Supporting Information

Catalytic arylsulfonyl radical-triggered 1,5-enyne-bicyclizations and hydrosulfonylation of α,β-conjugates

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**General Information**

Unless otherwise noted all starting materials were either known compounds or were obtained from commercial sources and used without purification. Melting points were determined in open capillaries. IR spectra were taken on a FT-IR-Tensor 27 spectrometer. $^1$H NMR ($^{13}$C NMR) spectra were measured on a Bruker DPX 400 MHz spectrometer in CDCl$_3$ (or DMSO-d$_6$) with chemical shift (δ) given in ppm relative to TMS as internal standard [(s = singlet, d = doublet, t = triplet, brs = broad singlet, m = multiplet), coupling constant (Hz)]. HRMS (APCI-TOF) was determined by using microTOF-Q II HRMS/MS instrument (BRUKER). X-Ray crystallographic analysis was performed with a Siemens SMART CCD and a Siemens P4 diffractometer.

![Fig 1, X-ray Structure of 3f](image-url)
Table 1 Optimization of the reaction conditions

| Entry | Oxidant (equiv) | Catalyst (mol%) | Additives (equiv) | Solvent | T (°C) | Yieldb (%) |
|-------|----------------|----------------|------------------|---------|--------|------------|
| 1     | BPO(4.0)       | TBAI (20)      | -                | MeCN    | 70     | 18         |
| 2     | BPO(4.0)       | TBAI (20)      | -                | DCM     | 70     | 10         |
| 3     | BPO(4.0)       | TBAI (20)      | -                | 1,4-Dioxane | 70 | trace |
| 4     | BPO(4.0)       | TBAI (20)      | -                | Toluene | 70     | 0          |
| 5     | BPO(4.0)       | TBAI (20)      | -                | MeCN    | 100    | 25         |
| 6     | BPO(4.0)       | I2 (15)        | -                | MeCN    | 100    | messy      |
| 7     | BPO(4.0)       | KI (20)        | -                | MeCN    | 100    | messy      |
| 8     | BPO(4.0)       | CuI (20)       | -                | MeCN    | 100    | 16         |
| 9     | BPO(4.0)       | TBAI (20)      | HOAc (1.0)       | MeCN    | 100    | 28         |
| 10    | BPO(4.0)       | TBAI (20)      | L-proline (1.0)  | MeCN    | 100    | 33         |
| 11    | BPO(4.0)       | TBAI (20)      | PivOH (1.0)      | MeCN    | 100    | 35         |
| 12    | BPO(4.0)       | TBAI (20)/ CuI (5) | PivOH (1.0) | MeCN    | 100    | 49         |
| 13    | BPO(4.0)       | TBAI (30)/ Cu(OAc)2 (5) | PivOH (1.0) | MeCN    | 100    | 53         |
| 14    | BPO(4.0)       | TBAI (20)/ Cu(OAc)2 (5) | PivOH (1.0) | MeCN    | 100    | 61         |
| 15    | BPO(4.0)       | TBAI (20)/ Cu(OAc)2 (5) | PivOH (2.0) | MeCN    | 100    | 71         |
| 16    | BPO(4.0)       | TBAI (20)/ Cu(OAc)2 (10) | PivOH (2.0) | MeCN    | 100    | 63         |
| 17    | TBHP (4.0)     | TBAI (20)/ Cu(OAc)2 (5) | PivOH (2.0) | MeCN    | 100    | 64         |
| 18    | DTBP (4.0)     | TBAI (20)/ Cu(OAc)2 (5) | PivOH (2.0) | MeCN    | 100    | trace      |
| 19    | H2O2 (4.0)     | TBAI (20)/ Cu(OAc)2 (5) | PivOH (2.0) | MeCN    | 100    | trace      |
| 20    | BPO(4.0)       | Cu(OAc)2 (5)   | PivOH (2.0)      | MeCN    | 100    | 20         |
| 21    | BPO(4.0)       | TBAI (20)/ Cu(OAc)2 (5) | -                | MeCN    | 100    | 33         |

aReaction conditions: 1,5-conjugated enyne (1a, 0.25 mmol), tosylhydrazide (2a, 0.50 mmol), Oxidant (1.0 mmol), solvent (2.5 mL), 12 h. bIsolated yields based on 1.
Experimental Section

Preparation of Compound 1a:
A mixture of 2-(phenylethynyl)benzaldehyde (5 mmol), p-tolylethanone (0.67 g, 5 mmol), NaOH (0.8 g, 20 mmol) and EtOH (20.0 mL) were added in a 50-mL reaction vial. Then, the mixture was stirred at 0°C for 20 min, and the followed reaction system was stirred at room temperature. After completion of the reaction monitored by TLC, the reaction system was poured into the cold water, and the solid product was collected by Büchner filtration. The desired pure 3-(2-(phenylethynyl)phenyl)-1-(p-tolyl)prop-2-en-1-one (1a) was obtained in 80% yield by recrystallization from 95% EtOH.

Preparation of Compound 3a: 7-Methyl-5-phenyl-11-tosyl-11H-benzo[b]fluoren-10-ol
A mixture of 3-(2-(phenylethynyl)phenyl)-1-(p-tolyl)prop-2-en-1-one (1a, 0.25 mmol, 1.0 equiv.), tosylhydrazide (2a, 0.5 mmol, 2.0 equiv.), TBAI (0.05 mmol, 0.2 equiv.), BPO (1.0 mmol, 4.0 equiv.), PivOH (0.5 mmol, 2.0 equiv.), TBAI (0.05 mmol, 0.20 equiv.), Cu(OAc)₂ (0.0125 mmol, 0.05 equiv), MeCN (2.0 mL) were added in a sealed 10-mL reaction vial. Then, the mixture was stirred at 100 °C for 12 h until complete consumption of the starting material 1a as detected by TLC. The mixture was cooled to room temperature and evaporated under vacuum. The crude mixture was purified by flash column chromatography (petroleum ether /ethyl acetate) to afford the desired product 7-methyl-5-phenyl-11-tosyl-11H-benzo[b]fluoren-10-ol (3a) in 71% yield.

![Chemical Structure](image)

Red solid, mp 188-189 °C; IR (KBr, ν, cm⁻¹): 3447, 2917, 1625, 1598, 1586, 1507, 1472, 1301.

