An Unexpected Silver Triflate-Catalyzed Tandem Reaction of *N'*-(2-Alkynylbenzylidene)hydrazide with Ketene

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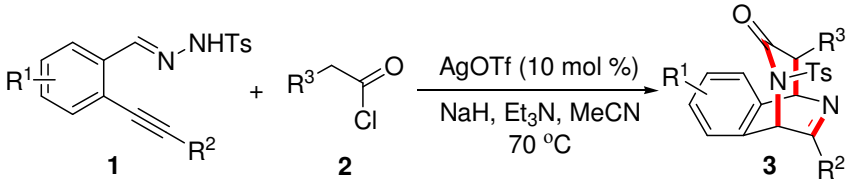
Supporting Information

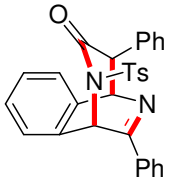
General experimental methods:

Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63μm, 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 at 25–35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

General experimental procedure for silver triflate-catalyzed tandem reaction of N'-(2-alkynylbenzylidene)hydrazide with ketene.

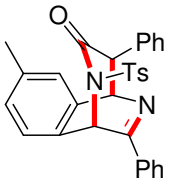


Silver triflate (0.03 mmol, 7.7 mg) was added to a solution of *N*'-(2-alkynylbenzylidene)hydrazide **1** (0.3 mmol) in MeCN (1.0 mL), and the solution was stirred at 70 °C under N_2 for 1 hour. Subsequently, NaH (0.45 mmol, 10.8 mg) with Et_3N (0.045 mmol, 4.6 mg) in MeCN (0.5 mL), and ketene **2** (0.45 mmol) in MeCN (0.5 mL) were added. The reaction was stirred at 70 °C under N_2. After completion of reaction as indicated by TLC, the mixture was diluted with ethyl acetate (5.0 mL) and quenched with water (5.0 mL). The organic layer was washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash column chromatograph (eluting with PE/EA = 4/1) to give the desired product **3**.



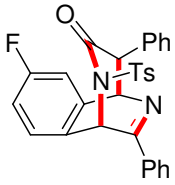
Compound **3a**

White solid. Melting point: 191-192 ℃. Yield: 90% (132.9 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (d, *J* = 7.2 Hz, 2H), 7.70-7.65 (m, 3H), 7.58-7.51 (m, 3H), 7.49-7.45 (m, 1H), 7.43-7.35 (m, 2H), 7.21-7.13 (m, 6H), 6.98-6.95 (m, 2H), 5.53 (s, 1H), 4.09 (s, 1H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.5, 170.4, 144.9, 141.7, 138.2, 135.9, 134.8, 134.6, 131.6, 129.1, 129.0, 128.8, 128.5, 128.4, 128.2, 127.5, 127.4, 126.8, 125.5, 65.0, 60.9, 50.5, 21.6; HRMS calcd. for $C_{30}H_{25}N_2O_3S^+$ $[M+H]^+$: 493.1580, found 493.1600.



Compound **3b**

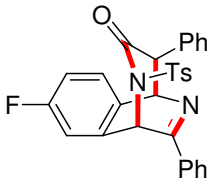
White solid. Melting point: 226-227 ℃. Yield: 73% (110.9 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (d, *J* = 6.8 Hz, 2H), 7.70 (d, *J* = 7.2 Hz, 2H), 7.58- 7.50 (m, 4H), 7.29-7.27 (m, 1H), 7.20-7.11 (m, 7H), 6.9 8-6.96 (m, 2H), 5.47 (s, 1H), 4.07 (s, 1H), 2.40 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 170.6, 144.8, 141.8, 139.3, 138.4, 136.0, 131.6, 131.5, 129.1, 129.0, 128.8, 128.7, 128.5, 128.4, 127.4, 126.7, 126.2, 65.1, 61.1, 50.5, 21.7, 21.4; HRMS calcd. for $C_{31}H_{27}N_2O_3S^+$ $[M+H]^+$: 507.1737, found 507.1721.



Compound **3c**

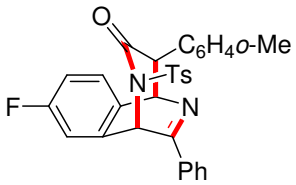
White solid. Melting point: 91-92 ℃. Yield: 58% (88.8 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15-8.11 (m, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 7.58-7.52 (m, 3H), 7.46-7.39

(m, 2H), 7.21-7.19 (m, 3H), 7.16 (d, J = 8.0 Hz, 2H), 7.11-7.10 (m, 1H), 7.08-7.06 (m, 1H), 6.96-6.94 (m, 2H), 5.53 (s, 1H), 4.07 (s, 1H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.3, 170.0, 162.3 (d, $^1J_{CF}$ = 251.9 Hz), 145.2, 138.2, 137.6, 135.9, 134.7, 131.9, 129.3, 129.2, 129.0, 128.7, 128.5, 127.7, 127.5, 127.4, 127.3, 116.0 (d, $^2J_{CF}$ = 21.4 Hz), 114.2 (d, $^2J_{CF}$ = 22.7 Hz), 64.5, 61.2, 50.3, 21.7; HRMS calcd. for $C_{30}H_{24}FN_2O_3S^+$ $[M+H]^+$: 511.1486, found 511.1481.



Compound **3d**

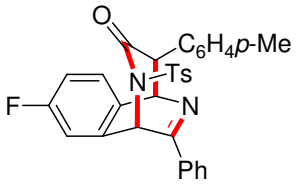
White solid. Melting point: 184-185 °C. Yield: 60% (91.8 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (d, J = 6.4 Hz, 2H), 7.70 (d, J = 7.2 Hz, 2H), 7.58-7.51 (m, 3H), 7.45-7.39 (m, 2H), 7.20-7.15 (m, 5H), 7.11-7.07 (m, 2H), 6.94 (m, 2H), 5.53 (s, 1H), 4.07 (s, 1H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.3, 170.0, 164.2 (d, $^1J_{CF}$ = 246.1 Hz), 145.2, 138.2, 135.9, 134.7, 131.9, 129.3, 129.2, 129.0, 128.7, 128.5, 127.7, 127.6, 127.5, 127.4, 116.1 (d, $^2J_{CF}$ = 21.6 Hz), 114.2 (d, $^2J_{CF}$ = 23.1 Hz), 64.5, 61.2, 50.2, 21.7; HRMS calcd. for $C_{30}H_{24}FN_2O_3S^+$ $[M+H]^+$: 511.1486, found 511.1472.



Compound **3e**

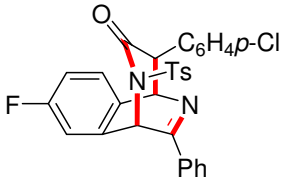
White solid. Melting point: 153-154 °C. Yield: 65% (102.3 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (d, J = 6.8Hz, 2H), 7.69 (d, J = 8.0 Hz, 2H), 7.59-7.54 (m, 3H), 7.43 (d, J = 7.6 Hz, 2H), 7.16-7.07 (m, 6H), 6.88 (t, J = 7.2 Hz, 1H), 6.40 (d, J = 7.2 Hz, 1H), 5.47 (s, 1H), 4.31 (s, 1H), 2.41 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 169.8, 162.1 (d, $^1J_{CF}$ = 249.0 Hz), 145.0, 137.6, 136.5, 136.5, 135.6, 135.2, 134.5, 131.7, 130.7, 129.1, 128.8, 128.1, 127.6, 127.3, 127.0, 126.9, 126.0, 115.9 (d, $^2J_{CF}$ = 21.7 Hz), 114.1 (d, $^2J_{CF}$ = 22.7 Hz), 62.5, 57.3, 50.0, 21.5, 19.4;

HRMS calcd. for $C_{31}H_{26}N_2O_3S^+$ $[M+H]^+$: 525.1643, found 525.1647.



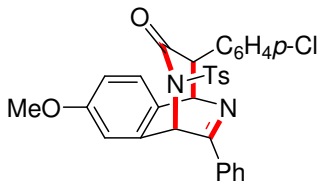
Compound **3f**

White solid. Melting point: 183-184 °C. Yield: 65% (102.3 mg). ^{1}H NMR (400 MHz, $CDCl_3$): 8.13 (d, J = 6.8 Hz, 2H), 7.69 (d, J = 7.6 Hz, 2H), 7.67-7.53 (m, 3H), 7.43-7.38 (m, 2H), 7.16-7.06 (m, 4H), 7.00 (d, J = 7.2 Hz, 2H), 6.84 (d, J = 7.2 Hz, 2H), 5.51 (s, 1H), 4.03 (s, 1H), 2.34 (s, 3H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): 170.5, 170.0, 162.3 (d, $^1J_{CF}$ = 249.1 Hz), 145.2, 137.4, 135.9, 135.2, 134.8, 131.9, 129.4, 129.3, 129.2, 129.0, 128.3, 127.5, 127.4, 127.3, 116.0 (d, $^2J_{CF}$ = 21.4 Hz), 114.2 (d, $^2J_{CF}$= 23.0 Hz), 64.7, 60.8, 50.3, 21.7, 21.2; HRMS calcd. for $C_{31}H_{26}FN_2O_3S^+$ $[M+H]^+$:525.1643, found 525.1621.



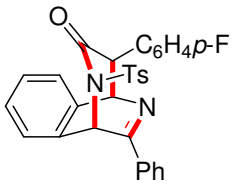
compound **3g**

White solid. Melting point: 89-90 °C. Yield: 72% (117.6 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (d, J = 6.8 Hz, 2H), 7.68 (d, J = 7.2 Hz, 2H), 7.58-7.55 (m, 3H), 7.47-7.38 (m, 2H), 7.15 (t, J = 7.6 Hz, 3H), 7.11-7.03 (m, 2H), 6.90 (d, J = 7.6 Hz, 2H), 6.39 (d, J = 7.2 Hz, 1H), 5.49 (s, 1H), 4.04 (s, 1H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.5, 170.2, 162.2 (d, $^1J_{CF}$ = 257.4 Hz), 145.2, 136.5, 135.6, 134.4, 133.5, 131.9, 130.7, 123.0, 129.8, 129.2, 128.9, 128.7, 128.6, 127.3, 127.1, 116.0 (d, $^2J_{CF}$ = 21.9 Hz), 114.2 (d, $^2J_{CF}$ = 23.5 Hz), 64.2, 57.3, 50.1, 21.6; HRMS calcd. for $C_{30}H_{23}ClFN_2O_3S^+$ $[M+H]^+$: 545.1096, found 545.1072.



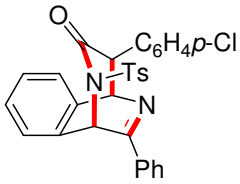
Compound **3i**

White solid. Melting point: 90-91 °C. Yield: 54% (93.7 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (d, *J* = 7.2 Hz, 2H), 7.69 (d, *J* = 7.6 Hz, 2H), 7.58-7.53 (m, 3H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.20-7.07 (m, 6H), 6.89 (d, *J* = 8.0 Hz, 3H), 5.44 (s, 1H), 4.04 (s, 1H), 3.87 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.3, 170.2, 159.6, 145.0, 136.8, 135.8, 134.7, 133.3, 131.7, 130.8, 129.8, 128.8, 128.6, 127.3, 126.6, 114.6, 112.3, 64.2, 60.5, 55.7, 50.7, 21.6; HRMS calcd. for $C_{31}H_{25}ClN_2NaO_4S^+$ $[M+Na]^+$: 579.1116, found 579.1096.



Compound **3j**

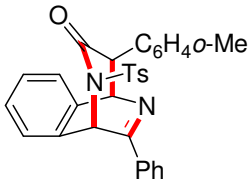
White solid. Melting point: 94-95 °C. Yield: 65% (99.5 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (d, *J* = 7.2 Hz, 2H), 7.70-7.66 (m, 3H), 7.57-7.53 (m, 3H), 7.49-7.43 (m, 1H), 7.41-7.37 (m, 2H), 7.15-7.13 (m, 3H), 6.99-6.96 (m, 2H), 6.89 (t, *J* = 7.2 Hz, 2H), 5.50 (s, 1H), 4.06 (s, 1H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 170.3, 162.0 (d, $^1J_{CF}$ = 246.3 Hz), 145.0, 141.5, 135.8, 134.7, 134.5, 134.1, 131.7, 130.1, 130.1, 129.1, 129.1, 128.8, 128.3, 127.3, 126.9, 125.5, 115.4 (d, $^2J_{CF}$ = 21.7 Hz), 64.9, 60.1, 50.6, 21.6; HRMS calcd. for $C_{30}H_{24}FN_2O_3S^+$ $[M+H]^+$: 511.1486, found 511.1470.



Compound **3k**

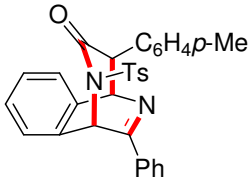
White solid. Melting point: 183-184 °C. Yield: 63% (103.6 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 2H), 7.69-7.66 (m, 3H), 7.57-7.52 (m, 3H), 7.46-7.37 (m, 3H), 7.18-7.13(m, 5H), 6.93 (d, J = 8.0 Hz, 2H), 5.49 (s, 1H), 4.05 (s, 1H), 2.33 (s, 3H); 13C NMR (100 MHz, CDCl3): δ 170.6, 167.0, 145.0, 141.4, 136.7, 135.8, 134.7, 134.4, 133.4, 131.7, 129.8, 129.1, 129.1, 128.8, 128.6, 128.3, 127.3, 126.8, 125.5, 64.8, 60.2, 50.5, 21.5; HRMS calcd. for $C_{30}H_{23}ClN_2NaO_3S^+$ $[M+Na]^+$: 549.1010,

found 549.0994.



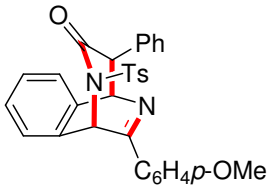
Compound **3l**

White solid. Melting point: 91-92 °C. Yield: 74% (112.4 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (d, *J* = 6.4 Hz, 2H), 7.68 (d, *J* = 7.2 Hz, 3H), 7.55-7.53 (m, 3H), 7.44-7.39 (m, 3H), 7.19-7.09 (m, 5H), 6.88 (t, *J* = 6.8 Hz, 1H), 6.42 (d, *J* = 6.8 Hz, 1H), 5.47 (s, 1H), 4.32 (s, 1H), 2.41 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 170.5, 145.0, 142.0, 136.9, 136.0, 135.4, 135.0, 134.7, 131.8, 130.9, 129.3, 129.2, 129.0 128.4, 127.68, 127.6, 127.0, 126.2, 125.4, 63.3, 57.4, 50.6, 21.7, 19.7; HRMS calcd. for $C_{31}H_{27}N_2O_3S^+$ $[M+H]^+$: 507.1737, found 507.1712.



Compound **3m**

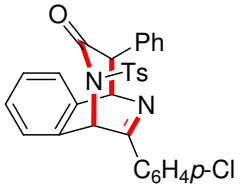
White solid. Melting point: 180-181 °C. Yield: 65% (98.7 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15 (d, *J* = 6.2 Hz, 2H), 7.70-7.66 (m, 3H), 7.54-7.53 (m, 3H), 7.47-7.38 (m, 3H), 7.13 (d, *J* = 8.4 Hz, 3H), 7.00 (d, *J* = 7.2 Hz, 2H), 6.86 (d, *J* = 7.2 Hz, 2H), 5.52 (s, 1H), 4.05 (s, 1H), 2.33 (s, 3H), 2.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 170.3, 144.8, 141.8, 137.1, 136.0, 135.3, 134.9, 134.6, 131.6, 129.2, 129.1, 129.0, 128.8, 128.2, 128.1, 127.4, 126.8, 125.5, 65.1, 60.6, 50.6, 21.6, 21.0; HRMS calcd. for $C_{31}H_{26}N_2NaO_3S^+$ $[M+Na]^+$: 529.1556, found 529.1543.



Compound **3p**

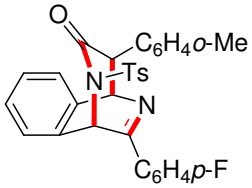
White solid. Melting point: 103-104 °C. Yield: 50% (78.4 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (d, *J* = 7.6 Hz, 2H), 7.72-7.66 (m, 3H), 7.45-7.43 (m, 1H), 7.39-7.38

(m, 2H), 7.20-7.15 (m, 6H), 7.04 (d, J = 7.6 Hz, 2H), 6.97 (m, 2H), 5.47 (s, 1H), 4.07 (s, 1H), 3.89 (s, 3H), 2.33 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 169.4, 162.4, 144.8, 141.9, 138.4, 136.0, 134.7, 129.2, 129.1, 129.0, 128.8, 128.5, 128.4, 128.1, 127.5, 127.4, 126.8, 125.5, 114.3, 64.8, 61.0, 55.4, 50.4, 21.6; HRMS calcd. for $C_{31}H_{27}N_2O_4S^+$ $[M+H]^+$: 523.1686, found 523.1660.



