Supporting Information

Super electron donor-mediated reductive desulfurization reactions

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1. General comments
Melting points (mp) were determined with a Yazawa micro melting point apparatus and uncorrected. Infrared (IR) data were recorded on SensIR ATR (Attenuated Total Reflectance) FT-IR. Absorbance frequencies are reported in reciprocal centimeters (cm$^{-1}$). NMR data were recorded on a JEOL AL400 spectrometer and a JEOL ECA600 spectrometer. Chemical shifts are expressed in $\delta$ (parts per million, ppm) values and coupling constants are expressed in hertz (Hz). $^1$H NMR spectra were referenced to tetramethylsilane as an internal standard or to a solvent signal (CDCl$_3$: 7.26 ppm, DMSO-$d_6$: 2.49 ppm, Acetone-$d_6$: 2.05 ppm). $^{13}$C NMR spectra were referenced to a solvent signal (CDCl$_3$: 77.0 ppm, DMSO-$d_6$: 39.5 ppm, Acetone-$d_6$: 29.8 ppm). The following abbreviations are used: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = double doublet. Low and high resolution mass spectra (LRMS and HRMS) were obtained from Mass Spectrometry Resource, Graduate School of Pharmaceutical Sciences, Tohoku University, on a JEOL JMS-DX 303 and JMS-700/JMS-T 100 GC spectrometer.

2. Materials
Materials were purchased from Tokyo Kasei Co., Aldrich Inc., and other commercial suppliers and were used as received. Flash column chromatography was performed with Kanto silica gel 60 N (spherical, neutral, 70–230 mesh).

3. Preparation of starting materials
1,3-Bis(N,N-dimethyl-4-aminopyridinium)propane diiodide (3')

Under an Ar atmosphere, 1,3-diiodopropane (30 mmol, 8.9 g) was added to a solution of 4-dimethylaminopyridine (75 mmol, 9.2 g) in acetonitrile (300 mL). The mixture was heated under reflux overnight. After cooling, diethyl ether (100 mL) was added to the mixture. The resulting precipitates were filtered and washed with diethyl ether (30 mL x 3) and recrystallized from dichloromethane/ethanol to give 3' (15 g, 89%) as a white solid; mp 303–308 °C (decomp.); IR (neat, cm$^{-1}$): 1645, 1570, 1539, 1404, 1374, 1236, 1202, 1174, 1036, 946, 830, 743, 738; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ (ppm): 8.29 (4H, d, $J = 7.3$ Hz), 7.04 (4H, d, $J = 7.3$ Hz), 4.27–4.23 (4H, m), 3.18 (12H, s), 2.36–2.32 (2H, m); $^{13}$C{$^1$}H NMR (100 MHz, DMSO-$d_6$) $\delta$ (ppm): 155.9, 141.9, 107.8, 53.8, 39.8, 31.0; LRMS (FAB, m/z): 413 (M–I)$^+$; HRMS (FAB, m/z): Calcd. for C$_{17}$H$_{26}$IN$_4$: 413.1197, found: 413.1212.
1,3-Bis(3-methyl-3H-benzimidazolium)propane diiodide (4')

Under an Ar atmosphere, 1,3-diiodopropane (5.0 mmol, 1.5 g) was added to a solution of 1-methylbenzimidazole (20 mmol, 2.6 g) in acetonitrile (40 mL). The mixture was heated under reflux for 2 days. After cooling, diethyl ether (40 mL) was added to the mixture. The resulting precipitates were filtered and washed with diethyl ether (30 mL × 3) to give 4' (2.2 g, quant.) as a white solid; mp 268–273 °C; IR (neat, cm⁻¹): 1570, 1487, 1351, 1201, 1126, 1008, 857, 762; ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 9.73 (2H, s), 8.09–8.03 (4H, m), 7.73–7.67 (4H, m), 4.68 (4H, t, J = 7.3 Hz), 4.07 (6H, s), 2.59 (2H, quintet, J = 7.3 Hz); ¹³C{¹H} NMR (100 MHz, DMSO-d₆) δ (ppm): 142.8, 131.8, 130.8, 126.6, 126.5, 113.6, 113.5, 43.8, 33.4, 28.1; LRMS (FAB, m/z): 433 (M–I)⁺; HRMS (FAB, m/z): Calcd. for C₁₉H₂₂IN₄: 433.0884, found: 433.0894.

1,3-Bis(5,6-dimethoxy-3-methyl-3H-benzimidazolium)butane diiodide (5')

Under an Ar atmosphere, 1,4-diiodobutane (3.0 mmol, 0.93 g) was added to a solution of 5,6-dimethoxy-1-methylbenzimidazole (5.0 mmol, 0.99 g) in acetonitrile (25 mL). The mixture was heated under reflux for 3 days. After cooling, diethyl ether (40 mL) was added to the mixture. The resulting precipitates were filtered and washed with diethyl ether (30 mL × 3) to give 5' (1.4 g, 76%) as a white solid; mp 293 °C (decomp.); IR (neat, cm⁻¹): 3056, 3023, 2995, 2961, 1560, 1500, 1436, 1262, 1232, 1165, 1016, 852, 824; ¹H NMR (400 MHz, DMSO-d₆) δ (ppm): 9.43 (2H, s), 7.51 (4H, d, J = 7.8 Hz), 4.49 (4H, br.s), 3.99 (6H, s), 3.90 (6H, s), 3.86 (6H, s), 1.91 (4H, s); ¹³C{¹H} NMR (100 MHz, DMSO-d₆) δ (ppm): 149.6, 149.5, 139.8, 125.7, 124.6, 95.3 (2C), 56.6, 56.5, 45.9, 33.4, 25.2; LRMS (FAB, m/z): 567 (M–I)⁺; HRMS (FAB, m/z): Calcd. for C₂₄H₃₂IN₄O₄: 567.1468, found: 567.1463.

1,1′-(1,3-Propanediyl)bis[2,6-dideutero-4(dimethylamino)pyridinium] diiodide (3′-d)

To a solution of 1,3-bis(N,N-dimethyl-4-aminopyridinium)propane diiodide (3′, 4.5 mmol, 2.4 g) in D₂O (10 mL) was added Et₃N (10 mL). The solution was stirred at 80 °C for 5 days. After cooling, the mixture was evaporated. Recrystallization from dichloromethane/ethanol to give 3′-d (2.2 g, 90%, 99%D) as colorless prisms; mp 270–280 °C (decomp.); IR (neat, cm⁻¹): 3052, 2925, 1627, 1560, 1425, 1366, 1220, 1184, 1044,
Representative procedure for the synthesis of benzophenones

Under an Ar atmosphere, the solution of 4-iodoanisole (4.8 mmol, 1.1 g) in anhydrous THF (10 mL) was cooled to −20 °C and isopropylmagnesium chloride (2.0 M in Et₂O, 5.2 mmol, 2.6 mL) was added slowly. The reaction mixture was stirred at this temperature for 30 min. The resulting solution was then transferred dropwise by cannula into a second flask, which contained a solution of benzoyl cyanide (4.0 mmol, 0.52 g) and [Fe(acac)₃] (0.20 mmol, 72 mg) in anhydrous THF (20 mL) stirred at −10 °C. At the end of the addition, the reaction mixture was quenched with aq. NH₄Cl (10 mL), diluted with water (25 mL), and extracted with Et₂O (30 mL × 3). The combined organic layer was washed with aq. NaHCO₃ (15 mL), brine (15 mL × 2), and then dried over Na₂SO₄. The solvent was removed under reduced pressure and the residue was purified by SiO₂ column chromatography (eluent: hexane/AcOEt = 100/1) to give 4-methoxybenzophenone (0.64 g, 75%) as colorless prisms.

4-Methoxybenzophenone

Recrystallized from AcOEt/hexane, colorless prisms, mp 54−58 °C; IR (neat, cm⁻¹): 3016, 2971, 1740, 1647, 1595, 1506, 1257, 1170, 841; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.83 (2H, d, J = 8.9 Hz), 7.76 (2H, d, J = 6.8 Hz), 7.60−7.55 (1H, m), 7.47 (2H, t, J = 7.5 Hz) , 6.97 (2H, d, J = 8.9 Hz), 3.89 (3H, s); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 195.5, 163.2, 138.3, 132.5, 131.9, 130.2, 129.7, 128.2, 113.5, 55.5; LRMS (EI, m/z): 212 (M⁺); HRMS (EI, m/z): Calcd. for C₁₄H₁₂O₂ 212.0837, found: 212.0837.

Ethyl 4-benzoylbenzoate

Colorless oil; IR (neat, cm⁻¹): 2970, 1718, 1654, 1560, 1507, 1267, 1102, 1019; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 8.15 (2H, d, J = 8.2 Hz), 7.84 (2H, d, J = 8.2 Hz), 7.81 (2H, d, J = 6.8 Hz), 7.61 (1H, t, J = 7.5 Hz), 7.50 (2H, t, J = 7.5 Hz), 4.43 (2H, q, J = 7.5 Hz), 1.42 (3H, t, J = 7.5 Hz); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 195.9, 165.7, 141.2, 136.9, 133.5, 132.9, 130.0, 129.7, 129.4, 128.4, 61.4, 14.2; LRMS (EI, m/z): 254 (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₄O₂ 254.0943, found: 254.0934.
4-Cyanobenzophenone

Recrystallized from AcOEt/hexane, colorless needles, mp 114–116 °C; IR (neat, cm\(^{-1}\)): 2981, 2228, 1734, 1653, 1560, 1507, 1457, 1420, 857; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.88 (2H, d, \(J = 8.2\) Hz), 7.80–7.78 (4H, m), 7.65 (1H, t, \(J = 7.5\) Hz), 7.52 (2H, t, \(J = 8.0\) Hz); \(^1^C\){\(^1\)H} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 195.0, 141.2, 136.3, 133.3, 132.2, 130.2, 130.1, 128.6, 118.0, 115.7; LRMS (EI, \(m/z\)): 207 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_9\)NO 207.0684, found: 207.0686.

Representative procedure for the preparation of 1,3-dithianes

**Method A**

\[\text{O} \quad \text{BF}_3 \cdot \text{OEt}_2 \quad \text{CHCl}_3, \text{Na}_2\text{SO}_4 \quad 0 \degree \text{C} \to \text{rt overnight} \]

Under an Ar atmosphere, 1,3-propanedithiol (10.5 mmol, 1.1 g) was added to a mixture of benzophenone (10.0 mmol, 1.8 g) and Na\(_2\)SO\(_4\) (1.0 g) in CHCl\(_3\) (15 mL) at room temperature. After distilled BF\(_3\)•OEt\(_2\) (11.0 mmol, 1.4 mL) was added dropwise to the mixture at 0 °C, the whole reaction mixture was gradually warmed up to room temperature and then stirred overnight. After the addition of saturated aqueous NaHCO\(_3\) (50 mL), the mixture was extracted with CHCl\(_3\) (50 mL × 2) and the organic layer was washed with NaHCO\(_3\) (50 mL), H\(_2\)O (50 mL), and brine (50 mL). The organic layer was dried over MgSO\(_4\) and concentrated under reduced pressure. The residue was purified by SiO\(_2\) column chromatography (eluent: hexane/AcOEt = 5/1) followed by recrystallization (AcOEt/hexane) to give 2,2-diphenyl-1,3-dithiane 1\(a\) (1.6 g, 60%) as colorless plates.

