

Supporting Information
for

**Synthesis of Substituted Indoles from
2-Azidoacrylates and *ortho*-Silyl Aryltriflates**

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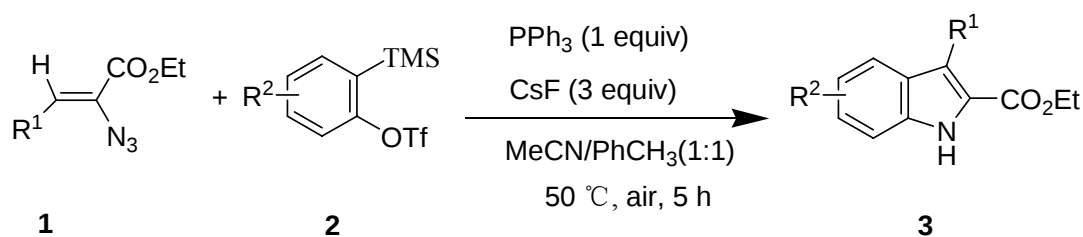
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General Information:

Infrared spectra were obtained on a FTIR spectrometer. ^1H NMR spectra were recorded on 500 MHz spectrometer in CDCl_3 solution and the chemical shifts were reported relative to internal standard TMS (0 ppm). The following abbreviations are used to describe peak patterns where appropriate: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants are reported in Hertz (Hz). ^{13}C NMR were recored on 125 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm). ^{31}P NMR were recored on 200 MHz in toluene- d_8 /CD $_3$ CN (1:1) solution. MS and HRMS were obtained using ESI ionization. Melting points were measured with micro melting point apparatus.

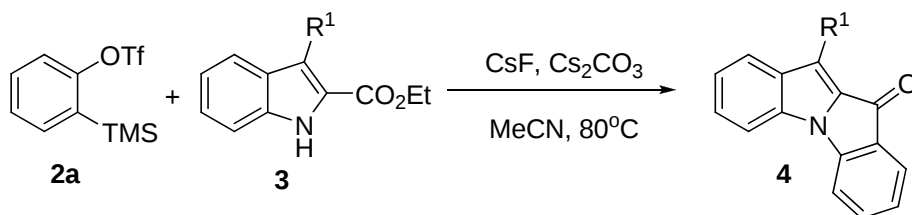
The 2-azidoacrylates and *ortho*-silyl aryltriflates were prepared according to the literature.^{1,2} PPh_3 , CsF and Cs_2CO_3 were commercial available. Toluene and CH_3CN were distilled over phosphorous pentoxide.

General Procedure for the Synthesis of 3:



To a suspension of PPh_3 (0.5 mmol) and CsF (1.5 mmol) in MeCN/PhMe (1:1, 5 mL) was added a solution of **1** (0.5 mmol) and **2** (0.75 mmol) in MeCN/PhMe (1:1, 5 mL) over 30 min at $50\text{ }^\circ\text{C}$ under air. The reaction mixture was stirred at $50\text{ }^\circ\text{C}$ for 5 h and then filtered through a Celite plug. The filtrate was concentrated in vacuum, and the residue was purified by chromatography on silica gel column using hexane/ EtOAc (5:1) as the eluent to afford **3**.

General Procedure for the Synthesis of 4:



The **3** (0.5 mmol), Cs_2CO_3 (1.0 mmol) and CsF (1.5 mmol) were added to a dried round bottom flask and put under dried air. CH_3CN (4 mL) were added and the suspension was stirred and heated to $80\text{ }^\circ\text{C}$. Then, a solution of **2a** (0.75 mmol) in CH_3CN (6 mL) was added over 10 min. The reaction mixture was stirred at $80\text{ }^\circ\text{C}$ for 1 h and then filtered through a Celite plug. The filtrate was concentrated in vacuum, and the residue was purified by chromatography on silica gel column using hexane/ EtOAc (5:1) as the eluent to afford **4**.

Procedure and Data for ^{31}P NMR Trapping Experiment

To a dried round bottom flask were added PPh_3 (0.5 mmol), CsF (1.5 mmol), toluene- d_8 (2.5 mL) and CD_3CN (2.5 mL) under N_2 atmosphere. A solution of **1a** (0.5 mmol) and **2a** (0.75 mmol) in toluene- d_8 and CD_3CN (1:1, 5 mL) was added to the stirring mixture over 30 min at 50 $^\circ\text{C}$. The reaction mixture was stirred at 50 $^\circ\text{C}$ for 10 h, then D_2O (0.5 mL) and silica gel (0.5 g) were added to stop the reaction. The reaction was detected after 2 h, 5 h, 10 h and addition of D_2O using ^{31}P NMR (under N_2 atmosphere). The LRMS and HRMS (ESI) detections for intermediates **B** were performed after 10 h. The ^{31}P NMR spectra of pure compounds of PPh_3 , **A** and Ph_3PO were recorded in toluene- d_8 and CD_3CN (1:1) solution.

Characterization data:

^{31}P NMR data (toluene- d_8 / CD_3CN (1:1), 200 MHz) for PPh_3 : δ -4.9 ppm;

^{31}P NMR data (toluene- d_8 / CD_3CN (1:1), 200 MHz) for Ph_3PO : δ 26.4 ppm;

^{31}P NMR data (toluene- d_8 / CD_3CN (1:1), 200 MHz) for **A**: δ 7.7 ppm;

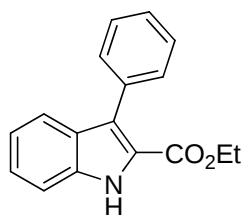
^{31}P NMR data (toluene- d_8 / CD_3CN (1:1), 200 MHz) for **B** (two possible stereoisomers): δ 23.45, 23.42 ppm;

LRMS (ESI) of **B**: m/z 528 ($[\text{M}+\text{H}]^+$);

HRMS (ESI) of **B** calcd for ($[\text{C}_{35}\text{H}_{30}\text{NO}_2\text{P}+\text{H}]^+$): 528.2087, found: 528.2094.

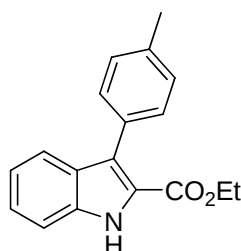
Characterization of 3

Ethyl 3-phenyl-1*H*-indole-2-carboxylate (3a).³



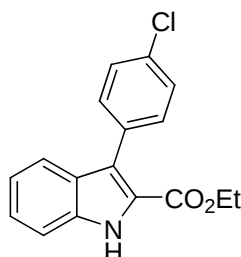
White solid, m.p. 132-133 °C; IR (KBr): 3320, 1677, 1450, 1384, 1145, 1019, 745 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 9.09 (s, 1 H), 7.63 (d, J = 8.2 Hz, 1 H), 7.57-7.55 (m, 2 H), 7.46-7.43 (m, 3 H), 7.39-7.34 (m, 2 H), 7.16-7.13 (m, 1 H), 4.29 (q, J = 7.1 Hz, 2 H), 1.23 (t, J = 7.1 Hz, 3 H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 162.3, 136.0, 133.8, 130.9, 128.2, 128.0, 127.5, 126.0, 124.5, 123.0, 122.0, 121.1, 111.9, 61.1, 14.3 ppm; MS (ESI): m/z ([M+Na]⁺) 288; HRMS (ESI) calcd for ([C₁₇H₁₅NO₂+Na]⁺): 288.0995; found: 288.0990.

Ethyl 3-*p*-tolyl-1*H*-indole-2-carboxylate (3b).



Yellow solid, m.p. 142-143 °C; IR (KBr): 3321, 1677, 1440, 1384, 1141, 1016, 743 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 9.09 (s, 1 H), 7.65-7.63 (m, 1 H), 7.46 (d, J = 8.0 Hz, 2 H), 7.42 (d, J = 8.4 Hz, 1 H), 7.35-7.32 (m, 1 H), 7.26-7.23 (m, 2 H), 7.15-7.11 (m, 1 H), 4.30 (q, J = 7.1 Hz, 2 H), 2.42 (s, 3 H), 1.25 (t, J = 7.1 Hz, 3 H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 162.3, 137.1, 136.0, 130.8, 130.7, 128.8, 128.2, 126.0, 124.6, 122.9, 122.1, 121.0, 111.9, 61.1, 21.6, 14.4 ppm; MS (ESI): m/z ([M+Na]⁺) 302; HRMS (ESI) calcd for ([C₁₈H₁₇NO₂+Na]⁺): 302.1151; found: 302.1147.

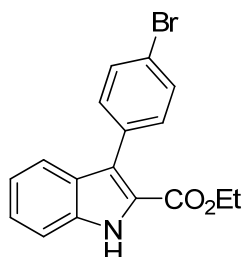
Ethyl 3-(4-chlorophenyl)-1*H*-indole-2-carboxylate (3c).⁴



Light yellow solid, m.p. 143-144 °C; IR (KBr): 3320, 1676, 1439, 1382, 1141, 1017, 744 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 9.14 (s, 1 H), 7.58 (d, J = 8.1 Hz, 1 H), 7.50-7.48 (m, 2 H), 7.45-7.41 (m, 3 H), 7.38-7.36 (m, 1 H), 7.17-7.14 (m, 1 H), 4.31 (q, J = 7.1 Hz, 2 H), 1.26 (t, J = 7.1 Hz, 3 H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 162.1, 135.9, 133.4, 132.2, 128.2, 128.0, 126.2, 123.1,

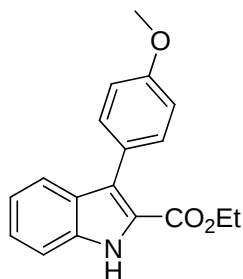
121.7, 121.3, 112.1, 61.3, 14.4 ppm; MS (ESI): m/z ($[M+Na]^+$) 322; HRMS (ESI) calcd for ($[C_{17}H_{14}ClNO_2+Na]^+$): 322.0605; found: 322.0604.

Ethyl 3-(4-bromophenyl)-1H-indole-2-carboxylate (3d).



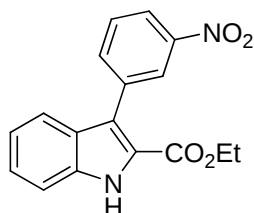
White solid, m.p. 163-164 °C; IR (KBr): 3339, 1673, 1439, 1381, 1144, 1007, 743 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$): δ 9.06 (s, 1 H), 7.59-7.56 (m, 3 H), 7.46-7.42 (m, 3 H), 7.38-7.35 (m, 1 H), 7.18-7.15 (m, 1 H), 4.31 (q, J = 7.1 Hz, 2 H), 1.26 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.0, 135.9, 132.7, 132.5, 131.2, 127.9, 126.2, 123.12, 123.08, 121.7, 121.4, 112.0, 61.3, 14.4 ppm; MS (ESI): m/z ($[M-H]^-$) 342; HRMS (ESI) calcd for ($[C_{17}H_{14}BrNO_2-H]^-$): 342.0135; found: 342.0120.

Ethyl 3-(4-methoxyphenyl)-1H-indole-2-carboxylate (3e).⁵



White solid, m.p. 115-116 °C; IR (KBr): 3345, 1696, 1453, 1378, 1144, 1032, 748 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$): δ 9.09 (s, 1 H), 7.65-7.63 (m, 1 H), 7.51-7.49 (m, 2 H), 7.42 (d, J = 8.4 Hz, 1 H), 7.36-7.34 (m, 1 H), 7.15-7.12 (m, 1 H), 7.01-6.99 (m, 2 H), 4.31 (q, J = 7.1 Hz, 2 H), 3.87 (s, 3 H), 1.26 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.3, 159.1, 136.0, 132.0, 128.2, 126.0, 125.3, 124.3, 122.8, 122.0, 120.9, 113.5, 111.9, 61.1, 55.5, 14.4 ppm; MS (ESI): m/z ($[M+Na]^+$) 318; HRMS (ESI) calcd for ($[C_{18}H_{17}NO_3+Na]^+$): 318.1101; found: 318.1095.

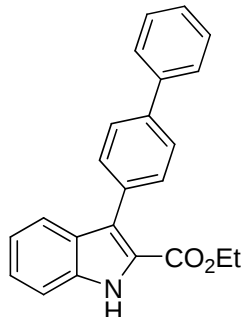
Ethyl 3-(3-nitrophenyl)-1H-indole-2-carboxylate (3f).



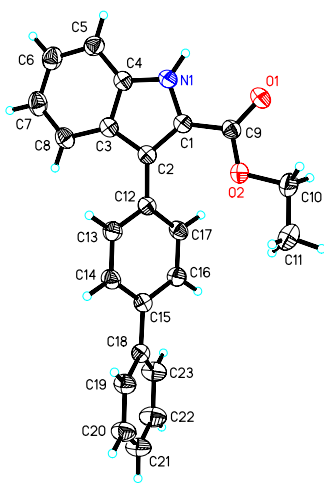
Yellow solid, m.p. 149-150 °C; IR (KBr): 3398, 1714, 1431, 1349, 1160, 1038, 737 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$): δ 9.37 (s, 1 H), 8.47-8.46 (m, 1 H), 8.25-8.23 (m, 1 H), 7.90 (d, J = 7.7 Hz, 1 H), 7.63-7.57 (m, 2 H), 7.49 (d, J = 8.4 Hz, 1 H), 7.41-7.38 (m, 1 H), 7.21-7.18 (m, 1 H), 4.32 (q, J = 7.1 Hz, 2 H), 1.23 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 161.9, 148.2, 137.0,

135.9, 135.7, 128.9, 127.6, 126.4, 126.0, 123.5, 122.3, 121.8, 121.4, 121.1, 112.3, 61.6, 29.9, 14.2 ppm; MS (ESI): m/z ($[M-H]^-$) 309; HRMS (ESI) calcd for ($[C_{17}H_{14}N_2O_4-H]^-$): 309.0881; found: 309.0872.

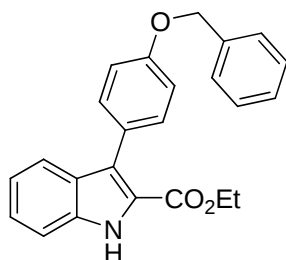
Ethyl 3-(biphenyl-4-yl)-1H-indole-2-carboxylate (3g).



Colorless crystal, m.p. 227-228 °C; IR (KBr): 3309, 1678, 1453, 1376, 1145, 1011, 752 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$): δ 9.09 (s, 1 H), 7.71-7.64 (m, 7 H), 7.49-7.45 (m, 3 H), 7.39-7.36 (m, 2 H), 7.19-7.17 (m, 1 H), 4.33 (q, J = 7.1 Hz, 2 H), 1.27 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.2, 141.3, 140.2, 136.0, 132.8, 131.3, 129.0, 128.2, 127.5, 127.4, 126.7, 126.1, 124.1, 123.1, 122.0, 121.2, 112.0, 61.2, 14.4 ppm; MS (ESI): m/z ($[M+Na]^+$) 364; HRMS (ESI) calcd for ($[C_{23}H_{19}NO_2+Na]^+$): 364.1308; found: 364.1300.



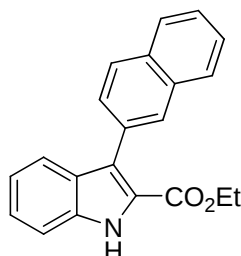
Ethyl 3-(4-(benzyloxy)phenyl)-1H-indole-2-carboxylate (3h).



Yellow solid, m.p. 134-135 °C; IR (KBr): 3305, 1669, 1454, 1384, 1179, 1014, 747 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$): δ 9.03 (s, 1 H), 7.64 (d, J = 8.3 Hz, 1 H), 7.50-7.47 (m, 4 H), 7.42-7.38 (m, 3 H), 7.35-7.33 (m, 2 H), 7.14-7.13 (m, 1 H), 7.07-7.06 (m, 2 H), 5.13 (s, 2 H), 4.30 (q, J = 7.1 Hz, 2 H).

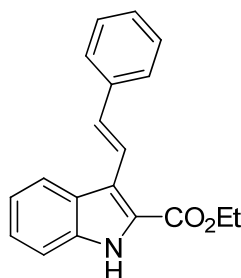
H), 1.24 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.3, 158.4, 137.4, 136.0, 132.0, 128.8, 128.3, 128.2, 127.8, 126.3, 126.0, 124.3, 122.8, 122.0, 121.0, 114.5, 111.9, 70.3, 61.1, 14.4 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 394; HRMS (ESI) calcd for ($[\text{C}_{24}\text{H}_{21}\text{NO}_3+\text{Na}]^+$): 394.1414; found: 394.1409.

Ethyl 3-(naphthalen-2-yl)-1H-indole-2-carboxylate (3i).



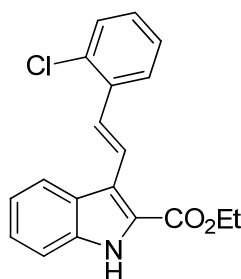
White solid, m.p. 155-156 °C; IR (KBr): 3330, 1676, 1438, 1381, 1143, 1011, 738 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.19 (s, 1 H), 8.03 (s, 1 H), 7.91-7.86 (m, 3 H), 7.70-7.68 (m, 2 H), 7.51-7.49 (m, 2 H), 7.46 (d, J = 8.4 Hz, 1 H), 7.38-7.37 (m, 1 H), 7.17-7.14 (m, 1 H), 4.29 (q, J = 7.1 Hz, 2 H), 1.18 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.4, 136.1, 133.4, 132.9, 131.4, 129.6, 129.4, 128.33, 128.27, 127.9, 127.3, 126.2, 126.1, 124.3, 123.3, 122.0, 121.2, 112.0, 61.2, 14.3 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 338; HRMS (ESI) calcd for ($[\text{C}_{21}\text{H}_{17}\text{NO}_2+\text{Na}]^+$): 338.1151; found: 338.1147.

(E)-Ethyl 3-styryl-1H-indole-2-carboxylate (3j).⁶



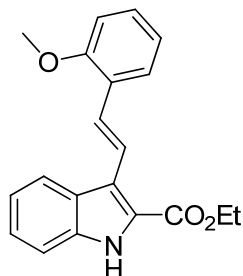
Light yellow solid, m.p. 159-160 °C; IR (KBr): 3306, 1679, 1460, 1380, 1196, 1022, 740 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.96 (s, 1 H), 8.14 (d, J = 8.2 Hz, 1 H), 8.06 (d, J = 16.8 Hz, 1 H), 7.59 (d, J = 7.6 Hz, 2 H), 7.42-7.36 (m, 5 H), 7.28-7.22 (m, 2 H), 4.47 (q, J = 7.1 Hz, 2 H), 1.47 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.3, 138.4, 136.6, 131.2, 128.9, 127.7, 126.6, 126.1, 126.0, 124.3, 122.9, 122.1, 121.5, 121.1, 112.3, 61.4, 14.7 ppm; MS (ESI): m/z ($[\text{M}-\text{H}]$) 290; HRMS (ESI) calcd for ($[\text{C}_{19}\text{H}_{17}\text{NO}_2-\text{H}]$): 290.1187; found: 290.1177.

(E)-Ethyl 3-(2-chlorostyryl)-1H-indole-2-carboxylate (3k).



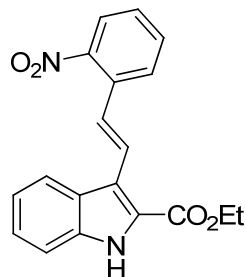
Light yellow solid, m.p. 176-177 °C; IR (KBr): 3319, 1675, 1454, 1380, 1197, 1023, 738 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 8.96 (s, 1 H), 8.19 (d, J = 8.1 Hz, 1 H), 8.05 (d, J = 16.7 Hz, 1 H), 7.80-7.76 (m, 2 H), 7.43-7.38 (m, 3 H), 7.29-7.25 (m, 2 H), 7.19-7.18 (m, 1 H), 4.47 (q, J = 7.1 Hz, 2 H), 1.47 (t, J = 7.2 Hz, 3 H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 162.1, 136.6, 136.4, 133.5, 130.0, 128.5, 127.2, 127.0, 126.3, 126.2, 125.9, 124.5, 124.3, 122.9, 121.9, 121.0, 112.3, 61.5, 14.7 ppm; MS (ESI): m/z ([M-H]⁻) 324; HRMS (ESI) calcd for ([C₁₉H₁₆ClNO₂-H]⁻): 324.0797; found: 324.0785.

(E)-Ethyl 3-(2-methoxystyryl)-1H-indole-2-carboxylate (3l).



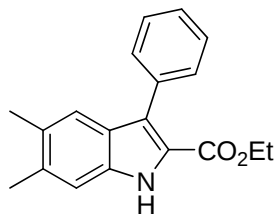
Yellow solid, m.p. 187-188 °C; IR (KBr): 3331, 1674, 1459, 1381, 1189, 1025, 741 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 8.89 (s, 1 H), 8.19 (d, J = 8.2 Hz, 1 H), 8.02 (d, J = 17.0 Hz, 1 H), 7.76 (d, J = 16.9 Hz, 1 H), 7.71-7.69 (m, 1 H), 7.42-7.35 (m, 2 H), 7.25-7.22 (m, 2 H), 7.01-6.98 (m, 1 H), 6.92 (d, J = 8.2 Hz, 1 H), 4.46 (q, J = 7.1 Hz, 2 H), 3.92 (s, 3 H), 1.46 (t, J = 7.1 Hz, 3 H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 162.4, 157.1, 136.6, 128.7, 127.6, 126.3, 126.03, 126.01, 124.1, 123.2, 122.2, 121.9, 121.4, 121.1, 112.2, 111.2, 61.3, 55.9, 14.7 ppm; MS (ESI): m/z ([M-H]⁻) 320; HRMS (ESI) calcd for ([C₂₀H₁₉NO₃-H]⁻): 320.1292; found: 320.1282.

(E)-Ethyl 3-(2-nitrostyryl)-1H-indole-2-carboxylate (3m).



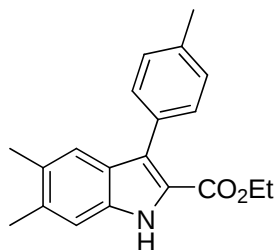
Yellow solid, m.p. 209-210 °C; IR (KBr): 3323, 1676, 1456, 1379, 1202, 1027, 737 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 8.95 (s, 1 H), 8.19 (d, J = 8.2 Hz, 1 H), 8.12 (d, J = 16.5 Hz, 1 H), 8.01-7.99 (m, 1 H), 7.93-7.90 (m, 2 H), 7.62-7.61 (m, 1 H), 7.45-7.38 (m, 3 H), 7.31-7.28 (m, 1 H), 4.48 (q, J = 7.1 Hz, 2 H), 1.48 (t, J = 7.1 Hz, 3 H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 161.9, 148.0, 136.4, 134.2, 133.4, 128.1, 127.9, 127.2, 126.4, 125.8, 125.4, 125.1, 124.9, 122.8, 122.3, 120.6, 112.3, 61.5, 14.7 ppm; MS (ESI): m/z ([M+Na]⁺) 359; HRMS (ESI) calcd for ([C₁₉H₁₆N₂O₄+Na]⁺): 359.1002; found: 359.0995.

Ethyl 5,6-dimethyl-3-phenyl-1H-indole-2-carboxylate (3n).



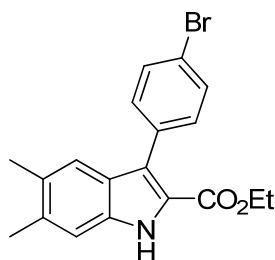
White solid, m.p. 152-153 °C; IR (KBr): 3326, 1676, 1445, 1370, 1158, 1003, 768 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.87 (s, 1 H), 7.55-7.54 (m, 2 H), 7.45-7.42 (m, 2 H), 7.38-7.35 (m, 2 H), 7.20 (s, 1 H), 4.27 (q, J = 7.1 Hz, 2 H), 2.37 (s, 3 H), 2.30 (s, 3 H), 1.22 (t, J = 7.2 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.4, 135.9, 135.1, 134.2, 130.9, 130.3, 127.9, 127.3, 126.7, 124.1, 122.3, 121.4, 112.0, 60.9, 21.0, 20.3, 14.3 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 316; HRMS (ESI) calcd for ($[\text{C}_{19}\text{H}_{19}\text{NO}_2+\text{Na}]^+$): 316.1308; found: 316.1304.

Ethyl 5,6-dimethyl-3-p-tolyl-1H-indole-2-carboxylate (3o).



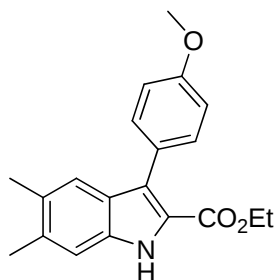
White solid, m.p. 182-183 °C; IR (KBr): 3335, 1674, 1440, 1369, 1158, 1003, 779 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.82 (s, 1 H), 7.50 (d, J = 8.0 Hz, 2 H), 7.37 (s, 1 H), 7.26-7.23 (m, 2 H), 7.18 (s, 1 H), 4.28 (q, J = 7.1 Hz, 2 H), 2.42 (s, 3 H), 2.37 (s, 3 H), 2.30 (s, 3 H), 1.24 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.3, 136.9, 135.8, 135.1, 131.1, 130.7, 130.1, 128.7, 126.7, 124.2, 122.1, 121.5, 111.9, 60.9, 21.6, 20.9, 20.3, 14.4 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 330; HRMS (ESI) calcd for ($[\text{C}_{20}\text{H}_{21}\text{NO}_2+\text{Na}]^+$): 330.1465; found: 330.1460.

Ethyl 3-(4-bromophenyl)-5,6-dimethyl-1H-indole-2-carboxylate (3p).



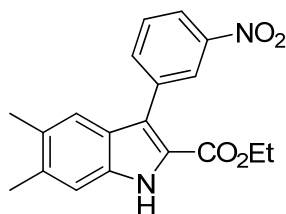
White solid, m.p. 201-202 °C; IR (KBr): 3331, 1676, 1443, 1369, 1161, 1002, 779 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.88 (s, 1 H), 7.57-7.55 (m, 2 H), 7.43-7.41 (m, 2 H), 7.31 (s, 1 H), 7.20 (s, 1 H), 4.28 (q, J = 7.1 Hz, 2 H), 2.38 (s, 3 H), 2.31 (s, 3 H), 1.25 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.1, 136.1, 135.1, 133.1, 132.5, 131.1, 130.6, 126.4, 122.7, 122.3, 121.4, 121.1, 112.1, 61.1, 21.0, 20.4, 14.4 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 394; HRMS (ESI) calcd for ($[\text{C}_{19}\text{H}_{18}\text{BrNO}_2+\text{Na}]^+$): 394.0413; found: 394.0412.

Ethyl 3-(4-methoxyphenyl)-5,6-dimethyl-1H-indole-2-carboxylate (3q).



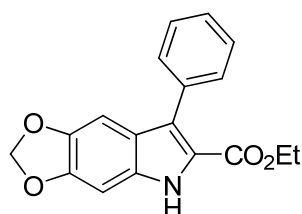
Yellow solid, m.p. 175-176 °C; IR (KBr): 3332, 1673, 1445, 1370, 1161, 1002, 778 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.83 (s, 1 H), 7.50-7.48 (m, 2 H), 7.36 (s, 1 H), 7.18 (s, 1 H), 7.00-6.99 (m, 2 H), 4.28 (q, J = 7.1 Hz, 2 H), 3.87 (s, 3 H), 2.37 (s, 3 H), 2.30 (s, 3 H), 1.25 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.4, 159.0, 135.9, 135.1, 132.0, 130.1, 126.7, 126.4, 123.9, 122.1, 121.5, 113.5, 112.0, 60.9, 55.5, 20.9, 20.3, 14.4 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 346; HRMS (ESI) calcd for ($[\text{C}_{20}\text{H}_{21}\text{NO}_3+\text{Na}]^+$): 346.1414; found: 346.1407.

Ethyl 5,6-dimethyl-3-(3-nitrophenyl)-1H-indole-2-carboxylate (3r).



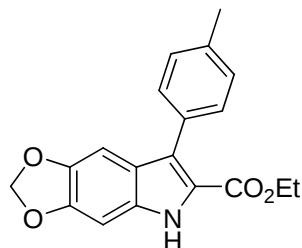
Yellow solid, m.p. 174-175 °C; IR (KBr): 3339, 1676, 1442, 1369, 1157, 1001, 779 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.04 (s, 1 H), 8.45-8.44 (m, 1 H), 8.24-8.22 (m, 1 H), 7.90-7.88 (m, 1 H), 7.62-7.59 (m, 1 H), 7.30 (s, 1 H), 7.25-7.24 (m, 1 H), 4.29 (q, J = 7.1 Hz, 2 H), 2.39 (s, 3 H), 2.32 (s, 3 H), 1.22 (t, J = 7.2 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 161.9, 148.2, 137.0, 136.5, 136.1, 135.1, 131.1, 128.8, 126.1, 126.0, 122.7, 122.1, 121.0, 120.5, 112.3, 61.3, 21.0, 20.4, 14.3 ppm; MS (ESI): m/z ($[\text{M}-\text{H}]^-$) 337; HRMS (ESI) calcd for ($[\text{C}_{19}\text{H}_{18}\text{N}_2\text{O}_4-\text{H}]^-$): 337.1194; found: 337.1179.

Ethyl 7-phenyl-5H-[1,3]dioxolo[4,5-f]indole-6-carboxylate (3s).⁷



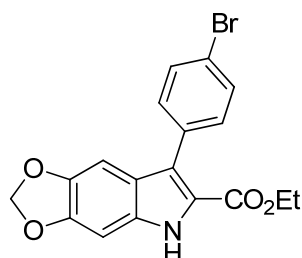
White solid, m.p. 210-211 °C; IR (KBr): 3320, 1675, 1461, 1259, 1180, 1038, 957 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.09 (s, 1 H), 7.52-7.50 (m, 2 H), 7.44-7.41 (m, 2 H), 7.37-7.35 (m, 1 H), 6.93 (s, 1 H), 6.83 (s, 1 H), 5.95 (s, 2 H), 4.26 (q, J = 7.1 Hz, 2 H), 1.21 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.1, 148.5, 144.6, 133.9, 131.9, 130.7, 128.0, 127.4, 124.9, 122.5, 121.9, 101.3, 99.3, 91.8, 60.9, 14.3 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 332; HRMS (ESI) calcd for ($[\text{C}_{18}\text{H}_{15}\text{NO}_4+\text{Na}]^+$): 332.0893; found: 332.0889.

Ethyl 7-p-tolyl-5H-[1,3]dioxolo[4,5-f]indole-6-carboxylate (3t).



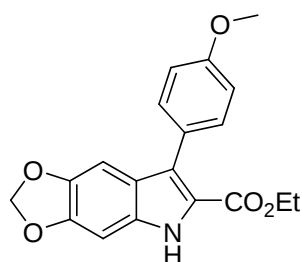
White solid, m.p. 181-182 °C; IR (KBr): 3320, 1673, 1464, 1259, 1181, 1039, 951 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.03 (s, 1 H), 7.41 (d, J = 7.8 Hz, 2 H), 7.24 (d, J = 8.0 Hz, 2 H), 6.94 (s, 1 H), 6.82 (s, 1 H), 5.94 (s, 2 H), 4.27 (q, J = 7.1 Hz, 2 H), 2.41 (s, 3 H), 1.23 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.0, 148.4, 144.5, 137.1, 131.9, 130.8, 130.6, 128.8, 125.0, 122.5, 121.8, 101.3, 99.4, 91.8, 60.8, 21.6, 14.4 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 346; HRMS (ESI) calcd for ($[\text{C}_{19}\text{H}_{17}\text{NO}_4+\text{Na}]^+$): 346.1050; found: 346.1045.

Ethyl 7-(4-bromophenyl)-5H-[1,3]dioxolo[4,5-f]indole-6-carboxylate (3u).



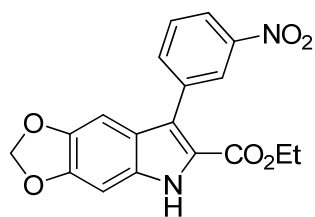
Light yellow solid, m.p. 223-224 °C; IR (KBr): 3309, 1668, 1465, 1256, 1181, 1038, 949 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.11 (s, 1 H), 7.56-7.54 (m, 2 H), 7.39-7.37 (m, 2 H), 6.87 (s, 1 H), 6.83 (s, 1 H), 5.96 (s, 2 H), 4.27 (q, J = 7.1 Hz, 2 H), 1.24 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 161.7, 148.4, 144.6, 132.7, 132.2, 131.7, 131.0, 123.3, 122.0, 121.7, 121.3, 101.2, 98.7, 91.7, 60.8, 14.2 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 410; HRMS (ESI) calcd for ($[\text{C}_{18}\text{H}_{14}\text{BrNO}_4+\text{Na}]^+$): 409.9998; found: 409.9998.

