Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information for:

Total Synthesis of Ascididemin via Anionic Cascade Ring Closure

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Table of contents:

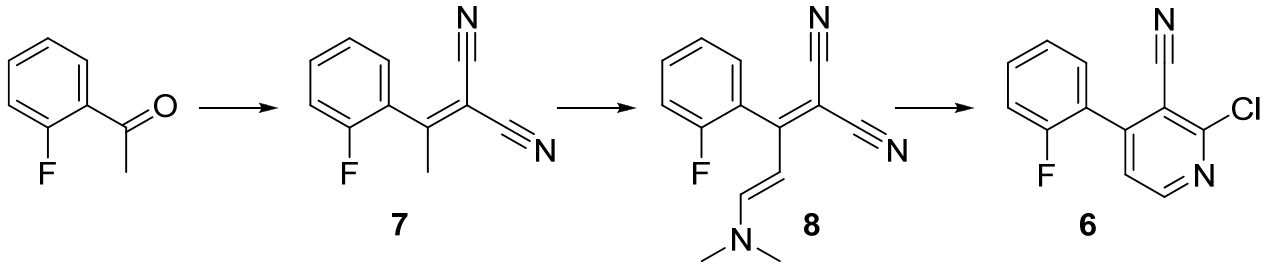
Materials and Methods.

Starting materials and reagents were obtained from commercial suppliers and used without further purifications. Syringes which were used to transfer anhydrous solvents or reagents were purged with nitrogen prior to use. Other solvents were analytical or HPLC grade and were used as received. *n*-BuLi was titrated following the procedure described by Burchat and co-workers.[1] Yields refer to isolated compounds estimated to be > 95 % pure as determined by ^{1}H NMR (25 °C). Thin-layer chromatography (TLC) was carried out on silica gel 60 F_{254} plates from Merck (Germany). Visualization was accomplished by UV lamp (254 nm). Column chromatography was performed on chromatography grade silica 60Å particle size 35–70 micron from Fisher Scientific using the solvent system as stated; where otherwise noted activated neutral alumina (approx. 150 mesh) from Aldrich was used instead. Microwave-assisted synthesis was carried out in a Biotage Initiator apparatus operating in single mode; the microwave cavity producing controlled irradiation at 2.45 GHz (Biotage AB, Uppsala, Sweden). The reactions were run in sealed vessels. These experiments were performed by employing magnetic stirring and a fixed hold time using variable power to reach (during 1-2 min) and then maintain the desired temperature in the vessel for the programmed time period. The temperature was monitored by an IR sensor focused on a point on the reactor vial glass. The IR sensor was calibrated to internal solution reaction temperature by the manufacturer. ^{1}H NMR and ^{13}C NMR spectra were recorded on Varian 300 Mercury instrument, using $CDCl_3$ as deuterated solvent and with the residual solvent as the internal reference; where otherwise noted a mixture $CDCl_3$/CD_3OD (4:1) was used instead. For all NMR experiences the $CDCl_3$ signal was used as the internal lock. Coupling constants (*J* values) are given in Hertz (Hz). Multiplicities of ^{1}H NMR signals are reported as follows: s, singlet; d, doublet; dd, doublet of doublets; ddd, doublet of

[1] Burchat, A. F.; Chong, J. M.; Nielsen, N. *J. Organomet. Chem.* **1997**, *542*, 281–283.

doublets of doublets; m, multiplet; br, broad signal. Melting points (mp) were measured using a MPA100 Optimelt melting point apparatus and are uncorrected. High-resolution mass spectra (HRMS) were obtained using a Micromass Q-TOF 2 instrument. PEPPSI-iPr catalyst ([1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene](3-chloropyridyl)palladium(II) dichloride) was obtained from Aldrich. The following abbreviations are used: MW: microwave; PE: petroleum ether; DCM: dichloromethane; THF: tetrahydrofuran; EtOAc: ethyl acetate.

2-Chloro-4-(2-fluorophenyl)nicotinonitrile (6).



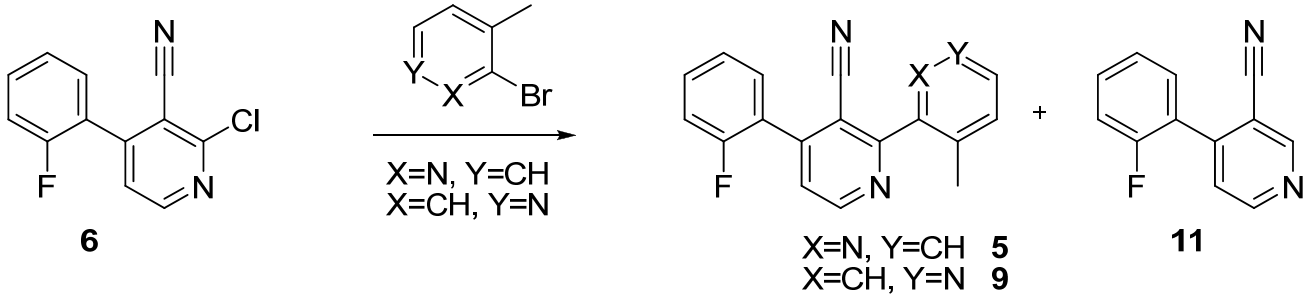
To a solution of 2'-fluoroacetophenone (5.0 g, 36.2 mmol, 1 equiv) in a mixture toluene/glacial acetic acid (15:1, 48 mL) were successively added malononitrile (4.8 g, 72.4 mmol, 2 equiv) and ammonium acetate (2.8 g, 36.2 mmol, 1 equiv) at room temperature. The resulting mixture was heated under reflux conditions using a Dean-Stark trap placed under the reflux condenser to remove the water formed during the reaction. After completion of the reaction (approximately 12 h), solvents were removed *in vacuo* and the residue was taken up in EtOAc (100 mL) and washed successively with water (100 mL) and brine (100 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated *in vacuo* to lead to crude 2-(2-fluorobenzylidene)malononitrile **7** which was used in the next step without any further purifications. An analytically pure sample of malononitrile **7** has been obtained as a pale yellow oil which slowly solidified when stored in the refrigerator; mp 57–60 °C; R_f = 0.5 (EtOAc–PE, 1:4); ^{1}H NMR (300 MHz): δ 7.47–7.57 (m, 1H), 7.17–7.40 (m, 3H), 2.64 (m, 3H); ^{13}C NMR (75 MHz): δ 172.0, 158.6 ($J_{C\text{-}F}$ = 251.9), 133.8 ($J_{C\text{-}F}$ =

8.8), 129.1 ($J_{C\text{-}F}$ = 2.0), 125.1 ($J_{C\text{-}F}$ = 3.5), 124.4 ($J_{C\text{-}F}$ = 13.7), 117.1 ($J_{C\text{-}F}$ = 21.2), 112.2, 112.1, 88.5, 24.7 ($J_{C\text{-}F}$ = 3.5); HRMS (EI): MNa^+ found 209.0506. $C_{11}H_7FN_2Na$ requires 209.0491.

To a solution of crude malononitrile **7** in dry DCM (100 mL) under N_2 was added dimethylformamide dimethyl acetal (8.6 g, 72.4 mmol, 2 equiv) at room temperature. The resulting solution was stirred for 12 h at this temperature then concentrated *in vacuo* to give crude (*E*)-2-[3-(dimethylamino)-1-(2-fluorophenyl)allylidene]malononitrile **8** which was used without any further purifications in the next step. An analytically pure sample of enamine **8** has been obtained as an orange solid; mp 214–216 °C; R_f = 0.35 (EtOAc–PE, 1:1); ^{1}H NMR (300 MHz): δ 7.40–7.50 (m, 1H), 7.13–7.28 (m, 3H) 6.58 (d, *J* = 12.4, 1H), 5.85 (d, *J* = 12.4, 1H), 3.06 (s, 3H), 3.03 (s, 3H); ^{13}C NMR (75 MHz): δ 165.5, 158.8 ($J_{C\text{-}F}$ = 249.1), 155.4, 132.1 ($J_{C\text{-}F}$ = 8.0), 130.8 ($J_{C\text{-}F}$ = 2.6), 124.7 ($J_{C\text{-}F}$ = 3.8), 122.6 ($J_{C\text{-}F}$ = 15.8), 116.6 ($J_{C\text{-}F}$ = 22.9), 116.3, 116.0, 98.1, 66.2, 46.3, 38.2. HRMS (EI): MNa^+ found 264.0890. $C_{14}H_{12}FN_3Na$ requires 264.0913.

To a solution of crude enamine **8** in glacial acetic acid (100 mL) was bubbled hydrochloric acid gas for 5 min at room temperature. The solution was stirred for 3 h at this temperature then solvents were removed *in vacuo*. The residue was taken up in DCM (100 mL) and washed with a saturated aqueous solution of K_2CO_3. The aqueous layer was extracted with DCM (100 mL) then the combined organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated *in vacuo*. The crude product was purified by chromatography column using a gradient elution (EtOAc–PE, 1:4 to 1:3) to provide pure 2-chloro-4-(2-fluorophenyl)nicotinonitrile **6** (6.8 g, 81% over 3 steps) as a pale yellow solid; mp 152–154 °C; R_f = 0.4 (EtOAc–PE, 1:4); ^{1}H NMR (300 MHz): δ 8.59 (d, *J* = 5.0, 1H), 7.39–7.56 (m, 3H), 7.21–7.34 (m, 2H); ^{13}C NMR (75 MHz): δ 159.1 ($J_{C\text{-}F}$ = 250.8), 154.0, 152.0, 150.9, 132.9 ($J_{C\text{-}F}$ = 8.3), 130.7 ($J_{C\text{-}F}$ = 2.0), 125.1 ($J_{C\text{-}F}$ = 3.7), 124.0 ($J_{C\text{-}F}$ = 2.3), 123.0 ($J_{C\text{-}F}$ = 14.3), 116.9 ($J_{C\text{-}F}$ = 21.2), 114.3, 110.9. HRMS (EI): MNa^+ found 255.0128. $C_{12}H_6ClFN_2Na$ requires 255.0101.

Typical procedure for the preparation of bipyridines 5 and 9 (TP1).



To a solution of *i*-PrMgCl (2.0 M in THF, 0.53 equiv) was added a solution of *n*-BuLi (1.49 M in hexanes, 1.05 equiv) at -10 °C under N_2. The mixture was stirred at this temperature for 10 min prior to the addition of a solution of the adequate bromopyridine (1 equiv) in dry THF (1 mL). After 30 min a solution of $ZnCl_2$ (0.5 M in THF, 1.05 equiv) was added over 1 min and the reaction mixture was allowed to warm up to room temperature over 40 min then transferred via cannula to a sealed MW vial containing 2-chloropyridine **6** (0.70 equiv) and PEPPSI-iPr (2 mol%) under N_2. The MW was heated at 120 °C for 1 h under MW conditions. The resulting mixture was filtered through a pad of Celite® and the pad was washed several times with EtOAc. The filtrate was washed with brine, dried over Na_2SO_4, filtered and concentrated *in vacuo*. The crude material was purified by column chromatography to afford the corresponding pure bipyridine. 2-Chloropyridine **6** (< 5%) and 3-cyanopyridine **11** (< 5%) were also recovered.