1HNMR (400 MHz, DMSO-d₆; δ, ppm) 8.29 (d, J = 8.4 Hz, 1H, ArH), 7.68 (d, J = 7.6 Hz, 1H, ArH), 7.61-7.07 (m, 7H, ArH), 7.07-6.90 (m, 4H, ArH), 6.74 (d, J = 8.4 Hz, 2H, ArH), 6.68-6.56 (m, 1H, ArH), 6.44 (s, 1H, CH), 5.85 (d, J = 8.0 Hz, 1H, OH), 2.33 (s, 3H, CH₃).

13CNMR (100 MHz, CDCl₃; δ, ppm) 150.3, 144.9, 142.2, 138.2, 137.3, 136.4, 135.5, 135.3, 130.2, 129.9, 129.4, 129.2, 129.1, 128.9, 128.5, 127.7, 127.4, 126.8, 129.7, 125.1, 123.7, 123.5, 122.9, 112.9, 70.9, 22.0, 21.5. HRMS (APCI): m/z Calcd. For: C₃₁H₂₃O₃S, 475.1367 [M-H]⁻; found: 475.1397.

11-((4-Bromophenyl)sulfonyl)-7-methyl-5-phenyl-11H-benzo[b]fluoren-10-ol (3b)

![Chemical Structure](image)

Red solid, mp 233-234 °C; IR (KBr, ν, cm⁻¹): 3466, 2936, 1689, 1565, 1543, 1511, 1488, 1321.

1HNMR (400 MHz, DMSO-d₆; δ, ppm) 7.94 (d, J = 7.6 Hz, 1H, ArH), 7.53-7.50 (m, 3H, ArH), 7.39-7.37 (m, 1H, ArH), 7.29 (d, J = 7.6 Hz, 1H, ArH), 7.25-7.11 (m, 5H, ArH), 7.06-7.02 (m, 1H,
ArH), 6.88 (d, J = 8.4 Hz, 2H, ArH), 6.79-6.75 (m, 1H, ArH), 6.01 (d, J = 8.0 Hz, 1H, CH), 5.78 (s, 1H, OH), 2.39 (s, 3H, CH₃).

HRMS (APCI): m/z Calcd. For: C₃₁H₂₄O₃S, 541.0296 [M-H]⁻, found: 541.0281.

7-Methyl-5-phenyl-11-(phenylsulfonyl)-11H-benzo[b]fluoren-10-ol (3c)

Yellow solid, mp 188-189 °C; IR (KBr, v, cm⁻¹): 3448, 2918, 1627, 1613, 1510, 1448, 1302, 1262.

¹H NMR (400 MHz, CDCl₃; δ, ppm) 8.78 (s, 1H, ArH), 8.34 (d, J = 8.4 Hz, 1H, ArH), 7.88 (d, J = 7.6 Hz, 1H, ArH), 7.51-7.36 (m, 3H, ArH), 7.33-7.29 (m, 2H, ArH), 7.09-7.07 (m, 2H, ArH), 6.06-6.09 (m, 6H, ArH), 6.67 (d, J = 7.2 Hz, 1H, ArH), 5.93 (d, J = 8.0 Hz, 1H, CH), 5.73 (s, 1H, OH), 2.32 (s, 3H, CH₃).

¹³C NMR (100 MHz, CDCl₃; δ, ppm) 149.2, 141.2, 137.1, 136.3, 135.3, 134.5, 134.1, 132.7, 131.8, 129.2, 129.2, 129.1, 128.4, 128.1, 128.0, 126.9, 126.7, 126.5, 126.4, 125.8, 125.7, 124.1, 122.6, 122.4, 121.9, 111.7, 69.8, 20.9.

HRMS (APCI): m/z Calcd. For: C₃₀H₂₂O₃S, 461.1211 [M-H]⁻, found: 461.1216.

11-((4-(Tert-butyl)phenyl)sulfonyl)-7-methyl-5-phenyl-11H-benzo[b]fluoren-10-ol (3d)

Yellow solid, mp 208-209 °C; IR (KBr, v, cm⁻¹): 3436, 2964, 1628, 1593, 1507, 1463, 1398, 1308.

¹H NMR (400 MHz, CDCl₃; δ, ppm) 8.29 (d, J = 8.4 Hz, 1H, ArH), 7.72 (d, J = 7.6 Hz, 1H, ArH), 7.69-7.29 (m, 5H, ArH), 7.22-7.12 (m, 3H, ArH), 7.03-6.94 (m, 2H, ArH), 6.80-6.72 (m, 2H, ArH), 6.66-6.64 (m, 1H, ArH), 6.44 (s, 1H, CH), 5.81 (d, J = 8.0 Hz, 1H, OH), 2.33 (s, 3H, CH₃), 1.17 (s, 9H, CH₃).

¹³C NMR (100 MHz, CDCl₃; δ, ppm) 157.9, 150.3, 142.2, 138.2, 137.2, 136.5, 135.4, 135.4, 130.3, 130.1, 129.6, 129.4, 129.1, 129.0, 128.8, 127.7, 127.5, 127.4, 126.8, 126.7, 125.2, 124.8, 123.7, 123.4, 122.9, 113.1, 70.8, 35.1, 30.9, 22.0.

HRMS (APCI): m/z Calcd. For: C₃₄H₂₀O₃S, 517.1837 [M-H]⁻, found: 517.1855.

5-(4-Methoxyphenyl)-7-methyl-11-tosyl-11H-benzo[b]fluoren-10-ol (3e)

Red solid, mp 209-210 °C; IR (KBr, v, cm⁻¹): 3483, 2919, 1646, 1577, 1547, 1518, 1457, 1328.

¹H NMR (400 MHz, DMSO-d₆; δ, ppm) 8.33 (d, J = 8.4 Hz, 1H, ArH), 7.86 (d, J = 7.6 Hz, 1H, ArH), 7.29 (d, J = 8.4 Hz, 1H, ArH), 7.19 (s, 1H, ArH), 7.09-7.04 (m, 2H, ArH), 7.00-6.96 (m, 2H, ArH), 6.93-6.88 (m, 3H, ArH), 6.76 (d, J = 8.1 Hz, 2H, ArH), 6.59-6.56 (m, 1H, ArH), 6.07 (d, J =
7.8 Hz, 1H, CH), 5.71 (s, 1H, OH), 3.86 (s, 3H, OCH₃), 2.33 (s, 3H, CH₃), 2.18 (s, 3H, CH₃). HRMS (APCI): m/z Calcd. For: C₃₂H₂₆O₃S, 505.1474 [M-H]⁻, found: 505.1482.

