Electronic Supplementary Information

Palladium on Carbon-catalyzed Synthesis of 2- and 2,3-Substituted Indoles under Heterogeneous Conditions

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General methods

All reagents were obtained from commercial sources and used without further purification. Analytical thin-layer chromatography (TLC) was carried out on pre-coated Silica gel 60 F-254 plates (32–63 μm particle size) and visualized with UV light (254 nm). The 10% Pd/C was obtained from the N.E. Chemcat Co. Flash column chromatography was performed with Silica gel 60 (40–63 μm particle size, Merck & Co., Inc.) or Silica gel 60N (100–210 μm, Kanto Chemical Co., Inc.). ^{1}H and ^{13}C NMR spectra were recorded by a JEOL JNM EX-400 or AL-400 spectrometer (400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR) using $CDCl_3$ as the solvent. Chemical shifts (δ) are expressed in ppm based on internal standards (TMS for ^{1}H NMR and $CDCl_3$ for ^{13}C NMR). The electron impact (EI) and fast-atom bombardment (FAB) mass spectra were taken using a JEOL JMS-SX 102A instrument.

1-Boc- (**1b**),[1] 1-acetyl- (**1c**),[2] 1-Cbz- (**1d**),[3] and 1-tosyl-2-iodoanilines (**1e**)[2] were perepared according to the reported procedures. 1-tosyl-2-iodo-4-methoxycarbonylaniline (**1f**) was synthesized by the *N*-tosylation of 2-iodo-4-methoxycarbonylaniline using tosylchloride, *N*.*N*-dimethylpyridine and pyridine.

1-Tosyl-2-iodo-4-methoxycarbonylaniline (1f)

^{1}H NMR: δ 8.54 (s, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 2H),7.10 (br s, 1H), 3.88 (s, 3H), 2.38 (s, 3H); ^{13}C NMR: δ 164.9, 144.7, 141.4, 140.5, 135.5, 130.8, 129.8, 127.6, 127.4, 119.5, 89.9, 52.3, 21.6; MS (FAB, NBA) m/z 432 [$(M+H)^+$, 17%], 432 (17%); HRMS (FAB, NBA) Calcd for $C_{15}H_{15}NO_4SI$ [$(M+H)^+$] 431.9769. Found 431.9772.

General procedure of the LiCl-free Pd/C-catalyzed annulation of alkynes with *N*-Ts-2-iodoaniline (for Table 5)

A mixture of *N*-Ts-2-iodoaniline (0.250 mmol), alkyne (0.300 mmol), 10% Pd/C (8.0 mg, 3 mol % of *N*-Ts-2-iodoaniline) and NaOAc (22.5 mg, 0.275 mmol) in NMP (1.00 cm^3) in a 15 cm^3-test tube was sealed with a septum and the air inside was replaced with Ar by five vacuum/Ar (balloon) cycles. The mixture was stirred at 110–130 °C for 24 h using Chemist Plaza personal organic synthesizer (Shibata Scientific Technology, Ltd., Tokyo). The suspension was vigorously stirred with additional EtOAc (40 cm^3) and saturated NH_4Cl solution (10 cm^3), and filtered through a membrane filter (Millipore, Millex®-LH, 0.45 μm). The filtered aqueous layers were separated and the water layer was extracted with another EtOAc (20 cm^3). The EtOAc layer was combined with the filtered organic layer and washed with saturated NH_4Cl solution (20 cm^3 × 2), H_2O (5 cm^3 × 2), and brine (5 cm^3), and dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column

chromatography.

1-Boc-2,3-diphenylindole (2b)[4]

^{1}H NMR: δ 8.32 (d, *J* = 8.4 Hz, 1H), 7.57 (d, *J* = 7.9 Hz, 1H), 7.36 (t, *J* = 7.9 Hz, 1H), 7.25–7.18 (m, 11H), 1.2 (s, 9H); ^{13}C NMR: δ 150.2, 136.6, 135.7, 133.9, 133.4, 130.3, 130.3, 130.1, 129.3, 128.1, 127.6, 127.4, 126.6 , 124.7, 123.0, 121.9, 119.6, 115.2, 83.2, 27.4; MS (EI) m/z 369 (M^+, 20%), 313 (45%), 269 (100%), 57 (15%); HRMS (EI) Calcd for $C_{25}H_{23}NO_2$ (M^+) 369.1729. Found 369.1735.

1-Acetyl-2,3-diphenylindole (2c)[5]

^{1}H NMR: δ 8.47 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.55 (td, *J* = 8.4, 0.8 Hz, 1H), 7.42–7.20 (m, 12H), 2.0 (s, 3H) ; ^{13}C NMR: δ 172.3, 137.5, 137.8, 133.7, 131.5, 130.8, 130.0, 129.3, 129.0, 127.6, 126.2, 124.5, 124.5, 124.1, 120.3, 116.9, 28.6; MS (EI) m/z 311 (M^+, 41%), 269 (100%), 84 (58%); HRMS (EI) Calcd for $C_{22}H_{17}NO$ (M^+) 311.1313. Found 311.1310.

2,3-Dipheny-1-tosylindole (2e)[6]

^{1}H NMR: δ 8.41 (d, *J* = 8.8 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.41 (dd, *J* = 7.4, 7.4 Hz, 1H), 7.34–7.17 (m, 11H), 7.09–7.06 (m, 4H); ^{13}C NMR: δ;144.5, 137.2, 136.8, 135.2, 132.5, 132.0, 130.8, 130.4, 129.7, 129.2, 128.4, 128.1, 127.2, 126.9, 126.8, 125.1, 124.7, 124.1, 120.0, 116.1, 21.4; MS (EI) m/z 423 (M^+, 39%), 268 (100%); HRMS (EI) Calcd for $C_{27}H_{21}NO_2S$ (M^+) 423.1288. Found 423.1293.

2,3-Dipentyl-1-tosylindole (2f)

^{1}H NMR: δ 8.17 (dd, *J* = 7.2, 1.6 Hz, 1H), 7.50 (d, *J* = 8.6 Hz, 2H), 7.36 (dd, *J* = 6.8, 1.6 Hz, 1H), 7.24–7.17 (m, 2H), 7.08 (d, *J* = 8.6 Hz, 2H), 2.94 (t, *J* = 7.8 Hz, 2H), 2.57 (t, *J* = 7.8 Hz, 2H), 2.26 (s, 3H), 1.70 (m, 2H), 1.51 (m, 2H), 1.36–1.20 (m, 8H), 0.91–0.82 (m, 6H); ^{13}C NMR: δ 144.1, 137.8, 136.9, 135.9, 131.1, 129.4, 126.1, 123.7, 123.3, 121.8, 118.5, 115.4, 31.8, 31.6, 30.7, 29.5, 26.5, 24.1, 22.4, 22.4, 21.3, 14.0, 13.9; MS (EI) m/z 411 (M^+, 100%), 256 (41%), 91 (25%); HRMS (EI) Calcd for $C_{25}H_{33}NO_2S$ (M^+) 411.2232. Found 411.2239.

2,3-Diethy-1-tosylindole (2g)

^{1}H NMR: δ 8.17 (dd, *J* = 7.2, 1.6 Hz, 1H), 7.55 (d, *J* = 8.4 Hz, 2H), 7.40 (dd, *J* = 6.8, 2.0 Hz, 1H), 7.27–7.20 (m, 2H), 7.13 (d, *J* = 8.4 Hz, 2H), 2.99 (q, *J* = 7.6 Hz, 2H), 2.61 (q, *J* = 7.6 Hz, 2H), 1.30 (t *J* = 7.6 Hz, 3H), 1.15 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR: δ 144.2, 138.5, 136.8, 136.2, 130.6, 130.0, 126.1, 123.8, 123.2, 122.5, 118.4, 115.2, 21.4, 19.7, 17.4, 15.7, 14.7; MS (EI) m/z 327 (M^+, 54%), 172 (100%), 91 (20%); HRMS (EI) Calcd for $C_{19}H_{21}NO_2S$ (M^+) 327.1293.

Found 372.1298.

2,3-Dipropy-1-tosylindole (2h)[6]

^{1}H NMR: δ 8.15 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.37 (dd, *J* = 8.0, 2.4 Hz, 1H), 7.25–7.18 (m, 2H), 7.10 (d, *J* = 8.4 Hz, 2H), 2.93 (t, *J* = 7.6 Hz, 2H), 2.56 (t, *J* = 7.6 Hz, 2H), 2.27 (s, 3H), 1.73 (q, *J* = 7.6 Hz, 2H), 1.55 (q, *J* = 7.6 Hz, 2H), 0.98 (t, *J* = 7.6 Hz, 3H), 0.85 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR: δ 144.2, 137.7, 136.9, 135.9, 131.1, 129.4, 126.0, 123.8, 123.3, 121.7, 118.5, 115.4, 28.4, 26.2, 24.2, 23.0, 21.4, 14.0, 13.8 ; MS (EI) m/z 355 (M^+, 100%), 326 (46%), 200 (85%), 91 (35%); HRMS (EI) Calcd for $C_{21}H_{25}NO_2S$ (M^+) 355.1606. Found 355.1599.

