Supporting information

Enantioselective synthesis of substituted pyrans *via* amine-catalyzed Michael addition and subsequent enolization/cyclisation

Utpal Das,[a] Chan-Hui Huang[a] and Wenwei Lin*[a]

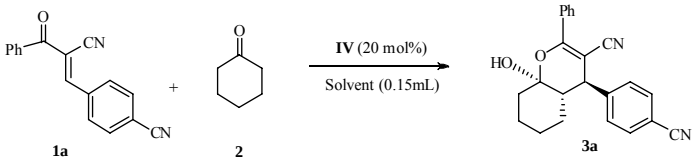
[a] Department of Chemsitry, National Taiwan Normal University

88, Sec. 4, Tingchow Road, Taipei 11677, Taiwan, ROC.

wenweilin@ntnu.edu.tw

General methods: All reagents were used as purchased from commercial suppliers without further purification. IR spectra were recorded on a Perkin Elmer 500 spectrometer. NMR spectra were recorded on a Bruker Avance 400/500 NMR spectrometer. Chemical shifts are reported in δ ppm referenced to an internal TMS standard for ^{1}H NMR and chloroform-*d* (δ 77.0 ppm) for ^{13}C NMR. Enantioselectivities were determined by high performance liquid chromatography (HPLC) analysis. Optical rotations were measured in $CHCl_3$ on a JASCO co. DIP-1000 Digital polarimeter with a 50 mm cell (c given in g/100 mL). HRMS spectra were recorded on JEOL SX-102A. The X-ray diffraction measurements were carried out at 298 K on a KAPPA APEX II CCD area detector system equipped with a graphite monochromator and a Mo-Kα fine-focus sealed tube (*k* = 0.71073 Å). Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Flash-chromatography was performed using Merck silica gel 60 (70–230 mesh).

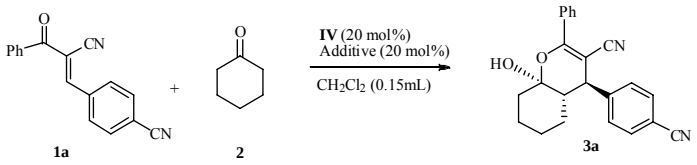
Table 1: Optimization (solvent screening) for the enantioselective synthesis of pyrans using **1a** and **2**[a]



Entry	Solvent	t (d)	dr[b]	Yield (%)[c]	ee (%)[d]
1	CH_2Cl_2	1.5	3.4:1	95	92
2	THF	3.5	4.1:1	83	84
3	EtOAc	3.0	3.8:1	87	90
4	$CHCl_3$	2.0	2.6:1	70	93
5	EtOH	2.0	3.6:1	87	90
6	Toluene	2.0	4.6:1	95	90
7	$(CH_2Cl)_2$	2.5	2.5:1	72	91
8	CH_3CN	3.5	10.8:1	72	84
9	DMF	3.5	5.3:1	91	82
10[e]	Neat	1.0	2.9:1	74	-82
11[f]	Neat	2.5	4.7:1	69	-75

[a] The reaction was conducted with **1a** (0.15 mmol) and 0.30 mmol **2** with at 25 °C. [b] Diastereomeric ratio was measured by ^{1}H NMR of crude reaction mixture. [c] Yields were determined by ^{1}H NMR analysis of the crude reaction mixture with dibromomethane as internal standard. [d] Enantiomeric excess was determined by HPLC. [e] Catalyst **III** was used. [f] 10 mol% **III** was used.

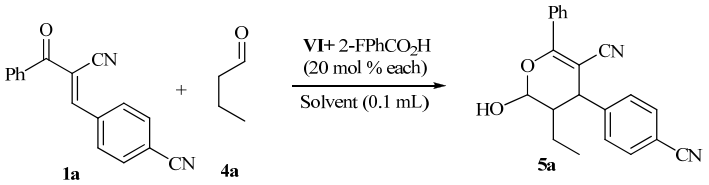
Table 2: Optimization (additive screening) for the enantioselective synthesis of pyrans using **1a** and **2**[a]



Entry	Additive	t (d)	dr[b]	Yield (%)[c]	ee (%)[d]
1	$PhCO_2H$	1.5	5.6:1	94	90
2	$2\text{-}FPhCO_2H$	2.5	5.3:1	97	96
3	CF_3CO_2H	4.5	-	-	-
4	CH_3CO_2H	2.0	4.4:1	99	96
5[e]	CH_3CO_2Na	3.0	-	-	91
6	$2\text{-}NO_2PhCO_2H$	5.0	6.5:1	67	94
7[f]	CH_3CO_2H	4d	-	-	85

[a] The reaction was conducted with **1a** (0.15 mmol) and 0.30 mmol of **2** with at 25 °C. [b] Diastereomeric ratio measured by ^{1}H NMR of crude reaction mixture. [c] Yields were determined by ^{1}H NMR analysis of the crude reaction mixture with dibromomethane as internal standard. [d] Enantiomeric excess was determined by HPLC. [e] Without internal standard. [f] Reaction was performed at 0 °C .

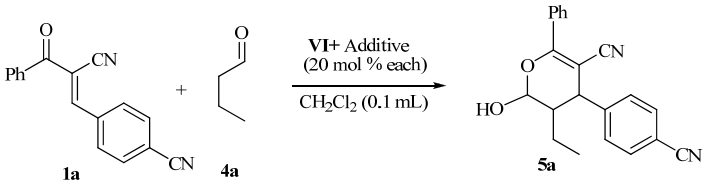
Table 3: Optimization (solvent screening) for the enantioselective synthesis of pyrans using **1a** and **4a**[a]



Entry	Solvent	t (d)	dr[b]	Yield (%)[c]	ee (%)[d]
1	CH_2Cl_2	3.5	1:0.9	73	90, 80
2	toluene	2.5	1:1.2	71	88, 72
3	$CHCl_3$	2.5	1:1.1	63	88, 76
4	EtOH	2.0	1:1	66	82, 64
5	Neat	3.5	1:1	40	84, 72

[a] The reaction was conducted with **1a** (0.1 mmol), **4a** (0.2 mmol) and 20 mol% of $2\text{-}FPhCO_2H$ at 25°C. [b] The dr ratio was determined from crude ^{1}H NMR analysis. [c] Isolated yields of the mixture of isomers. [d] Determined by HPLC analysis.

Table 4: Optimization (additive screening) for the enantioselective synthesis of pyrans using **1a** and **4a**[a]



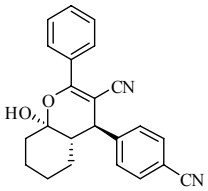
Entry	Additive	t (d)	dr[b]	Yield[c]	ee[d]
1	2-$FPhCO_2H$	3.5	1:0.9	73	90, 80
2	$PhCO_2H$	4.0	1:3.0	39	71, 28
3	4-NO_2PhCO_2H	3.5	1:0.9	73	91, 78
4	CF_3CO_2H	3.5	1:1.4	45	62, 87
5	CH_3CO_2H	4.0	1:0.7	33	88, 65
6[e]	2-$FPhCO_2H$	2.5	1:1.3	75	90, 87
7[f]	2-$FPhCO_2H$	5.0	1:1.8	20	80, 60

[a] The reaction was conducted with **2a** (0.1 mmol), **4a** (0.2 mmol), and 20 mol% of catalyst & additive at 30 ^oC. [b] The dr ratio was determined from crude ^1H NMR analysis. [c] Isolated yields of the mixture of isomers. [d] Determined by HPLC analysis. [e] 40 mol% 2-$FPhCO_2H$ was used. [f] 10 mol % **VI** and 20 mol% 2-$FPhCO_2H$ were used.

General procedure for the synthesis of substituted pyrans (**3a-n** and **5a-e**) **using amine catalysts:** In a glass vial equipped with a magnetic stirring bar was added *trans*-2-aroyl-3-arylacrylonitriles **1a-n** (0.25 mmol), cyclohexanone (or aldehyde, 0.5 mmol), organocatalyst **IV** or **VI** (20 mol%) and 2-$FC_6H_4CO_2H$ (20 or 40 mol%) in dichloromethane (0.25 mL) and the reaction mixture was stirred at 25°C for the indicated time (as in Table 2 and Table 4) . The reaction was monitored by using TLC and crude ^{1}H NMR data analyses. The reaction mixture was directly purified by flash column chromatography on silica gel (hexane:ethyl acetate = 70:30) to afford the substituted pyran derivatives.

Racemic compounds (**3a-n** and **5a-e**) were prepared following the general procedure using either pyrrolidine or n-propylamine as catalyst.

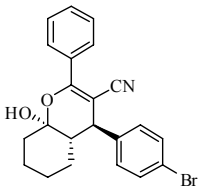
3a: The product was prepared by following the general procedure and was obtained as a colorless solid $[\alpha]^{15}_D$ = -31.96 (*c* = 0.5, $CHCl_3$); mp: 125-126 °C



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.80-7.73 (m, 2H), 7.69 (*pseudo* d, 2H, *J* = 8.2 Hz), 7.48-7.38 (m, 5H), 3.57 (d, 1H, *J* = 11.5 Hz), 2.72-2.64 (bs, 1H), 2.12-2.01 (m, 1H), 1.89-1.59 (m, 5H), 1.43-1.30 (m, 2H), 1.23-1.08 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 163.1, 146.0, 133.1, 132.6, 130.9, 129.6, 128.3, 128.1, 119.1, 118.6, 111.4, 99.3, 88.0, 45.2, 43.5, 37.5, 26.7, 25.0, 22.7; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3045 (s), 3059 (w), 2941 (s), 2861 (m), 2229 (s), 2206 (s), 1705 (w), 1609 (s), 1576 (m), 1504 (m), 1494 (m), 1447 (s), 1333 (s), 1276 (s), 1220 (s), 1140 (s), 1026 (s); **HRMS** (ESI) for $C_{23}H_{20}N_2O_2Na$, $[M+Na]^+$ (379.1422) found: 379.1429.

The enantiomeric excess was determined by HPLC. [CHIRALPAK AS-H column, 254 nm, *n*-Hexane: IPA = 85:15, flow rate: 1.0 mL/min: t_R (major) = 21.96 min; t_R (minor) = 38.71 min] ee 96%.

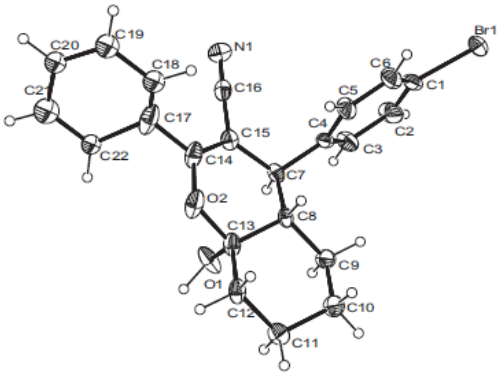
3b: The product was prepared by following the general procedure and was obtained as a colorless solid. $[\alpha]^{15}_{D}$ = -18.82 (*c* = 0.5, $CHCl_3$); mp: 159-160 ^{o}C



^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C) δ/ppm: 7.80-7.71 (m, 2H), 7.51 (*pseudo* d, 2H, *J* = 8.3 Hz), 7.48-7.37 (m, 3H), 7.17 (*pseudo* d, 2H, *J* = 8.4 Hz), 3.44 (d, 1H, *J* = 11.4 Hz), 2.85-2.64 (bs, 1H), 2.05-1.98 (m, 1H), 1.87-1.59 (m, 5H), 1.49-1.39 (m, 1H), 1.36-1.23 (m, 1H), 1.23-1.07 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 ^{o}C) δ/ppm: 162.6, 139.1, 133.3, 131.9, 130.7, 130.4, 128.3, 128.1, 121.4, 119.3, 99.3, 88.9, 45.3, 42.8, 37.6, 26.7, 25.0, 22.7; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3380 (m), 2939 (s), 2860 (m), 2206(s), 1612 (s), 1599 (s), 1575 (m), 1488 (m), 1446 (m), 1329 (s), 1219 (m), 1139 (s), 1026 (m); **HRMS** (ESI) $C_{22}H_{20}BrNO_2Na$, $[M+Na]^+$ (432.0575) found: 432.0560.

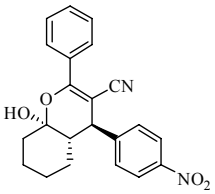
The enantiomeric excess was determined by HPLC. [CHIRALPAK AD-H column, 254 nm, *n*-Hexane: IPA = 90:10, flow rate: 1.0 mL/min: t_R (major) = 12.28 min; t_R (minor) = 34.13 min] ee 94%.

Crystallographic data for **3b** (CCDC 865351 contains the supplementary crystallographic data for **3b**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre): $C_{22}H_{20}BrNO_2$, M = 410.29, orthorhombic, space group P 21 21 21, T = 200 K, *a* = 5.5785 (4), *b* = 15.6744 (8), *c* = 22.2663 (14) Å, α = β = γ = 90, *V* = 1947.0 (2) Å^3; λ(Mo-Kα) = 0.71073 Å, *Z* = 4, D = 1.400 g/cm^3; R = 0.0500, wR2 = 0.0922



ORTEP diagram of **3b**

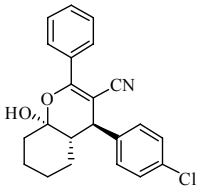
3c: The product was prepared by following the general procedure and was obtained as a colorless solid. $[\alpha]^{15}_{D}$ = -16.42 (*c* = 0.5, $CHCl_3$); mp: 105-106 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 8.26 (*pseudo* d, 2H, *J* = 8.6 Hz), 7.81-7.73 (m, 2H), 7.53-7.40 (m, 5H), 3.64 (d, 1H, *J* = 11.4 Hz), 2.81-2.72 (bs, 1H), 2.12-2.03 (m, 1H), 1.90-1.61 (m, 5H), 1.43-1.28 (m, 2H), 1.23-1.09 (m, 1H). **^{13}C NMR** (125 MHz, $CDCl_3$, 25 °C) δ/ppm: 163.0, 147.9, 147.5, 133.0, 131.0, 129.7, 128.4, 128.1, 124.1, 119.0, 99.2, 88.0, 45.2, 43.3, 37.7, 26.8, 25.0, 22.7; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3389 (m), 2940 (m), 2860 (m), 2207 (m), 1610 (m), 1598 (s), 1575 (m), 1520 (s), 1347 (s), 1140 (m); **HRMS** (ESI) $C_{22}H_{20}ClNO_2Na$, $[M+Na]^+$ (388.1080) found: 388.1050.

The enantiomeric excess was determined by HPLC. [CHIRALPAK AD-H column, 254 nm, *n*-Hexane: IPA = 85:15, flow rate: 1.0 mL/min, t_R (major) = 16.11 min, t_R (minor) = 29.78 min] ee 96%.

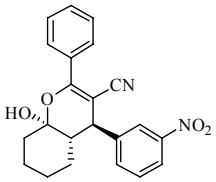
3d: The product was prepared by following the general procedure and was obtained as a colorless solid $[\alpha]^{15}_{D}$ = -22.09 (*c* = 0.5, $CHCl_3$); mp: 178-179 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.79-7.70 (m, 2H), 7.50-7.30 (m, 5H), 7.25-7.19 (m, 2H), 3.45 (d, 1H, *J* = 11.4 Hz), 2.77-2.62 (bs, 1H), 2.09-1.99 (m, 1H), 1.88-1.60 (m, 5H), 1.49-1.39 (m, 1H), 1.36-1.23 (m, 1H), 1.22-1.07 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 162.6, 138.6, 133.3, 133.2, 130.7, 130.0, 129.0, 128.3, 128.1, 119.4, 99.3, 89.0, 45.4, 42.7, 37.5, 26.7, 25.0, 22.7; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3380 (s), 2939 (m), 2858 (w), 2206 (s), 1613 (s), 1599 (s), 1574 (m), 1491 (s), 1330 (s), 1219 (m), 1139 (s); **HRMS** (ESI) for $C_{22}H_{20}N_2O_4Na$, $[M+Na]^+$ (399.1321) found: 399.1309.

The enantiomeric excess was determined by HPLC [CHIRALPAK AD-H column, 254 nm, *n*-Hexane: IPA = 90:10, flow rate: 1.0 mL/min]: t_R (major) = 10.73 min; t_R (minor) = 32.14 min] ee 94%.

3e: The product was prepared by following the general procedure and was obtained as a viscous liquid, $[\alpha]^{15}_{D}$ = -66.56 (*c* = 0.1, $CHCl_3$).

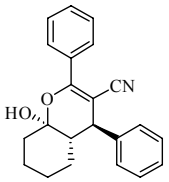


^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 8.24-8.16 (m, 2H), 7.77 (*pseudo* d, 2H, *J* = 6.6 Hz), 7.70-7.62 (m, 1H), 7.62-7.54 (m, 1H), 7.51-7.39 (m, 3H), 3.64 (d, 1H, *J* = 11.4 Hz), 2.80-2.71 (bs, 1H), 2.13-2.01 (m, 1H), 1.93-1.59 (m, 5H), 1.46-1.30 (m, 2H), 1.24-1.09 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 163.1, 148.7, 142.5, 135.2, 133.1, 130.9, 129.9, 128.4, 128.2, 123.4, 122.8, 119.1, 99.3, 88.2, 45.2, 43.3, 37.7, 26.8, 25.0, 22.7; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3388 (m), 2938 (m), 2862 (m), 2206 (m), 1612 (m), 1600 (m), 1575 (m), 1529 (s), 1350 (s); **HRMS** (ESI) $C_{22}H_{20}N_2O_4Na$, $[M+Na]^+$ (399.1321) found: 399.1313.

The enantiomeric excess was determined by HPLC. [OD-H column, 254 nm, *n*-Hexane: IPA = 90:10, flow rate: 1.0 mL/min, t_R (major) = 14.84 min; t_R (minor) = 31.32 min] ee 92%.

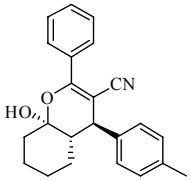
3f: The product was prepared by following the general procedure and was obtained as a colorless solid, $[\alpha]^{15}_{D}$ = -17.72 (*c* = 0.5, $CHCl_3$); mp: 174-175 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.91-7.67 (m, 2H), 7.49-7.34 (m, 5H), 7.34-7.26 (m, 3H), 3.45 (d, 1H, *J* = 11.4 Hz), 2.76-2.59 (bs, 1H), 2.11-1.97 (m, 1H), 1.87-1.58 (m, 5H), 1.51-1.42 (m, 1H), 1.37-1.23 (m, 1H), 1.22-1.06 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 162.1, 140.0, 133.5, 130.6, 128.8, 128.7, 128.3, 128.2, 127.6, 119.4, 99.3, 89.8, 45.4, 43.3, 37.8, 26.8, 25.1, 22.9; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3376 (m), 2939 (s), 2860 (w), 2207 (s), 1612 (s), 1600 (w), 1494 (w), 1139 (s); **HRMS** (ESI) $C_{22}H_{21}NO_2Na$, $[M+Na]^+$ (354.1470) found: 354.1444.

The enantiomeric excess was determined by HPLC. [CHIRALPAK AS-H column, 254 nm, *n*-Hexane: IPA = 85:15, flow rate: 1.0 mL/min, t_R (major) = 11.36 min; t_R (minor) = 23.33 min] ee 95%.

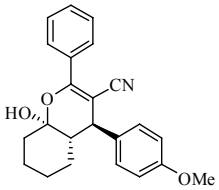
3g: The product was prepared by following the general procedure and was obtained as a colorless solid, $[\alpha]^{15}_{D}$ = -24.53 (*c* = 0.5, $CHCl_3$); mp: 168-169 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.80-7.72 (m, 2H), 7.49-7.35 (m, 3H), 7.23-7.12 (m, 4H), 3.41 (d, 1H, *J* = 11.4 Hz), 2.87-2.75 (bs, 1H), 2.35 (s, 3H), 2.09-1.97 (m, 1H), 1.84-1.56 (m, 5H), 1.52-1.43 (m, 1H), 1.36-1.21 (m, 1H), 1.21-1.05 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 162.0, 137.1, 136.8, 133.5, 130.5, 129.5, 128.5, 128.3, 128.2, 119.5, 99.3, 90.0, 45.4, 42.9, 37.8, 26.8, 25.1, 22.9, 21.1; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3392 (s), 2937 (s), 2856 (m), 2205 (s), 1612 (s), 1576 (m), 1447 (m), 1331 (s), 1138 (s); **HRMS** (ESI) $C_{23}H_{23}NO_2Na$, $[M+Na]^+$ (368.1626) found: 368.1598.

The enantiomeric excess was determined by HPLC. [CHIRALPAK AD-H column, 254 nm, *n*-Hexane: IPA = 85:15, flow rate: 1.0 mL/min, t_R (major) = 6.23 min; t_R (minor) = 14.96 min] ee 93%.

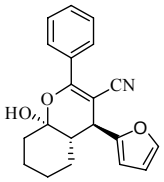
3h: The product was prepared by following the general procedure and was obtained as a colorless solid, $[\alpha]^{15}_{D}$ = -34.01 (*c* = 0.5, $CHCl_3$); Mp: 155-156 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.81-7.71 (m, 2H), 7.48-7.36 (m, 3H), 7.19 (*pseudo* d, 2H, *J* = 8.5 Hz), 6.91 (*pseudo* d, 2H, *J* = 8.5 Hz), 3.80 (s, 3H), 3.40 (d, 1H, *J* = 11.4 Hz), 3.08-2.84 (bs, 1H), 2.09-1.96 (m, 1H), 1.83-1.53 (m, 5H), 1.51-1.41 (m, 1H), 1.35-1.06 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 162.0, 158.9, 133.5, 131.9, 130.5, 129.6, 128.3, 128.2, 119.5, 114.2, 99.3, 90.0, 55.2, 45.5, 42.5, 37.7, 26.8, 25.1, 22.9; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3389 (w), 2934 (s), 2857 (m), 2205 (m), 1611 (s), 1599 (m), 1581 (m), 1513 (s), 1448 (m), 1250 (s); **HRMS** (ESI) $C_{23}H_{23}NO_3Na$, $[M+Na]^+$ (384.1576) found: 384.1531.

The enantiomeric excess was determined by HPLC [CHIRALPAK AD-H column, 254 nm, *n*-Hexane: IPA = 80:20, flow rate: 1.0 mL/min: t_R (major) = 5.82 min, t_R (minor) = 15.27 min] ee 94%.

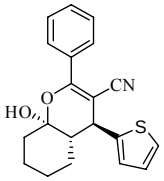
3i: The product was prepared by following the general procedure and was obtained as a colorless solid, $[\alpha]^{15}_{D}$ = -57.23 (*c* = 0.5, $CHCl_3$); mp: 165-166 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.79-7.69 (m, 2H), 7.49-7.36 (m, 4H), 6.41-6.27 (m, 2H), 3.65 (d, 1H, *J* = 11.7 Hz), 2.79-2.54 (bs, 1H), 2.12-1.97 (m, 2H), 1.89-1.48 (m, 5H), 1.39-1.18 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 162.0, 151.9, 142.4, 133.4, 130.6, 128.3, 128.2, 118.9, 110.4, 109.2, 99.2, 87.6, 42.0, 37.8, 37.0, 27.0, 25.0, 22.8; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3431 (s), 2952 (m), 2908 (m), 2863 (m), 2250 (w), 1710 (s), 1602 (w), 1506 (m), 1446 (m), 1363 (s); **HRMS** (ESI) $C_{20}H_{19}NO_3Na$, $[M+Na]^+$ (344.1263) found: 344.1278.

The enantiomeric excess was determined by HPLC. [CHIRALPAK AD-H column, 254 nm, *n*-Hexane: IPA = 85:15, flow rate: 1.0 mL/min, t_R (major) = 6.45 min, t_R (minor) = 19.78 min] ee 93%.

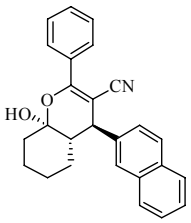
3j: The product was prepared by following the general procedure and was obtained as a colorless solid, $[\alpha]^{15}_{D}$ = -25.58 (*c* = 0.5, $CHCl_3$); Mp: 178-179 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.81-7.69 (m, 2H), 7.52-7.36 (m, 3H), 7.31-7.27 (m, 1H), 7.06-6.97 (m, 2H), 3.81 (d, 1H, *J* = 11.4 Hz), 2.78-2.60 (bs, 1H), 2.10-1.98 (m, 1H), 1.90-1.59 (m, 6H), 1.38-1.15 (m, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 161.7, 143.1, 133.3, 130.7, 128.4, 128.3, 127.1, 126.8, 124.9, 119.1, 99.4, 89.9, 46.0, 38.6, 37.6, 27.0, 25.1, 22.8; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3357 (s), 2935 (s), 2861 (m), 2207 (s), 1608 (s), 1575 (m), 1447 (m); **HRMS** (ESI) $C_{20}H_{19}NO_2SNa$, $[M+Na]^+$ (360.1034) found: 360.1002.

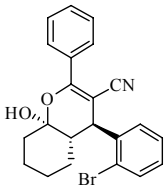
The enantiomeric excess was determined by HPLC [CHIRALPAK AD-H column, 254 nm, *n*-Hexane: IPA = 90:10, flow rate: 1.0 mL/min, t_R (major) = 11.86 min t_R (minor) = 41.63 min] ee 95%.

3k: The product was prepared by following the general procedure and was obtained as a colorless solid, $[\alpha]^{15}_{D}$ = -44.14 (*c* = 0.5, $CHCl_3$); mp: 152-153 °C.



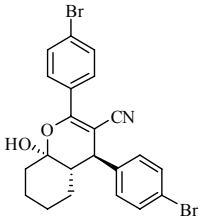
^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.93-7.77 (m, 5H), 7.74 (s, 1H), 7.53-7.37 (m, 6H), 3.62 (d, 1H, *J* = 11.4 Hz), 2.86-2.72 (bs, 1H), 2.09-1.99 (m, 1H), 1.94-1.58 (m, 5H), 1.50-1.40 (m, 1H), 1.40-1.27 (m, 1H), 1.19-1.03 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 162.4, 137.3, 133.5, 133.5, 133.0, 130.6, 128.8, 128.3, 128.2, 127.8, 127.7, 126.2, 125.8, 119.5, 99.3, 89.5, 45.3, 43.5, 37.7, 26.9, 25.1, 22.8; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3379 (m), 3056 (w), 2936 (s), 2860 (m), 2206 (s), 1600 (s), 1576 (m), 1446 (m); **HRMS** (ESI) $C_{26}H_{23}NO_2Na$, $[M+Na]^+$ (404.1626) found: 404.1623.
The enantiomeric excess was determined by HPLC. [CHIRALPAK AD-H column, 254 nm, *n*-Hexane: IPA = 90:10, flow rate: 1.0 mL/min: t_R (major) = 6.01 min, t_R (minor) = 26.63 min] ee 60%.

3l: The product was prepared by following the general procedure and was obtained as a colorless solid (mixture of diastereomer).



3l (major diastereomer): **^{1}H NMR** (400 MHz, d_6-DMSO, 25 °C) δ/ppm: 7.71-7.63 (m, 3H), 7.52-7.45 (m, 5H), 7.27-7.20 (m, 1H), 4.12 (d, 2H, *J* = 11.3 Hz), 2.18-2.10 (m, 1H), 1.90-1.76 (m, 1H), 1.72-1.50 (m, 5H), 1.43-1.28 (m, 1H), 1.19-1.10 (m, 1H); **^{13}C NMR** (100 MHz, d_6-DMSO, 25 °C) δ/ppm: 162.7, 139.8, 133.6, 132.4, 130.6, 129.6, 129.1, 128.6, 128.3, 128.0, 125.6, 119.2, 99.9, 87.9, 45.9, 40.9, 35.9, 26.3, 24.6, 22.6; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3390 (s), 3072 (m), 2942 (s), 2925 (s), 2891 (m), 2861 (m), 2260 (m), 1715 (s), 1492 (m), 1470 (s); **HRMS** (ESI) $C_{22}H_{20}BrNO_2Na$, $[M+Na]^+$ (432.0575) found: 432.0536.
The enantiomeric excess was determined by HPLC [CHIRALPAK AS-H column, 254 nm, *n*-Hexane: IPA = 95:5, flow rate: 0.5 mL/min, t_R (major) = 75.20 min, t_R (minor) = 180.08 min] ee 85%.

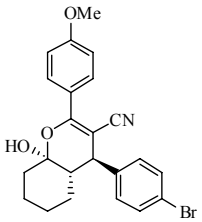
3m: The product was prepared by following the general procedure and was obtained as a colorless solid, $[\alpha]^{15}_{D}$ = -11.57 (*c* = 0.2, $CHCl_3$); mp: 179-180 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.63 (*pseudo* d, 2H, *J* = 8.6 Hz), 7.55 (*pseudo* d, 2H, *J* = 8.6 Hz), 7.51 (*pseudo* d, 2H, *J* = 8.3 Hz), 7.15 (*pseudo* d, 2H, *J* = 8.4 Hz), 3.42 (d, 1H, *J* = 11.5 Hz), 2.68-2.63 (bs, 1H), 2.08-1.99 (m, 1H), 1.87-1.59 (m, 5H), 1.49-1.39 (m, 1H), 1.36-1.22 (m, 1H), 1.22-1.07 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 161.5, 138.8, 132.2, 132.0, 131.5, 130.3, 129.7, 125.1, 121.5, 119.1, 99.5, 89.3, 45.2, 42.8, 37.5, 26.7, 25.0, 22.7; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3378 (m), 2939 (s), 2861 (m), 2207 (s), 1609 (s), 1590 (s), 1488 (s); **HRMS** (ESI) $C_{22}H_{19}Br_2NO_2Na$, $[M+Na]^+$ (511.9660) found: 511.9646.

The enantiomeric excess was determined by HPLC [CHIRALCEL OD-H column, 254 nm, *n*-Hexane: IPA = 92:8, flow rate: 1.0 mL/min, t_R (major) = 11.09 min, t_R (minor) = 16.71 min] ee 65%.

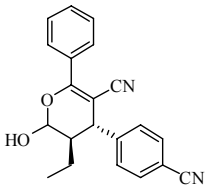
3n: The product was prepared by following the general procedure and was obtained as a colorless solid, $[\alpha]^{15}_{D}$ = -22.67 (*c* = 0.1, $CHCl_3$); mp: 108-109 °C.



^{1}H NMR (400 MHz, $CDCl_3$, 25 °C) δ/ppm: 7.71 (*pseudo* d, 2H, *J* = 8.9 Hz), 7.49 (*pseudo* d, 2H, *J* = 8.4 Hz), 7.15 (*pseudo* d, 2H, *J* = 8.4 Hz), 6.90 (*pseudo* d, 2H, *J* = 8.9 Hz), 3.81 (s, 3H), 3.40 (d, 1H, *J* = 11.4 Hz), 3.22-2.67 (bs, 1H), 2.04-1.96 (m, 1H), 1.77-1.55 (m, 5H), 1.44-1.36 (m, 1H), 1.31-1.20 (m, 1H), 1.19-1.04 (m, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$, 25 °C) δ/ppm: 162.3, 161.4, 139.4, 131.9, 130.4, 129.8, 125.6, 121.3, 119.8, 113.6, 99.1, 87.3, 55.3, 45.4, 42.9, 37.5, 26.7, 25.0, 22.7; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3384 (s), 2939 (s), 2862 (m), 2204 (s), 1609 (s), 1574 (m); **HRMS** (ESI) $C_{23}H_{22}BrNO_3Na$, $[M+Na]^+$ (462.0681) found: 462.0644.

The enantiomeric excess was determined by HPLC [CHIRALPAK AS-H column, 254 nm, *n*-Hexane: IPA = 90:10, flow rate: 1.0 mL/min, t_R (major) = 23.06 min, t_R (minor) = 45.56 min] ee 75%.

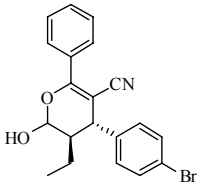
5a: The product was prepared by following the general procedure and was obtained as a colorless solid (mixture of diastereomer).



5a (major diastereomer): **^{1}H NMR** (500 MHz, $CDCl_3$, 25 ^{o}C) δ/ppm: 7.79-7.75 (m, 2H), 7.72-7.68 (m, 2H), 7.48-7.41 (m, 5H), 5.72 (dd, 1H, J = 4.1 Hz, J = 2.3 Hz), 3.64 (d, 1H, J = 10.8 Hz), 3.57 (dd, 1H, J = 4.3 Hz, J = 1.4 Hz), 1.93-1.85 (m, 1H), 1.53-1.43 (m, 1H), 1.33-1.23 (m, 1H), 0.88 (t, 3H, J = 7.5 Hz); **^{13}C NMR** (125 MHz, $CDCl_3$, 25 ^{o}C) δ/ppm: 162.4, 146.2, 132.8, 132.7, 131.1, 129.6, 128.4, 128.1, 118.9, 118.6, 111.7, 93.3, 87.7, 44.1, 42.5, 21.5, 11.1; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3388 (m), 2965 (w), 2936 (w), 2879 (w), 2230 (m), 2207 (m), 1610 (s), 1599 (s), 1575 (m); **HRMS** (ESI) $C_{21}H_{18}N_2O_2Na$, $[M+Na]^+$ (353.1266) found: 353.1282.

The enantiomeric excess was determined by HPLC [CHIRALPAK IA column, 254 nm, *n*-Hexane: EtOH = 90:10, flow rate: 1.0 mL/min] for major diastereomer: t_R (major) = 23.59 min, t_R (minor) = 49.24 min, ee 87%; for minor diastereomer: t_R (minor) = 20.26 min, t_R (major) = 33.13 min, ee 90%.

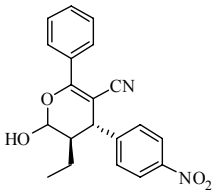
5b: The product was prepared by following the general procedure and was obtained as a colorless solid (mixture of diastereomer).



5b (major diastereomer): **^{1}H NMR** (400 MHz, $CDCl_3$, 25 ^{o}C) δ/ppm: 7.81, 7.75 (m, 2H), 7.51-7.35 (m, 5H*), 7.15- 7.09 (m, 2H), 5.51-5.41 (m, 1H), 4.32 (br, 1H), 4.03 (d, 1H, J = 5.7 Hz), 1.93-1.83 (m, 1H), 1.34-1.16 (m, 2H), 0.87-0.76 (m, 3H*) [* some peak from the minor isomer is merged in this area.]; **^{13}C NMR** (100 MHz, $CDCl_3$, 25 ^{o}C) δ/ppm: 163.9, 137.0, 132.8, 132.0, 131.6, 130.9, 128.3, 128.0, 121.4, 119.7, 95.0, 85.8, 42.6, 40.4, 19.0, 11.6; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3373 (m), 2966 (m), 2937 (w), 2878 (w), 2208 (s), 1614 (s), 1598 (s), 1575 (m); **HRMS** (ESI) $C_{20}H_{18}BrNO_2Na$, $[M+Na]^+$ (406.0419) found: 406.0400.

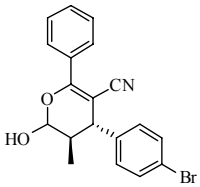
The enantiomeric excess was determined by HPLC [CHIRALPAK IA column, 254 nm, *n*-Hexane: EtOH = 95:5, flow rate: 1.00 mL/min]: for major diastereomer: t_R (major) = 27.82 min, t_R (minor) = 40.40 min, ee 83%; for minor diastereomer: t_R (minor) = 22.24 min, t_R (major) = 31.76 min, ee 89%.

5c: The product was prepared by following the general procedure and was obtained as a colorless solid (mixture of diastereomer).



5c (major diastereomer): **^{1}H NMR** (400 MHz, $CDCl_3$, 25 ^{o}C) δ/ppm: 8.34-8.21 (m, 2H), 7.86-7.79 (m, 2H), 7.54-7.42 (m, 5H*), 5.69-5.58 (m, 1H), 4.30 (d, 1H, J = 5.9 Hz), 3.98 (br, 1H), 2.09-1.97 (m, 1H), 1.40-1.27 (m, 2H*), 0.89-0.81 (m, 3H*); **^{13}C NMR** (125 MHz, $CDCl_3$, 25 ^{o}C) δ/ppm: 164.0, 147.4, 145.9, (132.7, 131.5, 131.2, 130.2, 128.5, 128.1, 123.8, 123.0, 119.3)*, 94.6, 85.3, 42.4, 40.5, 18.9, 11.6 [* some peak from the minor isomer is merged in this area.]; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3373 (w), 2968 (w), 2937 (w), 2879 (w), 2207 (m), 1614 (m), 1598 (m), 1519 (s), 1495 (m), 1348 (s); **HRMS** (ESI) $C_{20}H_{18}N_2O_4Na$, $[M+Na]^+$ (373.1164) found: 373.1127.
The enantiomeric excess was determined by HPLC. [CHIRALPAK IA column, 254 nm, *n*-Hexane: EtOH = 90:10, flow rate: 0.8 mL/min]: The enantiomeric excess was determined by HPLC [CHIRALPAK IA column, 254 nm, *n*-Hexane: EtOH = 90:10, flow rate: 0.8 mL/min], for major diastereomer: t_R (major) = 28.28 min, t_R (minor) = 51.97 min, ee 87%; for minor diastereomer: t_R (minor) = 24.67 min, t_R (major) = 39.32 min, ee 88%.

