

# Supporting Information

for

## A chemoselective and continuous synthesis of *m*-sulfamoylbenzamide analogues

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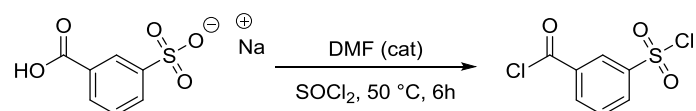
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## Experimental part

### *Synthesis of compound 1*

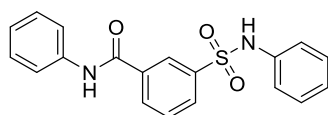


To a suspension of *m*-sulfobenzoic acid monosodium salt (10 g, 44.6 mmol) in thionyl chloride (200 mL), a catalytic amount of dimethylformamide (1 mL, 13.4 mmol) was added. Subsequently, the reaction was heated to 50 °C. After 6 hours, the reaction

mixture was filtered and the filtrate was concentrated in vacuo resulting in the desired final product. The compound was used without further purification.

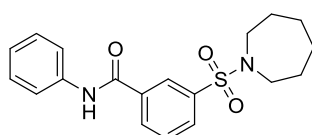
Yellow oil (95%);  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.75 (m, 1H), 8.50 (ddd, 1H,  $J = 7.9, 1.6, 1.1$  Hz), 8.31-8.28 (ddd, 1H,  $J = 8.0, 1.6, 1.1$  Hz), 7.80 (dd, 1H  $J = 8.0$  Hz);  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  166.5 (C), 145.3 (C), 137.0 (CH), 135.0 (CH), 132.8 (C), 130.9 (CH), 129.4 (CH); IR (neat):  $\nu = 3076$  (w), 1753 (s), 1593 (m), 1377 (s), 1159 (s), 1082 (m), 997 (m), 930 (s), 806 (m), 689 (s), 660 (s), 571 (s)  $\text{cm}^{-1}$ .

#### Compound **3aa**



White solid (no further purification; 140 mg 95%); m.p. = 155 - 156  $^{\circ}\text{C}$ ;  $^1\text{H-NMR}$  (400 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  10.48 (s, 1H), 10.41 (s, 1H), 8.34 (dd, 1H,  $J = 1.7$  Hz), 8.18 (ddd, 1H,  $J = 7.9, 1.7$  Hz), 7.92 (ddd, 1H,  $J = 7.8, 1.6$  Hz), 7.75 (d, 2H,  $J = 7.8$  Hz), 7.71 (dd, 1H,  $J = 7.9$  Hz), 7.37 (dd, 2H,  $J = 7.8$  Hz), 7.24 (dd, 2H,  $J = 7.8$  Hz), 7.16 – 7.09 (m, 3H), 7.03 (dd, 1H,  $J = 7.3$  Hz);  $^{13}\text{C-NMR}$  (100 MHz,  $\text{DMSO-}d_6$ ):  $\delta$  164.4 (C), 140.3 (C), 139.1 (C), 137.8 (C), 136.2 (C), 132.1 (CH), 129.8 (CH), 129.7 (CH), 129.6 (CH), 129.0 (CH), 126.5 (CH), 124.6 (CH), 124.4 (CH), 120.8 (CH), 120.6 (CH); IR (neat):  $\nu = 3379$  (w), 3136 (w), 3074 (w), 1655 (s), 1599 (m), 1533 (s), 1493 (m), 1445 (m), 1327 (m), 1159 (s), 1080 (m), 922 (m), 746 (s), 691 (s), 556 (s), 513 (m)  $\text{cm}^{-1}$ ; MS:  $m/z = 353.0$   $[\text{M}+\text{H}]^+$ .

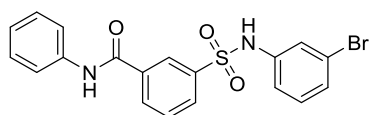
#### Compound **3ab**



White crystals (recrystallization in methanol; 197 mg, 75%); m.p. = 160 – 161  $^{\circ}\text{C}$ ;  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.26 (dd, 1H,  $J = 1.4$  Hz), 8.13 (ddd, 1H,  $J = 1.3, 7.8$  Hz),

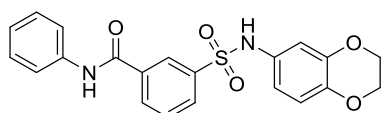
7.99 (s, br, 1H) 7.98 (ddd, 1H,  $J = 1.3, 7.7$  Hz), 7.70 – 7.65 (m, 3H), 7.45 – 7.39 (m, 2H), 7.24 – 7.19 (m, 1H), 3.33 (t, 4H,  $J = 5.9$  Hz), 1.75 (m, 4H), 1.66 – 1.60 (m, 4H);  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.9 (C), 140.4 (C), 138.0 (C), 136.6 (C), 131.9 (CH), 130.1 (CH), 129.5 (CH), 129.4 (CH), 125.5 (CH), 125.3 (CH), 121.0 (CH), 48.7 ( $\text{CH}_2$ ), 29.5 ( $\text{CH}_2$ ), 27.2 ( $\text{CH}_2$ ); IR (neat):  $\nu = 3311$  (w), 2918 (w), 2848 (w), 1651 (m), 1597 (m), 1530 (s), 1441 (m), 1329 (s), 1153 (s), 1038 (m), 743 (s), 687 (s), 565 (s), 513 (s)  $\text{cm}^{-1}$ ; MS:  $m/z = 359.1$   $[\text{M}+\text{H}]^+$ .

#### Compound **3ac**



White-yellow solid (recrystallization in methanol; 94 mg, 78%); m.p. = 203 – 204 °C;  $^1\text{H}$ -NMR (400 MHz,  $\text{DMSO}-d_6$ ):  $\delta$  10.53 (s, 1H), 10.45 (s, 1H), 8.39 (dd, 1H,  $J = 1.6$  Hz), 8.24-8.22 (m, 1H), 7.99-7.96 (m, 1H), 7.81-7.79 (m, 2H), 7.76 (dd, 1H,  $J = 7.9$  Hz), 7.44-7.40 (m, 2H), 7.29-7.27 (m, 2H), 7.17-7.15 (m, 2H), 7.11-7.06 (m, 1H);  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.5 (C), 140.4 (C), 139.2 (C), 136.3 (C), 132.2 (CH), 129.9 (CH), 129.8 (CH), 129.7 (CH), 129.1 (CH), 126.6 (C), 124.8 (CH), 124.5 (CH), 120.96 (CH), 120.7 (CH); IR (neat):  $\nu = 3345$  (m), 3127 (w), 1645 (s), 1597 (m), 1524 (s), 1447 (s), 1393 (m), 1339 (m), 1263 (m), 1161 (s), 1124 (m), 951 (s), 878 (m), 762 (m), 677 (s), 588 (s), 507 (m)  $\text{cm}^{-1}$ ; MS:  $m/z = 430.9$   $[\text{M}+\text{H}]^+$ ; 433.0  $[\text{M}+\text{H}]^+$ .

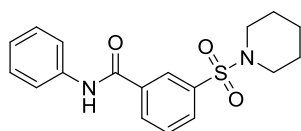
#### Compound **3ae**



White solid (column chromatography: 55% Ethyl acetate:hexane; 92 mg, 76%); m.p. = 165 – 166 °C;  $^1\text{H}$ -NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.30 (dd, 1H,  $J = 1.6$  Hz), 8.26 (s, br, 1H), 8.08-8.05 (m, 1H), 7.78-7.76 (m, 1H), 7.62-7.59 (m, 1H), 7.48 (dd, 1H,  $J = 7.9$

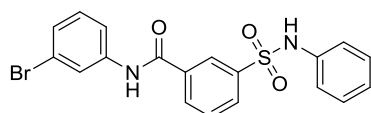
Hz), 7.28-7.23 (m, 2H), 7.26-7.25 (m, 1H), 7.16-7.11 (m, 1H), 6.69 (d, 1H,  $J = 8.7$  Hz), 6.67 (d, 1H,  $J = 2.5$  Hz), 6.53 (dd, 1H,  $J = 8.5, 2.5$  Hz), 4.17-4.16 (m, 4H);  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.2 (C), 143.7 (C), 142.2 (C), 139.5 (C), 137.5 (C), 135.9 (C), 134.2 (CH), 132.2 (CH), 130.3 (CH), 129.6 (CH), 129.1 (C), 129.1 (CH), 125.4 (CH), 125.0 (CH), 120.7 (CH), 117.6 (CH), 117.0 (CH), 113.1 (CH), 64.2 ( $\text{CH}_2$ ), 64.2 ( $\text{CH}_2$ ); IR (neat):  $\nu = 3368$  (w), 3138 (w), 1649 (m), 1599 (m), 1533 (m), 1503 (m), 1445 (m), 1339 (m), 1300 (m), 1258 (m), 1157 (s), 1065 (m), 883.4 (m), 758 (m), 681 (s), 590 (s), 554 (s), 503 (m)  $\text{cm}^{-1}$ ; MS:  $m/z = 411.0$   $[\text{M}+\text{H}]^+$ .

### Compound **3ag**



White-yellow solid (recrystallization in methanol; 88 mg, 74%); m.p. = 158 – 159 °C;  $^1\text{H}$ -NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.20 (dd, 1H,  $J = 1.6$  Hz), 8.16-8.13 (m, 1H), 8.01 (s, br, 1H), 7.93-7.90 (m, 1H), 7.70-7.65 (m, 3H), 7.42-7.37 (m, 2H), 7.21-7.17 (m, 1H), 3.02 (t, 4H,  $J = 5.4$  Hz), 1.64 (tt, 4H,  $J = 5.6$  Hz), 1.45-1.40 (m, 2H);  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.2 (C), 137.5 (C), 137.2 (C), 136.2 (C), 131.7 (CH), 130.5 (CH), 129.8 (CH), 129.2 (CH), 125.6 (CH), 125.1 (CH), 120.1 (CH), 47.0 ( $\text{CH}_2$ ), 25.1 ( $\text{CH}_2$ ), 23.4 ( $\text{CH}_2$ ); IR (neat):  $\nu = 3321$  (w), 2922 (w), 2851 (w), 1653 (s), 1597 (m), 1521 (s), 1323 (s), 1260 (m), 1161 (s), 1092 (m), 1053 (m), 932 (s), 812 (m), 773 (m), 745 (s), 681 (s), 608 (s), 579 (s)  $\text{cm}^{-1}$ ; MS:  $m/z = 345.0$   $[\text{M}+\text{H}]^+$ .

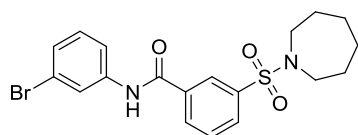
### Compound **3ca**



White solid (Column chromatography: 55% ethyl acetate:hexane; 83 mg, 70%); m.p. = 139 – 140 °C;  $^1\text{H}$ -NMR (400 MHz  $\text{DMSO}-d_6$ ):  $\delta$  10.62 (s, 1H), 10.42 (s, 1H), 8.34

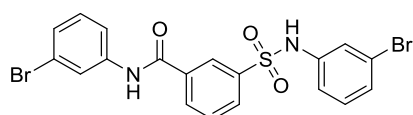
(dd, 1H,  $J=1.6$  Hz), 8.19-8.17 (m, 1H), 8.08-8.07 (m, 1H), 7.96-7.93 (m, 1H), 7.75-7.70 (m, 2H), 7.35-7.33 (m, 2H), 7.27-7.22 (m, 2H), 7.12-7.10 (m, 2H), 7.06-7.02 (m, 1H);  $^{13}\text{C}$ -NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  164.7 (C), 140.8 (C), 140.5 (C), 137.8 (C), 135.9 (C), 132.3 (CH), 131.2 (CH), 130.1 (CH), 130.1 (CH), 129.7 (CH), 127.1 (CH), 126.6 (CH), 124.8 (CH), 123.2 (CH), 121.9 (C), 120.7 (CH), 119.7 (CH); IR (neat):  $\nu$  = 3377 (w), 3167 (w), 1659 (s), 1593 (m), 1528 (m), 1412 (m), 1346 (m), 1161 (s), 1076 (m), 918 (m), 779 (s), 748 (s), 696 (s), 588 (s), 544 (s), 466 (m)  $\text{cm}^{-1}$ ; MS:  $m/z$  = 431.0  $[\text{M}+\text{H}]^+$ , 433.0  $[\text{M}+\text{H}]^+$

### Compound **3cb**



White crystals (recrystallization in methanol; 93 mg, 78%); m.p. = 152 – 153 °C  $^1\text{H}$ -NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  8.25 (dd, 1H,  $J= 1.6$  Hz), 8.14-8.10 (m, 2H), 8.00-7.96 (m, 2H), 7.68 (dd, 1H,  $J= 7.8$  Hz), 7.61-7.59 (m, 1H), 7.35-7.33 (m, 1H), 7.29 (d, 1H,  $J= 7.8$  Hz), 3.32 (t, 4H,  $J= 5.9$  Hz), 1.78-1.71 (m, 4H), 1.64-1.60 (m, 4H);  $^{13}\text{C}$ -NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  164.3 (C), 140.4 (C), 138.8 (C), 135.7 (C), 131.4 (CH), 130.4 (CH), 130.0 (CH), 129.9 (CH), 128.0 (CH), 125.0(CH), 123.4 (CH), 122.8 (C), 118.8 (CH), 48.4 (CH<sub>2</sub>), 29.1 (CH<sub>2</sub>), 26.9 (CH<sub>2</sub>); IR (neat):  $\nu$ = 3292 (w), 3075 (w), 2934 (w), 2853 (w) 1651 (s), 1587 (m), 1414 (m), 1332 (s), 1159 (s), 876 (m), 773 (s), 687 (s), 582 (s), 567 (s)  $\text{cm}^{-1}$ ; MS:  $m/z$ = 437.0  $[\text{M}+\text{H}]^+$ , 439.0  $[\text{M}+\text{H}]^+$ .

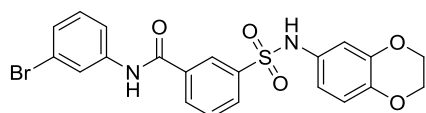
### Compound **3cc**



Yellow solid (no further purification; 118 mg, 99%); m.p. = 188 – 189 °C;  $^1\text{H}$ -NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  10.71 (s, 1H), 10.63 (s, 1H), 8.36 (dd, 1H,  $J= 1.7$  Hz), 8.22-

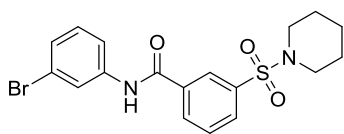
8.20 (m, 1H), 8.08-8.06 (m, 1H), 7.97 (ddd, 1H,  $J$  = 7.9, 1.7, 1.0 Hz), 7.75 (dd, 1H,  $J$  = 7.7 Hz), 7.75-7.72 (m, 1H), 7.37-7.33 (m, 2H), 7.28-7.27 (m, 1H), 7.23-7.19 (m, 2H), 7.14-7.12 (m, 1H);  $^{13}\text{C}$ -NMR (100 MHz, DMSO- $d_6$ ): 164.6 (C), 140.8 (C), 140.1 (C), 139.6 (C), 136.0 (C), 132.6 (CH), 131.8 (CH), 131.2 (CH), 130.3 (CH), 130.0 (CH), 127.4 (CH), 127.1 (CH), 126.6 (CH), 123.2 (CH), 122.6 (CH), 122.3 (C), 121.9 (C), 119.7 (CH), 119.1 (CH); IR (neat):  $\nu$  = 3375 (w), 3146 (w), 1657 (m), 1589 (m), 1526 (m), 1477 (m), 1335 (m), 1159 (s), 1072 (m), 883 (m), 777 (s), 707 (m), 590 (s), 436 (m)  $\text{cm}^{-1}$ ; MS:  $m/z$  = 508.8  $[\text{M}+\text{H}]^+$ , 510.9  $[\text{M}+\text{H}]^+$ .

### Compound **3ce**



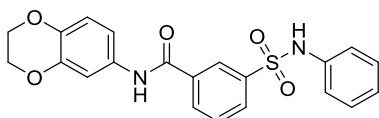
White-brown solid (column chromatography: 55% ethyl acetate:hexane; 97 mg, 81%); m.p. = 145 – 146 °C;  $^1\text{H}$ -NMR (400 MHz, DMSO- $d_6$ ):  $\delta$  10.62 (s, 1H), 10.10 (s, 1H), 8.29 (dd, 1H,  $J$  = 1.6 Hz), 8.20-8.17 (m, 1H), 8.08-8.07 (m, 1H), 7.90 (ddd, 1H,  $J$  = 7.9, 1.6, 1.0 Hz), 7.76-7.71 (m, 2H), 7.37-7.31 (m, 2H), 6.71 (d, 1H,  $J$  = 8.6 Hz), 6.58 (d, 1H,  $J$  = 2.4 Hz), 6.54 (dd, 1H,  $J$  = 8.6, 2.5 Hz), 4.17-4.13 (m, 4H);  $^{13}\text{C}$ -NMR (100 MHz, DMSO- $d_6$ ):  $\delta$  164.8 (C), 143.7 (C), 141.2 (C), 140.9 (C), 140.4 (C), 135.8 (C), 132.2 (CH), 131.2 (CH), 130.9 (C), 130.1 (CH), 130.0 (CH), 127.1 (CH), 126.6 (CH), 123.2 (CH), 121.9 (C), 119.7 (C), 117.8 (CH), 115.1 (CH), 111.0 (CH), 64.5 ( $\text{CH}_2$ ), 64.3 ( $\text{CH}_2$ ); IR (neat):  $\nu$  = 3333 (w), 3118 (w), 2965 (w), 2878 (w), 1649 (m), 1593 (m), 1510 (s), 1333 (m), 1254 (m), 1157 (s), 1070 (m), 980 (m), 856 (m), 775 (s), 675 (s), 583 (s), 490 (m)  $\text{cm}^{-1}$ ; MS:  $m/z$  = 488.9  $[\text{M}+\text{H}]^+$ , 490.9  $[\text{M}+\text{H}]^+$ .