4-(2-Fluorophenyl)-3'-methyl-2,2'-bipyridine-3-carbonitrile (5). Starting from 2-bromo-3-methylpyridine (200 mg, 1.2 mmol), following typical procedure **TP1** and using EtOAc–DCM (1:1) as eluent, bipyridine **5** was obtained as a pale brownish oil (169 mg, 72 %) which slowly solidified when stored in the refrigerator; mp 123–126 °C; R_f = 0.35 (EtOAc–DCM, 1:1); ^{1}H NMR (300 MHz): δ 8.82 (d, J = 5.2, 1H), 8.55 (dd, J = 4.7 and J = 0.8, 1H), 7.64 (dd, J = 8.0 and J = 0.9,

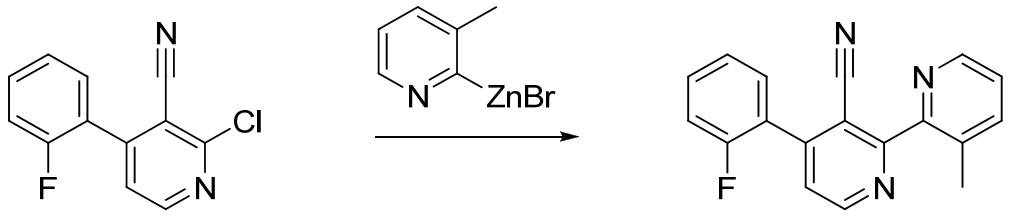
1H), 7.40–7.50 (m, 3H), 7.16–7.31 (m, 3H), 2.37 (s, 3H); ^{13}C NMR (75 MHz): δ 162.2, 159.3 ($J_{C\text{-}F}$ = 250.0), 154.4, 151.3, 148.9, 147.0, 139.2, 132.6, 132.3 ($J_{C\text{-}F}$ = 8.3), 131.0 ($J_{C\text{-}F}$ = 2.3), 124.9 ($J_{C\text{-}F}$ = 3.7), 124.6, 124.1 ($J_{C\text{-}F}$ = 2.0), 123.9 ($J_{C\text{-}F}$ = 14.6), 116.6 ($J_{C\text{-}F}$ = 21.5), 115.7, 110.0, 19.2. HRMS (EI): MNa^+ found 312.0930. $C_{18}H_{12}FN_3Na$ requires 312.0913.

4-(2-Fluorophenyl)-4'-methyl-2,3'-bipyridine-3-carbonitrile (9). Starting from 3-bromo-4-methylpyridine (250 mg, 1.5 mmol), following typical procedure **TP1** and using EtOAc–DCM (1:1) as eluent, bipyridine **9** was obtained as a pale yellow oil (237 mg, 80 %) which slowly solidified when stored in the refrigerator; mp 72–75 °C; R_f = 0.15 (EtOAc–DCM, 1:1); ^{1}H NMR (300 MHz): δ 8.91 (d, J = 5.0, 1H), 8.62 (s, 1H), 8.57 (d, J = 5.0, 1H), 7.46–7.56 (m, 3H), 7.21–7.35 (m, 3H), 2.36 (s, 3H); ^{13}C NMR (75 MHz): δ 159.7, 159.2 ($J_{C\text{-}F}$ = 250.0), 152.3, 151.8*, 149.8, 149.3, 148.7, 148.3 ($J_{C\text{-}F}$ = 13.1), 134.9, 132.6 ($J_{C\text{-}F}$ = 8.3), 130.9 ($J_{C\text{-}F}$ = 2.0), 126.5, 126.3*, 125.1 ($J_{C\text{-}F}$ = 3.8), 124.3 ($J_{C\text{-}F}$ = 1.7), 123.5 ($J_{C\text{-}F}$ = 14.3), 116.7 ($J_{C\text{-}F}$ = 21.5), 115.7, 110.6, 19.9. *signal corresponding to the minor rotamer; HRMS (EI): MNa^+ found 312.0920. $C_{18}H_{12}FN_3Na$ requires 312.0913.

4-(2-Fluorophenyl)nicotinonitrile (11).[2] Off-white solid; mp 100–102 °C; R_f = 0.3 (EtOAc–PE, 1:3); ^{1}H NMR (300 MHz): δ 8.96 (s, 1H), 8.82 (d, J = 5.2, 1H), 7.41–7.56 (m, 3H), 7.21–7.34 (m, 2H); ^{13}C NMR (75 MHz): δ 159.3 ($J_{C\text{-}F}$ = 250.2), 153.7, 152.8, 147.2, 132.5 ($J_{C\text{-}F}$ = 8.3), 130.8 ($J_{C\text{-}F}$ = 2.3), 125.1 ($J_{C\text{-}F}$ = 2.3), 125.0 ($J_{C\text{-}F}$ = 4.0), 123.4 ($J_{C\text{-}F}$ = 14.0), 116.8 ($J_{C\text{-}F}$ = 21.5), 116.3, 110.3.

Synthesis of bipyridine 5 using commercially available zinc reagent.

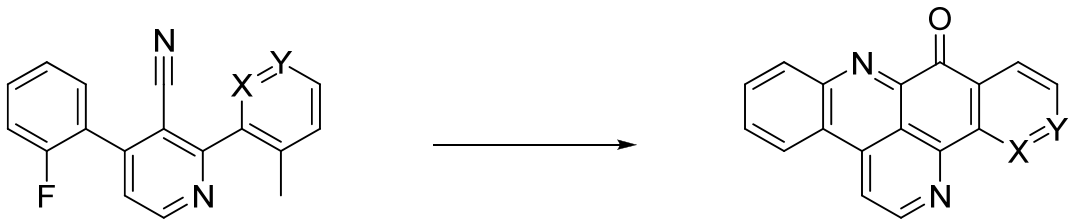
[2] a) Hansen, H. M.; Lysén, M.; Begtrup, M.; Kristensen, J. L. *Tetrahedron* **2005**, *61*, 9955–9960; b) Cailly, T.; Fabis, F.; Rault, S. *Tetrahedron* **2006**, *62*, 5862–5867.



6 **5**

To a sealed MW vial containing 2-chloropyridine **6** (977 mg, 4.2 mmol, 0.7 equiv) and PEPPSI-iPr (82 mg, 2 mol%) under N_2 was added a solution of 3-methyl-2-pyridylzinc bromide (0.5 M in THF, 12 mL, 6.0 mmol, 1 equiv). The MW was heated at 120 °C for 1 h under MW conditions. The resulting mixture was filtered through a pad of Celite® and the pad was washed several times with EtOAc. The filtrate was washed with brine, dried over Na_2SO_4, filtered and concentrated *in vacuo*. The crude material was purified by column chromatography EtOAc–DCM (1:1) as eluent to furnish pure bipyridine **5** (971 mg, 80%) as a brown oil which slowly solidified when stored in the refrigerator.

Typical procedure for the preparation of pyridoacridines 1 and 10 (TP2).



X=N, Y=CH **5**
X=CH, Y=N **9**

X=N, Y=CH **1**
X=CH, Y=N **10**

To a MW vial containing the adequate bipyridine (1 equiv) and dry DMF (with a concentration of 0.05 M) was added NaH (60% dispersion in mineral oil, 10 equiv). After few minutes the vial was sealed and heated at 95 °C for 25 min under MW conditions. After cooling to room temperature the resulting mixture was bubbled for 30 min with O_2, stirred for 3 h under O_2

atmosphere and concentrated *in vacuo*. The crude material was purified by column chromatography on activated neutral alumina to afford the corresponding pure pyridoacridine.

Ascididemin (1).[3] Starting from bipyridine **5** (200 mg, 0.69 mmol), following typical procedure **TP2** and using 100% DCM as eluent then a gradient elution (DCM–MeOH, 100:1 to 10:1), ascididemin (**1**) was obtained as pale yellow flakes (136 mg, 69%); mp 302–304 °C; R_f = 0.3 DCM–MeOH (20:1); ^{1}H NMR (300 MHz): δ 9.28 (d, *J* = 5.6, 1H), 9.17 (dd, *J* = 4.7 and *J* = 1.9, 1H), 8.80 (dd, *J* = 8.0 and *J* = 1.9, 1H), 8.70 (dd, *J* = 8.0 and *J* = 1.1, 1H), 8.64 (dd, *J* = 8.0 and *J* = 1.1, 1H), 8.55 (d, *J* = 5.6, 1H), 8.02 (ddd, *J* = 8.0 and *J* = 7.1 and *J* = 1.4, 1H), 7.95 (ddd, *J* = 8.0 and *J* = 7.1 and *J* = 1.4, 1H), 7.67 (dd, *J* = 8.0 and *J* = 4.7, 1H); ^{13}C NMR (75 MHz): δ 181.9, 155.7, 152.5, 149.92, 149.86, 146.1, 145.9, 138.2, 136.8, 133.4, 132.1, 131.1, 129.2, 125.9, 123.7, 123.1, 118.3, 117.1.

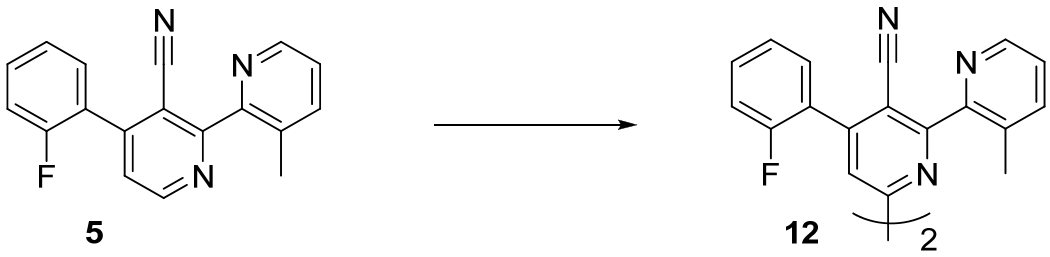
9H-Quino[4,3,2-*de*][1,9]phenanthrolin-9-one (10). Starting from bipyridine **9** (63 mg, 0.22 mmol), following typical procedure **TP2** and using 100% DCM as eluent then a gradient elution (DCM–MeOH, 100:1 to 20:1), 9H-quino[4,3,2-*de*][1,9]phenanthrolin-9-one **10** was obtained as a yellow solid (40 mg, 65%); mp >308 °C (dec); R_f = 0.5 DCM–MeOH (30:1); ^{1}H NMR (300 MHz): δ 10.20 (s, 1H), 9.14 (d, *J* = 5.6, 1H), 9.02 (d, *J* = 5.2, 1H), 8.70 (dd, *J* = 8.2 and *J* = 1.3, 1H), 8.65 (dd, *J* = 8.2 and *J* = 1.3, 1H), 8.50 (d, *J* = 5.6, 1H), 8.26 (d, *J* = 5.2, 1H), 8.04 (ddd, *J* = 8.0 and *J* = 7.1 and *J* = 1.1, 1H), 7.97 (ddd, *J* = 8.0 and *J* = 7.1 and *J* = 1.1, 1H); ^{1}H NMR [300 MHz, $CDCl_3$/CD_3OD (4:1)]: δ 10.03 (s, 1H), 9.03 (d, *J* = 5.8, 1H), 8.87 (d, *J* = 5.2, 1H), 8.63 (dd, *J* = 8.2 and *J* = 0.8, 1H), 8.49 (dd, *J* = 8.2 and *J* = 0.8, 1H), 8.44 (d, *J* = 5.8, 1H), 8.15 (d, *J* = 5.1, 1H),

[3] a) Kobayashi, J.; Cheng, J.-F.; Nakamura, H.; Ohizumi, Y. *Tetrahedron Lett.* **1988**, *29*, 1177–1180; b) Moody, C. J.; Rees, C. W.; Thomas, R. *Tetrahedron* **1992**, *48*, 3589–3602.

7.96 (ddd, J = 8.2 and J = 7.2 and J = 1.4, 1H), 7.89 (ddd, J = 8.2 and J = 7.2 and J = 1.4, 1H);^{13}C NMR [75 MHz, $CDCl_3/CD_3OD$ (4:1)]: δ 181.7, 151.8, 149.3, 148.7, 148.4, 145.7, 145.6, 138.2, 137.2, 132.8, 132.3, 131.3, 129.6, 123.8, 123.2, 120.3, 117.3, 116.5; HRMS (EI): MNa^+ found 306.0651. $C_{18}H_9N_3ONa$ requires 306.0643.

Attempts for preparation of 12-deoxyascididemin (4). Synthesis of a dimeric derivative 12.

