Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Chemo- and Enantioselective Hetero-coupling of Hydroxycarbazoles Catalyzed by Chiral Vanadium(V) Complex

Makoto Sako,[†] Keigo Higashida,[†] Ganesh Tatya Kamble,[†] Kevin Kaut,[†,‡]Ankit Kumar,[†] Yuka Hirose,[†,¶] Da-Yang Zhou,[†] Takeyuki Suzuki,[†] Magnus Rueping,[‡,#] Tomohiro Maegawa,[¶] Shinobu Takizawa,*[,†] and Hiroaki Sasai*[,†]

[†]The Institute of Scientific and Industrial Research (ISIR), Osaka University, Mihogaoka, Ibaraki, Osaka 567-0047, Japan

[‡]Institute of Organic Chemistry, RWTH Aachen University, Landoltweg 1, 52074 Aachen, Germany

#Catalysis Center, King Abdullah University of Science and Technology (KAUST), Thuwal 23955-6900, Saudi Arabia

[¶]School of Pharmaceutical Sciences, Kindai University, Higashi-Osaka, Osaka 577-8502, Japan

E-mail: taki@sanken.osaka-u.ac.jp

E-mail: sasai@sanken.osaka-u.ac.jp

Tel: +81-6-6879-8465; Fax: +81-6-6879-8469

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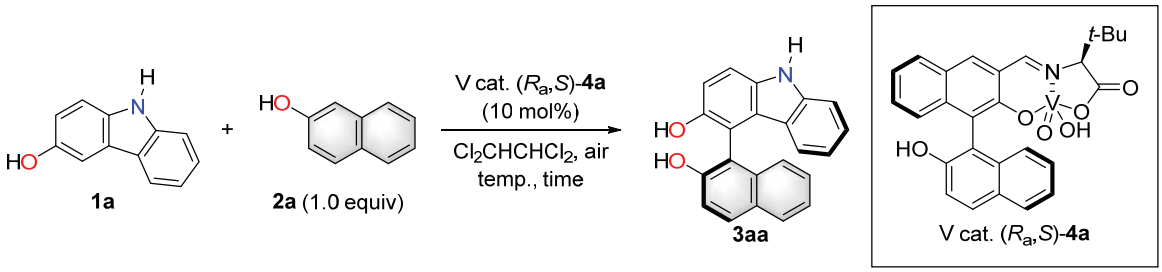
General information

^{1}H-, ^{13}C-, and ^{19}F-NMR were recorded with JEOL JMN ECS400 FT NMR, JNM ECA600 FT NMR or Bruker AVANCE II (^{1}H-NMR 400, 600 or 700 MHz, ^{13}C-NMR 100, 150 or 175 MHz, ^{19}F-NMR 565 MHz.) ^{1}H-NMR spectra are reported as follows: chemical shift in ppm relative to the chemical shift of tetramethylsilane (TMS) at 0 ppm, integration, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), and coupling constants (Hz). ^{13}C-NMR spectra reported in ppm relative to the central line of triplet for $CDCl_3$ at 77 ppm. CF_3CO_2H was used as an external standard or hexafluorobenzene (C_6F_6) was used as an internal standard for ^{19}F-NMR. FT-MS spectra were obtained with LTQ Orbitrap XL (Thermo Fisher Scientific). ESI-MS spectra were obtained with JMS-T100LC (JEOL). Optical rotations were measured with JASCO P-1030 polarimeter. HPLC analyses were performed on a JASCO HPLC system (JASCO PU 980 pump and UV-975 UV/Vis detector) using a mixture of hexane and 2-propanol as eluents. FT-IR spectra were recorded on a JASCO FT-IR system (FT/IR4100). Column chromatography on SiO_2 was performed with Kanto Silica Gel 60 (63-210 μm). Commercially available organic and inorganic compounds were used without further purification.

Optimization of reaction conditions

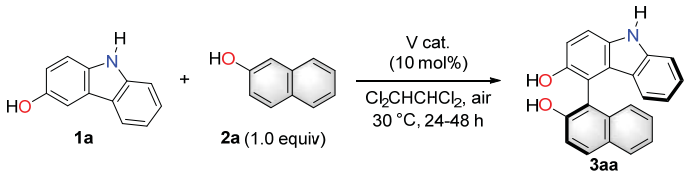
Table S1. Screening of reaction temperature[a].

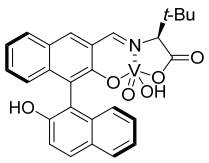


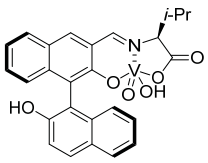
entry	temp. (°C)	time (h)	yield (%)[b]	er[c]
1	10	24	53	70:30
2	20	48	69	70:30
3	30	48	81	69:31
4	40	48	91	66:34
5	50	48	90	61:39

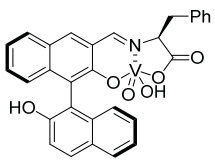
[a]The reaction of **1a** (0.05 mmol) and **2a** (0.05 mmol) with 10 mol% of (R_a,*S*)-**4a** (0.005 mmol) was carried out in $Cl_2CHCHCl_2$ (0.5 mL) at 30 °C under air (1 atm). [b]yields were determined by 1H NMR spectroscopy using 1,3,5-trimethoxybenzene as an internal standard. [c]ers were determined using HPLC (DAICEL CHIRALPAK IA).

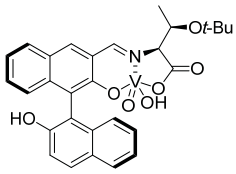
Table S2. Screening of mononuclear vanadium complexes.

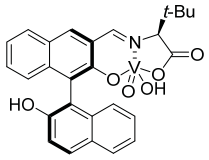



81%, 69:31 er


95%, 65:35 er


88%, 60:40 er


63%, 68:32 er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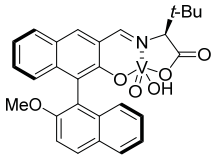
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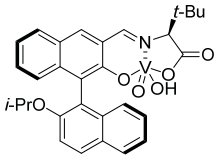
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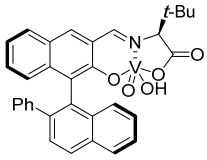
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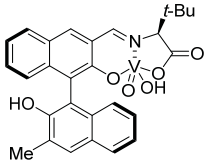
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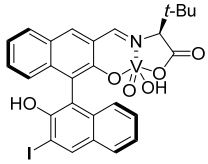
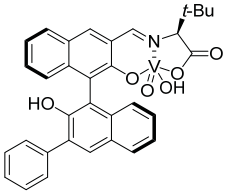

73%, 72:28 er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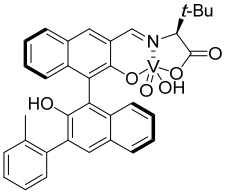
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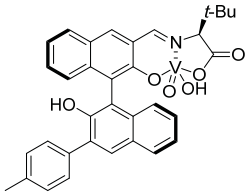
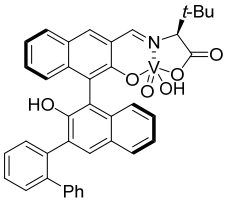

88%, 73:27 er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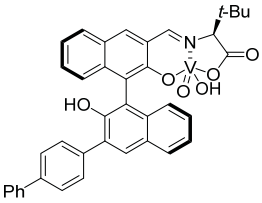
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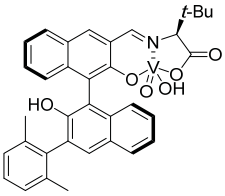
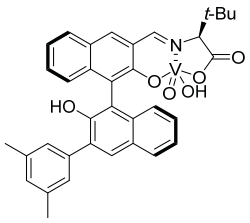
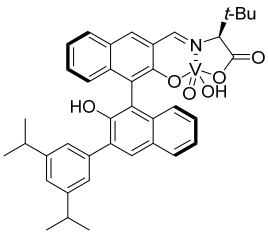
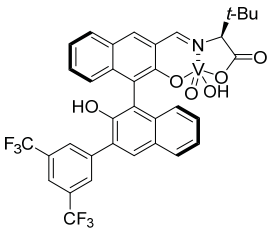
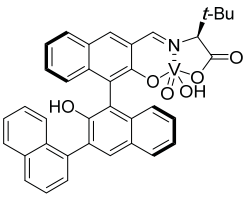
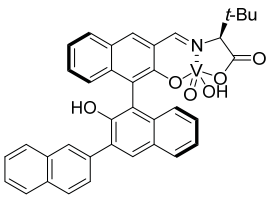

92%, 82:18 er

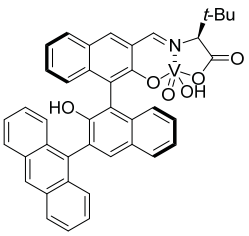

76%, 73:27 er

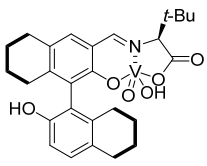

66%, 70:30 er


85%, 55:45 er


88%, 77:23 er


84%, 71:29 er


80%, 74:26 er


90%, 60:40 er

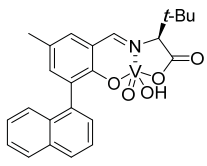
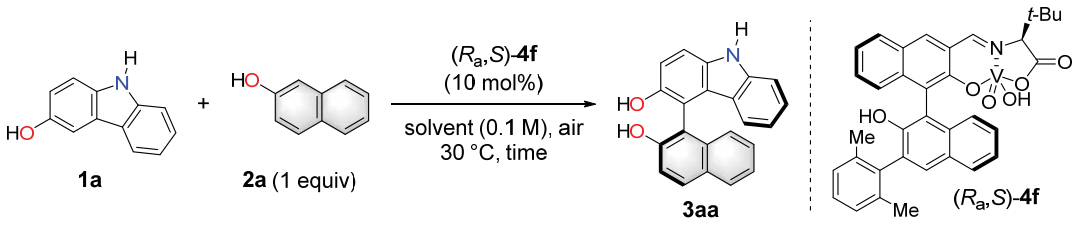

87%, 63:37 er

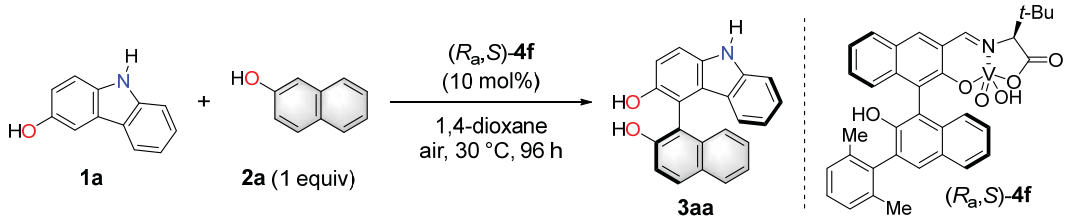
Table S3. Screening of reaction solvent.



entry	Solvent	Time (h)	NMR yield (%)	er
1	CH_2Cl_2	24	92	81:19
2	$CHCl_3$	24	69	80:20
3	CCl_4	24	57	78:22
4	$ClCH_2CH_2Cl$	24	78	81:19
5	$Cl_2CHCHCl_2$	24	>95	82:18
6	$Cl_2C{=}CCl_2$	24	67	80:20
7	PhCl	24	81	80:20
8	Toluene	24	79	79:21
9	*o*-xylene	24	77	79:21
10	*m*-xylene	24	72	79:21
11	*p*-xylene	24	79	81:19
12	Mesitylene	24	79	80:20
13	THF	48	20	88:12
14	2-MeTHF	48	59	76:14
15	MTHP[a]	48	59	85:15
16	1,4-dioxane	48	40	92:8
17	1,4-dioxane	96	74	91:9
18	Et_2O	48	49	74:26
19	*i*-Pr_2O	48	15	78:22
20	DME[b]	48	27	77:23
21	CPME[c]	48	>95	79:21
22	MTBE[d]	48	66	77:23
23	EtOAc	48	71	84:16
24	Acetone	48	40	85:15
25	MeCN	48	68	81:19
26	MeOH	48	9	58:42
27	H_2O	72	9	58:42

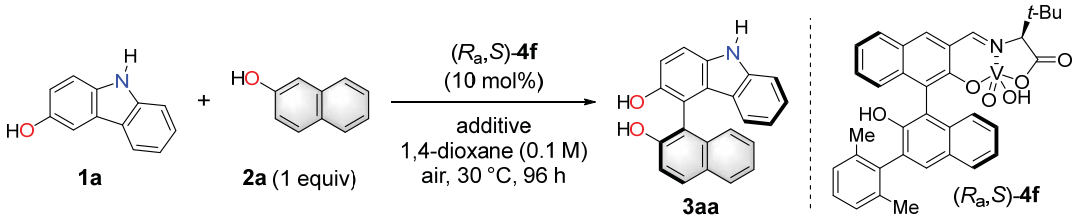
[a]MTHP = 4-Methyltetrahydropyran. [b]DME = 1,2-Dimethoxyethane. [c]CPME = Cyclopentyl Methyl Ether. [c]MTBE = Methyl tert-Butyl Ether.

Table S4. Screening of concentration.



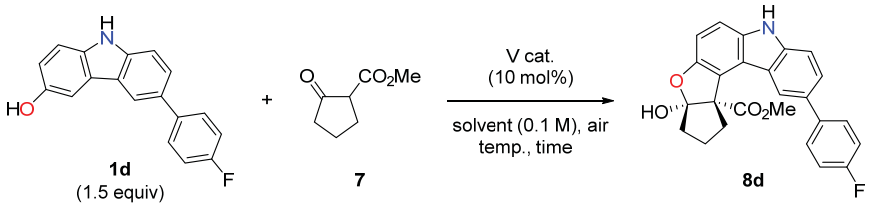
entry	C (M)	NMR yield (%)	er
1	0.05	61	91:9
2	0.1	74	91:9
3	0.2	80	87:13

Table S5. Screening of additive.



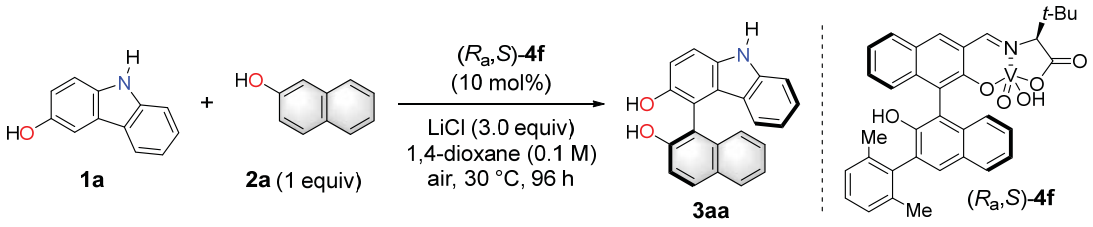
entry	Additive	amount of additive (equiv)	NMR yield (%)	er
1	None	-	74	91:9
2	AcOH	1.0	77	91:9
3	AcOH	2.0	70	87:13
4	AcOH	5.0	74	81:19
5	AcOH	10.0	83	76:24
6	PhCOOH	0.5	85	90:10
7	TMSCl	0.5	20	58:42
8	LiCl	0.5	85	93:7
9	LiCl	1.0	84	93:7
10	LiCl	2.0	84	93:7
11	LiCl	2.5	83	93:7
12	LiCl	3.0	85	94:6
13	LiCl	5.0	84	94:6
14	LiCl	10.0	78	94:6
15	LiBr	1.0	82	91:9
16	$LiClO_4$	1.0	77	87:13
17	$Mg(ClO_4)_2$	1.0	63	88:12
18	NH_4Cl	3.0	75	91:9

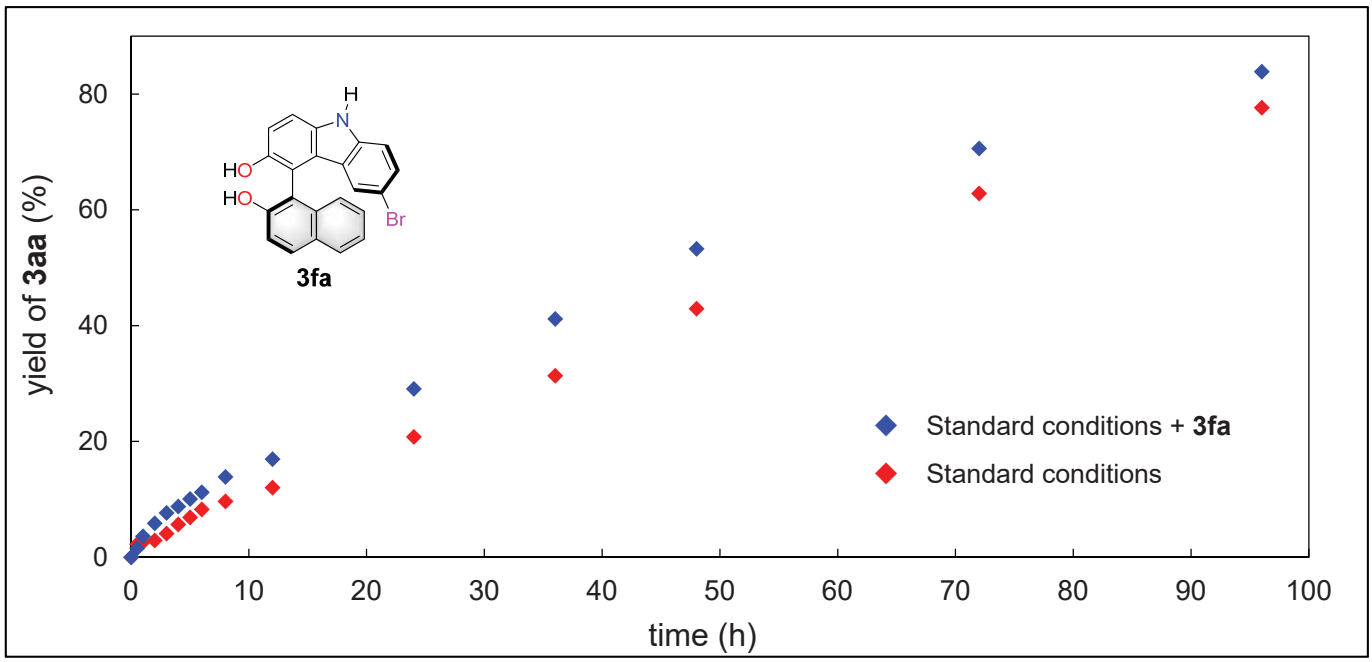
Table S6. Screening of additive.



entry	solvent	temp. (°C)	V cat.	time (h)	additive (3.0 equiv)	yield (%)	er	dr
1	1,4-dioxane	30	(R_a,S)-**4f**	96	LiCl	52	49:51	—
2	1,4-dioxane	50	(S_a,S)-**4a**	24	—	34	64:36	10:1
3	PhCl	30	(S_a,S)-**4a**	24	—	70	91:9	10:3
4	PhCl	50	(R_a,S)-**4f**	24	—	48	92:8	—
5	PhCl	50	(S_a,S)-**4a**	24	LiCl	20	81:19	10:1
6	PhCl	50	(S_a,S)-**4a**	24	—	78	90:10	>20:1

Mechanistic studies





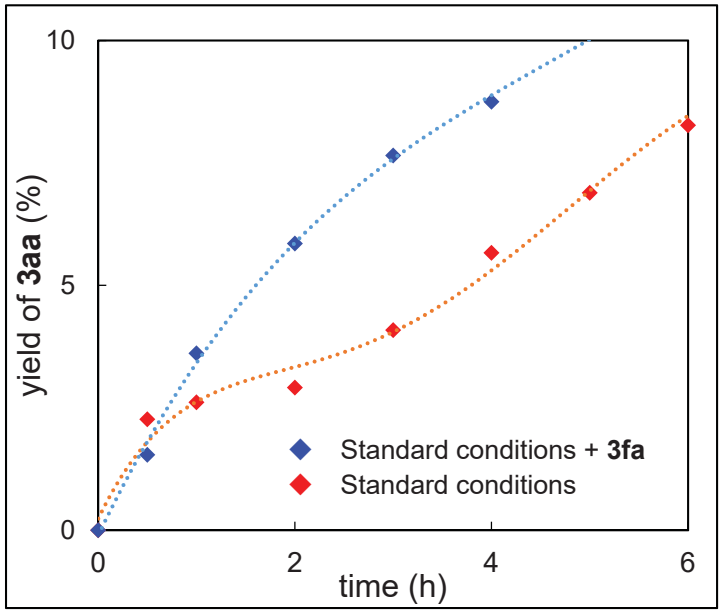


Figure S1. Reaction profile (Red makers = standard conditions. Blue markers = added 0.5 equiv of hetero-coupling product **3fa**).

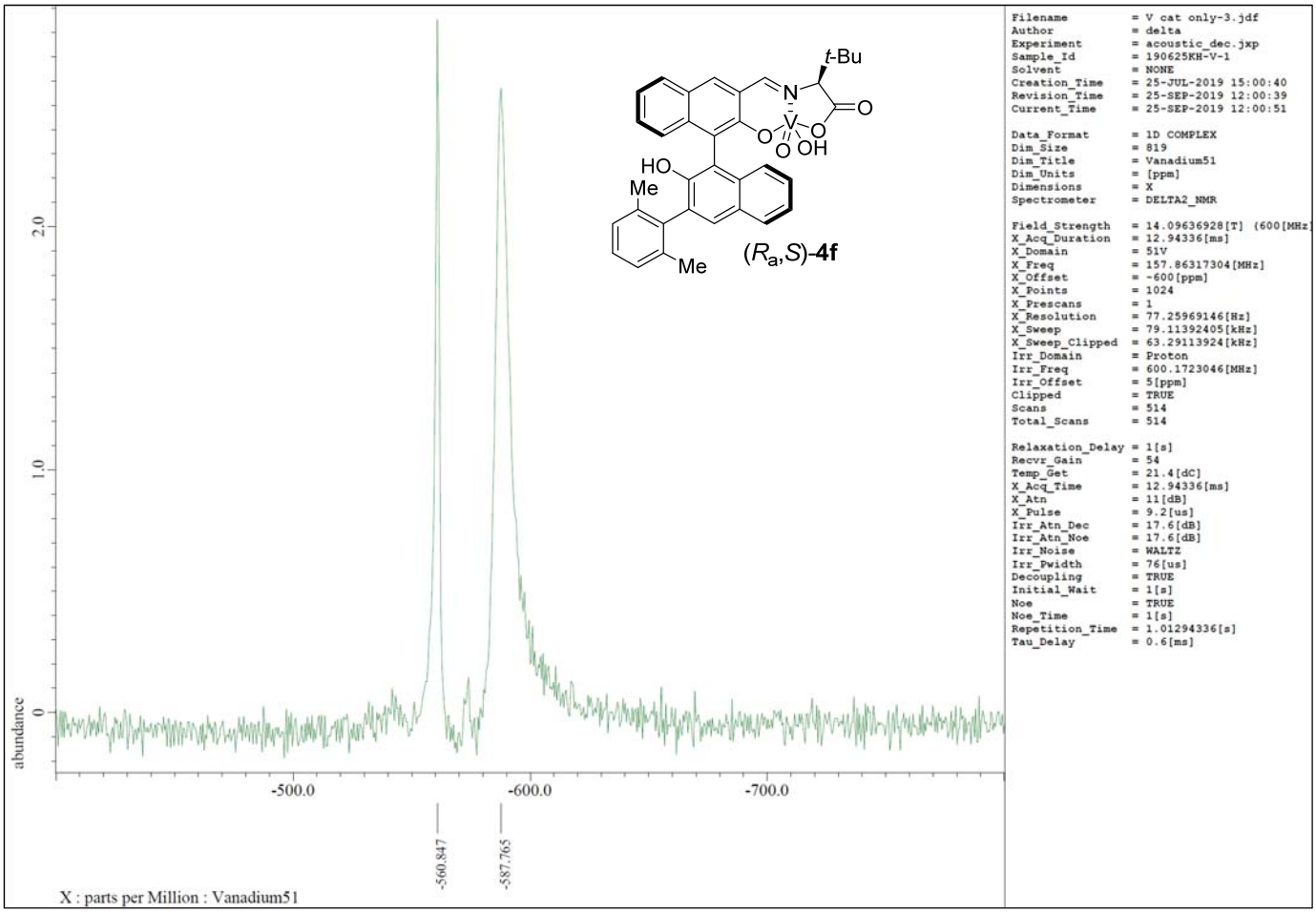
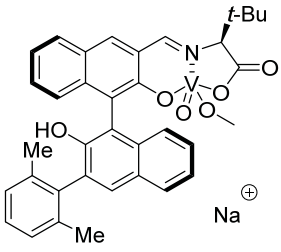
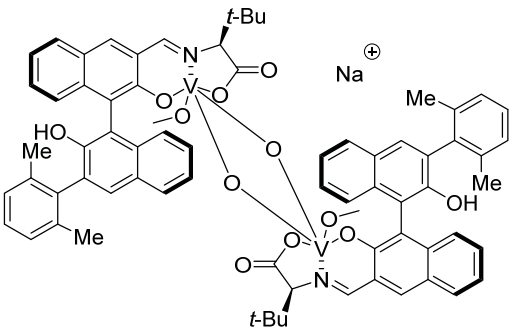


Figure S2. ^{51}V-NMR chart of V cat. (R_a,*S*)-**4f** in 1,4-dioxane at room temperature.



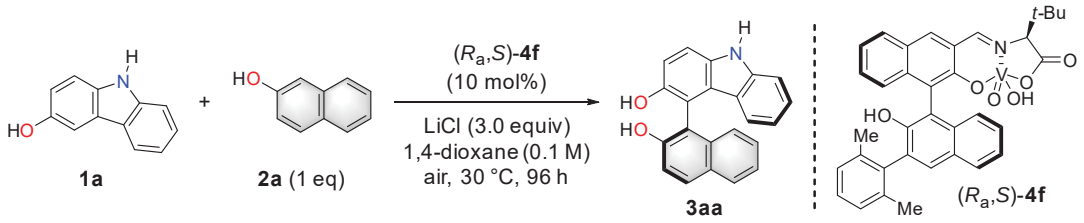
HRMS (ESI): calcd for $C_{36}H_{34}NNaO_6V$:
m/z 650.1723 [M + Na]$^+$, found 650.1724.



HRMS (ESI): calcd for $C_{72}H_{68}N_2NaO_{12}V_2$:
m/z 1277.3549 [M + Na]$^+$, found 1277.3560.

Figure S3. Mass result of V cat. (R_a,*S*)-**4f** in $CHCl_3$ and one drop of MeOH.

Table S7. Product ee vs. catalyst ee.



entry	ee of (R_a,S)-**4f** (%)	ee of **3aa** (%)	NMR yield (%)
1	16	14	79
2	38	30	73
3	58	48	72
4	77	62	75
5	100 (R_a,S)	88 (R)	85

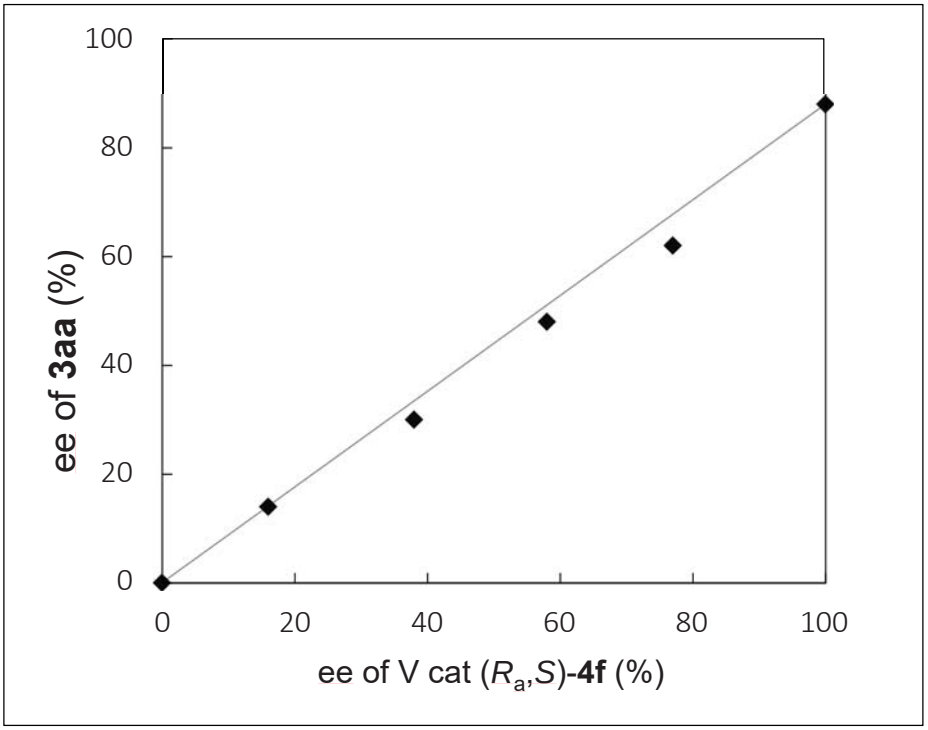


Figure S4. Product ee vs. catalyst ee.

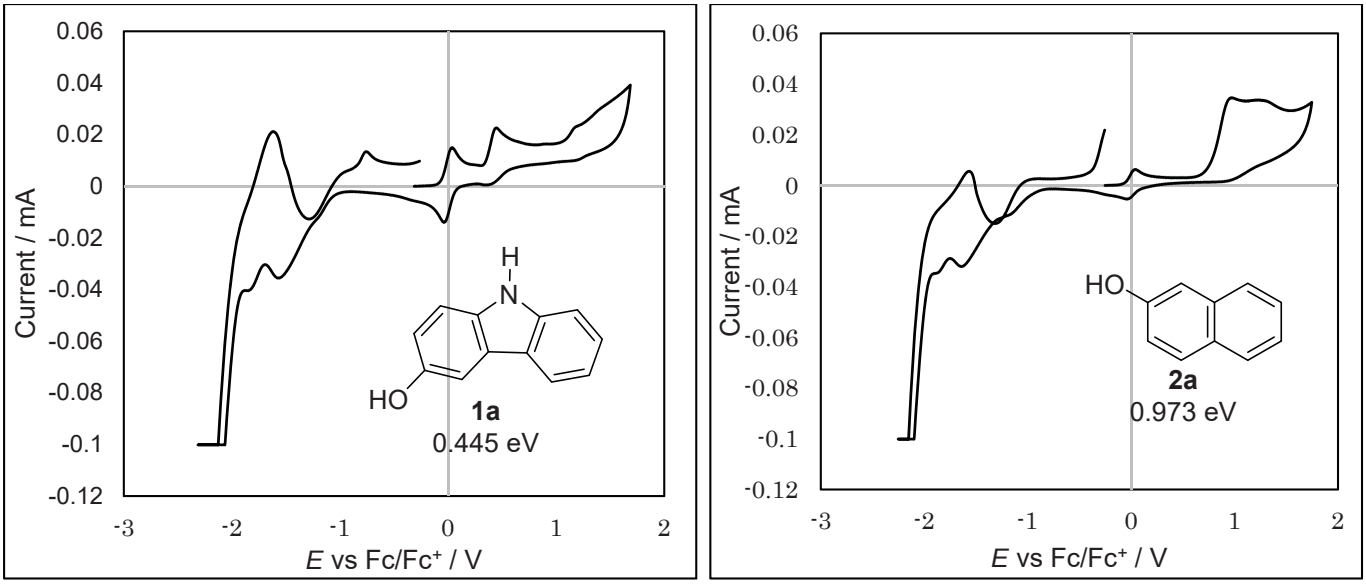
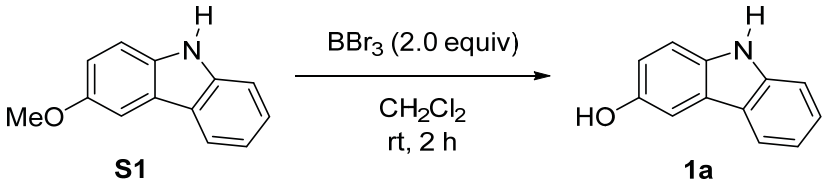


Figure S5. CV experiments.

Experimental procedures

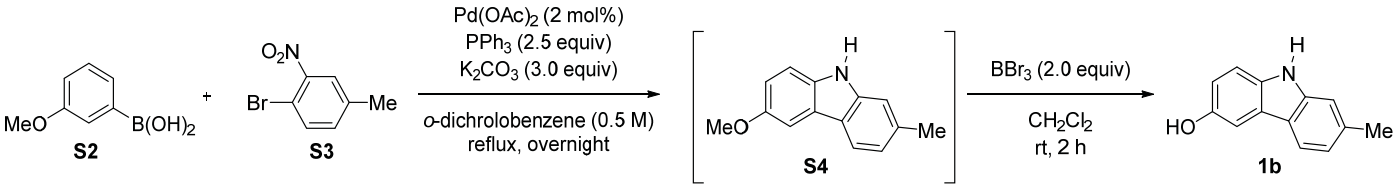
Synthesis of coupling precursor 1 and 2



9*H*-carbazol-3-ol (1a)[1)]

1a was prepared following the method described by Fujioka.[1)] Product **1a** was obtained in 92% yield as a brown solid

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.06 (s, 1H), 8.03 (d, J = 7.6 Hz, 1H), 7.93 (s, 1H), 7.56 (d, J = 2.3 Hz, 1H), 7.47 (d, J = 8.5 Hz, 1H), 7.38-7.34(m, 2H), 7.13 (ddd, J = 8.5, 7.6, 0.9 Hz, 1H), 7.00 (dd, J = 8.5, 2.3 Hz, 1H).



7-Methyl-9*H*-carbazol-3-ol (1b)[2)]

S4 was prepared according to the literature procedure.[3)] The mixture of 1-bromo-4-methyl-2-nitrobenzene (**S2**) (500 mg, 2.31 mmol), (3-methoxyphenyl)boronic acid (**S3**) (457 mg, 3.01 mmol), $Pd(OAc)_2$ (10 mg, 5.79 mmol), PPh_3 (1.52 g, 5.79 mmol), K_2CO_3 (640 mg, 4.63 mmol) and *o*-DCB (4.6 mL). The mixture was heated at 180 °C (oil bath temperature) for overnight, the reaction mixture was allowed to cool to ambient temperature. The reaction mixture was filtered through a pad of celite and the resulting liquor was concentrated under vacuum. The crude product was purified by silica column chromatography to afford 6-methoxy-2-methyl-9*H*-carbazole (**S4**). A subsequent deprotection of methyl group was performed following the procedure for preparation of compound **1a** to give 7-methyl-9*H*-carbazol-3-ol (**1b**) in 24% overall yield in 2 steps as a white solid.

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 9.90 (s, 1H), 7.89 (d, J = 7.8 Hz, 1H), 7.56 (d, J = 0.9 Hz, 1H), 7.50 (d, J = 2.8 Hz, 1H),

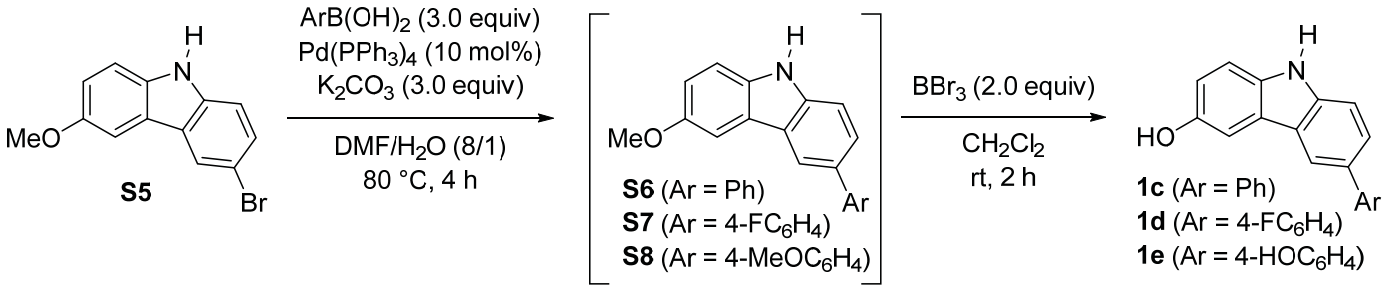
7.32 (d, J = 8.7 Hz, 1H), 7.27 (s, 1H), 6.98-6.94 (m, 2H), 2.49 (s, 3H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 151.4, 142.1, 135.7, 135.0, 124.6, 121.4, 120.32, 120.29, 114.9, 111.7, 111.4, 105.4, 21.8.

HRMS (APCI): calcd for $C_{13}H_{12}NO$: *m/z* 198.0919 $[M + H]^+$, found 198.0908.

IR (KBr): 3498, 3399, 3271, 3029, 1493, 1458, 1445, 1188, 809, 442 cm^{-1}.

mp: 142-144 °C (white solid).



6-Phenyl-9*H*-carbazol-3-ol (1c)

S5[4] (420 mg, 1.52 mmol), phenyl boronic acid (556 mg, 4.56 mmol), $Pd(PPh_3)_4$ (176 mg, 0.15 mmol) and potassium carbonate (631 mg, 4.56 mmol) in DMF (15.0 mL) and water (1.9 mL) was stirred for 4 h at 80 °C. After cooling, the reaction mixture was quenched with 0.5 M NaOH aq., and extracted with EtOAc. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and evaporated under vacuum. The crude product was purified by silica column chromatography to afford 3-methoxy-6-phenyl-9*H*-carbazole (**S6**). A subsequent deprotection of methyl group was performed following the procedure for preparation of compound **1a** to give 6-phenyl-9*H*-carbazol-3-ol (**1c**) in 86% overall yield in 2 steps as a brown solid.

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.16 (s, 1H), 8.34 (d, J = 0.9 Hz, 1H), 8.05-8.01 (m, 1H), 7.79 (s, 1H), 7.70 (dd, J = 8.2, 1.8 Hz, 1H), 7.67 (d, J = 1.8 Hz, 1H), 7.56 (d, J = 8.2 Hz, 1H), 7.50 (t, J = 7.6 Hz, 2H), 7.40 (d, J = 8.7 Hz, 1H), 7.35 (t, J = 7.6 Hz, 1H), 7.04 (dd, J = 8.7, 1.8 Hz, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 151.4, 142.7, 140.9, 135.4, 131.9, 129.2, 127.3, 126.6, 125.2, 124.5, 124.1, 118.8, 115.7, 111.9, 111.6, 105.7.

HRMS (APCI): calcd for $C_{18}H_{14}NO$: *m/z* 260.1075 $[M + H]^+$, found 260.1068.

IR (KBr): 3532, 3407, 3030, 2962, 1490, 1459, 1170, 820, 756, 696, 597 cm^{-1}.

mp: 223-225 °C (brown solid).

6-(4-Fluorophenyl)-9*H*-carbazol-3-ol (1d)

The procedure followed that for preparation of **1c**. Product **1d** was obtained in 83% yield as a brown solid.

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.15 (s, 1H), 8.32 (d, J = 1.4 Hz, 1H), 7.99 (s, 1H), 7.83-7.78 (m, 2H), 7.68-7.65 (m, 2H), 7.56 (d, J = 8.2 Hz, 1H), 7.40 (d, J = 8.7 Hz, 1H), 7.29-7.23 (m, 2H), 7.04 (dd, J = 8.7, 2.3 Hz, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 162.4 (d, $^1J_{C\text{-}F}$ = 243.5 Hz), 151.5, 141.0, 139.2 (d, $^4J_{C\text{-}F}$ = 3.8 Hz), 135.5, 131.0, 129.2 (d, $^3J_{C\text{-}F}$ = 7.7 Hz), 125.2, 124.5, 124.2, 118.9, 115.9 (d, $^2J_{C\text{-}F}$ = 21.1 Hz), 115.9, 112.0, 111.7, 105.8.

^{19}F NMR (565 MHz, $(CD_3)_2CO$): δ –118.3.

HRMS (APCI): calcd for $C_{18}H_{13}FNO$: *m/z* 278.0981 $[M + H]^+$, found 278.0972.

IR (KBr): 3519, 3406, 3075, 3025, 1491, 1458, 1240, 1216, 1166, 816, 600, 521 cm^{-1}.

mp: 214-216 °C (brown solid).

6-(4-Hydroxyphenyl)-9*H*-carbazol-3-ol (1e)

The procedure followed that for preparation of **1c**. Product **1e** was obtained in 70% yield as a white solid

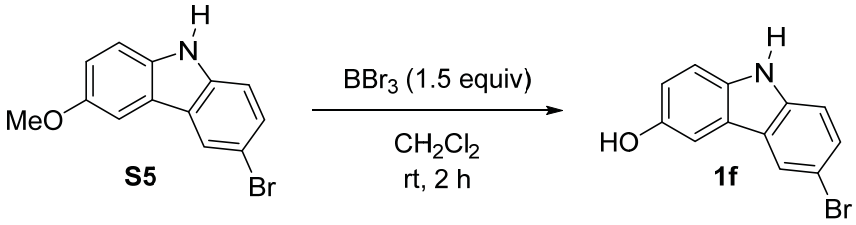
^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.07 (s, 1H), 8.34 (s, 1H), 8.24 (d, J = 1.8 Hz, 1H), 7.96 (s, 1H), 7.64-7.60 (m, 4H), 7.52 (d, J = 8.7 Hz, 1H), 7.38 (d, J = 8.6 Hz, 1H), 7.02 (dd, J =8.6, 2.5 Hz, 1H), 6.98 (dd, J = 6.4, 1.8 Hz, 2H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 156.8, 151.4, 140.5, 135.5, 134.3, 132.2, 128.5, 125.0, 124.6, 124.2, 118.2, 116.2, 115.6, 111.9, 111.5, 105.7.

HRMS (APCI): calcd for $C_{18}H_{14}NO_2$: *m/z* 276.1025 $[M + H]^+$, found 276.1016.

IR (KBr): 3395, 3296, 1496, 1462, 1220, 1168, 811, 602, 593 cm^{-1}.

mp: 261-263 °C (white solid).



6-Bromo-9*H*-carbazol-3-ol (1f)[5)]

1f was prepared following the method described by Ohizumi.[5)] Product **1f** was obtained in 92% yield as a white solid.

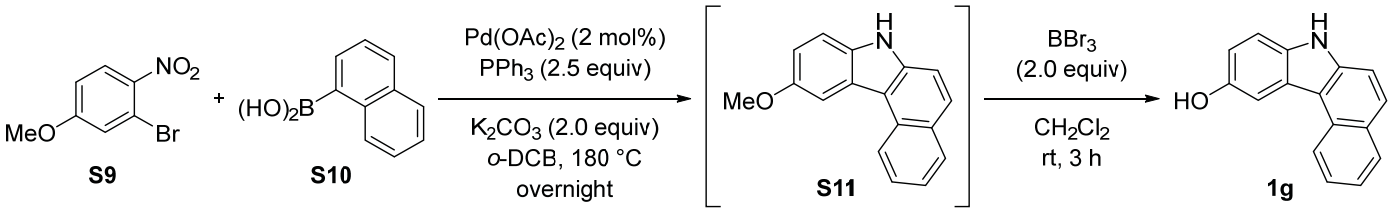
^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.25 (s, 1H), 8.21 (d, J = 1.6 Hz, 1H), 8.08 (s, 1H), 7.59 (d, J = 2.5 Hz, 1H), 7.45 (dd, J = 8.6, 1.6 Hz, 1H), 7.45 (d, J = 8.7 Hz, 1H), 7.41 (d, J = 8.7 Hz, 1H), 7.06 (dd, J = 8.7, 2.5 Hz, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 151.6, 139.9, 135.3, 128.2, 125.3, 123.2, 123.1, 116.5, 113.0, 112.1, 110.8, 105.6.

HRMS (APCI): calcd for $C_{12}H_9BrNO$: *m/z* 182.0600 $[M - Br]^+$, found 182.0602.

IR (KBr): 3532, 3397, 3080, 1585, 1487, 1150, 1130, 850, 812, 575 cm^{-1}.

mp: 171-173 °C (white solid).



7*H*-Benzo[*c*]carbazol-10-ol (1g)

A mixture of 2-bromo-4-methoxy-1-nitrobenzene **S9**[6)] (574.6 mg, 2.48 mmol), commercially available aryl boronic acid **S10** (553.7 mg, 3.22 mmol), $Pd(OAc)_2$ (11.1 mg, 0.05 mmol), PPh_3 (1.62 g, 6.19 mmol) and K_2CO_3 (684.5 mg, 4.95 mmol) in *o*-DCB (5.0 mL) was heated at 180 °C (oil bath temperature) for 20 h. After cooling, the reaction mixture was filtrated through celite and the resulting liquor was concentrated in vacuo. The crude product was purified by silica column chromatography to afford 10-methoxy-7*H*-benzo[*c*]carbazole (**S11**). A subsequent deprotection of methyl group was performed following the procedure for preparation of compound 1a to give 7*H*-benzo[*c*]carbazol-10-ol (**1g**) in 56% overall yield in 2 steps as a brown solid.

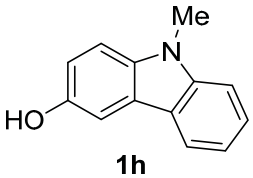
^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.62 (s, 1H), 8.71 (d, J = 8.7 Hz, 1H), 8.06-8.03 (m, 3H), 7.89 (d, J = 8.7 Hz, 1H), 7.76 (d, J = 8.7 Hz, 1H), 7.72 (ddd, J =6.6, 5.7, 0.9 Hz, 1H), 7.54 (d, J = 7.9 Hz, 1H), 7.47 (ddd, J =6.6, 5.7, 0.9 Hz, 1H), 7.07 (ddd, J = 8.7, 2.3, 0.9 Hz, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 151.9, 138.9, 134.0, 130.7, 129.6, 129.5, 127.3, 127.0, 124.8, 123.1, 122.8, 114.9, 114.2, 113.8, 112.4, 107.0.

HRMS (APCI): calcd for $C_{16}H_{12}NO$: *m/z* 234.0919 $[M + H]^+$, found 234.0910.

IR (KBr): 3524, 3400, 3348, 1492, 1209, 1164, 832, 803, 753, 737 cm^{-1}.

mp: 212-214 °C (brown solid).

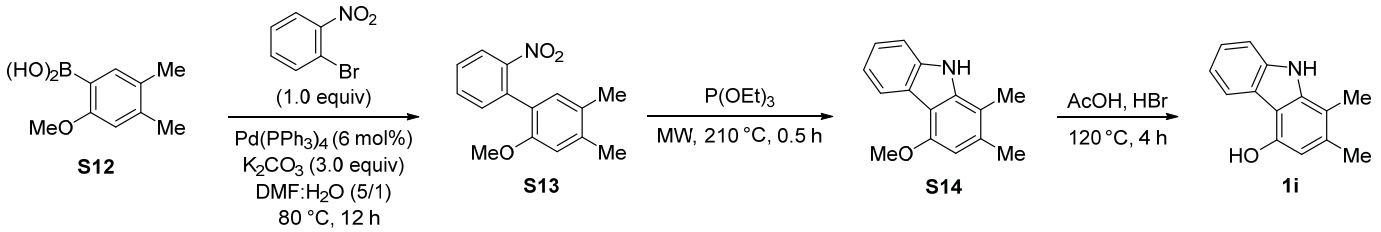


1h

9-Methyl-9*H*-carbazol-3-ol (1h)[7)]

1h was prepared following the method described by Takizawa.[7)]

Synthesis of 1,2-dimethyl-9H-carbazol-4-ol (1i)



2-Methoxy-4,5-dimethyl-2'-nitro-1,1'-biphenyl (S13)

In a dry round bottom flask, commercially available aryl boronic acid **S12** (1.1 equiv), K_2CO_3 (3.0 equiv), and $Pd(PPh_3)_4$ (6 mol%) were dissolved in DMF/H_2O (5:1, 0.25 M for **S12**). Then, 1-bromo-2-nitrobenzene (1.0 equiv, commercially available) was added, and the resulting mixture was heated at 80 °C under N_2 atmosphere. After 12 h, the mixture was poured into water and then extracted with EtOAc and dried over Na_2SO_4. After concentration of the mixture under reduced pressure, the residue was purified by column chromatography on silica gel (*n*-hexane:EtOAc = 5/1 to 1/1) to give **S13** in 72% yield as a light yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.60 (ddd, *J* = 7.8, 7.6, 1.2 Hz, 1H), 7.39-7.49 (m, 2H), 7.08 (s, 1H), 6.70 (s, 1H), 3.67 (s, 3H), 2.31 (s, 3H), 2.26 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 153.89, 149.88, 138.40, 133.16, 132.72, 132.60, 130.87, 129.18, 127.82, 124.11, 123.98, 112.50, 55.36, 20.32, 19.05.

HRMS (ESI): calcd for $C_{15}H_{15}NO_3$: *m/z* 280.1052 $[M + Na]^+$, found 280.0943.

IR (KBr): 2980, 2915, 1616, 1512, 1446, 1359, 1140, 813 cm^{-1}.

mp: 88-90 °C (light yellow solid).

4-Methoxy-1,2-dimethyl-9H-carbazole (S14)[8)]

S13 (1.0 equiv) was dissolved in $P(OEt)_3$ (0.3 M for **S13**) and stirred at 210 °C for 0.5 h in microwave reactor. After the completion, the mixture was concentrated under reduced pressure. Then, the residue was purified by column chromatography on silica gel (*n*-hexane:EtOAc = 7/1 to 1/3) to give **S14** in 80% yield as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, *J* = 7.8 Hz, 1H), 7.88 (s, 1H), 7.41 (d, *J* = 7.8 Hz, 1H), 7.34 (ddd, *J* = 7.8, 7.3, 0.9 Hz, 1H), 7.21 (ddd, *J* = 7.6, 7.3, 1.2 Hz, 1H), 6.53 (s, 1H), 4.04 (s, 3H), 2.46 (s, 3H), 2.39 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 153.89, 140.72, 138.90, 134.42, 124.46, 123.58, 122.86, 119.67, 110.63, 110.17, 110.02, 103.24, 55.54, 20.37, 12.86.

HRMS (ESI): calcd for $C_{15}H_{15}NO_3$: *m/z* 248.1154 $[M + Na]^+$, found 248.1047.

IR (KBr): 3403, 3381, 2997, 2849, 1605, 1523, 1515, 1415, 1150 cm^{-1}.

mp: 92-94 °C (white solid).

1,2-Dimethyl-9H-carbazol-4-ol (1i)[9)]

In a dry round bottom flask, **S14** was dissolved in AcOH (0.16 M for **S14**) at room temperature under N_2 atmosphere. The reaction mixture was reflux at 120 ºC for 15 min. Then aq. HBr (47%) (20 equiv) was added dropwise over 10 min. After 4 h, the reaction mixture was diluted with water and extracted with EtOAc, dried over Na_2SO_4. After the concentration under reduced pressure, the residue was purified by column chromatography on silica gel (*n*-hexane:EtOAc = 5/1 to 1/2) to give **1i** in 50% yield as a light yellow solid.

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.01 (s, 1H), 8.55 (s, 1H), 8.20 (d, J = 7.8 Hz, 1H), 7.41 (d, J = 8.2 Hz, 1H), 7.26 (ddd, J = 7.8, 7.6, 1.2 Hz, 1H), 7.10 (ddd, J = 7.8, 7.3, 0.9 Hz, 1H), 6.50 (s, 1H), 2.37 (s, 3H), 2.33 (s, 3H).

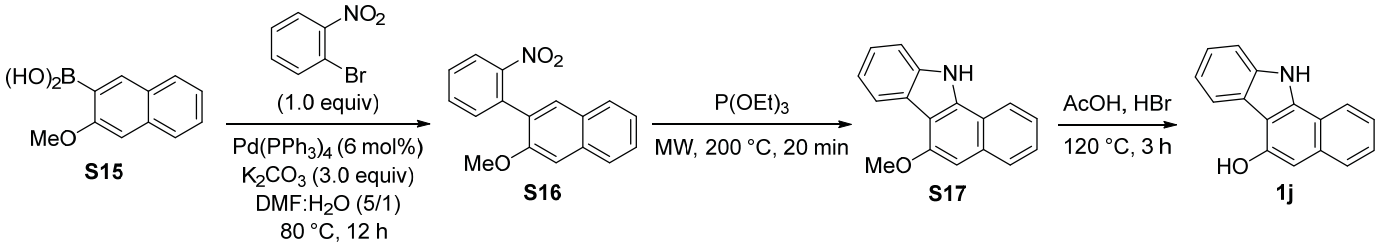
^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 151.84, 142.37, 140.48, 134.67, 124.65, 124.32, 123.13, 119.40, 110.89, 110.63, 109.72, 107.65, 19.95, 12.81.

HRMS (ESI): calcd for $C_{15}H_{15}NO_3$: *m/z* 234.0997 $[M + Na]^+$, found 234.0888.

IR (KBr): 3462, 3358, 1629, 1591, 1455, 1330, 1129, 1025, 748, 732 cm^{-1}.

mp: 170-172 °C (light yellow solid).

Synthesis of 11*H*-benzo[*a*]carbazol-6-ol (1j)



2-Methoxy-3-(2-nitrophenyl)naphthalene (S16)

In an oven dried round bottom flask, (3-methoxynaphthalen-2-yl)boronic acid[11)] (**S15**, 680 mg, 3.6 mmol), K_2CO_3 (3.0 equiv), and $Pd(PPh_3)_4$ (6.5 mol%) were dissolved in DMF:H_2O (5/1, 0.25 M for **S15**). Then, 1-bromo-2-nitrobenzene (1.0 equiv.) was added, and the resulting mixture was heated to 80 °C under N_2 atmosphere. After 12 h, the mixture was then poured into water, and extracted with EtOAc (3 times) and dried over anhydrous Na_2SO_4. After concentration of the mixture under reduced pressure, the residue was purified by column chromatography on silica gel (*n*-hexane:EtOAc = 9/1) to provide **S62** (750 mg, 74% yield) as a yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (dd, J = 8.4, 1.1 Hz, 1H), 7.79 (dd, J = 17.2, 8.0 Hz, 3H), 7.68 (td, J = 7.6, 1.4 Hz, 1H), 7.46-7.54 (m, 3H), 7.38 (t, J = 7.2 Hz, 1H), 7.15 (s, 1H), 3.80 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 154.28, 149.77, 134.63, 133.81, 133.12, 132.84, 130.17, 129.35, 129.08, 128.48, 127.97, 126.99, 126.73, 124.46, 124.28, 124.08, 123.84, 105.59, 77.49, 77.16, 76.85, 55.39.

HRMS (ESI): calcd for $C_{17}H_{13}NO_3$: *m/z* 302.0787 $[M + Na]^+$, found 302.0787.

IR (KBr): 3063, 3003, 2948, 2839, 2374 ,1958, 1781, 1573, 1349, 1123, 980 cm^{-1}.

mp: 137-139 ºC (yellow solid).

6-Methoxy-11*H*-benzo[*a*]carbazole (S17)

S16 (700 mg, 2.5 mmol) was dissolved in $P(OEt)_3$ (0.3 M for **S16**) and stirred at 210 ºC for 20 min in microwave reactor. After the completion, the mixture was concentrated under reduced pressure. Then, the residue was purified by column chromatography on silica gel (*n*-hexane:EtOAc = 5/1) to provide **S17** (510 mg, 82% yield) as a white solid.

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 11.19 (s, 1H), 8.18-8.25 (m, 2H), 7.79-7.81 (m, 1H), 7.48 (d, J = 8.4 Hz, 1H), 7.22-7.37 (m, 3H), 6.93 (s, 1H), 4.04 (s, 3H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 205.40, 155.37, 138.73, 136.79, 134.01, 127.58, 125.63, 124.16, 123.06, 123.00, 122.47, 121.26, 119.57, 117.93, 110.94, 110.59, 96.74, 54.94, 29.61, 29.42, 29.22, 29.03, 28.83, 28.64, 28.45.

HRMS (ESI): calcd for $C_{17}H_{13}NO$: *m/z* 270.0889 $[M + Na]^+$, found 271.1878.

IR (KBr): 3419, 2970, 2931, 2361, 1628, 1528, 1293, 1046, 931 cm^{-1}.

mp: 205-207 °C (white solid).

11*H*-Benzo[*a*]carbazol-6-ol (1j)

In an oven dried round bottom flask, **S17** (490 mg, 1.98 mmol) was dissolved in AcOH (0.16 M) at room temperature under N_2 atmosphere. The reaction mixture was reflux at 120 ºC. After 15 min, aq. HBr (47%) (20 equiv) was added dropwise over 10 min. After stirring fof 3 h, the reaction mixture was diluted with water (20 mL) and the organic layer was extracted with dichloromethane (3 times) and dried over anhydrous Na_2SO_4. After concentration under reduced pressure, the residue was purified by column chromatography on silica gel (*n*-hexane:EtOAc = 6/1) to give **1j** (358 mg, 77% yield) as a red solid.

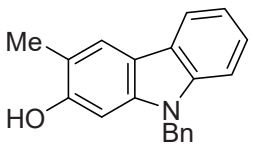
^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 12.08 (s, 1H), 10.25 (s, 1H), 8.30 (d, J = 8.0 Hz, 1H), 8.20 (d, J = 7.6 Hz, 1H), 7.71 (d, J = 7.6 Hz, 1H), 7.54 (d, J = 7.6 Hz, 1H), 7.28-7.37 (m, 3H), 7.14 (t, J = 7.2 Hz, 1H), 6.81 (s, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 153.16, 138.81, 137.45, 134.01, 126.97, 125.94, 124.39, 123.10, 122.53, 122.44, 122.18, 119.68, 117.13, 111.45, 110.39, 100.10, 40.63, 40.43, 40.22, 40.01, 39.80, 39.59, 39.38.

HRMS (ESI): calcd for $C_{16}H_{11}NO$: *m/z* 256.0733 $[M + Na]^+$, found 256.0732.

IR (KBr): 3211, 3107, 2767, 1600, 1546, 1452, 1315, 1195, 921 cm^{-1}.

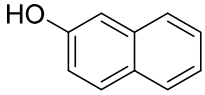
mp: 258-260 °C (red solid).



1k

9-Benzyl-3-methyl-9*H*-carbazol-2-ol (1k)[10)]

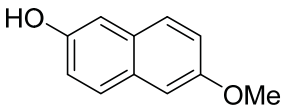
1k was prepared following the method described by Kozlowski.[10)]



2a

2-Naphthol (2a)

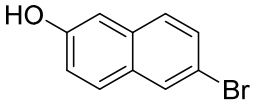
2a was purchased from Tokyo Chemical Industry CO., LTD.



2b

6-Methoxy-2-naphthol (2b)[11)]

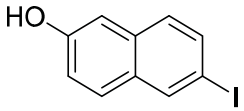
2a was prepared following the method described by Mei.[11)]



2c

6-Bromo-2-naphthol (2c)

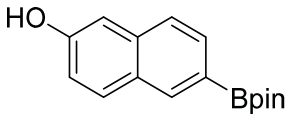
2c was purchased from Tokyo Chemical Industry CO., LTD.



2d

6-Iodo-2-naphthol (2d)[12)]

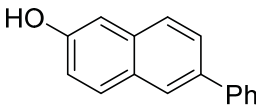
2d was prepared following the method described by Ball.[12)]



2e

6-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)-2-naphthol (2e)[11)]

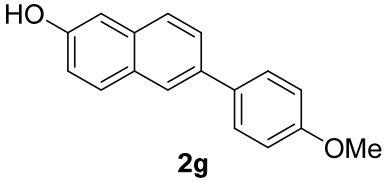
2e was prepared following the method described by Mei.[11)]



2f

6-Phenyl-2-naphthol (2f)[13)]

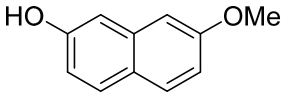
2e was prepared following the method described by Bin and You.[13)]



2g

6-(4-Anisyl)--2-naphthol (2g)[14)]

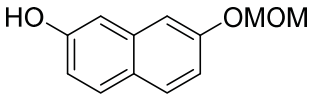
2g was prepared following the method described by Gao, Zhang, and Shi.[14)]



2h

7-Methoxy-2-naphthol (2h)[13)]

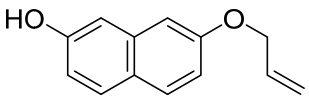
2h was prepared following the method described by Bin and You.[13)]



2i

7-(Methoxymethoxy)-2-naphthol (2i)[15)]

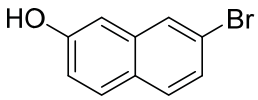
2i was prepared following the method described by Takizawa and Sasai.[15)]



2j

7-(Allyloxy)-2-naphthol (2j)[15)]

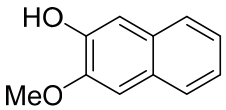
2j was prepared following the method described by Takizawa and Sasai.[15)]



2k

7-Bromo-2-naphthol (2j)[13)]

2k was prepared following the method described by Bin and You.[13)]

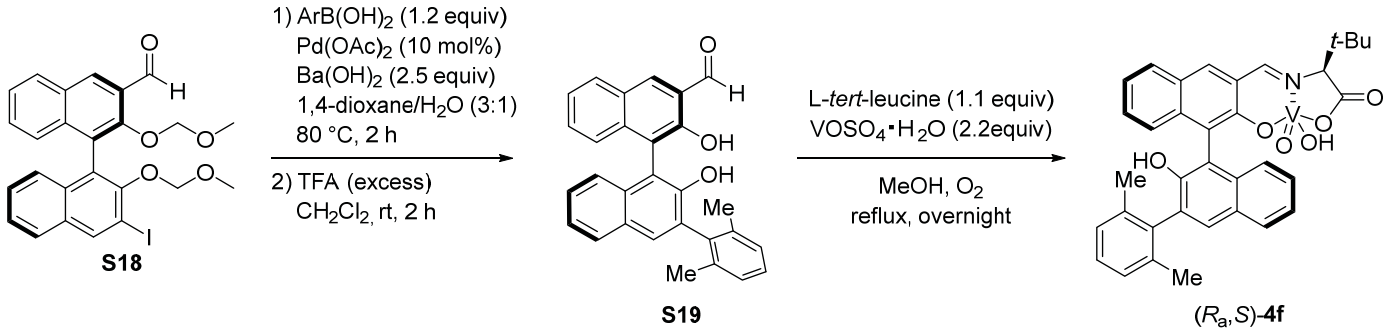


2l

3-Methoxy-2-naphthol (2l)[13)]

2l was prepared following the method described by Bin and You.[13)]

Preparation of mononuclear vanadium complexes



(*R*)-3'-(2,6-Dimethylphenyl)-2,2'-dihydroxy-[1,1'-binaphthalene]-3-carbaldehyde (S19)

A suspension of **S18**[16] (250.0 mg, 0.47 mmol), (2,6-dimethylphenyl)boronic acid (78.1 mg, 0.52 mmol), $Pd(PPh_3)_4$ (54.7 mg, 0.047 mmol) and $Ba(OH)_2$ (202.7 mg, 1.18 mmol) in degassed water:1,4-dioxane (1/3, 4.7 mL) was stirred for 3 h at 80 °C. After cooling, the reaction mixture was then filtered through a short pad of silica gel and the solvent was evaporated. The crude product was purified by silica column chromatography to give coupling product. A subsequent deprotection of methoxymethyl group was performed. Trifluoroacetic acid (excess) was added to a solution of coupling product in CH_2Cl_2 (4.7 mL) at room temperature. The mixture was stirred for 2 h. After concentrated, the reaction mixture was purified by silica column chromatography to afford **S19** in 77% yield in 2 steps as a yellow solid.

^{1}H NMR (400 MHz, $CDCl_3$): δ 10.54 (s, 1H), 10.20 (s, 1H), 8.35 (s, 1H), 8.02-7.99 (m, 1H), 7.87 (d, J = 7.8 Hz, 1H), 7.74 (s, 1H), 7.47-7.17 (m, 10H), 4.41 (s, 1H), 2.18 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 196.6, 154.0, 149.0, 138.5, 137.9, 137.7, 137.4, 135.4, 133.3, 130.8, 130.0, 129.7, 129.2, 129.1, 128.3, 128.2, 127.8, 127.7, 126.5, 125.0, 124.5, 123.6, 122.1, 116.8, 113.9, 20.6.

HRMS (APCI): calcd for $C_{29}H_{23}O_3$: *m/z* 419.1647 $[M + H]^+$, found 419.1632.

$[\alpha]_D^{26}$ +50.93 (*c* 0.30, $CHCl_3$)

Mononuclear vanadium complex (R_a,*S*)-**4f**

A round-bottomed flask was charged with **S14** (152.2 mg, 0.36 mmol), L-*tert*-leucine (52.5 mg, 0.40 mmol), $VOSO_4$•xH_2O (144.8 mg, 0.80 mmol), MS 3A (360 mg), MeOH (9.1 mL) under O_2 (balloon). The reaction mixture was refluxed, and the consumption of **S14** was monitored by TLC. The resulting solution was gradually cooled down to r.t. and filtered through Celite to remove MS 3A. The filtrate was evaporated, and the resulting black solid was dissolved in CH_2Cl_2 and washed with H_2O. The organic phase was dried over anhydrous Na_2SO_4 and concentrated in vacuum to give desired vanadium complex (R_a,*S*)-**4f** in 73% yield as a dark green solid.

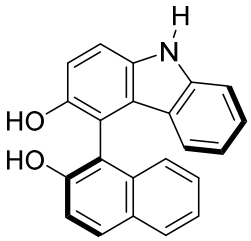
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.90 (s, 1H), 8.43 (s, 1H), 7.97 (s, 1H), 7.80-7.78 (m, 1H), 7.59-7.58 (m, 1H), 7.32-7.10 (m, 9H), 4.22 (s, 1H), 2.12 (s, 6H), 1.22 (s, 9H).

^{13}C NMR (100 MHz, CD_3OD): δ 180.6, 169.2, 151.9, 140.2, 139.7, 139.3, 139.24, 139.18, 139.0, 136.0, 132.4, 131.7, 131.5, 131.4, 130.6, 129.8, 129.4, 129.2, 127.97, 127.95, 127.3, 127.0, 126.1, 125.2, 118.9, 118.4, 85.0, 39.4, 29.1, 21.9.

HRMS (ESI): calcd for $C_{36}H_{34}NNaO_6V$: *m/z* 650.1723 $[M + OMe - OH + Na]^+$, found 650.1710.

Enantioselective oxidative hetero-coupling of 3-hydroxycarbazole and 2-naphthol catalyzed by vanadium(V) complex

A test tube was charged with 3-hydroxycarbazole **1** (1.0 equiv), 2-naphthol **2** (1.0 equiv), mononuclear vanadium catalyst (10 mol%), lithium chloride (3.0 equiv) and 1,4-dioxane (0.1 M for **1**) under air at 30 ℃. After stirring for 96 h, the reaction mixture was then filtered through a short pad of silica gel and the solvent was evaporated. The crude product was purified by silica gel column chromatography to afford hetero-coupling product.



3aa

4-(2-Hydroxynaphthalen-1-yl)-9*H*-carbazol-3-ol (3aa)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (s, 1H), 8.03 (d, J = 8.7 Hz, 1H), 7.93 (d, J = 8.2 Hz, 1H), 7.53 (d, J = 8.7 Hz, 1H), 7.44-7.25 (m, 7H), 6.77 (t, J = 7.3 Hz, 1H), 6.66 (d, J = 7.8 Hz, 1H), 5.26 (s, 1H), 4.72 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.3, 148.2, 140.4, 134.6, 132.8, 131.3, 129.4, 128.4, 127.5, 126.0, 124.1, 124.0, 122.6, 122.4, 121.4, 119.3, 117.8, 115.0, 112.6, 111.8, 110.7, 110.5.

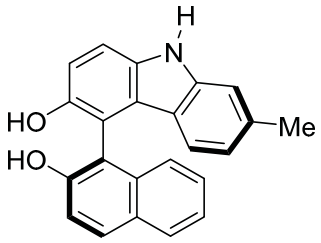
HRMS (APCI): calcd for $C_{22}H_{16}NO_2$: *m/z* 326.1181 $[M + H]^+$, found 326.1176.

IR (KBr): 3409, 3059, 2926, 1620, 1499, 1442, 1276, 1168, 1147, 898, 817, 748 cm^{-1}.

mp: 113-115 °C (white solid).

Enantiomeric ratio: 94:6, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 16.8 min, second peak: t_R = 20.4 min.

$[\alpha]_D^{20}$ –82.90 (*c* 0.18, $CHCl_3$) for 94:6 er.



3ba

4-(2-Hydroxynaphthalen-1-yl)-7-methyl-9*H*-carbazol-3-ol (3ba)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, J = 9.2 Hz, 1H), 7.99 (s, 1H), 7.92 (d, J = 8.2 Hz, 1H), 7.49 (d, J = 8.7 Hz, 1H), 7.42 (d, J = 8.7 Hz, 1H), 7.39-7.23 (m, 4H), 7.17 (s, 1H), 6.59 (dd, J = 8.2, 0.9 Hz, 1H), 6.52 (d, J = 8.2 Hz, 1H), 5.25 (s, 1H), 4.68 (s, 1H), 2.38 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.3, 148.2, 140.9, 136.3, 134.6, 132.8, 131.3, 129.4, 128.4, 127.5, 124.2, 124.0, 122.8, 121.0, 120.1, 117.8, 114.4, 112.5, 111.9, 110.6, 110.4, 21.9 (one carbon overlapped).

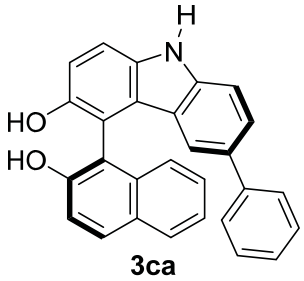
HRMS (APCI): calcd for $C_{23}H_{18}NO_2$: *m/z* 340.1338 $[M + H]^+$, found 340.1327.

IR (KBr): 3503, 3408, 3060, 2923, 1621, 1496, 1442, 1171, 810, 751 cm^{-1}.

mp: 128-130 °C (white solid).

Enantiomeric ratio: 92:8, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 19.2 min, second peak: t_R = 26.1 min.

$[\alpha]_D^{20}$ –41.54 (*c* 0.72, $CHCl_3$) for 92:8 er.



3ca

4-(2-Hydroxynaphthalen-1-yl)-6-phenyl-9*H*-carbazol-3-ol (3ca)

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.27 (s, 1H), 8.05 (s, 1H), 8.03 (d, *J* = 8.7 Hz, 1H), 7.98 (d, *J* = 7.8 Hz, 1H), 7.89 (s, 1H), 7.56-7.42 (m, 4H), 7.36-7.10 (m, 9H), 6.79 (s, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$: δ 153.7, 149.7, 142.5, 141.0, 135.8, 134.7, 131.2, 130.2, 129.7, 129.0, 128.5, 126.8, 126.7, 126.4, 125.3, 124.6, 124.2, 123.8, 123.4, 120.5, 119.2, 116.1, 116.0, 114.8, 112.0, 111.3.

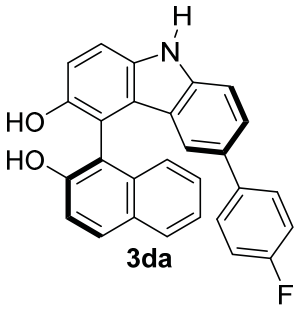
HRMS (APCI): calcd for $C_{28}H_{20}NO_2$: *m/z* 402.1494 $[M + H]^+$, found 402.1482.

IR (KBr): 3501, 3410, 3057, 1498, 1469, 1463, 1291, 1269, 1163, 816, 758 cm^{-1}.

mp: 152-154 °C (white solid).

Enantiomeric ratio: 90:10, determined by HPLC (Chiralpak OD-H, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 21.9 min, second peak: t_R = 38.8 min.

$[\alpha]_D^{21}$ –119.24 (*c* 0.79, Ethyl acetate) for 90:10 er.



3da

6-(4-Fluorophenyl)-4-(2-hydroxynaphthalen-1-yl)-9*H*-carbazol-3-ol (3da)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (s, 1H), 8.03 (d, *J* = 9.2 Hz, 1H), 7.94 (d, *J* = 8.2 Hz, 1H), 7.55 (d, *J* = 8.7 Hz, 1H), 7.47-7.36 (m, 5H), 7.33-7.30 (m, 2H), 7.04-6.99 (m, 2H), 6.97-6.92 (m, 2H), 6.73 (d, *J* = 1.37 Hz, 1H), 5.30 (s, 1H), 4.80 (s, 1H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 161.8 (d, $^1J_{C\text{-}F}$ = 244.2 Hz), 152.3, 148.3, 139.8, 137.6, 135.1, 132.9, 131.5, 131.4, 129.4, 128.4, 128.1 (d, $^3J_{C\text{-}F}$ = 7.2 Hz), 127.6, 125.2, 124.2, 124.1, 123.0, 122.7, 119.9, 117.7, 115.4, 115.3 (d, $^2J_{C\text{-}F}$ = 21.7 Hz), 112.8, 111.8, 111.0, 110.7.

^{19}F NMR (565 MHz, $CDCl_3$): δ –117.6.

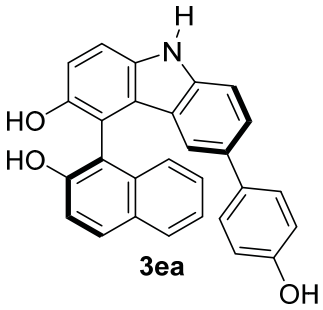
HRMS (APCI): calcd for $C_{28}H_{19}FNO_2$: *m/z* 420.1400 $[M + H]^+$, found 420.1389.

IR (KBr): 3413, 3060, 2925, 1513, 1498, 1471, 1231, 1161, 814 cm^{-1}.

mp: 154-156 °C (white solid).

Enantiomeric ratio: 93:7, determined by HPLC (Chiralpak IB, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 21.0 min, second peak: t_R = 27.5 min.

$[\alpha]_D^{19}$ –133.33 (*c* 0.83, $CHCl_3$) for 93:7 er.



3ea

4-(2-Hydroxynaphthalen-1-yl)-6-(4-hydroxyphenyl)-9*H*-carbazol-3-ol (3ea)

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.19 (s, 1H), 8.20 (s, 1H), 8.02 (d, *J* = 9.2 Hz, 1H), 7.97 (d, *J* = 8.7 Hz, 1H), 7.85 (s, 1H), 7.53 (d, *J* = 8.7 Hz, 1H), 7.45-7.43 (m, 3H), 7.37-7.30 (m, 3H), 7.25-7.20 (m, 2H), 6.96-6.93 (m, 2H), 6.77-6.73 (m, 2H), 6.70 (s, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 156.4, 153.6, 149.5, 140.5, 135.7, 134.7, 133.9, 131.4, 130.1, 129.6, 128.4, 127.8, 126.6, 125.2, 124,2, 124.1, 123.8, 123.3, 119.8, 119.1, 116.0, 115.9, 115.8, 114.7, 111.8, 111.0.

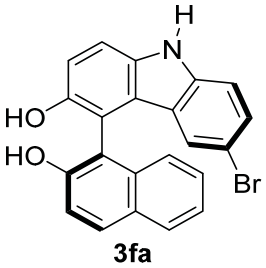
HRMS (APCI): calcd for $C_{28}H_{20}NO_3$: *m/z* 418.1443 $[M + H]^+$, found 418.1439.

IR (KBr): 3408, 2925, 1704, 1469, 1269, 1237, 1269, 1172, 811 cm^{-1}.

mp: 165-167°C (white solid).

Enantiomeric ratio: 92:8, determined by HPLC (Chiralpak IC-3, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 10.5 min, second peak: t_R = 16.1 min.

$[\alpha]_D^{23}$ –72.48 (*c* 0.63, EtOAc) for 92:8 er.



3fa

6-Bromo-4-(2-hydroxynaphthalen-1-yl)-9*H*-carbazol-3-ol (3fa)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (s, 1H), 8.06 (d, *J* = 9.2 Hz, 1H), 7.94 (d, *J* = 7.8 Hz, 1H), 7.53 (d, *J* = 8.7 Hz, 1H), 7.45 (d, *J* = 9.2 Hz, 1H), 7.40 (ddd, *J* = 8.0, 6.4, 1.6 Hz, 1H), 7.36-7.22 (m, 5H), 6.72 (d, *J* = 1.8 Hz, 1H), 5.19 (s, 1H), 4.74 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.3, 148.5, 138.9, 135.0, 132.6, 131.7, 129.4, 128.8, 128.6, 127.7, 124.2, 124.1, 124.0, 123.9, 121.6, 117.8, 116.0, 112.8, 112.0, 111.9, 111.1, 110.9.

HRMS (APCI): calcd for $C_{22}H_{15}NO_2$: *m/z* 325.1103 $[M – Br + H]^+$, found 325.1096.

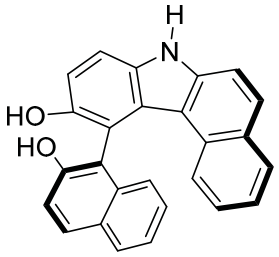
IR (KBr): 3412, 3060, 2925, 1499, 1448, 1286, 1275, 1165, 1147, 1045, 808 cm^{-1}.

mp: 122-124 °C (white solid).

Enantiomeric ratio: 89:11, determined by HPLC (Chiralpak IE, *n*-hexane:2-propanol = 9/1; flow rate 1.0 mL/min; 25 °C;

230 nm) first peak: t_R = 12.5 min, second peak: t_R = 16.2 min.

$[\alpha]_D^{24}$ –43.17 (*c* 0.63, $CHCl_3$) for 89:11 er.



3ga

11-(2-Hydroxynaphthalen-1-yl)-7*H*-benzo[*c*]carbazol-10-ol (3ga)

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.87 (s, 1H), 8.11 (d, *J* = 9.2 Hz, 1H), 7.96 (d, *J* = 8.2 Hz, 1H), 7.87 (s, 1H), 7.78-7.70 (m, 4H), 7.44 (d, *J* = 8.7 Hz, 1H), 7.30 (d, *J* = 8.7 Hz, 2H), 7.25 (ddd, *J* = 8.4, 6.9, 1.4 Hz, 1H), 7.15 (ddd, *J* = 8.4 , 6.9, 0.9 Hz, 1H), 7.08 (s, 1H), 7.05 (ddd, *J* = 8.2, 6.9, 0.9 Hz, 1H), 6.76 (d, *J* = 8.7 Hz, 1 H), 6.60 (ddd, *J* = 8.7, 6.9, 1.8 Hz, 1H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 154.3, 150.0, 139.4, 135.8, 134.8, 130.4, 129.6, 129.5, 128.9, 128.4, 128.0, 126.71, 125.66, 125.7, 125.6, 125.5, 123.4, 122.1, 119.14, 119.11, 116.6, 114.9, 113.8, 113.6, 112.8. (one carbon overlapped).

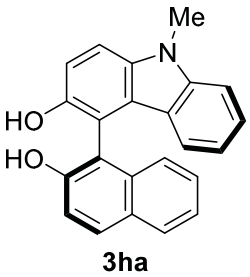
HRMS (APCI): calcd for $C_{26}H_{18}NO_2$: *m/z* 376.1338 $[M + H]^+$, found 376.1334.

IR (KBr): 3510, 3491, 3400, 3062, 1619, 1389, 1344, 1195, 1151, 816, 753, 505 cm^{-1}.

mp: 268-270 °C (white solid).

Enantiomeric ratio: 87:13, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 40.4 min, second peak: t_R = 46.8 min.

$[\alpha]_D^{24}$ –9.48 (*c* 0.68, EtOAc) for 87:13 er.



3ha

4-(2-Hydroxynaphthalen-1-yl)-9-methyl-9*H*-carbazol-3-ol (3ha)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, *J* = 9.2 Hz, 1H), 7.92 (d, *J* = 8.2 Hz, 1H), 7.52 (d, *J* = 8.7 Hz, 1H), 7.43 (d, *J* = 9.2 Hz, 1H), 7.37-7.30 (m, 6H), 6.77 (ddd, *J* = 8.0, 6.2, 1.8 Hz, 1H), 6.69 (d, *J* = 8.0 Hz, 1H), 5.22 (s, 1H), 4.69 (s, 1H), 3.91 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.3, 147.9, 141.6, 136.3, 132.8, 131.3, 129.4, 128.4, 127.5, 125.9, 124.1, 124.0, 121.8, 121.6, 121.4, 118.7, 117.8, 114.8, 111.9, 110.8, 110.4, 108.3, 29.2.

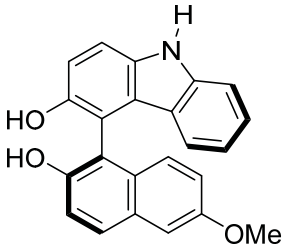
HRMS (APCI): calcd for $C_{23}H_{18}NO_2$: *m/z* 340.1338 $[M + H]^+$, found 340.1331.

IR (KBr): 3506, 3479, 3398, 3066, 2927, 1496, 1466, 1238, 1204, 1173, 1145, 806, 747 cm^{-1}.

mp: 145-147 °C (white solid).

Enantiomeric ratio: 74:26, determined by HPLC (Chiralpak IC-3, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 9.7 min, second peak: t_R = 16.6 min.

$[\alpha]_D^{19}$ –3.30 (*c* 0.58, $CHCl_3$) for 74:26 er.



3ab

4-(2-Hydroxy-6-methoxynaphthalen-1-yl)-9*H*-carbazol-3-ol (3ab)

^{1}H NMR (400 MHz, $(CD_3)_2CO$): δ 10.20 (s, 1H), 7.91 (d, *J* = 8.7 Hz, 1H), 7.56 (s, 1H), 7.51 (d, *J* = 8.2 Hz, 1H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.39-7.36 (m, 2H), 7.26-7.14 (m, 4H), 6.90 (dd, *J* = 9.2, 1.8 Hz, 1H), 6.64 (t, *J* = 7.8 Hz, 1H), 6.58 (d, *J* = 7.8 Hz, 1H), 3.91 (s, 3H).

^{13}C NMR (100 MHz, $(CD_3)_2CO$): δ 156.4, 151.9, 149.5, 141.5, 135.3, 130.5, 129.8, 128.9, 126.7, 125.4, 123.7, 122.0, 119.5, 119.1, 118.3, 116.2, 115.8, 114.7, 111.8, 110.9, 106.9, 78.9, 55.1.

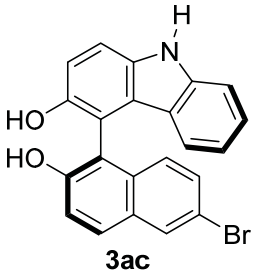
HRMS (APCI): calcd for $C_{23}H_{18}NO_3$: *m/z* 356.1287 $[M + H]^+$, found 356.1279.

IR (KBr): 3395, 3003, 2945, 1605, 1503, 1345, 1231, 1157, 810, 751, 604 cm^{-1}.

mp: 131-133°C (brown solid).

Enantiomeric ratio: 87:13, determined by HPLC (Chiralpak IC-3, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 10.3 min, second peak: t_R = 13.5 min.

$[\alpha]_D^{19}$–19.87 (*c* 0.77, $CHCl_3$) for 87:13 er.



3ac

4-(6-Bromo-2-hydroxynaphthalen-1-yl)-9*H*-carbazol-3-ol (3ac)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (s, 1H), 8.08 (d, *J* = 2.3 Hz, 1H), 7.94 (d, *J* = 8.7 Hz, 1H), 7.54 (d, *J* = 8.9 Hz, 1H), 7.45 (d, *J* = 8.9 Hz, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.34 (dd, *J* = 8.8, 2.3 Hz, 1H), 7.30-7.26 (m, 2H), 7.19 (d, *J* = 8.8 Hz, 1H), 6.78 (ddd, *J* = 8.0, 7.1, 0.9 Hz, 1H), 6.60 (d, *J* = 7.8 Hz, 1H), 5.28 (s, 1H), 4.64 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.6, 148.1, 140.4, 134.6, 131.5, 130.7, 130.5, 130.4, 126.13, 126.07, 122.5, 122.2, 121.2, 119.5, 119.0, 117.9, 115.2, 112.9, 112.3, 110.6, 110.1 (one carbon overlapped).

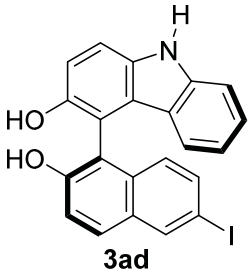
HRMS (APCI): calcd for $C_{22}H_{15}BrNO_2$: *m/z* 404.0286 $[M + H]^+$, found 404.0281.

IR (KBr): 3410, 3059, 2928, 1588, 1499, 1441, 1274, 1340, 1164, 899, 810, 749 cm^{-1}.

mp: 117-119 °C (white solid).

Enantiomeric ratio: 89:11, determined by HPLC (Chiralpak IC-3, *n*-hexane:2-propanol = 9/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 14.2 min, second peak: t_R = 22.5 min.

$[\alpha]_D^{19}$ –99.33 (*c* 0.75, $CHCl_3$) for 89:11 er.



3ad

4-(2-Hydroxy-6-iodonaphthalen-1-yl)-9*H*-carbazol-3-ol (3ad)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.30 (d, J = 1.8 Hz, 1H), 8.13 (s, 1H), 7.91 (d, J = 8.7 Hz, 1H), 7.54 (d, J = 8.7 Hz, 1H), 7.50 (dd, J = 8.7, 1.8 Hz, 1H), 7.43 (d, J = 9.2 Hz, 1H) , 7.39 (d, J = 8.2 Hz, 1H), 7.30-7.28 (m, 2H), 7.06 (d, J = 8.7 Hz, 1H), 6.78 (ddd, J = 8.3, 7.3, 0.9 Hz, 1H), 6.60 (d, J = 8.2 Hz, 1H), 5.32 (s, 1H), 4.66 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.7, 148.1, 140.4, 136.9, 135.9, 134.6, 131.8, 131.1, 130.2, 126.12, 126.06, 122.6, 122.3, 121.2, 119.4, 118.8, 115.2, 112.8, 112.4, 110.6, 110.1, 89.0.

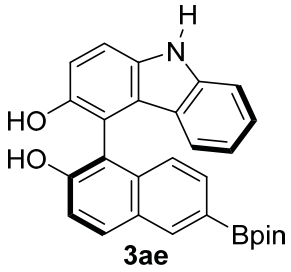
HRMS (APCI): calcd for $C_{22}H_{15}INO_2$: *m/z* 452.0147 $[M + H]^+$, found 452.0128.

IR (KBr): 3410, 3058, 2953, 1581, 1500, 1442, 1338, 1276, 1150, 898, 816 cm^{-1}.

mp: 133-135 °C (white solid).

Enantiomeric ratio: 85:15, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 14.9 min, second peak: t_R = 26.0 min.

$[\alpha]_D^{23}$ –83.71 (*c* 0.89, $CHCl_3$) for 85:15 er.



3ae

4-(2-Hydroxy-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)naphthalen-1-yl)-9*H*-carbazol-3-ol (3ae)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (s, 1H), 8.11 (s, 1H), 8.07 (d, J = 8.7 Hz, 1H), 7.65 (dd, J = 8.4, 1.4 Hz, 1H), 7.53 (d, J = 8.9 Hz, 1H), 7.43 (d, J = 9.2 Hz, 1H), 7.37 (d, J = 8.9 Hz, 1H), 7.31-7.23 (m, 3H), 6.75(ddd, J = 8.3, 7.8, 0.9 Hz, 1H), 6.61 (d, J = 8.3 Hz, 1H), 5.32 (s, 1H), 4.69 (s, 1H), 1.36 (s, 12H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 153.3, 148.2, 140.4, 136.7, 134.7, 134.6, 132.3, 132.2, 128.8, 126.0, 123.2, 122.7, 122.5, 121.4, 119.4, 117.7, 115.1, 112.6, 111.9, 110.6, 110.5, 83.9, 24.9 (one carbon overlapped).

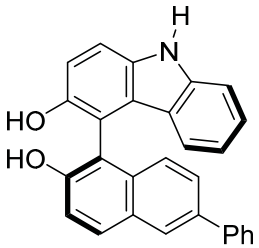
HRMS (APCI): calcd for $C_{28}H_{27}BNO_4$: *m/z* 452.2033 $[M + H]^+$, found 452.2033.

IR (KBr): 3351, 3212, 2980, 1719, 1482, 1373, 1333, 1297, 1256, 1171, 1145, 1045, 849, 738 cm^{-1}.

mp: 118-120 °C (brown solid).

Enantiomeric excess: 93:7, determined by HPLC (Chiralpak IC-3, hexane/2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 10.8 min, second peak: t_R = 15.7 min.

$[\alpha]_D^{20}$ –74.45 (*c* 0.87, $CHCl_3$) for 93:7 er.



3af

4-(2-Hydroxy-6-phenylnaphthalen-1-yl)-9*H*-carbazol-3-ol (3af)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13-8.08 (m, 3H), 7.67 (dd, J = 8.7, 0.9 Hz, 2H), 7.58-7.54 (m, 2H), 7.48-7.28 (m, 8H), 6.80 (ddd, J = 8.0, 7.3, 0.9 Hz, 1H), 6.73 (d, J = 8.0 Hz, 1H), 5.28 (s, 1H), 4.74 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 152.4, 148.2, 140.8, 140.4, 136.8, 134.6, 132.0, 131.6, 129.7, 128.8, 127.20, 127.15, 126.3, 126.1, 124.7, 122.6, 122.4, 121.4, 119.4, 118.2, 115.1, 112.7, 111.8, 110.6, 110.5 (one carbon overlapped).

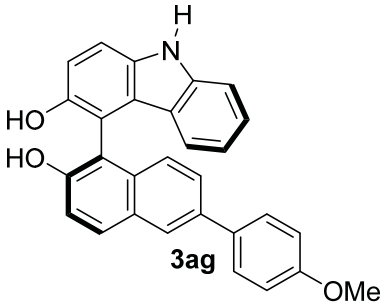
HRMS (APCI): calcd for $C_{28}H_{20}NO_2$: *m/z* 402.1494 $[M + H]^+$, found 402.1489.

IR (KBr): 3410, 3058, 1707, 1596, 1494, 1286, 1258, 1151, 901, 757 cm^{-1}.

mp: 134-136 °C (white solid).

Enantiomeric ratio: 89:11, determined by HPLC (Chiralpak IC-3, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 255 nm) first peak: t_R = 7.5 min, second peak: t_R = 10.2 min.

$[\alpha]_D^{18}$ –94.15 (*c* 1.06, $CHCl_3$) for 89:11 er.



3ag

4-(2-Hydroxy-6-(4-methoxyphenyl)naphthalen-1-yl)-9*H*-carbazol-3-ol (3ag)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12 (s, 1H), 8.08-8.06 (m, 2H), 7.60 (d, J = 8.7 Hz, 2H), 7.56-7.52 (m, 2H), 7.45 (d, J = 8.7 Hz, 1H), 7.40-7.37 (m, 2H), 7.31-7.27 (m, 2H), 7.00 (d, J = 9.2 Hz, 2H), 6.79 (ddd, J = 8.2, 7.8, 0.9 Hz, 1H), 6.73 (d, J = 8.2 Hz, 1H), 5.25 (s, 1H), 4.75 (s, 1H), 3.86 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 152.2, 148.2, 140.4, 136.4, 134.6, 133.4, 131.7, 131.5, 129.7, 128.2, 127.0, 126.0, 125.6, 124.7, 122.6, 122.4, 121.4, 119.4, 118.2, 115.1, 114.3, 112.6, 111.8, 110.7, 110.5, 55.4.

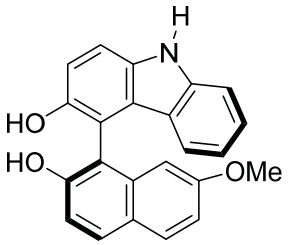
HRMS (APCI): calcd for $C_{29}H_{22}NO_3$: *m/z* 432.1600 $[M + H]^+$, found 432.1588.

IR (KBr): 3410, 3052, 2931, 1607, 1503, 1282, 1248, 1179, 1152, 821 cm^{-1}.

mp: 115-117 °C (brown solid).

Enantiomeric ratio: 78:22, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 32.5 min, second peak: t_R = 42.2 min.

$[\alpha]_D^{27}$ –45.86 (*c* 0.70, $CHCl_3$) for 78:22 er.



3ah

4-(2-Hydroxy-7-methoxynaphthalen-1-yl)-9*H*-carbazol-3-ol (3ah)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (s, 1H), 7.94 (d, *J* = 8.7 Hz, 1H), 7.82 (d, *J* = 9.2 Hz, 1H), 7.53 (d, *J* = 8.2 Hz, 1H), 7.38 (d, *J* = 8.2 Hz, 1H), 7.30-7.25 (m, 3H), 7.03 (dd, *J* = 8.9, 2.8 Hz, 1H), 6.80 (td, *J* = 8.1, 0.9 Hz, 1H), 6.75 (d, *J* = 7.3 Hz, 1H), 6.63 (d, *J* =2.3 Hz, 1H), 5.21 (s, 1H), 4.76 (s, 1H), 3.55 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 153.0, 148.2, 140.4, 134.7, 134.3, 131.1, 123.0, 126.0, 124.7, 122.5, 121.4, 119.4, 116.1, 115.2, 115.0, 112.6, 111.0, 110.8, 110.5, 103.1, 55.1 (one carbon overlapped).

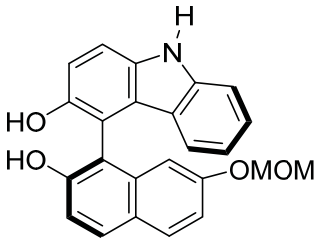
HRMS (APCI): calcd for $C_{23}H_{18}NO_3$: *m/z* 356.1287 $[M + H]^+$, found 356.1270.

IR (KBr): 3479, 3408, 2927, 1622, 1513, 1442, 1273, 1221, 1165, 831 cm^{-1}.

mp: 116-118 °C (brown solid).

Enantiomeric ratio: 86:14, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 15.6 min, second peak: t_R = 20.7 min.

$[\alpha]_D^{25}$ –39.36 (*c* 0.78, $CHCl_3$) for 86:14 er.



3ai

4-(2-Hydroxy-7-(methoxymethoxy)naphthalen-1-yl)-9*H*-carbazol-3-ol (3ai)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (s, 1H), 7.95 (d, *J* = 9.2 Hz, 1H), 7.85 (d, *J* = 9.2 Hz, 1H), 7.51 (d, *J* = 6.9 Hz, 1H), 7.37 (d, *J* = 8.2 Hz, 1H), 7.30-7.24 (m, 3H), 7.16 (dd, *J* = 8.7, 2.3 Hz, 1H), 6.83 (d, *J* = 2.3 Hz, 1H), 6.79 (ddd, *J* = 8.2, 7.3, 0.9 Hz, 1H), 6.72 (d, *J* = 8.2 Hz, 1H), 5.24 (s, 1H), 4.97 (s, 2H), 4.73 (s, 1H), 3.26 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 156.5, 152.9, 148.2, 140.4, 134.7, 134.2, 131.1, 130.1, 125.9, 125.3, 122.5, 121.5, 119.3, 116.0, 115.8, 115.1, 112.6, 111.1, 110.7, 110.4, 107.8, 94.4, 56.0 (one carbon overlapped)

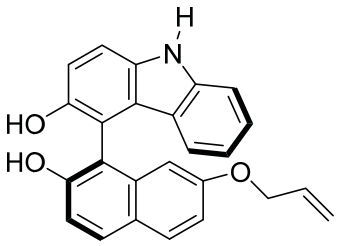
HRMS (APCI): calcd for $C_{23}H_{16}NO_3$: *m/z* 354.1125 $[M - OMe]^+$, found 354.1128.

IR (KBr): 3409, 2932, 1622, 1514, 1455, 1290, 1269, 1156 cm^{-1}.

mp: 170-172 °C (brown solid).

Enantiomeric ratio: 92:8, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 15.0 min, second peak: t_R = 19.9 min.

$[\alpha]_D^{21}$ –86.75 (*c* 0.80, $CHCl_3$) for 92:8 er.



3aj

4-(7-(Allyloxy)-2-hydroxynaphthalen-1-yl)-9*H*-carbazol-3-ol (3aj)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (s, 1H), 7.94 (d, J = 9.2 Hz, 1H), 7.82 (d, J = 8.7 Hz, 1H), 7.53 (d, J = 8.7 Hz, 1H), 7.38 (d, J = 8.2 Hz, 1H), 7.23-7.25 (m, 3H), 7.05 (dd, J = 8.9, 2.3 Hz, 1H), 6.79 (ddd, J = 7.8, 6.9, 0.9 Hz, 1H), 6.73 (d, J = 7.8 Hz, 1H), 6.64 (d, J = 2.8 Hz, 1H), 5.87-5.77 (m, 1H), 5.21 (s, 1H), 5.15-5.05 (m, 2H), 4.75 (s, 1H), 4.31-4.19 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 158.0, 153.0, 148.2, 140.4, 134.6, 134.3, 132.8, 131.0, 123.0, 126.0, 124.7, 122.49, 122.45, 121.4, 119.4, 118.0, 116.4, 115.2, 115.0, 112.5, 111.0, 110.8, 110.4, 104.4, 68.6.

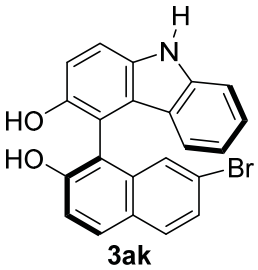
HRMS (APCI): calcd for $C_{25}H_{20}NO_3$: *m/z* 382.1443 $[M + H]^+$, found 382.1429.

IR (KBr): 3491, 3409, 2925, 1620, 1512, 1441, 1190, 834 cm^{-1}.

mp: 101-103 °C (brown solid).

Enantiomeric ratio: 91:9, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 12.5 min, second peak: t_R = 19.2 min.

$[\alpha]_D^{25}$ –88.21 (*c* 0.87, $CHCl_3$) for 91:9 er.



3ak

4-(7-Bromo-2-hydroxynaphthalen-1-yl)-9*H*-carbazol-3-ol (3ak)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 1H), 7.99 (d, J = 8.7 Hz, 1H), 7.78 (d, J = 9.2 Hz, 1H), 7.55 (d, J = 8.7 Hz, 1H), 7.46-7.39 (m, 4H), 7.30-7.26 (m, 2H), 6.79 (ddd, J = 8.7, 7.8, 0.9 Hz, 1H), 6.63 (d, J = 7.8 Hz, 1H), 5.29 (s, 1H), 4.63 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 153.2, 148.2, 140.4, 134.7, 134.3, 131.3, 130.0, 127.8, 127.5, 126.2, 126.1, 122.5, 122.3, 122.2, 121.2, 119.4, 118.2, 115.3, 113.0, 111.5, 110.6, 109.9.

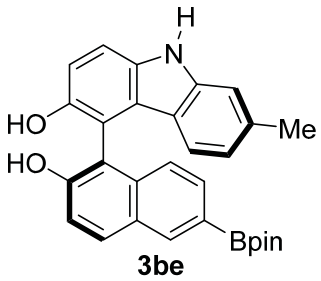
HRMS (APCI): calcd for $C_{22}H_{15}NO_2$: *m/z* 325.1103 $[M – Br + H]^+$, found 325.1096.

IR (KBr): 3411, 3058, 2926, 1614, 1499, 1441, 1286, 1164, 900, 836, 748 cm^{-1}.

mp: 126-128°C (white solid).

Enantiomeric ratio: 88:12, determined by HPLC (Chiralpak IC-3, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 230 nm) first peak: t_R = 10.2 min, second peak: t_R = 17.0 min.

$[\alpha]_D^{20}$ –78.76 (*c* 0.65, $CHCl_3$) for 88:12 er.



3be

4-(2-Hydroxy-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)naphthalen-1-yl)-7-methyl-9*H*-carbazol-3-ol (3be)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (s, 1H), 8.06 (d, *J* = 8.7 Hz, 1H), 7.99 (s, 1H), 7.65 (dd, *J* = 8.5, 0.9 Hz, 1H), 7.49 (d, *J* = 8.7 Hz, 1H), 7.41 (d, *J* = 8.7 Hz, 1H), 7.30 (d, *J* = 8.2 Hz, 1H), 7.24 (d, *J* = 8.7 Hz, 1H), 7.16 (s, 1H), 6.57 (dd, *J* = 8.2, 0.9 Hz, 1H), 6.47 (d, *J* = 8.2 Hz, 1H), 5.33 (s, 1H), 4.67 (s, 1H), 2.37 (s, 3H), 1.36 (s, 12H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 153.3, 148.2, 140.9, 136.7, 136.3, 134.7, 134.6, 132.2, 132.1, 128.8, 123.3, 122.8, 121.0, 120.1, 117.7, 114.4, 112.5, 112.0, 110.6, 110.3, 83.9, 24.9, 21.9 (two carbons overlapped).

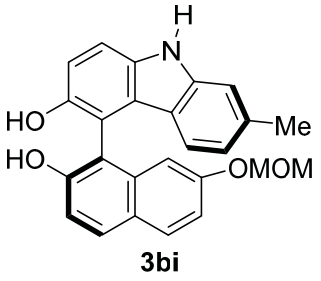
HRMS (APCI): calcd for $C_{29}H_{29}BNO_4$: *m/z* 466.2190 $[M + H]^+$, found 466.2182.

IR (KBr): 3404, 2921, 1622, 1514, 1442, 1153, 984, 809 cm^{-1}.

mp: 129-131 °C (brown solid).

Enantiomeric ratio: 87:13, determined by HPLC (Chiralpak IG-3, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 9.6 min, second peak: t_R = 16.7 min.

$[\alpha]_D^{26}$ –93.42 (*c* 0.76, $CHCl_3$) for 87:13 er.



3bi

4-(2-Hydroxy-7-(methoxymethoxy)naphthalen-1-yl)-7-methyl-9*H*-carbazol-3-ol (3bi)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (s, 1H), 7.94 (d, *J* = 9.2 Hz, 1H), 7.84 (d, *J* = 8.7 Hz, 1H), 7.48 (d, *J* = 8.7 Hz, 1H), 7.28 (d, *J* = 8.7 Hz, 1H), 7.23 (d, *J* = 8.7 Hz, 1H), 7.17-7.14 (m, 2H), 6.82 (d, *J* = 2.8 Hz, 1H), 6.63-6.57 (m, 2H), 5.27 (s, 1H), 4.98 (s, 2H), 4.72 (s, 1H), 3.28 (s, 3H), 2.39 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4, 152.9, 148.1, 140.9, 136.2, 134.6, 134.2, 130.9, 130.0, 125.3, 122.6, 121.1, 120.9, 120.2, 116.0, 115.8, 114.4, 112.5, 111.3, 110.5, 110.4, 107.8, 94.4, 56.0, 21.9.

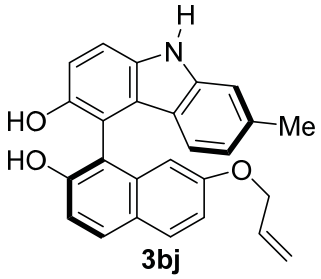
HRMS (APCI): calcd for $C_{25}H_{22}NO_4$: *m/z* 400.1549 $[M + H]^+$, found 400.1538.

IR (KBr): 3344, 3206, 2979, 1715, 1625, 1481, 1378, 1334, 1145, 806 cm^{-1}.

mp: 165-167 °C (brown solid).

Enantiomeric ratio: 88:12, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 20.2 min, second peak: t_R = 31.8 min.

$[\alpha]_D^{26}$ –77.84 (*c* 0.84, $CHCl_3$) for 88:12 er.



3bj

4-(7-(Allyloxy)-2-hydroxynaphthalen-1-yl)-7-methyl-9*H*-carbazol-3-ol (3bj)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (s, 1H), 7.93 (d, J = 8.7 Hz, 1H), 7.81 (d, J = 9.2 Hz, 1H), 7.49 (d, J = 8.7 Hz, 1H), 7.27-7.23 (m, 2H), 7.17 (s, 1H), 7.04 (dd, J = 8.9, 2.3 Hz, 1H), 6.64-6.58 (m, 3H), 5.88-5.78 (m, 1H), 5.22 (s, 1H), 5.16-5.06 (m, 2H), 5.73 (s, 1H), 4.30-4.19 (m, 2H), 2.39 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 158.0, 153.0, 148.1, 140.9, 136.3, 134.6, 134.3, 132.8, 130.9, 129.9, 124.7, 122.6, 121.1, 121.0, 120.2, 118.0, 116.4, 115.2, 114.4, 112.4, 111.1, 110.5, 104.4, 68.6, 21.9 (one carbon overlapped).

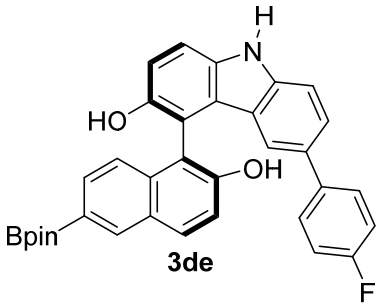
HRMS (APCI): calcd for $C_{26}H_{22}NO_3$: *m/z* 396.1600 $[M + H]^+$, found 396.1587.

IR (KBr): 3493, 3406, 2923, 1621, 1514, 1442, 1191, 834, 809 cm^{-1}.

mp: 102-104 °C (brown solid).

Enantiomeric ratio: 85:15, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 14.9 min, second peak: t_R = 26.0 min.

$[\alpha]_D^{27}$ –-103.45 (*c* 0.87, $CHCl_3$) for 85:15 er.



3de

6-(4-Fluorophenyl)-4-(2-hydroxy-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)naphthalen-1-yl)-9*H*-carbazol-3-ol (3de)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.46 (s, 1H), 8.13 (s, 1H), 8.07 (d, J = 8.7 Hz, 1H), 7.67 (dd, J = 8.5, 0.9 Hz, 1H), 7.55 (d, J = 8.7 Hz, 1 H), 7.46-7.39 (m, 3H), 7.35-7.30 (m, 2H), 7.01-6.91 (m, 4H), 6.69 (d, J = 1.4 Hz, 1H), 5.38 (s, 1H), 4.79 (s, 1H), 1.36 (d, J = 4.4 Hz, 12H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 161.8 (d, $^1J_{C\text{-}F}$ = 245.6 Hz), 153.4, 148.3, 139.7, 137.6 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 136.8, 135.1, 134.7, 132.3, 131.4, 128.8, 128.1 (d, $^3J_{C\text{-}F}$ = 8.7 Hz), 125.2, 123.4, 122.9, 122.7, 119.8, 117.7, 115.4, 115.3 (d, $^2J_{C\text{-}F}$ = 21.7 Hz), 112.8, 111.9, 110.9, 110.7, 83.9, 24.9 (two carbons overlapped).