2-Phenyl-1-tosylindole (2i)[7]

^{1}H NMR: δ 8.31 (d, *J* = 8.4 Hz, 1H), 7.50–7.24 (m, 10H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.53 (s, 1H), 2.27 (s, 3H); ^{13}C NMR: δ 144.4, 142.3, 138.2, 137.0, 134.8, 132.3, 131.0, 130.5, 129.4, 129.1, 127.4, 127.4, 126.8, 124.6, 124.2, 120.6, 116.6, 113.3, 21.5, 21.4; MS (EI) m/z 361 (M^+, 88%), 206 (100%); HRMS (EI) Calcd for $C_{22}H_{19}NO_2S$ (M^+) 361.1137. Found 361.1144

2-Butyl-1-tosylindole (2j)[7]

^{1}H NMR: δ 8.16 (d, *J* = 8.8 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.0 Hz), 7.25–7.15 (m, 4H), 6.37 (s, 1H), 2.98 (t, *J* = 7.6 Hz, 2H), 2.31 (s, 3H), 1.72 (m, 2H), 1.44 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H) ; ^{13}C NMR: δ 144.5, 142.5, 137.2, 136.3, 129.8, 129.7, 126.2, 123.7, 123.4, 120.0, 114.8, 108.6, 31.0, 28.7, 22.4, 21.5, 13.9; MS (EI) m/z 327 (M^+, 80%), 130 (100%), 91 (43%); HRMS (EI) Calcd for $C_{19}H_{21}NO_2S$ (M^+) 327.1293. Found 327.1298

3-Butyl-1-tosylindole[8]

^{1}H NMR: δ 7.97 (d, *J* = 8.4 Hz, 1H), 7.73 (d, *J* = 8.8 Hz, 2H), 7.47 (d, *J* = 8.0 Hz), 7.30–7.18 (m, 5H), 2.64 (t, *J* = 7.6 Hz, 2H), 2.33 (s, 3H), 1.66 (m, 2H), 1.38 (m, 2H), 0.94 (t, *J* = 7.6 Hz, 3H); MS (EI) m/z 327 (M^+, 100%), 130 (83%), 91 (63%); HRMS (EI) Calcd for $C_{19}H_{21}NO_2S$ (M^+)327.1293. Found 327.1298.

3-Butyl-2-phenyl-1-tosylindole (2k)

m.p. 111–112 ºC; ^{1}H NMR: δ 8.32 (d, *J* = 8.0 Hz, 1H), 7.48–7.24 (m, 10H), 7.05 (d, *J* = 8.0 Hz, 2H), 2.45 (t, *J* = 7.6 Hz, 2H), 2.29 (s, 3H), 1.41 (m, 2H), 1.16 (m, 2H), 0.75 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR: δ 144.3, 137.4, 136.5, 135.2, 131.6, 131.3, 131.0, 129.1, 128.4, 127.4, 126.8, 124.7, 124.5, 123.7, 119.3, 116.2, 31.9, 23.9, 22.4, 21.5, 13.7; MS (EI) m/z 403 (M^+, 100%), 248 (60%), 206 (73%); HRMS (EI) Calcd for $C_{25}H_{25}NO_2S$ (M^+) 403.1606. Found 403.1602.

2-Butyl-3-phenyl-1-tosylindole

m.p. 100–101 ºC; ^{1}H NMR: δ 8.21 (d, *J* = 8.4 Hz, 1H), 7.62 (d, *J* = 8.8 Hz, 2H), 7.47–7.17 (m, 10H), 2.98 (t, *J* = 8.0 Hz), 2.33 (s, 3H), 1.71 (m, 2H), 1.28 (m, *J* = 8.0, 8.0 Hz, 2H), 0.82 (t, *J* = 8.0 Hz, 3H); ^{13}C NMR: δ 144.5, 138.7, 136.7, 136.0, 133.3, 130.7, 130.0, 130.0, 129.7, 128.6, 127.4, 126.3, 124.3, 123.7, 119.3, 115.3, 33.4, 26.6, 22.5, 21.5, 13.7; MS (EI) m/z 403 (M^+, 54%), 248 (15%), 206 (100%); HRMS (EI) Calcd for $C_{25}H_{25}NO_2S$ (M^+) 403.1606. Found 403.1597.

3-Methyl-2-phenyl-1-tosylindole (2l)

m.p. 163–164 ºC; ^{1}H NMR: δ 8.32 (d, *J* = 8.4 Hz, 1H), 7.44–7.30 (m, 10H), 7.04 (d, *J* = 8.0 Hz, 2H), 2.28 (s, 3H), 2.03 (s, 3H); ^{13}C NMR: δ 144.3, 137.2, 136.6, 135.1, 131.7, 131.5, 131.3, 129.1, 128.3, 127.4, 126.8, 124.9, 123.9, 119.7, 119.0, 116.2, 21.5, 9.4; MS (EI) m/z 361 (M^+, 52%), 206 (100%); HRMS (EI) Calcd for $C_{22}H_{19}NO_2S$ (M^+) 361.1137. Found 361.1146.

2-Methyl-3-phenyl-1-tosylindole[9]

^{1}H NMR: δ 8.26 (d, *J* = 8.0 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.47–7.20 (m, 10H), 2.59 (s, 3H), 2.36 (s, 3H); ^{13}C NMR: δ 144.7, 136.4, 136.3, 133.1, 133.1, 120.0, 130.0, 129.9, 128.5, 127.3, 126.4, 124.2, 123.5, 122.5, 119.2, 114.5, 21.6, 13.5; MS (EI) m/z 361(M^+, 41%), 206 (100%); HRMS (EI) Calcd for $C_{22}H_{19}NO_2S$ (M^+) 361.1137. Found 361.1144.

2,3-Dibuty-1-tosylindole (2m)

^{1}H NMR: δ 8.16 (dd, *J* = 7.6, 2.0 Hz, 1H), 7.50 (d, *J* = 8.2 Hz, 2H), 7.37 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.25–7.18 (m, 2H), 7.10 (d, *J* = 8.2 Hz, 2H), 2.93 (t, *J* = 7.6 Hz, 2H), 2.56 (t, *J* = 7.6 Hz, 2H), 2.27 (s, 3H), 1.73 (q, *J* = 7.6 Hz, 2H), 1.55 (q, *J* = 7.6 Hz, 2H), 0.98 (t, *J* = 7.6 Hz, 3H), 0.85 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR: δ 144.2, 137.8, 137.0, 136.0, 131.1, 129.5, 126.1, 123.8, 123.3, 121.8, 118.5, 115.5, 33.1, 32.0, 26.3, 23.9, 22.7, 22.5, 21.4, 13.9, 13.8; MS (EI) m/z 383 (M^+, 100%), 228 (30%), 91 (35%); HRMS (EI) Calcd for $C_{23}H_{29}NO_2S$ (M^+) 383.1919. Found 383.1926.

2-(4-Methoxylphenyl)-1-tosylindole (2n)[7]

^{1}H NMR: δ 8.31 (d, *J* = 8.8 Hz, 1H), 7.43–7.35 (m, 3H), 7.32 (dd, *J* = 8.4, 1.5 Hz, 1H), 7.27–7.23 (m, 3H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.95 (d, *J* = 8.8 Hz, 2H), 6.48 (s, 1H), 3.89 (s, 3H), 2.28 (s, 3H); ^{13}C NMR: δ 160.0, 144.4, 142.0, 138.2, 134.7, 131.6, 130.6, 129.1, 126.8, 124.7, 124.5, 124.2, 120.5, 116.7, 113.0, 112.9, 55.3, 21.5; MS (EI) m/z 377 (M^+, 39%), 222 (100%); HRMS (EI) Calcd for $C_{22}H_{19}NO_3S$ (M^+) 377.1086. Found 377.1096.

2-(3-Methylphenyl)-1-tosylindole (2o)

m.p. 104–105 ºC; ^{1}H NMR: δ 8.30 (d, *J* = 8.8 Hz, 1H), 7.43 (d, *J* = 7.6 Hz, 1H), 7.36–7.23 (m, 8H), 7.04 (d, *J* = 8.0 Hz, 2H), 6.52 (s, 1H), 2.41 (s, 3H), 2.29 (s, 3H); ^{13}C NMR: δ 144.4, 142.3, 138.2, 137.0, 134.8, 132.3, 131.0, 130.5, 129.4, 129.1, 127.4, 127.4, 126.8, 124.6, 124.2, 120.6, 116.6, 113.3, 21.5, 21.4; MS (EI) m/z 361 (M^+, 88%) ,206 (100%); HRMS (EI) Calcd for $C_{22}H_{19}NO_2S$ (M^+) 361.1137. Found 361.1144.

2-(4-Methylphenyl)-1-tosylindole (2p)[10]

^{1}H NMR: δ 8.30 (d, *J* = 8.4 Hz, 1H), 7.42–7.38 (m, 3H), 7.35–7.21(m, 7.03, 6H), 7.01 (d, *J* = 8.0 Hz, 2H), 6.49 (s, 1H), 2.43 (s, 3H), 2.26 (s, 3H); ^{13}C NMR: δ 144.4, 142.3, 138.6, 138.2, 134.7, 130.6, 130.2, 129.5, 129.1, 128.2, 126.8, 124.6, 124.2, 120.5, 116.6, 113.2, 21.5, 21.4; MS (EI) m/z 361 (M^+, 60%), 206 (100%); HRMS (EI) Calcd for $C_{22}H_{19}NO_2S$ (M^+) 361.1137. Found 361.1130.

2-(1-Naphthyl)-1-tosylindole (2q)[10]

^{1}H NMR: δ 8.39 (d, *J* = 8.8 Hz, 1H),7.93 (d, *J* = 8.4 Hz, 1H), 7.86 (d, *J* =7.6 Hz, 1H), 7.63(d, *J* = 8.4 Hz, 1H), 7.54–7.39 (m, 5H), 7.33–7.29 (m, 2H), 7.24 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 6.64(s, 1H), 2.23 (s, 3H); ^{13}C NMR: δ 144.5, 138.8, 137.6, 135.3, 133.4, 133.0, 130.0, 129.8, 129.5,129.3, 129.2, 128.0, 126.9, 126.2, 126.1, 125.7, 124.8, 124.4, 123.9, 120.8, 115.8, 113.7, 21.4; MS (EI) m/z 397 (M^+, 41%), 242 (100%), 91(44%); HRMS (EI) Calcd for $C_{25}H_{19}NO_2S$ (M^+) 397.1137. Found 397.1129.