**Method B**

\[\text{OH} \quad \text{TFAA (1.5 equiv.)} \]

Under an Ar atmosphere, \(n\)-BuLi (1.6 M in hexane, 5.5 mmol, 3.5 mL) was added to a solution of 2-phenyl-1,3-dithiane (5.5 mmol, 1.0 g) in THF (40 mL) at −78 °C, and the mixture was warmed up to −20 °C. The reaction mixture was stirred at −20 °C for 1 h. Benzaldehyde (5.5 mmol, 0.58 g) was added to the mixture at −78 °C, and the reaction mixture was stirred at room temperature overnight. The reaction mixture was quenched by saturated aqueous NH\(_4\)Cl (50 mL). The mixture was extracted with CH\(_2\)Cl\(_2\) (50 mL × 2) and the organic layer was washed with H\(_2\)O (50 mL), and brine (50 mL). The organic layer was dried over MgSO\(_4\) and
concentrated under reduced pressure. The residue was purified by SiO$_2$ column chromatography.

A solution of trifluoroacetic anhydride (5.3 mmol, 1.1 g) in CH$_2$Cl$_2$ (10 mL) was added to a stirring solution of DMSO (7.7 mmol, 0.60 g) in CH$_2$Cl$_2$ (30 mL) at −78 °C via cannula. After stirring at −78 °C for 30 min, a solution of the crude (3.5 mmol, 0.99 g) in CH$_2$Cl$_2$ (6 mL) was added dropwise via cannula, and stirring continued at −78 °C for 1 h. Triethylamine (10.5 mmol, 1.5 mL) was added and the solution was warmed up to room temperature and stirred overnight. After the addition of 5% aqueous HCl (30 mL), the organic layer was washed with saturated aqueous NaHCO$_3$ (50 mL × 2). The organic layer was dried over MgSO$_4$ and concentrated under reduced pressure. The residue was purified by SiO$_2$ column chromatography to give phenyl(2-phenyl-1,3-dithian-2-yl)methanone 1s (0.45 g, 45%) as colorless plates.

**Method C**

\[
\begin{align*}
\text{PhMgCl (1.5 equiv.)} & \quad \text{THF, rt, overnight} \\
\text{PhH-OH} & \quad \text{PhH-SH} \\
\text{PhH-CO} & \quad \text{PhH-SH}
\end{align*}
\]

A solution of 2-napthaldehyde (10.0 mmol, 1.6 g) in THF (12.5 mL) was slowly added to phenyl magnesium chloride (1.2 M in THF, 15.0 mmol, 12.5 mL) at 0 °C, and the mixture was gradually warmed up to room temperature and then stirred overnight. After the addition of saturated aqueous NH$_4$Cl (50 mL), the mixture was extracted with AcOEt (50 mL × 3) and the organic layer was washed with brine (50 mL). The combined organic layer was dried over MgSO$_4$. After filtration, the filtrate was concentrated under reduced pressure and dried in vacuo. The crude was directly used for next step without further purification.

Diethyl azocarboxylate (2.2 M in toluene, 13.0 mmol, 5.3 mL) was slowly added to a solution of (n-Bu)$_3$P (13.0 mmol) in toluene (30 mL) at −10 °C. After the mixture was stirred for 1 h, a solution of the crude and benzenethiol (13.0 mmol, 1.3 mL) in toluene (6 mL) was slowly added to the mixture at −10 °C. After being stirred at −10 °C for overnight, the reaction was quenched by adding 1 M aqueous NaOH (50 mL). The mixture was extracted with Et$_2$O (50 mL × 3) and the organic layer was washed with brine (50 mL). The combined organic layer was dried over MgSO$_4$ and the solvent was removed under reduced pressure after filtration. The residue was purified by SiO$_2$ column chromatography (hexane/AcOEt = 40/1) followed by recrystallization (MeOH) to give (naphthalen-2-yl)(phenyl)methyl)(phenyl)sulfane 9aa (1.7 g, 52%) as colorless prisms.

**Method D**

\[
\begin{align*}
1) (n-Bu)_2Li & \quad \text{THF, 0 °C, 2 h} \\
2) \text{diethyldisulphide (1.1 equiv.)}
\end{align*}
\]

Diphenylmethane (18.0 mmol, 3.0 g) was dissolved in THF (15 mL) and cooled to 0 °C. n-BuLi (1.6 M in hexane, 19.6 mmol, 12.3 mL) was then added and the mixture stirred for 2 h at 0 °C. Diethyldisulphide (20.0
mmol, 2.4 g) was then added and the mixture warmed up to room temperature, treated with H₂O, extracted with Et₂O (50 mL × 3) and the organic layer was washed with brine (50 mL). The combined organic layer was dried over MgSO₄ and concentrated under reduced pressure. The residue was purified by SiO₂ column chromatography (eluent: hexane/AcOEt = 40/1) to give benzhydryl(ethyl)sulfide 9da as colorless oil.

2,2-Diphenyl-1,3-dithiane (1a)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless plates, mp 112–113 °C; IR (neat, cm⁻¹): 3032, 2892, 1507, 1444, 1281, 1035, 930, 864, 737; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.70 (4H, d, J = 7.9 Hz), 7.34 (4H, t, J = 7.9 Hz), 7.28–7.26 (2H, m), 2.80–2.78 (4H, m), 2.03–1.99 (2H, m); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 142.5, 129.3, 128.4, 127.6, 62.8, 29.4, 24.5; LRMS (EI, m/z): 272 (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₆S₂ 272.0693, found: 272.0704.

2-Phenyl-2-(p-tolyl)-1,3-dithiane (1b)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless prisms, mp 115–116 °C; IR (neat, cm⁻¹): 2971, 1740, 1560, 1507, 1420, 1364, 1217, 1206, 742; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.70 (4H, d, J = 7.5 Hz), 7.56 (2H, d, J = 7.5 Hz), 7.34 (2H, t, J = 7.5 Hz), 7.27–7.25 (1H, m), 7.14 (2H, d, J = 7.5 Hz), 2.79–2.77 (4H, m), 2.34 (3H, s), 2.01–1.98 (2H, m); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 142.7, 139.6, 137.3, 129.3, 129.2, 129.1, 128.4, 127.5, 62.6, 29.4, 24.5, 21.0; LRMS (EI, m/z): 286 (M⁺); HRMS (EI, m/z): Calcd. for C₁₇H₁₈S₂ 286.0850, found: 286.0835.

2-Phenyl-2-(m-tolyl)-1,3-dithiane (1c)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless prisms, mp 82–83 °C; IR (neat, cm⁻¹): 2971, 1740, 1560, 1507, 1366, 1217, 770, 738; ¹H NMR (600 MHz, DMSO-d₆) δ (ppm): 7.56 (2H, d, J = 7.5 Hz), 7.42 (1H, s), 7.37 (1H, d, J = 7.5 Hz), 7.32 (2H, t, J = 7.5 Hz), 7.29–7.23 (2H, m), 7.10 (1H, d, J = 7.5 Hz), 2.73–2.71 (4H, m), 2.31 (3H, s).
2.27 (3H, s), 1.90−1.86 (2H, m); $^{13}$C{$^1$H} NMR (150 MHz, DMSO-$d_6$) δ (ppm): 142.4, 142.3, 137.7, 129.2, 128.7, 128.6, 128.4, 128.3, 127.6, 125.9, 62.1, 28.8, 23.9, 21.3; LRMS (EI, $m/z$): 286 (M$^+$); HRMS (EI, $m/z$): Calcd. for C$_{17}$H$_{18}$S$_2$ 286.0850, found: 286.0849.

2-Phenyl-2-(o-tolyl)-1,3-dithiane (1d)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless prisms, mp 92−94 °C; IR (neat, cm$^{-1}$): 2971, 1740, 1684, 1560, 1507, 1366, 1217, 754, 735; $^1$H NMR (600 MHz, CDCl$_3$/TMS) δ (ppm): 8.11 (1H, d, $J$ = 7.5 Hz), 7.52 (2H, d, $J$ = 7.5 Hz), 7.30−7.21 (5H, m), 7.13 (1H, d, $J$ = 7.5 Hz), 2.97−2.93 (2H, m), 2.81 (2H, dt, $J$ = 14.4, 4.5 Hz), 2.08−1.99 (5H, m); $^{13}$C{$^1$H} NMR (150 MHz, CDCl$_3$/TMS) δ (ppm): 143.6, 140.2, 138.0, 133.3, 130.8, 128.3, 128.0, 127.6, 127.5, 125.0, 61.2, 29.6, 24.3, 22.8; LRMS (EI, $m/z$): 286 (M$^+$); HRMS (EI, $m/z$): Calcd. for C$_{17}$H$_{18}$S$_2$ 286.0850, found: 286.0843.

2-(4-Methoxyphenyl)-2-phenyl-1,3-dithiane (1e)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless needles, mp 101−102 °C; IR (neat, cm$^{-1}$): 2954, 2911, 1604, 1506, 1445, 1249, 1176, 1027, 849, 811, 740; $^1$H NMR (400 MHz, CDCl$_3$/TMS) δ (ppm): 7.72 (2H, d, $J$ = 7.8 Hz), 7.58 (2H, d, $J$ = 8.8 Hz), 7.35 (2H, t, $J$ = 7.8 Hz), 7.28−7.25 (1H, m), 6.85 (2H, d, $J$ = 8.8 Hz), 3.80 (3H, s), 2.79−2.76 (4H, m), 2.03−1.97 (2H, m); $^{13}$C{$^1$H} NMR (100 MHz, CDCl$_3$/TMS) δ (ppm): 158.8, 142.7, 134.6, 130.6, 129.4, 128.4, 127.5, 113.6, 62.4, 55.3, 29.5, 24.5; LRMS (EI, $m/z$): 302 (M$^+$); HRMS (EI, $m/z$): Calcd. for C$_{17}$H$_{18}$OS 302.0799, found: 302.0819.

