Electronic Supplementary Information

Graphene Oxide (GO)–catalyzed Multi–component Reactions: Green Synthesis of Library of pharmacophore 3–Sulfenylimidazo[1,2–a]pyridines

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S1. Procedure for the preparation of Graphene Oxide (GO).

GO was prepared according to the modified Hummers method.[1a-b]

To an ice-cold concentrated sulfuric acid (46 mL) was slowly added 0.1 g of sodium nitrate portion-wise and then graphite powder (2 g) with vigorous magnetic stirring. After the complete addition of graphite powder, potassium permanganate (6 g) was added to the reaction mixture very slowly, maintaining the temperature within 0–5 ^{o}C to avoid any possible explosion. The mixture was allowed to stir at room temperature for 6 h forming a thick paste. It was diluted with distilled water (92 mL) under stirred condition. The temperature of the solution was then raised to about 90 ^{o}C and the mixture was allowed to stir for another 30 min. Finally, water (280 mL) was added followed by slow addition of H_2O_2 (30%, 3 mL). The colour of the solution gradually changes from dark brown to yellowish brown. The overall solution was exfoliated under sonication for about 30 min followed by centrifuged at 15000 rpm to collect the solid mass at the bottom. This process was continued for several times until the pH of the supernatant aqueous part becomes neutral (using pH paper). Finally, the brown mass was collected and dried at 60 ^{o}C under vacuum to obtain solid graphene oxide (GO). A comparable literature data,[1c] were obtained when characterized by FT-IR spectroscopy: ν_{max}(KBr): 3359, 1719, 1618, 1411, 1218, 1052 cm^{-1}.

S2. NMR Spectral data and scanned copies for compounds in Table 2; entries (5a – 5i) & Table 3; entries (8a – 8r):

Table 2:

2-Phenyl*H*-imidazo[1,2-*a*]pyridine (5a): Pale yellow solid, mp 134-136 ^{o}C (lit.[2] 135 ^{o}C). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.60 (t, *J* = 6.6 Hz, 1H), 7.02-7.07 (m, 1H), 7.26-7.31 (m, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.56 (d, *J* = 9.0 Hz, 1H), 7.68 (s, 1H), 7.89-7.93 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 107.9, 112.0, 117.0, 124.4, 125.3, 125.7, 127.6, 128.4, 133.5, 145.3.

2-(4-Ethylphenyl)*H*-imidazo[1,2-*a*]pyridine (5b): Brown solid, mp 115-117 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 1.25 (t, J = 7.5 Hz, 3H), 2.66 (q, *J* = 7.5 Hz, 2H), 6.64 (t, *J* = 6.6 Hz, 1H), 7.06-7.07 (m, 1H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.58 (d, *J* = 9.0 Hz, 1H), 7.70 (s, 1H), 7.84 (d, *J* = 8.1 Hz, 2H), 7.94-7.97 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 15.5, 28.7, 107.8, 112.2, 117.2, 124.5, 125.6, 126.0, 128.2, 131.1, 144.1, 145.5, 145.8.

2-(4-Bromophenyl)*H*-imidazo[1,2-*a*]pyridine (5c): Pale yellow solid, mp 215-217 ^{o}C (lit.[3] 216 ^{o}C). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.75-6.80 (m, 1H), 7.15-7.20 (m, 1H), 7.52-7.64 (m, 3H), 7.78-7.82

(m, 3H), 8.07-8.09 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 108.2, 112.7, 117.4, 121.9, 125.1, 125.6, 127.5, 131.8, 132.5, 144.4, 145.5.

2-(4-Iodophenyl)*H*-imidazo[1,2-*a*]pyridine (5d): White solid, mp 225-227 ^{o}C (lit.[4] 227 ^{o}C). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.75-6.80 (m, 1H), 7.14-7.20 (m, 1H), 7.60-7.85 (m, 6H), 8.08-8.10 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 93.4, 108.3, 112.6, 117.6, 124.9, 125.6, 127.8, 133.3, 137.8, 144.7, 145.7.

2-(3-Bromophenyl)*H*-imidazo[1,2-*a*]pyridine (5e): Pale yellow solid, mp 135-137 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.70-6.75 (m, 1H), 7.11-7.16 (m, 1H), 7.22-7.28 (m, 1H), 7.39-7.43 (m, 1H), 7.59 (d, J = 9.0 Hz, 1H), 7.76-7.83 (m, 2H), 8.02-8.10 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 108.4, 112.6, 117.3, 122.8, 124.4, 124.9, 125.6, 128.8, 130.1, 130.6, 135.7, 143.9, 145.5.

2-(2-Bromophenyl)*H*-imidazo[1,2-*a*]pyridine (5f): Pale yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.77-6.80 (m, 1H), 7.15-7.20 (m, 2H), 7.39-7.44 (m, 1H), 7.63-7.68 (m, 2H), 8.13-8.16 (m, 2H), 8.27 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 112.0, 112.6, 117.6, 121.5, 124.9, 125.8, 127.6, 128.9, 131.7, 133.7, 134.3, 143.1, 144.5.

2-(4-Ethylphenyl)-5-methyl*H*-imidazo[1,2-*a*]pyridine (5g): Brown liquid. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 1.27 (t, J = 7.5 Hz, 3H), 2.59 (s, 3H), 2.69 (q, J = 7.5 Hz, 2H), 6.58-6.60 (m, 1H), 7.10-7.15 (m, 1H), 7.26-7.28 (m, 2H), 7.55 (d, J = 9.0 Hz, 1H), 7.69 (s, 1H), 7.89-7.92 (m, 2H) .^{13}C NMR (75 MHz, $CDCl_3$), δ: 15.4, 18.7, 28.7, 104.9, 111.5, 114.8, 124.7, 126.1, 128.2, 131.3, 134.3, 144.1, 145.9, 146.1.

6-Chloro-2-(4-chlorophenyl)*H*-imidazo[1,2-*a*]pyridine (5h): Pale yellow solid, mp 204-206 ^{o}C (lit.[5] 209 ^{o}C). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 7.13-7.17 (m, 1H), 7.37-7.41 (m, 2H), 7.57 (d, J = 9.6 Hz, 1H), 7.78 (s, 1H), 7.82-7.86 (m, 2H), 8.14 (s, 1H).^{13}C NMR (75 MHz, $CDCl_3$), δ: 108.5, 117.7, 120.9, 123.4, 126.5, 127.3, 129.0, 131.6, 134.1, 143.9, 145.5.

2-(3-Bromophenyl)-5-methyl*H*-imidazo[1,2-*a*]pyridine (5i): Pale yellow solid, mp 106-108 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 2.49 (s, 3H), 6.61-6.63 (m, 1H), 7.13-7.18 (m, 1H), 7.26-7.31 (m, 1H), 7.42-7.45 (m, 1H), 7.53 (d, J = 9.0 Hz, 1H), 7.71 (s, 1H), 7.88-7.91 (m, 1H), 8.13-8.15 (m, 1H).^{13}C NMR (75 MHz, $CDCl_3$), δ: 18.7, 105.7, 111.8, 115.0, 122.9, 124.6, 125.2, 129.0, 130.2, 130.7, 134.5, 136.1, 144.2, 146.2.

Table 3:

2-Phenyl-3-(phenylthio)*H*-imidazo[1,2-*a*]pyridine (8a): Pale yellow solid, mp 106-108 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.81-6.86 (m, 1H), 6.97-7.09 (m, 2H), 7.11-7.25 (m, 3H), 7.28-7.46 (m, 4H),

7.73 (d, *J* = 9.0 Hz, 1H), 8.19-8.27 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 106.4, 113.1, 117.6, 124.5, 125.6, 126.1, 126.7, 128.4, 128.5, 128.6, 129.5, 133.3, 135.2, 147.1, 151.4.

3-(*p*-Tolylthio)-2-phenyl*H*-imidazo[1,2-*a*]pyridine (8b): Pale yellow solid, mp 141-143 °C (lit.[6] 142-143 °C). ^{1}H NMR (300 MHz, $CDCl_3$), δ: 2.22 (s, 3H), 6.78-7.00 (m, 5H), 7.23-7.44 (m, 4H), 7.70 (d, *J* = 9.0 Hz, 1H), 8.20-8.25 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 20.8, 106.8, 112.9, 117.5, 124.4, 125.7, 126.5, 128.3, 128.5, 130.1, 131.4, 133.3, 135.9, 146.9, 151.0.

3-(4-Methoxyphenylthio)-2-phenyl*H*-imidazo[1,2-*a*]pyridine (8c): Pale yellow solid, mp 124-126 °C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 3.67 (s, 3H), 6.71-6.75 (m, 2H), 8.82-6.85 (m, 1H), 6.96-6.99 (m, 2H), 7.25-7.31 (m, 1H), 7.36-7.47 (m, 3H), 7.70 (d, *J* = 9.3 Hz, 1H), 8.24-8.30 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 55.2, 107.8, 112.9, 115.1, 117.5, 124.4, 125.4, 126.4, 128.0, 128.3, 128.4, 128.5, 133.4, 146.7, 150.7, 158.5.

3-(4-Chlorophenylthio)-2-phenyl*H*-imidazo[1,2-*a*]pyridine (8d): Pale yellow solid, mp 161-163 °C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.86-6.93 (m, 3H), 7.16-7.47 (m, 6H), 7.74 (d, *J* = 9.0 Hz, 1H), 8.16-8.24 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 105.8, 113.3, 117.8, 124.3, 126.8, 126.9, 128.3, 128.5, 128.8, 129.6, 132.1, 133.1, 133.7, 147.2, 151.5.

3-(2,5-Dimethylphenylthio)-2-(4-ethylphenyl)*H*-imidazo[1,2-*a*]pyridine (8e): Pale yellow solid, mp 131-133 °C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 1.22 (t, *J* = 7.5 Hz, 3H), 1.97 (s, 3H), 2.44 (s, 3H), 2.64 (q, *J* = 7.5 Hz, 2H), 6.24 (s, 1H), 6.71-6.81 (m, 2H), 7.03 (d, *J* = 8.4 Hz, 1H), 7.20-7.26 (m, 3H), 7.70 (d, *J* = 9.0 Hz, 1H), 8.12-8.14 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 15.5, 19.4, 21.1, 28.8, 105.4, 112.9, 117.5, 124.5, 124.6, 126.5, 126.6, 128.0, 128.4, 130.6, 130.9, 131.9, 133.8, 136.7, 144.8, 147.2, 151.8.

3-(2-Methoxyphenylthio)-2-(4-ethylphenyl)*H*-imidazo[1,2-*a*]pyridine (8f): Brown solid, mp 146-148 °C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 1.24 (t, *J* = 7.5 Hz, 3H), 2.66 (q, *J* = 7.5 Hz, 2H), 3.92 (s, 3H), 6.37-6.40 (m, 1H), 6.66-6.67 (m, 1H), 6.82-6.89 (m, 2H), 6.09-6.10 (m, 1H), 7.24-7.33 (m, 3H), 7.73 (d, *J* = 9.0 Hz, 1H), 8.11-8.25 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 15.4, 28.7, 55.8, 104.9, 110.8, 112.8, 117.4, 121.5, 123.3, 124.7, 125.5, 126.5, 126.8, 127.9, 128.3, 130.7, 144.7, 147.1, 151.7, 156.1.

2-(4-Methoxyphenyl)-3-(naphthalen-3-ylthio)*H*-imidazo[1,2-*a*]pyridine (8g): Pale yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.79-7.84 (m, 1H), 6.94-6.97 (m, 2H), 7.14-7.18 (m, 1H), 7.29-7.40 (m, 4H), 7.55-7.58 (m, 1H), 7.68-7.77 (m, 3H), 8.18-8.28 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 55.2, 105.2, 113.1, 113.9, 117.3, 123.3, 123.8, 124.4, 125.6, 125.7, 126.7, 126.8, 126.9, 127.7, 129.2, 129.7, 131.7, 132.6, 133.8, 146.9, 151.2, 160.1.

3-(*p*-tolylthio)-2-(naphthalen-3-yl)*H*-imidazo[1,2-*a*]pyridine (8h): Pale yellow solid, mp 134-136 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.80-6.85 (m, 1H), 6.96-7.04 (m, 4H), 7.27-7.33 (m, 1H), 7.46-7.51 (m, 2H), 7.76-7.95 (m, 4H), 8.30-8.32 (m, 1H), 8.45-8.48 (m, 1H), 8.80-8.81 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 20.7, 107.5, 112.9, 117.4, 124.4, 125.8, 125.9, 125.95, 125.98, 126.0, 126.2, 126.5, 127.5, 127.7, 128.6, 130.1, 130.8, 131.4, 133.3, 136.0, 146.9, 150.8.

3-(4-Fluorophenylthio)-2-*p*-tolylH-imidazo[1,2-*a*]pyridine (8i): Pale yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 3.38 (s, 3H), 6.81-6.87 (m, 1H), 6.89-6.92 (m, 2H), 6.95-7.00 (m, 2H), 7.25-7.33 (m, 3H), 7.71 (d, J = 9.0 Hz, 1H), 8.13-8.15 (m, 2H), 8.23-8.26 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 21.4, 106.3, 113.1, 116.4, 116.7, 117.6, 124.3, 126.7, 127.6, 127.7, 128.2, 129.2, 130.2, 130.3, 130.4, 138.6, 147.0, 151.4, 159.9, 163.1.

3-(4-Chlorophenylthio)-2-(4-methoxyphenyl)*H*-imidazo[1,2-*a*]pyridine (8j): Pale yellow solid, mp 143-145 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 3.81 (s, 3H), 6.81-6.98 (m, 5H), 7.12-7.16 (m, 2H), 7.27-7.33 (m, 1H), 7.70 (d, J = 8.7 Hz, 1H), 8.14-8.21 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 55.1, 104.6, 112.9, 113.8, 117.3, 124.1, 125.6, 126.6, 126.7, 129.4, 129.5, 131.8, 133.8, 147.0, 151.3, 160.0.

3-(4-Chlorophenylthio)-2-(3,4-dimethoxyphenyl)*H*-imidazo[1,2-*a*]pyridine (8k): Pale yellow solid, mp 156-158 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 3.89 (s, 3H), 3.90 (s, 3H), 6.85-6.94 (m, 4H), 7.15-7.18 (m, 2H), 7.28-7.36 (m, 1H), 7.70-7.82 (m, 3H), 8.24-8.26 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 55.8, 104.9, 111.0, 111.4, 113.0, 117.4, 120.9, 124.1, 125.9, 126.6, 126.7, 129.5, 132.0, 133.9, 147.0, 148.8, 149.6, 151.3.

3-(2-Bromophenylthio)-2-*p*-tolylH-imidazo[1,2-*a*]pyridine (8l): White solid, mp 159-161 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 2.34 (s, 3H), 6.33-6.36 (m, 1H), 6.80-6.83 (m, 1H), 6.93-6.96 (m, 2H), 7.21 (d, J = 8.1 Hz, 2H), 7.27-7.32 (m, 1H), 7.51-7.54 (m, 1H), 7.72 (d, J = 9.0 Hz, 1H), 8.07 (d, J = 8.4 Hz, 2H), 8.16 (d, J = 6.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 21.4, 105.0, 113.2, 117.6, 120.4, 124.4, 125.6, 126.9, 127.0, 128.2, 129.2, 130.2, 133.3, 136.4, 138.7, 147.4, 152.1.

3-(*p*-tolylthio)-2-(4-bromophenyl)*H*-imidazo[1,2-*a*]pyridine (8m): White solid, mp 147-149 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 2.24 (s, 3H), 6.82-6.89 (m, 3H), 6.99-7.01 (m, 2H), 7.25-7.34 (m, 1H), 7.53-7.56 (m, 2H), 7.70 (d, J = 9.0 Hz, 1H), 8.10-8.15 (m, 2H), 8.26 (d, J = 6.9 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 20.8, 107.1, 113.1, 117.5, 122.8, 124.4, 125.8, 126.8, 129.8, 130.2, 131.0, 131.5, 132.3, 136.2, 146.9, 149.7.

3-(*p*-tolylthio)-2-(4-iodophenyl)*H*-imidazo[1,2-*a*]pyridine (8n): Pale yellow solid, mp 134-136 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 2.24 (s, 3H), 6.82-6.89 (m, 3H), 6.99-7.02 (m, 2H), 7.25-7.34 (m, 1H),

7.69-7.76 (m, 3H), 7.97-8.00 (m, 2H), 8.24-8.27 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 20.8, 94.8, 107.2, 113.2, 117.5, 124.4, 125.8, 126.8, 129.9, 130.2, 131.0, 132.8, 136.2, 137.5, 146.8, 149.7.

3-(*p*-tolylthio)-2-(4-chlorophenyl)*H*-imidazo[1,2-*a*]pyridine (8o): Pale yellow solid, mp 133-135 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 2.25 (s, 3H), 6.84-6.90 (m, 3H), 7.01 (d, *J* = 8.1 Hz, 2H), 7.30-7.41 (m, 3H), 7.70-7.74 (m, 1H), 8.18-8.20 (m, 2H), 8.26-8.29 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 20.8, 107.1, 113.2, 117.5, 124.5, 125.8, 126.9, 128.6, 129.5, 130.2, 131.0, 131.7, 134.5, 136.2, 146.8, 149.6.

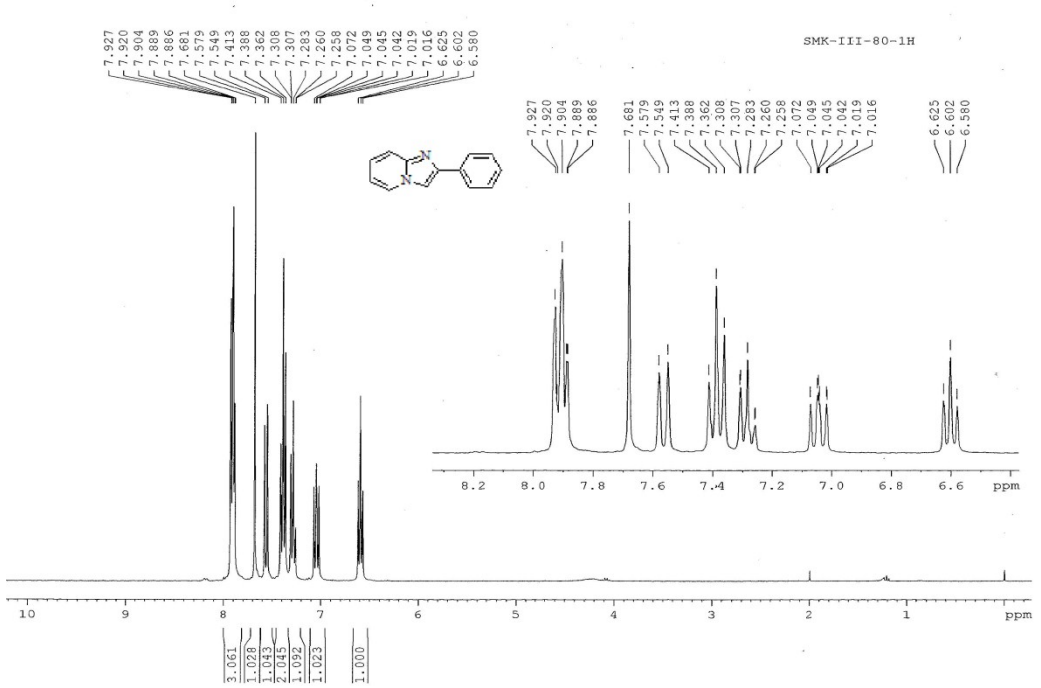
3-(4-Chlorothio)-2-(naphthalen-1-yl)*H*-imidazo[1,2-*a*]pyridine (8p): White solid, mp 208-210 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.82-6.95 (m, 3H), 7.10-7.14 (m, 2H), 7.36-7.60 (m, 5H), 7.80-7.92 (m, 3H), 8.17-8.17 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 108.7, 113.4, 117.9, 124.5, 124.9, 125.8, 126.1, 126.4, 126.8, 127.1, 128.2, 128.7, 129.2, 129.4, 130.4, 131.9, 132.2, 133.7, 133.8, 147.0, 152.7.