2-(6-Methoxynaphth-2-yl)-1-tosylindole (2r)

m.p. 193–194 ºC; ^{1}H NMR: δ 8.33 (d, *J* = 7.6 Hz, 1H), 7.79–7.75 (m, 3H), 6.65 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.45 (d, *J* = 7.6 Hz, 1H), 7.36 (td, *J* = 8.4, 1.2 Hz, 1H), 7.29–7.24 (m, 3H), 7.21–7.18 (m, 2H), 7.00 (d, *J* = 7.6 Hz, 2H), 6.60 (s, 1H), 3.96 (s, 3H), 2.27 (s, 3H); ^{13}C NMR: δ 158.3, 144.5, 142.3, 138.3, 134.7, 134.5, 130.7, 129.7, 129.1, 129.1, 128.7, 128.1, 127.8, 126.8, 125.6, 124.7, 124.3, 120.6, 119.1, 116.7, 113.7, 105.8, 55.4, 21.5; MS (EI) m/z 427 (M^+, 52%), 272 (100%); HRMS (EI) Calcd for $C_{26}H_{21}NO_3S$ (M^+) 427.1242. Found 427.1249.

2-(1-Cyclehexene-1-yl)-1-tosylindole (2s)[11]

^{1}H NMR: δ 8.17 (d, *J* = 8.0 Hz, 1H), 7.51 (d, *J* = 8.4 Hz), 7.37 (d, *J* = 7.8 Hz, 1H), 7.26 (dd, *J* = 7.8, 7.8 Hz, 1H), 7.19 (dd, *J* = 8.0, 8.0 Hz, 1H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.34 (s, 1H), 5.74 (s, 1H), 2.44 (m, 2H), 2.23 (m, 2H), 1.81 (t, *J* = 5.6 Hz, 2H), 1.72 (t, *J* = 5.6 Hz, 2H) ; ^{13}C NMR: δ 144.9, 144.3, 137.7, 135.1, 132.4, 130.7, 129.4, 129. 1, 126.7, 124.2, 123.9, 120.4, 116.0, 110.9,

30.5, 25.6, 22.7, 21.9, 21.5; MS (EI) m/z 351 (M^+, 100%), 287 (33%), 196 (59%); HRMS (EI) Calcd for $C_{21}H_{21}NO_2S$ (M^+) 351.1298. Found 351.1293.

Methyl 2,3-dipentyl-1-tosylindole-5-carboxylate (2t)

^{1}H NMR: δ 8.20 (d, *J* = 8.8 Hz, 1H), 8.09 (d, *J* = 1.4 Hz, 1H), 7.93 (dd, *J* = 8.8 Hz, 1.4 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 3.92 (s, 3H), 2.94 (t, *J* = 7.8 Hz, 2H), 2.61 (t, *J* = 7.8 Hz, 2H), 2.30 (s, 3H), 1.69 (brd, 2H), 1.53 (td, *J* = 7.8, 7.8 Hz, 2H), 1.37–1.26 (m, 8H), 0.92-0.83 (m, 6H) ; ^{13}C NMR: δ 167.4, 144.6, 139.6, 139.2, 135.8, 130.9, 129.6, 126.1, 125.3, 125.1, 121.9, 120.6, 115.0, 52.0, 31.9, 31.6, 30.6, 29.6, 29.6, 26.6, 24.1, 22.4, 22.4, 21.5, 14.0, 14.0; MS (EI) m/z 469 (M^+, 100%) , 314 (37%); HRMS (EI) Calcd for $C_{27}H_{35}NO_4S$ (M^+) 469.2280. Found 469.2287.

2,3-Diphenylindole (3)[6]

^{1}H NMR: δ 8.13 (s, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.44–7.18 (m, 13H); ^{13}C NMR: δ 135.8, 135.0, 134.0, 132.6, 130.1, 128.7, 128.6, 128.5, 128.2, 127.6, 126.2, 122.7, 122.6, 119.7, 115.0, 110.9; MS (EI) m/z 269 (M^+, 100%), 84 (33%); HRMS (EI) Calcd for $C_{20}H_{15}N$ (M^+) 269.1205. Found 269.1199.

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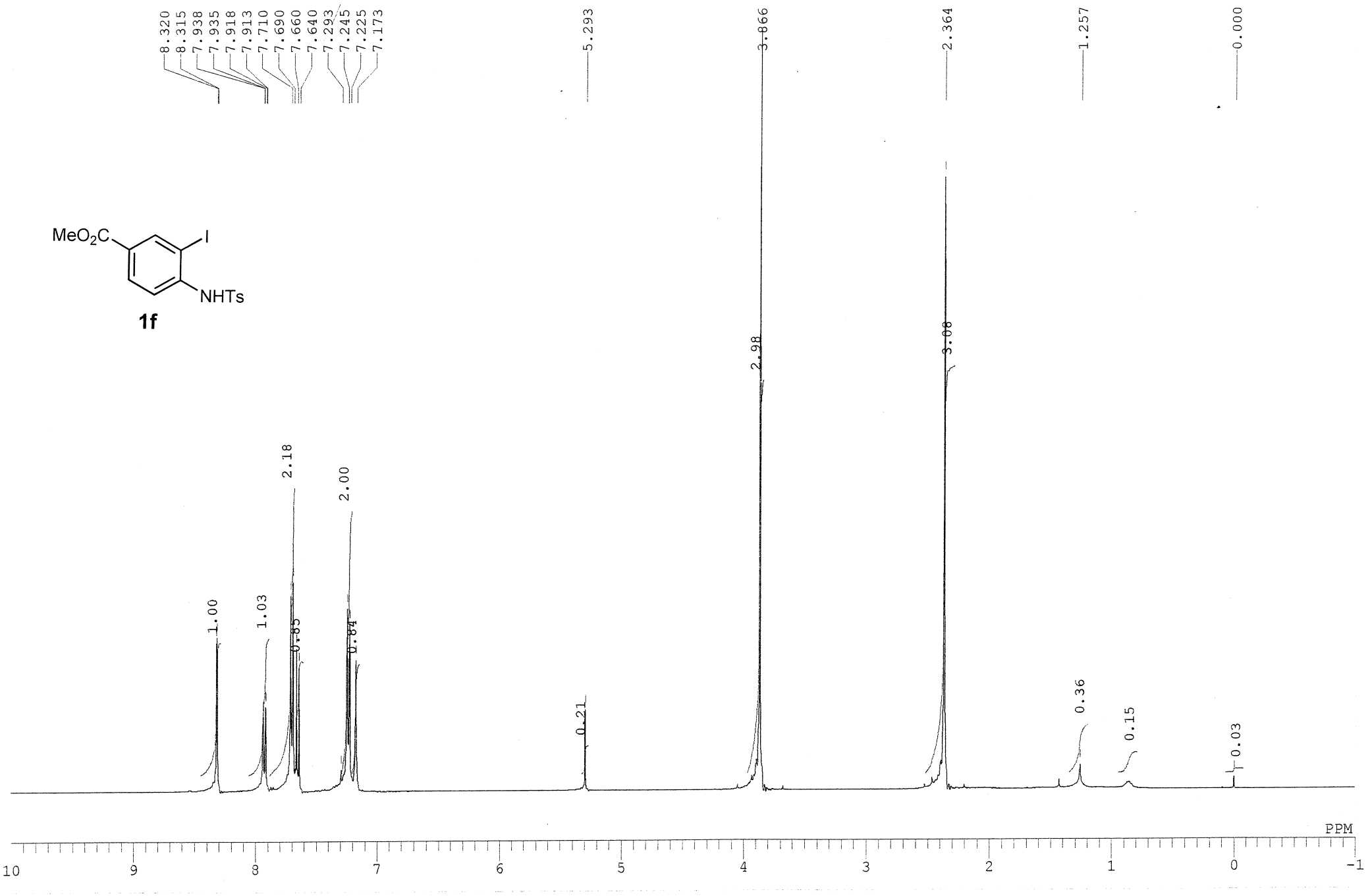
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7.710
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7.640
7.293
7.245
7.225
7.173
5.293
3.866
2.364
1.257
0.000
MeO2C
I
NHTs
1f
1.00
1.03
2.18
0.85
2.00
0.84
0.21
2.98
3.00
0.36
0.15
0.03
PPM
10
9
8
7
6
5
4
3
2
1
0
-1

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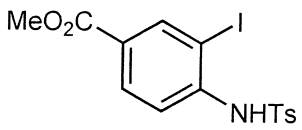

1f

DFILE C:\
COMNT azu
DATIM Tue
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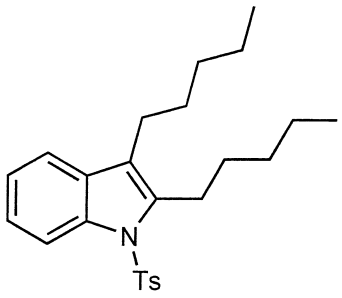

2f

azu-127-2-Re

2f

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136.926
135.938
131.097
129.442
126.059
123.737
123.284
121.786
118.476
115.422