5d: The product was prepared by following the general procedure and was obtained as a colorless solid (mixture of diastereomer).

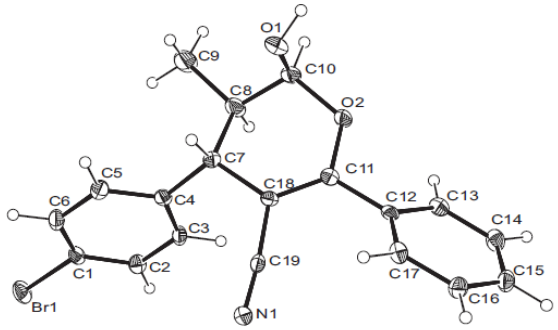


5d (major diastereomer):**^{1}H NMR** (500 MHz, d_6-DMSO, 25 ^{o}C) δ/ppm: 7.79-7.28 (m, 9H*), 5.58-5.49 (m, 1H), 3.45 (d, 1H, J = 10.7 Hz), 2.06-1.94 (m, 1H), 0.82 (d, 3H, J = 6.6 Hz) [* some peak from the minor isomer is merged in this area.]; **^{13}C NMR** (125 MHz, d_6-DMSO, 25 ^{o}C) δ/ppm: Major, 162.4, 140.4, 133.4, 131.5, 130.9, 130.7, 128.3, 128.0, 120.3, 119.3, 95.3, 87.4, 42.1, 37.6, 14.0; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3378 (m), 2970 (w), 2932 (w), 2209 (s), 1612 (s), 1600 (s), 1577 (m), 1489 (m); **HRMS** (ESI) $C_{19}H_{16}BrNO_2Na$, $[M+Na]^+$ (392.0262) found: 392.0237.

The enantiomeric excess was determined by HPLC. [CHIRALPAK IA column, 254 nm, *n*-Hexane: EtOH = 93:7, flow rate: 0.8 mL/min] for major diastereomer: t_R (major) = 26.48 min, t_R (minor) = 60.73 min, ee 95%; for minor diastereomer: t_R

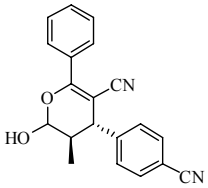
(minor) = 23.51 min, t_R (major) = 31.56 min, ee 55%.

Crystallographic data for **5d** [major diastereomer (CCDC 865350 contains the supplementary crystallographic data for **5d**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre)]: $C_{19}H_{16}BrNO_2$, M = 370.23, orthorhombic, space group P 21 21 21, T = 200 K, *a* = 5.2806 (2), *b* = 8.1975 (2), *c* = 37.8235 (11) Å, α = β = γ = 90, *V* = 1637.29 (9) Å^3; λ(Mo-Kα) = 0.71073 Å, *Z* = 4, D = 1.502 g/cm^3; R = 0.0300, wR2 = 0.0627.



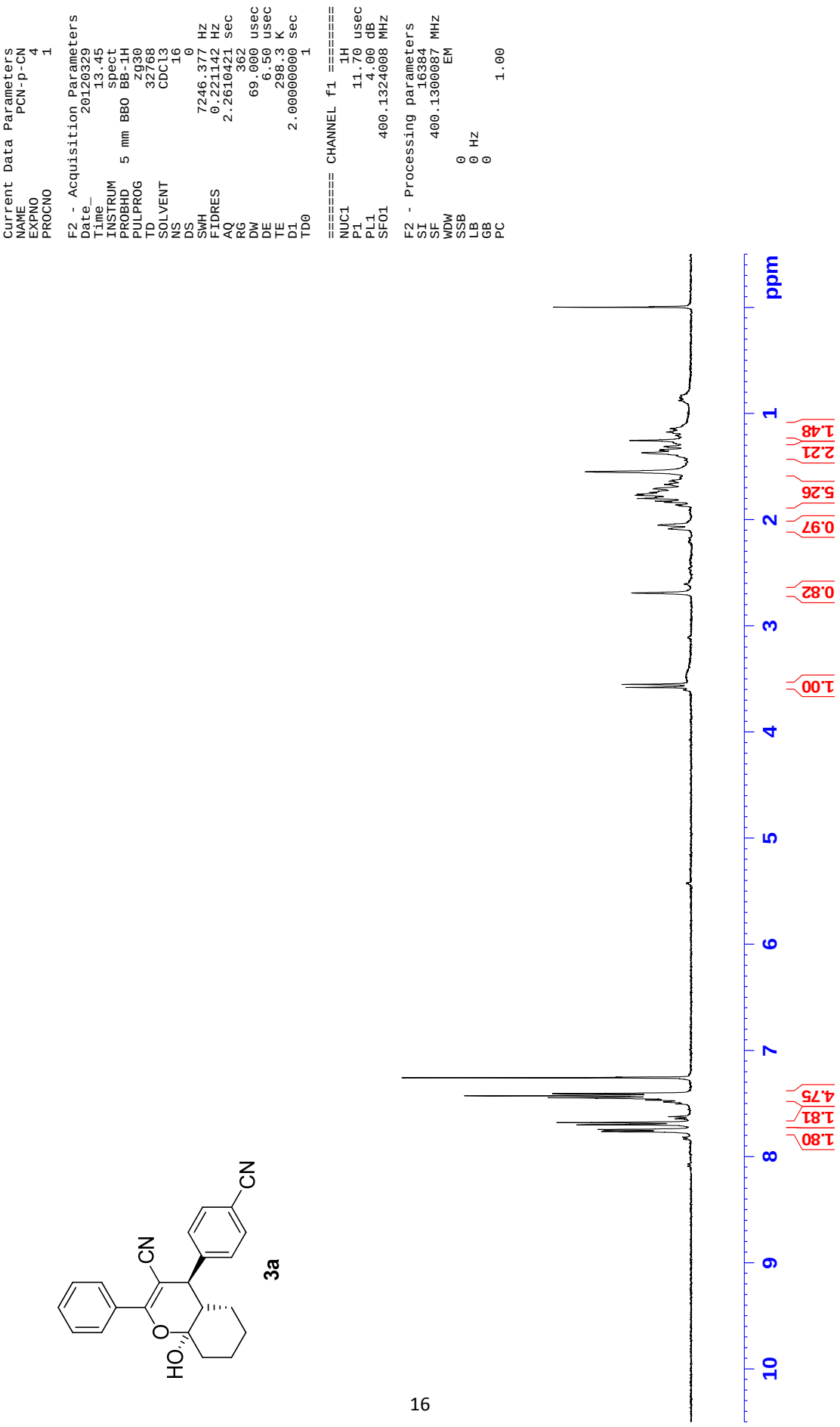
ORTEP diagram of **5d**

5e: The product was prepared by following the general procedure and was obtained as a colorless solid (mixture of diastereomer).



5e (major diastereomer): **^{1}H NMR** (400 MHz, d_6-DMSO, 25 ^{o}C) δ/ppm: 7.97-7.45 (m, 9H*), 5.61-5.50 (m, 1H), 3.59 (d, 1H, *J* = 10.8 Hz), 2.08-1.85 (m, 1H), 0.93-0.72 (m, 3H) [* some peak from the minor isomer is merged in this area.]; **^{13}C NMR** (100 MHz, d_6-DMSO, 25 ^{o}C) δ/ppm: 162.8, 146.9, 133.3, 132.5, 130.7, 129.9, 128.3, 128.0, 119.2, 118.7, 110.2, 95.3, 86.8, 42.6, 37.4, 13.9; **IR** (KBr) $\tilde{\nu}$ (cm^{-1}): 3379 (s), 2968 (w), 2931 (w), 2876 (w), 2230 (m), 2217 (s), 1611 (s), 1599 (m), 1576 (w); **HRMS** (ESI) $C_{20}H_{16}N_2O_2Na$, $[M+Na]^+$ (339.1109) found: 339.1094.

The enantiomeric excess was determined by HPLC. [CHIRALPAK IA column, 254 nm, *n*-Hexane: EtOH = 82:18, 1.0 mL/min]: for major diastereomer: t_R (major) = 11.48 min, t_R (minor) = 29.08 min, ee 87%; for minor diastereomer: t_R (minor) = 10.28 min, t_R (major) = 15.17 min, ee 61%.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ppm
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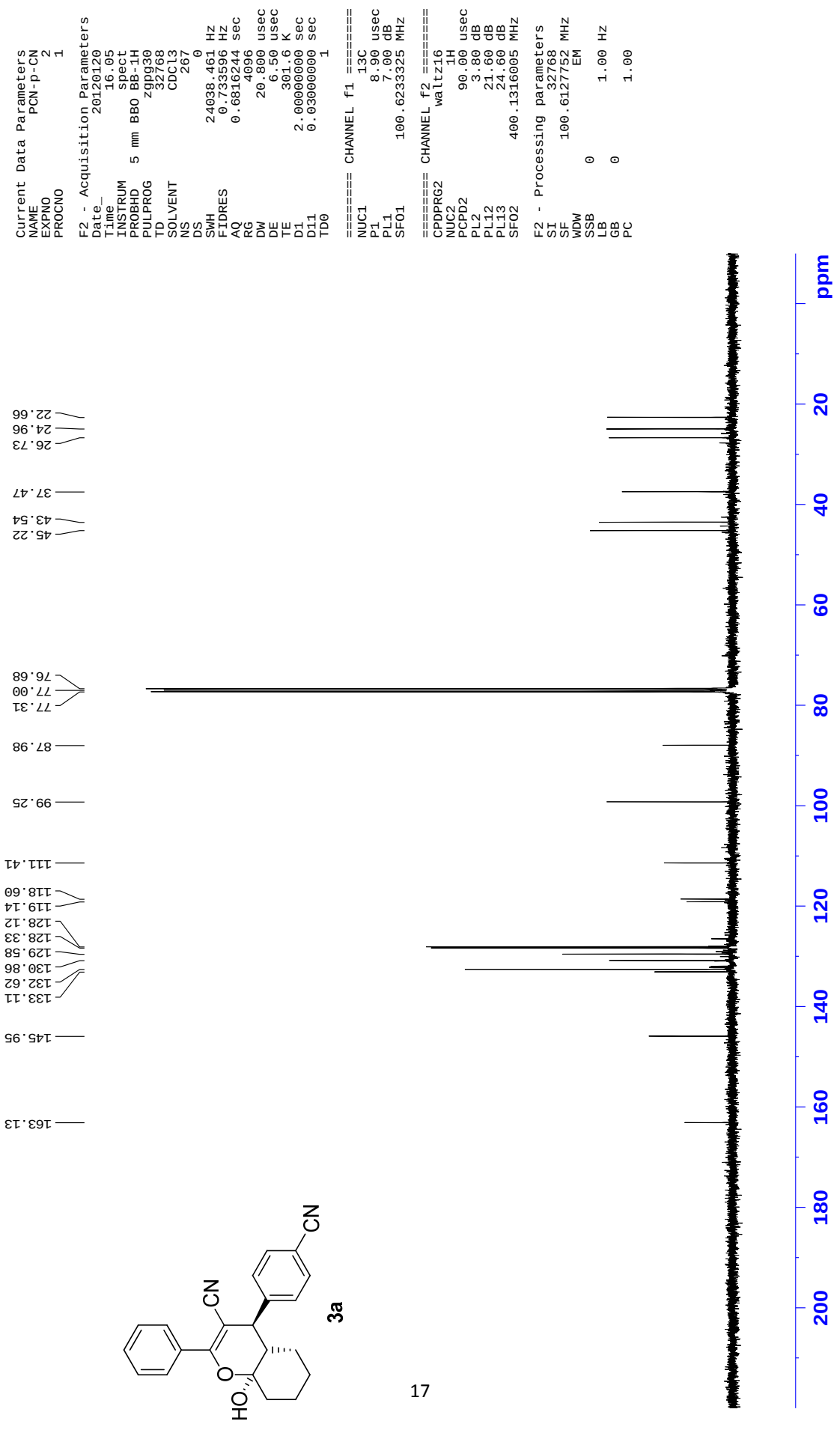
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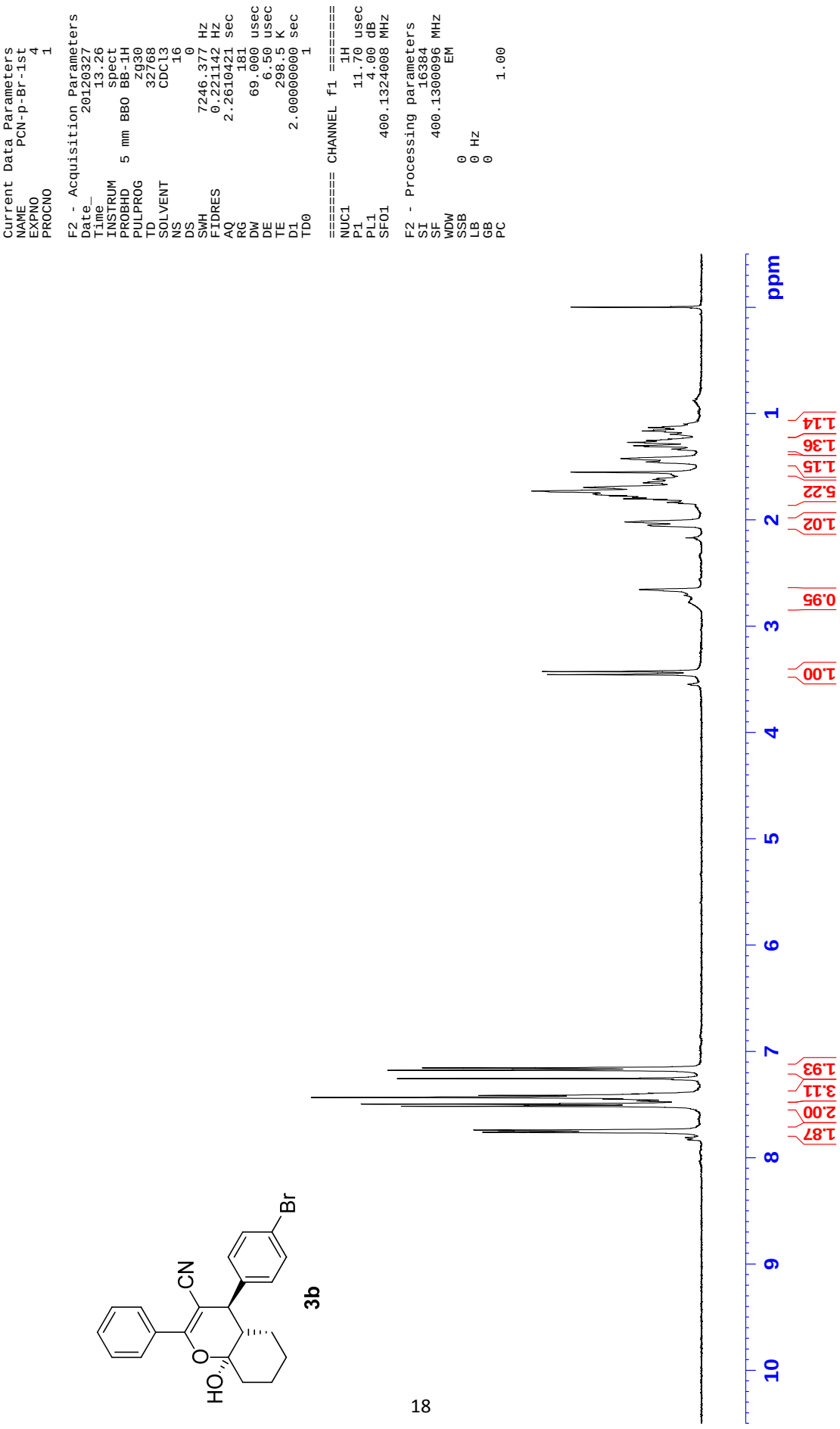
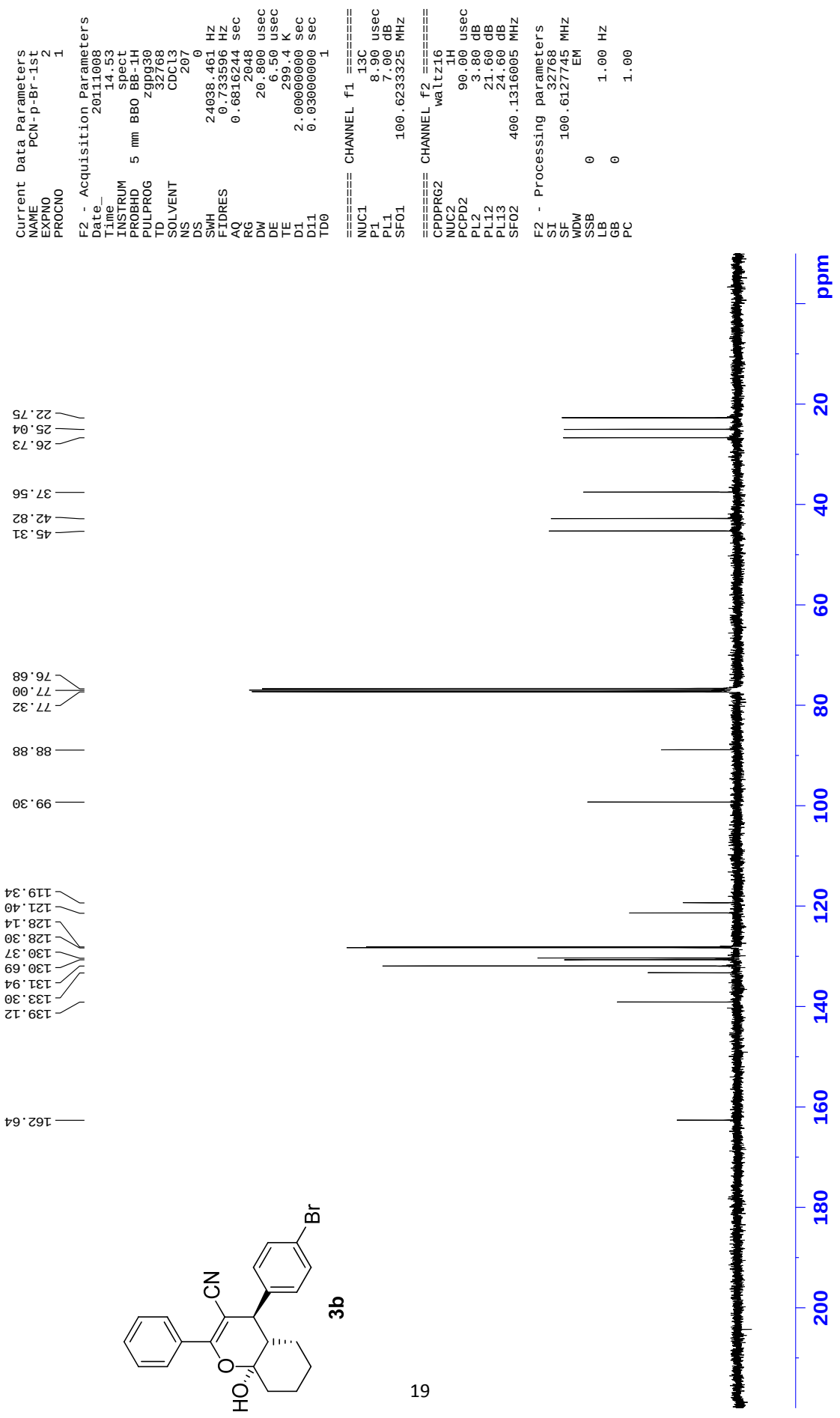
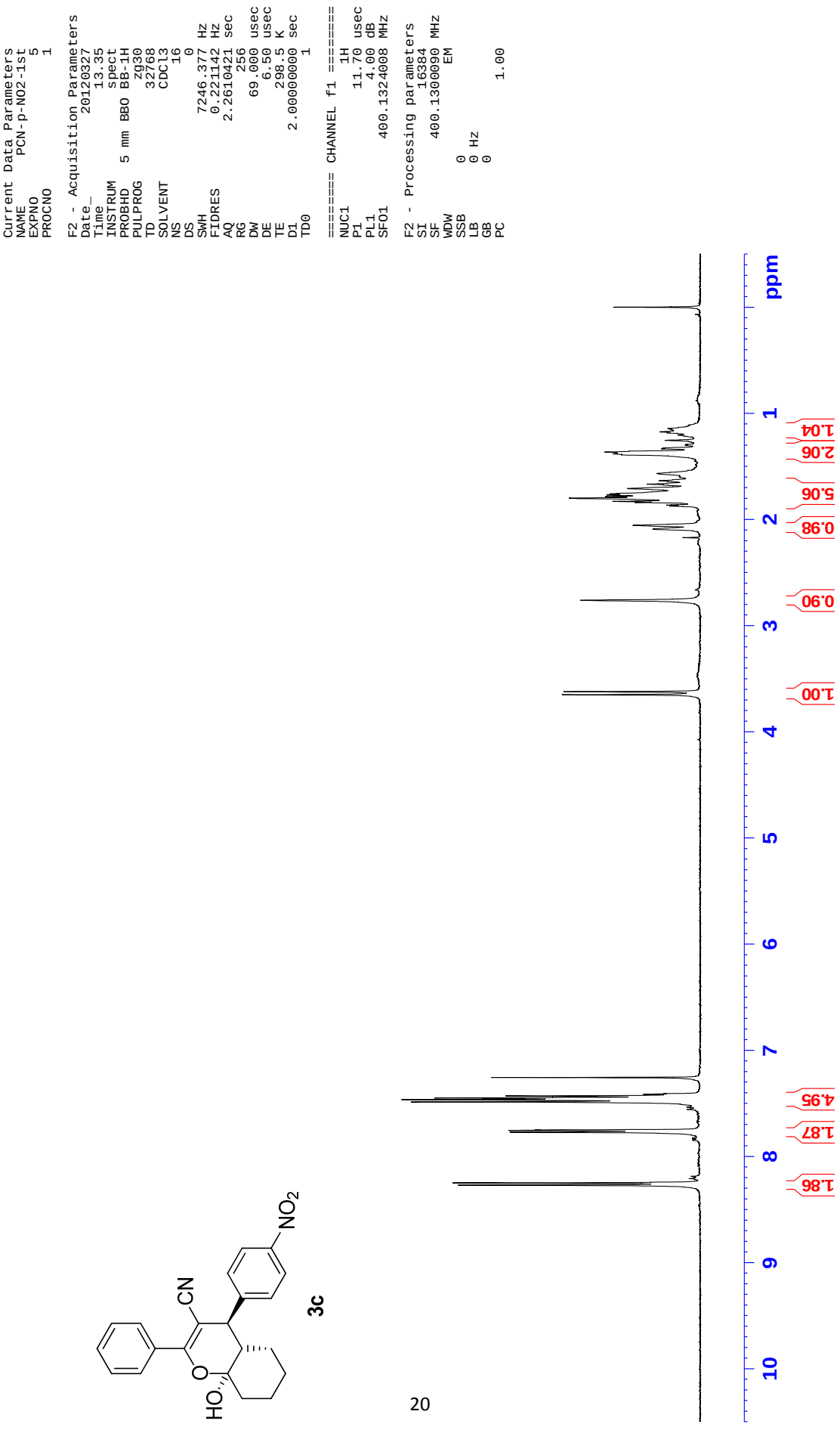
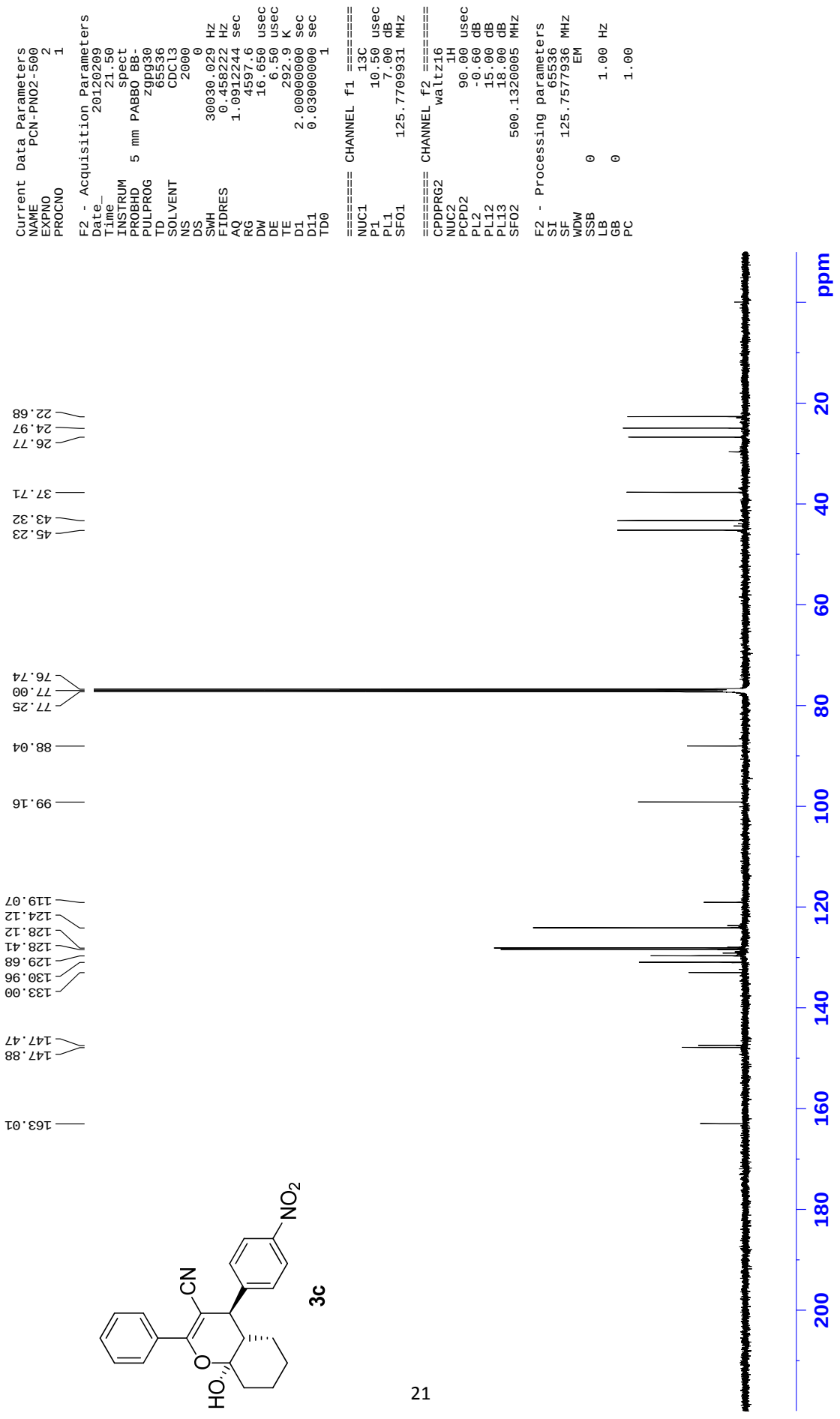
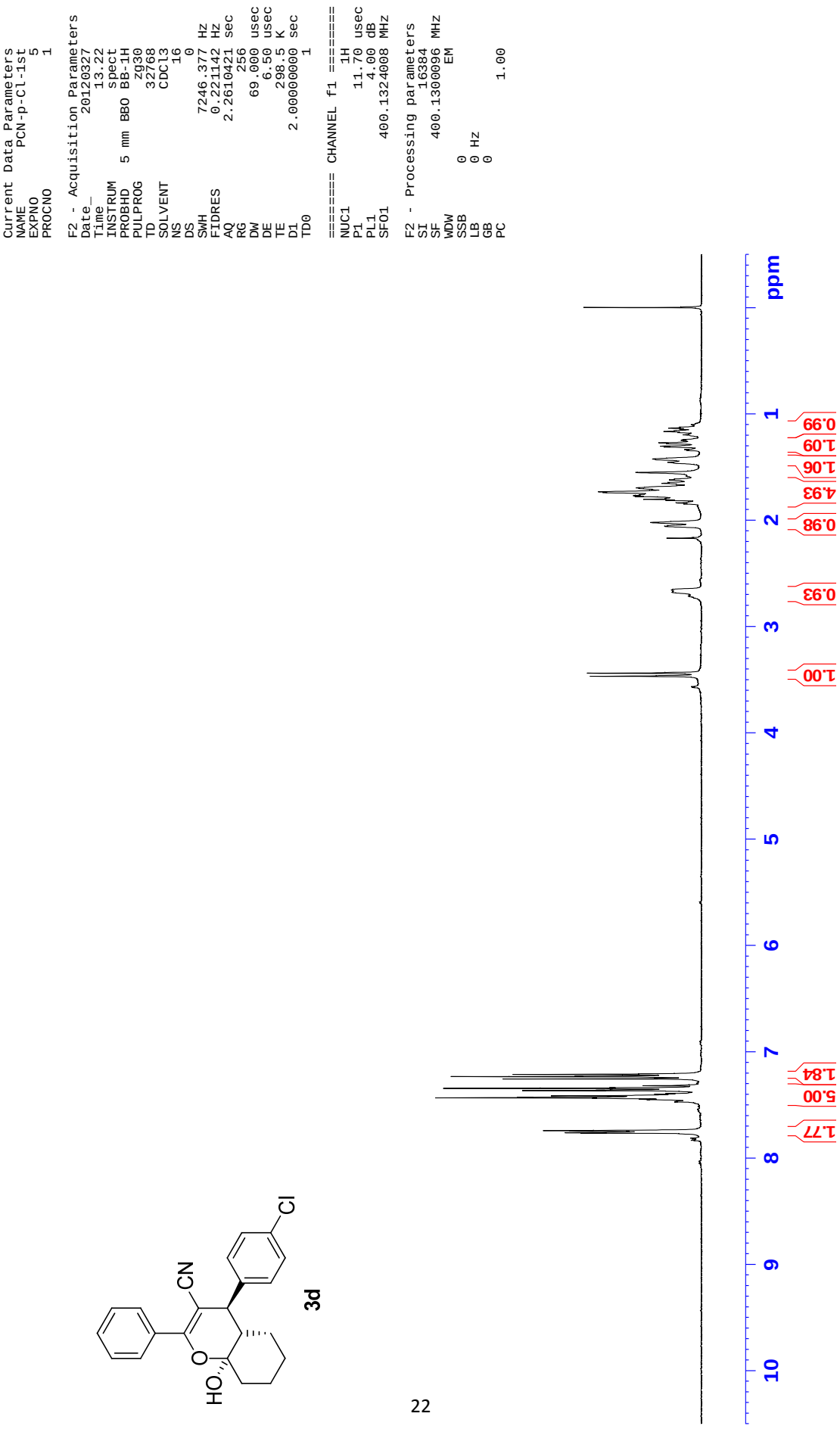
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127752 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.66
24.96
26.73
37.47
43.54
45.22
76.68
77.00
77.31
87.98
99.25
111.41
118.60
119.14
128.12
128.33
129.58
130.86
132.62
133.11
145.95
163.13
ppm
20
40
60
80
100
120
140
160
180
200
CN
CN
O
HO
3a

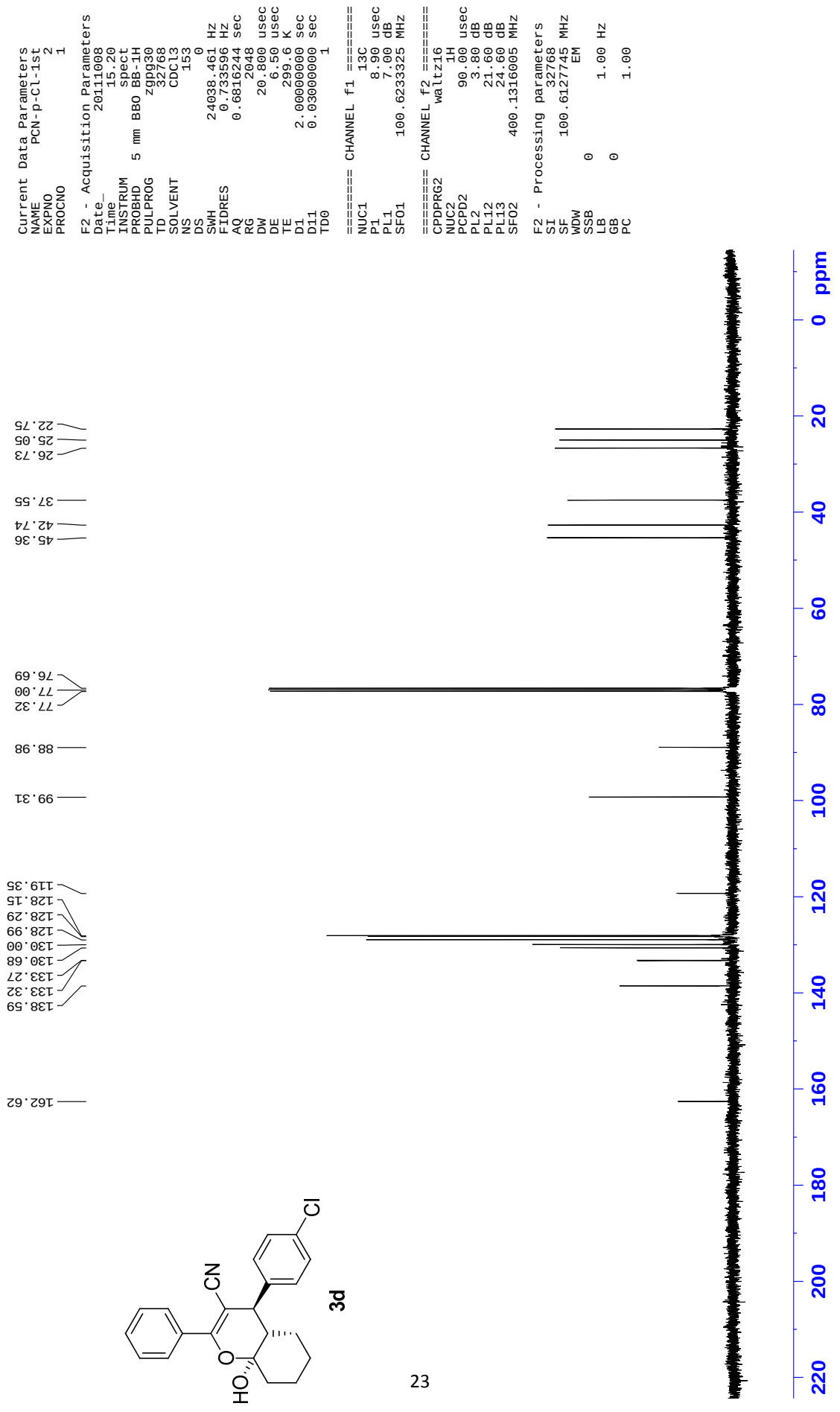

Current Data Parameters
NAME PCN-p-Br-1st
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120327
Time 13.26
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 181
DW 69.000 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
1.14
1.36
1.15
5.22
1.02
0.95
1.00
1.93
3.11
2.00
1.87
CN
Br
O
HO
3b


Current Data Parameters
NAME PCN-p-Br-1st
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111008
Time_ 14.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 207
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 2048
DW 20.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.75
25.04
26.73
37.56
42.82
45.31
76.68
77.00
77.32
88.88
99.30
119.34
121.40
128.14
128.30
130.37
130.69
131.94
133.30
139.12
162.64
ppm
20
40
60
80
100
120
140
160
180
200
Br
CN
O
HO
3b


Current Data Parameters
NAME PCN-p-NO2-1st
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120327
Time 13.35
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 256
DW 69.000 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
1.04
2.06
5.06
0.98
0.90
1.00
4.95
1.87
1.86
CN
NO2
O
HO
3c


Current Data Parameters
NAME PCN-PNO2-500
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120209
Time_ 21.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2000
DS 0
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 4597.6
DW 16.650 usec
DE 6.50 usec
TE 292.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 125.7709931 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577936 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.68
24.97
26.77
37.71
43.32
45.23
76.74
77.00
77.25
88.04
99.16
119.07
124.12
128.12
128.41
129.68
130.96
133.00
147.47
147.88
163.01
ppm
20
40
60
80
100
120
140
160
180
200
CN
NO2
HO
O
3c