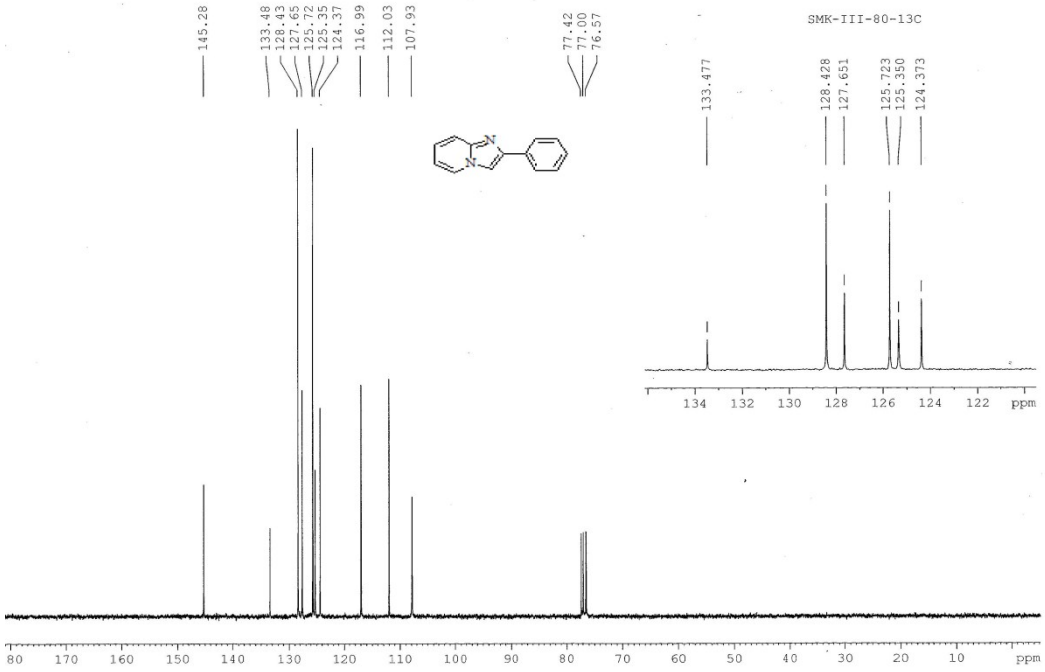
3-(2-Bromophenylthio)-2-(2-bromophenyl)*H*-imidazo[1,2-*a*]pyridine (8q): White solid, mp 185-187 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 6.31-6.34 (m, 1H), 6.85-6.99 (m, 3H), 7.23-7.56 (m, 4H), 7.74 (d, *J* = 9.0 Hz, 1H), 8.09-8.20 (m, 2H), 8.36-8.37 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 106.1, 113.5, 117.7, 120.5, 122.6, 124.4, 125.5, 126.6, 127.1, 127.2, 128.1, 129.8, 131.1, 131.5, 133.3, 135.0, 135.8, 147.2, 150.0.

3-(Pentylthio)-2-*p*-tolyl*H*-imidazo[1,2-*a*]pyridine (8r): Pale yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$), δ: 0.77 (t, *J* = 6.9 Hz, 3H), 1.10-1.28 (m, 4H), 1.42 (qnt, *J* = 7.5 Hz, 2H), 2.40 (s, 3H), 2.62 (t, *J* = 7.2 Hz, 2H), 6.86 (t, *J* = 6.9 Hz, 1H), 7.22-7.29 (m, 3H), 7.65 (d, *J* = 9.0 Hz, 1H), 8.22 (d, *J* = 8.1 Hz, 2H), 8.46-8.49 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$), δ: 13.7, 21.2, 22.0, 29.0, 30.6, 35.6, 109.9, 112.4, 117.3, 124.2, 125.7, 128.1, 128.9, 130.9, 137.9, 146.1, 149.4.

Scanned 1H & 13C NMR Spectra:

Table 2, 5a:





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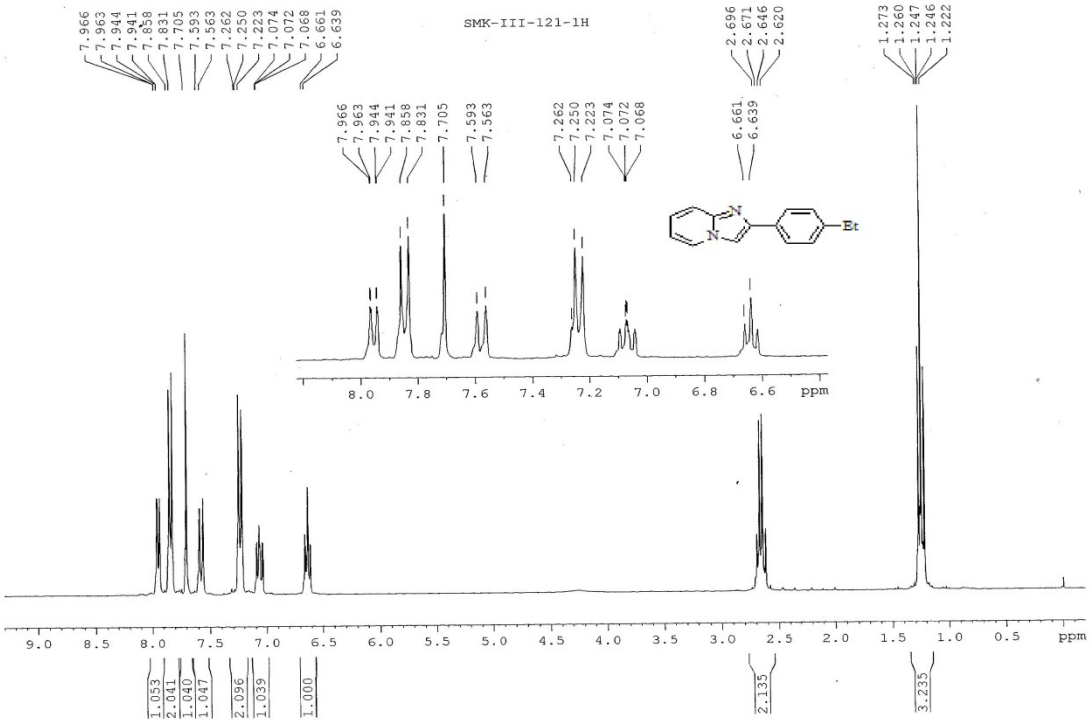
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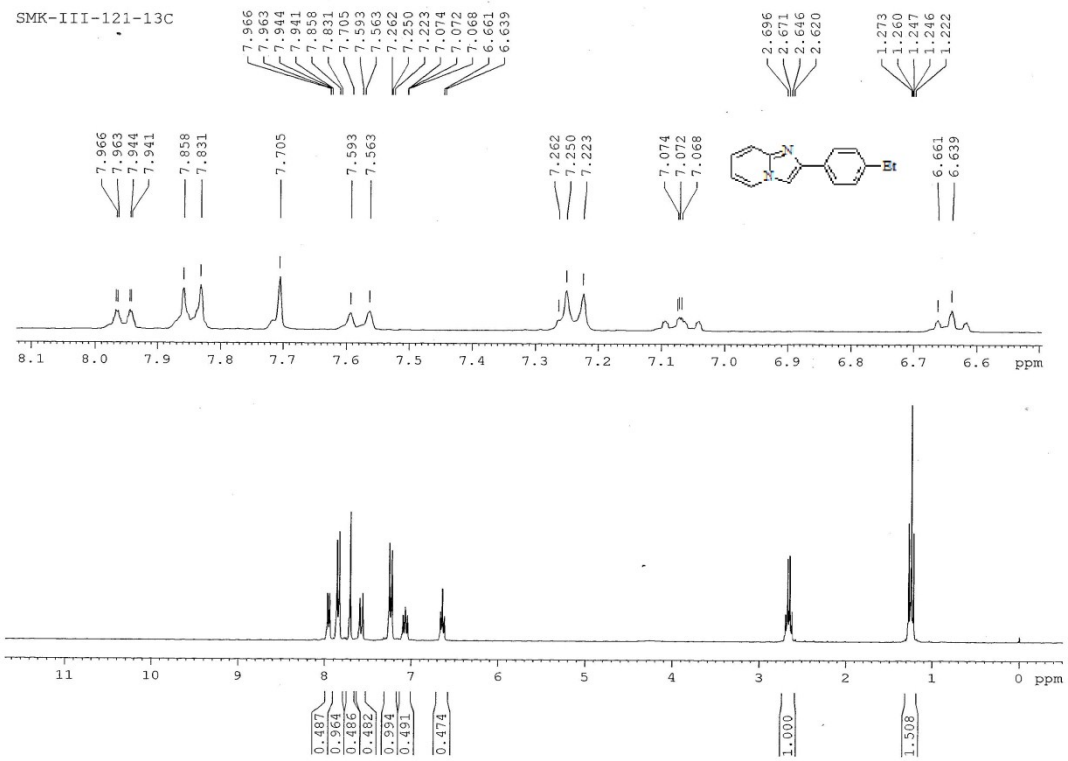
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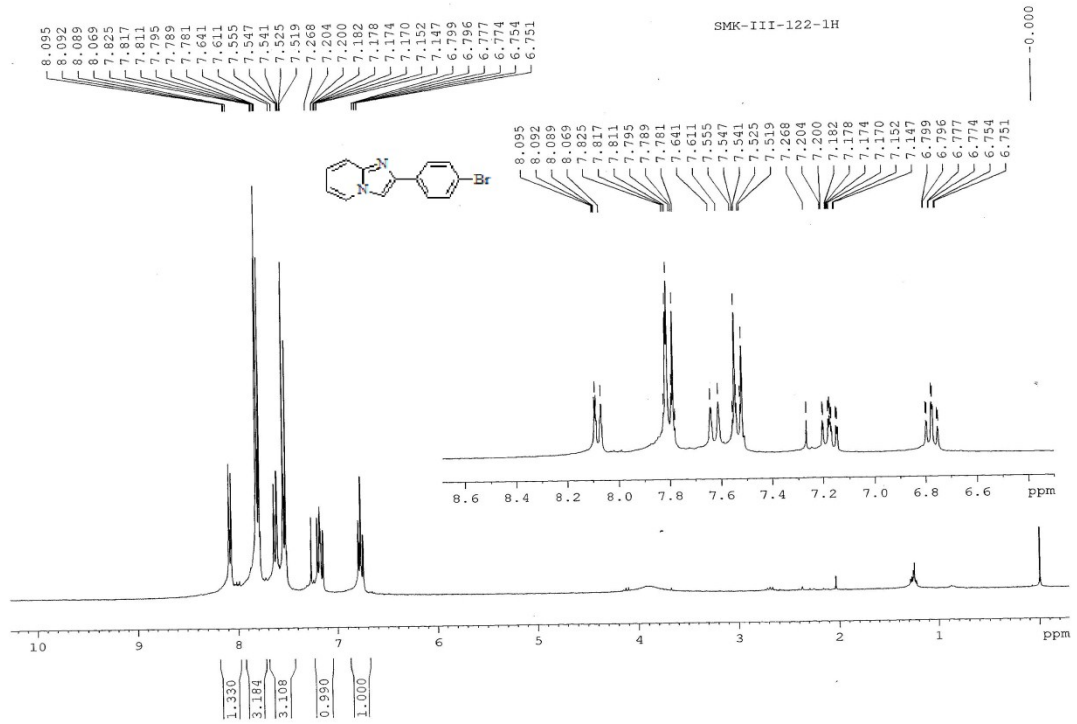
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7.817
7.811
7.795
7.789
7.781
7.641
7.611
7.555
7.547
7.541
7.525
7.519
7.268
7.204
7.200
7.182
7.178
7.174
7.170
7.152
7.147
6.799
6.796
6.777
6.774
6.754
6.751
0.000
Br
1.330
3.184
3.108
0.990
1.000
ppm

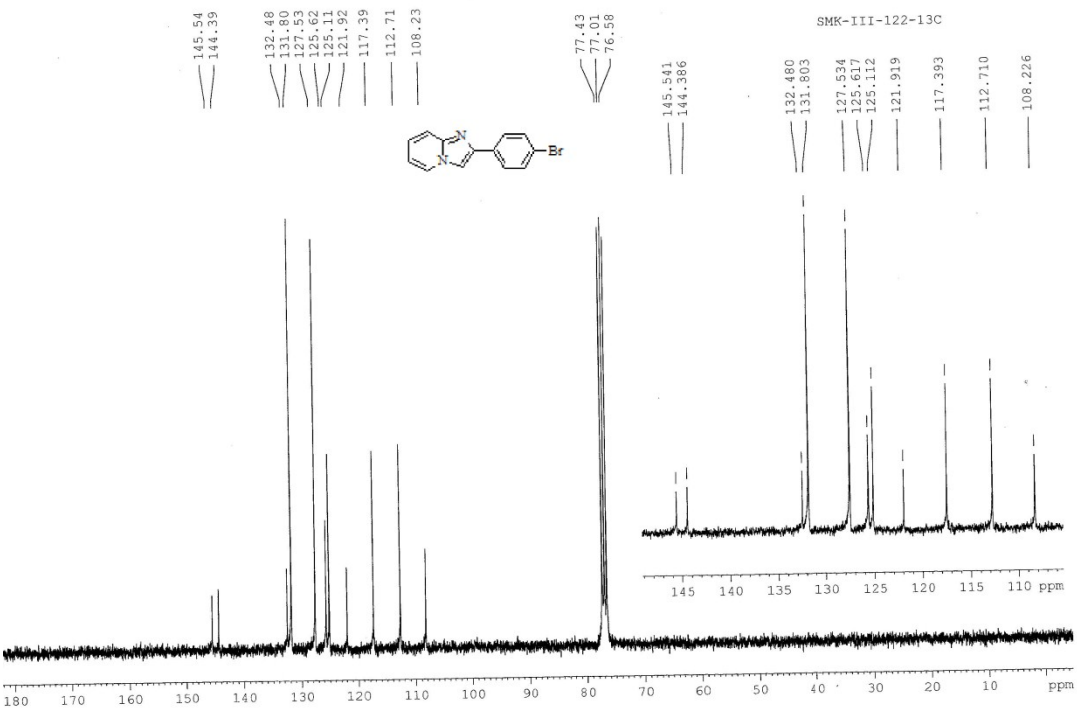
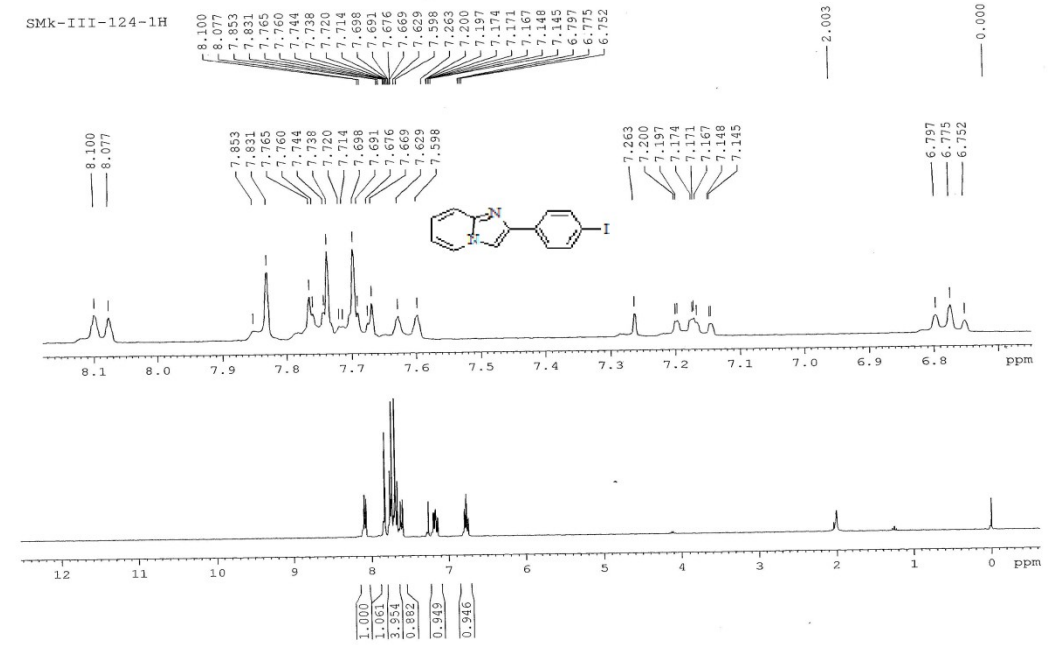

SMK-III-122-13C
145.54
144.39
132.48
131.80
127.53
125.62
125.11
121.92
117.39
112.71
108.23
77.43
77.01
76.58
145.541
144.386
132.480
131.803
127.534
125.617
125.112
121.919
117.393
112.710
108.226
Br
ppm

Table 2, 5d:


SMk-III-124-1H
8.100
8.077
7.853
7.831
7.765
7.760
7.744
7.738
7.720
7.714
7.698
7.691
7.676
7.669
7.629
7.598
7.263
7.200
7.197
7.174
7.171
7.167
7.148
7.145
6.797
6.775
6.752
2.003
0.000
1.000
1.061
3.854
0.882
0.949
0.946
ppm