5-(4-Chlorophenyl)-7-methyl-11-tosyl-11H-benzo[b]fluoren-10-ol (3f)

Red solid, mp 194-195 °C; IR (KBr, v, cm⁻¹): 3441, 2972, 1627, 1576, 1560, 1446, 1398, 1321. 
¹H NMR (400 MHz, DMSO-d₆; δ, ppm) 8.29 (d, J = 8.4 Hz, 1H, ArH), 7.69 (d, J = 7.6 Hz, 1H, ArH), 7.65-7.56 (m, 2H, ArH), 7.53-7.20 (m, 4H, ArH), 7.11-7.07 (m, 1H, ArH), 7.04-6.86 (m, 3H, ArH), 6.98 (d, J = 8.4 Hz, 2H, ArH), 6.66-6.56 (m, 1H, ArH), 6.45 (s, 1H, CH), 5.94 (d, J = 8.0 Hz, 1H, OH), 2.35 (s, 3H, CH₃), 2.27 (s, 3H, CH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 150.6, 144.9, 141.9, 137.5, 136.7, 136.5, 135.4, 135.3, 133.7, 131.8, 131.7, 129.9, 129.5, 129.4, 129.3, 129.2, 128.6, 127.6, 127.6, 126.9, 125.2, 124.8, 123.7, 123.3, 123.1, 112.9, 70.8, 22.0, 21.5. HRMS (APCI): m/z Calcd. For: C₃₁H₂₅ClO₃S, 509.0977 [M-H]⁻, found: 509.0991.

7-Methyl-5-(naphthalen-1-yl)-11-tosyl-11H-benzo[b]fluoren-10-ol (3g)

Yellow solid, mp 210-211 °C; IR (KBr, v, cm⁻¹): 3442, 2933, 1663, 1586, 1534, 1478, 1397, 1202. 
¹H NMR (400 MHz, CDCl₃; δ, ppm) 9.09 (s, 1H, ArH), 8.46 (d, J = 8.4 Hz, 1H, ArH), 8.00-7.94 (m, 3H, ArH), 7.68-7.56 (m, 1H, ArH), 7.46-7.42 (m, 1H, ArH), 7.37 (d, J = 8.4 Hz, 1H, ArH), 7.31 (d, J = 6.8 Hz, 1H, ArH), 7.22-7.19 (m, 1H, ArH), 7.05-7.00 (m, 3H, ArH), 6.95-6.75 (m, 5H, ArH), 6.54 (d, J = 8.4 Hz, 1H, ArH), 5.85 (s, 1H, CH), 5.63 (d, J = 8.0 Hz, 1H, OH), 2.29 (s, 3H, CH₃), 2.27 (s, 3H, CH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 150.6, 144.8, 141.8, 137.4, 136.1, 135.7, 135.2, 133.8, 132.8, 130.2, 129.5, 129.2, 128.7, 128.3, 128.2, 128.2, 127.7, 127.4, 126.7, 126.1, 125.9, 125.7, 125.3, 125.2, 124.0, 123.8, 123.4, 122.9, 112.6, 70.8, 21.9, 21.8. HRMS (APCI): m/z Calcd. For: C₃₃H₂₇ClO₃S, 525.1524 [M-H]⁻, found: 525.1542.

7-Bromo-5-(4-methoxyphenyl)-11-tosyl-11H-benzo[b]fluoren-10-ol (3h)

Yellow solid, mp 218-219 °C; IR (KBr, v, cm⁻¹): 3446, 2943, 622, 581, 1511, 1426, 1363, 1243. 
¹H NMR (400 MHz, CDCl₃; δ, ppm) 8.36 (d, J = 8.8 Hz, 1H, ArH), 7.93 (d, J = 7.6 Hz, 1H, ArH), 7.60-7.57 (m, 1H, ArH), 7.52 (d, J = 1.6 Hz, 1H, ArH), 7.32-7.28 (m, 1H, ArH), 7.16-7.07 (m, 2H,
7-Benzyl-5-(p-tolyl)-11-tosyl-11H-benzo[b]fluoren-10-ol (3i)

Red solid, mp 176-177 °C; IR (KBr, v, cm⁻¹): 3469, 2920, 1621, 1577, 1418, 1354, 1289, 1232. 
\(^1\)HNMR (400 MHz, CDCl₃; δ, ppm) 8.36 (d, J = 8.8 Hz, 1H, ArH), 7.93 (d, J = 7.6 Hz, 1H, ArH), 7.60-7.57 (m, 1H, ArH), 7.51 (s, 1H, ArH), 7.37-7.26 (m, 3H, ArH), 7.24 (d, J = 6.4 Hz, 1H, ArH), 7.10-7.04 (m, 2H, ArH), 6.94 (d, J = 8.0 Hz, 2H, ArH), 6.84 (d, J = 8.0 Hz, 2H, ArH), 6.57 (d, J = 8.0 Hz, 1H, ArH), 6.11 (d, J = 8.0 Hz, 1H, CH), 5.77 (s, 1H, OH), 2.49 (s, 3H, CH₃), 2.26 (s, 3H, CH₃). 
\(^1\)CNMR (100 MHz, CDCl₃; δ, ppm) 150.3, 144.9, 142.2, 138.2, 137.2, 136.4, 135.4, 135.3, 130.3, 130.2, 129.8, 129.4, 129.1, 129.0, 128.9, 128.5, 127.7, 127.5, 127.4, 126.8, 126.7, 125.1, 123.6, 123.5, 122.9, 70.9, 21.3, 21.5. HRMS (APCI): m/z Calcd. For: C₃₁H₂₃BrO₅S, 553.0472 [M-H]⁻, found: 553.0490.