Current Data Parameters
NAME PCN-p-Cl-1st
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120327
Time 13.22
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 256
DW 69.000 usec
DE 6.50 usec
TE 298.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
0.99
1.09
1.06
4.93
0.98
0.93
1.00
1.84
5.00
1.77
CN
Cl
O
HO
3d


Current Data Parameters
NAME PCN-p-Cl-1st
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111008
Time_ 15.20
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 153
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 2048
DW 20.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.75
25.05
26.73
37.55
42.74
45.36
76.69
77.00
77.32
88.98
99.31
119.35
128.15
128.29
128.99
130.00
130.68
133.27
133.32
138.59
162.62
CN
Cl
HO
O
3d
0
20
40
60
80
100
120
140
160
180
200
220
ppm

```
Current Data Parameters
NAME          PCN-m-NO2
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20120328
Time               21.25
INSTRUM            spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2610421 sec
RG                   256
DW                69.000 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                11.70 usec
PL1                4.00 dB
SFO1        400.1324008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300084 MHz
WDW                   EM
SSB                    0
LB                 0 Hz
GB                     0
PC                  1.00
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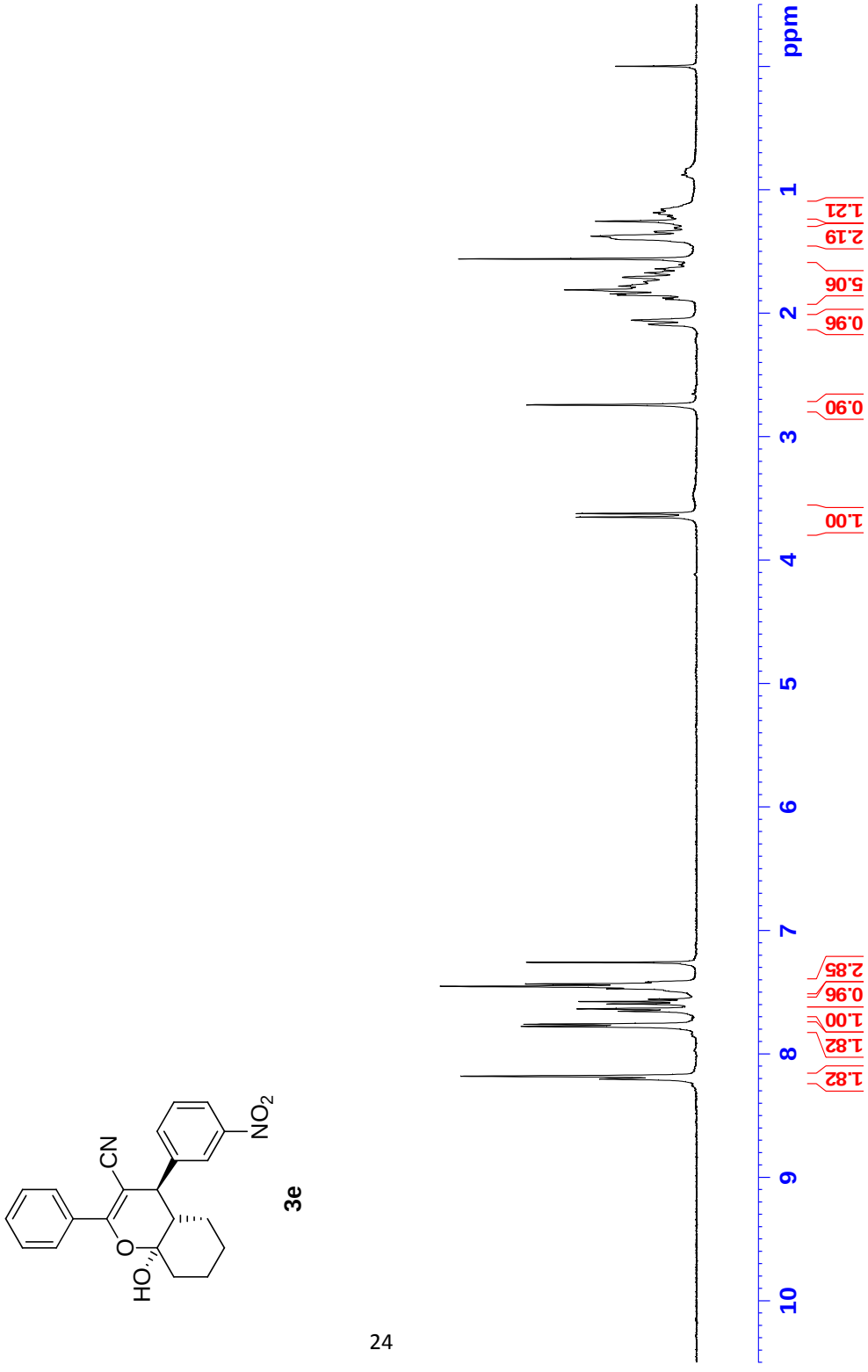
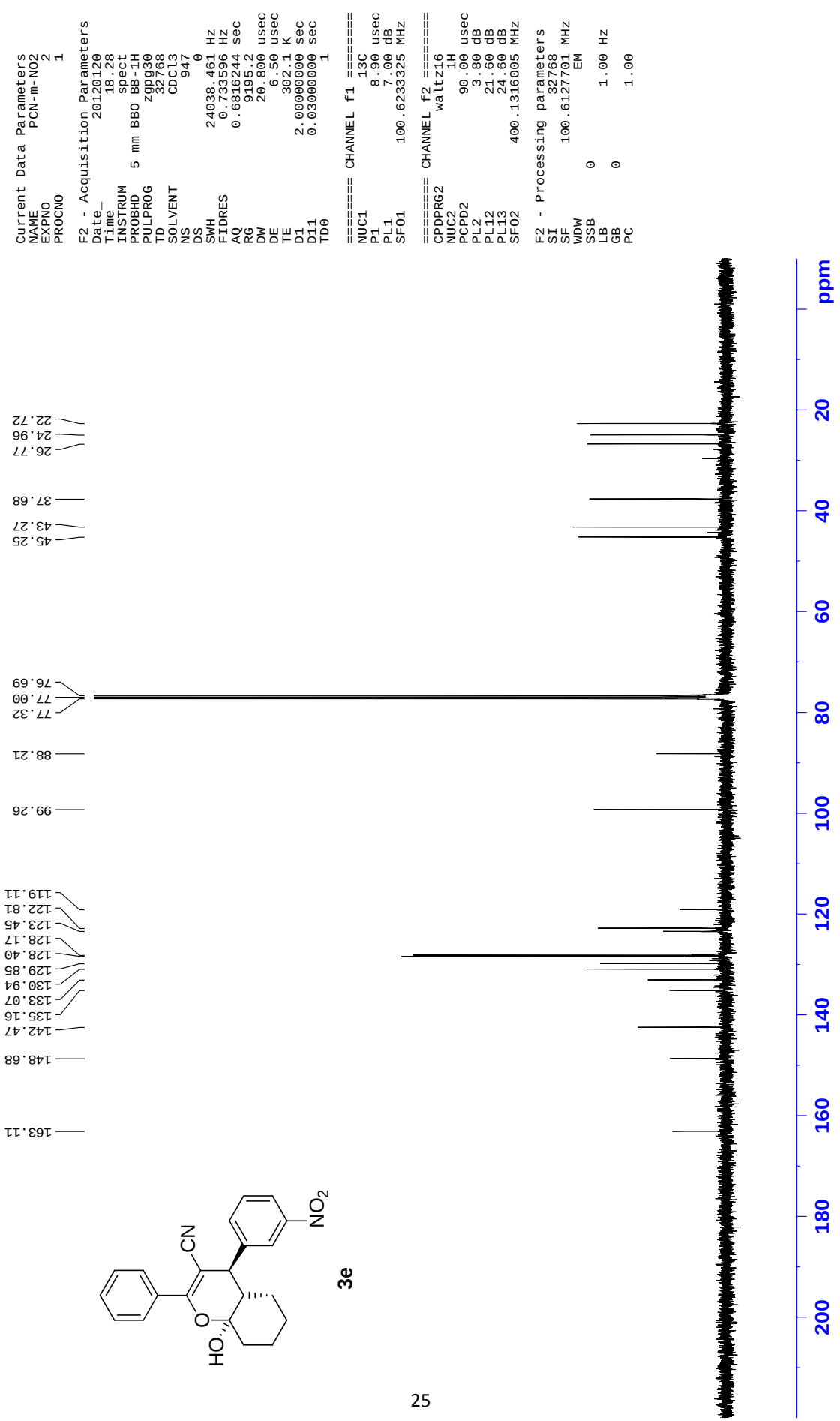
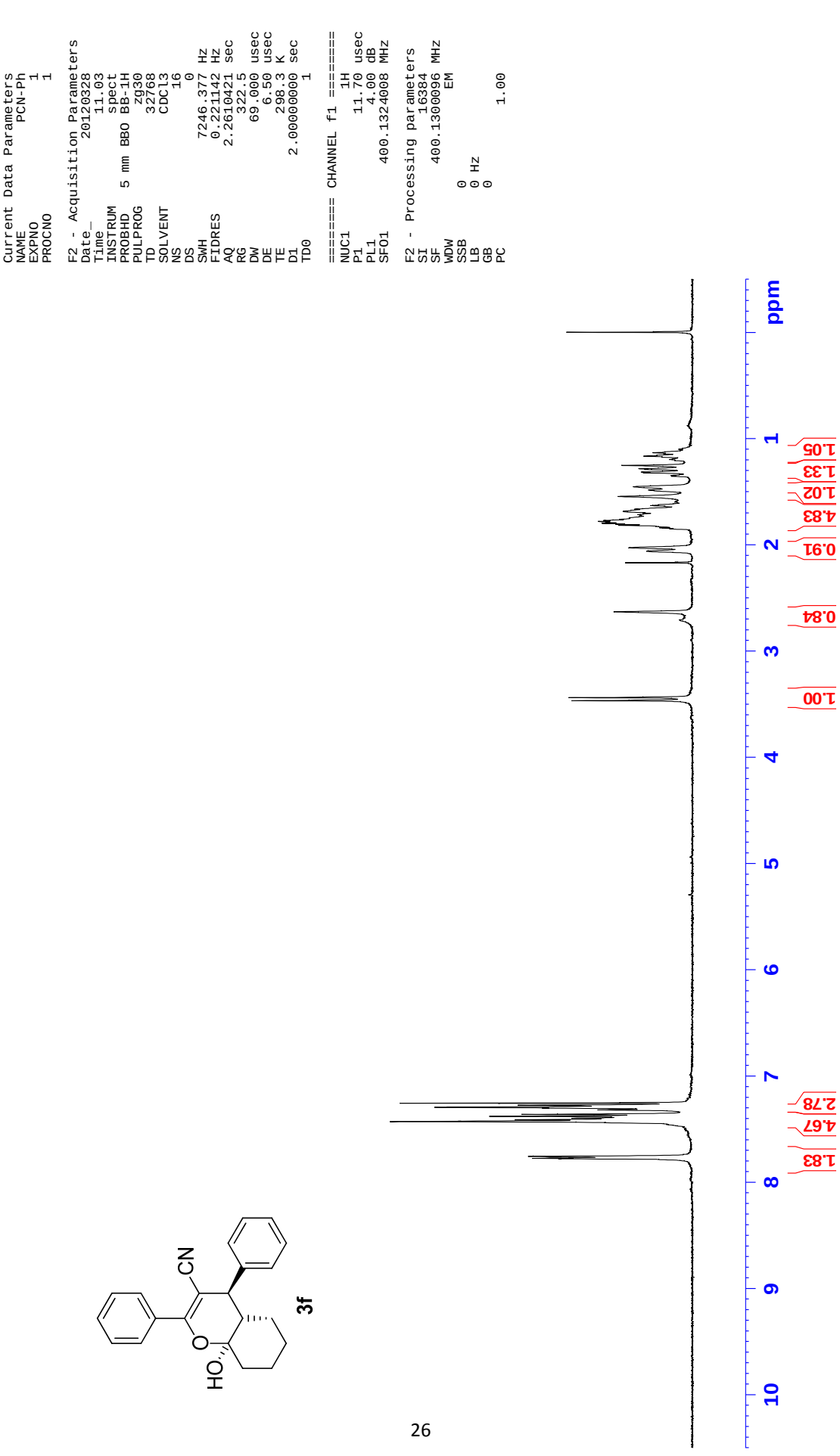
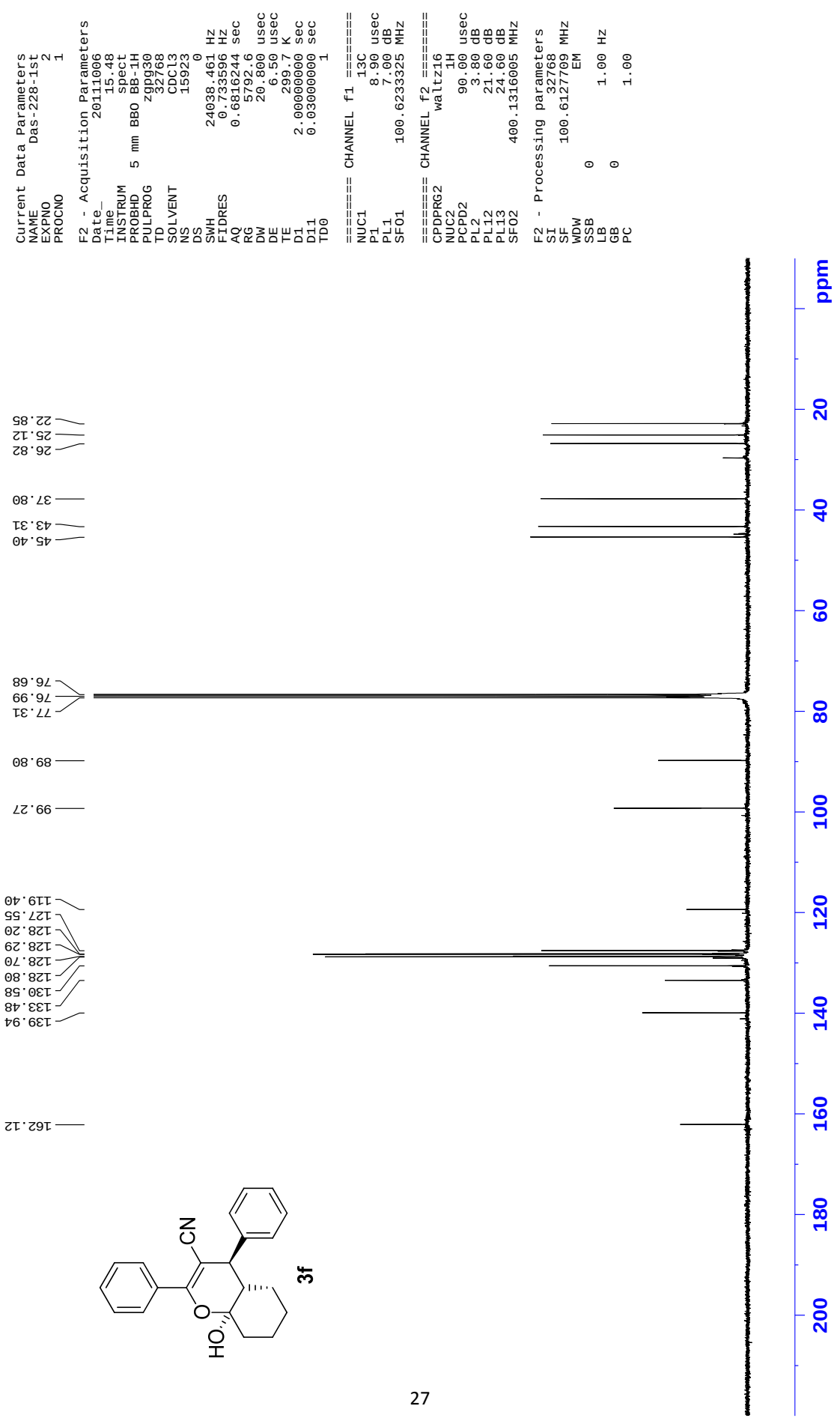
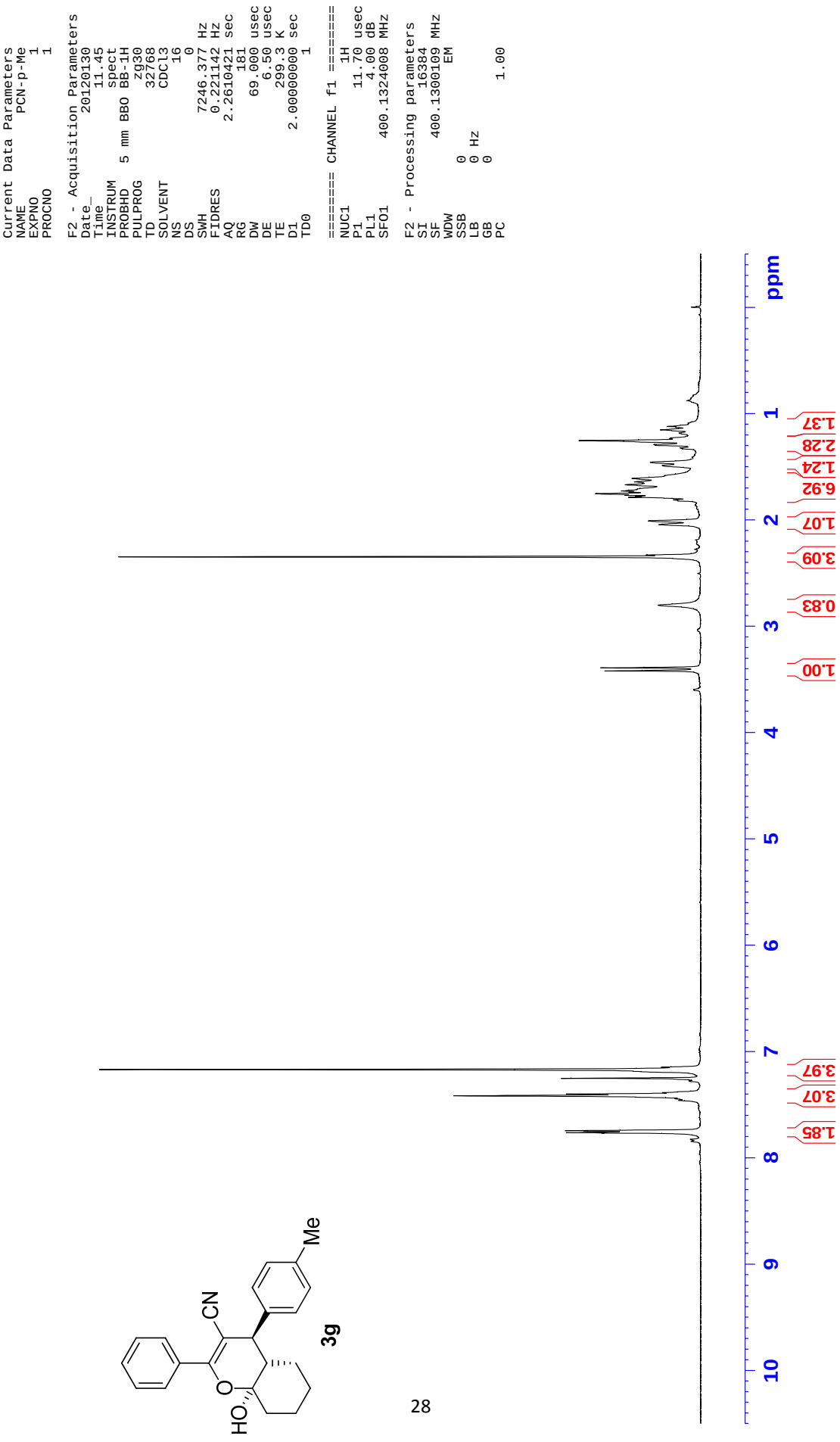


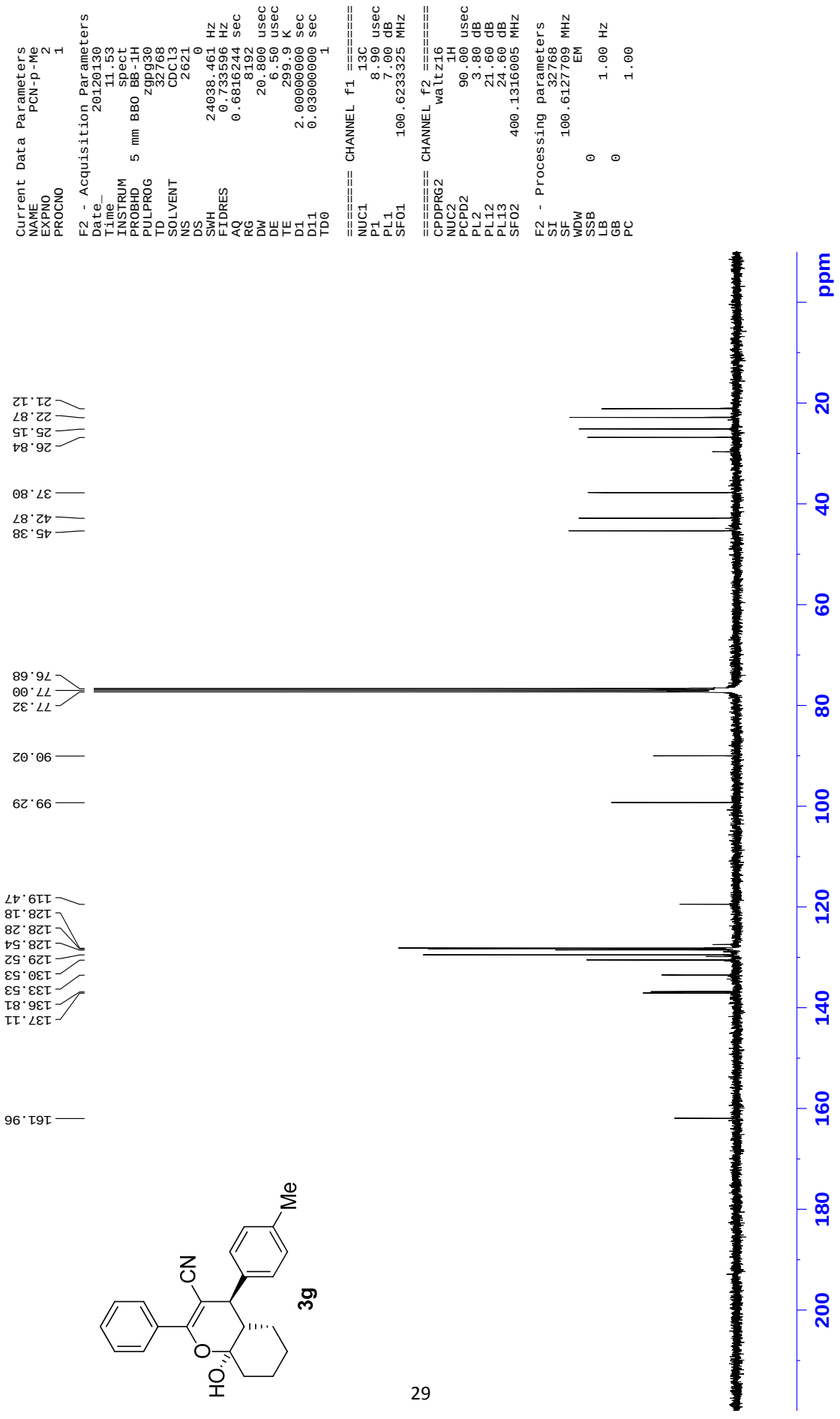
3e

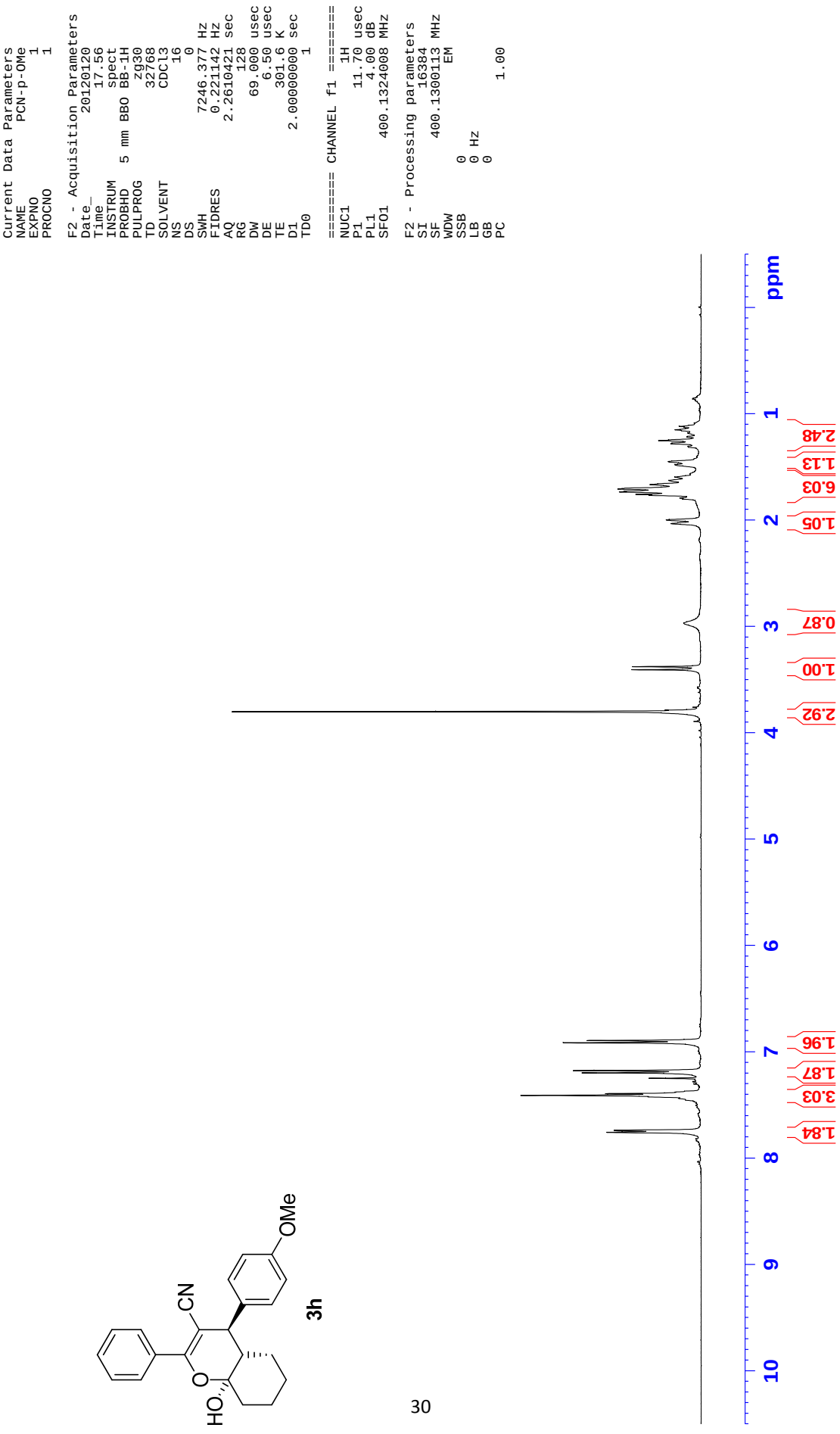

Current Data Parameters
NAME PCN-m-NO2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120120
Time_ 18.28
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 947
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 302.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127701 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.72
24.96
26.77
37.68
43.27
45.25
76.69
77.00
77.32
88.21
99.26
119.11
122.81
123.45
128.17
128.40
129.85
130.94
133.07
135.16
142.47
148.68
163.11
ppm
20
40
60
80
100
120
140
160
180
200
CN
NO2
HO
O
3e

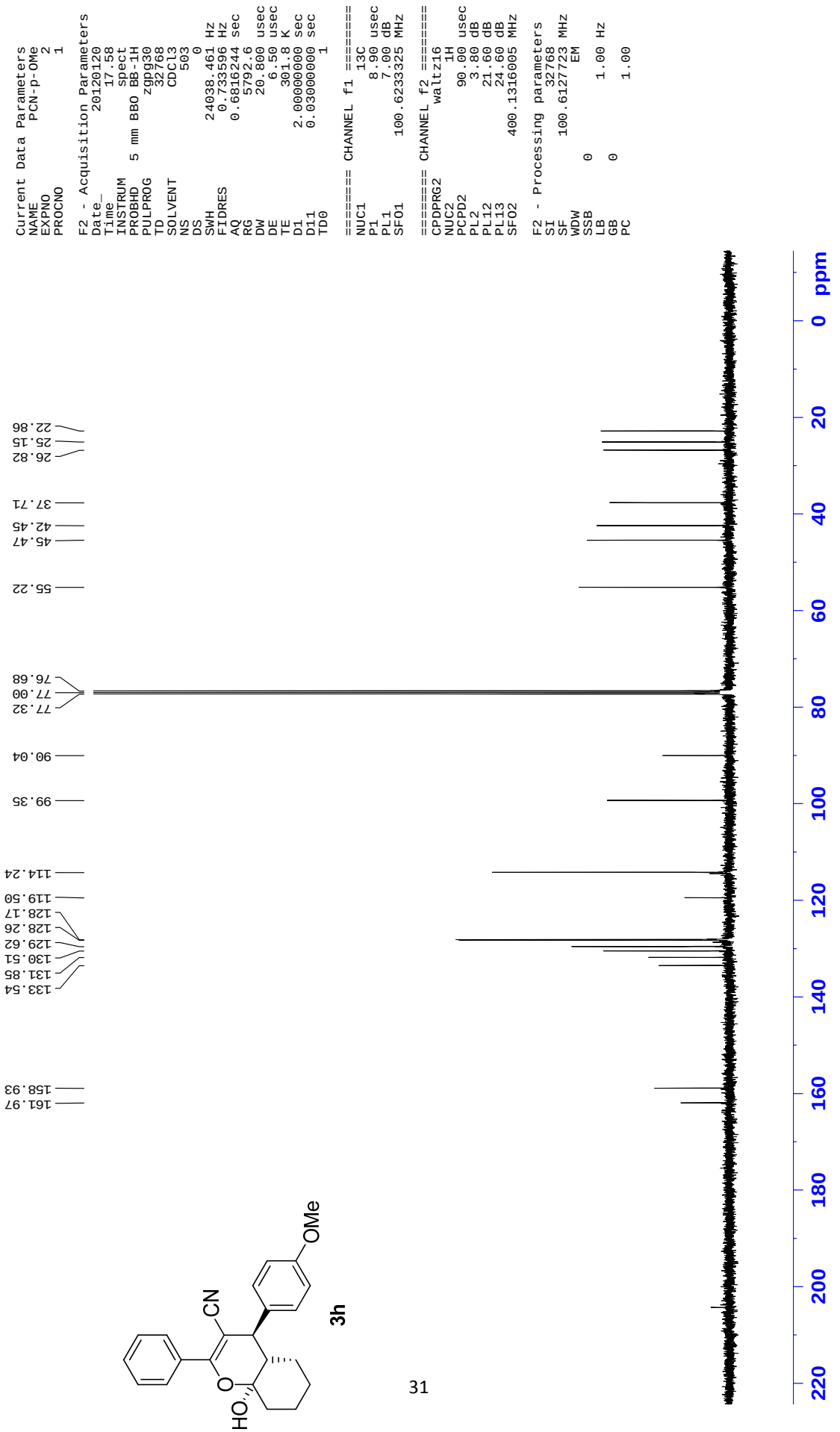

Current Data Parameters
NAME PCN-Ph
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120328
Time 11.03
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 322.5
DW 69.000 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1.05
1.33
1.02
4.83
0.91
0.84
1.00
2.78
4.67
1.83
CN
O
HO
3f

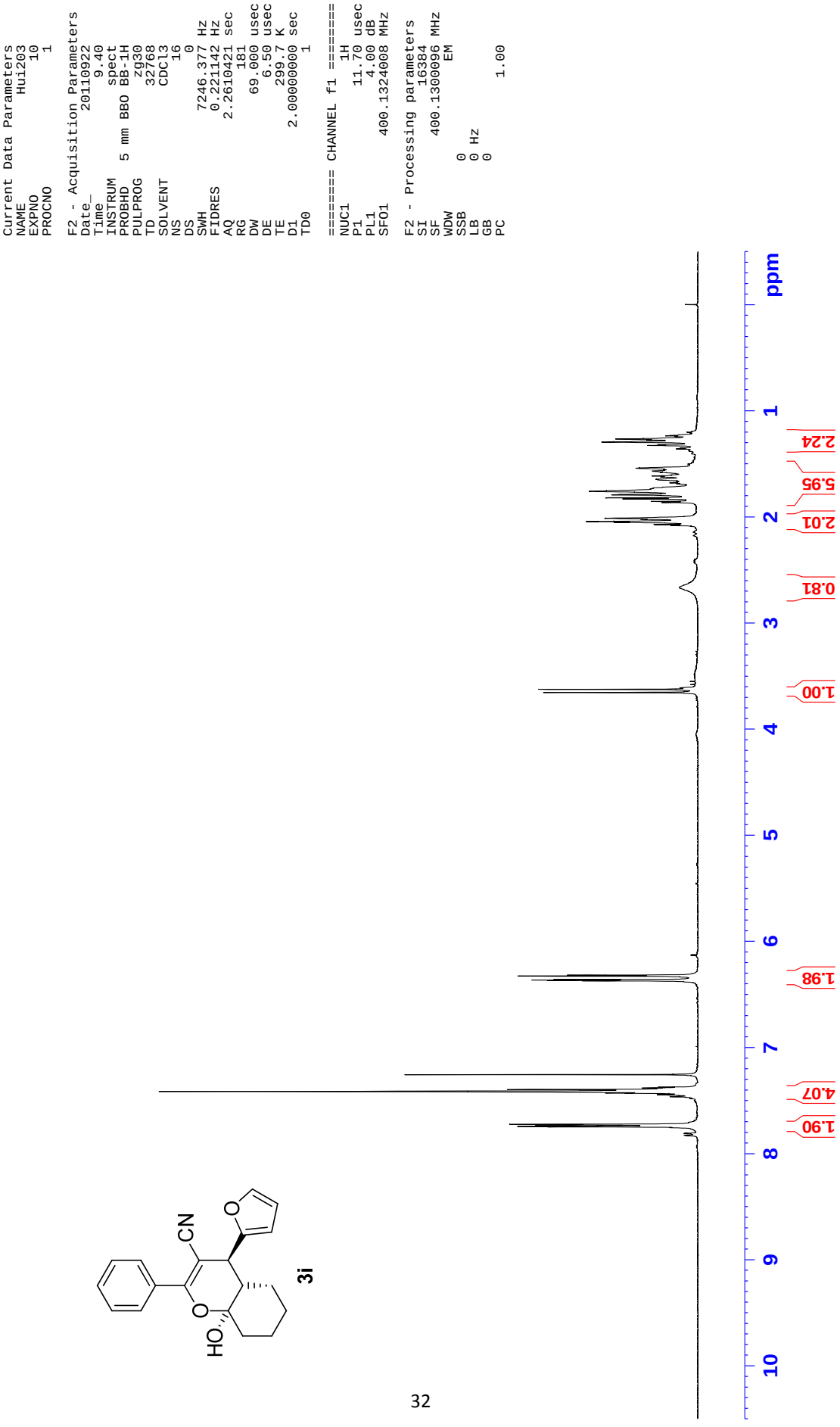

Current Data Parameters
NAME Das-228-1st
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111006
Time_ 15.48
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 15923
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127709 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.85
25.12
26.82
37.80
43.31
45.40
76.68
76.99
77.31
89.80
99.27
119.40
127.55
128.20
128.29
128.70
128.80
130.58
133.48
139.94
162.12
ppm
20
40
60
80
100
120
140
160
180
200
CN
O
HO
3f

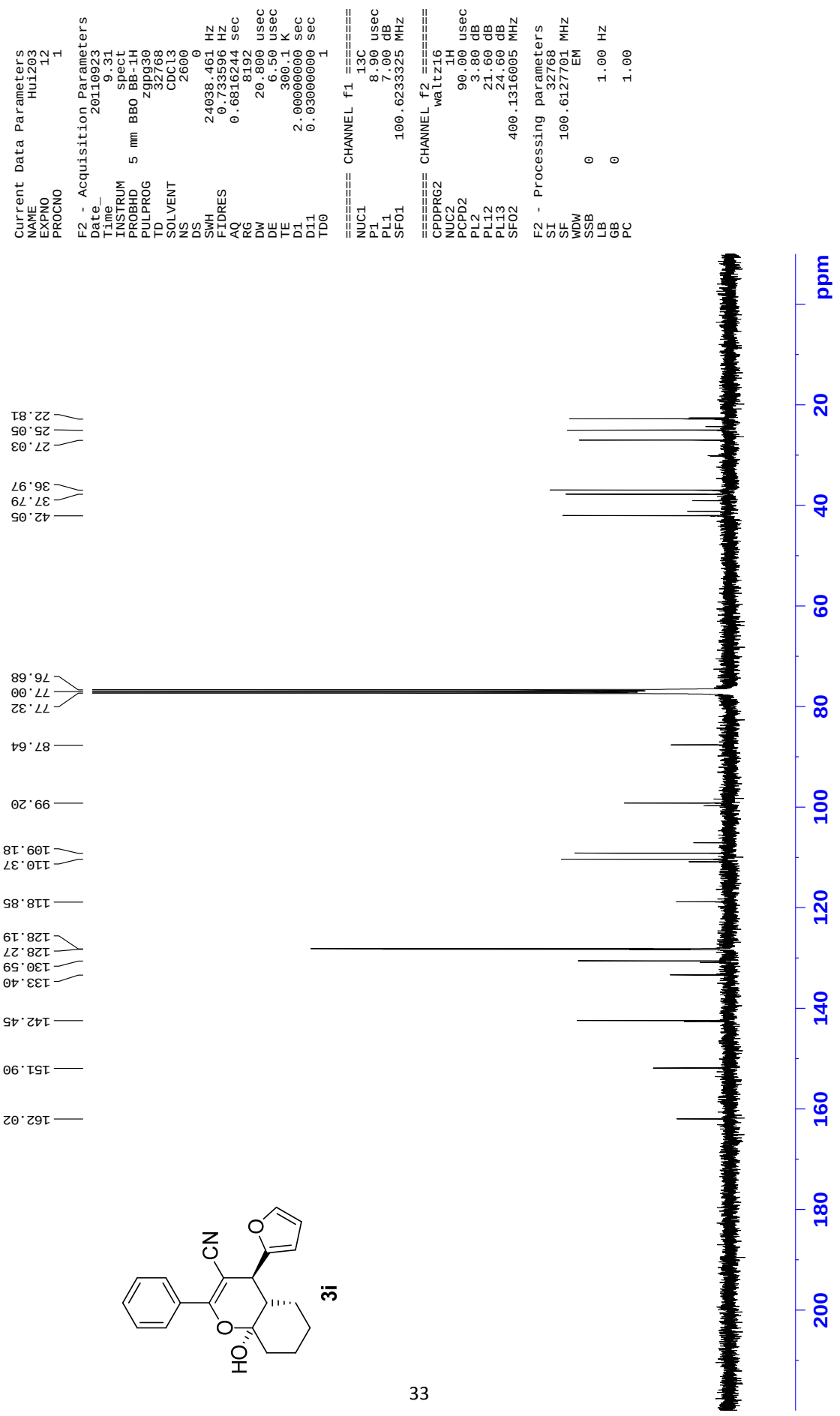

Current Data Parameters
NAME PCN-p-Me
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120130
Time 11.45
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 181
DW 69.000 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300109 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1.37
2.28
1.24
6.92
1.07
3.09
0.83
1.00
3.97
3.07
1.85
CN
Me
HO
O
3g


Current Data Parameters
NAME PCN-p-Me
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120130
Time_ 11.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2621
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127709 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
21.12
22.87
25.15
26.84
37.80
42.87
45.38
76.68
77.00
77.32
90.02
99.29
119.47
128.18
128.28
128.54
129.52
130.53
133.53
136.81
137.11
161.96
ppm
20
40
60
80
100
120
140
160
180
200
CN
Me
HO
O
3g


Current Data Parameters
NAME PCN-p-OMe
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120120
Time 17.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 128
DW 69.000 usec
DE 6.50 usec
TE 301.6 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300113 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
2.48
1.13
6.03
1.05
0.87
1.00
2.92
1.96
1.87
3.03
1.84
CN
OMe
HO
O
3h


Current Data Parameters
NAME PCN-p-OMe
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120120
Time_ 17.58
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 503
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 5792.6
DW 20.800 usec
DE 6.50 usec
TE 301.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127723 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.86
25.15
26.82
37.71
42.45
45.47
55.22
76.68
77.00
77.32
90.04
99.35
114.24
119.50
128.17
128.26
129.62
130.51
131.85
133.54
158.93
161.97
0 ppm
20
40
60
80
100
120
140
160
180
200
220
CN
OMe
O
HO
3h


Current Data Parameters
NAME Hui203
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110922
Time 9.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 181
DW 69.000 usec
DE 6.50 usec
TE 299.7 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
2.24
5.95
2.01
0.81
1.00
1.98
4.07
1.90
CN
O
O
HO
3i

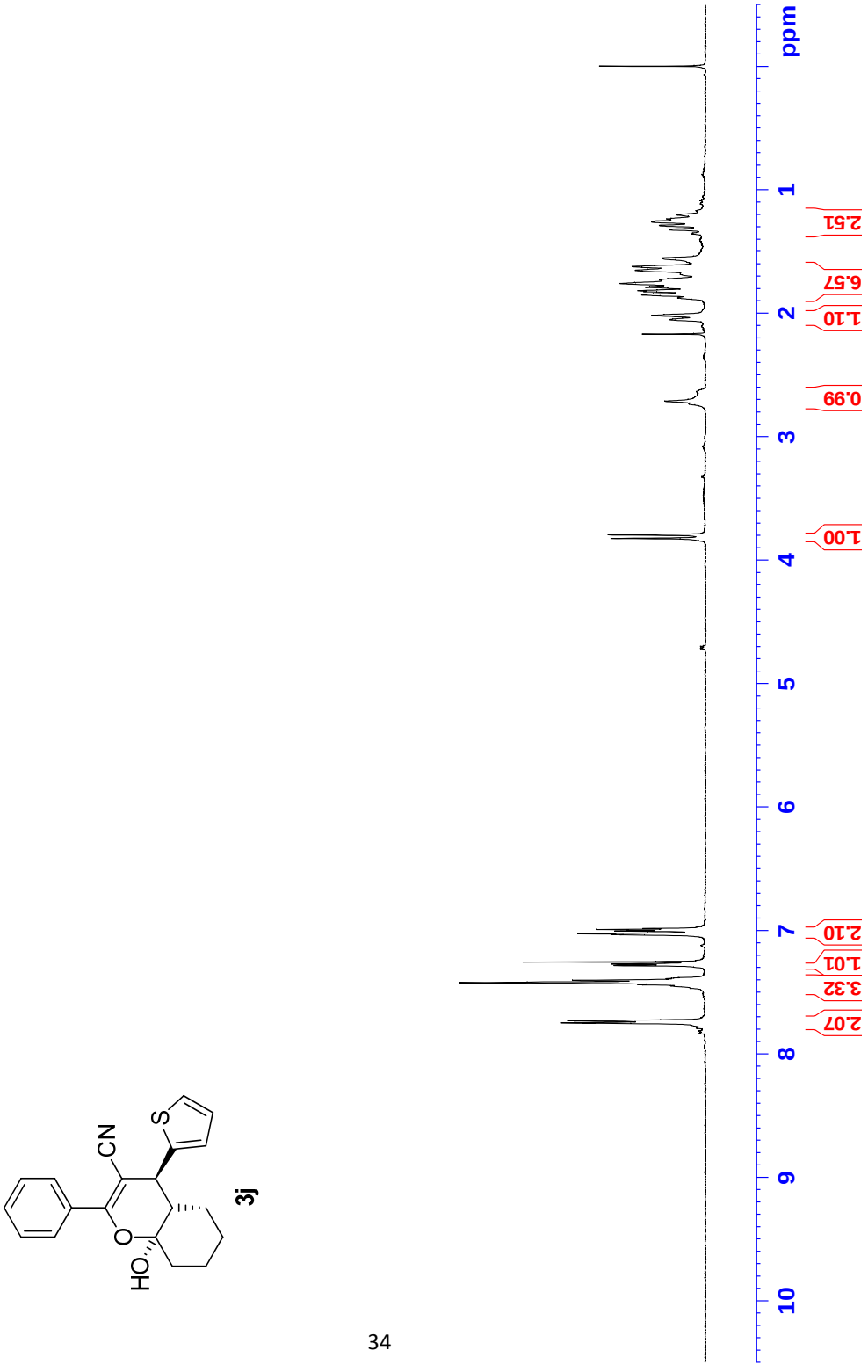

Current Data Parameters
NAME Hui203
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110923
Time_ 9.31
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 2600
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127701 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.81
25.05
27.03
36.97
37.79
42.05
76.68
77.00
77.32
87.64
99.20
109.18
110.37
118.85
128.19
128.27
130.59
133.40
142.45
151.90
162.02
ppm
20
40
60
80
100
120
140
160
180
200
CN
O
O
HO
3i