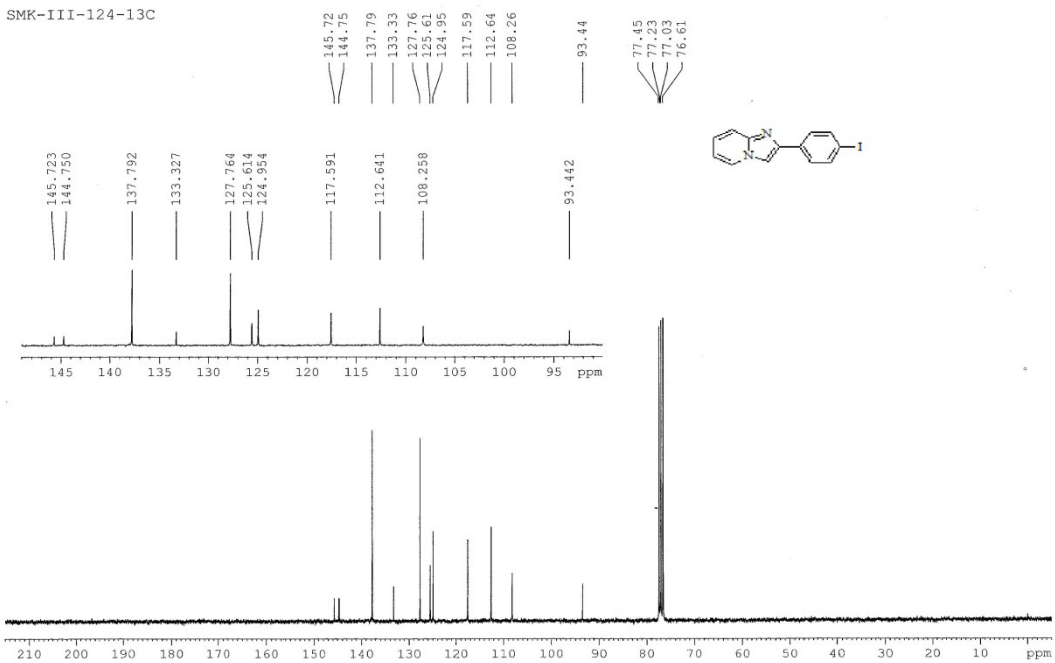
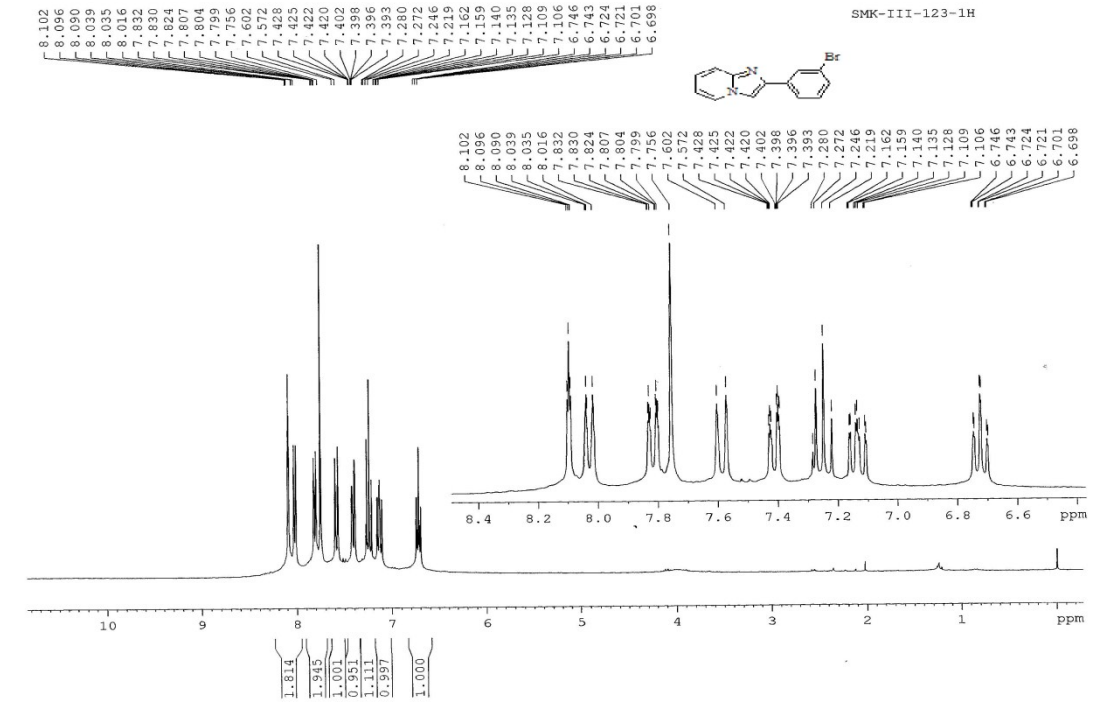

SMK-III-124-13C
145.72
144.75
137.79
133.33
127.76
125.61
124.95
117.59
112.64
108.26
93.44
77.45
77.23
77.03
76.61
145.723
144.750
137.792
133.327
127.764
125.614
124.954
117.591
112.641
108.258
93.442
ppm

Table 2, 5e:


SMK-III-123-1H
8.102
8.096
8.090
8.039
8.035
8.016
7.832
7.830
7.824
7.807
7.804
7.799
7.756
7.602
7.572
7.428
7.425
7.422
7.420
7.402
7.398
7.396
7.393
7.280
7.272
7.246
7.219
7.162
7.159
7.140
7.135
7.128
7.109
7.106
6.746
6.743
6.724
6.721
6.701
6.698
Br
N
N
1.814
1.945
1.001
0.951
1.111
0.997
1.000
ppm

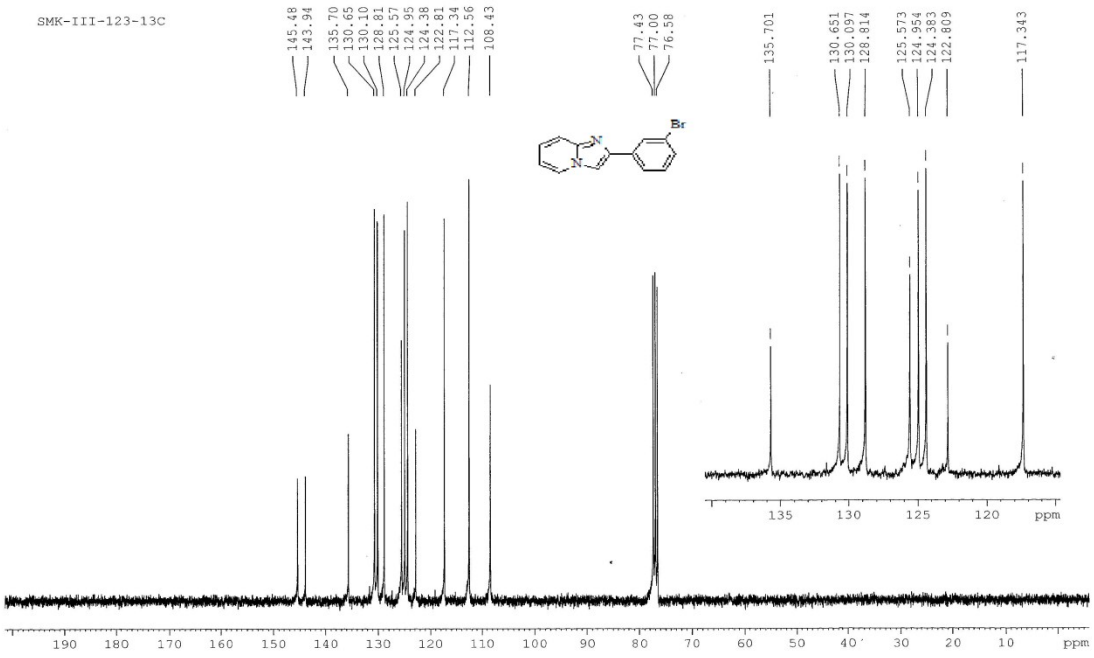
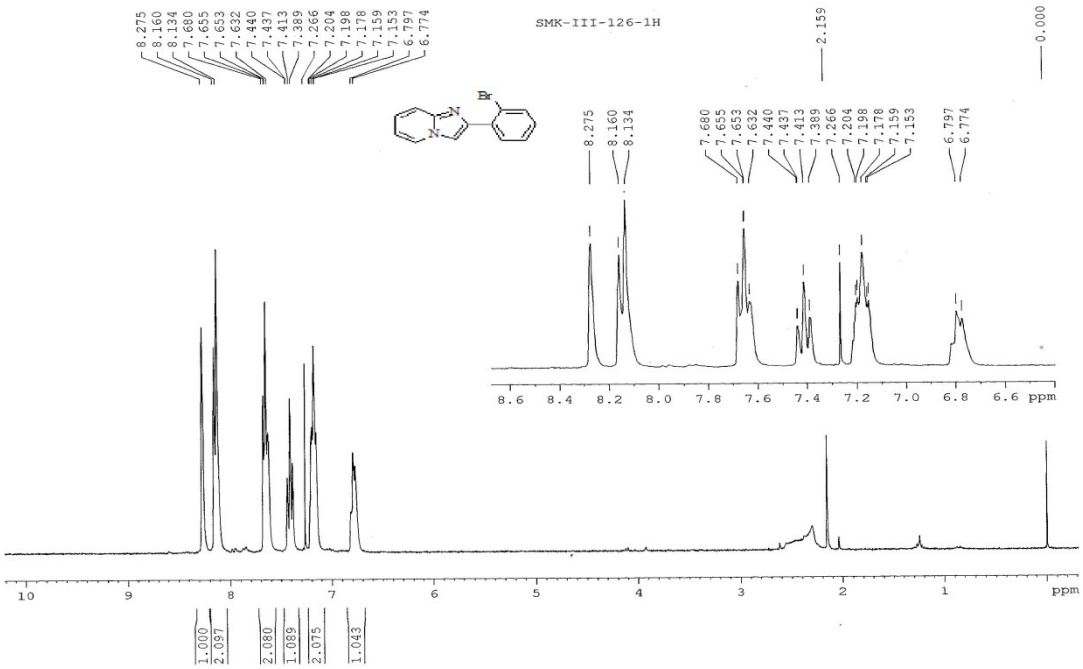

SMK-III-123-13C
145.48
143.94
135.70
130.65
130.10
128.81
125.57
124.95
124.38
122.81
117.34
112.56
108.43
77.43
77.00
76.58
135.701
130.651
130.097
128.814
125.573
124.954
124.383
122.809
117.343
Br
N
N
ppm

Table 2, 5f:


SMK-III-126-1H
8.275
8.160
8.134
7.680
7.655
7.653
7.632
7.440
7.437
7.413
7.389
7.266
7.204
7.198
7.178
7.159
7.153
6.797
6.774
2.159
0.000
Br
1.000
2.097
2.080
1.089
2.075
1.043
ppm

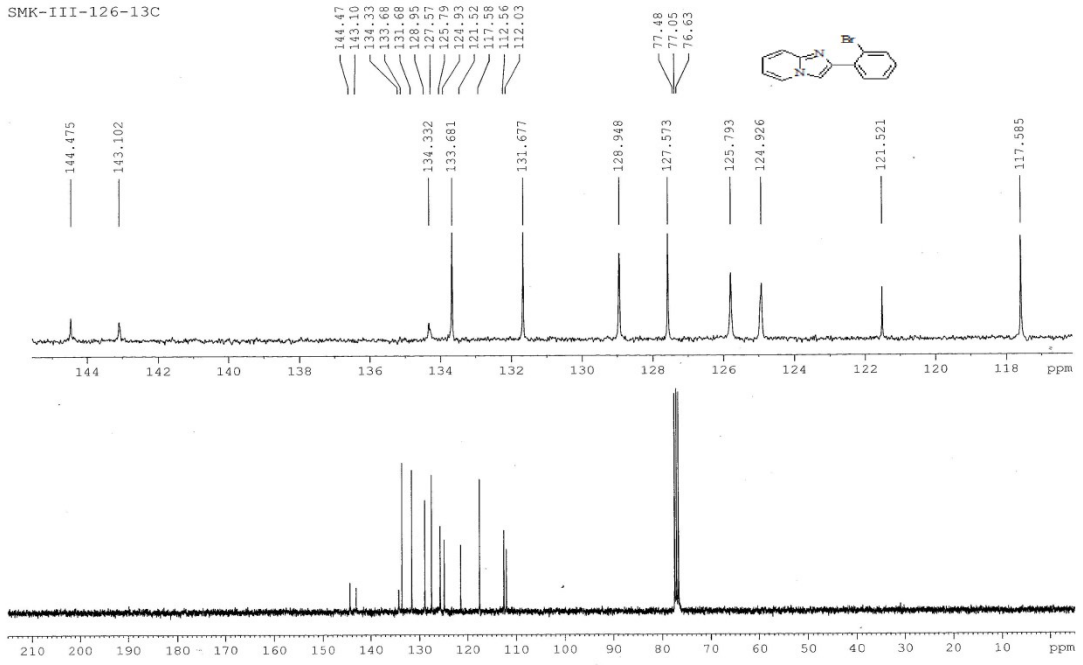
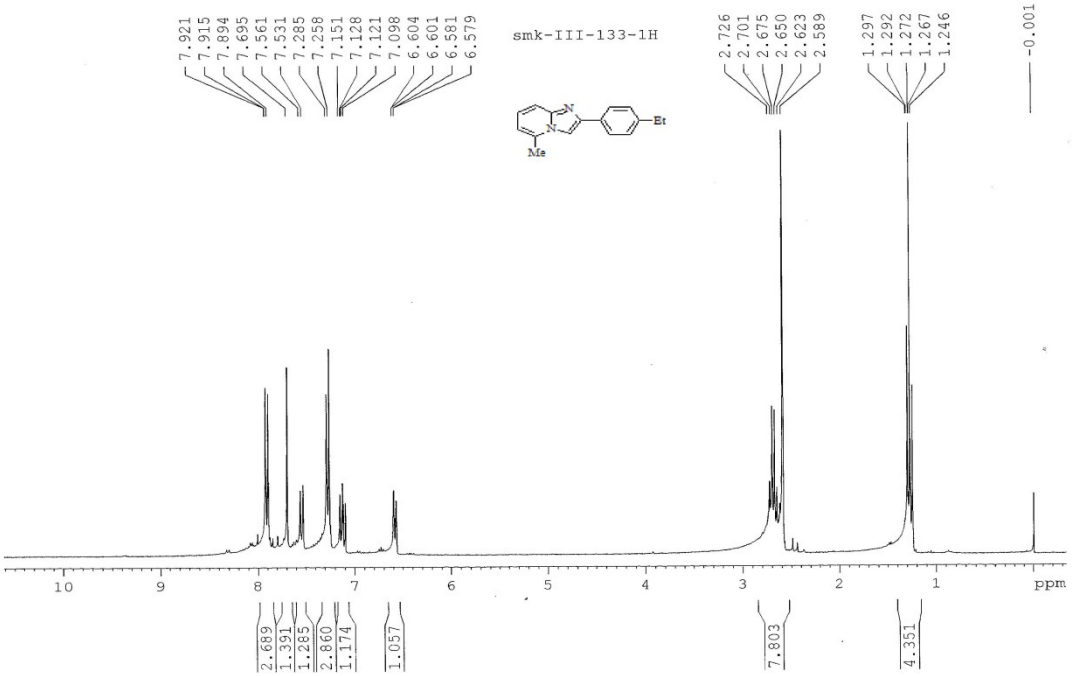

SMK-III-126-13C
144.475
143.102
134.332
133.681
131.677
128.948
127.573
125.793
124.926
121.521
117.585
77.48
77.05
76.63
Br
ppm

Table 2, 5g:


smk-III-133-1H
7.921
7.915
7.894
7.695
7.561
7.531
7.285
7.258
7.151
7.128
7.121
7.098
6.604
6.601
6.581
6.579
2.726
2.701
2.675
2.650
2.623
2.589
1.297
1.292
1.272
1.267
1.246
-0.001
Et
Me
ppm
2.689
1.391
1.285
2.860
1.174
1.057
7.803
4.351

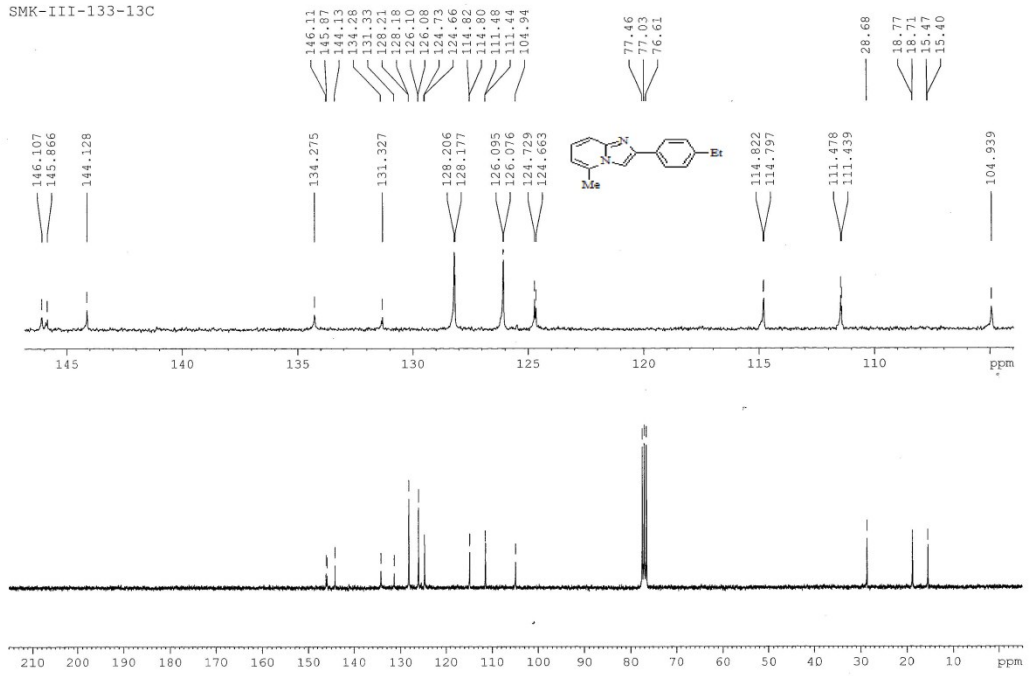
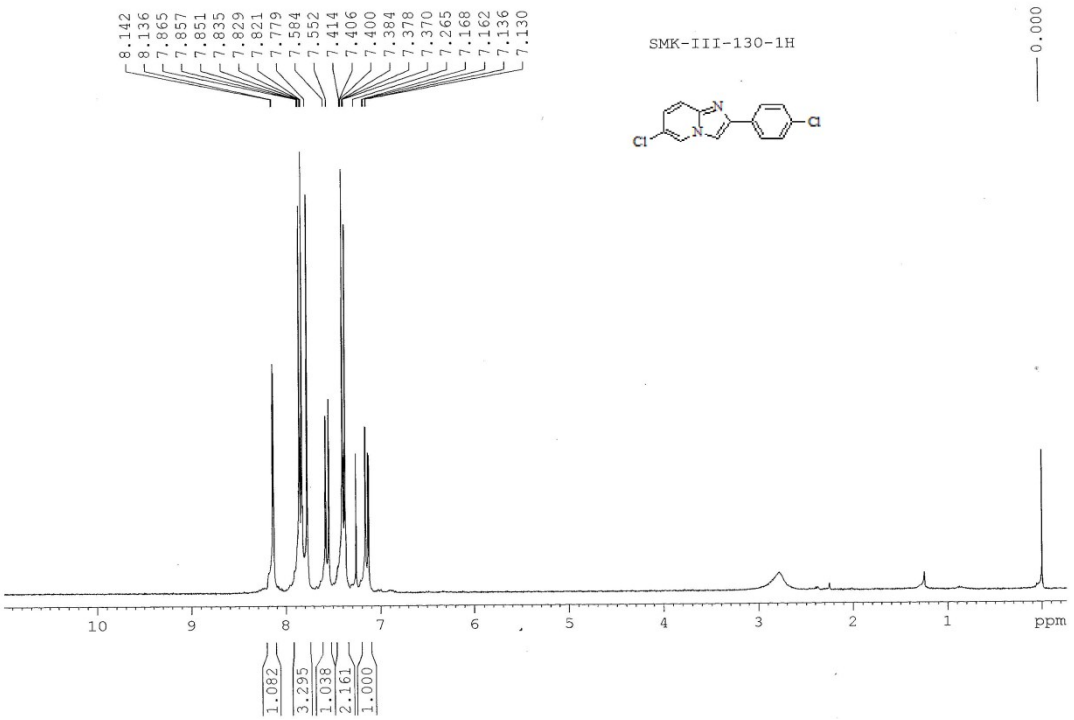

SMK-III-133-13C
146.11
145.87
144.13
134.28
131.33
128.21
128.18
126.10
126.08
124.73
124.66
114.82
114.80
111.48
111.44
104.94
77.46
77.03
76.61
28.68
18.77
18.71
15.47
15.40
146.107
145.866
144.128
134.275
131.327
128.206
128.177
126.095
126.076
124.729
124.663
114.822
114.797
111.478
111.439
104.939
Et
Me
ppm