7-Benzyl-5-phenyl-11-tosyl-11H-benzo[b]fluoren-10-ol (3j)

Red solid, mp 200-201 °C; IR (KBr, v, cm⁻¹): 3443, 3024, 1623, 1581, 1490, 1447, 1415, 1305, 1215. 
\(^1\)HNMR (400 MHz, CDCl₃; δ, ppm) 8.37 (d, J = 8.8 Hz, 1H, ArH), 7.93 (d, J = 7.6 Hz, 1H, ArH), 7.65-7.40 (m, 5H, ArH), 7.30 (d, J = 7.6 Hz, 1H, ArH), 7.23-7.16 (m, 1H, ArH), 7.03 (m, 1H, ArH), 6.95 (d, J = 8.4 Hz, 2H, ArH), 6.85 (d, J = 8.0 Hz, 2H, ArH), 6.70 (d, J = 7.2 Hz, 1H, ArH), 6.03 (d, J = 8.0 Hz, 1H, CH), 5.77 (s, 1H, OH), 2.27 (s, 3H, CH₃). 
\(^1\)CNMR (100 MHz, CDCl₃; δ, ppm) 150.3, 145.2, 141.7, 137.6, 137.18, 136.5, 135.4, 130.2, 130.1, 129.7, 129.6, 129.3, 129.1, 129.1, 128.7, 128.6, 128.2, 127.9, 126.8, 126.4, 124.9, 123.9, 123.7, 122.3, 114.2, 70.8, 21.5. HRMS (APCI): m/z Calcd. For: C₃₀H₂₁BrO₅S, 539.0316 [M-H]⁻, found: 539.0343.

5-Benzyl-11-tosyl-11H-benzo[b]fluoren-10-ol (3k)

Red solid, mp 212-213 °C; IR (KBr, v, cm⁻¹): 3457, 3011, 1624, 1596, 1443, 1422, 1380, 1283.
1\(^1\)HNMR (400 MHz, CDCl\(_3\); δ, ppm) 8.52 (d, J = 8.4 Hz, 1H, ArH), 7.93 (d, J = 7.6 Hz, 1H, ArH), 7.57-7.39 (m, 6H, ArH), 7.36 (d, J = 8.4 Hz, 1H, ArH), 7.28 (d, J = 7.6 Hz, 1H, ArH), 7.24-7.20 (m, 1H, ArH), 7.07-6.92 (m, 3H, ArH), 6.83 (d, J = 8.0 Hz, 2H, ArH), 6.75 (d, J = 7.2 Hz, 1H, ArH), 6.07 (d, J = 8.0 Hz, 1H, CH), 5.80 (s, 1H, HOH), 2.25 (s, 3H, CH\(_3\)). 1\(^3\)CNMR (100 MHz, CDCl\(_3\); δ, ppm) 150.3, 145.0, 142.2, 138.1, 136.3, 135.3, 135.2, 130.3, 130.2, 129.9, 129.5, 129.2, 129.1, 129.0, 128.6, 127.8, 127.5, 127.3, 127.3, 126.8, 126.1, 125.5, 125.3, 123.5, 123.1, 113.7, 70.9, 21.5. HRMS (APCI): m/z Calcd. For: C\(_{30}\)H\(_{22}\)O\(_3\)S, 461.1211 [M-H]; found: 461.1219.

7-Bromo-5-phenyl-11-(phenylsulfonyl)-11\(^H\)-benzo[b]fluoren-10-ol (3I)

Yellow solid, mp 194-195 °C; IR (KBr, v, cm\(^{-1}\)): 3449, 2953, 1644, 1578, 1545, 1483, 1324, 1298, 1206. 1\(^1\)HNMR (400 MHz, CDCl\(_3\); δ, ppm) 8.38 (d, J = 8.8 Hz, 1H, ArH), 7.95 (d, J = 7.6 Hz, 1H, ArH), 7.61-7.58 (m, 1H, ArH), 7.55-7.50 (m, 2H, ArH), 7.49-7.28 (m, 5H, ArH), 7.20-7.18 (m, 1H, ArH), 7.11-7.00 (m, 5H, ArH), 6.72 (d, J = 7.2 Hz, 1H, ArH), 6.02 (d, J = 8.0 Hz, 1H, CH), 5.79 (s, 1H, HOH). 1\(^3\)CNMR (100 MHz, CDCl\(_3\); δ, ppm) 150.3, 141.7, 137.5, 137.1, 136.5, 135.2, 133.9, 132.7, 130.2, 130.0, 129.6, 129.3, 129.2, 129.1, 128.7, 128.2, 128.2, 128.0, 127.0, 126.5, 124.9, 124.0, 123.7, 122.3, 114.0, 70.8. HRMS (APCI): m/z Calcd. For: C\(_{29}\)H\(_{19}\)BrO\(_3\)S, 525.0159 [M-H]; found: 525.0163.

5-(4-Chlorophenyl)-11-(phenylsulfonyl)-11\(^H\)-benzo[b]fluoren-10-ol (3m)

Yellow solid, mp 192-193 °C; IR (KBr, v, cm\(^{-1}\)): 3444, 2978, 1682, 1598, 1455, 1378, 1289, 1211. 1\(^1\)HNMR (400 MHz, CDCl\(_3\); δ, ppm) 8.51 (d, J = 8.0 Hz, 1H, ArH), 7.88 (d, J = 1.2 Hz, 1H, ArH), 7.56-7.48 (m, 3H, ArH), 7.47-7.40 (m, 2H, ArH), 7.37-7.34 (m, 1H, ArH), 7.21-7.19 (m, 1H, ArH), 7.05-6.94 (m, 3H, ArH), 6.88 (d, J = 8.0 Hz, 2H, ArH), 6.76-6.67 (m, 1H, ArH), 5.96 (d, J = 8.4 Hz, 1H, CH), 5.74 (s, 1H, HOH), 2.27 (s, 3H, CH\(_3\)). 1\(^3\)CNMR (100 MHz, CDCl\(_3\); δ, ppm) 150.3, 145.3, 140.6, 137.7, 137.0, 135.2, 133.3, 130.1, 130.0, 129.7, 129.7, 129.2, 129.1, 129.0, 128.7, 128.0, 127.5, 127.4, 126.7, 126.1, 125.6, 124.3, 123.1, 113.4, 70.6, 21.5. HRMS (APCI): m/z Calcd. For: C\(_{29}\)H\(_{19}\)ClO\(_3\)S, 481.0664 [M-H]; found: 481.0681.