^{19}F NMR (565 MHz, $CDCl_3$): δ –115.9.

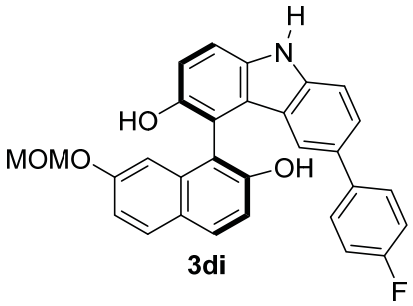
HRMS (APCI): calcd for $C_{34}H_{30}BFNO_4$: *m/z* 546.2252 $[M + H]^+$, found 546.2231.

IR (KBr): 3410, 2996, 1620, 1472, 1376, 1333, 1292, 1143, 810 cm^{-1}.

mp: 147-149 °C (brown solid).

Enantiomeric ratio: 89:11, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 11.3 min, second peak: t_R = 13.5 min.

$[\alpha]_D^{27}$ –221.46 (*c* 0.89, $CHCl_3$) for 89:11 er.



3di

6-(4-Fluorophenyl)-4-(2-hydroxy-7-(methoxymethoxy)naphthalen-1-yl)-9*H*-carbazol-3-ol (3di)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 1H), 7.95 (d, *J* = 8.7 Hz, 1H), 7.87 (d, *J* = 9.2 Hz, 1H), 7.53 (d, *J* = 8.7 Hz, 1H), 7.46 (dd, *J* = 8.2, 1.8 Hz, 1H), 7.41 (d, *J* = 8.7 Hz, 1H), 7.30 (d, *J* = 8.7 Hz, 1H), 7.29 (d, *J* = 8.7 Hz, 1H), 7.17 (dd, *J* = 8.9, 2.3 Hz, 1H), 7.07-7.03 (m, 2H), 6.98-6.93 (m, 2H), 6.87 (d, *J* = 2.8 Hz, 1H), 6.80 (d, *J* = 1.4 Hz, 1H), 5.34 (s, 1H), 4.96 (d, *J* = 2.3 Hz, 2H), 4.87 (s, 1H), 3.20 (s, 3H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 161.8 (d, $^1J_{C\text{-}F}$ = 245.6 Hz), 156.5, 152.9, 148.2, 139.7, 137.7 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 135.1, 134.2, 131.3, 131.2, 130.1, 128.1 (d, $^3J_{C\text{-}F}$ = 8.7 Hz), 125.3, 125.1, 123.0, 122.5, 120.0, 116.1, 115.8, 115.4, 115.3 (d, $^2J_{C\text{-}F}$ = 21.7 Hz), 112.8, 111.1, 111.0, 110.7, 107.9, 94.3, 55.8.

^{19}F NMR (565 MHz, $CDCl_3$): δ –115.9.

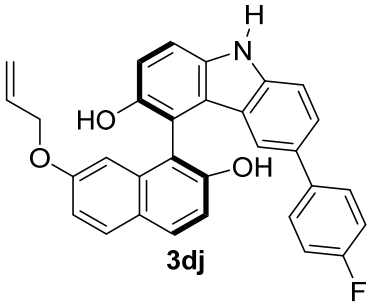
HRMS (APCI): calcd for $C_{30}H_{23}FNO_4$: *m/z* 480.1611 $[M + H]^+$, found 480.1593.

IR (KBr): 3409, 3070, 2925, 1621, 1513, 1231, 1220, 1160, 837, 811 cm^{-1}.

mp: 112-114 °C (brown solid).

Enantiomeric ratio: 83:17, determined by HPLC (Chiralpak IB, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 23.8 min, second peak: t_R = 29.5 min.

$[\alpha]_D^{23}$ –160.70 (*c* 1.15, $CHCl_3$) for 83:17 er.



3dj

4-(7-(Allyloxy)-2-hydroxynaphthalen-1-yl)-6-(4-fluorophenyl)-9*H*-carbazol-3-ol (3dj)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (s, 1H), 7.93 (d, *J* = 8.7 Hz, 1H), 7.83 (d, *J* = 8.7 Hz, 1H), 7.54 (d, *J* = 8.7 Hz, 1H), 7.47 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.41 (d, *J* = 8.2 Hz, 1H), 7.31 (d, *J* = 8.7 Hz, 1H), 7.26 (d, *J* = 8.7 Hz, 1H), 7.09-7.05 (m, 3H), 6.97-6.93 (m, 2H), 6.81 (d, *J* = 1.8 Hz, 1H), 6.67 (d, *J* = 2.3 Hz, 1H), 5.84-5.74 (m, 1H), 5.29 (s, 1H), 5.12-5.01 (m, 2H), 4.87 (s, 1H), 4.31-4.19 (m, 2H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 161.8 (d, $^1J_{C\text{-}F}$ = 244.2 Hz), 158.0, 153.0, 148.3, 139.7, 137.7 (d, $^4J_{C\text{-}F}$ = 2.9 Hz), 135.1, 134.3, 132.7, 131.4, 131.2, 130.0, 128.1 (d, $^3J_{C\text{-}F}$ = 7.2 Hz), 125.2, 124.7, 123.0, 122.5, 119.9, 118.0, 116.4, 115.3, 115.3 (d, $^2J_{C\text{-}F}$ = 21.7 Hz), 115.2, 112.7, 111.1, 110.9, 110.7, 104.6, 68.62.

^{19}F NMR (565 MHz, $CDCl_3$): δ –115.9.

HRMS (APCI): calcd for $C_{31}H_{23}FNO_3$: *m/z* 476.1662 $[M + H]^+$, found 476.1642.

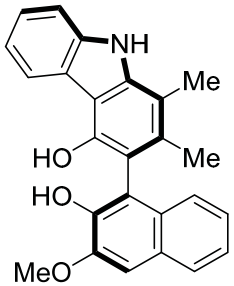
IR (KBr): 3406, 2957, 2925, 1622, 1513, 1227, 1173, 1159, 811 cm^{-1}.

mp: 101-103 °C (brown solid).

Enantiomeric ratio: 82:18, determined by HPLC (Chiralpak IB, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 220 nm) first peak: t_R = 20.4 min, second peak: t_R = 27.9 min.

$[\alpha]_D^{23}$ –58.52 (*c* 1.08, $CHCl_3$) for 82:18 er.

Enantioselective oxidative hetero-coupling of 4-hydroxycarbazole 1i and 2-naphthol 2l catalyzed by vanadium(V) complex



3il

A test tube was charged with 1,2-dimethyl-9*H*-carbazol-4-ol (**1i**) (1.0 equiv), 3-methoxynaphthalen-2-ol (**2l**) (2.0 equiv), mononuclear vanadium catalyst (10 mol%), lithium chloride (3.0 equiv) and 1,4-dioxane (0.1 M for **1i**) under air at 30 ℃. The mixture was stirred for 96 h. The reaction mixture was then filtered through a short pad of silica gel and the solvent was evaporated. The crude product was purified by silica gel column chromatography to afford hetero-coupling product **3il**.

3-(2-Hydroxy-3-methoxynaphthalen-1-yl)-1,2-dimethyl-9H-carbazol-4-ol (3il)

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.23 (d, *J* = 8.2 Hz, 1H), 8.01 (brs, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.46 (d, *J* = 8.2 Hz, 1H), 7.38-7.35 (m, 2H), 7.25 (m, 4H), 5.94 (brs, 1H), 5.05 (brs, 1H), 4.11 (s, 3H), 2.48 (s, 3H), 2.05 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 147.6, 144.6, 138.9, 134.0. 129.3, 129.2, 126.9, 125.0, 124.6, 124.5, 124.4, 123.4, 122.9, 119.7, 114.6, 110.4, 110.2, 109. 9, 109.3, 106.7, 56.0, 16.8, 13.6.

HRMS (APCI): calcd for $C_{25}H_{22}NO_3$: *m/z* 384.1594 $[M + H]^+$, found 384.1595.

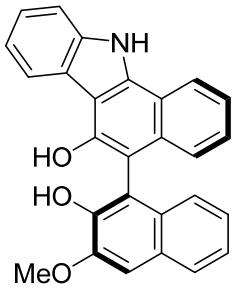
IR (KBr): 3403, 2922, 1737, 1524, 884, 743 cm^{-1}.

mp: 168-170 °C (brown solid).

Enantiomeric ratio: 95:5 er, determined by HPLC (Chiralpak IB, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 235 nm) first peak: t_R = 24.8 min, second peak: t_R = 29.7 min.

$[\alpha]_D^{15}$ –80.4 (*c* 1.6, $CHCl_3$) for 95:5 er.

Enantioselective oxidative hetero-coupling of 1j and 2-naphthol 2l catalyzed by vanadium(V) complex



3jl

A test tube was charged with 11H-benzo[*a*]carbazol-6-ol (**1j**) (1.0 equiv), 3-methoxynaphthalen-2-ol (**2l**) (2.0 equiv), mononuclear vanadium catalyst (10 mol%), lithium chloride (3.0 equiv) and 1,4-dioxane (0.1 M for **1j**) under air at 30 °C. The mixture was stirred for 96 h. The reaction mixture was then filtered through a short pad of silica gel and the solvent was evaporated. The crude product was purified by silica gel column chromatography to afford hetero-coupling product **3jl**.

5-(2-hydroxy-3-methoxynaphthalen-1-yl)-11*H*-benzo[*a*]carbazol-6-ol (3ji)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.95 (brs, 1H), 8.39 (d, J = 7.8 Hz, 1H), 8.16 (d, J = 7.8 Hz. 1H), 7.83 (d, J = 8.2 Hz, 1H), 7.63 (d, J = 8.2 Hz, 1H), 7.45 (d, J = 7.5 Hz, 2H), 7.38-7.18 (m, 7H), 5.95 (brs, 1H), 5.49 (brs, 1H), 4.14 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 149.1, 147.6, 145.4, 138.1, 137.0, 132.3, 129.5 x 2, 127.0, 126.2, 125.7, 125.1, 124.8, 124.7, 124.6, 123.4, 123.0 x 2, 120.7, 120.5, 117.5, 112.5, 110.6, 110.0, 107.2, 104.1, 56.1.

HRMS (ESI): calcd for $C_{27}H_{19}NO_3Na$: *m/z* 428.1257 $[M + Na]^+$, found 428.1251.

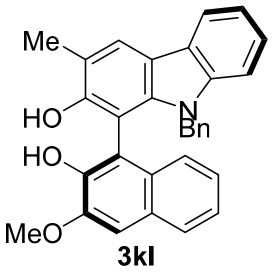
IR (KBr): 3411, 2921, 2847, 2375, 1459, 1255, 1125, 1073, 885, 746 cm^{-1}.

mp: 250 °C (decomp.) (black green solid).

Enantiomeric ratio: 91:9 er, determined by HPLC (Chiralpak IC, *n*-hexane:2-propanol = 3/1; flow rate 1.0 mL/min; 25 °C; 297 nm) first peak: t_R = 11.4 min, second peak: t_R = 19.0 min.

$[\alpha]_D^{15}$ –80.8 (*c* 1.2, $CHCl_3$) for 91:9 er.

Enantioselective oxidative hetero-coupling of 2-hydroxycarbazole 1k and 2-naphthol 2l catalyzed by vanadium(V) complex



3kl

A test tube was charged with 9-benzyl-3-methyl-9*H*-carbazol-2-ol (**1k**) (1.0 equiv), 3-methoxynaphthalen-2-ol (**2l**) (1.0 equiv), mononuclear vanadium catalyst (10 mol%), lithium chloride (3.0 equiv) and 1,4-dioxane (0.1 M for **1k**) under air at 30 °C. The mixture was stirred for 96 h. The reaction mixture was then filtered through a short pad of silica gel and the solvent was evaporated. The crude product was purified by silica gel column chromatography to afford hetero-coupling product **3kl**.

9-Benzyl-1-(2-hydroxy-3-methoxynaphthalen-1-yl)-3-methyl-9*H*-carbazol-2-ol (3kl)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, J = 7.8 Hz, 1H), 8.01 (s, 1H), 7.73 (d, J = 8.2 Hz, 1H), 7.33-7.20 (m, 3H), 7.15 (dd, J = 15.1, 8.2 Hz, 4H), 6.99 (t, J = 6.9 Hz, 1H), 6.92 (t, J = 7.8 Hz, 2H), 6.34 (d, J = 7.8 Hz, 2H), 5.47 (s, 1H), 4.89 (d, J = 17.4 Hz, 1H), 4.84 (s, 1H), 4.55 (d, J = 17.4 Hz, 1H), 3.97 (d, J = 1.4 Hz, 3H), 2.47 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 151.2, 147.1, 145.1, 141.7, 138.0, 137.8, 129.7, 128.8, 127.7, 126.9, 126.3, 125.3, 125.1, 124.6, 124.5, 124.3, 123.1, 122.1, 119.3, 119.1, 117.3, 117.1, 111.2, 108.6, 106.9, 102.3, 55.8, 46.9, 16.7.

HRMS (ESI): calcd for $C_{31}H_{25}NO_3Na$: *m/z* 482.1727 $[M + Na]^+$, found 482.1722.

IR (KBr): 3446, 2373, 2345, 1655, 1563, 1264, 1073, 908, 865, 735 cm^{-1}.

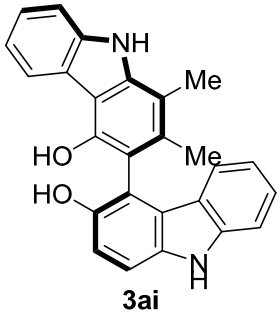
mp: 182-184 °C (yellow brown solid).

Enantiomeric ratio: 82:18, determined by HPLC (Chiralpak IC, *n*-hexane:2-propanol = 4/1; flow rate 1.0 mL/min; 25 °C; 303 nm) first peak: t_R = 9.2 min, second peak: t_R = 31.0 min.

$[\alpha]_D^{21}$ +33.7 (*c* 4.4, $CHCl_3$) for 82:18 er.

Enantioselective oxidative hetero-coupling of 3-hydroxycarbazole and 4-hydroxycarbazole catalyzed by vanadium(V) complex

A test tube was charged with **1i** (1.0 equiv), **1a** (1.0 equiv), mononuclear vanadium catalyst (10 mol%), lithium chloride (3.0 equiv) and 1,4-dioxane (0.1 M for **1i**) under air at 30 ℃. The mixture was stirred for 96 h. The reaction mixture was then filtered through a short pad of silica gel and the solvent was evaporated. The crude product was purified by silica gel column chromatography to afford hetero-coupling product **3ia**.



3ai

1,2-Dimethyl-9*H*,9'*H*-[3,4'-bicarbazole]-3',4-diol (3ia)

^{1}H NMR (600 MHz, ACETONE-D_6) δ: 10.25 (brs, 1H), 10.16 (brs, 1H), 8.20 (d, *J* = 7.6 Hz, 1H), 7.51 (d, *J* = 8.2 Hz, 1H), 7.44 (d, *J* = 8.9 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 1H), 7.20 (brs, 1H), 7.19-7.10 (m, 3H), 7.03 (brs, 1H), 6.75 (d, *J* = 8.2 Hz, 1H), 6.65 (t, *J* = 7.6 Hz, 1H), 2.52 (s, 3H), 2.08 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$): δ 150.0, 149.5, 142.0, 141.6, 140.3, 135.4, 134.5, 125.6, 124.7, 124.5, 124.1, 124.0, 123.2, 121.9, 119.4, 118.6, 115.9, 115.7, 112.6, 112.0, 111.1, 110.8, 110.6, 110.4, 16.7, 13.7.

HRMS (ESI): calcd for $C_{26}H_{20}N_2O_2Na$: *m/z* 415.1417 $[M + Na]^+$, found 415.1410.

IR (KBr): 3446, 3419, 3349, 3057, 2921, 1709, 1610, 1498, 1439, 1350, 1166, 1113, 902, 756 cm^{-1}.

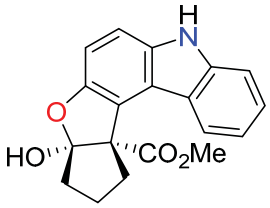
mp: 206-207 °C (yellow brown solid).

Enantiomeric ratio: 71:29, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 3/1; flow rate 2.0 mL/min; 25 °C; 339 nm) first peak: t_R = 4.8 min, second peak: t_R = 7.7 min.

$[\alpha]_D^{16}$ –129.5. (*c* 2.6, acetone) for 71:29 er.

Enantioselective oxidative hetero-coupling of 3-hydroxycarbazole and *β*-ketoester catalyzed by vanadium(V) complex

To a solution of 3-hydroxycarbazole **1** (0.52 mmol, 1.5 eq) and methyl 2-oxocyclopentanecarboxylate (**7**) (0.35 mmol) in chlorobenzene (0.1 M), mononuclear vanadium catalyst (10 mol%) was added. The mixture was stirred under air at 50 ℃ for 24 h. The reaction mixture was then filtered through a short pad of silica gel and the solvent was evaporated. The crude product was purified by silica gel column chromatography to afford hetero-coupling product **8**.



8a
(major)

Compound 8a (major diastereomer)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (s, 1H), 7.80 (dd, J = 7.8, 0.9 Hz, 1H), 7.43-7.38 (m, 2H), 7.28 (d, J = 8.7 Hz, 1H), 7.18 (ddd, J = 8.0, 5.5, 2.5 Hz,1H), 6.97 (d, J = 8.7 Hz, 1H), 3.90 (s, 1H), 3.71 (s, 3H), 3.07 (td, J = 12.4, 6.4 Hz, 1H), 2.43-2.34 (m, 2H), 2.17 (td, J = 12.4, 6.4 Hz, 1H), 1.91-1.83 (m, 1H), 1.61-1.51 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.6, 152.9, 140.4, 135.3, 126.0, 122.2, 121.2, 120.7, 120.6, 119.4, 119.2, 110.9, 110.7, 108.0, 65.1, 53.0, 39.6, 34.7, 22.7.

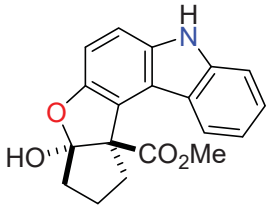
HRMS (ESI): calcd for $C_{19}H_{17}NO_4Na$: *m/z* 346.1050 $[M + Na]^+$, found 346.1046.

IR (KBr): 3413, 2953, 1720, 1496, 1442, 1321, 1277, 1101, 937 cm^{-1}.

mp: 98-100 °C (white solid).

Enantiomeric ratio: 93:7, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 350 nm) first peak: t_R = 16.7 min, second peak: t_R = 19.3 min.

$[\alpha]_D^{29}$ +72.46 (*c* 1.0, THF) for 93:7 er.



8a'
(minor)

Compound 8a' (minor diastereomer)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (dd, J = 7.8, 0.9 Hz, 1H), 7.90 (s, 1H), 7.42 (s, 1H), 7.39-7.38 (m, 2H), 7.20-7.16 (m, 2H), 5.19 (s, 1H), 3.81 (s, 3H), 2.65 (td, J = 12.4, 6.4 Hz, 1H), 2.41 (ddt, J = 13.3, 6.4, 1.8 Hz, 1H), 2.23 (ddt, J = 13.3, 6.4, 1.8 Hz, 1H), 2.17-2.08 (m, 1H), 1.83-1.75 (m, 1H), 1.55-1.46 (m, 1H)

^{13}C NMR (150 MHz, $CDCl_3$): δ 173.3, 152.6, 140.3, 134.8, 128.2, 126.0, 124.5, 123.2, 120.4, 119.8, 119.2, 110.6, 106.1, 99.8, 62.9, 52.9, 40.3, 37.5, 21.9.

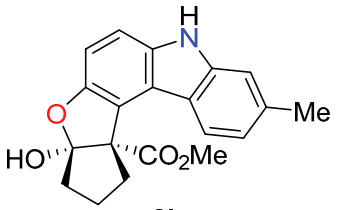
HRMS (ESI): calcd for $C_{19}H_{17}NO_4Na$: *m/z* 346.1050 $[M + Na]^+$, found 346.1046.

IR (KBr): 3413, 2959, 1715, 1458, 1250, 1096, 860 cm^{-1}.

mp: 196-198 °C (white solid).

Enantiomeric ratio: 88:12, determined by HPLC (Chiralpak IA, *n*-hexane/2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 360 nm) first peak: t_R = 28.5 min, second peak: t_R = 48.9 min.

$[\alpha]_D^{29}$ +25.6 (*c* 0.32, $CHCl_3$) for 88:12 er.



8b
(major)

Compound 8b (major diastereomer)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (s, 1H), 7.66 (d, J = 8.2 Hz, 1H), 7.22 (d, J = 8.7 Hz, 1H), 7.18 (s, 1H), 7.00 (d, J = 8.2 Hz, 1H), 6.92 (d, J = 8.7 Hz, 1H), 3.94 (s, 1H), 3.69 (s, 3H), 3.05 (td, J = 12.4, 6.4 Hz, 1H), 2.50 (s, 3H), 2.42-2.32 (m, 2H), 2.16 (td, J = 12.4, 6.4 Hz, 1H), 1.89-1.82 (m, 1H), 1.59-1.53 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.7, 152.8, 141.0, 136.3, 135.3, 121.9, 121.0, 120.6, 120.3, 119.2, 118.9, 110.8, 110.7, 107.3, 65.0, 52.9, 39.6, 34.6, 22.7, 22.0.

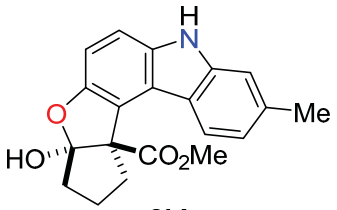
HRMS (ESI): calcd for $C_{20}H_{19}NO_4Na$: *m/z* 360.1206 $[M + Na]^+$, found 360.1201.

IR (KBr): 3485, 3392, 2948, 1726, 1616, 1436, 1260, 1123, 937, 805 cm^{-1}.

mp: 215-217 °C (white solid).

Enantiomeric ratio: 92:8, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 315 nm) first peak: t_R = 18.5 min, second peak: t_R = 23.6 min.

$[\alpha]_D^{29}$ +26.7 (*c* 0.33, $CHCl_3$) for 92:8 er.



8b'
(minor)

Compound 8b' (minor diastereomer)

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 (d, J = 8.2 Hz, 1H), 7.78 (s, 1H), 7.37 (s, 1H), 7.16 (s, 1H), 7.13 (s, 1H), 7.01 (d, J = 8.2 Hz, 1H), 5.20 (s, 1H), 3.81 (s, 3H), 2.64 (td, J = 12.8, 6.4 Hz, 1H), 2.50 (s, 3H), 2.40 (ddt, J = 13.5, 6.4, 1.8 Hz, 1H), 2.22 (ddt, J = 13.5, 6.4, 1.8 Hz, 1H), 2.11 (td, J = 12.8, 6.4 Hz, 1H), 1.81-1.75 (m, 1H), 1.57-1.44 (m, 1H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 173.4, 152.6, 140.8, 136.2, 134.8, 127.4, 124.6, 120.9, 120.8, 120.0, 119.8, 110.7, 105.9, 99.5, 62.8, 52.9, 40.3, 37.5, 22.1, 21.9.

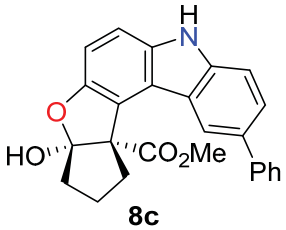
HRMS (ESI): calcd for $C_{20}H_{19}NO_4Na$: *m/z* 360.1206 $[M + Na]^+$, found 360.1202.

IR (KBr): 3485, 3392, 2948, 1726, 1616, 1436, 1260, 1123, 937, 805 cm^{-1}.

mp: 178-180 °C (white solid).

Enantiomeric ratio: 90:10, determined by HPLC (Chiralpak IA, hexane/2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 300 nm) first peak: t_R = 34.3 min, second peak: t_R = 55.6 min.

$[\alpha]_D^{29}$ +59.4 (*c* 0.32, $CHCl_3$) for 90:10 er.



8c

Compound 8c

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (d, J = 1.4 Hz, 1H), 8.07 (s, 1H), 7.70-7.67 (m, 3H), 7.51-7.45 (m, 3H), 7.34 (tt, J = 7.3, 1.4 Hz, 1H), 7.28 (d, J = 8.2 Hz, 1H), 6.98 (d, J = 8.7 Hz, 1H), 4.03 (s, 1H), 3.72 (s, 3H), 3.10 (td, J = 12.6, 6.4 Hz, 1H), 2.44-2.38 (m, 2H), 2.18 (td, J = 12.6, 6.4 Hz, 1H), 1.91-1.83 (m, 1H), 1.62-1.50 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.6, 153.0, 141.6, 139.9, 135.7, 132.6, 128.9, 126.9, 126.5, 125.5, 121.8, 120.8, 120.7, 120.5, 119.3, 111.1, 111.0, 108.2, 65.1, 53.0, 39.7, 34.7, 22.7.

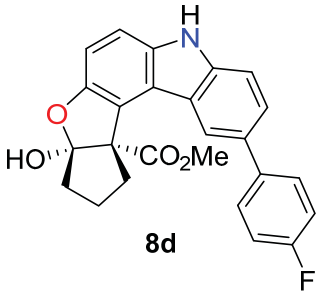
HRMS (ESI): calcd for $C_{25}H_{21}NO_4Na$: *m/z* 422.1363 $[M + Na]^+$, found 422.1357.

IR (KBr): 3409, 3376, 2943, 1704, 1600, 1491, 1260, 1118, 937, 756 cm^{-1}.

mp: 118-120 °C (white solid).

Enantiomeric ratio: 92:8, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 315 nm) first peak: t_R = 17.1 min, second peak: t_R = 23.2 min.

$[\alpha]_D^{29}$ +15.89 (*c* 0.1, THF) for 92:8 er.



8d

Compound 8d

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (s, 1H), 8.03 (t, J = 1.4 Hz, 1H), 7.64-7.59 (m, 3H), 7.47 (d, J = 8.7 Hz, 1H), 7.30 (d, J = 8.7 Hz, 1H), 7.20-7.14 (m, 2H), 6.99 (d, J = 8.7 Hz, 1H), 4.99 (s, 1H), 3.71 (s, 3H), 3.09 (td, J = 12.6, 6.4 Hz, 1H), 2.43-2.39 (m, 2H), 2.18 (td, J = 12.6, 6.4 Hz, 1H), 1.90-1.84 (m, 1H), 1.63-1.50 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.6, 162.0 (d, $^1J_{C\text{-}F}$ = 246.3 Hz), 153.0, 139.8, 137.8, 135.8, 131.7, 128.40 (d, $^3J_{C\text{-}F}$ = 8.6 Hz), 125.3, 121.80, 120.80, 120.71, 120.40, 119.21, 115.7 (d, $^2J_{C\text{-}F}$ = 21.7 Hz), 111.1, 111.0, 108.3, 65.0, 53.0, 39.7, 34.7, 22.6.

^{19}F NMR (565 MHz, $CDCl_3$): δ –120.2.

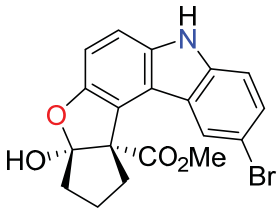
HRMS (ESI): calcd for $C_{25}H_{20}FNO_4Na$: *m/z* 440.1269 $[M + Na]^+$, found 440.1263.

IR (KBr): 3408, 2951, 1716, 1488, 1254, 1227, 1091, 944, 808 cm^{-1}.

mp: 116-118 °C (white solid).

Enantiomeric ratio: 90:10, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 315 nm) first peak: t_R = 17.6 min, second peak: t_R = 26.7 min.

$[\alpha]_D^{28}$ –10.5 (*c* 0.40, $CHCl_3$) for 90:10 er.



8e

Compound 8e (major diastereomer)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (s, 1H), 7.95 (d, J = 1.8 Hz, 1H), 7.49 (dd, J = 8.7, 1.8 Hz, 1H), 7.30 (d, J = 8.7 Hz, 1H), 7.28 (d, J = 8.7 Hz, 1H), 6.99 (d, J = 8.7 Hz, 1H), 4.11 (s, 1H), 3.78 (s, 3H), 3.04 (td, J = 12.6, 6.4 Hz, 1H), 2.43-2.33 (m, 2H), 2.17 (td, J = 12.6, 6.4 Hz, 1H), 1.91-1.85 (m, 1H), 1.61-1.49 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 172.7, 153.1, 138.9, 135.7, 128.6, 124.7, 122.8, 120.6, 118.1, 112.2, 111.8, 111.3, 108.9, 64.9, 53.0, 39.7, 34.7, 22.6 (one carbon overlapped).

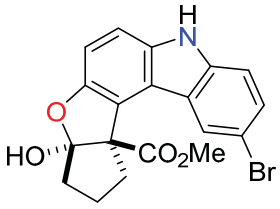
HRMS (ESI): calcd for $C_{19}H_{16}BrNO_4Na$: *m/z* 424.0155 $[M + Na]^+$, found 424.0150.

IR (KBr): 3408, 2951, 1721, 1493, 1439, 1281, 1096, 944, 797 cm^{-1}.

mp: 105-108 °C (white solid).

Enantiomeric ratio: 91:9, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 309 nm) first peak: t_R = 15.0 min, second peak: t_R = 20.1 min.

$[\alpha]_D^{29}$ –78.95 (*c* 1.0, THF) for 91:9 er.



8e'
(minor)

Compound 8e' (minor diastereomer)

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (d, J = 1.8 Hz, 1H), 7.91 (s, 1H), 7.46 (dd, J = 8.7, 1.8 Hz, 1H), 7.35 (s, 1H), 7.26 (d, J = 7.8 Hz, 1H), 7.17 (s, 1H), 5.13 (s, 1H), 3.81 (s, 3H), 3.04 (td, J = 12.4, 6.4 Hz, 1H), 2.44-2.39 (m, 1H), 2.24-2.19 (m, 1H), 2.12 (td, J = 12.4, 6.4 Hz, 1H), 1.83-1.76 (m, 1H), 1.56-1.47 (m, 1H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 173.2, 152.8, 138.8, 135.1, 129.2, 128.6, 124.9, 123.4, 123.0, 112.0, 112.1, 111.9, 106.4, 99.7, 62.9, 53.0, 40.2, 37.6, 22.0.

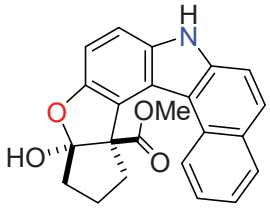
HRMS (ESI): calcd for $C_{19}H_{16}BrNO_4Na$: *m/z* 424.0155 $[M + Na]^+$, found 424.0149.

IR (KBr): 3418, 2951, 1721, 1466, 1270, 1096, 949, 868, 802, 753 cm^{-1}.

mp: 220-222 °C (white solid).

Enantiomeric ratio: 90:10, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 315 nm) first peak: t_R = 23.8 min, second peak: t_R = 54.0 min.

$[\alpha]_D^{28}$ +39.5 (*c* 0.40, $CHCl_3$) for 90:10 er.



8f'

Compound 8f'

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.62 (d, J = 8.2 Hz, 1H), 8.28 (s, 1H), 7.97 (d, J = 8.2 Hz, 1H), 7.91 (s, 1H), 7.82 (d, J = 8.7 Hz, 1H), 7.69 (ddd, J = 8.0, 7.1, 1.2 Hz, 1H), 7.57 (d, J = 8.7 Hz, 1H), 7.46 (ddd, J = 8.0, 7.1, 1.2 Hz, 1H), 7.30 (s, 1H), 5.20 (s, 1H), 3.83 (s, 3H), 2.68 (td, J = 12.4, 6.4 Hz, 1H), 2.40 (ddt, J = 13.5, 6.4, 1.8 Hz, 1H), 2.28 (ddt, J = 13.5, 6.4, 1.8 Hz, 1H), 2.15 (td, J = 12.4, 6.4 Hz, 1H), 1.84-1.78 (m, 1H), 1.60-1.48 (m, 1H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 173.4, 153.3, 137.9, 133.9, 129.8, 129.1, 129.0, 127.6, 127.0, 126.9, 124.8, 123.0, 122.8, 119.9, 115.2, 112.6, 106.5, 101.2, 62.9, 52.9, 40.3, 37.6, 22.0.

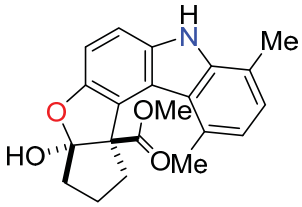
HRMS (ESI): calcd for $C_{23}H_{19}NO_4Na$: *m/z* 396.1206 $[M + Na]^+$, found396.1201.

IR (KBr): 3403, 2953, 1720, 1463, 1254, 1085, 937, 800 cm^{-1}.

mp: 146-148 °C (white solid).

Enantiomeric ratio: 94:6, determined by HPLC (Chiralpak IA, *n*-hexane:2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 315 nm) first peak: t_R = 17.4 min, second peak: t_R = 29.6 min.

$[\alpha]_D^{28}$ +38.6 (*c* 0.35, $CHCl_3$) for 94:6 er.



8g'

Compound 8g'

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (s, 1H), 7.55 (s, 1H), 7.21 (s, 1H), 7.09 (d, J = 7.3 Hz, 1H), 6.88 (d, J = 7.3 Hz, 1H), 5.20 (s, 1H), 3.81 (s, 3H), 2.78 (s, 3H),2.65 (td, J = 12.4, 6.4 Hz, 1H), 2.49 (s, 3H), 2.42 (ddt, J = 13.3, 6.4, 1.8 Hz, 1H), 2.24 (ddt, J = 13.3, 6.4, 1.8 Hz, 1H), 2.12 (td, J = 12.4, 6.4 Hz, 1H), 1.83-1.76 (m, 1H), 1.57-1.45 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 173.4, 152.5, 139.7, 134.8, 130.8, 127.0, 126.2, 125.3, 121.1, 120.5, 119.8, 117.0, 105.8, 102.0, 62.9, 52.9, 40.3, 37.5, 21.9, 20.3, 16.5.

HRMS (ESI): calcd for $C_{21}H_{21}NO_4Na$: *m/z* 374.1363 $[M + Na]^+$, found 374.1358.

IR (KBr): 3403, 2959, 1715, 1458, 1304, 1096, 855, 805 cm^{-1}.

mp: 215-217 °C (white solid).

Enantiomeric ratio: 84:16, determined by HPLC (Chiralpak IA, *n*-hexane/2-propanol = 7/1; flow rate 1.0 mL/min; 25 °C; 315 nm) first peak: t_R = 22.1 min, second peak: t_R = 35.4 min.

$[\alpha]_D^{28}$ +13.1 (*c* 0.40, $CHCl_3$) for 84:16 er.

References

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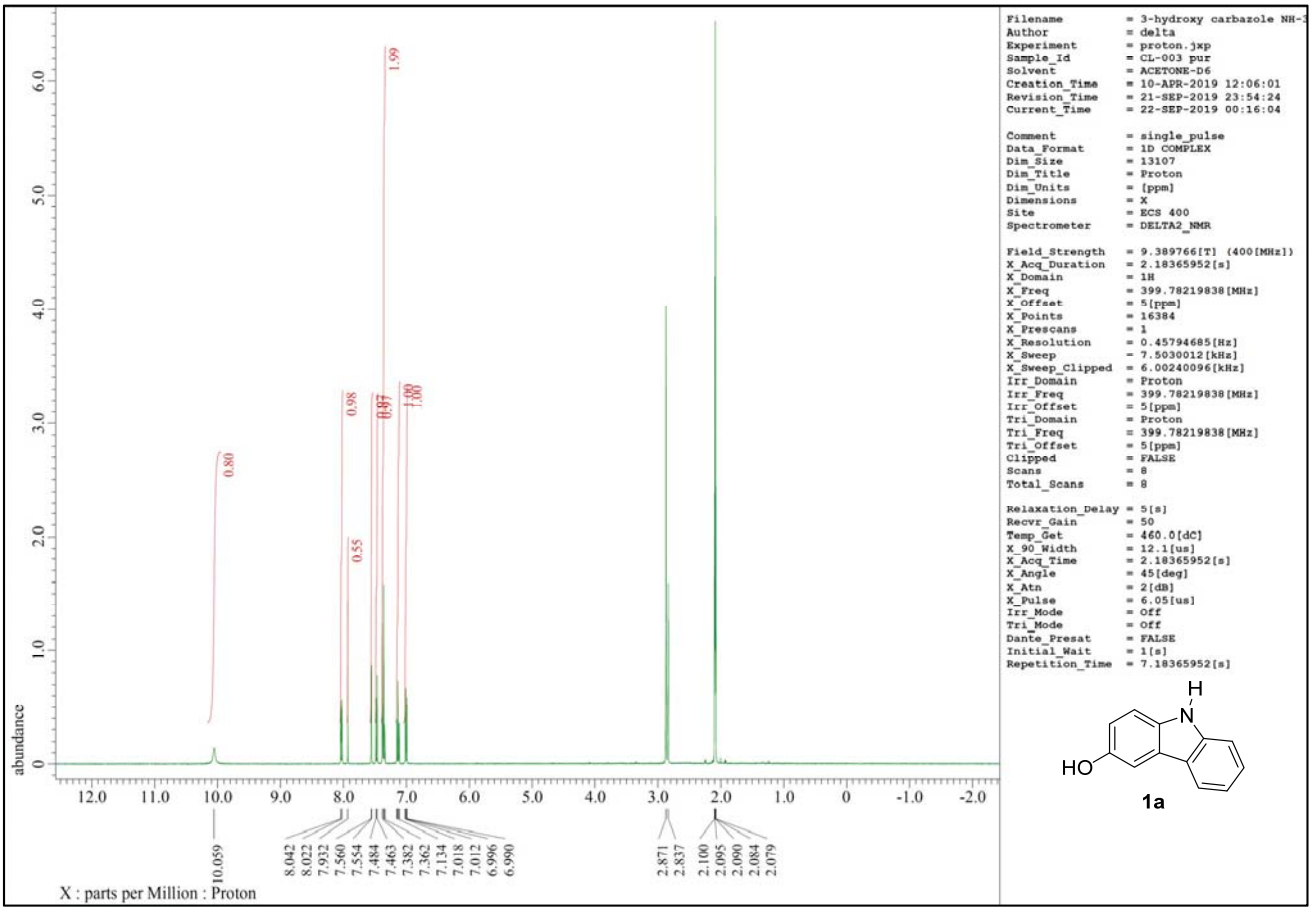
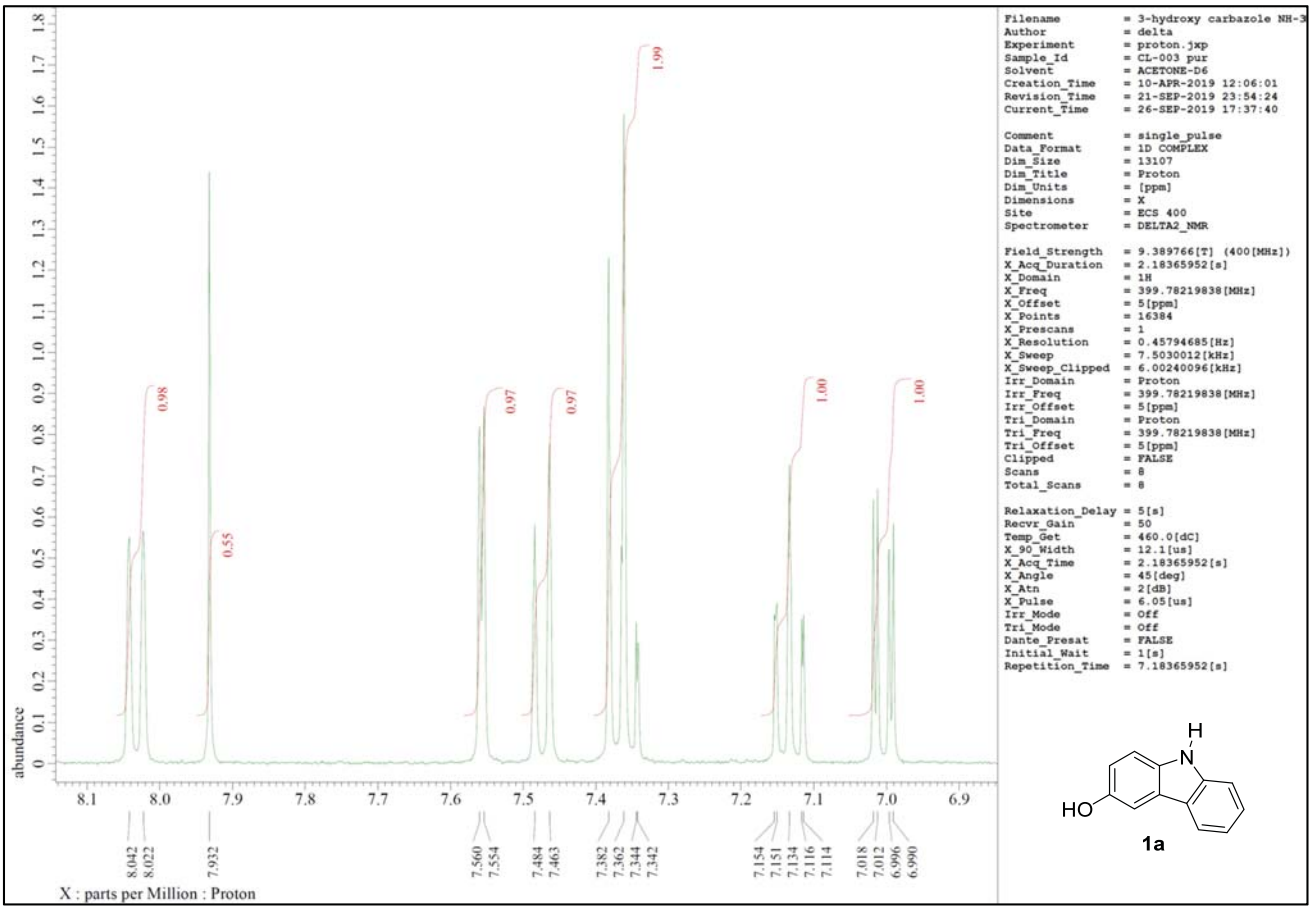
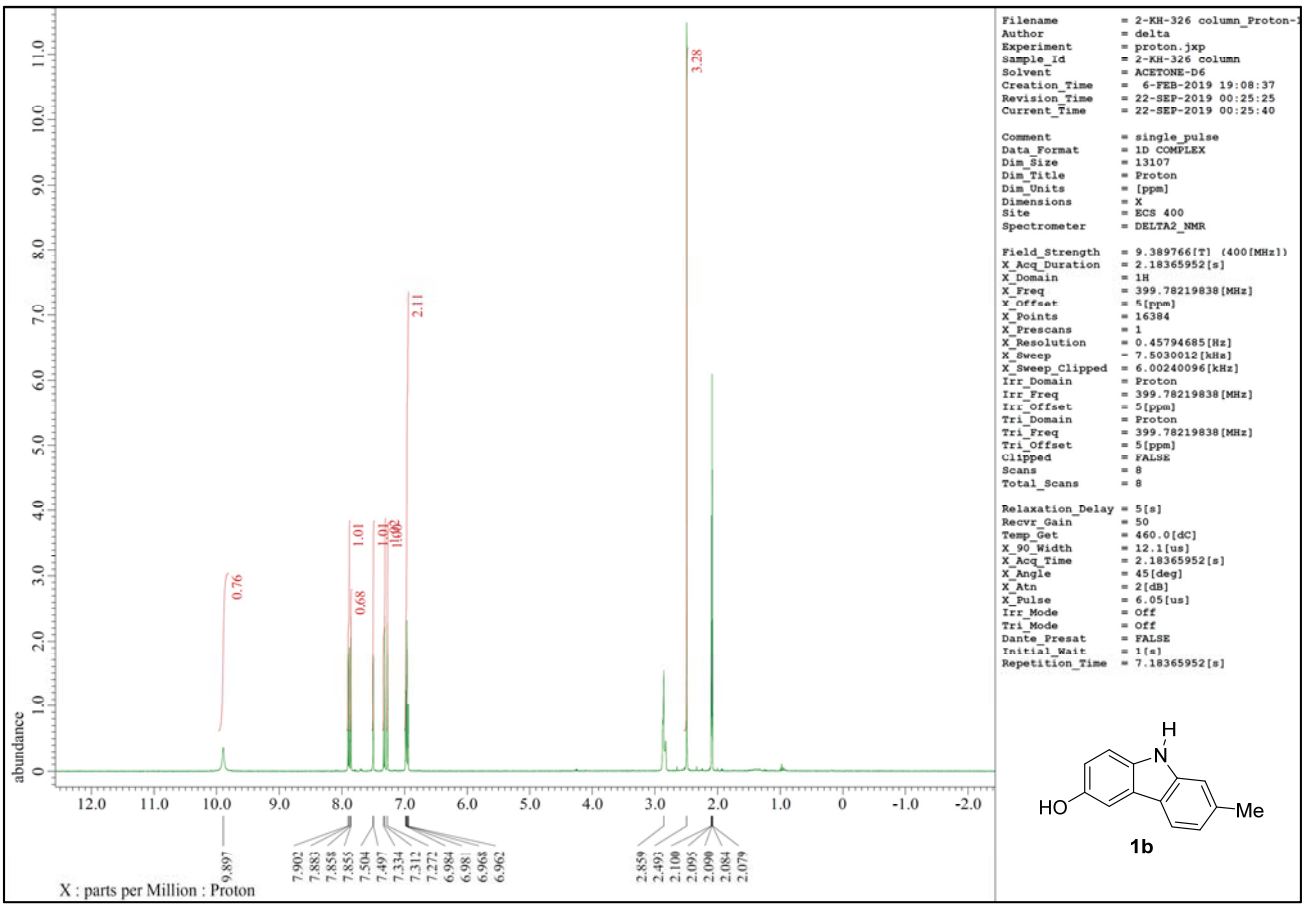
NMR spectra





Compound **1a** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$)

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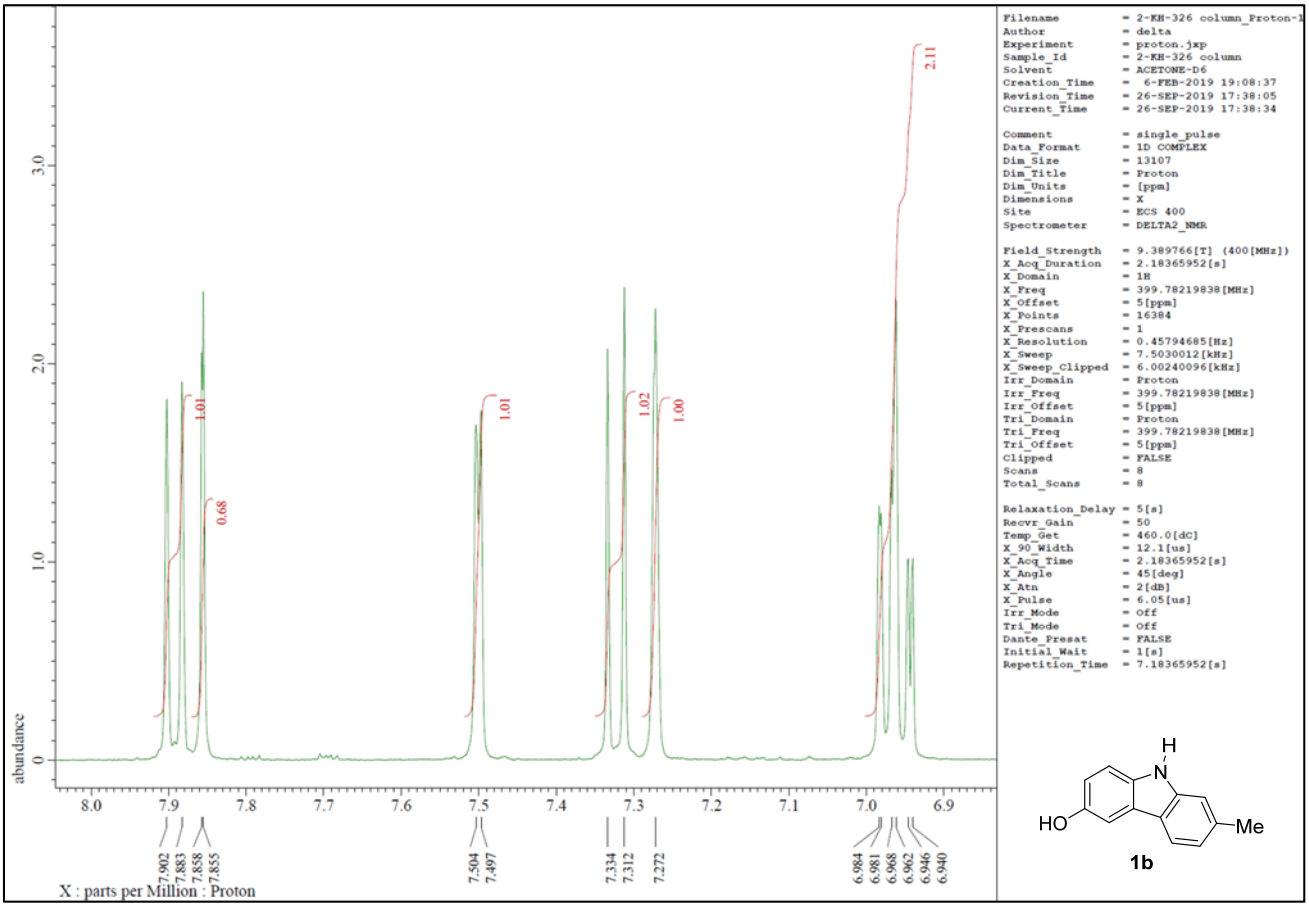
```


1b



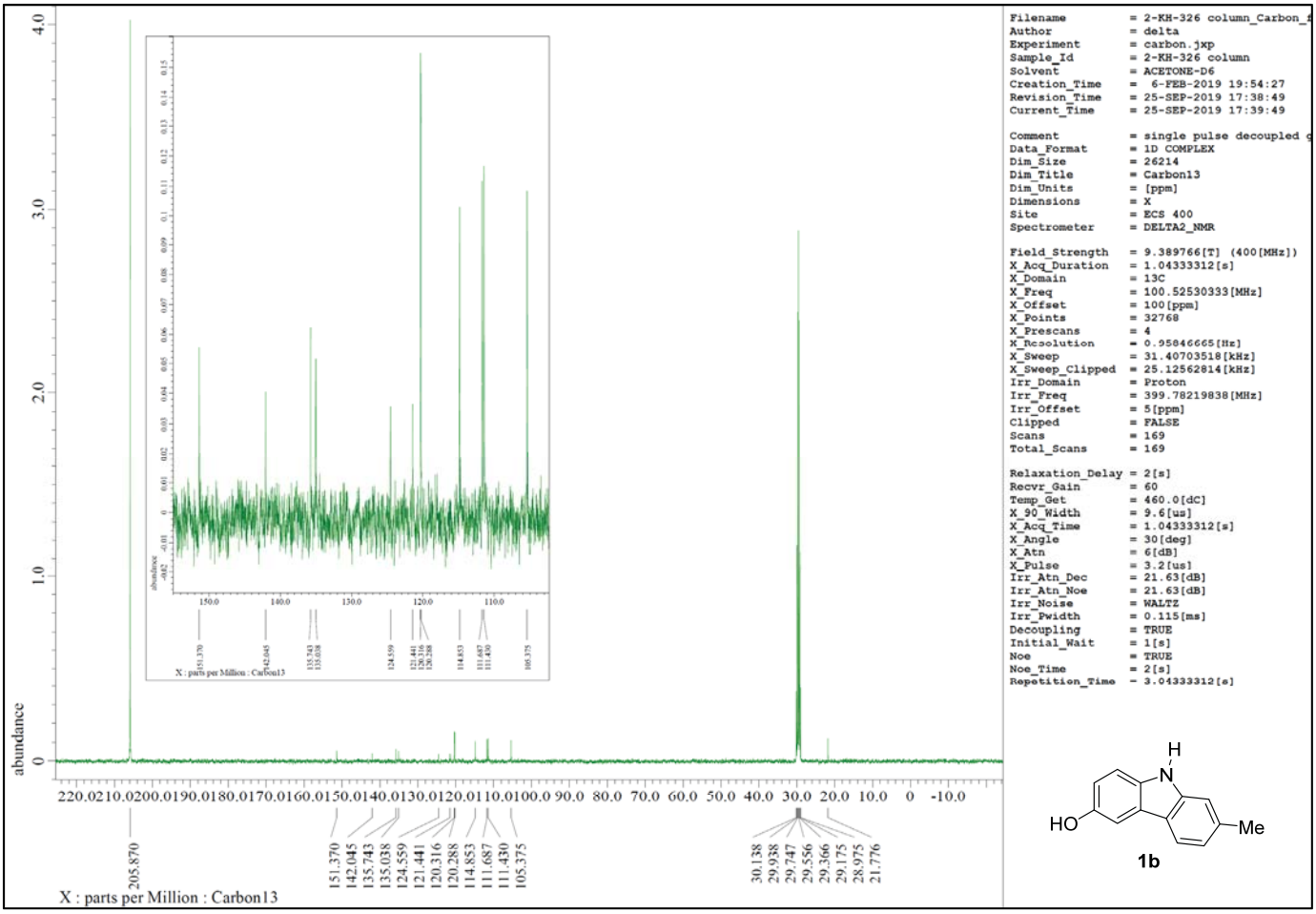

```
Filename          = 2-KH-326 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-326 column
Solvent           = ACETONE-D6
Creation_Time     =  6-FEB-2019 19:08:37
Revision_Time     = 26-SEP-2019 17:38:05
Current_Time      = 26-SEP-2019 17:38:34

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

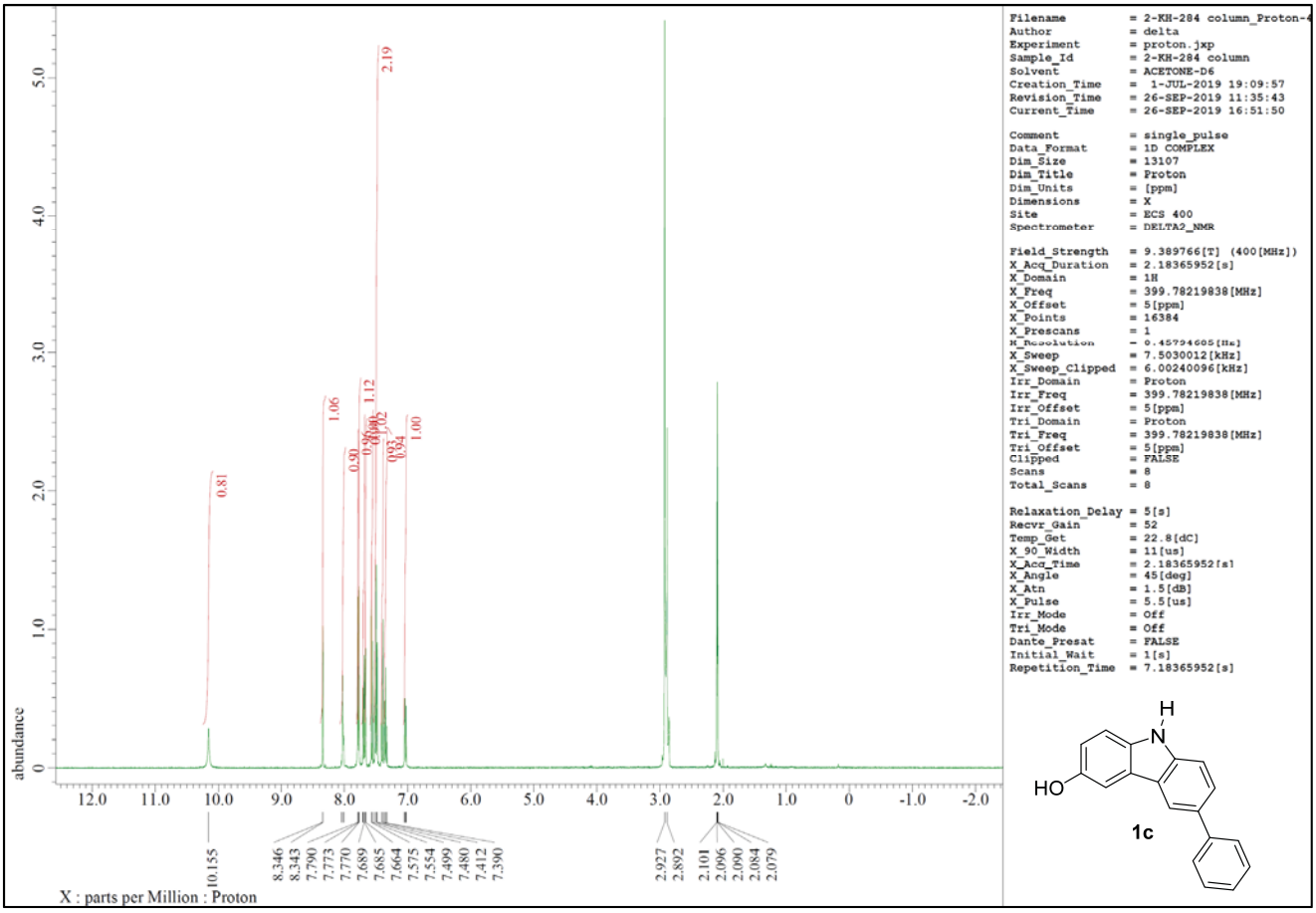
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 460.0[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```


1b



Compound **1b** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)

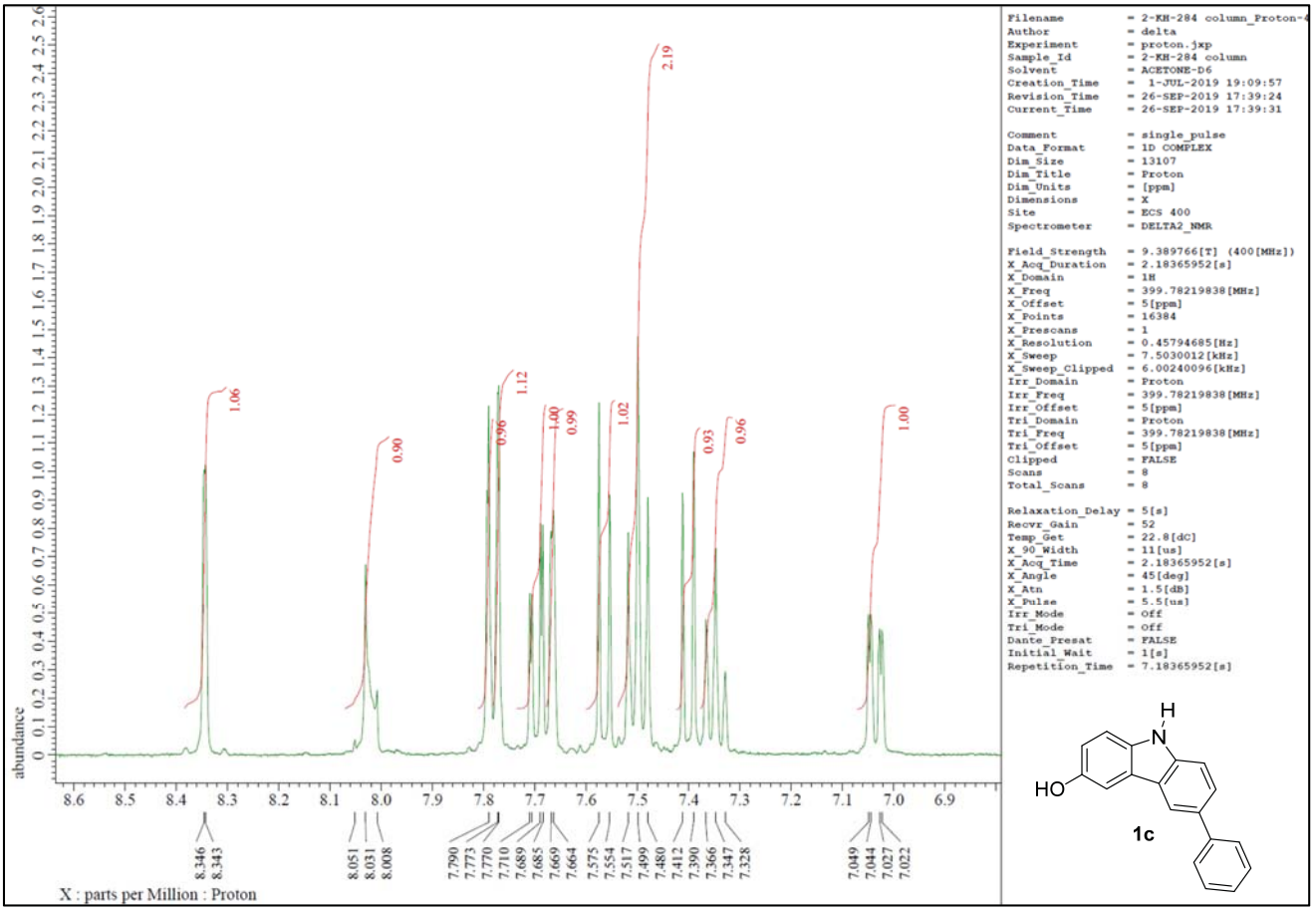



```
Filename          = 2-KH-284 column_Proton-4
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-284 column
Solvent           = ACETONE-D6
Creation_Time     =  1-JUL-2019 19:09:57
Revision_Time     = 26-SEP-2019 11:35:43
Current_Time      = 26-SEP-2019 16:51:50

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 52
Temp_Get          = 22.8[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

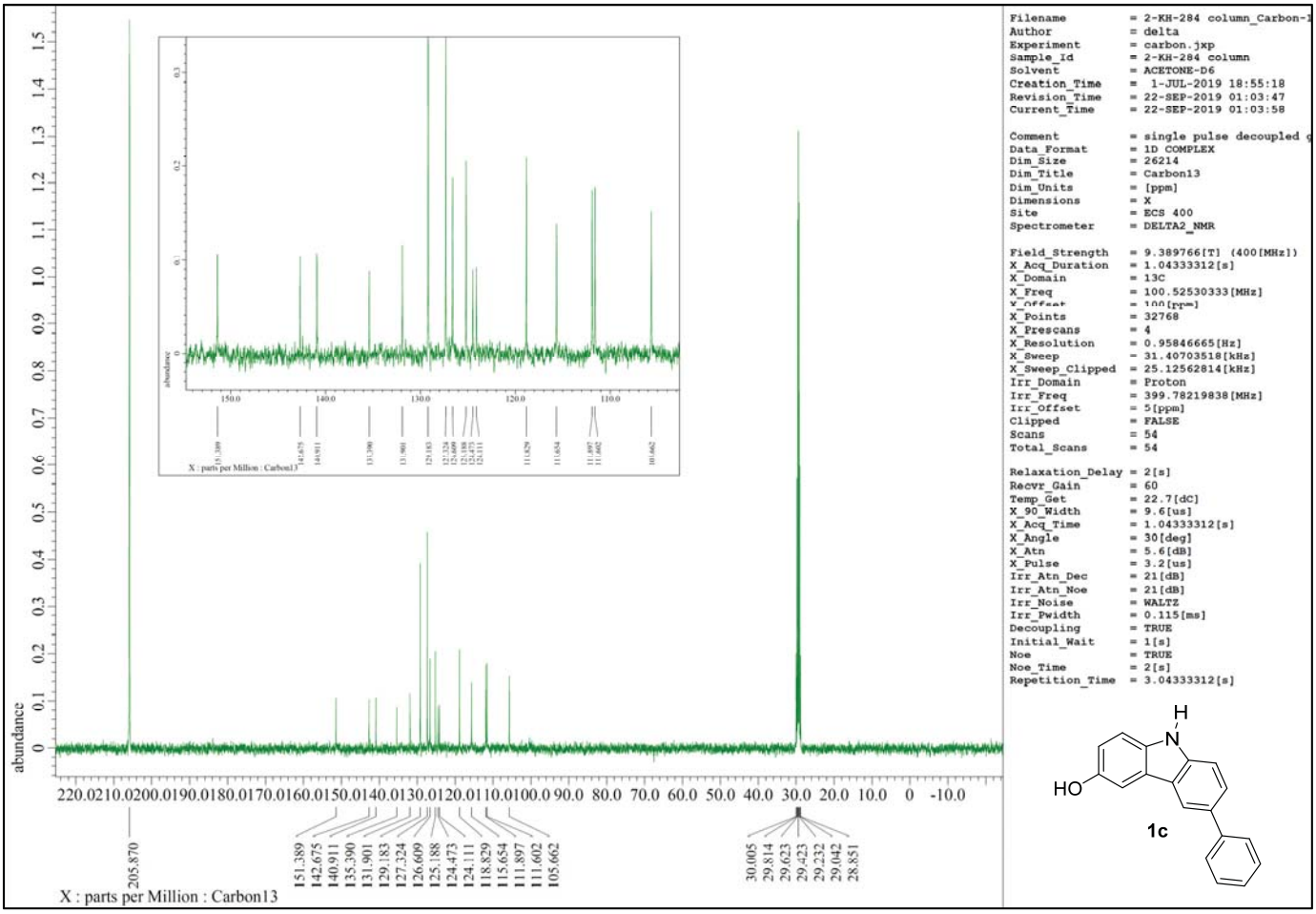



```
Filename          = 2-KH-284 column_Proton-4
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-284 column
Solvent           = ACETONE-D6
Creation_Time     =  1-JUL-2019 19:09:57
Revision_Time     = 26-SEP-2019 17:39:24
Current_Time      = 26-SEP-2019 17:39:31

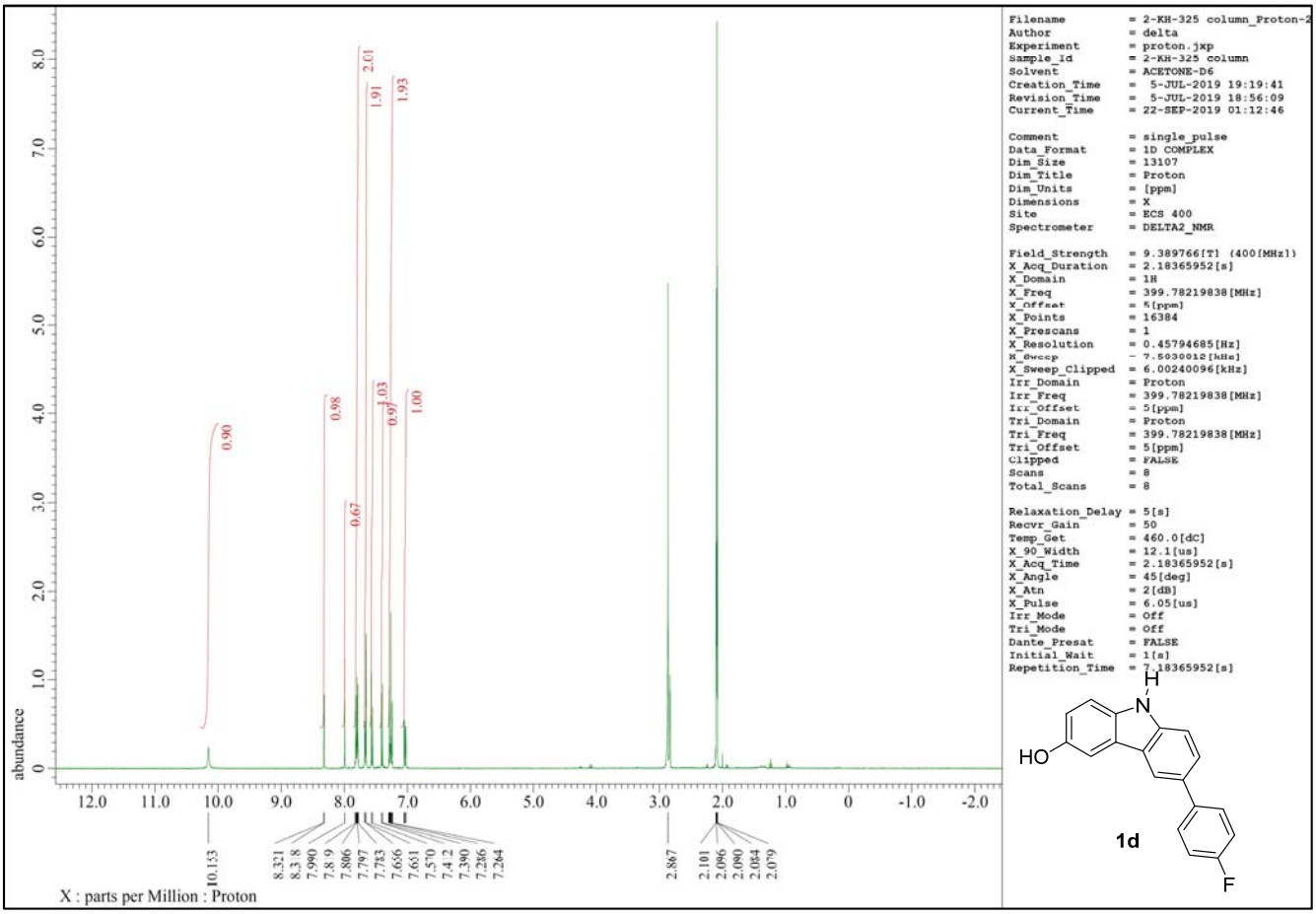
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

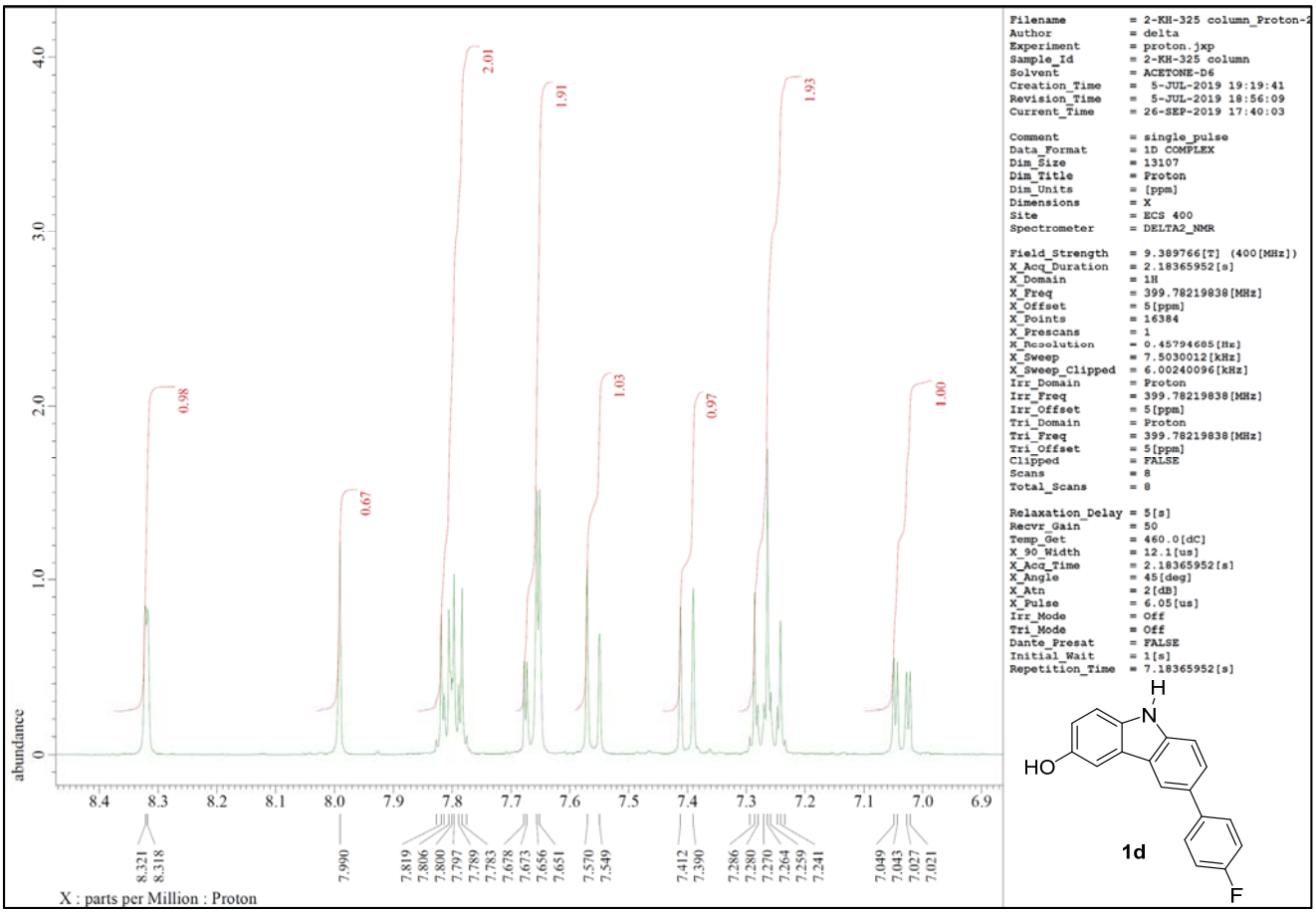
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

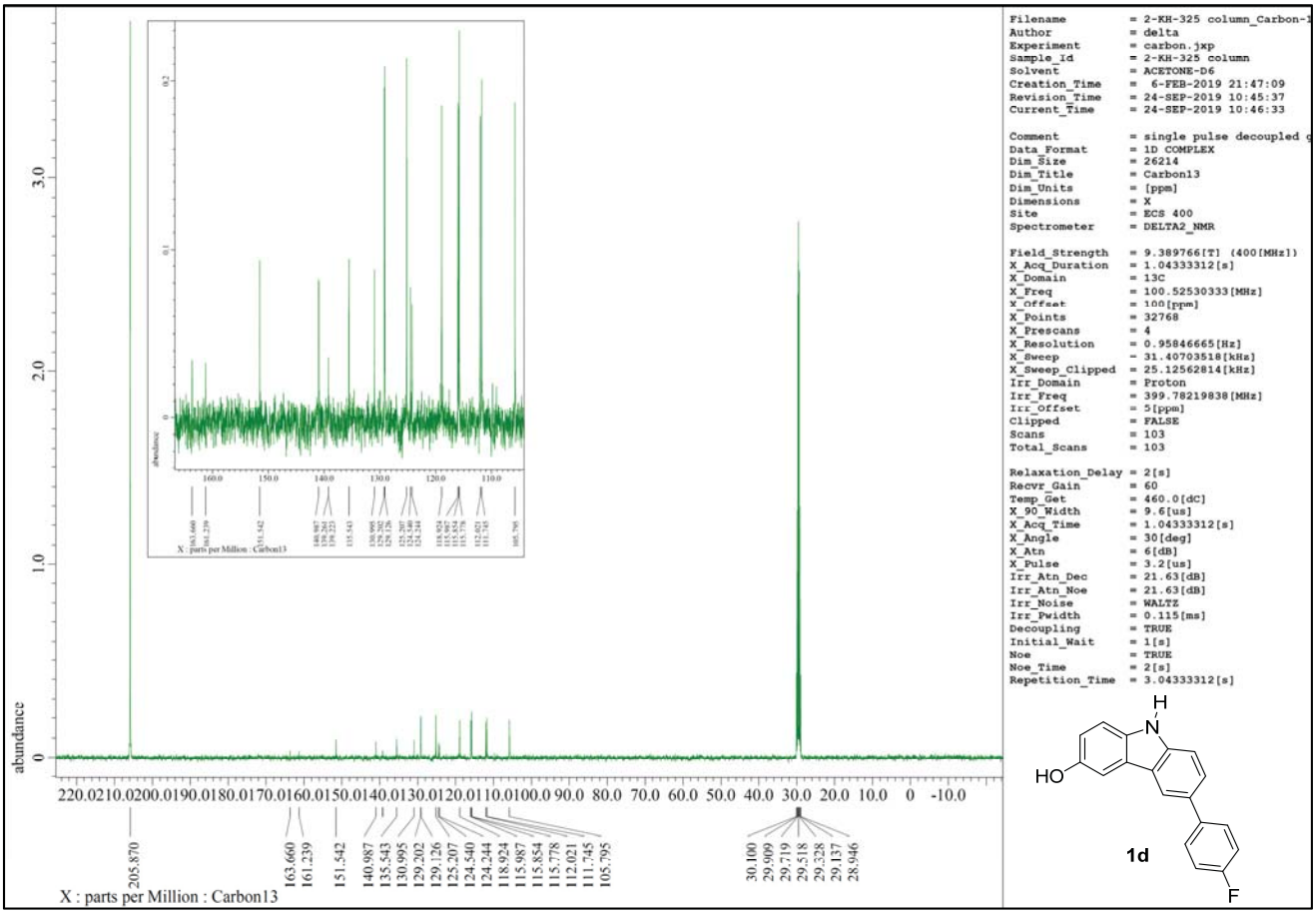
Relaxation_Delay  = 5[s]
Recvr_Gain        = 52
Temp_Get          = 22.8[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

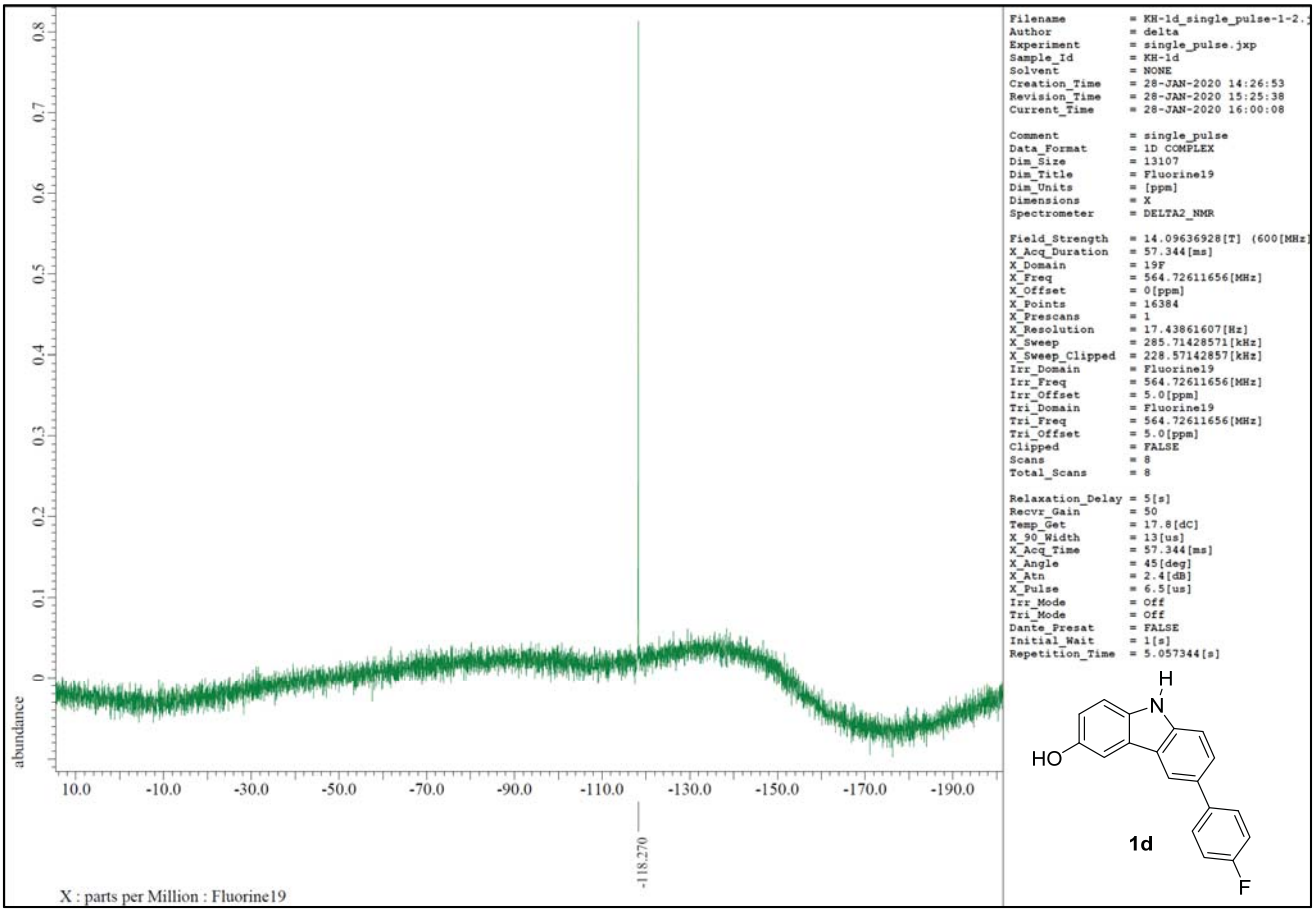


Compound **1c** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)

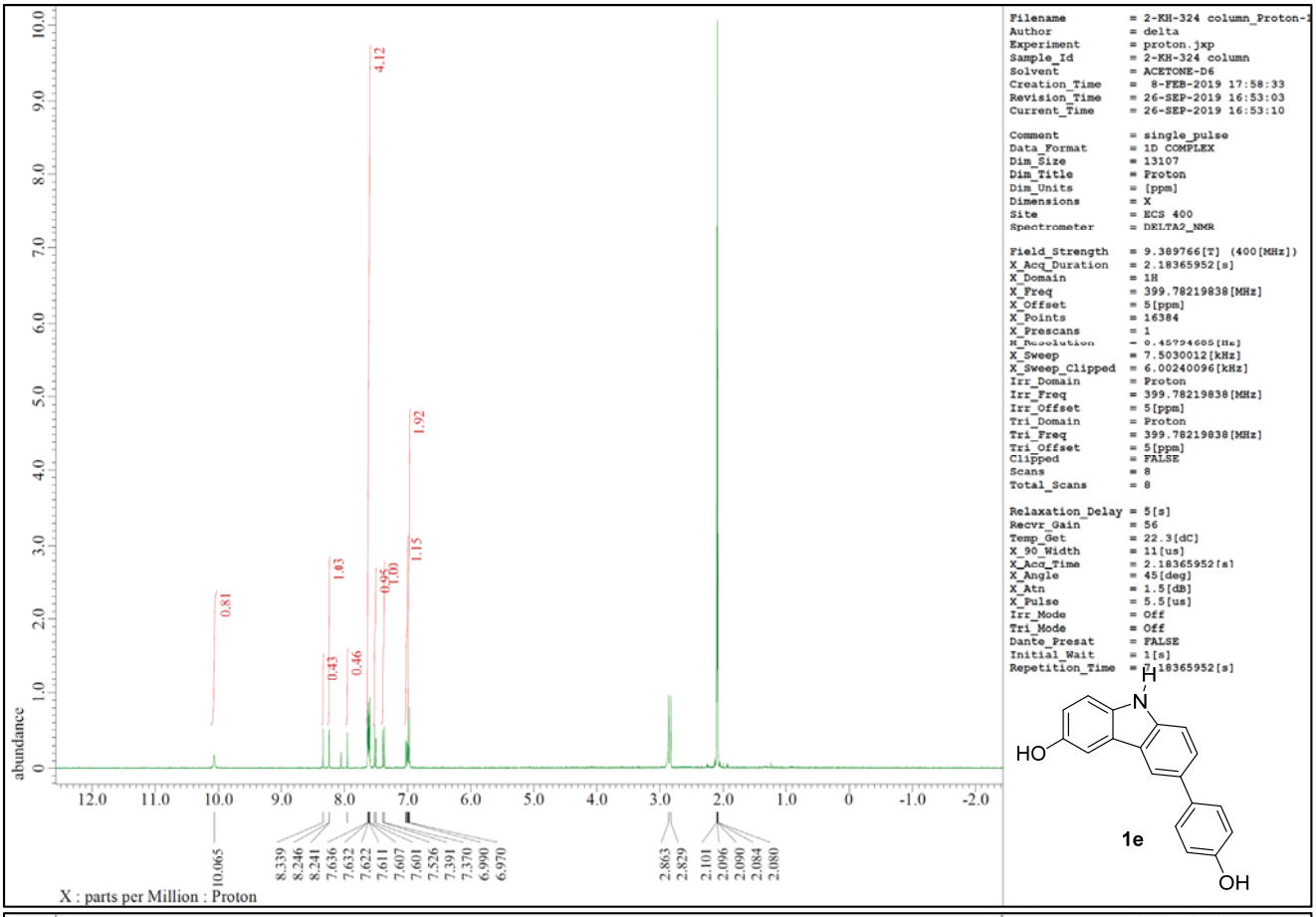

abundance
X : parts per Million : Proton
0.90
0.98
0.67
2.01
1.91
1.93
1.03
0.97
1.00
10.153
8.321
8.318
7.990
7.819
7.806
7.800
7.797
7.789
7.783
7.678
7.673
7.656
7.651
7.570
7.549
7.412
7.390
7.286
7.280
7.270
7.264
7.259
7.241
2.867
2.101
2.096
2.090
2.084
2.079
Filename = 2-KH-325 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-325 column
Solvent = ACETONE-D6
Creation_Time = 5-JUL-2019 19:19:41
Revision_Time = 5-JUL-2019 18:56:09
Current_Time = 22-SEP-2019 01:12:46
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
F
1d

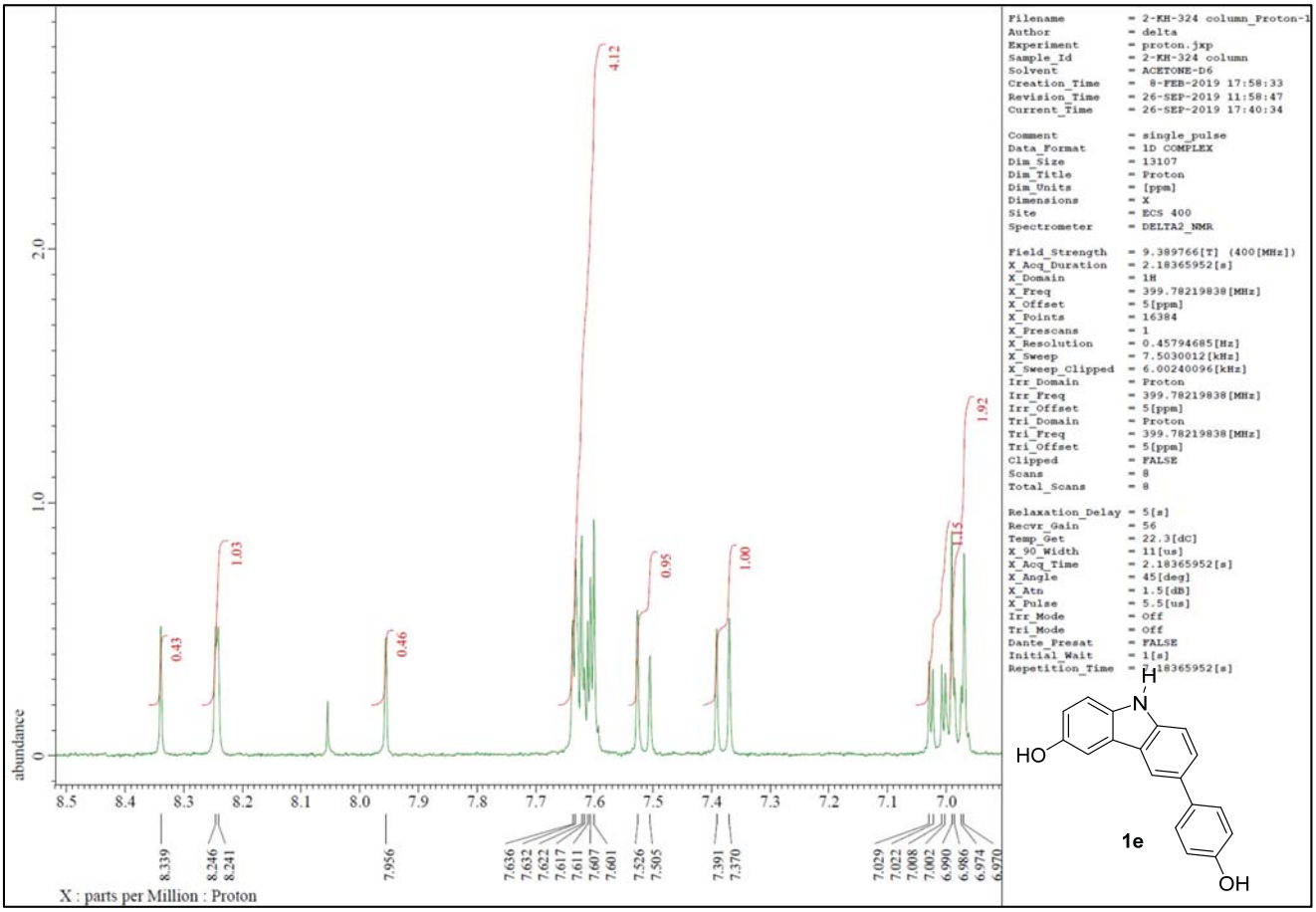

abundance
X : parts per Million : Proton
0.98
0.67
2.01
1.91
1.03
0.97
1.93
1.00
8.321
8.318
7.990
7.819
7.806
7.800
7.797
7.789
7.783
7.678
7.673
7.656
7.651
7.570
7.549
7.412
7.390
7.286
7.280
7.270
7.264
7.259
7.241
7.049
7.043
7.027
7.021
Filename = 2-KH-325 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-325 column
Solvent = ACETONE-D6
Creation_Time = 5-JUL-2019 19:19:41
Revision_Time = 5-JUL-2019 18:56:09
Current_Time = 26-SEP-2019 17:40:03
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
F
1d

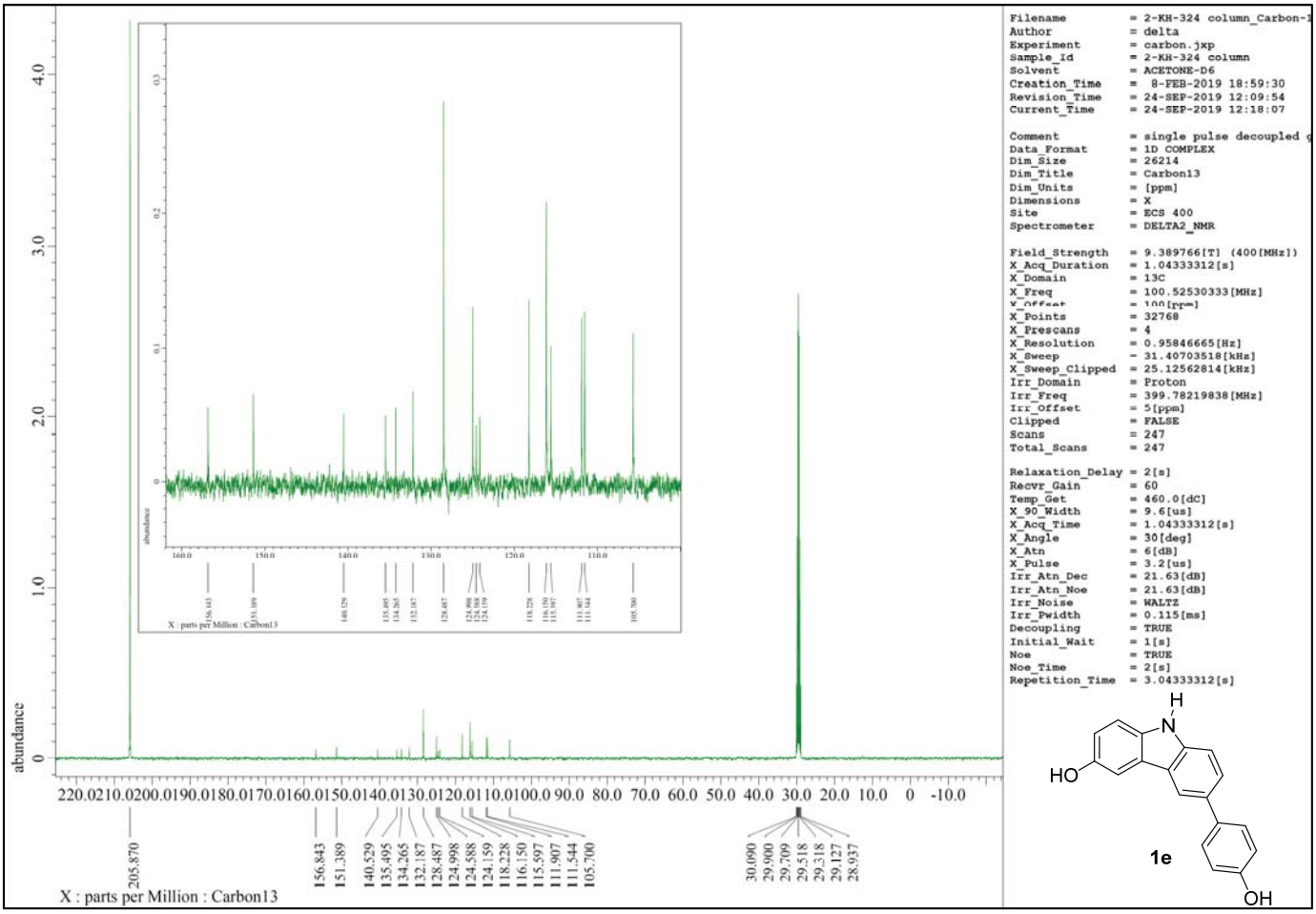




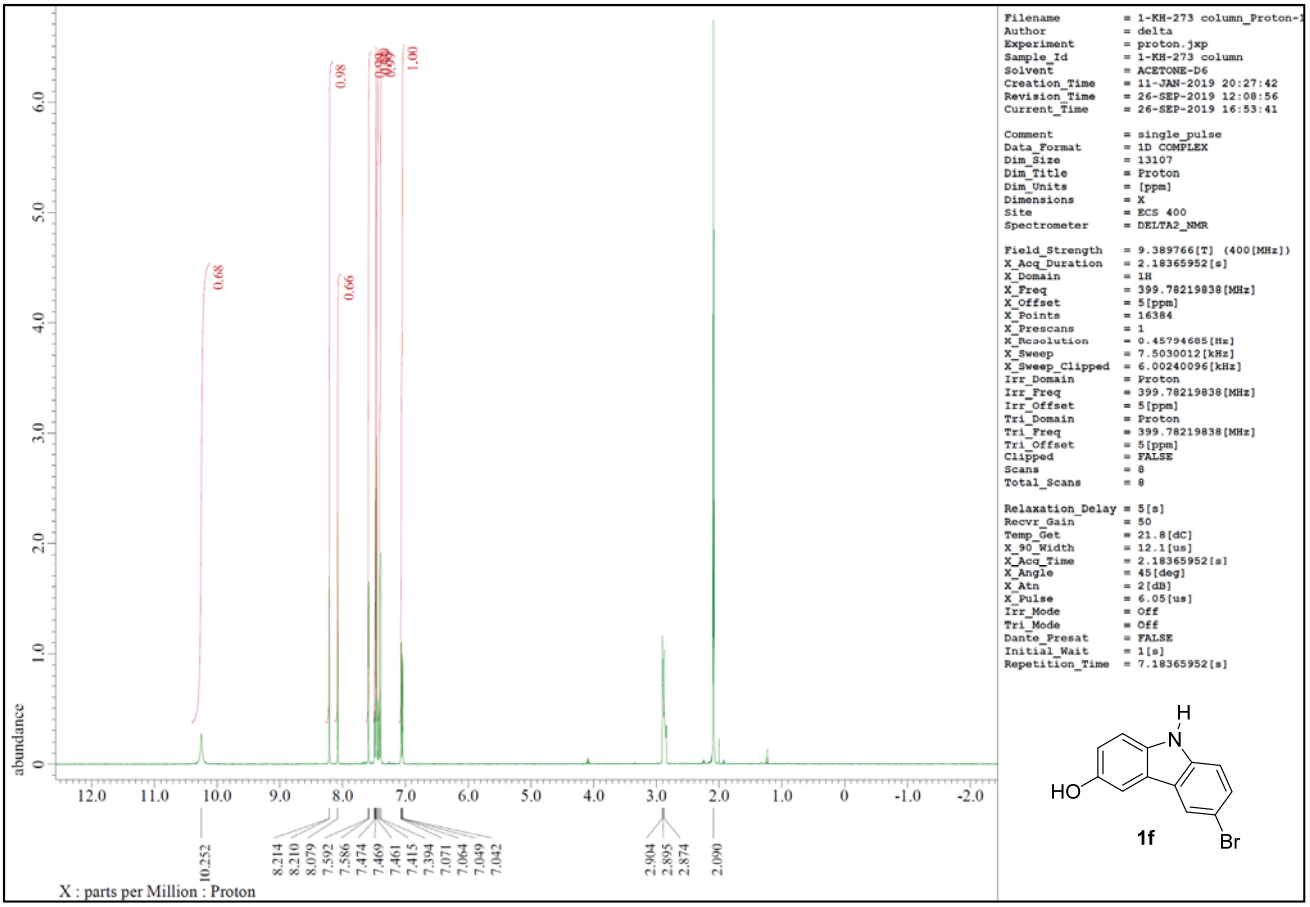
Compound **1d** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$, ^{13}C NMR, 100 MHz, $(CD_3)_2CO$ and ^{19}F NMR, 565 MHz, $(CD_3)_2CO$)

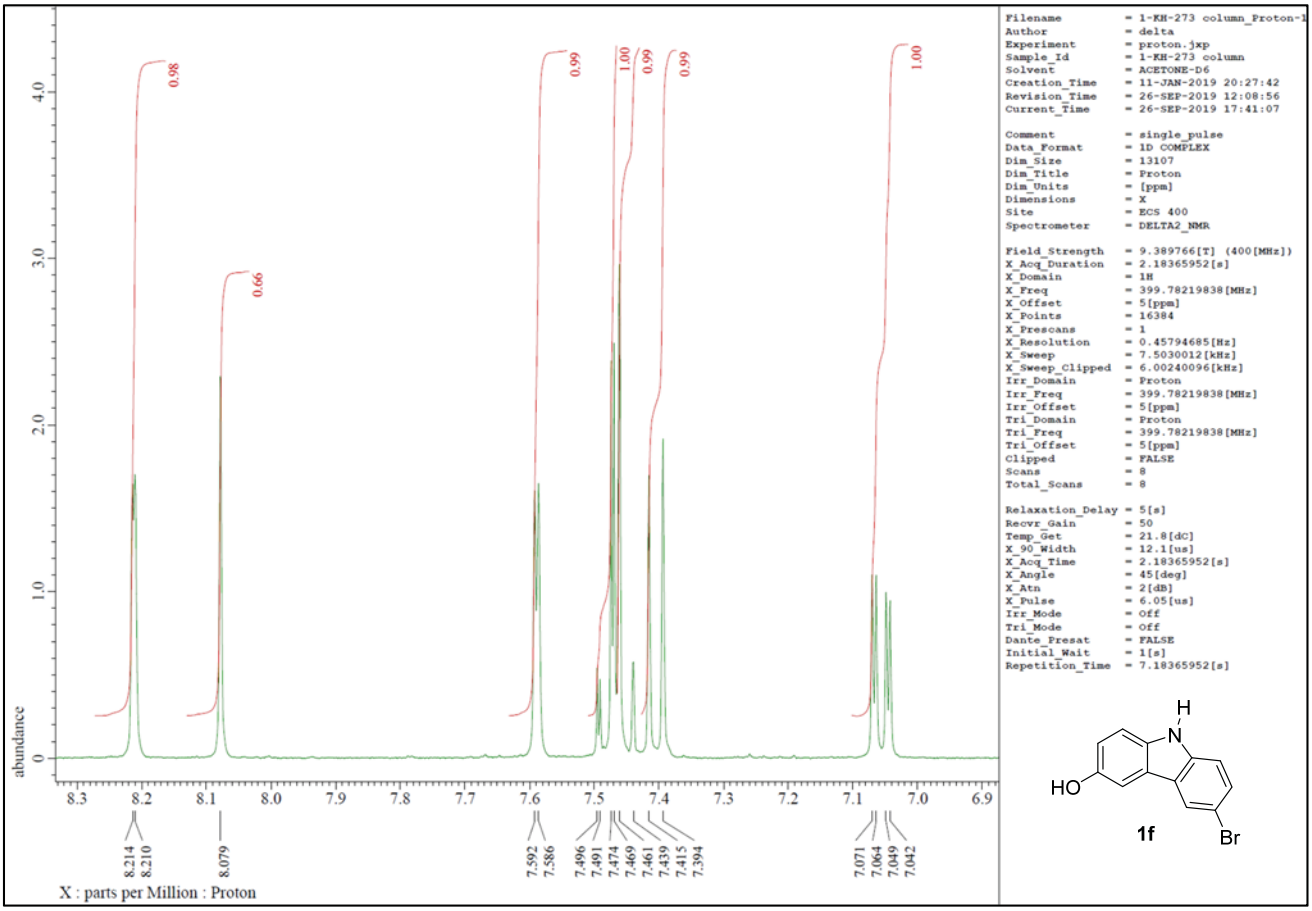

Filename = 2-KH-324 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-324 column
Solvent = ACETONE-D6
Creation_Time = 8-FEB-2019 17:58:33
Revision_Time = 26-SEP-2019 16:53:03
Current_Time = 26-SEP-2019 16:53:10
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.3[dC]
X_90_Width = 11[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.5[dB]
X_Pulse = 5.5[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
1e
HO
OH
H
N

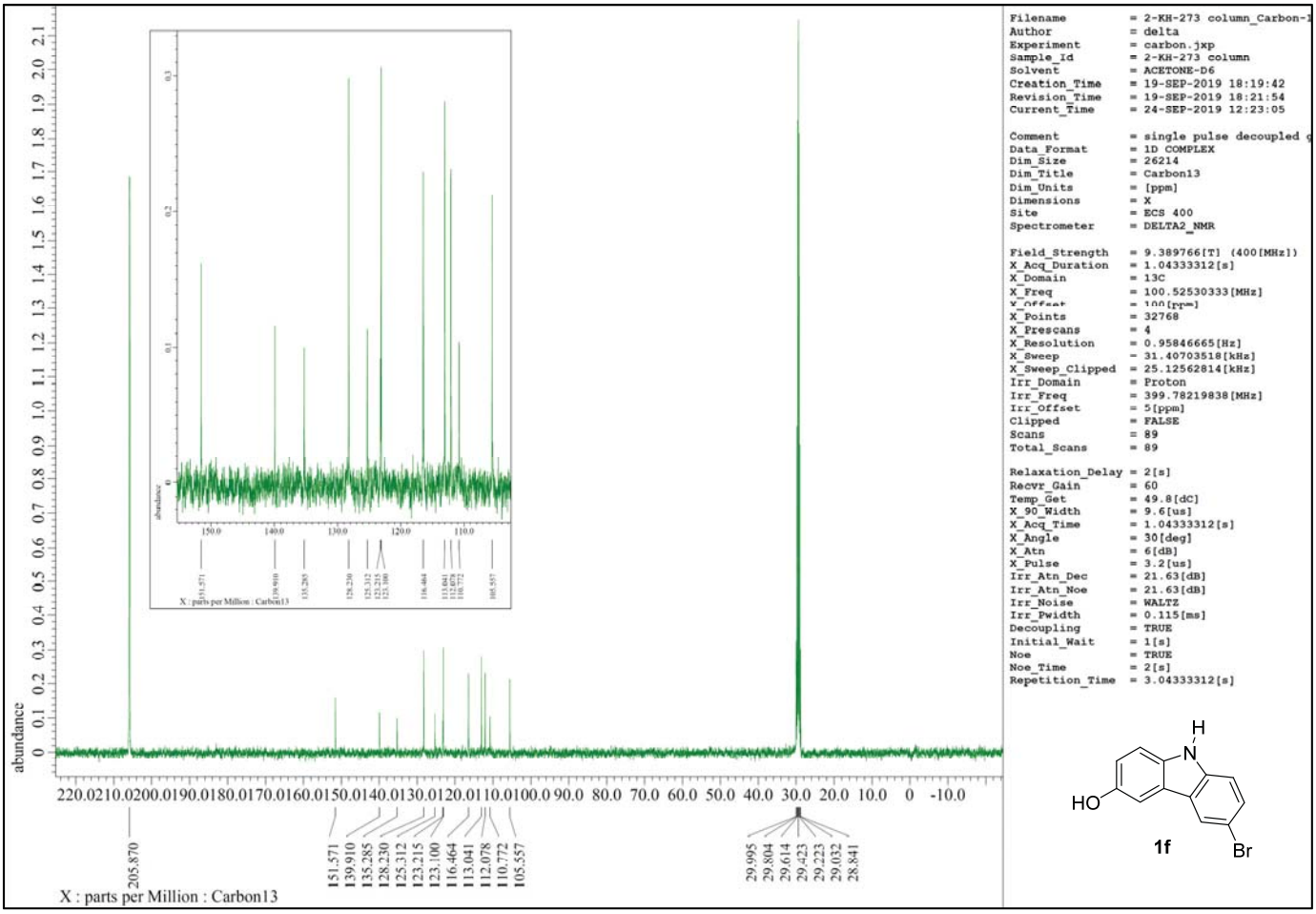

Filename = 2-KH-324 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-324 column
Solvent = ACETONE-D6
Creation_Time = 8-FEB-2019 17:58:33
Revision_Time = 26-SEP-2019 11:58:47
Current_Time = 26-SEP-2019 17:40:34
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.3[dC]
X_90_Width = 11[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.5[dB]
X_Pulse = 5.5[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
1e
HO
OH
H
N