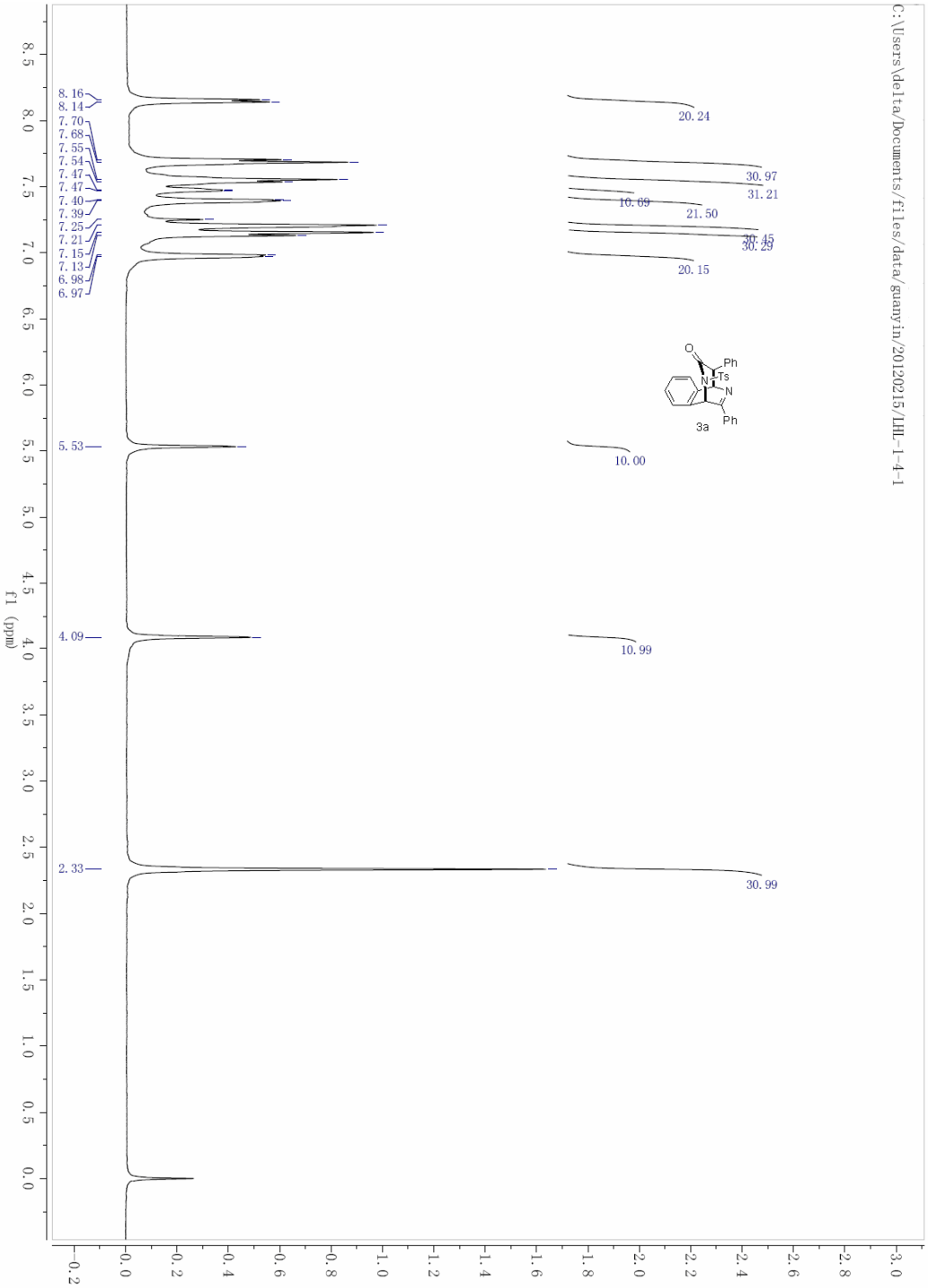
Compound **3q**

White solid. Melting point: 99-100 °C. Yield: 51% (80.5 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, J = 8.0 Hz, 2H), 7.72-7.66 (m, 3H), 7.52-7.45 (m, 3H), 7.41-7.38 (m, 2H), 7.22-7.16 (m, 5H), 7.09-7.07 (m, 1H), 6.98-6.95 (m, 2H), 5.53 (s, 1H), 4.09 (s, 1H), 2.35 (s, 3H).; ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.4, 169.3, 145.0, 141.6, 138.1, 138.0, 135.9, 134.4, 133.2, 129.3, 129.2, 128.8, 128.7, 128.6, 128.3, 127.6, 126.8, 125.6, 65.1, 60.9, 50.4, 21.6; $C_{30}H_{24}ClN_2O_3S^+$ $[M+H]^+$: 527.1191, found 527.1179.