```
Current Data Parameters
NAME       PCN-2-thienyl
EXPNO                  2
PROCNO                 1

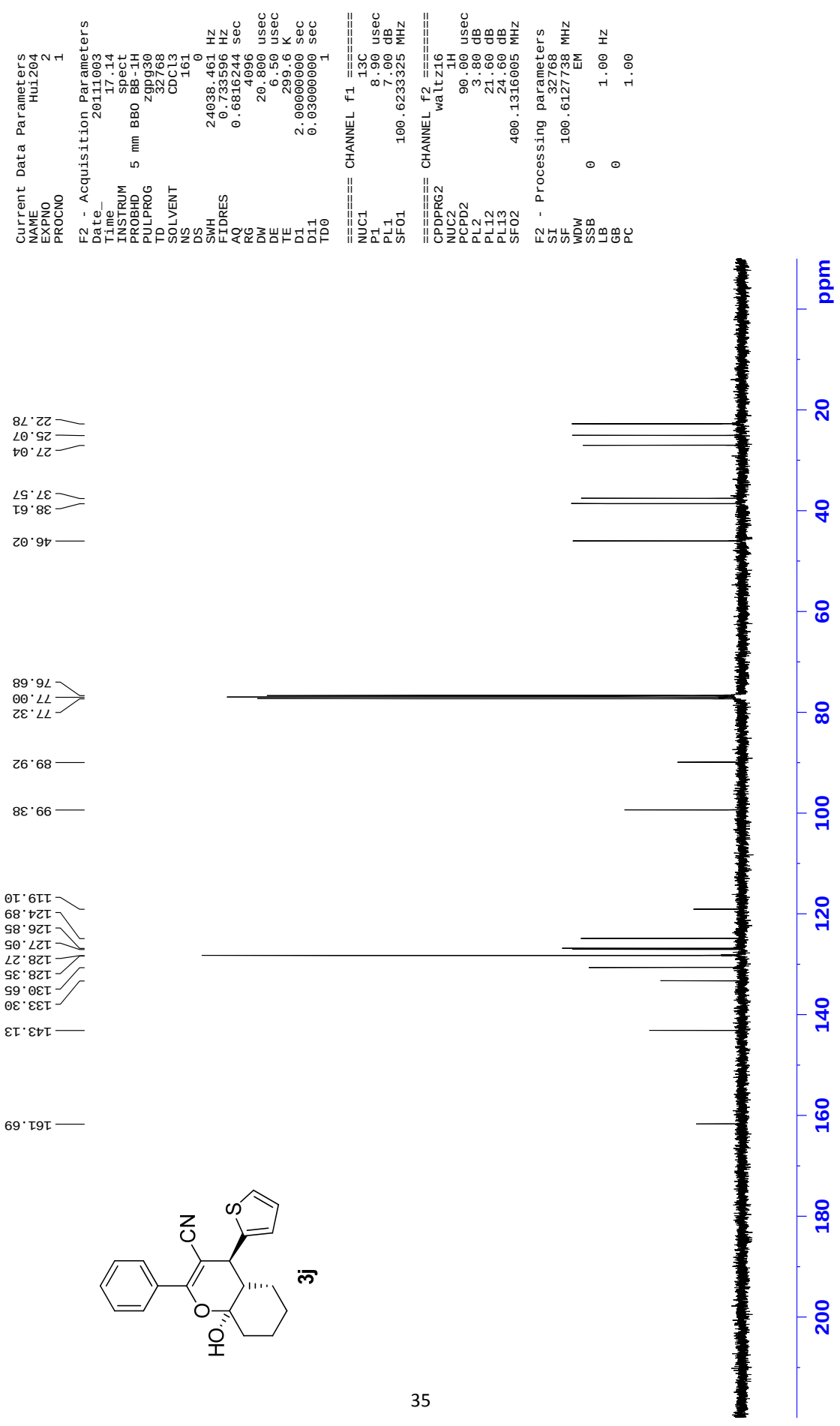
F2 - Acquisition Parameters
Date_           20120327
Time                13.30
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2610421 sec
RG                   181
DW                69.000 usec
DE                 6.50 usec
TE                298.4 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 1H
P1               11.70 usec
PL1               4.00 dB
SFO1        400.1324008 MHz

F2 - Processing parameters
SI                 16384
SF          400.1300096 MHz
WDW                   EM
SSB                    0
LB                 0 Hz
GB                     0
PC                  1.00
```



3j

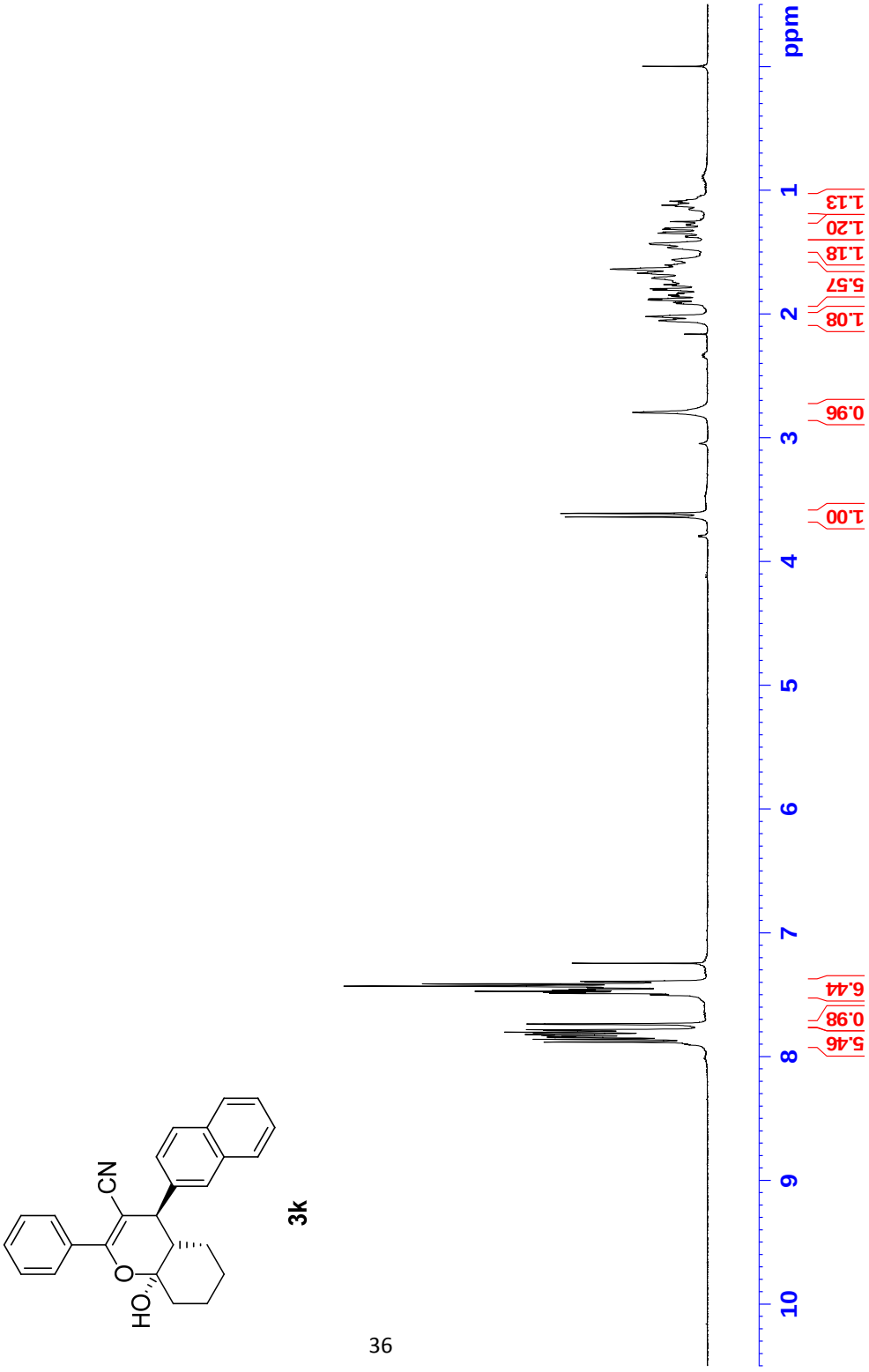

Current Data Parameters
NAME Hui204
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111003
Time_ 17.14
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 161
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 4096
DW 20.800 usec
DE 6.50 usec
TE 299.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127738 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.78
25.07
27.04
37.57
38.61
46.02
76.68
77.00
77.32
89.92
99.38
119.10
124.89
126.85
127.05
128.27
128.35
130.65
133.30
143.13
161.69
ppm
20
40
60
80
100
120
140
160
180
200
CN
S
O
HO
3j

```
Current Data Parameters
NAME         PCN-2-Nath
EXPNO                 2
PROCNO                1

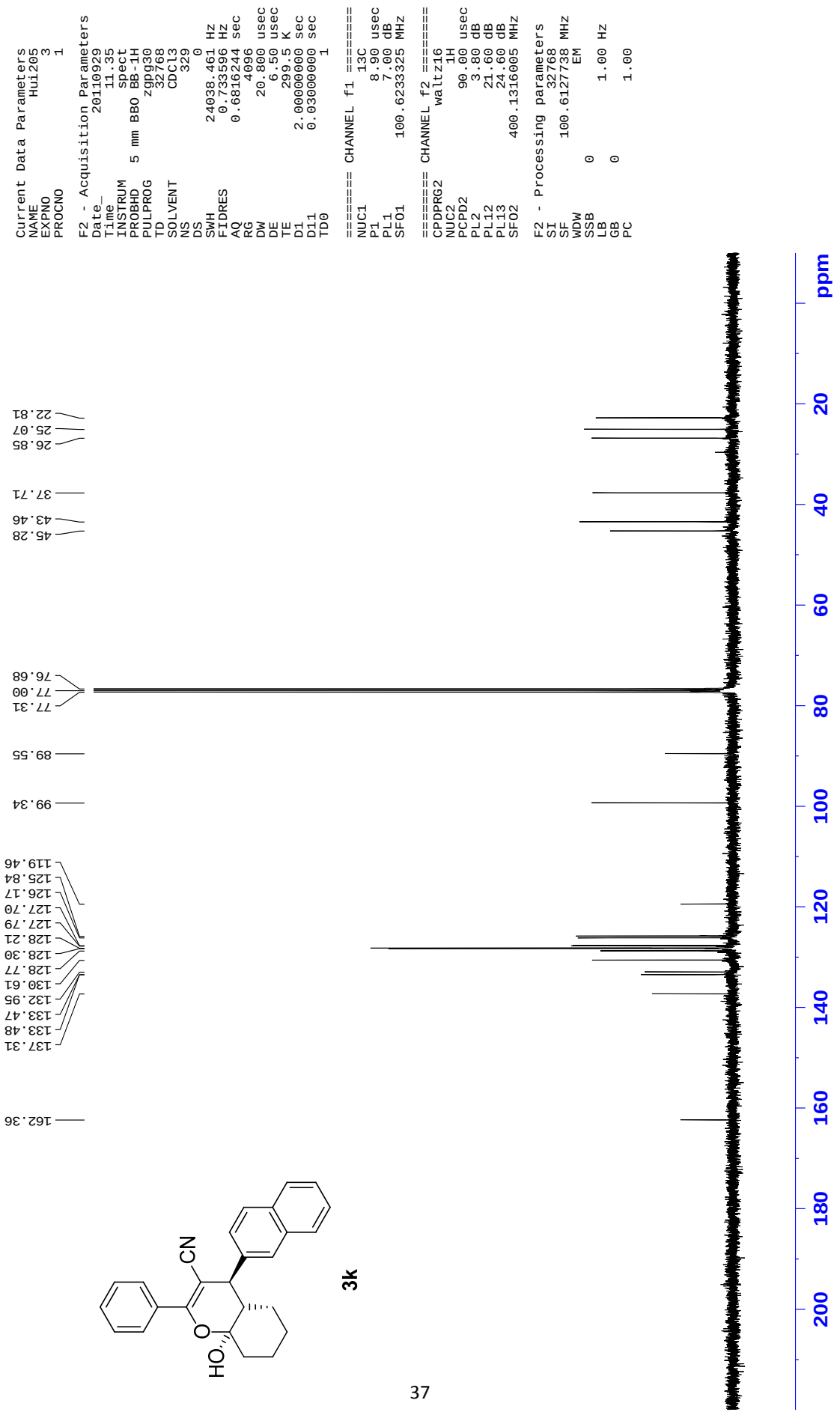
F2 - Acquisition Parameters
Date_           20120327
Time                13.11
INSTRUM             spect
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             7246.377 Hz
FIDRES          0.221142 Hz
AQ              2.2610421 sec
RG                   128
DW                69.000 usec
DE                 6.50 usec
TE                298.5 K
D1              2.00000000 sec
TD0                    1

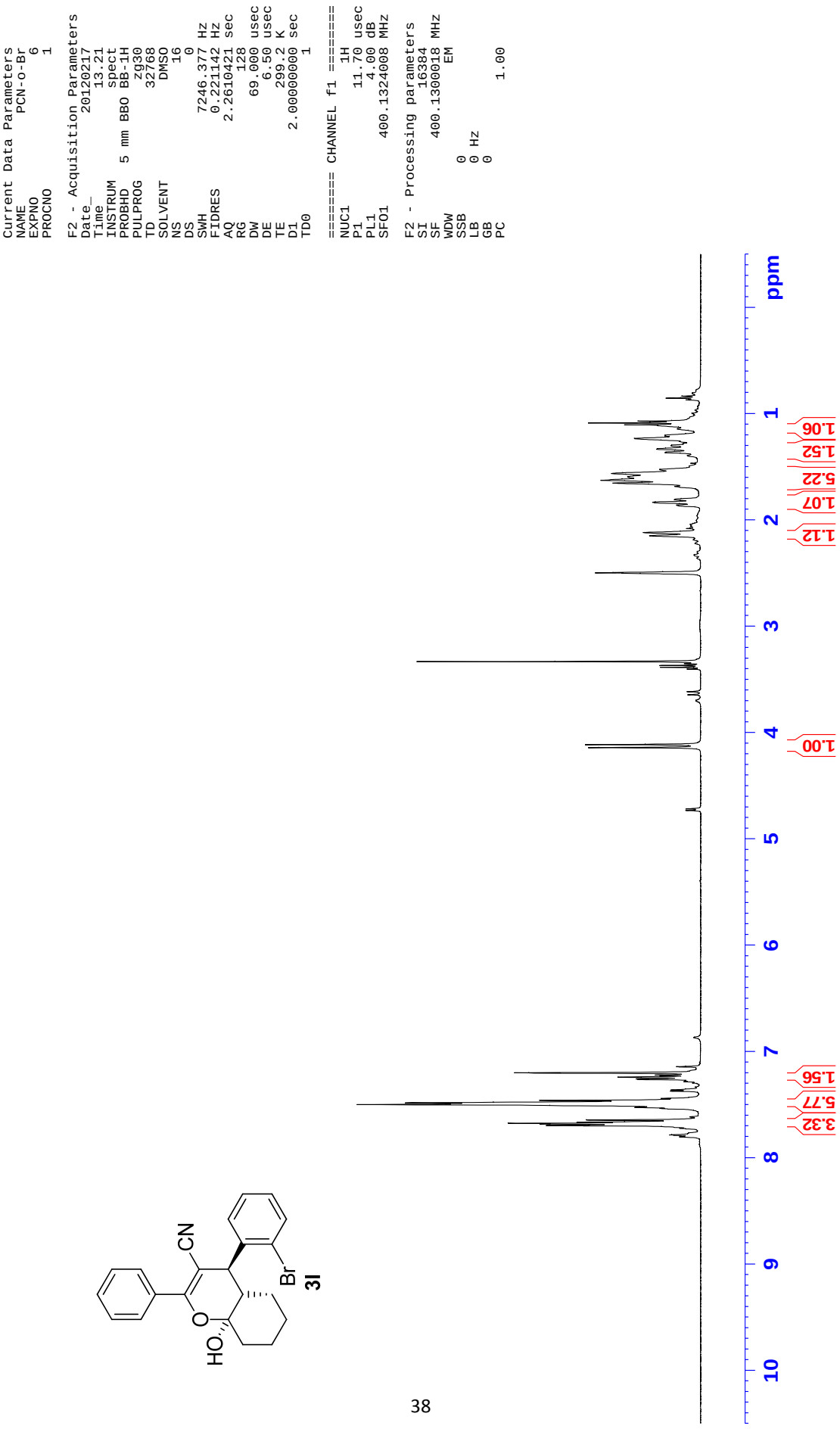
======== CHANNEL f1 ========
NUC1                  1H
P1                11.70 usec
PL1                4.00 dB
SFO1        400.1324008 MHz

