

The ESI for *Org. Biomol. Chem.*, 2017, **15**, 1913–1920, DOI: 10.1039/C6OB02479C, originally published on 7th February 2017, was updated on 2nd August 2022.

Direct access to the optically active VACHT inhibitor vesamicol and its analogues *via* the asymmetric aminolysis of *meso*-epoxides with secondary aliphatic amines

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Correction Notice for Revised ESI

The NMR spectra of **4a**, **4b**, **4c**, **4i**, **4j**, **4k** and **4p** provided earlier were those of racemic compounds. Now this oversight has been corrected by replacing them with NMR spectra of optically active compounds.

ELECTRONIC SUPPLEMENTARY INFORMATION

NMR data of compounds 4a , 4b , 4c , 4i , 4j , 4k and 4p	S2
Copies of ^1H and ^{13}C NMR spectra of compounds 4a – 4p	S3–S18
HPLC Analysis data of compounds 4a – 4p	S19–S20
Copies of HPLC chromatograms of compounds 4a – 4p	S21–S36
CD-Spectra of selected compounds.....	S37

NMR data of compounds 4a, 4b, 4c, 4i, 4j, 4k and 4p

(1S,2S)-2-(4-Phenylpiperidin-1-yl)cyclohexanol (4a). ^1H NMR (500 MHz, CDCl_3): δ 7.33–7.28 (m, 2H), 7.25–7.21 (m, 3H), 3.39 (dt, $J = 4.5, 10.0$ Hz, 1H), 2.94 (d, $J = 11.0$ Hz, 1H), 2.77–2.70 (m, 2H), 2.49 (tt, $J = 4.0, 12.0$ Hz, 1H), 2.28–2.20 (m, 2H), 2.18–2.10 (m, 1H), 1.91–1.76 (m, 5H), 1.74–1.61 (m, 2H), 1.30–1.15 (m, 4H) ppm. ^{13}C NMR (125 MHz, CDCl_3) δ 146.2, 128.3, 126.7, 126.0, 70.6, 68.6, 53.4, 45.4, 42.9, 34.2, 33.9, 33.2, 25.5, 24.0, 22.2 ppm.

(1S,2S)-2-(4-p-Tolylpiperidin-1-yl)cyclohexanol (4b). ^1H NMR (500 MHz, CDCl_3): δ 7.12 (s, 4H), 3.47–3.40 (m, 1H), 3.00 (d, $J = 11.5$ Hz, 1H), 2.87–2.71 (m, 2H), 2.47 (tt, $J = 4.0, 12.0$ Hz, 1H), 2.32 (s, 3H), 2.31–2.26 (m, 2H), 2.17–2.11 (m, 1H), 1.90–1.78 (m, 4H), 1.75–1.68 (m, 2H), 1.30–1.18 (m, 5H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 142.9, 135.7, 129.1, 126.6, 70.6, 68.7, 53.3, 45.6, 42.3, 33.9, 33.6, 33.4, 29.6, 25.5, 24.0, 22.4, 20.9 ppm.

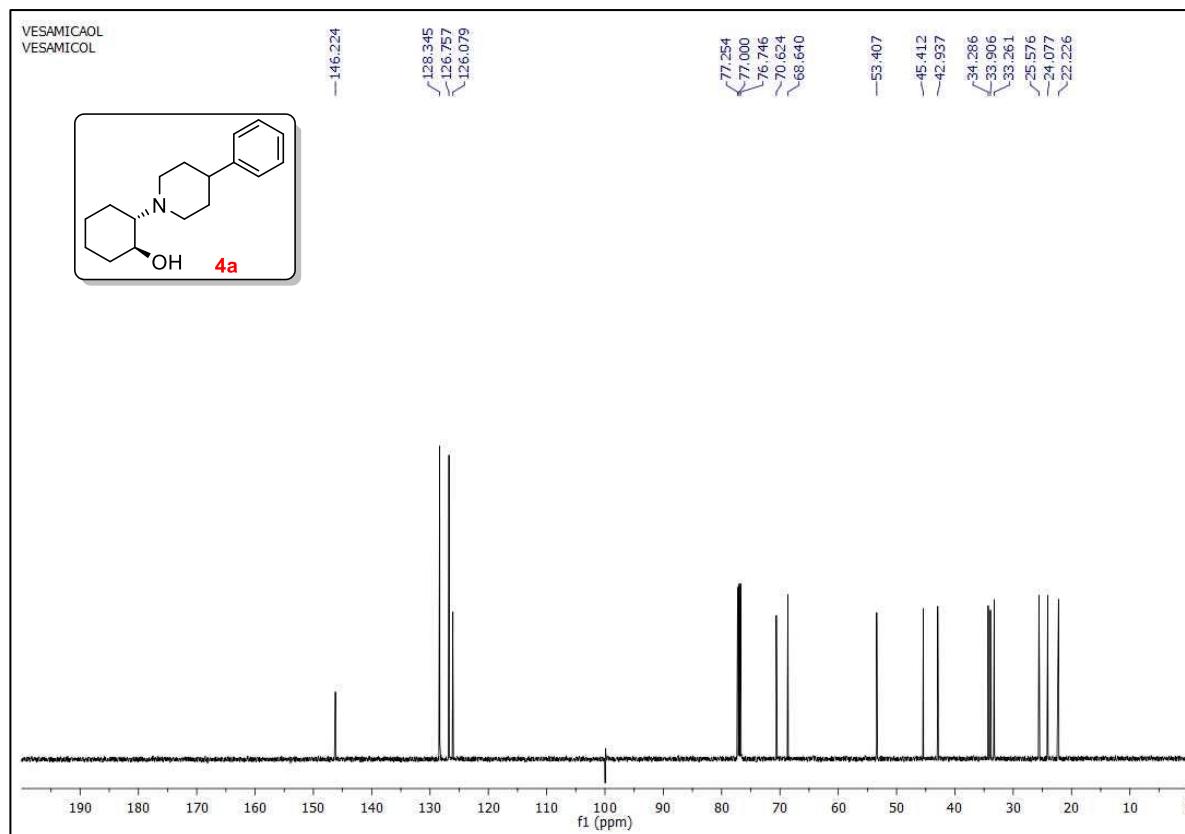
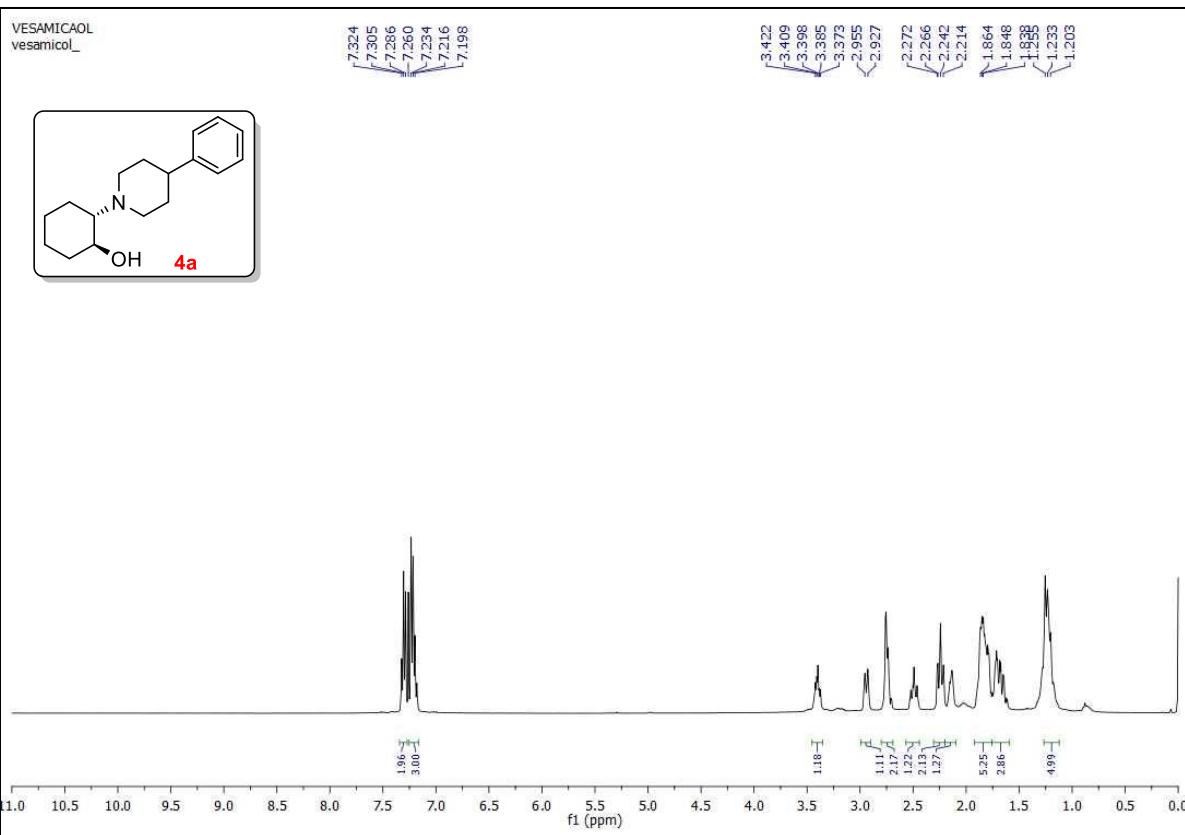
(1S,2S)-2-(4-(4-Methoxyphenyl)piperidin-1-yl)cyclohexanol (4c). ^1H NMR (400 MHz, CDCl_3): δ 7.14 (d, $J = 8.5$ Hz, 2H), 6.85 (d, $J = 8.5$ Hz, 2H), 3.79 (s, 3H), 3.45–3.36 (m, 1H), 2.94 (d, $J = 10.0$ Hz, 1H), 2.81–2.70 (m, 2H), 2.49–2.40 (m, 1H), 2.30–2.20 (m, 2H), 2.18–2.10 (m, 1H), 1.89–1.76 (m, 6H), 1.75–1.50 (m, 2H), 1.32–1.15 (m, 3H) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ 157.9, 138.4, 127.6, 113.7, 70.6, 68.6, 55.2, 53.4, 45.4, 42.0, 34.5, 34.1, 33.2, 25.6, 24.1, 22.2 ppm.

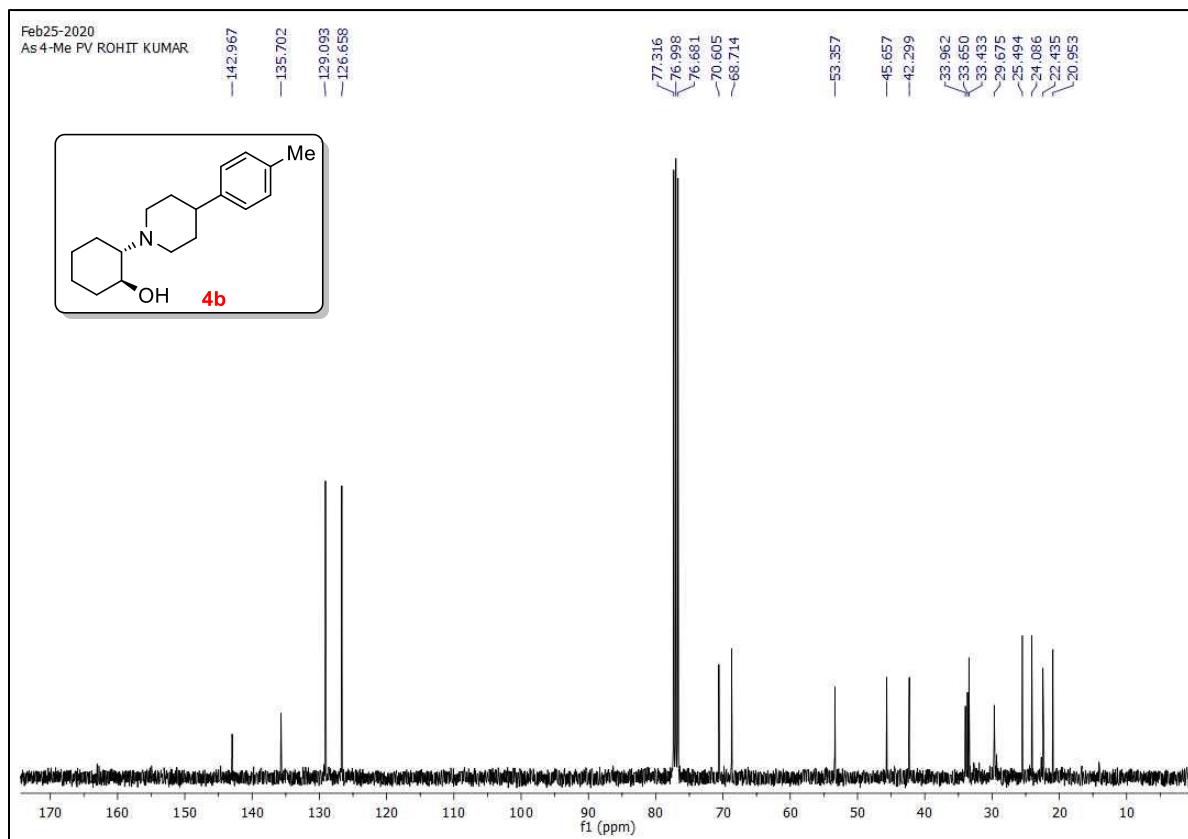
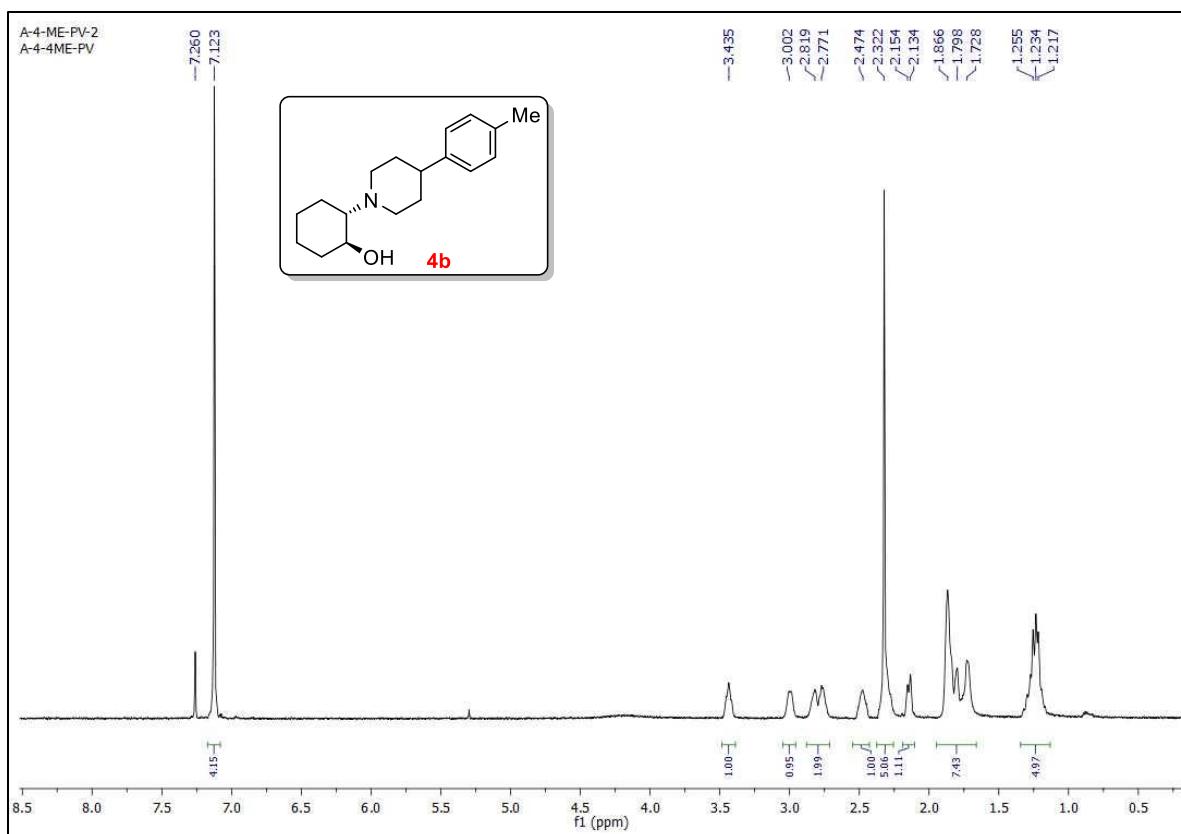
(2S,3S)-3-(4-Phenylpiperidin-1-yl)-1,2,3,4-tetrahydronaphthalen-2-ol (4i). ^1H NMR (500 MHz, CDCl_3): δ 7.35–7.31 (m, 2H), 7.27–7.19 (m, 3H), 7.16–7.08 (m, 4H), 4.38 (s, 1H, –OH), 3.90 (dt, $J = 6.0, 10.0$ Hz, 1H), 3.33 (dd, $J = 6.0, 16.0$ Hz, 1H), 3.12–2.78 (m, 7H), 2.56 (tt, $J = 3.5, 12.0$ Hz, 1H), 2.39 (dt, $J = 2.0, 11.5$ Hz, 1H), 1.97–1.81 (m, 3H), 1.75 (ddd, $J = 4.0, 12.5, 25.0$ Hz, 1H) ppm. ^{13}C NMR (125 MHz, CDCl_3) δ 146.0, 134.8, 134.0, 129.2, 129.1, 128.4, 126.8, 126.2, 126.1, 125.9, 66.5, 65.6, 53.6, 45.0, 42.9, 37.8, 34.3, 33.8, 26.0 ppm.

