

Visible-Light-Induced Transition-Metal and Photosensitizer Free Decarbonylative Addition of Amino-Arylaldehydes to Ketones

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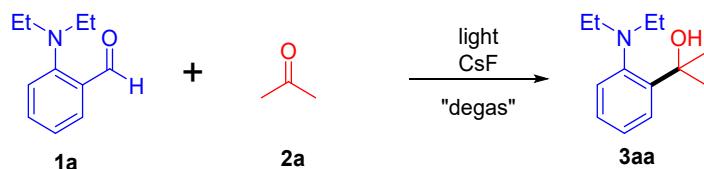
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All reagents and solvents were purchased from commercial sources and used without further purification unless otherwise stated. All reactions were monitored by thin-layer chromatography (TLC). All reactions were carried out in argon atmosphere unless otherwise stated. Column chromatography was performed on silica gel (200-300 mesh) and visualized with ultraviolet light. Ethyl acetate and petroleum ether were used as eluents. ^1H , ^{13}C and ^{19}F NMR spectra were recorded at room temperature on Varian Mercury plus 300, Bruker AV400 and Agilent INOVA 600 MHz with TMS as an internal standard and CDCl_3 or CD_3OD as solvent. Fourier transform infrared spectra (FT-IR) were recorded on Agilent Technologies Cary 630 instrument or Bruker TENSOR 27 instrument. HRMS analyses were made by Lanzhou University by means of ESI. Melting points were measured on SGWX-4 micro melting point apparatus and uncorrected.

2. Optimizing reaction conditions(1) Effects of base on yields^a

Entry	Base	Yield ^b /%
1	-	70
2	K_2CO_3	67
3	NaOH	n.p.
4	Cs_2CO_3	75
5	CsF	88
6	DIPEA	70
7	Pyridine	70
8	DMAP	85

^aGeneral conditions: **1a** (0.1 mmol) and base (1.0 equiv.) in acetone (1.0 mL) were irradiated by 405 nm light for 24 h under argon atmosphere. ^bYields were determined by ^1H NMR using nitromethane as an internal standard.

(2) Effects of Cs⁺ sources on yields^a

Entry	[Cs ⁺]	Yield ^b /%
1	CsBr	76
2	CsI	80
3	CsAc	50
4	CsF	88

^aGeneral conditions: **1a** (0.1 mmol) and [Cs⁺] (1.0 equiv.) in acetone (1.0 mL) were irradiated by 405 nm light for 24 h under argon atmosphere. ^bYields were determined by ¹H NMR using nitromethane as an internal standard.

(3) Effects of F⁻ sources on yields^a

Entry	[F ⁻]	Yield ^b /%
1	LiF	68
2	KF	76
3	ZnF ₂	67
4	CsF	88

^aGeneral conditions: **1a** (0.1 mmol) and [F⁻] (1.0 equiv.) in acetone (1.0 mL) were irradiated by 405 nm light for 24 h under argon atmosphere. ^bYields were determined by ¹H NMR using nitromethane as an internal standard.

(4) The effects of the amount of CsF on yields^a

Entry	Conditions	Yield ^b /%
1	0.5 equiv.	88
2	1.0 equiv.	88
3	2.0 equiv.	86

^aGeneral conditions: **1a** (0.1 mmol) and CsF (X equiv.) in acetone (1.0 mL) were irradiated by 405 nm light for 24 h under argon atmosphere. ^bYields were determined by ¹H NMR using nitromethane as an internal standard.

(5) Effects of light on yields^a

Entry	Light	Yield ^b /%
1	380 nm	83
2	425 nm	41
3	465 nm	40
4	dark	n. r.

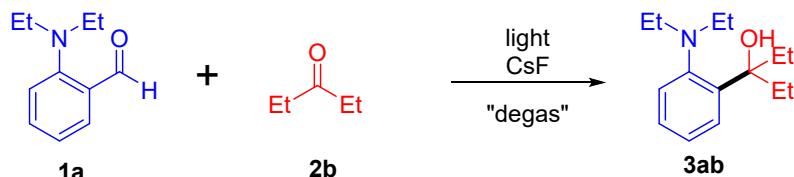
^aGeneral conditions: **1a** (0.1 mmol) and CsF (0.5 equiv.) in acetone (1.0 mL) were irradiated by light for 24 h under argon atmosphere. ^bYields were determined by ¹H NMR using nitromethane as an internal standard.

(6) Effects of reaction time on yields^a

Entry	Time	Yield ^b /%
1	4 h	65
2	8 h	78
3	12 h	89 (83)

^aGeneral conditions: **1a** (0.1 mmol) and CsF (0.5 equiv.) in acetone (1.0 mL) were irradiated by 405 nm light under argon atmosphere. ^bYields were determined by ¹H NMR using nitromethane as an internal standard; isolated yields were shown in parentheses.

Evaluation of various conditions with 3-pentanone.



(1) Effects of solvent on yields^a

Entry	Solvent	Yield ^b /%
1	cyclohexane	56
2	THF	61
3	toluene	73
4	1,4-dioxane	47
5	HFIP	n.r.
6	Acetonitrile	n.p.
7	CH ₃ OH	n.r.

^aGeneral conditions: **1a** (0.1 mmol), **2b** (5.0 equiv.), CsF (0.5 equiv.) and solvent (1 mL) for 12 h under argon atmosphere, light (405 nm). ^bYields were determined by ¹H NMR using nitromethane as an internal standard.

(2) Effects of **2b** amount on yields^a

Entry	2b	Yield ^b /%
1	2.0 equiv.	53
2	5.0 equiv.	73
3	8.0 equiv.	87 (77)
3	10.0 equiv.	82

^aGeneral conditions: **1a** (0.1 mmol), **2b**, CsF (0.5 equiv.) and toluene (1 mL) for 12 h under argon atmosphere, light (405 nm). ^bYields were determined by ¹H NMR using nitromethane as an internal standard.

3. General procedures for preparation of starting materials

i. General procedures for preparation of starting materials **1a~1h**, **1l** and **1o**

The synthesis of starting materials **1a~1h**, **1l** and **1o** were following the general procedure.¹ 2-Fluorobenzaldehyde (1.24 g, 10 mmol), secondary amine (12 mmol), potassium carbonate (1.66 g, 12 mmol) and DMF (10 mL) were added to a 25 mL round-bottom flask and charged with a magnetic stir bar. The reaction mixture was stirred in an oil bath and the bath temperature was slowly increased to the reflux temperature of DMF (ca. 153 °C) and stirred for 24 h. The reaction was monitored by thin-layer chromatography (TLC). After the reaction was completed, the mixture was cooled to room temperature and extracted with ethyl acetate (3 × 20 mL). The combined organic extracts were washed with brine, dried over anhydrous Na₂SO₄, concentrated, and further purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 80: 1, v/v) to afford the corresponding products.



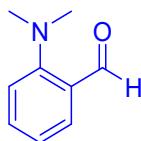
2-(diethylamino)benzaldehyde (1a)

Yellow oil, 88%

¹H NMR (600 MHz, CDCl₃) δ 10.36 (s, 1H), 7.81 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.51 – 7.46 (m, 1H), 7.16 (d, *J* = 8.2 Hz, 1H), 7.11 – 7.07 (m, 1H), 3.19 (q, *J* = 7.1 Hz, 4H), 1.07 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 192.2, 154.6, 134.3, 130.8, 128.9, 122.3, 121.7, 48.9,

12.4. The spectra data were consistent with the literature.¹



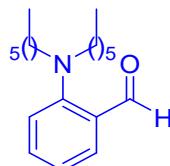
2-(dimethylamino)benzaldehyde (1b)

Yellow oil, 82%

¹H NMR (400 MHz, CDCl₃) δ 10.23 (s, 1H), 7.76 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.49 – 7.43 (m, 1H), 7.07 – 6.96 (m, 2H), 2.92 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 191.2, 155.8, 134.6, 131.0, 127.0, 120.6, 117.6, 45.6.

The spectra data were consistent with the literature.²



2-(dihexylamino)benzaldehyde (1c)

Yellow oil, 84%

IR (KBr): 2956, 2929, 2857, 2732, 1688, 1596, 1481, 1454, 1376, 1287, 1189, 1161, 1092, 831, 766, 731, 643, 456 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.32 (s, 1H), 7.80 (dd, *J* = 7.7, 1.5 Hz, 1H), 7.50 – 7.46 (m, 1H), 7.15 (d, *J* = 8.2 Hz, 1H), 7.06 (t, *J* = 7.4 Hz, 1H), 3.15 – 3.09 (m, 4H), 1.47 (q, *J* = 7.4, 6.9 Hz, 4H), 1.28 – 1.17 (m, 12H), 0.85 (t, *J* = 6.9 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 192.1, 155.3, 134.2, 130.2, 128.9, 121.9, 121.4, 55.2, 31.6, 27.1, 26.8, 22.6, 14.0.

HRMS (ESI): calcd. for C₁₉H₃₁NO ([M+H]⁺): 304.2635, found: 304.2633.

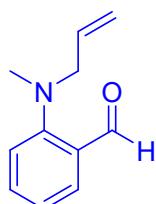


2-(diallylamino)benzaldehyde (1d)

Yellow oil, 60%

¹H NMR (600 MHz, CDCl₃) δ 10.35 (s, 1H), 7.80 (d, *J* = 7.7 Hz, 1H), 7.47 (t, *J* = 7.3 Hz, 1H), 7.12 – 7.04 (m, 2H), 5.82 (td, *J* = 10.3, 5.1 Hz, 2H), 5.23 (s, 1H), 5.21 – 5.16 (m, 3H), 3.79 (d, *J* = 5.9 Hz, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 191.7, 154.2, 134.2, 133.9, 129.5, 129.3, 122.1, 121.3, 118.1, 57.3. The spectra data were consistent with the literature.¹



2-(allyl(methyl)amino)benzaldehyde (1e)

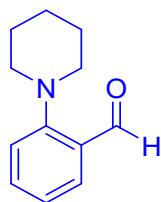
Yellow oil, 65%

IR (KBr): 3069, 2920, 2852, 1684, 1597, 1484, 1453, 1281, 1140, 924, 831, 761 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.26 (s, 1H), 7.79 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.48 (ddd, *J* = 8.3, 7.2, 1.8 Hz, 1H), 7.09 (d, *J* = 8.3 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 5.92 (ddt, *J* = 17.2, 10.2, 5.9 Hz, 1H), 5.38 – 5.16 (m, 2H), 3.74 (d, *J* = 5.9 Hz, 2H), 2.86 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 191.3, 155.5, 134.5, 134.0, 130.2, 127.7, 121.2, 118.8, 117.9, 62.0, 41.0.

HRMS (ESI): calcd. for C₁₁H₁₃NO ([M+H]⁺): 176.1070, found: 176.1070.

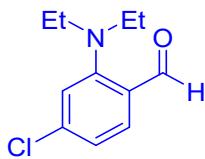


2-(piperidin-1-yl)benzaldehyde (1f)

Yellow oil, 86%

¹H NMR (600 MHz, CDCl₃) δ 10.30 (d, *J* = 0.7 Hz, 1H), 7.80 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.50 (ddd, *J* = 8.3, 7.3, 1.8 Hz, 1H), 7.15 – 7.03 (m, 2H), 3.11 – 2.99 (m, 4H), 1.77 (p, *J* = 5.9 Hz, 4H), 1.64 – 1.57 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 191.7, 157.0, 134.8, 129.2, 128.6, 122.0, 119.0, 55.6, 26.2, 24.1. The spectra data were consistent with the literature.¹



4-chloro-2-(diethylamino)benzaldehyde (1g)

Yellow oil, 86%

IR (KBr): 2974, 2934, 2844, 2736, 1690, 1587, 1468, 1411, 1381, 1242, 1189, 1173, 1086, 1011, 919, 838, 811 cm⁻¹.

¹H NMR (300 MHz, CDCl₃) δ 10.22 (s, 1H), 7.74 (d, *J* = 8.3 Hz, 1H), 7.10 (d, *J* = 1.8 Hz, 1H), 7.04 (dd, *J* = 8.3, 1.2 Hz, 1H), 3.21 (q, *J* = 7.1 Hz, 4H), 1.09 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 190.7, 155.3, 140.5, 130.5, 128.4, 122.4, 121.5, 48.7, 12.2.

HRMS (ESI): calcd. for C₁₁H₁₄ClNO ([M+H]⁺): 212.0836, found: 212.0840.



4-bromo-2-(diethylamino)benzaldehyde (1h)

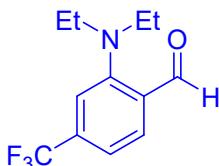
Yellow oil, 86%

IR (KBr): 2973, 2933, 2844, 1689, 1581, 1467, 1409, 1362, 1241, 1190, 1173, 1146, 1083, 1009, 912, 834, 809, 663, 592 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.23 (s, 1H), 7.66 (d, *J* = 8.3 Hz, 1H), 7.27 (d, *J* = 1.6 Hz, 1H), 7.20 (d, *J* = 8.3 Hz, 1H), 3.20 (q, *J* = 7.1 Hz, 4H), 1.09 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 190.8, 155.2, 130.5, 129.3, 128.8, 125.3, 124.5, 48.7, 12.3.

HRMS (ESI): calcd. for C₁₁H₁₄BrNO ([M+H]⁺): 256.0332, found: 256.0337.



2-(diethylamino)-4-(trifluoromethyl)benzaldehyde (1l)

Yellow oil, 86%

IR (KBr): 2977, 2936, 2875, 2850, 1694, 1613, 1576, 1494, 1427, 1382, 1318, 1241,

1170, 1130, 1074, 1015, 940, 882, 845, 830, 778, 731, 667 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.30 (s, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.35 (s, 1H), 7.29 (d, *J* = 7.9 Hz, 1H), 3.25 (q, *J* = 7.0 Hz, 4H), 1.11 (t, *J* = 7.0 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 190.9, 154.4, 135.4 (q, *J* = 32.2 Hz), 132.3, 129.9, 123.6 (d, *J* = 273.2 Hz), 118.3 (q, *J* = 3.7 Hz), 118.1 (q, *J* = 3.7 Hz), 48.7, 12.3.

¹⁹F NMR (282 MHz, CDCl₃) δ -64.2.

HRMS (ESI): calcd. for C₁₂H₁₄F₃NO ([M+H]⁺): 246.1100, found: 246.1099.



2-allyl-6-(diethylamino)benzaldehyde (1m)

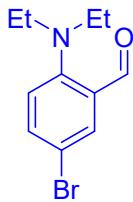
Yellow oil, 88%

IR (KBr): 3077, 2974, 2933, 2871, 2838, 1686, 1639, 1585, 1466, 1443, 1382, 1250, 1173, 1086, 1066, 1032, 996, 912, 856, 795, 739, 667 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.35 (s, 1H), 7.40 (t, *J* = 7.8 Hz, 1H), 7.07 (d, *J* = 8.1 Hz, 1H), 6.94 (d, *J* = 7.5 Hz, 1H), 6.01 (dt, *J* = 16.8, 8.4 Hz, 1H), 5.11 – 4.95 (m, 2H), 3.84 – 3.64 (m, 2H), 3.14 (q, *J* = 7.0 Hz, 4H), 1.04 (t, *J* = 7.0 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 194.4, 155.5, 142.3, 137.6, 132.8, 129.8, 124.7, 119.9, 115.4, 48.9, 37.6, 12.3.

HRMS (ESI): calcd. for C₁₄H₁₉NO ([M+H]⁺): 218.1538, found: 218.1539.



5-bromo-2-(diethylamino)benzaldehyde (1n)

Yellow oil

¹H NMR (400 MHz, CDCl₃) δ 10.3 (s, 1H), 7.9 (d, *J* = 2.5 Hz, 1H), 7.6 (dd, *J* = 8.7, 2.6 Hz, 1H), 7.0 (d, *J* = 8.7 Hz, 1H), 3.2 (q, *J* = 7.1 Hz, 4H), 1.1 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 190.5, 153.4, 136.8, 132.0, 131.6, 123.6, 115.5, 48.9, 12.3.

HRMS (ESI): calcd. for C₁₁H₁₄BrNO ([M+H]⁺): 256.0332 found: 256.0329.



(¹³C)-2-(diethylamino)benzaldehyde (1a')

Yellow oil

IR (KBr): 3730, 2973, 2931, 2850, 2322, 1652, 1594, 1483, 1452, 1378, 1274, 1242, 1187, 1144, 1093, 819, 764, 638, 419 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.36 (d, *J* = 179.2 Hz, 1H), 7.81 (dd, *J* = 8.0, 4.1 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 1H), 7.16 (d, *J* = 8.2 Hz, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 3.19 (q, *J* = 7.1 Hz, 4H), 1.07 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 192.2, 154.6 (d, *J* = 2.6 Hz), 137.2 (d, *J* = 2.9 Hz), 134.3, 130.8 (d, *J* = 54.2 Hz), 128.9 (d, *J* = 1.7 Hz), 122.0 (dd, *J* = 65.0, 3.2 Hz), 49.0, 12.4.

HRMS (ESI): calcd. for C₁₀¹³CH₁₆NO ([M+H]⁺): 179.1260, found: 179.1265.

ii. Preparation of starting materials 1k

The compound **1k** was prepared following the literature procedure.³ **1h** (25.6 mg, 1.0 mmol), Cs₂CO₃ (977 mg, 3.0 mmol) and Pd(dppf)Cl₂ (15 mg, 2 mol%) were added to a 25 mL Schlenk tube, and the mixture was dissolved in THF (2.0 mL). Triethylborane (3.0 mL, 1 M solution in THF, 3.0 mmol) was then added, and the mixture was refluxed for 6 h. The reaction mixture was cooled to 0 °C and quenched by 10% aq NaOH and 30% aq H₂O₂, and further stirring for 30 min at rt. The mixture was acidified by dilute aq HCl, and extracted with ether (3×10 mL). The combined organic extracts were dried over anhydrous Na₂SO₄, concentrated, and purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 80: 1, v/v) to afford the products **1k**.



2-(diethylamino)-4-ethylbenzaldehyde (1k)

Yellow oil, 86%

IR (KBr): 3737, 2970, 2933, 2871.8, 2843, 2310, 1681, 1601, 1567, 1546, 1511,

1491, 1455, 1423, 1378, 1278, 1247, 1202, 1100, 1013, 934, 849, 828, 751, 419 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.31 (s, 1H), 7.74 (d, *J* = 7.9 Hz, 1H), 6.96 (s, 1H), 6.93 (d, *J* = 7.9 Hz, 1H), 3.18 (q, *J* = 7.1 Hz, 4H), 2.66 (q, *J* = 7.6 Hz, 2H), 1.25 (t, *J* = 7.6 Hz, 3H), 1.06 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 191.8, 154.9, 151.5, 129.1, 128.7, 122.3, 121.0, 48.9, 29.4, 15.2, 12.4.

HRMS (ESI): calcd. for C₁₃H₁₉NO ([M+H]⁺): 206.1539, found: 206.1540.

iii. Preparation of starting materials **1i**

The compound **1i** was prepared following the literature procedure.⁴ CuI (19 mg, 5 mol %), 8-hydroxyquinoline (29 mg, 10 mol %) and K₃PO₄ (425mg, 2.0mmol) were added to a 25 mL round-bottom flask, and the flask was evacuated and backfilled with argon (3 cycles). **1h** (256 mg, 1.0 mmol) and ethanol (1 mL) were added by syringe at room temperature. The oil bath temperature was slowly increased to 110 °C and stirred for 24 h. The reaction was monitored by TLC. After the reaction was completed, the organic layer was extracted with ethyl acetate (3 × 20 mL) and dried over anhydrous Na₂SO₄. The combined organic layers were concentrated under vacuo and purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 80: 1) to afford the product **1i**.



2-(diethylamino)-4-ethoxybenzaldehyde (1i**)**

Yellow oil, 60%

IR (KBr): 2976, 2933, 2872, 2838, 1677, 1595, 1491, 1476, 1383, 1301, 1287, 1254, 1212, 1100, 1043, 851, 805 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.18 (s, 1H), 7.79 (d, *J* = 8.4 Hz, 1H), 6.60 (d, *J* = 8.5 Hz, 2H), 4.09 (d, *J* = 7.0 Hz, 2H), 3.18 (q, *J* = 7.1 Hz, 4H), 1.44 (t, *J* = 7.0 Hz, 3H), 1.07 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 190.6, 164.2, 156.7, 131.4, 124.3, 108.3, 107.4, 63.7, 48.7, 14.7. 12.3.

HRMS (ESI): calcd. for C₁₃H₁₉NO₂ ([M+H]⁺): 222.1489, found: 222.1489.

iv. Preparation of starting materials **1j**

The compound **1j** was prepared following the literature procedure.⁵ Pd₂(dba)₃ (9.2 mg, 0.01 mmol), DPPF (11.1 mg, 0.02 mmol) and toluene (1.0 mL) were added to a 25 mL round-bottom flask. **1h** (256 mg, 1.0 mmol), *i*-Pr₂NEt (0.19 mL, 1.1 mmol) and ethanol (0.5 mL) were added at room temperature. The bath temperature was slowly increased to 110 °C and stirred for 24 h. The reaction was monitored by TLC. After the reaction was completed, the mixture was extracted with ethyl acetate (3 × 20 mL) and dried over anhydrous Na₂SO₄. The combined organic layers were concentrated under vacuo and purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 80: 1, v/v) to afford the product **1j**.



2-(diethylamino)-4-(ethylthio)benzaldehyde (1j**)**

Yellow oil, 70%

IR (KBr): 3730, 2972, 2930, 2871, 2841, 1672, 1582, 1551, 1473, 1453, 1405, 1379, 1364, 1291, 1263, 1239, 1150, 1076, 840, 811, 419 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.23 (s, 1H), 7.72 (d, *J* = 8.2 Hz, 1H), 6.99 – 6.92 (m, 2H), 3.19 (q, *J* = 7.1 Hz, 4H), 3.01 (q, *J* = 7.4 Hz, 2H), 1.37 (t, *J* = 7.4 Hz, 3H), 1.07 (t, *J* = 7.1 Hz, 6H).

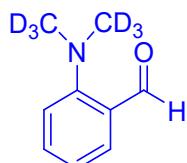
¹³C NMR (101 MHz, CDCl₃) δ 191.0, 154.6, 146.0, 129.5, 127.5, 120.0, 119.1, 48.7, 26.0, 14.0, 12.3.

HRMS (ESI): calcd. for C₁₃H₁₉NOS ([M+H]⁺): 238.1260, found: 238.1260.

v. Preparation of starting materials **1b'**

To a 25 mL round-bottom flask, NaH (ca. 60% dispersion in mineral oil, 3.0 mmol) was added and the flask was evacuated and backfilled with argon. 2-aminobenzaldehyde (121 mg, 1 mmol) was added to the flask, and the mixture was cooled to 0 °C by ice/water bath. Iodomethane-D3 (187 μL, 3.0 mmol) was added slowly to the flask. The ice/water bath was removed, and the mixture was stirred overnight at room temperature. The reaction was monitored by TLC. After the reaction was completed, the organic layer was extracted with ethyl acetate (3 × 20 mL) and dried over anhydrous Na₂SO₄. The combined organic layers were concentrated

under vacuo and purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 80: 1, v/v) to afford the products **1b'**.



2-(bis(methyl-d3)amino)benzaldehyde (1b')

Yellow oil, 70%

IR (KBr): 2847, 2740, 2241, 2193, 2055, 1684, 1597, 1482, 1453, 1388, 1321, 1292, 1275, 1192, 1177, 1117, 1100, 843, 820, 760, 639 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.22 (s, 1H), 7.77 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.47 (ddd, *J* = 8.3, 7.2, 1.8 Hz, 1H), 7.07 – 6.97 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 191.1, 155.7, 134.5, 131.0, 126.9, 120.5, 117.5, 44.5 (hept, *J* = 20.1).

HRMS (ESI): calcd. for C₉H₅D₆NO ([M+H]⁺): 156.1290, found: 156.1290.

vi. Preparation of starting materials **1a''**

LiAlD₄ (205 mg, 5.0 mmol) and THF (10 mL) were added to a 25 mL round-bottom flask and the mixture was cooled to 0 °C by ice/water bath. **1a** (5.0 mmol, 885.3 mg) in THF (2 mL) was slowly added to the flask. The mixture was further stirred for 2 h at room temperature. The reaction was monitored by TLC. After the reaction was completed, the reaction mixture was slowly quenched with H₂O and the extracted with ethyl acetate (3×10 mL) and dried over anhydrous Na₂SO₄. The combined organic layers were concentrated under vacuo. The residue product was further dissolved in 40 mL CH₂Cl₂ and MnO₂ (20 mmol, 1.74 g) was added. The mixture was further stirred for 48 h at room temperature. After the reaction was completed, the reaction mixture was filtered and the filtrate was concentrated under vacuo. The crude product was further purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 80: 1, v/v) to afford the products **1a''**.



(D)-2-(diethylamino)benzaldehyde (1a'')

IR (KBr): 2974, 2927, 2851, 1667, 1595, 1482, 1451, 1381, 1355, 1291, 1274, 1243, 1209, 1175, 1147, 1105, 1023, 942, 797, 764, 413 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 7.82 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.50 (ddd, *J* = 8.2, 7.1, 1.8 Hz, 1H), 7.18 – 7.09 (m, 1H), 7.08 (d, *J* = 7.7 Hz, 1H), 3.19 (q, *J* = 7.1 Hz, 4H), 1.07 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 192.0 (t, *J* = 27.4 Hz), 154.6, 134.3, 130.5 (t, *J* = 3.1), 128.9, 122.3, 121.6, 48.9, 12.3.

HRMS (ESI): calcd. for C₁₁H₁₄DNO ([M+H]⁺): 179.1289, found: 179.1288.

4. General procedures for decarbonylative cross-coupling

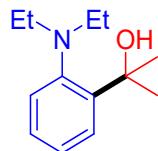
i. Decarbonylative cross-coupling of aldehyde with acetone

The starting materials **1** (0.1 mmol), CsF (0.05 mmol) and acetone (1 mL) were added to a 20 mL tube with a magnetic stir-bar. Then the tube was evacuated by three freeze-pump-thaw cycles and back-filled with ultra-purified argon. The reaction mixture was stirred at room temperature under 405nm LED for 12 h and monitored by TLC. After the reaction was completed, the mixture was filtered through a pad of diatomite. The organic layers were then concentrated under vacuo and purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1, v/v) to afford the products **3**.

ii. Decarbonylative cross-coupling of aldehyde with other ketones

The starting materials **1** (0.1 mmol), CsF (0.05 mmol), ketone (0.8 mmol) and toluene (1.0 mL) were added to a 20 mL tube with a magnetic stir-bar. Then the tube was evacuated by three freeze-pump-thaw cycles and back-filled with ultra-purified argon. The reaction was stirred at room temperature under 405nm LED for 12 h. After the reaction was completed, the mixture was filtered through a pad of diatomite. The organic layers were then concentrated under vacuo and purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1, v/v) to afford the products **3**.

Characterization data of products



2-(2-(diethylamino)phenyl)propan-2-ol (3aa)

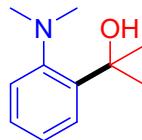
Colourless oil. 17 mg, 82%

IR (KBr): 3062, 2975, 2931, 2837, 1483, 1442, 1379, 1356, 1278, 1217, 1167, 1120, 1083, 1047, 964, 758, 679, 558 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.33 (br, 1H), 7.29 (d, *J* = 8.2 Hz, 1H), 7.27 – 7.22 (m, 2H), 7.21 – 7.17 (m, 1H), 3.05 (dq, *J* = 14.2, 7.2 Hz, 2H), 2.94 (dq, *J* = 14.1, 7.2 Hz, 2H), 1.58 (s, 6H), 1.09 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.2, 145.4, 127.4, 127.3, 126.1, 124.1, 74.7, 50.5, 33.9, 12.9.

HRMS (ESI): calcd. for C₁₃H₂₁NO ([M+H]⁺): 208.1696, found: 208.1700.



2-(2-(dimethylamino)phenyl)propan-2-ol (3ba)

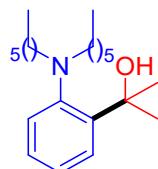
Colourless oil. 14 mg, 78%

IR (KBr): 3101, 3065, 2977, 2945, 2928, 2866, 2833, 2790, 1731, 1682, 1598, 1485, 1457, 1404, 1377, 1357, 1283, 1181, 1118, 1096, 1037, 934, 758, 679, 552 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 9.52 (br, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.29 – 7.24 (m, 2H), 7.17 (t, *J* = 7.5 Hz, 1H), 2.71 (s, 6H), 1.58 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 151.1, 144.1, 127.7, 126.7, 126.3, 123.5, 74.0, 46.6, 32.7.

HRMS (ESI): calcd. for C₁₁H₁₇NO ([M+H]⁺): 180.1383, found: 180.1387.



2-(2-(dihexylamino)phenyl)propan-2-ol (3ca)

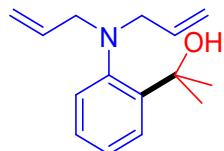
Colourless oil. 20 mg, 63%

IR (KBr): 3062, 2957, 2930, 2857, 1466, 1441, 1377, 1356, 1279, 1213, 1175, 1118, 1094, 1084, 1048, 964, 757, 725, 684, 559 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.29 (br, 1H), 7.28 – 7.24 (m, 2H), 7.22 (td, *J* = 7.4, 1.9 Hz, 1H), 7.16 (td, *J* = 7.3, 1.7 Hz, 1H), 2.91 (td, *J* = 12.2, 10.7, 5.2 Hz, 2H), 2.82 (td, *J* = 12.2, 10.7, 5.2 Hz, 2H), 1.65 – 1.57 (m, 2H), 1.55 (s, 6H), 1.41 – 1.31 (m, 2H), 1.30 – 1.18 (m, 12H), 0.88 – 0.81 (m, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 148.3, 144.8, 127.4, 127.3, 125.9, 124.1, 74.6, 57.3, 33.9, 31.6, 27.6, 27.2, 22.2, 14.0.

HRMS (ESI): calcd. for C₂₁H₃₇NO ([M+H]⁺): 320.2948, found: 320.2950.



2-(2-(diallylamino)phenyl)propan-2-ol (3da)

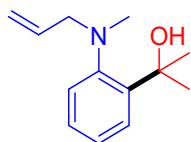
Colourless oil. 11 mg, 48%

IR (KBr): 3078, 2977, 2927, 2848, 1735, 1687, 1644, 1597, 1575, 1483, 1442, 1421, 1377, 1357, 1278, 1174, 1084, 991, 962, 924, 757, 694, 661, 597, 550 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 9.56 (br, 1H), 7.29 – 7.26 (m, 1H), 7.26 – 7.14 (m, 3H), 5.90 (ddt, *J* = 17.0, 10.2, 6.7 Hz, 2H), 5.18 (q, *J* = 1.5 Hz, 1H), 5.16 – 5.10 (m, 3H), 3.57 (dd, *J* = 12.1, 6.8 Hz, 4H), 1.57 (s, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.7, 144.8, 133.9, 127.3, 127.2, 126.3, 125.1, 119.0, 74.5, 59.0, 33.4.

HRMS (ESI): calcd. for C₁₅H₂₁NO ([M+H]⁺): 232.1696, found: 232.1700.



2-(2-(allyl(methyl)amino)phenyl)propan-2-ol (3ea)

Colourless oil. 10 mg, 49%

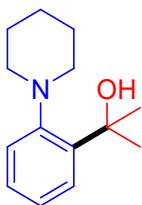
IR (KBr): 3074, 2977, 2926, 2799, 1737, 1643, 1597, 1578, 1484, 1453, 1423, 1377, 1357, 1276, 1222, 1176, 1119, 996, 960, 910, 758, 697, 552 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 9.43 (br, 1H), 7.30 (t, *J* = 8.4 Hz, 2H), 7.27 – 7.23 (m,

1H), 7.18 (t, J = 7.6 Hz, 1H), 5.97 – 5.87 (m, 1H), 5.23 (dd, J = 34.4, 13.7 Hz, 2H), 3.59 (d, J = 11.7 Hz, 1H), 3.42 (t, J = 10.7 Hz, 1H), 2.67 (s, 3H), 1.58 (s, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 150.4, 144.3, 134.1, 127.5, 126.8, 126.3, 124.2, 118.8, 74.1, 61.7, 43.5, 32.9, 32.7.

HRMS (ESI): calcd. for $\text{C}_{13}\text{H}_{19}\text{NO}$ ($[\text{M}+\text{H}]^+$): 206.1539, found: 206.1542.



2-(2-(piperidin-1-yl)phenyl)propan-2-ol (3fa)

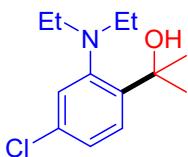
Yellow oil, 18 mg, 82%

IR (KBr): 2934, 2853, 2825, 1576, 1484, 1442, 1377, 1355, 1311, 1275, 1212, 1119, 1049, 1033, 959, 913, 863, 757, 695, 539 cm^{-1} .

^1H NMR (600 MHz, CDCl_3) δ 9.62 (br, 1H), 7.32 (d, J = 8.0 Hz, 1H), 7.28 (d, J = 7.9 Hz, 1H), 7.24 (t, J = 7.6 Hz, 1H), 7.17 (d, J = 7.5 Hz, 1H), 3.05 (d, J = 11.6 Hz, 2H), 2.78 (td, J = 11.6, 3.2 Hz, 2H), 1.85 (d, J = 13.7 Hz, 1H), 1.79 – 1.67 (m, 4H), 1.58 (s, 6H), 1.39 – 1.32 (m, 1H).

^{13}C NMR (101 MHz, CDCl_3) δ 150.8, 144.2, 127.5, 126.7, 126.3, 124.1, 74.0, 55.4, 32.7, 26.4, 23.8.

HRMS (ESI): calcd. for $\text{C}_{14}\text{H}_{21}\text{NO}$ ($[\text{M}+\text{H}]^+$): 220.1696, found: 220.1700.



2-(4-chloro-2-(diethylamino)phenyl)propan-2-ol (3ga)

Yellow oil, 22 mg, 91%

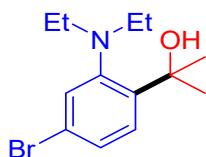
IR (KBr): 3064, 2976, 2929, 2838, 1589, 1564, 1473, 1450, 1383, 1358, 1297, 1277, 1214, 1175, 1120, 1028, 965, 918, 867, 820, 804, 787, 637, 549 cm^{-1} .

^1H NMR (600 MHz, CDCl_3) δ 9.89 (br, 1H), 7.23 – 7.19 (m, 2H), 7.16 (d, J = 8.5 Hz, 1H), 3.02 (dq, J = 14.3, 7.3 Hz, 2H), 2.88 (dq, J = 14.0, 7.2 Hz, 2H), 1.53 (s, 6H), 1.08 (t, J = 7.2 Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 148.7, 144.1, 132.5, 128.6, 126.5, 124.5, 74.4, 50.7,

33.7, 12.9.

HRMS (ESI): calcd. for C₁₃H₂₀ClNO ([M+H]⁺): 242.1306, found: 242.1311.



2-(4-bromo-2-(diethylamino)phenyl)propan-2-ol (3ha)

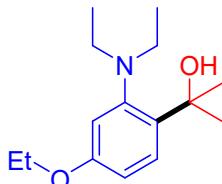
Yellow oil. 25 mg, 88%

IR (KBr): 3062, 2975, 2928, 2839, 1582, 1561, 1478, 1382, 1297, 1275, 1212, 1175, 1121, 1100, 1068, 1028, 965, 908, 866 819 780, 626 549 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 9.85 (br, 1H), 7.35 (s, 1H), 7.30 (d, *J* = 8.7 Hz, 1H), 7.15 (d, *J* = 8.5 Hz, 1H), 3.02 (dq, *J* = 14.2, 7.3 Hz, 2H), 2.88 (dq, *J* = 14.0, 7.4 Hz, 2H), 1.53 (s, 6H), 1.08 (t, *J* = 7.3 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 148.9, 144.6, 129.4, 129.0, 127.5, 120.3, 74.4, 50.7, 33.7, 12.9.

HRMS (ESI): calcd. for C₁₃H₂₀BrNO ([M+H]⁺): 286.0801, found: 286.0806.



2-(2-(diethylamino)-4-ethoxyphenyl)propan-2-ol (3ia)

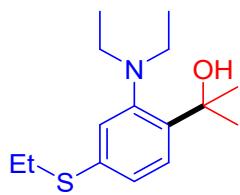
Colourless oil. 17 mg, 68%

IR (KBr): 3066, 2975, 2927, 2874, 2850, 1608, 1573, 1478, 1448, 1381, 1312, 1286, 1243, 1197, 1167, 1116, 1047, 963, 866, 851, 820, 672, 549 cm⁻¹.

¹H NMR (300 MHz, CDCl₃) δ 10.05 (br, 1H), 7.17 (d, *J* = 8.3 Hz, 1H), 6.77 – 6.70 (m, 2H), 4.08 – 3.93 (m, 2H), 2.95 (dt, *J* = 27.5, 13.3, 12.5, 7.2 Hz, 4H), 1.53 (s, 6H), 1.41 (td, *J* = 7.0, 1.1 Hz, 3H), 1.07 (td, *J* = 7.2, 1.1 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 157.9, 148.4, 137.7, 128.0, 112.0, 110.0, 74.2, 63.5, 50.5, 34.0, 14.8, 12.9.

HRMS (ESI): calcd. for C₁₅H₂₅NO₂ ([M+H]⁺): 252.1958, found: 252.1963.



2-(2-(diethylamino)-4-(ethylthio)phenyl)propan-2-ol (3ja)

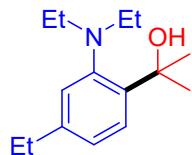
Colourless oil. 22 mg, 82%

IR (KBr): 3063, 2974, 2928, 2872, 2836, 1590, 1551, 1477, 1449, 1381, 1357, 1280, 1262, 1213, 1167, 1143, 1120, 1086, 1065, 1029, 964, 921 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.07 (br, 1H), 7.22 – 7.17 (m, 2H), 7.14 (dd, *J* = 8.4, 1.8 Hz, 1H), 3.08 – 2.97 (m, 2H), 2.97 – 2.86 (m, 4H), 1.54 (s, 6H), 1.31 (t, *J* = 7.4 Hz, 3H), 1.07 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.7, 143.1, 135.2, 127.9, 126.9, 125.1, 74.5, 50.5, 33.8, 27.8, 14.4, 12.9.

HRMS (ESI): calcd. for C₁₅H₂₅NOS ([M+H]⁺): 268.1730, found: 268.1734.



2-(2-(diethylamino)-4-ethylphenyl)propan-2-ol (3ka)

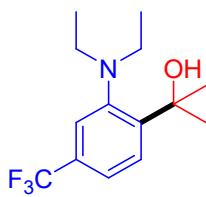
Colourless oil. 14 mg, 60%

IR (KBr): 2972, 2927, 2851, 1611, 1585 1492 1451, 1407, 1378, 1356 1282, 1190, 1167, 1125, 1073, 1030, 964, 827, 792, 747, 703, 668, 554 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.33 (br, 1H), 7.17 (d, *J* = 8.1 Hz, 1H), 7.04 (d, *J* = 1.8 Hz, 1H), 7.01 (dd, *J* = 8.1, 1.9 Hz, 1H), 3.02 (dq, *J* = 12.1, 7.2 Hz, 2H), 2.92 (dq, *J* = 12.2, 7.2 Hz, 2H), 2.61 (q, *J* = 7.6 Hz, 2H), 1.54 (s, 6H), 1.22 (t, *J* = 7.6 Hz, 3H), 1.07 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.1, 143.3, 142.4, 127.2, 125.7, 123.1, 74.5, 50.4, 33.9, 28.2, 15.4, 12.9.

HRMS (ESI): calcd. for C₁₅H₂₅NO ([M+H]⁺): 236.2009, found: 236.2013.



2-(2-(diethylamino)-4-(trifluoromethyl)phenyl)propan-2-ol (3la)

Colourless oil. 17 mg, 62%

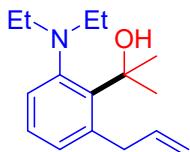
IR (KBr): 2978, 2932, 2842, 1498, 1452, 1406, 1359, 1330, 1292, 1170, 1127, 1096, 1065, 1030, 966, 926, 893, 833, 729, 631 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 9.79 (br, 1H), 7.32 (s, 1H), 7.28 – 7.21 (m, 2H), 2.97 – 2.84 (m, 2H), 2.81 – 2.70 (m, 2H), 1.40 (s, 6H), 0.91 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 149.4, 148.0, 129.8 (q, *J* = 32.7 Hz), 123.8 (q, *J* = 272.7 Hz), 122.9 (q, *J* = 3.6 Hz), 121.5 (q, *J* = 3.8 Hz), 74.7, 50.6, 33.6, 12.8.

¹⁹F NMR (282 MHz, CDCl₃) δ -62.9.

HRMS (ESI): calcd. for C₁₄H₂₀F₃NO ([M+H]⁺): 276.1570, found: 276.1573.



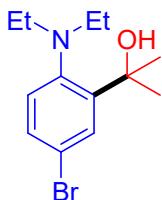
2-(2-allyl-6-(diethylamino)phenyl)propan-2-ol (3ma)

Colorless oil. 19 mg, 78%

¹H NMR (600 MHz, CDCl₃) δ 12.12 (s, 1H), 7.18 (t, *J* = 7.9 Hz, 1H), 7.11 (d, *J* = 8.7 Hz, 2H), 6.01 – 5.91 (m, 1H), 5.09 (d, *J* = 9.8 Hz, 1H), 5.01 (d, *J* = 17.0 Hz, 1H), 3.56 (d, *J* = 5.4 Hz, 2H), 3.07 – 2.98 (m, 2H), 2.93 – 2.82 (m, 2H), 1.66 (s, 6H), 1.07 (t, *J* = 7.1 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 147.8, 144.1, 136.8, 133.7, 130.2, 126.4, 125.4, 123.0, 75.4, 50.6, 32.1, 14.3, 12.7.

HRMS (ESI): calcd. for C₁₆H₂₅NO ([M+H]⁺): 248.2009, found: 248.2006.



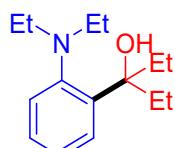
2-(5-bromo-2-(diethylamino)phenyl)propan-2-ol (3na)

Colourless oil. 20mg, 70 %

¹H NMR (400 MHz, CDCl₃) δ 10.0 (s, 1H), 7.4 (d, *J* = 2.4 Hz, 1H), 7.3 (dd, *J* = 8.6, 2.4 Hz, 1H), 7.1 (d, *J* = 8.6 Hz, 1H), 3.0 (dq, *J* = 12.3, 7.2 Hz, 2H), 2.9 (dq, *J* = 12.3, 7.2 Hz, 2H), 1.5 (s, 6H), 1.1 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.8, 146.5, 130.5, 130.4, 126.1, 119.7, 74.5, 50.5, 33.7, 12.8.

HRMS (ESI): calcd. for C₁₃H₂₀BrNO ([M+H]⁺): 286.0801, found: 286.0805.



3-(2-(diethylamino)phenyl)pentan-3-ol (3ab)

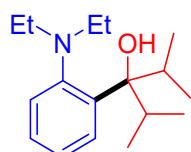
Colourless oil. 18 mg, 77%

IR (KBr): 3026, 2973, 2935, 2875, 2843, 1482, 1441, 1384, 1297, 1271, 1218, 1170, 1121, 1089, 1061, 1037, 975, 754, 674, 594, 559 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.26 (br, 1H), 7.25 – 7.09 (m, 4H), 3.01 (dq, *J* = 12.4, 7.2 Hz, 2H), 2.88 (dq, *J* = 12.4, 7.3 Hz, 2H), 1.86 – 1.68 (m, 4H), 1.10 (t, *J* = 7.2 Hz, 6H), 0.80 (t, *J* = 7.3 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 149.8, 141.7, 128.0, 127.0, 125.9, 124.1, 80.3, 50.4, 37.1, 13.1, 8.5.

HRMS (ESI): calcd. for C₁₅H₂₅NO ([M+H]⁺): 236.2009, found: 236.2012.



3-(2-(diethylamino)phenyl)-2,4-dimethylpentan-3-ol (3ac)

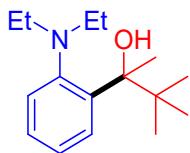
Colourless oil. 15 mg, 57 %

IR (KBr): 2972, 2934, 2875, 2832, 1475, 1441, 1381, 1195, 1179, 1165, 1119, 1101, 1061, 1032, 1012, 998, 865, 771, 753, 673, 629 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.58 (br, 1H), 7.24 – 7.18 (m, 2H), 7.19 – 7.10 (m, 2H), 3.07 – 2.98 (m, 2H), 2.92 – 2.83 (m, 2H), 2.26 (hept, *J* = 6.8 Hz, 2H), 1.12 (t, *J* = 7.2 Hz, 6H), 0.86 (dd, *J* = 22.0, 6.8 Hz, 12H).

¹³C NMR (101 MHz, CDCl₃) δ 151.4, 139.1, 128.1, 126.9, 125.1, 124.3, 84.2, 50.5, 36.5, 18.6, 17.1, 13.2.

HRMS (ESI): calcd. for C₁₇H₂₉NO ([M+H]⁺): 264.2322, found: 264.2325.



2-(2-(diethylamino)phenyl)-3,3-dimethylbutan-2-ol (3ad)

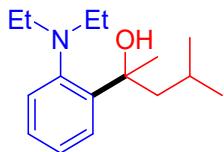
Colourless oil. 14 mg, 56 %

IR (KBr): 3064, 2978, 2873, 2829, 1787, 1483, 1443, 1388, 1368, 1294, 1276, 1215, 1188, 1167, 1061, 1029, 1004, 935, 759, 729 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.44 (br, 1H), 7.25 – 7.18 (m, 3H), 7.13 (ddd, *J* = 8.0, 6.1, 2.5 Hz, 1H), 3.15 (dq, *J* = 12.0, 7.3 Hz, 1H), 2.98 (q, *J* = 7.1 Hz, 2H), 2.69 (dq, *J* = 12.0, 7.2 Hz, 1H), 1.53 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H), 0.95 (m, 12H).

¹³C NMR (101 MHz, CDCl₃) δ 149.9, 141.7, 129.7, 127.3, 124.7, 124.1, 81.7, 51.2, 49.8, 40.3, 26.5, 26.3, 13.7, 12.1.

HRMS (ESI): calcd. for C₁₆H₂₇NO ([M+H]⁺): 250.2165, found: 250.2170.



2-(2-(diethylamino)phenyl)-4-methylpentan-2-ol (3ae)

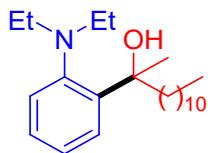
Colourless oil. 17 mg, 68 %

IR (KBr): 3062, 3027, 2973, 2926, 2867, 2837, 1482, 1443, 1383, 1367, 1282, 1218, 1167, 1123, 1098, 1048, 1029, 867, 757, 702, 683, 596 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.26 (br, 1H), 7.25 – 7.19 (m, 3H), 7.15 (t, *J* = 7.1 Hz, 1H), 3.07 – 2.98 (m, 2H), 2.96 – 2.83 (m, 2H), 1.82 – 1.73 (m, 2H), 1.64 (dd, *J* = 13.6, 5.1 Hz, 1H), 1.50 (s, 3H), 1.11 (d, *J* = 7.2 Hz, 3H), 1.06 (t, *J* = 7.2 Hz, 3H), 0.94 (d, *J* = 6.3 Hz, 3H), 0.76 (d, *J* = 6.3 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 148.0, 144.4, 128.0, 127.2, 125.8, 124.1, 77.7, 54.0, 50.4, 50.4, 33.6, 24.9, 24.7, 24.6, 13.0, 12.8.

HRMS (ESI): calcd. for C₁₆H₂₇NO ([M+H]⁺): 250.2165, found: 250.2170.