13-Phenyl-8-tosyl-8\(^H\)-indenol[2,1-b]phenanthren-7-ol (3n)

Yellow solid, mp 156-157 °C; IR (KBr, v, cm\(^{-1}\)): 3445, 2911, 1668, 1599, 1538, 1505, 1373, 1302.
\(^1\)H NMR (400 MHz, CDCl\(_3\); \(\delta\), ppm) 8.77 (s, 1H, ArH), 8.47 (d, \(J = 8.8\) Hz, 1H, ArH), 7.87 (s, 1H, ArH), 7.81-7.65 (m, 2H, ArH), 7.56-7.40 (m, 4H, ArH), 7.40-7.34 (m, 1H, ArH), 7.18-7.10 (m, 2H, ArH), 6.97 (m 1H, ArH), 6.89 (m, 3H, ArH), 6.77 (m, 3H, ArH), 5.71 (s, 1H, CH), 5.55 (d, \(J = 8.0\) Hz, 1H, OH), 2.17 (s, 3H, CH\(_3\)). \(^{13}\)CNMR (100 MHz, CDCl\(_3\); \(\delta\), ppm) 149.4, 144.3, 141.1, 137.0, 136.9, 135.9, 134.6, 133.2, 129.3, 129.3, 129.2, 129.2, 129.1, 129.1, 128.9, 128.8, 128.3, 127.8, 127.4, 127.1, 126.0, 125.7, 124.1, 123.2, 123.0, 121.4, 113.5, 70.1, 20.7. HRMS (APCI): m/z Calcd. For: C\(_{34}\)H\(_{25}\)O\(_3\)S, 511.1376 [M+H]-, found: 511.1375.

**Preparation of Compound 5a: 1-(4-Chlorophenyl)-3-phenyl-3-tosylpropan-1-one**

A mixture of 1-(4-chlorophenyl)-3-phenylprop-2-en-1-one (4a, 0.25 mmol, 1.0 equiv.), tosyldiazide (2a, 0.5 mmol, 2.0 equiv.), BPO (0.50 mmol, 2.0 equiv.), TBHP (0.25 mol, 1.0 equiv., 70% in water), TBAI (0.05 mmol, 0.2 equiv.), MeCN (2.5 mL) were added in a sealed 10-mL reaction vial. Then, the mixture was stirred in oil bath at 100 °C for 6 h until complete consumption of the starting material 4a as detected by TLC. The mixture was cooled to room temperature and evaporated under vacuum. The crude mixture was purified by flash column chromatography (petroleum ether/ethyl acetate) to afford the desired product 5a in 77% yield.

![Chemical Structure of 5a](image)

White solid, mp 199-201 °C; IR (KBr, v, cm\(^{-1}\)): 2949, 2921, 1689, 1589, 1489, 1311, 1229, 1142, 1086. \(^1\)HNMR (400 MHz, CDCl\(_3\); \(\delta\), ppm) 7.81 (d, \(J = 8.4\) Hz, 2H, ArH), 7.42-7.28 (m, 4H, ArH), 7.19-7.05 (m, 7H, ArH), 4.83-4.77 (m, 1H, CH), 4.01 (d, \(J = 3.6\) Hz, 1H, CH\(_2\)), 3.82 (d, \(J = 9.6\) Hz, 1H, CH\(_2\)), 2.32 (s, 3H, CH\(_3\)). \(^{13}\)CNMR (100 MHz, CDCl\(_3\); \(\delta\), ppm) 193.9, 144.8, 140.2, 134.5, 133.9, 132.6, 129.7, 129.5, 129.4, 129.1, 128.8, 128.5, 66.4, 37.0, 21.6. HRMS (ESI): m/z Calcd. For: C\(_{22}\)H\(_{19}\)ClO\(_2\)S, 399.0821 [M+H]+, found: 399.0813.

**1-(4-Chlorophenyl)-3-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-1-one (5b)**

![Chemical Structure of 5b](image)

White solid, mp 182-183 °C; IR (KBr, v, cm\(^{-1}\)): 3060, 2920, 1689, 1595, 1497, 1314, 1258, 1139, 1089. \(^1\)HNMR (400 MHz, CDCl\(_3\); \(\delta\), ppm) 7.95-7.83 (m, 2H, ArH), 7.43 (d, \(J = 8.4\) Hz, 4H, ArH), 7.25-7.14 (m, 5H, ArH), 6.83 (d, \(J = 8.8\) Hz, 2H, ArH), 4.88-4.85 (m, 1H, CH), 4.15-4.03 (m, 1H, CH\(_2\)), 3.94-3.86 (m, 1H, CH\(_2\)), 3.83 (s, 3H, OCH\(_3\)). \(^{13}\)CNMR (100 MHz, CDCl\(_3\); \(\delta\), ppm) 193.9, 163.8, 140.2, 134.5, 133.9, 132.7, 131.2, 129.7, 129.6, 129.1, 128.8, 128.5, 113.9, 66.6, 55.6, 37.0. HRMS (ESI): m/z Calcd. For: C\(_{22}\)H\(_{19}\)ClO\(_4\)S, 415.0771 [M+H]+, found: 415.0774.

**1-(4-Chlorophenyl)-3-phenyl-3-(phenylsulfonfonyl)propan-1-one (5c)**
White solid, mp 190-192 °C; IR (KBr, v, cm⁻¹): 3063, 2921, 1588, 1447, 1306, 1226, 1139, 1085. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 7.95-7.85 (m, 2H, ArH), 7.58-7.53 (m, 3H, ArH), 7.45-7.37 (m, 4H, ArH), 7.26-7.16 (m, 5H, ArH), 4.93-4.89 (m, 1H, CH), 4.15-4.09 (m, 1H, CH₂), 3.92-3.85 (m, 1H, CH₂). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 193.8, 140.3, 136.8, 134.4, 133.8, 132.4, 129.7, 129.6, 129.1, 129.0, 128.9, 128.7, 128.5, 66.4, 36.9.