### Compound **3cg**



White crystals (recrystallization in methanol; 92 mg, 77%); m.p. = 167 – 168 °C;  $^1\text{H}$ -NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.41 (s, br, 1H), 8.20 (dd, 1H,  $J$  = 1.6 Hz), 8.16-8.13 (m, 1H), 7.97 (dd, 1H,  $J$  = 1.7 Hz), 7.90-7.87 (m, 1H), 7.67 (dd, 1H,  $J$  = 7.8 Hz), 7.61 (d, 1H,  $J$  = 8.0 Hz), 7.32-7.29 (m, 1H), 7.24 (dd, 1H,  $J$  = 8.0 Hz), 2.99 (t, 4H,  $J$  = 5.4 Hz), 1.61 (tt, 4H,  $J$  = 5.6 Hz), 1.41 (tt, 2H,  $J$  = 5.7 Hz);  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.4 (C), 138.9 (C), 137.0 (C), 135.8 (C), 132.0 (CH), 130.6 (CH), 130.4 (CH), 129.8 (CH), 127.9 (CH), 125.7 (CH), 123.5 (CH), 122.7 (C), 119.1 (CH), 47.0 ( $\text{CH}_2$ ), 25.1 ( $\text{CH}_2$ ), 23.4 ( $\text{CH}_2$ ); IR (neat):  $\nu$  = 3296 (w), 2947 (w), 2845 (w), 1653 (m), 1589 (m), 1520 (m), 1476 (m), 1414 (m), 1337 (s), 1294 (m), 1161 (s), 1051 (m), 935 (s), 775 (s), 685 (s), 571 (s), 486 (m)  $\text{cm}^{-1}$ ; MS:  $m/z$  = 423.0  $[\text{M}+\text{H}]^+$ ; 425.0  $[\text{M}+\text{H}]^+$ .

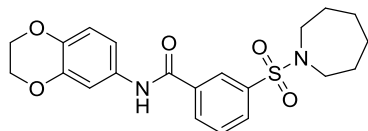
### Compound **3ea**



White-brown solid (column chromatography: 65% ethyl acetate: hexane; 96 mg, 80%); m.p. = 192 – 193 °C;  $^1\text{H}$ -NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.20 (dd, 1H,  $J$  = 1.5 Hz), 8.08-8.05 (m, 1H), 7.87-7.84 (m, 1H), 7.68 (s, br, 1H), 7.54 (dd, 1H,  $J$  = 7.9 Hz), 7.29-7.25 (m, 2H), 7.23 (d, 1H,  $J$  = 2.0 Hz), 7.19-7.15 (m, 1H), 7.11-7.08 (m, 1H), 6.98 (dd, 1H,  $J$  = 8.8, 2.2 Hz), 6.84 (d, 1H,  $J$  = 8.7 Hz), 4.28-4.24 (m, 4H);  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.0 (C), 143.5 (C), 141.0 (C), 139.5 (C), 136.1 (C), 136.0 (C), 132.1 (CH), 131.0 (C), 130.1 (CH), 129.5 (CH), 129.4 (CH), 125.9 (CH), 125.4 (CH), 122.4 (CH), 117.3 (CH), 114.3 (CH), 110.5 (CH), 64.4 ( $\text{CH}_2$ ), 64.3 ( $\text{CH}_2$ ); IR (neat):  $\nu$  = 3389 (w), 3076 (w), 2889 (w), 1639 (s), 1597 (m), 1506 (s), 1429 (m), 1342 (m), 1157

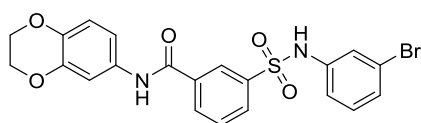
(s), 1065 (s), 920 (s), 889 (s), 767 (m), 729 (m), 679 (s), 543 (m)  $\text{cm}^{-1}$ ; MS:  $m/z$  = 411.0  $[\text{M}+\text{H}]^+$ .

#### Compound **3eb**



White crystals (recrystallization in methanol; 94 mg, 78%); m.p. = 115 – 116 °C;  $^1\text{H}$ -NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.2 (dd, 1H,  $J$  = 1.6 Hz), 8.09-8.07 (m, 1H), 7.95-7.92 (m, 1H), 7.83 (s, br, 1H), 7.63 (dd, 1H,  $J$  = 7.8 Hz), 7.29 (d, 1H,  $J$  = 1.9 Hz), 7.03 (dd, 1H,  $J$  = 8.7, 2.1 Hz), 6.86 (d, 1H,  $J$  = 8.7 Hz), 4.28-4.25 (m, 4H), 3.29 (t, 4H,  $J$  = 5.9 Hz), 1.75-1.69 (m, 4H), 1.61-1.57 (m, 4H)  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.0 (C), 143.6 (C), 141.0 (C), 140.3 (C), 136.2 (C), 131.3 (C), 131.1 (CH), 129.8 (CH), 129.7 (CH), 124.9 (CH), 117.4 (CH), 114.1 (CH), 110.3 (CH), 64.4 ( $\text{CH}_2$ ), 64.3 ( $\text{CH}_2$ ), 48.4 ( $\text{CH}_2$ ), 29.1 ( $\text{CH}_2$ ), 26.9 ( $\text{CH}_2$ ); IR (neat):  $\nu$  = 3302 (w), 2920 (w), 2853 (w), 1641 (m), 1607 (s), 1506 (s), 1412 (s), 1328 (s), 1279 (s), 1152 (s), 1069 (s), 920 (m), 862 (m), 733 (m), 577 (s)  $\text{cm}^{-1}$ ; MS:  $m/z$  = 417.0  $[\text{M}+\text{H}]^+$ .

#### Compound **3ec**

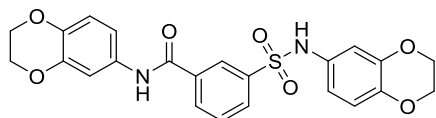


White-yellow solid (column chromatography: 60% ethyl acetate: hexane; 87 mg, 72%); m.p. = /;  $^1\text{H}$ -NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.52 (s, br, 1H), 8.47 (m, 1H), 8.04-8.01 (m, 1H), 7.77-7.74 (m, 1H), 7.41 (dd, 1H,  $J$  = 7.8 Hz), 7.32-7.31 (m, 1H), 7.19-7.16 (m, 2H), 7.05-7.02 (m, 2H), 7.00-6.97 (m, 1H), 6.74 (d, 1H,  $J$  = 8.7 Hz), 4.18-4.17 (m, 4H);  $^{13}\text{C}$ -NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  164.3 (C), 143.4 (C), 141.0 (C), 139.6 (C), 138.4 (C), 136.0 (C), 132.1 (CH), 131.0 (C), 130.6 (CH), 130.1 (CH), 129.4 (CH), 128.2 (CH), 125.6 (CH), 124.6 (CH), 122.7 (C), 120.3 (CH), 117.2 (CH), 114.6 (CH), 110.8



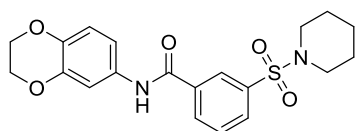
(CH), 64.4 (CH<sub>2</sub>), 64.3 (CH<sub>2</sub>); IR (neat):  $\nu$  = 3271 (w), 2930 (w), 1649 (m), 1593 (m), 1506 (s), 1477 (m), 1333 (m), 1283 (m), 1159 (s), 1066 (m), 921 (m), 887 (m), 736 (m), 682 (m), 586 (m), cm<sup>-1</sup>; MS:  $m/z$  = 488.9 [M+H]<sup>+</sup>; 490.9 [M+H]<sup>+</sup>

#### Compound **3ee**



Brown solid (no further purification; 117 mg, 98%); m.p. = /; <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  8.25 (dd, 1H,  $J$  = 1.5 Hz), 8.09-8.05 (m, 1H), 8.01 (s, br, 1H) 7.80-7.78 (m, 1H), 7.76 (s, br, 1H), 7.50 (dd, 1H,  $J$  = 7.8 Hz), 7.22 (d, 1H,  $J$  = 1.9 Hz), 7.05 (s, br, 1H), 7.01 (dd, 1H,  $J$  = 8.6, 2.0 Hz), 6.81 (d, 1H,  $J$  = 8.7 Hz), 6.70 (d, 1H,  $J$  = 8.6 Hz), 6.67 (d, 1H,  $J$  = 2.5 Hz), 6.52 (dd, 1H,  $J$  = 8.7, 2.6 Hz), 4.25-4.22 (m, 4H), 4.19-4.17 (m, 4H); <sup>13</sup>C-NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  163.9 (C), 143.7 (C), 143.756 (C), 142.734 (C), 141.0 (C), 139.4 (C), 135.9 (C), 132.1 (CH), 131.0 (C), 130.2 (CH), 129.5 (CH), 129.1 (C), 125.3 (CH), 117.6 (CH), 117.3 (CH), 117.1 (CH), 114.3 (CH), 113.2 (CH), 110.4 (CH), 64.4 (CH<sub>2</sub>), 64.3 (CH<sub>2</sub>), 64.2 (CH<sub>2</sub>), 64.17 (CH<sub>2</sub>); IR (neat):  $\nu$  = 3237 (s), 2932 (s), 1601 (m), 1502 (s), 1300 (m), 1281 (m), 1240 (m), 1204 (m), 1152 (s), 1063 (s), 966 (m), 920 (m), 885 (s), 808.17 (m), 681 (s), 621 (m), 580 (s), 548 (s) cm<sup>-1</sup>; MS:  $m/z$  = 469.0 [M+H]<sup>+</sup>.

#### Compound **3eg**

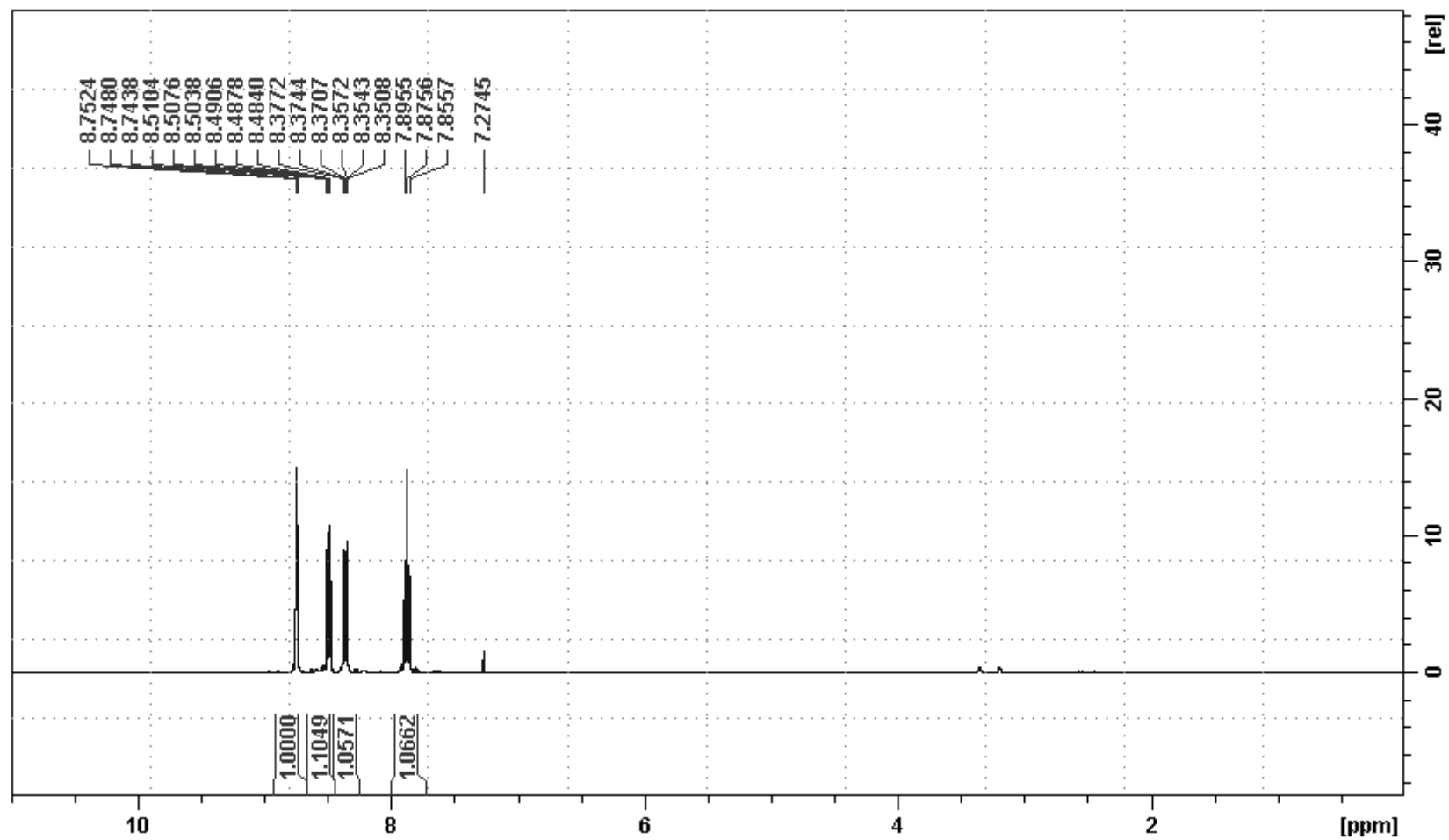


White solid (recrystallization in methanol; 93 mg, 77%); m.p. = 186 - 187 °C; <sup>1</sup>H-NMR (400 MHz, DMSO-d<sub>6</sub>):  $\delta$  10.35 (s, 1H), 8.27-8.23 (m, 2H), 7.93-7.91 (m, 1H), 7.80 (dd, 1H,  $J$  = 7.8 Hz), 7.38 (d, 1H,  $J$  = 2.4 Hz), 7.19 (dd, 1H,  $J$  = 8.7, 2.4 Hz), 6.85 (d, 1H,  $J$  = 8.7 Hz), 4.27-4.22 (m, 4H); <sup>13</sup>C-NMR (100 MHz, DMSO-d<sub>6</sub>):  $\delta$  164.0 (C),

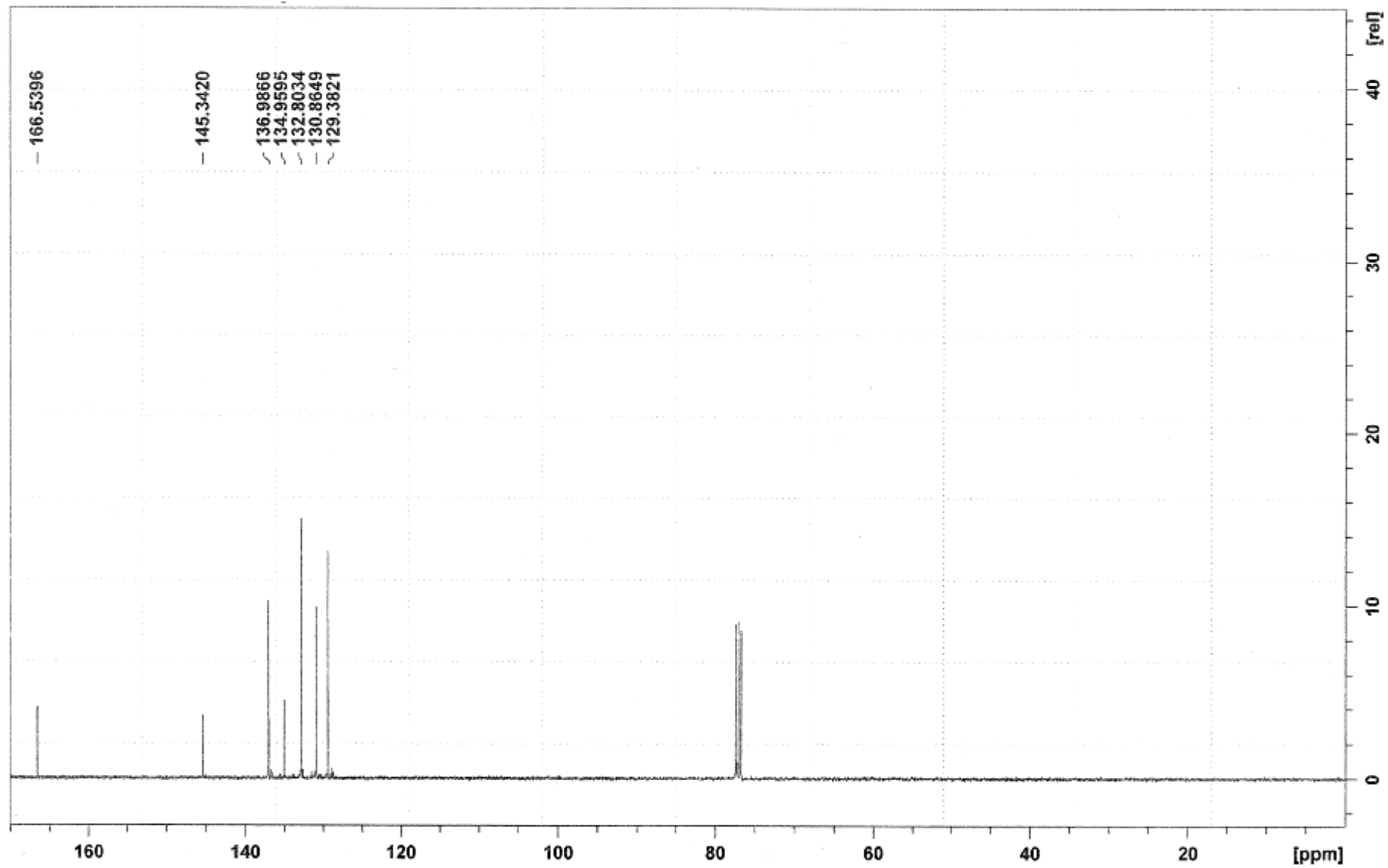
143.3 (C), 140.4 (C), 136.5 (C), 136.4 (C), 132.8 (C), 132.4 (CH), 130.6 (CH), 126.7 (CH), 117.2 (CH), 114.4 (CH), 110.2 (CH), 64.6 (CH<sub>2</sub>), 64.4 (CH<sub>2</sub>), 47.1 (CH<sub>2</sub>), 25.1 (CH<sub>2</sub>), 23.4 (CH<sub>2</sub>); IR (neat):  $\nu$ = 3273 (w), 2941 (w), 2853 (w), 1643 (m), 1611 (m), 1506 (s), 1429 (m), 1416 (m), 1337 (s), 1300 (s), 1281 (s), 1242 (m), 1204 (m), 1180 (s), 1069 (s), 932 (s), 862 (m), 737 (m), 710 (s), 596 (s), 579 (s), 501 (m) cm<sup>-1</sup>; MS:  $m/z$ = 403.0 [M+H]<sup>+</sup>.