2-(2-(diethylamino)phenyl)tridecan-2-ol (3af)

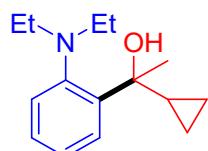
Colourless oil. 24 mg, 69 %

IR (KBr): 3061, 3026, 2971, 2925, 2853, 1482, 1465, 1382, 1294, 1278, 1217, 1168, 1123, 1087, 1064, 1045, 757, 723, 682, 592, 558 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.26 (br, 1H), 7.20 (dq, *J* = 21.3, 7.1, 6.0 Hz, 4H), 3.06 – 2.97 (m, 2H), 2.94 – 2.84 (m, 2H), 1.83 (td, *J* = 12.9, 4.3 Hz, 1H), 1.69 (td, *J* = 12.9, 4.4 Hz, 1H), 1.50 (s, 3H), 1.46 – 1.39 (m, 1H), 1.31 – 1.19 (m, 17H), 1.07 (dt, *J* = 15.2, 7.2 Hz, 6H), 0.87 (t, *J* = 7.0 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 148.1, 144.1, 127.8, 127.1, 126.0, 124.1, 50.5, 50.4, 45.7, 33.0, 31.9, 30.1, 29.7, 29.7, 29.6, 29.4, 24.3, 22.7, 14.1, 13.1, 12.9.

HRMS (ESI): calcd. for C₂₃H₄₁NO ([M+H]⁺): 348.2361, found: 348.2362.



1-cyclopropyl-1-(2-(diethylamino)phenyl)ethan-1-ol (3ag)

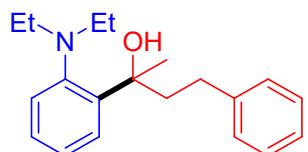
Colourless oil. 15 mg, 64%

IR (KBr): 3063, 2974, 2926, 2834, 1598, 1576, 1483, 1447, 1383, 1278, 1218, 1169, 1122, 1102, 1064, 1044, 1018, 823, 756, 736, 591, 558, 528 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.26 (br, 1H), 7.42 (dt, *J* = 6.6, 1.5 Hz, 1H), 7.25 – 7.17 (m, 3H), 3.09 – 2.97 (m, 2H), 2.97 – 2.81 (m, 2H), 1.48 (s, 3H), 1.25 (ddd, *J* = 8.5, 5.6, 2.9 Hz, 1H), 1.07 (dt, *J* = 9.8, 7.2 Hz, 6H), 0.59 (ddd, *J* = 9.2, 5.8, 3.5 Hz, 1H), 0.53 – 0.39 (m, 2H), 0.28 (tdd, *J* = 8.6, 5.8, 3.4 Hz, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 147.3, 145.4, 127.9, 127.1, 126.0, 124.0, 74.7, 50.5, 50.2, 31.5, 24.2, 12.9, 12.7, 1.6, 0.9.

HRMS (ESI): calcd. for C₁₅H₂₃NO ([M+H]⁺): 234.1852, found: 234.1856.



2-(2-(diethylamino)phenyl)-4-phenylbutan-2-ol (3ah)

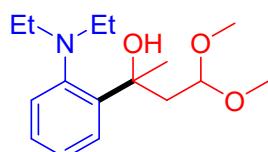
Colourless oil. 19 mg, 64 %

IR (KBr): 3061, 3026, 2973, 2928, 2837, 1602, 1575, 1483, 1451, 1383, 1294, 1277, 1216, 1166, 1121, 1100, 1064, 1028, 759, 699, 622, 604 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.58 (br, 1H), 7.23 (dq, *J* = 20.5, 7.6 Hz, 6H), 7.15 (dd, *J* = 14.8, 7.5 Hz, 3H), 3.05 (dq, *J* = 14.3, 7.2 Hz, 2H), 2.92 (ddq, *J* = 26.6, 13.9, 7.2 Hz, 2H), 2.82 (td, *J* = 13.1, 4.8 Hz, 1H), 2.38 (td, *J* = 13.1, 4.2 Hz, 1H), 2.15 (td, *J* = 13.2, 4.2 Hz, 1H), 2.04 (td, *J* = 13.3, 4.8 Hz, 1H), 1.56 (s, 3H), 1.10 (q, *J* = 7.7 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 148.1, 143.5, 143.3, 128.4, 128.3, 127.6, 127.4, 126.2, 125.4, 124.2, 50.8, 50.3, 47.9, 33.3, 30.9, 13.2, 12.9.

HRMS (ESI): calcd. for C₂₀H₂₇NO ([M+H]⁺): 298.2165, found: 298.2167.



2-(2-(diethylamino)phenyl)-4,4-dimethoxybutan-2-ol (3ai)

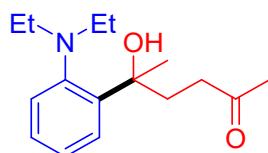
Colourless oil. 17 mg, 60 %

IR (KBr): 3060, 2975, 2933, 2830, 1483, 1444, 1382, 1296, 1279, 1211, 1192, 1165, 1122, 1074, 1050, 972, 941, 911, 898, 820, 758, 705, 620 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.57 (br, 1H), 7.23 (d, *J* = 3.8 Hz, 3H), 7.20 – 7.15 (m, 1H), 4.62 (t, *J* = 4.9 Hz, 1H), 3.34 (s, 3H), 3.17 (s, 3H), 3.02 (ddt, *J* = 20.8, 14.1, 7.0 Hz, 2H), 2.91 (ddq, *J* = 26.8, 13.8, 7.2 Hz, 2H), 2.16 – 2.06 (m, 2H), 1.55 (s, 3H), 1.11 (t, *J* = 7.2 Hz, 3H), 1.05 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (151 MHz, CDCl₃) δ 147.5, 143.9, 127.9, 127.4, 125.9, 124.1, 102.7, 96.7, 75.5, 53.1, 52.1, 50.6, 50.2, 48.4, 32.1, 13.0, 12.7.

HRMS (ESI): calcd. for C₁₆H₂₇NO₃ ([M+H]⁺): 282.2064, found: 282.2068.



5-(2-(diethylamino)phenyl)-5-hydroxyhexan-2-one (3aj)

Yellow oil. 15 mg, 57 %

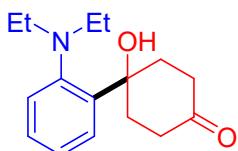
IR (KBr): 3060, 3025, 2974, 2930, 2875, 2836, 1483, 1443, 1367, 1280, 1217, 1164, 1121, 1085, 1043, 1029, 936, 894, 760, 683, 561, 548 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.53 (br, 1H), 7.28 – 7.20 (m, 2H), 7.21 – 7.14 (m, 2H), 3.09 – 2.97 (m, 2H), 2.90 (ddq, *J* = 30.1, 12.3, 7.2 Hz, 2H), 2.66 (ddd, *J* = 16.6,

10.9, 5.0 Hz, 1H), 2.28 (ddd, $J = 16.7$, 10.9, 4.7 Hz, 1H), 2.21 – 2.13 (m, 1H), 2.10 (s, 3H), 2.00 (ddd, $J = 14.0$, 10.9, 5.0 Hz, 1H), 1.51 (s, 3H), 1.08 (dt, $J = 11.3$, 7.2 Hz, 6H)

^{13}C NMR (101 MHz, CDCl_3) δ 209.7, 147.9, 143.0, 127.6, 127.5, 126.3, 124.3, 76.4, 50.9, 50.1, 39.2, 38.8, 33.2, 29.9, 13.1, 12.8.

HRMS (ESI): calcd. for $\text{C}_{16}\text{H}_{25}\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 264.1958, found: 264.1960.



4-(2-(diethylamino)phenyl)-4-hydroxycyclohexan-1-one (3ak)

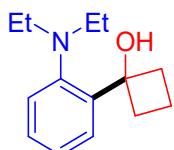
White solid. 11 mg, 42%, m.p. 71.4 - 73.2 °C

IR (KBr): 3026, 2973, 2931, 2873, 2835, 1711, 1598, 1483, 1436, 1382, 1338, 1308, 1279, 1247, 1124, 1087, 1053, 1026, 866, 770, 755, 716, 671 cm^{-1} .

^1H NMR (600 MHz, CDCl_3) δ 11.12 (br, 1H), 7.29 (s, 2H), 7.26 – 7.19 (m, 2H), 3.12 – 3.00 (m, 4H), 2.99 – 2.90 (m, 2H), 2.33 – 2.24 (m, 4H), 2.13 (d, $J = 12.3$ Hz, 2H), 1.08 (t, $J = 7.2$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 213.1, 147.4, 142.8, 128.0, 126.7, 126.4, 124.3, 74.4, 50.8, 41.3, 37.6, 12.8.

HRMS (ESI): calcd. for $\text{C}_{16}\text{H}_{23}\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 262.1802, found: 262.1804.



1-(2-(diethylamino)phenyl)cyclobutan-1-ol (3al)

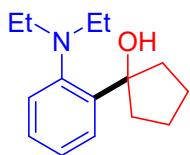
Colourless oil. 15 mg, 68 %

IR (KBr): 3064, 3028, 2976, 2935, 2873, 2837, 1598, 1575.0, 1484, 1446, 1384, 1291, 1223, 1168, 1146, 1129, 1086, 1059, 1028, 897, 752, 683 cm^{-1} .

^1H NMR (600 MHz, CDCl_3) δ 9.64 (br, 1H), 7.51 (d, $J = 7.5$ Hz, 1H), 7.27 – 7.19 (m, 3H), 2.97 (q, $J = 7.2$ Hz, 4H), 2.56 – 2.50 (m, 2H), 2.49 – 2.42 (m, 2H), 2.04 (ddq, $J = 14.8$, 9.9, 4.8 Hz, 1H), 1.85 – 1.75 (m, 1H), 1.04 (t, $J = 7.2$ Hz, 6H).

^{13}C NMR (101 MHz, CDCl_3) δ 147.4, 142.8, 127.3, 125.9, 125.7, 123.8, 78.4, 49.10, 38.9, 13.3, 12.5.

HRMS (ESI): calcd. for C₁₄H₂₁NO ([M+H]⁺): 220.1696, found: 220.1698.



1-(2-(diethylamino)phenyl)cyclopentan-1-ol (3am)

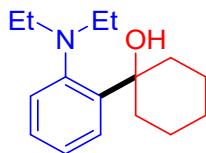
Colourless oil. 16 mg, 69%

IR (KBr): 3061, 3025, 2971, 2870, 2845, 1722, 1481, 1383, 1340, 1290, 1168, 1121, 1013, 946, 911, 891, 753, 602, 571, 540, 472, 458 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.08 (br, 1H), 7.27 – 7.20 (m, 3H), 7.16 (ddd, *J* = 8.4, 6.8, 1.9 Hz, 1H), 3.06 – 2.98 (m, 2H), 2.97 – 2.89 (m, 2H), 2.07 – 1.92 (m, 6H), 1.77 (s, 2H), 1.07 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 148.1, 143.9, 127.2, 126.9, 125.9, 124.0, 84.9, 50.3, 44.9, 24.7, 12.8.

HRMS (ESI): calcd. for C₁₅H₂₃NO ([M+H]⁺): 234.1852, found: 234.1854.



1-(2-(diethylamino)phenyl)cyclohexan-1-ol (3an)

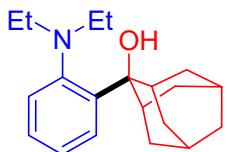
Colourless oil. 18 mg, 73%

IR (KBr): 3060, 2974, 2930, 2845, 1481, 1383, 1281, 1218, 1168, 1141, 1129, 1064, 1047, 1032, 1016, 989, 906, 884, 831, 752, 695, 601, 573, 495 cm⁻¹.

¹H NMR (400 MHz, CDCl₃) δ 10.40 (br, 1H), 7.32 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.24 – 7.21 (m, 2H), 7.21 – 7.15 (m, 1H), 3.01 (dq, *J* = 12.2, 7.2 Hz, 2H), 2.90 (dq, *J* = 12.2, 7.2 Hz, 2H), 1.94 – 1.69 (m, 7H), 1.61 – 1.53 (m, 2H), 1.24 (qt, *J* = 13.1, 3.8 Hz, 1H), 1.05 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.3, 145.7, 127.4, 127.2, 126.0, 124.0, 75.7, 50.7, 41.3, 25.8, 22.0, 12.9.

HRMS (ESI): calcd. for C₁₆H₂₅NO ([M+H]⁺): 248.2009, found: 248.2010.



(1R,3S,5r,7r)-2-(2-(diethylamino)phenyl)adamantan-2-ol (3ao)

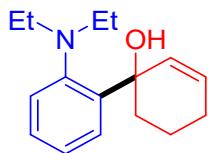
White solid. 23 mg, 77%

IR (KBr): 2968, 2902, 2851, 1595, 1573, 1481, 1450, 1384, 1334, 1289, 1186, 1163, 1139, 1118, 1103, 1044, 1011, 857, 794, 757, 736, 686, 665, 531 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 7.59 (d, *J* = 8.0 Hz, 1H), 7.27 (d, *J* = 7.8 Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 6.01 (br, 1H), 3.01 (dq, *J* = 14.1, 7.2 Hz, 2H), 2.84 (dq, *J* = 13.4, 7.3 Hz, 2H), 2.62 (d, *J* = 12.4 Hz, 2H), 2.45 (s, 2H), 1.90 (d, *J* = 12.5 Hz, 2H), 1.87 – 1.76 (m, 4H), 1.73 (s, 2H), 1.59 (d, *J* = 12.3 Hz, 2H), 1.04 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 151.7, 144.2, 127.9, 127.3, 125.0, 124.9, 51.0, 38.3, 37.0, 35.4, 33.5, 27.4, 26.9, 13.2.

HRMS (ESI): calcd. for C₂₀H₂₉NO ([M+H]⁺): 300.2322, found: 300.2323.



2'-(diethylamino)-3,4-dihydro-[1,1'-biphenyl]-1(2H)-ol (3ap)

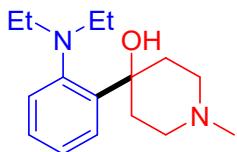
Colourless oil. 15 mg, 61%

IR (KBr): 3022, 2933, 2832, 1728, 1715, 1650, 1575, 1481, 1451, 1436, 1382, 1338, 1274, 1219, 1195, 1167, 1121, 1086, 1063, 853, 771, 755, 718, 662 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.16 (br, 1H), 7.27 – 7.19 (m, 3H), 7.18 – 7.12 (m, 1H), 5.97 – 5.90 (m, 1H), 5.75 (d, *J* = 9.9 Hz, 1H), 3.10 – 2.87 (m, 4H), 2.18 – 2.10 (m, 1H), 2.09 – 1.92 (m, 3H), 1.79 (td, *J* = 12.6, 3.0 Hz, 1H), 1.67 – 1.59 (m, 1H), 1.12 (t, *J* = 7.2 Hz, 3H), 1.04 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 148.0, 144.1, 133.8, 129.1, 128.5, 127.4, 125.9, 123.9, 74.4, 51.2, 49.8, 42.1, 25.0, 18.9, 13.0, 12.8.

HRMS (ESI): calcd. for C₁₆H₂₃NO ([M+H]⁺): 246.1852, found: 246.1854.



4-(2-(diethylamino)phenyl)-1-methylpiperidin-4-ol (3aq)

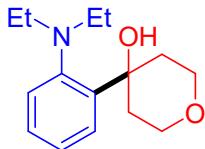
Colourless oil. 14 mg, 53%

IR (KBr): 3424, 2931, 2669, 2595, 2515, 1726, 1470, 1380, 1285, 1261, 1207, 1063, 1038, 980, 886, 773, 756, 609, 575, 546, 510, 474 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 11.56 (br, 1H), 7.45 (d, *J* = 7.5 Hz, 1H), 7.32 – 7.24 (m, 3H), 3.39 – 3.28 (m, 4H), 3.04 (dt, *J* = 14.3, 7.2 Hz, 2H), 2.92 (dt, *J* = 12.6, 7.0 Hz, 2H), 2.86 – 2.79 (m, 2H), 2.78 (s, 3H), 1.83 (d, *J* = 14.0 Hz, 2H), 1.03 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (151 MHz, CD₃OD) δ 146.8, 140.9, 128.5, 127.6, 127.1, 123.9, 71.4, 51.0, 50.8, 43.6, 38.3, 12.7.

HRMS (ESI): calcd. for C₁₆H₂₆N₂O ([M+H]⁺): 263.2118, found: 263.2119.



4-(2-(diethylamino)phenyl)tetrahydro-2H-pyran-4-ol (3ar)

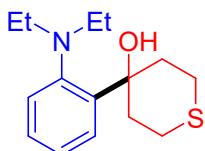
Colourless oil. 17 mg, 68%

IR (KBr): 2951, 2867, 1598, 1575, 1481, 1449, 1425, 1383, 1280, 1238, 1169, 1129, 1099, 1064, 1030, 1017, 978, 840, 769, 753, 707, 554, 537 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.78 (br, 1H), 7.32 (d, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 17.9 Hz, 3H), 4.02 (t, *J* = 11.8 Hz, 2H), 3.85 (dd, *J* = 11.2, 4.8 Hz, 2H), 3.03 (dq, *J* = 14.0, 7.2 Hz, 2H), 2.91 (dq, *J* = 13.7, 7.3 Hz, 2H), 2.18 (td, *J* = 12.8, 4.8 Hz, 2H), 1.64 (d, *J* = 13.1 Hz, 2H), 1.05 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.2, 143.8, 127.7, 127.3, 126.4, 124.1, 73.2, 64.1, 50.8, 41.5, 12.8.

HRMS (ESI): calcd. for C₁₅H₂₃NO₂ ([M+H]⁺): 250.1802, found: 250.1803.



4-(2-(diethylamino)phenyl)tetrahydro-2H-thiopyran-4-ol (3as)

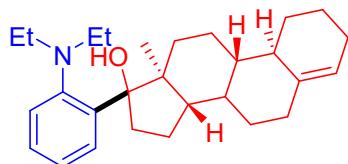
Colourless oil. 16 mg, 60%

IR (KBr): 3058, 3025, 2972, 2924, 2831, 1481, 1448, 1422, 1381, 1274, 1214, 1123, 1077, 1065, 970, 941, 895, 769, 752, 698, 657, 602, 574, 539 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 10.75 (br, 1H), 7.32 (d, *J* = 7.8 Hz, 1H), 7.27 – 7.20 (m, 3H), 3.34 (t, *J* = 13.3 Hz, 2H), 3.01 (dt, *J* = 14.0, 7.1 Hz, 2H), 2.90 (dq, *J* = 14.0, 7.2 Hz, 2H), 2.39 (d, *J* = 13.0 Hz, 2H), 2.13 (td, *J* = 13.0, 3.6 Hz, 2H), 2.00 (d, *J* = 13.2 Hz, 2H), 1.04 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 146.8, 144.4, 127.7, 127.4, 126.3, 124.1, 74.2, 50.8, 41.9, 24.3, 12.8.

HRMS (ESI): calcd. for C₁₅H₂₃NOS ([M+H]⁺): 266.1573, found: 266.1574.



(9S,10R,13S,14S,17S)-17-(2-(diethylamino)phenyl)-13-methyl-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-17-ol (3at)

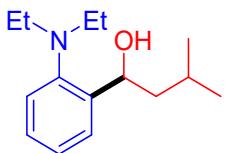
Yellow solid. 23 mg, 59%; $[\alpha]_D^{28.0} = +79.37$ (*c* 0.015, CHCl₃), m.p. 165.8–167.0 °C

IR (KBr): 2923, 2851, 1741, 1596, 1541, 1472, 1447, 1381, 1336, 1299, 1265, 1217, 1191, 1143, 1120, 1086, 1054, 1030, 837, 756, 739, 432 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 9.52 (br, 1H), 7.26 – 7.18 (m, 2H), 7.17 – 7.11 (m, 1H), 7.05 (d, *J* = 7.8 Hz, 1H), 5.36 (d, *J* = 2.7 Hz, 1H), 3.14 (dq, *J* = 12.1, 7.3 Hz, 1H), 2.96 (ddq, *J* = 26.6, 12.9, 7.0, 6.6 Hz, 2H), 2.75 (dq, *J* = 12.1, 7.2 Hz, 1H), 2.29 – 2.16 (m, 2H), 2.15 – 2.06 (m, 1H), 2.01 (t, *J* = 13.5 Hz, 1H), 1.92 – 1.80 (m, 3H), 1.78 – 1.68 (m, 3H), 1.64 – 1.58 (m, 2H), 1.53 (qd, *J* = 12.5, 5.4 Hz, 1H), 1.47 – 1.36 (m, 2H), 1.34 – 1.28 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.12 (qd, *J* = 13.4, 4.3 Hz, 1H), 1.05 (s, 3H), 0.99 – 0.94 (m, 1H), 0.93 (t, *J* = 7.2 Hz, 3H), 0.87 (td, *J* = 12.6, 3.5 Hz, 1H), 0.43 – 0.31 (m, 2H).

¹³C NMR (151 MHz, CDCl₃) δ 150.6, 142.9, 140.6, 128.9, 127.2, 125.1, 123.95, 119.7, 89.3, 51.8, 49.8, 49.7, 49.2, 49.0, 42.0, 41.9, 39.9, 35.6, 33.5, 31.9, 28.6, 26.2, 25.5, 24.2, 22.0, 15.8, 13.9, 12.3.

HRMS (ESI): calcd. for C₂₈H₄₁NO ([M+H]⁺): 408.3261, found: 408.3260.



1-(2-(diethylamino)phenyl)-3-methylbutan-1-ol (3au)

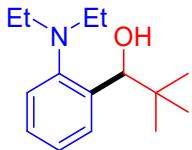
Colourless oil. 13mg, 55%

IR (KBr): 3026, 2955, 2933, 2868, 2818, 1959, 1597, 1486, 1465, 1449, 1383, 1367, 1293, 1234, 1170, 1120, 1064, 1045, 838, 793, 749, 545, 419 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 7.37 (br, 1H), 7.23 (dt, *J* = 3.5, 1.6 Hz, 2H), 7.14 (dd, *J* = 3.6, 2.0 Hz, 2H), 4.96 (dd, *J* = 10.2, 3.7 Hz, 1H), 3.05 – 2.90 (m, 4H), 2.03 – 1.90 (m, 1H), 1.73 (ddd, *J* = 14.0, 10.1, 4.5 Hz, 1H), 1.48 (ddd, *J* = 13.2, 9.1, 3.7 Hz, 1H), 1.06 (t, *J* = 7.2 Hz, 6H), 0.99 (dd, *J* = 10.5, 6.7 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 148.6, 141.5, 127.5, 127.2, 125.4, 123.6, 72.0, 49.6, 49.2, 24.7, 23.9, 21.7, 12.8.

HRMS (ESI): calcd. for C₁₅H₂₅NO ([M+H]⁺): 236.2009, found: 236.2010.



1-(2-(diethylamino)phenyl)-2,2-dimethylpropan-1-ol (3av)

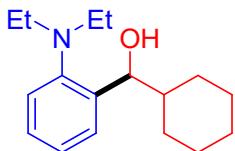
Colourless oil. 11 mg, 47%

IR (KBr): 2971, 2869, 1483, 1463, 1447, 1385, 1361, 1294, 1235, 1215, 1179, 1169, 1119, 1099, 1069, 1042, 1009, 825, 794, 767 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 7.25 – 7.20 (m, 2H), 7.15 (d, *J* = 7.3 Hz, 2H), 7.11 (ddd, *J* = 8.0, 5.7, 2.6 Hz, 1H), 4.67 (s, 1H), 3.05 (dq, *J* = 12.9, 7.4 Hz, 2H), 2.85 (dq, *J* = 14.3, 7.1 Hz, 2H), 1.07 (t, *J* = 7.1 Hz, 6H), 0.95 (s, 9H).

¹³C NMR (151 MHz, CDCl₃) δ 150.7, 137.4, 130.0, 127.4, 124.3, 123.9, 82.5, 50.0, 37.6, 26.5, 12.8.

HRMS (ESI): calcd. for C₁₅H₂₅NO ([M+H]⁺): 236.2009, found: 236.2010.



Cyclohexyl(2-(diethylamino)phenyl)methanol (**3aw**)

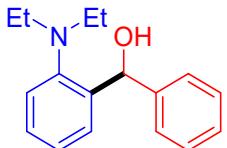
Colourless oil. 15 mg, 57%

IR (KBr): 3358, 2971, 2851, 1959, 1594, 1509, 1450, 1419, 1385, 1365, 1315, 1083, 1042, 925, 866, 837, 817, 668, 611, 579, 484, 472, 419 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 7.26 – 7.20 (m, 2H), 7.16 – 7.07 (m, 2H), 4.55 (d, *J* = 7.2 Hz, 1H), 2.99 (ddd, *J* = 14.4, 9.7, 6.5 Hz, 2H), 2.91 (ddt, *J* = 11.6, 7.0, 3.7 Hz, 2H), 1.96 (d, *J* = 12.3 Hz, 1H), 1.80 – 1.68 (m, 2H), 1.66 – 1.55 (m, 2H), 1.43 – 1.36 (m, 1H), 1.22 – 1.10 (m, 5H), 1.08 – 1.01 (m, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 149.4, 139.7, 128.7, 127.2, 125.1, 123.8, 79.3, 49.8, 46.1, 30.3, 28.5, 26.5, 26.3, 12.8.

HRMS (ESI): calcd. for C₁₇H₂₇NO ([M+H]⁺): 262.2165, found: 262.2166.



(2-(diethylamino)phenyl)(phenyl)methanol (**3ax**)

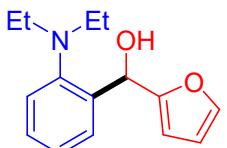
Colourless oil. 10 mg, 39%

IR (KBr): 3061, 3028, 2972, 2931, 2872, 1650, 1598, 1580, 1487, 1451, 1383, 1293, 1234, 1172, 1120, 1097, 1062, 1026, 794, 765, 744, 699, 650, 419 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 8.24 (br, 1H), 7.39 (d, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.28 – 7.23 (m, 3H), 7.10 (ddd, *J* = 8.3, 5.7, 2.8 Hz, 1H), 6.97 (d, *J* = 7.7 Hz, 1H), 6.00 (s, 1H), 2.90 (qd, *J* = 12.7, 6.3 Hz, 4H), 1.02 (t, *J* = 7.2 Hz, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 148.7, 144.6, 140.0, 129.3, 128.1, 127.7, 126.9, 126.9, 125.2, 123.8, 76.1, 49.0, 12.5.

HRMS (ESI): calcd. for C₁₇H₂₁NO ([M+H]⁺): 256.1696, found: 256.1697.



(2-(diethylamino)phenyl)(furan-2-yl)methanol (**3ay**)

Colourless oil. 13 mg, 53%

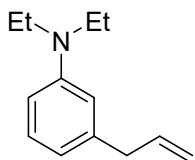
IR (KBr): 2972, 2930, 2872, 1663, 1597, 1581, 1488, 1449, 1382, 1295, 1238, 1224, 1171, 1085, 1064, 1031, 1008, 939, 834, 815, 793, 766, 733, 598, 563, 459 cm⁻¹.

¹H NMR (600 MHz, CDCl₃) δ 8.71 (br, 1H), 7.38 – 7.36 (m, 1H), 7.29 (dd, *J* = 16.5,

8.2 Hz, 2H), 7.18 – 7.13 (m, 1H), 7.10 (d, J = 7.6 Hz, 1H), 6.31 (dd, J = 3.3, 1.8 Hz, 1H), 6.18 (d, J = 3.2 Hz, 1H), 6.02 (s, 1H), 3.03 – 2.89 (m, 4H), 1.04 (t, J = 7.1 Hz, 6H).

^{13}C NMR (151 MHz, CDCl_3) δ 157.2, 148.6, 141.9, 137.1, 128.9, 128.0, 125.5, 123.8, 110.0, 106.39, 71.1, 49.2, 12.5.

HRMS (ESI): calcd. for $\text{C}_{15}\text{H}_{19}\text{NO}_2$ ($[\text{M}+\text{H}]^+$): 246.1489, found: 246.1491.



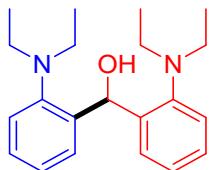
3-allyl-N,N-diethylaniline (5)

Yellow oil, 16 mg, 85%

^1H NMR (600 MHz, CDCl_3) δ 7.14 (t, J = 7.8 Hz, 1H), 6.55 – 6.45 (m, 3H), 6.03 – 5.91 (m, 1H), 5.10 (d, J = 16.2 Hz, 1H), 5.04 (d, J = 10.0 Hz, 1H), 3.37 – 3.29 (m, 6H), 1.15 (t, J = 7.1 Hz, 6H).

^{13}C NMR (151 MHz, CDCl_3) δ 148.0, 141.1, 137.9, 129.2, 115.7, 115.3, 112.1, 109.8, 44.3, 40.8, 12.6.

HRMS (ESI): calcd. for $\text{C}_{13}\text{H}_{19}\text{N}$ ($[\text{M}+\text{H}]^+$): 190.1590, found: 190.1590.



bis(2-(diethylamino)phenyl)methanol (8)

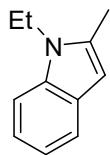
Colourless oil.

IR (KBr): 3384, 3061, 3027, 2971, 2932, 2872, 2816, 1598, 1485, 1450, 1382, 1295, 1237, 1174, 1118, 1093, 1017, 794, 756, 712, 668, 646, 484, 472, 419 cm^{-1} .

^1H NMR (600 MHz, CDCl_3) δ 8.11(br, 1H), 7.23 (dt, J = 13.5, 7.7 Hz, 4H), 7.05 (t, J = 7.2 Hz, 2H), 6.99 (d, J = 7.7 Hz, 2H), 6.75 (s, 1H), 3.05 (q, J = 7.1 Hz, 8H), 1.05 (t, J = 7.1 Hz, 12H).

^{13}C NMR (151 MHz, CDCl_3) δ 149.3, 141.4, 129.3, 127.4, 124.7, 122.8, 69.4, 48.7, 12.7.

HRMS (ESI): calcd. for $\text{C}_{21}\text{H}_{30}\text{N}_2\text{O}$ ($[\text{M}+\text{H}]^+$): 327.2430, found: 327.2435.

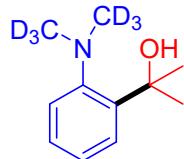


1-ethyl-2-methyl-1H-indole(9)

Colorless oil.

¹H NMR (600 MHz, CDCl₃) δ 7.52 (d, *J* = 7.8 Hz, 1H), 7.32 – 7.20 (m, 1H), 7.13 (t, *J* = 7.5 Hz, 1H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.23 (s, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 1.34 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 136.2, 136.0, 128.2, 120.4, 119.7, 119.1, 108.8, 99.8, 37.7, 15.3, 12.6. The spectra data were consistent with the literature.⁶



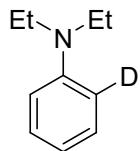
2-(2-(bis(methyl-d3)amino)phenyl)propan-2-ol (3b'a)

Colorless oil, 13 mg, 70%

¹H NMR (600 MHz, CDCl₃) δ 9.58 (s, 1H), 7.35 (d, *J* = 7.9 Hz, 1H), 7.30 – 7.22 (m, 2H), 7.20 – 7.14 (m, 1H), 1.58 (s, 6H).

¹³C NMR (151 MHz, CDCl₃) δ 151.0, 144.0, 127.6, 126.6, 126.2, 123.4, 73.9, 45.6 (hept, *J* = 21.0 Hz), 32.6.

HRMS (ESI): calcd. for C₁₁H₁₁D₆NO ([M+H]⁺): 186.1760, found: 186.1758.



N,N-diethylaniline-2-d (4a')

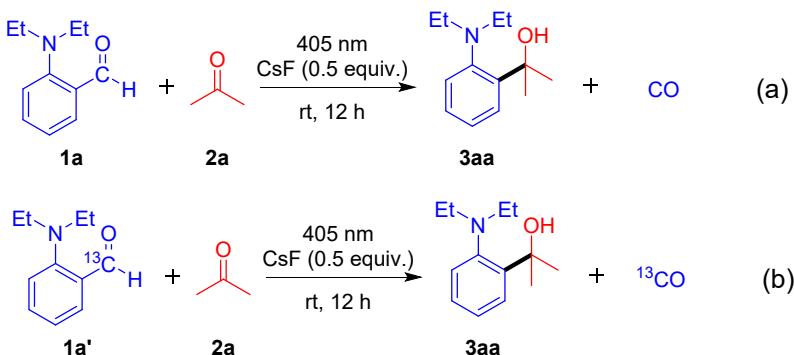
Colorless oil.

¹H NMR (600 MHz, CDCl₃) δ 7.2 (t, *J* = 6.3 Hz, 2H), 6.7 (d, *J* = 8.5 Hz, 1H), 6.6 (t, *J* = 7.3 Hz, 1H), 3.4 (q, *J* = 7.0 Hz, 4H), 1.2 (t, *J* = 6.9 Hz, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 147.7, 129.2, 129.1, 115.3, 111.8, 111.4 (t, *J* = 22.8 Hz), 44.3, 12.6.

HRMS (ESI): calcd. for C₁₀H₁₄DN ([M+H]⁺): 152.1374, found: 152.1377.

5. GC-MS data of CO gas



Scheme S1 Analysis of the reaction atmosphere by GC-MS

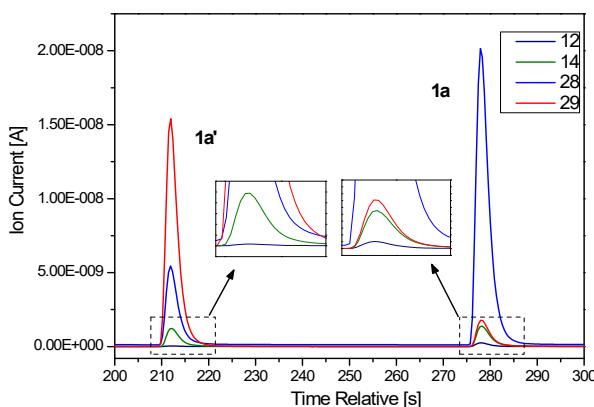


Figure S1 GC-MS analysis of the gas phase mixture of $1\mathbf{a}'$ and $1\mathbf{a}$

It can be seen from Figure S1 that the intensity of the signal peak with mass-to-charge ratio (m/e) of 29 in the gas phase component of $1\mathbf{a}'$ (Scheme S1-b) was significantly higher than that of the system of $1\mathbf{a}$ (Scheme S1-a), which indicated that the ^{13}C isotope labelled carbon monoxide is formed in $1\mathbf{a}'$ system.

6. Emission spectra of blue LED

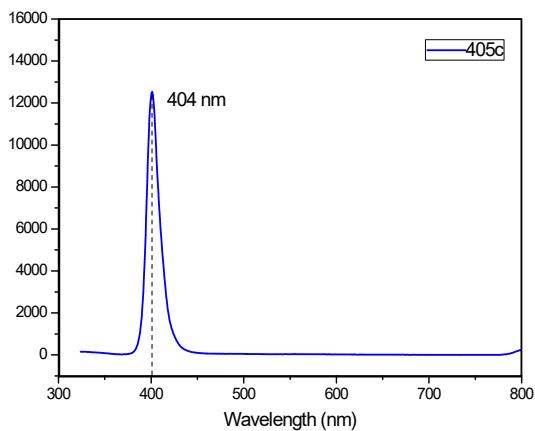


Figure S2 Emission spectra of blue LED

7. UV-vis absorption spectra

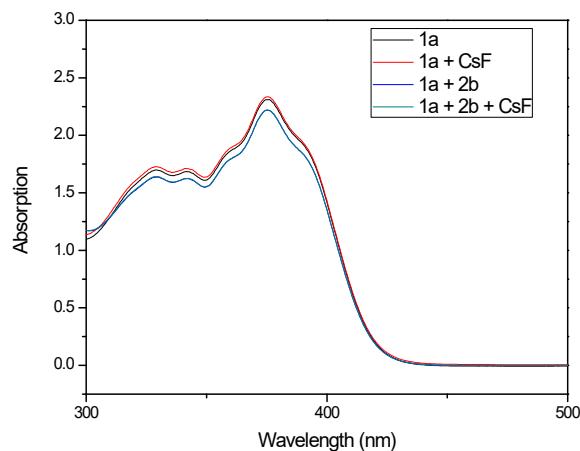


Figure S3 UV-vis absorption spectra

The UV-vis absorption spectra were recorded in toluene with the combination of **1a**, **2b** and CsF. **[1a]** = 1.0 M: 1.77 mg dissolved in 10 ml toluene; **[1a + CsF]**: solution of 1.77 mg of **1a** and 0.07 mg of CsF in 10 ml toluene; **[1a + 2b]** : solution of 1.77 mg of **1a** and 6.88 mg of **2b** in 10 ml toluene; **[1a + 2b + CsF]** : solution of 1.77 mg of **1a** , 6.88 mg of **2b** and 0.07 mg of CsF in 10 ml toluene.

8. Stern-Volmer quenching studies with ketone **2b**

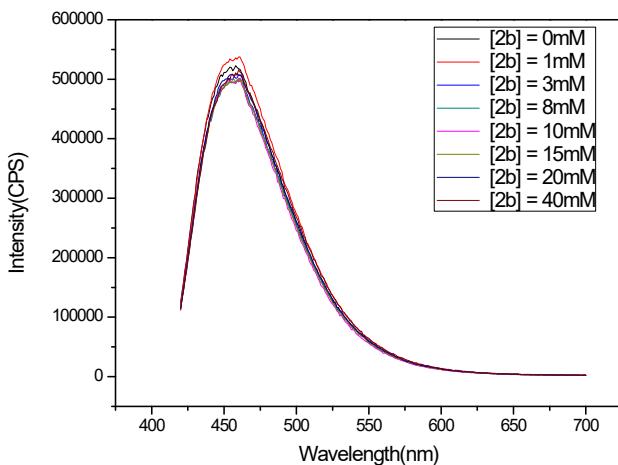


Figure S4 Quenching of the **1a** in the presence of **2b**

The samples were prepared by mixing compound **1a** ($[1\mathbf{a}] = 10^{-3}$ M) with the different amounts of **2b** in 2 mL toluene in a 10×10 mm light path quartz fluorescence cuvette equipped with Silicone/PTFE 3.2 mm septum. The excitation wavelength was fixed at 405 nm (incident light slit regulated to 1 mm), and the emission light was acquired from 400 nm to 700 nm (emission light slit regulated to 5 mm). A solvent blank was acquired from all measurements.

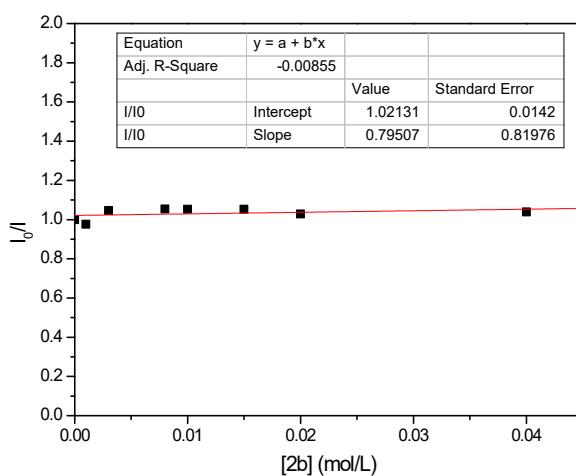
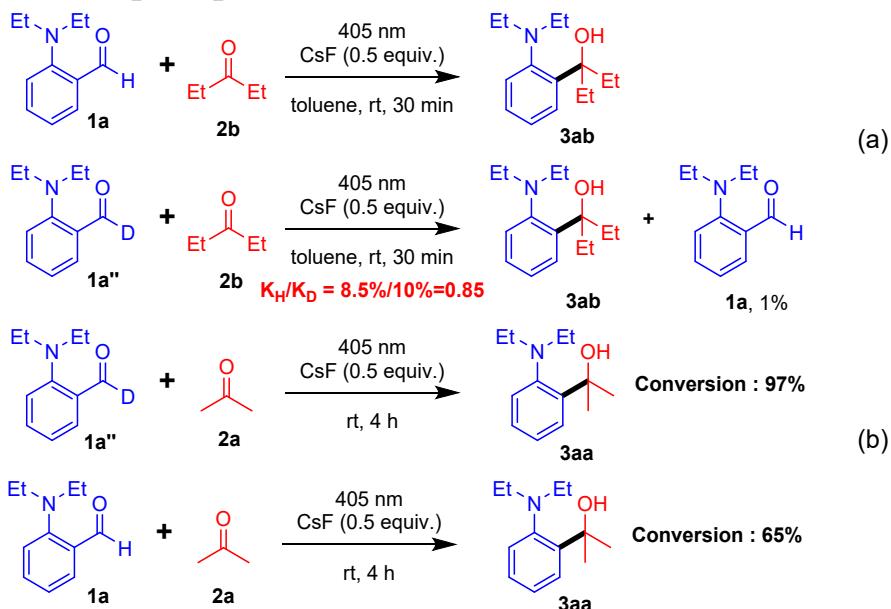


Figure S5 Stern-Vomer quenching plot

The Stern-Vomer plot shows a linear correlation between the concentration of **2b** and the ratio I_0/I . The Stern-Vomer constant K_{sv} could be calculated of 0.795 M^{-1} , based on the following Equation S1.

$$I_0/I = 1 + K_{sv}[Q] \quad \text{Eq. S1}$$

9. Kinetic isotope experiments



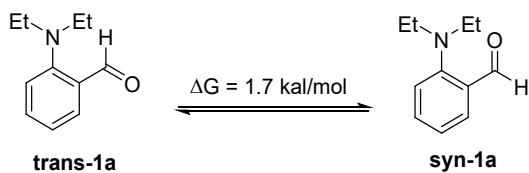
Scheme S2 Kinetic isotope experiments

The conversion of isotope labelled substrate **1a''** was significantly higher than that of normal substrate **1a** (Scheme S2), which maybe due to the much higher bond energy of C-D bond than C-H bond, thus hindering the vibrational relaxation process of excited state and prolonging the lifetime of single excited state (S_1)⁷

10. Computational details

All the calculations were carried out with Gaussian 16 software package.⁸ Geometry optimizations were carried out at CAM-B3LYP/6-311G(d,p)¹⁰, SDD¹¹ for Cs/IEFPCM¹²(toluene) level of theory. Vibrational frequencies were also calculated for all stationary points to verify them as energy minima or transition states. Intrinsic reaction coordinate (IRC)¹³ calculations were carried out as well to confirm whether the transition states were connected with expected reactants and products. Time-dependent density functional theory (TD-DFT)¹⁴ calculation was also performed at CAM-B3LYP/6-311G(d,p), SDD for Cs /IEFPCM (toluene) level of theory and first 6 excited states were reported. The orbitals were displayed using VMD 1.9.3.¹⁵ The optimized geometries were displayed using CYLview20.¹⁶

i. Configuration study



1a has two possible conformations, where the Gibbs free energy of **syn-1a** is 1.7 kcal/mol higher than that of **trans-1a**. Thus, **trans-1a** is the more stable conformation and use as the main conformation for the next calculation.

ii Time-dependent density functional theory calculations

TD-DFT calculations of **1a**. Orbital 48 is HOMO and orbital 49 is LUMO.

Excited State 1: Singlet-A 3.7037 eV 334.76 nm f=0.0326 <S**2>=0.000

45 -> 49	0.12242
46 -> 49	0.42599 (n _O , π*) 36% contribution
46 -> 51	0.11102
48 -> 49	0.51333 (n _N , π*) 53% contribution

Excited State 2: Singlet-A 4.2036 eV 294.95 nm f=0.0782 <S**2>=0.000

45 -> 49	-0.10892
46 -> 49	-0.46490
46 -> 51	-0.10131
47 -> 49	-0.10916
48 -> 49	0.46887

Excited State 3: Singlet-A 5.0698 eV 244.55 nm f=0.0267 <S**2>=0.000

45 -> 49	0.15919
47 -> 49	-0.44822
47 -> 50	0.10534
48 -> 50	0.49328

Excited State 4: Singlet-A 5.5426 eV 223.69 nm f=0.5119 <S**2>=0.000

46 -> 49	-0.10934
47 -> 49	0.50531
48 -> 50	0.45457

Excited State 5: Singlet-A 5.8194 eV 213.05 nm f=0.0159 <S**2>=0.000

45 -> 49	0.58074
46 -> 49	-0.16689
47 -> 50	0.26556
48 -> 50	-0.18310

Excited State 6: Singlet-A 6.5345 eV 189.74 nm f=0.1066 <S**2>=0.000

45 -> 49	0.21773
46 -> 49	-0.13475
47 -> 50	-0.41334
48 -> 51	0.42619
48 -> 52	-0.21983

Orbital of **1a**, (localized by PM-Mulliken method¹⁶ with Multiwfn 3.7¹⁷)

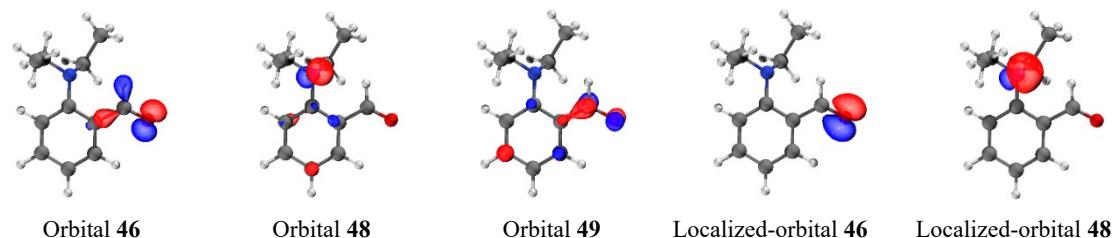


Figure S6

TD-DFT Calculations of **1a-Cs**. Orbital 52 is HOMO and orbital 53 is LUMO.

Excited State 1: Singlet-A 3.5734 eV 346.97 nm f=0.0840 <S**2>=0.000

49 -> 53	0.14972	(n _O , π*) 4% contribution
50 -> 53	-0.16679	
52 -> 53	0.65922	(n _N , π*) 87% contribution

Excited State 2: Singlet-A 4.2130 eV 294.29 nm f=0.0342 <S**2>=0.000

49 -> 53	-0.39608
49 -> 60	0.12867
50 -> 53	0.49254
50 -> 60	-0.111153
52 -> 53	0.22902

Excited State 3: Singlet-A 4.9578 eV 250.08 nm f=0.0490 <S**2>=0.000

49 -> 53 0.15407
51 -> 53 0.55445
52 -> 58 0.36756

Excited State 4: Singlet-A 5.4238 eV 228.59 nm f=0.4908 <S**2>=0.000

49 -> 53 0.17168
50 -> 53 0.15787
51 -> 53 -0.41752
52 -> 58 0.47914

Excited State 5: Singlet-A 5.5371 eV 223.91 nm f=0.0015 <S**2>=0.000

49 -> 53 0.12684
50 -> 53 0.11469
52 -> 54 0.63385
52 -> 57 0.22531

Excited State 6: Singlet-A 5.5976 eV 221.49 nm f=0.0408 <S**2>=0.000

49 -> 53 0.43653
50 -> 53 0.36385
51 -> 58 -0.17096
52 -> 54 -0.16348
52 -> 58 -0.31865

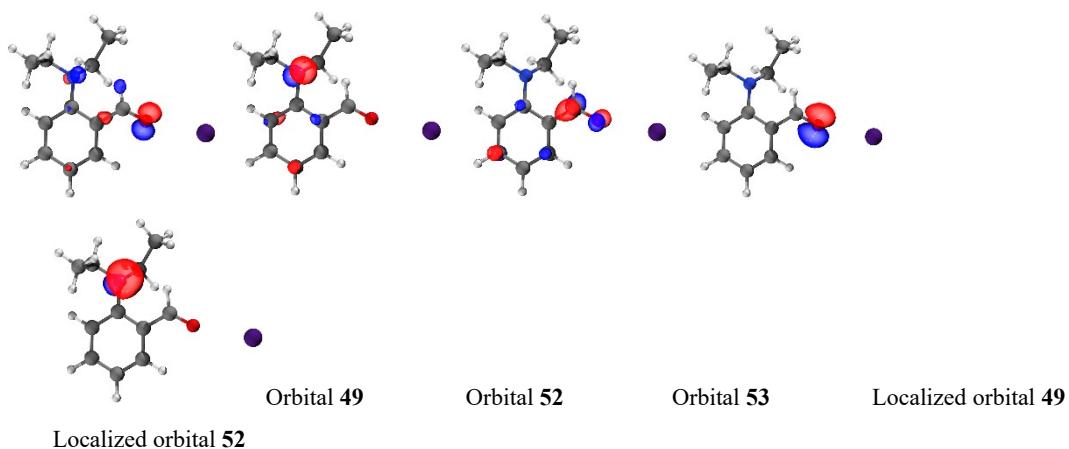


Figure S7

TD-DFT Calculations of **1n**. Orbital 48 is HOMO and orbital 49 is LUMO.