Compound **3r**

White solid. Melting point: 109-110 °C. Yield: 62% (97.5 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19-8.16 (m, 2H), 7.70 (d, J = 7.6 Hz, 3H), 7.46-7.40 (m, 3H), 7.26-7.09 (m, 7H), 6.89 (t, J = 7.2 Hz, 1H), 6.41 (d, J = 7.2 Hz, 1H), 5.44 (s, 1H), 4.31 (s, 1H), 2.41 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.1, 169.2, 162.7 (d, $^1J_{CF}$ = 239.7 Hz), 145.1, 141.9, 136.8, 136.0, 135.3, 134.5, 130.9, 129.8, 129.7, 129.4, 129.2, 129.0, 128.4, 128.2, 127.7, 127.0, 126.2, 125.4, 116.3 (d, $^2J_{CF}$ = 21.9 Hz), 63.2, 57.3, 50.5, 21.7, 19.6; HRMS calcd. for $C_{31}H_{26}FN_2O_3S^+$ $[M+H]^+$: 525.1643, found 525.1617.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8.14
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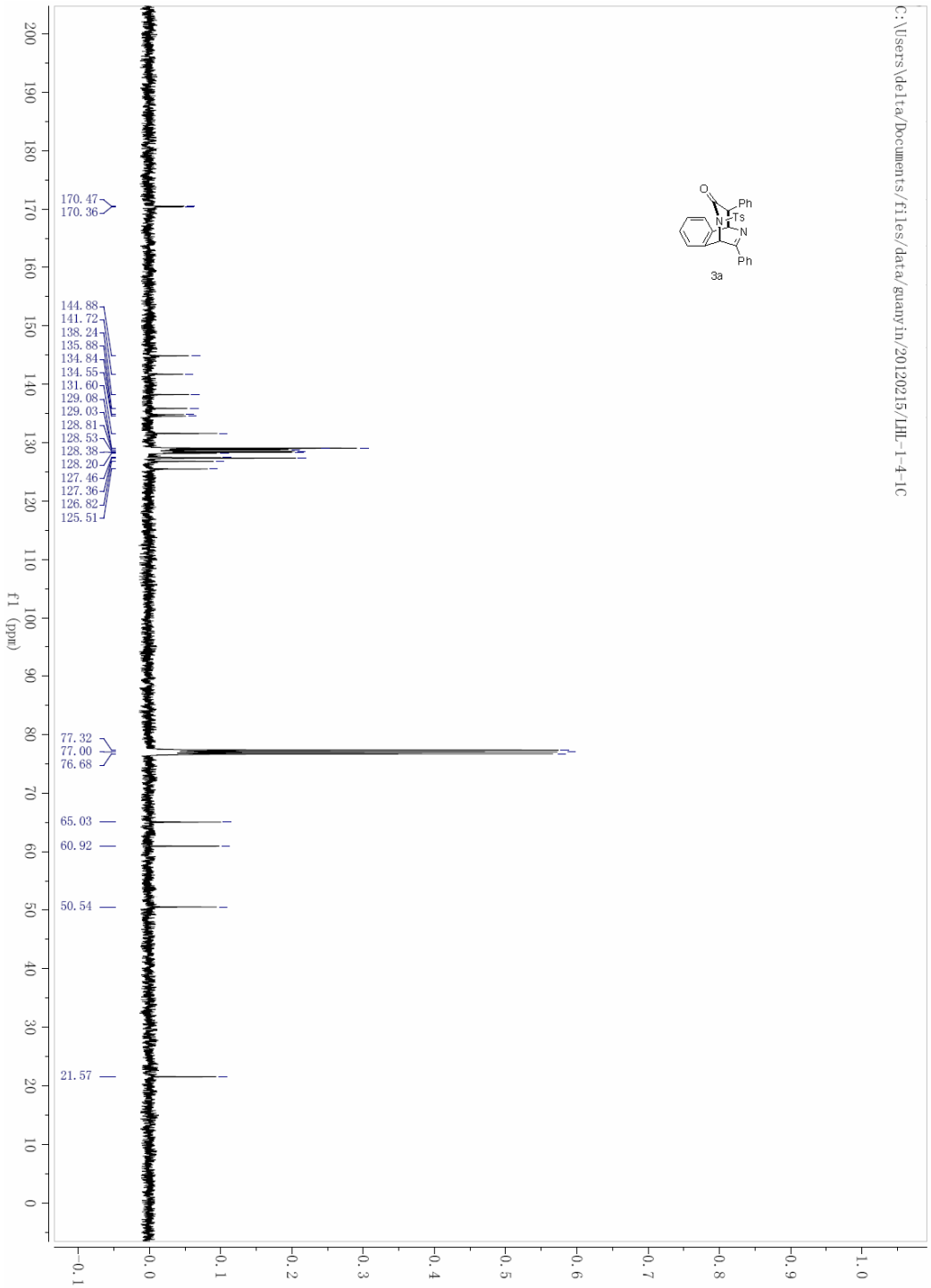
7.68
7.55
7.54
7.47
7.47
7.40
7.39
7.25
7.21
7.15
7.13
6.98
6.97
5.53
4.09
2.33
20.24
30.97
31.21
16.69
21.50
30.45
30.29
20.15
10.00
10.99
30.99
O
Ph
Ts
N
N
Ph
3a
f1 (ppm)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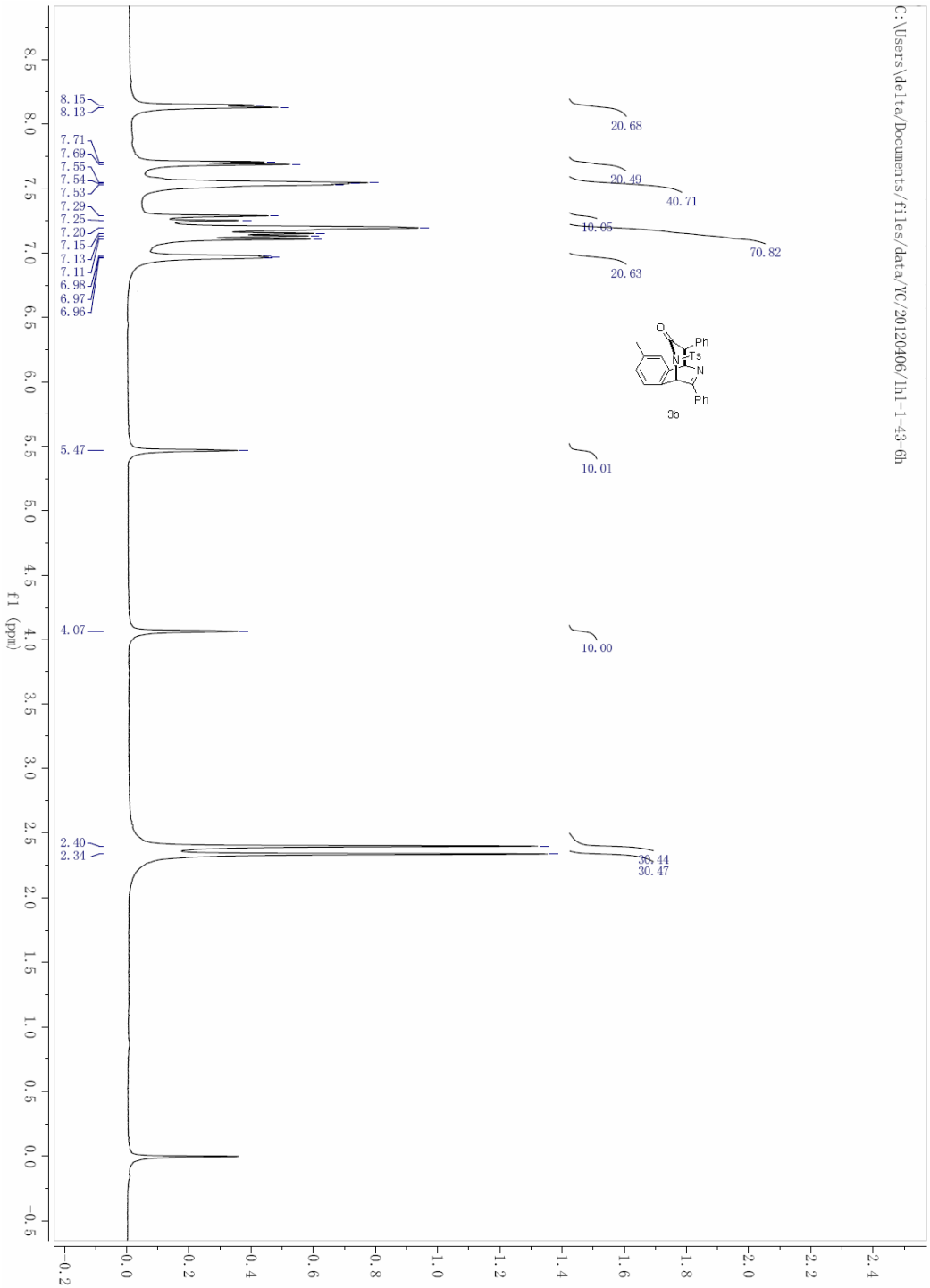
f1 (ppm)
170.47
170.36
144.88
141.72
138.24
135.88
134.84
134.55
131.60
129.08
129.03
128.81
128.53
128.38
128.20
127.46
127.36
126.82
125.51
77.32
77.00
76.68
65.03
60.92
50.54
21.57
O
Ph
Ts
N
N
Ph
3a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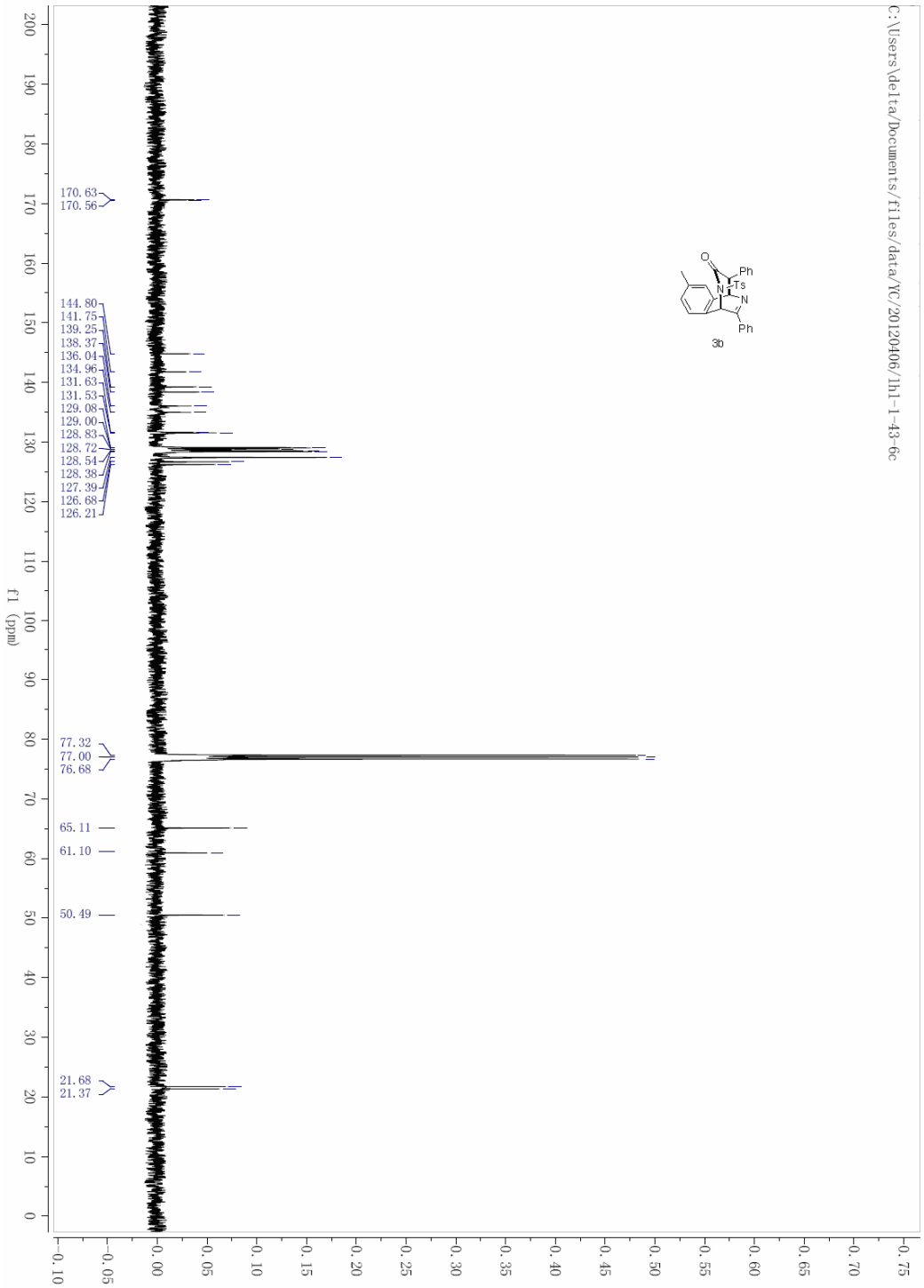
8.15
8.13
7.71
7.69
7.55
7.54
7.53
7.29
7.25
7.20
7.15
7.13
7.11
6.98
6.97
6.96
5.47
4.07
2.40
2.34
20.68
20.49
40.71
10.05
70.82
20.63
10.01
10.00
30.44
30.47
O
Ph
Ts
N
N
Ph
3b
f1 (ppm)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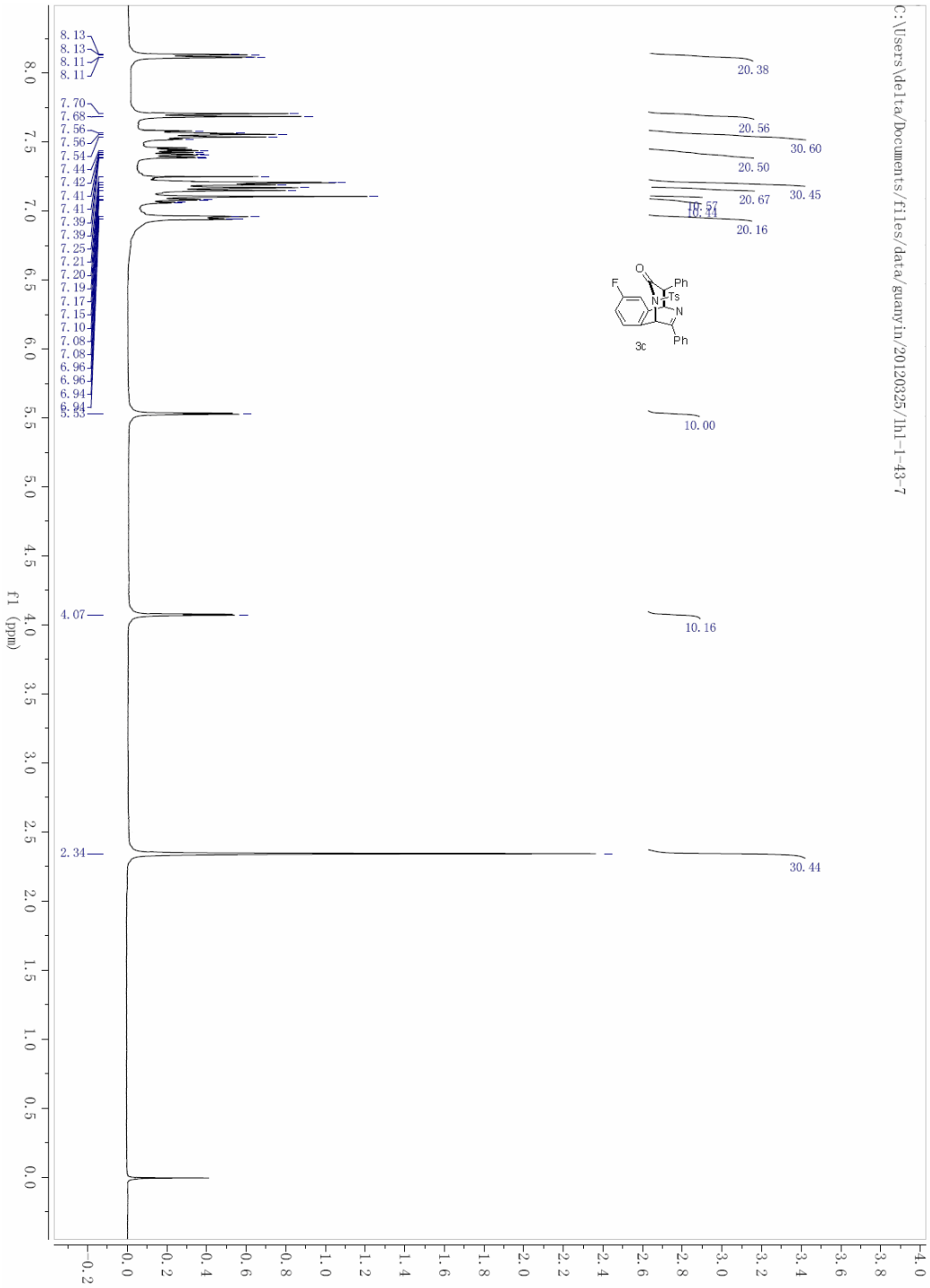
170.63
170.56
144.80
141.75
139.25
138.37
136.04
134.96
131.63
131.53
129.08
129.00
128.83
128.72
128.54
128.38
127.39
126.68
126.21
77.32
77.00
76.68
65.11
61.10
50.49
21.68
21.37
f1 (ppm)
Ph
Ts
N
N
Ph
O
3b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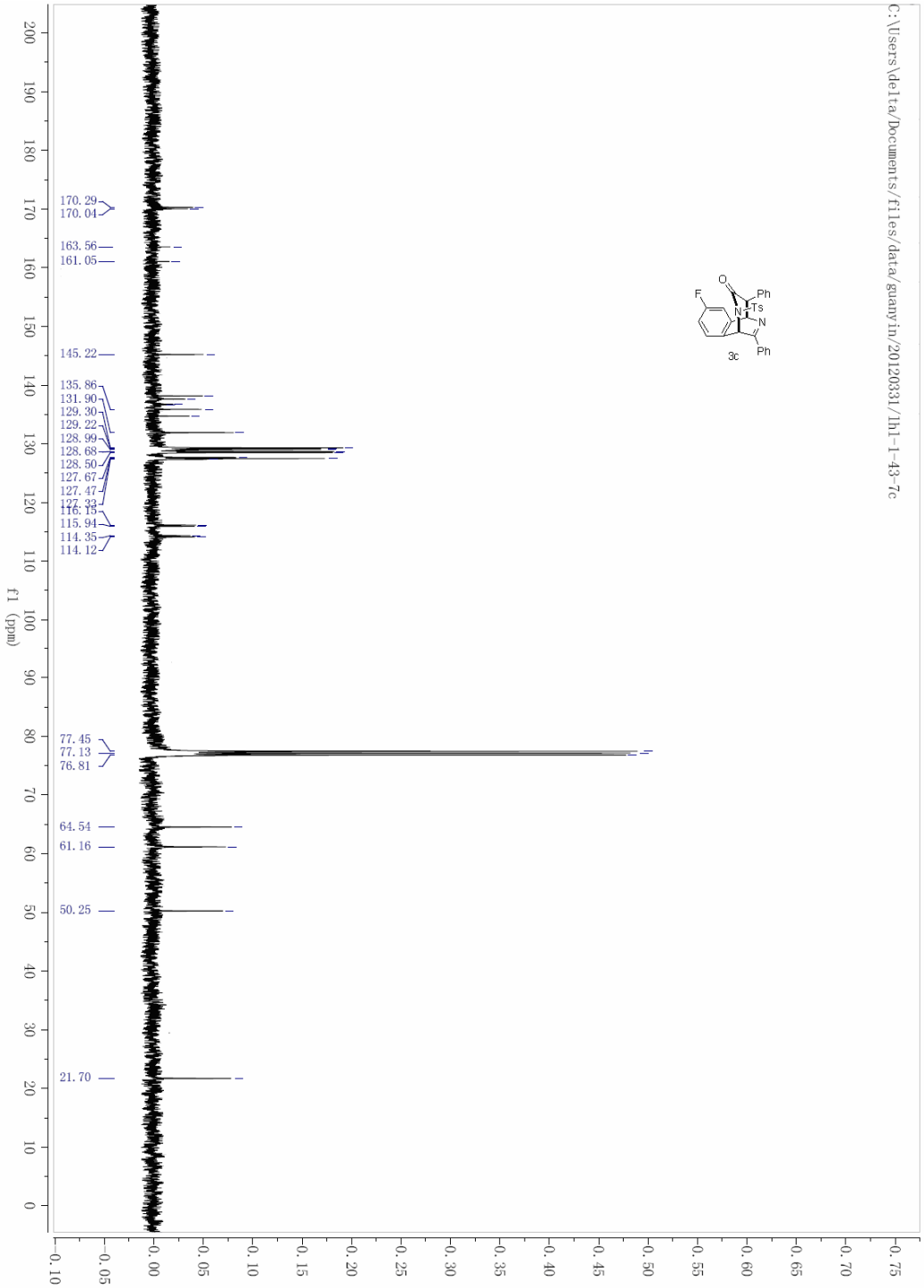
8.13
8.13
8.11
8.11
7.70
7.68
7.56
7.56
7.54
7.44
7.42
7.41
7.41
7.39
7.39
7.25
7.21
7.20
7.19
7.17
7.15
7.10
7.08
7.08
6.96
6.96
6.94
6.94
5.33
4.07
2.34
20.38
20.56
30.60
20.50
30.45
20.67
20.16
10.00
10.16
30.44
F
O
Ph
Ts