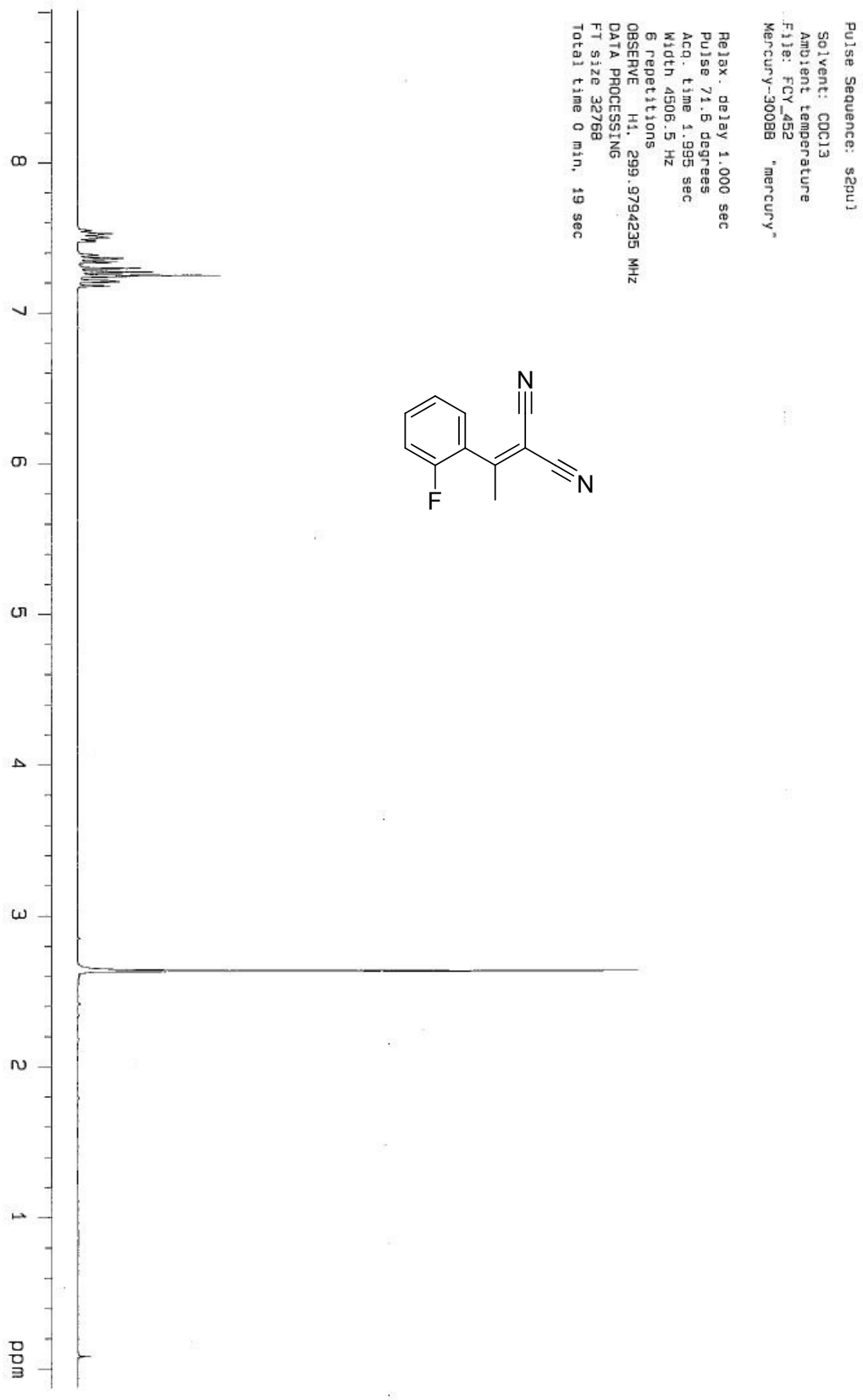
To promote the formation of **1** several different bases (t-BuOK, NaHMDS in DMSO or DMF. LDA, t-BuLi, LiHMDS in THF) was screened followed by immediately oxidation due to the instability of **4**. Attempts to purify **4** before oxidation were unsuccessful. Different oxidation methods (I_2/TBHP or Fe(III)/TBHP, air, MnO_2, Oxone and $KMnO_4$) was also screened. Successful cyclisation attempts were obtained with LiTMP and NaHMDS in THF followed by oxidation with I_2/TBHP or air. This though, only gave ascididemin in 10 % and 25 % yield along with recovered starting material. Treatment with LiTMP also gave dimer **12.**



4',4''-Bis(2-fluorophenyl)-3,3'''-dimethyl-[2,2':6',2'':6'',2'''-quaterpyridine]-3',5''-dicarbonitrile (12). Pale beige solid; mp 290 °C (dec); R_f = 0.85 (EtOAc–DCM, 1:1); ^{1}H NMR (300 MHz): δ 8.70 (s, 1H), 8.69 (s, 1H), 8.61–8.65 (m, 2H), 7.70–7.75 (m, 2H), 7.47–7.59 (m, 4H), 7.23–7.40 (m, 6H), 2.48 (s, 6H); ^{13}C NMR (75 MHz): δ 161.9, 159.4 ($J_{C\text{-}F}$ = 250.5), 155.3, 154.1,

150.4, 147.1, 139.3, 132.8, 132.4 ($J_{C\text{-}F}$ = 8.6), 131.0 ($J_{C\text{-}F}$ = 2.3), 124.9 ($J_{C\text{-}F}$ = 3.7), 124.8, 124.1 ($J_{C\text{-}F}$ = 14.6), 122.3 ($J_{C\text{-}F}$ = 2.0), 116.7 ($J_{C\text{-}F}$ = 21.5), 115.8, 110.8, 19.6.[4]

[4] All ^{13}C signals are counting for two carbons.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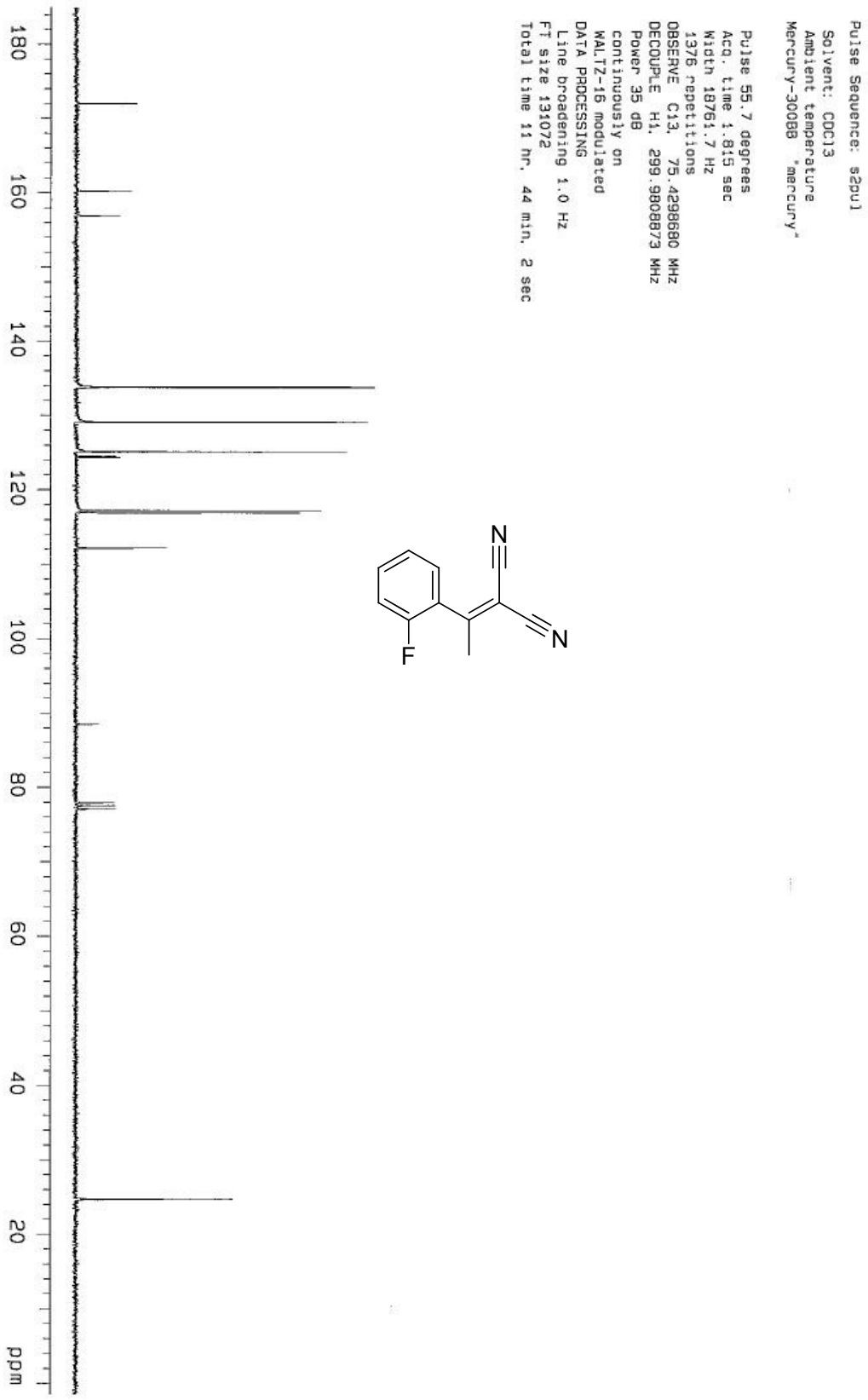