2-(4-Dimethylaminophenyl)-2-phenyl-1,3-dithiane (1f)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless plates, mp 141−142 °C; IR (neat, cm$^{-1}$): 2971, 1740, 1715, 1653, 1559, 1507, 1363, 1437, 1355, 792, 764, 728; $^1$H NMR (600 MHz, CDCl$_3$/TMS) δ (ppm): 7.74 (2H, d, $J$ = 7.5 Hz), 7.47 (2H, d, $J$ = 8.2 Hz), 7.33 (2H, t, $J$ = 7.9 Hz), 7.26−7.24 (1H, m), 6.65 (2H, d, $J$ = 8.9 Hz), 2.95 (6H,
2-(4-Fluorophenyl)-2-phenyl-1,3-dithiane (1g)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless prisms, mp 102–104 °C; IR (neat, cm⁻¹): 2971, 1740, 1560, 1507, 1366, 1217, 1103, 1017, 815, 783, 752; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.67 (2H, d, J = 8.1 Hz), 7.40–7.38 (2H, m), 7.36–7.34 (2H, m), 7.29–7.27 (1H, m), 2.77–2.75 (4H, m), 1.97–1.94 (2H, m); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 143.4, 142.6, 133.8, 132.0, 129.8, 129.3, 129.2, 128.6, 128.5, 62.6, 29.9, 25.0; LRMS (EI, m/z): 306 (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₅FClS₂ 306.0304, found: 306.0316.

2-(4-Chlorophenyl)-2-phenyl-1,3-dithiane (1h)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless prisms, mp 99–100 °C; IR (neat, cm⁻¹): 2971, 1740, 1560, 1490, 1366, 1217, 1093, 1017, 815, 783, 752; ¹H NMR (600 MHz, Acetone-d₆) δ (ppm): 7.67 (2H, d, J = 8.8 Hz), 7.61 (2H, d, J = 8.1 Hz), 7.40–7.38 (2H, m), 7.36–7.34 (2H, m), 7.29–7.27 (1H, m), 2.77–2.75 (4H, m), 1.97–1.94 (2H, m); ¹³C{¹H} NMR (150 MHz, Acetone-d₆) δ (ppm): 143.4, 142.6, 133.8, 132.0, 129.8, 129.3, 129.2, 128.6, 62.6, 29.9, 25.0; LRMS (EI, m/z): 306 (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₅ClS₂ 306.0304, found: 306.0316.

2-(4-Bromophenyl)-2-phenyl-1,3-dithiane (1i)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless prisms, mp 117 °C; IR (neat, cm⁻¹): 2971, 1740, 1653, 1560, 1507, 1366, 1217, 1012, 892, 784; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.65 (2H, d, J = 7.5 Hz), 7.60 (2H, d, J = 8.2 Hz), 7.46 (2H, d, J = 8.9 Hz), 7.34 (2H, t, J = 7.5 Hz), 7.29–7.25 (1H, m), 2.78–2.76 (4H, m),
2.02–1.99 (2H, m); \(^{13}C\{^1H\} NMR (150 MHz, CDCl\textsubscript{3}/TMS) \delta (ppm): 142.1, 141.7, 131.5, 131.3, 129.1, 128.5, 127.8, 121.8, 62.2, 29.3, 24.3; LRMS (EI, \(m/z\)): 350 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{16}\)H\(_{15}\)BrS\(_2\) 349.9799, found: 349.9782.

**Ethyl 4-(2-phenyl-1,3-dithian-2-yl)benzoate (1j)**

![Ethyl 4-(2-phenyl-1,3-dithian-2-yl)benzoate (1j)]

Prepared according to the Method A.

Recrystallized from AcOEt/hexane, colorless prisms, mp 98–99 °C; IR (neat, cm\(^{-1}\)): 2971, 1734, 1719, 1653, 1560, 1507, 1457, 1363, 1275, 1217, 1108, 758; \(^1H\) NMR (600 MHz, Acetone-\(d_6\)) \(\delta (ppm): 8.04–8.02 (2H, m), 7.86 (2H, d, \(J = 8.9\) Hz), 7.62 (2H, d, \(J = 8.3\) Hz), 7.39–7.37 (2H, m), 7.32–7.30 (1H, m), 4.36 (2H, q, \(J = 7.2\) Hz), 2.82–2.80 (4H, m), 2.03–1.96 (2H, m), 1.37 (3H, t, \(J = 7.2\) Hz); \(^{13}C\{^1H\} NMR (150 MHz, Acetone–\(d_6\)) \(\delta (ppm): 166.3, 148.6, 143.4, 130.6, 130.5, 130.3, 129.8, 129.3, 128.7, 62.9, 61.5, 25.0, 14.6; LRMS (EI, \(m/z\)): 340 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{19}\)H\(_{20}\)O\(_2\)S\(_2\) 344.0905, found: 344.0895.

2-(4-Cyanophenyl)-2-phenyl-1,3-dithiane (1k)

![2-(4-Cyanophenyl)-2-phenyl-1,3-dithiane (1k)]

Prepared according to the Method A.

Recrystallized from AcOEt/hexane, colorless prisms, mp 111–113 °C; IR (neat, cm\(^{-1}\)): 2945, 2905, 2895, 2228, 1596, 1496, 1447, 856, 744; \(^1H\) NMR (600 MHz, CDCl\textsubscript{3}/TMS) \(\delta (ppm): 7.85 (2H, d, \(J = 8.3\) Hz), 7.64 (4H, d, \(J = 8.8\) Hz), 7.36 (2H, t, \(J = 7.9\) Hz), 7.31–7.29 (1H, m), 2.83–2.73 (4H, m), 2.04–2.01 (2H, m); \(^{13}C\{^1H\} NMR (150 MHz, CDCl\textsubscript{3}/TMS) \(\delta (ppm): 166.3, 148.6, 143.4, 130.6, 130.5, 130.3, 129.8, 129.3, 128.7, 62.9, 61.5, 29.9, 25.0, 14.6; LRMS (EI, \(m/z\)): 297 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{17}\)H\(_{15}\)NS\(_2\) 297.0646, found: 297.0632.

2-(4-Nitrophenyl)-2-phenyl-1,3-dithiane (1l)

![2-(4-Nitrophenyl)-2-phenyl-1,3-dithiane (1l)]

Prepared according to the Method A.

Recrystallized from AcOEt/hexane, colorless needles, mp 123–124 °C; IR (neat, cm\(^{-1}\)): 2961, 2911, 1602, 1511, 1485, 1347, 843, 740; \(^1H\) NMR (600 MHz, CDCl\textsubscript{3}/TMS) \(\delta (ppm): 8.19–8.18 (2H, m), 7.91 (2H, d, \(J = 8.2\) Hz), 7.65 (2H, d, \(J = 8.2\) Hz), 7.37 (2H, t, \(J = 7.5\) Hz), 7.32–7.30 (1H, m), 2.85–2.75 (4H, m), 2.06–2.02 (2H, m); \(^{13}C\{^1H\} NMR (150 MHz, CDCl\textsubscript{3}/TMS) \(\delta (ppm): 150.2, 147.1, 141.4, 130.5, 129.0, 128.8, 128.2,
123.5, 62.0, 29.3, 24.1; LRMS (EI, m/z): 317 (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₅NO₂S₂ 317.0544, found: 317.0543.

**2,2-Bis(4-fluorophenyl)-1,3-dithiane (1m)**

![Chemical Structure](image)

Prepared according to the Method A.

Recrystallized from AcOEt/hexane, colorless plates, mp 91–93 °C; IR (neat, cm⁻¹): 2971, 1793, 1749, 1734, 1684, 1653, 1560, 1507, 1457, 1420, 1374, 1340, 1217; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.67–7.65 (4H, m), 7.04–7.01 (4H, m), 2.78–2.76 (4H, m), 2.03–1.99 (2H, m); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 162.0 (d, J_FC = 247.8 Hz), 138.2, 131.2 (d, J_FC = 7.2 Hz), 115.3 (d, J_FC = 21.5 Hz), 61.5, 29.4, 24.3; LRMS (EI, m/z): 308 (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₄F₂S₂ 308.0505, found: 308.0515.

**2,2-Bis(4-chlorophenyl)-1,3-dithiane (1n)**

![Chemical Structure](image)

Prepared according to the Method A.

Recrystallized from AcOEt/hexane, colorless prisms, mp 103–105 °C; IR (neat, cm⁻¹): 2929, 2894, 1585, 1487, 1475, 1398, 1273, 1241, 1092, 1014, 897, 852, 805, 787; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.61 (4H, d, J = 8.2 Hz), 7.32–7.31 (4H, m), 2.78–2.76 (4H, m), 2.03–1.99 (2H, m); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 140.8, 133.8, 130.8, 128.6, 61.5, 29.3, 24.2; LRMS (EI, m/z): 308 (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₄Cl₂S₂ 339.9914, found: 339.9906.

**2,2-Bis(4-bromophenyl)-1,3-dithiane (1o)**

![Chemical Structure](image)

Prepared according to the Method A.

Recrystallized from AcOEt/hexane, colorless prisms, mp 135–137 °C; IR (neat, cm⁻¹): 2929, 2893, 1484, 1391, 1074, 1008, 800, 783; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.55 (4H, d, J = 8.5 Hz), 7.47 (4H, d, J = 8.5 Hz), 2.77–2.76 (4H, m), 2.03–1.99 (2H, m); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 141.3, 131.6, 131.1, 122.1, 61.7, 29.3, 24.2; LRMS (EI, m/z): 427 (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₄Br₂S₂ 427.8904,
found: 427.8899.

2,2-**Bis**(4-methylphenyl)-1,3-dithiane (1p)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless prisms, mp 136–137 °C; IR (neat, cm\(^{-1}\)): 2971, 1793, 1734, 1684, 1560, 1507, 1457, 1374, 1340, 1217; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.56 (4H, d, \(J = 7.9\) Hz), 7.14 (4H, d, \(J = 7.9\) Hz), 2.78–2.76 (4H, m), 2.34 (6H, s), 2.00–1.97 (2H, m); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 139.7, 137.2, 129.2, 129.1, 62.4, 29.4, 24.5, 21.0; LRMS (EI, \(m/z\)): 300 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{18}\)H\(_{20}\)S\(_2\) 300.1006, found: 300.1035.

2,2-**Bis**(4-methoxyphenyl)-1,3-dithiane (1q)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless needles, mp 96–97 °C; IR (neat, cm\(^{-1}\)): 2905, 2835, 1606, 1503, 1302, 1272, 1248, 1173, 1114, 1026, 870, 820, 793; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.60 (4H, d, \(J = 9.2\) Hz), 6.86 (4H, d, \(J = 9.2\) Hz), 3.81 (6H, s), 2.78–2.76 (4H, m), 2.01–1.97 (2H, m); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 158.8, 134.8, 130.6, 113.6, 62.0, 55.3, 29.5, 24.5; LRMS (EI, \(m/z\)): 332 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{18}\)H\(_{20}\)O\(_2\)S\(_2\) 332.0905, found: 332.0897.

