Facile assembly of indeno[1,2-*c*]chromenes via a palladium-catalyzed reaction of 2-alkynylhalobenzene

Xiaolin Pan,[a] Hongming Nie,[b] Yong Luo,[a] Yueqiu Gao,*[,b] and Jie Wu*[,a,c]

[a] *Department of Chemistry, Fudan University, 220 Handan Road, Shanghai 200433, China*

[b] *Department of hepatology disease, Shanghai Shuguang hospital, Shanghai University of T.C.M., Shanghai 201203, China*

[c] *State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Road, Shanghai 200032, China*

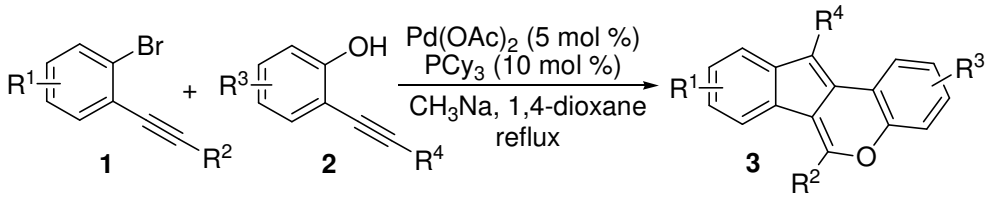
jie_wu@fudan.edu.cn; gaoyueqiu@hotmail.com

Supporting Information

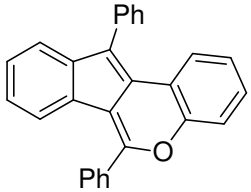
General experimental methods:

All reactions were performed in reaction tubes under air atmosphere. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr (house vacuum) at 25–35°C. Commercial reagents and solvents were used as received. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale.

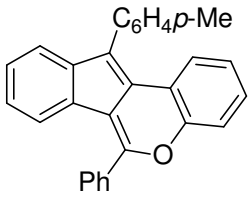
General experimental procedure for the synthesis of indeno[1,2-c]chromenes via a Pd-catalyzed reaction of 2-alkynylbromobenzene with 2-alkynylphenol.



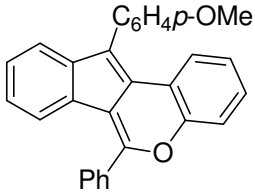
2-Alkynylhalobenzene (0.40 mmol) was added to a mixture of $Pd(OAc)_2$ (5 mol %), tricyclohexylphosphine (10 mol %), CH_3ONa (0.80 mmol), and 2-alkynylphenol (0.60 mmol) in 1,4-dioxane (3.0 mL). The mixture was stirred under reflux. After completion of the reaction as indicated by TLC, the mixture was cooled and diluted by EtOAc (10 mL), washed with saturated brine (2 × 10 mL), and dried by anhydrous Na_2SO_4. Evaporation of the solvent followed by purification on silica gel provided the product **3**.



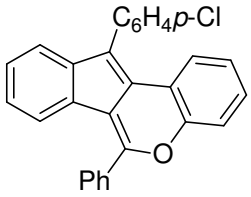
6,11-Diphenylindeno[1,2-c]chromene (**3a**). Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.87 (m, 2H), 7.69 (dd, J = 8.0, 1.2 Hz, 1H), 7.62-7.44 (m, 10H), 7.38-7.29 (m, 3H), 7.11-7.04 (m, 2H). ^{13}C NMR (100 MHz) δ 152.6, 150.1, 144.4, 136.6, 133.8, 130.5, 130.0, 129.7, 129.5, 128.9, 128.8, 127.7, 127.4, 126.7, 126.5, 124.9, 124.3, 123.8, 122.8, 121.5, 120.1, 119.3, 117.7, 117.3. HRMS (ESI) calculated for $C_{28}H_{18}O$ $[M+H]^+$ requires 371.1436, found 371.1424.



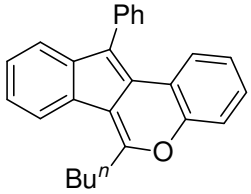
6-Phenyl-11-(*p*-tolyl)indeno[1,2-c]chromene (**3b**). Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89-7.87 (m, 2H), 7.75-7.73 (m, 1H), 7.63-7.60 (m, 3H), 7.50-7.43 (m, 4H), 7.39-7.35 (m, 3H), 7.33-7.29 (m, 2H), 7.10-7.05 (m, 2H), 2.49 (s, 3H). ^{13}C NMR (100 MHz) δ 152.4, 150.1, 144.5, 137.1, 133.9, 133.5, 130.5, 129.8, 129.6, 129.5, 128.8, 127.6, 126.65, 126.60, 124.9, 124.2, 123.6, 122.7, 121.4, 120.2, 119.4, 117.6, 117.3, 21.5. HRMS (ESI) calculated for $C_{29}H_{20}O$ $[M+H]^+$ requires 385.1592, found 385.1599.



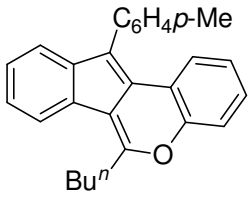
11-(4-Methoxyphenyl)-6-phenylindeno[1,2-c]chromene (**3c**). Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.88 (m, 2H), 7.73 (dd, J = 8.0, 1.2 Hz, 1H), 7.64-7.62 (m, 3H), 7.52-7.49 (m, 3H), 7.46-7.44 (m, 1H), 7.39-7.31 (m, 3H), 7.12-7.07 (m, 4H), 3.95 (s, 3H). ^{13}C NMR (100 MHz) δ 158.9, 134.2, 131.1, 130.5, 129.5, 128.7, 127.6, 126.7, 124.8, 124.2, 122.7, 121.4, 119.3, 117.7, 114.3, 55.3. HRMS (ESI) calculated for $C_{29}H_{20}O_2$ $[M+H]^+$ requires 401.1542, found 401.1530.



11-(4-Chlorophenyl)-6-phenylindeno[1,2-c]chromene (**3d**). Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88-7.86 (m, 2H), 7.66 (dd, J = 8.4, 1.2Hz, 1H), 7.62-7.60 (m, 3H), 7.54-7.49 (m, 5H), 7.46-7.44 (m, 1H), 7.35-7.32 (m, 3H), 7.11-7.08 (m, 2H). ^{13}C NMR (100 MHz) δ 152.9, 150.1, 143.9, 135.1, 133.7, 133.2, 131.4, 130.6, 129.7, 129.5, 129.2, 128.8, 127.9, 126.8, 125.0, 124.7, 124.4, 124.1, 122.9, 121.5, 119.8, 119.0, 117.8, 117.2. HRMS (ESI) calculated for $C_{28}H_{17}ClO$ $[M+H]^+$ requires 405.1046, found 405.1045.

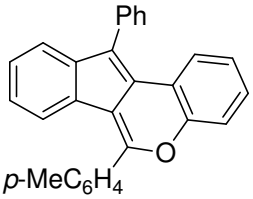


6-Butyl-11-phenylindeno[1,2-c]chromene (**3e**). Yellow solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, J = 7.6 Hz, 1H), 7.64 (d, J = 8.0Hz, 1H), 7.55-7.51 (m, 4H), 7.47-7.27 (m, 6H), 7.05-7.00 (m, 1H), 3.23 (t, J = 7.6 Hz, 2H), 1.99-1.91 (m, 2H), 1.61-1.55 (m, 2H), 1.02 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz) δ 157.2, 149.9, 144.1, 136.8, 130.1, 129.8, 128.8, 127.4, 127.3, 125.9, 125.2, 124.9, 124.1, 123.4, 122.9, 121.5, 120.2, 119.5, 117.4, 116.2, 32.4, 29.6, 22.7, 13.9. HRMS (ESI) calculated for $C_{26}H_{22}O$ $[M+H]^+$ requires 351.1749, found 351.1746.

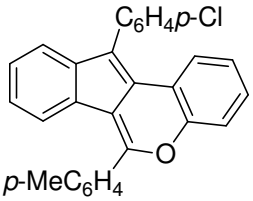


6-Butyl-11-(p-tolyl)indeno[1,2-c]chromene (**3f**). Yellow solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J = 7.2 Hz, 1H), 7.69 (dd, J = 8.0, 0.9 Hz, 1H), 7.44-7.32(m, 8H), 7.29-7.25(m, 1H), 7.04-7.01 (m, 1H), 3.21 (t, J = 7.6 Hz, 2H), 2.47 (s, 3H), 1.97-1.90 (m, 2H), 1.59-1.53 (m, 2H), 1.01 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz) δ 157.0, 149.9,

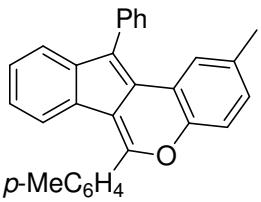
144.2, 136.8, 133.7, 129.9, 129.8, 129.5, 127.3, 125.9, 125.2, 124.9, 124.0, 123.2, 122.8, 121.5, 120.3, 119.6, 117.4, 116.3, 32.3, 29.6, 22.7, 21.4, 14.0. HRMS (ESI) calculated for $C_{27}H_{24}O$ $[M+H]^+$ requires 365.1905, found 365.1890.



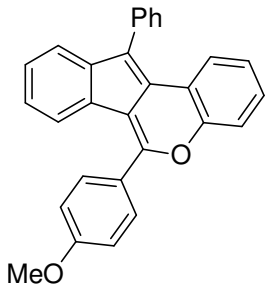
11-Phenyl-6-(p-tolyl)indeno[1,2-c]chromene **(3g)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78 (d, J = 8.0 Hz, 2H), 7.68 (dd, J = 8.0, 1.2 Hz, 1H), 7.60-7.53 (m, 5H), 7.48-7.40 (m, 4H), 7.37-7.28 (m, 3H), 7.11-7.03 (m, 2H), 2.51 (s, 3H). ^{13}C NMR (100 MHz) δ 152.9, 150.1, 144.3, 140.8, 136.7, 130.9, 130.0, 129.8, 129.45, 129.43, 128.9, 127.6, 127.4, 126.6, 126.3, 124.8, 124.2, 123.8, 122.7, 121.5, 120.1, 119.3, 117.7, 117.0, 21.7. HRMS (ESI) calculated for $C_{29}H_{20}O$ $[M+H]^+$ requires 385.1592, found 385.1579.



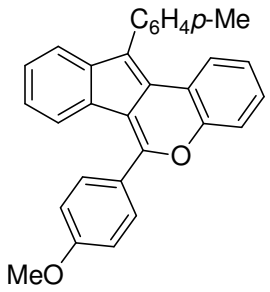
11-(4-Chlorophenyl)-6-(*p*-tolyl)indeno[1,2-c]chromene **(3h)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (d, J = 8.0 Hz, 2H), 7.66 (dd, J = 8.0, 1.2Hz, 1H), 7.58-7.56 (m, 1H), 7.56-7.52(m, 4H), 7.47-7.41 (m, 3H), 7.36-7.32 (m, 3H), 7.12-7.10 (m, 2H), 2.52 (s, 3H). ^{13}C NMR (100 MHz) δ 153.2, 150.2, 143.9, 140.9, 135.2, 134.2, 133.2, 131.5, 130.8, 129.8, 129.5, 129.4, 129.2, 127.9, 126.6, 124.7, 124.3, 122.8, 121.6, 119.8, 119.0, 117.8, 117.0, 21.7. HRMS (ESI) calculated for $C_{29}H_{19}ClO$ $[M+H]^+$ requires 419.1203, found 419.1178.



2-Methyl-11-phenyl-6-(*p*-tolyl)indeno[1,2-c]chromene **(3i)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, J = 8.0 Hz, 2H), 7.60-7.52 (m, 5H), 7.47-7.44(m, 2H), 7.41-7.37(m, 3H), 7.34-7.29 (m, 2H), 7.11-7.07 (m, 2H), 2.50 (s, 3H), 2.18 (s, 3H). ^{13}C NMR (100 MHz) δ 153.0, 148.3, 144.2, 140.7, 136.7, 133.6, 131.0, 130.0, 129.8, 129.4, 128.73, 128.67, 127.4, 126.4, 125.9, 124.8, 124.0, 122.5, 121.4, 119.7, 119.2, 117.4, 116.9, 21.6, 21.2. HRMS (ESI) calculated for $C_{30}H_{22}O$ $[M+H]^+$ requires 399.1749, found 399.1760.

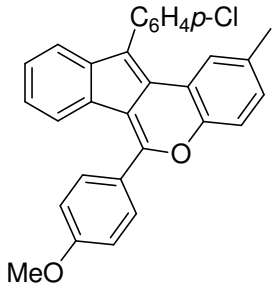


6-(4-Methoxyphenyl)-11-phenylindeno[1,2-c]chromene **(3j)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, J = 8.4 Hz, 2H), 7.68 (d, J = 8.0Hz, 1H), 7.63-7.53 (m, 5H), 7.48-7.42 (m, 2H), 7.38-7.28 (m, 3H), 7.13-7.10 (m, 3H), 7.07-7.03 (m, 1H), 3.92 (s, 3H). ^{13}C NMR (100 MHz) δ 161.3, 152.7, 150.1, 144.2, 136.7, 131.1, 130.0, 129.9, 128.9, 127.6, 127.4, 126.5, 126.0, 124.8, 124.1, 123.9, 122.6, 121.4, 120.1, 119.3, 117.6, 116.8, 114.1, 113.5, 55.4. HRMS (ESI) calculated for $C_{29}H_{20}O_2$ $[M+H]^+$ requires 401.1542, found 401.1525.

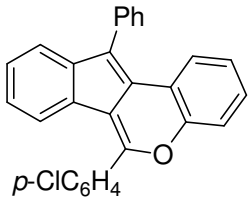


6-(4-Methoxyphenyl)-11-(*p*-tolyl)indeno[1,2-c]chromene **(3k)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.81 (m, 2H), 7.73 (dd, J = 8.0, 1.2Hz, 1H), 7.60 (d, J = 7.6Hz, 1H), 7.47-7.45 (m, 2H), 7.43-7.26 (m, 6H), 7.11-7.03 (m, 4H), 3.90 (s, 3H), 2.48 (s, 3H). ^{13}C NMR (100 MHz) δ 161.2, 152.6, 150.1, 144.3, 136.9, 133.6, 131.1, 129.8, 129.6, 127.5, 126.5, 126.2, 126.1, 124.8, 124.1, 123.8, 122.6, 121.4, 120.2, 119.3, 117.6, 116.9, 114.1,

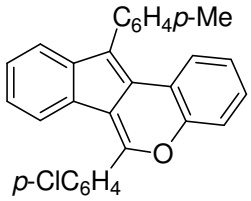
55.4, 21.4. HRMS (ESI) calculated for $C_{30}H_{22}O_2$ $[M+H]^+$ requires 415.1698, found 415.1694.



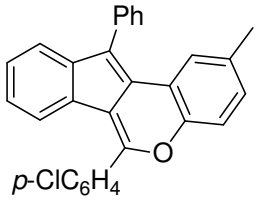
11-(4-Chlorophenyl)-6-(4-methoxyphenyl)-2-methylindeno[1,2-c]chromene **(3l)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.4 Hz, 2H), 7.61-7.59 (m, 1H), 7.53(m, 4H), 7.47 (s, 1H), 7.37-7.32 (m, 3H), 7.16-7.10 (m, 4H), 3.95 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (100 MHz) δ 161.3, 153.1, 148.3, 143.8, 135.3, 133.8, 133.1, 131.5, 131.1, 129.8, 128.9, 126.5, 126.0, 124.5, 124.4, 124.2, 122.7, 121.4, 119.5, 118.9, 117.5, 116.6, 114.1, 55.5, 21.3. HRMS (ESI) calculated for $C_{30}H_{21}ClO_2$ $[M+H]^+$ requires 449.1308, found 449.1326.



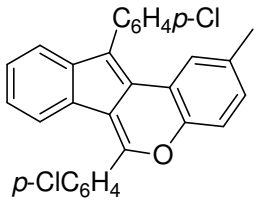
6-(4-Chlorophenyl)-11-phenylindeno[1,2-c]chromene **(3m)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.4 Hz, 2H), 7.67 (dd, J = 8.0, 1.2Hz, 1H), 7.60-7.56 (m, 6H), 7.52-7.42 (m, 3H), 7.37-7.30 (m, 3H), 7.14-7.05 (m, 2H). ^{13}C NMR (100 MHz) δ 151.2, 150.0, 144.5, 136.7, 136.5, 132.2, 130.9, 129.9, 129.5, 129.1, 128.9, 127.8, 127.5, 126.92, 126.86, 124.9, 124.4, 123.7, 122.9, 121.3, 119.9, 119.5, 117.6. HRMS (ESI) calculated for $C_{28}H_{17}ClO$ $[M+H]^+$ requires 405.1046, found 405.1024.



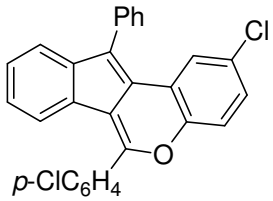
6-(4-Chlorophenyl)-11-(*p*-tolyl)indeno[1,2-c]chromene **(3n)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, J = 8.4 Hz, 2H), 7.72 (dd, J = 8.0, 1.2Hz, 1H), 7.60-7.58 (m, 2H), 7.51-7.41 (m, 4H), 7.37-7.29 (m, 5H), 7.13-7.06 (m, 2H), 2.50 (s, 3H). ^{13}C NMR (100 MHz) δ 151, 150.0, 144.6, 137.2, 136.6, 134.2, 133.3, 132.2, 131.0, 129.7, 129.6, 129.1, 128.8, 127.7, 126.9, 124.9, 124.3, 123.5, 122.9, 121.3, 120.1, 119.5, 117.6, 21.5. HRMS (ESI) calculated for $C_{29}H_{19}ClO$ $[M+H]^+$ requires 419.1203, found 419.1191.



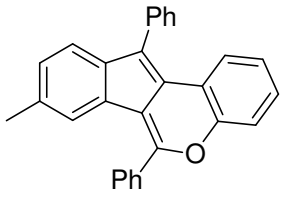
6-(4-Chlorophenyl)-2-methyl-11-phenylindeno[1,2-c]chromene **(3o)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.0 Hz, 2H), 7.60-7.56 (m, 6H), 7.51-7.45 (m, 3H), 7.39-7.32 (m, 3H), 7.14-7.09 (m, 2H), 2.19 (s, 3H). ^{13}C NMR (100 MHz) δ 151.2, 148.2, 144.4, 136.5, 133.9, 132.3, 130.9, 129.9, 129.54, 129.51, 129.1, 128.8, 127.5, 126.8, 126.5, 124.8, 123.8, 122.8, 121.3, 119.6, 119.4, 117.3, 21.2. HRMS (ESI) calculated for $C_{29}H_{19}ClO$ $[M+H]^+$ requires 419.1203, found 419.1178.



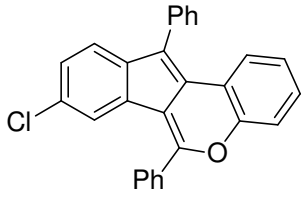
6,11-Bis(4-chlorophenyl)-2-methylindeno[1,2-c]chromene **(3p)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.81 (m, 2H), 7.61-7.59 (m, 2H), 7.55-7.46 (m, 6H), 7.36-7.33 (m, 3H), 7.18-7.10 (m, 2H), 2.24 (s, 3H). ^{13}C NMR (100 MHz) δ 151.5, 148.2, 144.1, 136.6, 135.0, 134.1, 133.3, 132.2, 131.4, 130.9, 129.4, 129.13, 129.09, 129.07, 126.9, 124.9, 124.6, 124.1, 122.9, 121.4, 119.4, 119.1, 117.5, 21.3. HRMS (ESI) calculated for $C_{29}H_{18}Cl_2O$ $[M+H]^+$ requires 453.0813, found 453.0780.



2-Chloro-6-(4-chlorophenyl)-11-phenylindeno[1,2-c]chromene **(3q)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (m, 2H), 7.62-7.51 (m, 9H), 7.38-7.36 (3H), 7.28-7.27 (m, 1H), 7.17-7.13 (m, 1H). ^{13}C NMR (100 MHz) δ 148.4, 136.8, 135.6, 134.3, 131.9, 130.9, 129.7, 129.5, 129.2, 129.1, 127.9, 127.7, 127.1, 124.2, 123.4, 121.4, 121.3, 119.8, 118.9. HRMS (ESI) calculated for $C_{28}H_{16}Cl_2O$ $[M+H]^+$ requires 439.0656, found 439.0635.

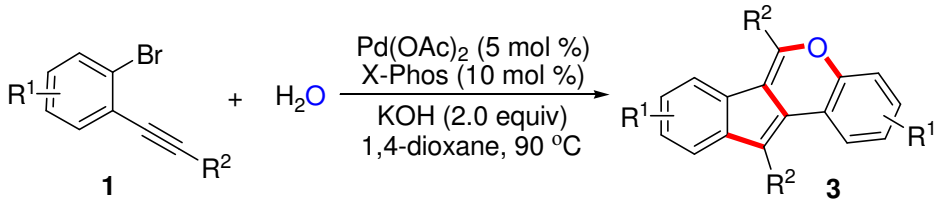


8-Methyl-6,11-diphenylindeno[1,2-c]chromene **(3r)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89-7.87 (m, 2H), 7.67-7.53 (m, 8H), 7.47-7.41 (m, 2H), 7.31-7.21 (m, 3H), 7.16-7.14 (m, 1H), 7.06-7.02 (m, 1H), 2.30 (s, 3H). ^{13}C NMR (100 MHz) δ 152.3, 150.0, 142.1, 136.8, 133.9, 132.4, 130.5, 130.1, 129.9, 129.6, 128.8, 128.7, 127.8, 127.5, 127.4, 126.5, 124.7, 124.2, 123.0, 121.9, 120.2, 119.1, 117.6, 117.3, 21.8. HRMS (ESI) calculated for $C_{29}H_{20}O$ $[M+H]^+$ requires 385.1592, found 385.1590.

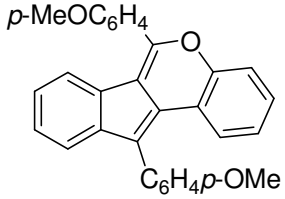


8-Chloro-6,11-diphenylindeno[1,2-c]chromene **(3s)**. Red solid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89-7.87 (m, 2H), 7.67-7.66 (m, 4H), 7.59-7.56 (m, 4H), 7.51-7.45 (m, 3H), 7.36-7.32 (m, 1H), 7.30-7.25 (m, 2H), 7.10-7.07 (m, 1H). ^{13}C NMR (100 MHz) δ 153.7, 150.1, 142.6, 136.2, 134.3, 133.3, 130.9, 129.9, 129.4, 128.98, 128.95, 128.5, 127.9, 127.6, 126.7, 125.8, 124.8, 124.5, 124.2, 121.4, 120.1, 119.9, 117.7, 116.5. HRMS (ESI) calculated for $C_{28}H_{17}ClO$ $[M+H]^+$ requires 405.1046, found 405.1033.

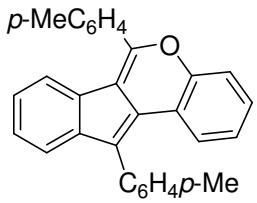
General procedure of the synthesis of indeno[1,2-c]chromenes via a palladium-catalyzed reaction of 2-alkynylbromobenzenes with water



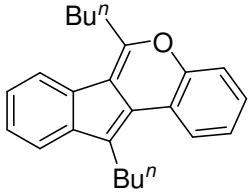
To a mixture of $Pd(OAc)_2$ (0.02 mmol, 5 mol %), X-Phos (0.04 mmol, 10 mol %), KOH (0.8 mmol), and 2-alkynylhalobenzene **1** (0.4 mmol) in 1,4-dioxane (2.0 mL) was added water (1.0 mmol). The mixture was heated at 90 °C. After completion of the reaction as indicated by TLC, the solvent was diluted by EtOAc (10 mL), washed with saturated brine (2 x 10 mL), and dried by Na_2SO_4. Evaporation the solvent followed by purification on silica gel provided the product **3**.



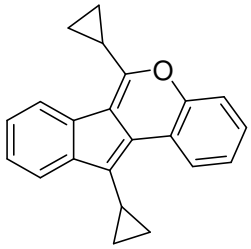
6,11-Bis(4-methoxyphenyl)indeno[1,2-*c*]chromene (**3t**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.8 Hz, 2H), 7.73-7.71 (m, 1H), 7.61-7.59 (m, 1H), 7.49 (d, *J* = 8.2 Hz, 2H), 7.44-7.42 (m, 1H), 7.38-7.36 (m, 1H), 7.29 (d, *J* = 6.9 Hz, 2H), 7.12-7.05 (m, 6H), 3.93 (s, 3H), 3.92 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.3, 158.9, 152.5, 150.2, 144.5, 131.1, 129.9, 128.8, 127.5, 126.5, 126.1, 125.9, 124.8, 124.1, 123.8, 122.6, 121.4, 120.3, 119.3, 117.6, 114.3, 114.1, 113.8, 55.4, 55.3. HRMS (ESI) calcd for $C_{30}H_{23}O$: 431.1647 ($M + H^+$), found: 431.1640.



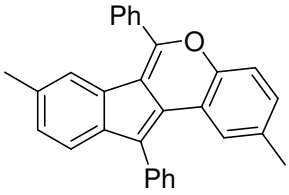
6,11-Di-p-tolylindeno[1,2-*c*]chromene (**3u**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.72 (m, 3H), 7.56 (d, *J* = 7.8 Hz, 1H), 7.48-7.45 (m, 2H), 7.42-7.39 (m, 2H), 7.36-7.35 (m, 3H), 7.33-7.30 (m, 3H), 7.09-7.06 (m, 2H), 2.51 (s, 3H), 2.49 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 150.2, 144.4, 140.7, 137.0, 133.6, 131.0, 129.8, 129.6, 129.4, 128.4, 127.6, 126.5, 126.4, 124.9, 124.1, 123.7, 122.7, 121.5, 120.3, 119.3, 117.6, 117.1, 21.6, 21.5. HRMS (ESI) calcd for $C_{30}H_{23}O$: 399.1749 ($M + H^+$), found: 399.1758.



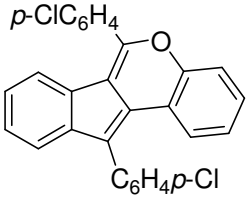
6,11-Dibutylindeno[1,2-*c*]chromene (**3v**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (s, 1H), 7.90 (d, *J* = 7.4 Hz, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.44-7.41 (m, 2H), 7.34-7.32 (m, 3H), 3.17-3.11 (m, 4H), 1.91-1.88 (m, 2H), 1.74-1.73 (m, 2H), 1.57-1.53 (m, 4H), 1.25(m, 4H), 1.00 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.5, 149.9, 144.0, 130.1, 126.8, 125.7, 125.0, 124.3, 122.6, 122.2, 121.5, 121.2, 118.4, 117.5, 116.2, 32.1, 31.3, 29.6, 26.1, 23.2, 22.7, 14.2, 13.9. HRMS (ESI) calcd for $C_{24}H_{27}O$: 331.2062 ($M + H^+$), found: 331.2066.



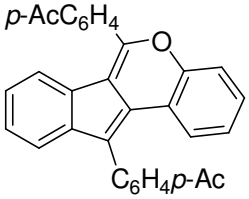
6,11-Dicyclopropylindeno[1,2-*c*]chromene (**3w**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67-8.66 (m, 1H), 8.07 (d, *J* = 7.3 Hz, 1H), 7.93 (d, *J* = 7.3 Hz, 1H), 7.45-7.42 (m, 1H), 7.33-7.29 (m, 4H), 2.76-2.75 (m, 1H), 2.08-2.07 (m, 1H), 1.39-1.28 (m, 2H), 1.24-1.18 (m, 4H), 0.75-0.74 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.9, 149.5, 144.3, 130.2, 126.9, 126.7, 125.4, 125.2, 124.2, 123.9, 122.4, 121.5, 120.9, 119.9, 116.8, 116.2, 12.8, 8.2, 7.7. HRMS (ESI) calcd for $C_{22}H_{19}O$: 299.1436 ($M + H^+$), found: 299.1440.



