Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Imino Exchange Reaction in Dearomatization Strategy: Synthesis of *N*-Acyl Diarylamines and Phenothiazines from Two Anilines

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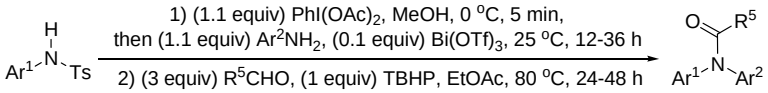
Supporting Information

1. General Information

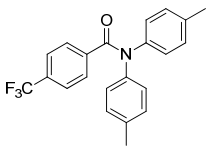
All reactions were performed in Schlenk tubes under nitrogen atmosphere. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated 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.

2. General Procedure and Spectral Data of Products

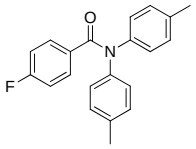
1) Synthesis of *N*-acyl diarylamines from anilines and aldehydes



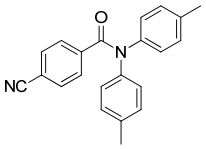
$PhI(OAc)_2$ (0.22 mmol) was added into the solution of *N*-sulfonyl protected *para*-substituted aniline (0.2 mmol) in MeOH (2 mL) at 0 °C. After 5 min, the second aniline (0.22 mmol) and $Bi(OTf)_3$ (0.02 mmol) were added. The reaction was stirred at 25 °C until the consumption of *N*-sulfonyl cyclohexadienimine determined by TLC, then was quenched with saturated $NaHCO_3$ (20 mL), and extracted by ethyl acetate (25 mL x 3). The organic layer was dried over anhydrated Na_2SO_4, and concentrated in vacuo. The crude product was treated EtOAc (2 mL), TBHP (0.2 mmol), and aldehyde (0.6 mmol). The resulting mixture was stirred at 80 °C until the consumption of *N*-aryl cyclohexadienimine determined by TLC. The reaction was quenched with saturated $NaHCO_3$ (20 mL), and extracted by ethyl acetate (25 mL x 3). The organic layer was dried over anhydrated Na_2SO_4, and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (hexanes/ethyl acetate = 10:1) to afford the pure product.



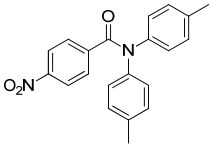
***N,N*-Di-*p*-tolyl-4-(trifluoromethyl)benzamide 3:** white solid; m.p. 126-127 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, J = 8.1 Hz, 2 H), 7.46 (d, J = 8.2 Hz, 2 H), 6.85-7.18 (m, 8 H), 2.30 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.0, 140.7, 139.9, 136.5, 131.6, 131.3, 129.8, 129.3, 124.8, 124.7, 122.3, 20.9; HRMS m/z calcd for $C_{22}H_{19}F_3NO$ ($[M+H]^+$): 370.1413, found 370.1412.



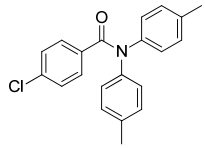
4-Fluoro-*N,N*-di-*p*-tolylbenzamide 4: brown solid; m.p. 165-166 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 - 7.48 (m, 2 H), 7.00 – 7.10 (m, 8 H), 8.85-6.90 (m, 2 H), 2.29 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.4, 164.6, 162.1, 141.3, 136.1, 132.3, 131.5, 131.4, 129.7, 127.0, 20.9; HRMS m/z calcd for $C_{21}H_{19}FNO$ ($[M+H]^+$): 320.1445, found 320.1439.



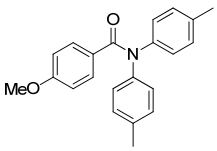
4-Cyano-*N,N*-di-*p*-tolylbenzamide 5: white solid; m.p. 180-181 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, J = 8.5 Hz, 2 H), 7.49 (d, J = 8.5 Hz, 2 H), 6.82-7.12 (m, 8 H), 2.30 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 140.9, 140.5, 136.8, 131.8, 130.0, 129.2, 127.1, 118.2, 113.4, 21.0; HRMS m/z calcd for $C_{22}H_{19}N_2O$ ($[M+H]^+$): 327.1492, found 327.1501.



4-Nitro-*N,N*-di-*p*-tolylbenzamide 6: brown solid; m.p. 146-147 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.7 Hz, 2 H), 7.59 (d, J = 8.8 Hz, 2 H), 6.75-7.27 (m, 8 H), 2.31 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.3, 148.1, 142.7, 140.4, 137.0, 130.0, 129.8, 127.2, 123.2, 21.0; HRMS m/z calcd for $C_{21}H_{19}N_2O_3$ ($[M+H]^+$): 347.1390, found 347.1389.

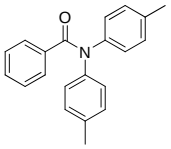


4-Chloro-*N,N*-di-*p*-tolylbenzamide 7: white solid; m.p. 139-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, J = 8.5 Hz, 2 H), 7.17 (d, J = 8.5 Hz, 2 H), 6.90 -7.12 (m, 8 H), 2.29 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.3, 141.1, 136.2, 135.9, 134.6, 130.5, 129.7, 128.0, 127.0, 20.9; HRMS m/z calcd for $C_{21}H_{19}ClNO$ ($[M+H]^+$): 336.1150, found 336.1162

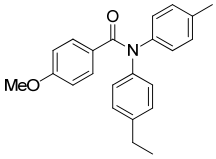


4-Methoxy-*N,N*-di-*p*-tolylbenzamide 8: white solid; m.p. 131-132 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 8.9 Hz, 2 H), 6.98-7.10 (m, 8 H), 6.70 (d, J = 8.9 Hz, 2 H) , 3.75 (s, 3 H), 2.30 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 160.9, 141.9, 135.8,

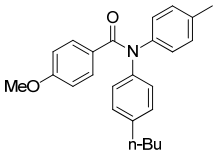
131.4, 129.7, 128.3, 127.2, 55.2, 21.0; HRMS m/z calcd for $C_{22}H_{22}NO_2$ ($[M+H]^+$): 332.1645, found 332.1648.



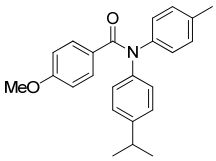
***N,N*-Di-*p*-tolylbenzamide 9:** yellow solid; m.p. 136-137 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 7.2 Hz, 2 H), 7.16 -7.27 (m, 3 H), 6.92-7.10 (m, 8 H), 2.28 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 141.5, 136.4, 136.1, 130.0, 129.7, 129.2, 127.8, 127.2, 21.0; HRMS m/z calcd for $C_{21}H_{20}NO$ ($[M+H]^+$): 302.1539, found 302.1540.



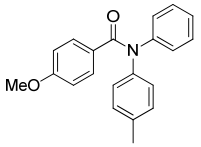
***N*-(4-Ethylphenyl)-4-methoxy-*N*-(*p*-tolyl)benzamide 11:** brown solid; m.p. 124-125 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.45 (m, 2 H), 6.97 -7.13 (m, 8 H), 6.67-6.74 (m, 2 H), 3.75 (s, 3 H), 2.62 (t, J = 15.2 Hz. J = 7.6 Hz, 2 H), 2.29 (s, 3 H), 1.20 (t, J = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 160.8, 142.0, 141.9, 141.8, 135.7, 131.3, 129.6, 128.4, 128.2, 127.1, 127.1, 112.8, 55.1, 28.2, 20.9, 15.3; HRMS m/z calcd for $C_{23}H_{24}NO_2$ ($[M+H]^+$): 346.1802, found 346.1797.



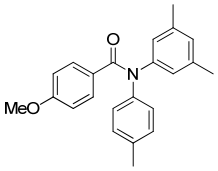
***N*-(4-Butylphenyl)-4-methoxy-*N*-(*p*-tolyl)benzamide 12:** yellow solid; m.p. 94-95 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.8 Hz, 2 H), 6.96 -7.10 (m, 8 H), 6.68 (d, J = 8.8 Hz, 2 H), 3.72 (s, 3 H), 2.55 (t, J = 7.6 Hz, 2 H), 2.28 (s, 3 H), 1.52-1.59 (m, 2 H), 1.27-1.36 (m, 2 H), 0.90 (t, J = 7.3 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 160.7, 141.8, 141.7, 140.6, 135.6, 131.2, 129.6, 128.8, 128.1, 127.0, 126.9, 112.8, 55.0, 35.0, 33.3, 22.2, 20.9, 13.8; HRMS m/z calcd for $C_{25}H_{28}NO_2$ ($[M+H]^+$): 374.2115, found 374.2133.



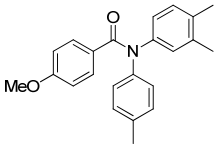
***N*-(4-Isopropylphenyl)-4-methoxy-*N*-(*p*-tolyl)benzamide 13:** yellow solid; m.p. 124-125 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.8 Hz, 2 H), 6.96-7.15 (m, 8 H), 6.69 (d, J = 8.8 Hz, 2 H), 3.74 (s, 3 H), 2.82-2.89 (m, 1 H), 2.29 (s, 3 H), 1.21 (d, J = 6.9 Hz, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 160.8, 146.5, 141.9, 141.8, 135.7, 131.2, 129.6, 128.2, 127.2, 126.9, 126.8, 112.9, 55.1, 33.5, 23.8, 20.9; HRMS m/z calcd for $C_{24}H_{26}NO_2$ ($[M+H]^+$): 360.1958, found 360.1950.



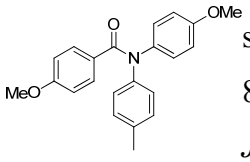
4-Methoxy-*N*-phenyl-*N*-(*p*-tolyl)benzamide 14: white solid; m.p. 151-152 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 8.8 Hz, 2 H), 7.24 -7.30 (m, 2 H), 7.05-7.18 (m, 5 H), 7.15 (d, J = 8.3 Hz, 2 H), 6.70 (d, J = 8.8 Hz, 2 H), 3.75 (s, 3 H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 161.0, 144.5, 141.8, 136.0, 131.4, 129.8, 129.1, 128.2, 127.3, 126.0, 113.1, 55.2, 21.0; HRMS m/z calcd for $C_{21}H_{20}NO_2$ ($[M+H]^+$): 318.1489, found 318.1484.



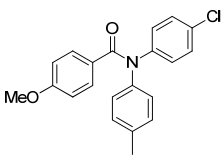
***N*-(3,5-Dimethylphenyl)-4-methoxy-*N*-(*p*-tolyl)benzamide 15;** white solid; m.p. 143-144 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, J = 8.8 Hz, 2 H), 7.06 (d, J = 8.2 Hz, 2 H), 6.99 (d, J = 8.2 Hz, 2 H), 6.81 (s, 1 H), 6.77 (s, 2 H), 6.70 (d, J = 8.8 Hz, 2 H), 3.75 (s, 3 H), 2.29 (s, 3H), 2.22 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 161.0, 144.2, 142.0, 138.7, 135.8, 131.4, 129.8, 128.4, 128.0, 127.2, 125.1, 113.0, 55.2, 21.2, 21.0; HRMS m/z calcd for $C_{23}H_{24}NO_2$ ($[M+H]^+$): 346.1802, found 346.1787.



***N*-(3,4-Dimethylphenyl)-4-methoxy-*N*-(*p*-tolyl)benzamide 16:** yellow solid; m.p. 140-141 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.46 (m, 2 H), 6.98-7.10 (m, 7 H), 6.64 - 6.72 (m, 2 H), 3.74 (s, 3 H), 2.29 (s, 3 H), 2.20 (s, 3 H), 2.16 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 160.8, 142.0, 141.9, 140.2, 137.3, 135.6, 134.6, 131.2, 130.0, 129.6, 128.2, 127.0, 124.7, 55.1, 20.9, 19.8, 19.2; HRMS m/z calcd for $C_{23}H_{24}NO_2$ ($[M+H]^+$): 346.1802, found 346.1798.

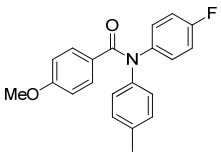


4-Methoxy-*N*-(4-methoxyphenyl)-*N*-(*p*-tolyl)benzamide 17: white solid; m.p. 112-113 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.7 Hz, 2 H), 6.96 – 7.10 (m, 6 H), 6.80 (d, J = 8.8 Hz, 2 H), 6.70 (d, J = 8.8 Hz, 2 H), 3.76 (s, 3 H), 3.75 (s, 3 H), 2.29 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 160.9, 157.6, 142.0, 137.4, 135.8, 131.3, 129.7, 128.5, 128.3, 127.0, 114.4, 113.1, 55.2, 55.3, 21.0; HRMS m/z calcd for $C_{22}H_{22}NO_3$ ($[M+H]^+$): 348.1594, found 348.1593.



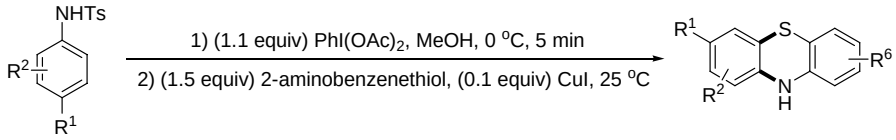
***N*-(4-chlorophenyl)-4-methoxy-*N*-(*p*-tolyl)benzamide 18:** yellow solid; m.p. 127-128 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.9 Hz, 2 H), 7.24 (d, J = 8.7 Hz, 2 H), 7.02-7.12 (m, 4 H), 6.98 (d, J = 8.3 Hz, 2 H), 6.72 (d, J = 8.8 Hz, 2 H), 3.77 (s, 3 H), 2.30 (s, 3

H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 161.1, 142.9, 141.4, 136.3, 131.3, 129.9, 129.1, 128.2, 127.7, 127.3, 113.1, 55.2, 20.9; HRMS m/z calcd for $C_{21}H_{19}ClNO_2$ ($[M+H]^+$): 352.1099, found 352.1108.

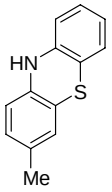


***N*-(4-Fluorophenyl)-4-methoxy-*N*-(*p*-tolyl)benzamide 19:** yellow solid; m.p. 151-152 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.8 Hz, 2 H), 6.94 -7.15 (m, 8 H), 6.71 (d, J = 8.8 Hz, 2 H), 3.76 (s, 3 H), 2.30 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 161.7, 161.0, 159.2, 141.6, 140.4, 136.1, 131.3, 129.8, 128.7, 128.6, 127.8, 127.1, 116.0, 115.7, 113.1, 55.2, 20.9; HRMS m/z calcd for $C_{21}H_{19}FNO_2$ ($[M+H]^+$): 336.1394, found 336.1389.

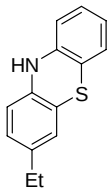
2) Synthesis of phenothiazines



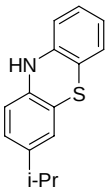
$PhI(OAc)_2$ (0.22 mmol) was added into the solution of *N*-sulfonyl protected *para*-substituted aniline (0.2 mmol) in MeOH (2 mL) in ice-water bath. After 5 minutes, 2-aminobenzenethiol (0.30 mmol) and CuI (0.02 mmol) were added into the reaction system. Upon completion determined by TLC, the reaction mixture was quenched with saturated $NaHCO_3$ (25 mL), and extracted by ethyl acetate (25 mL x 3). The organic layer was dried over Na_2SO_4, and concentrated under vacuum. The residue was purified by flash column chromatography on silica gel (hexanes/ethyl acetate = 20:1) to afford the pure product.



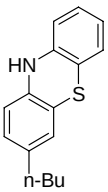
3-Methyl-10*H*-phenothiazine 23: ^{1}H NMR (400 MHz, DMSO-d6) δ 8.46 (s, 1 H), 6.96 (t, J = 7.6 Hz, 1 H), 6.89 (d, J = 7.5 Hz, 1 H), 6.78 (d, J = 7.9 Hz, 1 H), 6.70-6.73 (m, 2 H), 6.67 (d, J = 7.9 Hz, 1 H), 6.59 (d, J = 8.0 Hz, 1 H), 2.12 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 142.9, 140.0, 131.1, 128.4, 127.9, 127.0, 126.7, 121.9, 116.8, 116.7, 114.8, 20.4; HRMS m/z calcd for $C_{13}H_{12}NS$ ($[M+H]^+$): 214.0685, found 214.0669.



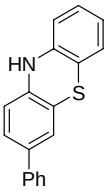
3-Ethyl-10*H*-phenothiazine 24: ^{1}H NMR (400 MHz, DMSO-d6) δ 8.47 (s, 1 H), 6.97 (dt, J = 7.7, 1.4 Hz, 1 H), 6.89 (dd, J = 7.6, 1.2 Hz, 1 H), 6.81 (dd, J = 8.1, 1.9 Hz, 1 H), 6.75 (dd, J = 1.7 Hz, 1 H), 6.72 (dt, J = 7.6, 1.2 Hz, 1 H), 6.67 (dd, J = 7.9, 1.1 Hz, 1 H), 6.61 (d, J = 8.0 Hz, 1 H), 2.39-2.44 (m, 2 H), 1.09 (t, J = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 142.9, 140.3, 137.8, 127.9, 127.3, 126.7, 125.9, 121.9, 116.8, 116.7, 114.8, 114.7, 27.7, 16.2; HRMS m/z calcd for $C_{14}H_{14}NS$ ($[M+H]^+$): 228.0841, found 228.0852.



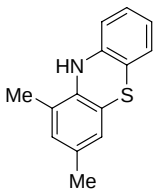
3-Isopropyl-10*H*-phenothiazine 25: ^{1}H NMR (400 MHz, DMSO-d6) δ 8.48 (s, 1 H), 6.96 (t, J = 7.6 Hz, 1 H), 6.89 (d, J = 7.6 Hz, 1 H), 6.85 (dd, J = 8.1, 1.8 Hz, 1 H), 6.77 (d, J = 1.7 Hz, 1 H), 6.72 (dd, J = 10.8, 4.2 Hz, 1 H), 6.67 (d, J = 7.3 Hz, 1 H), 6.62 (d, J = 8.1 Hz, 1 H), 2.70 (m, 1 H), 1.12 (s, 3 H), 1.10 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 142.9, 142.5, 140.4, 127.9, 126.7, 125.8, 124.4, 121.9, 116.8, 116.7, 114.8, 114.7, 33.0, 24.3; HRMS m/z calcd for $C_{15}H_{16}NS$ ($[M+H]^+$): 242.0998, found 242.0987.



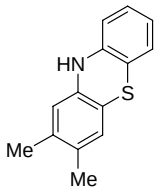
3-Butyl-10*H*-phenothiazine 26: ^{1}H NMR (400 MHz, DMSO-d6) δ 8.48 (s, 1 H), 6.96 (t, J = 7.6 Hz, 1 H), 6.89 (d, J = 6.9 Hz, 1 H), 6.79 (dd, J = 8.0, 1.3 Hz, 1 H), 6.72 (s, 2 H), 6.67 (d, J = 7.8 Hz, 1 H), 6.60 (d, J = 7.9 Hz, 1 H), 2.38 (s, 2 H), 1.49-1.39 (m, 2 H), 1.24 (m, 2 H), 0.86 (t, J = 7.3 Hz, 3 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 142.8, 140.3, 136.3, 127.9, 127.8, 126.7, 126.3, 121.9, 116.8, 116.7, 114.8, 34.3, 33.6, 22.1, 14.3; HRMS m/z calcd for $C_{16}H_{18}NS$ ($[M+H]^+$): 256.1154, found 256.1151.

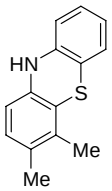


3-Phenyl-10*H*-phenothiazine 27: ^{1}H NMR (400 MHz, DMSO-d6) δ 8.72 (s, 1 H), 7.56 (d, J = 7.4 Hz, 2 H), 7.39 (t, J = 7.6 Hz, 2 H), 7.34-7.24 (m, 2 H), 7.22 (s, 1 H), 7.00 (t, J = 7.4 Hz, 1 H), 6.93 (d, J = 7.4 Hz, 1 H), 6.74-6.77 (m, 2 H), 6.70 (d, J = 7.8 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 142.2, 141.9, 139.6, 134.2, 129.3, 128.1, 127.3, 126.7, 126.3, 126.2, 124.6, 122.3, 117.5, 116.6, 115.2, 115.0; HRMS m/z calcd for $C_{18}H_{14}NS$ ($[M+H]^+$): 276.0841, found 276.0824.

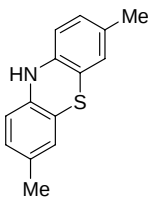


1,3-Dimethyl-10*H*-phenothiazine 28: ^{1}H NMR (400 MHz, DMSO-d6) δ 7.54 (s, 1 H), 7.02-6.94 (m, 2 H), 6.90 (d, J = 7.3 Hz, 1 H), 6.74 (dt, J = 7.7, 1.8 Hz, 1 H), 6.68 (s, 1 H), 6.60 (s, 1 H), 2.16 (s, 3 H), 2.09 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 142.9, 138.0, 130.8, 130.3, 127.7, 126.5, 124.9, 122.7, 122.2, 117.6, 116.9, 116.0, 20.3, 18.0; HRMS m/z calcd for $C_{14}H_{14}NS$ ($[M+H]^+$): 228.0841, found 228.0813.

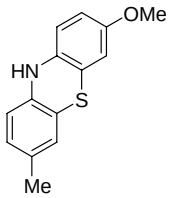




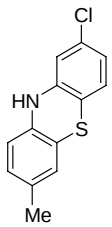
2,3-Dimethyl-10*H*-phenothiazine **a**nd **3,4-dimethyl-10*H*-phenothiazine 29:** ^{1}H NMR (400 MHz, DMSO-d6): **I**: δ 8.37 (s, 1 H, NH, peaks of two isomers overlapped), 6.95 (dt, J = 7.8, 1.3 Hz, 1 H, CHar, peaks of two isomers overlapped), 6.88 (d, J = 7.6 Hz, 1 H, CHar), 6.71 (d, J = 7.4 Hz, 1 H, CHar), 6.65-6.68 (m, 2 H, CHar, peaks of two isomers overlapped), 6.48 (s, 1 H, CHar), 2.07 (s, 3 H, CH), 2.04 (s, 3 H, CH); **II**: δ 8.37 (s, 1 H, NH, peaks of two isomers overlapped), 6.95 (dt, J = 7.8, 1.3 Hz, 2 H, CHar, peaks of two isomers overlapped), 6.76 (d, J = 8.0 Hz, 1 H, CHar), 6.65-6.68 (m, 2 H, CHar, peaks of two isomers overlapped), 6.44 (d, J = 7.9 Hz, 1H, CHar), (s, 6 H, CH); HRMS m/z calcd for $C_{14}H_{14}NS$ ($[M+H]^+$): 228.0841, found 228.0832.



3,7-dimethyl-10*H*-phenothiazine 30: gray oil; ^{1}H NMR (400 MHz, DMSO-d6) δ 8.31 (s, 1 H), 6.76 (d, J = 8.1 Hz, 2 H), 6.72 (s, 2 H), 6.56 (d, J = 8.0 Hz, 2 H), 2.11 (s, 6 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 140.3, 130.8, 128.3, 126.9, 116.6, 114.6, 20.4; HRMS m/z calcd for $C_{14}H_{14}NS$ ($[M+H]^+$): 228.0841, found 228.0816.

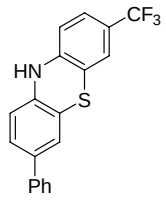


3-Methoxy-7-methyl-10*H*-phenothiazine 31: ^{1}H NMR (400 MHz, DMSO-d6) δ 8.23 (s, 1 H), 6.77 (dd, J = 8.0, 1.2 Hz, 1 H), 6.73 (s, 1H), 6.61 (s, 1 H), 6.60 (d, J = 2.6 Hz, 1 H), 6.57-6.56 (m, 2 H), 3.64 (s, 3 H), 2.12 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 154.9, 140.7, 136.4, 130.6, 128.4, 126.9, 118.0, 116.1, 115.4, 114.6, 113.5, 112.1, 55.8, 20.4; HRMS m/z calcd for $C_{14}H_{14}NOS$ ($[M+H]^+$): 244.0791, found 244.0779.

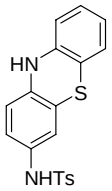


2-Chloro-7-methyl-10*H*-phenothiazine 32: pale yellow solid; m.p. 229-231 °C; ^{1}H NMR (400 MHz, DMSO-d6) δ 8.64 (s, 1 H), 6.89 (d, J = 8.2 Hz, 1 H), 6.80 (d, J = 8.0 Hz, 1 H), 6.75-6.73 (m, 2 H), 6.67 (d, J

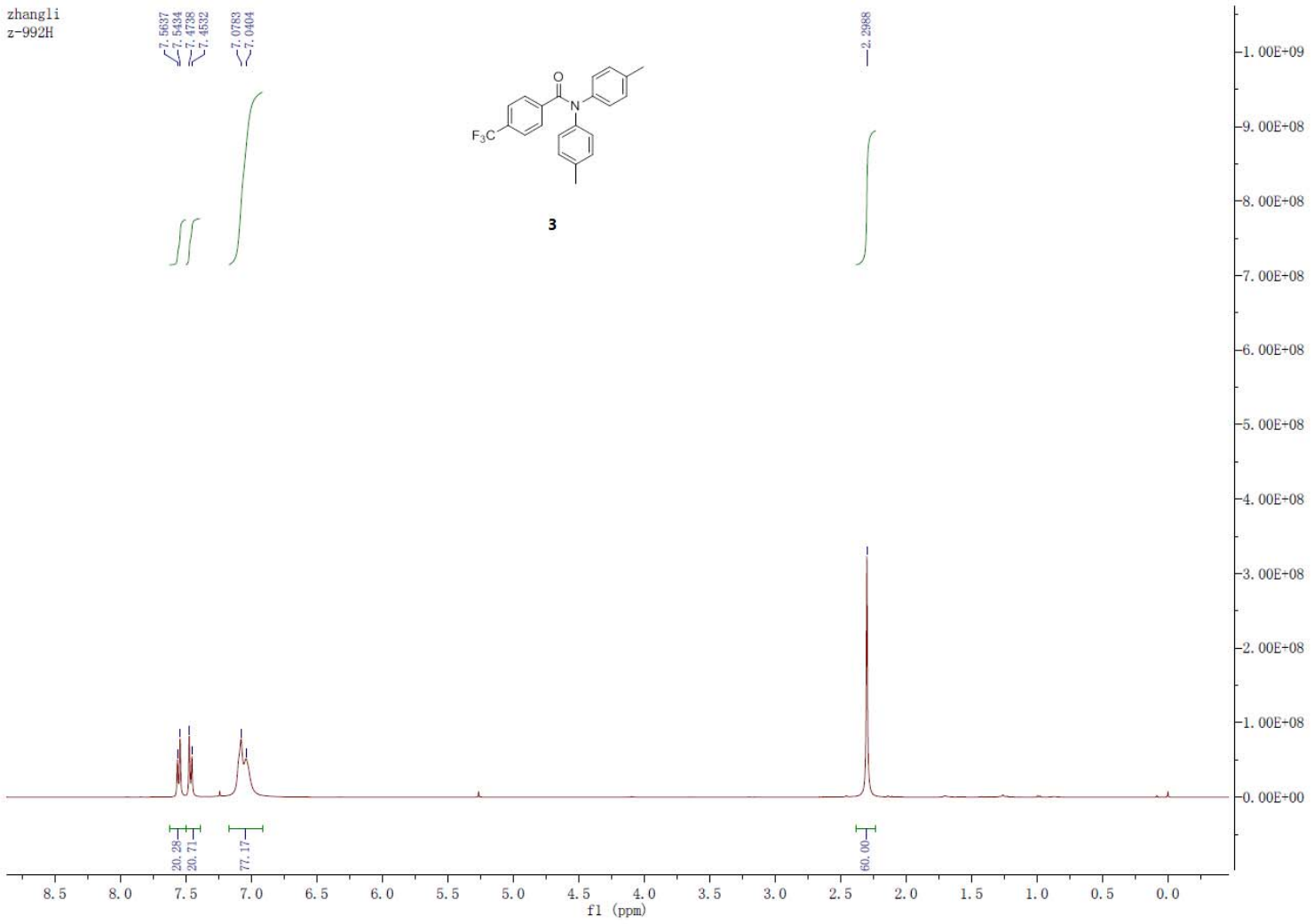
= 2.1 Hz, 1 H), 6.56 (d, *J* = 8.0 Hz, 1 H), 2.12 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 144.2, 139.0, 132.2, 131.8, 128.6, 127.9, 127.0, 121.3, 116.4, 115.9, 115.0, 114.0, 20.4; HRMS m/z calcd for $C_{13}H_{11}ClNS$ ($[M+H]^+$): 248.0295, found 248.0264.



3-Phenyl-7-(trifluoromethyl)-10*H*-phenothiazine 33: pale yellow solid; m.p. 226-228 °C; ^{1}H NMR (400 MHz, DMSO-d6) δ 8.99 (s, 1 H), 7.56 (d, *J* = 7.5 Hz, 2 H), 7.39 (t, *J* = 7.6 Hz, 2 H), 7.35-7.25 (m, 2 H), 7.23 (d, *J* = 1.6 Hz, 1 H), 7.11 (d, *J* = 8.0 Hz, 1 H), 7.03 (d, *J* = 8.0 Hz, 1 H), 6.92 (s, 1 H), 6.73 (d, *J* = 8.2 Hz, 1 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 142.7, 140.6, 139.4, 135.0, 129.3, 128.6 (*J* = 31.8 Hz), 127.4 (*J* = 6.9 Hz), 126.8, 126.3, 124.8, 124.5 (*J* = 271.8 Hz), 122.3, 118.5 (*J* = 3.4 Hz), 116.6, 115.5, 110.6 (*J* = 3.4 Hz); HRMS m/z calcd for $C_{19}H_{13}F_3NS$ ($[M+H]^+$): 344.0715, found 344.0689.



