125 MHz, CDCl₃) δ 153.68, 151.21, 142.95, 142.38, 132.58, 129.53, 125.69, 124.00, 34.22, 29.40, 24.66, 23.60, 21.55. HRMS (ESI) calcd for C₂₂H₃₀O₂S (M + H)⁺ 358.1967, found 359.2042.

5. ¹H NMR and ¹³C NMR spectra for compounds





71.96 71.95 71.94 71.57 71.57 71.53 71.53 71.53























2.42













8.00 8.00 8.00 8.00 8.00 7.19 8.00 7.10 7.10 7.00 7.10 7.00















-2.41







 $\overbrace{2.37}^{2.41}$









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