77.313
77.000
76.679

31.819
31.564
30.666
29.464
26.542
24.146
22.434
22.393
21.347
13.970
13.929

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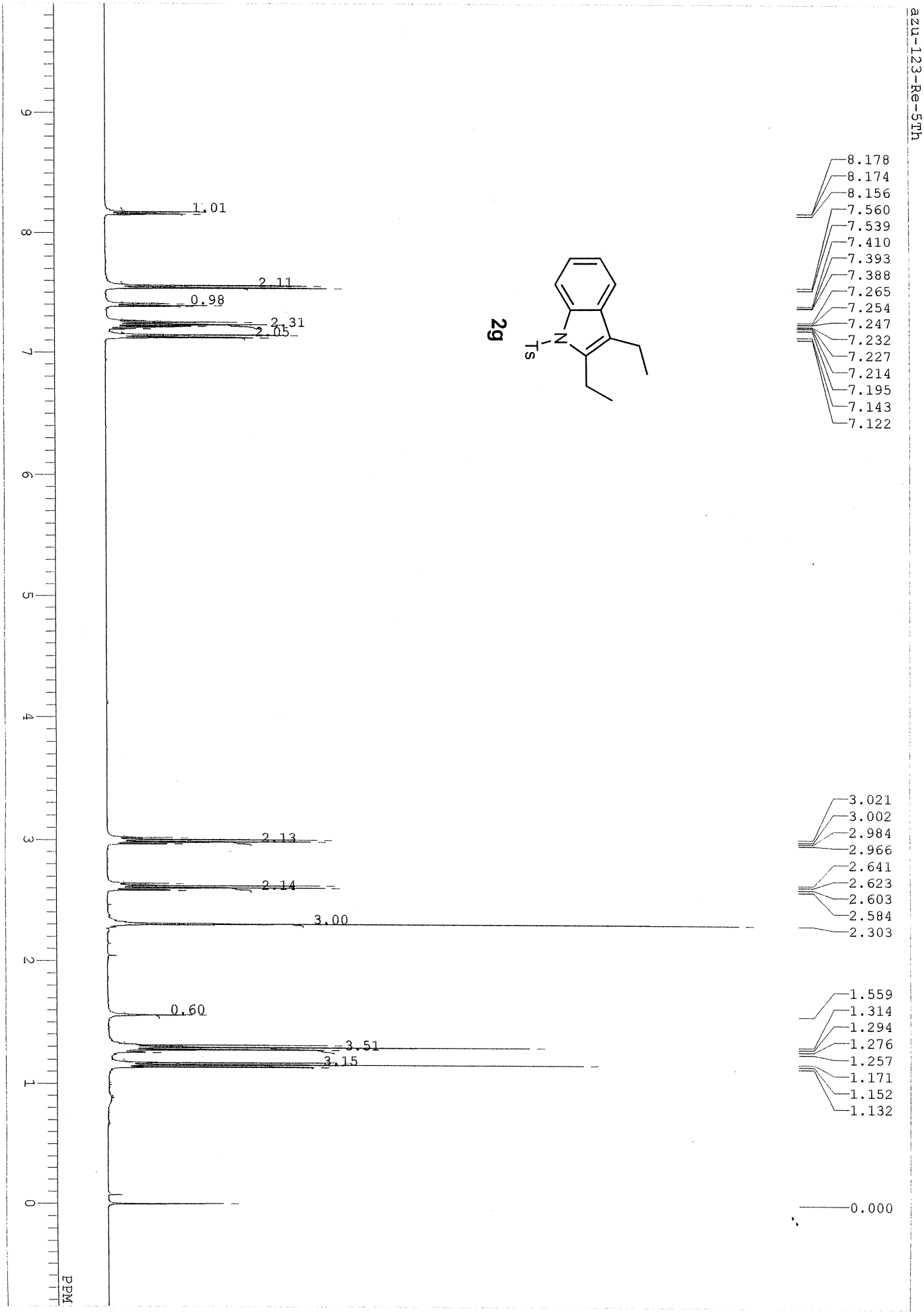
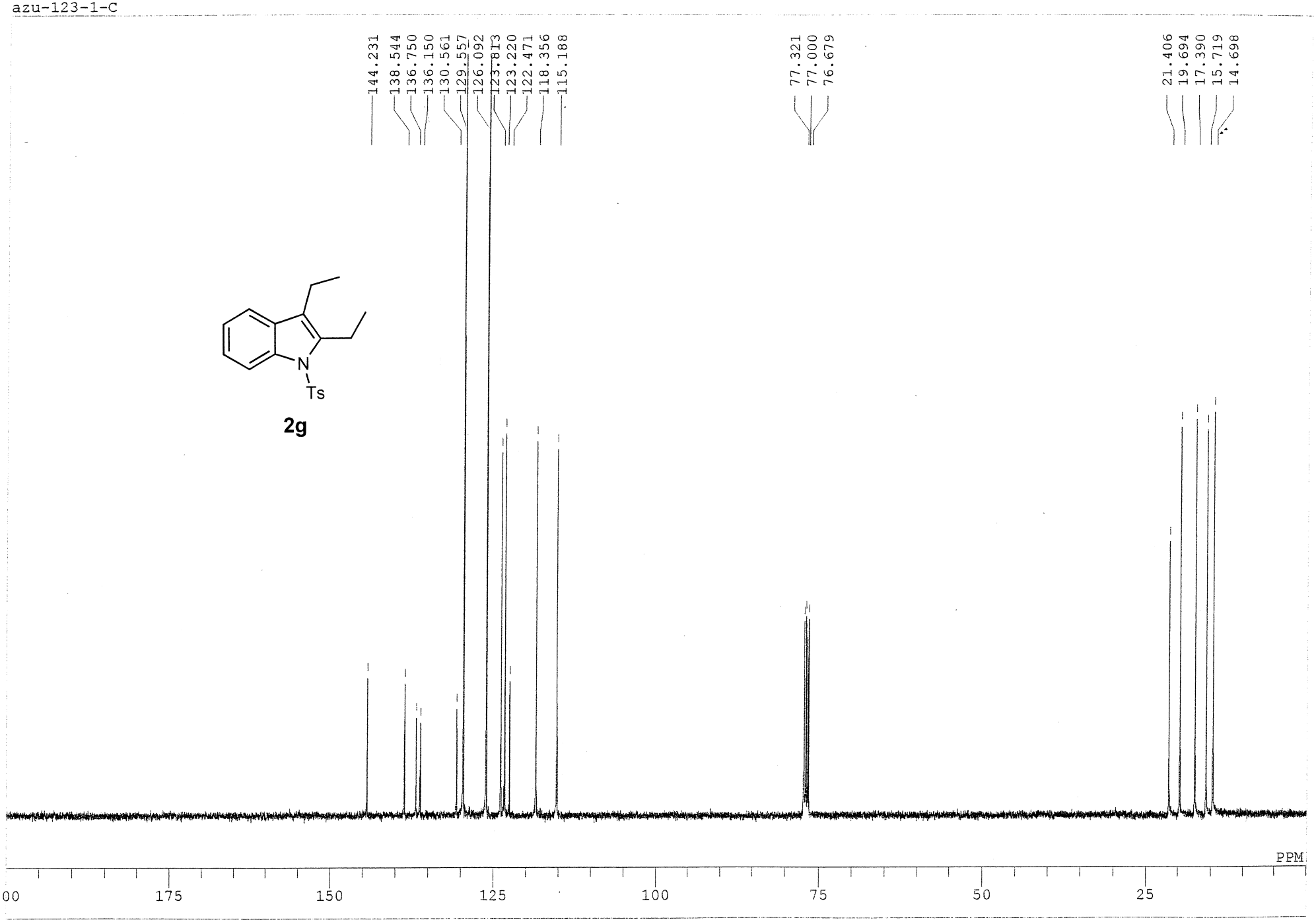


2g

azu-123-1-C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136.150
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123.813
123.220
122.471
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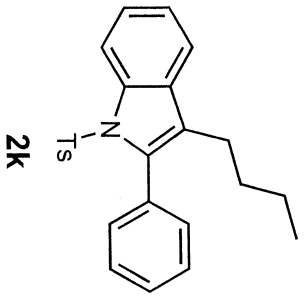
115.188
77.321
77.000
76.679
21.406
19.694
17.390
15.719
14.698
N
Ts
2g
PPM
200
175
150
125
100
75
50
25