HRMS (ESI): m/z Calcd. For: C₂₁H₁₇ClO₃S, 385.0665 [M+H]+, found: 385.0650.

3-(4-Methoxyphenyl)-1-phenyl-3-tosylpropan-1-one (5d)

White solid, mp 186-187 °C; IR (KBr, v, cm⁻¹): 3071, 2938, 1670, 1601, 1498, 1310, 1243, 1145, 1087. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 7.95-7.87 (m, 2H, ArH), 7.42 (d, J = 8.4 Hz, 2H, ArH), 7.24-7.20 (m, 7H, ArH), 6.98-6.88 (m, 2H, ArH), 4.92-4.89 (m, 1H, CH), 4.09-4.01 (m, 1H, CH₂), 3.91 (d, J = 9.6 Hz, 1H, CH₂), 3.86 (s, 3H, OCH₃), 2.38 (s, 3H, CH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 193.4, 163.9, 144.7, 134.1, 132.7, 130.5, 129.8, 129.4, 129.3, 129.0, 128.7, 128.4, 113.9, 66.6, 55.5, 36.6, 21.6. HRMS (ESI): m/z Clcd. For: C₂₃H₂₂O₄S, 395.1317 [M+H]+, found: 395.1318.

3-(4-Methoxyphenyl)-3-(4-methoxyphenyl)sulfonyl)-1-phenylpropan-1-one (5e)

White solid, mp 181-182 °C; IR (KBr, v, cm⁻¹): 3061, 2920, 1671, 1598, 1499, 1305, 1238, 1138, 1088. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 7.92 (d, J = 8.8 Hz, 2H, ArH), 7.48-7.36 (m, 2H, ArH), 7.24-7.11 (m, 5H, ArH), 6.92 (d, J = 8.8 Hz, 2H, ArH), 6.82 (d, J = 8.8 Hz, 2H, ArH), 4.93 (s, 1H, CH), 4.08-4.00 (m, 1H, CH₂), 3.90 (d, J = 9.6 Hz, 1H, CH₂), 3.86 (s, 3H, OCH₃), 3.83 (s, 3H, OCH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 193.4, 163.9, 163.7, 132.9, 131.2, 130.5, 129.8, 129.3, 128.7, 128.4, 113.9, 113.9, 66.8, 55.6, 55.5, 36.6. HRMS (ESI): m/z Clcd. For: C₂₃H₂₂O₅S, 411.1266 [M+H]+, found: 411.1262.
White solid, mp 178-179 °C; IR (KBr, v, cm⁻¹): 3060, 2939, 1671, 1598, 1499, 1306, 1256, 1138, 1089. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 7.95 (d, J = 8.8 Hz, 2H, ArH), 7.61-7.52 (m, 3H, ArH), 7.41 (d, J = 8.0 Hz, 2H, ArH), 7.26-7.20 (m, 5H, ArH), 6.94 (d, J = 8.8 Hz, 2H, ArH), 4.97-4.94 (m, 1H, CH), 4.12-4.06 (m, 1H, CH₂), 3.98-3.90 (m, 1H, CH₂), 3.88 (s, 3H, CH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 193.3, 163.9, 137.0, 133.7, 132.6, 130.5, 129.8, 129.0, 128.75, 128.7, 128.4, 113.9, 66.6, 55.5, 36.4. HRMS (ESI): m/z Calcd. For: C₂₂H₂₀O₄S, 381.1160 [M+H]⁺, found: 381.1161.

3-(4-Bromophenyl)-1-(m-tolyl)-3-tosylpropan-1-one (5g)

White solid, mp 181-182 °C; IR (KBr, v, cm⁻¹): 3073, 2939, 1688, 1595, 1486, 1302, 1286, 1084. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 7.74-7.70 (m, 2H, ArH), 7.45 (d, J = 8.4 Hz, 2H, ArH), 7.40-7.32 (m, 4H, ArH), 7.22 (d, J = 8.0 Hz, 2H, ArH), 7.11-7.07 (m, 2H, ArH), 4.87-4.84 (m, 1H, CH), 4.10-4.05 (m, 1H, CH₂), 3.91-3.84 (m, 1H, CH₂), 2.40 (d, J = 2.8 Hz, 6H, CH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 194.9, 145.0, 138.6, 136.0, 134.5, 133.8, 131.8, 131.6, 131.3, 129.6, 129.0, 128.6, 128.6, 125.3, 123.0, 65.9, 37.0, 21.7, 21.3. HRMS (ESI): m/z Calcd. For: C₂₃H₂₁BrO₃S, 457.0473 [M+H]⁺, found: 457.0465.

3-(4-Bromophenyl)-3-((4-methoxyphenyl)sulfonyl)-1-(m-tolyl)propan-1-one (5h)

White solid, mp 173-175 °C; IR (KBr, v, cm⁻¹): 3079, 2941, 1685, 1596, 1498, 1298, 1261, 1147, 1086, 1008. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 7.72 (s, 2H, ArH), 7.48 (d, J = 8.8 Hz, 2H, ArH), 7.40-7.32 (m, 4H, ArH), 7.09 (d, J = 8.4 Hz, 2H, ArH), 6.87 (d, J = 8.8 Hz, 2H, ArH), 4.86-4.81 (m, 1H, CH), 4.06 (d, J = 3.2 Hz, 1H, CH₂), 3.94-3.87 (m, 1H, CH₂), 3.85 (s, 3H, OCH₃), 2.40 (s, 3H, CH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 195.0, 163.9, 138.7, 136.0, 134.6, 133.9, 132.0, 131.7, 131.3, 131.2, 128.7, 128.2, 125.3, 123.0, 114.1, 66.1, 55.7, 37.1, 21.3. HRMS (ESI): m/z Calcd. For: C₂₃H₂₁BrO₄S, 473.0422 [M+H]⁺, found: 473.0440.
3-(4-Bromophenyl)-3-(phenylsulfonyl)-1-(m-tolyl)propan-1-one (5i)

White solid, mp 174-175 °C; IR (KBr, ν, cm⁻¹): 3064, 2949, 1684, 1585, 1490, 1307, 1255, 1142, 1085. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 7.75-7.70 (m, 2H, ArH), 7.62-7.56 (m, 3H, ArH), 7.46-7.31 (m, 6H, ArH), 7.07 (d, J = 8.4 Hz, 2H, ArH), 4.90-4.86 (m, 1H, CH), 4.12-4.07 (m, 1H, CH₂), 3.94-3.83 (m, 1H, CH), 2.40 (s, 3H, CH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 194.8, 138.7, 136.8, 136.0, 134.6, 133.9, 131.7, 131.3, 129.0, 128.9, 128.7, 128.7, 125.3, 123.1, 65.9, 36.9, 21.3. HRMS (ESI): m/z Calcd. For: C₂₂H₁₉BrO₃S, 443.0316 [M+H]^+, found: 443.0327.