Excited State 1: Singlet-A 3.9498 eV 313.90 nm f=0.0162 <S**2>=0.000

30 -> 33 0.61830 (n_O, π*) 76% contribution

30 -> 36 0.13937

30 -> 37 -0.15007

32 -> 33 -0.25287

Excited State 2: Singlet-A 4.1025 eV 302.22 nm f=0.1317 <S**2>=0.000

30 -> 33 0.23969

31 -> 34 -0.11368

32 -> 33 0.64532

Excited State 3: Singlet-A 5.2623 eV 235.61 nm f=0.1152 <S**2>=0.000

31 -> 33 0.61811

32 -> 34 0.30600

Excited State 4: Singlet-A 5.9383 eV 208.79 nm f=0.5305 <S**2>=0.000

31 -> 33 -0.31506

32 -> 34 0.61213

Excited State 5: Singlet-A 6.4529 eV 192.14 nm f=0.0579 <S**2>=0.000

29 -> 33 0.12390

29 -> 35 0.10969

31 -> 34 0.23941

32 -> 34 0.11341

32 -> 35 0.62117

Excited State 6: Singlet-A 6.5721 eV 188.65 nm f=0.0830 <S**2>=0.000

29 -> 33 0.19148

31 -> 34 0.48472

32 -> 35 -0.26525

32 -> 36 0.21205

32 -> 37 -0.27909

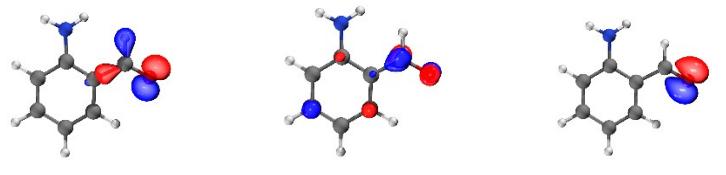


Figure S8

TD-DFT Calculations of **1o**. Orbital 48 is HOMO and orbital 49 is LUMO.

Excited State 1: Singlet-A 4.1194 eV 300.98 nm f=0.0004 <S**2>=0.000

47 -> 49 0.66603 (n_O, π*) 89% contribution

47 -> 52 0.11394

47 -> 54 0.14562

47 -> 55 0.10150

Excited State 2: Singlet-A 4.3911 eV 282.35 nm f=0.6913 <S**2>=0.000

48 -> 49 0.68506

48 -> 50 0.10369

Excited State 3: Singlet-A 4.7677 eV 260.05 nm f=0.0696 <S**2>=0.000

46 -> 49 -0.29970

48 -> 49 -0.10383

48 -> 50 0.62350

Excited State 4: Singlet-A 5.8062 eV 213.54 nm f=0.1841 <S**2>=0.000

46 -> 49 0.62578

48 -> 50 0.30920

Excited State 5: Singlet-A 6.3108 eV 196.46 nm f=0.0207 <S**2>=0.000

45 -> 51 0.10168

48 -> 51 0.67026

48 -> 53 -0.13266

Excited State 6: Singlet-A 6.7195 eV 184.51 nm f=0.0807 <S**2>=0.000

45 -> 49 0.40374

46 -> 50 0.25301

48 -> 52	-0.38623
48 -> 53	0.18317
48 -> 54	-0.21441
48 -> 55	-0.10921

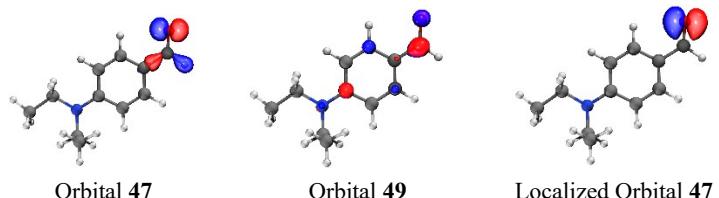


Figure S9

iii Potential complete energy surface

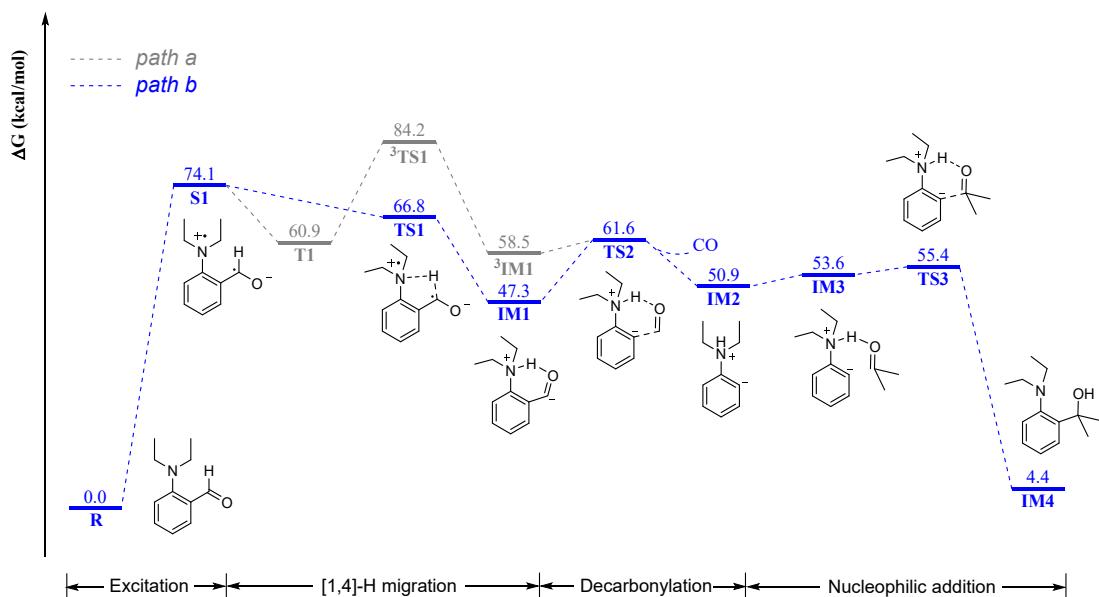


Figure S10 Potential complete energy surface

iv Cartesian coordinate

trans-1a

Charge = 0 Multiplicity = 1

C	0.73638100	-1.67813800	-0.46015600
C	2.10556000	-1.87053100	-0.55143700
C	2.99131800	-0.82190600	-0.33202800
C	2.48504200	0.41549000	0.01330800
C	1.11173400	0.61979200	0.13789500
C	0.20714800	-0.42472800	-0.13742300
N	-1.18209400	-0.17454500	-0.09048600
C	-2.06048800	-1.31829000	0.14452300
C	-1.88014000	-1.93628900	1.52276600
C	-1.69154300	0.72904900	-1.13558800

C	-2.84019600	1.61434000	-0.67391300
C	0.64936600	1.92062800	0.66236200
O	1.35930000	2.89200300	0.78772600
H	0.07625900	-2.50967900	-0.66299000
H	2.48405900	-2.85209100	-0.81250500
H	4.05945400	-0.97707900	-0.41594300
H	3.13851600	1.25267300	0.22646600
H	-3.08283500	-0.95023300	0.06276700
H	-1.95411600	-2.08013100	-0.64241400
H	-2.58750000	-2.75649200	1.66142600
H	-0.87448900	-2.33286900	1.66662700
H	-2.06198700	-1.18919500	2.29721600
H	-0.86716600	1.35554800	-1.47464200
H	-2.00489100	0.13779500	-2.00930000
H	-3.16233400	2.25860500	-1.49483800
H	-3.70669500	1.03296300	-0.35510000
H	-2.53638300	2.25037200	0.15904300
H	-0.40472500	1.94909800	0.98390900

syn-1a

Charge = 0 Multiplicity = 1

C	-1.20995900	1.40903100	-0.37405300
C	-2.58957200	1.32106900	-0.40645100
C	-3.23398100	0.10641700	-0.21186300
C	-2.45817100	-1.00192300	0.05404100
C	-1.06136900	-0.94425100	0.10925000
C	-0.39611100	0.28531400	-0.15251300
N	0.99237400	0.39122900	-0.17234400
C	1.58450400	1.72070600	-0.04548400
C	1.38513500	2.33834000	1.33151600
C	1.73012000	-0.48252600	-1.09680400
C	3.06764100	-0.96453400	-0.55674000
C	-0.42760100	-2.17568900	0.60708200
O	0.74182800	-2.37815800	0.83080200
H	-0.75531000	2.37141900	-0.55419200
H	-3.16778500	2.21683300	-0.60228700
H	-4.31316900	0.03524100	-0.24671700
H	-2.93525100	-1.95501000	0.25687900
H	2.65321400	1.60226100	-0.21451000
H	1.23387000	2.39874200	-0.83684500
H	1.88232600	3.30944300	1.38271900
H	0.33151400	2.48753400	1.56948000
H	1.81343200	1.68901200	2.09654100

H	1.10821000	-1.34207900	-1.32704000
H	1.87997800	0.06032700	-2.04233100
H	3.53362000	-1.63570100	-1.28229200
H	3.76541700	-0.14354700	-0.38108800
H	2.91434500	-1.50688600	0.37333500
H	-1.16946600	-2.97265100	0.82045000

1n

Charge = 0 Multiplicity = 1

C	-0.04843900	-1.41161700	0.02651800
C	1.29991300	-1.68794900	0.01652200
C	2.19999300	-0.62173900	-0.01560900
C	1.75480700	0.68314000	-0.02201900
C	0.38345100	0.97897900	-0.00078300
C	-0.52729400	-0.09687200	0.00556400
H	-0.78571400	-2.20481800	0.03868900
H	1.65639100	-2.70963600	0.02800100
H	3.26643300	-0.81499300	-0.02882200
H	2.46772000	1.50007400	-0.03594000
N	-0.02328800	2.29856700	-0.03200100
H	-0.90077000	2.53518800	0.39957600
H	0.69630200	2.97980800	0.15135400
C	-1.98054900	0.10793000	-0.05123000
H	-2.32503700	1.15095800	-0.18489900
O	-2.80045100	-0.77972200	0.01278300

1o

Charge = 0 Multiplicity = 1

C	0.31949600	-0.01574400	-0.18552000
C	-0.36238700	1.19747800	-0.43225700
C	-1.73760300	1.26330200	-0.37674100
C	-2.50837200	0.14590800	-0.06066000
C	-1.84574500	-1.05165900	0.20979200
C	-0.47367300	-1.13753900	0.15815200
H	0.18474200	2.09660900	-0.66993900
H	-2.22870500	2.20999500	-0.57916500
H	-2.43646500	-1.92149200	0.47133100
H	-0.01116000	-2.08301600	0.39386100
C	-3.96590400	0.23496800	-0.00235300
H	-4.37581900	1.24073600	-0.23154200
O	-4.71199000	-0.67967600	0.26769800
N	1.68756300	-0.11019600	-0.28196100

C	2.49588200	1.10024800	-0.37081200
H	2.05112500	1.76678500	-1.10945600
C	2.33143200	-1.36881300	0.10465500
H	2.19259000	-1.55301100	1.17867500
H	1.82087500	-2.17807600	-0.41997100
C	3.81001500	-1.46820900	-0.23418900
H	4.42497900	-0.77081500	0.33602500
H	4.14976000	-2.47628000	0.00902600
H	3.99011300	-1.30436400	-1.29828600
H	3.46569800	0.83013500	-0.78094200
C	2.68046000	1.82210900	0.96171600
H	3.18562500	1.17940200	1.68613100
H	3.28700700	2.72060700	0.82744400
H	1.72100400	2.11927500	1.38825700

S1

Charge = 0 Multiplicity = 1

C	0.95264700	-1.67369100	-0.33596500
C	2.34201000	-1.71801500	-0.42092300
C	3.07635500	-0.52963100	-0.31395200
C	2.45516800	0.68306300	-0.12518300
C	1.04210400	0.74185700	-0.03081100
C	0.30453300	-0.46966500	-0.14251000
N	-1.11318100	-0.35271300	-0.07158800
C	-1.77446900	-1.01392600	1.05187700
C	-1.26094600	-0.54911100	2.40395400
C	-1.81143700	-0.39457300	-1.36123900
C	-3.11753900	0.38536900	-1.38513900
C	0.26610200	1.90668400	0.16694400
O	0.59023800	3.09643000	0.30553300
H	0.37115500	-2.58500900	-0.42438900
H	2.84600600	-2.66410500	-0.56741700
H	4.15820300	-0.56557800	-0.37597400
H	3.03133400	1.59460600	-0.03730100
H	-2.84405000	-0.82268600	0.97134200
H	-1.63494500	-2.09968200	0.94925800
H	-1.77533100	-1.09583000	3.19571200
H	-0.19072600	-0.72903800	2.50556900
H	-1.44522900	0.51661000	2.54532500
H	-1.12355600	0.01644200	-2.09908200
H	-1.98811700	-1.44462100	-1.63311000
H	-3.53165700	0.35737100	-2.39426800
H	-3.86889500	-0.02832900	-0.71202000

H	-2.94793800	1.42961100	-1.11728900
H	-0.83307000	1.60762100	0.20818300

T1

Charge = 0 Multiplicity = 3

C	0.68220700	-1.79705700	-0.23990000
C	2.03197100	-2.05996100	-0.25879700
C	2.95264800	-0.95690300	-0.25458600
C	2.52836400	0.32735800	-0.13643700
C	1.14338900	0.63175200	0.04985600
C	0.18447500	-0.48497000	-0.17952400
N	-1.15853900	-0.21426200	-0.27950800
C	-2.13764900	-1.28551900	-0.08872000
C	-2.22811000	-1.75129600	1.35799500
C	-1.63544600	0.93421400	-1.06491200
C	-2.73574500	1.73794800	-0.38872600
C	0.78785900	1.90567400	0.57459100
O	1.61416700	2.82338100	0.74927300
H	-0.00802100	-2.62176900	-0.35120400
H	2.39576800	-3.07348000	-0.35072600
H	4.01498500	-1.161111000	-0.33271900
H	3.22543600	1.151113200	-0.07097200
H	-3.10259900	-0.89966000	-0.41033900
H	-1.90658200	-2.12068500	-0.75707100
H	-2.97923900	-2.53840300	1.44494900
H	-1.27667000	-2.14615200	1.71364200
H	-2.51834200	-0.92469700	2.00814300
H	-0.78329200	1.56908800	-1.29202900
H	-1.99107200	0.53830900	-2.02349900
H	-3.03105400	2.56011200	-1.04269400
H	-3.62649000	1.14059900	-0.18960400
H	-2.39360000	2.16299000	0.55524900
H	-0.26657300	2.06907900	0.83620500

TS1

Charge = 0 Multiplicity = 1

Imaginary frequency = -205.78 cm⁻¹

C	1.13962900	-1.29107700	-0.74455000
C	2.51668100	-1.19163900	-0.86534500
C	3.17340200	-0.06586800	-0.36823200
C	2.46597200	0.94759900	0.25232000

C	1.07540800	0.88489900	0.36976900
C	0.45572100	-0.25120000	-0.13573700
N	-1.02411400	-0.25714200	-0.00812500
C	-1.62555600	-1.57277800	0.38186600
C	-1.06716600	-2.08749300	1.69445300
C	-1.66314900	0.38621700	-1.21694900
C	-3.14253900	0.66144800	-1.03653700
H	0.62571500	-2.16398700	-1.12884900
H	3.07529900	-1.98818700	-1.33979200
H	4.25013100	0.01158000	-0.46819100
H	2.98325500	1.81029100	0.65925900
C	0.24067400	1.86230600	1.21624400
H	-1.20058300	0.39932300	0.77214700
H	-1.45806500	-2.26744000	-0.44021700
H	-2.69733800	-1.40783400	0.46491700
H	-1.60993100	-2.98942500	1.97937700
H	-1.19411400	-1.35117700	2.49036100
H	-0.00877800	-2.33141400	1.62074900
H	-1.45534800	-0.26681200	-2.06473100
H	-1.12058000	1.32233000	-1.33439200
H	-3.48224100	1.25825300	-1.88379700
H	-3.32190700	1.24952800	-0.13403800
H	-3.75175300	-0.24175700	-1.00620400
O	-0.48492800	2.63278100	0.57330800

³TS1

Charge = 0 Multiplicity = 3

Imaginary frequency = -406.10 cm⁻¹

C	1.28820600	-1.25317400	-0.78564200
C	2.65836100	-1.02479000	-0.80988900
C	3.17399300	0.13912100	-0.24567800
C	2.34205700	1.08011000	0.33862600
C	0.95164100	0.89190400	0.37708100
C	0.47715500	-0.30211400	-0.20194900
N	-0.97941600	-0.41610500	-0.10855800
C	-1.49684400	-1.72677900	0.37793000
C	-1.01294200	-2.04851100	1.77864500
C	-1.69046500	0.08815300	-1.33537900
C	-3.01091200	0.74572800	-0.98415800
H	0.87724700	-2.15687600	-1.22094900
H	3.31880400	-1.75320500	-1.26280900
H	4.24373500	0.31264900	-0.26587200
H	2.75258400	1.97967600	0.77978700

C	-0.00967700	1.77517100	1.02850700
H	-1.11672500	0.34870200	0.65060600
H	-1.19406900	-2.49076200	-0.33964300
H	-2.58378600	-1.66056300	0.35239100
H	-1.45218800	-2.99344100	2.10039400
H	-1.31475900	-1.27449000	2.48600100
H	0.07168900	-2.14539200	1.81719200
H	-1.80264700	-0.75694100	-2.01695300
H	-1.02423500	0.81572600	-1.79274400
H	-3.48396000	1.10502000	-1.89894600
H	-2.82433300	1.60366000	-0.33537700
H	-3.70789000	0.06112400	-0.49814900
O	-0.74087500	2.71611700	0.62205200

IM1

Charge = 0 Multiplicity = 1

C	1.08594300	-1.48550200	-0.22996900
C	2.47198900	-1.50421200	-0.32730600
C	3.19129700	-0.31815600	-0.30761600
C	2.51342000	0.88441700	-0.18944700
C	1.12271200	0.93157500	-0.08837400
C	0.41117300	-0.27892600	-0.11159300
N	-1.03811400	-0.23436700	-0.03038900
C	-1.57999100	-0.97550800	1.12354400
C	-1.11014300	-0.40857700	2.45303900
C	-1.66346400	-0.62239400	-1.31330100
C	-3.09101500	-0.11752100	-1.45905300
H	0.53359200	-2.41730500	-0.24909500
H	2.98860200	-2.45215600	-0.42059700
H	4.27148700	-0.33365200	-0.38360400
H	3.04194100	1.83000200	-0.16772900
C	0.55779900	2.32075900	0.04806900
H	-1.11291200	1.39305500	0.13280800
H	-1.32124900	-2.04034900	1.04954800
H	-2.66631000	-0.90934200	1.06718100
H	-1.56679700	-0.96461900	3.27347200
H	-1.39379000	0.64051400	2.55377600
H	-0.02703700	-0.48007200	2.56153000
H	-1.62811800	-1.71332500	-1.43619400
H	-1.04982800	-0.19374500	-2.10679200
H	-3.46556100	-0.36951700	-2.45290300
H	-3.13225800	0.96766000	-1.34804800
H	-3.77037500	-0.56414200	-0.73215700

O -0.73661400 2.33647800 0.16044500

³IM1

Charge = 0 Multiplicity = 3

C 1.12151300 -1.49805400 -0.03665300
C 2.50997100 -1.52807000 -0.12757900
C 3.20882200 -0.31131400 -0.26681100
C 2.55127500 0.88802100 -0.31116300
C 1.12696800 0.96488000 -0.21562100
C 0.45330600 -0.29488900 -0.07728600
N -1.02085800 -0.24472500 -0.01091000
C -1.57987900 -0.75731500 1.28263400
C -1.08158700 0.05196200 2.46388100
C -1.66483100 -0.85527700 -1.22355700
C -3.10490100 -0.41531200 -1.41301700
H 0.57046900 -2.42721400 0.06502700
H 3.03894700 -2.47085900 -0.09406000
H 4.29039600 -0.32639200 -0.34052200
H 3.10111400 1.81543500 -0.41745500
C 0.41785100 2.17253200 -0.24681100
H -1.22636300 0.79176200 -0.04865600
H -1.30094700 -1.80801400 1.36368600
H -2.66449200 -0.69625000 1.20722600
H -1.55605600 -0.31528300 3.37463500
H -1.33169100 1.10737100 2.34574100
H -0.00162800 -0.03513100 2.57795600
H -1.57363100 -1.93806400 -1.13263700
H -1.05547600 -0.53704200 -2.06755300
H -3.46350900 -0.79538200 -2.37042800
H -3.17858900 0.67355900 -1.43847800
H -3.77182500 -0.79602600 -0.63956000
O -0.81022000 2.37095100 -0.15958200

TS2

Charge = 0 Multiplicity = 1

Imaginary frequency = -116.66 cm⁻¹

C 1.33763300 -1.45921000 -0.09421000
C 2.71383300 -1.32271700 -0.18897400
C 3.27131600 -0.04902400 -0.29225100
C 2.45481400 1.07547800 -0.30228900
C 1.05974400 0.99447300 -0.21296600
C 0.58230600 -0.29176700 -0.10765400

N	-0.91865500	-0.39601600	-0.03248400
C	-1.42322300	-1.08453900	1.19835600
C	-0.99933500	-0.36289600	2.46245800
C	-1.50053600	-0.94311300	-1.30310800
C	-2.98225600	-0.65369600	-1.45779900
H	0.89291100	-2.44635100	-0.01463800
H	3.34723300	-2.20173200	-0.18225100
H	4.34958300	0.05508300	-0.36469800
H	2.92511700	2.05497800	-0.38167000
C	-0.26683900	2.89069400	-0.06039700
H	-1.20846500	0.58957100	0.02666400
H	-1.04582700	-2.10646700	1.17075600
H	-2.50906500	-1.12148800	1.12553400
H	-1.42483400	-0.87727900	3.32499500
H	-1.36281900	0.66624100	2.46774700
H	0.08418500	-0.34478600	2.56873000
H	-1.28585700	-2.01172500	-1.32077900
H	-0.93280200	-0.47302700	-2.10415400
H	-3.29650400	-0.96444100	-2.45490900
H	-3.18455200	0.41540500	-1.36549600
H	-3.59935600	-1.19153000	-0.73813100
O	-1.35038800	2.49594600	0.07783600

IM2

Charge = 0 Multiplicity = 1

C	1.04388800	-0.12107600	1.16090100
C	2.42587400	-0.21691800	1.20402500
C	3.13493500	-0.30390100	0.00665700
C	2.46694200	-0.29645100	-1.21320600
C	1.06427300	-0.20009200	-1.33291700
C	0.45454000	-0.11562000	-0.09750200
N	-1.04550300	-0.02804900	-0.20259400
C	-1.64339000	1.20726500	0.38946000
C	-1.07272300	2.47083600	-0.22408900
C	-1.71018900	-1.29402300	0.24194900
C	-3.16149200	-1.40995800	-0.18687700
H	0.47109600	-0.05360000	2.08144100
H	2.94343400	-0.22445100	2.15605600
H	4.21830400	-0.37765900	0.03583400
H	3.07519600	-0.36790600	-2.11411200
H	-1.10245200	0.01994400	-1.23331700
H	-1.45842400	1.16471900	1.46331300
H	-2.71944500	1.15866400	0.23011100
H	-1.56363300	3.33545800	0.22486000

H	-1.24802600	2.50085900	-1.30098600
H	-0.00054300	2.54912300	-0.05366100
H	-1.59972300	-1.34900700	1.32515800
H	-1.11409900	-2.09281200	-0.19654500
H	-3.51646100	-2.41355700	0.05092700
H	-3.26733000	-1.27081300	-1.26500100
H	-3.81531700	-0.70299000	0.32368200

IM3

Charge = 0 Multiplicity = 1

C	2.58433100	-0.35031000	-0.00779900
C	3.50823500	0.67717100	-0.10861100
C	3.04964800	1.97684400	-0.31028100
C	1.68657100	2.23232200	-0.41074300
C	0.68934100	1.23789700	-0.31570200
C	1.23791000	-0.01569400	-0.11067300
N	0.23149400	-1.13290600	-0.02425000
C	0.29307000	-1.93285200	1.23773700
C	0.06001800	-1.07268700	2.46401000
C	0.23893600	-1.97561300	-1.26388700
C	-0.97975000	-2.87264800	-1.39449400
H	2.92423000	-1.37034100	0.14597700
H	4.56875600	0.46766500	-0.03243100
H	3.76650000	2.78910600	-0.38976900
H	1.38934200	3.26831000	-0.57211900
H	-0.66138200	-0.61729100	-0.01860200
H	1.26682600	-2.42188700	1.26361100
H	-0.47306400	-2.70302600	1.16815500
H	0.09017700	-1.70415600	3.35326600
H	-0.92048900	-0.59777200	2.41197700
H	0.81915900	-0.29800900	2.56035000
H	1.17034900	-2.54265800	-1.26323100
H	0.28228400	-1.26360600	-2.08669200
H	-0.99803500	-3.29456400	-2.40037800
H	-1.89700900	-2.30022300	-1.24681100
H	-0.97005200	-3.70551500	-0.69112400
O	-2.57930600	-0.20431200	0.24811700
C	-2.72099900	0.98297600	0.00994100
C	-2.74925700	1.49505700	-1.39677400
H	-3.05010300	0.70676200	-2.08532600
H	-3.38157700	2.37516500	-1.51365100
H	-1.70747500	1.77264300	-1.60362700
C	-2.74283600	1.99523200	1.11534800

H	-1.73322100	2.42015000	1.14253700
H	-3.44989600	2.80315800	0.92495300
H	-2.95262400	1.51475900	2.06930300

TS3

Charge = 0 Multiplicity = 1

Imaginary frequency = -72.53 cm⁻¹

C	-2.48411500	0.30274600	-0.08439600
C	-3.34090800	-0.78042400	-0.19729100
C	-2.79981400	-2.05547100	-0.34498300
C	-1.42102300	-2.23192500	-0.38143700
C	-0.49224100	-1.17708200	-0.27400500
C	-1.11707800	0.04713900	-0.12227100
N	-0.17815900	1.22200000	-0.03223200
C	-0.35998900	2.07787400	1.17988100
C	-0.12495600	1.29629700	2.45791600
C	-0.15176300	2.00050600	-1.31204400
C	1.06074000	2.90634100	-1.43699700
H	-2.88950900	1.30379500	0.02801400
H	-4.41415400	-0.63260400	-0.17117500
H	-3.46453100	-2.91011300	-0.43276300
H	-1.05651300	-3.25269000	-0.50145100
H	0.74594700	0.76663900	0.05454300
H	-1.36521200	2.49682200	1.13783600
H	0.35178400	2.89851800	1.10463400
H	-0.22592700	1.96677900	3.31256500
H	0.88189200	0.87640400	2.46408400
H	-0.84190900	0.48353000	2.56626100
H	-1.09039600	2.55209900	-1.37782000
H	-0.14974800	1.24879800	-2.09966900
H	1.10400300	3.29901800	-2.45385400
H	1.97813600	2.34556600	-1.24906900
H	1.02592800	3.75892500	-0.75847300
O	2.42532000	0.11605700	0.32234500
C	2.36393800	-1.08243200	0.07474700
C	2.63136900	-1.60856300	-1.30819000
H	2.56446700	-0.80329200	-2.03690500
H	3.64669300	-2.02190500	-1.32823500
H	1.92950800	-2.39974600	-1.56134600
C	2.28068600	-2.09788000	1.18020200
H	1.71061000	-2.97209300	0.87349300
H	3.30186000	-2.41556200	1.42325300
H	1.83254600	-1.65409700	2.06614800

IM4

Charge = 0 Multiplicity = 1

C	0.01379500	2.15431000	-0.35261100
C	-1.19371200	2.81243100	-0.50319000
C	-2.37021200	2.08245600	-0.43925400
C	-2.32047200	0.71529700	-0.22404100
C	-1.11487900	0.02863600	-0.06107400
C	0.06882500	0.77863400	-0.13620400
N	1.36241800	0.13398400	-0.01315800
C	2.19573000	0.68394000	1.06887500
C	1.53205500	0.59490800	2.43421800
C	2.07608300	0.08106900	-1.30097100
C	3.11351100	-1.03076500	-1.36310800
H	0.93880800	2.71595400	-0.40648300
H	-1.21491200	3.88245400	-0.67105000
H	-3.32843200	2.57476300	-0.55464700
H	-3.25201600	0.16747800	-0.17814500
H	0.73170800	-1.40858400	0.58114500
H	2.47773800	1.72547800	0.85371000
H	3.12201400	0.10832300	1.08303500
H	2.22757100	0.93394400	3.20428400
H	1.23379100	-0.42969000	2.65770300
H	0.64024700	1.22104100	2.48632100
H	2.54708100	1.05132900	-1.51962200
H	1.32879100	-0.08316800	-2.07830400
H	3.55688700	-1.06678800	-2.36038700
H	2.65230400	-1.99882200	-1.15820600
H	3.92568900	-0.88237700	-0.65022300
O	-0.02558500	-1.95464100	0.87920200
C	-1.13702200	-1.50059100	0.12061000
C	-1.11342000	-2.16482900	-1.26409300
H	-0.20190700	-1.89787600	-1.80217900
H	-1.13219400	-3.24974100	-1.14259900
H	-1.96953400	-1.86091300	-1.87079400
C	-2.36023100	-1.97822300	0.90051800
H	-3.28906000	-1.87132900	0.33975500
H	-2.21870600	-3.03668600	1.12244700
H	-2.44842200	-1.43917900	1.84468200

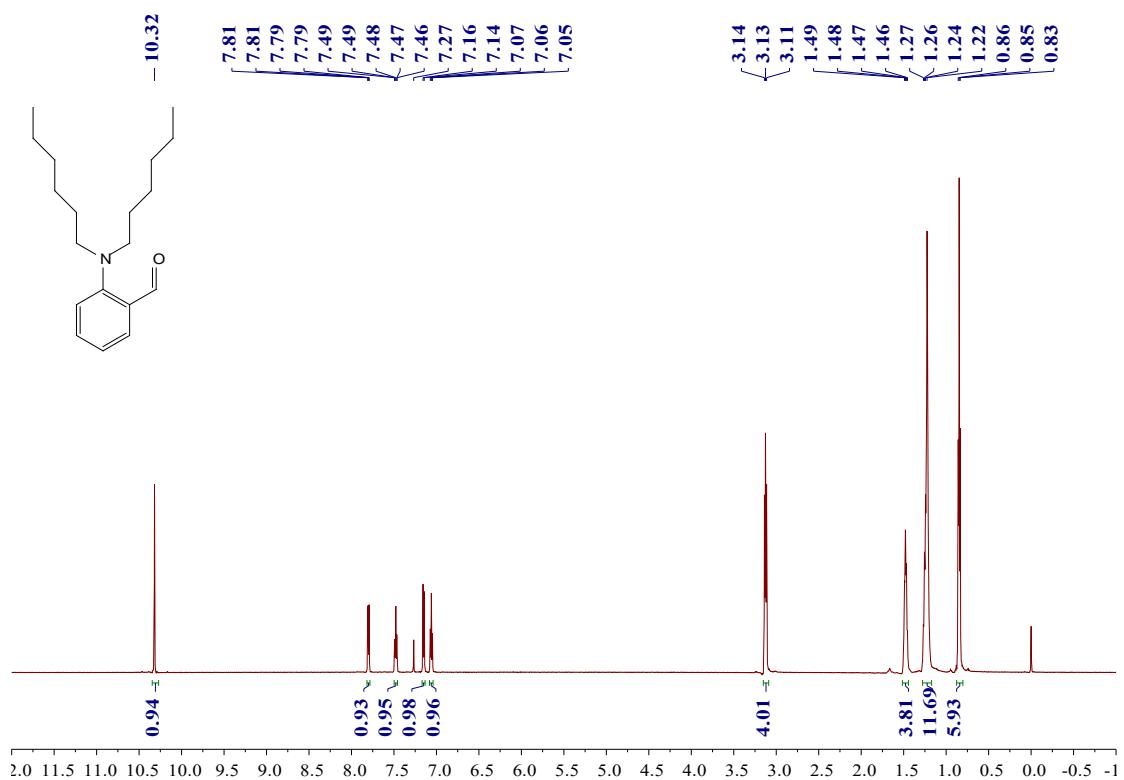
11. Notes and references

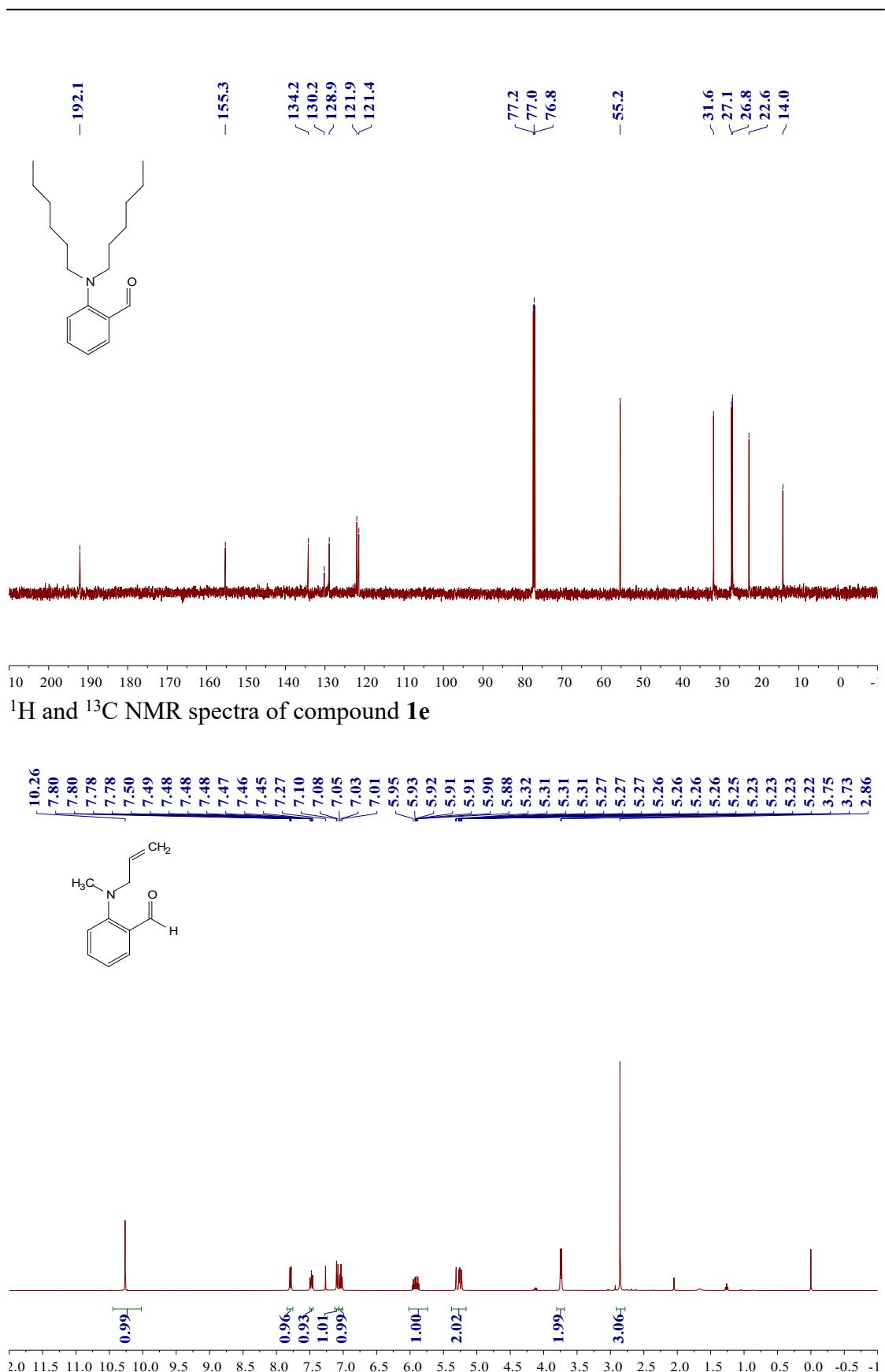
- [1] L. Josa-Culleré, M. G. Hirst, J. P. Lockett, A. L. Thompson, M. G. Moloney, *J. Org. Chem.* **2019**,

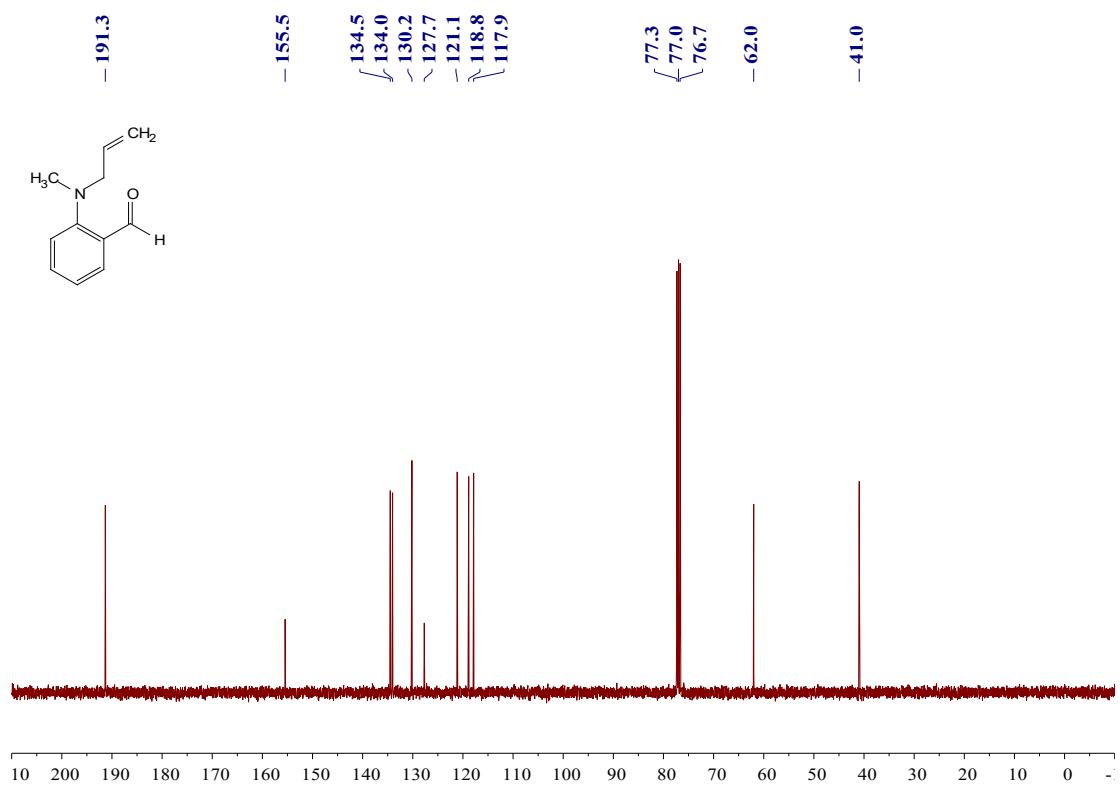
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12. Copies of ^1H , ^{13}C and ^{19}F NMR spectra.

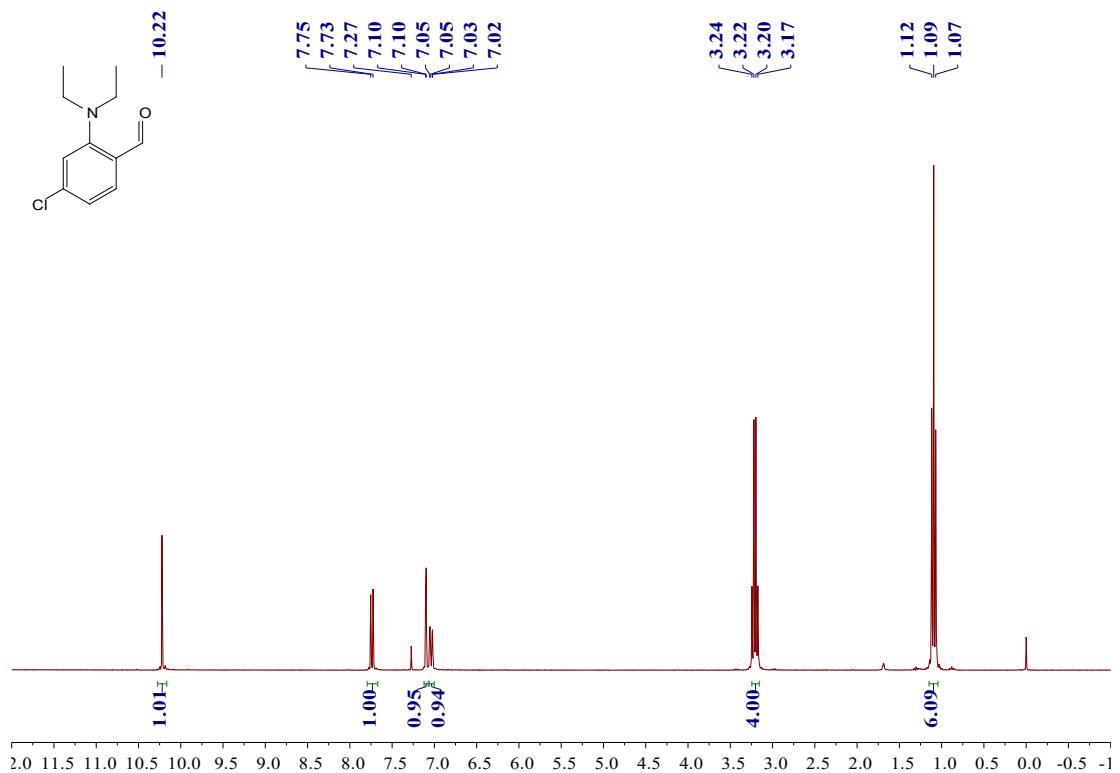
^1H and ^{13}C NMR spectra of compound **1c**

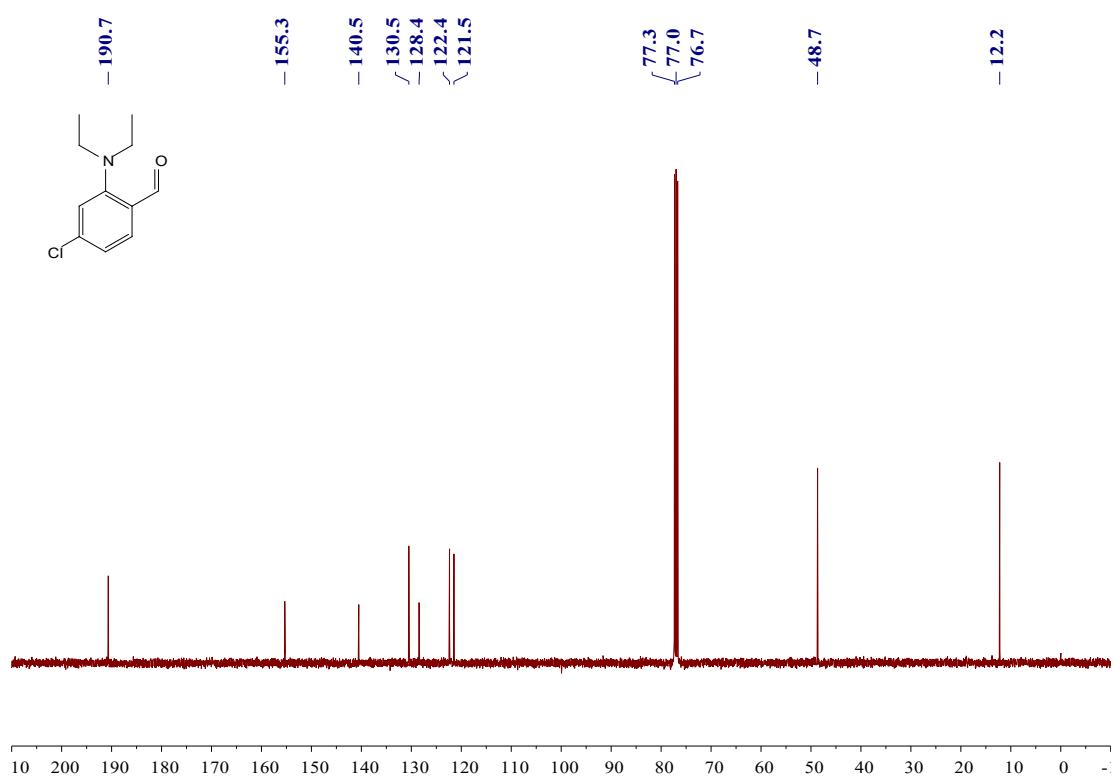




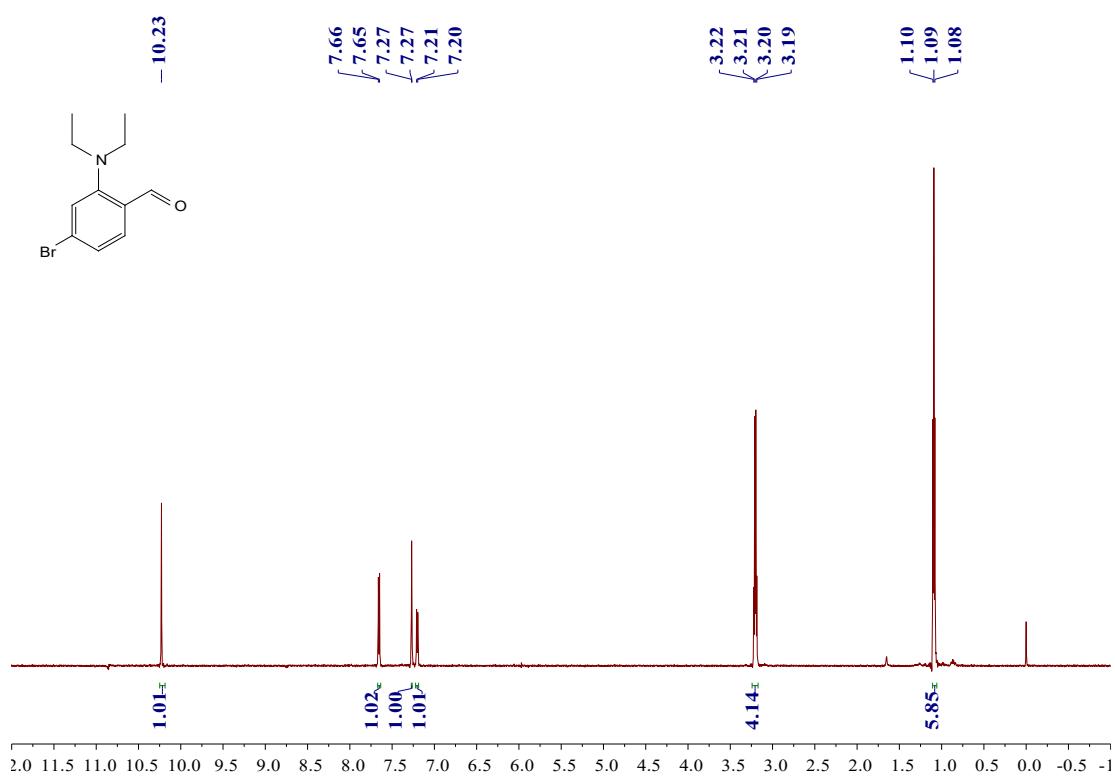


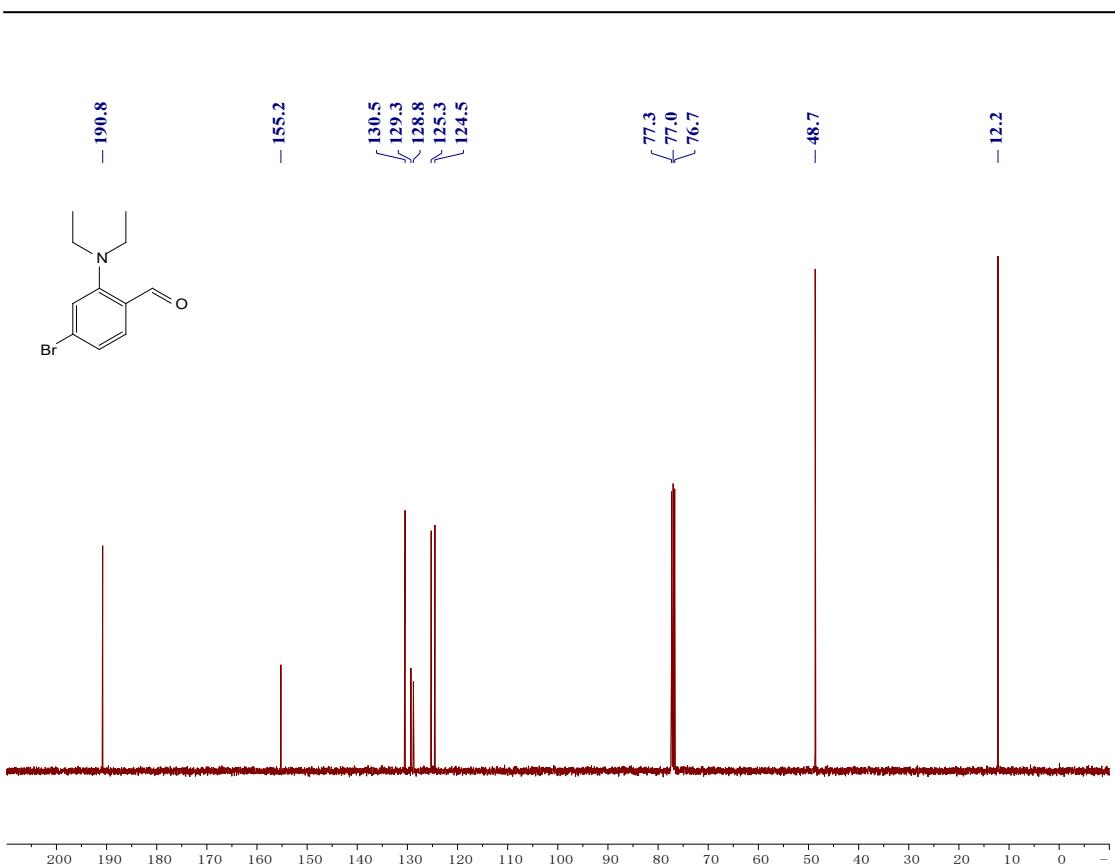
¹H and ¹³C NMR spectra of compound **1g**