2,2-**Bis**(4-dimethylaminophenyl)-1,3-dithiane (1r)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless needles, mp 186–188 °C; IR (neat, cm\(^{-1}\)): 2954, 2909, 2808, 1607, 1516, 1361, 1233, 1186, 1166, 1121, 949, 808, 783; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.53 (4H, d, \(J = 8.2\) Hz), 6.67 (4H, d, \(J = 8.2\) Hz), 2.95 (12H, s), 2.78–2.76 (4H, m), 1.98–1.94 (2H, m); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 149.5, 130.5, 130.2, 111.9, 62.5, 40.4, 29.7, 24.7; LRMS (EI, \(m/z\)): 358 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{20}\)H\(_{26}\)N\(_2\)S\(_2\) 358.1537, found: 358.1540.
**2,2-Bis(2,4-dimethylphenyl)-1,3-dithiane (1s)**

Prepared according to the Method A.

Recrystallized from AcOEt/hexane, colorless needles, mp 112–115 °C; IR (neat, cm⁻¹): 2954, 2906, 1610, 1486, 1448, 1411, 1278, 1170, 1035, 874, 833, 797, 781; \(^1\)H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 8.04 (2H, d, \(J = 8.2\) Hz), 7.04 (2H, d, \(J = 8.2\) Hz), 6.83 (2H, s), 2.92 (4H, s), 2.29 (6H, s), 2.05 (2H, quintet, \(J = 6.2\) Hz), 1.77 (6H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 138.6, 136.9, 136.5, 133.6, 129.0, 126.0, 61.1, 29.2, 24.3, 21.9, 20.7; LRMS (EI, \(m/z\)): 328 (M⁺); HRMS (EI, \(m/z\)): Calcd. for C\(_{20}\)H\(_{24}\)S\(_2\) 328.1319, found: 328.1327.

**Phenyl(2-phenyl-1,3-dithian-2-yl)methanone (1t)**

Prepared according to the Method B.

Recrystallized from MeOH, colorless prisms, mp 98–99 °C; IR (neat, cm⁻¹): 3081, 2898, 1670, 1593, 1576, 1444, 1218, 1018, 768; \(^1\)H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.68 (2H, d, \(J = 8.2\) Hz), 7.58–7.56 (2H, m), 7.36–7.33 (1H, m), 3.29–3.24 (2H, m), 2.77 (2H, dt, \(J = 14.7, 3.6\) Hz), 2.15–2.10 (1H, m), 1.98–1.90 (1H, m); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 169.6, 126.6, 123.0, 121.0, 120.0, 118.6, 118.4, 117.5, 117.3, 66.1, 38.7, 34.6; LRMS (EI, \(m/z\)): 300 (M⁺); HRMS (EI, \(m/z\)): Calcd. for C\(_{17}\)H\(_{16}\)O\(_2\)S 300.0643, found: 300.0665.

**2,2-Dimethyl-1-(2-phenyl-1,3-dithian-2-yl)propan-1-one (1u)**

Prepared according to the Method B.

Recrystallized from AcOEt/hexane, colorless needles, mp 111–113 °C; IR (neat, cm⁻¹): 2991, 2933, 2907, 1681, 1446, 1273, 1067; \(^1\)H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.46 (2H, d, \(J = 7.5\) Hz), 7.38 (2H, t, \(J = 7.5\) Hz), 7.35–7.33 (1H, m), 3.21–3.16 (2H, m), 2.75 (2H, dt, \(J = 13.8, 3.6\) Hz), 2.11–2.06 (1H, m), 1.91–1.83 (1H, m), 1.05 (9H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 206.0, 138.0, 128.9, 128.6, 126.8, 64.6, 45.6, 29.9, 29.3, 23.9; LRMS (EI, \(m/z\)): 280 (M⁺); HRMS (EI, \(m/z\)): Calcd. for C\(_{15}\)H\(_{20}\)O\(_2\)S 280.0956, found: 280.0947.
2,2-Diphenyl-1,3-dithiolane (6a)

Prepared according to the Method A using 1,2-ethanediethiol instead of 1,3-propanediethiol.
Recrystallized from AcOEt/hexane, colorless prisms, mp 105–106 °C; IR (neat, cm\(^{-1}\)): 3015, 2924, 1577, 1482, 1442, 1414, 1315, 1275, 1077, 951, 756; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.60 (4H, d, \(J = 7.5\) Hz), 7.28 (4H, t, \(J = 7.5\) Hz), 7.22 (2H, t, \(J = 7.5\) Hz), 3.41 (4H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 144.6, 128.2, 127.9, 127.2, 76.8, 40.1; LRMS (EI, \(m/z\)): 258 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{15}\)H\(_{14}\)S\(_2\) 258.0537, found: 258.0542.

2-Benzyl-2-phenyl-1,3-dithiane (7a)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless needles, mp 120–121 °C; IR (neat, cm\(^{-1}\)): 3056, 2952, 2903, 1309, 1280, 1246, 1150, 1077, 963, 784, 750; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.72 (2H, d, \(J = 6.9\) Hz), 7.31 (2H, t, \(J = 7.6\) Hz), 7.26–7.23 (1H, m), 7.16 (1H, t, \(J = 7.2\) Hz), 7.09 (2H, t, \(J = 7.5\) Hz), 6.73 (2H, d, \(J = 7.6\) Hz), 3.27 (2H, s), 2.71–2.63 (4H, m), 1.95–1.91 (2H, m); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 140.6, 134.4, 130.9, 129.5, 128.3, 127.3, 127.0, 126.9, 59.7, 51.6, 27.5, 25.0; LRMS (EI, \(m/z\)): 286 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{17}\)H\(_{18}\)S\(_2\) 286.0850, found: 286.0865.

2-Methyl-2-phenyl-1,3-dithiane (7b)

Prepared according to the Method A.
Colorless oil; IR (neat, cm\(^{-1}\)): 2906, 1487, 1443, 1276, 1065, 1049, 907, 758; \(^1\)H NMR (400 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.94 (2H, d, \(J = 7.8\) Hz), 7.37 (2H, t, \(J = 7.8\) Hz), 7.27–7.25 (1H, m), 2.76–2.69 (4H, m), 1.97–1.93 (2H, m), 1.80 (3H, s); \(^{13}\)C\{\(^1\)H\} NMR (100 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 143.8, 128.5, 127.8, 127.0, 54.0, 32.8, 28.1, 24.7; LRMS (EI, \(m/z\)): 210 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{11}\)H\(_{14}\)S\(_2\) 210.0537, found: 210.0542.
2-Butyl-2-phenyl-1,3-dithiane (7c)

Prepared according to the Method A.

Colorless oil; IR (neat, cm\(^{-1}\)): 2954, 2933, 2871, 1487, 1442, 1423, 1277, 1173, 1044, 909, 866, 800, 752; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.91 (2H, dd, \(J = 8.3, 1.4\) Hz), 7.38 (2H, t, \(J = 7.8\) Hz), 7.27–7.24 (1H, m), 2.73–2.64 (4H, m), 2.00–1.97 (2H, m), 1.96–1.91 (2H, m), 1.25–1.17 (4H, m), 0.80 (3H, t, \(J = 7.2\) Hz); \(^13\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 142.0, 128.8, 128.4, 126.7, 59.1, 45.1, 27.6, 25.9, 25.3, 22.7, 13.8; LRMS (EI, \(m/z\)): 252 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{20}\)S\(_2\) 252.1006, found: 242.1009.

2-(\(\text{tert}-\)Butyl)-2-phenyl-1,3-dithiane (7d)

To a solution of 2,2-dimethylpropiophenone (2.0 mmol, 0.32 g) and 1,3-propanedithiol (2.0 mmol, 0.20 mL) in anhydrous dichloromethane (2 mL) was added BF\(_3\)•OEt\(_2\) (1.5 mmol, 0.19 mL) at 0 °C. After being stirred for 2 days at room temperature the reaction mixture was quenched by saturated aqueous NaHCO\(_3\) (10 mL) and extracted with CH\(_2\)Cl\(_2\) (50 mL \(\times\) 3). The combined organic layer was washed with brine (50 mL), dried with MgSO\(_4\) and concentrated under reduced pressure. The residue was purified by SiO\(_2\) column chromatography (eluent: hexane/AcOEt = 5/1) to give 2-(\(\text{tert}-\)butyl)-2-phenyl-1,3-dithiane 7d (0.12 g, 25%) as a white solid. Recrystallized from AcOEt/hexane, colorless prisms, mp 103–107 °C; IR (neat, cm\(^{-1}\)): 2977, 2911, 2869, 1441, 1413, 1363, 1278, 1209, 1030, 899, 854, 799, 746, 708; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.99 (2H, d, \(J = 8.3\) Hz), 7.38 (2H, t, \(J = 8.3\) Hz), 7.26–7.24 (1H, m), 2.63 (2H, dt, \(J = 13.7, 3.5\) Hz), 2.58–2.53 (2H, m), 1.83–1.79 (2H, m), 1.10 (9H, s); \(^13\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 138.4, 132.0, 127.7, 126.5, 70.1, 40.1, 27.9, 26.5, 25.0; LRMS (EI, \(m/z\)): 252 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{20}\)S\(_2\) 252.1006, found: 252.1030.

2-(Naphthalen-2-yl)-1,3-dithiane (7e)

Prepared according to the Method A.
Recrystallized from AcOEt/hexane, colorless prisms, mp 112–113 °C; IR (neat, cm⁻¹): 2893, 1599, 1506, 1421, 1279, 1186, 816, 786, 776; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.94 (1H, s), 7.81–7.78 (3H, m), 7.57–7.56 (1H, m), 7.46–7.43 (2H, m), 5.31 (1H, s), 3.09–3.05 (2H, t, J = 13.4 Hz), 2.92 (2H, dt, J = 14.4, 3.0 Hz), 2.17–2.14 (1H, m), 1.99–1.91 (1H, m); ¹³C {¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 136.4, 133.24, 133.17, 128.4, 128.0, 127.6, 126.8, 126.22, 126.21, 125.6, 51.5, 32.1, 25.1; LRMS (EI, m/z): 246 (M⁺); HRMS (EI, m/z): Calcd. for C₁₄H₁₄S₂ 246.0537, found: 246.0555.

(Naphthalen-2-yl(phenyl)methyl)(phenyl)sulfane (9aa)

Prepared according to the Method C.

