Electronic Supplementary Material (ESI) for Dalton Transactions.

Contents

1. Additional Figures

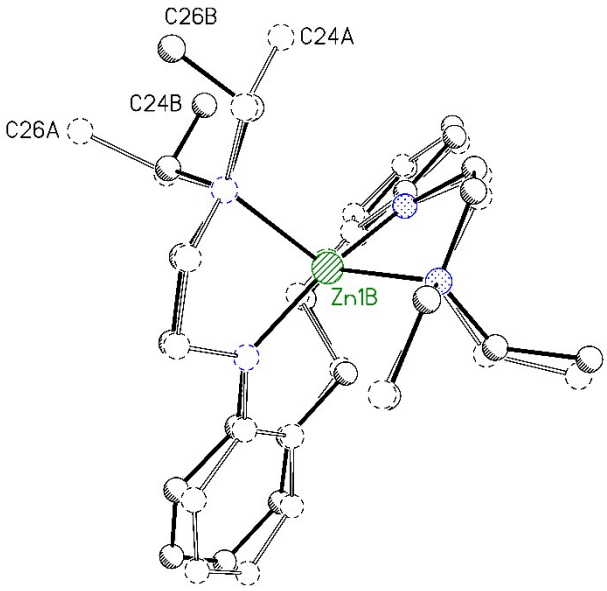

Figure S1 Fit of the two symmetry-independent molecules of 1nZn as observed by X-ray diffraction analysis.

2. General

All inorganic preparations were performed under an inert atmosphere of dinitrogen by means of standard Schlenk-line or glovebox techniques (Mb 200G). Traces of oxygen and moisture were removed from the inert gas by passing it over a BASF R 3-11 ($CuO/MgSiO_3$) catalyst, through concentrated sulfuric acid, over coarsely granulated silica gel, and finally through P_4O_{10}. Dichloromethane, diethyl ether, and *n*-pentane were freshly collected from a solvent purification system by M. Braun (MB SPS- 800). D_6-Benzene and toluene were used as p.a. grade and were distilled from Na/benzophenone prior to use. $CDCl_3$ was dried by distillation from calcium hydride.

2-chloro-N-methylethan-1-amine hydrochloride, 2-chloroacetyl chloride, 2,2´-ethylenedianiline, trimethylamine, diisobutylamine, diethyl oxalate, dimethyl succinate, 2,6-dimethylaniline, 2,6-diisopropylaniline, isophthalaldehyde, N,N-diethylethylendiamine, N,N-diisopropylethylendiamine, diethylamine, piperidine, lithium aluminum hydride, sodium borohydride, 1,2-dibromoethane, 1,3-dibromopropane, potassium carbonate, m-xylylene dichloride, $Zn(Et)_2$ (1.0 M in hexanes), $AlMe_3$ (2.0 M in toluene) and $Sn(HMDS)_2$ were purchased from Sigma- Aldrich, whereas $Mg(HMDS)_2$[1] and $AlH_3{\cdot}NMe_3$[2] were prepared according to literature known procedures.

Characterization. The NMR spectra were recorded with a Bruker Avance 300 and 400 spectrometers (T = 300 K) with δ referenced to external tetramethylsilane (^1H, ^{13}C). ^1H and ^{13}C NMR spectra were calibrated by using the solvent residual peak ($CDCl_3$: δ(^1H) = 7.26), (C_6D_6: δ(^1H) = 7.16)or (THF-d_8: δ(^1H) = 1.72 and 3.58) and the solvent peak ($CDCl_3$: δ(^{13}C) = 77.16), (C_6D_6: δ(^{13}C) = 128.06) or (THF-d_8: δ(^{13}C) = 67.2 and 25.3), respectively. Mass spectrometric data were measured with a Waters LCT Micromass spectrometer. IR spectra were recorded with a Bruker ALPHA spectrometer equipped with a diamond ATR unit. Elemental analysis was performed with a Vario MICRO cube (Elementar Analysensysteme GmbH); the presence of residual solvent molecules was verified by ^1H NMR spectroscopy.