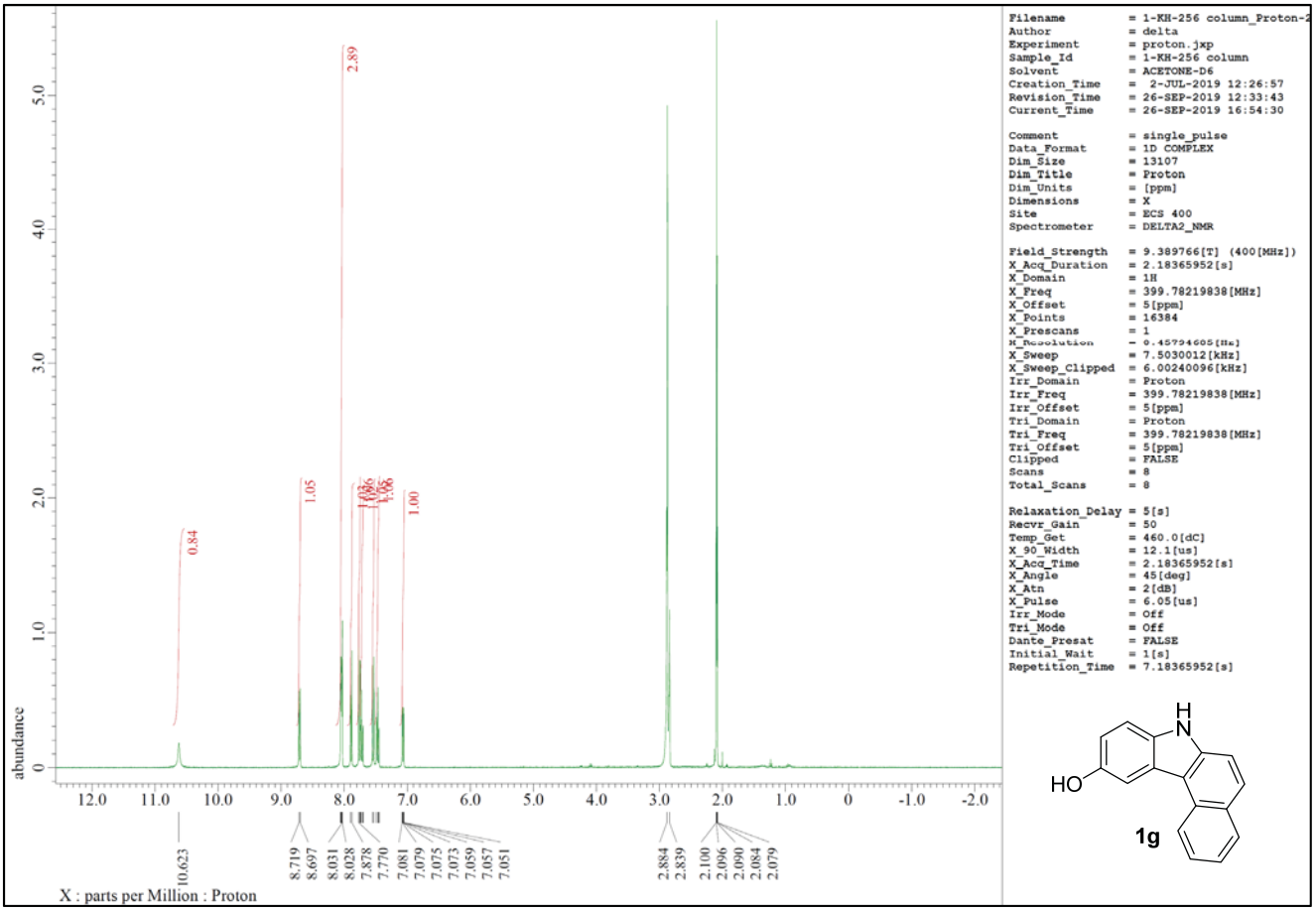
Compound **1e** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)

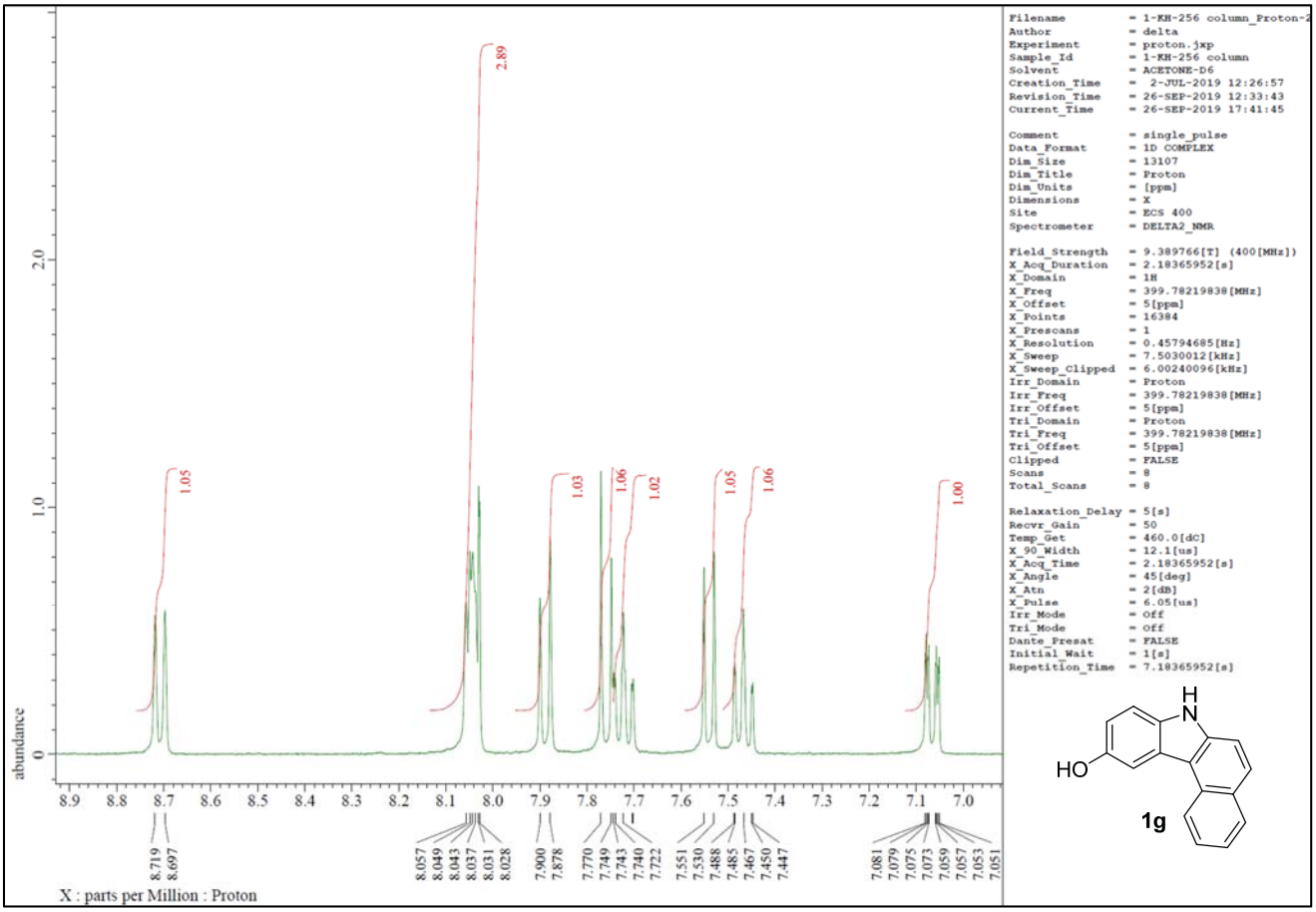

abundance
X : parts per Million : Proton
0.68
0.66
0.98
1.00
10.252
8.214
8.210
8.079
7.592
7.586
7.474
7.469
7.461
7.415
7.394
7.071
7.064
7.049
7.042
2.904
2.895
2.874
2.090
Filename = 1-KH-273 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 1-KH-273 column
Solvent = ACETONE-D6
Creation_Time = 11-JAN-2019 20:27:42
Revision_Time = 26-SEP-2019 12:08:56
Current_Time = 26-SEP-2019 16:53:41
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 21.8[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
Br
1f

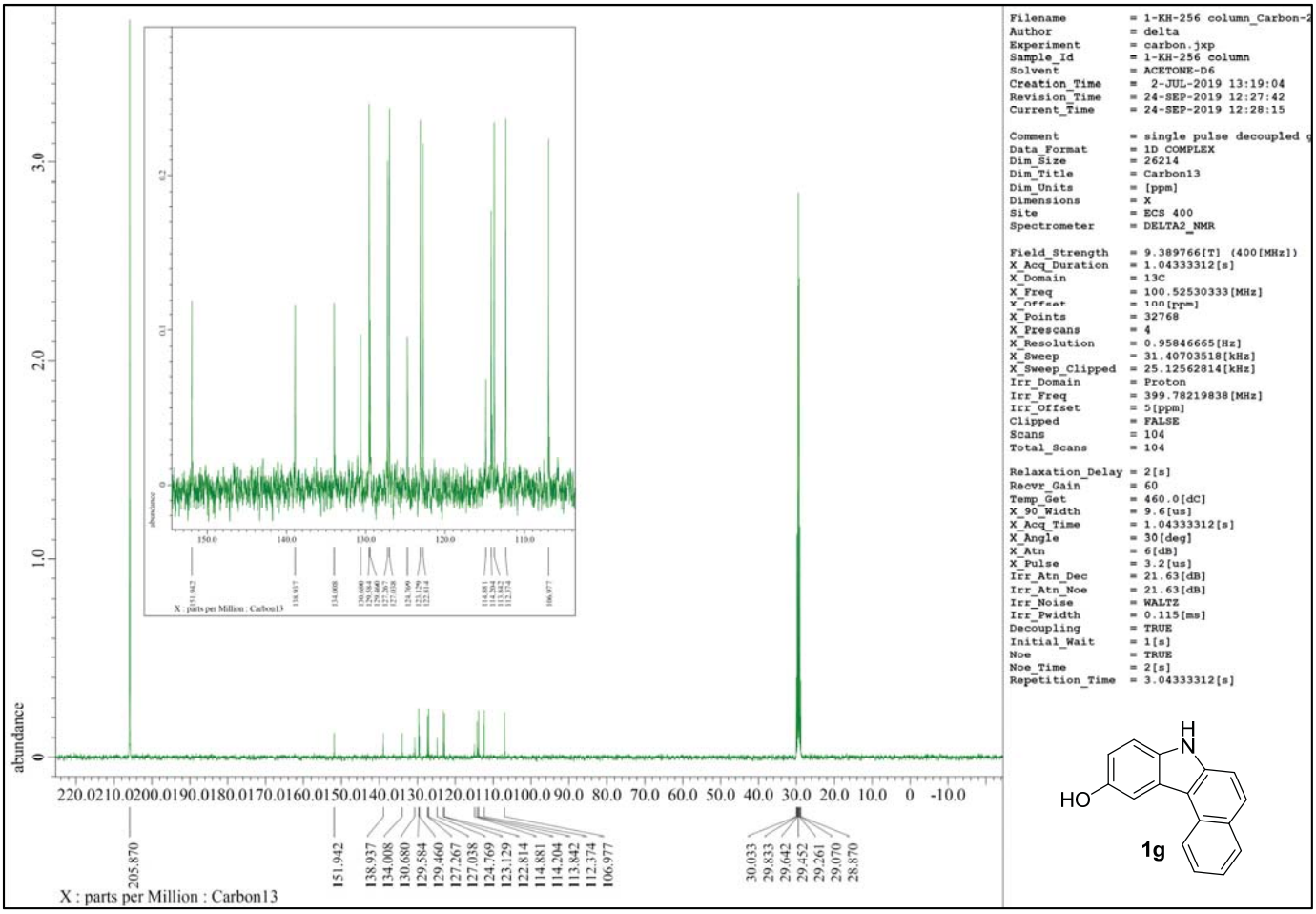

abundance
X : parts per Million : Proton
0.98
0.66
0.99
1.00
0.99
0.99
1.00
8.214
8.210
8.079
7.592
7.586
7.496
7.491
7.474
7.469
7.461
7.439
7.415
7.394
7.071
7.064
7.049
7.042
Filename = 1-KH-273 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 1-KH-273 column
Solvent = ACETONE-D6
Creation_Time = 11-JAN-2019 20:27:42
Revision_Time = 26-SEP-2019 12:08:56
Current_Time = 26-SEP-2019 17:41:07
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 21.8[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
Br
1f



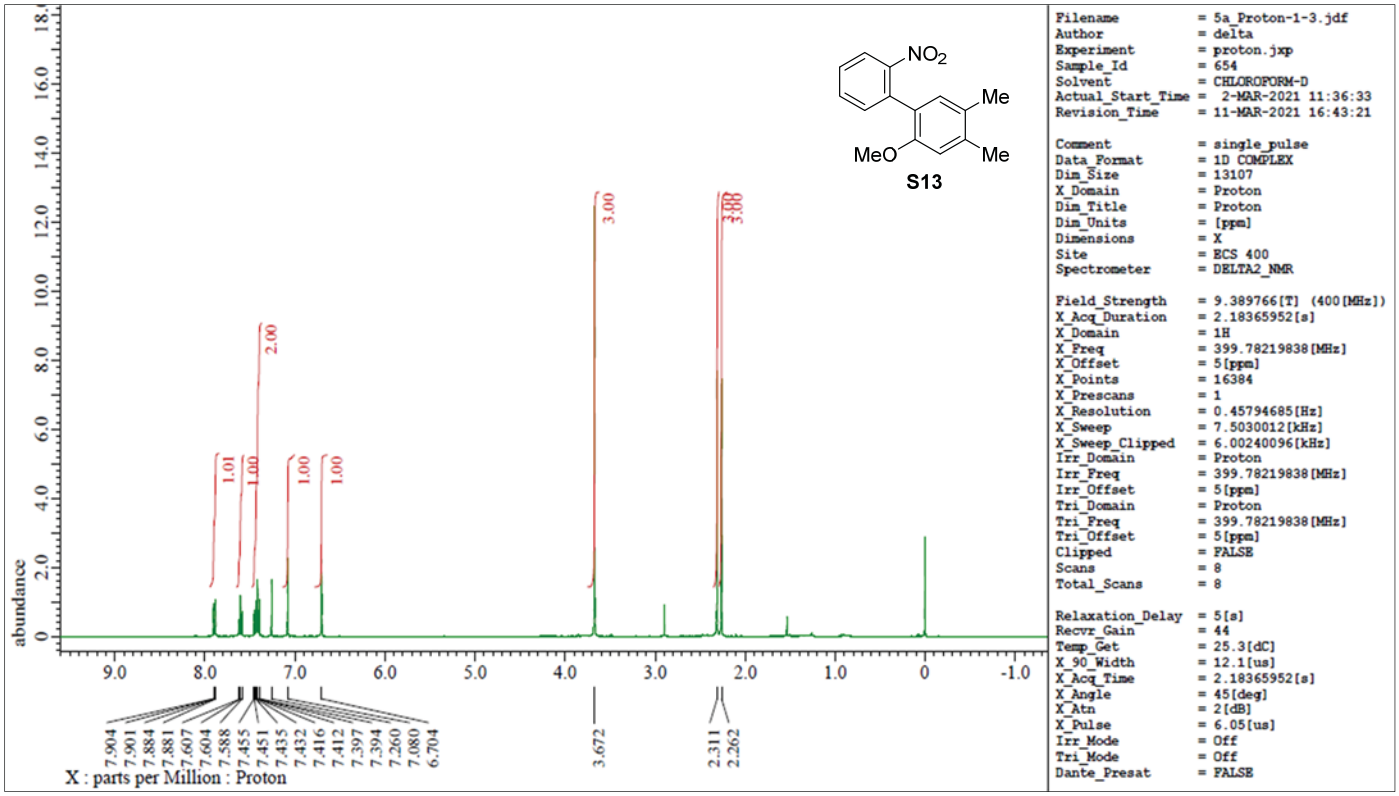
Compound **1f** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)

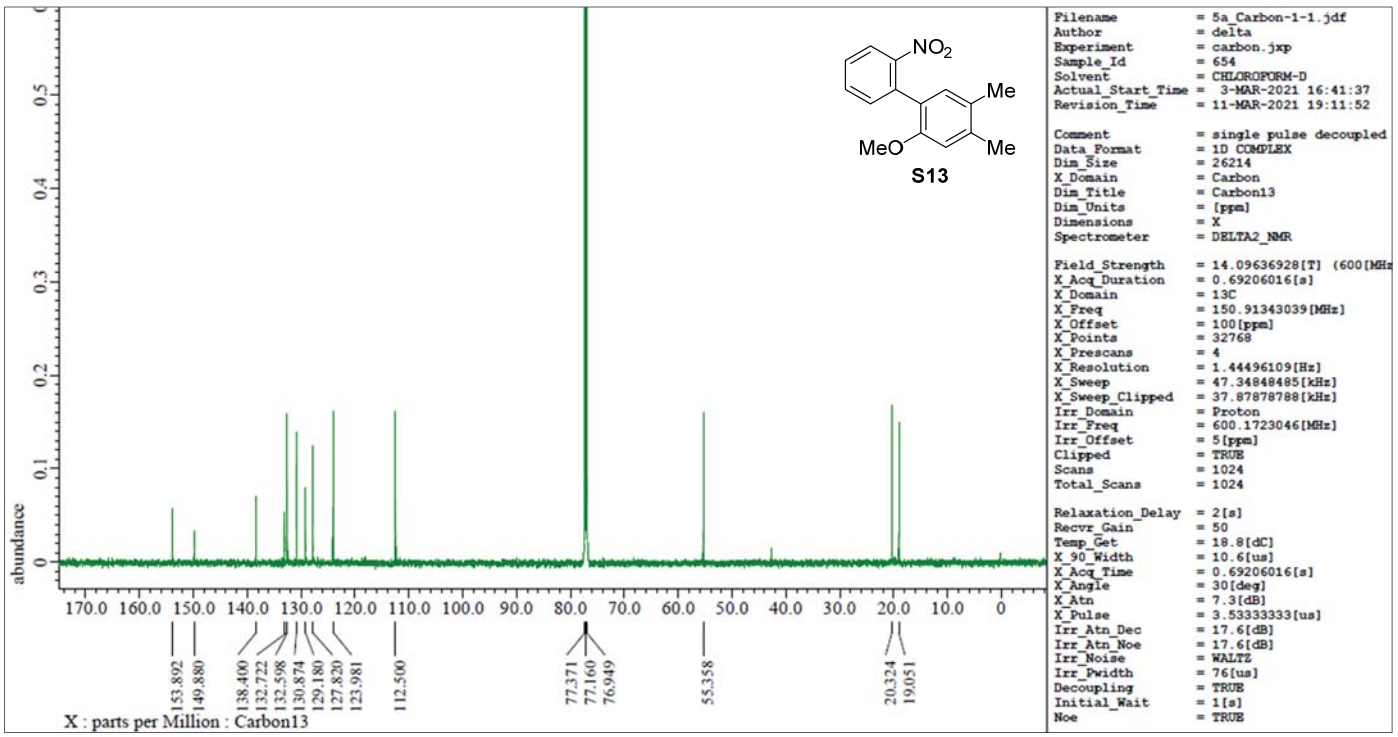

abundance
X : parts per Million : Proton
2.89
1.05
1.00
0.84
10.623
8.719
8.697
8.031
8.028
7.878
7.770
7.081
7.079
7.075
7.073
7.059
7.057
7.051
2.884
2.839
2.100
2.096
2.090
2.084
2.079
Filename = 1-KH-256 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 1-KH-256 column
Solvent = ACETONE-D6
Creation_Time = 2-JUL-2019 12:26:57
Revision_Time = 26-SEP-2019 12:33:43
Current_Time = 26-SEP-2019 16:54:30
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794605[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
HO
H
N
1g


abundance
X : parts per Million : Proton
2.89
1.05
1.03
1.06
1.02
1.05
1.06
1.00
8.719
8.697
8.057
8.049
8.043
8.037
8.031
8.028
7.900
7.878
7.770
7.749
7.743
7.740
7.722
7.551
7.530
7.488
7.485
7.467
7.450
7.447
7.081
7.079
7.075
7.073
7.059
7.057
7.053
7.051
Filename = 1-KH-256 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 1-KH-256 column
Solvent = ACETONE-D6
Creation_Time = 2-JUL-2019 12:26:57
Revision_Time = 26-SEP-2019 12:33:43
Current_Time = 26-SEP-2019 17:41:45
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
HO
H
N
1g

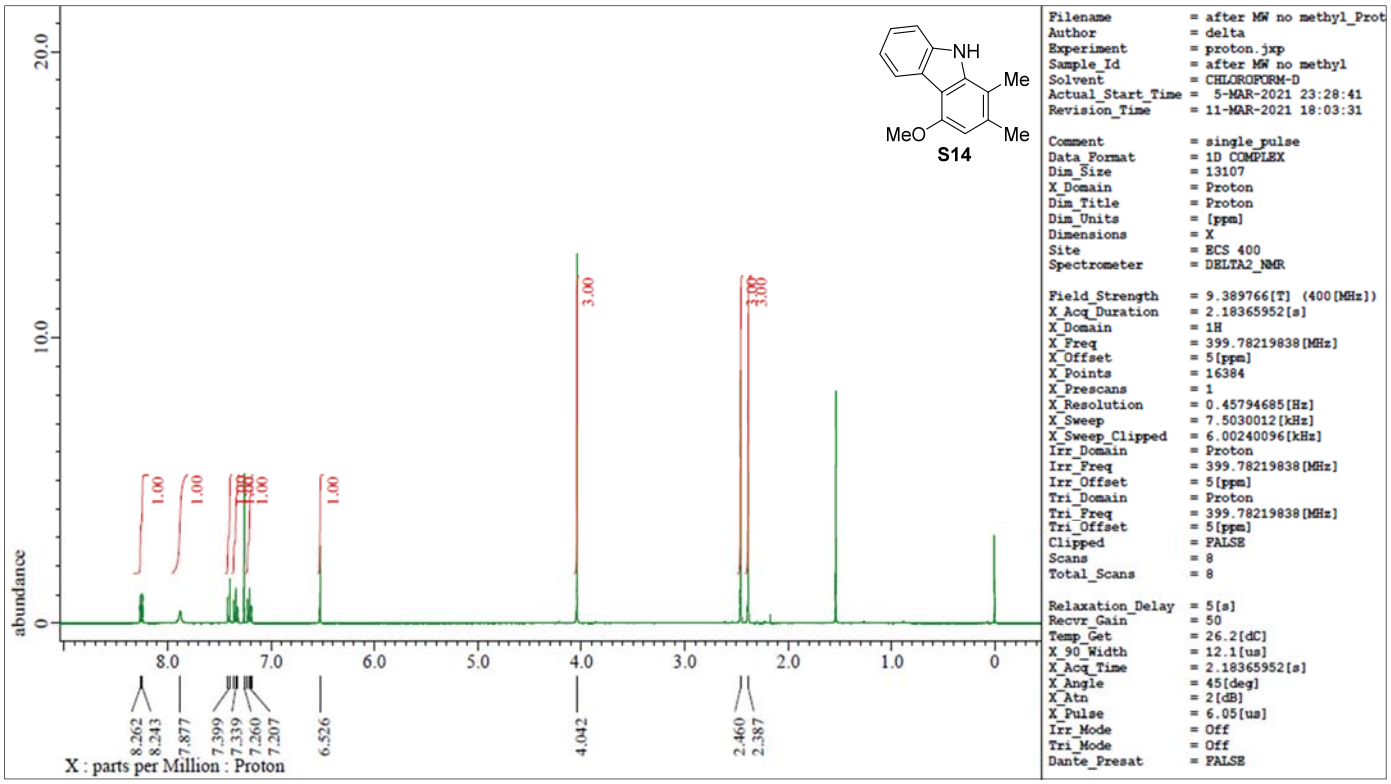


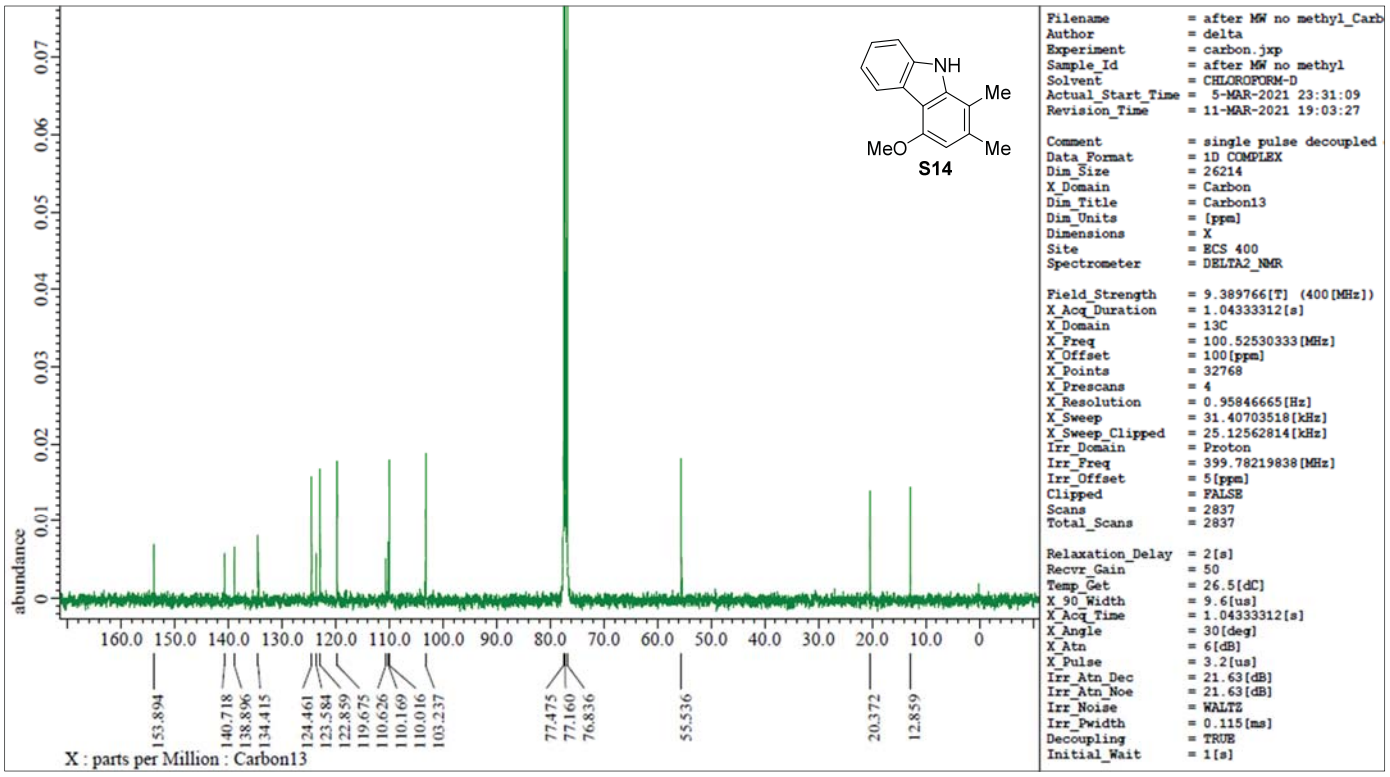
Compound **1g** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)



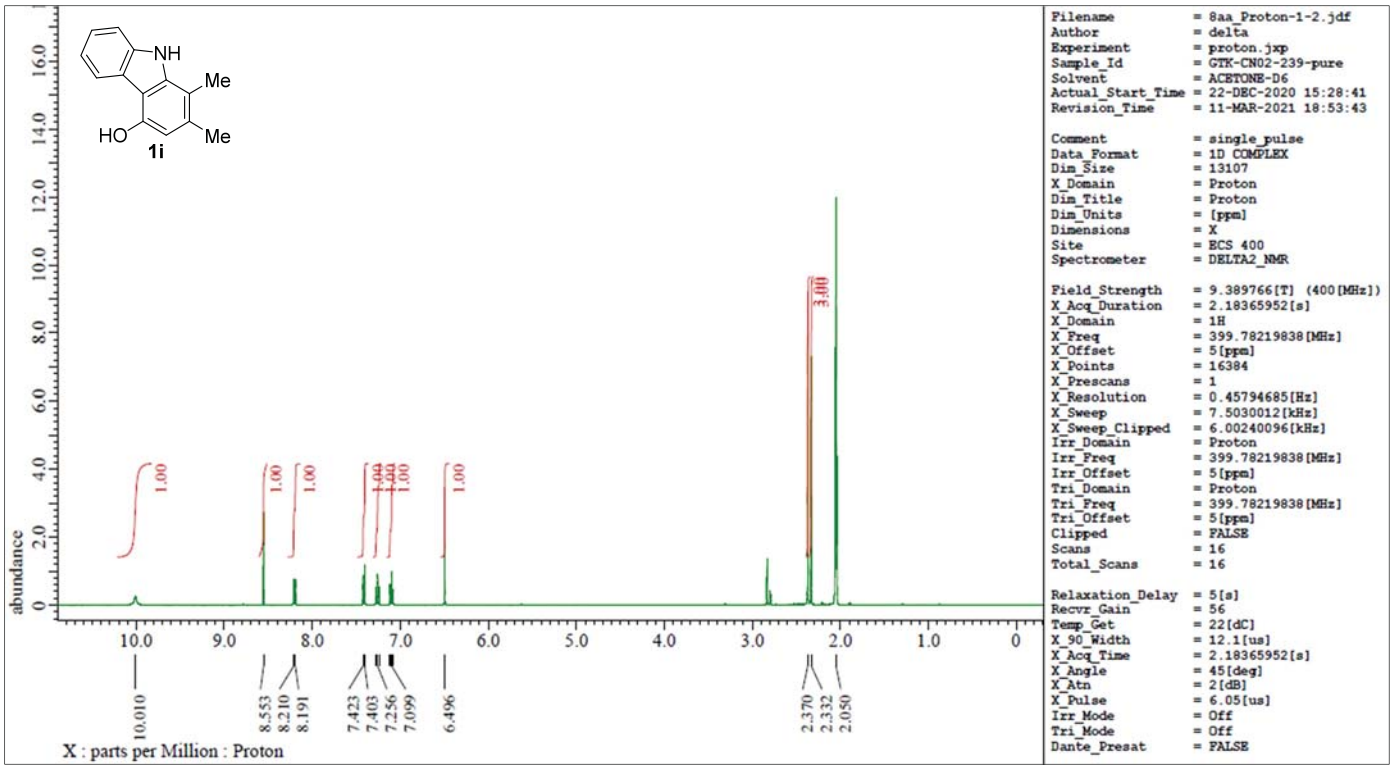


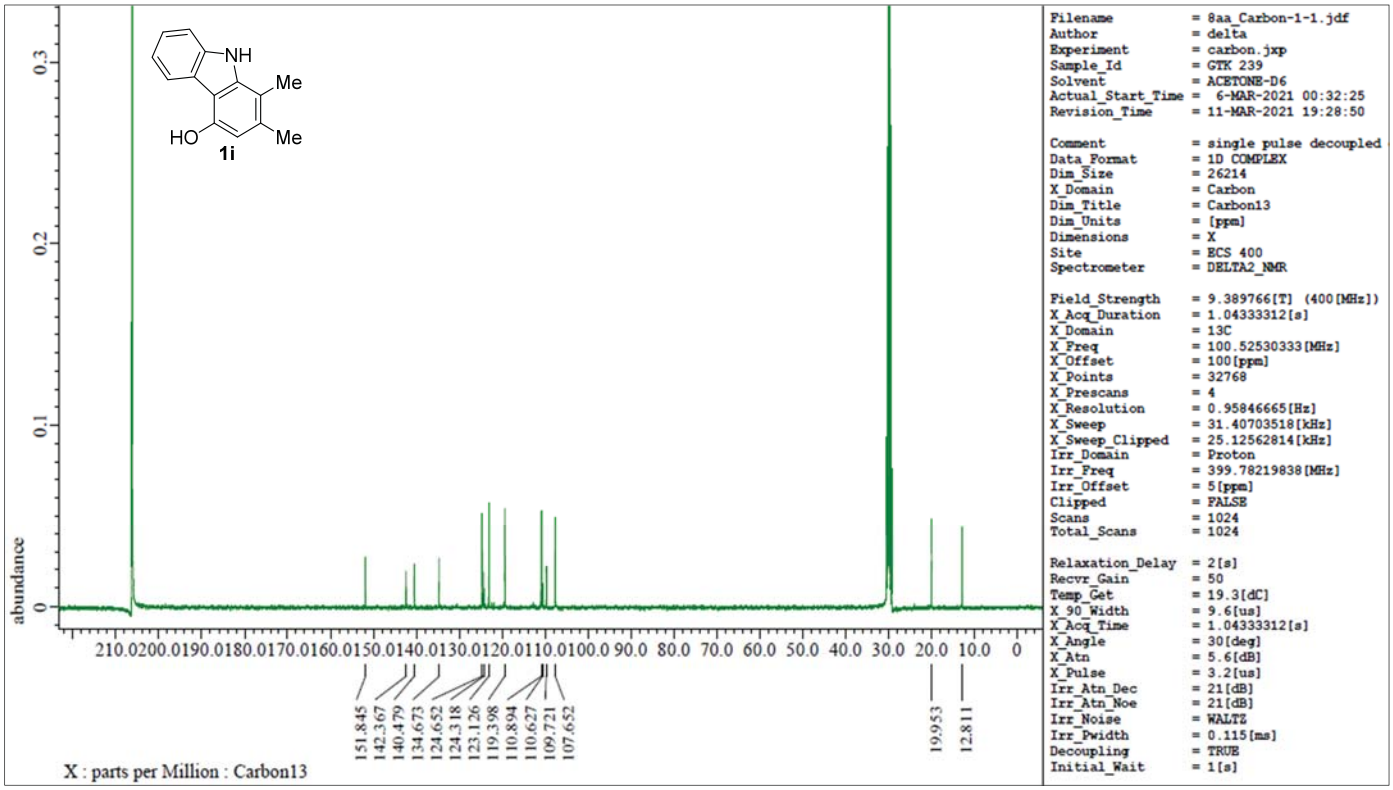
Compound **S13** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)



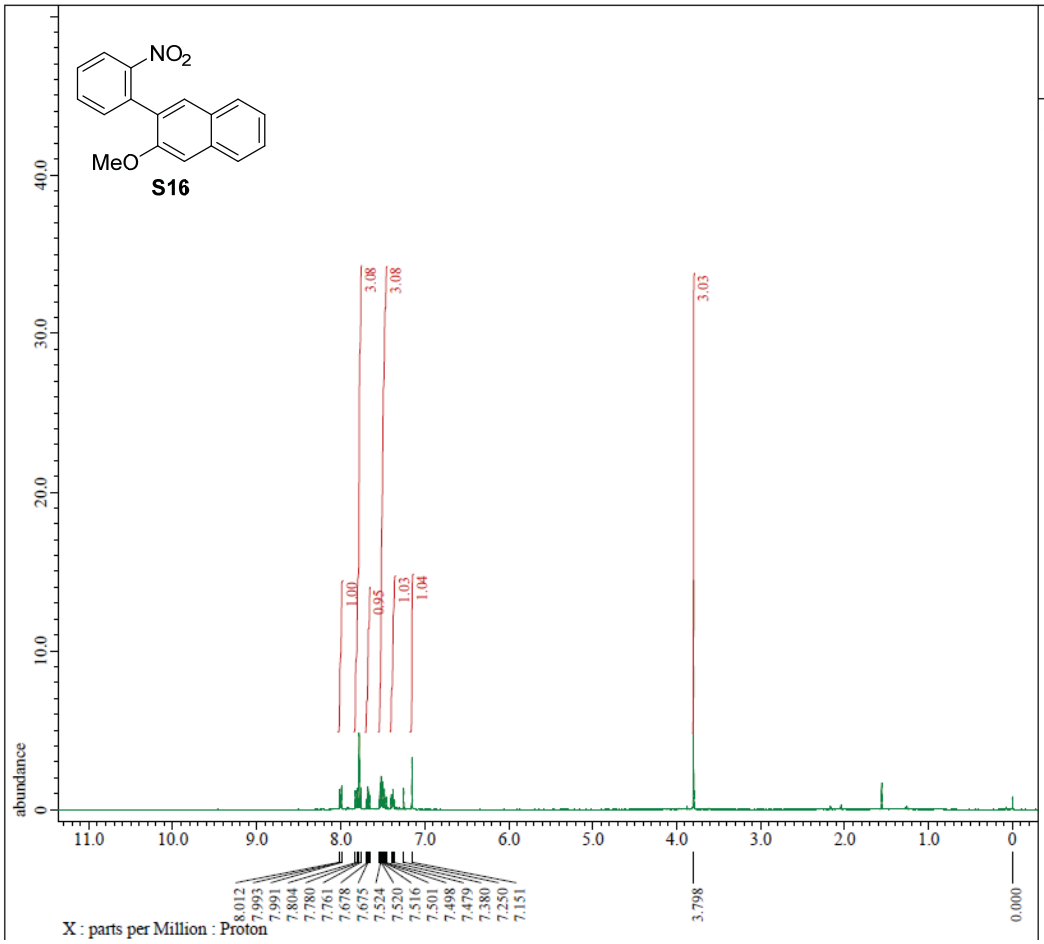


Compound **S14** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)





Compound **1i** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)

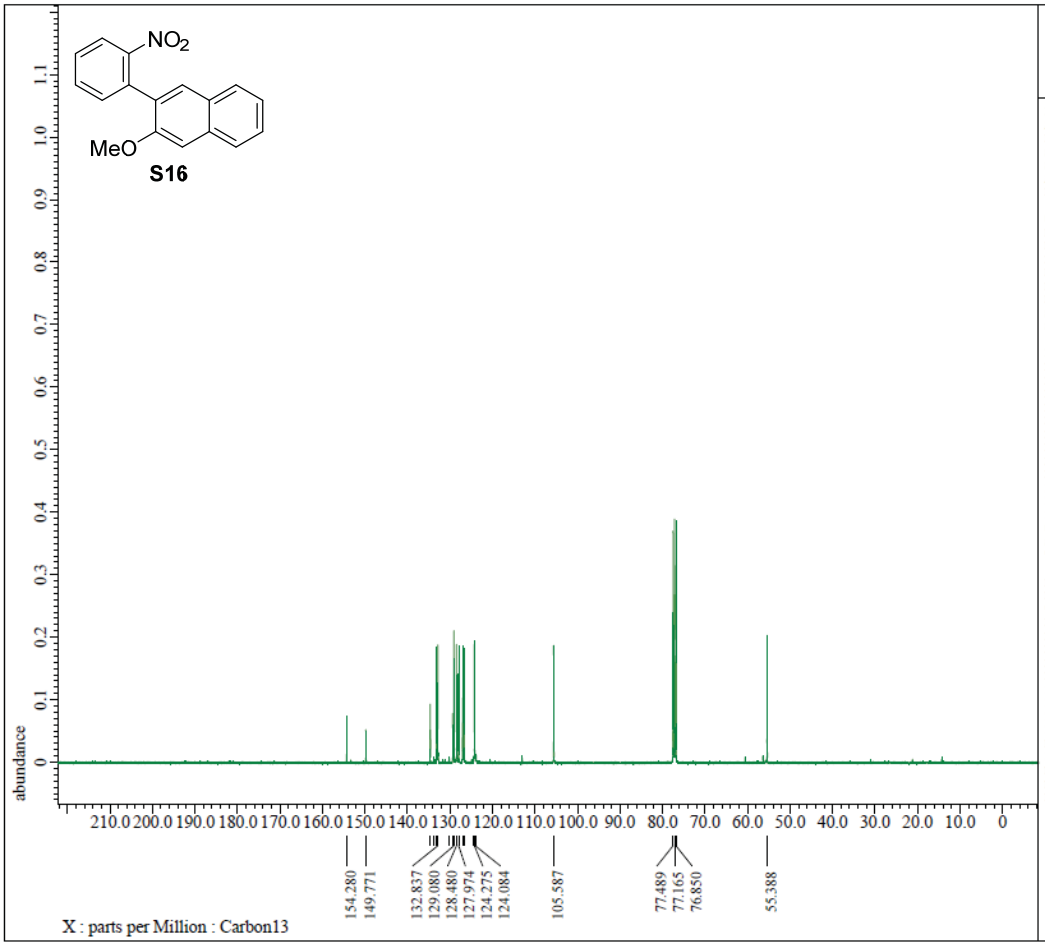



```
Filename          = ankit.exp-532-pure_Proton
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ankit.exp-532-pure
Solvent           = CHLOROFORM-D
Actual_Start_Time = 29-MAR-2021 18:10:28
Revision_Time     = 30-MAR-2021 10:37:17

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proton
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 18.6[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



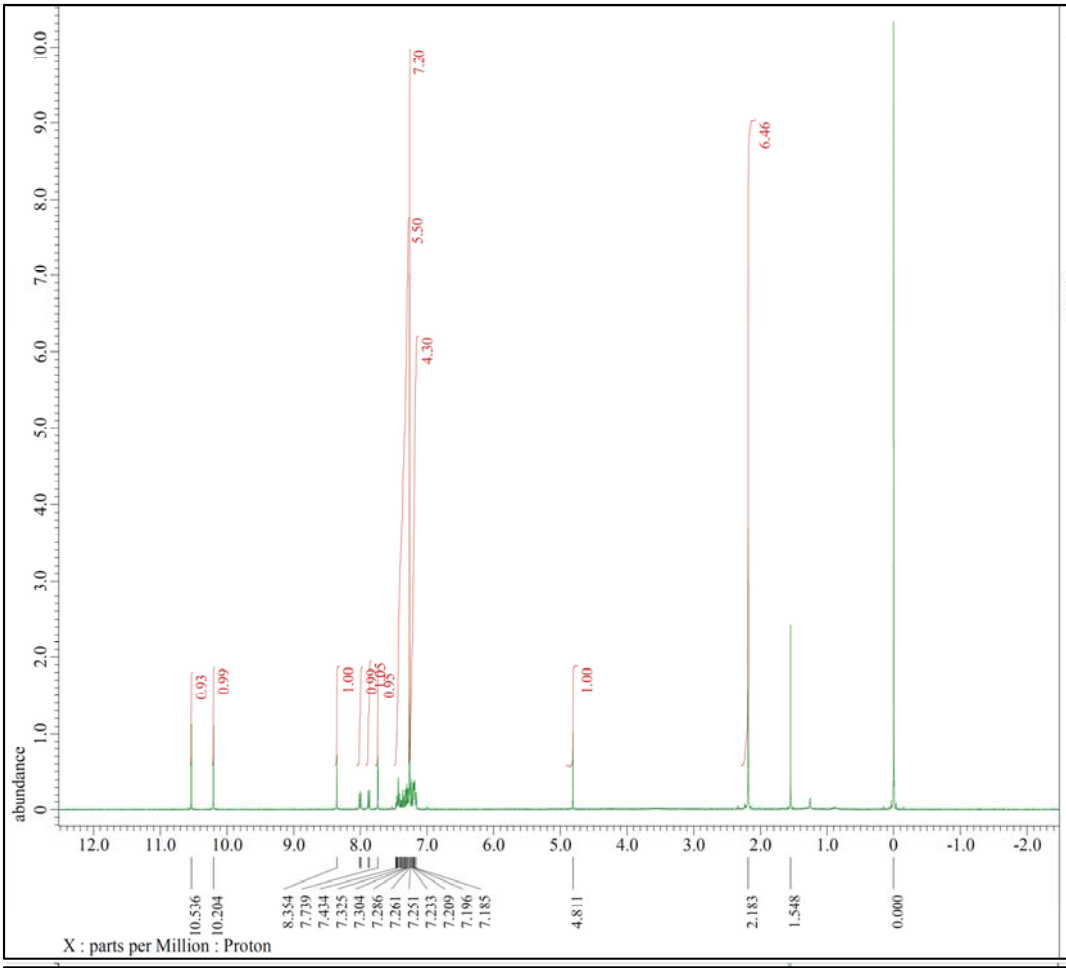

```
Filename          = ankit.exp-532-pure-13C_Ca
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ankit.exp-532-pure-13C
Solvent           = CHLOROFORM-D
Actual_Start_Time = 29-MAR-2021 22:07:05
Revision_Time     = 30-MAR-2021 10:40:12

Comment           = single pulse decoupled ga
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbon
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 18.7[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```

Compound **S16** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

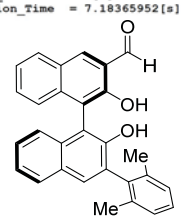



```
Filename          = 2-KH-293 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_id         = 2-KH-293 column
Solvent           = CHLOROFORM-D
Creation_Time     = 19-JAN-2019 18:41:14
Revision_Time     = 24-SEP-2019 15:36:13
Current_Time      = 24-SEP-2019 15:36:20

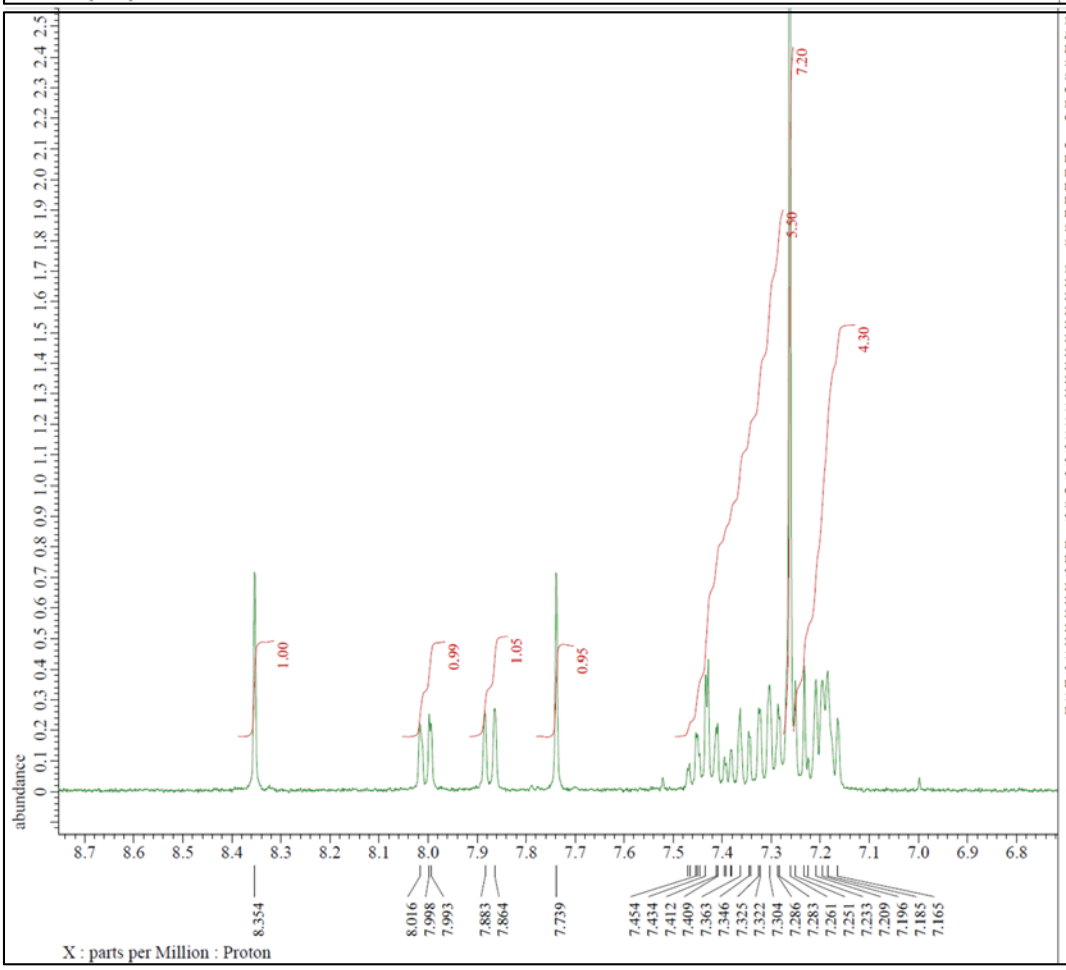
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 18.3[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



S19

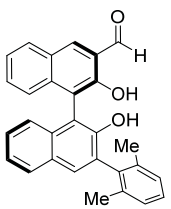



```
Filename          = 2-KH-293 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-293 column
Solvent           = CHLOROFORM-D
Creation_Time     = 19-JAN-2019 18:41:14
Revision_Time     =  2-JUL-2019 21:32:11
Current_Time      = 26-SEP-2019 18:01:42

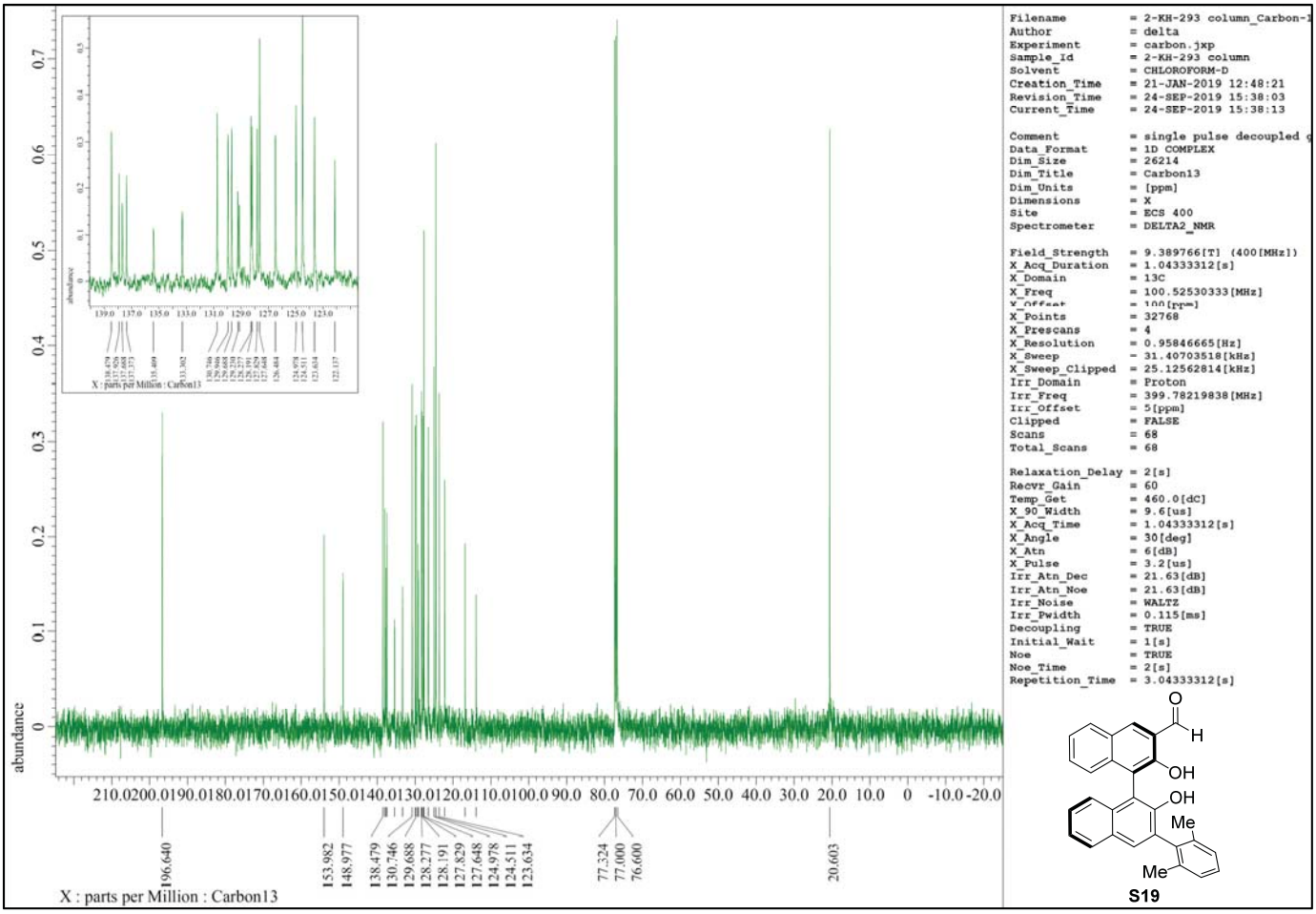
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

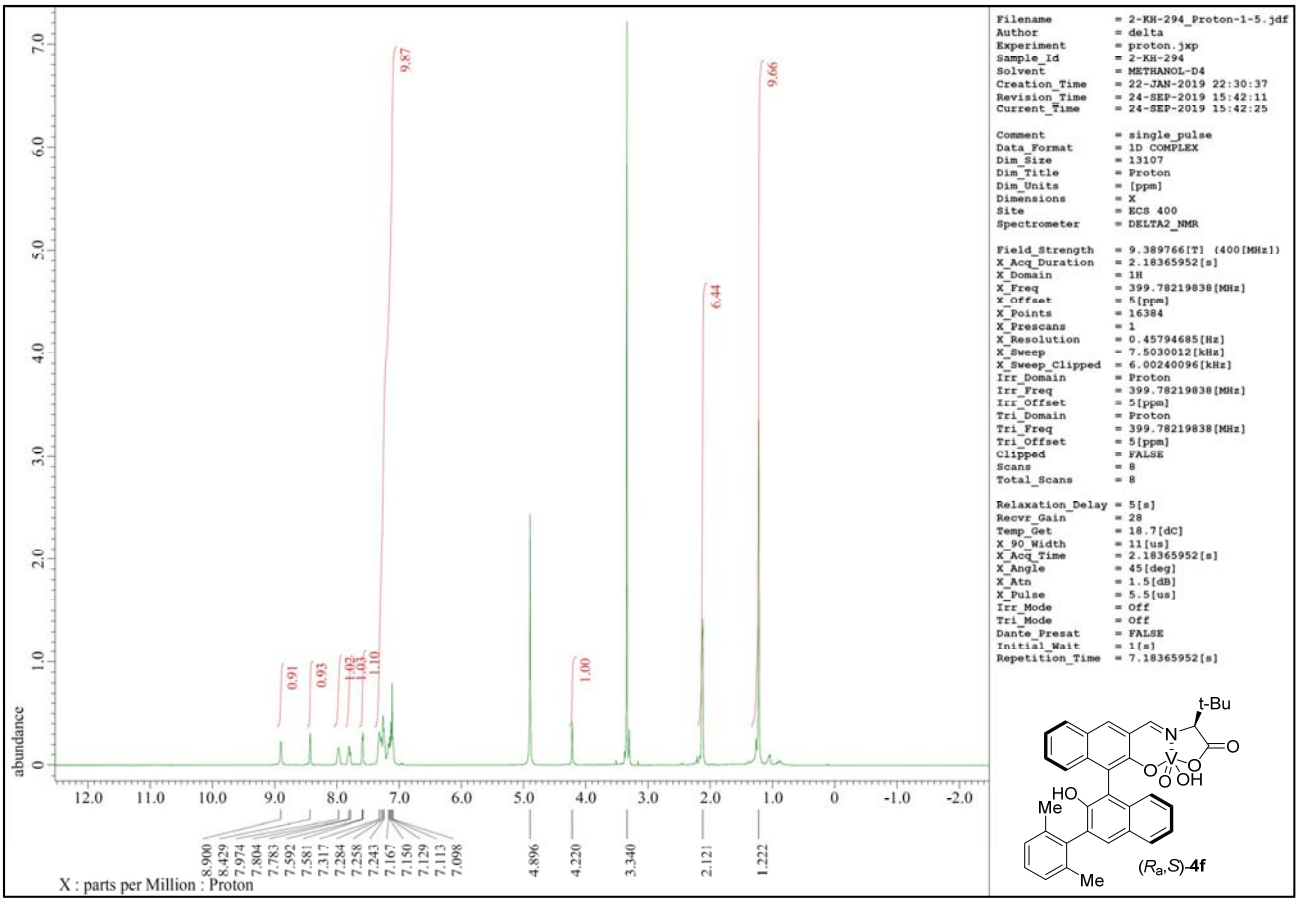
Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 18.3[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

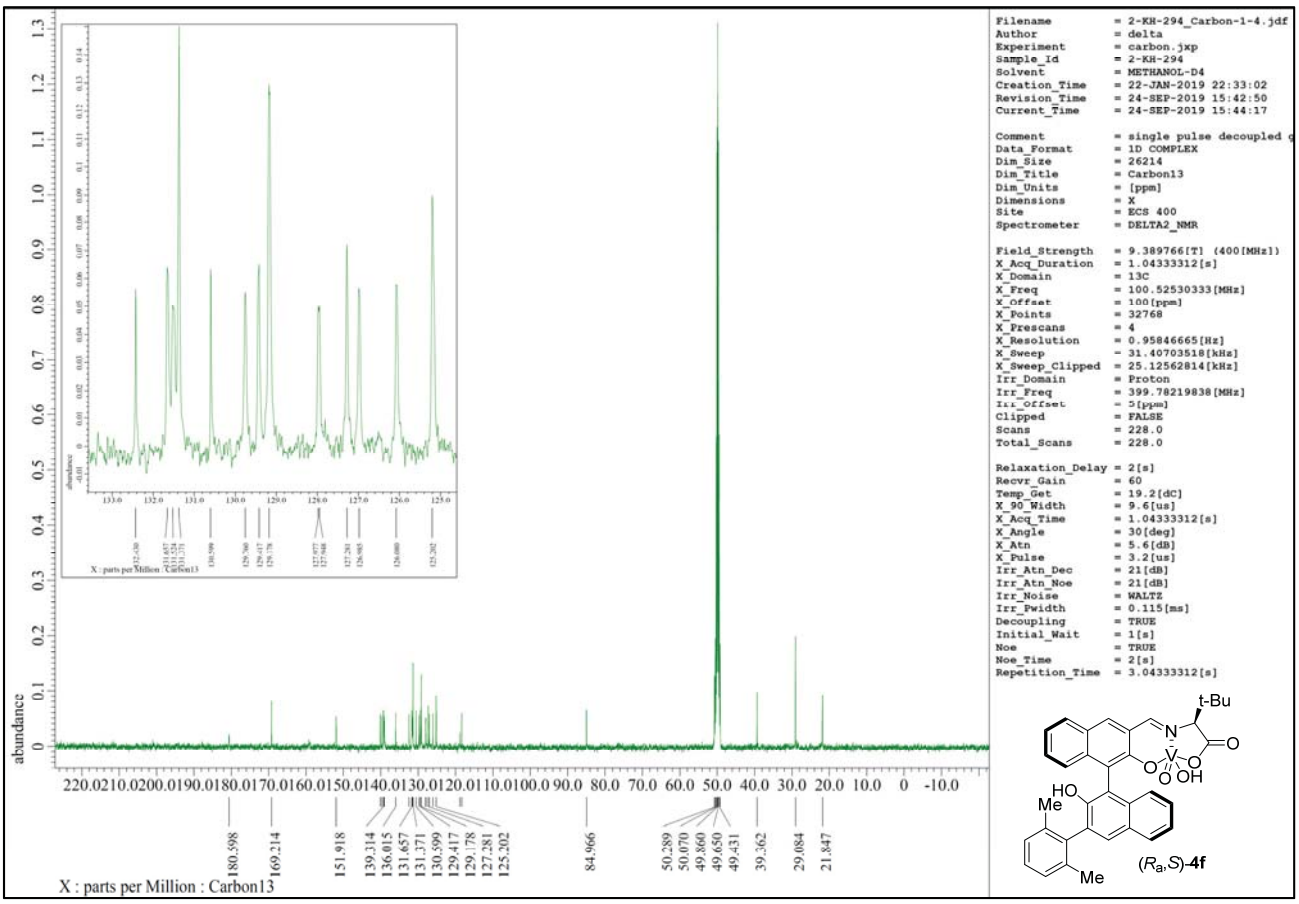


S19

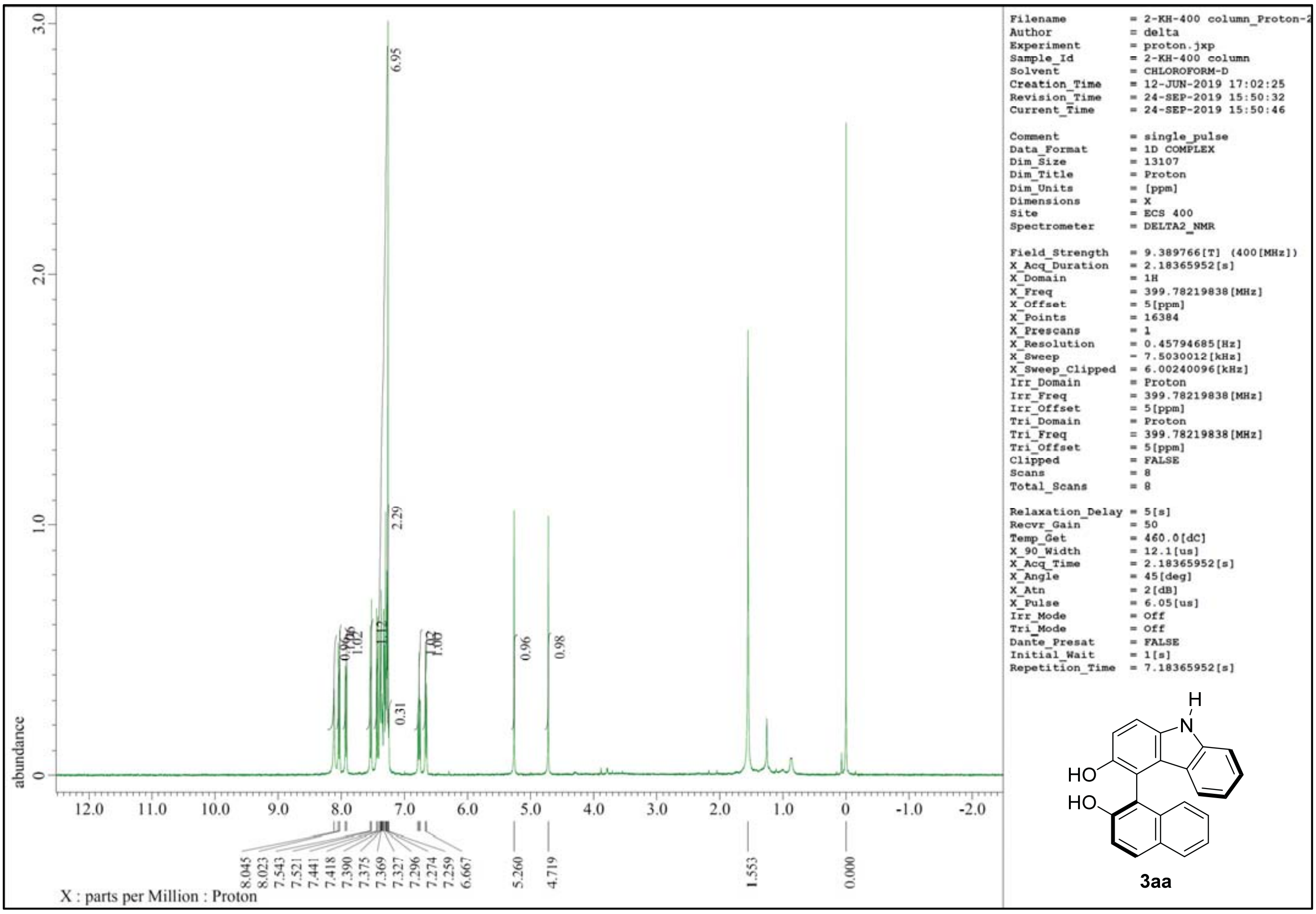


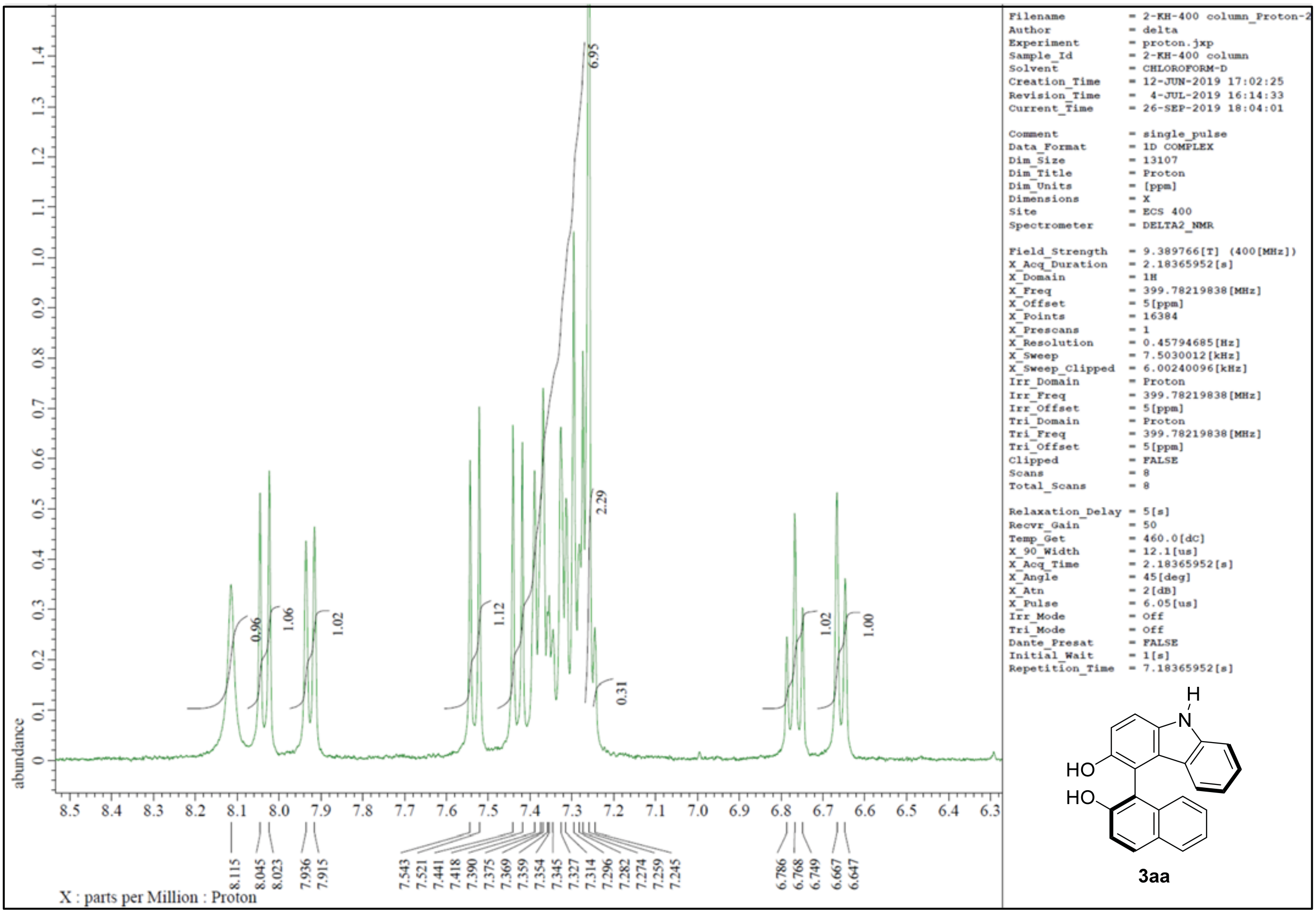
Compound **S19** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

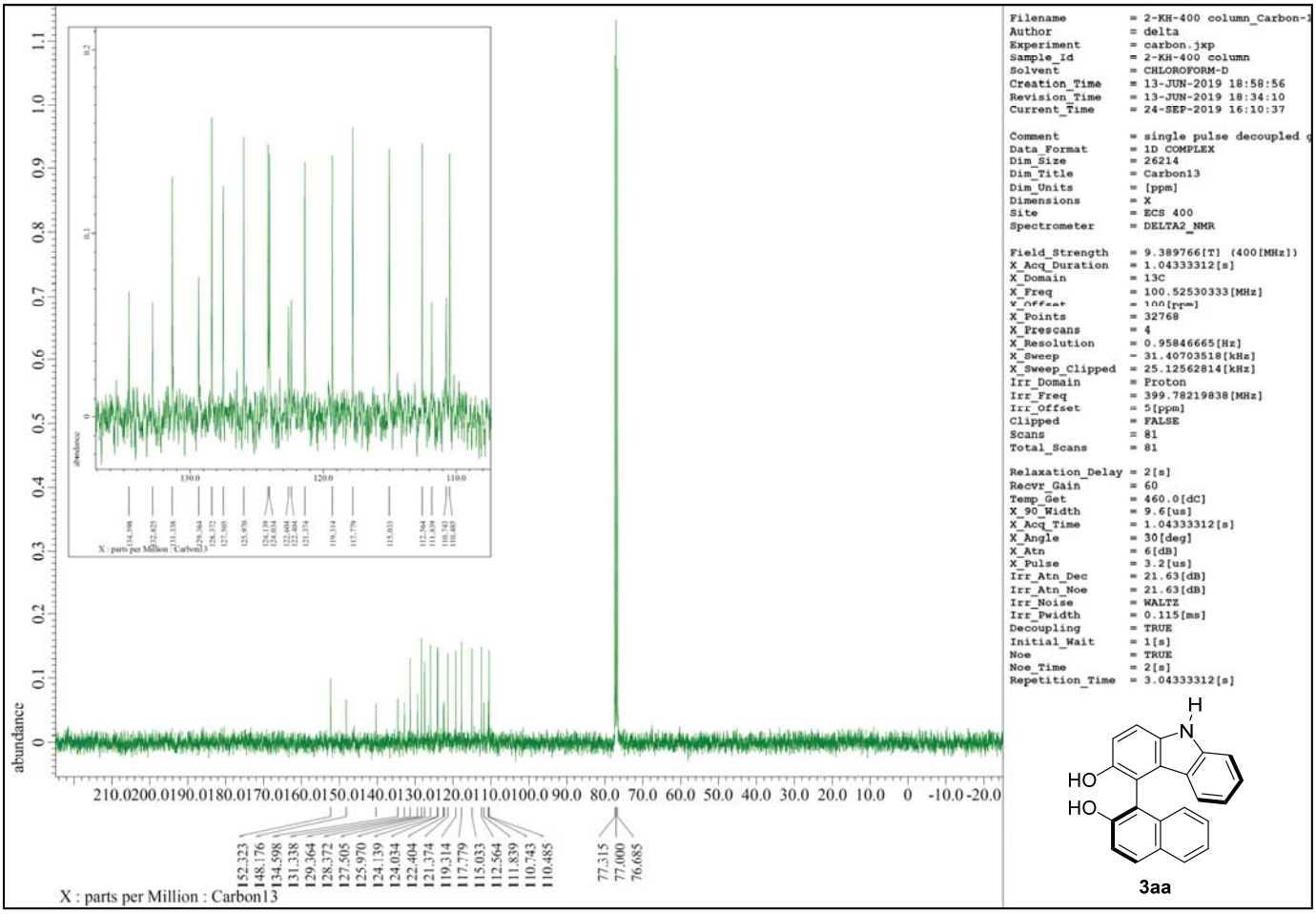




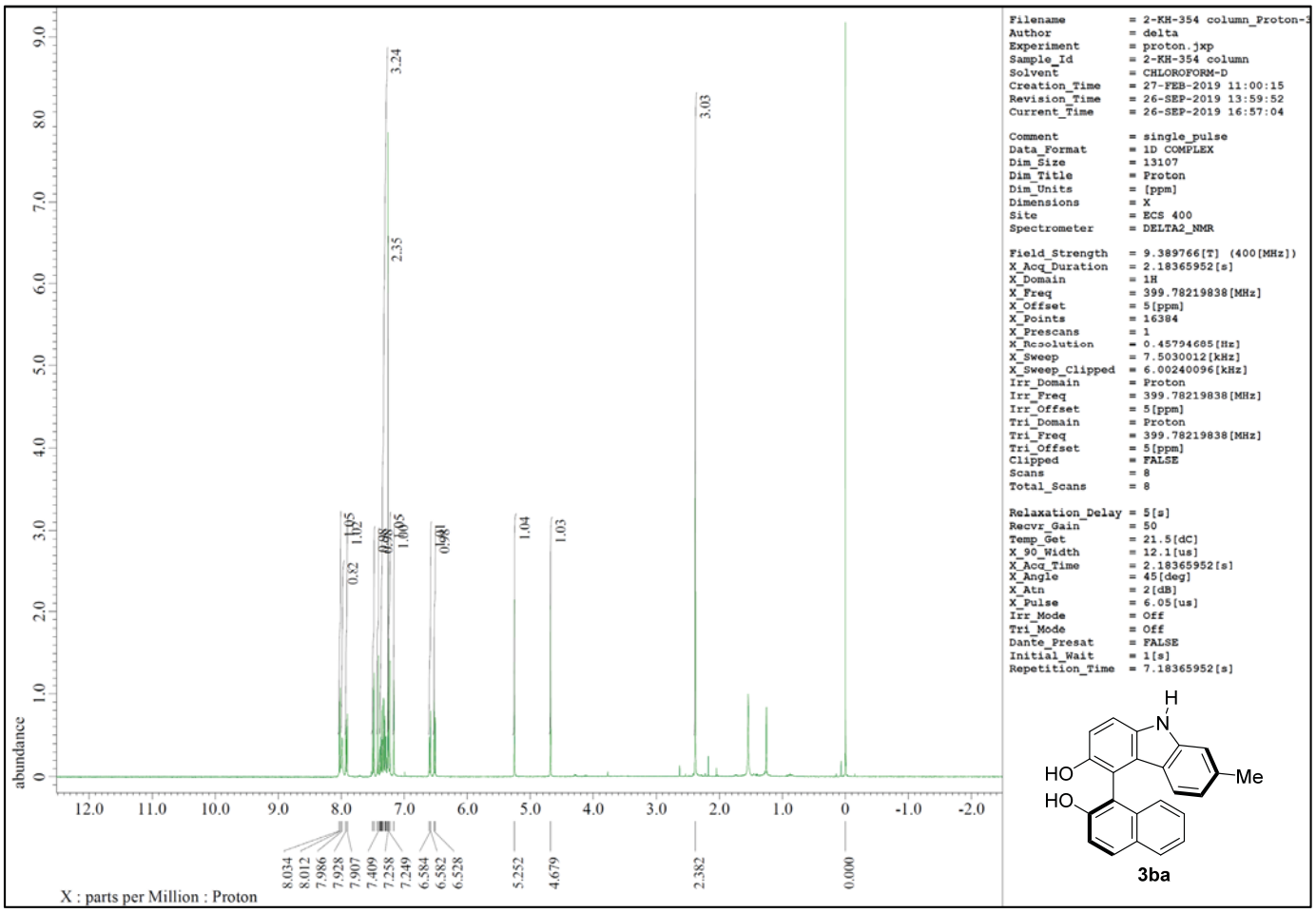
Compound (R_a,*S*)-**4f** (^{1}H NMR, 400 MHz, CD_3OD and ^{13}C NMR, 100 MHz, CD_3OD)

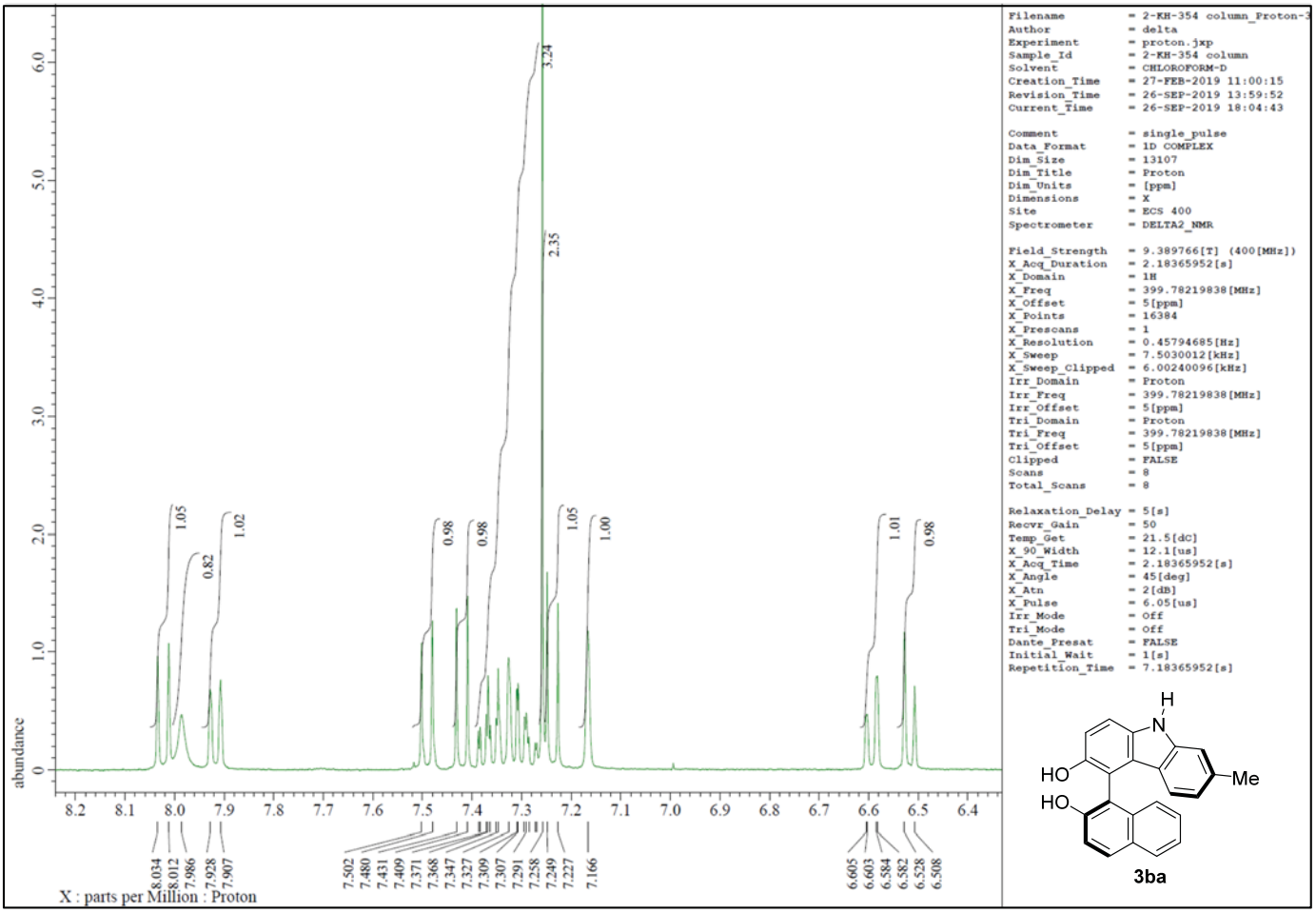

Filename = 2-KH-400 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-400 column
Solvent = CHLOROFORM-D
Creation_Time = 12-JUN-2019 17:02:25
Revision_Time = 24-SEP-2019 15:50:32
Current_Time = 24-SEP-2019 15:50:46
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
8.045 8.023 7.543 7.521 7.441 7.418 7.390 7.375 7.369 7.327 7.296 7.274 7.259 6.667 5.260 4.719 1.553 0.000
6.95 2.29 1.12 1.02 0.96 0.31 1.02 1.00 0.96 0.98
HO
HO
H
N
3aa

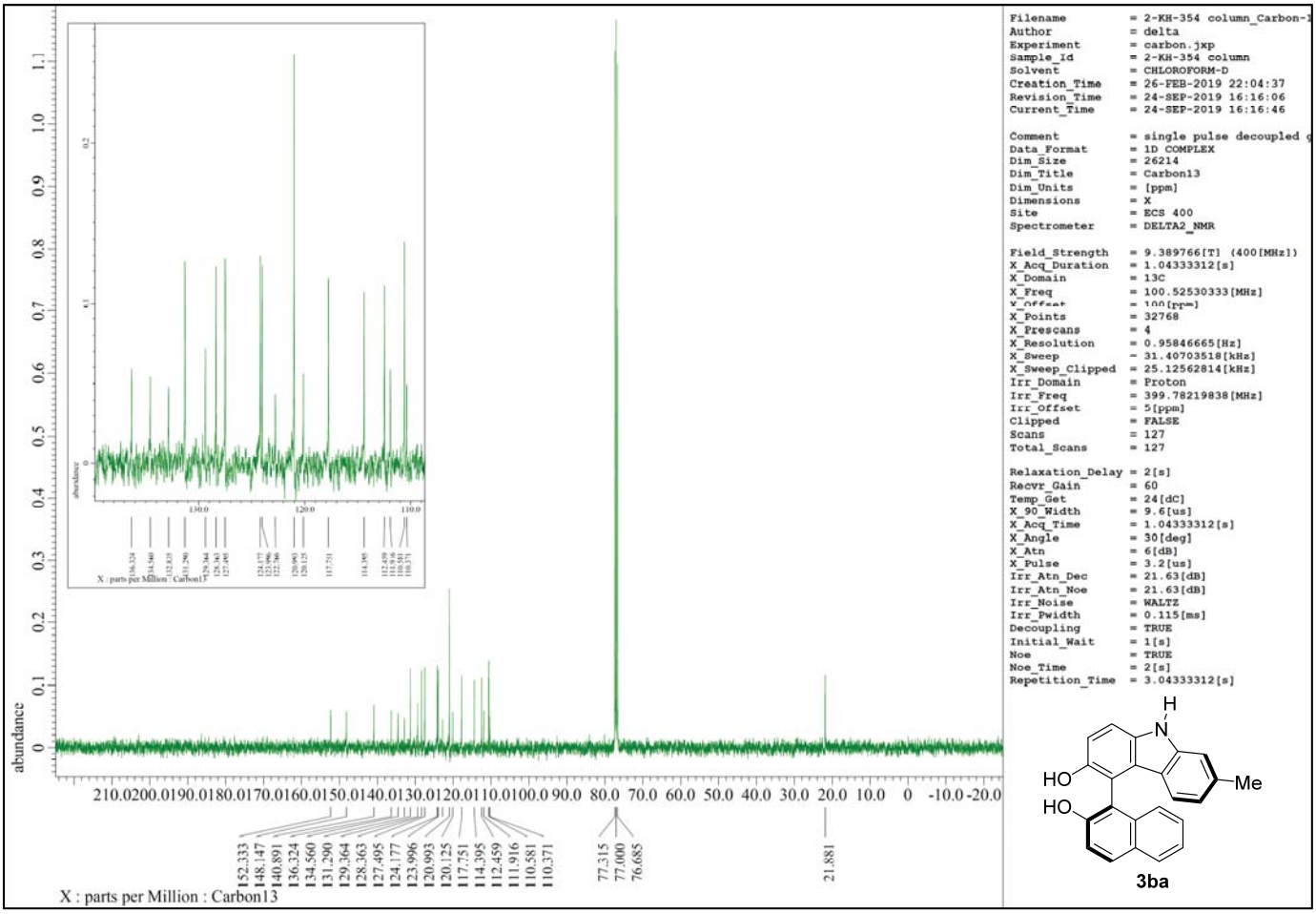

Filename = 2-KH-400 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-400 column
Solvent = CHLOROFORM-D
Creation_Time = 12-JUN-2019 17:02:25
Revision_Time = 4-JUL-2019 16:14:33
Current_Time = 26-SEP-2019 18:04:01
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
8.115 8.045 8.023 7.936 7.915 7.543 7.521 7.441 7.418 7.390 7.375 7.369 7.359 7.354 7.345 7.327 7.314 7.296 7.282 7.274 7.259 7.245 6.786 6.768 6.749 6.667 6.647
0.96 1.06 1.02 1.12 6.95 2.29 0.31 1.02 1.00
HO
HO
H
N
3aa



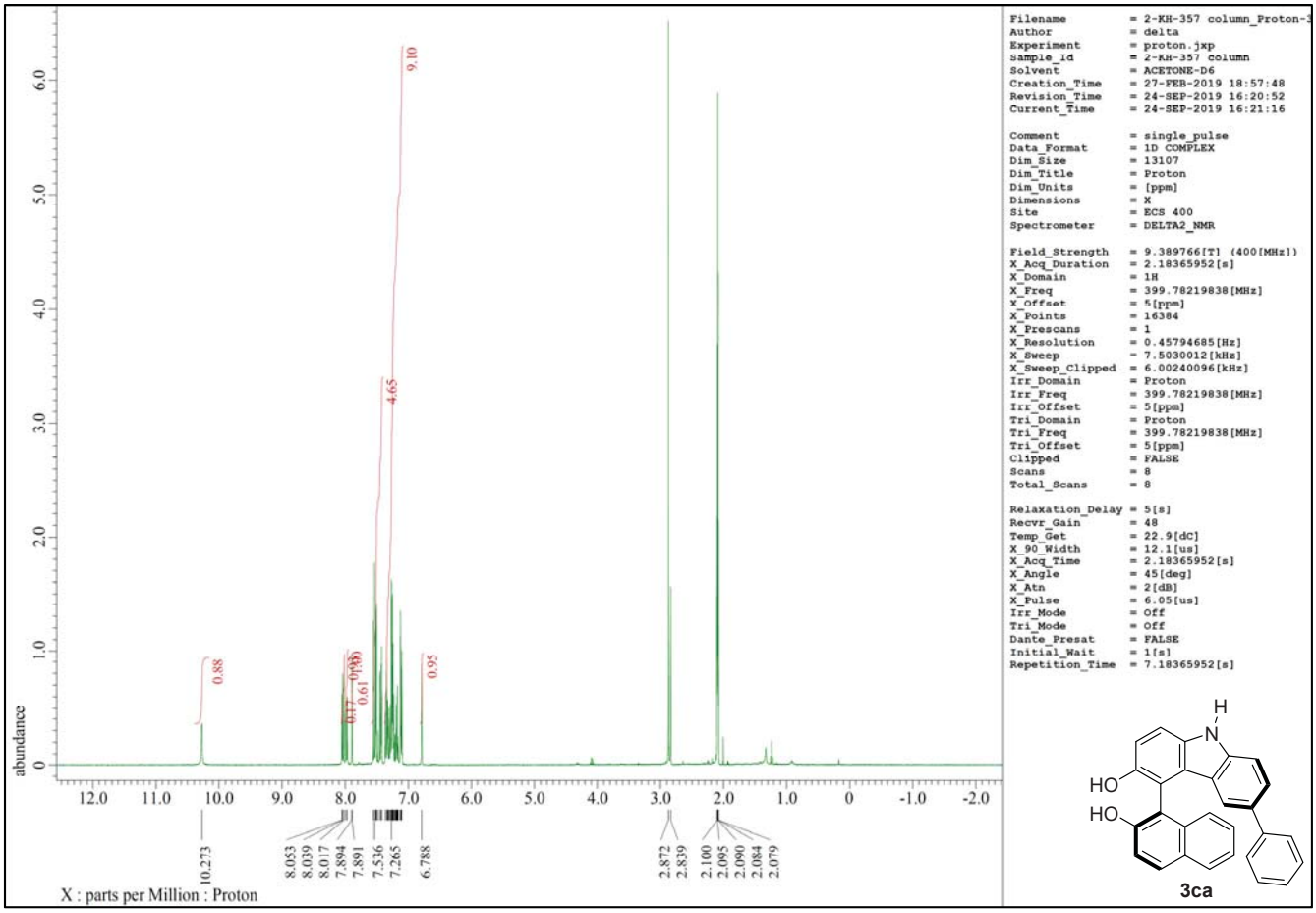
Compound **3aa** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

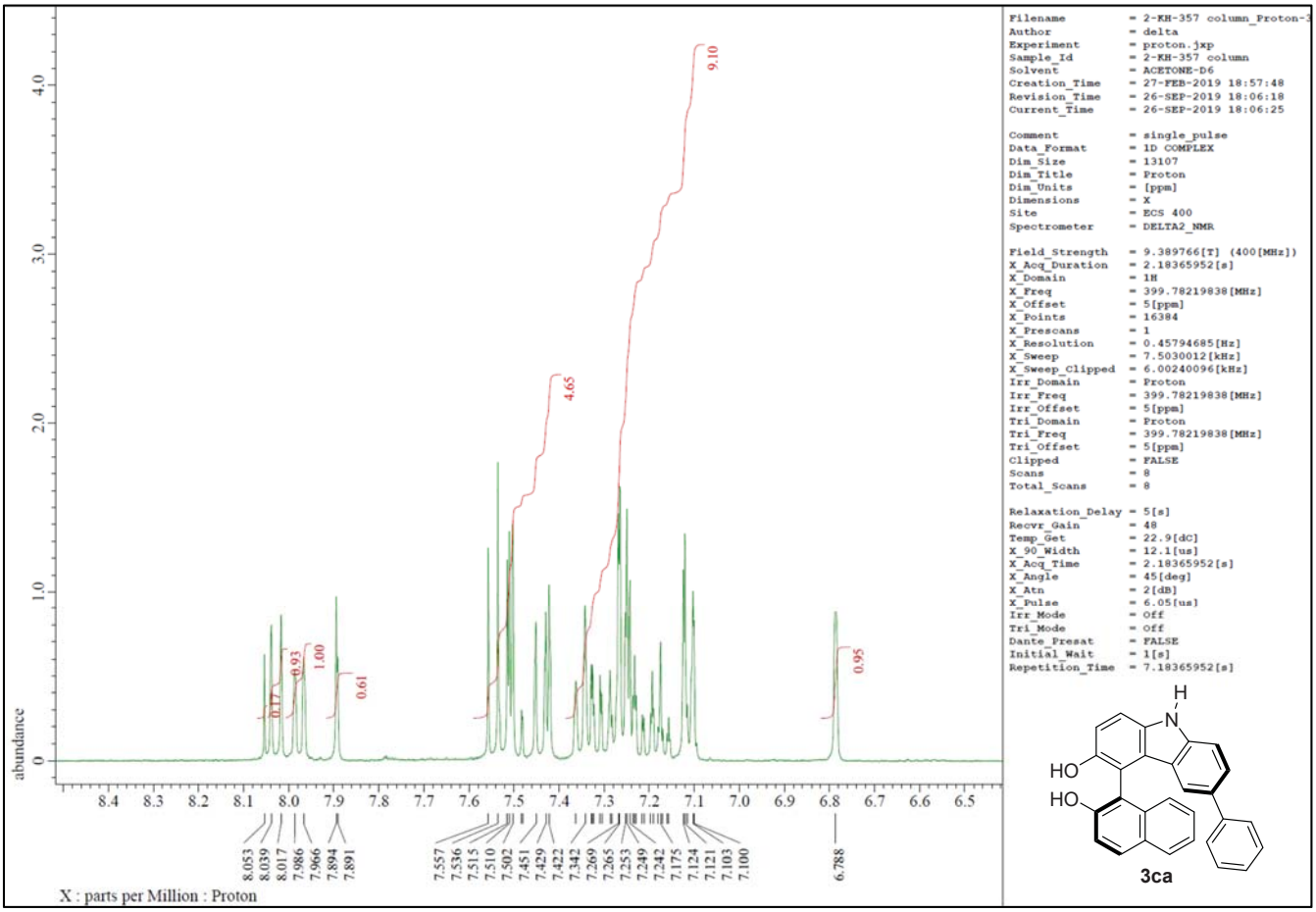

abundance
X : parts per Million : Proton
3.24
3.03
2.35
1.05
1.02
0.82
1.04
1.03
8.034
8.012
7.986
7.928
7.907
7.409
7.258
7.249
6.584
6.582
6.528
5.252
4.679
2.382
0.000
Filename = 2-KH-354 column_Proton-3
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-354 column
Solvent = CHLOROFORM-D
Creation_Time = 27-FEB-2019 11:00:15
Revision_Time = 26-SEP-2019 13:59:52
Current_Time = 26-SEP-2019 16:57:04
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 21.5[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
Me
3ba

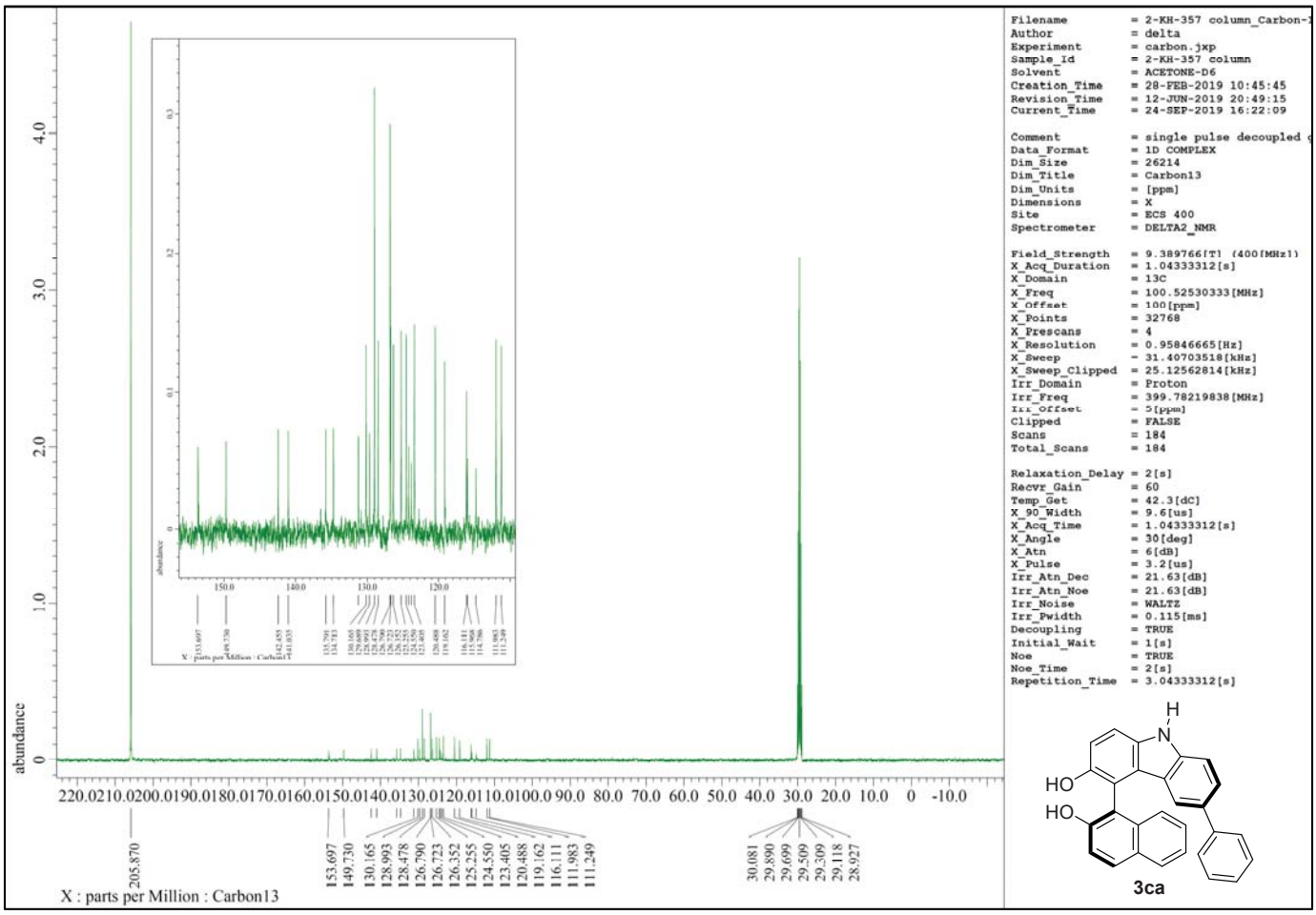

abundance
X : parts per Million : Proton
3.24
2.35
1.05
1.02
0.82
0.98
0.98
1.05
1.00
1.01
0.98
8.034
8.012
7.986
7.928
7.907
7.502
7.480
7.431
7.409
7.371
7.368
7.347
7.327
7.309
7.307
7.291
7.258
7.249
7.227
7.166
6.605
6.603
6.584
6.582
6.528
6.508
Filename = 2-KH-354 column_Proton-3
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-354 column
Solvent = CHLOROFORM-D
Creation_Time = 27-FEB-2019 11:00:15
Revision_Time = 26-SEP-2019 13:59:52
Current_Time = 26-SEP-2019 18:04:43
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 21.5[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
Me
3ba



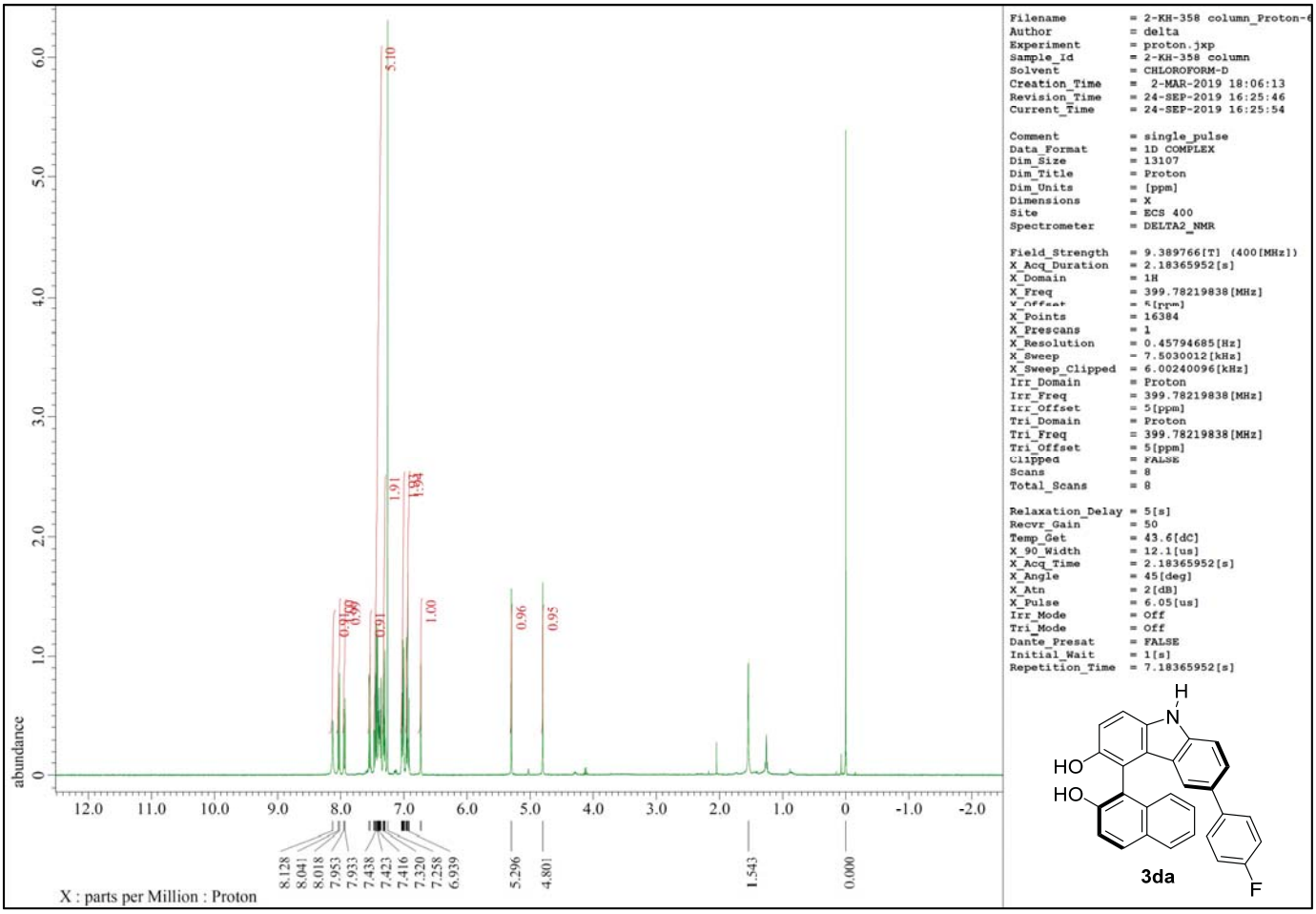
Compound **3ba** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

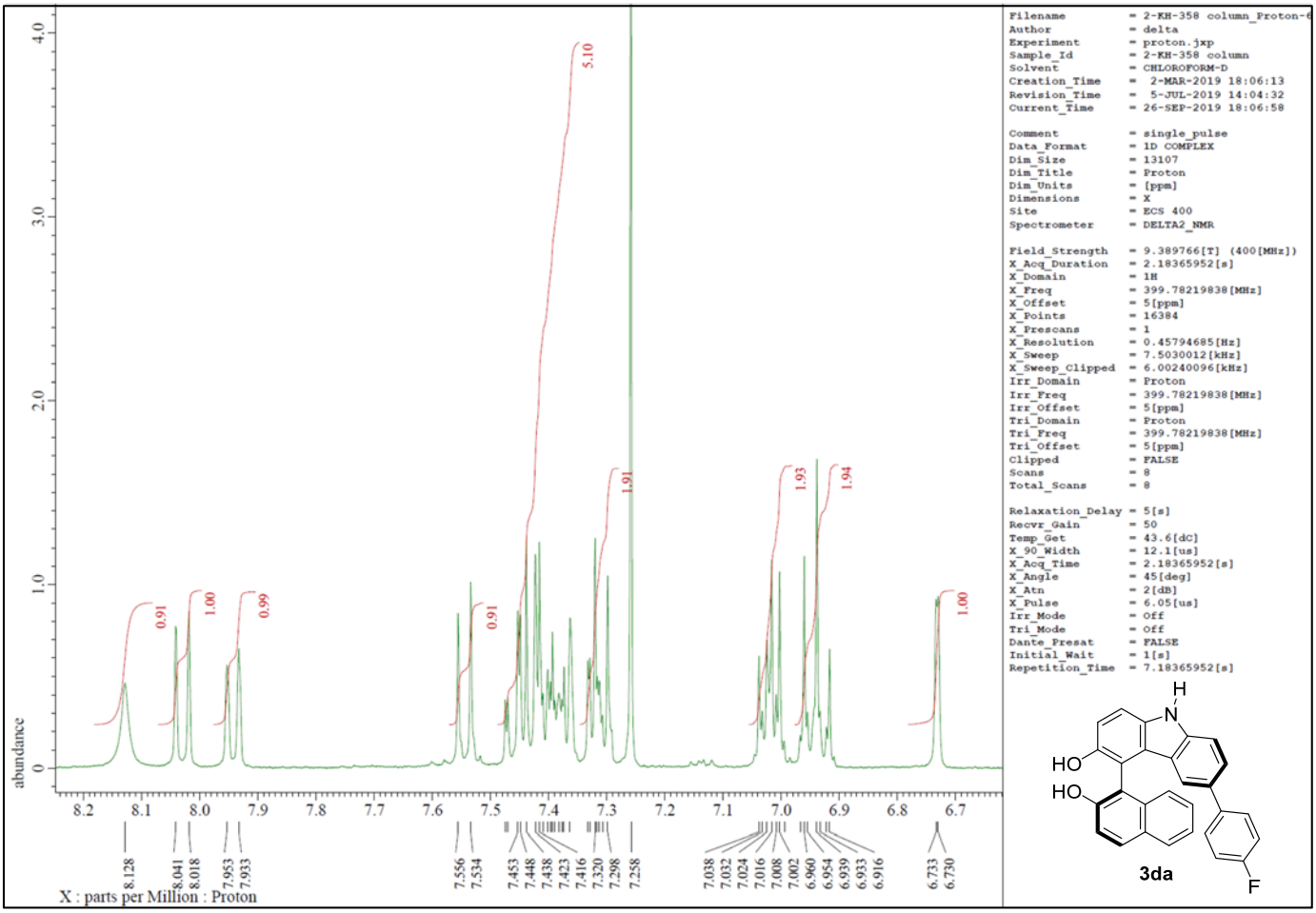

9.10
4.65
0.88
0.95
0.61
abundance
X : parts per Million : Proton
10.273
8.053
8.039
8.017
7.894
7.891
7.536
7.265
6.788
2.872
2.839
2.100
2.095
2.090
2.084
2.079
Filename = 2-KH-357 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-357 column
Solvent = ACETONE-D6
Creation_Time = 27-FEB-2019 18:57:48
Revision_Time = 24-SEP-2019 16:20:52
Current_Time = 24-SEP-2019 16:21:16
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 22.9[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
3ca

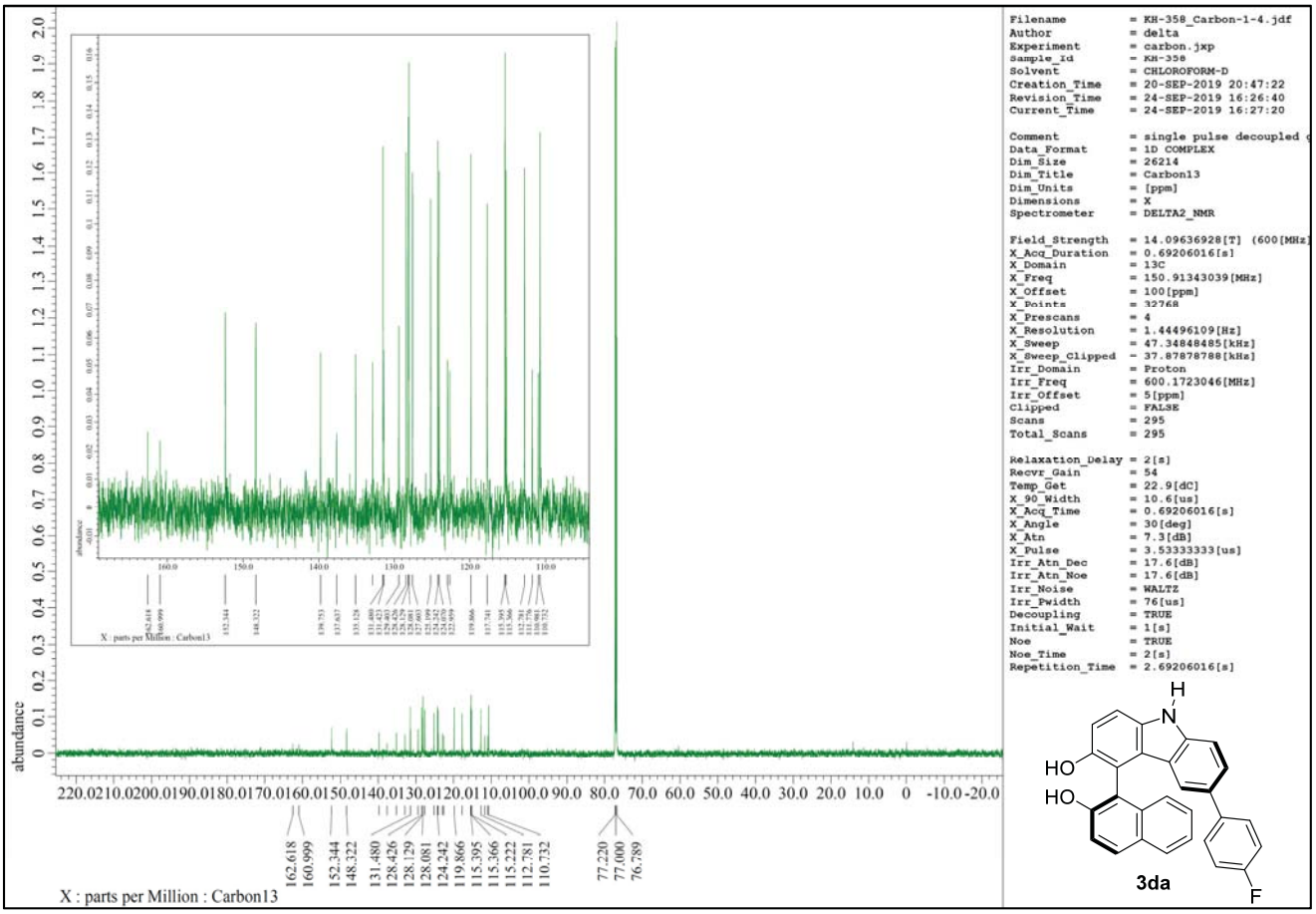

9.10
4.65
0.93
1.00
0.61
0.17
0.95
abundance
X : parts per Million : Proton
8.053
8.039
8.017
7.986
7.966
7.894
7.891
7.557
7.536
7.515
7.510
7.502
7.451
7.429
7.422
7.342
7.269
7.265
7.253
7.249
7.242
7.175
7.124
7.121
7.103
7.100
6.788
Filename = 2-KH-357 column_Proton-3
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-357 column
Solvent = ACETONE-D6
Creation_Time = 27-FEB-2019 18:57:48
Revision_Time = 26-SEP-2019 18:06:18
Current_Time = 26-SEP-2019 18:06:25
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 22.9[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
3ca