Recrystallized from MeOH, colorless prisms, mp 84–85 °C; IR (neat, cm⁻¹): 3042, 1596, 1507, 1453, 1440, 903, 872, 807, 747; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.83 (1H, s), 7.78–7.75 (3H, m), 7.56 (1H, dd, J = 8.4, 1.8 Hz), 7.45–7.43 (4H, m), 7.30 (2H, t, J = 7.8 Hz), 7.26–7.21 (3H, m), 7.16–7.14 (2H, m), 7.12–7.10 (1H, m), 5.69 (s, 1H); ¹³C {¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 140.9, 138.4, 136.1, 133.3, 132.6, 130.6, 128.8, 128.6, 128.5, 128.3, 128.0, 127.6, 127.3, 127.1, 126.62, 126.60, 126.1, 126.0, 57.7; LRMS (EI, m/z): 326 (M⁺); HRMS (EI, m/z): Calcd. for C₂₃H₁₈S 326.1129, found: 326.1160.

(4-Chlorophenyl)(naphthalen-2-yl(phenyl)methyl)sulfane (9ab)

Prepared according to the Method C.

Recrystallized from MeOH, colorless prisms, mp 110–112 °C; IR (neat, cm⁻¹): 3059, 1596, 1507, 1475, 1456, 1388, 1094, 1013, 821, 802; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.81–7.76 (4H, m), 7.55 (1H, d, J = 8.3 Hz), 7.47–7.42 (4H, m), 7.30 (2H, t, J = 7.3 Hz), 7.25–7.22 (1H, m), 7.17 (2H, d, J = 8.3 Hz), 7.11 (2H, d, J = 8.3 Hz), 5.65 (1H, s); ¹³C {¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 140.5, 137.9, 134.4, 133.2, 132.8, 132.6, 132.1, 128.9, 128.6, 128.5, 128.4, 128.0, 127.6, 127.5, 127.1, 126.5, 125.3, 124.9, 57.9; LRMS (EI, m/z): 360 (M⁺); HRMS (EI, m/z): Calcd. for C₂₃H₁₈³⁵ClS 360.0739, found: 360.0753.
(4-Chlorophenyl)(2-methyl-1-(naphthalen-2-yl)propyl)sulfane (9b)

Prepared according to the Method C.

Obtained as colorless oil; IR (neat, cm\(^{-1}\)): 3056, 2960, 1506, 1387, 1095, 1014, 860, 810, 774, 758, 745; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.77–7.73 (2H, m), 7.71–7.69 (1H, m), 7.51 (1H, s), 7.46 (1H, dd, \(J = 8.4, 1.8\) Hz), 7.42–7.39 (2H, m), 7.09 (2H, d, \(J = 8.2\) Hz), 7.02 (2H, d, \(J = 8.2\) Hz), 4.04 (1H, d, \(J = 8.3\) Hz), 2.28–2.22 (1H, m), 1.18 (3H, d, \(J = 6.3\) Hz), 0.90 (3H, d, \(J = 6.3\) Hz); \(^1\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 138.6, 134.1, 133.2, 132.9, 132.7, 132.5, 128.7, 127.9, 127.7, 127.5, 127.3, 126.3, 126.0, 125.7, 62.2, 33.9, 21.2, 20.9; LRMS (EI, m/z): 326 (M\(^+\)); HRMS (EI, m/z): Calcd. for C\(_{20}\)H\(_{19}\)ClS 326.0896, found: 326.0918.

(4-Chlorophenyl)(1-(naphthalen-2-yl)propyl)sulfane (9c)

Prepared according to the Method C.

Obtained as colorless oil; IR (neat, cm\(^{-1}\)): 3052, 2965, 2931, 1506, 1094, 1014, 854, 807, 774, 745; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.80–7.77 (2H, m), 7.73–7.71 (1H, m), 7.52 (1H, s), 7.46–7.42 (3H, m), 7.14 (2H, d, \(J = 9.0\) Hz), 7.10 (2H, d, \(J = 9.0\) Hz), 4.16–4.14 (1H, m), 2.09–1.98 (2H, m), 0.95 (3H, t, \(J = 7.5\) Hz); \(^1\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 139.0, 133.8, 133.4, 133.2, 133.1, 132.7, 128.7, 128.3, 127.7, 127.6, 126.7, 126.1, 125.8, 125.6, 56.0, 29.2, 12.4; LRMS (EI, m/z): 312 (M\(^+\)); HRMS (EI, m/z): Calcd. for C\(_{19}\)H\(_{17}\)ClS 312.0739, found: 312.0715.

Benzhydryl(ethyl)sulfide (9da)

Prepared according to the Method D.

Obtained as colorless oil; IR (neat, cm\(^{-1}\)): 3025, 2968, 2927, 1599, 1493, 1449, 1266, 1185, 1076, 1030, 971, 786, 747; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.41 (4H, d, \(J = 7.4\) Hz), 7.26 (4H, d, \(J = 7.4\) Hz), 7.17 (2H, t, \(J = 7.4\) Hz), 5.16 (1H, s), 2.36 (2H, q, \(J = 7.3\) Hz), 1.17 (3H, t, \(J = 7.3\) Hz); \(^1\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\) (TMS)) \(\delta\) (ppm): 138.6, 134.1, 133.2, 132.9, 132.7, 132.5, 128.7, 127.9, 127.7, 127.5, 127.3, 126.3, 126.0, 125.7, 62.2, 33.9, 21.2, 20.9; LRMS (EI, m/z): 312 (M\(^+\)); HRMS (EI, m/z): Calcd. for C\(_{20}\)H\(_{19}\)ClS 326.0896, found: 326.0918.
CDCl₃/TMS) δ (ppm): 141.4, 128.4, 128.2, 126.9, 53.7, 26.1, 14.1; LRMS (EI, m/z): 228 (M⁺); HRMS (EI, m/z): Calcd. for C₁₅H₁₆S 228.0973, found: 228.0978.

**Benzhydryl(isopropyl)sulfide (9db)**

Prepared according to the Method D.
Recrystallized from MeOH, colorless prisms, mp 61–62 °C; IR (neat, cm⁻¹): 2966, 1598, 1490, 1452, 1365, 1249, 1079, 1051, 1032, 754, 746; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.42–7.40 (4H, m), 7.26–7.23 (4H, m), 7.17–7.14 (2H, m), 5.19 (1H, d, J = 4.6 Hz), 2.67–2.62 (1H, m), 1.20–1.19 (6H, m); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 141.6, 128.4, 128.2, 126.9, 53.0, 34.9, 23.0; LRMS (EI, m/z): 242: (M⁺); HRMS (EI, m/z): Calcd. for C₁₆H₁₈S 242.1129, found: 242.1129.

**Benzhydryl(tert-butyl)sulfide (9dc)**

Prepared according to the Method D.
Recrystallized from MeOH, colorless prisms, mp 43–44 °C; IR (neat, cm⁻¹): 2962, 1598, 1490, 1449, 1365, 1156, 1080, 1030, 740; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.42 (4H, d, J = 7.6 Hz), 7.27 (4H, t, J = 7.6 Hz), 7.17 (2H, t, J = 7.6 Hz), 5.20 (1H, s), 1.26 (9H, s); ¹³C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 143.2, 128.4, 128.3, 126.7, 52.2, 44.6, 31.3; LRMS (EI, m/z): 256 (M⁺); HRMS (EI, m/z): Calcd. for C₁₇H₂₀S 256.1286, found: 256.1305.

**1-(Benzyloxy)hexan-3-yl)(methyl)sulfane (9e)**

To a solution of 3-(methylthio)-1-hexanol (10.0 mmol, 1.5 g) in DMF (10 mL) was added sodium hydride (15.0 mmol, 0.90 g, 60% oil suspension) at 0 °C under Ar. After stirring for 10 min, benzyl bromide (15.0 mmol, 1.8 mL) was added to the mixture and warmed to room temperature. After stirring 2 h, the reaction mixture was quenched with H₂O (10 mL) and extracted with Et₂O (30mL × 2). The combined organic layer was dried over MgSO₄ and concentrated in vacuo. The residue was purified by SiO₂ column chromatography (eluent: hexane/acetone = 100/1) to give 1-(benzyloxy)hexan-3-yl)(methyl)sulfane 9e (1.3 g, 53%) as colorless oil. Colorless oil; IR (neat, cm⁻¹): 3031, 2957, 2930, 2919, 2871, 2861, 1496, 1465, 1454, 1363, 1205, 1099, 1028, 734; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.37–7.33 (4H, m), 7.29–7.27 (1H, m), 4.52–4.51 (2H, m), 4.09–4.07 (2H, m), 3.83–3.81 (2H, m), 3.80–3.77 (2H, m), 3.51–3.48 (2H, m), 3.44–3.41 (2H, m), 3.35–3.32 (2H, m), 3.28–3.25 (2H, m), 3.13–3.10 (2H, m), 3.08–3.05 (2H, m), 3.05–3.02 (2H, m), 3.01–2.98 (2H, m), 2.94–2.91 (2H, m), 2.89–2.86 (2H, m), 2.85–2.82 (2H, m), 2.81–2.78 (2H, m), 2.77–2.74 (2H, m), 2.73–2.70 (2H, m), 2.69–2.66 (2H, m), 2.65–2.62 (2H, m), 2.61–2.58 (2H, m), 2.57–2.54 (2H, m), 2.54–2.51 (2H, m), 2.51–2.48 (2H, m), 2.47–2.44 (2H, m), 2.43–2.40 (2H, m), 2.40–2.37 (2H, m), 2.36–2.33 (2H, m), 2.33–2.30 (2H, m), 2.29–2.26 (2H, m), 2.26–2.23 (2H, m), 2.23–2.20 (2H, m), 2.20–2.17 (2H, m), 2.17–2.14 (2H, m), 2.14–2.11 (2H, m), 2.11–2.08 (2H, m), 2.08–2.05 (2H, m), 2.05–2.02 (2H, m), 2.02–1.99 (2H, m), 1.99–1.96 (2H, m), 1.96–1.93 (2H, m), 1.93–1.90 (2H, m), 1.90–1.87 (2H, m), 1.87–1.84 (2H, m), 1.84–1.81 (2H, m), 1.81–1.78 (2H, m), 1.78–1.75 (2H, m), 1.75–1.72 (2H, m), 1.72–1.69 (2H, m), 1.69–1.66 (2H, m), 1.66–1.63 (2H, m), 1.63–1.60 (2H, m), 1.60–1.57 (2H, m), 1.57–1.54 (2H, m), 1.54–1.51 (2H, m), 1.51–1.48 (2H, m), 1.48–1.45 (2H, m), 1.45–1.42 (2H, m), 1.42–1.39 (2H, m), 1.39–1.36 (2H, m), 1.36–1.33 (2H, m), 1.33–1.30 (2H, m), 1.30–1.27 (2H, m), 1.27–1.24 (2H, m), 1.24–1.21 (2H, m), 1.21–1.18 (2H, m), 1.18–1.15 (2H, m), 1.15–1.12 (2H, m), 1.12–1.09 (2H, m), 1.09–1.06 (2H, m), 1.06–1.03 (2H, m), 1.03–1.00 (2H, m), 1.00–0.97 (2H, m), 0.97–0.94 (2H, m), 0.94–0.91 (2H, m), 0.91–0.88 (2H, m), 0.88–0.85 (2H, m), 0.85–0.82 (2H, m), 0.82–0.79 (2H, m), 0.79–0.76 (2H, m), 0.76–0.73 (2H, m), 0.73–0.70 (2H, m), 0.70–0.67 (2H, m), 0.67–0.64 (2H, m), 0.64–0.61 (2H, m), 0.61–0.58 (2H, m), 0.58–0.55 (2H, m), 0.55–0.52 (2H, m), 0.52–0.49 (2H, m), 0.49–0.46 (2H, m), 0.46–0.43 (2H, m), 0.43–0.40 (2H, m), 0.40–0.37 (2H, m), 0.37–0.34 (2H, m), 0.34–0.31 (2H, m), 0.31–0.28 (2H, m), 0.28–0.25 (2H, m), 0.25–0.22 (2H, m), 0.22–0.19 (2H, m), 0.19–0.16 (2H, m), 0.16–0.13 (2H, m), 0.13–0.10 (2H, m), 0.10–0.07 (2H, m), 0.07–0.04 (2H, m), 0.04–0.01 (2H, m), 0.01–0.0 (2H, m).
3.68–3.59 (2H, m), 2.71–2.66 (1H, m), 2.01 (3H, s), 1.89–1.78 (2H, m), 1.53–1.43 (4H, m), 0.91 (3H, t, J = 7.2 Hz); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 138.5, 128.3, 127.6, 127.5, 73.0, 68.0, 43.3, 36.7, 34.4, 20.0, 14.0, 12.5; LRMS (EI, \(m/z\)): 238 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{22}\)OS 238.1391, found: 238.1416.