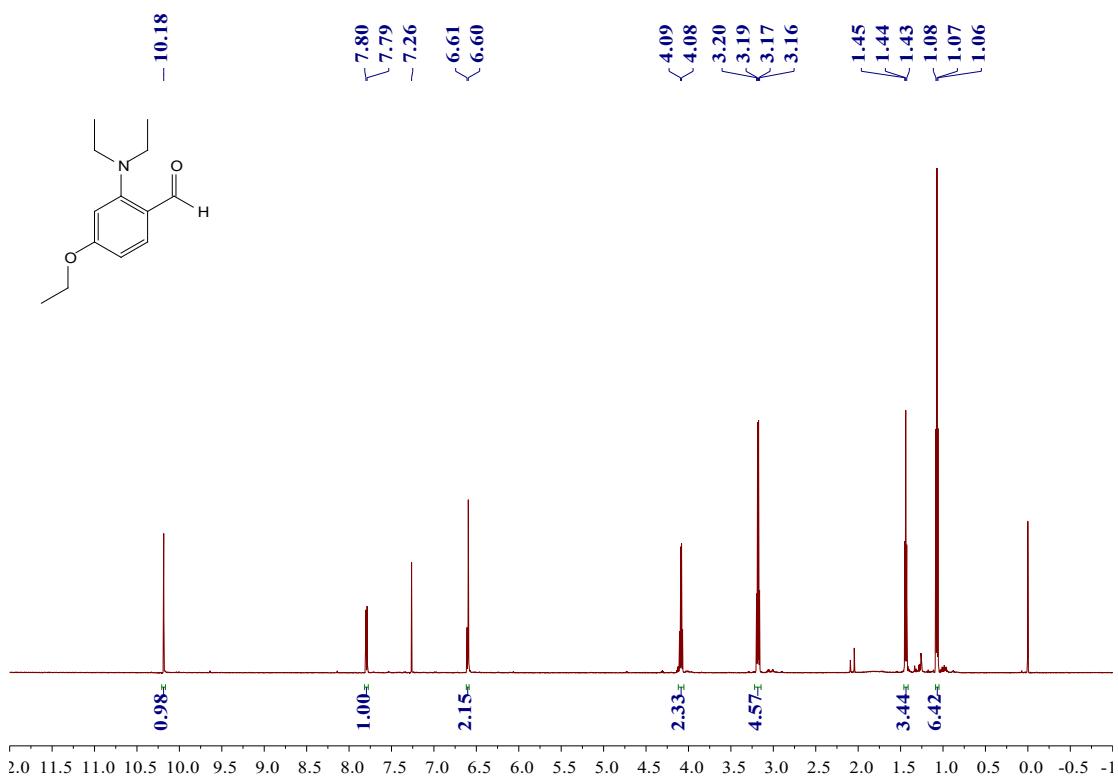


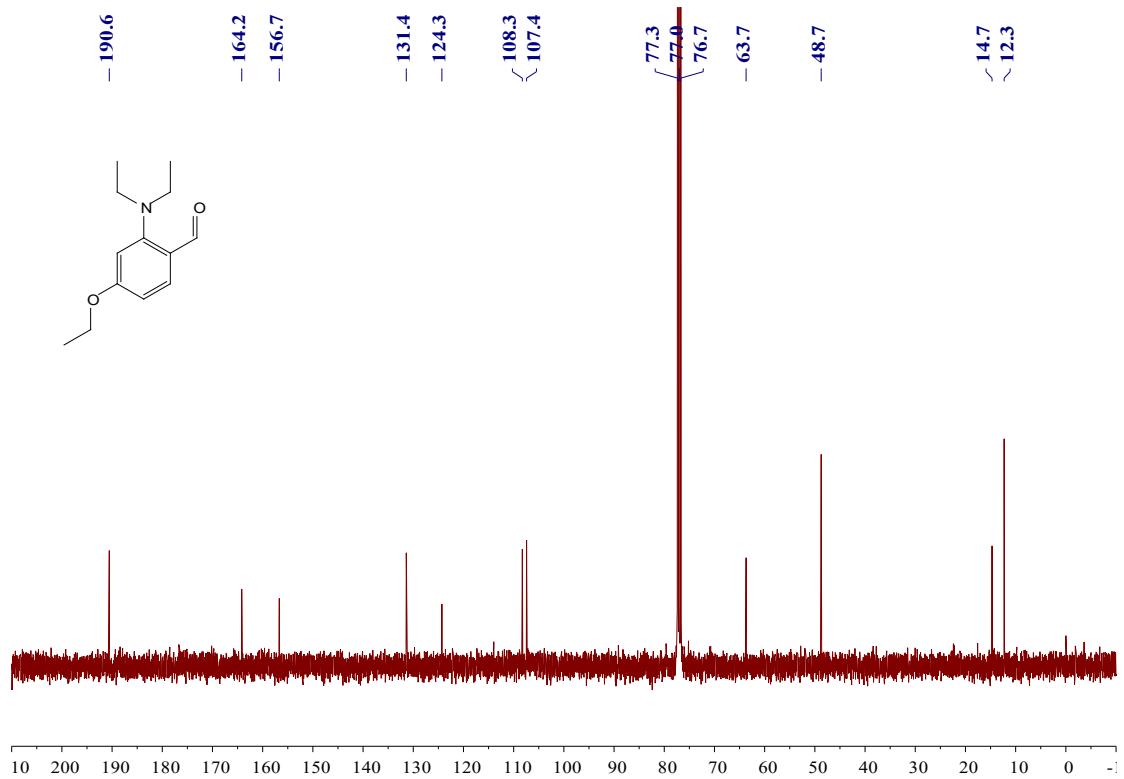
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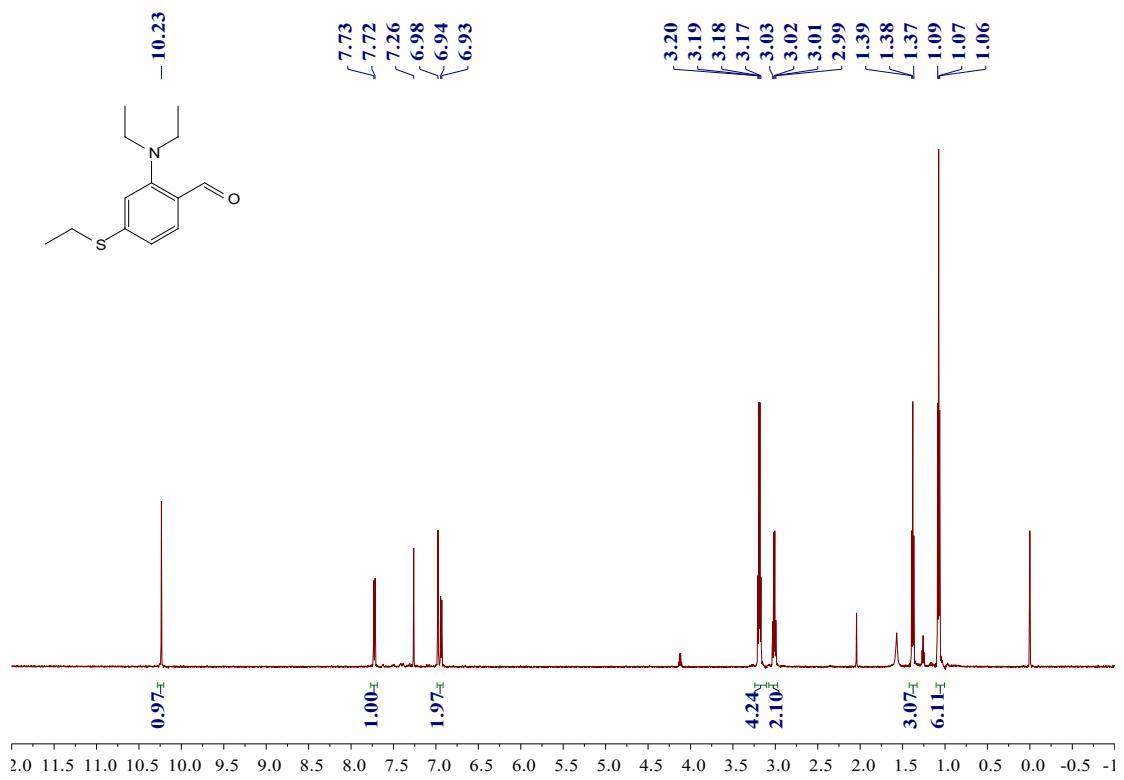


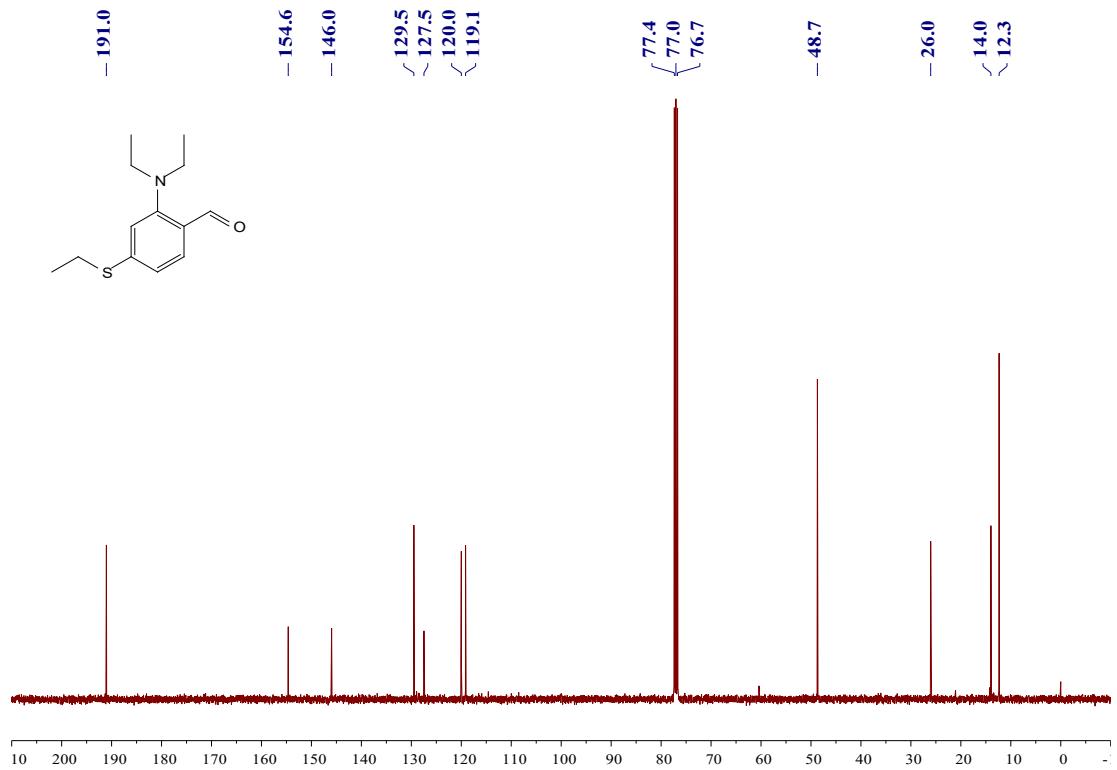
¹H and ¹³C NMR spectra of compound **1i**



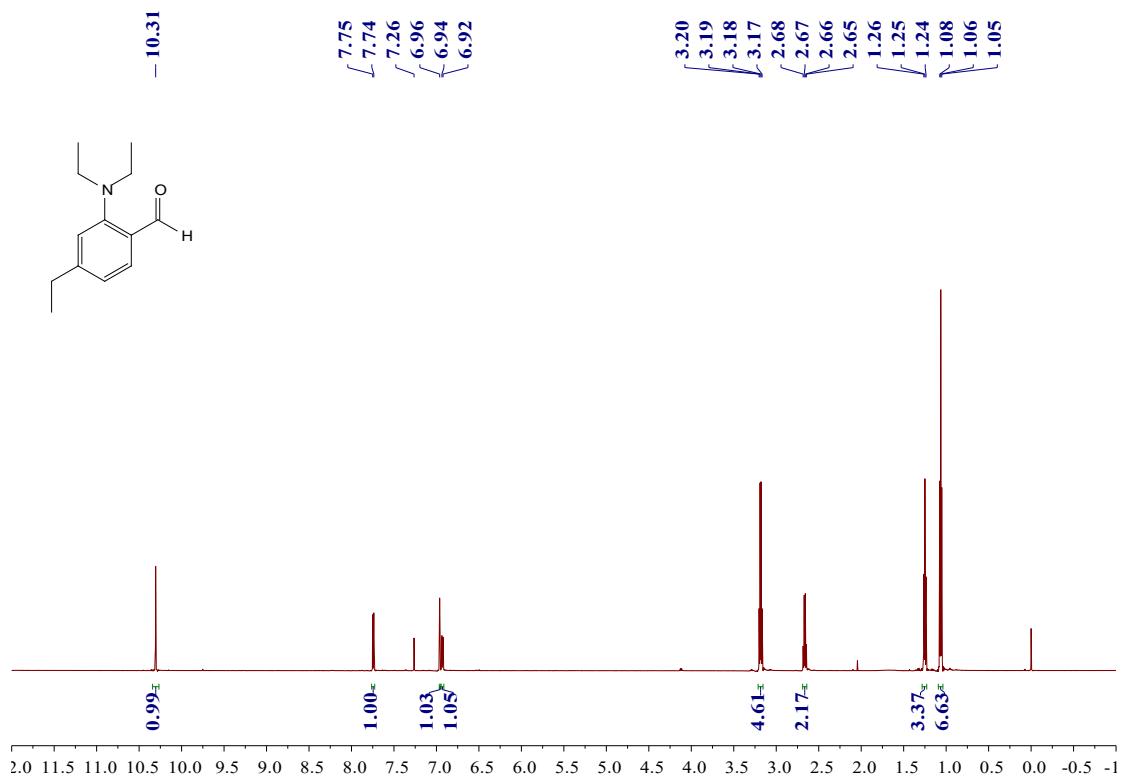


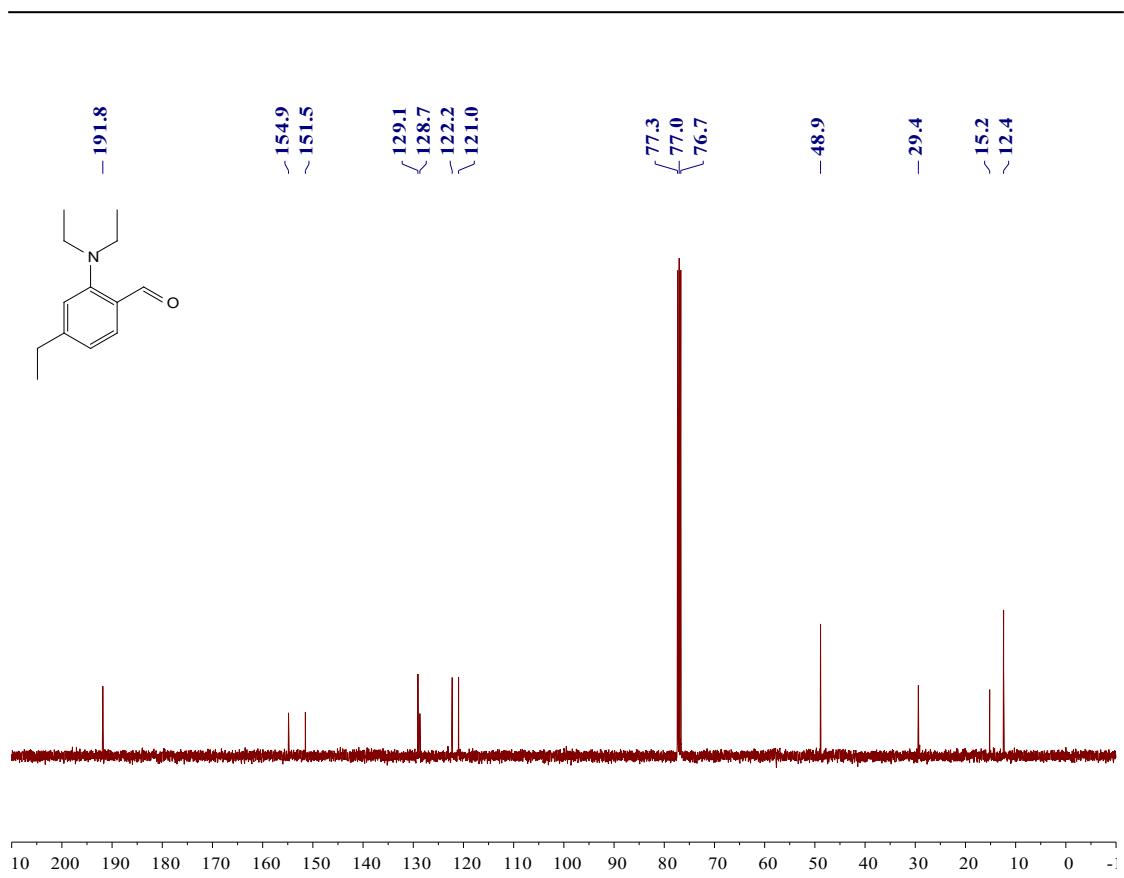
¹H and ¹³C NMR spectra of compound **1j**



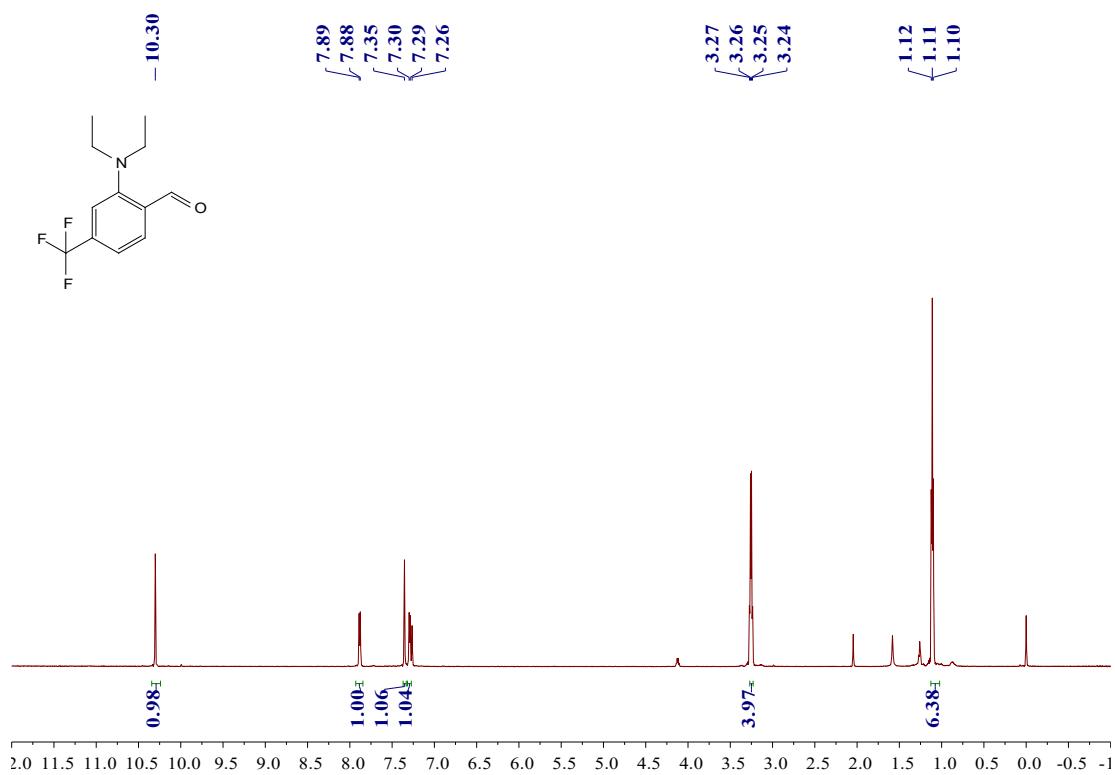


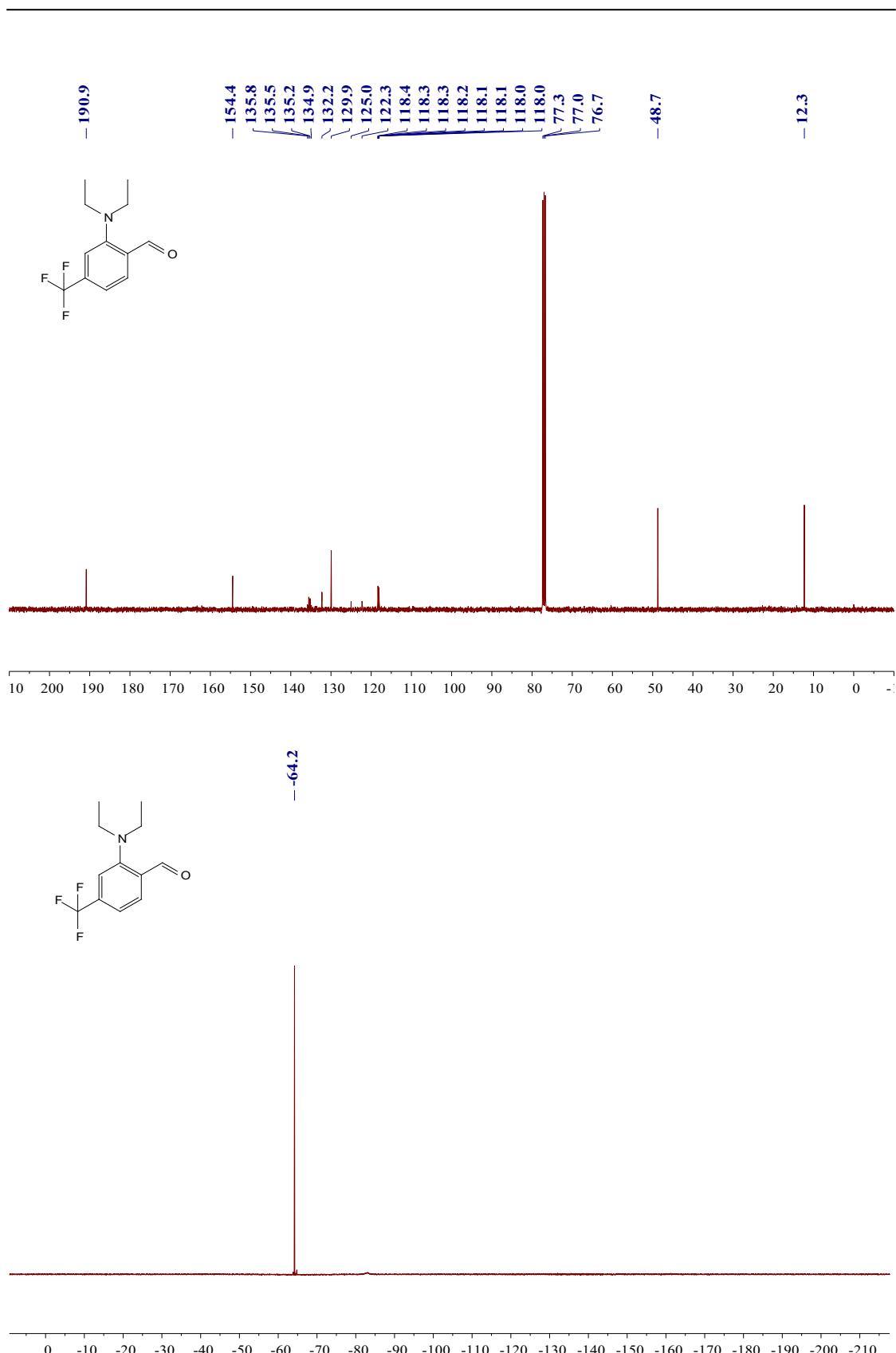
¹H and ¹³C NMR spectra of compound **1k**



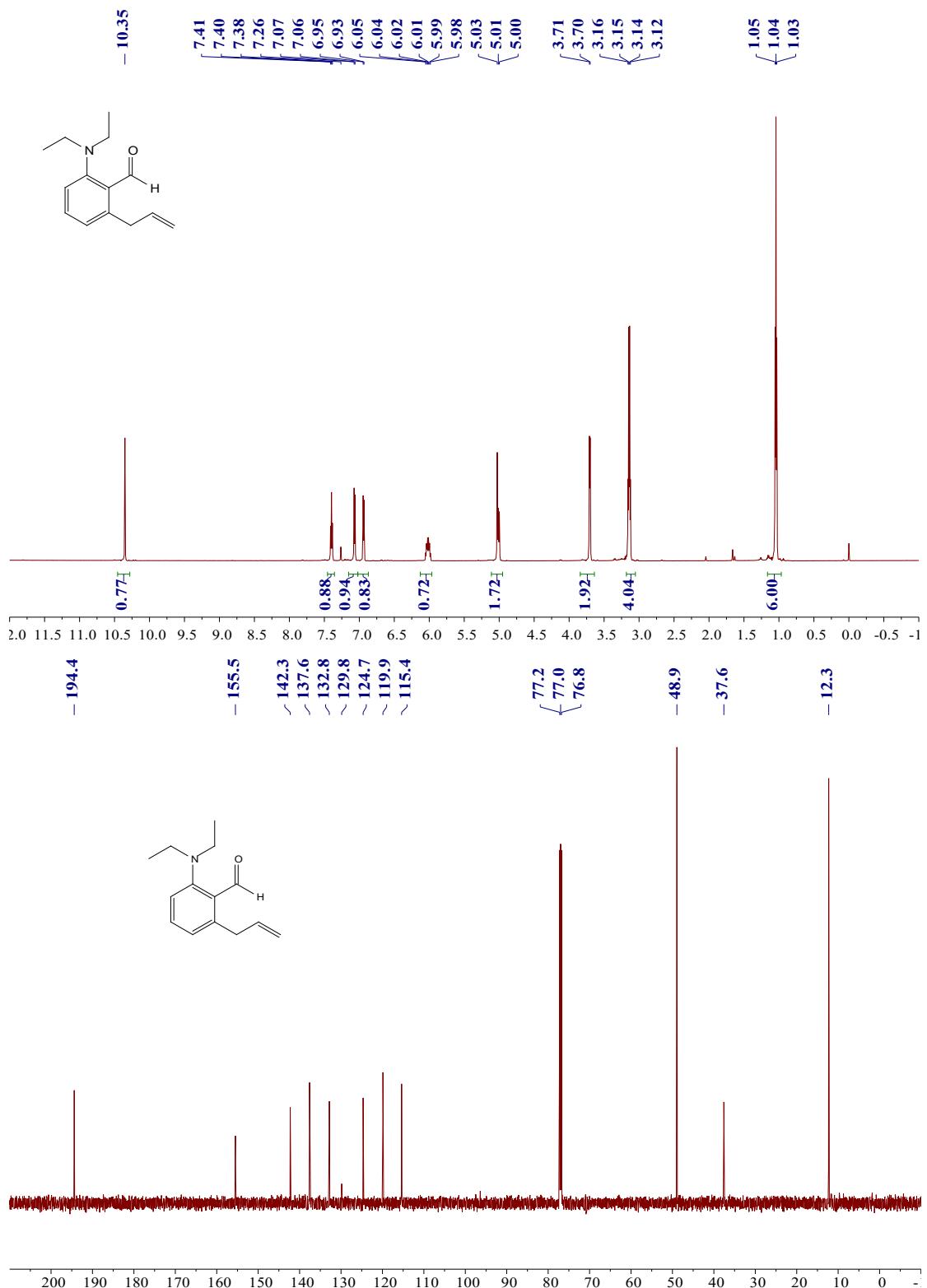


¹H, ¹³C and ¹⁹F NMR spectra of compound **1l**

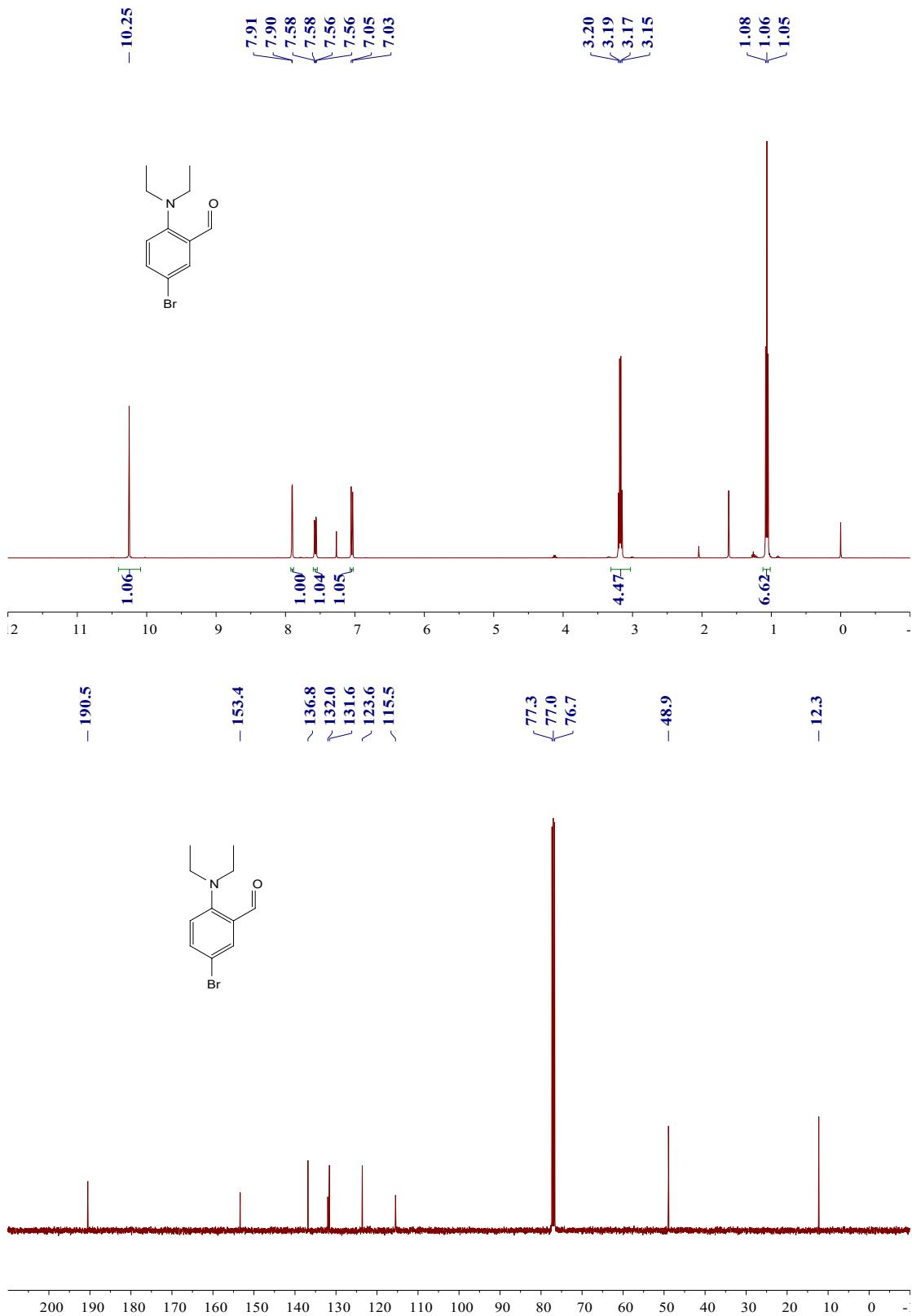




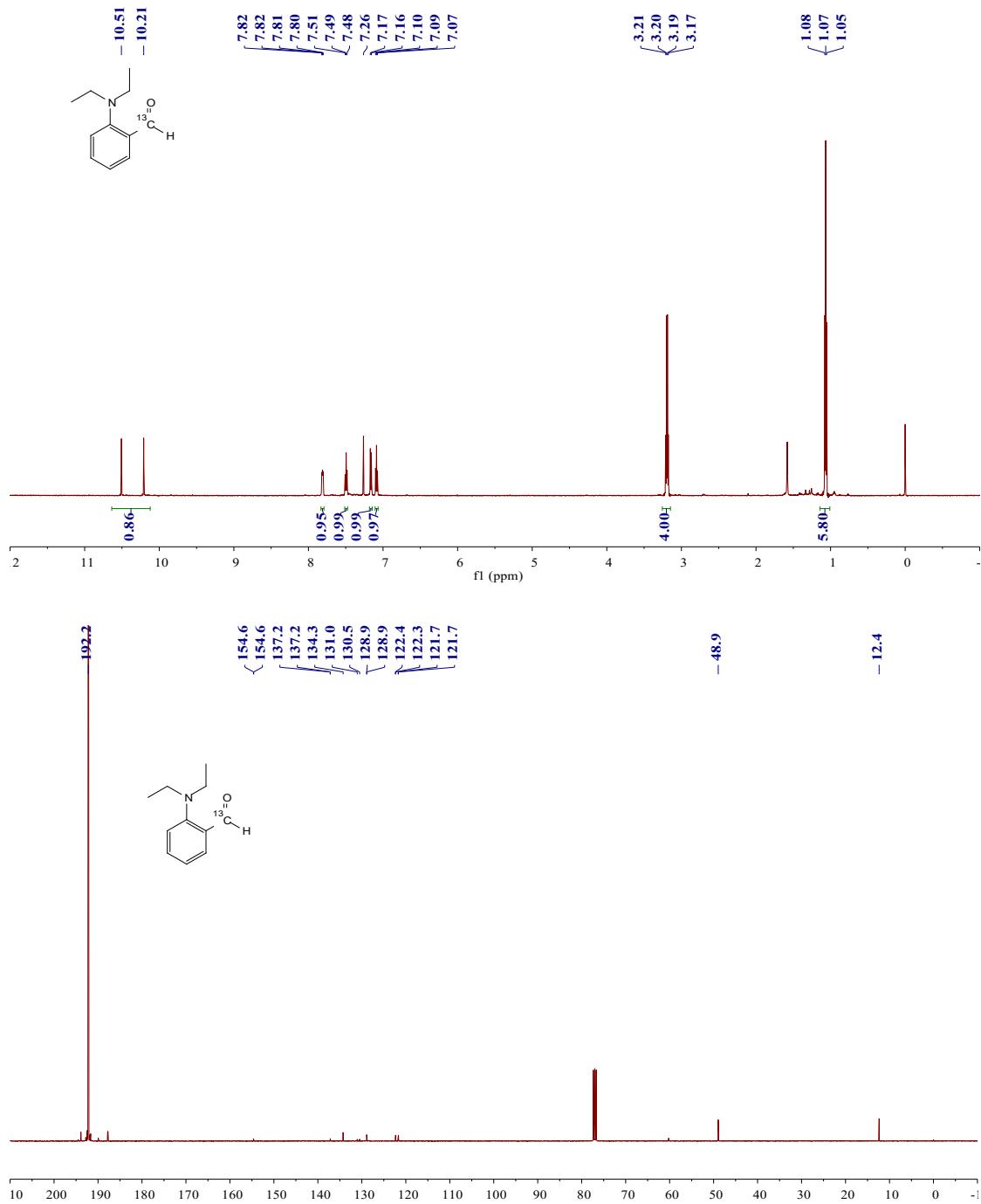
¹H and ¹³C NMR spectra of compound **1m**



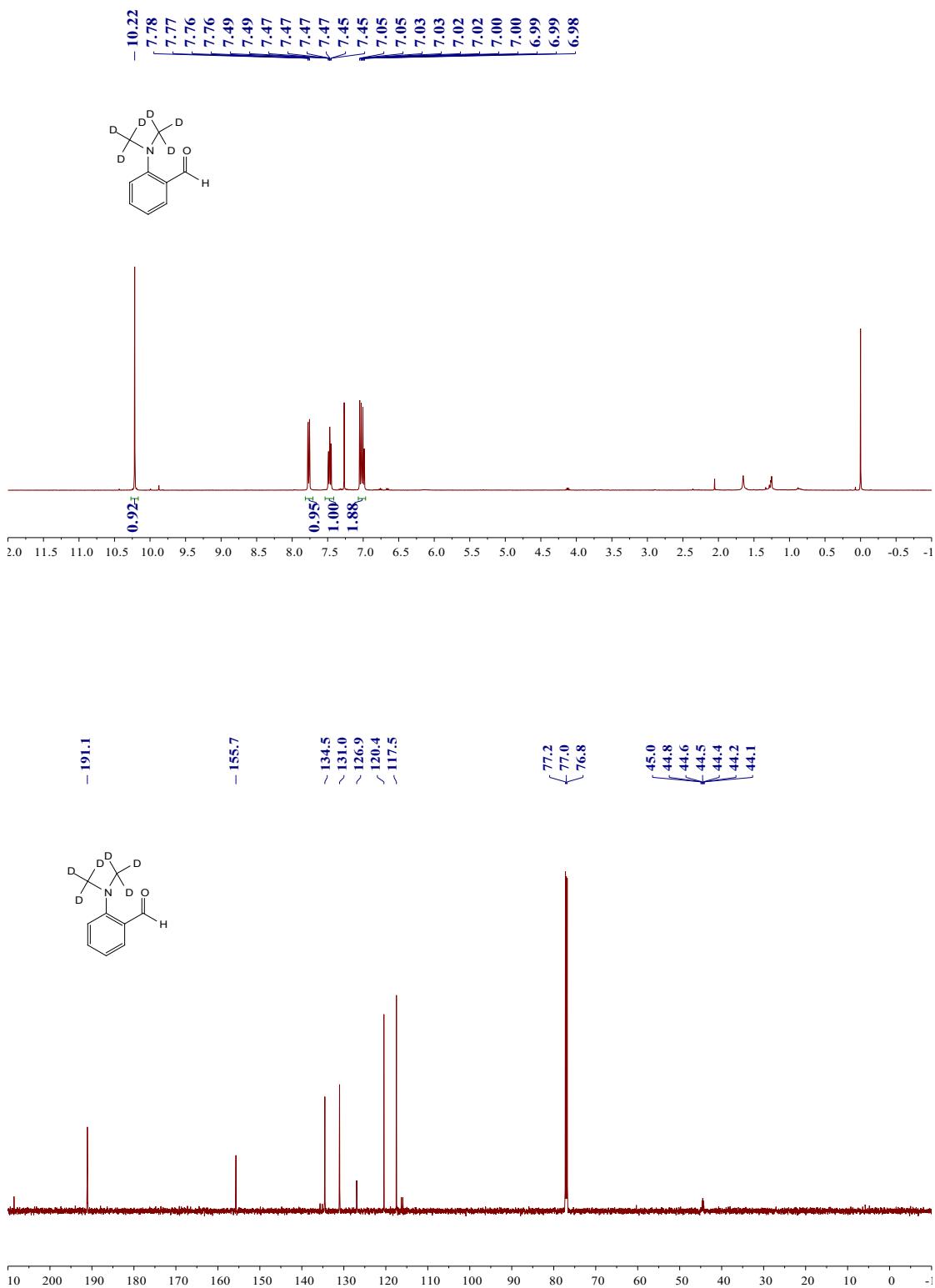
¹H and ¹³C NMR spectra of compound **1n**



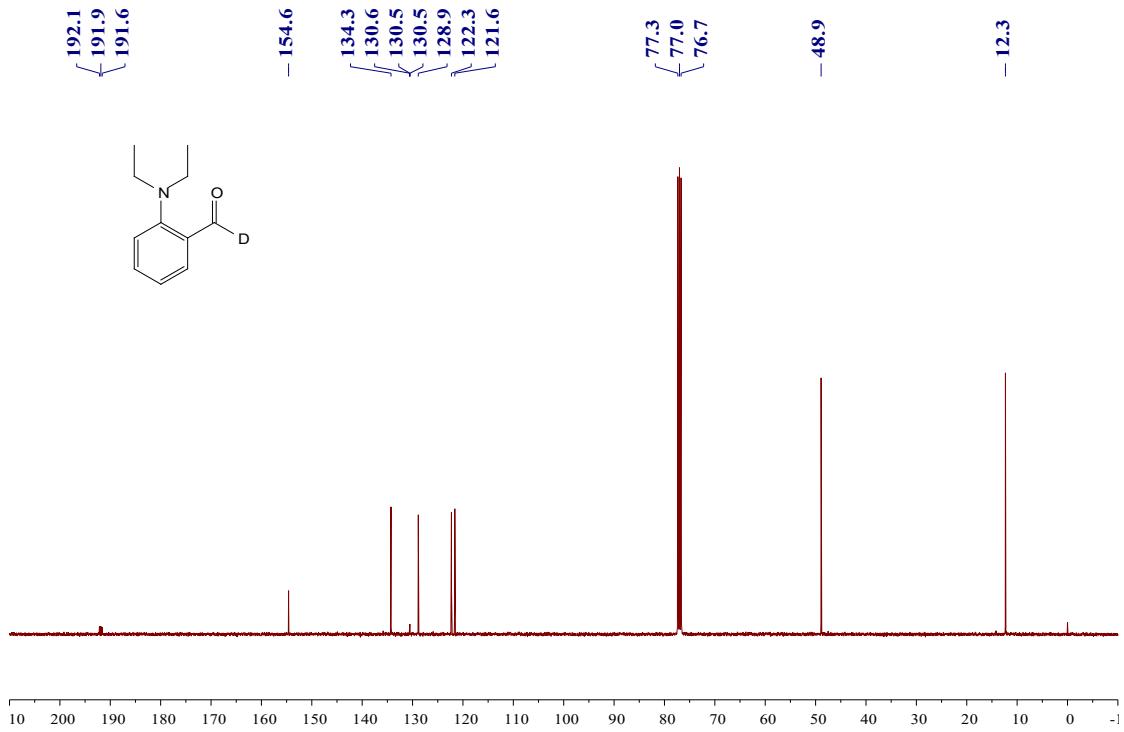
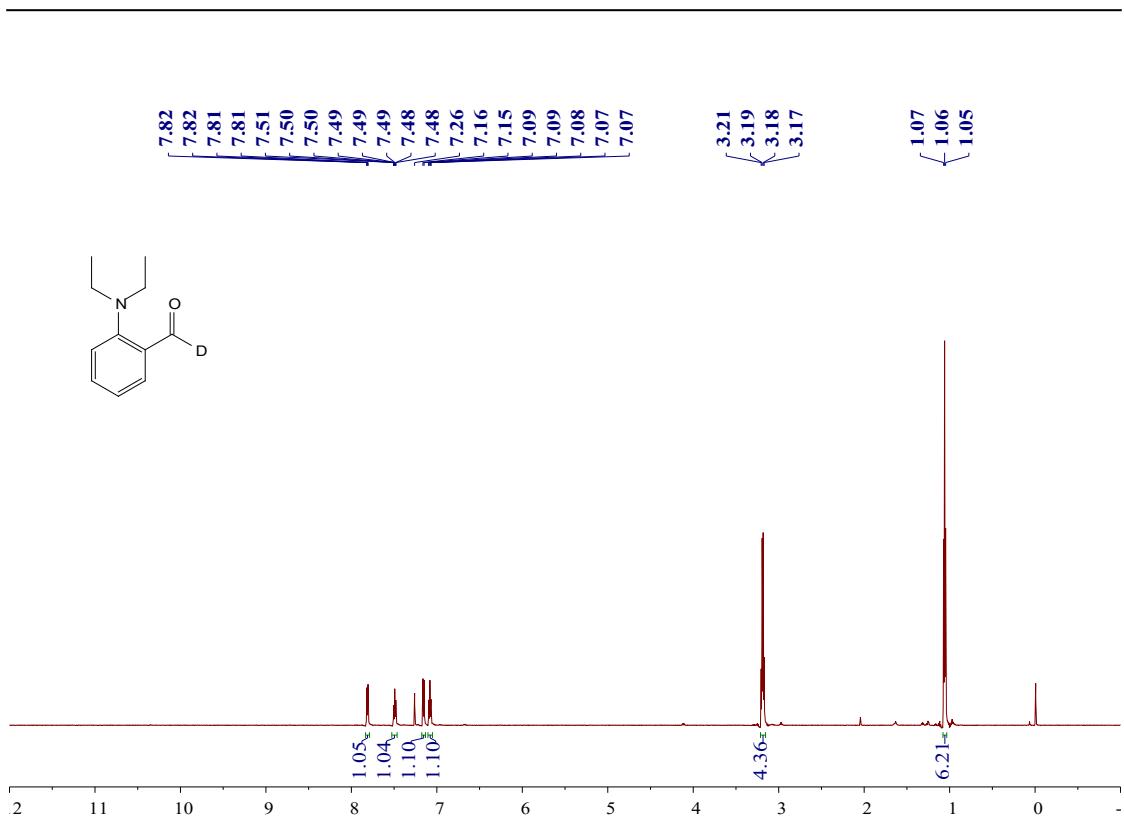
¹H and ¹³C NMR spectra of compound **1a'**