4-Methyl-*N*-(10*H*-phenothiazin-3-yl)benzenesulfonamide 35: black solid; m.p. 168-170 °C; ^{1}H NMR (400 MHz, DMSO-d6)δ 9.82 (s, 1 H), 8.53 (s, 1 H), 7.58 (d, *J* = 8.2 Hz, 2 H), 7.33 (d, *J* = 8.1 Hz, 2 H), 6.96 (t, *J* = 7.6 Hz, 1 H), 6.87 (d, *J* = 7.3 Hz, 1 H), 6.68-6.74 (m, 2 H), 6.64 (d, *J* = 7.8 Hz, 1 H), 6.60 (d, *J* = 2.1 Hz, 1 H), 6.53 (d, *J* = 8.5 Hz, 1 H), 2.33 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d6) δ 143.6, 142.5, 139.6, 137.0, 132.1, 130.1, 128.2, 127.2, 126.7, 122.3, 121.5, 119.9, 117.4, 116.0, 115.1, 114.9, 21.5; HRMS m/z calcd for $C_{19}H_{17}N_2O_2S_2$ ($[M+H]^+$): 369.0726, found 369.0692.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1.00E+08
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7.0
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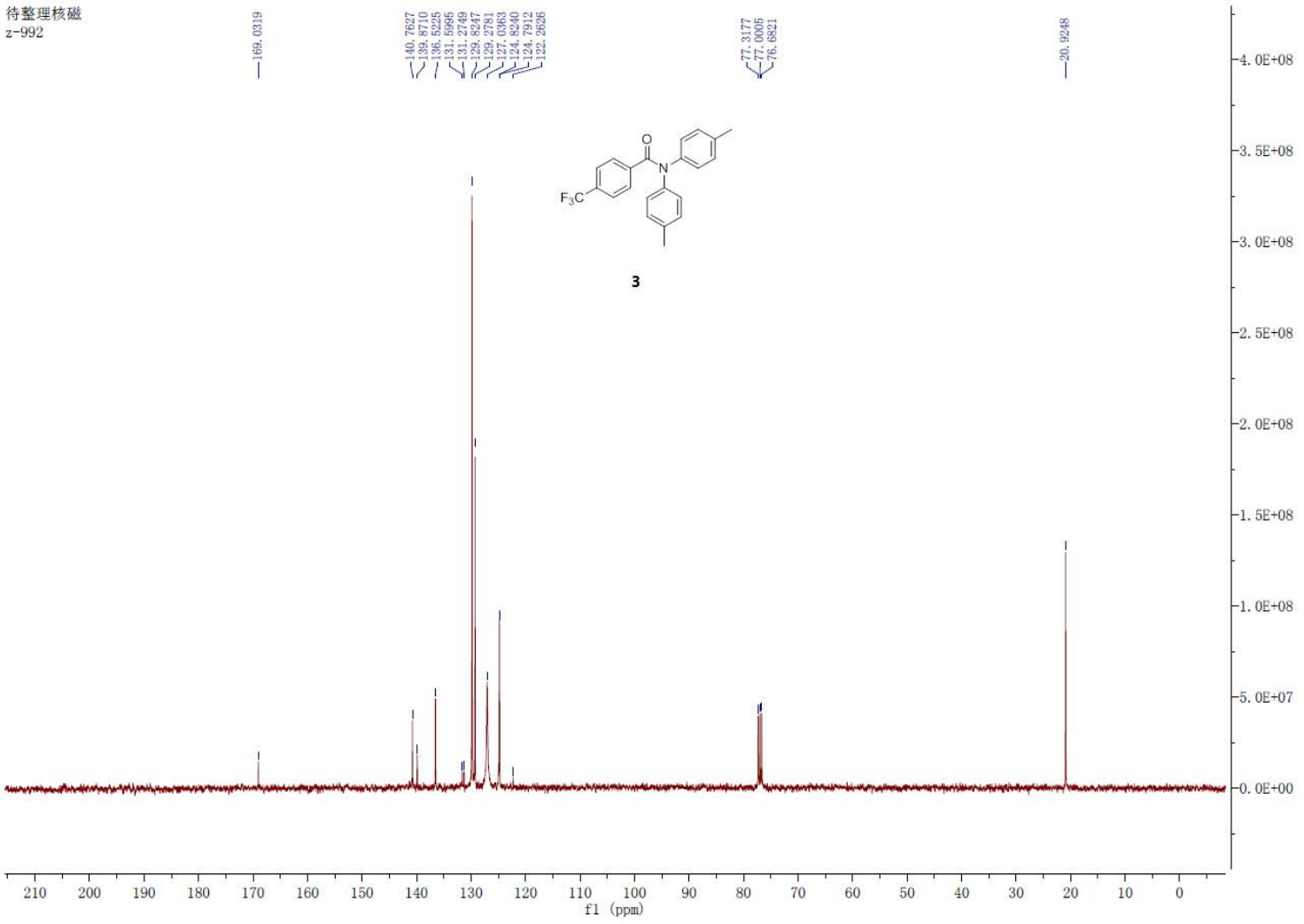
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77.3177
77.0005
76.6821
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O
N
F3C
3
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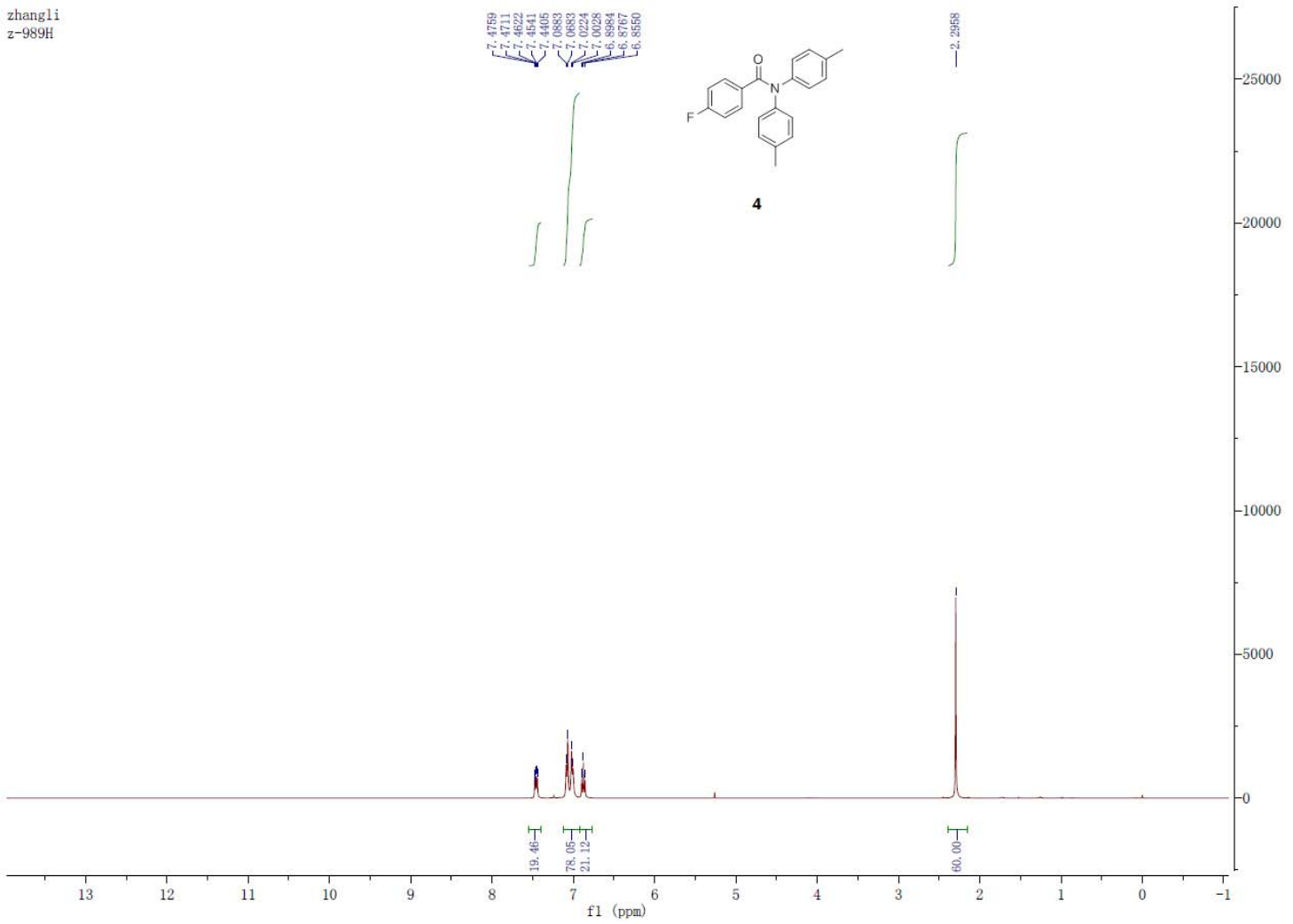
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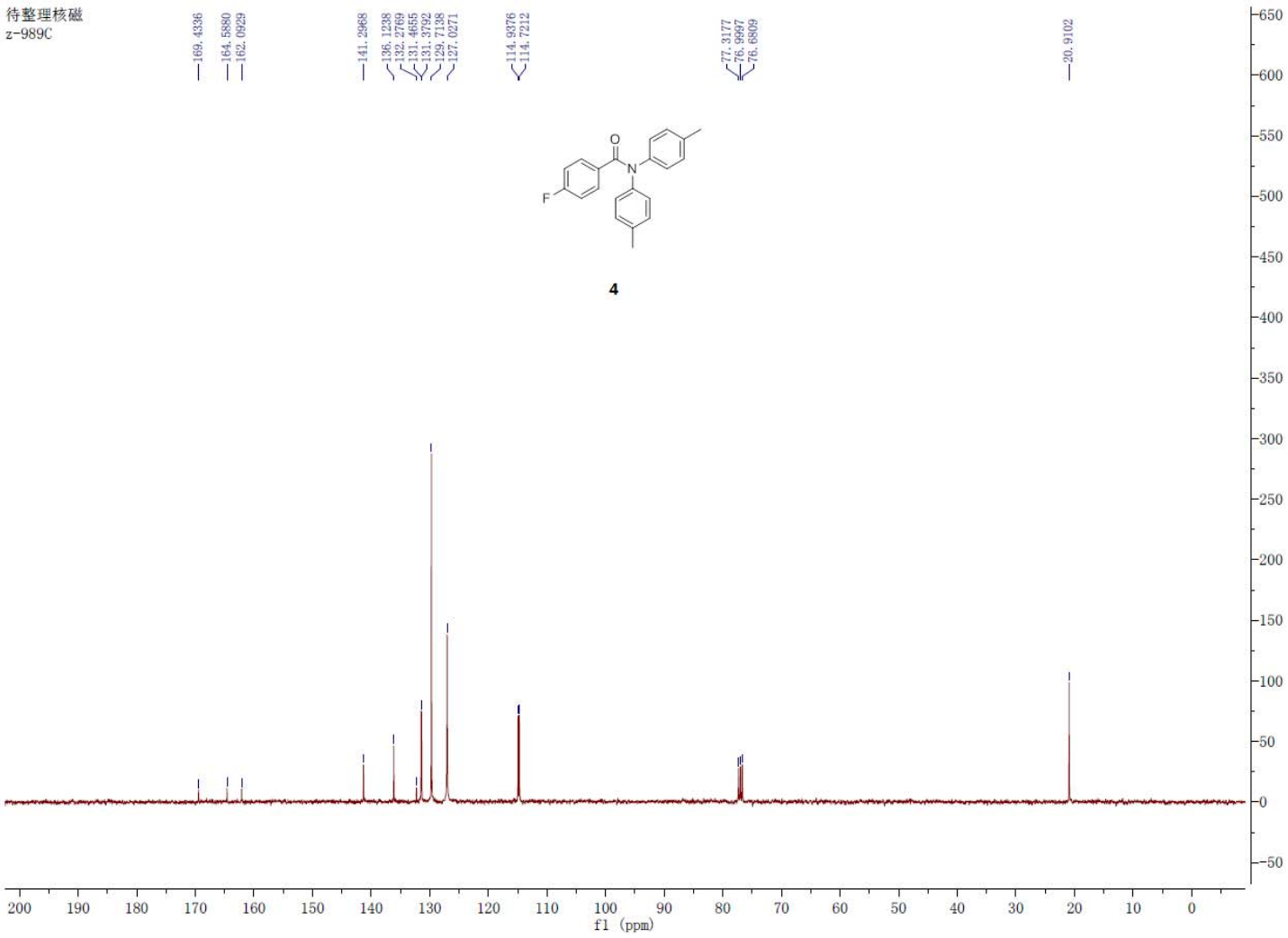
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O
N
F
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600
550
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450
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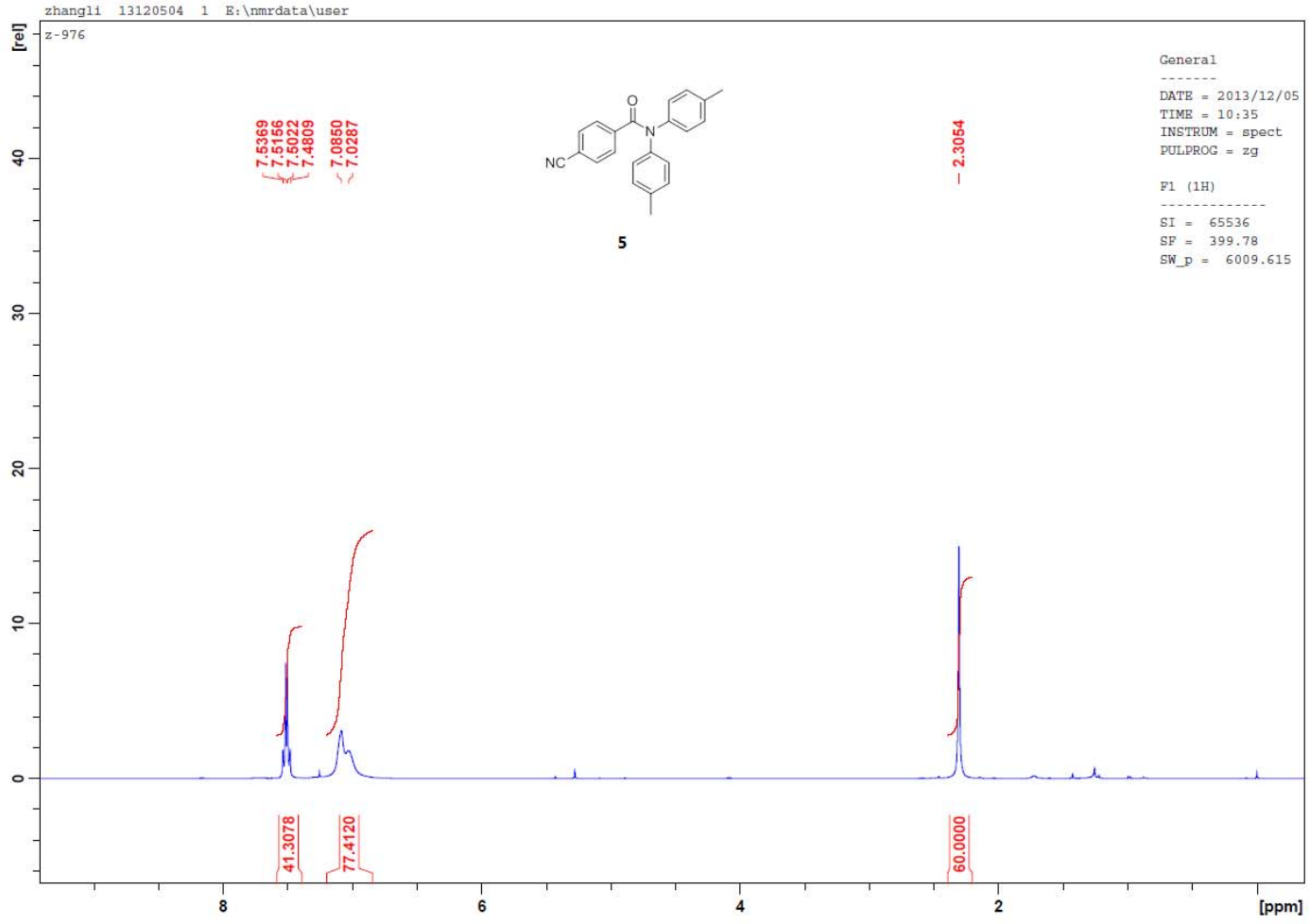
f1 (ppm)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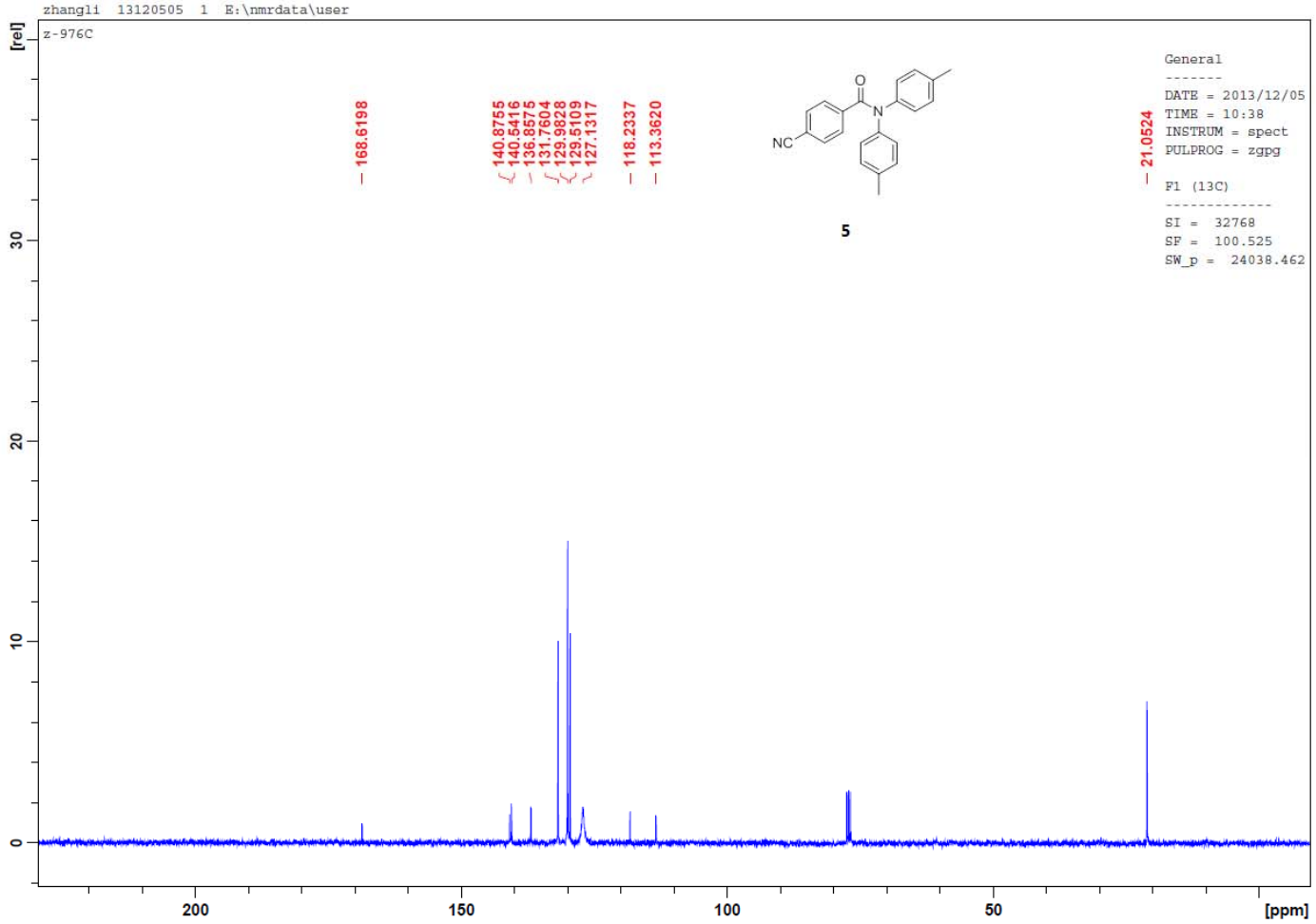
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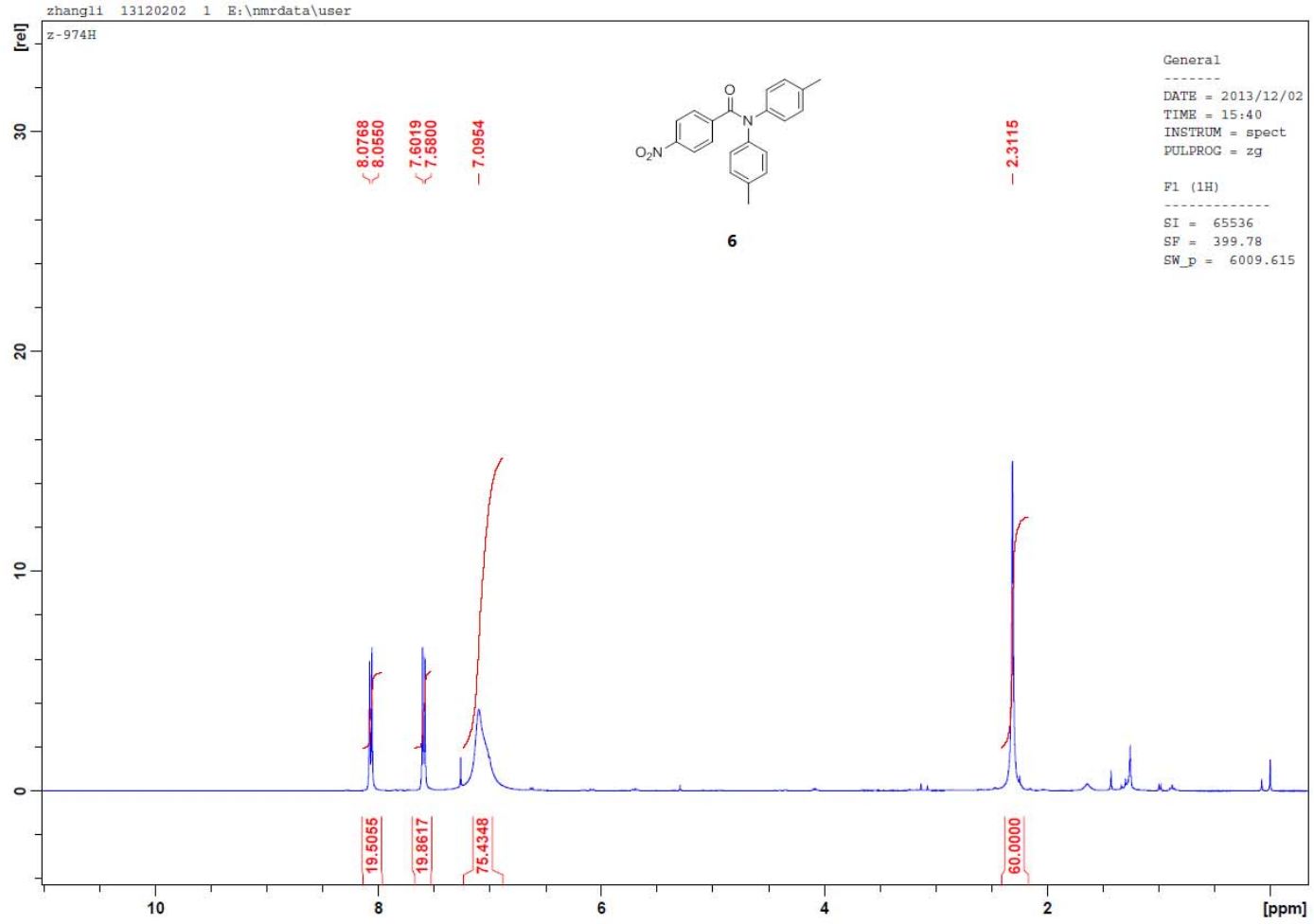
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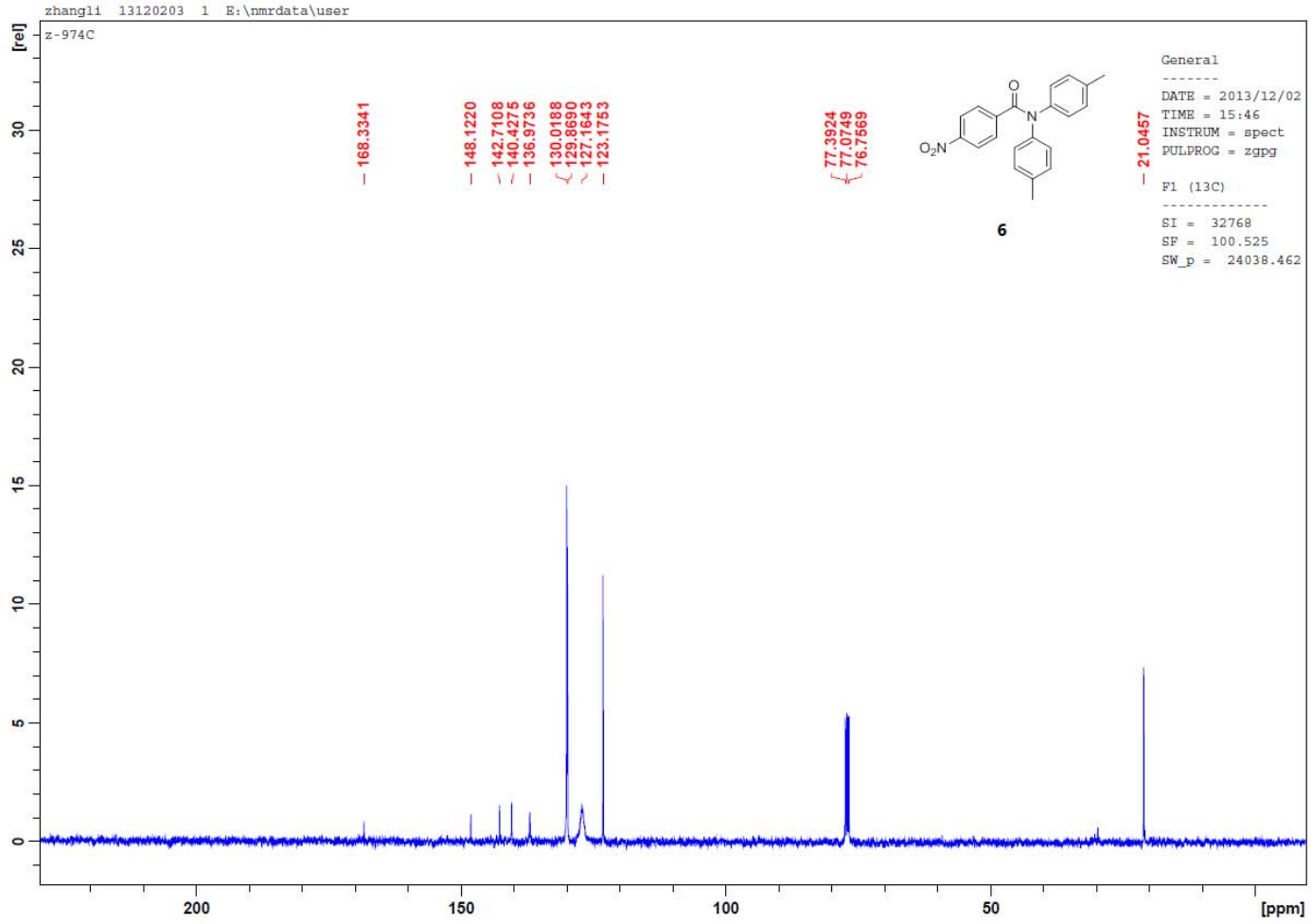
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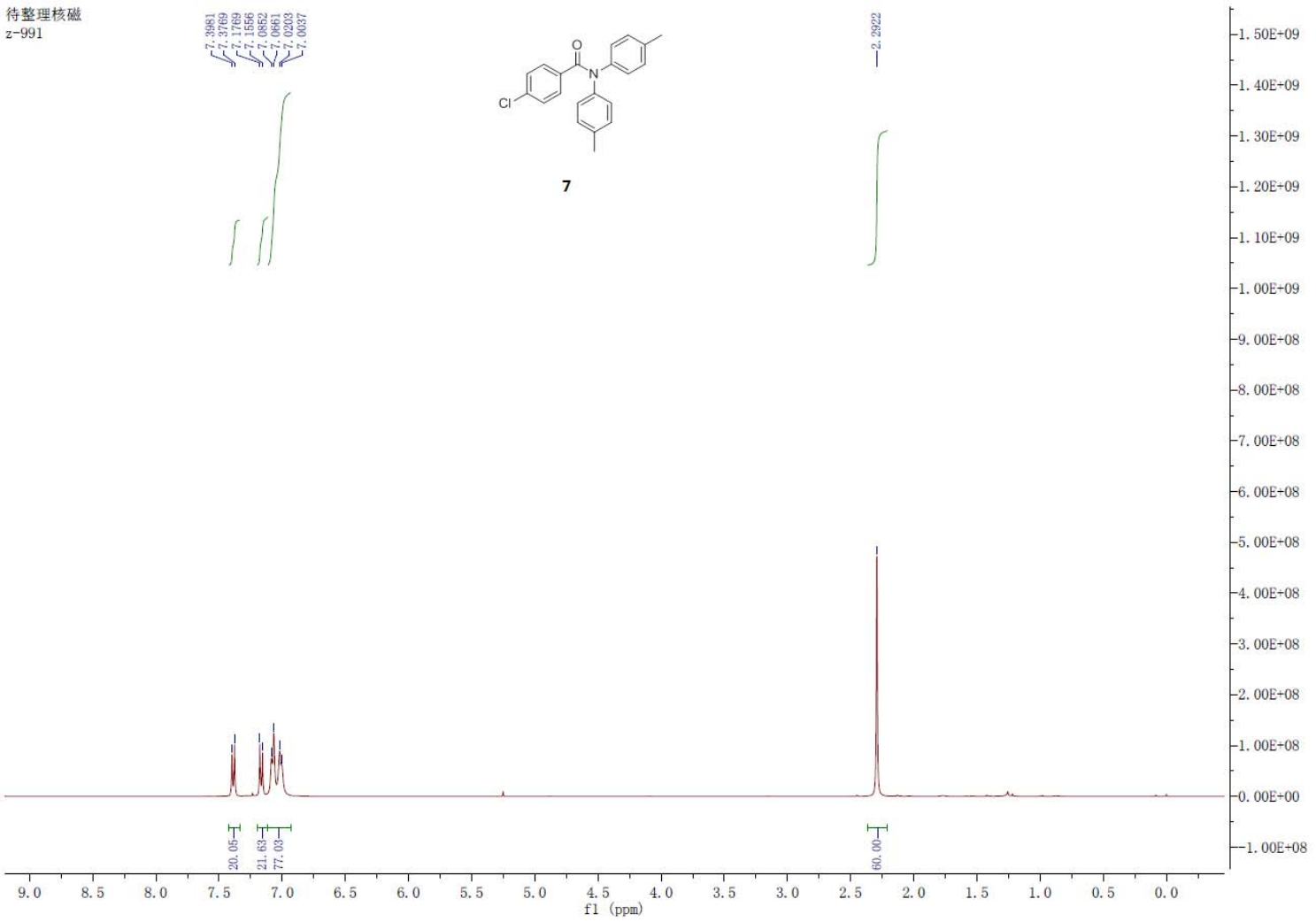
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5.00E+08
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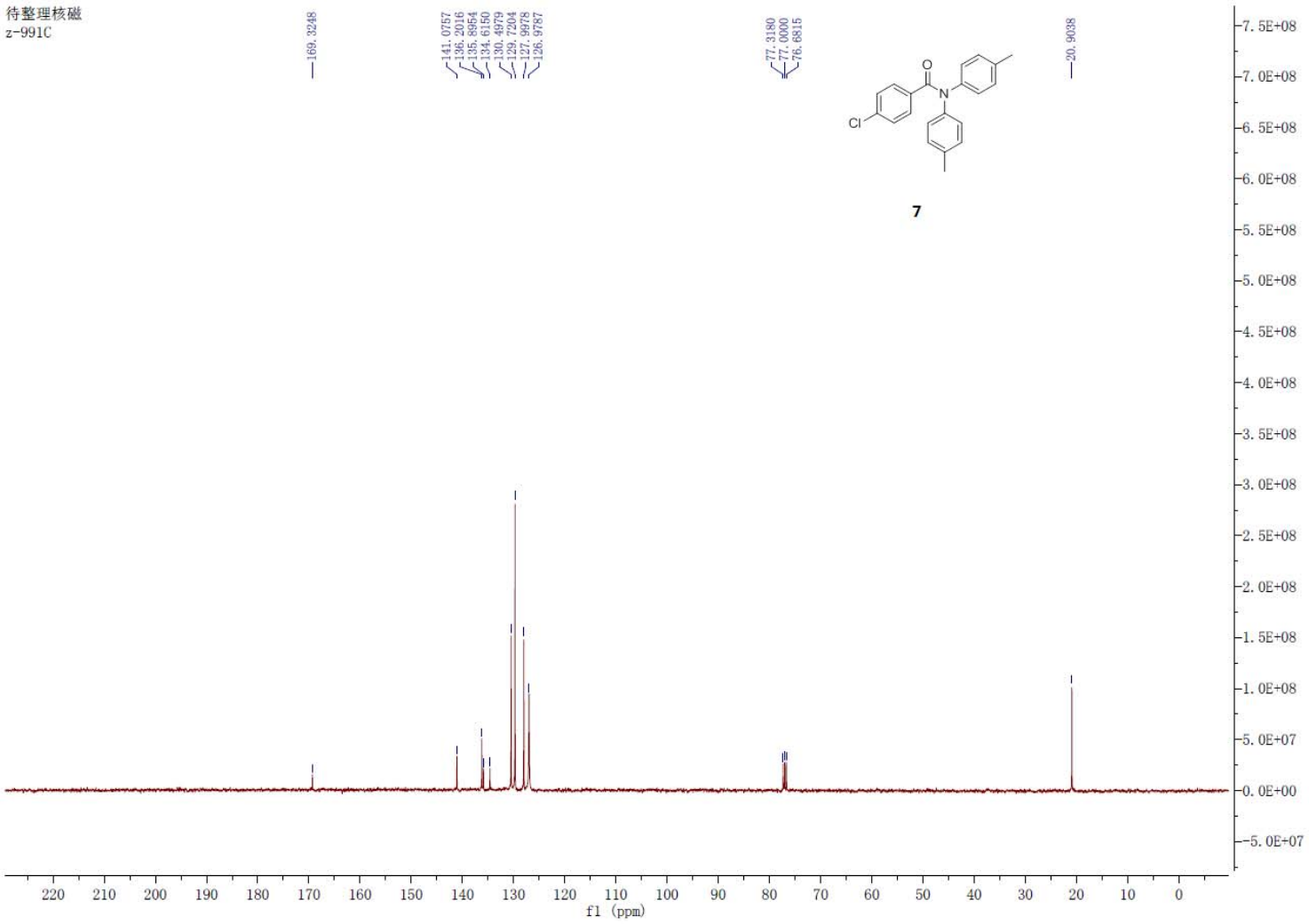
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N
Cl
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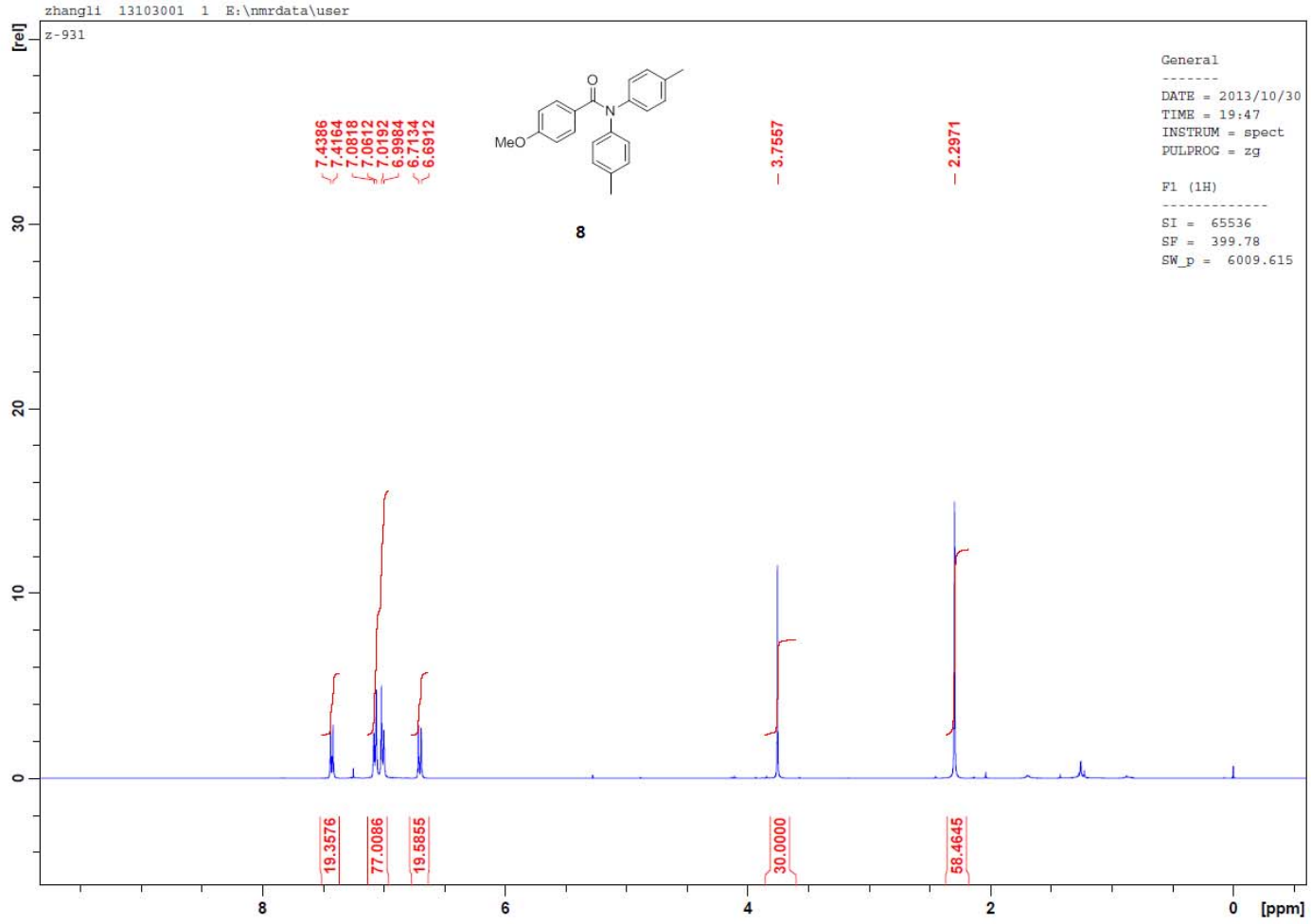
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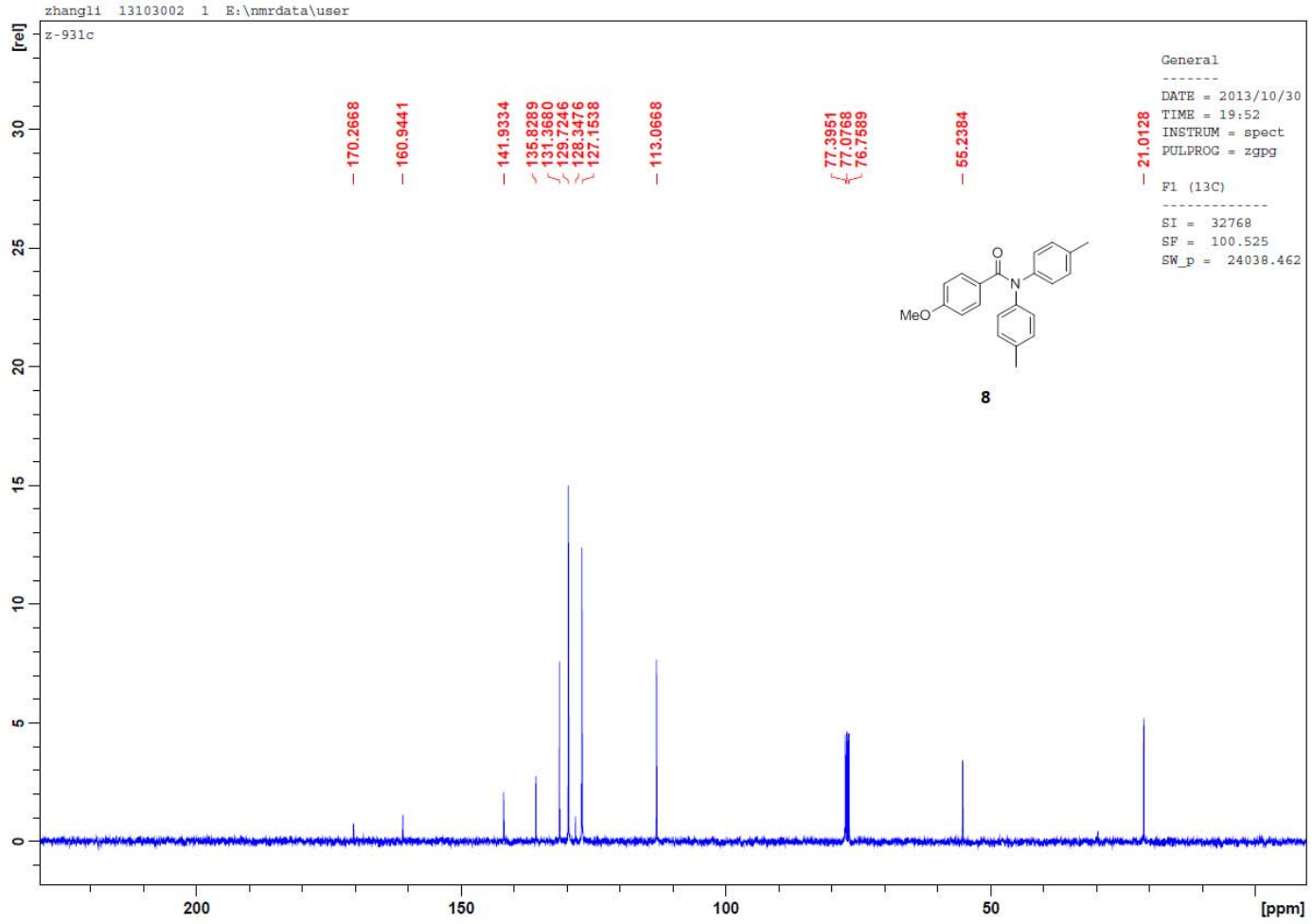
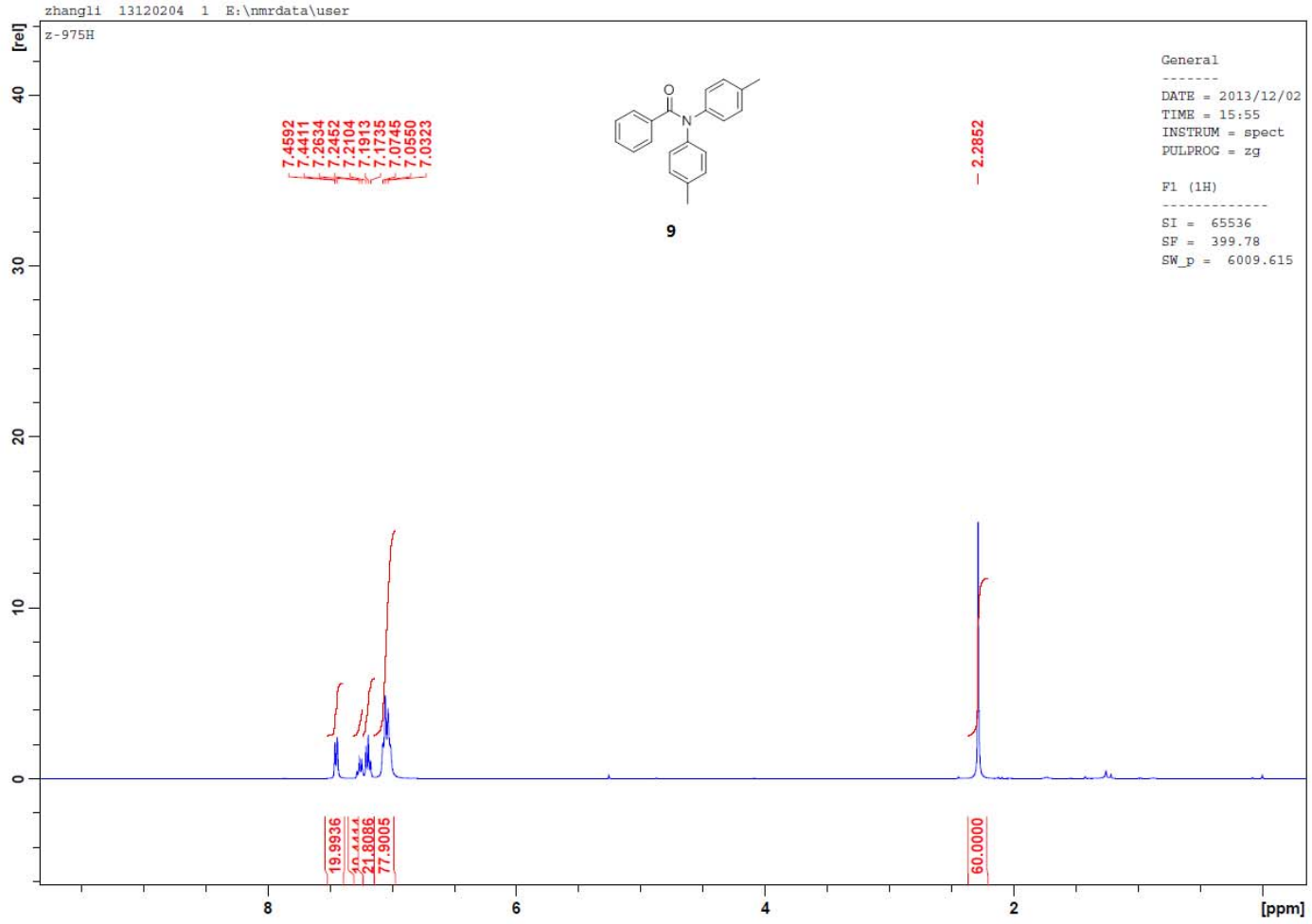
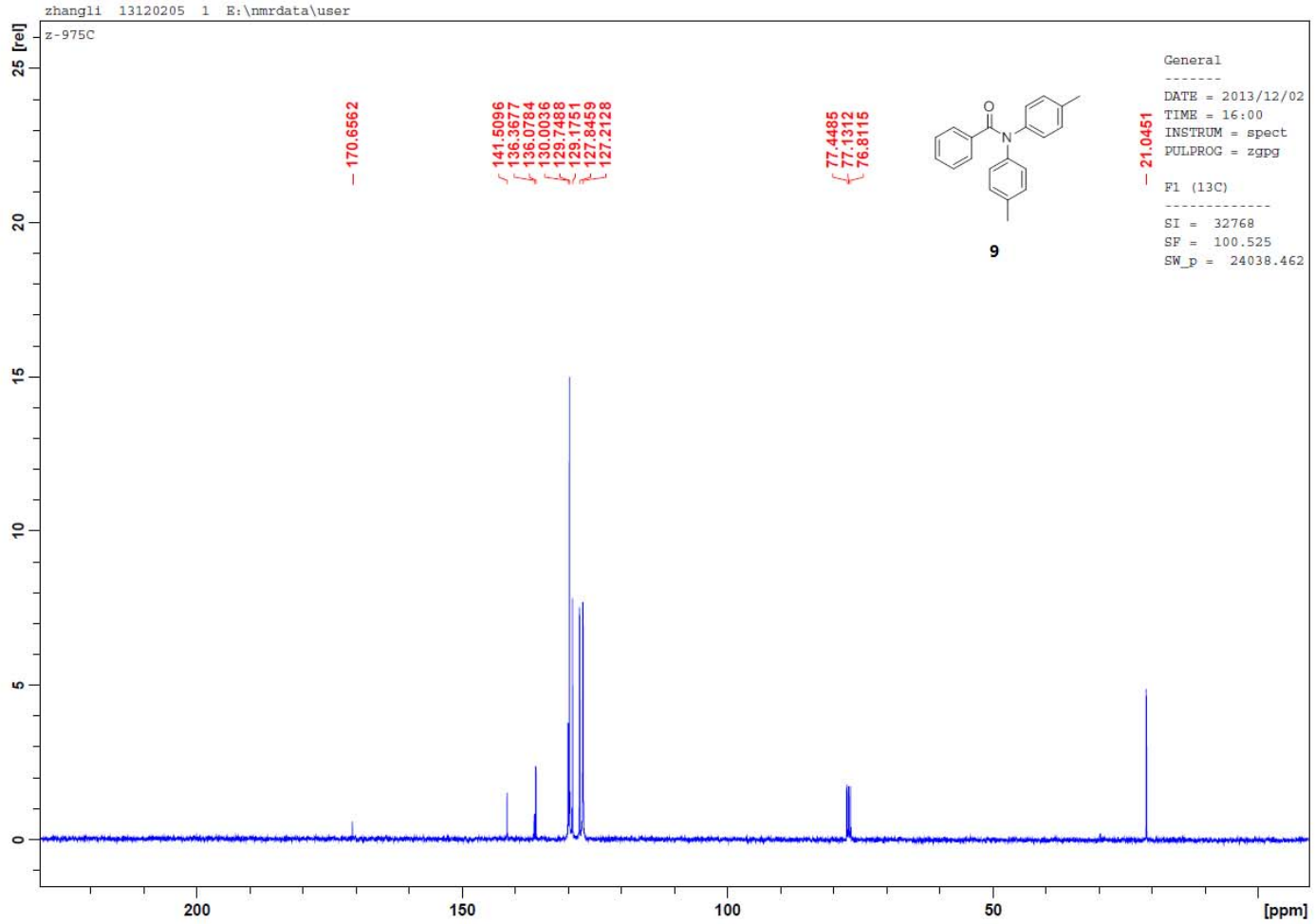
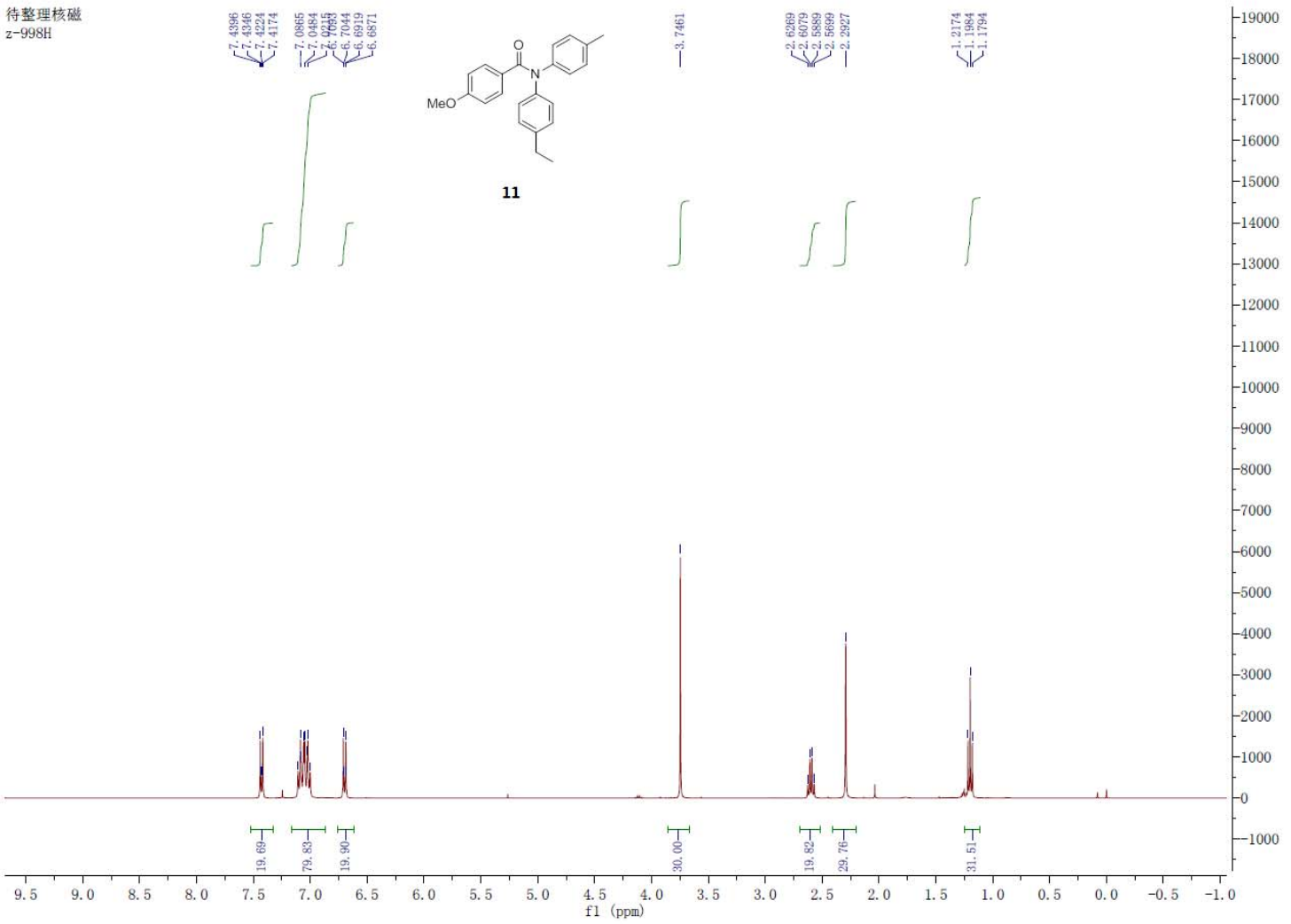
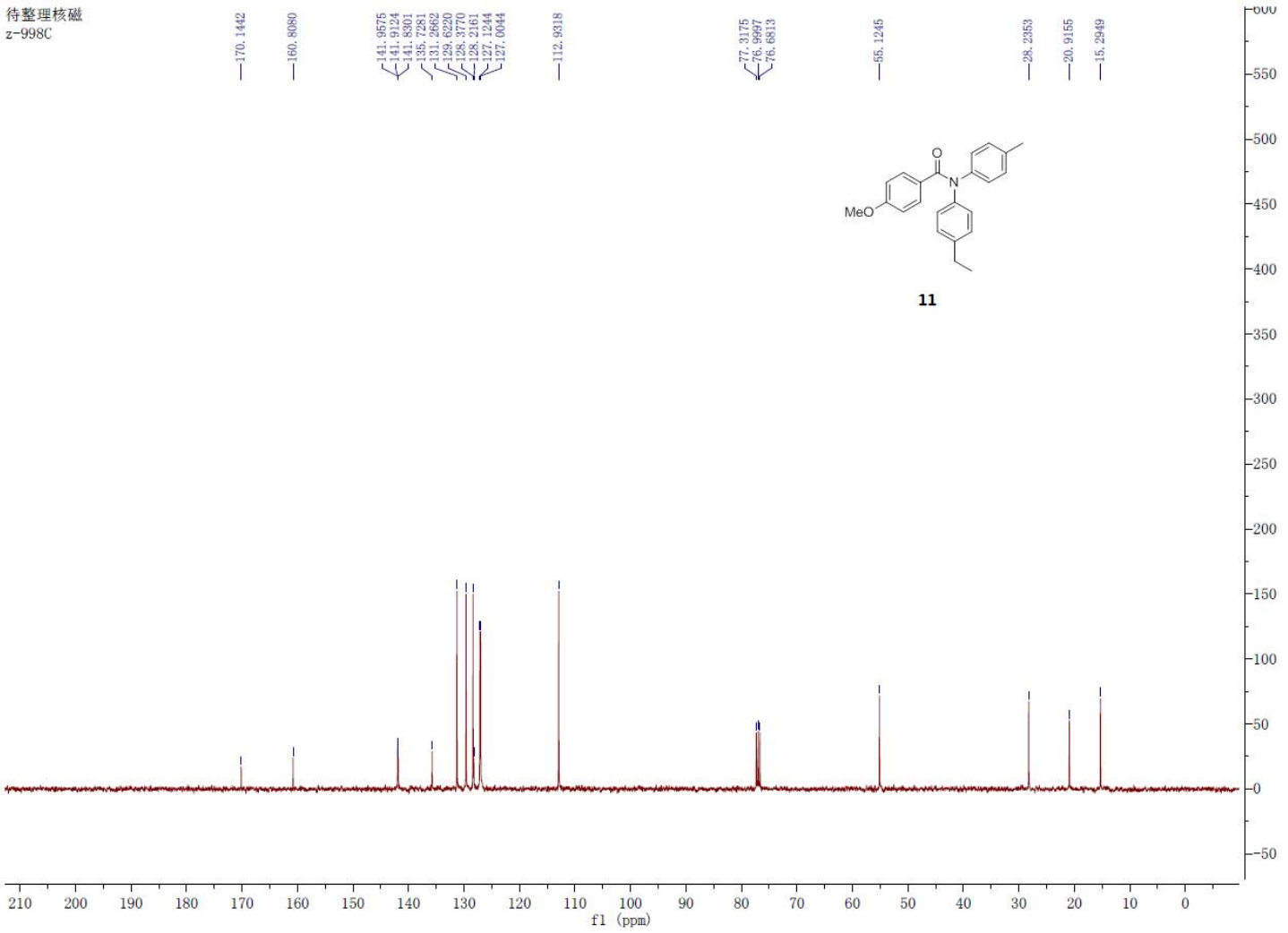
z-931
[rel]
7.4386
7.4164
7.0818
7.0612
7.0192
6.9984
6.7134
6.6912
3.7557
2.2971
MeO
O
N
8
General
DATE = 2013/10/30
TIME = 19:47
INSTRUM = spect
PULPROG = zg
F1 (1H)
SI = 65536
SF = 399.78
SW_p = 6009.615
19.3576
77.0086
19.5855
30.0000
58.4645
[ppm]