Compound 1

$^1\text{H}$ -NMR

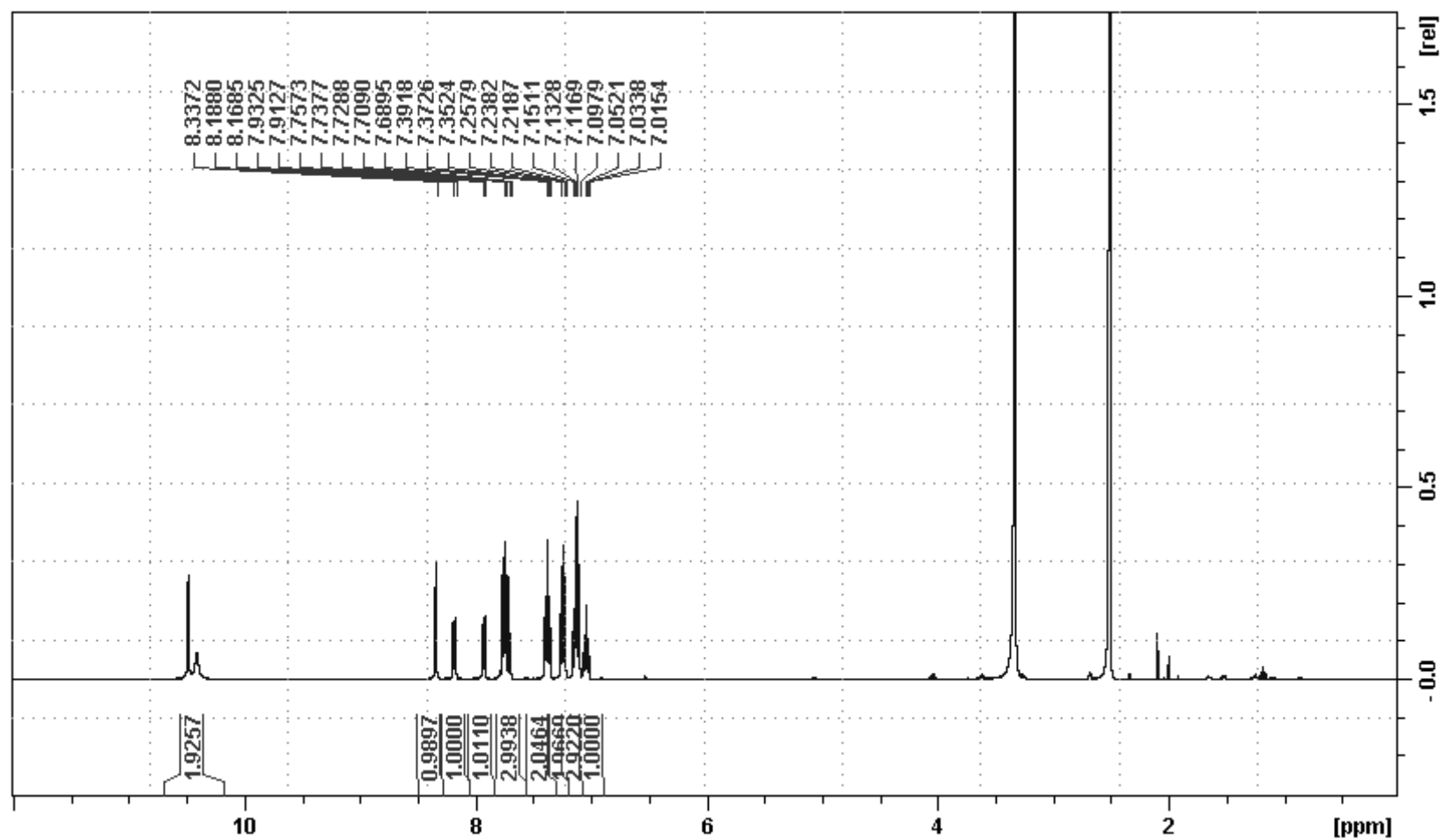


$^{13}\text{C}$ -NMR

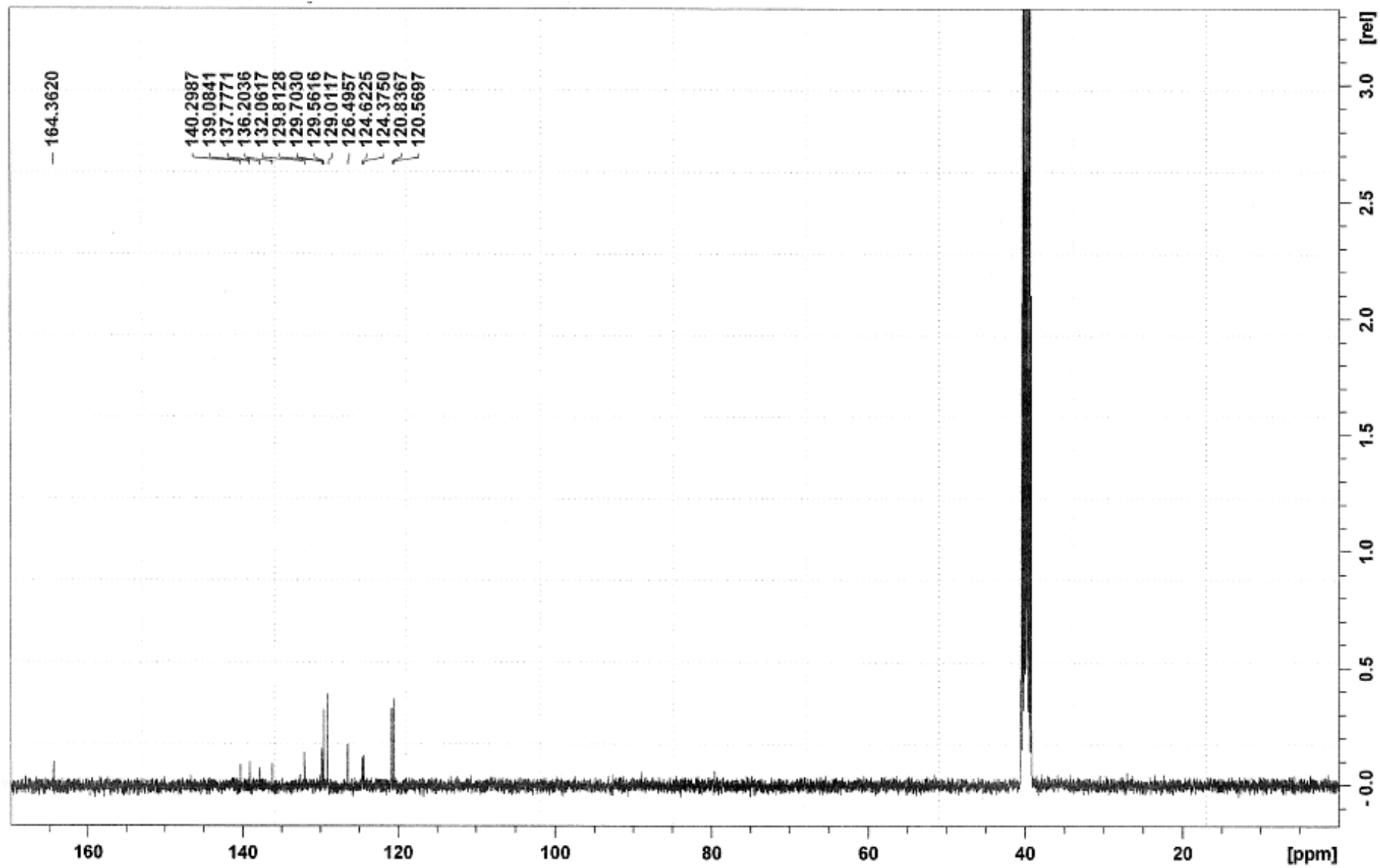


Compound **3aa**

$^1\text{H}$ -NMR

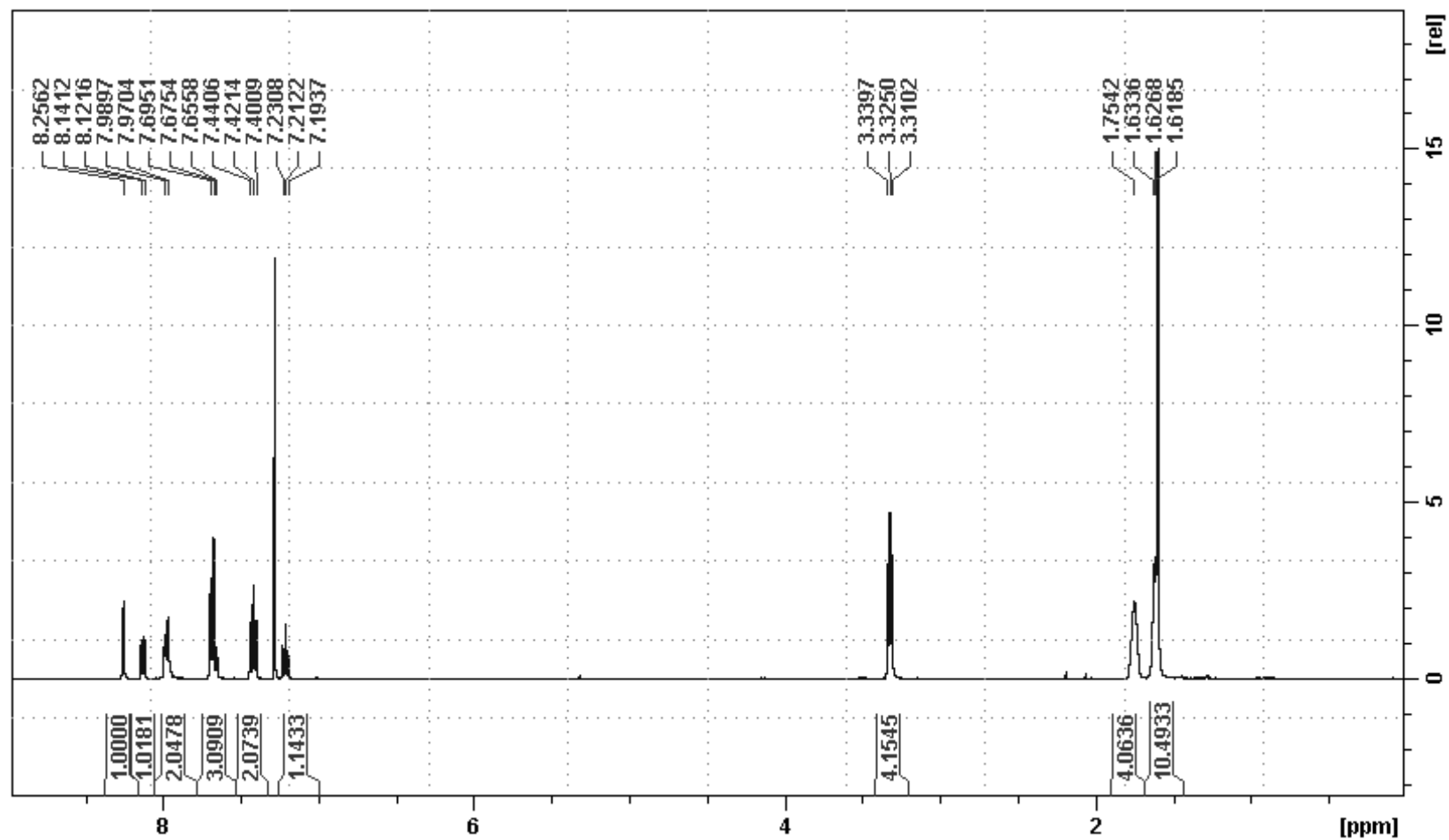


$^{13}\text{C}$ -NMR

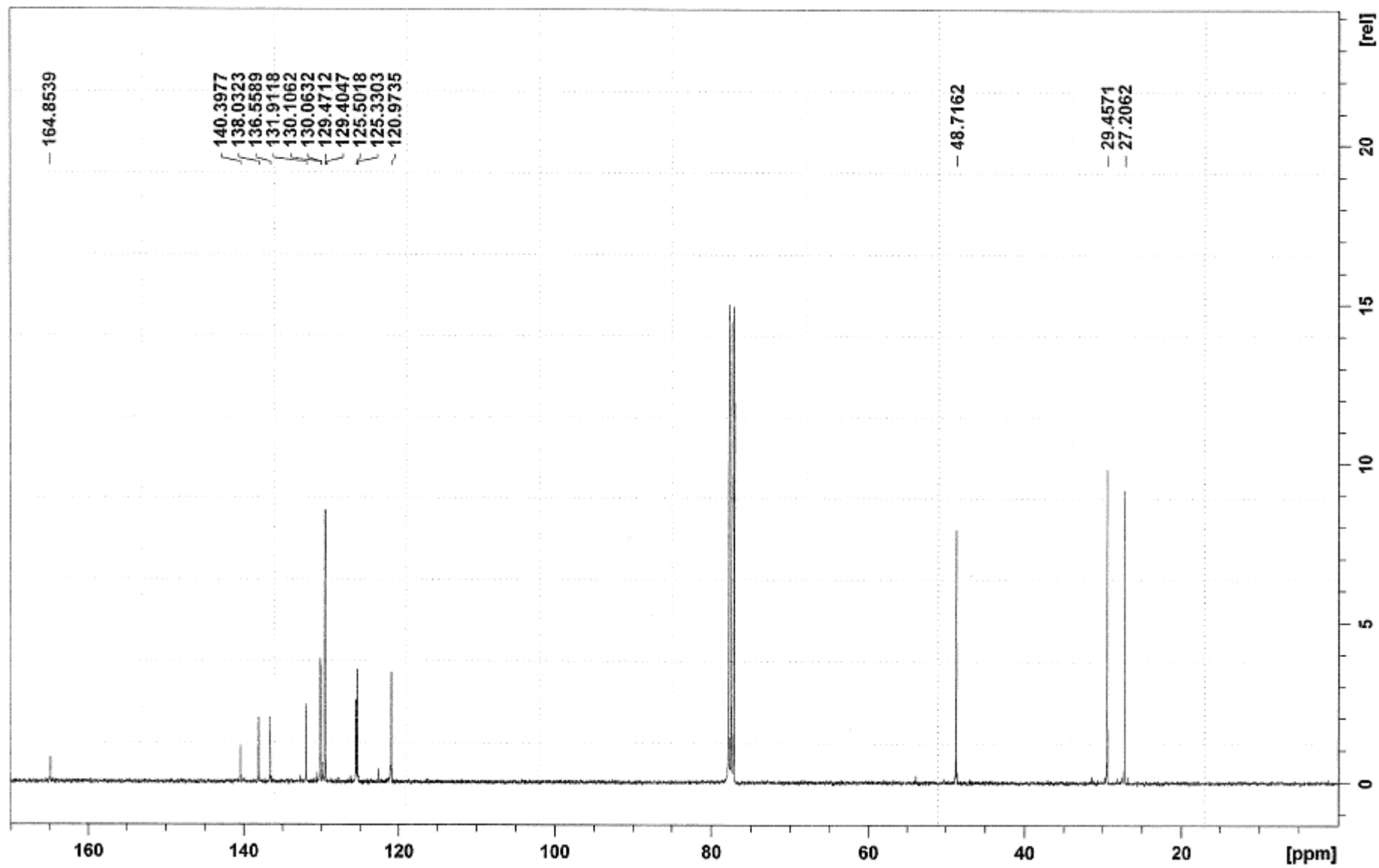


Compound **3ab**

$^1\text{H}$ -NMR



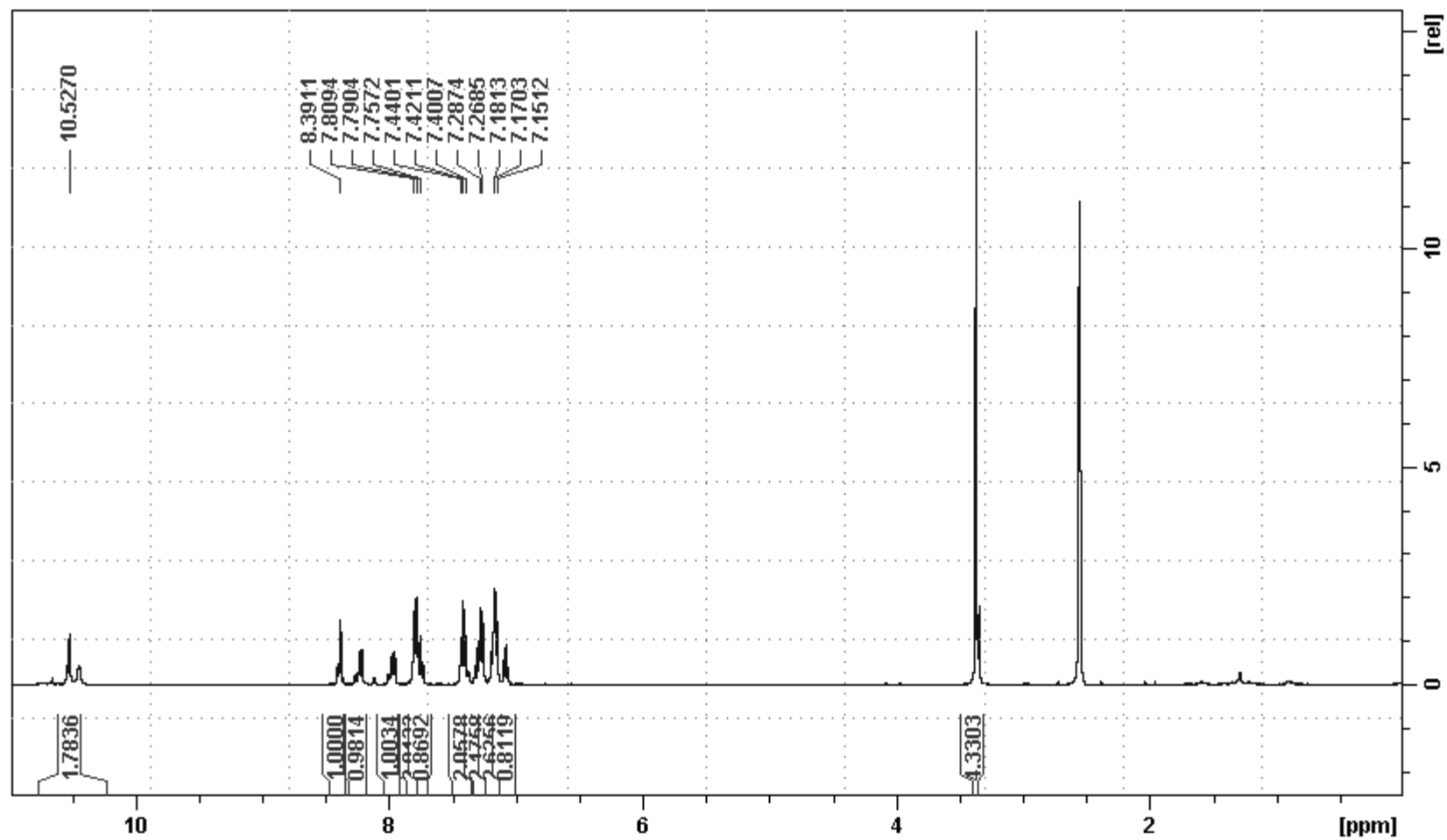
