

Disulfides as Efficient Thiolyating Reagents Enabling Selective Bis-sulfenylation of Aryl Dihalides Under Mild Copper-Catalyzed Conditions

Yunyun Liu*^a, Hang Wang,^a Jida Zhang,^b Jie-Ping Wan,^a Chengping Wen*^b

^aKey Laboratory of Functional Small Organic Molecules, Ministry of Education, College of Chemistry and Chemical Engineering, Jiangxi Normal University, Nanchang 330022, P. R. of China. Fax: +86 791 8812 0380; Tel: +86 791 8812 0380; E-mail: chemliuyunyun@gmail.com

^bCollege of Basic Medical Sciences, Zhejiang Chinese Medical University, Hangzhou 310053, P R China. E-mail: cpwen.zcmu@yahoo.com; Fax: +86 571 86633131; Tel: +86 571 86633131

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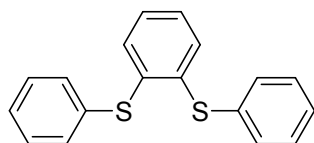
General information

Ligand **L5** and **L6** were synthesized following literature procedure,¹⁻² all other chemicals were obtained from commercial sources and used directly without further purification. Solvents used in the experiment have been treated following standard procedure. The reaction process was monitored by TLC. The ¹H and ¹³C NMR were recorded in 600 MHz or 400 MHz apparatus (as specified in the NMR spectra of every product) using CDCl₃ as solvent. The frequency used for measuring ¹H NMR was 600 MHz or 400 MHz, and for ¹³C NMR 150 MHz or 100 MHz, respectively. The chemical shifts were recorded in ppm using TMS as internal standard. Chemical shifts were recorded in ppm by employing TMS as internal standard. IR were performed with KBr film method. Melting points of all solid products were tested in X-4A apparatus without correcting temperature. HRMS data were obtained under EI or ESI model (specified in characterization data).

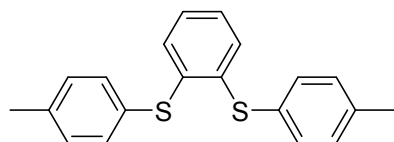
General procedure for the synthesis of bis-sulfenylated product 3. Diiodobenzene **1** (0.5 mmol), disulfide **2** (0.5 mmol), CuI (0.05 mmol), **L5** (0.1 mmol) and Cs₂CO₃ (1.0 mmol) were charged in a 25 mL round bottom flask equipped with stirring bar. Alcohol (2 mL) was added, and the resulting mixture was stirred at refluxing for 15h

(TLC). After cooling down to room temperature, the completed reaction mixture was added with 5 mL water and extracted with ethyl acetate (3 × 8 mL). The organic layers were combined and dried over anhydrous MgSO₄. After removing the solvent under reduced pressure, the residue was subjected to flash column chromatography to provide pure products. The reaction using dibromobenzene were performed in sealed tub at 110 °C under identical operation.

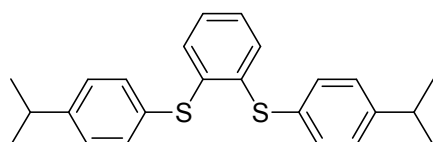
Characterization data



1,2-Bis(phenylthio)benzene (3a) Colorless liquid; ¹H NMR (CDCl₃, 600 MHz) δ = 7.41 (d, 4 H, *J* = 12 Hz), 7.37 (t, 4 H, *J* = 9 Hz), 7.32 (t, 2 H, *J* = 6 Hz), 7.19-7.14 (m, 4 H); ¹³C NMR (CDCl₃, 150 MHz) δ = 137.5, 134.6, 131.8, 131.4, 129.4, 127.5; IR (KBr, cm⁻¹): 3441, 3067, 1573, 1469, 725, 504; ESI-HRMS calcd for C₁₈H₁₅S₂ [M+H]⁺: 295.0610; found 295.0598.

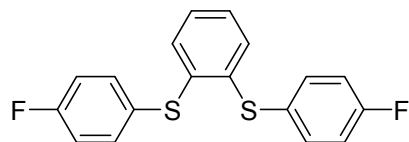


1,2-Bis(*p*-tolylthio)benzene (3b) White solid; mp 156-158 °C; ¹H NMR (CDCl₃, 600 MHz) δ = 7.31 (d, 4 H, *J* = 7.2 Hz), 7.17 (d, 4 H, *J* = 7.2 Hz), 7.06 (s, 4 H), 2.36 (s, 6 H); ¹³C NMR (CDCl₃, 150 MHz) δ = 137.8, 137.6, 132.5, 130.5, 130.2, 127.00, 21.2; IR (KBr, cm⁻¹): 3512, 3143, 2968, 1542, 701, 523; ESI-HRMS calcd for C₂₀H₁₉S₂ [M+H]⁺: 323.0923; found 323.0926.

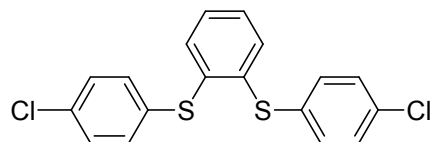


1,2-Bis(4-isopropylphenylthio)benzene (3c) White solid; mp 118-120 °C; ¹H NMR (CDCl₃, 600 MHz) δ = 7.37 (d, 4 H, *J* = 9.0 Hz), 7.25 (d, 4 H, *J* = 7.2 Hz), 7.11 (s, 4 H), 2.97-2.92 (m, 2 H), 1.30(d, 12 H, *J* = 9.0 Hz); ¹³C NMR (CDCl₃, 150 MHz) δ =

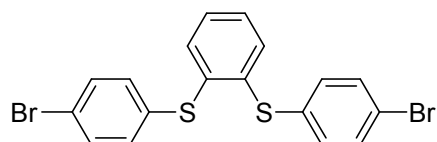
148.7, 137.6, 132.5, 130.9, 130.6, 127.6, 127.1, 33.6, 24.1; IR (KBr, cm^{-1}): 3524, 3028, 2963, 1543, 1036, 753; ESI-HRMS calcd for $\text{C}_{24}\text{H}_{27}\text{S}_2$ $[\text{M}+\text{H}]^+$: 379.1549; found 379.1549.



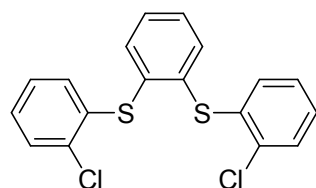
1,2-Bis(4-fluorophenylthio)benzene (3d) Yellow solid; mp 103-105 °C; ^1H NMR (CDCl_3 , 600 MHz) δ = 7.40 (q, 4 H, J = 6.0 Hz), 7.14 (q, 2 H, J = 6.0 Hz), 7.12-7.07 (m, 6 H); ^{13}C NMR (CDCl_3 , 150 MHz) δ = 163.5, 137.5, 134.3, 130.9, 129.0, 127.6, 116.6 (d, $J_{\text{C-F}}$ = 78.0 Hz); IR (KBr, cm^{-1}): 3501, 3023, 1374, 1026, 718, 532. EI-HRMS calcd for $\text{C}_{18}\text{H}_{12}\text{F}_2\text{S}_2$ $[\text{M}]^+$: 330.0343; found 330.0359.



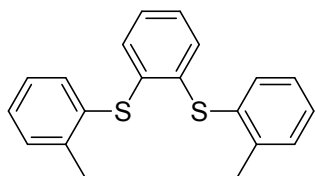
1,2-Bis(4-chlorophenylthio)benzene (3e) White solid; mp 106-108 °C; ^1H NMR (CDCl_3 , 600 MHz) δ = 7.33 (d, 4 H, J = 6.0 Hz), 7.29 (d, 4 H, J = 6.0 Hz), 7.19 (s, 4 H); ^{13}C NMR (CDCl_3 , 150 MHz) δ = 137.1, 133.7, 133.0, 132.9, 131.8, 129.6, 128.0; IR (KBr, cm^{-1}): 3469, 3006, 1546, 1004, 789, 517; EI-HRMS calcd for $\text{C}_{18}\text{H}_{12}\text{Cl}_2\text{S}_2$ $[\text{M}]^+$: 361.9752; found 361.9760.



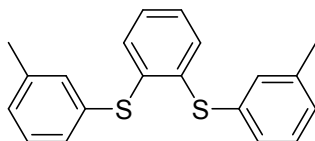
1,2-Bis(4-bromophenylthio)benzene (3f) Yellow solid; mp 111-113 °C; ^1H NMR (CDCl_3 , 600 MHz) δ = 7.47(d, 4 H, J = 12.0 Hz), 7.22-7.19 (m, 8 H); ^{13}C NMR (CDCl_3 , 150 MHz) δ = 137.0, 133.8, 133.0, 132.5, 131.9, 128.1, 121.7; IR (KBr, cm^{-1}): 3437, 3016, 1564, 1028, 704, 537; EI-HRMS calcd for $\text{C}_{18}\text{H}_{12}\text{Br}_2\text{S}_2$ $[\text{M}]^+$: 449.8742; found 449.8764.



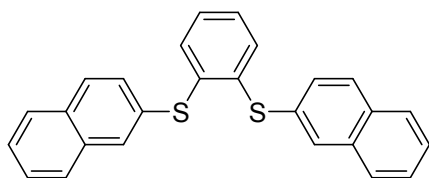
1,2-Bis(2-chlorophenylthio)benzene (3g) White solid; mp 69-71 °C; ^1H NMR (CDCl_3 , 600 MHz) $\delta = 7.30\text{-}7.28$ (m, 8 H), 7.23 (d, 1 H, $J = 6.0$ Hz), 7.16-7.14 (m, 3 H); ^{13}C NMR (CDCl_3 , 150 MHz) $\delta = 137.6, 133.9, 133.3, 133.0, 131.1, 129.8, 129.5, 128.5$; IR (KBr, cm^{-1}): 3469, 2979, 1504, 1062, 709, 517. EI-HRMS calcd for $\text{C}_{18}\text{H}_{12}\text{Cl}_2\text{S}_2$ $[\text{M}]^+$: 361.9752; found 361.9756.



1,2-Bis(o-tolylthio)benzene (3h) Colourless liquid; ^1H NMR (CDCl_3 , 600 MHz) $\delta = 7.31\text{-}7.27$ (m, 6 H), 7.19 (t, 2 H, $J = 6.0$ Hz), 7.11-7.08 (m, 2 H), 6.97 (d, 2 H, $J = 12.0$ Hz), 2.41 (s, 6 H); ^{13}C NMR (CDCl_3 , 150 MHz) $\delta = 140.4, 136.7, 133.1, 133.0, 130.7, 130.2, 128.1, 127.0, 126.8, 20.5$; IR (KBr, cm^{-1}): 3418, 3016, 2978, 1546, 757, 498. EI-HRMS calcd for $\text{C}_{20}\text{H}_{18}\text{S}_2$ $[\text{M}]^+$: 322.0844; found 322.0828.

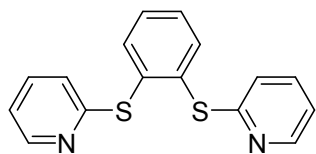


1,2-Bis(m-tolylthio)benzene (3i) Yellow liquid; ^1H NMR (CDCl_3 , 600 MHz) $\delta = 7.28\text{-}7.26$ (m, 4 H), 7.21 (d, 2 H, $J = 6.0$ Hz), 7.18 (t, 2 H, $J = 6.0$ Hz), 7.14 (d, 4 H, $J = 9.0$ Hz), 2.37 (s, 6 H); ^{13}C NMR (CDCl_3 , 150 MHz) $\delta = 139.2, 137.5, 134.2, 132.5, 131.3, 129.2, 129.0, 128.4, 127.4, 21.5$; IR (KBr, cm^{-1}): 3489, 2975, 7923, 1564, 709, 485; ESI-HRMS calcd for $\text{C}_{20}\text{H}_{19}\text{S}_2$ $[\text{M}+\text{H}]^+$: 323.0923; found 323.0931.

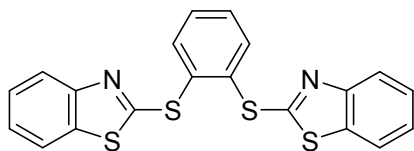


1,2-Bis(naphthalen-2-ylthio)benzene (3j) White solid; mp 98-100 °C; ^1H NMR (CDCl_3 , 400 MHz) $\delta = 7.94$ (s, 2 H), 7.87-7.83 (m, 4 H), 7.79 (t, 2 H, $J = 9.0$ Hz), 7.53-7.48 (m, 6 H), 7.27-7.24 (m, 2 H), 7.18-7.15 (m, 2 H); ^{13}C NMR (CDCl_3 , 100 MHz) $\delta = 137.5, 134.0, 132.6, 131.9, 131.7, 130.9, 129.2, 129.1, 127.9, 127.7, 127.6, 126.7, 126.5$; IR (KBr, cm^{-1}): 3443, 1567, 1443, 767, 738, 516; EI-HRMS calcd for

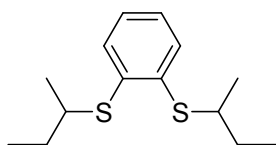
$C_{26}H_{18}S_2$ $[M]^+$: 394.0844; found 394.0851.



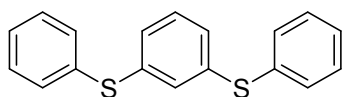
2-(2-(Pyridin-2-ylthio)phenylthio)pyridine (3k) Yellow solid; mp 71-73 °C; 1H NMR ($CDCl_3$, 600 MHz) δ = 8.40 (d, 2 H, J = 6.0 Hz), 7.64 (t, 2 H, J = 6.0 Hz), 7.02 (t, 2 H, J = 8.4 Hz), 7.02 (t, 2 H, J = 8.4 Hz), 7.00 (t, 4 H, J = 6.0 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ = 159.6, 149.8, 136.8, 136.6, 135.6, 129.6, 122.8, 120.5; IR (KBr, cm^{-1}): 3443, 3070, 1512, 1026, 738, 517; ESI-HRMS calcd for $C_{16}H_{13}N_2S_2$ $[M+H]^+$: 297.0515; found 297.0519.



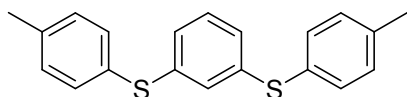
2-(2-(Benzo[d]thiazol-2-ylthio)phenylthio)benzo[d]thiazole (3l) Yellow liquid; 1H NMR ($CDCl_3$, 600 MHz) δ = 7.91 (d, 2 H, J = 6.0 Hz), 7.87 (d, 2 H, J = 6.0 Hz), 7.70 (d, 2 H, J = 9.0 Hz), 7.53 (q, 2 H, J = 6.0 Hz), 7.42 (t, 2 H, J = 6.0 Hz), 7.31 (t, 2 H, J = 9.0 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ = 166.0, 153.7, 136.1, 135.7, 131.0, 126.3, 124.8, 122.4, 121.0; IR (KBr, cm^{-1}): 3548, 3038, 1504, 1069, 727, 496. ESI-HRMS calcd for $C_{20}H_{13}N_2S_4$ $[M+H]^+$: 408.9956; found 408.9948.



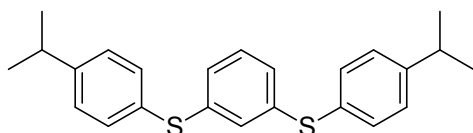
1,2-Bis(sec-butylthio)benzene (3m) Yellow liquid; 1H NMR ($CDCl_3$, 600 MHz) δ = 7.32 (t, 2 H, J = 6.0 Hz), 7.14 (t, 2 H, J = 6.0 Hz), 3.29 (q, 2 H, J = 6.0 Hz), 1.71-1.69 (m, 2 H), 1.61-1.56 (m, 2 H), 1.30 (d, 6 H, J = 6.0 Hz), 1.03 (t, 6 H, J = 9.0 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ = 137.6, 130.5, 126.5, 43.8, 29.7, 20.2, 11.4; IR (KBr, cm^{-1}): 3440, 2963, 2924, 1445, 1042, 746; ESI-HRMS calcd for $C_{14}H_{23}S_2$ $[M+H]^+$: 255.1236; found 255.1239.



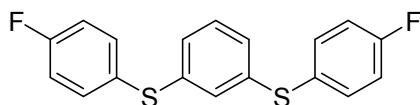
1,3-Bis(phenylthio)benzene (3n) Yellow liquid; ^1H NMR (CDCl_3 , 400 MHz) δ = 7.31-7.28 (m, 4 H), 7.23-7.15 (m, 7 H), 7.08-7.07 (m, 3 H); ^{13}C NMR (CDCl_3 , 100 MHz) δ = 137.3, 133.8, 133.3, 132.5, 131.6, 129.9, 128.8, 121.9; IR (KBr, cm^{-1}): 3447, 3078, 1574, 1456, 738, 517; ESI-HRMS calcd for $\text{C}_{18}\text{H}_{15}\text{S}_2$ $[\text{M}+\text{H}]^+$: 295.0610; found 295.0599.



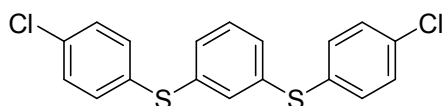
1,3-Bis(*p*-tolylthio)benzene (3o) Yellow solid; mp 79-81 °C; ^1H NMR (CDCl_3 , 600 MHz) δ = 7.32 (d, 4 H, J = 6.0 Hz), 7.16-7.11 (m, 6 H), 7.05 (d, 2 H, J = 6.0 Hz), 2.39 (s, 6 H); ^{13}C NMR (CDCl_3 , 150 MHz) δ = 138.7, 138.0, 132.9, 130.2, 130.1, 129.4, 129.1, 126.7, 21.5; IR (KBr, cm^{-1}): 3451, 3021, 2963, 1568, 1024, 738; ESI-HRMS calcd for $\text{C}_{20}\text{H}_{19}\text{S}_2$ $[\text{M}+\text{H}]^+$: 323.0923; found 323.0908.



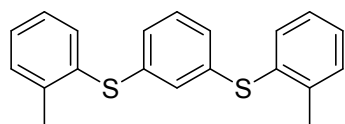
1,3-Bis(4-isopropylphenylthio)benzene (3p) Yellow liquid; ^1H NMR (CDCl_3 , 600 MHz) δ = 7.32 (d, 4 H, J = 6.0 Hz), 7.22 (s, 1 H), 7.19 (d, 4 H, J = 6.0 Hz), 7.15 (d, 1 H, J = 6.0 Hz), 7.06 (d, 2 H, J = 9.0 Hz), 2.93-2.89 (m, 2 H), 1.27 (d, 12 H, J = 6.0 Hz); ^{13}C NMR (CDCl_3 , 150 MHz) δ = 148.9, 138.2, 132.5, 130.9, 130.2, 129.5, 127.6, 127.4, 33.9, 23.9; IR (KBr, cm^{-1}): 3494, 3032, 2978, 1563, 1046, 726; ESI-HRMS calcd for $\text{C}_{24}\text{H}_{27}\text{S}_2$ $[\text{M}+\text{H}]^+$: 379.1549; found 379.1561.



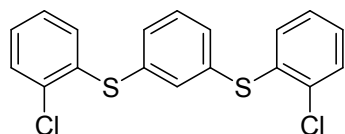
1,3-Bis(4-fluorophenylthio)benzene (3q) White solid; mp 69-71 °C; ^1H NMR (CDCl_3 , 600 MHz) δ = 7.40-7.38 (m, 4 H), 7.18 (t, 1 H, J = 12 Hz), 7.18 (m, 6 H), 6.95 (t, 1 H, J = 3 Hz); ^{13}C NMR (CDCl_3 , 150 MHz) δ = 163.6, 161.9, 138.7, 135.2, 129.5, 128.6, 126.7, 116.6 (d, $J_{\text{C-F}}$ = 90.0 Hz); IR (KBr, cm^{-1}): 3521, 3043, 1394, 1016, 703, 516; EI-HRMS calcd for $\text{C}_{20}\text{H}_{18}\text{F}_2\text{S}_2$ $[\text{M}]^+$: 330.0343; found 330.0347.



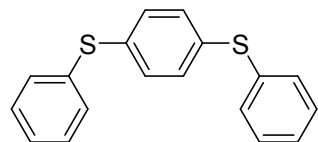
1,3-Bis(4-chlorophenylthio)benzene (3r) White solid; mp 71-73 °C; ^1H NMR (CDCl_3 , 600 MHz) $\delta = 7.47$ (d, 2 H, $J = 6$ Hz), 7.26-7.19 (m, 10 H); ^{13}C NMR (CDCl_3 , 150 MHz) $\delta = 136.2, 135.5, 133.8, 132.3, 130.1, 128.5, 128.4, 127.4$; IR (KBr, cm^{-1}): 3498, 3021, 1569, 1035, 712, 482; EI-HRMS calcd for $\text{C}_{18}\text{H}_{12}\text{Cl}_2\text{S}_2$ $[\text{M}]^+$: 361.9752; found 361.9769.