3. Protio-ligand syntheses

Synthesis of 2-chloro-N-methylethan-1-amine hydrochloride:

2-chloro-N-methylethan-1-amine hydrochloride was prepared according to the literature procedure of Hirokawa *et al.*[3].

Synthesis of N^1-(2,6-dimethylphenyl)-N^2-methylethane-1,2-diamine:

2-chloro-N-methylethan-1-amine hydrochloride (2.60 g, 20.0 mmol) and 2,6-dimethylaniline (7.40 mL, 60.0 mmol) were mixed in a closed reaction vessel and heated to 130 °C overnight before water (120 mL), EtOAc (20 mL) and an aqueous solution of NaOH (2M, 10 mL) were added to the reaction mixture. The organic phase was separated, the solvent was removed in vacuum and the resulting residue was dissolved in pentane (100 mL) followed by addition of acetic acid (4 mL) and water (50 mL). The phases were again separated, whereas the aqueous phase was further treated with NaOH (10 g), stirred and extracted with Et_2O. Afterwards, the combined organic layers were dried over $MgSO_4$ followed by solvent removal to obtain 2.16 g (76 %) of N^1-(2,6-dimethylphenyl)-N^2-methylethane-1,2-diamine as an orange viscous liquid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 6.99 (d, J=7.4 Hz, 2H, arom. meta H), 6.81 (t, J=7.4 Hz, 1H, arom. para H), 3.08 (t, J=5.7 Hz, 2H, NHC*H*$_2$CH$_2$NHCH$_3$), 2.80 (t, J= 5.7 Hz, 2H, NHCH$_2$C*H*$_2$NHCH$_3$), 2.48 (s, 3H, NC*H*$_3$), 2.31 (s, 6H, PhC*H*$_3$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 146.5 (*i*-C_6H_3), 129.5 (*o*-C_6H_3), 128.9 (*m*-C_6H_3), 121.8 (*p*-C_6H_3), 52.2 (NHCH$_2$*C*H$_2$NHCH$_3$), 47.8 (NH*C*H$_2$CH$_2$NHCH$_3$), 36.4 (N*C*H$_3$), 18.7 (Ph*C*H$_3$).

HRMS (Methode) [m/z]: 179.156 (ES+)

Synthesis of 2,2'-(piperazine-1,4-diyl)bis(N-(2,6-dimethylphenyl)acetamide):

2,2'-(piperazine-1,4-diyl)bis(N-(2,6-dimethylphenyl)acetamide) was prepared according to the literature procedure of Aalla *et al.*[4].

Synthesis of N^1-(2,6-diisopropylphenyl)-N^2-methylethane-1,2-diamine:

2-chloro-N-methylethan-1-amine hydrochloride (2.60 g, 20.0 mmol) and 2,6-diisopropylaniline (10.6 g, 60.0 mmol) were mixed in a closed reaction vessel and heated to 130 °C overnight before EtOAc (100 mL) and an aqueous solution of NaOH (2M, 100 mL) were added to the reaction mixture. The organic phase was separated, the solvent was removed in vacuum and the resulting residue was dissolved in pentane (100 mL) followed by addition of acetic acid (5 mL) and water (100 mL). The phases were again separated, whereas the aqueous phase was further treated with NaOH (10 g), stirred and extracted with EtOAc (50 mL). Afterwards, the combined organic layers were dried over Na_2SO_4 followed by solvent removal to obtain 3.01 g (64 %) of N^1-(2,6-diisopropylphenyl)-N^2-methylethane-1,2-diamine as a red liquid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 7.28-7.19 (m, 3H, arom. H), 3.51 (sept, J= 6.8Hz, 2H, $CH(CH_3)_2$), 3.15 (t, J= 6.0 Hz, 2H, $CH_3NHCH_2CH_2$), 2.99 (t, J= 6.0 Hz, 2H, $CH_3NHCH_2CH_2$), 2.65 (s, 3H, NCH_3), 1.42 (d, J=6.8Hz, 12H, $CH(CH_3)_2$.