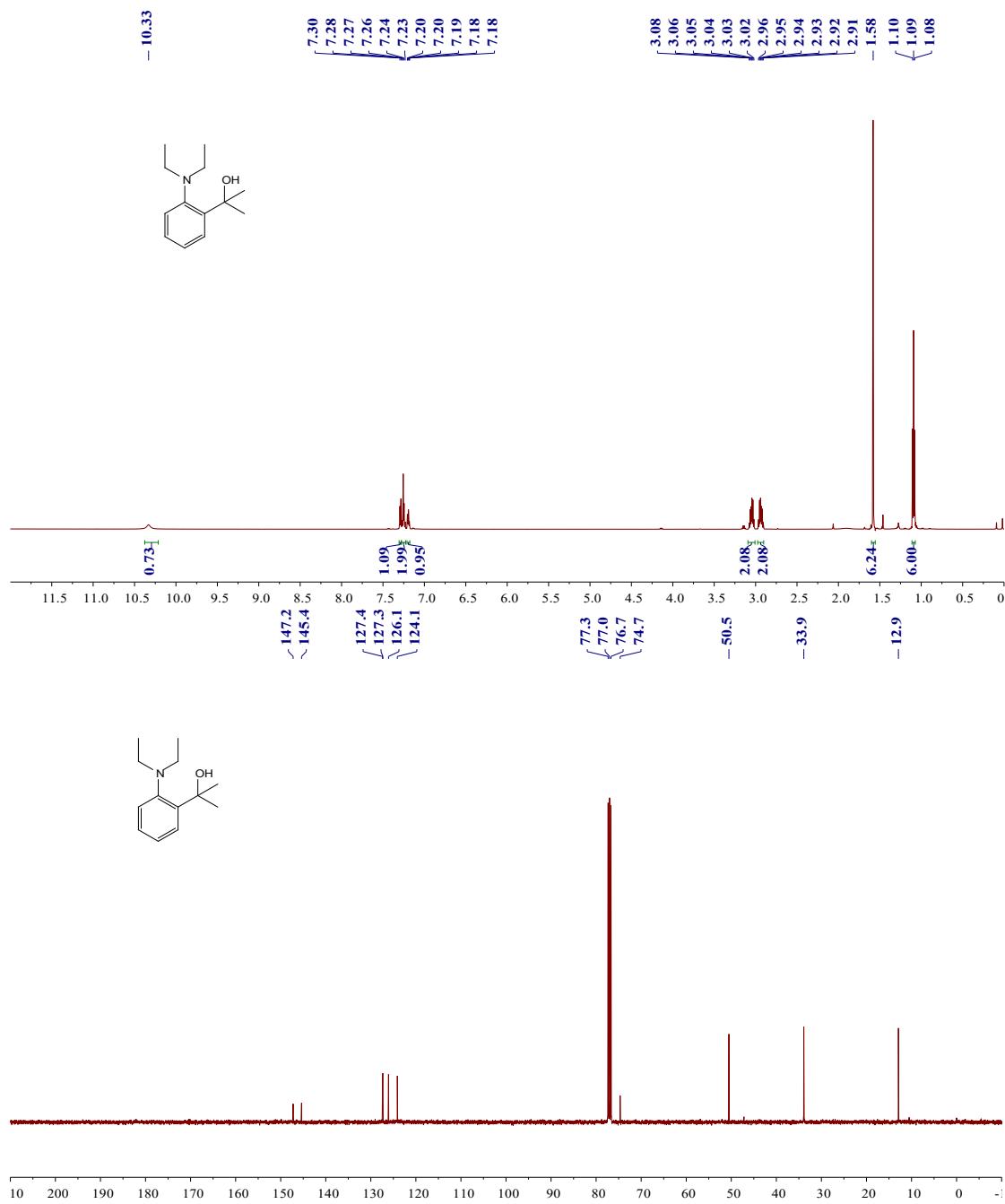
¹H and ¹³C NMR spectra of compound **1b'**



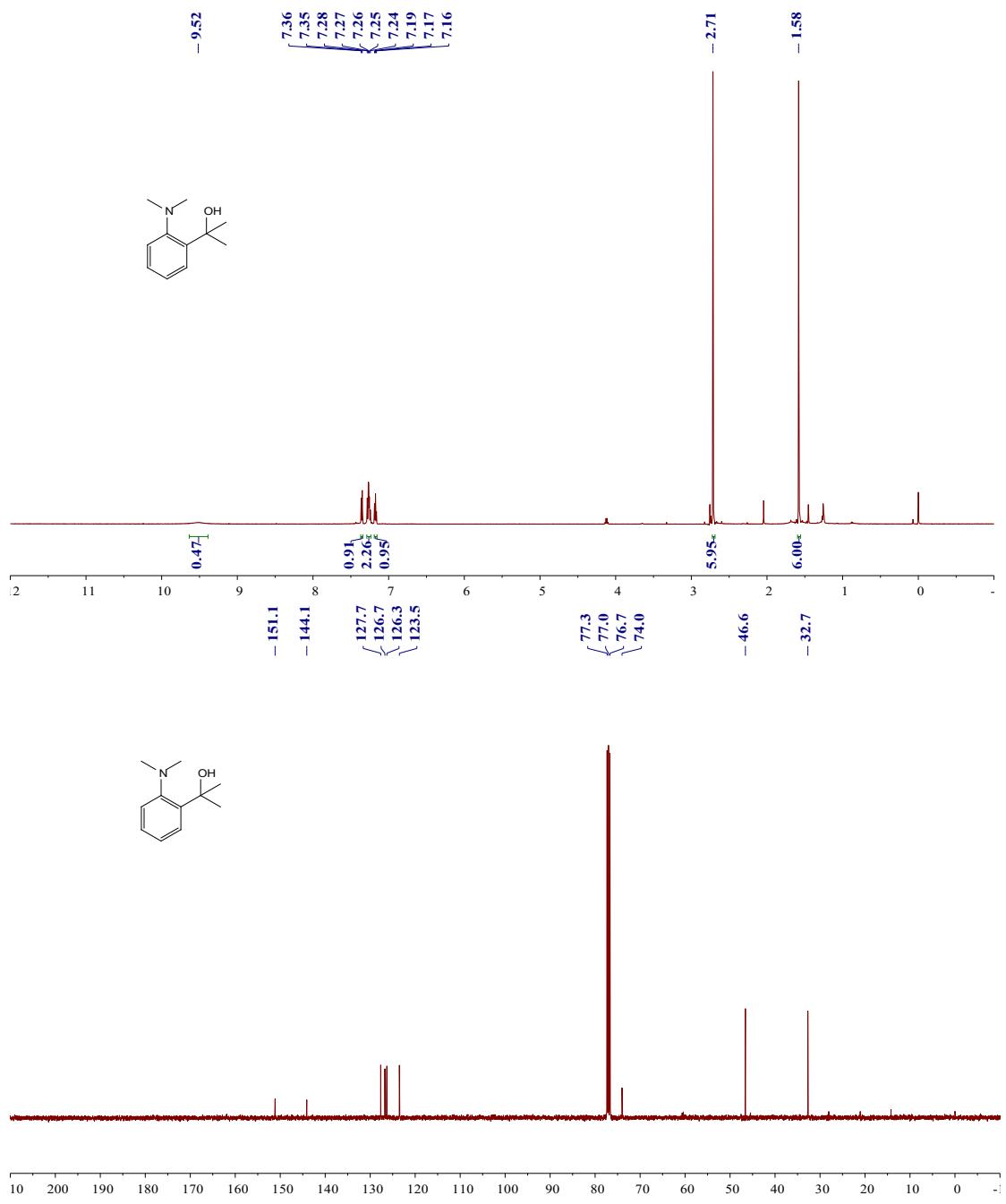
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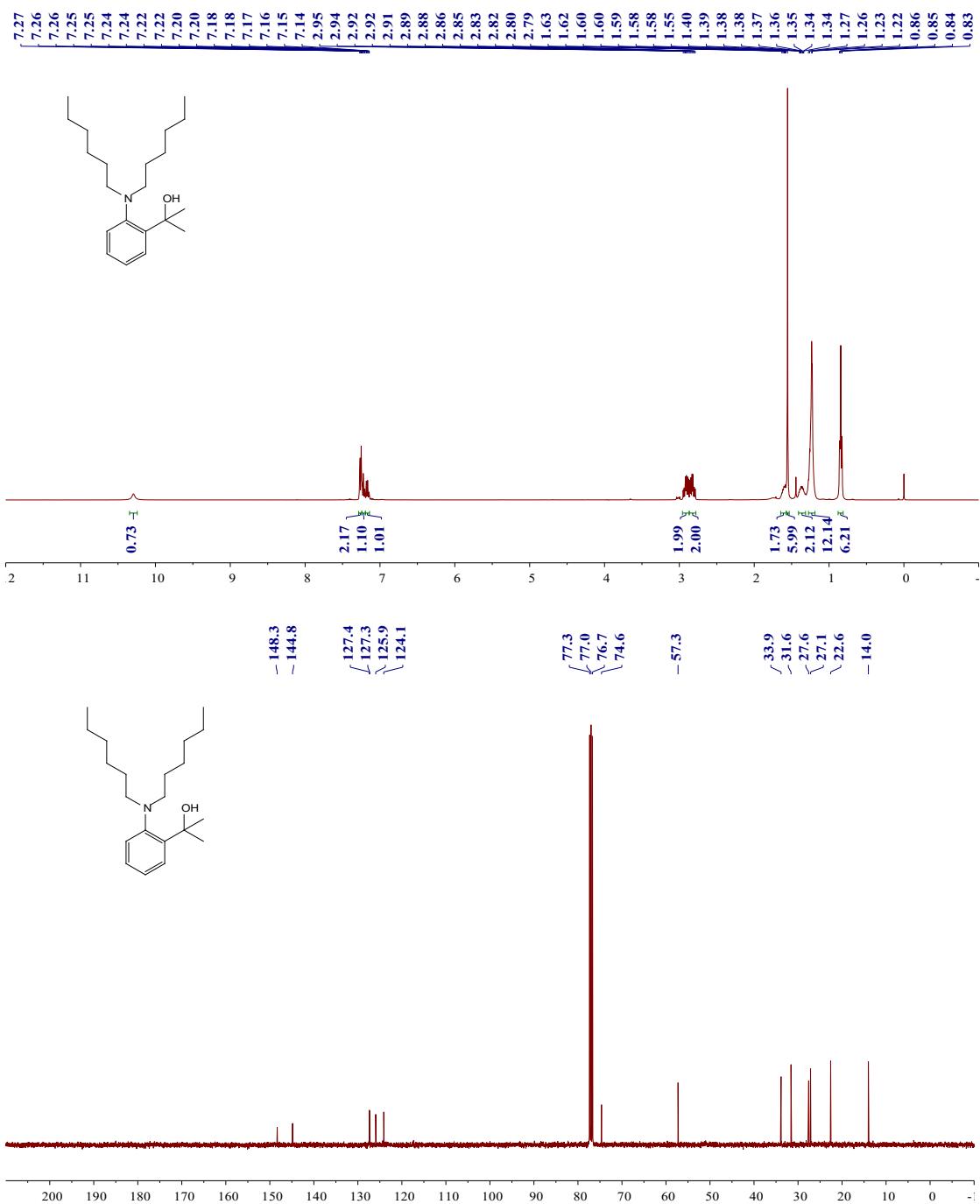
¹H and ¹³C NMR spectra of compound **3aa**



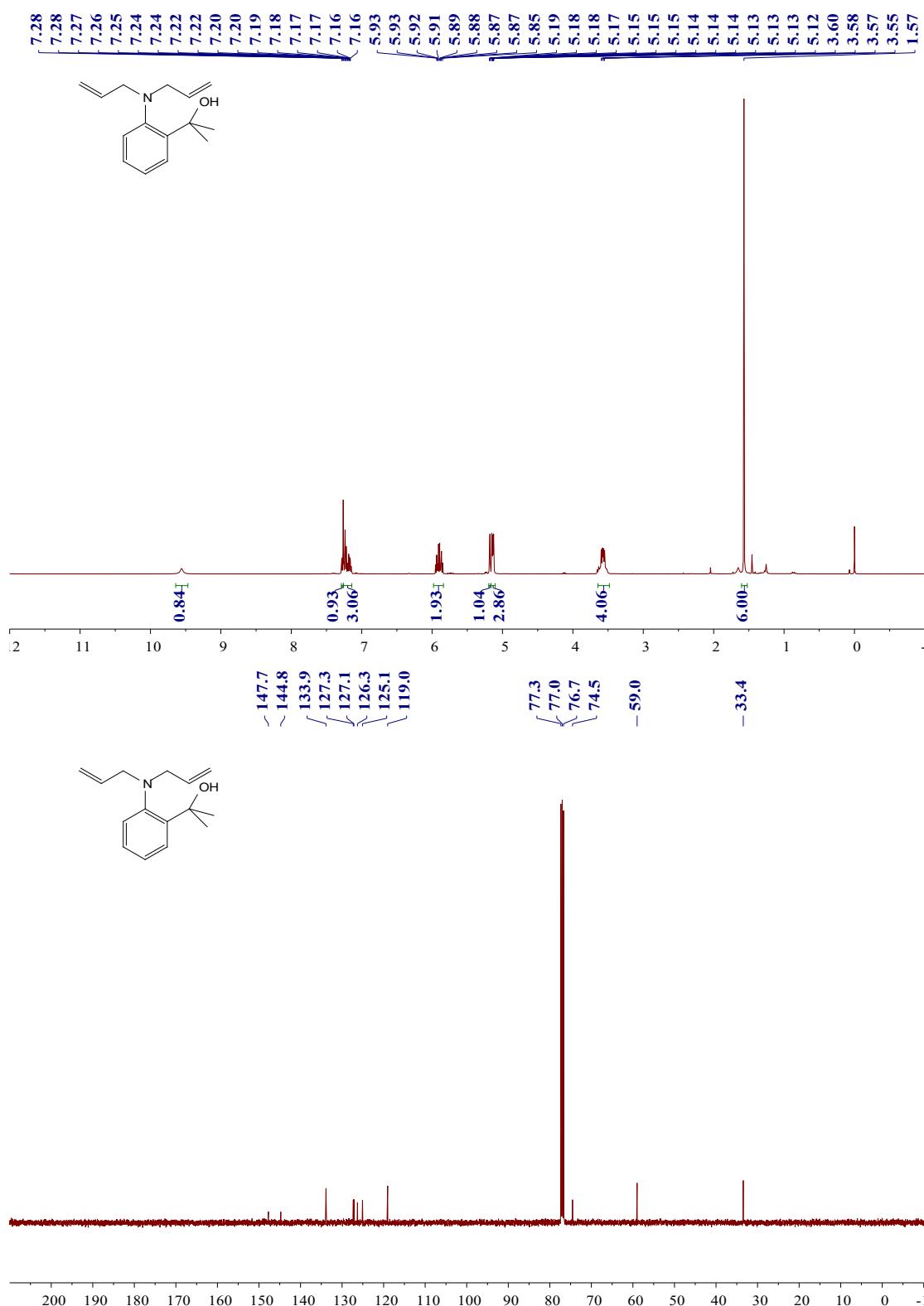
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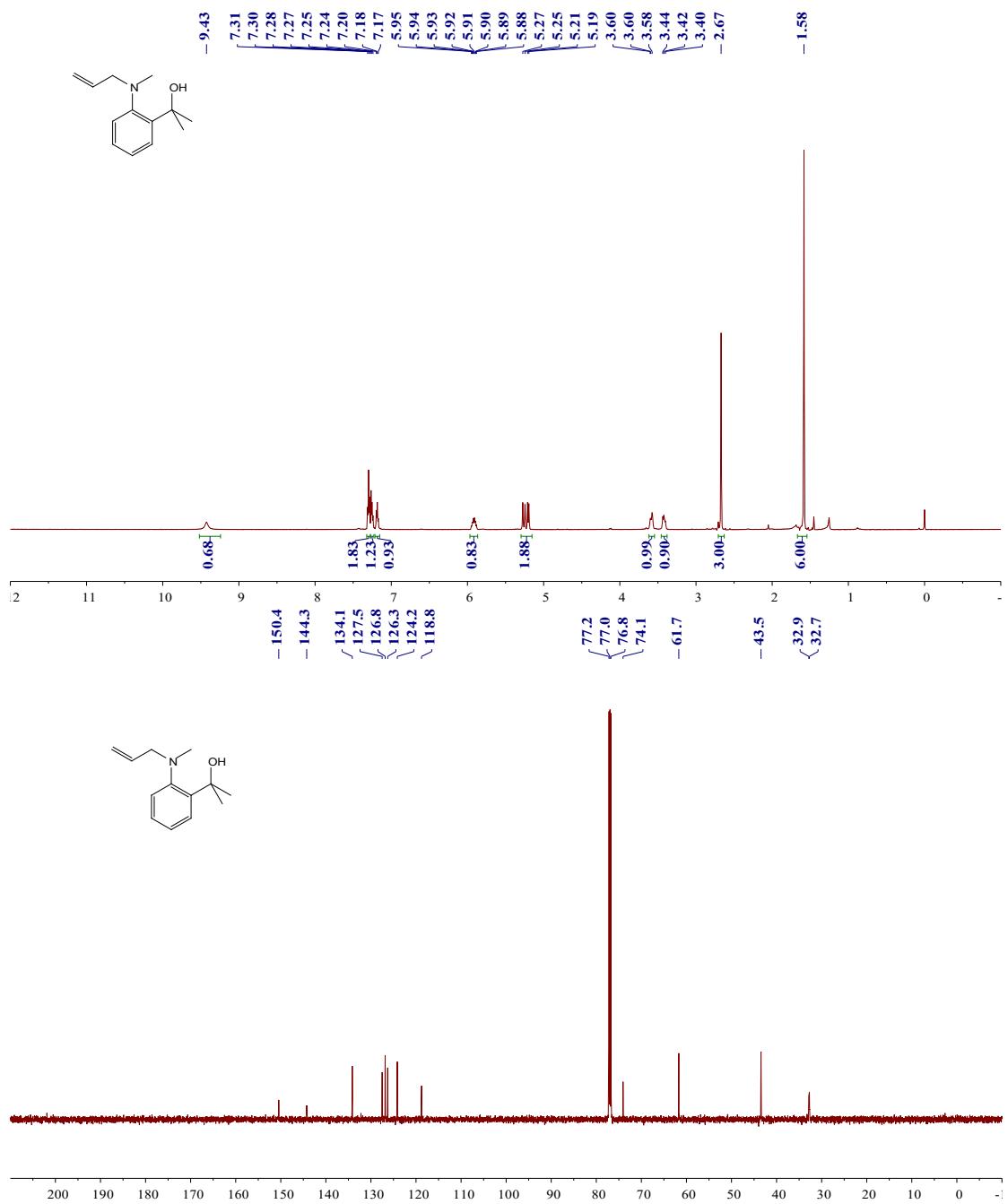
¹H and ¹³C NMR spectra of compound 3ca



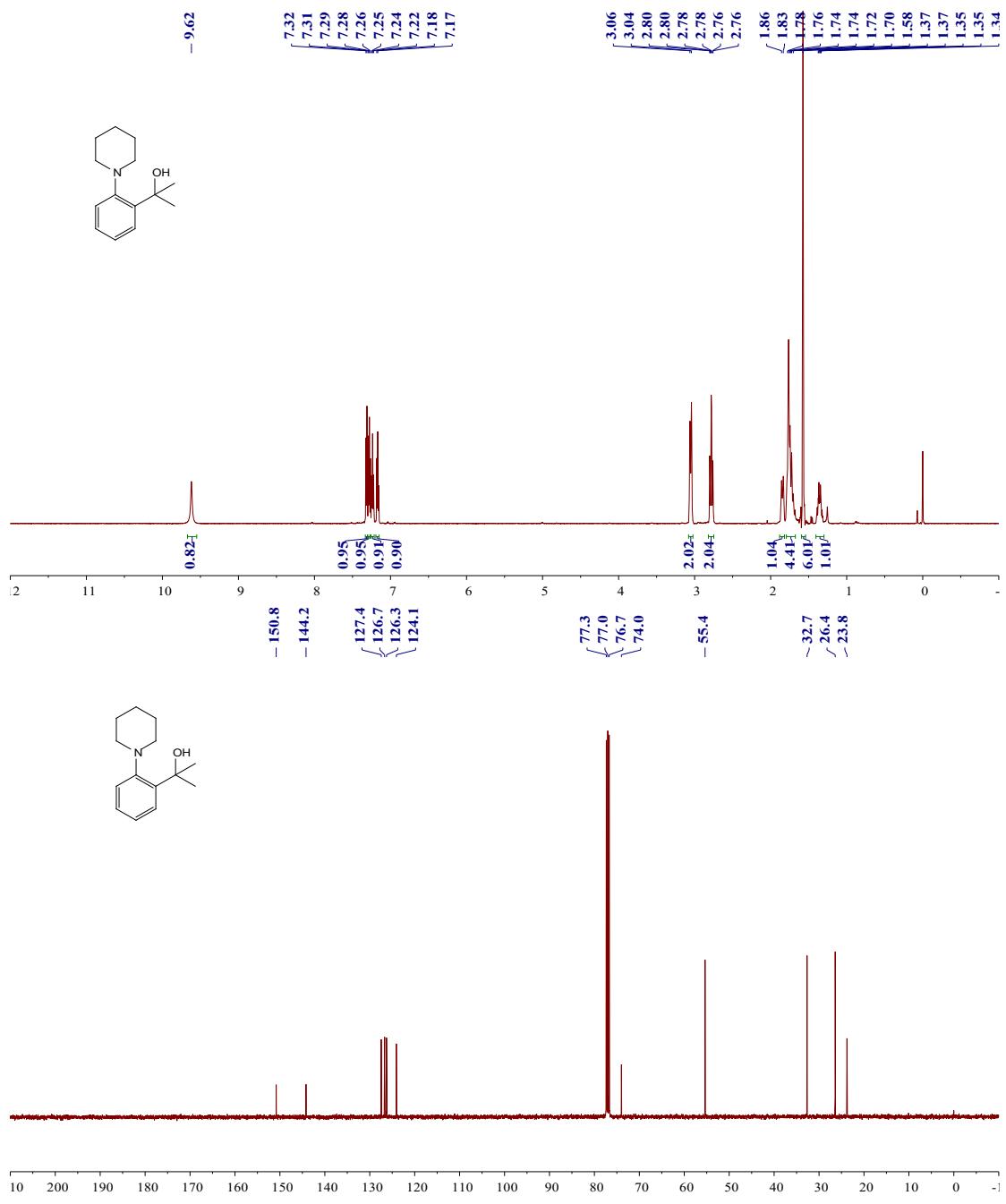
¹H and ¹³C NMR spectra of compound **3da**



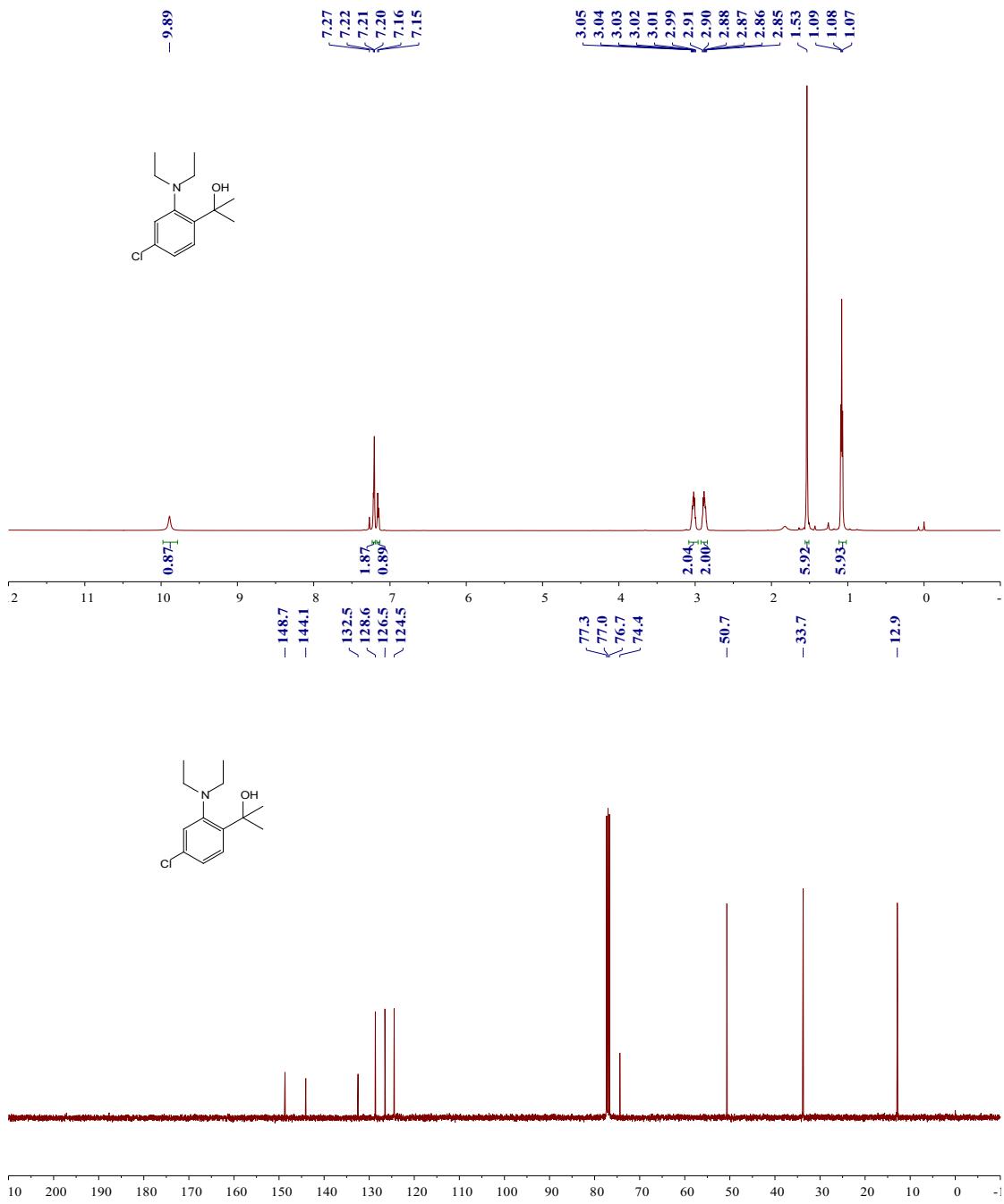
¹H and ¹³C NMR spectra of compound **3ea**



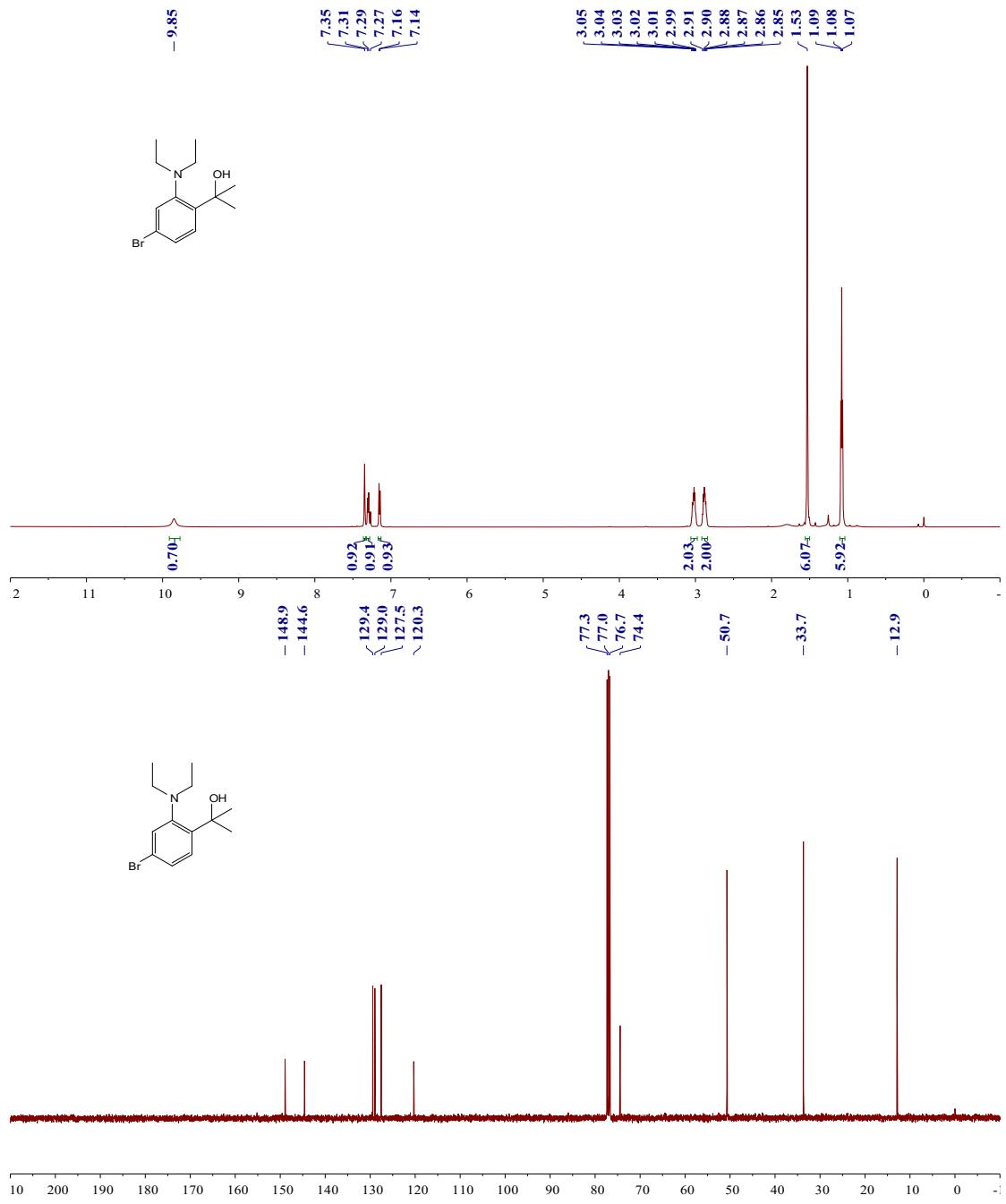
¹H and ¹³C NMR spectra of compound **3fa**



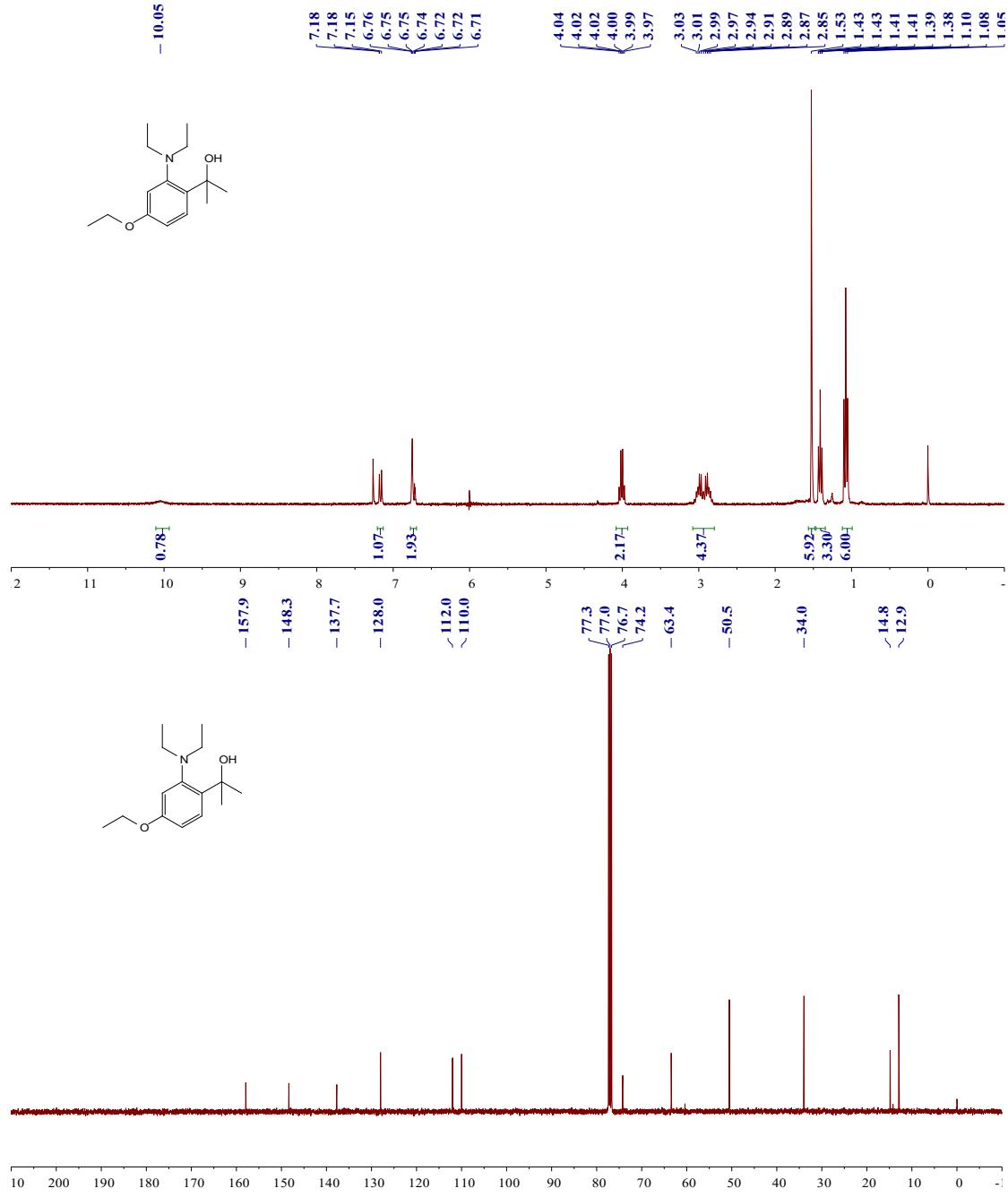
¹H and ¹³C NMR spectra of compound 3ga



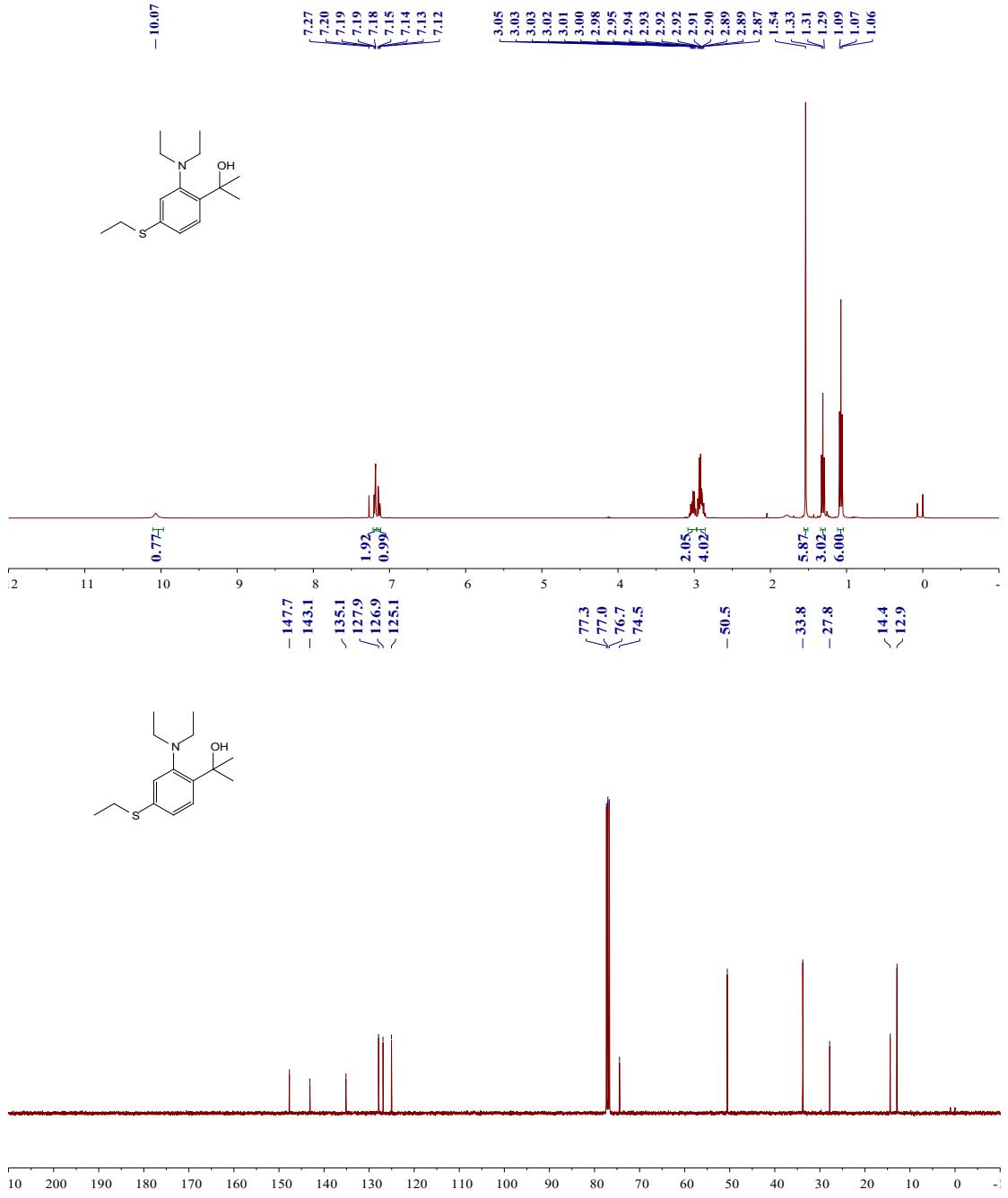
¹H and ¹³C NMR spectra of compound **3ha**



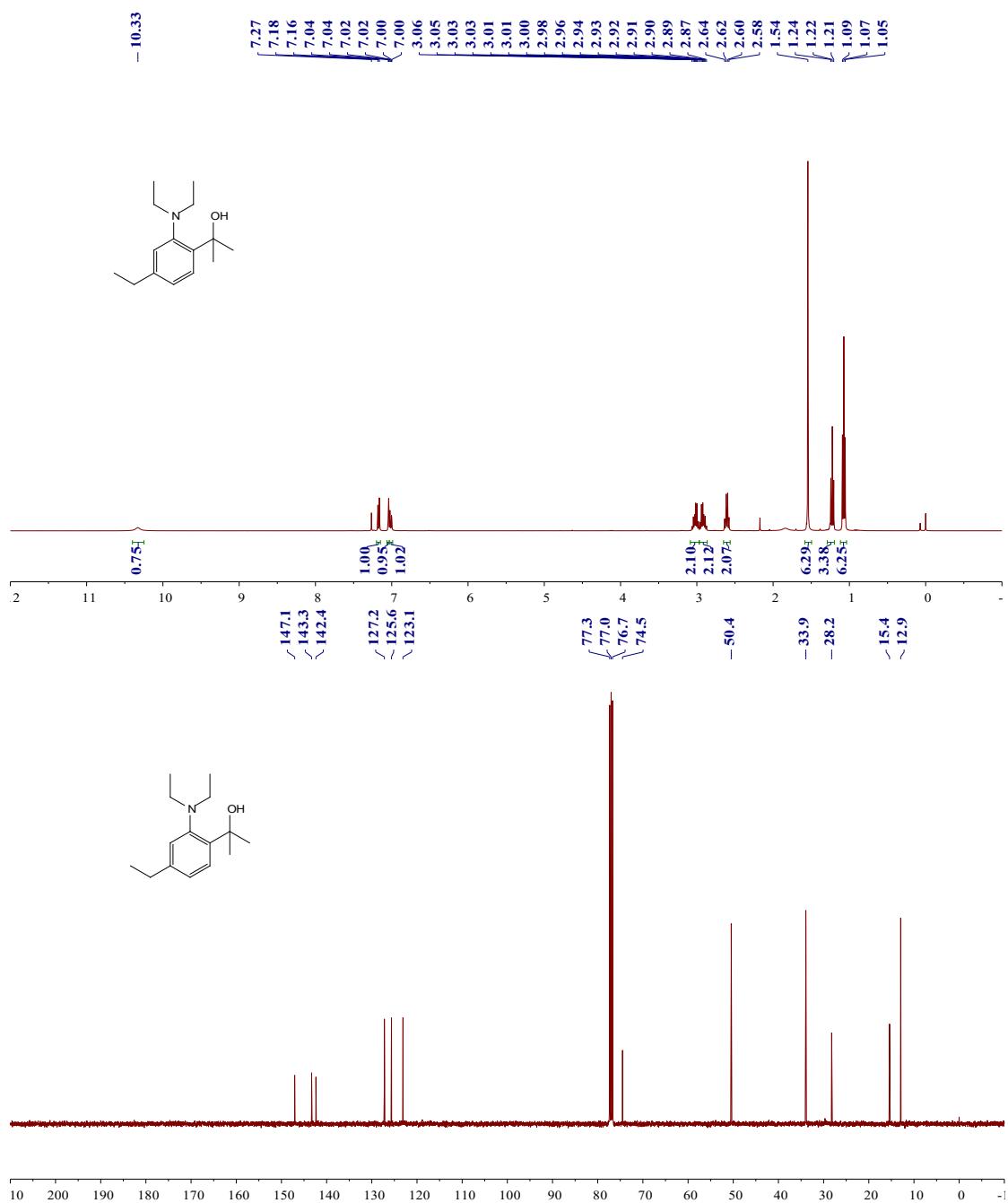
¹H and ¹³C NMR spectra of compound **3ia**



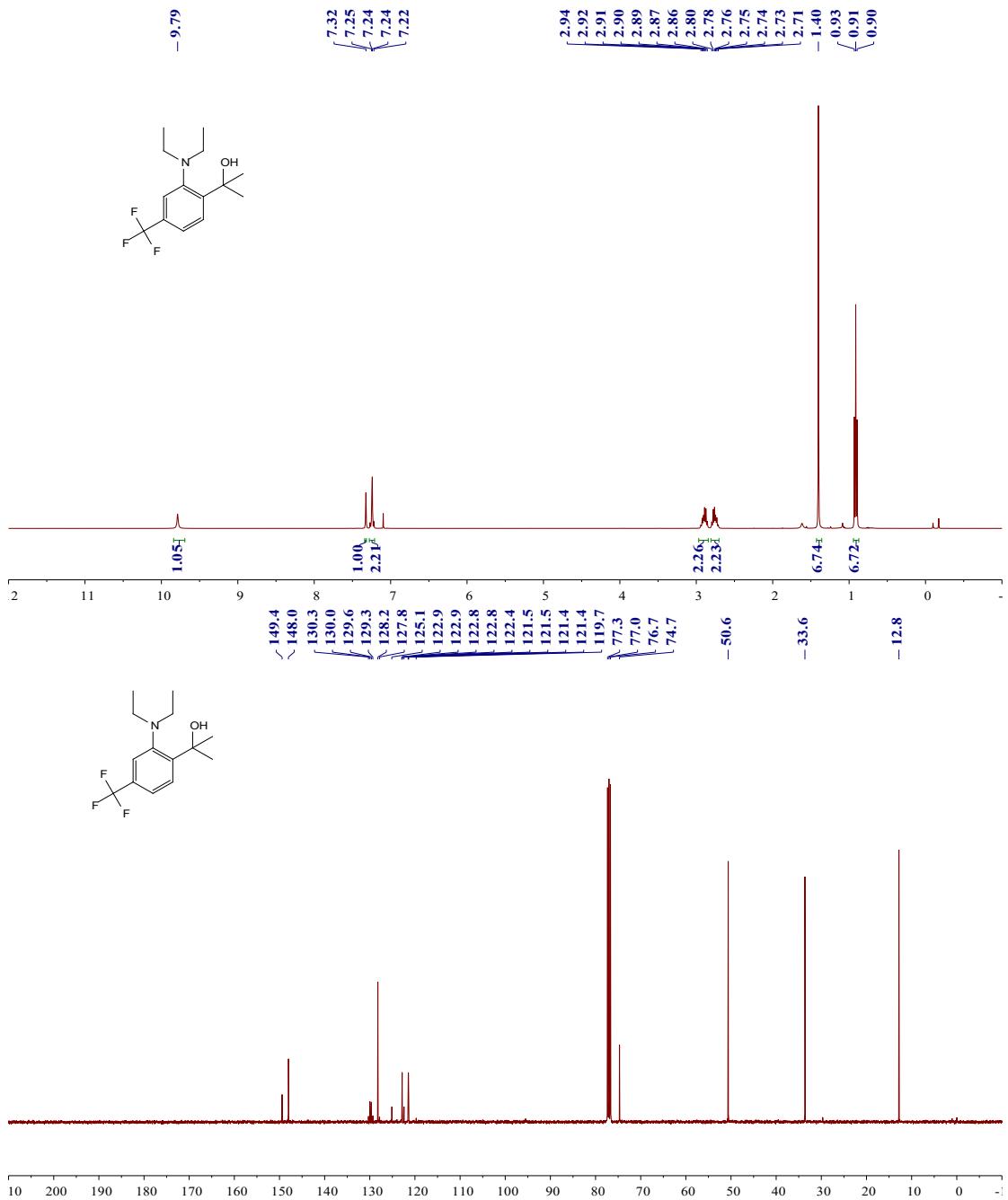
¹H and ¹³C NMR spectra of compound 3ja

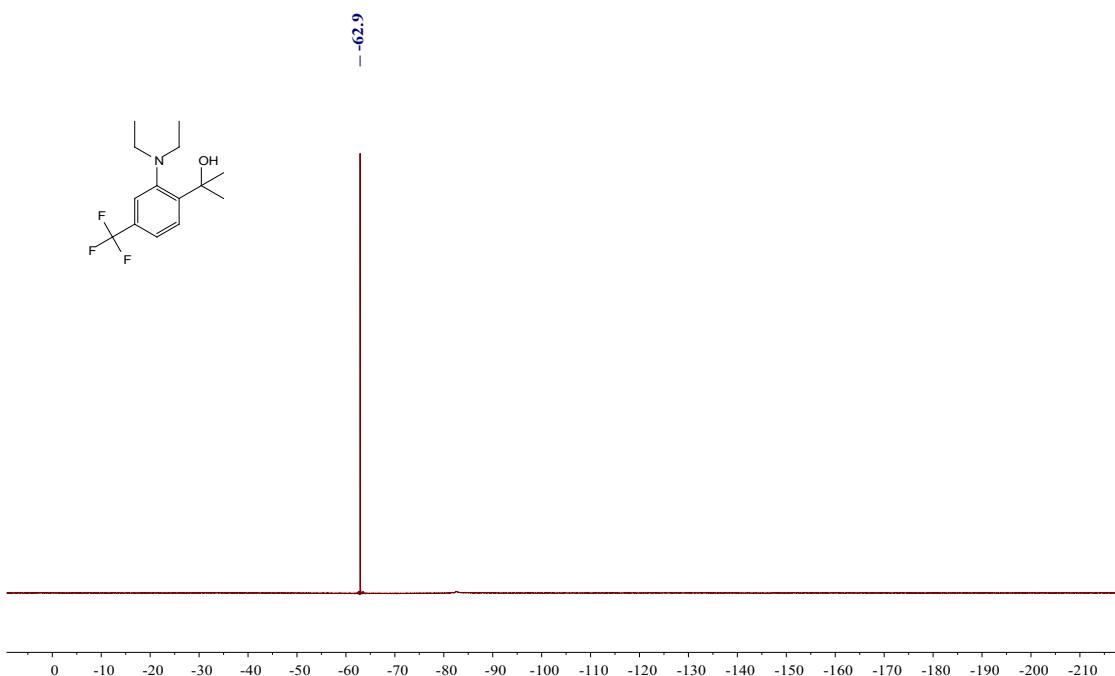


¹H and ¹³C NMR spectra of compound 3ka

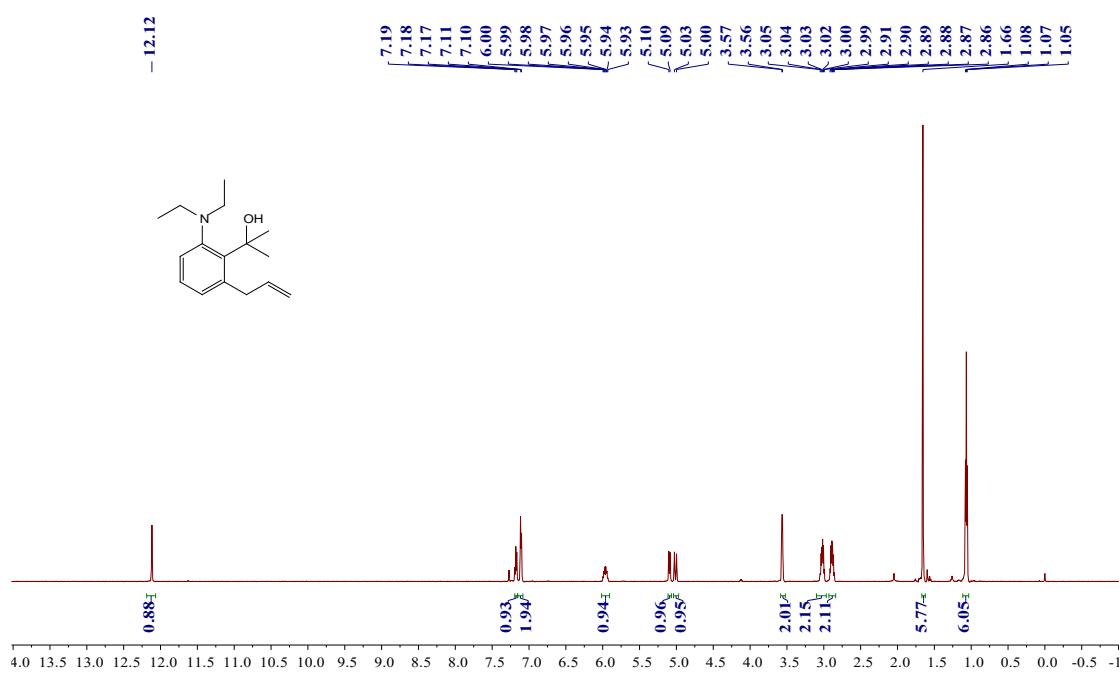


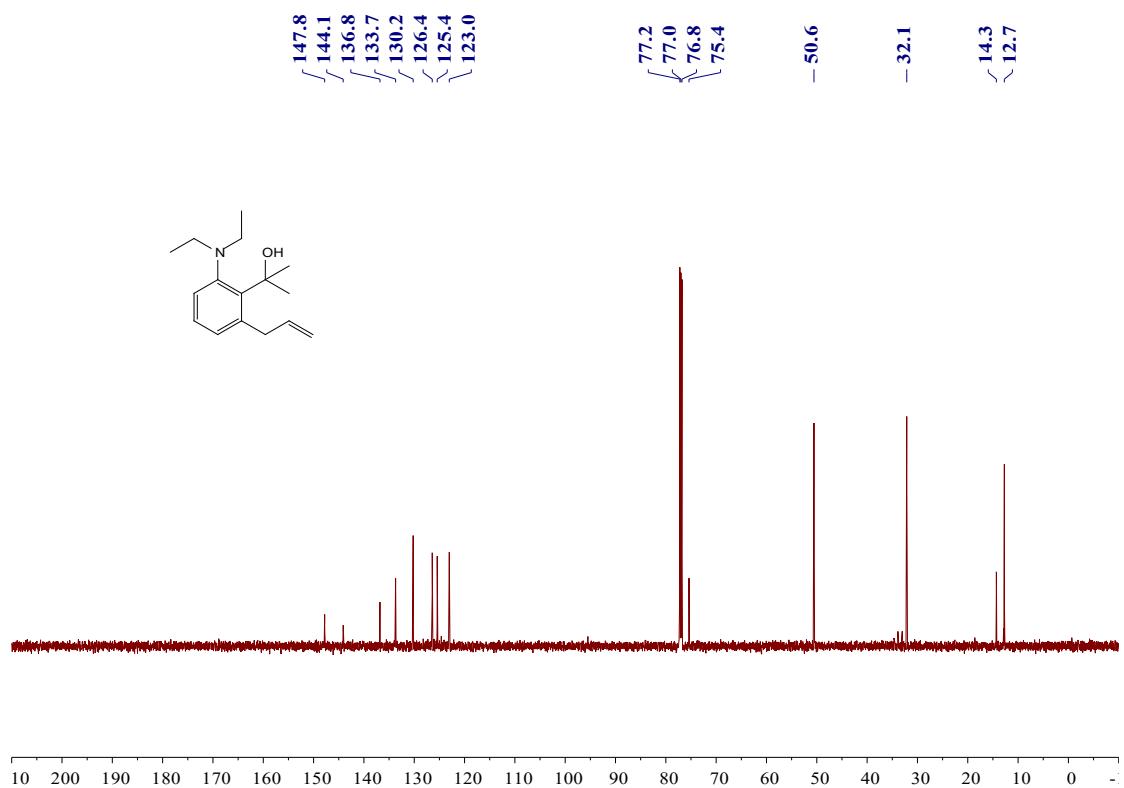
¹H, ¹³C and ¹⁹F NMR spectra of compound 3la