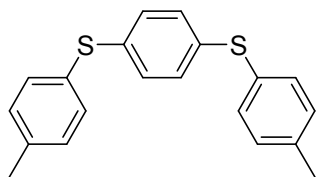
1,3-Bis(o-tolylthio)benzene (3s) Colourless liquid; ^1H NMR (CDCl_3 , 600 MHz) $\delta = 7.32$ (d, 4 H, $J = 6.0$ Hz), 7.25-7.24 (m, 4 H), 7.17- 7.14 (m, 3 H), 6.98 (d, 2 H, $J = 6.0$ Hz), 6.88 (s, 1 H), 2.34 (s, 6 H); ^{13}C NMR (CDCl_3 , 150 MHz) $\delta = 140.7, 138.0, 134.0, 132.5, 130.7, 129.5, 128.5, 128.3, 126.8, 126.2, 20.5$; IR (KBr, cm^{-1}): 3471, 3051, 2973, 1518, 1064, 728; ESI-HRMS calcd for $\text{C}_{20}\text{H}_{19}\text{S}_2$ $[\text{M}+\text{H}]^+$: 323.0923; found 323.0930.



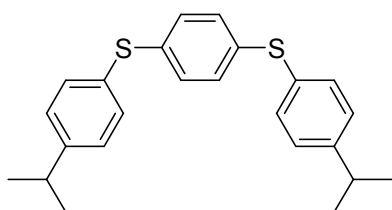
1,3-Bis(2-chlorophenylthio)benzene (3t) Colourless liquid; ^1H NMR (CDCl_3 , 600 MHz) $\delta = 7.40$ (d, 2 H, $J = 6.0$ Hz), 7.33-7.31 (m, 4 H), 7.19-7.14 (m, 6 H); ^{13}C NMR (CDCl_3 , 150 MHz) $\delta = 135.3, 134.8, 134.7, 134.6, 131.7, 131.1, 130.3, 130.0, 128.2, 127.4$; IR (KBr, cm^{-1}): 3429, 3076, 1549, 1434, 794, 519; EI-HRMS calcd for $\text{C}_{18}\text{H}_{12}\text{Cl}_2\text{S}_2$ $[\text{M}]^+$: 361.9752; found 361.9763.



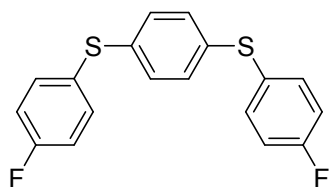
1,4-Bis(phenylthio)benzene (3u) White solid; mp 78-80 °C; ^1H NMR (CDCl_3 , 600 MHz) $\delta = 7.40$ (d, 4 H, $J = 9.0$ Hz), 7.35 (t, 4 H, $J = 6.0$ Hz), 7.30 (t, 2 H, $J = 9.0$ Hz), 7.26 (s, 4 H); ^{13}C NMR (CDCl_3 , 150 MHz) $\delta = 135.1, 135.0, 131.5, 131.2, 129.3, 127.4$; IR (KBr, cm^{-1}): 3442, 3073, 1578, 1437, 693, 509; EI-HRMS calcd for $\text{C}_{18}\text{H}_{14}\text{S}_2$ $[\text{M}]^+$: 294.0531; found 294.0533.



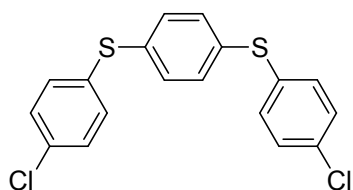
1,4-Bis(p-tolylthio)benzene (3v) White solid; mp 94-96 °C; ^1H NMR (CDCl_3 , 400 MHz) δ = 7.27 (d, 4 H, J = 8.0 Hz), 7.12 (d, 8 H, J = 8.0 Hz), 2.33 (s, 6 H); ^{13}C NMR (CDCl_3 , 100 MHz) δ = 137.8, 135.4, 132.3, 131.0, 130.3, 130.1, 21.1; IR (KBr, cm^{-1}): 3498, 3036, 2918, 1536, 747, 486; ESI-HRMS calcd for $\text{C}_{20}\text{H}_{19}\text{S}_2$ $[\text{M}+\text{H}]^+$: 323.0923; found 323.0917.



1,4-Bis(4-isopropylphenylthio)benzene (3w) Yellow solid; mp 76-77 °C; ^1H NMR (CDCl_3 , 600 MHz) δ = 7.31 (d, 4 H, J = 6.0 Hz), 7.17 (d, 4 H, J = 6.0 Hz), 7.06 (s, 4 H), 2.94-2.90 (m, 2 H), 1.28 (d, 12 H, J = 6.0 Hz); ^{13}C NMR (CDCl_3 , 150 MHz) δ = 135.3, 132.2, 131.4, 130.3, 127.5, 125.5, 33.70, 24.0; IR (KBr, cm^{-1}): 3456, 3018, 2978, 1568, 689, 479; EI-HRMS calcd for $\text{C}_{24}\text{H}_{26}\text{S}_2$ $[\text{M}]^+$: 378.1470; found 378.1458.

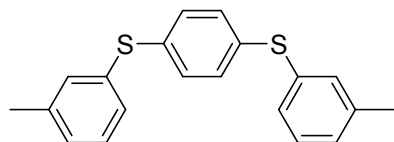


1,4-Bis(4-fluorophenylthio)benzene (3x) White solid; mp 83-84 °C; ^1H NMR (CDCl_3 , 600 MHz) δ = 7.41 (q, 4 H, J = 6.0 Hz), 7.17 (s, 4 H), 7.11 (t, 4 H, J = 9.0 Hz); ^{13}C NMR (CDCl_3 , 150 MHz) δ = 163.4, 135.4, 134.4, 130.3, 129.7, 116.6 (d, $J_{\text{C-F}}$ = 84.0 Hz); IR (KBr, cm^{-1}): 3521, 3013, 1354, 1056, 728, 507; EI-HRMS calcd for $\text{C}_{18}\text{H}_{12}\text{F}_2\text{S}_2$ $[\text{M}]^+$: 330.0343; found 330.0337.

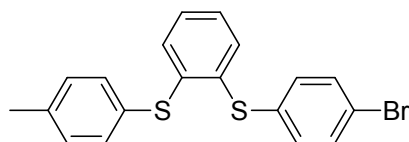


1,4-Bis(4-chlorophenylthio)benzene (3y) White solid; mp 142-144 °C; ^1H NMR

(CDCl₃, 400 MHz) δ = 7.27 (brs, 7 H), 7.21 (brs, 5 H); ¹³C NMR (CDCl₃, 100 MHz) δ = 134.8, 133.6, 132.7, 131.3, 129.5; IR (KBr, cm⁻¹): 3478, 3041, 1529, 1056, 732, 491; EI-HRMS calcd for C₁₈H₁₂Cl₂S₂ [M]⁺: 361.9752; found 361.9757.



1,4-Bis(m-tolylthio)benzene (3z) White solid; mp 84-86 °C; ¹H NMR (CDCl₃, 600 MHz) δ = 7.27-7.21 (m, 10 H), 7.13 (d, 2 H, *J* = 9.0 Hz), 2.37 (s, 6 H); ¹³C NMR (CDCl₃, 150 MHz) δ = 139.2, 135.0, 134.7, 132.2, 131.0, 129.2, 128.7, 128.4, 21.4; IR (KBr, cm⁻¹): 3508, 3012, 2978, 1507, 698, 507; ESI-HRMS calcd for C₂₀H₁₉S₂ [M+H]⁺: 323.0923; found 323.0914.

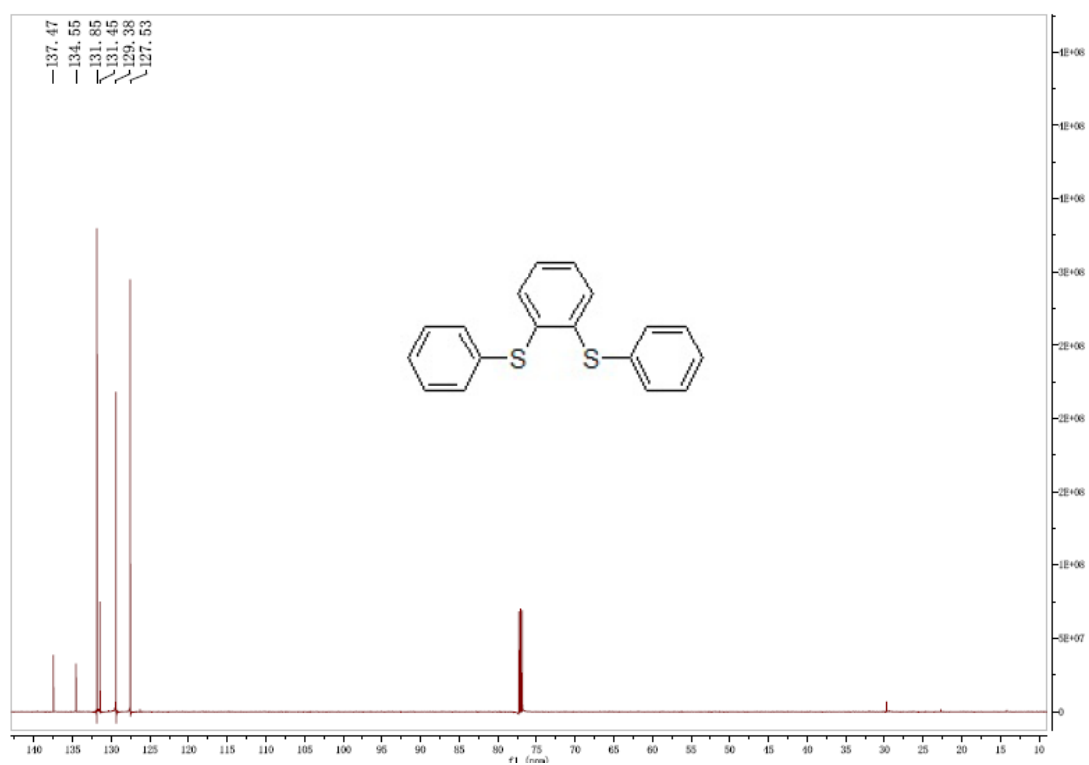
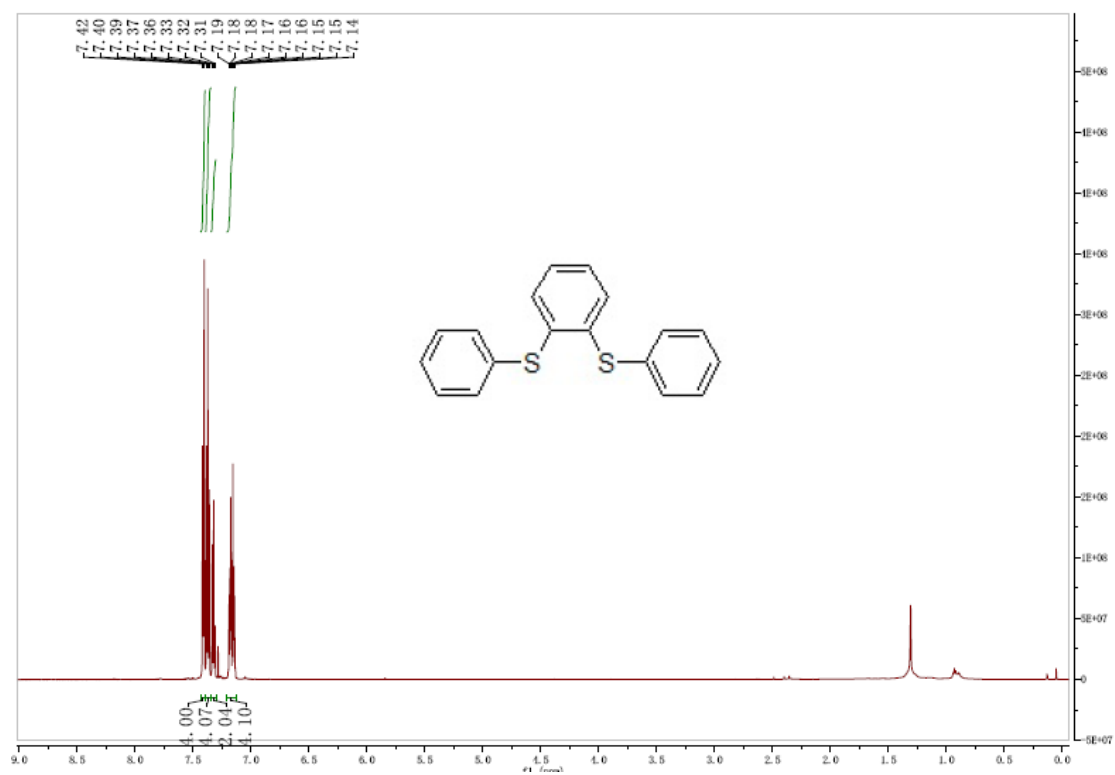


(4-bromophenyl)(2-(p-tolylthio)phenyl)sulfane (3bf) White solid; mp 72-74 °C; ¹H NMR (CDCl₃, 600 MHz) δ = 7.41 (d, 2 H, *J* = 6.0 Hz), 7.30 (d, 2 H, *J* = 6.0 Hz), 7.23 (d, 1 H, *J* = 6.0 Hz), 7.18-7.08 (m, 6 H), 6.97 (d, 1 H, *J* = 6.0 Hz), 2.37 (s, 3 H); ¹³C NMR (CDCl₃, 100 MHz) δ = 140.9, 138.4, 134.7, 133.6, 133.4, 133.0, 132.3, 132.0, 130.4, 129.7, 128.4, 126.7, 121.0, 21.0; IR (KBr, cm⁻¹): 3501, 3023, 1512, 670; EI-MS [M]⁺: 386.0; EI-HRMS calcd for C₂₀H₁₈S₂ [M]⁺: 385.9793; Found: 385.9797.

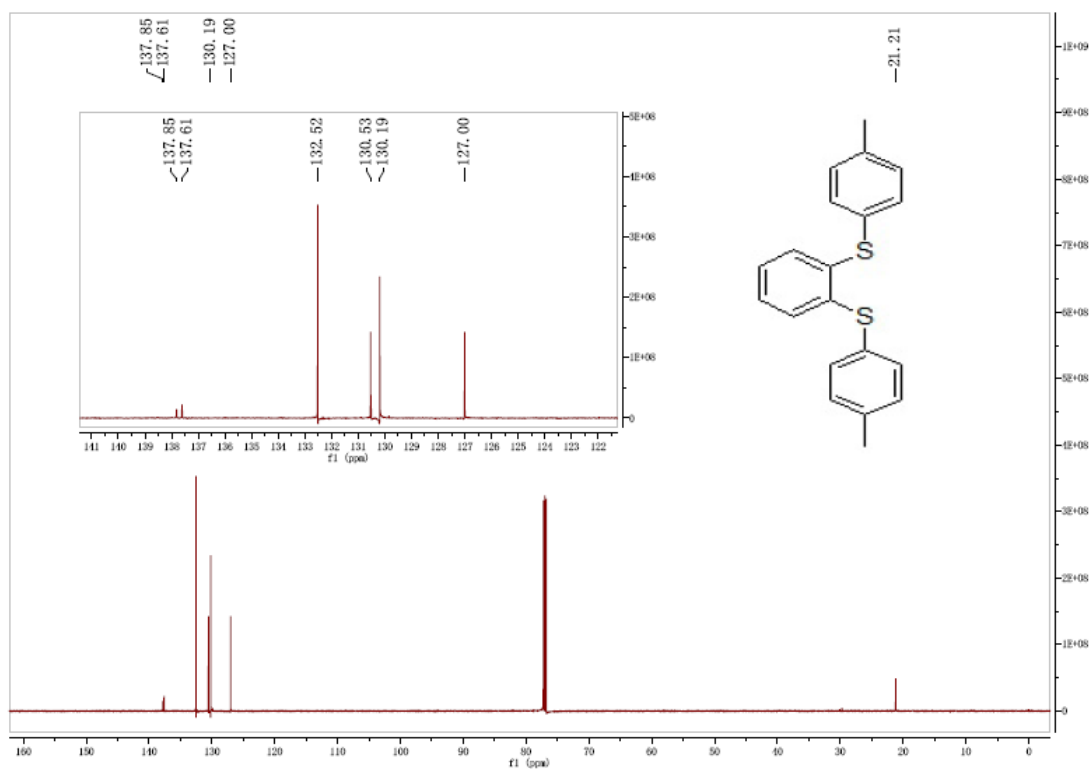
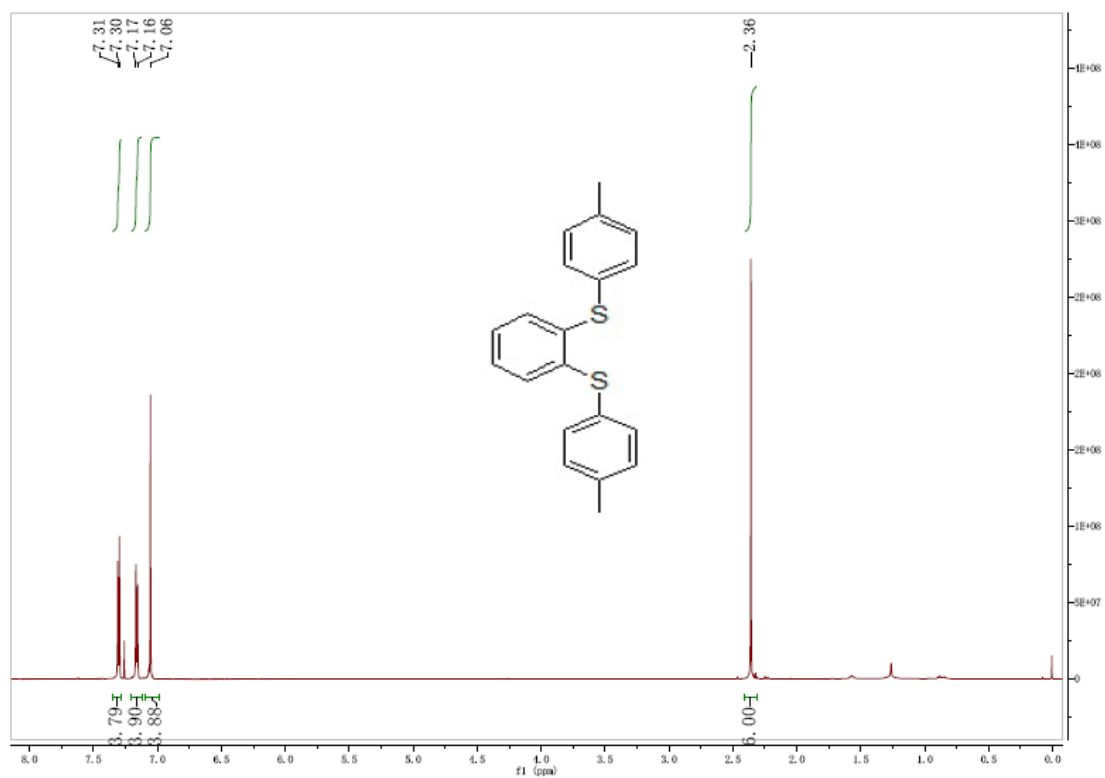
(1) F. M. A. A. El-Taweel, M. H. Elnagdi, *J. Heterocyclic Chem.* **2001**, *38*, 981.

(2) Y. Liu, R. Zhou, J.-P. Wan, *Synth. Commun.* **2013**, *43*, 2475.