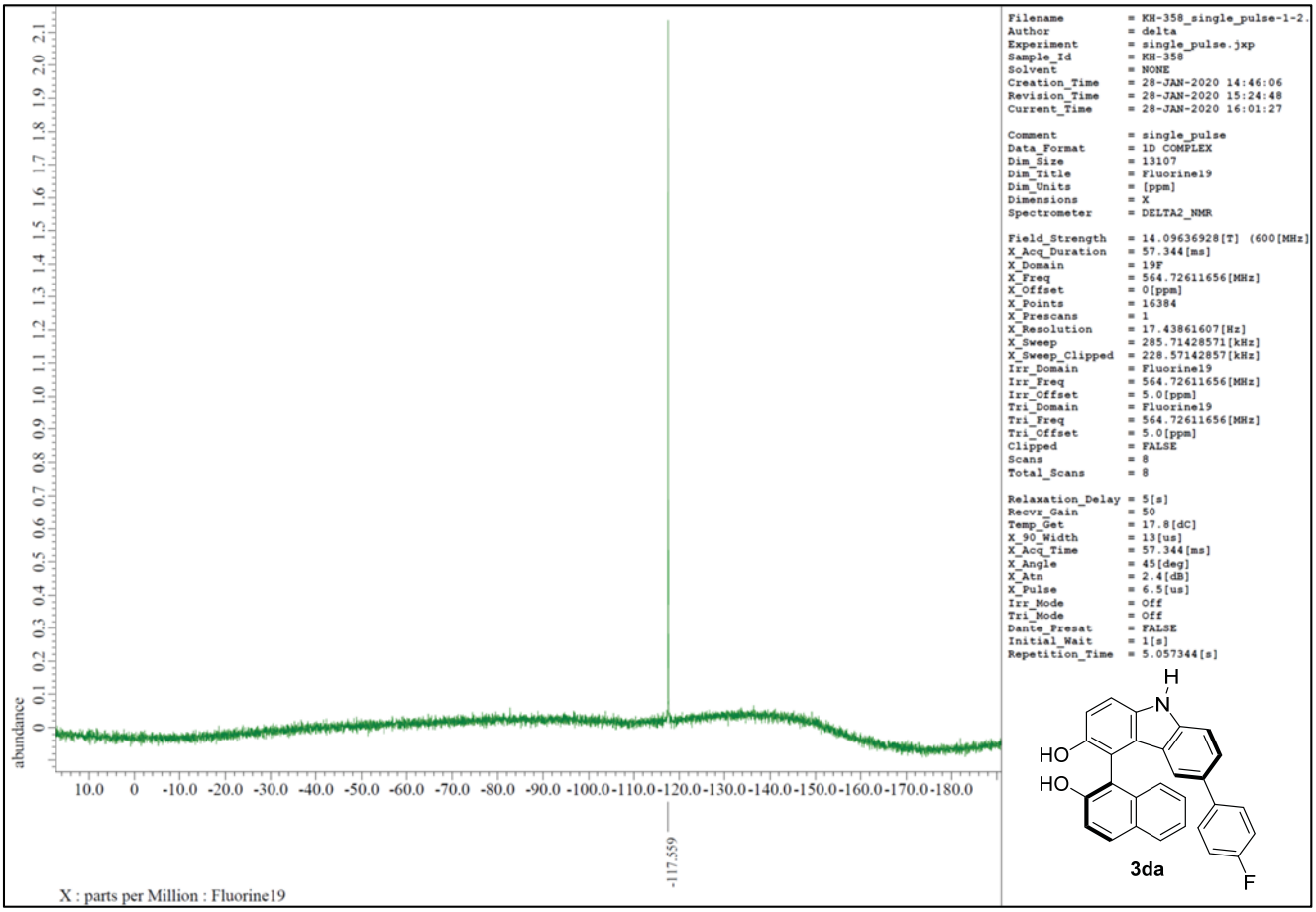


Compound **3ca** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)

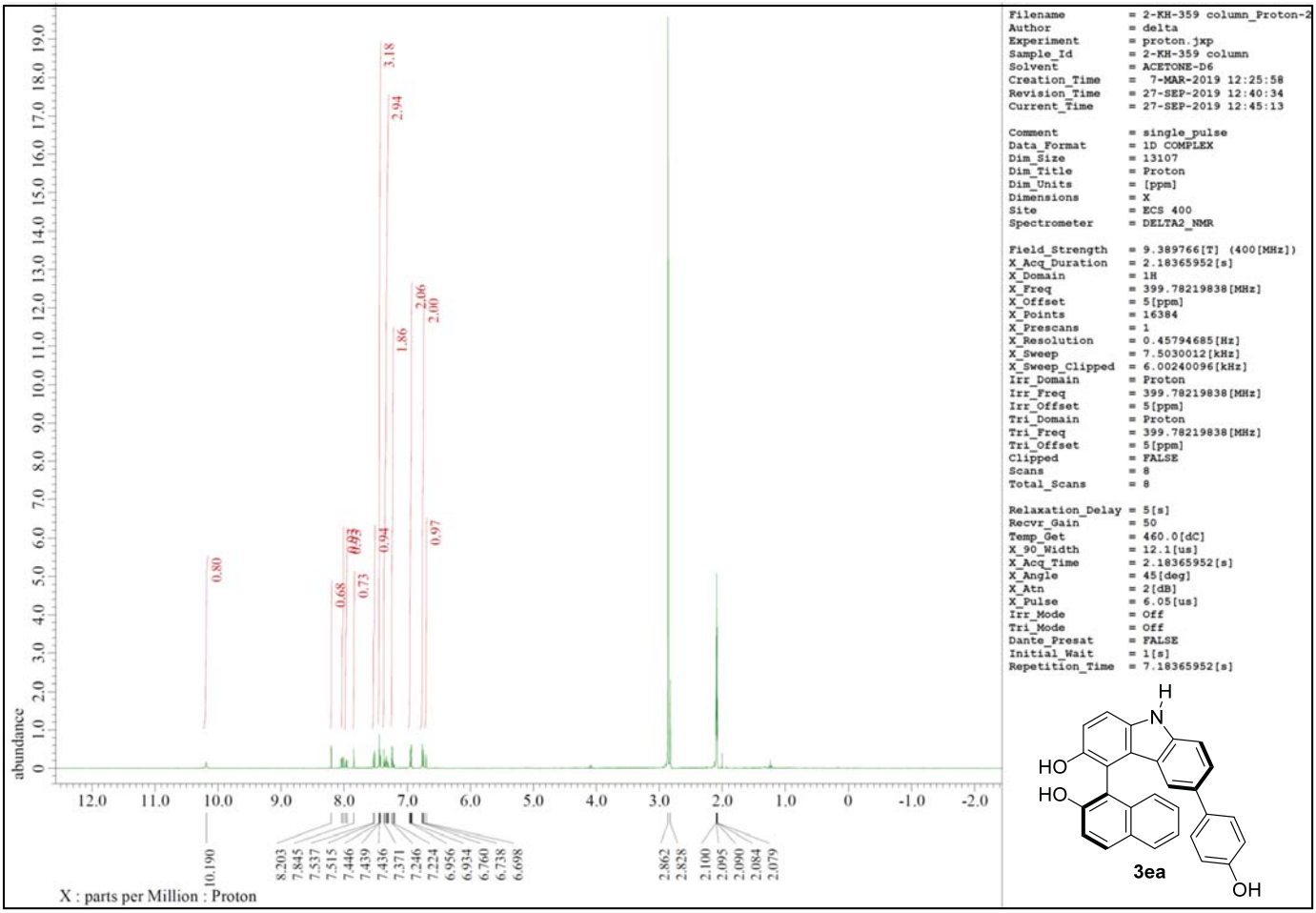

abundance
X : parts per Million : Proton
Filename = 2-KH-358 column_Proton-6
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-358 column
Solvent = CHLOROFORM-D
Creation_Time = 2-MAR-2019 18:06:13
Revision_Time = 24-SEP-2019 16:25:46
Current_Time = 24-SEP-2019 16:25:54
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 43.6[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
3da

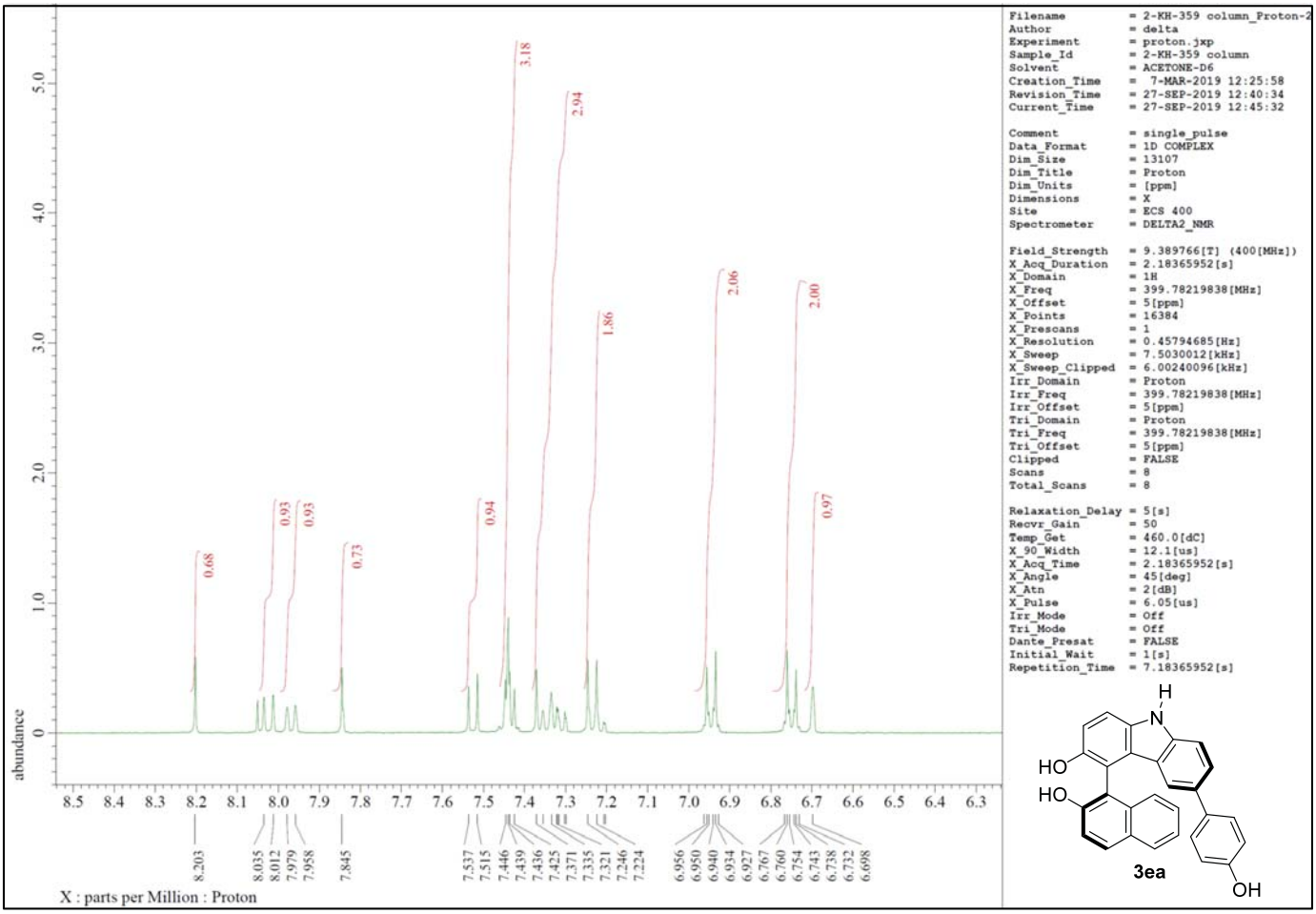

abundance
X : parts per Million : Proton
Filename = 2-KH-358 column_Proton-6
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-358 column
Solvent = CHLOROFORM-D
Creation_Time = 2-MAR-2019 18:06:13
Revision_Time = 5-JUL-2019 14:04:32
Current_Time = 26-SEP-2019 18:06:58
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 43.6[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
3da

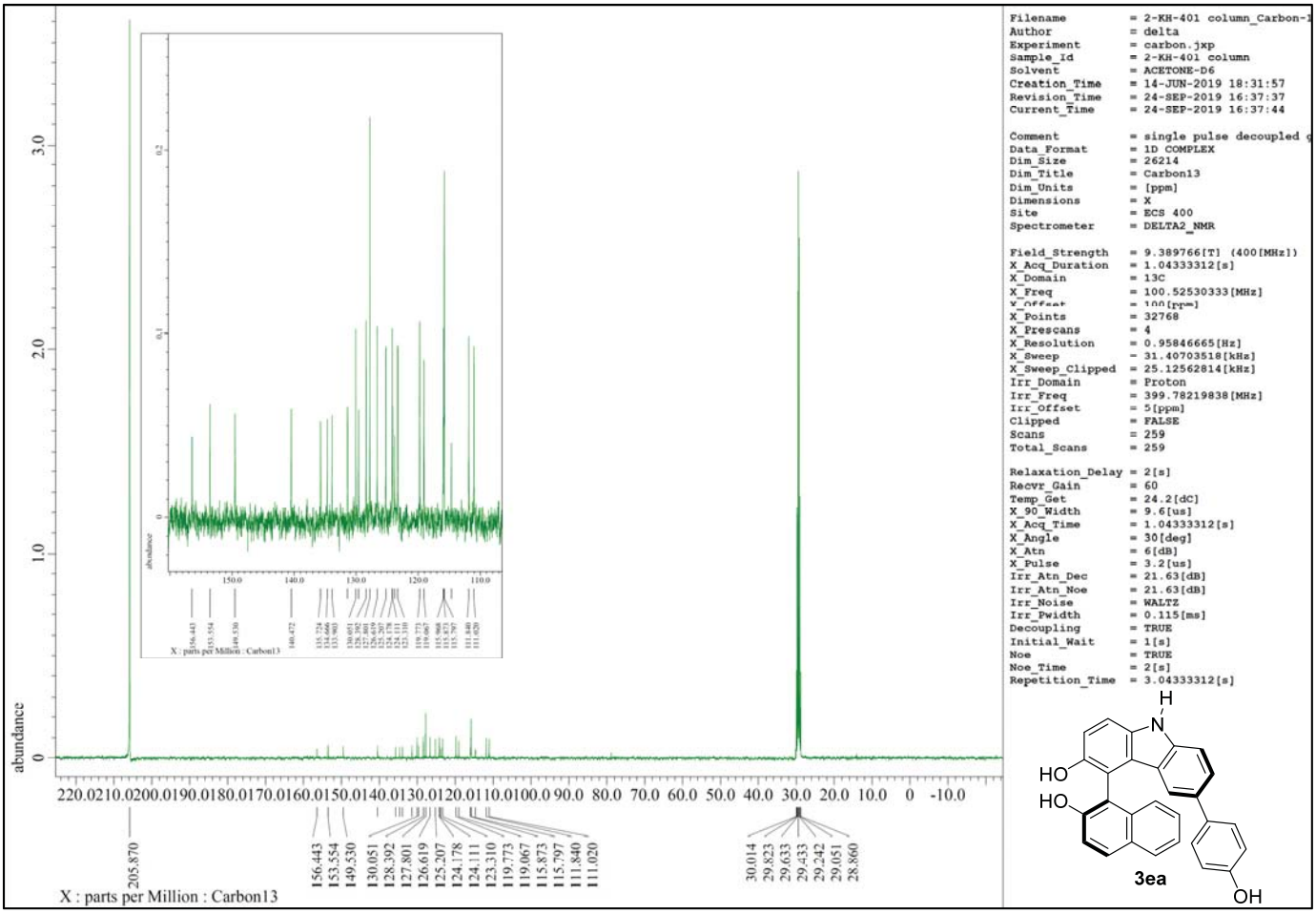




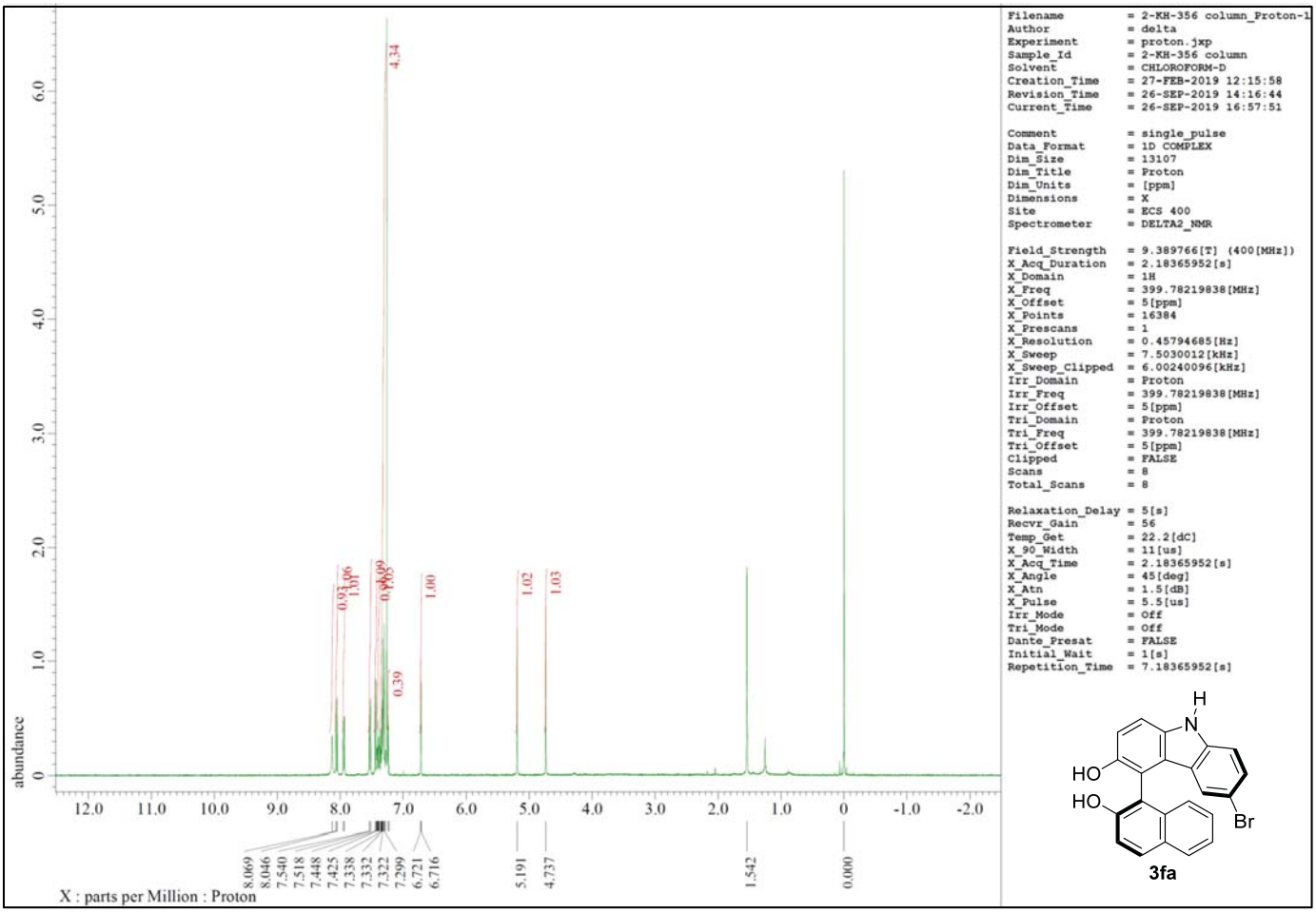
Compound **3da** (^{1}H NMR, 400 MHz, $CDCl_3$, ^{13}C NMR, 150 MHz, $CDCl_3$ and ^{19}F NMR, 565 MHz, $CDCl_3$)


abundance
X : parts per Million : Proton
3.18
2.94
2.06
2.00
1.86
0.97
0.94
0.93
0.93
0.80
0.73
0.68
10.190
8.203
7.845
7.537
7.515
7.446
7.439
7.436
7.371
7.246
7.224
6.956
6.934
6.760
6.738
6.698
2.862
2.828
2.100
2.095
2.090
2.084
2.079
Filename = 2-KH-359 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-359 column
Solvent = ACETONE-D6
Creation_Time = 7-MAR-2019 12:25:58
Revision_Time = 27-SEP-2019 12:40:34
Current_Time = 27-SEP-2019 12:45:13
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
OH
3ea


abundance
X : parts per Million : Proton
3.18
2.94
2.06
2.00
1.86
0.97
0.94
0.93
0.93
0.68
0.73
8.203
8.035
8.012
7.979
7.958
7.845
7.537
7.515
7.446
7.439
7.436
7.425
7.371
7.335
7.321
7.246
7.224
6.956
6.950
6.940
6.934
6.927
6.767
6.760
6.754
6.743
6.738
6.732
6.698
Filename = 2-KH-359 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-359 column
Solvent = ACETONE-D6
Creation_Time = 7-MAR-2019 12:25:58
Revision_Time = 27-SEP-2019 12:40:34
Current_Time = 27-SEP-2019 12:45:32
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
OH
3ea



Compound **3ea** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)



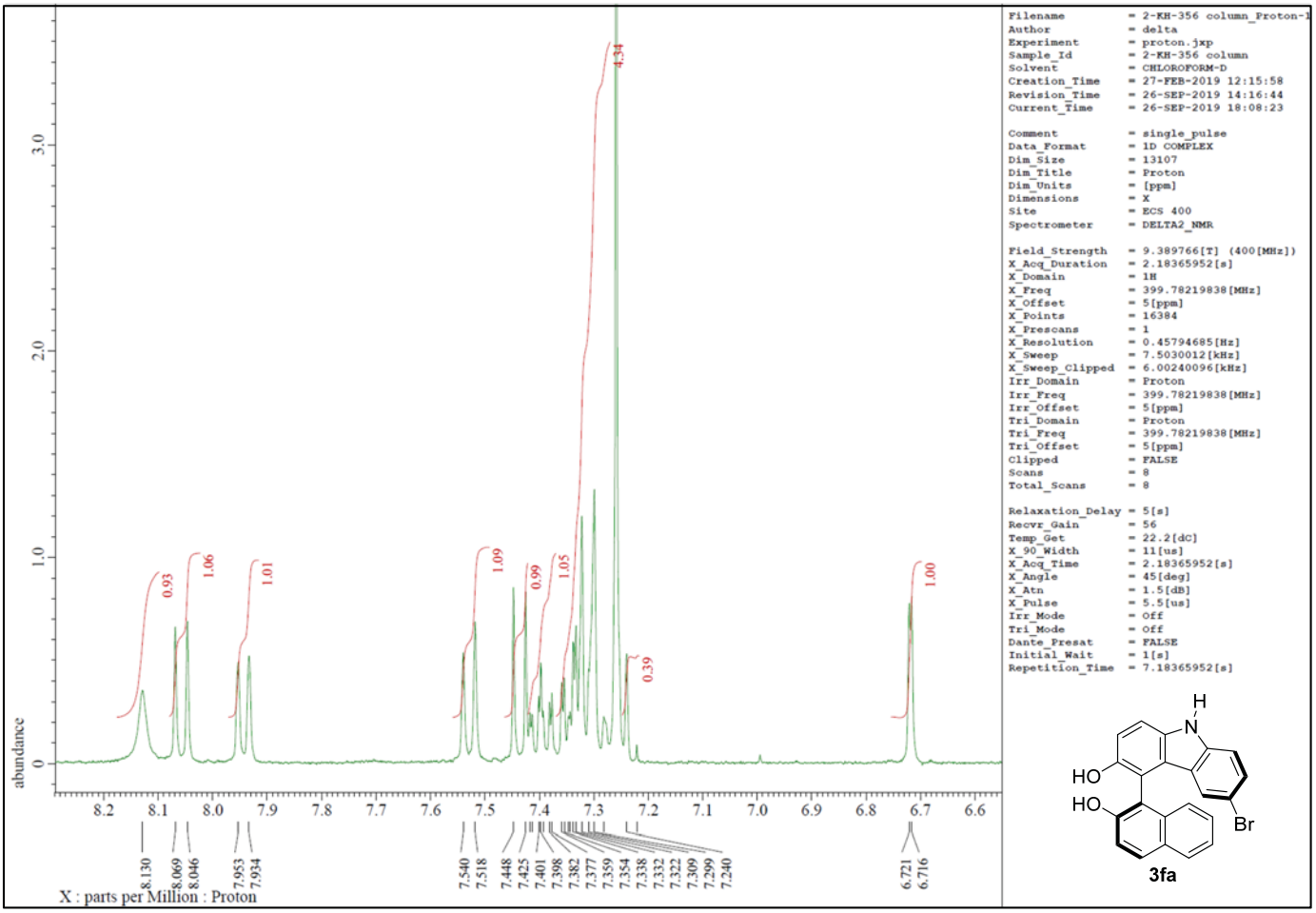

```
Filename          = 2-KH-356 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-356 column
Solvent           = CHLOROFORM-D
Creation_Time     = 27-FEB-2019 12:15:58
Revision_Time     = 26-SEP-2019 14:16:44
Current_Time      = 26-SEP-2019 16:57:51

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 56
Temp_Get          = 22.2[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```


3fa



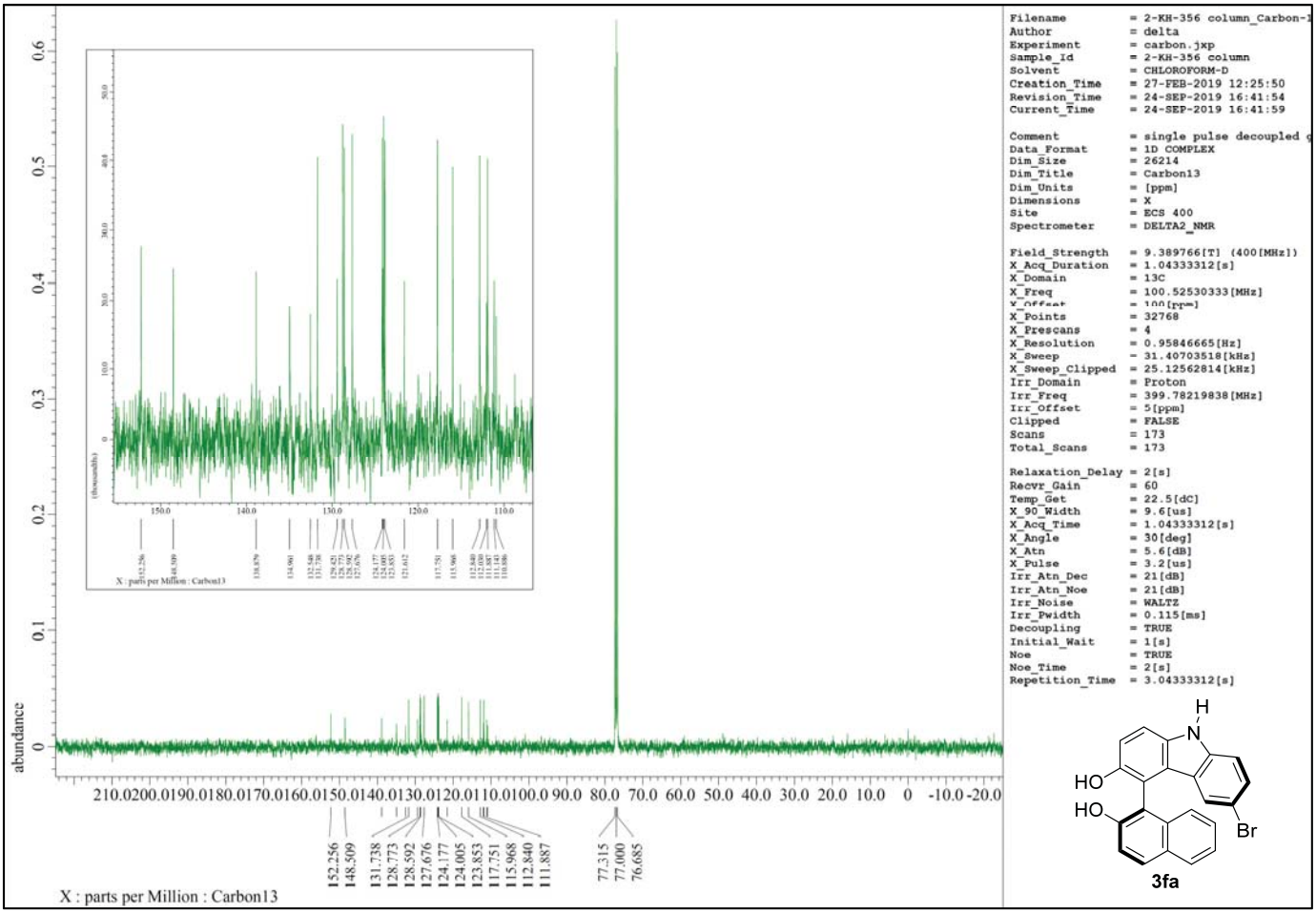

```
Filename          = 2-KH-356 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-356 column
Solvent           = CHLOROFORM-D
Creation_Time     = 27-FEB-2019 12:15:58
Revision_Time     = 26-SEP-2019 14:16:44
Current_Time      = 26-SEP-2019 18:08:23

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

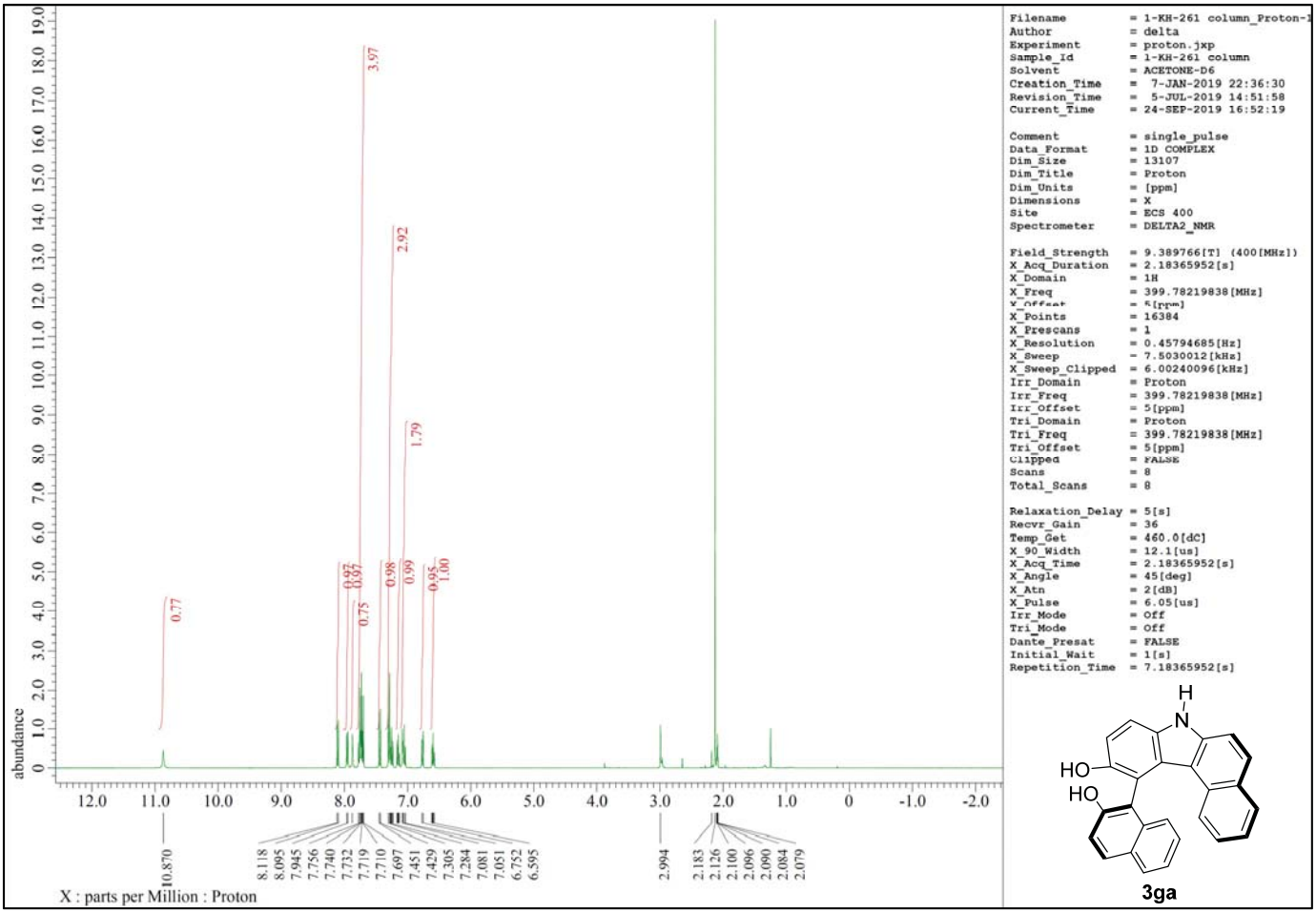
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

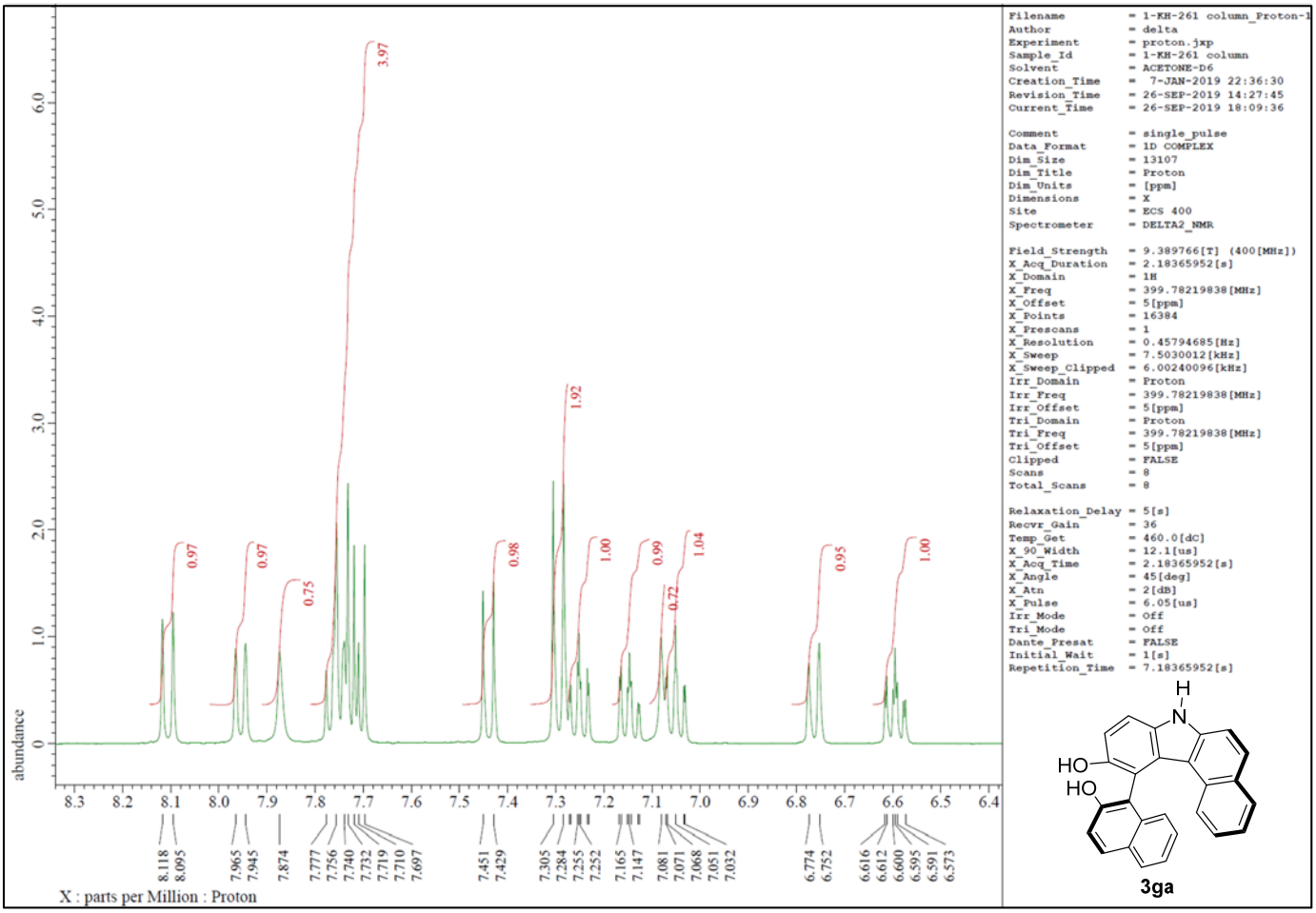
Relaxation_Delay  = 5[s]
Recvr_Gain        = 56
Temp_Get          = 22.2[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

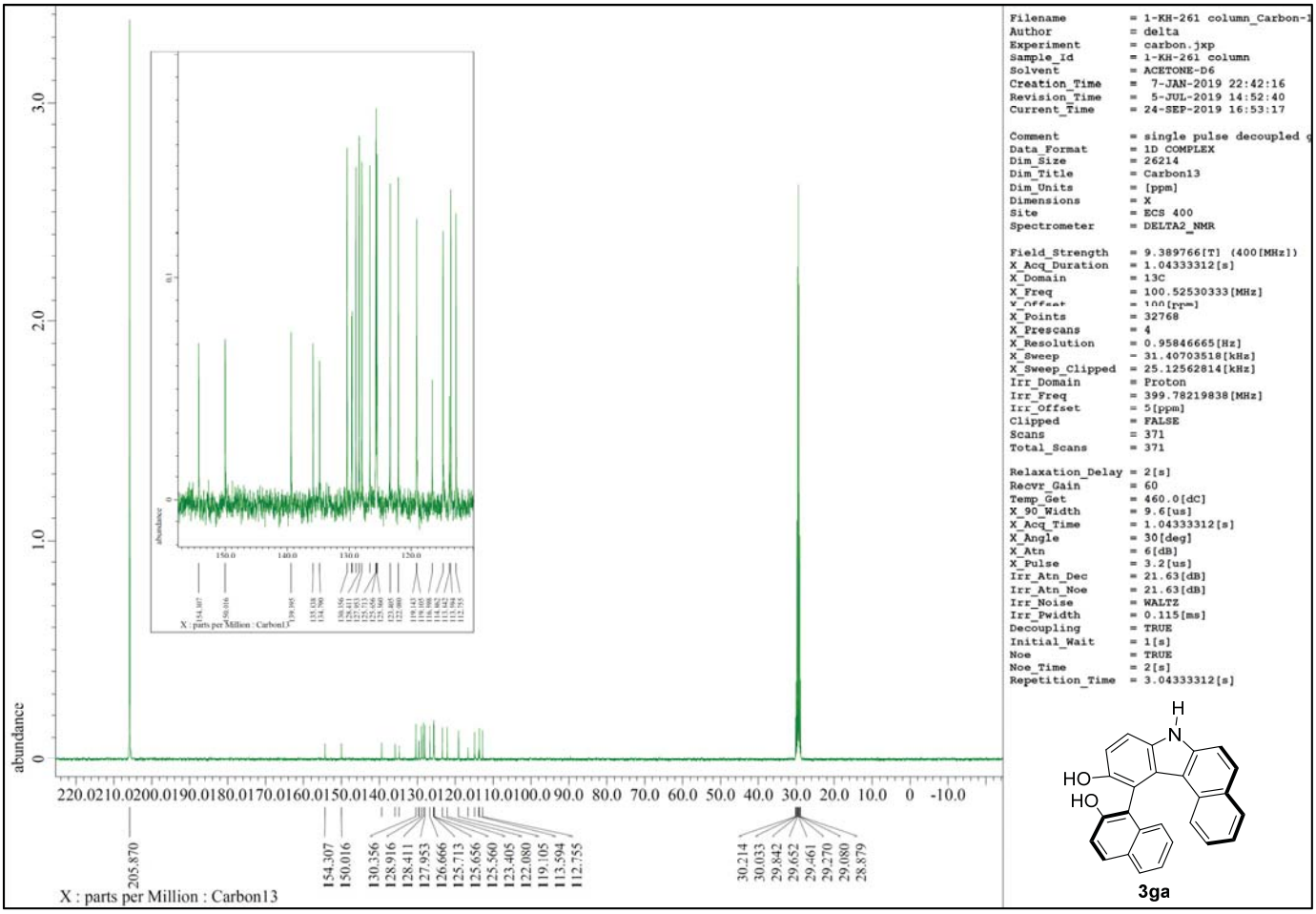

3fa



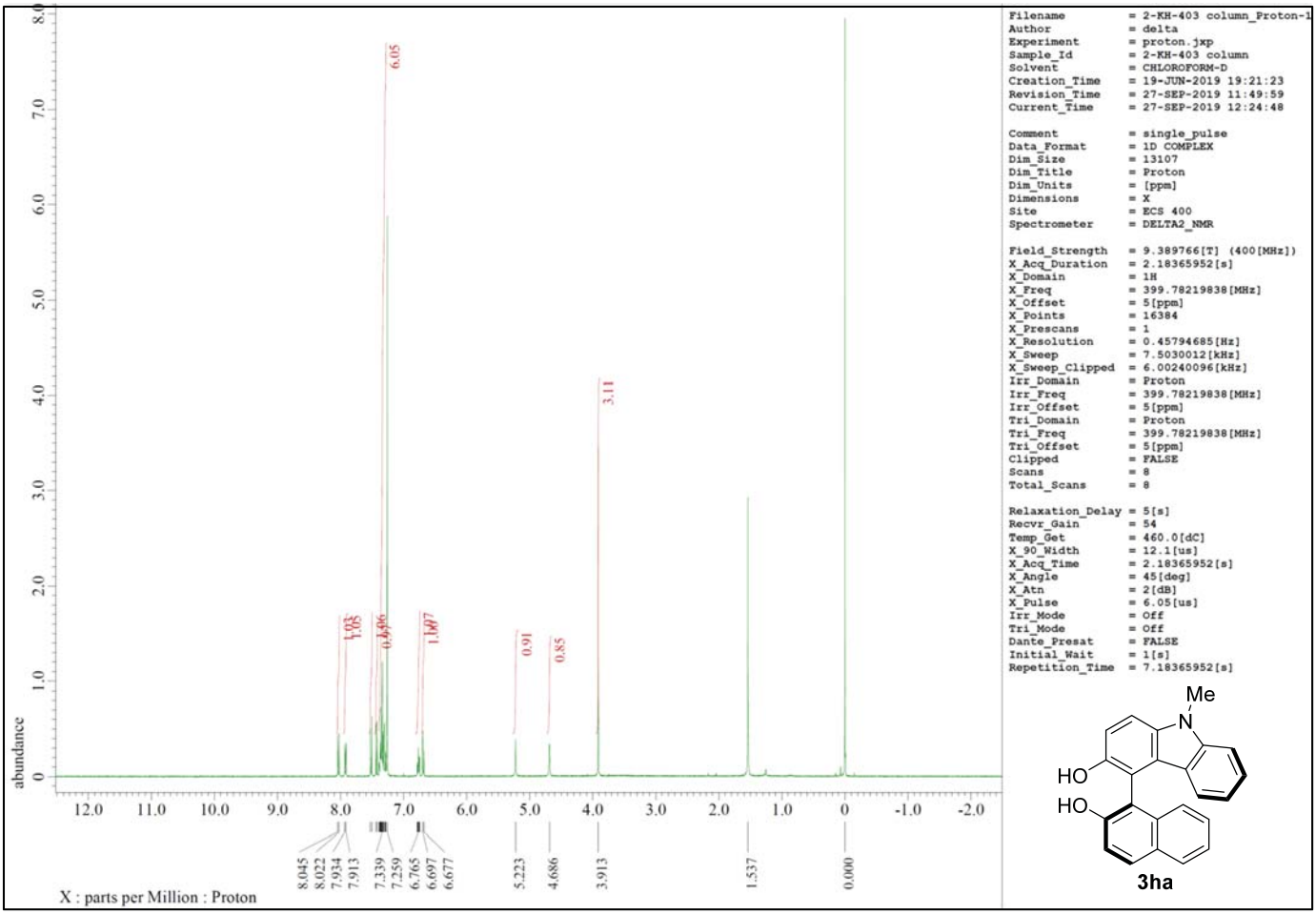
Compound **3fa** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)


abundance
X : parts per Million : Proton
3.97
2.92
1.79
0.77
0.97
0.97
0.75
0.98
0.99
0.95
1.00
10.870
8.118
8.095
7.945
7.756
7.740
7.732
7.719
7.710
7.697
7.451
7.429
7.305
7.284
7.081
7.051
6.752
6.595
2.994
2.183
2.126
2.100
2.096
2.090
2.084
2.079
Filename = 1-KH-261 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 1-KH-261 column
Solvent = ACETONE-D6
Creation_Time = 7-JAN-2019 22:36:30
Revision_Time = 5-JUL-2019 14:51:58
Current_Time = 24-SEP-2019 16:52:19
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
3ga


abundance
X : parts per Million : Proton
3.97
1.92
0.97
0.97
0.75
0.98
1.00
0.99
0.72
1.04
0.95
1.00
8.118
8.095
7.965
7.945
7.874
7.777
7.756
7.740
7.732
7.719
7.710
7.697
7.451
7.429
7.305
7.284
7.255
7.252
7.165
7.147
7.081
7.071
7.068
7.051
7.032
6.774
6.752
6.616
6.612
6.600
6.595
6.591
6.573
Filename = 1-KH-261 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 1-KH-261 column
Solvent = ACETONE-D6
Creation_Time = 7-JAN-2019 22:36:30
Revision_Time = 26-SEP-2019 14:27:45
Current_Time = 26-SEP-2019 18:09:36
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 36
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
3ga



Compound **3ga** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)



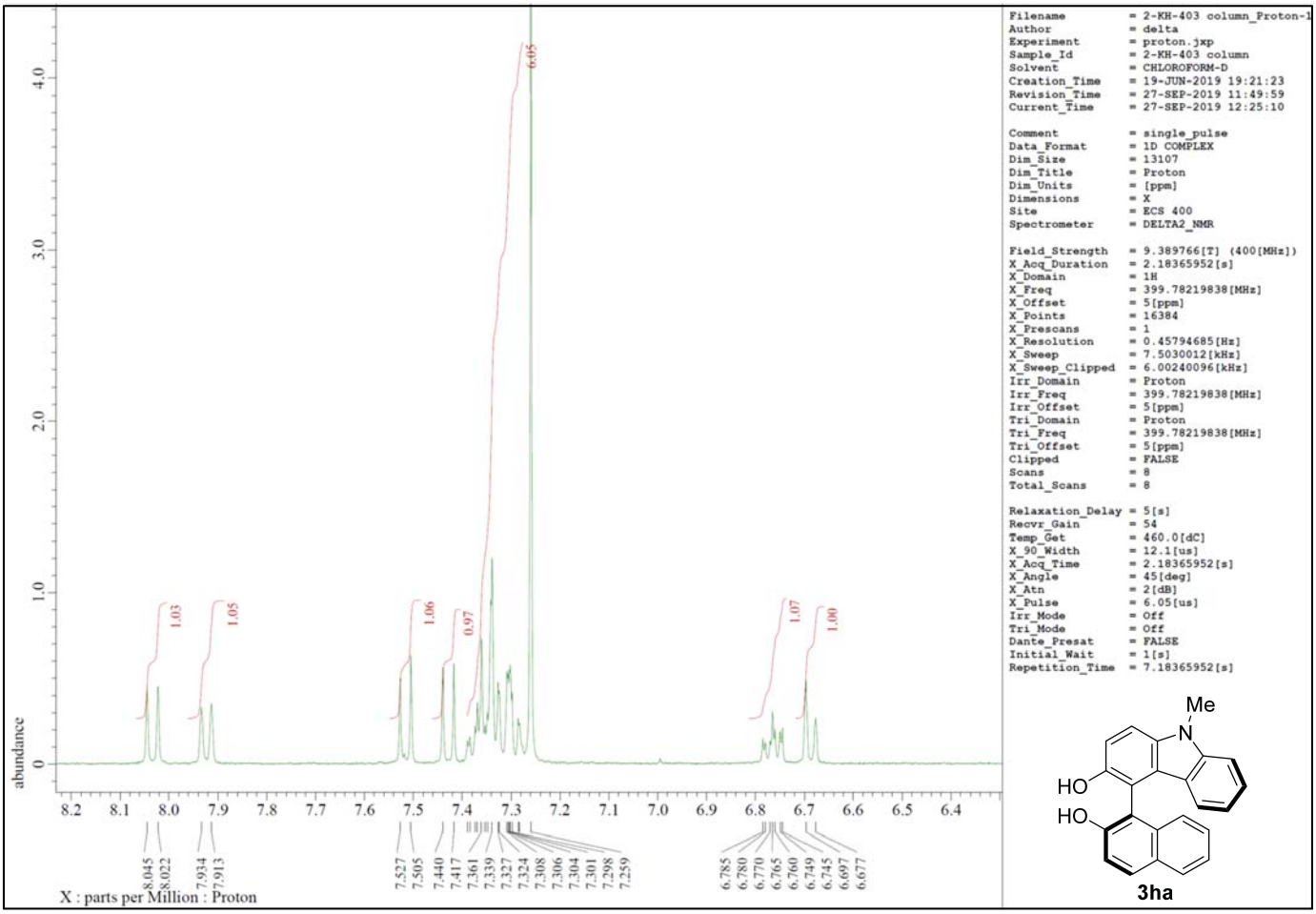

```
Filename          = 2-KH-403 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-403 column
Solvent           = CHLOROFORM-D
Creation_Time     = 19-JUN-2019 19:21:23
Revision_Time     = 27-SEP-2019 11:49:59
Current_Time      = 27-SEP-2019 12:24:48

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 460.0[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```


3ha



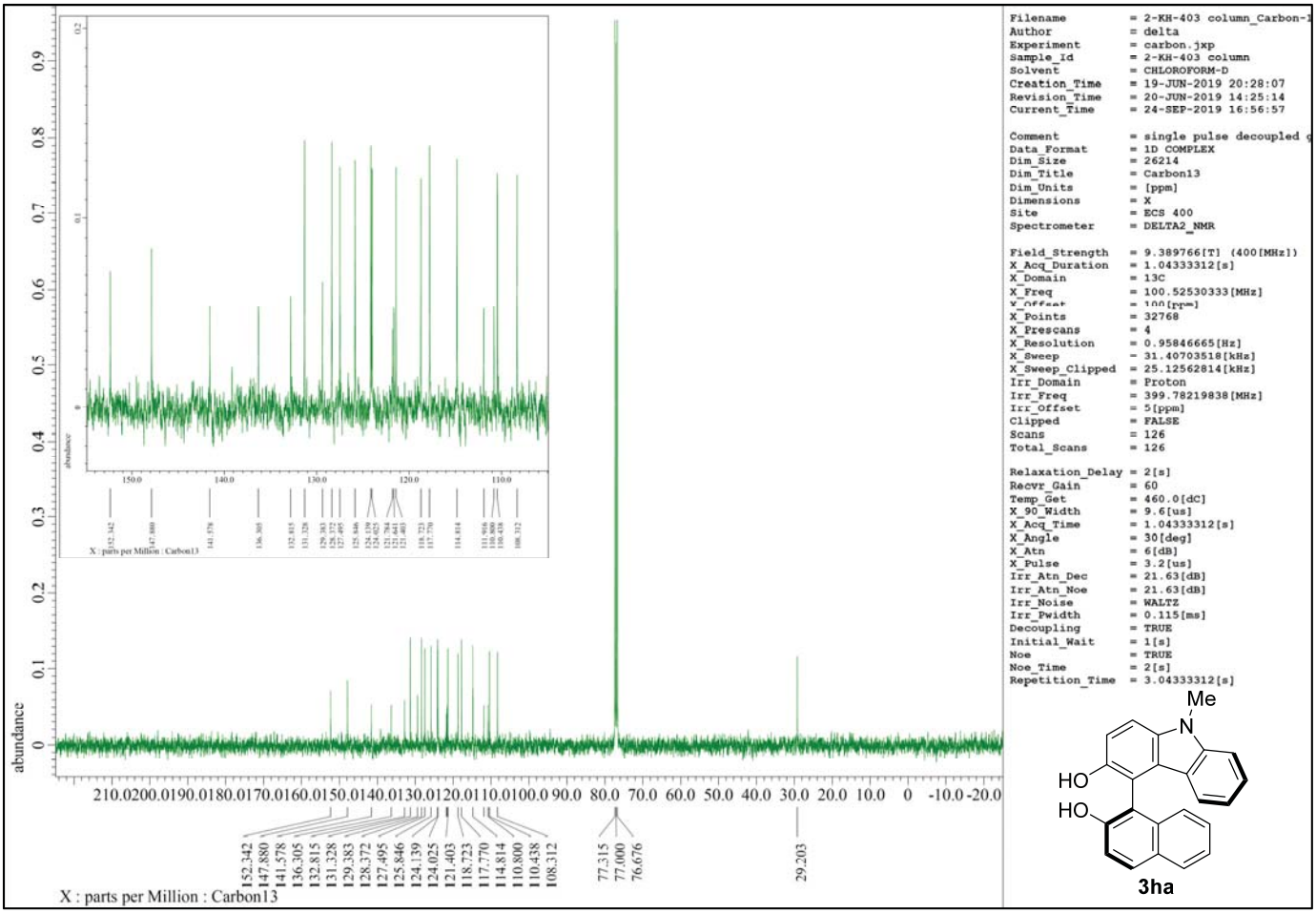

```
Filename          = 2-KH-403 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-403 column
Solvent           = CHLOROFORM-D
Creation_Time     = 19-JUN-2019 19:21:23
Revision_Time     = 27-SEP-2019 11:49:59
Current_Time      = 27-SEP-2019 12:25:10

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

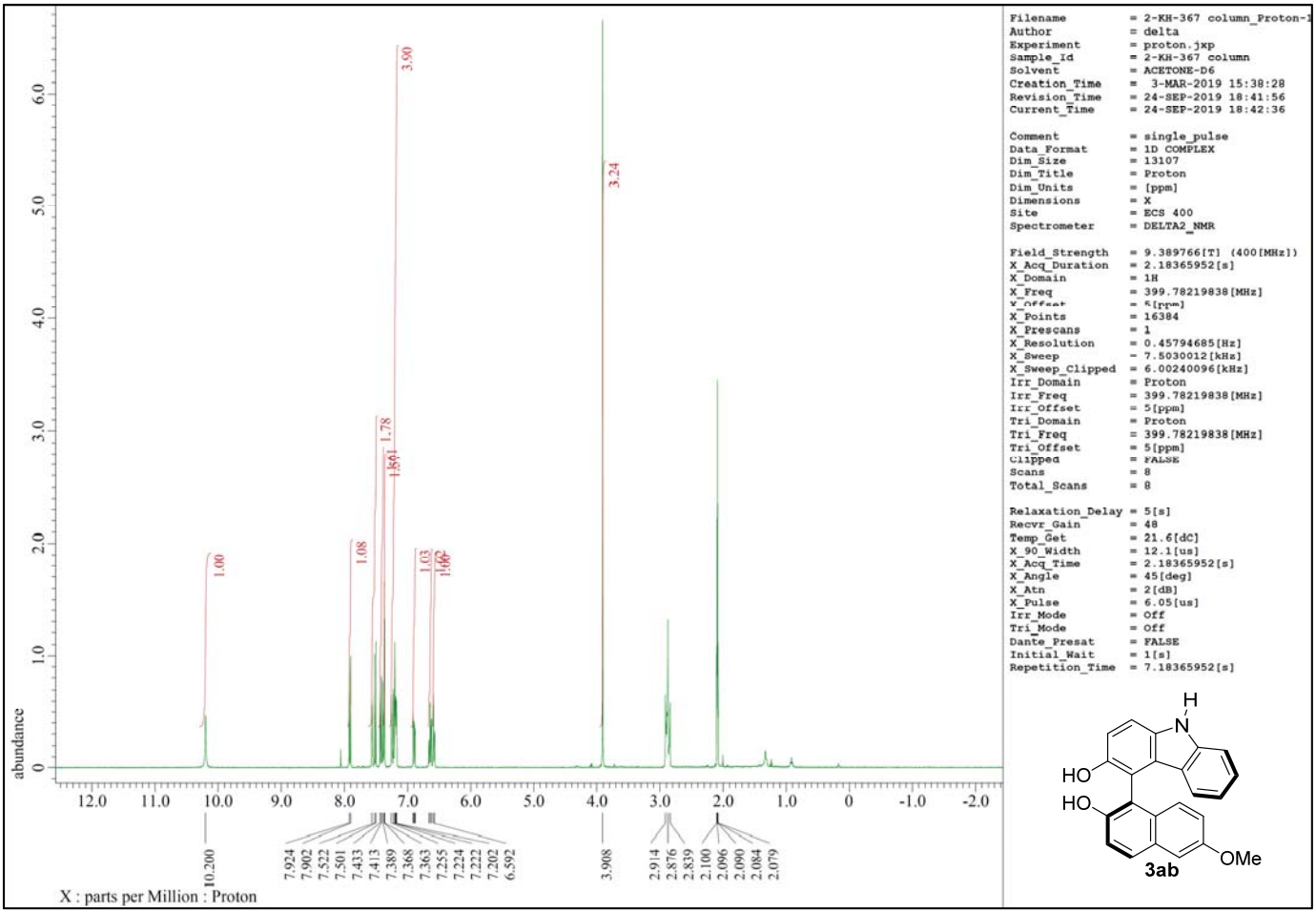
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

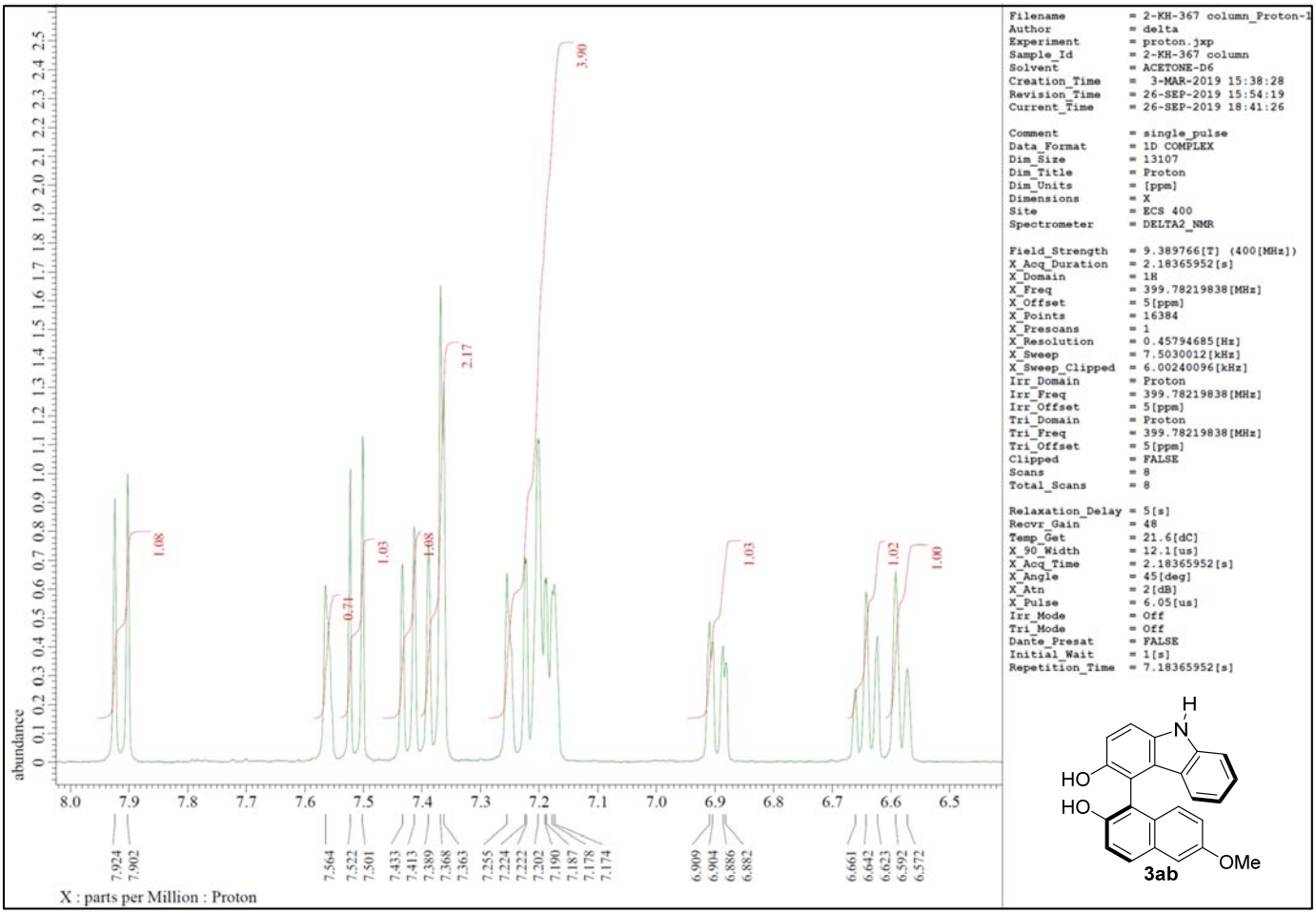
Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 460.0[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

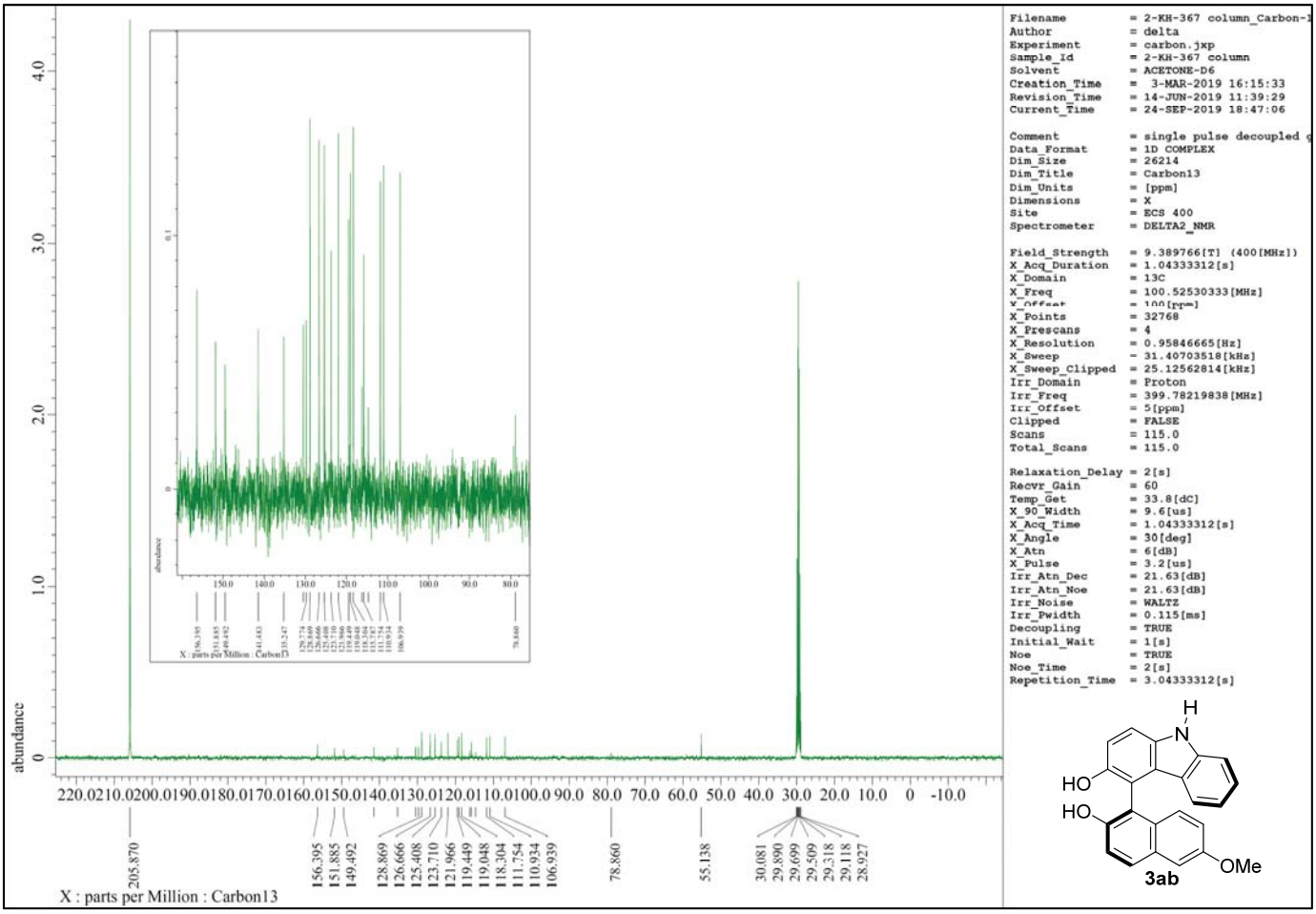

3ha



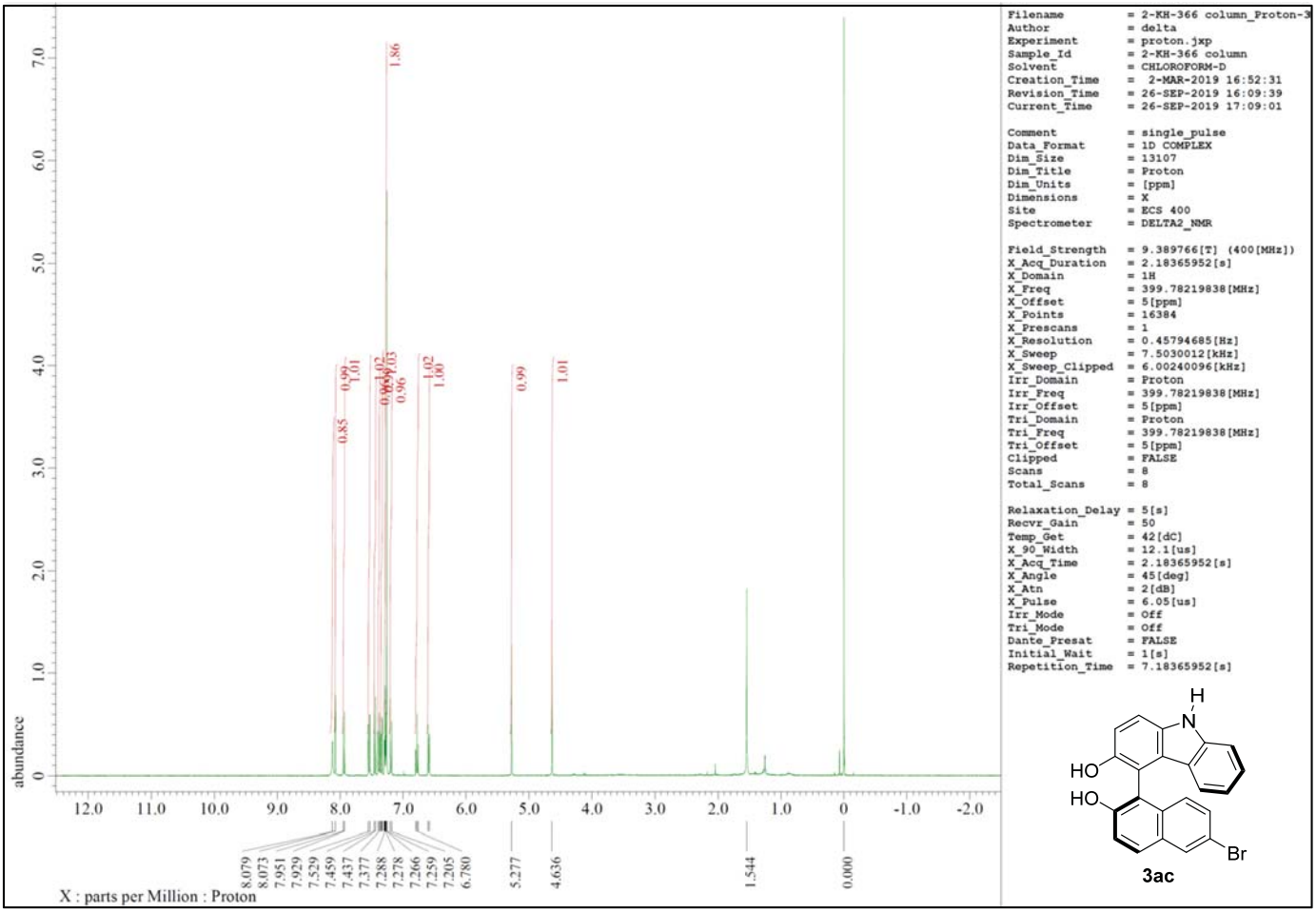
Compound **3ha** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

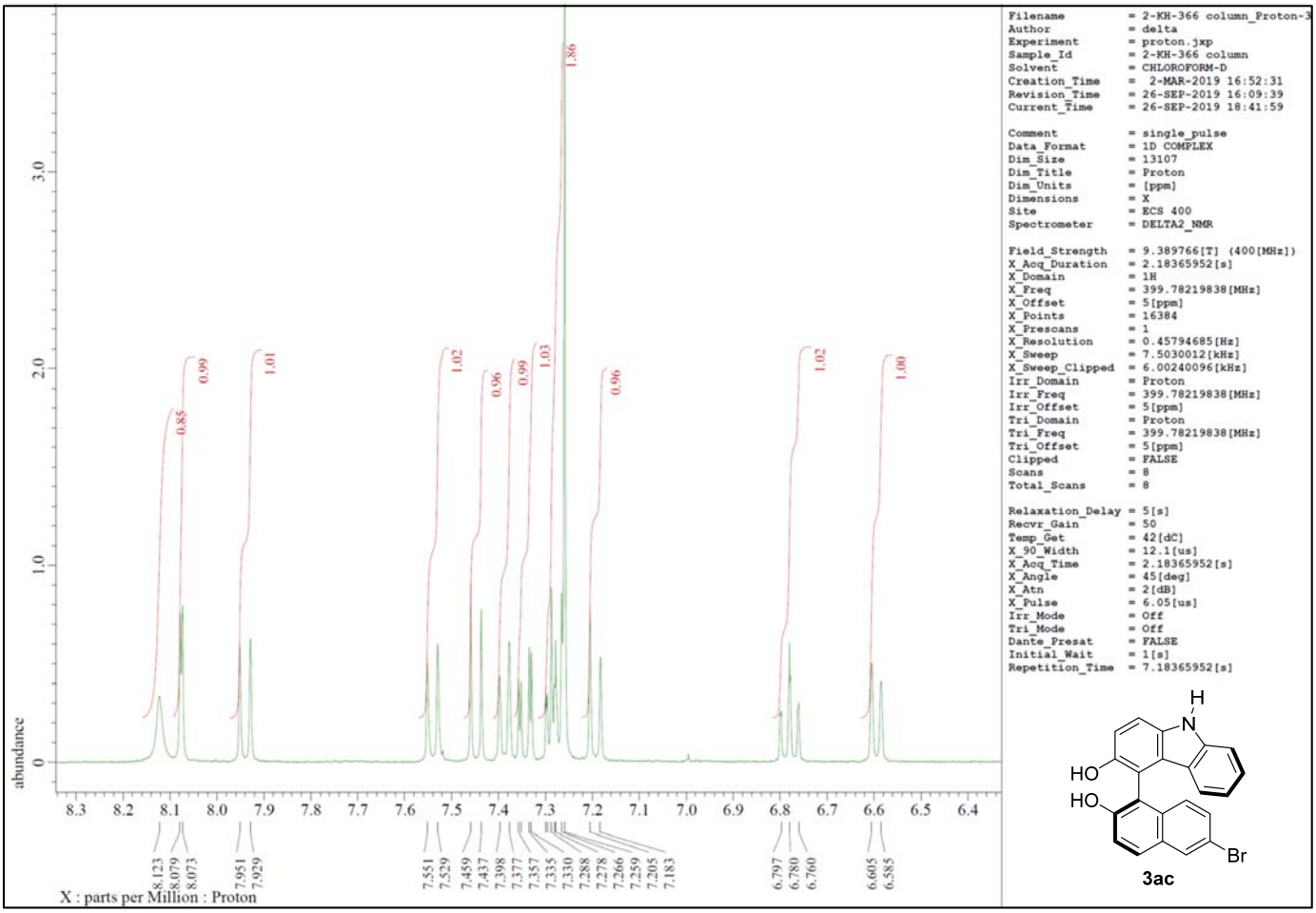

Filename = 2-KH-367 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-367 column
Solvent = ACETONE-D6
Creation_Time = 3-MAR-2019 15:38:28
Revision_Time = 24-SEP-2019 18:41:56
Current_Time = 24-SEP-2019 18:42:36
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 21.6[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
3ab

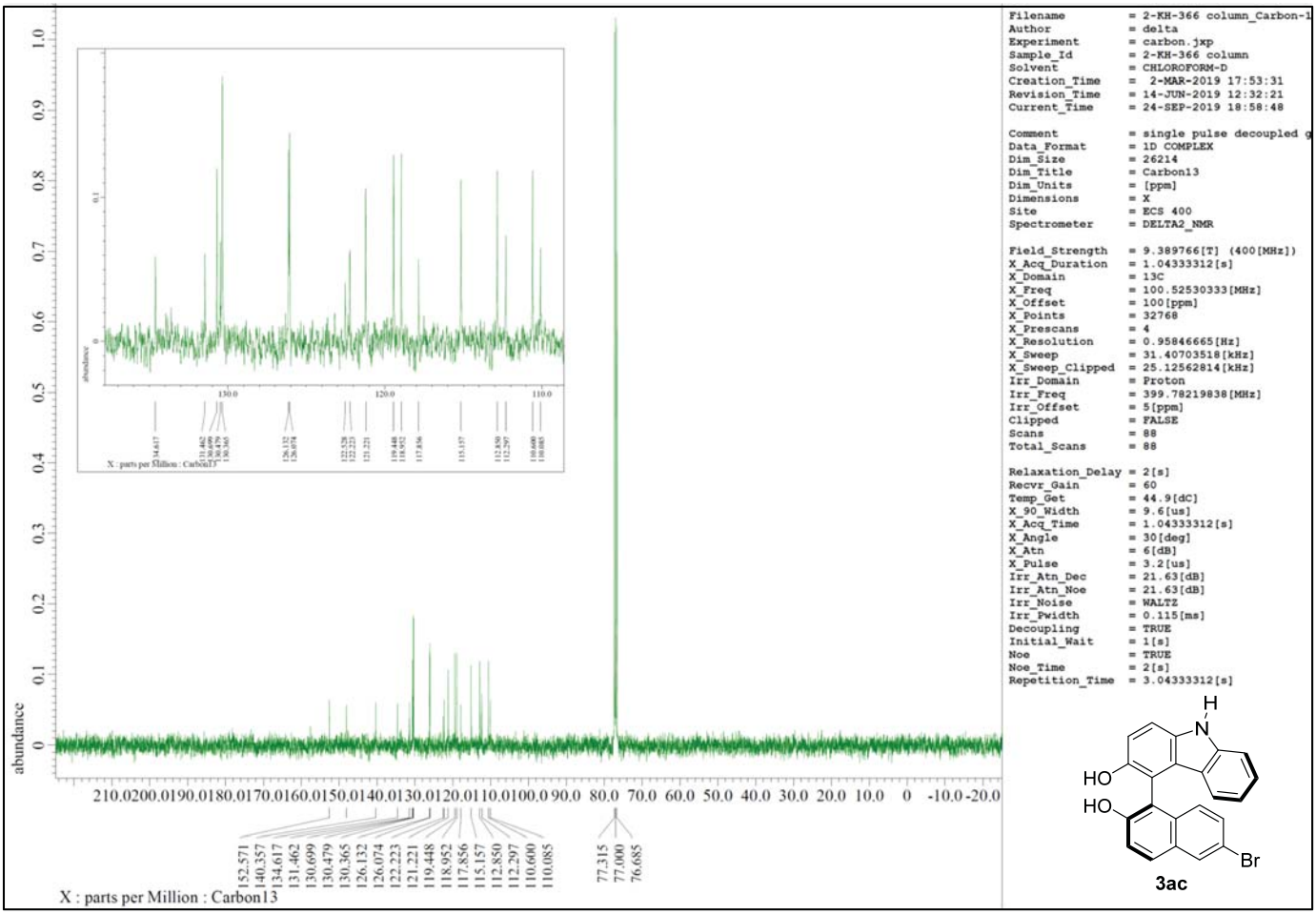

Filename = 2-KH-367 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-367 column
Solvent = ACETONE-D6
Creation_Time = 3-MAR-2019 15:38:28
Revision_Time = 26-SEP-2019 15:54:19
Current_Time = 26-SEP-2019 18:41:26
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 48
Temp_Get = 21.6[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
3ab



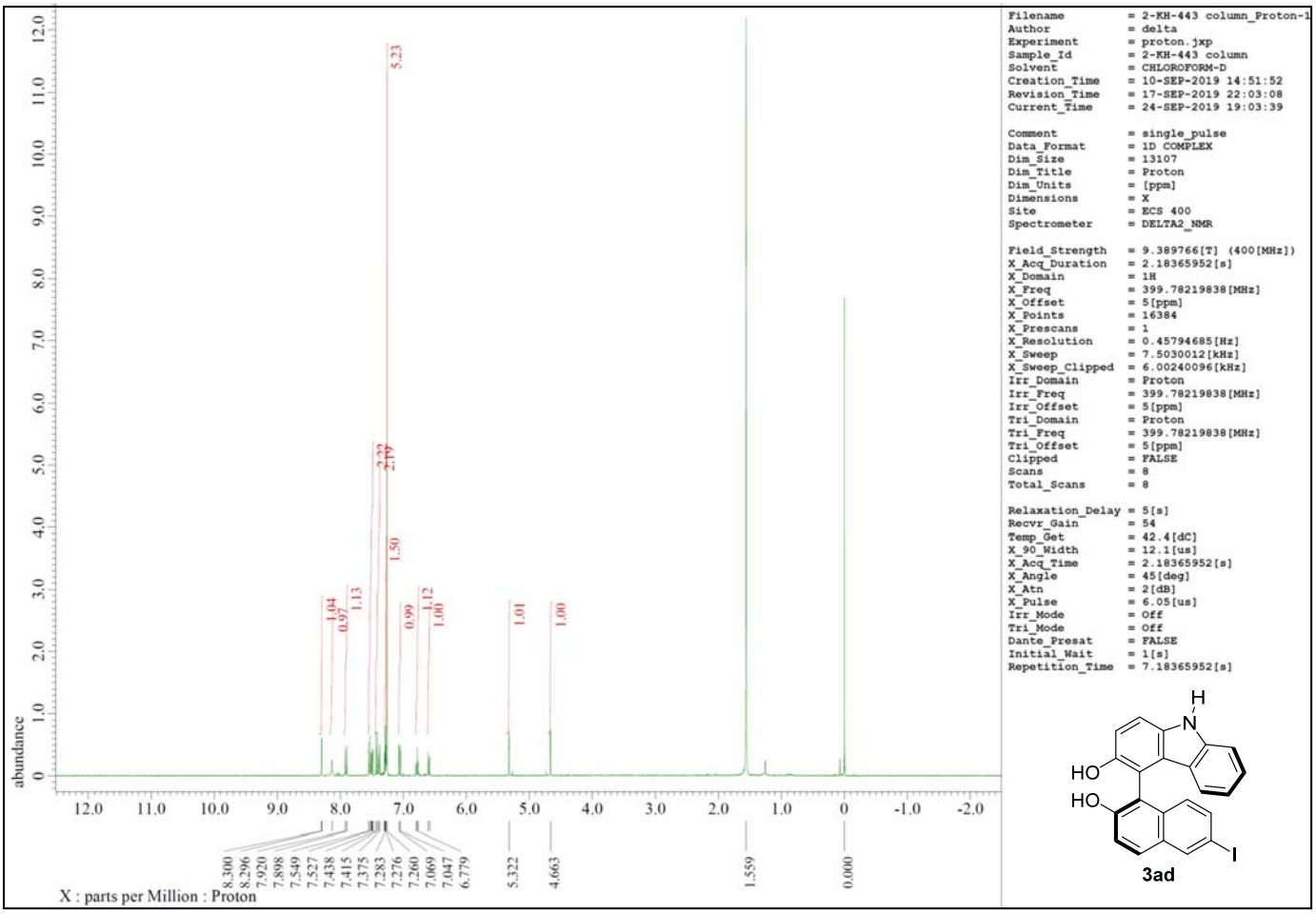
Compound **3ab** (^{1}H NMR, 400 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 100 MHz, $(CD_3)_2CO$)

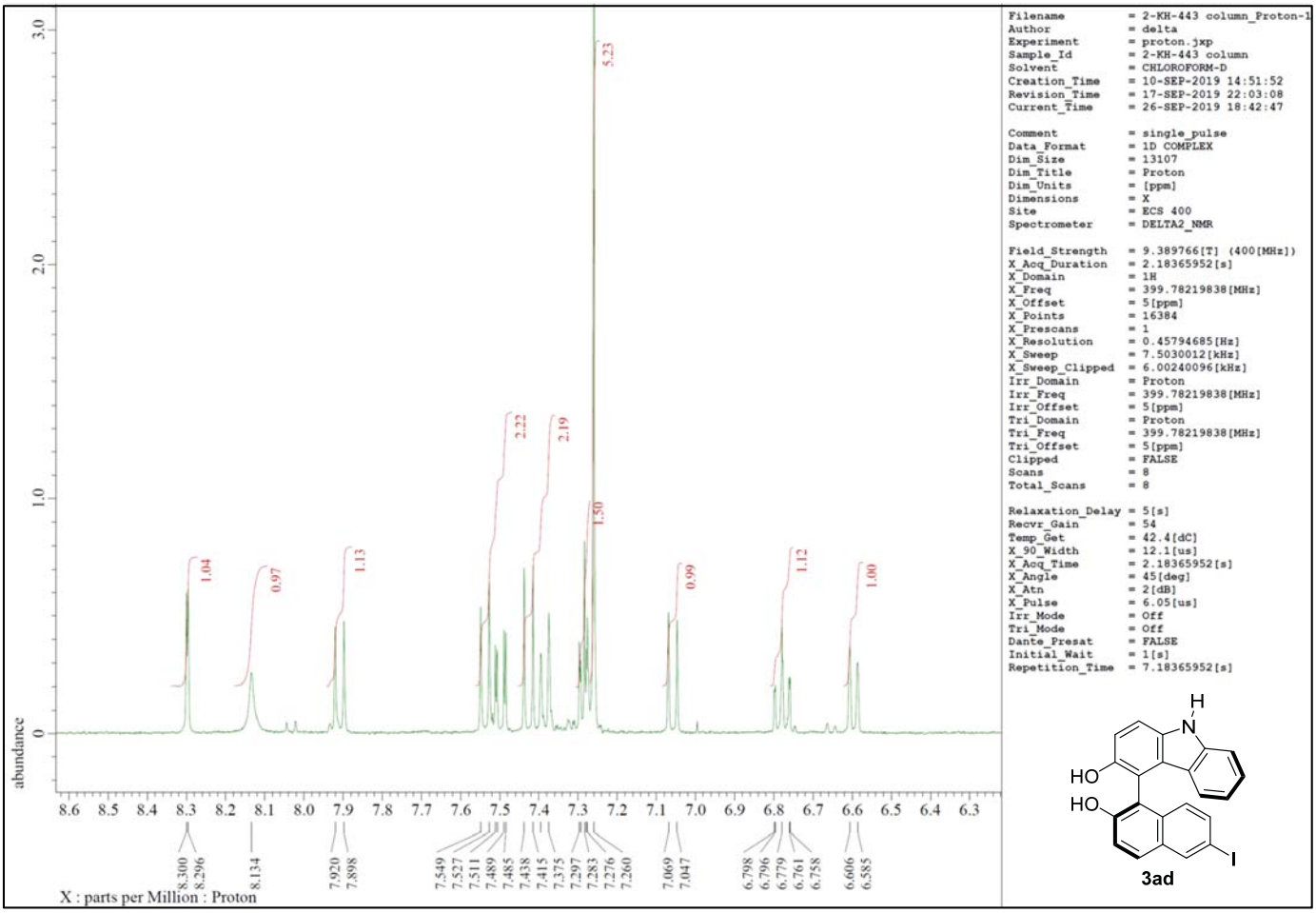

abundance
X : parts per Million : Proton
1.86
0.99
1.01
0.85
1.02
1.00
0.96
1.03
8.079
8.073
7.951
7.929
7.529
7.459
7.437
7.377
7.288
7.278
7.266
7.259
7.205
6.780
5.277
4.636
1.544
0.000
Filename = 2-KH-366 column_Proton-3
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-366 column
Solvent = CHLOROFORM-D
Creation_Time = 2-MAR-2019 16:52:31
Revision_Time = 26-SEP-2019 16:09:39
Current_Time = 26-SEP-2019 17:09:01
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 42[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
Br
3ac

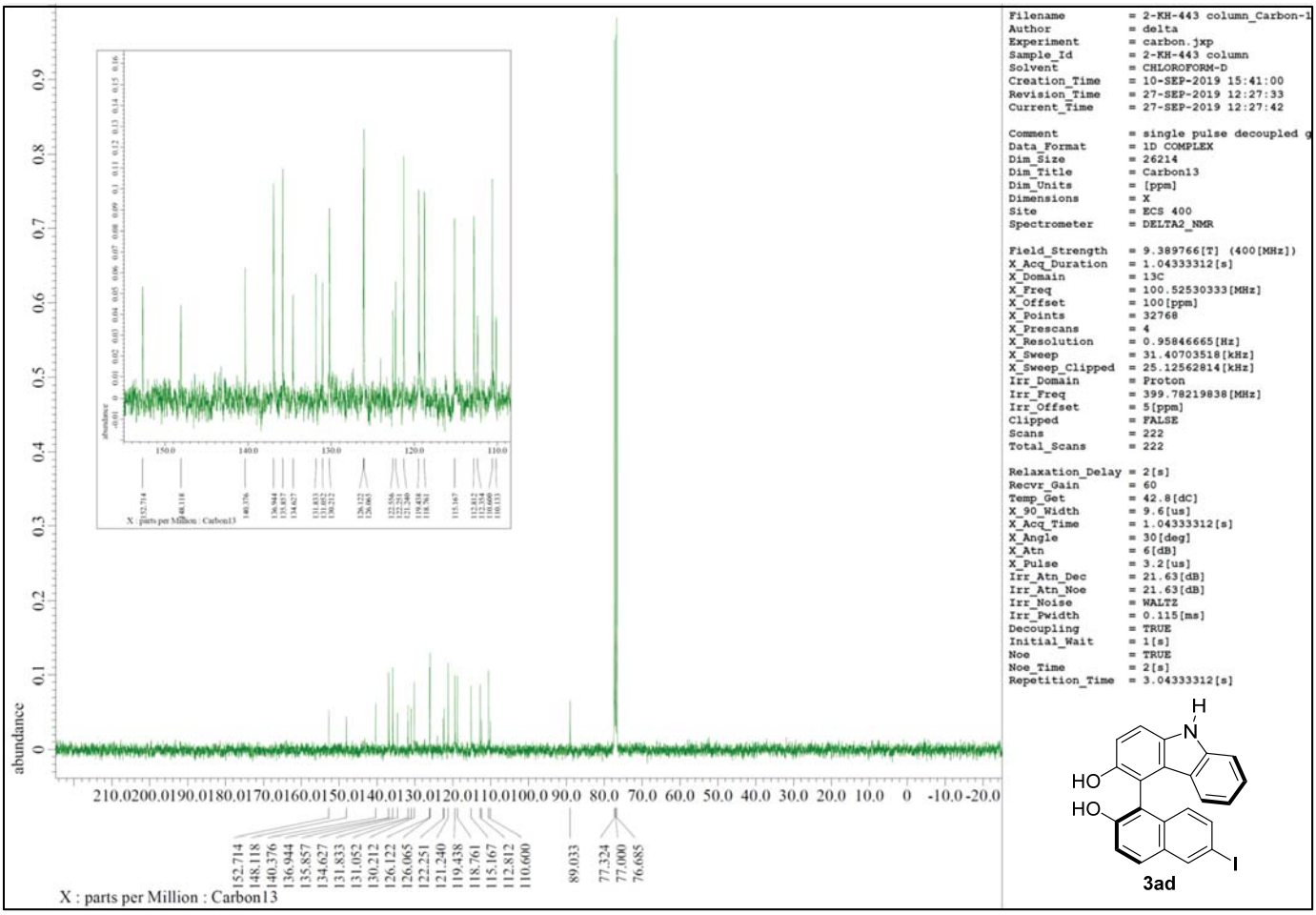

abundance
X : parts per Million : Proton
1.86
0.85
0.99
1.01
1.02
0.96
0.99
1.03
0.96
1.02
1.00
8.123
8.079
8.073
7.951
7.929
7.551
7.529
7.459
7.437
7.398
7.377
7.357
7.335
7.330
7.288
7.278
7.266
7.259
7.205
7.183
6.797
6.780
6.760
6.605
6.585
Filename = 2-KH-366 column_Proton-3
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-366 column
Solvent = CHLOROFORM-D
Creation_Time = 2-MAR-2019 16:52:31
Revision_Time = 26-SEP-2019 16:09:39
Current_Time = 26-SEP-2019 18:41:59
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 42[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
Br
3ac



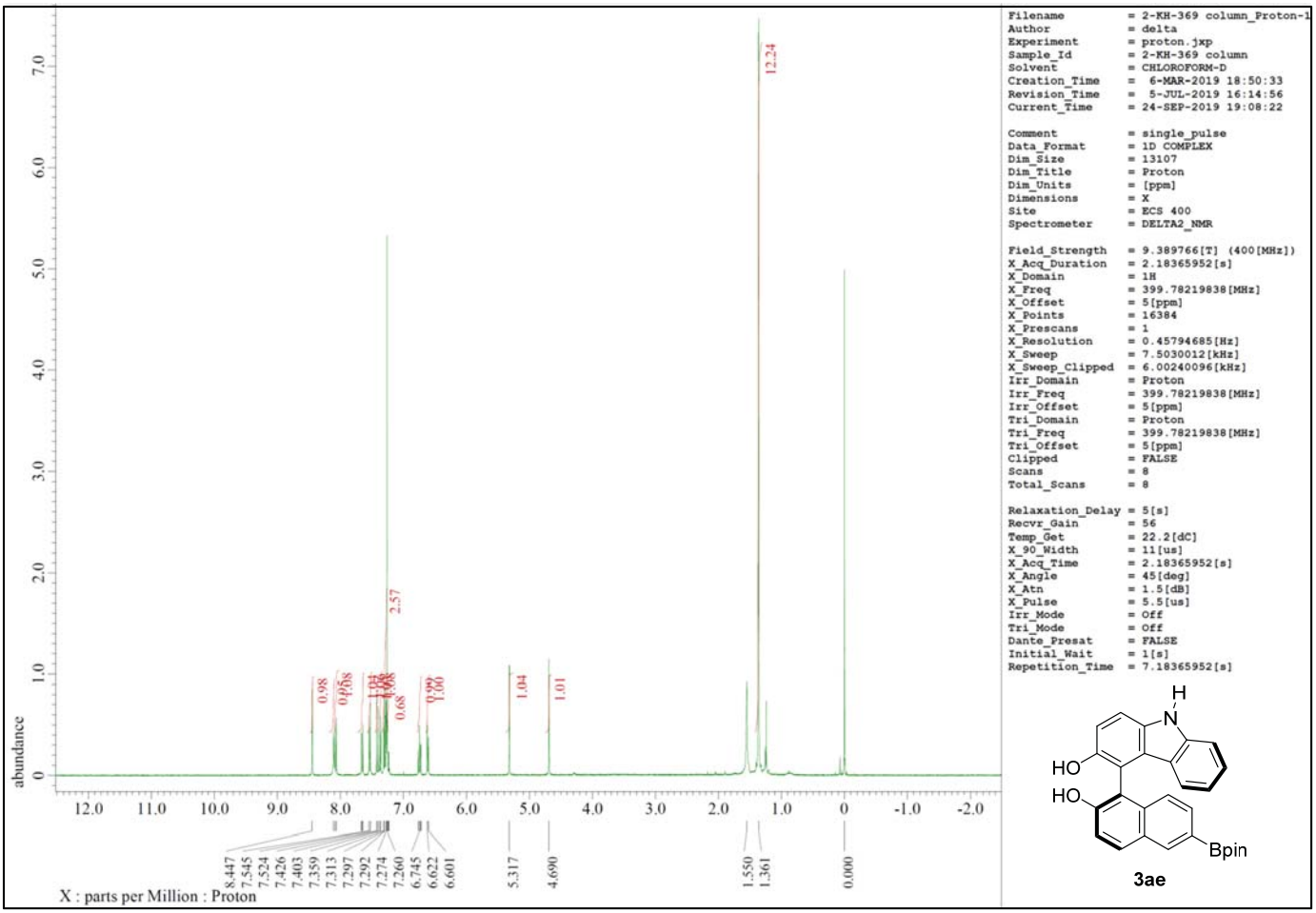
Compound **3ac** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

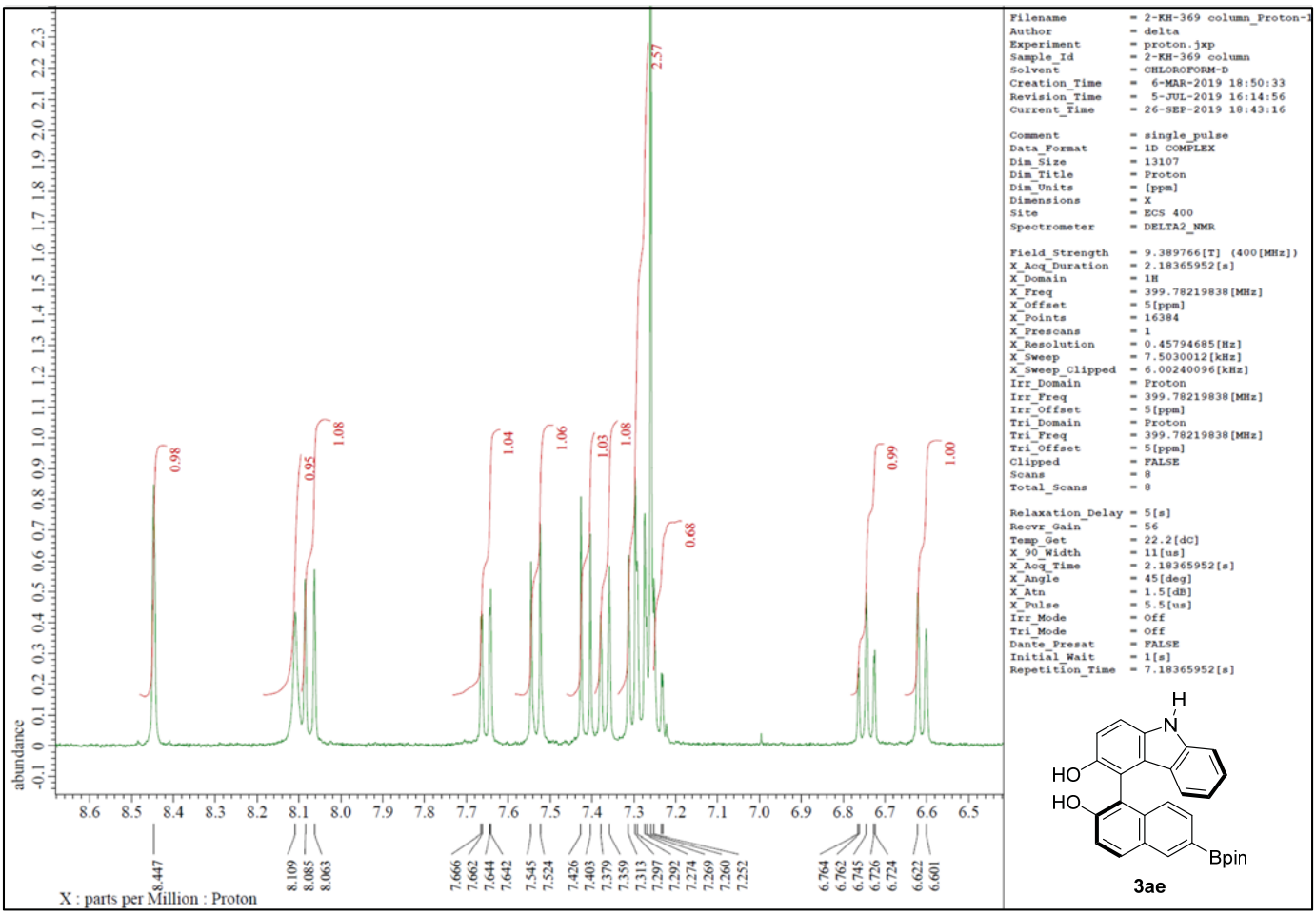

Filename = 2-KH-443 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-443 column
Solvent = CHLOROFORM-D
Creation_Time = 10-SEP-2019 14:51:52
Revision_Time = 17-SEP-2019 22:03:08
Current_Time = 24-SEP-2019 19:03:39
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Get = 42.4[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
HO
HO
H
N
I
3ad

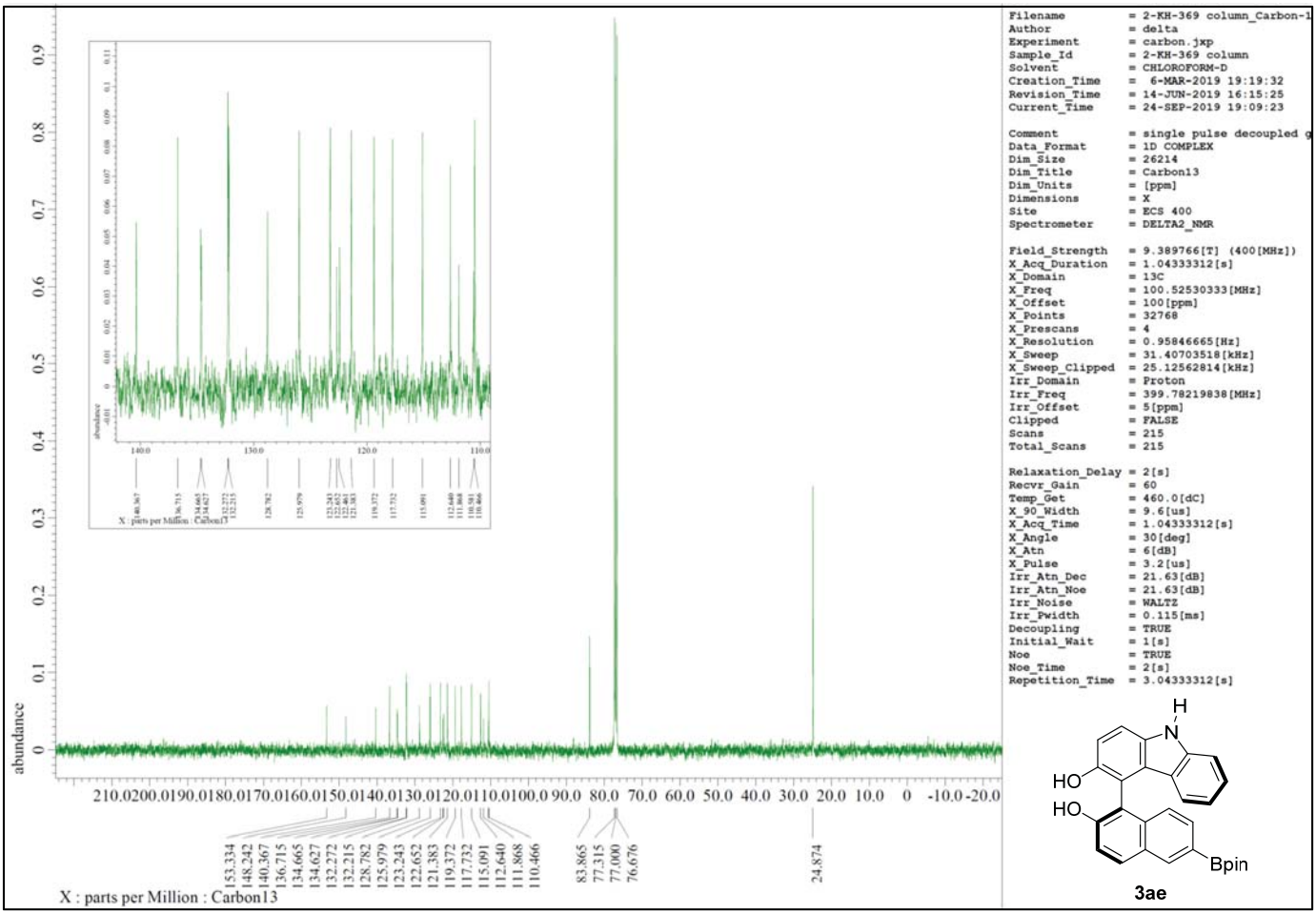

Filename = 2-KH-443 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-443 column
Solvent = CHLOROFORM-D
Creation_Time = 10-SEP-2019 14:51:52
Revision_Time = 17-SEP-2019 22:03:08
Current_Time = 26-SEP-2019 18:42:47
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Get = 42.4[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
HO
HO
H
N
I
3ad



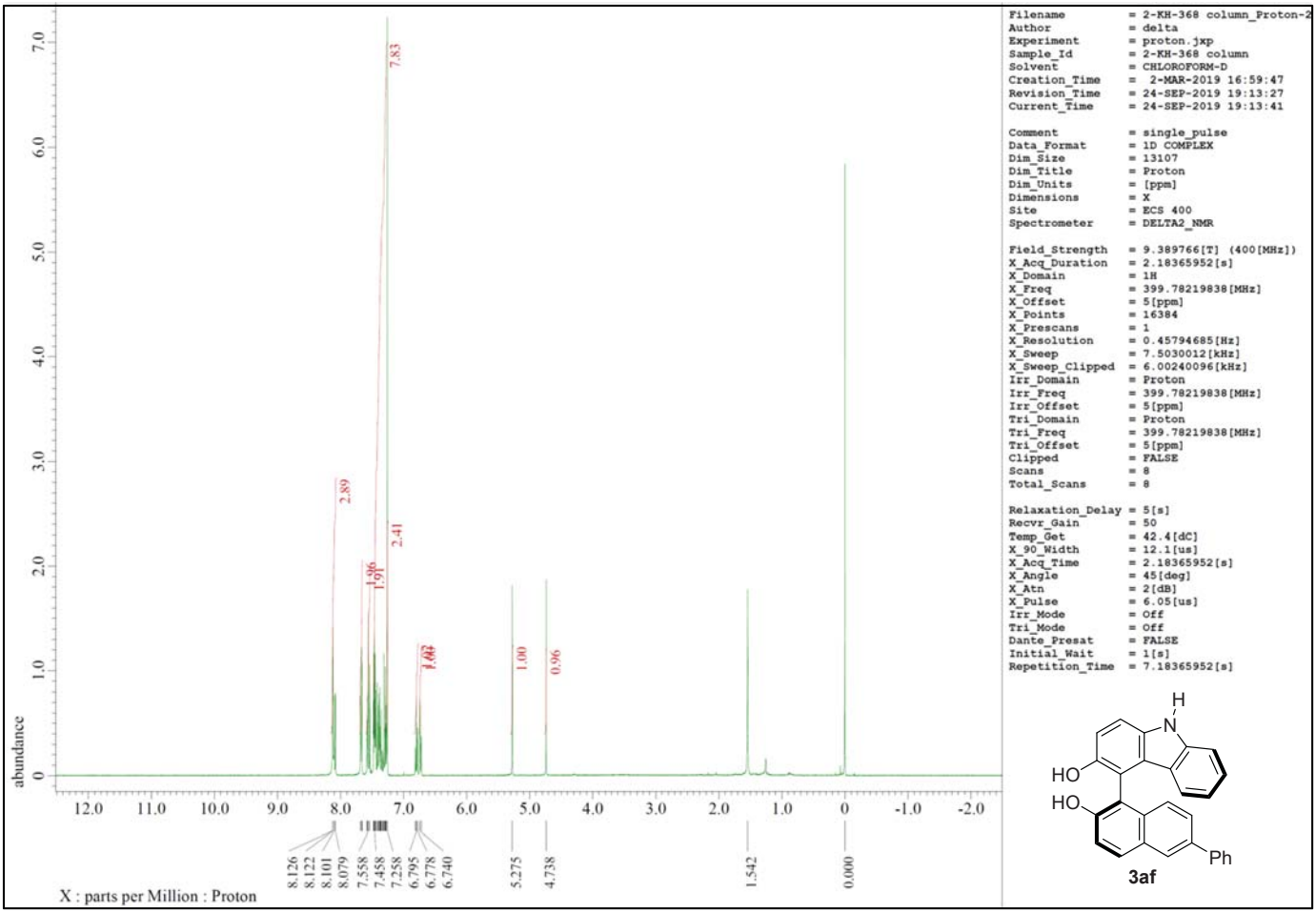
Compound **3ad** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)


Filename = 2-KH-369 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-369 column
Solvent = CHLOROFORM-D
Creation_Time = 6-MAR-2019 18:50:33
Revision_Time = 5-JUL-2019 16:14:56
Current_Time = 24-SEP-2019 19:08:22
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.2[dC]
X_90_Width = 11[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.5[dB]
X_Pulse = 5.5[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
12.24
2.57
0.98
1.04
1.01
0.68
8.447
7.545
7.524
7.426
7.403
7.359
7.313
7.297
7.292
7.274
7.260
6.745
6.622
6.601
5.317
4.690
1.550
1.361
0.000
H
N
HO
HO
Bpin
3ae