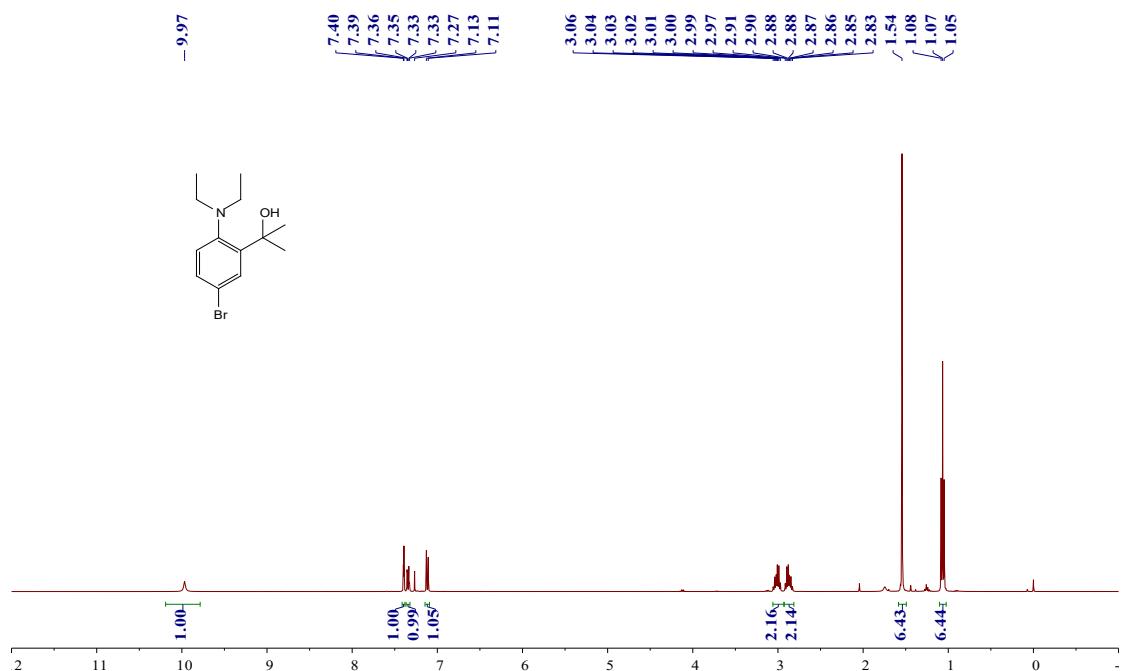


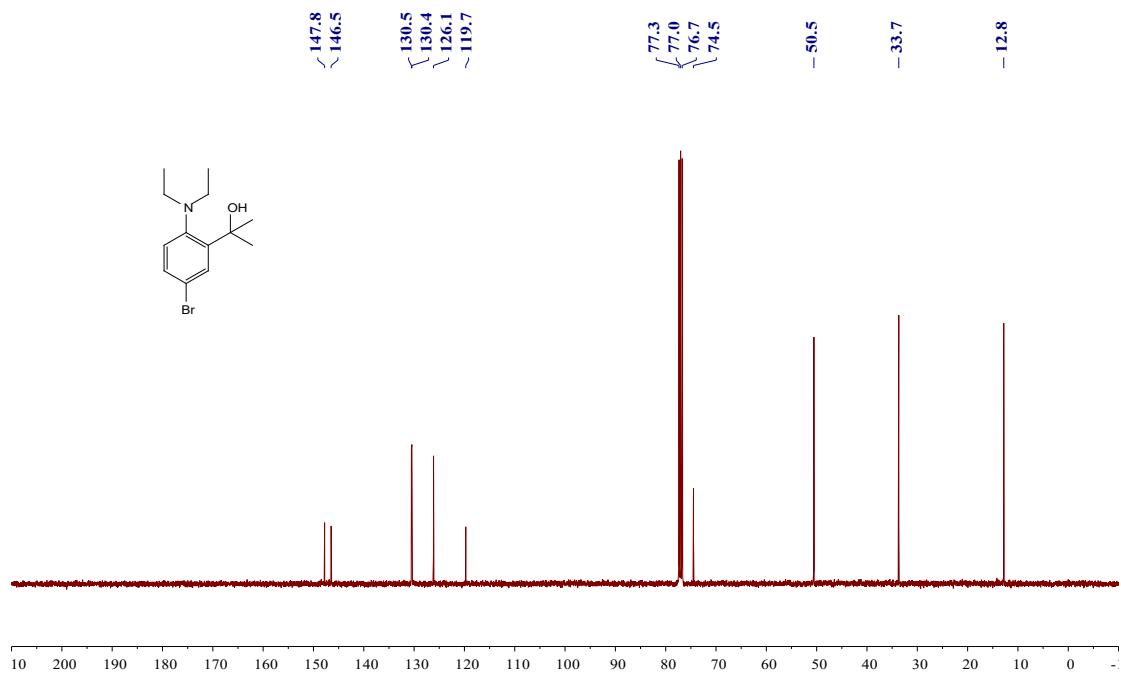
¹H and ¹³C NMR spectra of compound **3ma**



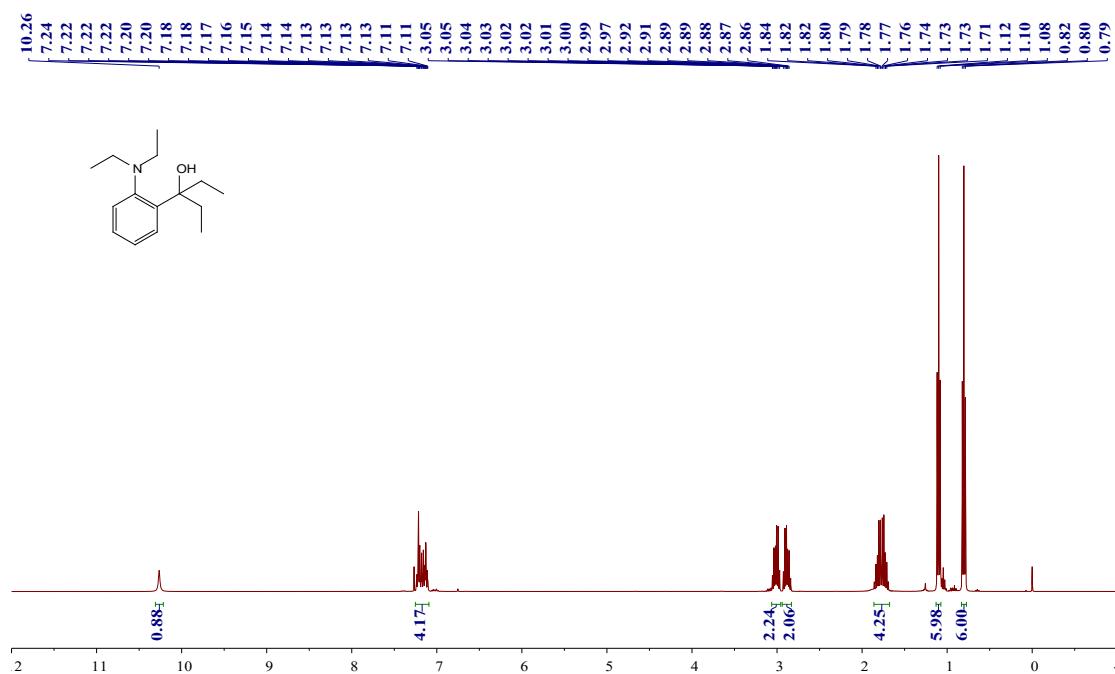


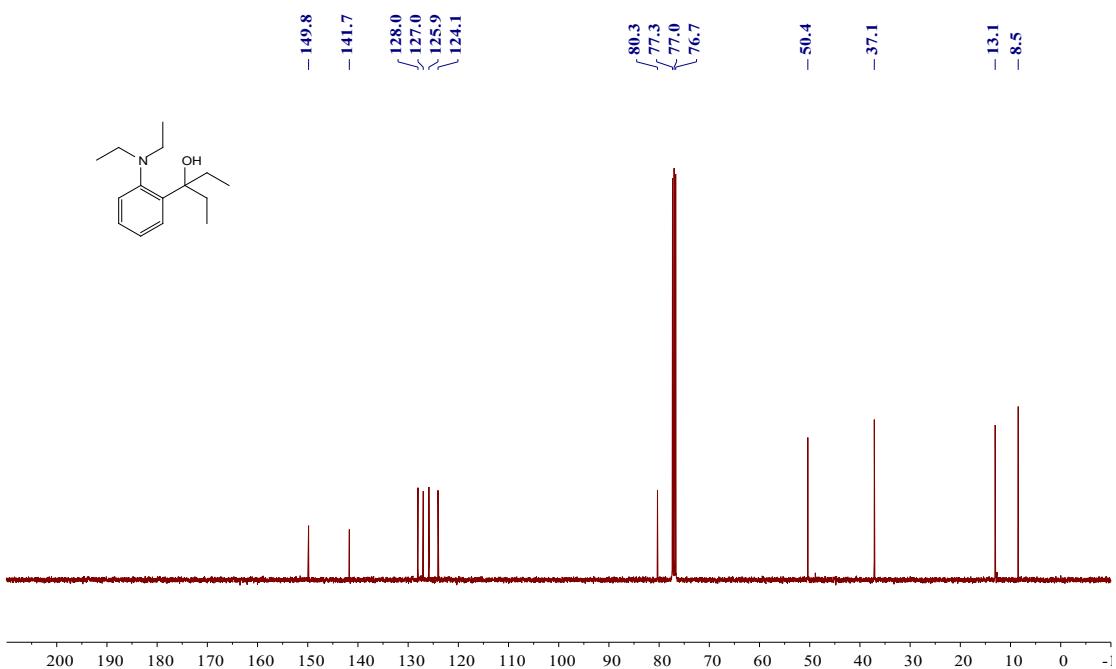
¹H and ¹³C NMR spectra of compound **1n**



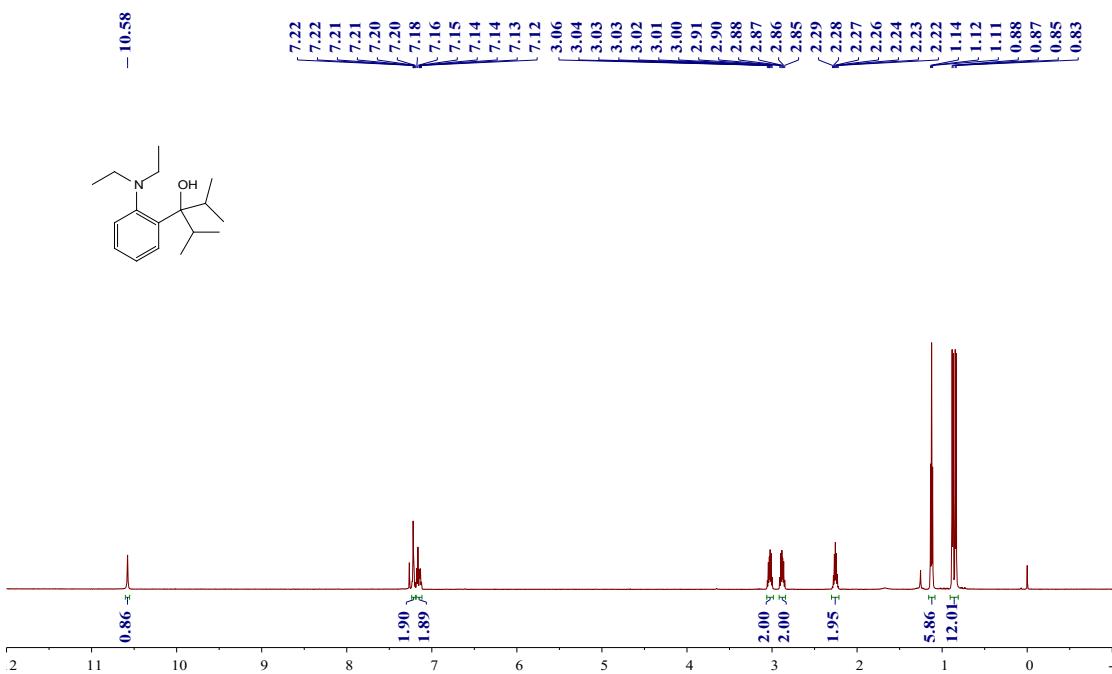


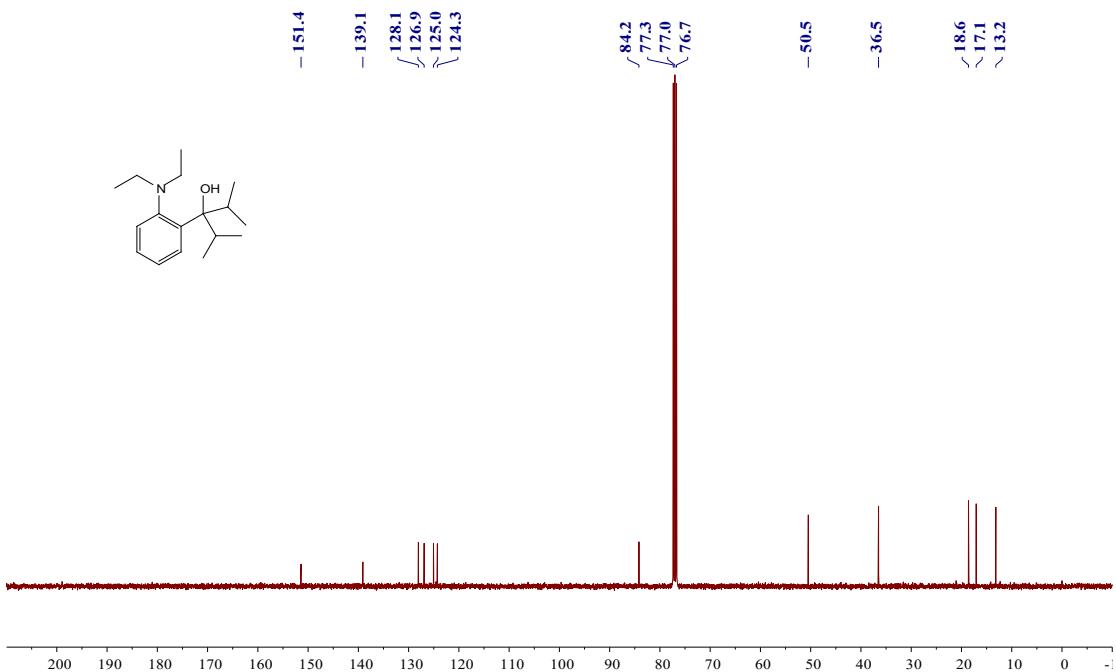
^1H and ^{13}C NMR spectra of compound **3ab**



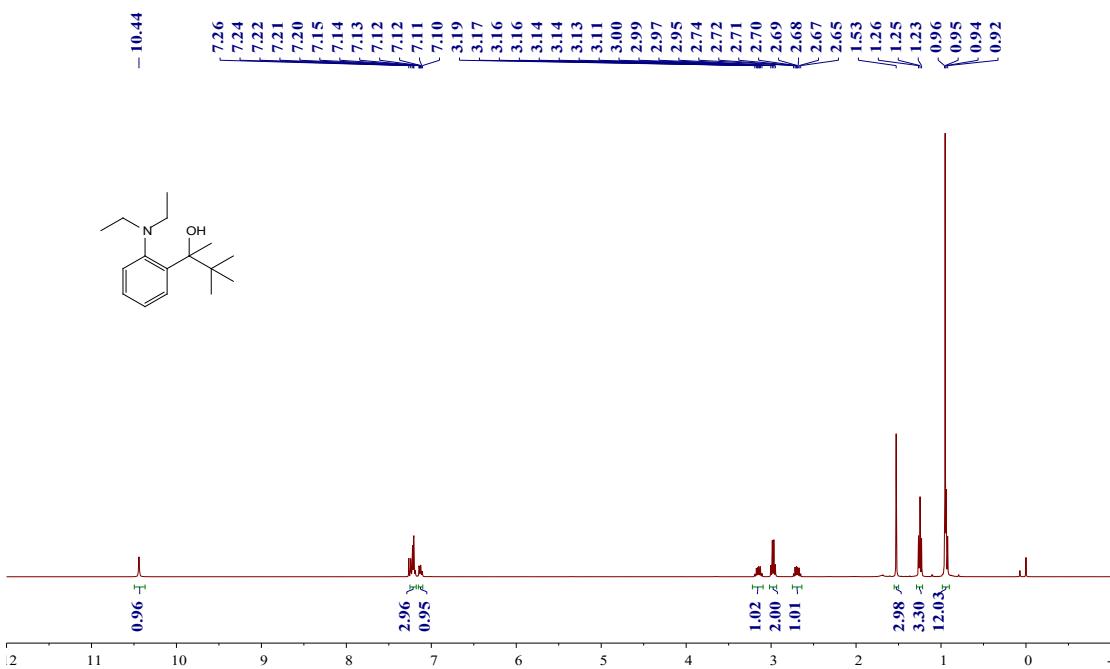


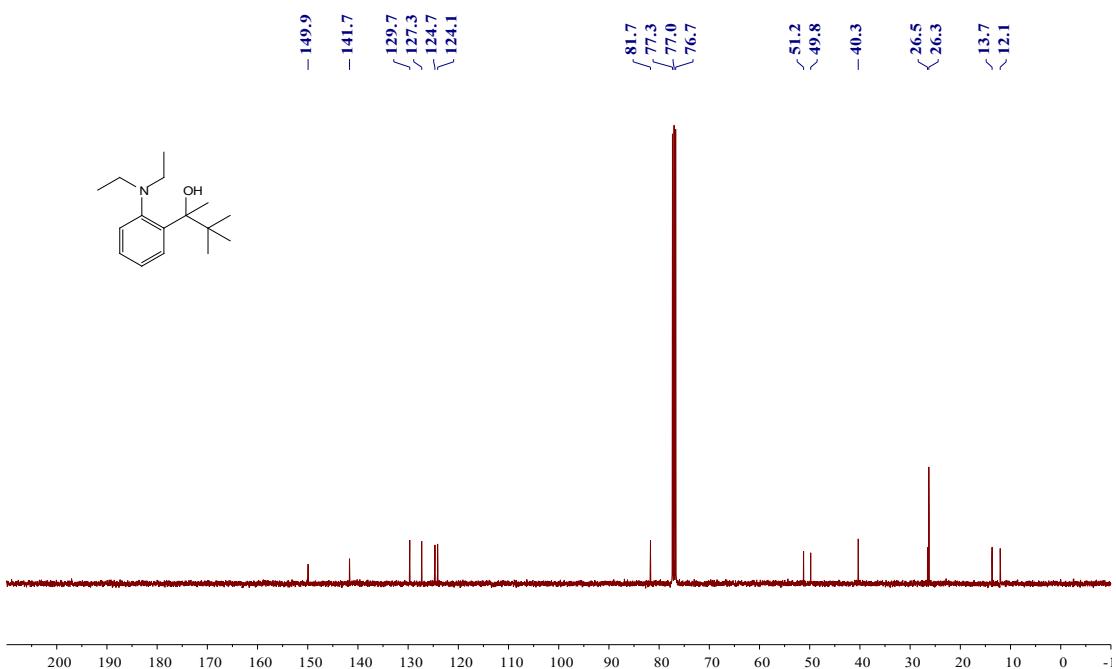
¹H and ¹³C NMR spectra of compound 3ac



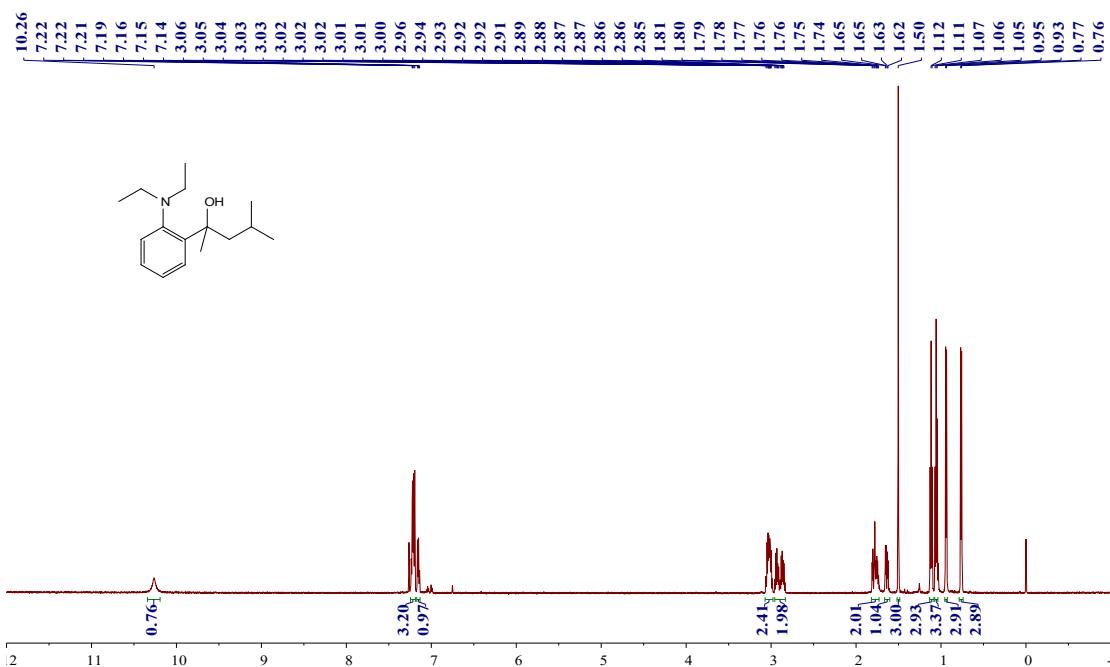


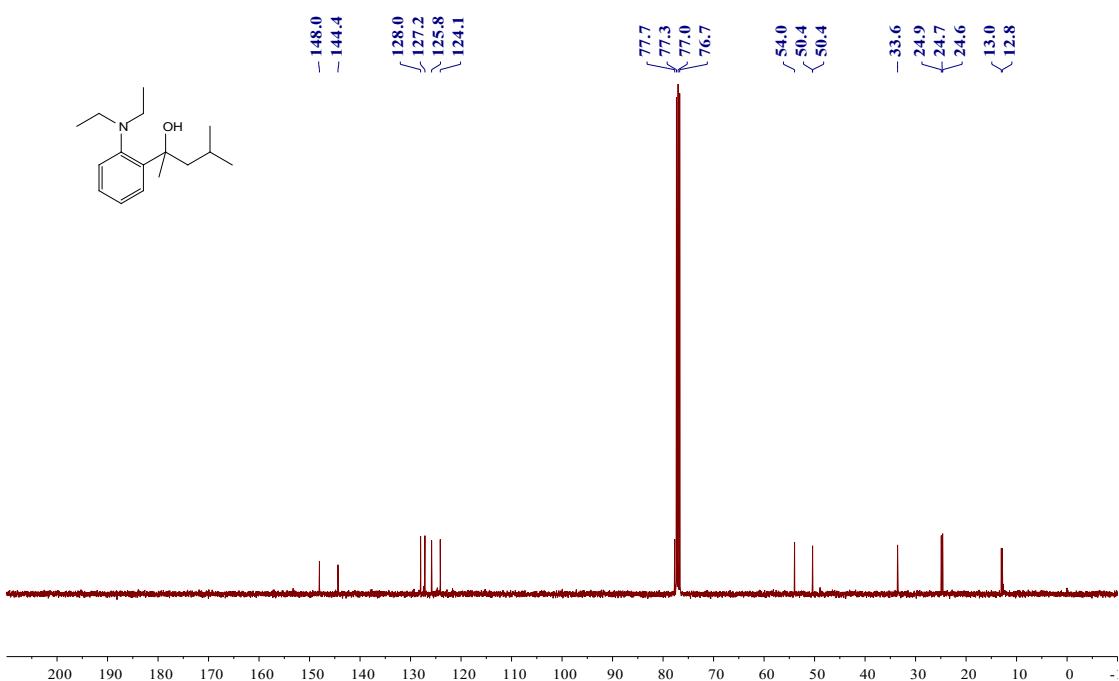
^1H and ^{13}C NMR spectra of compound 3ad



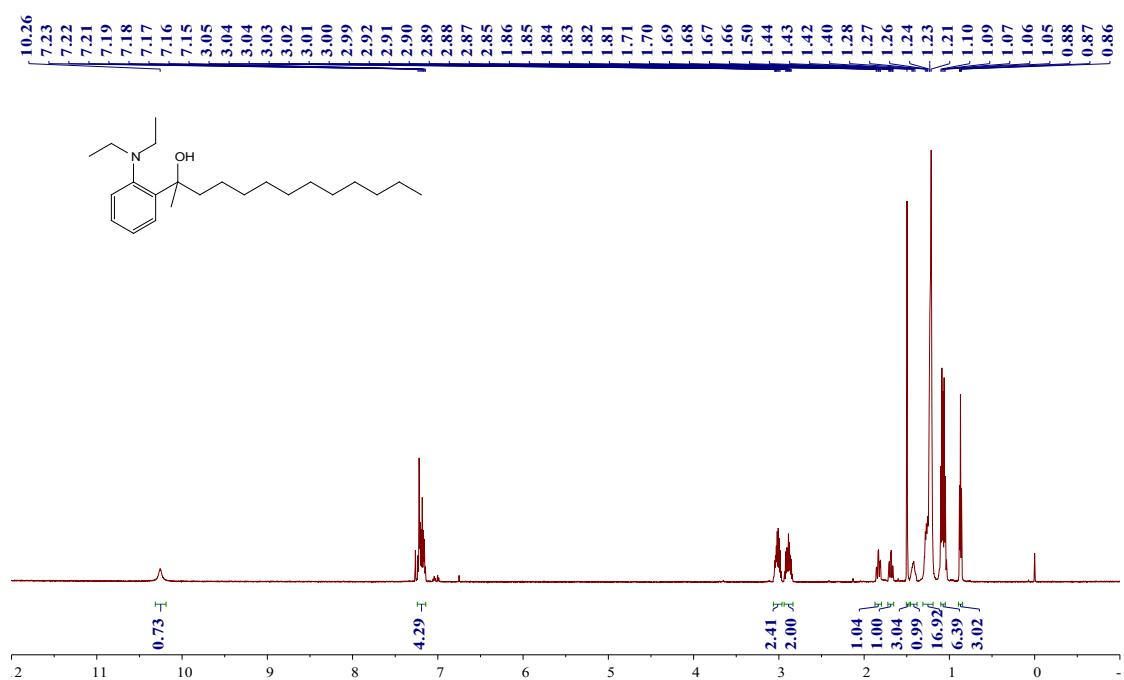


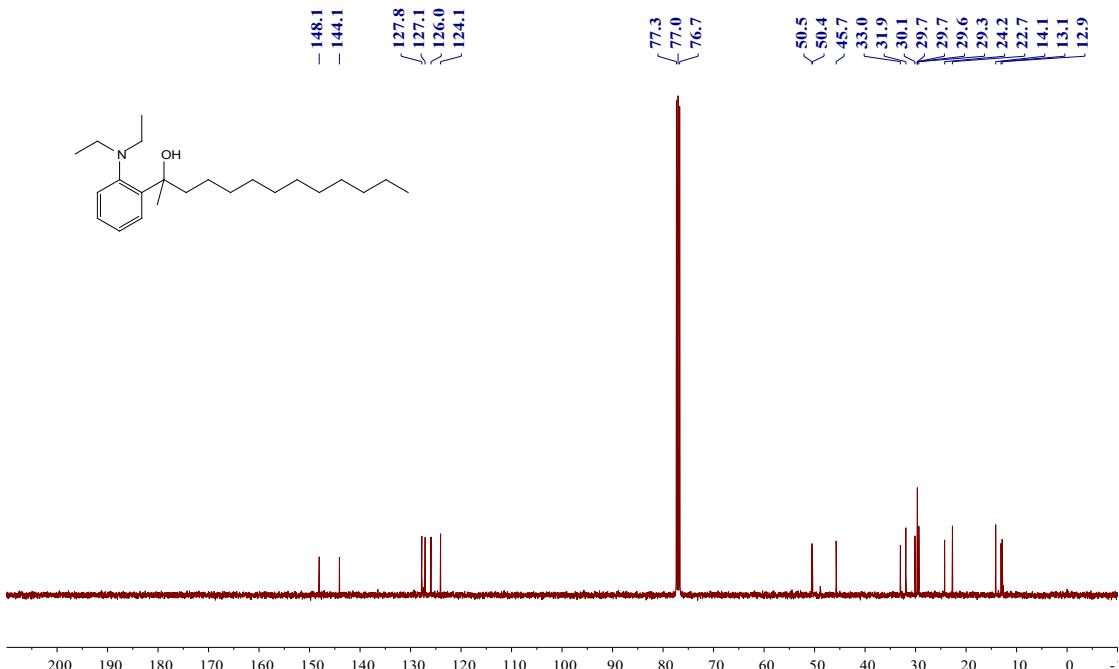
¹H and ¹³C NMR spectra of compound 3ae



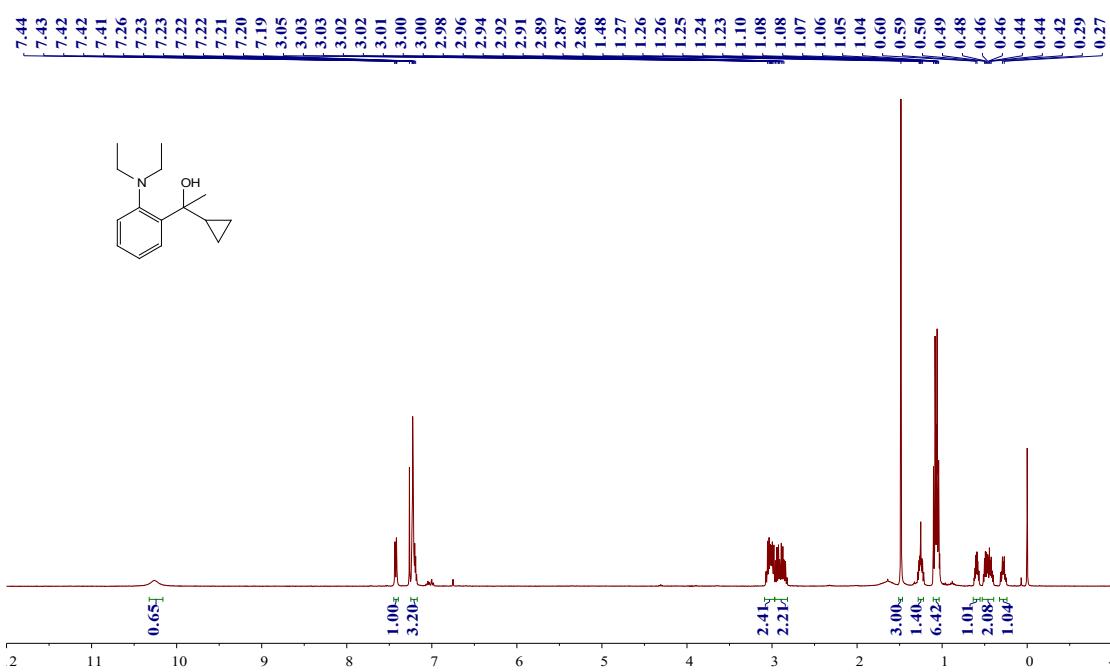


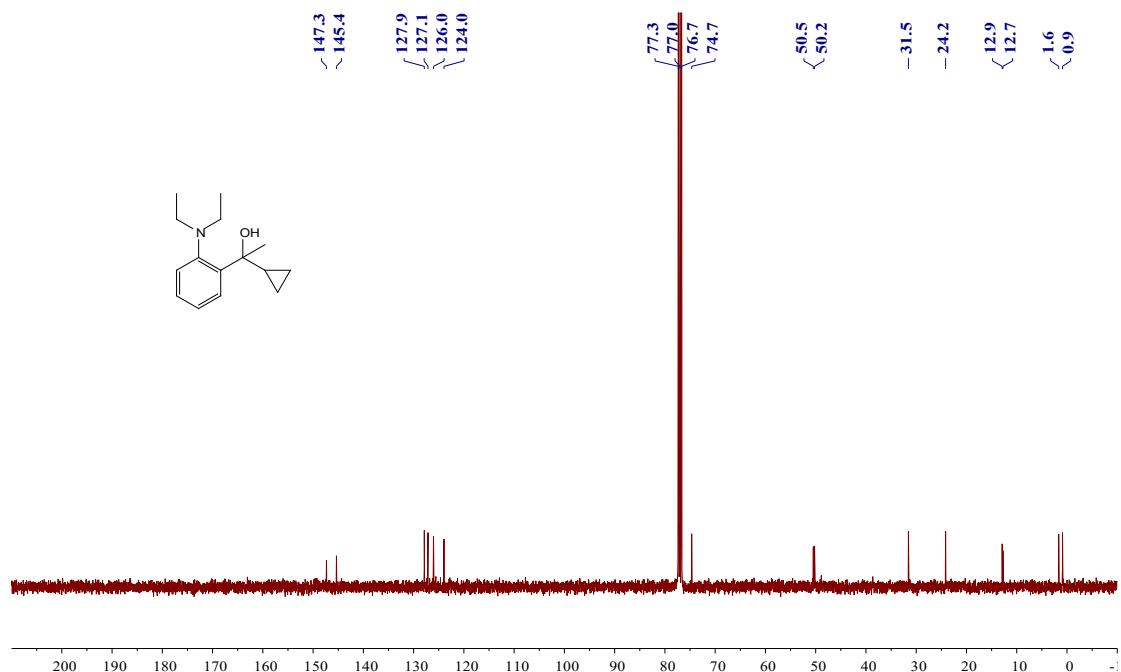
¹H and ¹³C NMR spectra of compound **3af**



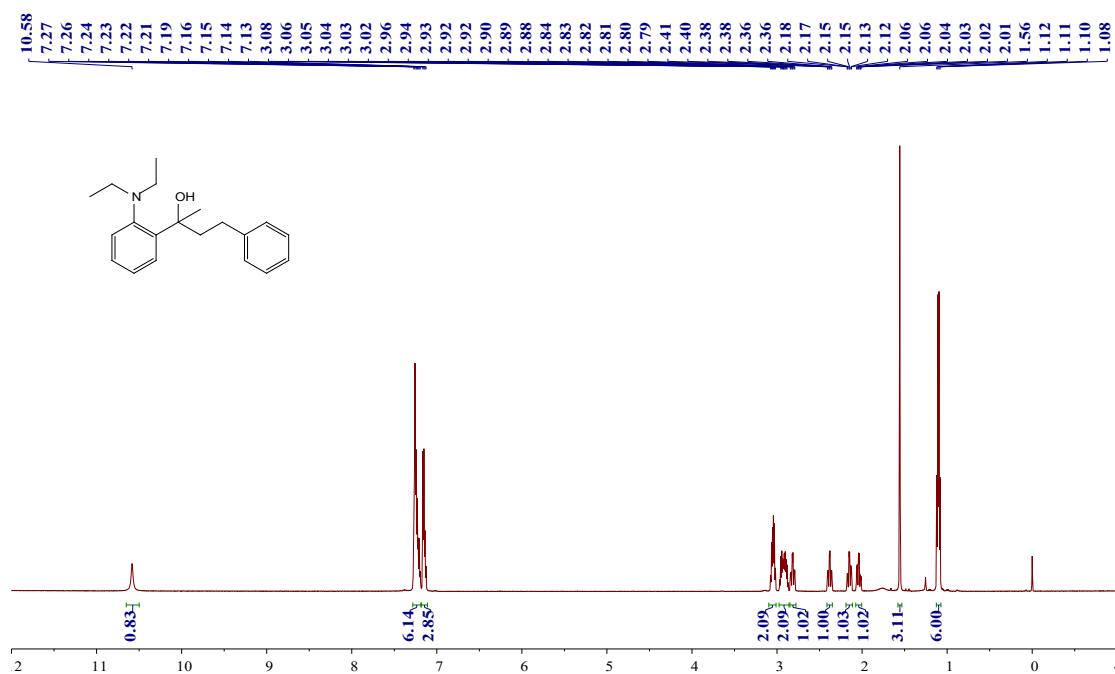


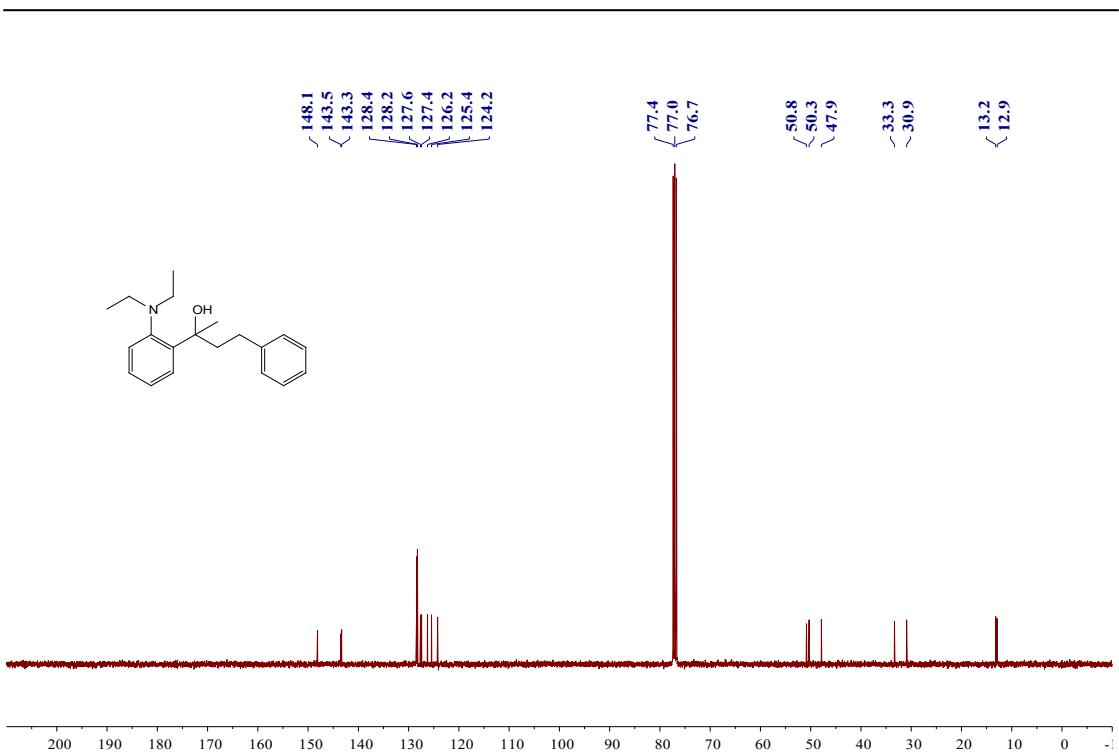
¹H and ¹³C NMR spectra of compound 3ag



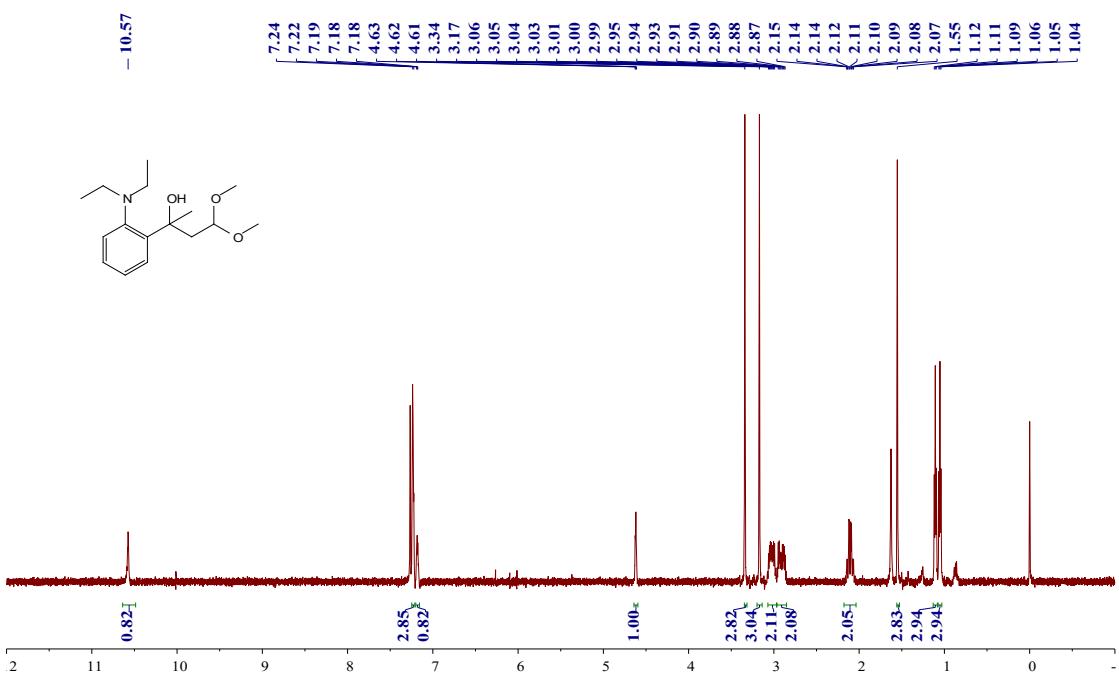


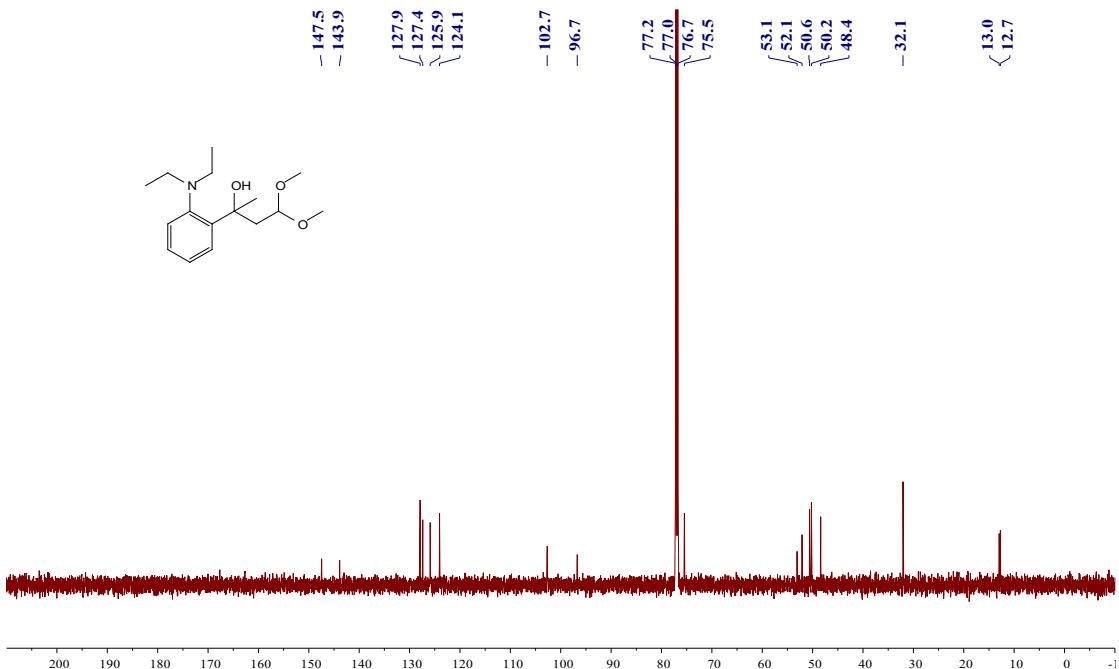
¹H and ¹³C NMR spectra of compound **3ah**