azu-2-5-3-2


N
Ts

2k

10
9
8
7
6
5
4
3
2
1
0
-1
PPM
1.00
13.21
1.95
2.07
3.10
0.49
0.06
0.24
2.09
2.82
3.20
0.12
8.331
8.311
7.475
7.455
7.427
7.421
7.414
7.381
7.360
7.342
7.338
7.328
7.324
7.309
7.304
7.287
7.244
7.061
7.041
2.469
2.450
2.430
2.292
2.161
2.039
1.554
1.433
1.414
1.396
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1.148
1.128
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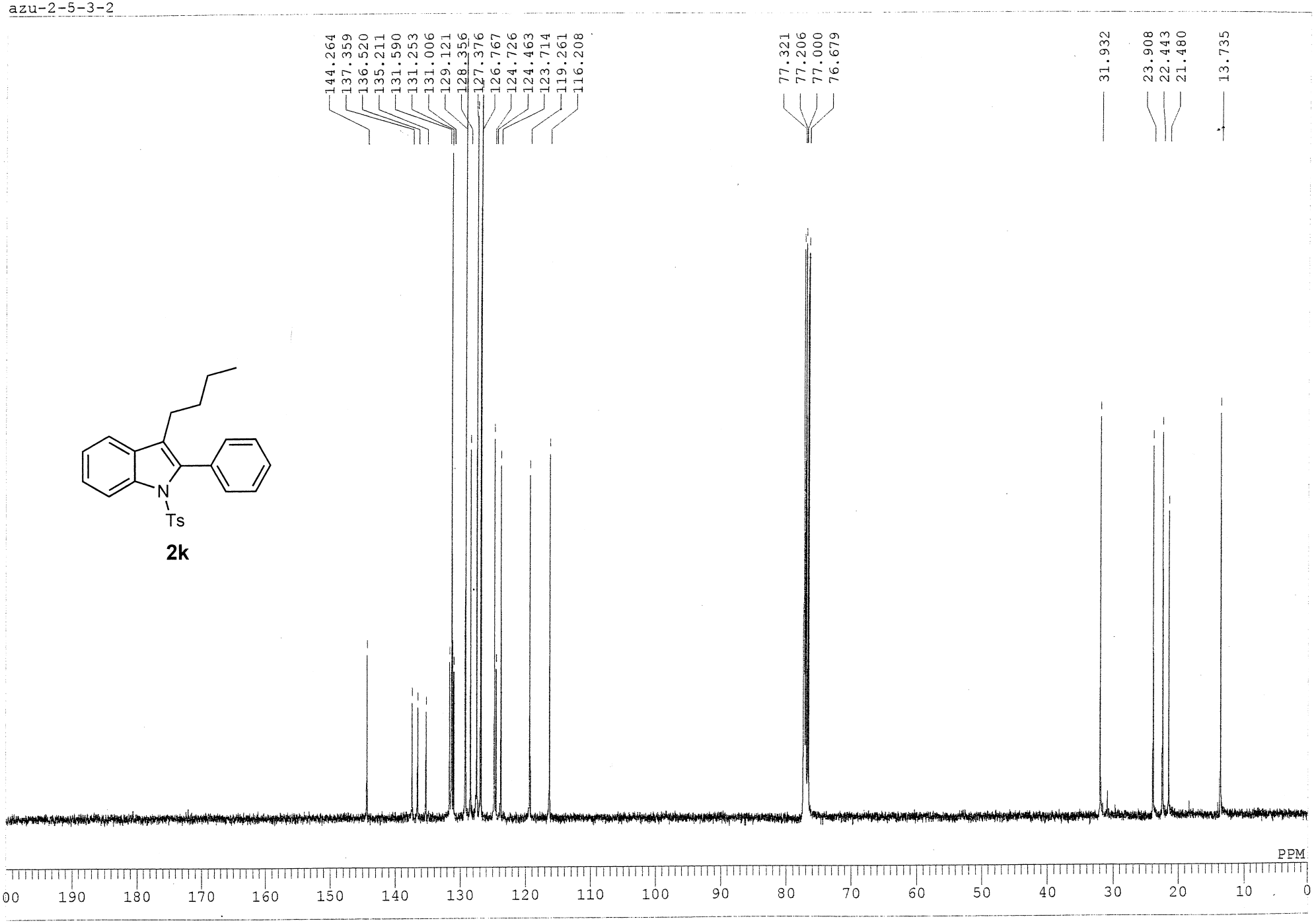
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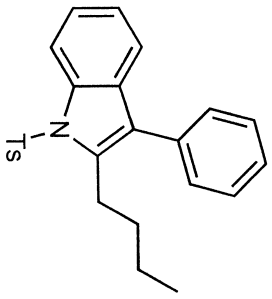
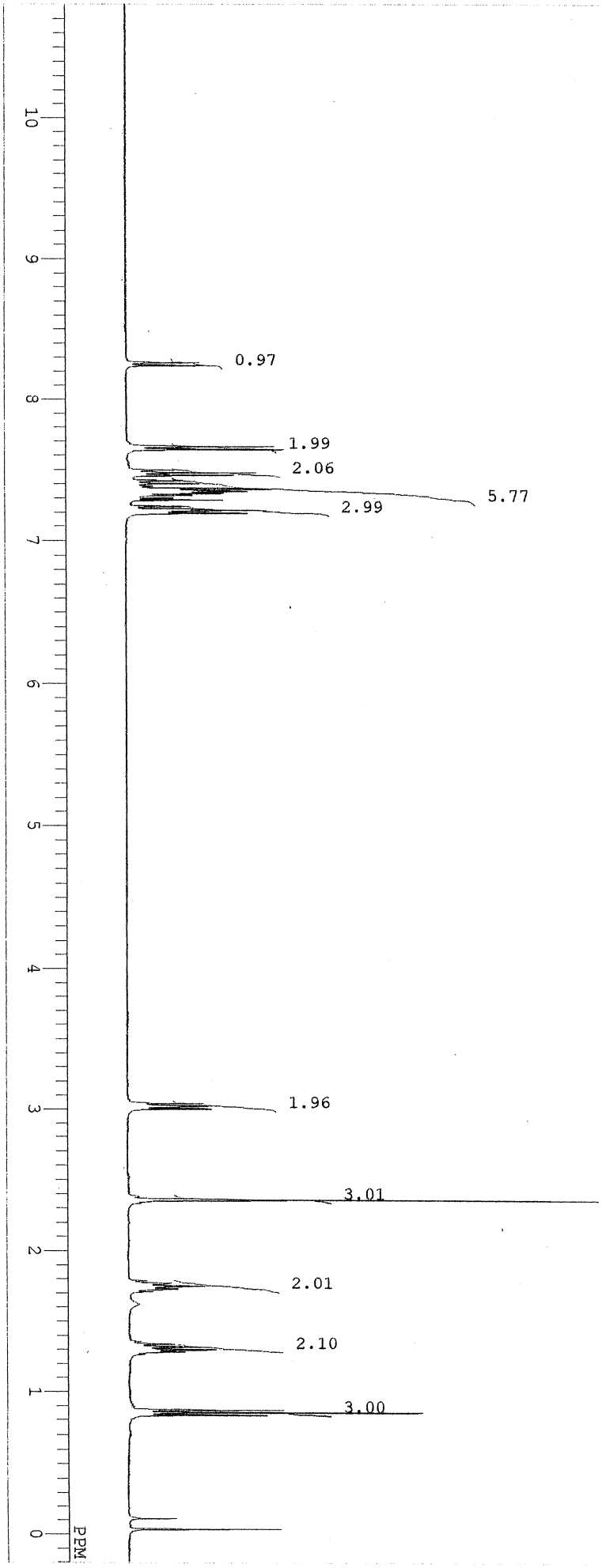
127.376
126.767
124.726
124.463
123.714
119.261
116.208
77.321
77.206
77.000
76.679
31.932
23.908
22.443
21.480
13.735
N
Ts
PPM
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

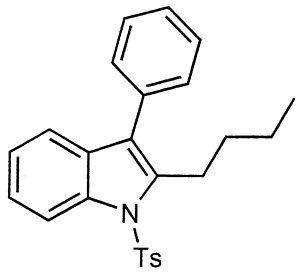
2k

azu 2



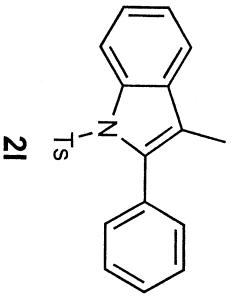


azu 2


N
Ts

PPM
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

azu2-45-P3-Re



2l

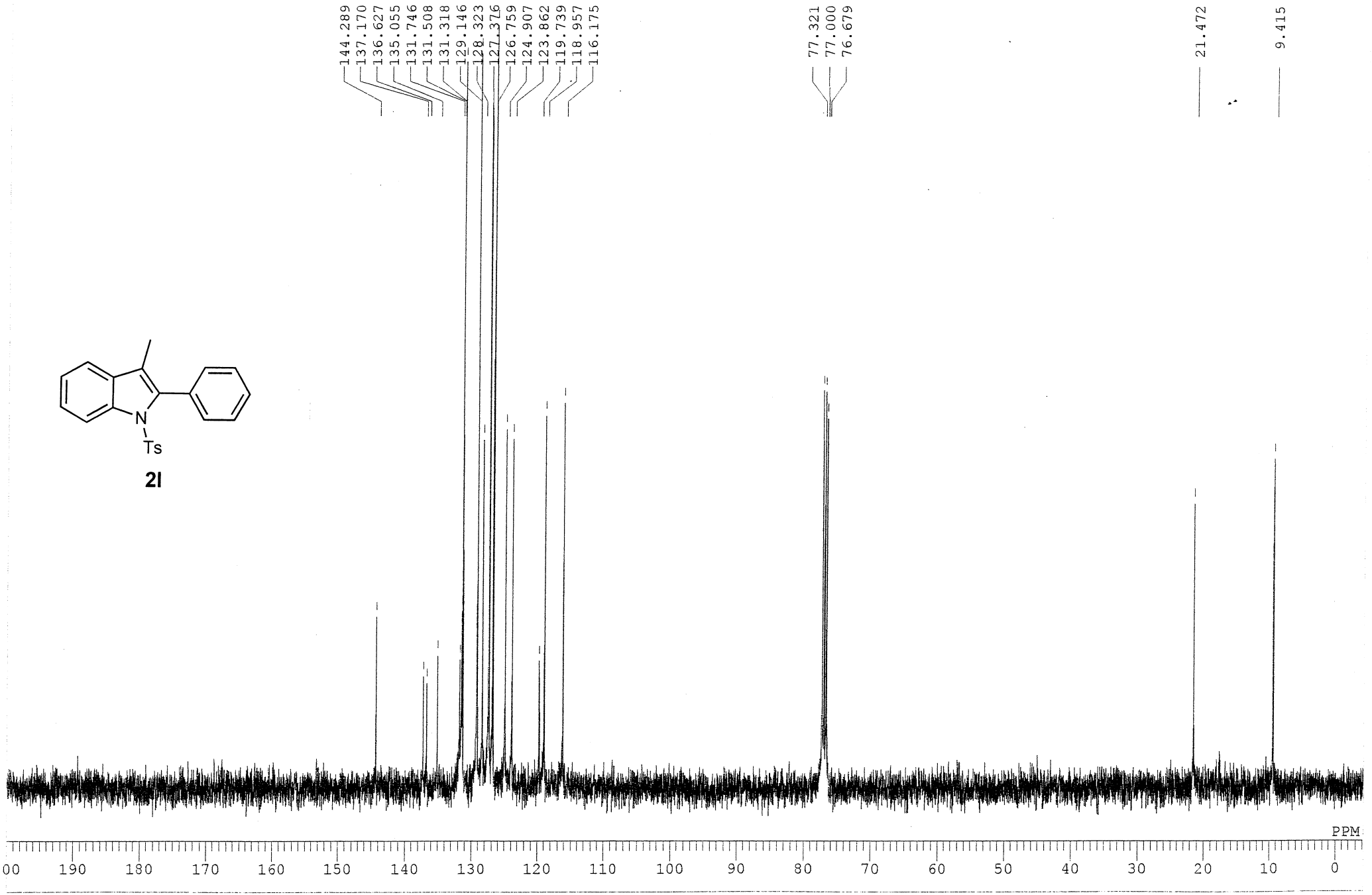
8.330
8.309
7.441
7.432
7.425
7.421
7.414
7.397
7.393
7.378
7.376
7.372
7.358
7.354
7.345
7.343
7.341
7.337
7.330
7.319
7.316
7.300
7.280
7.254
7.055
7.034