3-(4-Bromophenyl)-1-(naphthalen-2-yl)-3-tosylpropan-1-one (5j)

White solid, mp 179-180 °C; IR (KBr, ν, cm⁻¹): 3068, 2924, 1679, 1598, 1490, 1303, 1256, 1142, 1089. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 8.49 (s, 1H, ArH), 7.99 (d, J = 8.0 Hz, 1H, ArH), 7.93 (d, J = 8.4 Hz, 1H, ArH), 7.87 (d, J = 8.8 Hz, 2H, ArH), 7.65-7.58 (m, 2H, ArH), 7.48 (d, J = 8.0 Hz, 2H, ArH), 7.35 (d, J = 8.4 Hz, 2H, ArH), 7.23 (d, J = 8.0 Hz, 2H, ArH), 7.13 (d, J = 8.4 Hz, 2H, ArH), 4.94-4.89 (m, 1H, CH), 4.26-7.21 (m, 1H, CH₂), 4.07-4.00 (m, 1H, CH₂), 2.41 (s, 3H, CH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 194.6, 145.1, 135.8, 133.8, 133.8, 132.5, 131.8, 131.7, 131.0, 130.3, 129.7, 129.6, 129.0, 128.9, 128.7, 127.8, 127.7, 127.1, 123.5, 123.7, 66.0, 37.0, 21.7. HRMS (ESI): m/z Calcd. For: C₂₅H₁₉BrO₃S, 493.0473 [M+H]^+, found: 493.0482.

3-(4-Bromophenyl)-3-((4-methoxyphenyl)sulfonyl)-1-(naphthalen-2-yl)propan-1-one (5k)

White solid, mp 196-197 °C; IR (KBr, ν, cm⁻¹): 3059, 2923, 1682, 1594, 1497, 1315, 1259, 1141, 1090. ¹HNMR (400 MHz, CDCl₃; δ, ppm) 8.49 (s, 1H, ArH), 7.99 (d, J = 8.0 Hz, 1H, ArH), 7.93 (d, J = 8.8 Hz, 1H, ArH), 7.87 (d, J = 8.4 Hz, 2H, ArH), 7.64-7.56 (m, 2H, ArH), 7.51 (d, J = 8.4 Hz, 2H, ArH), 7.36 (d, J = 8.0 Hz, 2H, ArH), 7.13 (d, J = 8.4 Hz, 2H, ArH), 6.88 (d, J = 8.8 Hz, 2H, ArH), 4.92-4.87 (m, 1H, CH), 4.22 (d, J = 3.2 Hz, 1H, CH₂), 4.05 (d, J = 10.0 Hz, 1H, CH₂), 3.85 (s, 3H, OCH₃). ¹³CNMR (100 MHz, CDCl₃; δ, ppm) 194.5, 144.5, 137.4, 137.1, 136.7, 133.7, 133.6, 131.0, 130.0, 129.5, 129.4, 129.1, 128.7, 128.3, 127.0, 66.2, 36.8, 21.7, 19.7, 19.5. HRMS (ESI): m/z Calcd. For: C₂₆H₂₁BrO₃S, 509.0422 [M+H]^+, found: 509.0426.
3-(4-Bromophenyl)-1-(naphthalen-2-yl)-3-(phenylsulfonyl)propan-1-one (5l)

White solid, mp 191-192 °C; IR (KBr, v, cm⁻¹): 3065, 2923, 1675, 1589, 1490, 1307, 1225, 1123, 1086. §HNMR (400 MHz, CDCl₃; δ, ppm) 8.50 (s, 1H, ArH), 7.99 (d, J = 7.6 Hz, 1H, ArH), 7.95-7.92 (m, 1H, ArH), 7.88 (d, J = 8.8Hz, 2H, ArH), 7.65-7.57 (m, 5H, ArH), 7.46-7.42 (m, 2H, ArH), 7.35 (d, J = 8.4 Hz, 2H, ArH), 7.11 (d, J = 8.4 Hz, 2H, ArH), 4.96-4.92 (m, 1H, CH), 4.29-4.23(m, 1H, CH₂), 4.09-4.02 (m, 1H, CH₂). §CNRM (100 MHz, CDCl₃; δ, ppm) 194.5, 136.8, 135.9, 134.0, 133.3, 132.4, 131.7, 131.3, 130.2, 129.7, 129.0, 128.7, 127.8, 127.1, 123.5, 123.2, 66.1, 36.8. HRMS (ESI): m/z Calcd. For: C₂₅H₁₀Br₂O₃S, 481.0290 [M+H]⁺, found: 481.0302.

1,3-Bis(4-bromophenyl)-3-tosylpropan-1-one (5m)

White solid, mp 203-204 °C; IR (KBr, v, cm⁻¹): 3068, 2922, 1689, 1584, 1491, 1299, 1227, 1137, 1010. §HNMR (400 MHz, CDCl₃; δ, ppm) 7.79 (d, J = 8.4 Hz, 2H, ArH), 7.61 (d, J = 8.4 Hz, 2H, ArH), 7.44 (d, J = 8.4 Hz, 2H, ArH), 7.35 (d, J = 8.4 Hz, 2H, ArH), 7.22 (d, J = 8.0 Hz, 2H, ArH), 7.07 (d, J = 8.4 Hz, 2H, ArH), 4.84-4.81(m, 1H, CH), 4.05 (d, J = 3.6 Hz, 1H, CH₂), 3.83 (d, J = 10.0 Hz, 1H, CH₂), 2.41 (s, 3H, CH₃). §CNRM (100 MHz, CDCl₃; δ, ppm) 193.8, 145.1, 134.7, 133.6, 132.2, 131.7, 131.3, 129.6, 129.2, 129.0, 123.2, 65.8, 37.0, 21.7. HRMS (ESI): m/z Calcd. For: C₂₂H₁₈Br₂O₂S, 522.9396 [M+H]⁺, found: 522.9390.