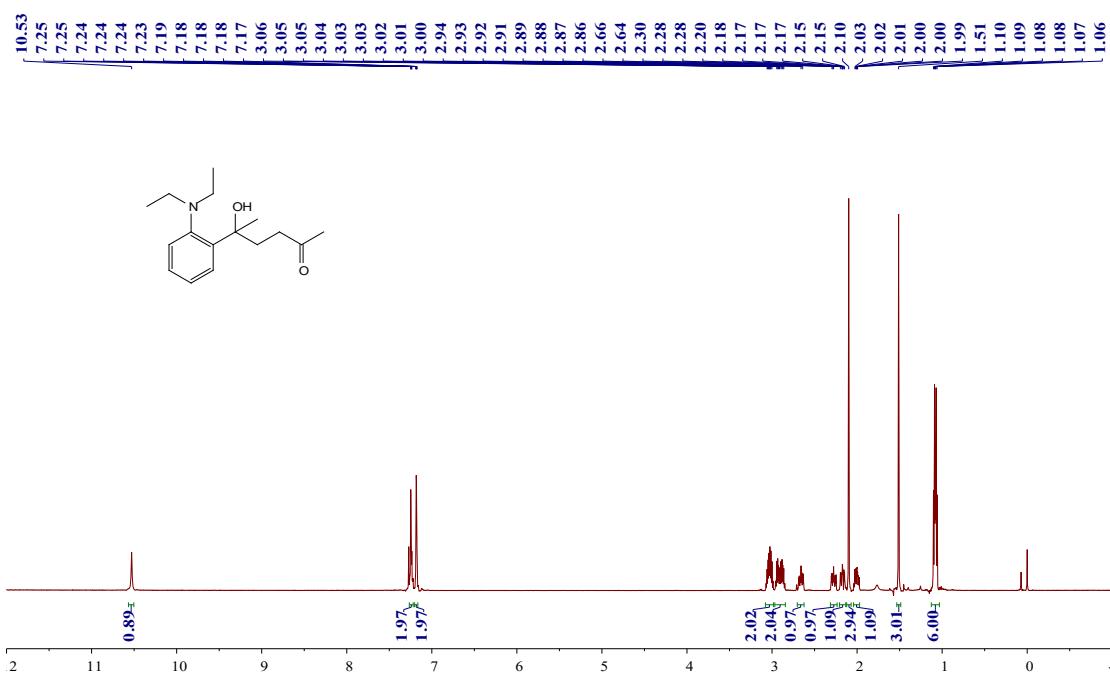


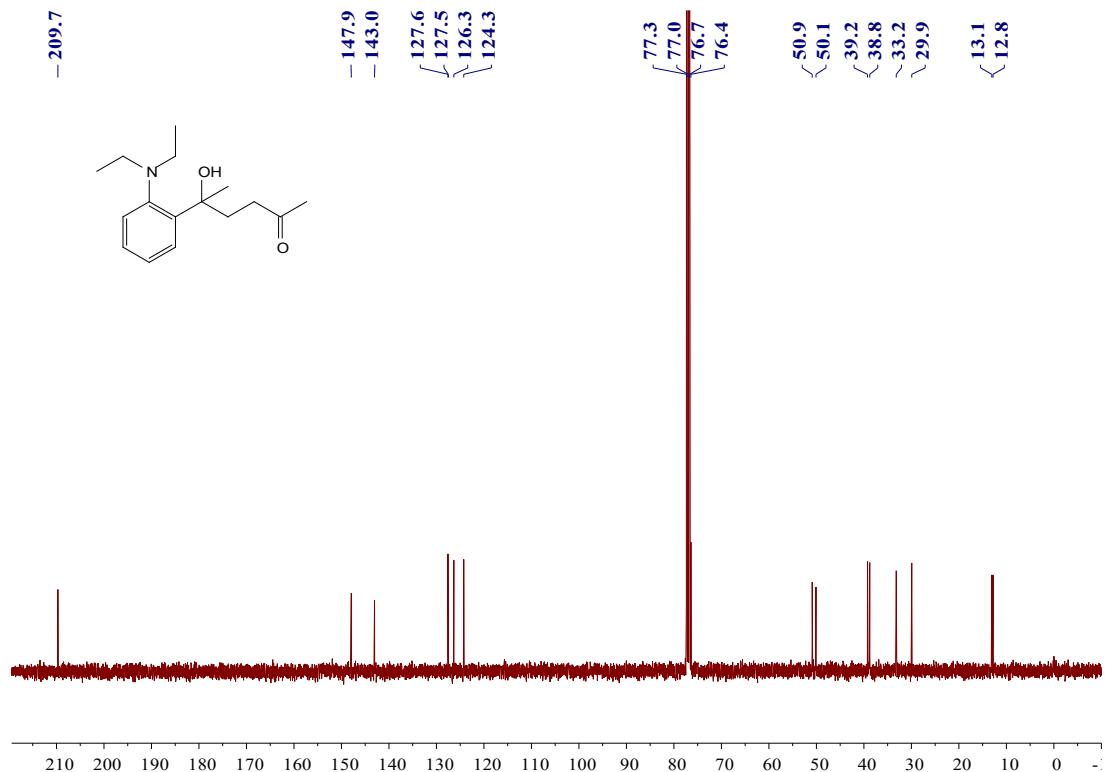
¹H and ¹³C NMR spectra of compound 3ai



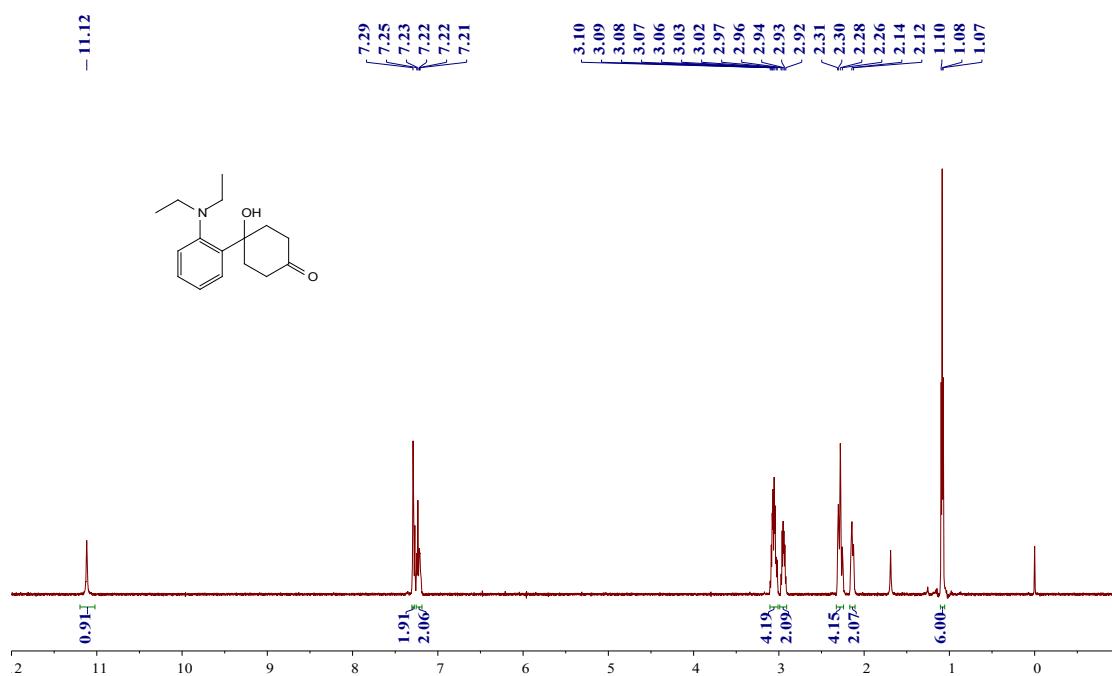


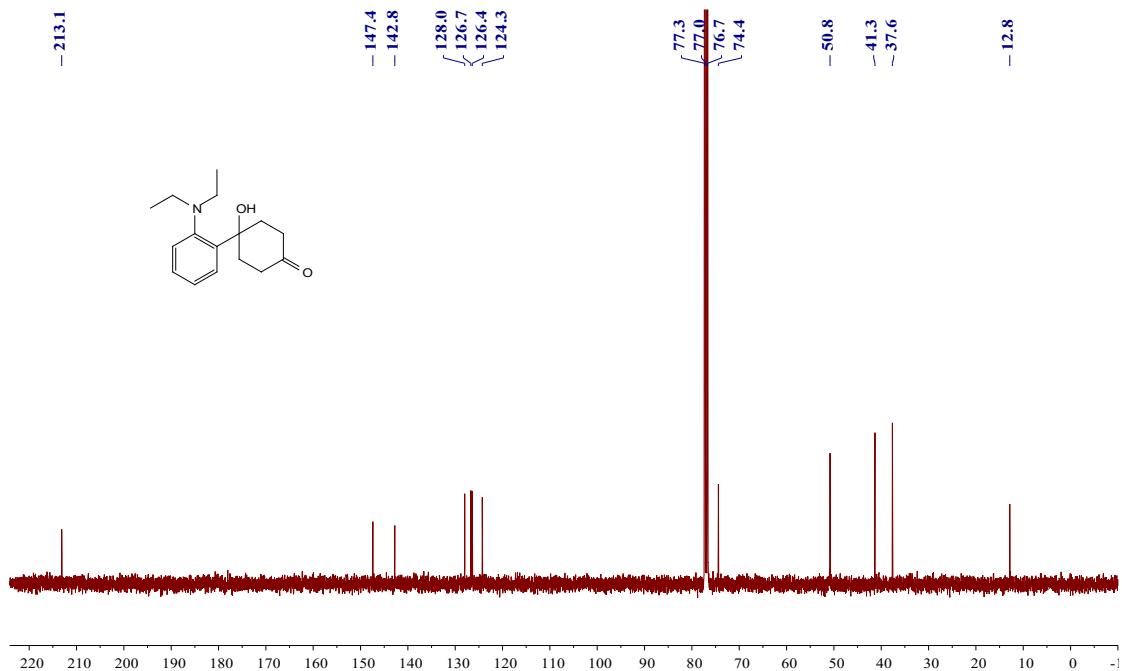
¹H and ¹³C NMR spectra of compound 3aj



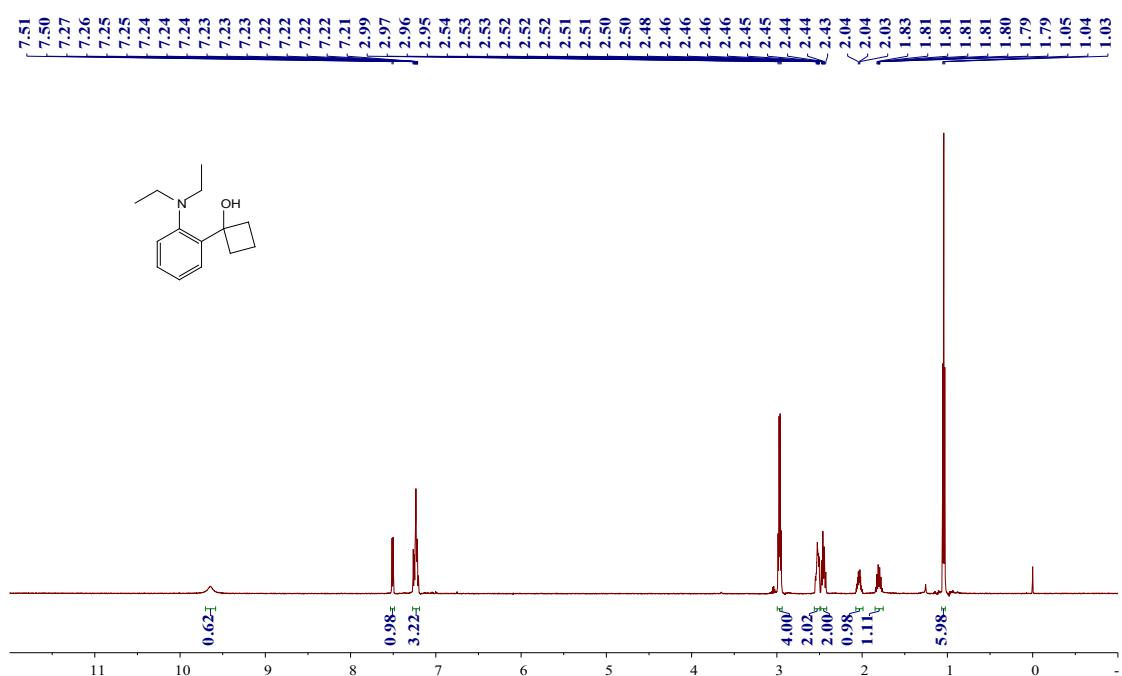


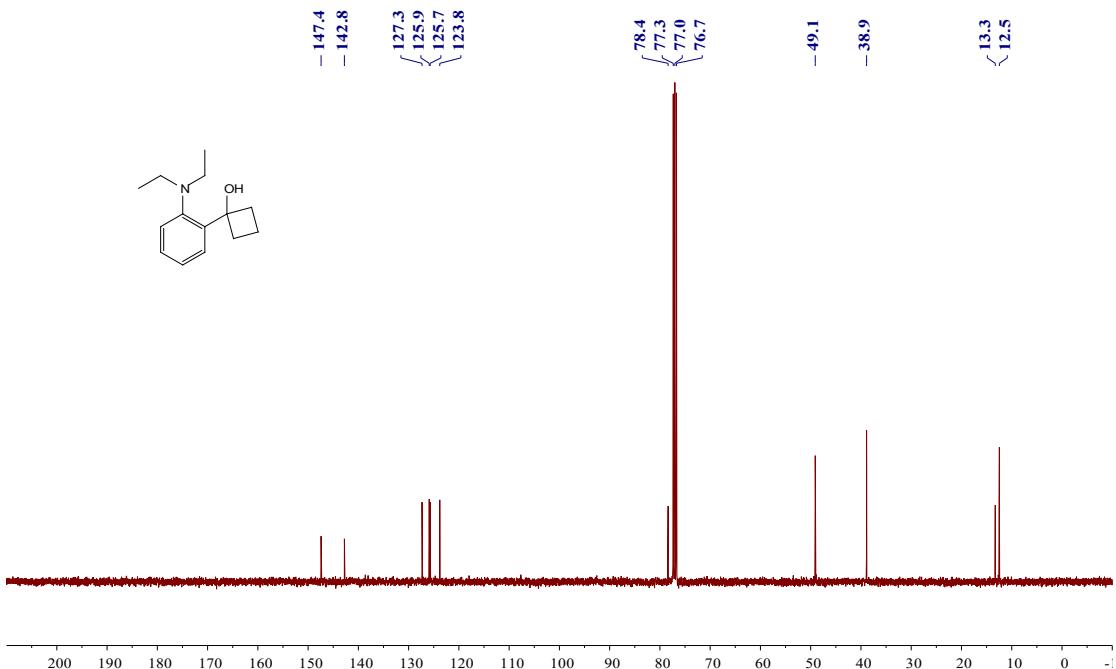
¹H and ¹³C NMR spectra of compound 3ak



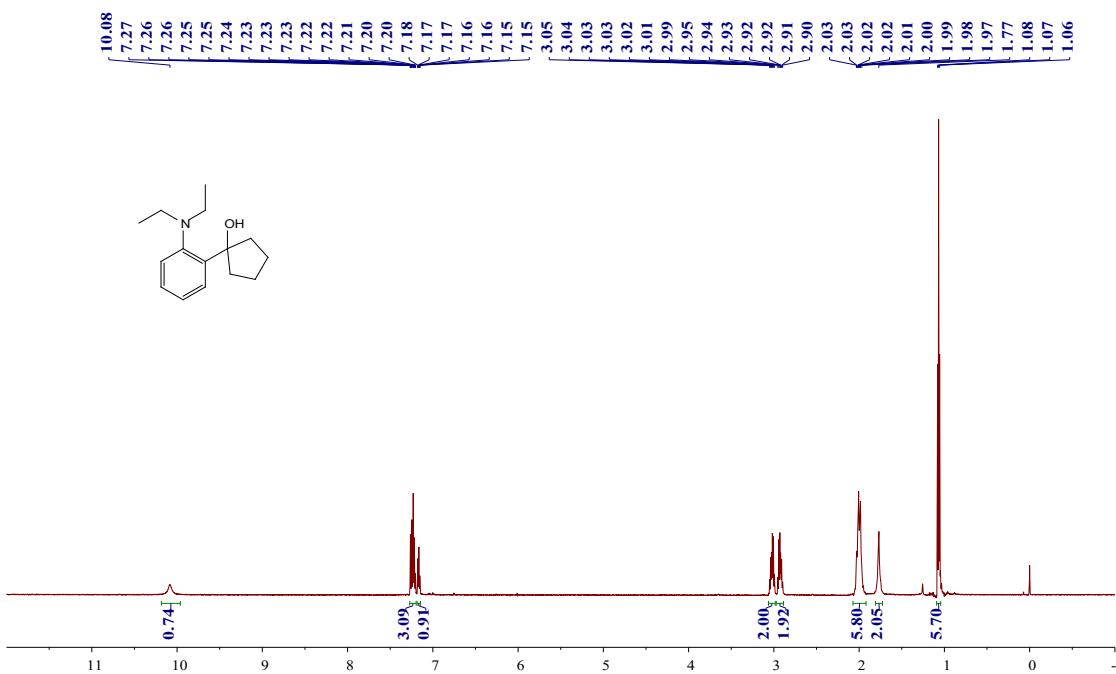


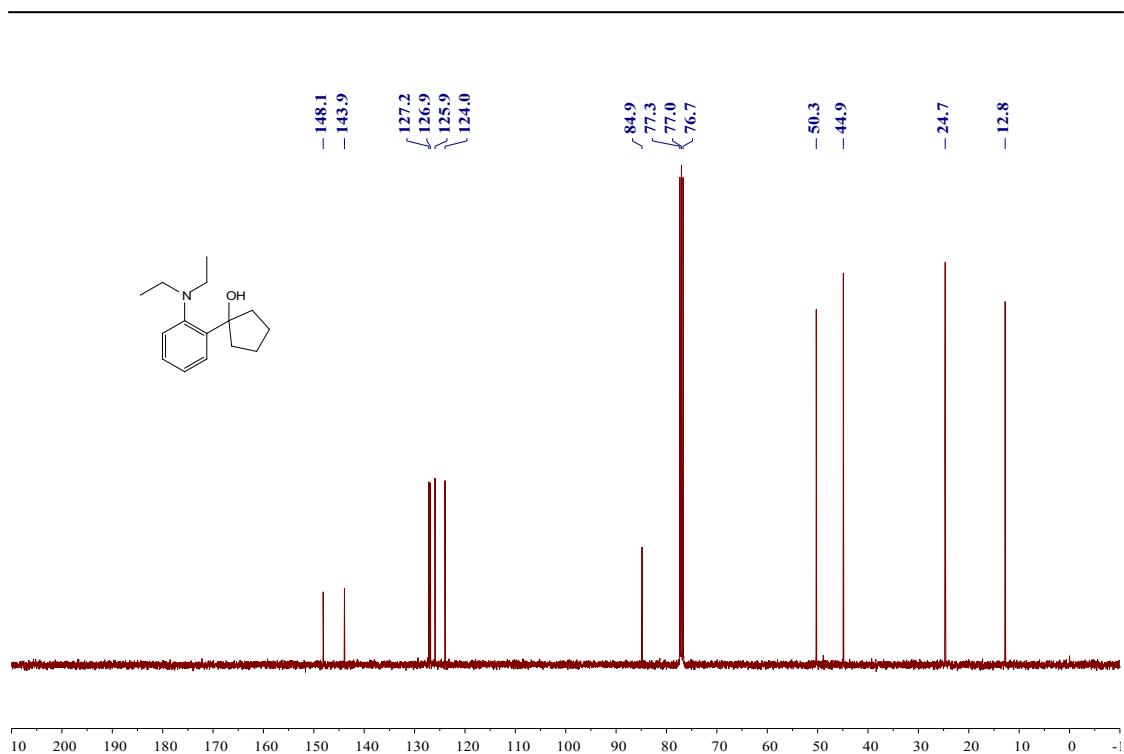
¹H and ¹³C NMR spectra of compound 3al



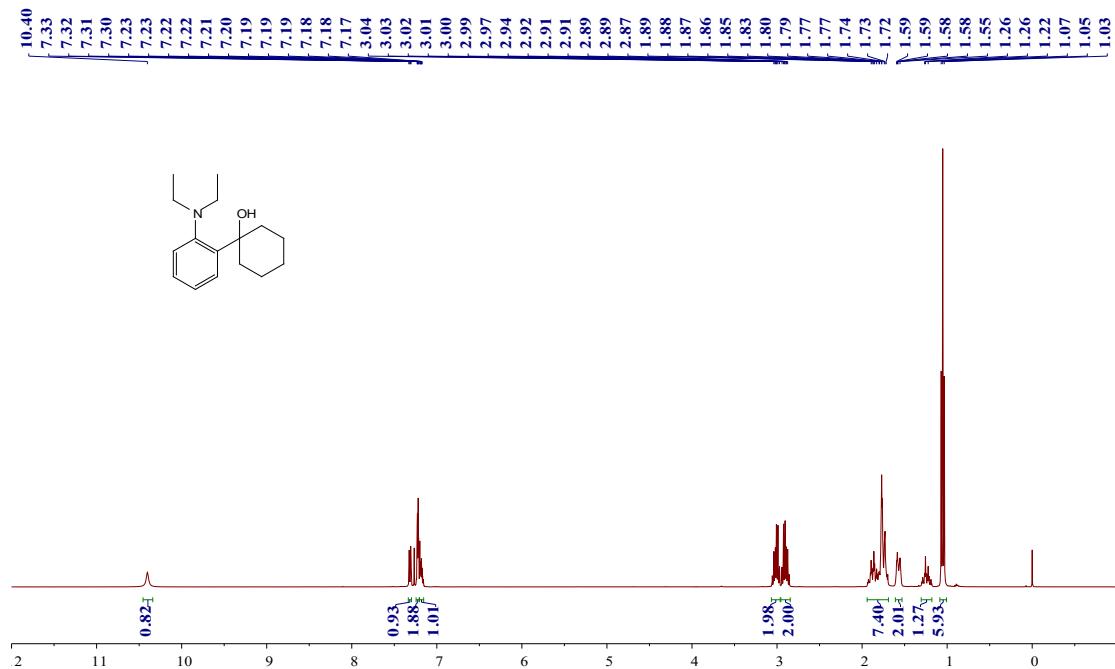


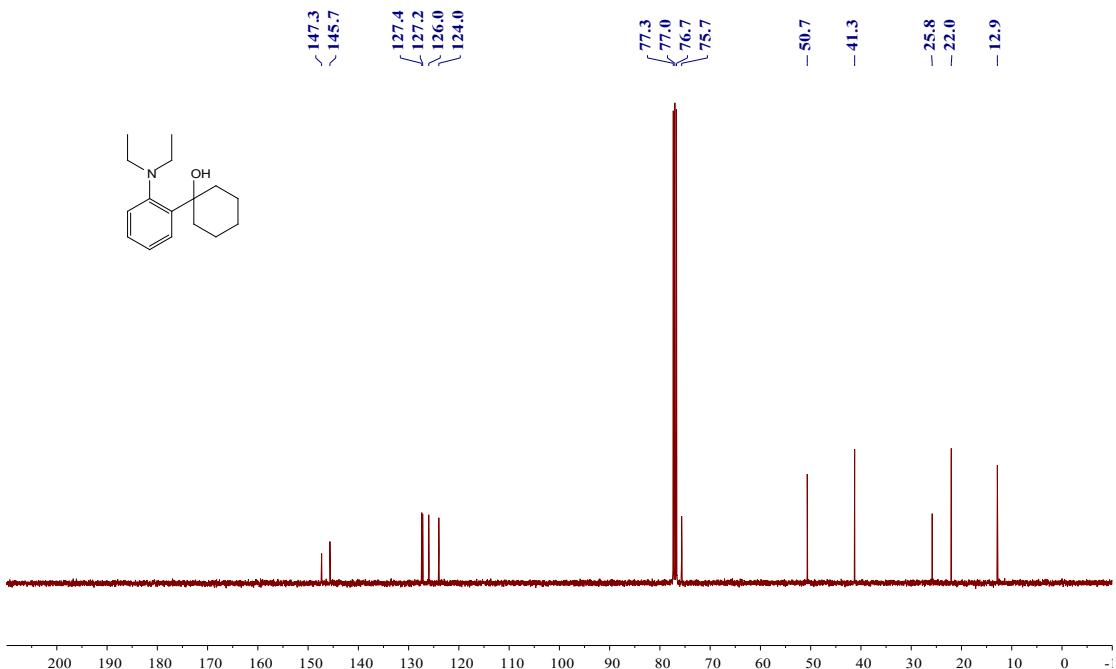
¹H and ¹³C NMR spectra of compound 3am



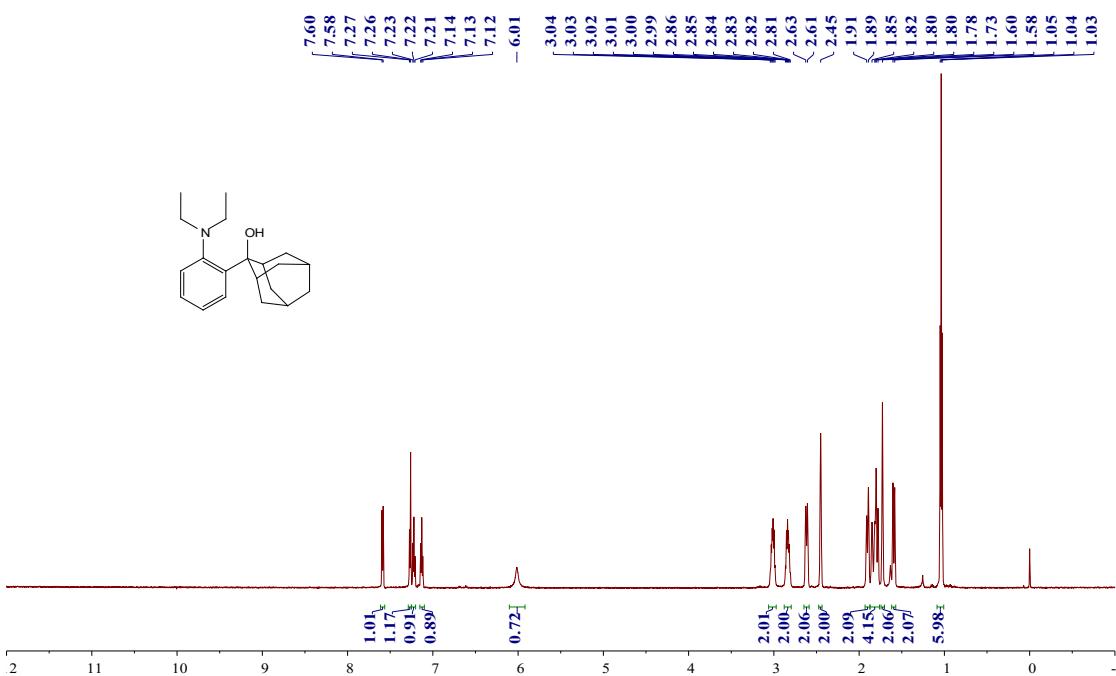


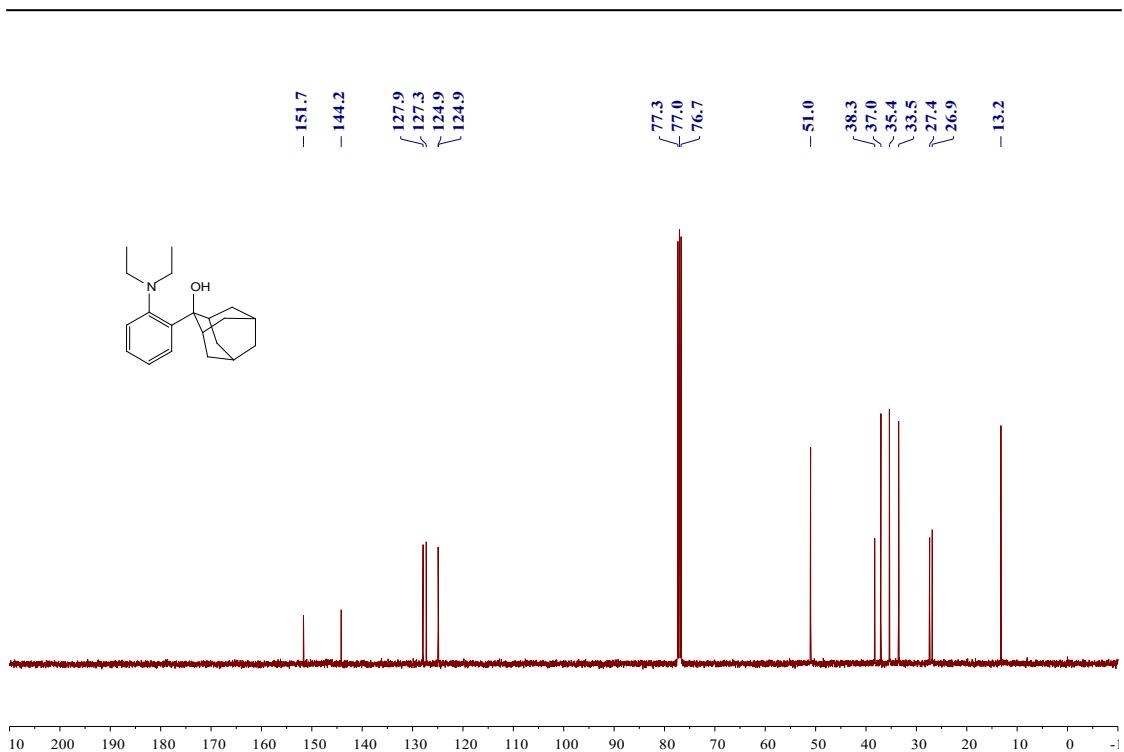
^1H and ^{13}C NMR spectra of compound **3an**



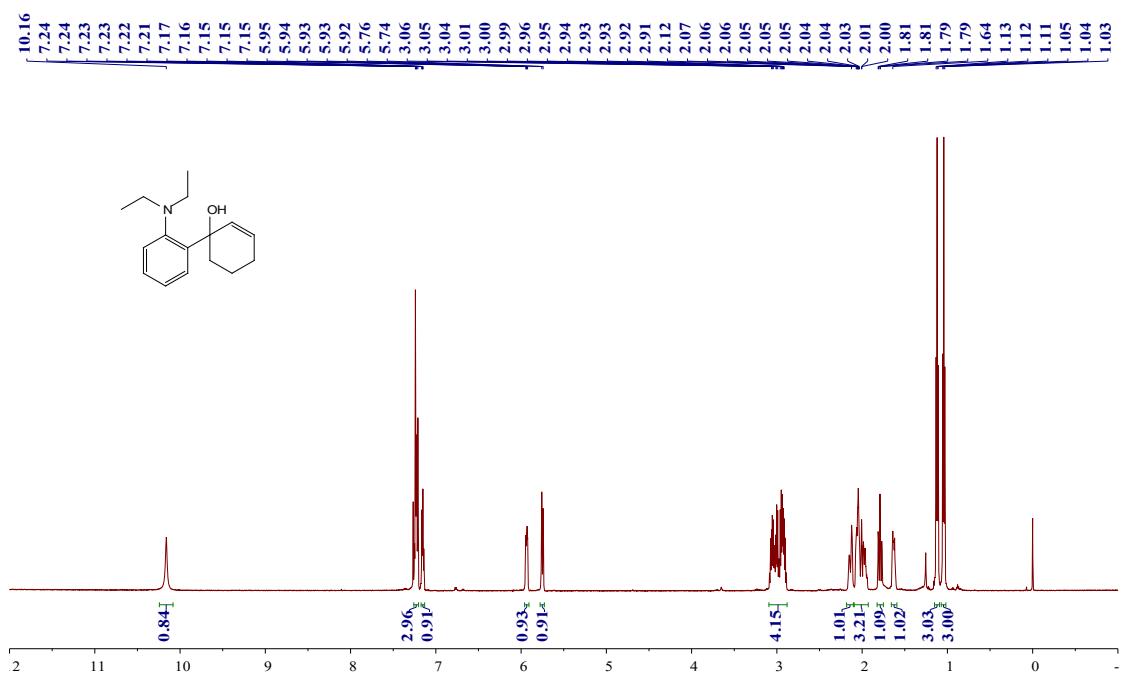


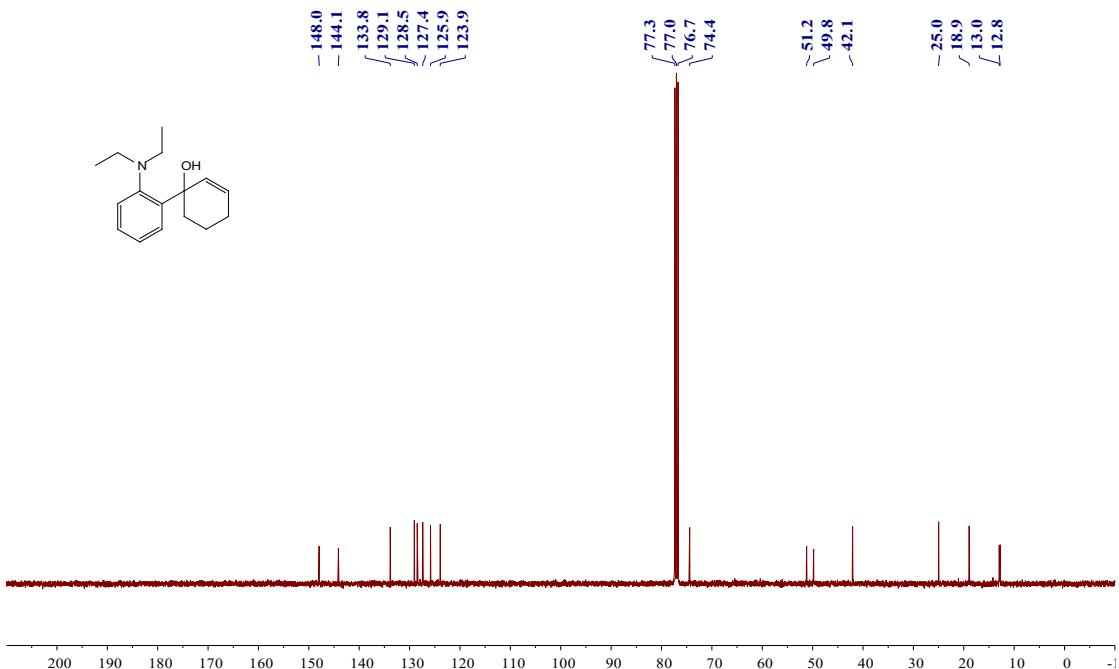
¹H and ¹³C NMR spectra of compound **3ao**



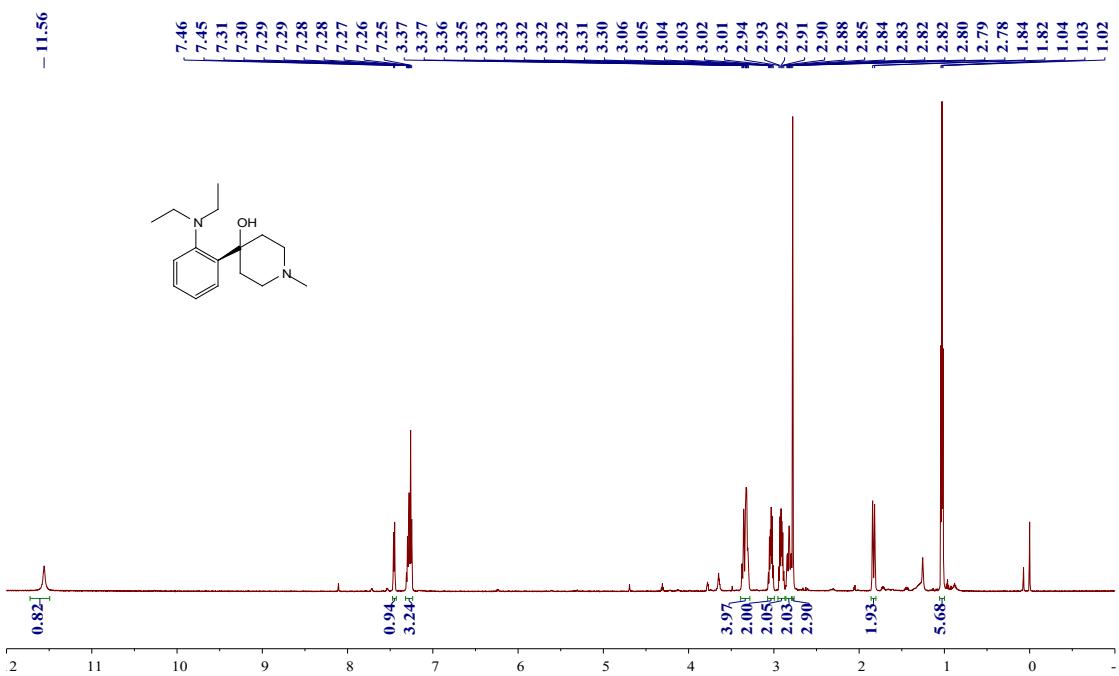


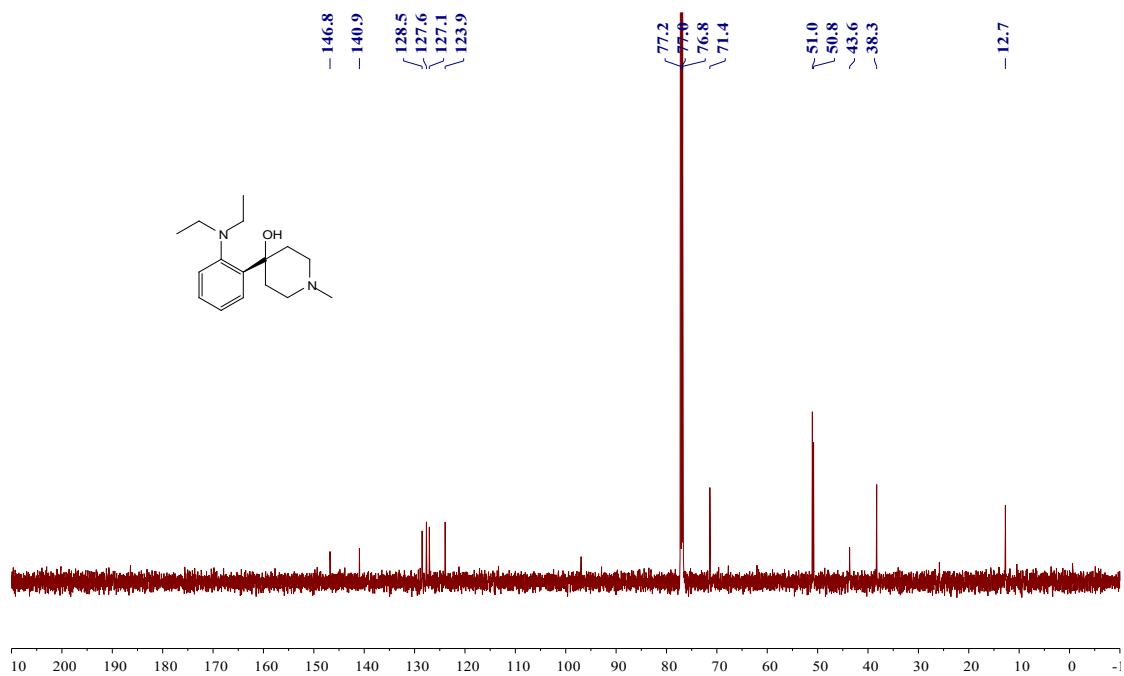
¹H and ¹³C NMR spectra of compound 3ap



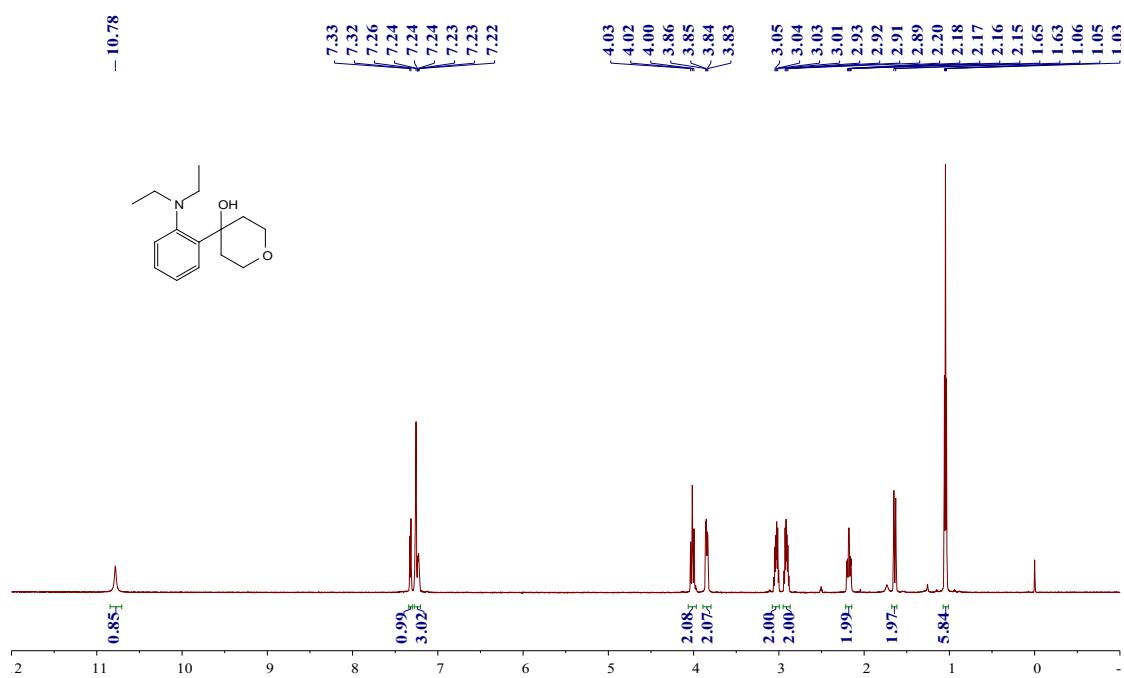


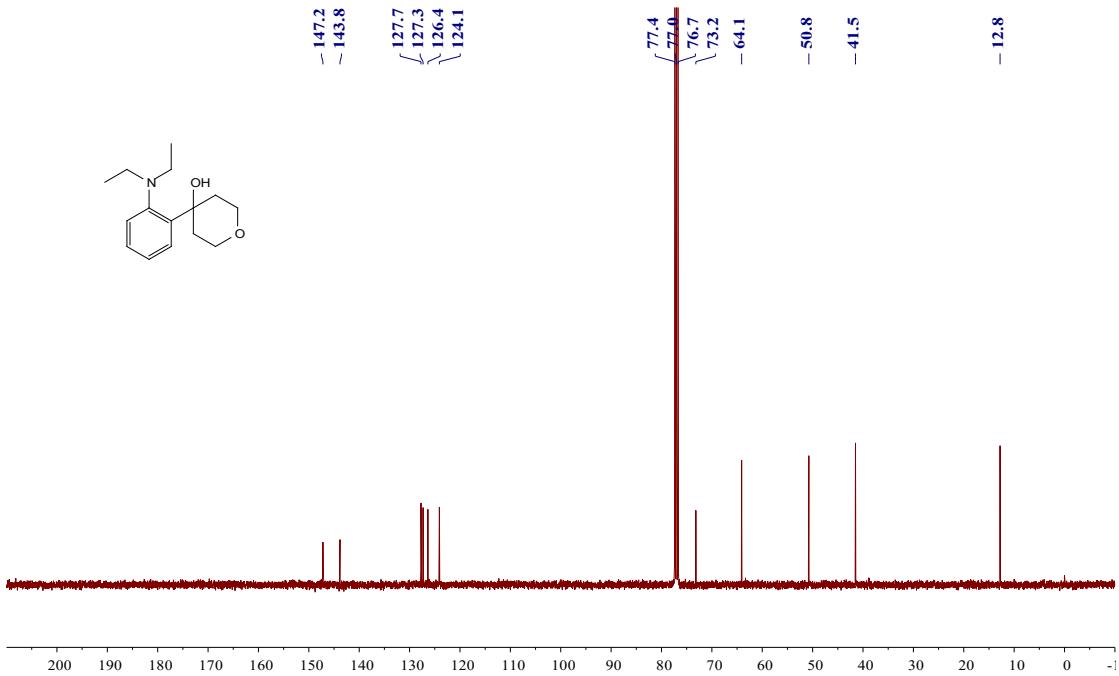
^1H and ^{13}C NMR spectra of compound **3aq**



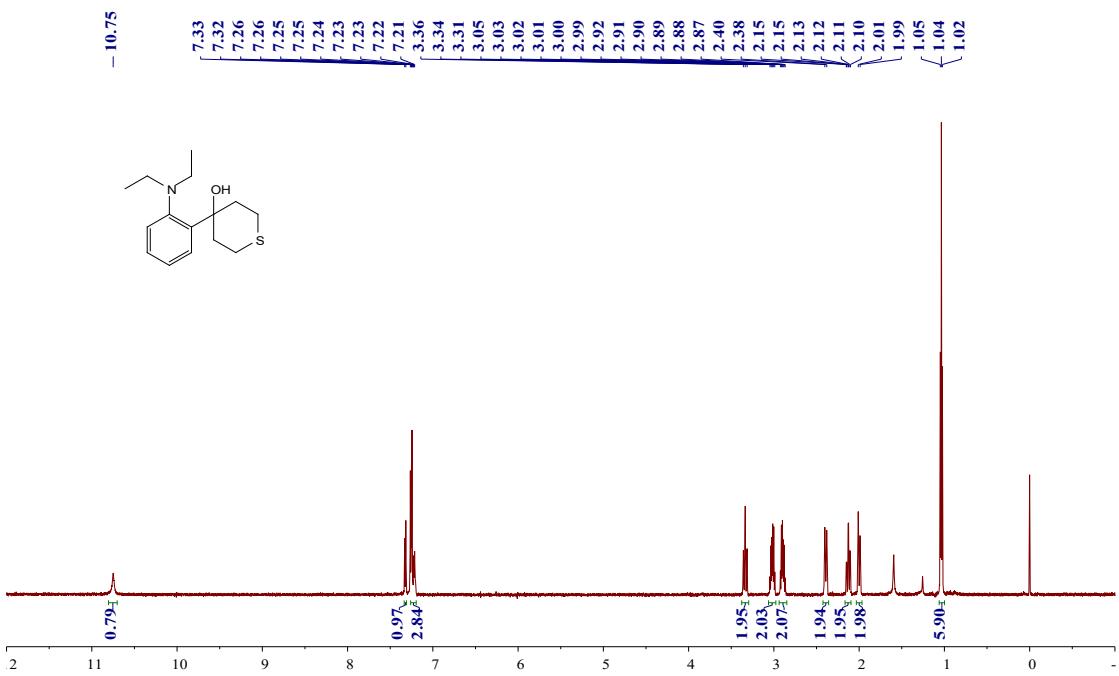


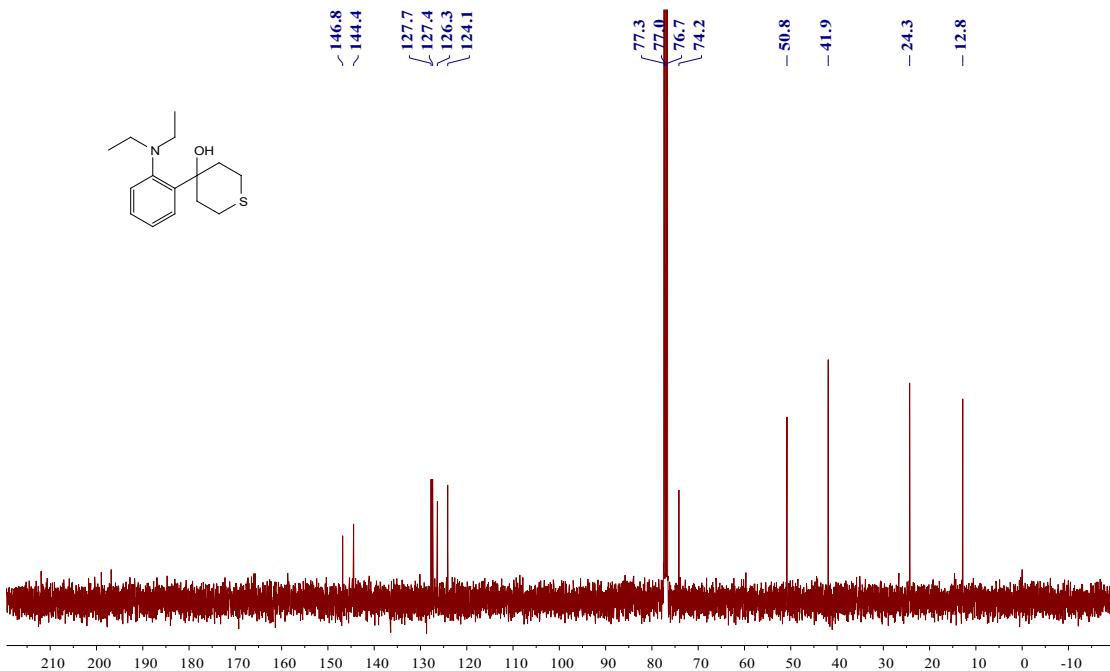
^1H and ^{13}C NMR spectra of compound **3ar**