N
N
Ph
3c
f1 (ppm)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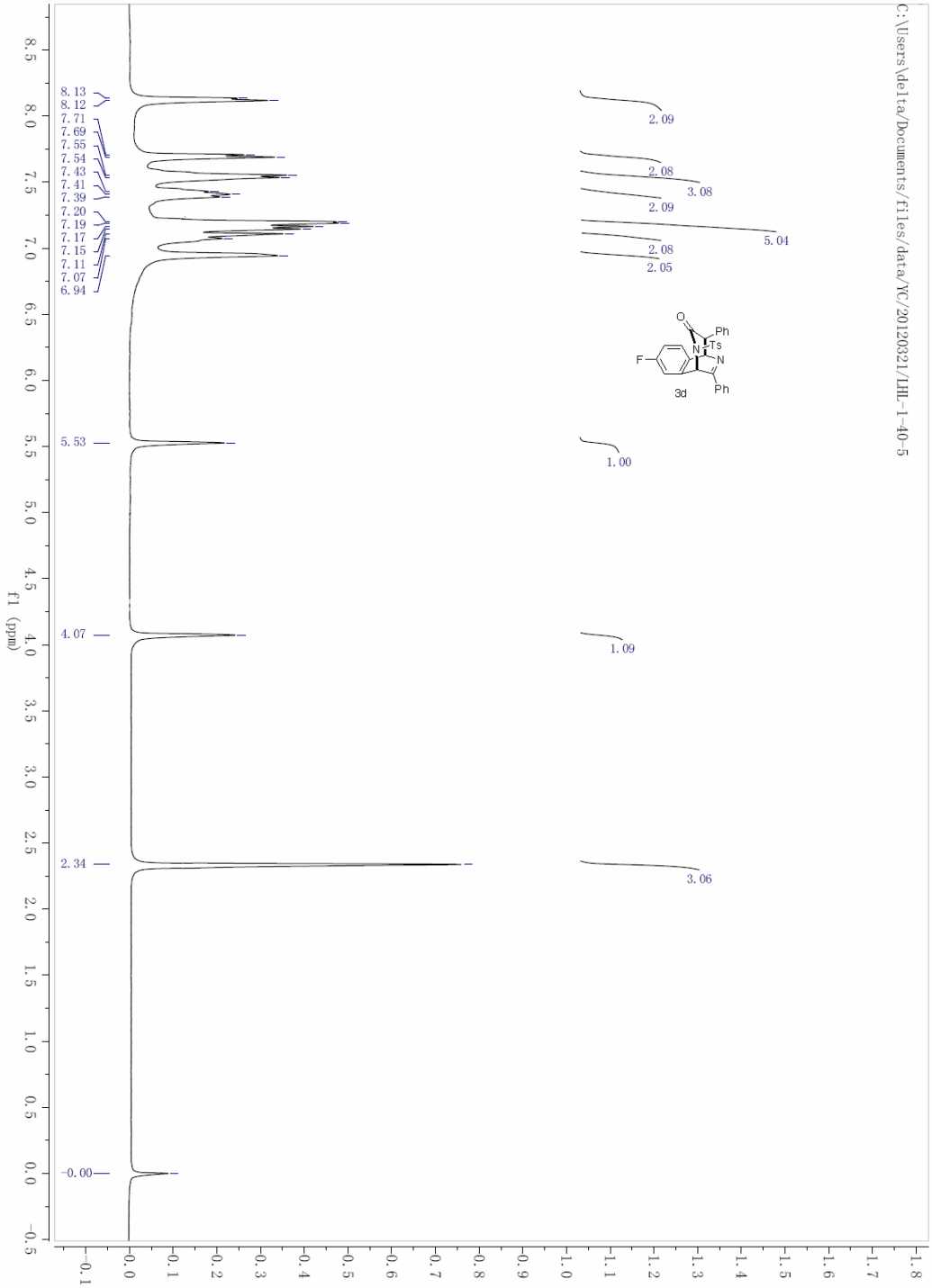
f1 (ppm)
170.29
170.04
163.56
161.05
145.22
135.86
131.90
129.30
129.22
128.99
128.68
128.50
127.67
127.47
127.33
116.15
115.94
114.35
114.12
77.45
77.13
76.81
64.54
61.16
50.25
21.70
F
O
Ph
Ts
N
N
Ph
3c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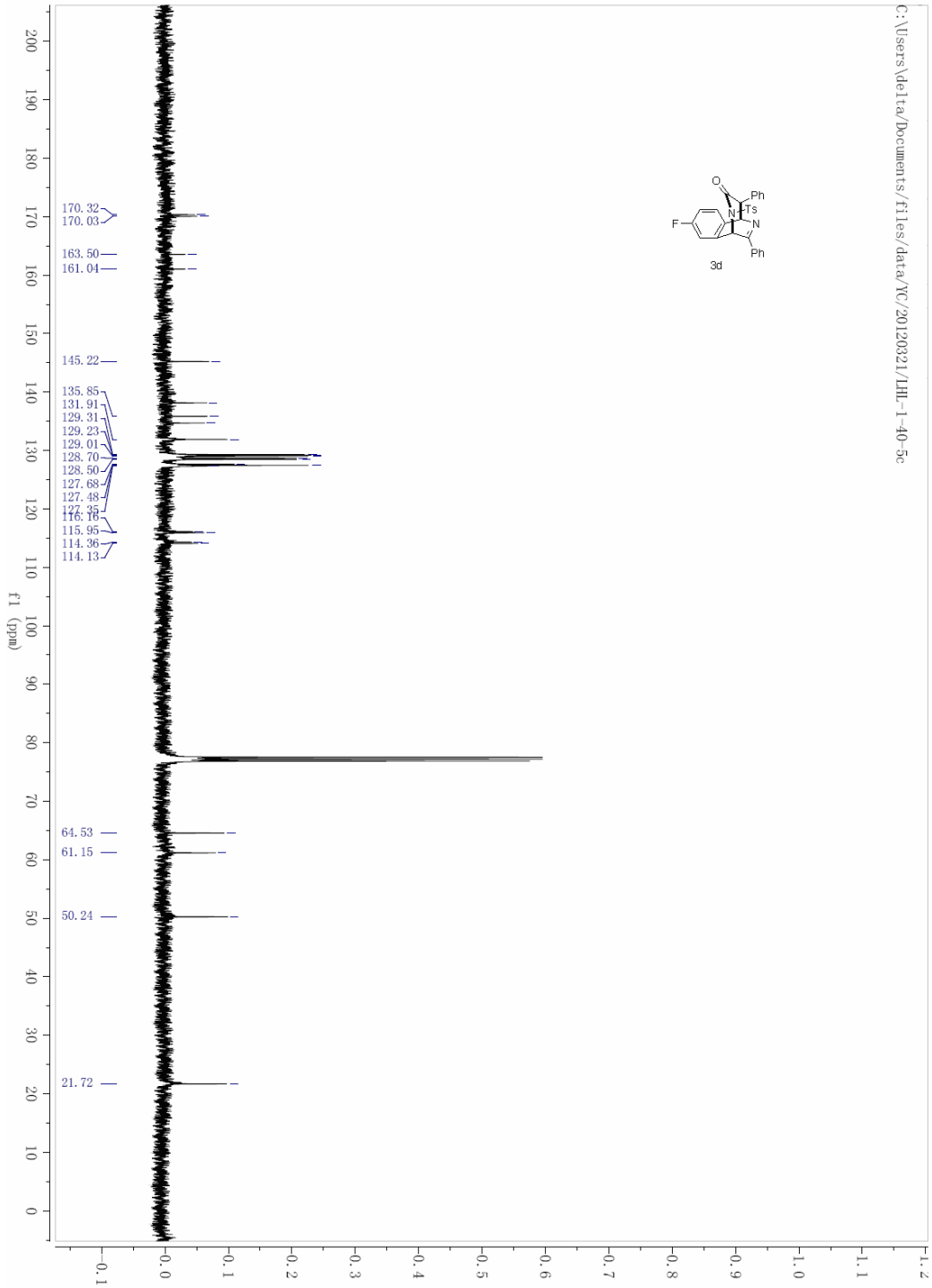
f1 (ppm)
8.13
8.12
7.71
7.69
7.55
7.54
7.43
7.41
7.39
7.20
7.19
7.17
7.15
7.11
7.07
6.94
5.53
4.07
2.34
-0.00
2.09
2.08
3.08
2.09
5.04
2.08
2.05
1.00
1.09
3.06
O
Ph
Ts
N
N
F
Ph
3d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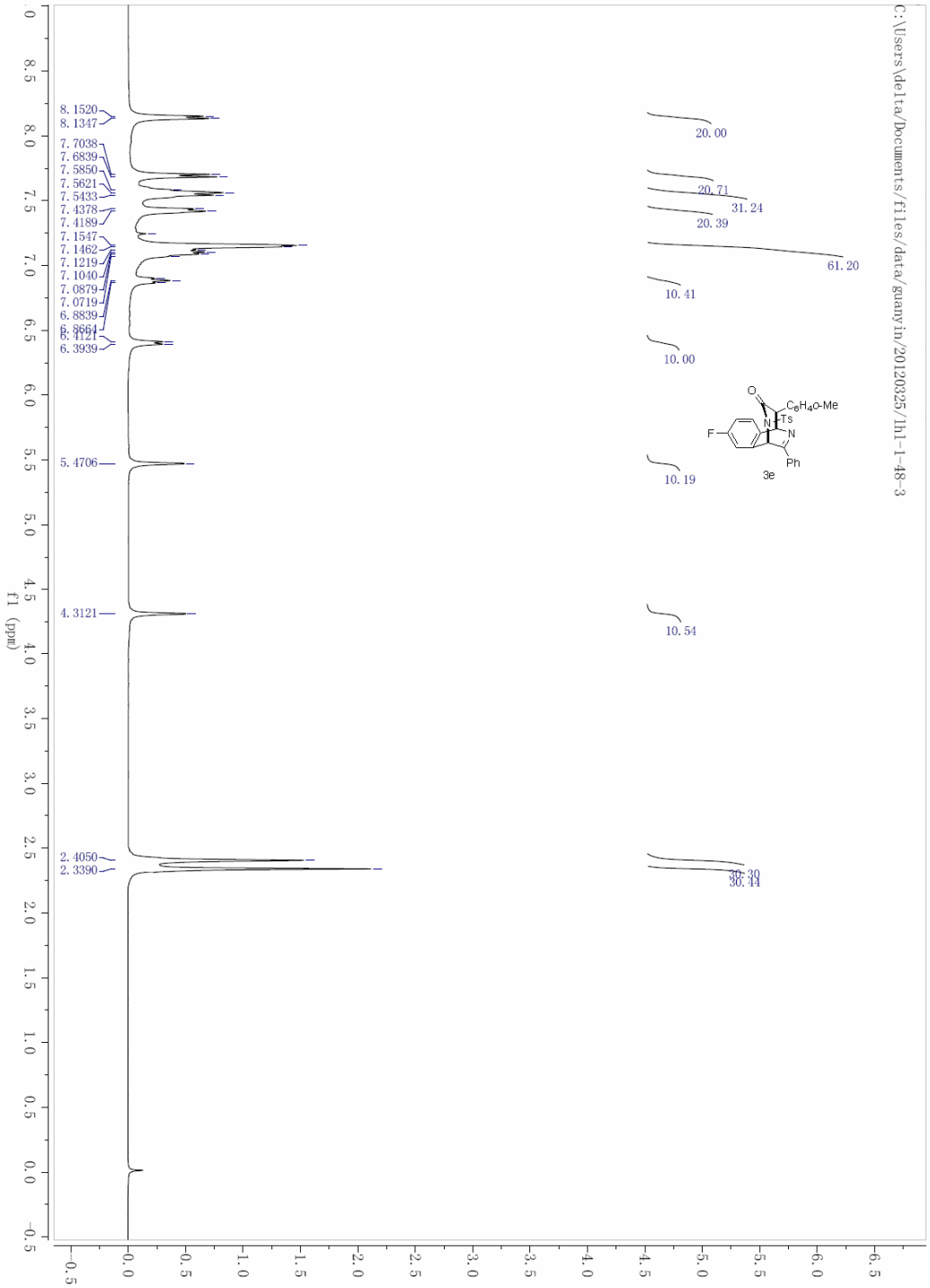
170.32
170.03
163.50
161.04
145.22
135.85
131.91
129.31
129.23
129.01
128.70
128.50
127.68
127.48
127.35
116.16
115.95
114.36
114.13
64.53
61.15
50.24
21.72
f1 (ppm)
O
Ph
Ts
N
N
F
Ph
3d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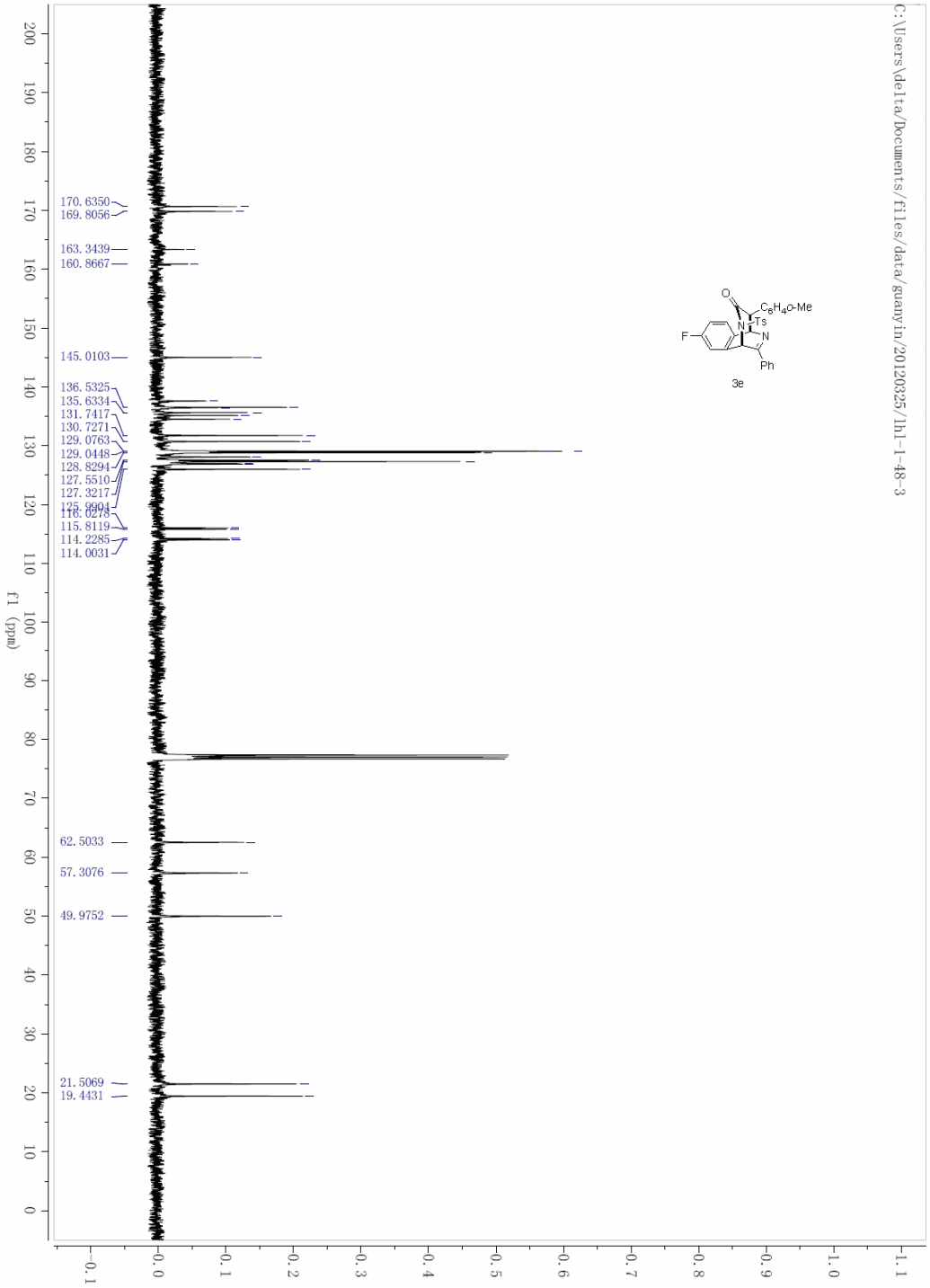
8.1347
7.7038
7.6839
7.5850
7.5621
7.5433
7.4378
7.4189
7.1547
7.1462
7.1219
7.1040
7.0879
7.0719
6.8839
6.8664
6.4121
6.3939
5.4706
4.3121
2.4050
2.3390
20.00
20.71
31.24
20.39
61.20
10.41
10.00
10.19
10.54
30.30
30.44
O
C6H4o-Me
Ts
N
F
N
Ph
3e
f1 (ppm)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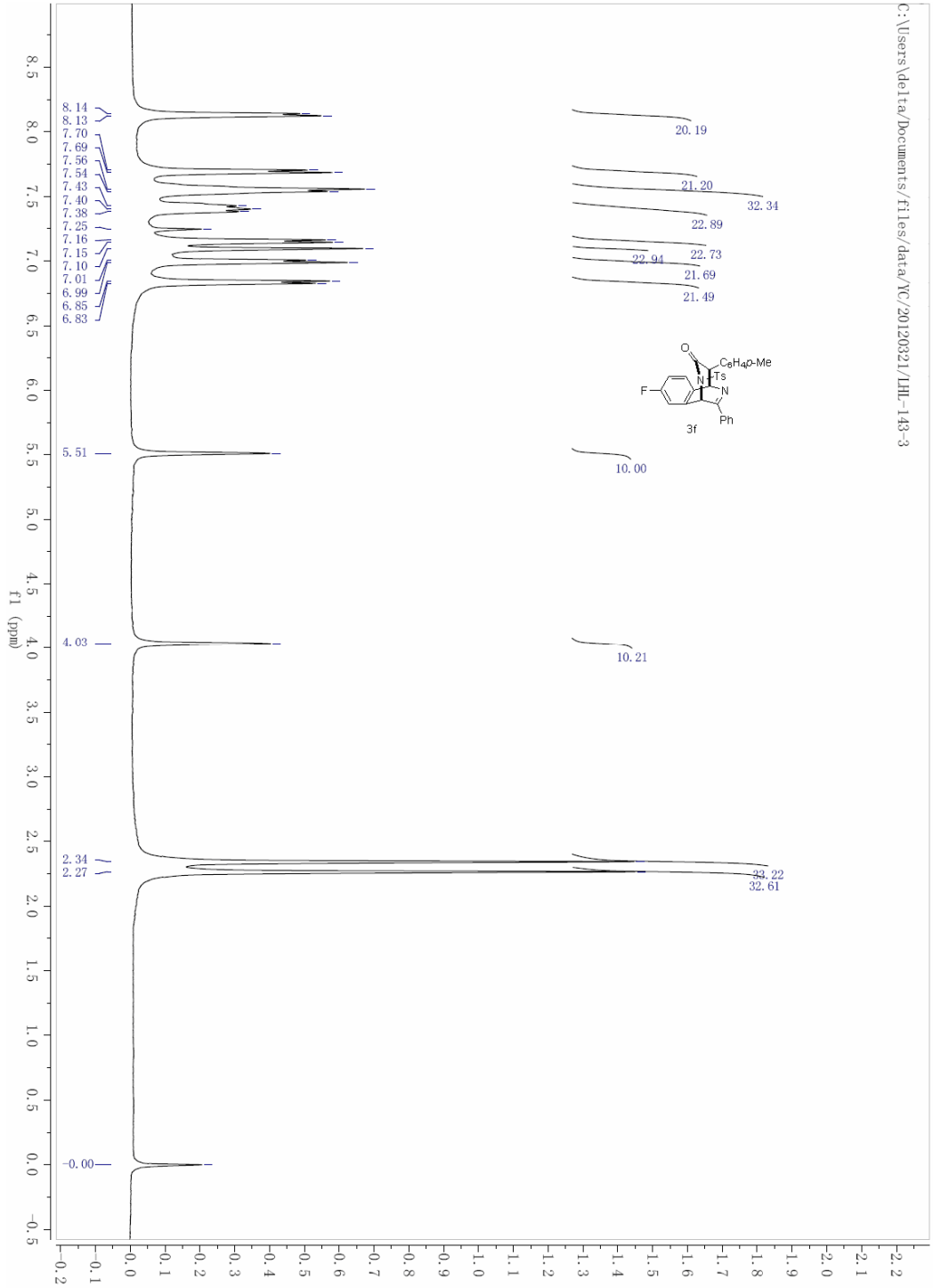
f1 (ppm)
170.6350
169.8056
163.3439
160.8667
145.0103
136.5325
135.6334
131.7417
130.7271
129.0763
129.0448
128.8294
127.5510
127.3217
125.9904
116.0278
115.8119
114.2285
114.0031
62.5033
57.3076
49.9752
21.5069
19.4431
O
C6H4O-Me
Ts
N
N
F
Ph
3e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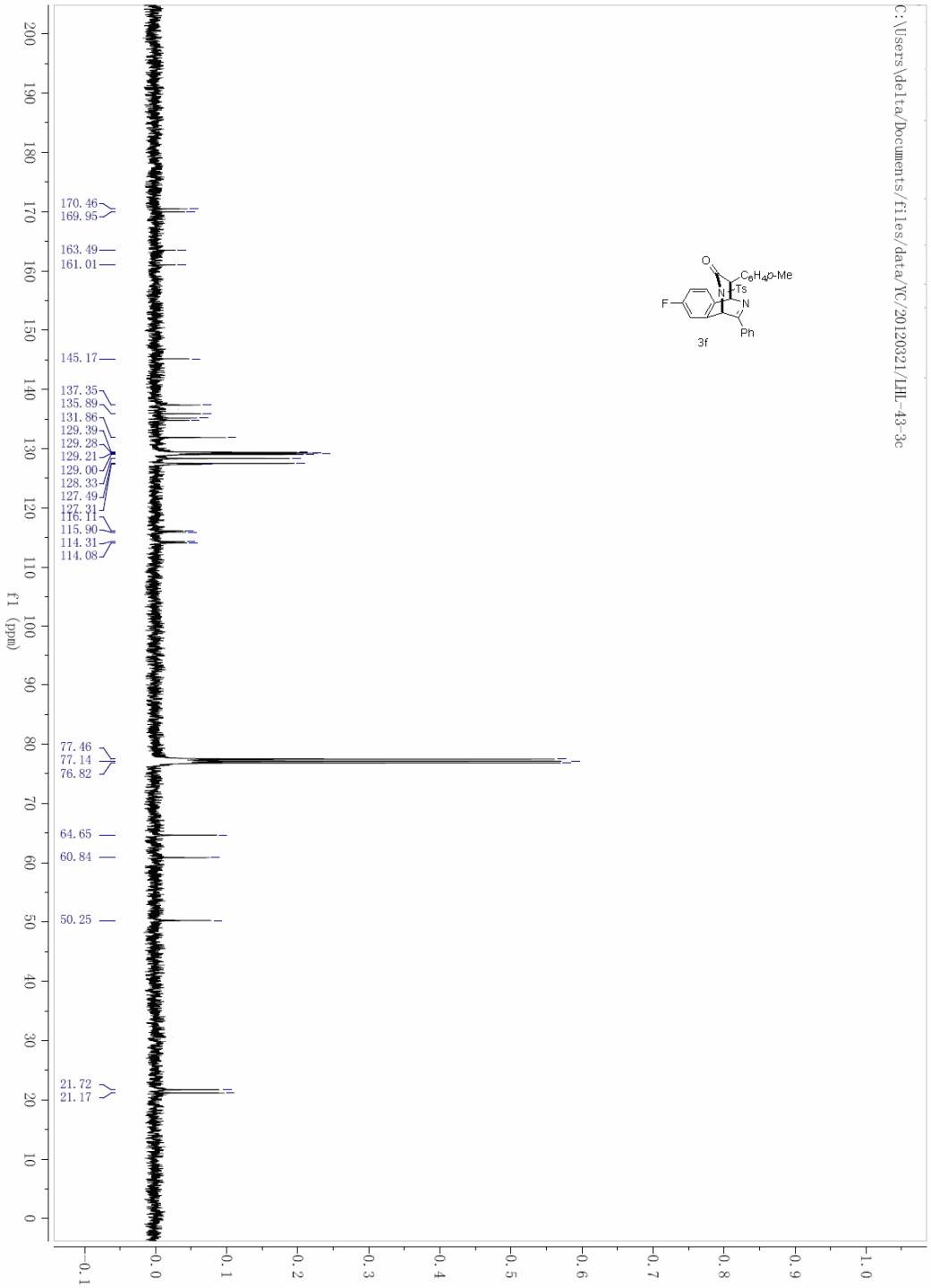
8.14
8.13
7.70
7.69
7.56
7.54
7.43
7.40
7.38
7.25
7.16
7.15
7.10
7.01
6.99
6.85
6.83
5.51
4.03
2.34
2.27
-0.00
20.19
21.20
32.34
22.89
22.94
22.73
21.69
21.49
10.00
10.21
32.22
32.61
O
C6H4p-Me
Ts
N
F
N
Ph
3f
f1 (ppm)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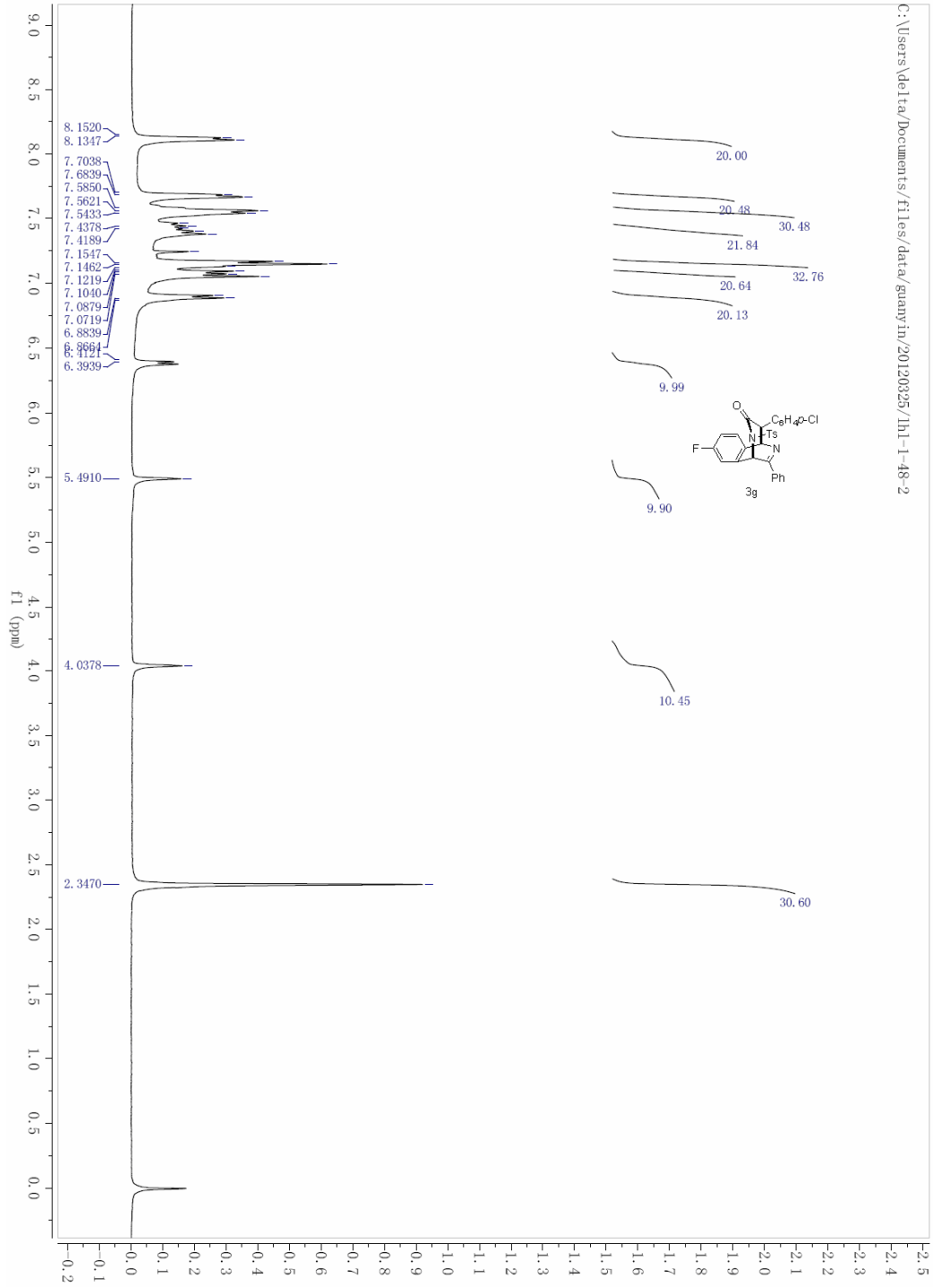
f1 (ppm)
170.46
169.95
163.49
161.01
145.17
137.35
135.89
131.86
129.39
129.28
129.21
129.00
128.33
127.49
127.31
116.11
115.90
114.31
114.08
77.46
77.14
76.82
64.65
60.84
50.25
21.72
21.17
O
C6H4p-Me
Ts
N
F
N
Ph
3f