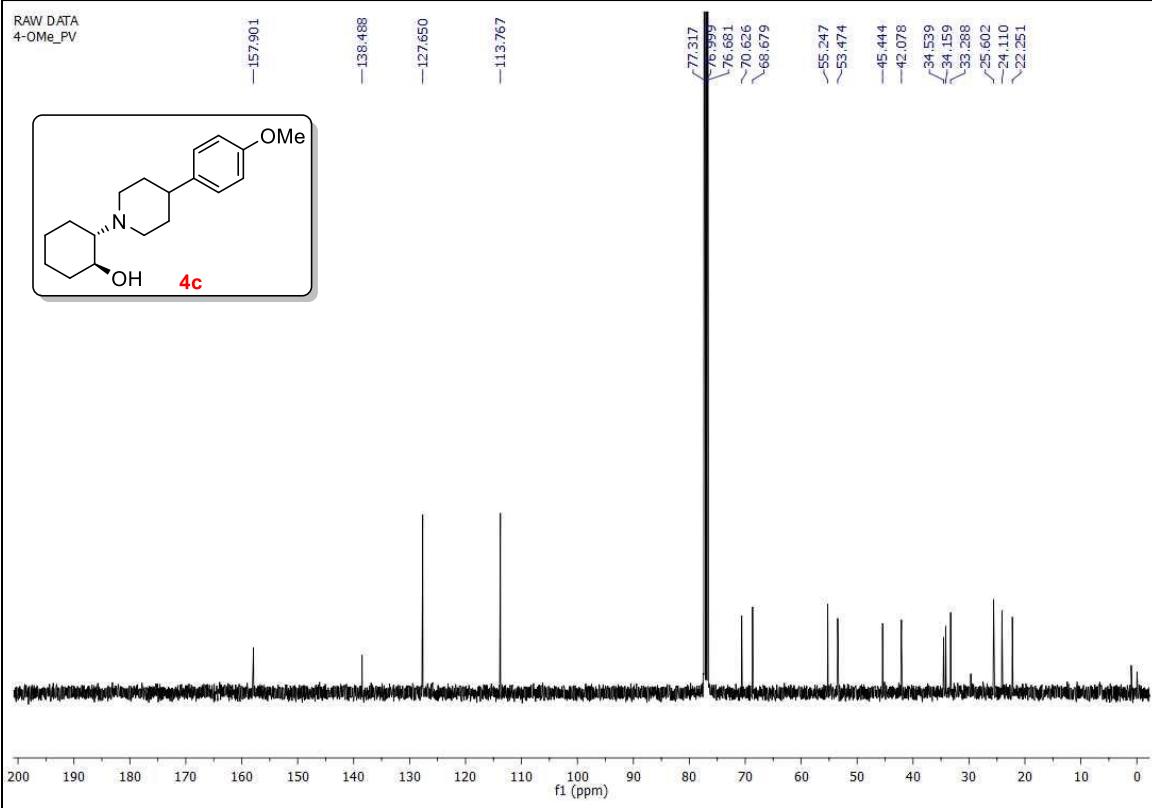
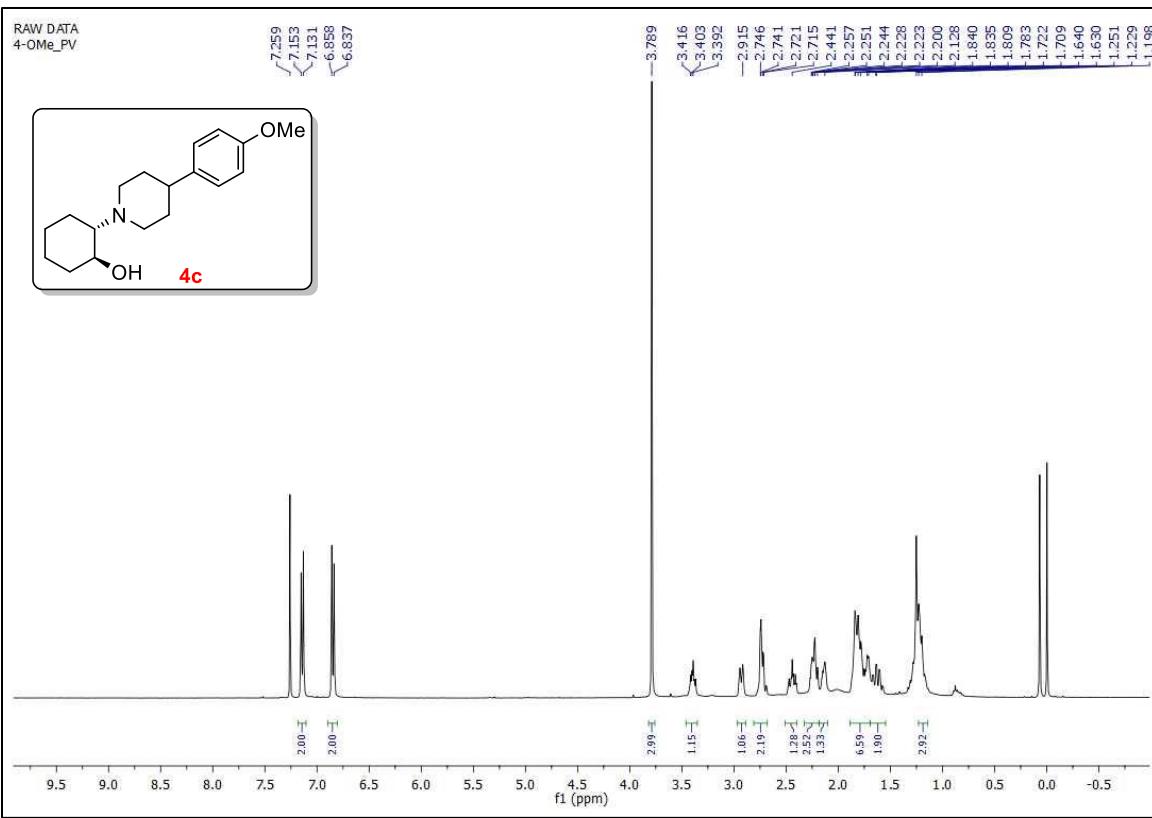
(2S,3S)-3-(4-p-Tolylpiperidin-1-yl)-1,2,3,4-tetrahydronaphthalen-2-ol (4j). ^1H NMR (500 MHz, CDCl_3): δ 7.18–7.08 (m, 8H), 3.93 (dt, $J = 6.0, 10.0$ Hz, 1H), 3.33 (dd, $J = 6.0, 16.0$ Hz, 1H), 3.08–2.78 (m, 7H), 2.55 (tt, $J = 8.0, 12.0$ Hz, 1H), 2.43 (dt, $J = 2.5, 11.5$ Hz, 1H), 2.35 (s, 3H), 1.99–1.78 (m, 3H), 1.83–1.72 (m, 1H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ 143.0, 135.7, 134.8, 134.0, 129.2, 129.1, 126.6, 126.1, 126.0, 66.5, 65.7, 53.6, 45.0, 42.4, 37.8, 34.3, 33.9, 26.0, 20.9 ppm.

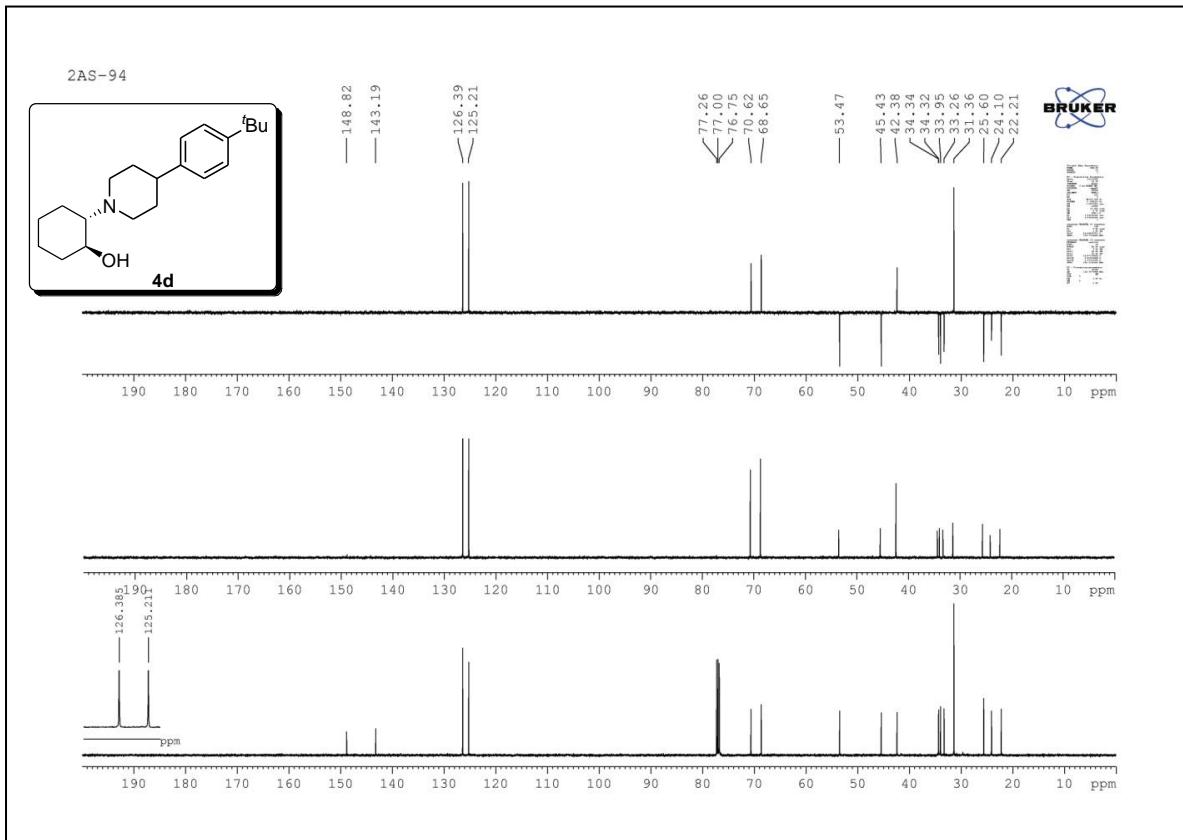
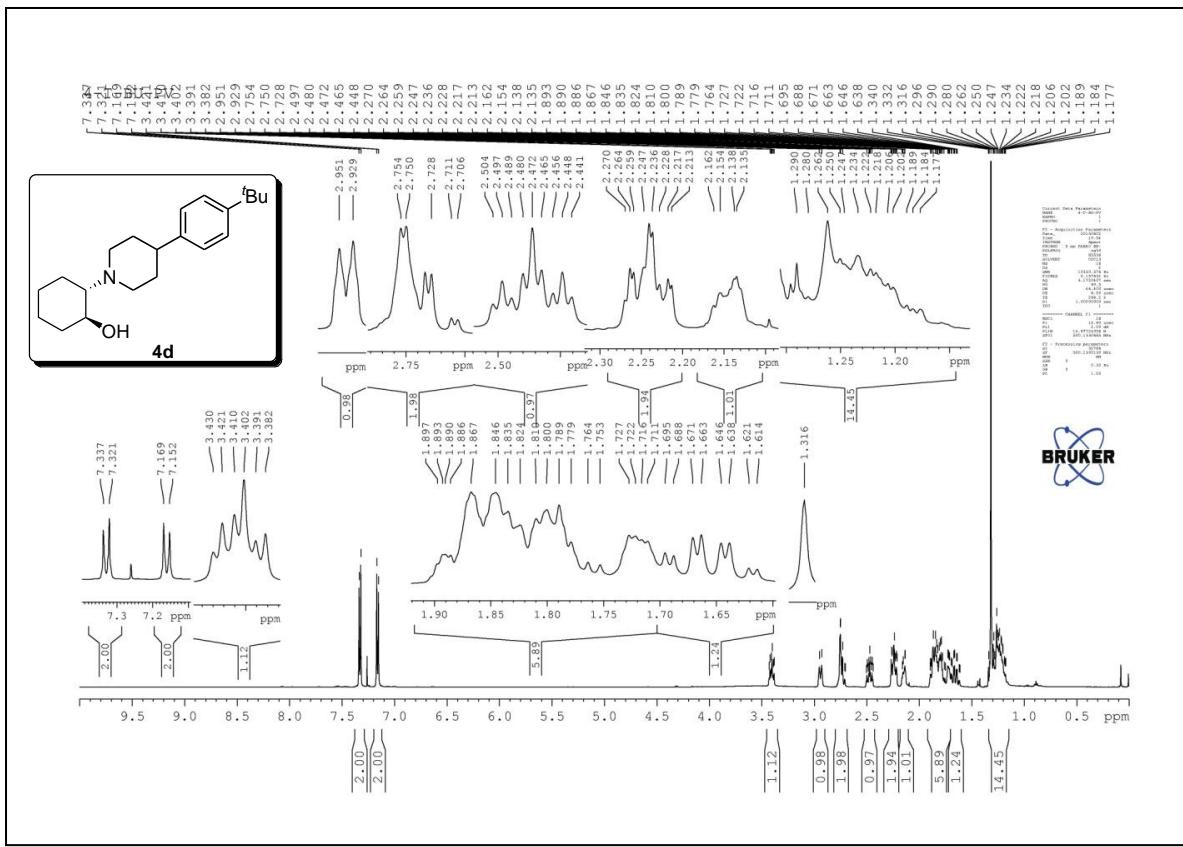
(2S,3S)-3-(4-(4-Methoxyphenyl)piperidin-1-yl)-1,2,3,4-tetrahydronaphthalen-2-ol (4k). ^1H NMR (500 MHz, CDCl_3): δ 7.17–7.08 (m, 6H), 6.87 (d, $J = 8.5$ Hz, 2H), 3.89 (tt, $J = 6.0, 10.0$ Hz, 1H), 3.80 (s, 3H), 3.32 (dd, $J = 6.0, 16.0$ Hz, 1H), 3.12–2.78 (m, 7H), 2.51 (tt, $J = 3.5, 12.0$ Hz, 1H), 2.38 (t, $J = 11.0$ Hz, 1H), 1.97–1.76 (m, 3H), 1.80–1.60 (m, 1H) ppm. ^{13}C NMR (125 MHz, CDCl_3): δ 157.9, 138.2, 134.8, 133.9, 129.2, 129.1, 127.6, 126.1, 125.9, 113.8, 66.5, 65.6, 55.2, 53.6, 45.0, 42.0, 37.8, 34.5, 34.0, 26.0 ppm.

(1S,2S)-2-(Spiro[1-H-indene-1,4-piperidin-1-yl)cyclohexanol (4p). ^1H NMR (500 MHz, CDCl_3): δ 7.38 (d, $J = 7.0$ Hz, 1H), 7.31 (d, $J = 7.0$ Hz, 1H), 7.25 (m, 2H), 6.82 (d, $J = 6.0$ Hz, 1H), 6.74 (d, $J = 6.0$ Hz, 1H), 3.48–3.41 (m, 1H), 3.00–2.92 (m, 2H), 2.79–2.72 (m, 1H), 2.50 (dt, $J = 2.0, 12.0$ Hz, 1H), 2.36–2.27 (m, 1H), 2.24–2.13 (m, 2H), 2.06 (dt, $J = 4.0, 13.0$ Hz, 1H), 1.98–1.90 (m, 1H), 1.86–1.79 (m, 1H), 1.76–1.71 (m, 1H), 1.43–1.36 (m, 2H), 1.33–1.21 (m, 4H) ppm.