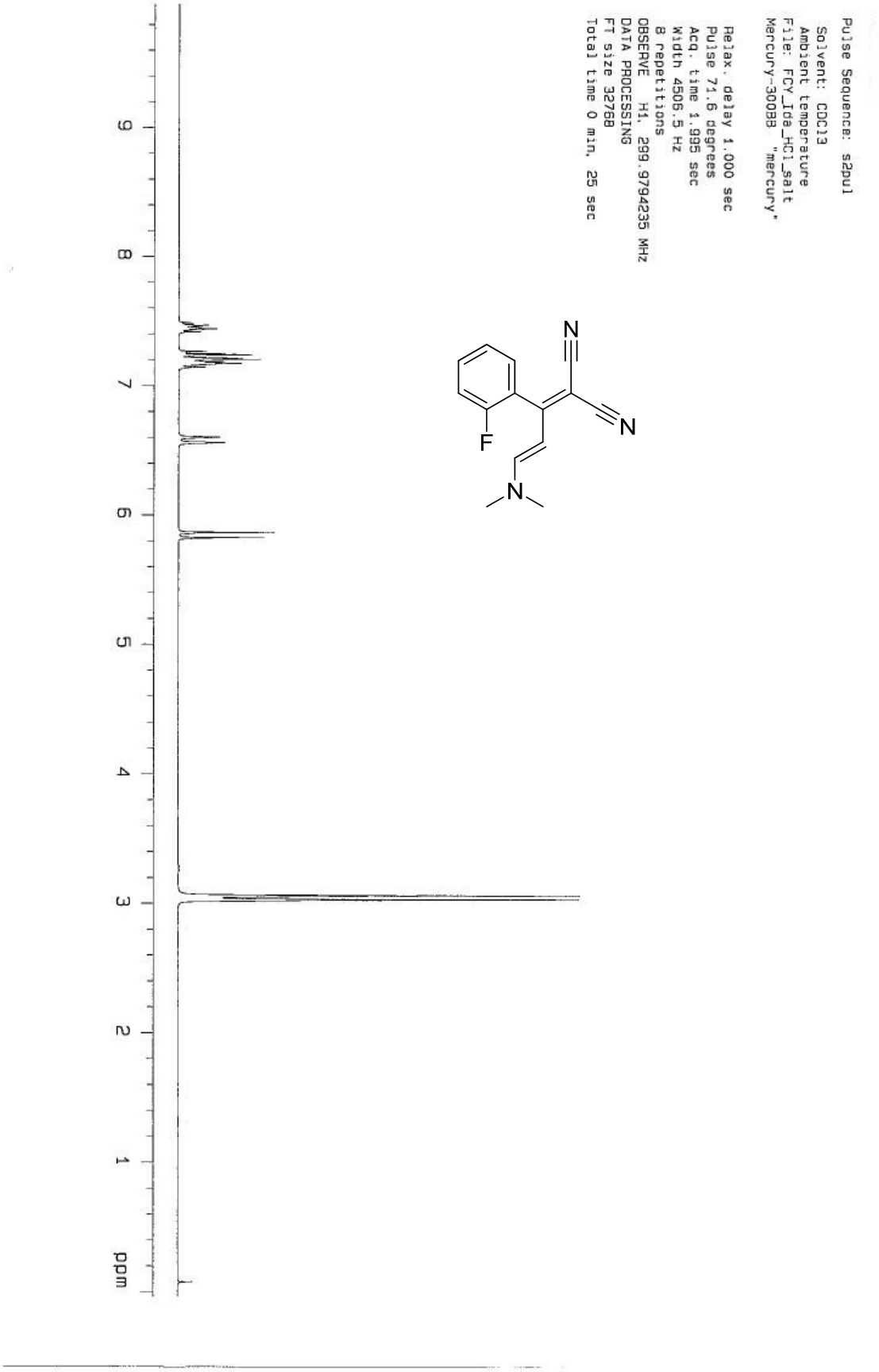
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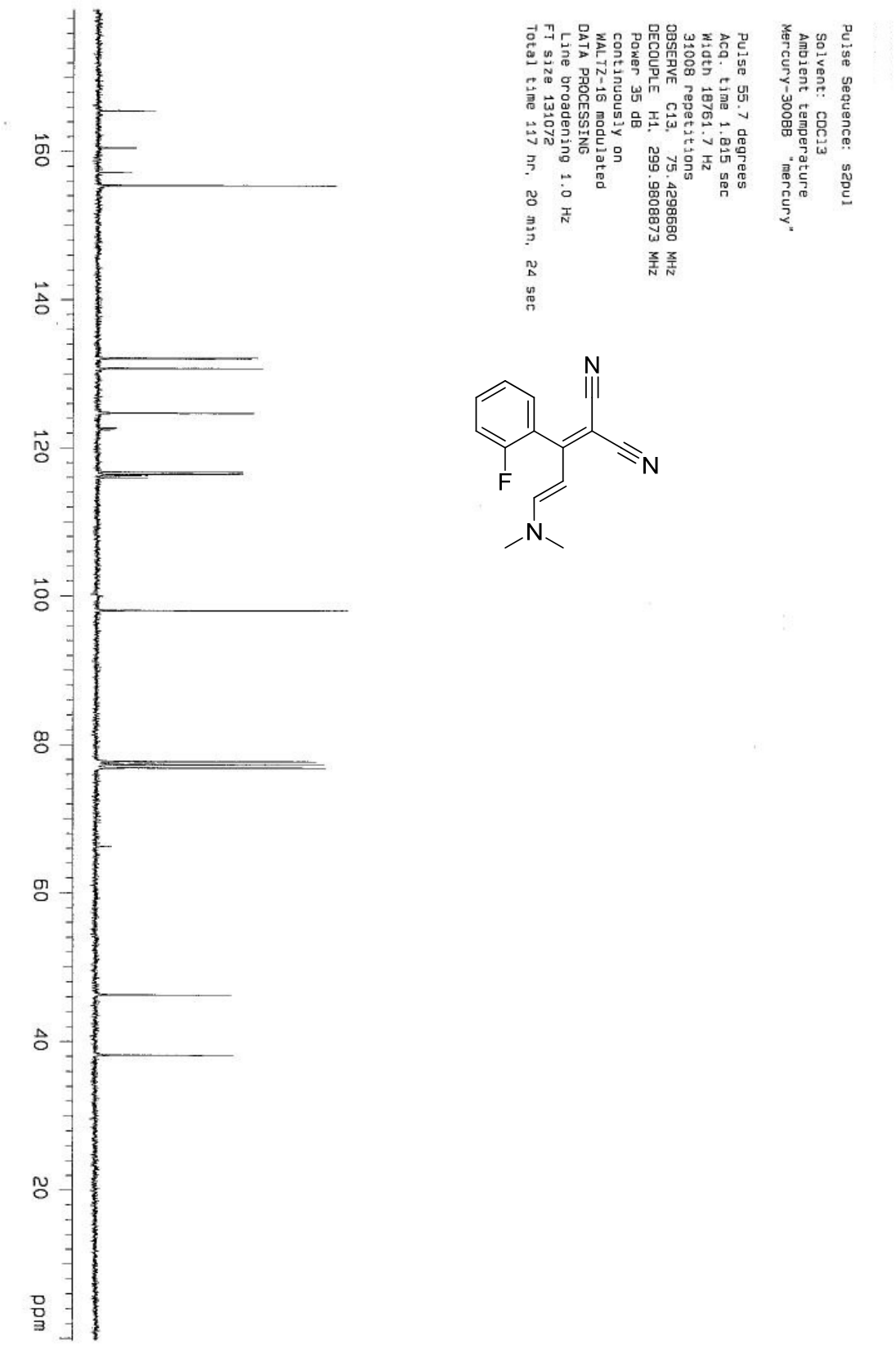
ppm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Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
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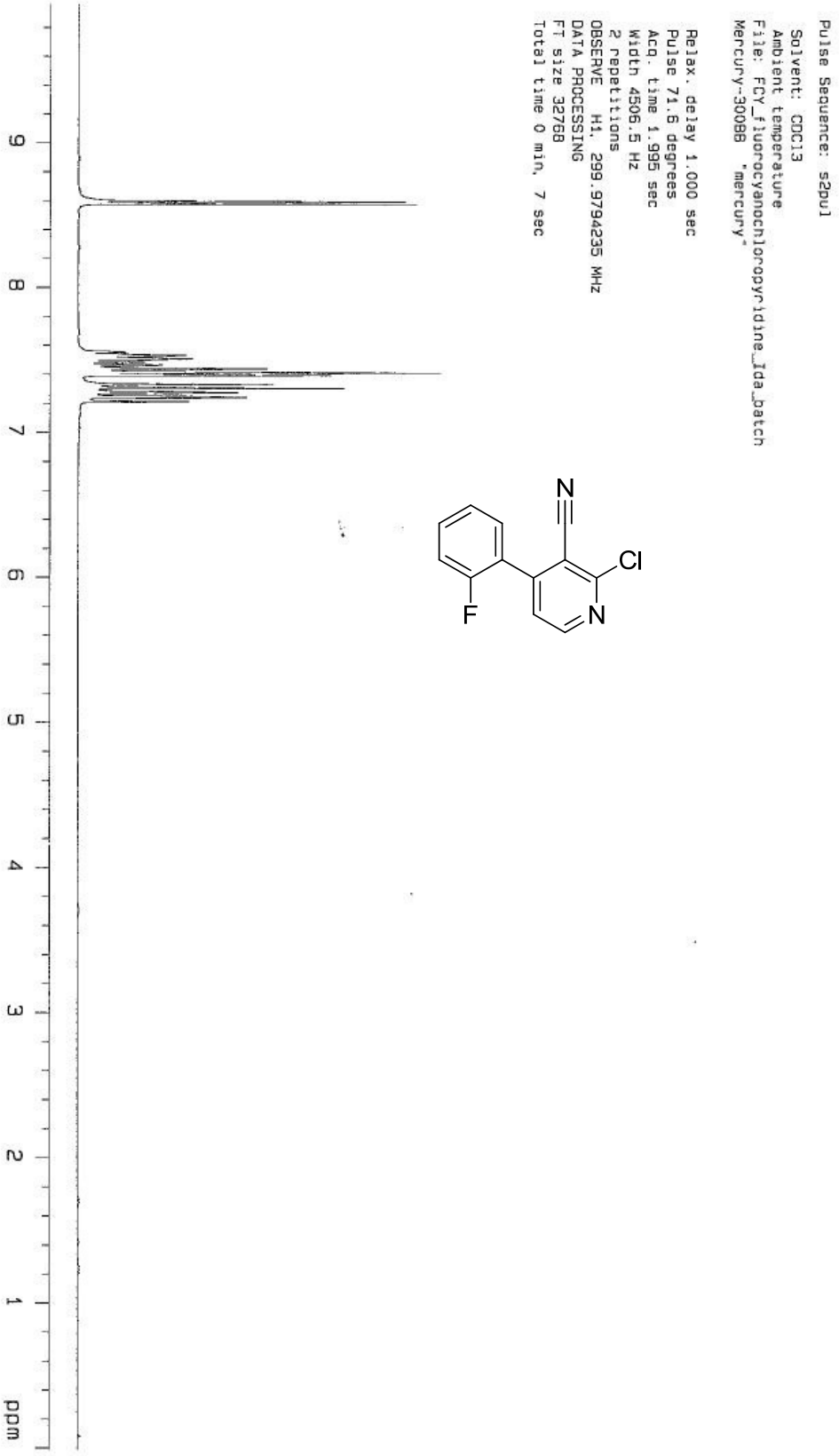
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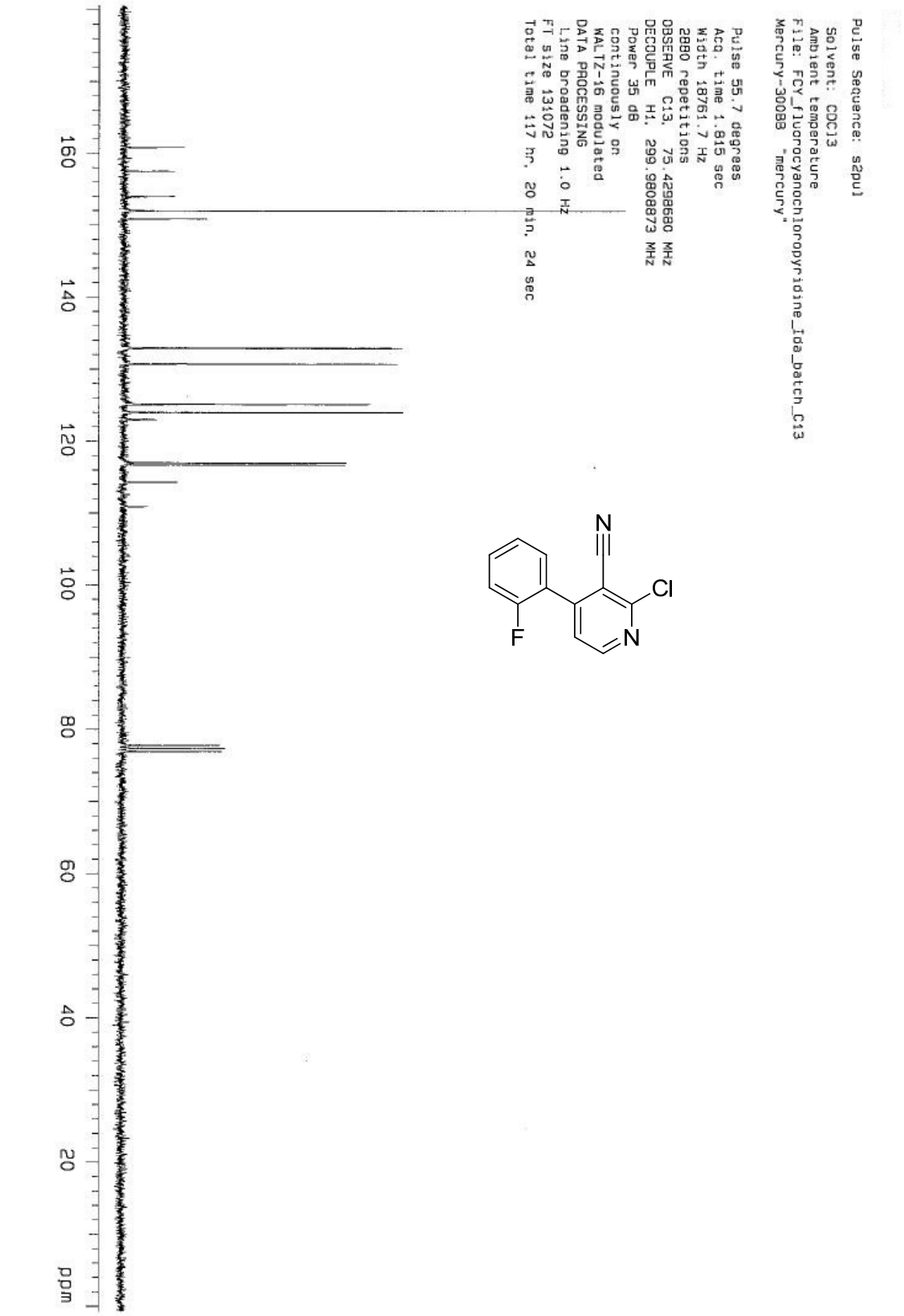
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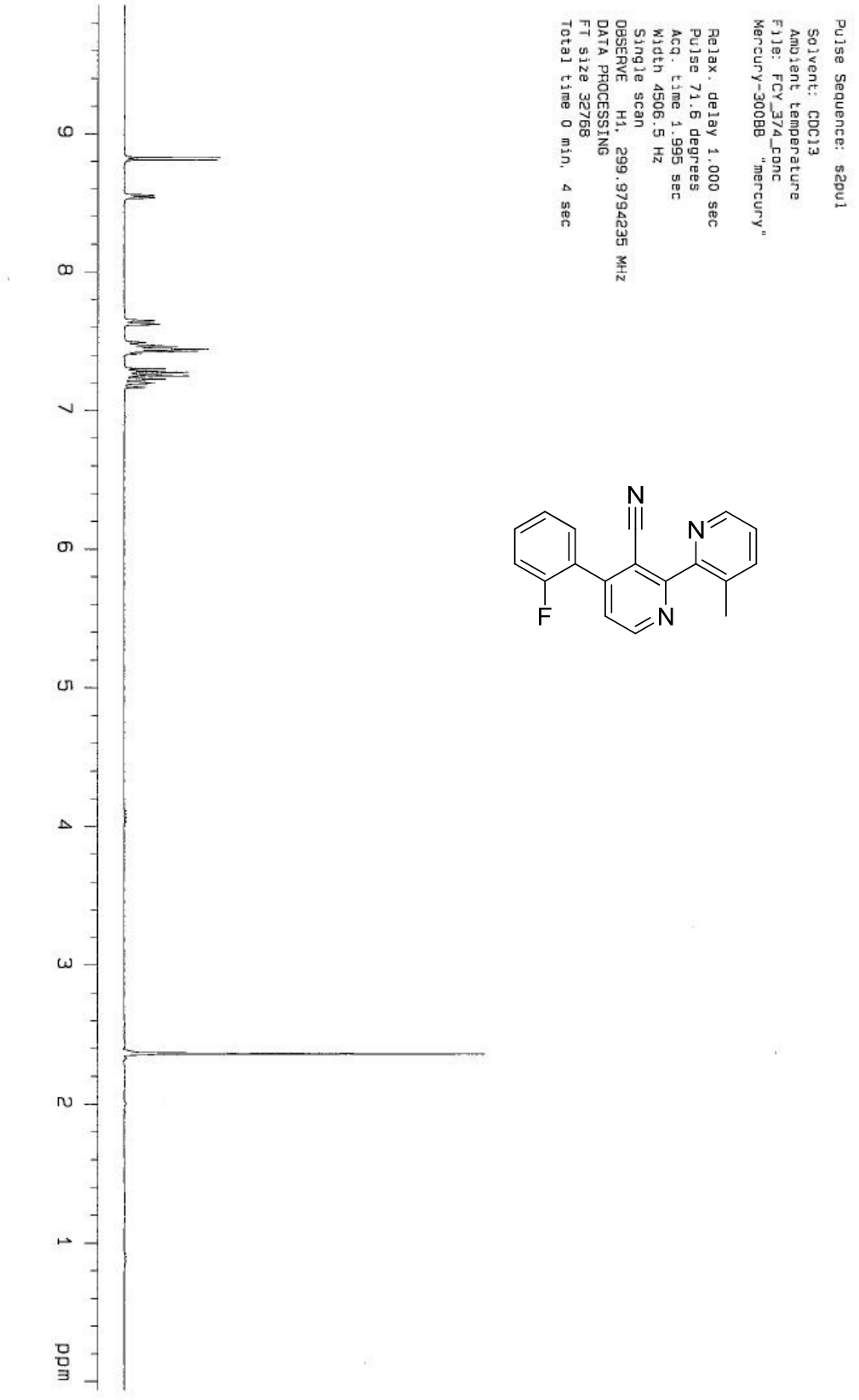
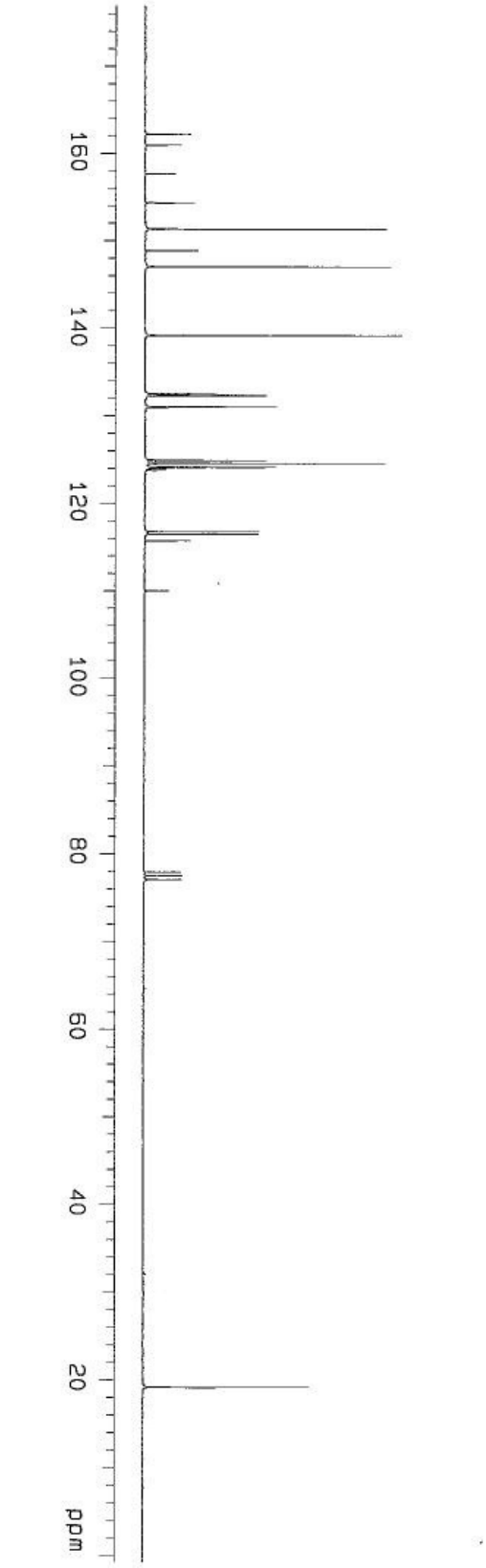
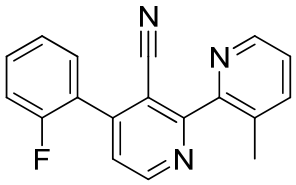
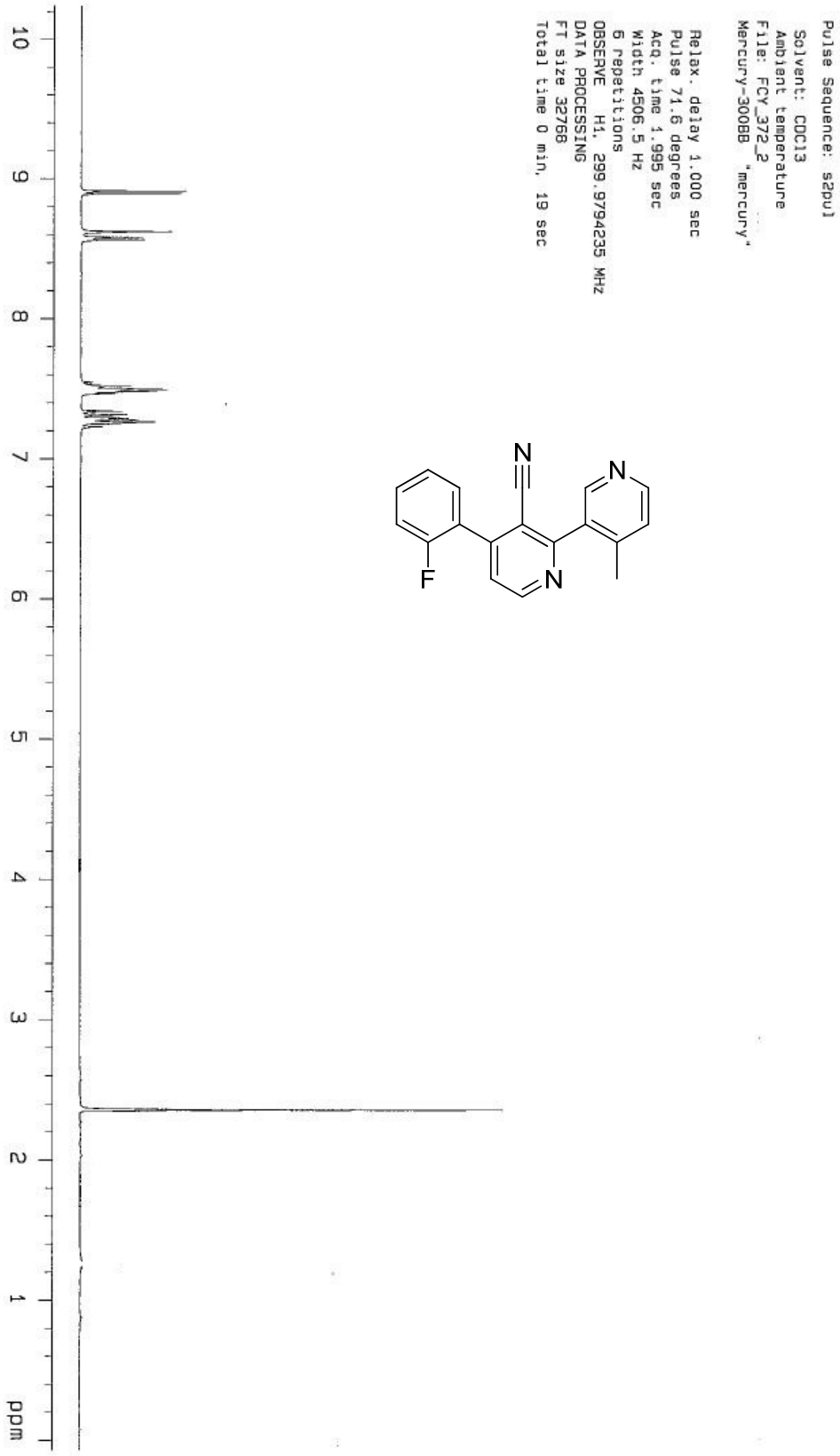
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
2880 repetitions
OBSERVE C13, 75.4298680 MHz
DECOUPLE H1, 299.9808873 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 117 hr, 20 min, 24 sec
N
Cl
F
N
160
140
120
100
80
60
40
20
ppm