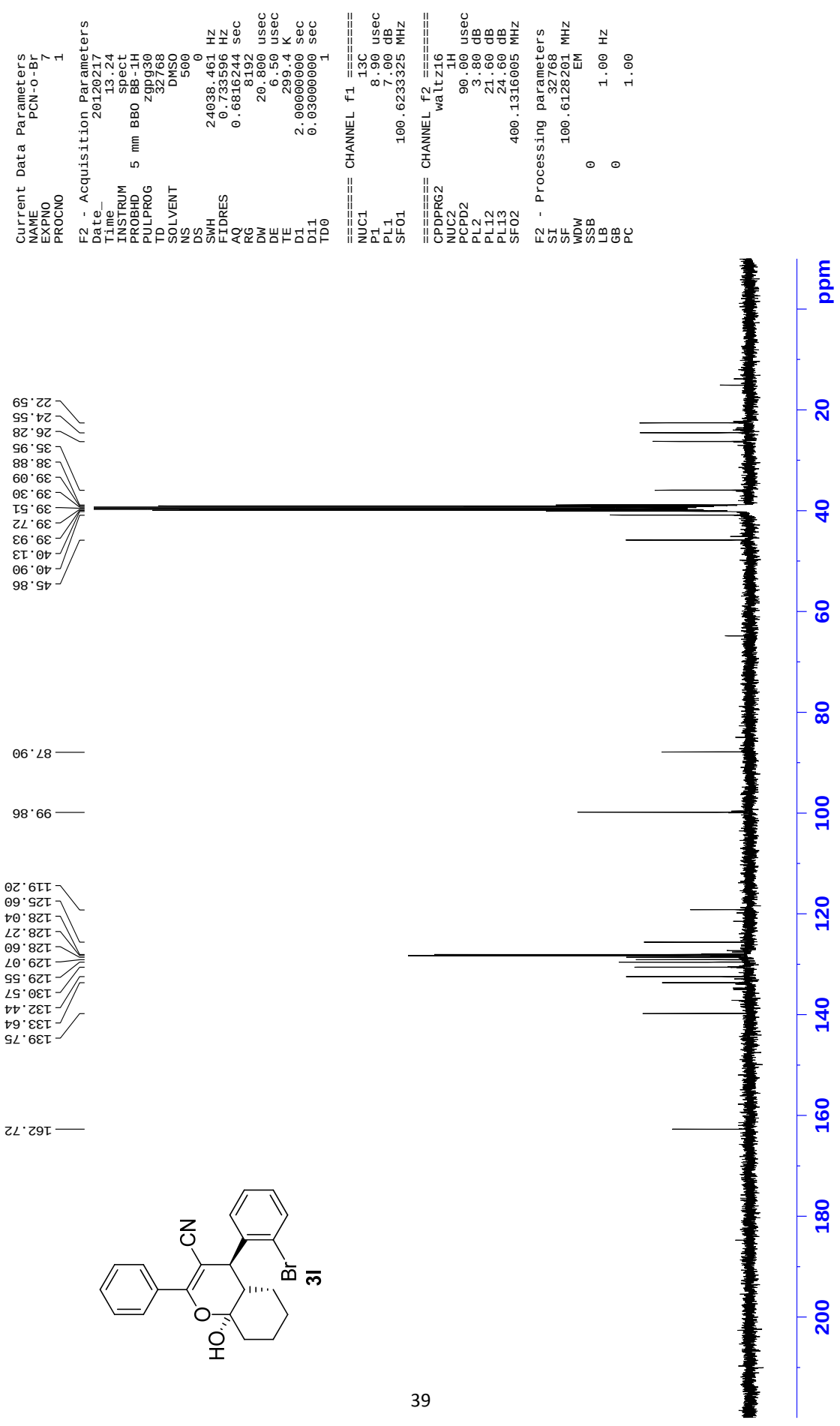
F2 - Processing parameters
SI                 16384
SF          400.1300131 MHz
WDW                   EM
SSB                    0
LB                 0 Hz
GB                     0
PC                  1.00
```

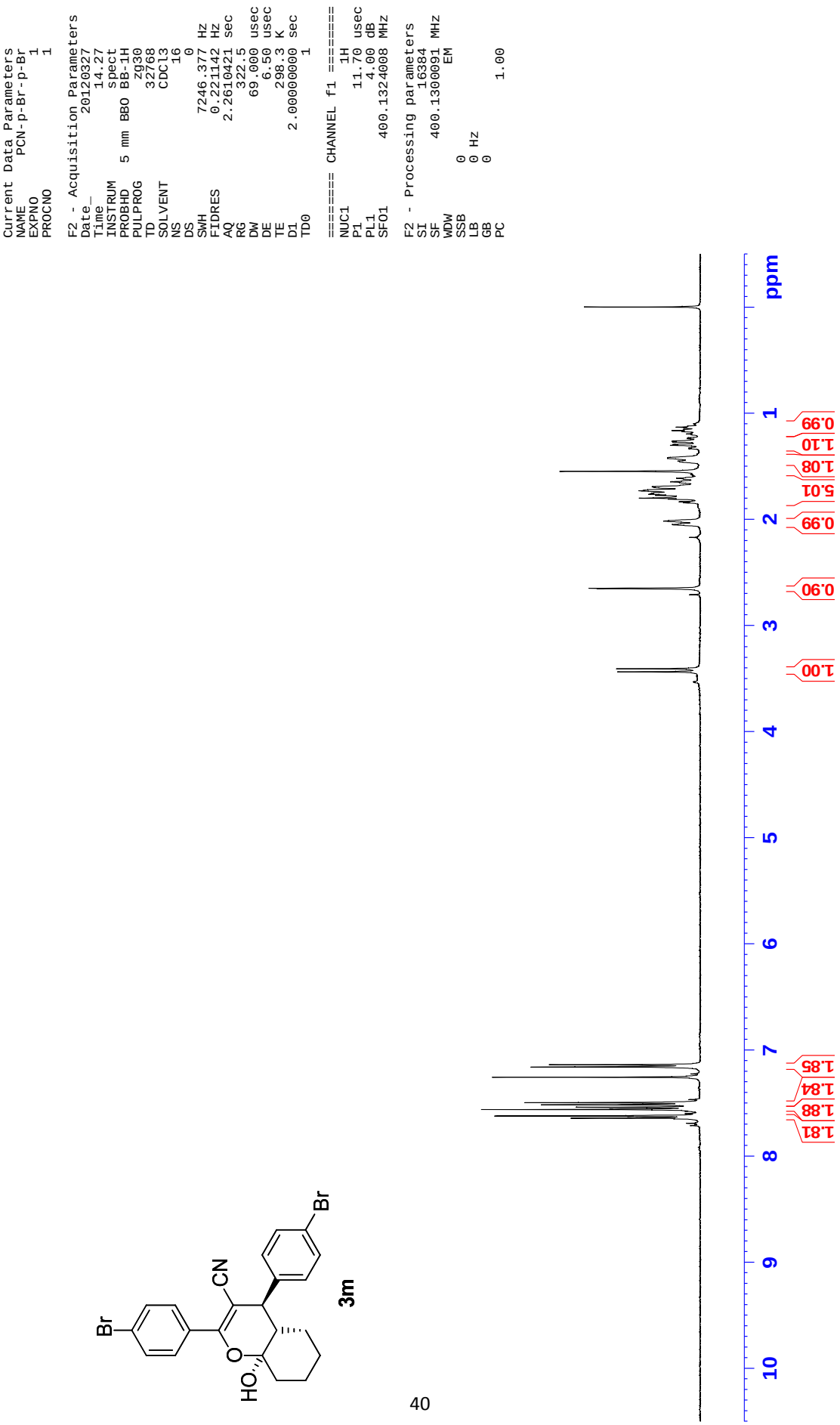


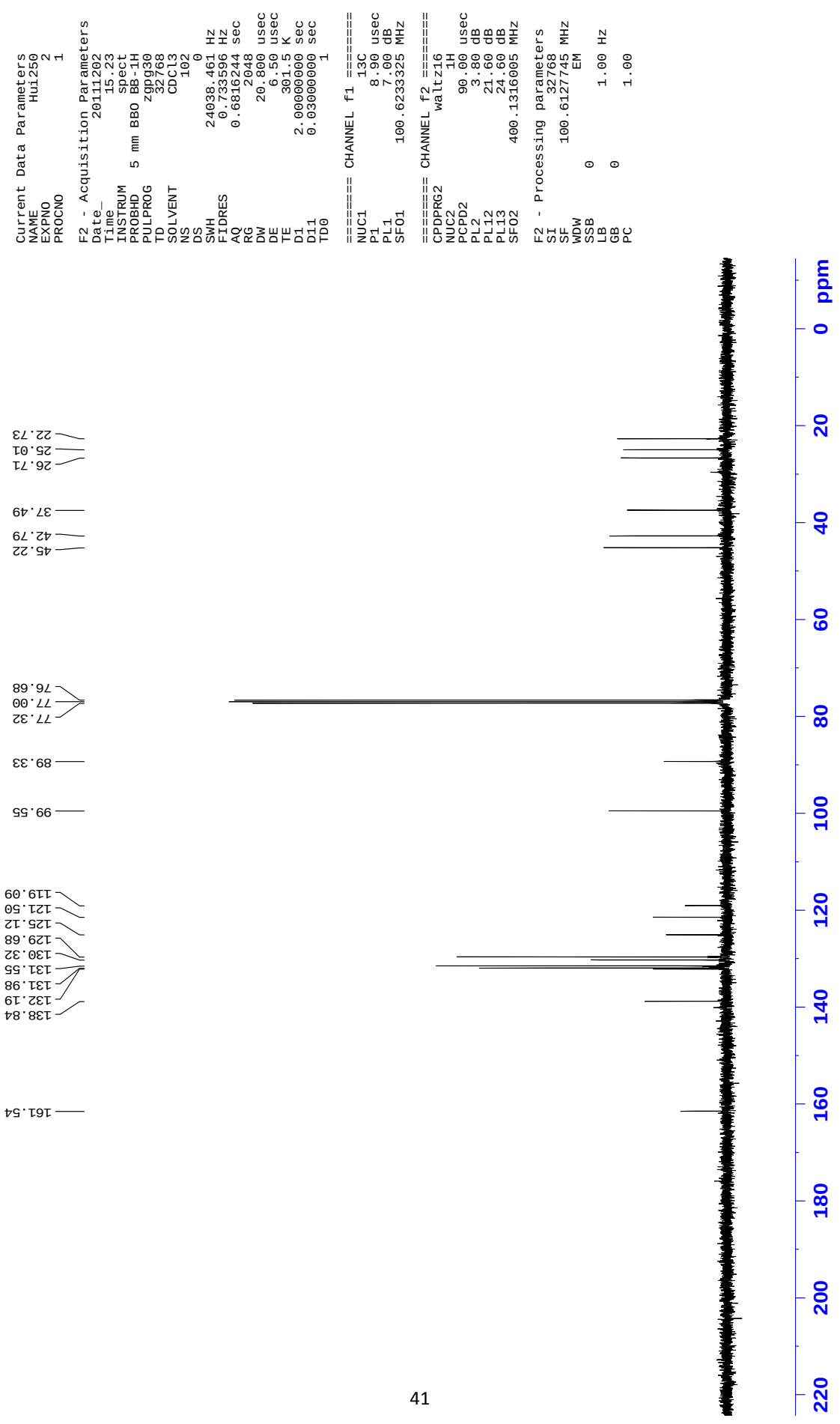
3k

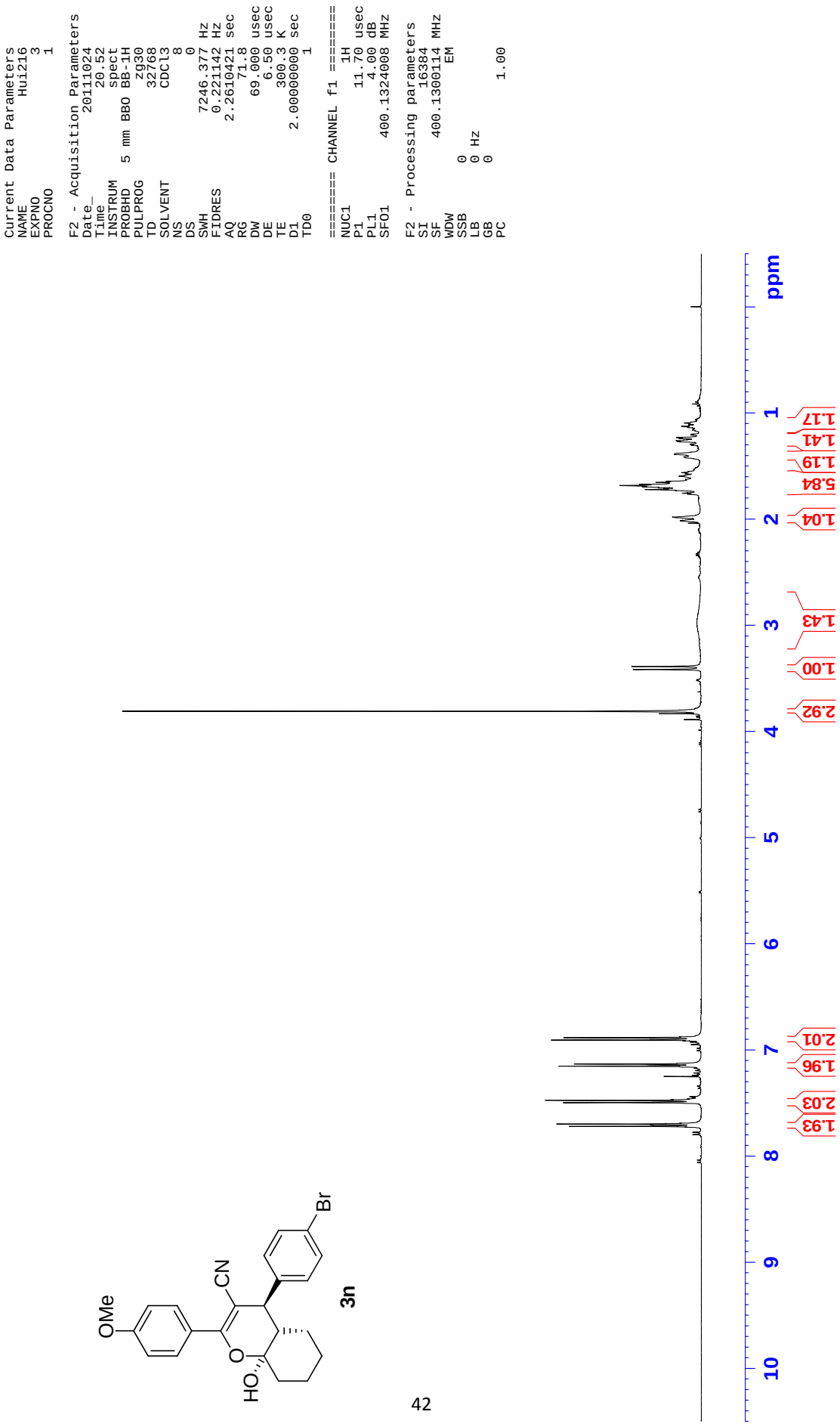

Current Data Parameters
NAME Hui205
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110929
Time_ 11.35
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 329
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 4096
DW 20.800 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127738 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.81
25.07
26.85
37.71
43.46
45.28
76.68
77.00
77.31
89.55
99.34
119.46
125.84
126.17
127.70
127.79
128.21
128.30
128.77
130.61
132.95
133.47
133.48
137.31
162.36
ppm
20
40
60
80
100
120
140
160
180
200
CN
O
HO
3k

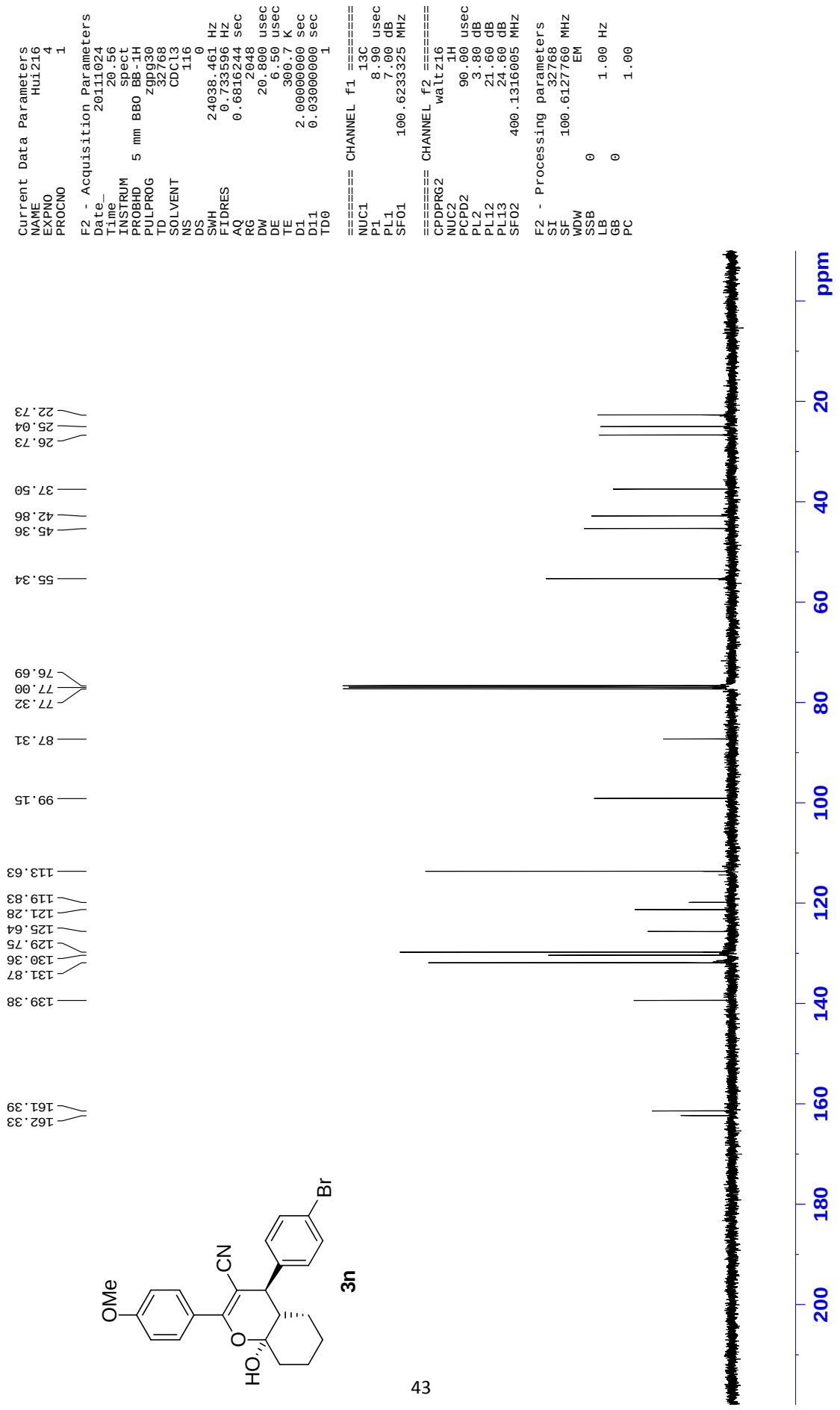

Current Data Parameters
NAME PCN-o-Br
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120217
Time 13.21
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 128
DW 69.000 usec
DE 6.50 usec
TE 299.2 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300018 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1.06
1.52
5.22
1.07
1.12
1.00
1.56
5.77
3.32
CN
Br
O
HO
3l

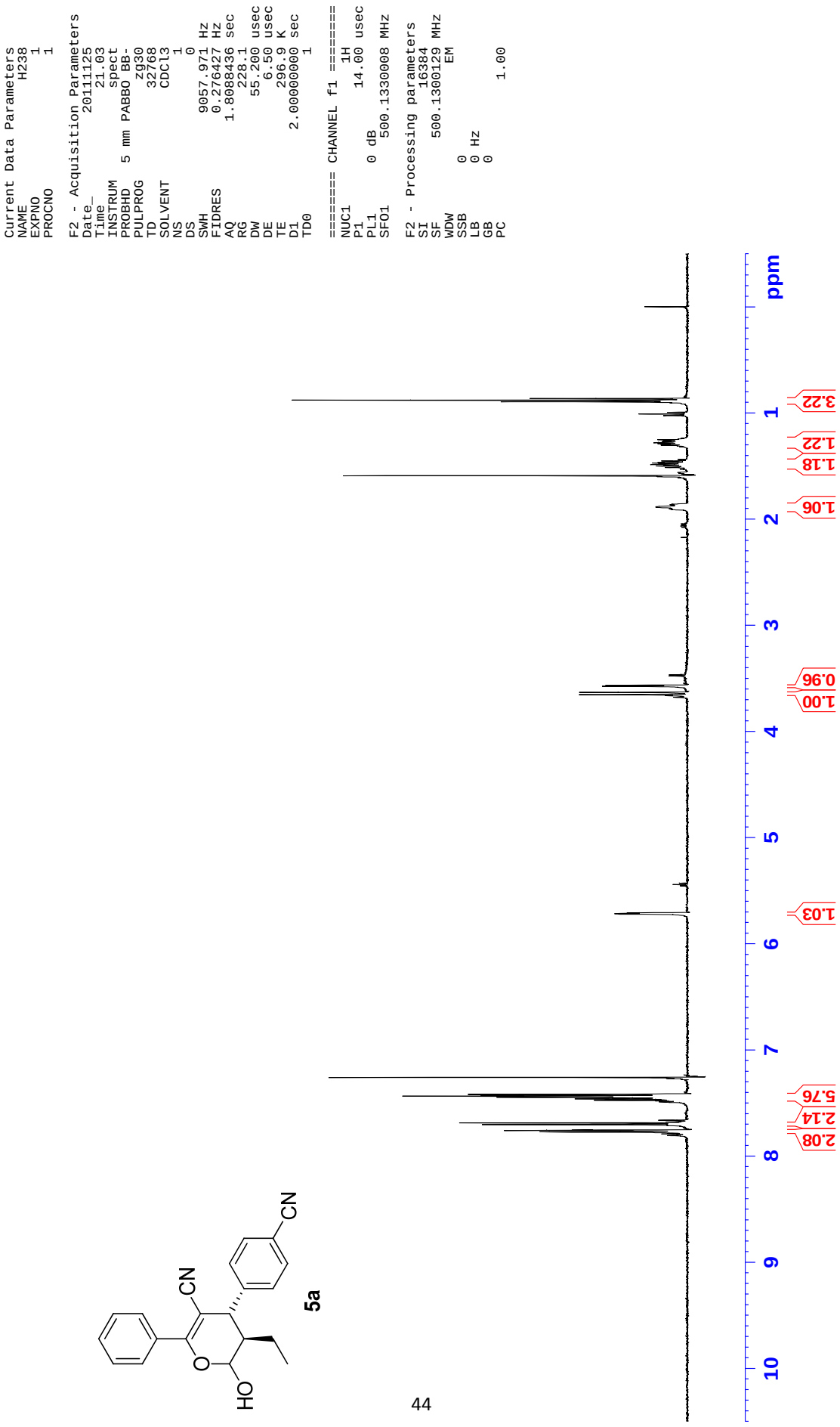

Current Data Parameters
NAME PCN-o-Br
EXPNO 7
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120217
Time_ 13.24
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 500
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 8192
DW 20.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128201 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.59
24.55
26.28
35.95
38.88
39.09
39.30
39.51
39.72
39.93
40.13
40.90
45.86
87.90
99.86
119.20
125.60
128.04
128.27
128.60
129.07
129.55
130.57
132.44
133.64
139.75
162.72
ppm
20
40
60
80
100
120
140
160
180
200
CN
Br
O
HO
3l

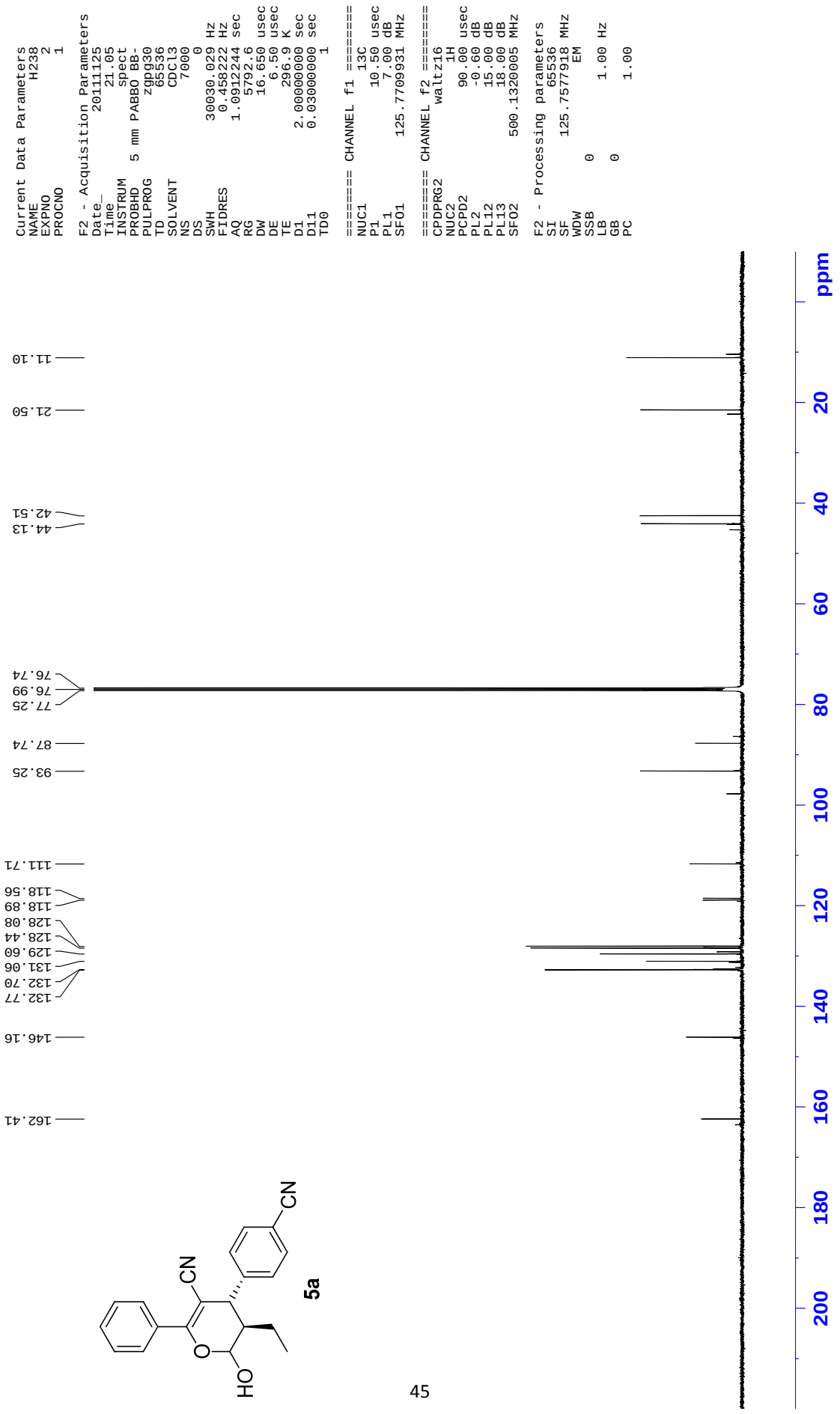

Current Data Parameters
NAME PCN-p-Br-p-Br
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120327
Time 14.27
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 322.5
DW 69.000 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
0.99
1.10
1.08
5.01
0.99
0.90
1.00
1.85
1.84
1.88
1.81
Br
CN
Br
O
HO
3m

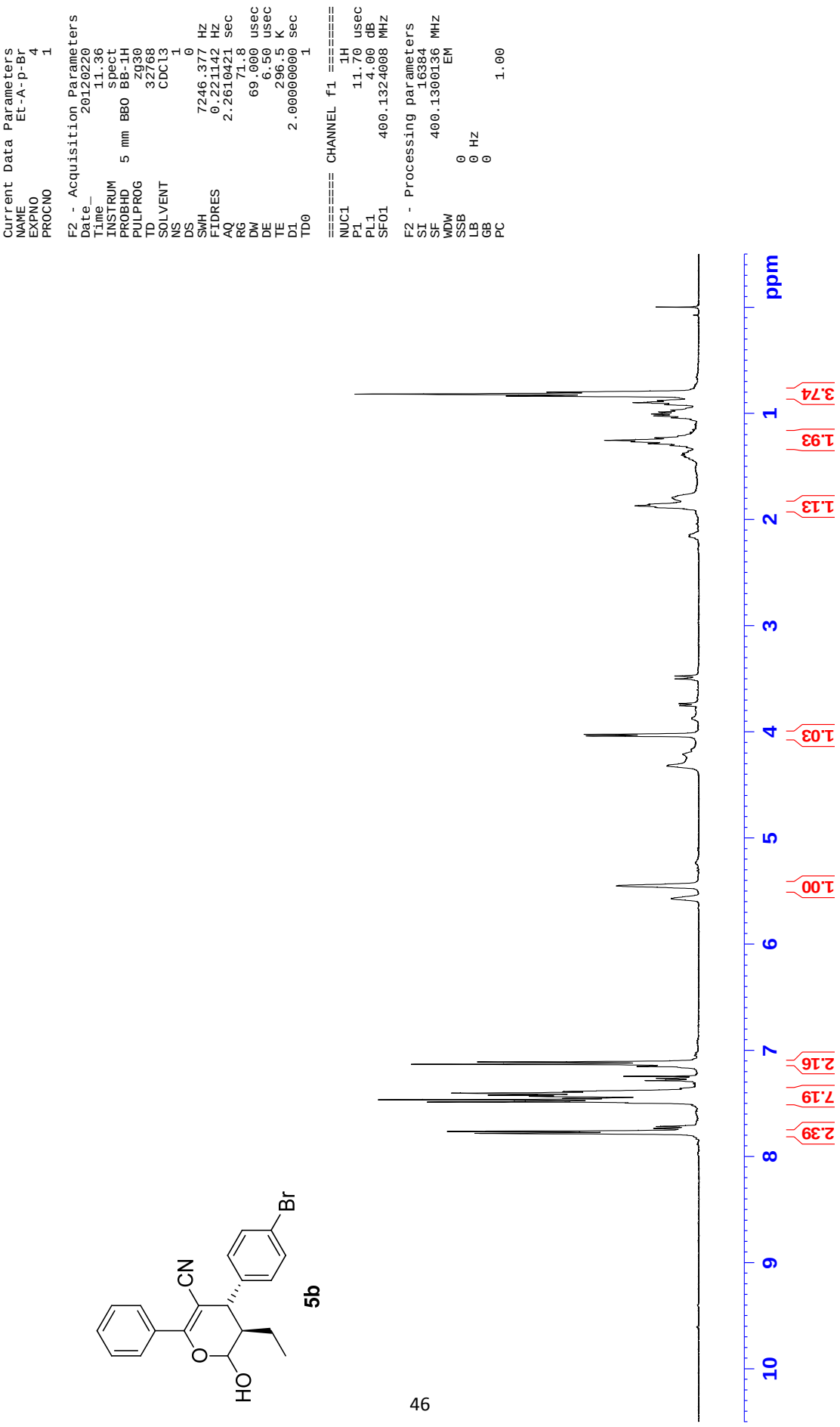

Current Data Parameters
NAME Hui250
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111202
Time_ 15.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 102
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 2048
DW 20.800 usec
DE 6.50 usec
TE 301.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.73
25.01
26.71
37.49
42.79
45.22
76.68
77.00
77.32
89.33
99.55
119.09
121.50
125.12
129.68
130.32
131.55
131.98
132.19
138.84
161.54
0 ppm
20
40
60
80
100
120
140
160
180
200
220

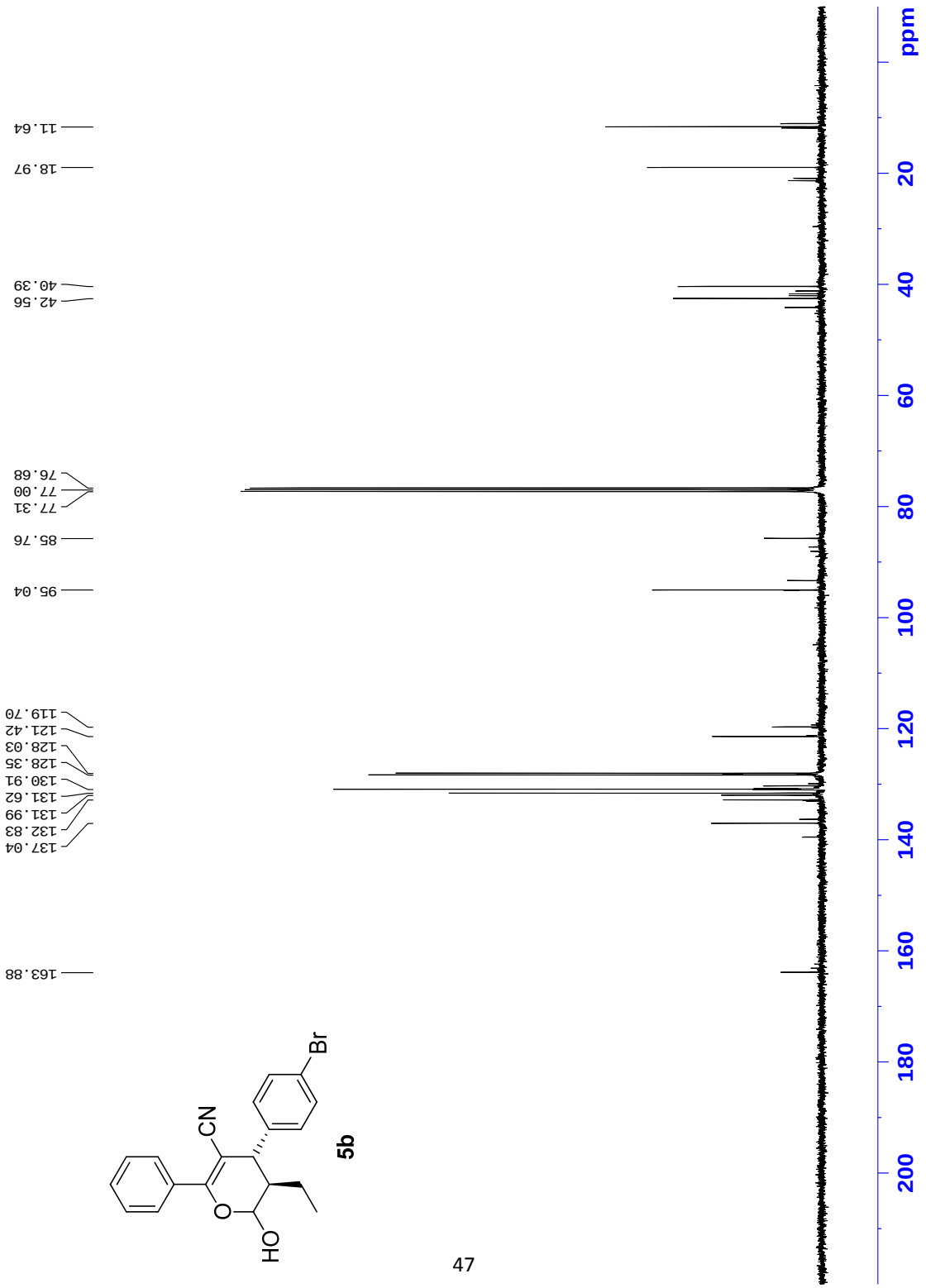

Current Data Parameters
NAME Hui216
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111024
Time 20.52
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 71.8
DW 69.000 usec
DE 6.50 usec
TE 300.3 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300114 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
1.17
1.41
1.19
5.84
1.04
1.43
1.00
2.92
2.01
1.96
2.03
1.93
OMe
CN
Br
O
HO
3n


Current Data Parameters
NAME Hui216
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111024
Time_ 20.56
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 116
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 2048
DW 20.800 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127760 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
22.73
25.04
26.73
37.50
42.86
45.36
55.34
76.69
77.00
77.32
87.31
99.15
113.63
119.83
121.28
125.64
129.75
130.36
131.87
139.38
161.39
162.33
ppm
20
40
60
80
100
120
140
160
180
200
OMe
CN
Br
O
HO
3n


Current Data Parameters
NAME H238
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111125
Time 21.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 1