$^{13}C\{^{1}H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 143.4 (*i*-C_6H_3), 142.4 (*o*-C_6H_3), 123.6 (*m*-C_6H_3), 123.4 (*p*-C_6H_3), 52.2 ($CH_3NHCH_2CH_2$), 51.0 ($CH_3NHCH_2CH_2$), 36.4 (NCH_3), 27.5 ($CH(CH_3)_2$), 24.3 ($CH(CH_3)_2$).

HRMS (Methode) [m/z]: 235.218 (ES+)

Synthesis of Bis[diethylaminoethyl]-N,N'-m-xylylendiimine:

Isophthalaldehyde (2.01 g, 15.0 mmol) was added to a stirred solution of N,N-diethylethylendiamine (3.50 g, 30.0 mmol) in EtOH (15 mL) and the solution was stirred overnight. Solvent removal and drying of the product under vacuum gives 4.95 g (99 %) of bis[diethylaminoethyl]-N,N'-m-xylylendiimine as an yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 8.26 (s, 2H, C*H*PhC*H*), 7.96 (s, 1H, 1), 7.73 (d, J=7.7 Hz, 2H, *3*), 7.37 (t, J=7.7 Hz, 1H, *4*), 3.66 (t, J=7.1 Hz, 4H, NCH_2CH_2), 2.72 (t, J=7.1 Hz, 4H, NCH_2CH_2), 2.54 (q, J=7.3 Hz, 8H, NCH_2CH_3), 0.98 (t, J=7.3 Hz, 12H, NCH_2CH_3).

$^{13}C\{^{1}H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 161.2 (*C*HPh*C*H), 136.7 (2), 129.7 (3), 128.8 (4), 128.0 (1), 59.8 (NCH_2CH_2), 53.4 (NCH_2CH_2), 47.5 (NCH_2CH_3), 11.9 (NCH_2CH_3).

HRMS (Methode) [m/z]: 331.286 (ES+)

Synthesis of Bis[diisopropylaminoethyl]-N,N'-m-xylylendiimine:

Isophthalaldehyde (2.01 g, 15.0 mmol) was added to a stirred solution of N,N-diisopropylethylendiamine (4.33 g, 30.0 mmol) in EtOH (15 mL) and the solution was stirred overnight. Solvent removal and drying of the product under vacuum gives 5.80 g (99 %) of bis[diisopropylaminoethyl]-N,N'-m-xylylendiimine as an yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 8.24 (s, 2H,C*H*PhC*H*), 7.99 (t, J=1.6 Hz, 2H, *1*), 7.76 (dd, J=1.6 Hz, J=7.6 Hz, 2H, *3*), 7.41 (t, J=7.6 Hz, 1H, *4*), 3.60 (t, J=7.2 Hz, 4H, $NHCH_2CH_2$), 2.99 (sept, J= 6.62, 4H, $CH(CH_3)_2$), 2.70 (t, J=7.2 Hz, 4H, $NHCH_2CH_2$), 0.98 (d, J=6.62 Hz, 24H, $CH(CH_3)_2$).

$^{13}C\{^{1}H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 161.0 (*C*HPh*C*H), 136.8 (2), 129.7 (3), 128.9 (4), 128.1 (1), 63.5 ($NHCH_2CH_2$), 49.0 ($CH(CH_3)_2$),46.2 ($NHCH_2CH_2$), 30.0 ($CH(CH_3)_2$).

HRMS (Methode) [m/z]: 387.348 (ES+)

Synthesis of N,N'-(1,3-phenylene)bis(2-chloroacetamide):

N,N'-(1,3-phenylene)bis(2-chloroacetamide) was prepared according to the literature procedure of Fyles *et al.*[5].