C:\Users\delta\Documents\files\data\guanyin\20120325\lhl-1-48-2
f1 (ppm)
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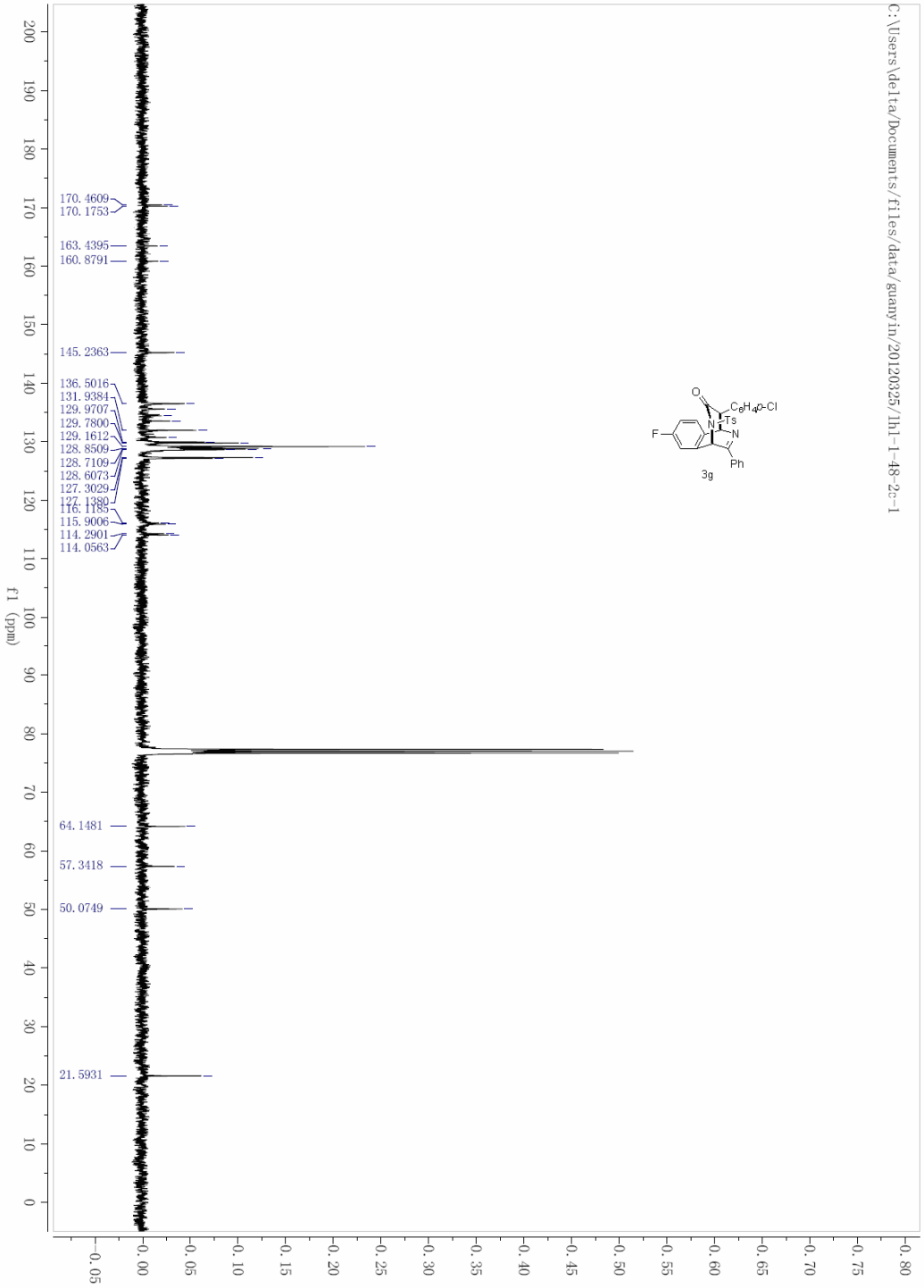
8.1347
7.7038
7.6839
7.5850
7.5621
7.5433
7.4378
7.4189
7.1547
7.1462
7.1219
7.1040
7.0879
7.0719
6.8839
6.8664
6.4121
6.3939
5.4910
4.0378
2.3470
20.00
20.48
30.48
21.84
32.76
20.64
20.13
9.99
9.90
10.45
30.60
O
C6H4o-Cl
Ts
N
N
F
Ph
3g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f1 (ppm)
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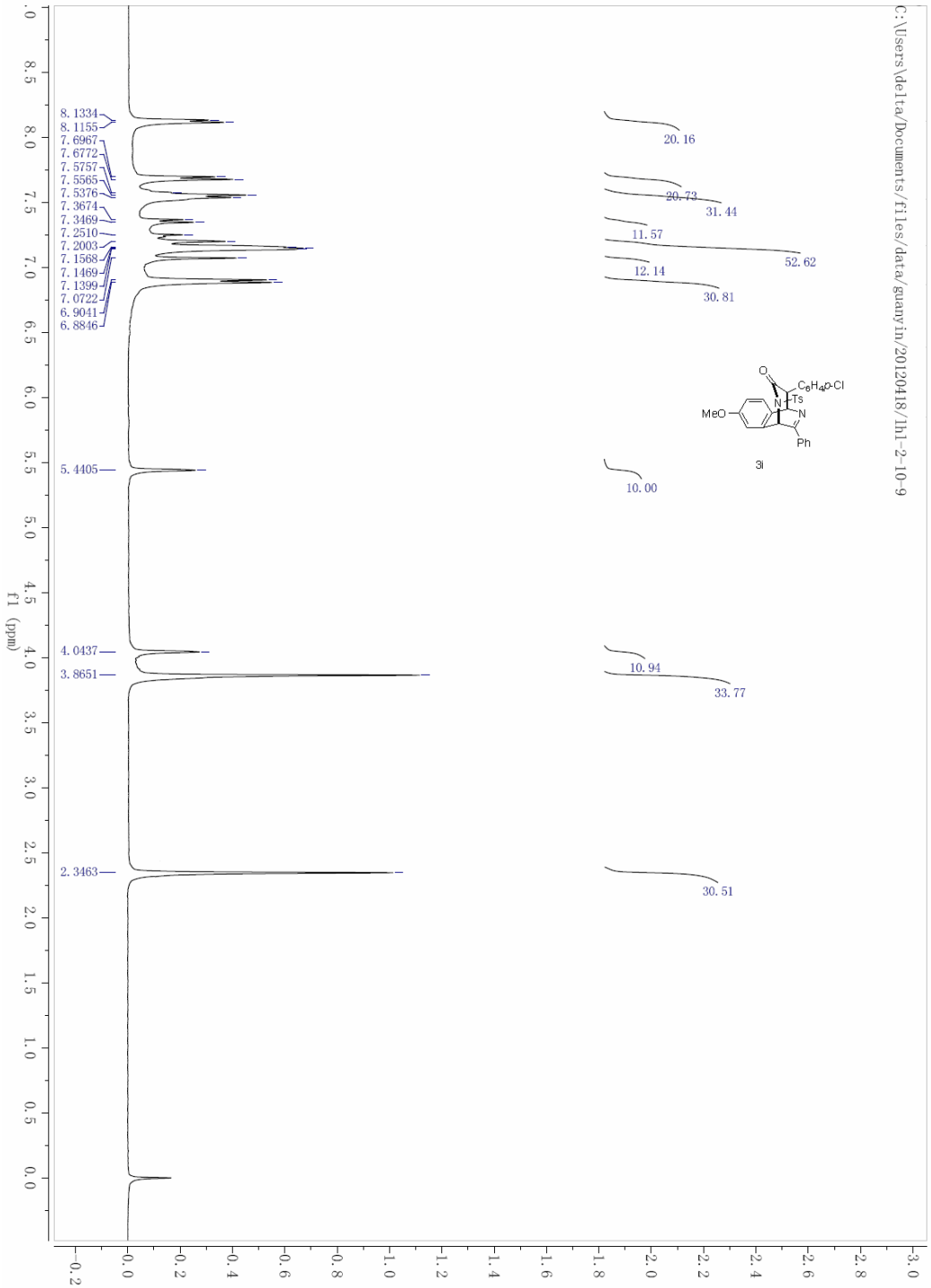
170.1753
163.4395
160.8791
145.2363
136.5016
131.9384
129.9707
129.7800
129.1612
128.8509
128.7109
128.6073
127.3029
127.1380
116.1185
115.9006
114.2901
114.0563
64.1481
57.3418
50.0749
21.5931
O
C6H4p-Cl
Ts
N
F
N
Ph
3g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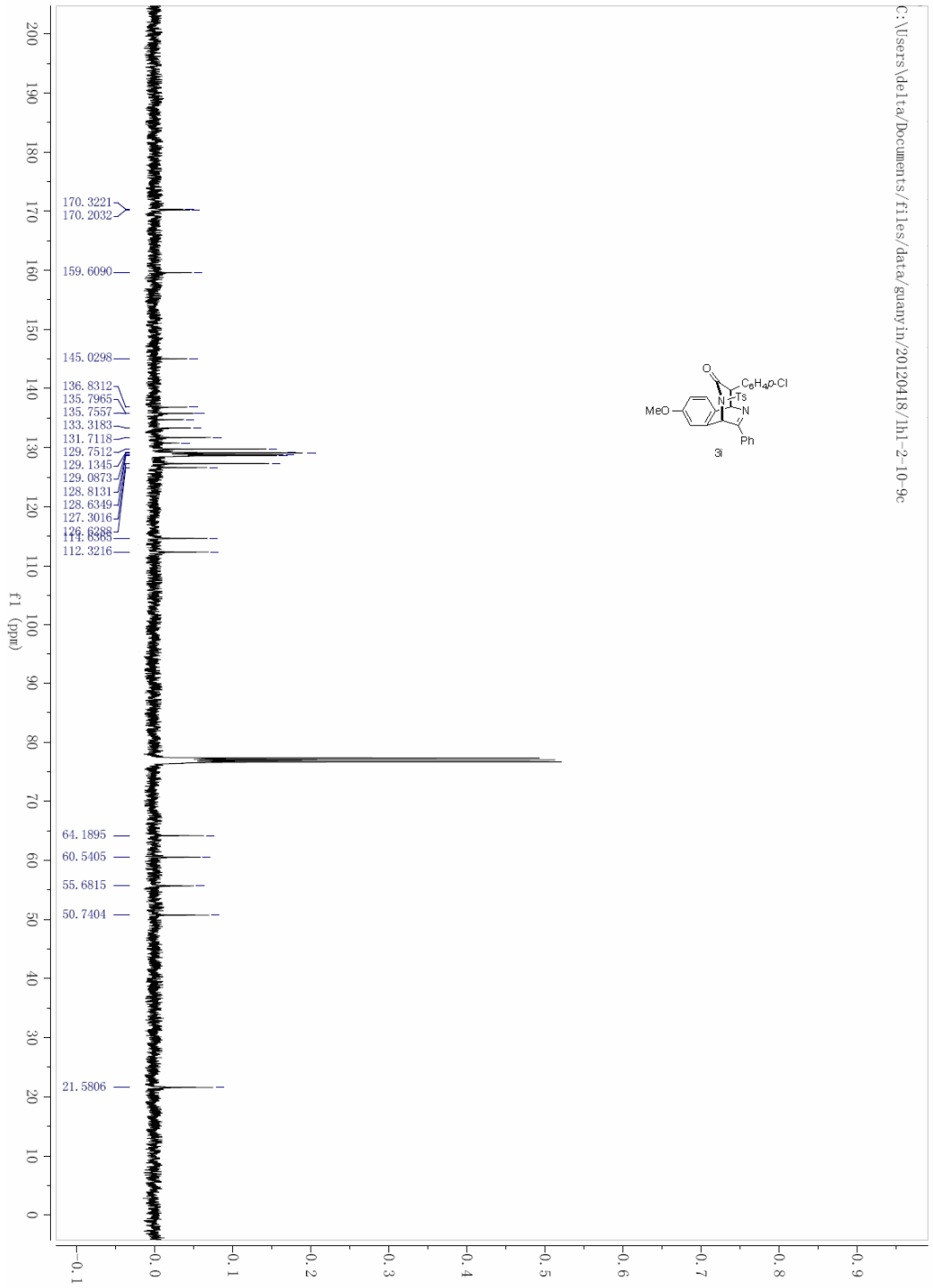
7.6772
7.5757
7.5565
7.5376
7.3674
7.3469
7.2510
7.2003
7.1568
7.1469
7.1399
7.0722
6.9041
6.8846
5.4405
4.0437
3.8651
2.3463
20.16
20.73
31.44
11.57
52.62
12.14
30.81
10.00
10.94
33.77
30.51
C6H4-2-Cl
Ts
N
N
MeO
O
Ph
3l
f1 (ppm)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f1 (ppm)
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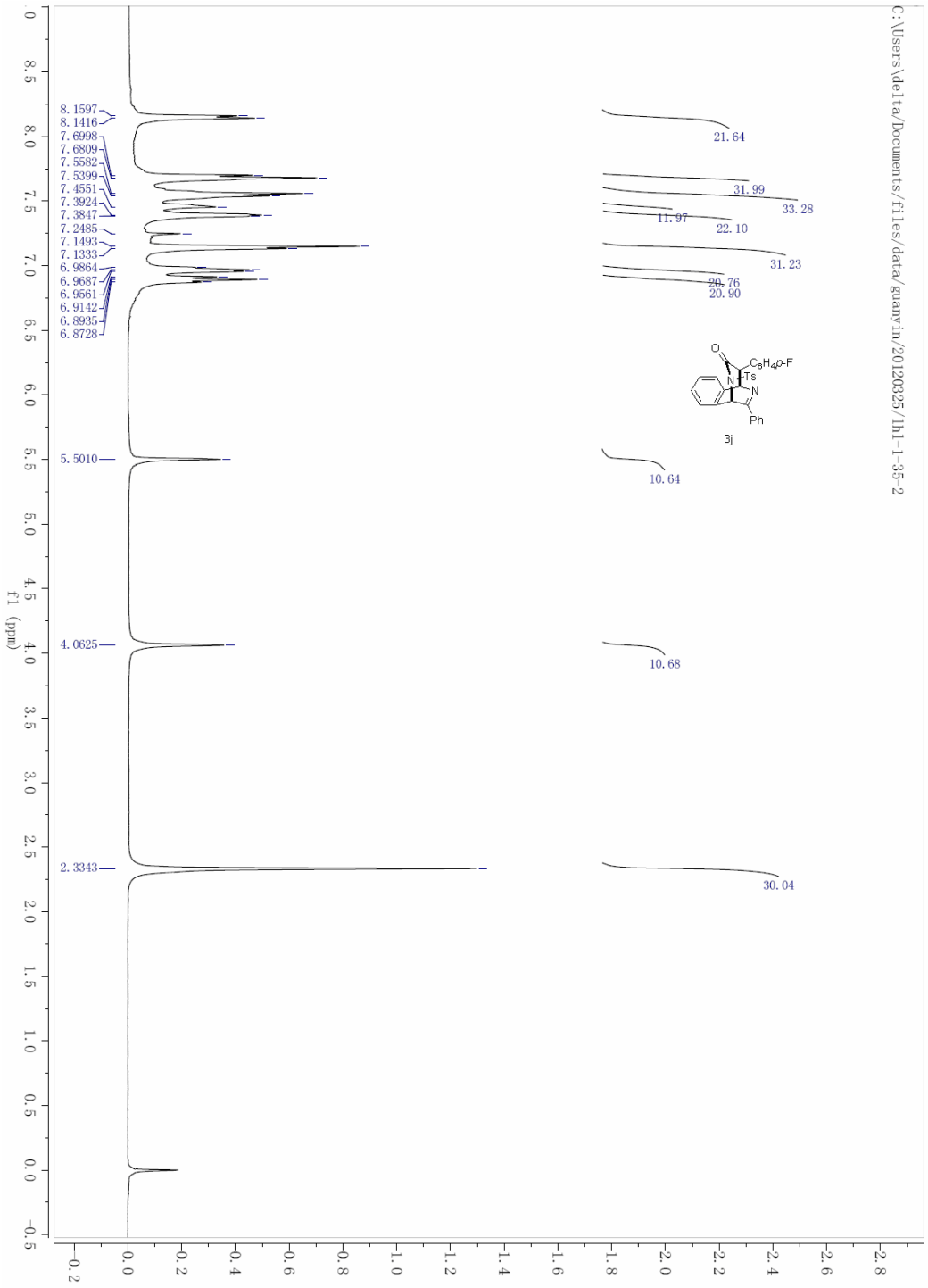
170.2032
159.6090
145.0298
136.8312
135.7965
135.7557
133.3183
131.7118
129.7512
129.1345
129.0873
128.8131
128.6349
127.3016
126.6288
114.6385
112.3216
64.1895
60.5405
55.6815
50.7404
21.5806
MeO
Ts
N
Ph
C6H4o-Cl
3l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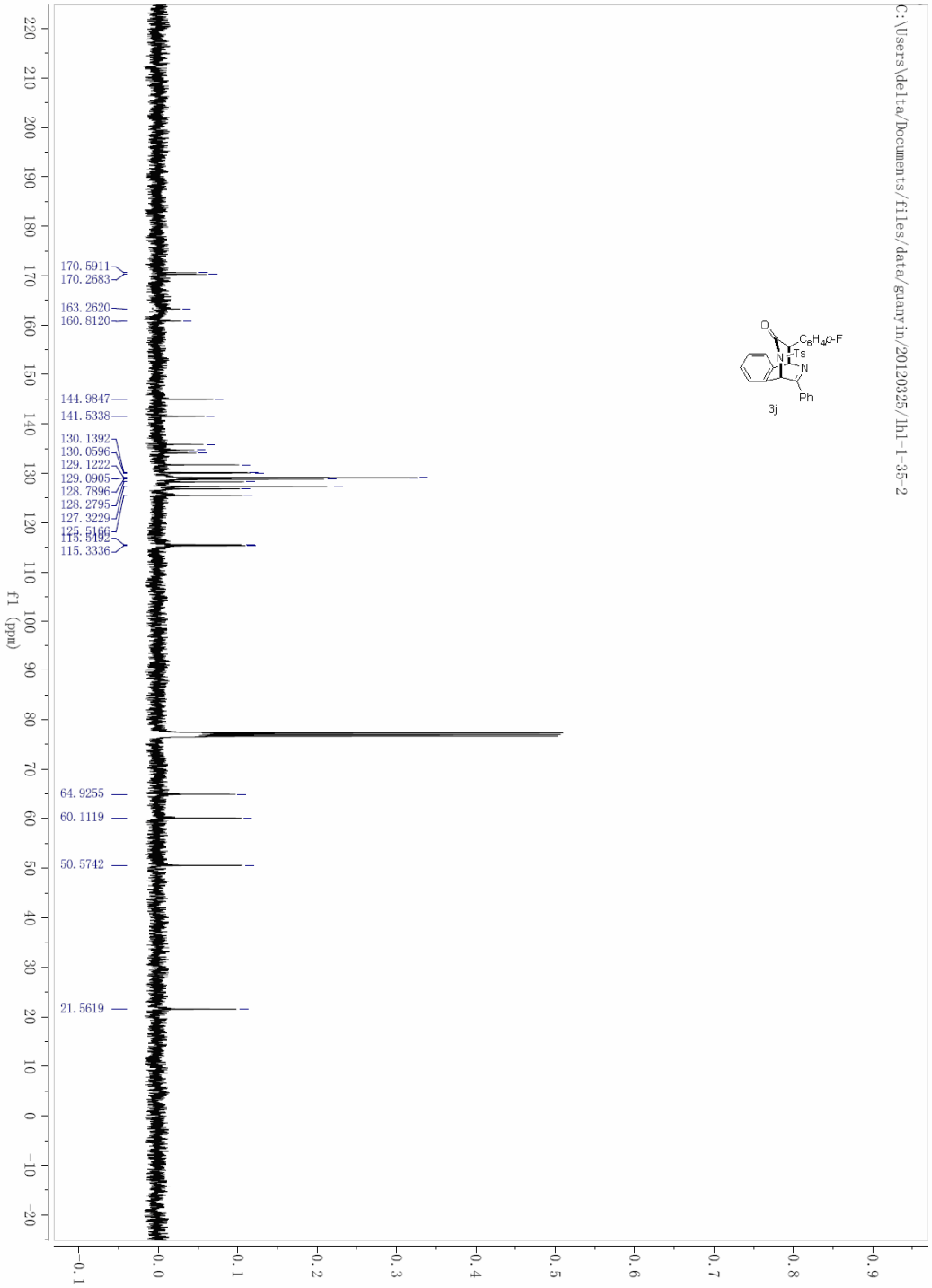
8.1416
7.6998
7.6809
7.5582
7.5399
7.4551
7.3924
7.3847
7.2485
7.1493
7.1333
6.9864
6.9687
6.9561
6.9142
6.8935
6.8728
5.5010
4.0625
2.3343
21.64
31.99
33.28
11.97
22.10
31.23
20.76
20.90
10.64
10.68
30.04
O
C6H4p-F
N
Ts
N
Ph
3j
f1 (ppm)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170.2683
163.2620
160.8120
144.9847
141.5338
130.1392
130.0596
129.1222
129.0905
128.7896
128.2795
127.3229
125.5166
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115.3336
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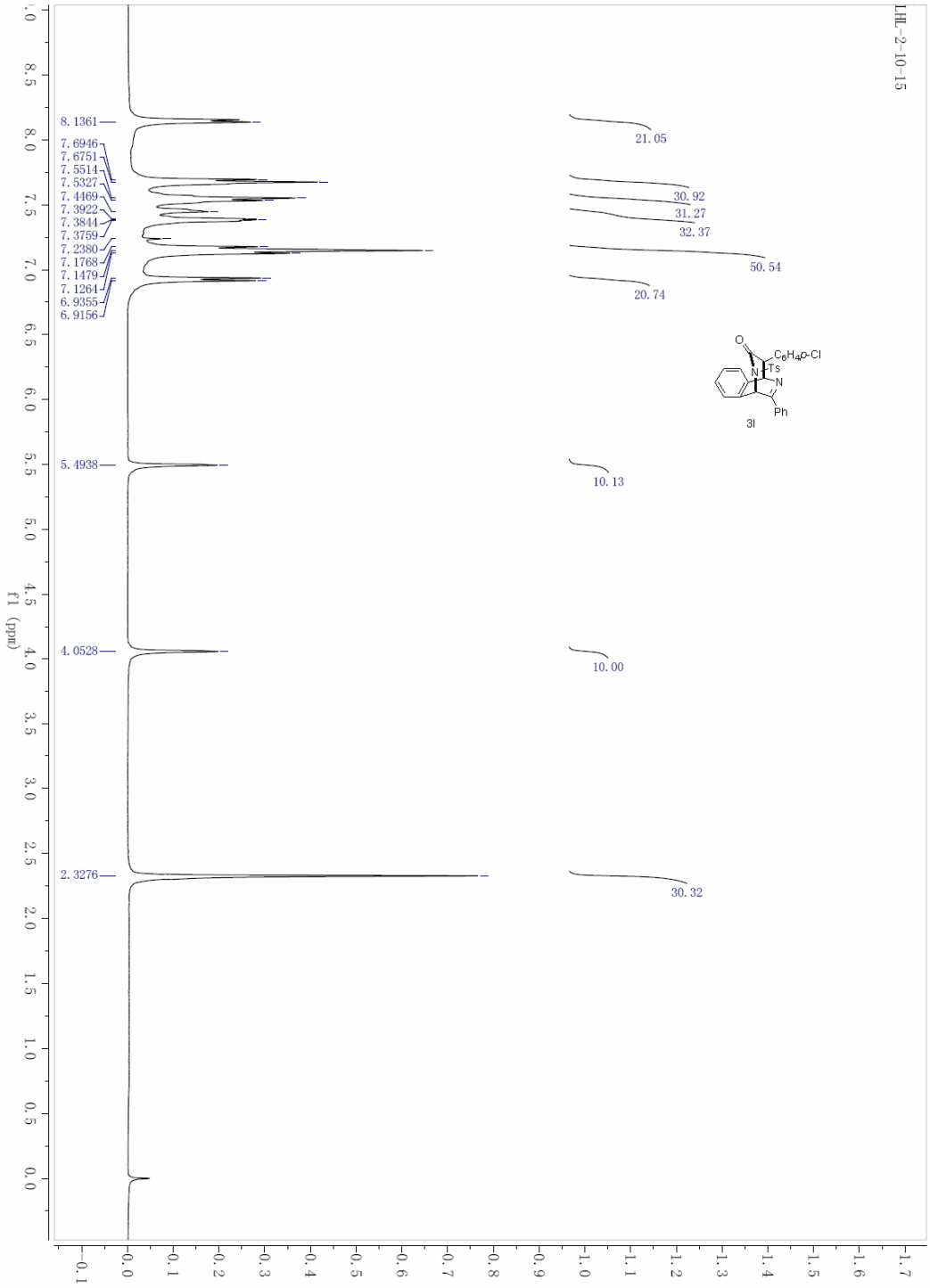
60.1119
50.5742
21.5619
f1 (ppm)
3j
Ph
Ts
N
O
F


