Pd/P(*t*-Bu)$_3$-Catalyzed Suzuki Cross-Couplings in the Presence of Water

Sha Lou and Gregory C. Fu*

Department of Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139

Supporting Information

Table of Contents

I. General Information

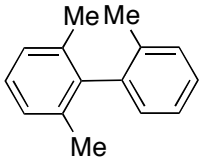
The cross-coupling partners were purchased and used without purification. THF (≥99.9%; inhibitor-free; Aldrich), $Pd_2(dba)_3$ (Strem), $HP(t\text{-}Bu)_3BF_4$ (Strem), and $Pd_2(dba)_3$/$[HP(t\text{-}Bu)_3]BF_4$ (Pd:P(*t*-Bu)$_3$ = 1:1.2; Strem) were purchased and used without purification. KF•2H$_2$O (Mallinckrodt Chemicals) was ground with a mortar and pestle before use.

Procedure for preparing a mixture of $Pd_2(dba)_3$/$[HP(t\text{-}Bu)_3]BF_4$ (Pd:P(*t*-Bu)$_3$ = 1:1.2). $Pd_2(dba)_3$ (458 mg, 0.50 mmol) and $HP(t\text{-}Bu)_3BF_4$ (348 mg, 1.20 mmol) were weighed into a mortar. The mixture was ground and mixed well with a pestle. The resulting fine powder was stored under air in a capped 4-mL vial.

II. Suzuki Cross-Couplings (Tables 2 and 3)

General Procedure for Suzuki cross couplings. $Pd_2(dba)_3$/$[HP(t\text{-}Bu)_3]BF_4$ (Pd:P(*t*-Bu)$_3$ = 1:1.2; 8.1 mg, 0.0050 mmol of $Pd_2(dba)_3$), the boronic acid (1.10 mmol), and KF•2H$_2$O (310 mg, 3.30 mmmol) were added to a 4-mL vial that contained a stir bar. The vial was purged with argon for 3 min, and then it was sealed with a septum cap. THF (2.0 mL) and the aryl halide (1.00 mmol) were added, and the reaction mixture was stirred at room temperature. Next, the mixture was diluted with Et$_2$O (2 mL) and filtered through a plug of silica gel (washed with Et$_2$O (10 mL)). The filtrate was concentrated, and the residue was purified by flash chromatography on silica gel.

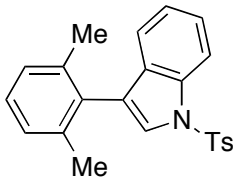
The first run was conducted with $Pd_2(dba)_3$/$[HP(t\text{-}Bu)_3]BF_4$ that we prepared. The second run employed commercially available $Pd_2(dba)_3$/$[HP(t\text{-}Bu)_3]BF_4$ (Strem).



2,2′,6-Trimethyl-1,1′biphenyl (Table 2, entry 1) [10273-87-7]. The title compound was prepared according to the General Procedure with 2-bromo-1,3-dimethylbenzene (133 μL; 185 mg, 1.00 mmol) and 2-methylbenzeneboronic acid (150 mg, 1.10 mmol). Reaction time: 2.5 h. After purification by flash chromatography (eluted with 5→15% Et_2O in hexanes), the title compound was isolated as a colorless oil (192 mg, 98% yield).

The second run furnished the product as a colorless oil (184 mg, 94% yield).

The ^1H NMR and ^{13}C NMR spectra are in agreement with reported values.[1]



3-(2,6-Dimethylphenyl)-1-tosyl-1*H*-indole (Table 2, entry 2). The title compound was prepared according to the General Procedure with 2-bromo-1,3-dimethylbenzene (133 μL; 185 mg, 1.00 mmol) and *N*-(toluenesulfonyl)indole-3-boronic acid (347 mg, 1.10 mmol). Reaction time: 4.0 h. After purification by flash chromatography (eluted with 15→50% Et_2O in hexanes), the title compound was isolated as a colorless oil (332 mg, 94% yield).

The second run furnished the product as a colorless oil (360 mg, 96% yield).

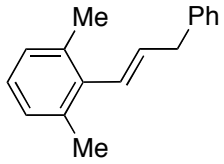
^1H NMR ($CDCl_3$, 500 MHz) δ 8.09 (d, J = 8.0 Hz, 1H), 7.78 (d, J = 8.0 Hz, 2H), 7.43 (s, 1H), 7.35 (t, J = 8.0 Hz, 1H), 7.24-7.18 (m, 4H), 7.12 (d, J = 8.0 Hz, 2H), 7.10 (d, J = 8.0 Hz, 1H), 2.35 (s, 3H), 1.99 (s, 6H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 145.1, 138.0 (2C), 135.4, 135.3, 131.5, 131.0, 130.0 (2C), 128.1, 127.5 (2C), 126.9 (2C), 125.0, 124.3, 123.6, 122.8, 120.6, 114.2, 21.7, 20.7 (2C);

IR (film) 3025, 1597, 1446, 1371, 1176, 1126, 1011, 931, 751, 693, 661 cm^{-1};

LRMS (ESI) calcd for $C_{23}H_{22}NO_2S$ (M+H) 376.1, found, 376.1.

[1] A. F. Littke, C. Dai, G. C. Fu, *J. Am. Chem. Soc.* **2000**, *122*, 4020–4028.



(*E*)-1,3-Dimethyl-2-(3-phenylprop-1-en-1-yl)benzene (Table 2, entry 3). The title compound was prepared according to the General Procedure with 2-bromo-1,3-dimethylbenzene (133 μL; 185 mg, 1.00 mmol) and *trans*-3-phenyl-1-propen-1-ylboronic acid (178 mg, 1.10 mmol). Reaction time: 4.0 h. After purification by flash chromatography (eluted with 5→15% Et_2O in hexanes), the title compound was isolated as a colorless oil (218 mg, 98% yield).

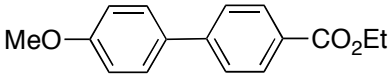
The second run furnished the product as a colorless oil (216 mg, 97% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.32-7.19 (m, 3H), 7.02-7.00 (m, 3H), 6.40 (d, *J* = 16.0 Hz, 1H), 5.85-5.78 (m, 1H), 3.58 (d, *J* = 7.0 Hz, 2H), 2.29 (s, 6H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 140.5, 137.4, 136.0, 134.1 (2C), 129.0, 128.8 (2C), 128.6 (2C), 127.8 (2C), 126.5, 126.3, 40.0, 21.2 (2C);

IR (film) 3026, 1603, 1494, 1465, 1453, 1376, 1029, 974, 768, 732, 698 cm^{-1};

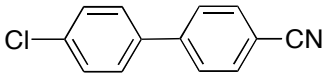
LRMS (ESI) calcd for $C_{17}H_{19}$ (M+H) 223.1, found, 223.1.



Ethyl 4'-methoxy-[1,1′-biphenyl]-4-carboxylate (Table 2, entry 4) [732-80-9]. The title compound was prepared according to the General Procedure with 4-bromoanisole (125 μL; 187 mg, 1.00 mmol) and 4-(ethoxycarbonyl)phenylboronic acid (213 mg, 1.10 mmol). Reaction time: 36 h. After purification by flash chromatography (eluted with 15→50% Et_2O in hexanes), the title compound was isolated as a colorless oil (223 mg, 87% yield).

The second run furnished the product as a colorless oil (208 mg, 81% yield).

The ^{1}H NMR and ^{13}C NMR spectra are in agreement with reported values.[2]



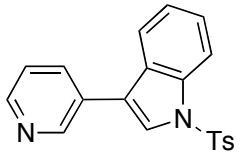
4'-Chloro-[1,1′-biphenyl]-4-carbonitrile (Table 2, entry 5) [57774-36-4]. The title compound was prepared according to the General Procedure with 4-bromochlorobenzene (192 mg, 1.00 mmol) and 4-cyanophenylboronic acid (162 mg, 1.10 mmol). Reaction time: 4.0 h. After

[2] S. E. Denmark, M. H. Ober, *Adv. Synth. Catal.* **2004**, *346*, 1703–1714.

purification by flash chromatography (eluted with 5→15% Et_2O in hexanes), the title compound was isolated as a colorless oil (212 mg, 99% yield).

The second run furnished the product as a colorless oil (207 mg, 97% yield).

The ^1H NMR and ^{13}C NMR spectra are in agreement with reported values.[3]



3-(Pyridin-3-yl)-1-tosyl-1*H*-indole (Table 2, entry 6). The title compound was prepared according to the General Procedure with 3-bromopyridine (96 μL; 158 mg, 1.00 mmol) and *N*-(toluenesulfonyl)indole-3-boronic acid (347 mg, 1.10 mmol). Reaction time: 2.5 h. After purification by flash chromatography (eluted with 20→80% Et_2O in hexanes), the title compound was isolated as a colorless oil (313 mg, 90% yield).

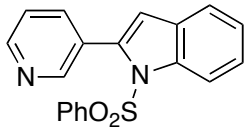
The second run furnished the product as a colorless oil (331 mg, 95% yield).

^1H NMR ($CDCl_3$, 500 MHz) δ 8.85 (d, *J* = 2.5 Hz, 1H), 8.59 (d, *J* = 4.5 Hz, 1H), 8.05 (d, *J* = 8.0 Hz, 1H), 7.88 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 2H), 7.73 (s, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 2H), 7.28 (t, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 149.0, 148.9, 145.5, 136.8, 135.6, 135.3, 135.2, 130.3 (2C), 127.2 (2C), 125.5, 124.0, 123.9, 123.7, 120.5, 120.2, 114.1, 86.7, 21.8;

IR (film) 3031, 1596, 1446, 1372, 1175, 1134, 1089, 1009, 811, 746, 675 cm^{-1};

LRMS (ESI) calcd for $C_{20}H_{17}N_2O_2S$ (M+H) 349.1, found 349.1.



1-(Phenylsulfonyl)-2-(pyridin-3-yl)-1*H*-indole (Table 2, entry 7). The title compound was prepared according to the General Procedure with 3-bromopyridine (96 μL; 158 mg, 1.00 mmol) and 1-(phenylsulfonyl)-2-indoleboronic acid (331 mg, 1.10 mmol). Reaction time: 36 h. After purification by flash chromatography (eluted with 20→80% Et_2O in hexanes), the title compound was isolated as a colorless oil (267 mg, 80% yield).

The second run furnished the product as a colorless oil (281 mg, 84% yield).

^1H NMR ($CDCl_3$, 500 MHz) δ 8.61 (d, *J* = 5.0 Hz, 1H), 8.55 (s, 1H), 8.27 (d, *J* = 8.0 Hz, 1H), 7.87 (dt, *J* = 8.0, 1.0 Hz, 1H), 7.43-7.29 (m, 6H), 7.25-7.20 (m, 3H), 6.57 (s, 1H);

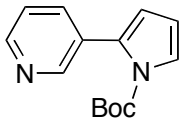
---

[3] R. Martin, S. L. Buchwald, *J. Am. Chem. Soc.* **2007**, *129*, 3844–3845.

^{13}C NMR ($CDCl_3$, 125 MHz) δ 150.0, 149.7, 138.5, 138.3, 137.4, 134.1 (2C), 130.4, 129.0 (2C), 128.7, 126.7 (2C), 125.6, 124.8, 122.6, 121.2, 116.7, 114.9;

IR (film) 3065, 1570, 1448, 1411, 1374, 1185, 1120, 1091, 753, 555 cm^{-1};

LRMS (ESI) calcd for $C_{19}H_{15}N_2O_2S$ (M+H) 335.1, found 335.1.