Synthesis of N,N'-(1,3-phenylene)bis(2-(diethylamino)acetamide):

Diethylamine (14.0 g, 200 mmol) was added to a stirred solution of N,N'-(1,3-phenylene)bis(2-chloroacetamide) (13.0 g, 50.0 mmol) in EtOH (120 mL) and heated to 80 °C for 15 hours. The mixture was allowed to cool to room temperature followed by solvent removal and addition of saturated $NaHCO_3$ solution (75 mL) as well as water (50 mL). The aqueous solution was extracted with $CHCl_3$ (3x50 mL), dried over $MgSO_4$ followed by solvent removal to obtain 15.0 g (89 %) of N,N'-(1,3-phenylene)bis(2-(diethylamino)acetamide).

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 9.43 (s, 2H, NH), 7.85 (t, J = 2.0 Hz, 1H, 1), 7.40-7.36 (m, 2H, 3), 7.30-7.25 (m, 1H, 4), 3.14 (s, 4H, C*H*$_2$CO), 2.65 (q, J = 7.21 Hz, 8H, C*H*$_2$CH$_3$), 1.08 (t, J = 7.21 Hz, 12H, CH$_2$C*H*$_3$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 170.5 (*C*O), 138.4 (2), 129.7 (4), 115.0 (3), 110.1 (1) , 58.0 (CO*C*H$_2$), 49.0 (*C*H$_2$CH$_3$), 12.4 (CH$_2$*C*H$_3$).

HRMS (Methode) [m/z]: 335.24 (ES+)

Synthesis of N,N'-(1,3-phenylene)bis(2-(piperidin-1-yl)acetamide):

Piperidine (7.80 g, 92.0 mmol) was added to a stirred solution of N,N'-(1,3-phenylene)bis(2-chloroacetamide) (6.00 g, 23.0 mmol) in EtOH (60 mL) and heated to 80 °C for 24 hours. The mixture was allowed to cool to room temperature followed by solvent removal and addition of saturated $NaHCO_3$ solution (30 mL). The aqueous solution was extracted with $CHCl_3$ (3x25 mL), dried over $MgSO_4$ followed by solvent removal to obtain 6.30 g (76 %) of N,N'-(1,3-phenylene)bis(2-(piperidin-1-yl)acetamide) as a brown solid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 9.26 (s, 2H, NH), 7.84 (t, J = 2.0 Hz, 1H, 4-C*H*$_{arom}$), 7.38-7.34 (m, 2H, 3-C*H*$_{arom}$), 7.30-7.24 (m, 1H, 1-C*H*$_{arom}$), 3.05 (s, 4H, COC*H*$_2$), 2.52 (m, 8H, α-pip), 1.63 (m, 8H, β-pip), 1.50-1.45 (m, 4H, γ-pip).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 169.3 (*C*O), 138.4 (2-C*H*$_{arom}$), 129.7 (4-C*H*$_{arom}$), 115.2 (3-*C*H$_{arom}$), 110.4 (1-C*H*$_{arom}$), 63.0 (CO*C*H$_2$), 55.0 (α-pip), 26.3 (β-pip), 23.7 (γ-pip).

Synthesis of N,N'-(1,2-phenylene)bis(2-chloroacetamide):

N,N'-(1,2-phenylene)bis(2-chloroacetamide) was prepared according to the literature procedure of Beer *et al.*[6].

Synthesis of N,N'-(1,2-phenylene)bis(2-(diethylamino)acetamide):

N,N'-(1,2-phenylene)bis(2-(diethylamino)acetamide) was prepared according to the literature procedure of Singh *et al.*[7].