\((4\text{-Chlorophenyl})(4\text{-phenylbutan-2-yl})\text{sulfane} \,(9f)\)

Prepared according to the Method C.

Colorless oil; IR (neat, \(\text{cm}^{-1}\)): 3026, 2957, 2924, 2861, 1946, 1885, 1603, 1496, 1476, 1453, 1388, 1375, 1095, 1013, 819, 746; \(^1\)H NMR (600 MHz, DMSO-\(d_6\)) \(\delta\) (ppm): 7.37–7.32 (4H, m), 7.27–7.24 (2H, m), 7.17–7.15 (3H, m), 3.31–3.26 (1H, m), 2.75–2.67 (2H, m), 1.82–1.73 (2H, m), 1.26 (3H, d, \(J = 6.2\) Hz); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, DMSO-\(d_6\)) \(\delta\) (ppm): 141.3, 133.9, 132.4, 131.2, 128.9, 128.3, 128.2, 125.8, 41.7, 37.7, 32.3, 20.6; LRMS (EI, \(m/z\)): 276 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{16}\)H\(_{17}\)ClS 276.0740, found: 276.0735.

\(\text{Ethyl}(1\text{-}(\text{naphthalen-2-yl})\text{ethyl})\text{sulfane} \,(9g)\)

Prepared according to the Method C.

Colorless oil; IR (neat, \(\text{cm}^{-1}\)): 3053, 2967, 2924, 2866, 1632, 1599, 1506, 1474, 1447, 1373, 1287, 1217, 1125, 1055, 891, 856, 818; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) (ppm): 7.88–7.86 (3H, m), 7.79 (s, 1H), 7.54 (1H, dd, \(J = 8.8, 1.9\) Hz), 7.51–7.44 (2H, m), 4.20 (1H, q, \(J = 6.8\) Hz), 2.28 (2H, q, \(J = 7.3\) Hz), 1.57 (3H, d, \(J = 6.8\) Hz), 1.08 (3H, d, \(J = 7.3\) Hz); \(^{13}\)C\{\(^1\)H\} NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) (ppm): 141.3, 132.7, 132.1, 128.0, 127.5, 127.4, 126.1, 125.6, 125.4, 125.3, 42.8, 24.4, 22.0, 14.4; LRMS (EI, \(m/z\)): 216 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{16}\)S 216.0973, found: 216.0992.

\(3\text{-}(\text{Benzhydrylthio})\text{propane-1-thiol} \,(11)\)\(^{11}\)

To a suspension of ZnI\(_2\) (3.0 mmol, 0.96 g) and benzhydrol (3.0 mmol, 0.55 g) in CH\(_2\)Cl\(_2\) was added 1,3-propanedithiol (6.0 mmol, 0.65 g), and the mixture was stirred at room temperature for 8 h. The reaction mixture was quenched with water and extracted with CH\(_2\)Cl\(_2\) (30 mL \(\times\) 3). The combined organic layer was washed
with brine (30 mL) and dried over MgSO₄. The filtrate was concentrated under reduced pressure. The residue was purified by SiO₂ column chromatography (eluent: hexane/AcOEt = 30/1) to give 3-(benzhydrylthio)propane-1-thiol 11 (0.30 g, 36%) as colorless oil.

IR (neat, cm⁻¹): 3059, 3025, 2919, 2849, 1599, 1490, 1449, 1262, 1076, 1030, 748; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.42 (4H, d, J = 7.5 Hz), 7.31 (4H, t, J = 7.5 Hz), 7.22 (2H, t, J = 7.5 Hz), 5.14 (1H, s), 2.58 (2H, q, J = 7.4 Hz), 2.50 (2H, t, J = 7.4 Hz), 1.83 (2H, quint, J = 7.4 Hz), 1.28 (1H, t, J = 7.4 Hz); ¹³C {¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 141.3, 128.6, 128.3, 127.2, 54.2, 32.8, 30.6, 23.4; LRMS (FAB, m/z): 273 (M–H)⁺; HRMS (FAB, m/z): Calcd. for C₁₆H₁₇S₂ 273.0772, found: 273.0778.

4. Procedure for the SED (3)-mediated desulfurization

4-1. Representative procedure for the SED (3)-mediated desulfurization under thermal conditions (Table 1, entry 1)

Under an Ar atmosphere, a mixture of 3' (0.30 mmol, 0.16 g) and NaH (0.60 mmol, 15.0 mg) in DMF (2.0 mL) was stirred at room temperature for 1 h. After the SED generated, 1a (0.20 mmol, 54.4 mg) was added to the mixture and stirred at 100 °C for 24 h. The reaction mixture was quenched with saturated NH₄Cl aq. and extracted with diethyl ether (15 mL × 3). The combined organic layer was washed by water (30 mL × 2) and brine (30 mL), and then dried over Na₂SO₄. The solvent was removed under reduced pressure and the residue was purified by SiO₂ column chromatography (eluent: hexane/AcOEt = 30/1) to give the reduced product 2a.

4-2. Representative procedure for the SED (3)-mediated desulfurization under UV photolysis conditions (Table 3, 8a)

Under an Ar atmosphere, a mixture of 3' (0.30 mmol, 0.16 g) and NaH (0.60 mmol, 15.0 mg) in DMF (2.0 mL) was stirred at room temperature for 1 h. After the SED generated, 7a (0.20 mmol, 57.2 mg) was added to the mixture. The reaction mixture was then submitted to UV irradiation for 72 h, using UV lamps (352 nm, 6 watts). The reaction mixture was quenched with saturated NH₄Cl aq. and extracted with diethyl ether (15 mL × 3). The combined organic layer was washed by water (30 mL × 2) and brine (30 mL), and then dried over Na₂SO₄. The solvent was removed under reduced pressure and the residue was purified by SiO₂ column chromatography (eluent: hexane/AcOEt = 50/1) to give the reduced product 8a.
4-3. Reaction conditions of reductive desulfurization of 9b (Table 4, entry 3)

\[
\begin{array}{c}
\text{S} \quad \text{Cl} \\
\text{9b} \\
\text{NaH (2x equiv), Solvent, rt, 1 h} \\
\text{Temp., 24 h} \\
\text{3} \\
\end{array}
\xrightarrow{\text{[from 3' (x equiv),} \text{Solvent, rt, 1 h]}}
\begin{array}{c}
\text{10b} \\
\end{array}
\]

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<tr>
<th>Entry</th>
<th>x (equiv.)</th>
<th>Solvent</th>
<th>Temp. (°C)</th>
<th>Yield (%)(^a)</th>
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<td>1.5</td>
<td>DMSO</td>
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<td>25</td>
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<td>1.5</td>
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<td>48</td>
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<tr>
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<td>3.0</td>
<td>DMSO</td>
<td>140</td>
<td>69 (70)(^b)</td>
</tr>
</tbody>
</table>

\(^a\) Determined by \(^1\)H-NMR using 1,1,2-trichloroethane as an internal standard.

\(^b\) Isolated yields are given in parentheses.

**Diphenylmethane (2a)**

Obtained as colorless oil, 28.4 mg (85%) (Table 1, entry 1), 23.1 mg (67%) (Scheme 1), 21.1 mg (65%) (Table 4, entry 5), 34.2 mg (95%) (Table 4, entry 6), 34.8 mg (93%) (Table 4, entry 7); IR (neat, cm\(^{-1}\)): 3063, 3026, 2916, 1740, 1600, 1494, 1451, 1217, 1076, 1030, 728; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.30–7.24 (4H, m), 7.21–7.17 (6H, m), 3.98 (2H, s); \(^{13}\)C{\(^1\)H} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 141.1, 128.9, 128.4, 126.0, 41.9; LRMS (EI, \(m/z\)): 168 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{13}\)H\(_{12}\): 168.0939, found: 168.0930.

**1-Benzyl-4-methylbenzene (2b)**

Obtained as colorless oil, 31.0 mg (86%); IR (neat, cm\(^{-1}\)): 3025, 2920, 1744, 1513, 1494, 1452, 1433, 1271, 1073, 830, 788, 720; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.26 (2H, t, \(J = 7.5\) Hz), 7.18–7.16 (3H, m), 7.09–7.06 (4H, m), 3.93 (2H, s), 2.30 (3H, s); \(^{13}\)C{\(^1\)H} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 141.4, 138.1, 135.5, 129.1, 128.9, 128.8, 128.4, 126.0, 41.5, 21.0; LRMS (EI, \(m/z\)): 182 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{14}\): 182.1096, found: 182.1092.

**1-Benzyl-3-methylbenzene (2c)**

Obtained as colorless oil, 32.7 mg (92%); IR (neat, cm\(^{-1}\)): 3026, 2917, 2362, 1734, 1602, 1495, 1452, 1378,
1218, 1073, 1030, 776, 721; 1H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.27 (2H, t, J = 7.5 Hz), 7.21–7.15 (4H, m), 7.00–6.97 (3H, m), 3.93 (2H, s), 2.30 (3H, s); 13C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 141.2, 141.0, 138.0, 129.7, 128.9, 128.4, 128.3, 126.8, 125.99, 125.96, 41.9, 21.4; LRMS (EI, m/z): 182 (M⁺); HRMS (EI, m/z): Calcd. for C₁₄H₁₄: 182.1096, found: 182.1088.