$^{13}\text{C}$ -NMR



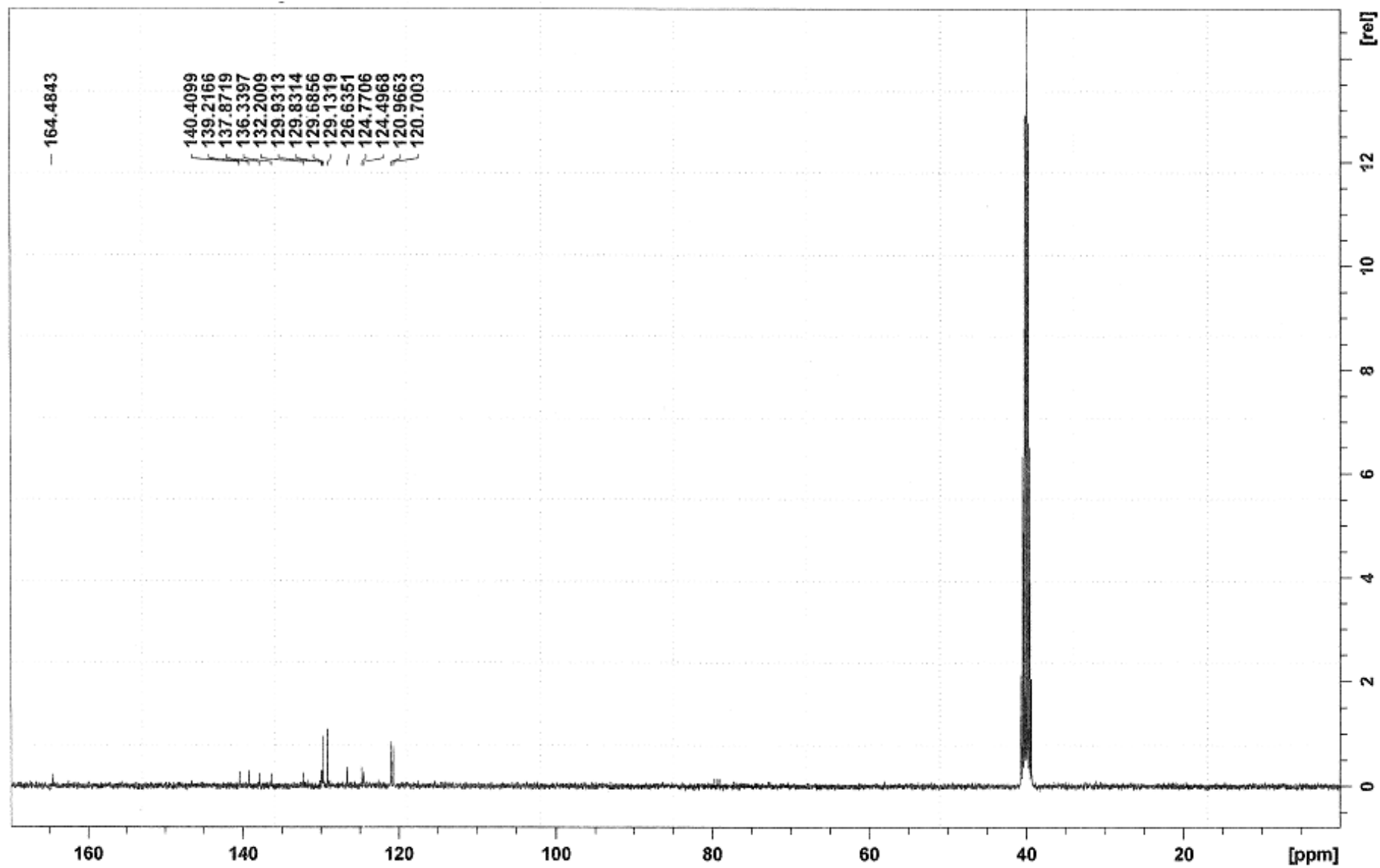


Compound **3ac**

$^1\text{H}$ -NMR

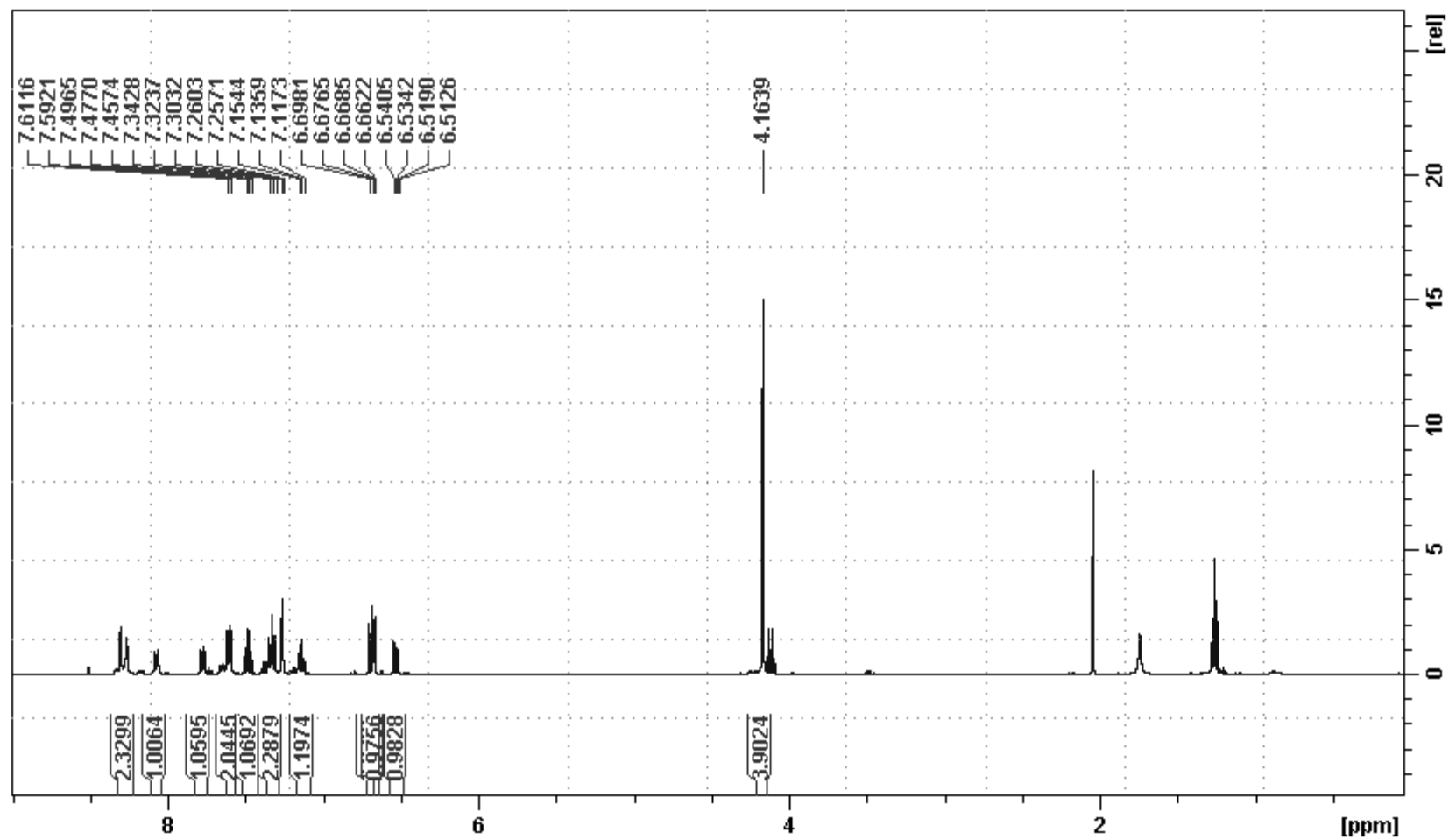


$^{13}\text{C}$ -NMR

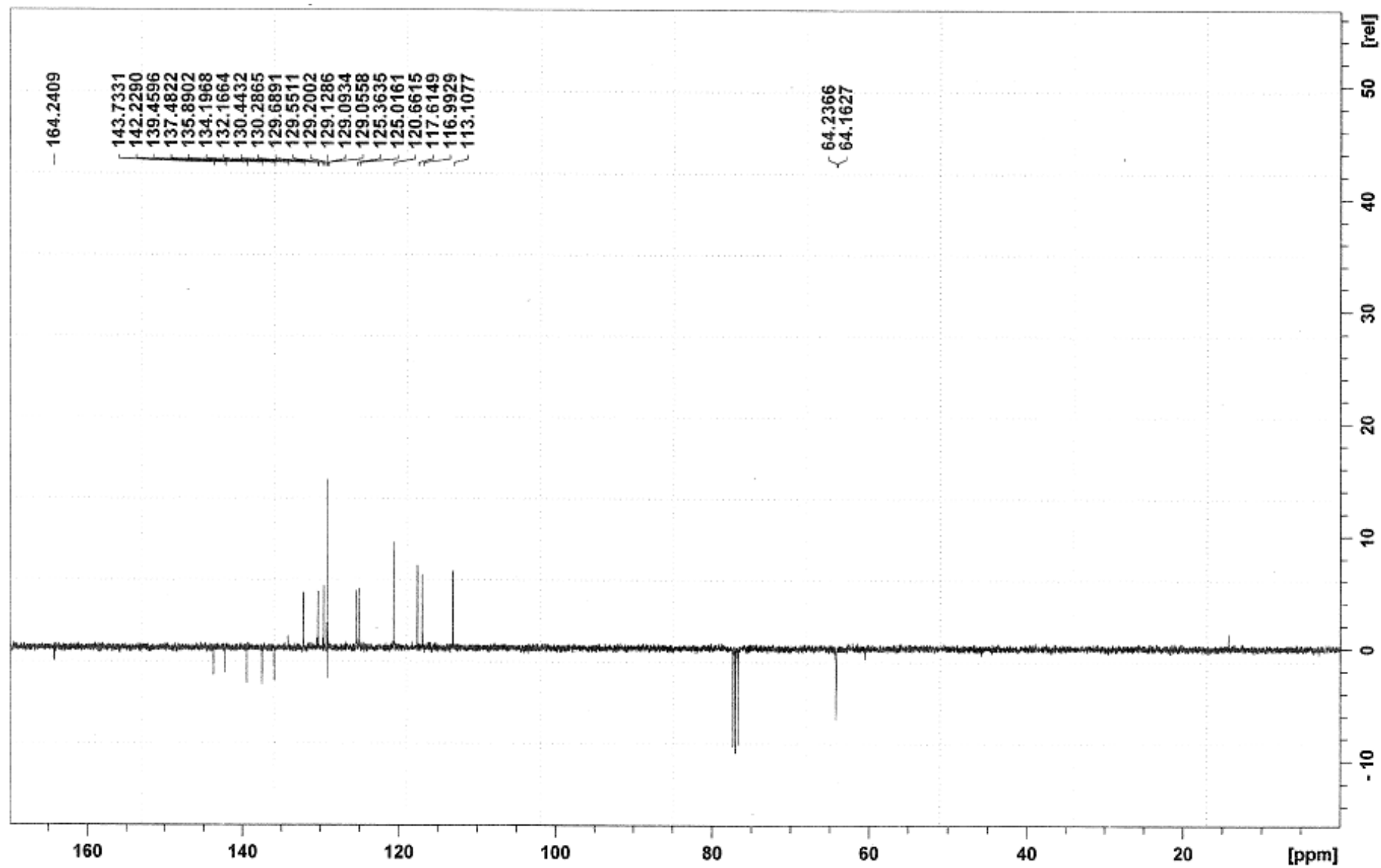


Compound **3ae**

$^1\text{H}$ -NMR

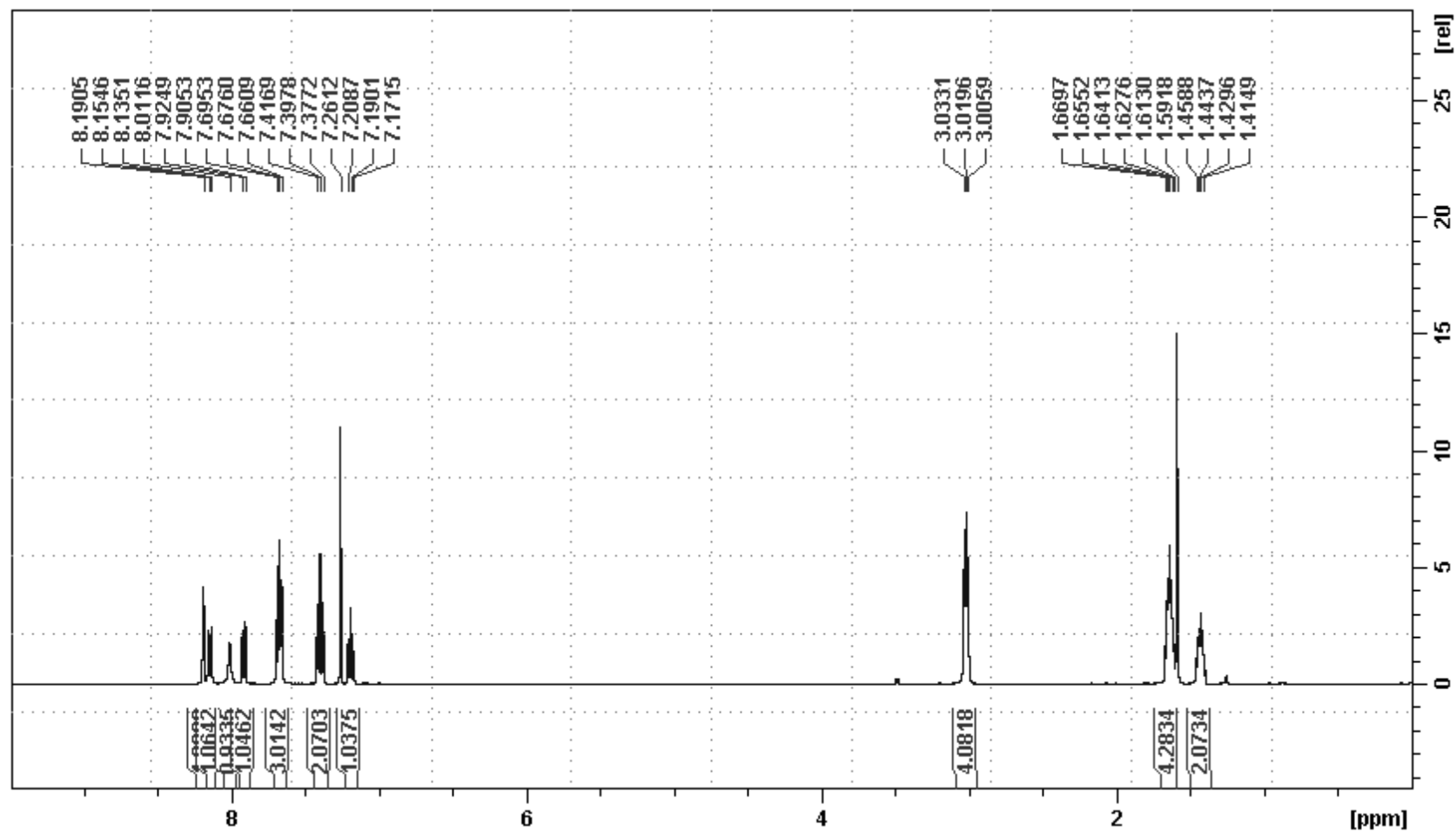


<sup>13</sup>C-NMR

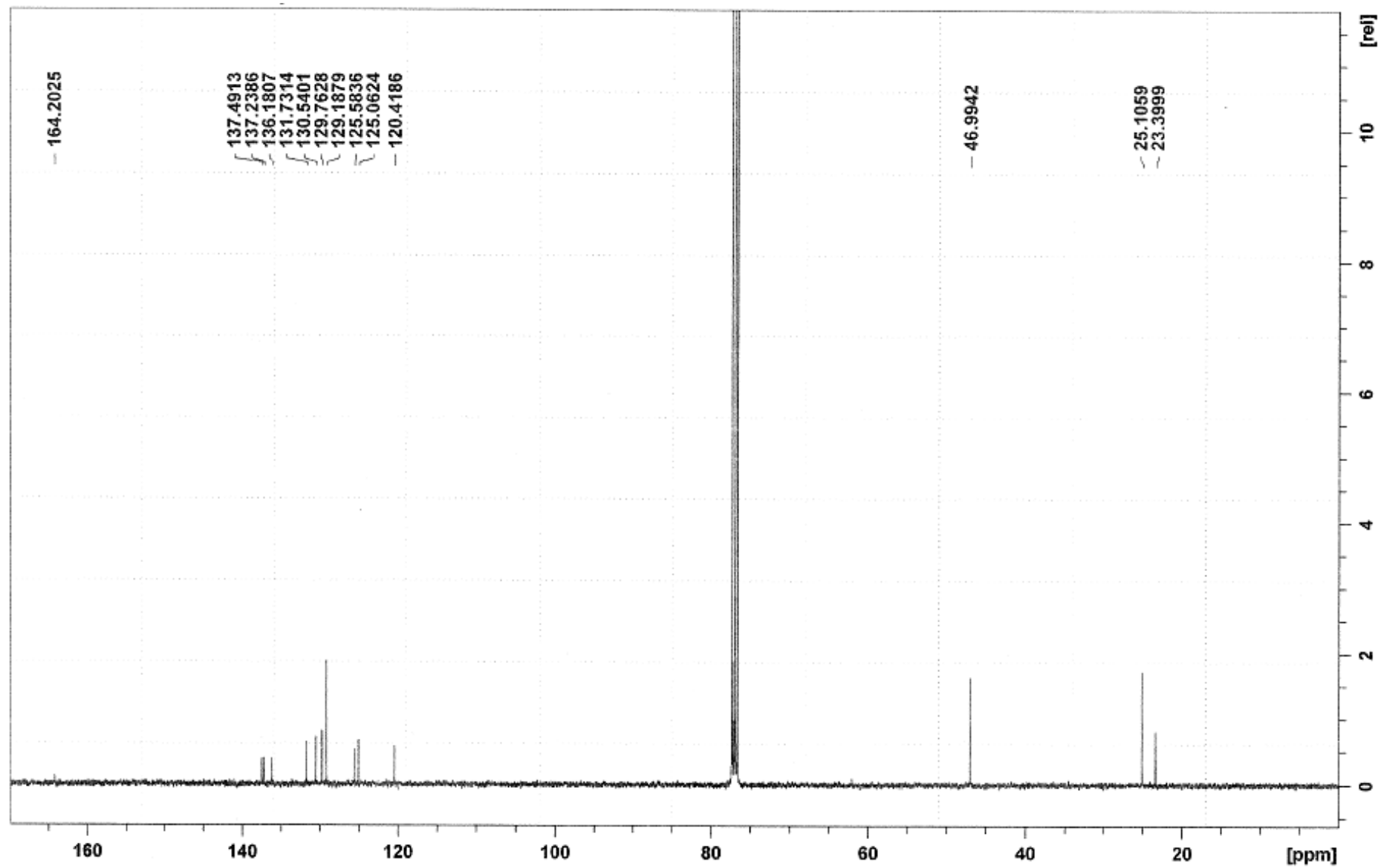


Compound **3ag**