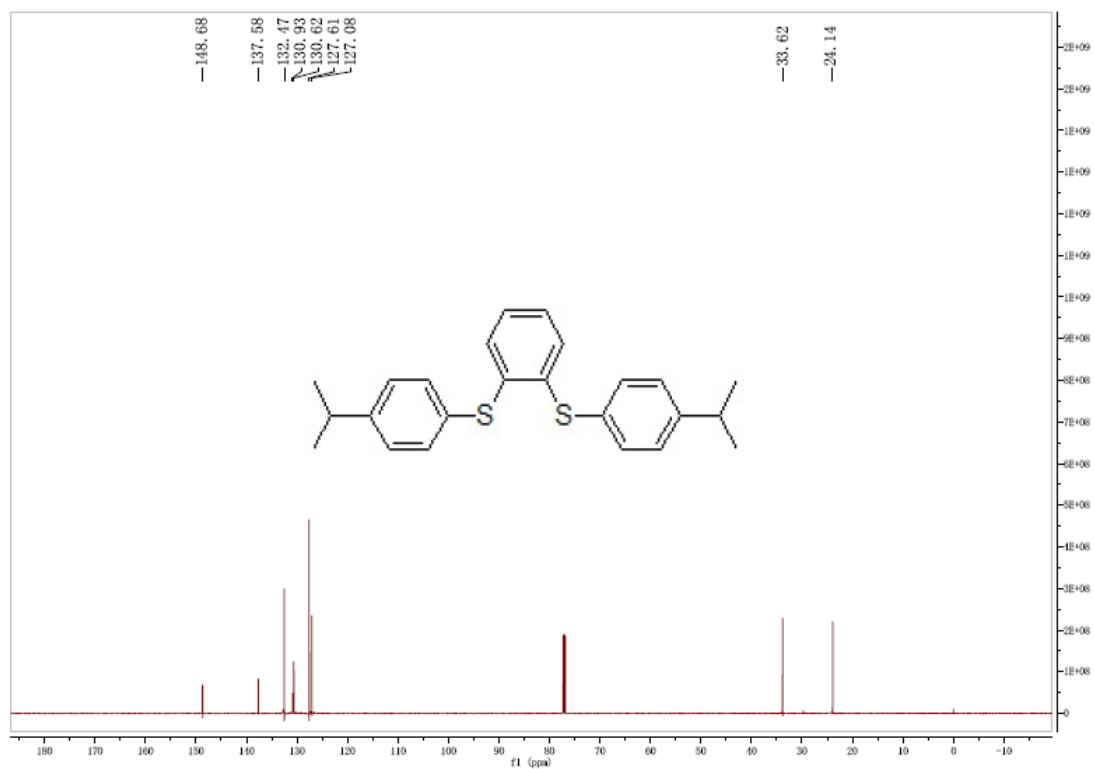
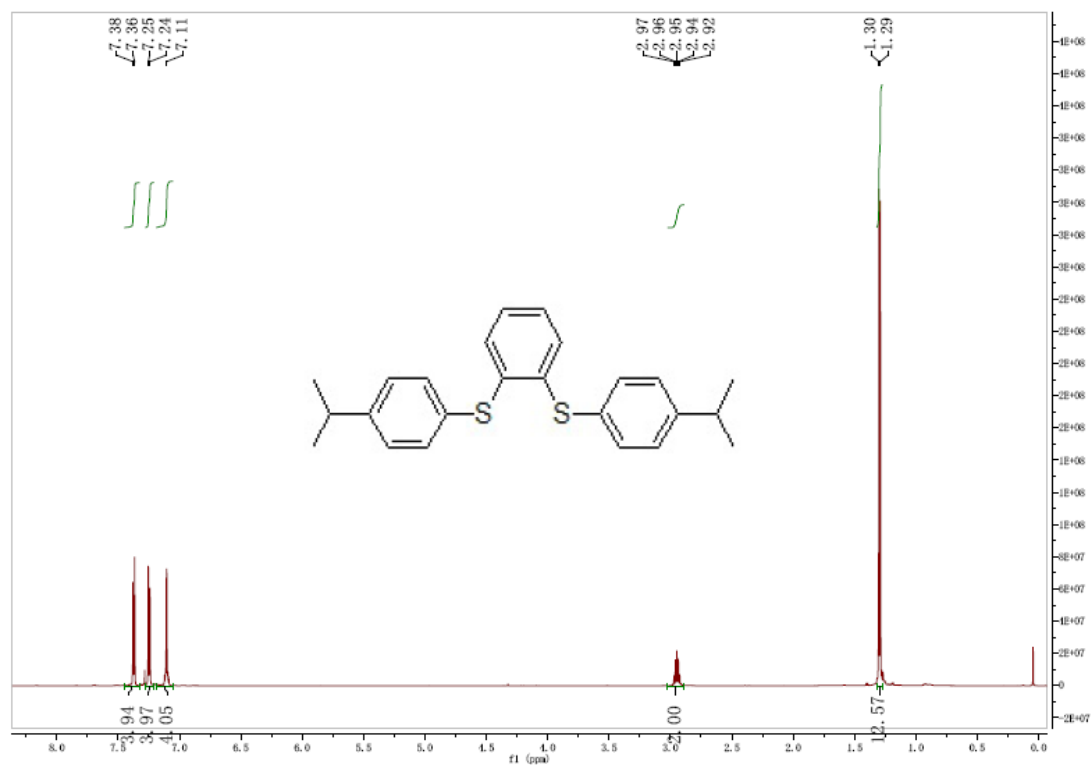
^1H and ^{13}C NMR of **3a** (600 MHz apparatus)



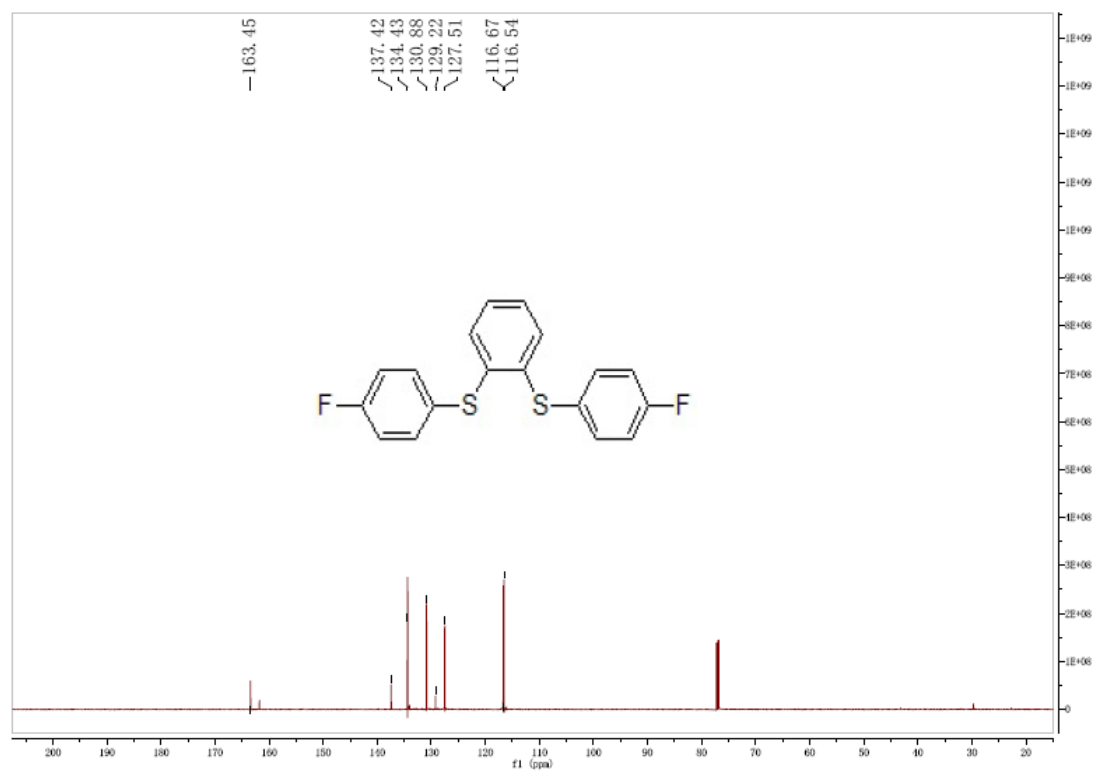
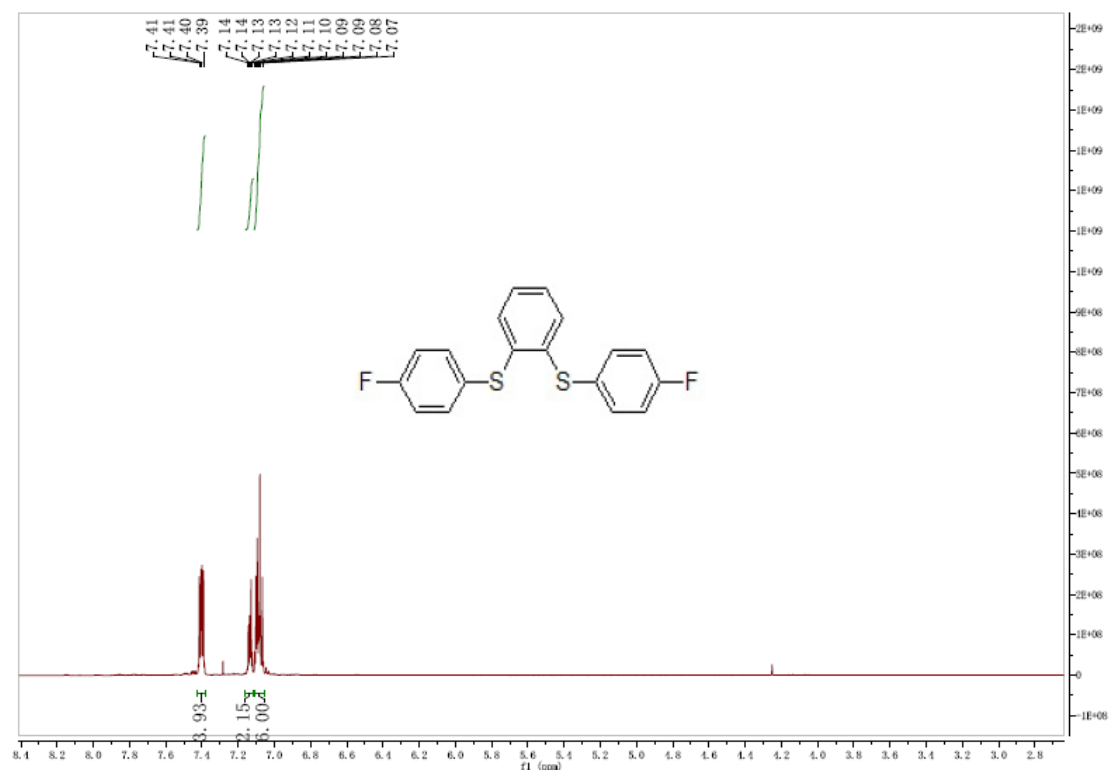
^1H and ^{13}C NMR of **3b** (600 MHz apparatus)



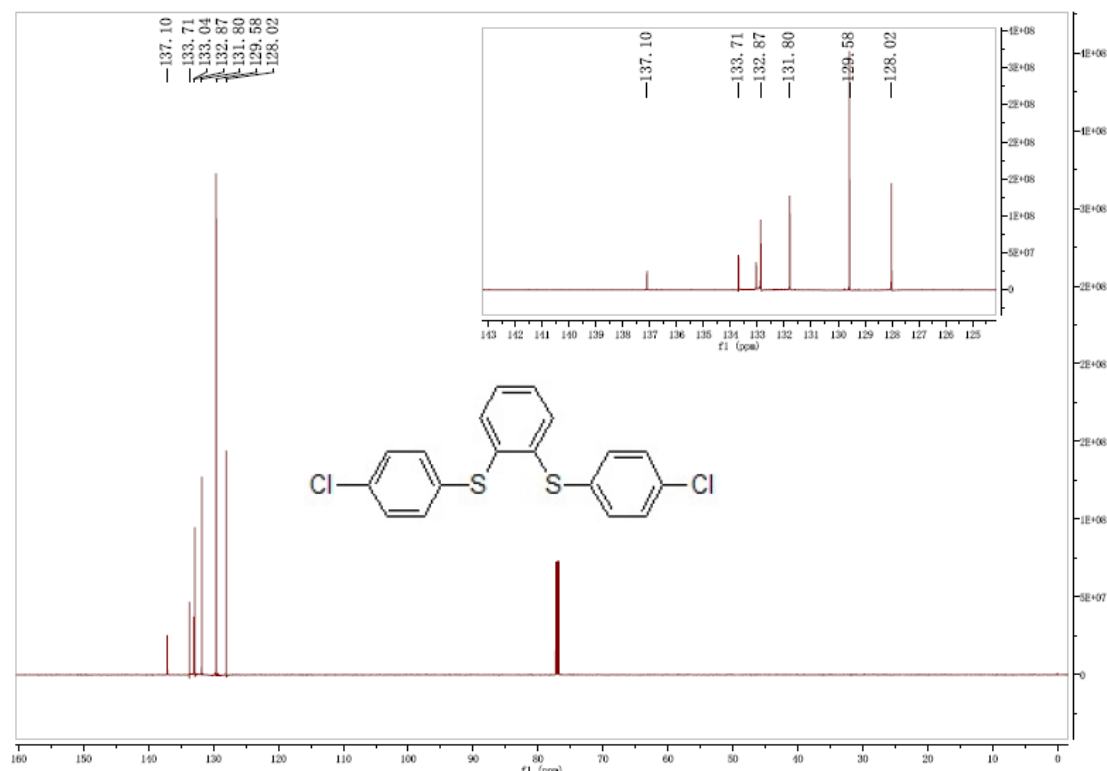
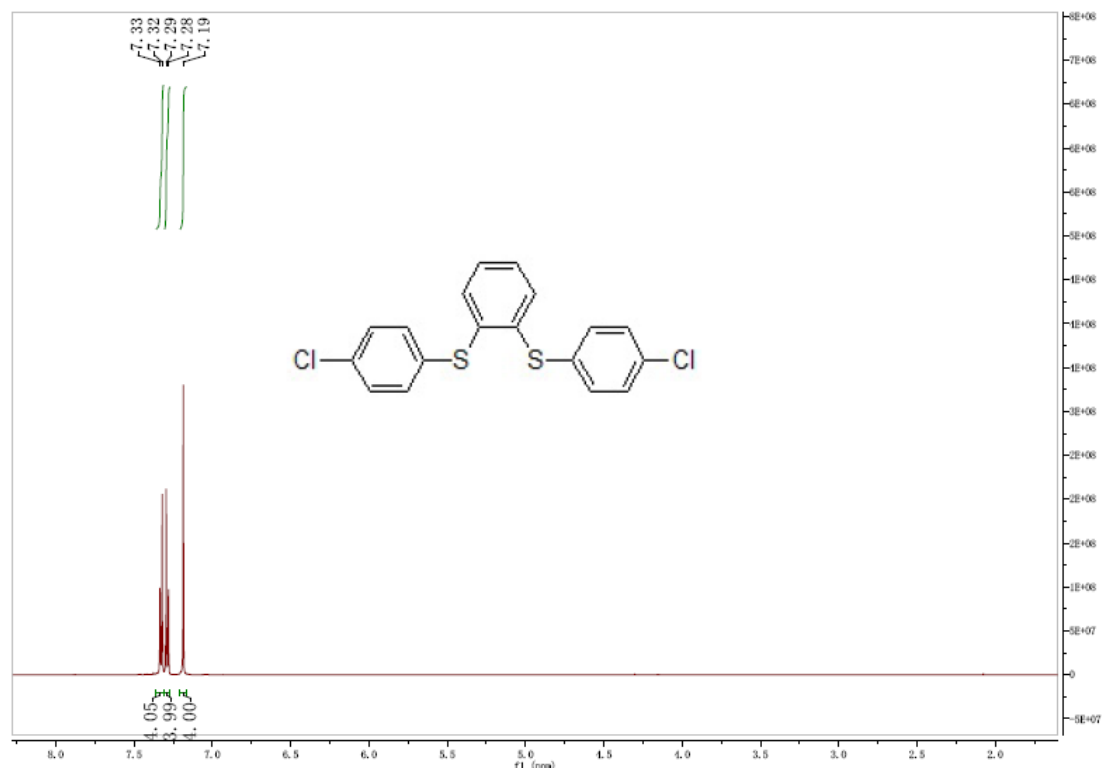
^1H and ^{13}C NMR of **3c** (600 MHz apparatus)



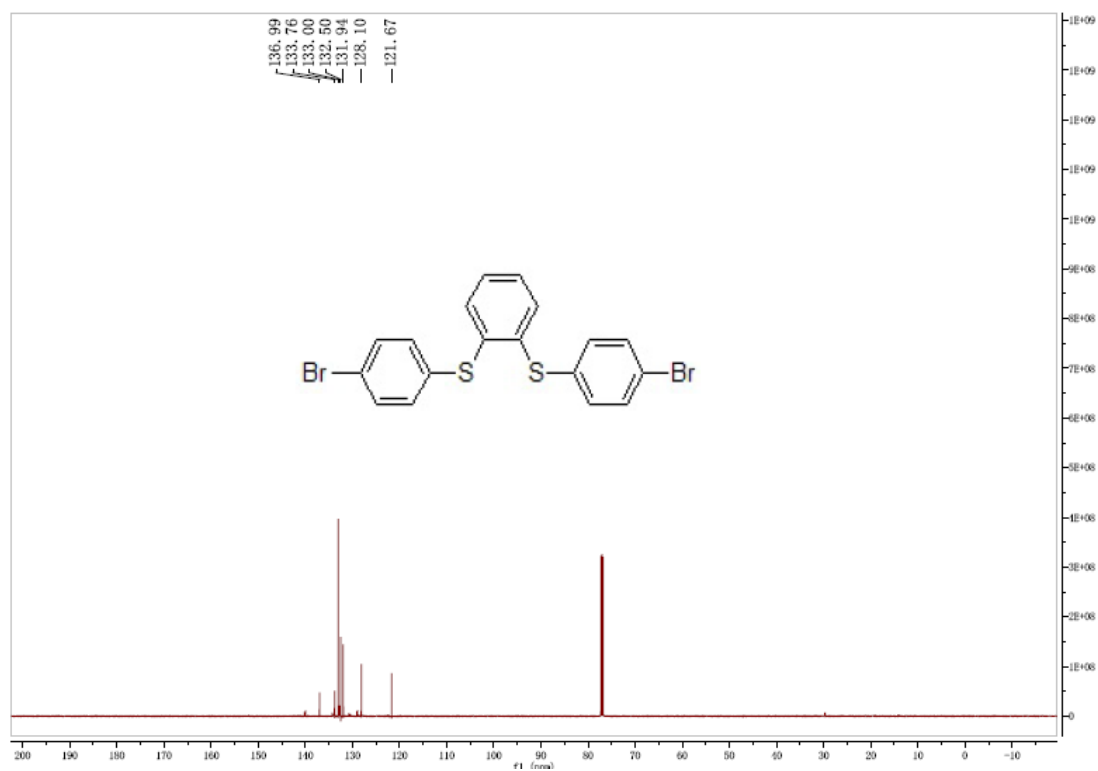
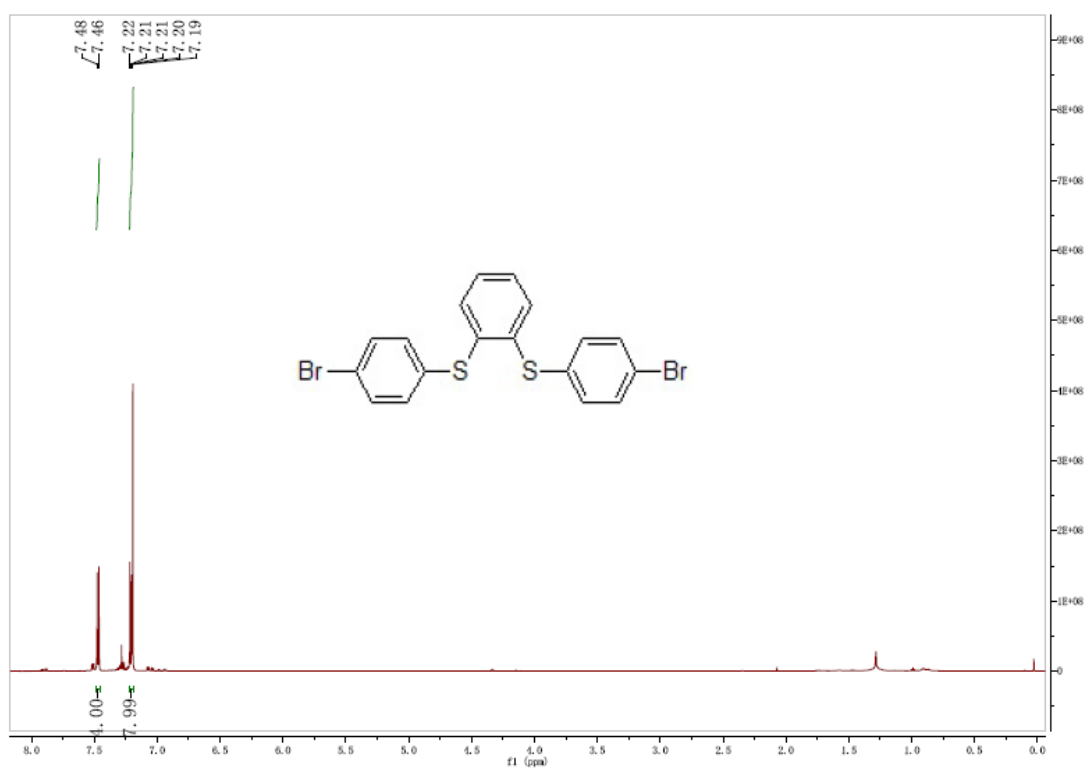
^1H and ^{13}C NMR of **3d** (600 MHz apparatus)