Synthesis of N,N'-(1,2-phenylene)bis(2-(piperidin-1-yl)acetamide):

Piperidine (8.50 g, 100 mmol) was added to a stirred solution of N,N'-(1,2-phenylene)bis(2-chloroacetamide) (6.50 g, 25.0 mmol) in EtOH (60 mL) and heated to 80 °C for 15 hours. The mixture was allowed to cool to room temperature followed by solvent removal and addition of saturated $NaHCO_3$ solution (38 mL) as well as water (25 mL). The aqueous solution was extracted with $CHCl_3$ (3x25 mL) and dried over $MgSO_4$. After solvent removal the crude product was recrystallized from EtOH to obtain 4.80 g (54 %) of N,N'-(1,2-phenylene)bis(2-(piperidin-1-yl)acetamide) as white needles.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 9.30 (s, 2H, N*H*), 7.60-7.56 (m, 2H, ortho-*H*), 7.21-7.17 (m, 2H, meta-*H*), 3.12 (s, 4H, CH_2CO), 2.53 (t, J = 5.0 Hz, 8H, α-pip), 1.67-1.59 (m, 8H, β-pip), 1.49-1.46 (m, 4H, γ-pip).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 169.9 (*C*O), 130.2 (ipso-*C*), 126.2 (meta-*C*), 124.9 (ortho-*C*), 62.8 (*C*H_2CO), 55.2 (α-pip), 26.2 (β-pip), 23.7 (γ-pip).

HRMS (Methode) [m/z]: 359.2 (ES+)

Synthesis of N,N'-(pyridine-2,6-diyl)bis(2-chloroacetamide):

N,N'-(pyridine-2,6-diyl)bis(2-chloroacetamide) was prepared according to the literature procedure of Wang *et al.*[8].

Synthesis of N,N'-(pyridine-2,6-diyl)bis(2-(diethylamino)acetamide):

Diethylamine (8.70 g, 120 mmol) was added to a stirred solution of N,N'-(pyridine-2,6-diyl)bis(2-chloroacetamide) (7.40 g, 28.0 mmol) in EtOH (90 mL) and heated to 80 °C for 24 hours. The mixture was allowed to cool to room temperature followed by solvent removal and addition of saturated $NaHCO_3$ solution (45 mL) as well as water (25 mL). The aqueous solution was extracted with $CHCl_3$ (3x30 mL), dried over $MgSO_4$ followed by solvent removal to obtain 9.30 g (99 %) of N,N'-(pyridine-2,6-diyl)bis(2-(diethylamino)acetamide) as a black solid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 9.56 (br, 2H, N*H*), 7.91 (d, J = 7.8 Hz, 2H, meta *H*), 7.65 (t, J = 7.8 Hz, 1H, para *H*), 3.12 (s, 4H, COCH_2), 2.62 (q, J = 7.2 Hz, 8H, CH_2CH_3), 1.06 (t, J = 7.2 Hz, CH_2CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 170.9 (*C*O), 149.4 (ortho *C*), 140.4 (para *C*), 109.2 (meta *C*), 58.1 (CO*C*H_2), 48.8 (*C*H_2CH_3), 12.3 (CH_2*C*H_3).

HRMS (Methode) [m/z]: 336.2 (ES+)

Synthesis of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-chloroacetamide):

2-chloroacetyl chloride (7.30 g, 65.0 mmol) was added to a stirred solution of 2,2´-ethylenedianiline (5.80 g, 26.0 mmol) and triethylamine (7.80 g, 78.0 mmol) in THF (150 mL) at 0 °C. The solution was allowed to warm to room temperature and was further stirred for 15 hours resulting in a grey precipitate. The precipitate was filtered off, washed with water, THF and again water to obtain 8.20 g (86 %) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-chloroacetamide).

^{1}H NMR (300 MHz, DMSO-d_6): *δ(ppm)* = 9.78 (s, 2H, N*H*), 7.38 (dd, J = 7.4 Hz, J = 1.5, 2H, arom *H*), 7.28 – 7.14 (m, 6H, arom *H*), 4.34 (s, 4H, COCH_2), 2.79 (s, 4H, CH_2CH_2).