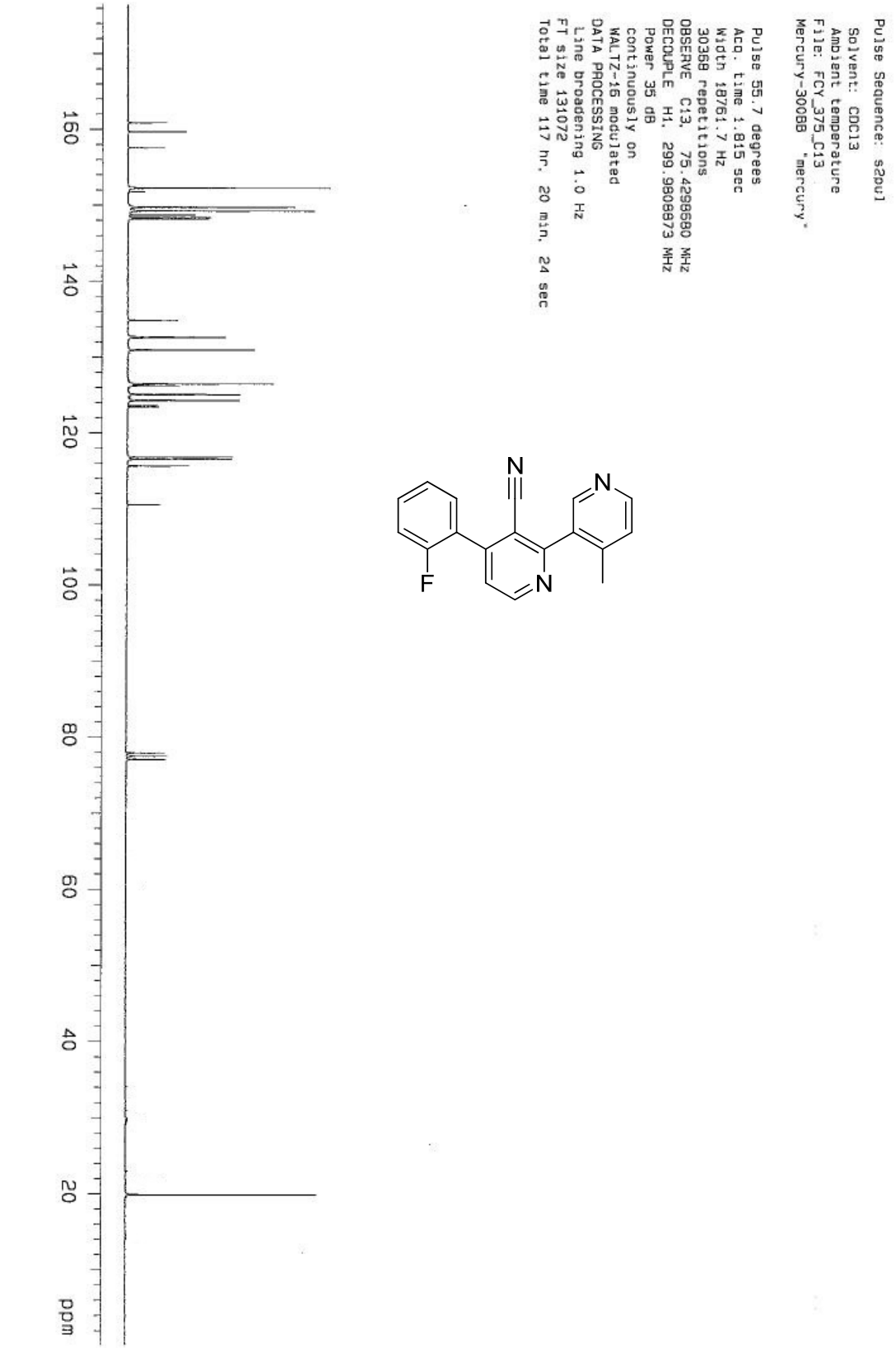

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: FCY_374_conc
Mercury-300BB "mercury"
Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
Single scan
OBSERVE H1, 299.9794235 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 4 sec
N
N
F
N
9
8
7
6
5
4
3
2
1
ppm