DS 0
SWH 9057.971 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 228.1
DW 55.200 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0 dB
SFO1 500.1330008 MHz
F2 - Processing parameters
SI 16384
SF 500.1300129 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
3.22
1.22
1.18
1.06
1.00
0.96
1.03
5.76
2.14
2.08
CN
CN
O
HO
5a


Current Data Parameters
NAME H238
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111125
Time_ 21.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 7000
DS 0
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 5792.6
DW 16.650 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 125.7709931 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577918 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
11.10
21.50
42.51
44.13
76.74
76.99
77.25
87.74
93.25
111.71
118.56
118.89
128.08
128.44
129.60
131.06
132.70
132.77
146.16
162.41
ppm
20
40
60
80
100
120
140
160
180
200
CN
CN
O
HO
5a


Current Data Parameters
NAME Et-A-p-Br
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120220
Time 11.36
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 1
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 71.8
DW 69.000 usec
DE 6.50 usec
TE 296.5 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300136 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
3.74
1.93
1.13
1.03
1.00
2.16
7.19
2.39
Br
CN
O
HO
5b


Current Data Parameters
NAME Et-A-p-Br
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120220
Time_ 15.13
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 318
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 4096
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127782 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
11.64
18.97
40.39
42.56
76.68
77.00
77.31
85.76
95.04
119.70
121.42
128.03
128.35
130.91
131.62
131.99
132.83
137.04
163.88
ppm
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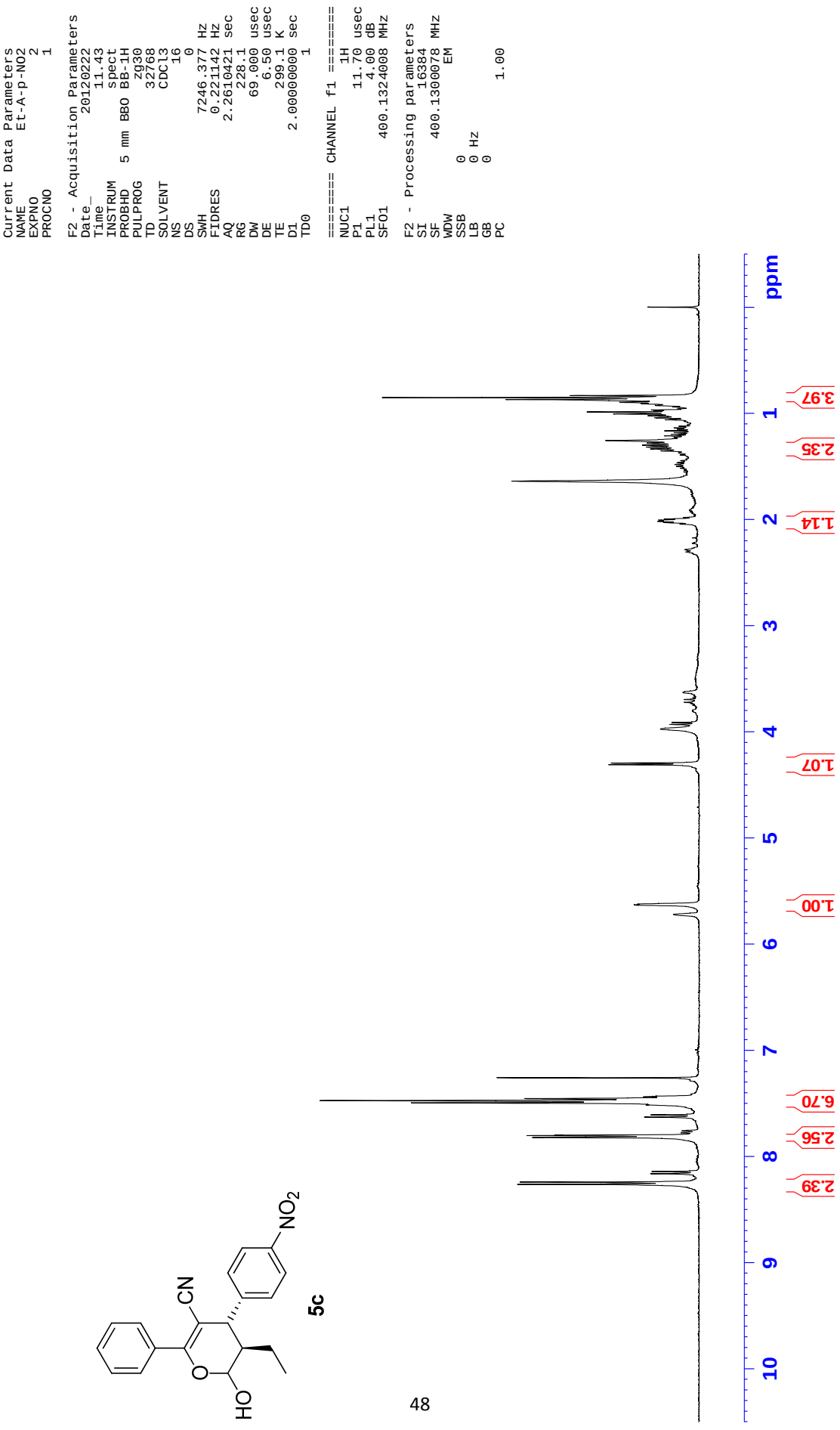
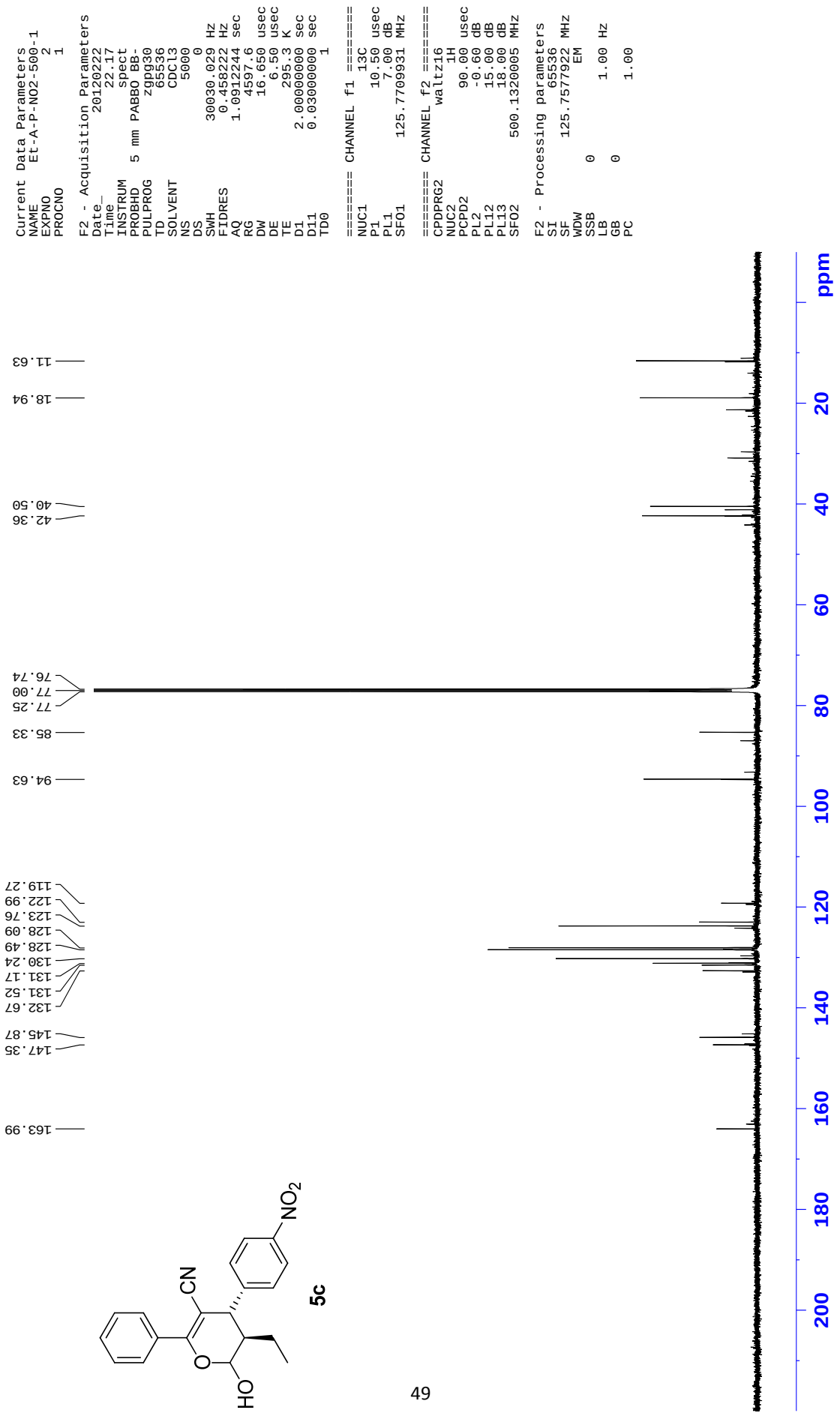
40
60
80
100
120
140
160
180
200
CN
Br
HO
O
5b


Current Data Parameters
NAME Et-A-p-NO2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120222
Time 11.43
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 228.1
DW 69.000 usec
DE 6.50 usec
TE 299.1 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
3.97
2.35
1.14
1.07
1.00
6.70
2.56
2.39
CN
NO2
HO
O
5c


Current Data Parameters
NAME Et-A-P-NO2-500-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120222
Time_ 22.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5000
DS 0
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 4597.6
DW 16.650 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 125.7709931 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 65536
SF 125.7577922 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
11.63
18.94
40.50
42.36
76.74
77.00
77.25
85.33
94.63
119.27
122.99
123.76
128.09
128.49
130.24
131.17
131.52
132.67
145.87
147.35
163.99
ppm
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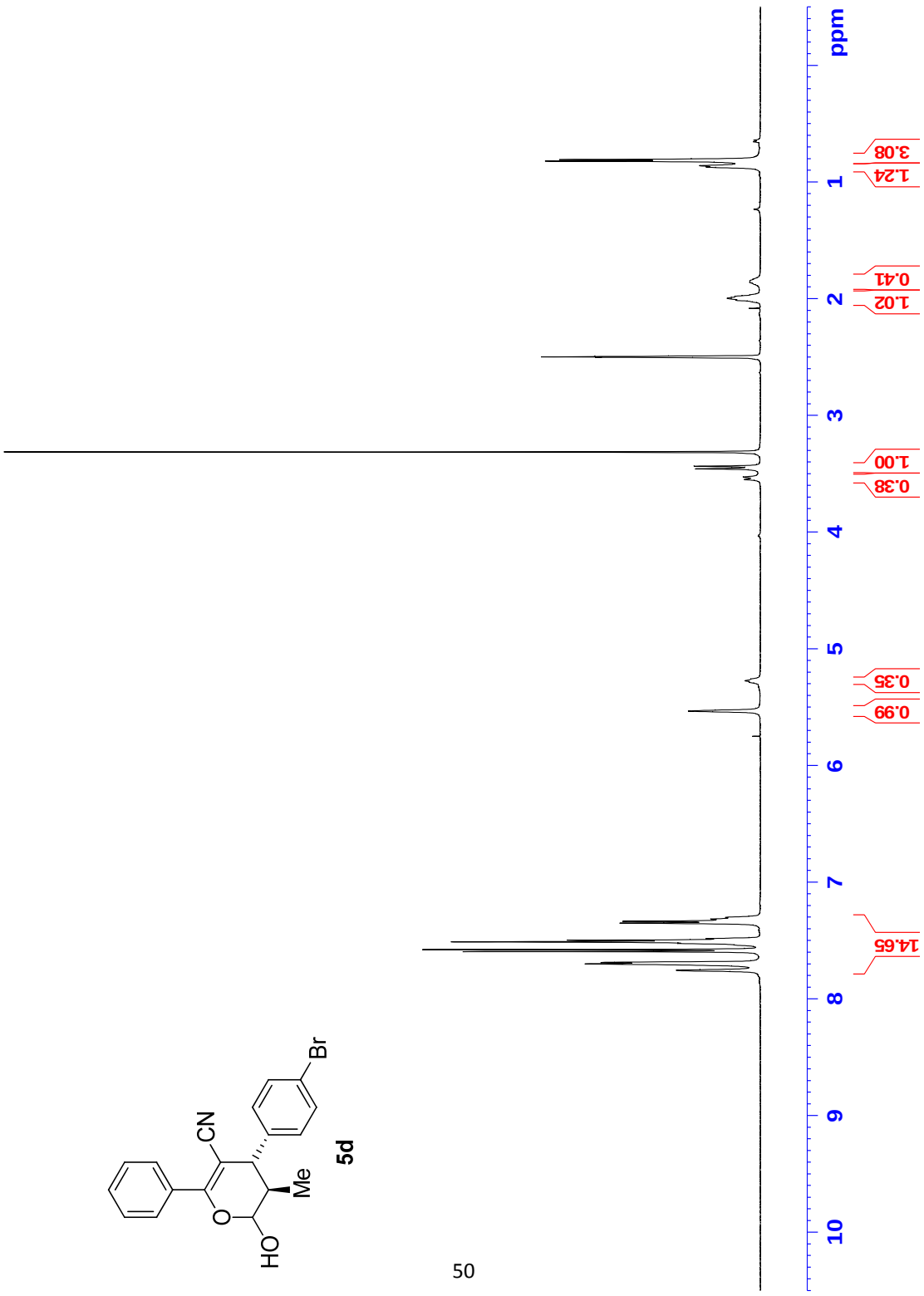
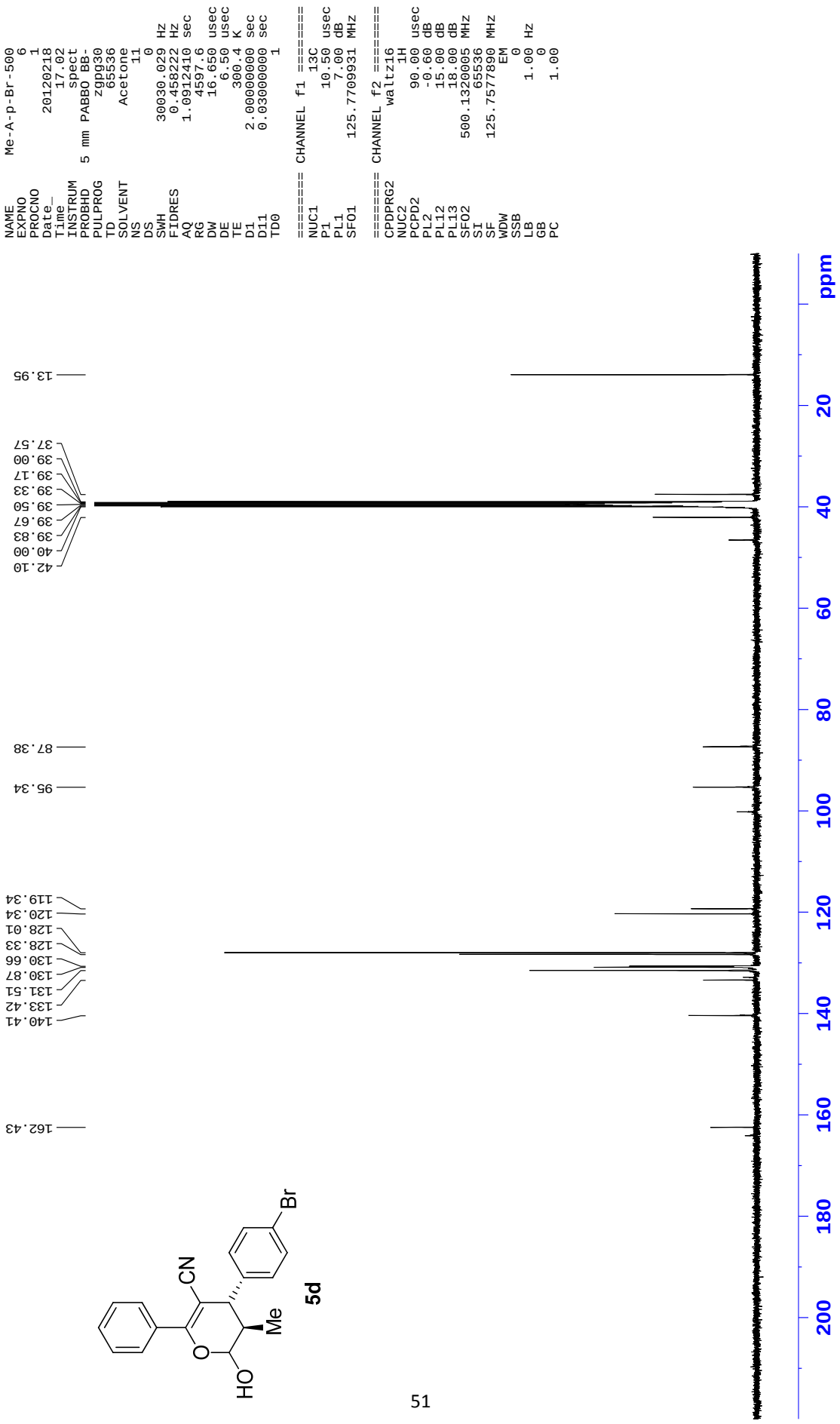
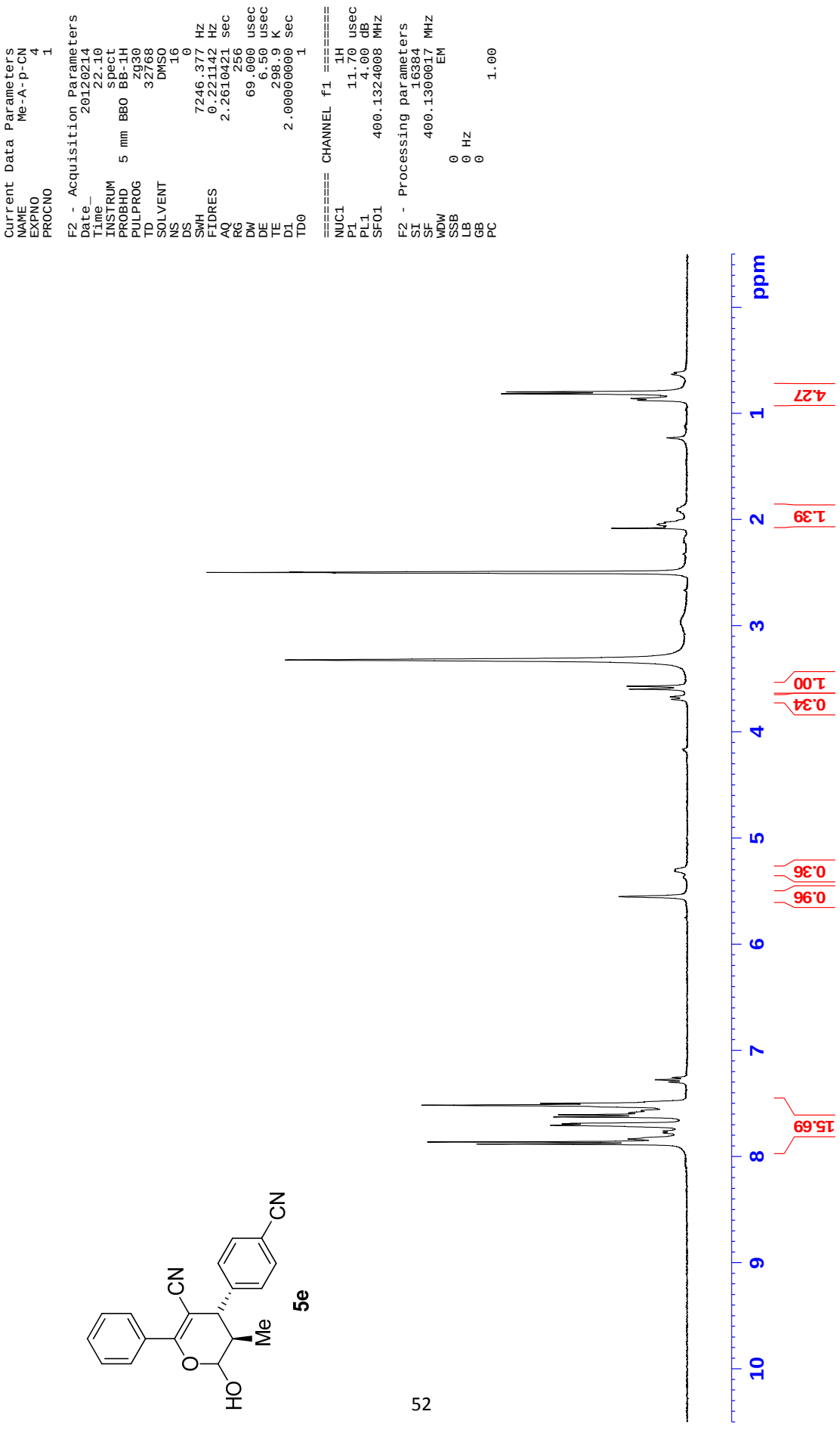
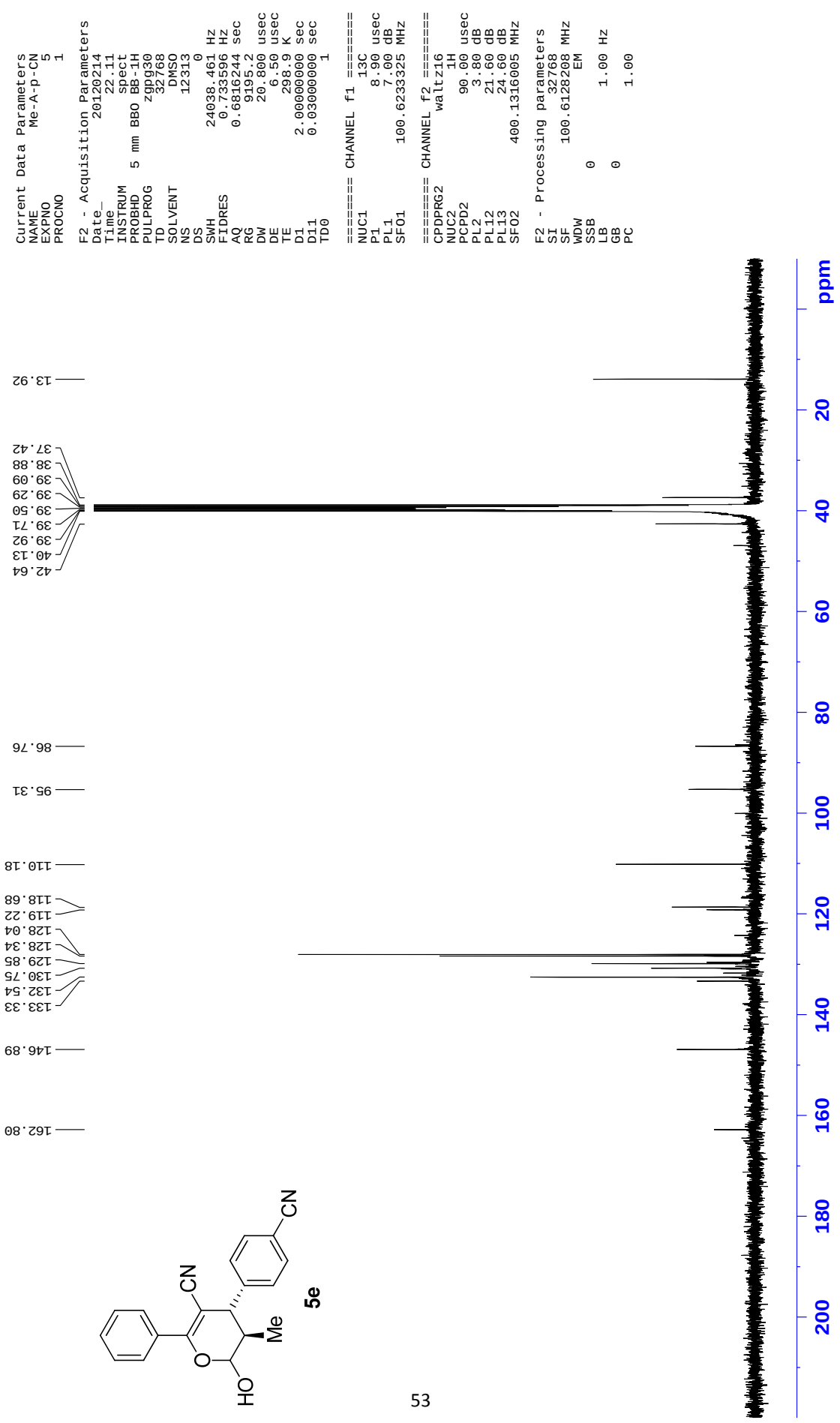
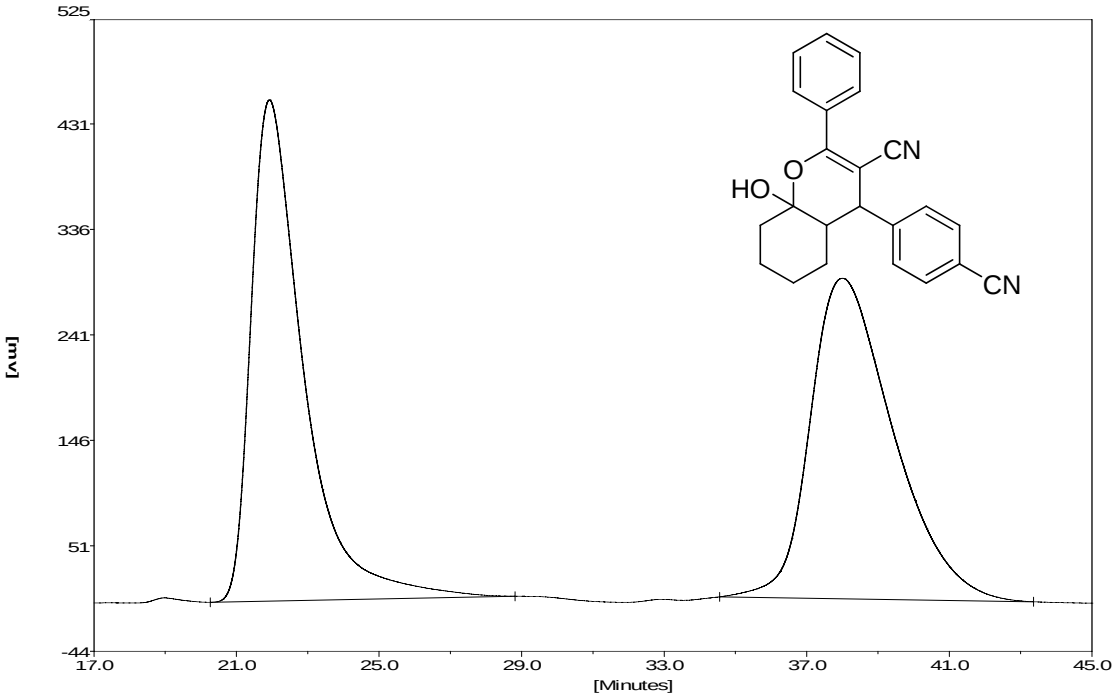
40
60
80
100
120
140
160
180
200
CN
NO2
HO
O
5c


Current Data Parameters
NAME Me-A-p-Br-500
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120218
Time_ 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT Acetone
NS 16
DS 0
SWH 9057.971 Hz
FIDRES 0.276427 Hz
AQ 1.8088436 sec
RG 161.3
DW 55.200 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 0 dB
SFO1 500.1330008 MHz
F2 - Processing parameters
SI 16384
SF 500.1297793 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
3.08
1.24
0.41
1.02
0.38
1.00
0.35
0.99
14.65
CN
Br
Me
HO
O
5d

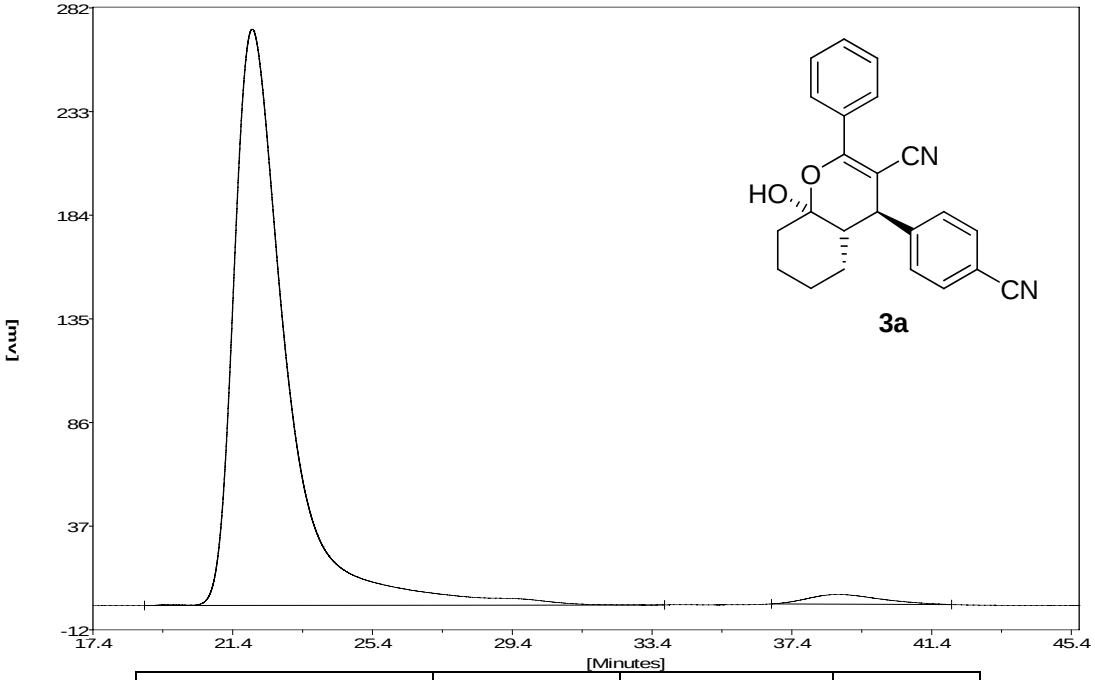

NAME Me-A-p-Br-500
EXPNO 6
PROCNO 1
Date_ 20120218
Time 17.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 11
DS 0
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 4597.6
DW 16.650 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 7.00 dB