Table 2, 5h:


8.142
8.136
7.865
7.857
7.851
7.835
7.829
7.821
7.779
7.584
7.552
7.414
7.406
7.400
7.384
7.378
7.370
7.265
7.168
7.162
7.136
7.130
SMK-III-130-1H
0.000
10
9
8
7
6
5
4
3
2
1
ppm
1.082
3.295
1.038
2.161
1.000

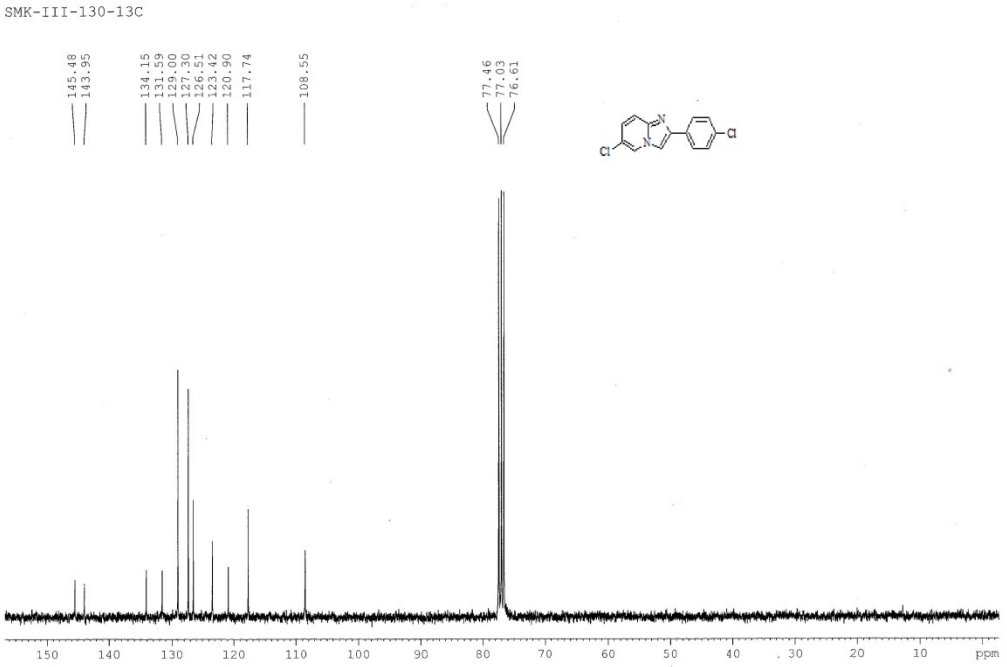
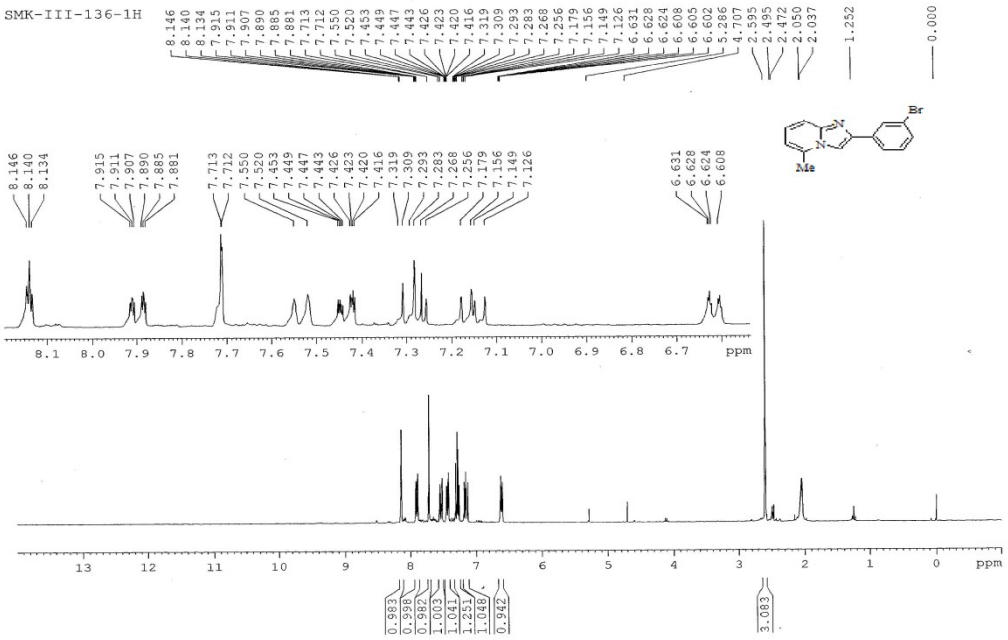

SMK-III-130-13C
145.48
143.95
134.15
131.59
129.00
127.30
126.51
123.42
120.90
117.74
108.55
77.46
77.03
76.61
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

Table 2, 5i:


SMK-III-136-1H
8.146
8.140
8.134
7.915
7.911
7.907
7.890
7.885
7.881
7.713
7.712
7.550
7.520
7.453
7.449
7.447
7.443
7.426
7.423
7.420
7.416
7.319
7.309
7.293
7.283
7.268
7.256
7.179
7.156
7.149
7.126
6.631
6.628
6.624
6.608
6.605
6.602
5.286
4.707
2.595
2.495
2.472
2.050
2.037
1.252
0.000
Br
N
N
Me
ppm
0.983
0.998
0.982
1.003
1.041
1.251
1.048
0.942
3.083

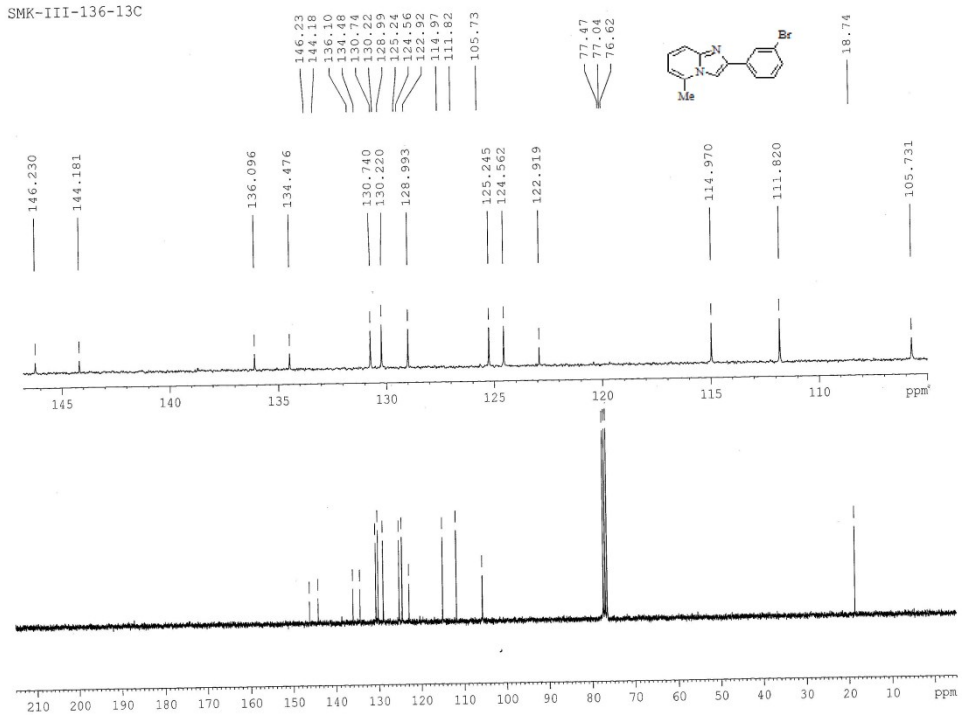
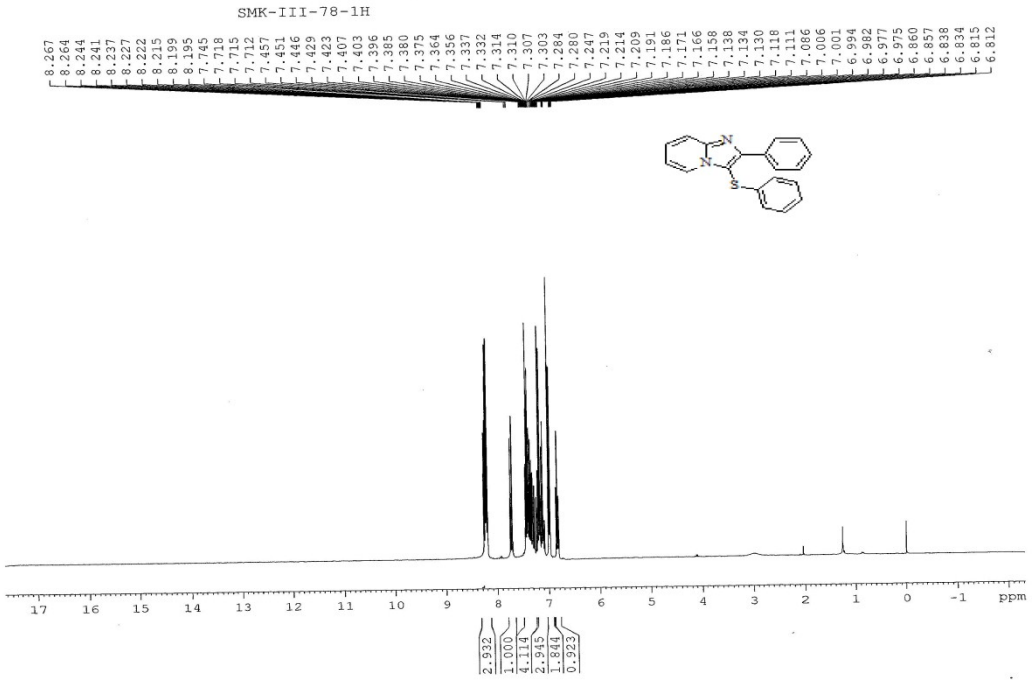

SMK-III-136-13C
146.23
144.18
136.10
134.48
130.74
130.22
128.99
125.24
124.56
122.92
114.97
111.82
105.73
77.47
77.04
76.62
18.74
Br
N
N
Me
146.230
144.181
136.096
134.476
130.740
130.220
128.993
125.245
124.562
122.919
114.970
111.820
105.731
ppm

Table 3, 8a:


SMK-III-78-1H
8.267
8.264
8.244
8.241
8.237
8.227
8.222
8.215
8.199
8.195
7.745
7.718
7.715
7.712
7.457
7.451
7.446
7.429
7.423
7.407
7.403
7.396
7.385
7.380
7.375
7.364
7.356
7.337
7.332
7.314
7.310
7.307
7.303
7.284
7.280
7.247
7.219
7.214
7.209
7.191
7.186
7.171
7.166
7.158
7.138
7.134
7.130
7.118
7.111
7.086
7.006
7.001
6.994
6.982
6.977
6.975
6.860
6.857
6.838
6.834
6.815
6.812
ppm
2.932
1.000
4.114
2.945
1.844
0.923

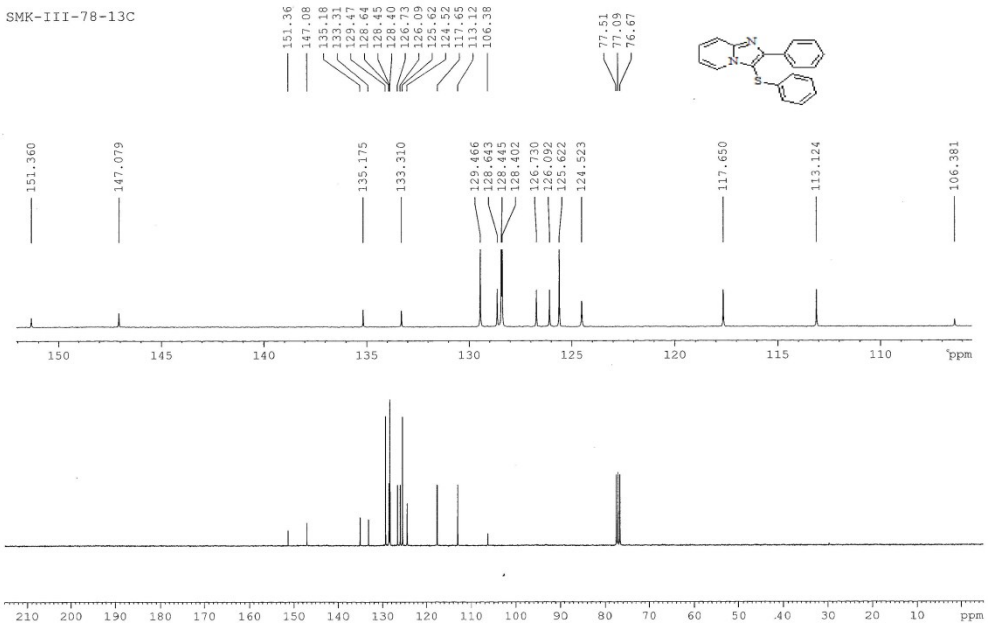
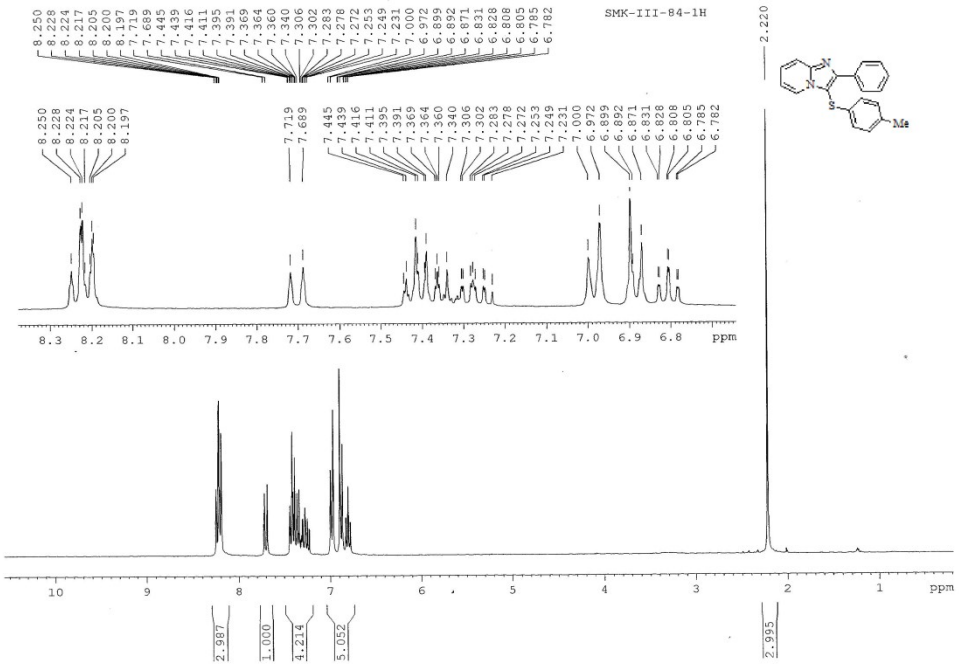

SMK-III-78-13C
151.36
147.08
135.18
133.31
129.47
128.64
128.45
128.40
126.73
126.09
125.62
124.52
117.65
113.12
106.38
77.51
77.09
76.67
151.360
147.079
135.175
133.310
129.466
128.643
128.445
128.402
126.730
126.092
125.622
124.523
117.650
113.124
106.381
ppm

Table 3, 8b:


SMK-III-84-1H
8.250
8.228
8.224
8.217
8.205
8.200
8.197
7.719
7.689
7.445
7.439
7.416
7.411
7.395
7.391
7.369
7.364
7.360
7.340
7.306
7.302
7.283
7.278
7.272
7.253
7.249
7.231
7.000
6.972
6.892
6.871
6.831
6.828
6.808
6.805
6.785
6.782
2.220
Me
ppm
2.987
1.000
4.214
5.052
2.995

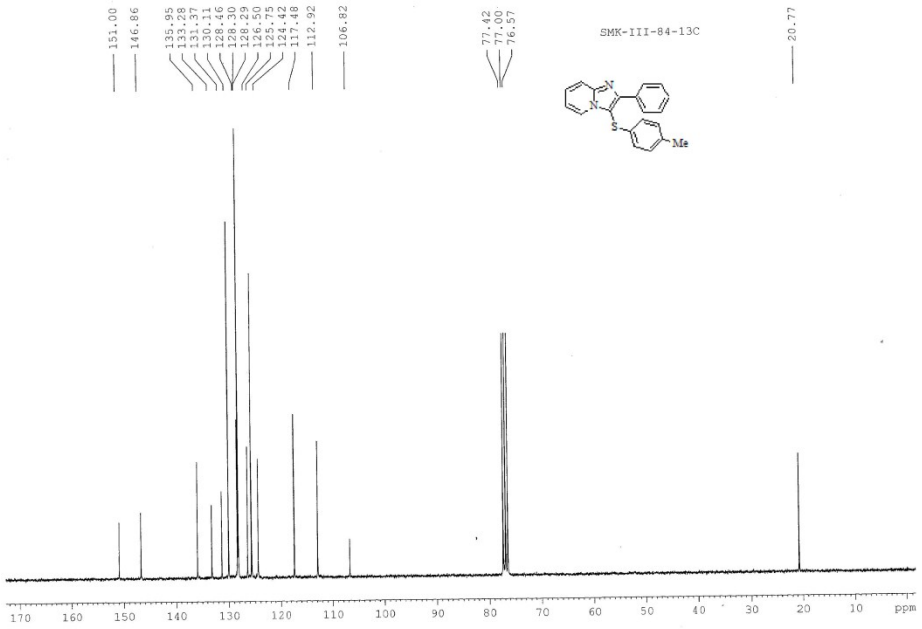
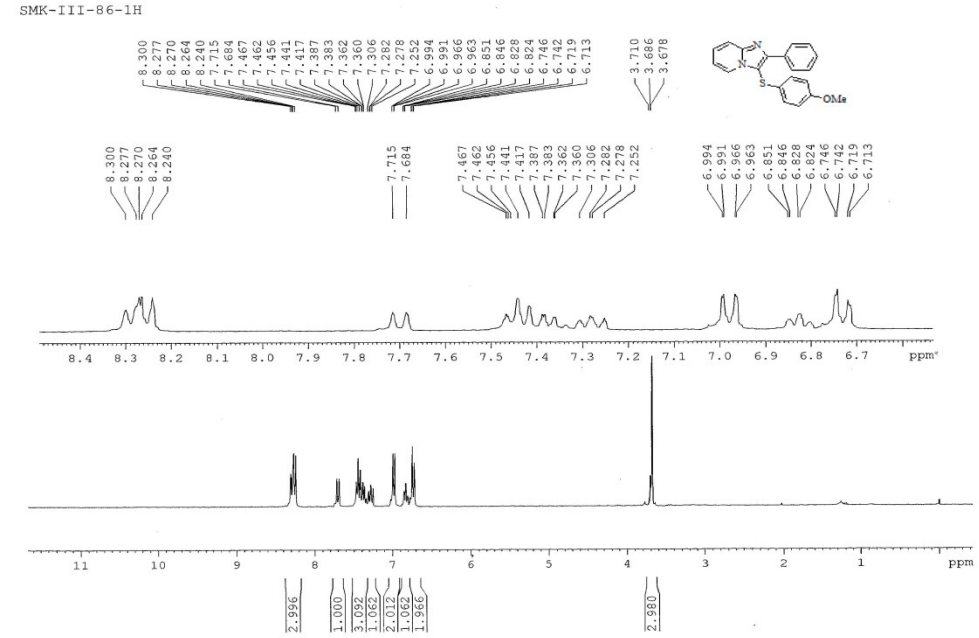