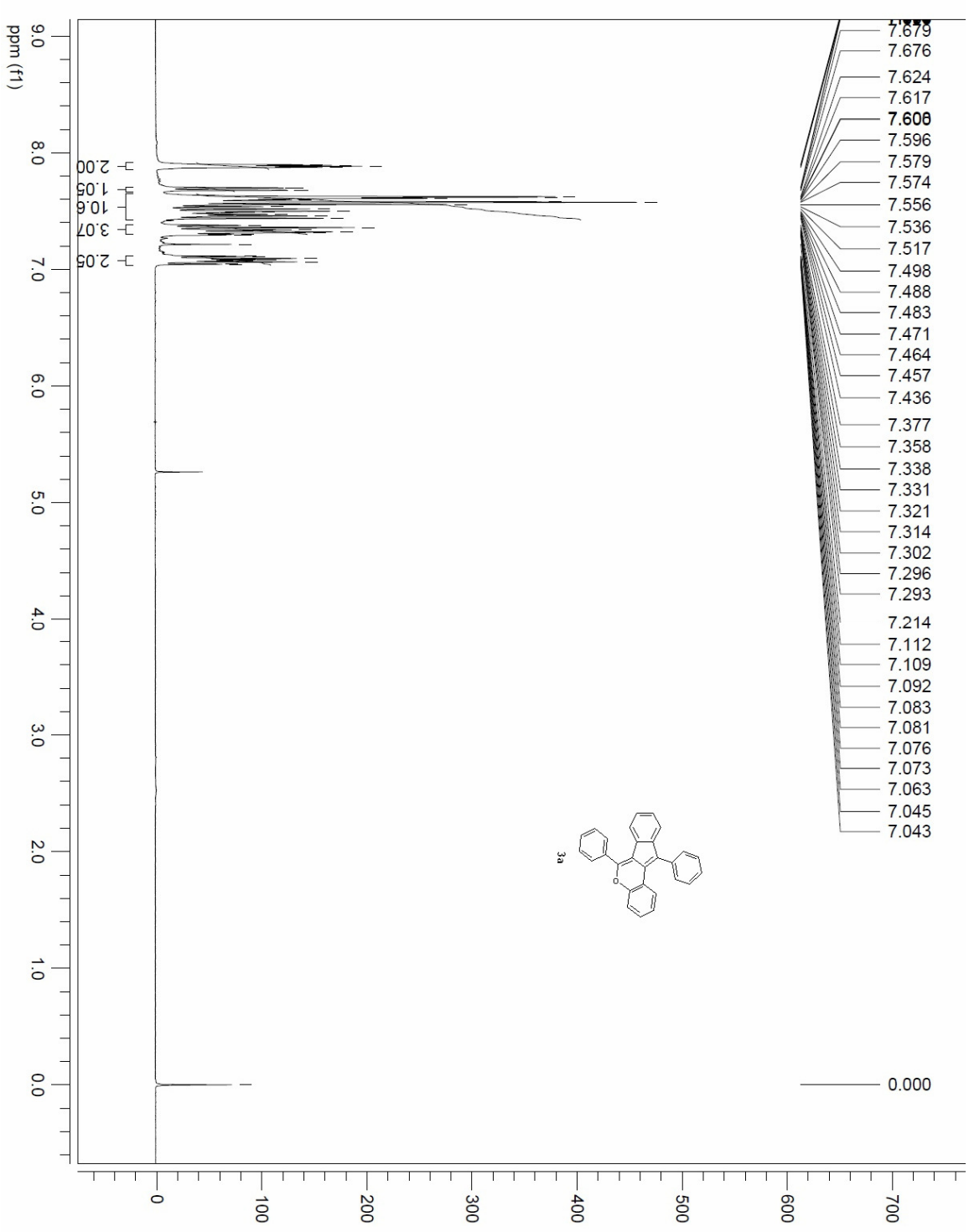
2,8-Dimethyl-6,11-diphenylindeno[1,2-*c*]chromene (**3x**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.8 Hz, 3H), 7.52-7.50 (m, 4H), 7.46-7.44 (m, 3H), 7.02 (m, 2H), 6.89 (d, *J* = 7.3 Hz, 2H), 6.69 (d, *J* = 7.3 Hz, 2H), 2.19 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.0, 142.4, 140.5, 137.3, 135.5, 134.1, 130.0, 130.3, 129.6, 129.1, 128.6, 128.5, 128.0, 127.9, 127.4, 127.3, 127.0, 122.8, 122.1, 119.0, 117.4, 110.5, 21.4. HRMS (ESI) calcd for $C_{30}H_{23}O$: 399.1749 ($M + H^+$), found: 399.1764.

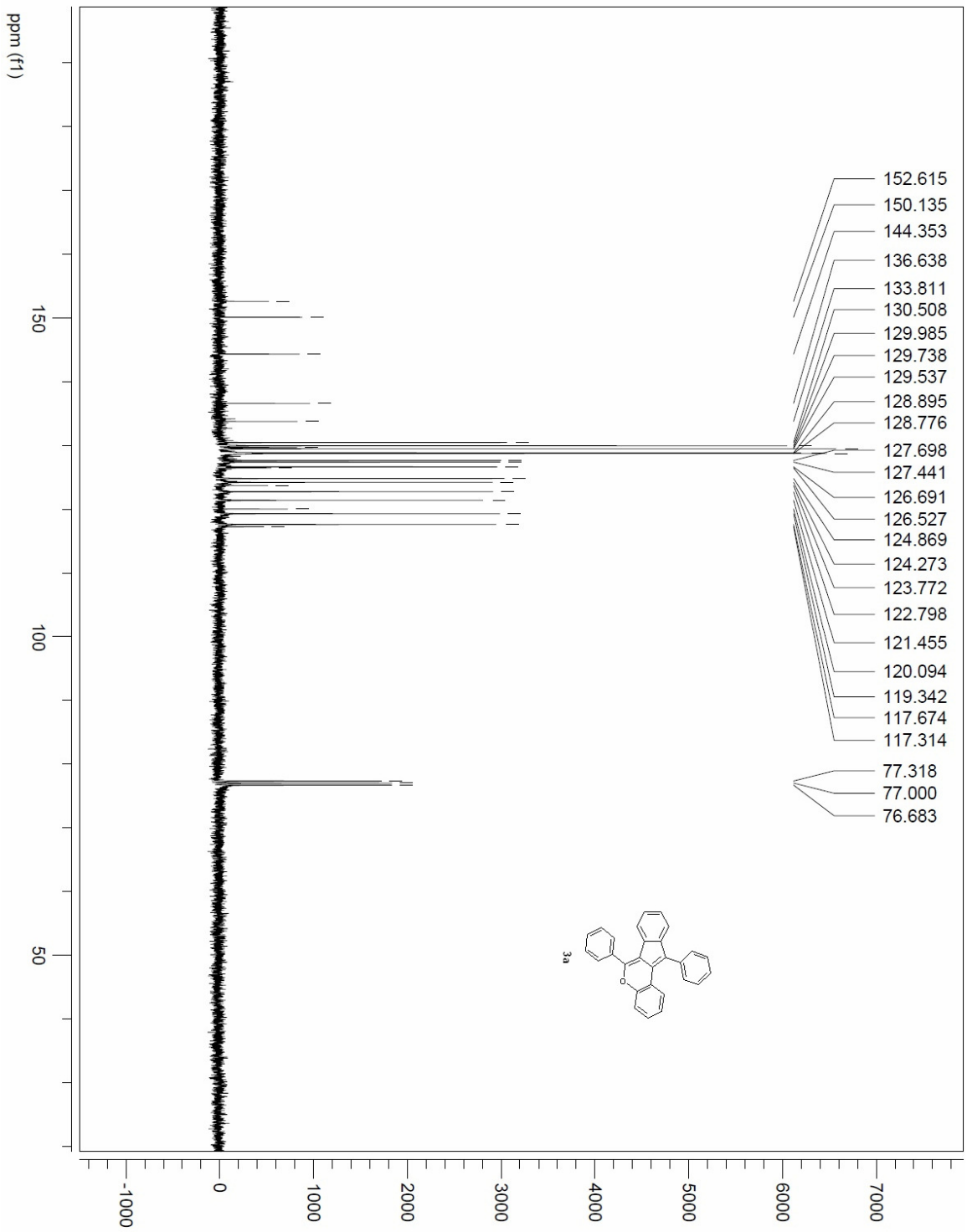


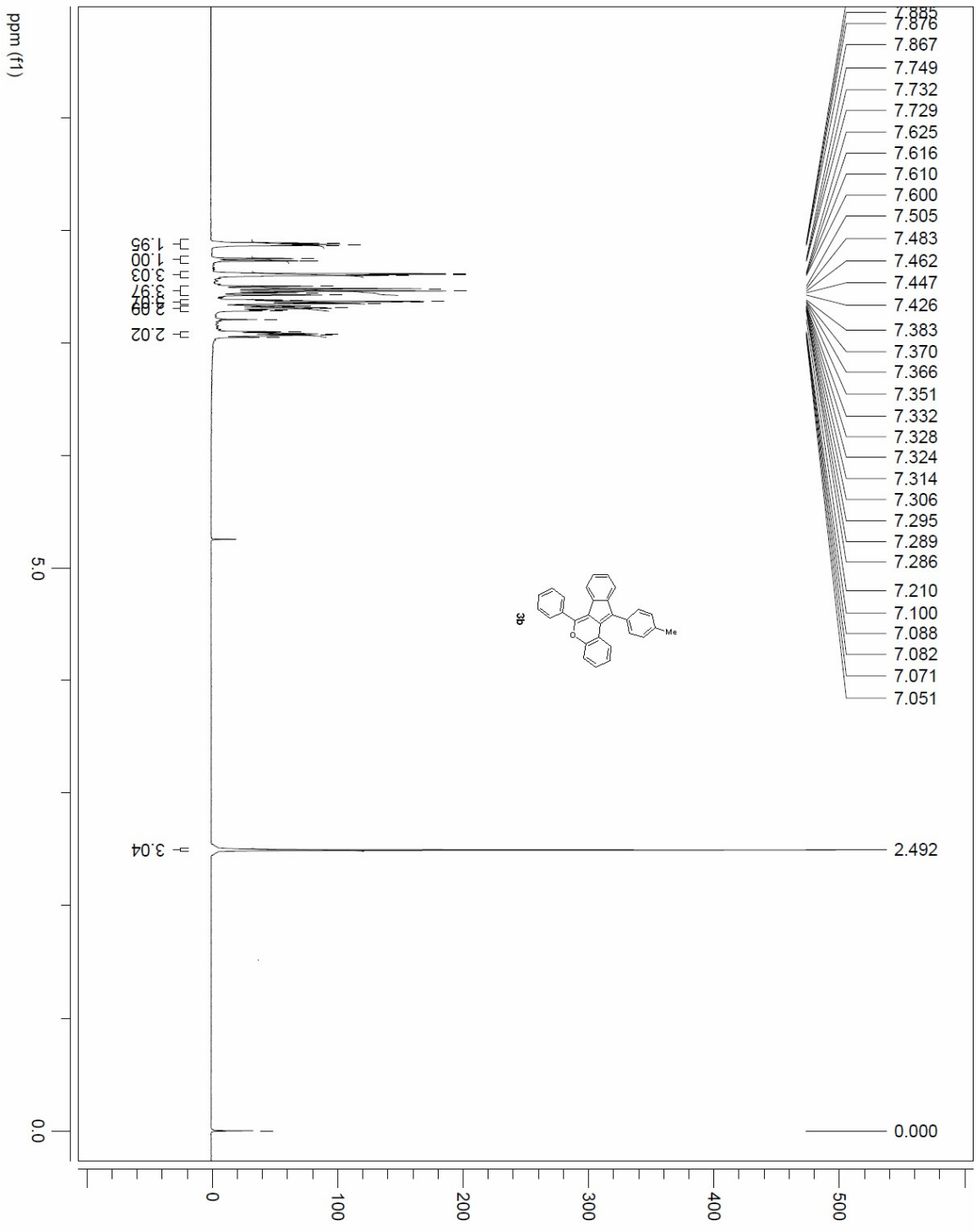
6,11-Bis(4-chlorophenyl)indeno[1,2-*c*]chromene (**3y**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 6.9 Hz, 2H), 7.64-7.60 (m, 3H), 7.55-7.50 (m, 6H), 7.35-7.33 (m, 3H), 7.13-7.10 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.8, 135.0, 133.3, 132.1, 131.4, 131.0, 129.2, 128.0, 127.0, 125.4, 124.8, 124.5, 123.1, 121.4, 119.8, 119.2, 117.8. HRMS (ESI) calcd for $C_{28}H_{17}Cl_2O$:439.0656 ($M + H^+$), found: 439.0648.

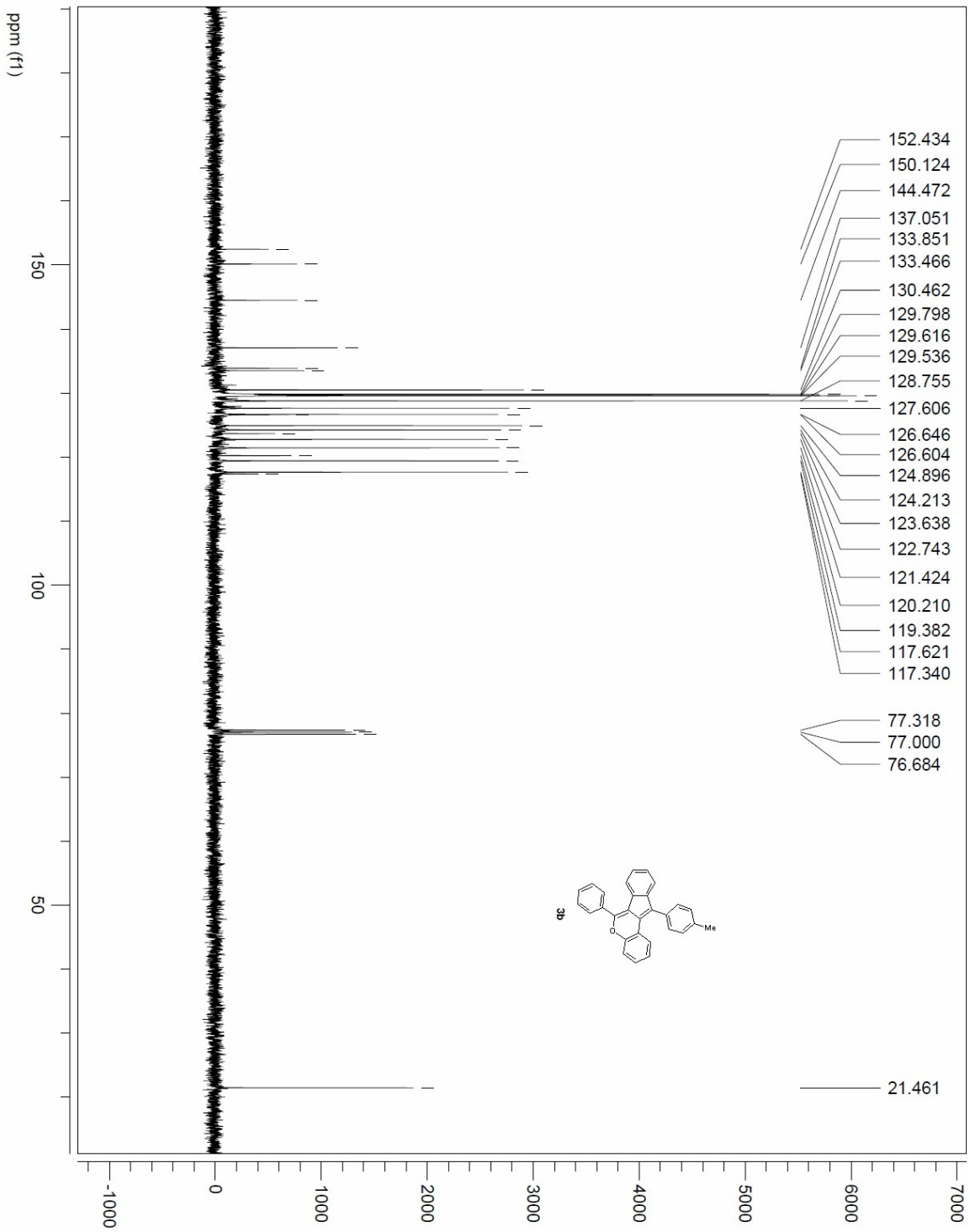


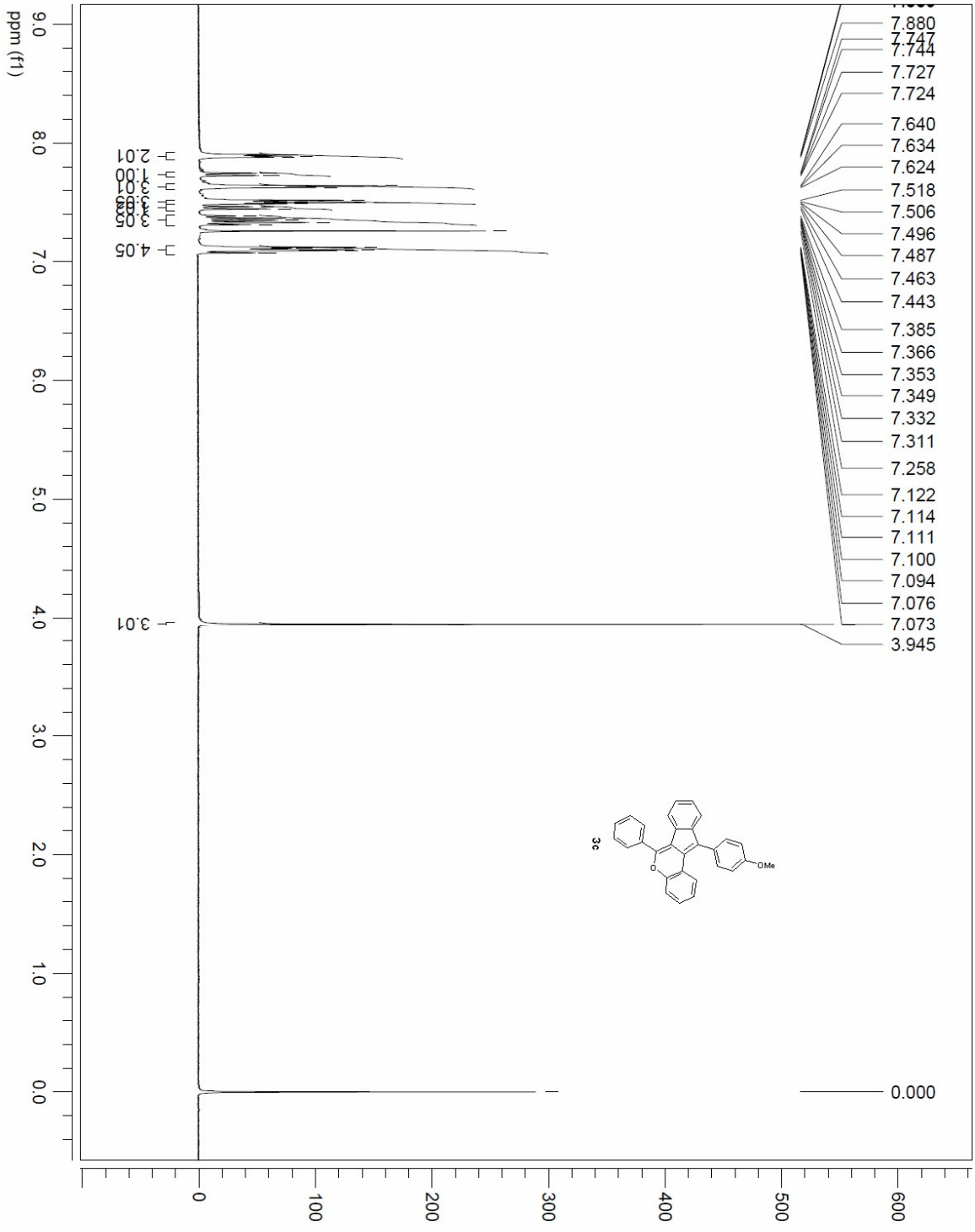
1,1'-(Indeno[1,2-*c*]chromene-6,11-diylbis(4,1-phenylene))diethanone (**3z**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21-8.15 (m, 4H), 8.02-8.00 (m, 2H), 7.71-7.69 (m, 3H), 7.51-7.49 (m, 2H), 7.35 (m, 3H), 7.12 (m, 2H), 2.74 (s, 3H), 2.72 (s, 3H) . ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.9, 197.4, 151.5, 150.0, 143.9, 141.9, 138.5, 137.9, 136.2, 130.3, 129.9, 129.4, 129.0, 128.7, 128.2, 127.2, 125.7, 124.8, 124.6, 124.3, 123.3, 121.5, 119.6, 119.2, 117.8, 26.8, 26.7. HRMS (ESI) calcd for $C_{32}H_{23}O_3$:455.1647 ($M + H^+$), found: 455.1662.