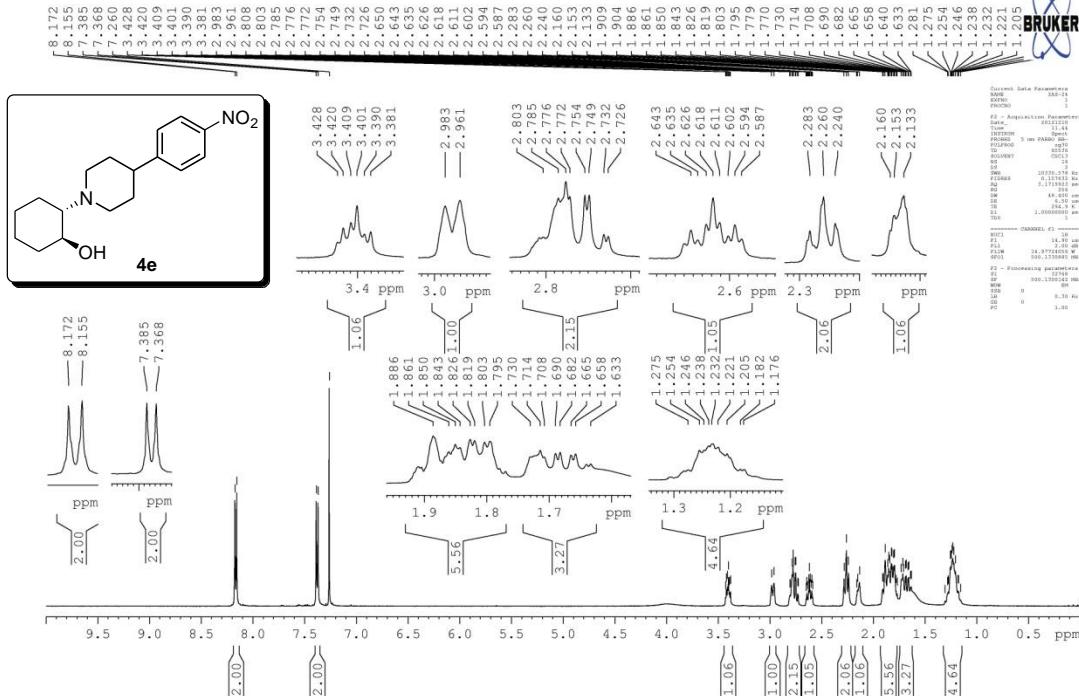




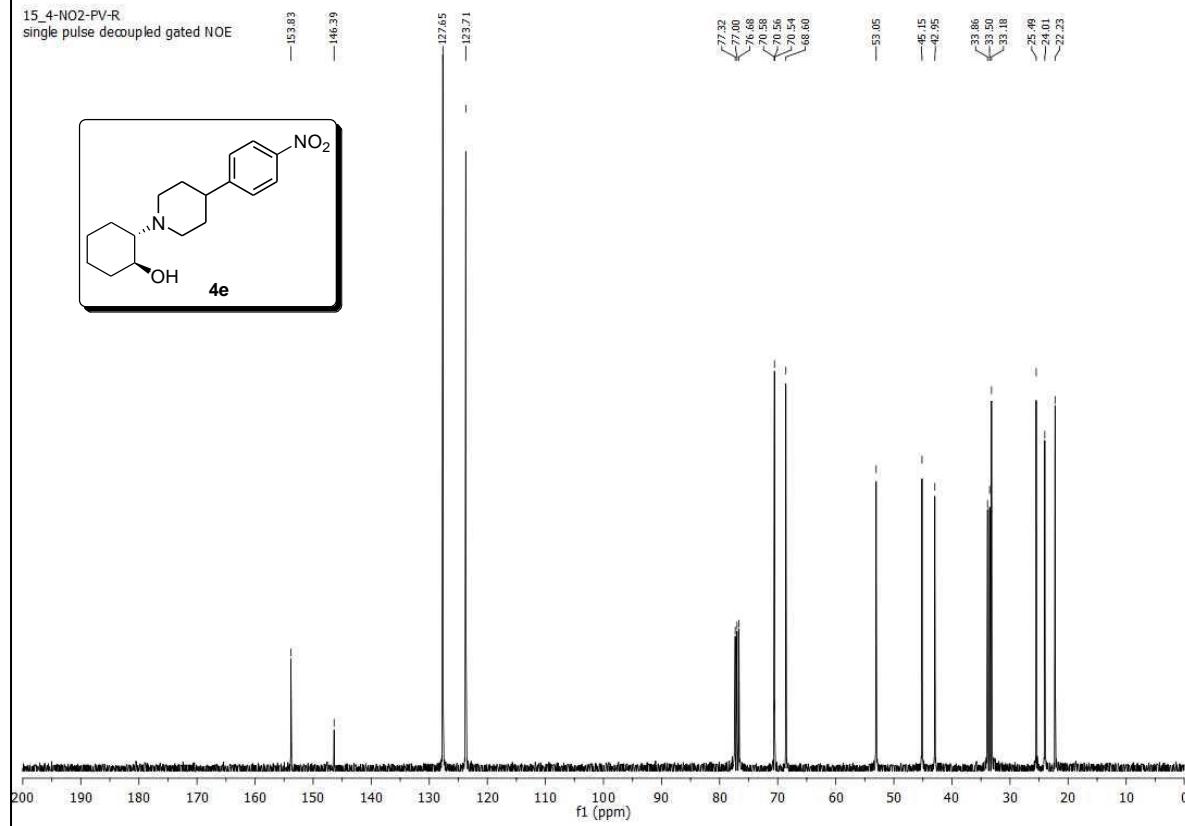


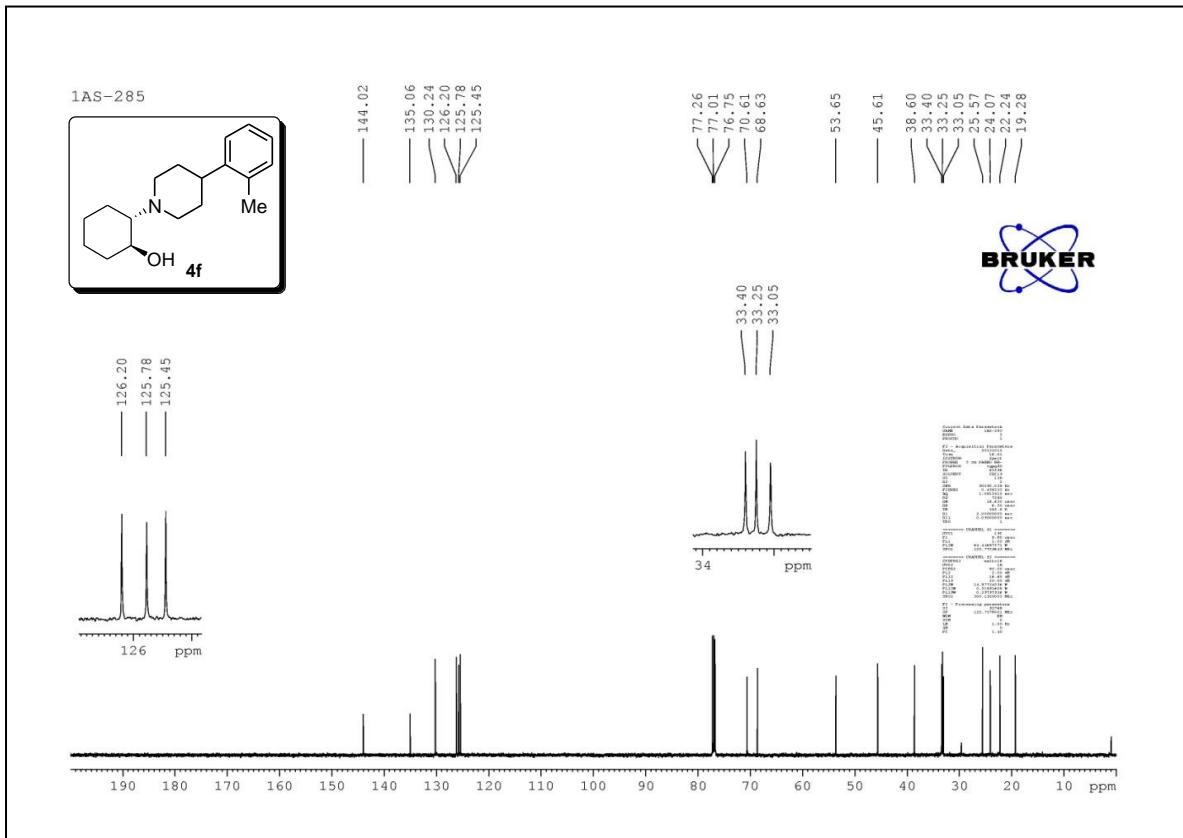
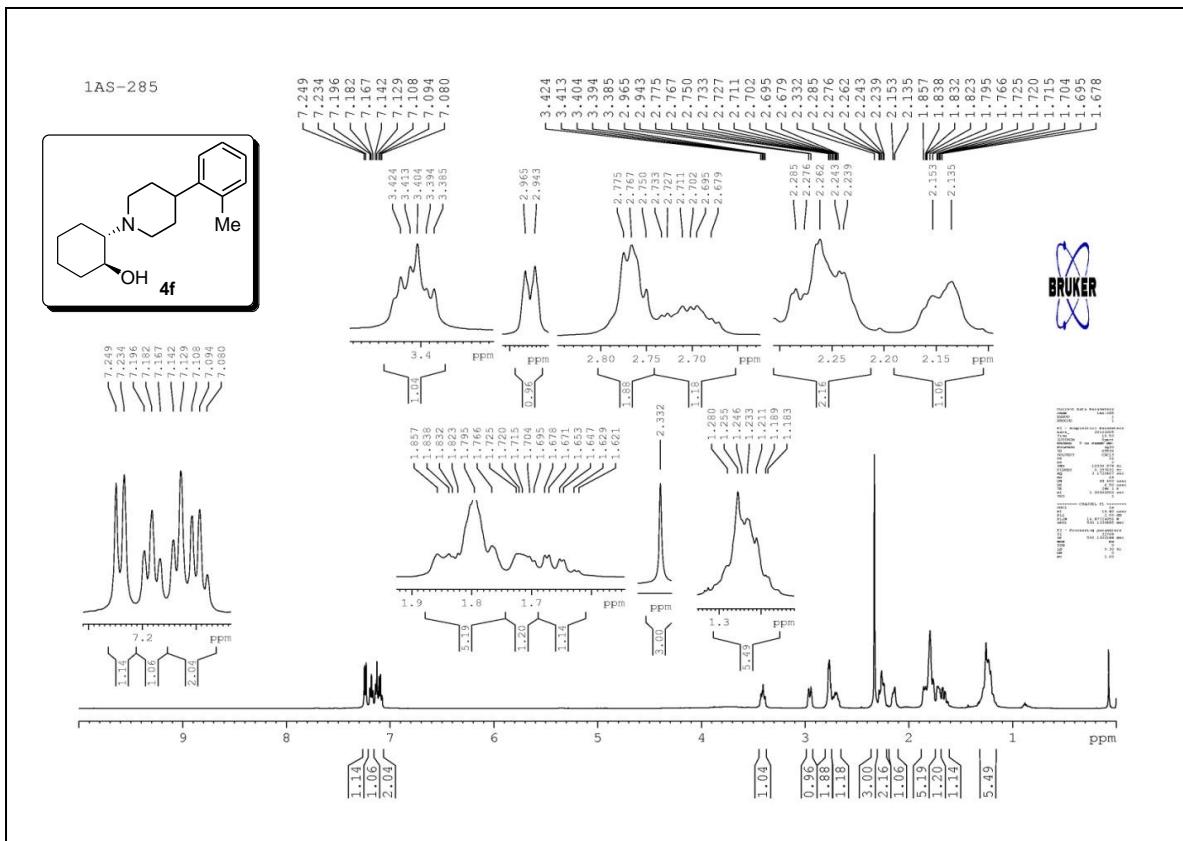


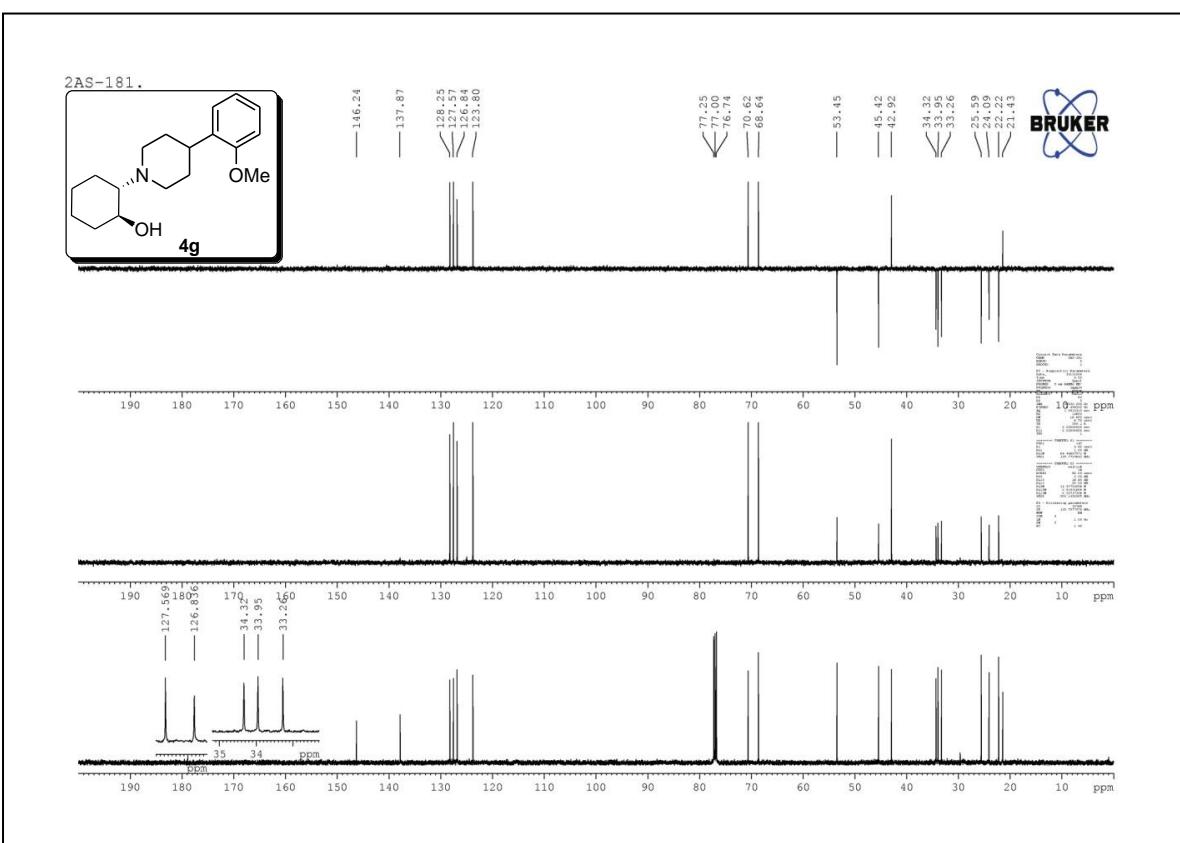
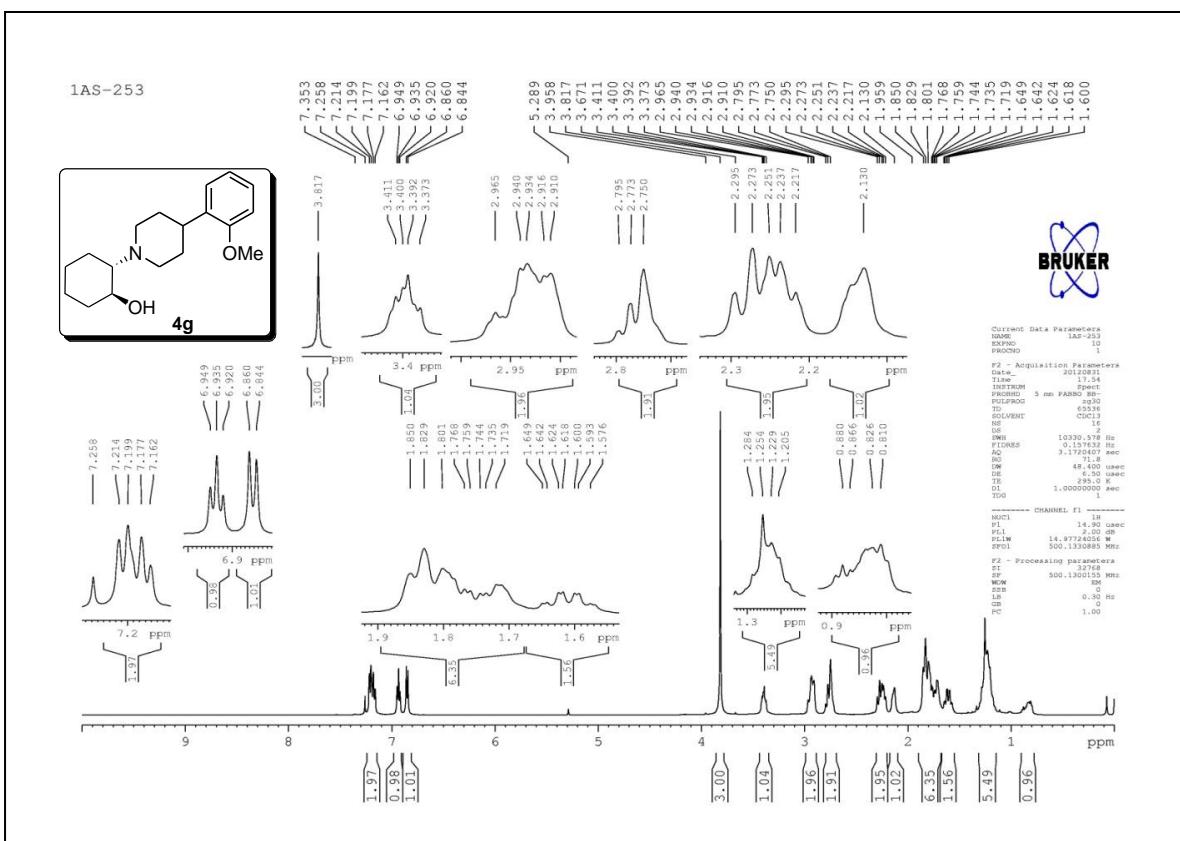
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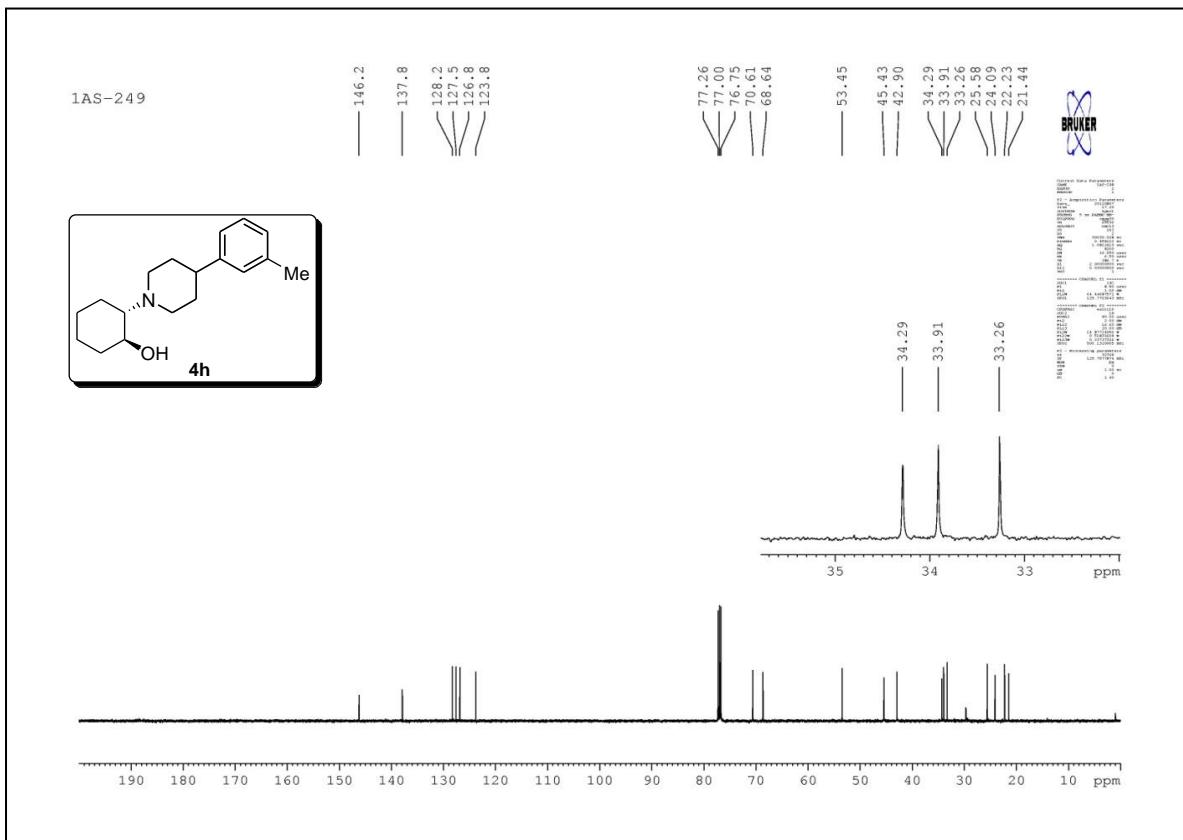
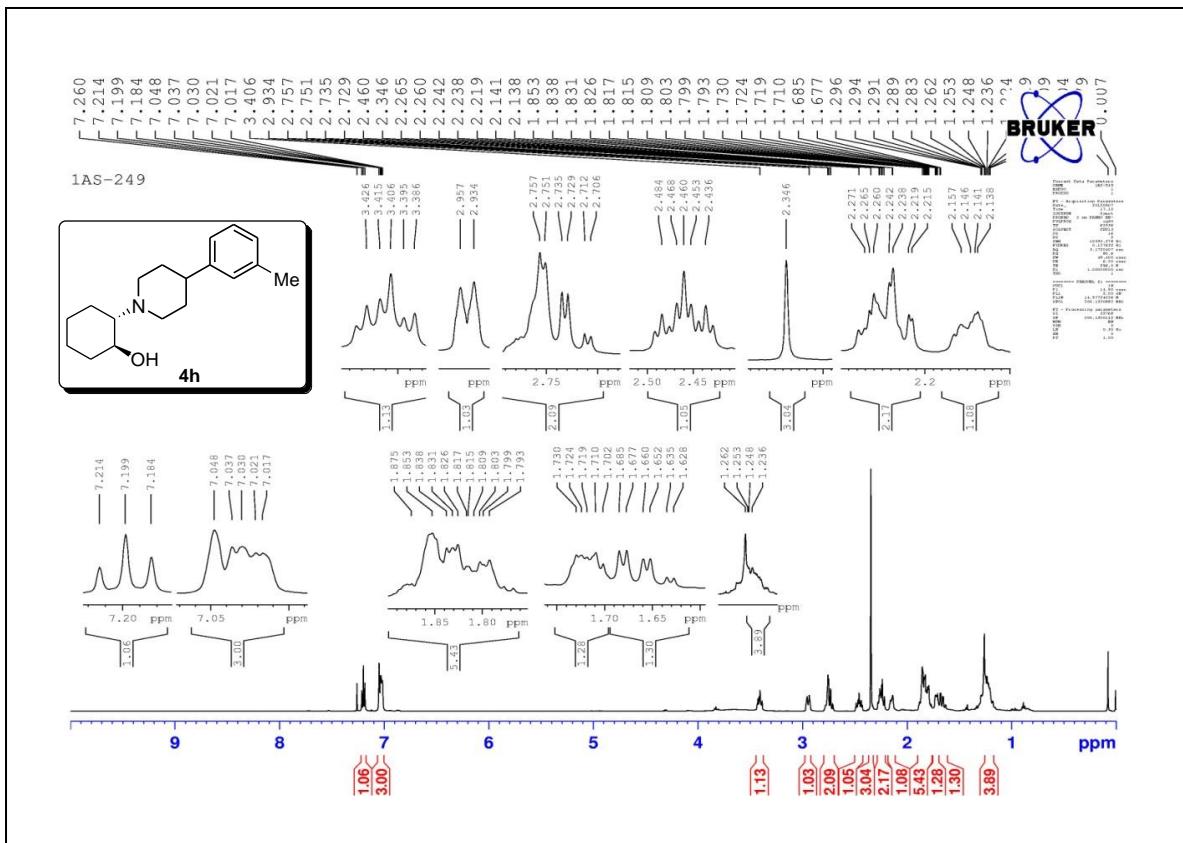