1.00
2.63
7.89
2.05
3.10
3.09
1.42
0.65

10 9 8 7 6 5 4 3 2 1 0

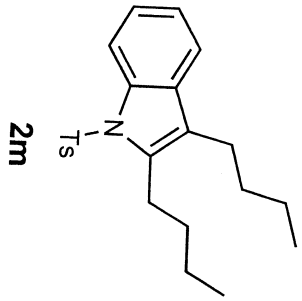
PPM

azu-3-163-2-13C



2l

azu-3-181-1



2m

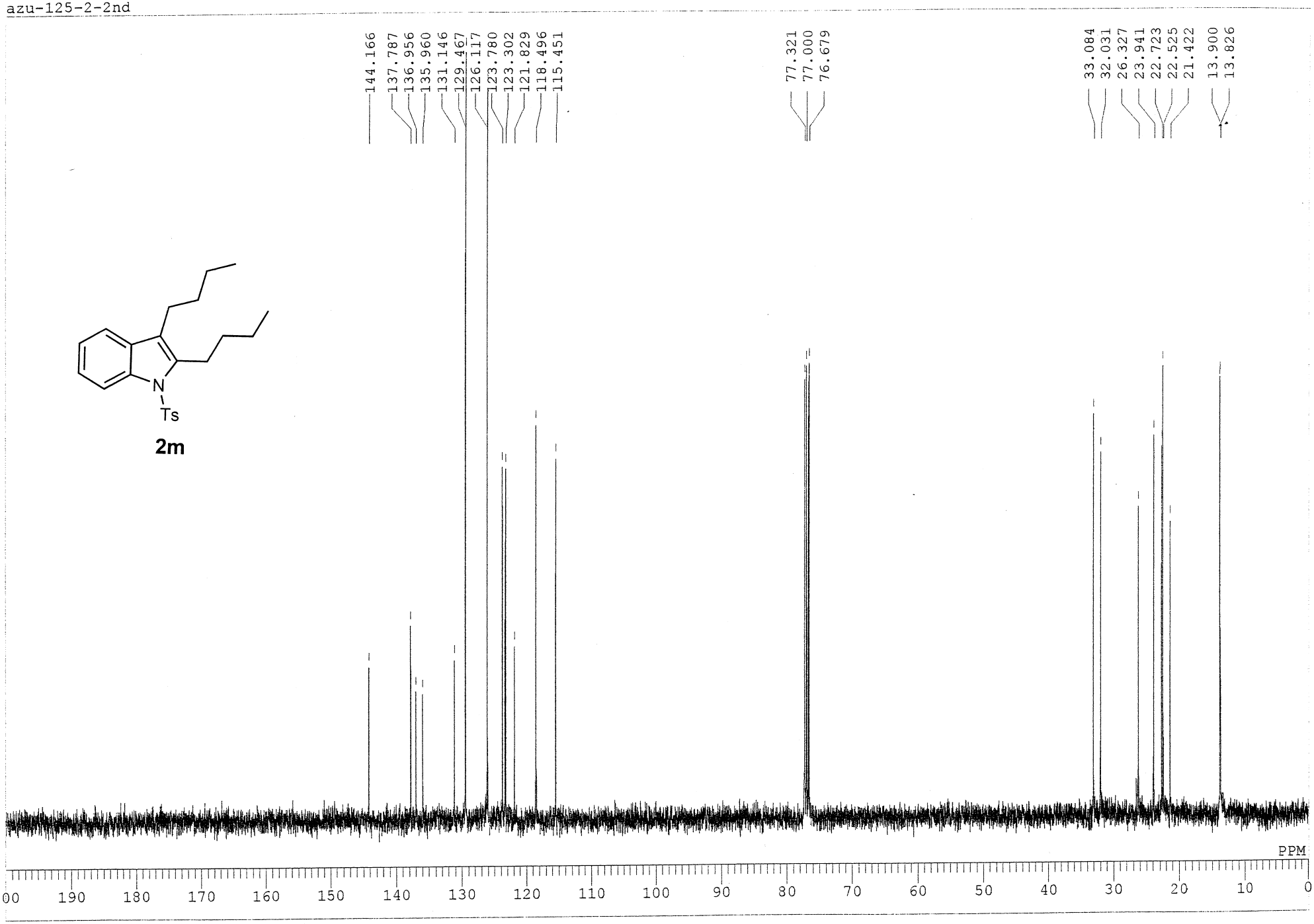
8.167
8.163
8.145
7.514
7.492
7.377
7.360
7.354
7.245
7.238
7.231
7.227
7.213
7.210
7.195
7.177
7.106
7.084

2.960
2.940
2.921
2.592
2.573
2.553
2.276
1.712
1.693
1.674
1.654
1.635
1.570
1.529
1.510
1.491
1.471
1.453
1.435
1.415
1.397
1.377
1.288
1.270
1.250
1.232
0.958
0.939
0.921
0.893
0.875
0.856
0.000

1.00
2.13
1.07
2.25
2.05
2.11
2.08
3.08
2.14
0.29
4.48
2.50
6.65
0.10

10 9 8 7 6 5 4 3 2 1 0 -1
PPM

azu-125-2-2nd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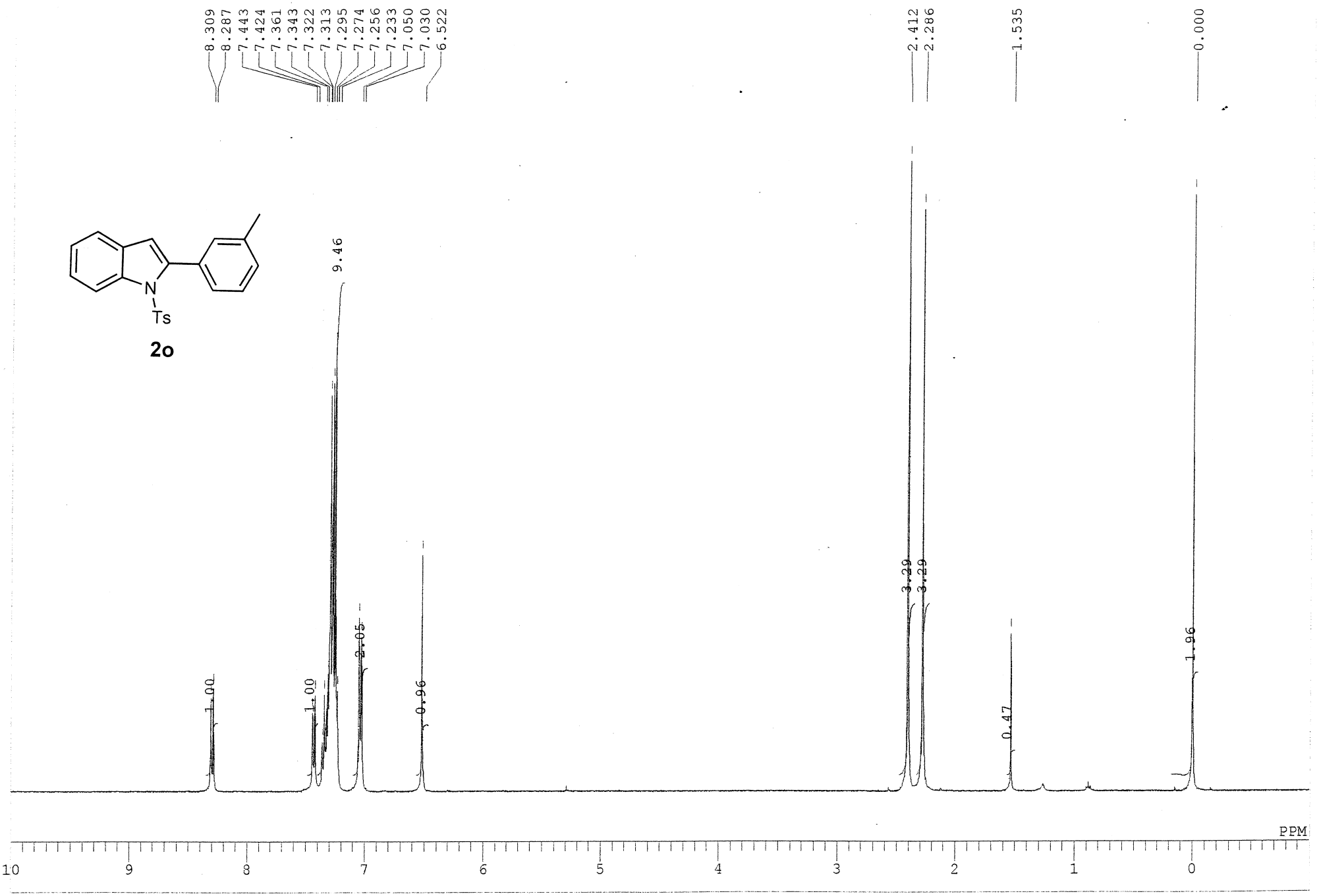
123.780
123.302
121.829
118.496
115.451
77.321
77.000
76.679
33.084
32.031
26.327
23.941
22.723
22.525
21.422
13.900
13.826
N
Ts
2m
PPM
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