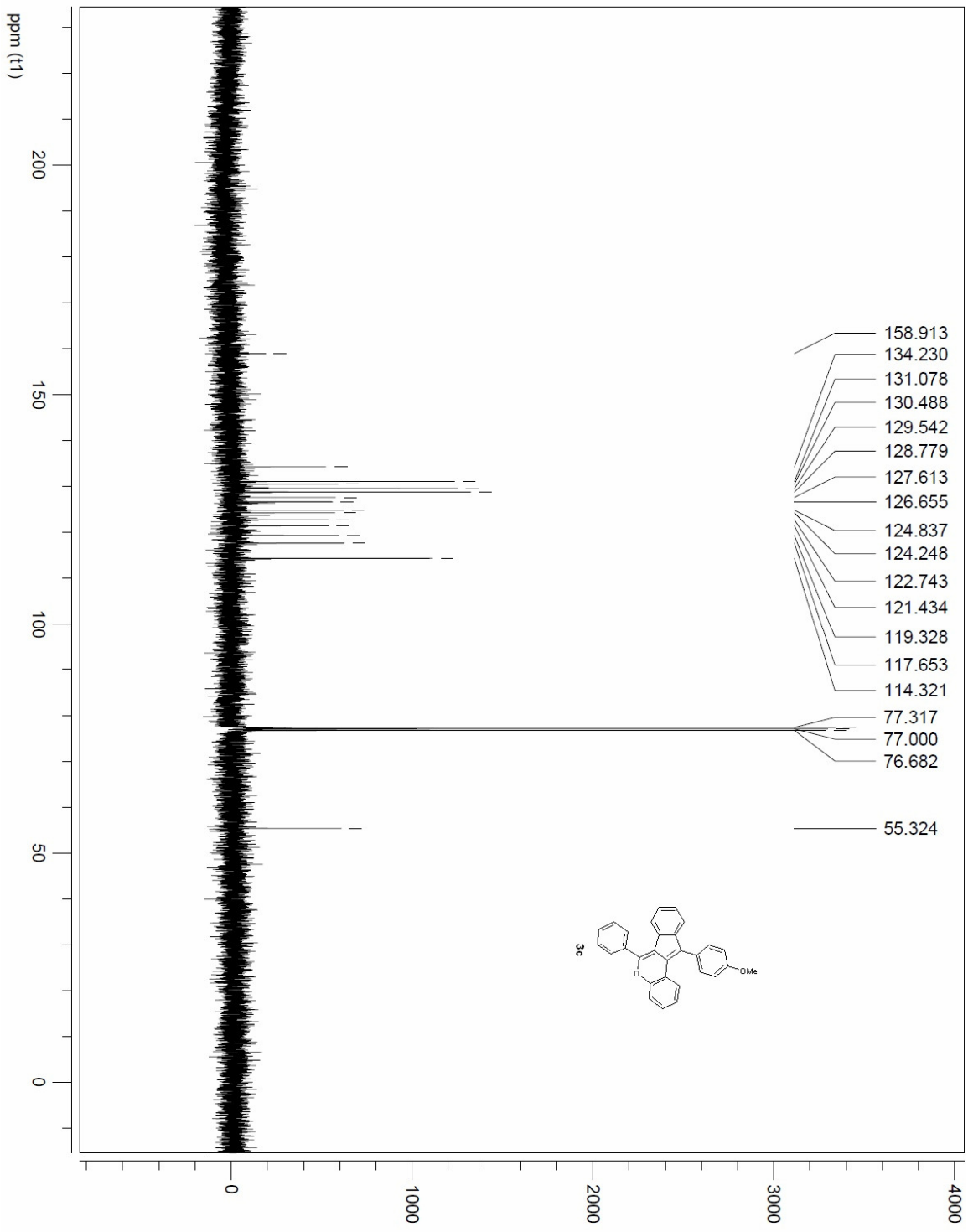


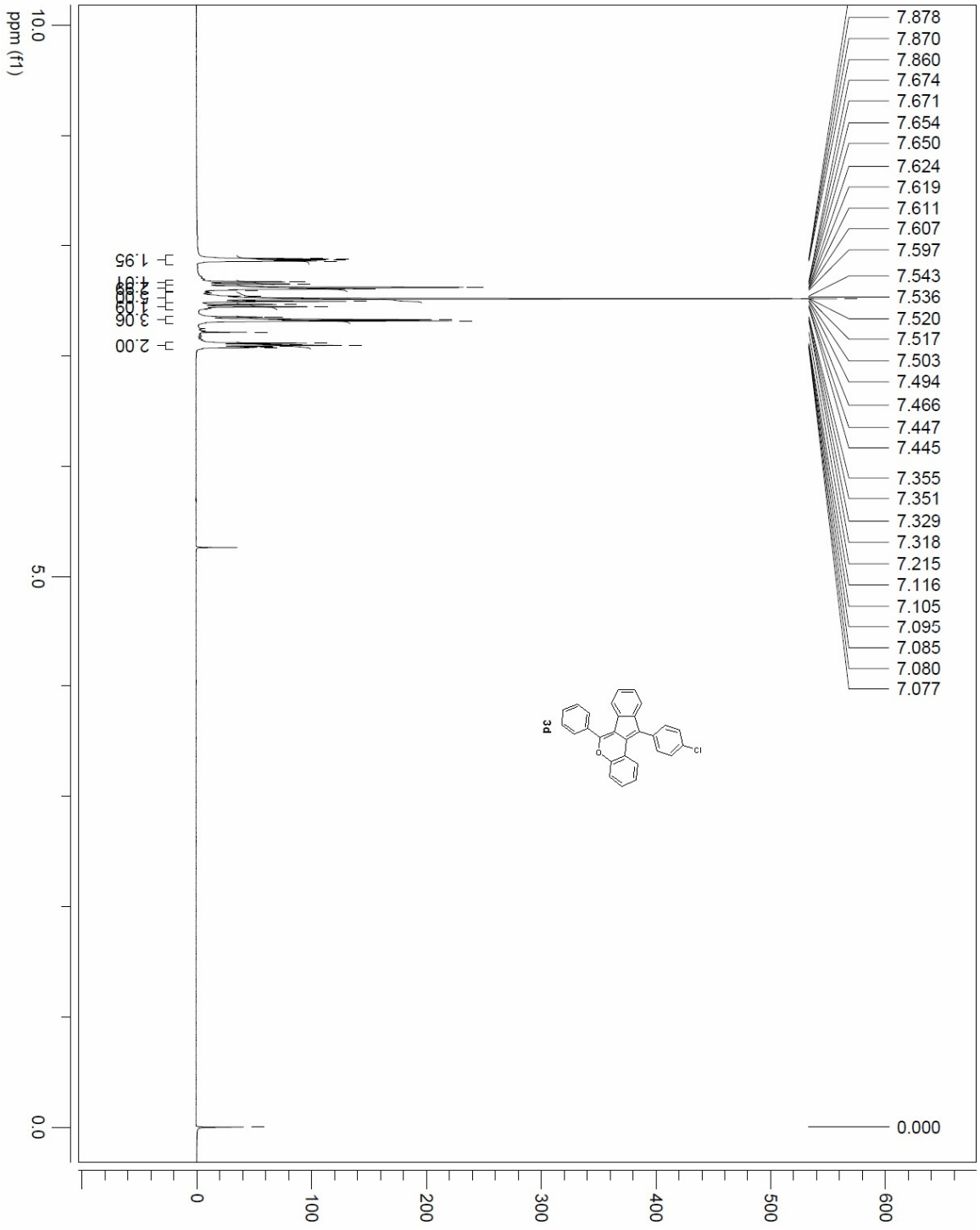


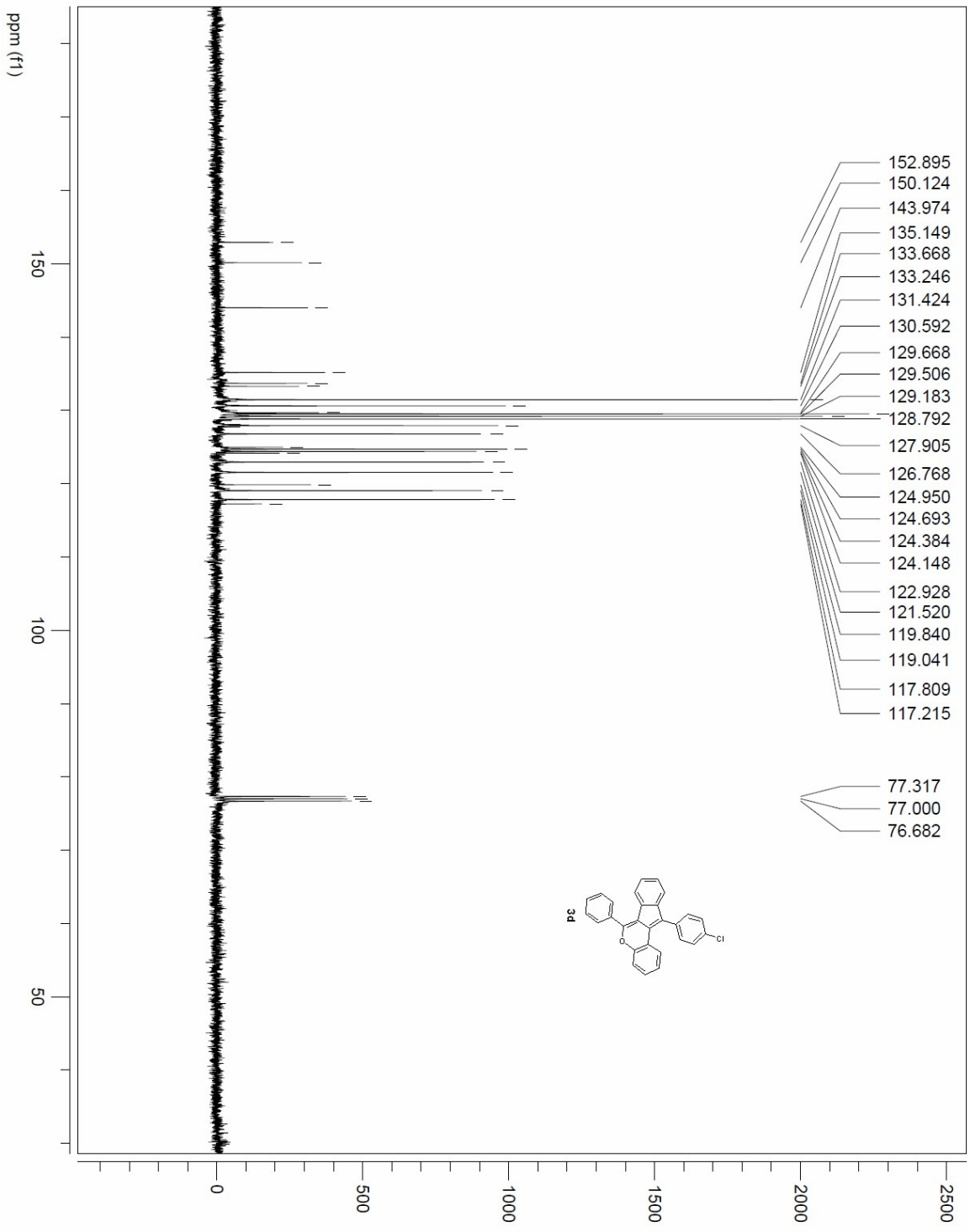


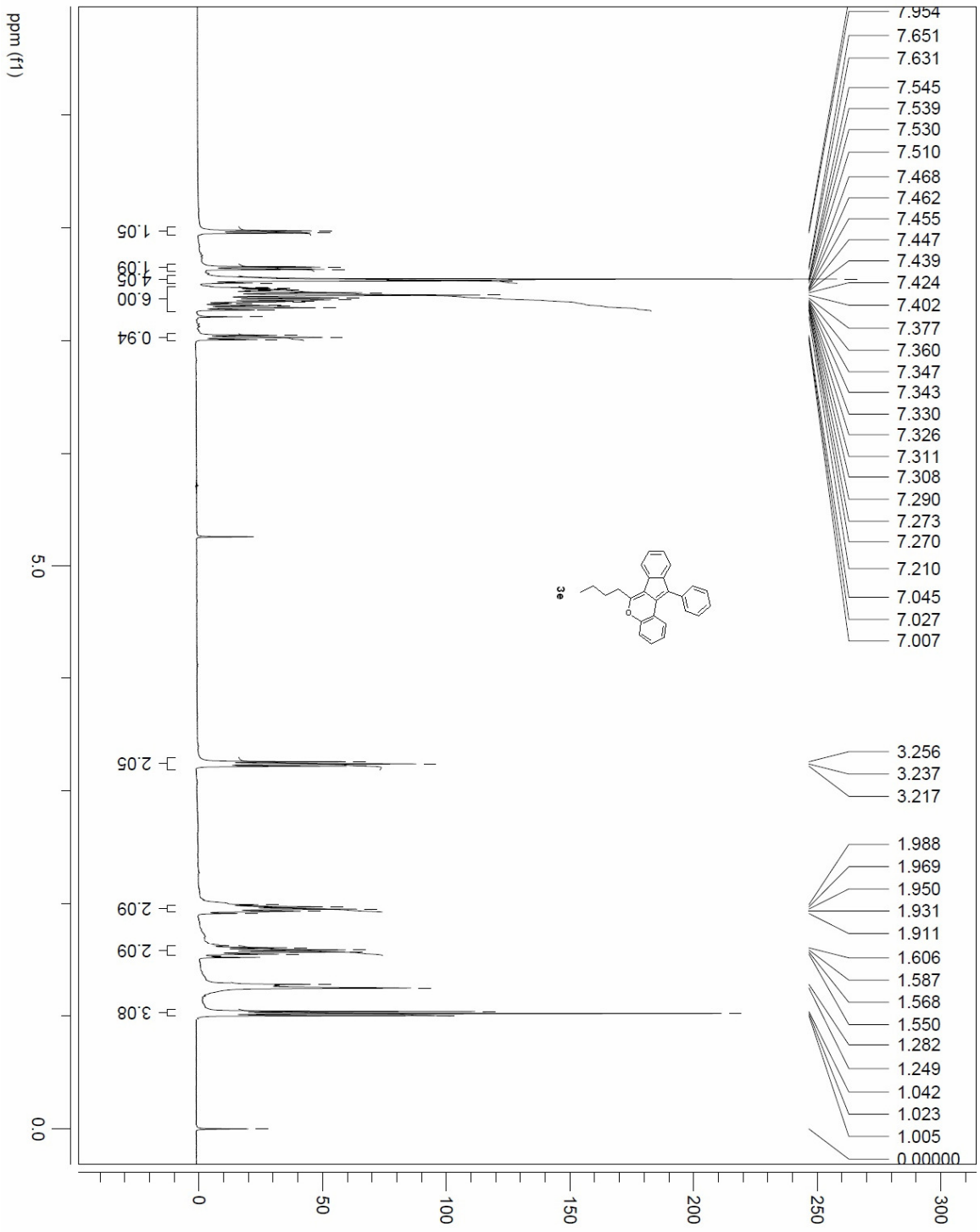


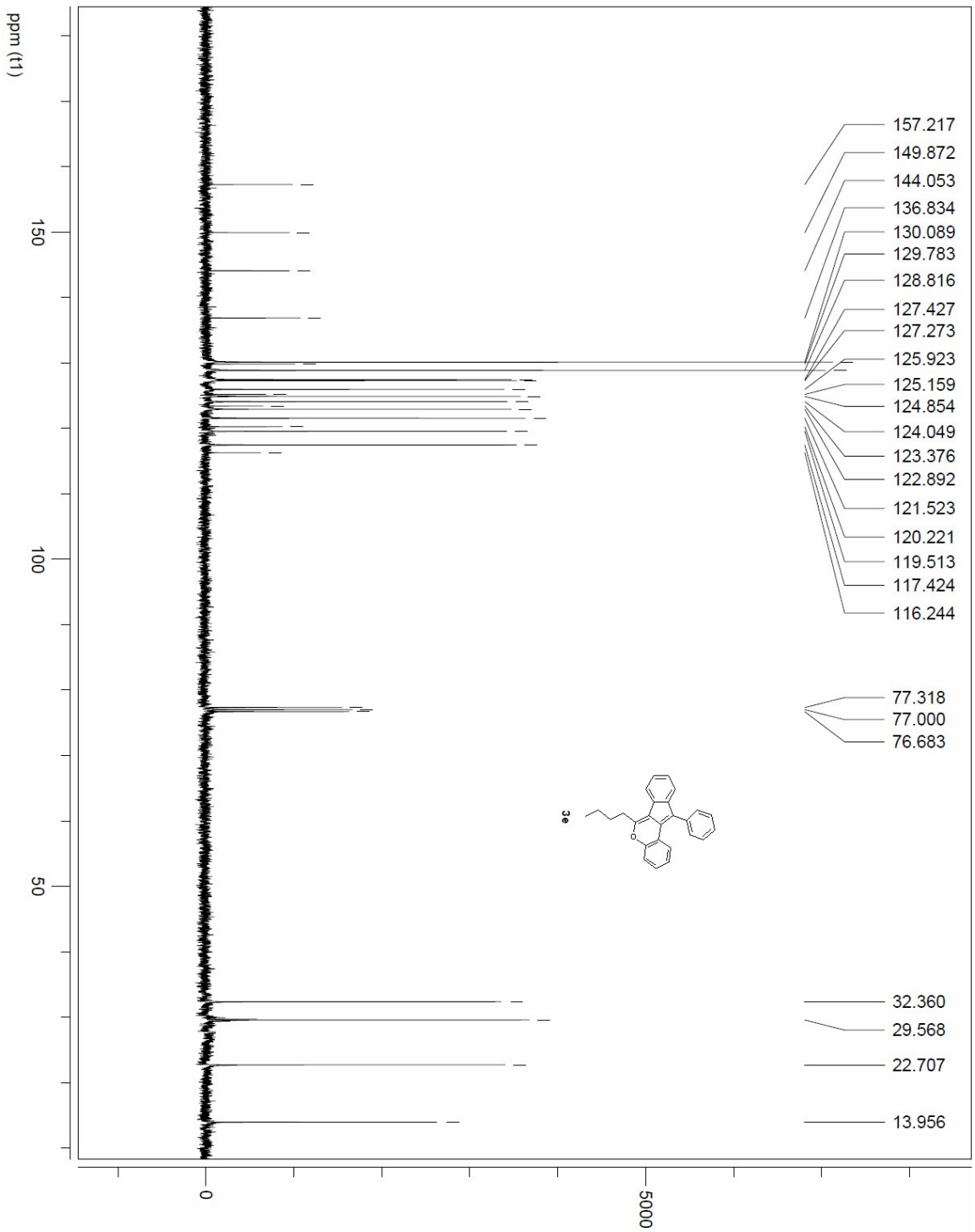


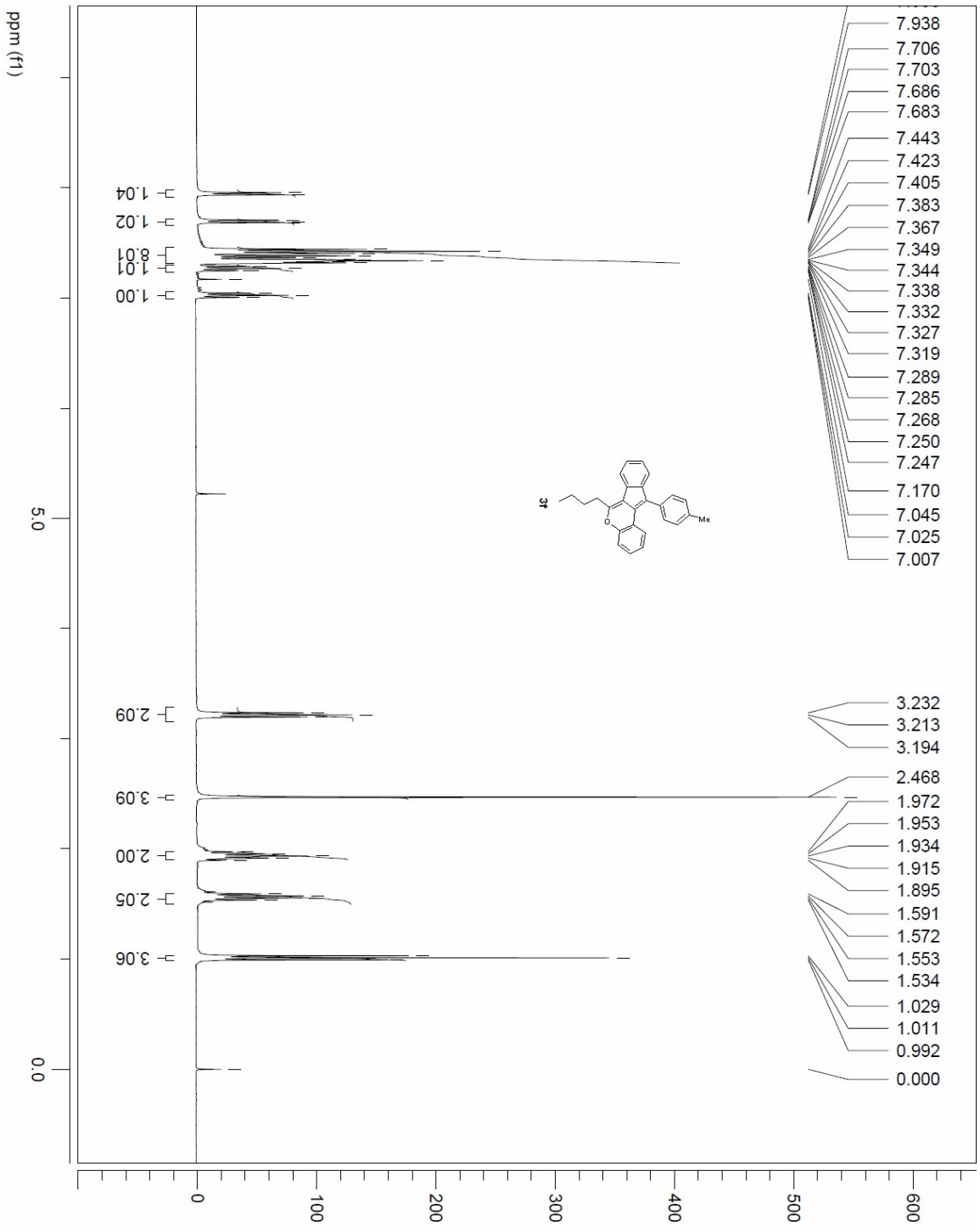


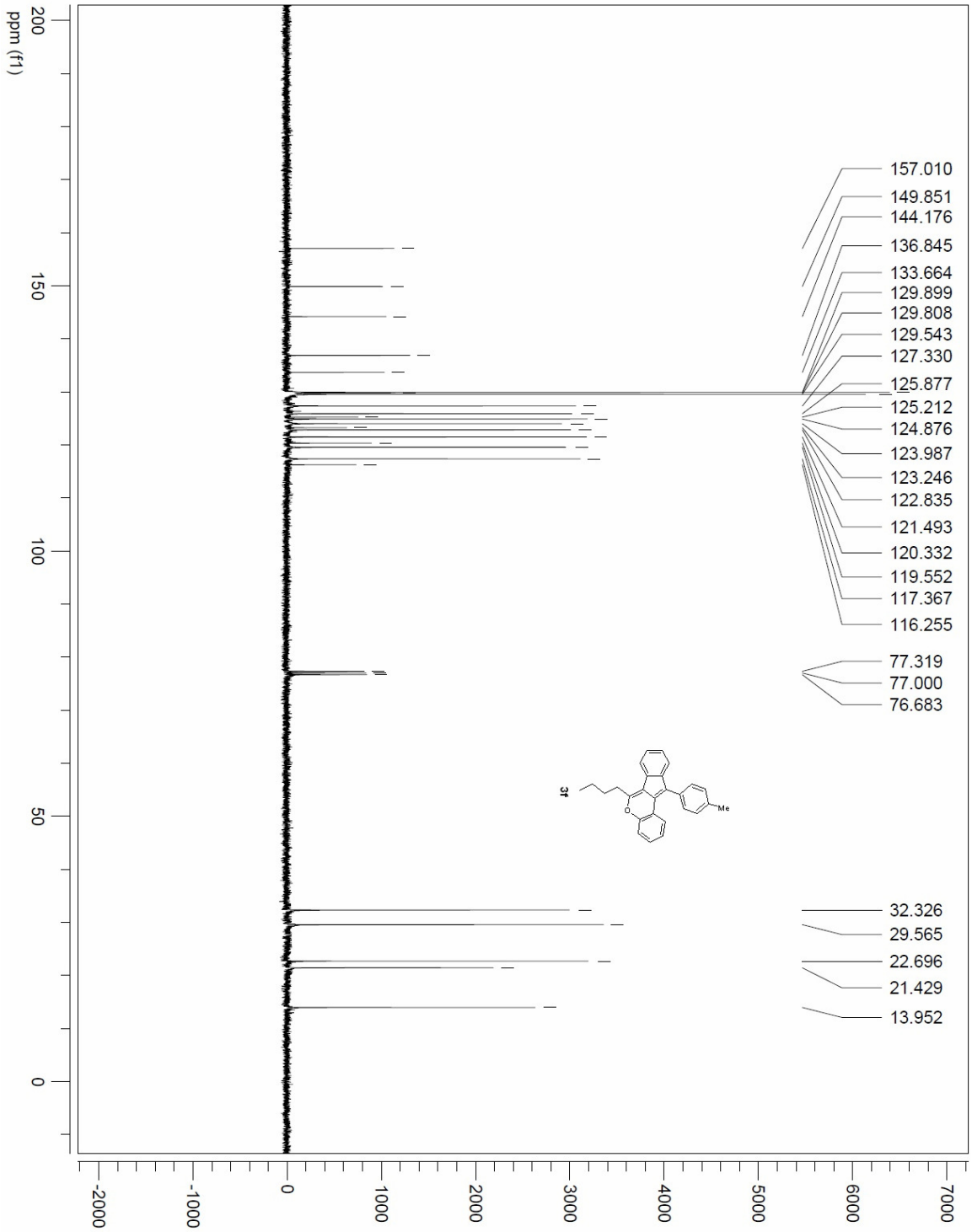


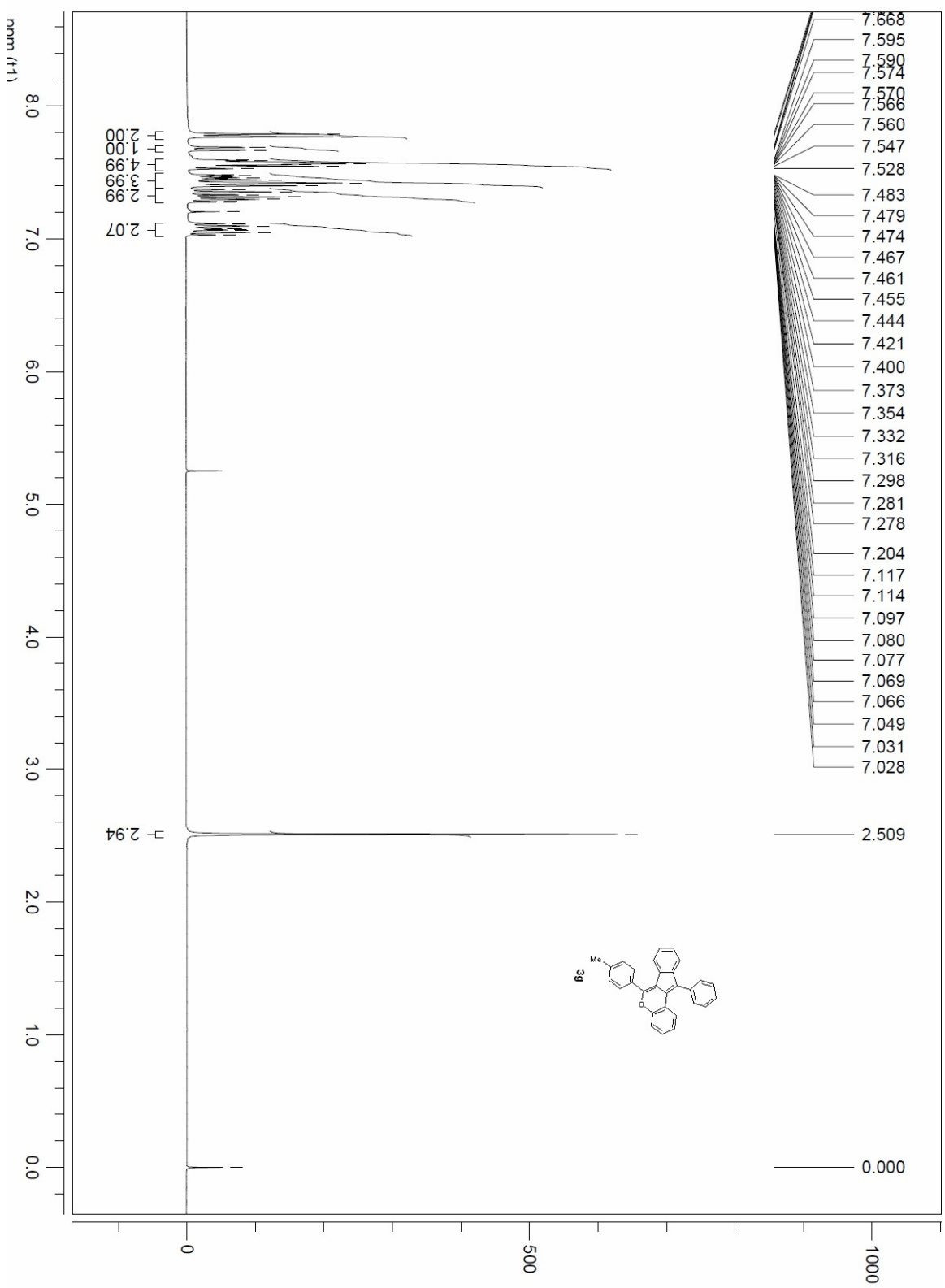


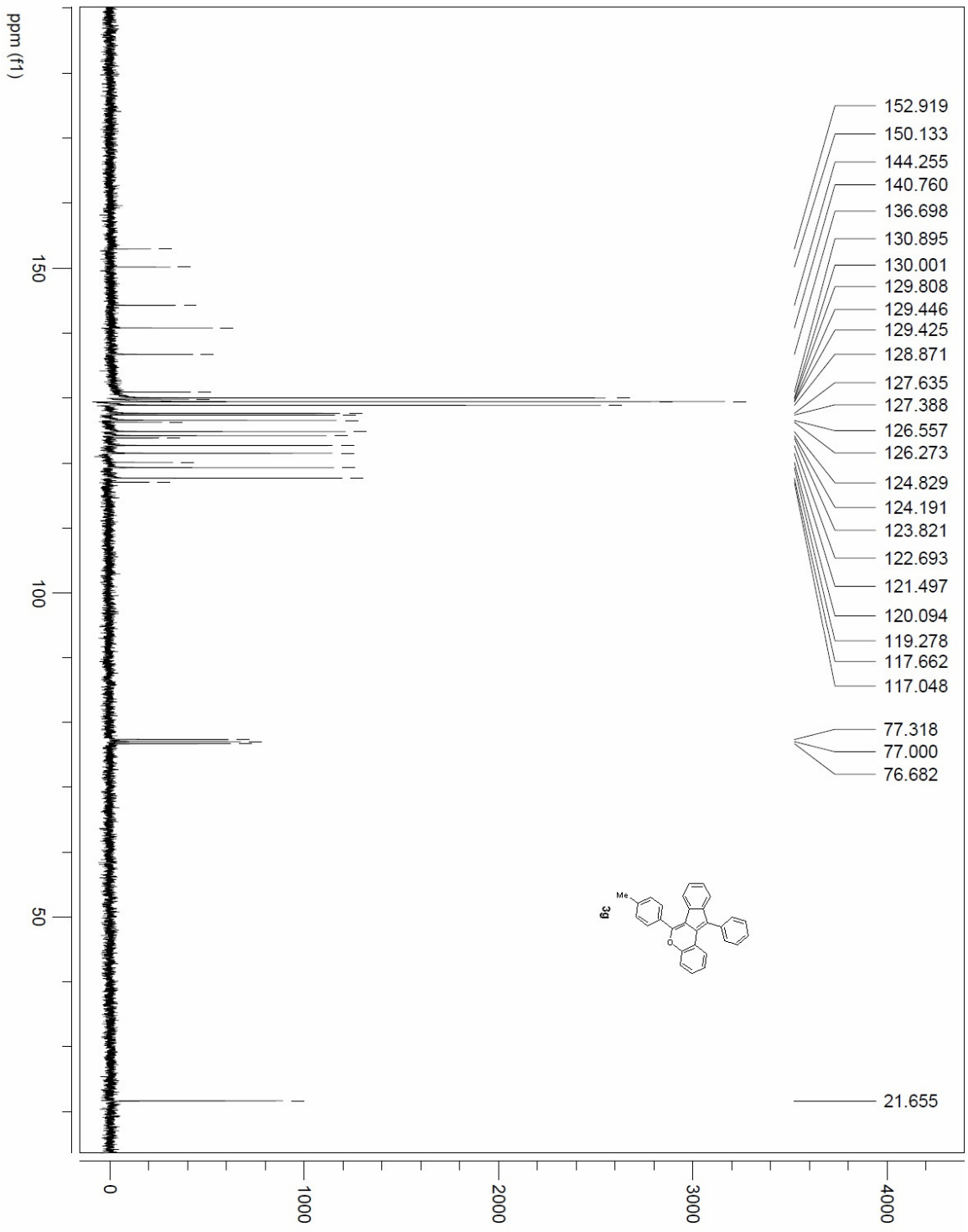


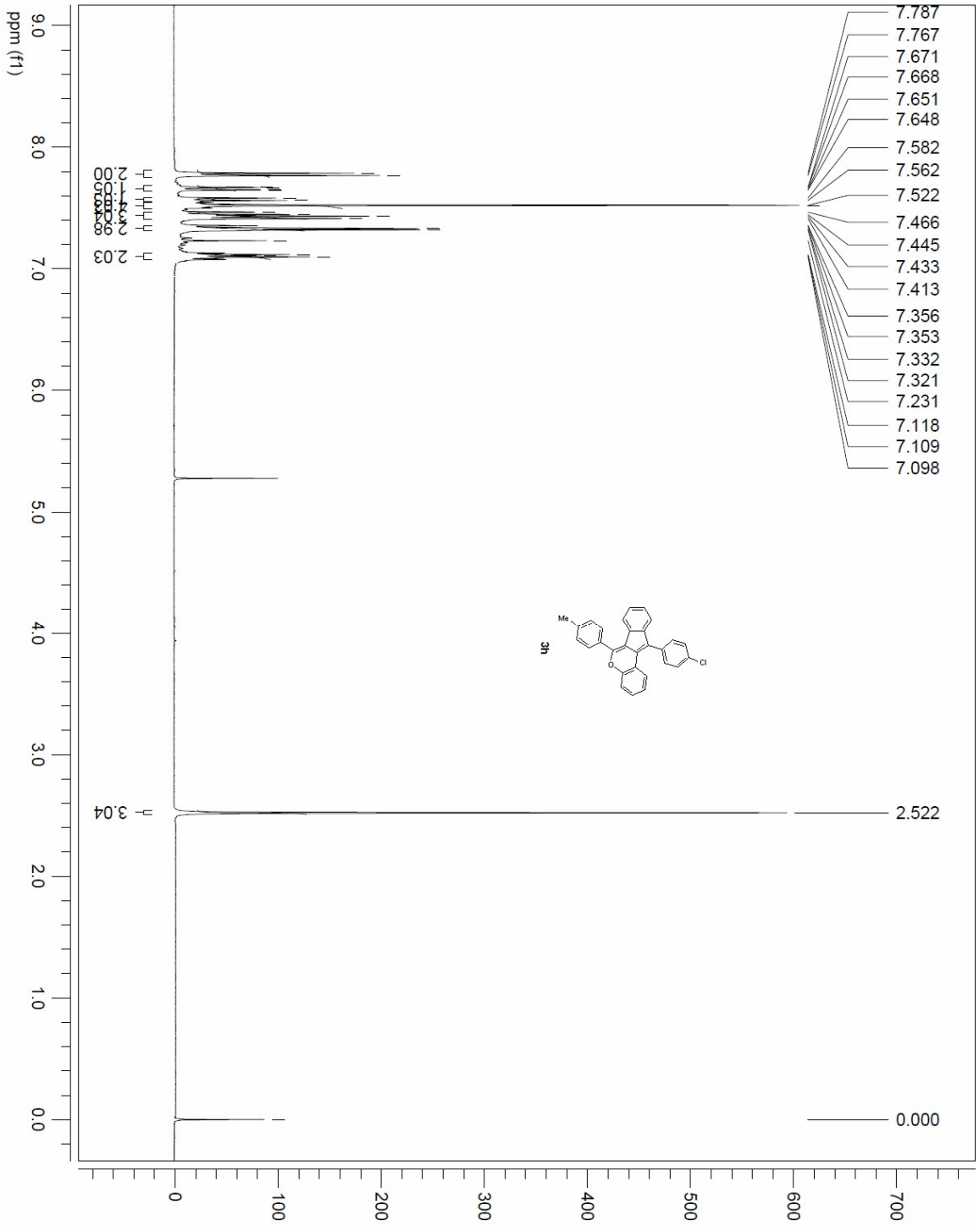


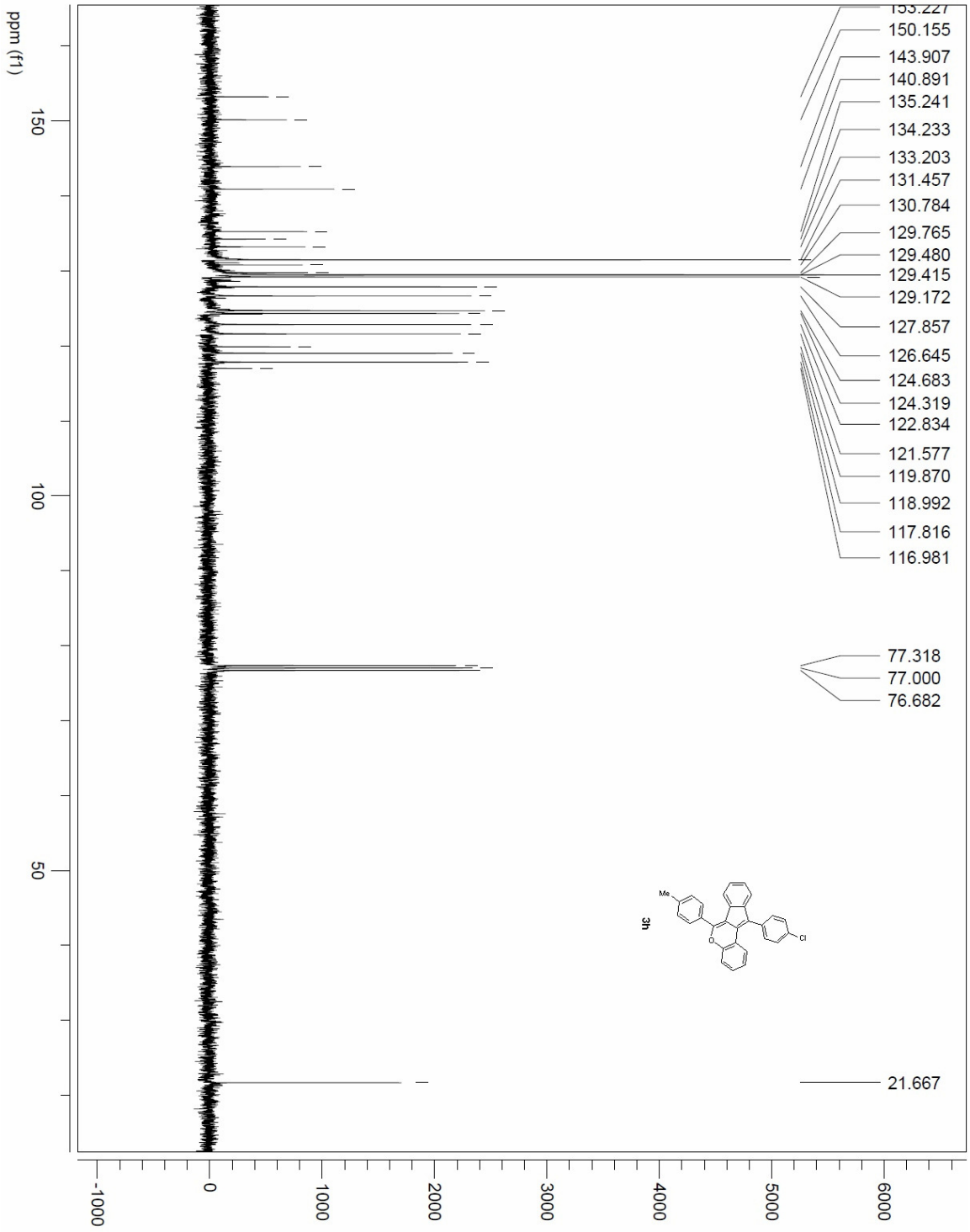


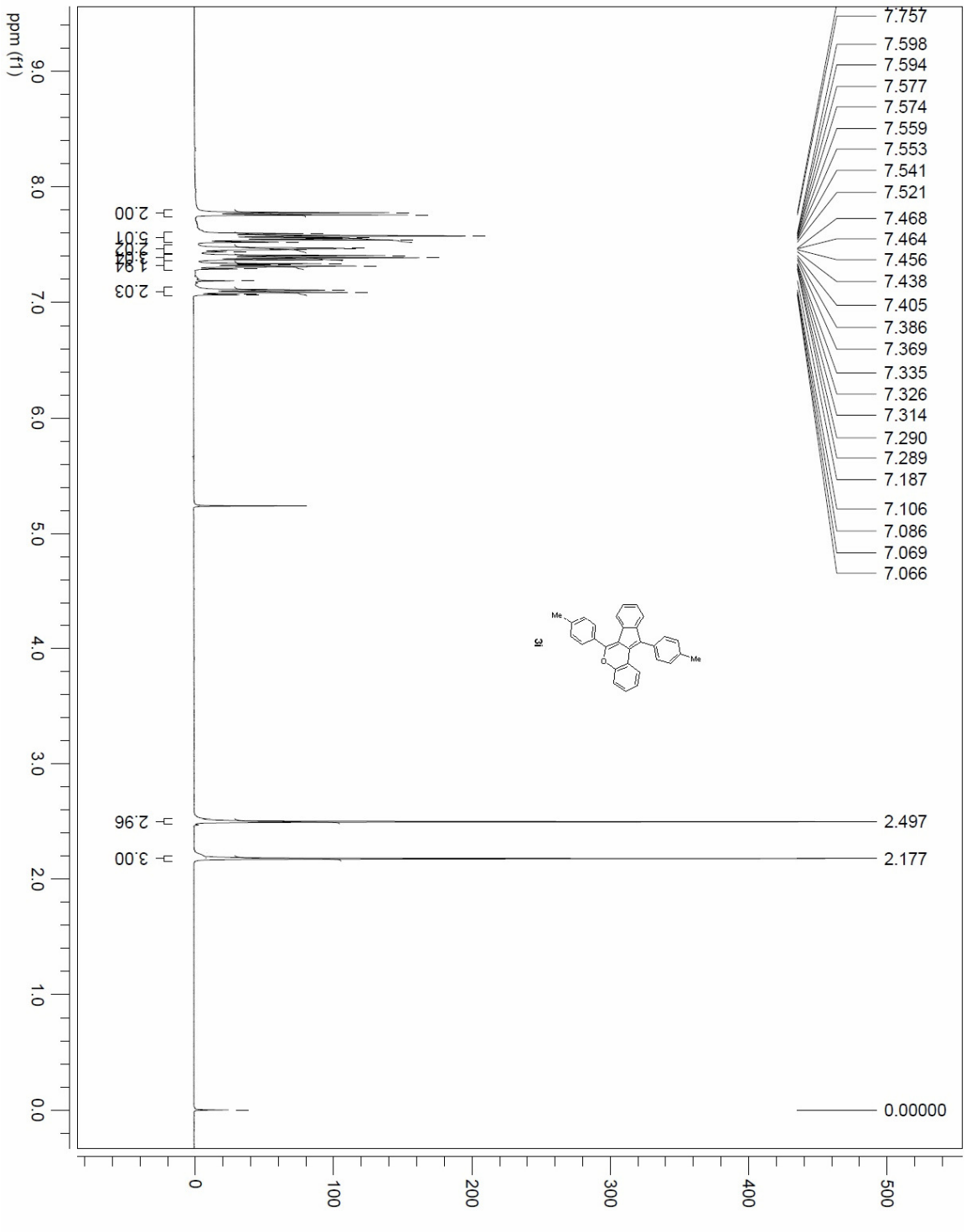


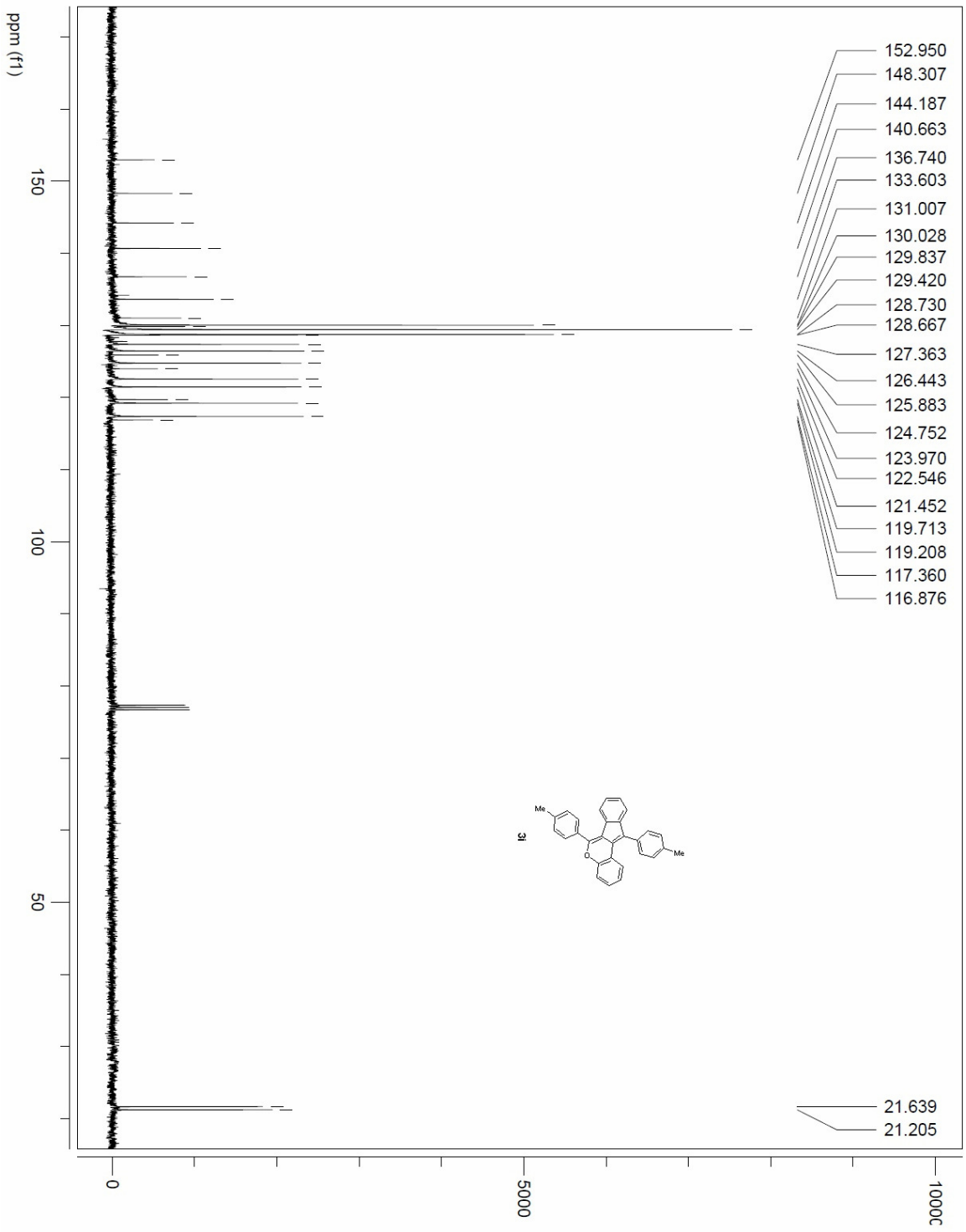


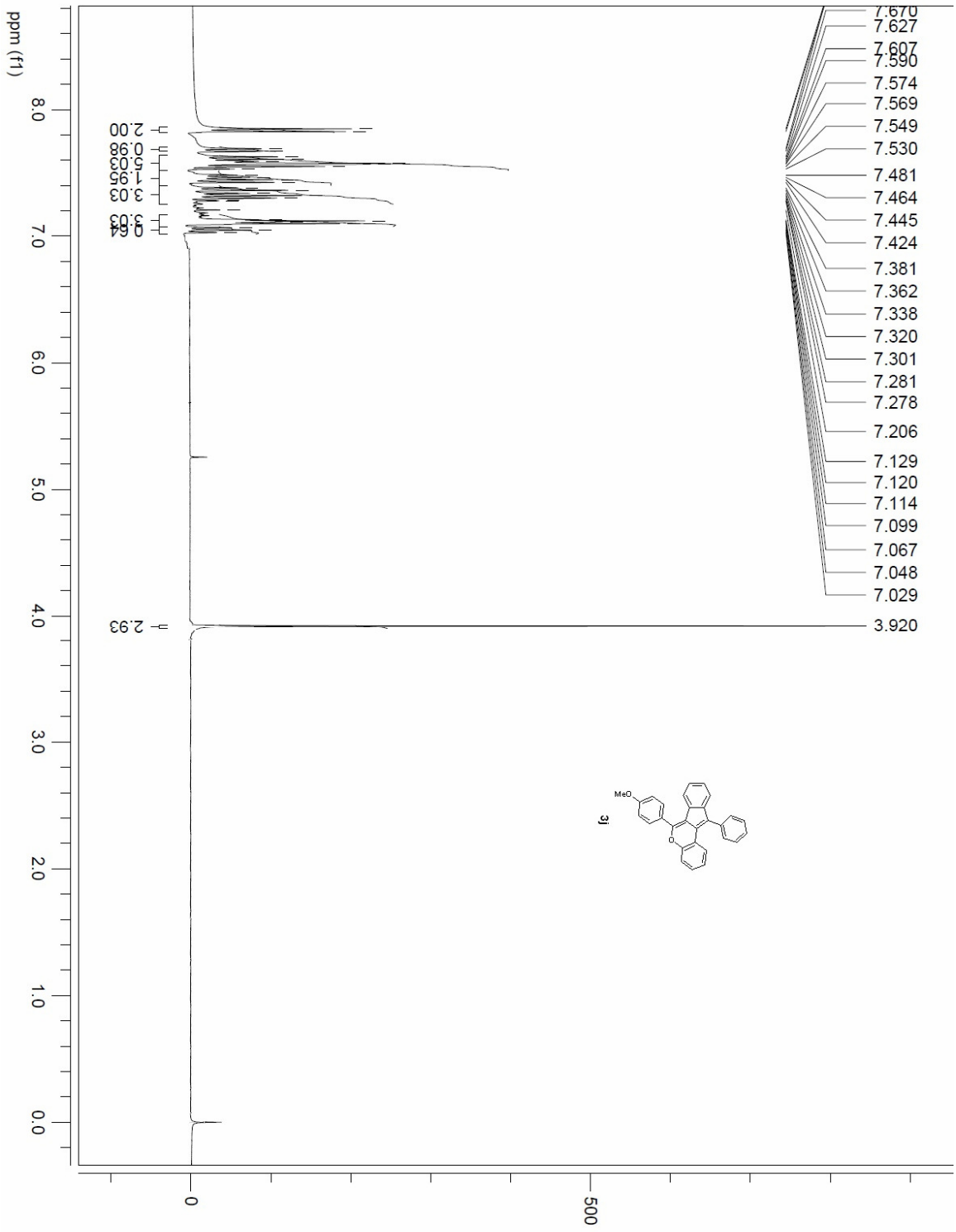


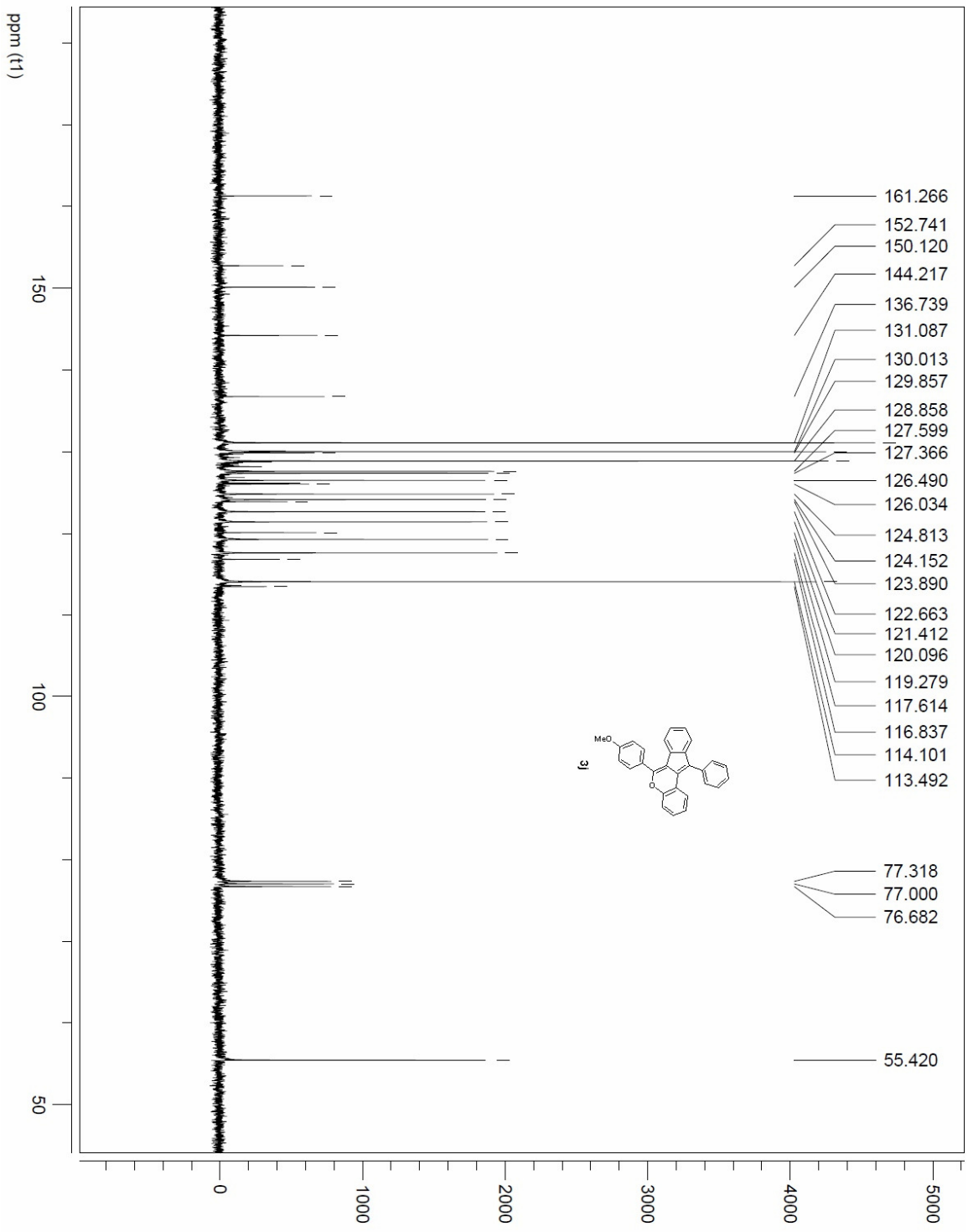


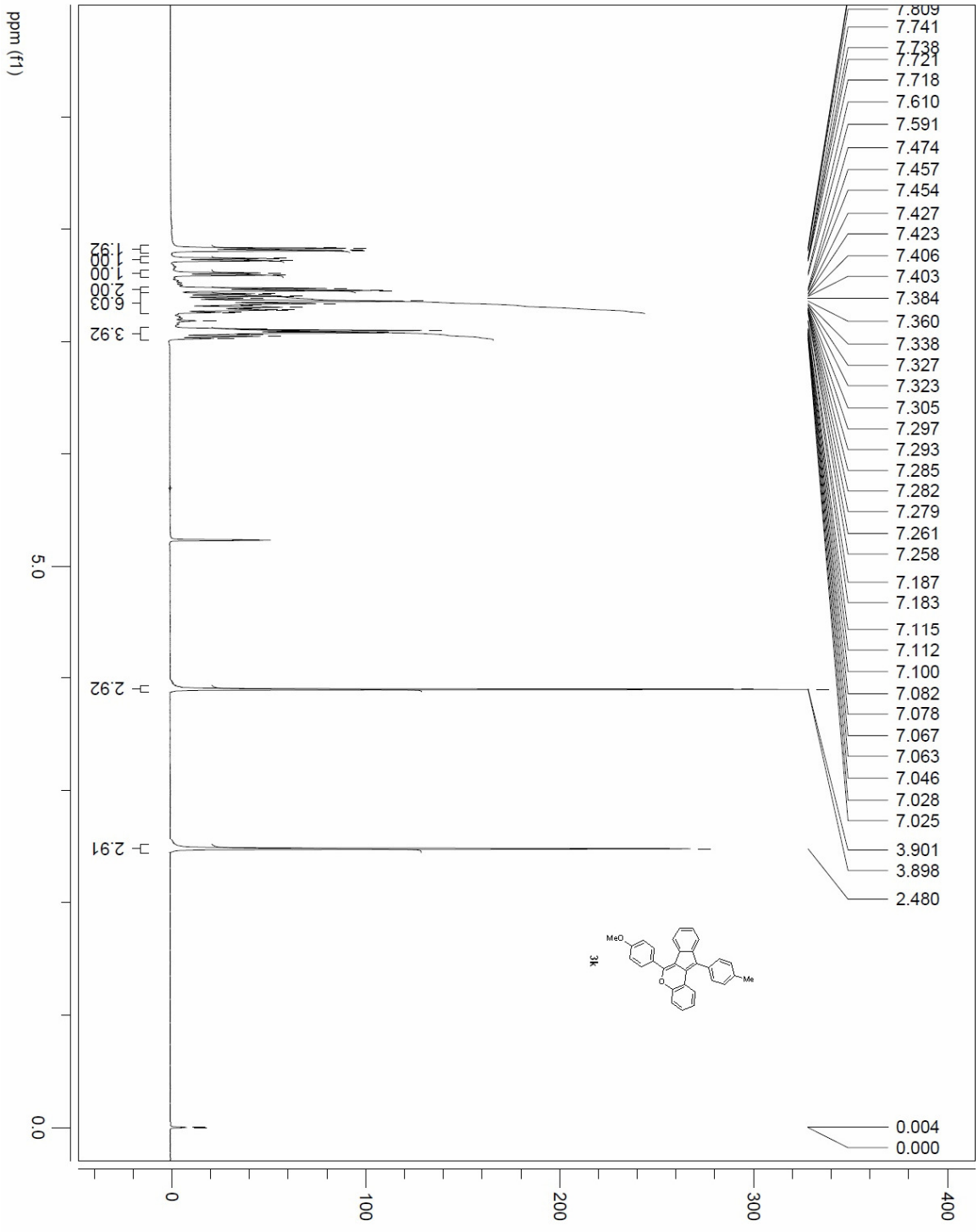