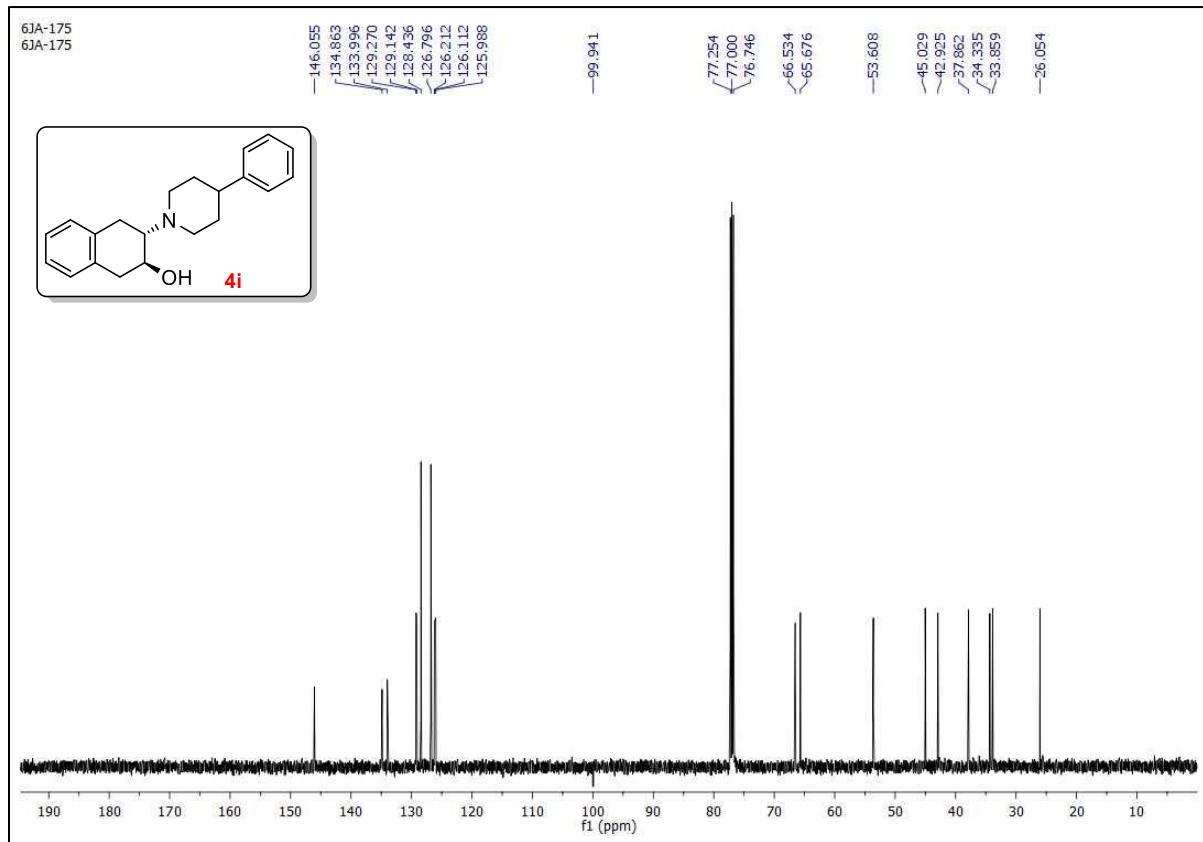
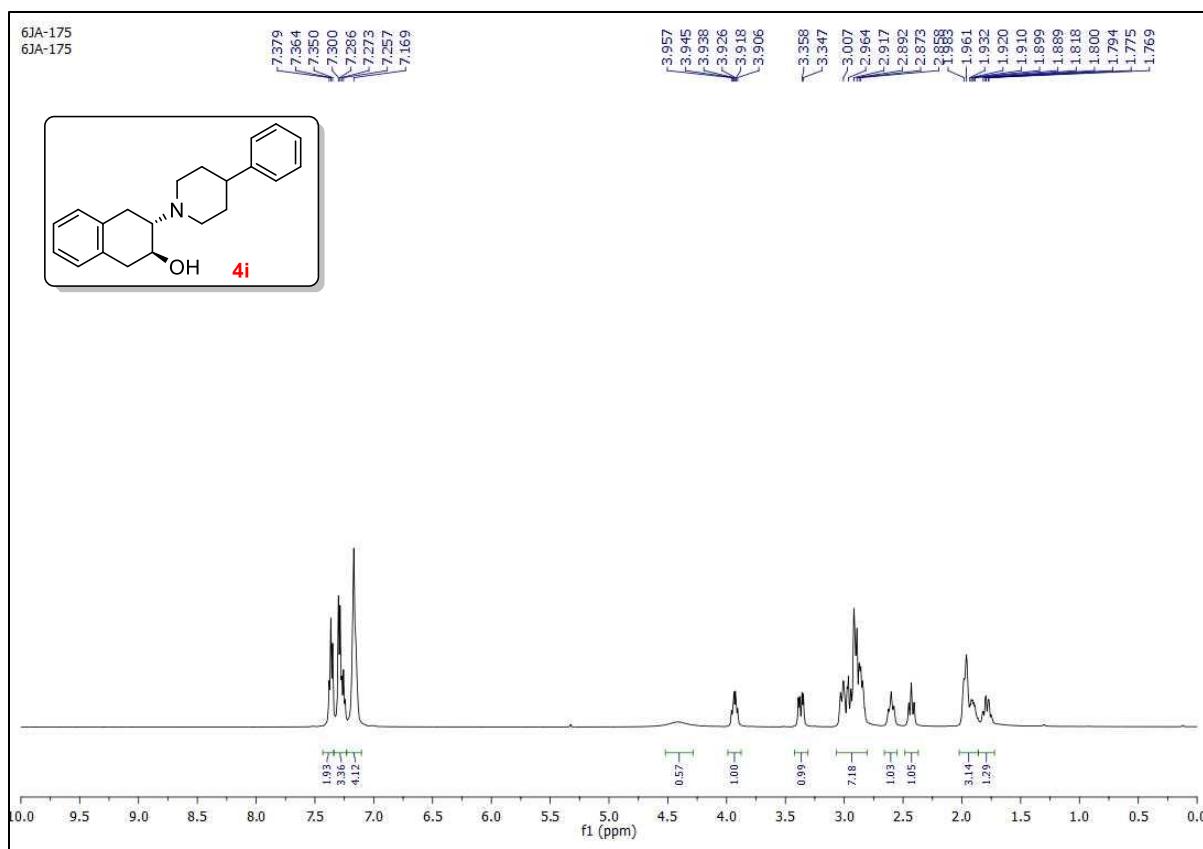
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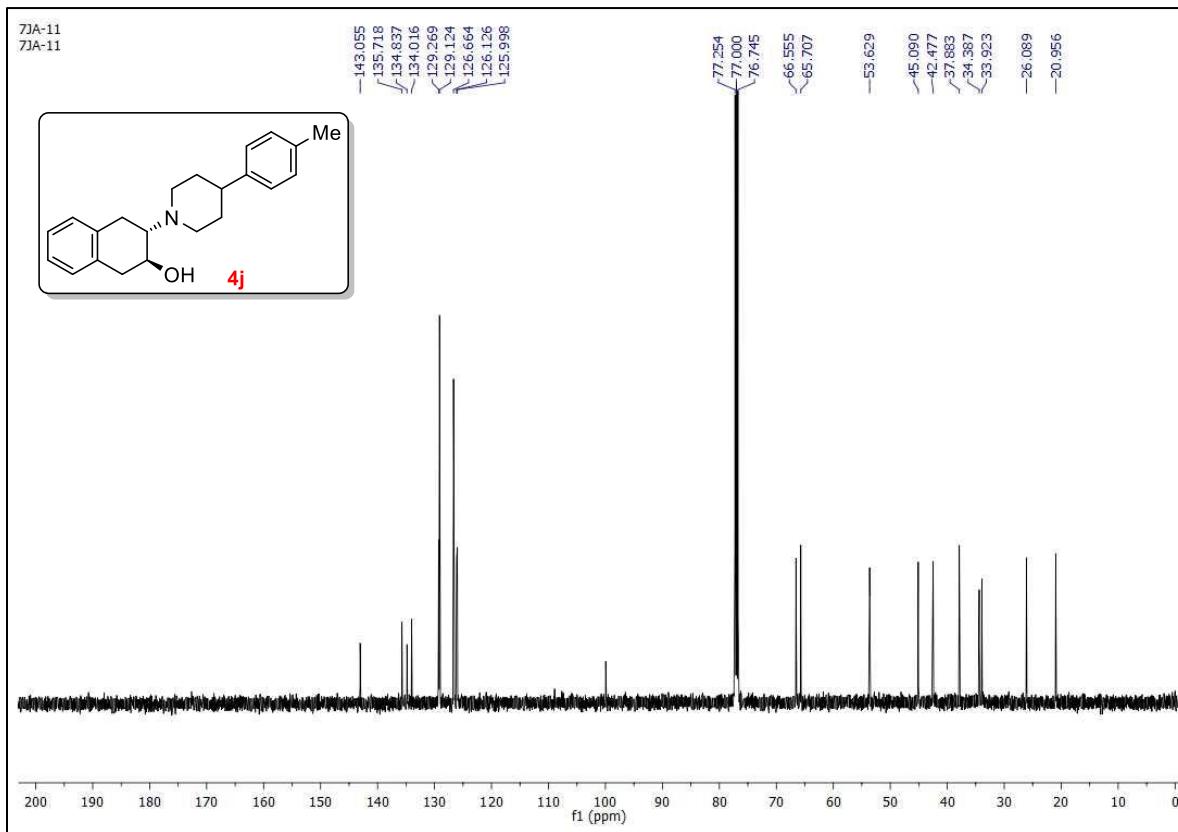
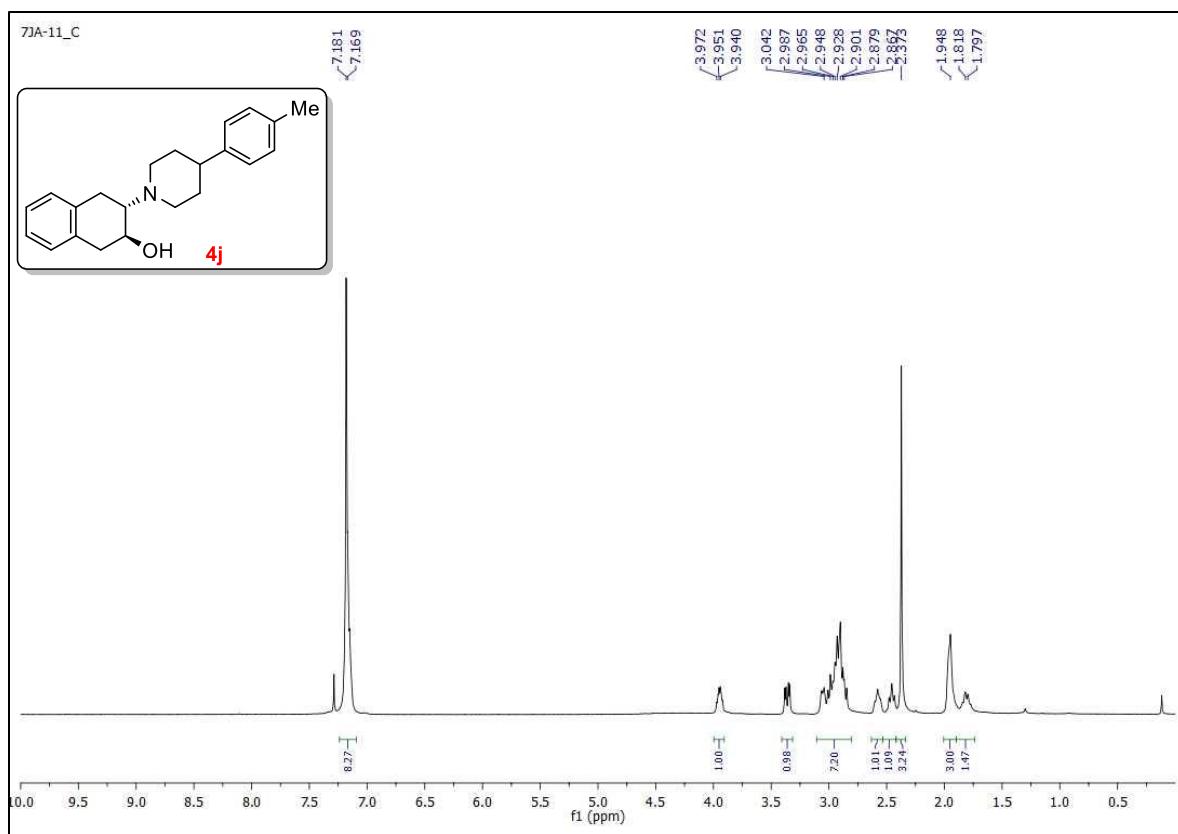