$^1\text{H}$ -NMR

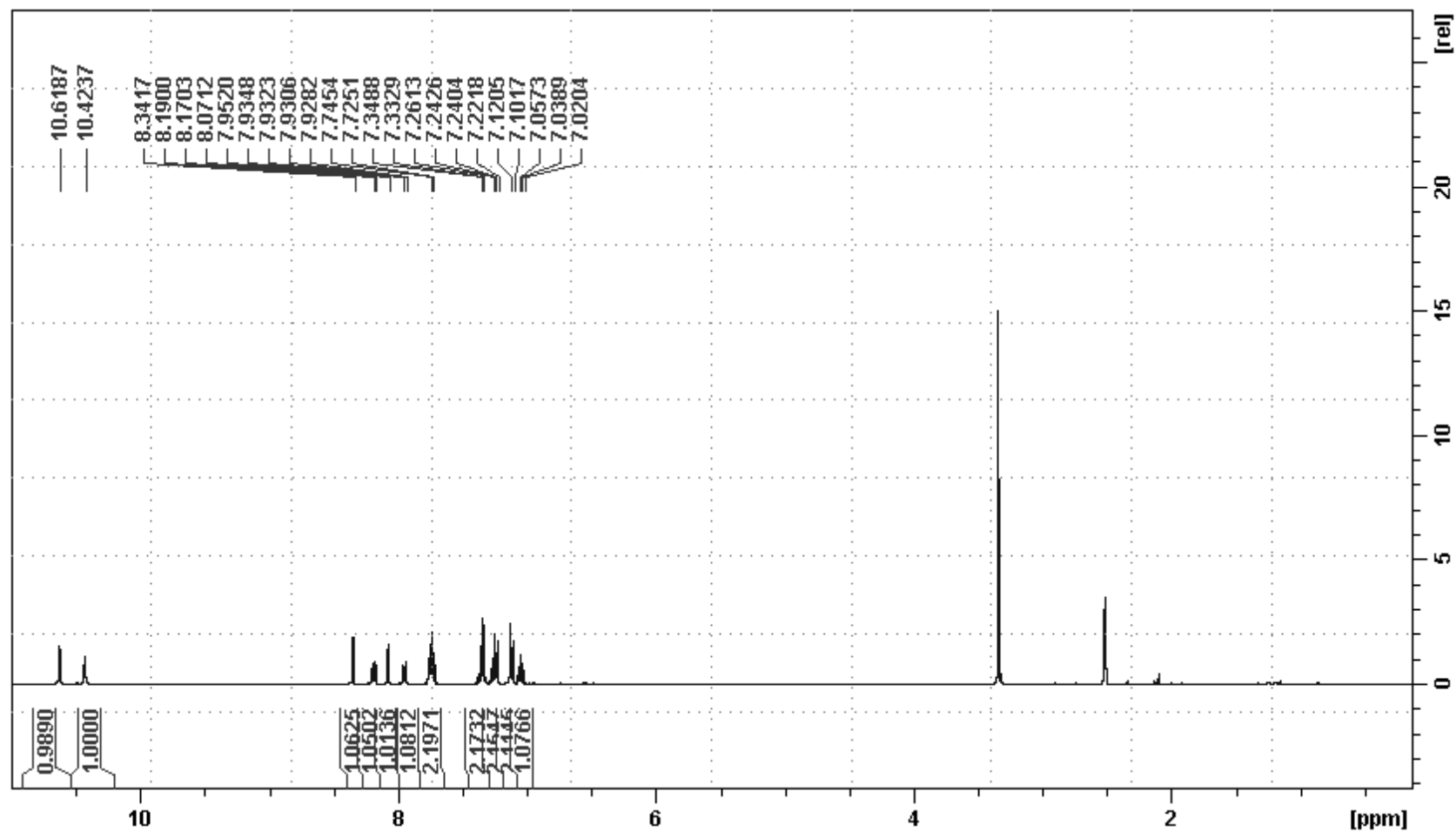


$^{13}\text{C}$ -NMR

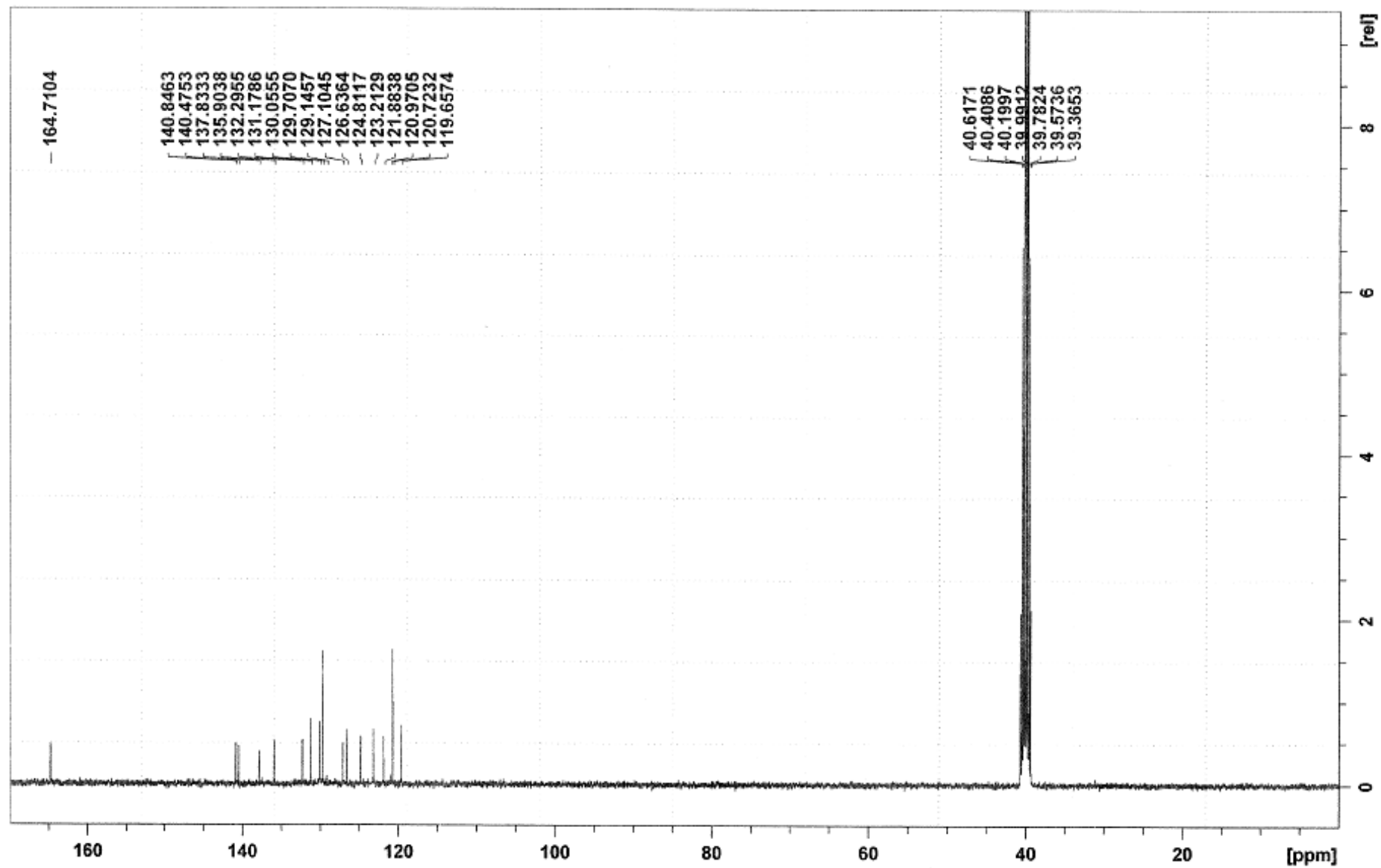


Compound **3ca**

$^1\text{H}$ -NMR



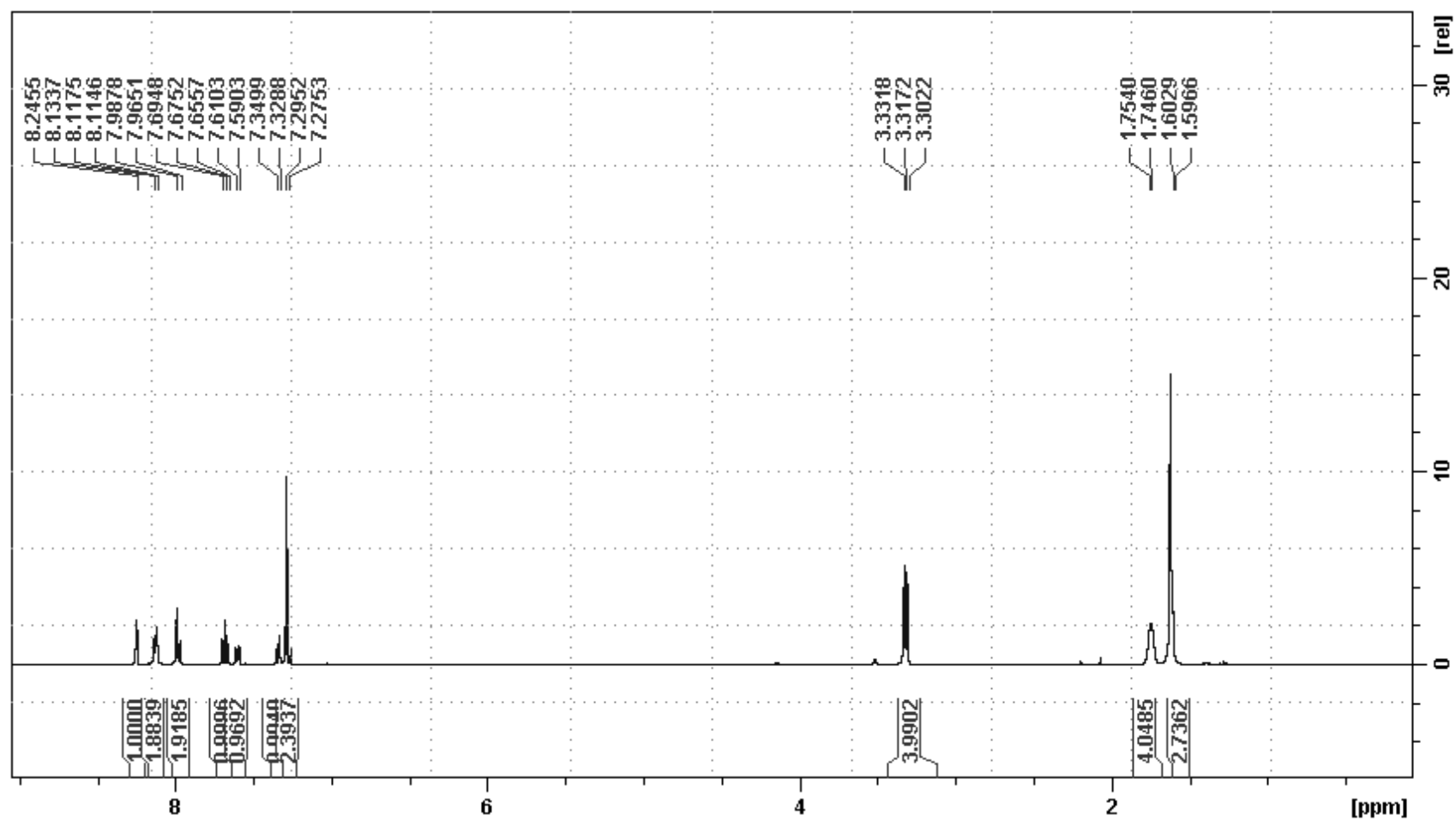
$^{13}\text{C}$ -NMR



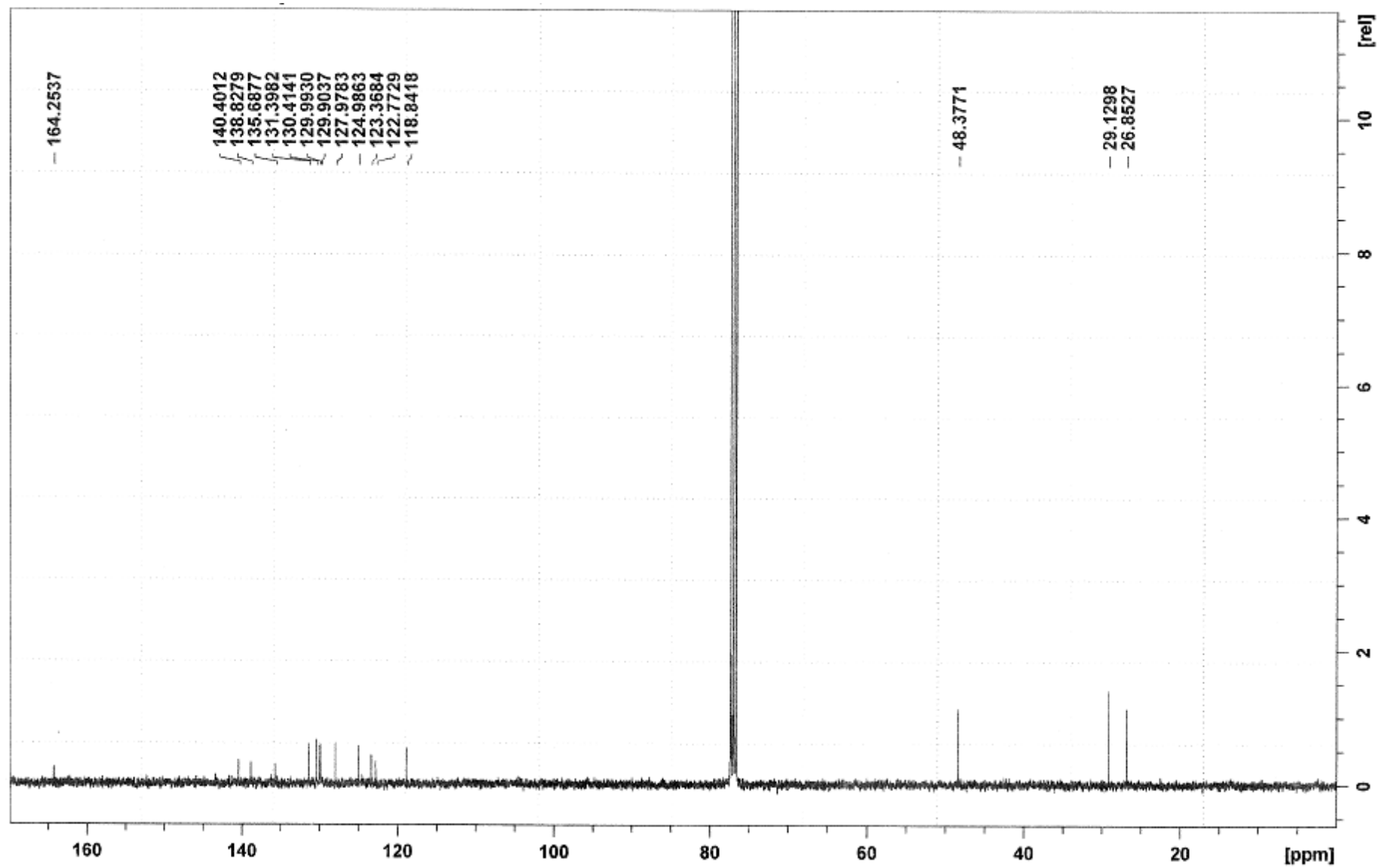


Compound **3cb**

$^1\text{H}$ -NMR

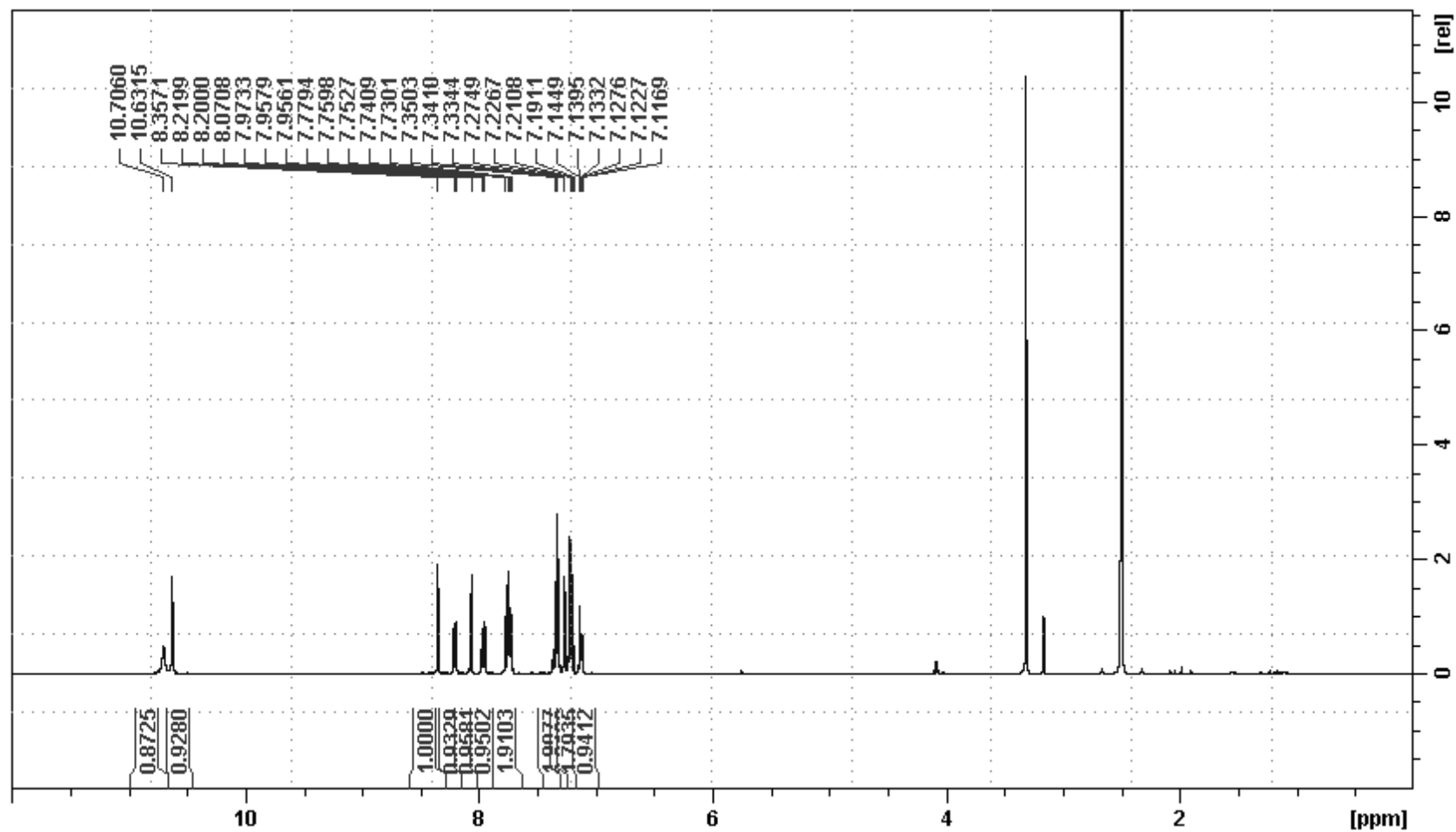