Ethyl 7-(4-methoxyphenyl)-5H-[1,3]dioxolo[4,5-f]indole-6-carboxylate (3v).⁸



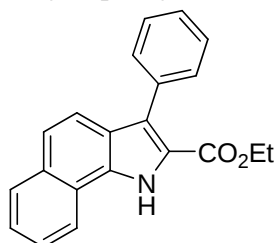
White solid, m.p. 179-180 °C; IR (KBr): 3323, 1675, 1463, 1257, 1177, 1037, 953 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.00 (s, 1 H), 7.48-7.46 (m, 2 H), 7.00 (d, J = 8.7 Hz, 2 H), 6.96 (s, 1 H), 6.84 (s, 1 H), 5.98 (s, 2 H), 4.30 (q, J = 7.1 Hz, 2 H), 3.89 (s, 3 H), 1.27 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.0, 159.1, 148.4, 144.5, 131.8, 126.1, 124.8, 122.6, 121.7, 113.5, 101.3, 99.3, 91.8, 60.8, 55.5, 14.4 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 362; HRMS (ESI) calcd for ($[\text{C}_{19}\text{H}_{17}\text{NO}_5+\text{Na}]^+$): 362.0999; found: 362.0994.

Ethyl 7-(3-nitrophenyl)-5H-[1,3]dioxolo[4,5-f]indole-6-carboxylate (3w).

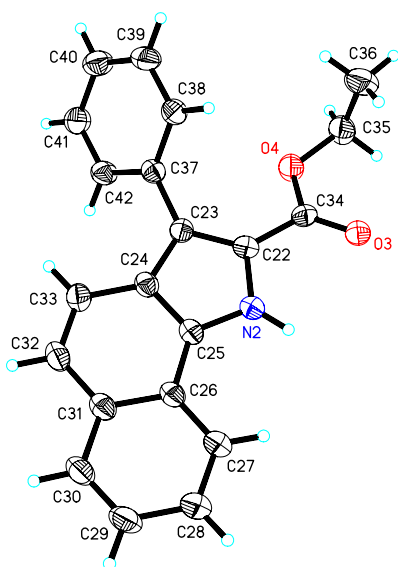


Yellow solid, m.p. 182-183 °C; IR (KBr): 3353, 1677, 1477, 1257, 1179, 1036, 951 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.25 (s, 1 H), 8.42-8.41 (m, 1 H), 8.23-8.21 (m, 1 H), 7.85 (d, J = 7.7 Hz, 1 H), 7.62-7.58 (m, 1 H), 6.87-6.86 (m, 2 H), 5.98 (s, 2 H), 4.28 (q, J = 7.1 Hz, 2 H), 1.21 (t, J = 7.1 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 161.7, 148.8, 148.2, 145.2, 136.8, 135.9, 131.9, 128.9, 125.9, 122.3, 122.0, 121.9, 101.6, 98.4, 92.1, 61.2, 14.3 ppm; MS (ESI): m/z ($[\text{M}+\text{Na}]^+$) 377; HRMS (ESI) calcd for ($[\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_6+\text{Na}]^+$): 377.0744; found: 377.0743.

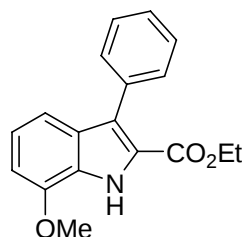
Ethyl 3-phenyl-1H-benzo[*g*]indole-2-carboxylate (3x).⁹



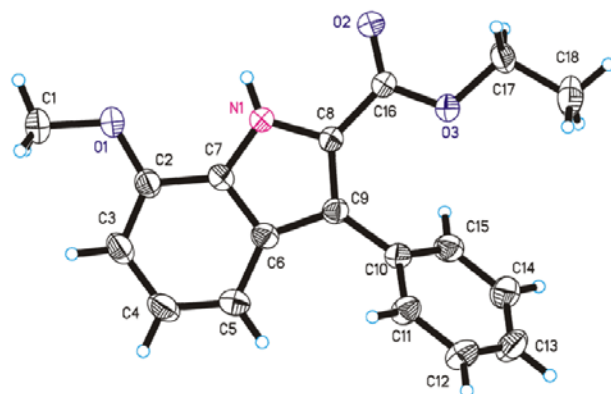
Colorless crystal, m.p. 185-186 °C; IR (KBr): 3323, 1674, 1450, 1365, 1180, 1034, 810 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.96 (s, 1 H), 8.22 (d, J = 8.1 Hz, 1 H), 7.91 (d, J = 7.9 Hz, 1 H), 7.62-7.58 (m, 4 H), 7.54-7.46 (m, 4 H), 7.41-7.40 (m, 1 H), 4.34 (q, J = 7.1 Hz, 2 H), 1.25 (t, J = 7.2 Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.5, 133.9, 132.5, 132.0, 131.0, 129.1, 128.0, 127.5, 126.3, 126.1, 124.3, 122.4, 121.8, 121.7, 120.9, 120.5, 61.2, 14.4 ppm; MS (ESI): m/z ($[\text{M}-\text{H}]^-$) 314; HRMS (ESI) calcd for ($[\text{C}_{21}\text{H}_{17}\text{NO}_2-\text{H}]^-$): 314.1187; found: 314.1175.



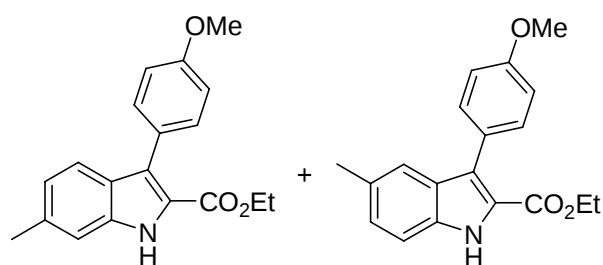
Ethyl 7-methoxy-3-phenyl-1H-indole-2-carboxylate (3y)



Colorless solid, m.p. 97-98 °C IR (KBr): 3294, 1697, 1443, 1390, 1328, 1261, 1094, 781, 695 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 9.20 (s, 1 H), 7.59 (d, $J = 7.0$ Hz, 2 H), 7.47 (t, $J = 7.5$ Hz, 2 H), 7.39 (t, $J = 7.5$ Hz, 1 H), 7.25 (d, $J = 8.0$ Hz, 1 H), 7.08 (t, $J = 7.5$ Hz, 1 H), 6.78 (t, $J = 7.5$ Hz, 1 H), 4.32 (q, $J = 7.5$ Hz, 2 H), 4.02 (s, 3 H), 1.27 (t, $J = 7.5$ Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.0, 146.6, 133.9, 130.9, 129.3, 127.9, 127.4, 127.2, 124.7, 122.8, 121.5, 114.1, 104.7, 61.0, 55.7, 14.3 ppm; MS (ESI): m/z ($[\text{M}+\text{H}]^+$) 296; HRMS (ESI) calcd for ($[\text{C}_{18}\text{H}_{17}\text{NO}_3 + \text{Na}]^+$): 318.1101; found: 318.1090.



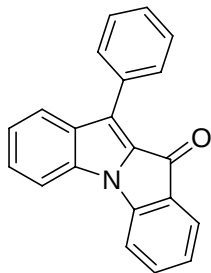
**Ethyl 3-(4-methoxyphenyl)-6-methyl-1H-indole-2-carboxylate +
Ethyl 3-(4-methoxyphenyl)-5-methyl-1H-indole-2-carboxylate (3z)**



White solid, m.p. 134-137 °C IR (KBr): 3329, 1672, 1547, 1505, 1383, 1333, 1263, 1179, 1039, 833 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.95 (s, 0.69 H), 8.80 (s, 0.27 H), 7.54-7.50 (m, 2.24 H), 7.42 (s, 0.71 H), 7.34 (d, $J = 8.5$ Hz, 0.73 H), 7.22-7.19 (m, 1 H), 7.03-7.00 (m, 2.25 H), 4.31 (q, $J = 7.0$ Hz, 2 H), 3.89 (s, 3 H), 2.49 (s, 0.83 H), 2.43 (s, 2.14 H), 1.28 (t, $J = 7.1$ Hz, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 162.3, 159.1, 136.4, 136.3, 134.4, 132.0, 130.4, 128.5, 127.9, 126.1, 123.8, 123.1, 122.9, 121.7, 121.2, 113.5, 111.6, 111.5, 60.9, 55.5, 22.2, 21.7, 14.4 ppm; MS (ESI): m/z ($[\text{M}-\text{H}]$) 308; HRMS (ESI) calcd for ($[\text{C}_{19}\text{H}_{19}\text{NO}_3 + \text{Na}]^+$): 332.1257; found: 332.1246.

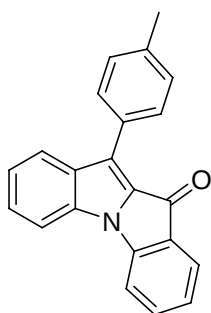
Characterization of 4

5-phenyl-iodolo[1,2-*a*]indol-5-one (4a)



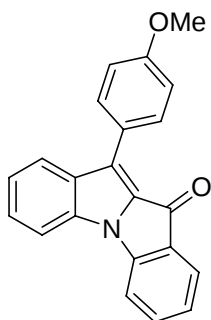
Yellow solid m.p. 189-191 °C IR (KBr): 1687, 1622, 1549, 1495, 1473, 1385, 1178, 939, 739, 687 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.01 (d, $J = 7.0$ Hz, 2 H), 7.92 (d, $J = 8.0$ Hz, 1 H), 7.68 (d, $J = 7.5$ Hz, 1 H), 7.57-7.50 (m, 4 H), 7.46 (t, $J = 7.5$ Hz, 2 H), 7.40 (d, $J = 7.5$ Hz, 1 H), 7.18 (t, $J = 7.5$ Hz, 1 H), 7.09 (t, $J = 7.5$ Hz, 1 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 181.0, 144.9, 135.3, 134.8, 131.6, 131.5, 131.4, 130.1, 129.4, 129.1, 128.9, 128.6, 126.0, 125.3, 124.6, 124.0, 122.4, 111.7, 111.6 ppm; ; MS (ESI): m/z ($[\text{M}+\text{H}]^+$) 296; HRMS (ESI) calcd for ($[\text{C}_{21}\text{H}_{13}\text{NO} + \text{Na}]^+$): 318.0889; found: 318.0886.

5-p-tolyl-iodolo[1,2-*a*]indol-5-one (4b)



Yellow solid m.p. 152-154 °C IR (KBr): 1688, 1620, 1554, 1508, 1475, 1385, 1323, 1176, 935, 733, 706 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 7.92 (d, $J = 8.0$ Hz, 3 H), 7.68 (d, $J = 7.5$ Hz, 1 H), 7.55 (d, $J = 8.0$ Hz, 1 H), 7.51 (t, $J = 8.0$ Hz, 1 H), 7.45 (t, $J = 7.5$ Hz, 1 H), 7.39 (d, $J = 8.0$ Hz, 1 H), 7.36 (d, $J = 8.0$ Hz, 2 H), 7.17 (t, $J = 7.5$ Hz, 1 H), 7.09 (t, $J = 7.5$ Hz, 1 H), 2.46 (s, 3 H) ppm; ^{13}C NMR (125 MHz, CDCl_3): δ 181.0, 144.9, 139.2, 135.2, 134.9, 131.5, 131.3, 130.2, 129.6, 129.3, 128.7, 128.6, 126.3, 125.2, 124.6, 123.8, 122.3, 111.7, 111.5, 21.8 ppm; ; MS (ESI): m/z ($[\text{M}+\text{H}]^+$) 310; HRMS (ESI) calcd for ($[\text{C}_{22}\text{H}_{15}\text{NO} + \text{H}]^+$): 310.1226; found: 310.1225.

5-(4-methoxyphenyl)-iodolo[1,2-*a*]indol-5-one (4c)

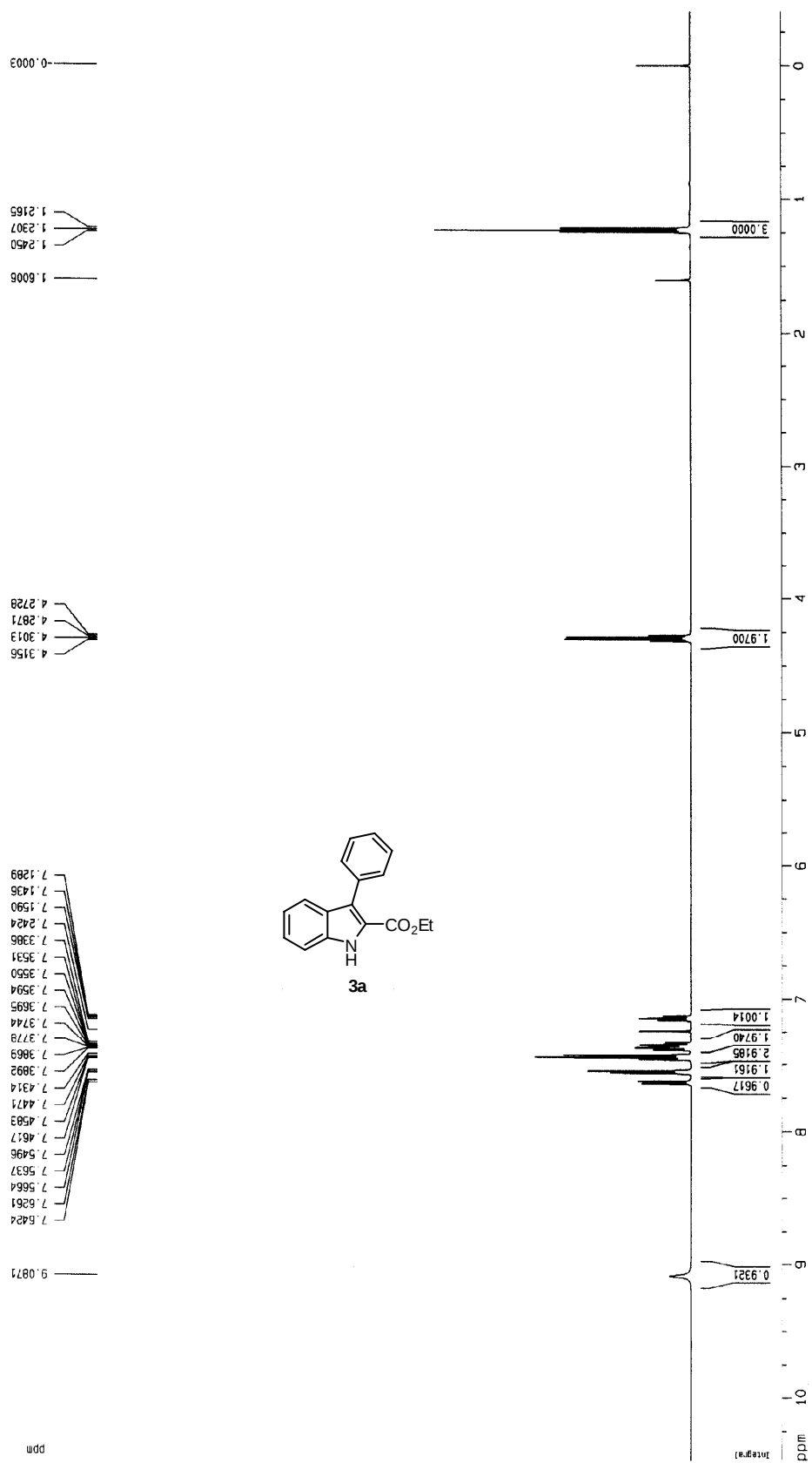


Yellow solid m.p. 171-173 °C IR (KBr): 1686, 1620, 1609, 1552, 1508, 1473, 1385, 1246, 1174, 937, 737 cm⁻¹; ¹H NMR (500 MHz, CDCl₃): δ 8.00 (d, *J* = 8.7 Hz, 2 H), 7.91 (d, *J* = 8.1 Hz, 1 H), 7.68 (d, *J* = 7.4 Hz, 1 H), 7.54 (d, *J* = 8.0 Hz, 1 H), 7.50 (t, *J* = 7.5 Hz, 1 H), 7.44 (t, *J* = 7.5 Hz, 1 H), 7.39 (d, *J* = 8.0 Hz, 1 H), 7.16 (t, *J* = 7.5 Hz, 1 H), 7.09-7.06 (m, 3 H), 3.91 (s, 3 H) ppm; ¹³C NMR (125 MHz, CDCl₃): δ 180.9, 160.4, 144.8, 135.1, 135.0, 131.5, 131.0, 130.8, 130.2, 128.6, 126.2, 125.2, 124.6, 124.2, 123.7, 122.3, 114.3, 111.7, 111.5, 55.6 ppm; MS (ESI): *m/z* ([M+Na]⁺) 348; HRMS (ESI) calcd for ([C₂₂H₁₅NO₂ +Na]⁺): 348.0995; found: 348.0981.

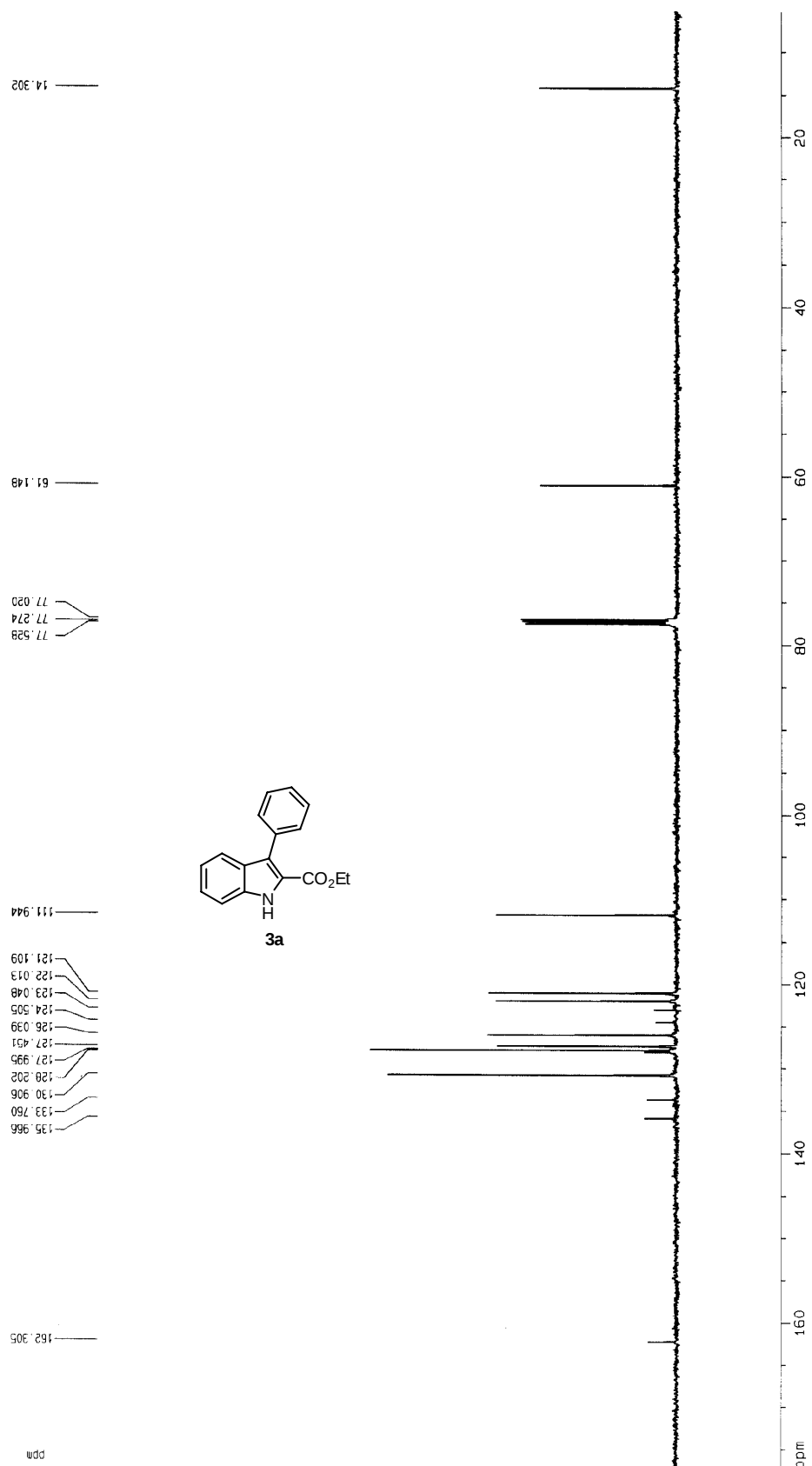
References

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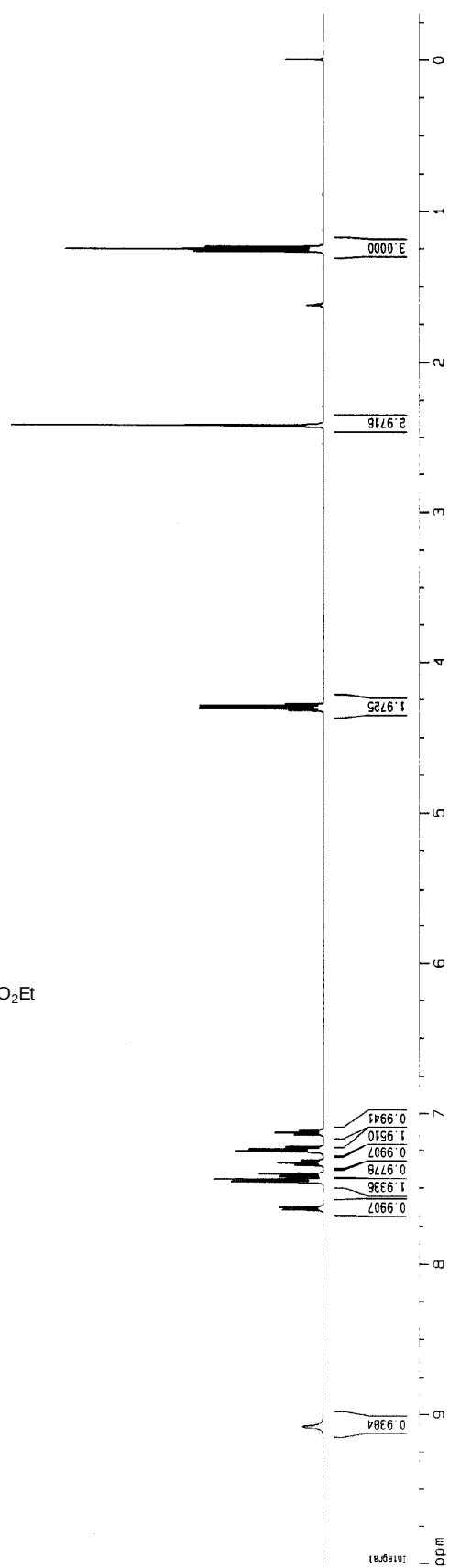
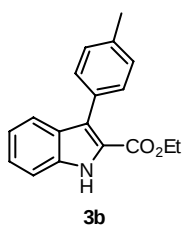
Zhejiang University Avance DMX 500
 GNP 5mm Sample: CZB-n-5 in CDCl₃



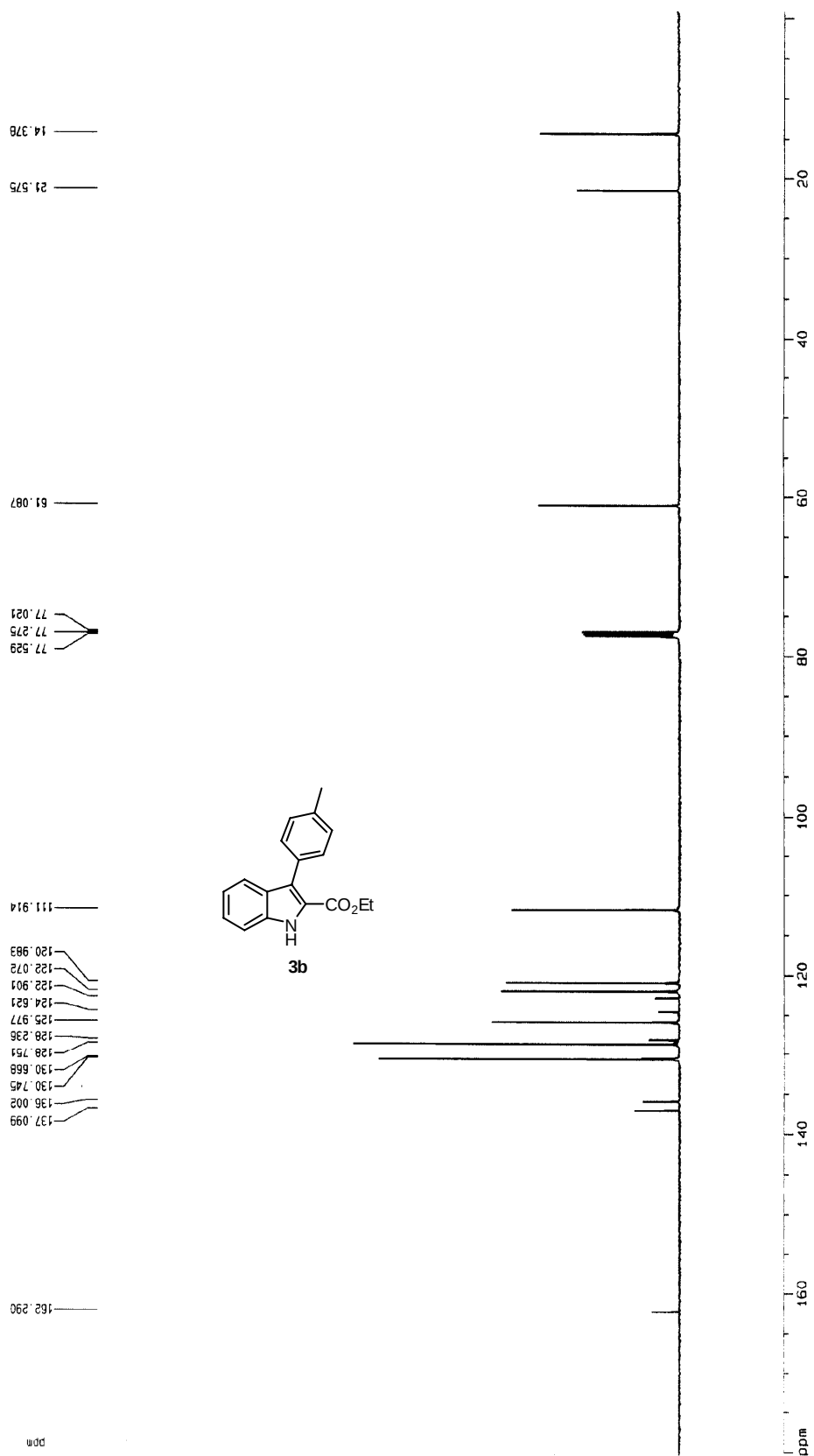
Zhejiang University Avance DMX 500
 GNP 5mm Sample: CZB-n-5 in CDCL3



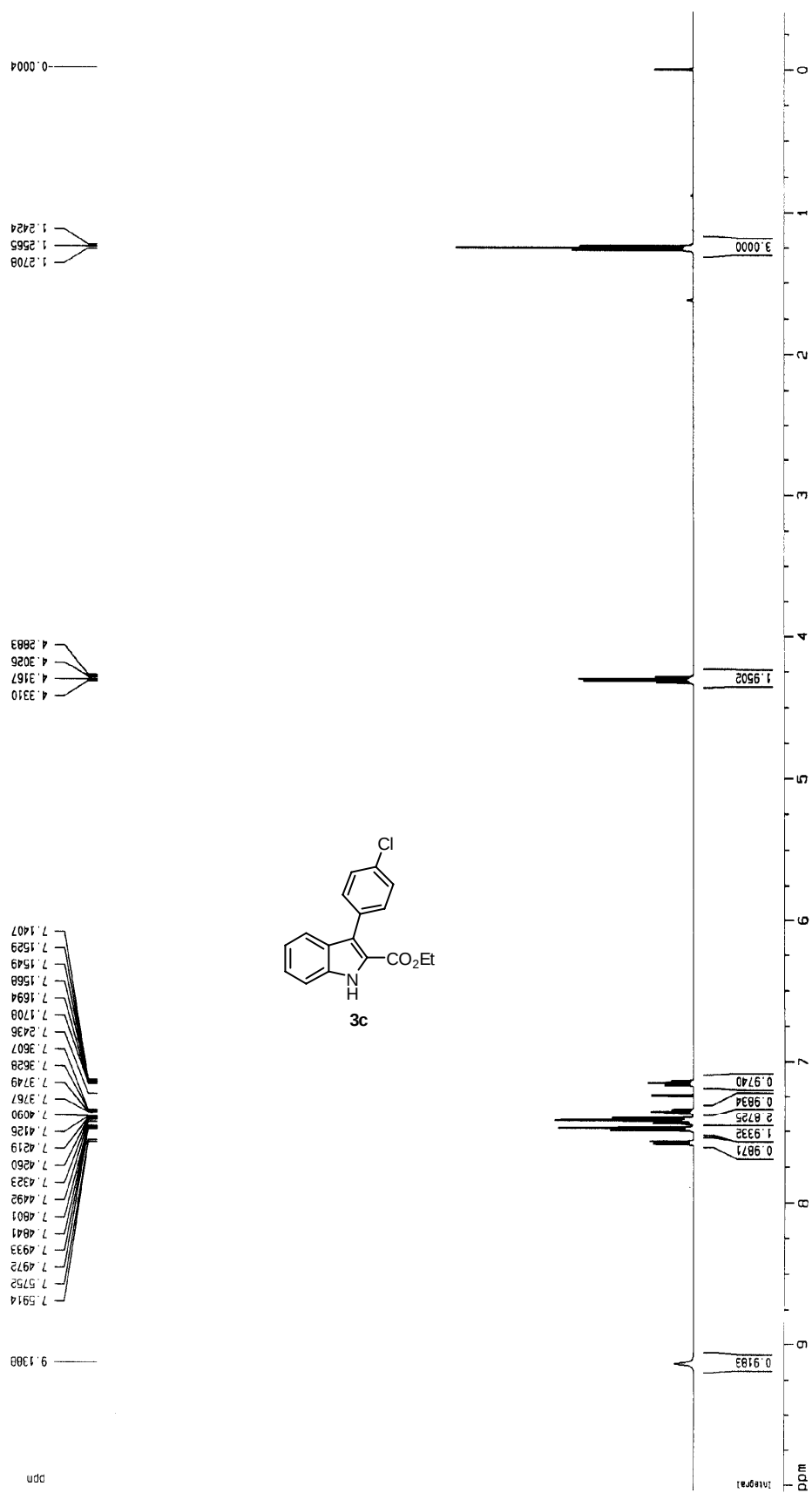
Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZB-n-24 in CDCl3



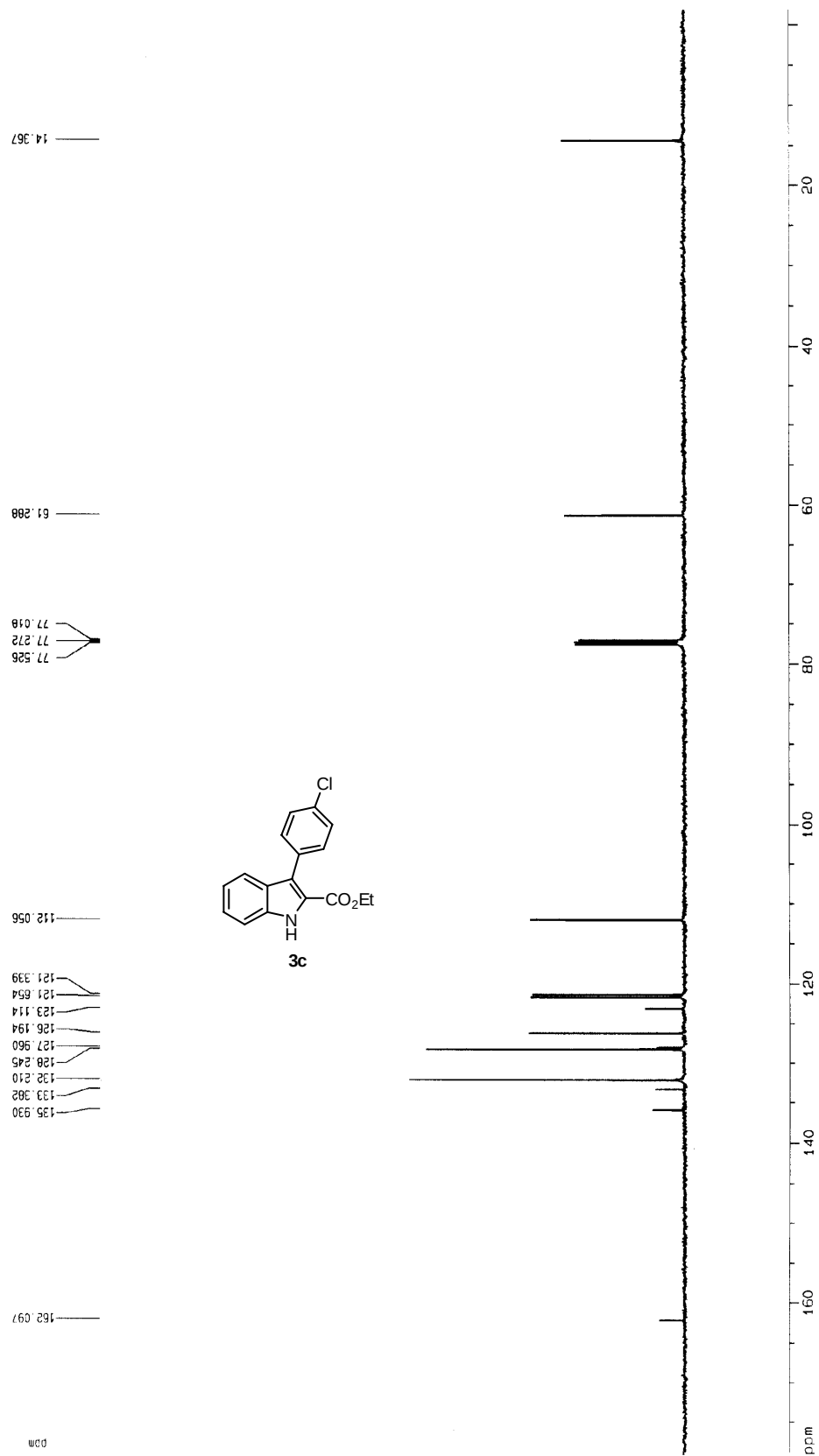
Zhejiang University Avance DMX 500
QNP 5mm Sample: C2B-n-24 in CDCl3