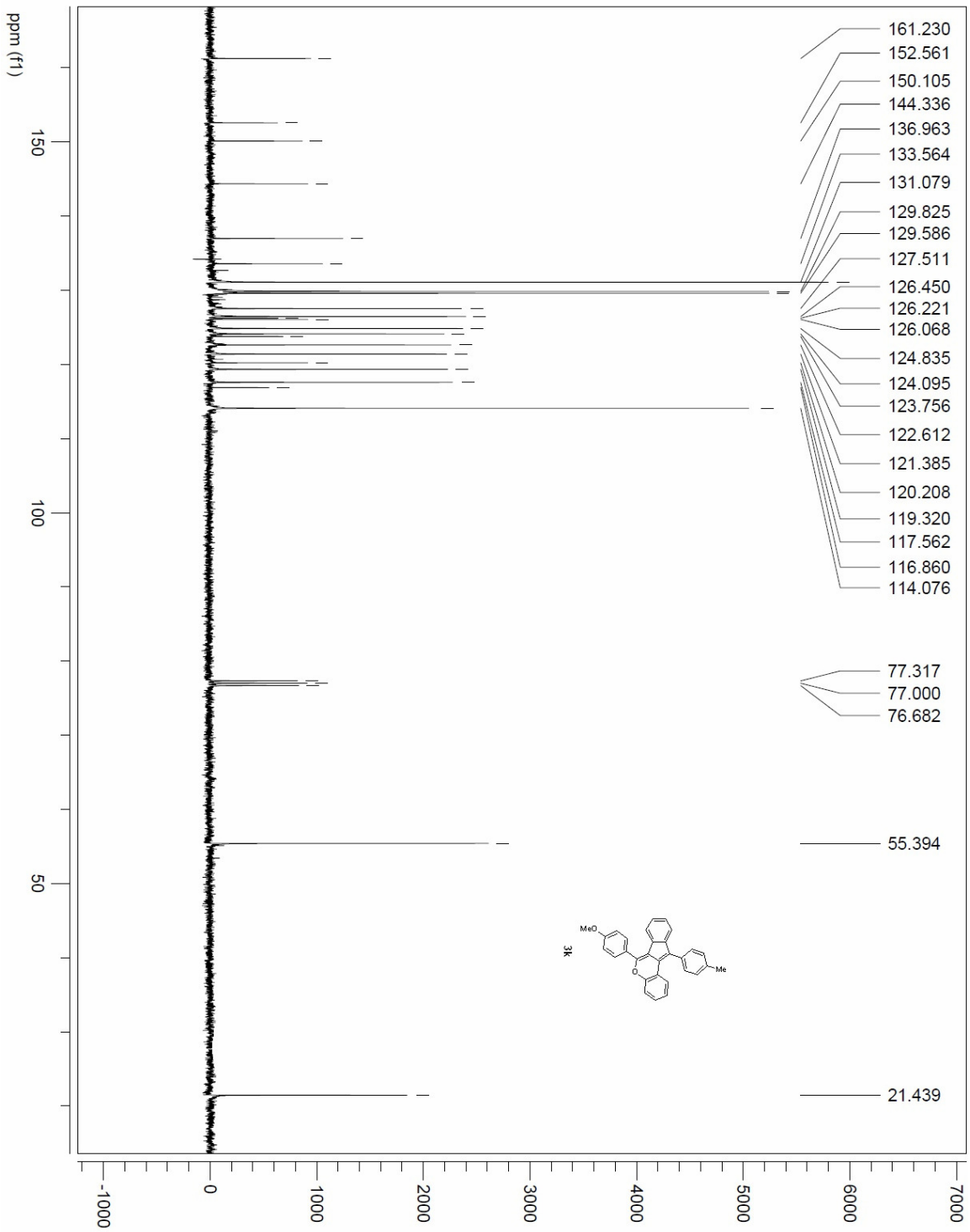


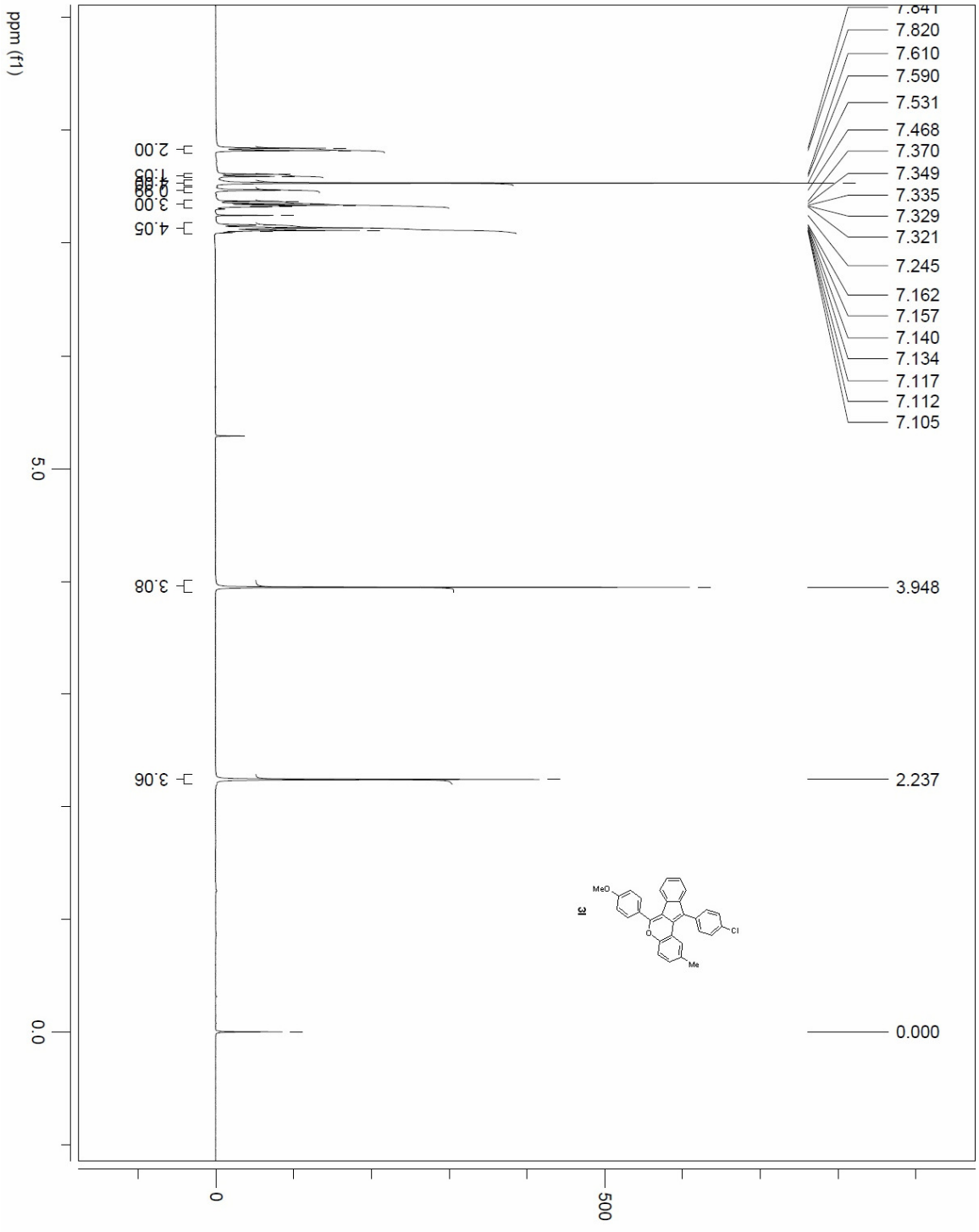


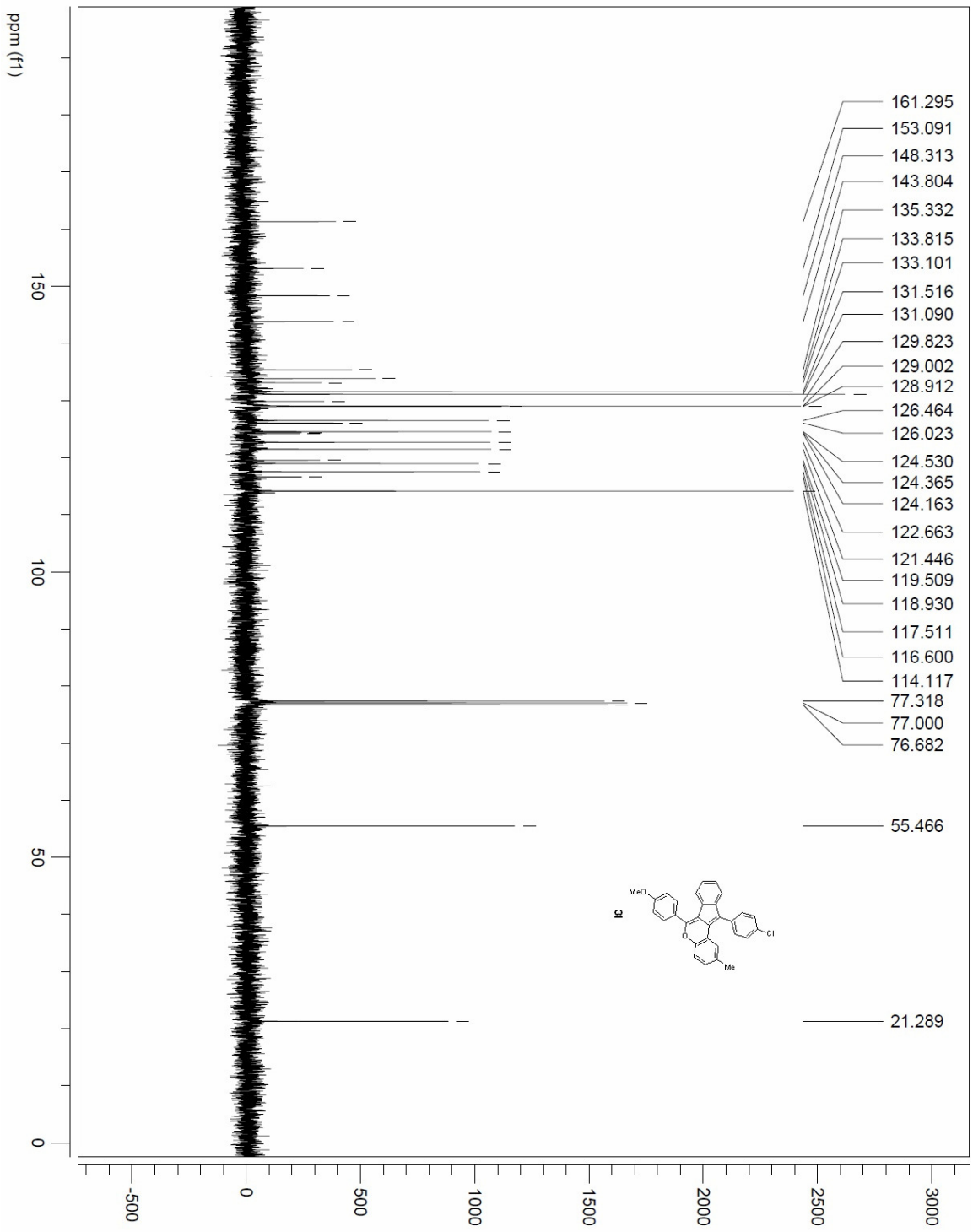


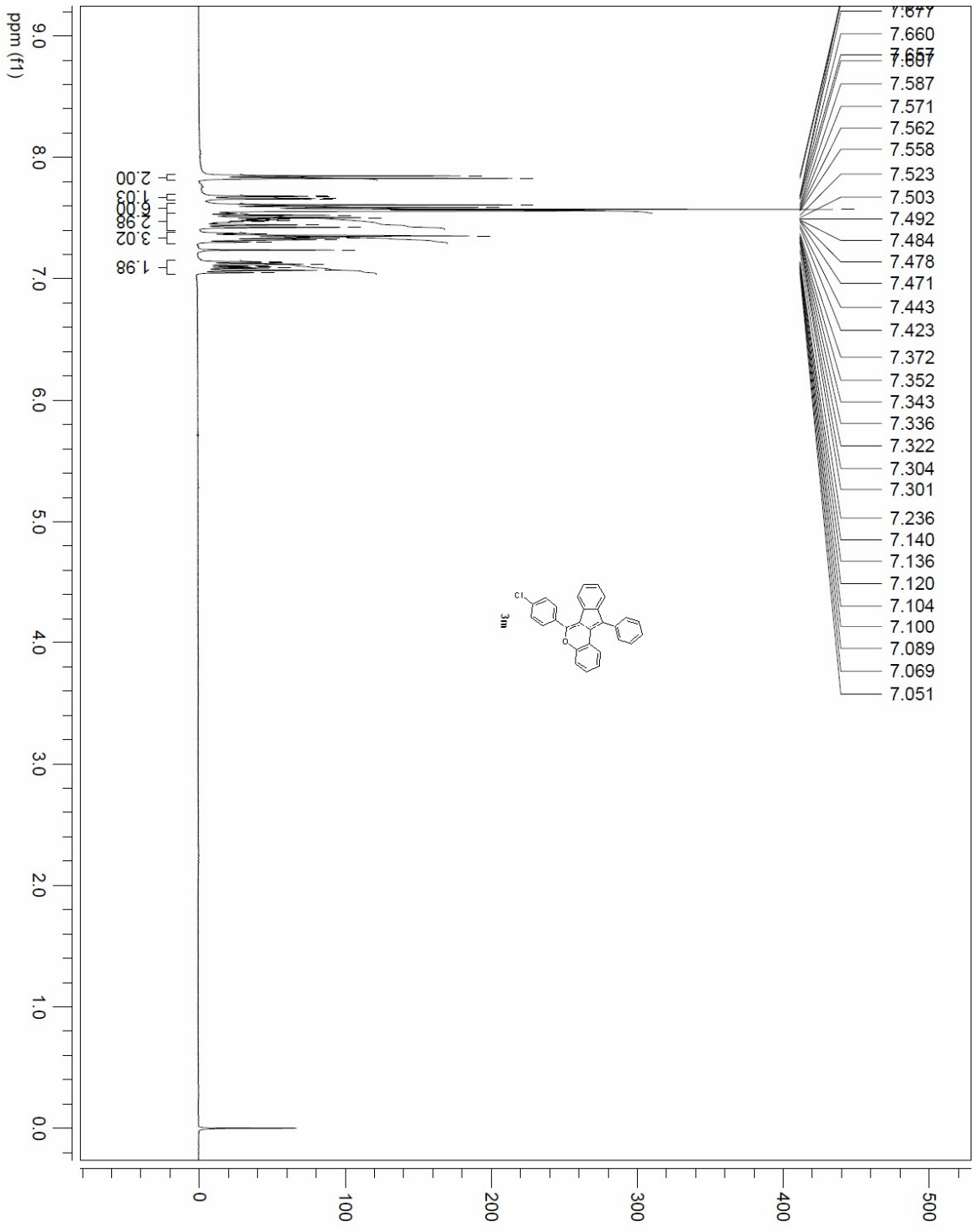


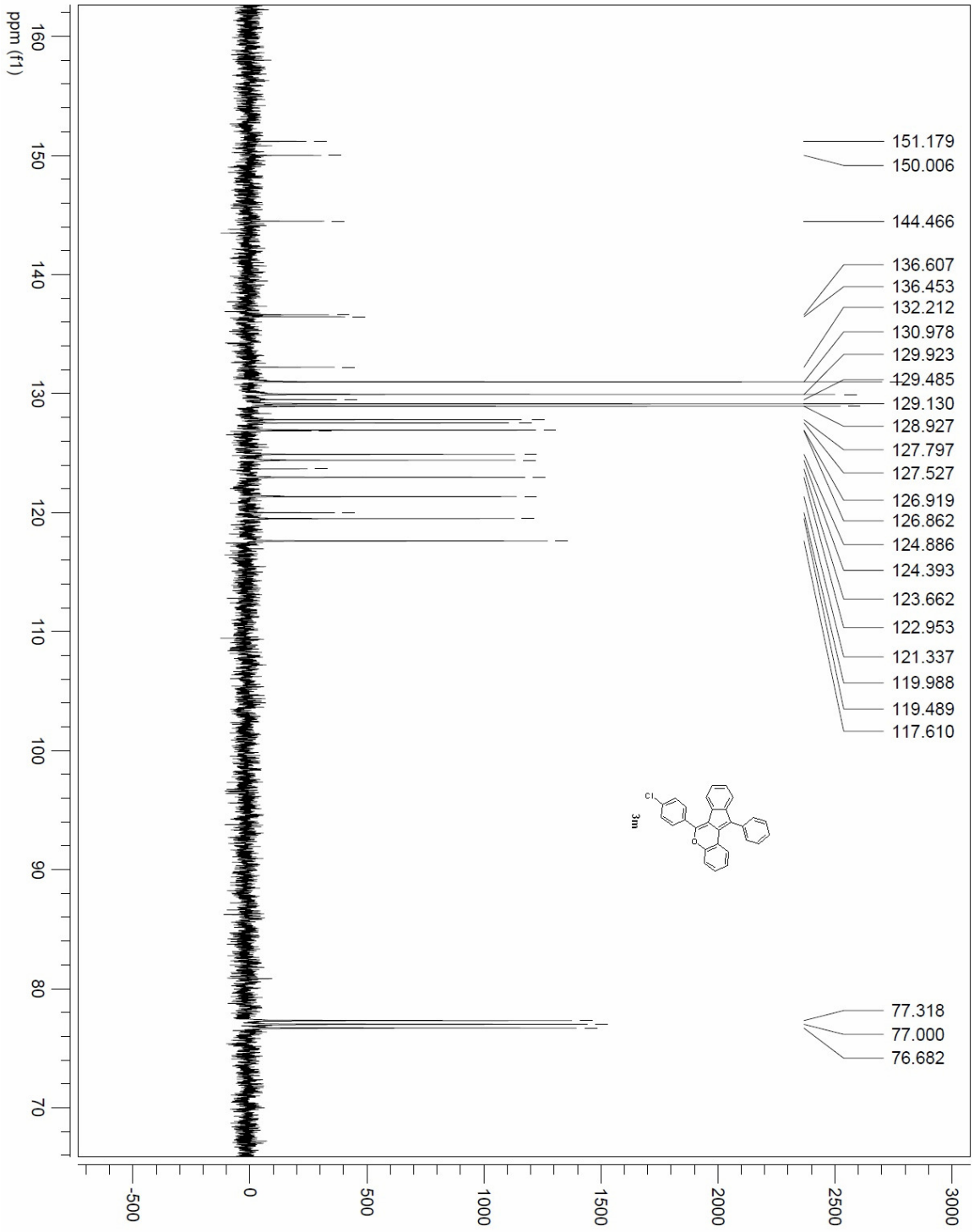


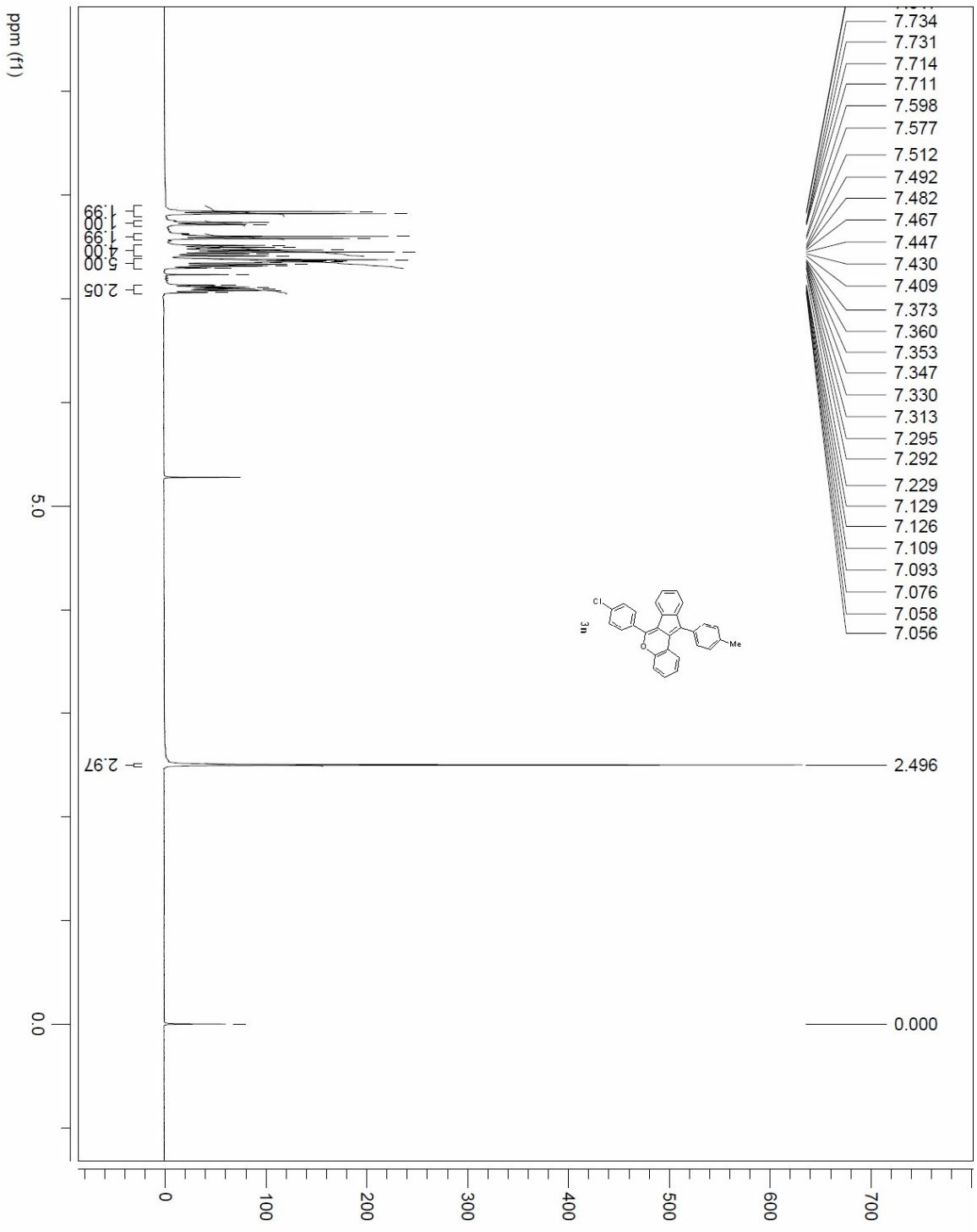


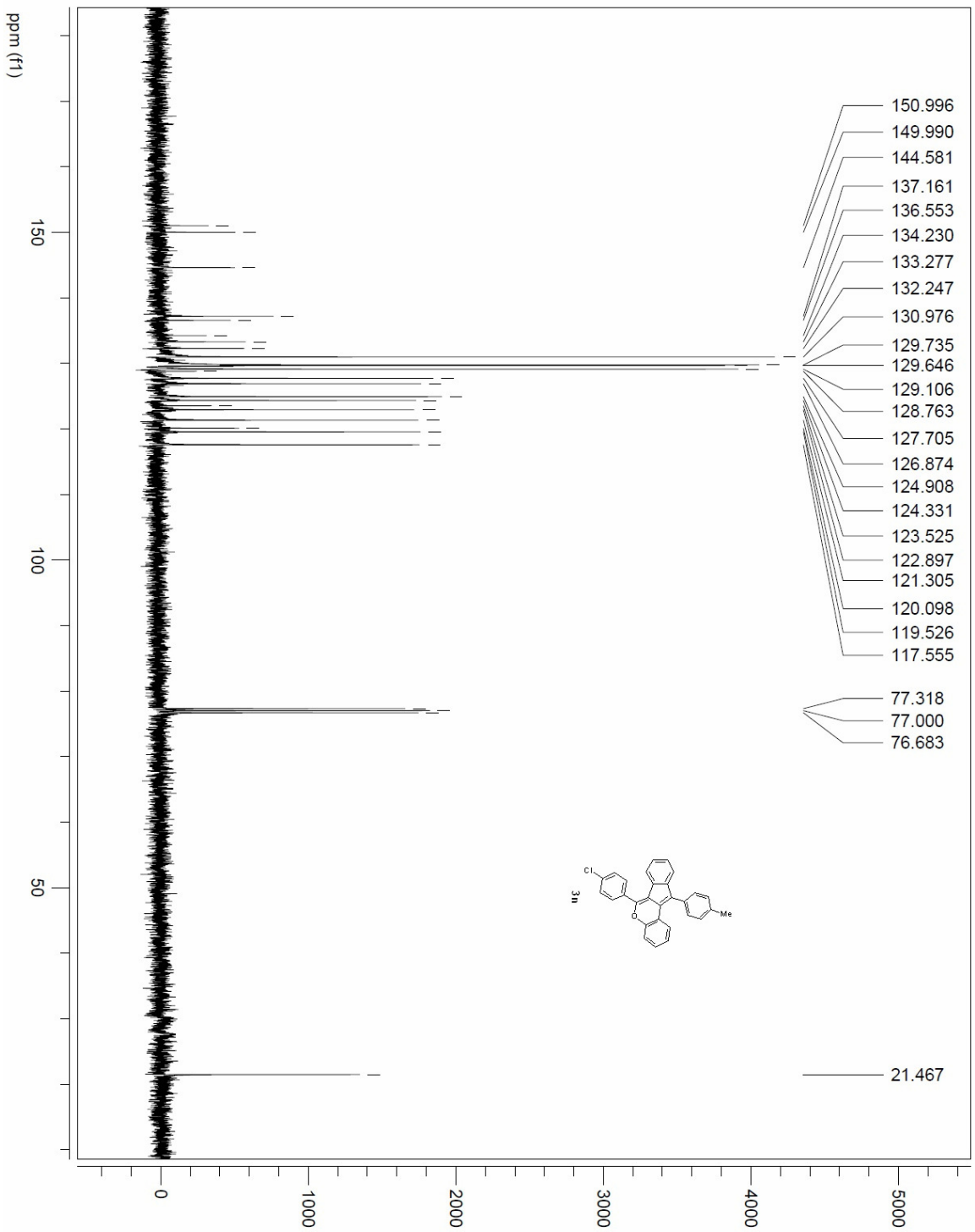


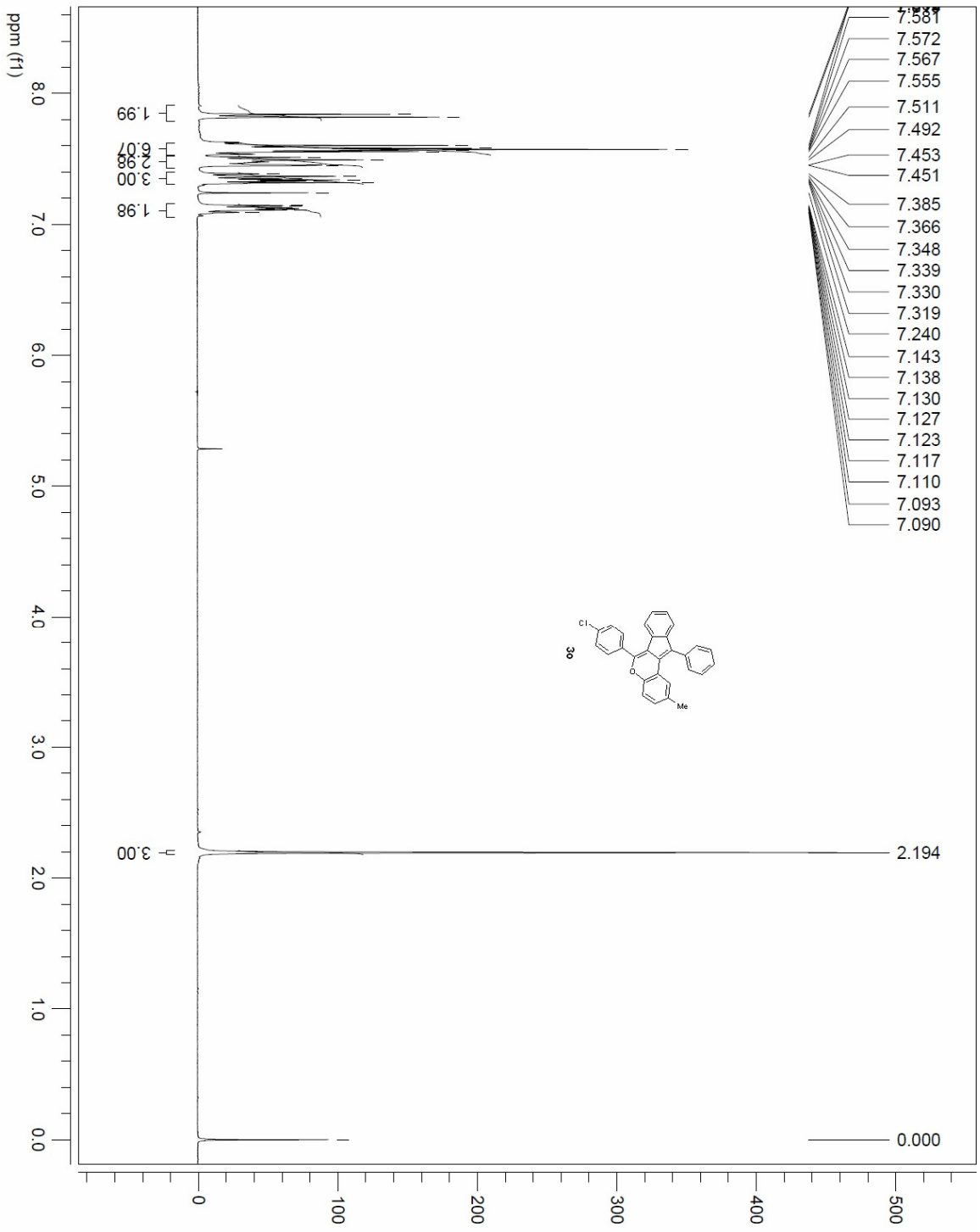


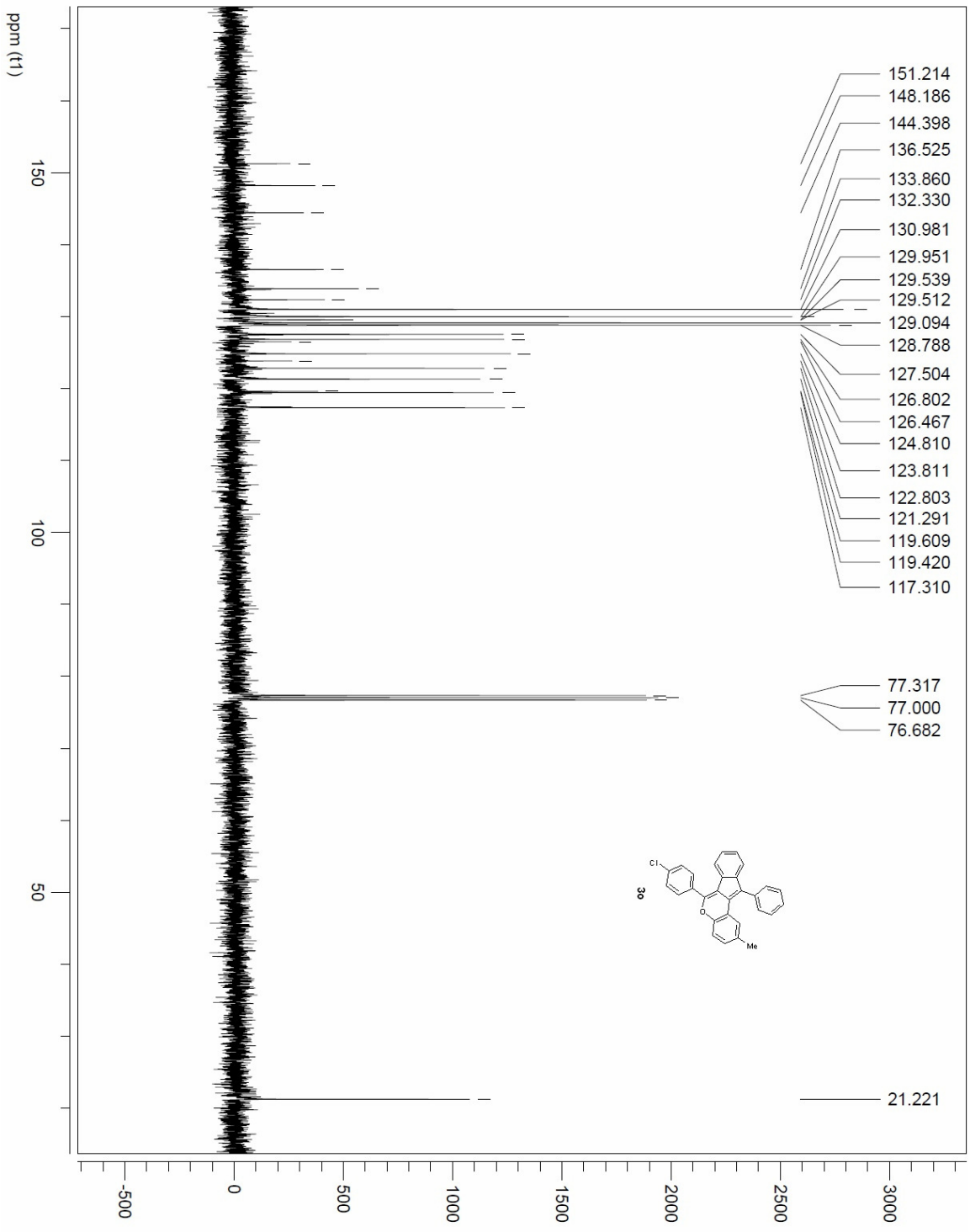


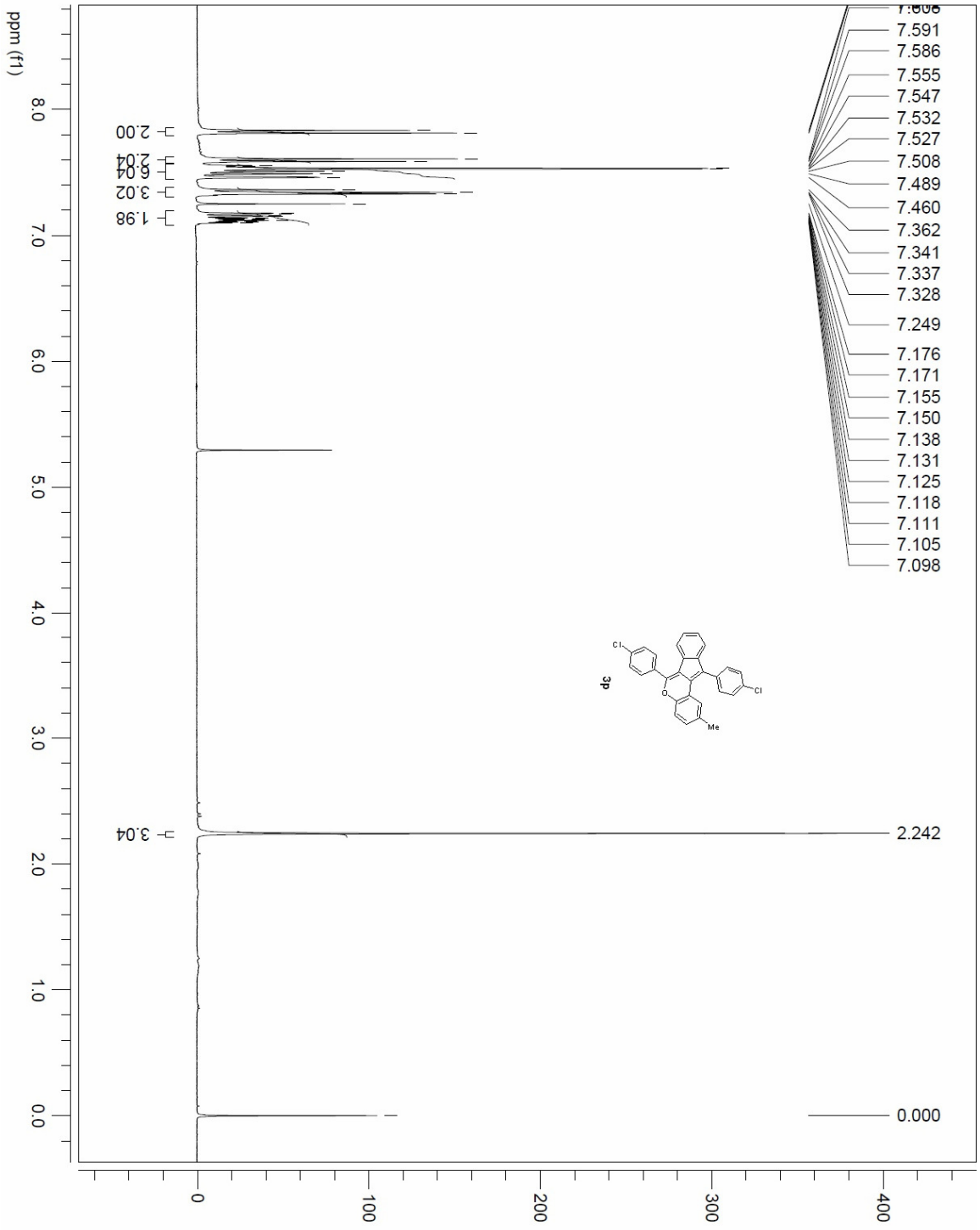


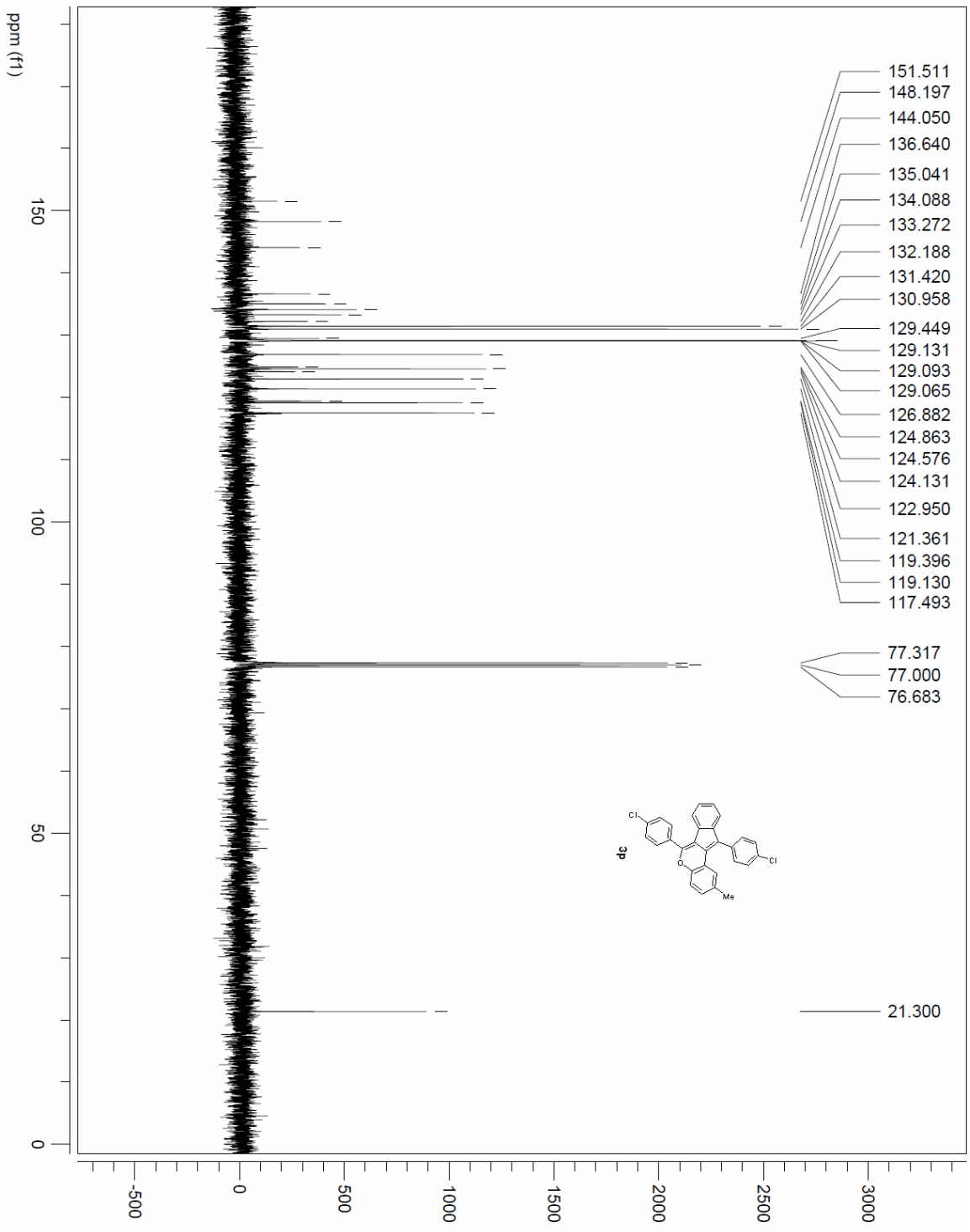


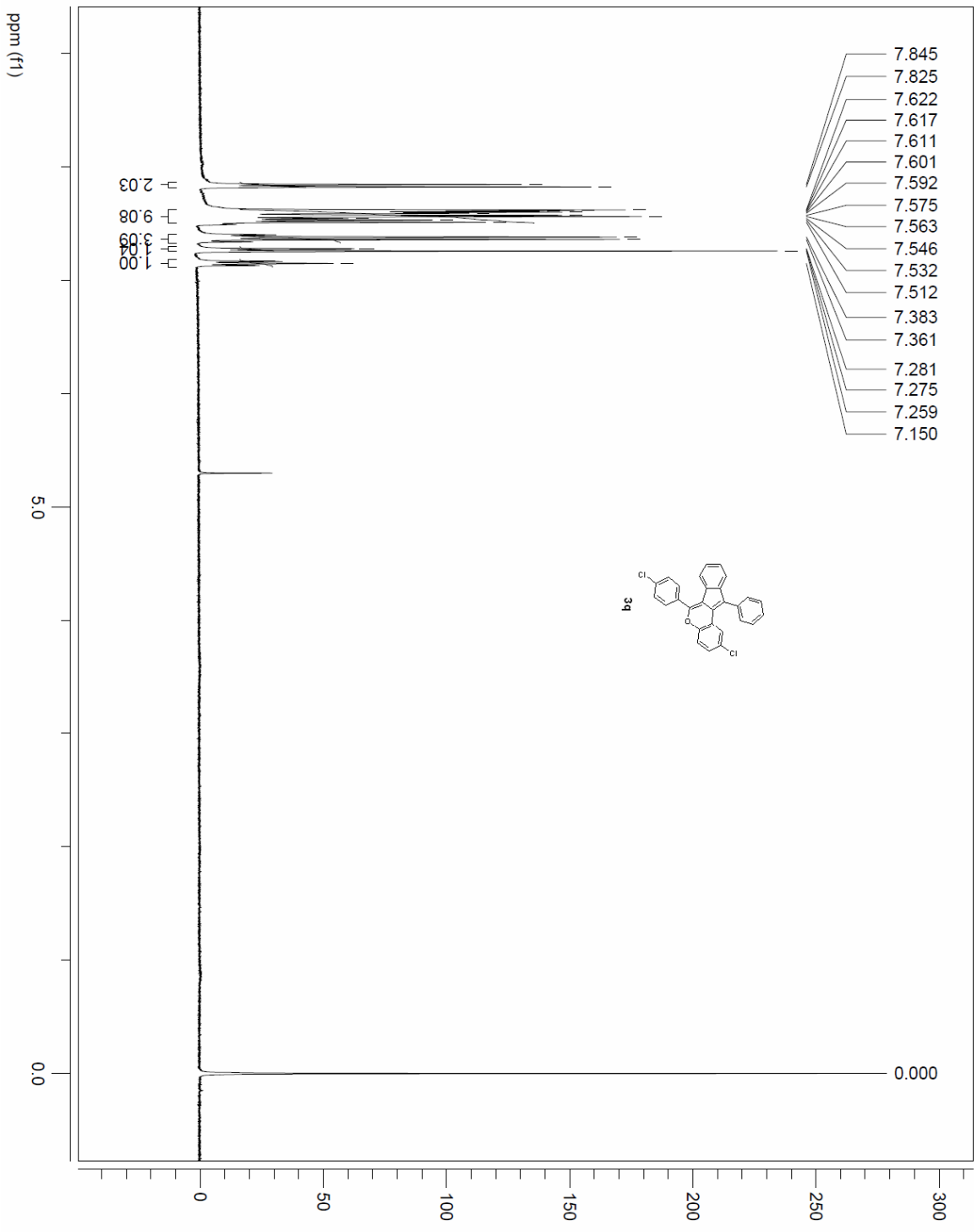


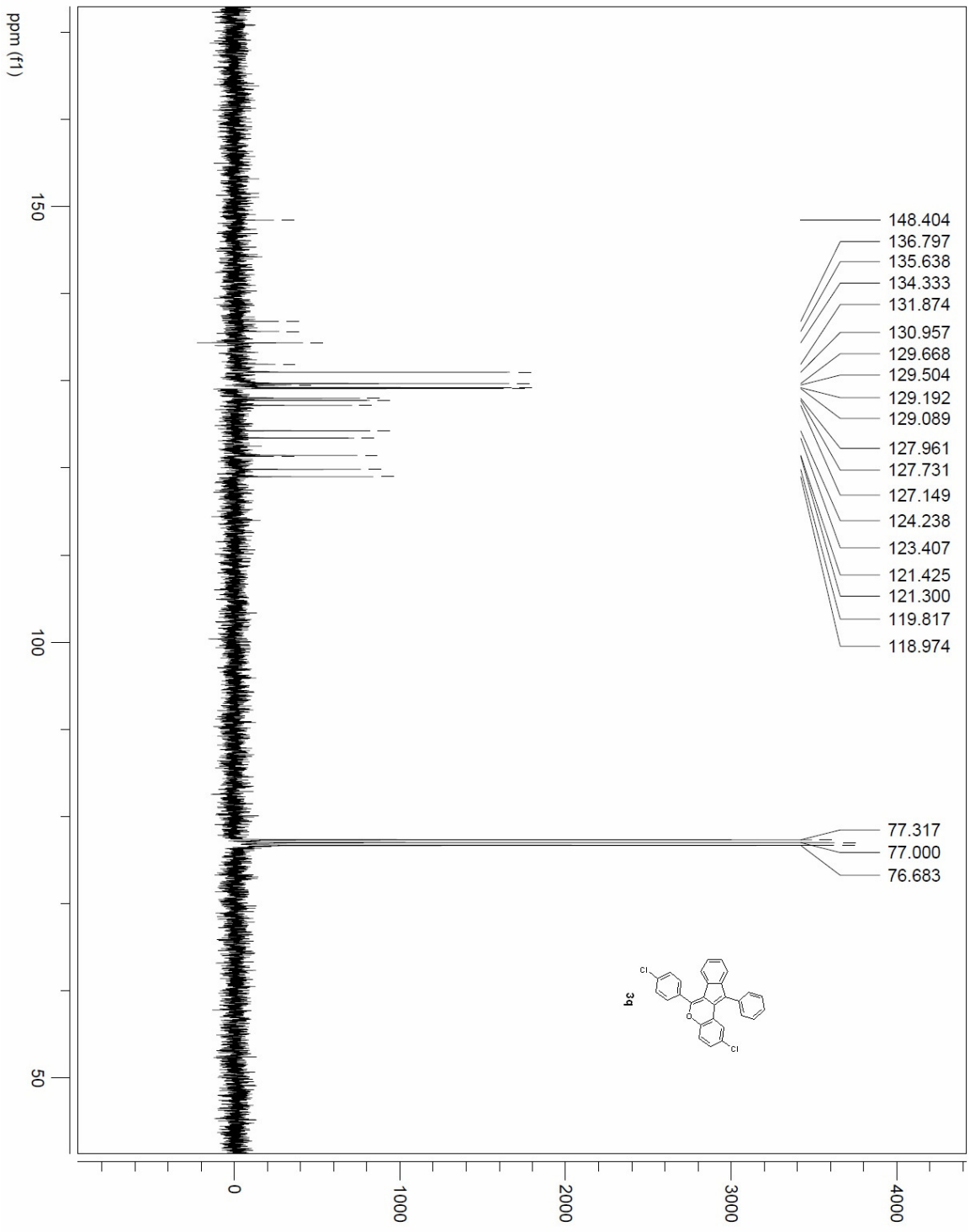


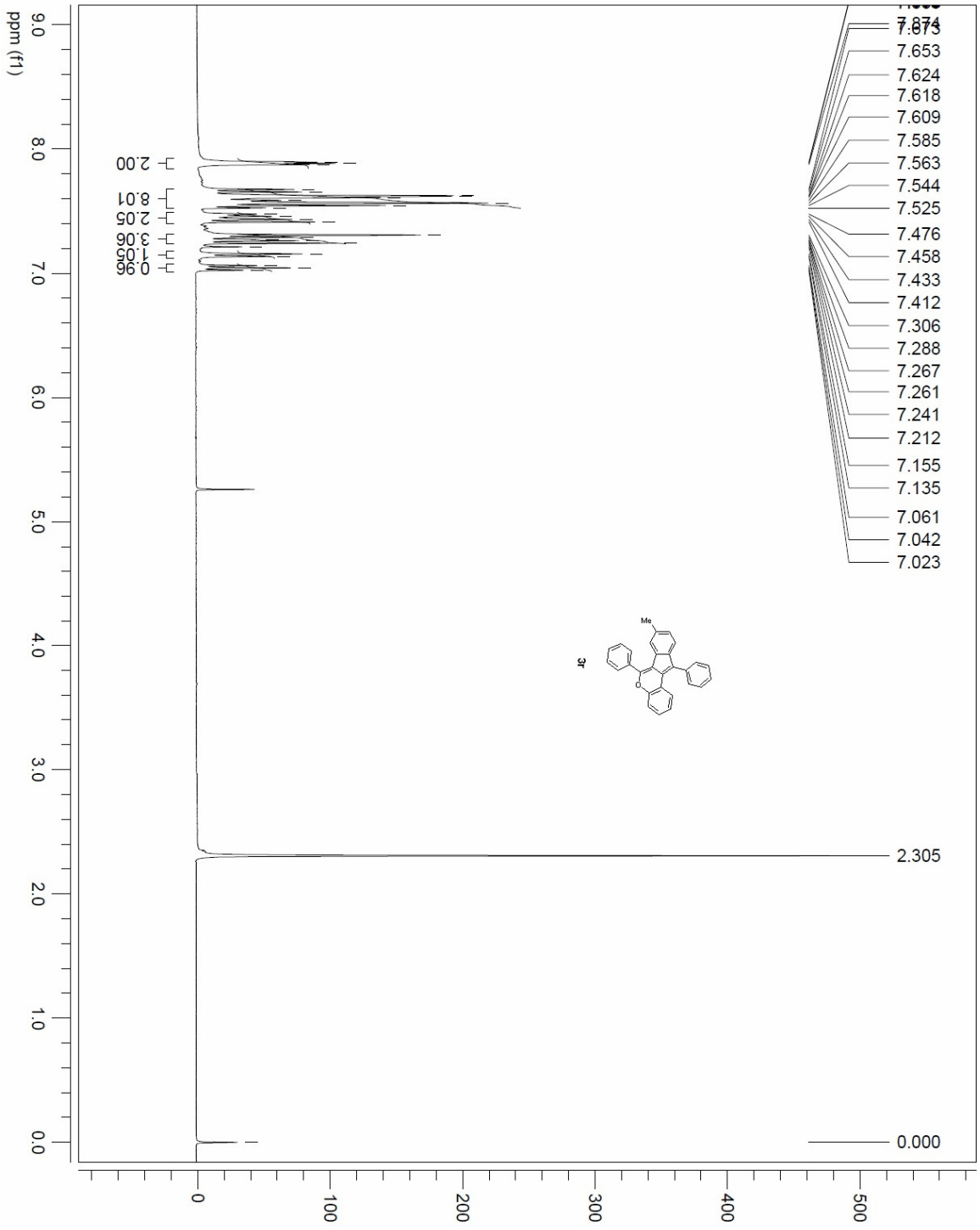


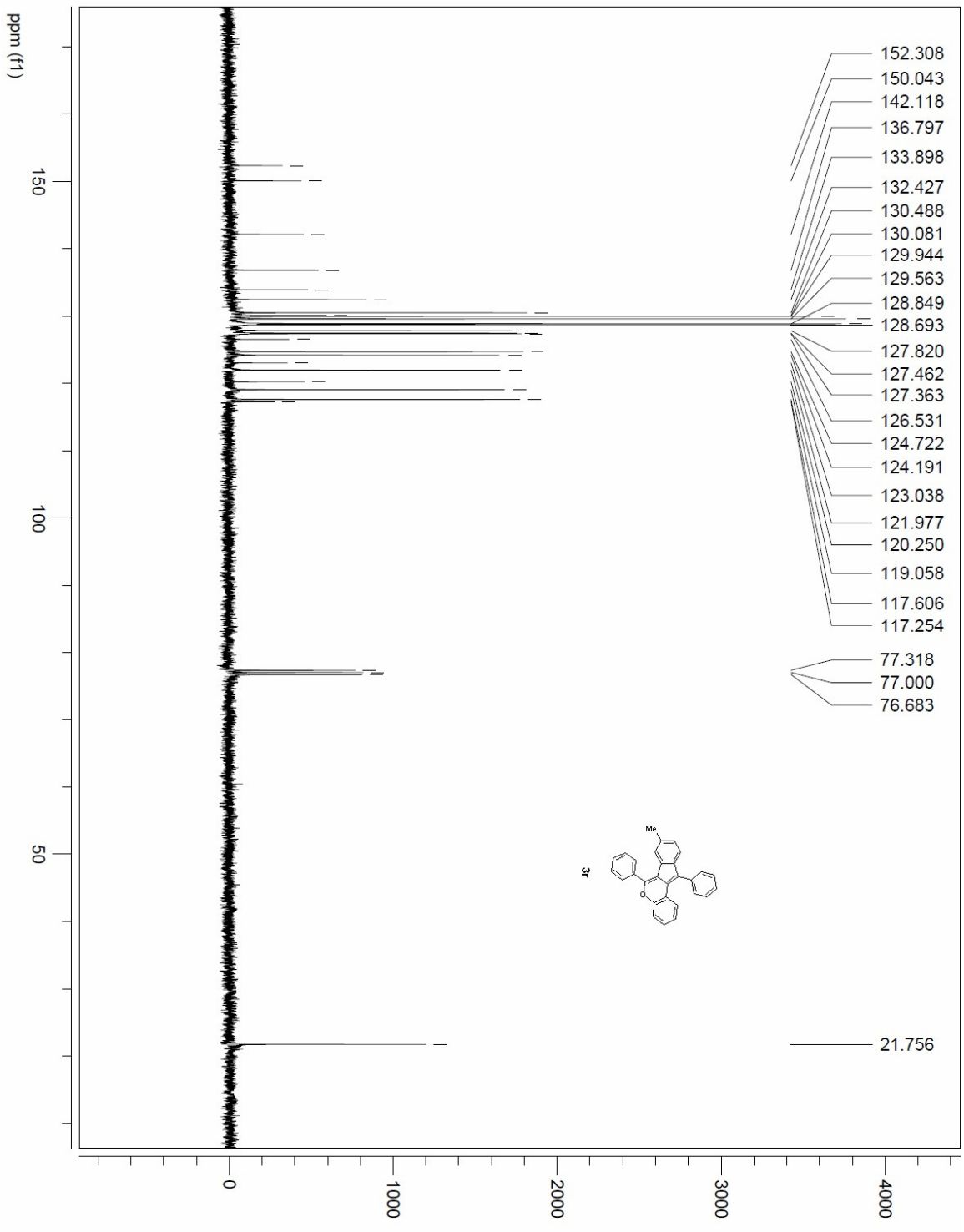


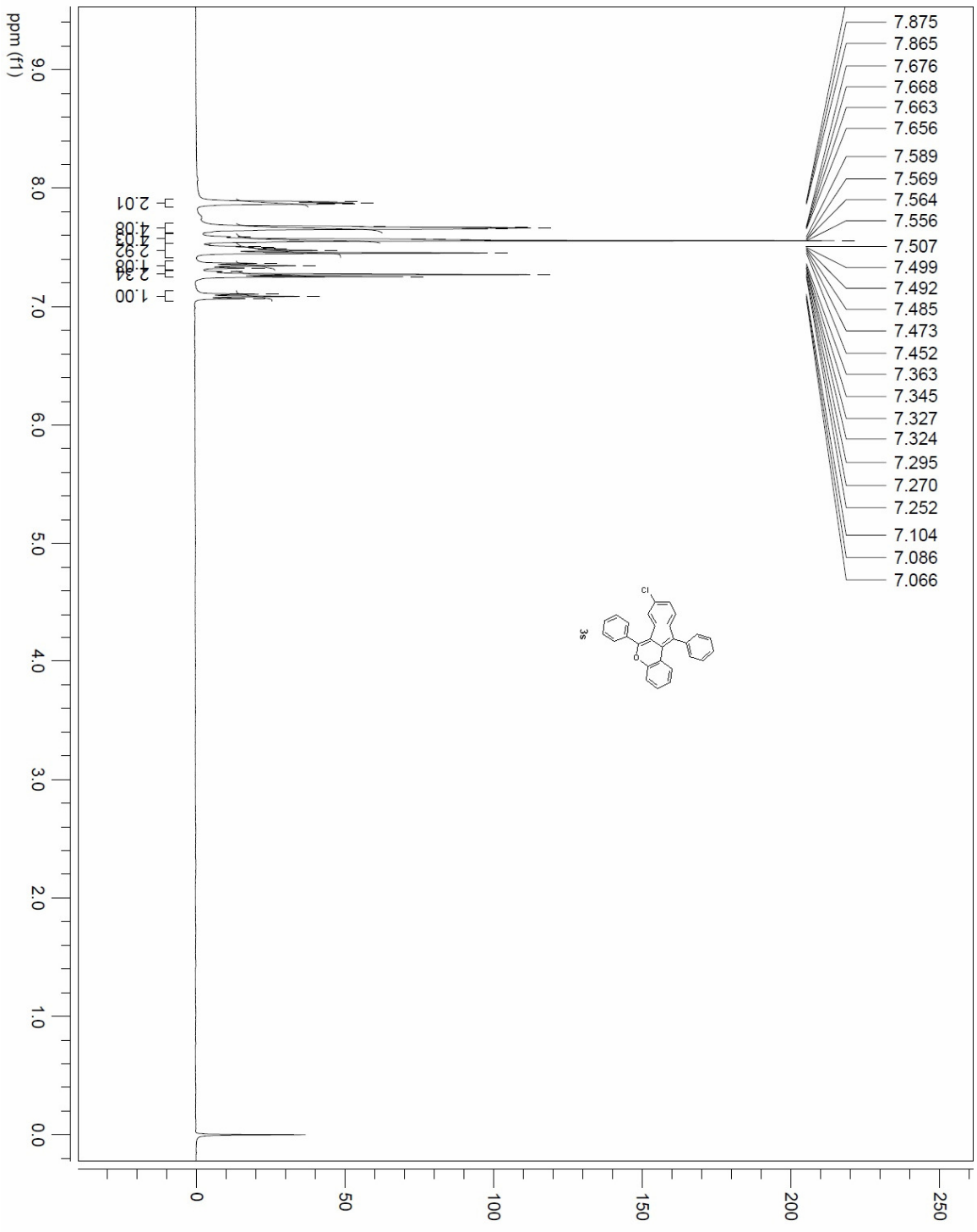