LHL-2-10-15
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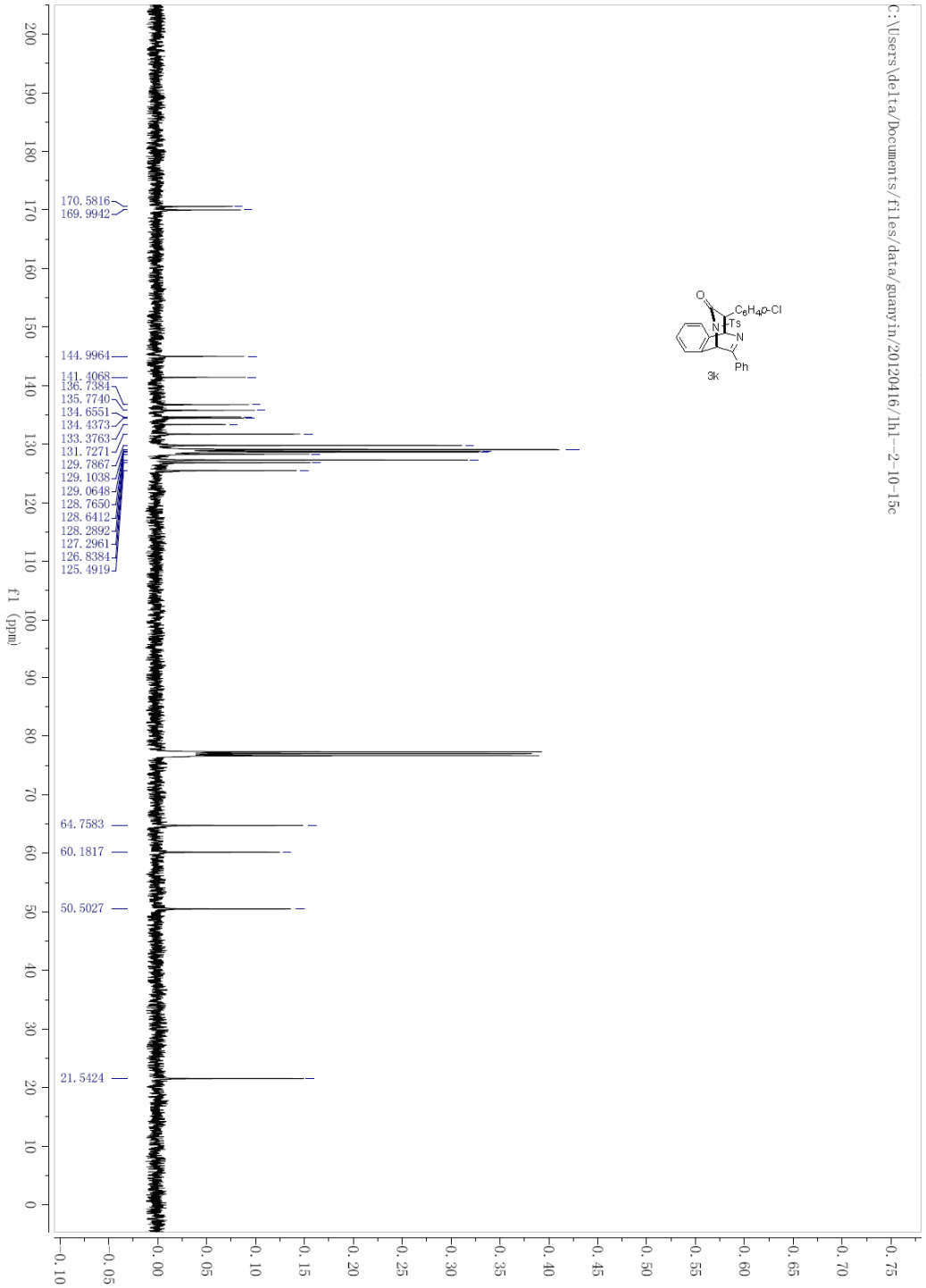
7.3922
7.3844
7.3759
7.2380
7.1768
7.1479
7.1264
6.9355
6.9156
5.4938
4.0528
2.3276
21.05
30.92
31.27
32.37
50.54
20.74
10.13
10.00
30.32
O
C6H4p-Cl
Ts
N
N
Ph
3l
f1 (ppm)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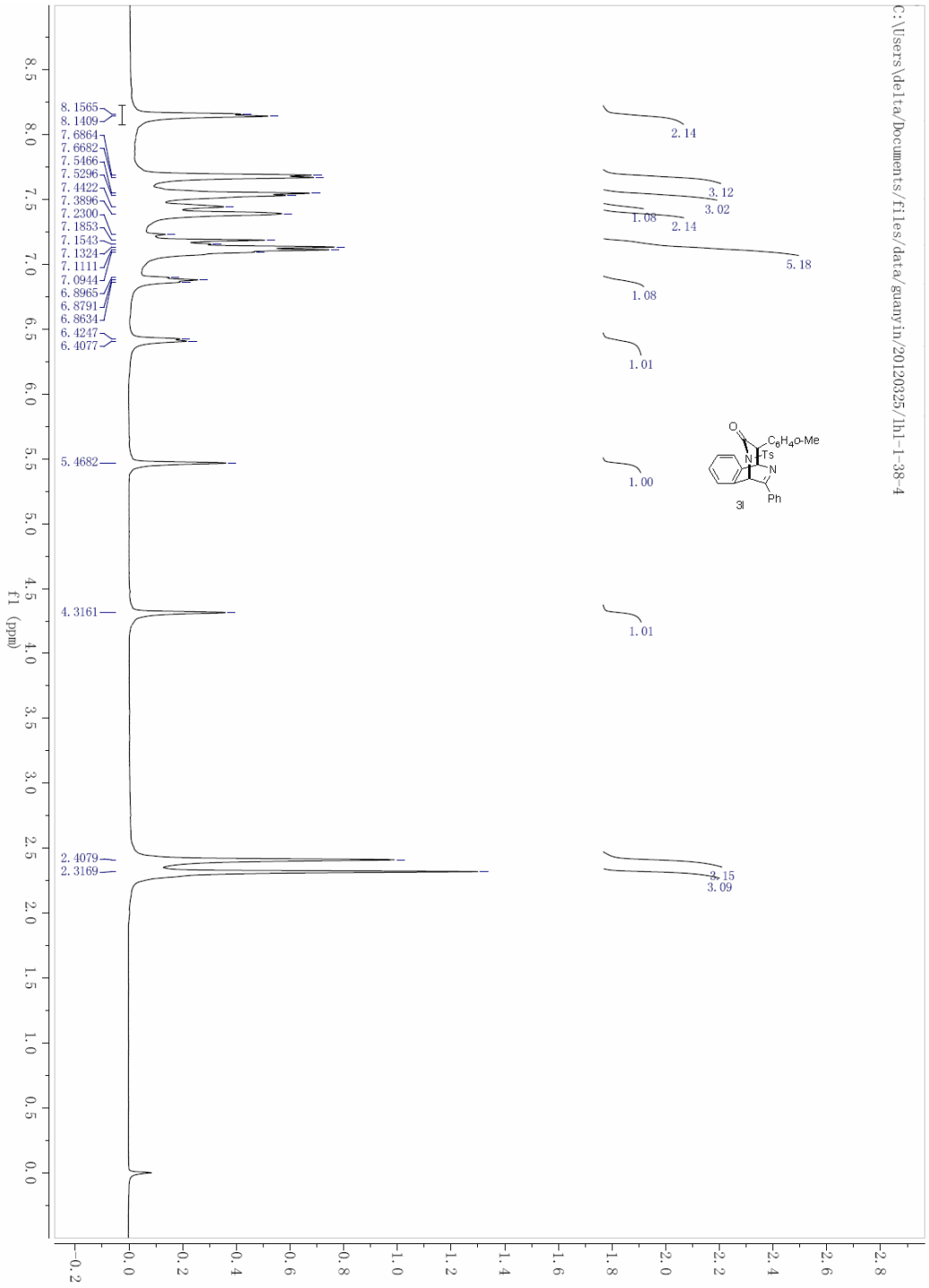
169.9942
144.9964
141.4068
136.7384
135.7740
134.6551
134.4373
133.3763
131.7271
129.7867
129.1038
129.0648
128.7650
128.6412
128.2892
127.2961
126.8384
125.4919
64.7583
60.1817
50.5027
21.5424
f1 (ppm)
O
C6H4p-Cl
Ts
N
N
Ph
3k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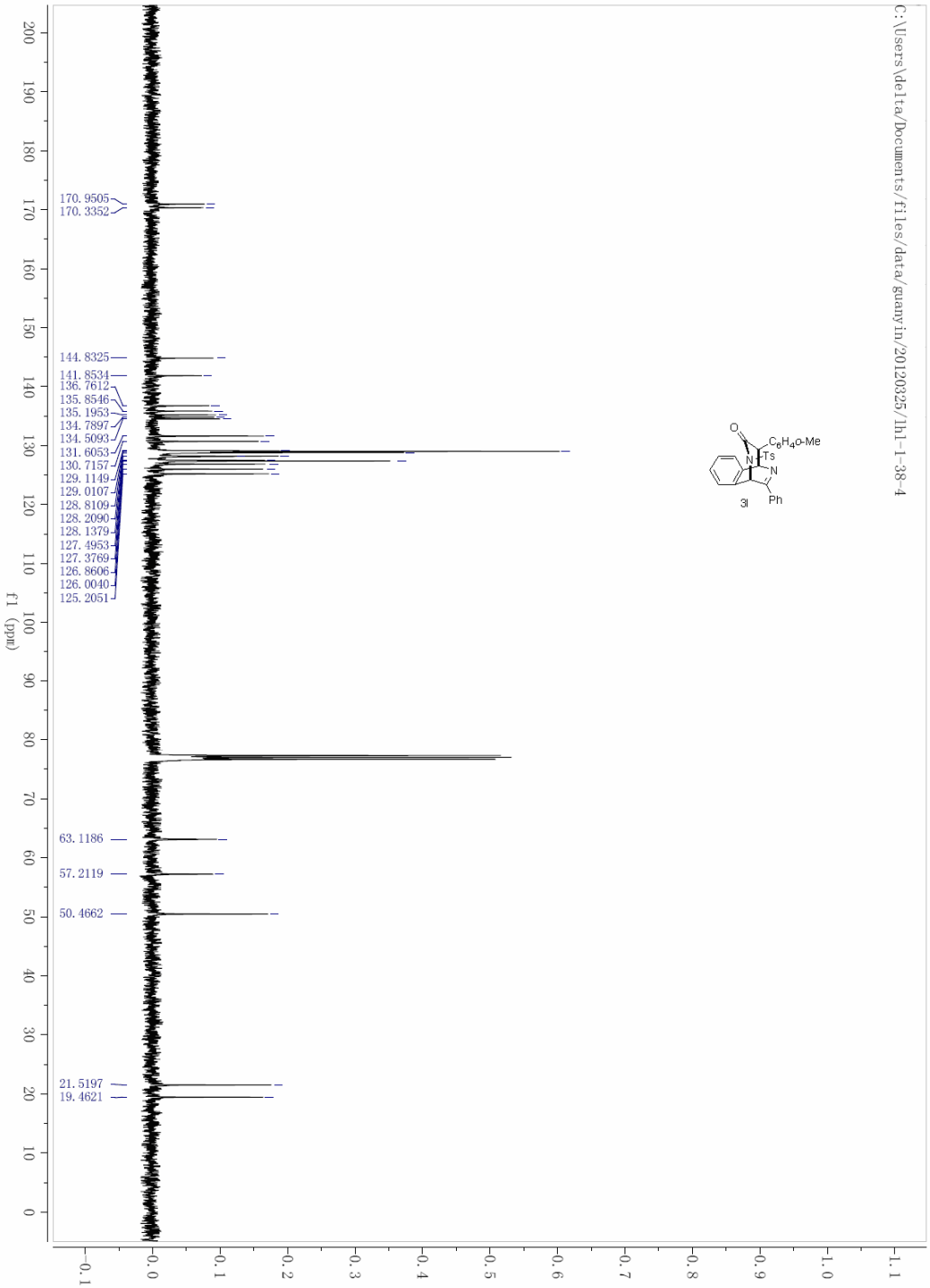
7.6682
7.5466
7.5296
7.4422
7.3896
7.2300
7.1853
7.1543
7.1324
7.1111
7.0944
6.8965
6.8791
6.8634
6.4247
6.4077
5.4682
4.3161
2.4079
2.3169
2.14
3.12
3.02
1.08
2.14
5.18
1.08
1.01
1.00
1.01
3.15
3.09
C6H4o-Me
Ts
N
N
Ph
3l