1-Benzyl-2-methylbenzene (2d)

Obtained as colorless oil, 30.7 mg (84%); IR (neat, cm⁻¹): 3025, 2920, 1734, 1601, 1494, 1452, 1379, 1073, 1030, 767, 742; 1H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.28–7.25 (2H, m), 7.19–7.16 (3H, m), 7.10 (2H, d, J = 8.9 Hz), 6.69 (2H, d, J = 8.2 Hz), 3.89 (2H, s), 2.91 (6H, s); 13C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 149.1, 142.0, 129.5, 129.3, 128.8, 128.3, 125.7, 113.0, 40.9, 40.8; LRMS (EI, m/z): 211 (M⁺); HRMS (EI, m/z): Calcd. for C₁₄H₁₄N: 211.1361, found: 211.1351.

1-Benzyl-4-methoxybenzene (2e)

Obtained as colorless oil, 27.2 mg (71%); IR (neat, cm⁻¹): 3027, 2906, 2835, 1610, 1510, 1452, 1301, 1244, 1175, 1035, 837, 797, 770; 1H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.28–7.25 (2H, m), 7.19–7.16 (3H, m), 7.10 (2H, d, J = 8.6 Hz), 6.82 (2H, d, J = 8.6 Hz), 3.92 (2H, s), 3.77 (3H, s); 13C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 158.0, 141.6, 133.2, 129.8, 128.8, 128.4, 125.9, 113.9, 55.2, 41.0; LRMS (EI, m/z): 198 (M⁺); HRMS (EI, m/z): Calcd. for C₁₄H₁₄O: 198.1045, found: 198.1036.

4-Benzyl-N,N’-dimethylaniline (2f)

Obtained as yellowish oil, 12.5 mg (30%); IR (neat, cm⁻¹): 3025, 2894, 1616, 1517, 1493, 1452, 1340, 1222, 1162, 947, 793; 1H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.28–7.25 (2H, m), 7.19–7.16 (3H, m), 7.06 (2H, d, J = 8.9 Hz), 6.69 (2H, d, J = 8.2 Hz), 3.89 (2H, s), 2.91 (6H, s); 13C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 149.1, 142.0, 129.5, 129.3, 128.8, 128.3, 125.7, 113.0, 40.9, 40.8; LRMS (EI, m/z): 211 (M⁺); HRMS (EI, m/z): Calcd. for C₁₄H₁₄N: 211.1361, found: 211.1351.

1-Benzyl-4-fluorolbenzene (2g)
Obtained as colorless oil, 29.2 mg (79%); IR (neat, cm\(^{-1}\)): 3029, 2917, 1607, 1507, 1453, 1220, 1157, 1094, 841, 775, 723; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.28 (2H, t, \(J = 7.5\) Hz), 7.20 (1H, t, \(J = 7.5\) Hz), 7.16–7.12 (4H, m), 6.98–6.94 (2H, m), 3.94 (2H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 161.4 (d, \(J_{FC} = 243.5\) Hz), 140.9, 136.8, 130.3 (d, \(J_{FC} = 7.2\) Hz), 128.8, 128.5, 126.2, 115.2 (d, \(J_{FC} = 20.1\) Hz), 41.1; LRMS (EI, \(m/z\)): 186 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{13}\)H\(_{11}\)F: 186.0845, found: 186.0842.

1-Benzyl-4-chlorobenzene (2h)

Obtained as colorless oil, 35.2 mg (88%); IR (neat, cm\(^{-1}\)): 3027, 2915, 1730, 1490, 1453, 1406, 1205, 1092, 1016, 844, 790, 746; \(^1\)H NMR (600 MHz, Acetone-\(d_6\)) \(\delta\) (ppm): 7.31–7.28 (4H, m), 7.25–7.22 (4H, m), 7.21–7.18 (1H, m), 3.97 (2H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, Acetone-\(d_6\)) \(\delta\) (ppm): 141.8, 141.3, 132.2, 131.3, 129.6, 129.3, 129.2, 127.0, 41.6; LRMS (EI, \(m/z\)): 202 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{13}\)H\(_{11}\)Cl: 202.0549, found: 202.0549.

1-Benzyl-4-bromobenzene (2i)

Obtained as colorless oil, 42.5 mg (87%); IR (neat, cm\(^{-1}\)): 3026, 2911, 1603, 1495, 1486, 1452, 1403, 1072, 1012, 842, 786, 742; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.39–7.38 (2H, m), 7.28 (2H, t, \(J = 7.6\) Hz), 7.22–7.19 (1H, m), 7.15 (2H, d, \(J = 7.6\) Hz), 7.05 (2H, d, \(J = 8.2\) Hz), 3.91 (2H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 140.4, 140.1, 131.5, 130.6, 128.8, 128.6, 126.3, 119.9, 41.3; LRMS (EI, \(m/z\)): 246 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{13}\)H\(_{11}\)Br: 246.0044, found: 246.0040.

Ethyl 4-benzylbenzoate (2j)

Obtained as colorless oil, 28.1 mg (60%); IR (neat, cm\(^{-1}\)): 2981, 1714, 1611, 1496, 1415, 1367, 1271, 1177, 1099, 1020, 855, 753; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.96 (2H, d, \(J = 7.9\) Hz), 7.29 (2H, t, \(J = 7.9\) Hz), 7.26–7.24 (2H, m), 7.21 (1H, t, \(J = 7.2\) Hz), 7.17 (2H, d, \(J = 7.6\) Hz), 4.35 (2H, q, \(J = 6.8\) Hz), 4.03 (2H, s), 1.37 (3H, t, \(J = 6.8\) Hz); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 166.5, 146.3, 140.2, 129.8, 128.91, 128.88, 128.6, 128.4, 126.3, 60.8, 41.9, 14.3; LRMS (EI, \(m/z\)): 240 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{16}\)H\(_{10}\)O\(_2\): 240.1150, found: 240.1144.
4-Benzylbenzonitrile (2k)

Obtained as colorless oil, 16.6 mg (57%); IR (neat, cm\(^{-1}\)): 2922, 2854, 2223, 1600, 1506, 1453, 1410, 1176, 1021, 857, 796, 768, 729; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.56 (2H, d, \(J = 8.2\) Hz), 7.32–7.27 (4H, m), 7.25–7.22 (1H, m), 7.15 (2H, d, \(J = 7.5\) Hz), 4.02 (2H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 146.7, 139.3, 132.3, 129.6, 128.9, 126.7, 119.0, 110.1, 42.0; LRMS (EI, \(m/z\)): 193 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{11}\)N: 193.0891, found: 193.0879.

Bis(4-fluorophenyl)methane (2m)

Obtained as colorless oil, 31.4 mg (79%); IR (neat, cm\(^{-1}\)): 3041, 2921, 1602, 1506, 1220, 1155, 1096, 1016, 816; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.11 (4H, dd, \(J = 8.9, 5.5\) Hz), 6.99–6.95 (4H, m), 3.91 (2H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 161.5 (d, \(J_{FC} = 243.5\) Hz), 136.6 (d, \(J_{FC} = 2.9\) Hz), 130.2 (d, \(J_{FC} = 7.2\) Hz), 115.3 (d, \(J_{FC} = 21.5\) Hz), 40.2; LRMS (EI, \(m/z\)): 204 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{13}\)H\(_{10}\)F\(_2\): 204.0751, found: 204.0757.

Bis(4-chlorophenyl)methane (2n)

Obtained as colorless oil, 34.9 mg (72%); IR (neat, cm\(^{-1}\)): 2922, 2854, 1490, 1405, 1091, 1081, 1015, 860, 810, 783, 731; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.25 (4H, d, \(J = 8.6\) Hz), 7.08 (4H, d, \(J = 8.6\) Hz), 3.90 (2H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 139.0, 132.1, 130.2, 128.7, 40.5; LRMS (EI, \(m/z\)): 236 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{13}\)H\(_{10}\)Cl\(_2\): 236.0160, found: 236.0157.

Bis(4-bromophenyl)methane (2o)

Obtained as colorless oil, 39.5 mg (64%); IR (neat, cm\(^{-1}\)): 2920, 2853, 1896, 1485, 1438, 1399, 1068, 1011, 858, 806; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.41–7.39 (4H, d, \(J = 7.9\) Hz), 7.02 (4H, d, \(J = 8.2\) Hz), 3.87 (2H, s); \(^{13}\)C\{\(^1\)H\} NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 139.4, 131.6, 130.6, 120.2, 40.7; LRMS (EI, \(m/z\)): 324 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{13}\)H\(_{10}\)Br\(_2\): 323.9149, found: 323.9141.
Di(p-tolyl)methane (2p)

![Structural formula of Di(p-tolyl)methane](image)

Obtained as colorless oil, 34.3 mg (85%); IR (neat, cm⁻¹): 3020, 2920, 1512, 1436, 1118, 1021, 793, 747; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.09–7.06 (8H, m), 3.90 (2H, s), 2.30 (6H, s); ¹³C {¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 138.4, 135.4, 129.1, 128.7, 41.1, 21.0; LRMS (EI, m/z): 196 (M⁺); HRMS (EI, m/z): Calcd. for C₁₅H₁₆: 196.1252, found: 196.1257.

Bis-(2,4-dimethyl-phenyl)methane (2s)

![Structural formula of Bis-(2,4-dimethyl-phenyl)methane](image)

Obtained as colorless oil, 35.1 mg (79%); IR (neat, cm⁻¹): 3005, 2968, 2918, 2731, 1898, 1744, 1616, 1503, 1456, 1441, 1377, 1157, 1034, 874, 829, 806, 773; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.00 (2H, s), 6.90 (2H, d, J = 7.5 Hz), 6.76 (2H, d, J = 7.5 Hz), 3.82 (2H, s), 2.29 (6H, s), 2.22 (6H, s); ¹³C {¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 136.3, 135.51, 135.49, 130.8, 129.0, 126.6, 35.9, 20.9, 19.5; LRMS (EI, m/z): 224 (M⁺); HRMS (EI, m/z): Calcd. for C₁₇H₂₀: 224.1565, found: 224.1579.

1,2-Diphenylethan-1-one (2t)

![Structural formula of 1,2-Diphenylethan-1-one](image)

Obtained as colorless oil, 21.7 mg (56%); IR (neat, cm⁻¹): 2928, 1679, 1597, 1581, 1334, 1274, 1218, 991, 753, 752; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 8.02–8.00 (2H, m), 7.57–7.54 (1H, m), 7.46 (2H, t, J = 7.9 Hz), 7.32 (2H, t, J = 7.9 Hz), 7.28–7.24 (3H, m), 4.29 (2H, s); ¹³C {¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 197.5, 136.6, 134.5, 133.1, 129.4, 128.63, 128.60, 128.58, 126.9, 45.5; LRMS (EI, m/z): 196 (M⁺); HRMS (EI, m/z): Calcd. for C₁₄H₁₂O: 176.0888, found: 176.0885.