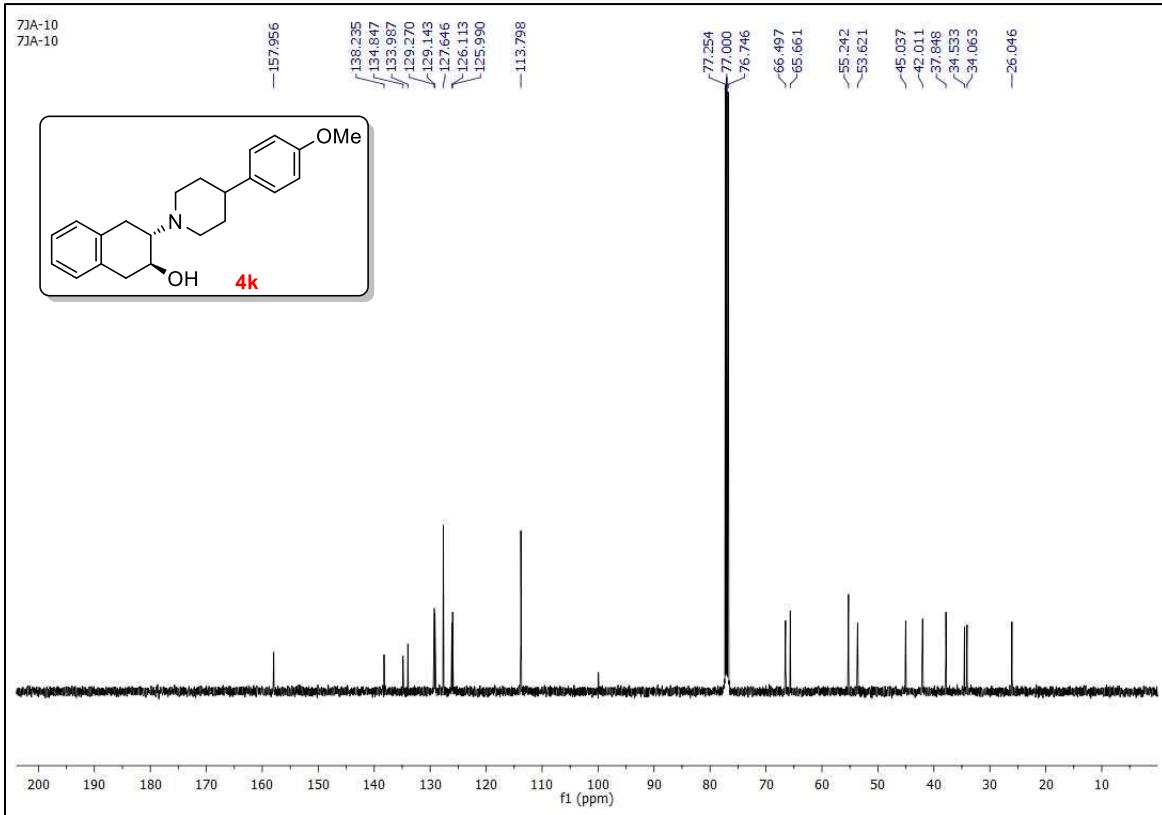
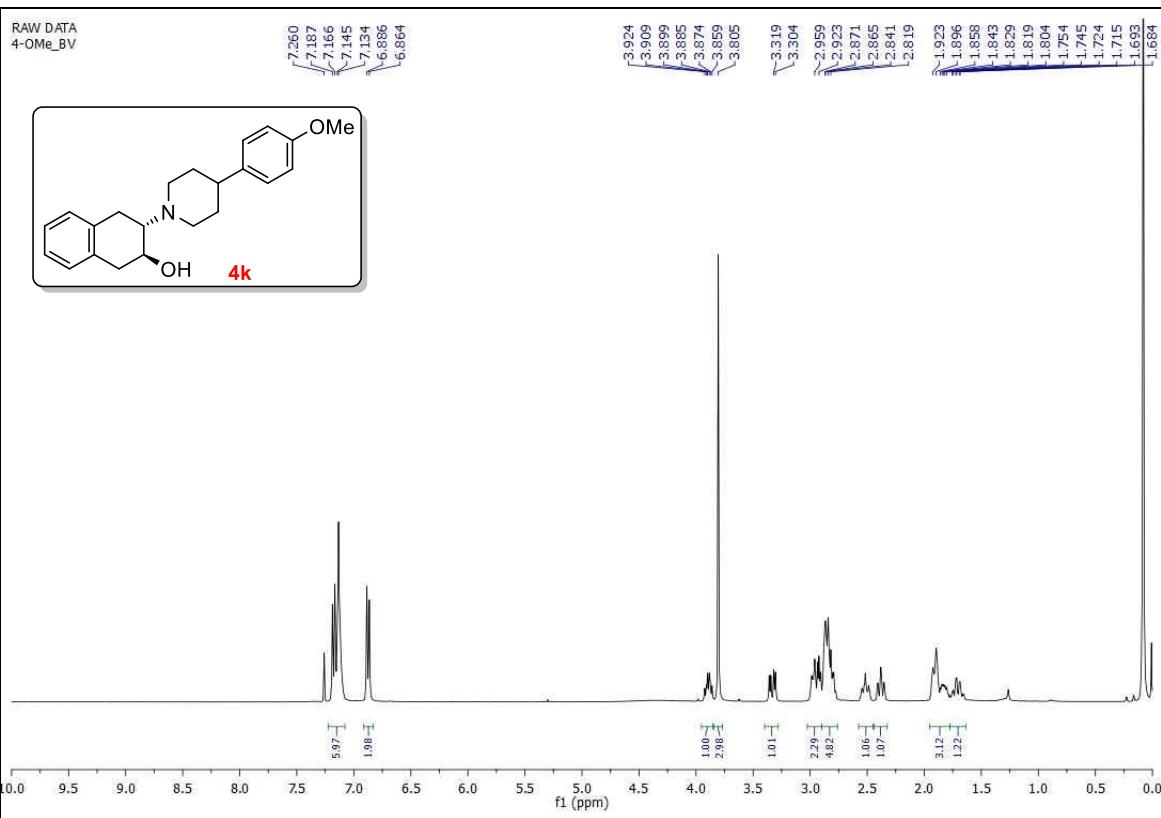


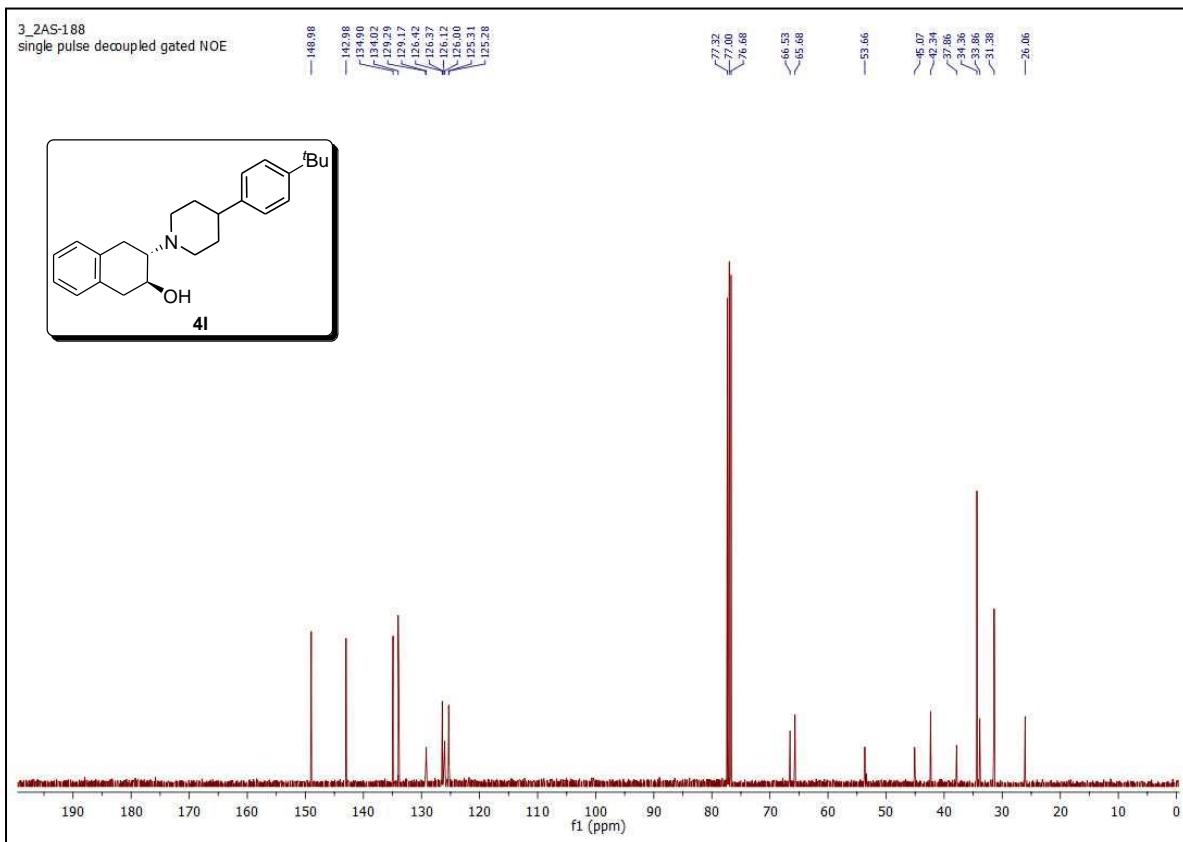
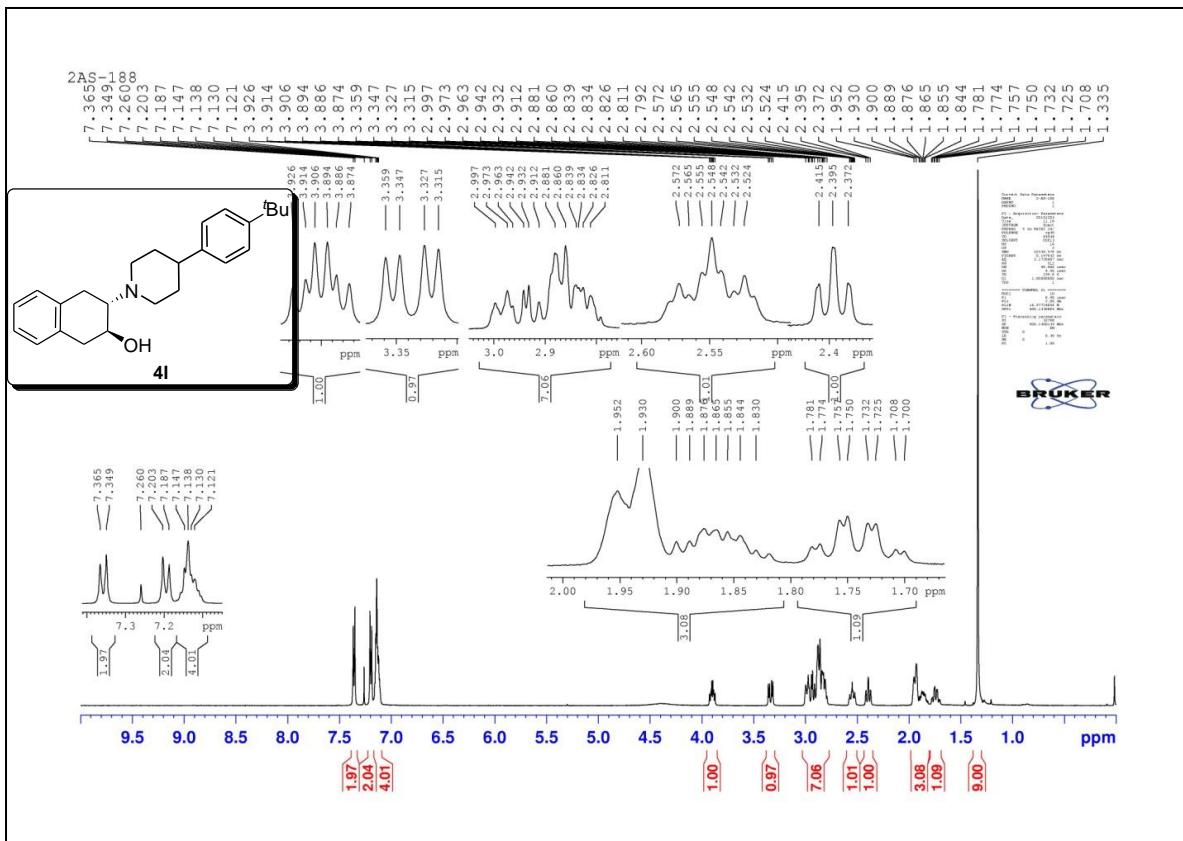


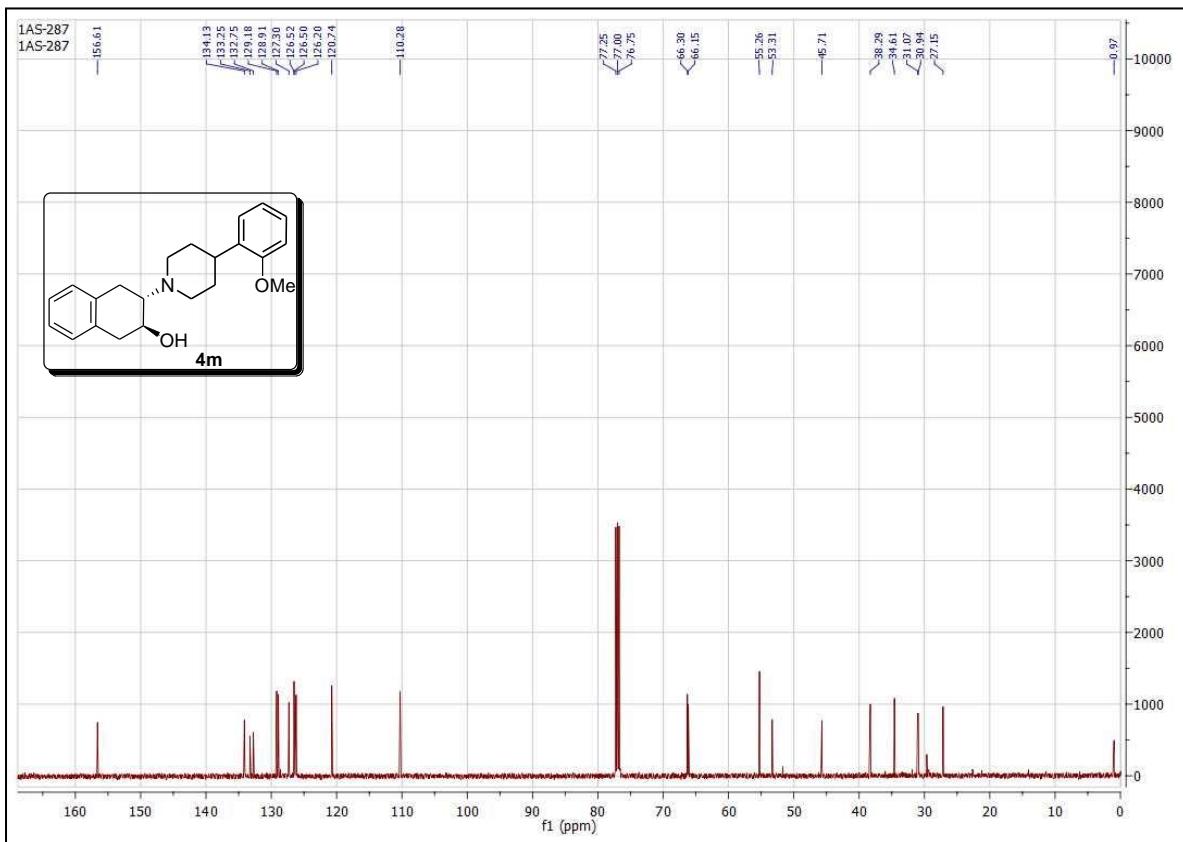
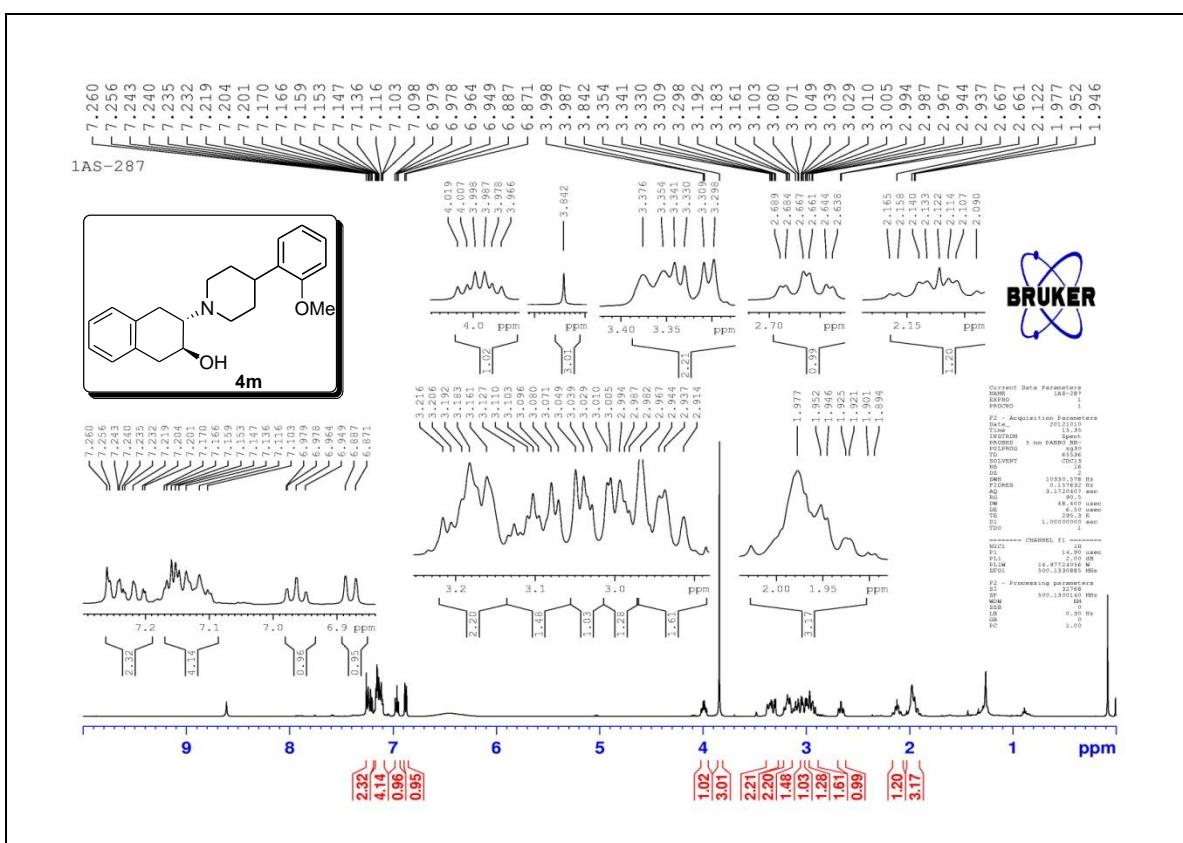