SMK-III-84-13C
151.00
146.86
135.95
133.28
131.37
130.11
128.46
128.30
128.29
126.50
125.75
124.42
117.48
112.92
106.82
77.42
77.00
76.57
20.77
Me
ppm

Table 3, 8c:


SMK-III-86-1H
8.300
8.277
8.270
8.264
8.240
7.715
7.684
7.467
7.462
7.456
7.441
7.417
7.387
7.383
7.362
7.360
7.306
7.282
7.278
7.252
6.994
6.991
6.966
6.963
6.851
6.846
6.828
6.824
6.746
6.742
6.719
6.713
3.710
3.686
3.678
OMe
ppm
2.996
1.000
3.092
1.062
2.012
1.062
1.966
2.980

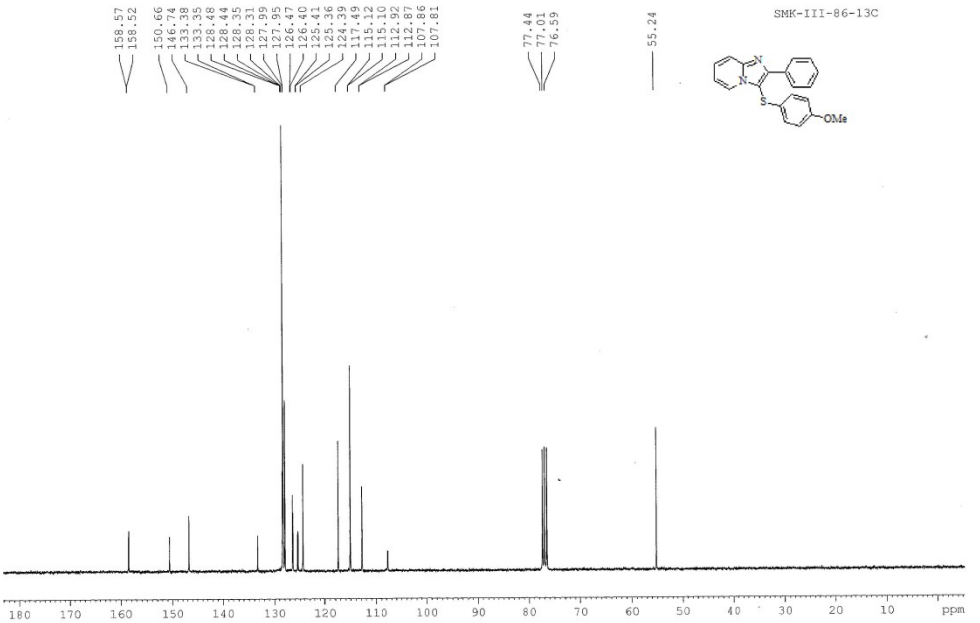
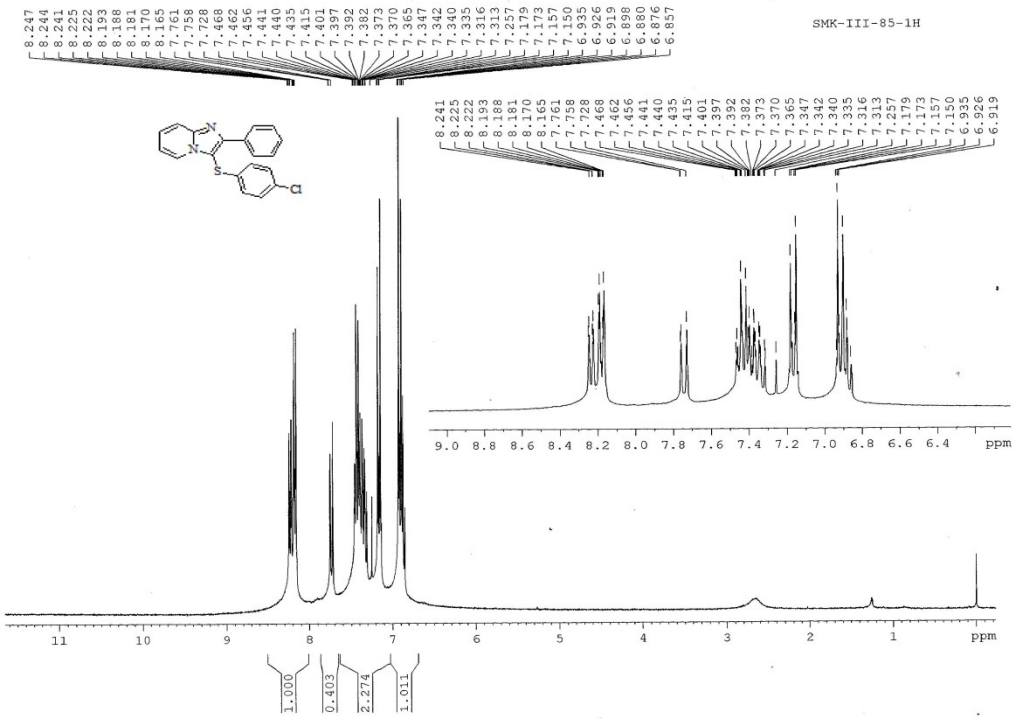

SMK-III-86-13C
158.57
158.52
150.66
146.74
133.38
133.35
128.48
128.44
128.35
128.31
127.99
127.95
126.47
126.40
125.41
125.36
121.49
117.49
115.12
115.10
112.92
112.87
107.86
107.81
77.44
77.01
76.59
55.24
OMe
ppm

Table 3, 8d:


SMK-III-85-1H
8.247
8.244
8.241
8.225
8.222
8.193
8.188
8.181
8.170
8.165
7.761
7.758
7.728
7.468
7.462
7.456
7.441
7.440
7.435
7.415
7.401
7.397
7.392
7.382
7.373
7.370
7.365
7.347
7.342
7.340
7.335
7.316
7.313
7.257
7.179
7.173
7.157
7.150
6.935
6.926
6.919
6.898
6.880
6.876
6.857
Cl
9.0 8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 ppm
11 10 9 8 7 6 5 4 3 2 1 ppm
1.000
0.403
2.274
1.011

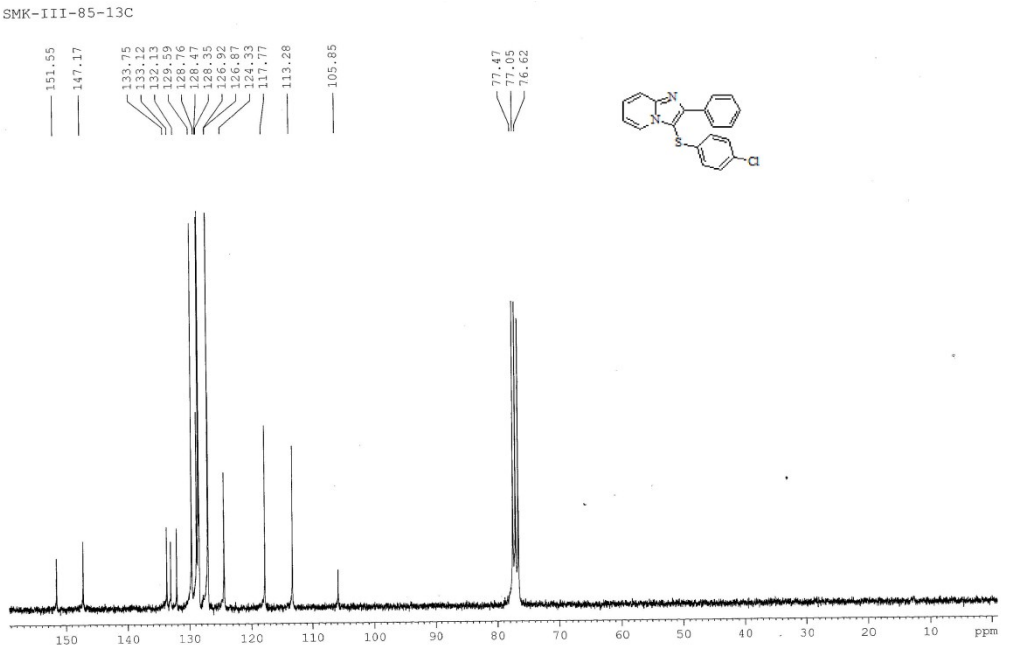
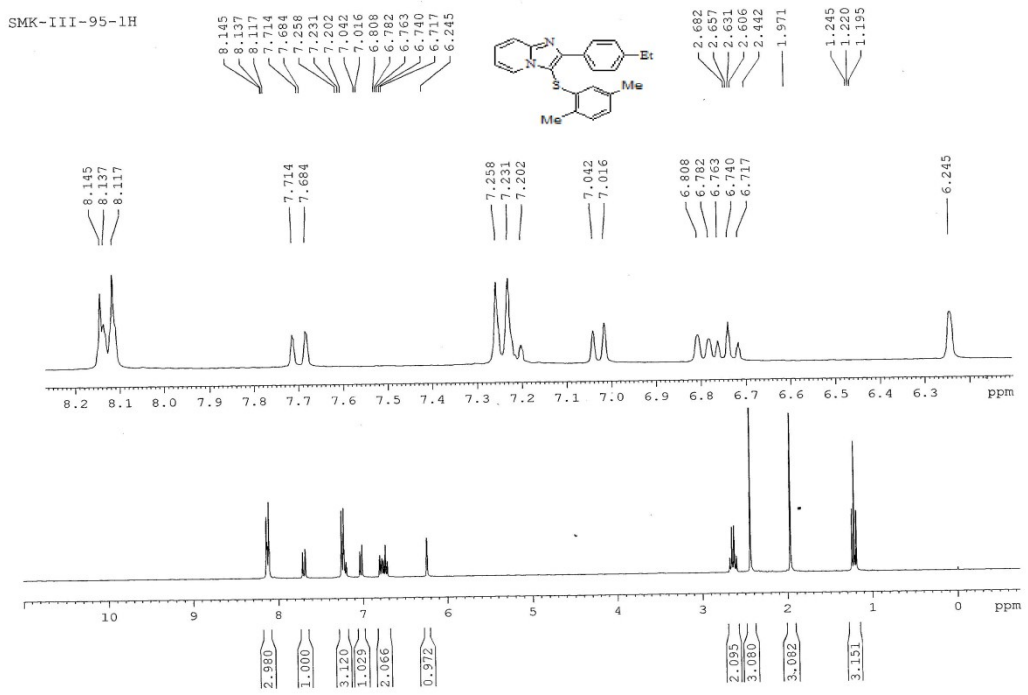

SMK-III-85-13C
151.55
147.17
133.75
133.12
132.13
129.59
128.76
128.47
128.35
126.92
126.87
124.33
117.77
113.28
105.85
77.47
77.05
76.62
Cl
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Table 3, 8e:


SMK-III-95-1H
8.145
8.137
8.117
7.714
7.684
7.258
7.231
7.202
7.042
7.016
6.808
6.782
6.763
6.740
6.717
6.245
2.682
2.657
2.631
2.606
2.442
1.971
1.245
1.220
1.195
Et
Me
Me
ppm
2.980
1.000
3.120
1.029
2.066
0.972
2.095
3.080
3.082
3.151

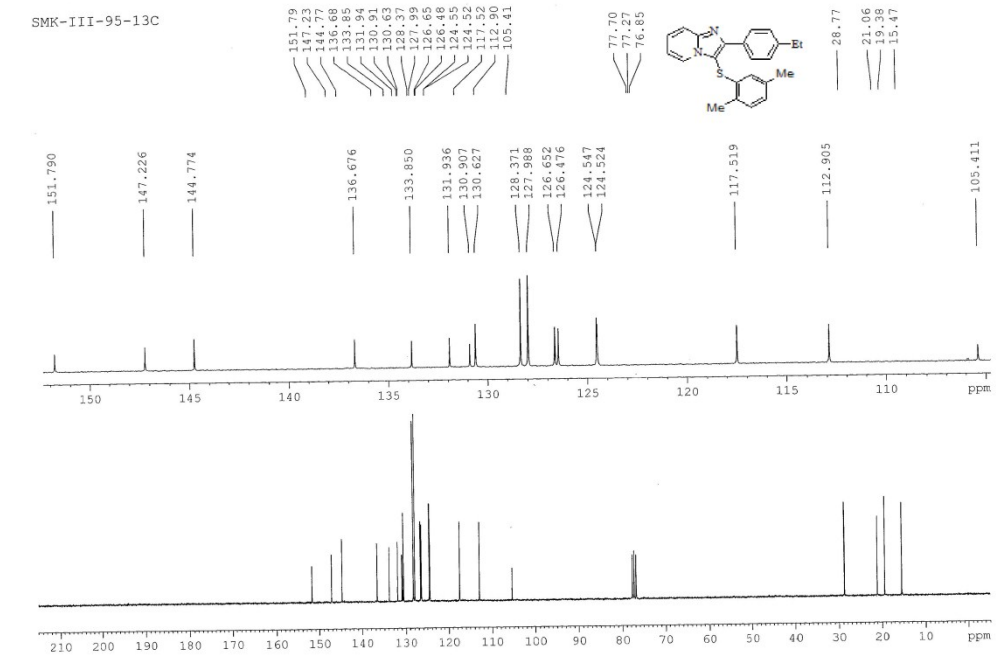
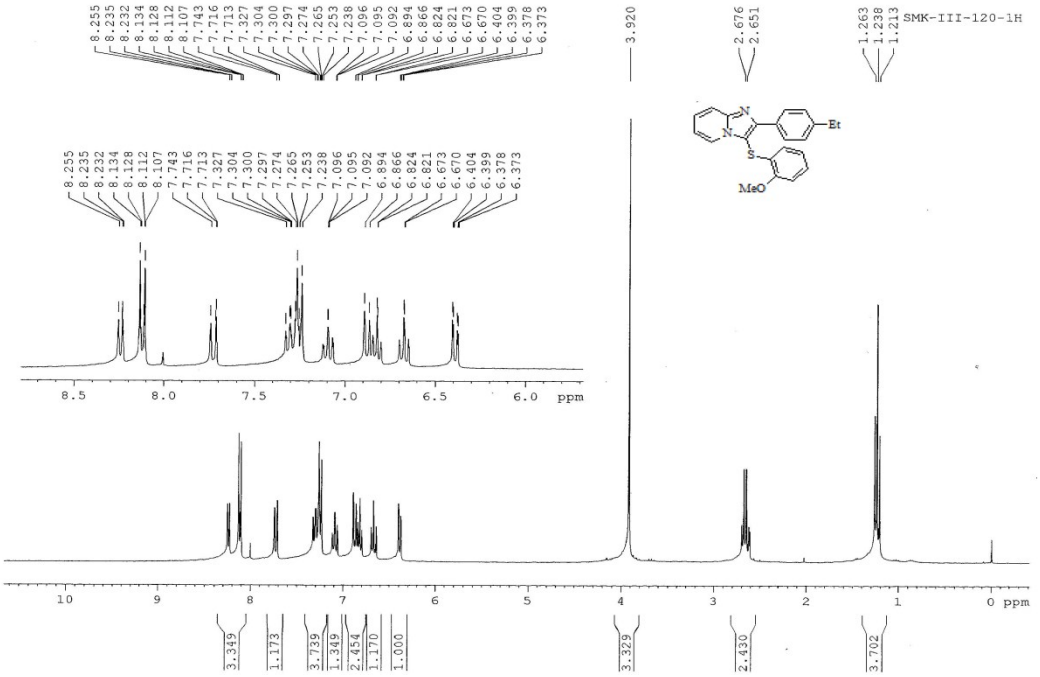

SMK-III-95-13C
151.79
147.23
144.77
136.68
133.85
131.94
130.91
130.63
128.37
127.98
126.48
124.55
124.52
117.52
112.90
105.41
77.70
77.27
76.85
28.77
21.06
19.38
15.47
Et
Me
Me
ppm

Table 3, 8f:


SMK-III-120-1H
8.255
8.235
8.232
8.134
8.128
8.112
8.107
7.743
7.716
7.713
7.327
7.304
7.300
7.297
7.274
7.265
7.253
7.238
7.096
7.095
7.092
6.894
6.866
6.824
6.821
6.673
6.670
6.404
6.399
6.378
6.373
3.920
2.676
2.651
1.263
1.238
1.213
Et
MeO
8.5
8.0
7.5
7.0
6.5
6.0 ppm
10
9
8
7
6
5
4
3
2
1
0 ppm
3.349
1.173
3.739
1.349
2.454
1.170
1.000
3.329
2.430
3.702

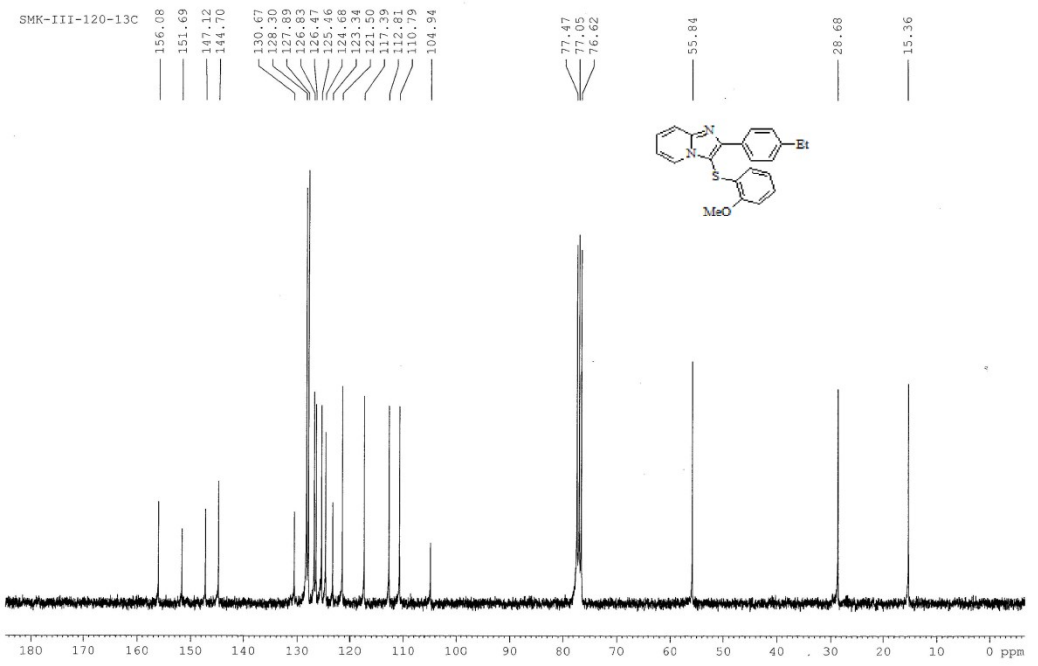
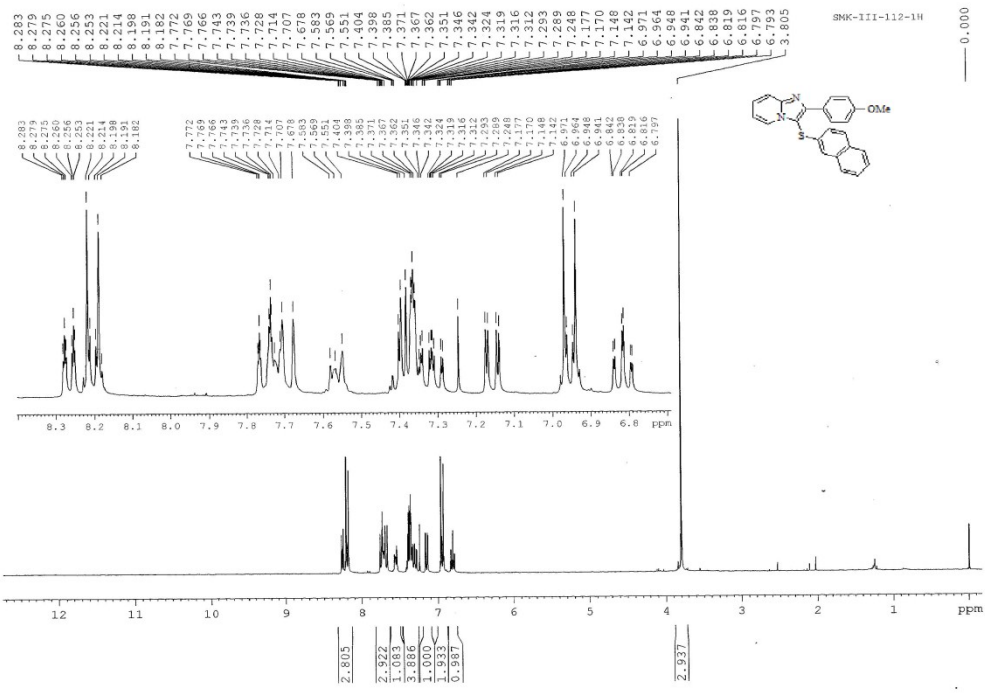