^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6): *δ (ppm)* = 165.4 (CO), 136.0 (arom. C), 135.1 (arom. C), 129.7 (arom. C), 126.4 (arom. C), 126.1 (arom. C), 126.1 (arom. C), 43.2 (CH_2Cl), 31.3 (CH_2CH_2).

HRMS (Methode) [m/z]: 365.1 (ES+).

Synthesis of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diethylamino)acetamide):

Diethylamine (3.20 g, 44.0 mmol) was added to a stirred solution of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-chloroacetamide) (4.00 g, 11.0 mmol) in EtOH (33 mL) and heated to 80 °C for 24 hours. The mixture was allowed to cool to room temperature followed by solvent removal and addition of saturated $NaHCO_3$ solution (17 mL) as well as water (11 mL). The aqueous solution was extracted with $CHCl_3$ (3x11 mL), dried over $MgSO_4$ followed by solvent removal to obtain 4.70 g (97 %) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diethylamino)acetamide) as a black solid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 9.50 (br, 2H, N*H*), 8.07 (d, J = 7.8 Hz, 2H, C$H_{arom.}$), 7.28-7.22 (m, 2H, C$H_{arom.}$), 7.12-7.03 (m, 4H, CH_{arom}), 3.20 (s, 4H, COCH_2), 2.89 (s, 4H, CH_2CH_2), 2.65 (q, J = 6.9 Hz, 8H, C$H_2$$CH_3$), 1.04 (t, J = 7.2 Hz, 12H, CH_2CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 170.1 (*C*O), 135.4 (arom. *C*), 130.9 (arom. *C*), 129.3 (arom. *C*), 127.5 (arom. *C*), 124.8 (arom. *C*), 122.3 (arom. *C*), 58.2 (CO*C*H_2), 48.8 (*C*$H_2$$CH_3$), 31.7 (*C*$H_2$*C*$H_2$), 12.4 ($CH_2$*C*$H_3$).

HRMS (Methode) [m/z]: 439.3 (ES+)

Synthesis of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diisobutylamino)acetamide):

Diisobutylamine (3.35 g, 26.0 mmol) was added to a stirred solution of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-chloroacetamide) (2.40 g, 6.50 mmol) in DMSO (10 mL) and heated to 70 °C for three days. The mixture was allowed to cool to room temperature followed by addition of water (100 mL). The aqueous solution was extracted with DCM (100 mL), followed by washing of the organic phase with brine (50 mL). Drying over $MgSO_4$ and solvent removal gave 3.20 g (90 %) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diisobutylamino)acetamide).

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 8.99 (br, 2H, NH), 7.85 (d, J = 7.4 Hz, 2H, *1*), 7.26-7.19 (m, 2H, *2*), 7.08-7.07 (m, 4H, *3+4*), 3.16 (s, 4H, COCH_2), 2.90 (s, 4H, CH_2CH_2), 2.22 (d, J = 7.5 Hz, 8H, CH_2CH(CH_3)$_2$), 1.76 (nonett, 4H, CH_2C*H*(CH_3)$_2$), 0.84 (d, J = 6.5 Hz, 24H, CH_2CH(CH_3)$_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 170.3 (*C*O), 135.1 (arom. *C*), 132.5 (arom. *C*), 129.0 (arom. *C*), 127.2 (arom. *C*), 125.4 (arom. *C*), 123.7 (arom. *C*), 64.3 (*C*H_2C(CH_3)$_2$), 60.8 (CO*C*H_2), 30.7(*C*H_2*C*H_2), 26.3 (CH_2*C*(CH_3)$_2$), 21.1 (CH_2C(*C*H_3)$_2$).

HRMS (Methode) [m/z]: 551.4 (ES+)

Synthesis of N,N'-(oxybis(2,1-phenylene))bis(2-chloroacetamide):

N,N'-(oxybis(2,1-phenylene))bis(2-chloroacetamide) was prepared according to the literature procedure of Kocina *et al.*[9].