Filename = 2-KH-369 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-369 column
Solvent = CHLOROFORM-D
Creation_Time = 6-MAR-2019 18:50:33
Revision_Time = 5-JUL-2019 16:14:56
Current_Time = 26-SEP-2019 18:43:16
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 22.2[dC]
X_90_Width = 11[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.5[dB]
X_Pulse = 5.5[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
2.57
0.98
0.95
1.08
1.04
1.06
1.03
1.08
0.68
0.99
1.00
8.447
8.109
8.085
8.063
7.666
7.662
7.644
7.642
7.545
7.524
7.426
7.403
7.379
7.359
7.313
7.297
7.292
7.274
7.269
7.260
7.252
6.764
6.762
6.745
6.726
6.724
6.622
6.601
H
N
HO
HO
Bpin
3ae



Compound **3ae** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)



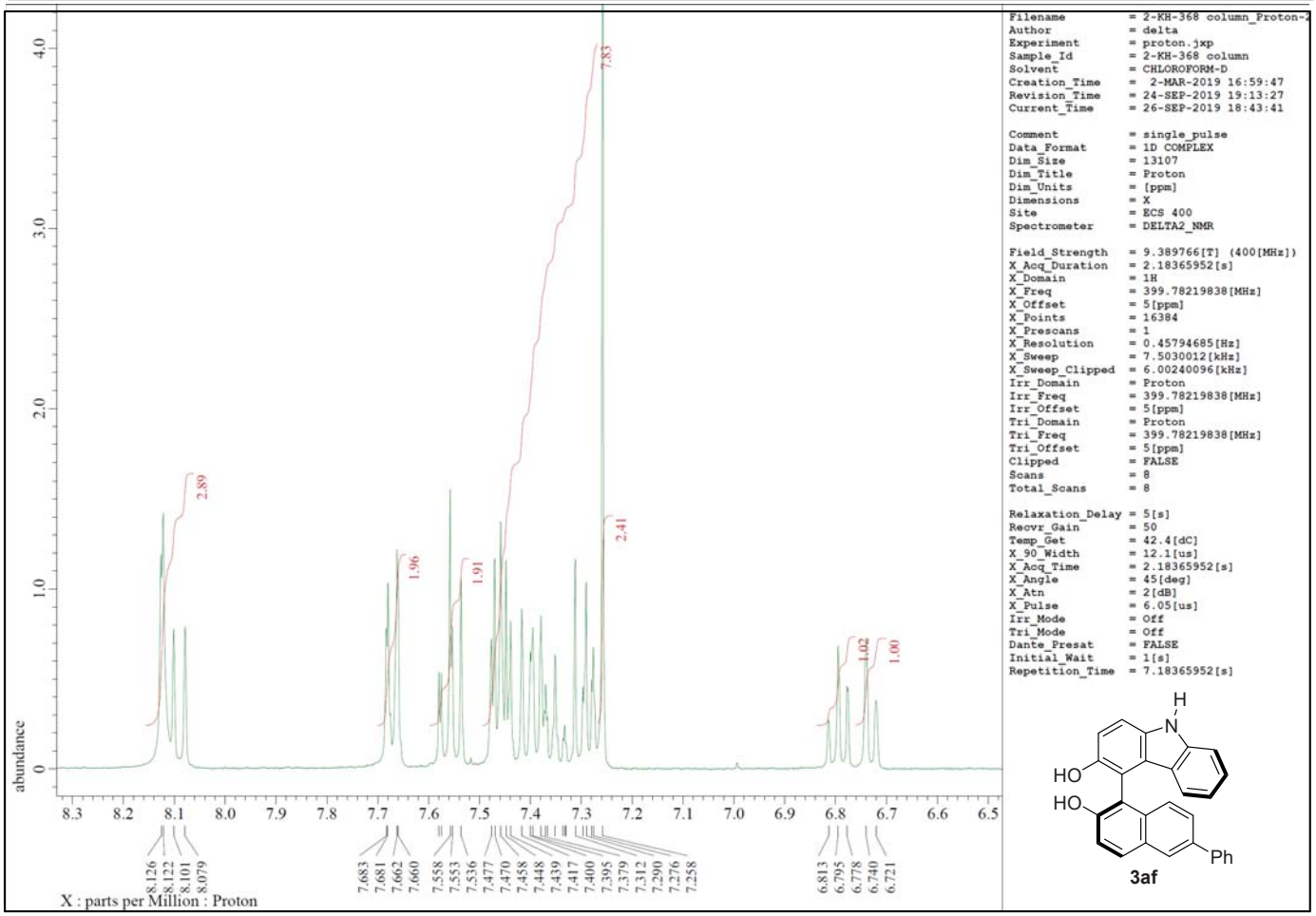

```
Filename          = 2-KH-368 column_Proton-2
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-368 column
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAR-2019 16:59:47
Revision_Time     = 24-SEP-2019 19:13:27
Current_Time      = 24-SEP-2019 19:13:41

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 42.4[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```


3af



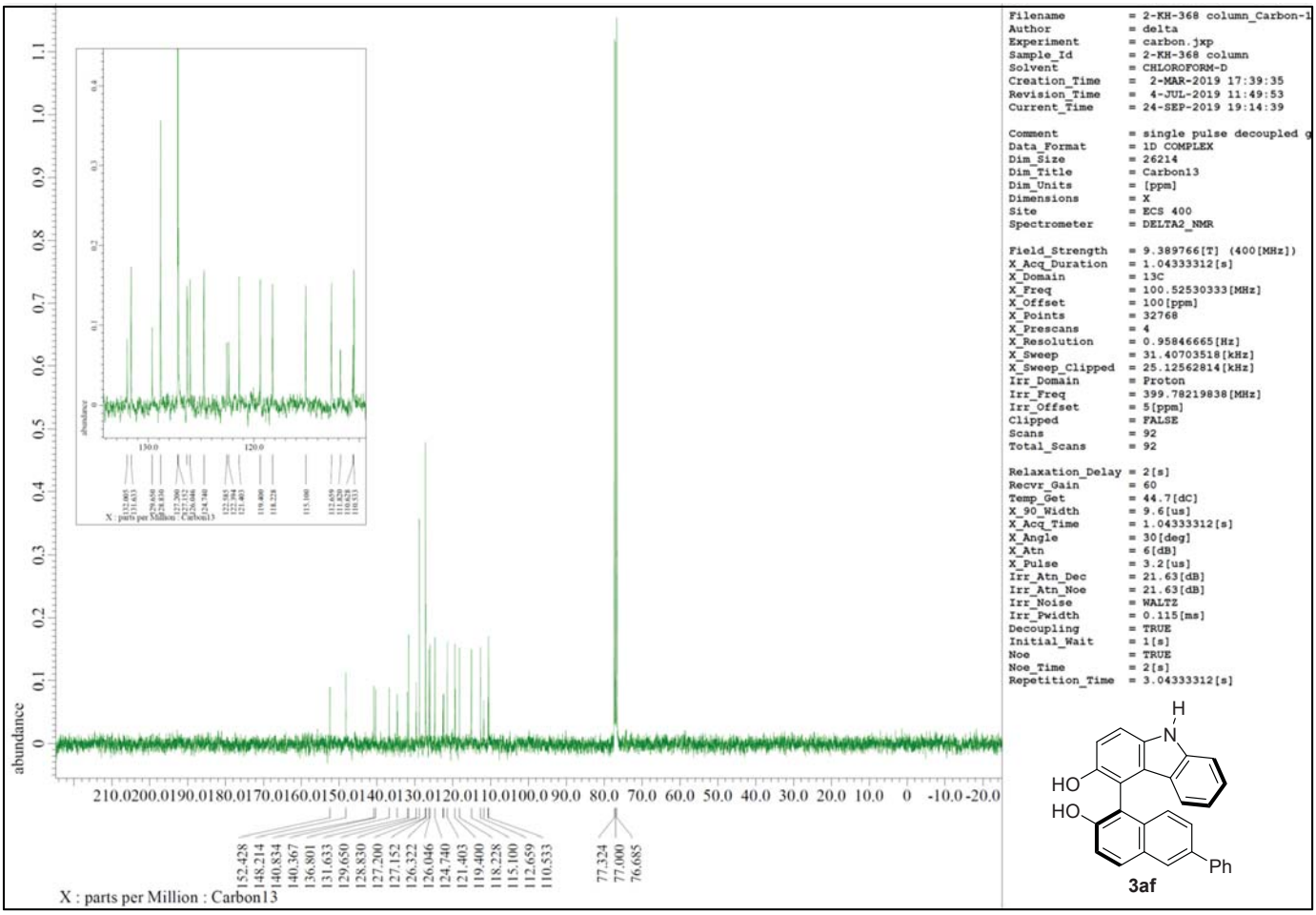

```
Filename          = 2-KH-368 column_Proton-2
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-368 column
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAR-2019 16:59:47
Revision_Time     = 24-SEP-2019 19:13:27
Current_Time      = 26-SEP-2019 18:43:41

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

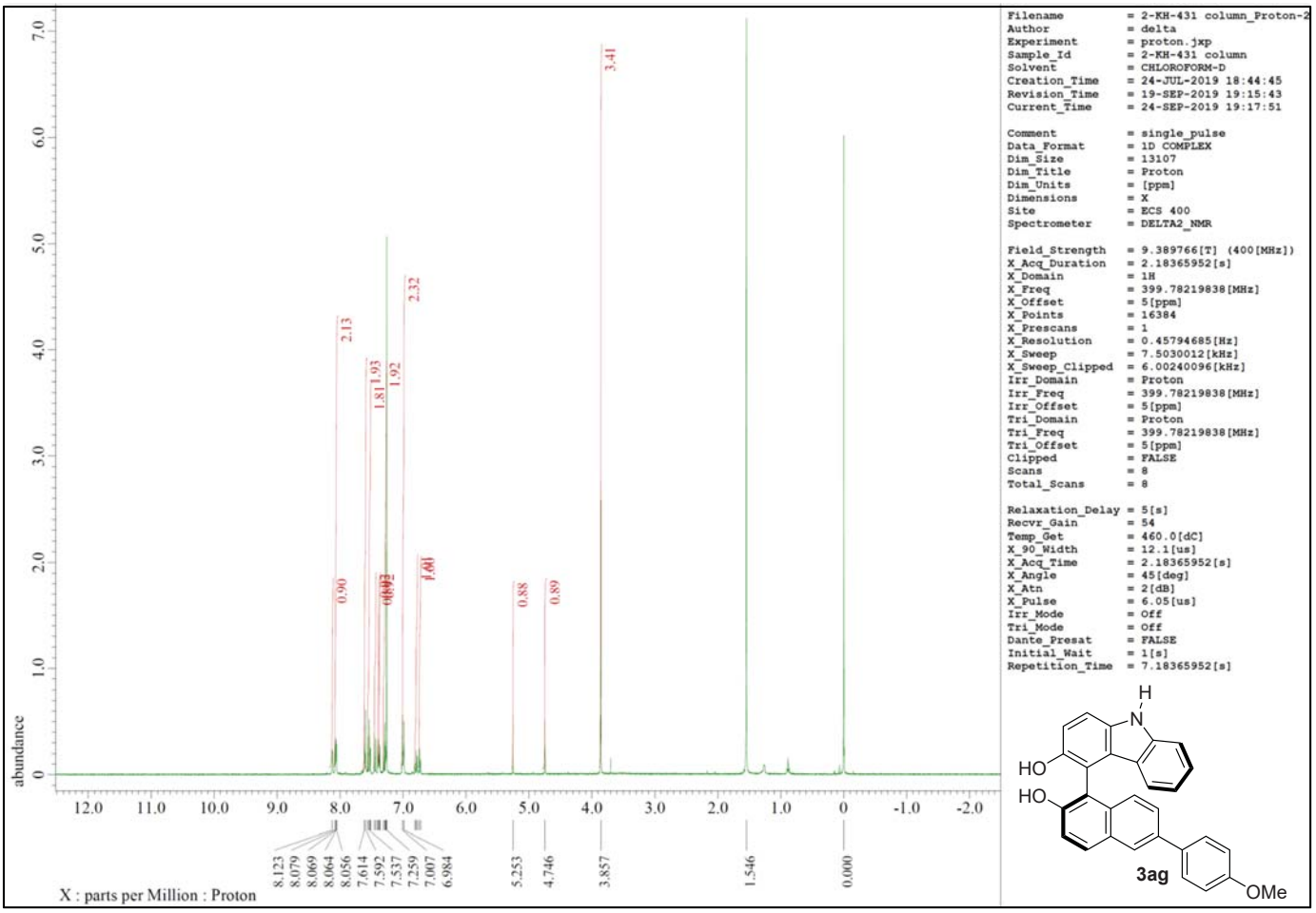
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 42.4[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```


3af



Compound **3af** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

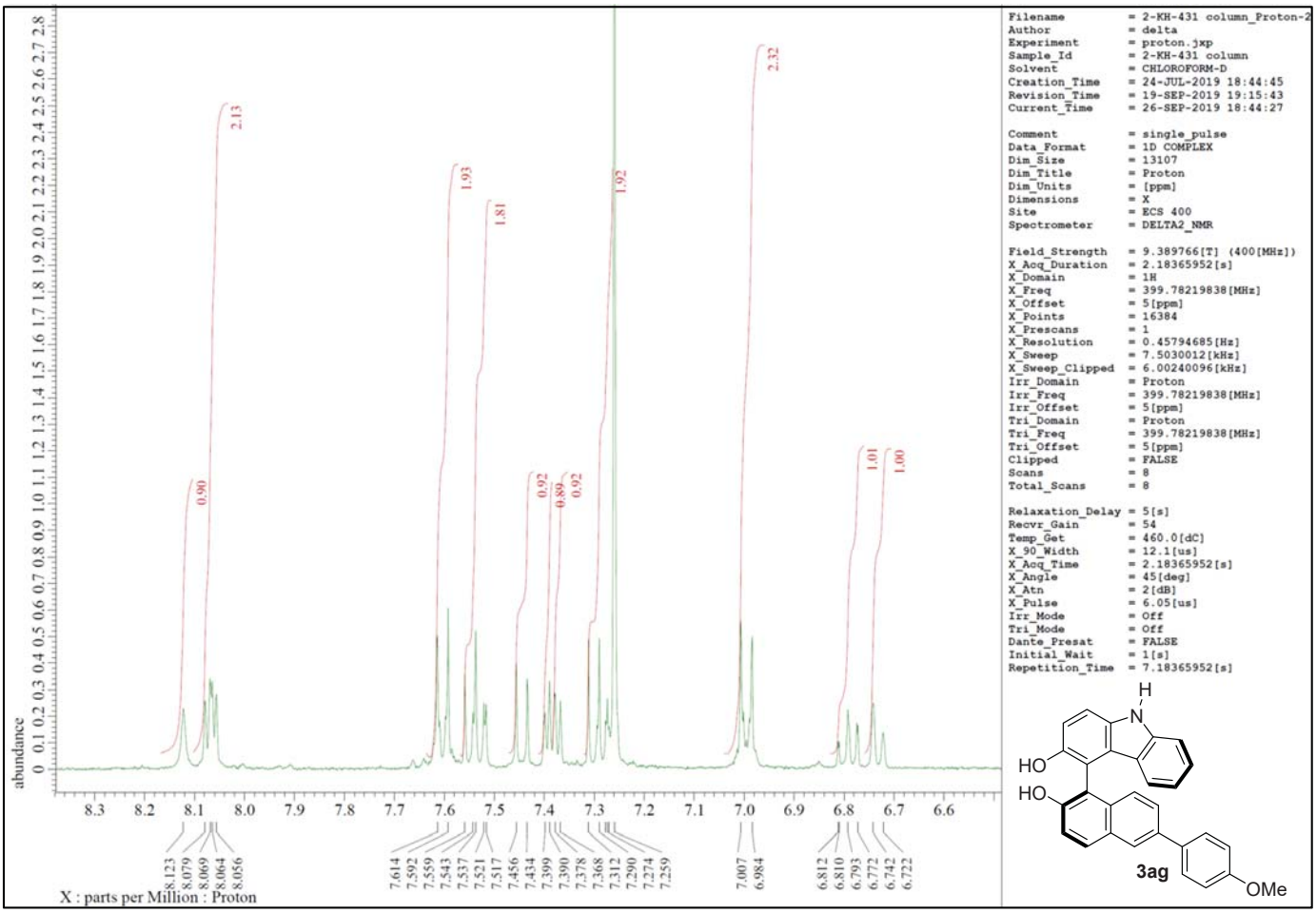



```
Filename          = 2-KH-431 column_Proton-2
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-431 column
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JUL-2019 18:44:45
Revision_Time     = 19-SEP-2019 19:15:43
Current_Time      = 24-SEP-2019 19:17:51

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 460.0[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```

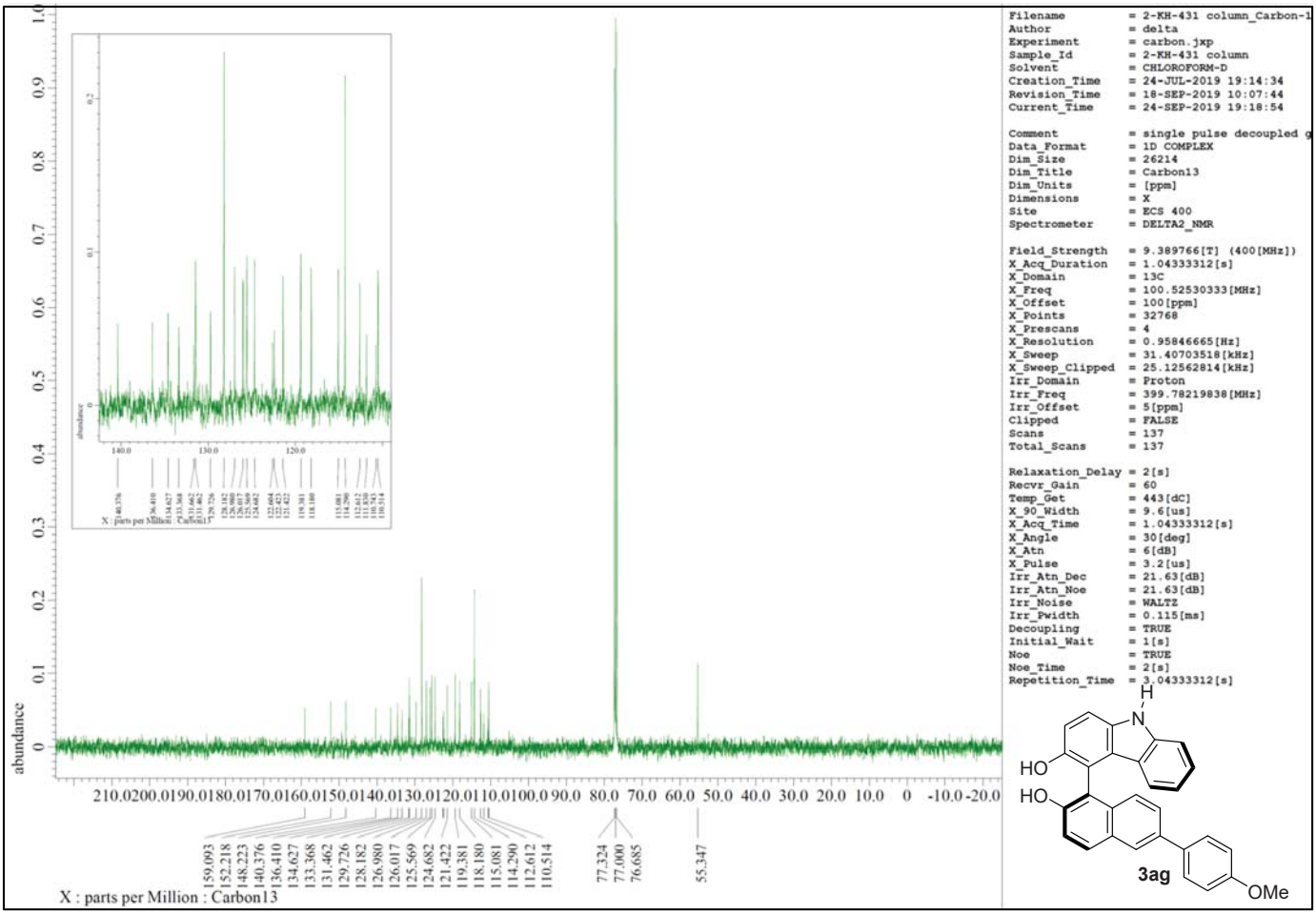



```
Filename          = 2-KH-431 column_Proton-2
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-431 column
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JUL-2019 18:44:45
Revision_Time     = 19-SEP-2019 19:15:43
Current_Time      = 26-SEP-2019 18:44:27

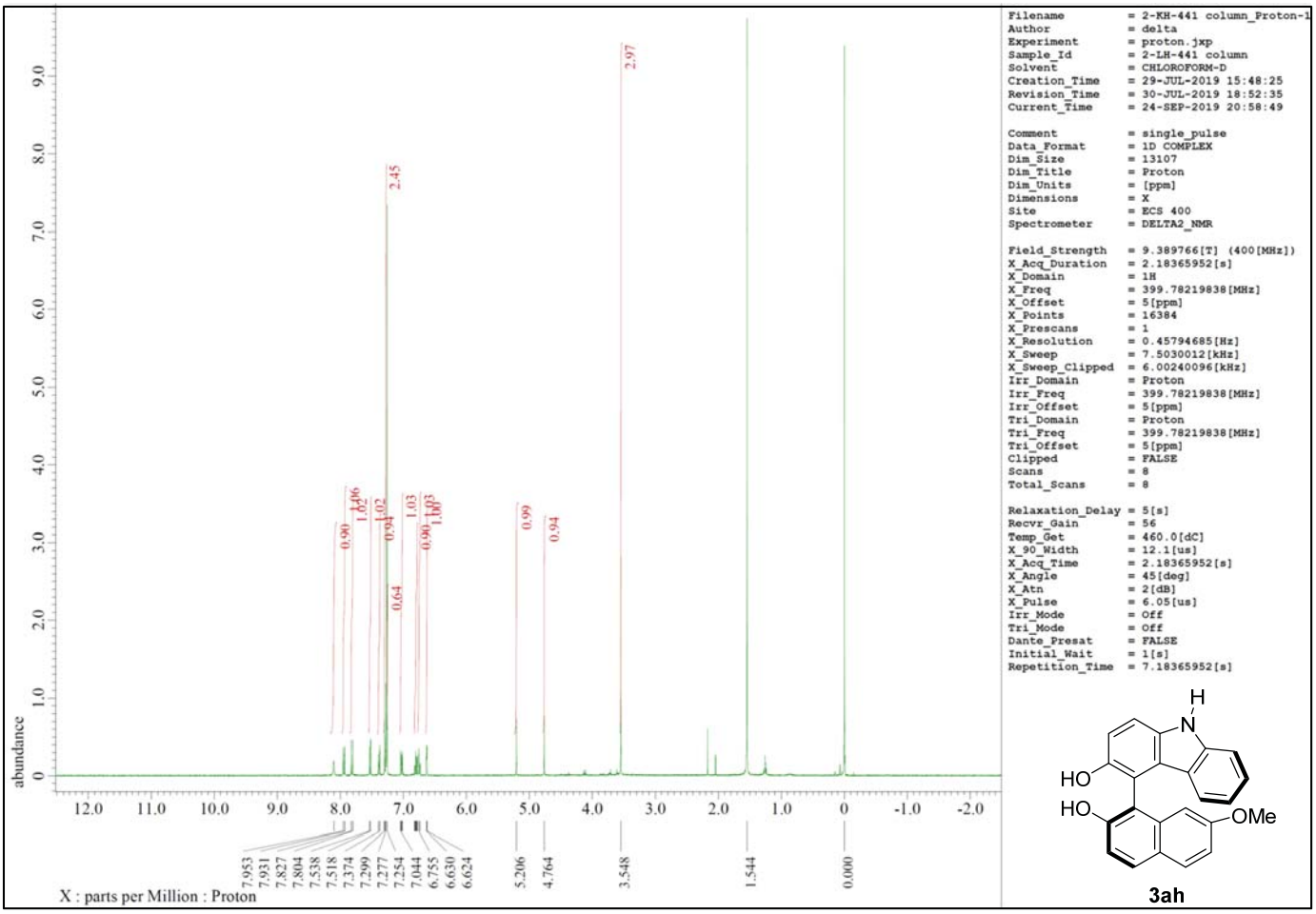
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

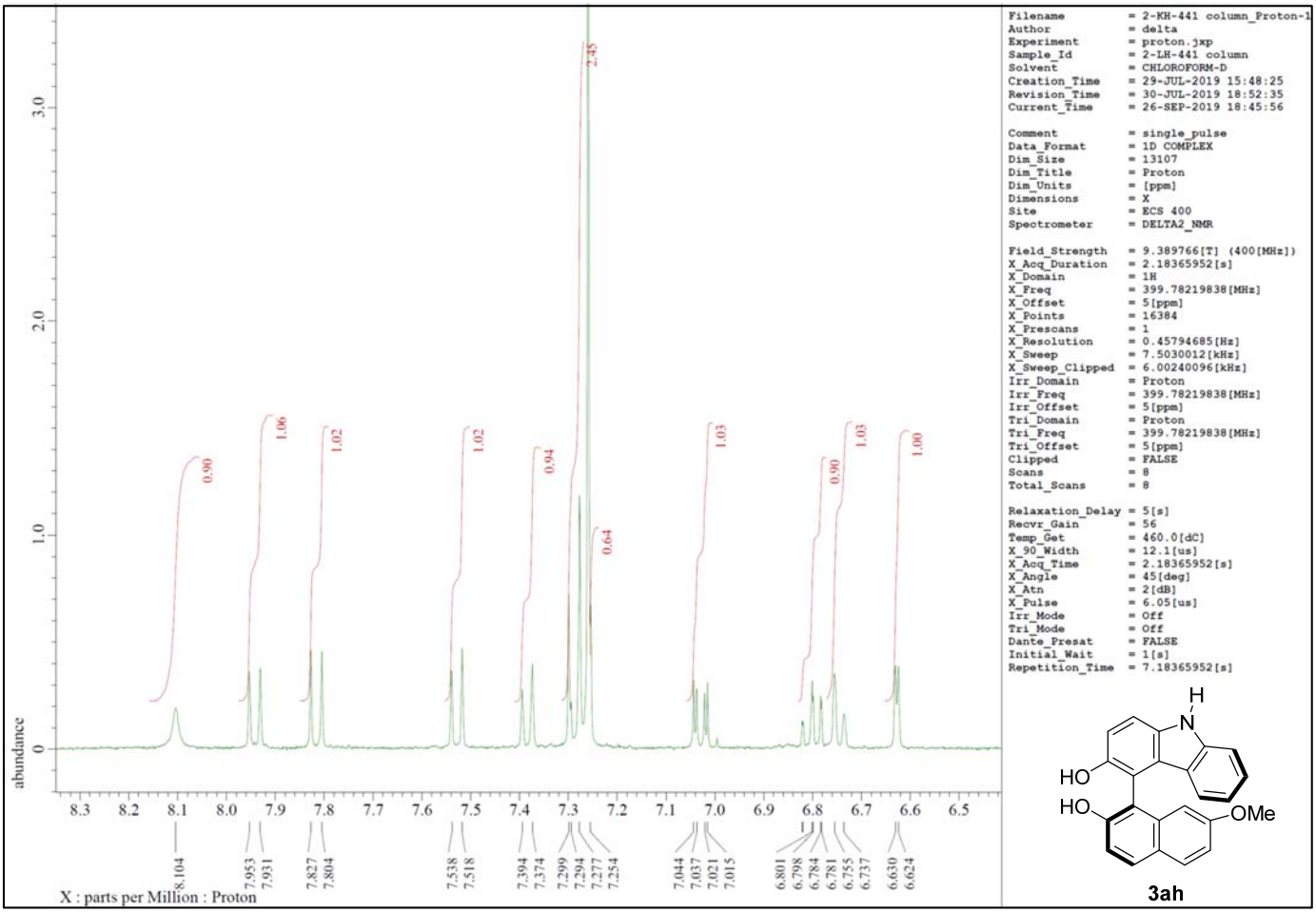
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

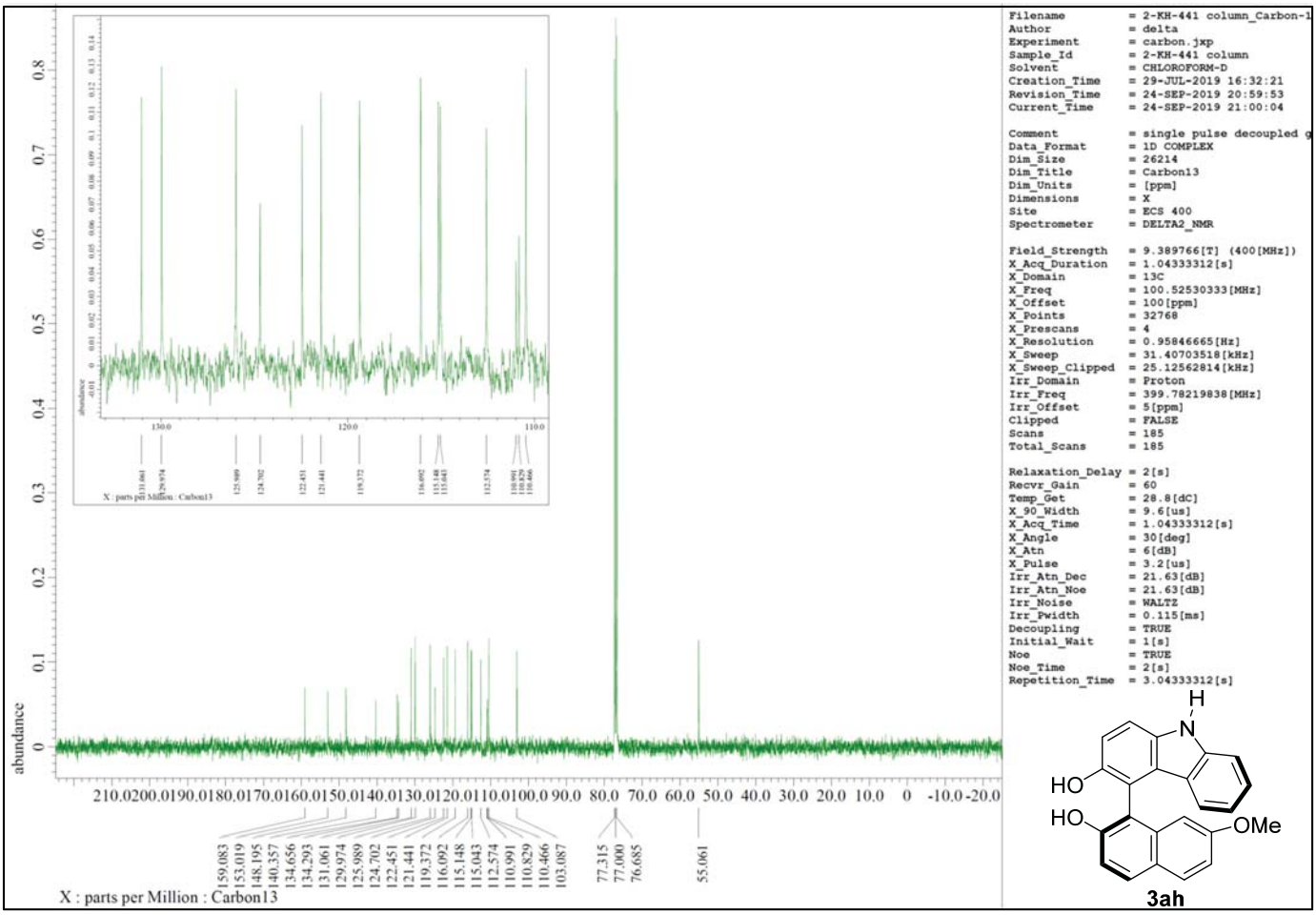
Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 460.0[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



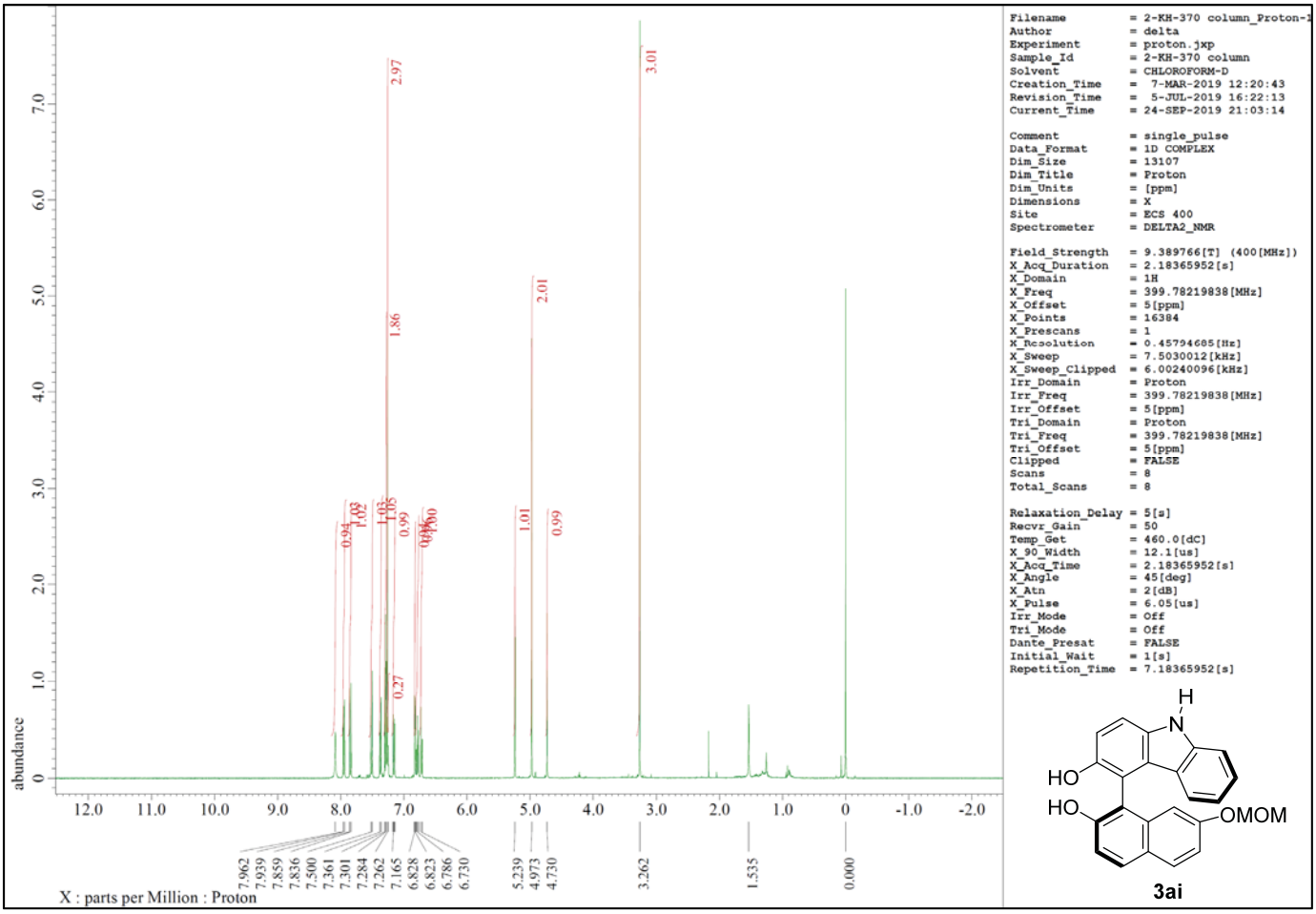
Compound **3ag** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

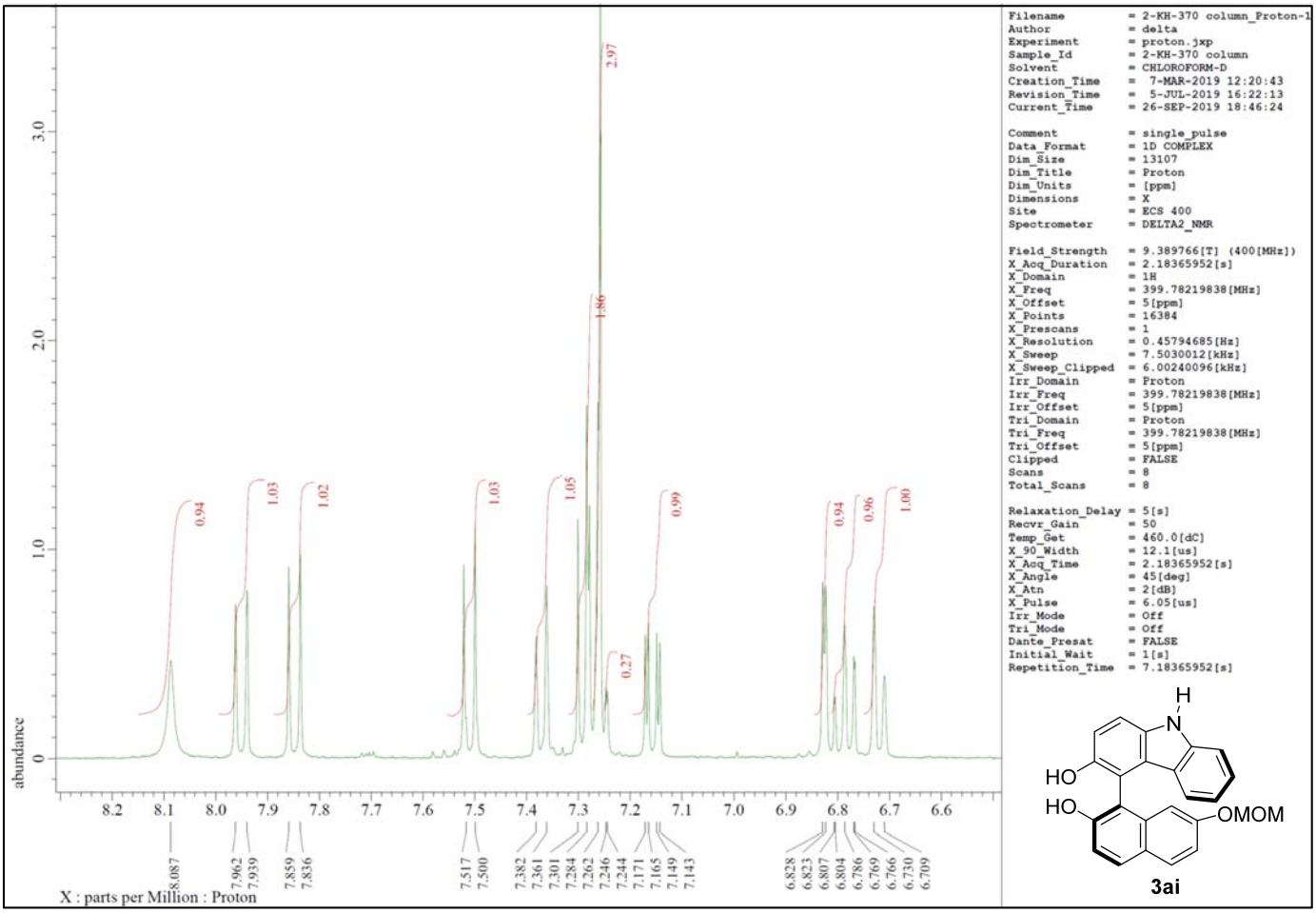

Filename = 2-KH-441 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-LH-441 column
Solvent = CHLOROFORM-D
Creation_Time = 29-JUL-2019 15:48:25
Revision_Time = 30-JUL-2019 18:52:35
Current_Time = 24-SEP-2019 20:58:49
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
7.953 7.931 7.827 7.804 7.538 7.518 7.374 7.299 7.277 7.254 7.044 6.755 6.630 6.624 5.206 4.764 3.548 1.544 0.000
HO
HO
OMe
H
N
3ah

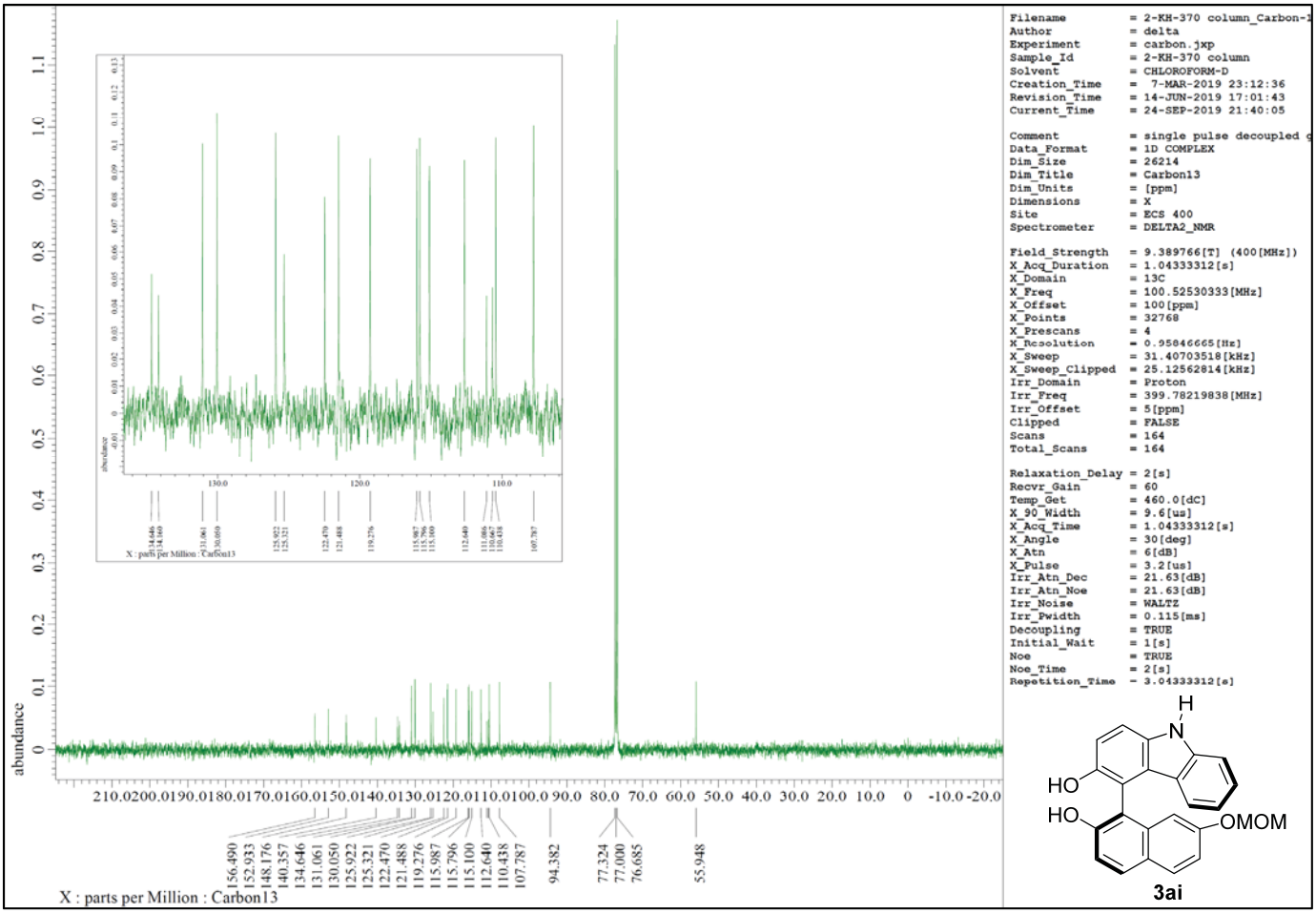

Filename = 2-KH-441 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-LH-441 column
Solvent = CHLOROFORM-D
Creation_Time = 29-JUL-2019 15:48:25
Revision_Time = 30-JUL-2019 18:52:35
Current_Time = 26-SEP-2019 18:45:56
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
8.104 7.953 7.931 7.827 7.804 7.538 7.518 7.394 7.374 7.299 7.294 7.277 7.254 7.044 7.037 7.021 7.015 6.801 6.798 6.784 6.781 6.755 6.737 6.630 6.624
HO
HO
OMe
H
N
3ah



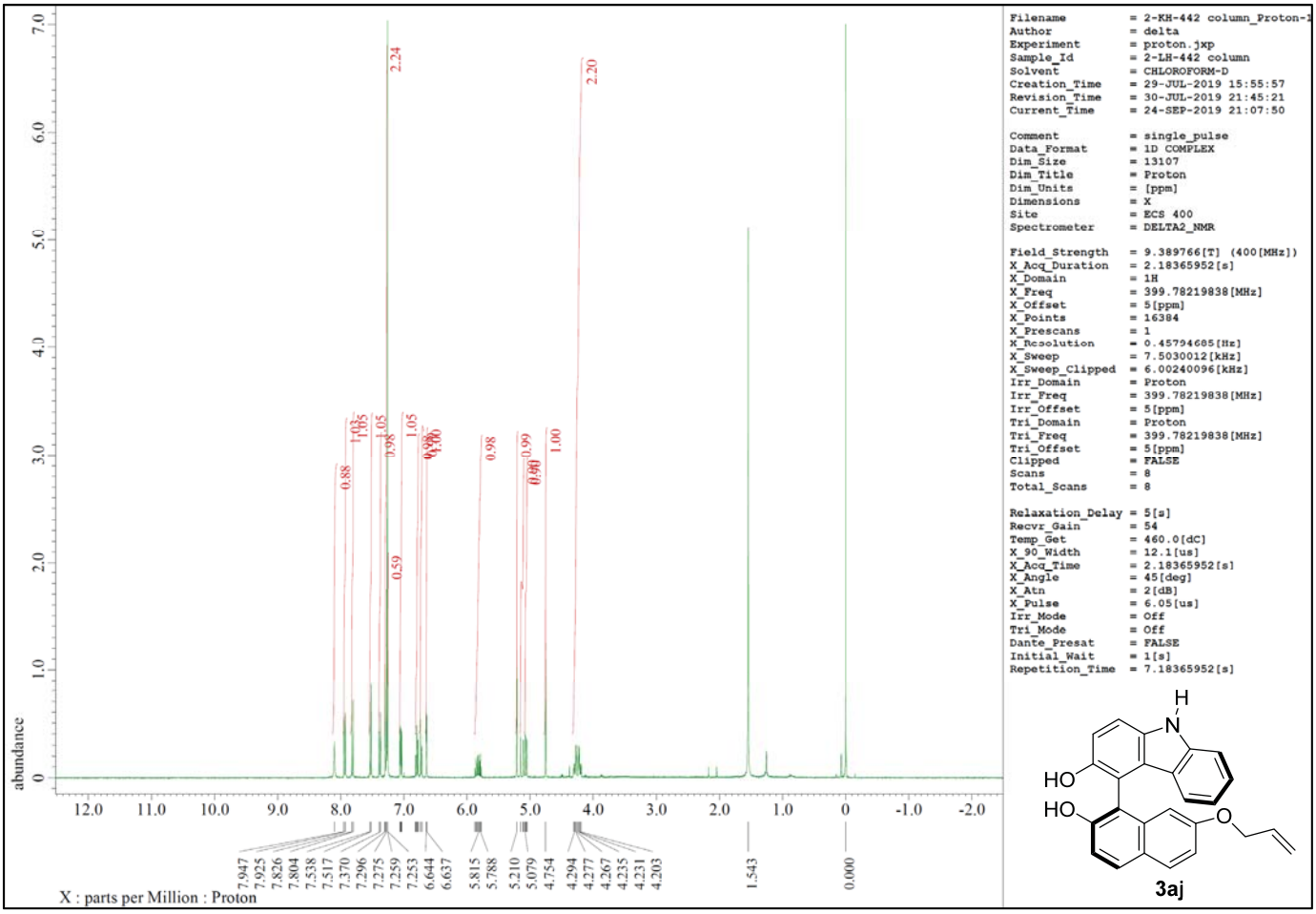
Compound **3ah** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

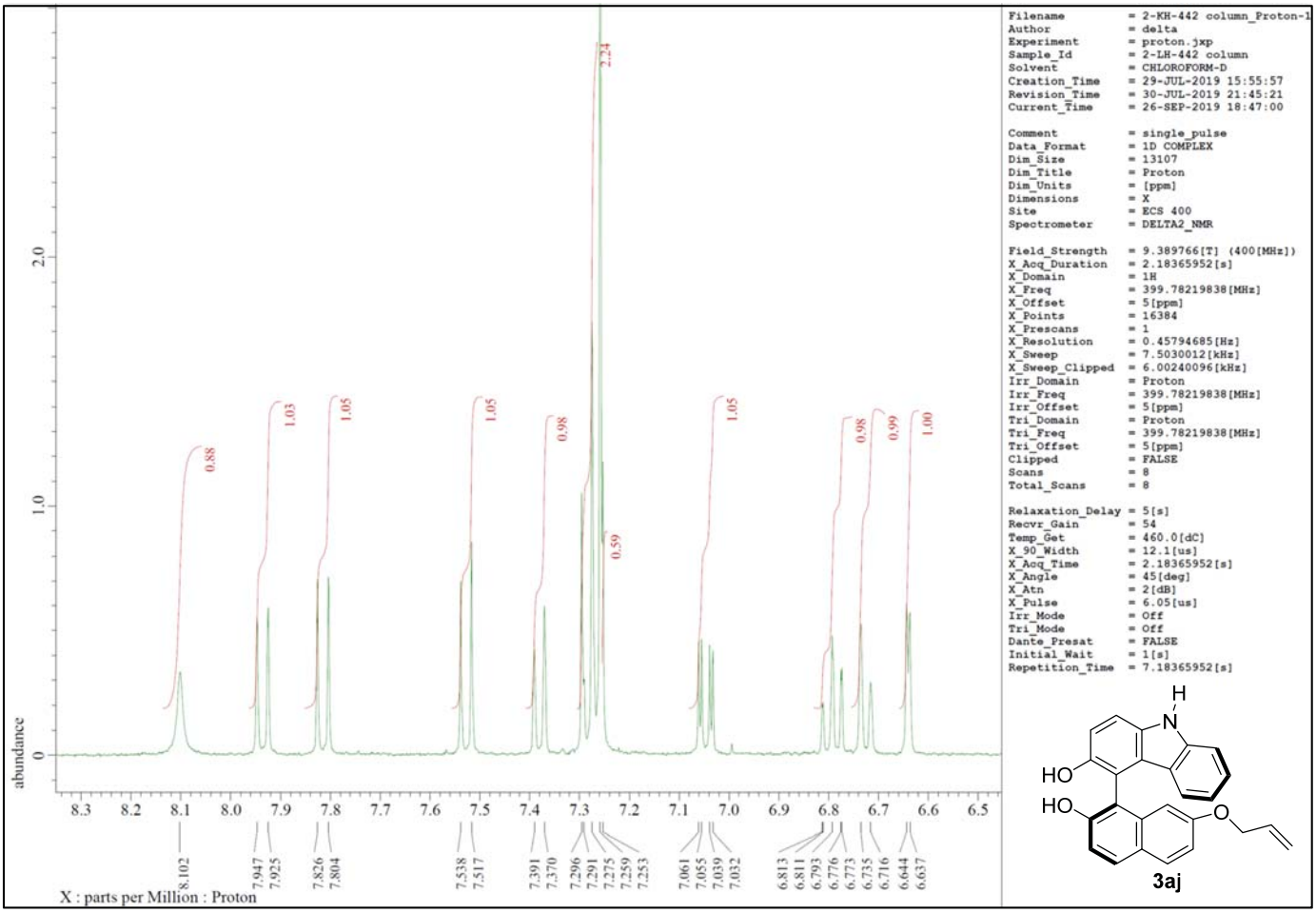

abundance
X : parts per Million : Proton
Filename = 2-KH-370 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-370 column
Solvent = CHLOROFORM-D
Creation_Time = 7-MAR-2019 12:20:43
Revision_Time = 5-JUL-2019 16:22:13
Current_Time = 24-SEP-2019 21:03:14
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
HO
HO
OMOM
3ai

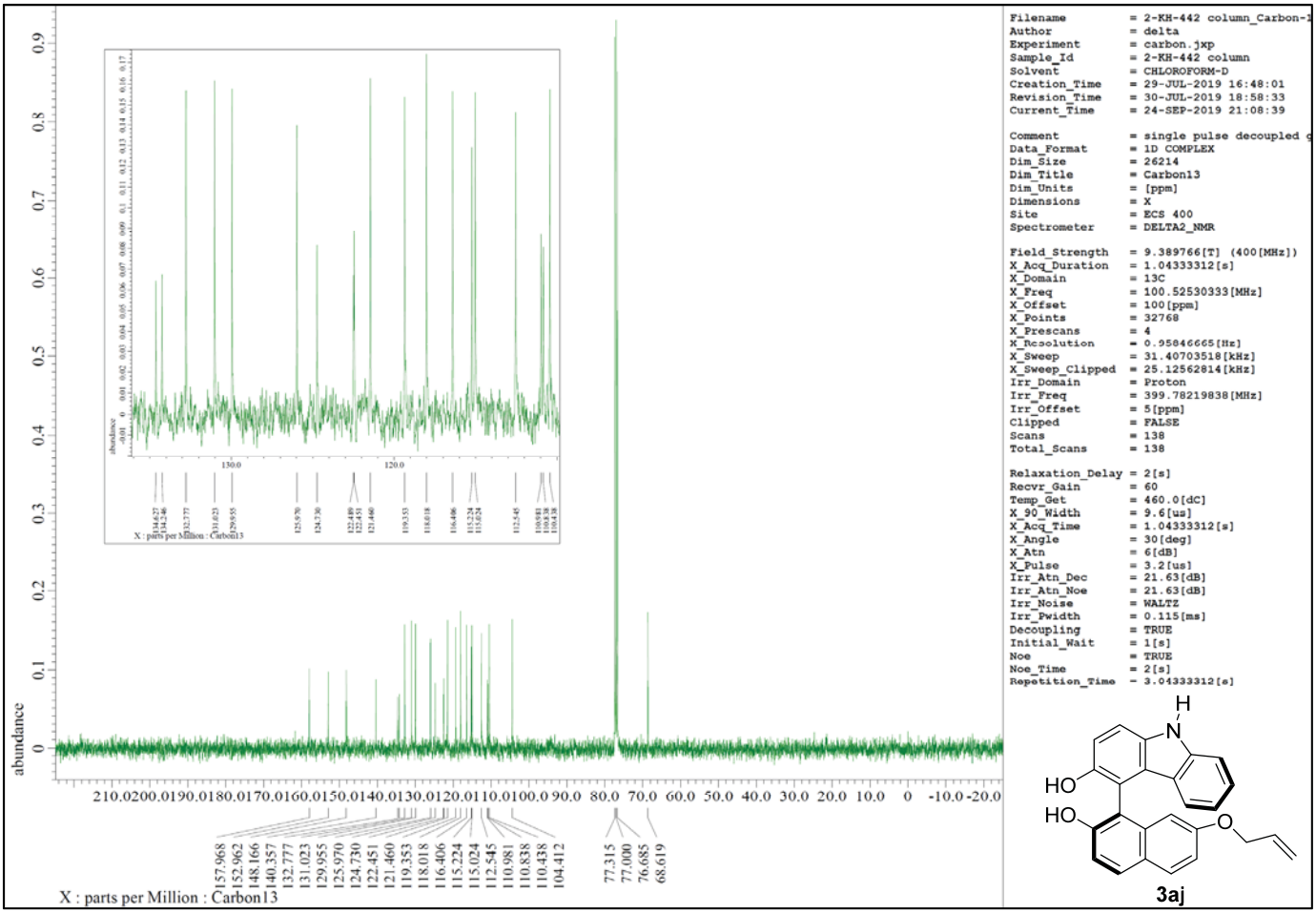

abundance
X : parts per Million : Proton
Filename = 2-KH-370 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-370 column
Solvent = CHLOROFORM-D
Creation_Time = 7-MAR-2019 12:20:43
Revision_Time = 5-JUL-2019 16:22:13
Current_Time = 26-SEP-2019 18:46:24
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
HO
HO
OMOM
3ai



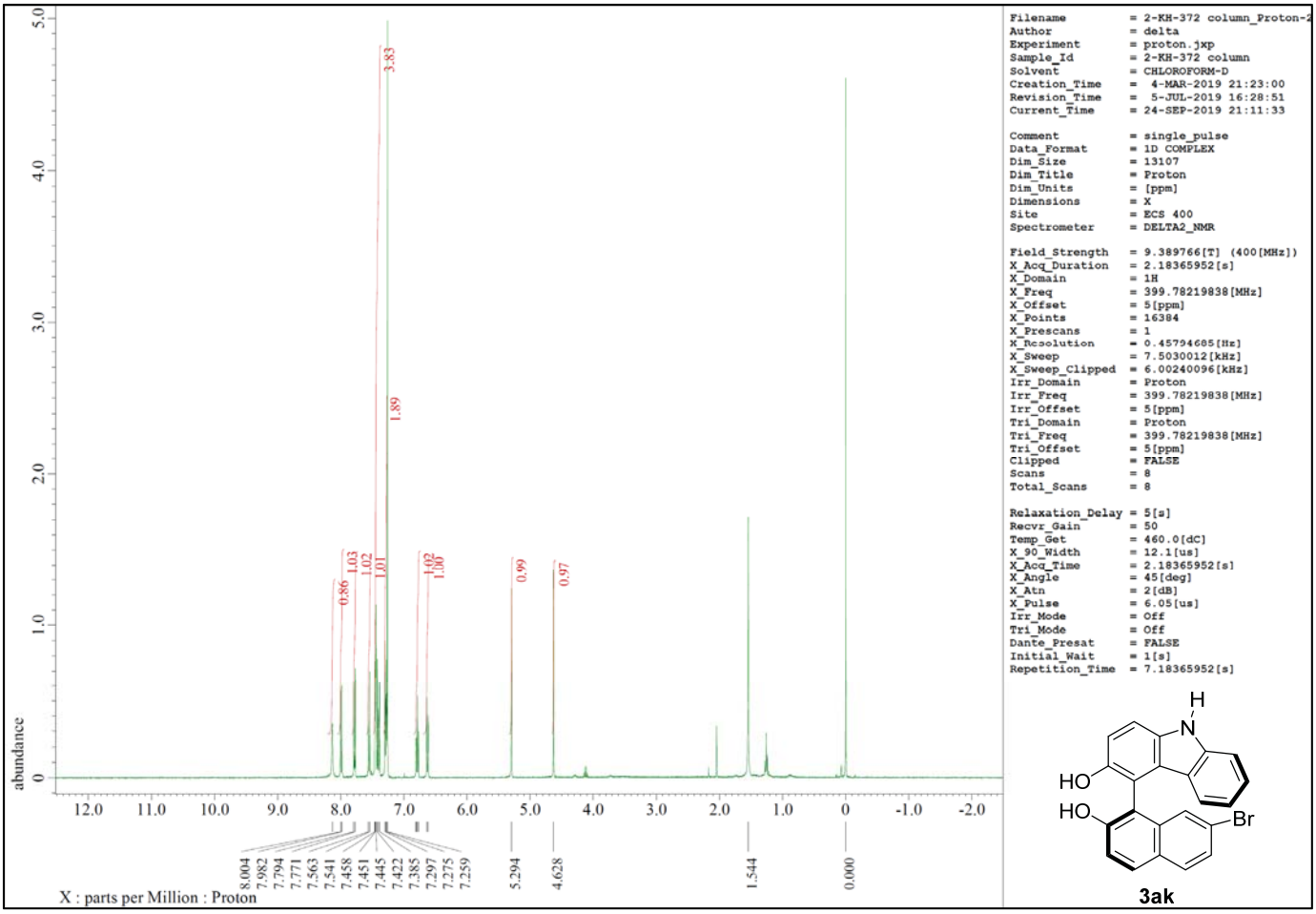
Compound **3ai** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)


abundance
X : parts per Million : Proton
Filename = 2-KH-442 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-LH-442 column
Solvent = CHLOROFORM-D
Creation_Time = 29-JUL-2019 15:55:57
Revision_Time = 30-JUL-2019 21:45:21
Current_Time = 24-SEP-2019 21:07:50
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
HO
HO
N
H
O
3aj

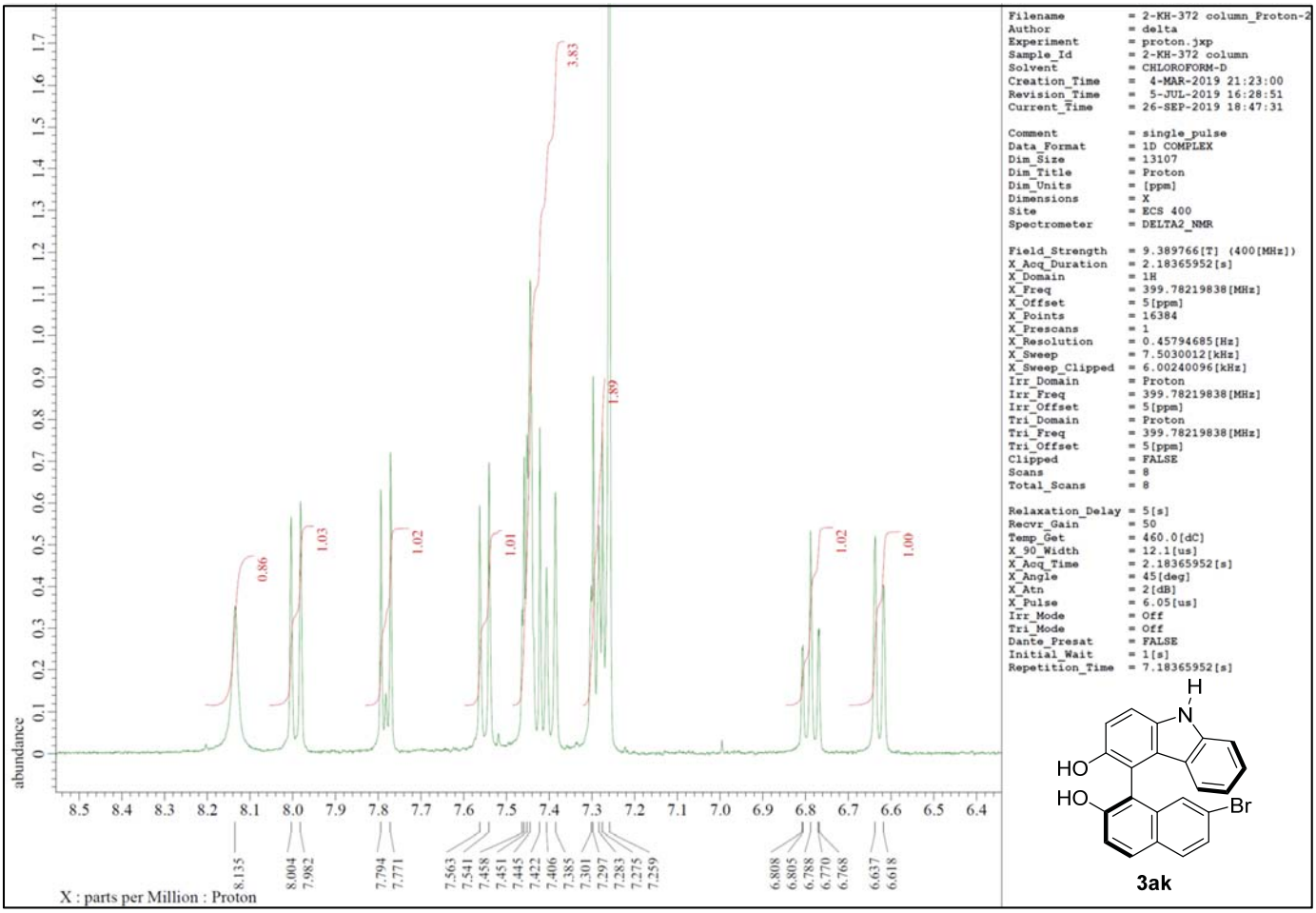

abundance
X : parts per Million : Proton
Filename = 2-KH-442 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-LH-442 column
Solvent = CHLOROFORM-D
Creation_Time = 29-JUL-2019 15:55:57
Revision_Time = 30-JUL-2019 21:45:21
Current_Time = 26-SEP-2019 18:47:00
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 54
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
HO
HO
N
H
O
3aj



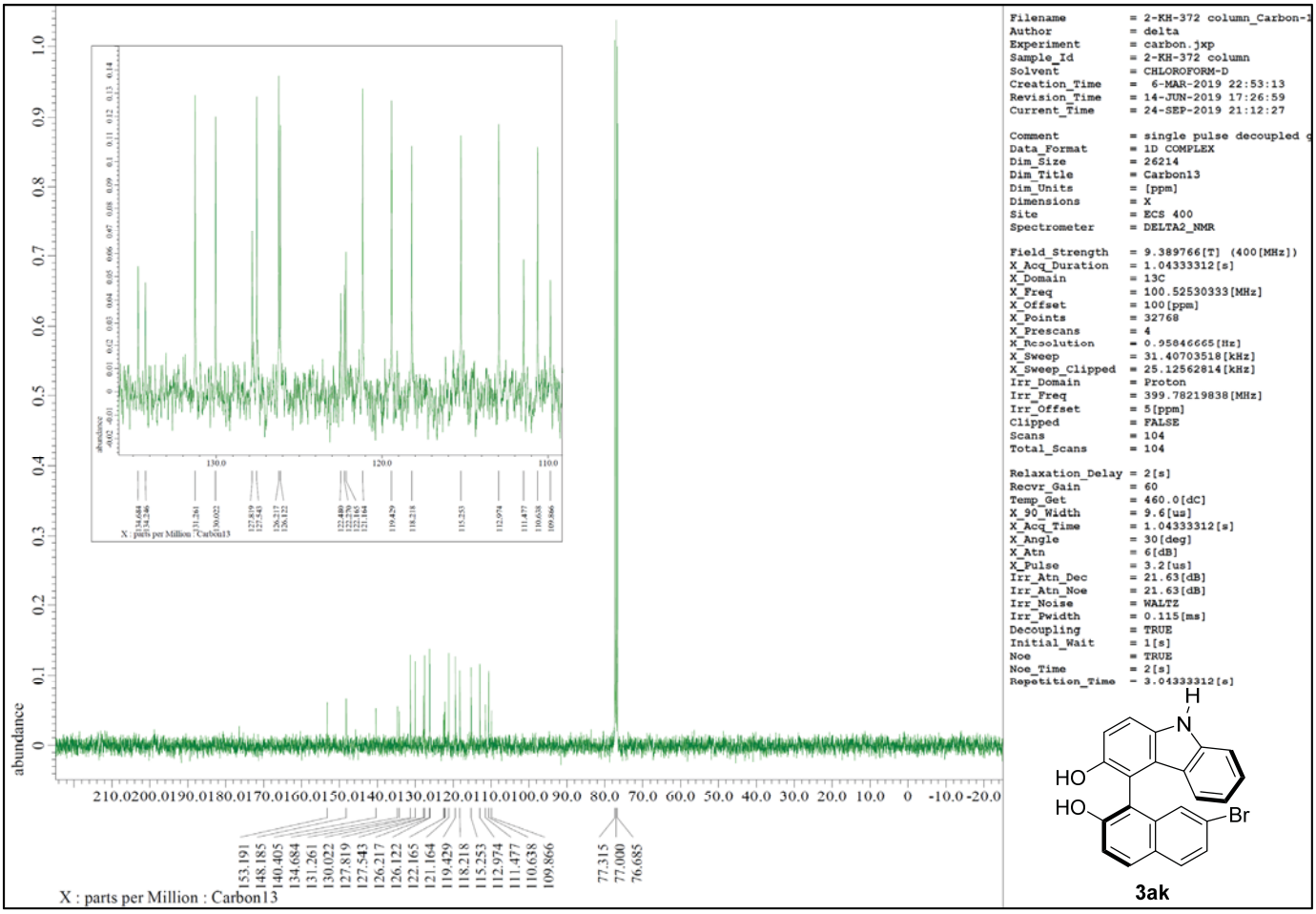
Compound **3aj** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)


abundance
X : parts per Million : Proton
3.83
1.89
0.86
1.03
1.02
1.01
1.02
1.00
0.99
0.97
8.004
7.982
7.794
7.771
7.563
7.541
7.458
7.451
7.445
7.422
7.385
7.297
7.275
7.259
5.294
4.628
1.544
0.000
Filename = 2-KH-372 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-372 column
Solvent = CHLOROFORM-D
Creation_Time = 4-MAR-2019 21:23:00
Revision_Time = 5-JUL-2019 16:28:51
Current_Time = 24-SEP-2019 21:11:33
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
Br

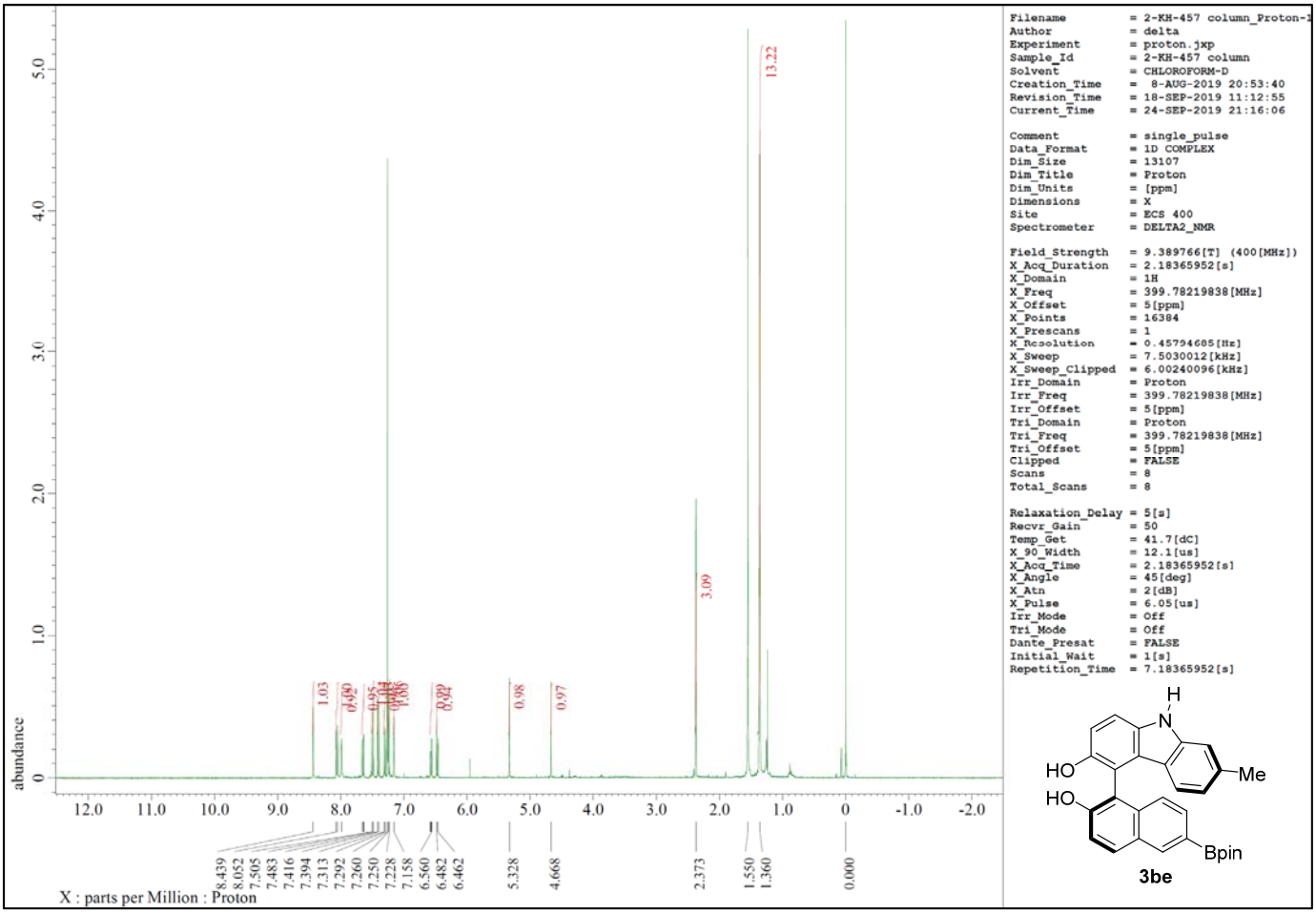
3ak

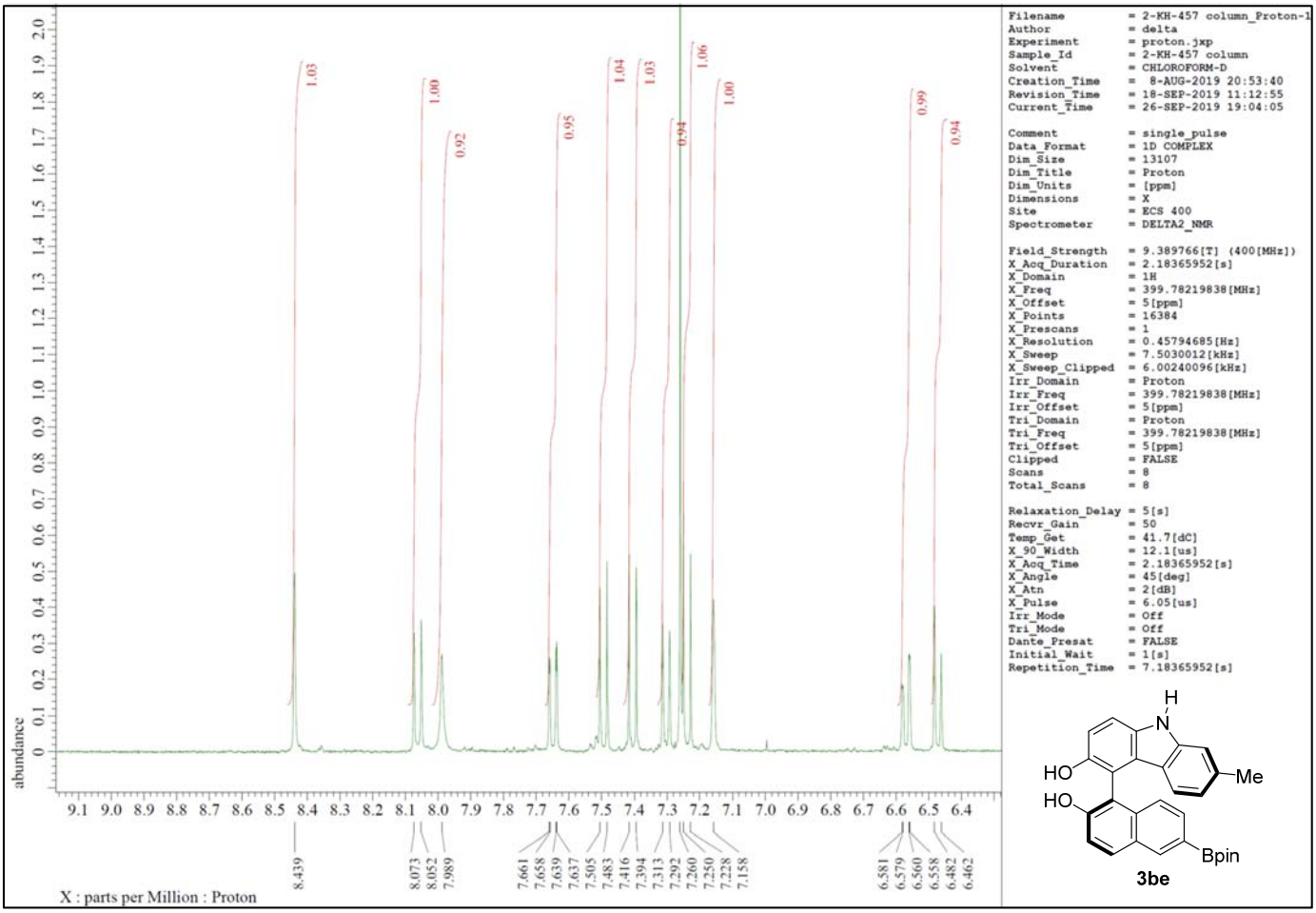

abundance
X : parts per Million : Proton
3.83
1.89
0.86
1.03
1.02
1.01
1.02
1.00
8.135
8.004
7.982
7.794
7.771
7.563
7.541
7.458
7.451
7.445
7.422
7.406
7.385
7.301
7.297
7.283
7.275
7.259
6.808
6.805
6.788
6.770
6.768
6.637
6.618
Filename = 2-KH-372 column_Proton-2
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-372 column
Solvent = CHLOROFORM-D
Creation_Time = 4-MAR-2019 21:23:00
Revision_Time = 5-JUL-2019 16:28:51
Current_Time = 26-SEP-2019 18:47:31
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 460.0[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
Br

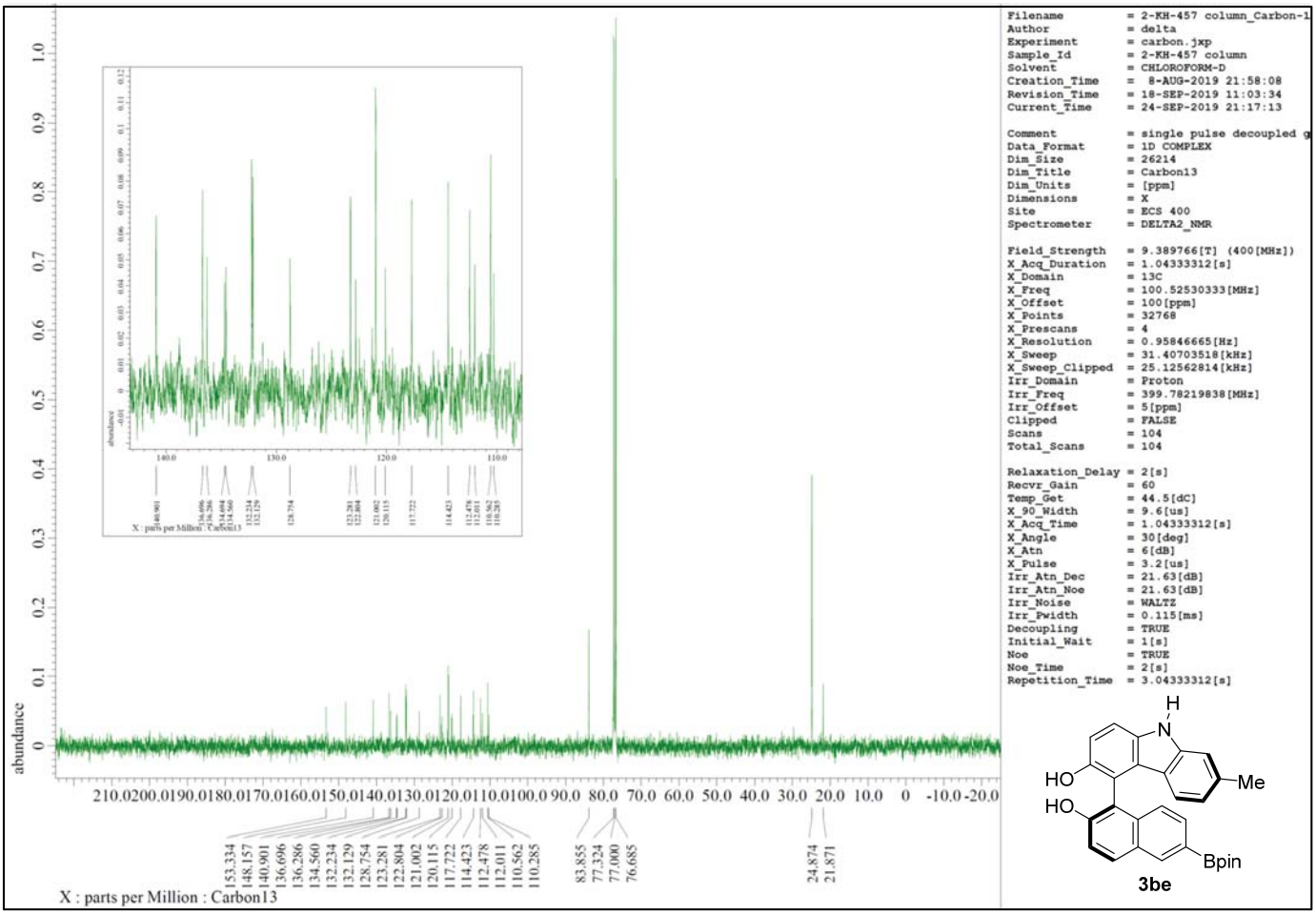
3ak



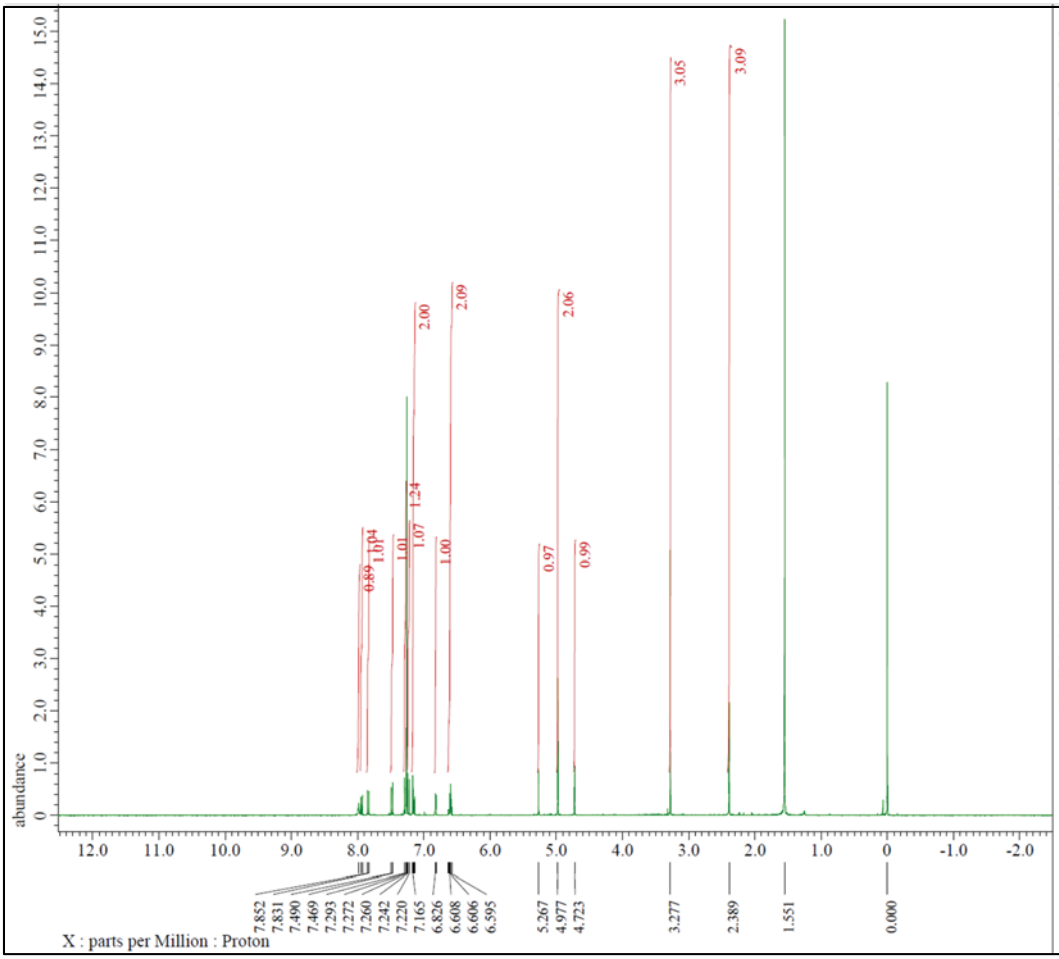
Compound **3ak** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)


abundance
X : parts per Million : Proton
1.03
0.92
0.95
0.98
0.97
3.09
13.22
Filename = 2-KH-457 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-457 column
Solvent = CHLOROFORM-D
Creation_Time = 8-AUG-2019 20:53:40
Revision_Time = 18-SEP-2019 11:12:55
Current_Time = 24-SEP-2019 21:16:06
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 41.7[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
Me
Bpin
3be


abundance
X : parts per Million : Proton
1.03
1.00
0.92
0.95
1.04
1.03
1.06
0.94
1.00
0.99
0.94
Filename = 2-KH-457 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-457 column
Solvent = CHLOROFORM-D
Creation_Time = 8-AUG-2019 20:53:40
Revision_Time = 18-SEP-2019 11:12:55
Current_Time = 26-SEP-2019 19:04:05
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 41.7[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
HO
Me
Bpin
3be



Compound **3be** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

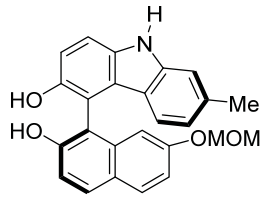



```
Filename          = 2-KH-456 column_Proton-2
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-456 column
Solvent           = CHLOROFORM-D
Creation_Time     = 20-AUG-2019 21:43:29
Revision_Time     = 18-SEP-2019 11:38:47
Current_Time      = 24-SEP-2019 21:51:31

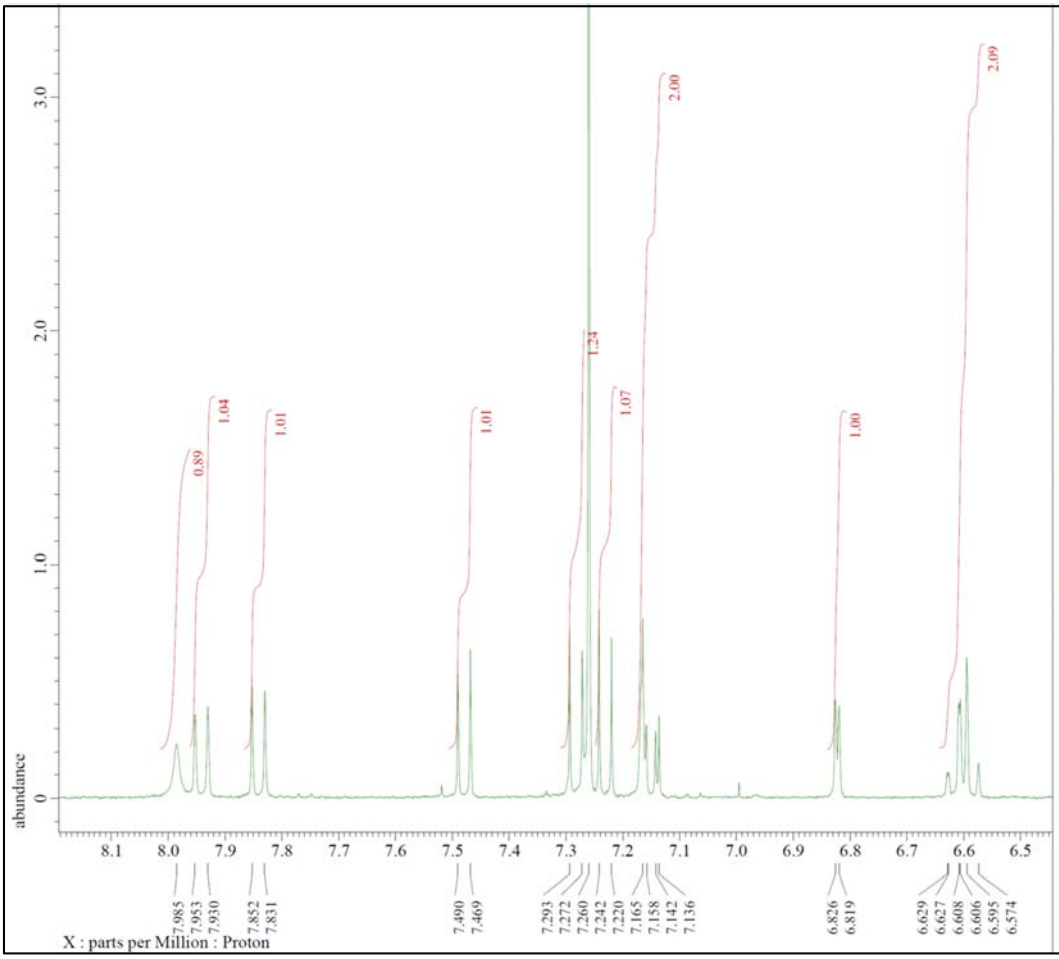
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 41.6[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



3bi

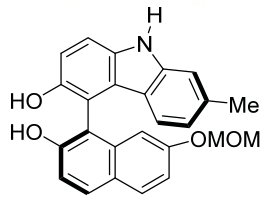



```
Filename          = 2-KH-456 column_Proton-2
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-456 column
Solvent           = CHLOROFORM-D
Creation_Time     = 20-AUG-2019 21:43:29
Revision_Time     = 18-SEP-2019 11:38:47
Current_Time      = 26-SEP-2019 19:04:50

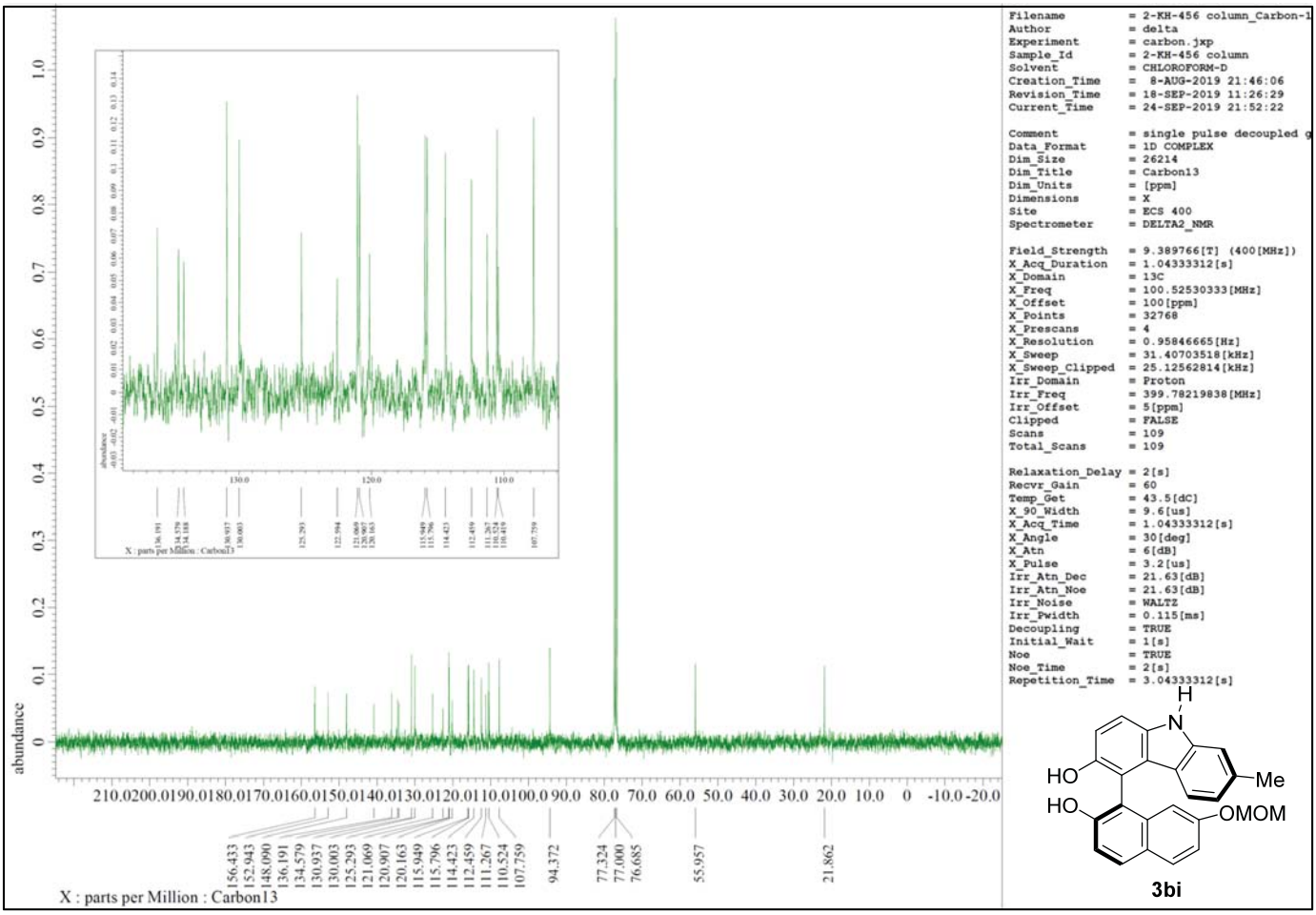
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

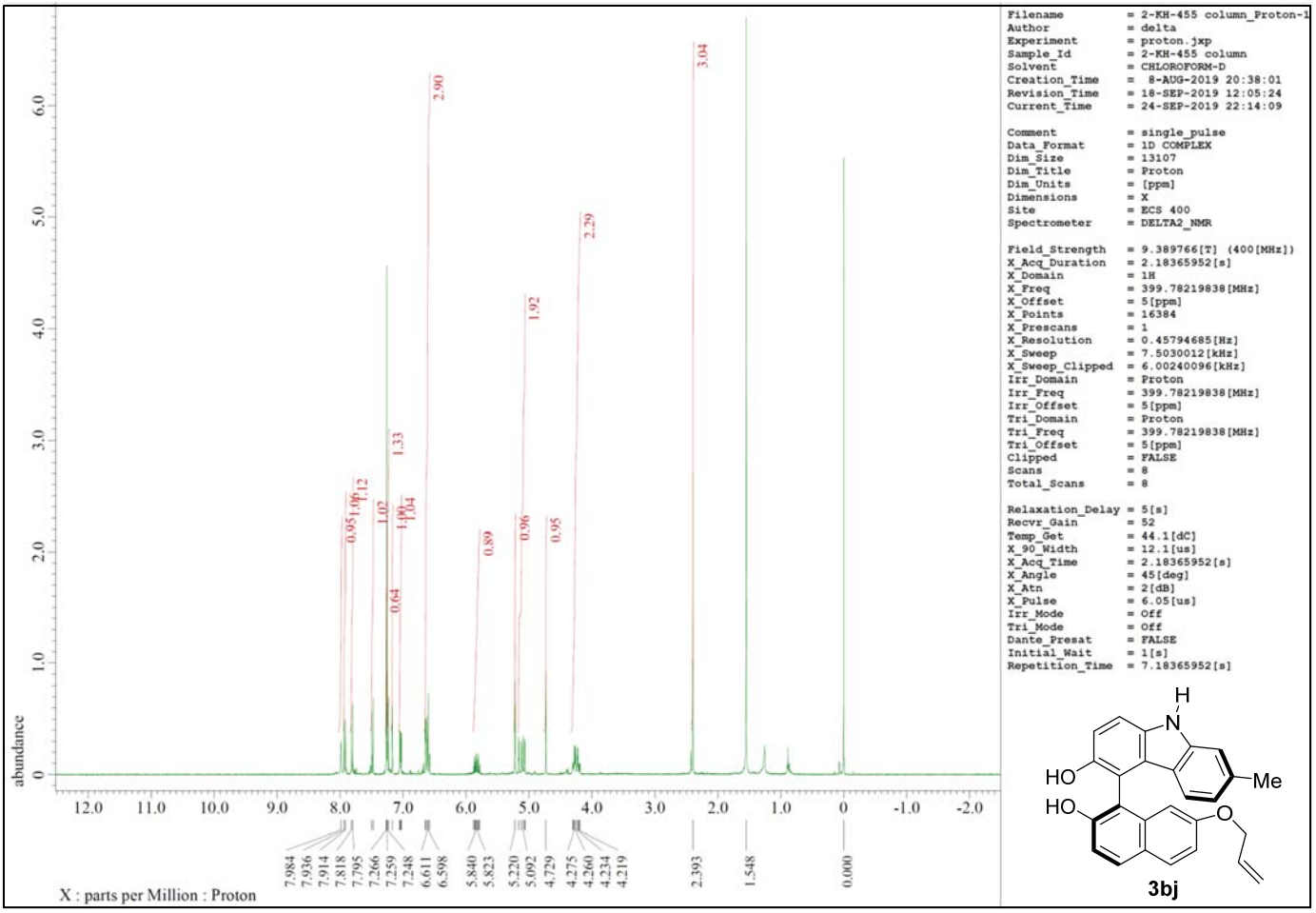
Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 41.6[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



3bi



Compound **3bi** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)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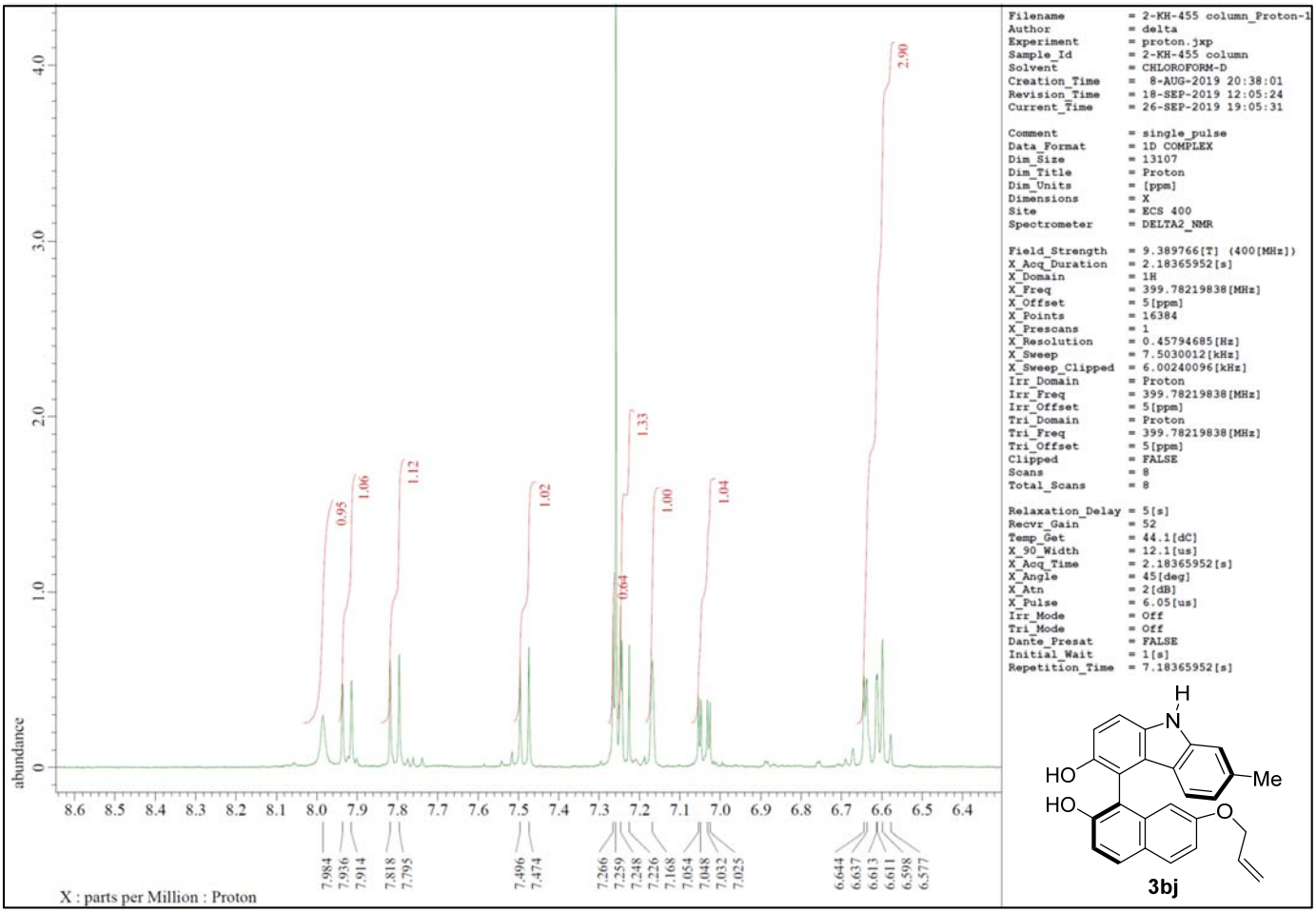
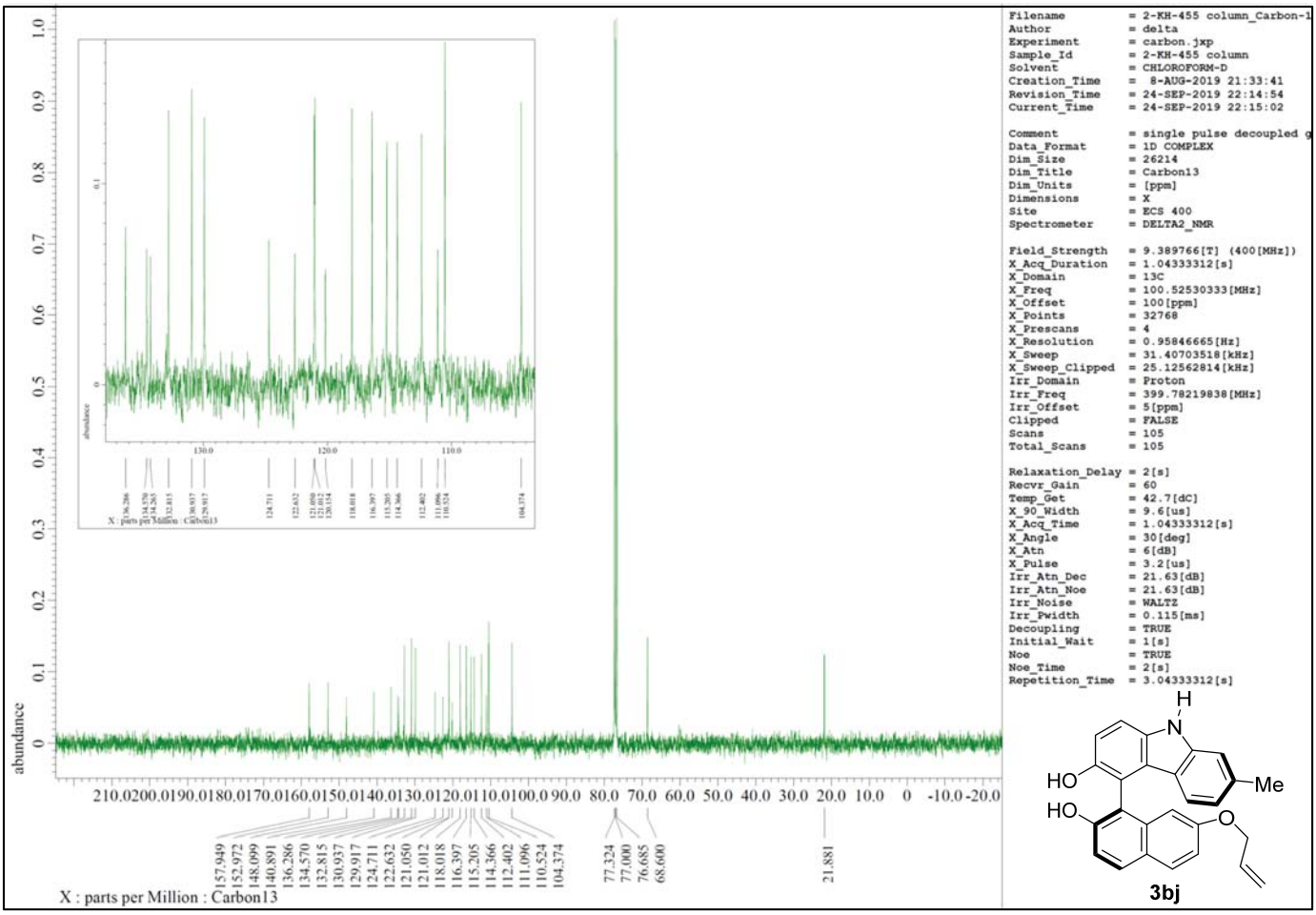
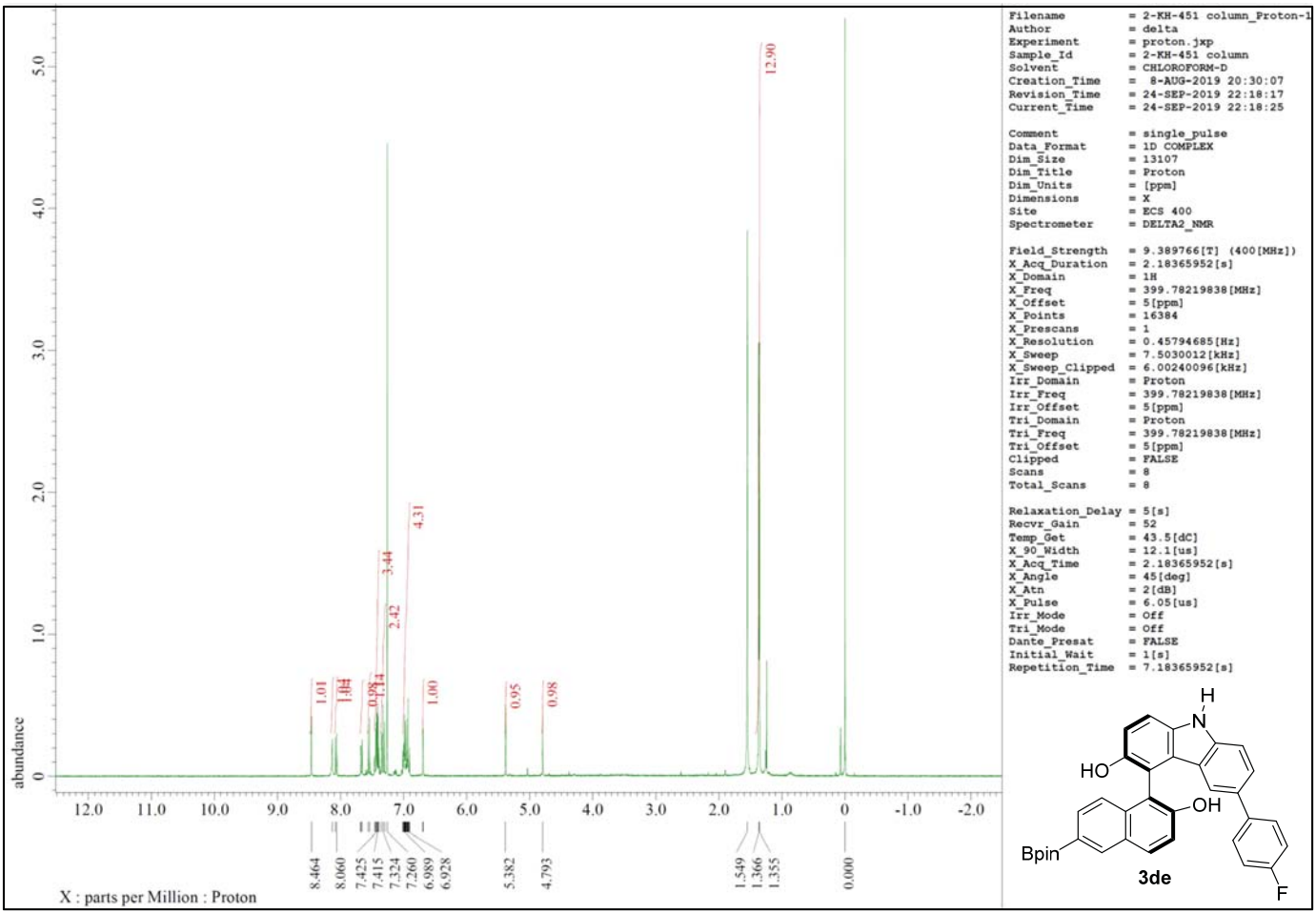
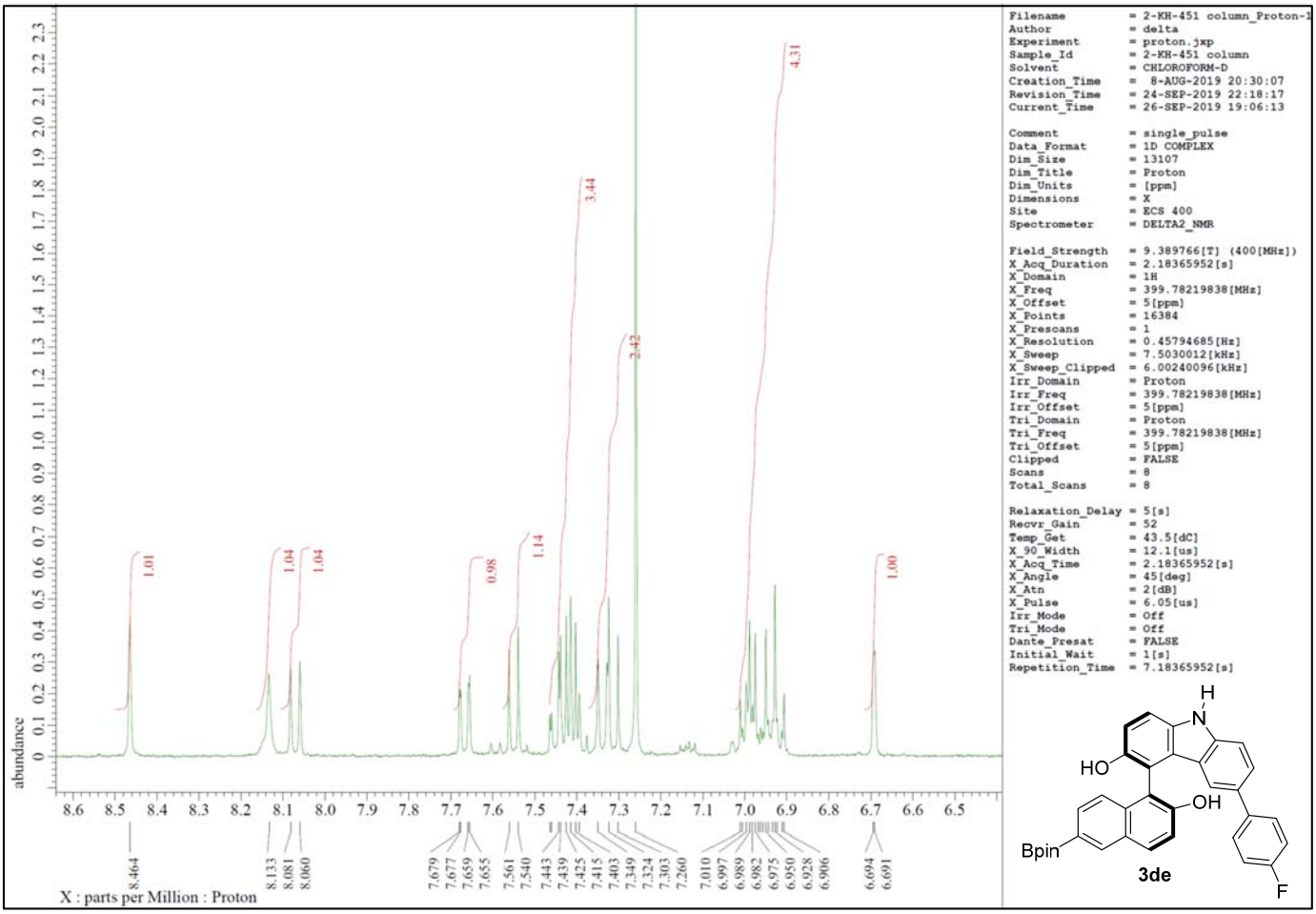
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-455 column
Solvent = CHLOROFORM-D
Creation_Time = 8-AUG-2019 20:38:01
Revision_Time = 18-SEP-2019 12:05:24
Current_Time = 24-SEP-2019 22:14:09
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 52
Temp_Get = 44.1[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
HO
HO
N
H
Me
O
3bj