***tert*-Butyl 2-(pyridin-3-yl)-1*H*-pyrrole-1-carboxylate (Table 2, entry 8).** The title compound was prepared according to the General Procedure with 3-bromopyridine (96 μL; 158 mg, 1.00 mmol) and 1-*N*-Boc-pyrrole-2-boronic acid (232 mg, 1.10 mmol). Reaction time: 4.0 h. After purification by flash chromatography (eluted with 10→80% Et_2O in hexanes), the title compound was isolated as a yellow oil (234 mg, 96% yield).

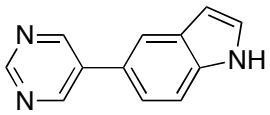
The second run furnished the product as a colorless oil (227 mg, 93% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 8.59 (dd, *J* = 2.5, 1.0 Hz, 1H), 8.52 (dd, *J* = 5.0, 2.0 Hz, 1H), 7.65 (dt, *J* = 8.0, 2.0 Hz, 1H), 7.40 (dd, *J* = 4.0, 2.0 Hz, 1H), 7.26 (dd, *J* = 8.0, 4.5 Hz, 1H), 6.26-6.23 (m, 2H), 1.37 (s, 9H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 145.0, 149.2, 148.3, 136.5, 131.4, 130.5, 123.4, 122.6, 115.7, 111.0, 84.3, 27.8 (3C);

IR (film) 2982, 1743, 1569, 1458, 1394, 1313, 1148, 972, 842, 756, 732 cm^{-1};

LRMS (ESI) calcd for $C_{14}H_{17}N_2O_2$ (M+H) 245.1, found 245.1.



5-(Pyrimidin-5-yl)-1*H*-indole (Table 2, entry 9). The title compound was prepared according to the General Procedure with 5-bromopyrimidine (159 mg, 1.00 mmol) and indole-5-boronic acid (177 mg, 1.10 mmol). Reaction time: 36 h. After purification by flash chromatography (eluted with 10→80% EtOAc in CH_2Cl_2), the title compound was isolated as a white solid (152 mg, 78% yield).

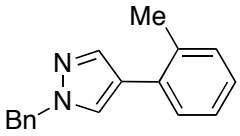
The second run furnished the product as a colorless oil (142 mg, 73% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 9.15 (s, 1H), 8.99 (s, 2H), 8.46 (br, 1H), 7.84 (s, 1H), 7.52 (d, *J* = 8.5 Hz, 1H), 7.39 (d, *J* = 8.5 Hz, 1H), 7.29 (t, *J* = 2.5 Hz, 1H), 6.64 (s, 1H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 156.8, 155.2 (2C), 136.2, 135.8, 128.9, 126.2, 125.8, 121.3, 119.7, 112.3, 103.4;

IR (film) 3179, 1597, 1562, 1408, 1314, 882, 765, 716 cm^{-1};

LRMS (ESI) calcd for $C_{12}H_{10}N_3$ (M+H) 196.1, found 196.1.



1-Benzyl-4-(*o*-tolyl)-1*H*-pyrazole (Table 2, entry 10). The title compound was prepared according to the General Procedure with 4-bromo-1-benzyl-1H-pyrazole (237 mg, 1.00 mmol) and 2-methylbenzeneboronic acid (150 mg, 1.10 mmol). Reaction time: 2.5 h. After purification by flash chromatography (eluted with 10→40% Et_2O in hexanes), the title compound was isolated as a colorless oil (219 mg, 88% yield).

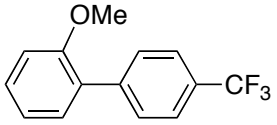
The second run furnished the product as a colorless oil (214 mg, 86% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.72 (s, 1H), 7.51 (s, 1H), 7.40-7.37 (m, 2H), 7.35-7.33 (m, 2H), 7.30 -7.24 (m, 3H), 7.22-7.19 (m, 2H), 5.38 (s, 2H), 2.40 (s, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 139.4, 136.7, 135.4, 132.3, 130.9, 129.2, 129.1 (2C), 128.32, 128.28, 127.9 (2C), 126.9, 126.1, 122.7, 56.3, 21.5;

IR (film) 2918, 1561, 1453, 1376, 1187, 995, 760, 723 cm^{-1};

LRMS (ESI) calcd for $C_{17}H_{17}N_2$ (M+H) 249.1, found 249.1.



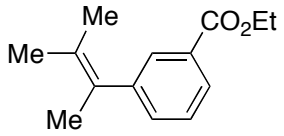
2-Methoxy-4'-(trifluoromethyl)biphenyl (eq 3) [122801-55-2]. The title compound was prepared according to the General Procedure with 2-iodoanisole (133 μL; 234 mg, 1.00 mmol) and 4-(trifluoromethyl)phenylboronic acid (209 mg, 1.10 mmol). Reaction time: 36 h. After purification by flash chromatography (eluted with 10→60% benzene in hexanes), the title compound was isolated as a colorless oil (222 mg, 88% yield).

The second run furnished the product as a colorless oil (207 mg, 82% yield).

The ^{1}H NMR and ^{13}C NMR spectra are in agreement with reported values.[4]

---

[4] S. E. Denmark, R. C. Smith, W.-T. T. Chang, J. M. Muhuhi, *J. Am. Chem. Soc.* **2009**, *131*, 3104–3118.



Ethyl 3-(3-methylbut-2-en-2-yl)benzoate (eq 4). The title compound was prepared according to the General Procedure with 2-bromo-3-methyl-2-butene (116 μL; 149 mg, 1.00 mmol) and 3-(ethoxycarbonyl)phenylboronic acid (213 mg, 1.10 mmol). Reaction time: 24 h. After purification by flash chromatography (eluted with 5→20% Et_2O in hexanes), the title compound was isolated as a colorless oil (135 mg, 62% yield).

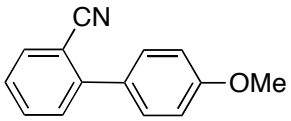
The second run furnished the product as a colorless oil (128 mg, 59% yield).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.89 (d, *J* = 7.5 Hz, 1H), 7.82 (s, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 7.5 Hz, 1H), 4.38 (q, *J* = 7.0 Hz, 2H), 1.98 (s, 3H), 1.83 (s, 3H), 1.58 (s, 3H), 1.41 (t, *J* = 7.0 Hz, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 167.1, 145.7, 133.2, 130.4, 129.7, 129.4, 128.4, 128.2, 127.2, 61.1, 22.3, 20.9, 20.8, 14.6;

IR (film) 2983, 1719, 1447, 1306, 1252, 1202, 1107, 1026, 758 cm^{-1};

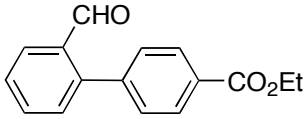
LRMS (ESI) calcd for $C_{14}H_{19}O_2$ (M+H) 219.1, found 219.1.



4'-Methoxy-[1,1′-biphenyl]-2-carbonitrile (Table 3, entry 1) [125610-78-8]. The title compound was prepared according to the General Procedure with 2-chlorobenzonitrile (138 mg, 1.00 mmol) and 4-methoxyphenylboronic acid (167 mg, 1.10 mmol). Reaction time: 4.0 h. After purification by flash chromatography (eluted with 5→40% Et_2O in hexanes), the title compound was isolated as a colorless oil (203 mg, 97% yield).

The second run furnished the product as a colorless oil (201 mg, 96% yield).

The ^{1}H NMR and ^{13}C NMR spectra are in agreement with reported values.[5]



Ethyl 2'-formyl-[1,1′-biphenyl]-4-carboxylate (Table 3, entry 2). The title compound was prepared according to the General Procedure with 2-chlorobenzaldehyde (113 μL; 141 mg, 1.00 mmol) and 4-(ethoxycarbonyl)phenylboronic acid (213 mg, 1.10 mmol). Reaction time: 24 h.

---

[5] G. Manolikakes, P. Knochel, *Angew. Chem. Int. Ed.* **2009**, *48*, 205–209.

After purification by flash chromatography (eluted with 5→50% Et_2O in hexanes), the title compound was isolated as a colorless oil (185 mg, 73% yield; 46 mg, 17% yield of the hydrate).

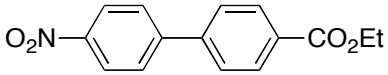
The second run furnished the product as a colorless oil (175 mg, 69% yield; 63 mg, 23% yield of the hydrate).

^{1}H NMR ($CDCl_3$, 500 MHz) δ 9.94 (s, 1H), 8.13 (d, *J* = 7.0 Hz, 2H), 8.03 (d, *J* = 7.5 Hz, 1H), 7.65 (t, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.44 (d, *J* = 7.0 Hz, 2H), 7.42 (d, *J* = 7.5 Hz, 1H), 4.40 (q, *J* = 7.0 Hz, 2H), 1.41 (t, *J* = 7.0 Hz, 3H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 192.0, 166.4, 144.9, 144.5, 142.5, 133.9, 130.8, 130.3 (2C), 129.8 (2C), 128.6, 128.1, 127.4, 51.4, 14.6;

IR (film) 2982, 1716, 1694, 1596, 1396, 1277, 1104, 760 cm^{-1};

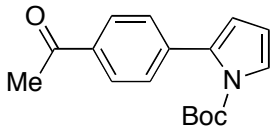
LRMS (ESI) calcd for $C_{16}H_{15}O_3$ (M+H) 255.1, found 255.1.



Ethyl 4'-nitro-[1,1′-biphenyl]-4-carboxylate (Table 3, entry 3) [6242-99-5]. The title compound was prepared according to the General Procedure with 1-chloro-4-nitrobenzene (133 μL; 158 mg, 1.00 mmol) and 4-(ethoxycarbonyl)phenylboronic acid (213 mg, 1.10 mmol). Reaction time: 2.0 h. After purification by flash chromatography (eluted with 20→80% Et_2O in hexanes), the title compound was isolated as a white solid (268 mg, 99% yield).

The second run furnished the product as a white solid (266 mg, 98% yield).

The ^{1}H NMR and ^{13}C NMR spectra are in agreement with reported values.[6]



***tert*-Butyl 2-(4-acetylphenyl)-1*H*-pyrrole-1-carboxylate (Table 3, entry 4).** The title compound was prepared according to the General Procedure with 4′-chloroacetophenone (130 μL; 155 mg, 1.00 mmol) and 1-*N*-Boc-pyrrole-2-boronic acid (232 mg, 1.10 mmol). Reaction time: 1.0 h. After purification by flash chromatography (eluted with 5→15% Et_2O in hexanes), the title compound was isolated as a colorless oil (280 mg, 98% yield).

The second run furnished the product as a colorless oil (277 mg, 97% yield).

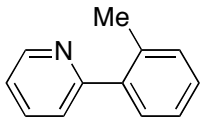
^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.88 (d, *J* = 7.0 Hz, 2H), 7.42 (d, *J* = 7.0 Hz, 2H), 7.21 (t, *J* = 2.0 Hz, 1H), 6.20 (d, *J* = 2.0 Hz, 1H), 6.19 (d, *J* = 2.0 Hz, 1H), 2.57 (s, 3H), 1.57 (s, 9H);

^{13}C NMR ($CDCl_3$, 125 MHz) δ 197.0, 156.0, 139.7, 135.6, 129.9 (2C), 129.1 (2C), 120.1 (2C), 112.0 (2C), 83.7, 28.2 (3C), 26.8;

---

[6] P. Lan, D. Berta, J. A. Porco, Jr, M. S. South, J. J. Parlow, *J. Org. Chem.* **2003**, *68*, 9678–9686.