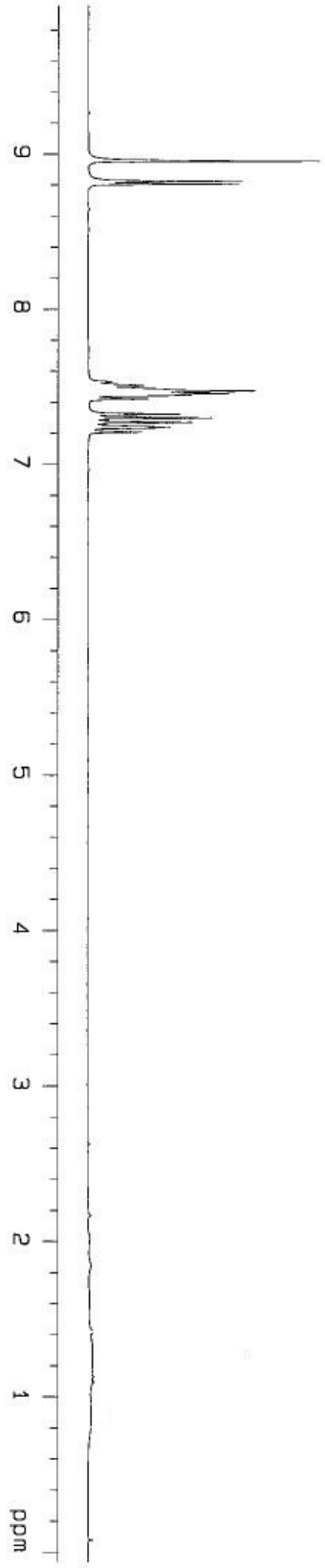



```
Pulse Sequence: s2pul

  Solvent: CDCl3
  Ambient temperature
  File: FCY_374_C13
Mercury-300BB "mercury"

  Pulse 55.7 degrees
  Acq. time 1.815 sec
  Width 18761.7 Hz
  28352 repetitions
OBSERVE   C13,  75.4298680 MHz
DECOUPLE  H1,  299.9808873 MHz
  Power 35 dB
  continuously on
  WALTZ-16 modulated
DATA PROCESSING
  Line broadening 1.0 Hz
  FT size 131072
Total time 117 hr, 20 min, 24 sec
```

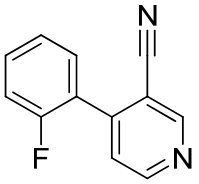


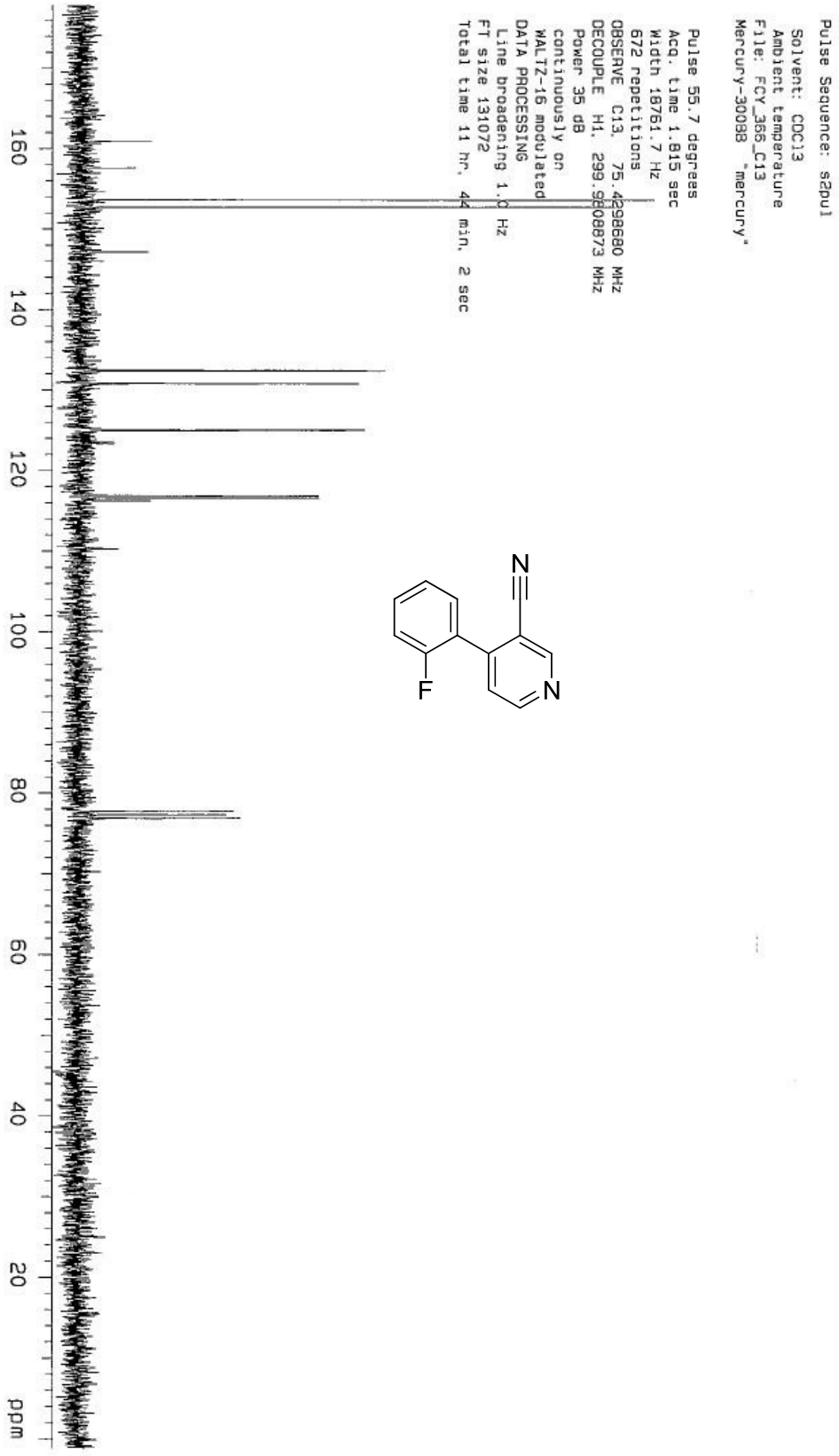


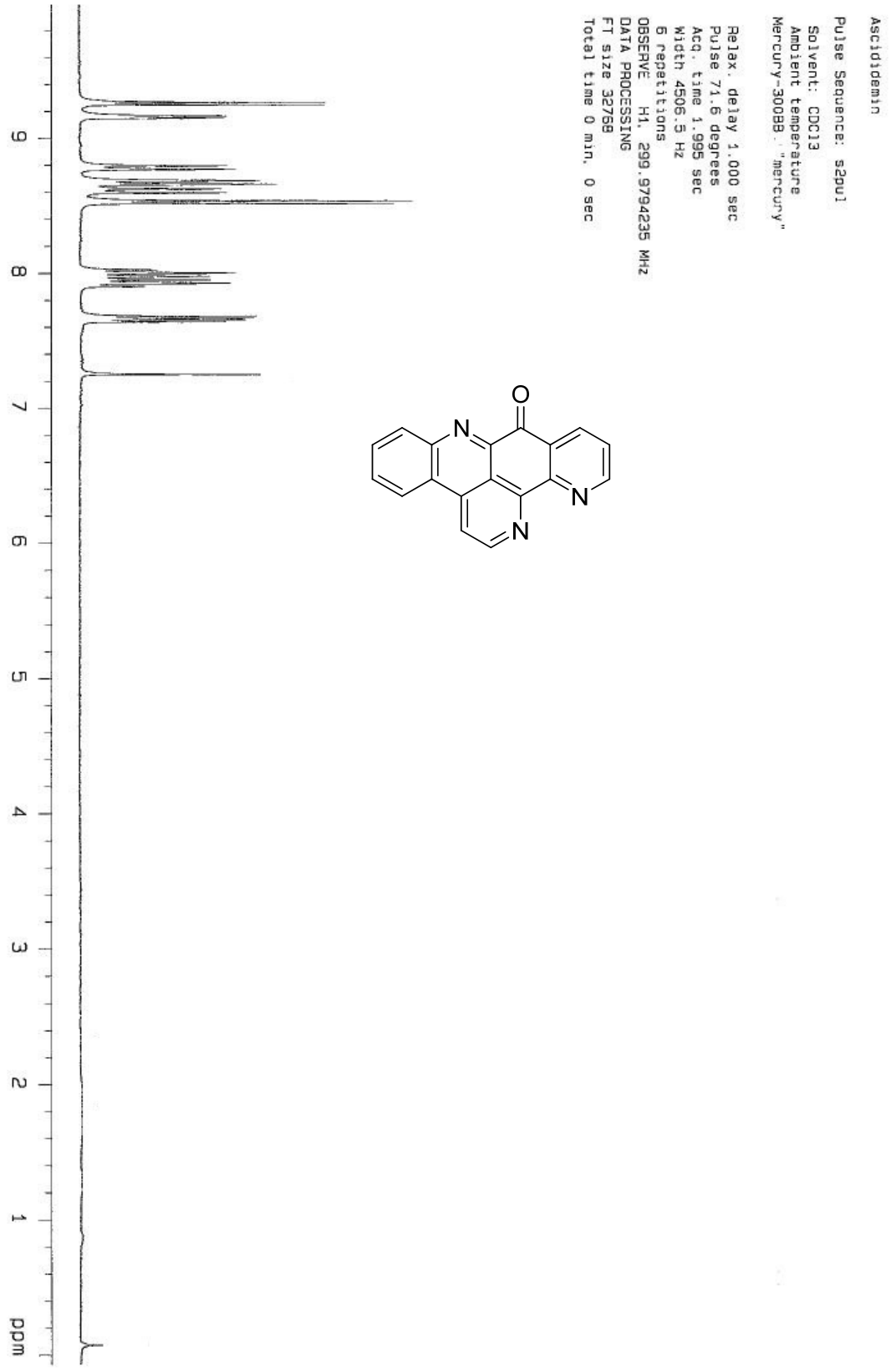

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: FCY_375_C13
Mercury-300BB "mercury"
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
30368 repetitions
OBSERVE C13, 75.4298580 MHz
DECOUPLE H1, 299.9806873 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 117 hr, 20 min, 24 sec
N
N
F
N
160
140
120
100
80
60
40
20
ppm