f1 (ppm)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f1 (ppm)
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170.3352
144.8325
141.8534
136.7612
135.8546
135.1953
134.7897
134.5093
131.6053
130.7157
129.1149
129.0107
128.8109
128.2090
128.1379
127.4953
127.3769
126.8606
126.0040
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57.2119
50.4662
21.5197
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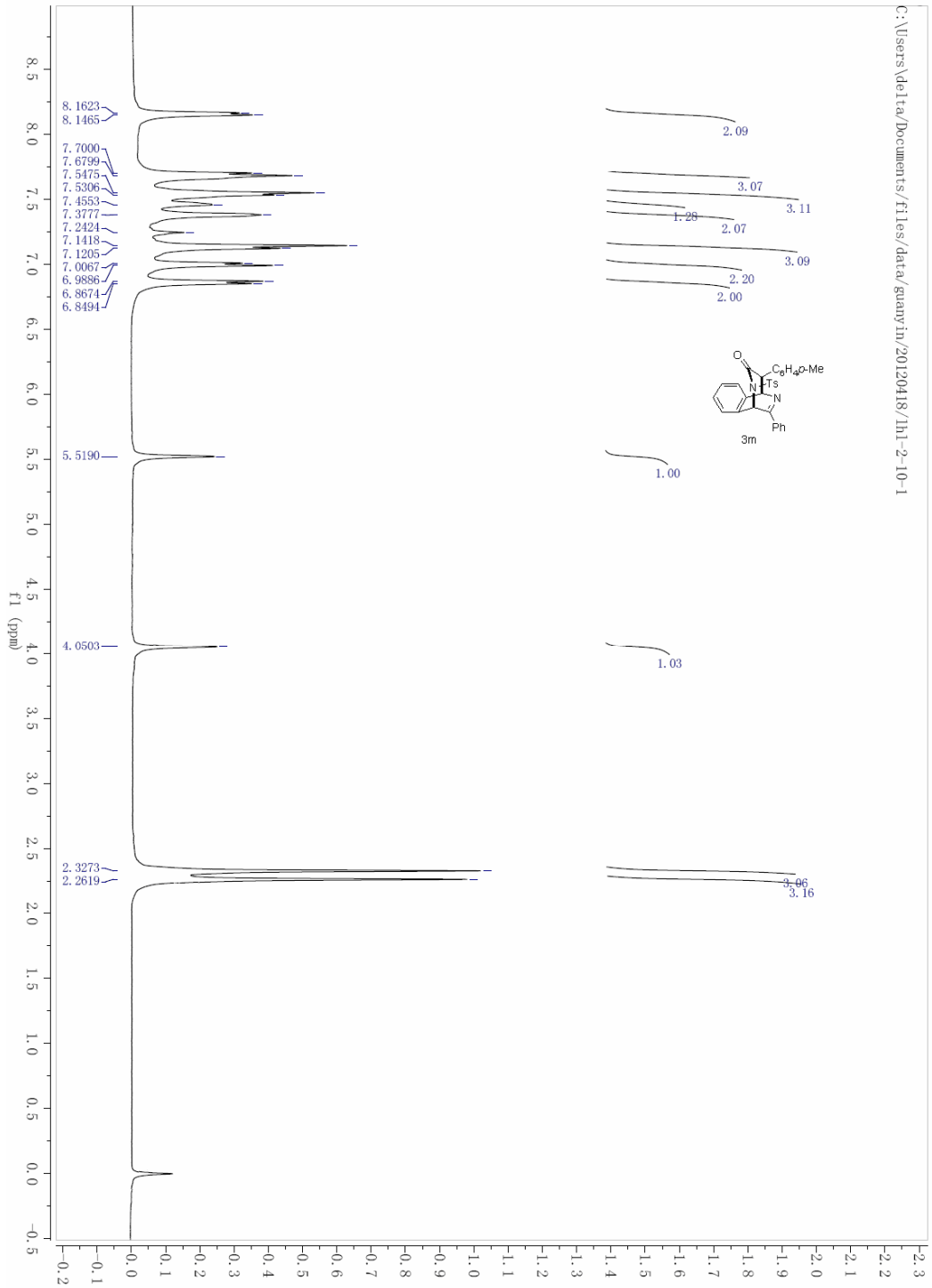
C6H4o-Me
Ts
N
N
Ph
3l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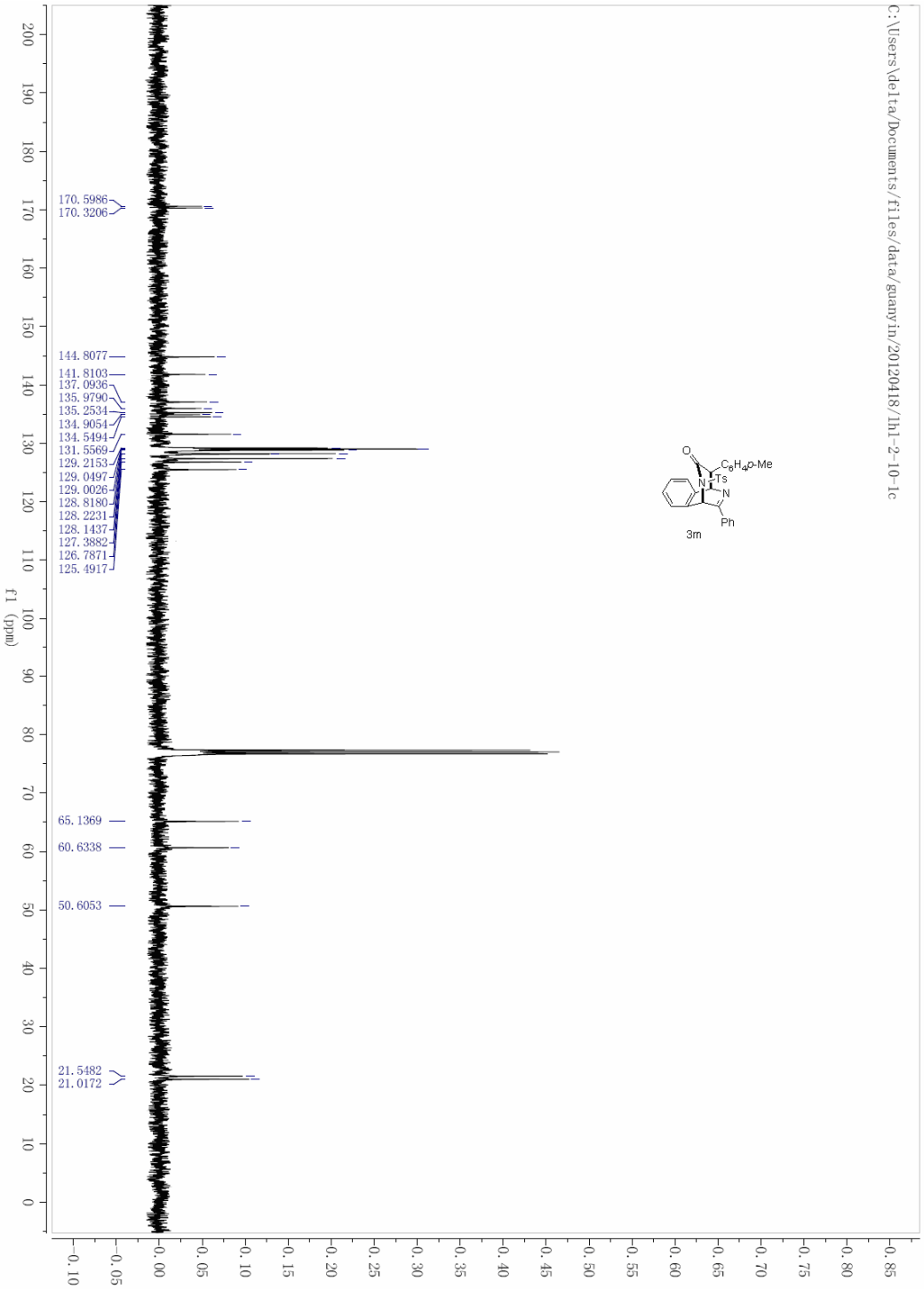
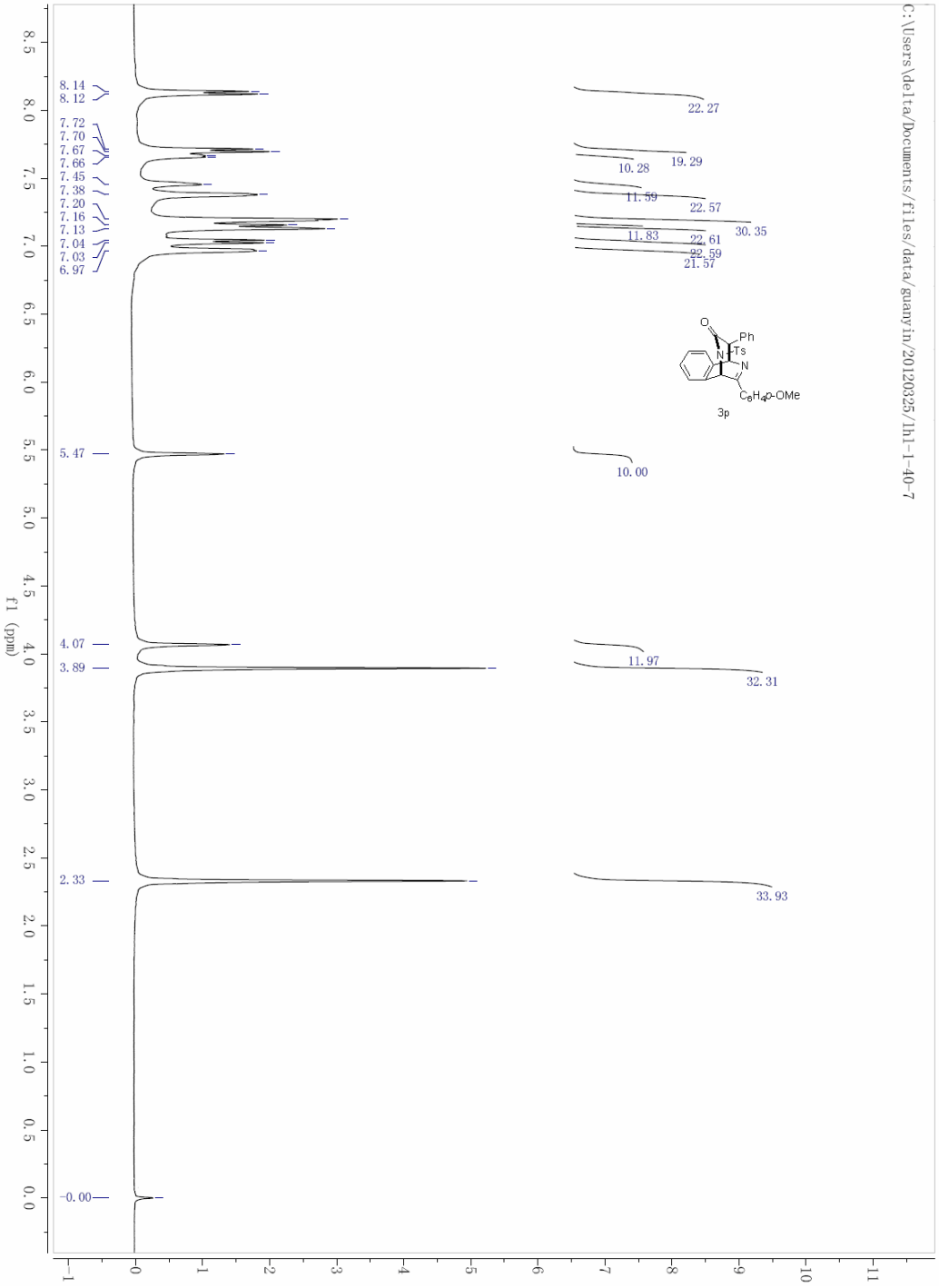
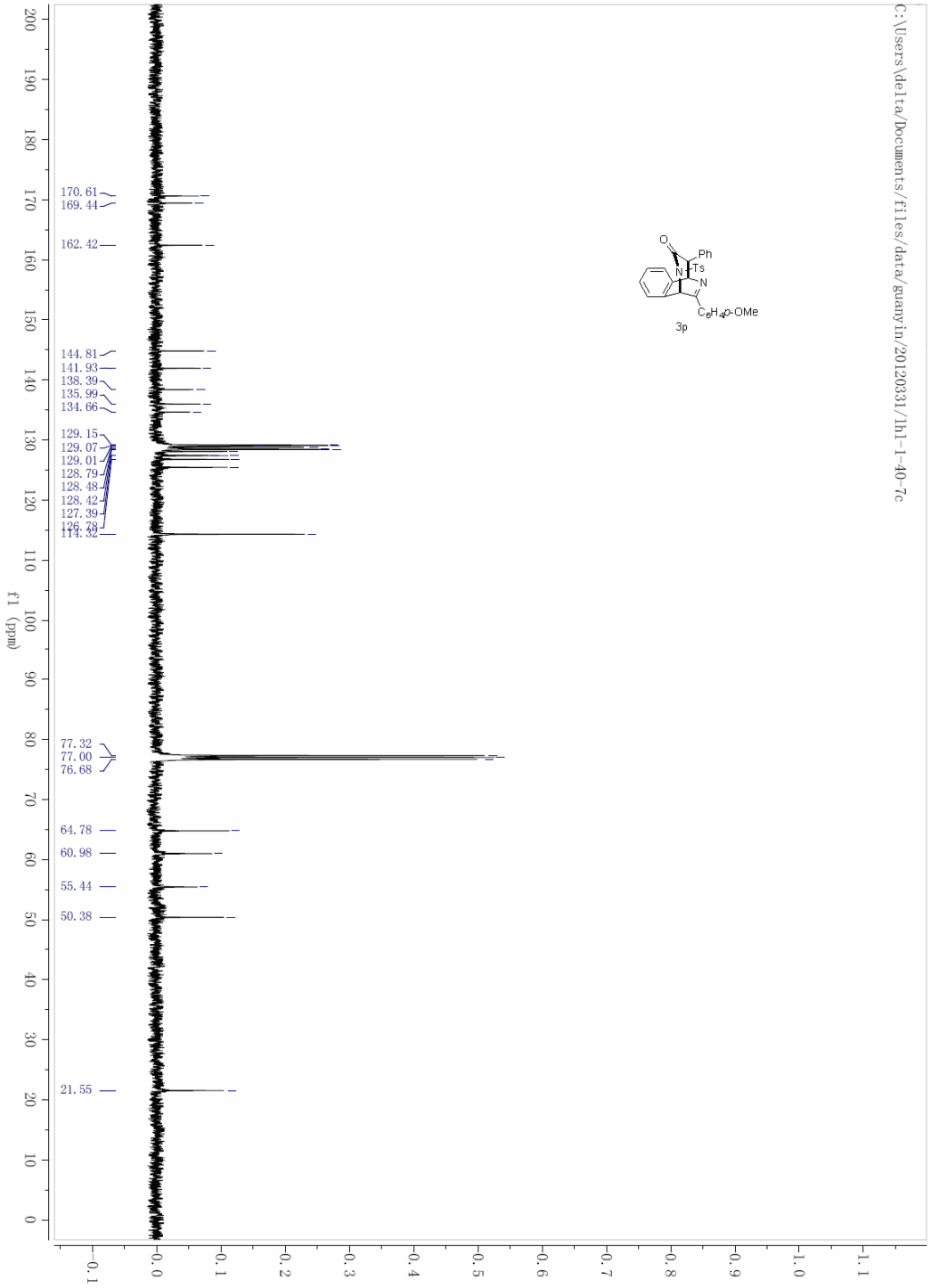
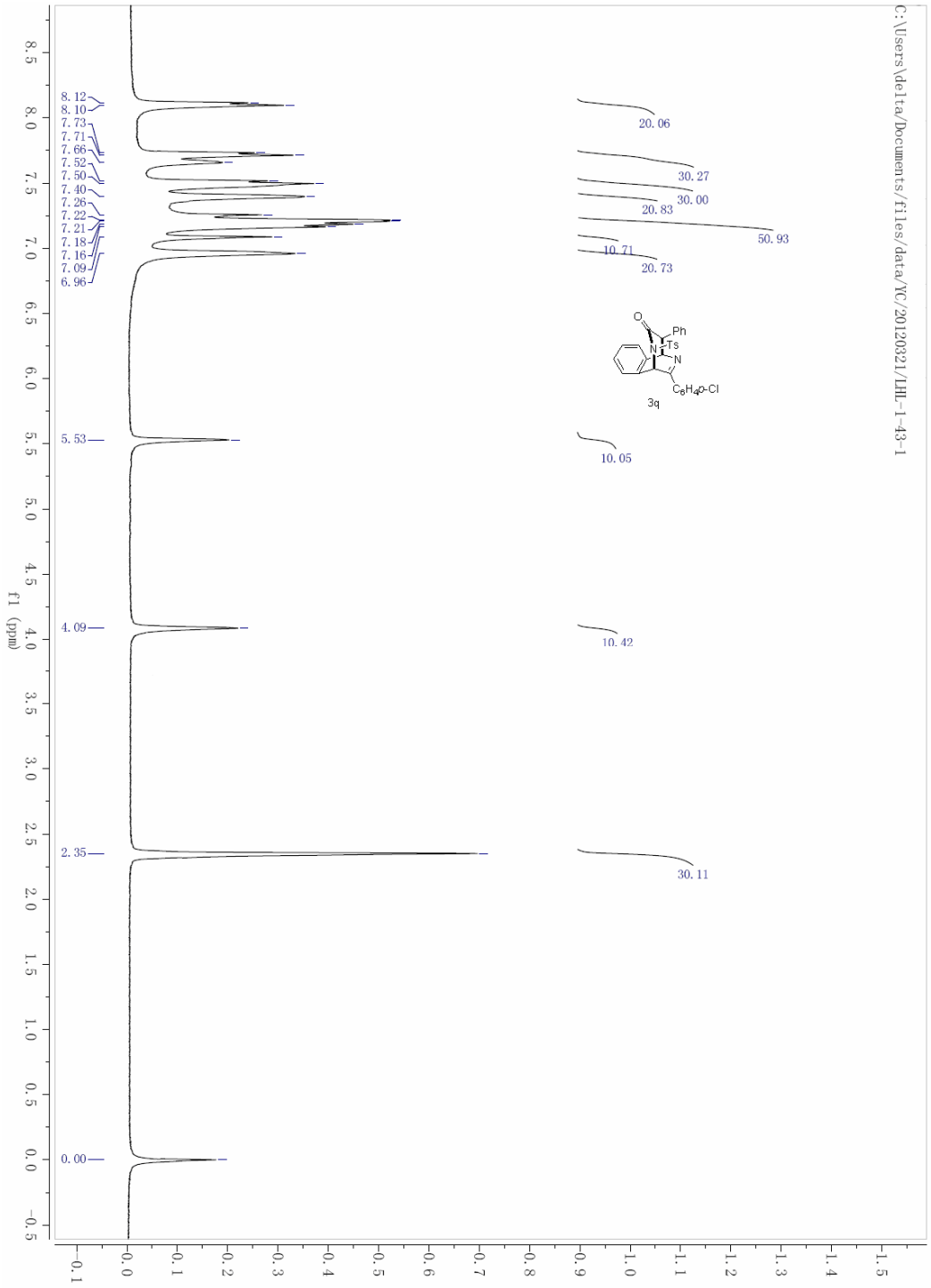
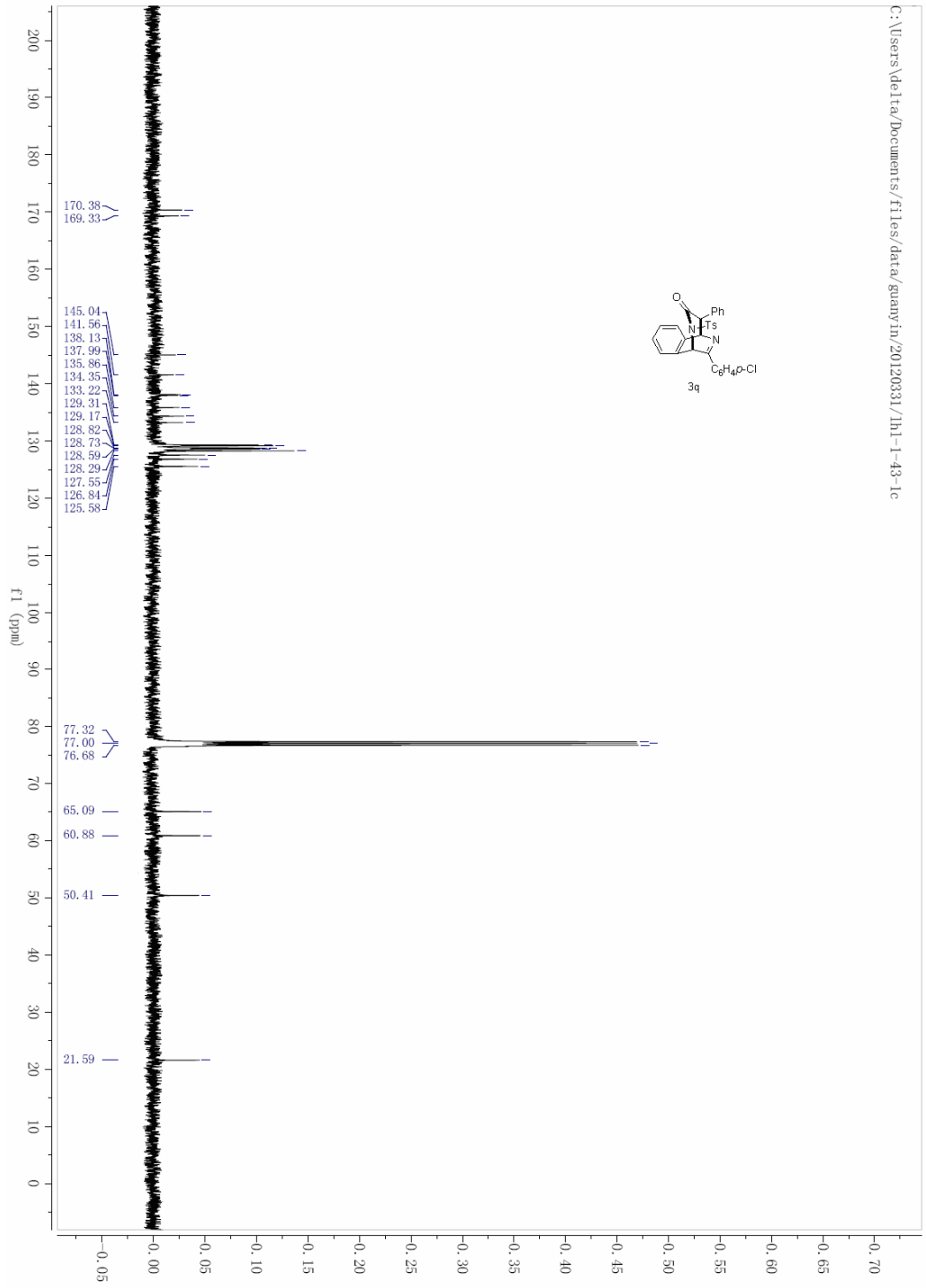
7.5475
7.5306
7.4553
7.3777
7.2424
7.1418
7.1205
7.0067
6.9886
6.8674
6.8494
5.5190
4.0503
2.3273
2.2619
2.09
3.07
3.11
1.28
2.07
3.09
2.20
2.00
1.00
1.03
3.06
3.16
O
C6H4p-Me
Ts
N
N
Ph
3m
f1 (ppm)