SMK-III-120-13C
156.08
151.69
147.12
144.70
130.67
128.30
127.89
126.83
126.47
125.46
124.68
123.34
121.50
117.39
112.81
110.79
104.94
77.47
77.05
76.62
55.84
28.68
15.36
Et
MeO
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

Table 3, 8g:


SMK-III-112-1H
OMe
ppm
2.805
2.922
1.083
3.886
1.000
1.933
0.987
2.937

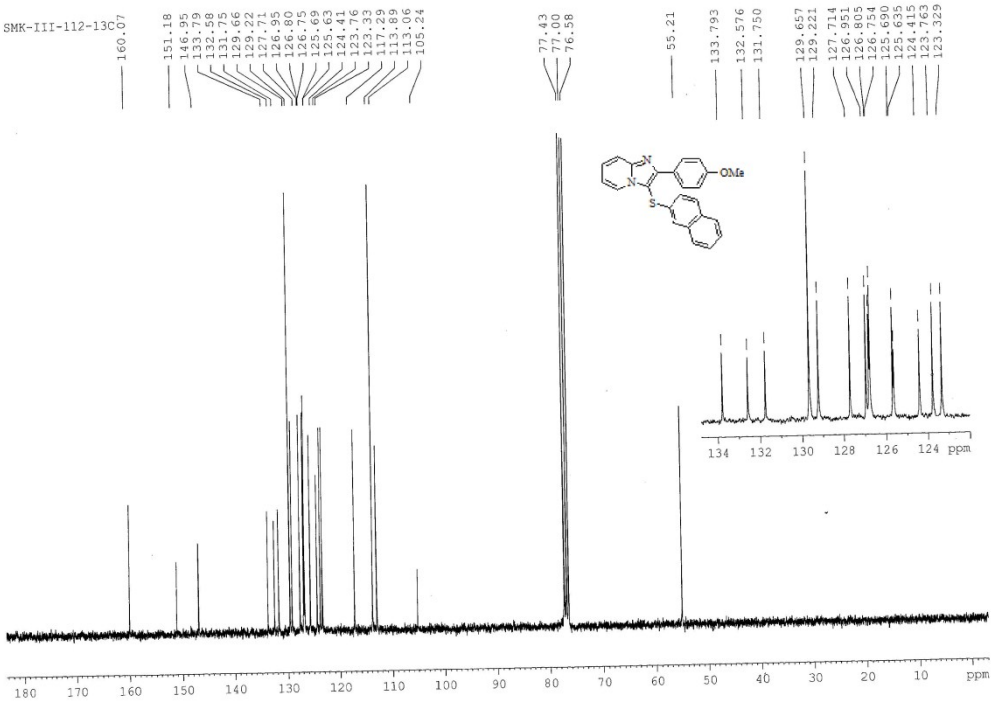
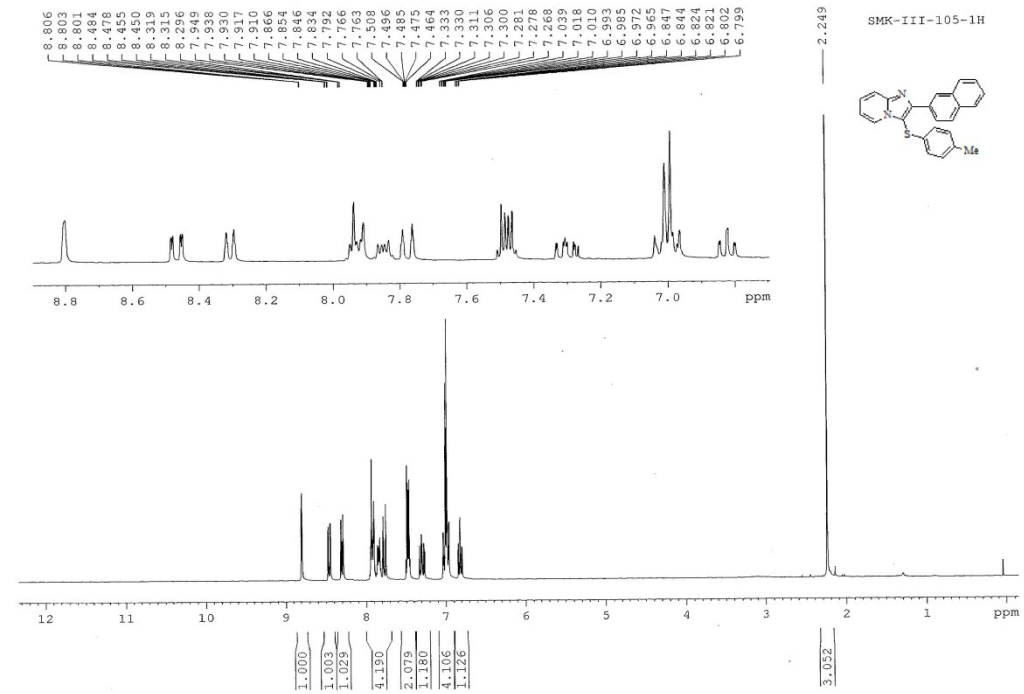

SMK-III-112-13C
OMe
ppm

Table 3, 8h:


SMK-III-105-1H
8.806
8.803
8.801
8.484
8.478
8.455
8.450
8.319
8.315
8.296
7.949
7.938
7.930
7.917
7.910
7.866
7.854
7.846
7.834
7.792
7.766
7.763
7.508
7.496
7.485
7.475
7.464
7.333
7.330
7.311
7.306
7.300
7.281
7.278
7.268
7.039
7.018
7.010
6.993
6.985
6.972
6.965
6.847
6.844
6.824
6.821
6.802
6.799
2.249
Me
ppm
1.000
1.003
1.029
4.190
2.079
1.180
4.106
1.126
3.052

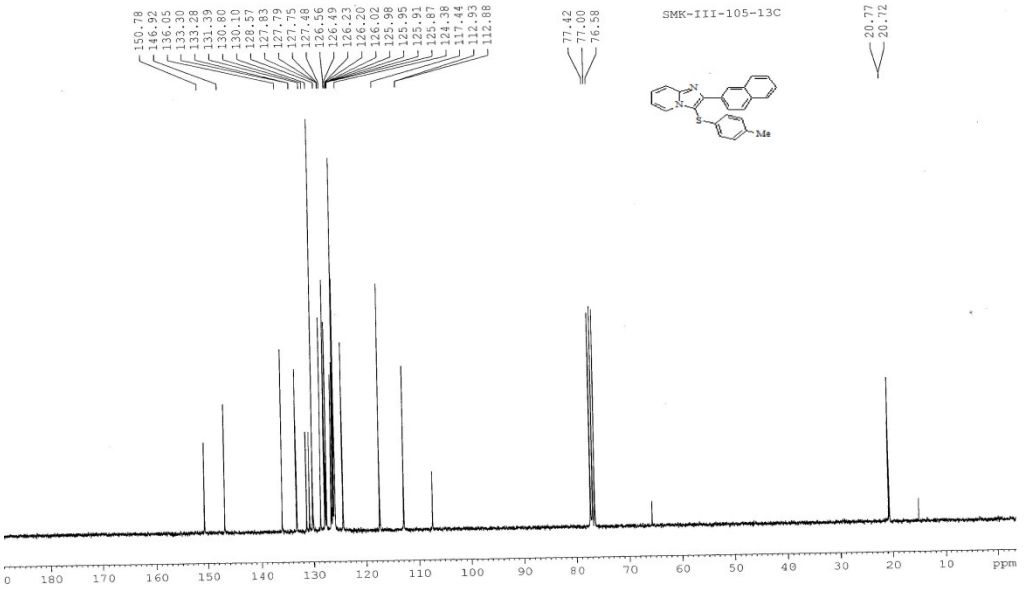
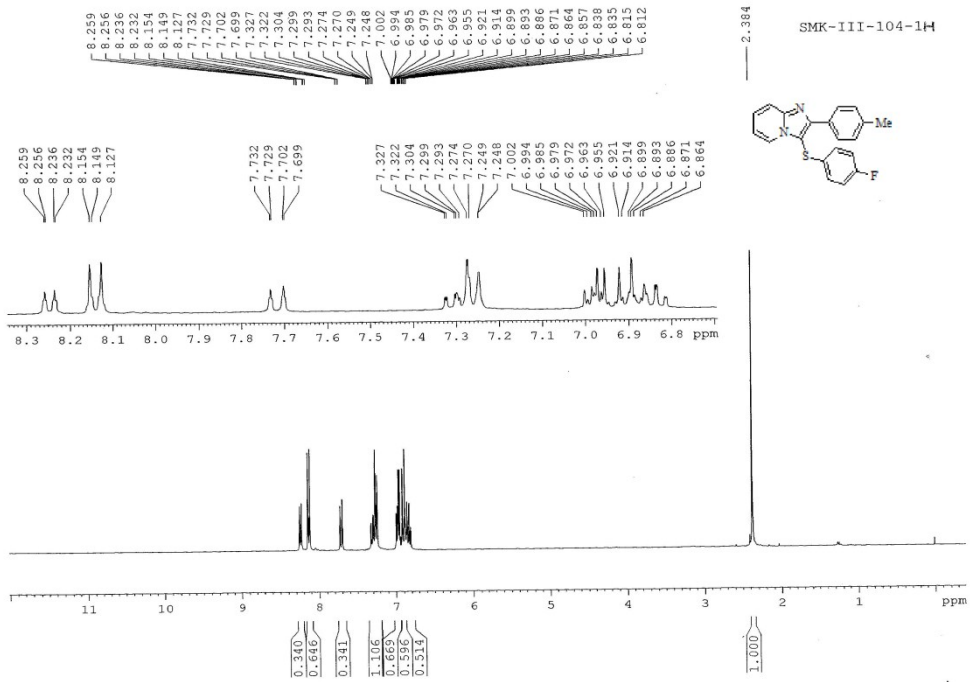

SMK-III-105-13C
150.78
146.92
136.05
133.30
133.28
131.39
130.80
130.10
128.57
127.83
127.79
127.75
127.48
126.56
126.49
126.23
126.20
126.02
125.98
125.95
125.91
125.87
124.38
117.44
112.93
112.88
77.42
77.00
76.58
20.77
20.72
Me
ppm

Table 3, 8i:


SMK-III-104-1H
8.259
8.256
8.236
8.232
8.154
8.149
8.127
7.732
7.729
7.702
7.699
7.327
7.322
7.304
7.299
7.293
7.274
7.270
7.249
7.248
7.002
6.994
6.985
6.979
6.972
6.963
6.955
6.921
6.914
6.899
6.893
6.886
6.871
6.864
6.857
6.838
6.835
6.815
6.812
2.384
Me
F
ppm
0.340
0.646
0.341
1.106
0.669
0.596
0.514
1.000

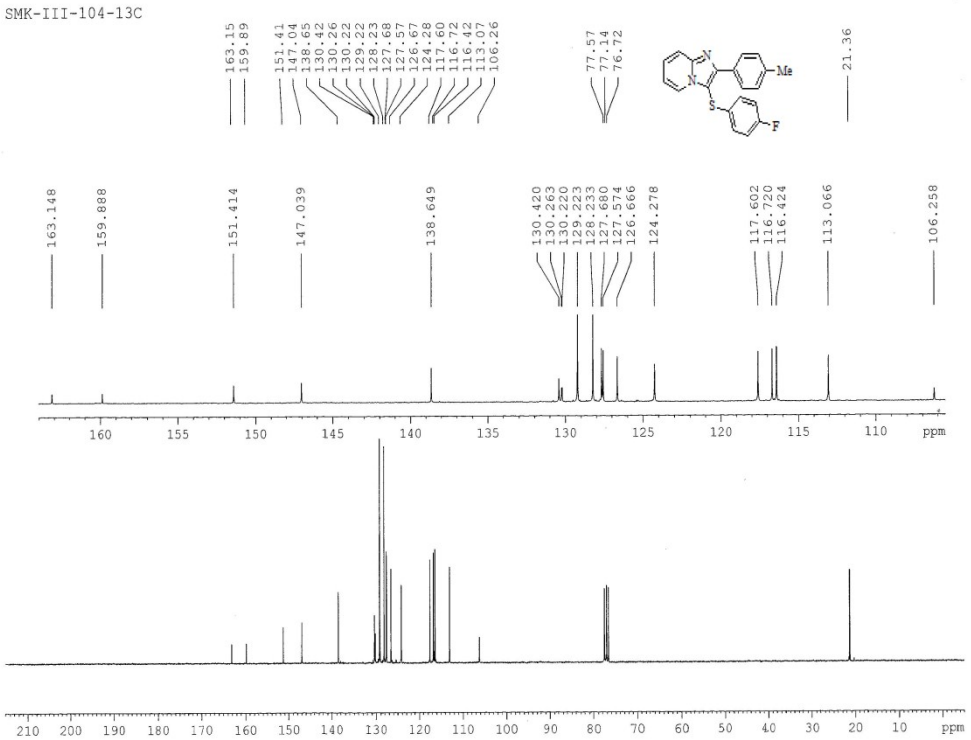
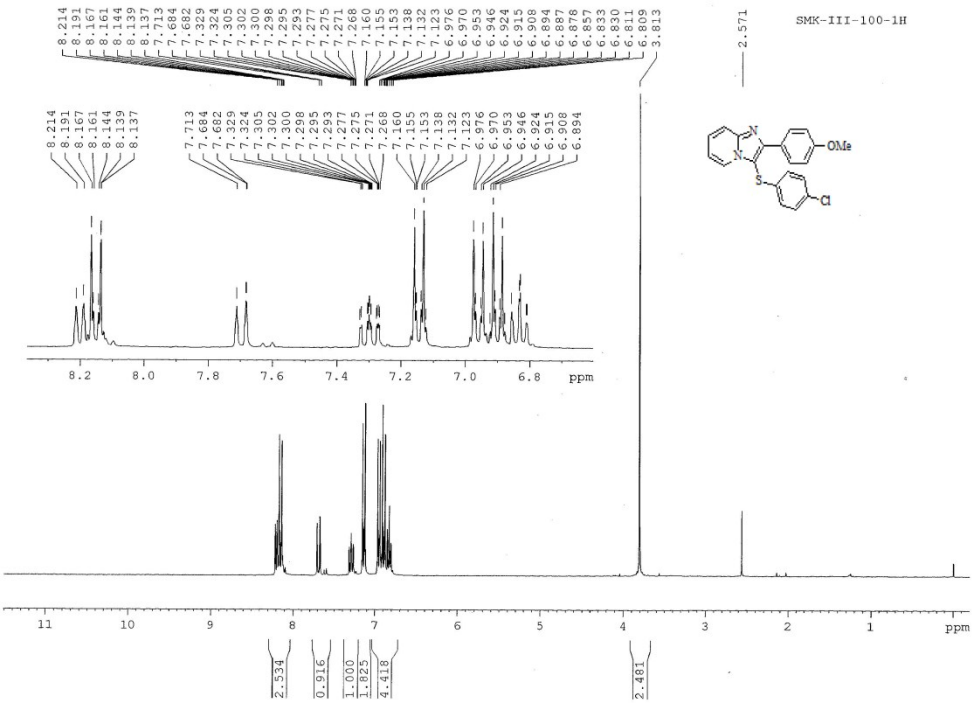

SMK-III-104-13C
163.15
159.89
151.41
147.04
138.65
130.42
130.26
130.22
129.22
128.23
127.68
127.57
126.67
124.28
117.60
116.42
113.07
106.26
77.57
77.14
76.72
21.36
Me
F
163.148
159.888
151.414
147.039
138.649
130.420
130.263
130.220
129.223
128.233
127.680
127.574
126.666
124.278
117.602
116.720
116.424
113.066
106.258
ppm

Table 3, 8j:


SMK-III-100-1H
8.214
8.191
8.167
8.161
8.144
8.139
8.137
7.713
7.684
7.682
7.329
7.324
7.305
7.302
7.300
7.298
7.295
7.293
7.277
7.275
7.271
7.268
7.160
7.155
7.153
7.138
7.132
7.123
6.976
6.970
6.953
6.946
6.924
6.915
6.908
6.894
6.887
6.878
6.857
6.833
6.830
6.811
6.809
3.813
2.571
OMe
Cl
S
N
ppm
2.534
0.916
1.000
1.825
4.418
2.481