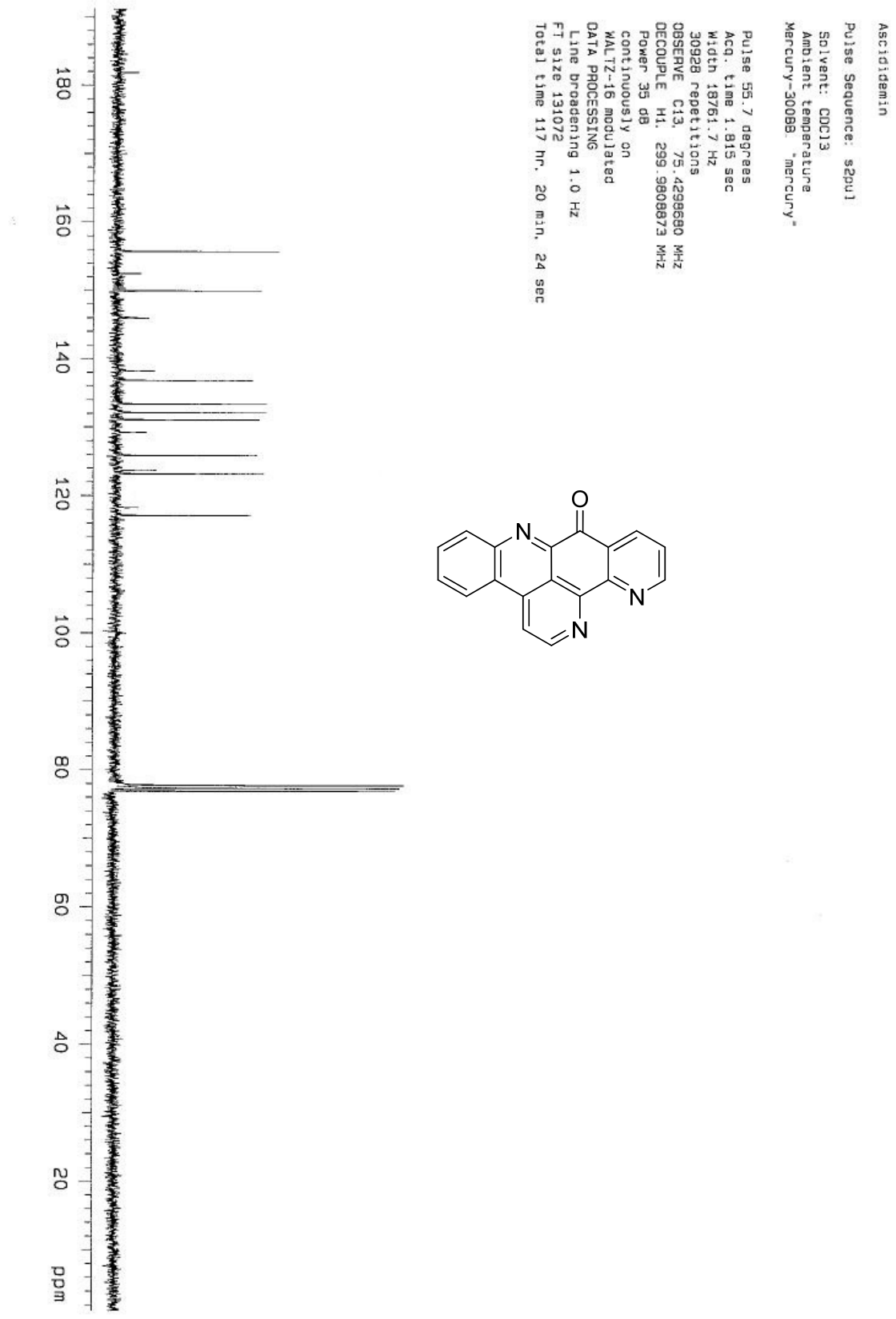


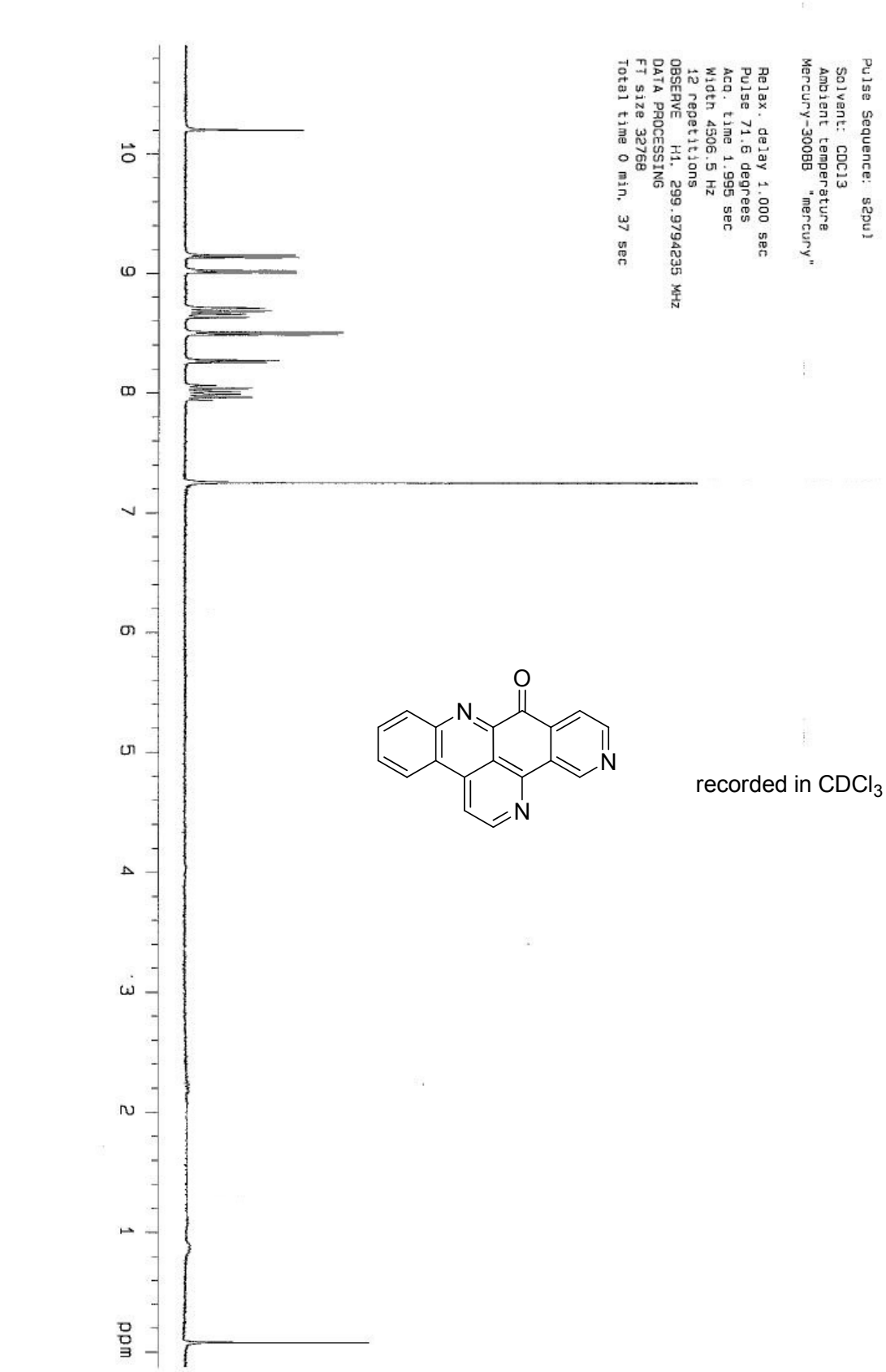

```
Pulse Sequence: s2pul

  Solvent:  CDCl3
  Ambient temperature
  File: FCY_366 .....
Mercury-300BB  "mercury"

Relax. delay 1.000 sec
Pulse 71.6 degrees
  Acq. time 1.995 sec
  Width 4506.5 Hz
  4 repetitions
OBSERVE   H1, 299.9794235 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 13 sec
```