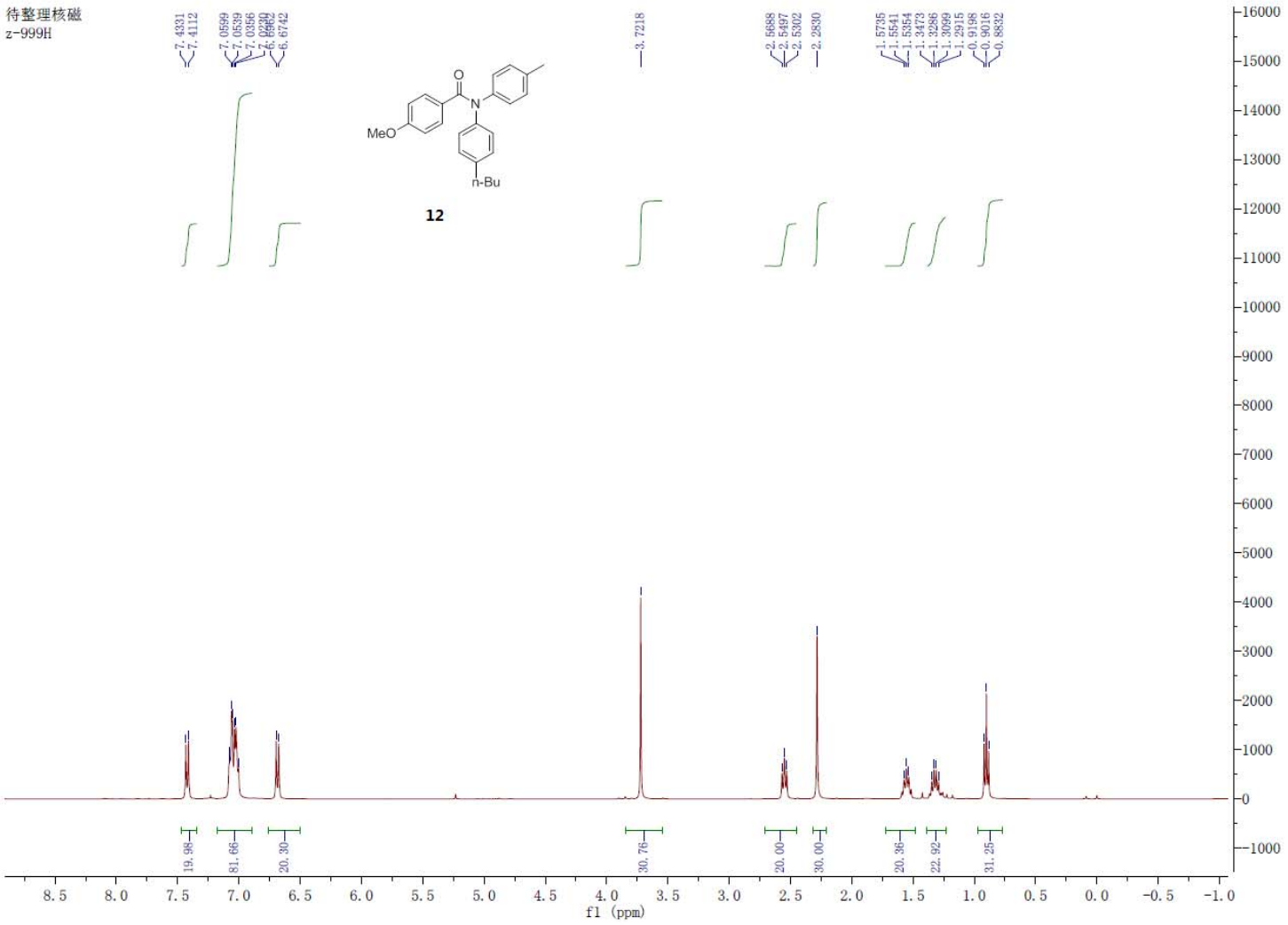

zhangli 13103002 1 E:\nmrdata\user
z-931c
[rel]
170.2668
160.9441
141.9334
135.8289
131.3680
129.7246
128.3476
127.1538
113.0668
77.3951
77.0768
76.7589
55.2384
21.0128
General
DATE = 2013/10/30
TIME = 19:52
INSTRUM = spect
PULPROG = zgpg
F1 (13C)
SI = 32768
SF = 100.525
SW_p = 24038.462
MeO
O
N
8
[ppm]