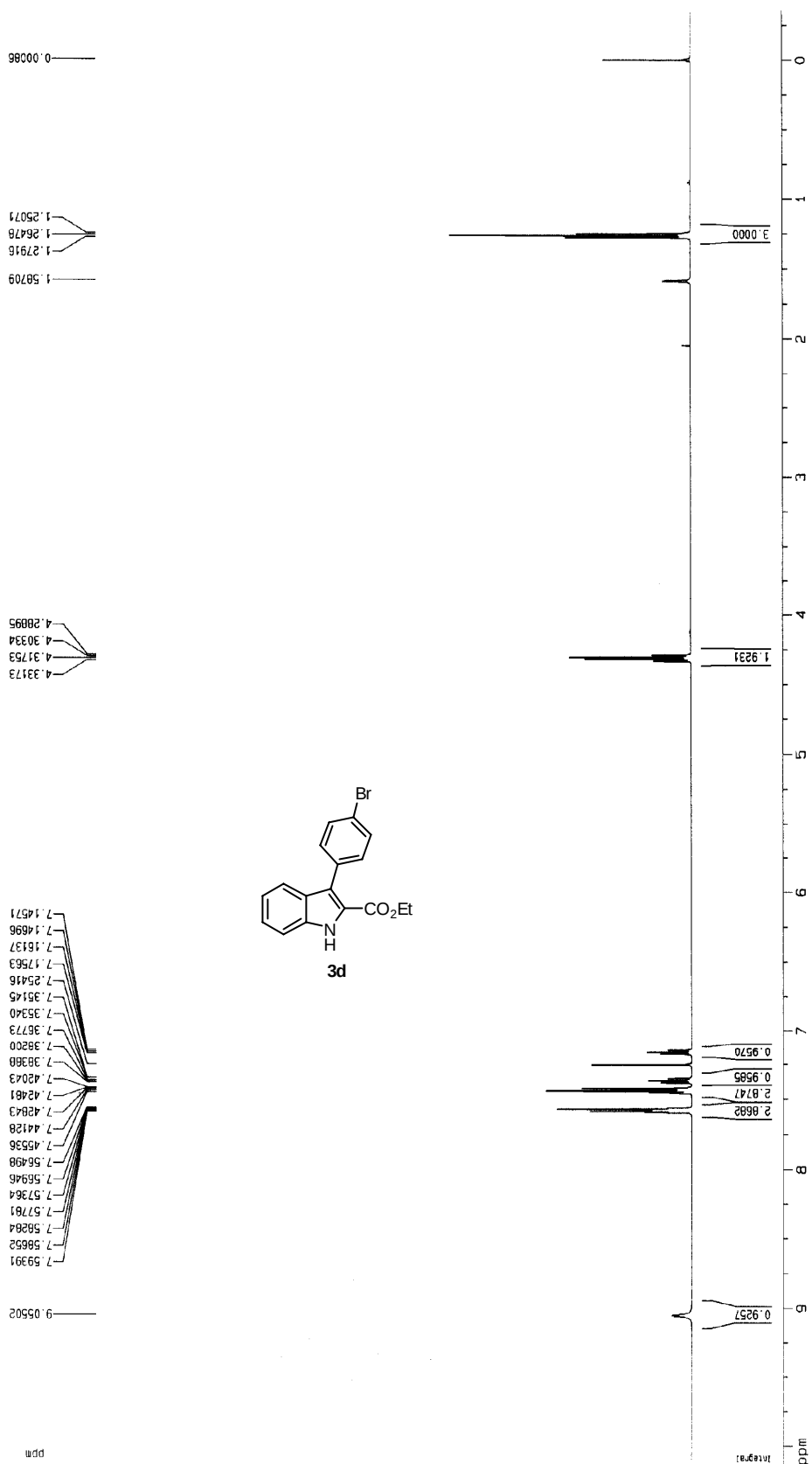
Zhejiang University Avance DMX 500
 GNP 5mm Sample: CZB-n-7 in CDCl₃



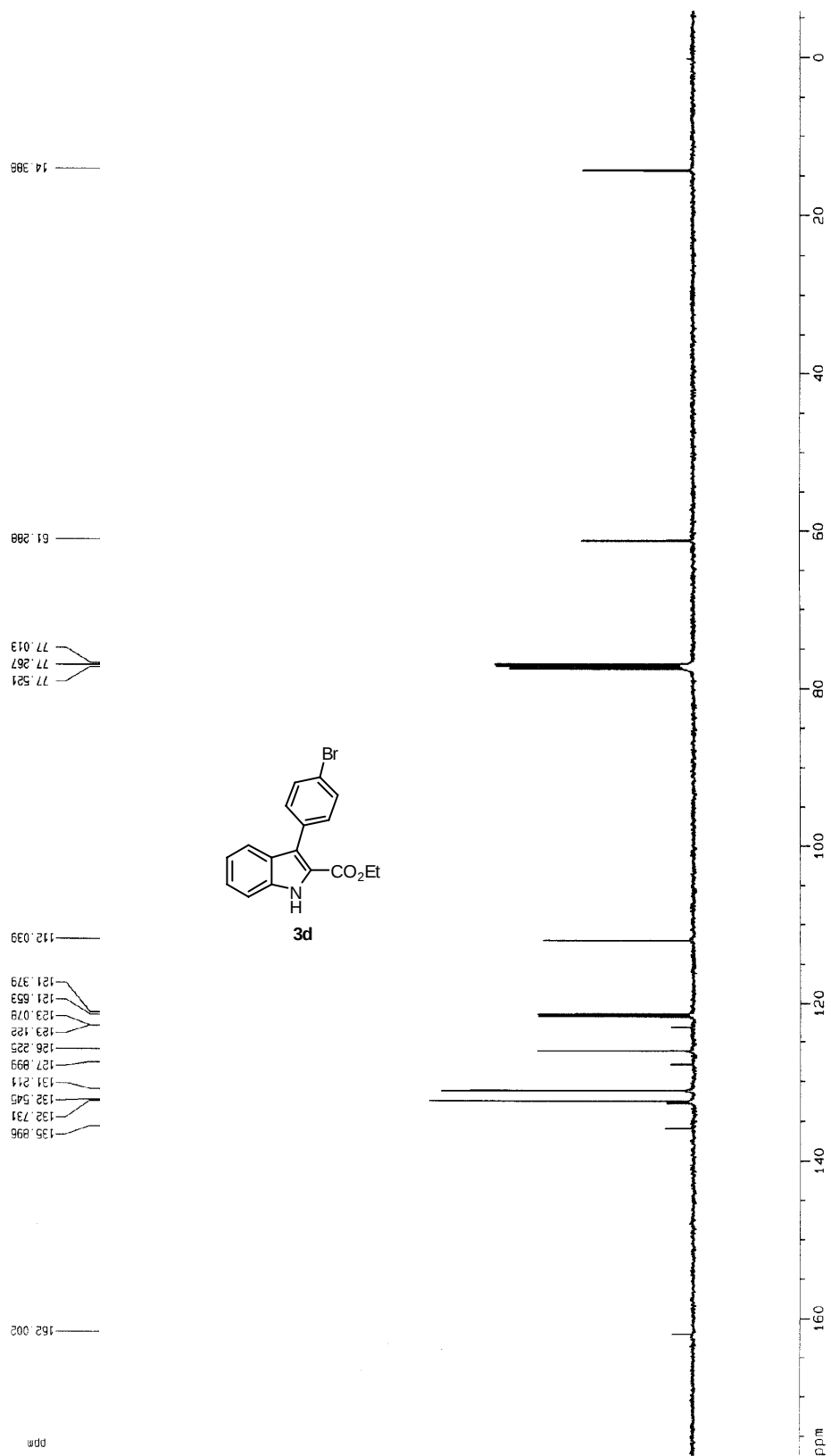
Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-7 in CDCl₃



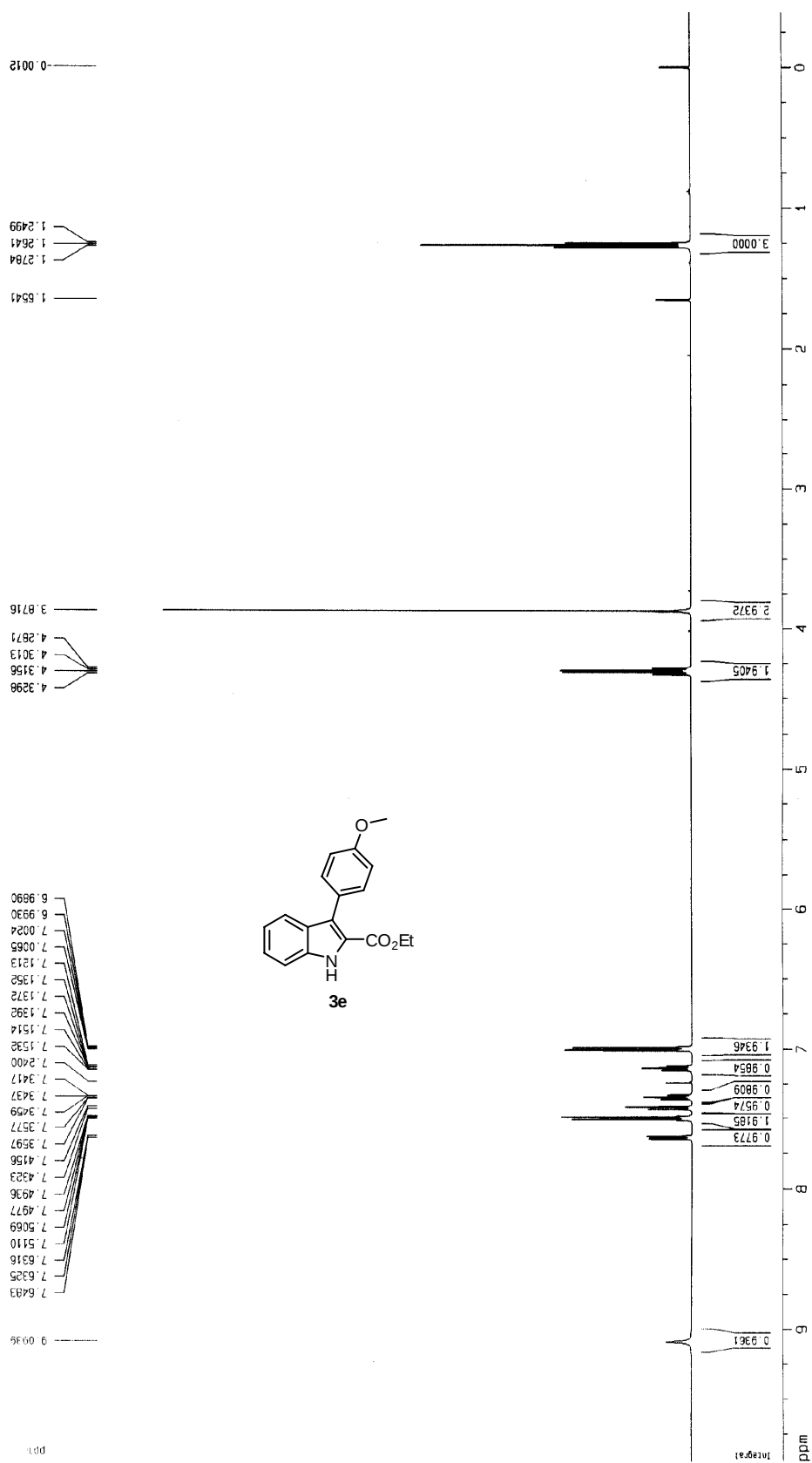
Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-4 in CDCl₃



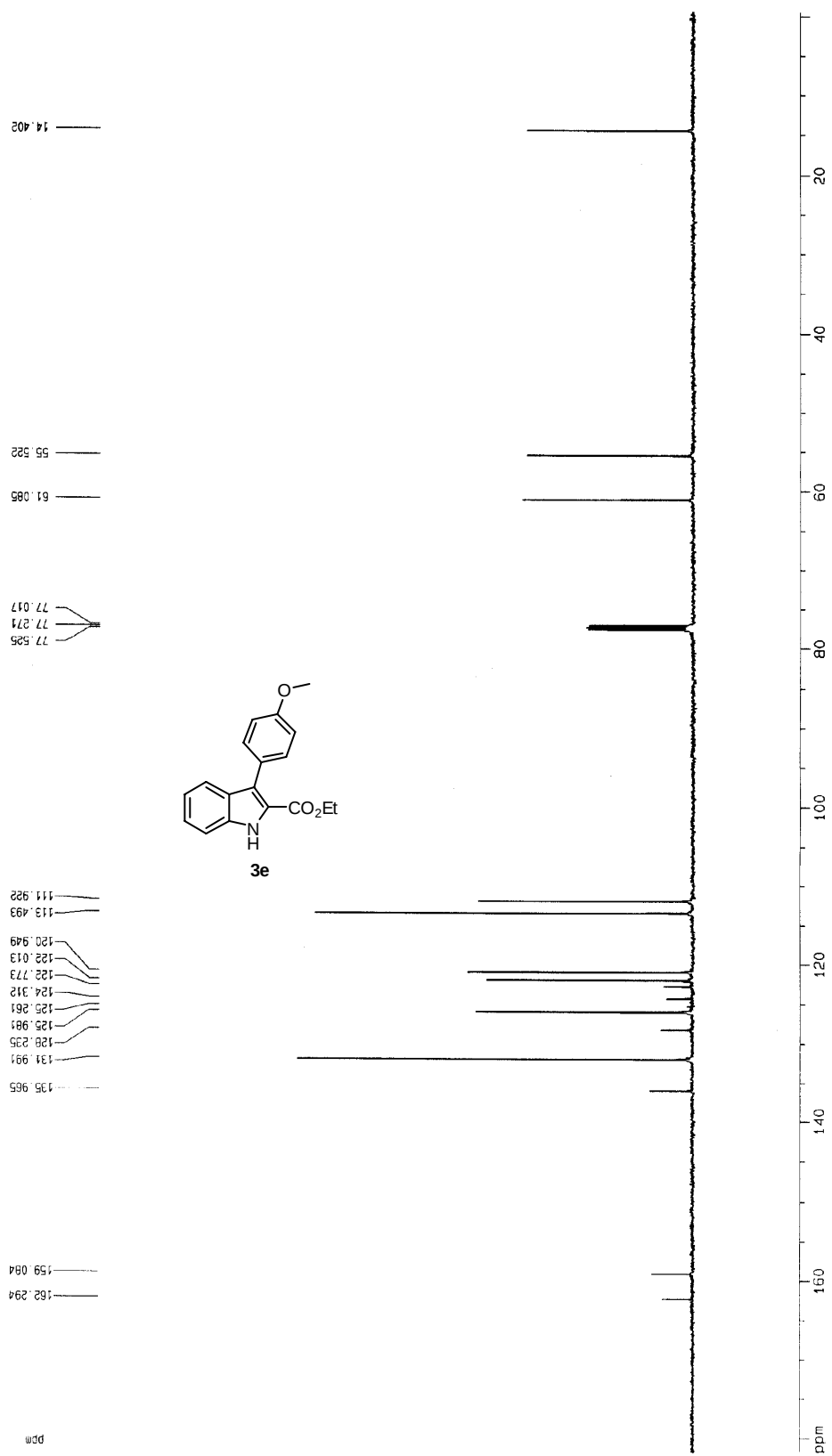
Zhejiang University Avance DMX 500
 QNP 5mm Sample: C2B-n-4 in CDCL3

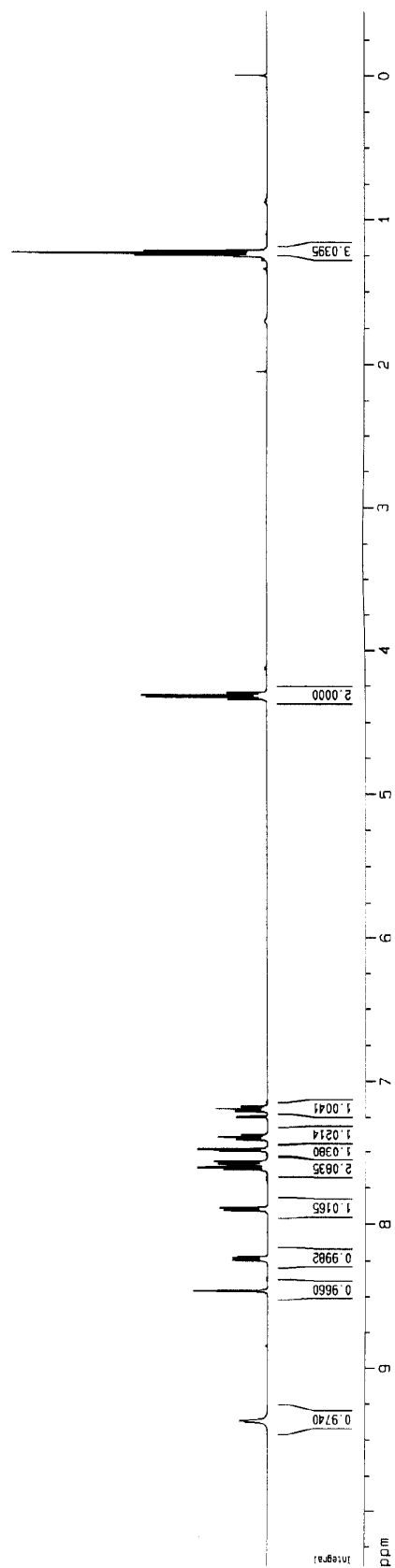
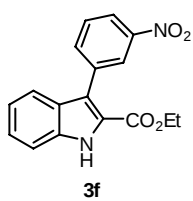


Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-1 in CDCl₃



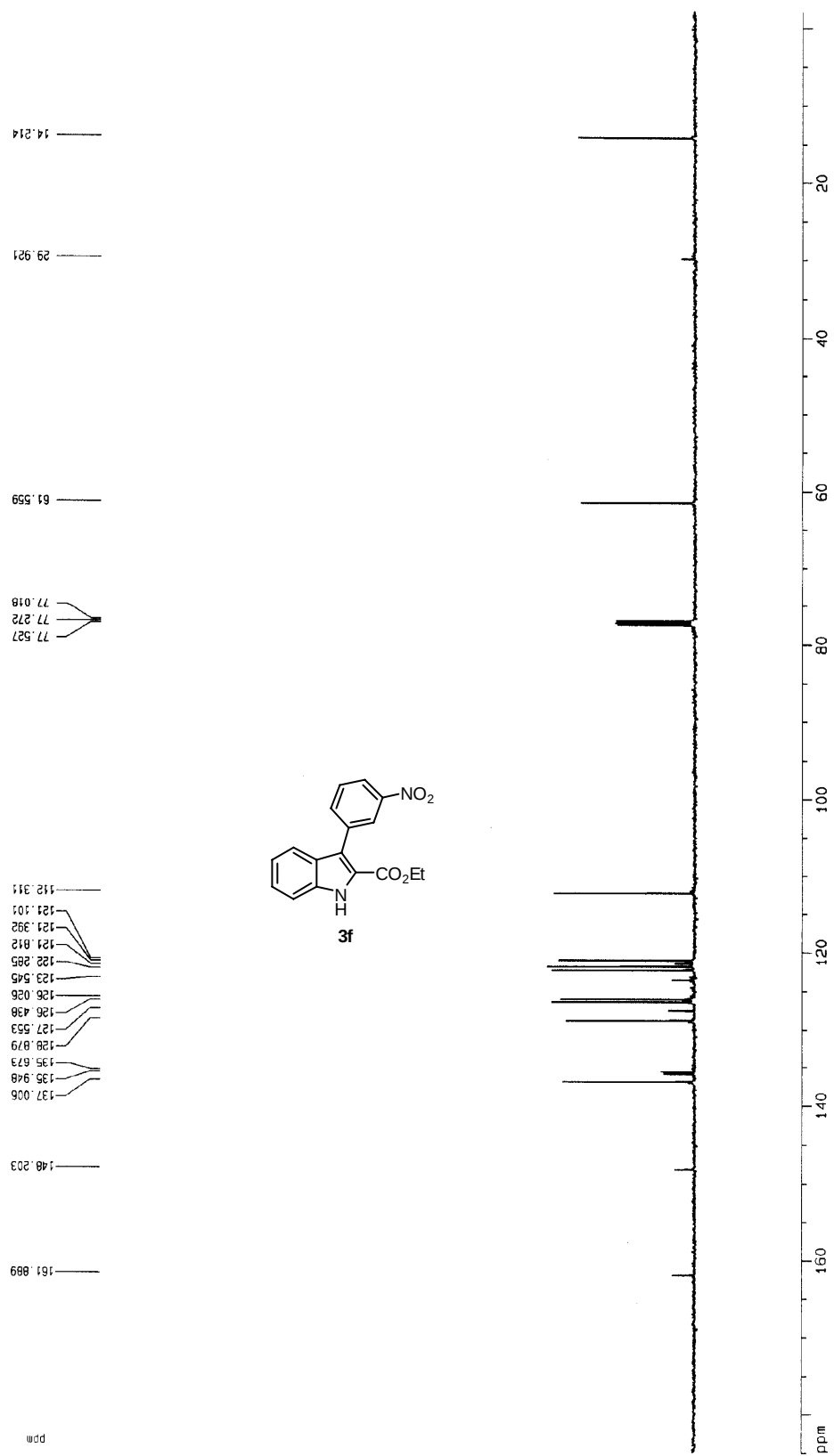
Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-1 in CDCl₃

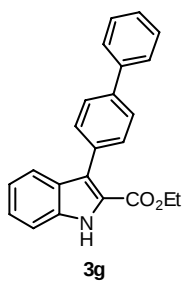
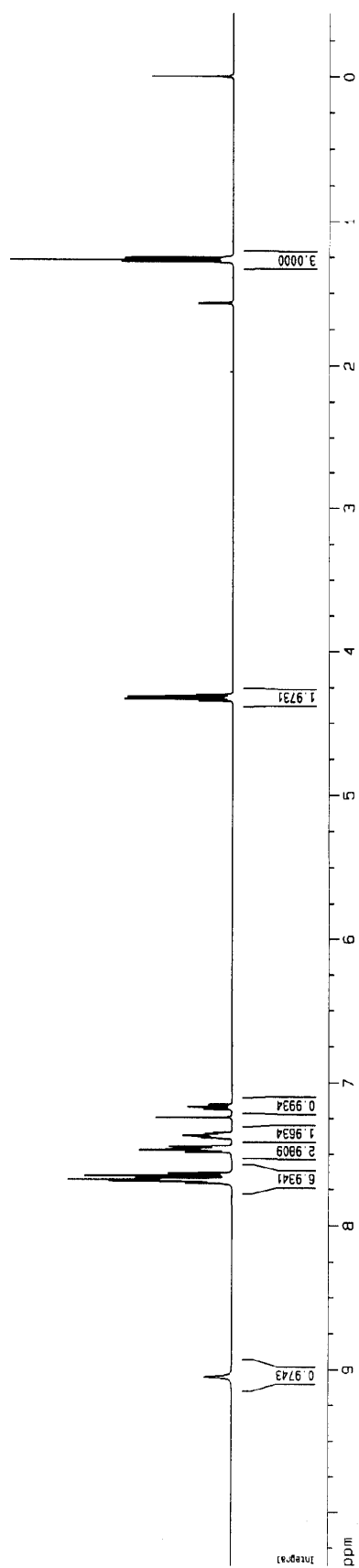




Zhejiang University Avance DMX 500
 GNP 5mm Sample: CZB-n-2 in CDCl₃

Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-2 in CDCl₃





0.0006
1.57246
1.29457
1.27027
1.25608

4.34687
4.32860
4.31034
4.30415

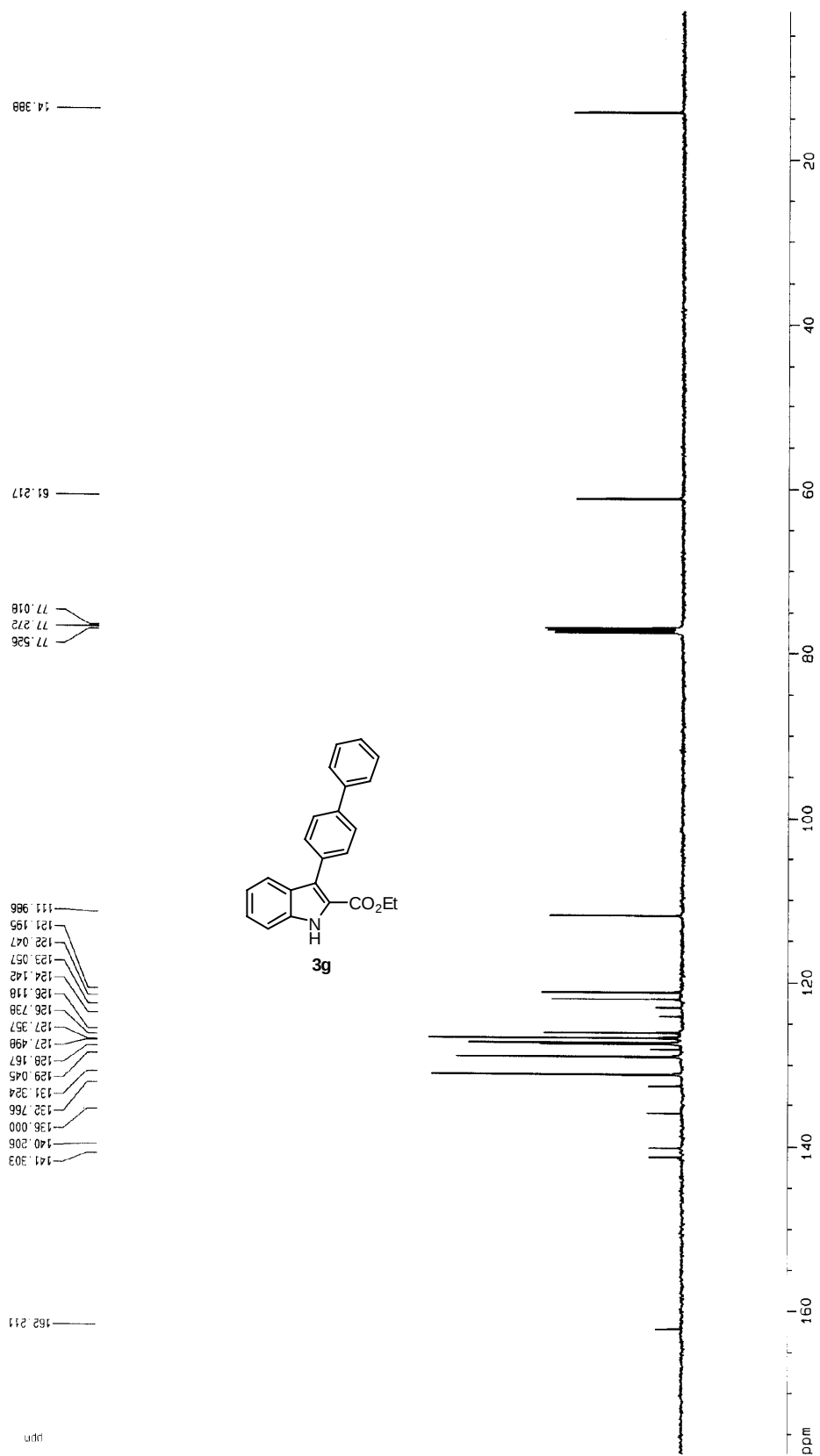
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7.69519
7.69330
7.68631
7.68276
7.67859
7.65937
7.64286
7.48680
7.47174
7.46379
7.45616
7.44711
7.36609
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7.37196
7.36919
7.36570
7.35730
7.24445
7.18525
7.16992

9.05096

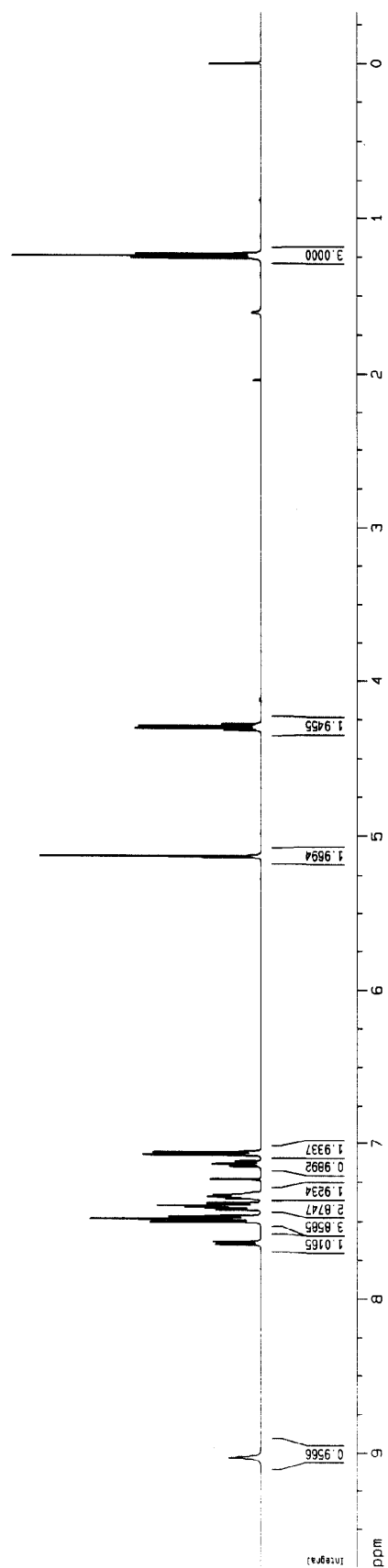
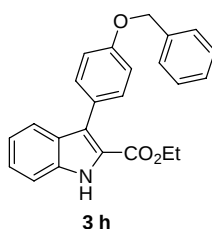
ppm

Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-6 in CDCl3

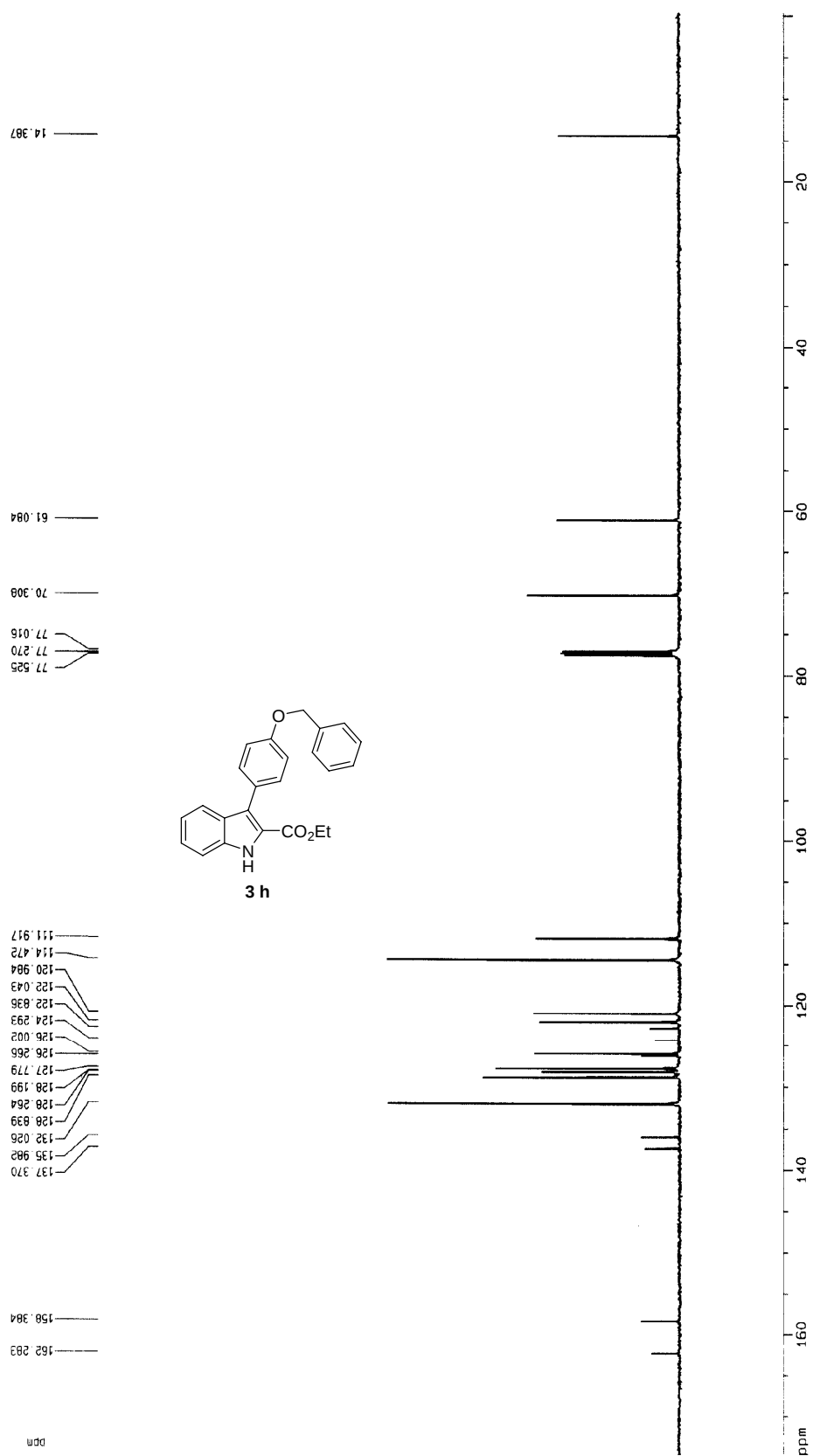
Zhejiang University Avance DMX 500
 GNP 5mm Sample: CZB-n-6 in CDCL3



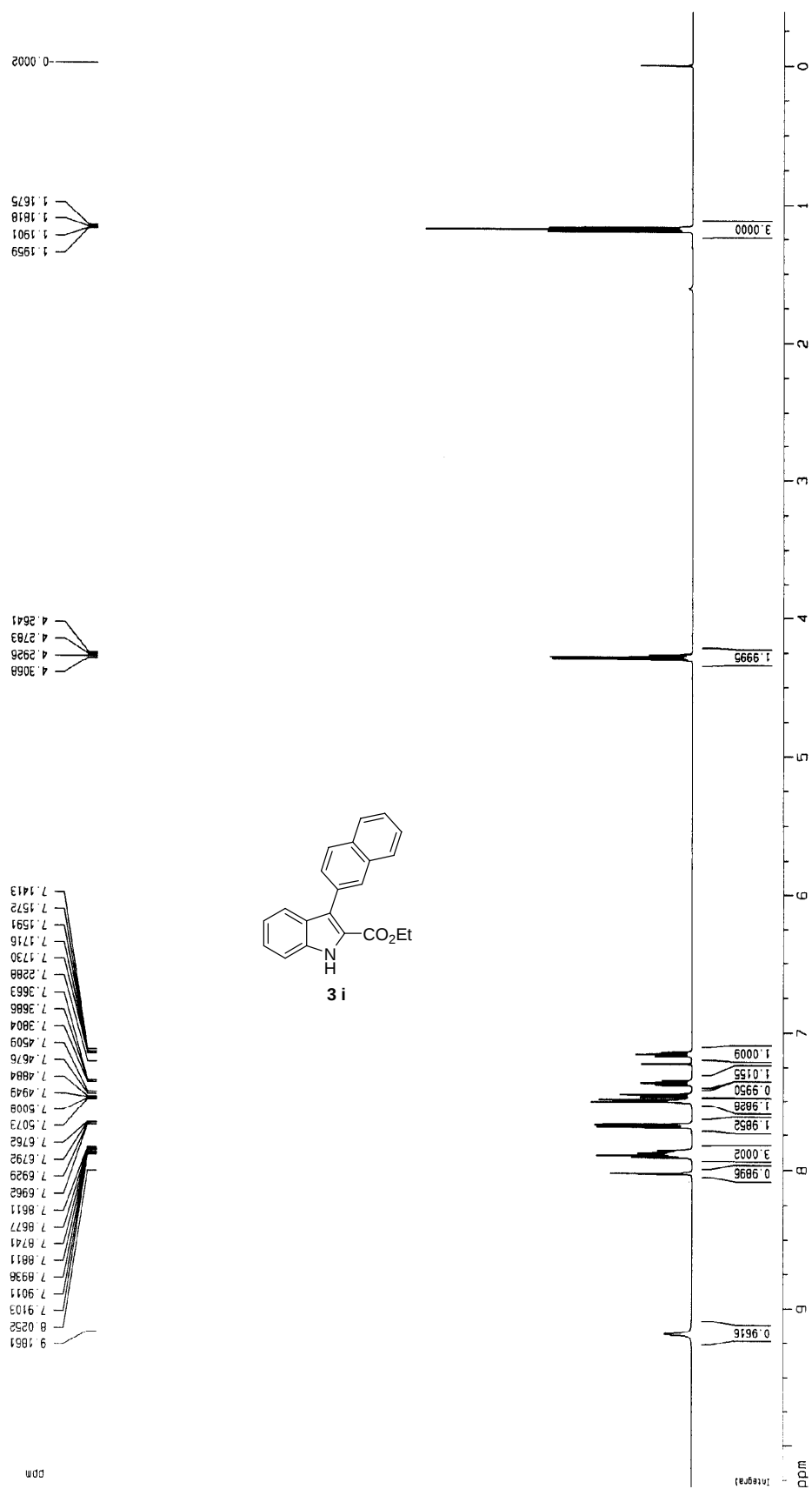
Zhejiang University Avance DMX 500
QNP 5mm Sample: C2B-n-10 in CDCL3



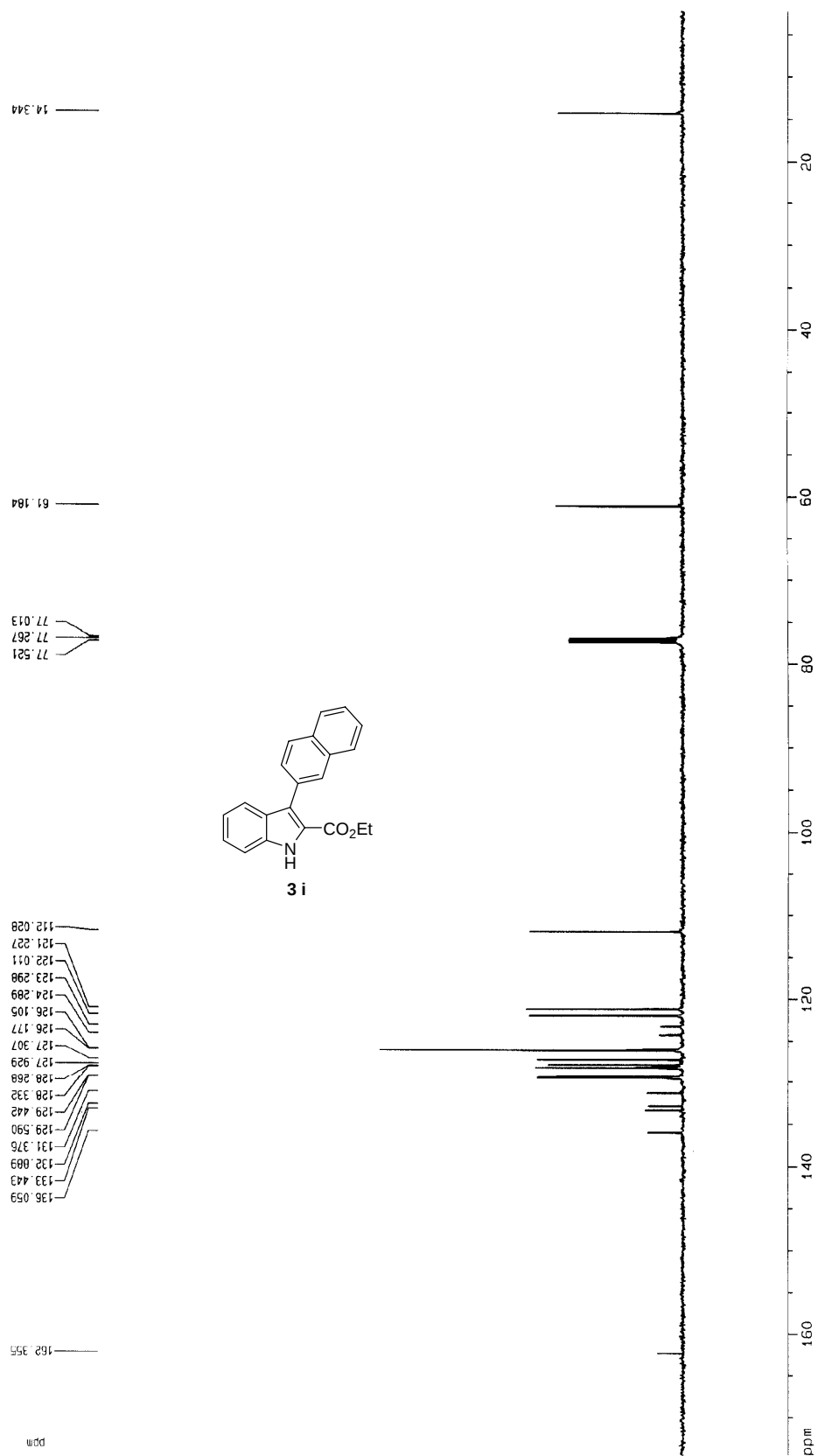
Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZB-n-10 in CDCl₃



Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-8 in CDCl₃



Zhejiang University Avance DMX 500
 GNP 5mm Sample: CZB-n-8 in CDCL3



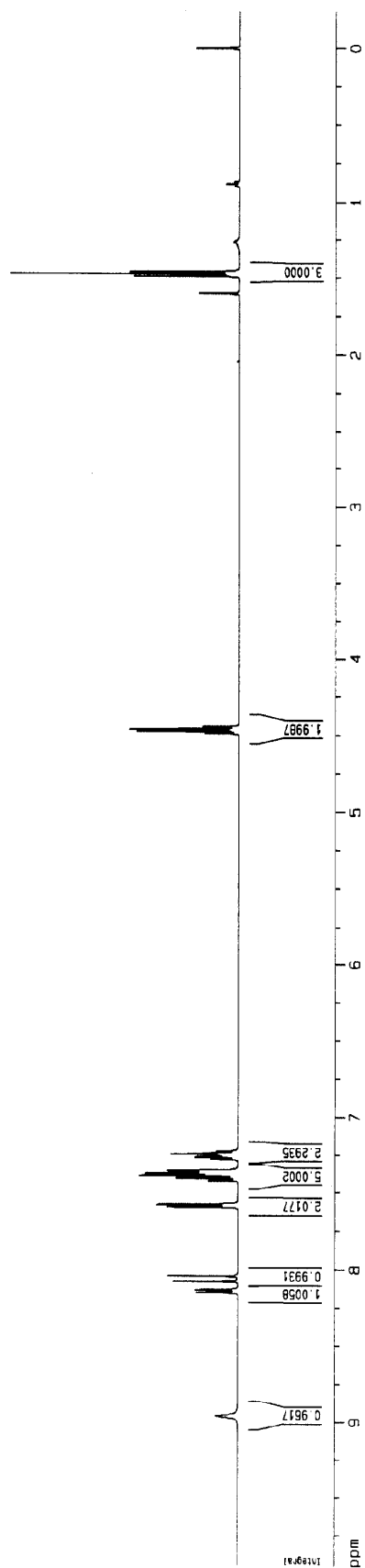
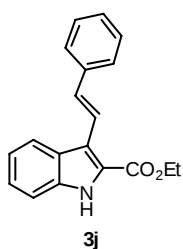
Zhejiang University Avance DMX 500
QNP 5mm Sample: C2B-n-11 in CDCl3

0.00111
1.60188
1.48769
1.47341
1.45910

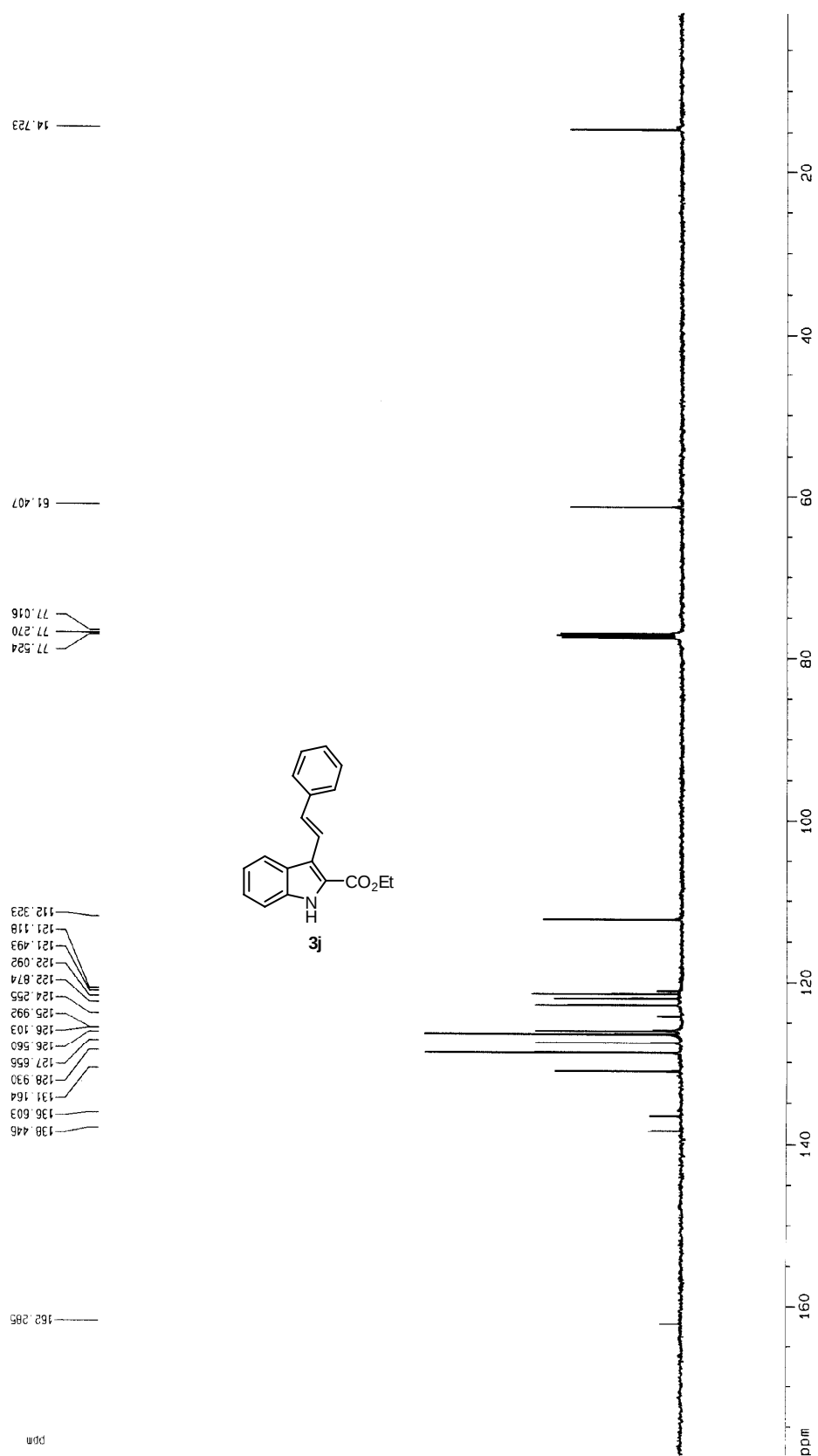
4.48699
4.47276
4.45854
4.44423

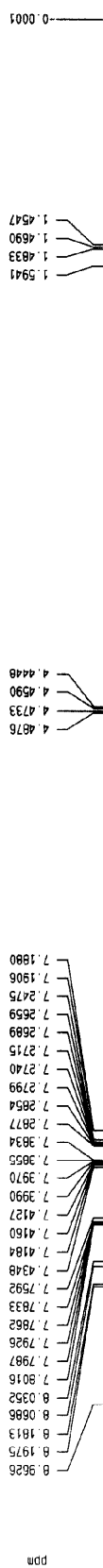
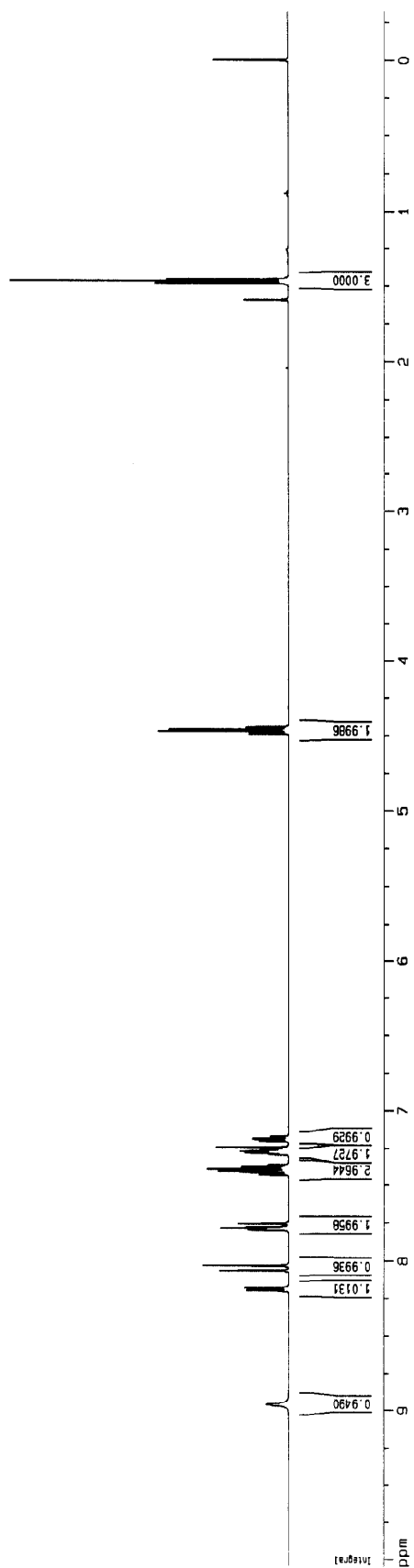
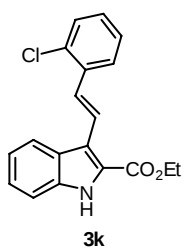
8.95992
8.14672
8.13029
8.07395
8.04043
7.59356
7.57643
7.42440
7.40769
7.39088
7.36407
7.36213
7.37527
7.37053
7.36840
7.36031
7.35741
7.27114
7.26239
7.25466
7.25255
7.24777
7.24577
7.24178
7.23863
7.22438
7.22247

ppm



Zhejiang University Avance DMX 500
 GNP 5mm Sample: C2B-n-11 in CDCl₃





Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-12 in CDCl₃

Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-12 in CDCl₃

135.560
135.436
133.528
130.002
128.511
127.194
126.965
126.343
126.225
125.910
124.542
124.339
122.802
121.878
121.036
112.320

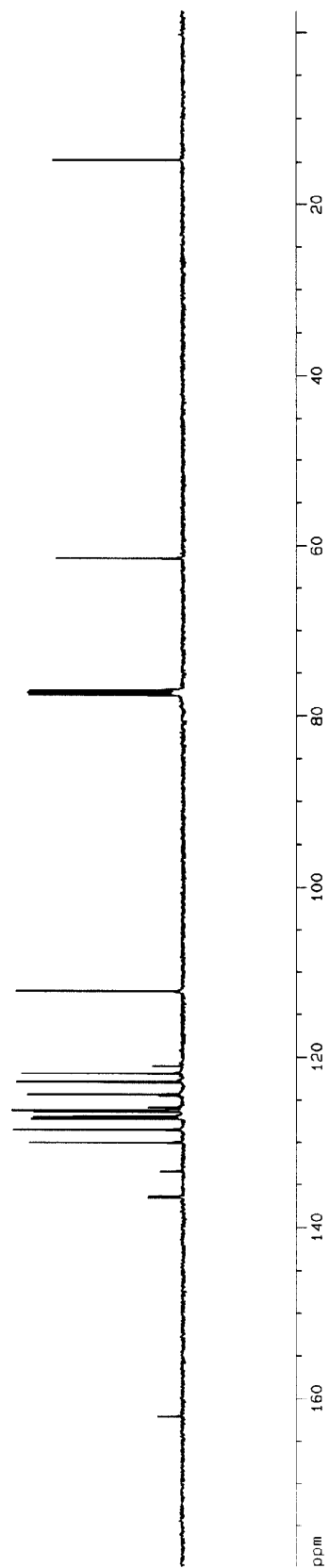
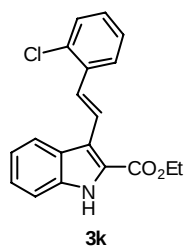
162.107

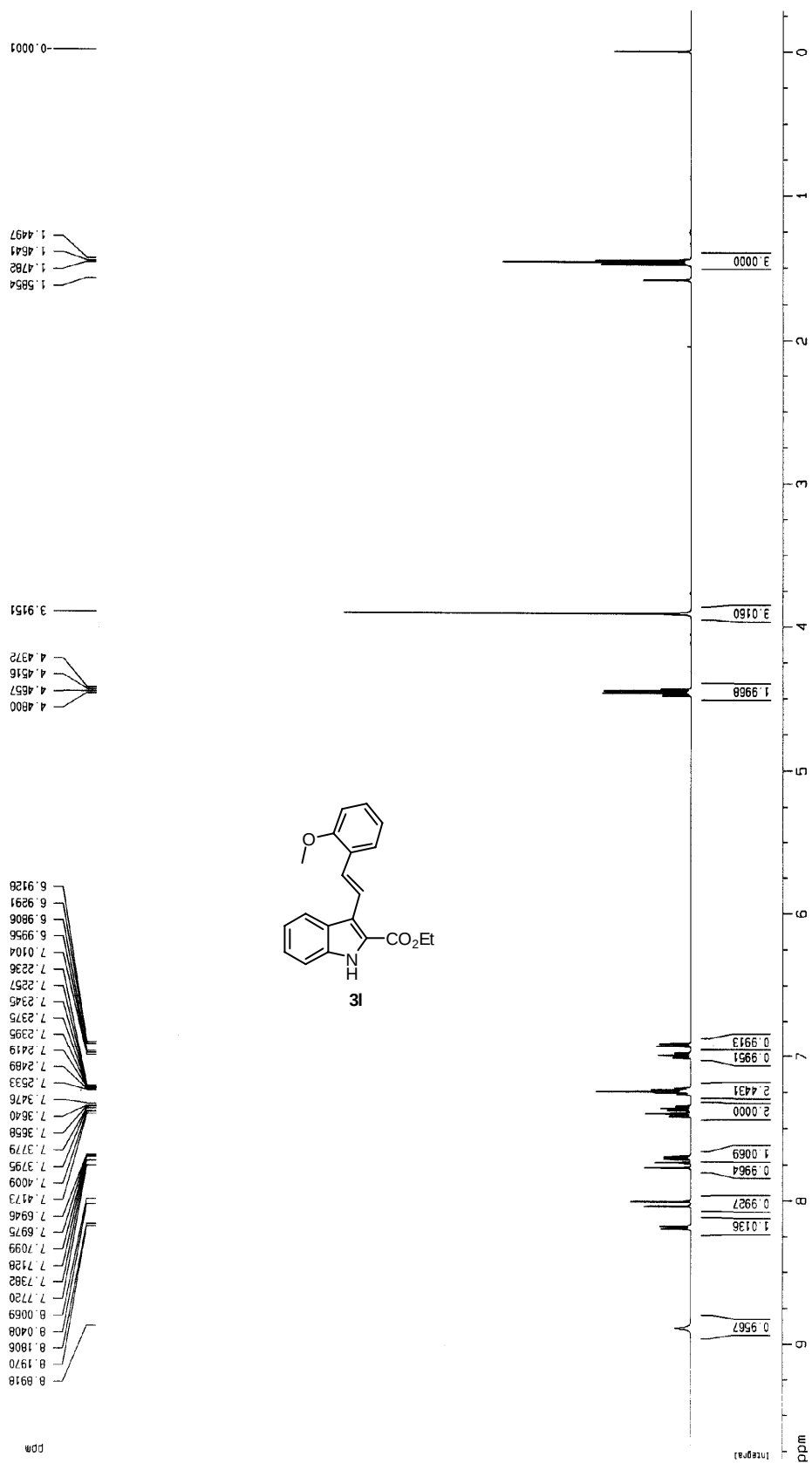
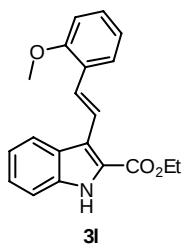
ppm

77.521
77.267
77.013

61.454

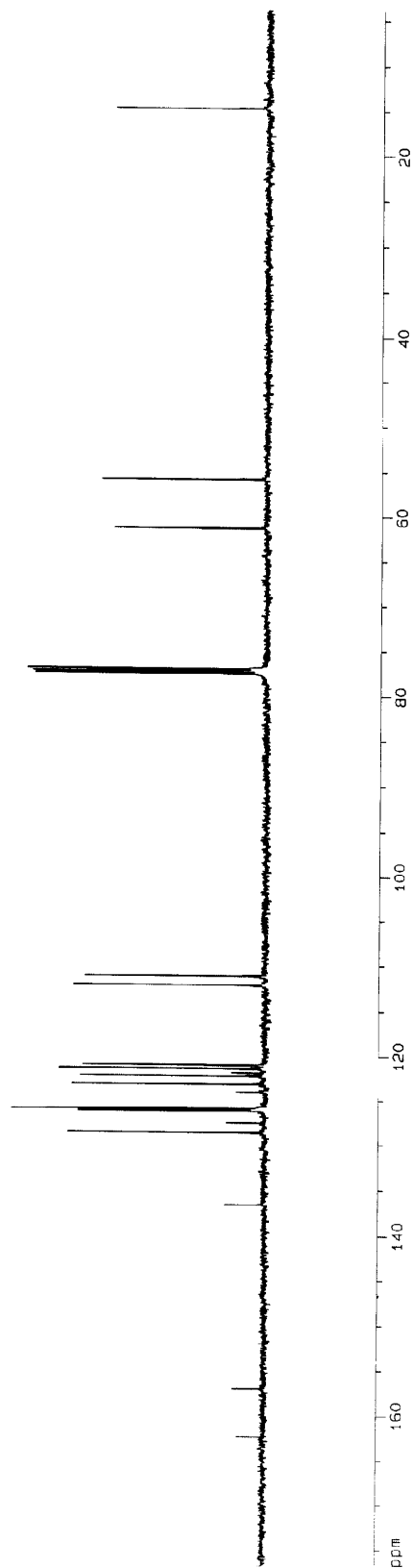
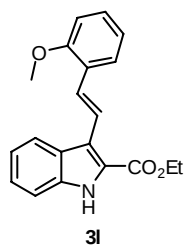
14.725



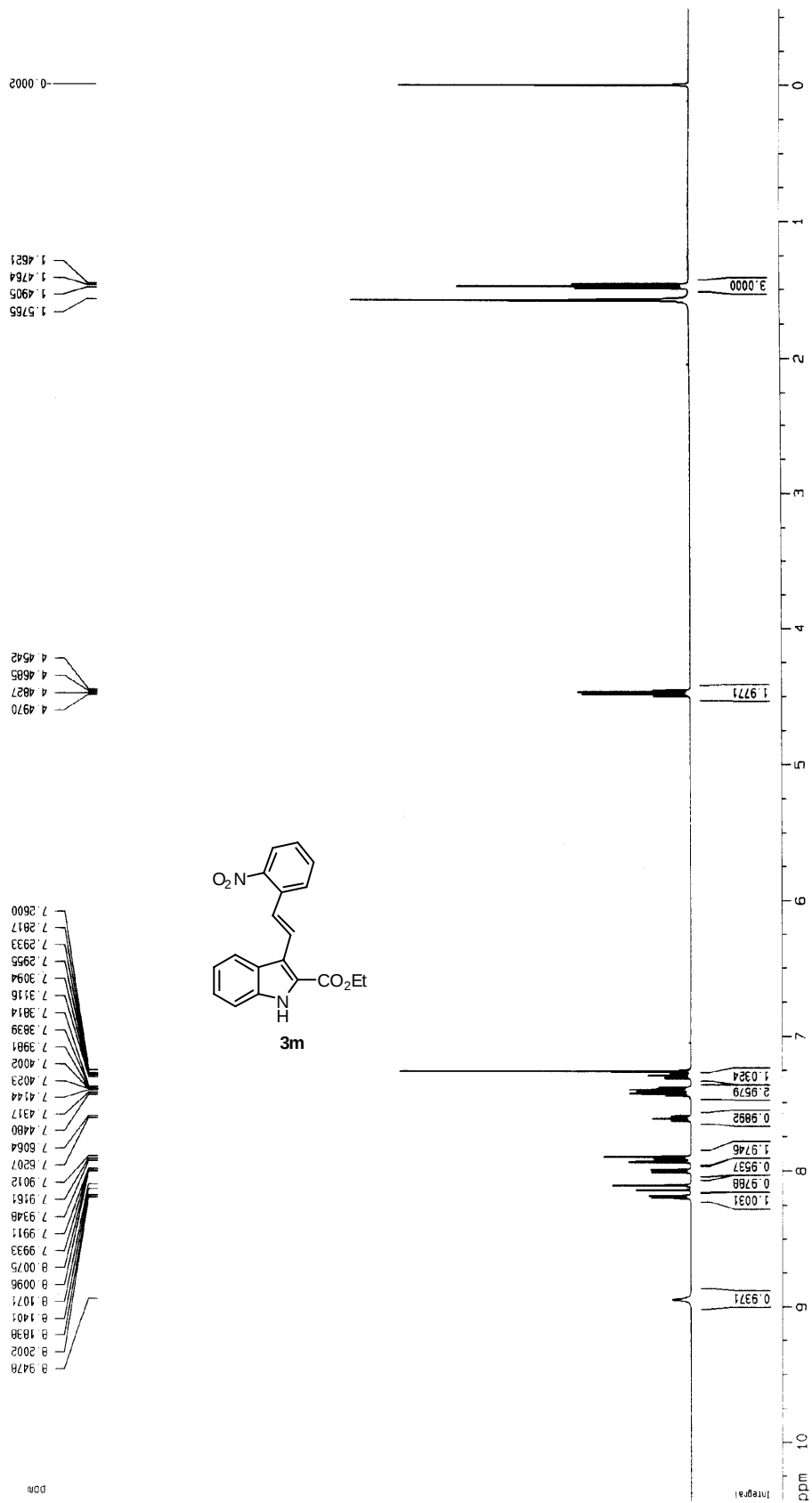


Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-3 in CDCl₃

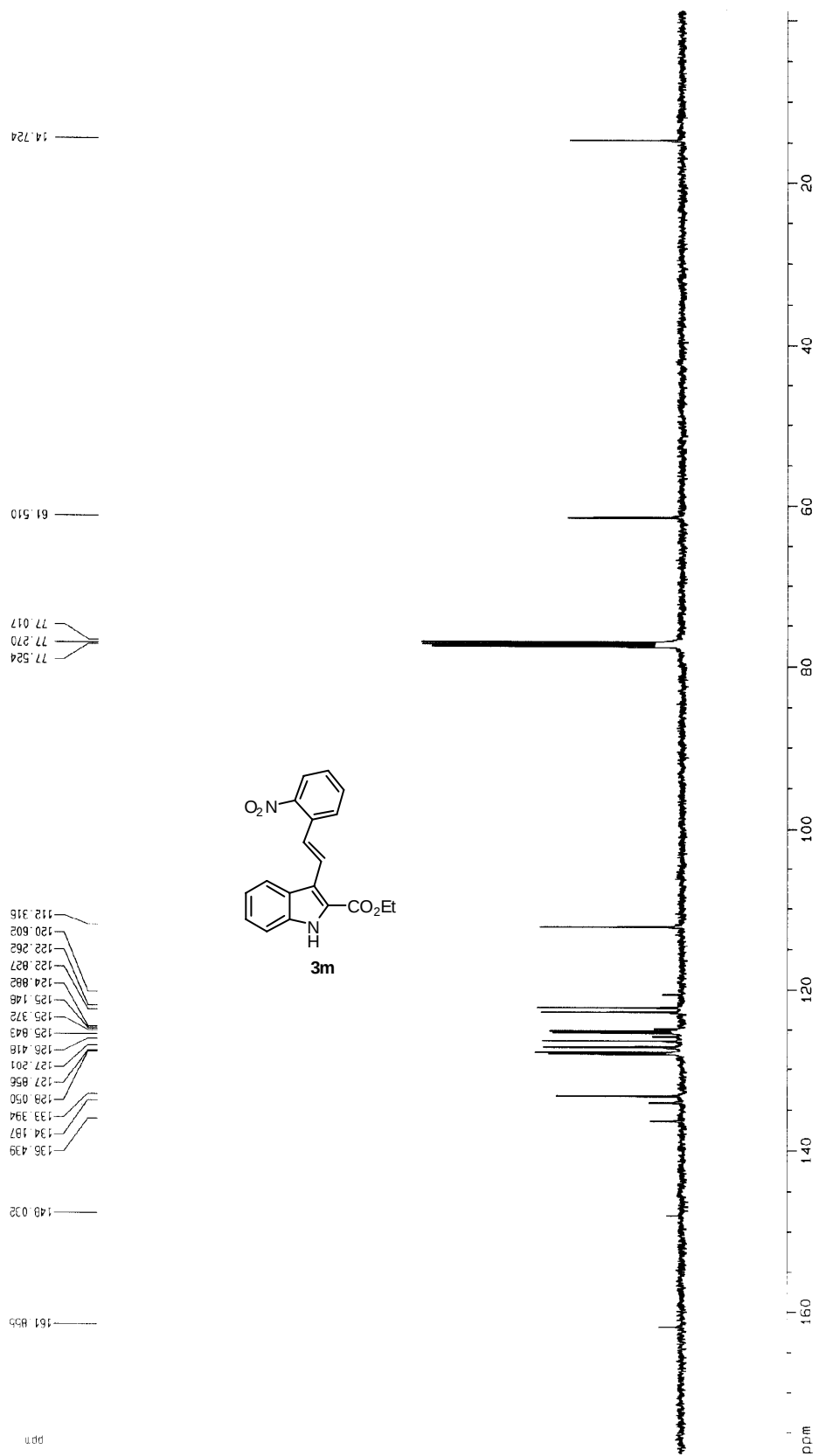
Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZB-n-3 in CDCL3