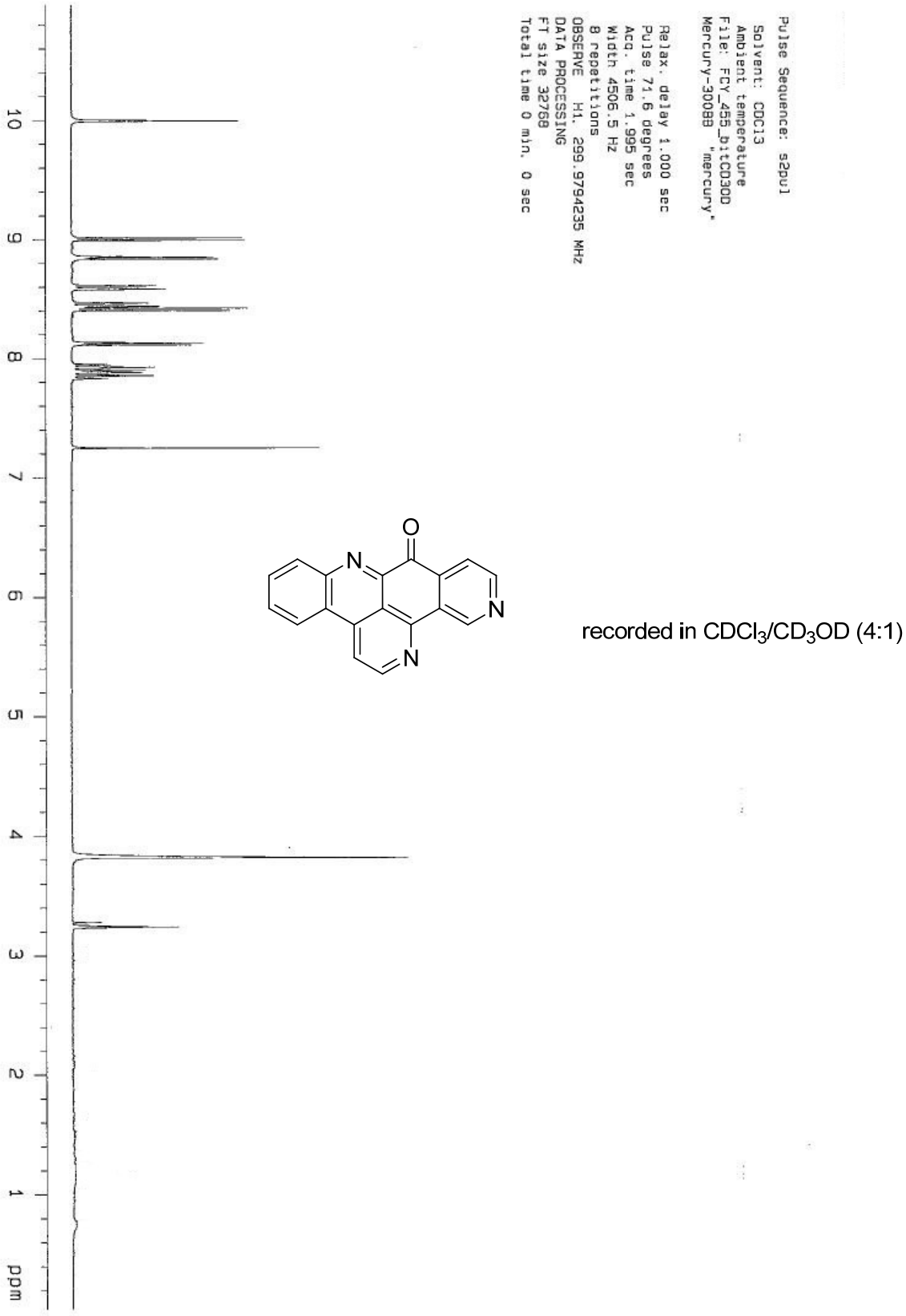


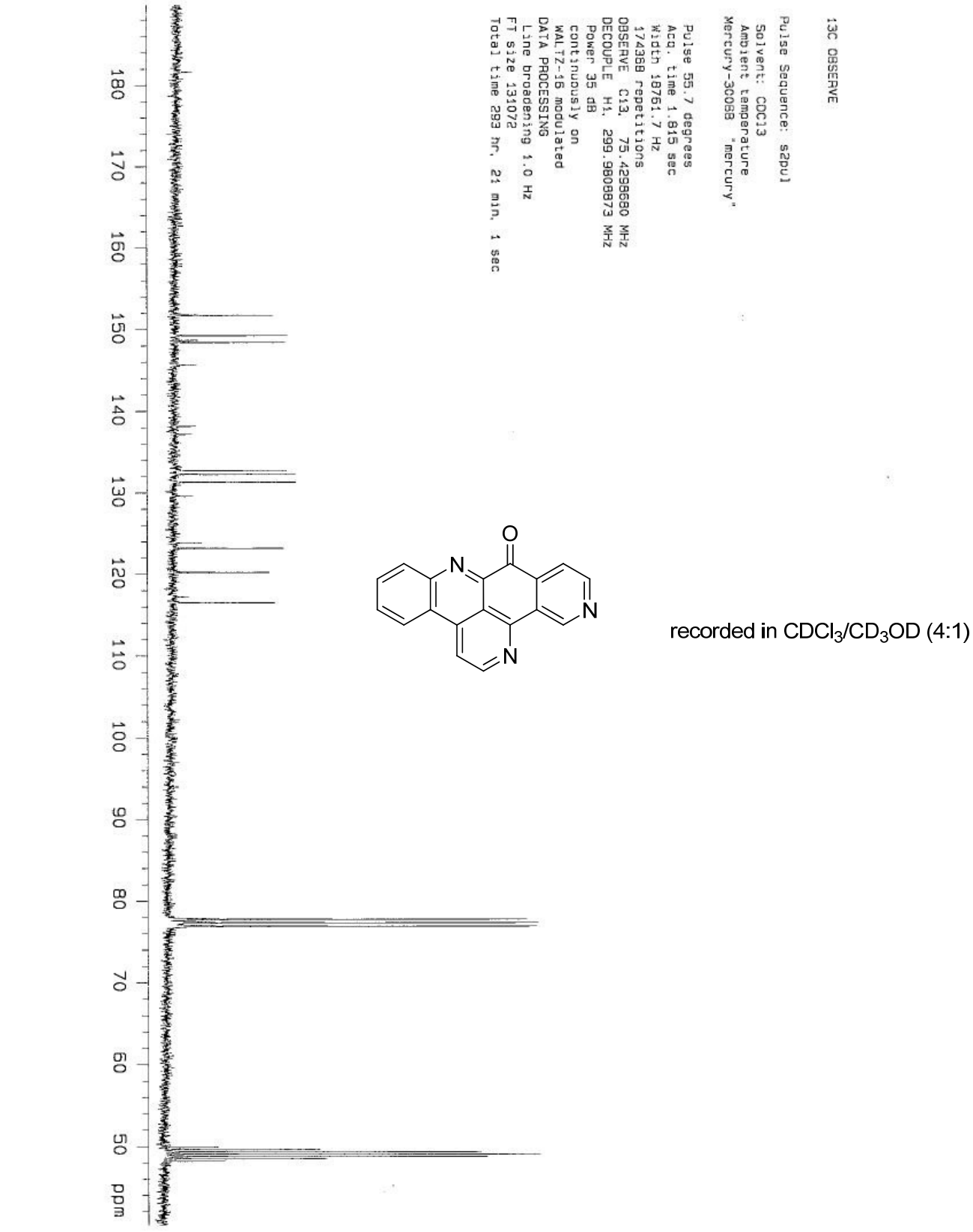

Asciidemin
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB: "mercury"
Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
6 repetitions
OBSERVE H1, 299.9794235 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 0 sec
O
N
N
N
9
8
7
6
5
4
3
2
1
ppm


Acididemin
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB: "mercury"
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
30928 repetitions
OBSERVE C13, 75.4298680 MHz
DECOUPLE H1, 299.9808873 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 117 hr, 20 min, 24 sec
O
N
N
N
180
160
140
120
100
80
60
40
20
ppm

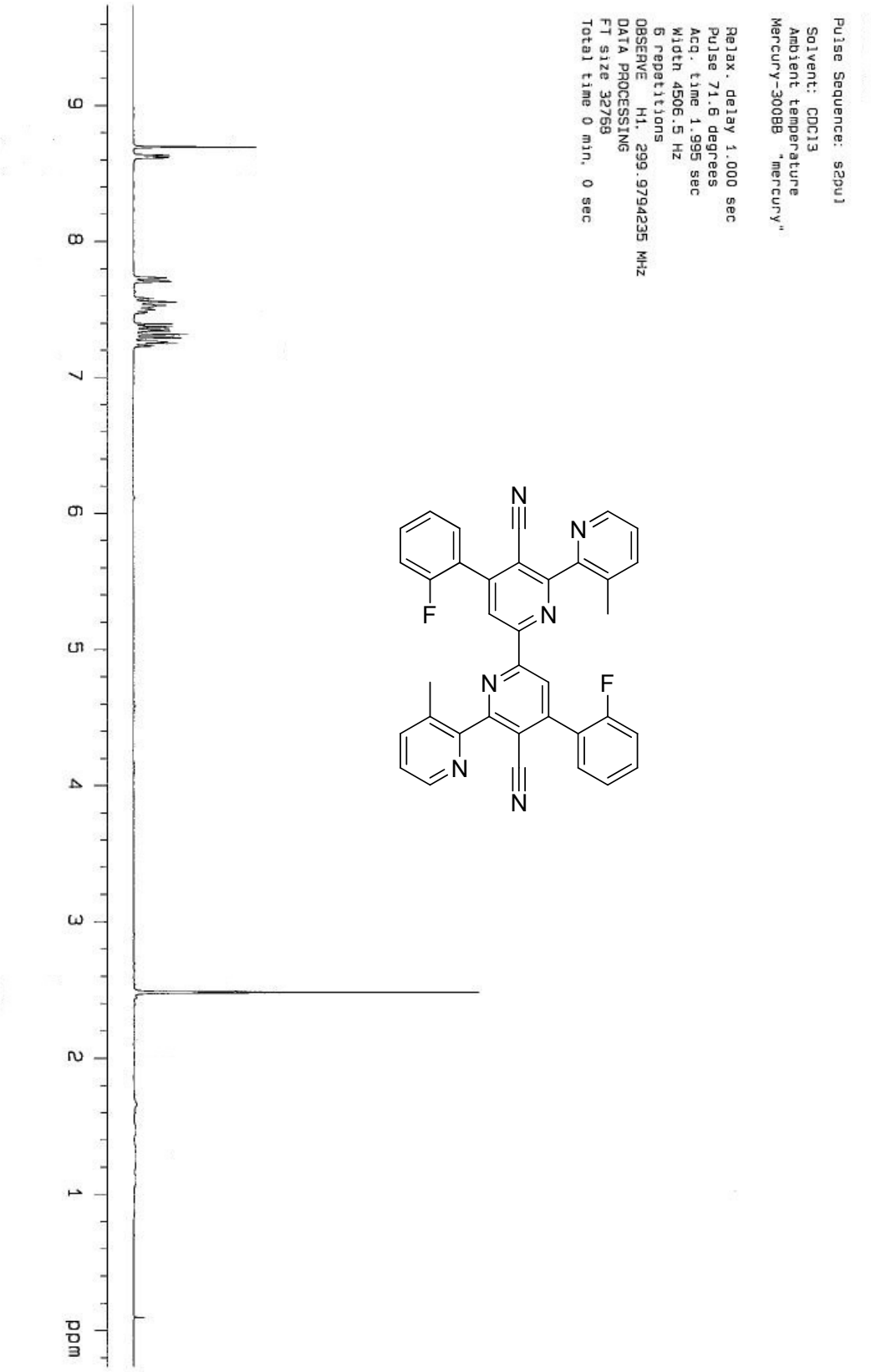


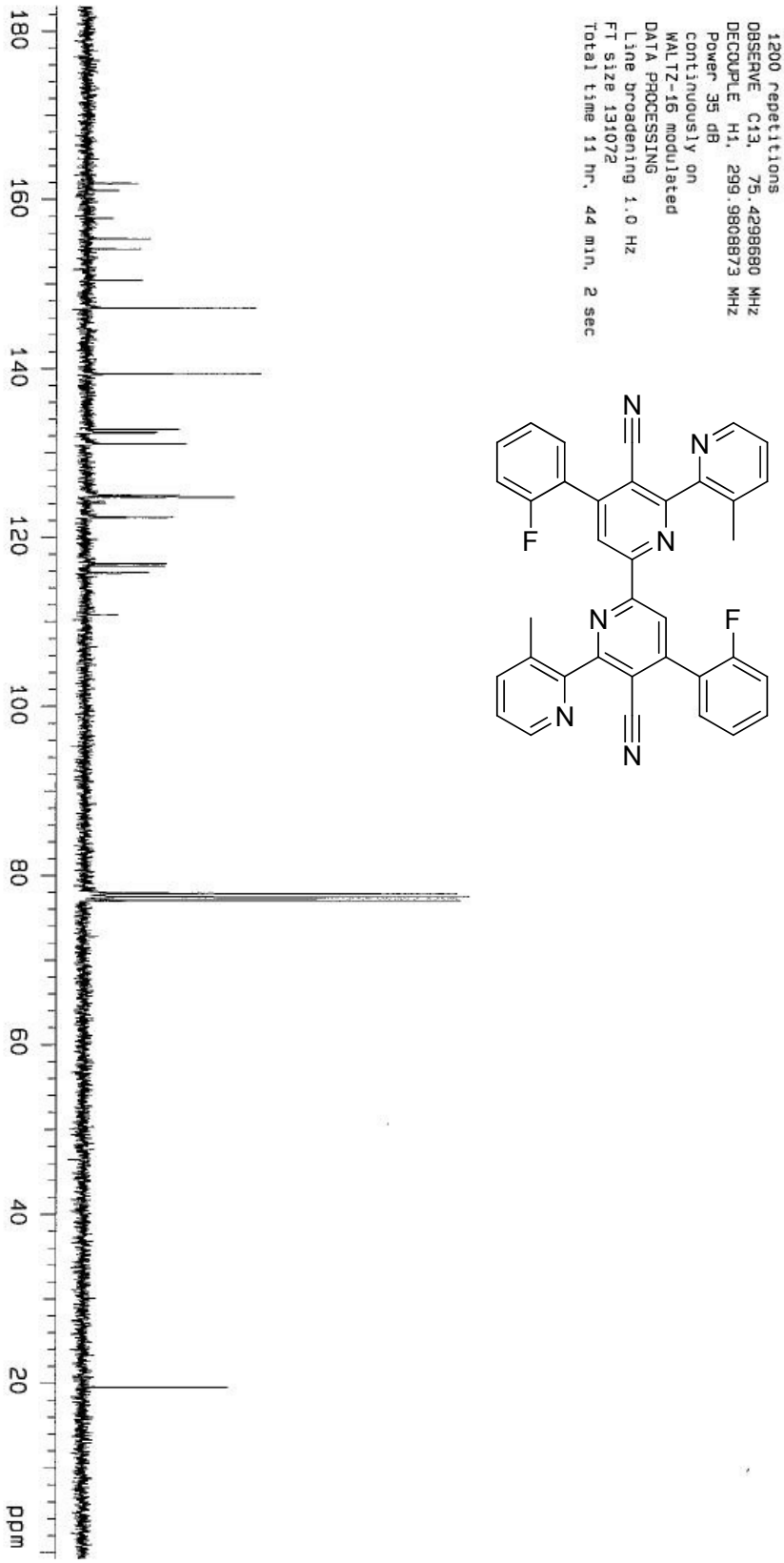
recorded in $CDCl_3$


Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: FCY_455_bitCD3OD
Mercury-300BB "mercury"
Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
8 repetitions
OBSERVE H1, 299.9794235 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 0 sec
O
N
N
N
recorded in CDCl3/CD3OD (4:1)
10
9
8
7
6
5
4
3
2
1
ppm



recorded in $CDCl_3/CD_3OD$ (4:1)


Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury"
Relax. delay 1.000 sec
Pulse 71.6 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
6 repetitions
OBSERVE H1, 299.9794235 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 0 sec
N
N
F
N
N
F
N
N
9
8
7
6
5
4
3
2
1
ppm


Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury"
Pulse 55.7 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
1200 repetitions
OBSERVE C13, 75.4298680 MHz
DECOUPLE H1, 299.9808873 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 11 hr, 44 min, 2 sec
N
N
F
N
N
F
N
N
180
160
140
120
100
80
60
40
20
ppm