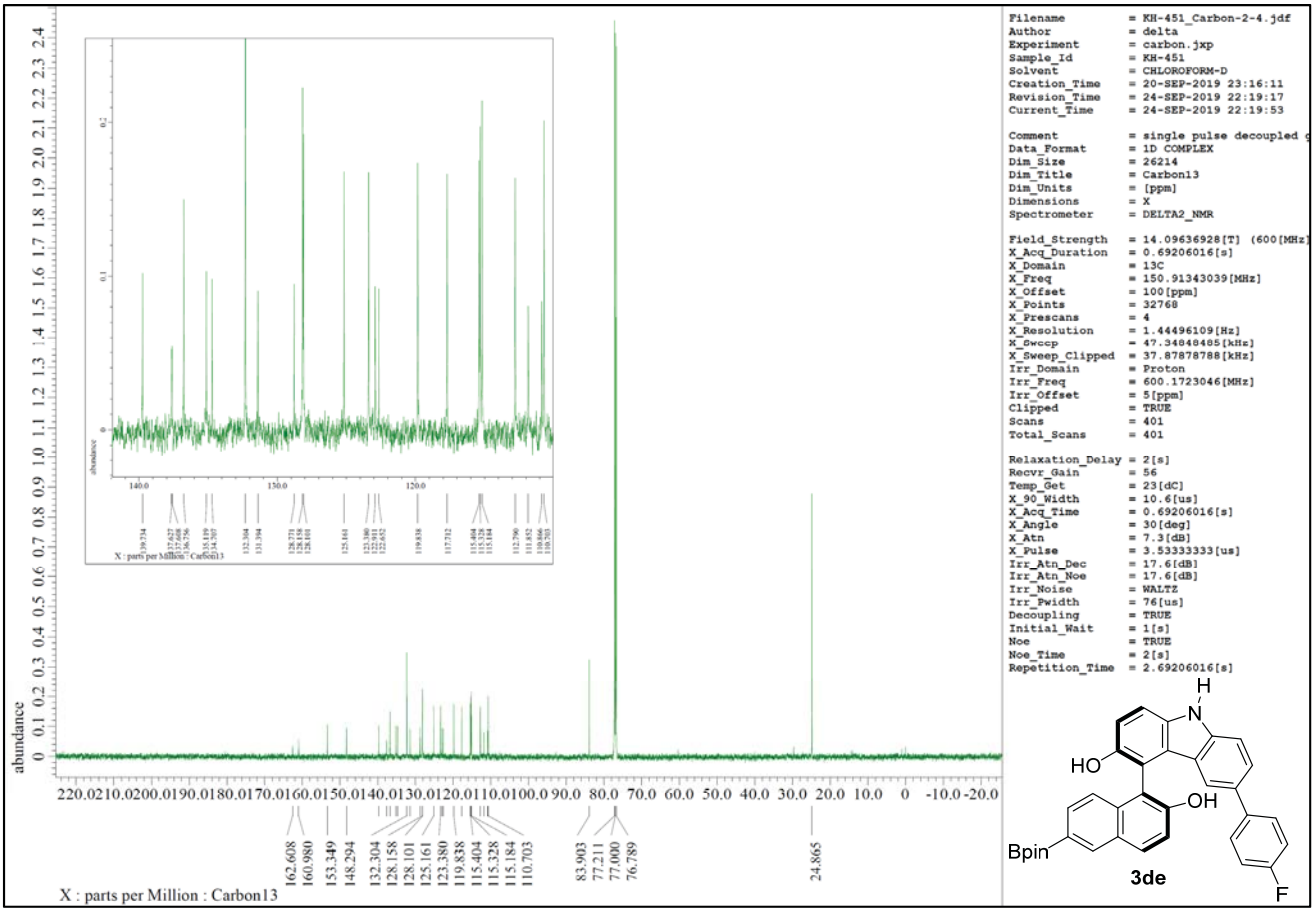

Filename = 2-KH-455 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-455 column
Solvent = CHLOROFORM-D
Creation_Time = 8-AUG-2019 20:38:01
Revision_Time = 18-SEP-2019 12:05:24
Current_Time = 26-SEP-2019 19:05:31
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 52
Temp_Get = 44.1[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
HO
HO
N
H
Me
O
3bj

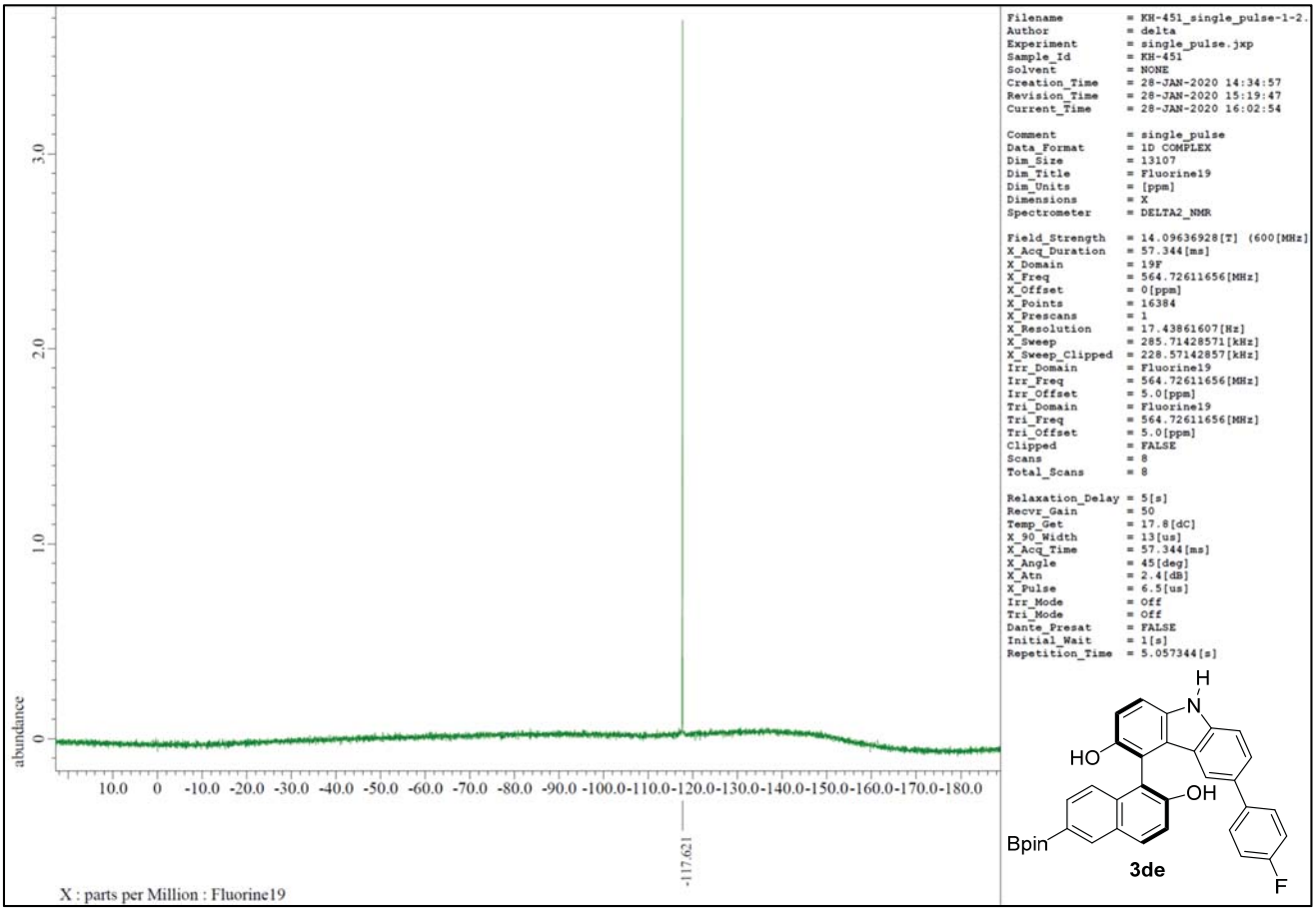


Compound **3bj** (^{1}H NMR, 400 MHz, $CDCl_3$ and ^{13}C NMR, 100 MHz, $CDCl_3$)

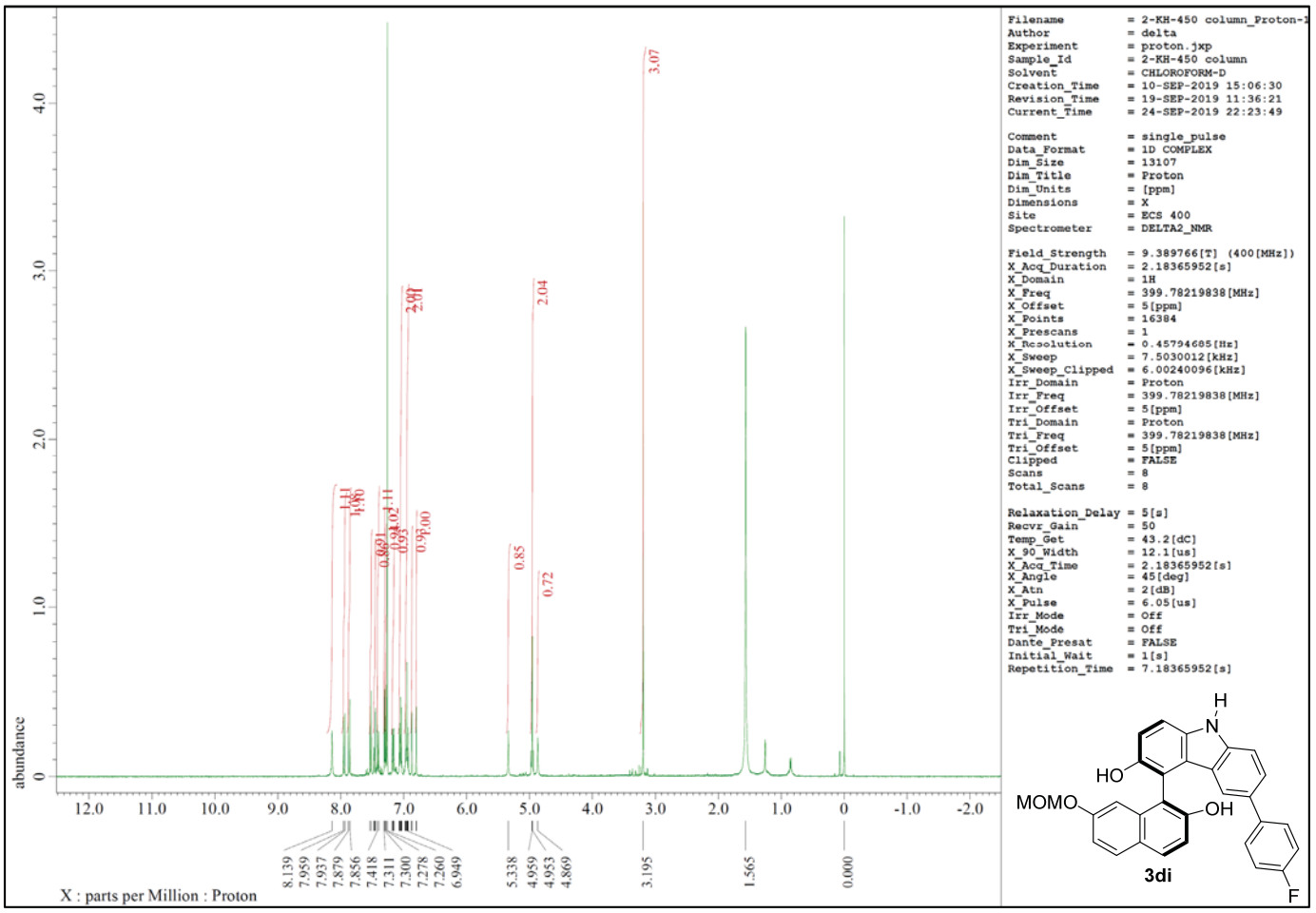

abundance
X : parts per Million : Proton
12.90
4.31
3.44
2.42
1.01
1.04
1.04
0.98
1.14
1.00
0.95
0.98
8.464
8.060
7.425
7.415
7.324
7.260
6.989
6.928
5.382
4.793
1.549
1.366
1.355
0.000
Filename = 2-KH-451 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-451 column
Solvent = CHLOROFORM-D
Creation_Time = 8-AUG-2019 20:30:07
Revision_Time = 24-SEP-2019 22:18:17
Current_Time = 24-SEP-2019 22:18:25
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 52
Temp_Get = 43.5[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
OH
Bpin
F
3de


abundance
X : parts per Million : Proton
4.31
3.44
2.42
1.01
1.04
1.04
0.98
1.14
1.00
8.464
8.133
8.081
8.060
7.679
7.677
7.659
7.655
7.561
7.540
7.443
7.439
7.425
7.415
7.403
7.349
7.324
7.303
7.260
7.010
6.997
6.989
6.982
6.975
6.950
6.928
6.906
6.694
6.691
Filename = 2-KH-451 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-451 column
Solvent = CHLOROFORM-D
Creation_Time = 8-AUG-2019 20:30:07
Revision_Time = 24-SEP-2019 22:18:17
Current_Time = 26-SEP-2019 19:06:13
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 52
Temp_Get = 43.5[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
H
N
HO
OH
Bpin
F
3de





Compound **3de** (^{1}H NMR, 400 MHz, $CDCl_3$, ^{13}C NMR, 150 MHz, $CDCl_3$ and ^{19}F NMR, 565 MHz, $CDCl_3$)