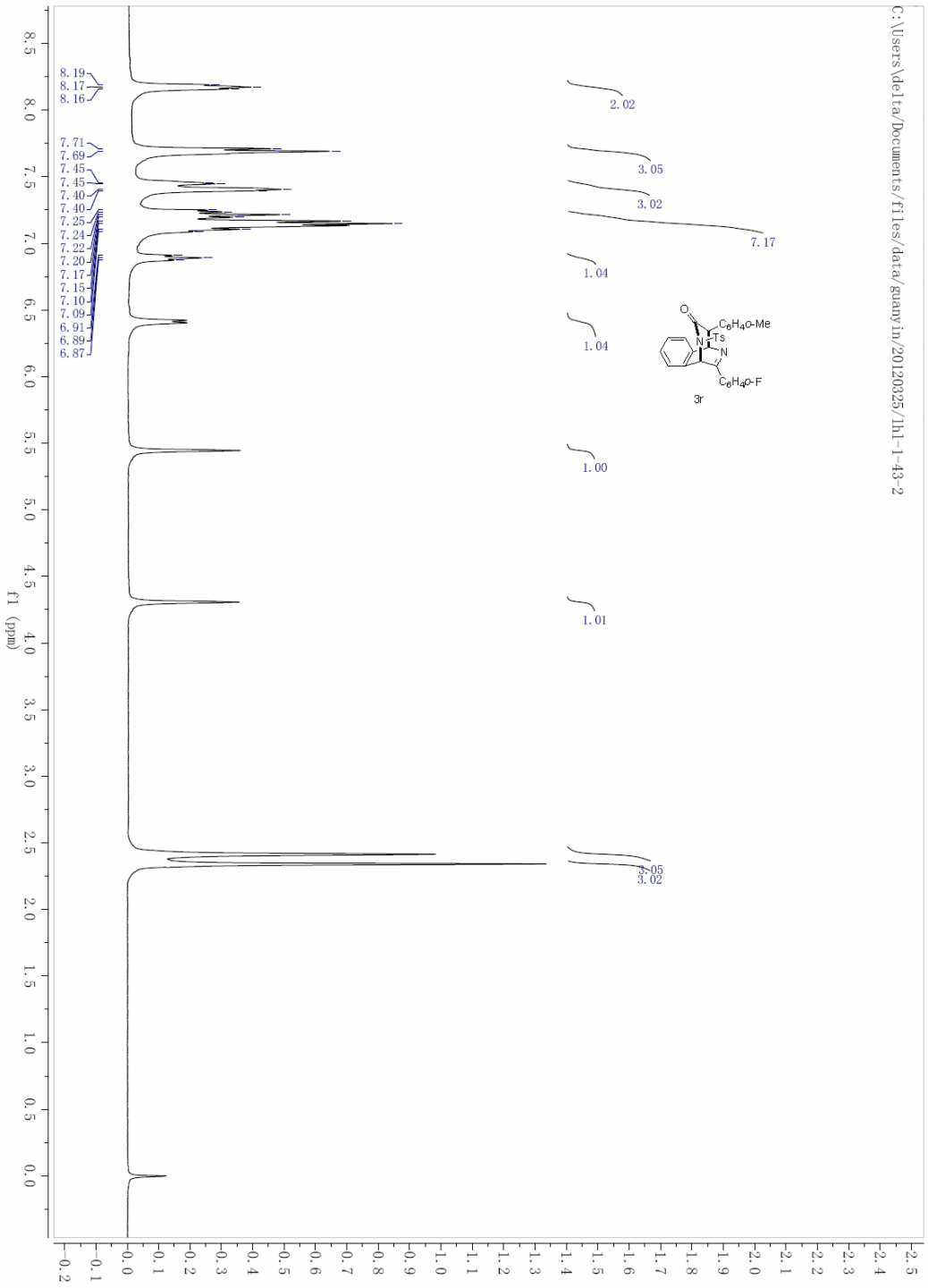

C:\Users\delta\Documents\files\data\guanyin\20120418\lhl-2-10-1c
f1 (ppm)
170.5986
170.3206
144.8077
141.8103
137.0936
135.9790
135.2534
134.9054
134.5494
131.5569
129.2153
129.0497
129.0026
128.8180
128.2231
128.1437
127.3882
126.7871
125.4917
65.1369
60.6338
50.6053
21.5482
21.0172
C6H4p-Me
Ts
N
N
O
Ph
3m

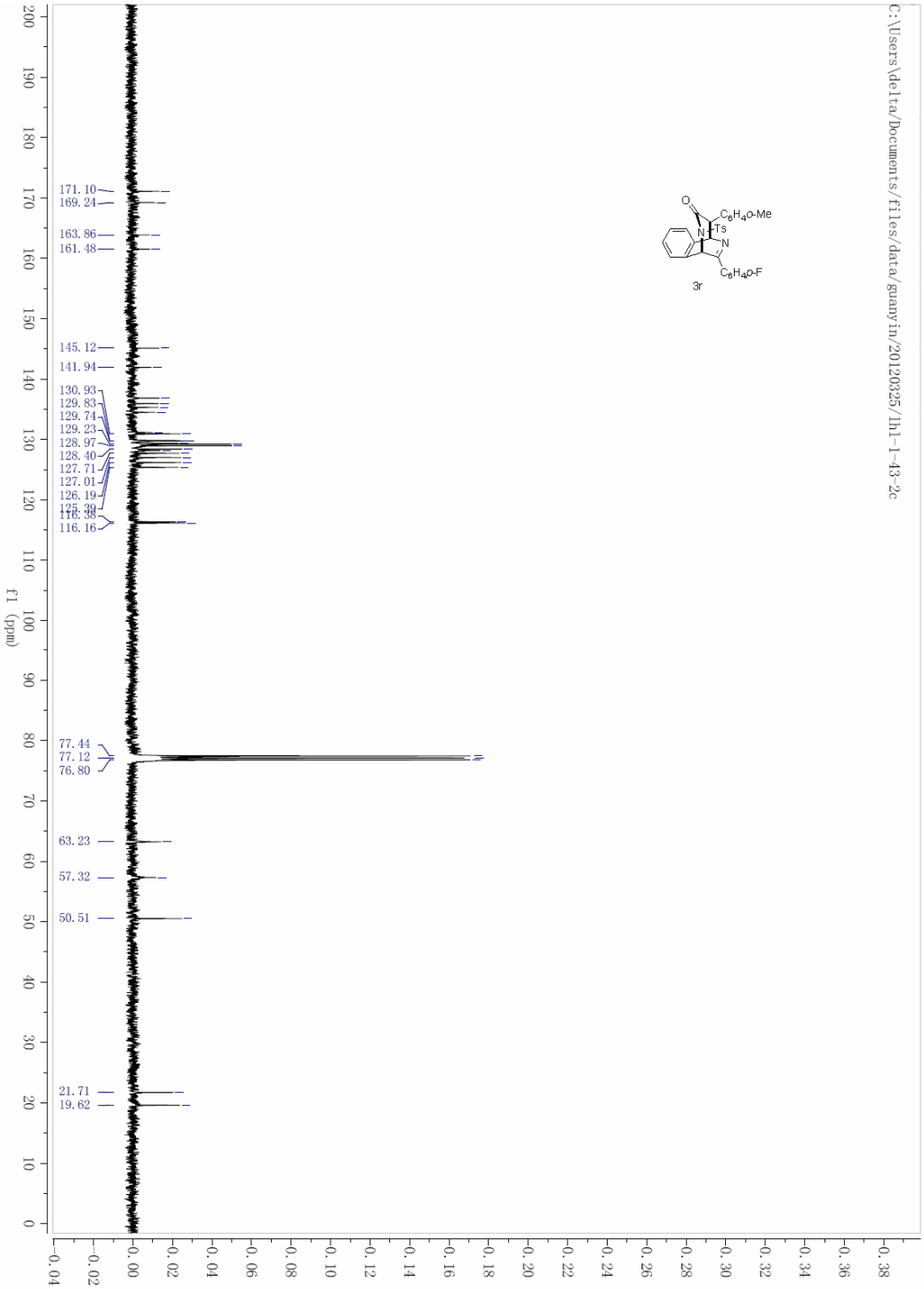

C:\Users\delta\Documents\files\data\guanyin\20120325\lh1-1-40-7
f1 (ppm)
8.14
8.12
7.72
7.70
7.67
7.66
7.45
7.38
7.20
7.16
7.13
7.04
7.03
6.97
5.47
4.07
3.89
2.33
-0.00
22.27
10.28
19.29
11.59
22.57
30.35
11.83
22.61
22.59
21.57
10.00
11.97
32.31
33.93
O
Ph
Ts
N
N
C6H4O-OMe
3p


C:\Users\delta\Documents\files\data\guanyin\20120331\lh1-1-40-7c
f1 (ppm)
170.61
169.44
162.42
144.81
141.93
138.39
135.99
134.66
129.15
129.07
129.01
128.79
128.48
128.42
127.39
77.32
77.00
76.68
64.78
60.98
55.44
50.38
21.55
Ph
Ts
N
C6H4p-OMe
3p


C:\Users\delta\Documents\files\data\YC\20120321\LHL-1-43-1
8.12
8.10
7.73
7.71
7.66
7.52
7.50
7.40
7.26
7.22
7.21
7.18
7.16
7.09
6.96
5.53
4.09
2.35
0.00
20.06
30.27
30.00
20.83
50.93
10.71
20.73
10.05
10.42
30.11
Ph
Ts
N
N
C6H4o-Cl
3q
f1 (ppm)


C:\Users\delta\Documents\files\data\guanyin\20120331\hl-1-43-1c
f1 (ppm)
170.38
169.33
145.04
141.56
138.13
137.99
135.86
134.35
133.22
129.31
129.17
128.82
128.73
128.59
128.29
127.55
126.84
125.58
77.32
77.00
76.68
65.09
60.88
50.41
21.59
O
Ph
Ts
N
N
C6H4p-Cl
3q


C:\Users\delta\Documents\files\data\guanyin\20120325\lhl-1-43-2
8.19
8.17
8.16
7.71
7.69
7.45
7.45
7.40
7.40
7.25
7.24
7.22
7.20
7.17
7.15
7.10
7.09
6.91
6.89
6.87
2.02
3.05
3.02
7.17
1.04
1.04
1.00
1.01
3.05
3.02
C6H4O-Me
Ts
N
N
C6H4O-F
3r
f1 (ppm)


C:\Users\delta\Documents\files\data\guanyin\20120325\lh1-1-43-2c
171.10
169.24
163.86
161.48
145.12
141.94
130.93
129.83
129.74
129.23
128.97
128.40
127.71
127.01
126.19
125.39
116.38
116.16
77.44
77.12
76.80
63.23
57.32
50.51
21.71
19.62
f1 (ppm)
C6H4o-Me
Ts
N
N
C6H4o-F
3r

checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: a20309a

```
Bond precision:  C-C = 0.0039 A          Wavelength=0.71073

Cell:            a=14.328(7)     b=11.798(6)          c=16.315(8)
                 alpha=90        beta=116.046(4)      gamma=90
Temperature:     293 K
```

	Calculated	Reported
Volume	2478(2)	2478(2)
Space group	P 21/c	P2(1)/c
Hall group	-P 2ybc	?
Moiety formula	C30 H24 N2 O3 S	?
Sum formula	C30 H24 N2 O3 S	C30 H24 N2 O3 S
Mr	492.58	492.57
Dx,g cm-3	1.320	1.320
Z	4	4
Mu (mm-1)	0.166	0.166
F000	1032.0	1032.0
F000’	1032.92	
h,k,lmax	17,14,19	17,14,19
Nref	4621	4593
Tmin,Tmax	0.971,0.980	0.967,0.980
Tmin’	0.967	

```
Correction method= MULTI-SCAN

Data completeness= 0.994          Theta(max)= 25.500

R(reflections)= 0.0742( 3440)     wR2(reflections)= 0.1825( 4593)

S = 1.021                 Npar= 326
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level B

PLAT093_ALERT_1_B No su’s on H-atoms, but refinement reported as . mixed

---

Alert level C

```
ABSTY02_ALERT_1_C  An _exptl_absorpt_correction_type has been given without
           a literature citation. This should be contained in the
           _exptl_absorpt_process_details field.
           Absorption correction given as multi-scan
```

Alert level G

```
PLAT005_ALERT_5_G No _iucr_refine_instructions_details in CIF ....          ?
PLAT072_ALERT_2_G SHELXL First  Parameter in WGHT Unusually Large.       0.11
PLAT199_ALERT_1_G Check the Reported _cell_measurement_temperature        293 K
PLAT200_ALERT_1_G Check the Reported   _diffrn_ambient_temperature        293 K
PLAT793_ALERT_4_G The Model has Chirality at C7      (Verify) ....          S
PLAT793_ALERT_4_G The Model has Chirality at C8      (Verify) ....          R
PLAT793_ALERT_4_G The Model has Chirality at C15     (Verify) ....          S
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   1 ALERT level B = A potentially serious problem, consider carefully
   1 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   7 ALERT level G = General information/check it is not something unexpected

   4 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   1 ALERT type 2 Indicator that the structure model may be wrong or deficient
   0 ALERT type 3 Indicator that the structure quality may be low
   3 ALERT type 4 Improvement, methodology, query or suggestion
   1 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica*, *Journal of Applied Crystallography*, *Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 28/03/2012; check.def file version of 18/03/2012

Datablock a20309a - ellipsoid plot