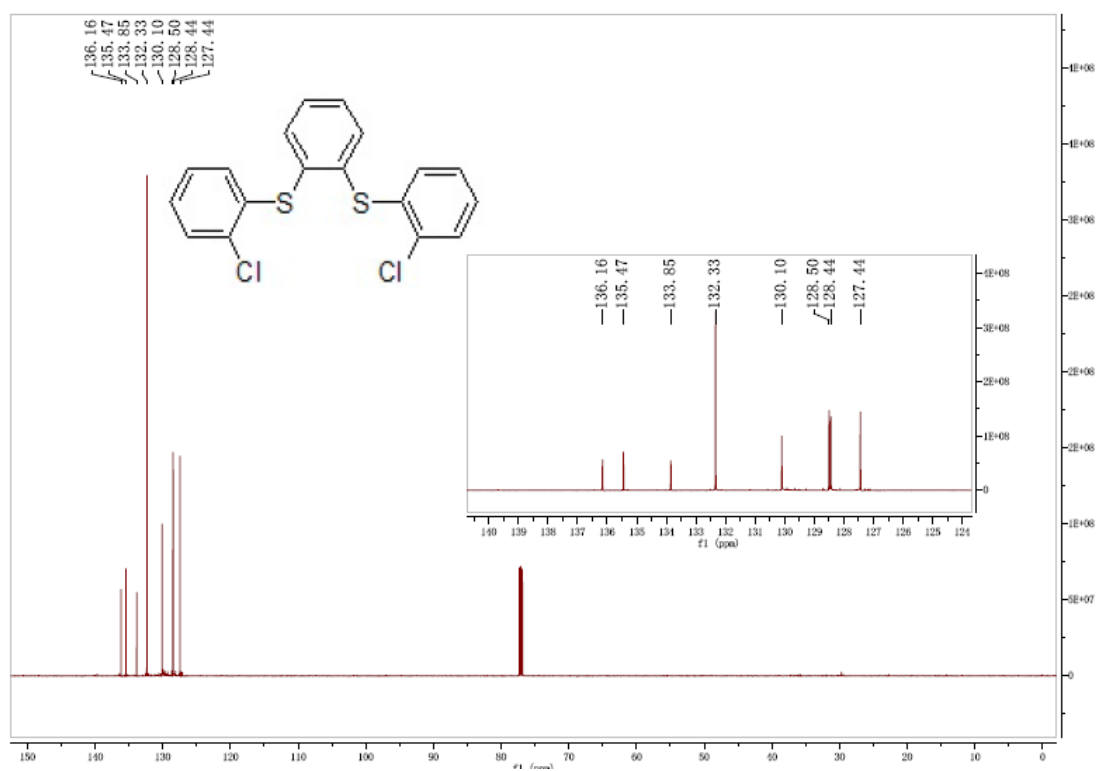
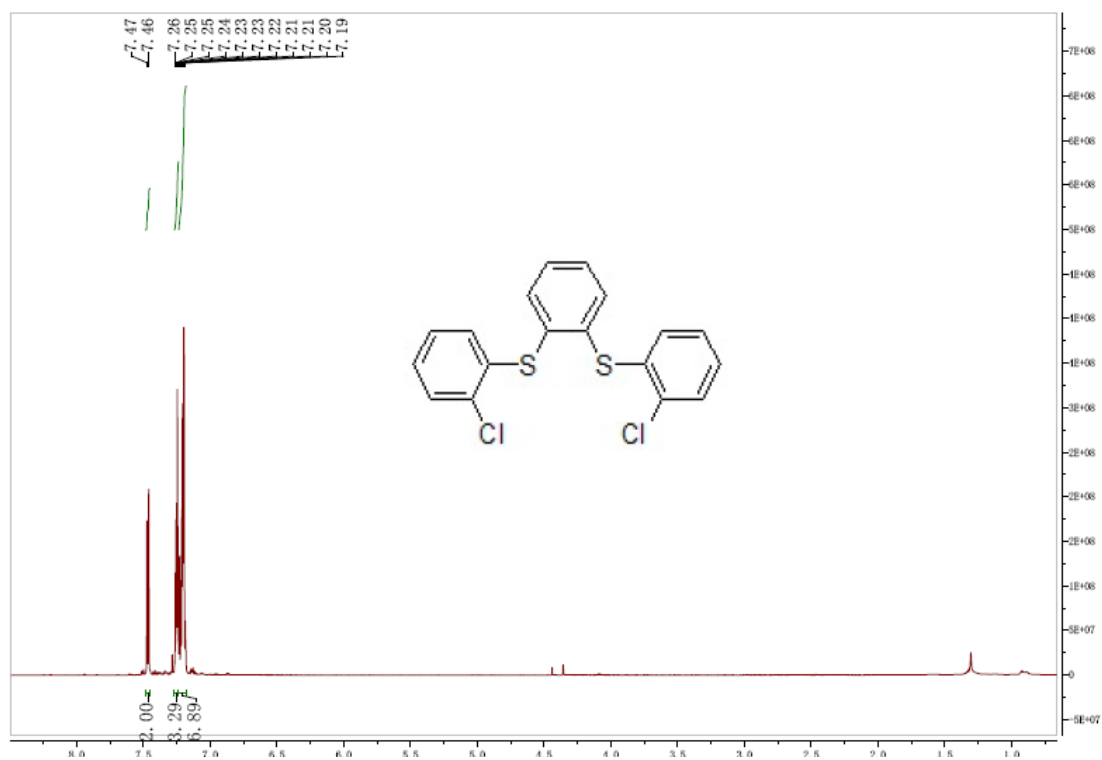
^1H and ^{13}C NMR of **3e** (600 MHz apparatus)



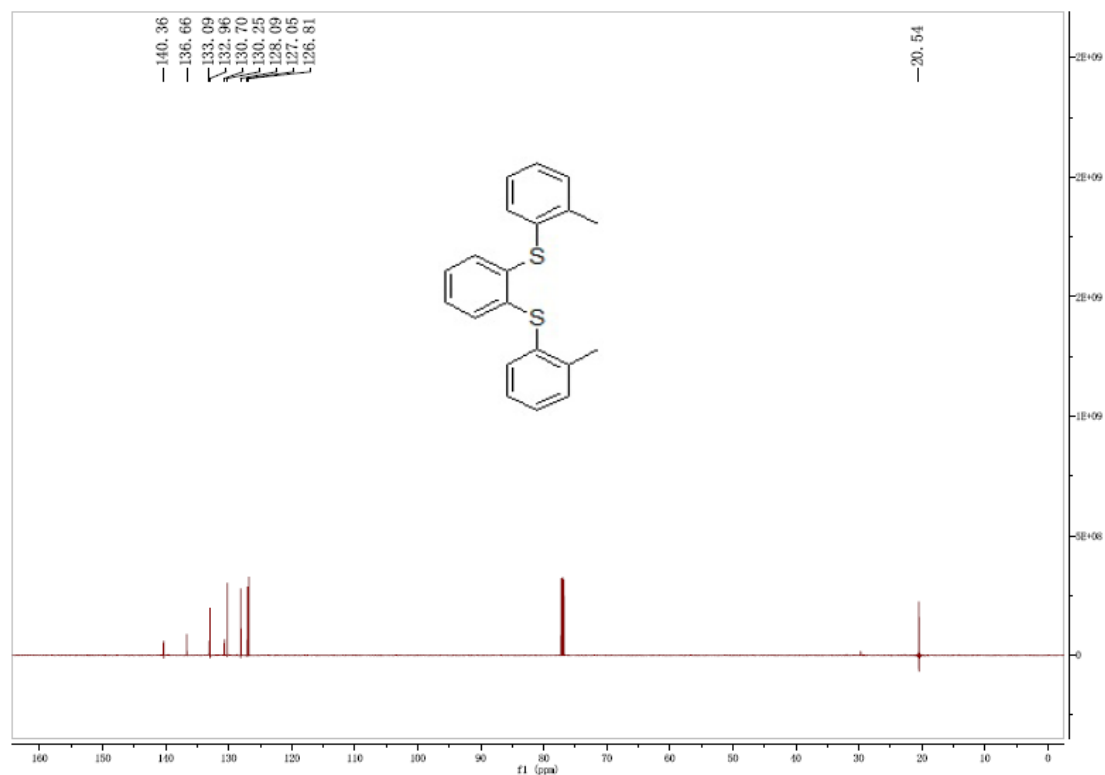
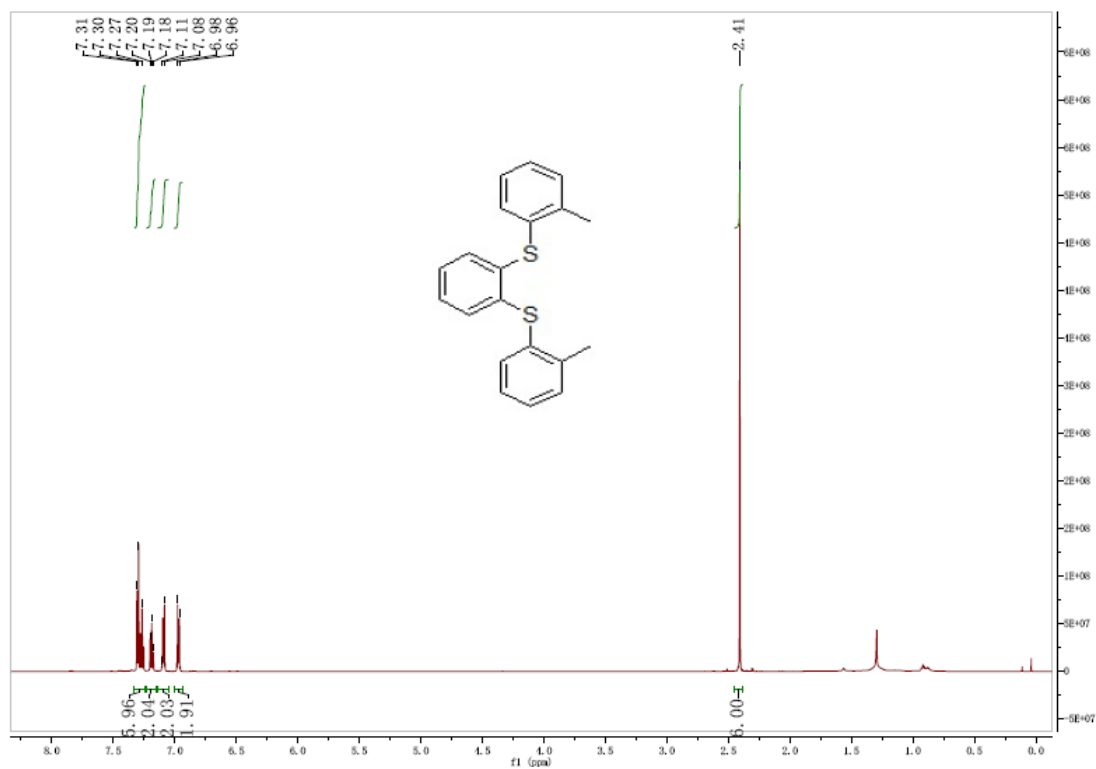
^1H and ^{13}C NMR of **3f** (600 MHz apparatus)



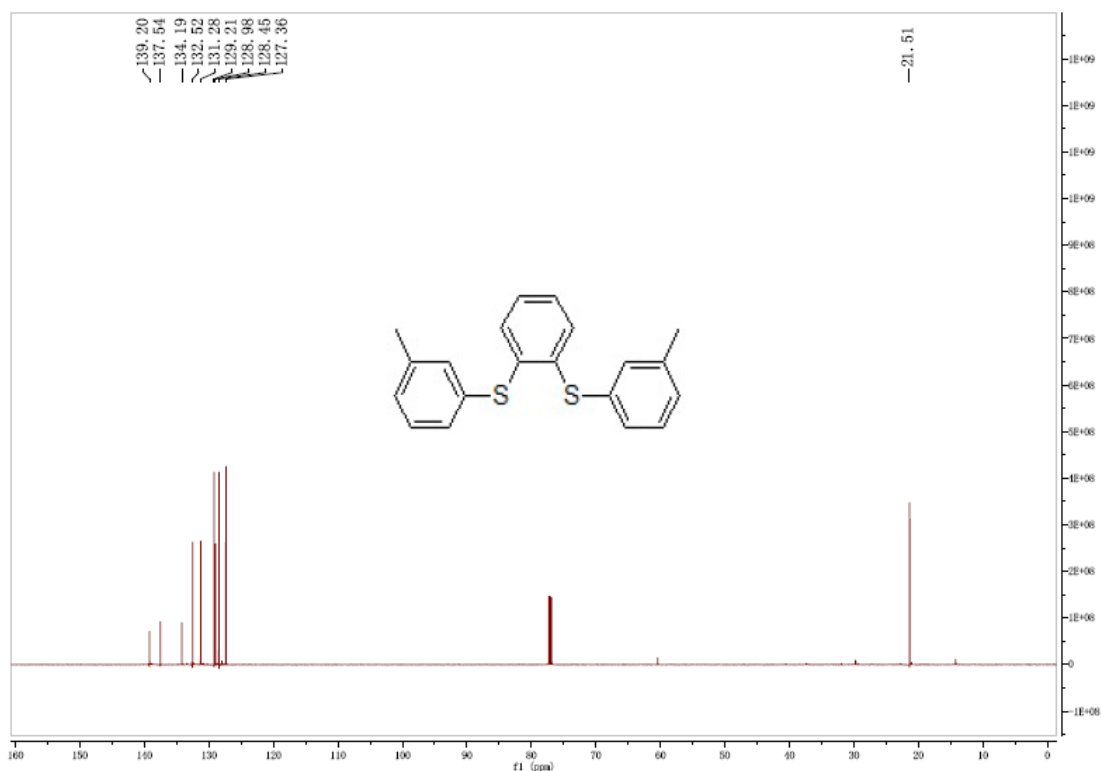
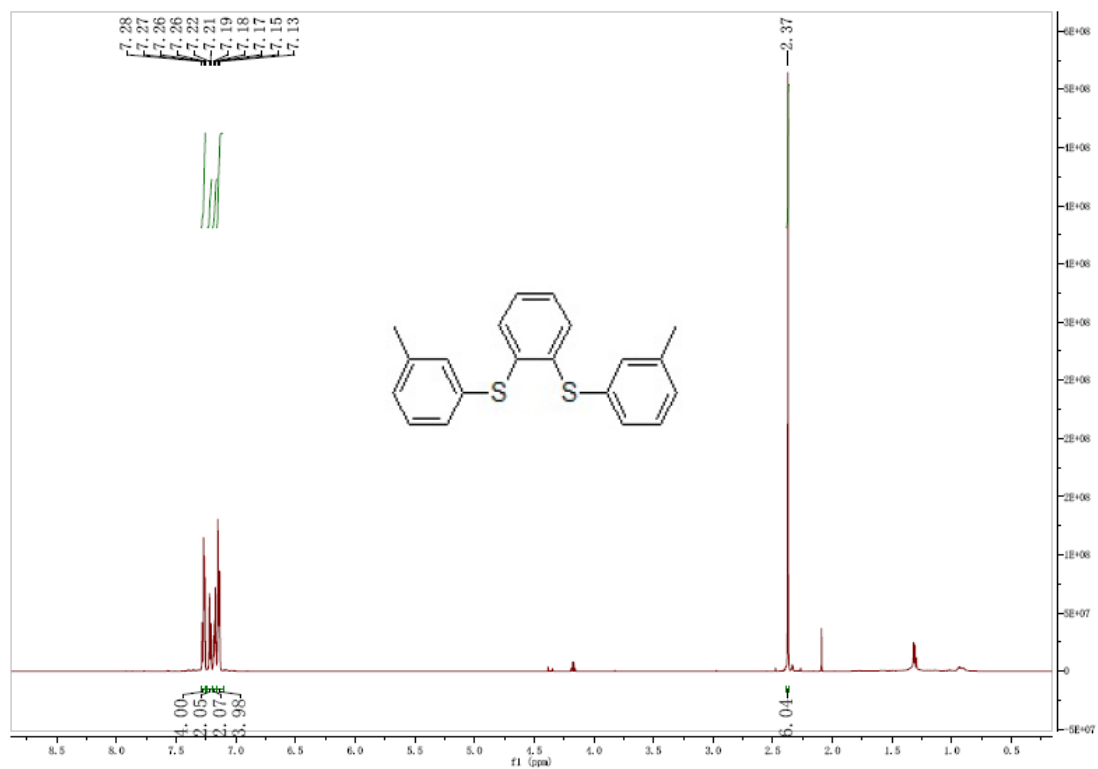
^1H and ^{13}C NMR of **3g** (600 MHz apparatus)



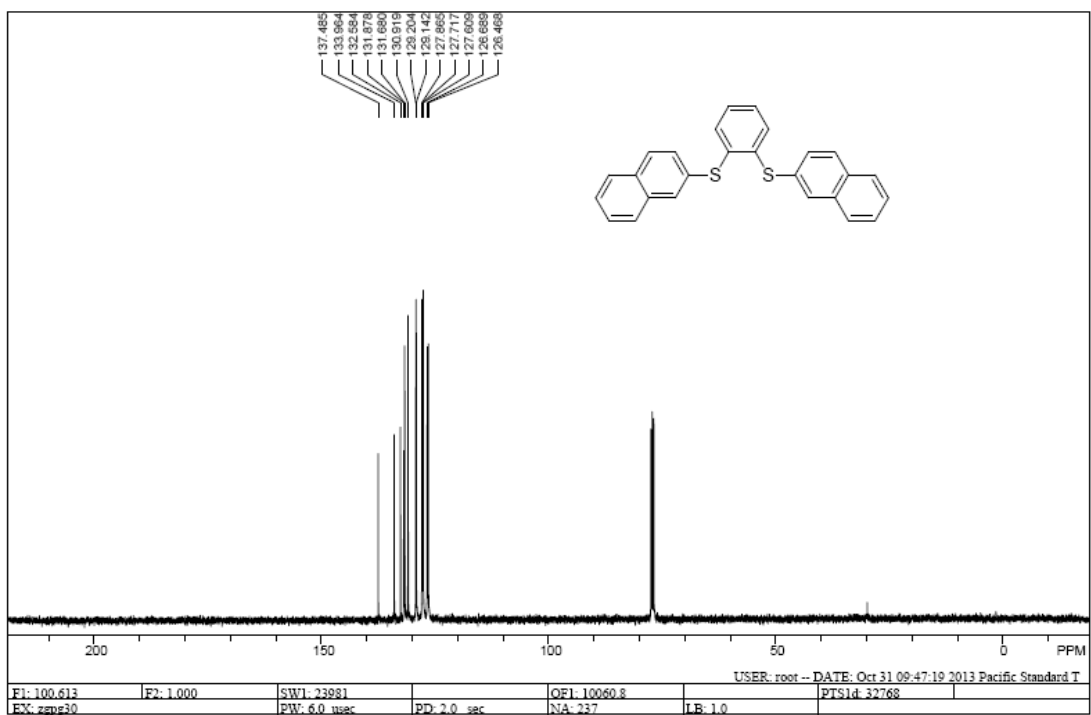
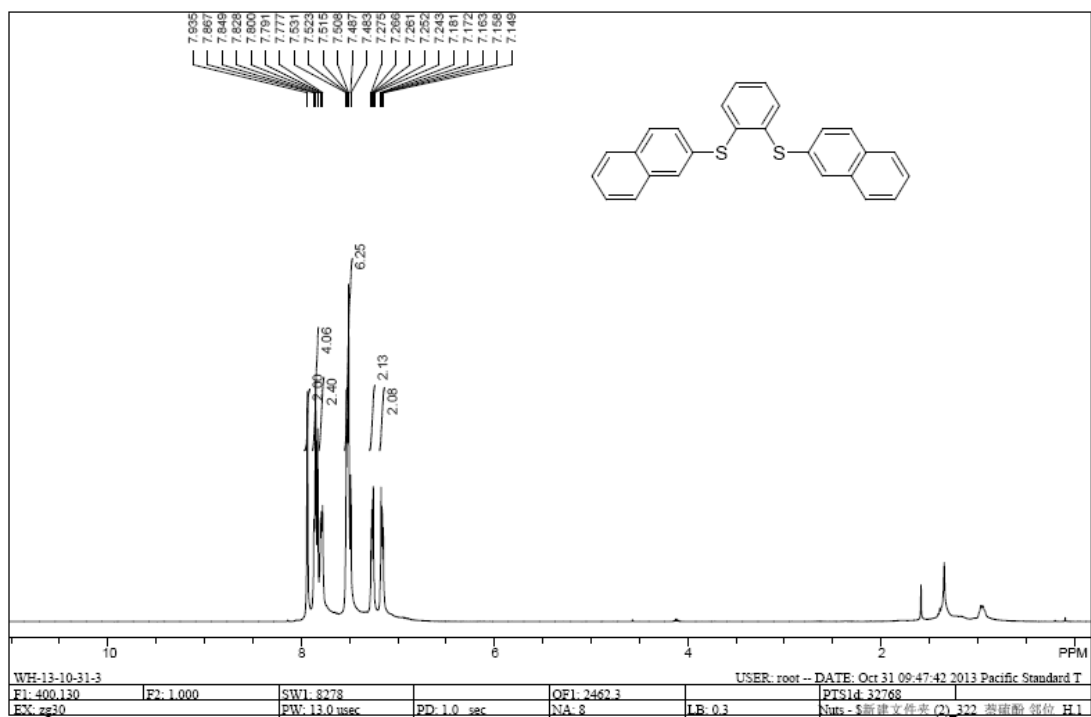
^1H and ^{13}C NMR of **3h** (600 MHz apparatus)