Synthesis of N,N'-(oxybis(2,1-phenylene))bis(2-(diethylamino)acetamide):

Diethylamine (3.00 g, 40.0 mmol) was added to a stirred solution of N,N'-(oxybis(2,1-phenylene))bis(2-chloroacetamide) (3.50 g, 10.0 mmol) in EtOH (30 mL) and heated to 80 °C for 15 hours. The mixture was allowed to cool to room temperature followed by solvent removal and addition of saturated $NaHCO_3$ solution (20 mL) as well as water (10 mL). The aqueous solution was extracted with DCM (2x15 mL), dried over $MgSO_4$ followed by solvent removal to obtain 3.70 g (86 %) of N,N'-(oxybis(2,1-phenylene))bis(2-(diethylamino)acetamide).

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 10.0 (br, 2H, N*H*), 8.51 (dd, J = 1,6 Hz, J = 8.2 Hz, 2H, *4*), 7.15 (dt, J = 1.3 Hz, J = 8.0 Hz, 2H, *2*), 7.01 (dt, J = 1.5 Hz, J = 8.2 Hz, 2H, *3*), 6.75 (dd, J = 1.8 Hz, J = 8.0 Hz, *1*), 3.12 (s, 4H, COCH_2), 2.49 (q, J = 7.2 Hz, 8H, CH_2CH$_3$), 0.90 (t, J = 7.2 Hz, CH$_2$CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 170.5 (CO), 145.3 (arom. *C*), 129.7 (arom. *C*), 124.8 (arom. *C*), 124.4 (arom. *C*), 120.8 (arom. *C*), 117.7 (arom. *C*), 58.4 (*C*H$_2$CH$_3$), 48.6 (CO*C*H$_2$), 12.4 (CH$_2$*C*H$_3$).

HRMS (Methode) [m/z]: 427.3 (ES+)

Synthesis of N^1,N^2-bis(2-(dimethylamino)ethyl)oxalamide:

N^1,N^2-bis(2-(dimethylamino)ethyl)oxalamide was prepared according to the literature procedure of Real *et al.*[10].

Synthesis of N^1,N^2-bis(2-(diethylamino)ethyl)oxalamide:

N,N-diethylethylenediamine (5.80 g, 50.0 mmol) and diethyl oxalate (2.90 g, 20.0 mmol) were mixed and stirred at 130 °C overnight. After drying at vacuum 5.60 g (98 %) of N^1,N^2-bis(2-(diethylamino)ethyl)oxalamide could be isolated as a pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 7.86 (br, 2H, NH), 3.33 (q, J = 6.0 Hz, 4H, NHCH_2CH$_2$), 2.57 (t, J = 6.0Hz, 4H, NHCH$_2$CH_2), 2.53 (q, J = 7.2 Hz, 8H, CH_2CH$_3$), 1.00 (t, J = 7.2 Hz, 12H, CH$_2$CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 159.9 (CO), 51.3 (NHCH$_2$*C*H$_2$), 47.0 (*C*H$_2$CH$_3$), 37.4 (NH*C*H$_2$CH$_2$), 12.0 (CH$_2$*C*H$_3$).

HRMS (Methode) [m/z]: 287.2 (ES+)

Synthesis of N^1,N^2-bis(2-(diisopropylamino)ethyl)oxalamide:

N,N-diisopropylethylenediamine (3.17 g, 22.0 mmol) and diethyl oxalate (1.46 g, 10.0 mmol) were mixed and stirred at 120 °C overnight. After drying at vacuum 3.40 g (99 %) of N^1,N^2-bis(2-(diisopropylamino)ethyl)oxalamide could be isolated as a pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 7.92 (br, 2H, NH), 3.27 (q, J = 5.9 Hz, 4H, $NHCH_2CH_2$), 3.01 (sept, J = 6.4 Hz, 4H, $CH(CH_3)_2$), 2.62 (t, J = 5.9 Hz, 4H, $NHCH_2CH_2$), 1.01 (d, J = 6.4 Hz, 24H, $CH(CH_3)_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 159.9 (CO), 48.1 ($NHCH_2CH_2$), 43.1 ($NHCH_2CH_2$), 38.7 ($CH(CH_3)_2$), 20.9 ($CH(CH_3)_2$).