<sup>13</sup>C-NMR

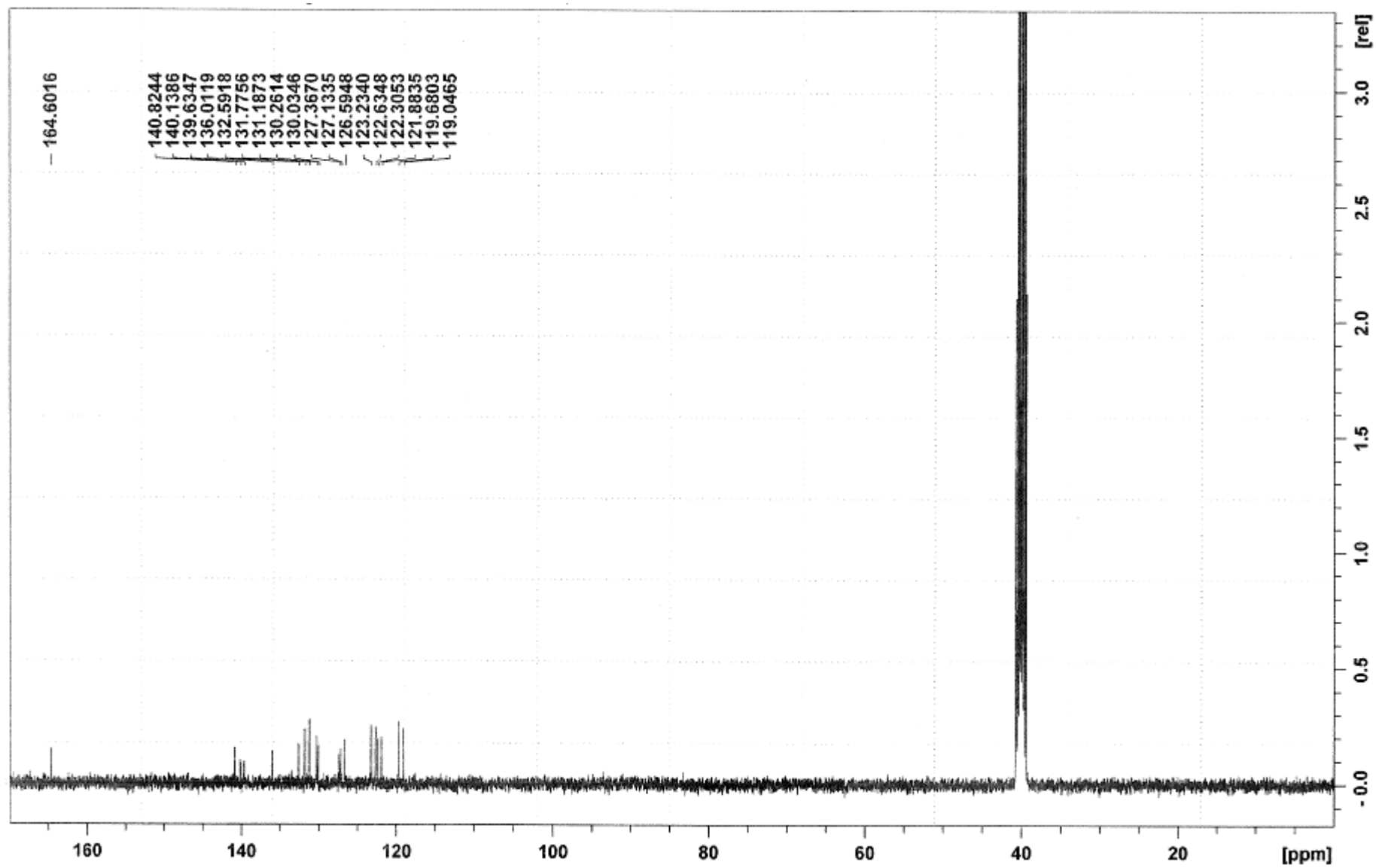


Compound **3cc**

$^1\text{H}$ -NMR

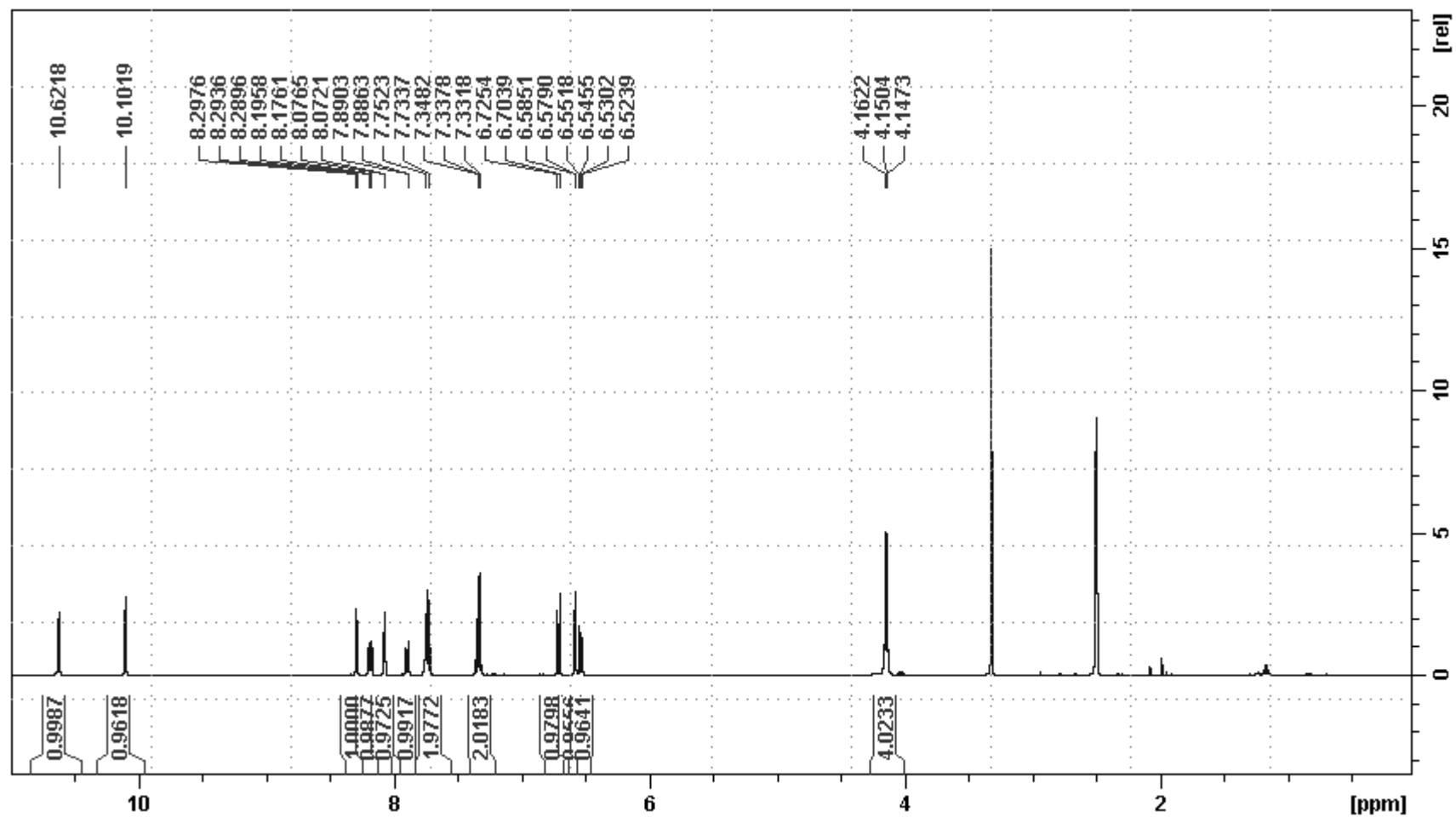


$^{13}\text{C}$ -NMR

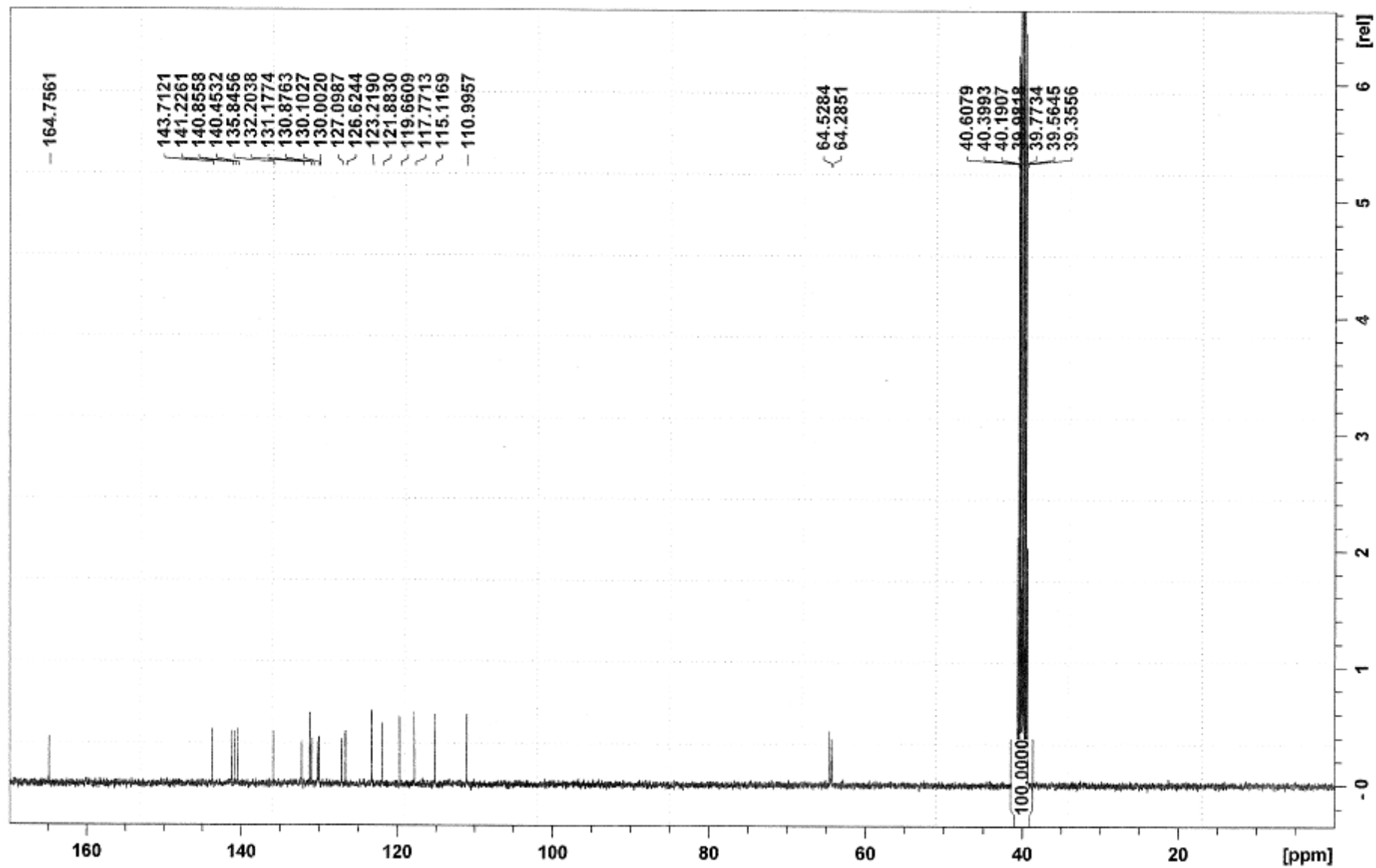


Compound **3ce**

<sup>1</sup>H-NMR

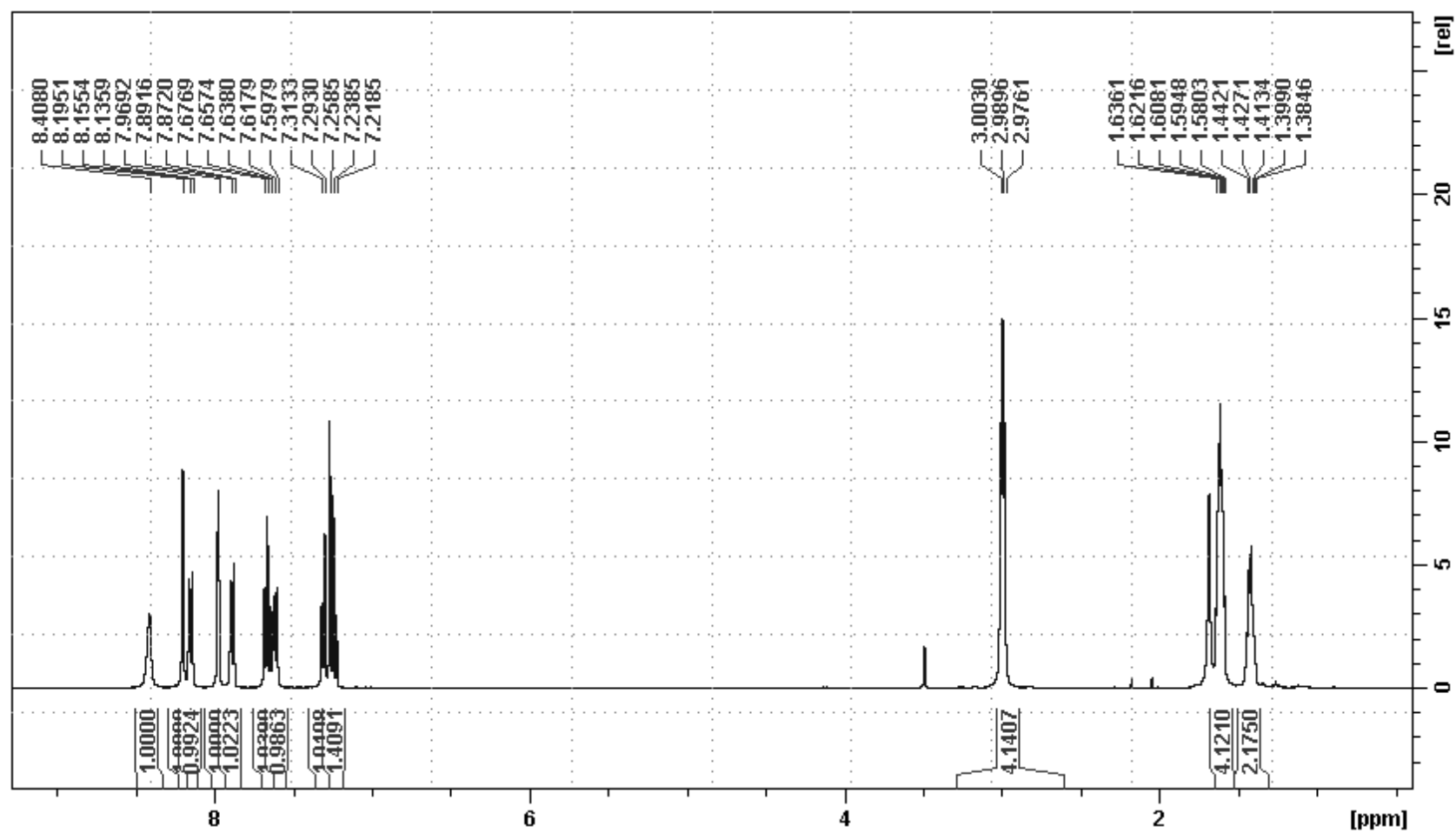


$^{13}\text{C}$ -NMR

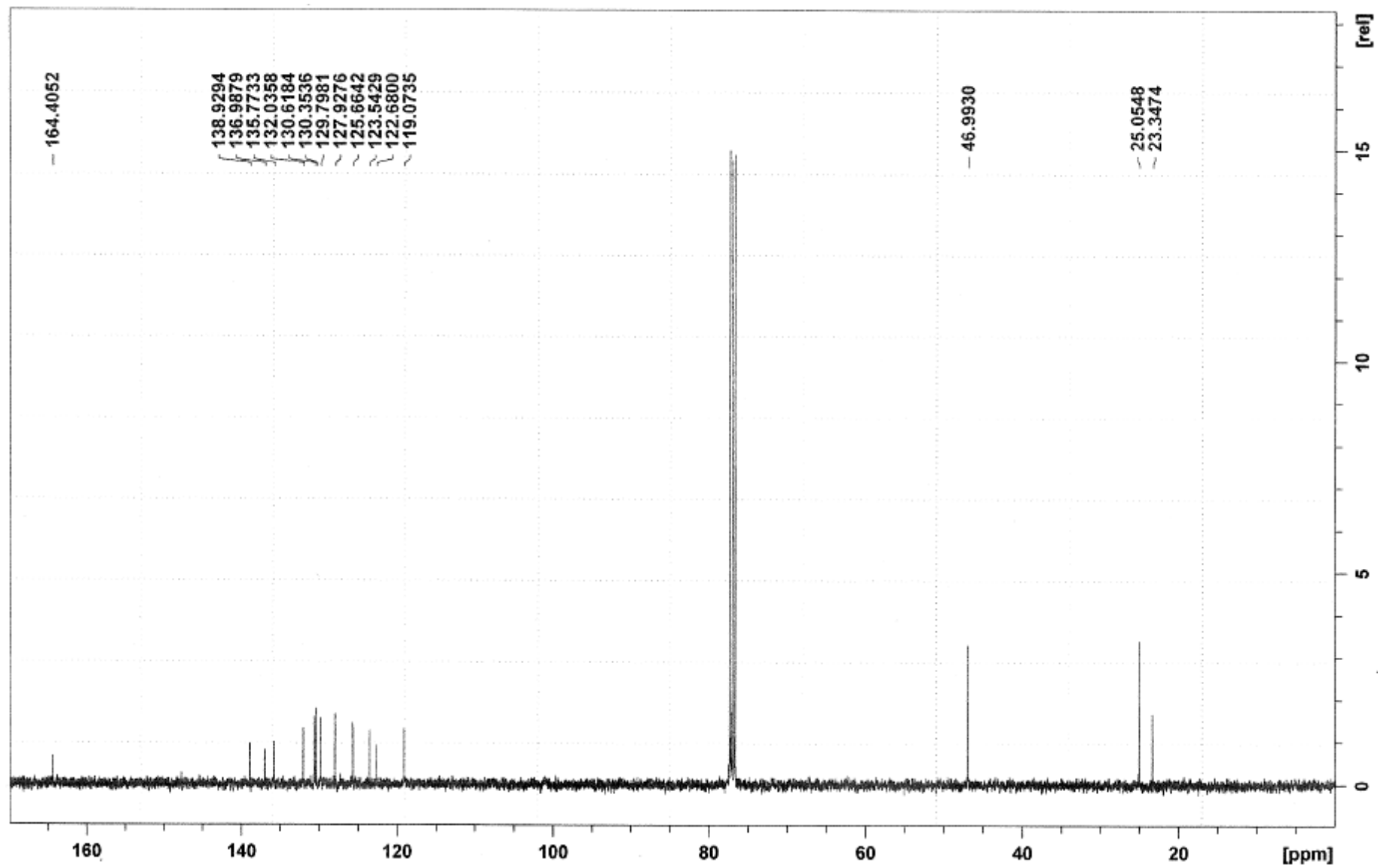


Compound **3cg**