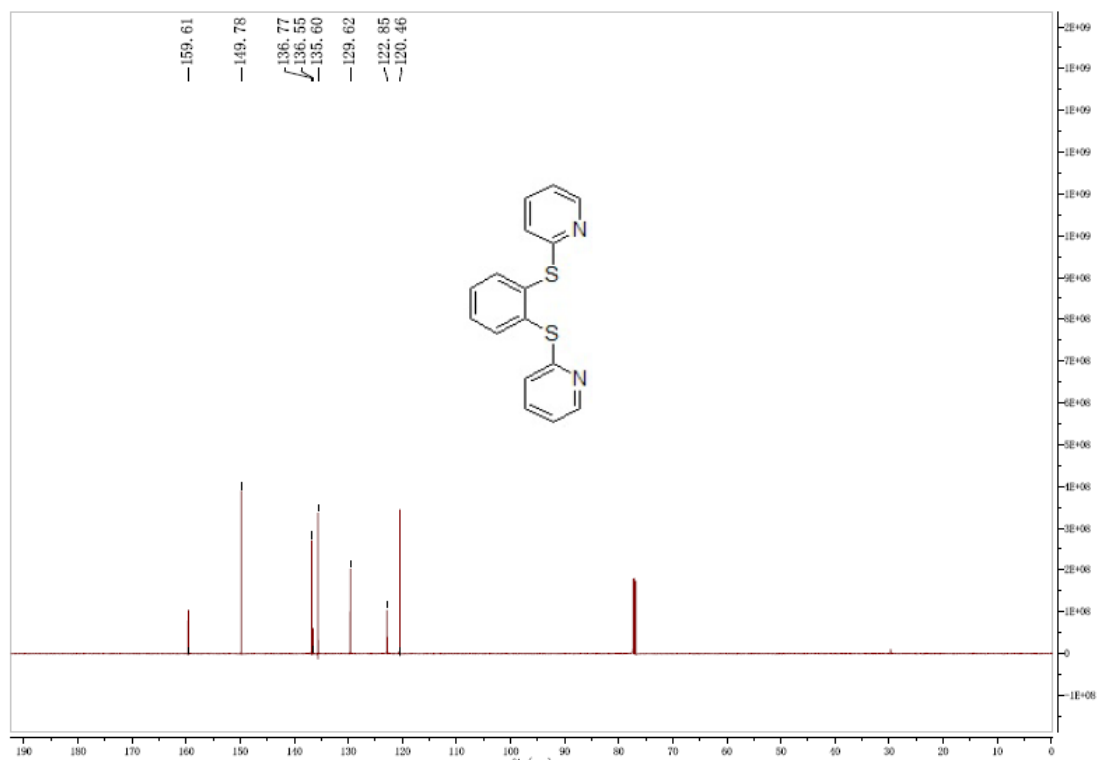
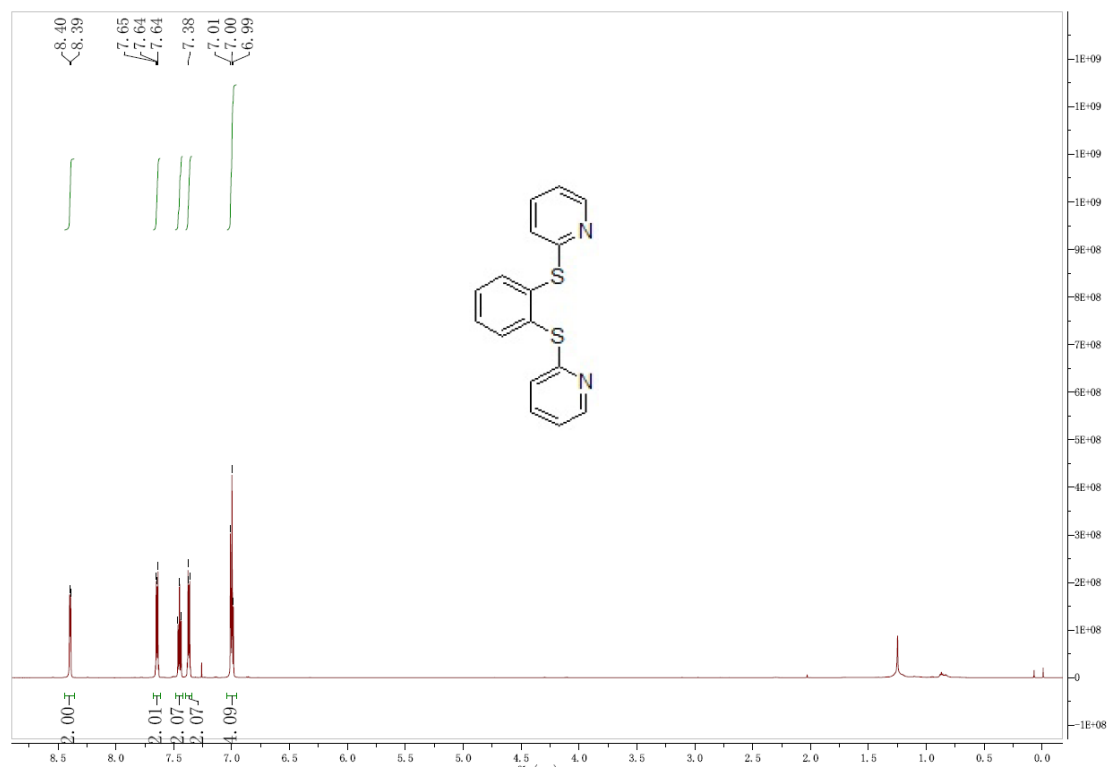
^1H and ^{13}C NMR of **3i** (600 MHz apparatus)



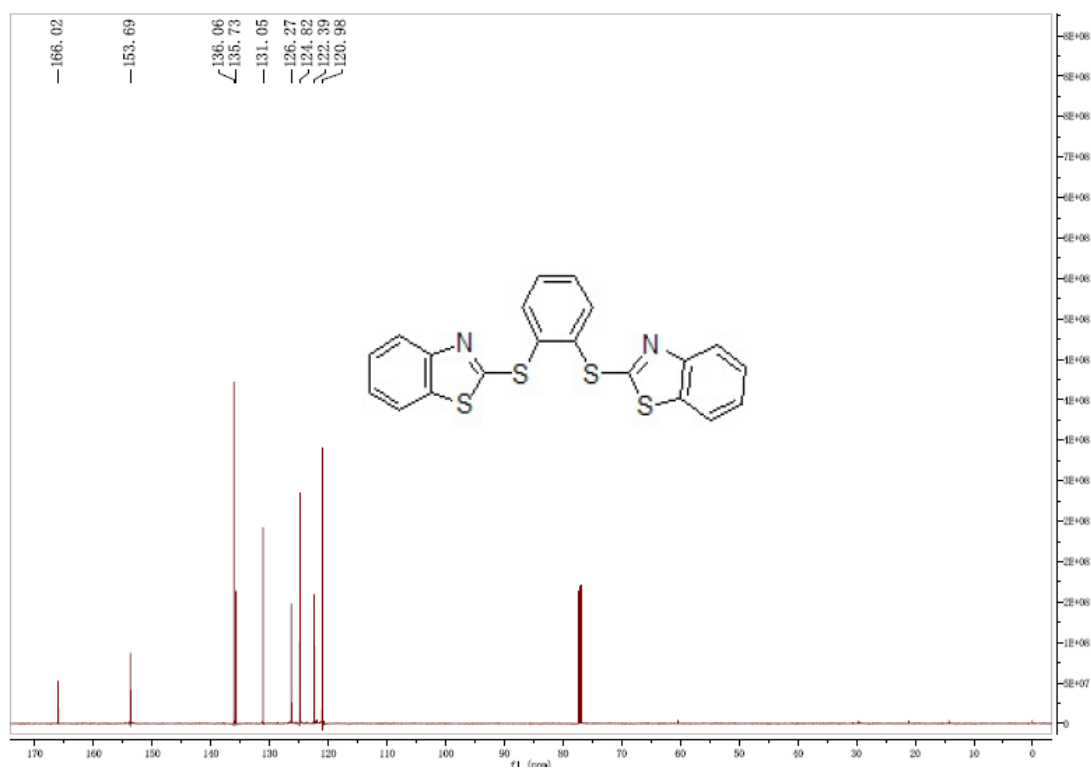
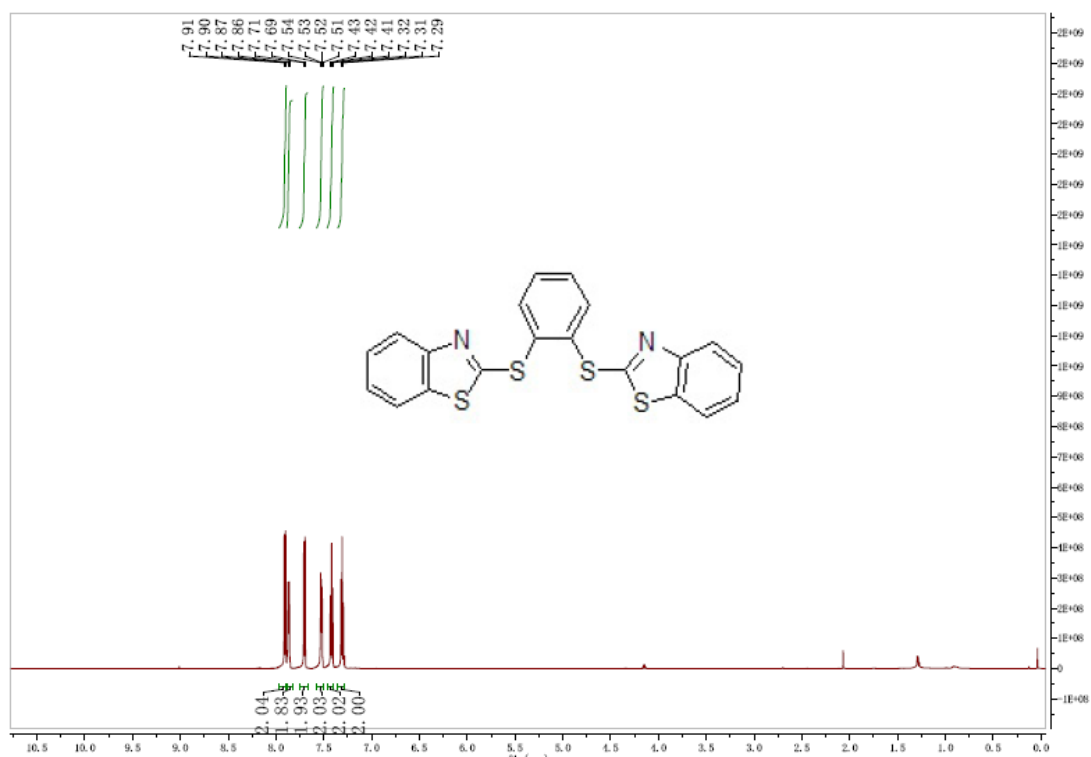
^1H and ^{13}C NMR of **3j** (400 MHz apparatus)



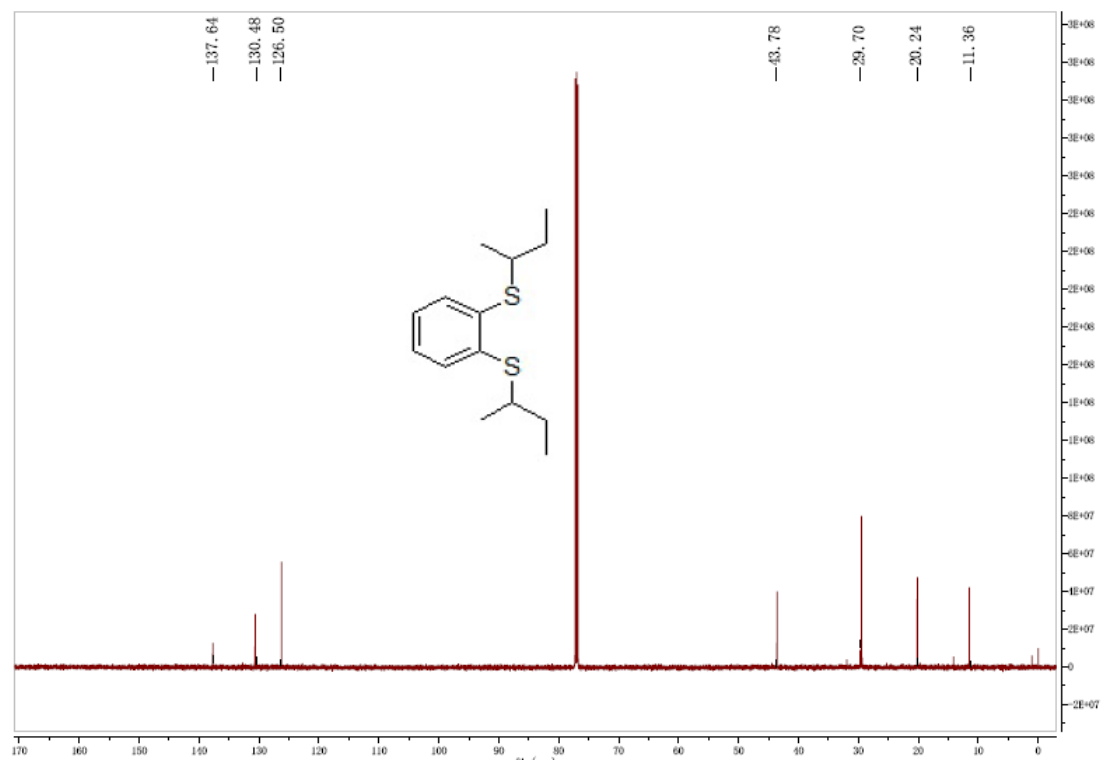
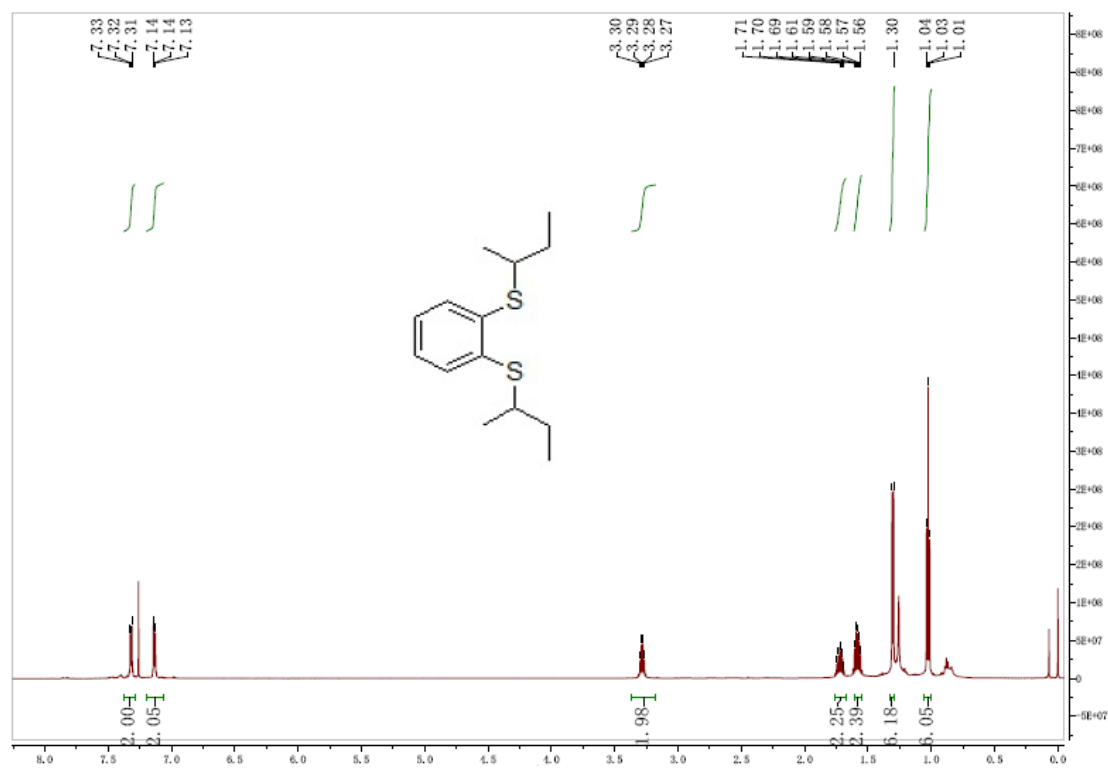
^1H and ^{13}C NMR of **3k** (600 MHz apparatus)



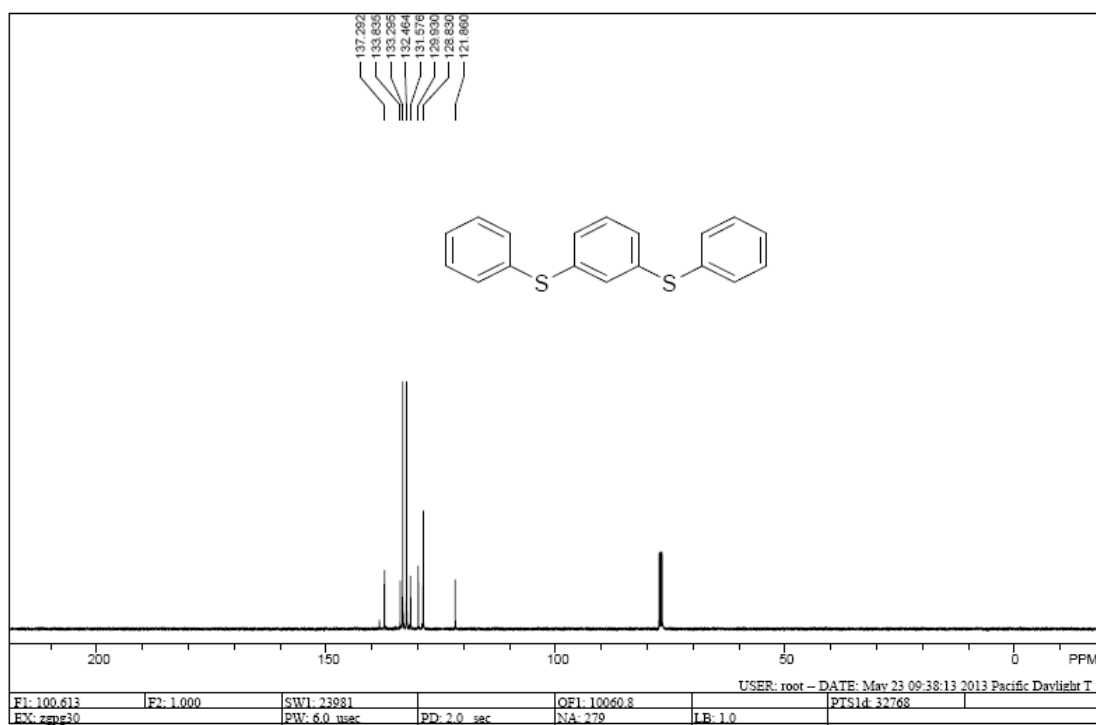
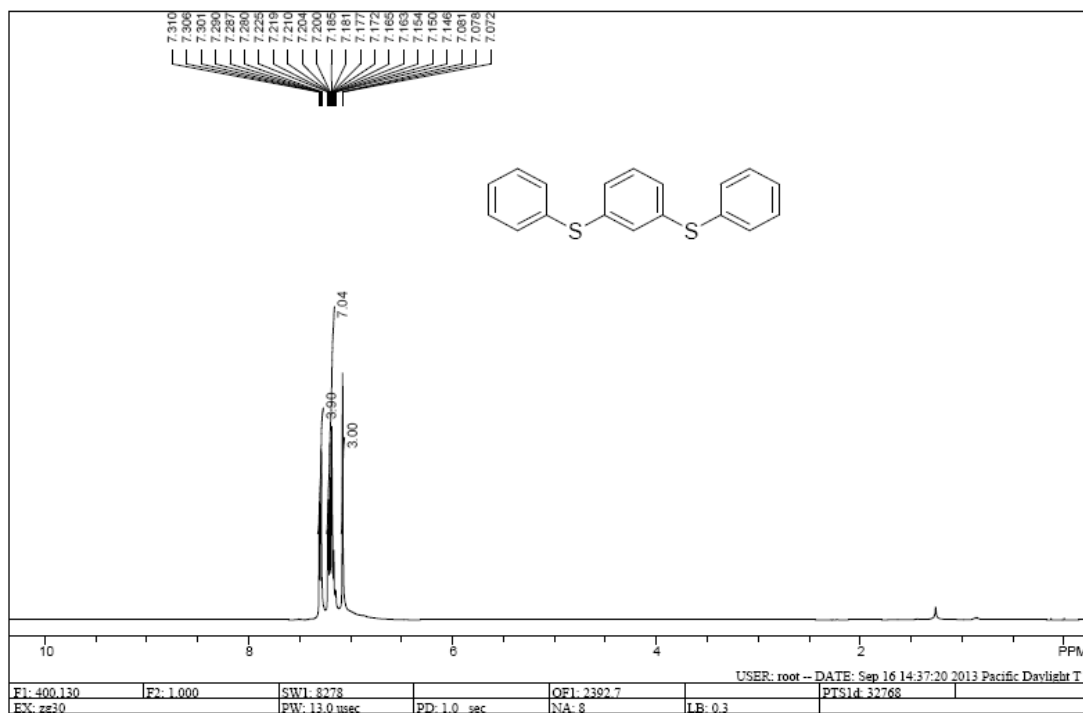
^1H and ^{13}C NMR of **31** (600 MHz apparatus)