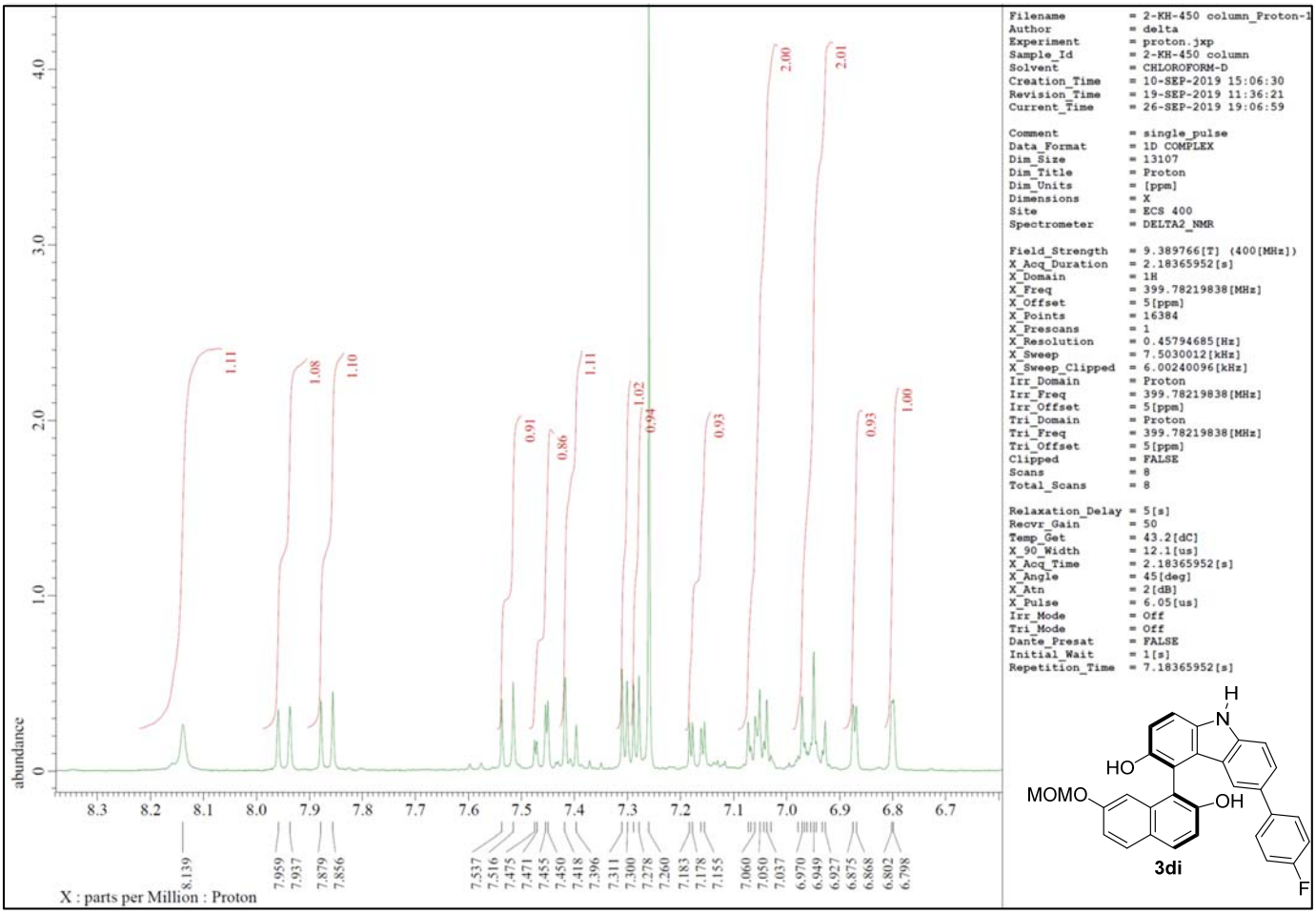



```
Filename          = 2-KH-450 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-450 column
Solvent           = CHLOROFORM-D
Creation_Time     = 10-SEP-2019 15:06:30
Revision_Time     = 19-SEP-2019 11:36:21
Current_Time      = 24-SEP-2019 22:23:49

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794605[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 43.2[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```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Filename          = 2-KH-450 column_Proton-1
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 2-KH-450 column
Solvent           = CHLOROFORM-D
Creation_Time     = 10-SEP-2019 15:06:30
Revision_Time     = 19-SEP-2019 11:36:21
Current_Time      = 26-SEP-2019 19:06:59

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 43.2[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```
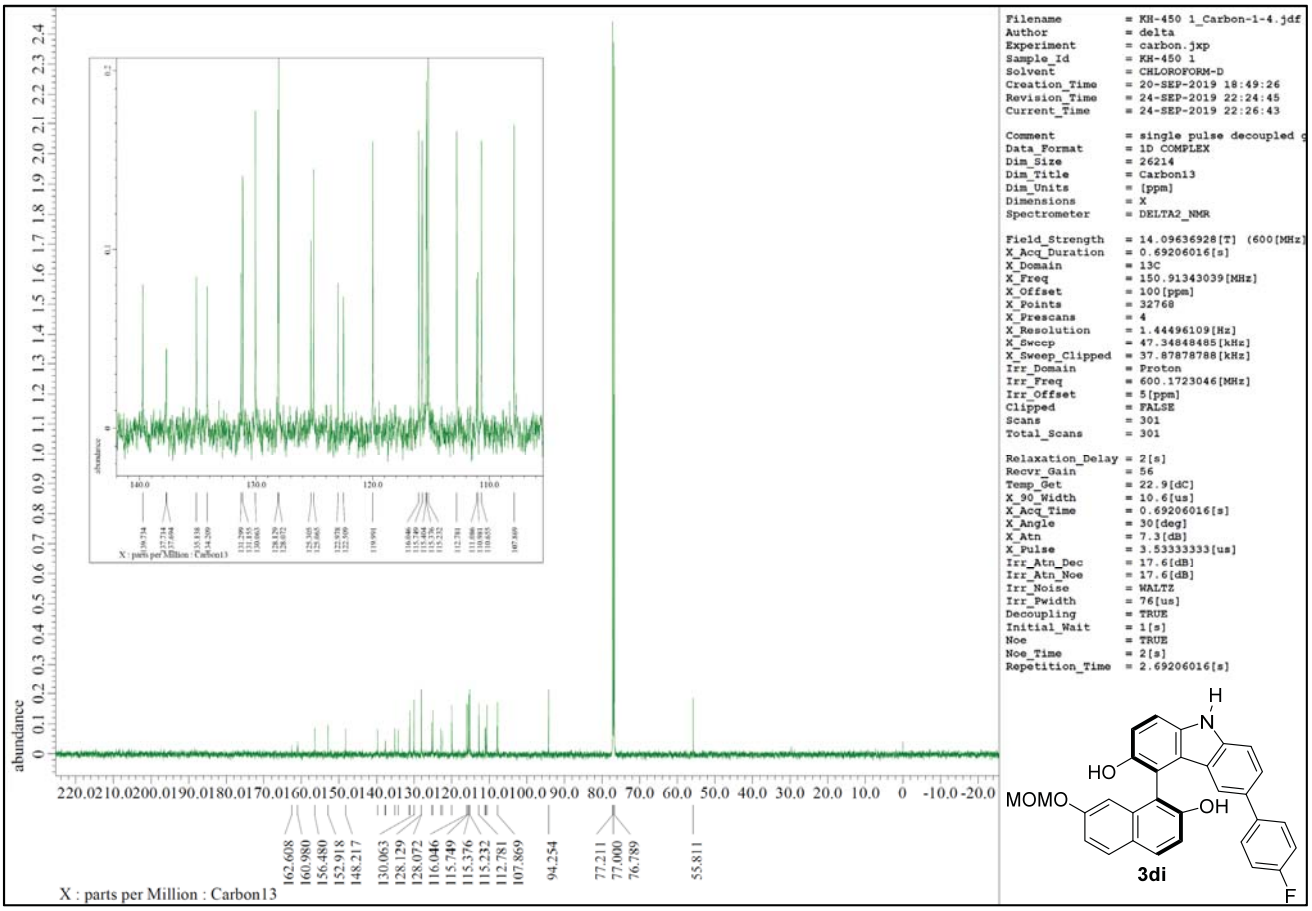
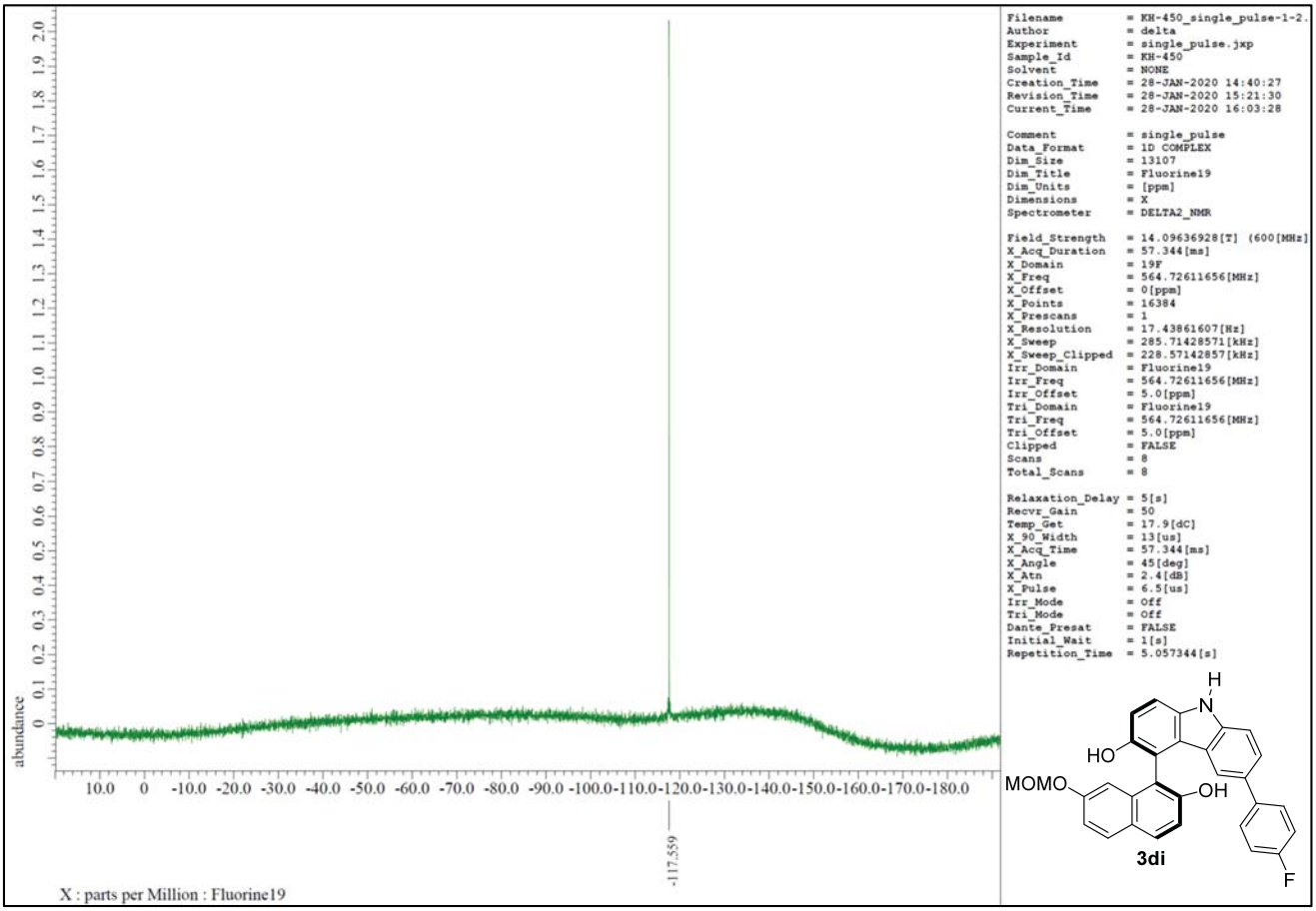
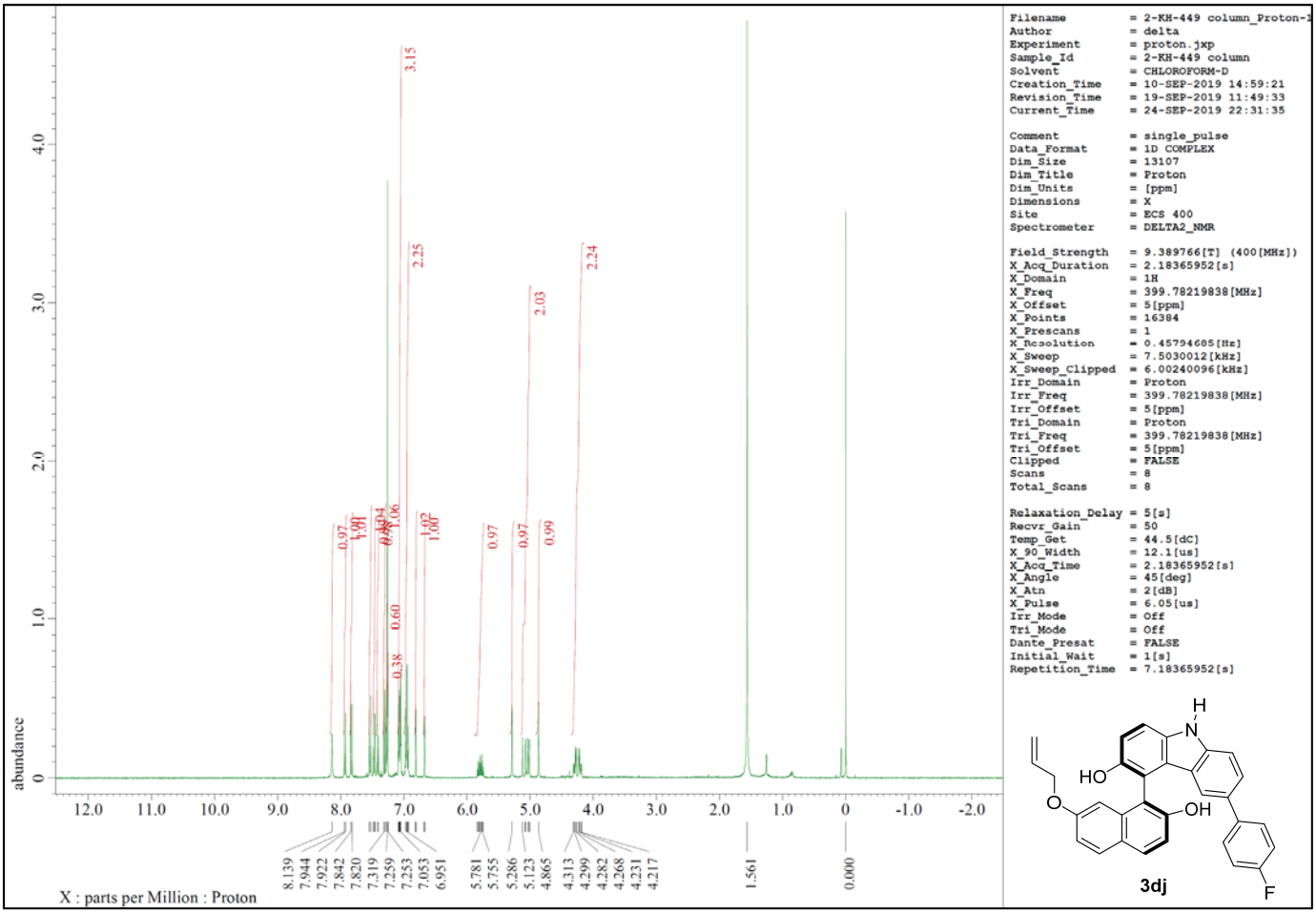
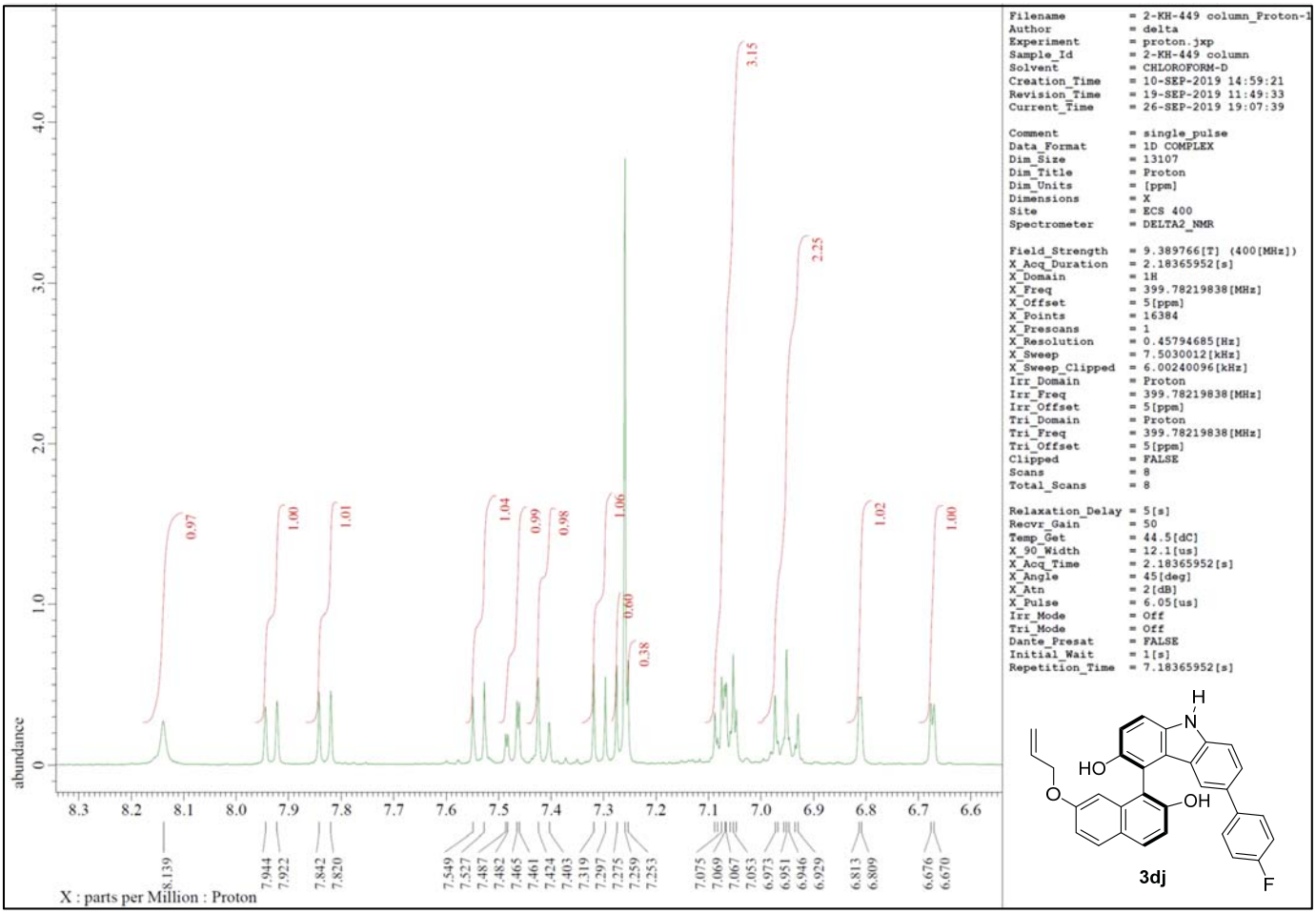





Compound **3di** (^{1}H NMR, 400 MHz, $CDCl_3$, ^{13}C NMR, 150 MHz, $CDCl_3$ and ^{19}F NMR, 565 MHz, $CDCl_3$)


Filename = 2-KH-449 column_Proton-1
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-449 column
Solvent = CHLOROFORM-D
Creation_Time = 10-SEP-2019 14:59:21
Revision_Time = 19-SEP-2019 11:49:33
Current_Time = 24-SEP-2019 22:31:35
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 44.5[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
3dj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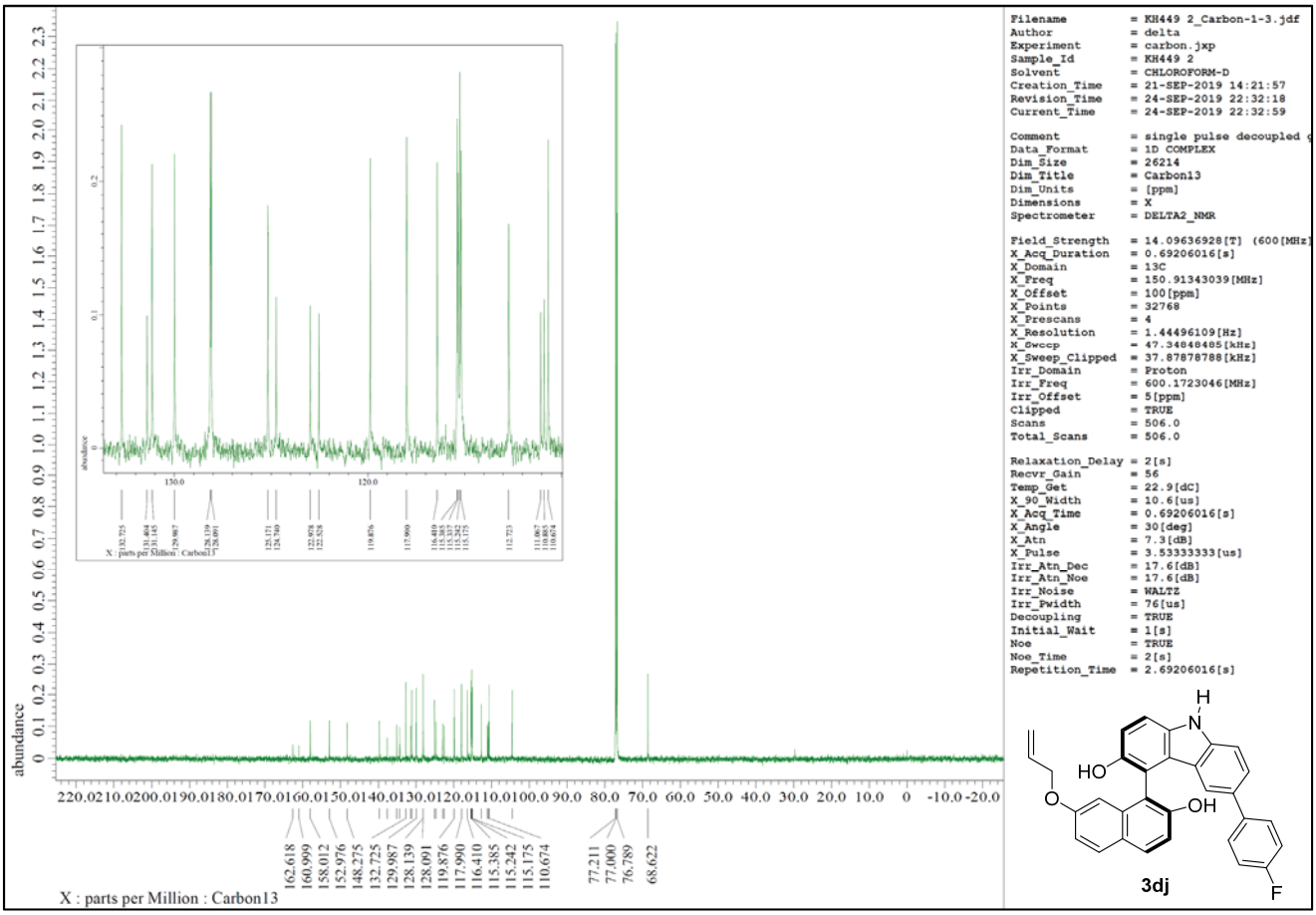
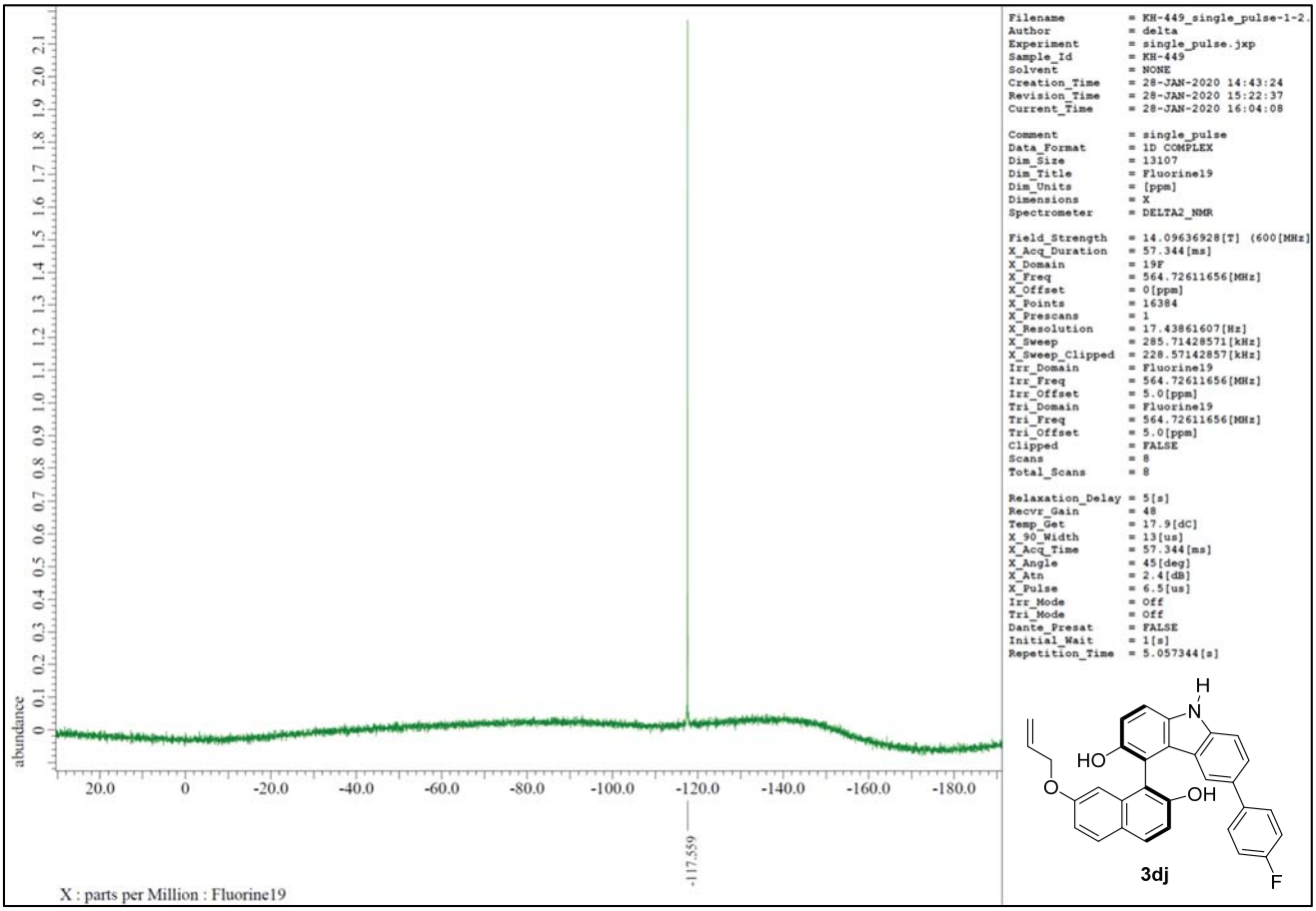
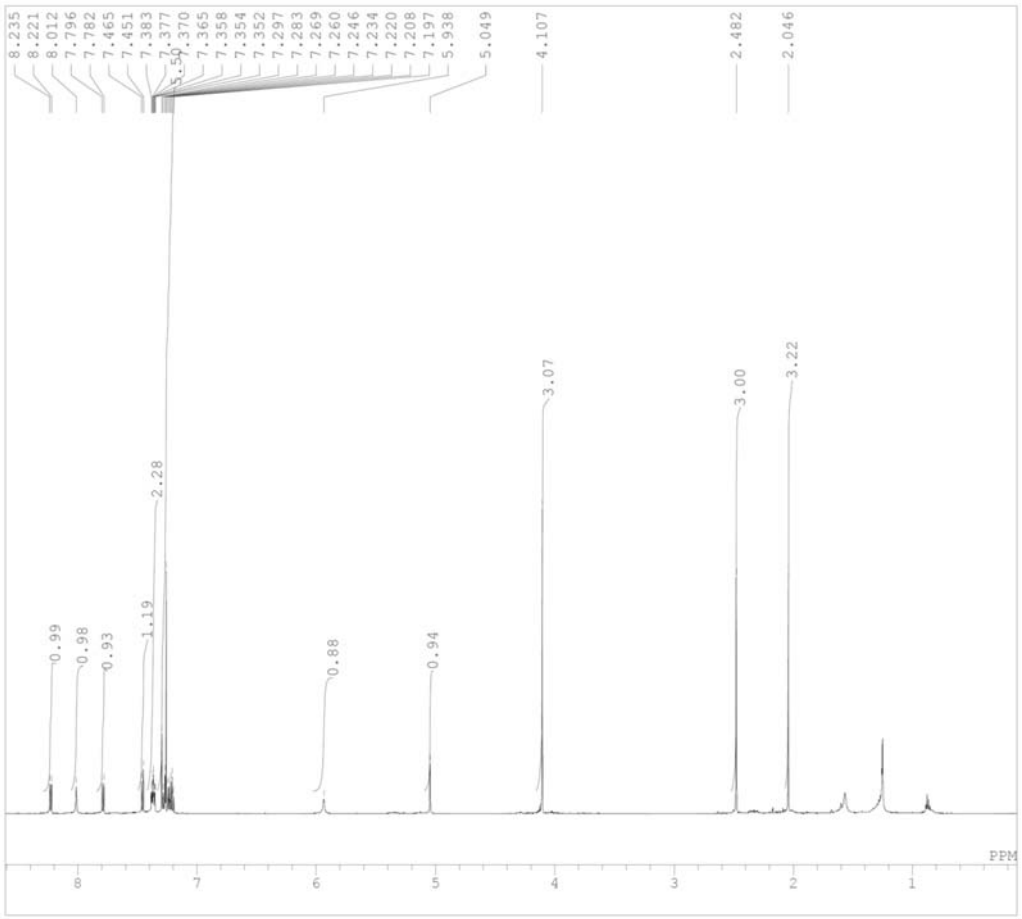
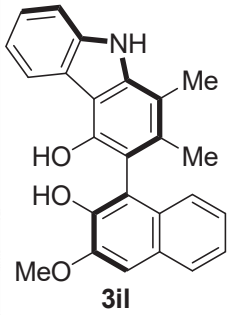
Author = delta
Experiment = proton.jxp
Sample_Id = 2-KH-449 column
Solvent = CHLOROFORM-D
Creation_Time = 10-SEP-2019 14:59:21
Revision_Time = 19-SEP-2019 11:49:33
Current_Time = 26-SEP-2019 19:07:39
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 50
Temp_Get = 44.5[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]
abundance
X : parts per Million : Proton
3dj



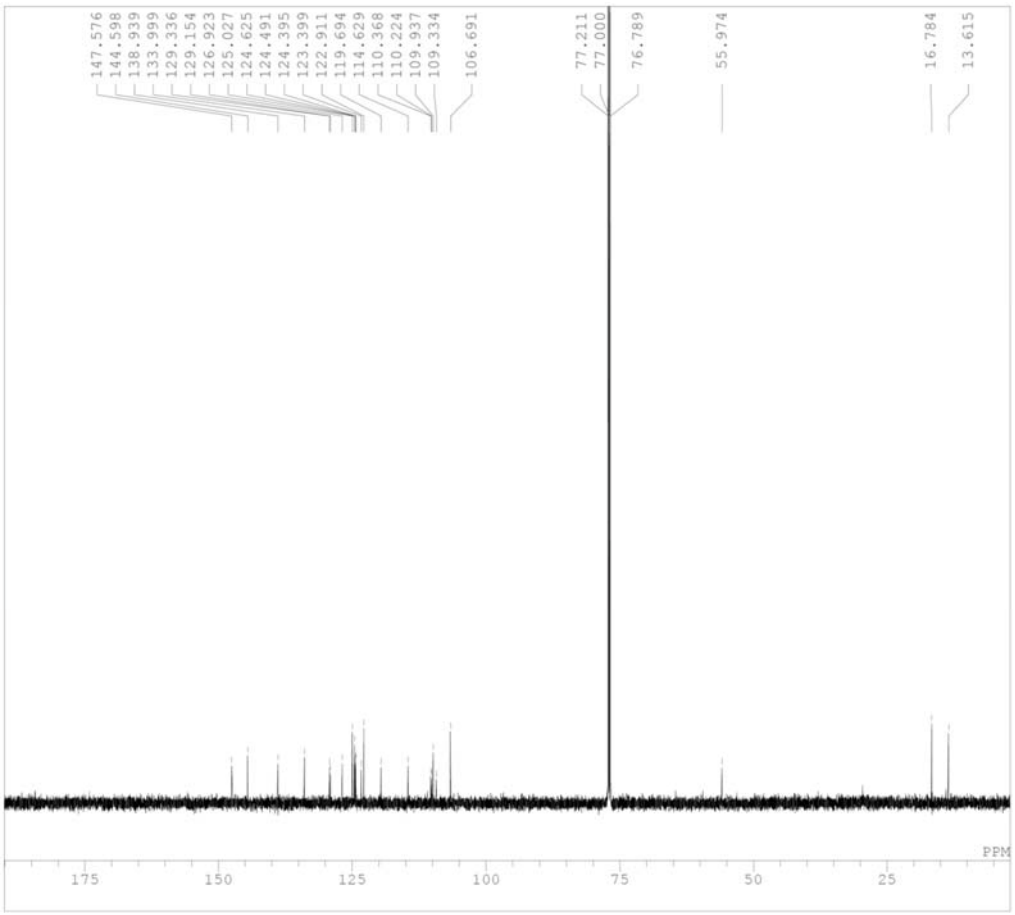


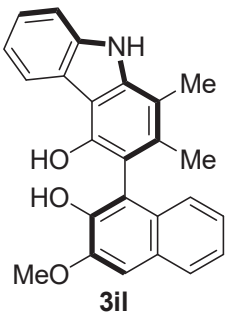
Compound **3dj** (^{1}H NMR, 400 MHz, $CDCl_3$, ^{13}C NMR, 150 MHz, $CDCl_3$ and ^{19}F NMR, 565 MHz, $CDCl_3$)





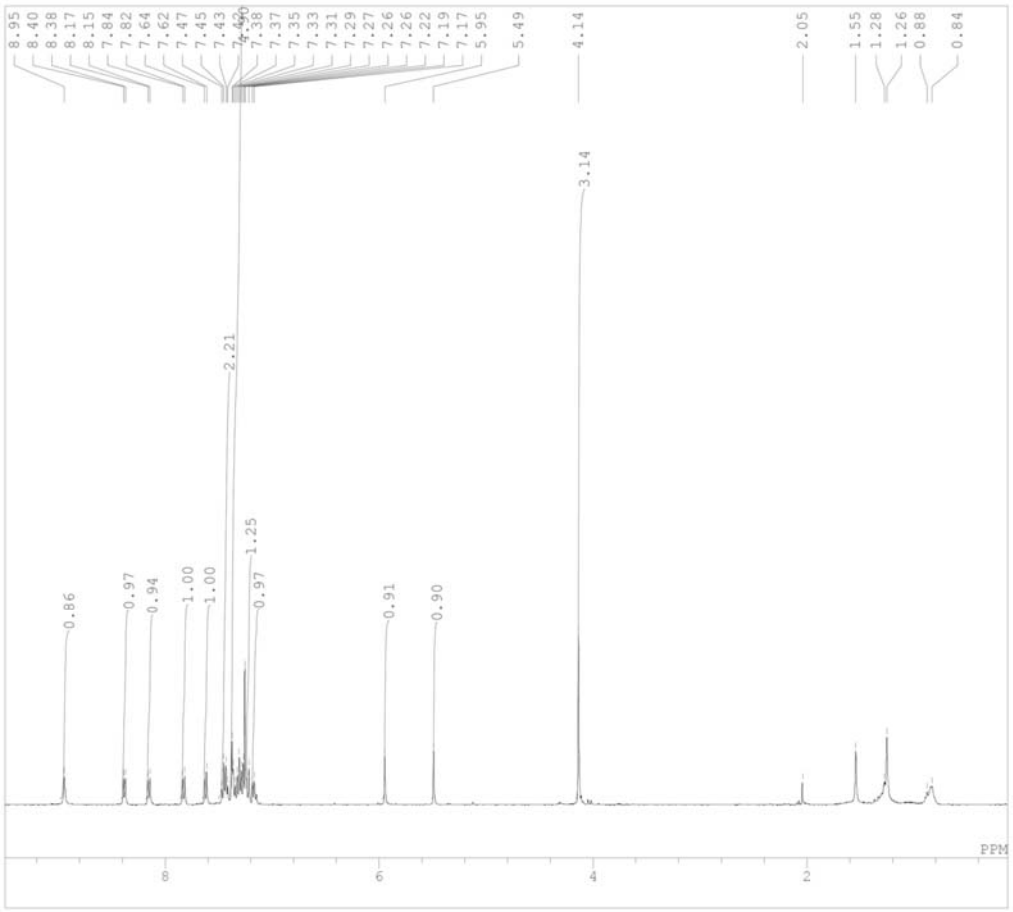
3il

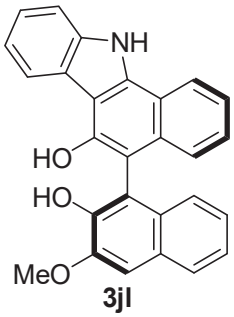




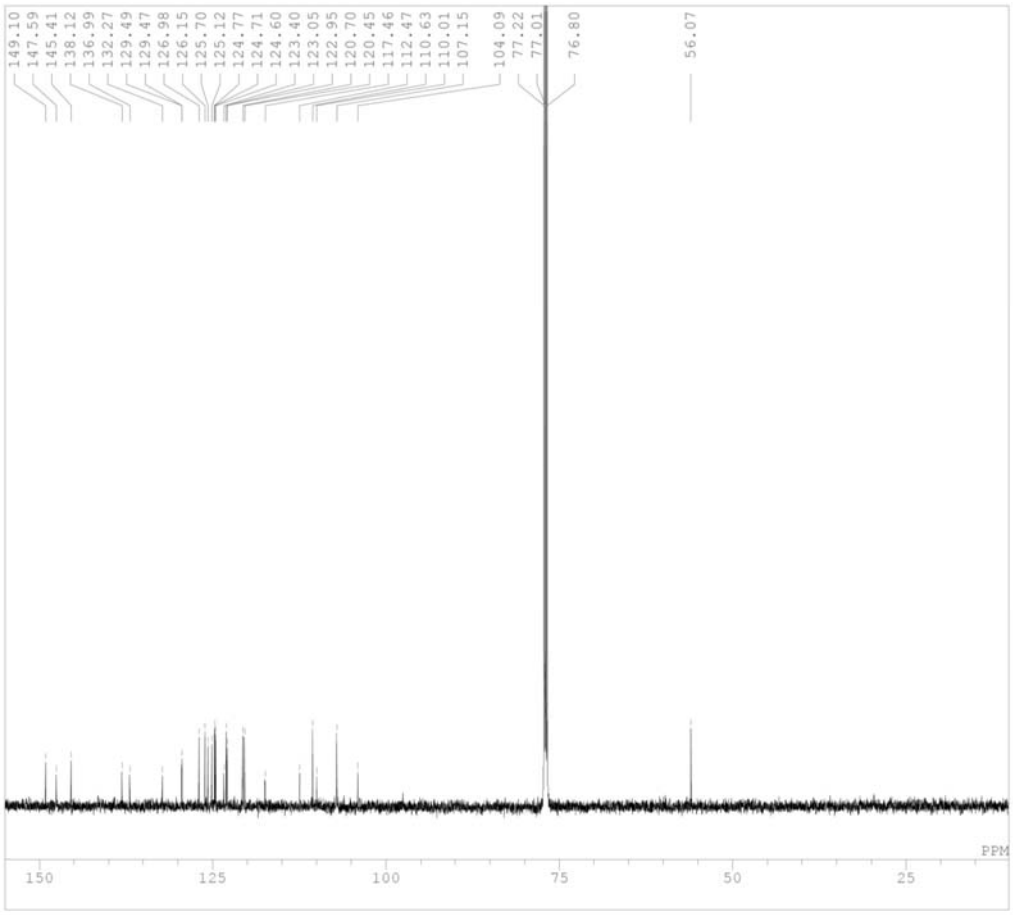
3il

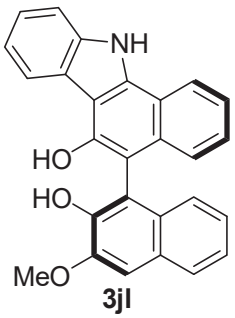
Compound **3il** (^{1}H NMR, 600 MHz, $CDCl_3$, ^{13}C NMR, 125 MHz, $CDCl_3$)





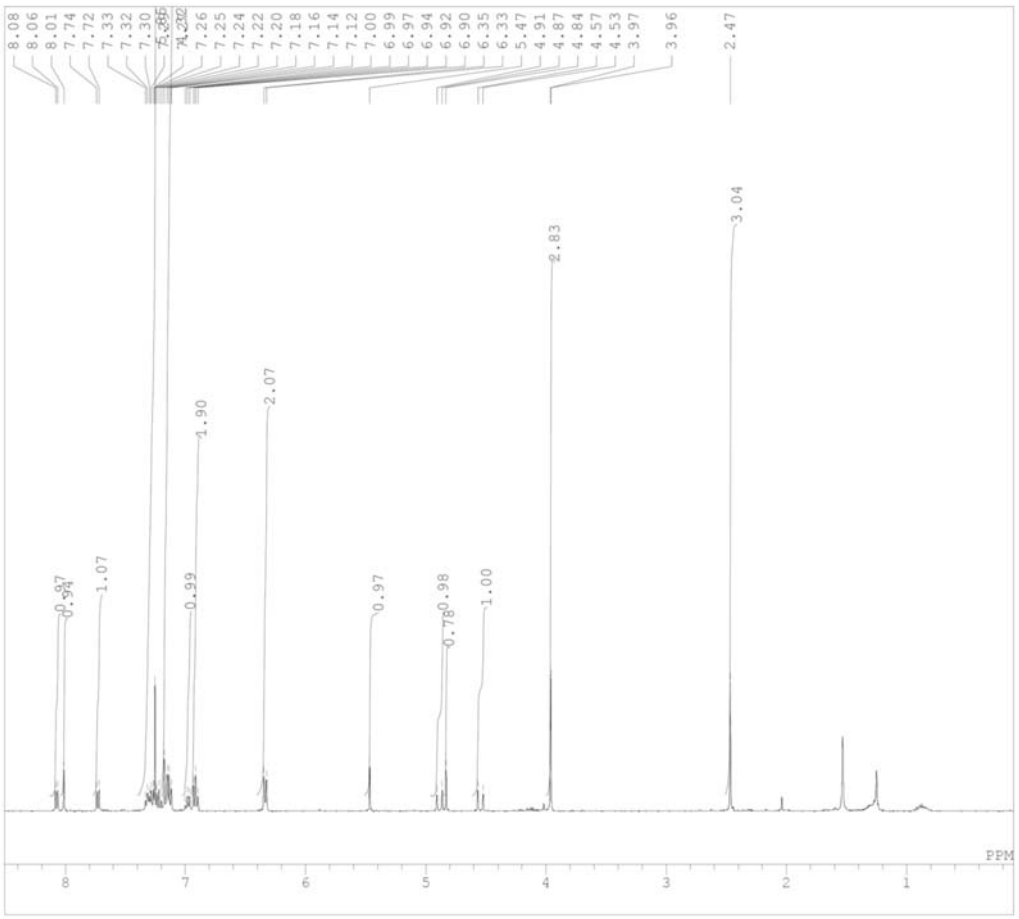
3jl

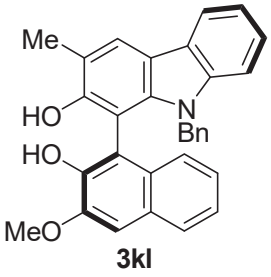




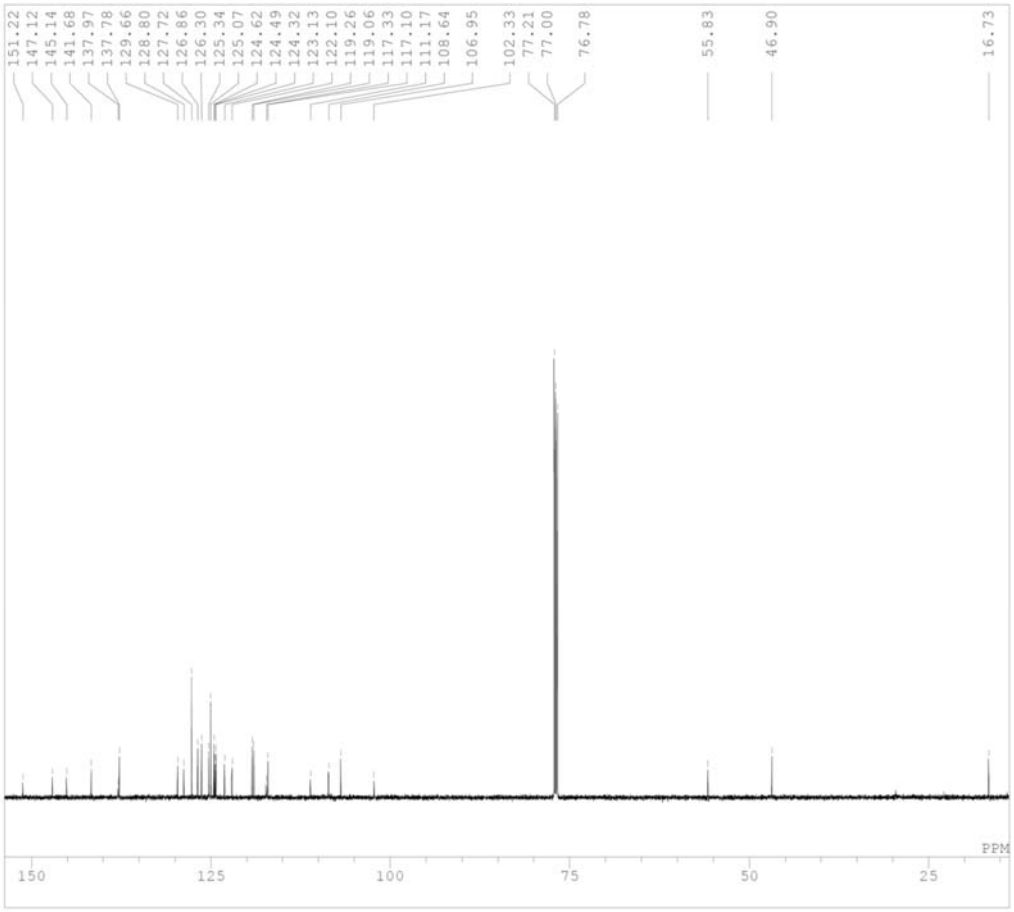
3jl

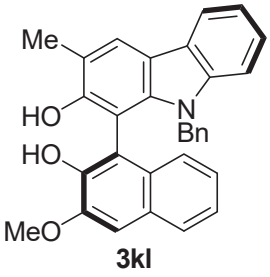
Compound **3jl** (^{1}H NMR, 400 MHz, $CDCl_3$, ^{13}C NMR, 125 MHz, $CDCl_3$)





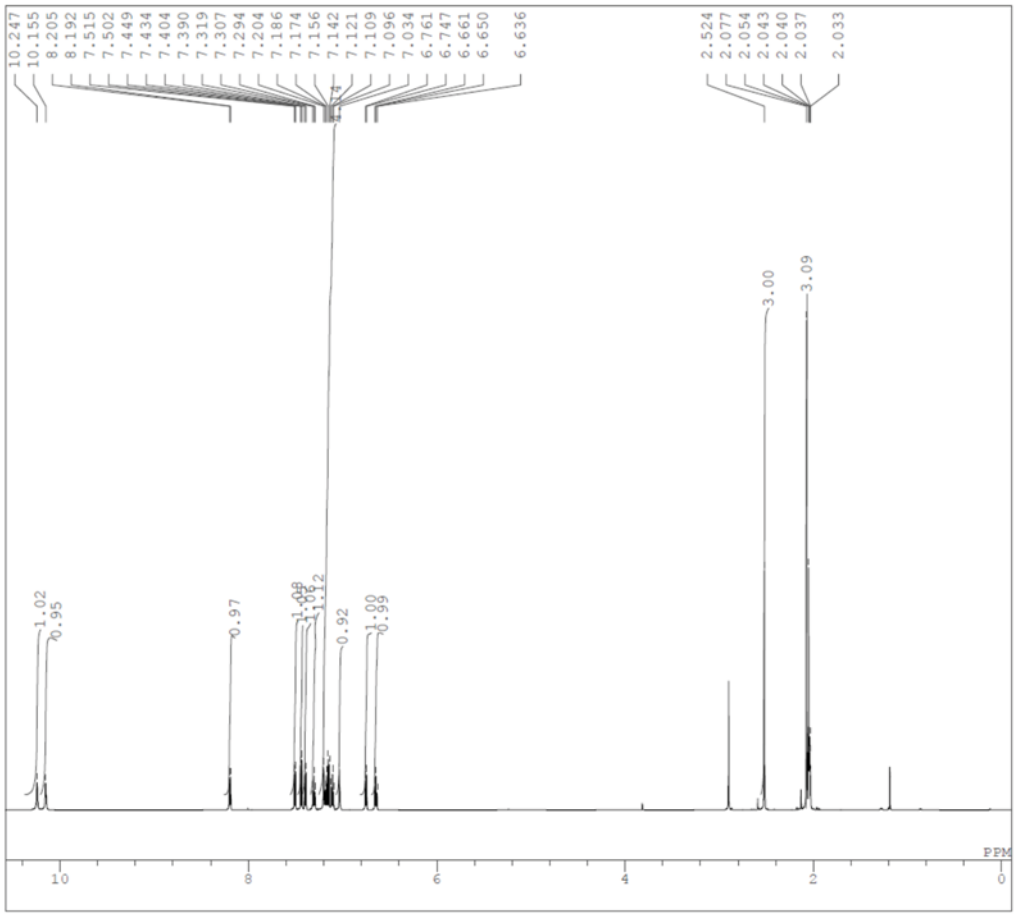
3kl

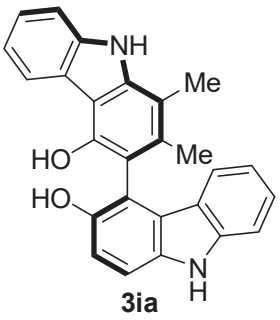




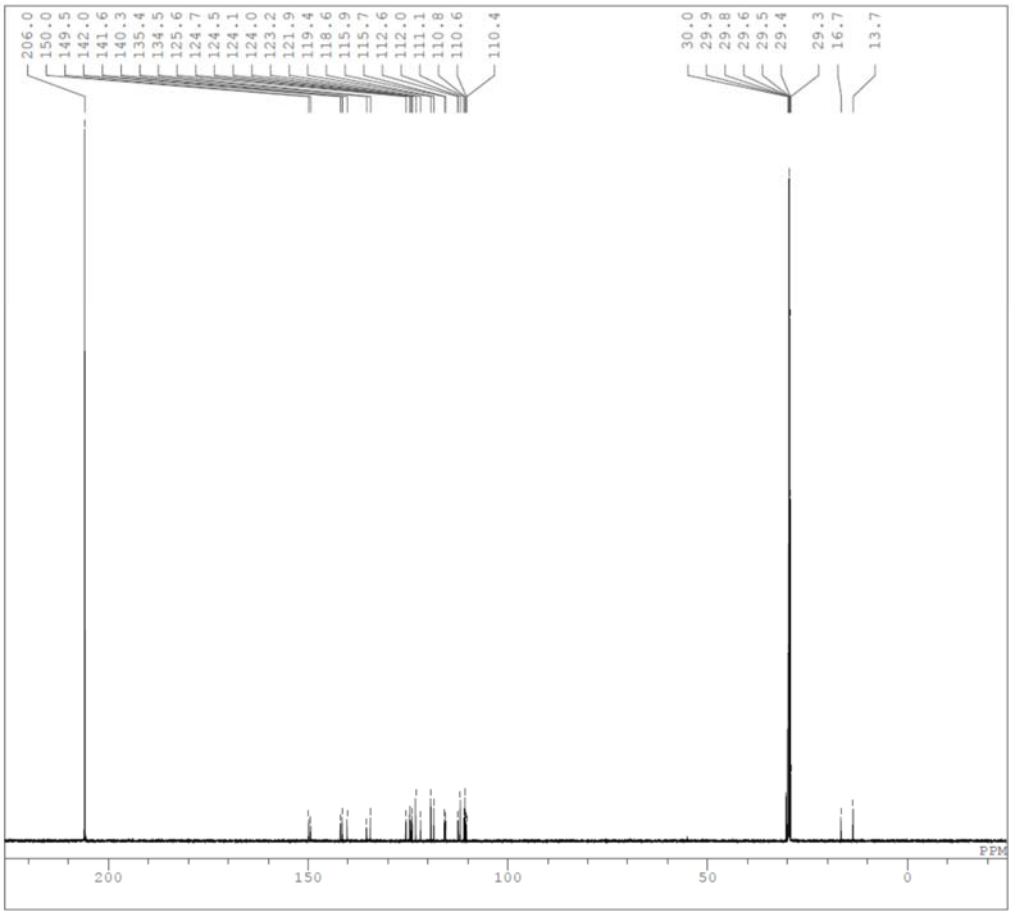
3kl

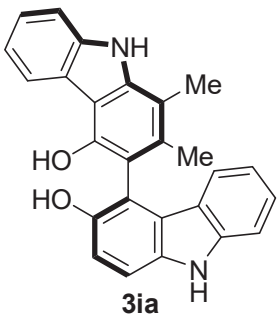
Compound **3kl** (^{1}H NMR, 400 MHz, $CDCl_3$, ^{13}C NMR, 125 MHz, $CDCl_3$)





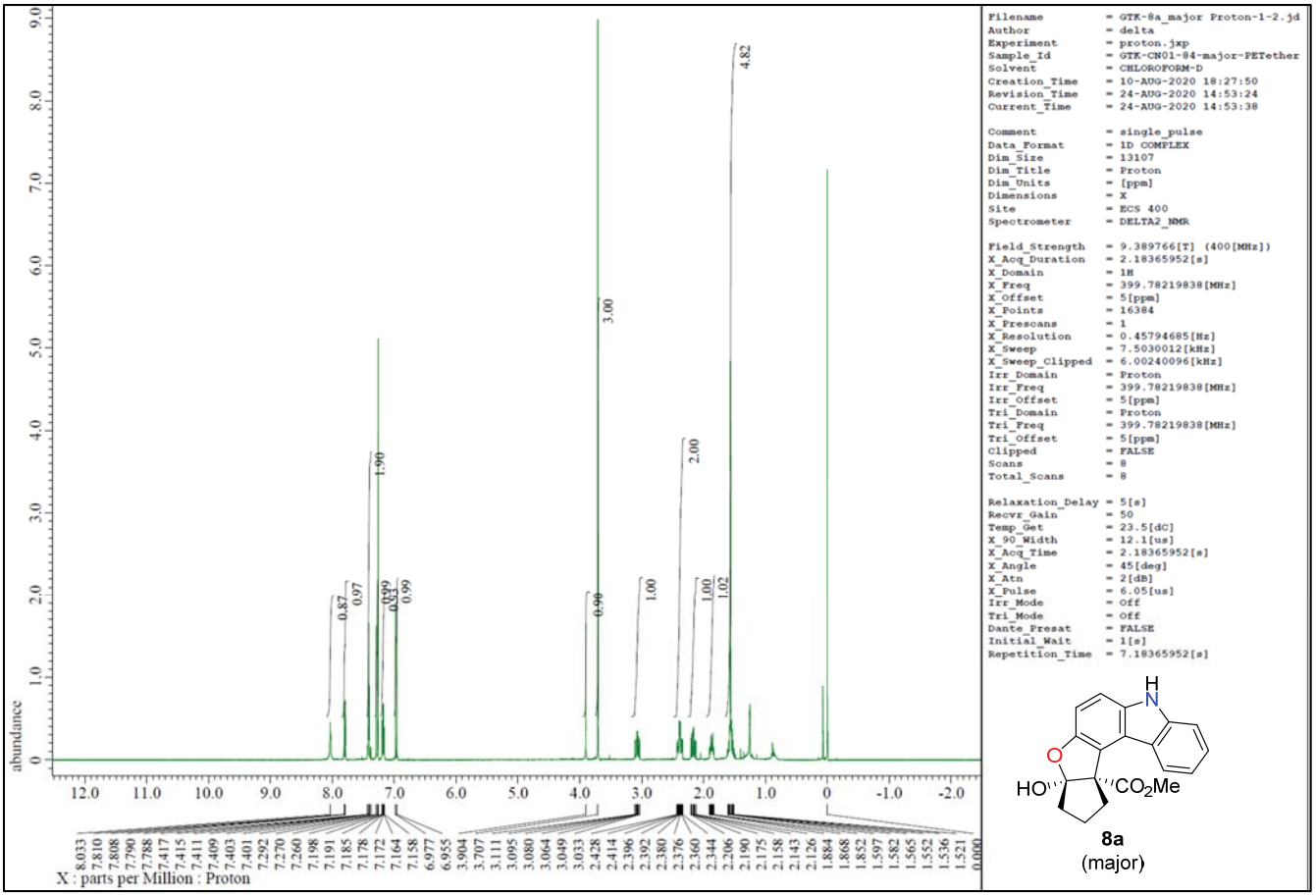
3ia



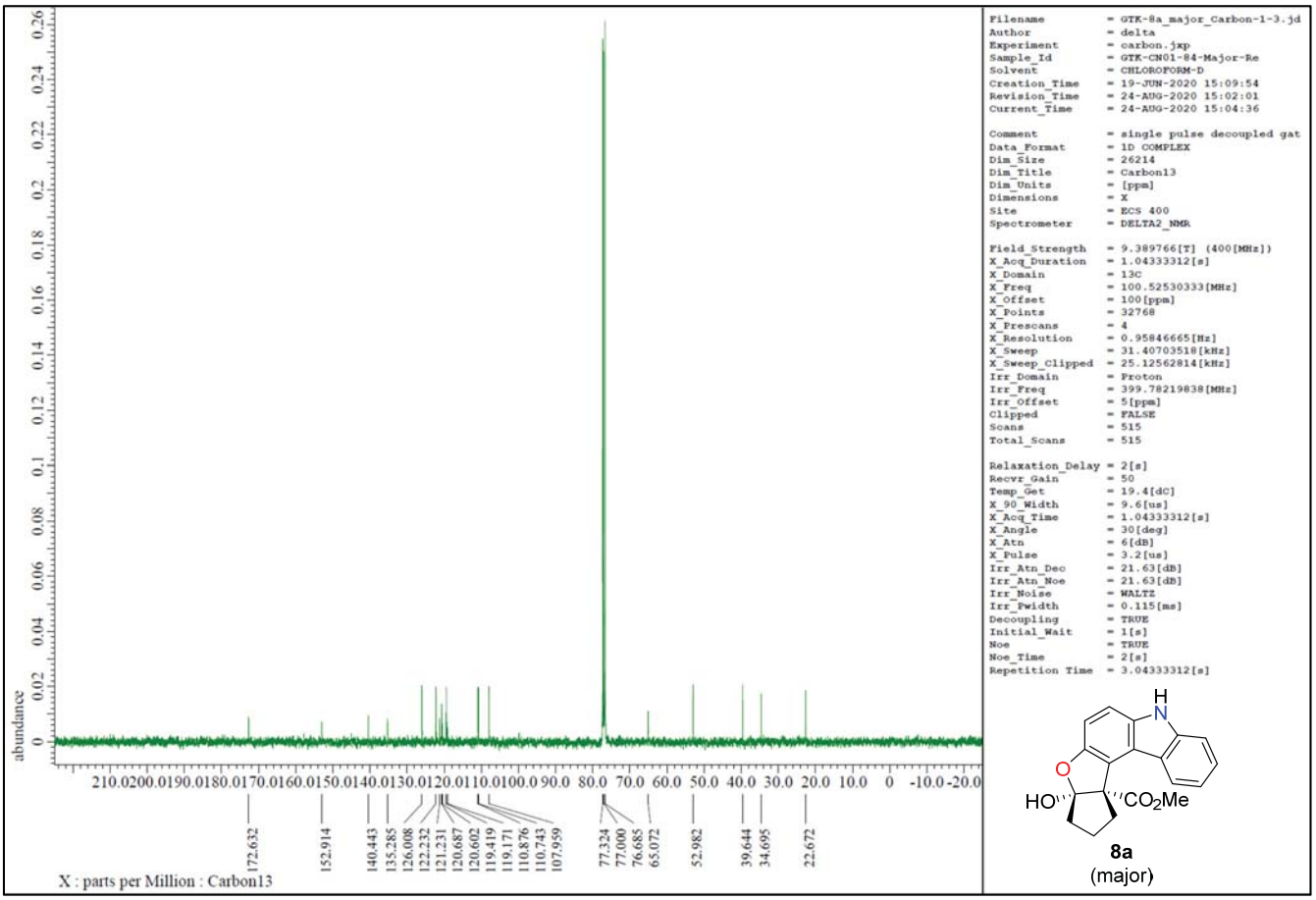


3ia

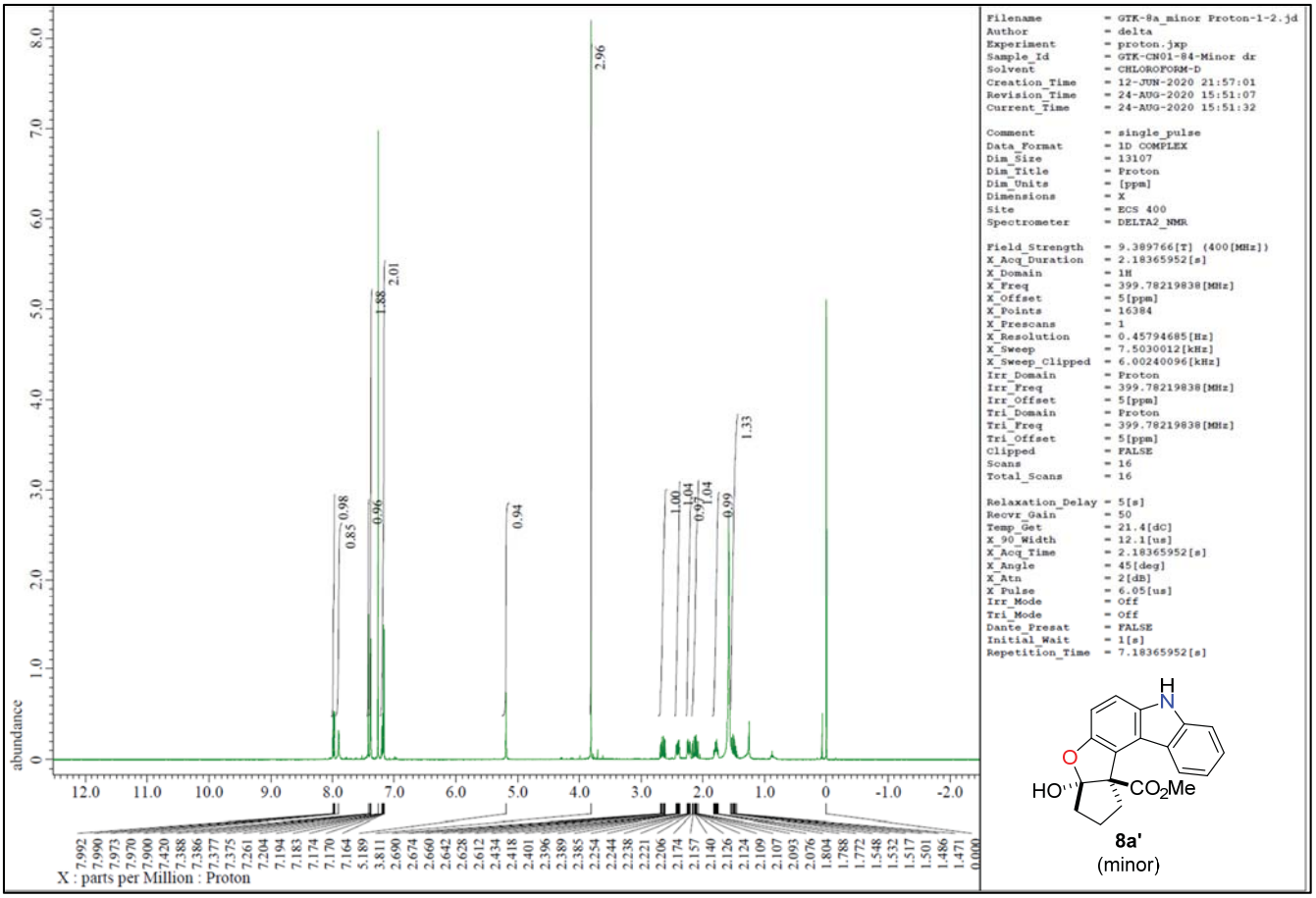
Compound **3ia** (^{1}H NMR, 600 MHz, $(CD_3)_2CO$ and ^{13}C NMR, 125 MHz, $(CD_3)_2CO$)



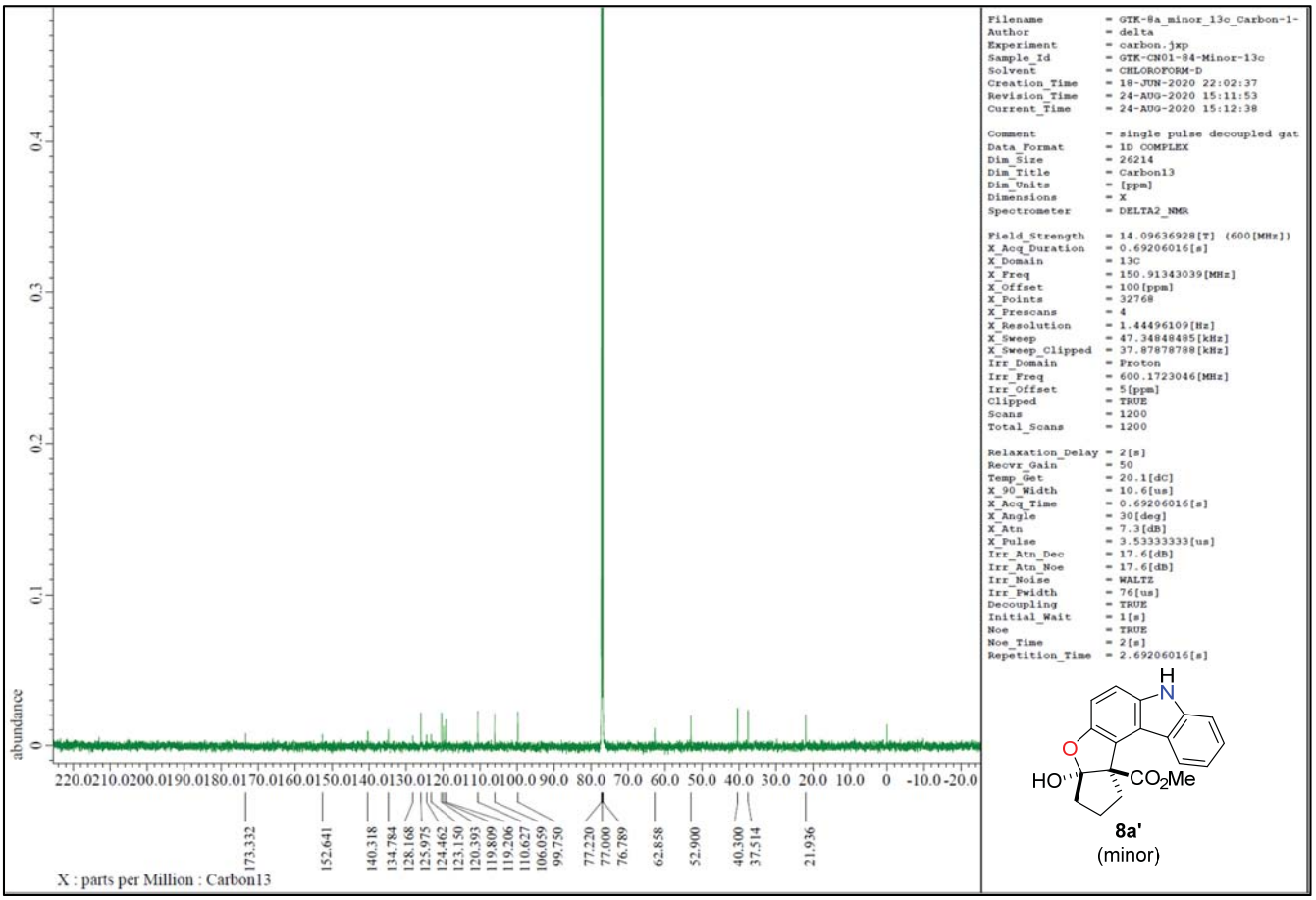
Compound **8a** (^{1}H NMR, 400 MHz, $CDCl_3$)



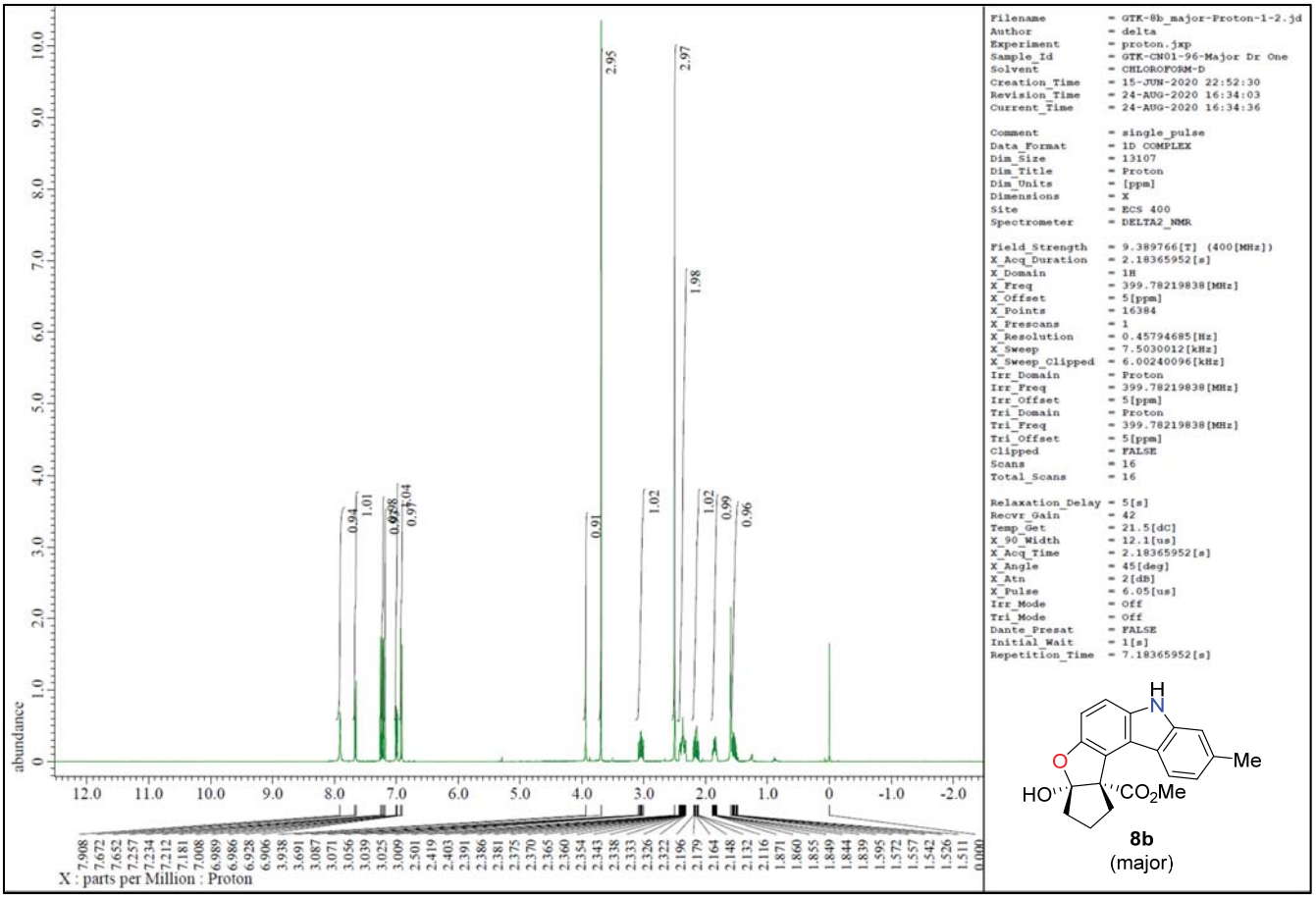
Compound **8a** (^{13}C NMR, 100 MHz, $CDCl_3$)



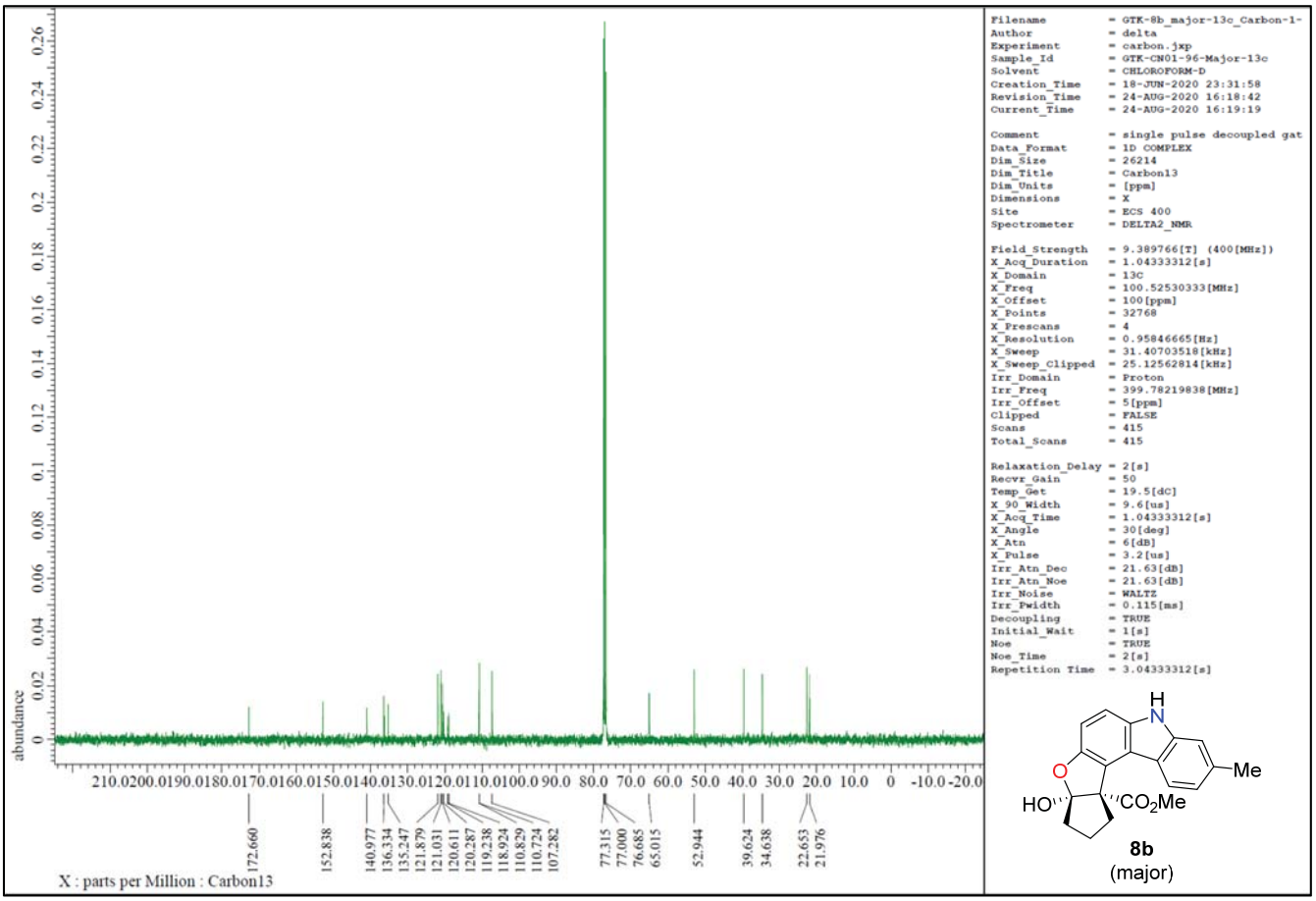
Compound **8a'** (^{1}H NMR, 400 MHz, $CDCl_3$)



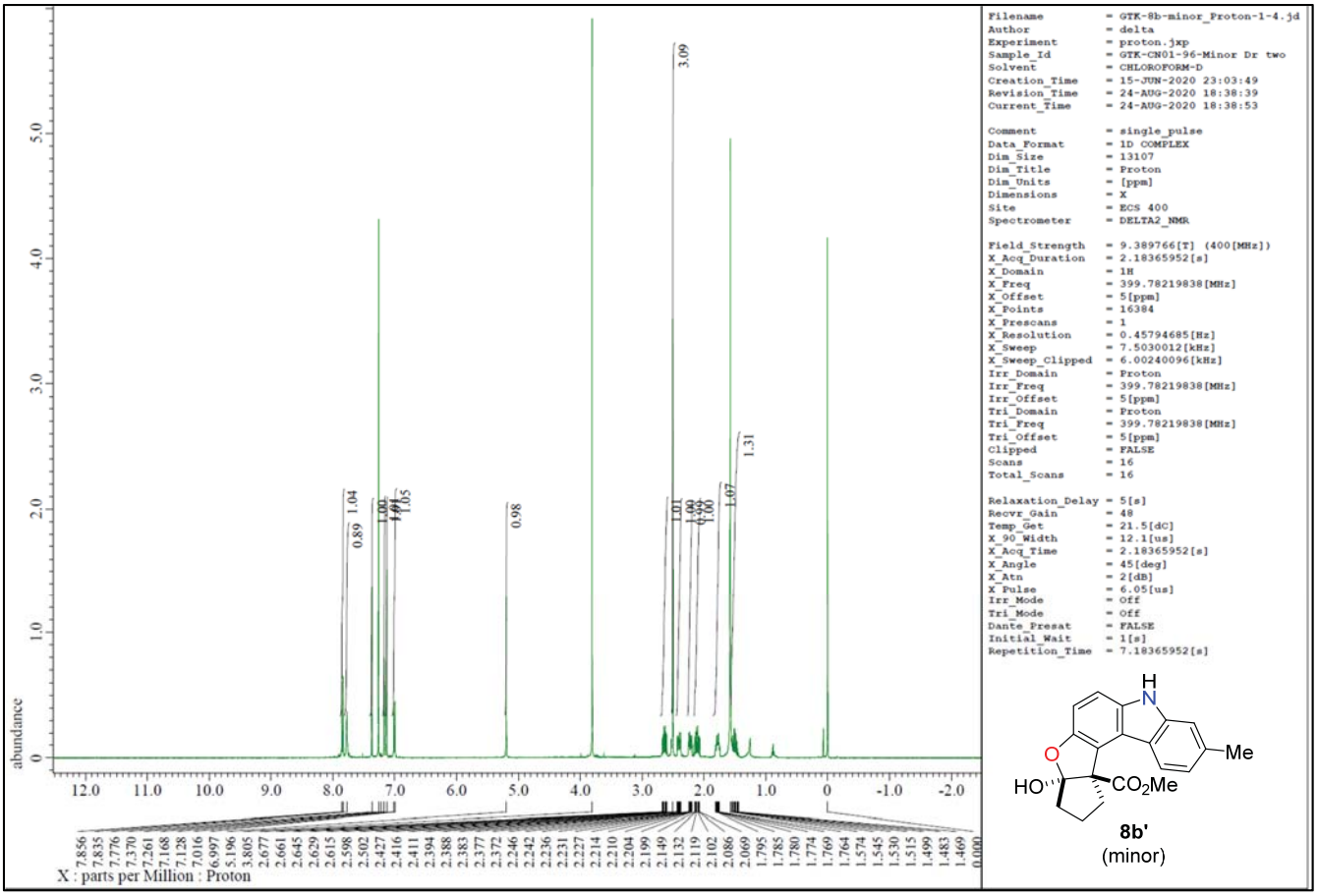
Compound **8a'** (^{13}C NMR, 150 MHz, $CDCl_3$)



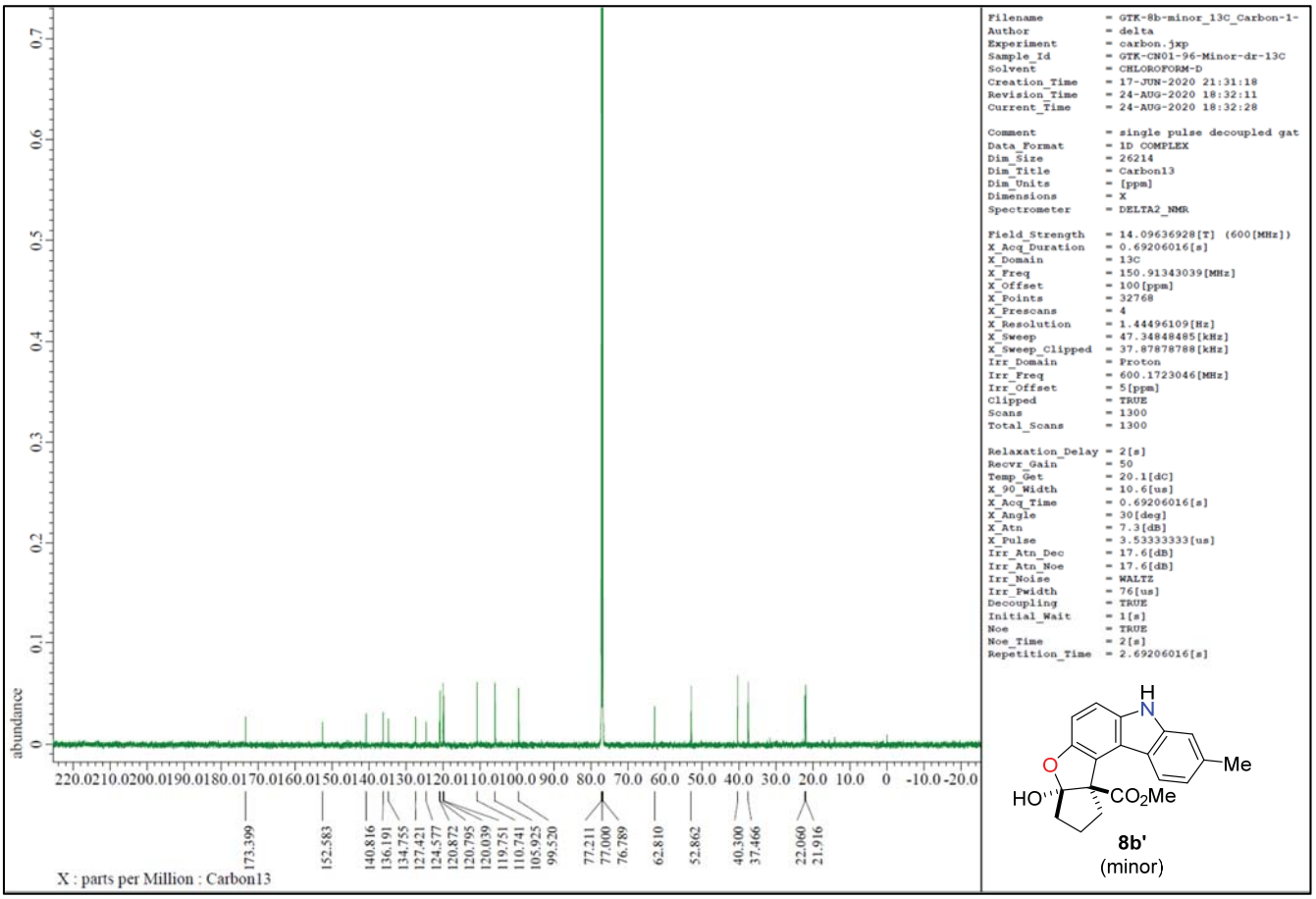
Compound **8b** (^{1}H NMR, 400 MHz, $CDCl_3$)



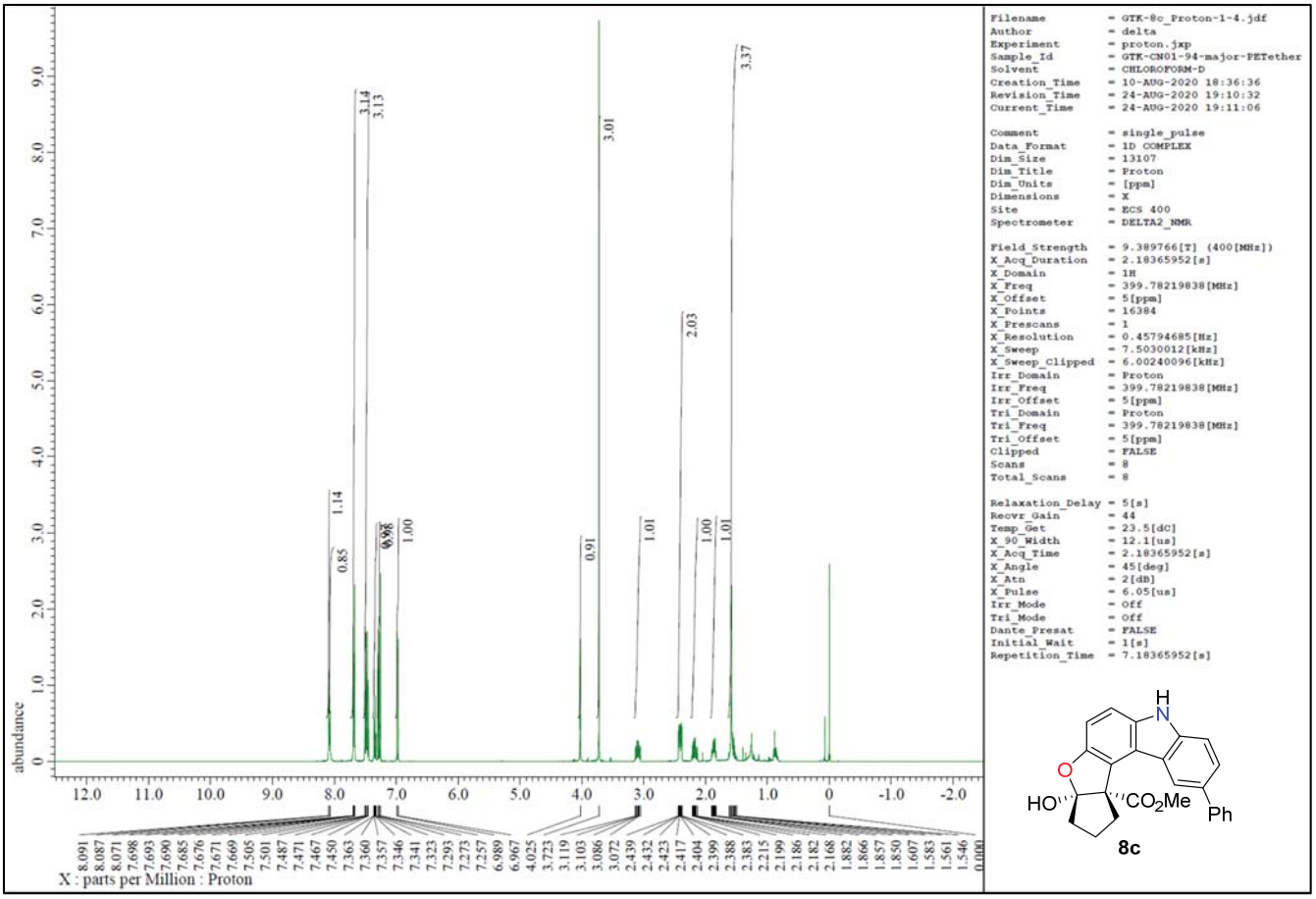
Compound **8b** (^{13}C NMR, 100 MHz, $CDCl_3$)



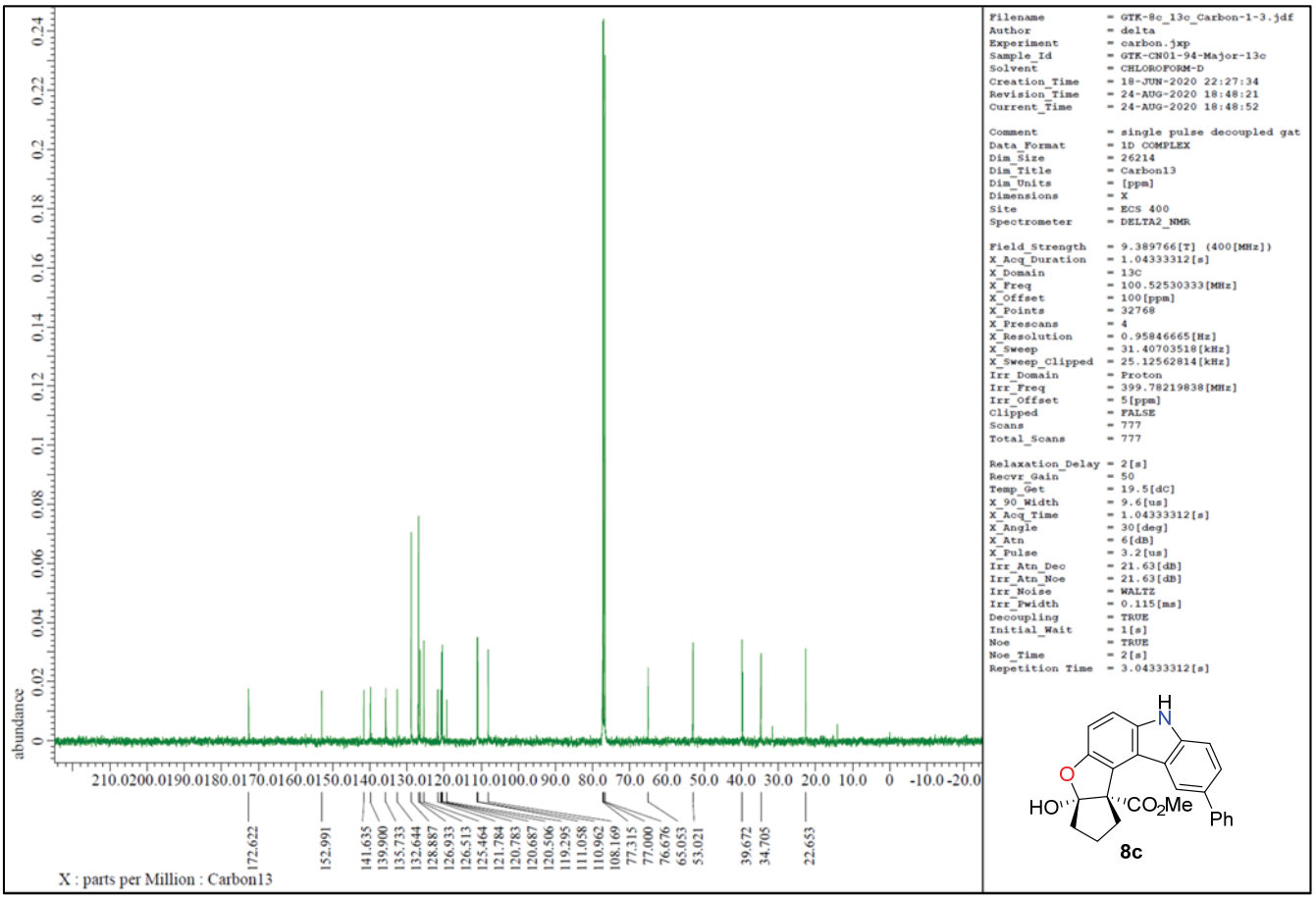
Compound **8b'** (^{1}H NMR, 400 MHz, $CDCl_3$)



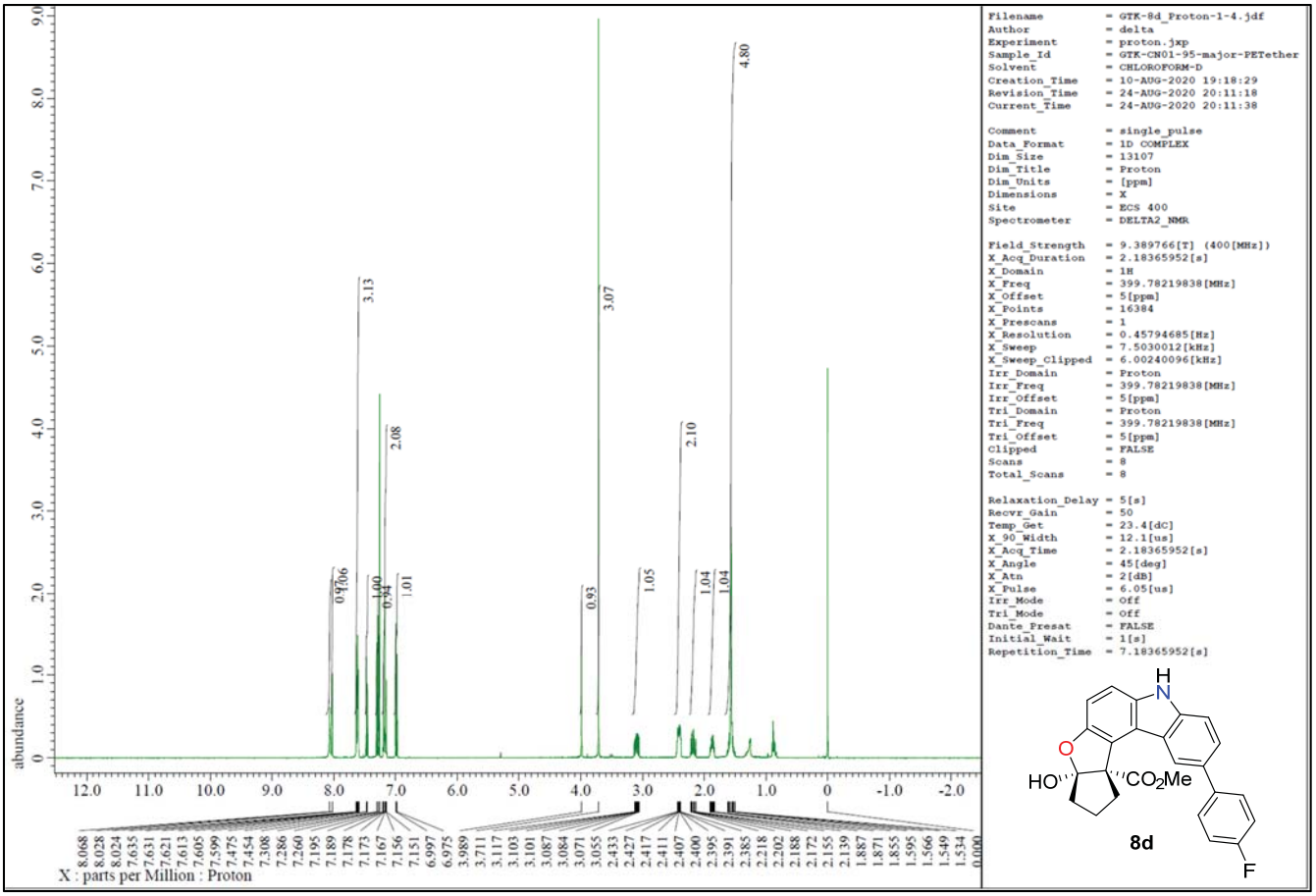
Compound **8b'** (^{13}C NMR, 150 MHz, $CDCl_3$)



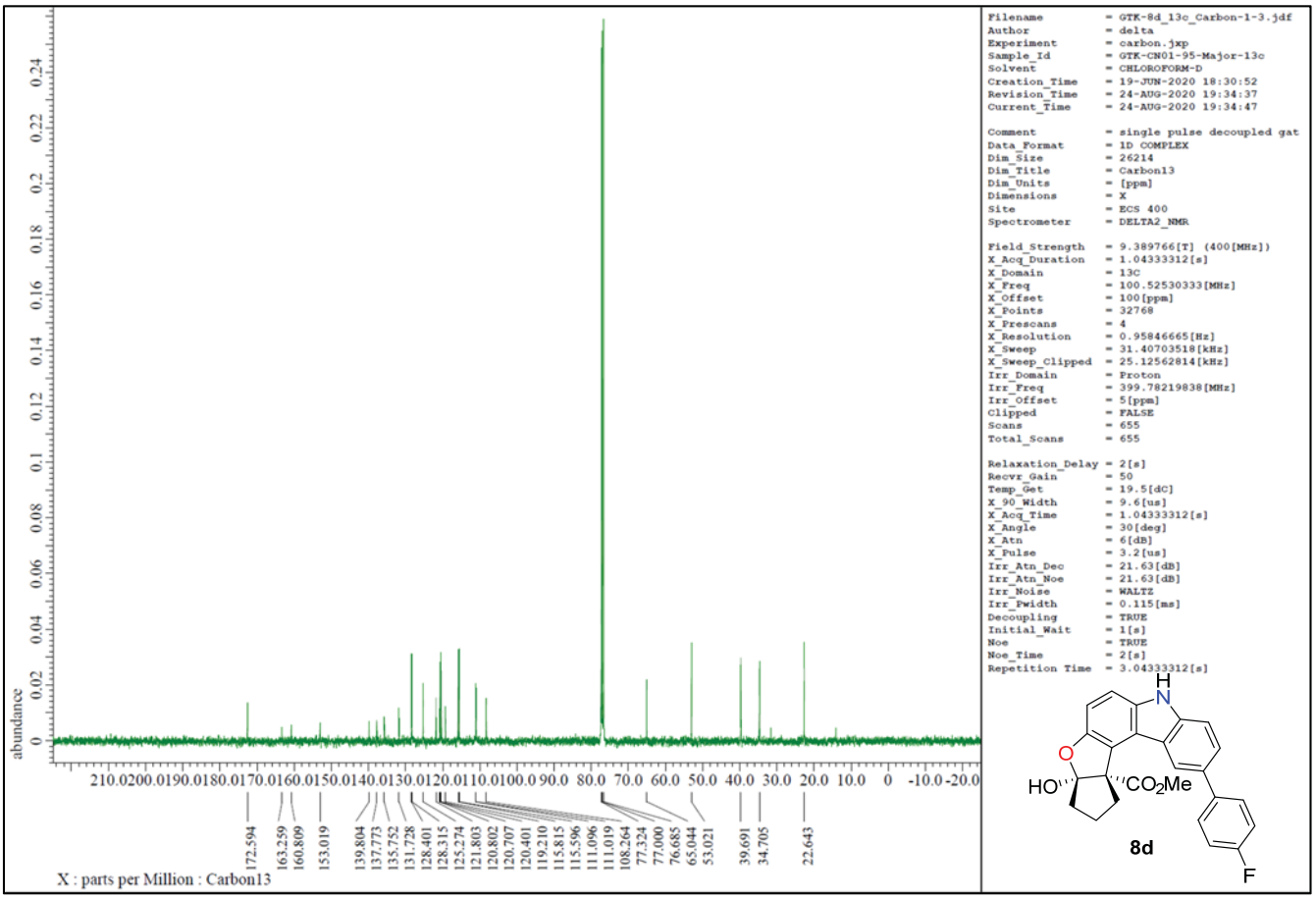
Compound **8c** (^{1}H NMR, 400 MHz, $CDCl_3$)



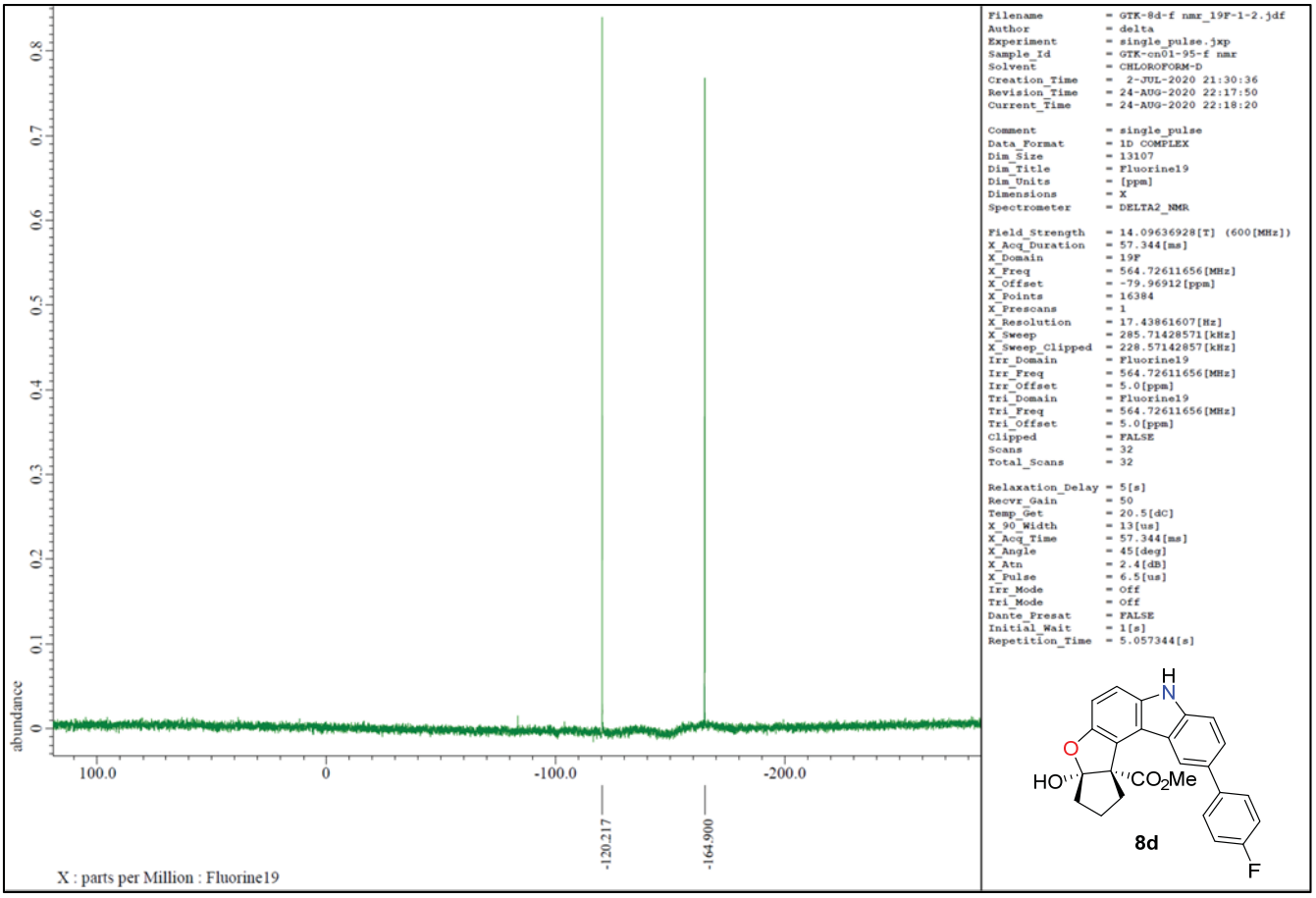
Compound **8c** (^{13}C NMR, 100 MHz, $CDCl_3$)



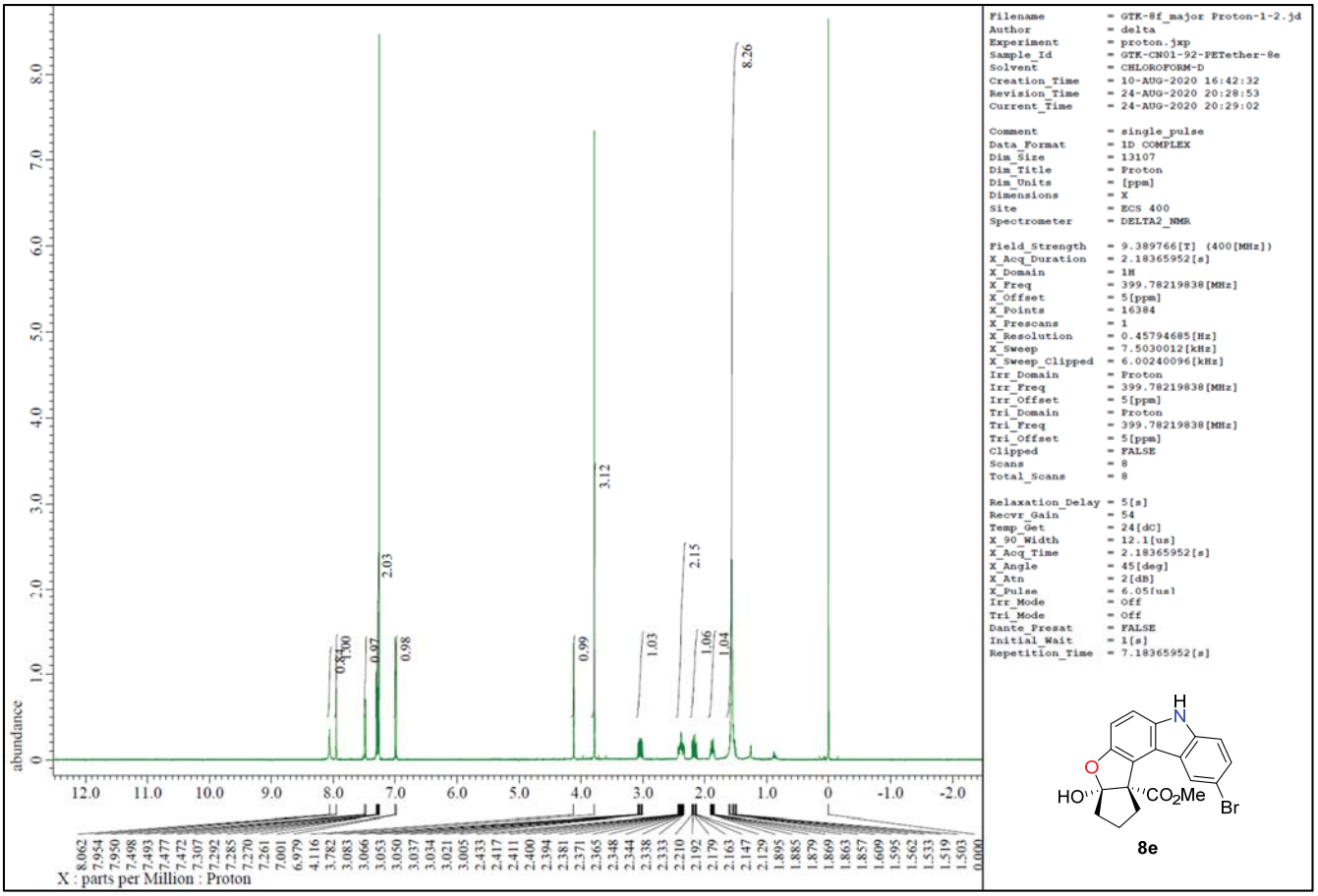
Compound **8d** (^{1}H NMR, 400 MHz, $CDCl_3$)



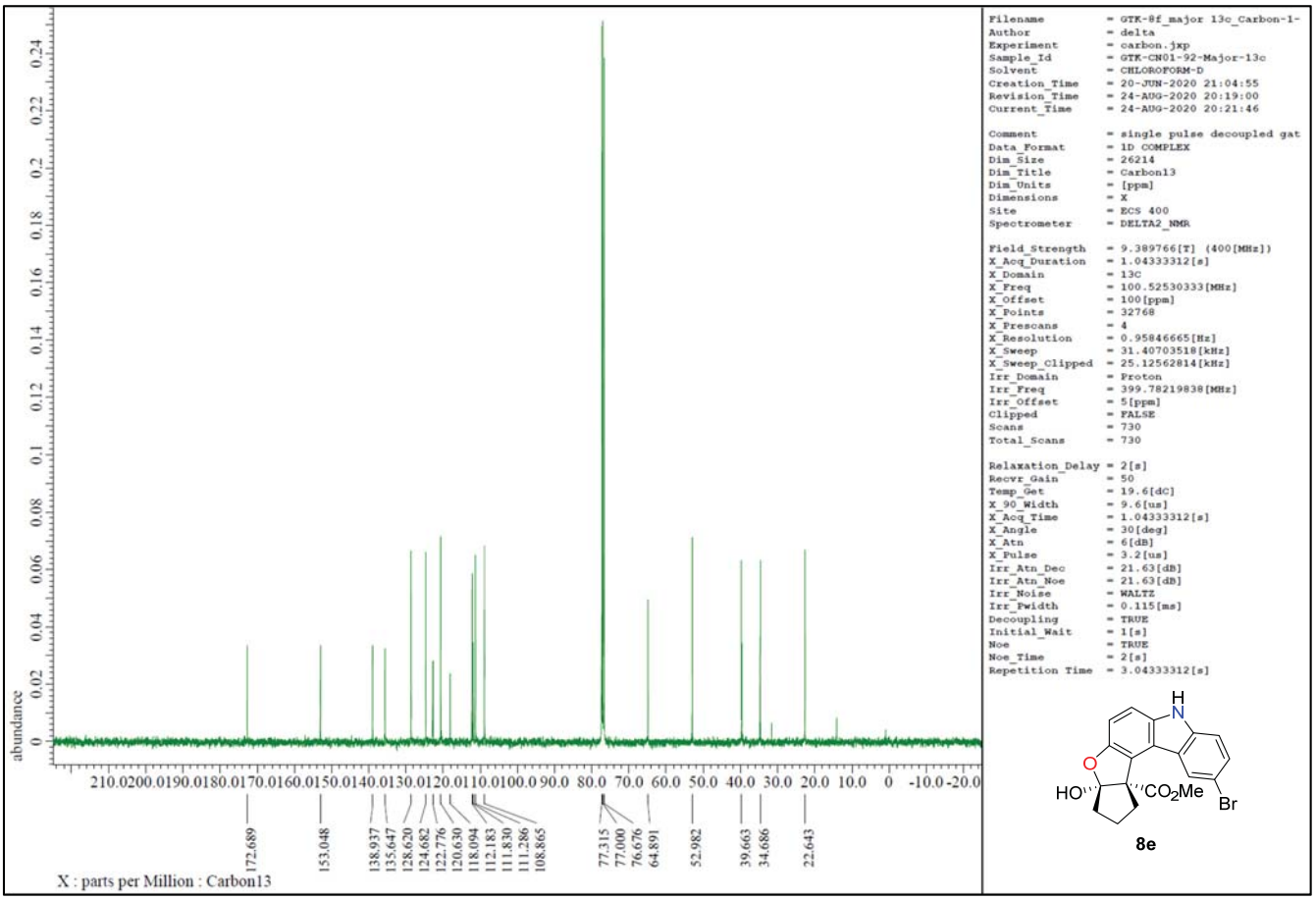
Compound **8d** (^{13}C NMR, 100 MHz, $CDCl_3$)



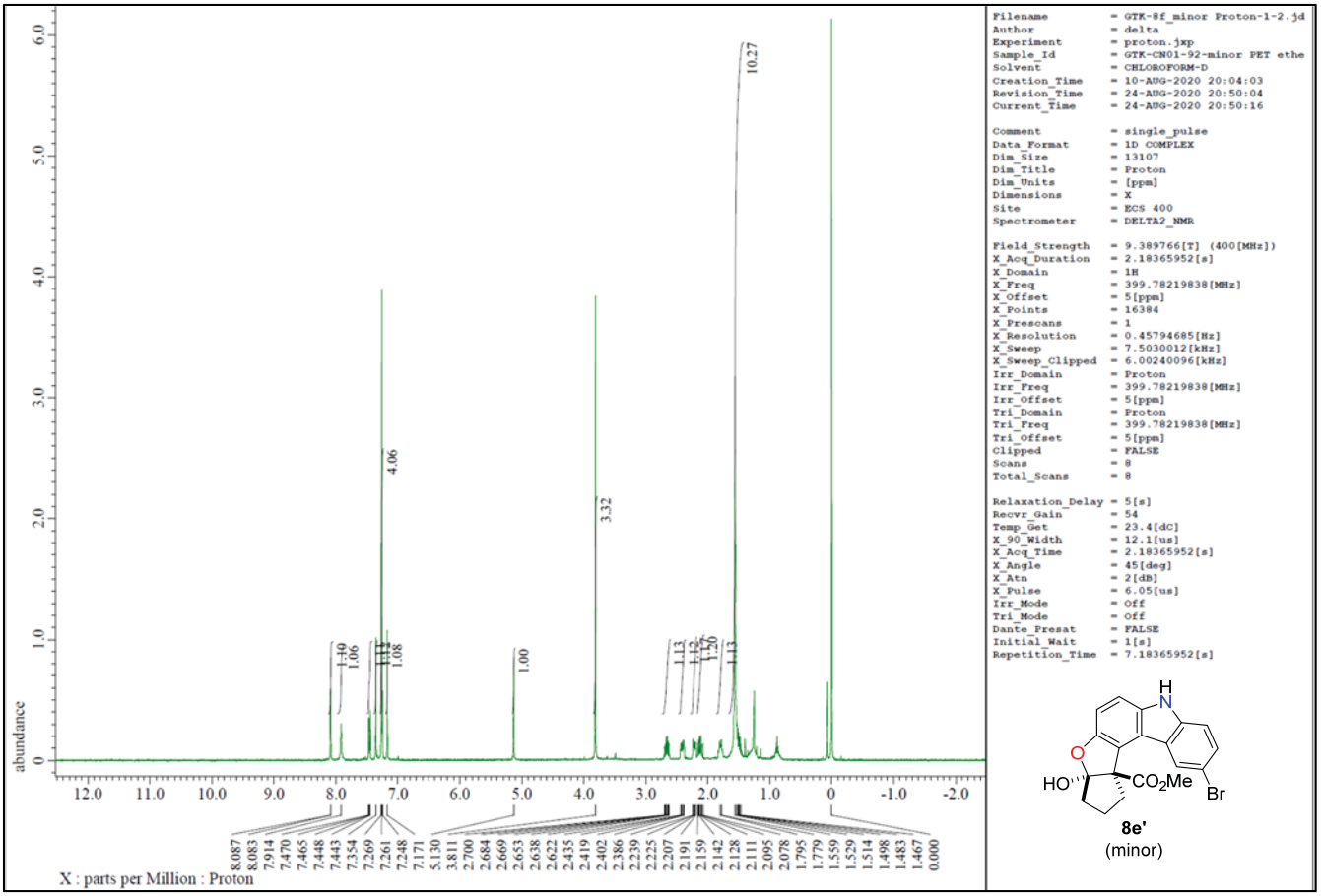
Compound **8d** (^{19}F NMR, 565 MHz, $CDCl_3$)



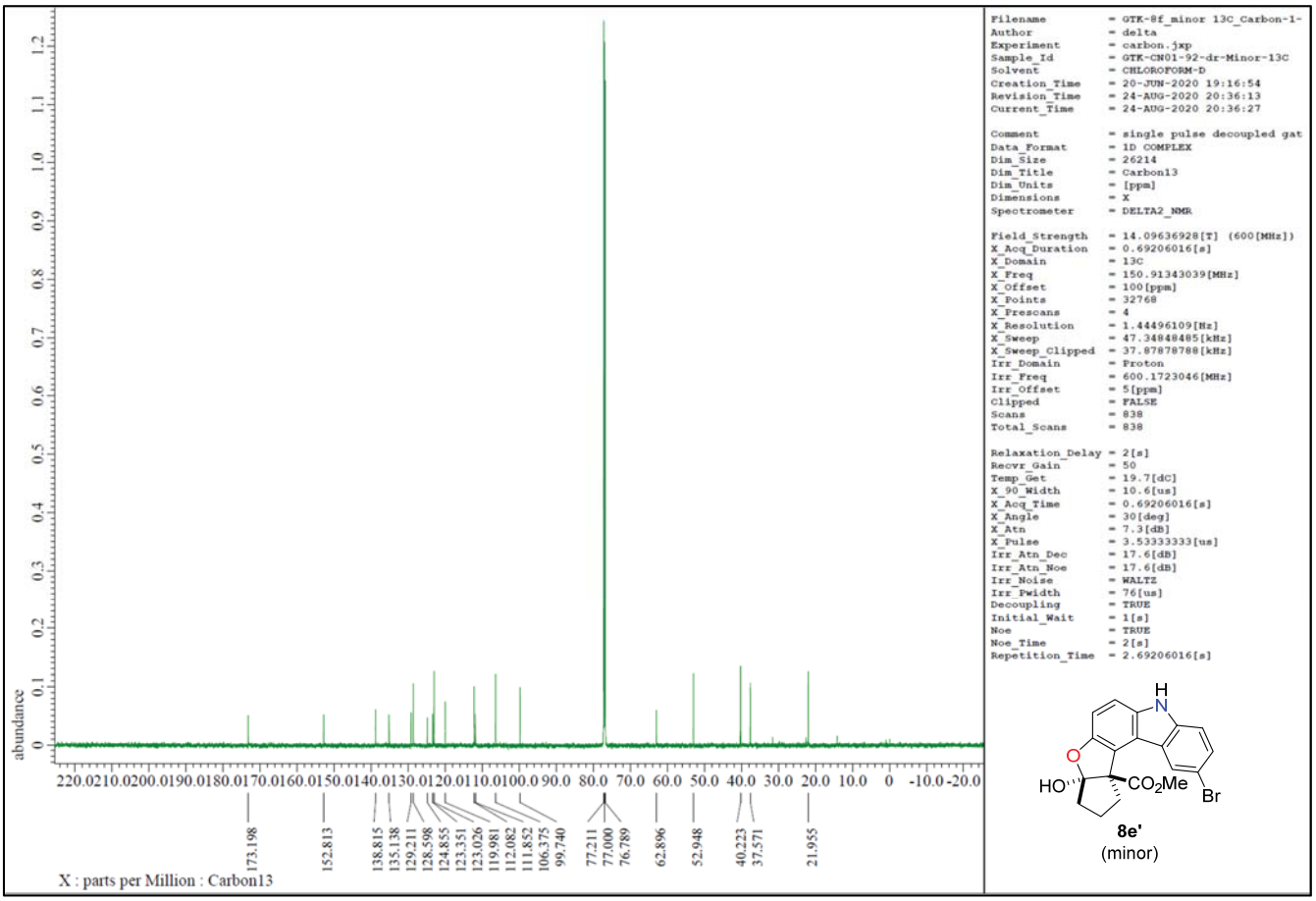
Compound **8e** (^{1}H NMR, 400 MHz, $CDCl_3$)



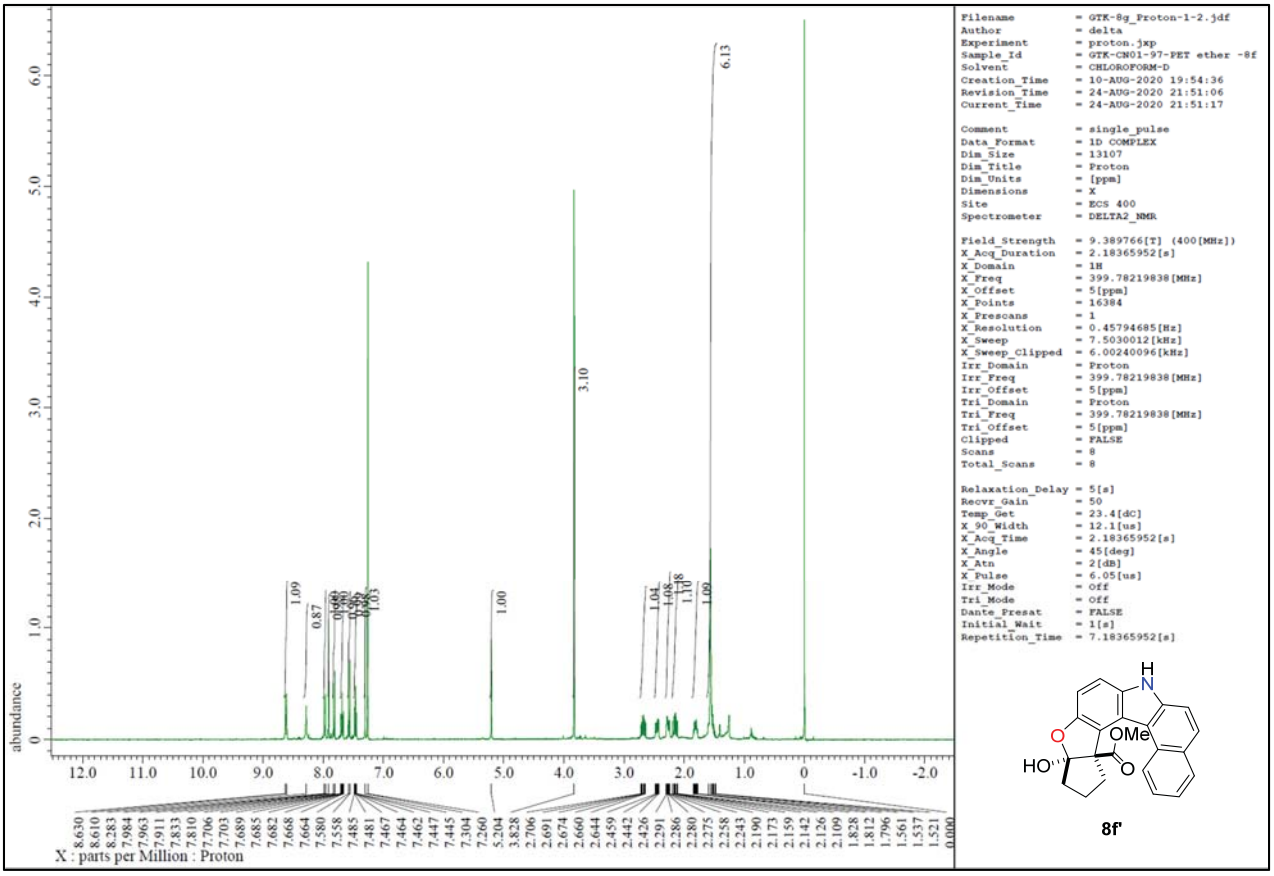
Compound **8e** (^{13}C NMR, 100 MHz, $CDCl_3$)



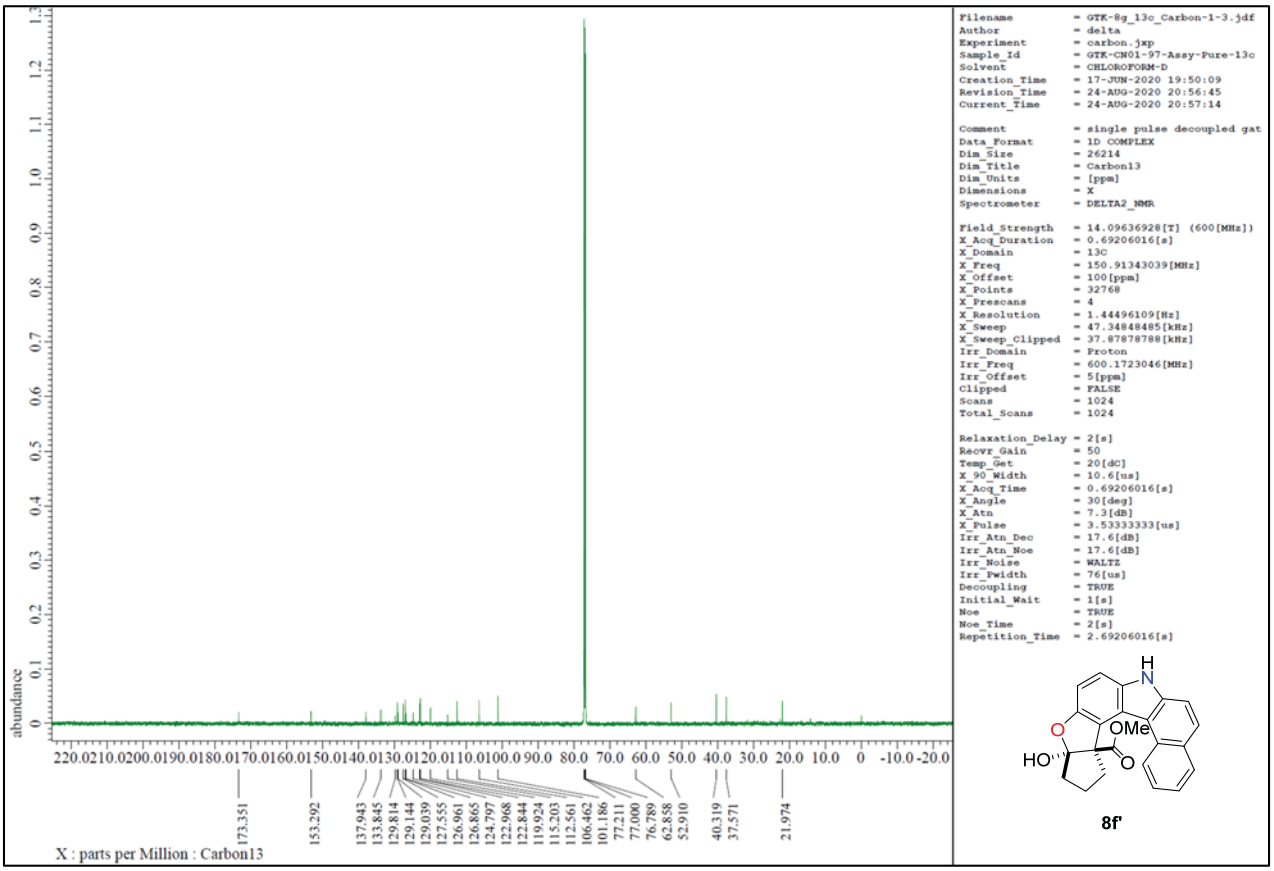
Compound **8e'** (^{1}H NMR, 400 MHz, $CDCl_3$)



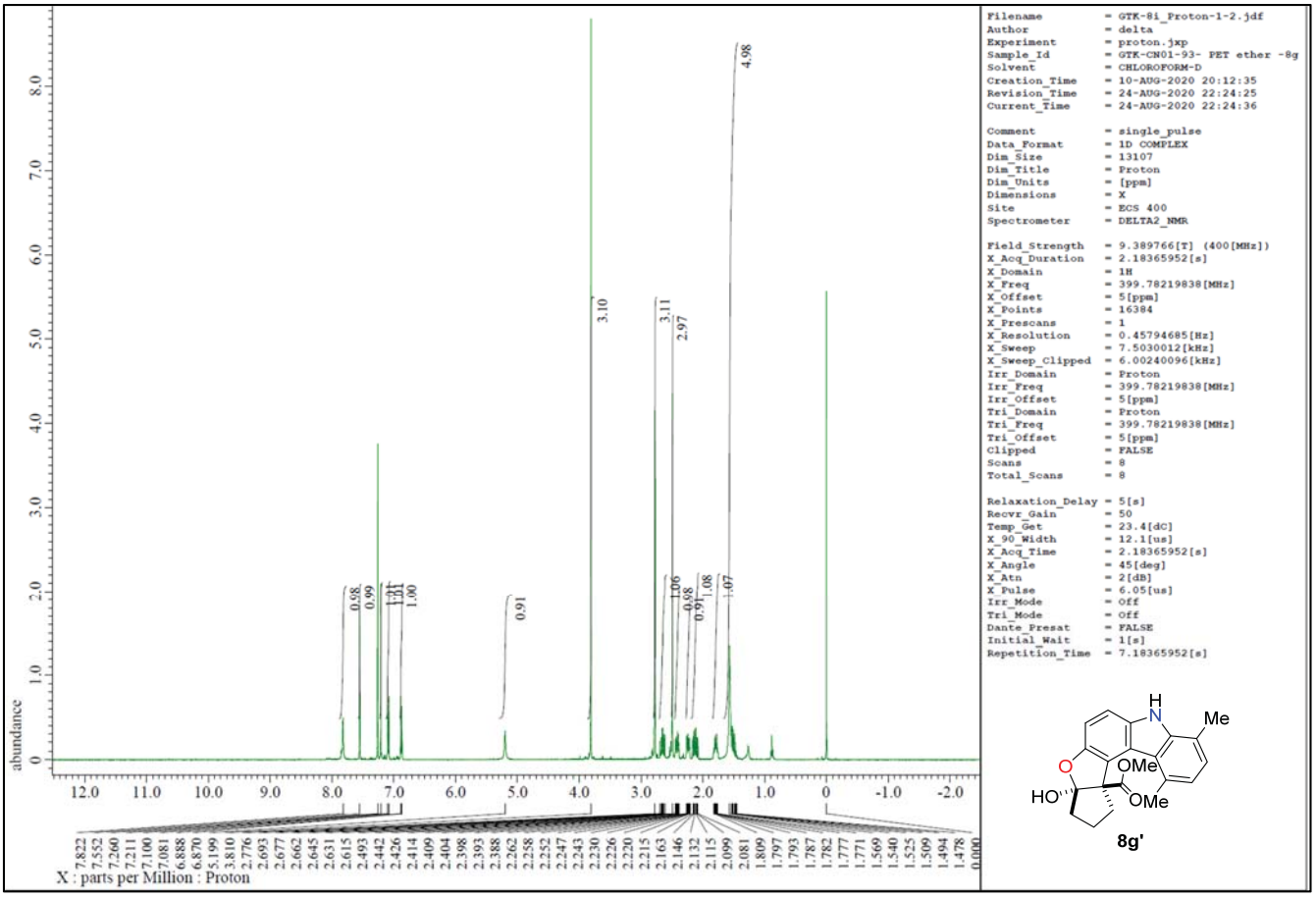
Compound **8e'** (^{13}C NMR, 150 MHz, $CDCl_3$)



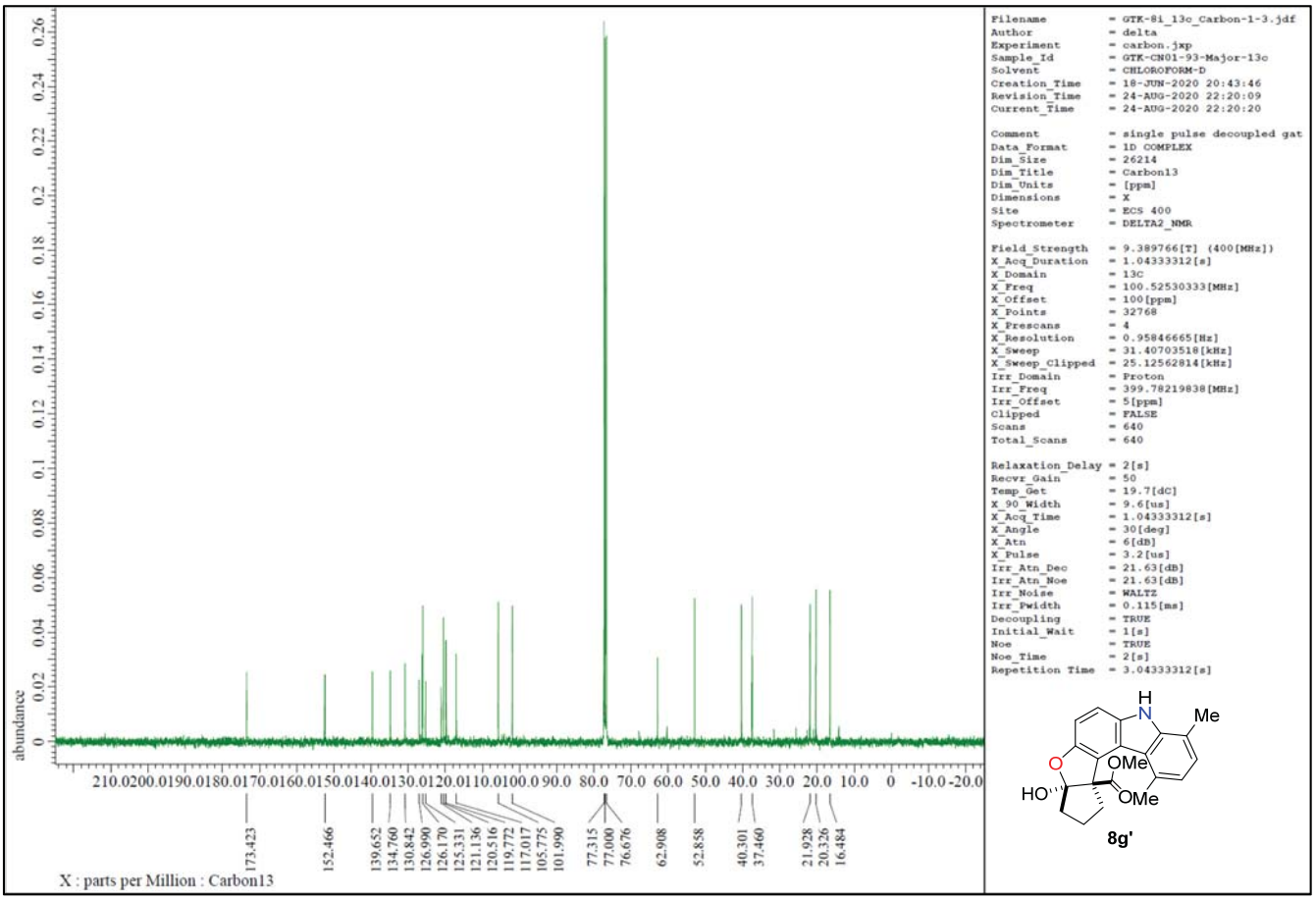
Compound **8f'** (^{1}H NMR, 400 MHz, $CDCl_3$)



Compound **8f'** (^{13}C NMR, 150 MHz, $CDCl_3$)

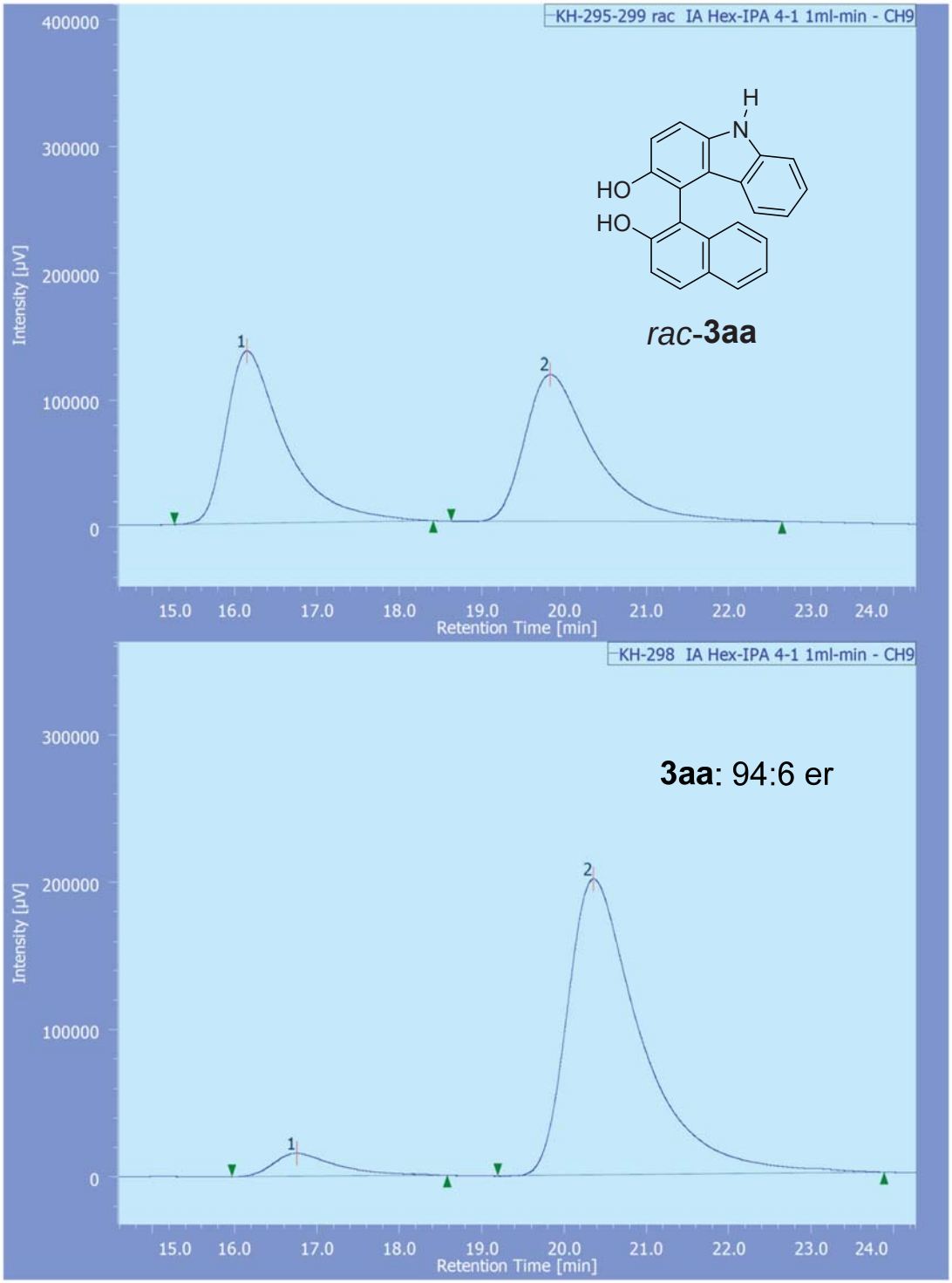


Compound **8g'** (^{1}H NMR, 400 MHz, $CDCl_3$)



Compound **8g'** (^{13}C NMR, 100 MHz, $CDCl_3$)

HPLC charts



Channel & Peak Information Table

Chromatogram Name KH-295-299 rac IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

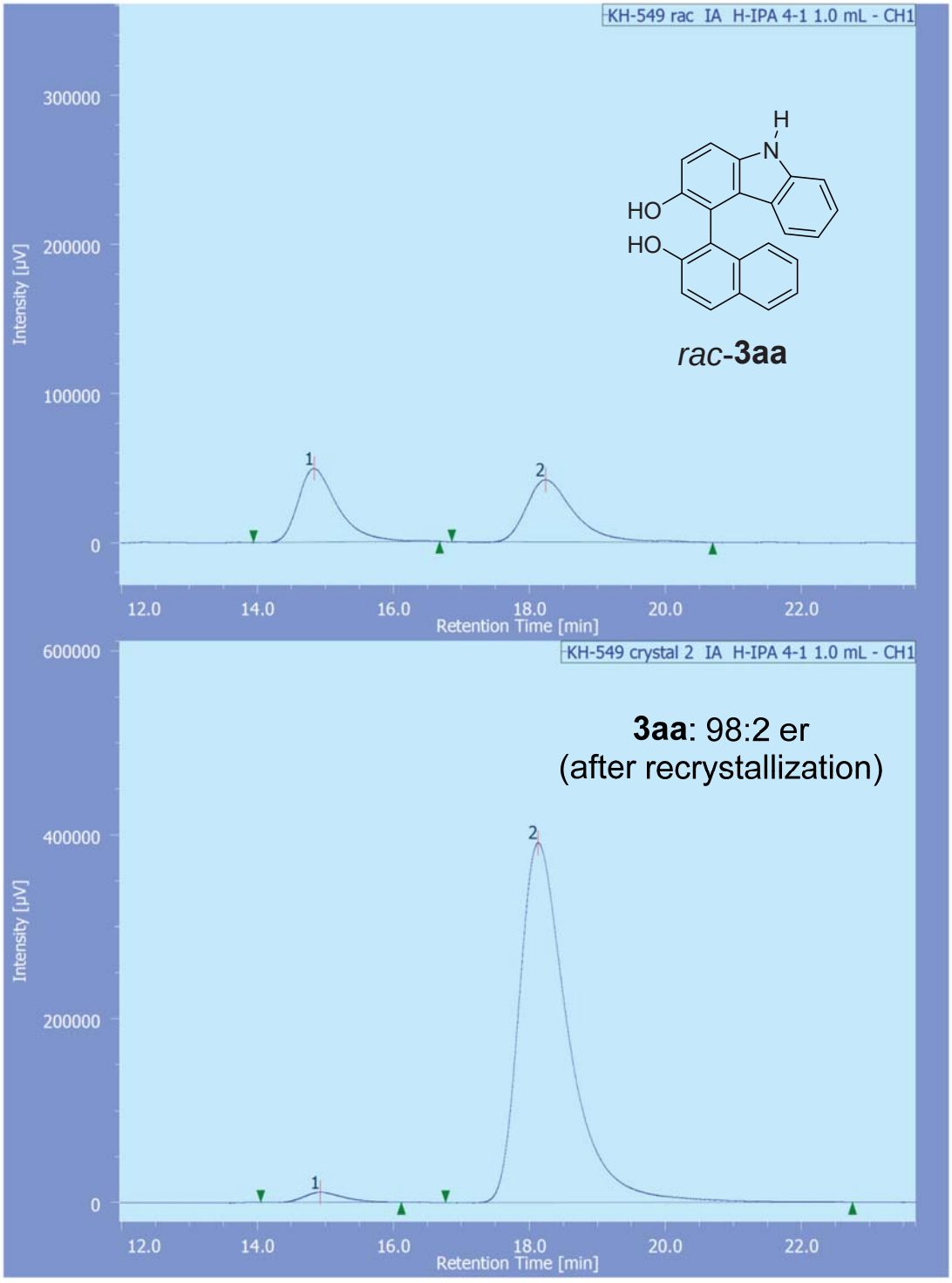
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	16.148	6793798	135586	49.878	53.933	N/A	2874	2.792	1.868	
2	Unknown	9	19.828	6827102	115811	50.122	46.067	N/A	3042	N/A	1.728	

Chromatogram Name KH-298 IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	16.753	832607	15541	6.219	7.185	N/A	2569	2.558	1.788	
2	Unknown	9	20.353	12555364	200764	93.781	92.815	N/A	2944	N/A	1.845	



Channel & Peak Information Table

Chromatogram Name KH-549 rac IA H-IPA 4-1 1.0 mL-CH1

Sample Name

Channel Name UV-2075

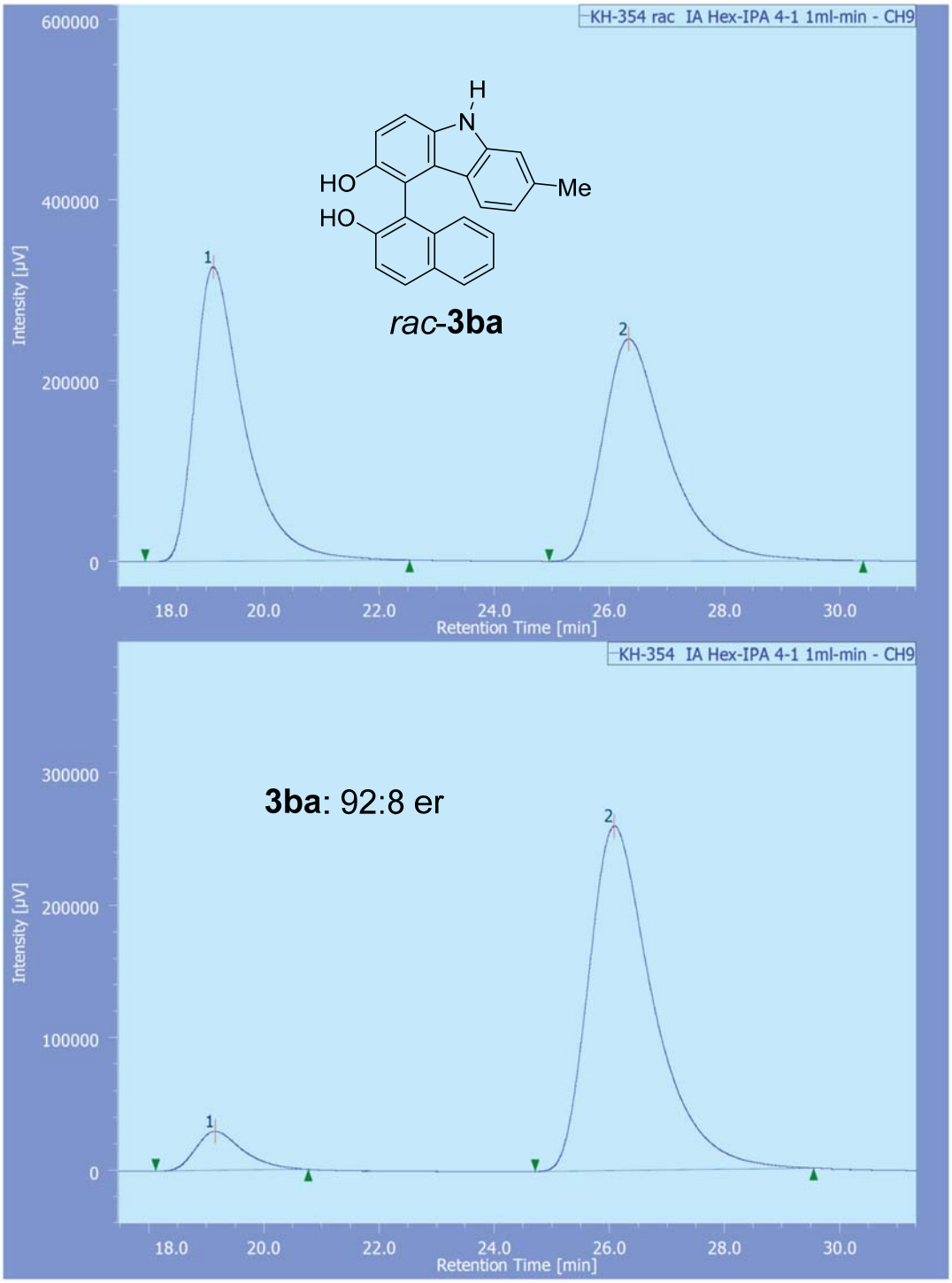
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	14.833	1990786	49152	49.353	54.246	N/A	3473	3.059	1.511	
2	Unknown	1	18.233	2043016	41457	50.647	45.754	N/A	3561	N/A	1.493	

Chromatogram Name KH-549 crystal 2 IA H-IPA 4-1 1.0 mL-CH1

Sample Name

Channel Name UV-2075

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	14.925	437396	10830	2.176	2.692	N/A	3177	2.805	1.354	
2	Unknown	1	18.125	19664290	391479	97.824	97.308	N/A	3483	N/A	1.595	



Channel & Peak Information Table

Chromatogram Name KH-354 rac IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

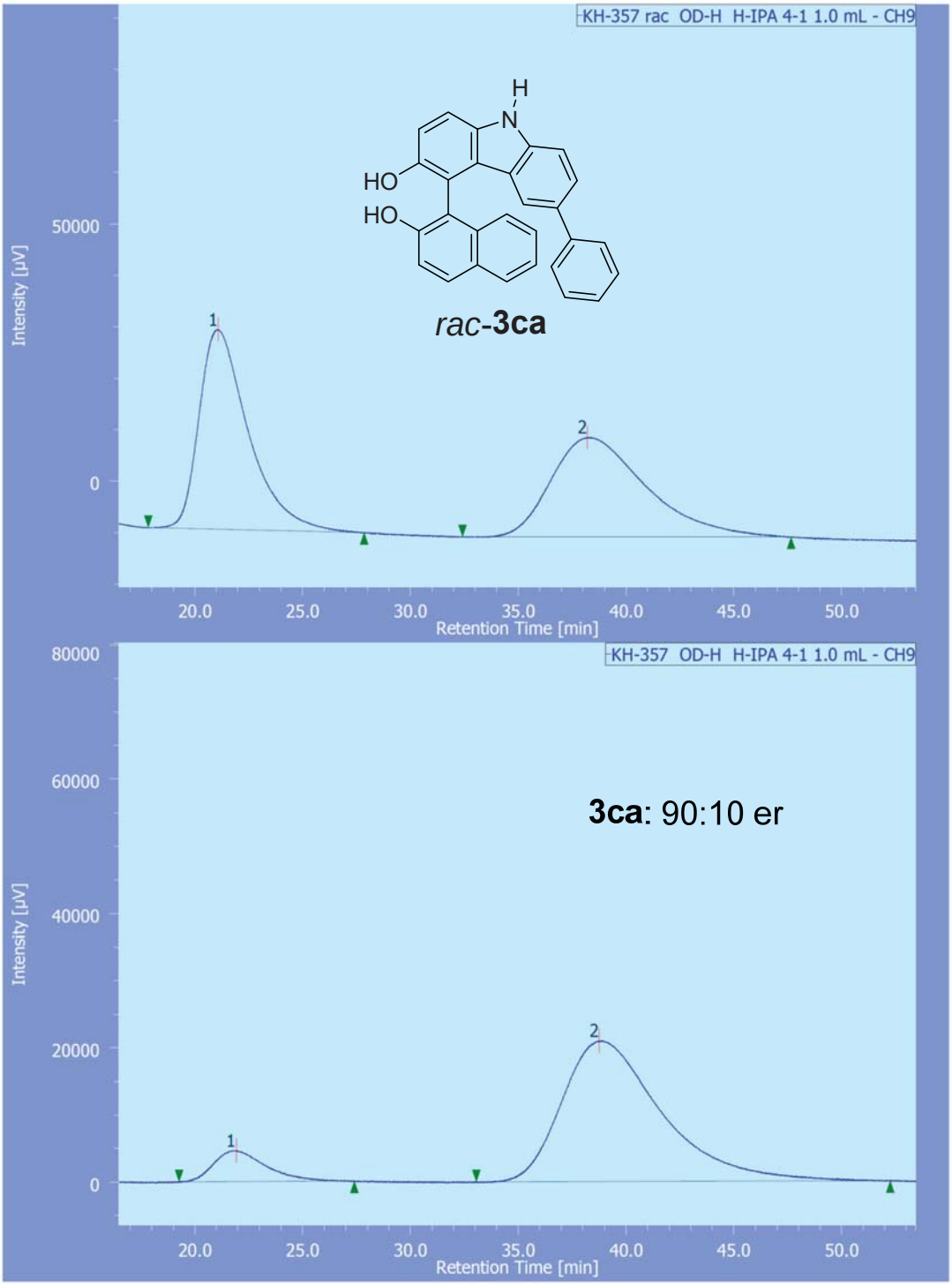
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	19.117	19444416	325735	49.923	56.997	N/A	2717	4.196	1.675	
2	Unknown	9	26.328	19504490	245765	50.077	43.003	N/A	2831	N/A	1.550	

Chromatogram Name KH-354 IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	19.157	1710540	29883	7.778	10.300	N/A	2724	4.059	1.388	
2	Unknown	9	26.083	20281863	260242	92.222	89.700	N/A	2852	N/A	1.530	



Channel & Peak Information Table

Chromatogram Name KH-357 rac OD-H H-IPA 4-1 1.0 mL-CH9

Sample Name

Channel Name 230.0nm

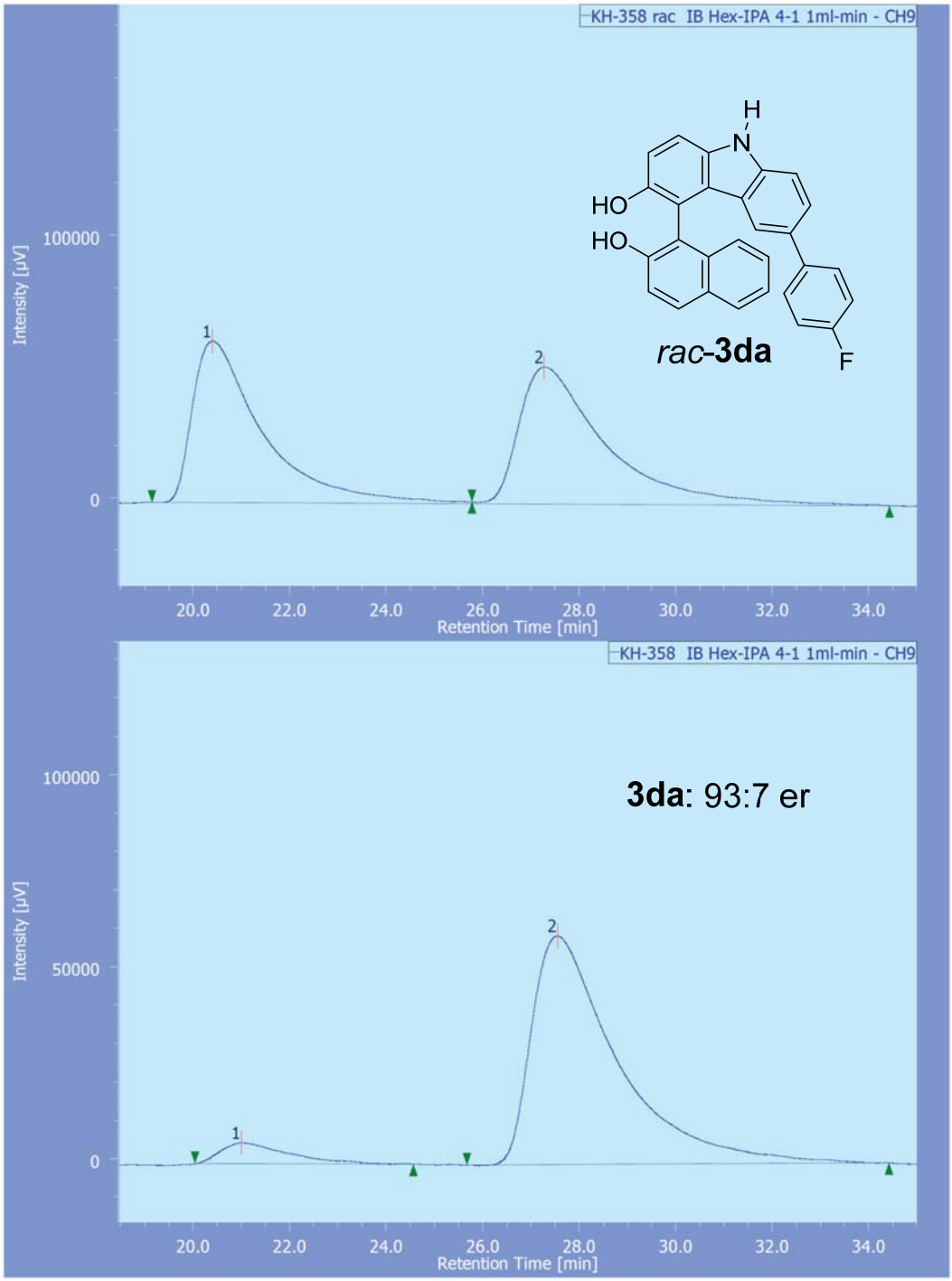
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	21.078	6142836	38706	51.109	66.733	N/A	450	2.911	1.597	
2	Unknown	9	38.188	5876261	19295	48.891	33.267	N/A	382	N/A	1.516	

Chromatogram Name KH-357 OD-H H-IPA 4-1 1.0 mL-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	21.913	789693	4662	10.461	18.193	N/A	409	2.709	1.462	
2	Unknown	9	38.753	6759045	20964	89.539	81.807	N/A	363	N/A	1.583	



Channel & Peak Information Table

Chromatogram Name KH-358 rac IB Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

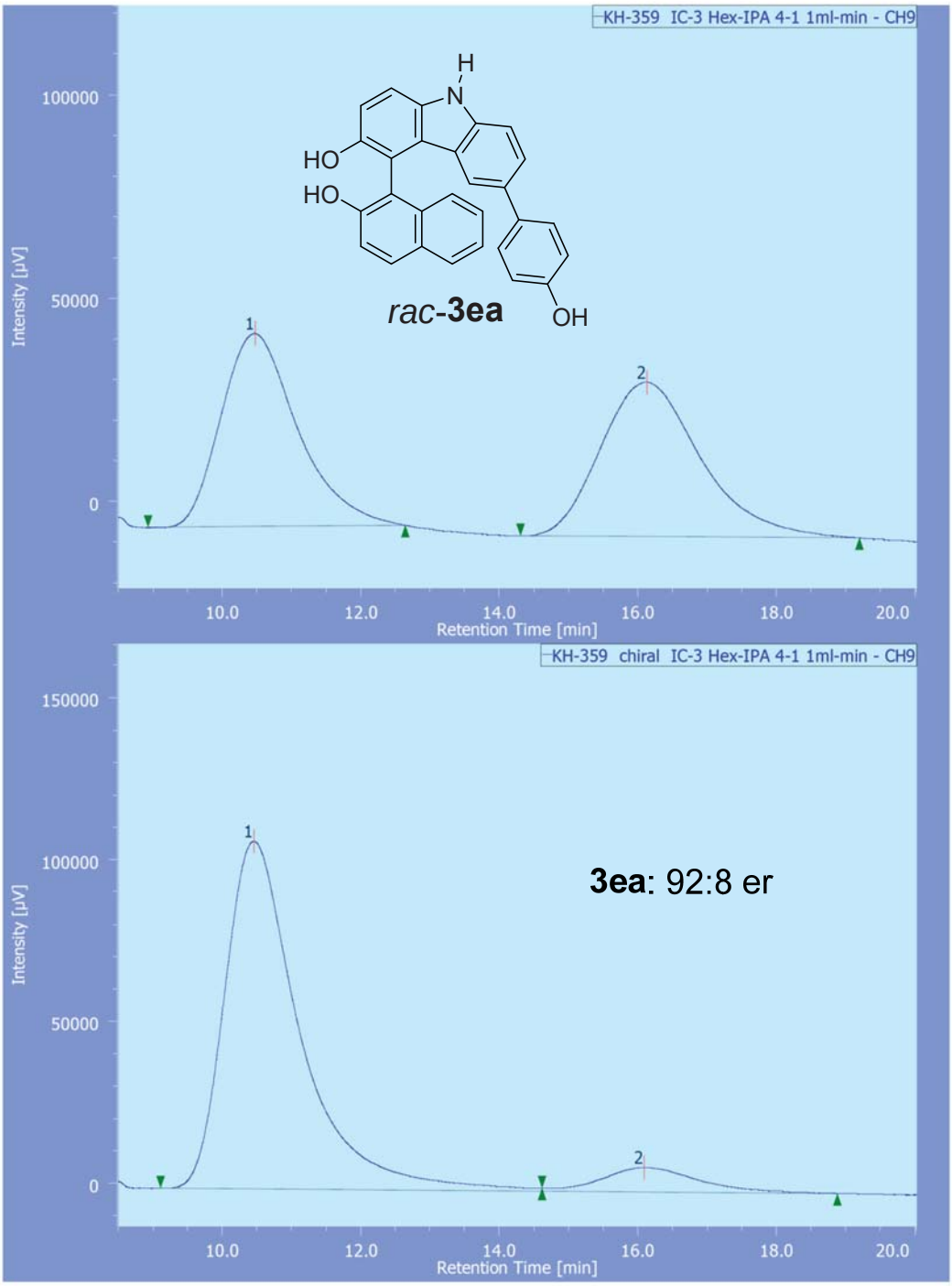
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	20.400	6149362	61423	48.820	54.097	N/A	1235	2.636	2.686	
2	Unknown	9	27.263	6446716	52118	51.180	45.903	N/A	1415	N/A	2.442	

Chromatogram Name KH-358 IB Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	20.998	543690	5516	6.869	8.467	N/A	1105	2.402	2.041	
2	Unknown	9	27.543	7371592	59626	93.131	91.533	N/A	1408	N/A	2.415	



Channel & Peak Information Table

Chromatogram Name KH-359 IC-3 Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

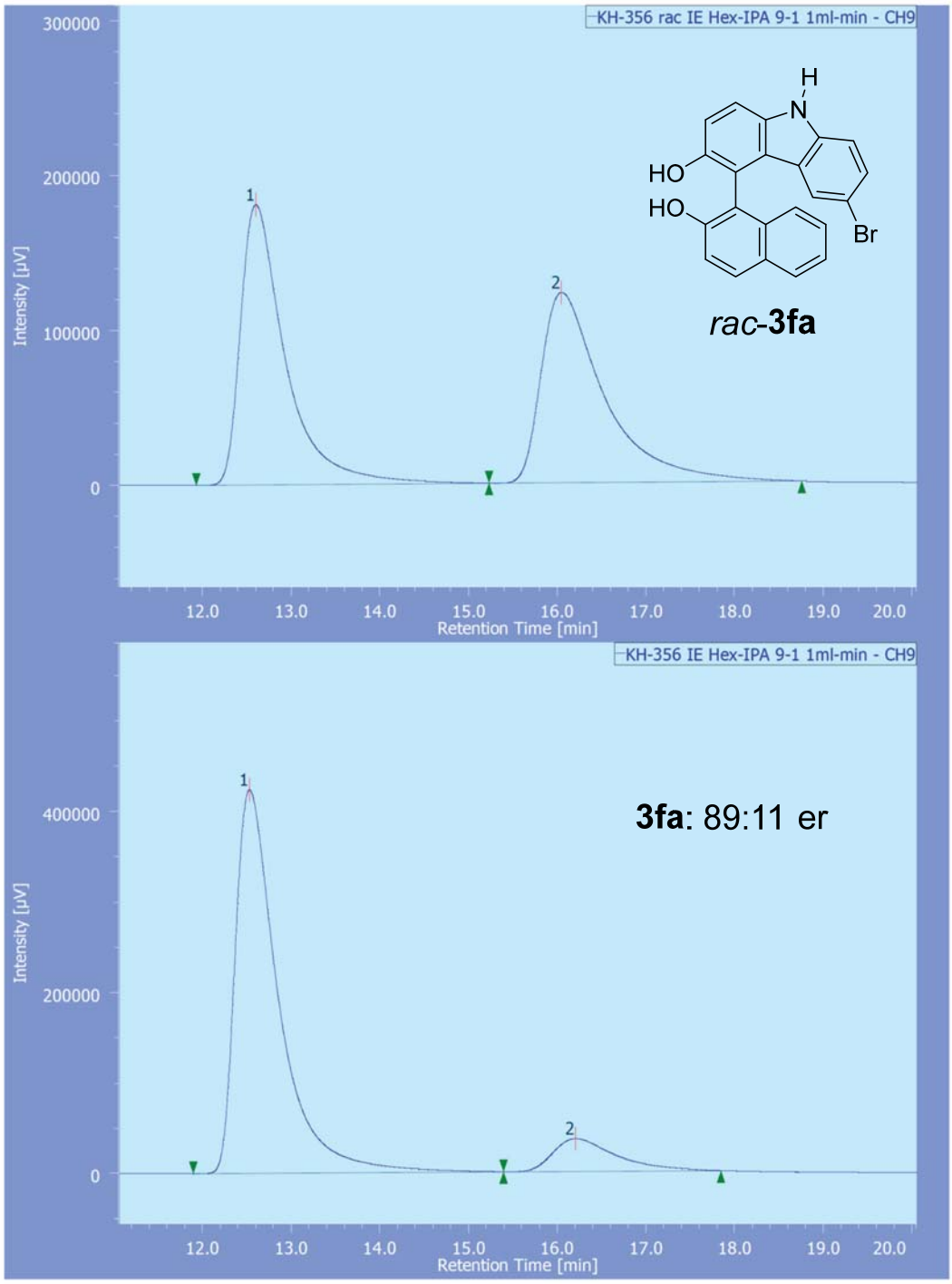
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	10.473	3642690	47514	48.754	55.579	N/A	446	2.469	1.332	
2	Unknown	9	16.122	3828944	37975	51.246	44.421	N/A	613	N/A	1.283	

Chromatogram Name KH-359 chiral IC-3 Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	10.455	8122936	107426	91.726	93.390	N/A	534	2.685	1.690	
2	Unknown	9	16.090	732687	7604	8.274	6.610	N/A	719	N/A	N/A	



Channel & Peak Information Table

Chromatogram Name KH-356 rac IE Hex-IPA 9-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

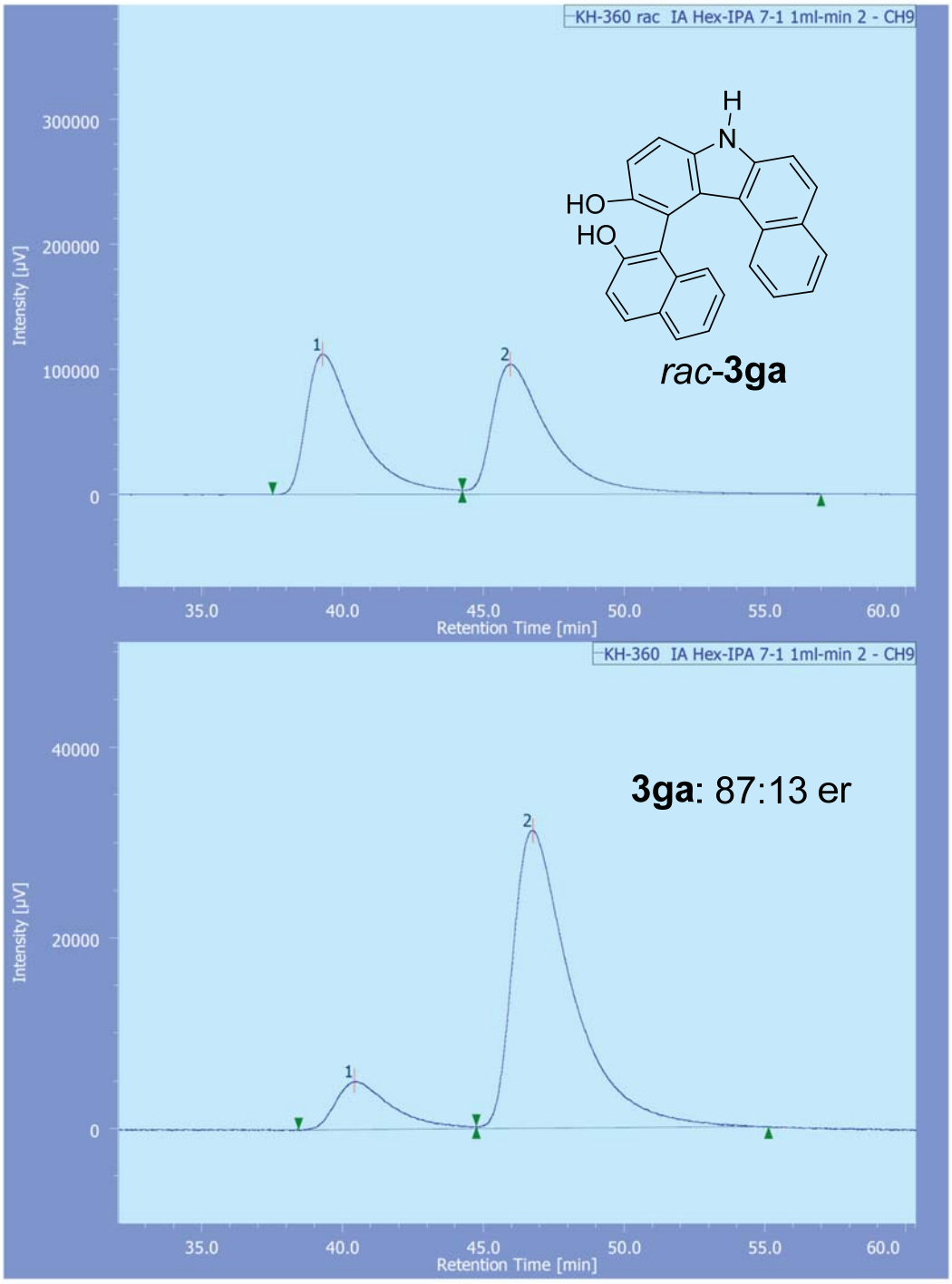
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	12.602	6383675	180837	51.052	59.520	N/A	3662	3.437	2.004	
2	Unknown	9	16.048	6120698	122989	48.948	40.480	N/A	2969	N/A	2.178	

Chromatogram Name KH-356 IE Hex-IPA 9-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	12.530	14443398	423861	88.921	92.072	N/A	3805	3.603	2.072	
2	Unknown	9	16.205	1799500	36495	11.079	7.928	N/A	2764	N/A	1.721	



Channel & Peak Information Table

Chromatogram Name KH-360 rac IA Hex-IPA 7-1 1ml-min 2-CH9

Sample Name

Channel Name 230.0nm

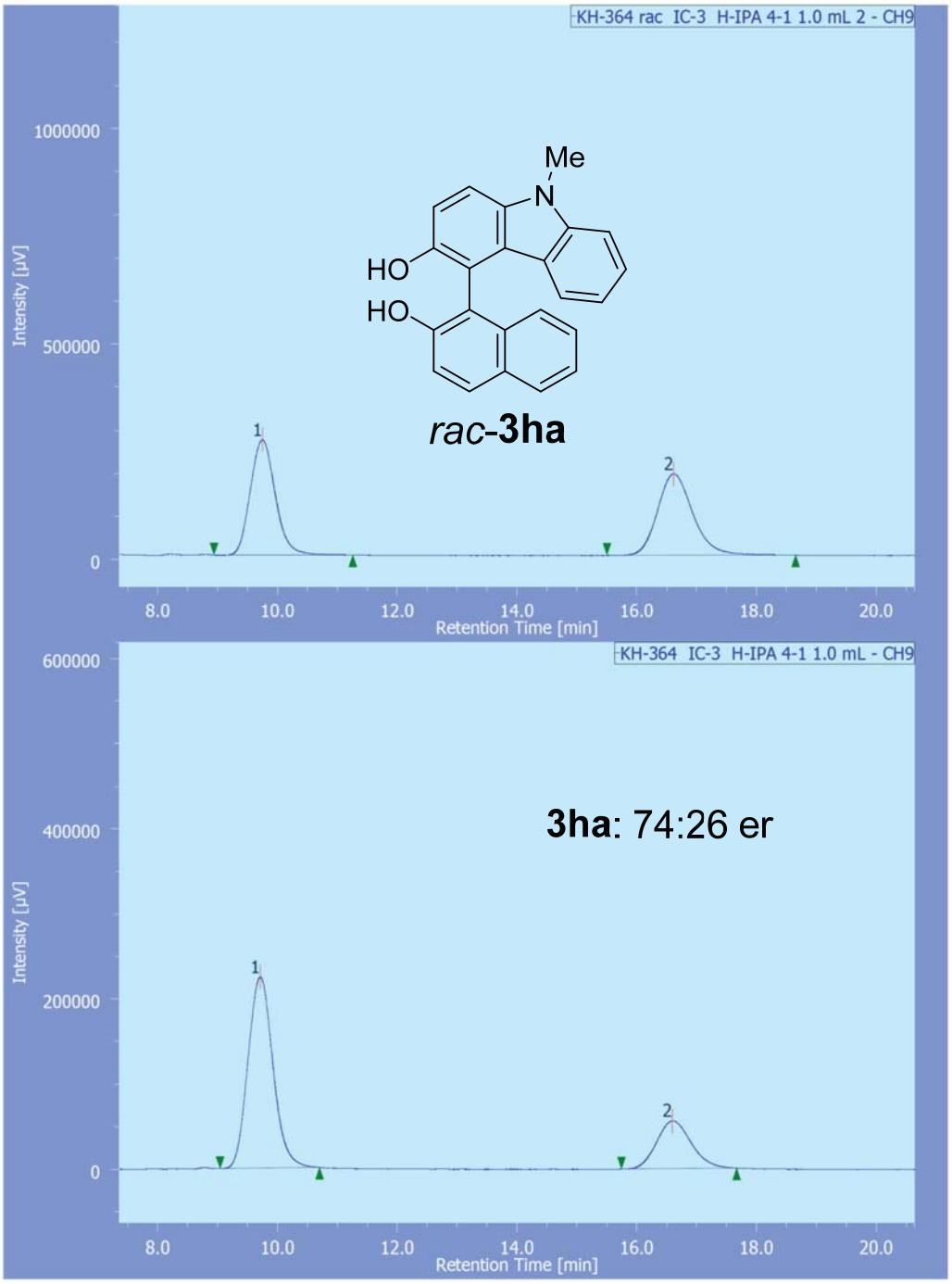
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	39.297	14247752	112080	48.704	51.924	N/A	2608	2.056	2.190	
2	Unknown	9	45.955	15005714	103773	51.296	48.076	N/A	2895	N/A	2.113	

Chromatogram Name KH-360 IA Hex-IPA 7-1 1ml-min 2-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	40.413	702134	5093	13.255	14.026	N/A	2223	1.821	1.927	
2	Unknown	9	46.757	4594843	31218	86.745	85.974	N/A	2765	N/A	2.018	



Channel & Peak Information Table

Chromatogram Name KH-364 rac IC-3 H-IPA 4-1 1.0 mL 2-CH9

Sample Name

Channel Name 230.0nm

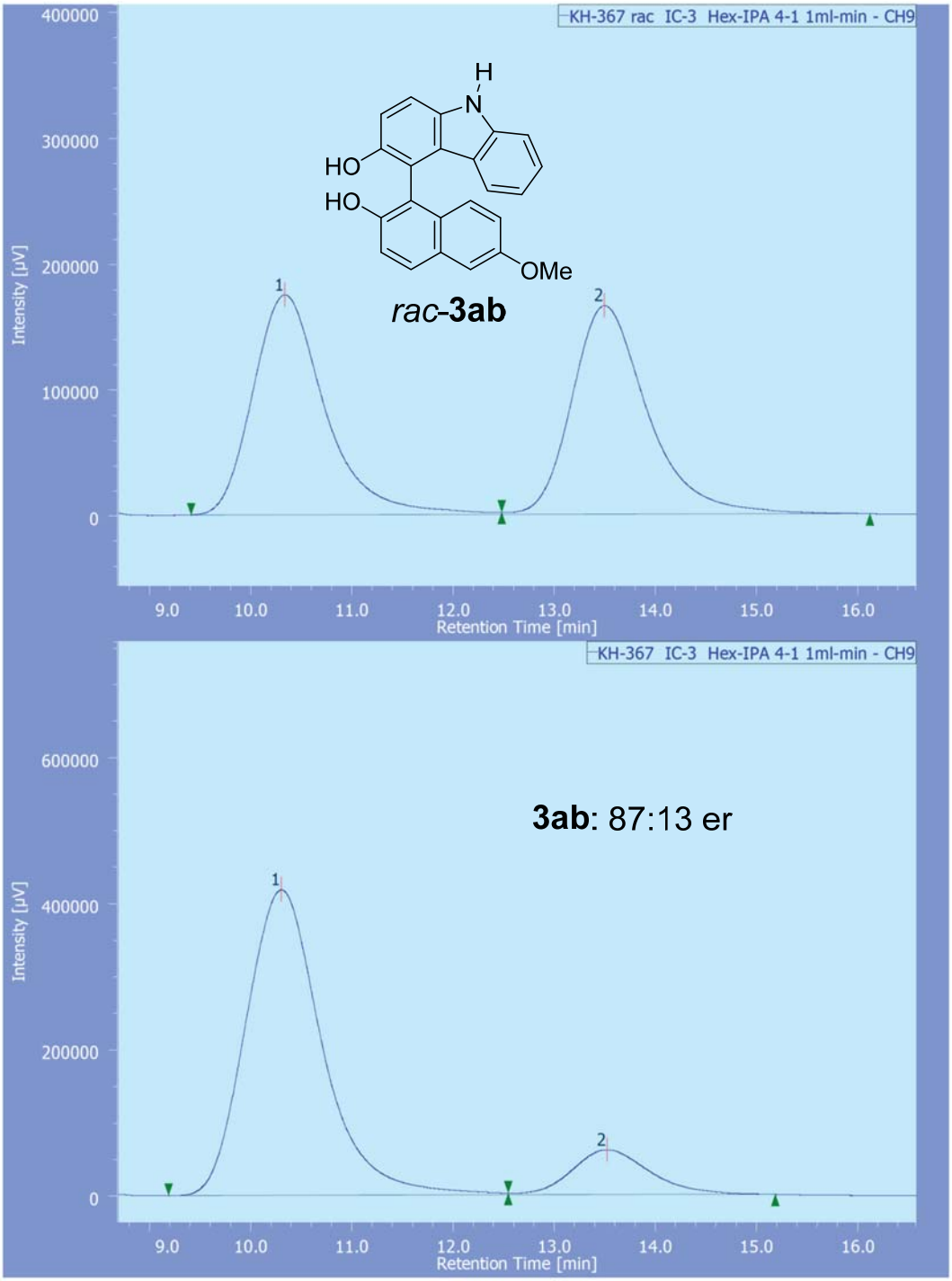
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	9.747	7762662	268283	49.950	58.610	N/A	2678	7.638	1.151	
2	Unknown	9	16.617	7778161	189463	50.050	41.390	N/A	4005	N/A	1.207	

Chromatogram Name KH-364 IC-3 H-IPA 4-1 1.0 mL-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	9.712	6532866	224812	74.189	79.929	N/A	2594	7.584	1.128	
2	Unknown	9	16.593	2272832	56454	25.811	20.071	N/A	3945	N/A	1.163	



Channel & Peak Information Table

Chromatogram Name KH-367 rac IC-3 Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

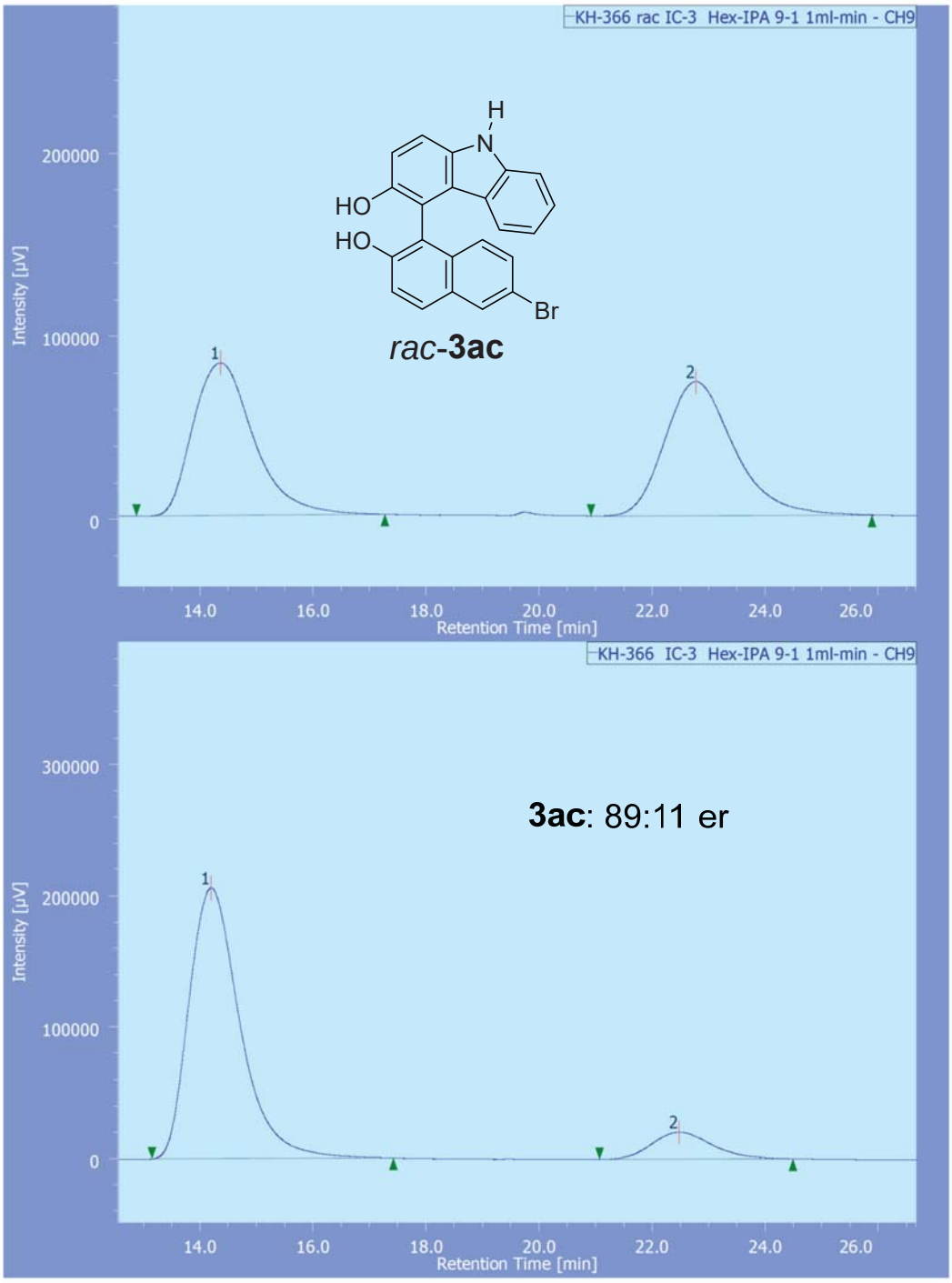
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	10.335	8673855	174977	50.100	51.331	N/A	1114	2.492	1.357	
2	Unknown	9	13.493	8639185	165905	49.900	48.669	N/A	1715	N/A	1.348	

Chromatogram Name KH-367 IC-3 Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	10.302	21973160	418929	86.879	87.166	N/A	959	2.386	1.272	
2	Unknown	9	13.515	3318411	61681	13.121	12.834	N/A	1557	N/A	1.184	



Channel & Peak Information Table

Chromatogram Name KH-366 rac IC-3 Hex-IPA 9-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

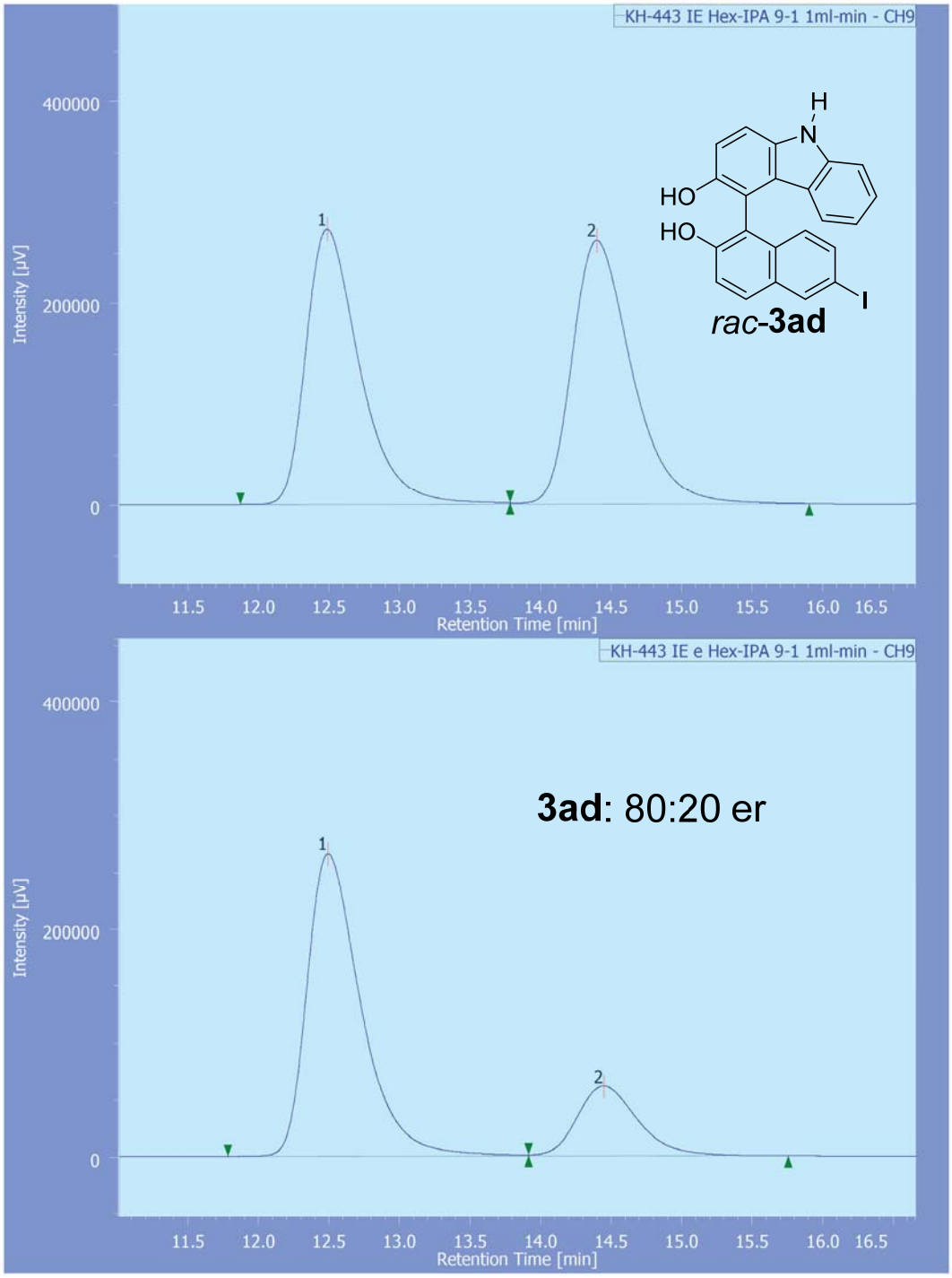
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	14.363	6258941	83488	49.991	53.290	N/A	873	4.072	1.364	
2	Unknown	9	22.773	6261232	73180	50.009	46.710	N/A	1718	N/A	1.281	

Chromatogram Name KH-366 IC-3 Hex-IPA 9-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	14.198	12926535	205902	89.197	90.791	N/A	1250	4.646	1.401	
2	Unknown	9	22.468	1565564	20884	10.803	9.209	N/A	2095	N/A	1.225	



Channel & Peak Information Table

Chromatogram Name KH-443 IE Hex-IPA 9-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

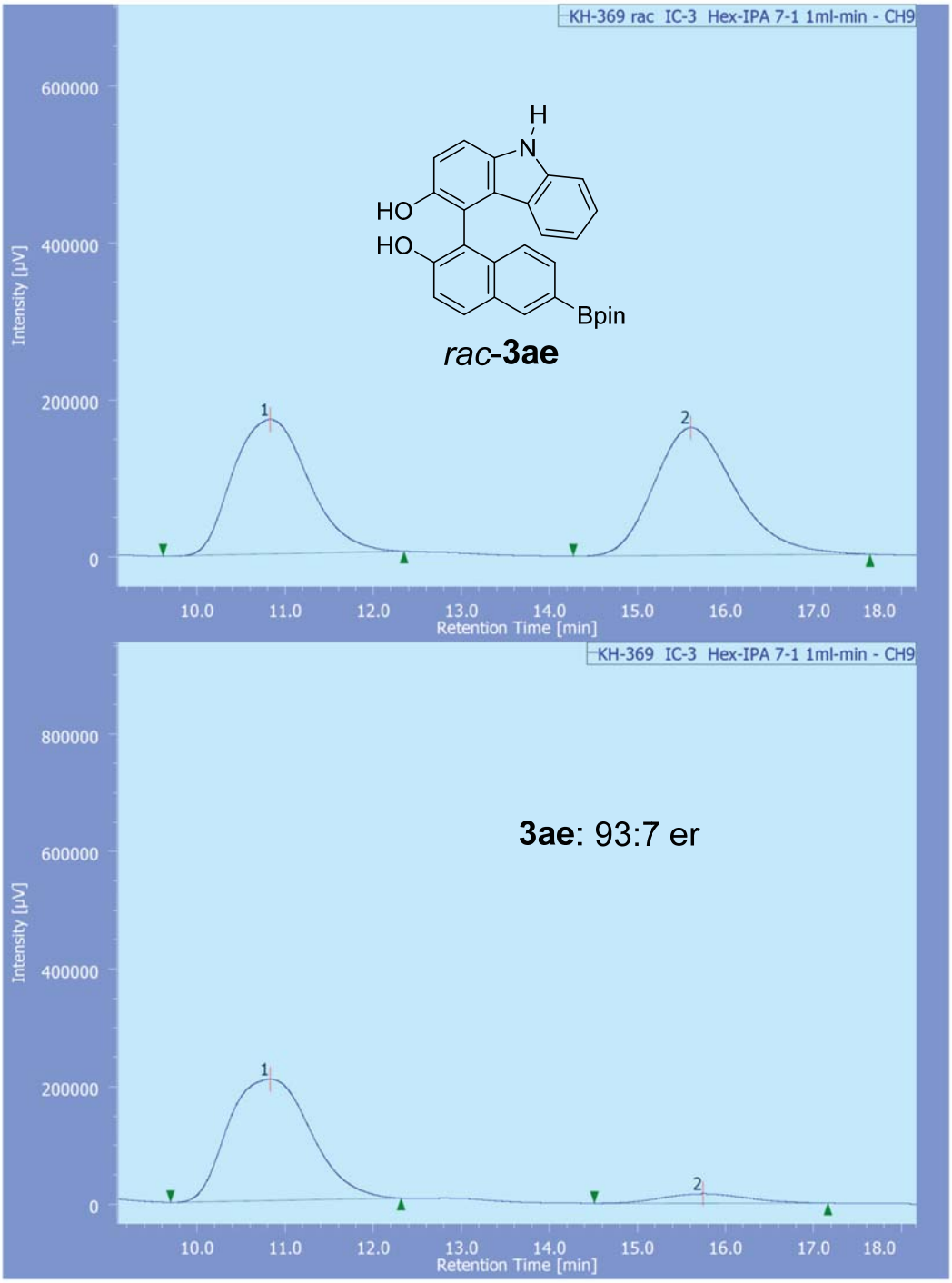
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	12.487	7223054	272612	48.496	51.061	N/A	5533	2.694	1.508	
2	Unknown	9	14.398	7670931	261284	51.504	48.939	N/A	5873	N/A	1.392	

Chromatogram Name KH-443 IE e Hex-IPA 9-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	12.492	7044239	265934	79.614	81.391	N/A	5585	2.742	1.526	
2	Unknown	9	14.448	1803769	60801	20.386	18.609	N/A	5747	N/A	1.265	



Channel & Peak Information Table

Chromatogram Name KH-369 rac IC-3 Hex-IPA 7-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

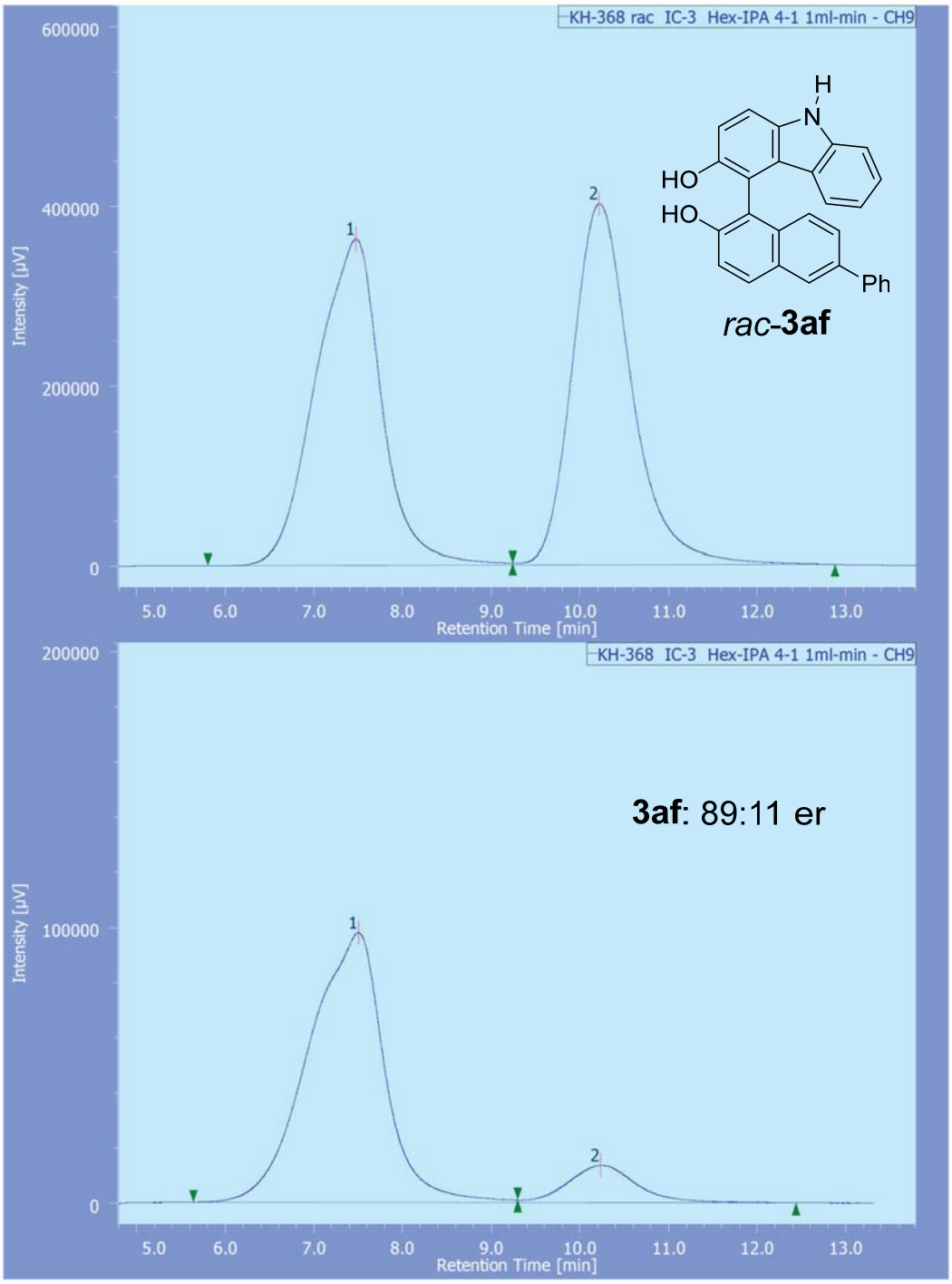
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	10.827	10186073	171557	49.350	51.266	N/A	726	2.929	1.187	
2	Unknown	9	15.602	10454532	163081	50.650	48.734	N/A	1409	N/A	1.256	

Chromatogram Name KH-369 IC-3 Hex-IPA 7-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	10.828	13516000	206962	92.571	92.746	N/A	592	2.742	1.159	
2	Unknown	9	15.738	1084613	16188	7.429	7.254	N/A	1208	N/A	1.117	



Channel & Peak Information Table

Chromatogram Name KH-368 rac IC-3 Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 255.0nm

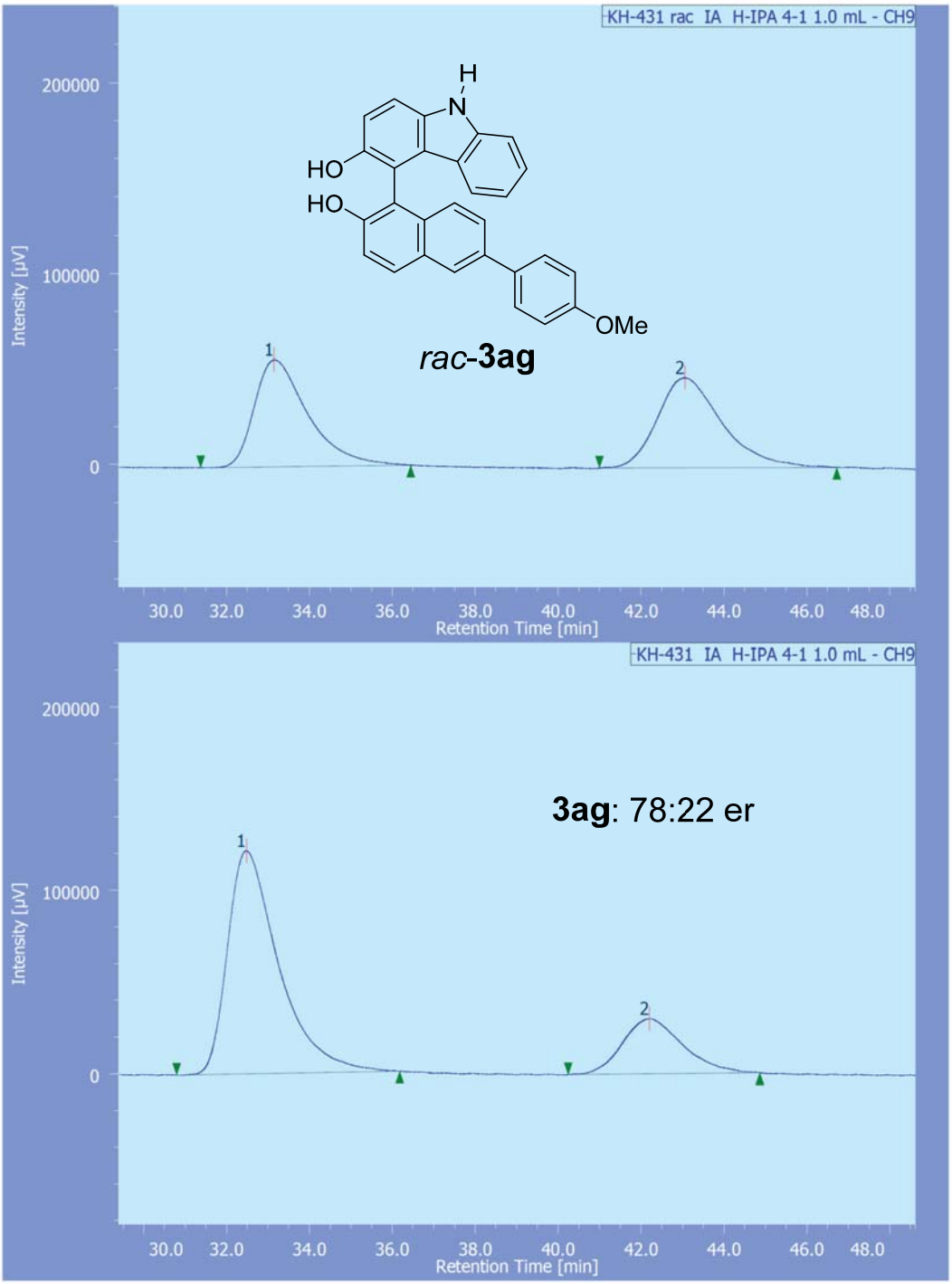
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	7.473	18662037	363033	50.039	47.490	N/A	487	2.176	0.941	
2	Unknown	9	10.217	18633066	401411	49.961	52.510	N/A	1214	N/A	1.270	

Chromatogram Name KH-368 IC-3 Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 255.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	7.502	5837622	97873	88.916	87.852	N/A	370	1.880	0.865	
2	Unknown	9	10.232	727690	13534	11.084	12.148	N/A	915	N/A	N/A	



Channel & Peak Information Table

Chromatogram Name KH-431 rac IA H-IPA 4-1 1.0 mL-CH9

Sample Name

Channel Name 220.0nm

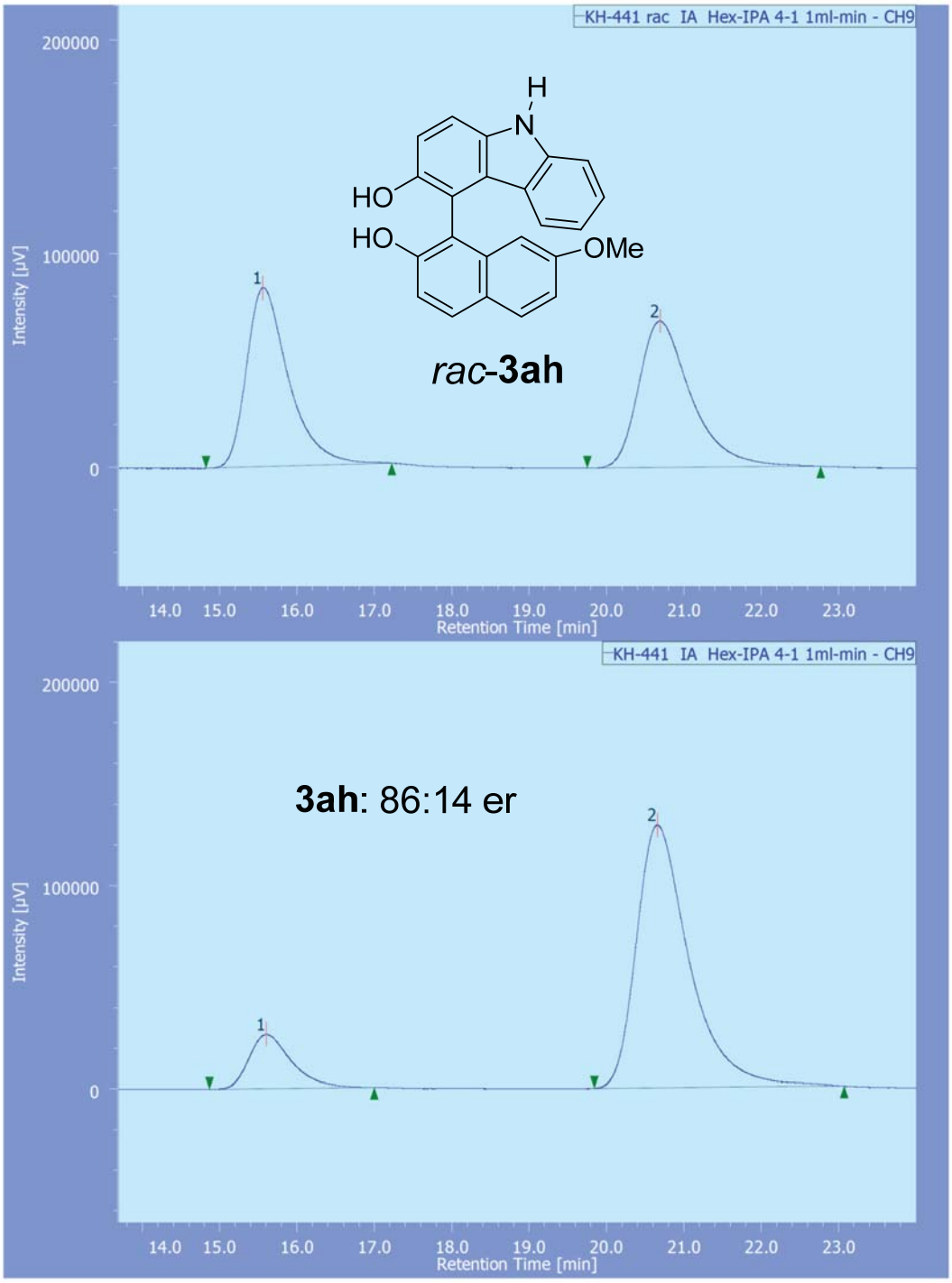
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	33.142	5028354	56265	49.458	54.281	N/A	3488	3.997	1.636	
2	Unknown	9	43.058	5138628	47389	50.542	45.719	N/A	3977	N/A	1.375	

Chromatogram Name KH-431 IA H-IPA 4-1 1.0 mL-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	32.478	10460520	121762	77.556	80.195	N/A	3686	4.106	1.704	
2	Unknown	9	42.200	3027187	30071	22.444	19.805	N/A	4188	N/A	1.247	



Channel & Peak Information Table

Chromatogram Name KH-441 rac IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

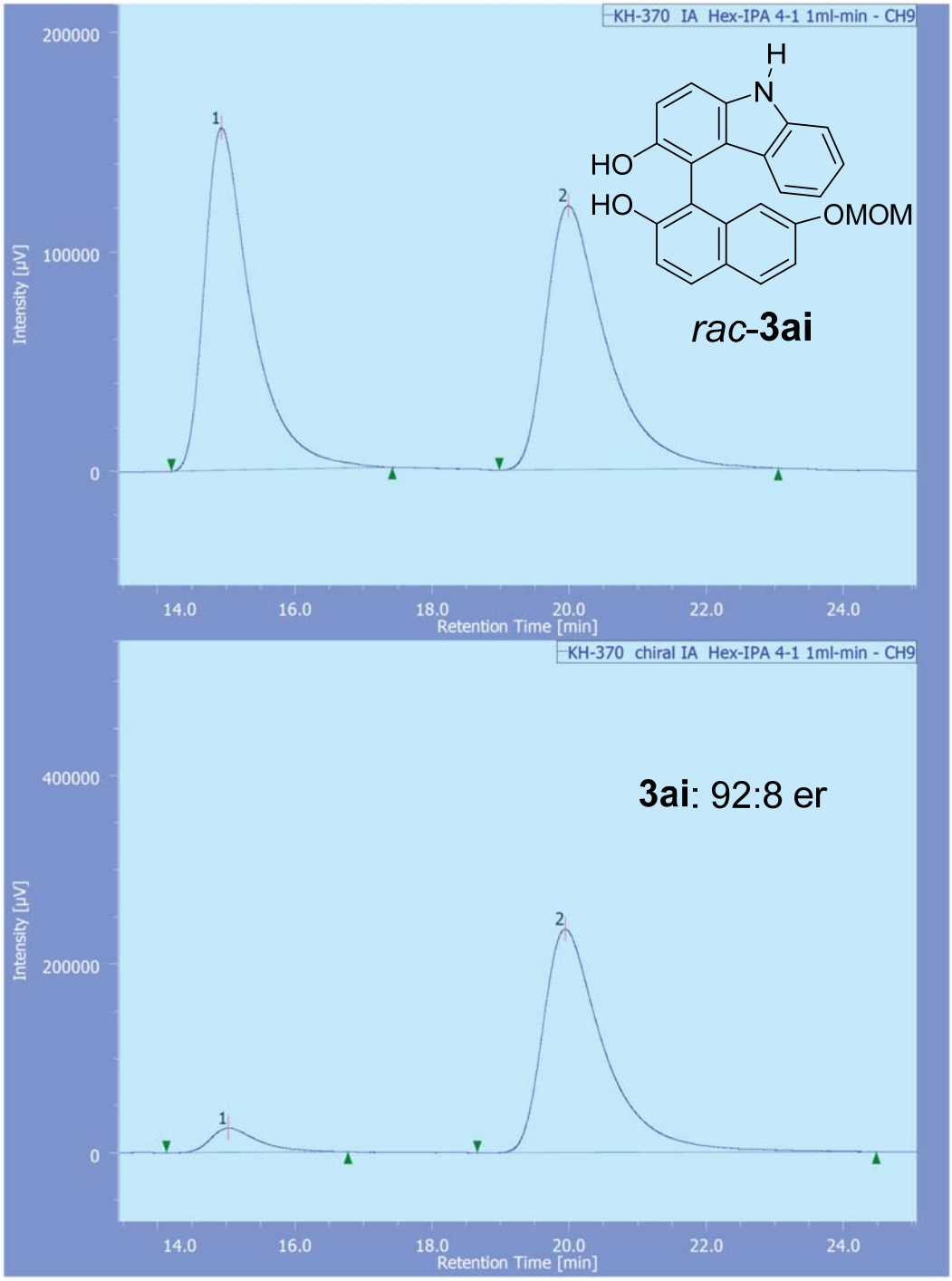
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	15.560	3167842	83932	49.446	55.046	N/A	4340	4.839	1.503	