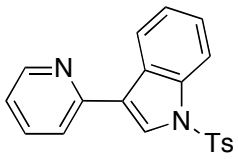
IR (film) 2981, 1747, 1685, 1590, 1472, 1399, 1343, 1319, 1260, 1154, 1094, 952, 828, 742 cm^{-1}; LRMS (ESI) calcd for $C_{17}H_{20}NO_3$ (M+H) 286.1, found 286.1.



2-*o*-Tolylpyridine (Table 3, entry 5) [10273-89-9]. The title compound was prepared according to the General Procedure with 2-chloropyridine (95 μL; 114 mg, 1.00 mmol) and 2-methylbenzeneboronic acid (150 mg, 1.10 mmol). Reaction time: 12 h. After purification by flash chromatography (eluted with 5→30% Et_2O in hexanes), the title compound was isolated as a colorless oil (156 mg, 92% yield).

The second run furnished the product as a colorless oil (154 mg, 91% yield).

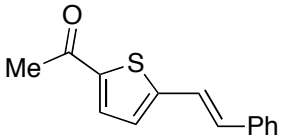
The ^1H NMR and ^{13}C NMR spectra are in agreement with reported values.[1]



3-(Pyridin-2-yl)-1-tosyl-1*H*-indole (Table 3, entry 6) [758686-21-4]. The title compound was prepared according to the General Procedure with 2-chloropyridine (95 μL; 114 mg, 1.00 mmol) and *N*-(toluenesulfonyl)indole-3-boronic acid (347 mg, 1.10 mmol). Reaction time: 12 h. After purification by flash chromatography (eluted with 10→60% Et_2O in hexanes), the title compound was isolated as a colorless oil (324 mg, 93% yield).

The second run furnished the product as a colorless oil (338 mg, 97% yield).

The ^1H NMR and ^{13}C NMR spectra are in agreement with reported values.[7]



(*E*)-1-(5-Styrylthiophen-2-yl)ethanone (Table 3, entry 7) [125972-78-3]. The title compound was prepared according to the General Procedure with 2-acetyl-5-chlorothiophene (161 mg, 1.00 mmol) and *trans*-2-phenylvinylboronic acid (163 mg, 1.10 mmol). Reaction time: 12 h. After

[7] T. Miyagi, Y. Hari, T. Aoyama, *Tetrahedron Lett.* **2004**, *45*, 6303–6305.

purification by flash chromatography (eluted with 5→15% Et_2O in hexanes), the title compound was isolated as a colorless oil (196 mg, 86% yield).

The second run furnished the product as a colorless oil (205 mg, 90% yield).

The ^{1}H NMR and ^{13}C NMR spectra are in agreement with reported values.[8]

[8] C. J. O'Brien, E. A. B. Kantchev, C. Valente, N. Hadei, G. A. Chass, A. Lough, A. C. Hopkinson, M. G. Organ, *Chem. Eur. J.* **2006**, *12*, 4743–4748.

Table 2, entry 1

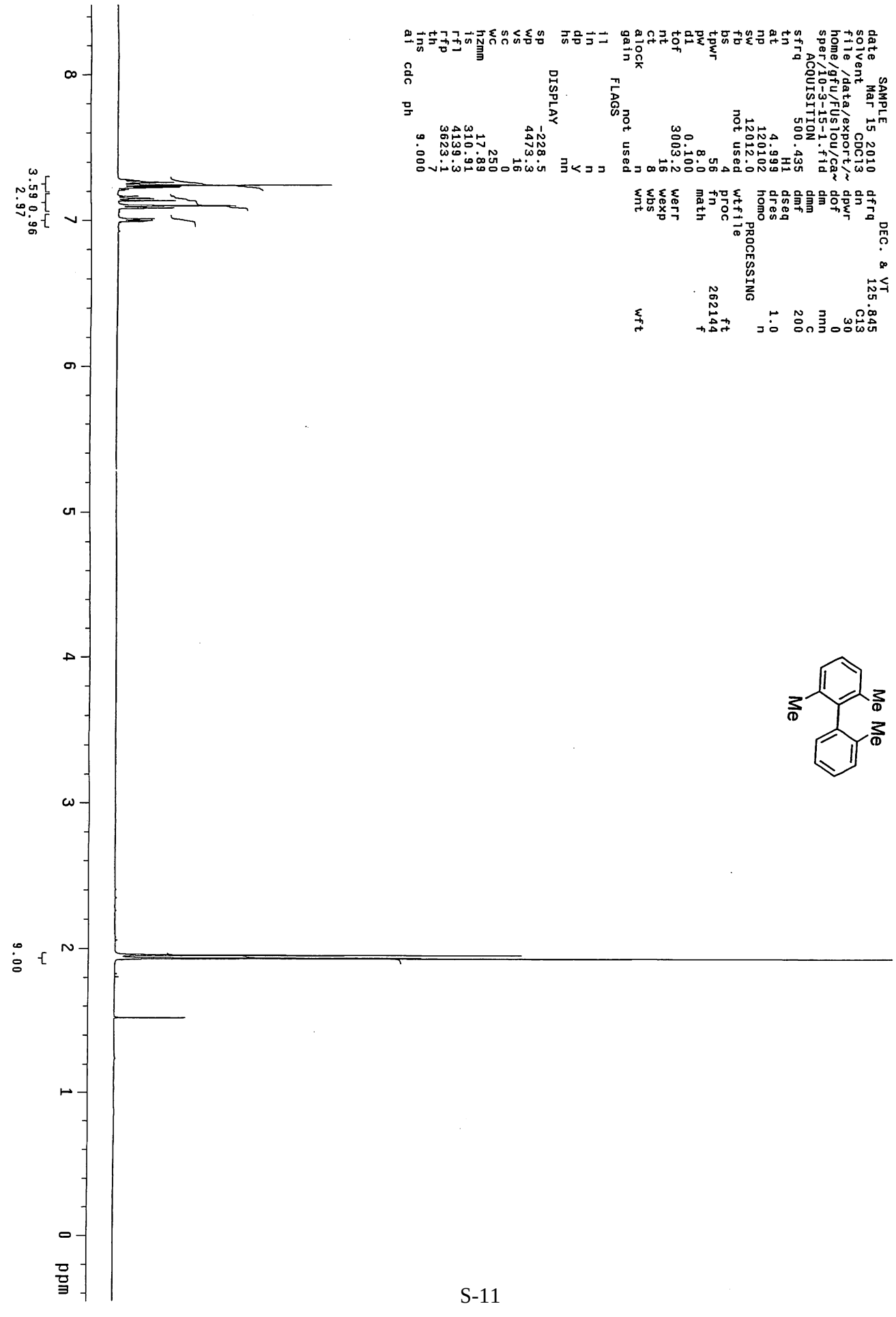


Table 2, entry 2

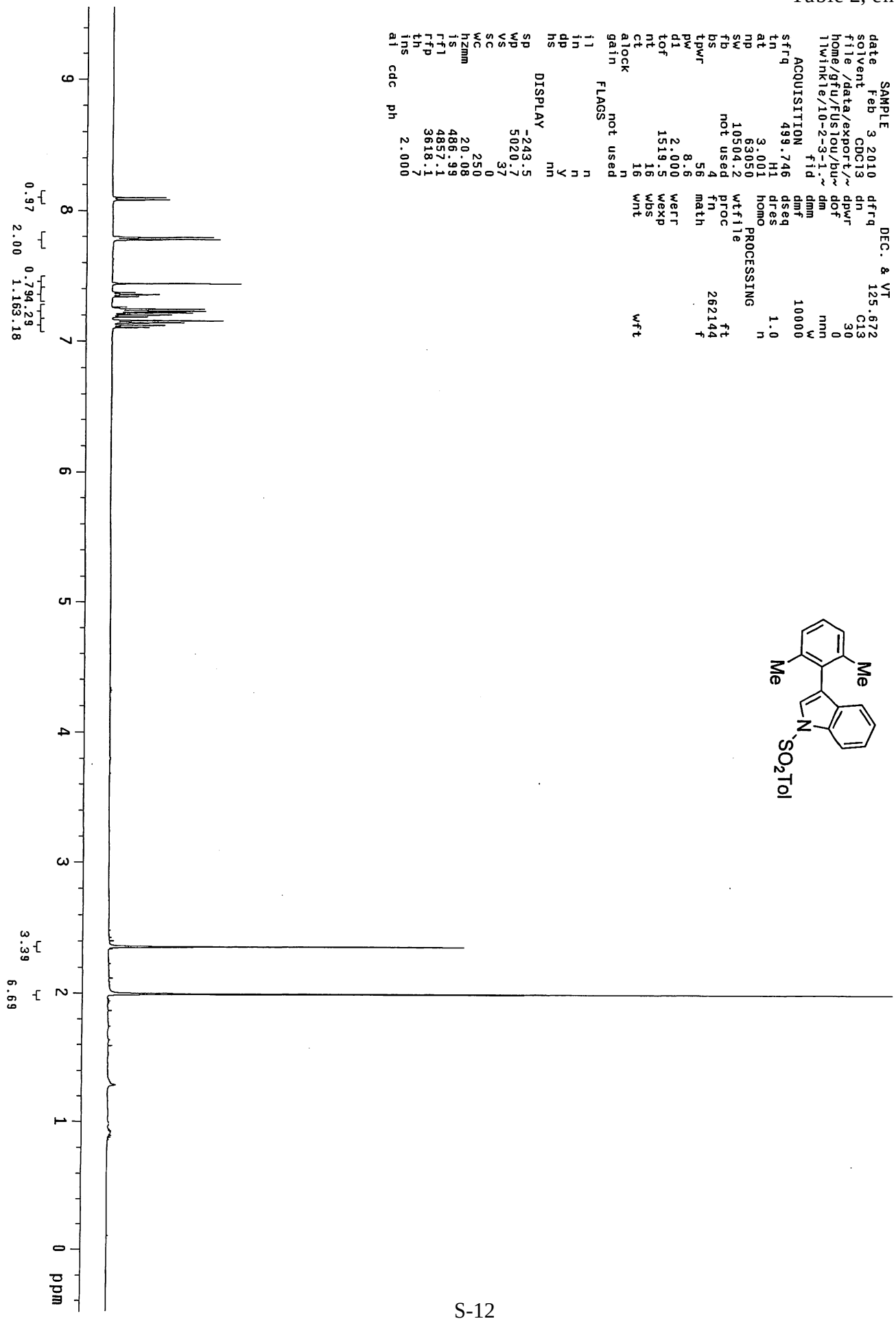


Table 2, entry 3

10-3-16-14

exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 16 2010	dfrq	125.845
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
	home/gfu/FUslou/ca~	dof	0
	sper/10-3-16-14.fi~	dm	nnn
	d	dmm	c
ACQUISITION		dmf	200
sfrq	500.435	dseq	
tn	H1	dres	1.0
at	4.999	homo	n
np	120102	PROCESSING	
sw	12012.0	wtfile	
fb	not used	proc	ft
bs	4	fn	262144
tpwr	56	math	f
pw	8.0		
d1	0.100	werr	
tof	3003.2	wexp	
nt	16	wbs	
ct	16	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-236.1		
wp	4842.7		
vs	15		
sc	0		
wc	250		
hzmm	19.37		
is	238.87		
rfl	4138.8		
rfp	3623.1		
th	6		
ins	6.000		
ai cdc ph			