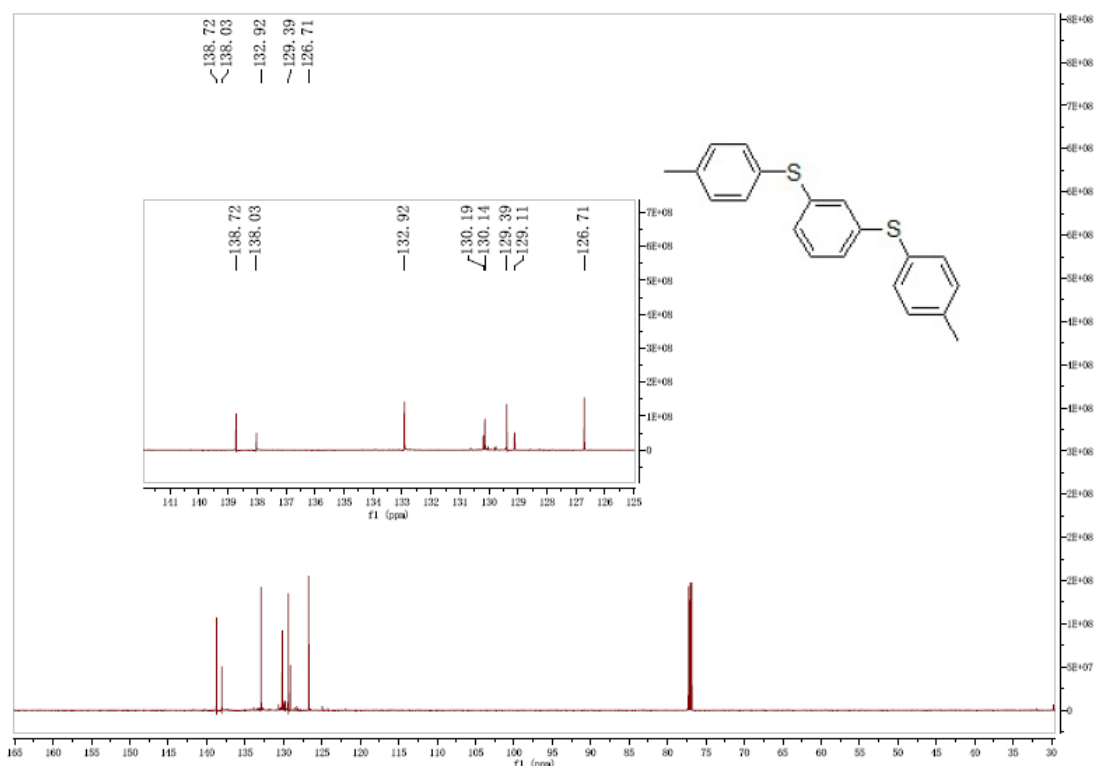
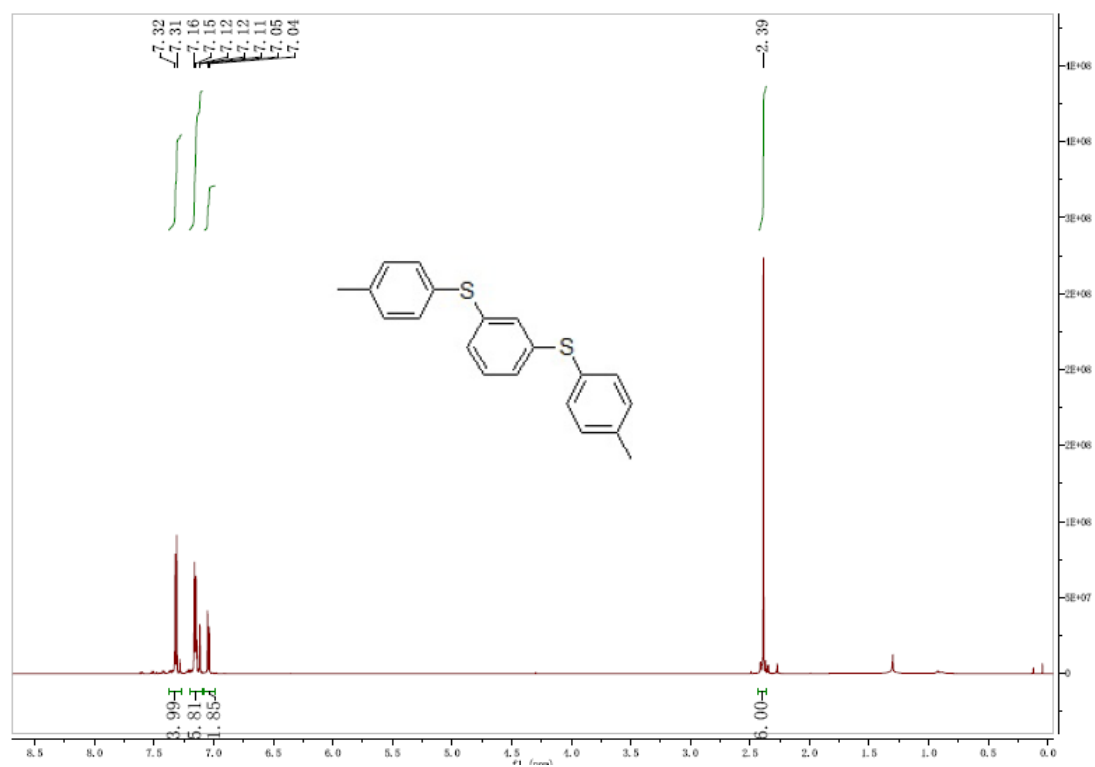
^1H and ^{13}C NMR of **3m** (600 MHz apparatus)



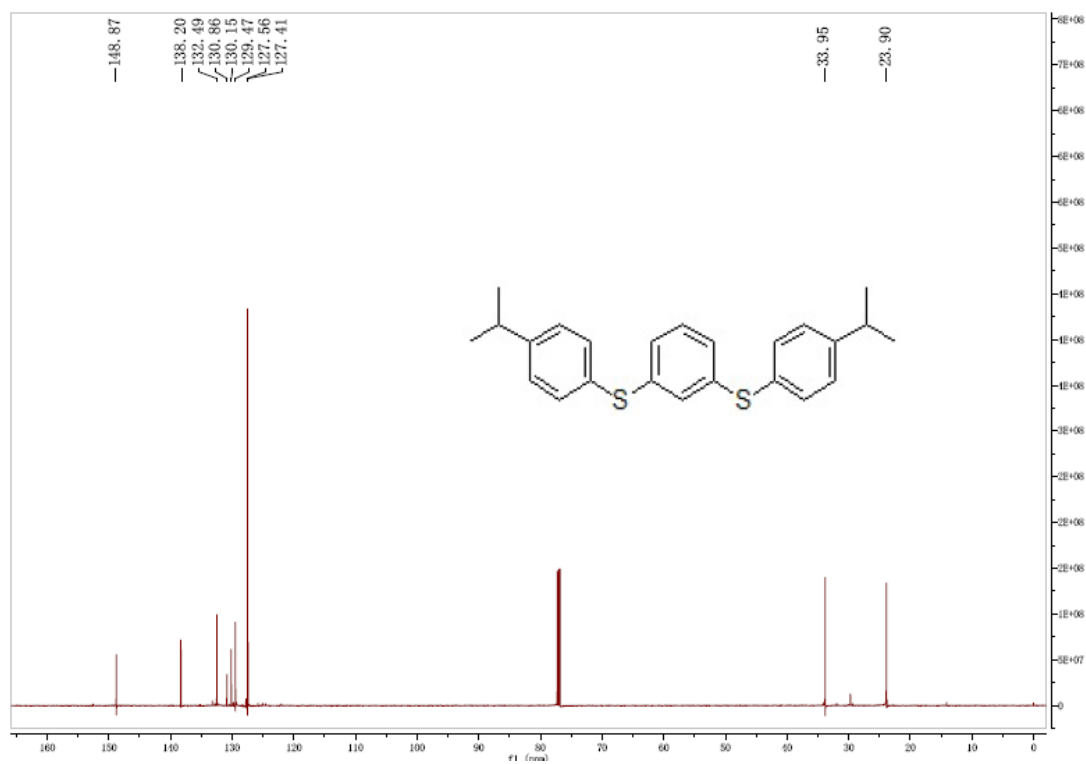
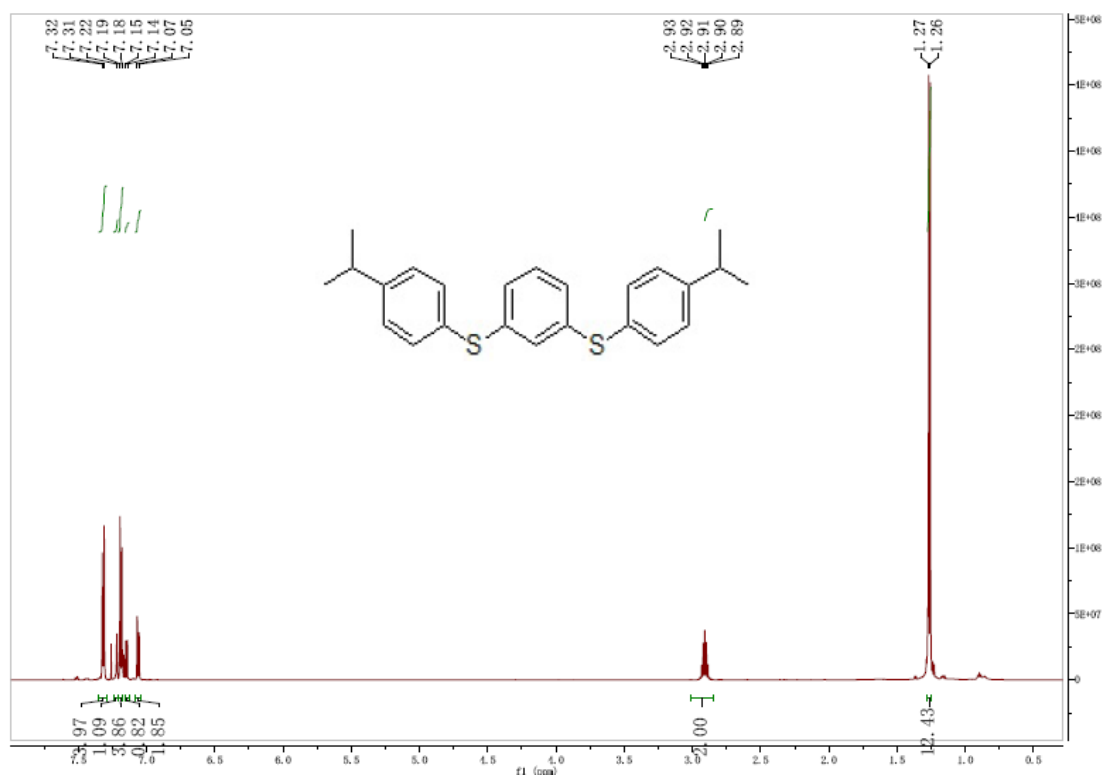
^1H and ^{13}C NMR of **3n** (400 MHz apparatus)



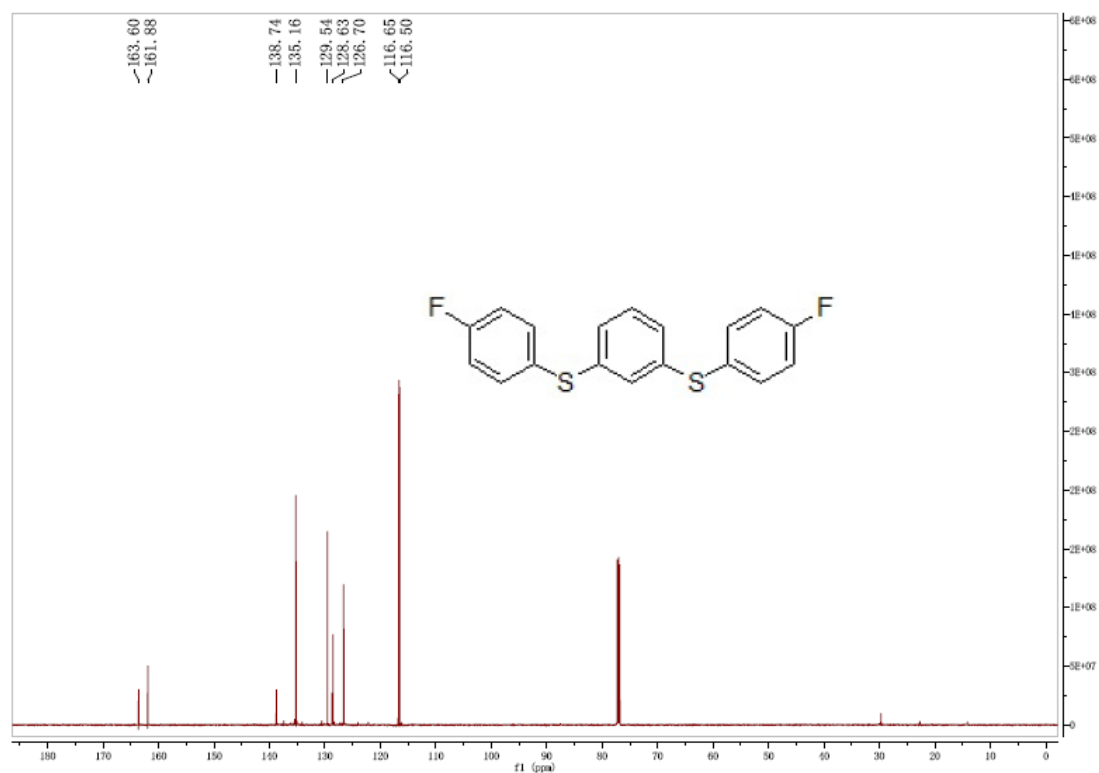
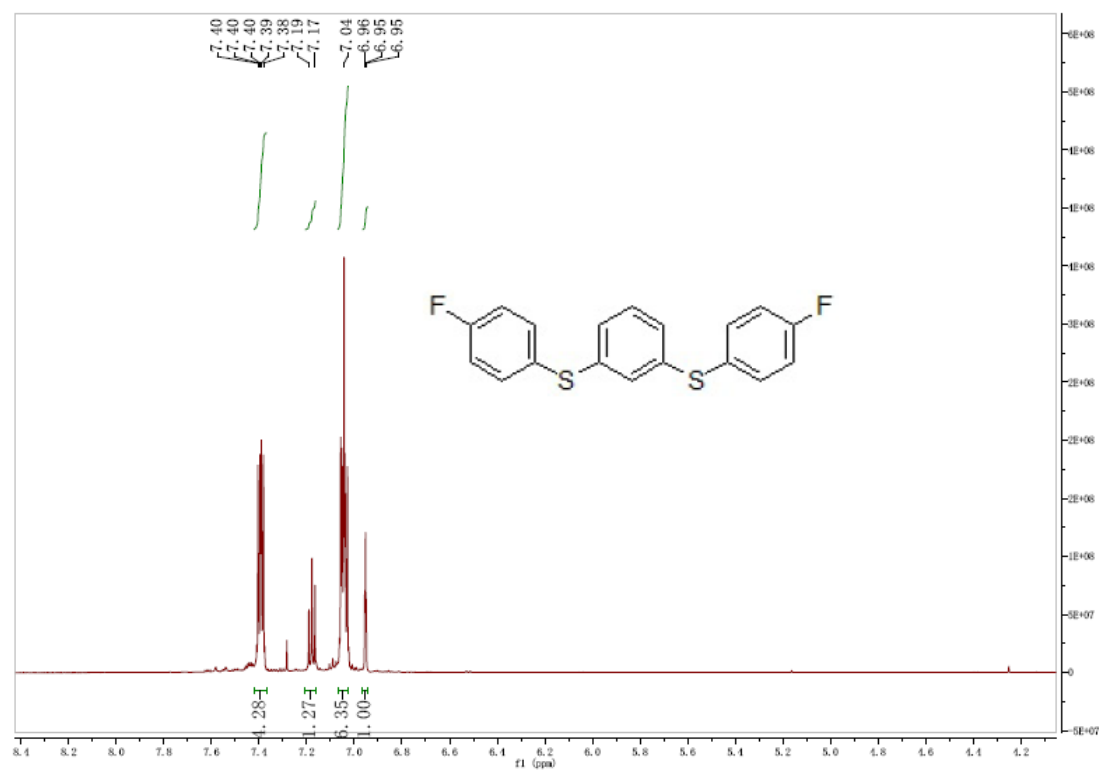
^1H and ^{13}C NMR of **3o** (600 MHz apparatus)



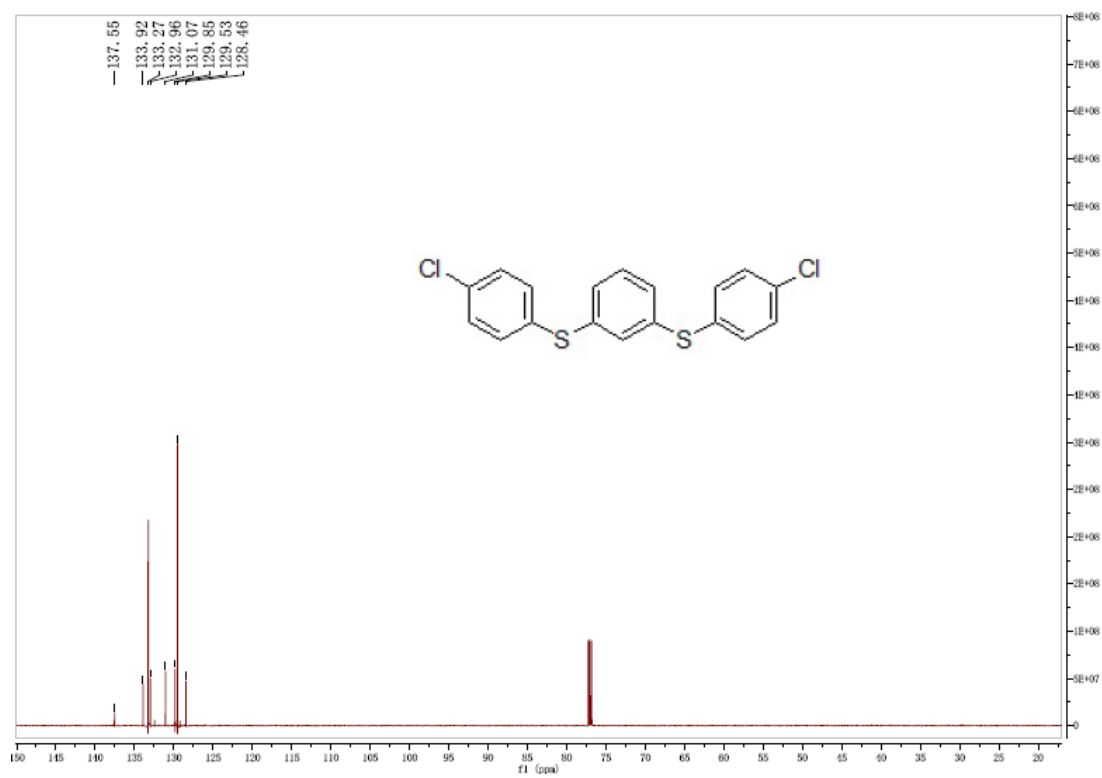
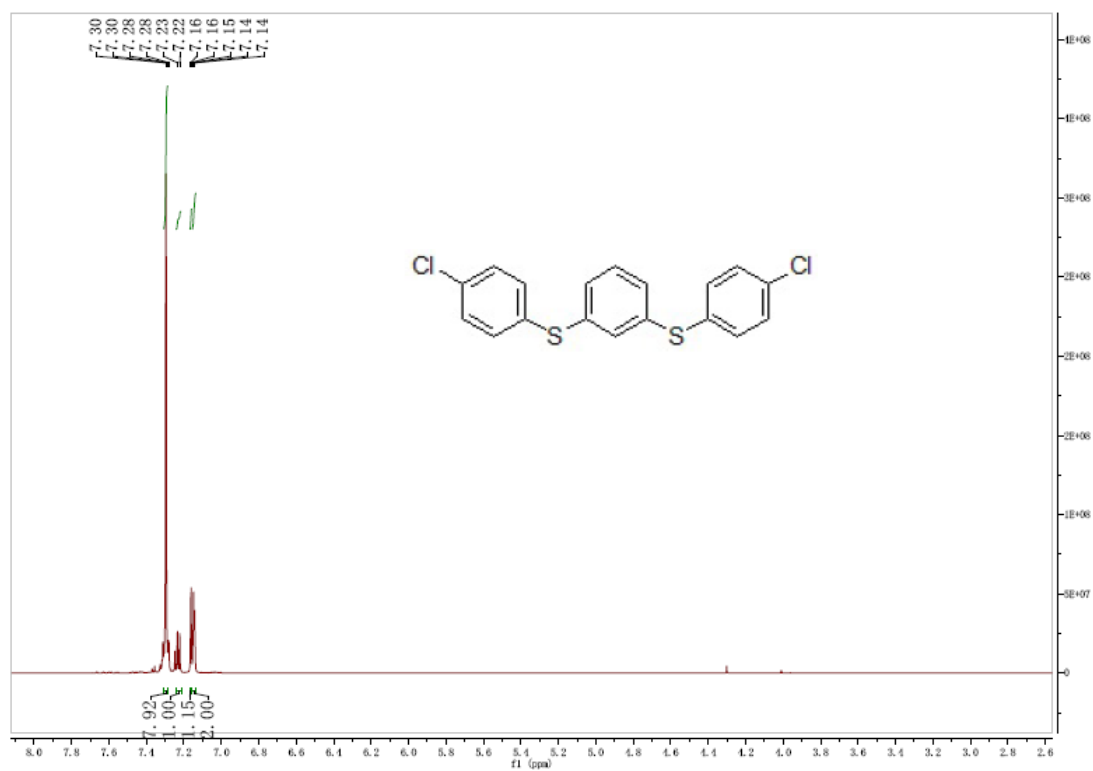
^1H and ^{13}C NMR of **3p** (600 MHz apparatus)