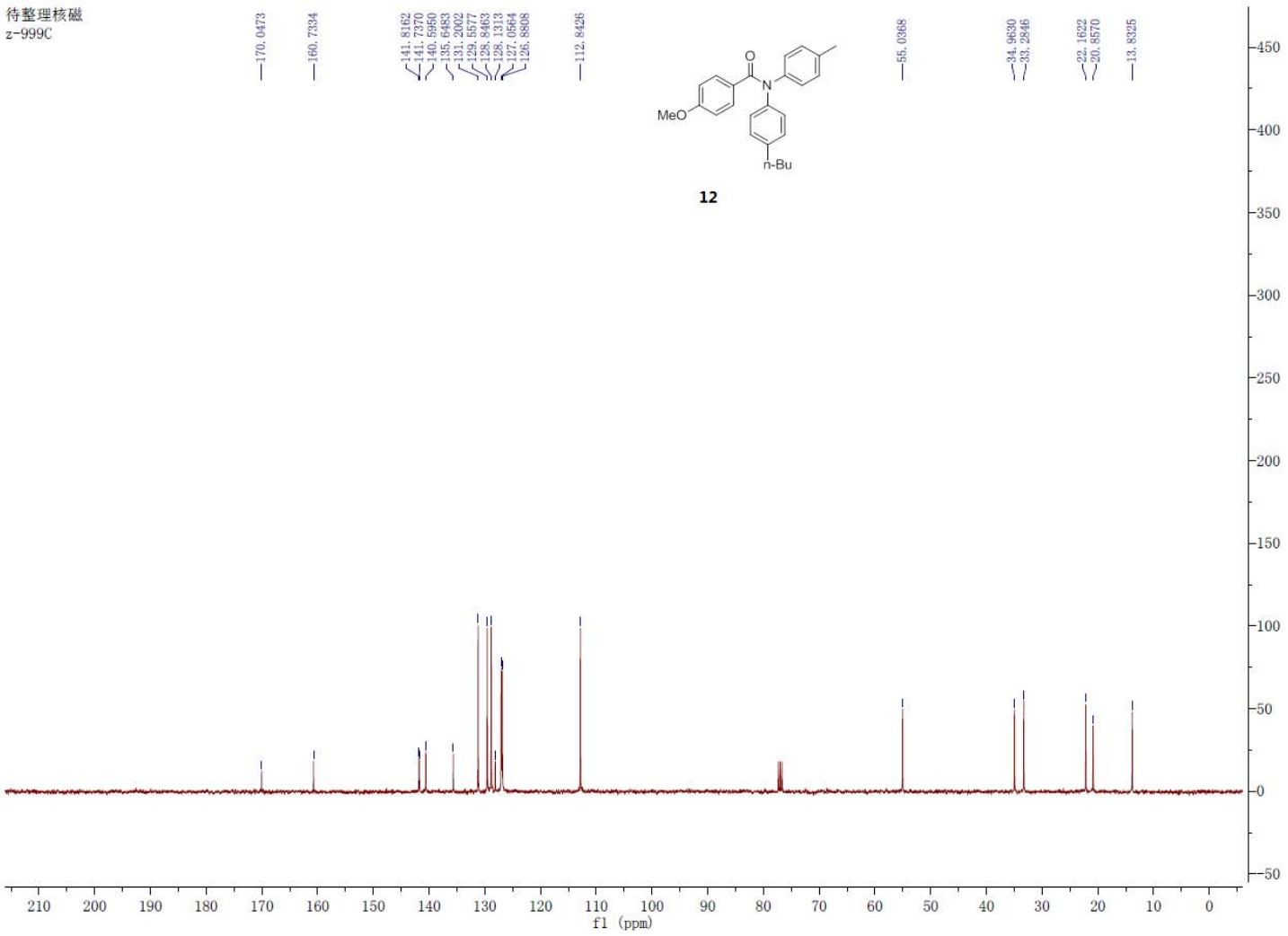

zhangli 13120204 1 E:\nmrdata\user
z-975H
[rel]
7.4592
7.4411
7.2634
7.2452
7.2104
7.1913
7.1735
7.0745
7.0550
7.0323
2.2852
9
General
DATE = 2013/12/02
TIME = 15:55
INSTRUM = spect
PULPROG = zg
F1 (1H)
SI = 65536
SF = 399.78
SW_p = 6009.615
19.9936
60.0000
[ppm]

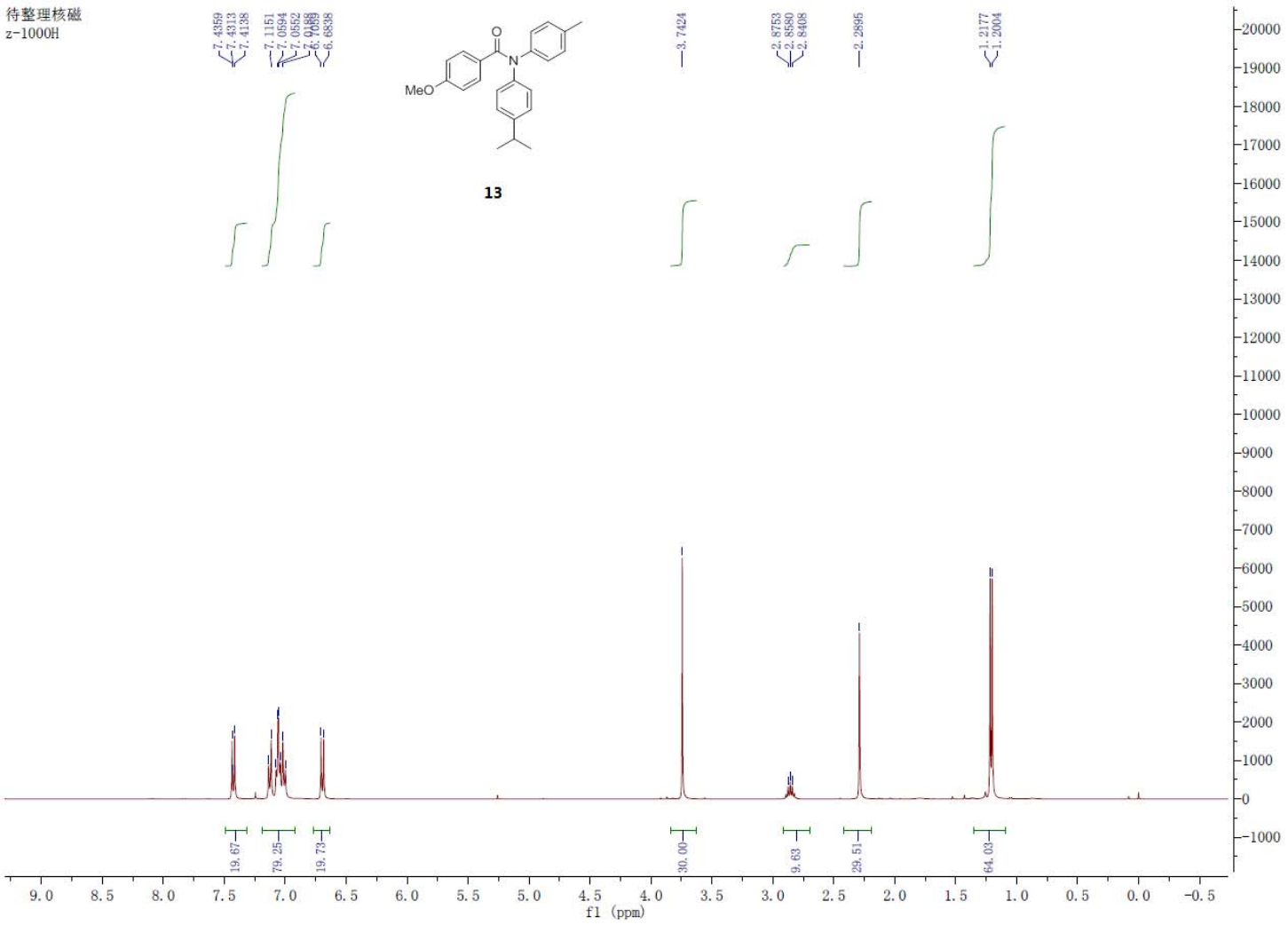

zhangli 13120205 1 E:\nmrdata\user
z-975C
170.6562
141.5096
136.3677
136.0784
130.0036
129.7488
129.1751
127.8459
127.2128
77.4485
77.1312
76.8115
21.0451
9
General
DATE = 2013/12/02
TIME = 16:00
INSTRUM = spect
PULPROG = zgpg
F1 (13C)
SI = 32768
SF = 100.525
SW_p = 24038.462
25 [rel]
20
15
10
5
0
200
150
100
50
[ppm]

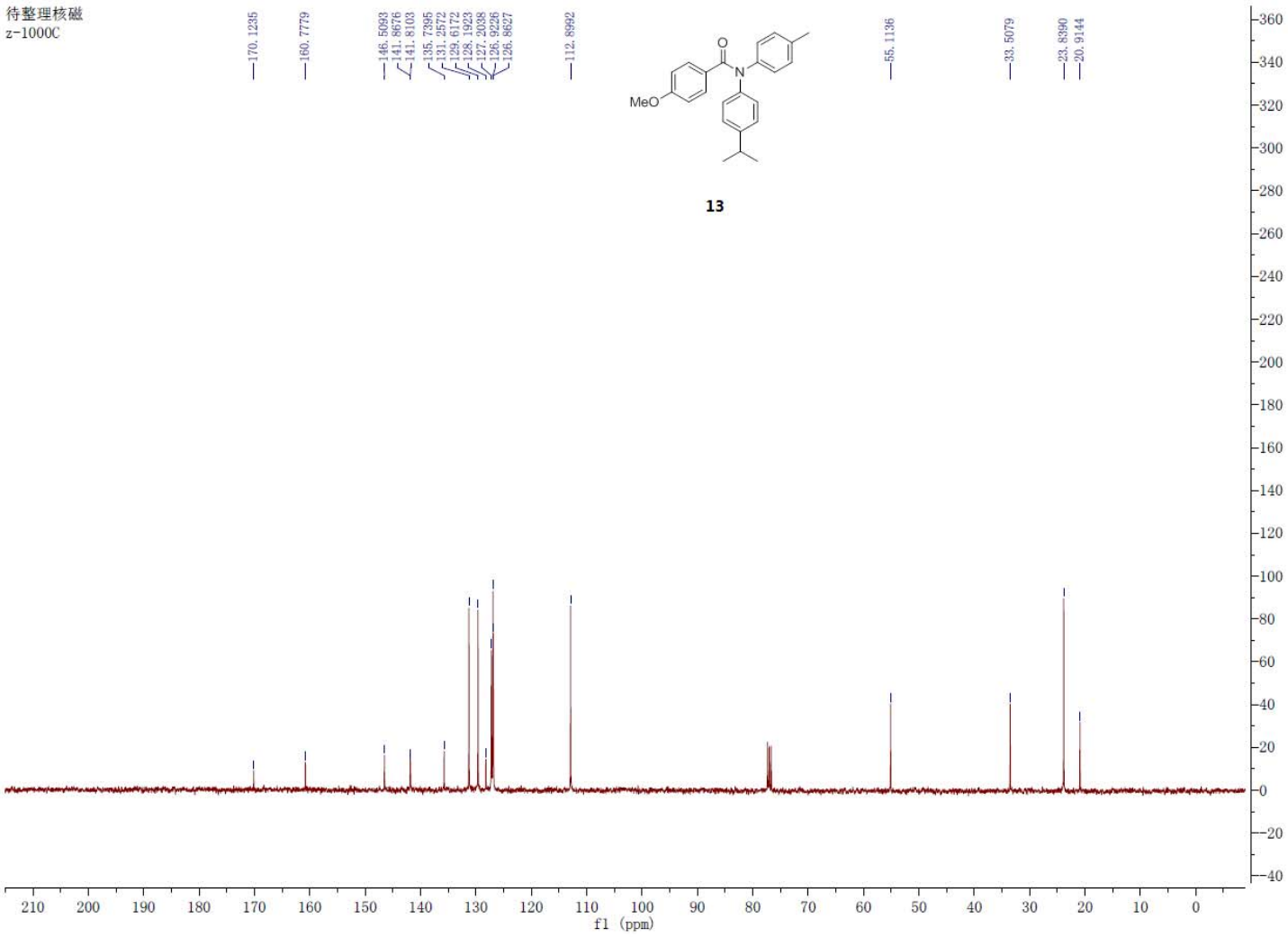

待整理核磁
z-998H
7.4396
7.4346
7.4224
7.4174
7.0865
7.0484
6.7044
6.6919
6.6871
3.7461
2.6269
2.6079
2.5889
2.5699
2.2927
1.2174
1.1984
1.1794
MeO
O
N
11
19.69
79.83
19.90
30.00
19.82
29.76
31.51
f1 (ppm)


待整理核磁
z-998C
170.1442
160.8080
141.9575
141.9124
141.8301
135.7281
131.2662
129.6220
128.3770
128.2161
127.1244
127.0044
112.9318
77.3175
76.9997
76.6813
55.1245
28.2353
20.9156
15.2949
MeO
O
N
11
f1 (ppm)

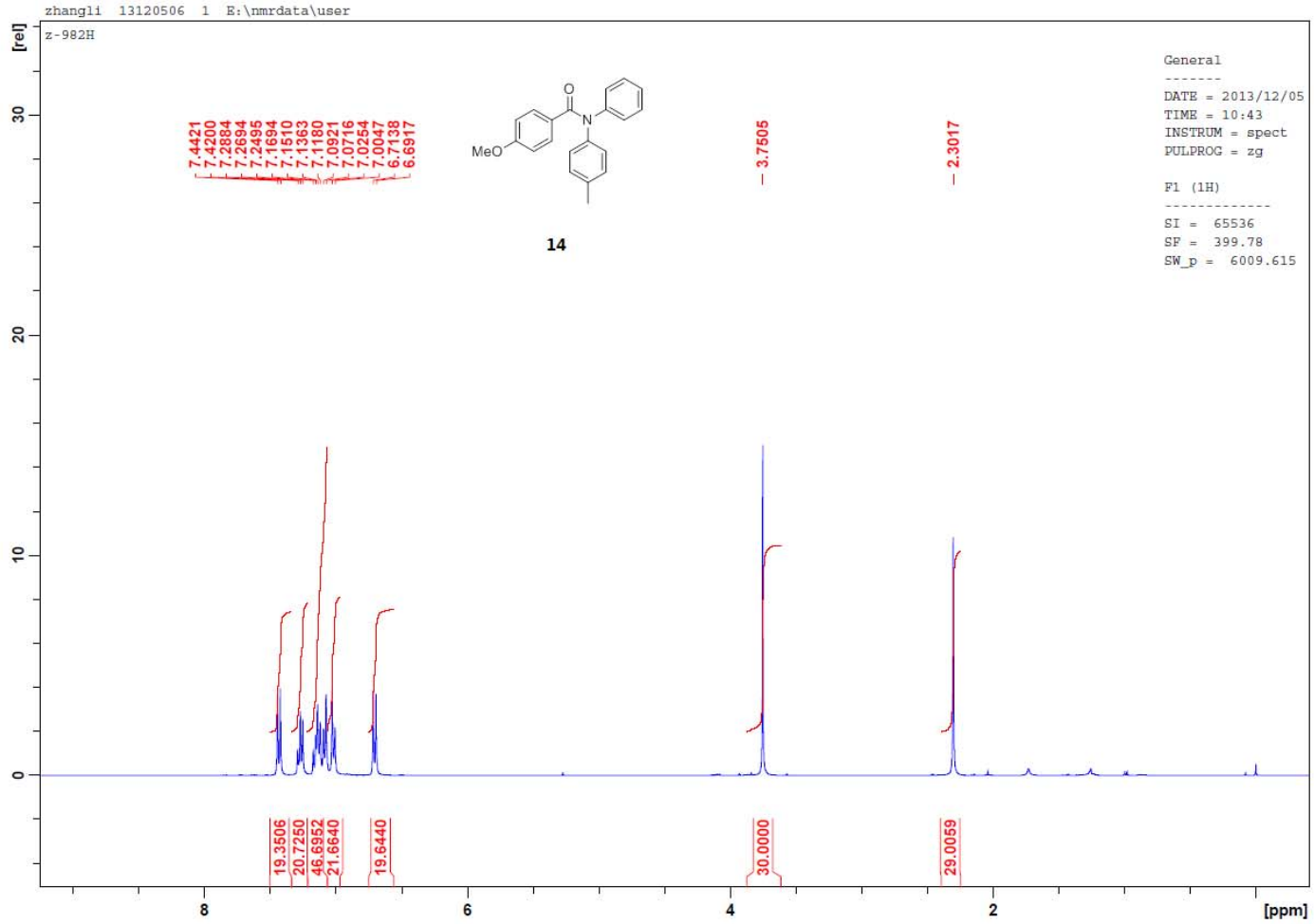

待整理核磁
z-999H
7.4331
7.4112
7.0599
7.0539
7.0356
6.6742
3.7218
2.5688
2.5497
2.5302
2.2830
1.5735
1.5541
1.5354
1.3473
1.3286
1.3099
1.2915
0.9198
0.9016
0.8832
MeO
N
O
n-Bu
12
19.98
81.66
20.30
30.76
20.00
30.00
20.36
22.92
31.25
f1 (ppm)

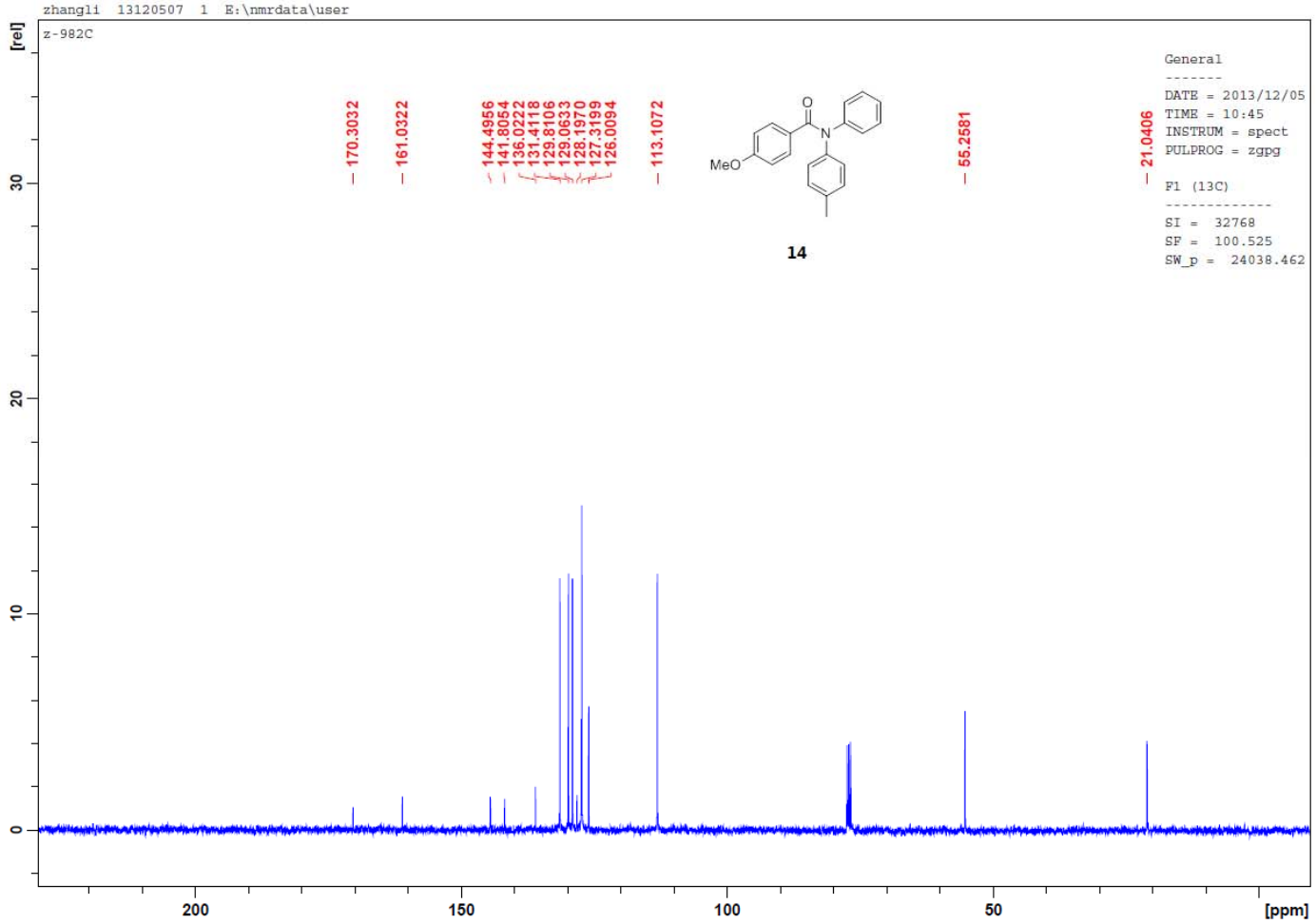

待整理核磁
z-999C
170.0473
160.7334
141.8162
141.7370
140.5950
135.6483
131.2002
129.5577
128.8463
128.1313
127.0564
126.8808
112.8426
55.0368
34.9630
33.2946
22.1622
20.8570
13.8325
MeO
N
O
n-Bu
12
f1 (ppm)

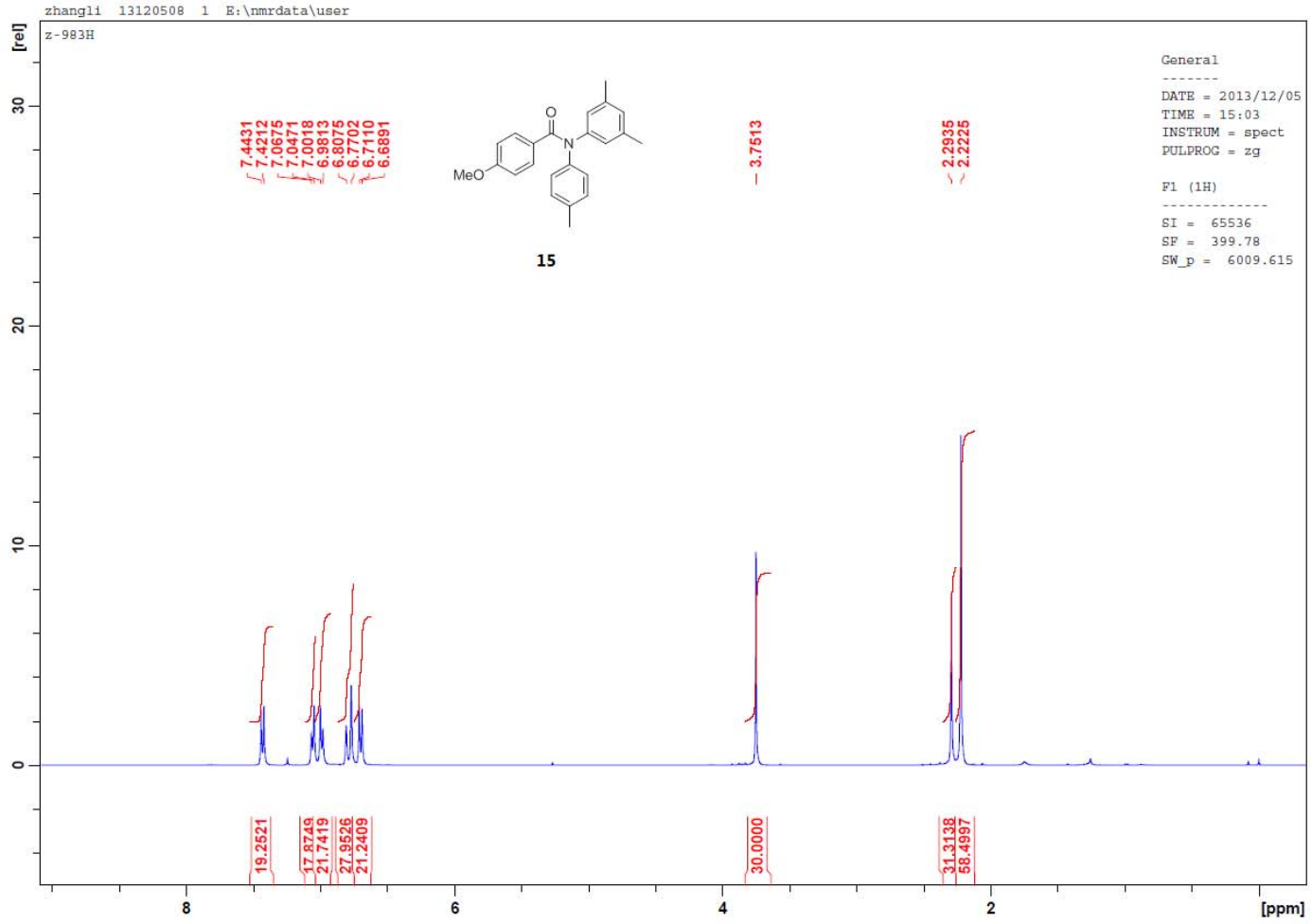

待整理核磁
z-1000H
7.4359
7.4313
7.4138
7.1151
7.0594
7.0552
7.0188
6.7089
6.6838
3.7424
2.8753
2.8580
2.8408
2.2895
1.2177
1.2004
MeO
O
N
13
19.67
79.25
19.73
30.00
9.63
29.51
64.03
f1 (ppm)

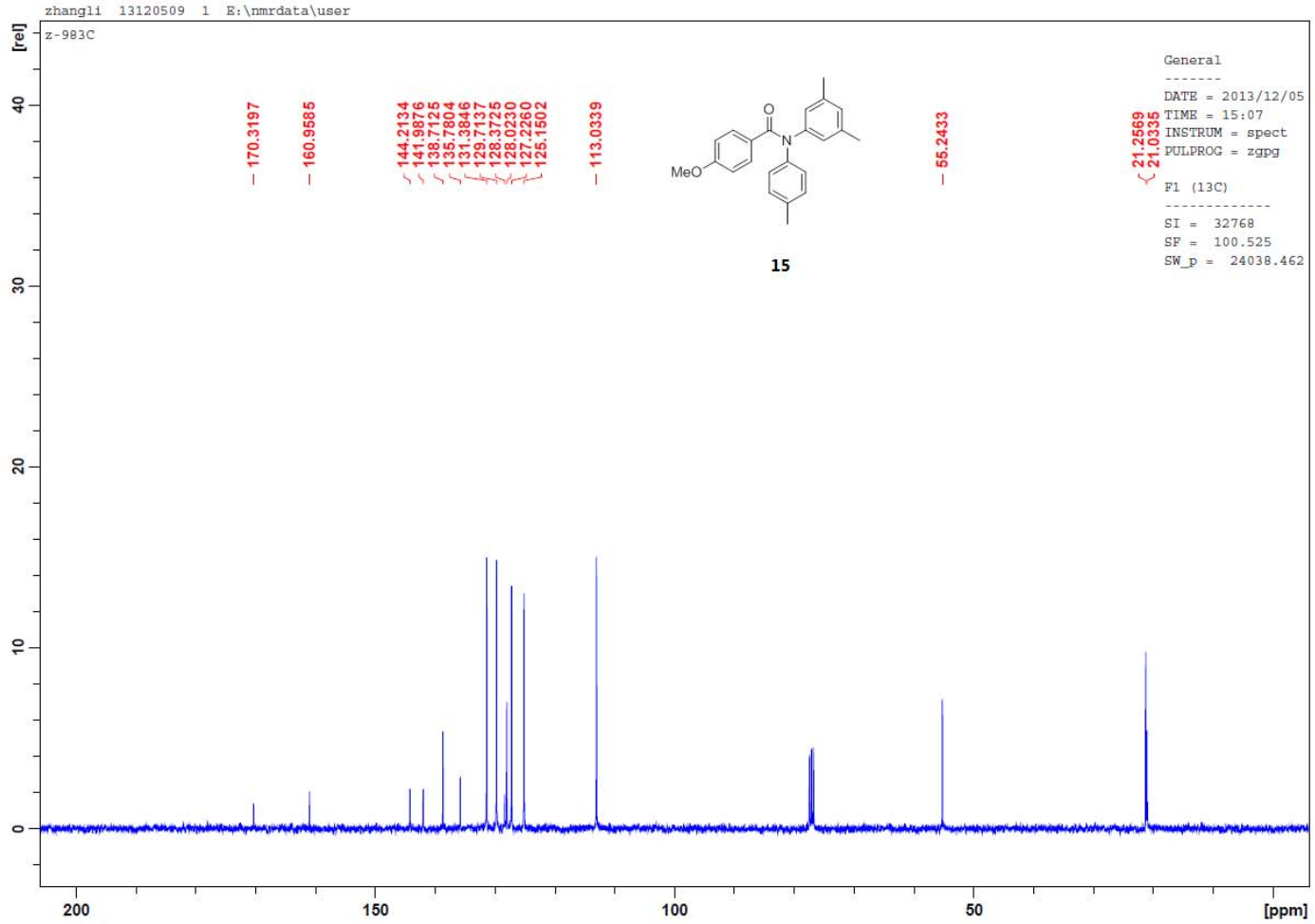

待整理核磁
z-1000C
170.1235
160.7779
146.5093
141.8676
141.8103
135.7395
131.2572
129.6172
128.1923
127.2038
126.9226
126.8627
112.8992
55.1136
33.5079
23.8390
20.9144
MeO
O
N
f1 (ppm)