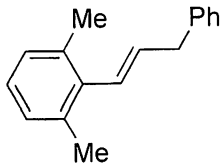


Table 2, entry 4

10-3-16-11

exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 16 2010	dfrq	125.845
solvent	CDCl3	dn	C13
file	/data/export/~	dpwr	30
home/gfu/FUslou/ca~		dof	0
sper/10-3-16-11.fi~		dm	nnn
	d	dmm	c
ACQUISITION		dmf	200
sfrq	500.435	dseq	
tn	H1	dres	1.0
at	4.999	homo	n
np	120102	PROCESSING	
sw	12012.0	wtfile	
fb	not used	proc	ft
bs	4	fn	262144
tpwr	56	math	f
pw	8.0		
d1	0.100	werr	
tof	3003.2	wexp	
nt	16	wbs	
ct	16	wnt	wft
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-255.9		
wp	4972.4		
vs	21		
sc	0		
wc	250		
hzmm	19.89		
is	409.66		
rfl	4138.7		
rfp	3623.1		
th	7		
ins	3.000		
ai cdc ph			

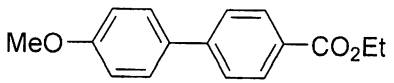


Table 2, entry 5

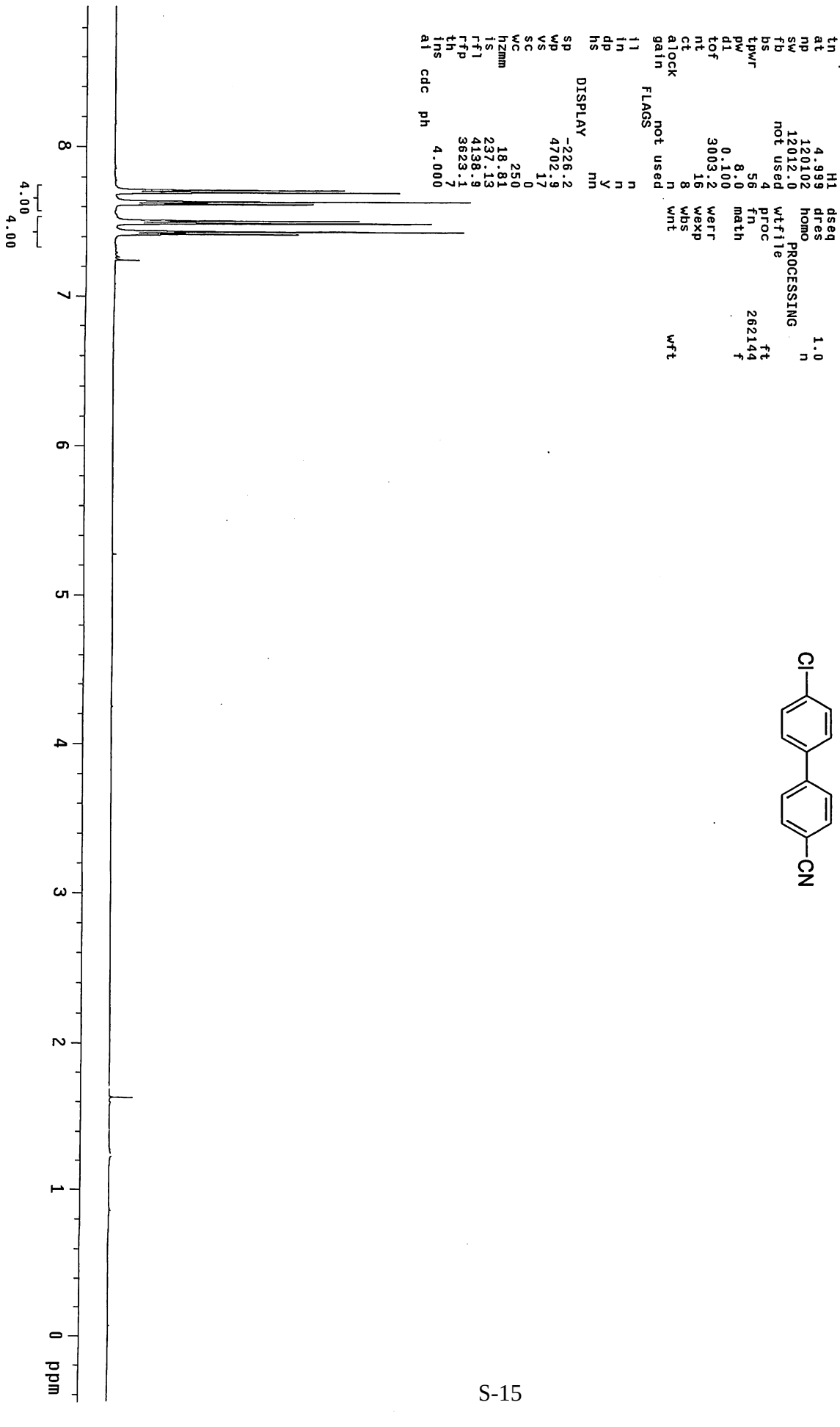


Table 2, entry 6

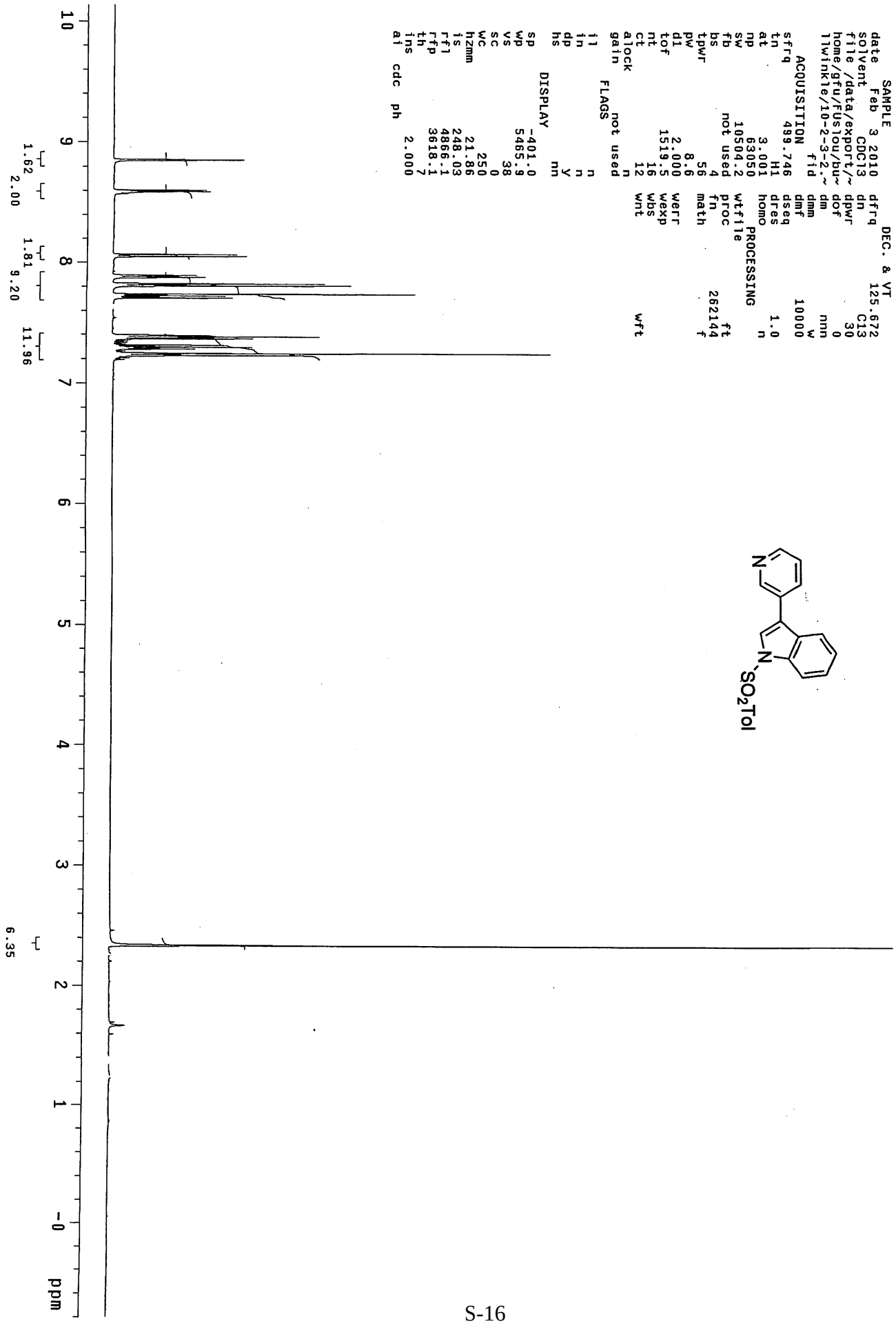


Table 2, entry 7

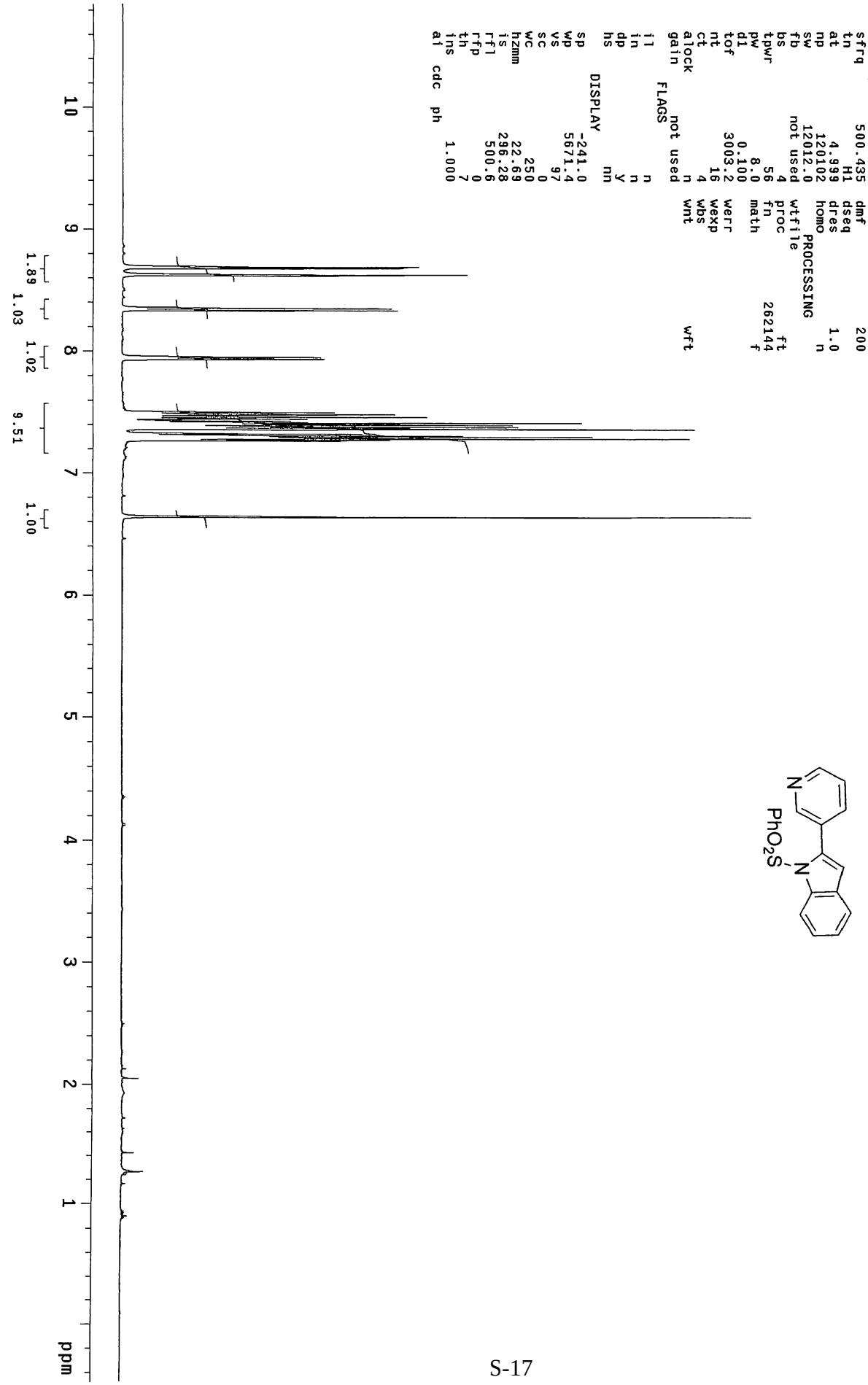


Table 2, entry 8

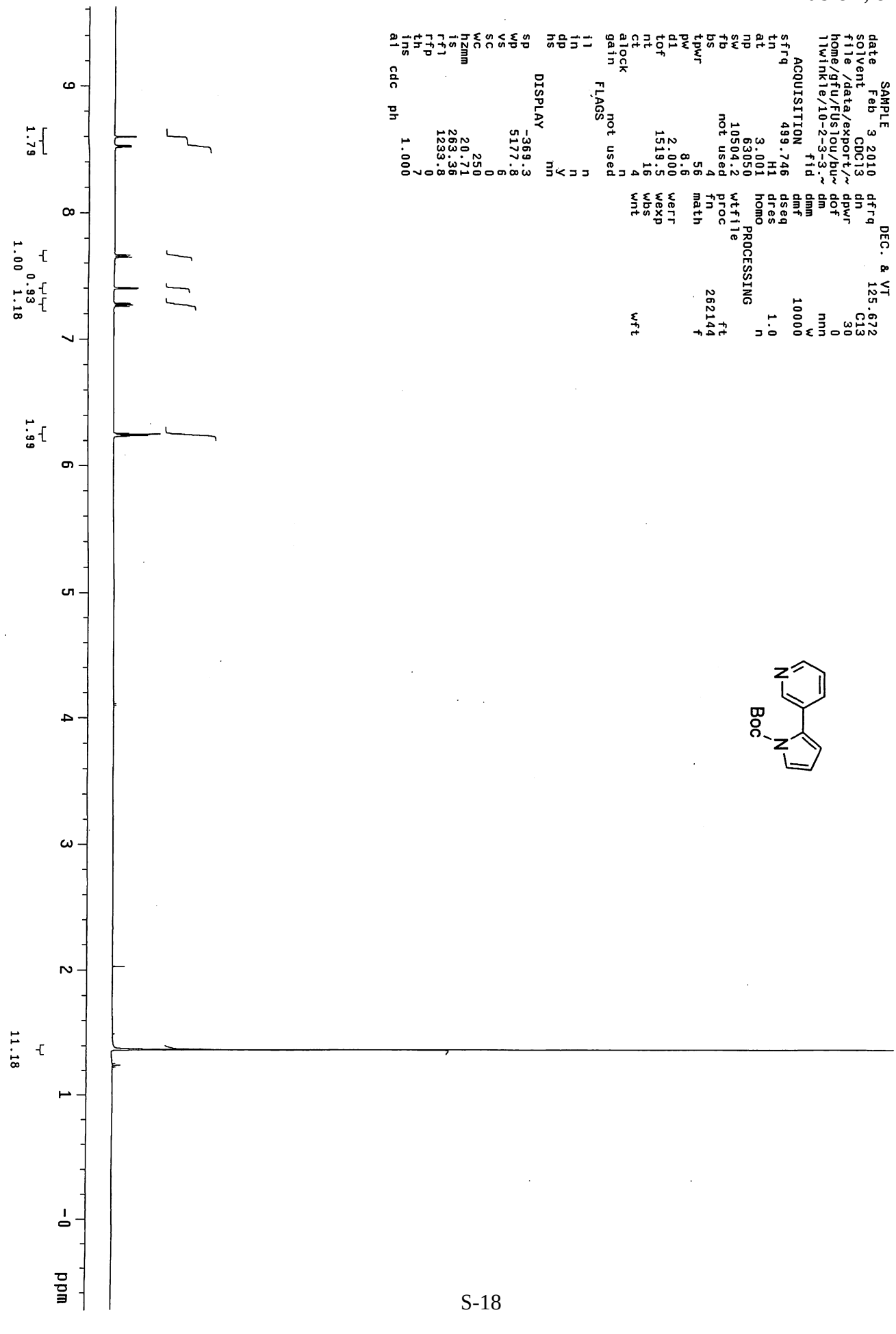


Table 2, entry 9

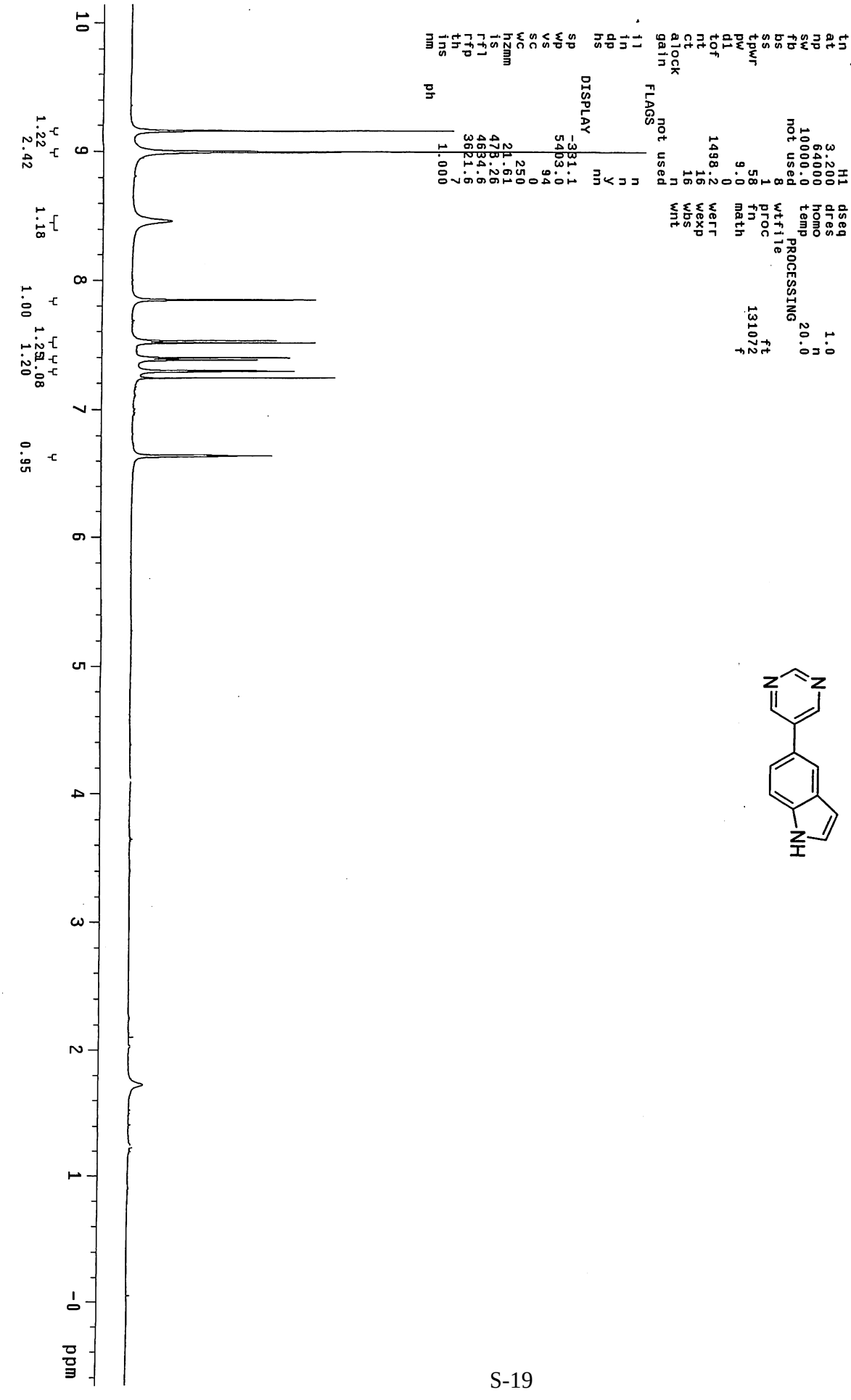
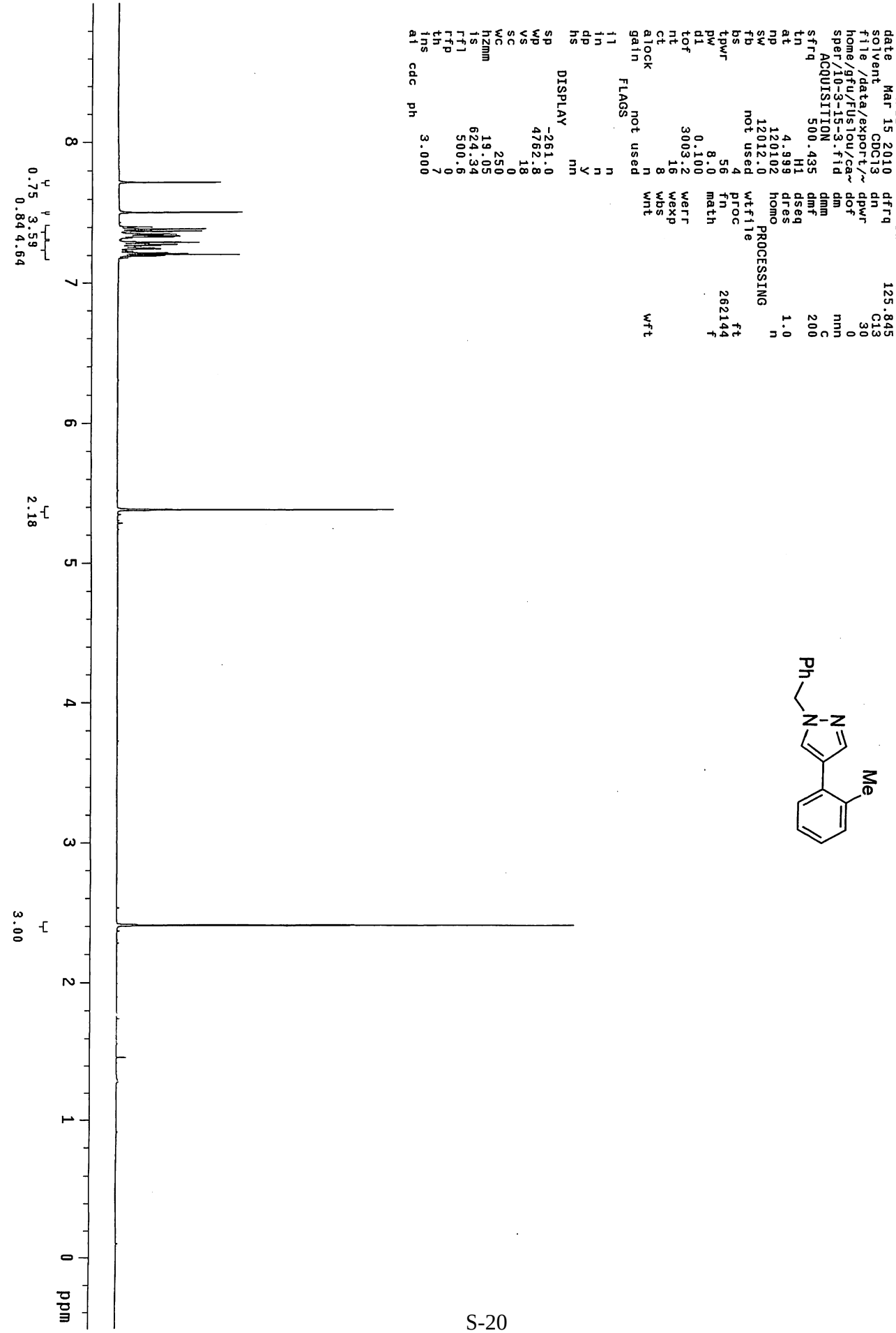
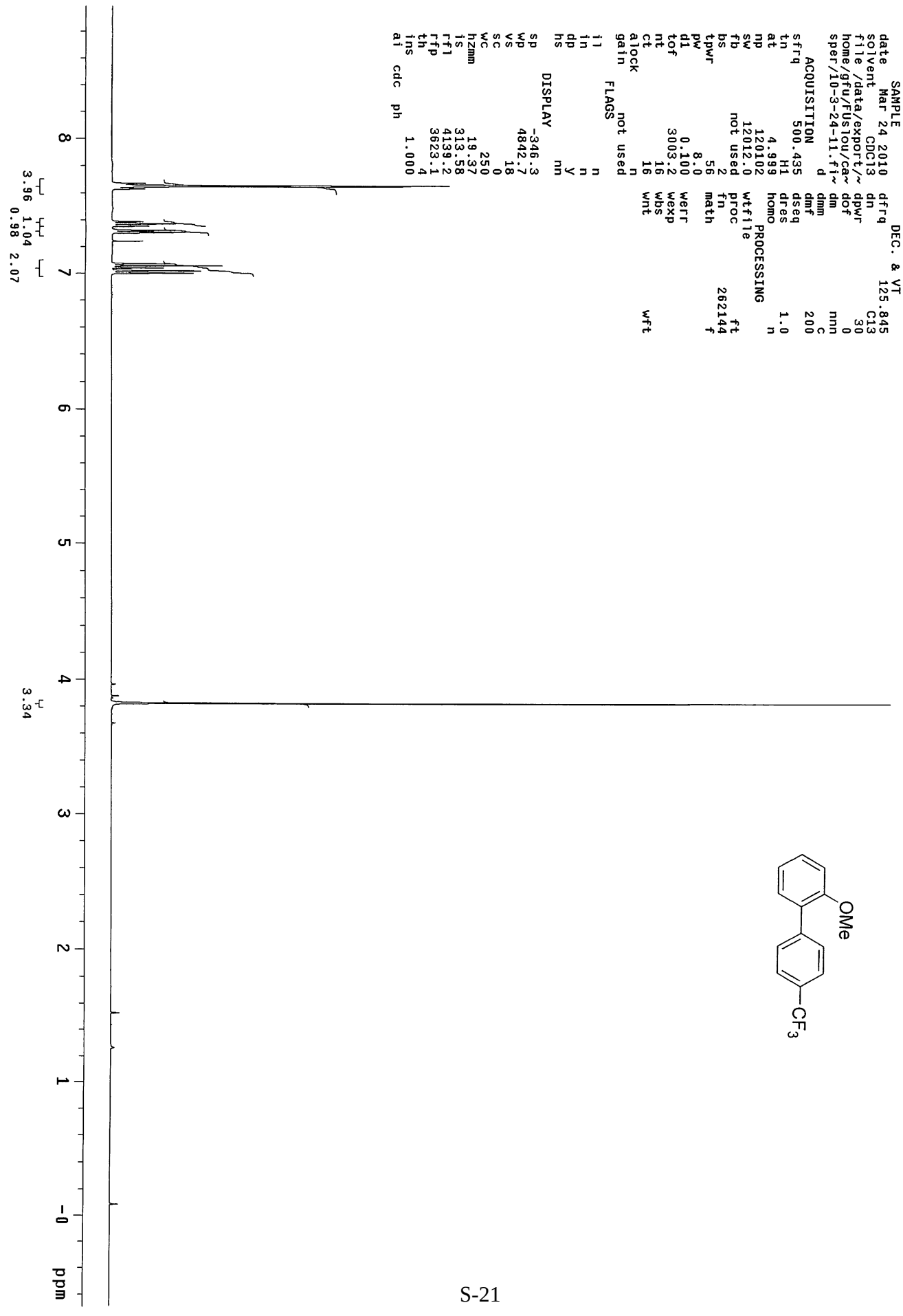