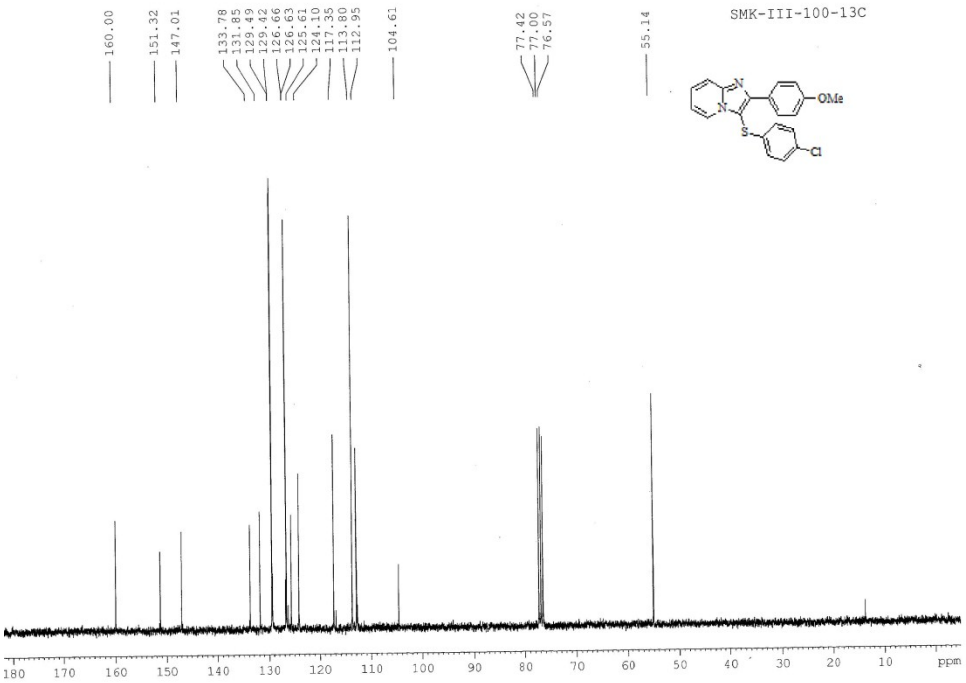
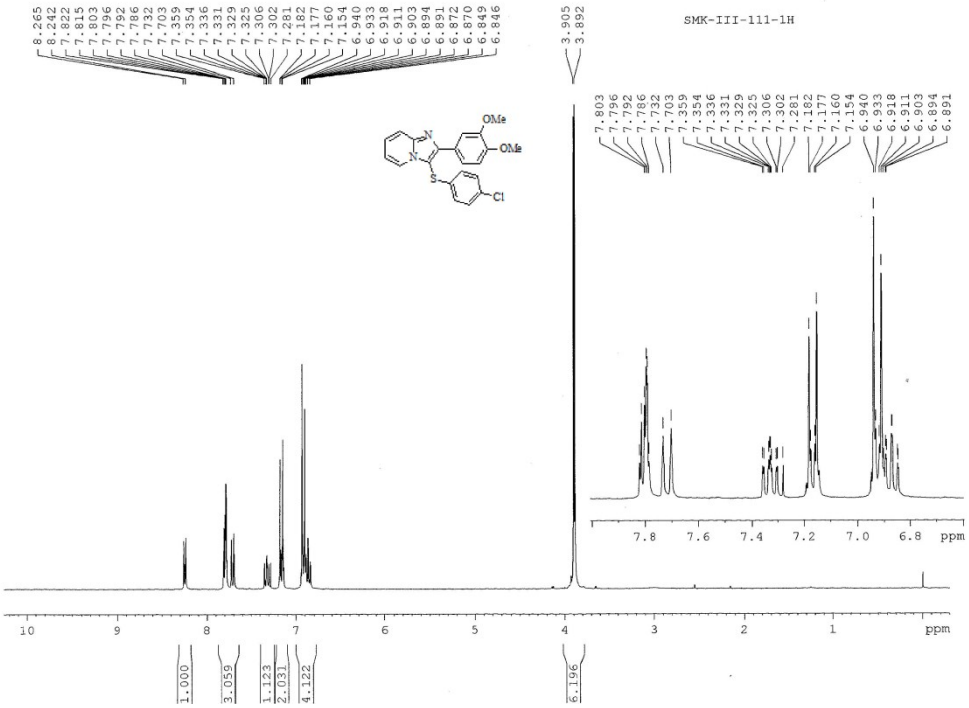

SMK-III-100-13C
160.00
151.32
147.01
133.78
131.85
129.49
129.42
126.66
126.63
125.61
124.10
117.35
113.80
112.95
104.61
77.42
77.00
76.57
55.14
OMe
Cl
S
N
ppm

Table 3, 8k:


SMK-III-111-1H
8.265
8.242
7.822
7.815
7.803
7.796
7.792
7.786
7.732
7.703
7.359
7.354
7.336
7.331
7.329
7.325
7.306
7.302
7.281
7.182
7.177
7.160
7.154
6.940
6.933
6.918
6.911
6.903
6.894
6.891
6.872
6.870
6.849
6.846
3.905
3.892
OMe
OMe
Cl
ppm
1.000
3.059
1.123
2.031
4.122
6.196

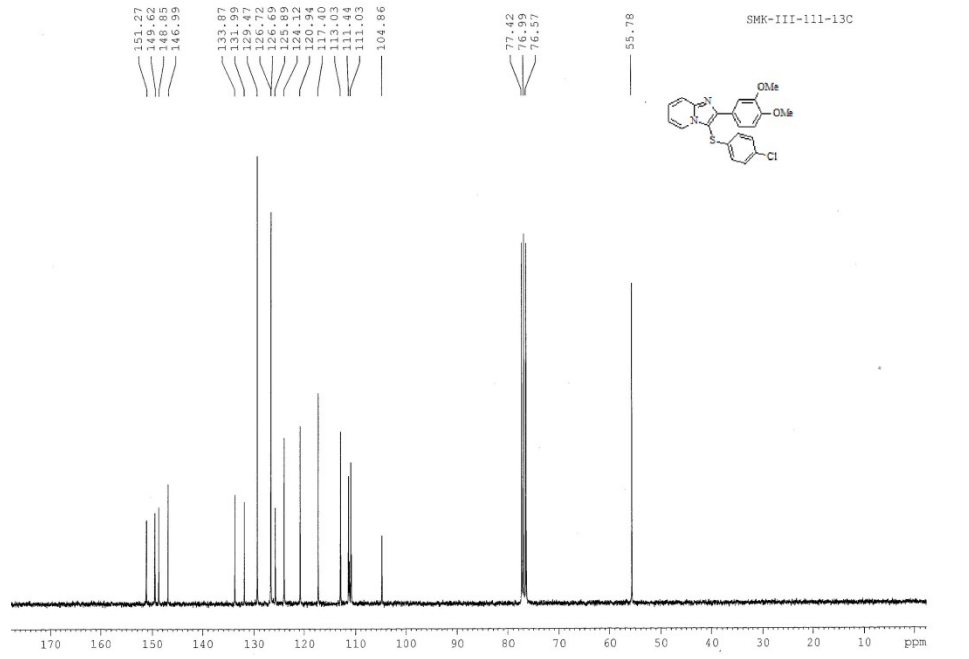
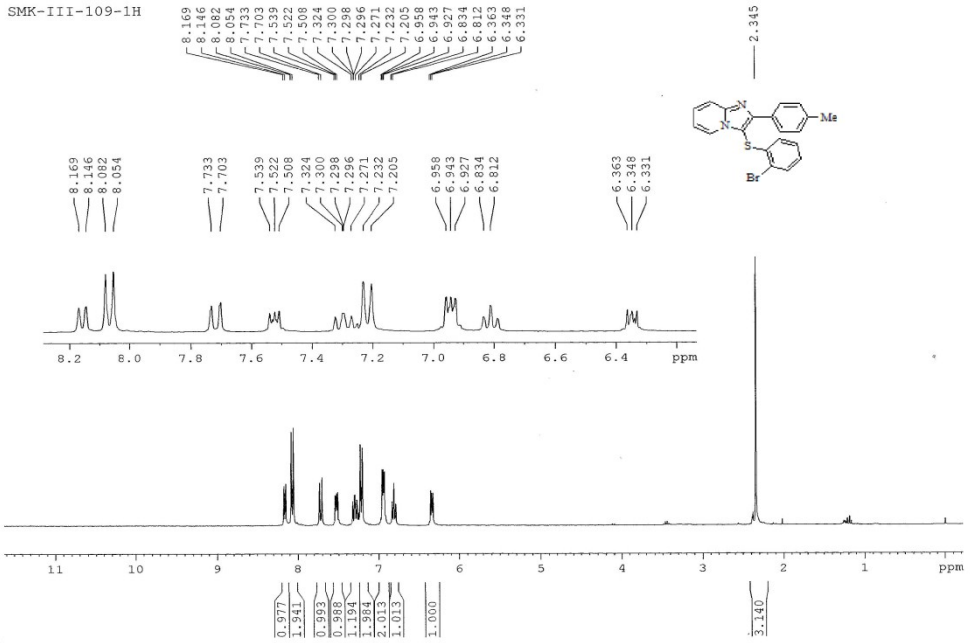

SMK-III-111-13C
151.27
149.62
148.85
146.99
133.87
131.99
129.47
126.72
126.69
125.89
124.12
120.94
117.40
113.03
111.44
111.03
104.86
77.42
76.99
76.57
55.78
OMe
OMe
Cl
ppm

Table 3, 8l:


SMK-III-109-1H
8.169
8.146
8.082
8.054
7.733
7.703
7.539
7.522
7.508
7.324
7.300
7.298
7.296
7.271
7.232
7.205
6.958
6.943
6.927
6.834
6.812
6.363
6.348
6.331
2.345
Me
N
S
Br
ppm
0.977
1.941
0.993
0.988
1.194
1.984
2.013
1.013
1.000
3.140

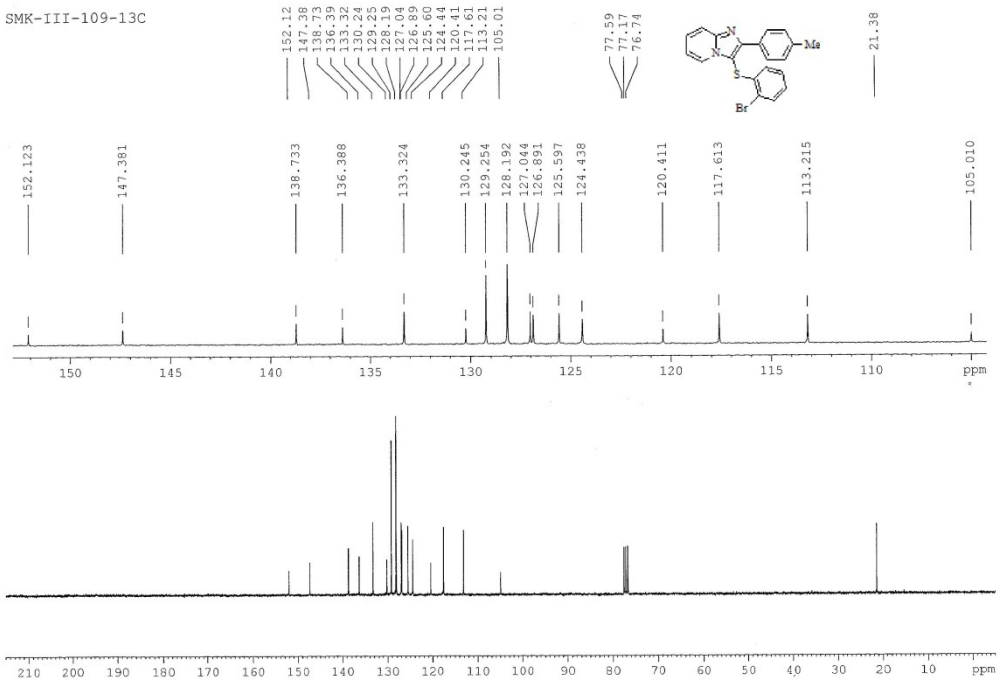
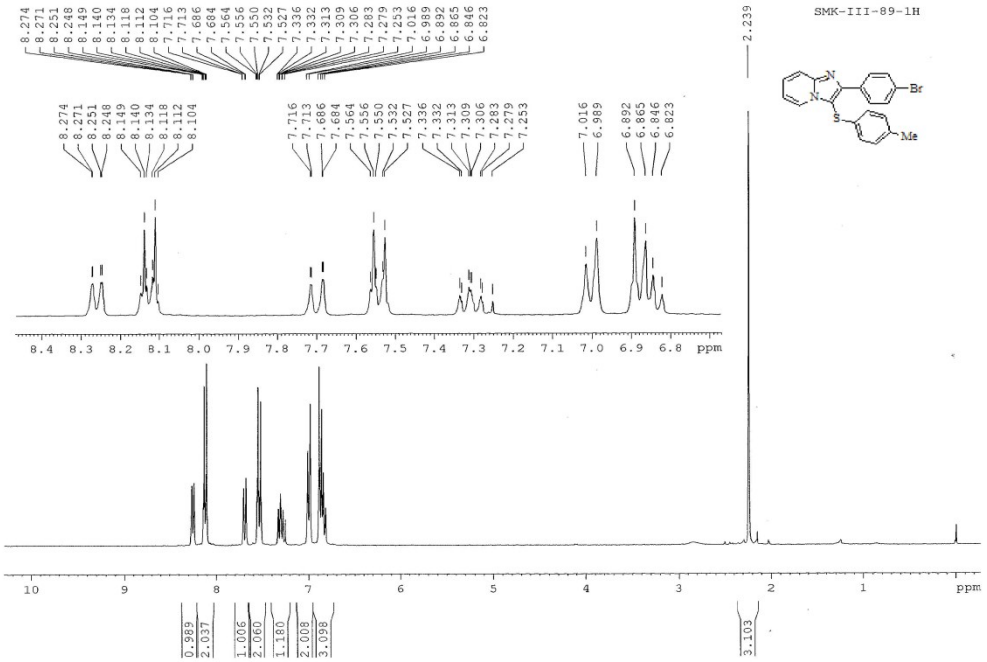

SMK-III-109-13C
152.12
147.38
138.73
136.39
133.32
130.24
129.25
128.19
127.04
126.89
125.60
124.44
120.41
117.61
113.21
105.01
77.59
77.17
76.74
21.38
Me
N
S
Br
ppm

Table 3, 8m:


SMK-III-89-1H
8.274
8.271
8.251
8.248
8.149
8.140
8.134
8.118
8.112
8.104
7.716
7.713
7.686
7.684
7.564
7.556
7.550
7.532
7.527
7.336
7.332
7.313
7.309
7.306
7.283
7.279
7.253
7.016
6.989
6.892
6.865
6.846
6.823
2.239
Br
Me
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 ppm
10 9 8 7 6 5 4 3 2 1 ppm
0.989
2.037
1.006
2.060
1.180
2.008
3.098
3.103

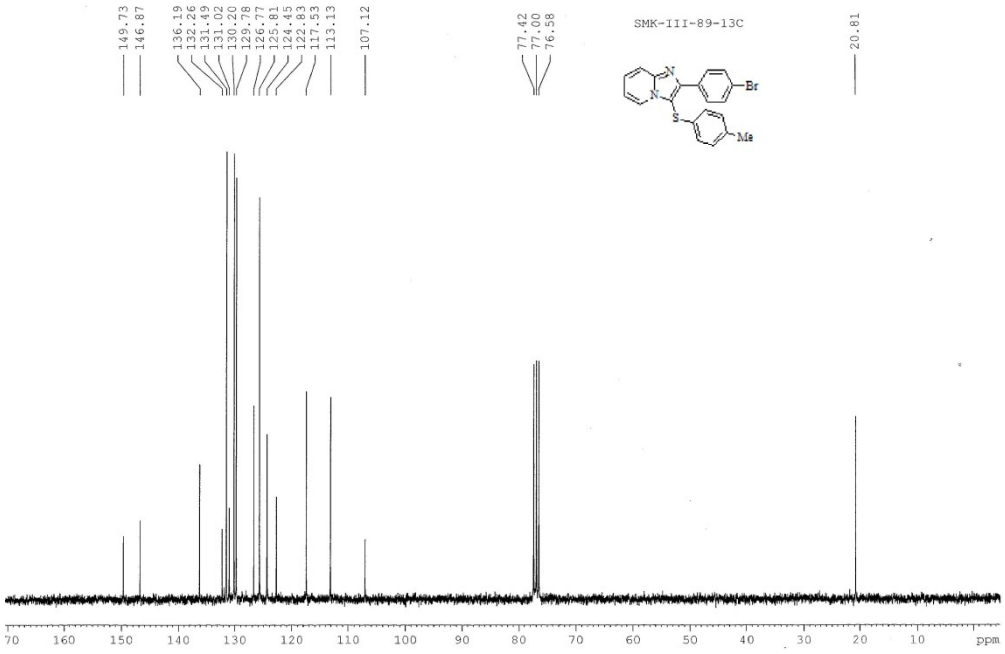
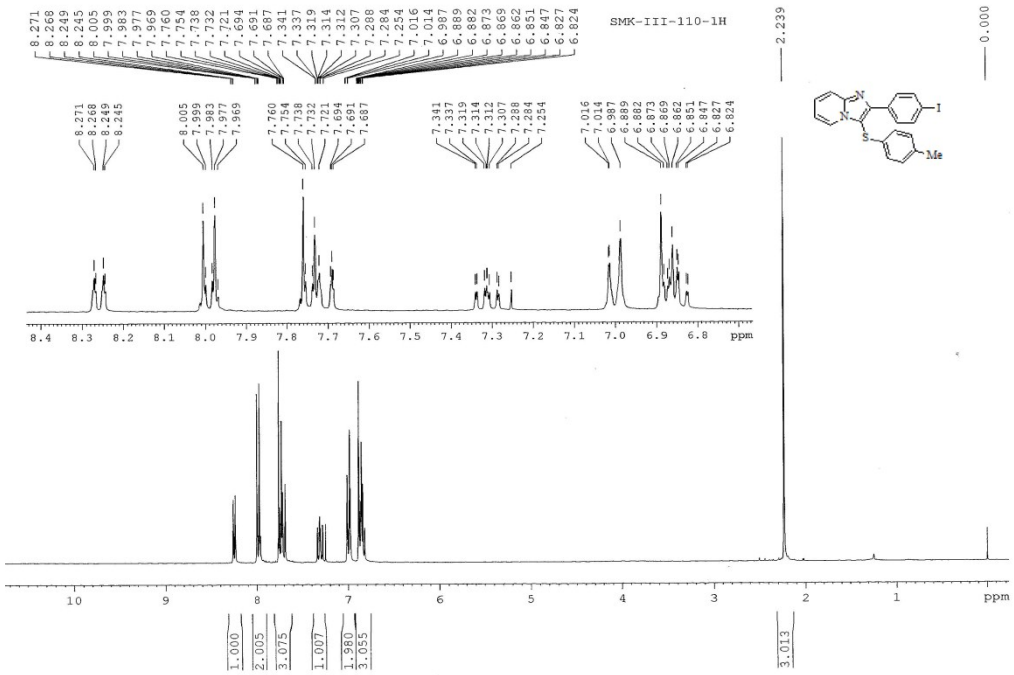

SMK-III-89-13C
149.73
146.87
136.19
132.26
131.49
131.02
130.20
129.78
126.77
125.81
124.45
122.83
117.53
113.13
107.12
77.42
77.00
76.58
20.81
Br
Me
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Table 3, 8n:



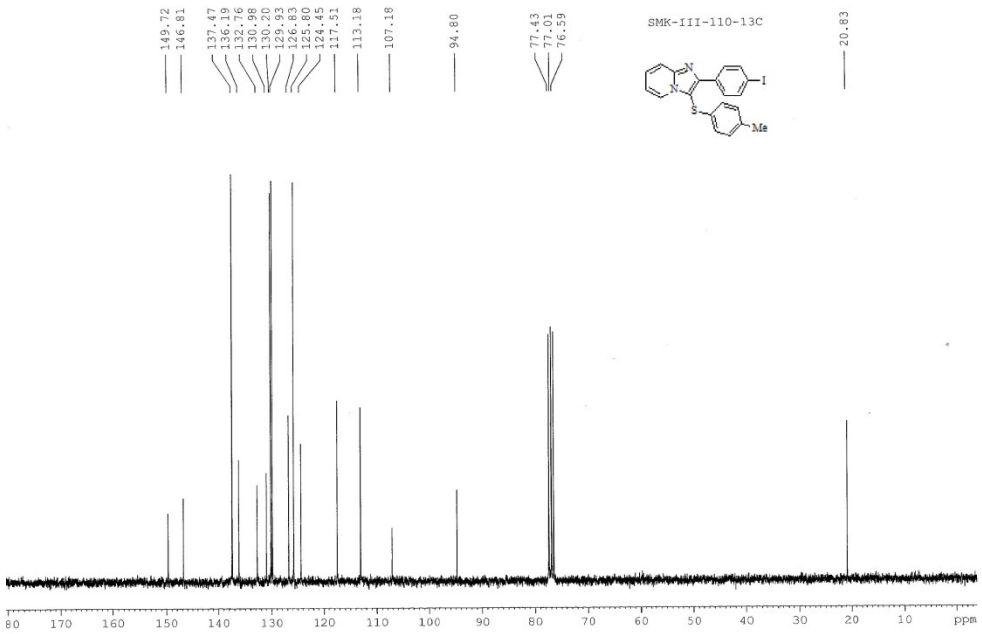
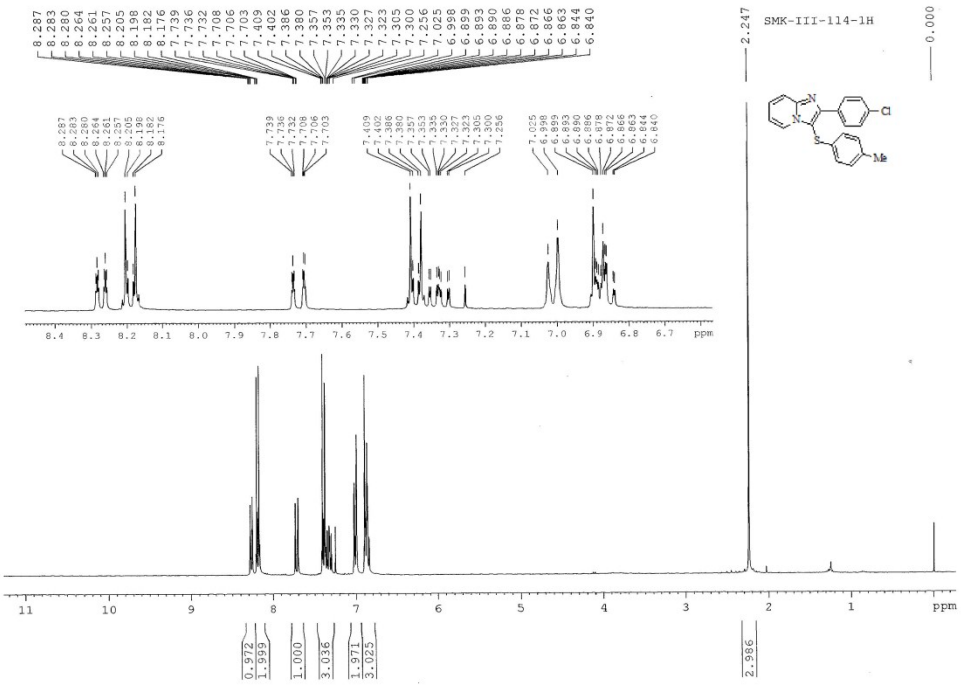


Table 3, 8o:


SMK-III-114-1H
8.287
8.283
8.280
8.264
8.261
8.257
8.205
8.198
8.182
8.176
7.739
7.736
7.732
7.708
7.706
7.703
7.409
7.402
7.386
7.380
7.357
7.353
7.335
7.330
7.327
7.323
7.305
7.300
7.256
7.025
6.998
6.899
6.893
6.890
6.886
6.878
6.872
6.866
6.863
6.844
6.840
2.247
0.000
Cl
S
Me
ppm
0.972
1.999
1.000
3.036
1.971
3.025
2.986

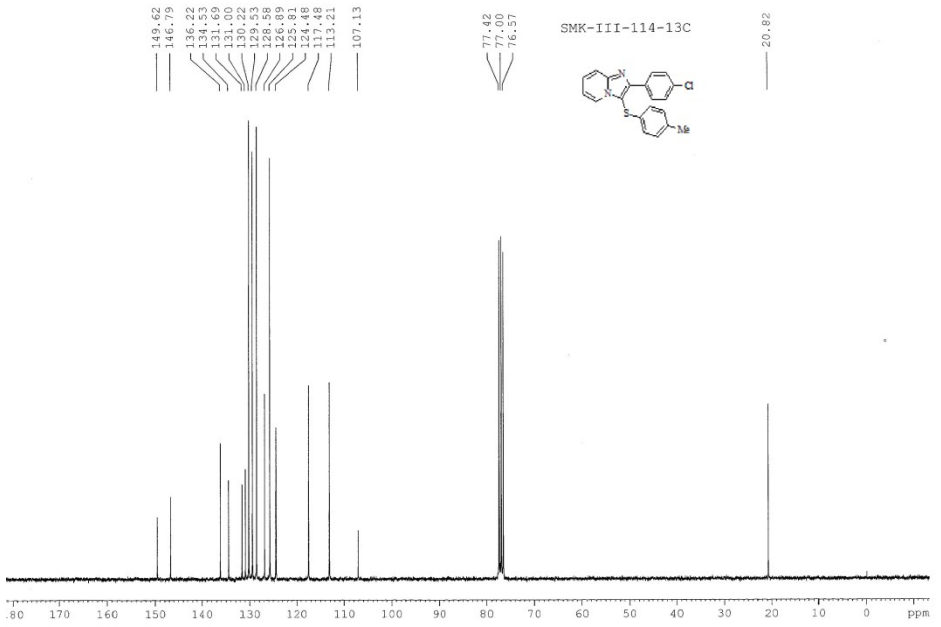
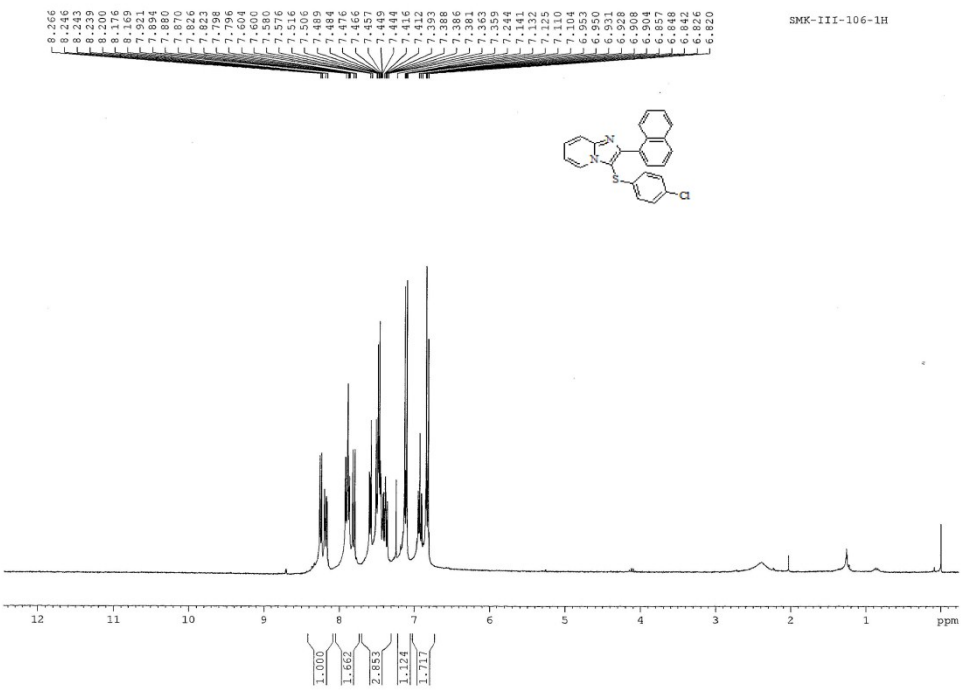

SMK-III-114-13C
149.62
146.79
136.22
134.53
131.69
131.00
130.22
129.53
128.58
126.89
125.81
124.48
117.48
113.21
107.13
77.42
77.00
76.57
20.82
Cl
S
Me
ppm

Table 3, 8p:


SMK-III-106-1H
8.266
8.246
8.243
8.239
8.200
8.176
8.169
7.921
7.894
7.880
7.870
7.826
7.823
7.798
7.796
7.604
7.600
7.580
7.576
7.516
7.506
7.489
7.484
7.476
7.466
7.457
7.449
7.444
7.416
7.412
7.393
7.388
7.386
7.381
7.363
7.359
7.244
7.141
7.132
7.125
7.110
7.104
6.953
6.950
6.931
6.928
6.908
6.904
6.857
6.848
6.842
6.826
6.820
N
N
S
Cl
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.662
2.853
1.124
1.717

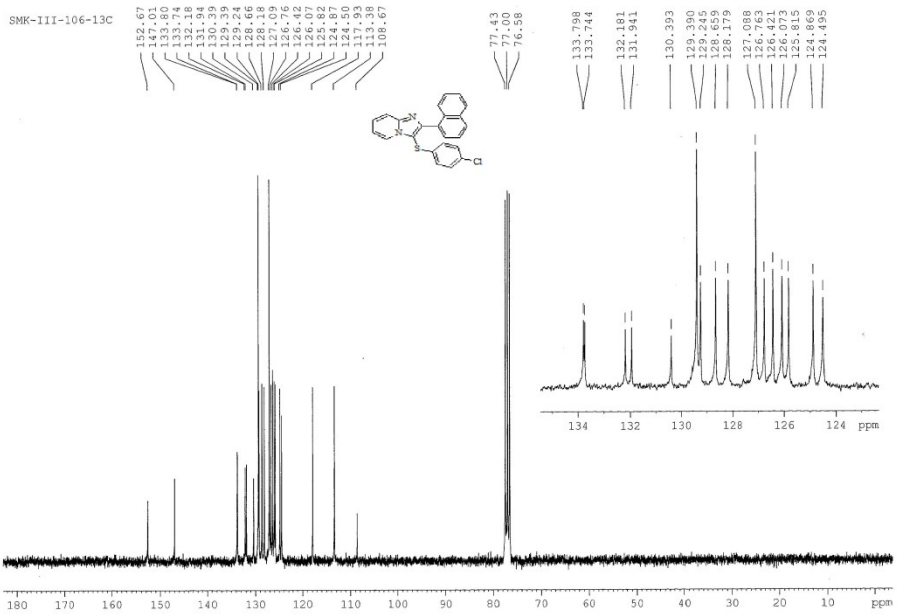
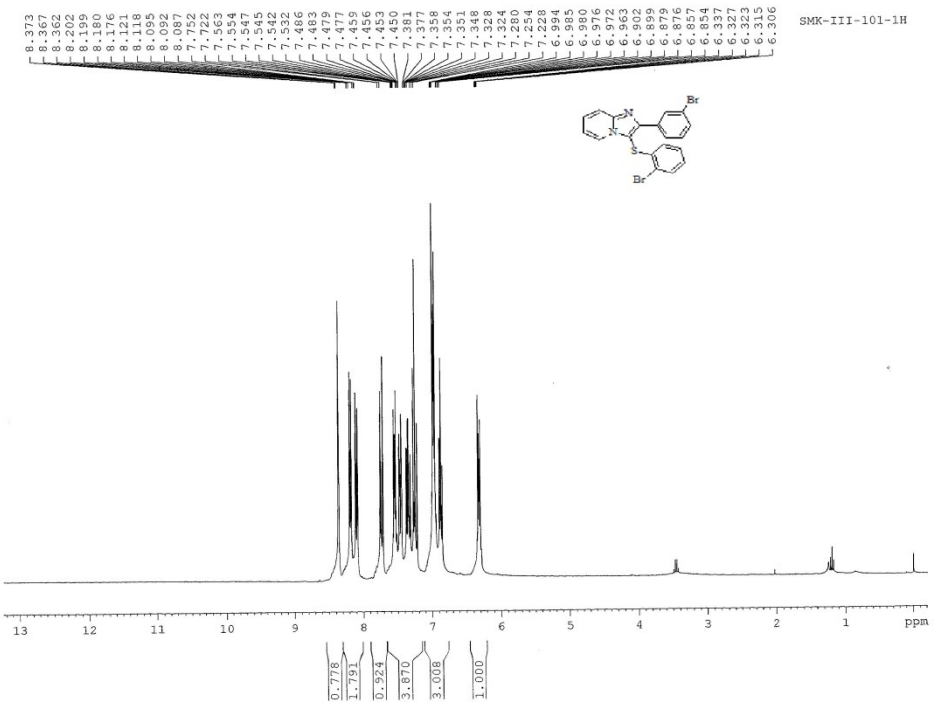

SMK-III-106-13C
152.67
147.01
133.80
133.74
132.18
131.94
130.39
129.39
129.24
128.66
128.18
127.09
126.76
126.42
126.07
125.82
125.67
124.50
117.93
113.38
108.67
77.43
77.00
76.58
133.798
133.744
132.181
131.941
130.393
129.390
129.245
128.659
128.179
127.088
126.763
126.421
126.073
126.015
124.869
124.495
N
N
S
Cl
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
134
132
130
128
126
124
ppm

Table 3, 8q:


SMK-III-101-1H
8.373
8.367
8.362
8.202
8.199
8.180
8.176
8.121
8.118
8.095
8.092
8.087
7.752
7.722
7.563
7.554
7.547
7.545
7.542
7.532
7.486
7.483
7.479
7.477
7.459
7.456
7.453
7.450
7.381
7.377
7.358
7.354
7.351
7.348
7.328
7.324
7.280
7.254
7.228
6.994
6.985
6.980
6.976
6.972
6.963
6.902
6.899
6.879
6.876
6.857
6.854
6.337
6.327
6.323
6.315
6.306
Br
N
S
Br
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.778
1.791
0.924
3.870
3.008
1.000

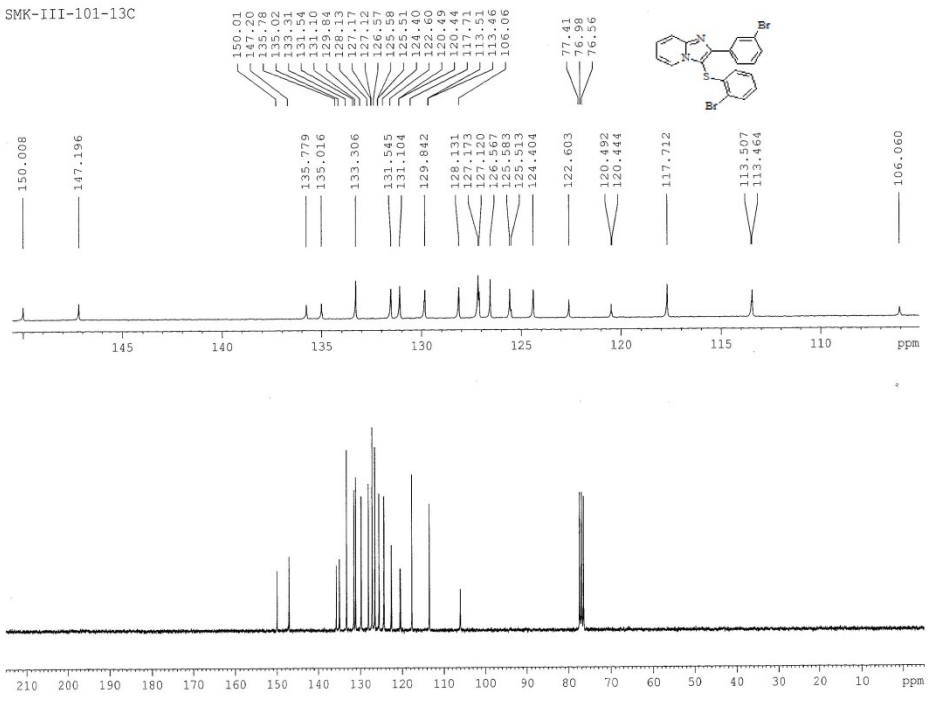
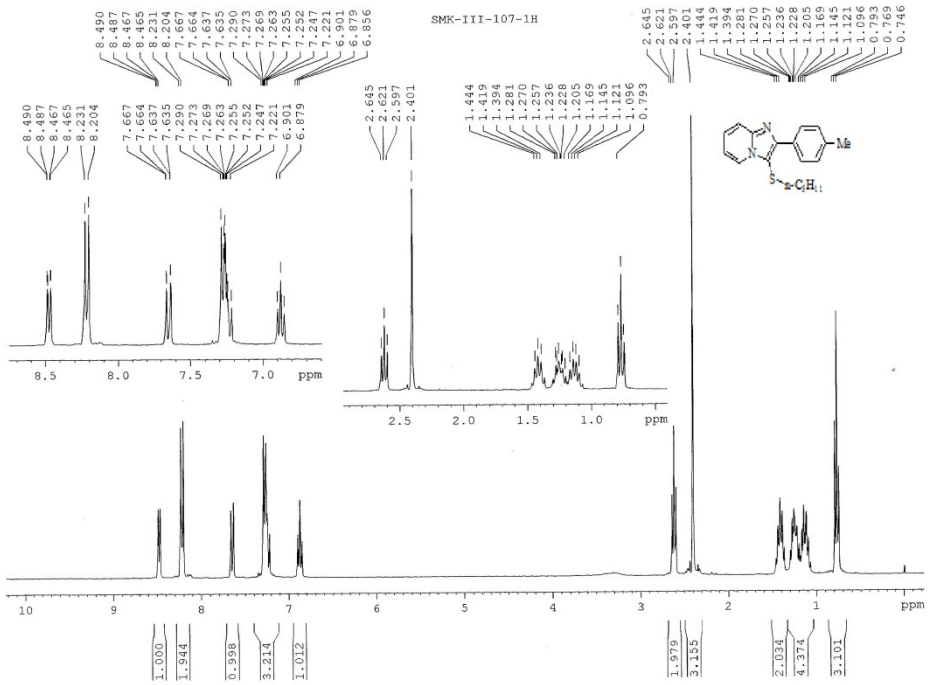
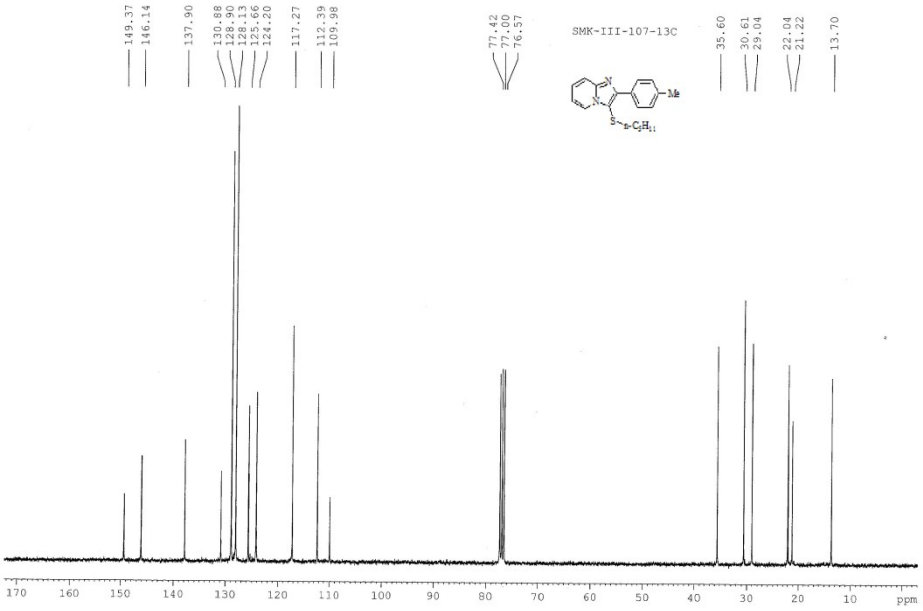

SMK-III-101-13C
150.01
147.20
135.78
135.02
133.31
131.54
131.10
129.84
128.13
127.17
127.12
126.57
125.58
125.51
124.40
122.60
120.49
120.44
117.71
113.51
113.46
106.06
77.41
76.98
76.56
Br
N
S
Br
150.008
147.196
135.779
135.016
133.306
131.545
131.104
129.842
128.131
127.173
127.120
126.567
125.583
125.513
124.404
122.603
120.492
120.444
117.712
113.507
113.464
106.060
145
140
135
130
125
120
115
110
ppm
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

Table 3, 8r:





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