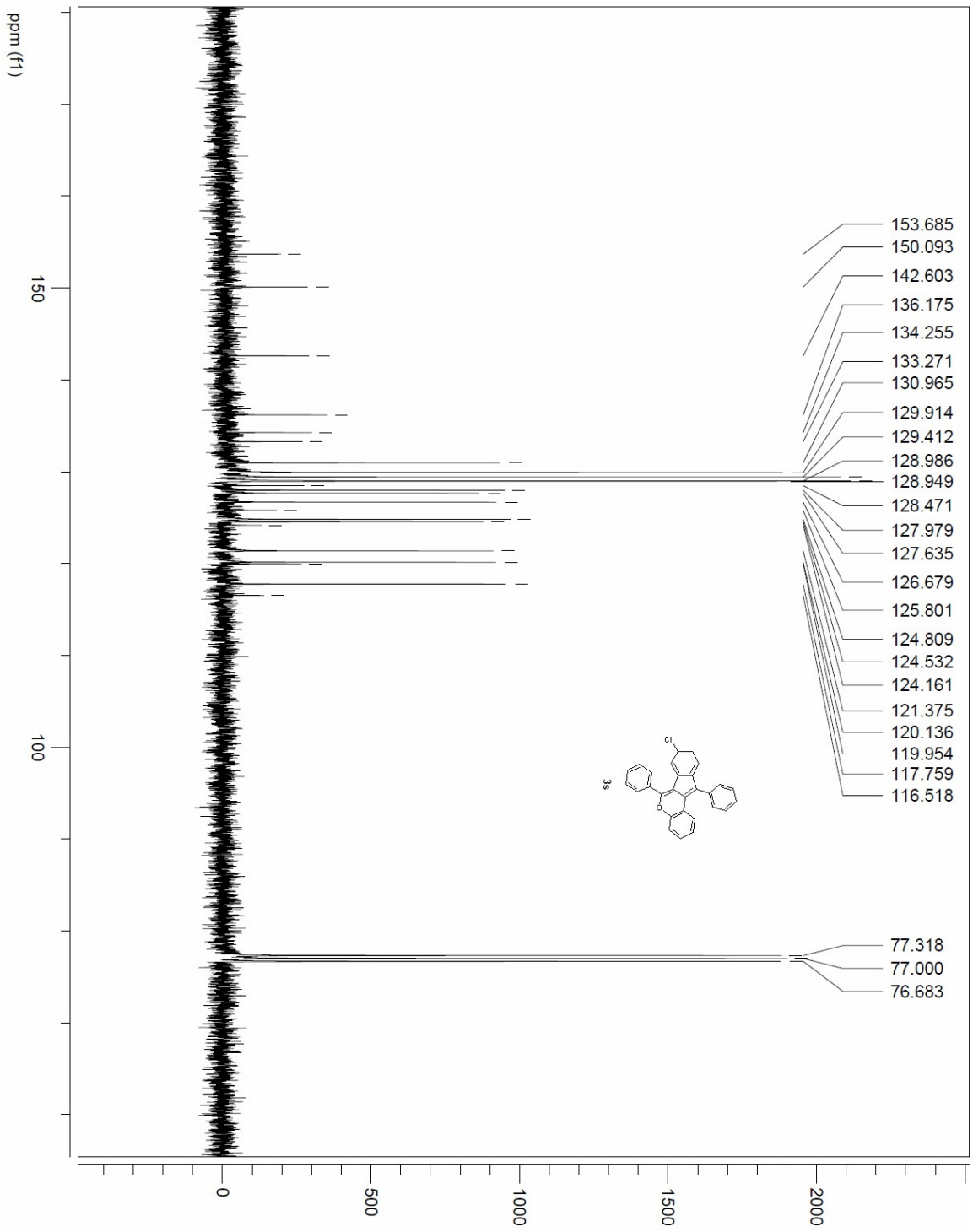


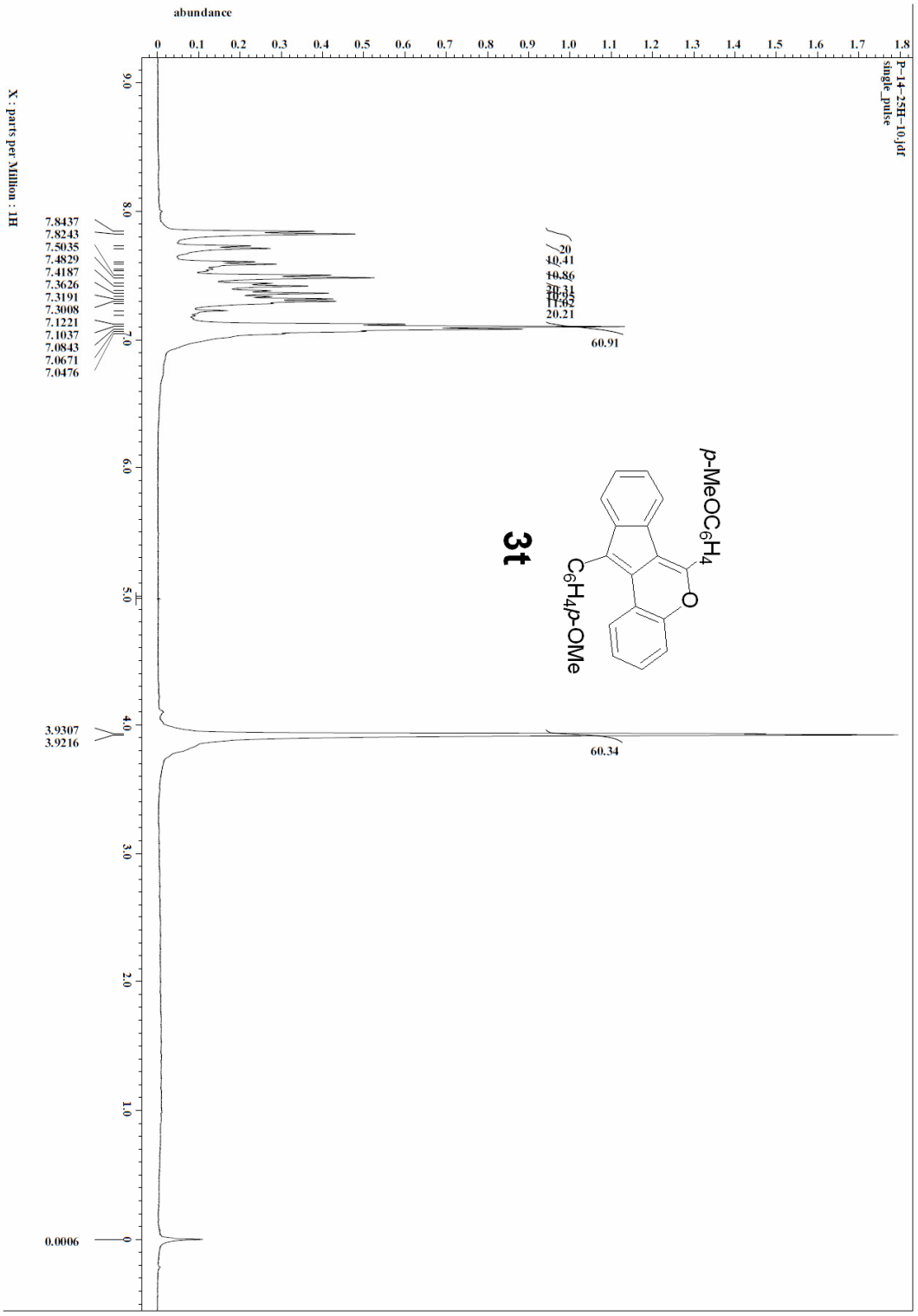


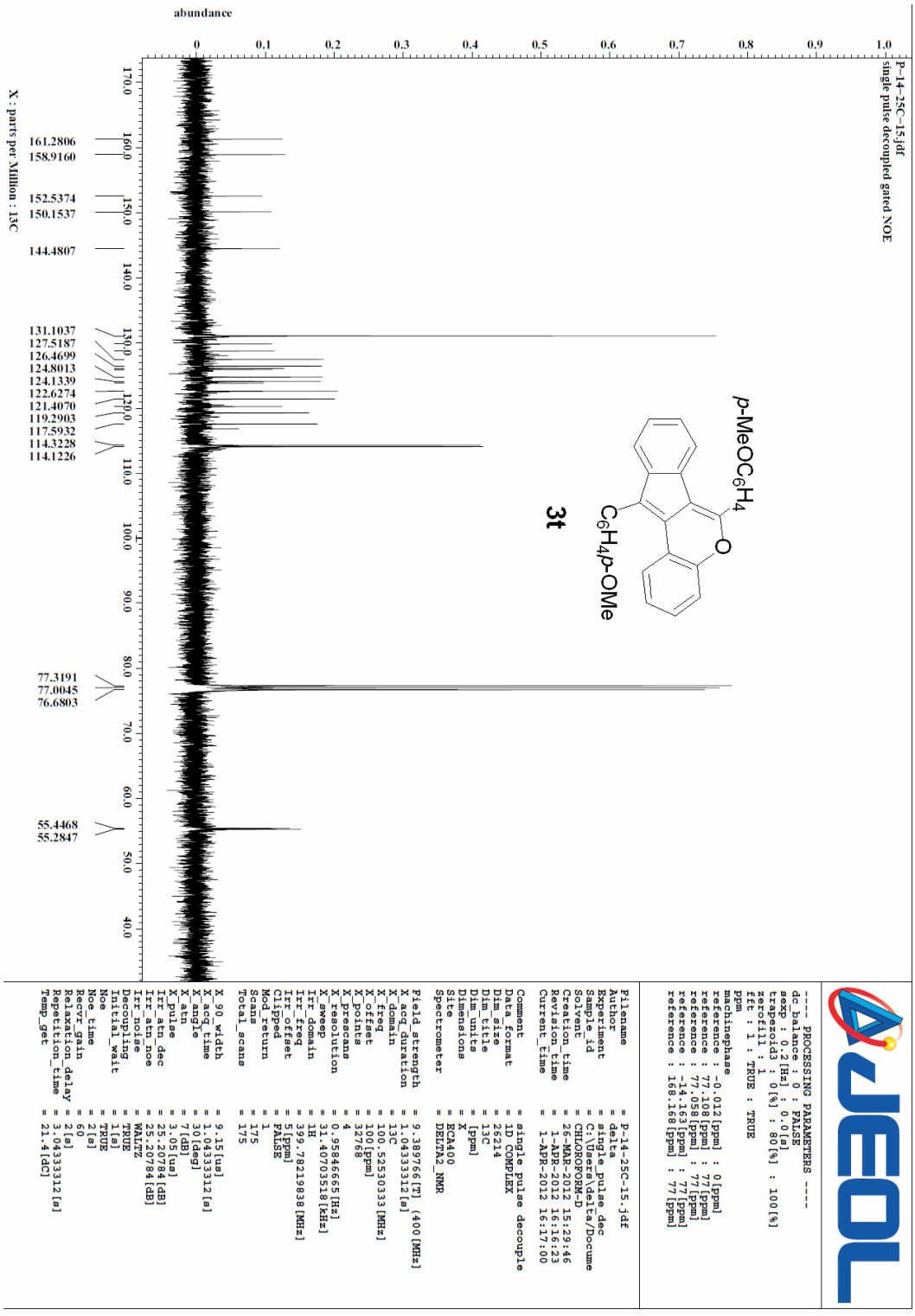


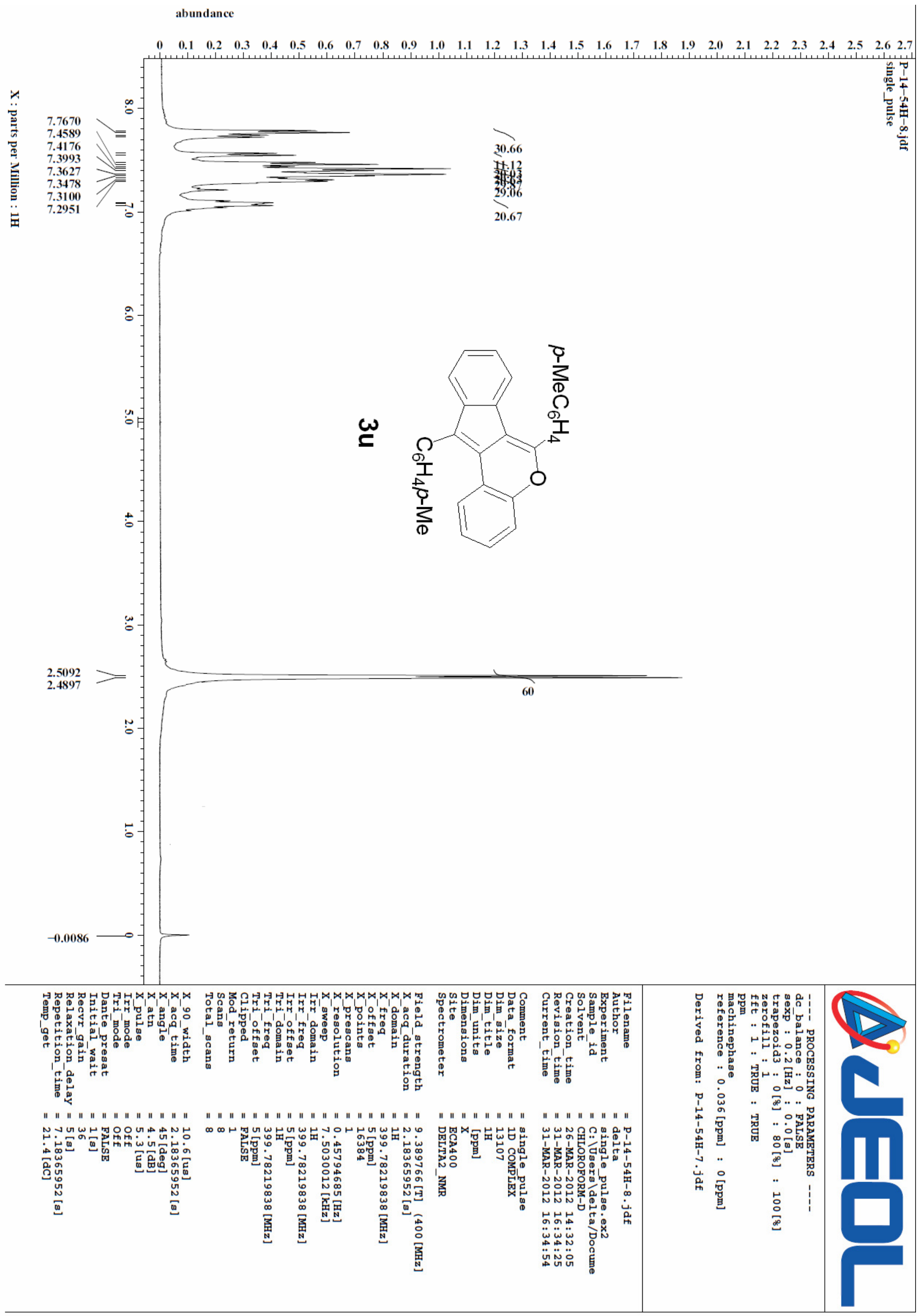


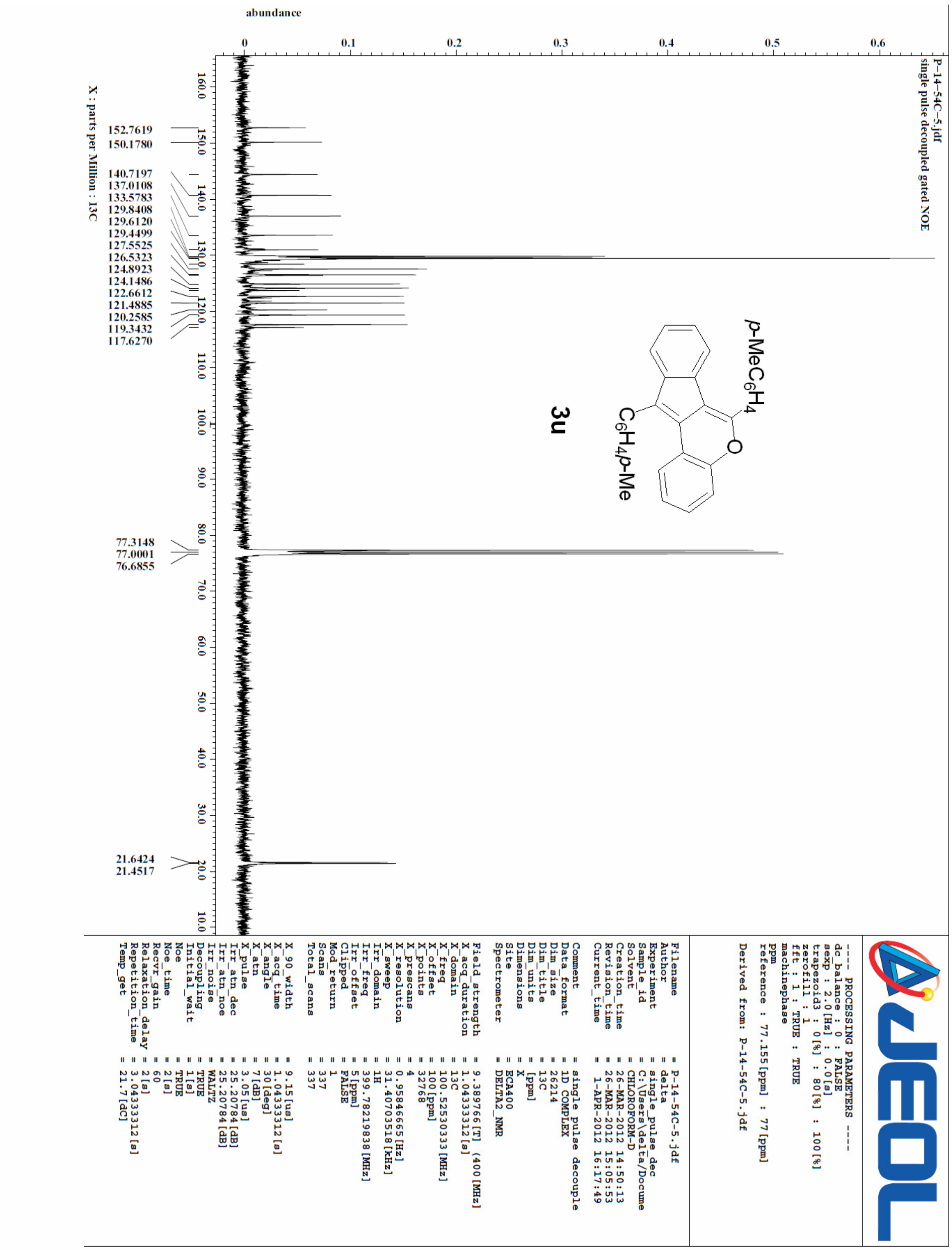


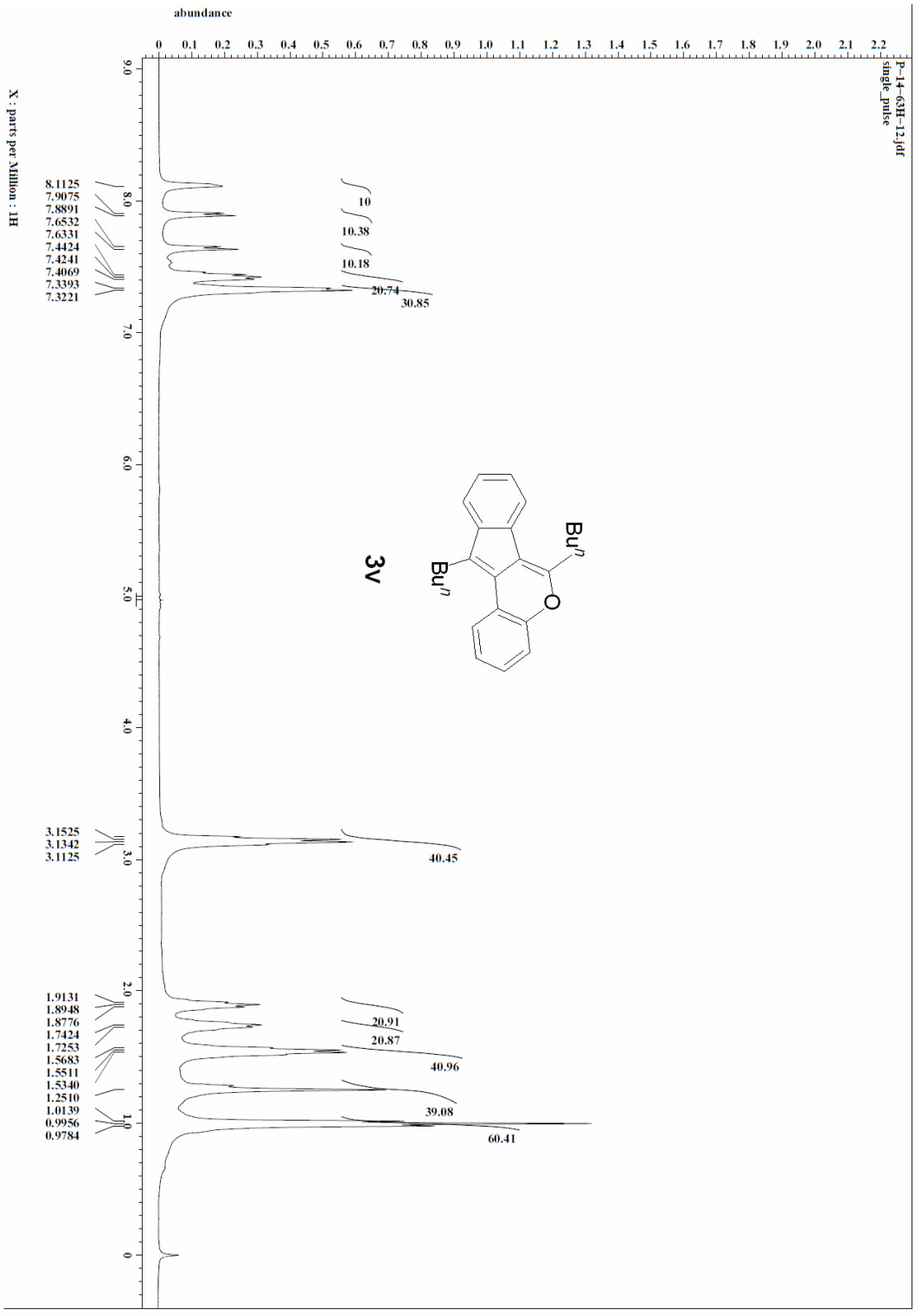


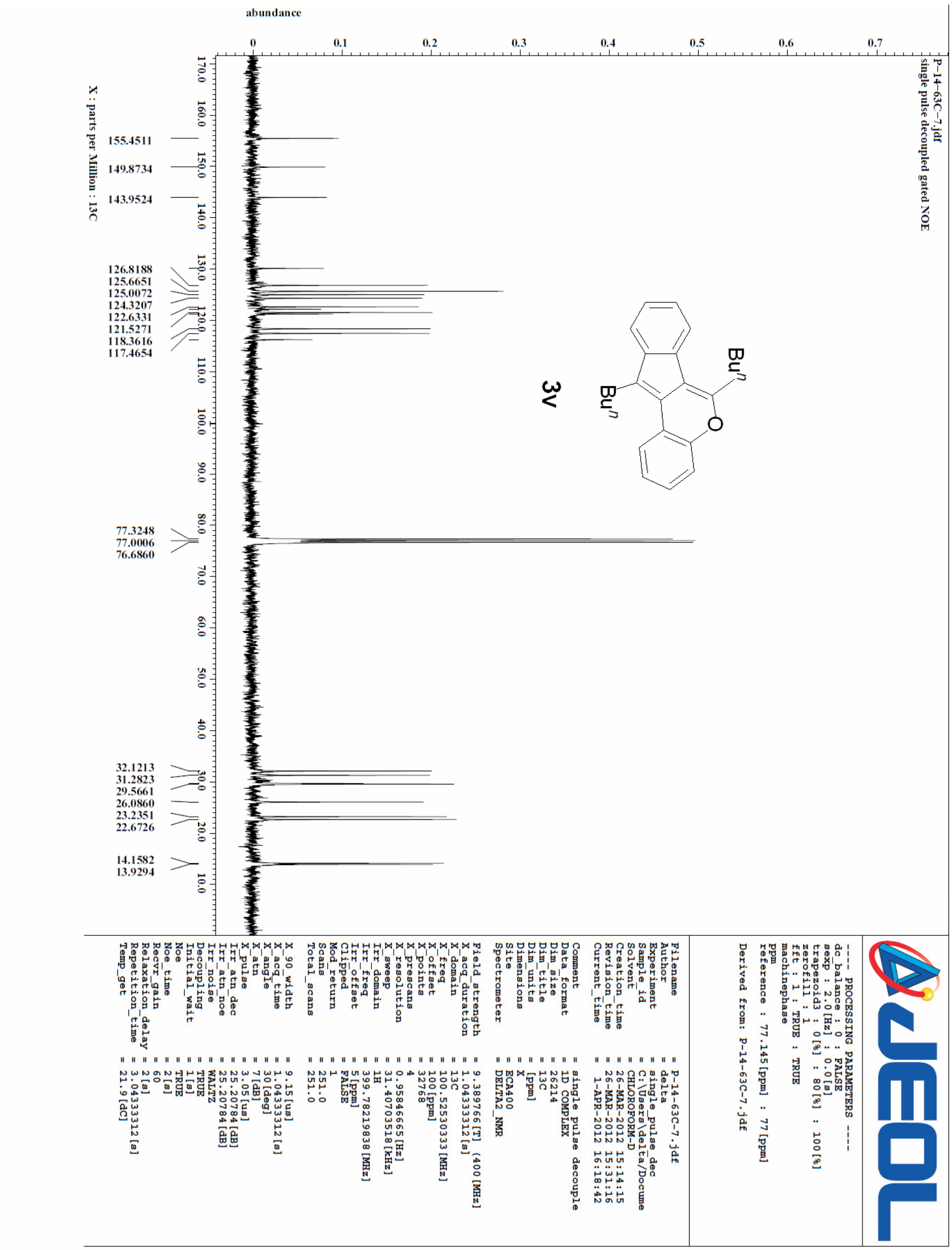


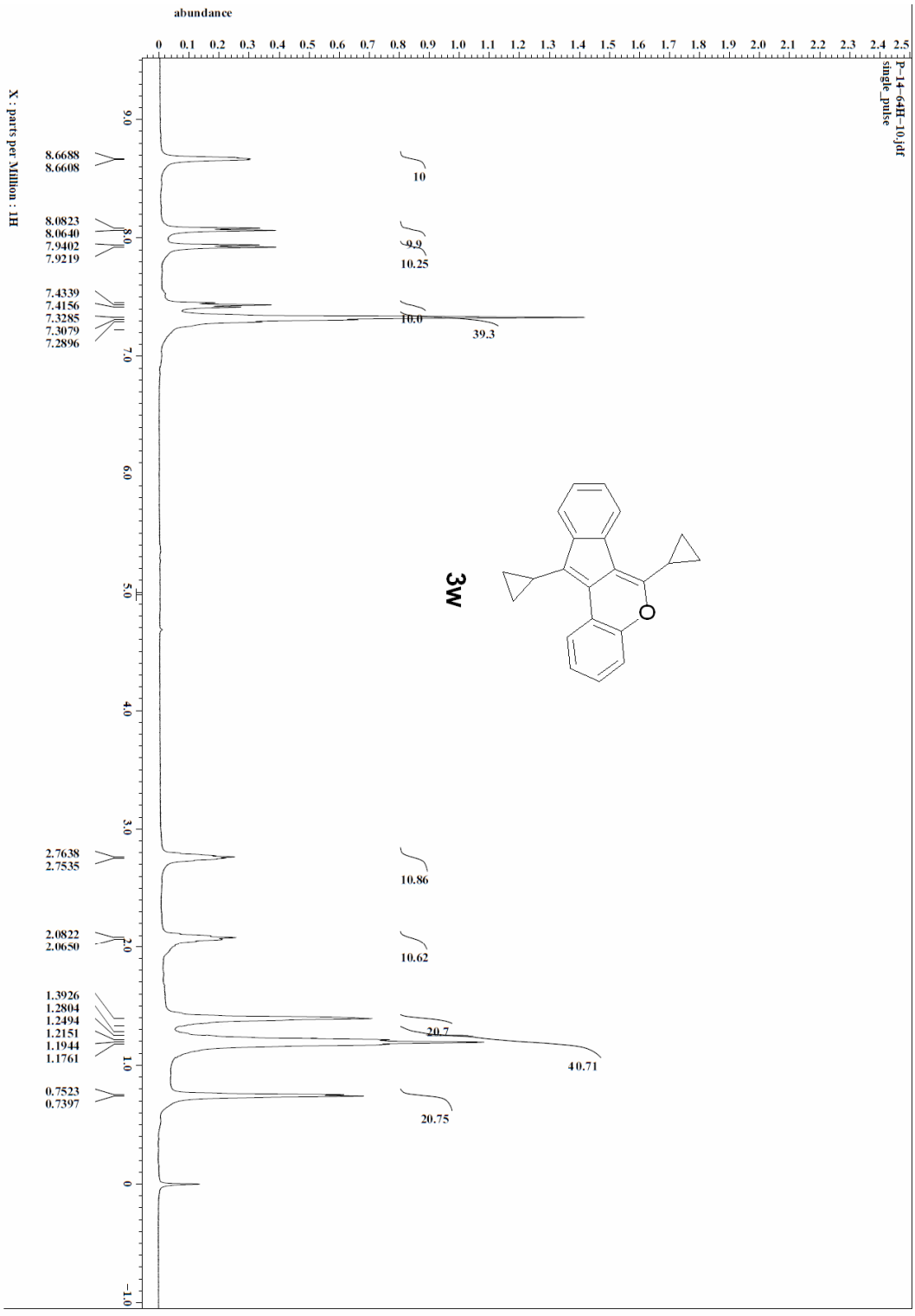

P-14-25C-15.jdf
single pulse decoupled gated NOE
abundance
X : parts per Million : 13C
161.2806
158.9160
152.5374
150.1537
144.4807
131.1037
127.5187
126.4699
124.8013
124.1339
122.6274
121.4070
119.2903
117.5932
114.3228
114.1226
77.3191
77.0045
76.6803
55.4468
55.2847
p-MeOC6H4
C6H4p-OMe
O
3t