azu-3-119-5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7.343
7.322
7.313
7.295
7.274
7.256
7.233
7.050
7.030
6.522
2.412
2.286
1.535
0.000
N
Ts
2o
9.46
3.29
3.29
2.05
1.96
1.00
1.00
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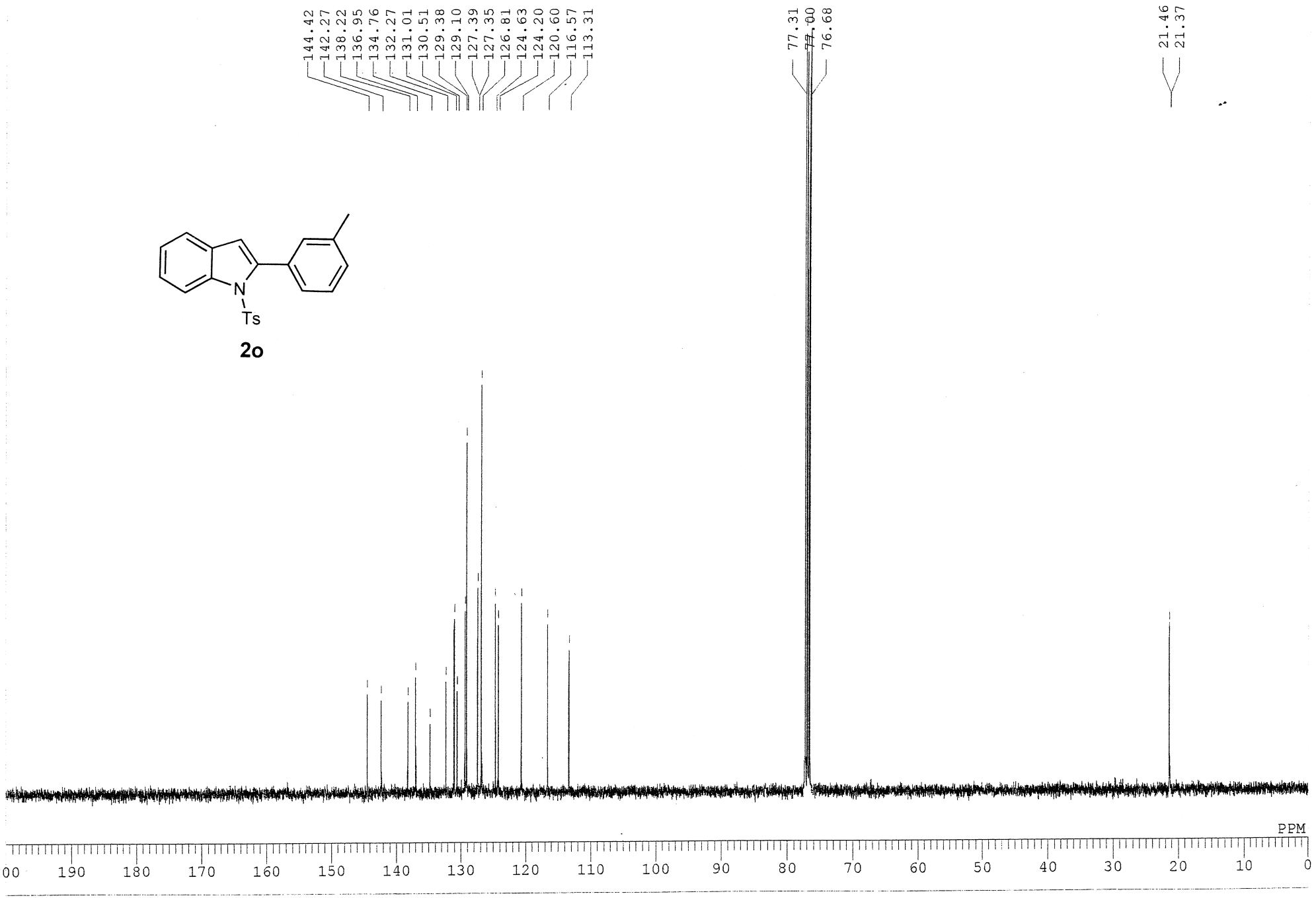
PPM
10
9
8
7
6
5
4
3
2
1
0

azu-3-119-2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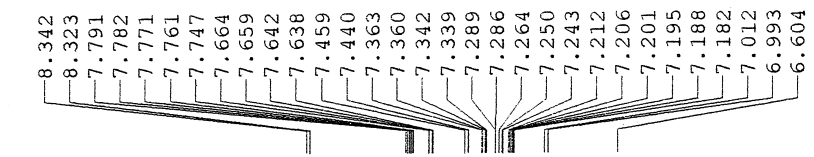
142.27
138.22
136.95
134.76
132.27
131.01
130.51
129.38
129.10
127.39
127.35
126.81
124.63
124.20
120.60
116.57
113.31
77.31
77.00
76.68
21.46
21.37
N
Ts
PPM
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

2o

azu-3-187-4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7.791
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7.771
7.761
7.747
7.664
7.659
7.642
7.638
7.459
7.440
7.363
7.360
7.342
7.339
7.289
7.286
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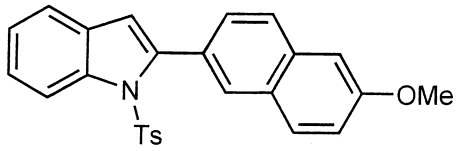
7.243
7.212
7.206
7.201
7.195
7.188
7.182
7.012
6.993
6.604


N
Ts
OMe

2r

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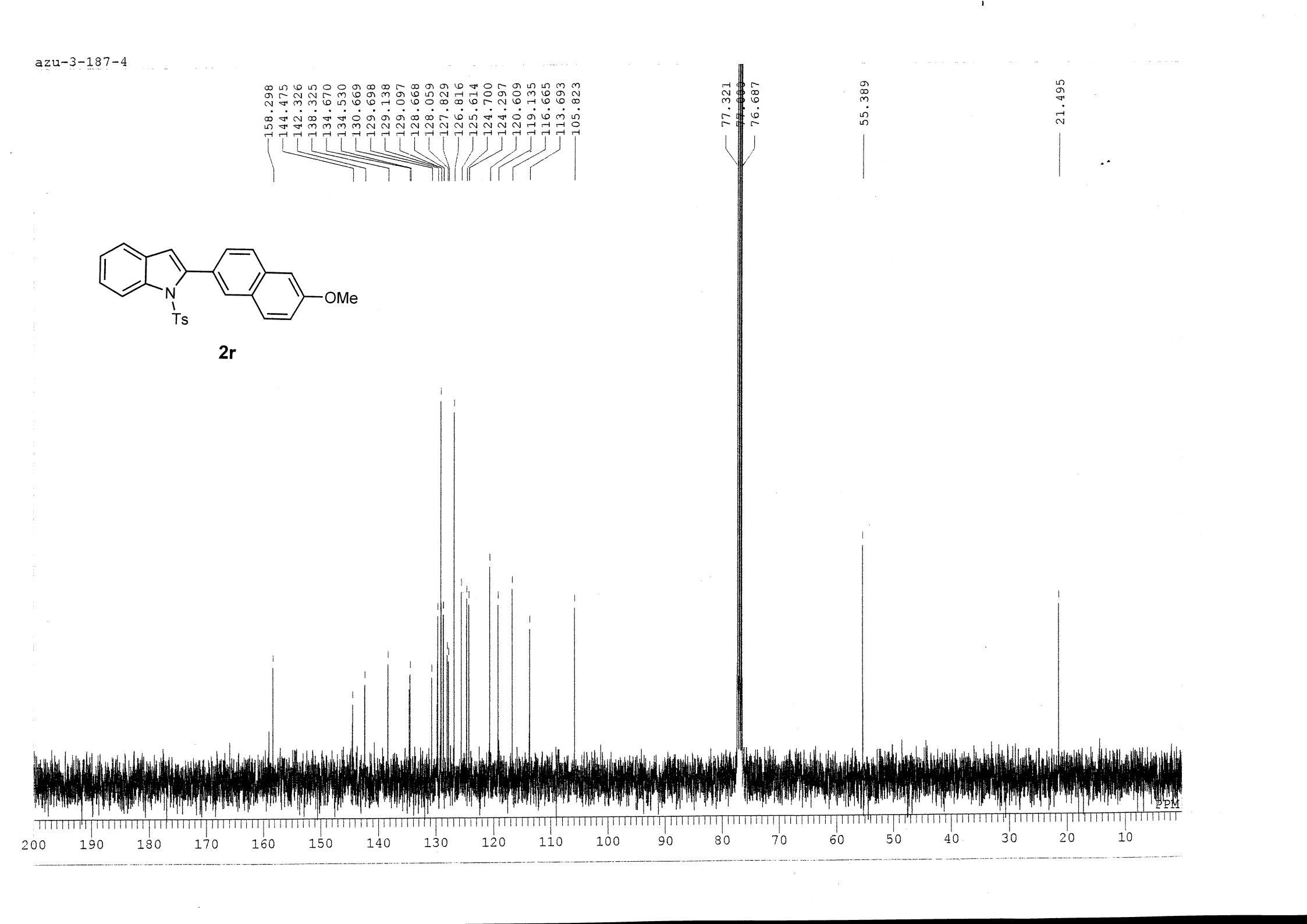
2.270
0.000
1.00
3.16
1.11
1.04
1.08
3.66
2.21
2.09
1.02
3.08
3.10
0.85
PPM
10
9
8
7
6
5
4
3
2
1
0
-1