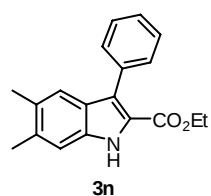
Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-13 in CDCL3



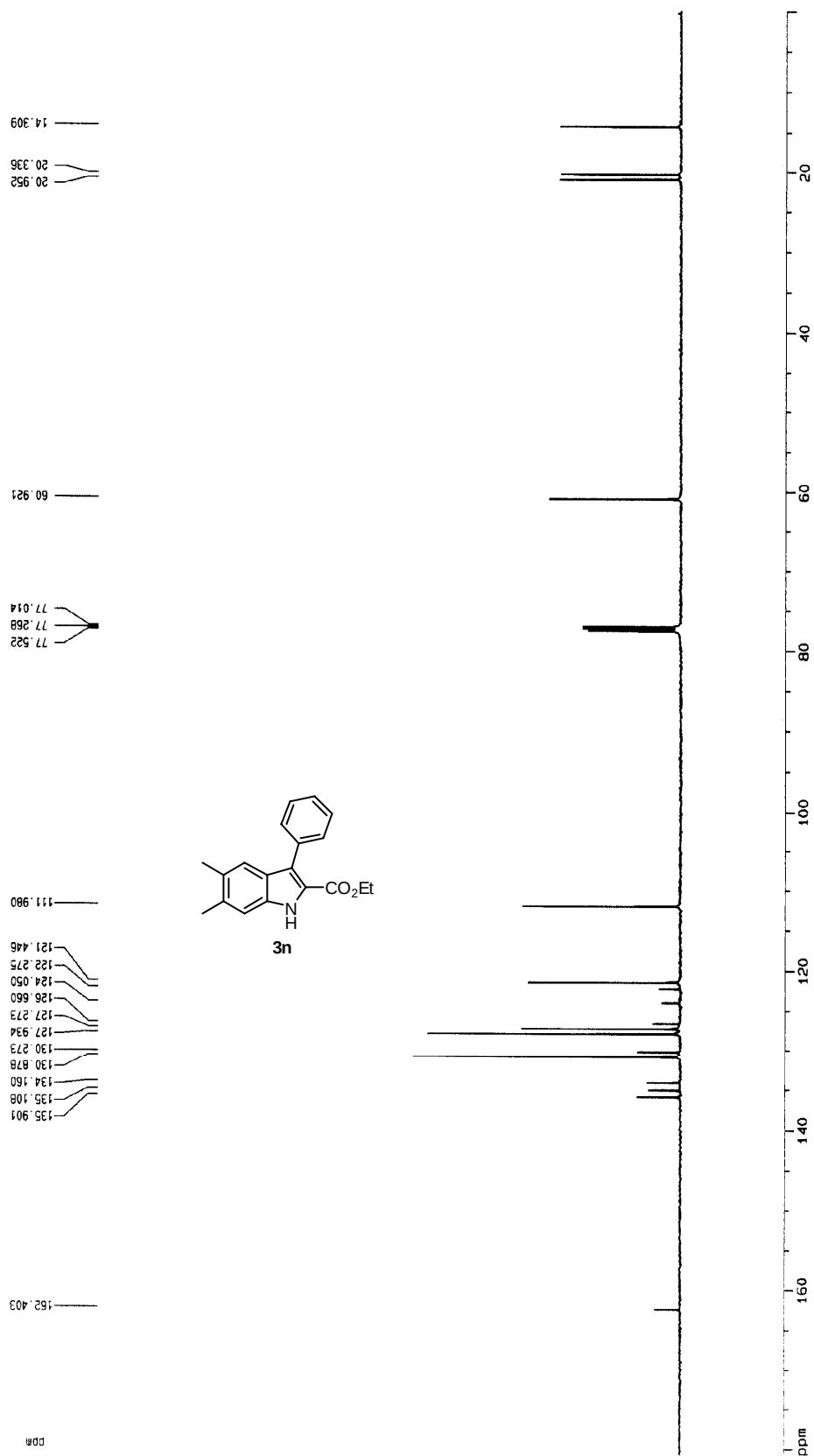
Zhejiang University Avance DMX 500
QNP 5mm Sample CZB-n-13 in CDCL3



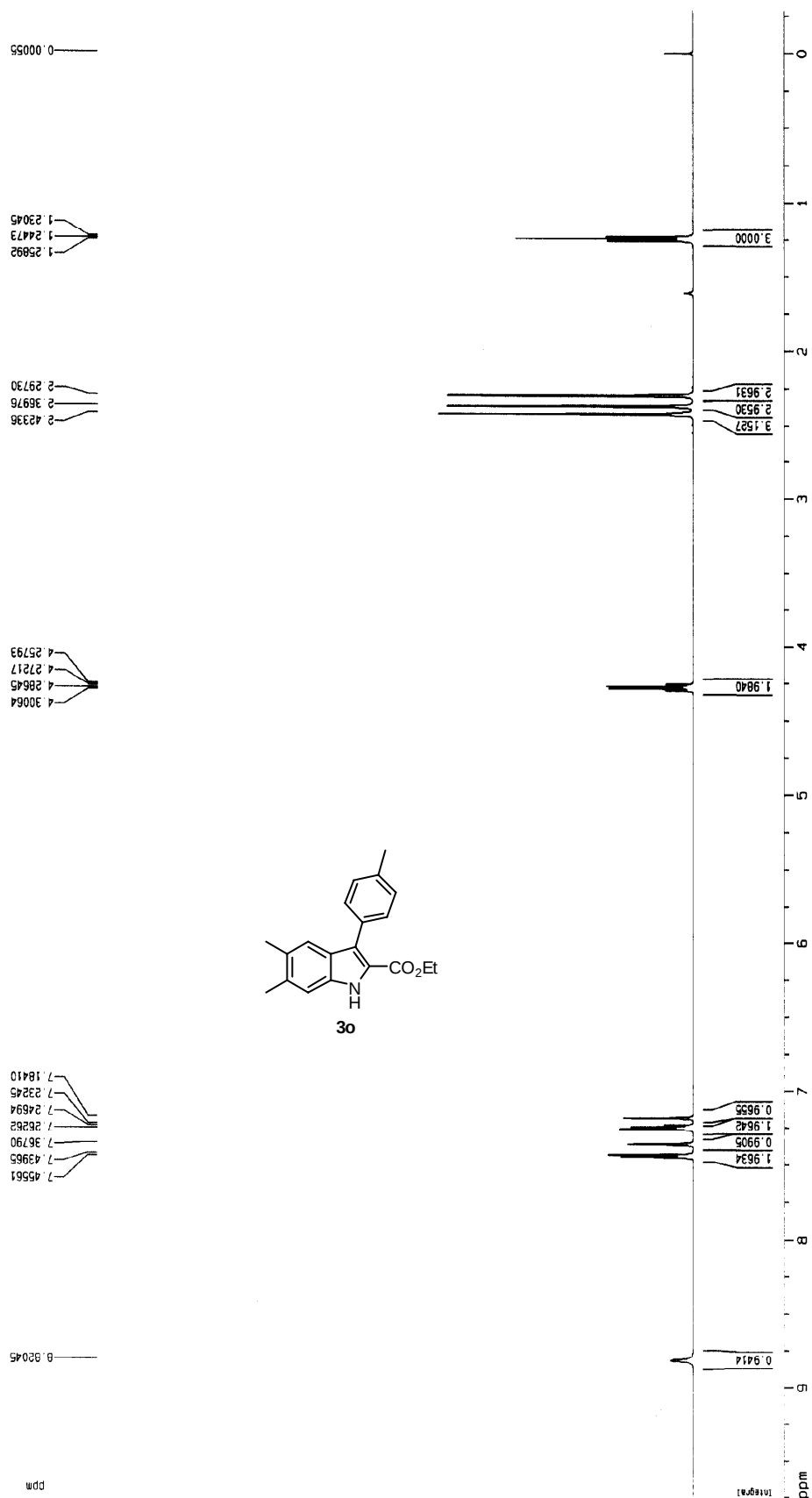
Zhejiang University Avance DMX 500
 QNP 5mm Sample: C2B-n-18 in CDCl3



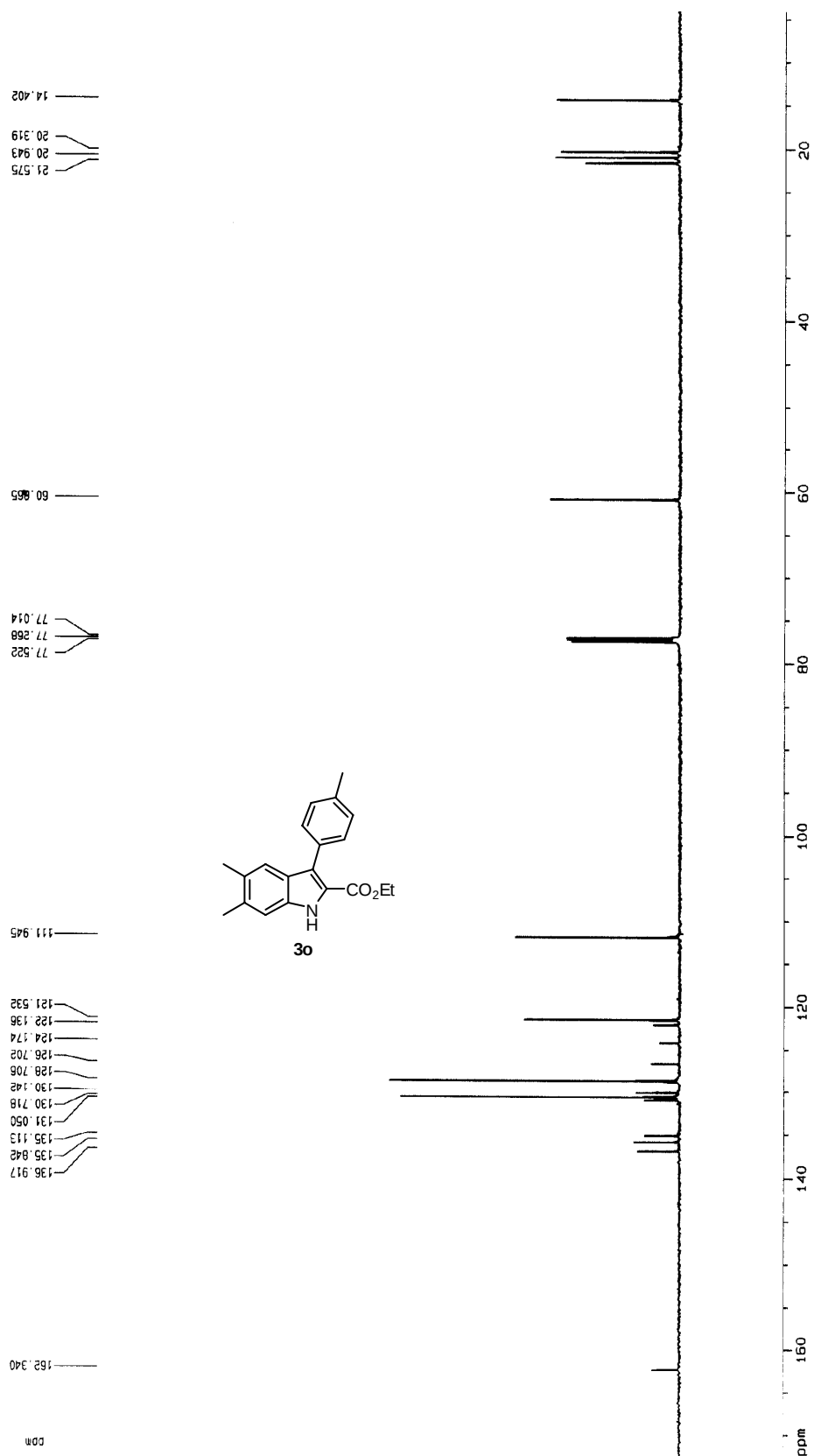
Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZ8-n-18 in CDCL3



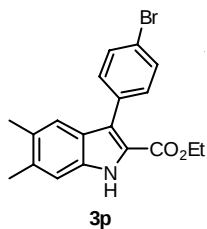
Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZB-n-22 in CDCl₃



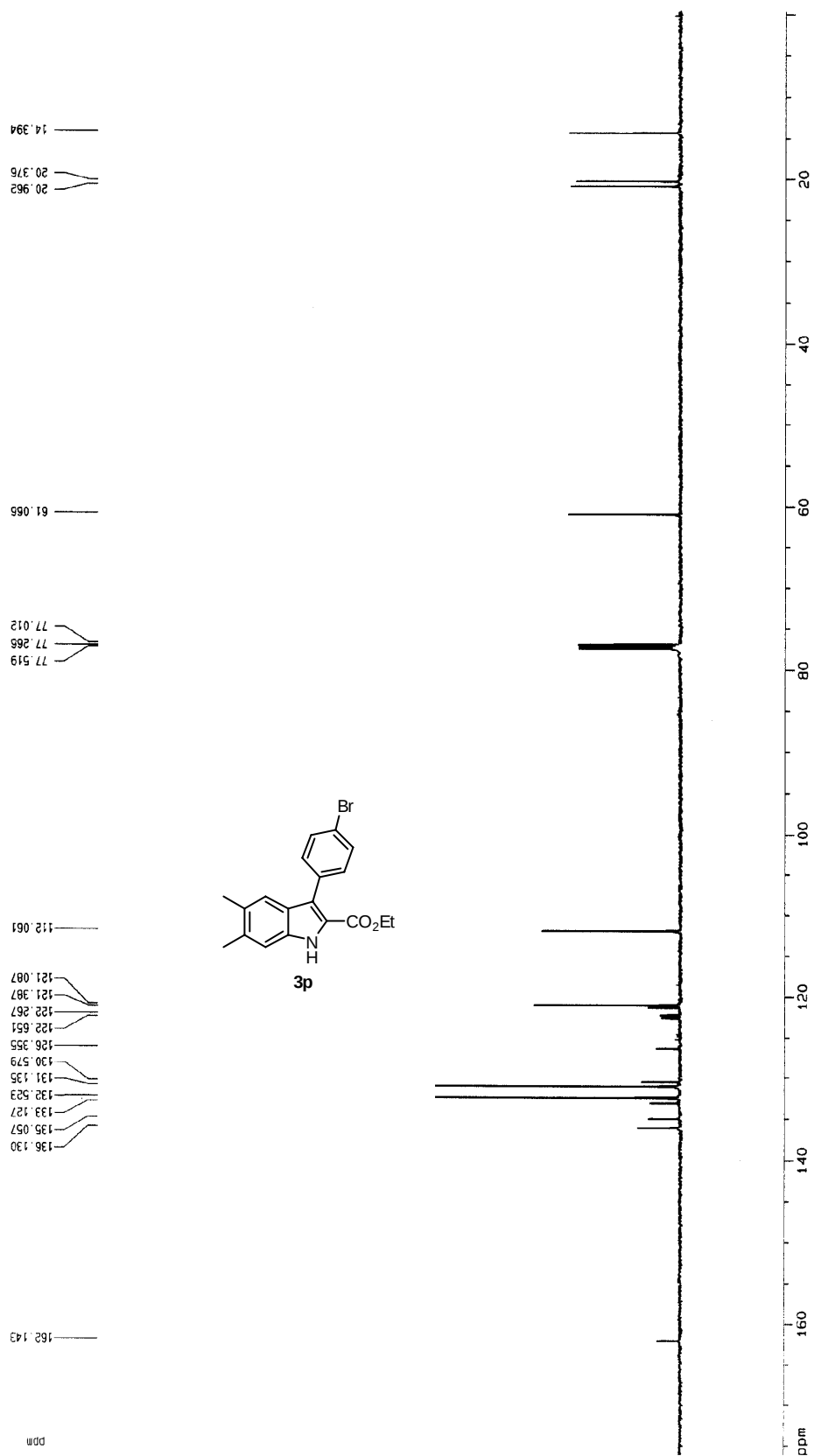
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 QNP 5mm Sample: CZB-n-22 in CDCl₃

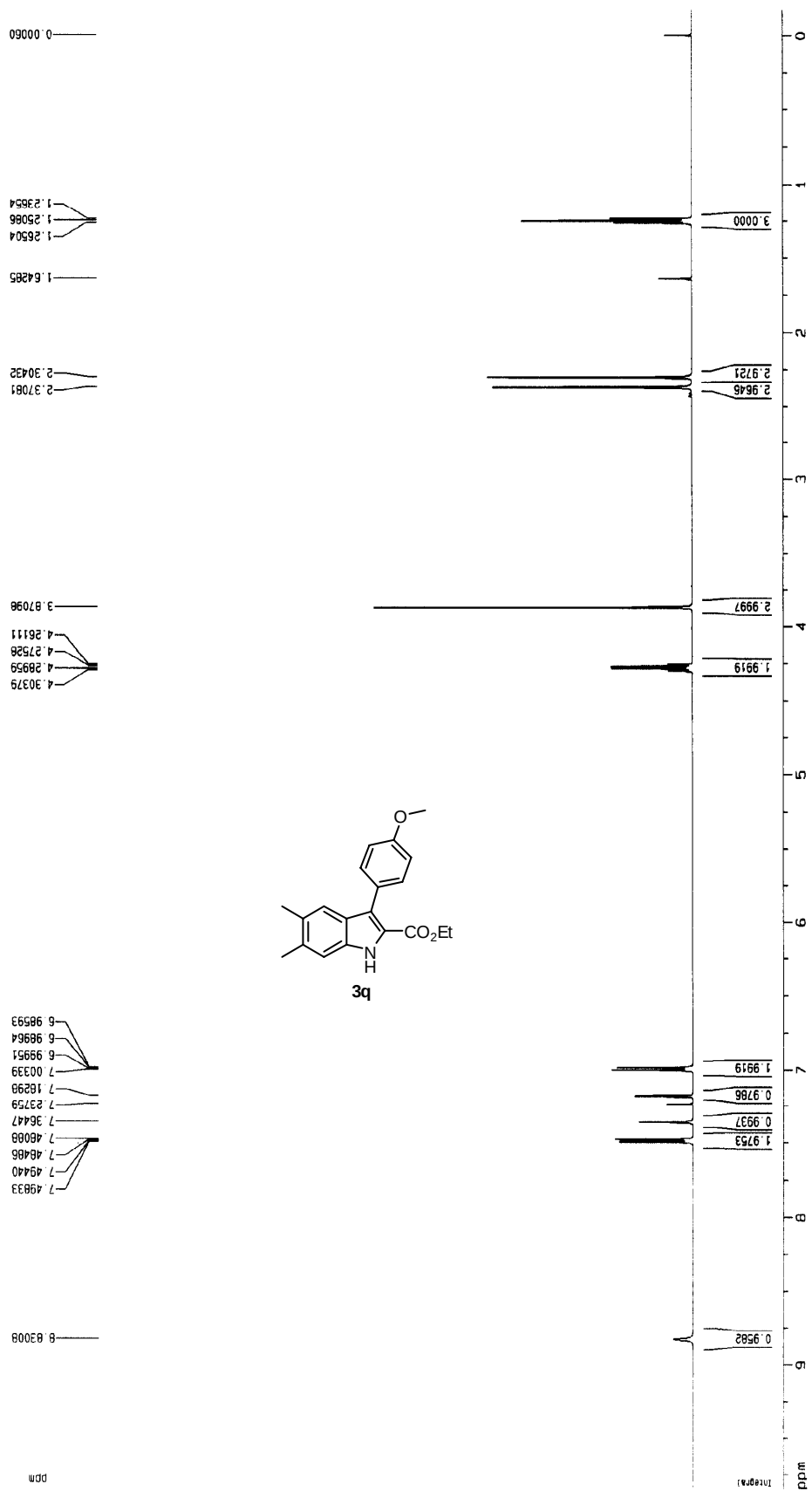
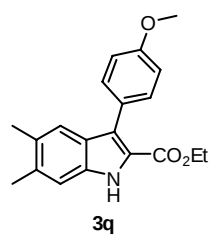


Zhejiang University Avance DMX 500
QNP 5mm Sample: C2B-n-19 in CDCl3



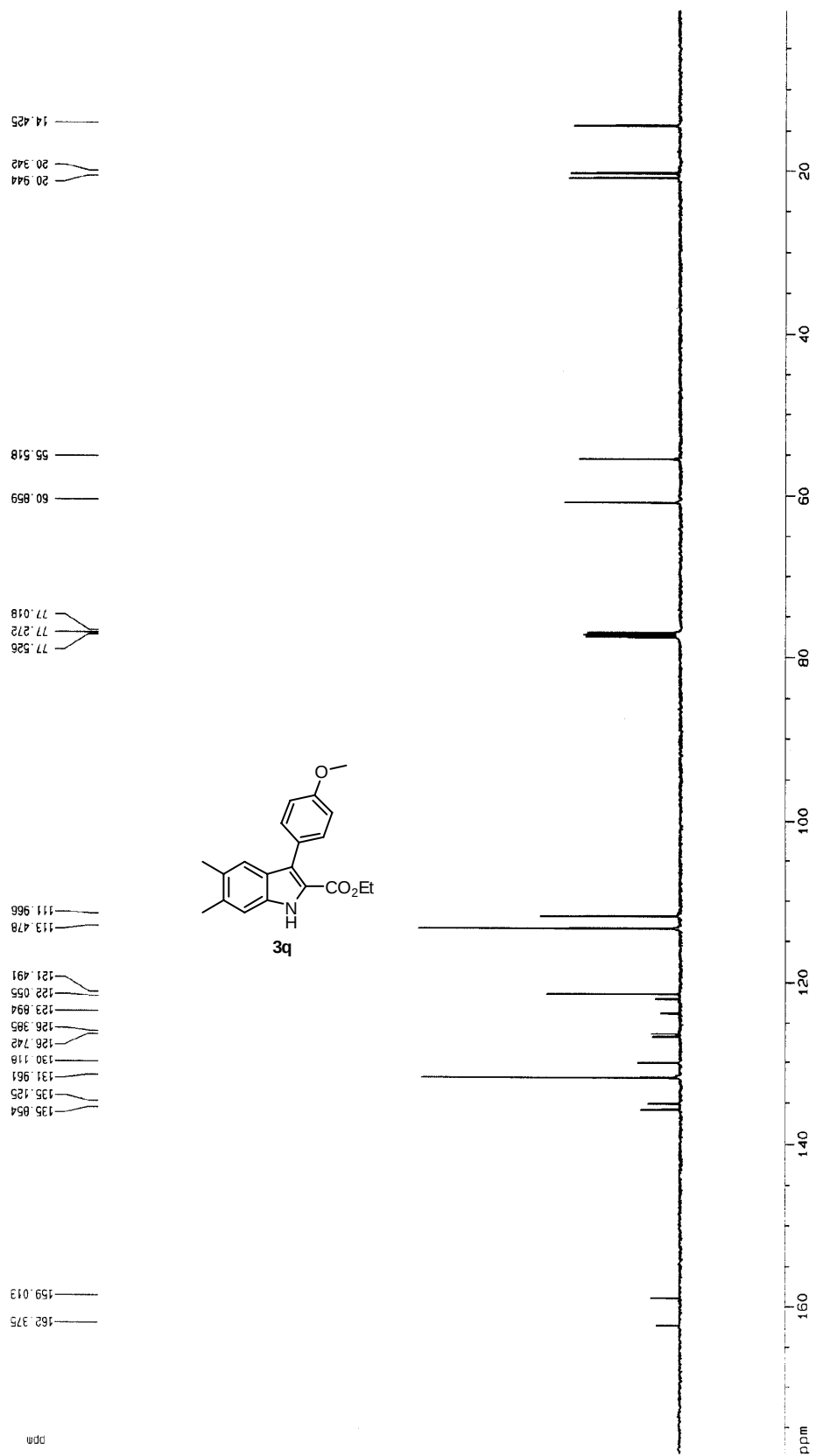
Zhejiang University Avance DMX 500
 GNP 5mm Sample: C7B-n-19 in CDCl₃

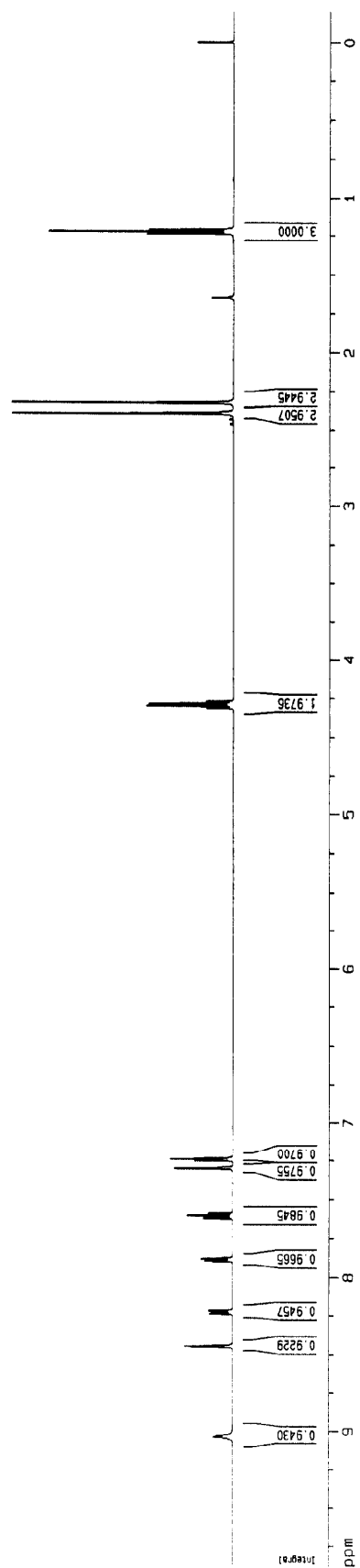
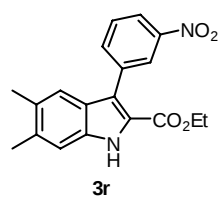




Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-20 in CDCl3

Zhejiang University Avance DMX 500
 GNP 5mm Sample: C2B-n-20 in CDCl3





Zhejiang University Avance DMX 500
QNP 5mm Sample: C7B-n-21 in CDCl₃

Zhejiang University Avance DMX 500
 QNP 5mm Sample: C7B-n-21 in CDCl3

14.252
 20.433
 20.977

61.319

77.013
 77.267
 77.520

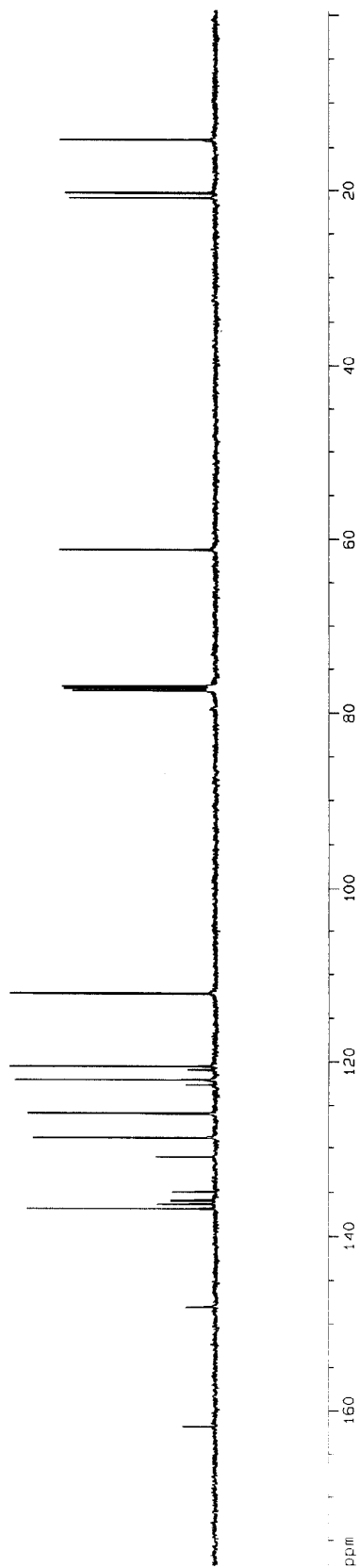
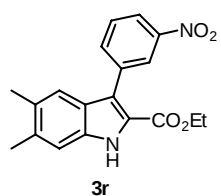
112.259

120.548
 120.971
 122.127
 122.723
 125.995
 126.059
 128.806
 131.131
 135.052
 136.057
 136.456
 136.990

148.208

161.934

0.071



Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZB-n-15 in CDCl₃

1.2202
 1.2059
 1.1917
 1.6302
 -0.0001

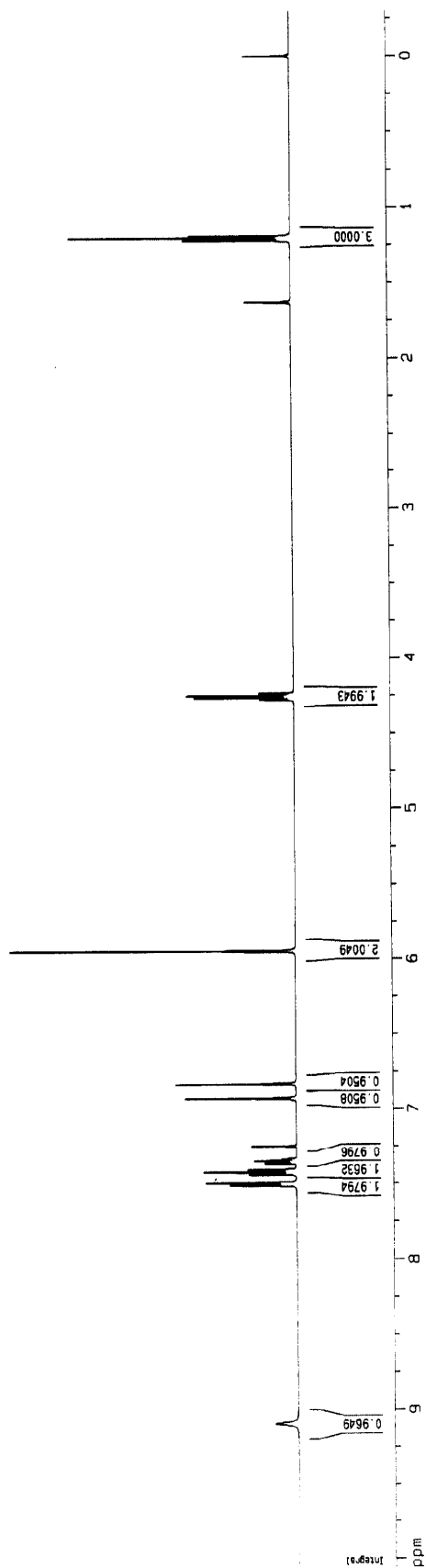
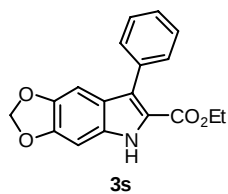
4.2806
 4.2664
 4.2521
 4.2379