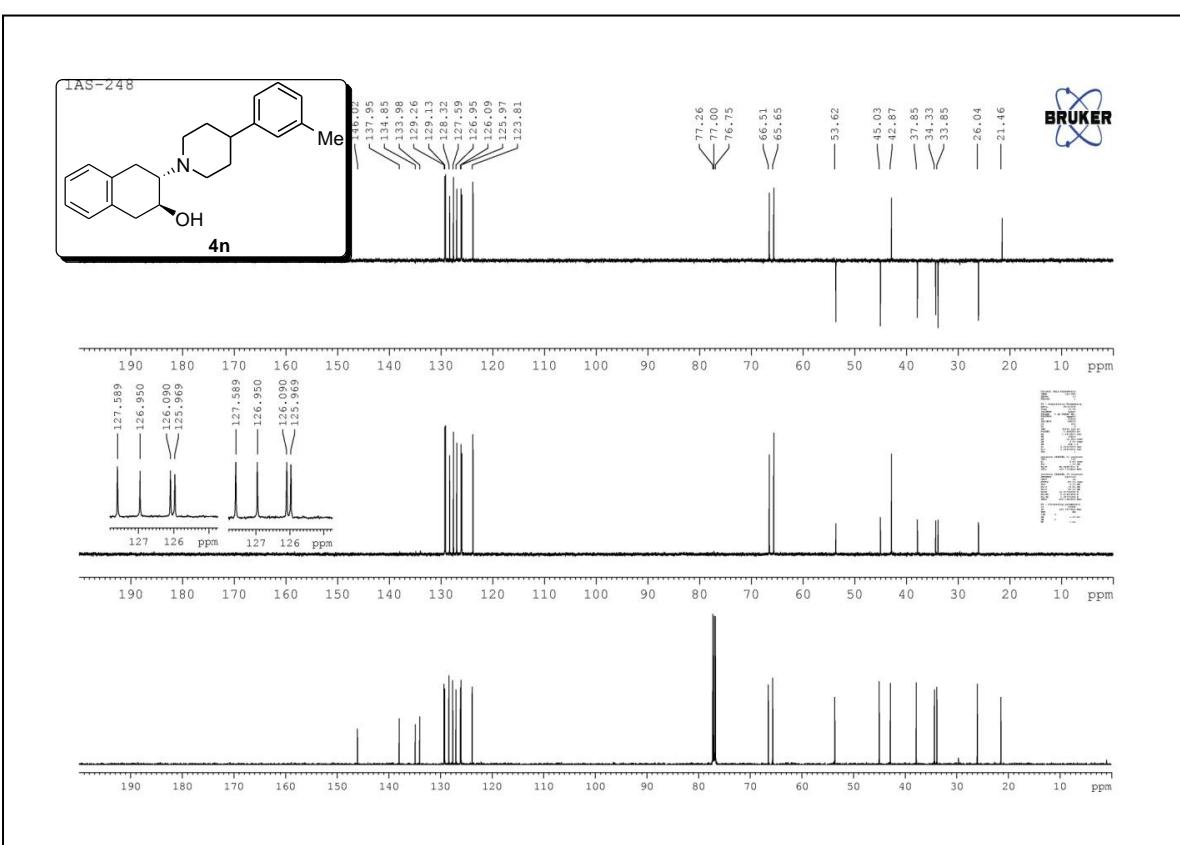
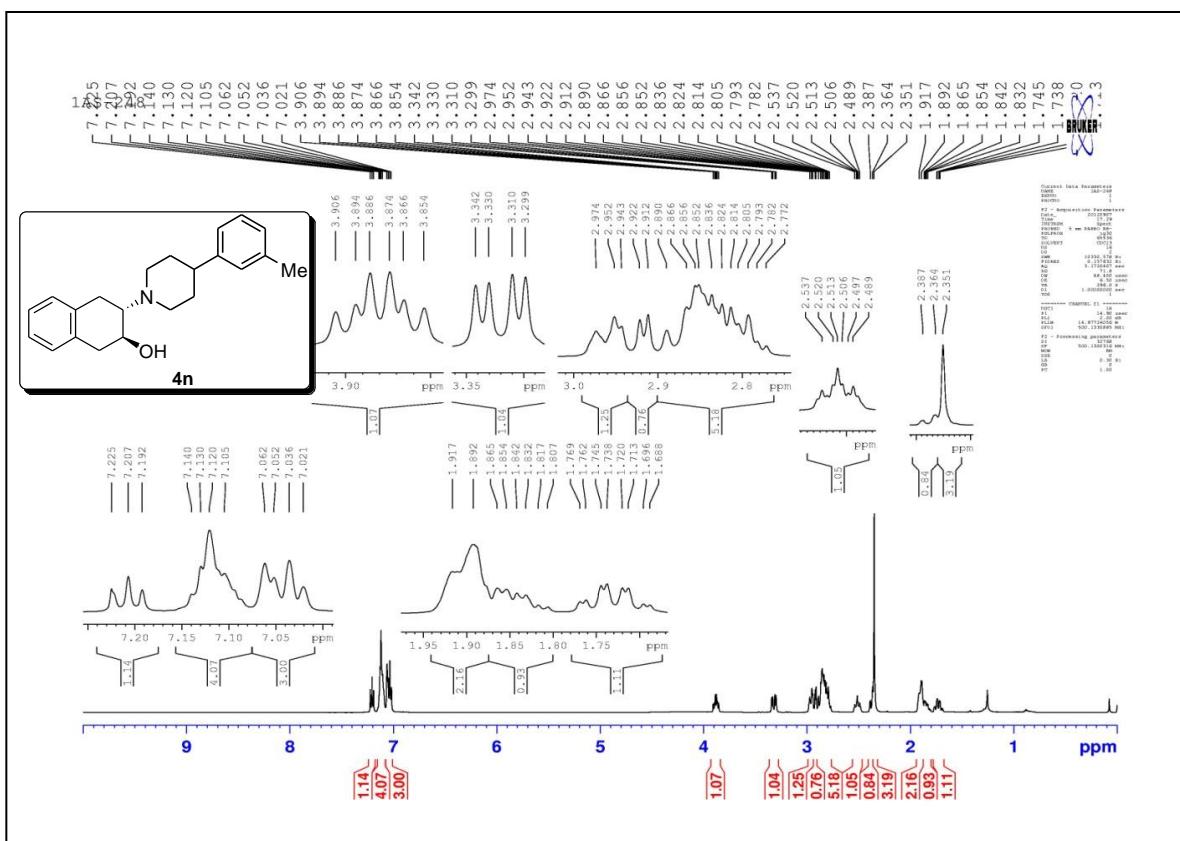


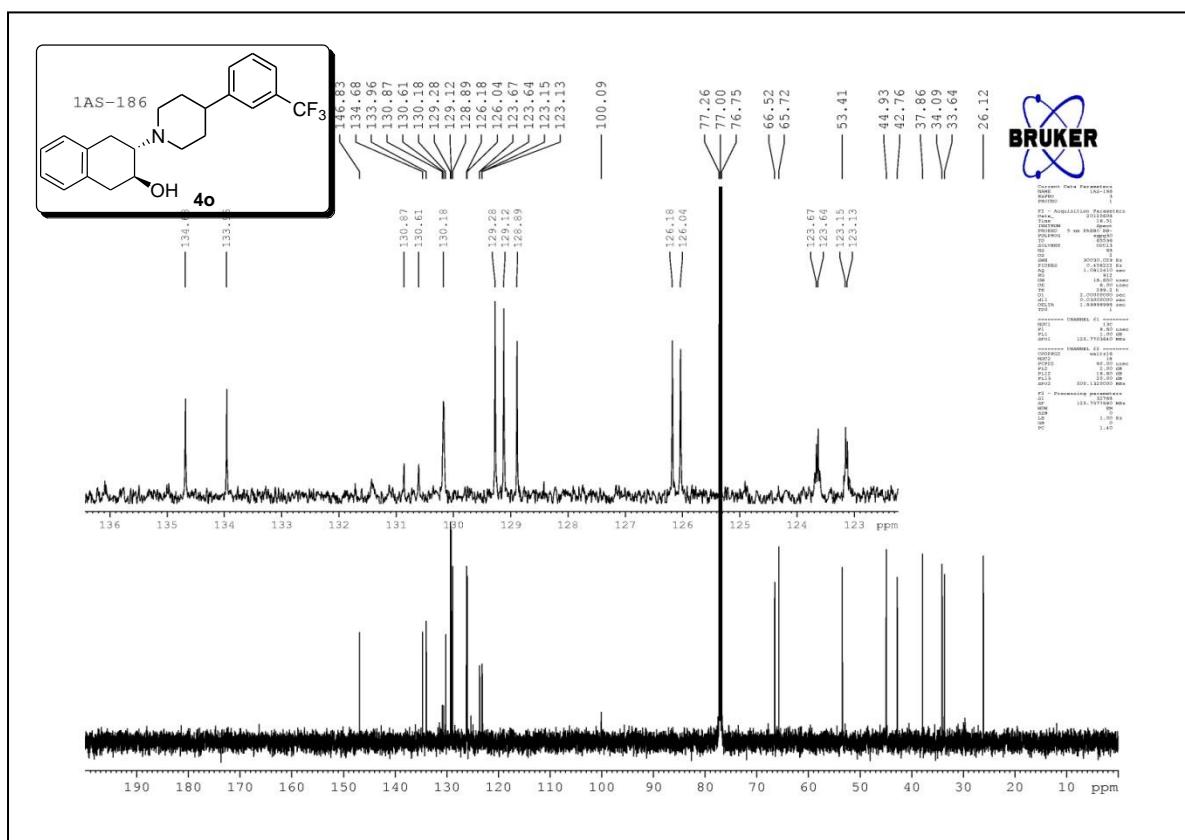
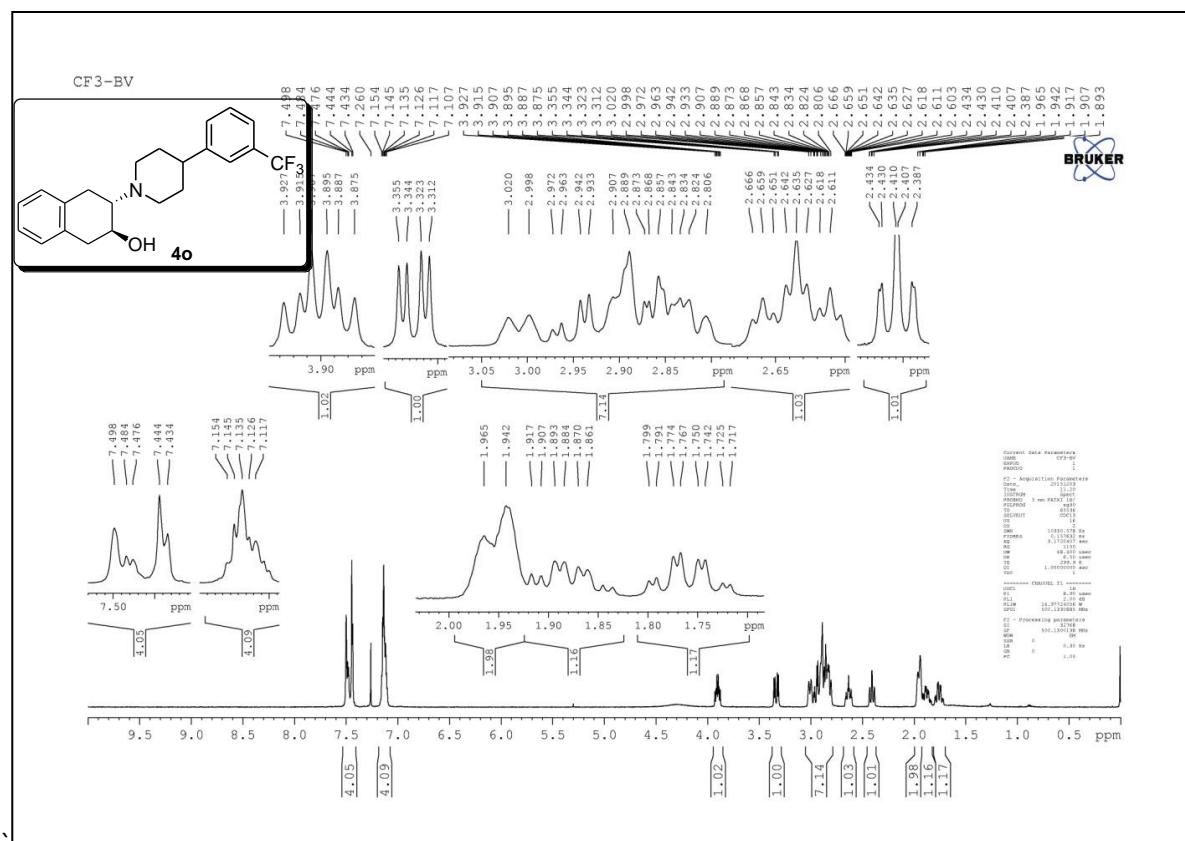


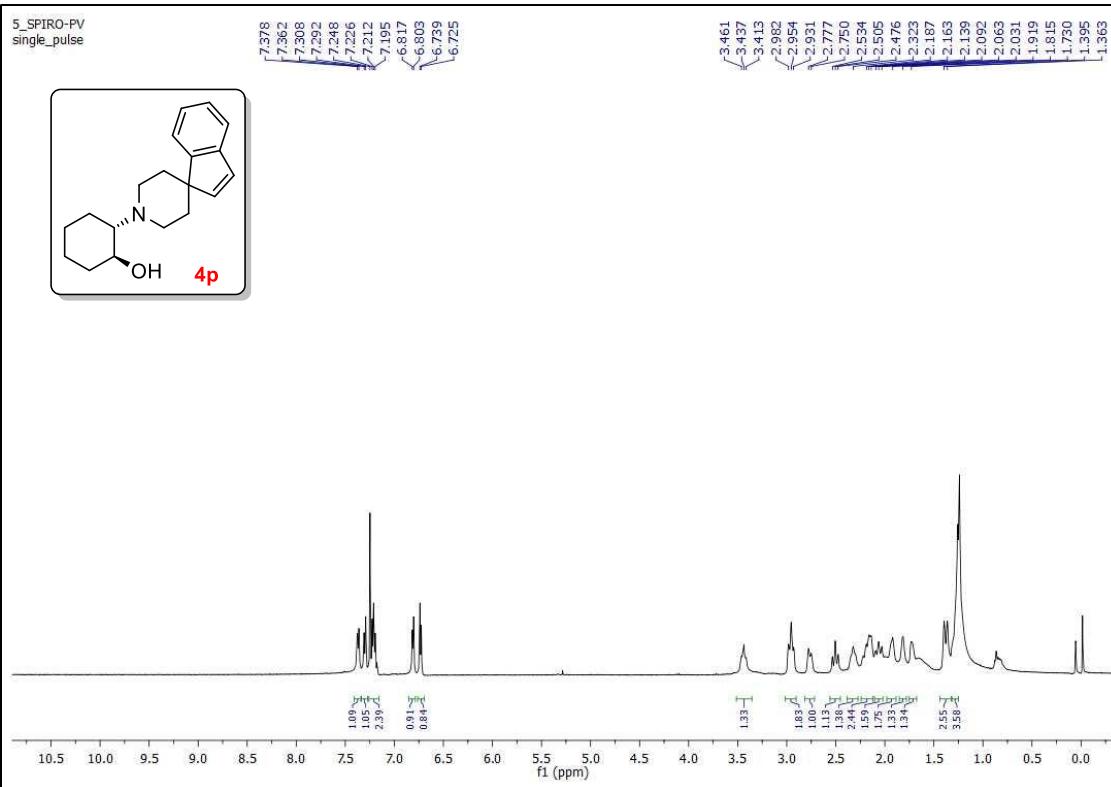




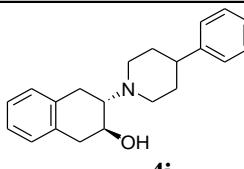
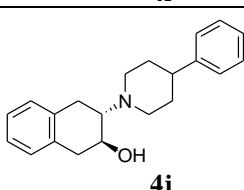
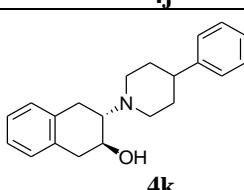
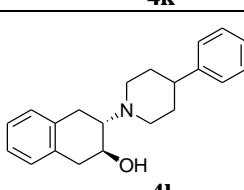
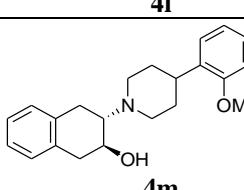
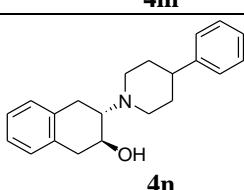
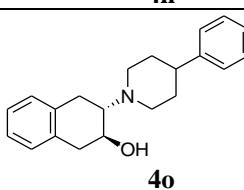
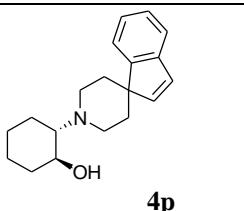