3,3-Dimethyl-1-phenylbutan-2-one (2u)

![Structural formula of 3,3-Dimethyl-1-phenylbutan-2-one](image)

Obtained as colorless oil, 20.6 mg (59%); IR (neat, cm⁻¹): 2971, 1711, 1497, 1478, 1455, 1366, 1060, 1010, 1001, 723; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.30 (2H, t, J = 7.3 Hz), 7.23 (1H, t, J = 7.3 Hz), 7.17 (2H, d, J = 7.3 Hz), 3.80 (2H, s), 1.20 (9H, s); ¹³C {¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 212.8, 134.9, 129.5, 128.3, 126.6, 44.7, 43.3, 26.4; LRMS (EI, m/z): 176 (M⁺); HRMS (EI, m/z): Calcd. for C₁₃H₁₈O: 176.1201,
3-((1,2-Diphenylethyl)thio)propane-1-thiol (8a)

Obtained as yellowish oil, 28.1 mg (49%); IR (neat, cm\(^{-1}\)): 3026, 2920, 1739, 1601, 1495, 1260, 1071, 1030, 765, 722; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.28–7.24 (4H, m), 7.21–7.18 (3H, m), 7.16–7.14 (1H, m), 7.04 (2H, d, \(J = 6.9\) Hz), 3.99 (1H, t, \(J = 7.6\) Hz), 3.16–3.08 (2H, m), 2.60–2.50 (2H, m), 2.38–2.28 (2H, m), 1.80–1.72 (2H, m); \(^{13}\)C\({\{^1}\}H\) NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 142.0, 138.9, 129.1, 128.4, 128.1, 128.0, 127.2, 126.3, 51.7, 43.2, 33.0, 29.6, 23.3; LRMS (EI, \(m/z\)): 288 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{17}\)H\(_{20}\)S\(_2\): 288.1006, found: 288.0967.

3-((1-Phenylethyl)thio)propane-1-thiol (8b)

Obtained as yellowish oil, 20.1 mg (48%); IR (neat, cm\(^{-1}\)): 2965, 2924, 1740, 1491, 1452, 1373, 1218, 1055, 1026, 764; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.35–7.30 (4H, m), 7.25–7.22 (1H, m), 3.94 (1H, q, \(J = 7.1\) Hz), 2.56–2.52 (2H, m), 2.47–2.37 (2H, m), 1.78–1.74 (2H, m), 1.57 (3H, d, \(J = 6.8\) Hz), 1.25 (1H, t, \(J = 8.0\) Hz); \(^{13}\)C\({\{^1}\}H\) NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 143.9, 128.5, 127.2, 127.1, 44.2, 33.1, 29.6, 23.4, 22.6; LRMS (EI, \(m/z\)): 212 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{11}\)H\(_{16}\)S\(_2\): 212.0693, found: 212.0687.

3-((1-Phenylpentyl)thio)propane-1-thiol (8c)

Obtained as yellowish oil, 25.1 mg (52%); IR (neat, cm\(^{-1}\)): 3055, 2955, 2929, 2858, 1740, 1491, 1452, 1378, 1261, 1107, 1103, 763; \(^1\)H NMR (400 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.31–7.27 (4H, m), 7.23–7.20 (1H, m), 3.74–3.72 (1H, m), 2.67–2.56 (2H, m), 2.39–2.28 (2H, m), 1.87–1.77 (4H, m), 1.33–1.19 (5H, m), 0.85 (3H, t, \(J = 7.2\) Hz); \(^{13}\)C\({\{^1}\}H\) NMR (100 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 142.9, 128.4, 127.8, 127.0, 49.8, 37.4, 36.3, 29.9, 29.5, 28.6, 22.5, 13.9; LRMS (EI, \(m/z\)): 254 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{22}\)S\(_2\): 254.1163, found: 254.1148.
3-((2,2-Dimethyl-1-phenylpropyl)thio)propane-1-thiol (8d)

Obtained as yellowish oil, 21.9 mg (46%); IR (neat, cm\(^{-1}\)): 2957, 2866, 1740, 1490, 1451, 1365, 1225, 1076, 844, 730; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.35–7.34 (2H, m), 7.28 (2H, t, \(J = 7.5\) Hz), 7.22 (1H, t, \(J = 7.5\) Hz), 3.62 (1H, s), 2.56–2.46 (2H, m), 2.34–2.22 (2H, m), 1.80–1.66 (2H, m), 1.22 (1H, t, \(J = 8.2\) Hz), 1.00 (9H, s); \(^{13}\)C\({\{^1\}H}\) NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 141.0, 129.7, 127.6, 126.8, 62.6, 35.5, 32.9, 30.0, 28.5, 23.4; LRMS (EI, \(m/z\)): 254 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{22}\)S\(_2\): 254.1163, found: 254.1131.

3-Naphthalen-2-ylsulfanylpropane-1-thiol (8e)

Obtained as yellowish oil, 20.3 mg (43%); IR (neat, cm\(^{-1}\)): 3052, 2916, 1599, 1508, 1233, 856, 817, 749; \(^1\)H NMR (600 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 7.82–7.79 (3H, m), 7.70 (1H, s), 7.50–7.44 (3H, m), 3.86 (2H, s), 2.96 (2H, q, \(J = 7.5\) Hz), 2.53 (2H, t, \(J = 7.5\) Hz), 1.85 (2H, quintet, \(J = 7.5\) Hz), 1.29 (1H, t, \(J = 7.5\) Hz); \(^{13}\)C\({\{^1\}H}\) NMR (150 MHz, CDCl\(_3\)/TMS) \(\delta\) (ppm): 135.6, 133.2, 132.5, 128.4, 127.64, 127.57, 127.2, 127.0, 126.2, 125.8, 36.5, 33.0, 29.5, 23.4; LRMS (EI, \(m/z\)): 248(M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{14}\)H\(_{16}\)S\(_2\): 248.0693, found: 248.0712.

2-Benzynaphthalene (10a)

Obtained as colorless oil, 41.0 mg (95%) (Table 4, entry 1), 40.4 mg (94%) (Table 4, entry 2); IR (neat, cm\(^{-1}\)):

\(\text{3025, 1601, 1507, 1495, 1453, 800, 754, 740; \(^1\)H NMR (600 MHz, Acetone–\(d_6\)) \(\delta\) (ppm): 7.84–7.80 (3H, m), 7.73 (1H, s), 7.47–7.42 (2H, m), 7.37 (1H, dd, \(J = 8.3, 1.4\) Hz), 7.29–7.28 (4H, m), 7.20–7.17 (1H, m), 4.14 (2H, s); \(^{13}\)C\({\{^1\}H}\) NMR (150 MHz, Acetone–\(d_6\)) \(\delta\) (ppm): 142.2, 139.9, 134.6, 133.1, 129.8, 129.3, 128.8, 128.43, 128.39, 128.3, 127.7, 126.9, 126.8, 126.2, 42.5; LRMS (EI, \(m/z\)): 218 (M\(^+\)); HRMS (EI, \(m/z\)): Calcd. for C\(_{17}\)H\(_{14}\): 218.1096, found: 218.1095.

2-Isobutylnaphthalene (10b)

Obtained as colorless oil, 25.8 mg (70%); IR (neat, cm\(^{-1}\)):

\(\text{2969, 1711, 1497, 1478, 1455, 1366, 1060, 1010; S27} \)
1H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.80–7.74 (3H, m), 7.57 (1H, s), 7.46–7.38 (2H, m), 7.30–7.29 (1H, m), 2.63 (2H, d, J = 7.6 Hz), 2.01–1.94 (1H, m), 0.94 (6H, d, J = 6.8 Hz); 13C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 139.3, 133.5, 132.0, 127.9, 127.55, 127.53, 127.4, 127.2, 125.7, 125.0, 45.6, 30.2, 22.5; LRMS (EI, m/z): 184 (M⁺); HRMS (EI, m/z): Calcd. for C₁₄H₁₆ 184.1252, found: 184.1248.

2-Propynaphthalene (10c)

![Structure](image_url)

Obtained as yellowish oil, 15.3 mg (43%); IR (neat, cm⁻¹): 3079, 1897, 1569, 1472, 1387, 1094, 1088, 1010, 814; ¹H NMR (600 MHz, CDCl₃/TMS) δ (ppm): 7.80–7.75 (3H, m), 7.60 (1H, s), 7.44–7.39 (2H, m), 7.33–7.31 (1H, m), 2.75 (2H, t, J = 7.6 Hz), 1.74 (2H, sextet, J = 7.6 Hz), 0.98 (3H, t, J = 7.6 Hz); 13C{¹H} NMR (150 MHz, CDCl₃/TMS) δ (ppm): 140.2, 133.6, 131.9 127.7, 127.6, 127.44, 127.37, 126.3, 125.8, 124.9, 38.2, 24.4, 13.9; LRMS (EI, m/z): 170 (M⁺); HRMS (EI, m/z): Calcd. for C₁₃H₁₄ 170.1096, found: 170.1121.

4-4. Unsuccessful sulfide substrates

<table>
<thead>
<tr>
<th>Entry</th>
<th>Substrate</th>
<th>Product</th>
<th>Conditions</th>
<th>Yield (%)</th>
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<tr>
<td>1</td>
<td>9e</td>
<td>10e</td>
<td>C</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>9f</td>
<td>10f</td>
<td>B</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>9g</td>
<td>10g</td>
<td>C</td>
<td>0</td>
</tr>
</tbody>
</table>

(Condition A: 3 (1.5 equiv) in DMF at 100 °C for 24 h), Condition B: 3 (3.0 equiv) in DMSO at 140 °C for 24 h, Condition C: 3 (3.0 equiv) in DMF under UV (352 nm) photolysis at room temperature for 72 h.)
5. Deuterium labelling experiment

\[
\begin{align*}
1a \quad \xrightarrow{\text{3-D}} \quad 2a \\
& \quad \text{[from 3'-D (1.5 equiv),} \\
& \quad \text{NaH (3.0 equiv), DMF, rt, 1 h]} \\
& \quad 100 \degree C, 24 h \\
& \quad 97\% (23\%D)
\end{align*}
\]

Determined by \(^1\text{H-NMR}\) using CH\(_2\)Br\(_2\) as an internal standard.
3
[from 3' (1.5 equiv), NaH (3.0 equiv), DMF-d_7, rt, 1 h]
100 °C, 24 h

2a
92% (<1% D)
Determined by ¹H-NMR using CH_2Br_2 as an internal standard.
6. $^1$H- and $^{13}$C-NMR spectra
$\text{NMe}_2$

2f
References