5.9480

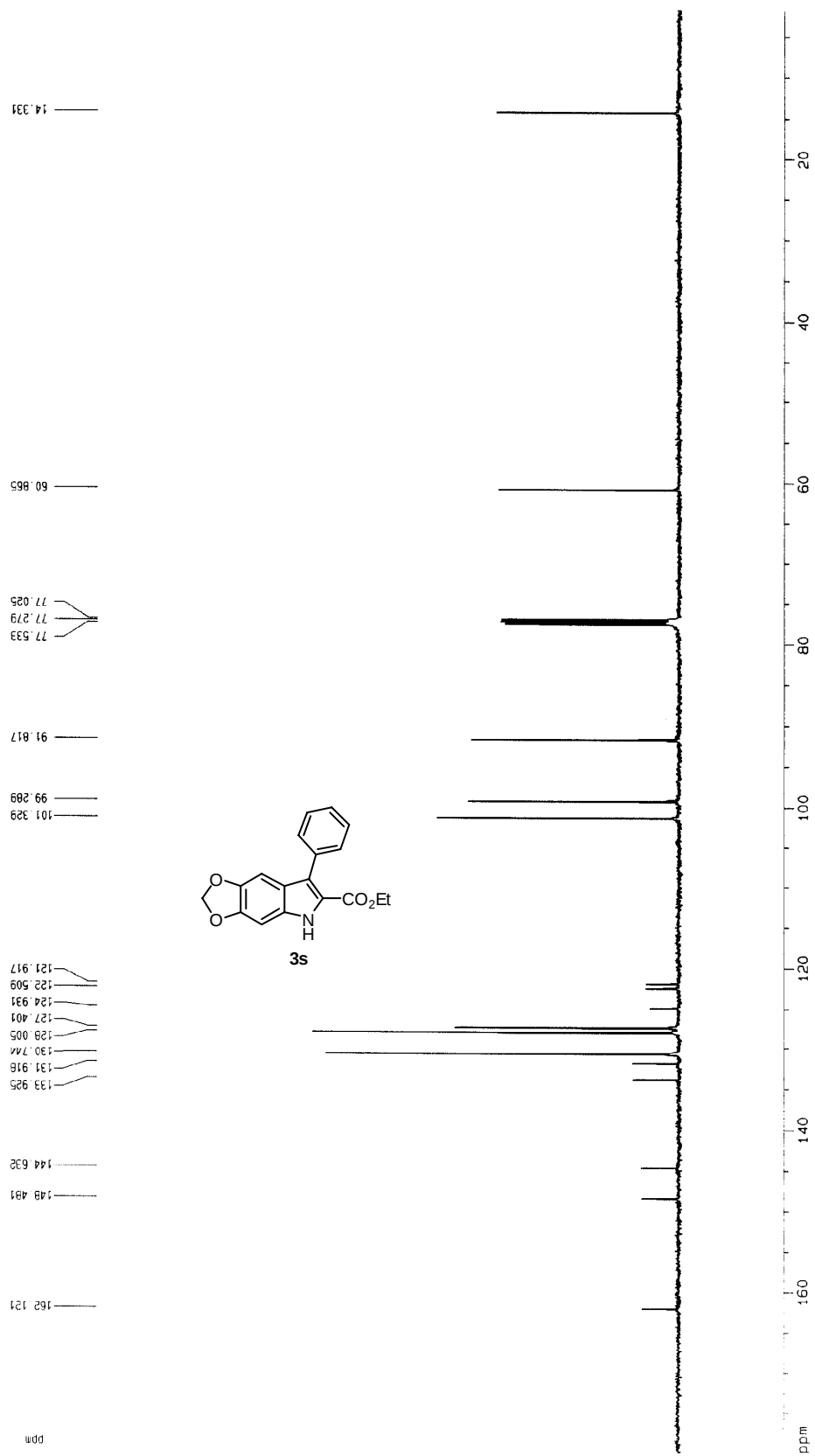
6.8918
 6.9283
 7.2497
 7.3545
 7.3670
 7.3693
 7.4115
 7.4270
 7.4416
 7.4991
 7.5133
 7.5158

9.0936

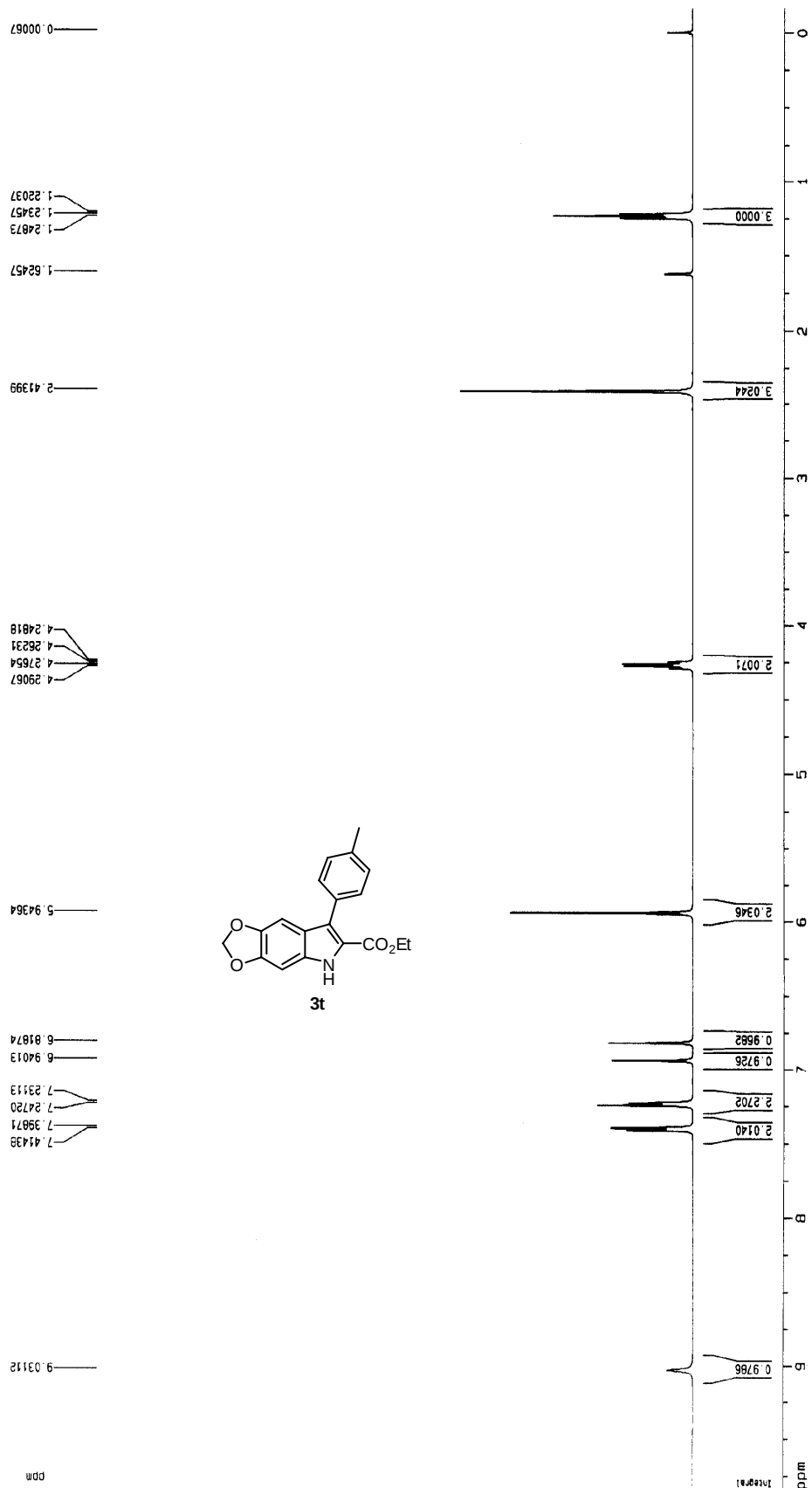
ppm



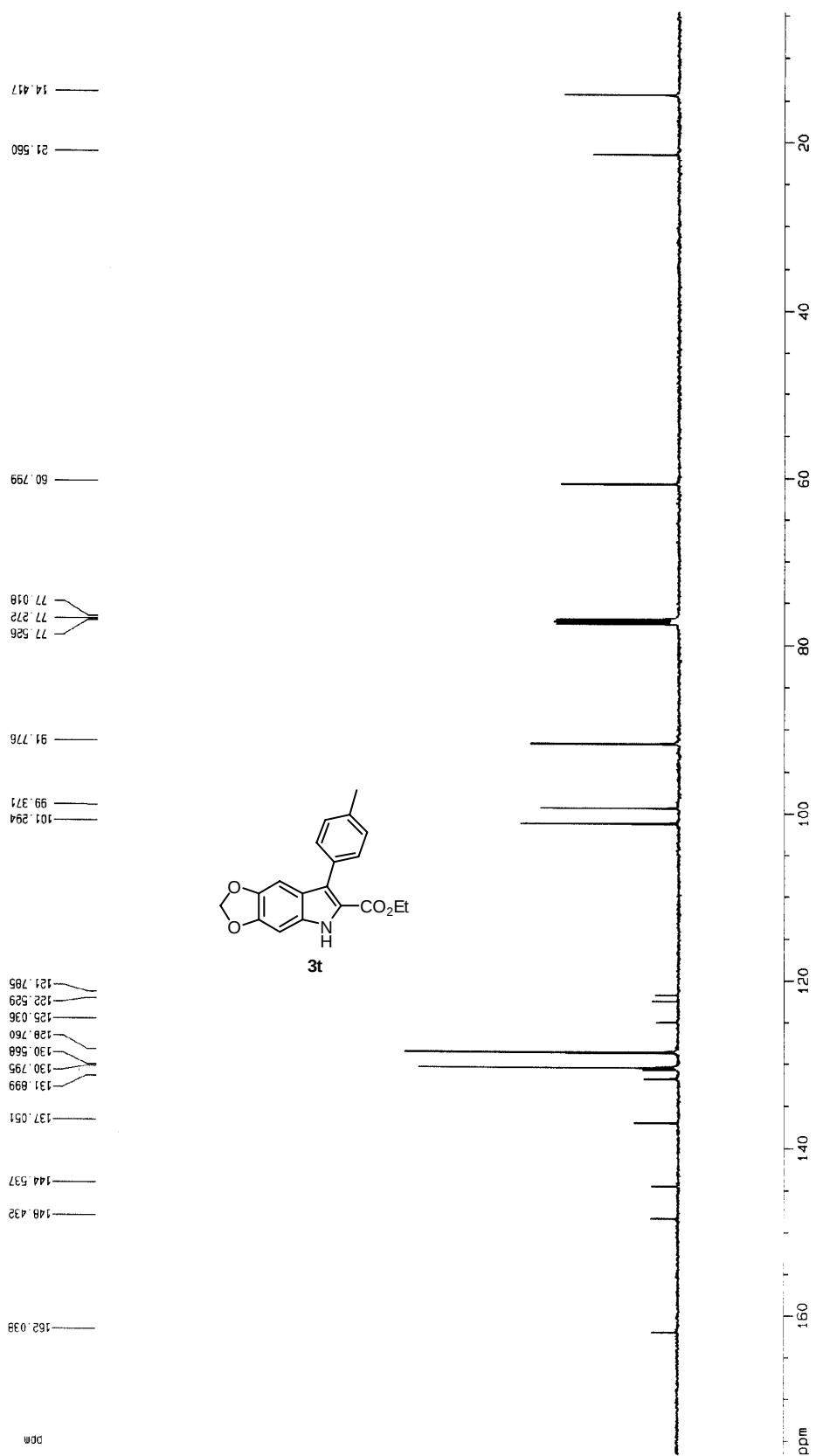
Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZB-n-15 in CDCl₃



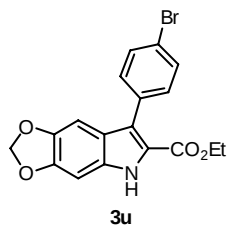
Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-23 in CDCl₃



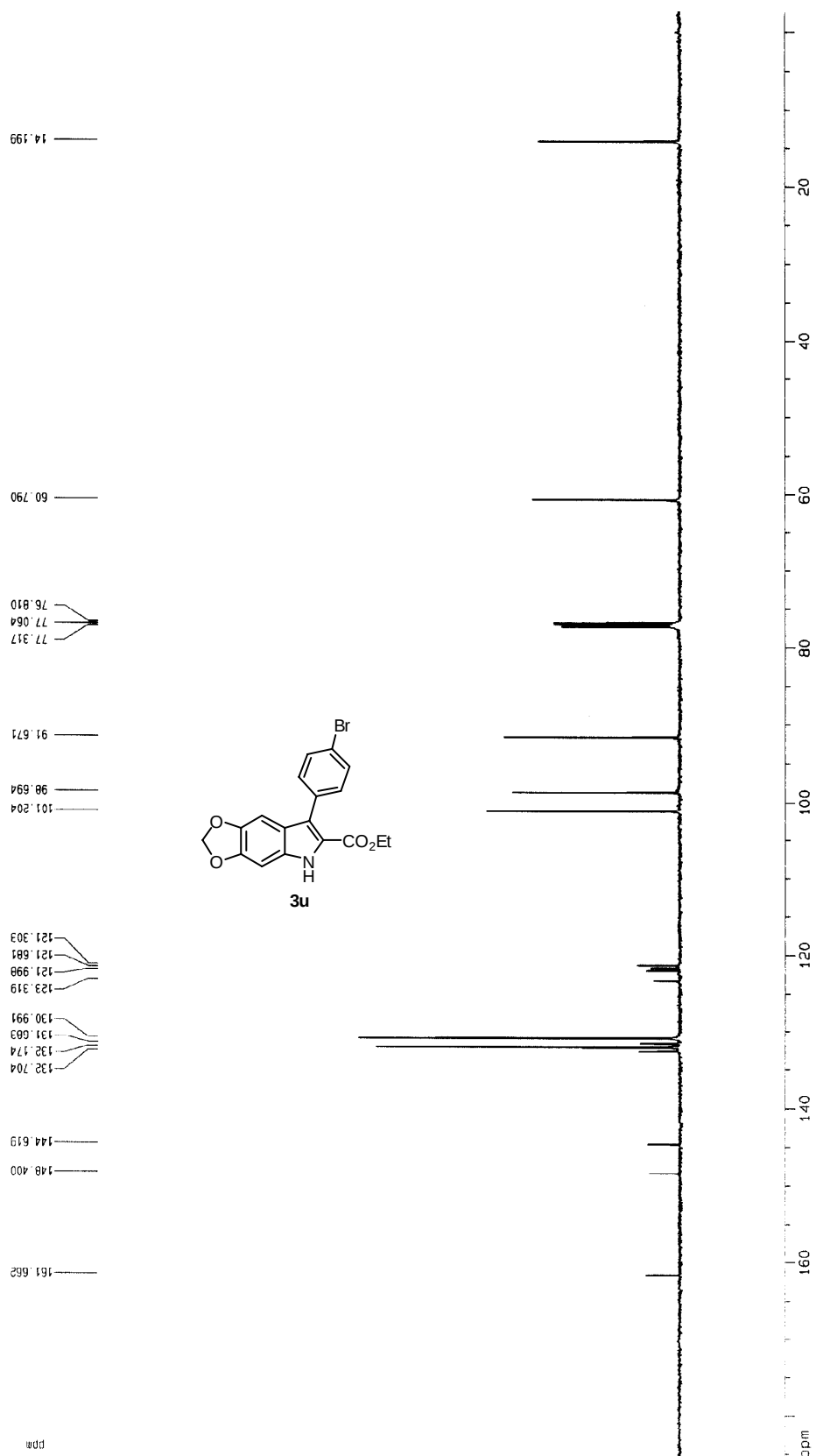
Zhejiang University Avance DMX 500
QNP 5mm Sample: CZB-n-23 in CDCl₃



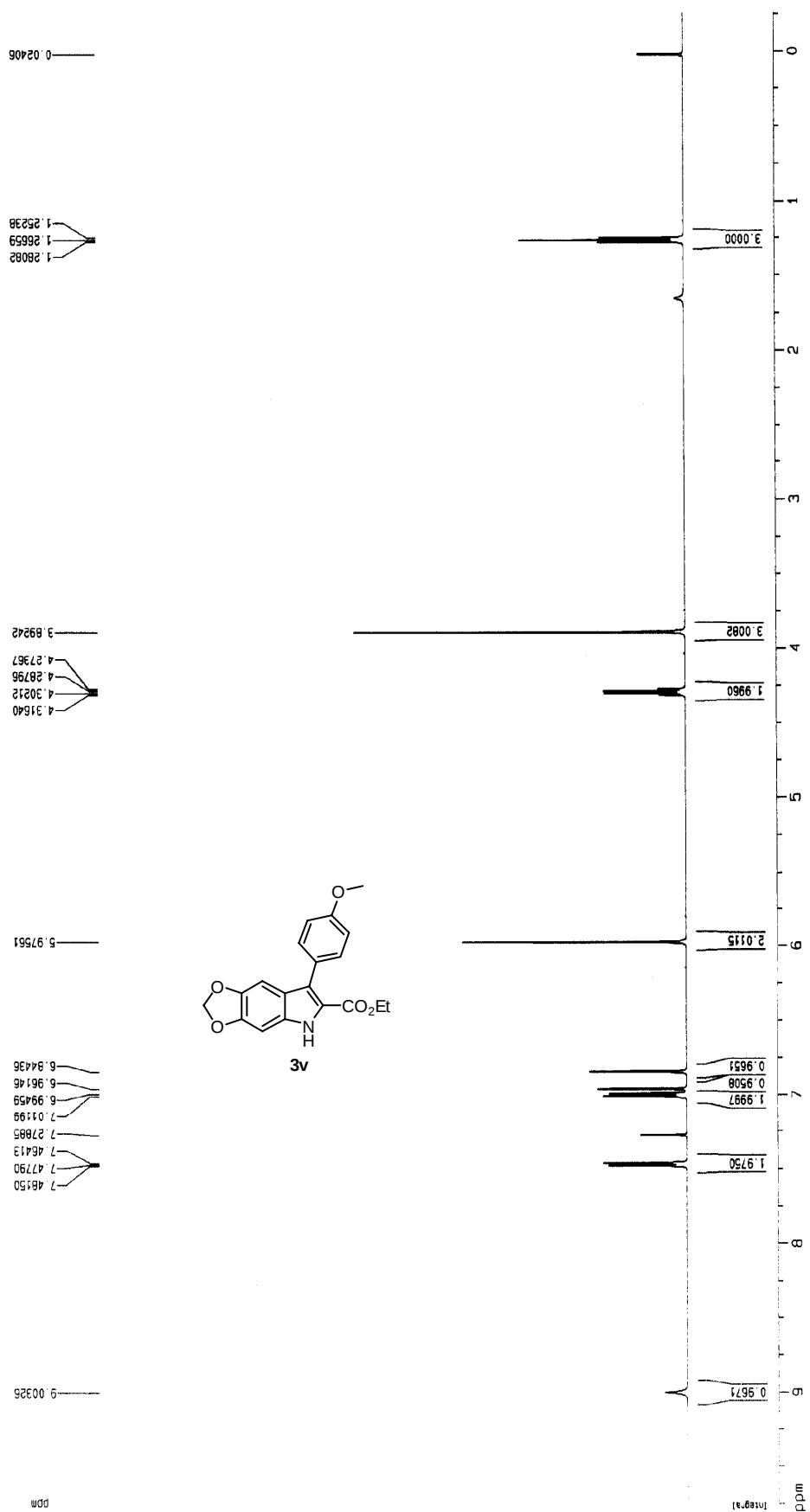
Zhejiang University Avance DMX 500
 5mm Sample: C2B-n-16 in CDCl3



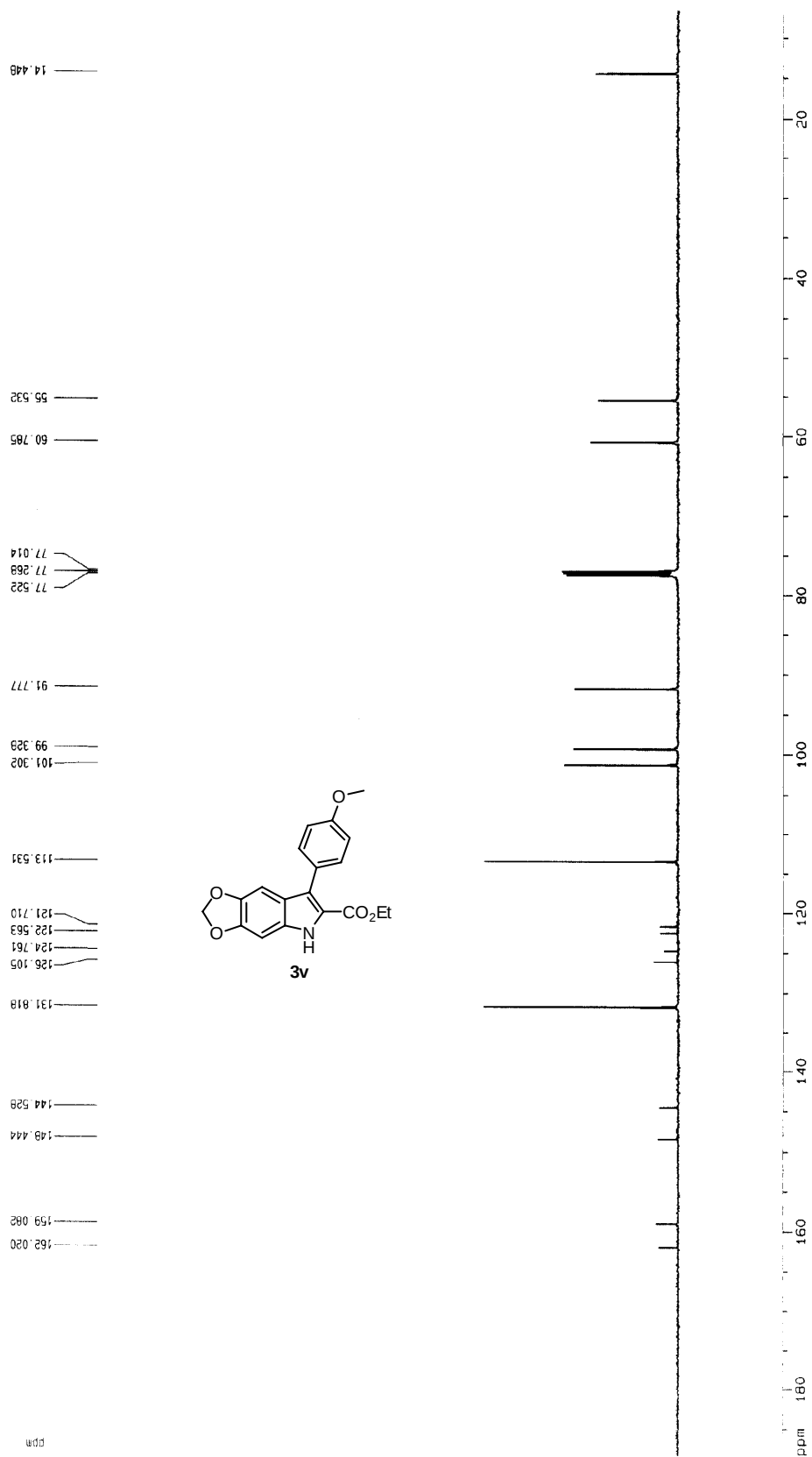
Zhejiang University Avance DMX 500
 5mm Sample: C7B-n-16 in CDCl₃



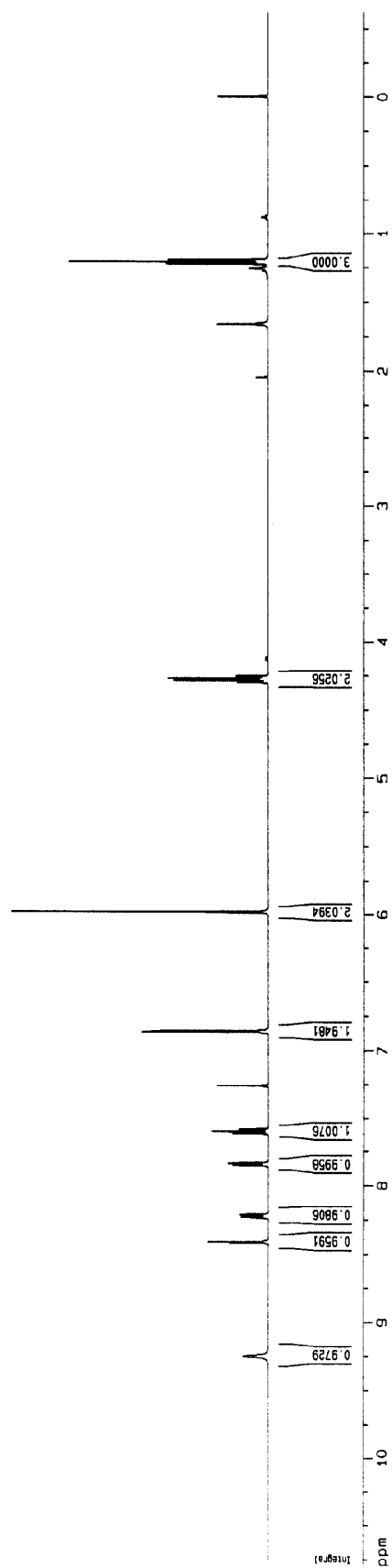
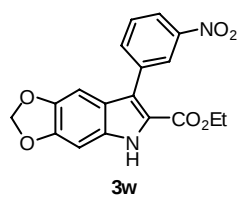
Zhejiang University Avance DMX 500
 QNP 5mm Sample: C2B-n-14 in CDCl₃



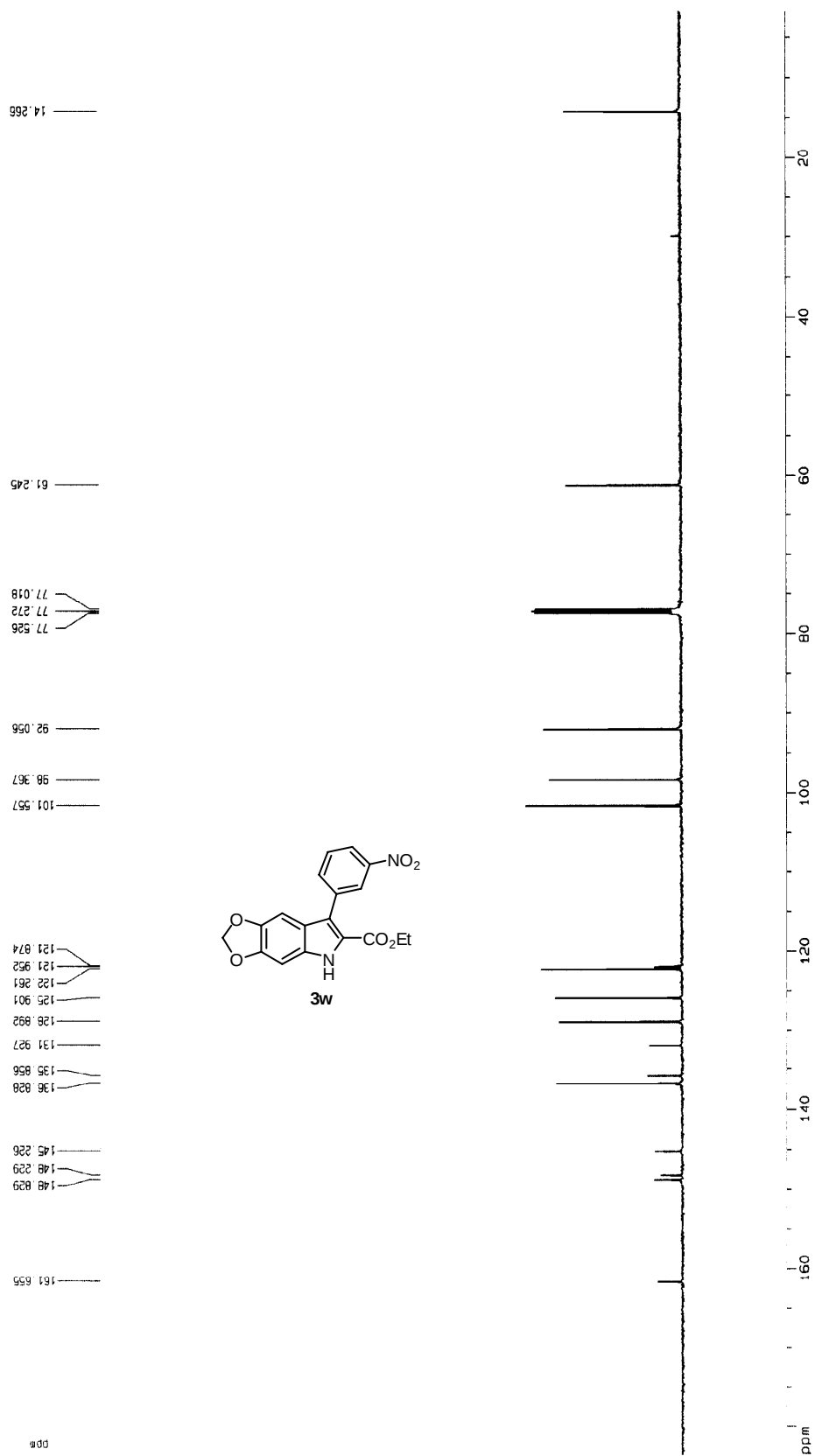
Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZB-n-14 in CDCL3



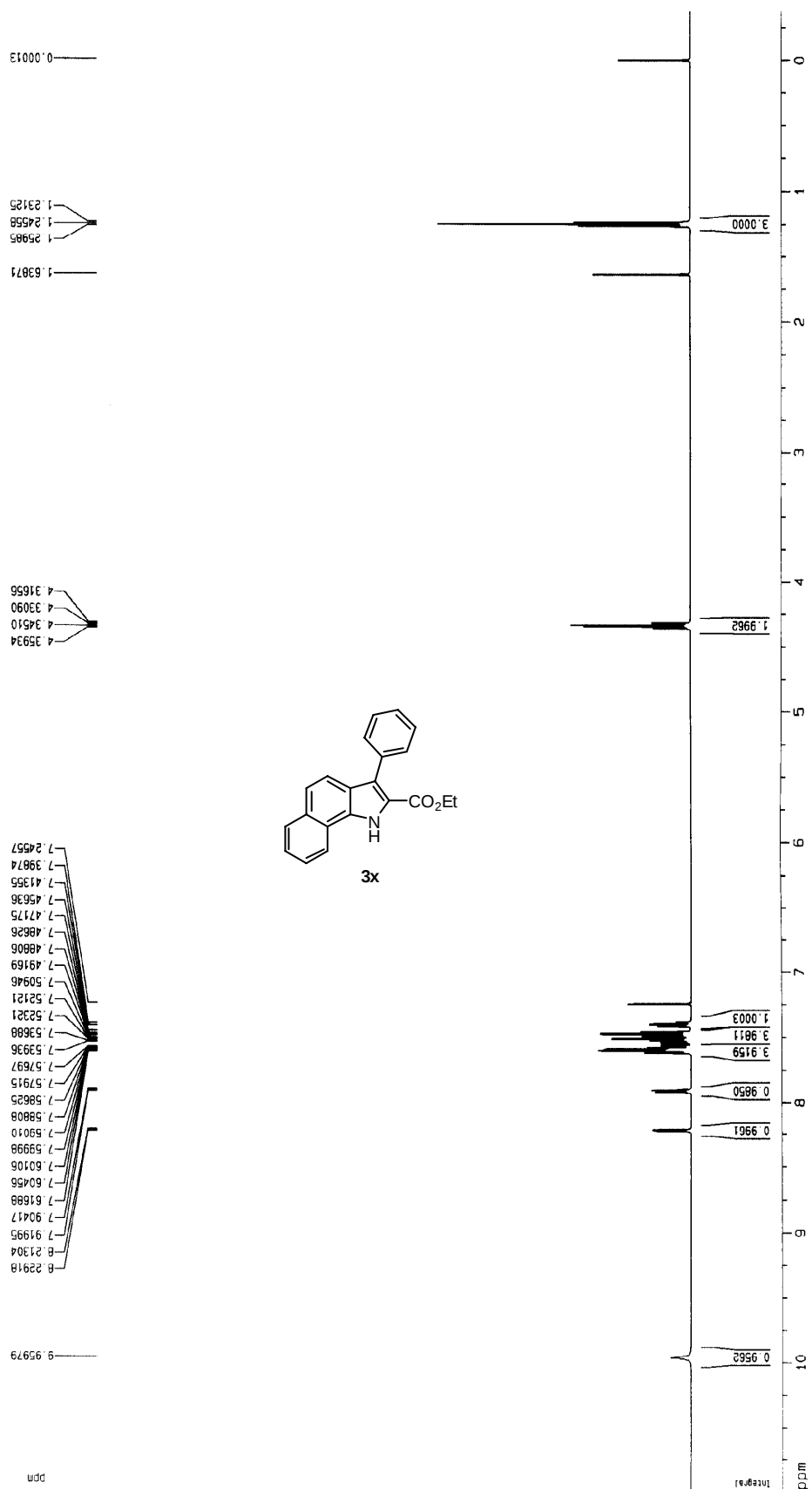
Zhejiang University Avance DMX 500
QNP 5mm Sample: C2B-n-17 in CDCl₃