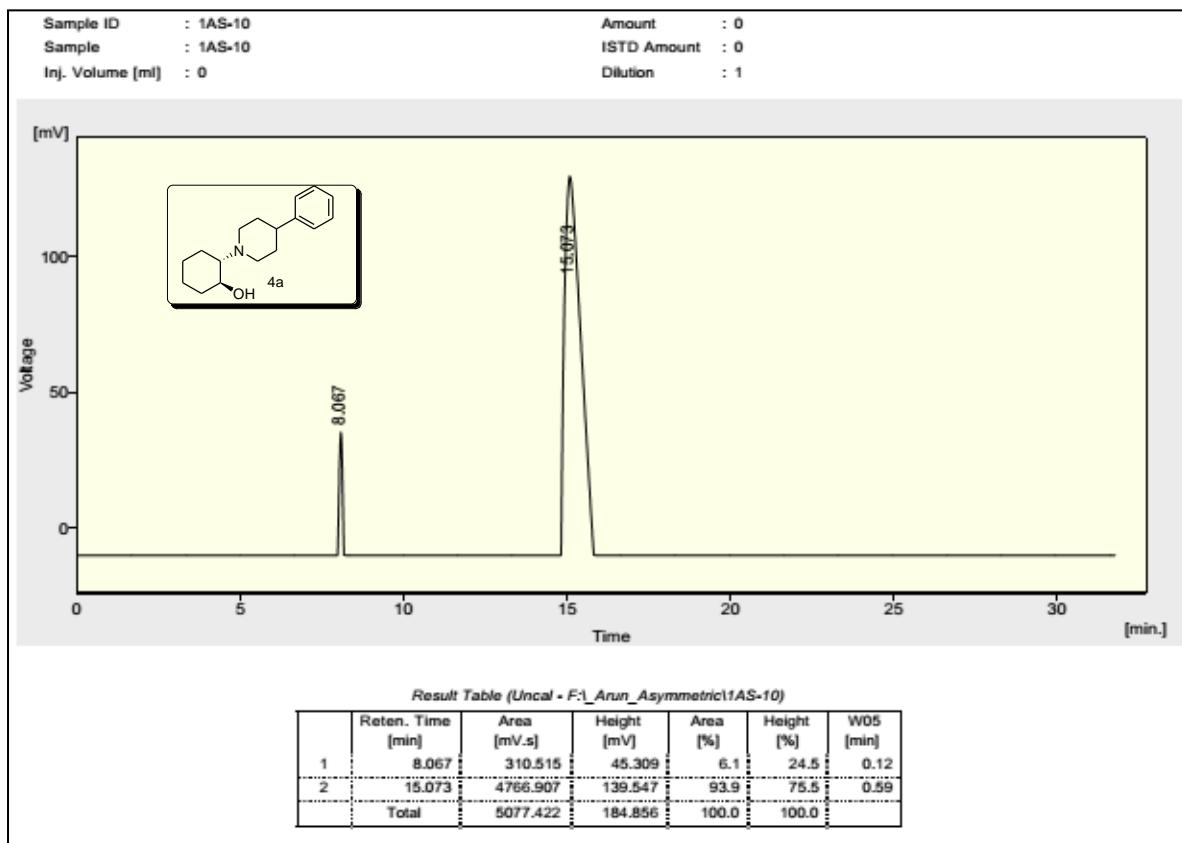
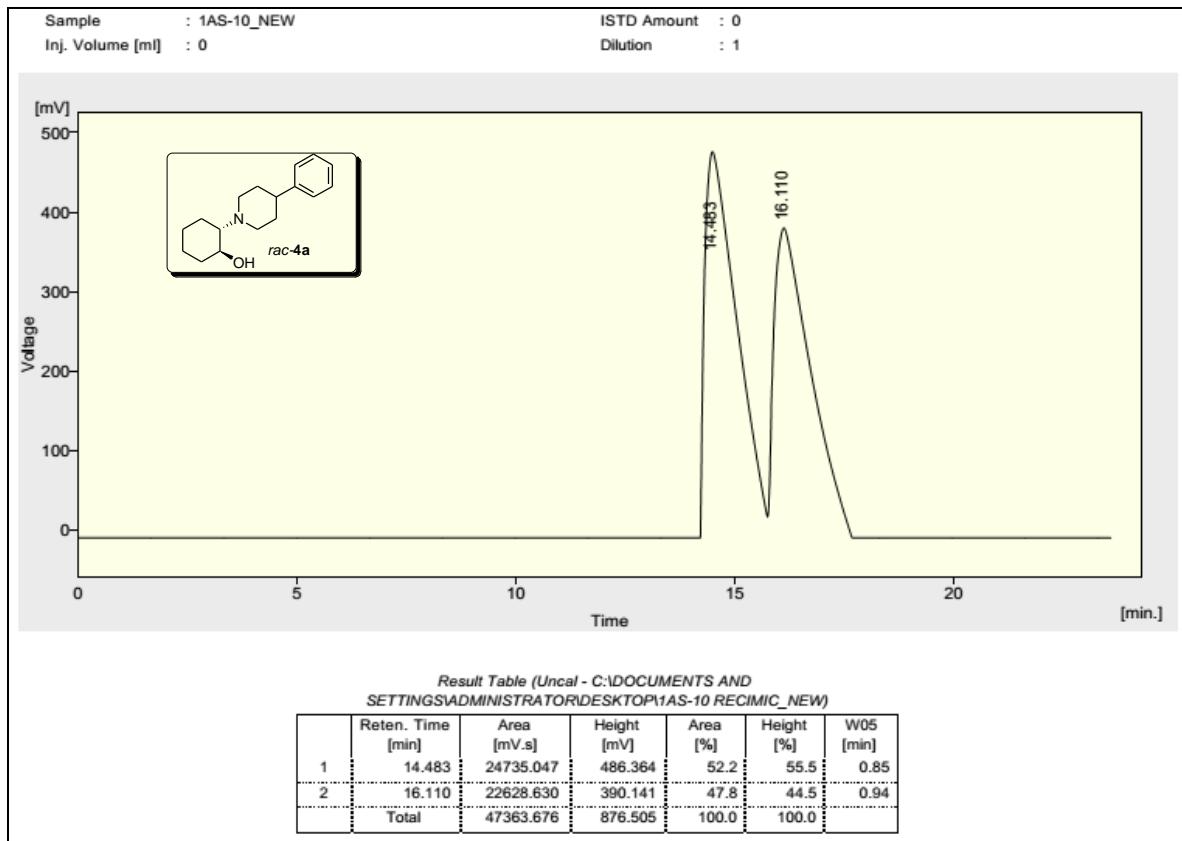


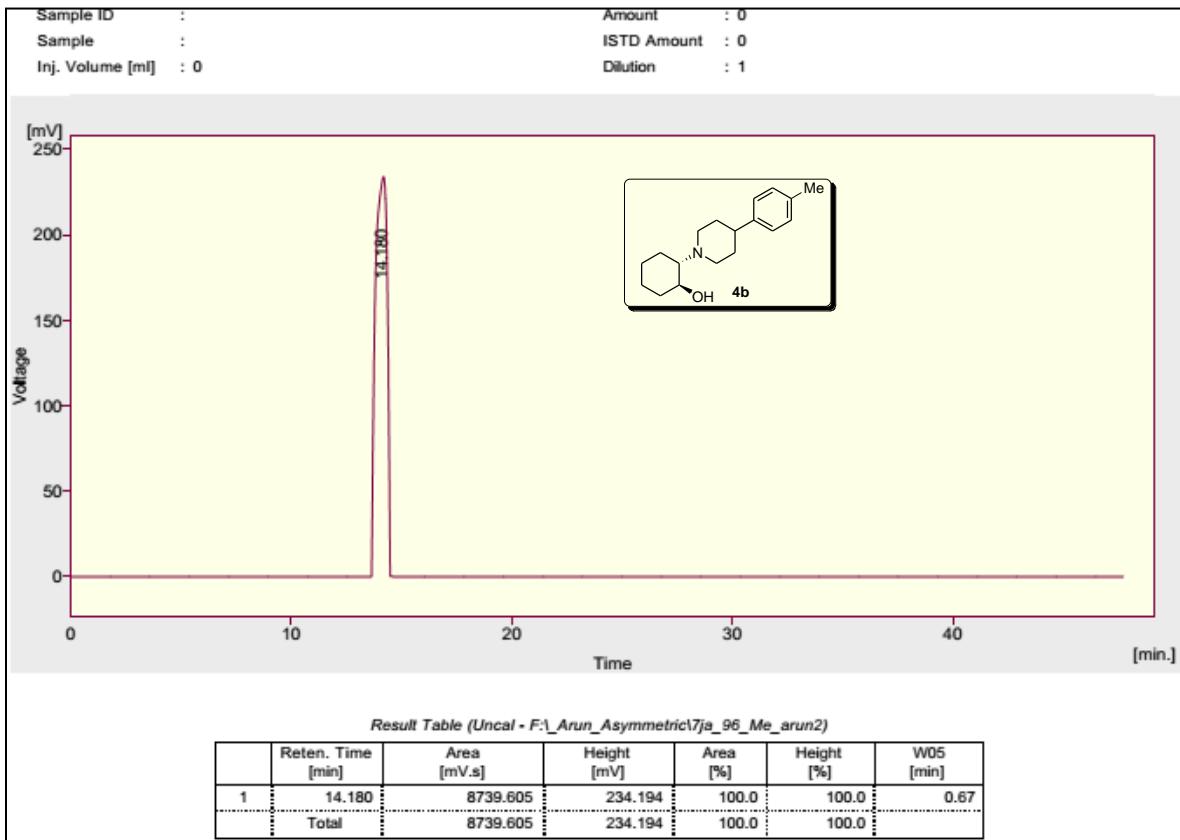
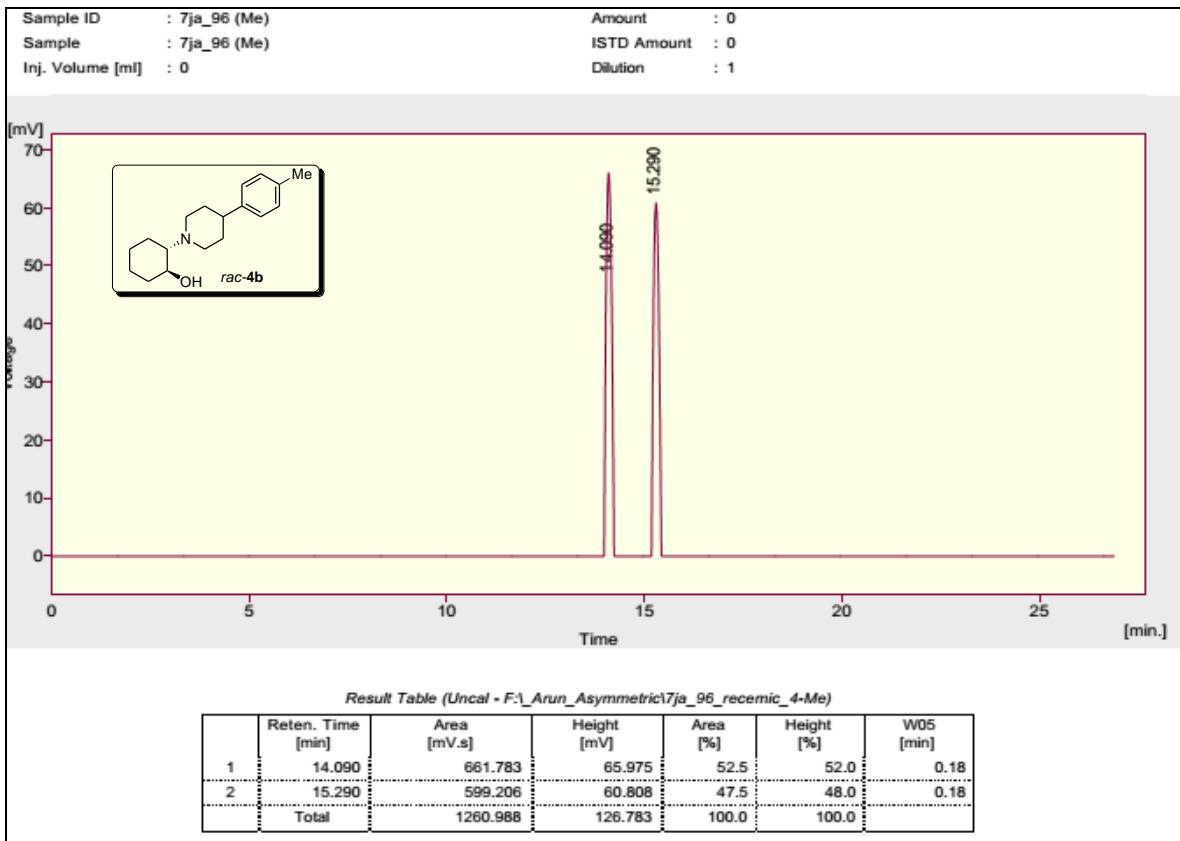


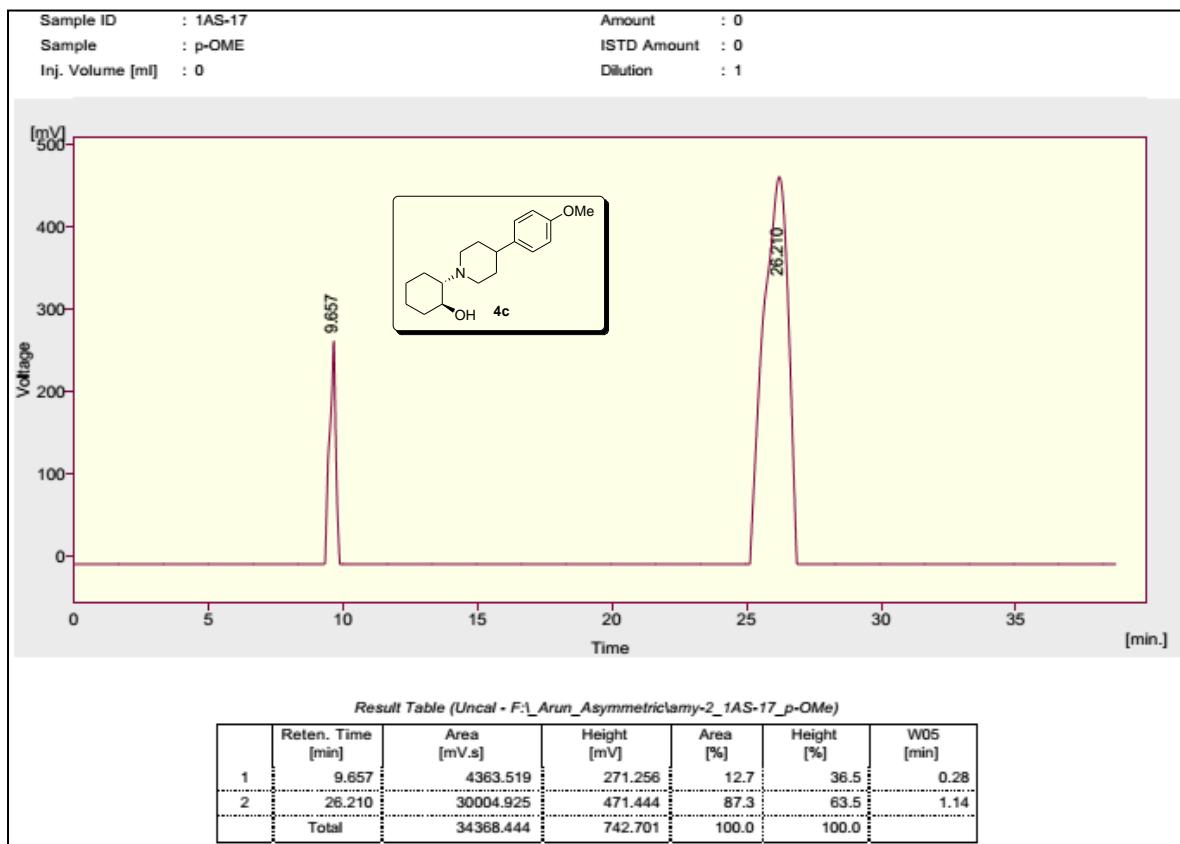
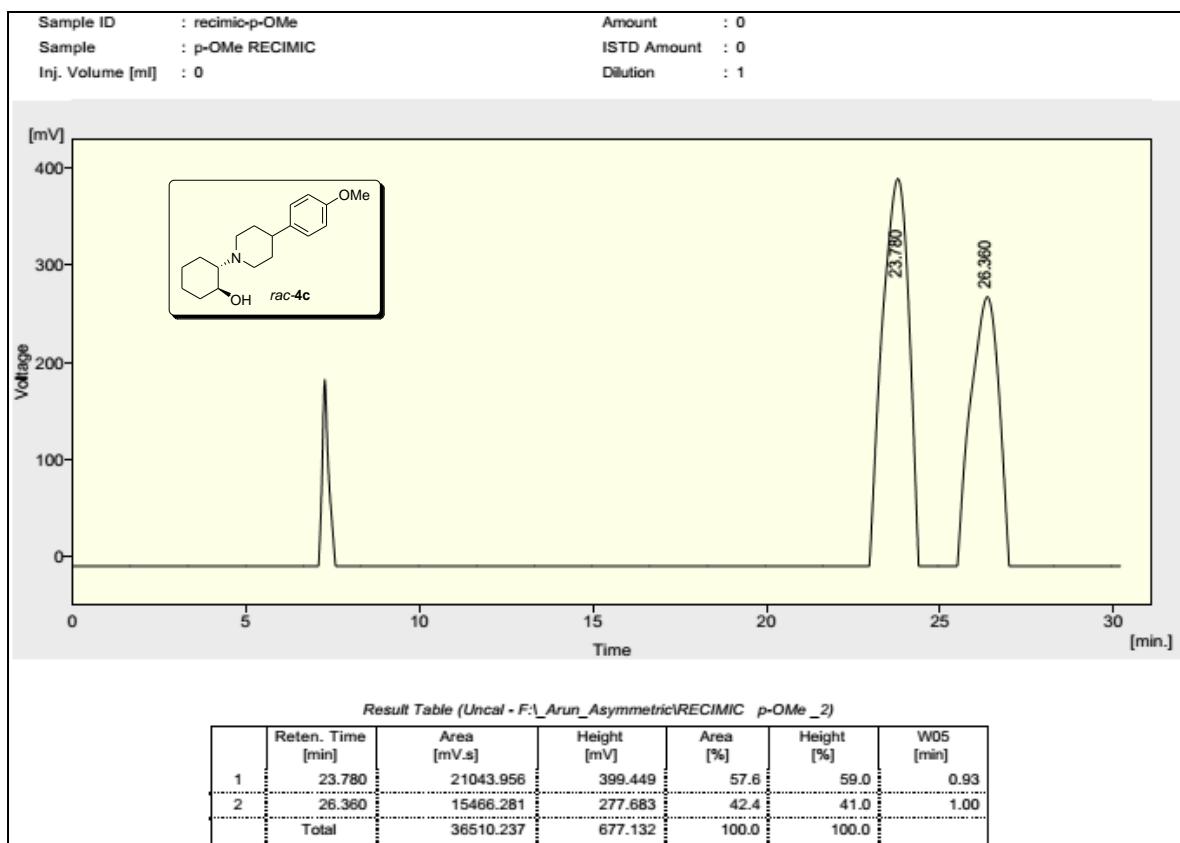