Table 2, entry 10



Eq. (3) product


10-3-24-11
exp1 s2pul
SAMPLE
date Mar 24 2010
solvent CDCl3
file /data/export/~
home/gfu/FUslou/ca~
sper/10-3-24-11.fi~
ACQUISITION
sfrq 500.435
tn H1
at 4.999
np 120102
sw 12012.0
fb not used
bs 2
tpwr 56
pw 8.0
d1 0.100
tof 3003.2
nt 16
ct 16
alock wnt
gain n
FLAGS
not used
DEC. & VT
dfrq 125.845
dn C13
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc
fn 262144
math f
wft
il n
in n
dp y
hs nn
DISPLAY
sp -346.3
wp 4842.7
vs 18
sc 0
wc 250
hzmm 19.37
is 313.58
rfl 4139.2
rfp 3623.1
th 4
ins
ai cdc ph 1.000
OMe
CF3
3.96 1.04
0.98 2.07
3.34
8
7
6
5
4
3
2
1
-0
ppm

Eq.(4) product

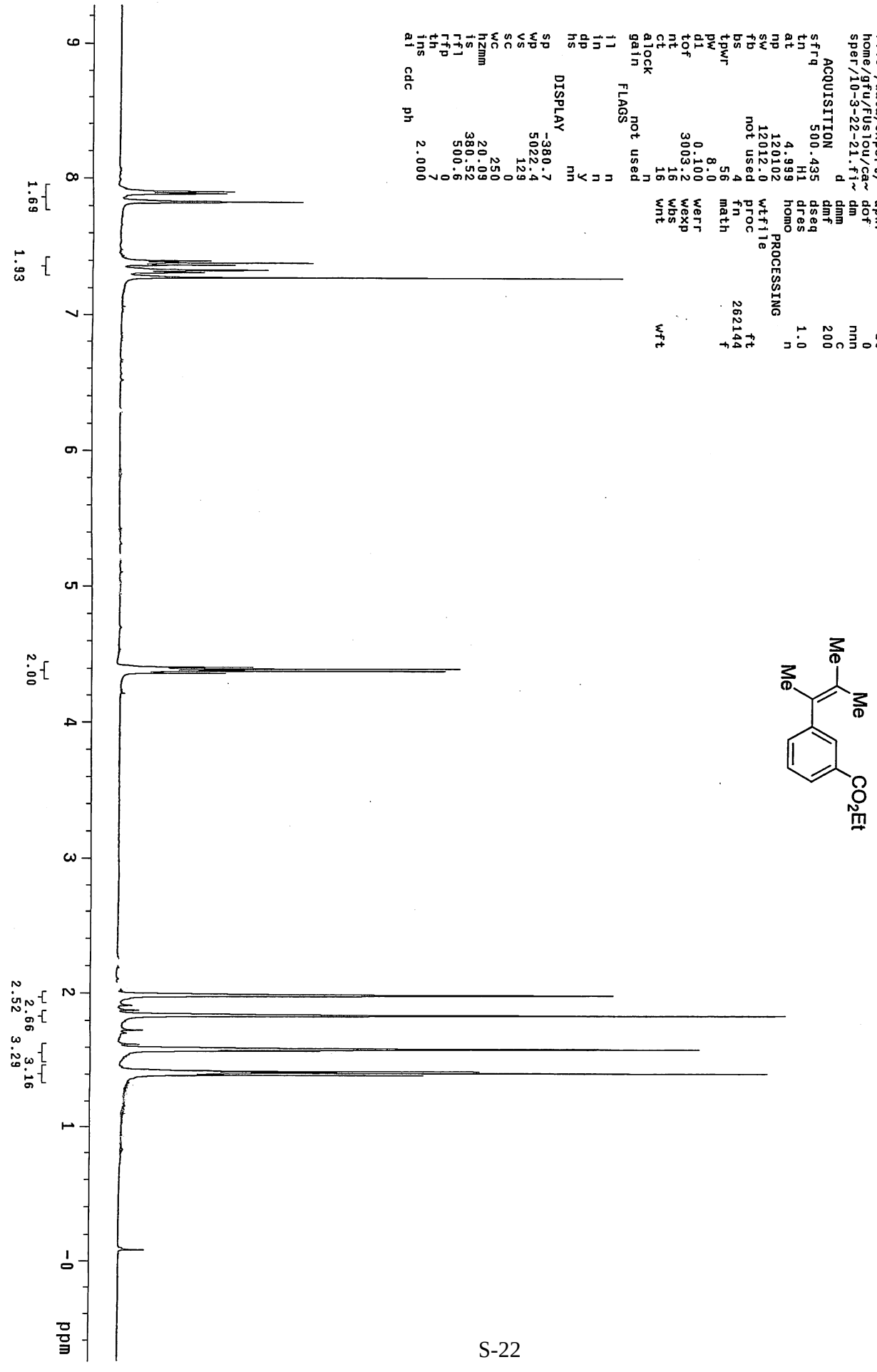

10-3-22-21
exp1 s2pul
SAMPLE
date Mar 23 2010
solvent CDCl3
file /data/export/~
home/gfu/Fuslou/ca~
sper/10-3-22-21.fi~
ACQUISITION
sfrq 500.435
tn H1
at 4.999
np 120102
sw 12012.0
fb not used
bs 4
tpwr 56
pw 8.0
d1 0.100
tof 3003.2
nt 16
ct 16
alock wnt
gain n
FLAGS
not used
il n
in n
dp y
hs nn
DISPLAY
sp -380.7
wp 5022.4
vs 129
sc 0
wc 250
hzmm 20.09
is 380.52
rfl 500.6
rfp 0
th 7
ins 2.000
ai cdc ph
DEC. & VT
dfrq 125.845
dn C13
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq 1.0
dres n
homo
PROCESSING
wtfile
proc ft
fn 262144
math f
wft
Me
Me
Me
CO2Et
1.69
1.93
2.00
2.66
3.16
2.52
3.29
9
8
7
6
5
4
3
2
1
-0
ppm