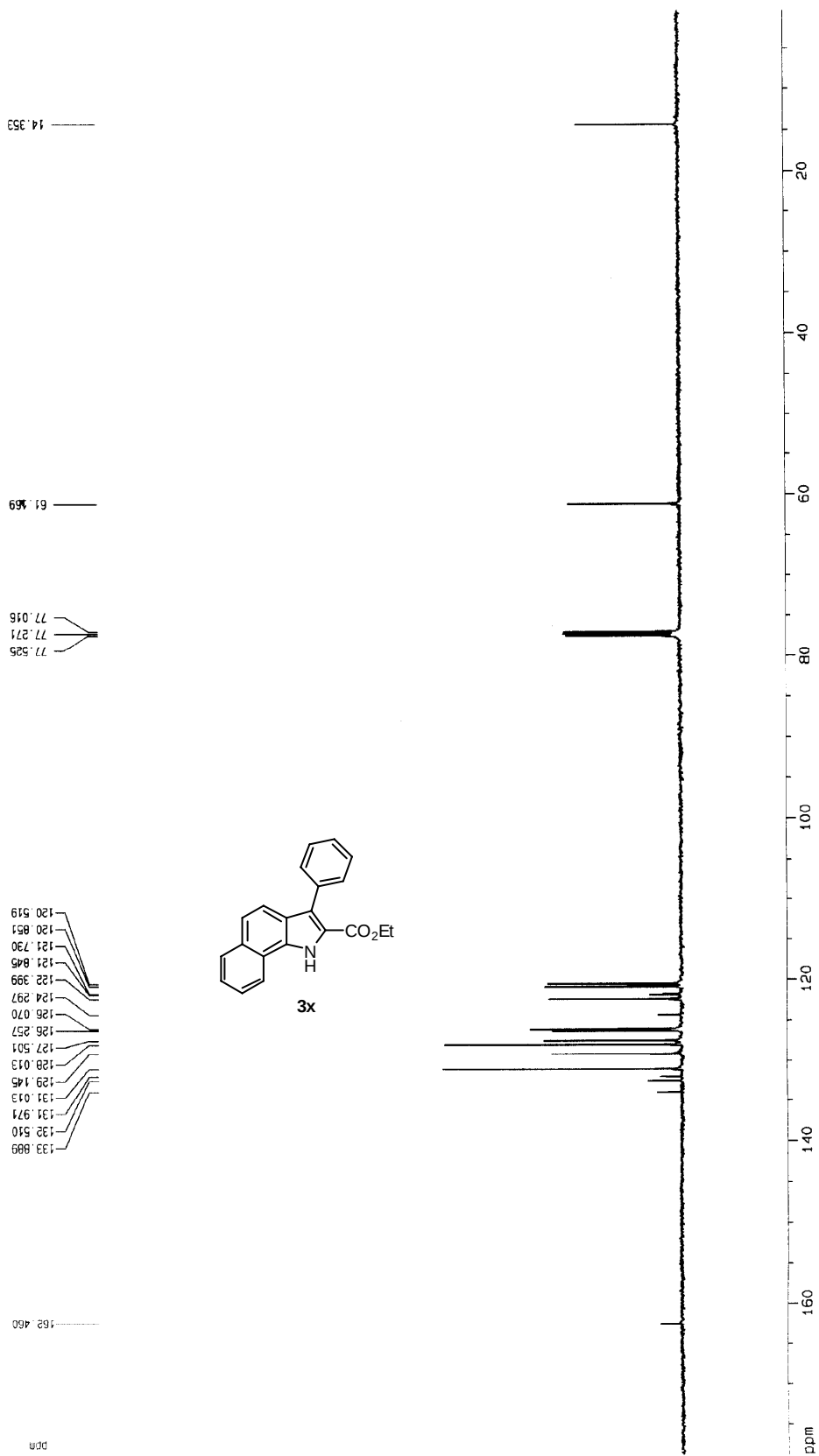
Zhejiang University Avance DMX 500
QNP 5mm Sample: C7B-n-17 in CDCL3



Zhejiang University Avance DMX 500
 QNP 5mm Sample: CZB-n-25 in CDCl₃



Zhejiang University Avance DMX 500
QNP 5mm Sample: C2B-n-25 in CDCL3



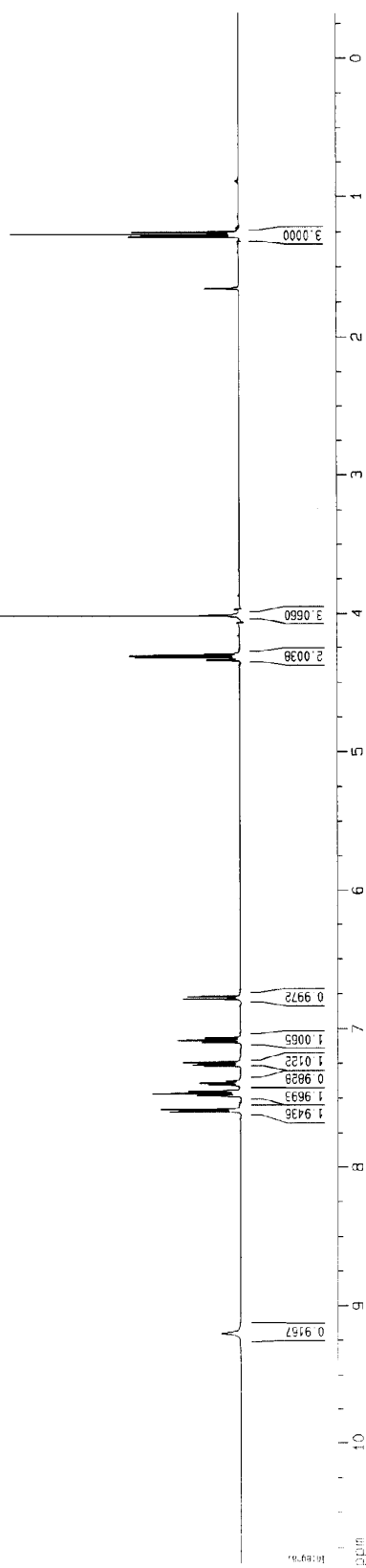
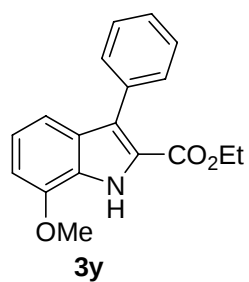
Zhejiang University Avance DMX 500
QNP 5mm Sample: HD-3OME in CDCl3

1.65543
1.28409
1.26979
1.25553

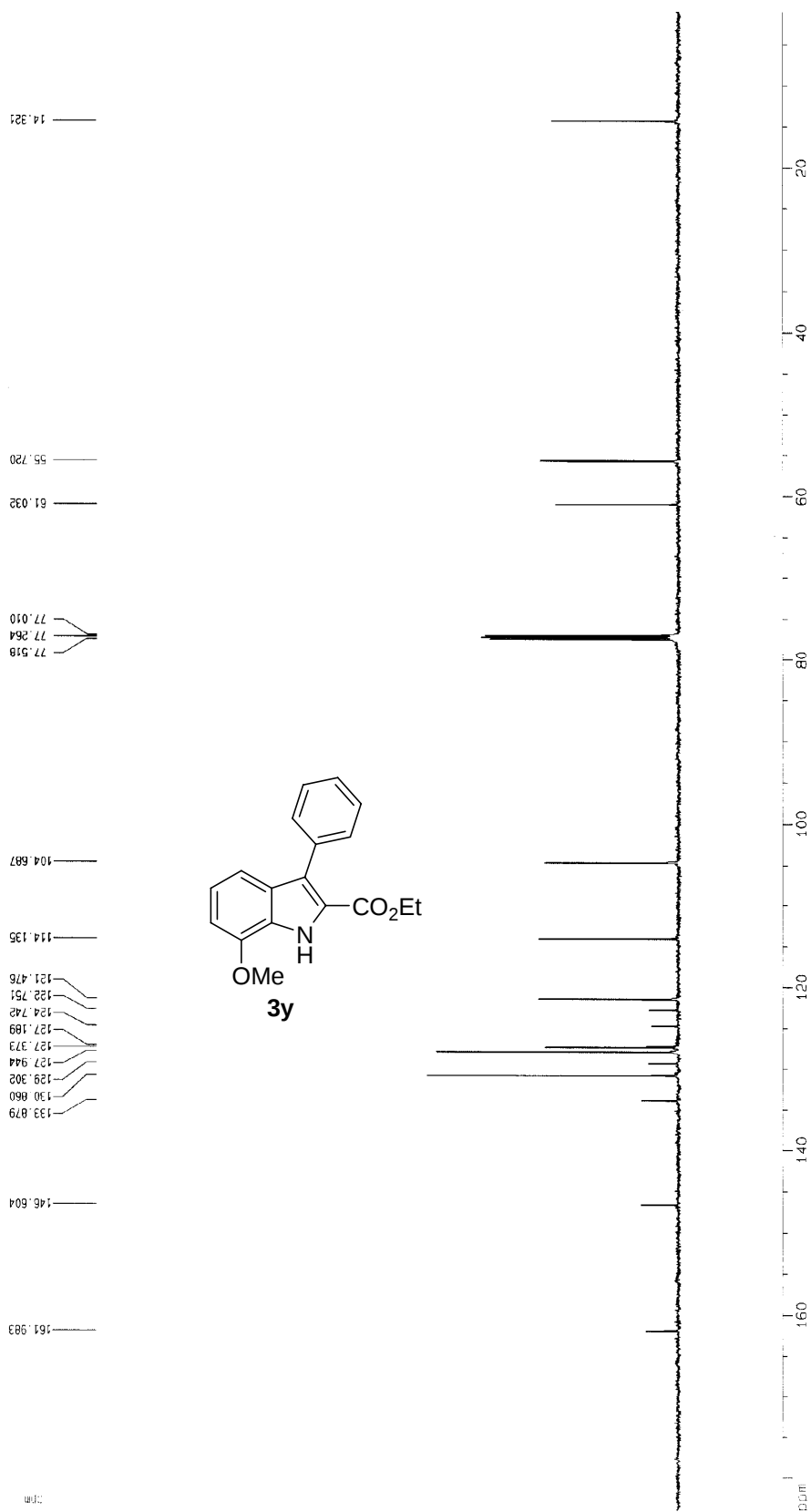
4.34076
4.32558
4.31229
4.29801
4.01770

7.59999
7.59724
7.58273
7.58159
7.48386
7.45921
7.45367
7.40917
7.40708
7.39430
7.27085
7.26085
7.24451
7.09929
7.08346
7.05758
6.78715
6.77196

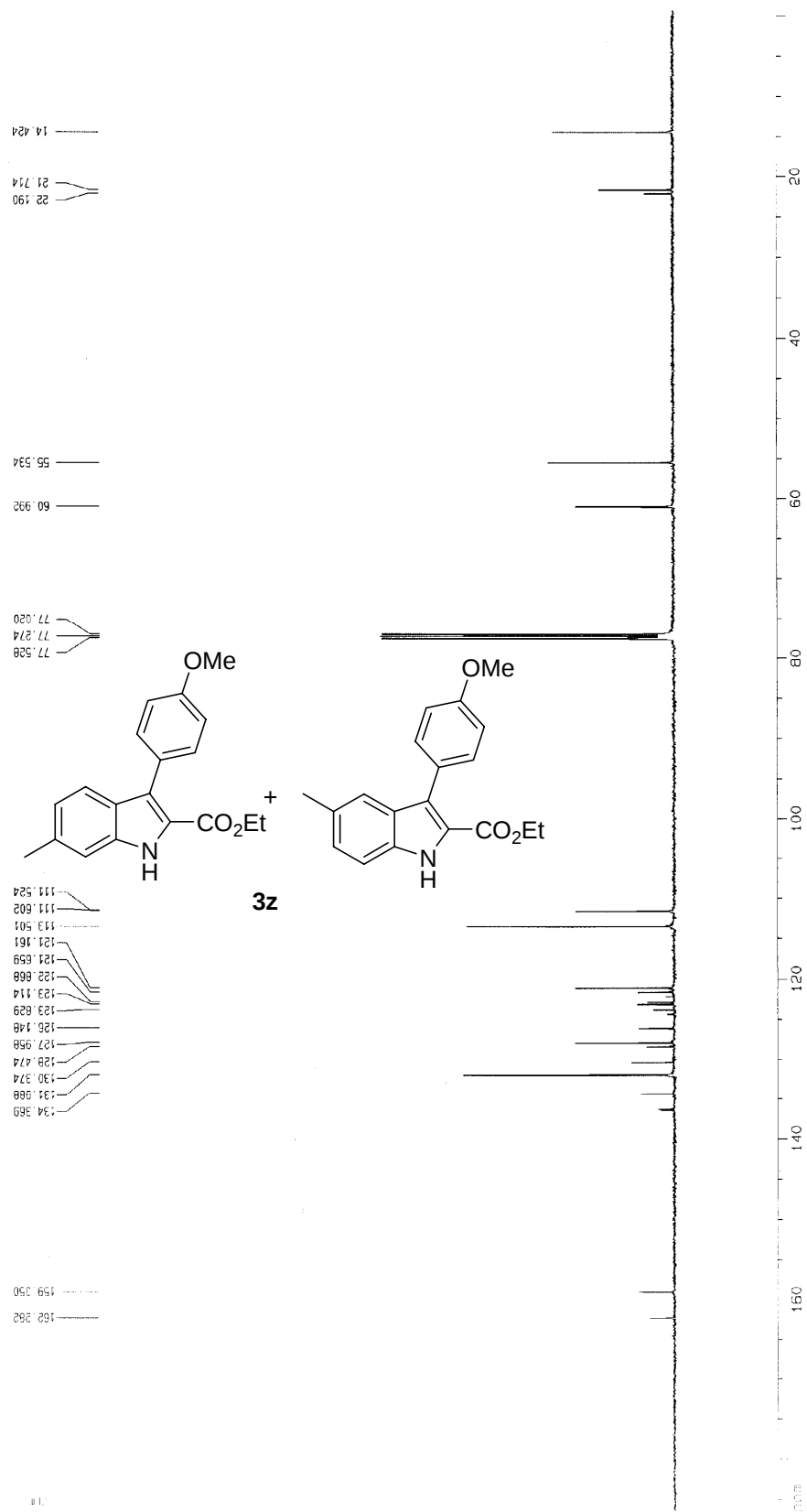
9.20046



Zhejiang University Avance DMX 500
 QNP 5mm Sample: HD-30Me in CDCL3



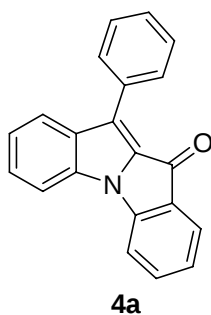
Zhejiang University Avance DMX 500
 QNP 5mm Sample: HD-0-0115 in CDCl₃



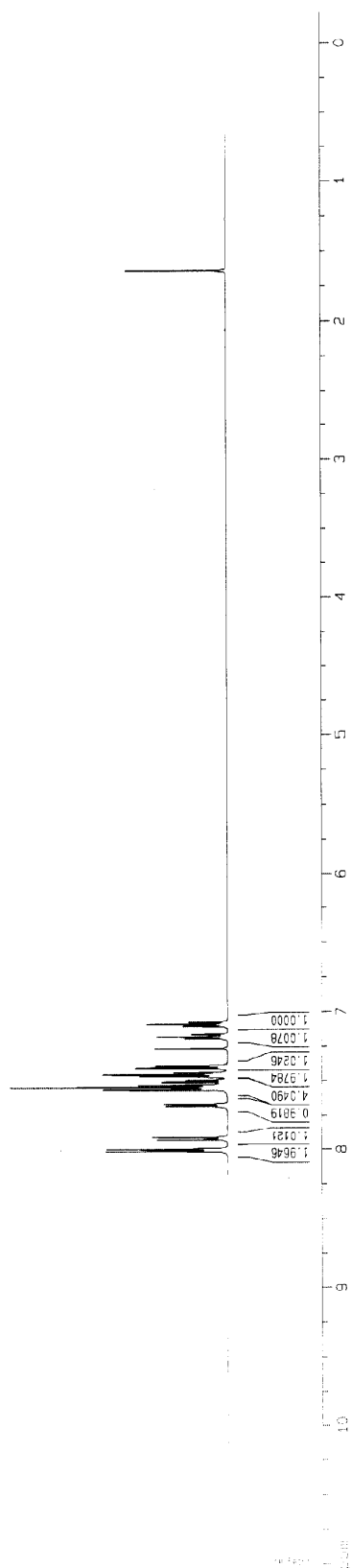
Zhejiang University Avance DMX 500
 GNP 5mm Smaple: HD-0-0109 in CDCl3

7.07838
 7.09295
 7.09378
 7.10812
 7.18151
 7.19608
 7.26945
 7.39450
 7.41021
 7.44134
 7.44310
 7.45780
 7.47333
 7.51393
 7.51585
 7.53164
 7.53675
 7.55202
 7.56768
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 8.01624
 8.01862

1.64292



4a



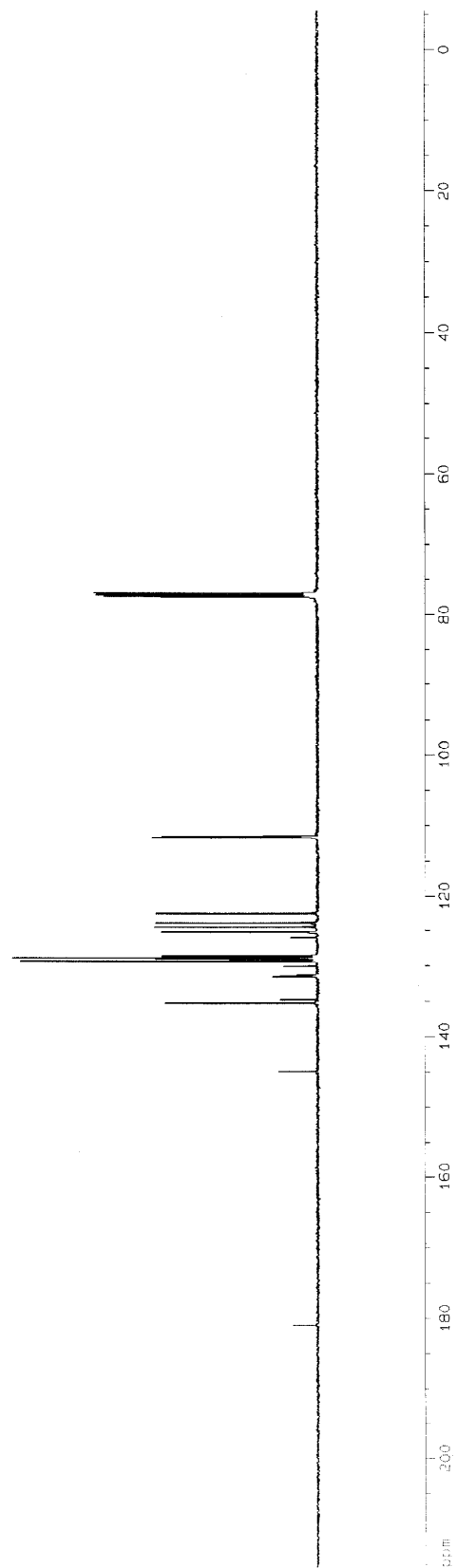
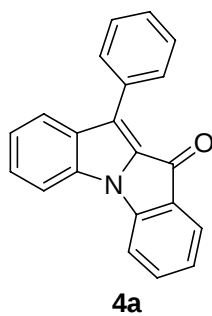
Zhejiang University Avance DMX 500
QNP 5mm Sample: HD-0-0109 in CDCl₃

77.530
77.276
77.022

144.945
135.318
134.816
131.560
131.452
131.359
130.098
129.386
129.050
128.905
128.621
125.934
125.273
124.545
123.952
122.438
111.727
111.595

181.042

ppm



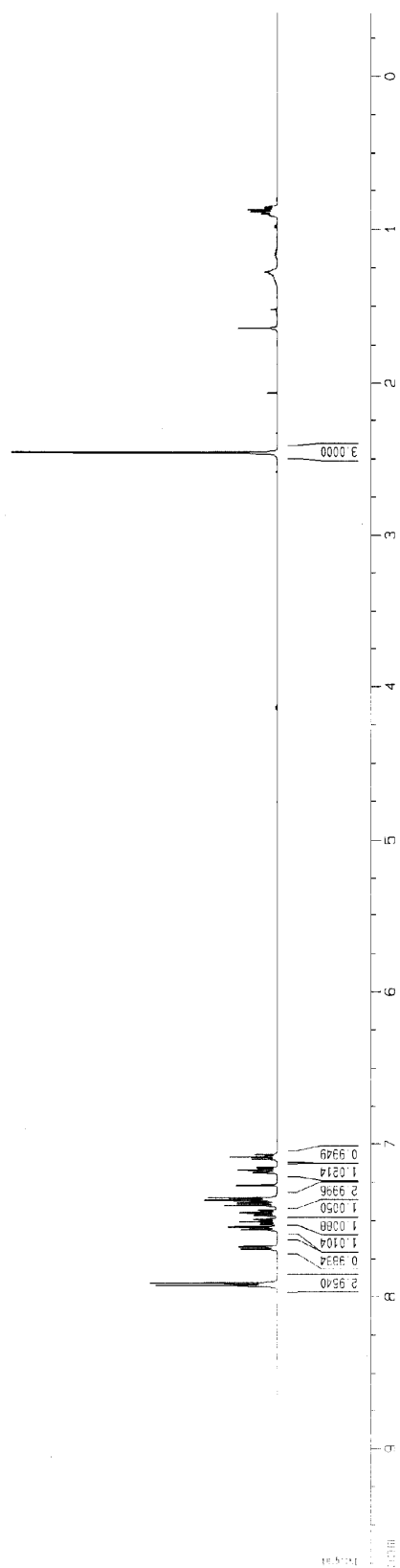
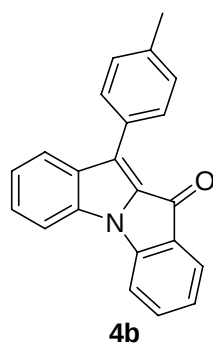
Zhejiang University Avance DMX 500
 GNP 5mm Smaple: HD-D-1215 in CDCL3

0.89013
 0.87614

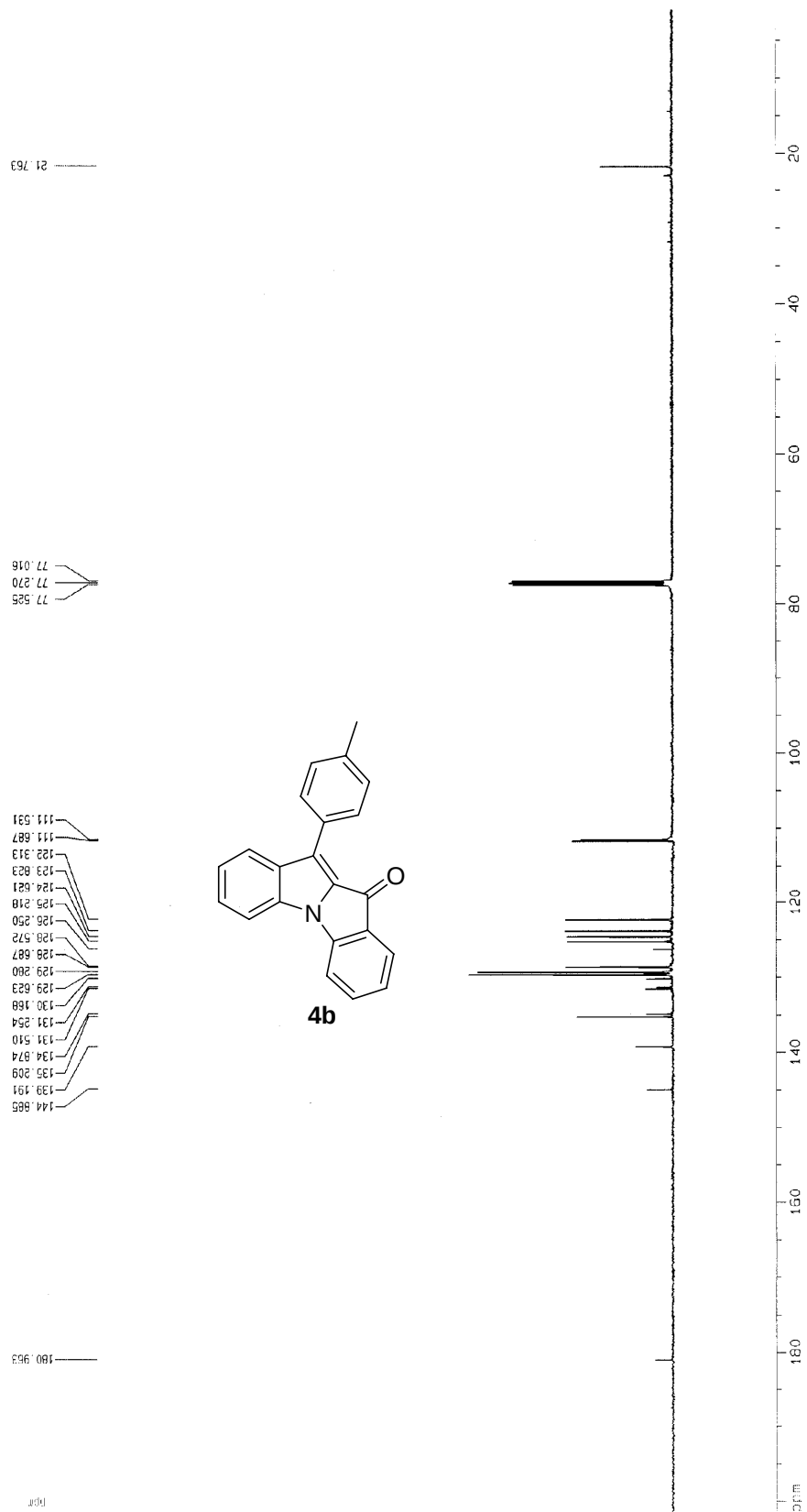
1.64318

2.46116

7.92904
 7.91289
 7.68616
 7.67135
 7.59811
 7.54255
 7.50755
 7.50642
 7.4928
 7.40374
 7.38793
 7.3651
 7.35263
 7.27012
 7.18608
 7.17109
 7.09998
 7.08524



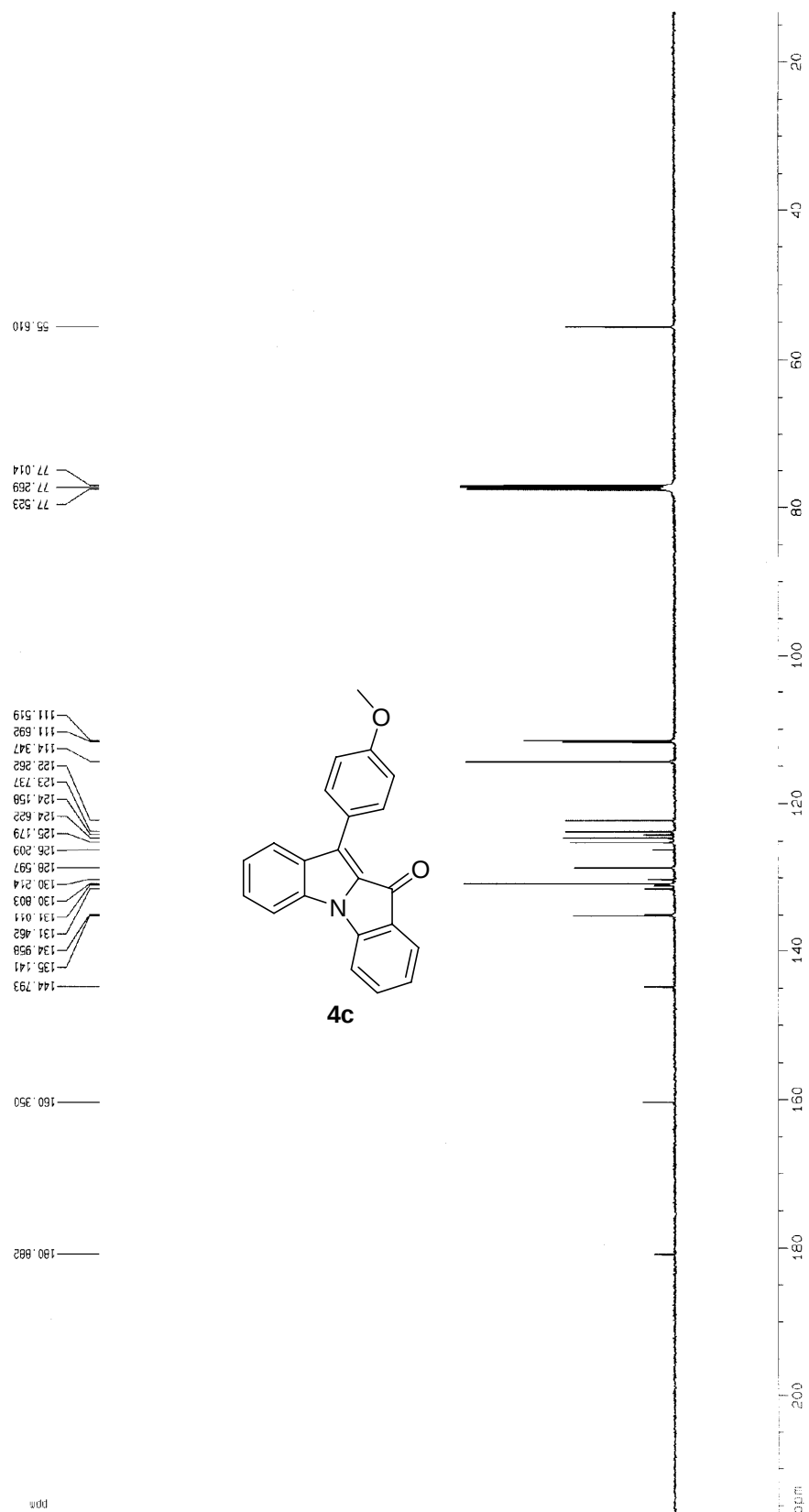
Zhejiang University Avance DMX 500
 QNP 5mm Sample: HD-D-1215 in CDCL3



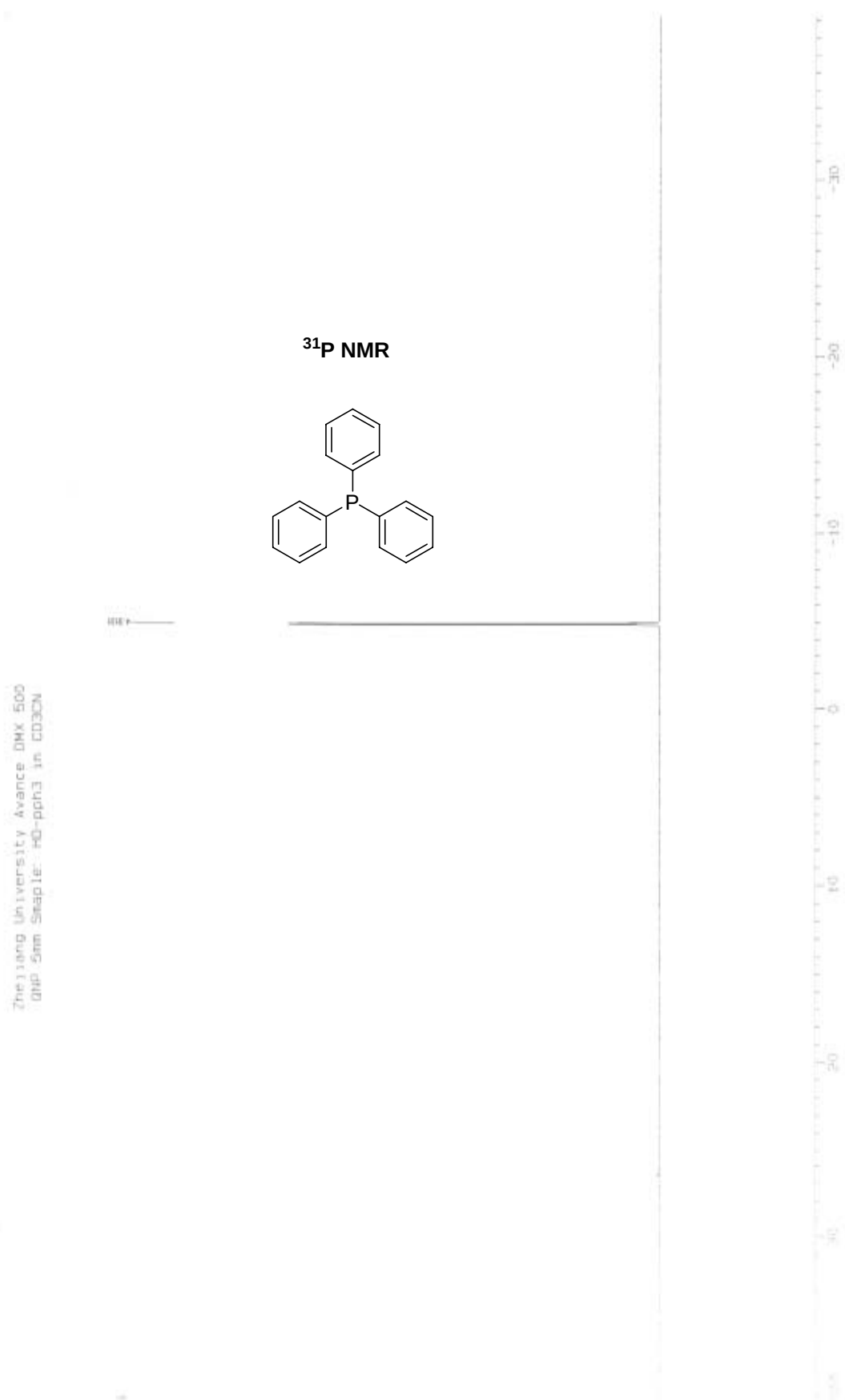
Zhejiang University Avance DMX 500
 GNP 5mm Sample: HO-D-12112 in CDCl₃