azu-3-187-4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134.670
134.530
130.669
129.698
129.138
129.097
128.668
128.059
127.829
126.816
125.614
124.700
124.297
120.609
119.135
116.665
113.693
105.823
77.321
77.000
76.687
55.389
21.495
N
Ts
OMe
PPM
200
190
180
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150
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130
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110
100
90
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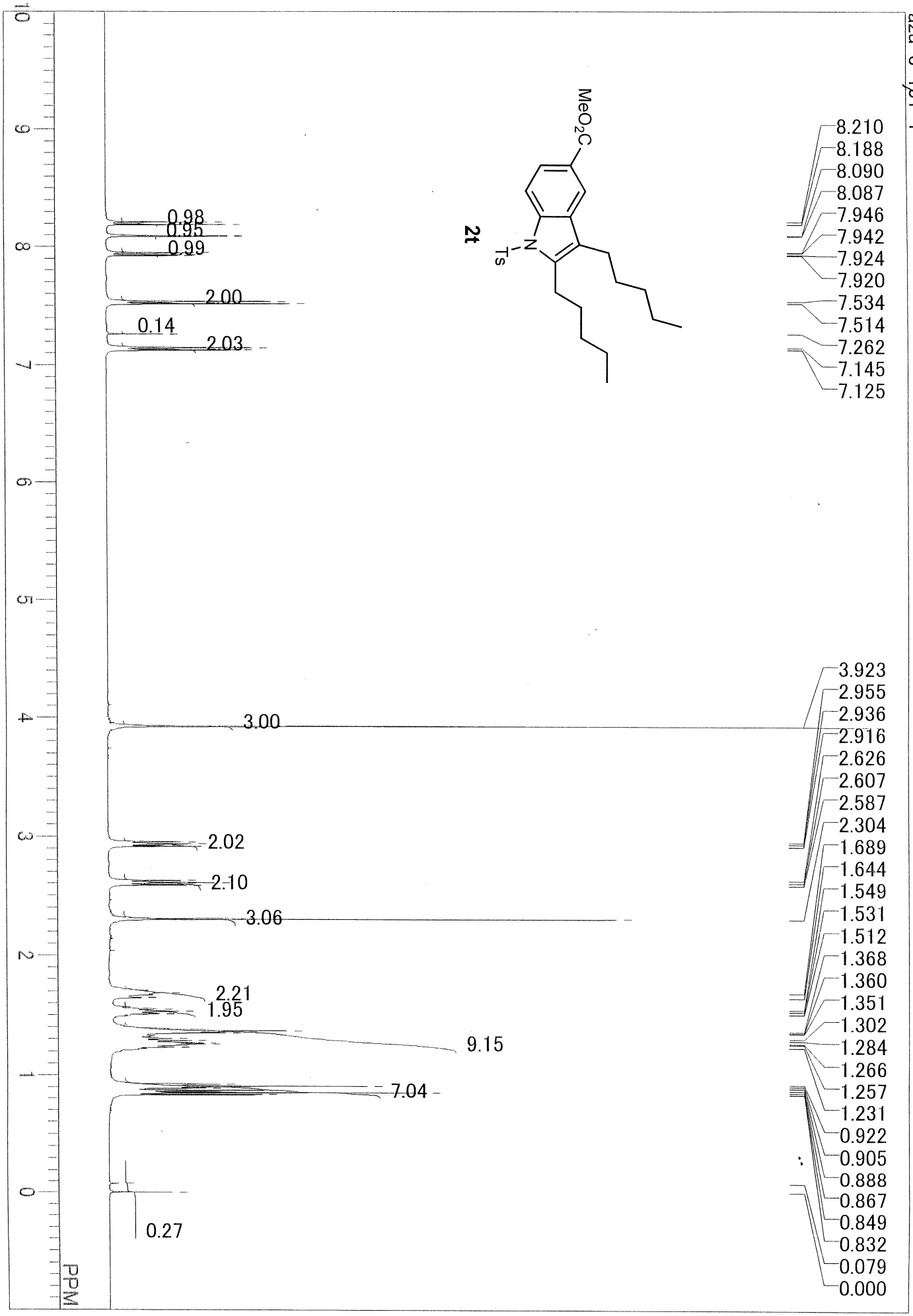
70
60
50
40
30
20
10

2r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7.920
7.534
7.514
7.262
7.145
7.125
3.923
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2.936
2.916
2.626
2.607
2.587
2.304
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1.644
1.549
1.531
1.512
1.368
1.360
1.351
1.302
1.284
1.266
1.257
1.231
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0.832
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0.000
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0.95
0.99
2.00
0.14
2.03
3.00
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7.04
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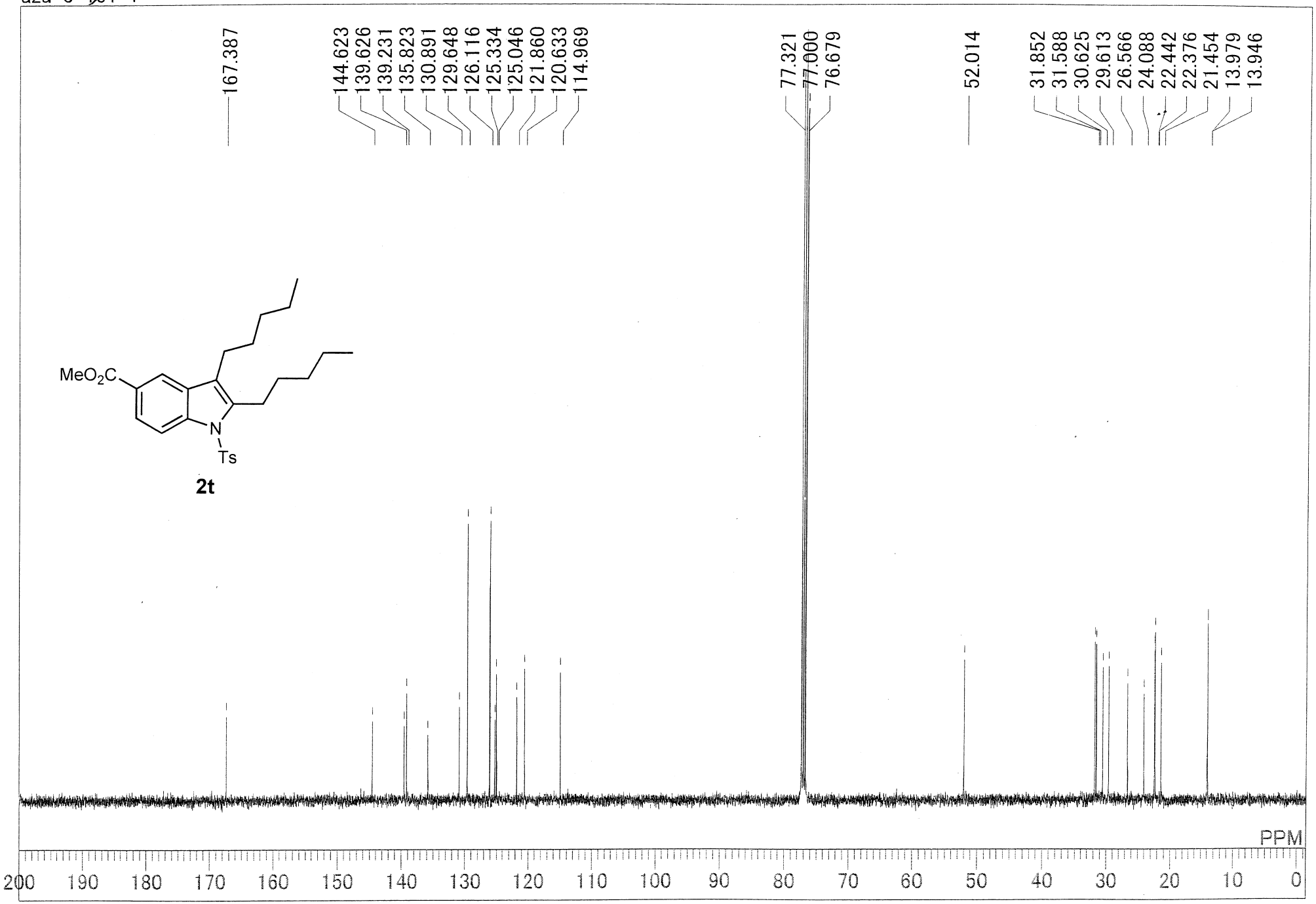
PPM

azu-3-151-1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130.891
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126.116
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125.046
121.860
120.633
114.969
77.321
77.000
76.679
52.014
31.852
31.588
30.625
29.613
26.566
24.088
22.442
22.376
21.454
13.979
13.946
MeO2C
N
Ts
2t
PPM
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0