Table 3, entry 1

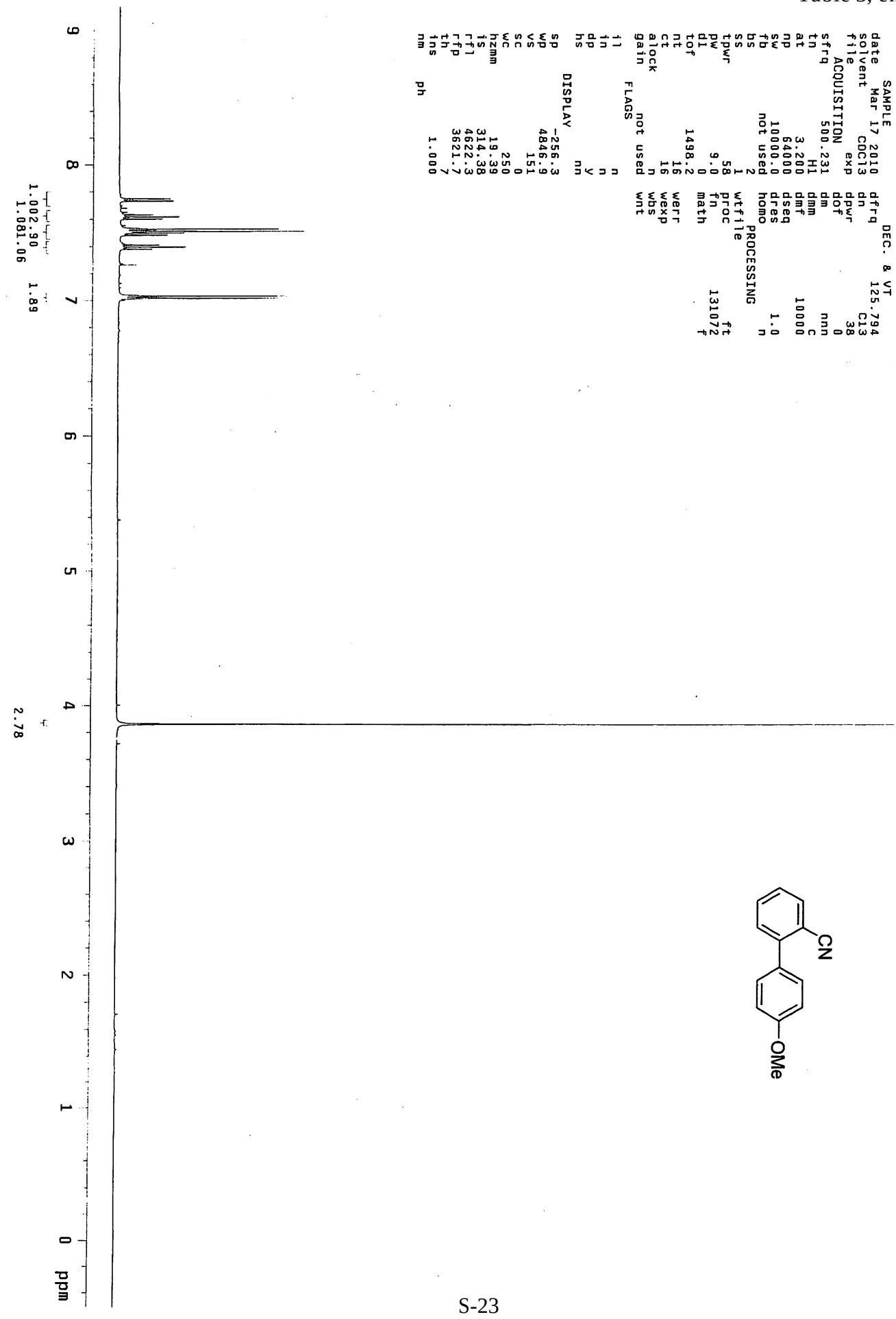


Table 3, entry 2

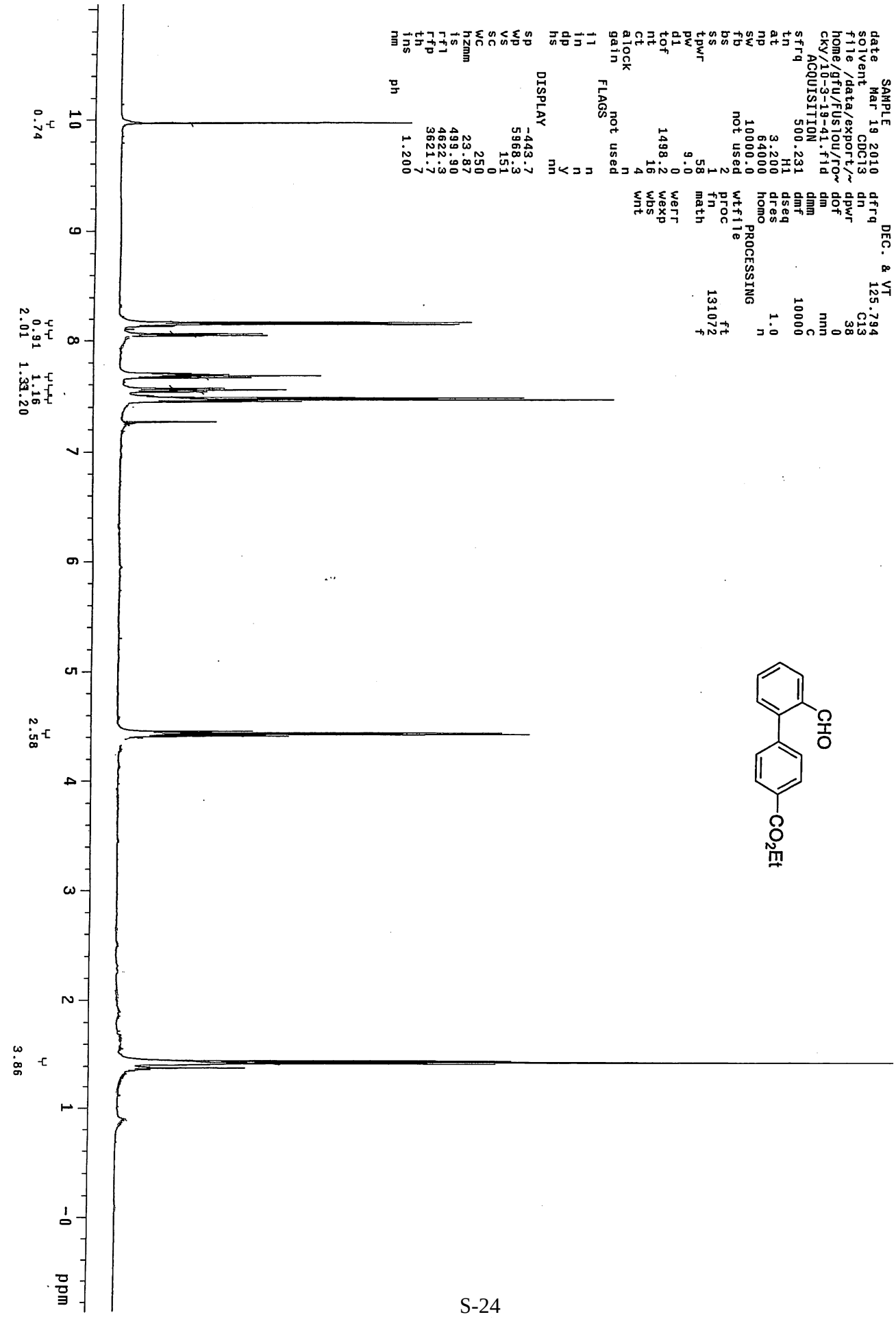


Table 3, entry 3

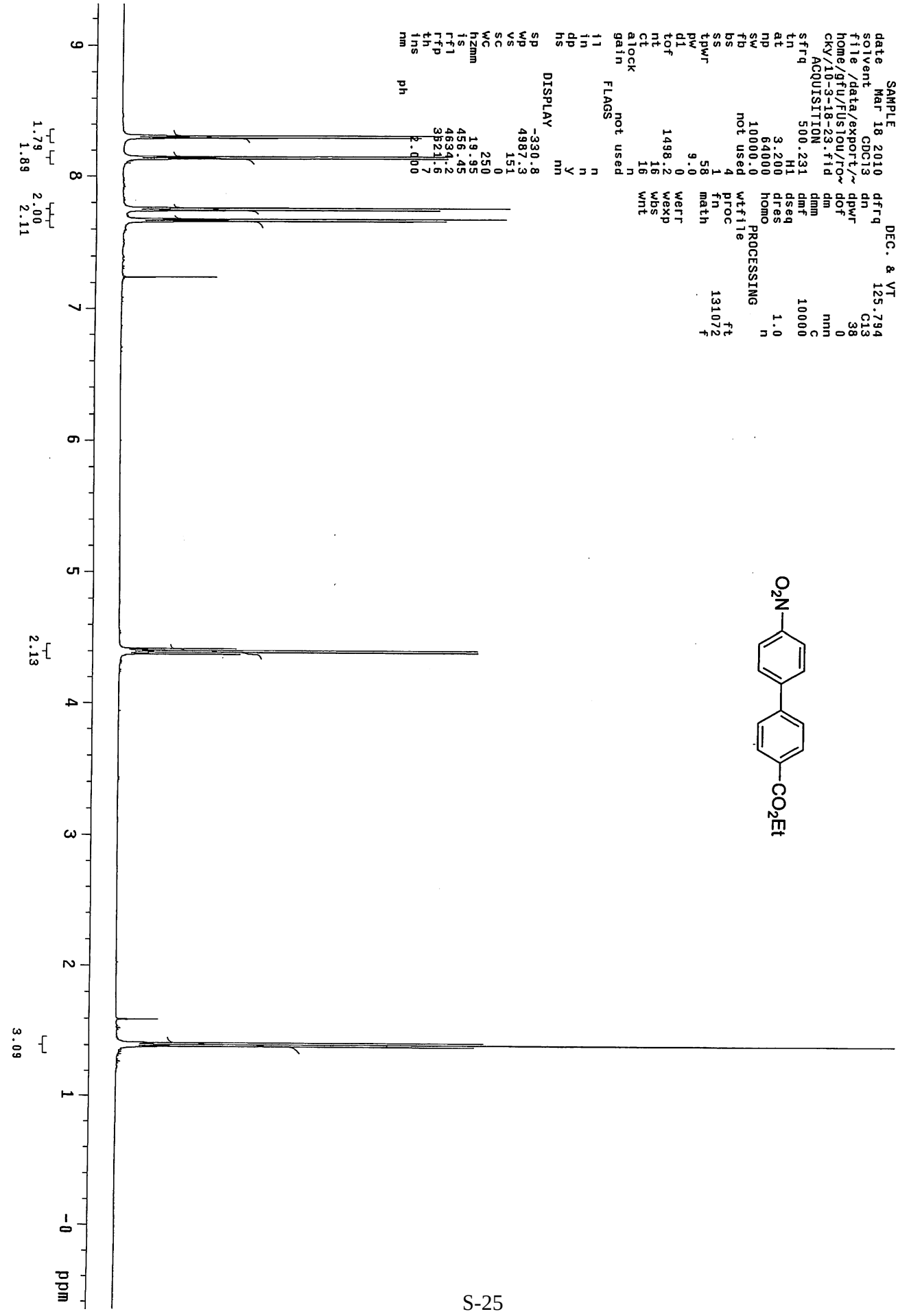