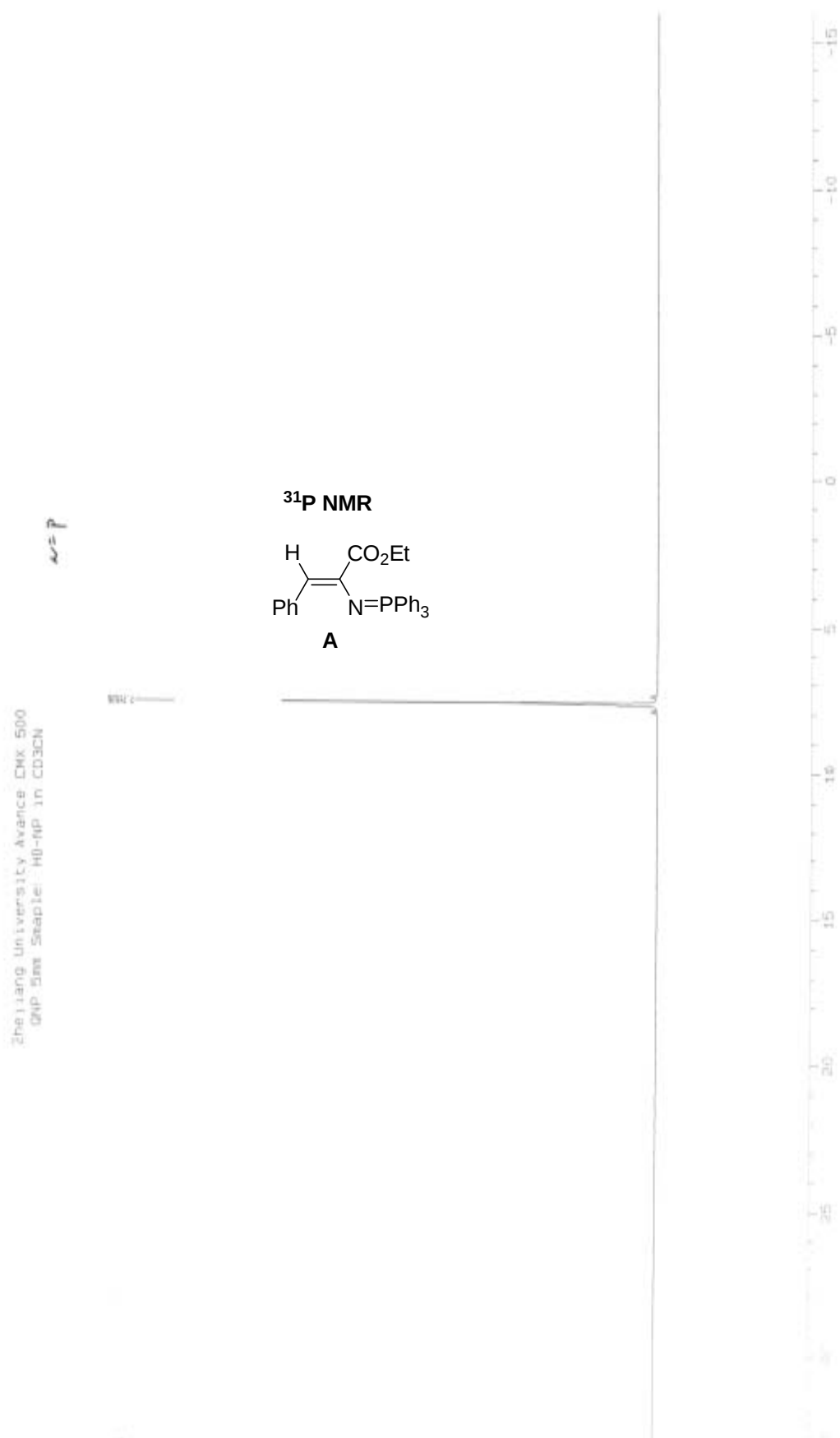
Zhejiang University Avance DMX 500
QNP 5mm Sample: HD-O-12112 in CDCl₃



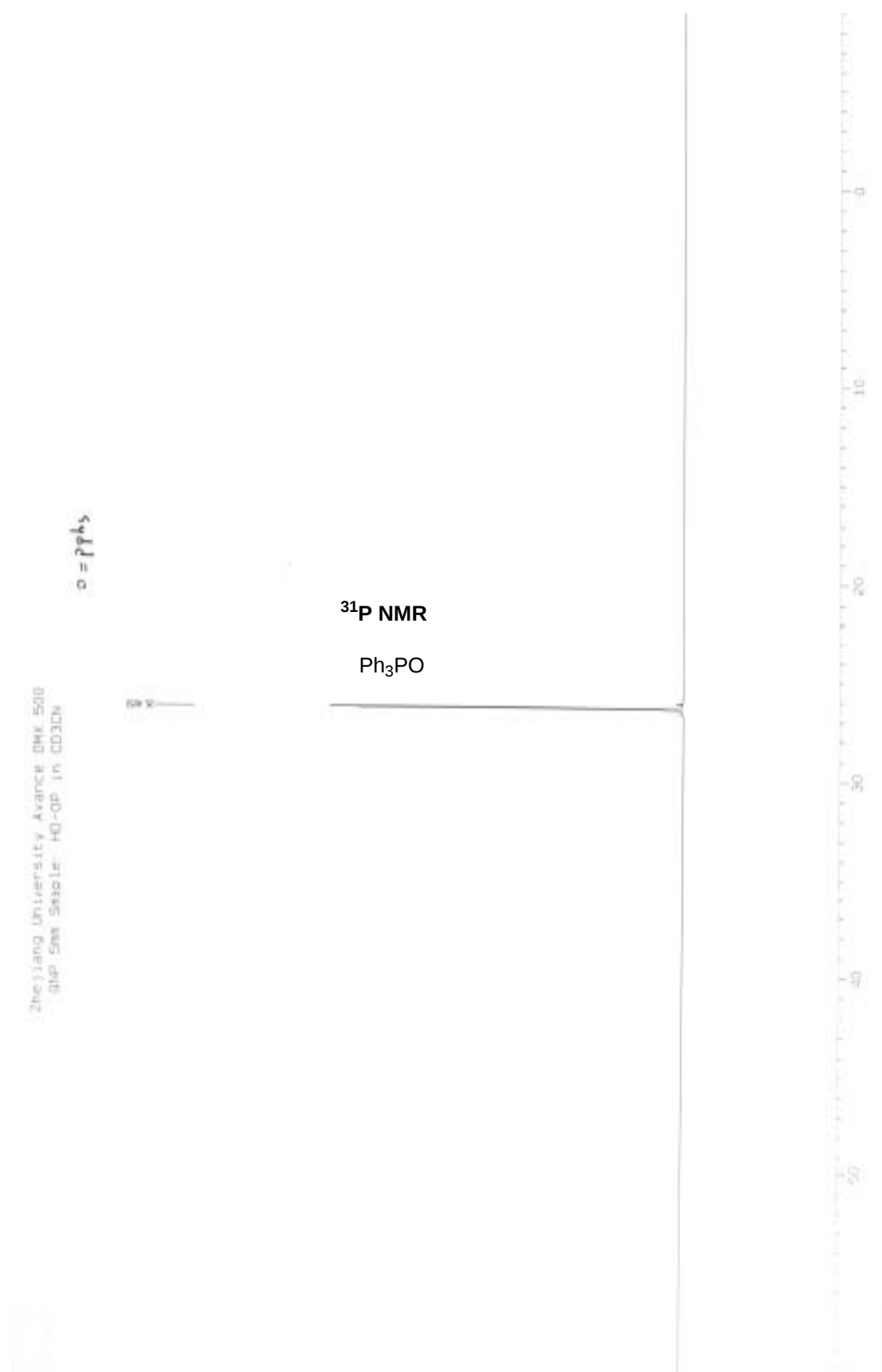
^{31}P NMR spectroscopy for PPh_3



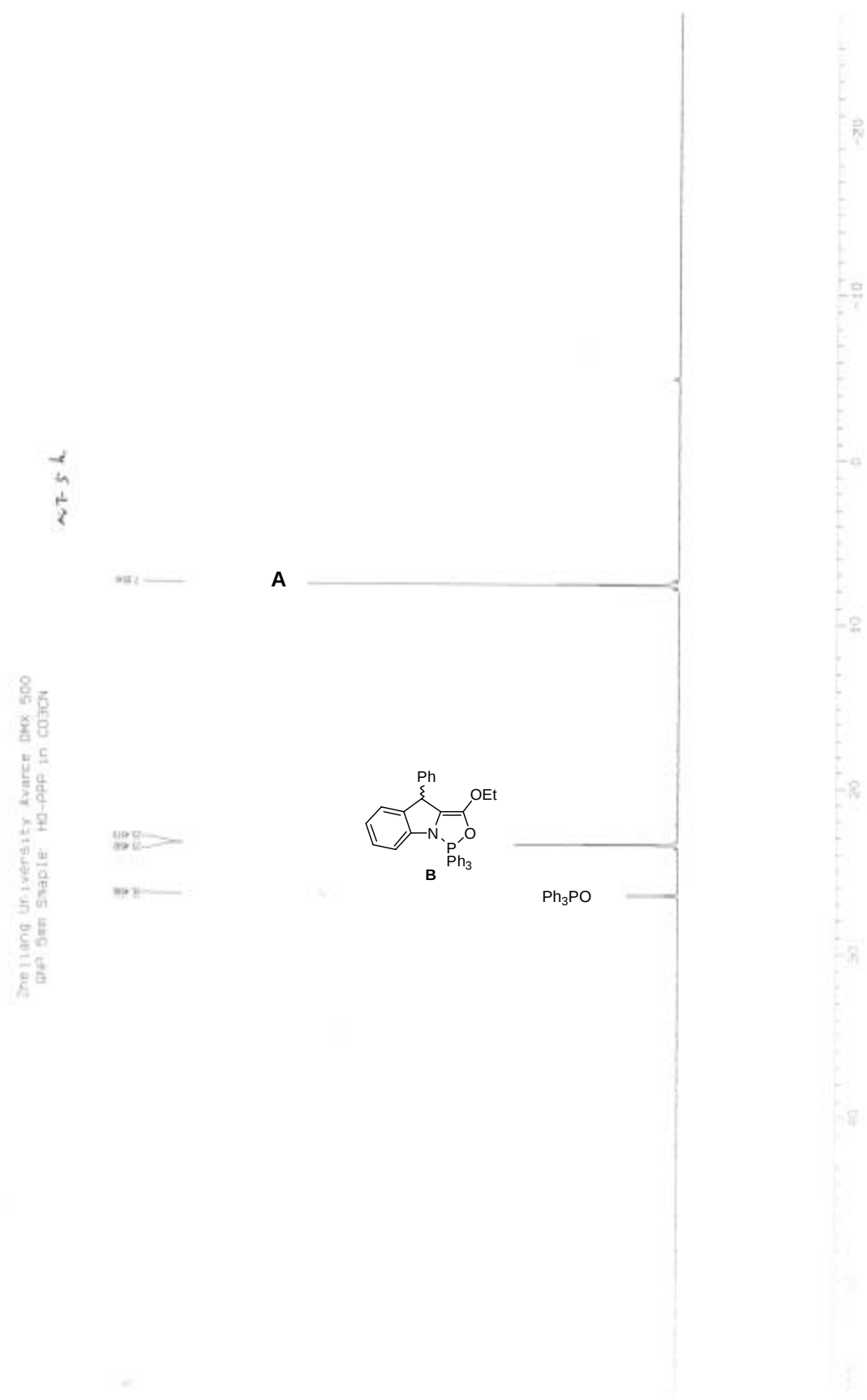
³¹P NMR spectroscopy for intermediate **A**



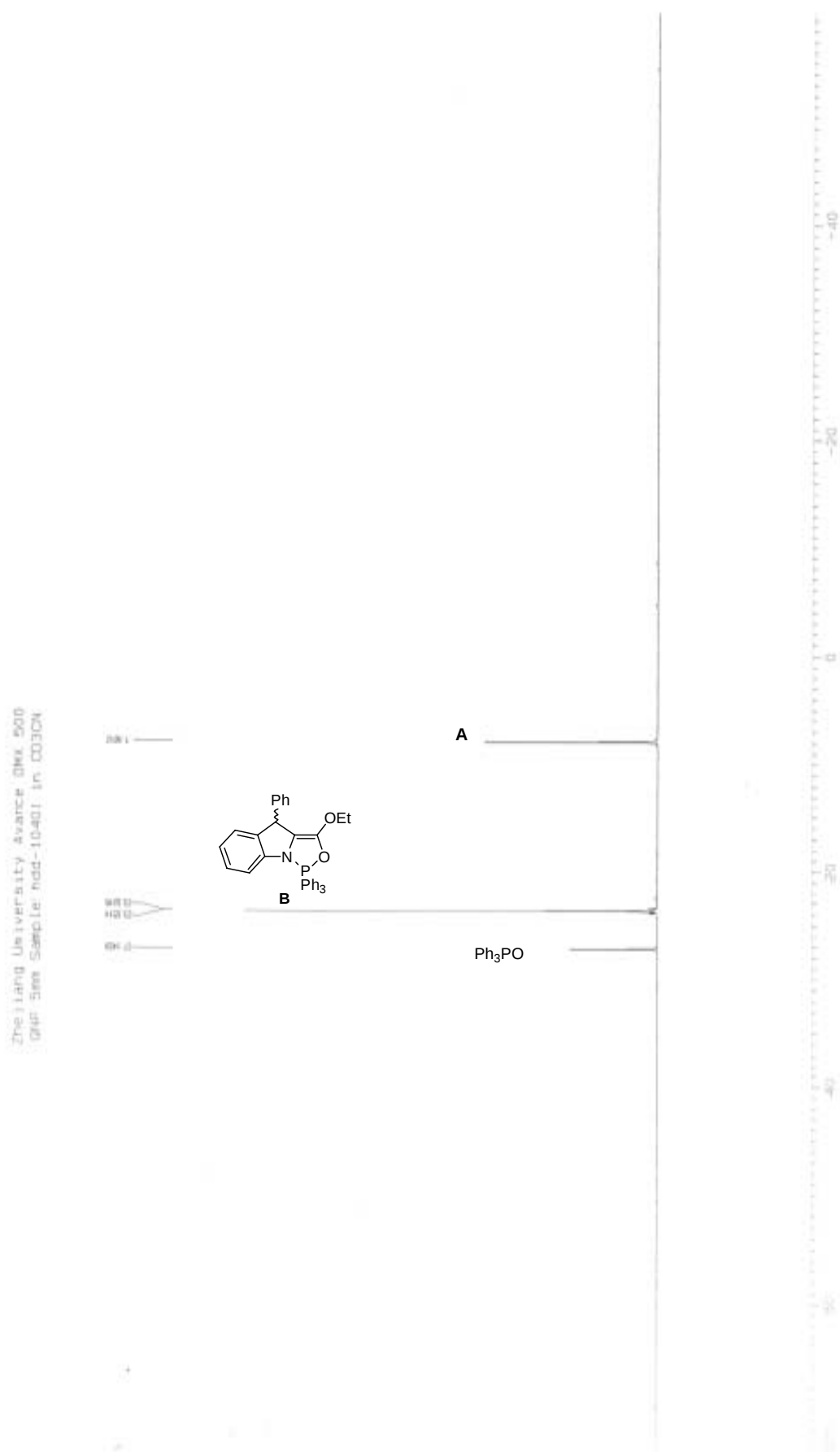
^{31}P NMR spectroscopy for Ph_3PO



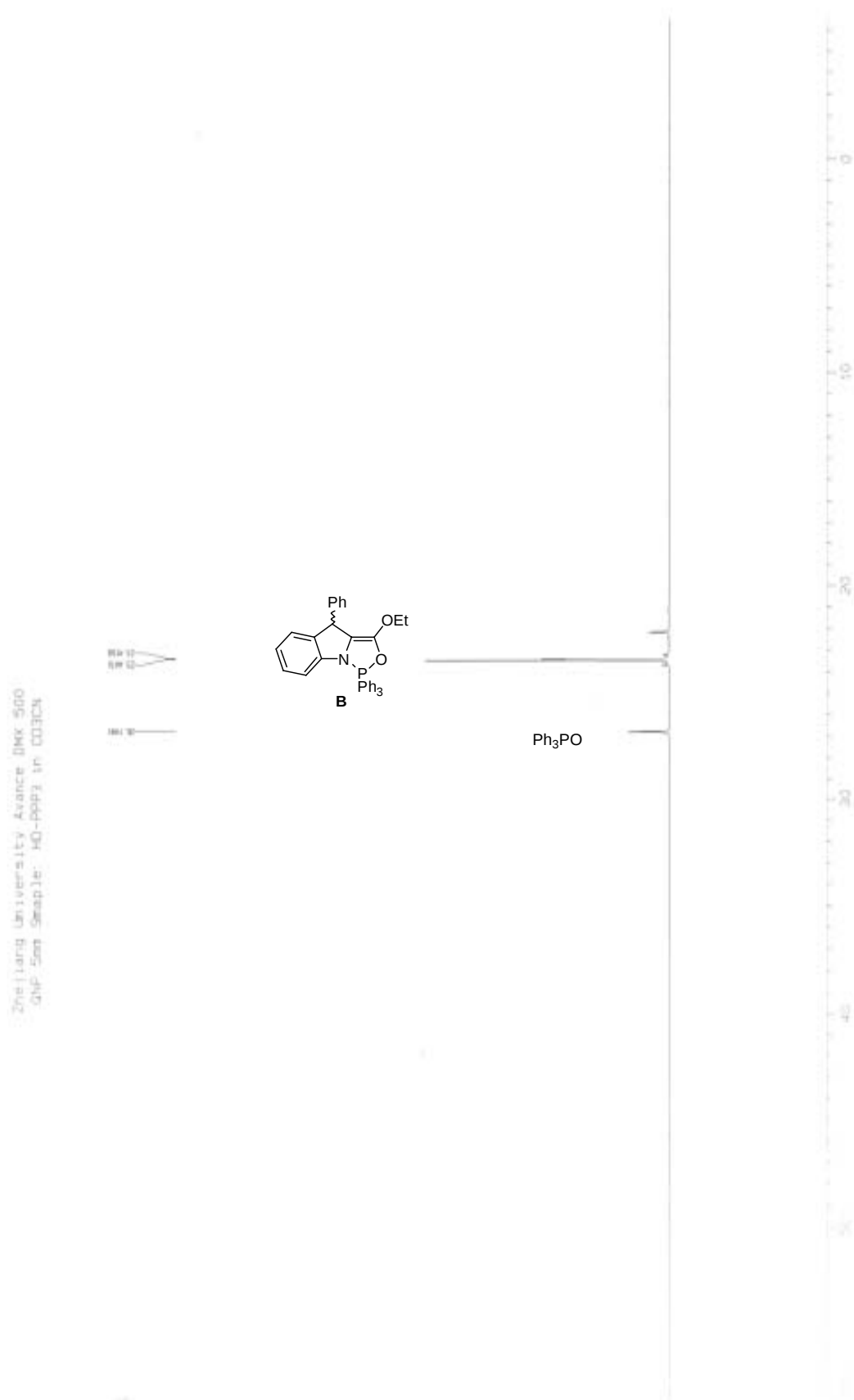
^{31}P NMR spectroscopy for the reaction mixture (after 2 h)



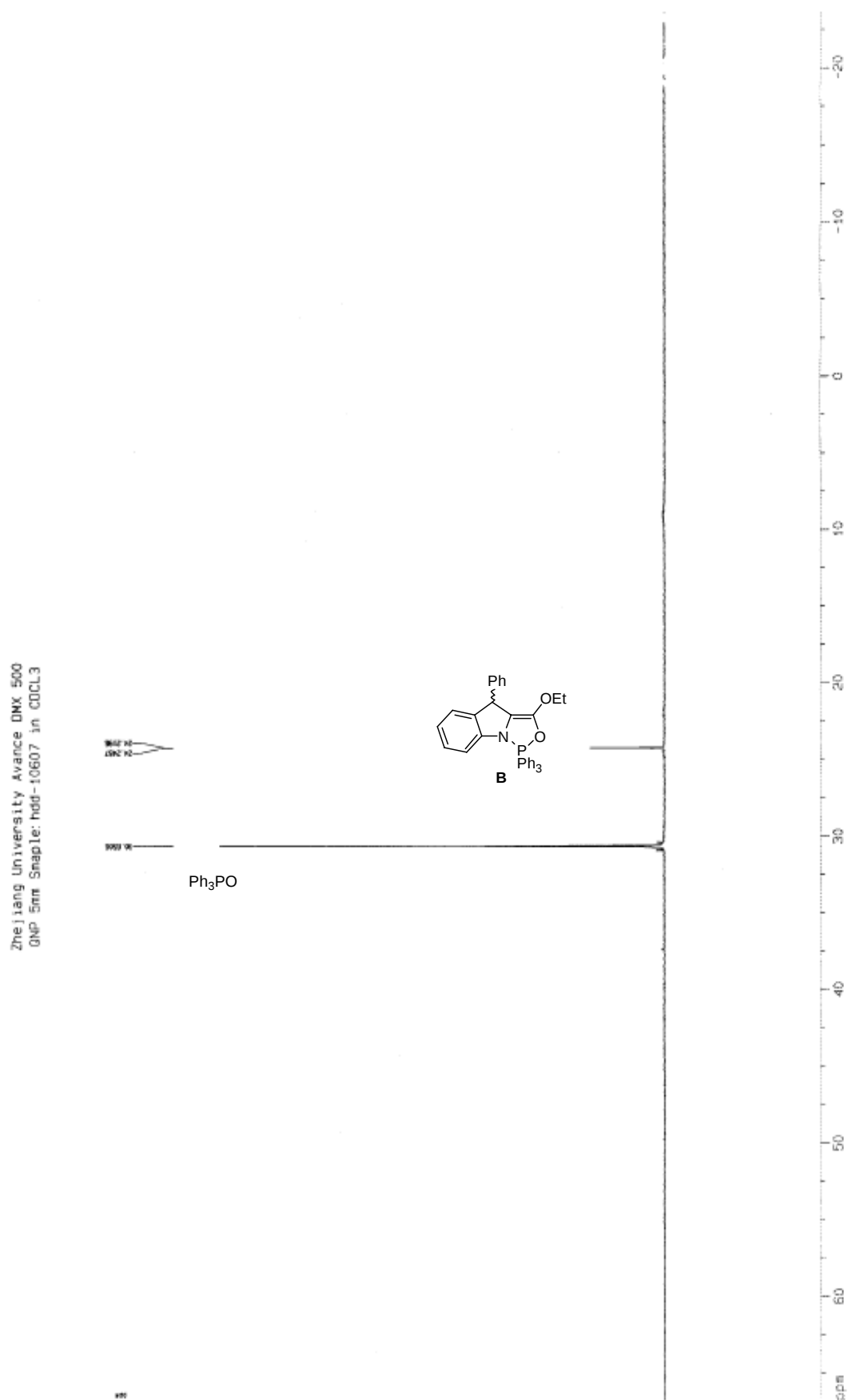
^{31}P NMR spectroscopy for the reaction mixture (after 5 h)



^{31}P NMR spectroscopy for the reaction mixture (after 10 h)



^{31}P NMR spectroscopy for the reaction mixture (after adding D_2O to the stirring mixture)



Display Report

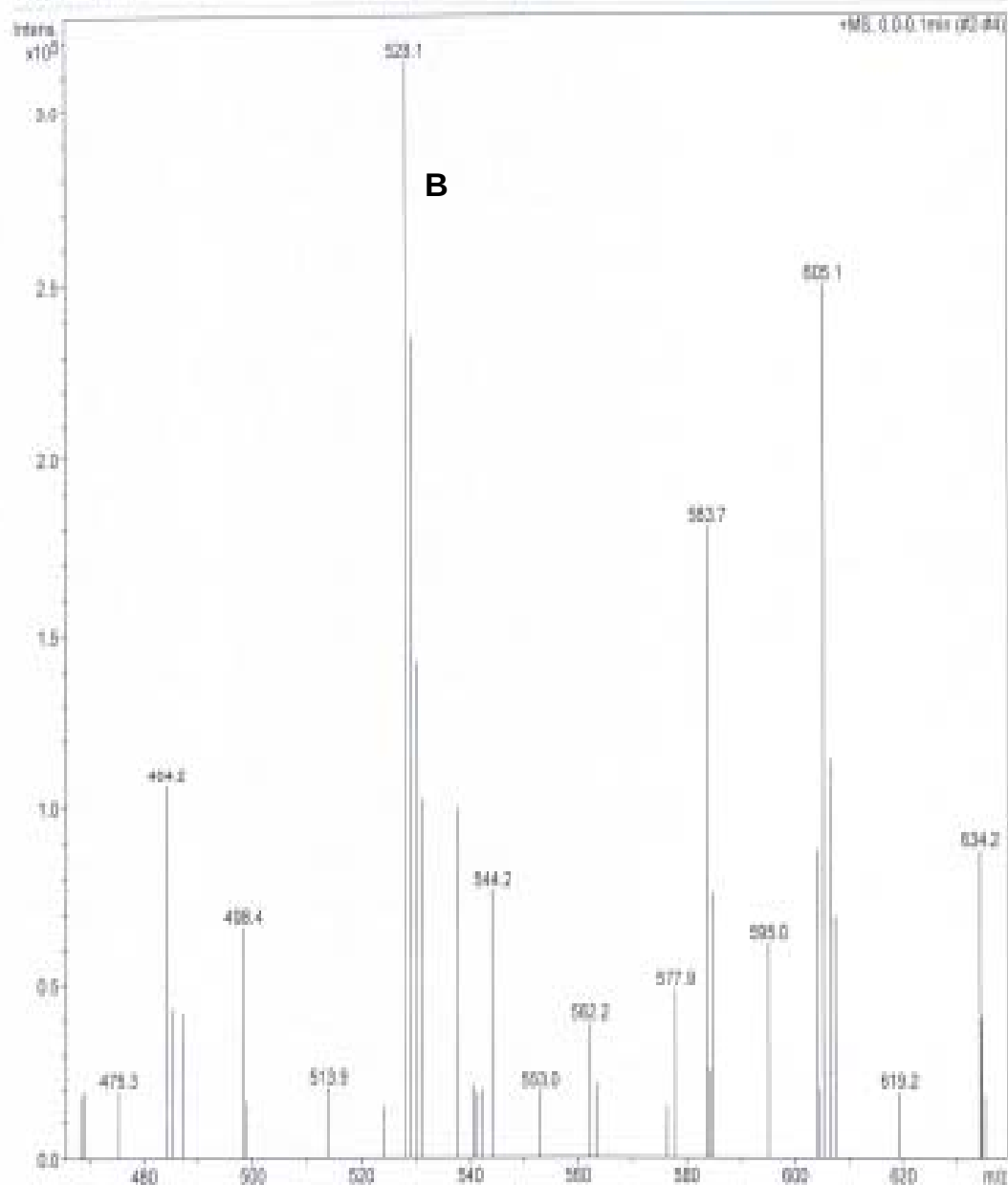
Analysis Info

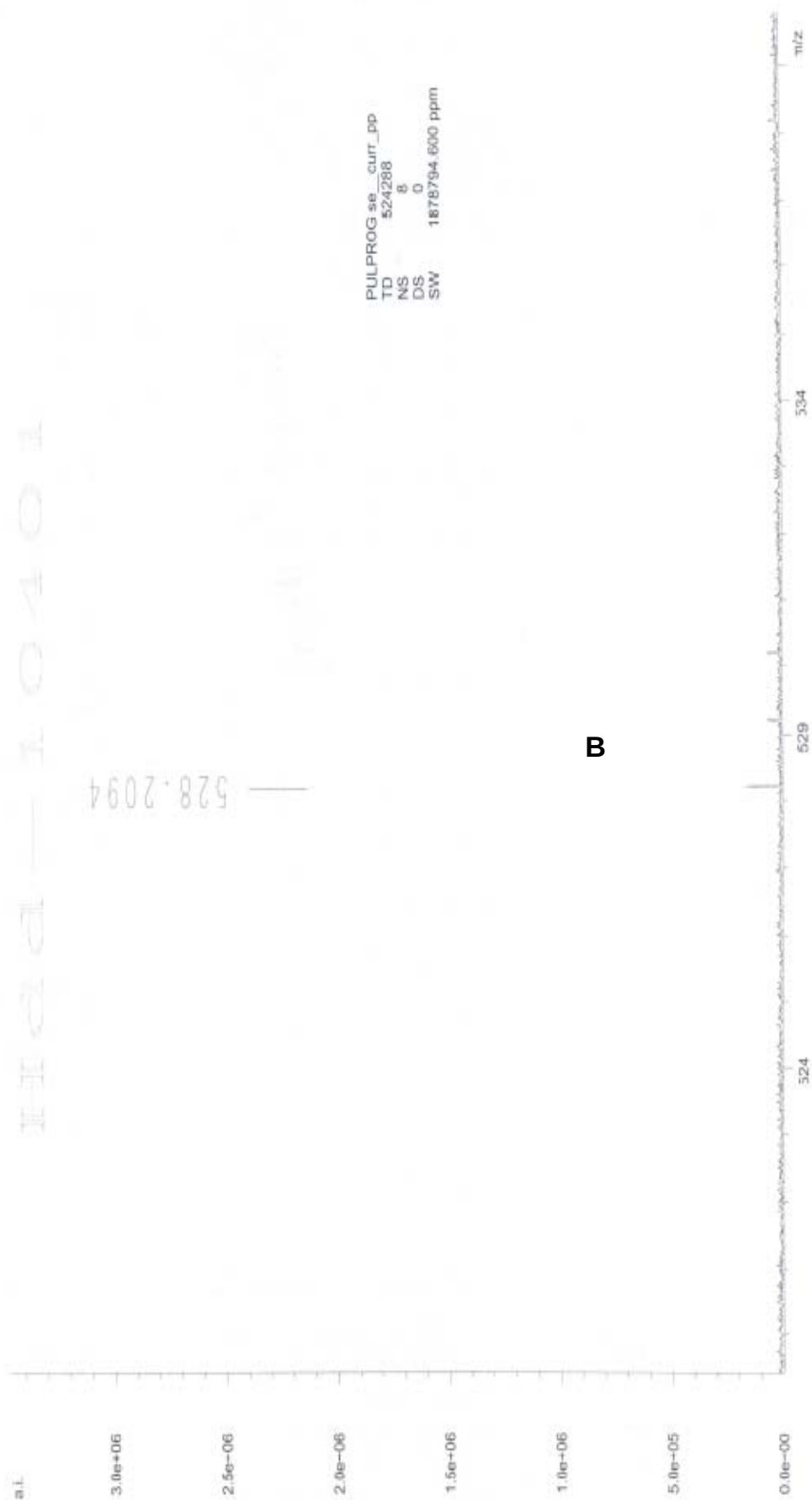
Analysis Name 10040215.d
Method Copy of ESI-0 Default.ms
Sample Name HDD-10401
Comment

Acquisition Date 04/02/10 10:08:41
Operator Administrator
Instrument esquire3000plus

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	off
Mass Range Mode	Std/Normal	Scan Begin	100 m/z	Scan End	2000 m/z
Capillary Exit	113.5 Volt	Skim 1	40.0 Volt	Trap Drive	42.0
Accumulation Time	90 s	Averages	8 Spectra	Auto MS/MS	off





/Data/go100402/hdd10401/pdata/1 Administrator Fri Apr 2 19:28:18 2010