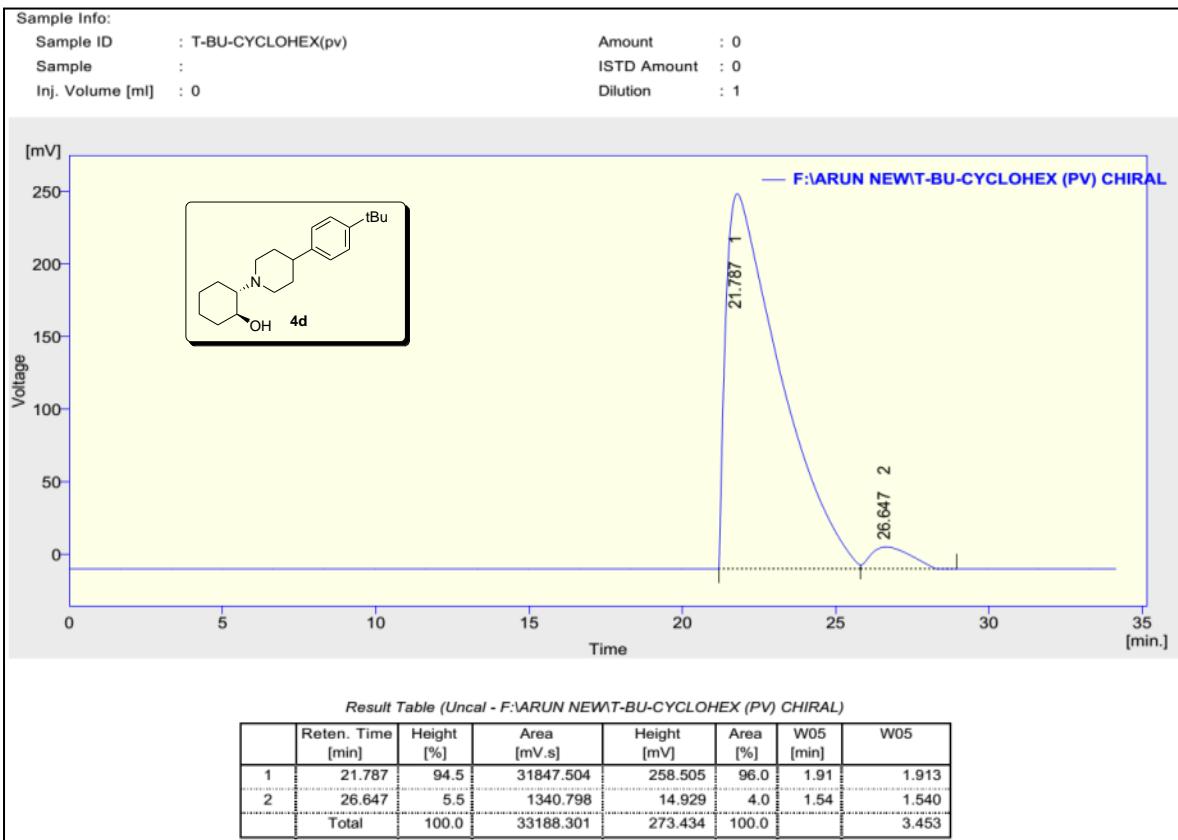
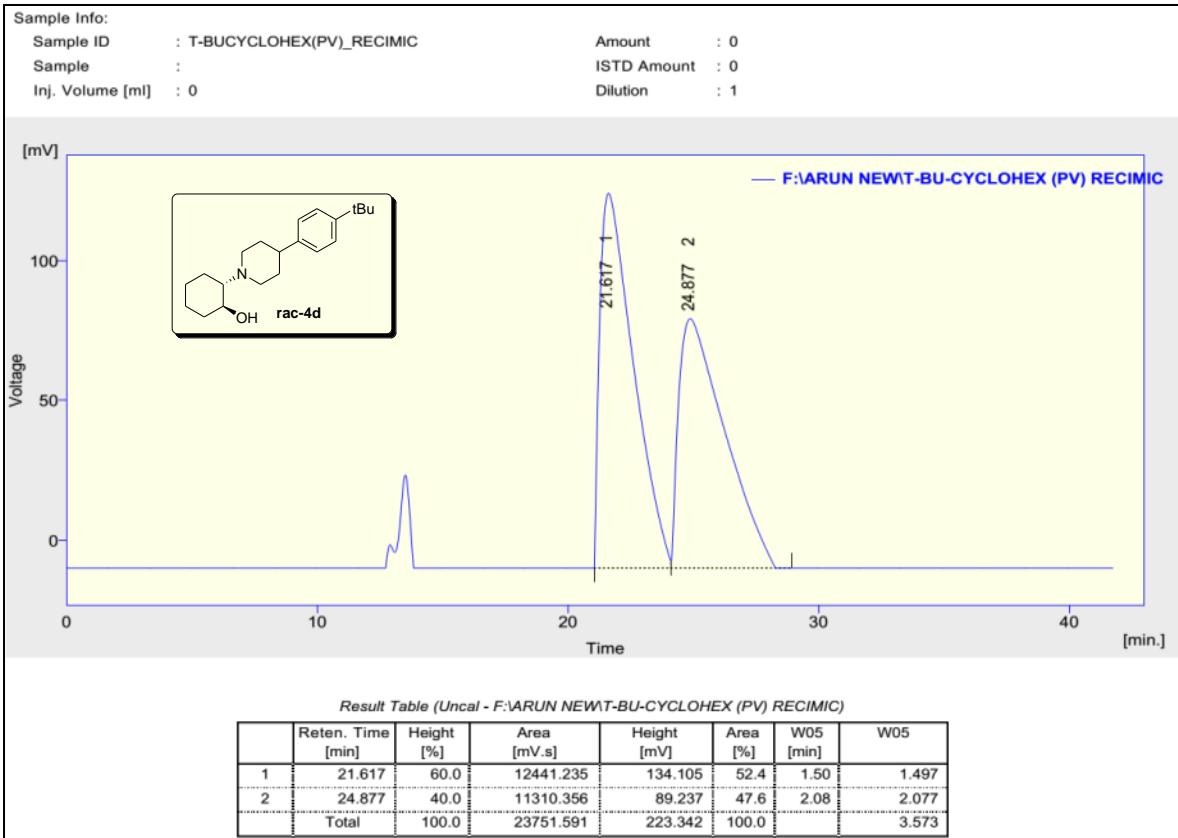
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			Column	Hex:IPA	flow rate mL/min	t _{R1}	t _{R2}
1		>99:1	AD-H	95:5	0.5 mL/min	14.4	16.1
2		>99:1	Amylose-2	97:3	0.5 mL/min	14.0	15.2
3		>99:1	Cellulose-2	97:3	0.5 mL/min	23.7	26.3
4		96:4	AD-H	95:5	0.3 mL/min	21.6	24.8
5		81:19	AD-H	90:10	0.5 mL/min	32.9	41.7
6		85:15	OD-H	98:2	0.5 mL/min	36.5	42.1
7		94:6	Cellulose-2	95:5	0.5 mL/min	15.6	18.6
8		92.5:7.5	Amylose-2	95:5	0.5 mL/min	15.4	17.5

S. No.	Product	Er	HPLC data				
			Column	Hex:IPA	flow rate mL/min	t _{R1}	t _{R2}
9		96:4	OD-H	95:5	0.5 mL/min	25.4	27.9
10		90:10	Cellulose-2	90:10	1.0 mL/min	6.7	8.1
11		>99:1	OD-H	97:3	0.4 mL/min	37.1	42.8
12		96:4	Cellulose-2	95:5	0.5 mL/min	12.4	16.1
13		99:1	AD-H	95:5	0.5 mL/min	21.0	23.2
14		>99:1	AD-H	95:5	0.5 mL/min	15.4	17.3
15		88:12	Cellulose-2	95:5	0.5 mL/min	18.7	26.5
16		>99:1	OD-H	99:1	0.4 mL/min	30.5	48.3





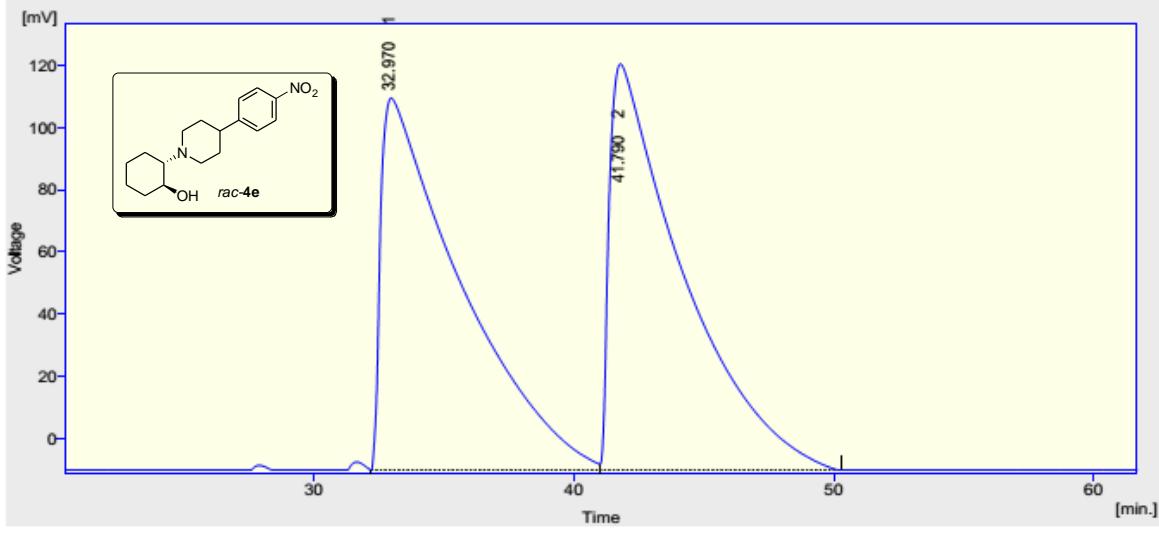




Sample Info:

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 Inj. Volume [ml] : 0

Amount : 0
 ISTD Amount : 0
 Dilution : 1

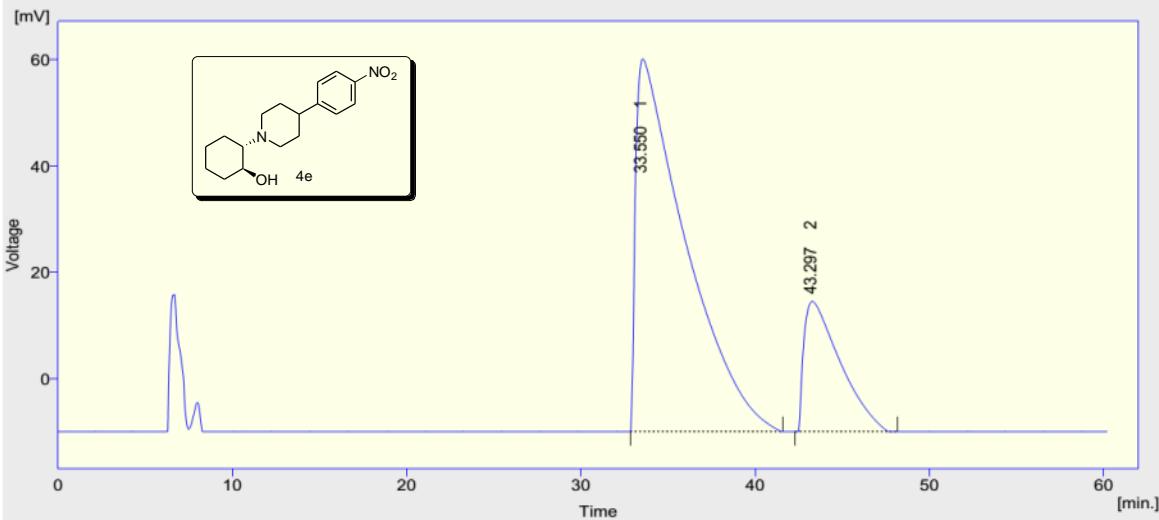
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	Reten. Time [min]	Height [%]	Area [mV.s]	Height [mV]	Area [%]	W05 [min]	W05
1	32.970	47.8	25442.995	119.436	50.0	3.16	3.160
2	41.790	52.2	25427.455	130.363	50.0	2.83	2.830
Total		100.0	50870.451	249.798	100.0		5.990

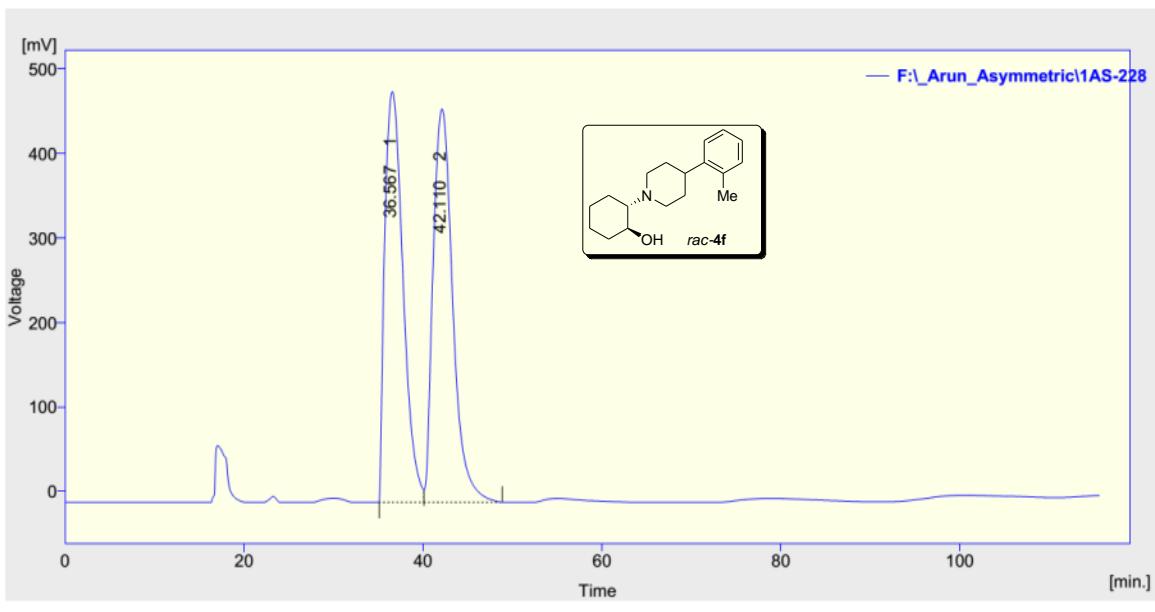
Sample Info:

Sample ID : A-4-NO₂-CHIRAL-2.
 Sample :
 Inj. Volume [ml] : 0

Amount : 0
 ISTD Amount : 0
 Dilution : 1

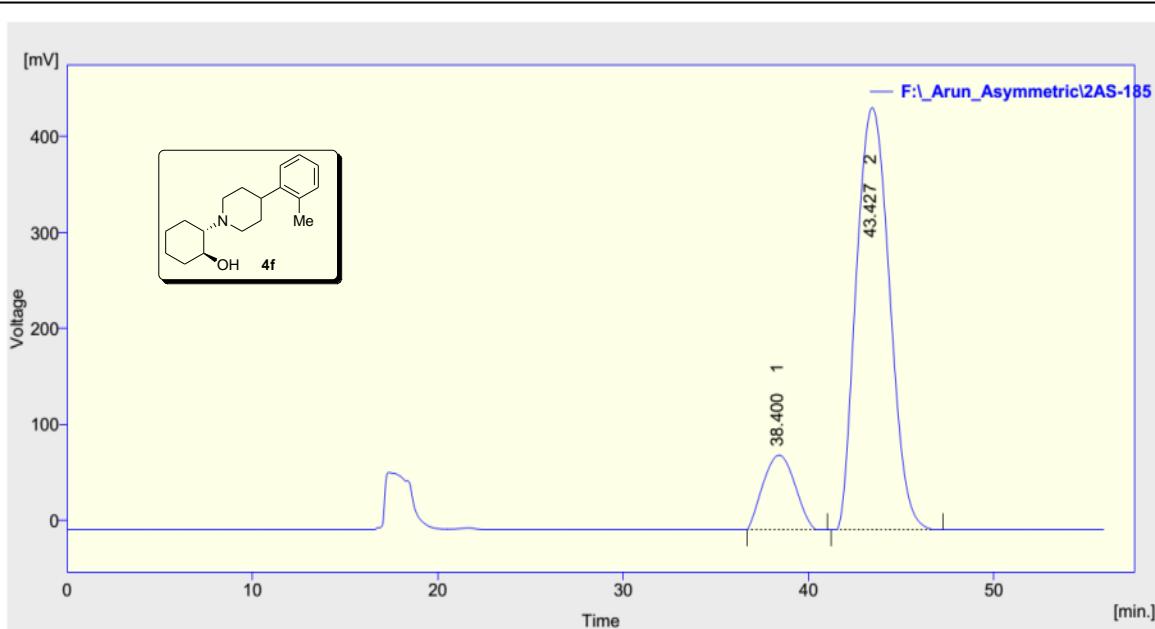
Result Table (Uncal - F:\arun\water\A-4-NO₂-CHIRAL-2.)

	Reten. Time [min]	Height [%]	Area [mV.s]	Height [mV]	Area [%]	W05 [min]	W05
1	33.550	74.1	13927.099	70.161	79.7	2.99	2.990
2	43.297	25.9	3542.199	24.469	20.3	2.30	2.303
Total		100.0	17469.297	94.630	100.0		5.293



Result Table (Uncal - F:\Arun_Asymmetric\1AS-228)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	36.567	68855.968	486.469	49.2	51.1	2.28
2	42.110	71057.467	465.940	50.8	48.9	2.36
Total		139913.436	952.409	100.0	100.0	



Result Table (Uncal - F:\Arun_Asymmetric\2AS-185)

	Reten. Time [min]	Area [mV.s]	Height [mV]	Area [%]	Height [%]	W05 [min]
1	38.400	9314.632	77.924	14.5	15.0	2.01
2	43.427	54796.318	440.304	85.5	85.0	2.03
Total		64110.950	518.229	100.0	100.0	