SFO1 125.7709931 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -0.60 dB
PL12 15.00 dB
PL13 18.00 dB
SFO2 500.1320005 MHz
SI 65536
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
13.95
37.57
39.00
39.17
39.33
39.50
39.67
39.83
40.00
42.10
87.38
95.34
119.34
120.34
128.01
128.33
130.66
130.87
131.51
133.42
140.41
162.43
ppm
20
40
60
80
100
120
140
160
180
200
HO
O
Me
CN
Br
5d


Current Data Parameters
NAME Me-A-p-CN
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120214
Time_ 22.10
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 0
SWH 7246.377 Hz
FIDRES 0.221142 Hz
AQ 2.2610421 sec
RG 256
DW 69.000 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 11.70 usec
PL1 4.00 dB
SFO1 400.1324008 MHz
F2 - Processing parameters
SI 16384
SF 400.1300017 MHz
WDW EM
SSB 0
LB 0 Hz
GB 0
PC 1.00
ppm
1
2
3
4
5
6
7
8
9
10
4.27
1.39
1.00
0.34
0.36
0.96
15.69
CN
CN
Me
HO
O
5e


Current Data Parameters
NAME Me-A-p-CN
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120214
Time_ 22.11
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 12313
DS 0
SWH 24038.461 Hz
FIDRES 0.733596 Hz
AQ 0.6816244 sec
RG 9195.2
DW 20.800 usec
DE 6.50 usec
TE 298.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.90 usec
PL1 7.00 dB
SFO1 100.6233325 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.80 dB
PL12 21.60 dB
PL13 24.60 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6128208 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
13.92
37.42
38.88
39.09
39.29
39.50
39.71
39.92
40.13
42.64
86.76
95.31
110.18
118.68
119.22
128.04
128.34
129.85
130.75
132.54
133.33
146.89
162.80
ppm
20
40
60
80
100
120
140
160
180
200
CN
CN
Me
HO
O
5e

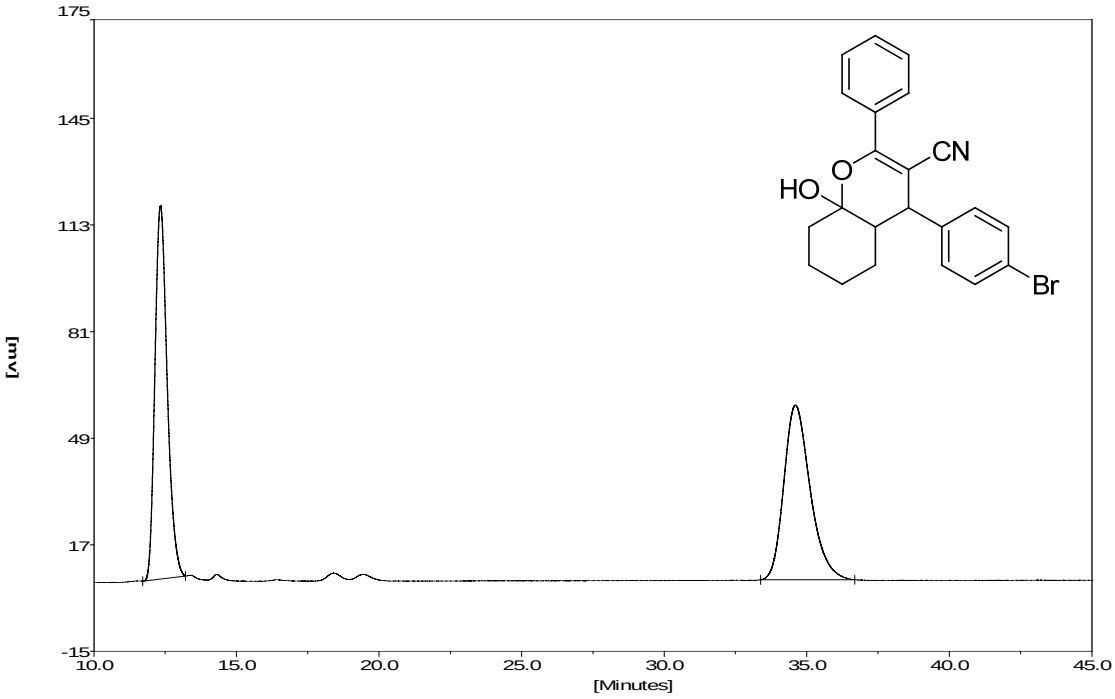


Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
21.92	451.46	46203.30	49.7420
38.00	288.70	46682.60	50.2580

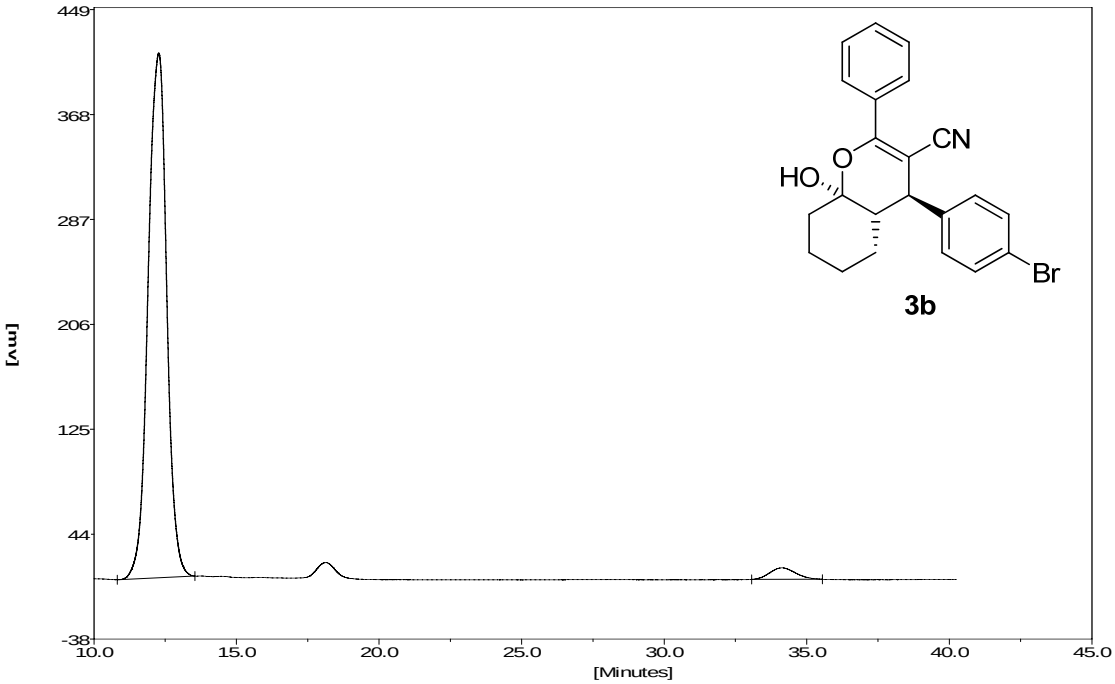


3a

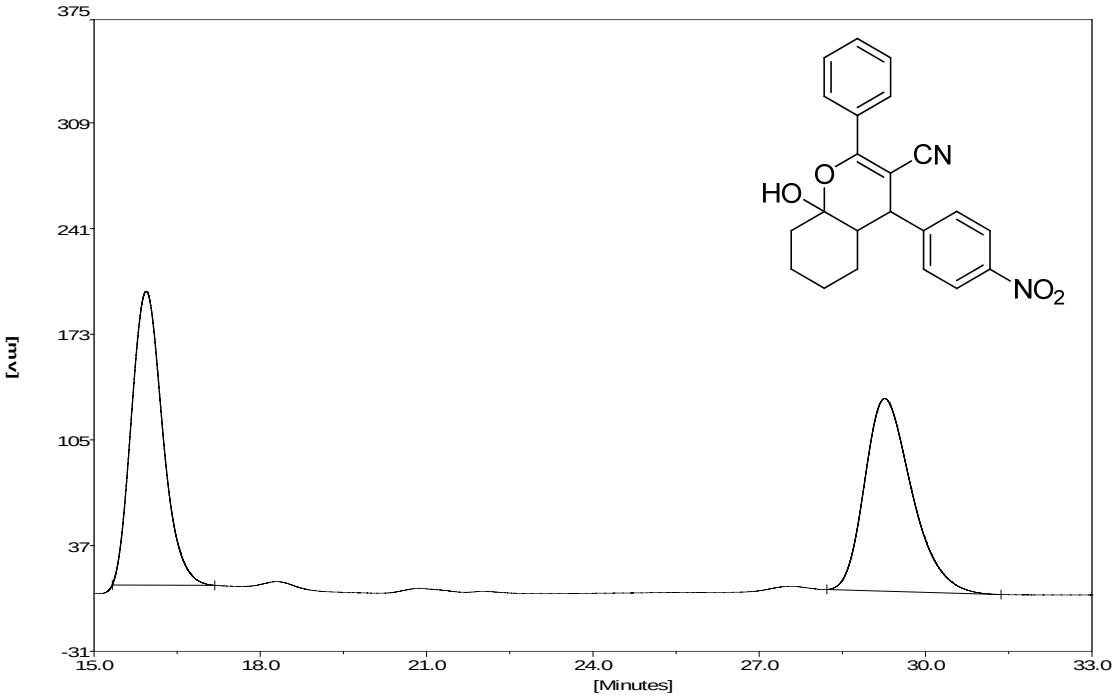
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
21.96	272.42	28482.83	97.7495
38.71	4.56	656.96	2.2545



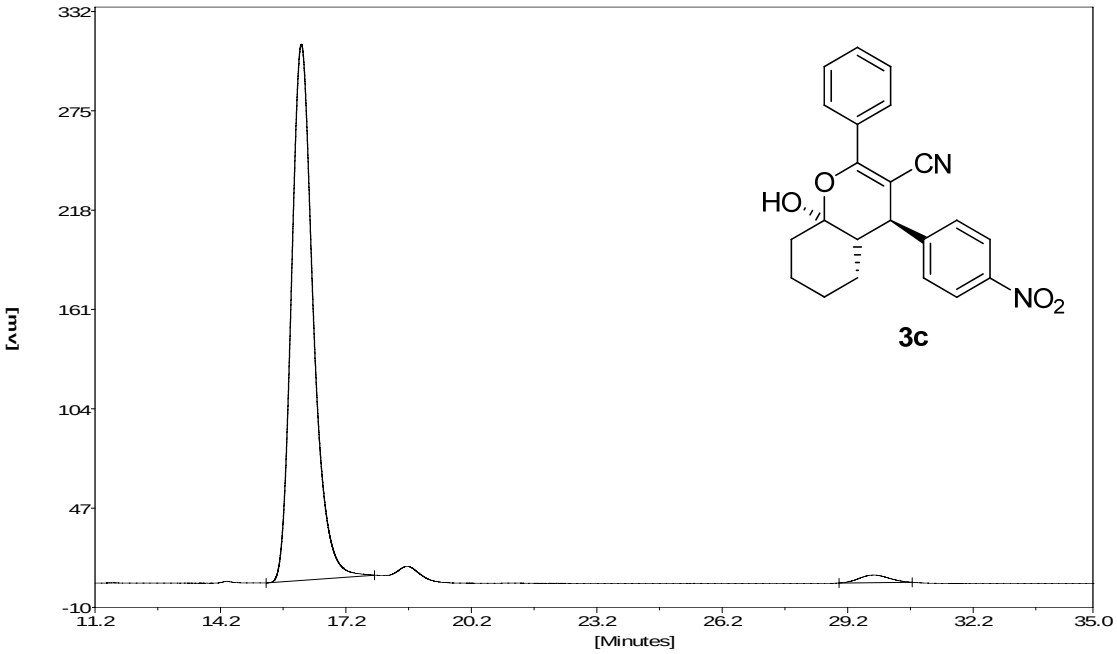
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
12.33	112.11	3358.02	49.9158
34.60	52.46	3369.35	50.0842



Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
12.28	405.12	18368.83	97.0338
34.13	8.87	561.51	2.9662

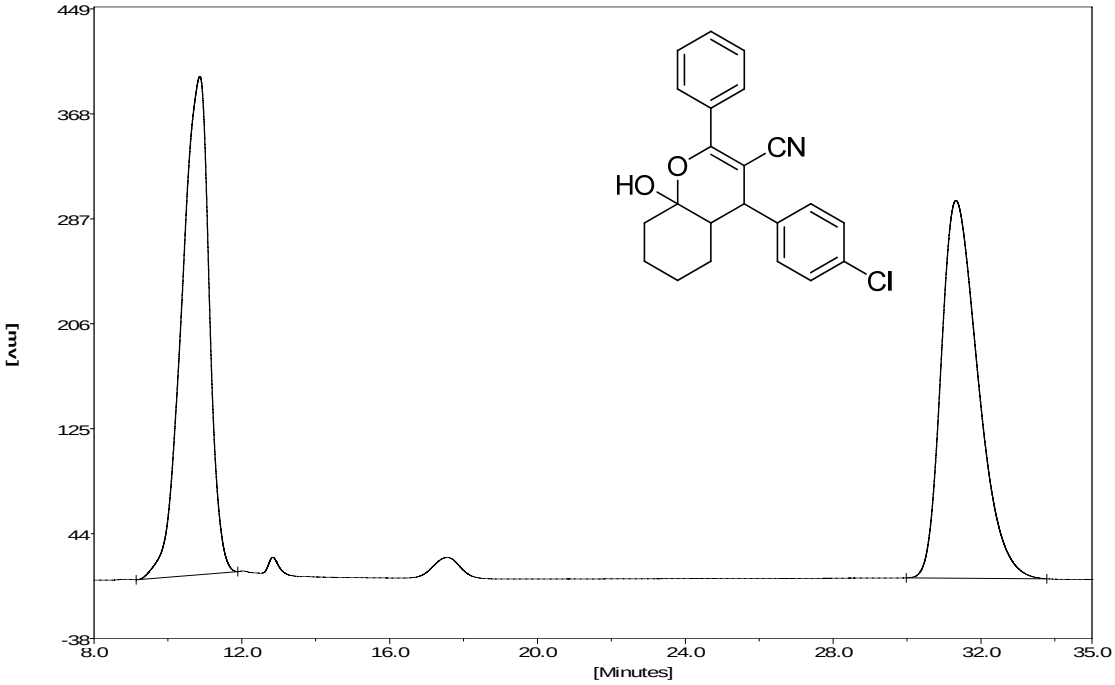


Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
15.94	189.06	7361.95	50.1724
29.26	123.92	7311.36	49.8276

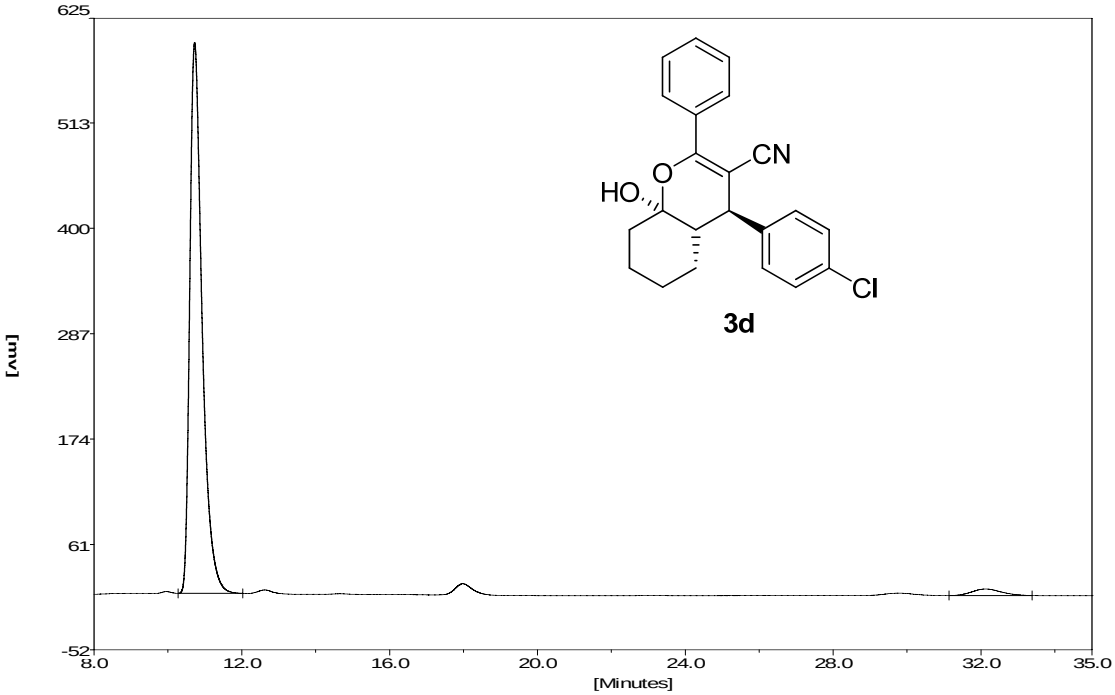


3c

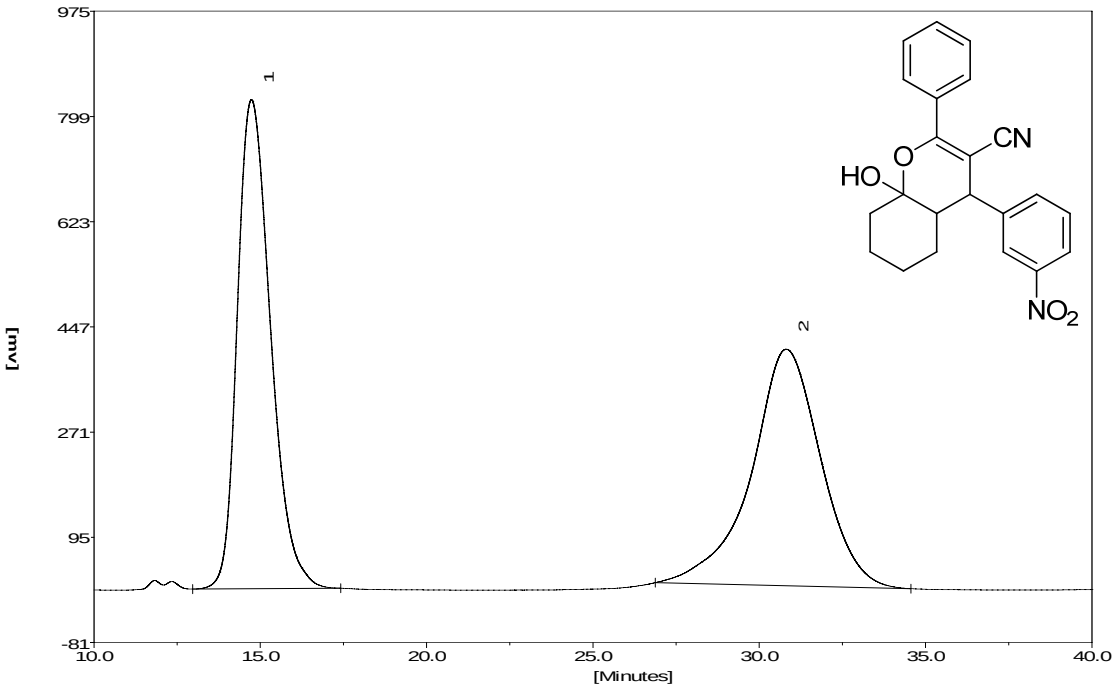
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
16.11	307.73	11133.64	98.0697
29.28	4.40	219.14	1.9303



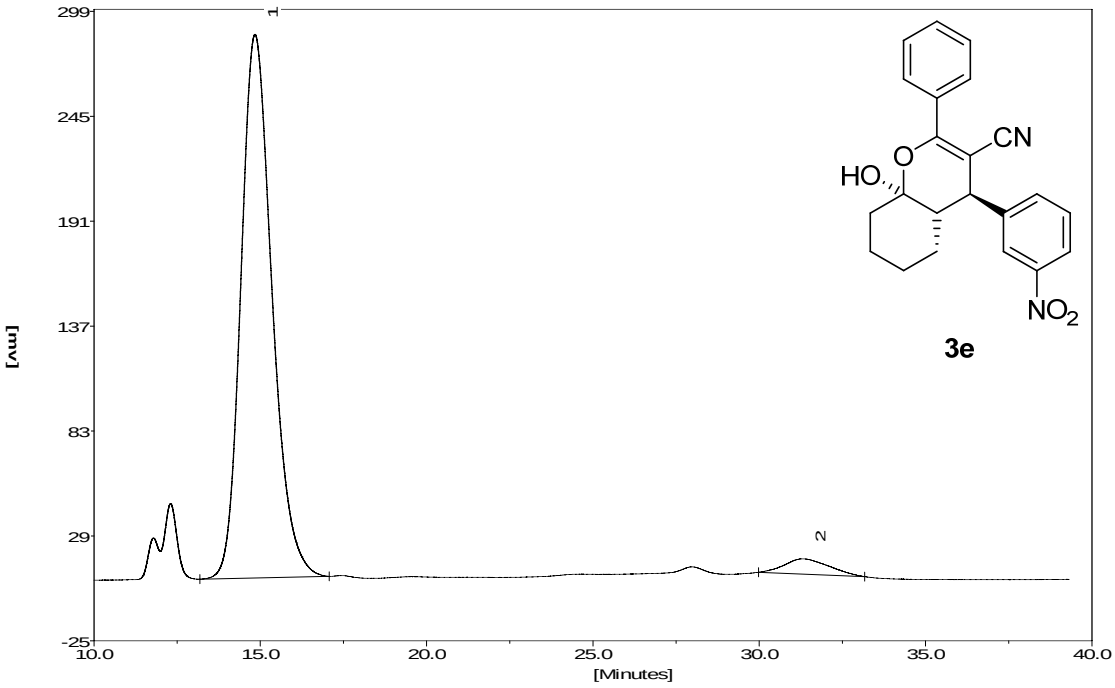
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
10.87	384.57	19834.13	49.5166
31.32	291.62	20221.43	50.4834



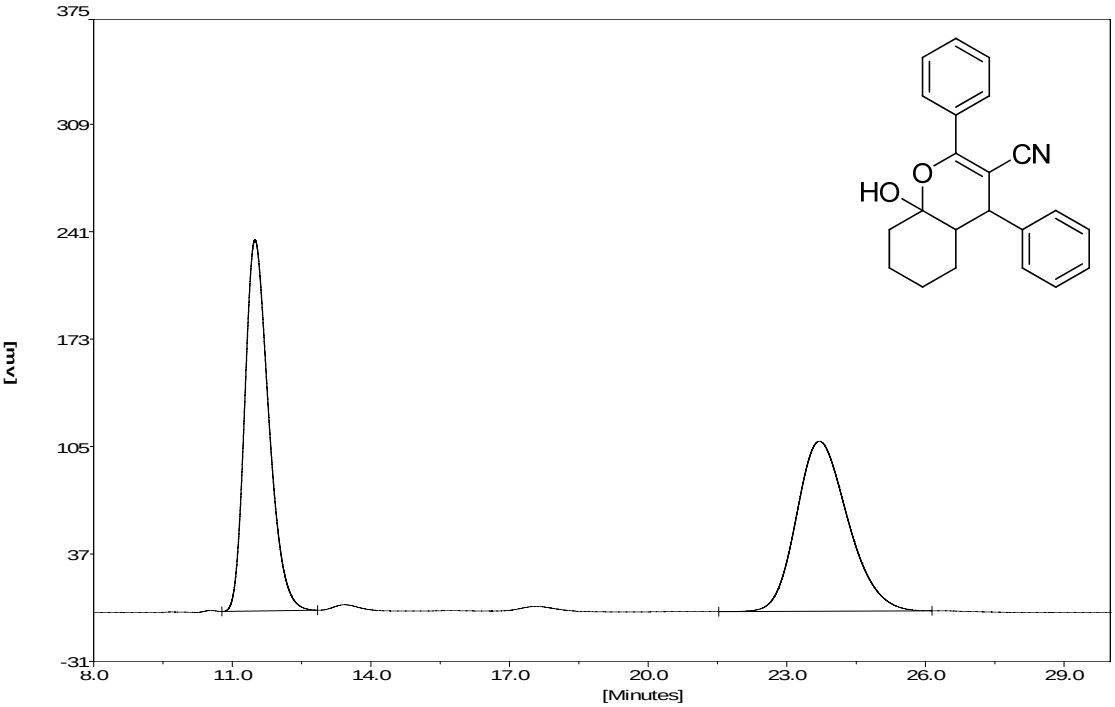
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
10.73	590.40	13670.84	97.2590
32.14	6.98	385.28	2.7410



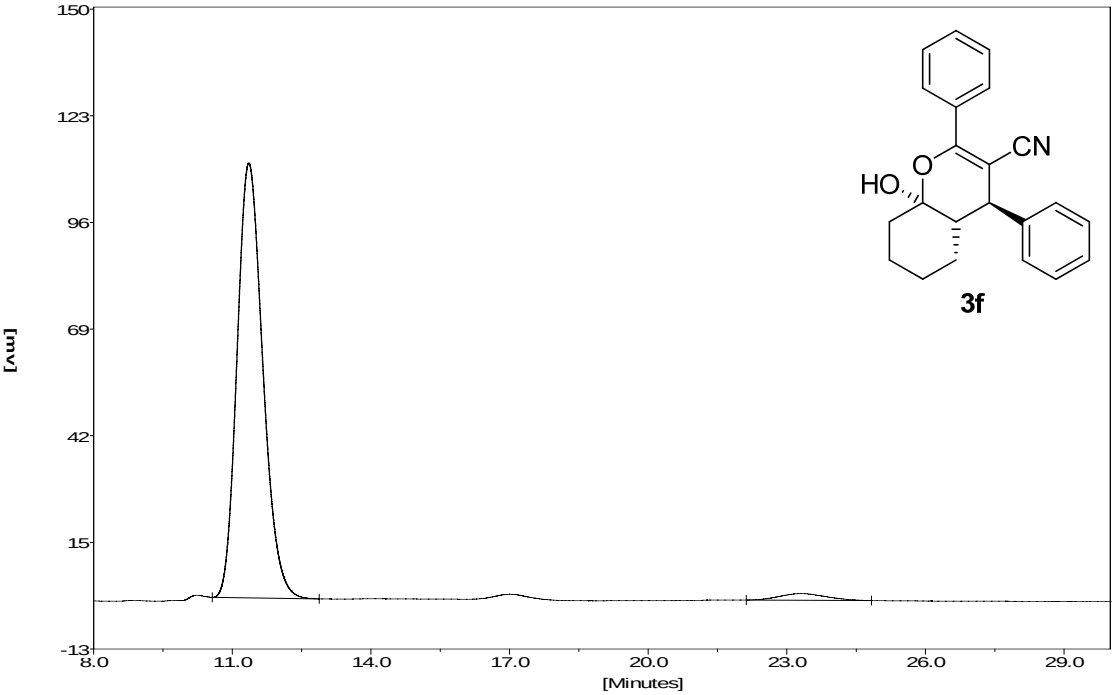
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
14.74	818.12	56825.68	49.7449
30.81	395.74	57408.46	50.2551



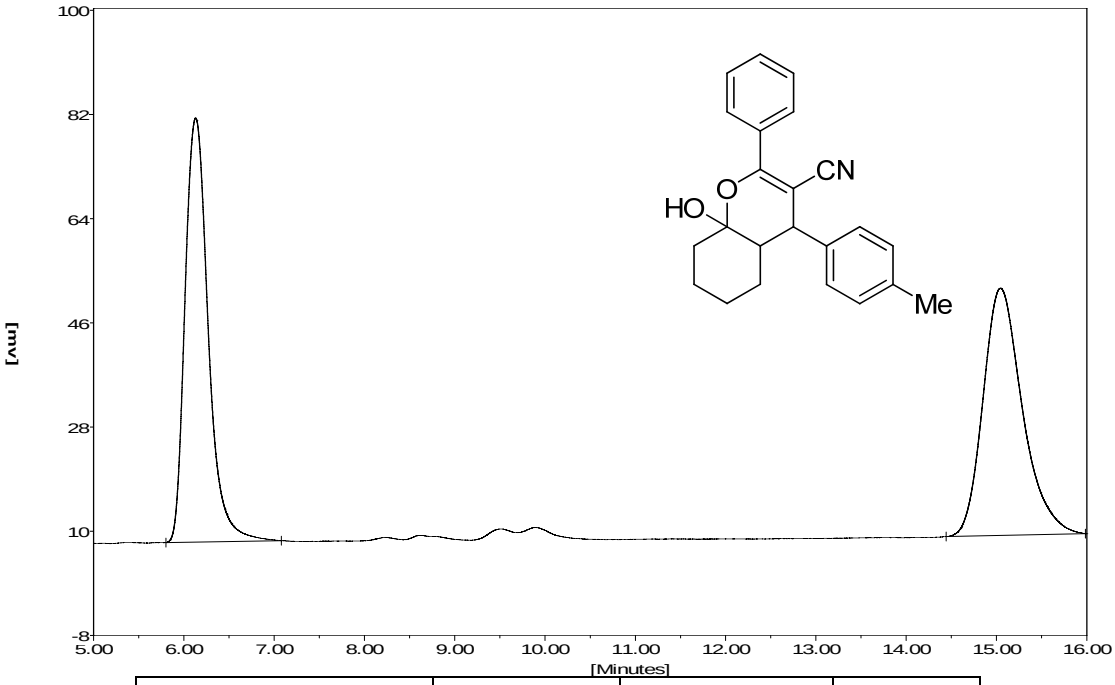
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
14.84	279.79	18139.53	96.1212
31.32	7.87	731.99	3.8788



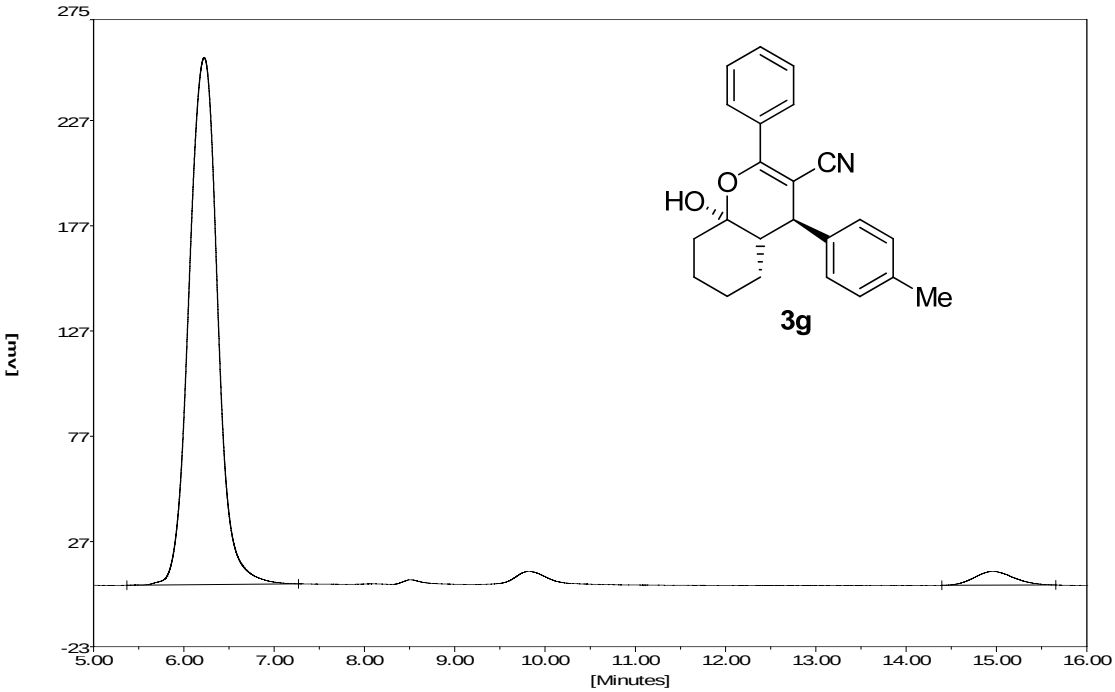
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
11.49	234.91	8436.59	50.3084
23.71	107.61	8333.16	49.6916



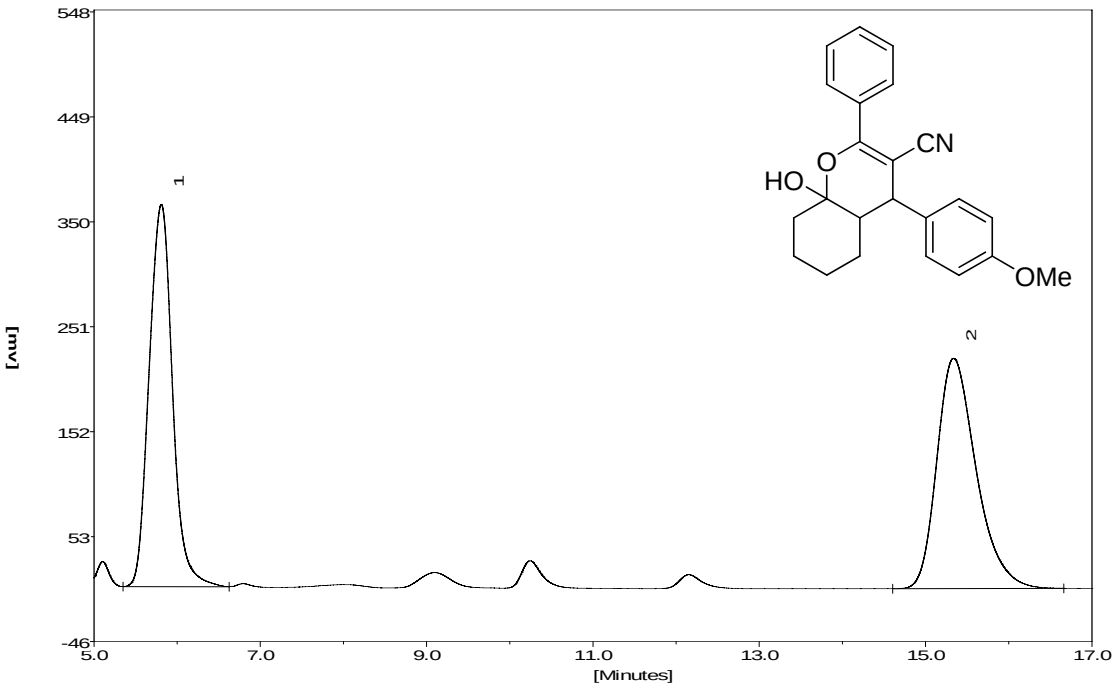
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
11.36	110.10	4263.38	97.3006
23.33	1.69	118.28	2.6994



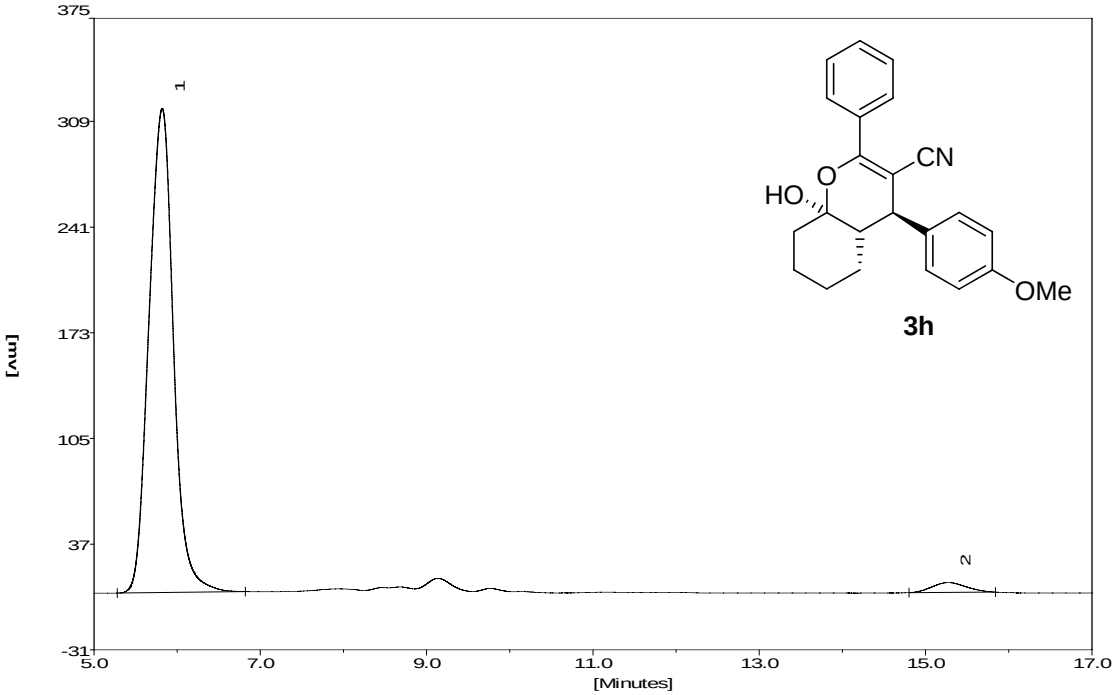
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
6.13	73.27	1304.67	50.2921
15.05	42.72	1289.51	49.7079



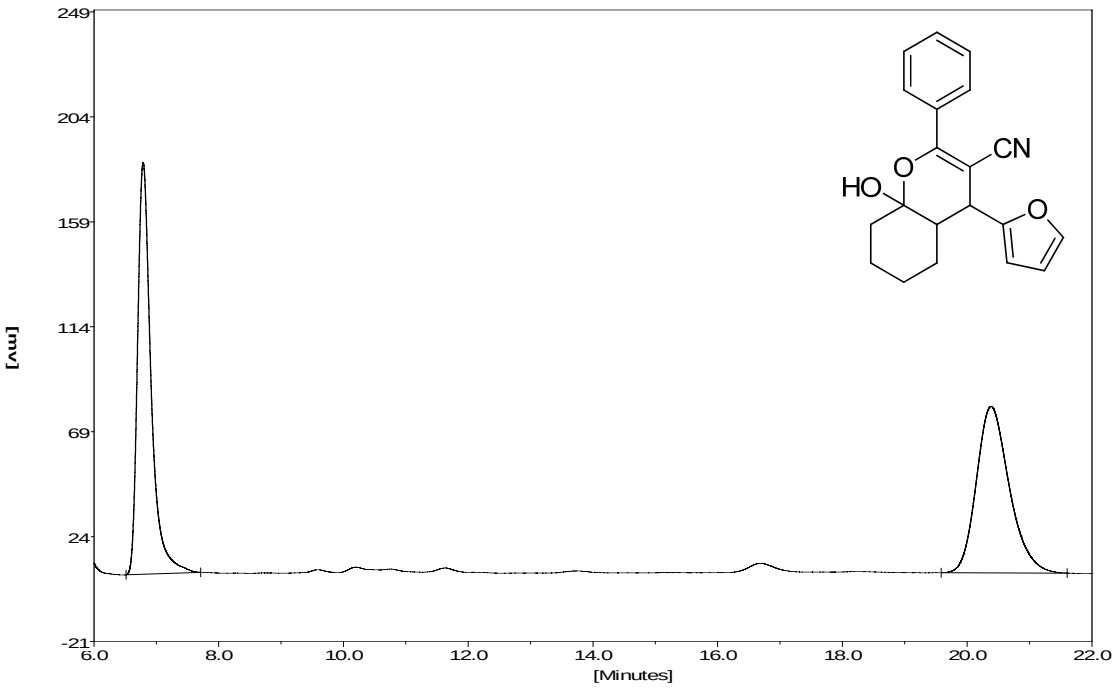
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
6.23	250.36	5527.57	96.5546
14.96	6.56	197.24	3.4454



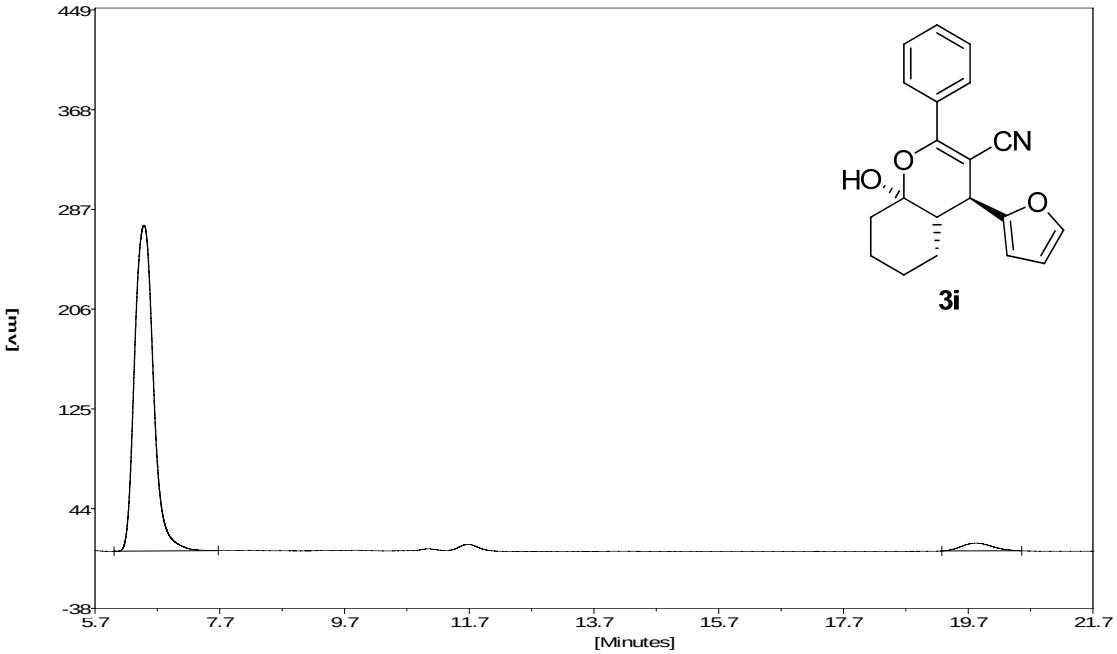
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
5.81	360.76	7028.28	49.6218