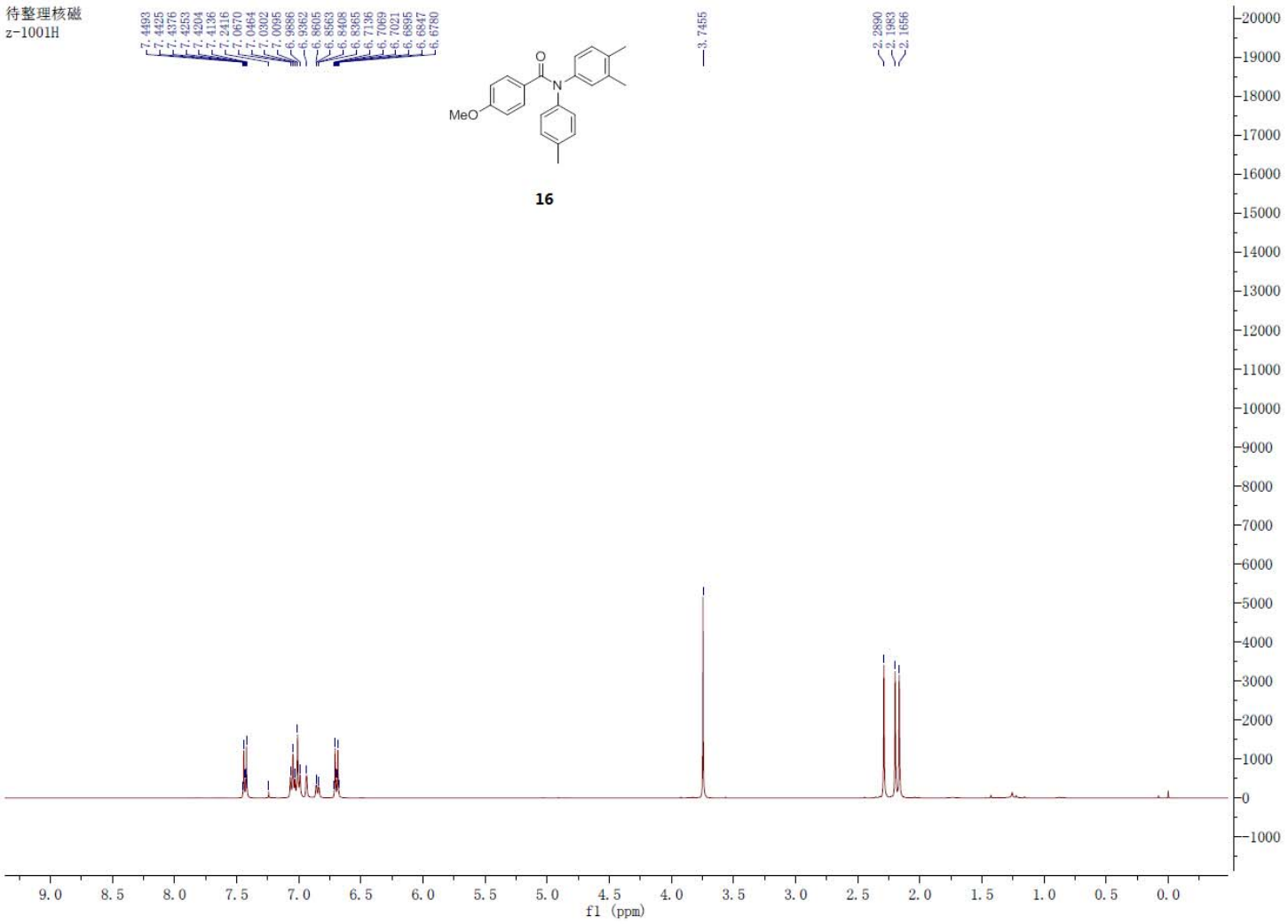
13

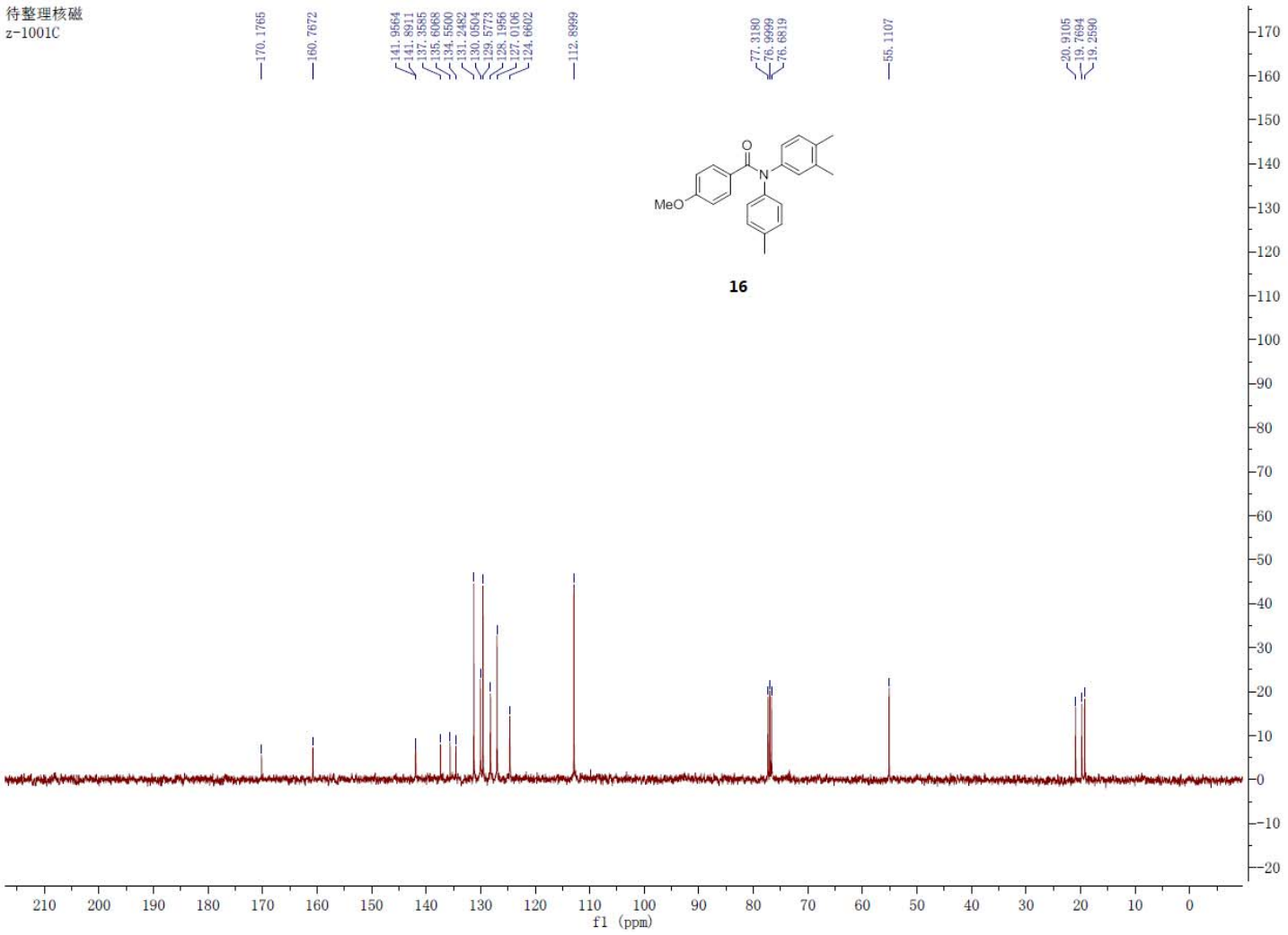

zhangli 13120506 1 E:\nmrdata\user
z-982H
[rel]
30
20
10
0
7.4421
7.4200
7.2884
7.2694
7.2495
7.1694
7.1510
7.1363
7.1180
7.0921
7.0716
7.0254
7.0047
6.7138
6.6917
MeO
O
N
14
3.7505
2.3017
General
DATE = 2013/12/05
TIME = 10:43
INSTRUM = spect
PULPROG = zg
F1 (1H)
SI = 65536
SF = 399.78
SW_p = 6009.615
19.3506
20.7250
46.6952
21.6640
19.6440
30.0000
29.0059
8
6
4
2
[ppm]

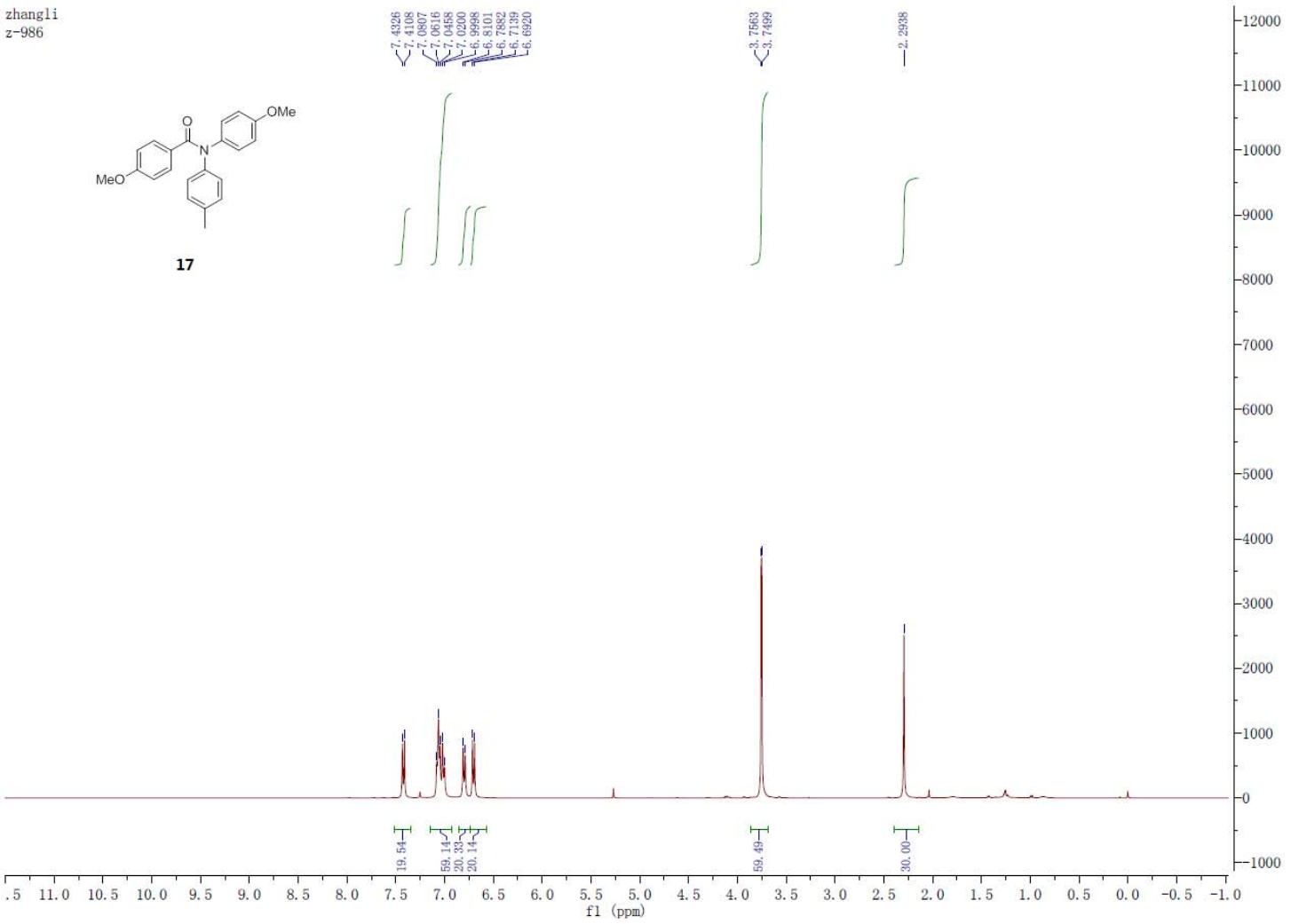

zhangli 13120507 1 E:\nmrdata\user
z-982C
[rel]
170.3032
161.0322
144.4956
141.8054
136.0222
131.4118
129.8106
129.0633
128.1970
127.3199
126.0094
113.1072
55.2581
21.0406
MeO
O
N
14
General
DATE = 2013/12/05
TIME = 10:45
INSTRUM = spect
PULPROG = zgpg
F1 (13C)
SI = 32768
SF = 100.525
SW_p = 24038.462
0
10
20
30
200
150
100
50
[ppm]

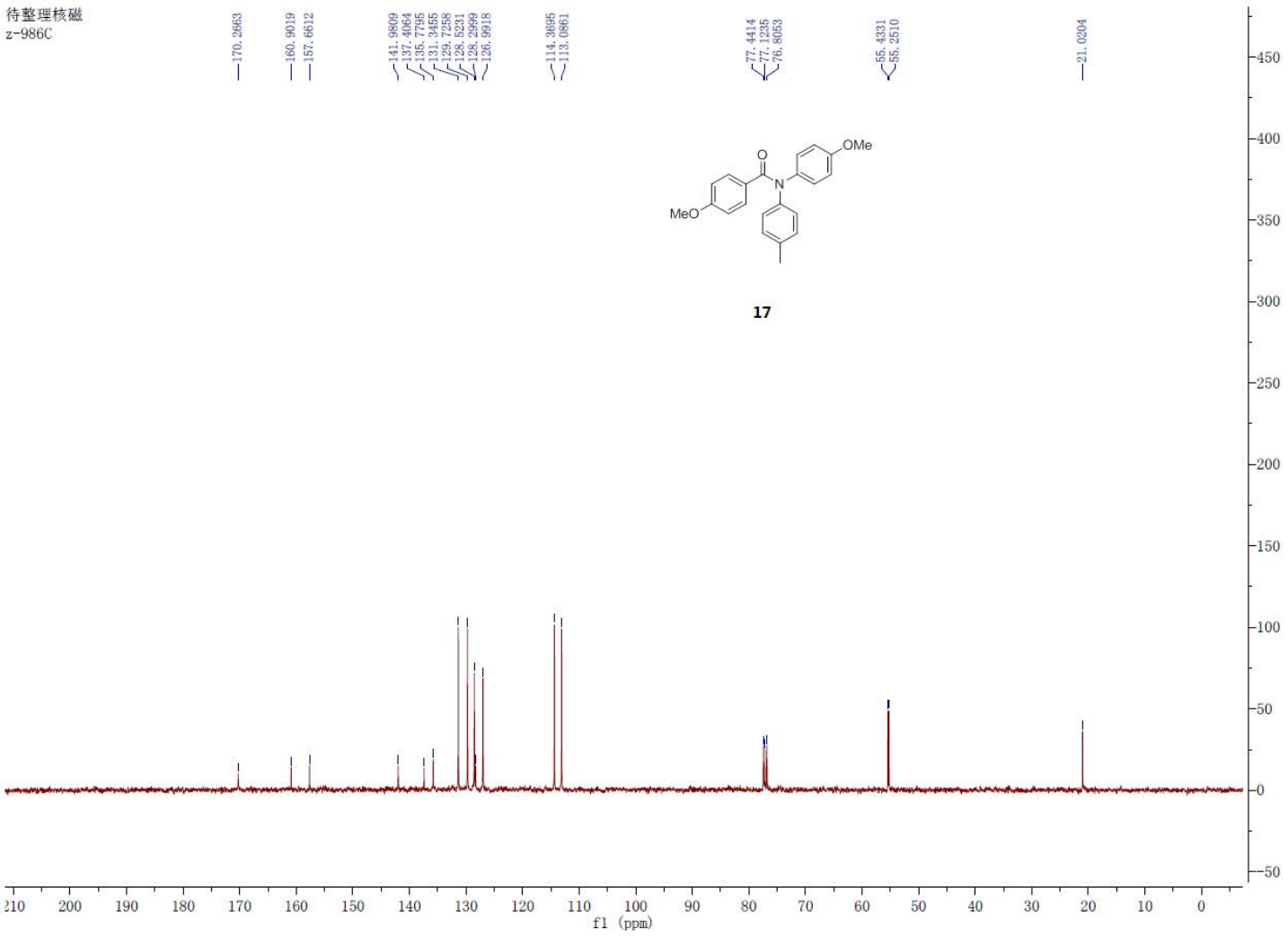

zhangli 13120508 1 E:\nmrdata\user
z-983H
[rel]
30
20
10
0
7.4431
7.4212
7.0675
7.0471
7.0018
6.9813
6.8075
6.7702
6.7110
6.6891
3.7513
2.2935
2.2225
MeO
O
N
15
General
DATE = 2013/12/05
TIME = 15:03
INSTRUM = spect
PULPROG = zg
F1 (1H)
SI = 65536
SF = 399.78
SW_p = 6009.615
19.2521
17.8749
21.7419
27.9526
21.2409
30.0000
31.3138
58.4997
8
6
4
2
[ppm]

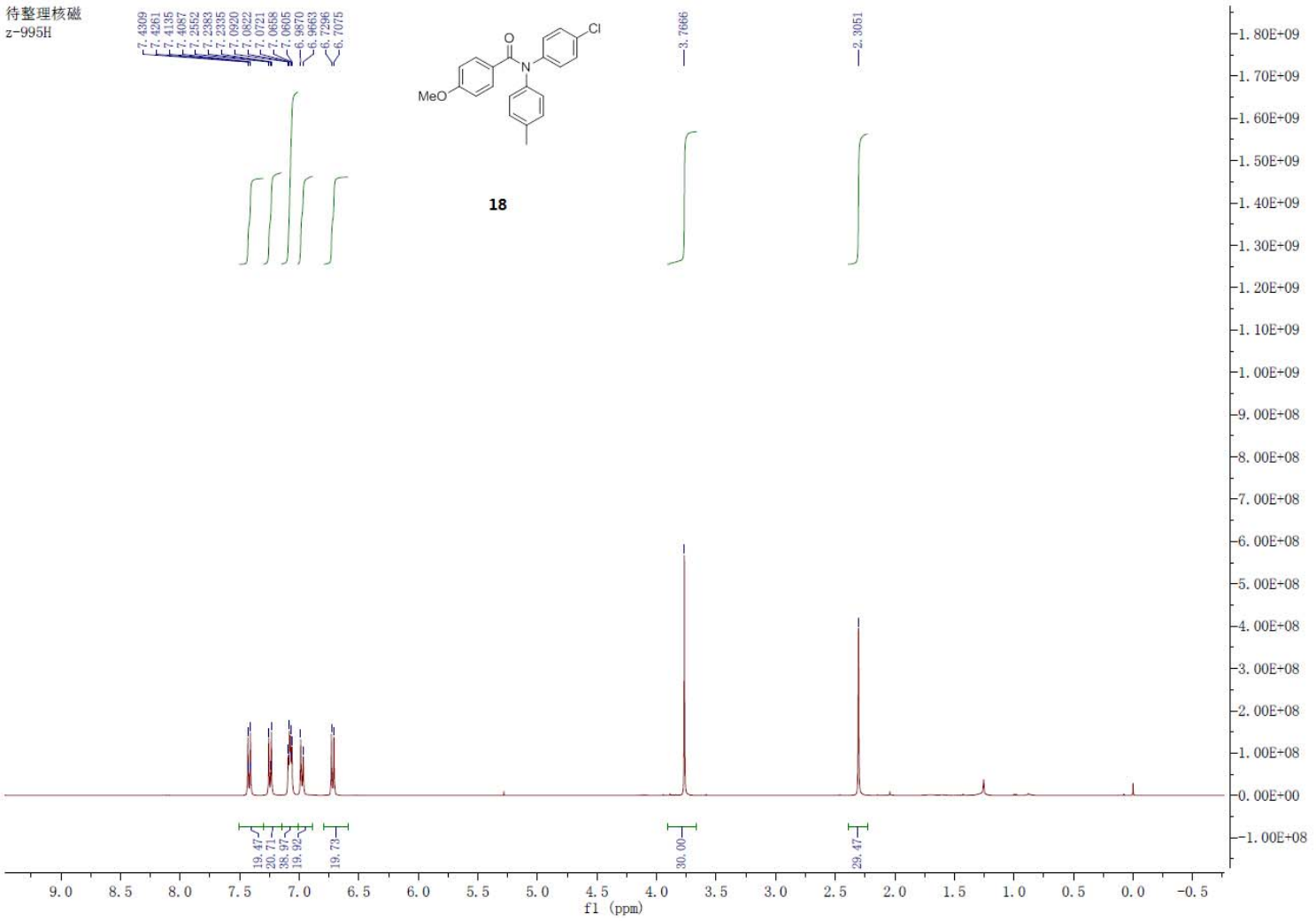

zhangli 13120509 1 E:\nmrdata\user
z-983C
[rel]
170.3197
160.9585
144.2134
141.9876
138.7125
135.7804
131.3846
129.7137
128.3725
128.0230
127.2260
125.1502
113.0339
55.2433
21.2569
21.0335
MeO
O
N
15
General
DATE = 2013/12/05
TIME = 15:07
INSTRUM = spect
PULPROG = zgpg
F1 (13C)
SI = 32768
SF = 100.525
SW_p = 24038.462
200
150
100
50
[ppm]

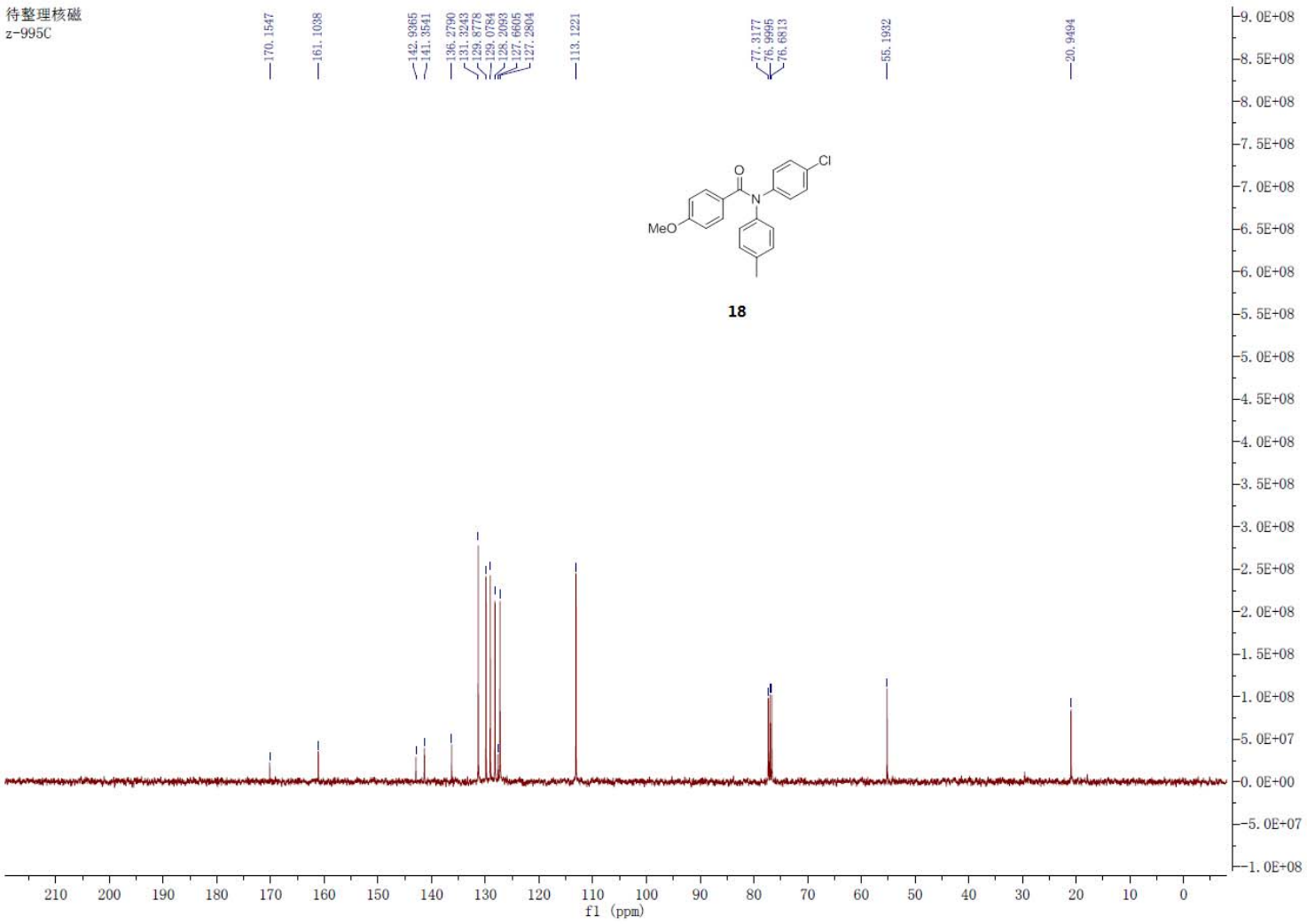

待整理核磁
z-1001H
7.4493
7.4425
7.4376
7.4253
7.4204
7.4136
7.2416
7.0670
7.0464
7.0302
7.0095
6.9886
6.9362
6.8605
6.8563
6.8408
6.8365
6.7136
6.7069
6.7021
6.6895
6.6847
6.6780
3.7455
2.2890
2.1983
2.1656
MeO
O
N
16
f1 (ppm)

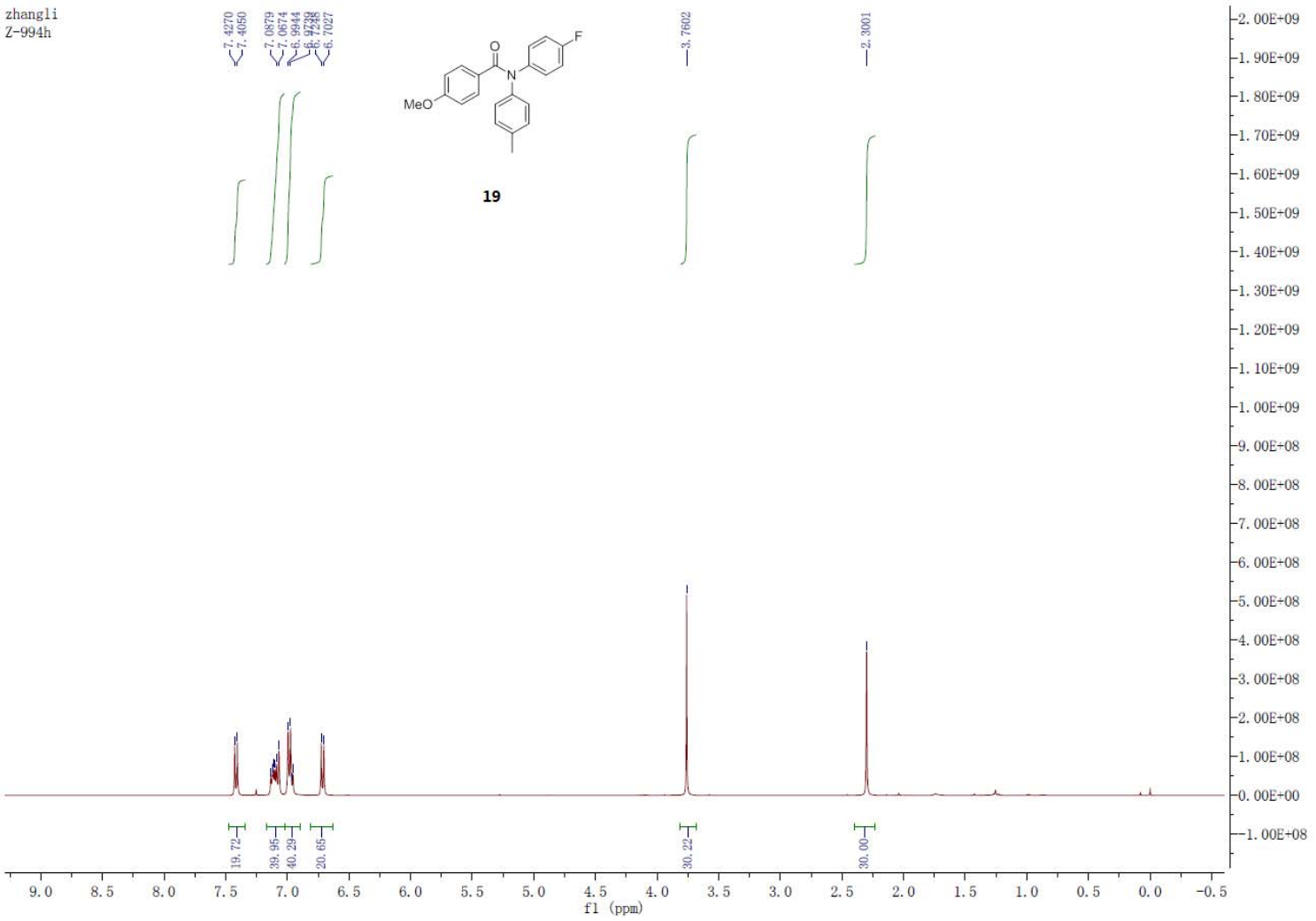

待整理核磁
z-1001C
170.1765
160.7672
141.9564
141.8911
137.3585
135.6068
134.5500
131.2482
130.0504
129.5773
128.1956
127.0106
124.6602
112.8999
77.3180
76.9999
76.6819
55.1107
20.9105
19.7694
19.2590
MeO
O
N
16
f1 (ppm)

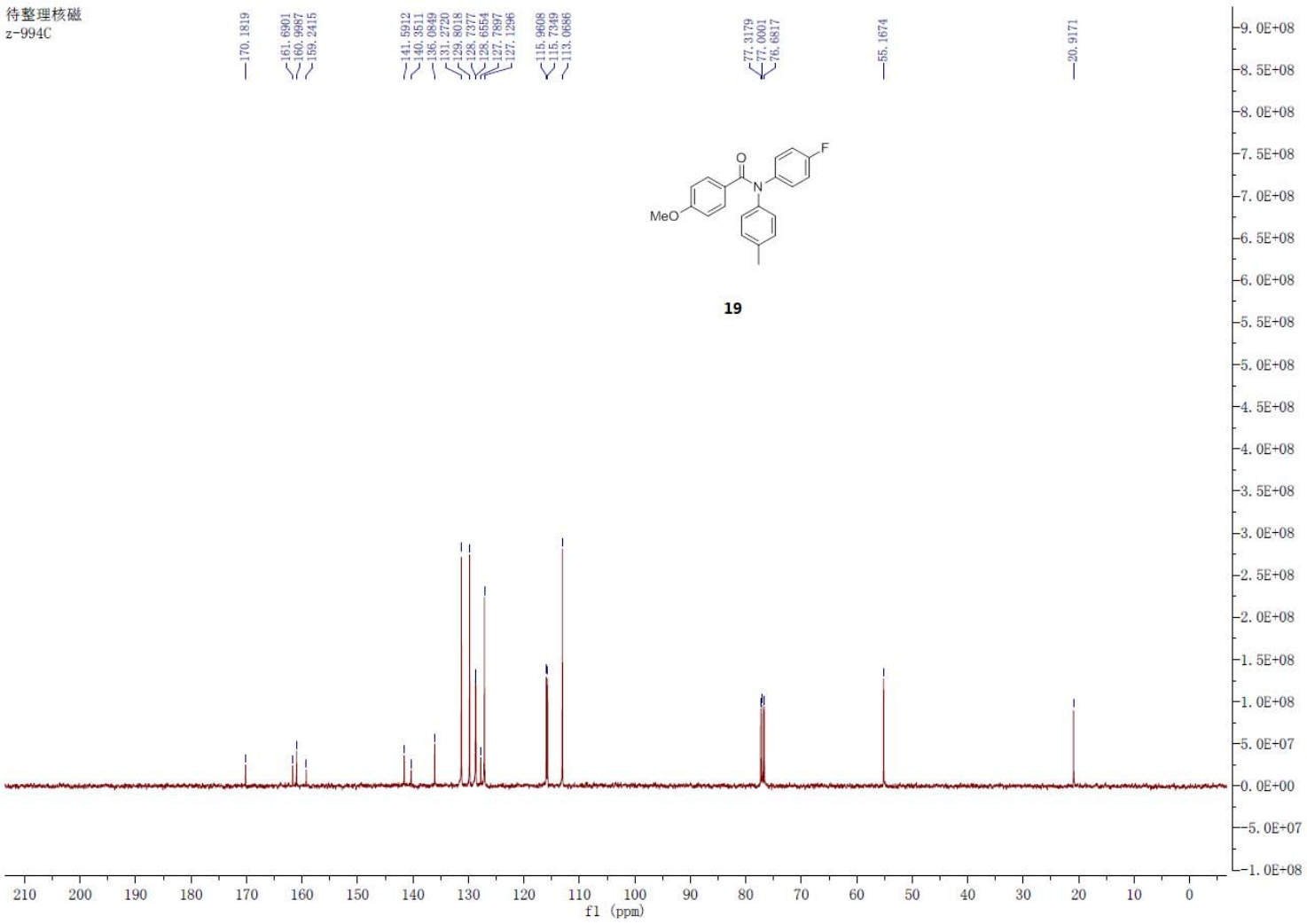

zhangli
z-986
OMe
MeO
O
N
17
7.4326
7.4108
7.0807
7.0616
7.0458
7.0200
6.9998
6.8101
6.7882
6.7139
6.6920
3.7563
3.7499
2.2938
19.54
59.14
20.33
20.14
59.49
30.00
f1 (ppm)