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2 [Hz] : 0.0 [s]
trapezoid3 : 1 : 0 [%] : 80 [%] : 100 [%]
zf : 1 : TRUE : TRUE
ppm : 1 : TRUE
machinephase
reference : -0.012 [ppm] : 0 [ppm]
reference : 77.108 [ppm] : 77 [ppm]
reference : 77.058 [ppm] : 77 [ppm]
reference : -14.163 [ppm] : 77 [ppm]
reference : 168.168 [ppm] : 77 [ppm]
Filename = P-14-25C-15.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = C:\Users\delta\Docume
Solvent = CHLOROFORM-D
Creation_time = 26-MAR-2012 15:29:46
Revision_time = 1-APR-2012 16:16:23
Current_time = 1-APR-2012 16:17:00
Comment = single_pulse_decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA400
Spectrometer = DELTA2_NMR
Field_strength = 9.389766 [T] (400 [MHz]
X_acq_duration = 1.043333312 [s]
X_domain = 13C
X_freq = 100.525303333 [MHz]
X_offset = 100 [ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.958466665 [Hz]
X_sweep = 31.40703518 [kHz]
Irr_domain = 1H
Irr_freq = 399.78219838 [MHz]
Irr_offset = 5 [ppm]
Mod_Fourier = FALSE
Scans = 1
Total_scans = 175
X_90_width = 9.15 [us]
X_acq_time = 1.043333312 [s]
X_angle = 30 [deg]
X_atn = 7 [dB]
X_pulse = 3.05 [us]
Irr_atn_dec = 25.20784 [dB]
Irr_atn_noe = 25.20784 [dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1 [s]
Noe = TRUE
Noe_time = 2 [s]
Recvr_gain = 60
Relaxation_delay = 2 [s]
Repetition_time = 3.043333312 [s]
Temp_get = 21.4 [dC]