1,3-Bis(4-bromophenyl)-3-((4-methoxyphenyl)sulfonyl)propan-1-one (5n)

White solid, mp 210-211 °C; IR (KBr, v, cm⁻¹): 3093, 2921, 1687, 1595, 1495, 1261, 1226, 1136, 1087. §HNMR (400 MHz, CDCl₃; δ, ppm) 7.79 (d, J = 8.4 Hz, 2H, ArH), 7.61 (d, J = 8.4 Hz, 2H, ArH), 7.47 (d, J = 8.8 Hz, 2H, ArH), 7.35 (d, J = 8.4 Hz, 2H, ArH), 7.07 (d, J = 8.4 Hz, 2H, ArH), 6.87 (d, J = 8.8 Hz, 2H, ArH), 4.81 (d, J = 6.0 Hz, 1H, CH), 4.10-4.05 (m, 1H, CH₂), 3.83 (s, 3H, OCH₃), 3.82 (d, J = 10.0 Hz, 1H, CH₂). §CNRM (100 MHz, CDCl₃; δ, ppm) 193.9, 163.9, 134.7, 132.2, 131.9, 131.7, 131.3, 131.2, 129.6, 129.2, 128.0, 123.2, 114.2, 66.0, 55.7, 37.0. HRMS (ESI): m/z Calcd. For: C₂₂H₁₈Br₂O₂S, 538.9345 [M+H]⁺, found: 538.9329.
1,3-Bis(4-bromophenyl)-3-(phenylsulfonyl)propan-1-one (5o)

![Structure](image)

White solid, mp 180-181 °C; IR (KBr, v, cm⁻¹): 3059, 2919, 1686, 1584, 1489, 1307, 1227, 1142, 1082. ¹H NMR (400 MHz, CDCl₃; δ, ppm) 7.79 (d, J = 8.4 Hz, 2H, ArH), 7.63-7.55 (m, 5H, ArH), 7.45-7.41 (m, 2H, ArH), 7.34 (d, J = 8.4 Hz, 2H, ArH), 7.06 (d, J = 8.4 Hz, 2H, ArH), 4.88-4.83 (m, 1H, CH), 4.13-4.06 (m, 1H, CH₂), 3.85 (d, J = 10.0 Hz, 1H, CH₂). ¹³C NMR (100 MHz, CDCl₃; δ, ppm) 193.7, 136.6, 134.7, 134.0, 132.2, 131.7, 131.6, 131.2, 129.6, 129.2, 129.0, 123.3, 65.8, 36.8. HRMS (ESI): m/z Calcd. For: C₂₁H₁₆Br₂O₃S, 508.9240 [M+H]^⁺, found: 508.9246.
Copies of $^1$H NMR and $^{13}$C NMR of Compounds 3 and 5

$^1$H NMR Spectrum of Compound 3a

$^{13}$C NMR Spectrum of Compound 3a
$^1$H NMR Spectrum of Compound 3b

$^1$H NMR Spectrum of Compound 3c
$^{13}$C NMR Spectrum of Compound $3c$

$^1$H NMR Spectrum of Compound $3d$
$^{13}$C NMR Spectrum of Compound 3d

$^1$H NMR Spectrum of Compound 3e
$^1$H NMR Spectrum of Compound 3f

$^{13}$C NMR Spectrum of Compound 3f

S19
$\text{H NMR Spectrum of Compound 3h}$

$\text{C NMR Spectrum of Compound 3h}$
$^1$H NMR Spectrum of Compound 3i

$^{13}$C NMR Spectrum of Compound 3i
$^1$H NMR Spectrum of Compound 3j

$^{13}$C NMR Spectrum of Compound 3j
$^{1}H$ NMR Spectrum of Compound 3k

$^{13}C$ NMR Spectrum of Compound 3k
$^{1}$H NMR Spectrum of Compound 3l

$^{13}$C NMR Spectrum of Compound 3l
$^1$H NMR Spectrum of Compound 3m

$^{13}$C NMR Spectrum of Compound 3m
H NMR Spectrum of Compound 3n

$^1$H NMR Spectrum of Compound 3n

$^{13}$C NMR Spectrum of Compound 3n
**1H NMR Spectrum of Compound 5b**

![1H NMR Spectrum of Compound 5b](image)

**13C NMR Spectrum of Compound 5b**

![13C NMR Spectrum of Compound 5b](image)
$^1$H NMR Spectrum of Compound 5c

$^{13}$C NMR Spectrum of Compound 5c
H NMR Spectrum of Compound 5d

\[ \text{H NMR Spectrum of Compound 5d} \]

\[ \text{13C NMR Spectrum of Compound 5d} \]

S31
$^{1}$H NMR Spectrum of Compound 5e

$^{13}$C NMR Spectrum of Compound 5e
$^{1}H$ NMR Spectrum of Compound 5f

$^{13}C$ NMR Spectrum of Compound 5f
$^{1}\text{H NMR Spectrum of Compound 5g}$

$^{13}\text{C NMR Spectrum of Compound 5g}$

S34
\[ \text{H NMR Spectrum of Compound 5h} \]

\[ \text{C NMR Spectrum of Compound 5h} \]
$^{1}H$ NMR Spectrum of Compound 5i

$^{13}C$ NMR Spectrum of Compound 5i
$^1$H NMR Spectrum of Compound 5j

$^{13}$C NMR Spectrum of Compound 5j
$^1$H NMR Spectrum of Compound 5k

$^{13}$C NMR Spectrum of Compound 5k
$^{1}$H NMR Spectrum of Compound 5l

$^{13}$C NMR Spectrum of Compound 5l
$^1$H NMR Spectrum of Compound 5m

$^1$C NMR Spectrum of Compound 5m
$^1$H NMR Spectrum of Compound 5n

$^{13}$C NMR Spectrum of Compound 5n

S41
$^1$H NMR Spectrum of Compound 5o

$^{13}$C NMR Spectrum of Compound 5o