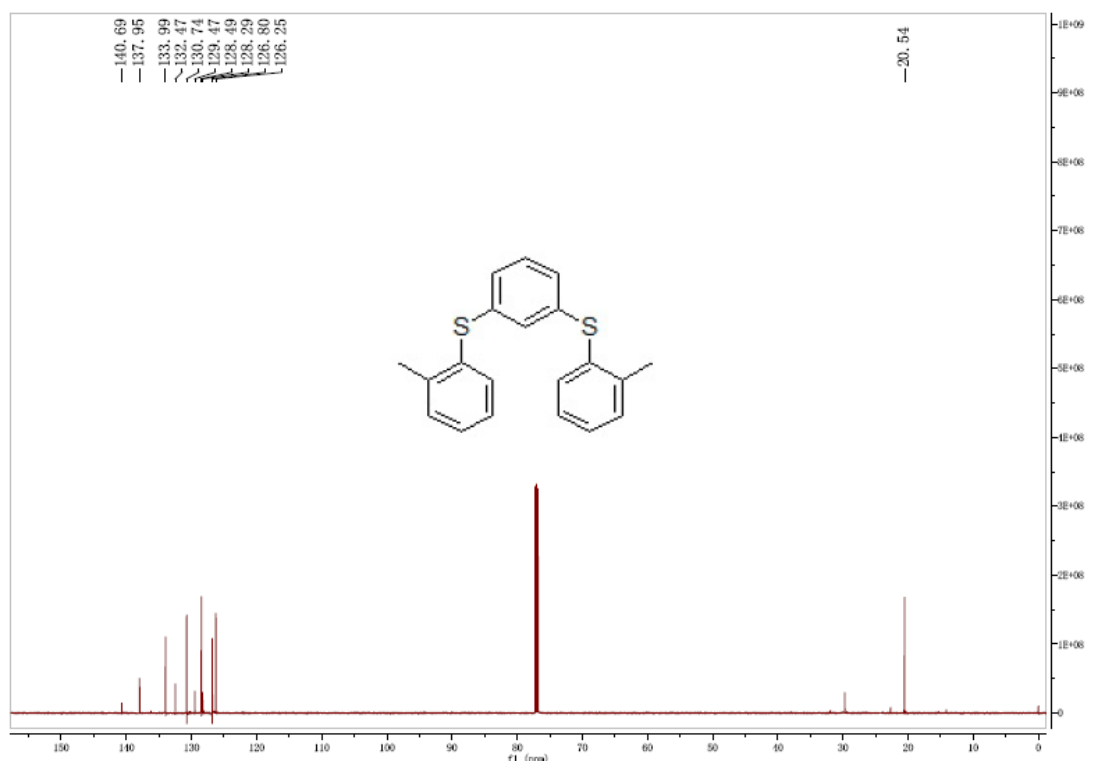
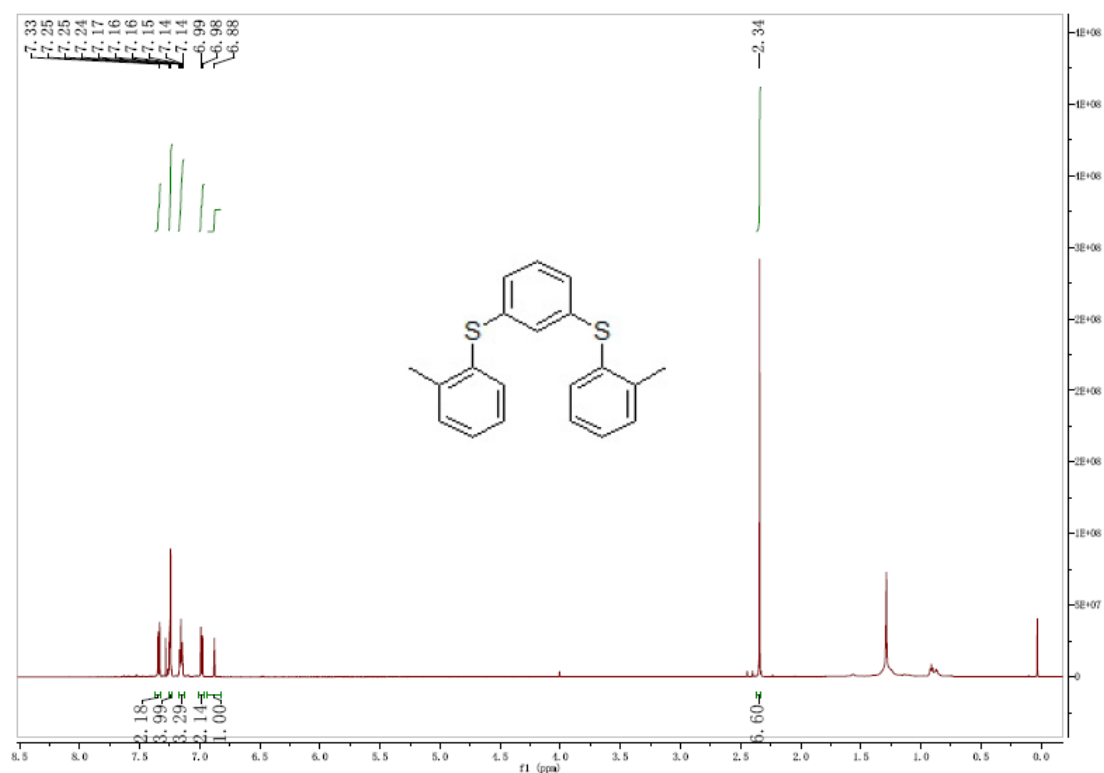
^1H and ^{13}C NMR of **3q** (600 MHz apparatus)



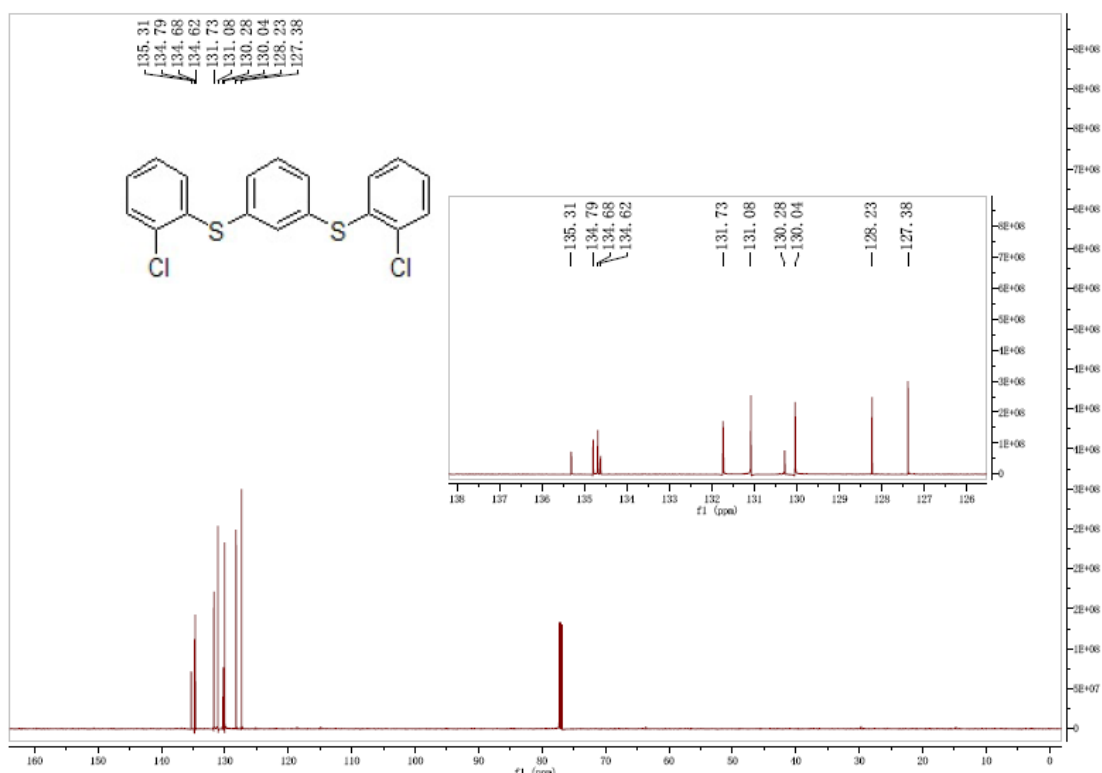
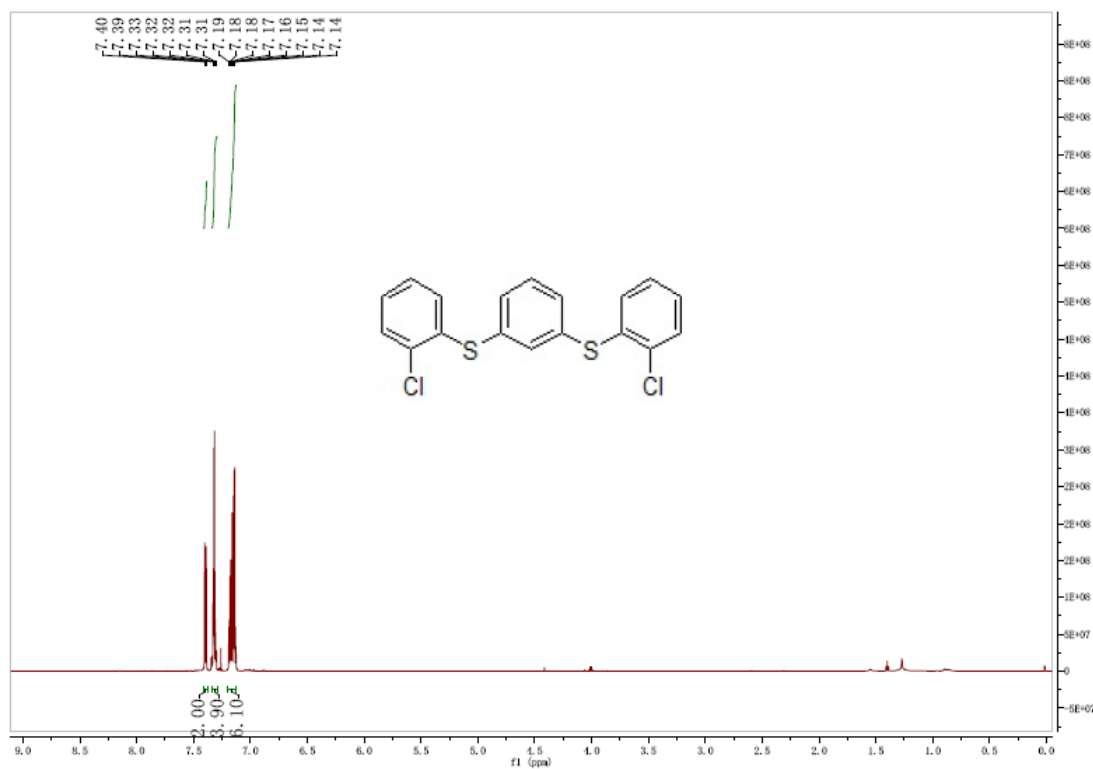
^1H and ^{13}C NMR of **3r** (600 MHz apparatus)



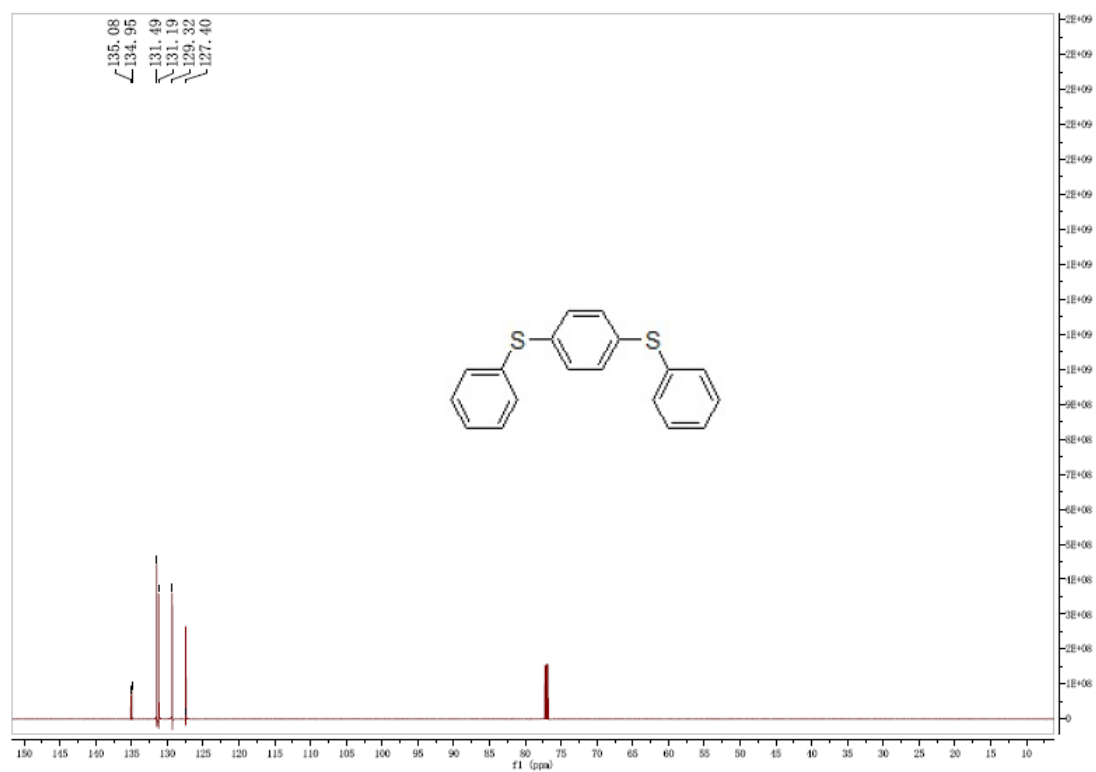
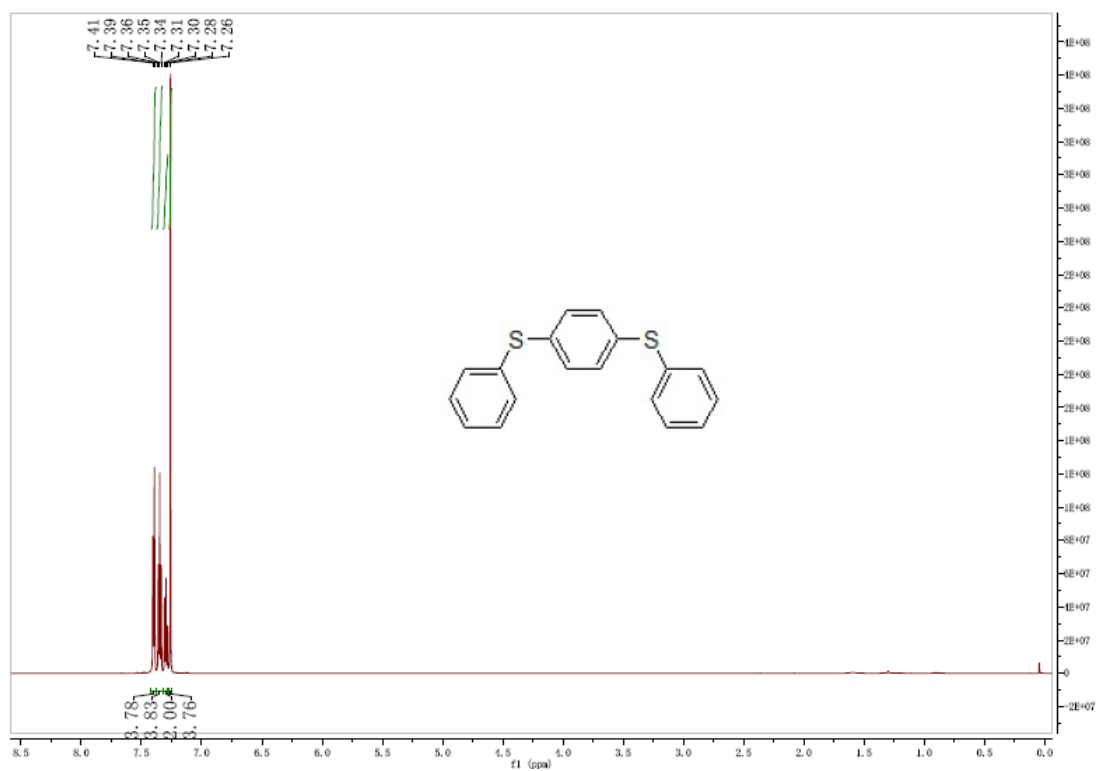
^1H and ^{13}C NMR of **3s** (600 MHz apparatus)



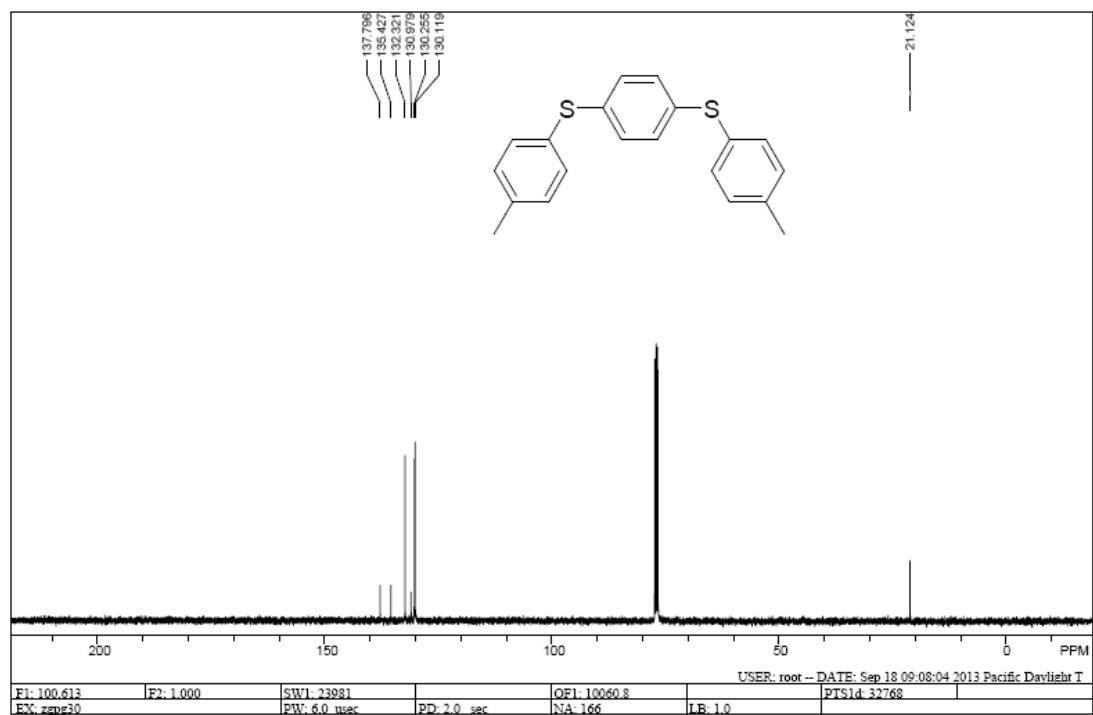
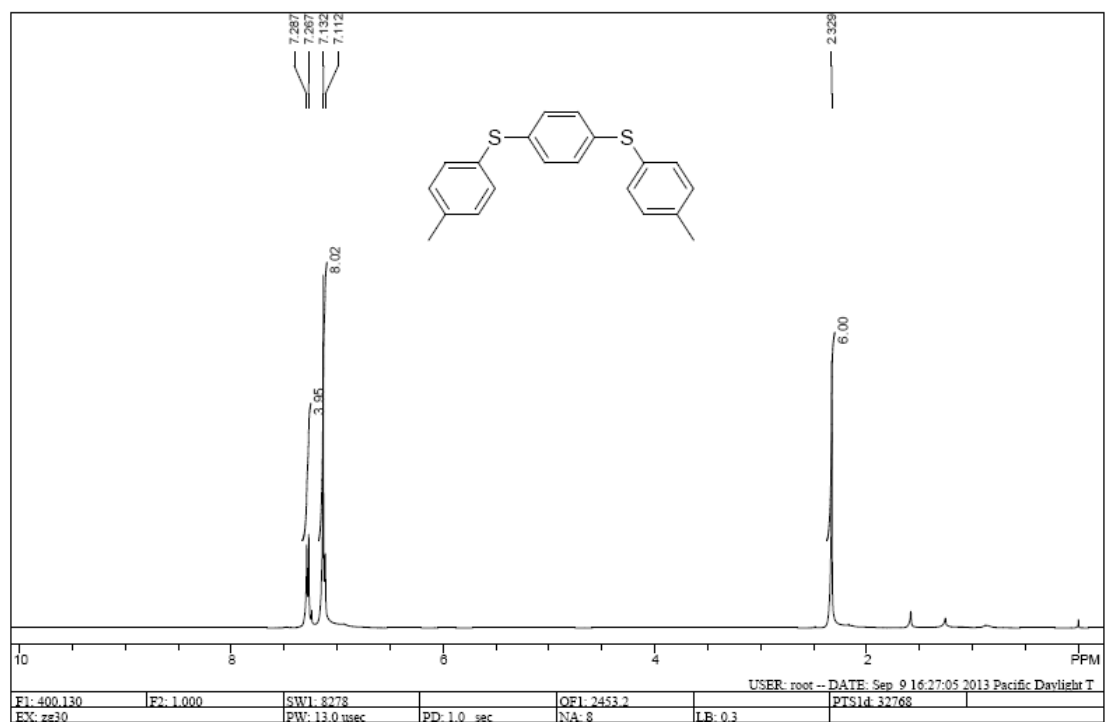
^1H and ^{13}C NMR of **3t** (600 MHz apparatus)