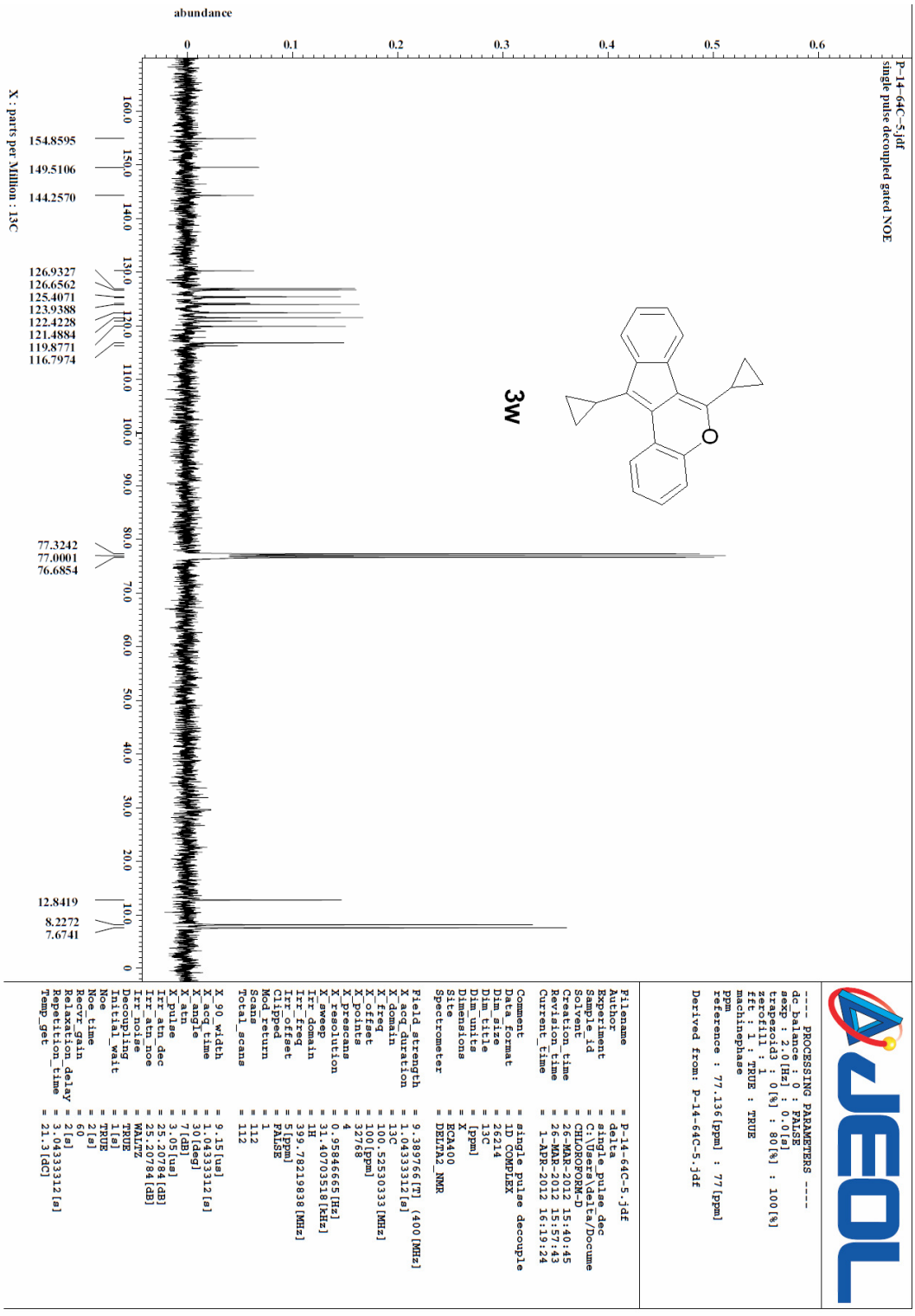

3u
p-MeC$_6$H$_4$
C$_6$H$_4$$p$-Me
O
abundance
X : parts per Million : 1H
7.7670
7.4589
7.4176
7.3993
7.3627
7.3478
7.3100
7.2951
2.5092
2.4897
−0.0086
30.66
29.06
20.67
60
P-14-54H-8.jdf
single_pulse
---- PROCESSING PARAMETERS ----
Derived from: P-14-54H-7.jdf
Filename = P-14-54H-8.jdf
Author = delta
Experiment = single_pulse.ex2
Solvent = CHLOROFORM-D
Creation_time = 26-MAR-2012 14:32:05
Revision_time = 31-MAR-2012 16:34:25
Current_time = 31-MAR-2012 16:34:54
Comment = single_pulse
Data_format = 1D COMPLEX
Spectrometer = DELTA2_NMR
Site = ECA400
Field_strength = 9.389766[T] (400[MHz])
X_freq = 399.78219838[MHz]
Scans = 8
Total_scans = 8
Temp_get = 21.4[dC]

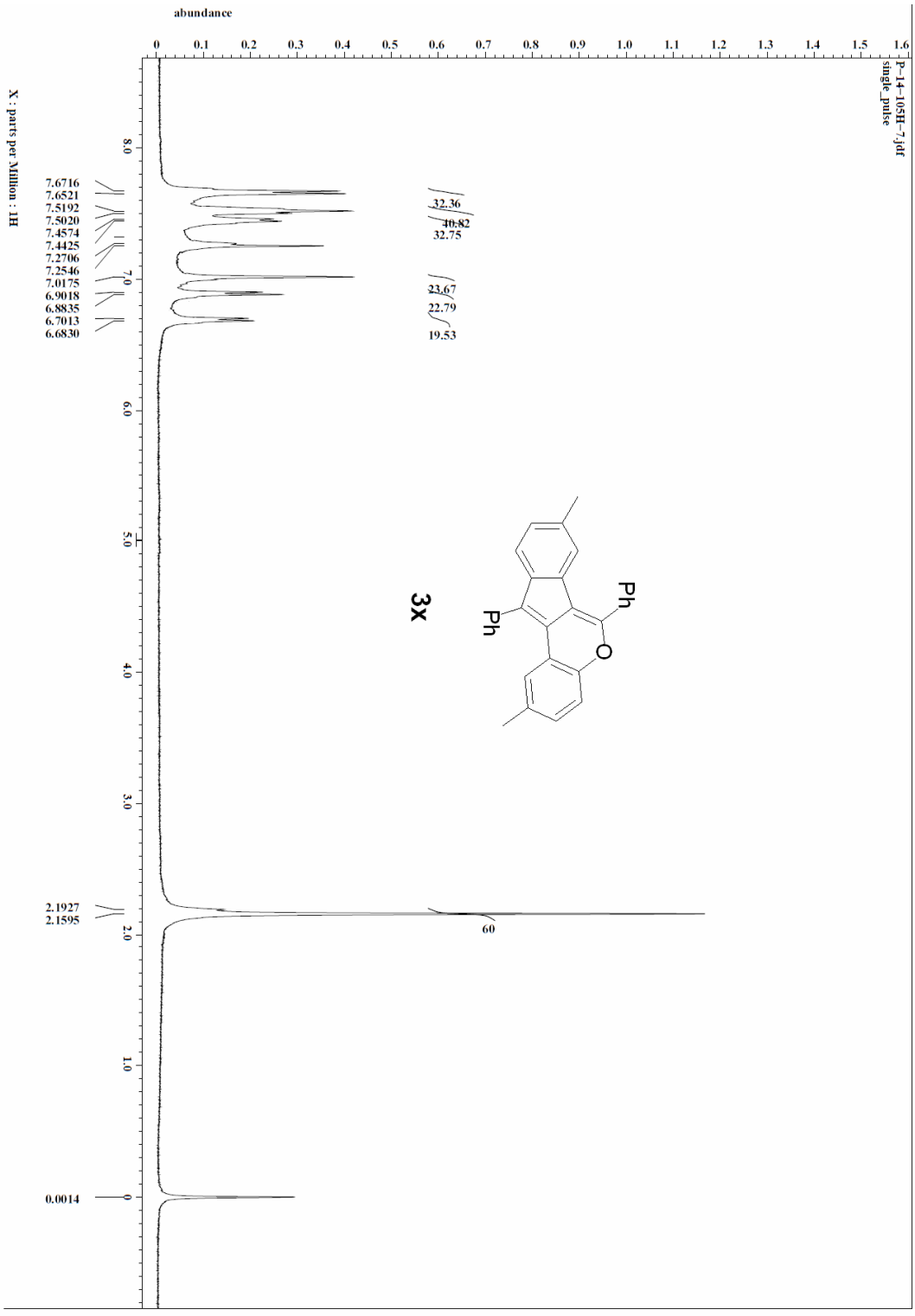

P-14-54C-5.jdf
single pulse decoupled gated NOE
abundance
X : parts per Million : 13C
152.7619
150.1780
140.7197
137.0108
133.5783
129.8408
129.6120
129.4499
127.5525
126.5323
124.8923
124.1486
122.6612
121.4885
120.2585
119.3432
117.6270
77.3148
77.0001
76.6855
21.6424
21.4517
p-MeC6H4
O
C6H4p-Me
3u
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
reference : 77.155[ppm] : 77[ppm]
Derived from: P-14-54C-5.jdf
Filename = P-14-54C-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = C:\Users\delta/Docume
Solvent = CHLOROFORM-D
Creation_time = 26-MAR-2012 14:50:13
Revision_time = 26-MAR-2012 15:05:53
Current_time = 1-APR-2012 16:17:49
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA400
Spectrometer = DELTA2_NMR
Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 337
Total_scans = 337
X_90_width = 9.15[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 7[dB]
X_pulse = 3.05[us]
Irr_atn_dec = 25.20784[dB]
Irr_atn_noe = 25.20784[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.04333312[s]
Temp_get = 21.7[dC]

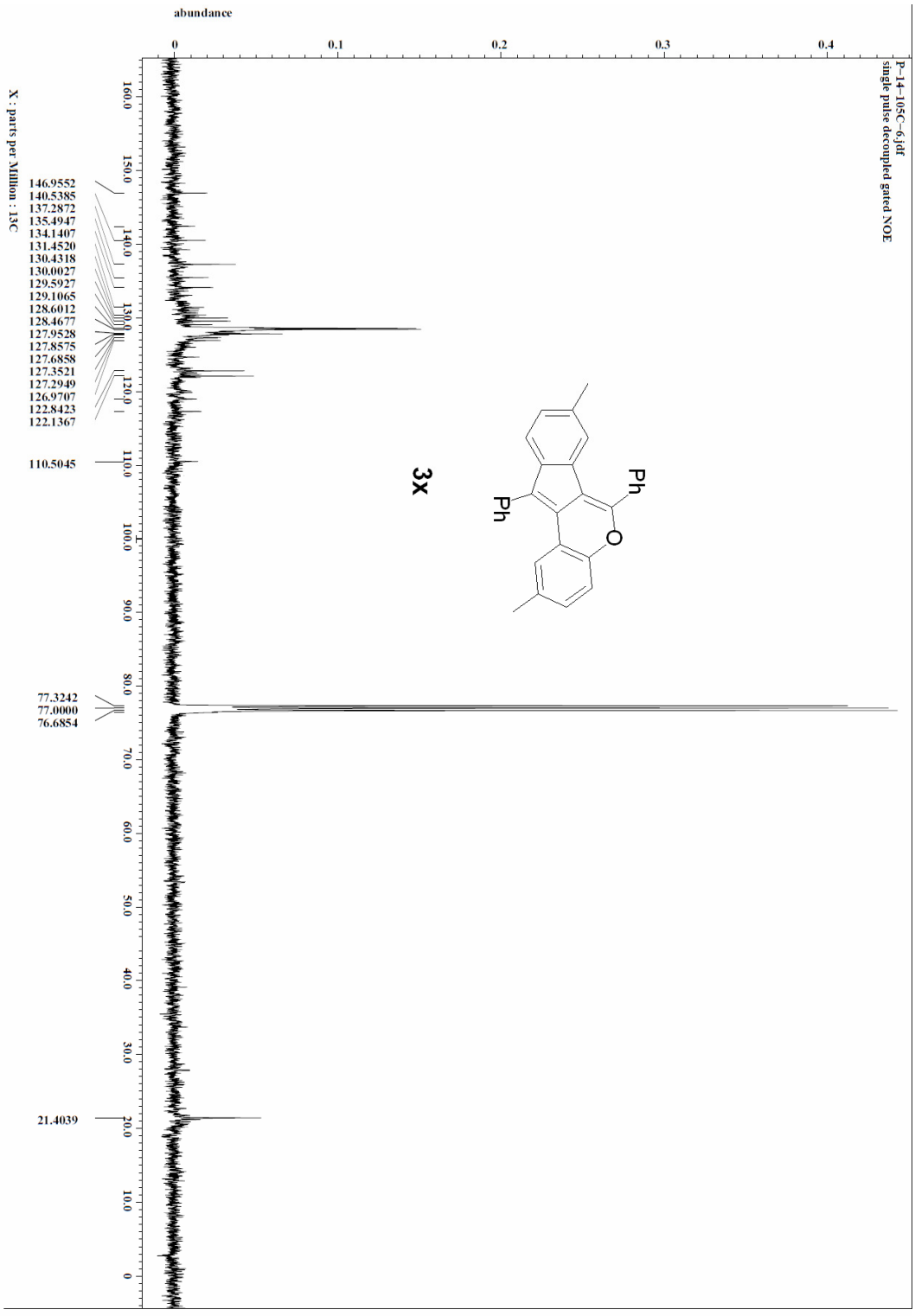


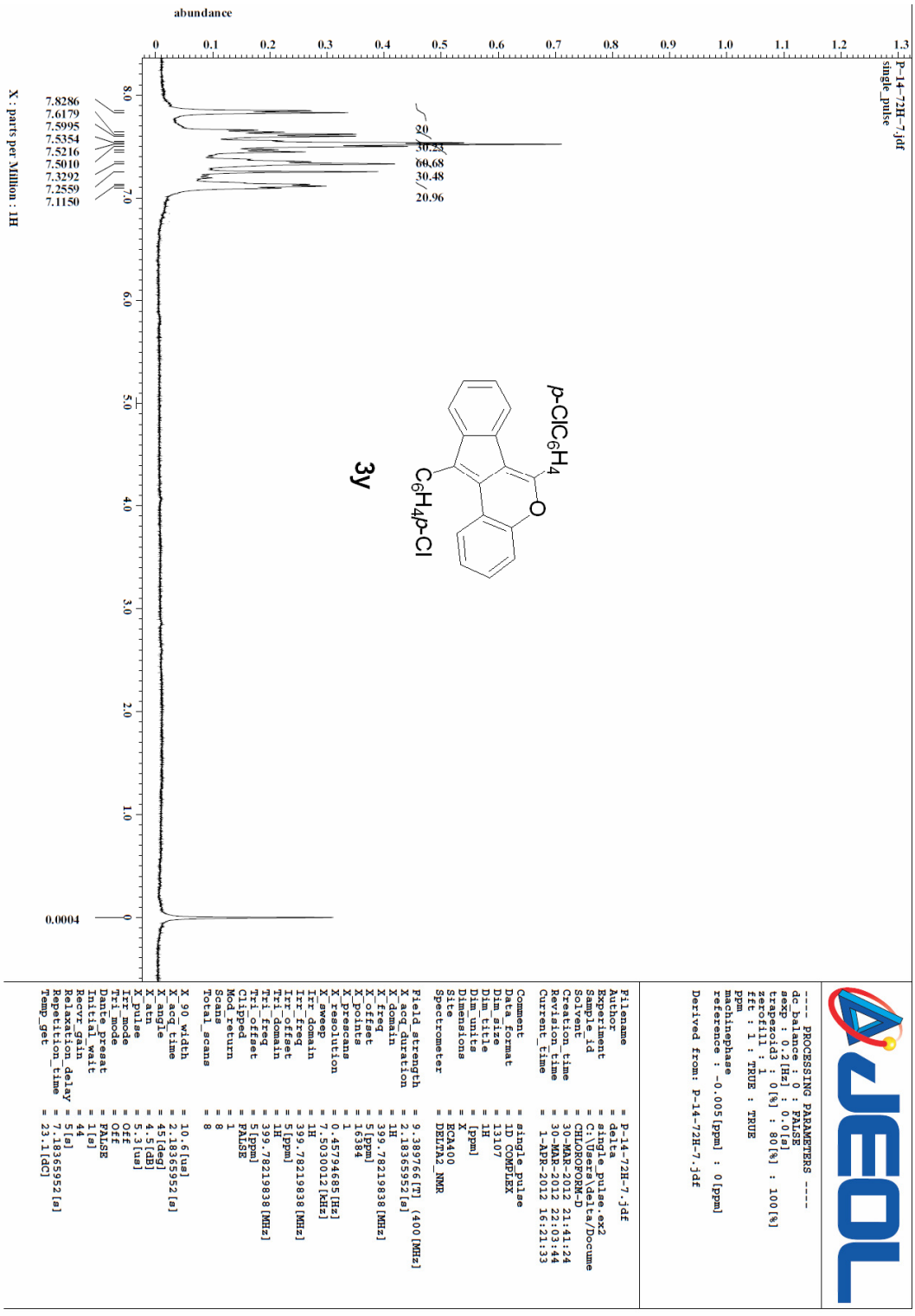

P-14-63C-7.jdf
single pulse decoupled gated NOE
abundance
X : parts per Million : 13C
3v

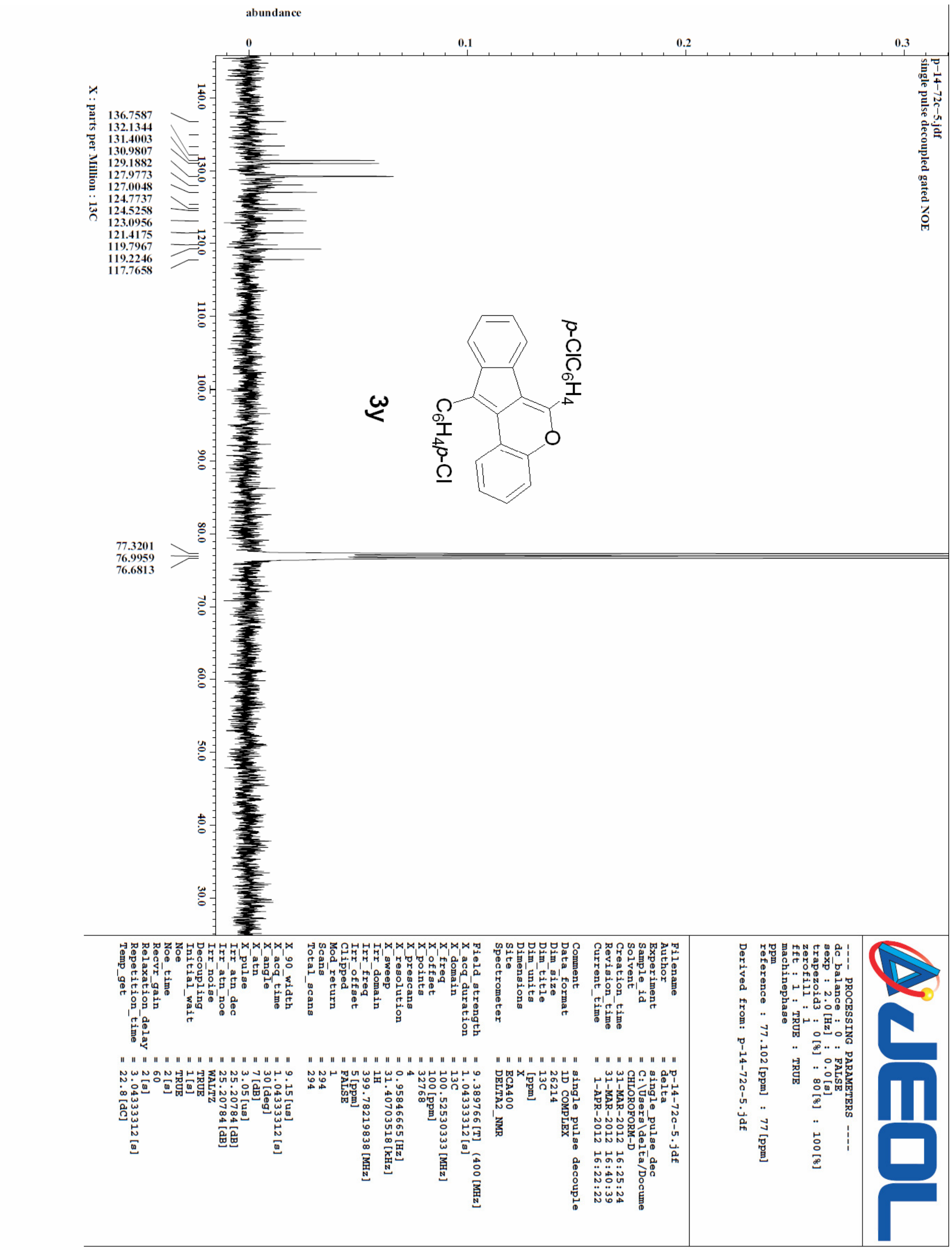



P-14-64C-5.jdf
single pulse decoupled gated NOE
abundance
X : parts per Million : 13C
154.8595
149.5106
144.2570
126.9327
126.6562
125.4071
123.9388
122.4228
121.4884
119.8771
116.7974
77.3242
77.0001
76.6854
12.8419
8.2272
7.6741
3w
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 2.0 [Hz] : 0.0 [s]
trapezoid : 1 : 0 [%] : 80 [%] : 100 [%]
zerofill : 1 : TRUE : TRUE
ffe : TRUE
machinephase
ppm
reference : 77.136 [ppm] : 77 [ppm]
Derived from: P-14-64C-5.jdf
Filename = P-14-64C-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = CHLOROFORM-D
Solvent = C:\Users\delta\Docume
Creation_time = 26-MAR-2012 15:40:45
Revision_time = 26-MAR-2012 15:57:43
Current_time = 1-APR-2012 16:19:24
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA400
Spectrometer = DELTA2_NMR
Field_strength = 9.389766 [T] (400 [MHz]
X_acq_duration = 1.04333312 [s]
X_domain = 13C
X_freq = 100.525030333 [MHz]
X_offset = 100 [ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.958466665 [Hz]
X_sweep = 31.40703518 [kHz]
Irr_domain = 1H
Irr_freq = 399.78219838 [MHz]
Irr_offset = 5 [ppm]
Mod_Preturn = FALSE
Scans = 112
Total_scans = 112
X_90_width = 9.15 [us]
X_acq_time = 1.04333312 [s]
X_angle = 30 [deg]
X_atn = 7 [dB]
X_pulse = 3.05 [us]
Irr_atn_dec = 25.20784 [dB]
Irr_atn_noe = 25.20784 [dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1 [s]
Noe = TRUE
Noe_time = 2 [s]
Recvr_gain = 60
Relaxation_delay = 2 [s]
Repetition_time = 3.04333312 [s]
Temp_get = 21.3 [dC]






P-14-72H-7.jdf
single_pulse
abundance
X : parts per Million : 1H
7.8286
7.6179
7.5995
7.5354
7.5216
7.5010
7.3292
7.2559
7.1150
0.0004
20
30.23
60.68
30.48
20.96
p-ClC6H4
C6H4p-Cl
O
3y
JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
exp : 0.2 [Hz] : 0.0 [s]
trapezoid : 1 : 0 [%] : 80 [%] : 100 [%]
zerofill : TRUE : TRUE
fft : 1
ppm
machinephase
reference : -0.005 [ppm] : 0 [ppm]
Derived from: P-14-72H-7.jdf
Filename = P-14-72H-7.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = C:\Users\delta\Docume
Solvent = CHLOROFORM-D
Creation_time = 30-MAR-2012 21:41:24
Revision_time = 30-MAR-2012 22:03:44
Current_time = 1-APR-2012 16:21:33
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA400
Spectrometer = DELTA2_NMR
Field_strength = 9.389766 [T] (400 [MHz]
X_acq_duration = 2.18355952 [s]
X_domain = 1H
X_freq = 399.78219838 [MHz]
X_offset = 5 [ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.45794685 [Hz]
X_sweep = 7.5030012 [kHz]
Irr_domain = 1H
Irr_freq = 399.78219838 [MHz]
Irr_offset = 5 [ppm]
Irr_resolution = 1
Tri_domain = 1H
Tri_freq = 399.78219838 [MHz]
Tri_offset = 5 [ppm]
Clipped = FALSE
Mod_Preturn = 1
Scans = 8
Total_scans = 8
X_90_width = 10.6 [us]
X_acq_time = 2.18355952 [s]
X_angle = 45 [deg]
X_atn = 4.5 [dB]
X_pulse = 5.3 [us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1 [s]
Recvr_gain = 44
Relaxation_delay = 5 [s]
Repetition_time = 7.18355952 [s]
Temp_get = 23.1 [dC]


p-14-72c-5.jdf
single pulse decoupled gated NOE
abundance
X : parts per Million : 13C
136.7587
132.1344
131.4003
130.9807
129.1882
127.9773
127.0048
124.7737
124.5258
123.0956
121.4175
119.7967
119.2246
117.7658
77.3201
76.9959
76.6813
3y