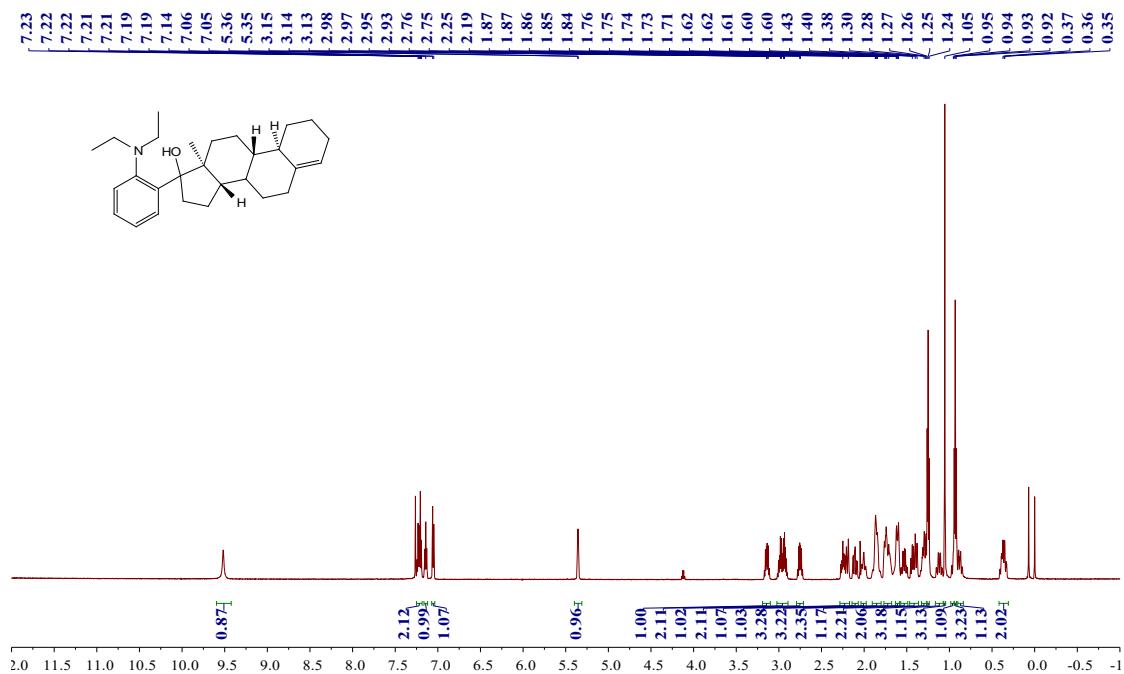


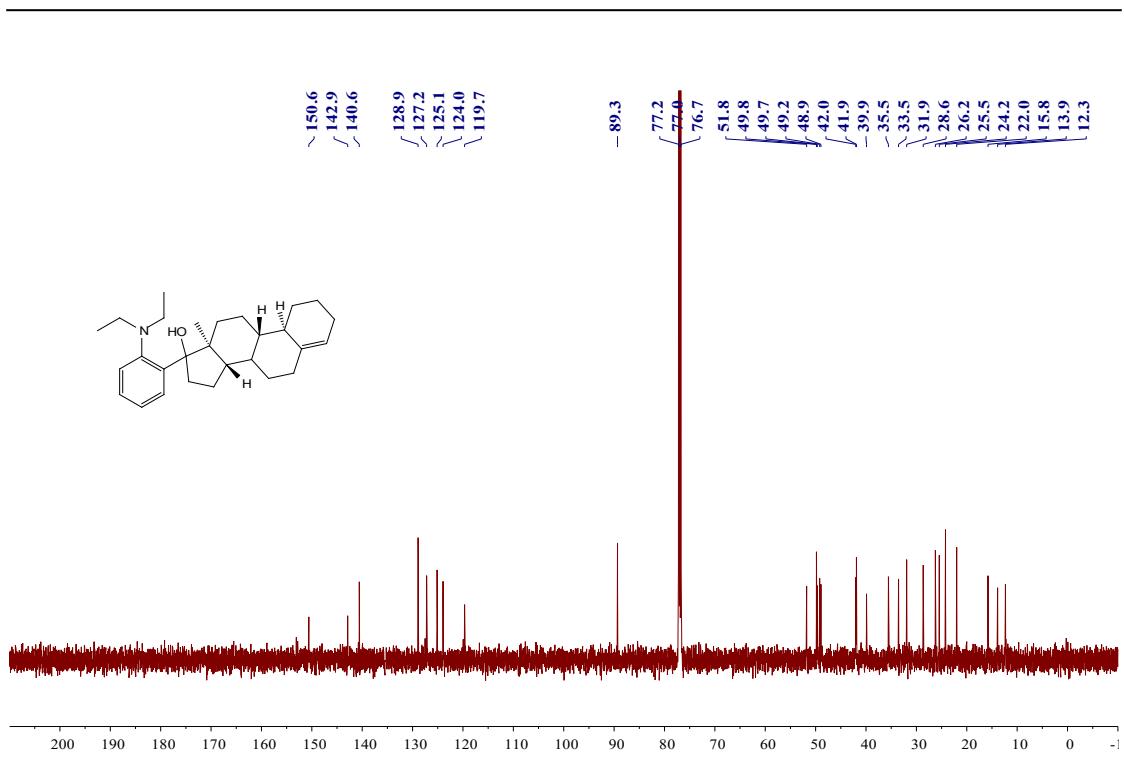
¹H and ¹³C NMR spectra of compound 3as



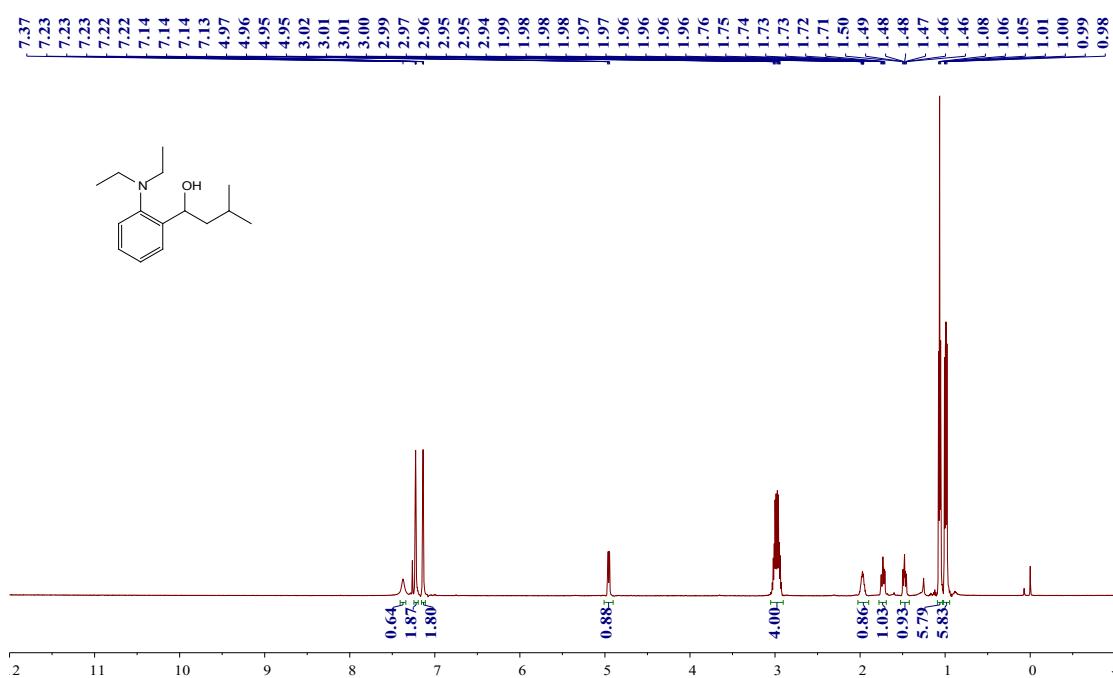


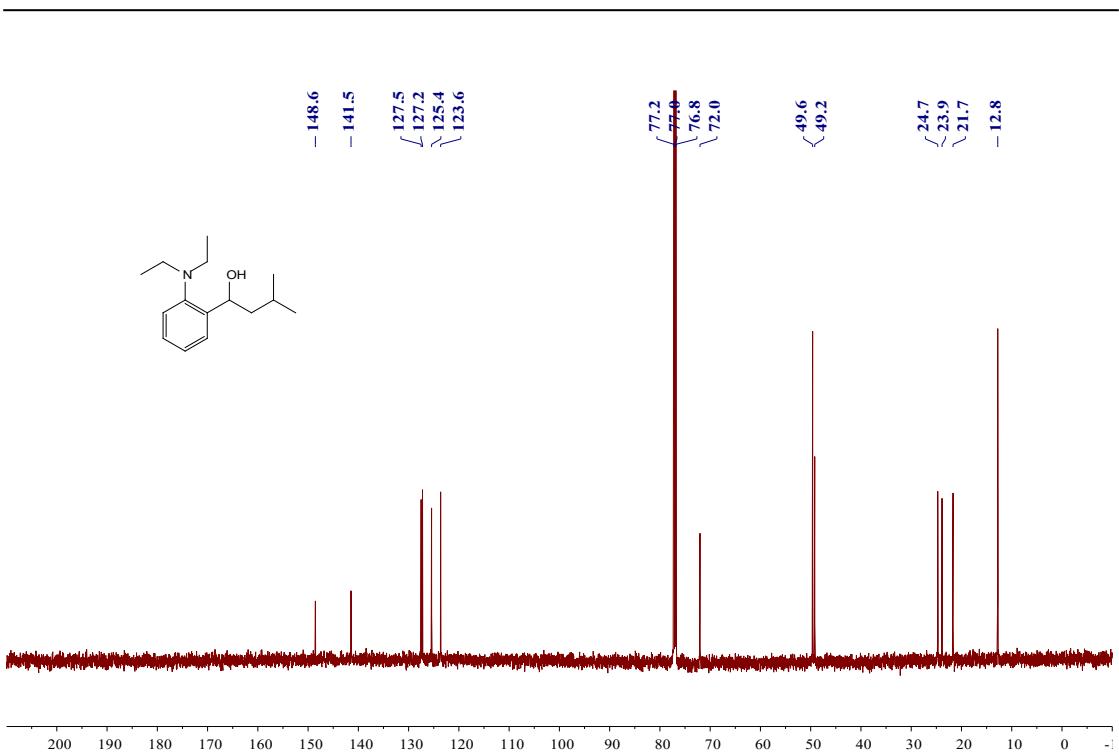
¹H and ¹³C NMR spectra of compound 3at



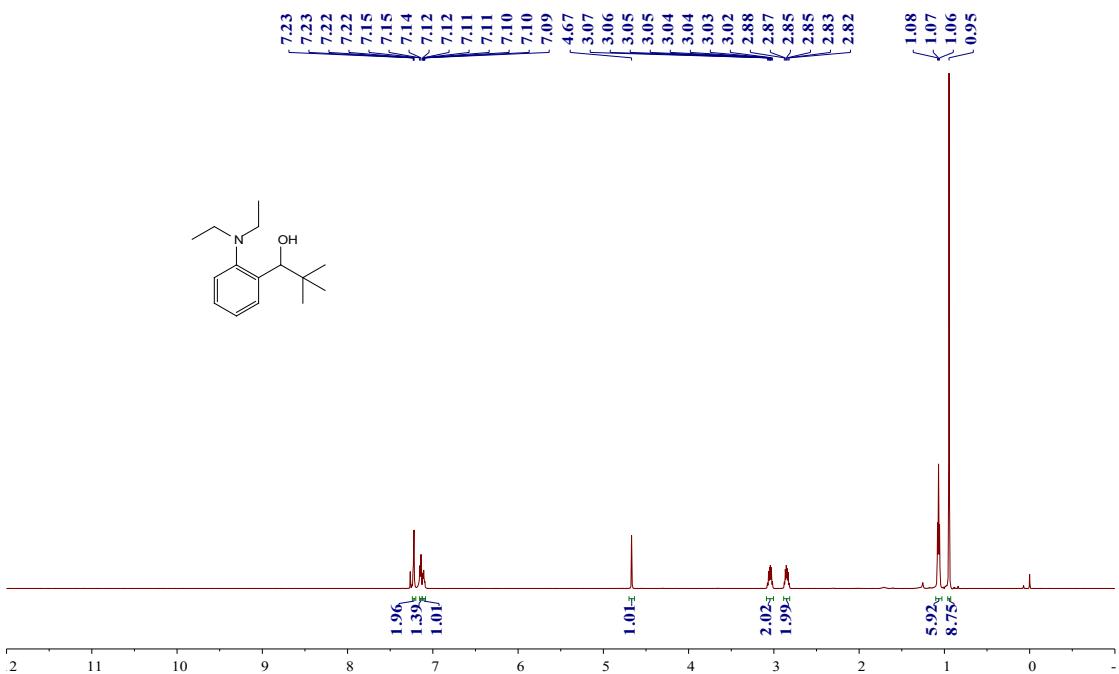


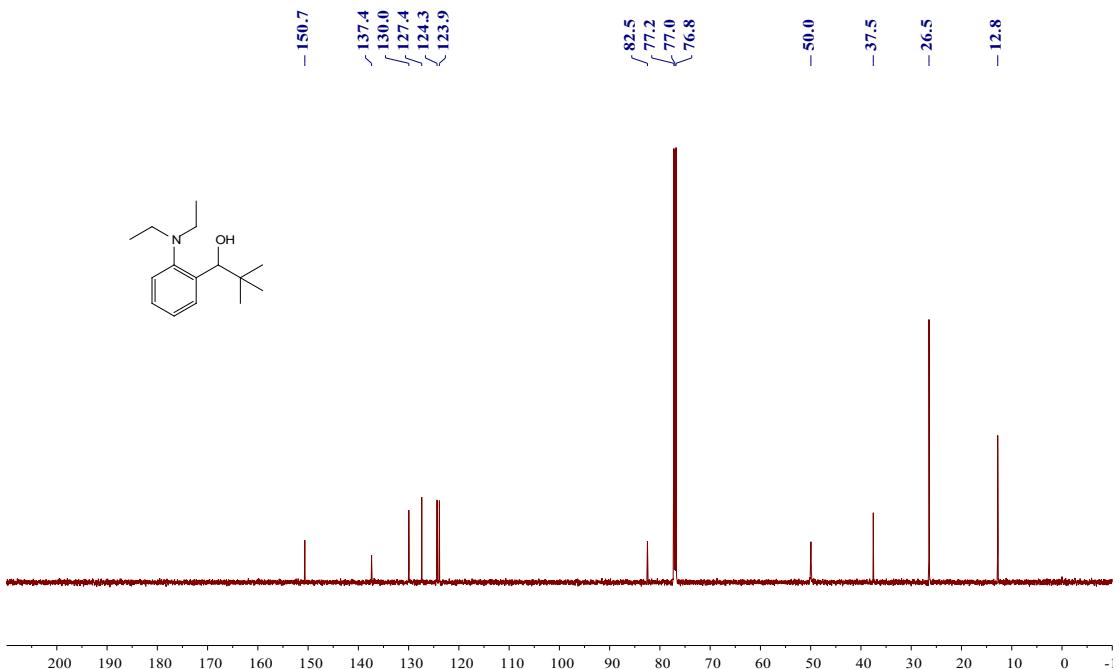
¹H and ¹³C NMR spectra of compound 3au



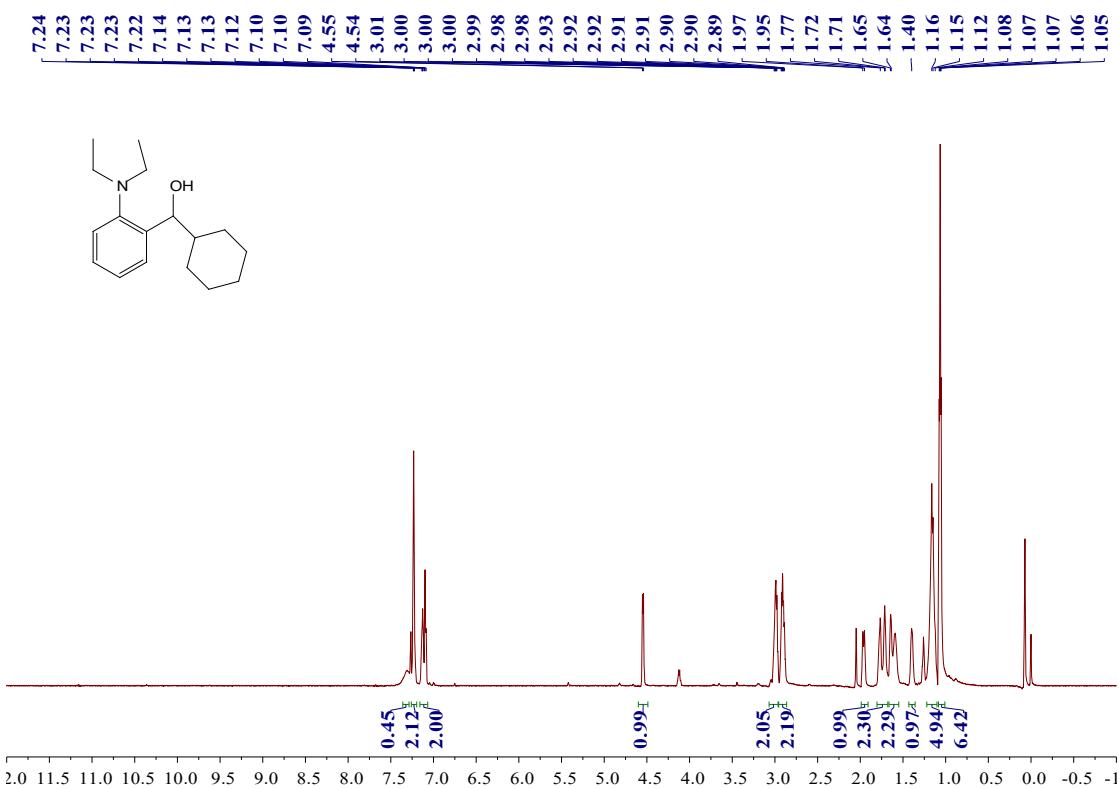


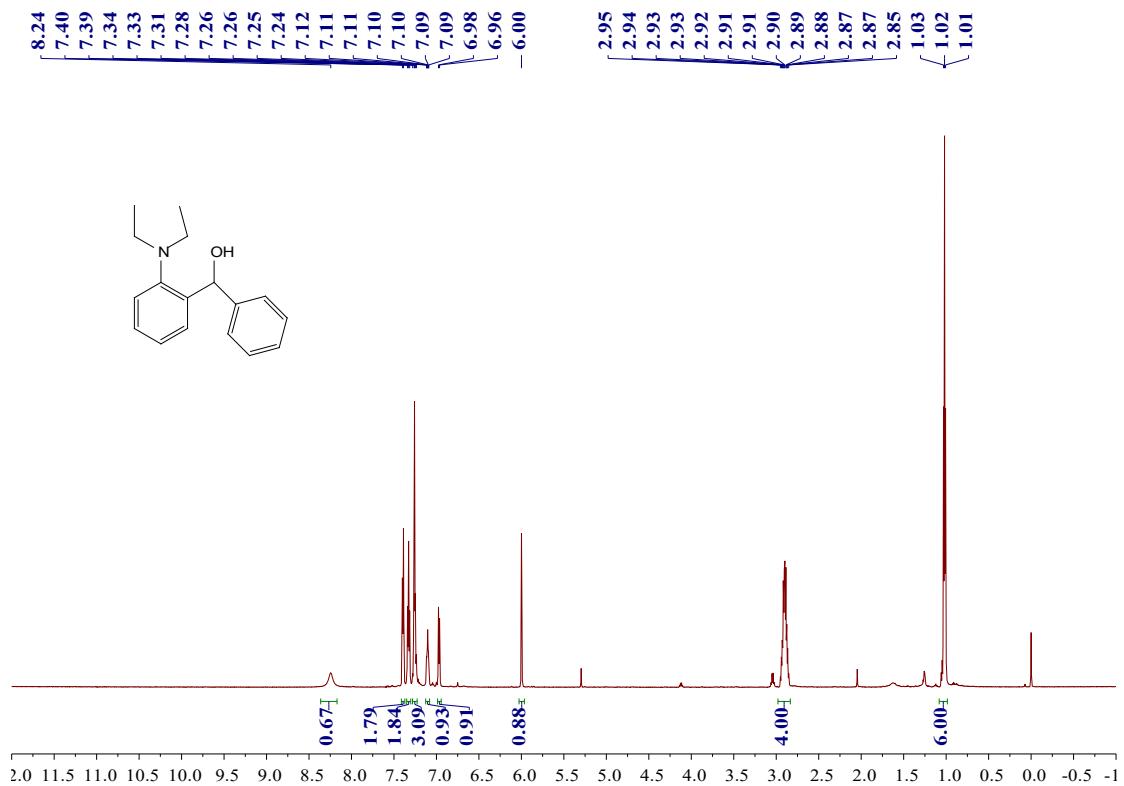
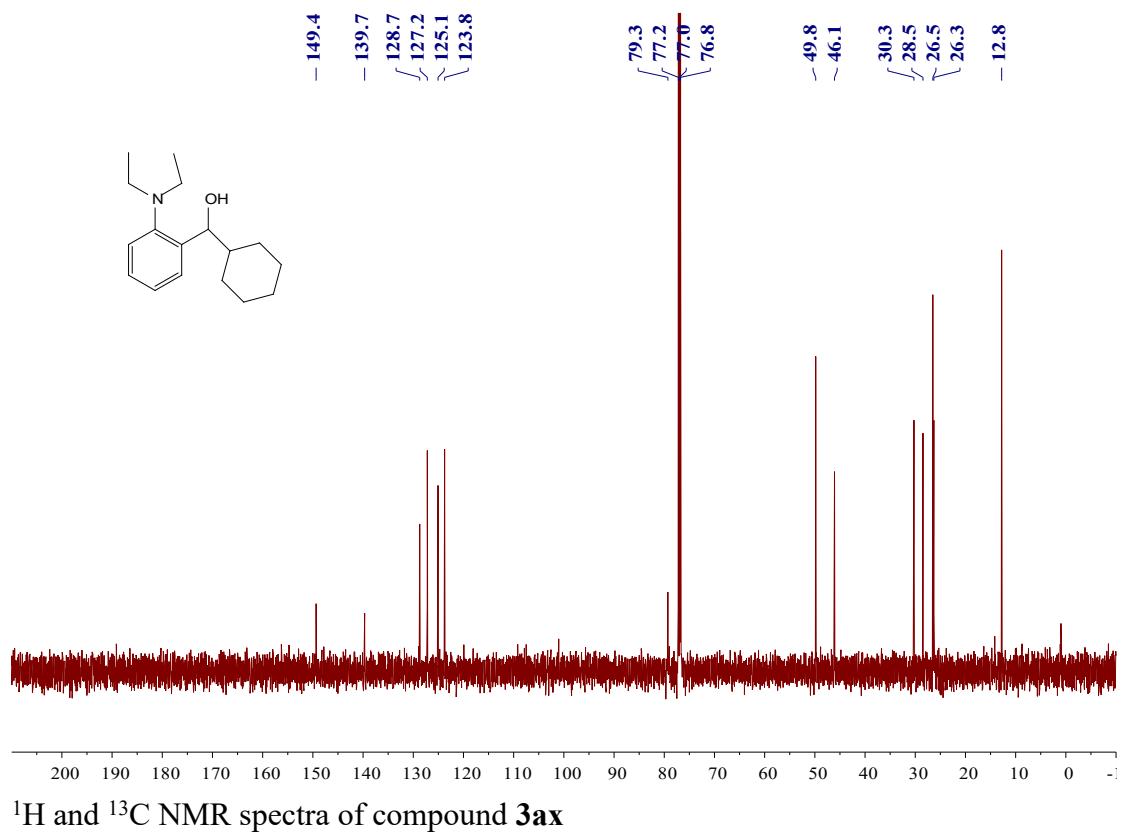
¹H and ¹³C NMR spectra of compound 3av

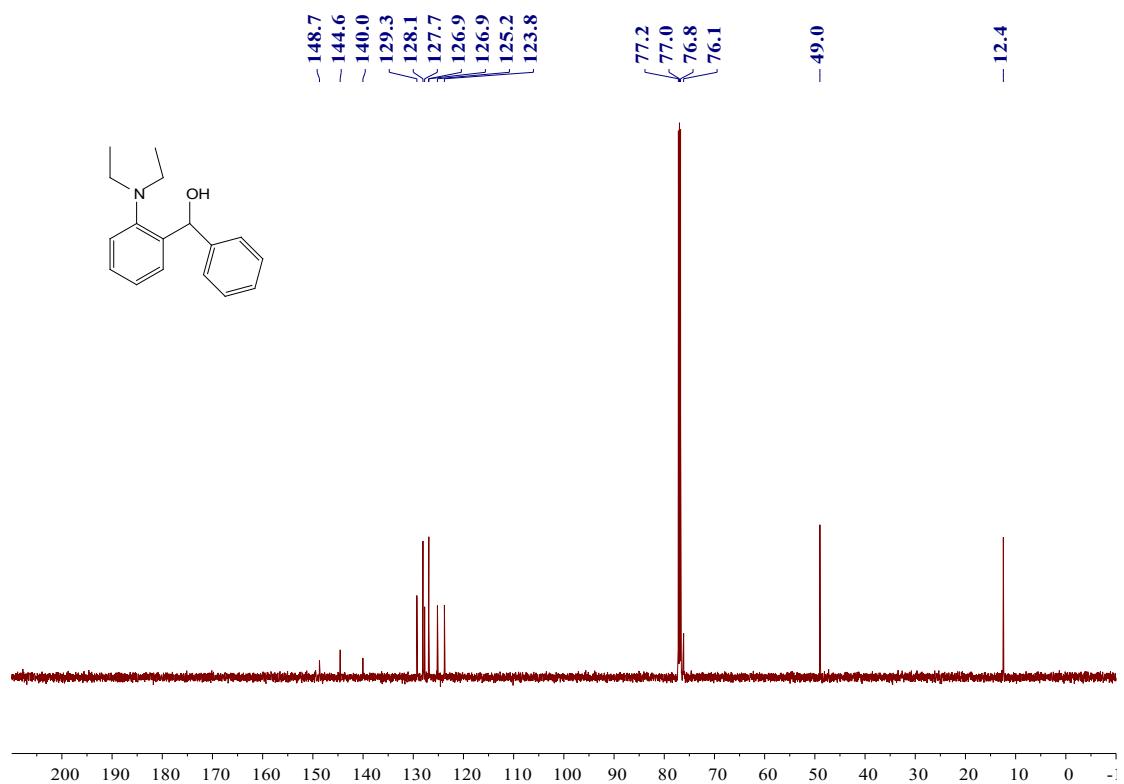




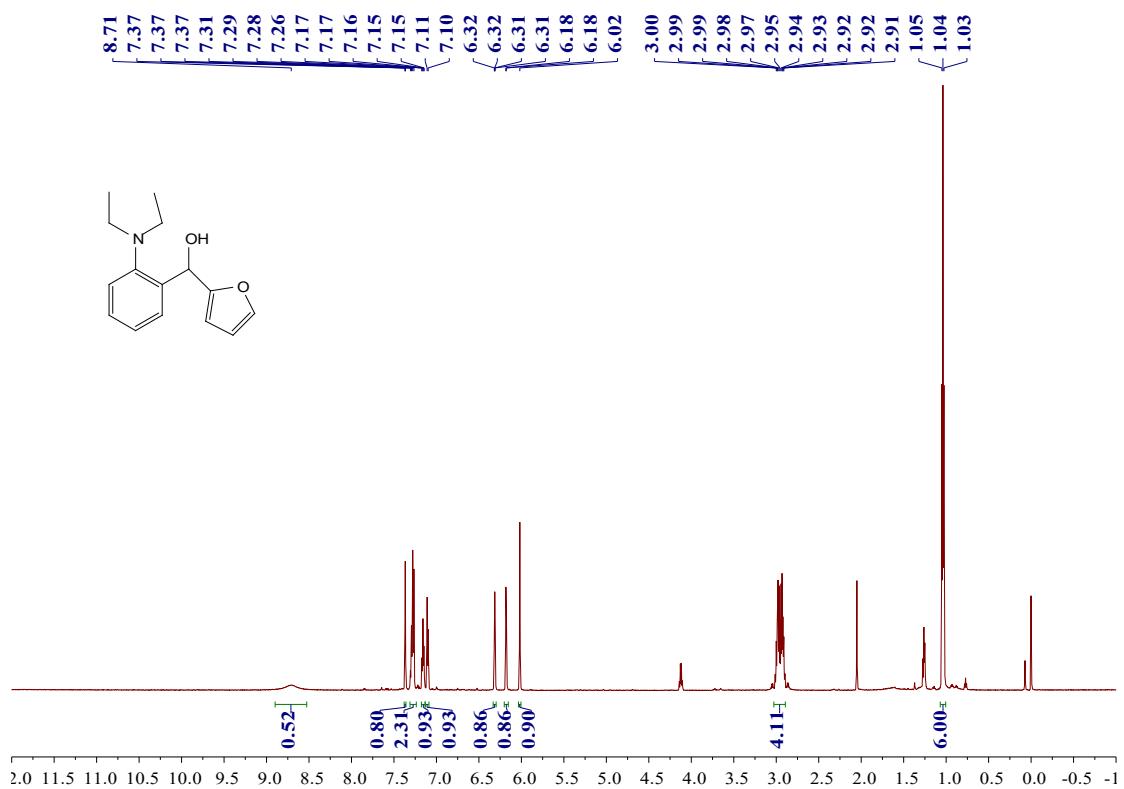
¹H and ¹³C NMR spectra of compound 3aw

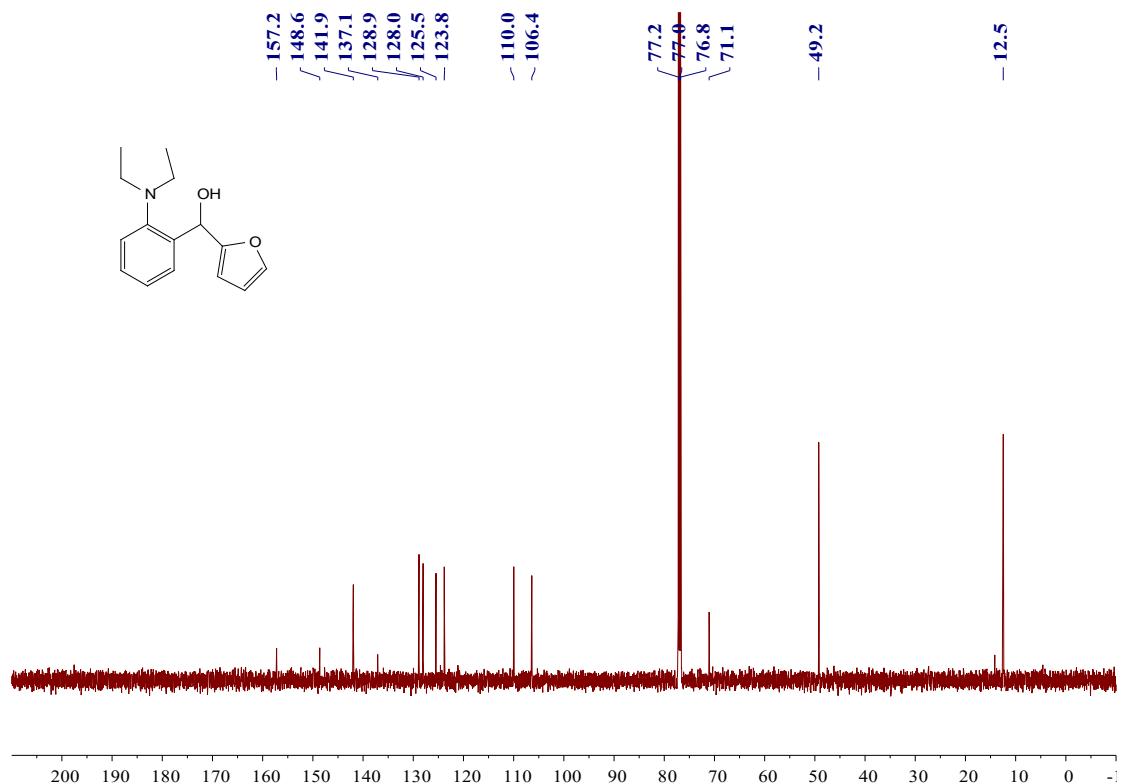




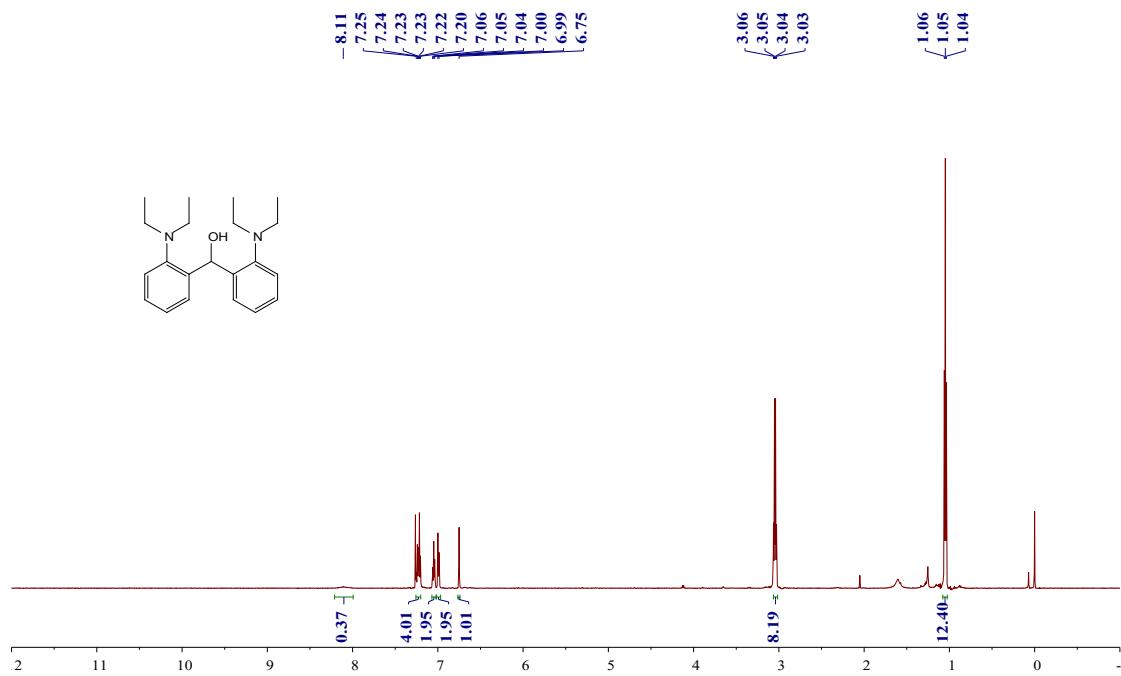


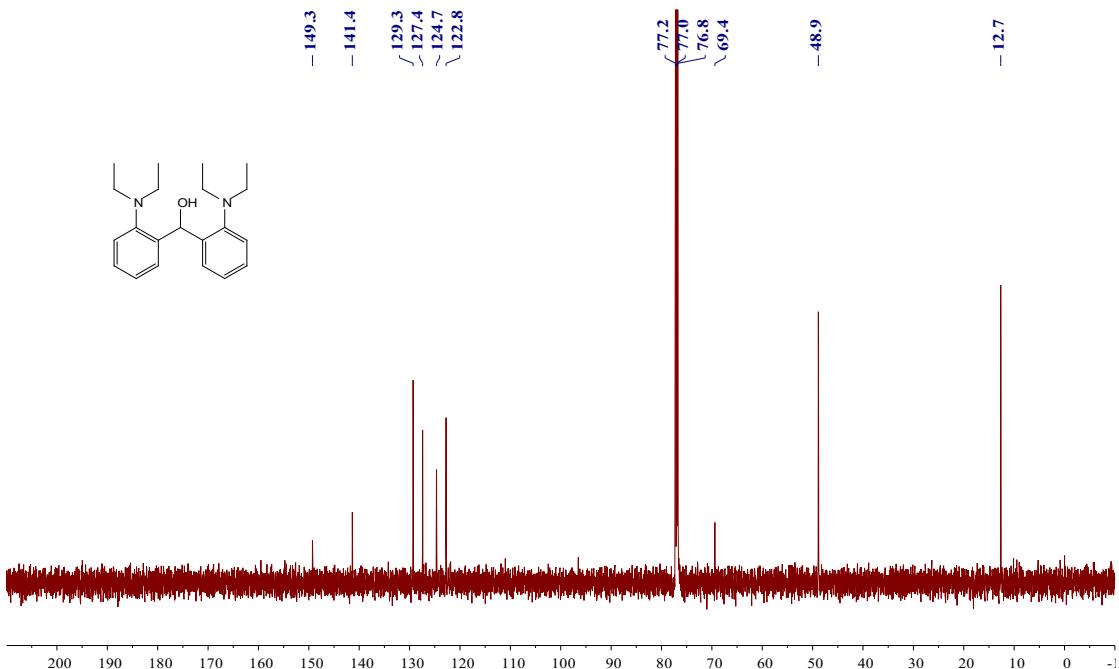
^1H and ^{13}C NMR spectra of compound 3ay



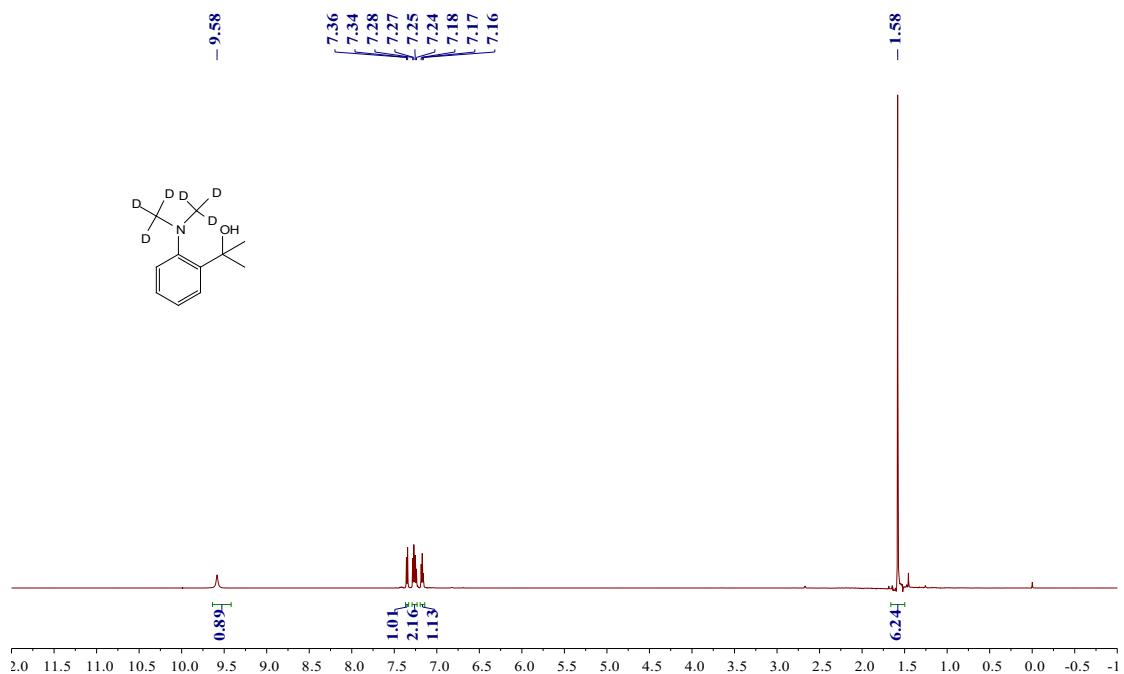


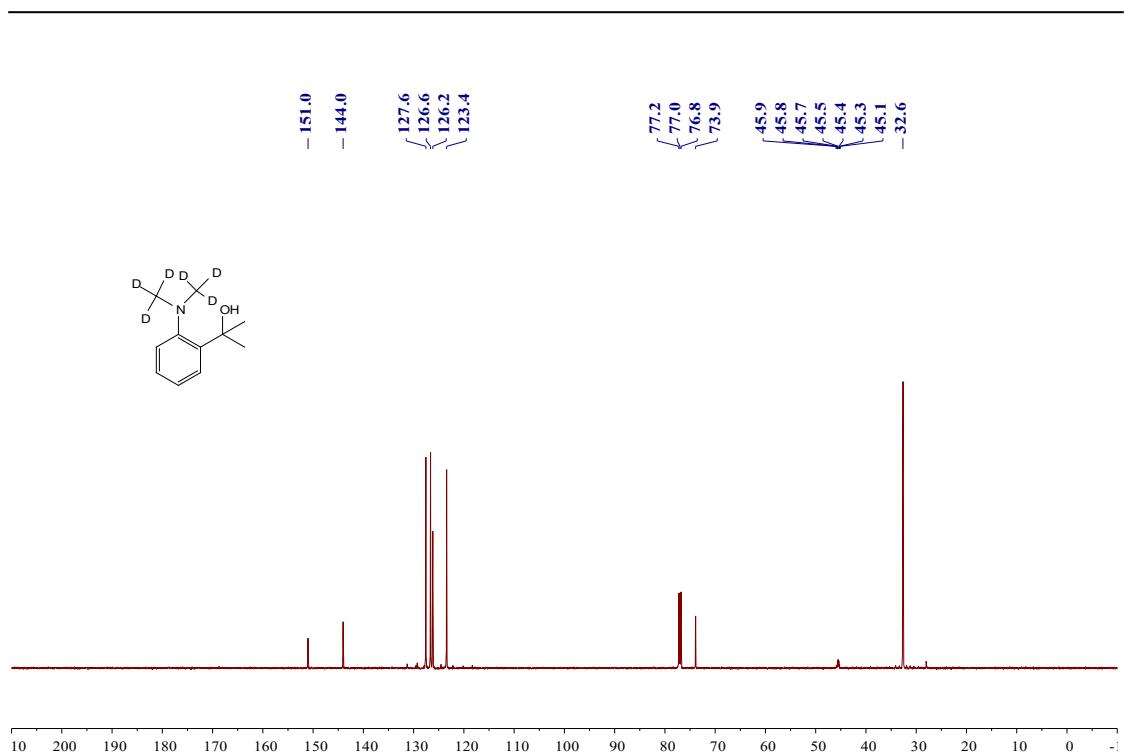
¹H and ¹³C NMR spectra of compound **bis(2-(diethylamino)phenyl)methanol (8)**



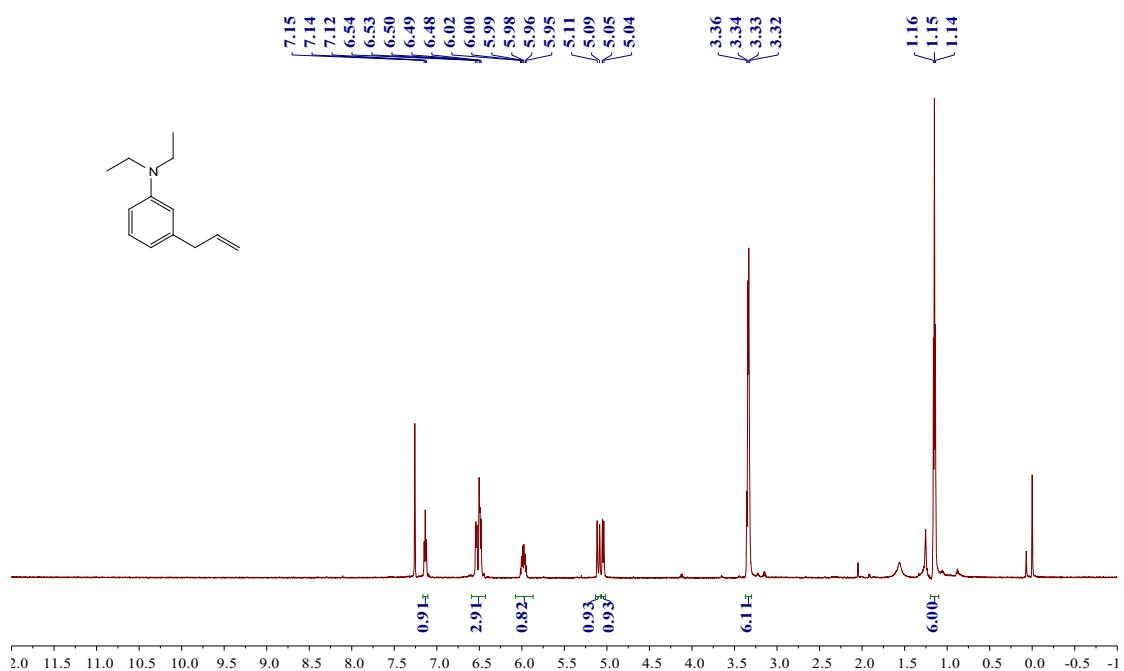


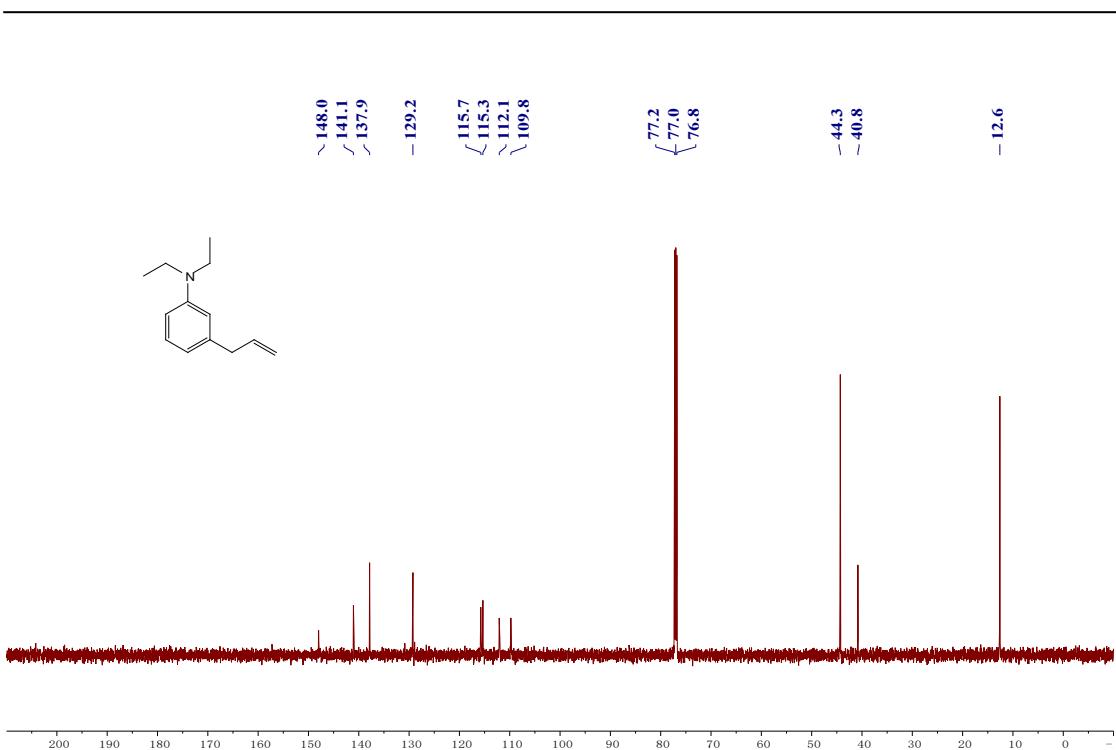
¹H and ¹³C NMR spectra of compound **3b'a**





¹H and ¹³C NMR spectra of compound **5**





¹H and ¹³C NMR spectra of compound 4a'