15.34	217.34	7135.41	50.3782



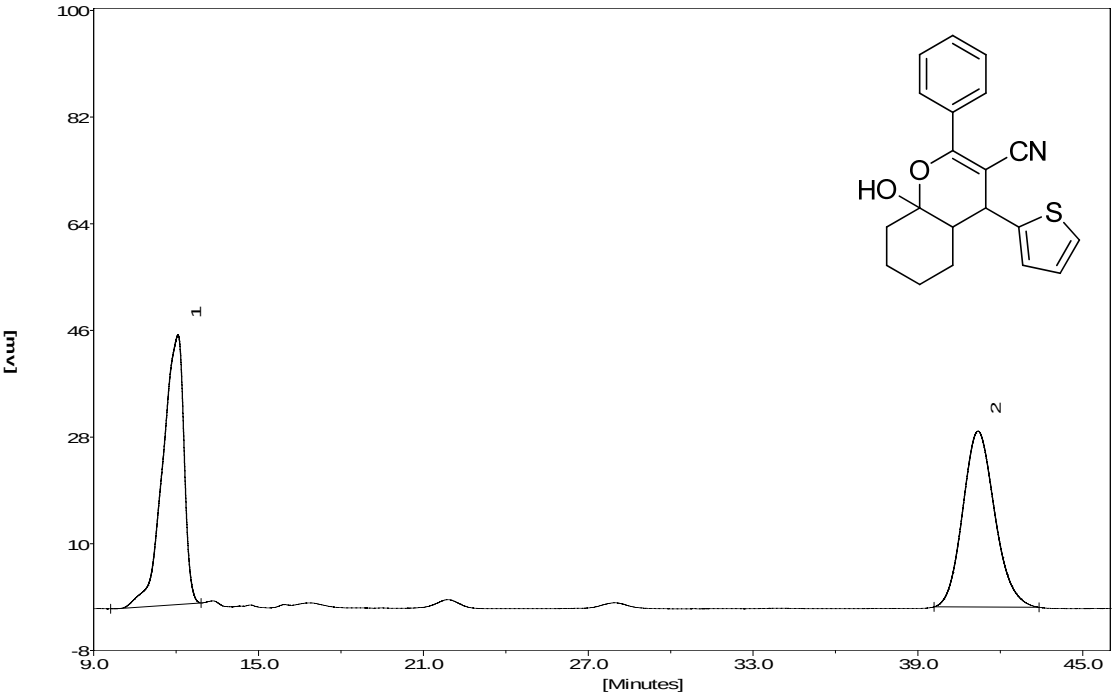
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
5.82	311.52	6348.44	97.2430
15.27	6.32	179.99	2.7570



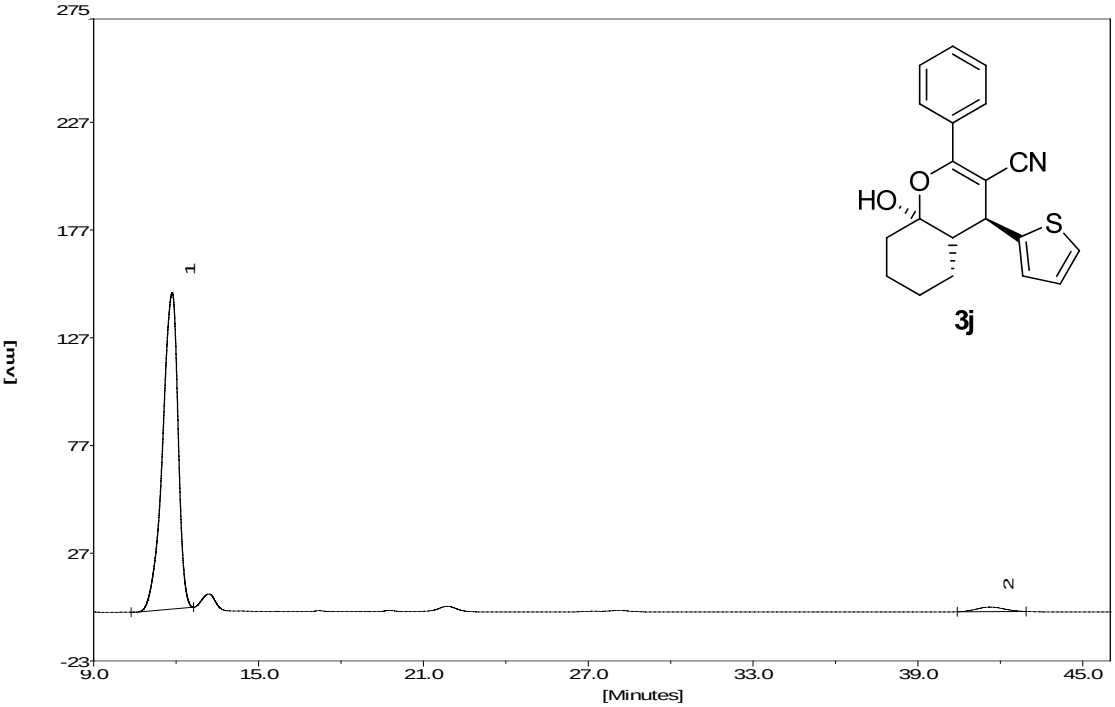
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
6.79	176.56	2649.64	50.3644
20.38	71.43	2611.30	49.6356



Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
6.45	264.69	5555.67	96.2470
19.78	6.29	216.63	3.7530

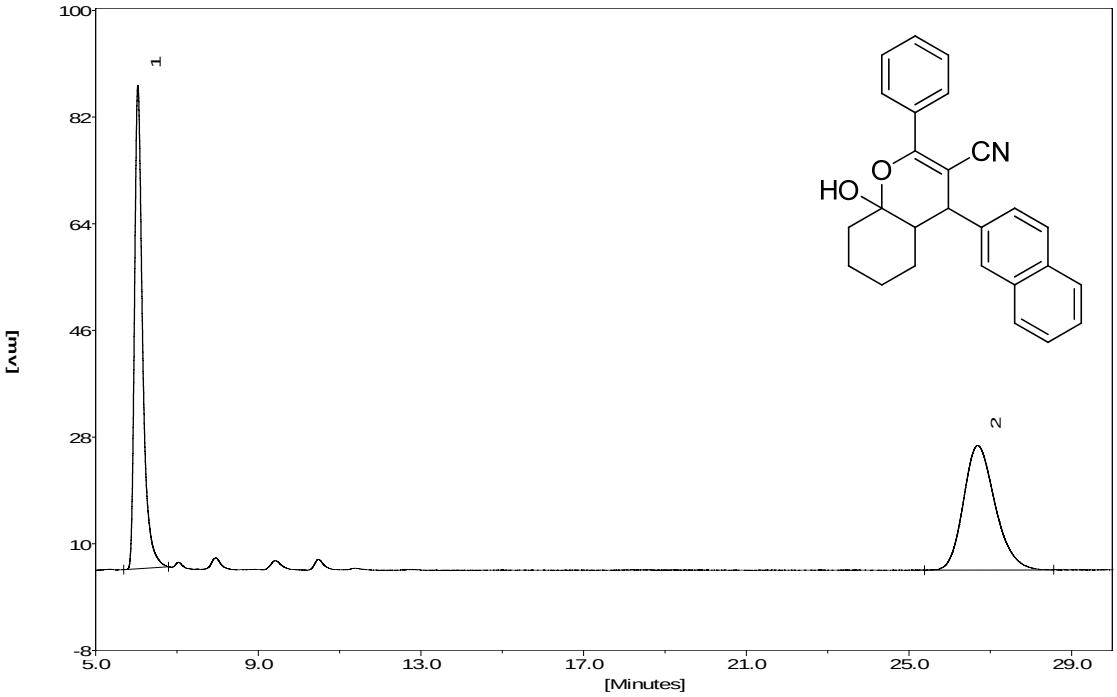


Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
12.07	45.47	2438.58	50.3891
41.18	29.70	2400.91	49.6109

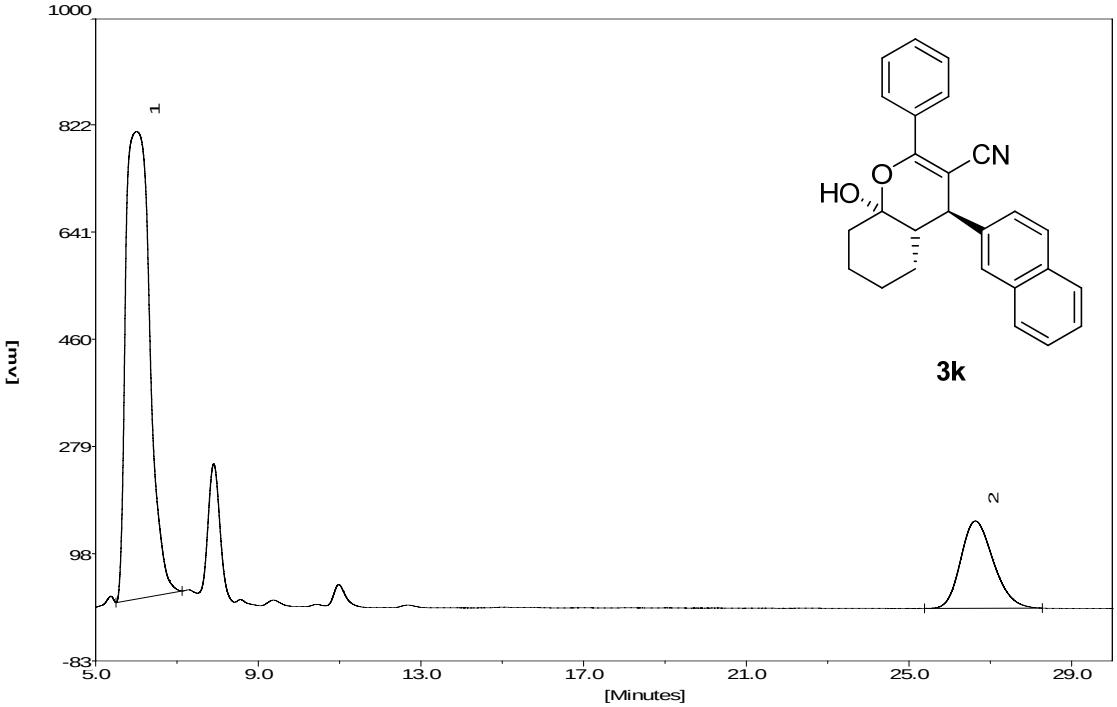


3j

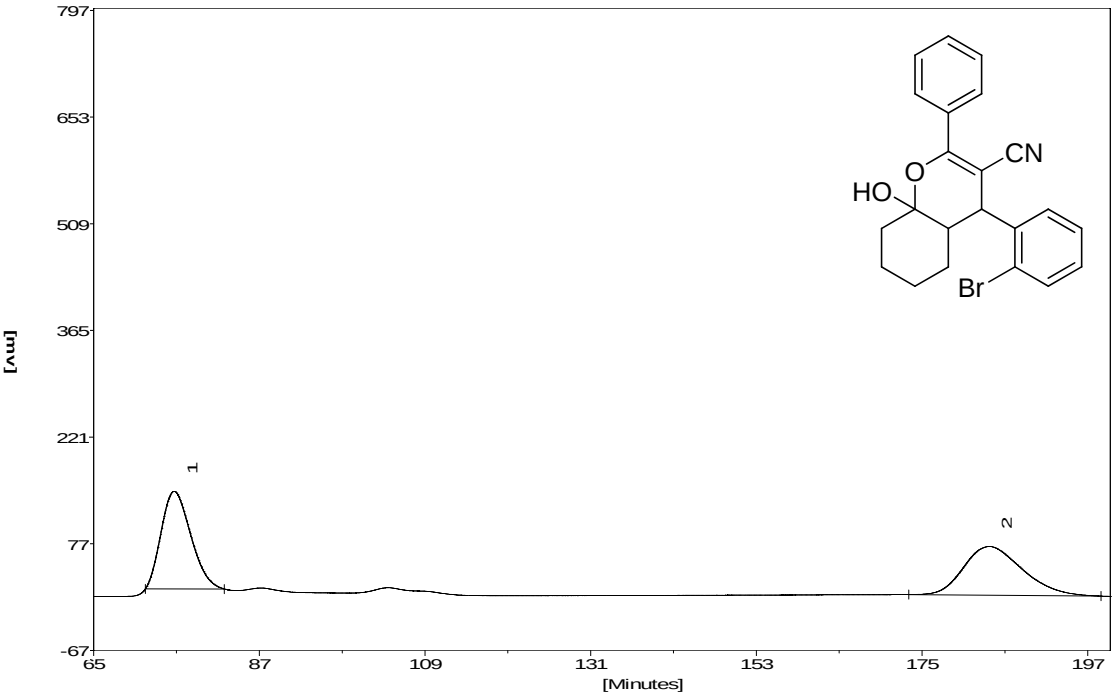
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
11.86	146.92	5769.78	97.4911
41.63	2.12	148.48	2.5089



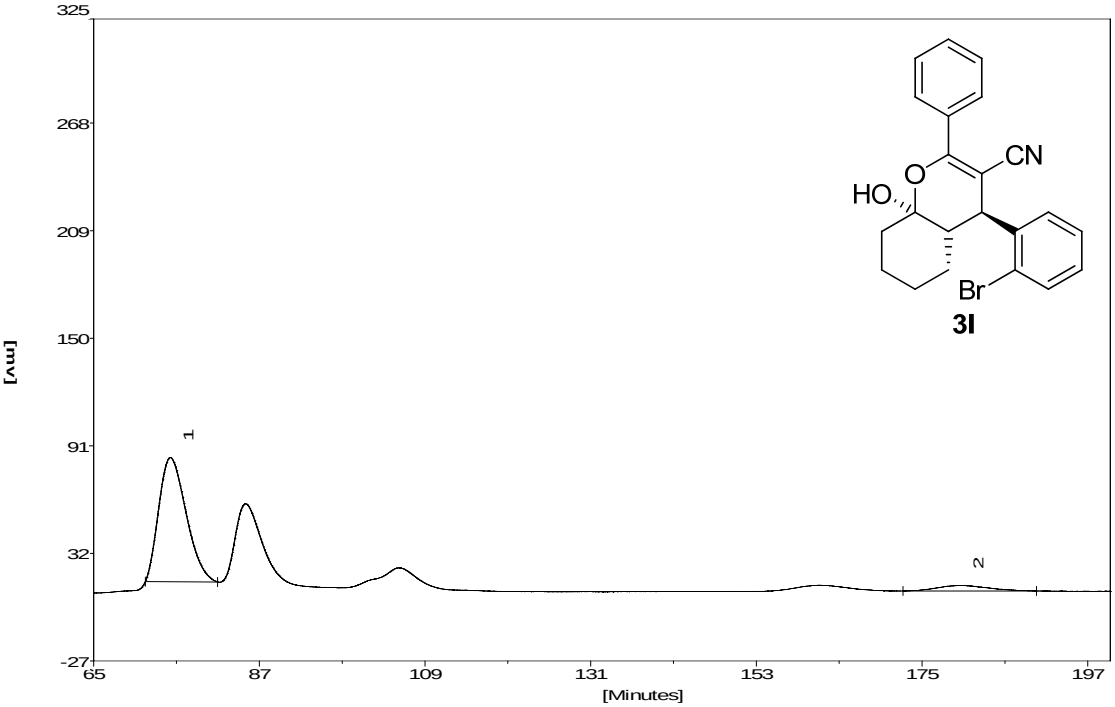
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
6.04	81.60	1130.17	49.8728
26.69	21.01	1135.94	50.1272



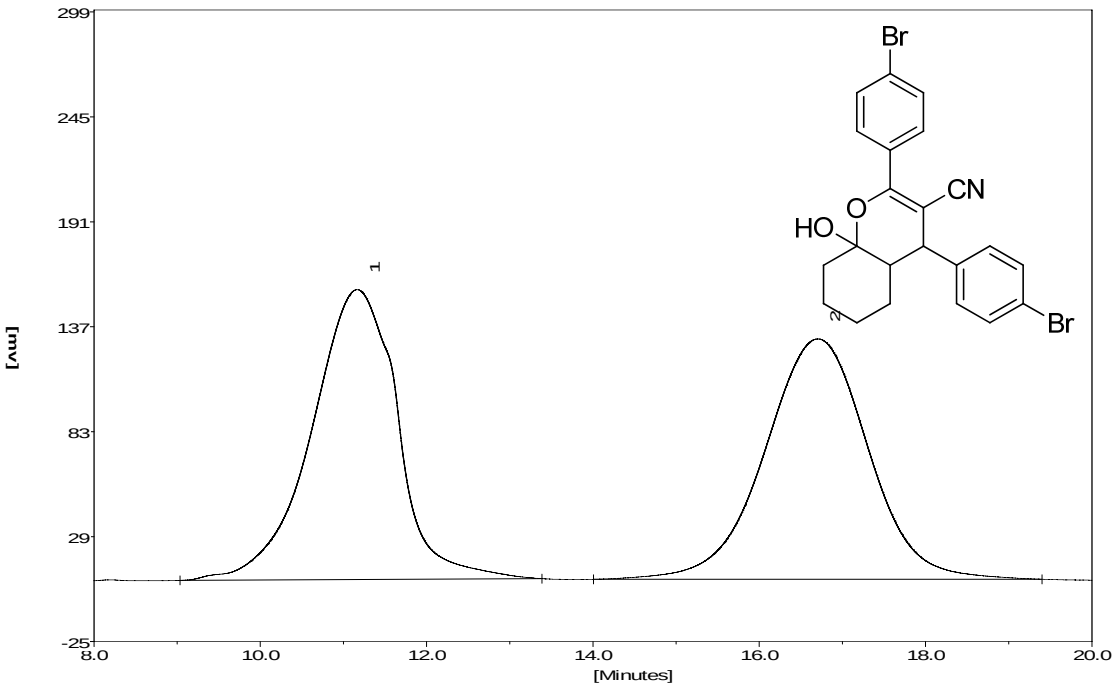
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
6.01	788.64	32349.40	79.7782
26.63	147.32	8199.79	20.2218



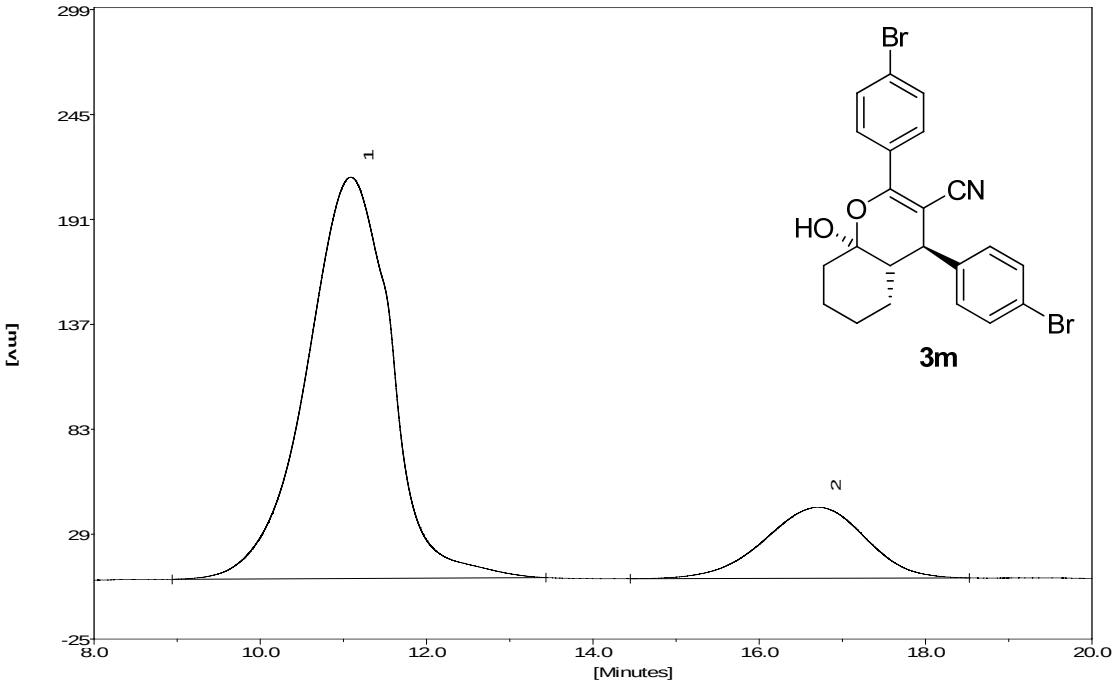
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
75.71	131.79	36027.70	50.3298
183.86	65.69	35555.60	49.6702



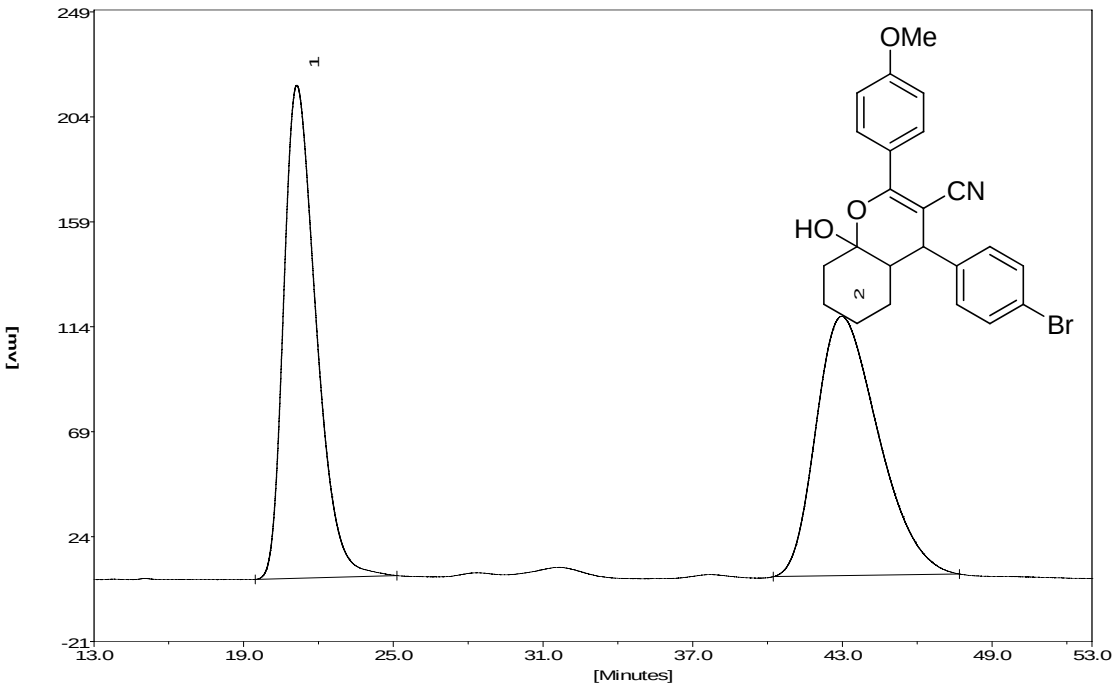
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
75.20	68.12	17222.94	92.4964
180.08	2.97	1397.19	7.5036



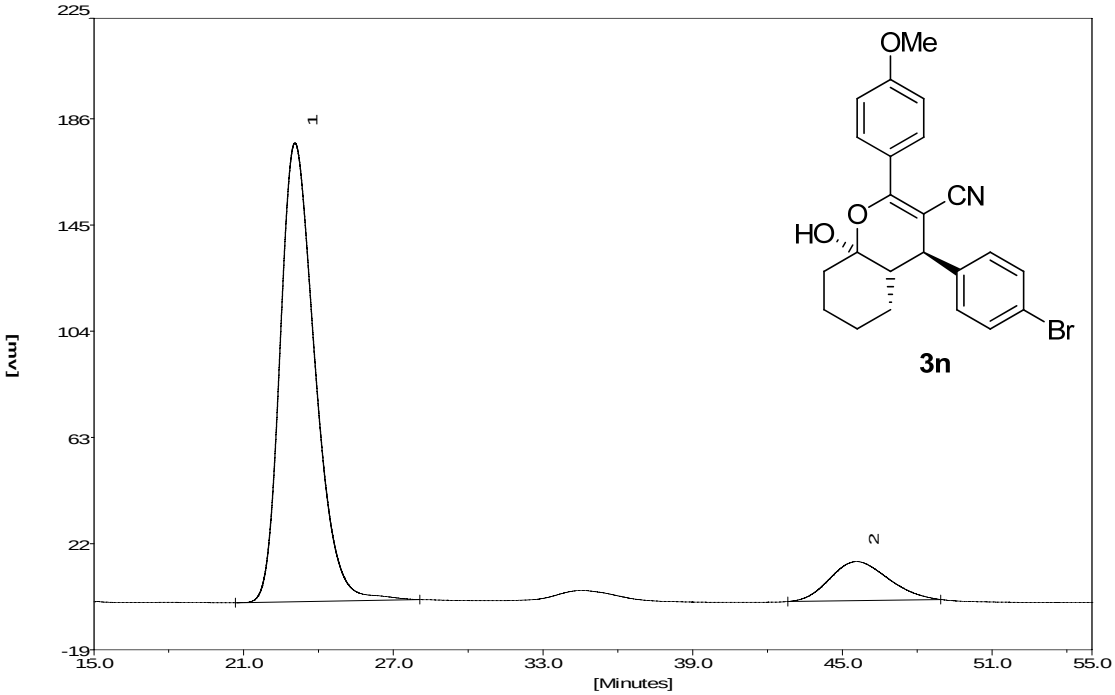
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
11.17	149.15	10713.74	50.0081
16.70	123.71	10710.28	49.9919



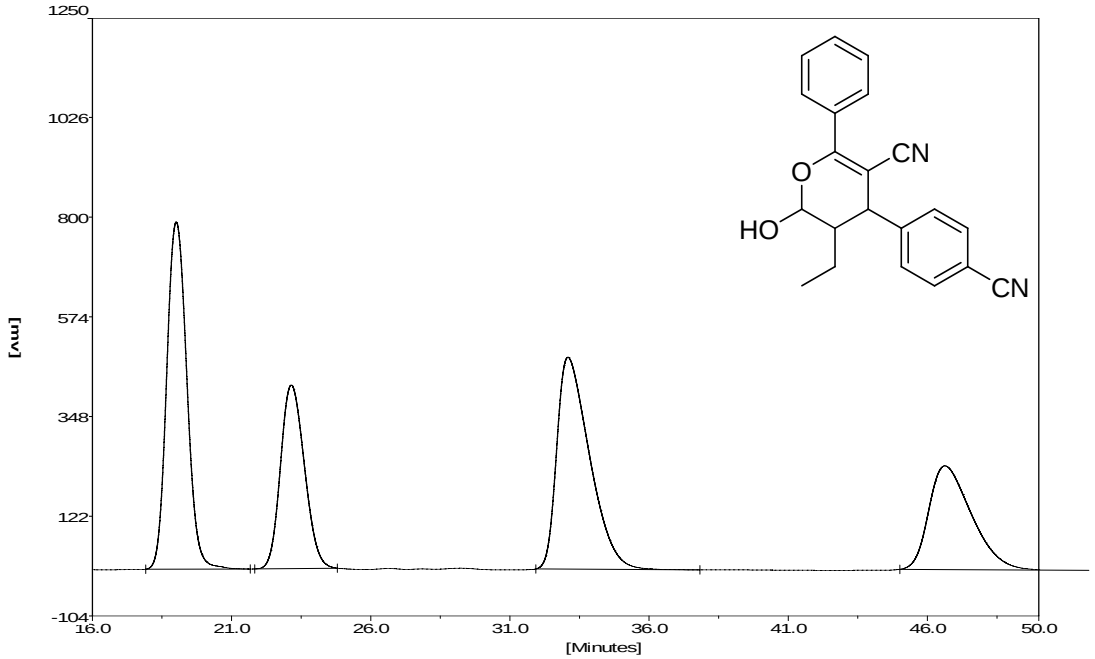
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
11.09	206.54	14601.79	82.5735
16.71	36.52	3081.60	17.4265



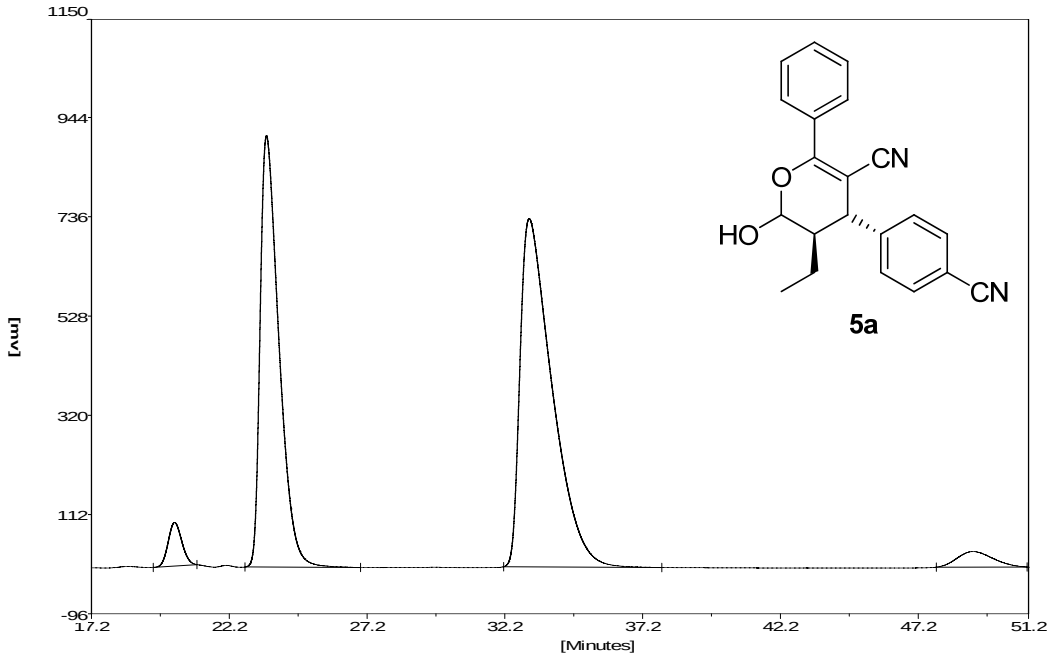
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
21.13	211.47	19059.06	49.6346
42.97	111.20	19339.72	50.3654



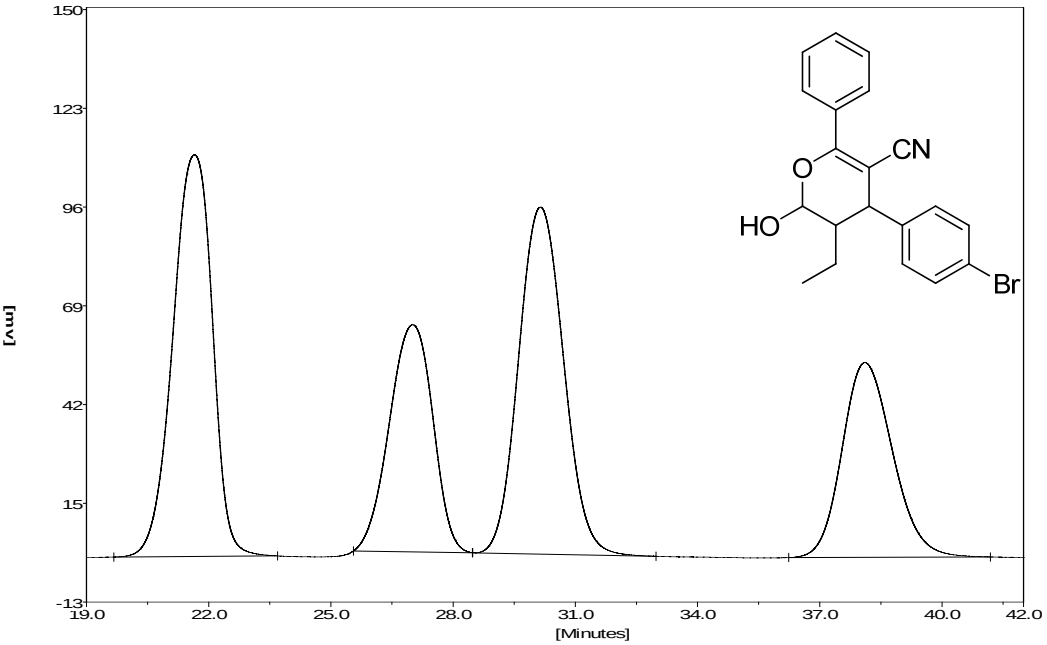
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
23.06	177.12	17102.36	87.4877
45.56	15.09	2445.93	12.5123



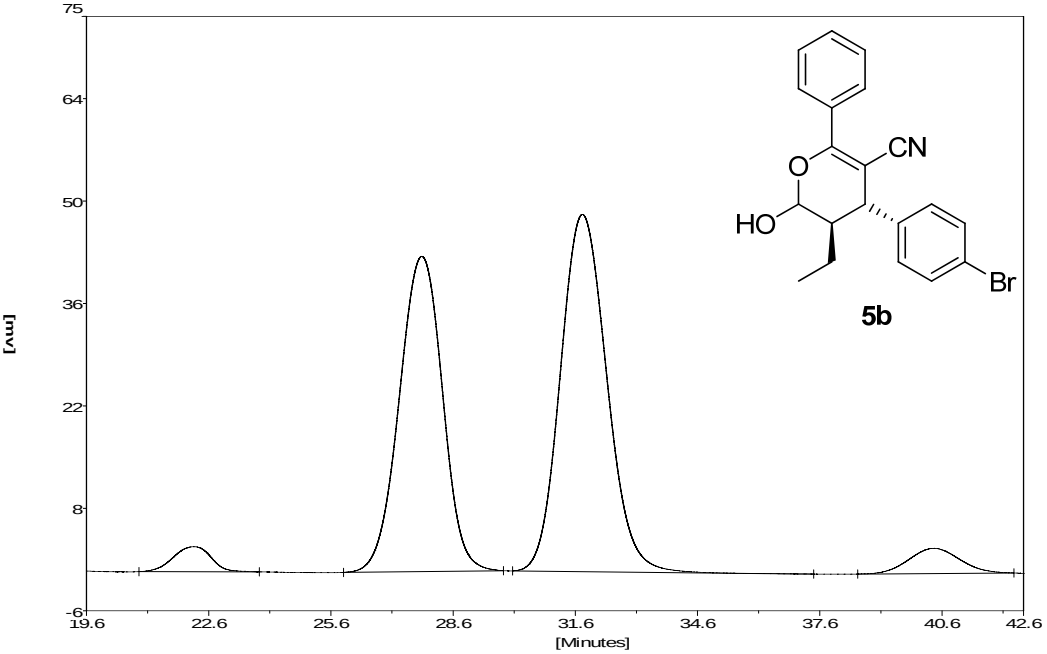
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
19.02	786.83	39500.15	30.7005
23.15	416.26	24864.74	19.3255
33.09	480.42	39292.28	30.5390
46.63	235.22	25005.67	19.4350



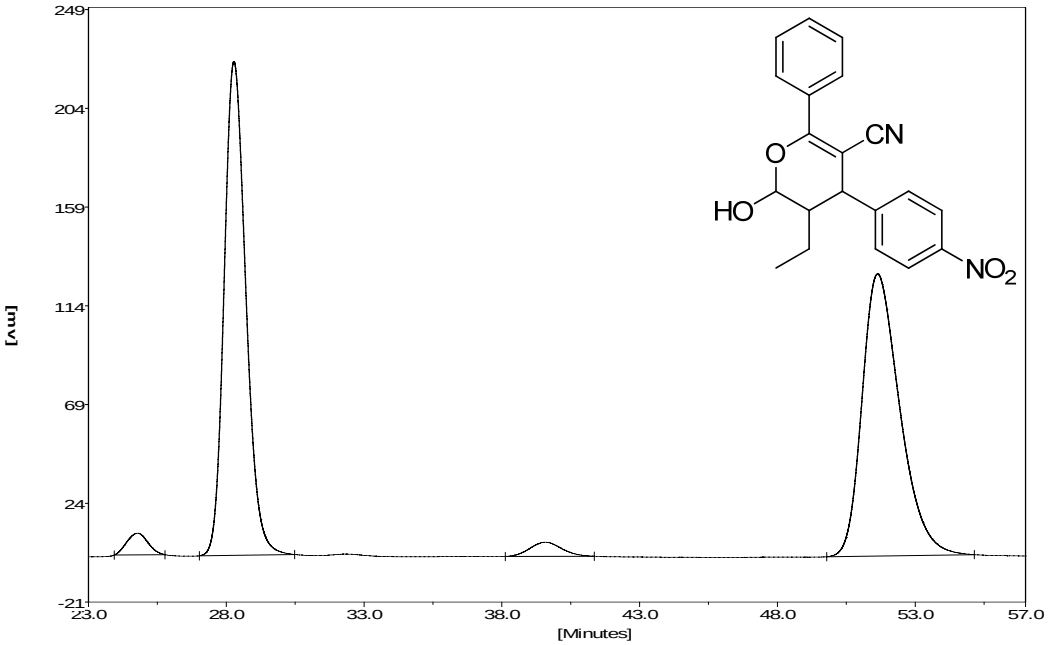
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
20.26	91.42	3153.86	2.9292
23.59	904.05	42842.24	39.7906
33.13	729.78	58739.51	54.5555
49.24	32.59	2933.56	2.7246



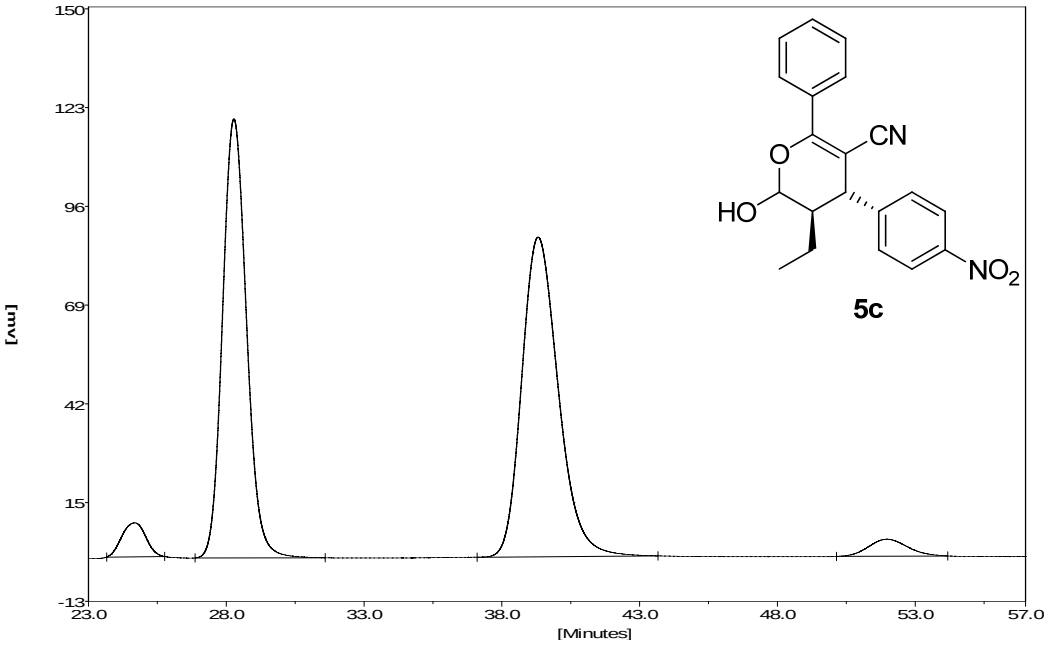
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
21.65	109.81	7409.56	31.0436
27.01	62.09	4603.23	19.2860
30.14	94.87	7270.83	30.4624
38.11	53.24	4584.61	19.2080



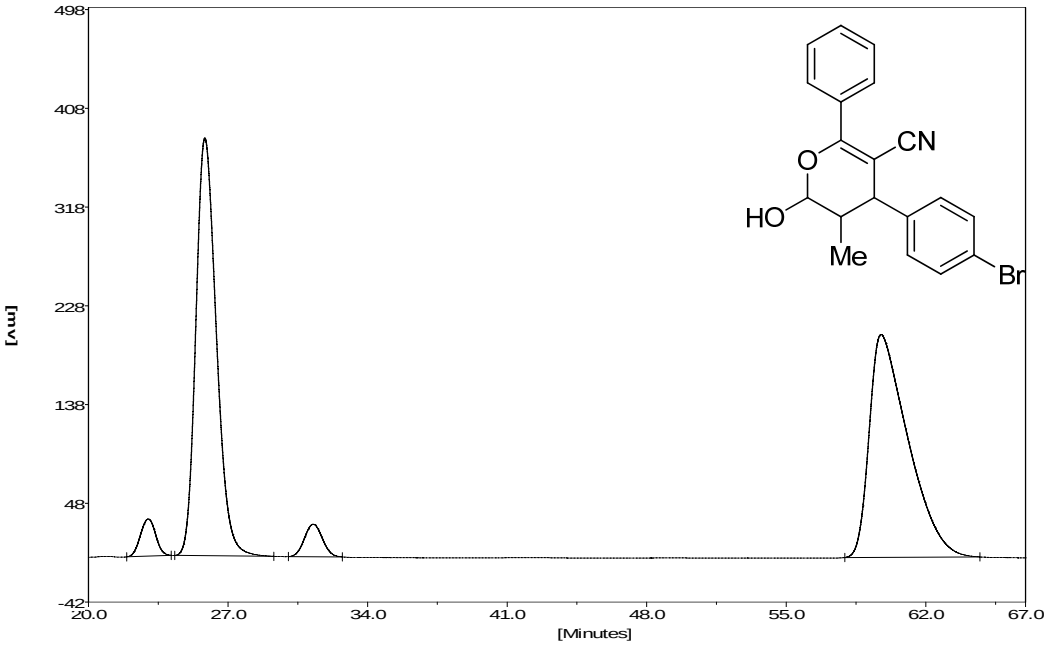
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
22.24	3.42	220.43	2.9228
27.82	43.08	3174.51	42.0912
31.76	48.82	3848.10	51.0224
40.40	3.45	298.93	3.9636



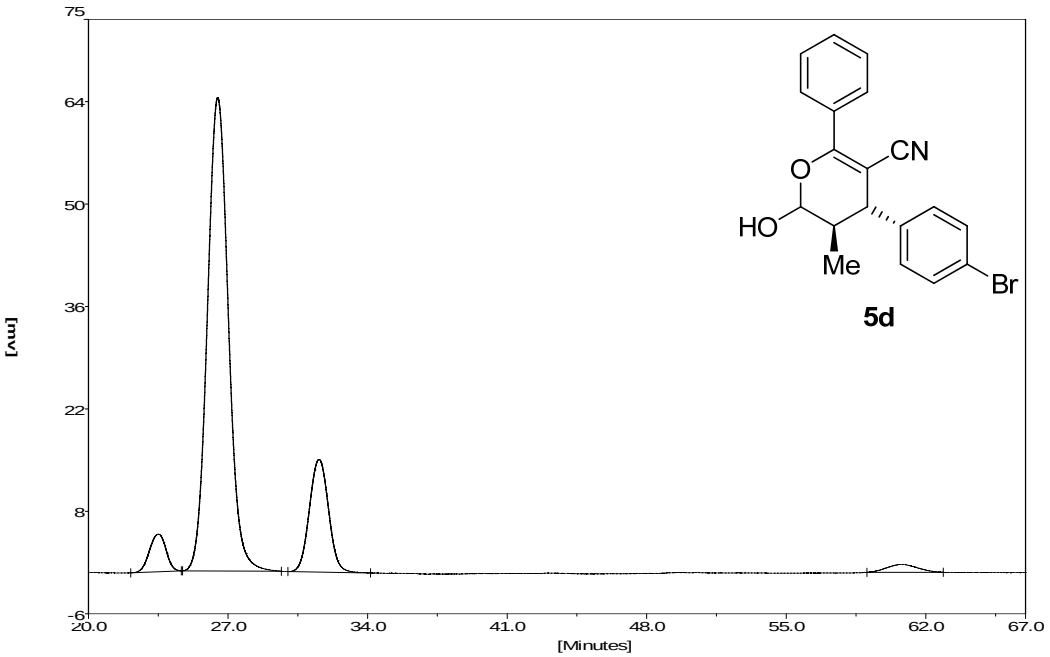
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
24.77	9.89	526.57	2.0588
28.28	225.05	12302.79	48.1019
39.59	6.45	520.35	2.0345
51.64	128.57	12226.83	47.8048



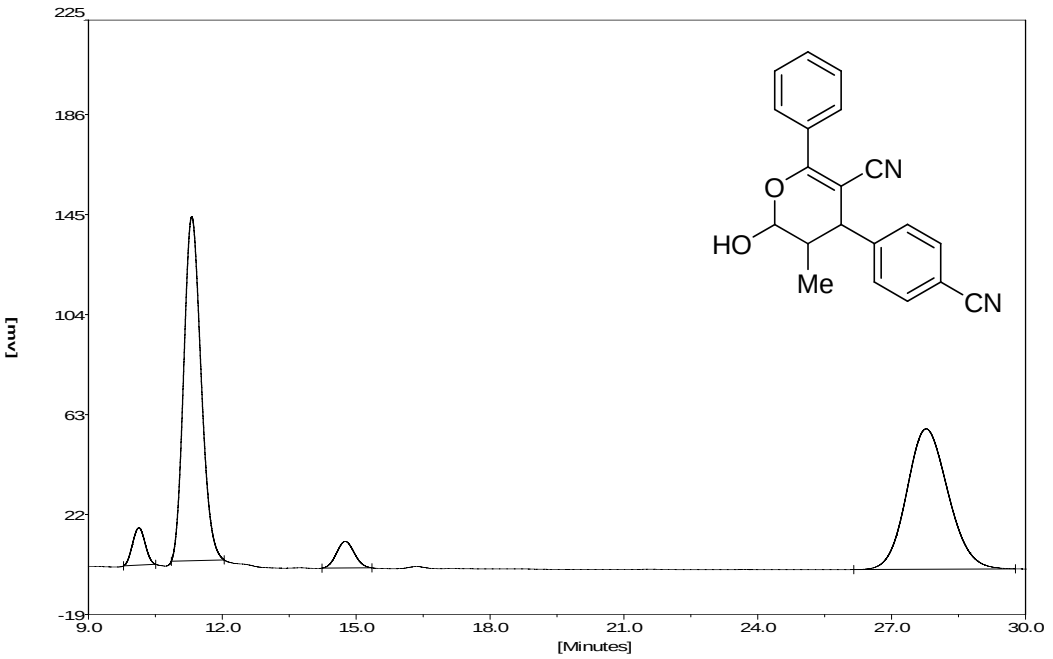
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
24.67	9.29	561.16	3.4404
28.28	120.00	7248.57	44.4402
39.32	87.41	8041.64	49.3024
51.97	4.67	459.46	2.8169



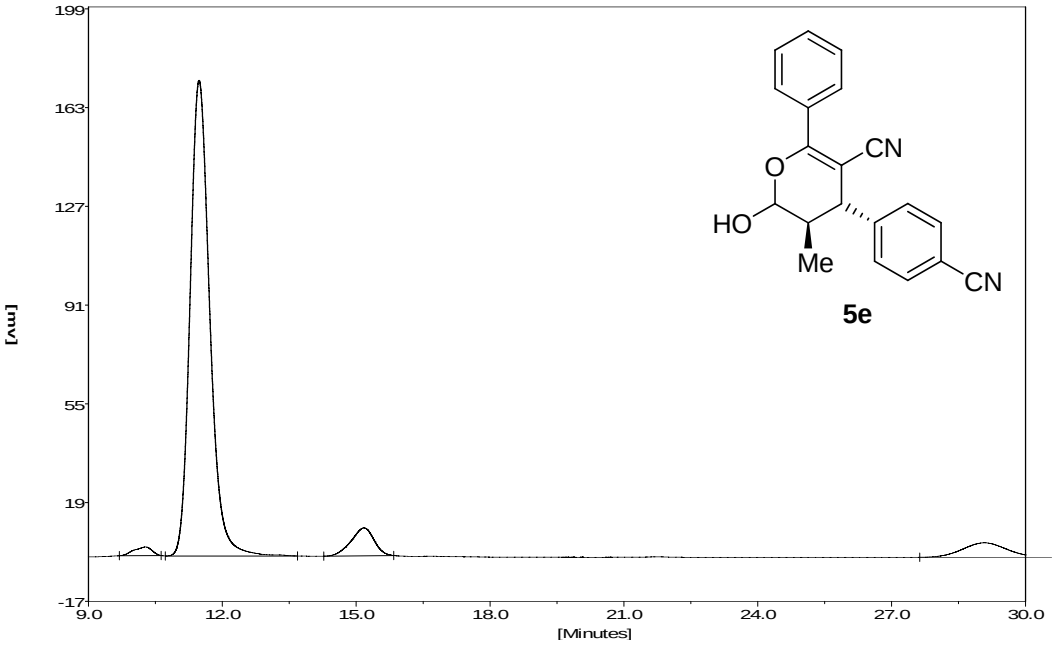
Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
23.00	33.87	1797.50	3.0856
25.84	380.65	27145.42	46.5983
31.27	29.66	1858.58	3.1905
59.76	203.17	27452.64	47.1256



Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
23.51	5.15	296.73	4.8174
26.48	64.73	4719.42	76.6196
31.56	15.41	1028.08	16.6908
60.73	1.07	115.32	1.8722



Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
10.13	15.30	299.97	3.6996
11.32	141.06	3790.57	46.7499
14.76	10.90	302.39	3.7294
27.77	57.58	3715.26	45.8210



Retention time (min)	Height (mv)	Area (mv•sec)	Area (%)
10.28	3.15	88.61	1.4506
11.48	173.41	5297.78	86.7281
15.17	10.16	364.16	5.9616
29.08	5.22	357.94	5.8597