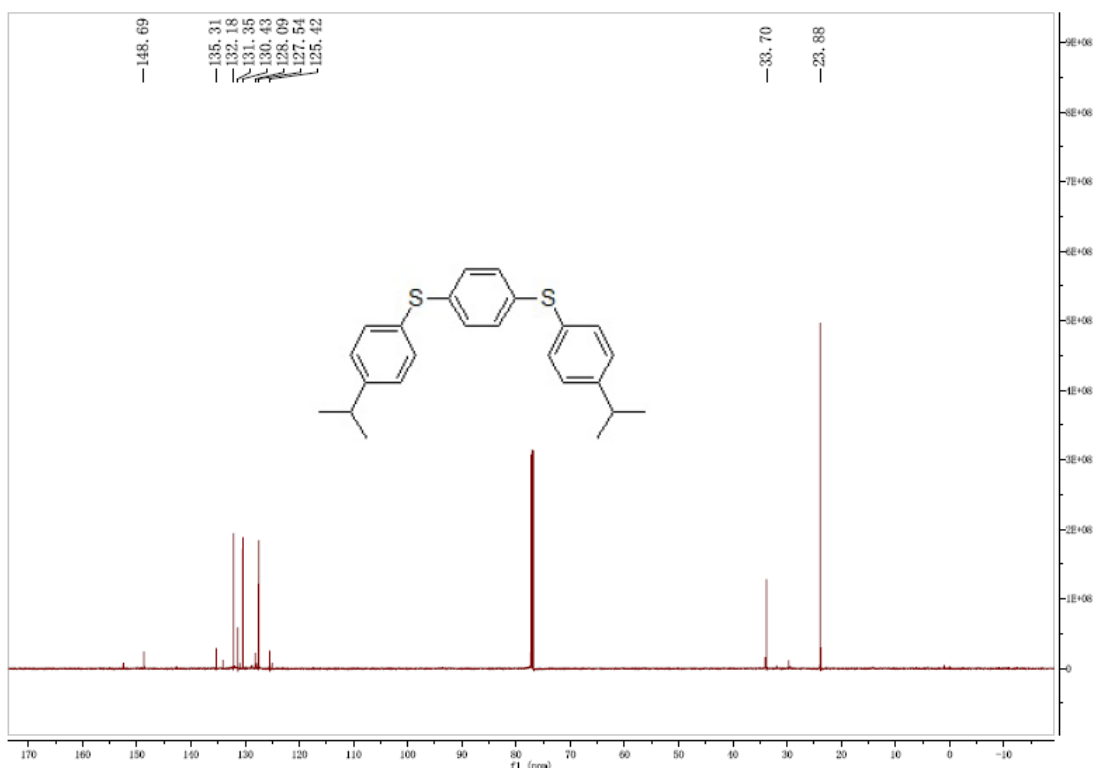
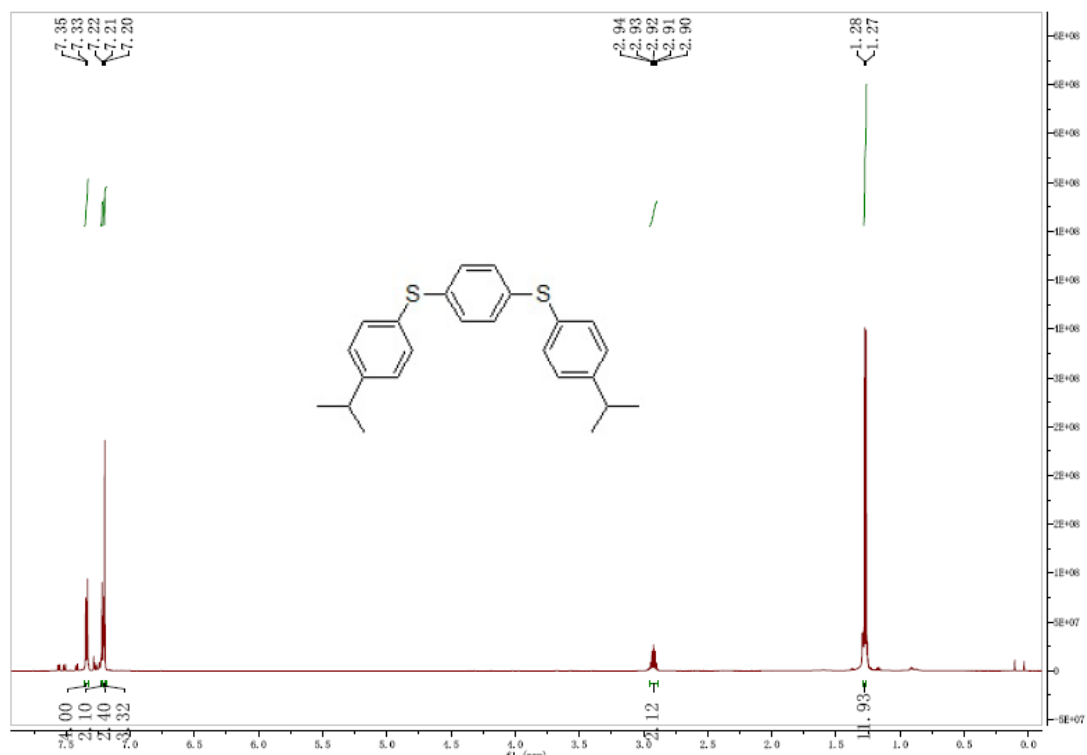
^1H and ^{13}C NMR of **3u** (600 MHz apparatus)



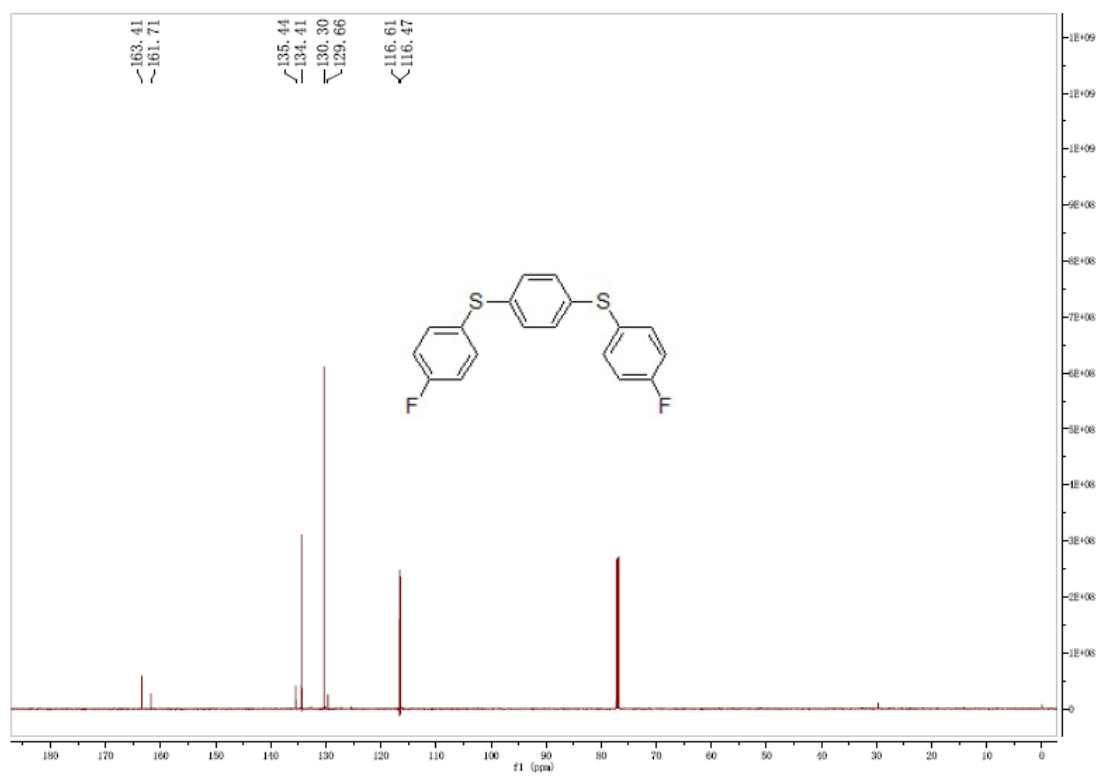
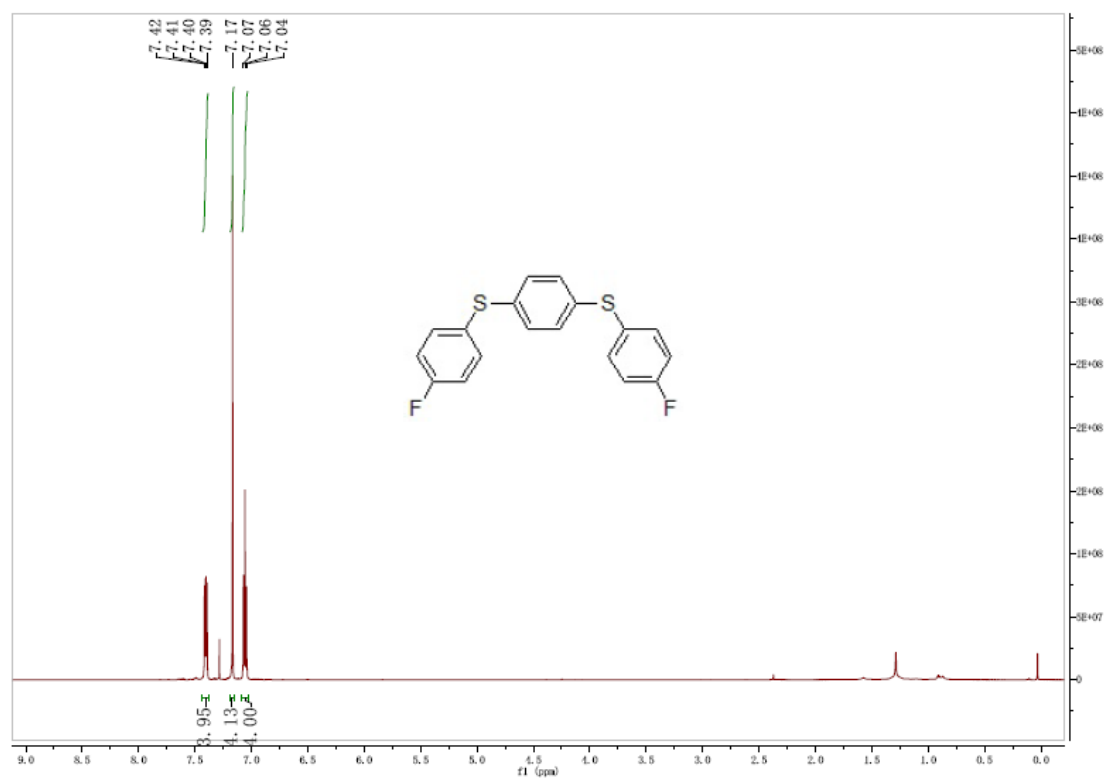
^1H and ^{13}C NMR of **3v** (400 MHz apparatus)



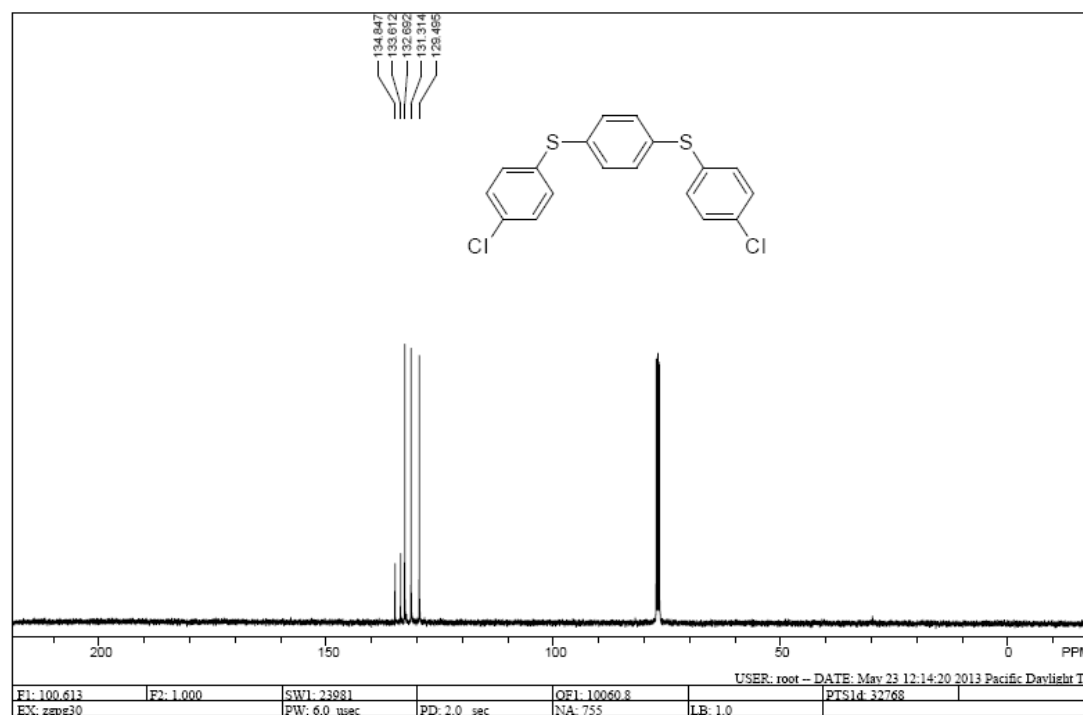
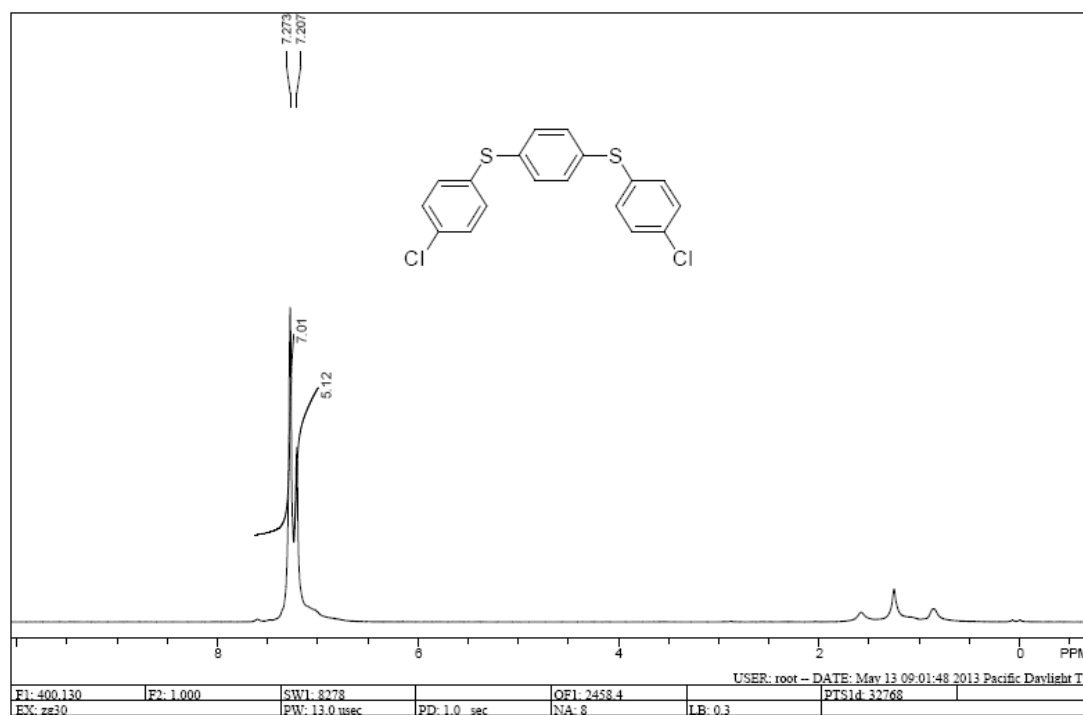
^1H and ^{13}C NMR of **3w** (600 MHz apparatus)



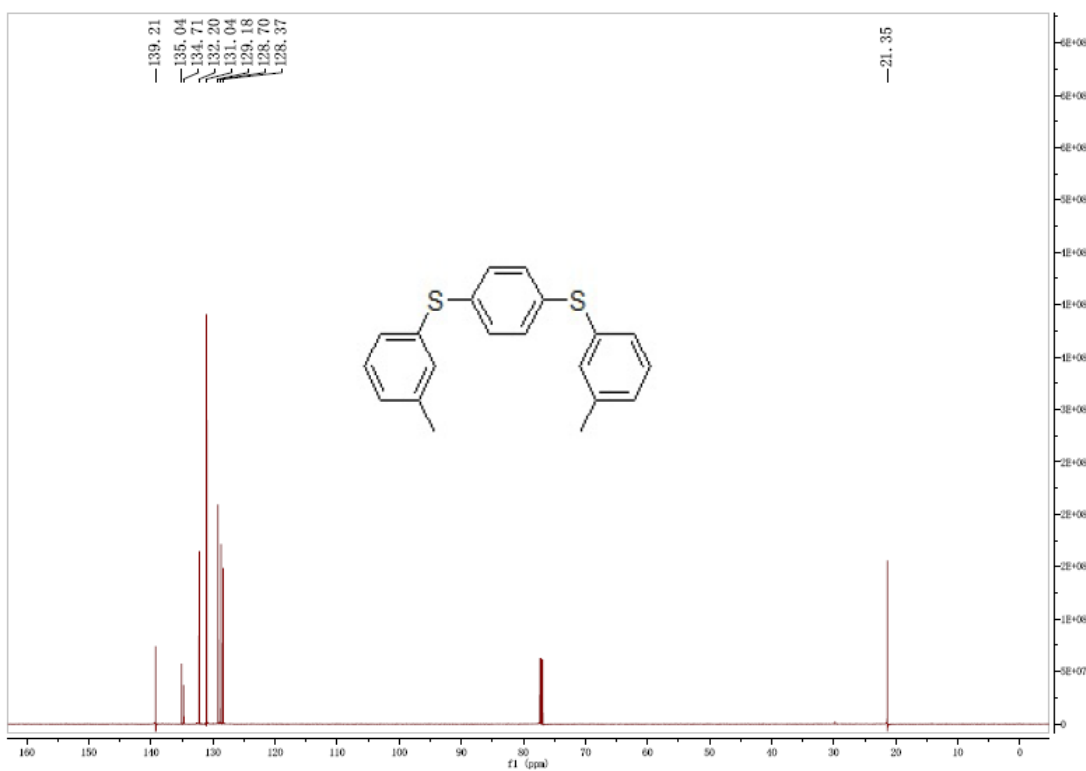
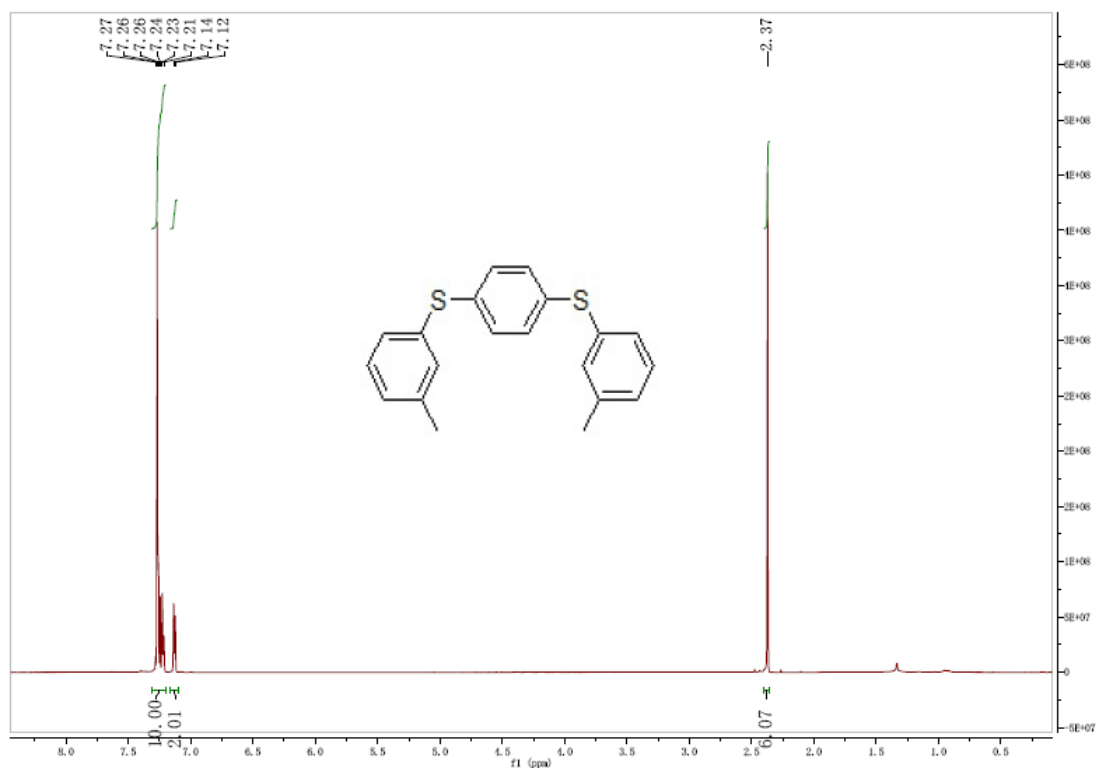
^1H and ^{13}C NMR of **3x** (600 MHz apparatus)



^1H and ^{13}C NMR of **3w** (400 Mz apparatus)



^1H and ^{13}C NMR of **3z**



^1H and ^{13}C NMR of **3bf** (recorded in 400 MHz apparatus)