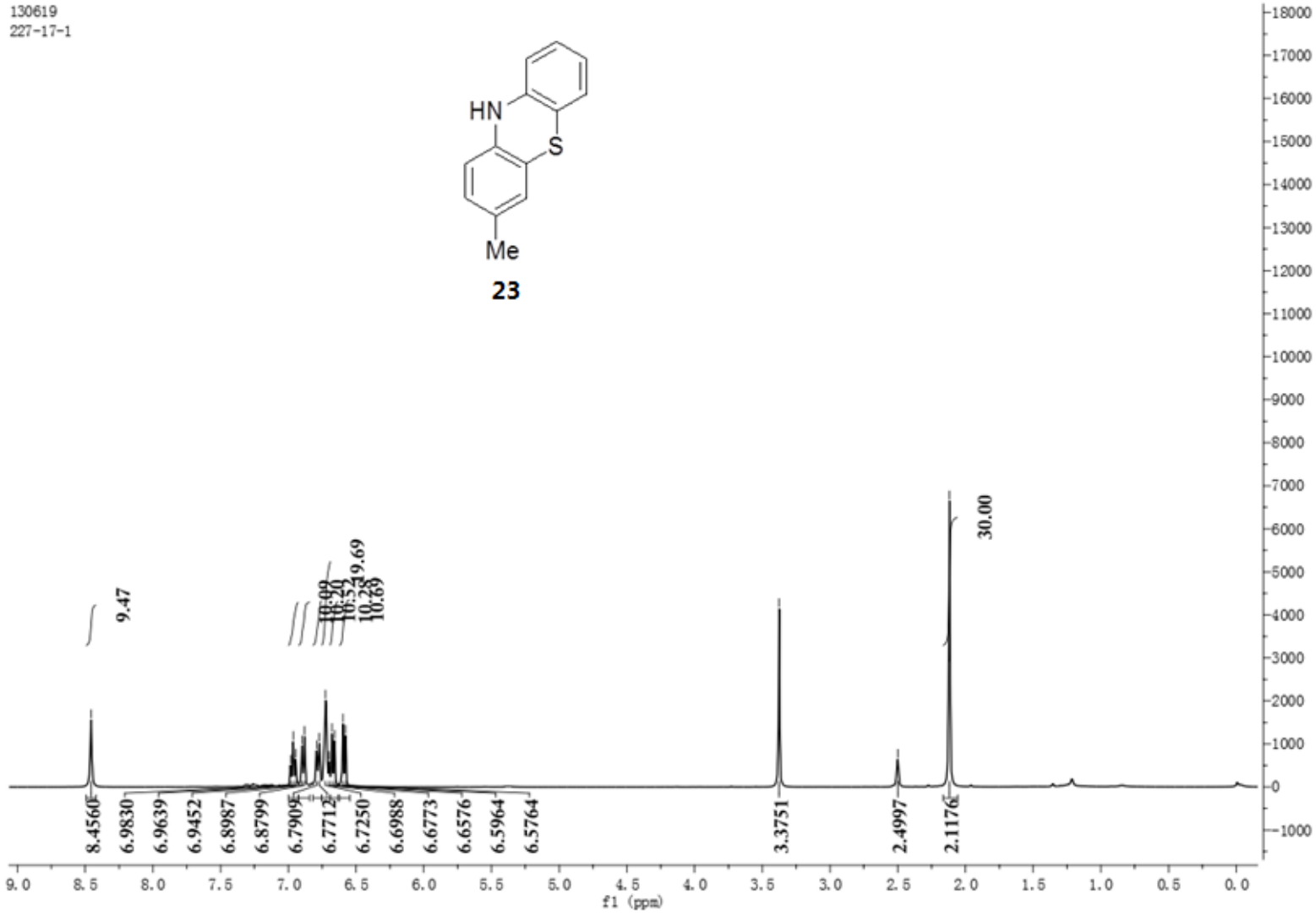

待整理核磁
z-986C
170.2663
160.9019
157.6612
141.9809
137.4064
135.7795
131.3455
129.7258
128.5231
128.2999
126.9918
114.3695
113.0861
77.4414
77.1235
76.8053
55.4331
55.2510
21.0204
OMe
MeO
O
N
17
f1 (ppm)

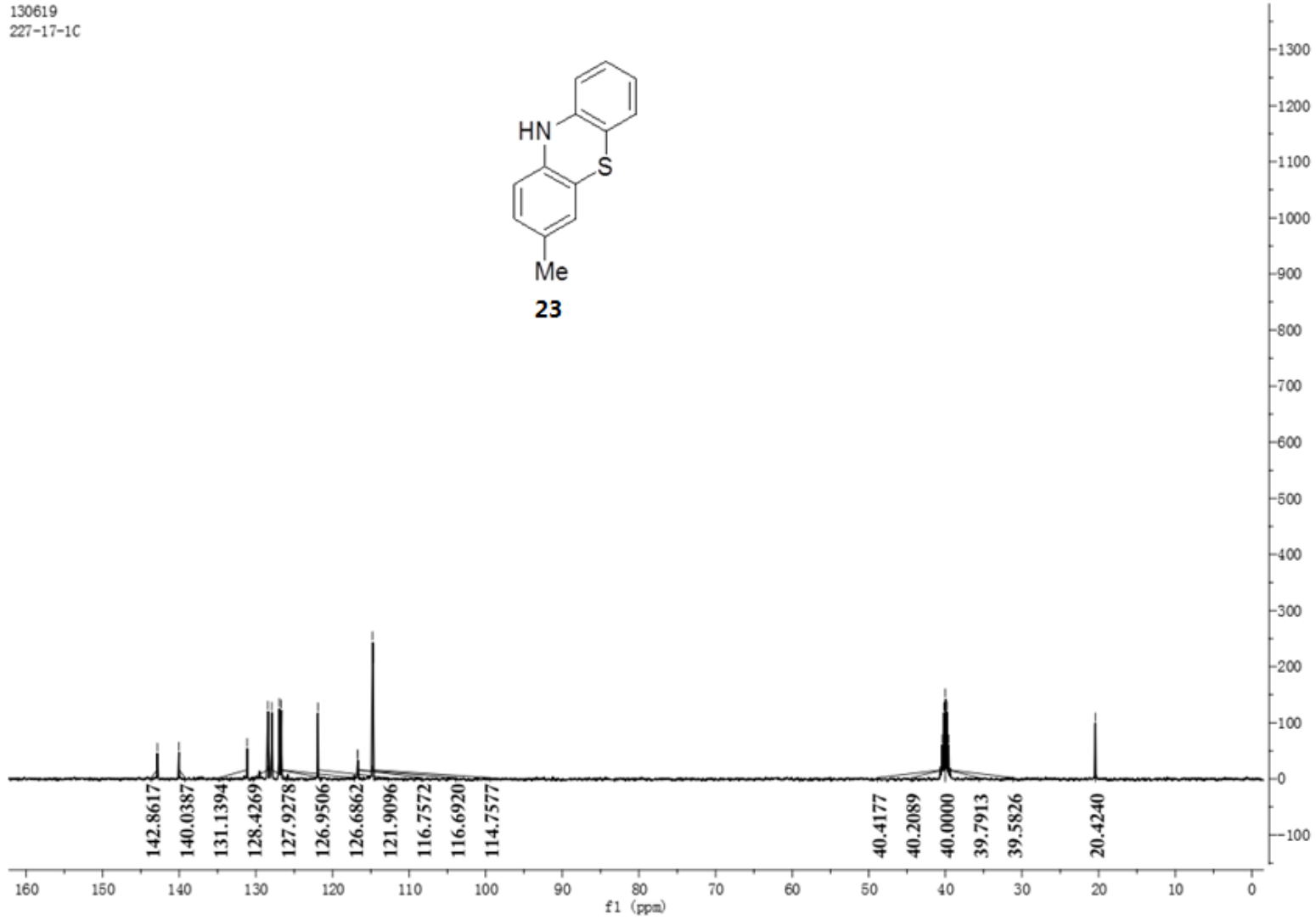

待整理核磁
z-995H
7.4309
7.4261
7.4135
7.4087
7.2552
7.2383
7.2335
7.0920
7.0822
7.0721
7.0658
7.0605
6.9870
6.9663
6.7296
6.7075
3.7666
2.3051
MeO
O
N
Cl
18
19.47
20.71
38.97
19.92
19.73
30.00
29.47
f1 (ppm)

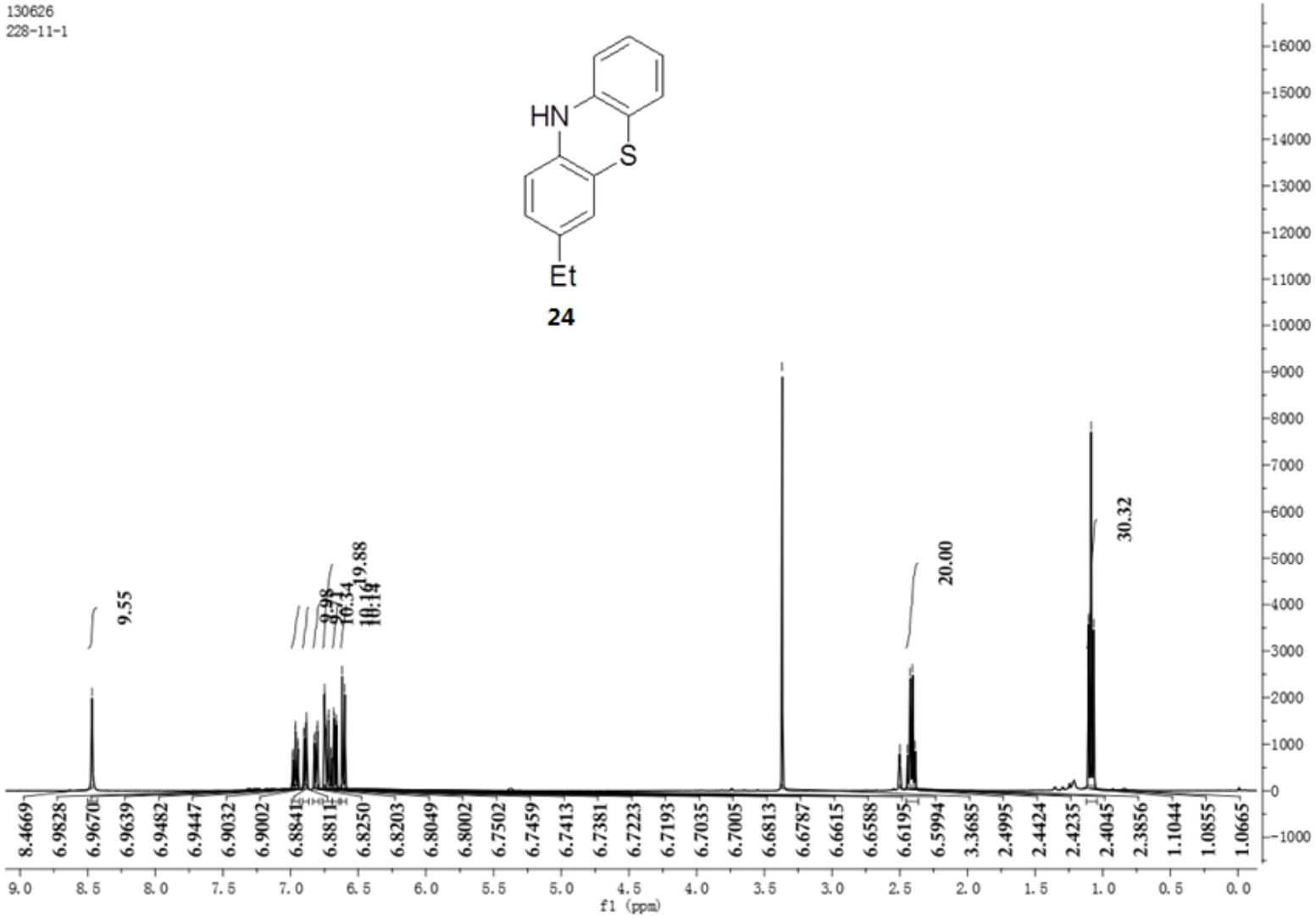

待整理核磁
z-995C
170.1547
161.1038
142.9365
141.3541
136.2790
131.3243
129.8778
129.0784
128.2093
127.6605
127.2804
113.1221
77.3177
76.9995
76.6813
55.1932
20.9494
O
N
Cl
MeO
18
f1 (ppm)

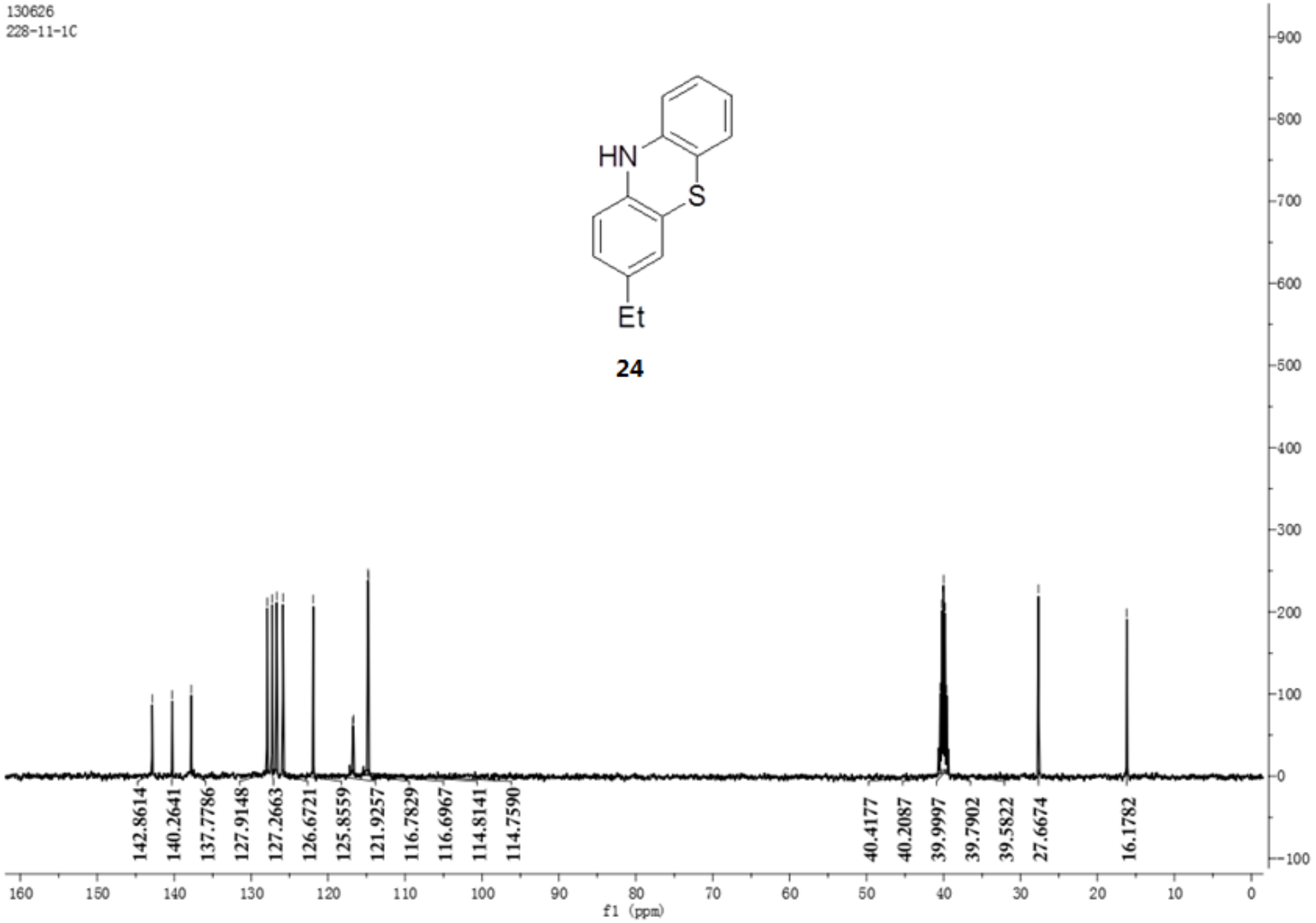

zhangli
Z-994h
7.4270
7.4050
7.0879
7.0674
6.9944
6.9738
6.9728
6.7027
3.7602
2.3001
O
N
F
MeO
19
19.72
39.95
40.29
20.65
30.22
30.00
f1 (ppm)


待整理核磁
z-994C
170.1819
161.6901
160.9967
159.2415
141.5912
140.3511
136.0849
131.2720
129.8018
128.7377
128.6554
127.7697
127.1296
115.9608
115.7349
113.0686
77.3179
77.0001
76.6817
55.1674
20.9171
MeO
O
N
F
19
9.0E+08
8.5E+08
8.0E+08
7.5E+08
7.0E+08
6.5E+08
6.0E+08
5.5E+08
5.0E+08
4.5E+08
4.0E+08
3.5E+08
3.0E+08
2.5E+08
2.0E+08
1.5E+08
1.0E+08
5.0E+07
0.0E+00
-5.0E+07
-1.0E+08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

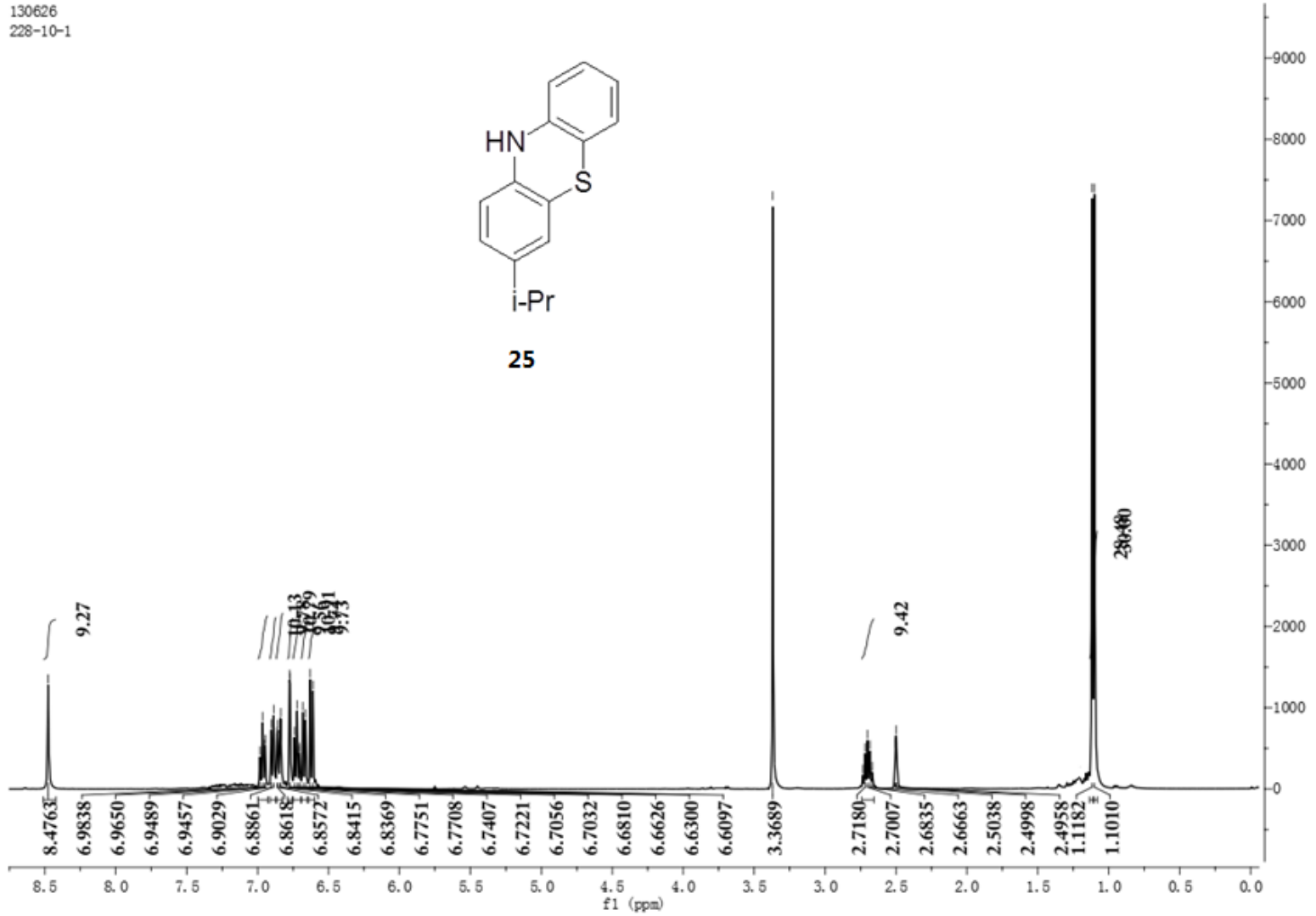

130619
227-17-1
HN
S
Me
23
9.47
10.09
10.20
10.52
10.73
10.89
9.69
30.00
8.4560
6.9830
6.9639
6.9452
6.8987
6.8799
6.7909
6.7712
6.7250
6.6988
6.6773
6.6576
6.5964
6.5764
3.3751
2.4997
2.1176
f1 (ppm)

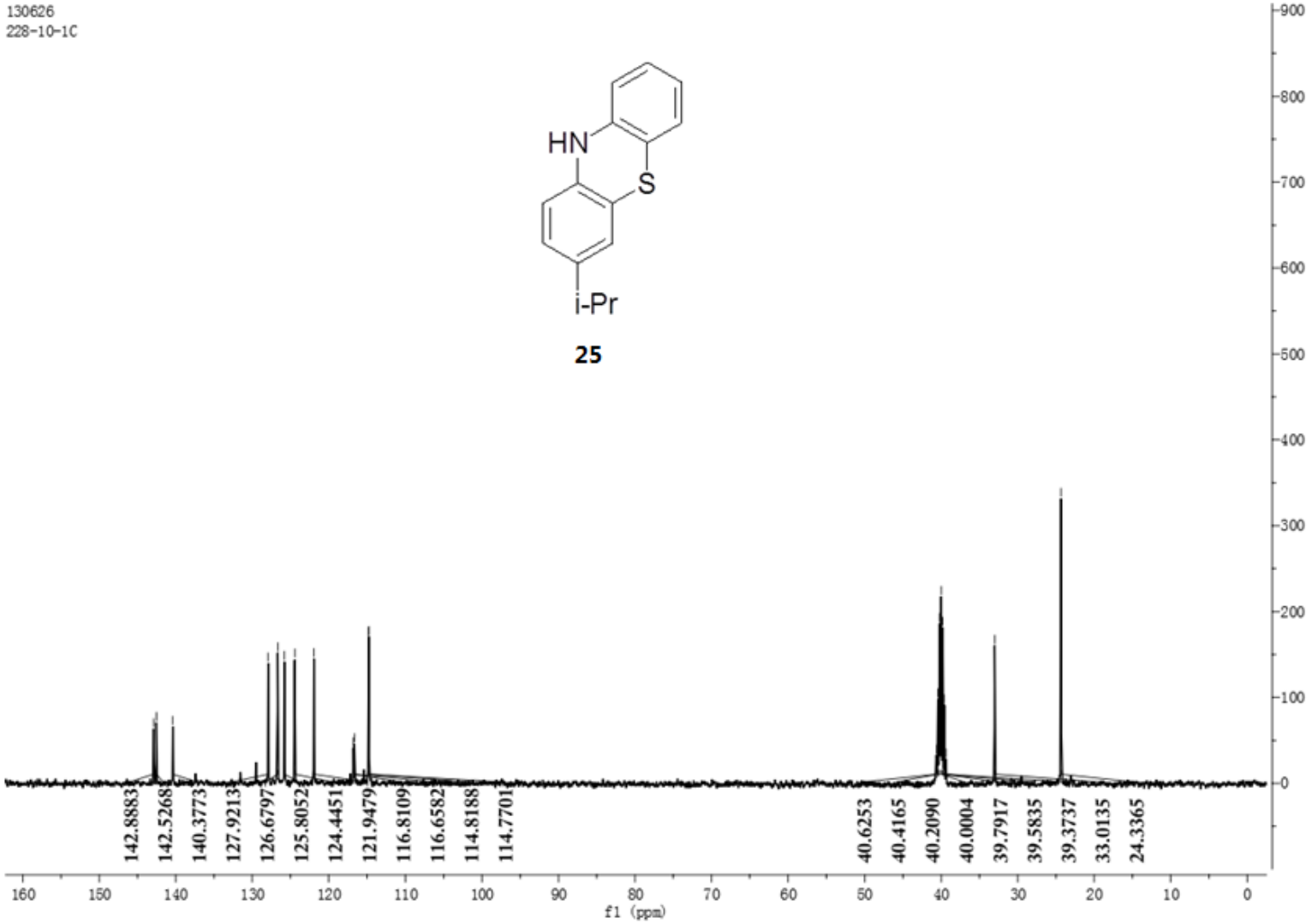

130619
227-17-1C
HN
S
Me
23
142.8617
140.0387
131.1394
128.4269
127.9278
126.9506
126.6862
121.9096
116.7572
116.6920
114.7577
40.4177
40.2089
40.0000
39.7913
39.5826
20.4240
f1 (ppm)


130626
228-11-1
HN
S
Et
24
9.55
20.00
30.32
f1 (ppm)


130626
228-11-1C
HN
S
Et
142.8614
140.2641
137.7786
127.9148
127.2663
126.6721
125.8559
121.9257
116.7829
116.6967
114.8141
114.7590
40.4177
40.2087
39.9997
39.7902
39.5822
27.6674
16.1782
f1 (ppm)

24


130626
228-10-1
HN
S
i-Pr
25
9.27
9.42
8.4763
6.9838
6.9650
6.9489
6.9457
6.9029
6.8861
6.8572
6.8415
6.8369
6.7751
6.7708
6.7407
6.7221
6.7056
6.7032
6.6810
6.6626
6.6300
6.6097
3.3689
2.7180
2.7007
2.6835
2.6663
2.5038
2.4998
2.4958
1.1182
1.1010
f1 (ppm)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228-10-1C
HN
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i-Pr
25
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142.5268
140.3773
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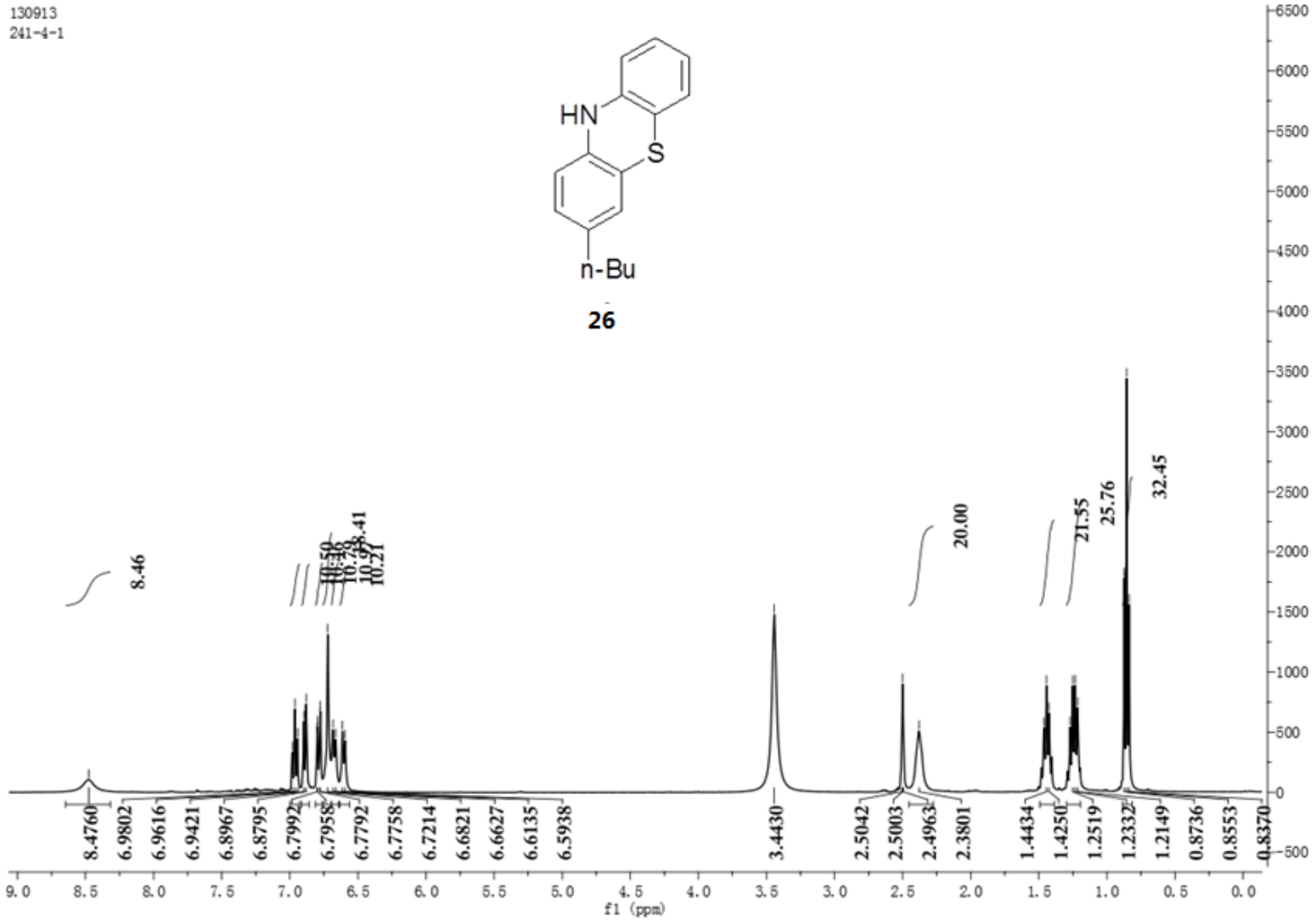
126.6797
125.8052
124.4451
121.9479
116.8109
116.6582
114.8188
114.7701
40.6253
40.4165
40.2090
40.0004
39.7917
39.5835
39.3737
33.0135
24.3365
f1 (ppm)


130913
241-4-1
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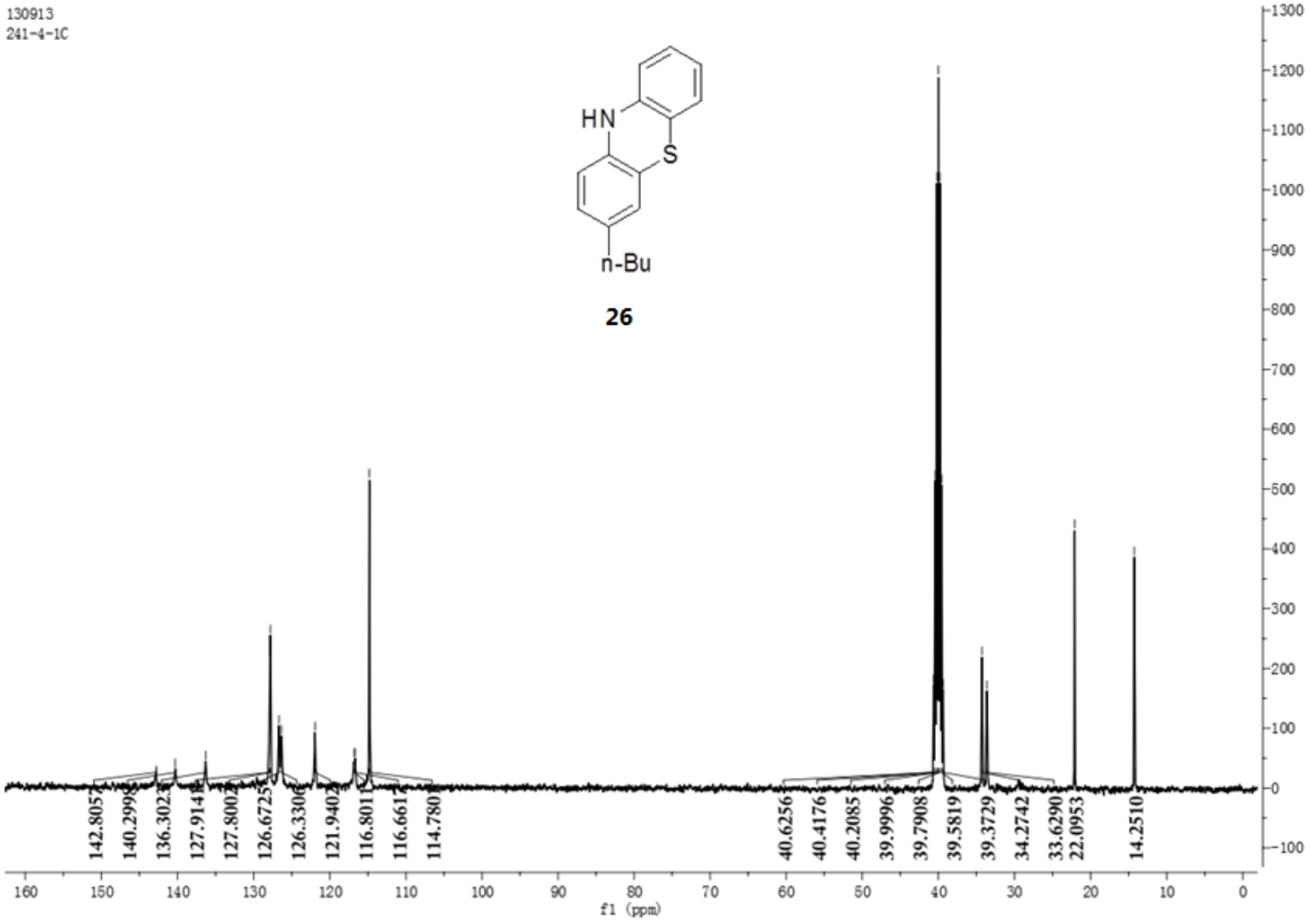
S
n-Bu
26
8.46
20.00
21.55
25.76
32.45
8.4760
6.9802
6.9616
6.9421
6.8967
6.8795
6.7992
6.7792
6.7758
6.7214
6.6821
6.6627
6.6135
6.5938
3.4430
2.5042
2.5003
2.4963
2.3801
1.4434
1.4250
1.2519
1.2332
1.2149
0.8736
0.8553
0.8370
f1 (ppm)