Table 3, entry 4

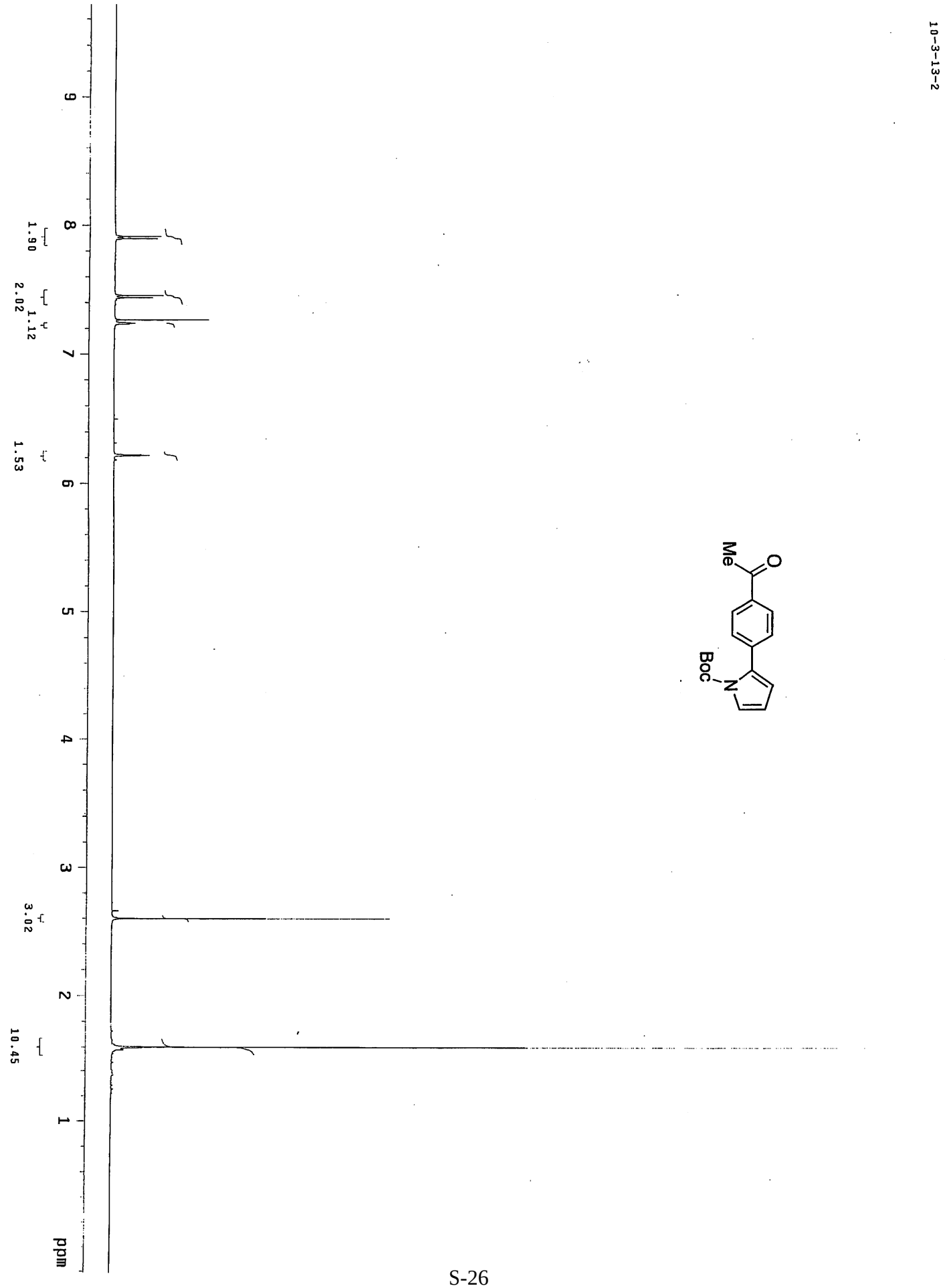


Table 3, entry 5

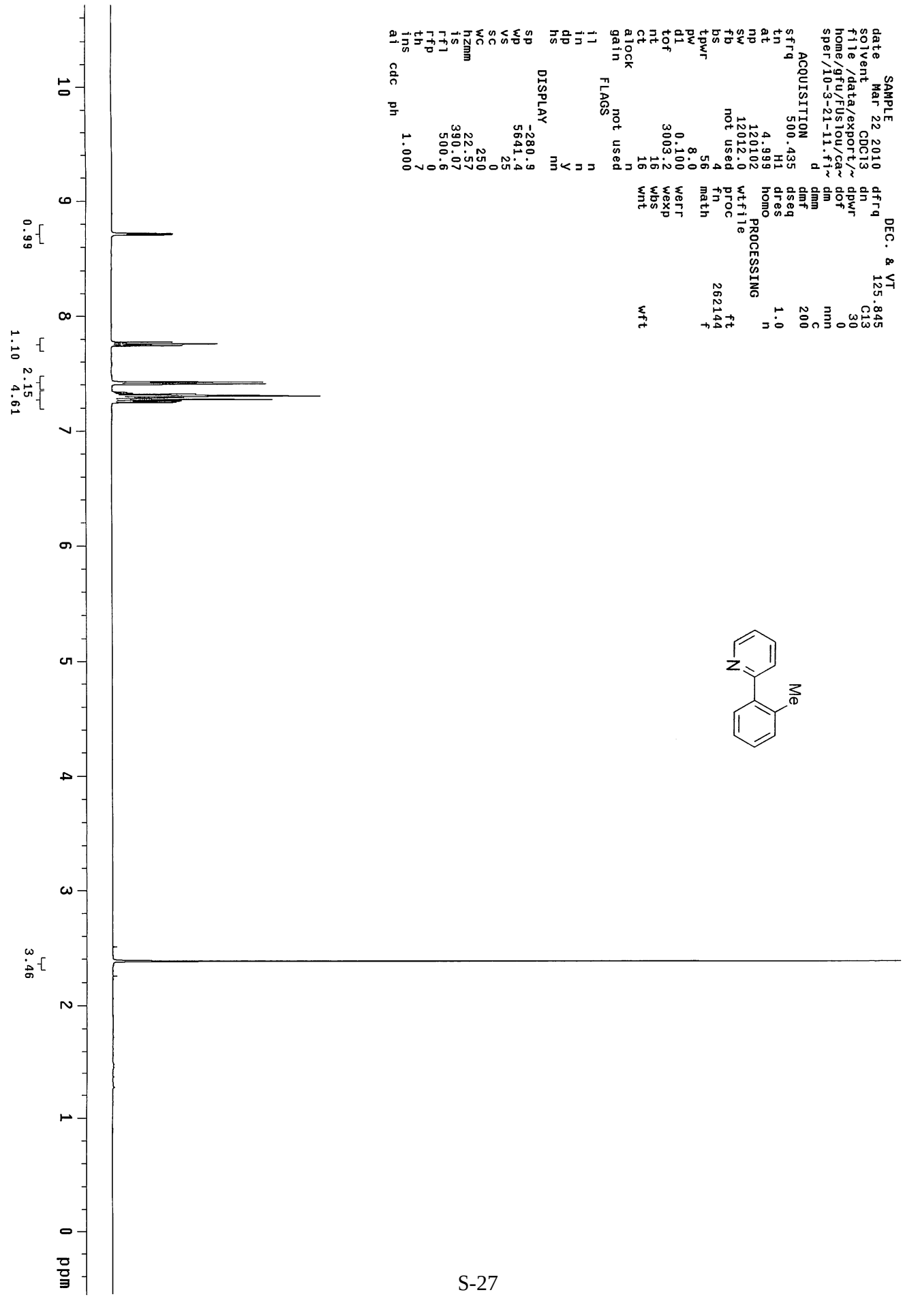


Table 3, entry 6

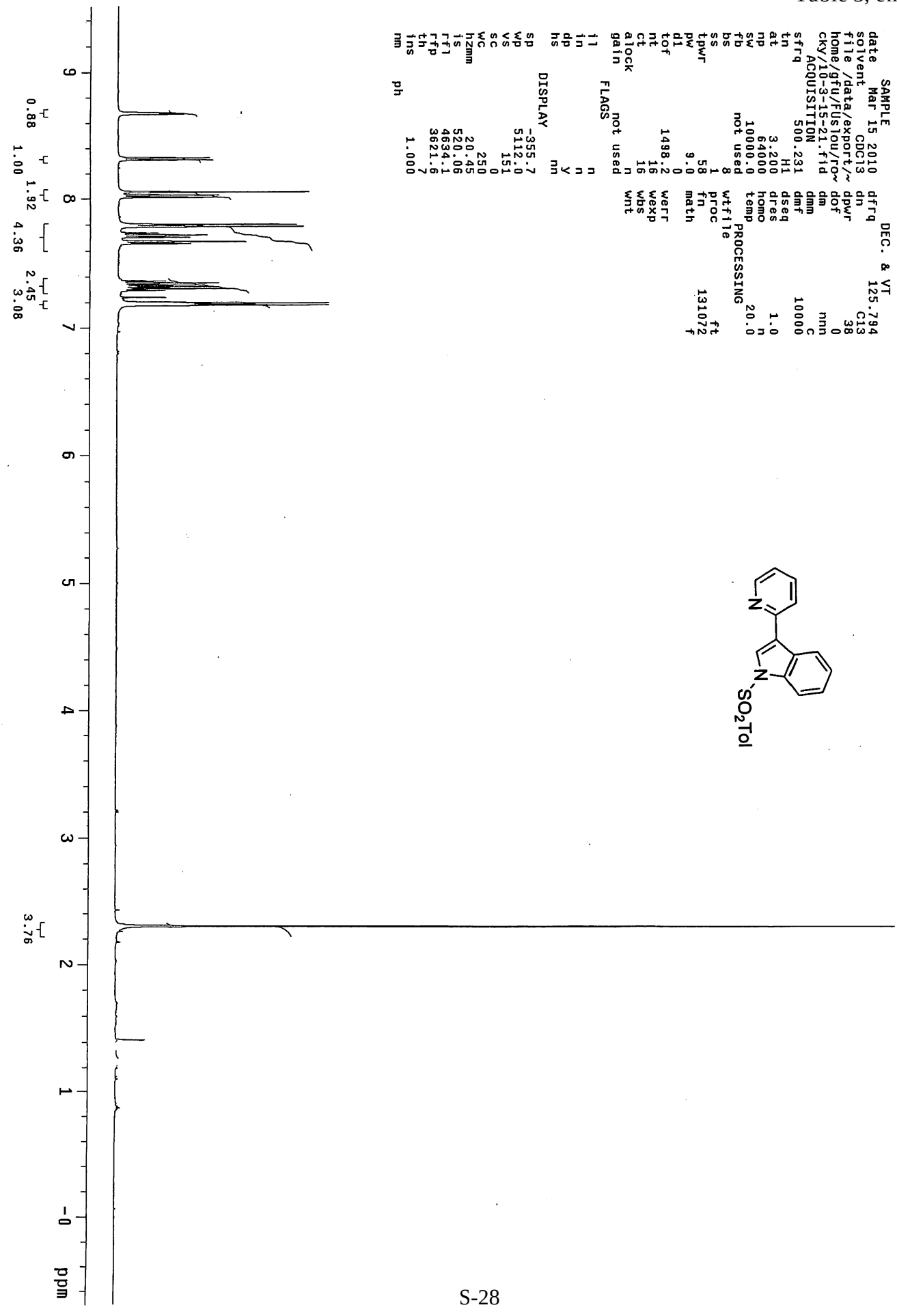


Table 3, entry 7