$^1\text{H}$ -NMR



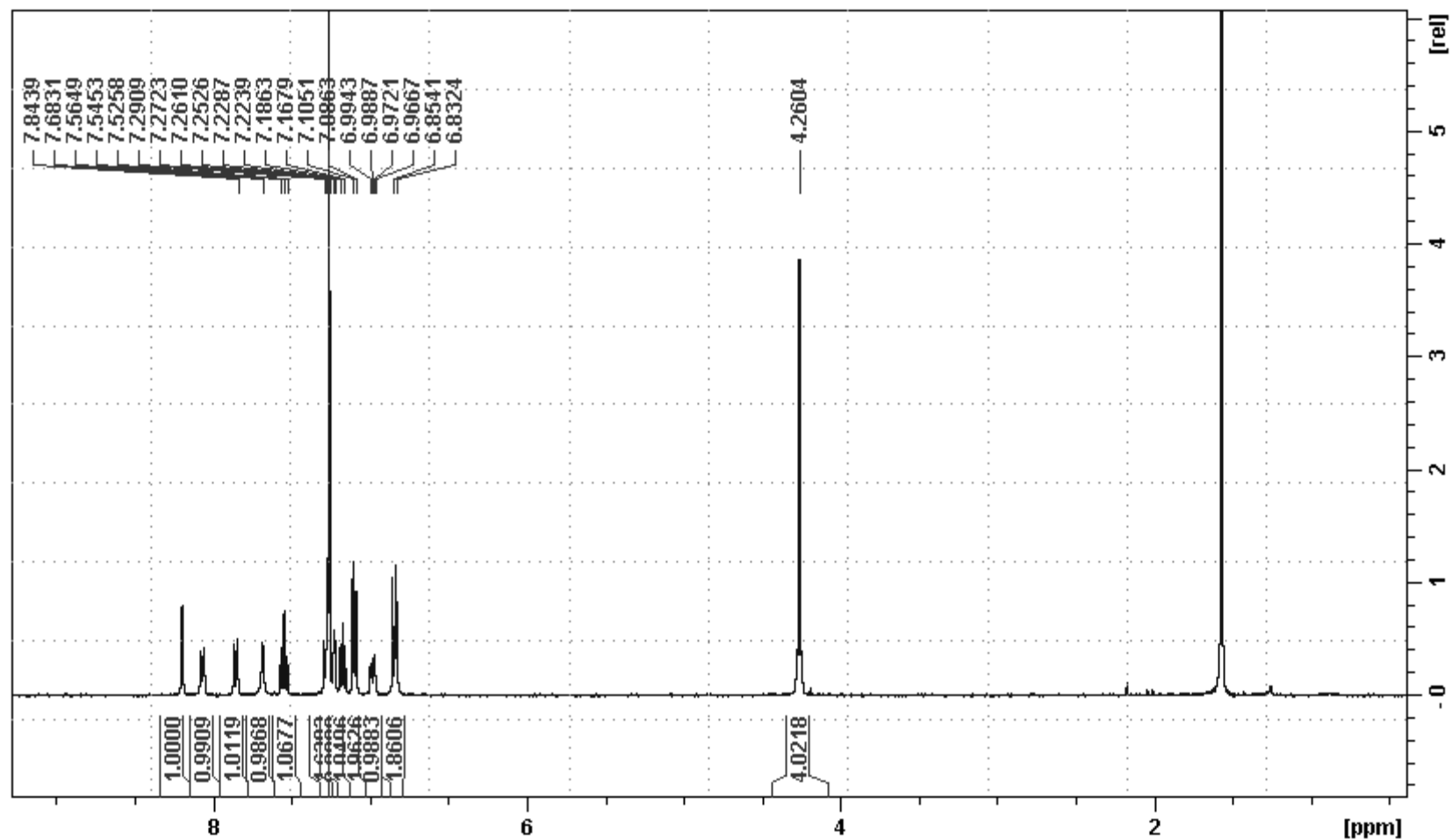
$^{13}\text{C}$ -NMR



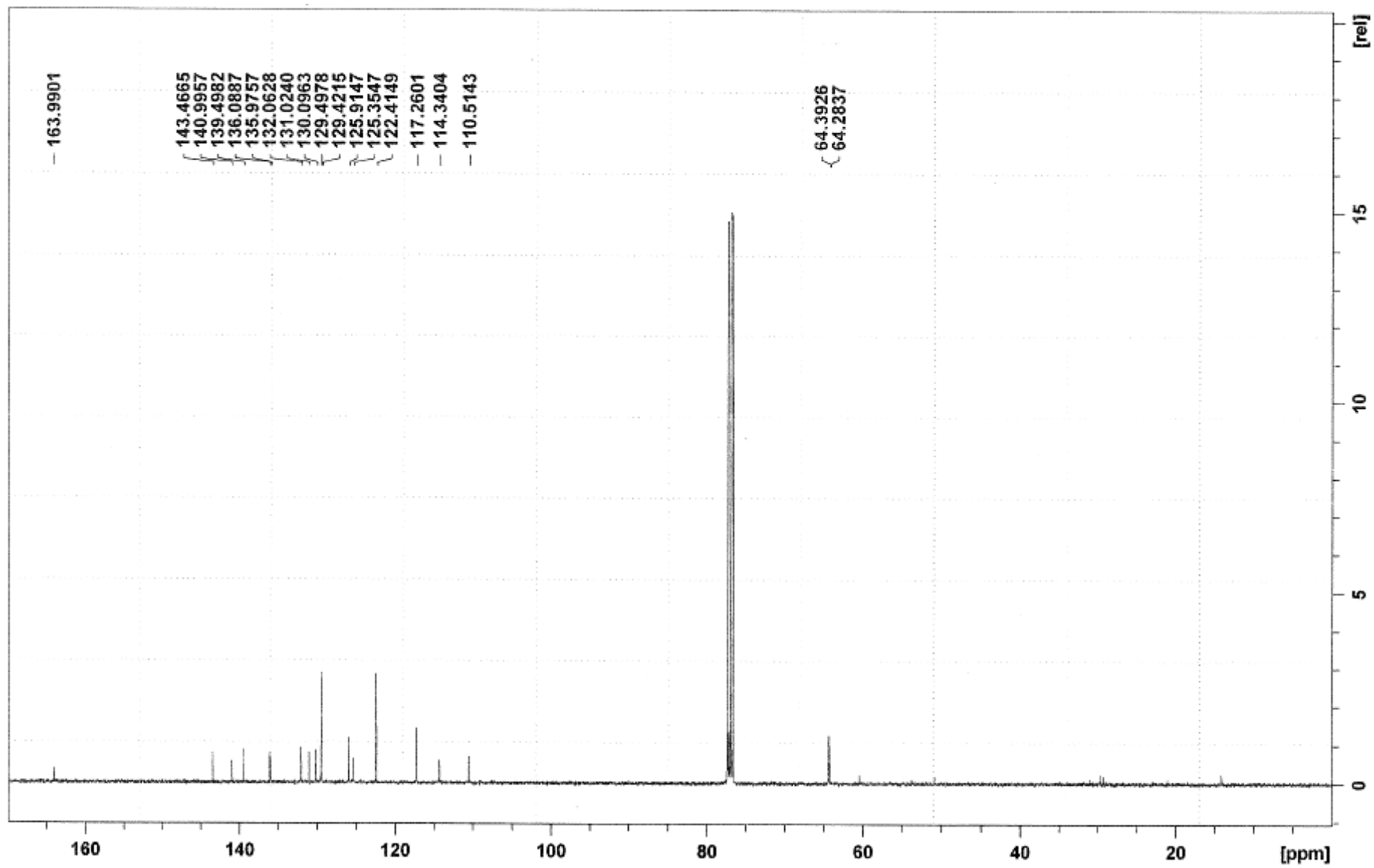


Compound **3ea**

$^1\text{H}$ -NMR

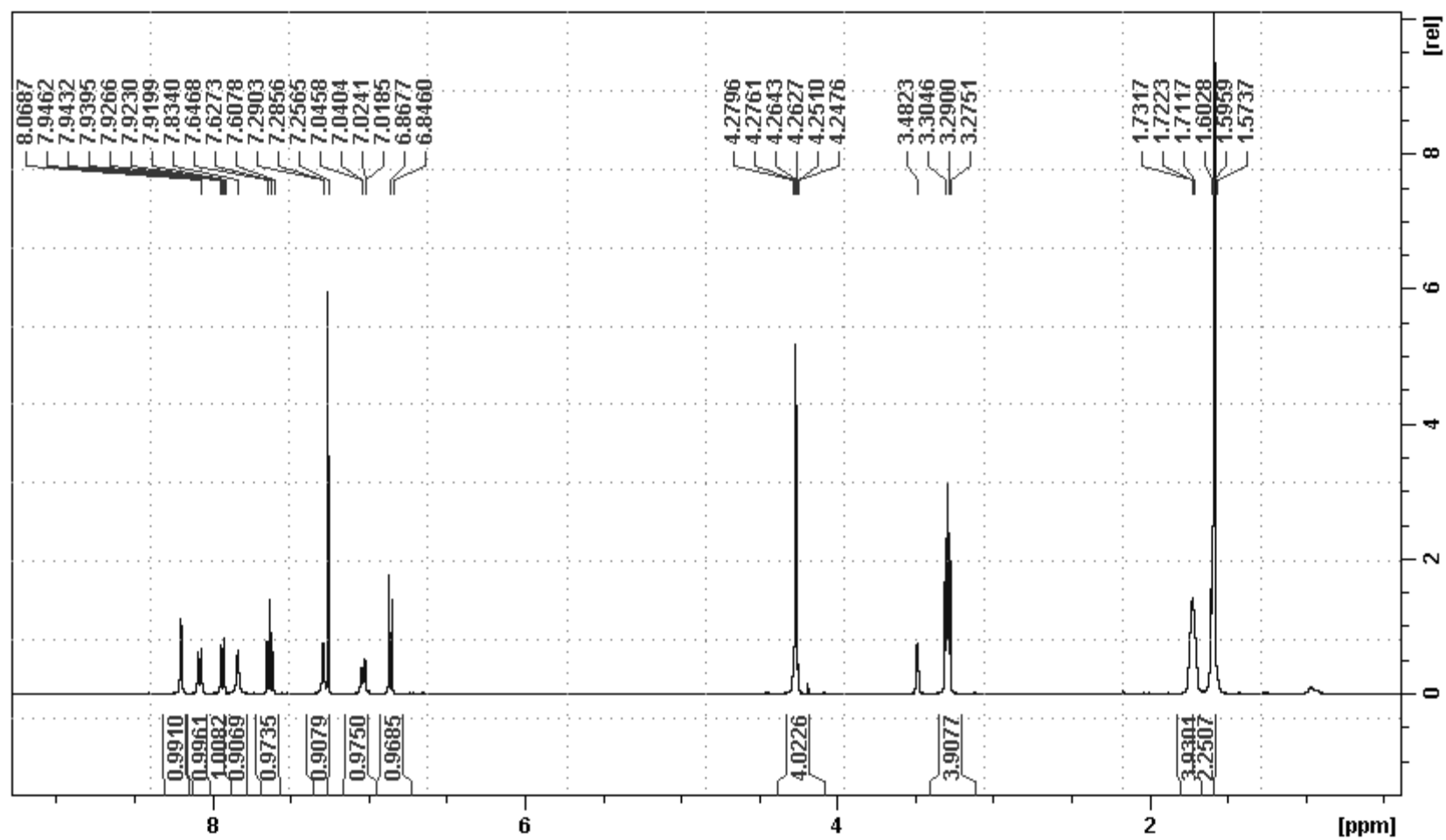


$^{13}\text{C}$ -NMR

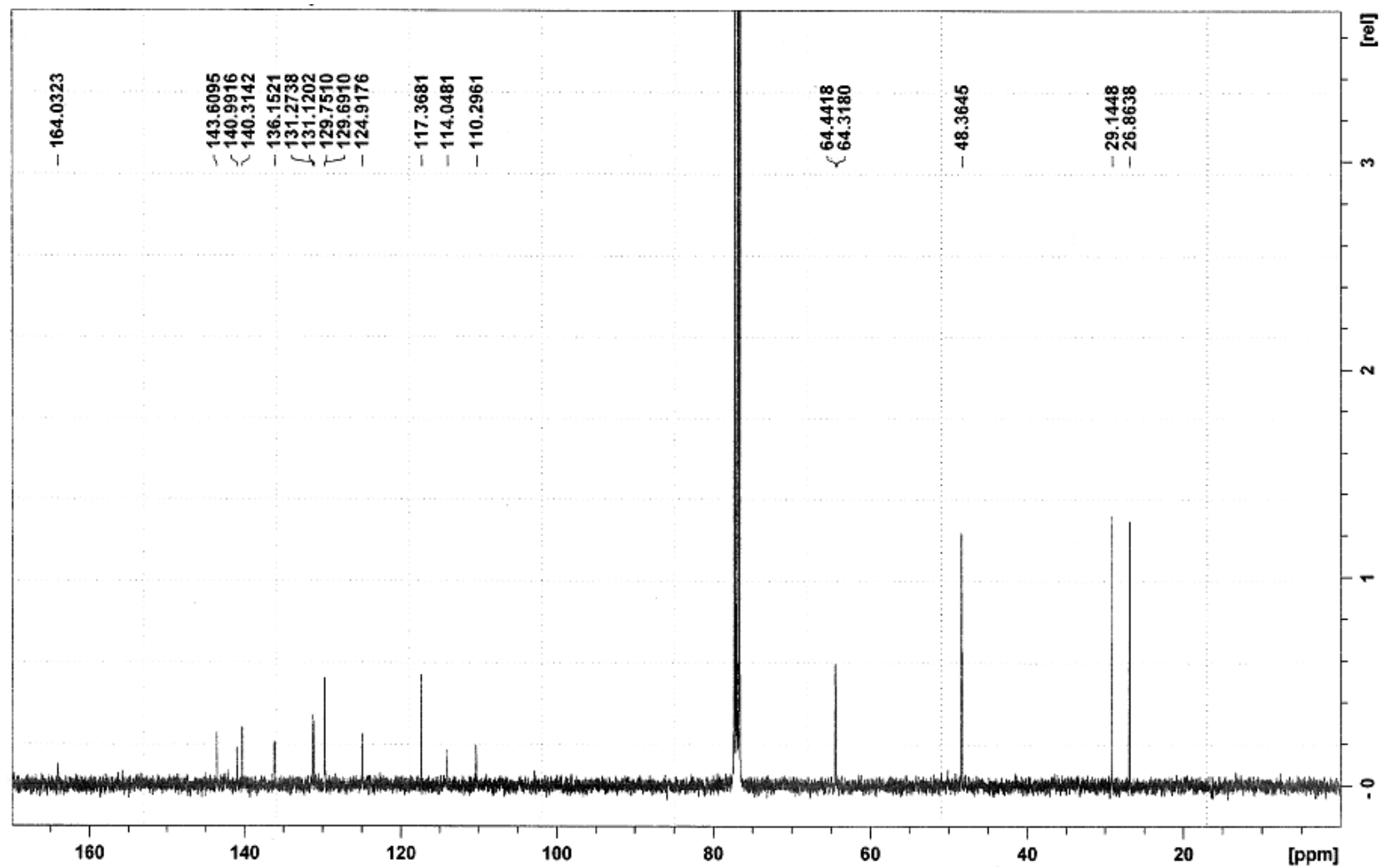


Compound **3eb**

<sup>1</sup>H-NMR

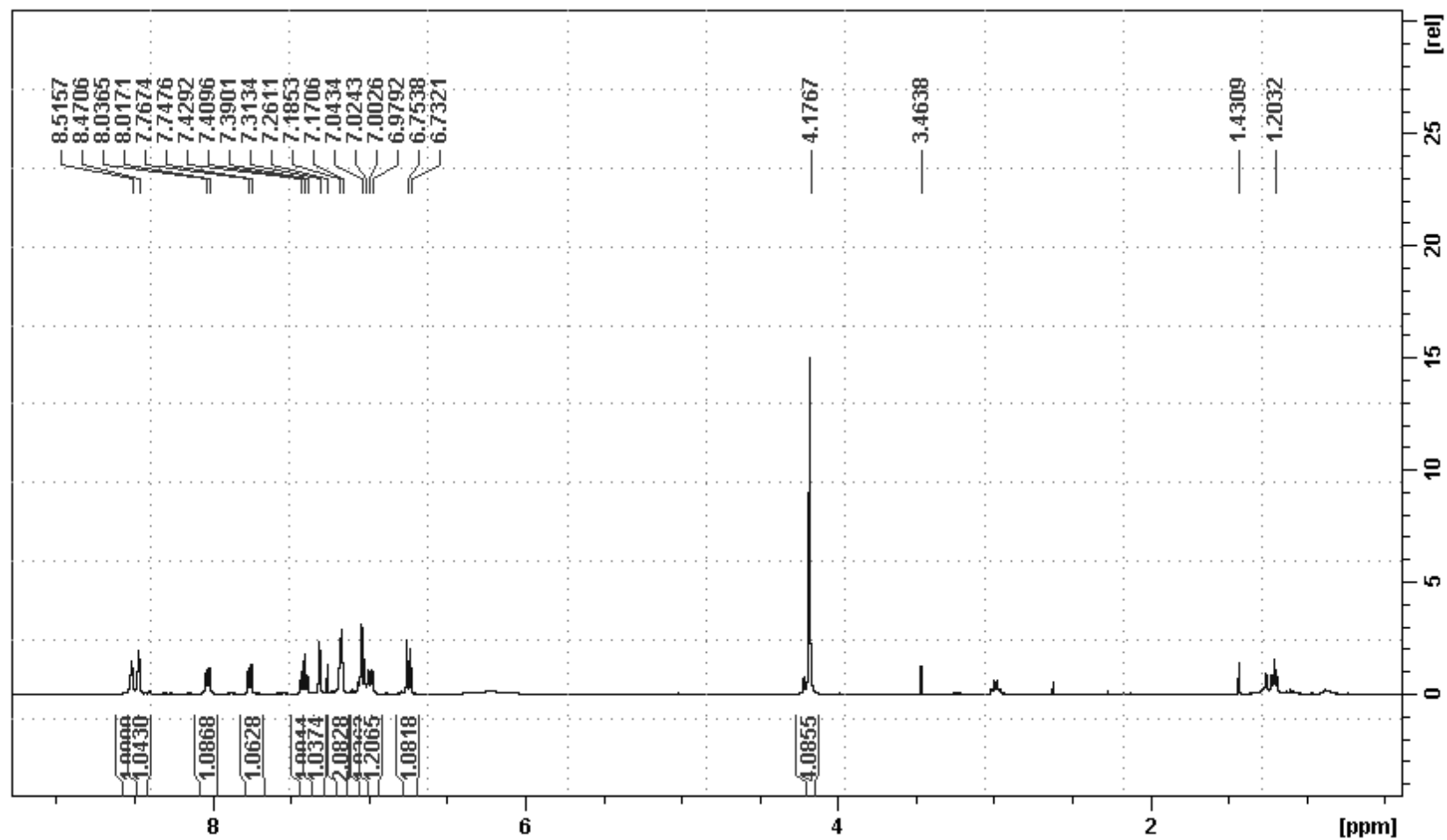