2	Unknown	9	20.690	3238848	68545	50.554	44.954	N/A	4908	N/A	1.461	

Chromatogram Name KH-441 IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	15.602	1019861	26660	14.244	17.107	N/A	4165	4.730	1.442	
2	Unknown	9	20.655	6139922	129181	85.756	82.893	N/A	4942	N/A	1.530	



Channel & Peak Information Table

Chromatogram Name KH-370 IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

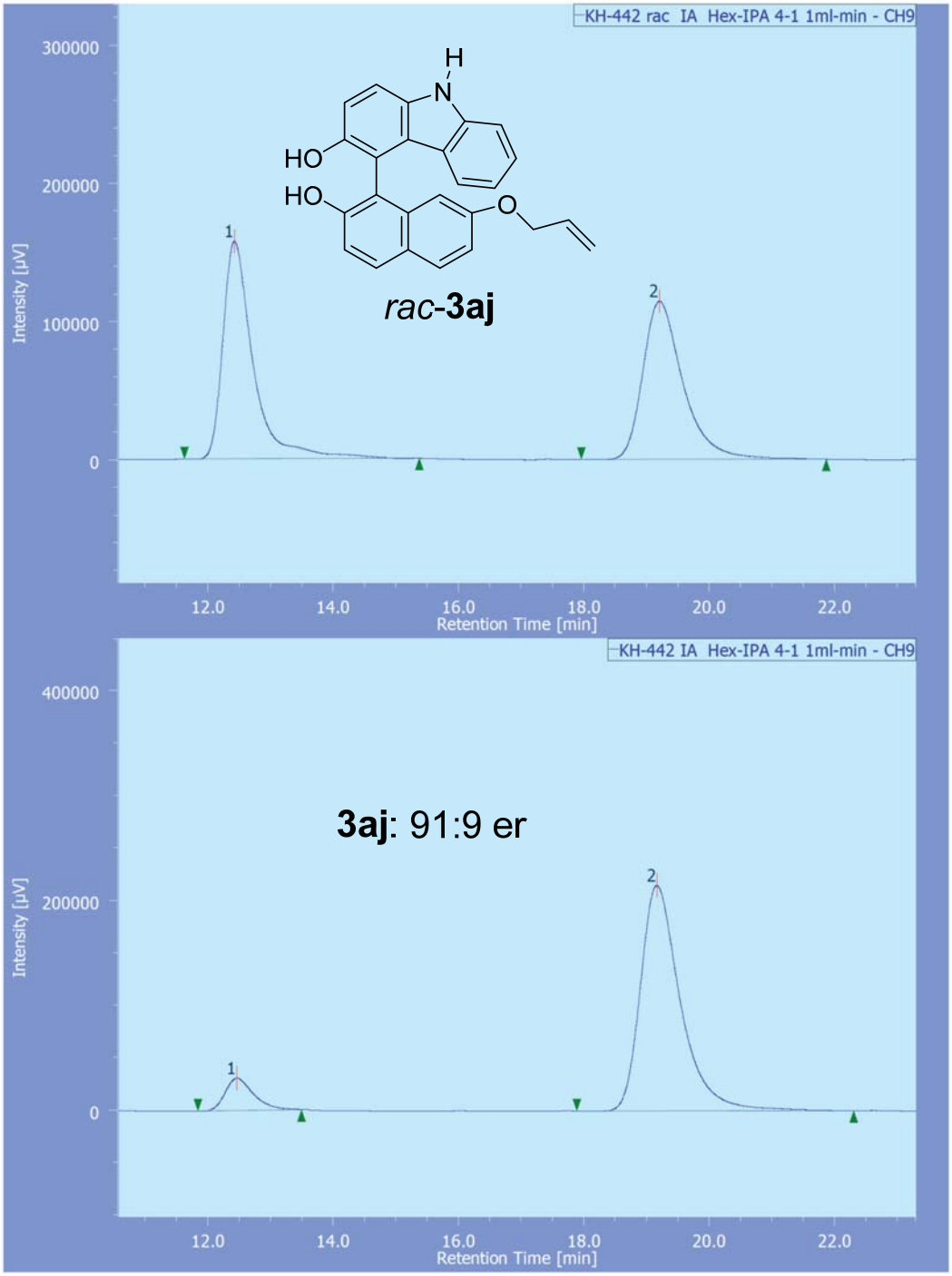
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	14.928	7413099	156085	50.008	56.497	N/A	2688	3.807	1.767	
2	Unknown	9	19.988	7410758	120185	49.992	43.503	N/A	2791	N/A	1.713	

Chromatogram Name KH-370 chiral IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	15.032	1266546	26107	7.916	9.920	N/A	2487	3.640	1.647	
2	Unknown	9	19.938	14733990	237069	92.084	90.080	N/A	2836	N/A	1.779	



Channel & Peak Information Table

Chromatogram Name KH-442 rac IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

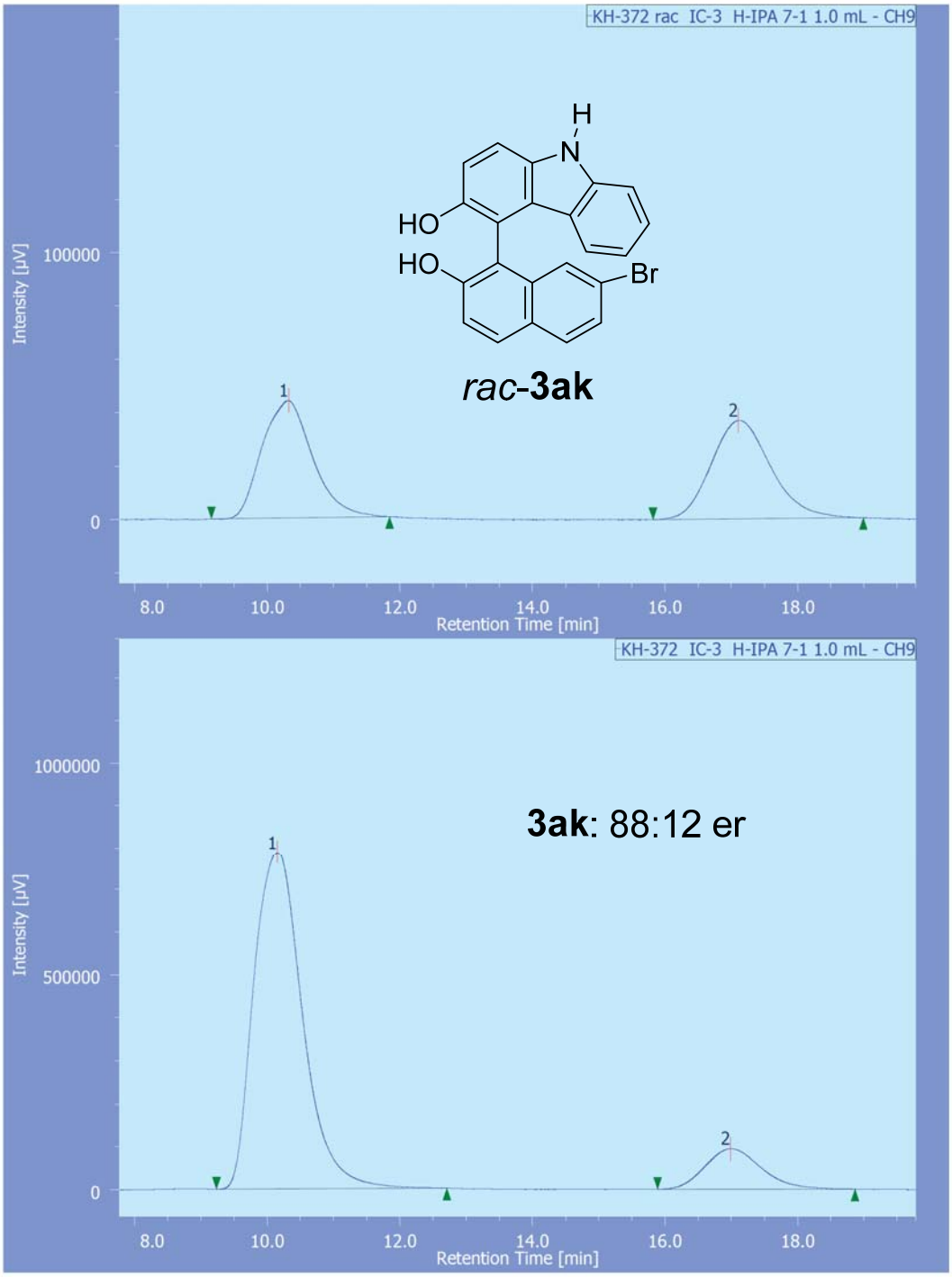
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	12.425	5290296	157474	50.952	57.931	N/A	4000	7.185	1.838	
2	Unknown	9	19.202	5092577	114356	49.048	42.069	N/A	4827	N/A	1.427	

Chromatogram Name KH-442 IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	12.460	971216	30801	9.260	12.539	N/A	3864	7.075	1.400	
2	Unknown	9	19.158	9517110	214838	90.740	87.461	N/A	4882	N/A	1.452	



Channel & Peak Information Table

Chromatogram Name KH-372 rac IC-3 H-IPA 7-1 1.0 mL-CH9

Sample Name

Channel Name 230.0nm

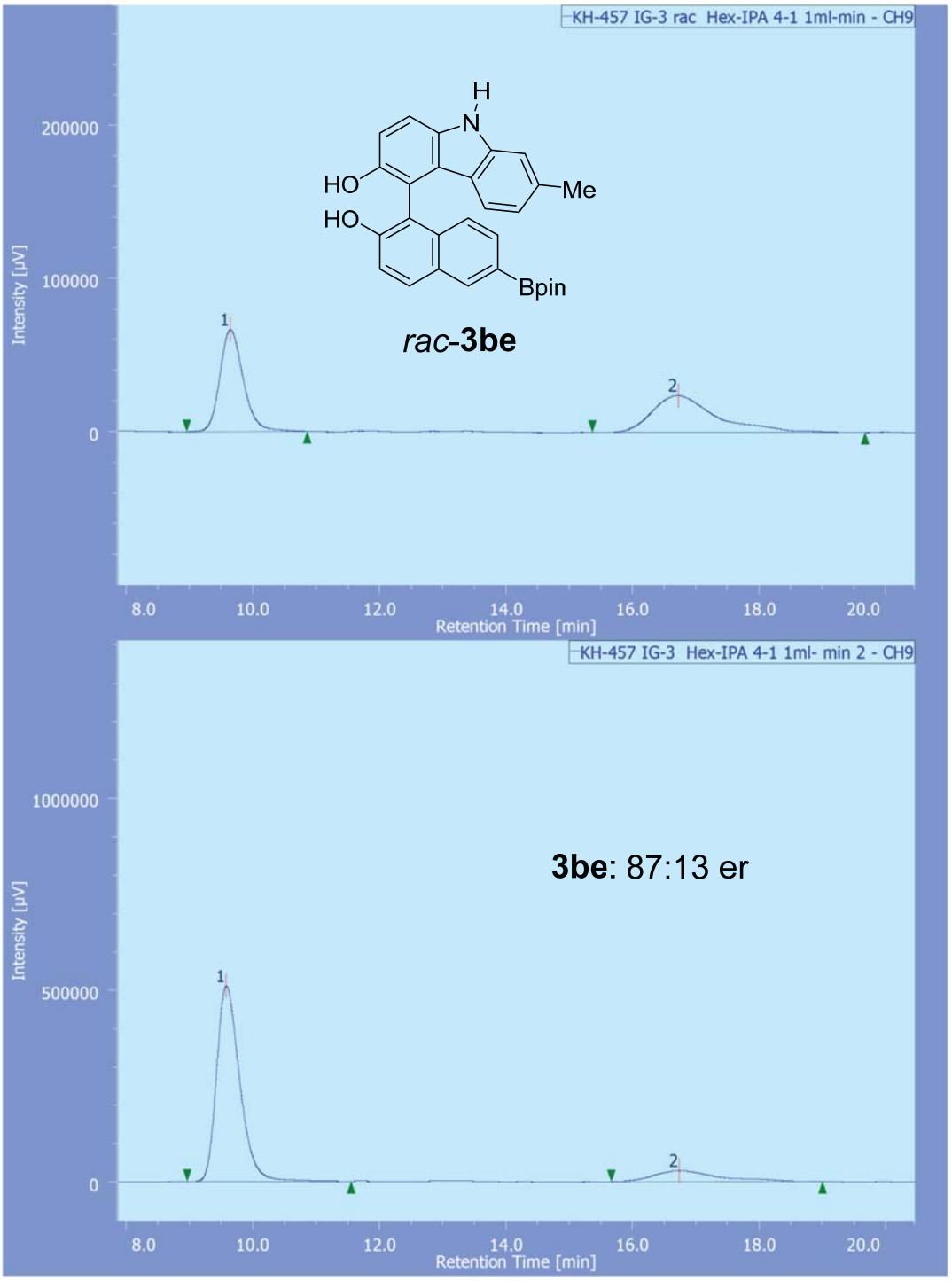
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	10.323	2315904	43922	50.227	54.391	N/A	857	4.477	1.157	
2	Unknown	9	17.103	2294927	36830	49.773	45.609	N/A	1769	N/A	1.200	

Chromatogram Name KH-372 IC-3 H-IPA 7-1 1.0 mL-CH9

Sample Name

Channel Name 230.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	10.153	40057814	787076	87.720	89.284	N/A	913	4.738	1.278	
2	Unknown	9	16.987	5607952	94468	12.280	10.716	N/A	1926	N/A	1.208	



Channel & Peak Information Table

Chromatogram Name KH-457 IG-3 rac Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

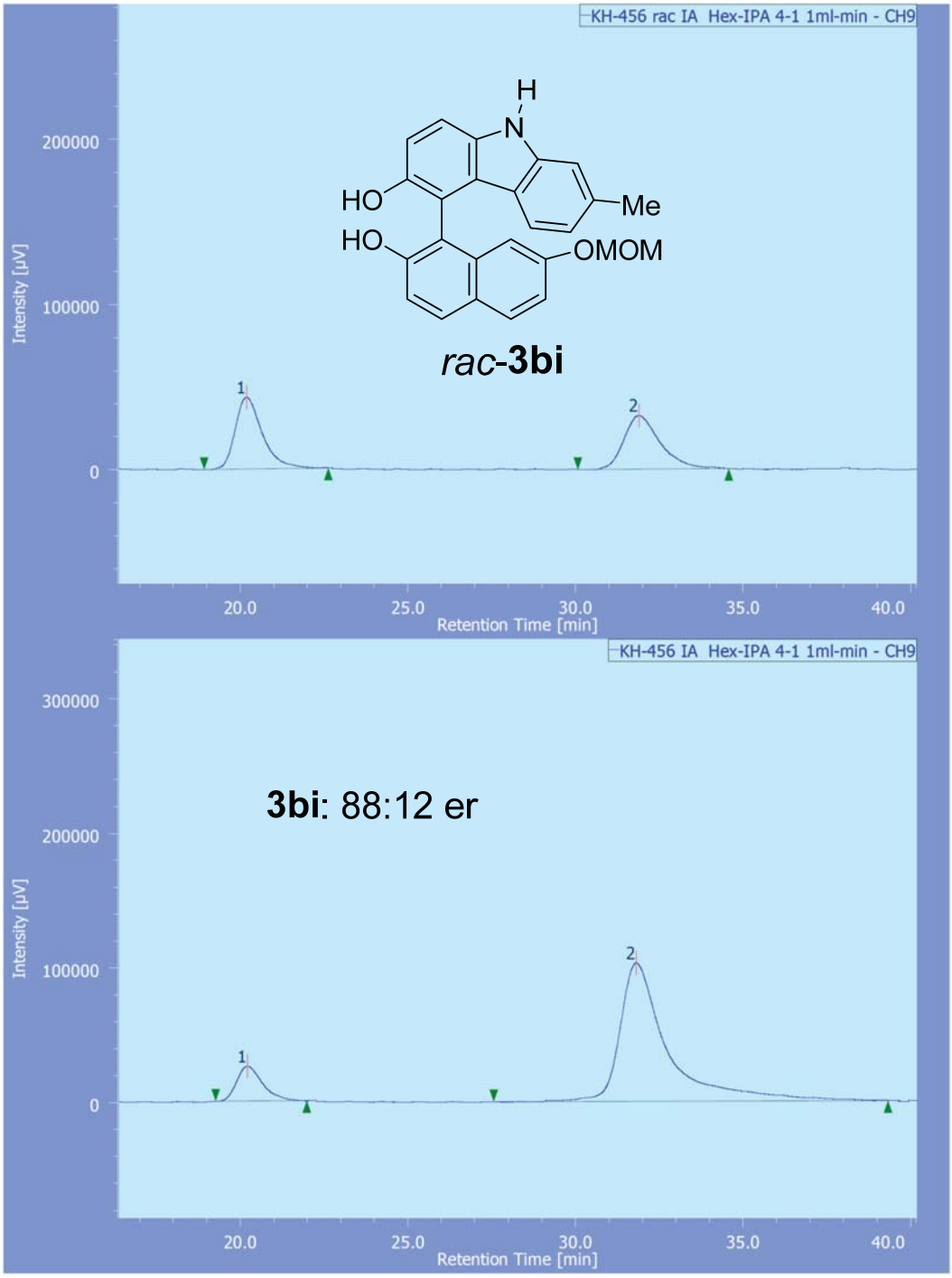
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	9.645	1710838	66590	49.925	73.479	N/A	3475	6.083	1.212	
2	Unknown	9	16.715	1715978	24034	50.075	26.521	N/A	1591	N/A	1.564	

Chromatogram Name KH-457 IG-3 Hex-IPA 4-1 1ml- min 2-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	9.580	13005867	509936	86.521	94.717	N/A	3432	6.102	1.335	
2	Unknown	9	16.732	2026171	28445	13.479	5.283	N/A	1557	N/A	1.504	



Channel & Peak Information Table

Chromatogram Name KH-456 rac IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

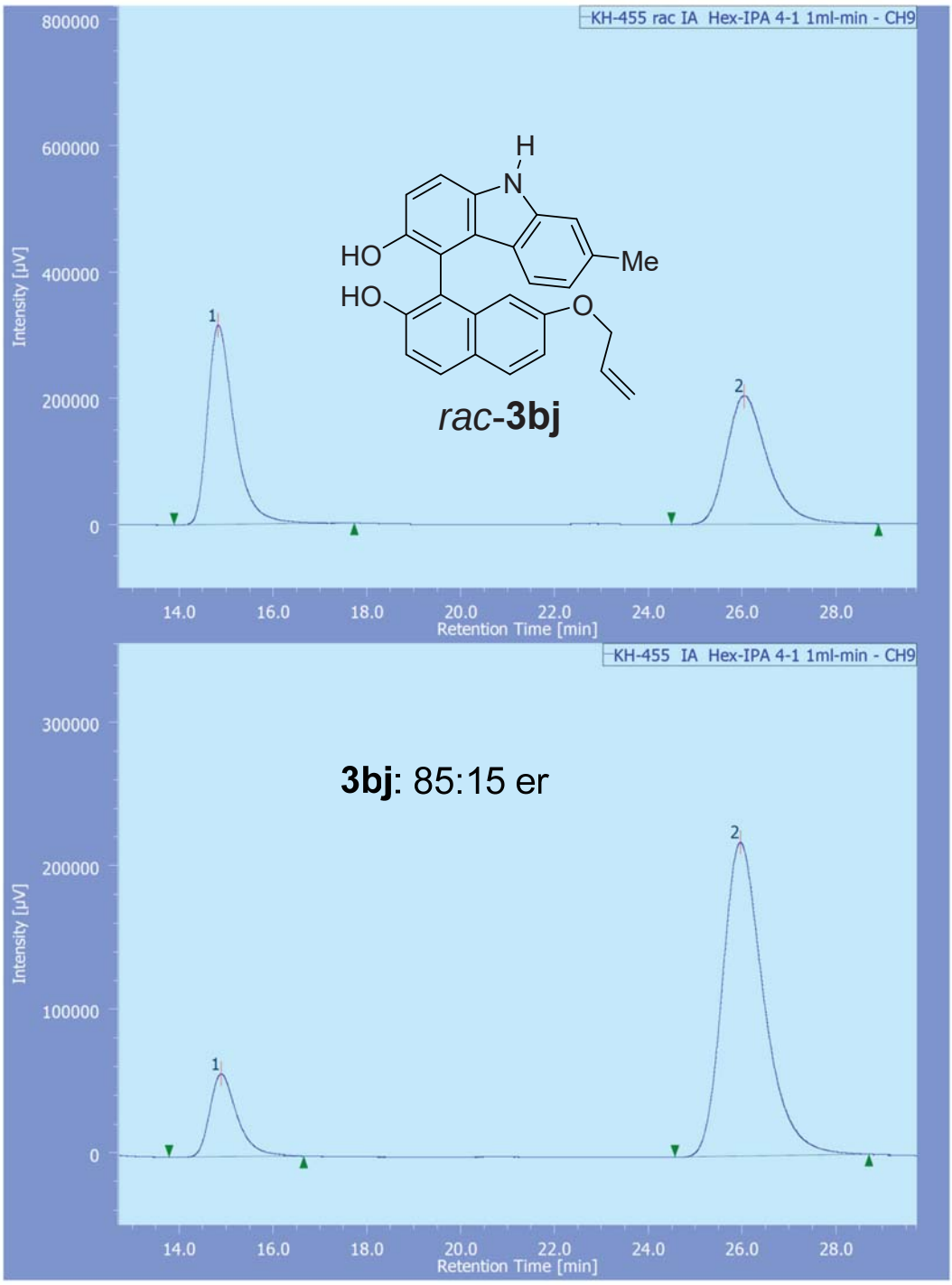
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	20.187	2445404	43579	50.070	57.350	N/A	3304	7.124	1.459	
2	Unknown	9	31.890	2438615	32408	49.930	42.650	N/A	4558	N/A	1.375	

Chromatogram Name KH-456 IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	20.217	1411239	26070	12.165	20.245	N/A	3447	6.788	1.402	
2	Unknown	9	31.812	10189133	102704	87.835	79.755	N/A	3861	N/A	2.232	



Channel & Peak Information Table

Chromatogram Name KH-455 rac IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

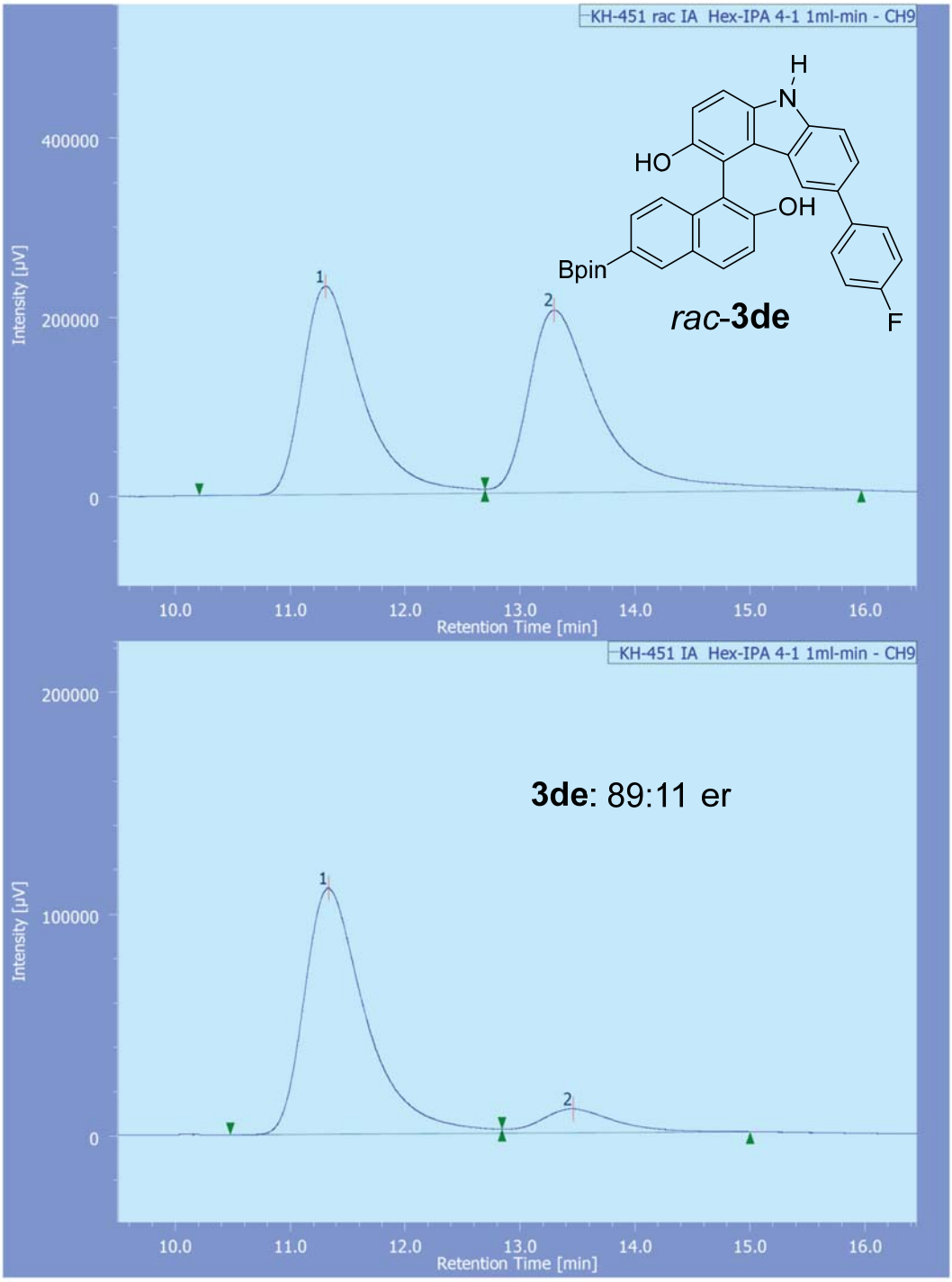
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	14.833	12569690	315278	49.946	60.768	N/A	3585	8.787	1.488	
2	Unknown	9	26.042	12597027	203541	50.054	39.232	N/A	4419	N/A	1.325	

Chromatogram Name KH-455 IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	14.897	2354697	58000	14.919	20.968	N/A	3430	8.613	1.431	
2	Unknown	9	25.958	13428185	218607	85.081	79.032	N/A	4440	N/A	1.321	



Channel & Peak Information Table

Chromatogram Name KH-451 rac IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

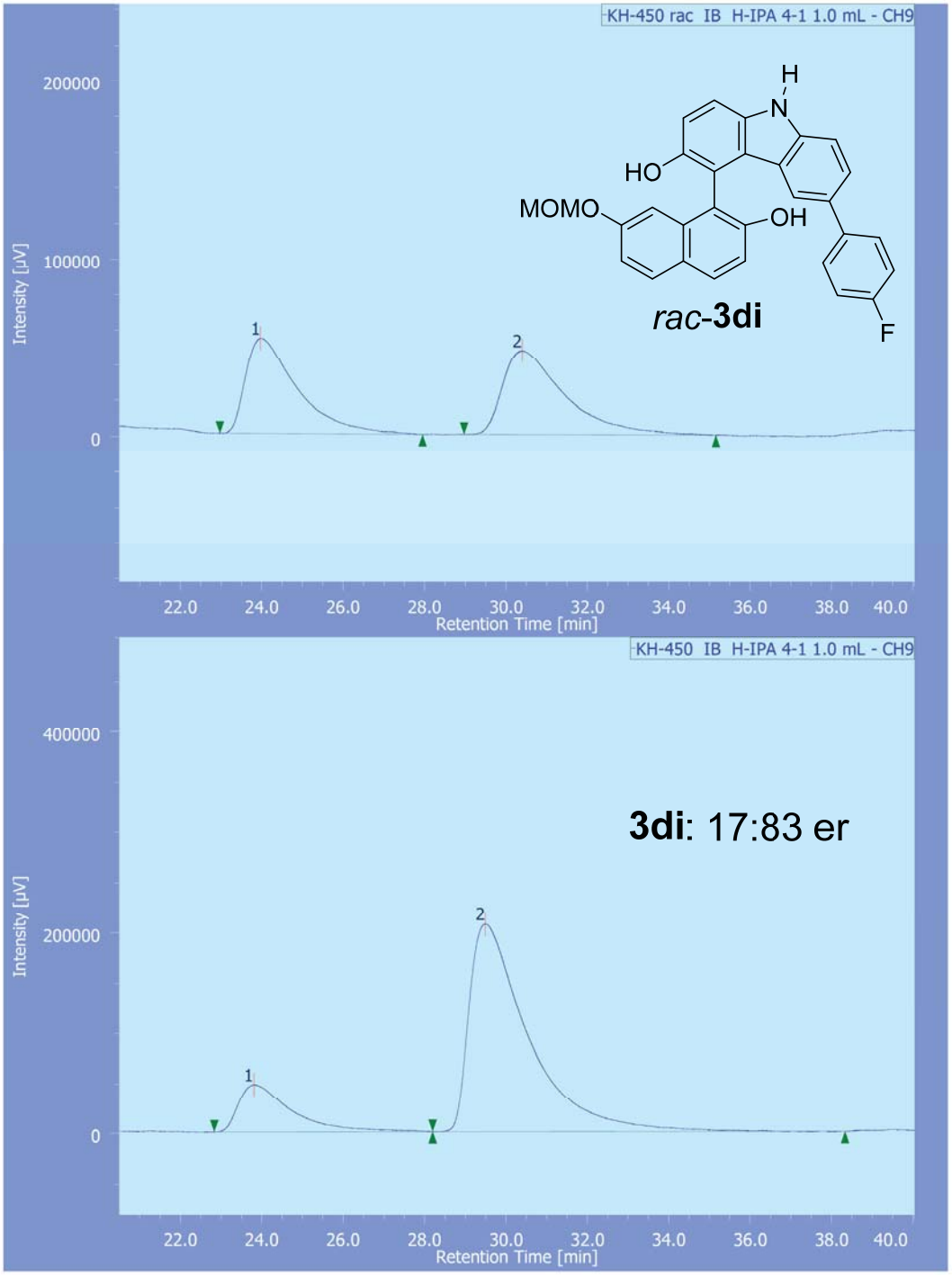
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	11.307	8361203	232324	48.539	53.310	N/A	2569	2.084	1.624	
2	Unknown	9	13.295	8864585	203476	51.461	46.690	N/A	2714	N/A	1.892	

Chromatogram Name KH-451 IA Hex-IPA 4-1 1ml-min-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	11.330	4135931	110807	89.499	91.155	N/A	2433	2.091	1.630	
2	Unknown	9	13.458	485293	10752	10.501	8.845	N/A	2302	N/A	N/A	



Channel & Peak Information Table

Chromatogram Name KH-450 rac IB H-IPA 4-1 1.0 mL-CH9

Sample Name

Channel Name 220.0nm

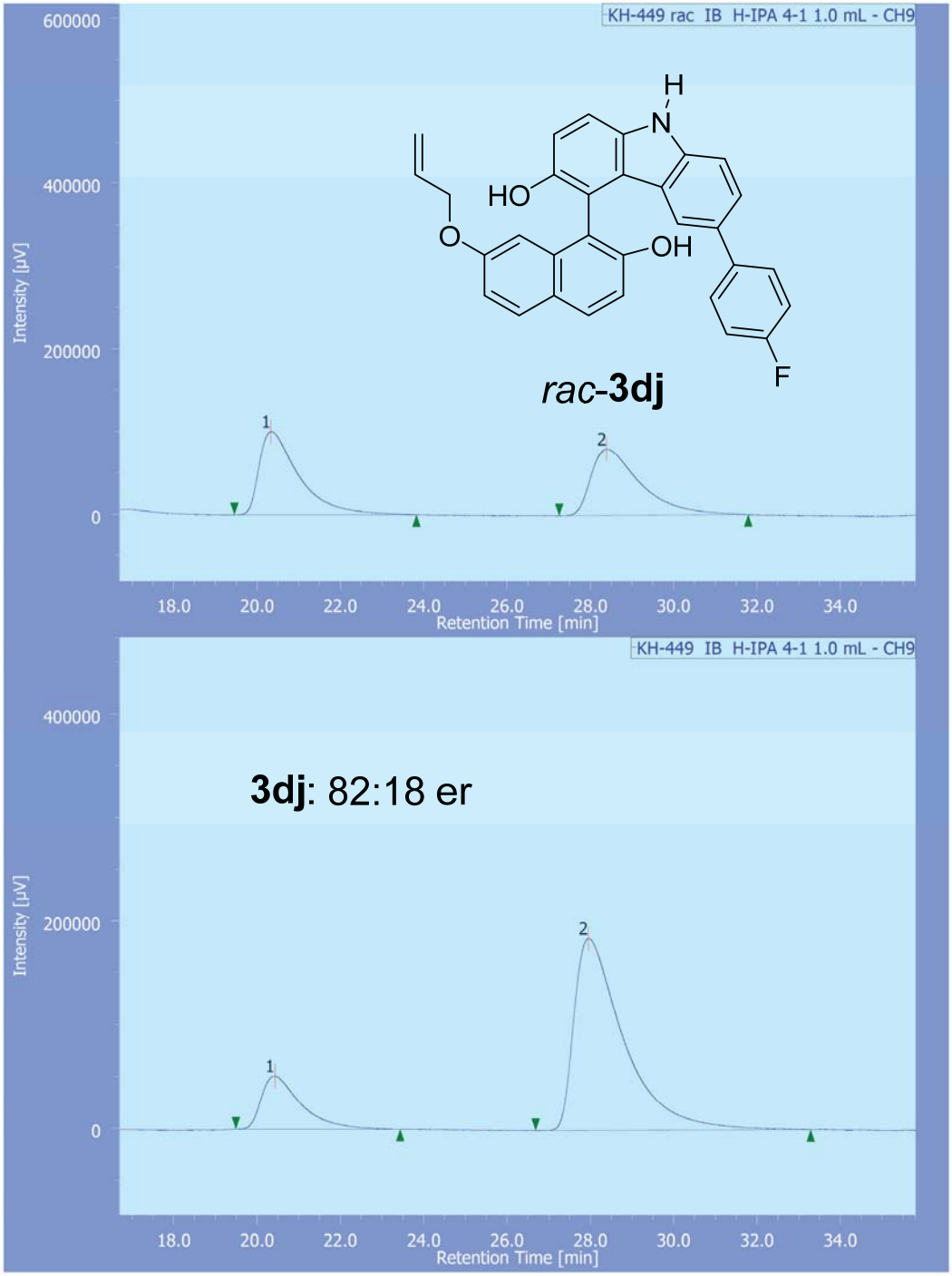
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	23.963	4717836	53748	48.692	53.169	N/A	1976	2.732	2.357	
2	Unknown	9	30.402	4971377	47341	51.308	46.831	N/A	2241	N/A	2.154	

Chromatogram Name KH-450 IB H-IPA 4-1 1.0 mL-CH9

Sample Name

Channel Name 220.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	23.807	4343980	48228	17.090	18.873	N/A	1985	2.501	2.501	
2	Unknown	9	29.490	21074535	207315	82.910	81.127	N/A	2376	N/A	2.700	



Channel & Peak Information Table

Chromatogram Name KH-449 rac IB H-IPA 4-1 1.0 mL-CH9

Sample Name

Channel Name 220.0nm

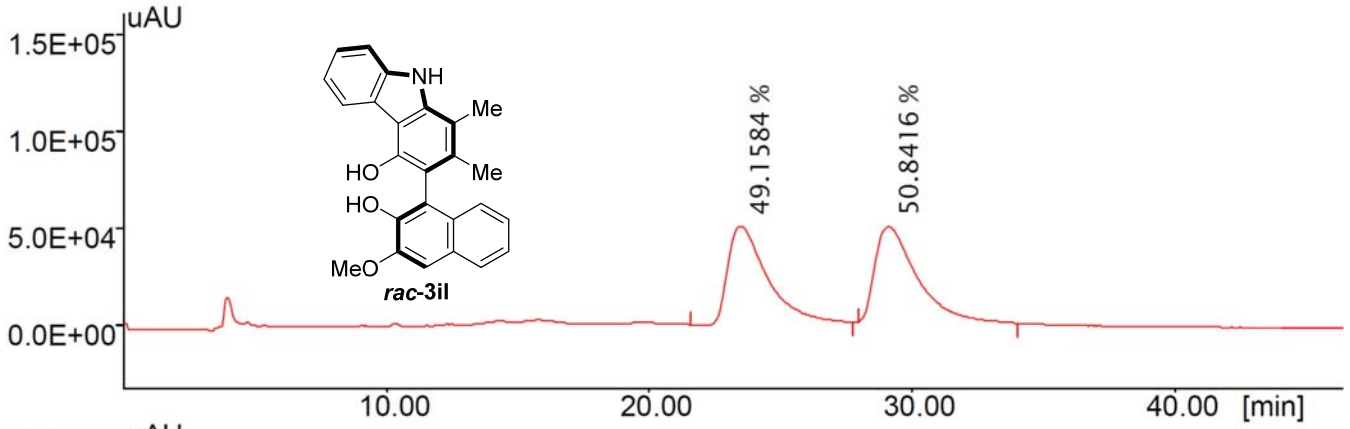
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	20.328	6672880	100715	50.294	55.623	N/A	2567	4.437	2.305	
2	Unknown	9	28.390	6594982	80354	49.706	44.377	N/A	3104	N/A	2.054	

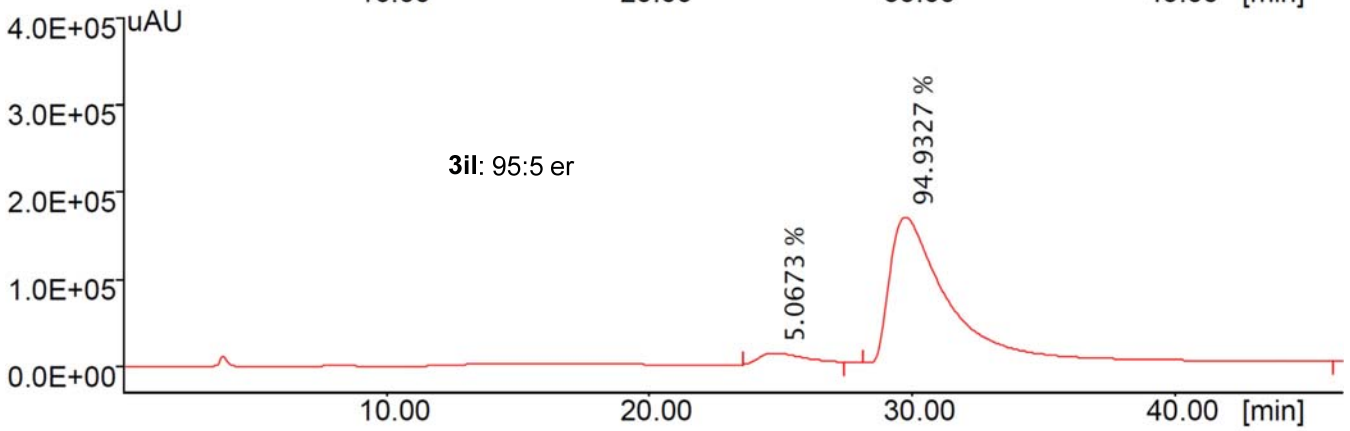
Chromatogram Name KH-449 IB H-IPA 4-1 1.0 mL-CH9

Sample Name

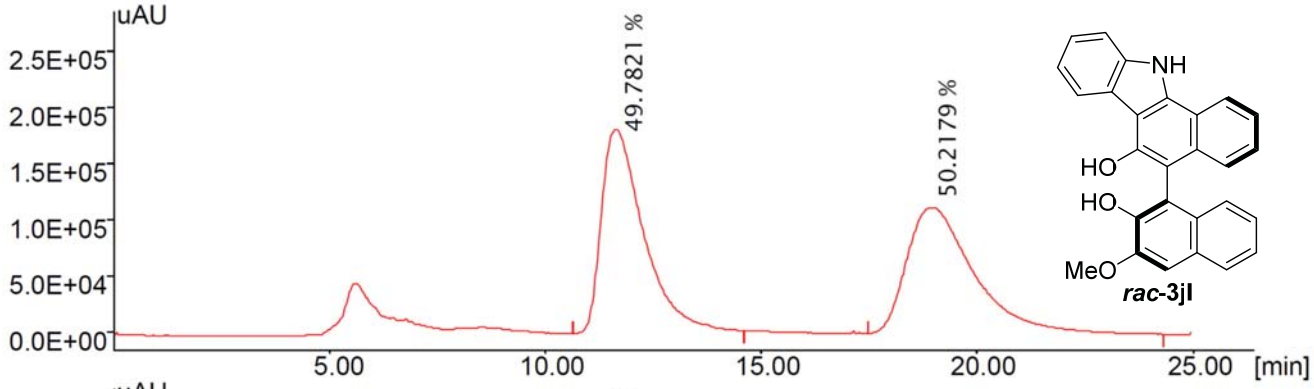
Channel Name 220.0nm

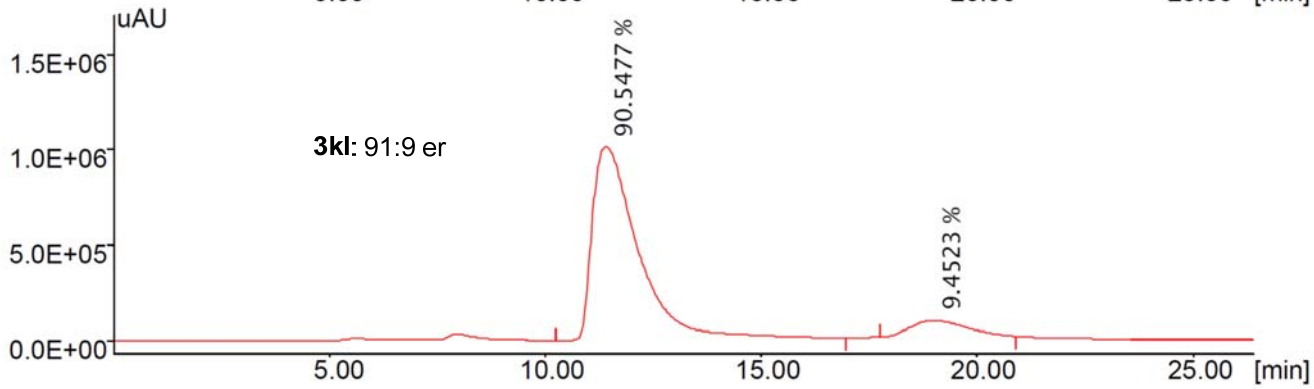
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	20.427	3383606	51457	17.959	21.757	N/A	2528	4.131	1.953	
2	Unknown	9	27.947	15457285	185046	82.041	78.243	N/A	3047	N/A	2.397	



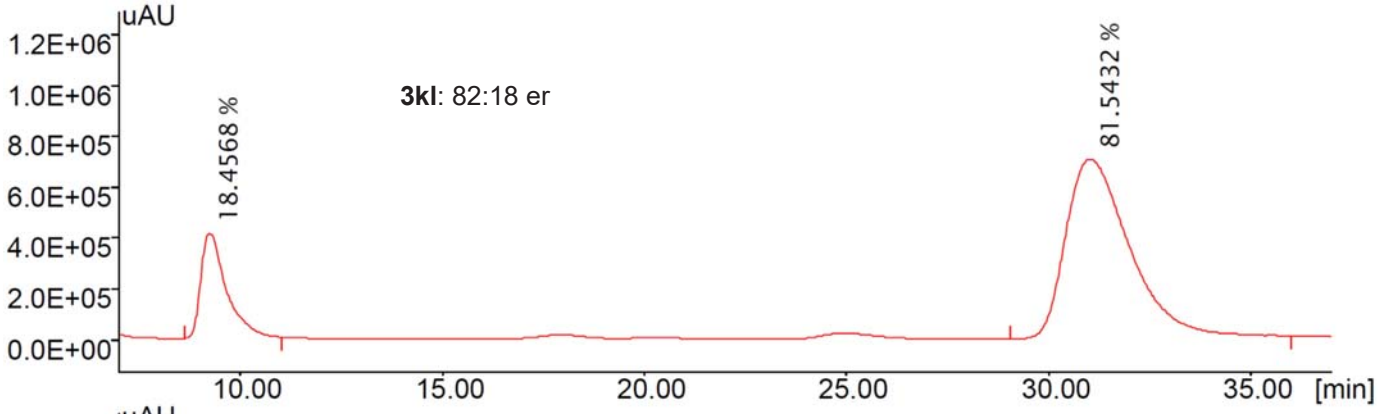


#	Name	RT	%Area
1		24.773	5.067
2		29.699	4.933

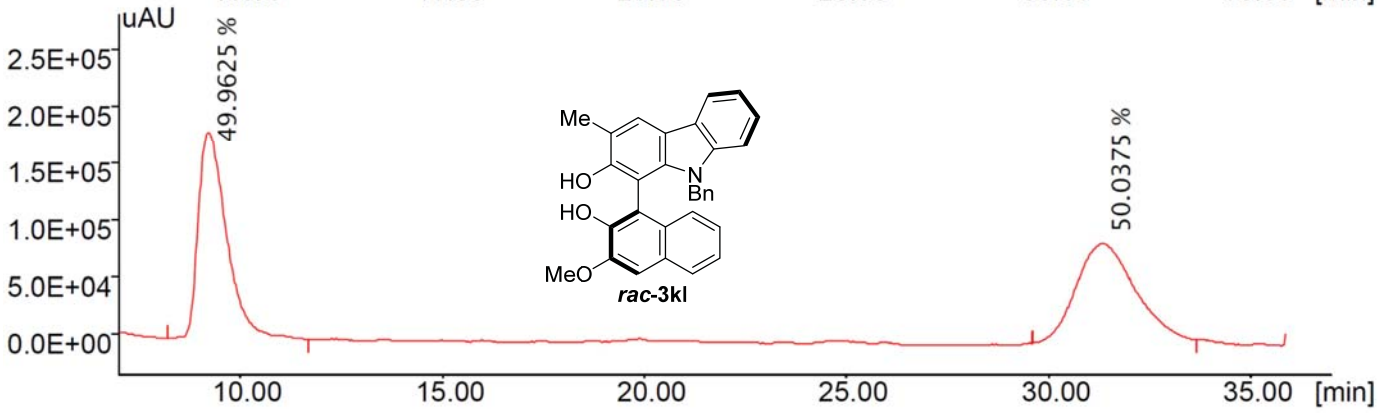




#	Name	RT	%Area
1		11.389	0.548
2		18.973	9.452

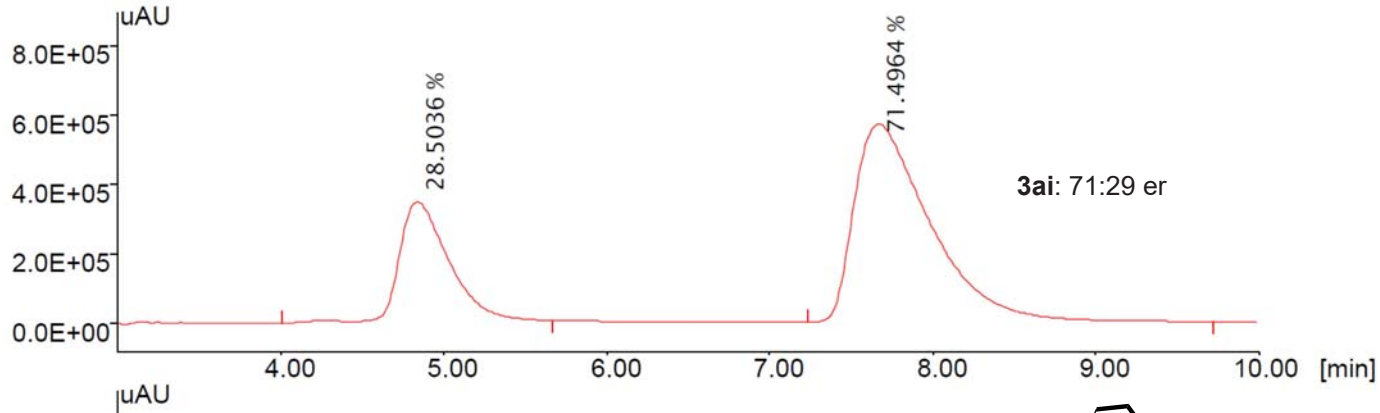


3kl: 82:18 er

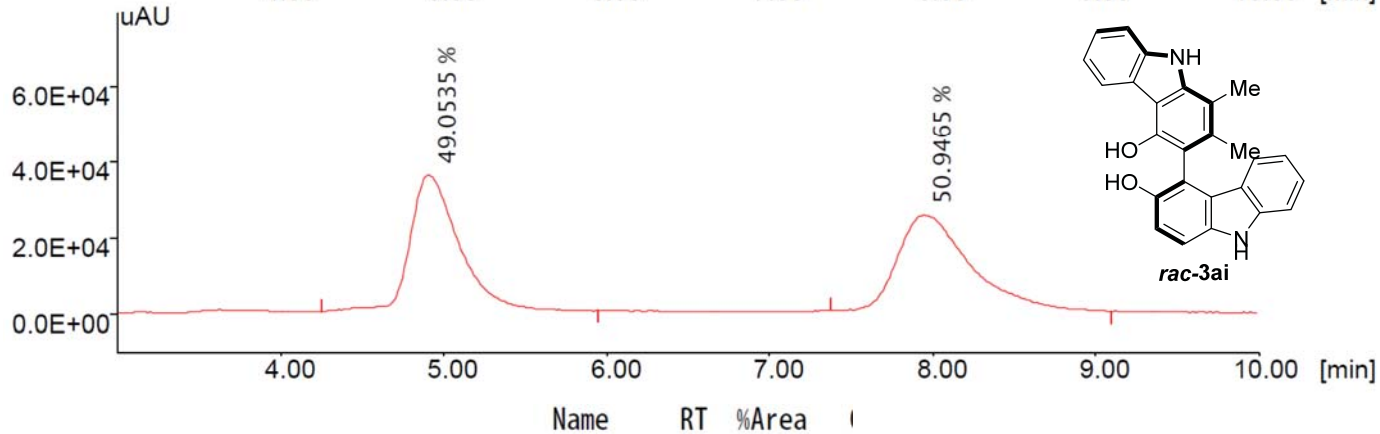


rac-**3kl**

#	Name	RT	%Area
1		9.227	18.457
2		31.048	1.543

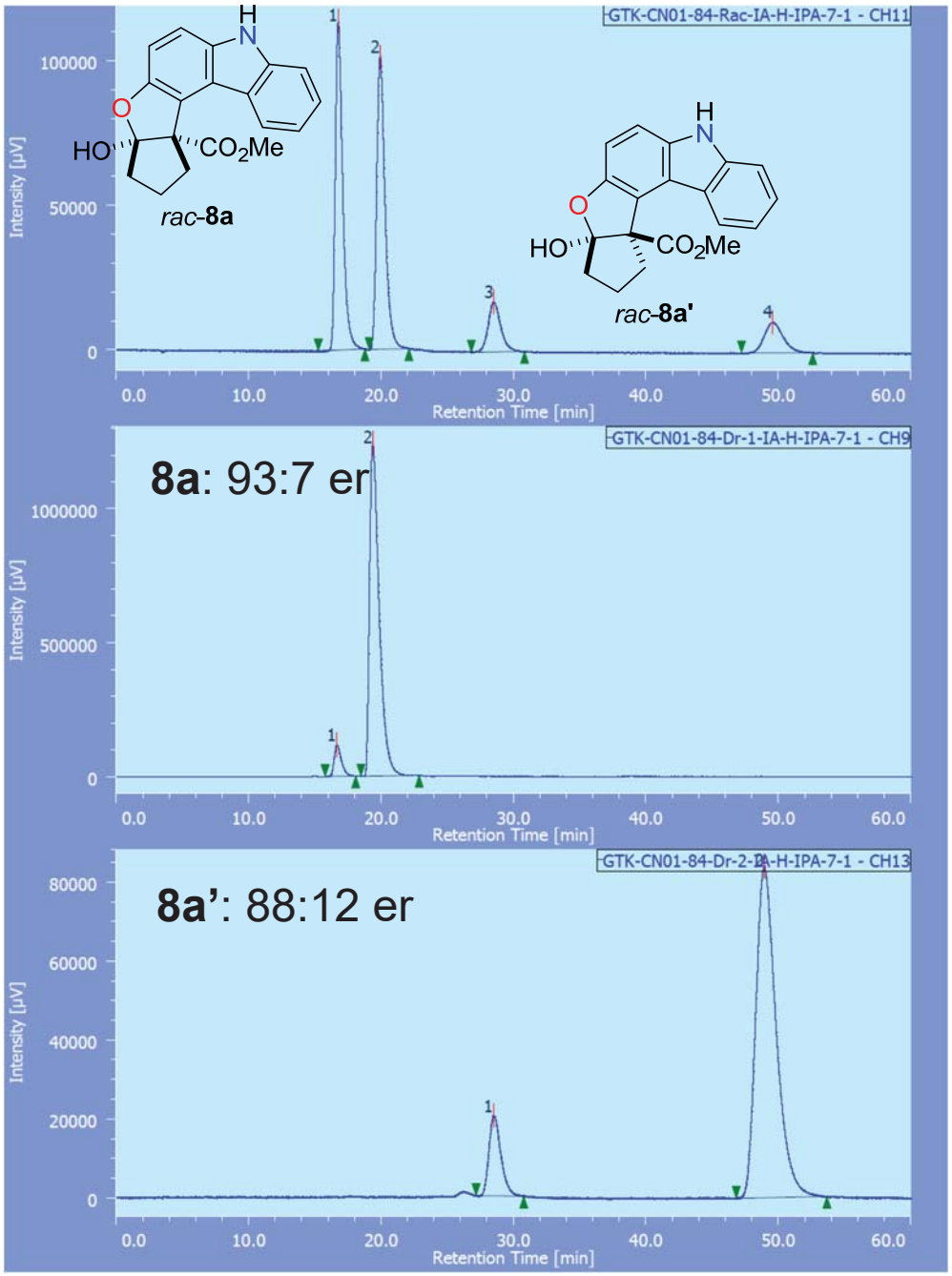


3ai: 71:29 er



rac-**3ai**

Name	RT	%Area
1	4.840	28.504
2	7.667	71.496



Channel & Peak Information Table

Chromatogram Name GTK-CN01-84-Rac-IA-H-IPA-7-1-CH11

Sample Name

Channel Name 300.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	11	16.765	4443087	113927	40.088	46.895	N/A	4725	3.042	1.495	
2	Unknown	11	19.927	4442112	101063	40.079	41.600	N/A	5172	6.247	1.431	
3	Unknown	11	28.515	1140863	17281	10.293	7.113	N/A	4786	9.988	1.223	
4	Unknown	11	49.575	1057382	10668	9.540	4.391	N/A	5910	N/A	1.209	

Chromatogram Name GTK-CN01-84-Dr-1-IA-H-IPA-7-1-CH9

Sample Name

Channel Name 350.0nm

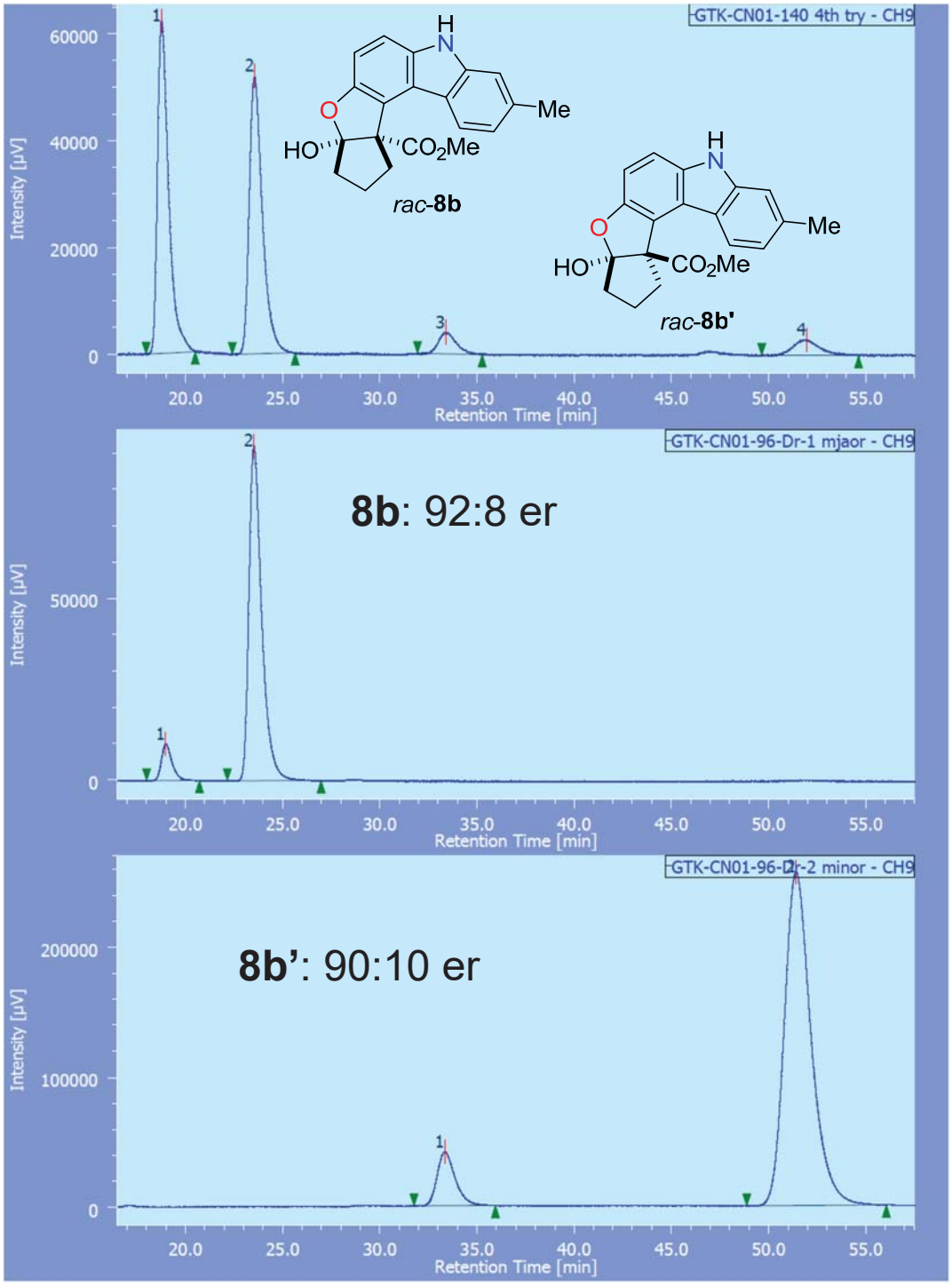
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	16.657	4509171	117336	6.715	8.631	N/A	4615	2.409	1.568	
2	Unknown	9	19.377	62638699	1242073	93.285	91.369	N/A	3648	N/A	1.869	

Chromatogram Name GTK-CN01-84-Dr-2-IA-H-IPA-7-1-CH13

Sample Name

Channel Name 360.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	13	28.480	1267203	20297	12.427	19.452	N/A	5077	9.502	1.464	
2	Unknown	13	48.947	8930060	84046	87.573	80.548	N/A	5179	N/A	1.398	



Channel & Peak Information Table

Chromatogram Name GTK-CN01-140 4th try-CH9

Sample Name

Channel Name 368.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	18.785	2484774	62286	45.344	51.267	N/A	5723	4.389	1.738	
2	Unknown	9	23.562	2452809	52137	44.761	42.914	N/A	6282	6.955	1.482	
3	Unknown	9	33.383	259431	4124	4.734	3.394	N/A	6606	9.274	1.259	
4	Unknown	9	51.940	282802	2946	5.161	2.425	N/A	7686	N/A	1.292	

Chromatogram Name GTK-CN01-96-Dr-1 mjaor-CH9

Sample Name

Channel Name 315.0nm

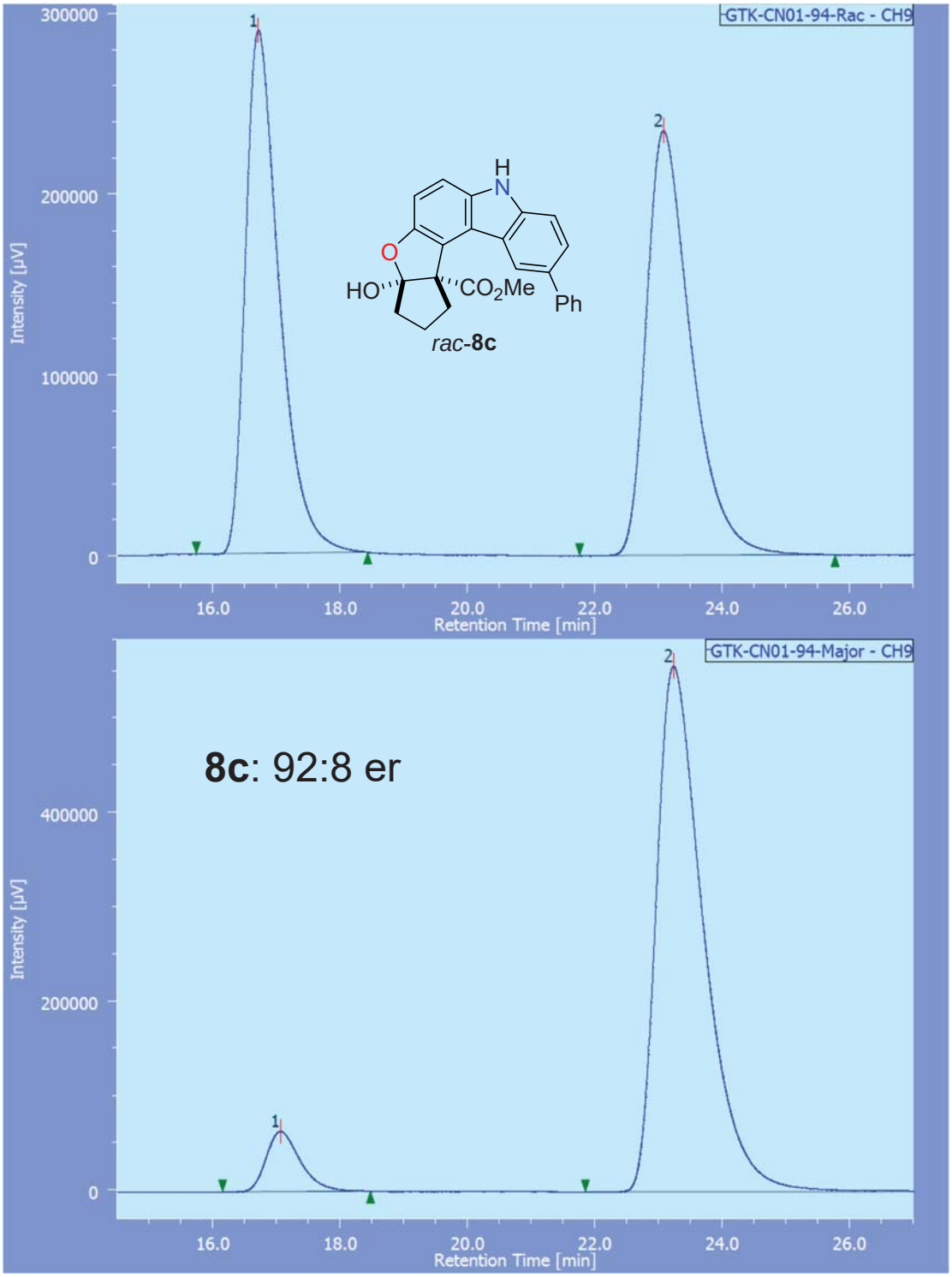
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	18.978	385423	10134	8.351	9.914	N/A	6359	4.349	1.436	
2	Unknown	9	23.530	4229912	92086	91.649	90.086	N/A	6733	N/A	1.519	

Chromatogram Name GTK-CN01-96-Dr-2 minor-CH9

Sample Name

Channel Name 315.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	33.342	2722855	41701	9.980	13.978	N/A	6470	8.850	1.345	
2	Unknown	9	51.397	24561366	256631	90.020	86.022	N/A	7141	N/A	1.299	



Channel & Peak Information Table

Chromatogram Name GTK-CN01-94-Rac-CH9

Sample Name

Channel Name 318.0nm

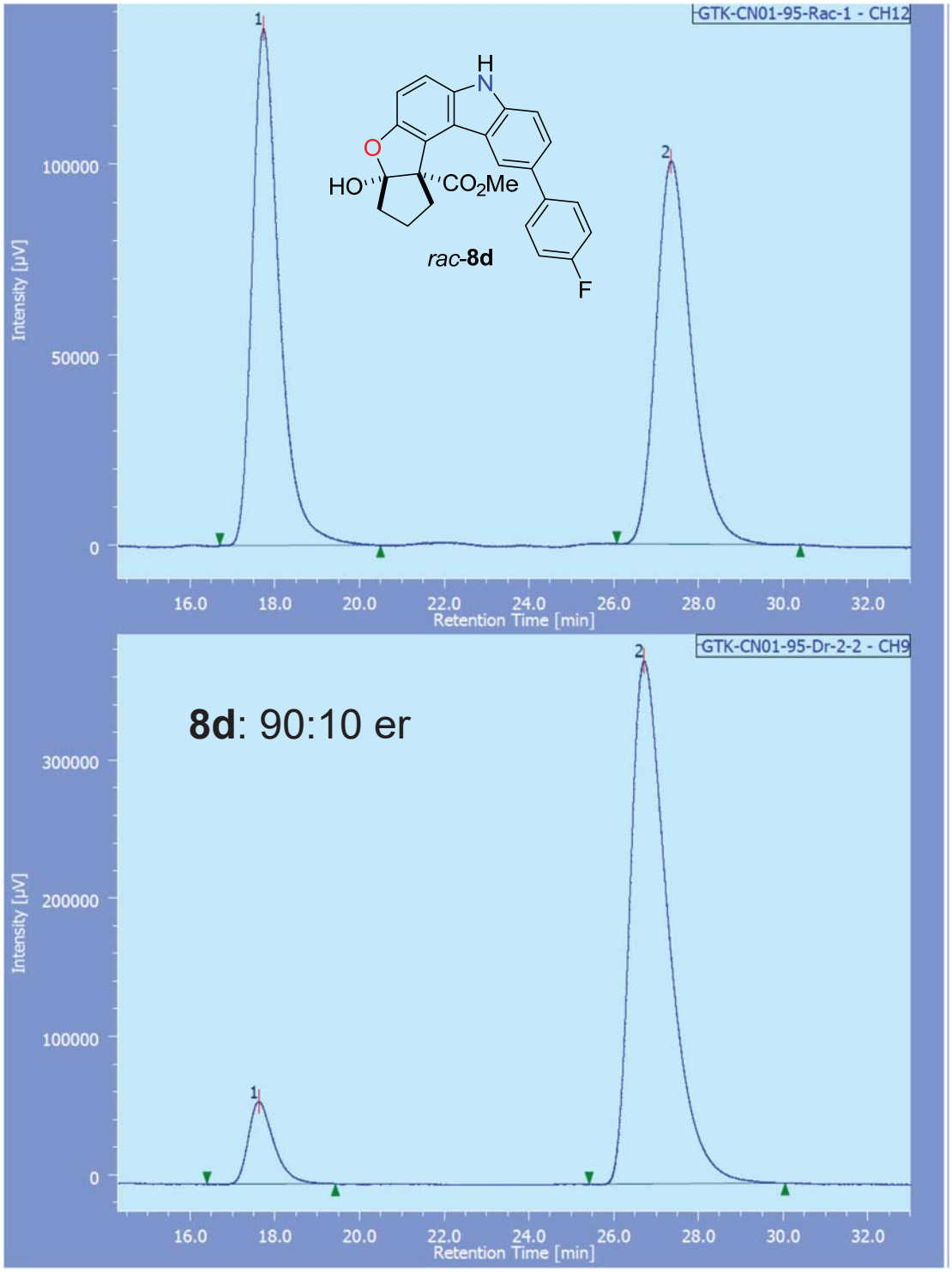
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	16.715	10705070	289335	48.450	55.187	N/A	5051	5.875	1.554	
2	Unknown	9	23.077	11389839	234945	51.550	44.813	N/A	5627	N/A	1.522	

Chromatogram Name GTK-CN01-94-Major-CH9

Sample Name

Channel Name 315.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	17.063	2390130	63929	7.688	10.298	N/A	5096	5.471	1.379	
2	Unknown	9	23.235	28699780	556858	92.312	89.702	N/A	5064	N/A	1.633	



Channel & Peak Information Table

Chromatogram Name GTK-CN01-95-Rac-1-CH12

Sample Name

Channel Name 290.0nm

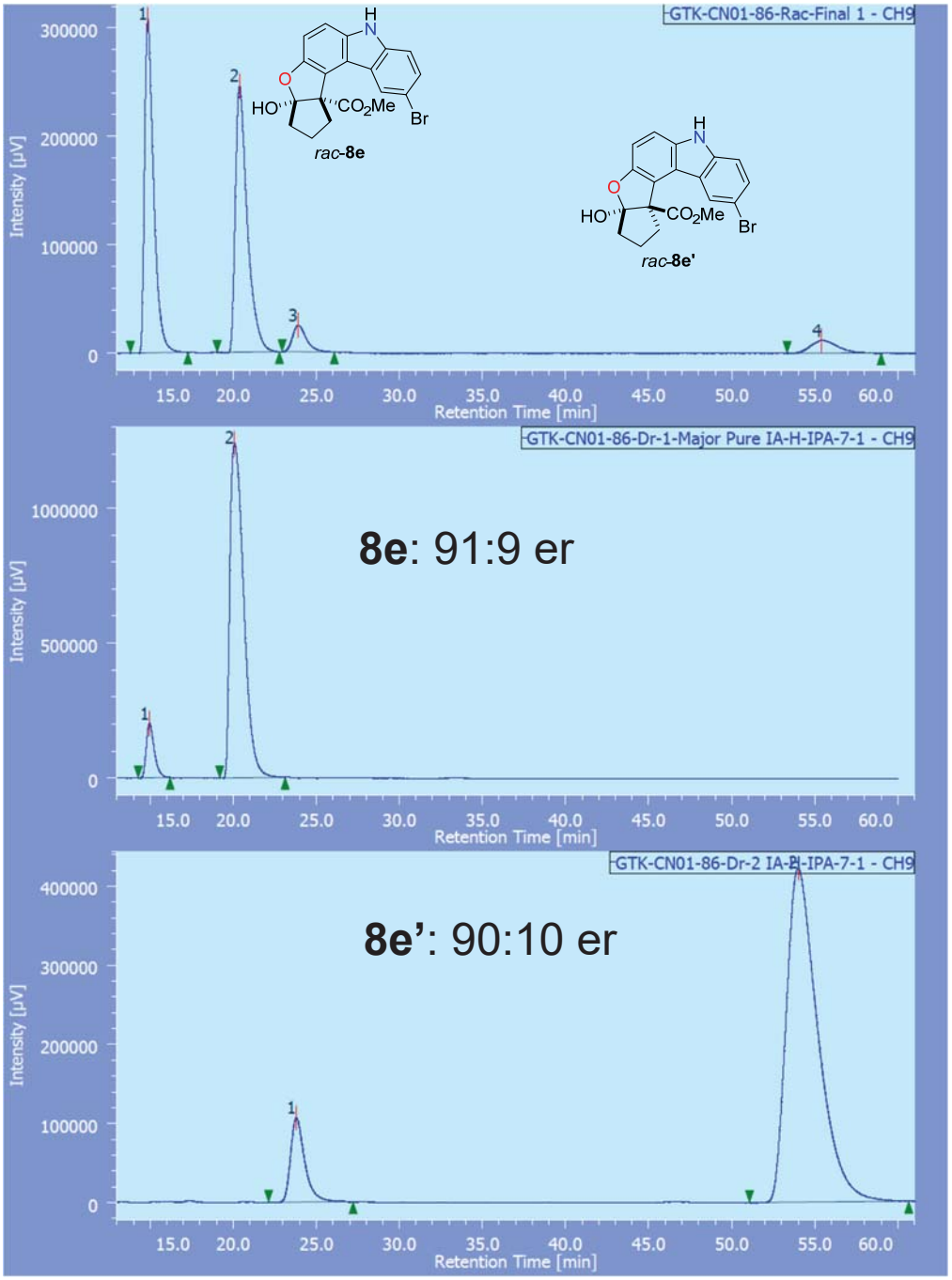
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	12	17.735	6011594	135797	50.519	57.456	N/A	4122	7.407	1.424	
2	Unknown	12	27.345	5887965	100554	49.481	42.544	N/A	5341	N/A	1.338	

Chromatogram Name GTK-CN01-95-Dr-2-2-CH9

Sample Name

Channel Name 315.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	17.623	2494376	59464	9.661	13.593	N/A	4408	6.929	1.431	
2	Unknown	9	26.715	23324737	377998	90.339	86.407	N/A	4635	N/A	1.638	



Channel & Peak Information Table

Chromatogram Name GTK-CN01-86-Rac-Final 1-CH9

Sample Name

Channel Name 315.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	14.858	11715099	307335	43.712	52.163	N/A	3761	4.986	1.769	
2	Unknown	9	20.378	12277965	245473	45.813	41.664	N/A	4246	2.596	1.820	
3	Unknown	9	23.902	1428342	24474	5.330	4.154	N/A	4226	14.040	1.439	
4	Unknown	9	55.403	1378964	11897	5.145	2.019	N/A	5354	N/A	1.305	

Chromatogram Name GTK-CN01-86-Dr-1-Major Pure IA-H-IPA-7-1-CH9

Sample Name

Channel Name 309.0nm

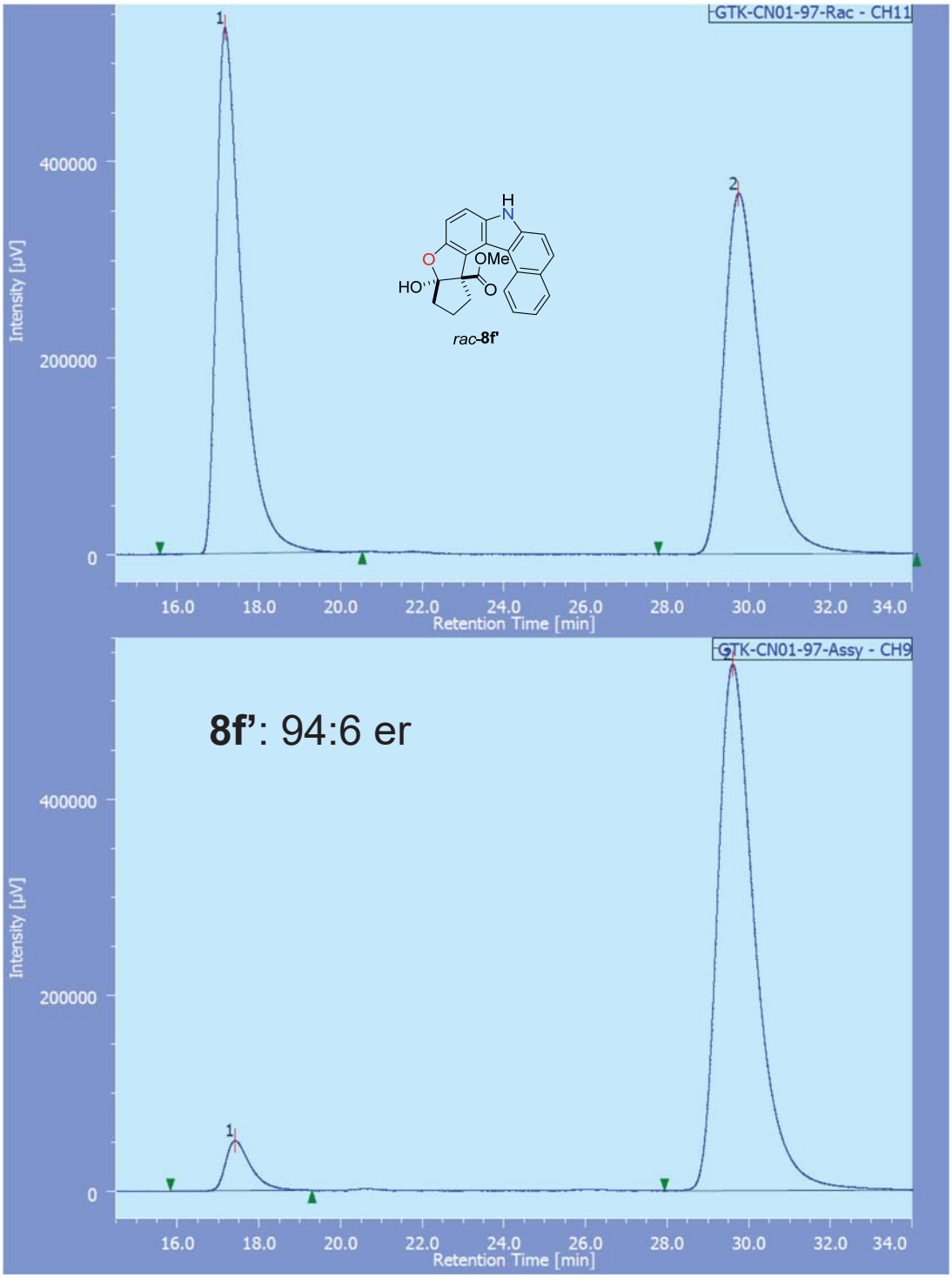
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	14.965	6846661	202269	8.742	14.047	N/A	4756	4.276	1.431	
2	Unknown	9	20.078	71472178	1237674	91.258	85.953	N/A	2755	N/A	1.833	

Chromatogram Name GTK-CN01-86-Dr-2 IA-H-IPA-7-1-CH9

Sample Name

Channel Name 315.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	23.805	6374381	106684	10.272	20.159	N/A	4202	12.454	1.498	
2	Unknown	9	53.987	55681354	422529	89.728	79.841	N/A	4056	N/A	1.599	



Channel & Peak Information Table

Chromatogram Name GTK-CN01-97-Rac-CH11

Sample Name

Channel Name 340.0nm

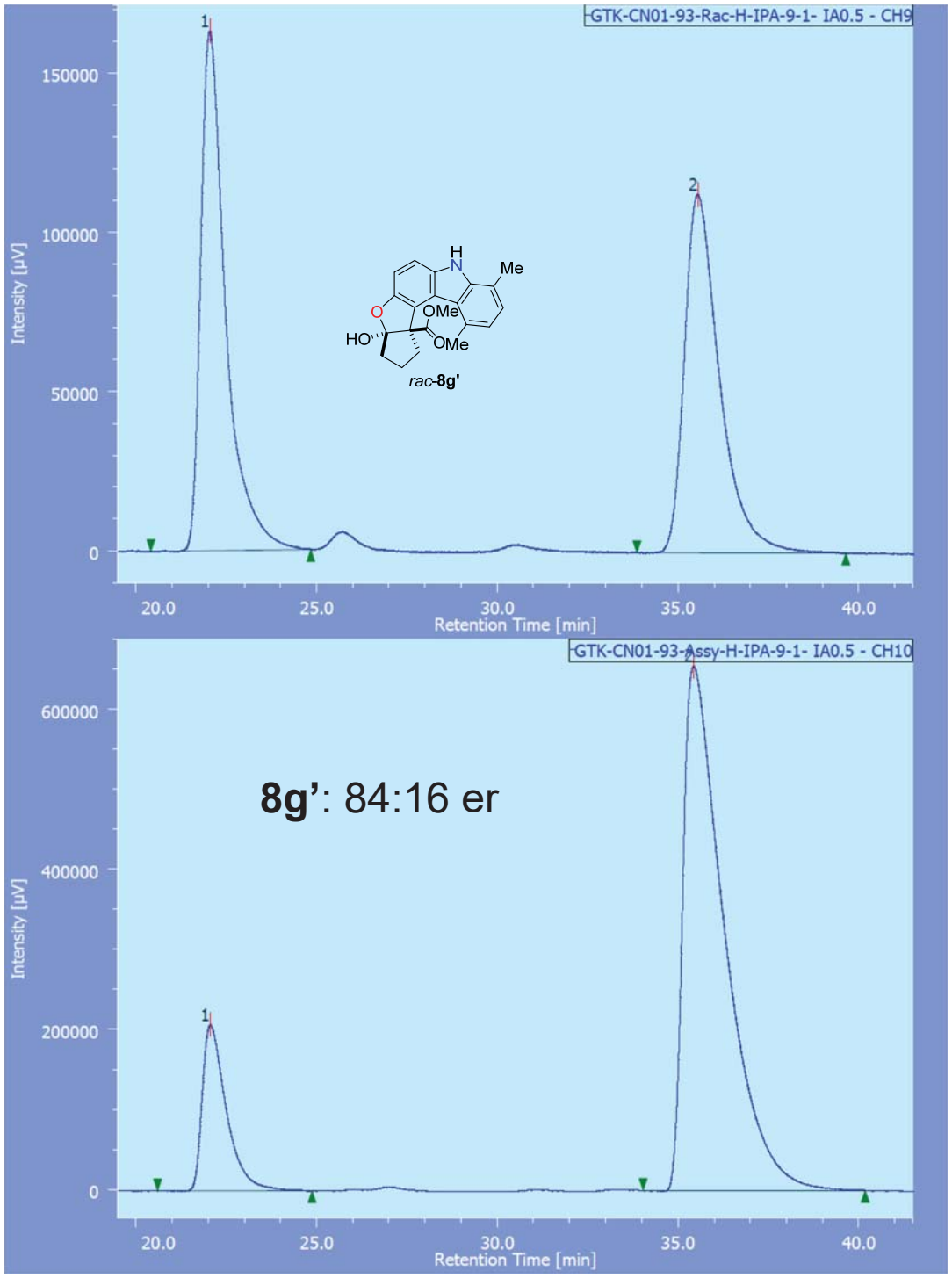
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	11	17.170	23233096	534907	49.516	59.336	N/A	4108	9.390	1.871	
2	Unknown	11	29.745	23687226	366585	50.484	40.664	N/A	5434	N/A	1.545	

Chromatogram Name GTK-CN01-97-Assy-CH9

Sample Name

Channel Name 315.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	17.412	2208525	51438	6.046	8.739	N/A	4228	9.177	1.641	
2	Unknown	9	29.600	34321116	537181	93.954	91.261	N/A	5529	N/A	1.494	



Channel & Peak Information Table

Chromatogram Name GTK-CN01-93-Rac-H-IPA-9-1- IA0.5-CH9

Sample Name

Channel Name 360.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	22.050	7831079	163288	50.764	59.196	N/A	5975	9.613	1.855	
2	Unknown	9	35.555	7595287	112553	49.236	40.804	N/A	7199	N/A	1.566	

Chromatogram Name GTK-CN01-93-Assy-H-IPA-9-1- IA0.5-CH10

Sample Name

Channel Name 315.0nm

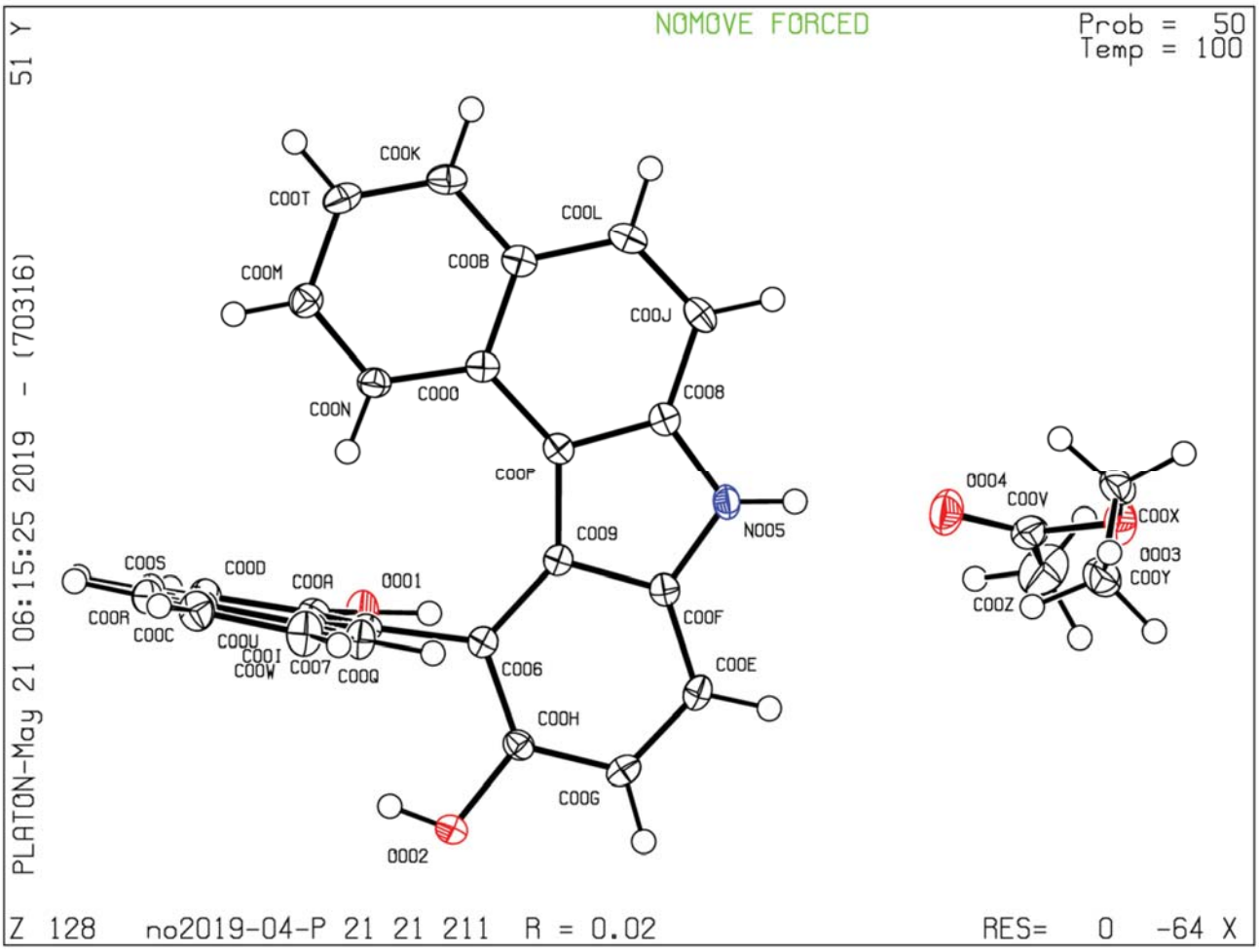
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	10	22.060	9605867	207573	15.577	24.059	N/A	5926	8.536	1.875	
2	Unknown	10	35.428	52060984	655194	84.423	75.941	N/A	5050	N/A	2.529	

X-ray Crystallographic Analysis

Compound (*R*)-**3ga**

Crystal data and structure refinement for CCDC 1966275

Identification code	No2019-04-Higashida-1
Empirical formula	$C_{30}H_{25}NO_4$
Formula weight	463.51
Temperature/K	100.15
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	9.01950(10)
b/Å	13.04640(10)
c/Å	19.72120(10)
α/°	90
ß/°	90
γ/°	90
Volume/Å^3	2320.63(3)
Z	4
ρ_{calc}g/cm^3	1.327
μ/mm^{-1}	0.707
F(000)	976.0
Crystal size/mm^3	0.218 × 0.15 × 0.11
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	8.126 to 151.788
Index ranges	-11 ≤ h ≤ 11, -12 ≤ k ≤ 16, -20 ≤ l ≤ 24
Reflections collected	22800
Independent reflections	4768 [R_{int} = 0.0210, R_{sigma} = 0.0143]
Data/restraints/parameters	4768/0/327
Goodness-of-fit on F^2	1.047
Final R indexes [I>=2σ (I)]	R_1 = 0.0244, wR_2 = 0.0638
Final R indexes [all data]	R_1 = 0.0246, wR_2 = 0.0640
Largest diff. peak/hole / e Å^{-3}	0.18/-0.16
Flack parameter	-0.01(4)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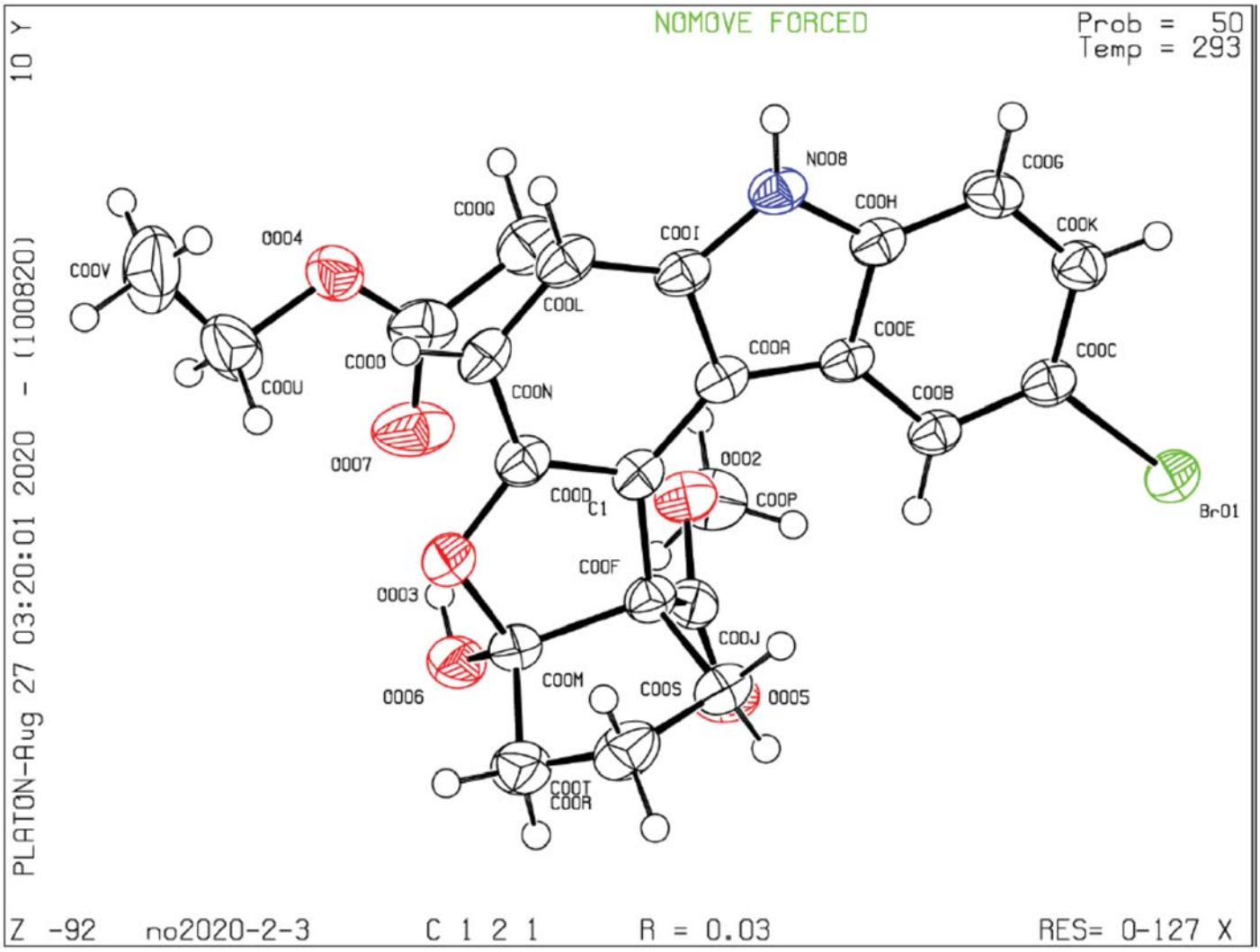
Temp = 100
51 Y
PLATON-May 21 06:15:25 2019 - (70316)
C00K
C00T
C00L
C00M
C00B
C00J
C00N
C00O
C00P
C008
C009
N005
C00S
C00D
C00A
0001
C00R
C00C
C00U
C00I
C00W
C007
C00Q
C006
C00F
C00E
C00H
C00G
0002
0004
C00V
C00X
0003
C00Y
C00Z
Z 128 no2019-04-P 21 21 211 R = 0.02
RES= 0 -64 X

Compound (*R*,*S*)-**8e**

Crystal data and structure refinement for CCDC 2025416

Identification code	No2020-2-3
Empirical formula	$C_{23}H_{24}BrNO_6$
Formula weight	490.34
Temperature/K	293(2)
Crystal system	monoclinic
Space group	C2
a/Å	17.0200(4)
b/Å	7.4729(2)
c/Å	17.5569(4)
α/°	90
ß/°	104.050(2)
γ/°	90
Volume/Å^3	2166.24(9)
Z	4
ρ_{calc}g/cm^3	1.503
μ/mm^{-1}	2.932
F(000)	1008.0
Crystal size/mm^3	0.096 × 0.064 × 0.022
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	5.188 to 150.998
Index ranges	$-20 \le h \le 21$, $-9 \le k \le 9$, $-21 \le l \le 22$
Reflections collected	14364
Independent reflections	4332 [R_{int} = 0.0339, R_{sigma} = 0.0358]
Data/restraints/parameters	4332/1/284
Goodness-of-fit on F^2	1.100
Final R indexes [I>=2σ (I)]	R_1 = 0.0293, wR_2 = 0.0770
Final R indexes [all data]	R_1 = 0.0306, wR_2 = 0.0776
Largest diff. peak/hole / e Å^{-3}	0.38/-0.41
Flack parameter	-0.023(13)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Temp = 293
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PLATON-Aug 27 03:20:01 2020 - (100820)
Z -92 no2020-2-3 C 1 2 1 R = 0.03 RES= 0-127 X
NO08
C00G
C00H
C00K
C00I
C00Q
0004
C00V
C00L
C00E
C00A
C00C
C00D
C00U
C00N
C00B
0007
0002
C00D C1
C00P
Br01
C00F
0003
C00J
C00M
C00S
0006
0005
C00T
C00R