130913
241-4-1C
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S
n-Bu
26
142.8057
140.2998
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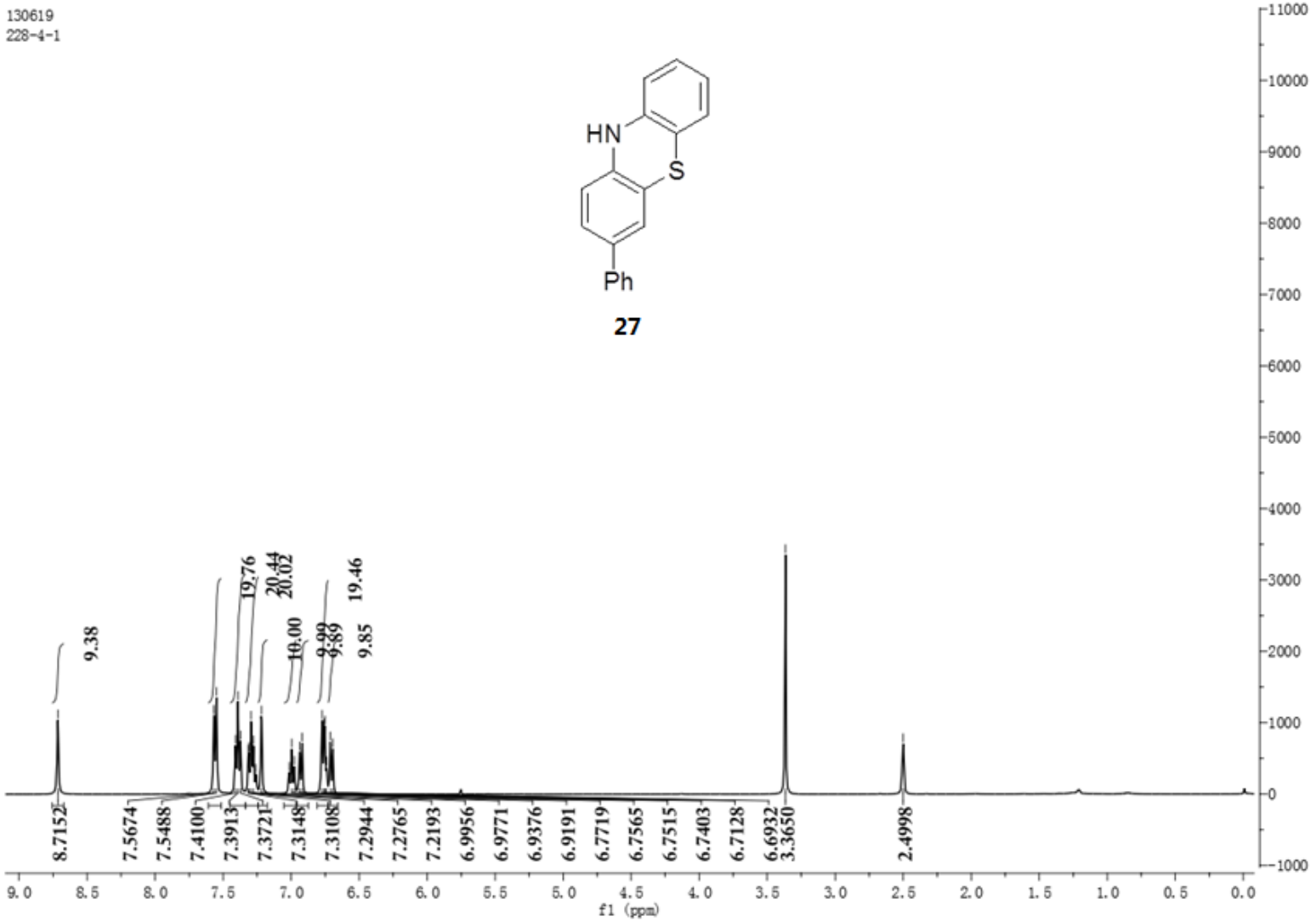
127.9148
127.8002
126.6725
126.3306
121.9402
116.8011
116.6614
114.7801
40.6256
40.4176
40.2085
39.9996
39.7908
39.5819
39.3729
34.2742
33.6290
22.0953
14.2510
f1 (ppm)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228-4-1
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S
Ph
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9.38
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20.44
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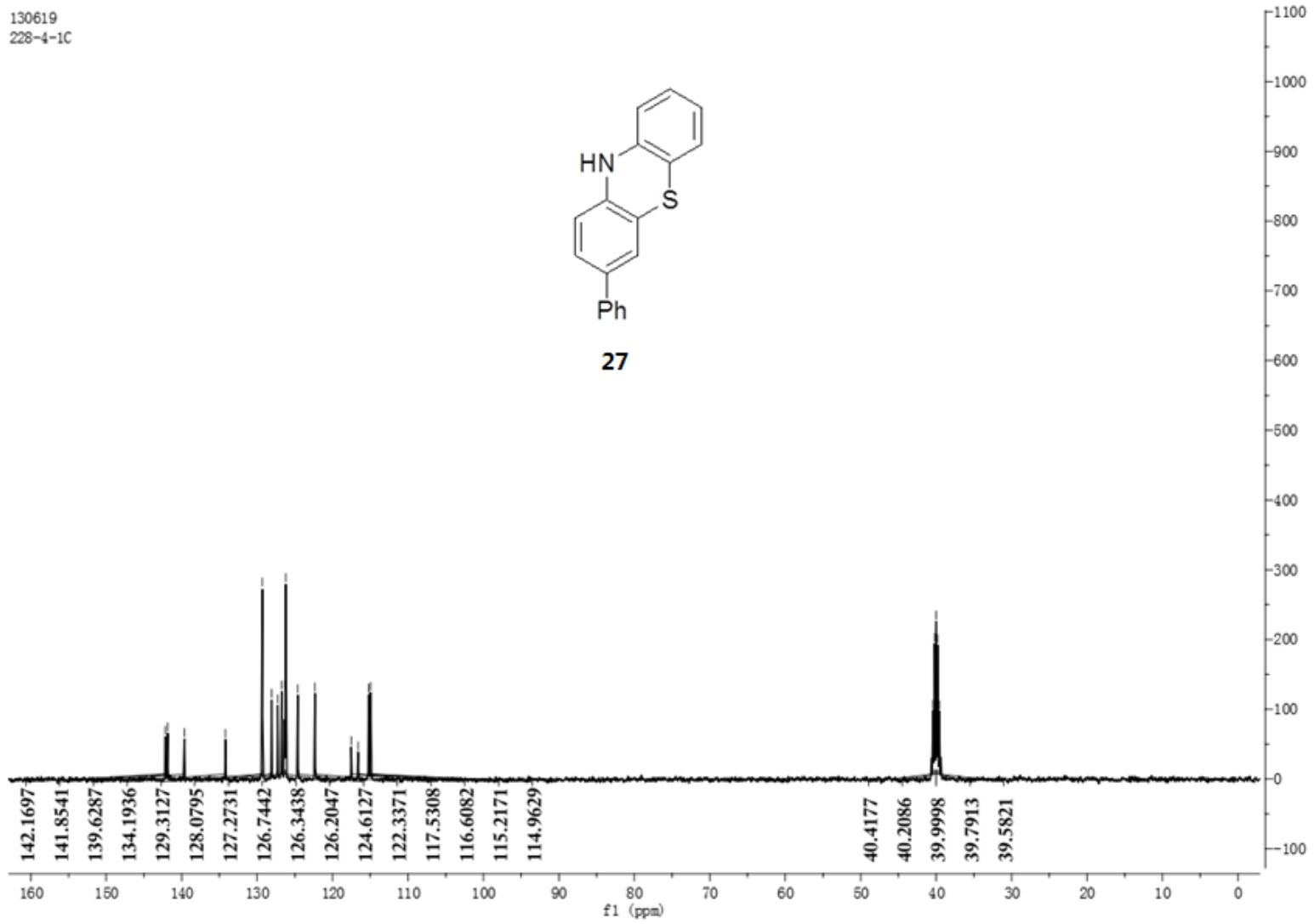
10.00
9.99
9.89
9.85
19.46
8.7152
7.5674
7.5488
7.4100
7.3913
7.3721
7.3148
7.3108
7.2944
7.2765
7.2193
6.9956
6.9771
6.9376
6.9191
6.7719
6.7565
6.7515
6.7403
6.7128
6.6932
3.3650
2.4998
f1 (ppm)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S
Ph
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142.1697
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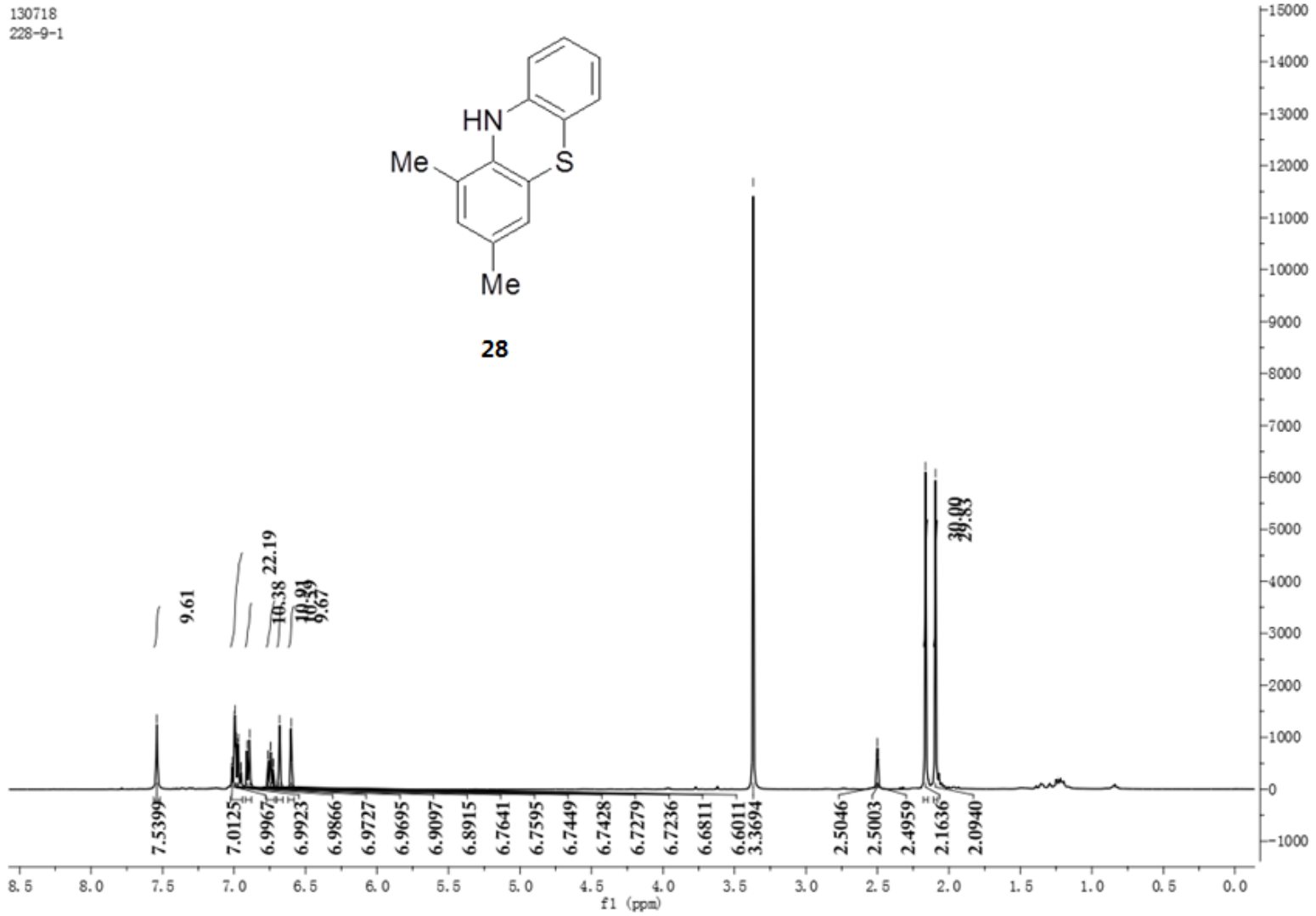
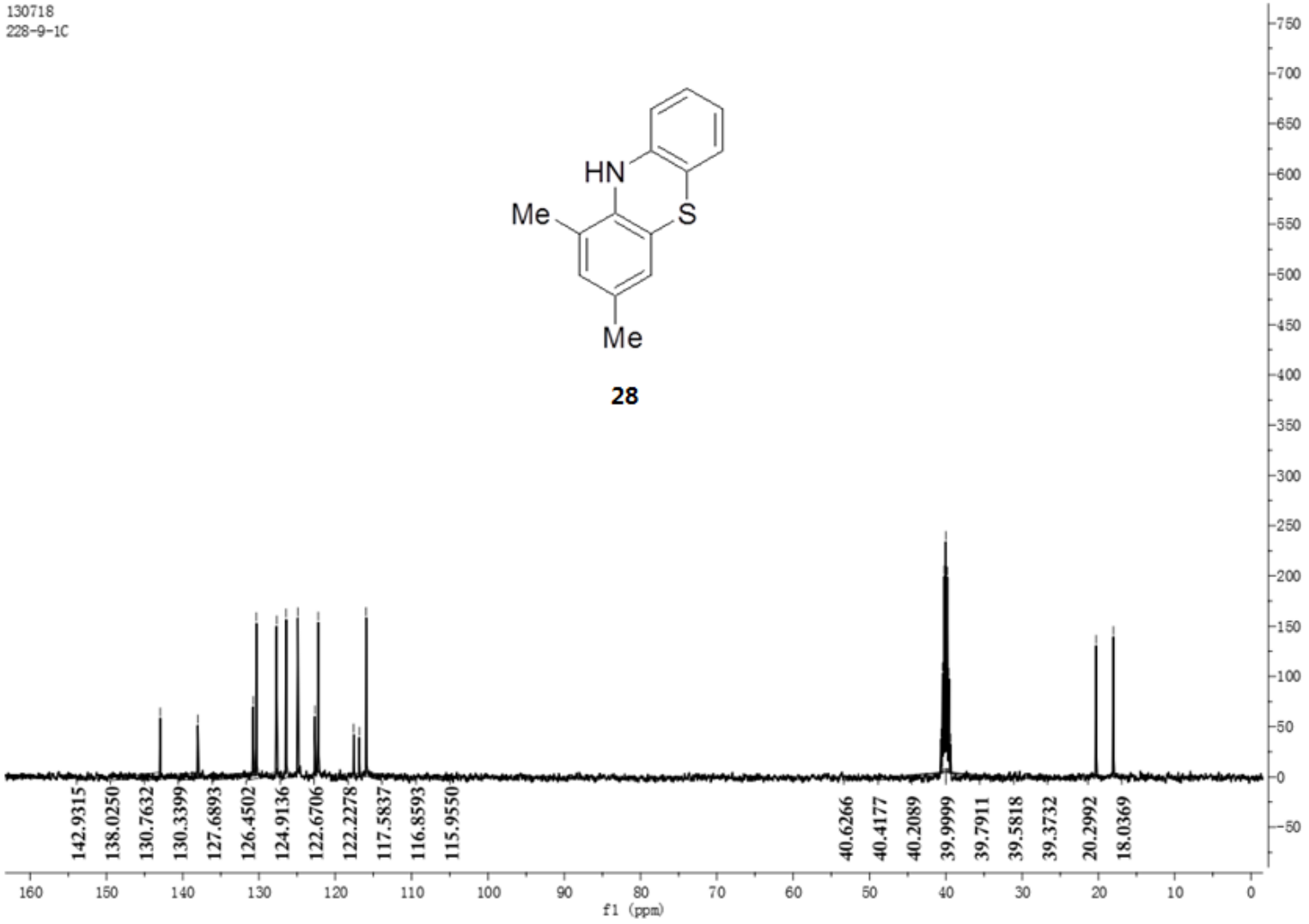
139.6287
134.1936
129.3127
128.0795
127.2731
126.7442
126.3438
126.2047
124.6127
122.3371
117.5308
116.6082
115.2171
114.9629
40.4177
40.2086
39.9998
39.7913
39.5821
f1 (ppm)


130718
228-9-1
HN
Me
S
Me
28
9.61
22.19
10.38
10.31
10.39
9.67
7.5399
7.0125
6.9967
6.9923
6.9866
6.9727
6.9695
6.9097
6.8915
6.7641
6.7595
6.7449
6.7428
6.7279
6.7236
6.6811
6.6011
3.3694
2.5046
2.5003
2.4959
2.1636
2.0940
f1 (ppm)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Me
S
Me
28
142.9315
138.0250
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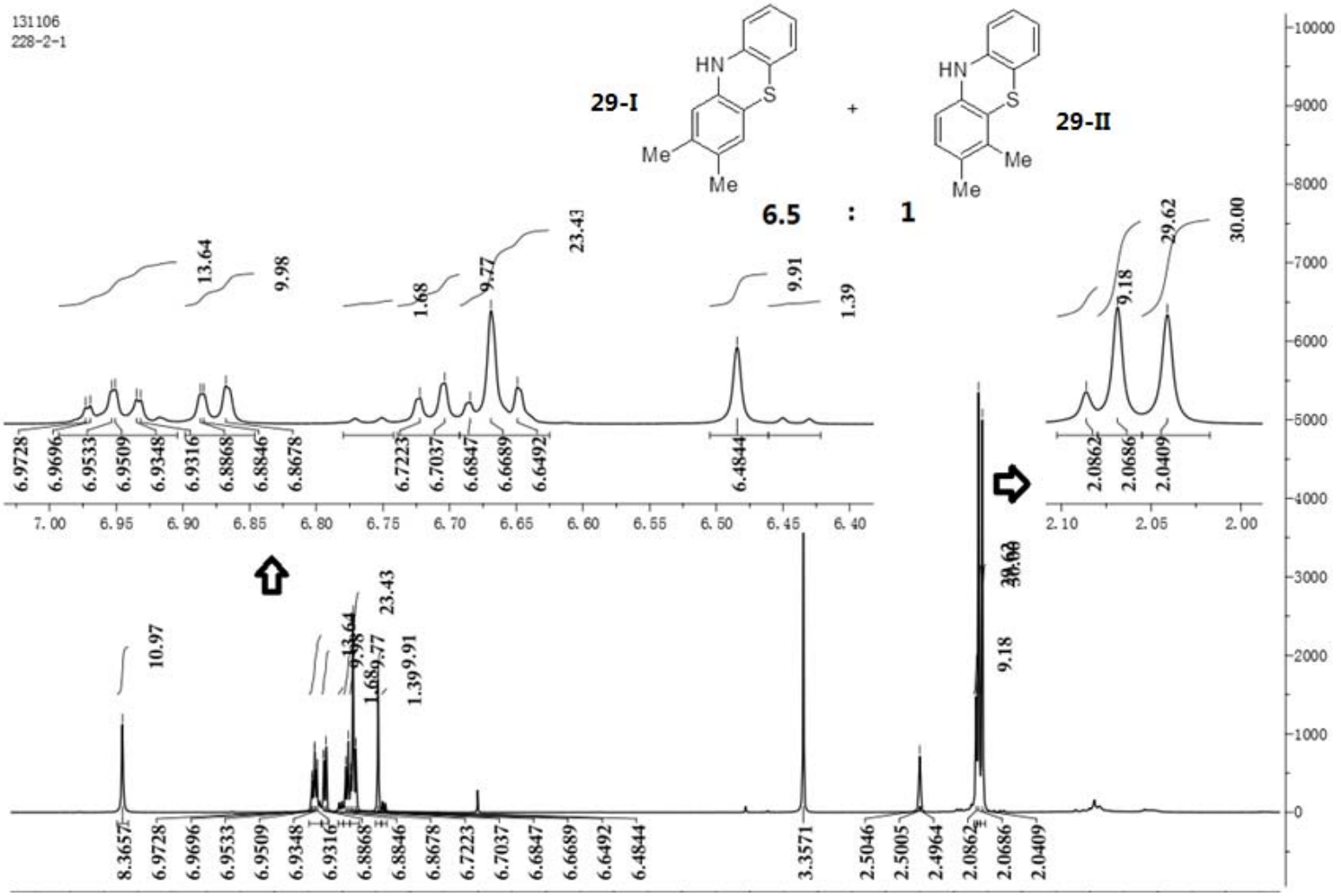
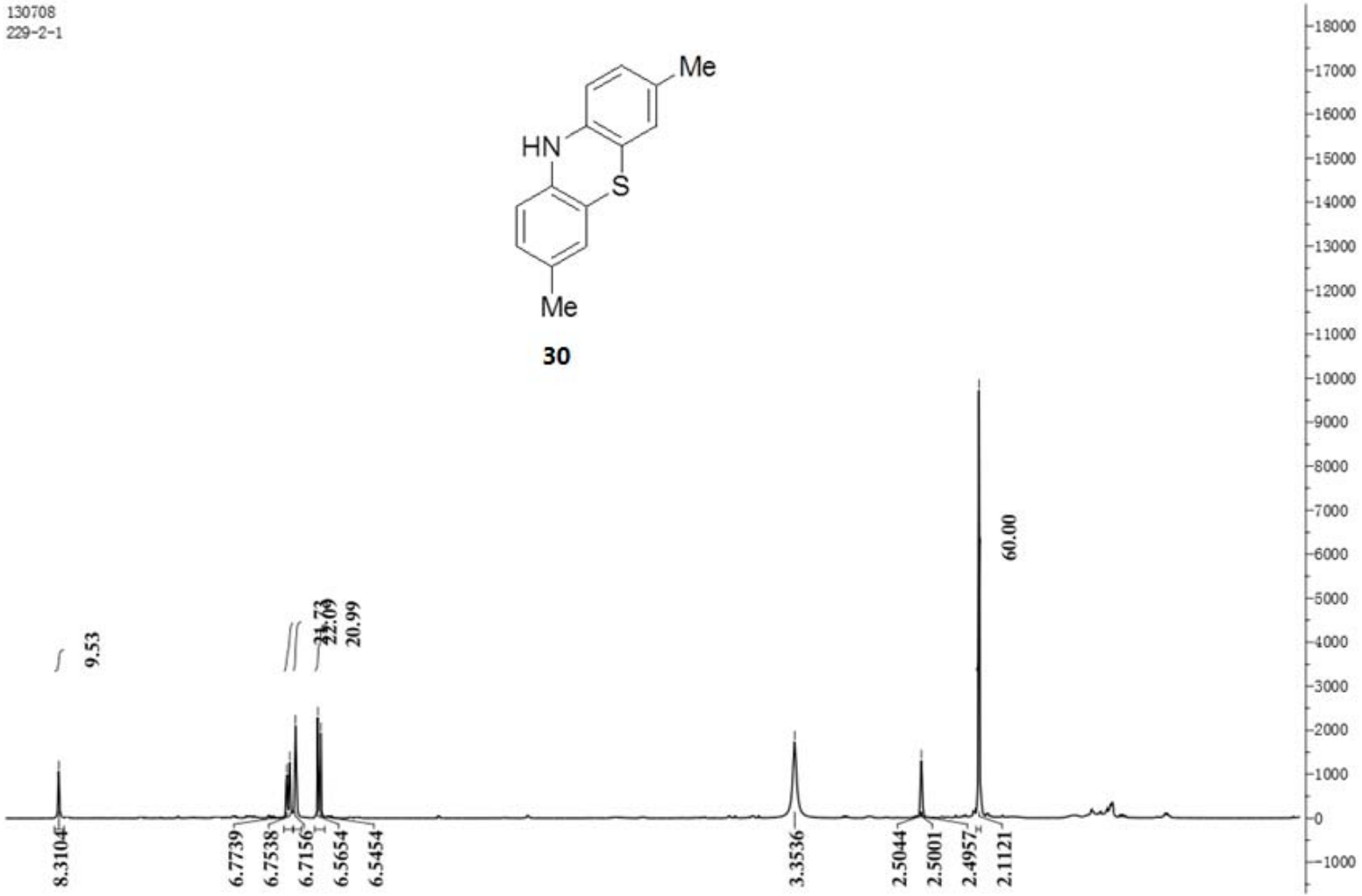
130.3399
127.6893
126.4502
124.9136
122.6706
122.2278
117.5837
116.8593
115.9550
40.6266
40.4177
40.2089
39.9999
39.7911
39.5818
39.3732
20.2992
18.0369
f1 (ppm)


131106
228-2-1
29-I
+
29-II
6.5 : 1
HN
S
Me
Me
HN
S
Me
Me
13.64
9.98
1.68
9.77
23.43
9.91
1.39
9.18
29.62
30.00
6.9728
6.9696
6.9533
6.9509
6.9348
6.9316
6.8868
6.8846
6.8678
6.7223
6.7037
6.6847
6.6689
6.6492
6.4844
2.0862
2.0686
2.0409
10.97
8.3657
3.3571
2.5046
2.5005
2.4964


130708
229-2-1
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HN
S
Me
30
9.53
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60.00
8.3104
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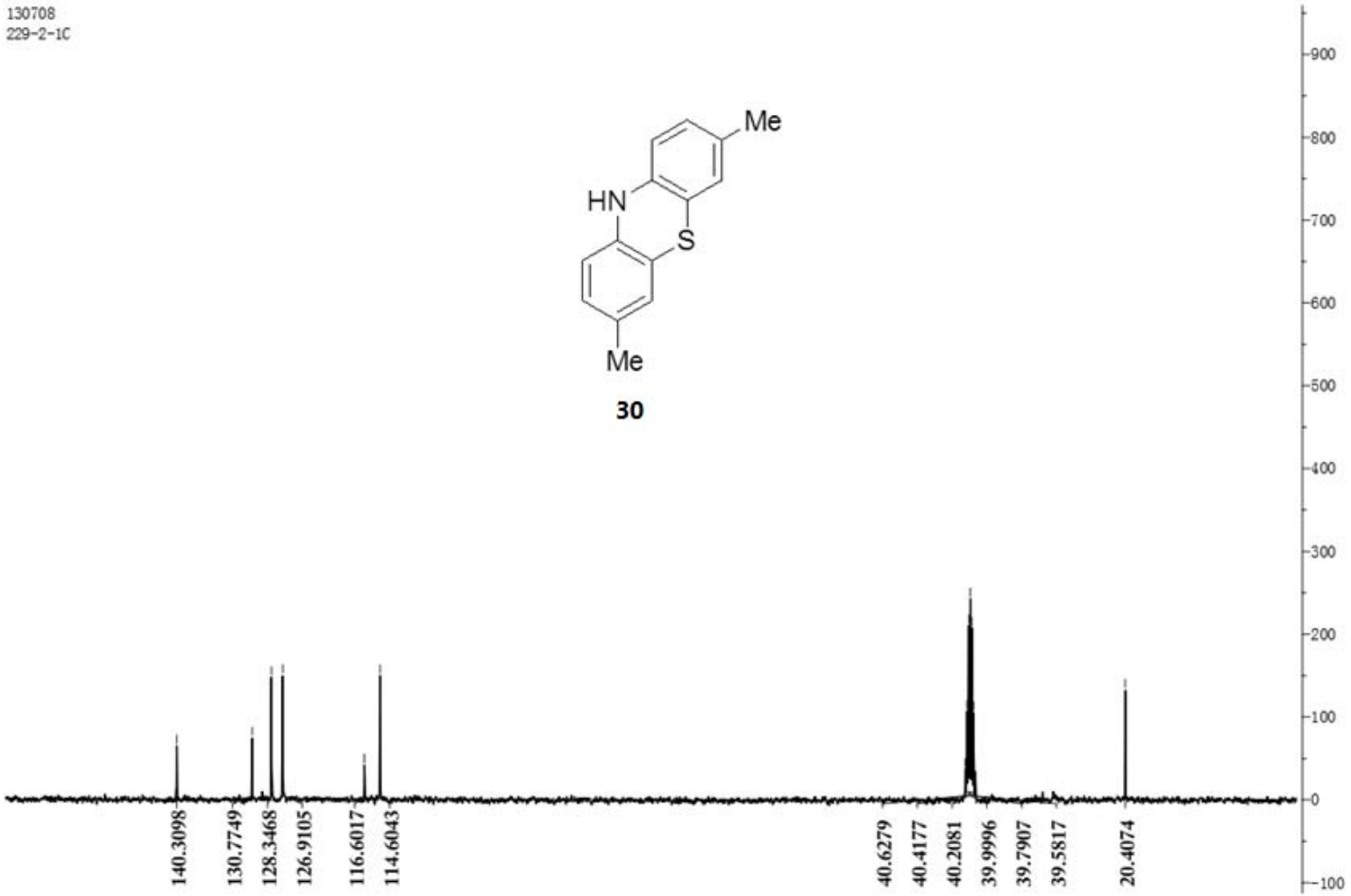
6.7538
6.7156
6.5654
6.5454
3.3536
2.5044
2.5001
2.4957
2.1121