<sup>13</sup>C-NMR

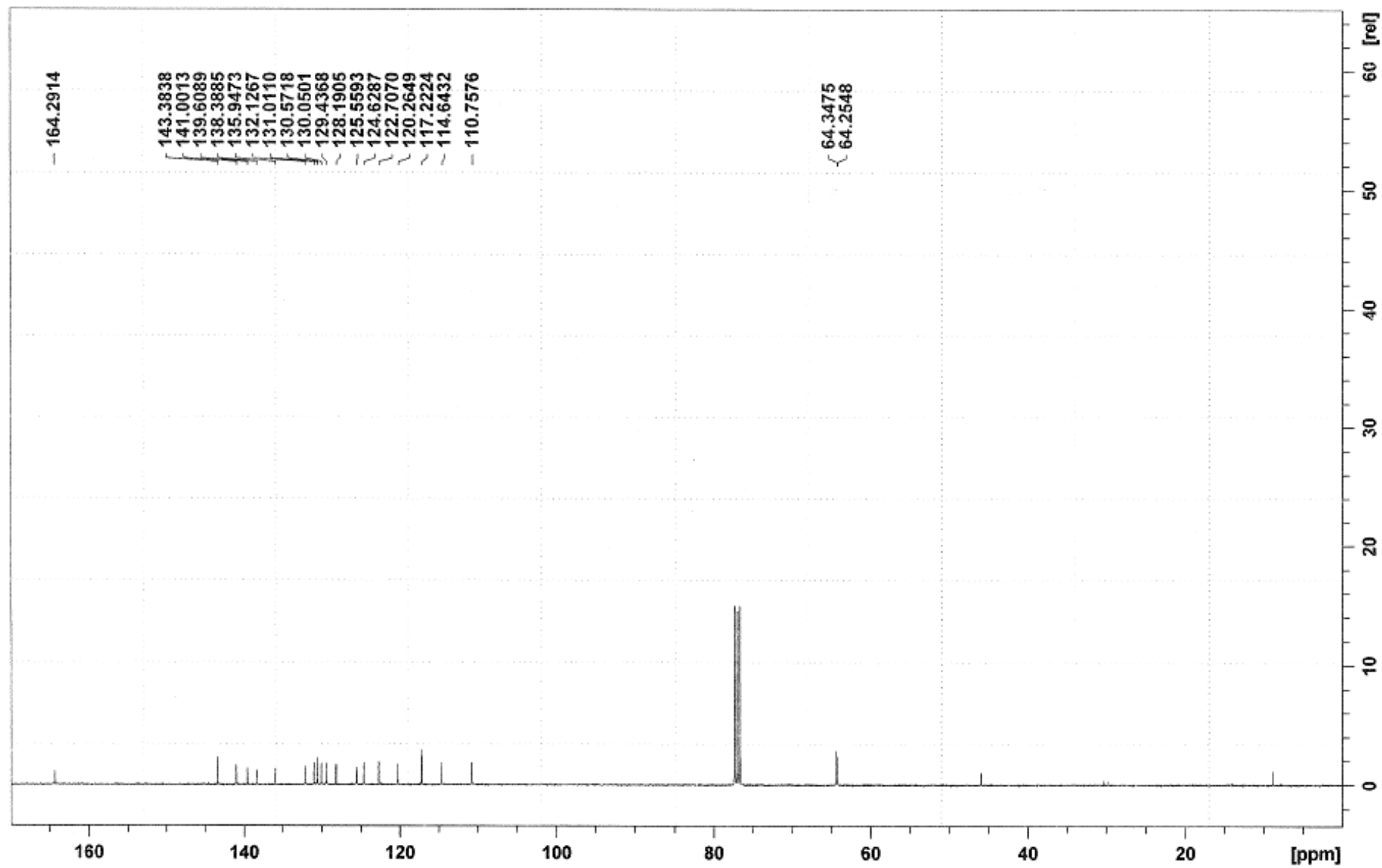


Compound **3ec**

$^1\text{H}$ -NMR

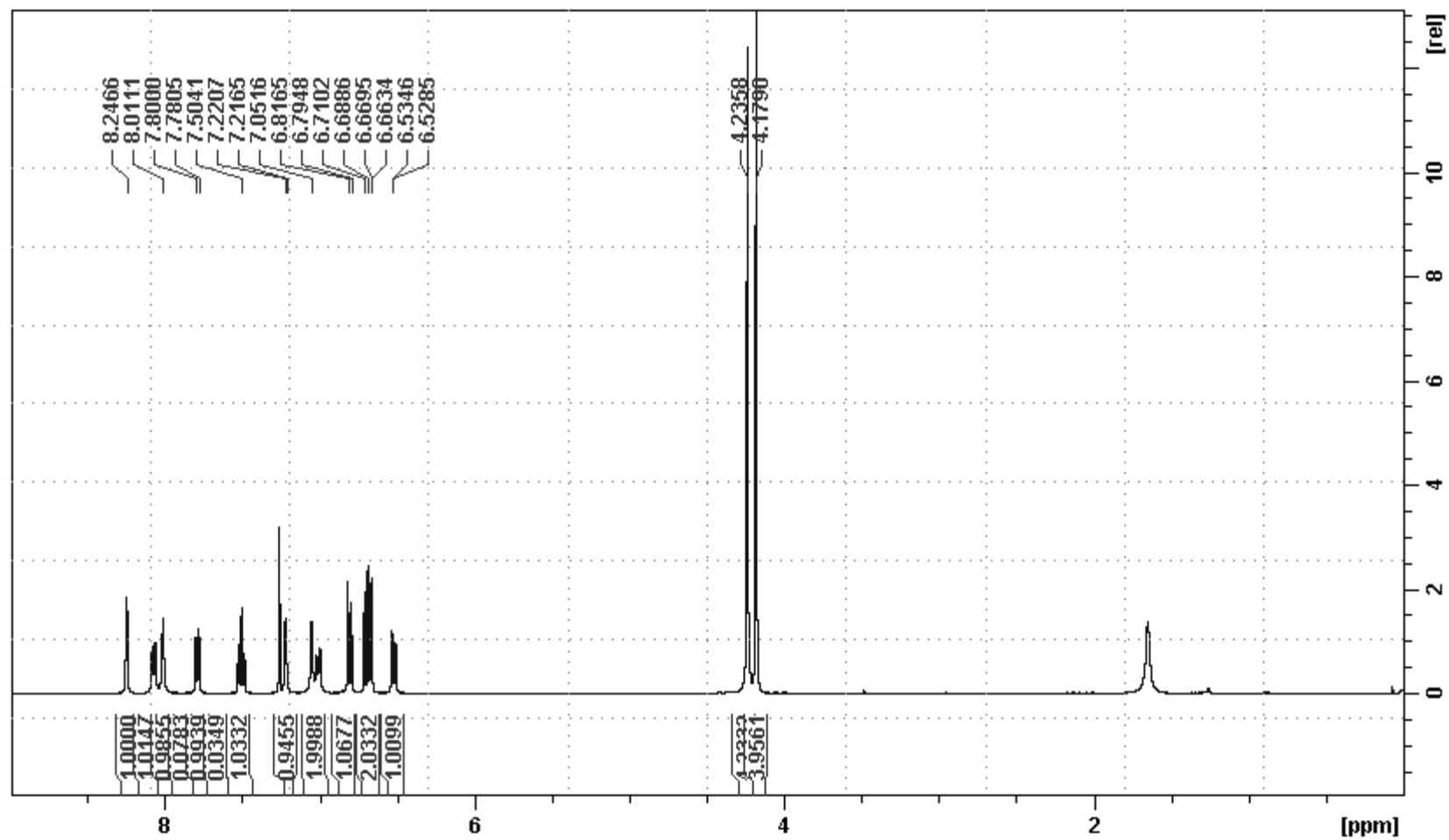


<sup>13</sup>C-NMR

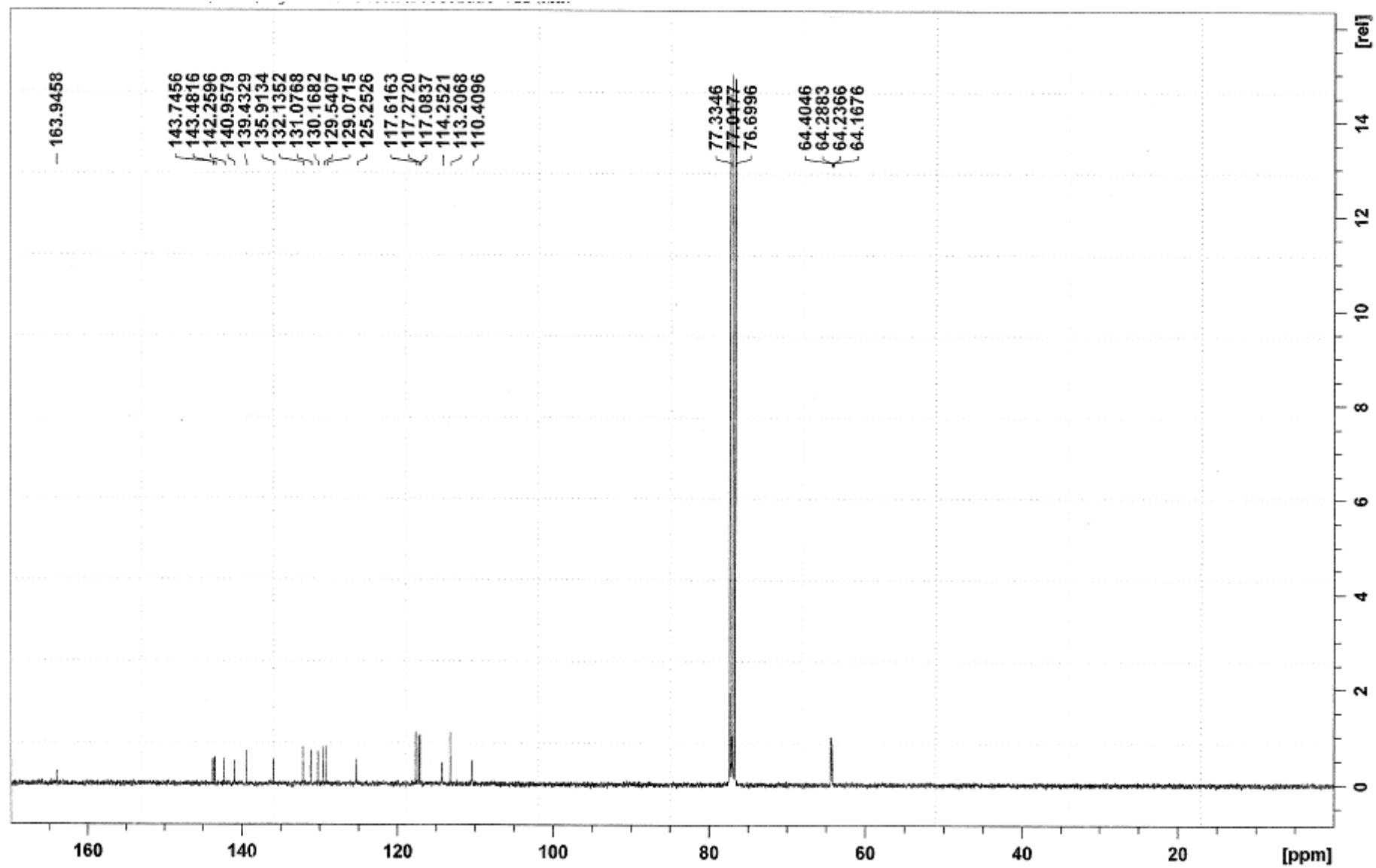


Compound **3ee**

$^1\text{H}$ -NMR



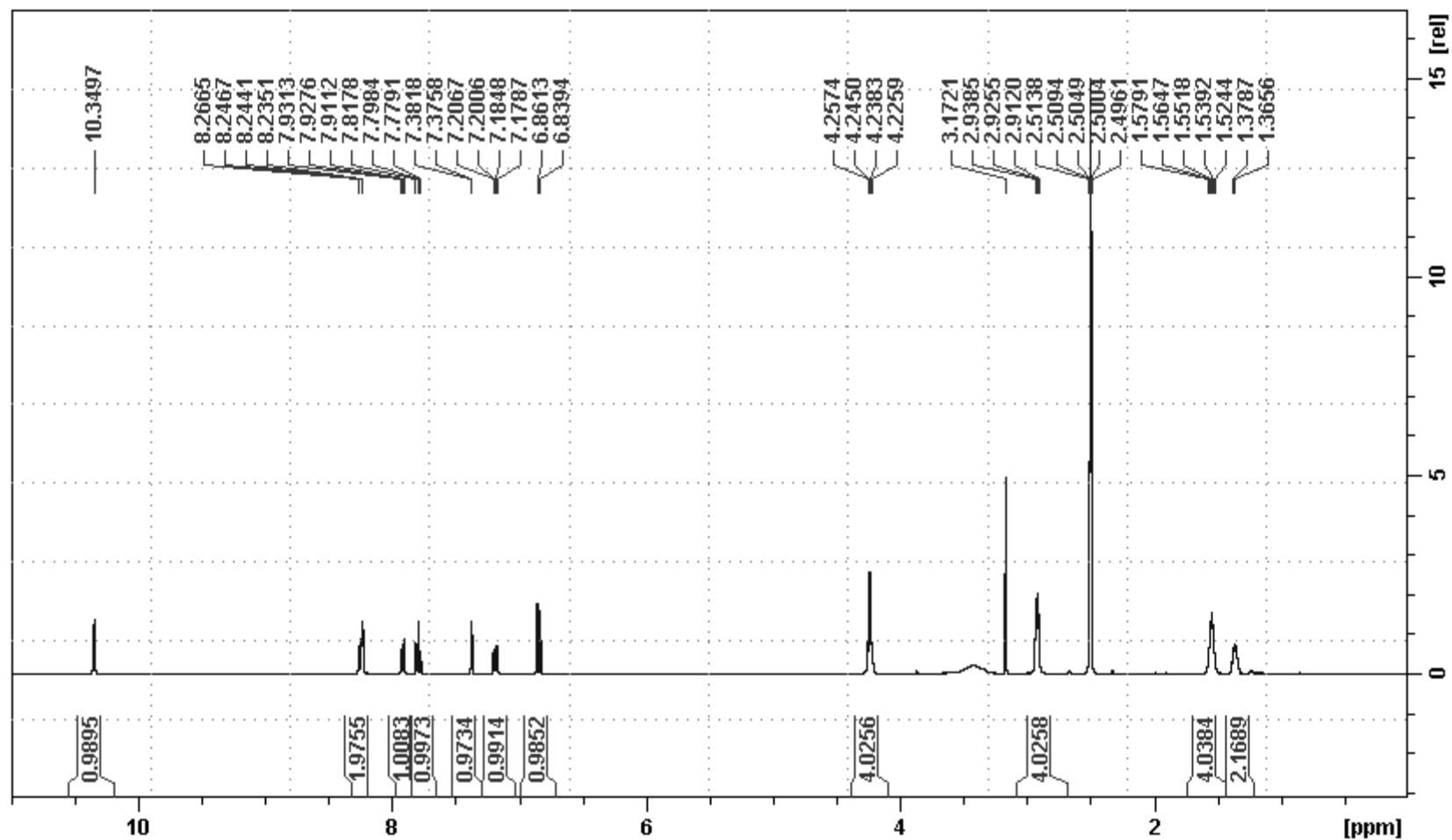
$^{13}\text{C}$ -NMR





Compound **3eg**

$^1\text{H}$ -NMR



<sup>13</sup>C-NMR