130708
229-2-1C
Me
HN
S
Me
30
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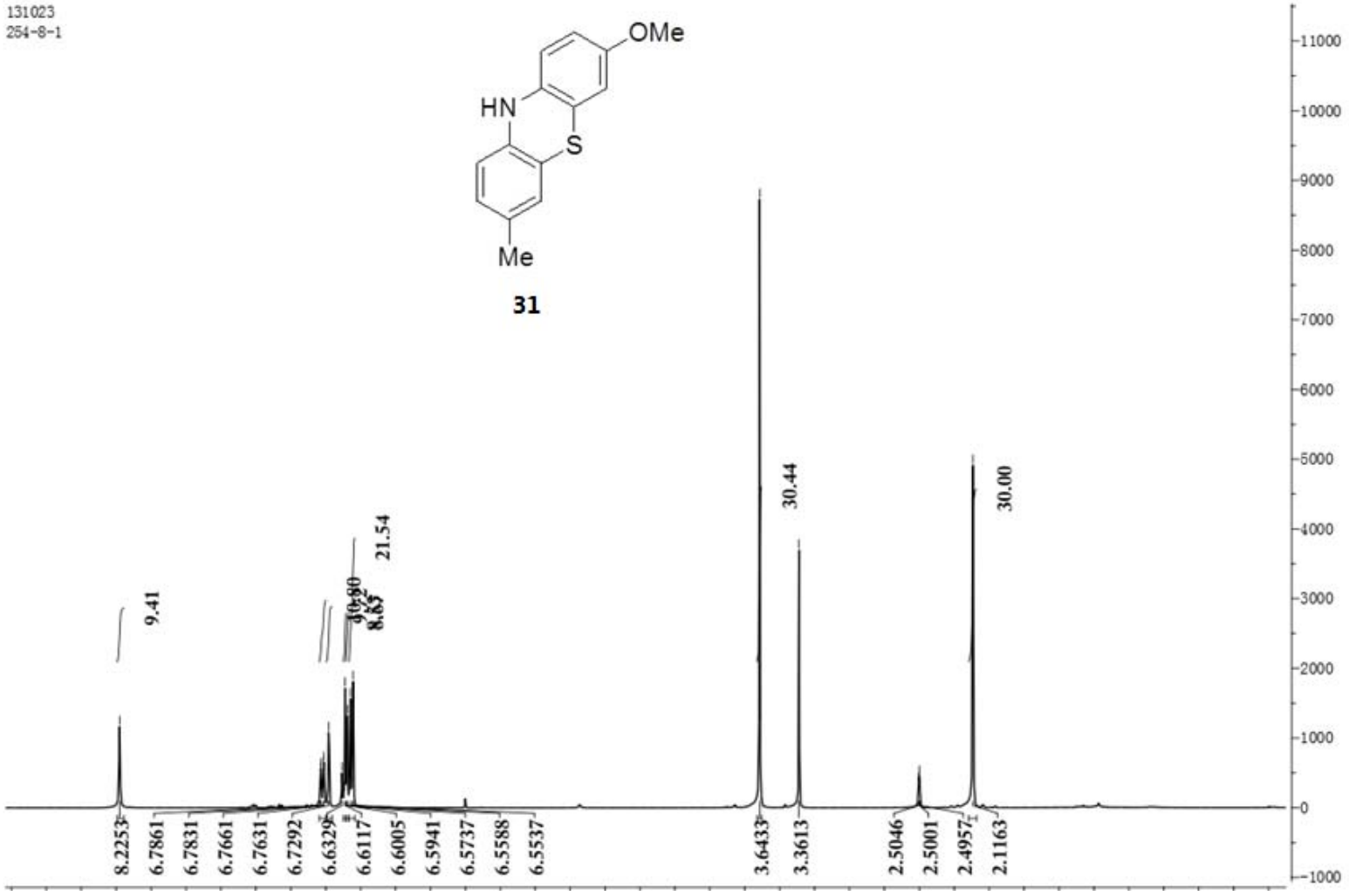
130.7749
128.3468
126.9105
116.6017
114.6043
40.6279
40.4177
40.2081
39.9996
39.7907
39.5817
20.4074
900
800
700
600
500
400
300
200
100
0
-100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254-8-1
OMe
HN
S
Me
31
9.41
21.54
30.44
30.00
8.2253
6.7861
6.7831
6.7661
6.7631
6.7292
6.6117
6.6005
6.5941
6.5737
6.5588
6.5537
3.6433
3.3613
2.5046
2.5001
2.4957
2.1163
11000
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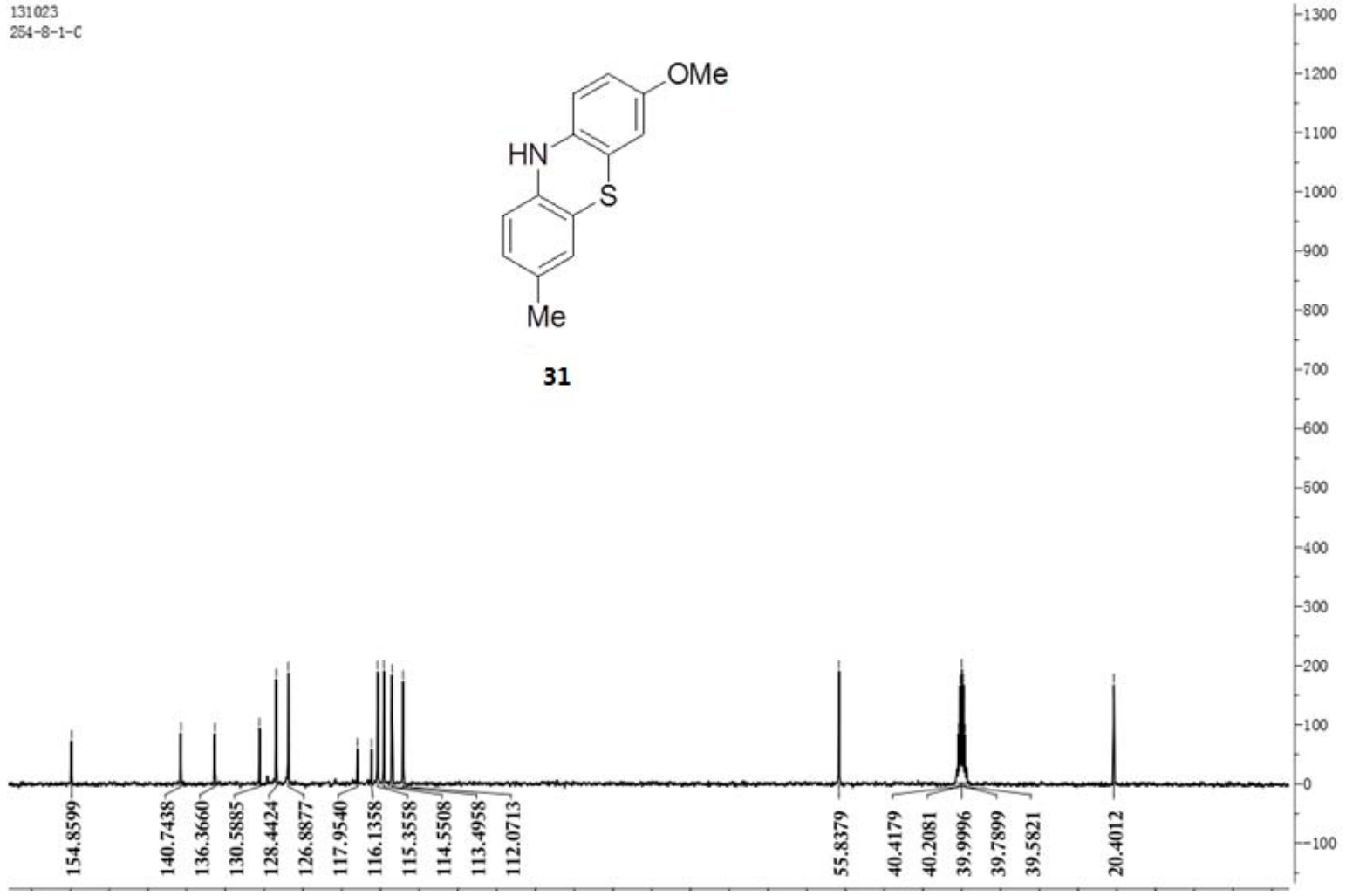
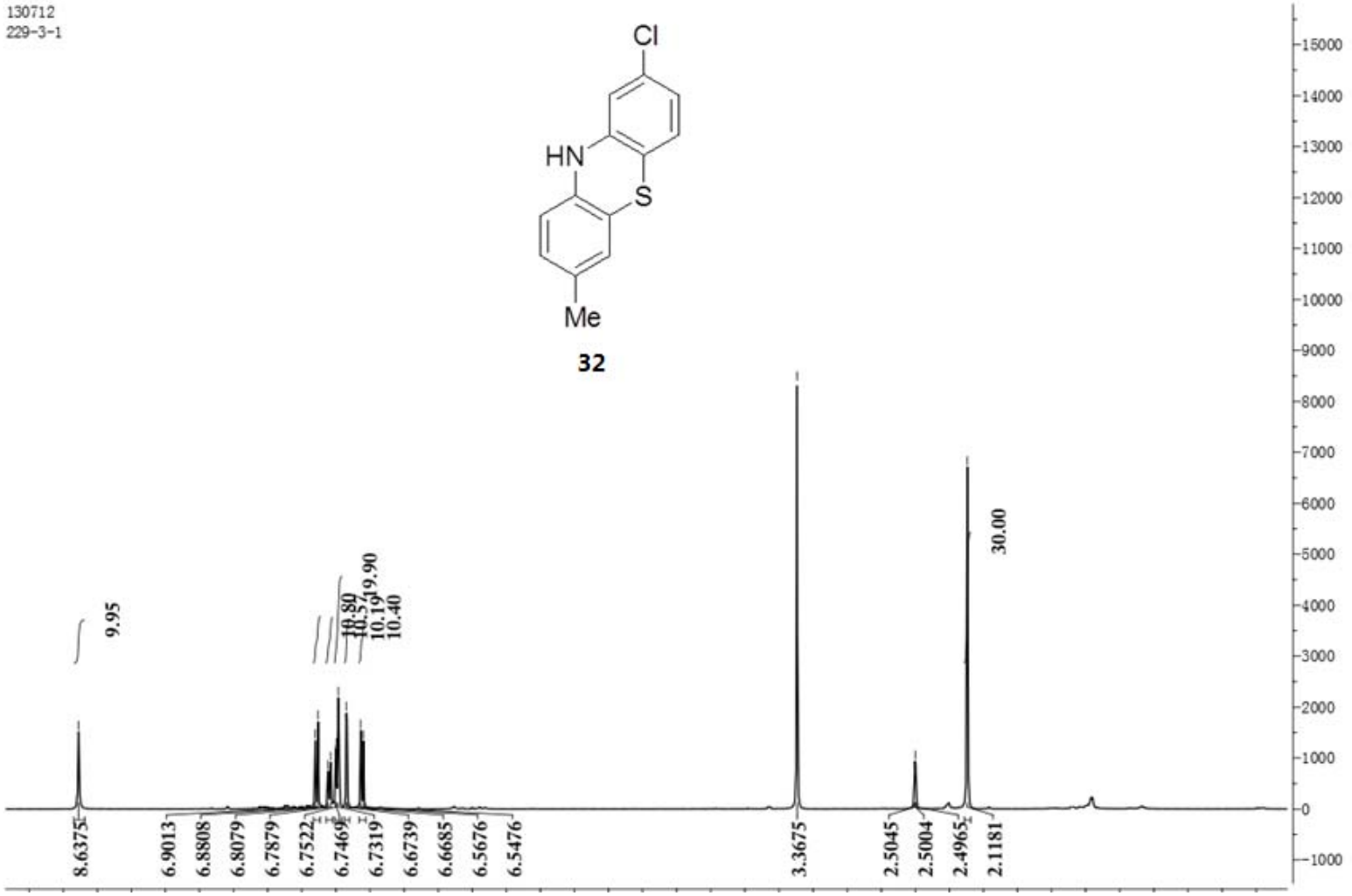
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

131023
254-8-1-C



31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Me
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9.90
10.40
30.00
8.6375
6.9013
6.8808
6.8079
6.7879
6.7522
6.7469
6.7319
6.6739
6.6685
6.5676
6.5476
3.3675
2.5045
2.5004
2.4965
2.1181
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14000
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12000
11000
10000
9000
8000
7000
6000
5000
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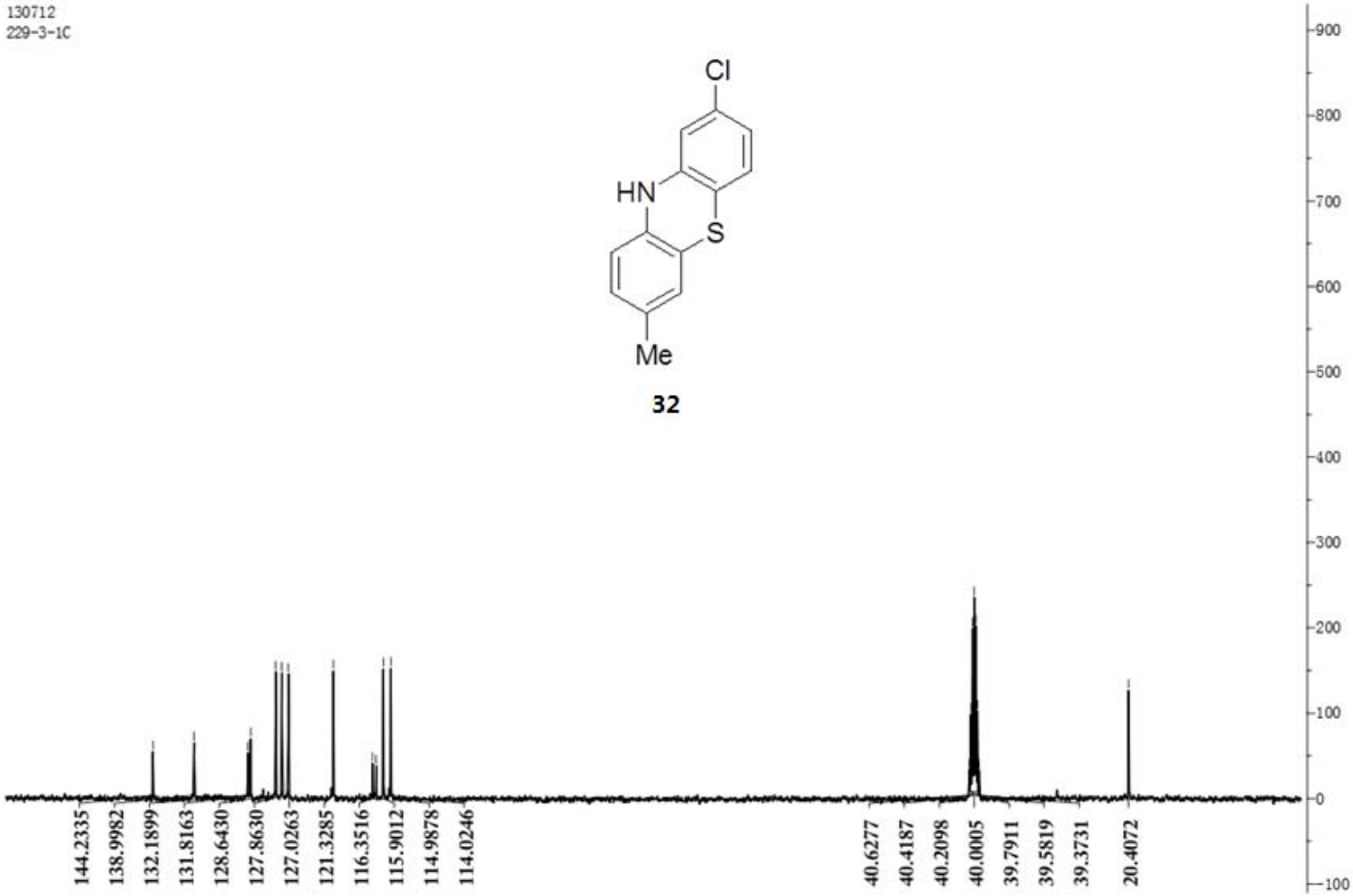
3000
2000
1000
0
-1000


130712
229-3-1C
Cl
HN
S
Me
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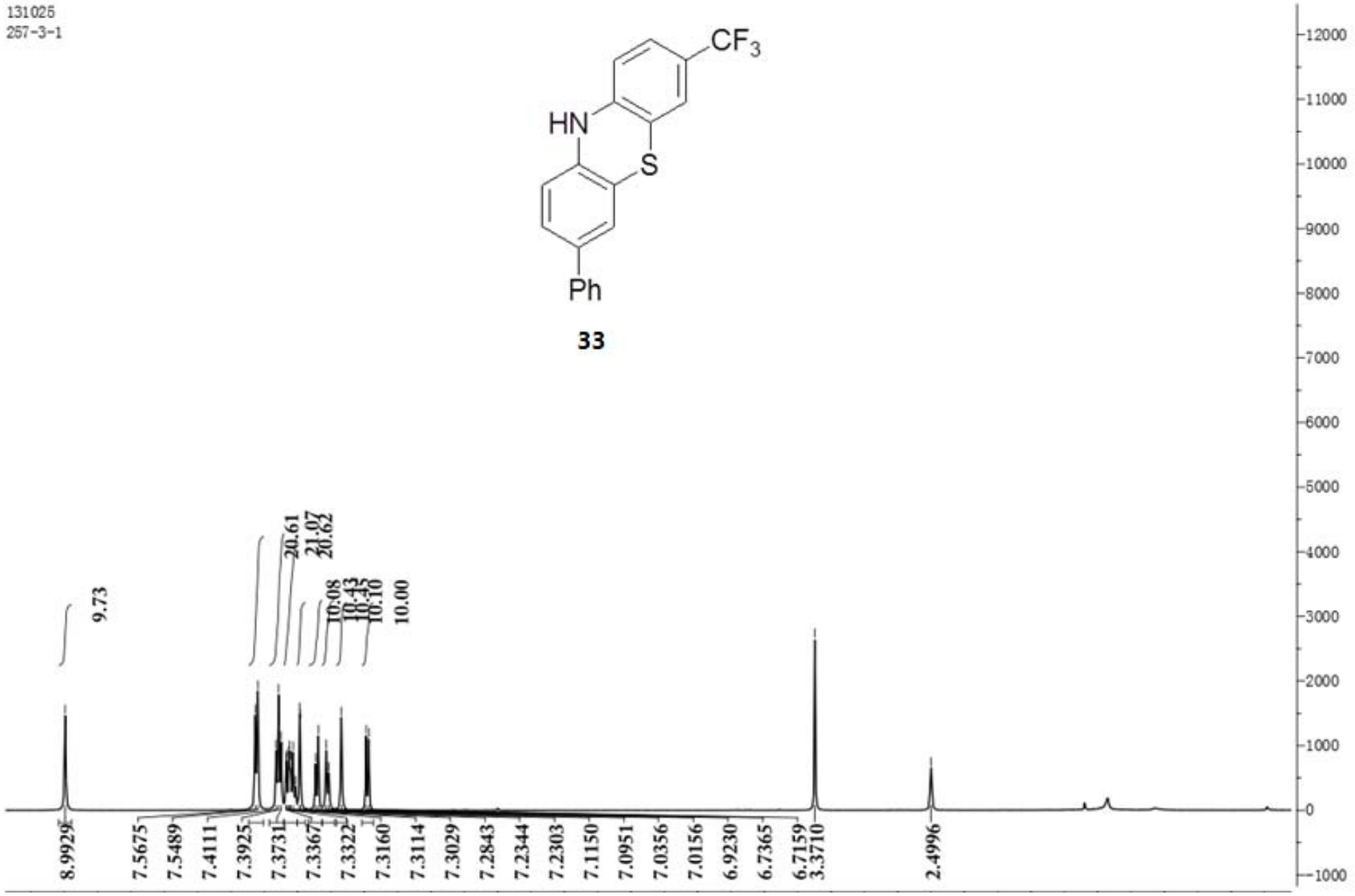
132.1899
131.8163
128.6430
127.8630
127.0263
121.3285
116.3516
115.9012
114.9878
114.0246
40.6277
40.4187
40.2098
40.0005
39.7911
39.5819
39.3731
20.4072
900
800
700
600
500
400
300
200
100
0
-100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10.43
10.45
10.10
10.00
8.9929
7.5675
7.5489
7.4111
7.3925
7.3731
7.3367
7.3322
7.3160
7.3114
7.3029
7.2843
7.2344
7.2303
7.1150
7.0951
7.0356
7.0156
6.9230
6.7365
6.7159
3.3710
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11000
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9000
8000
7000
6000
5000
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1000
0
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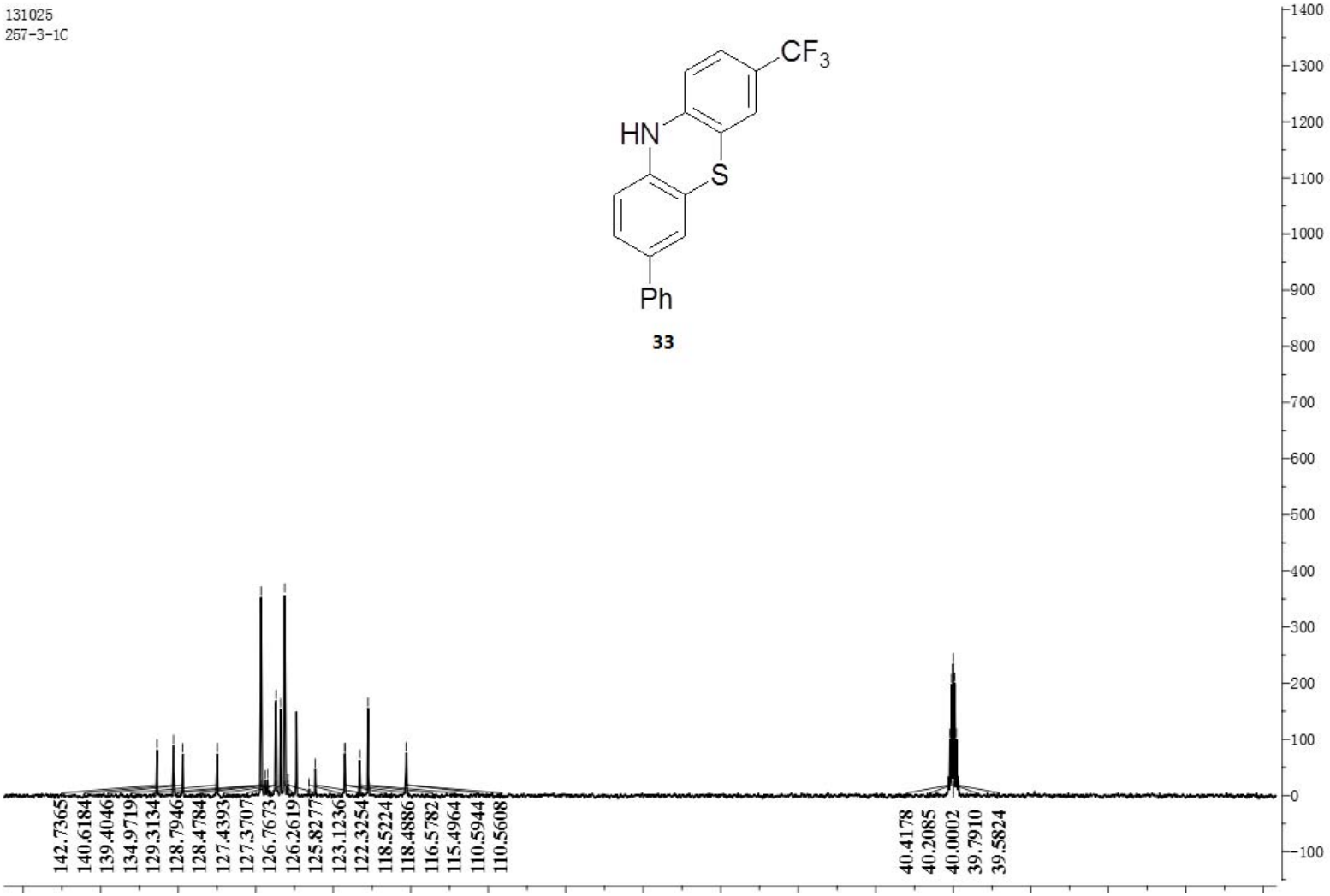
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128.4784
127.4393
127.3707
126.7673
126.2619
125.8277
123.1236
122.3254
118.5224
118.4886
116.5782
115.4964
110.5944
110.5608
40.4178
40.2085
40.0002
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39.5824
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1300
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1100
1000
900
800
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600
500
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200
100
0
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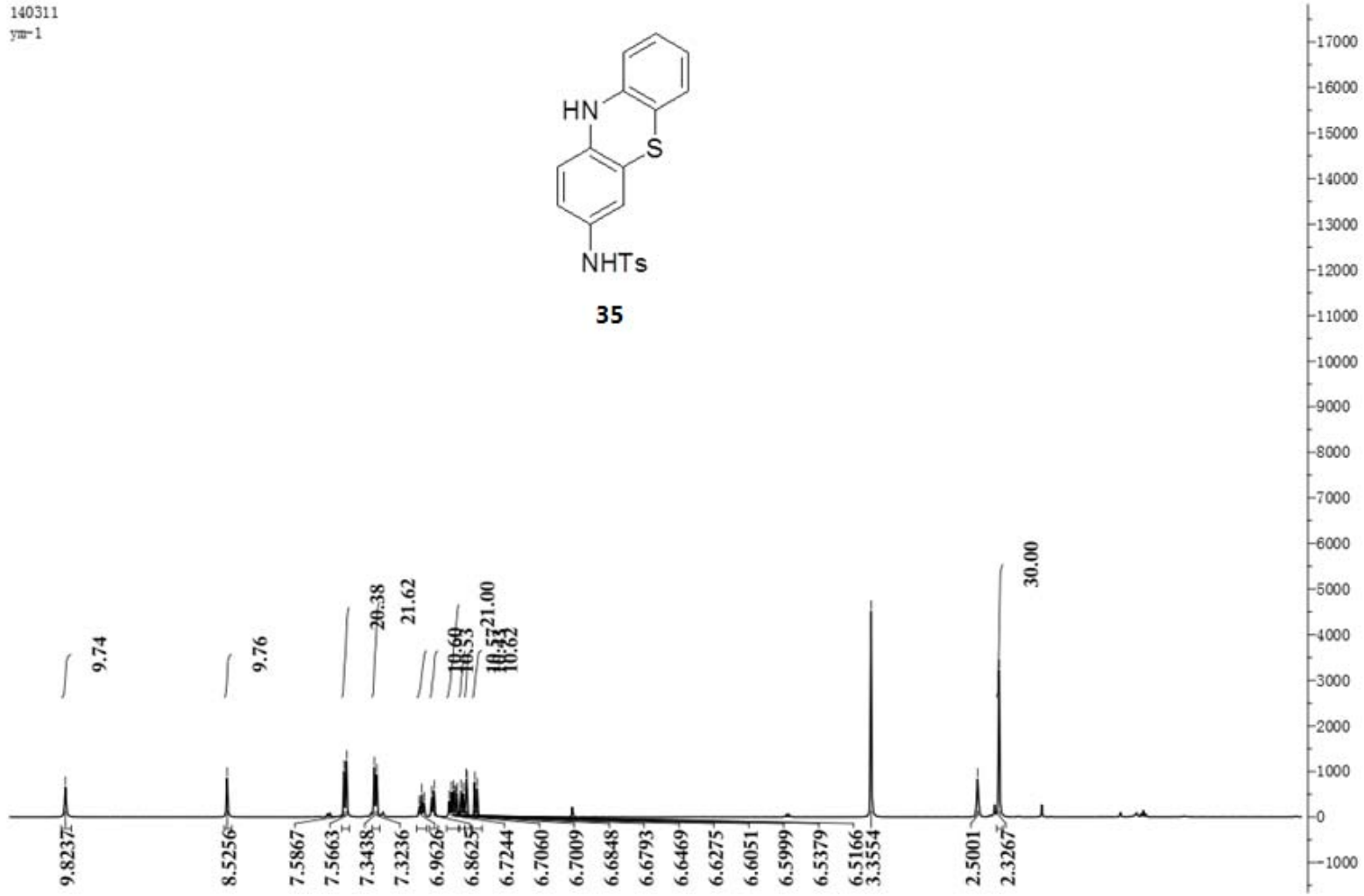
140311
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30.00
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7.5867
7.5663
7.3438
7.3236
6.9626
6.8625
6.7244
6.7060
6.7009
6.6848
6.6793
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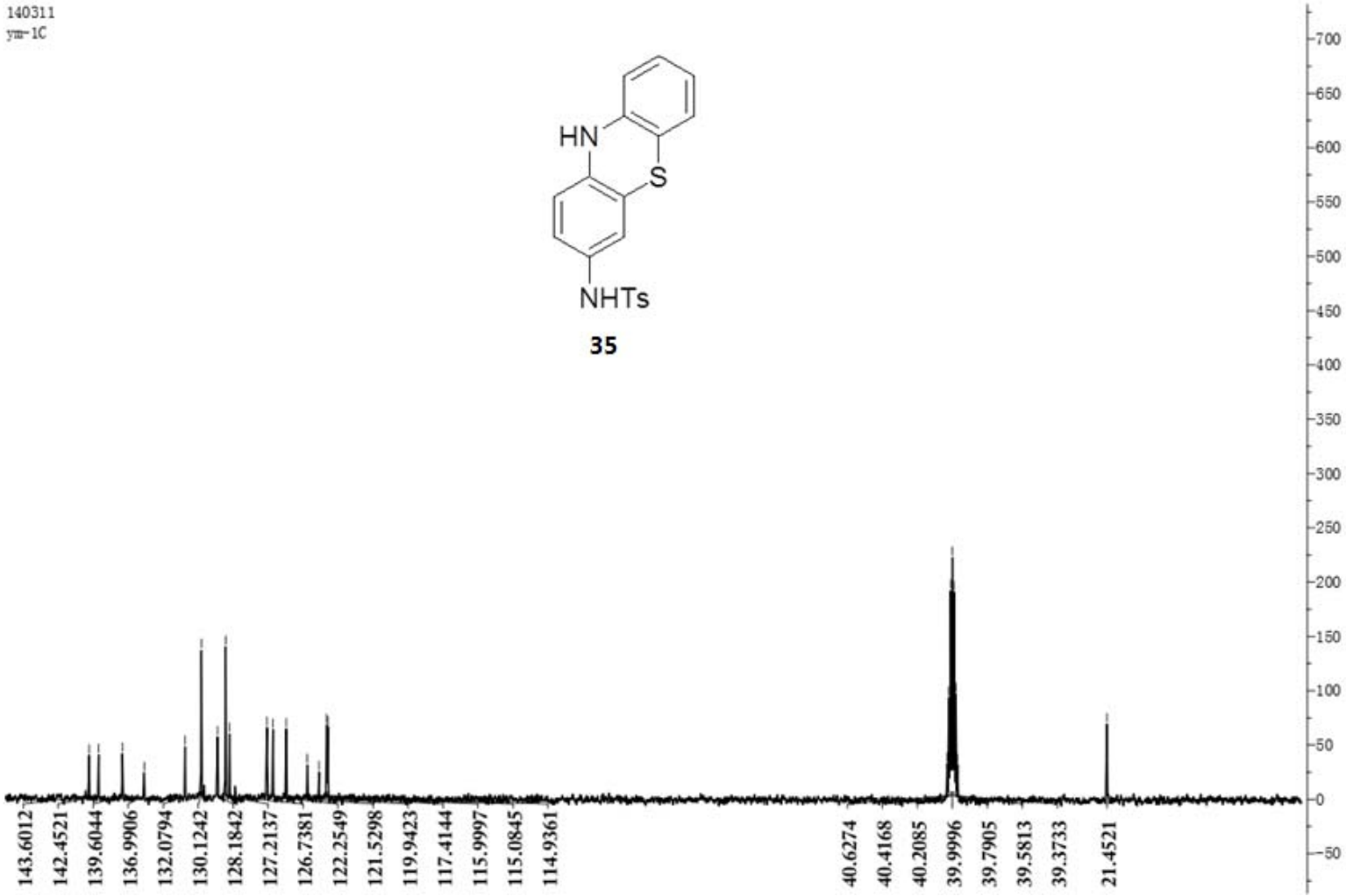
6.5379
6.5166
3.3554
2.5001
2.3267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132.0794
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126.7381
122.2549
121.5298
119.9423
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115.9997
115.0845
114.9361
40.6274
40.4168
40.2085
39.9996
39.7905
39.5813
39.3733
21.4521
700
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50