HRMS (Methode) [m/z]: 343.3 (ES+)

Synthesis of N^1,N^2-bis(2-(diisobutylamino)ethyl)oxalamide:

N,N-diisobutylethylenediamine (1.89 g, 11.0 mmol) and diethyl oxalate (730 mg, 5.00 mmol) were mixed and stirred at 120 °C overnight. After drying at vacuum 1.98 g (99 %) of N^1,N^2-bis(2-(diisobutylamino)ethyl)oxalamide could be isolated as a redish solid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 7.89 (br, 2H, NH), 3.32 (q, J = 5.8 Hz, 4H, $NHCH_2CH_2$), 2.49 (t, J = 5.8 Hz, 4H, $NHCH_2CH_2$), 2.09 (d, J = 7.3 Hz, 8H, $CH_2CH(CH_3)_2$), 1.69 (nonett, J = 6.6 Hz, 4H, $CH_2CH(CH_3)_2$), 0.89 (d, J = 6.6 Hz, 24 Hz, $CH_2CH(CH_3)_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 159.8 (CO), 63.9 ($CH_2CH(CH_3)_2$), 53.6 ($NHCH_2CH_2$), 37.3 ($NHCH_2CH_2$), 26.7 ($CH_2CH(CH_3)_2$), 21.0 ($CH_2CH(CH_3)_2$).

HRMS (Methode) [m/z]: 399.4 (ES+)

Synthesis of N^1,N^4-bis(2-(diethylamino)ethyl)succinamide:

N,N-diethylethylenediamine (5.80 g, 50.0 mmol) and dimethyl succinate (2.92 g, 20.0 mmol) were mixed and stirred at 120 °C overnight. After drying at vacuum 6.20 g (99 %) of N^1,N^4-bis(2-(diethylamino)ethyl)succinamide could be isolated as an orange solid.

^{1}H NMR (300 MHz, $CDCl_3$): *δ(ppm)* = 6.44 (br, 2H, NH), 3.27 (q, J = 5.7 Hz, 4H, $NHCH_2$), 2.52 (m, 16H, $CH_2N(CH_2CH_3)_2$ + CH_2CH_2), 1.00 (t, J = 7.1 Hz, 12H, CH_2CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 172.1 (CO), 51.5 ($NHCH_2CH_2$) , 46.8 (CH_2CH_3), 37.1 ($NHCH_2CH_2$), 31.9 (CH_2CH_2), 11.8 (CH_2CH_3).

HRMS (Methode) [m/z]: 315.3 (ES+)

*Synthesis of L(Et-Me_2)H_2 (**1a**):*

Lithium aluminum hydride (760 mg, 20.0 mmol) was added slowly to a stirred solution of N^1,N^2-bis(2-(dimethylamino)ethyl)oxalamide (1.84 g, 8.00 mmol) in dry THF (80 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C for three days, cooled down to room temperature and water (3 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 910 mg (56 %) of L(Et-Me_2)H_2 (**1a**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 2.74 (s, 4H, CH_2CH_2), 2.68 (t, J = 6.2 Hz, 4H, NHCH_2CH_2), 2.40 (t, J = 6.2 Hz, 4H, NHCH_2CH_2), 2.20 (s, 12H, N(CH_3)$_2$), 2.12 (br, 2H, N*H*CH_2CH_2).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 59.2 (NHCH_2*C*H_2), 49.6 (*C*H_2CH_2), 47.4 (NH*C*H_2CH_2), 45.7 (N(*C*H_3)$_2$).

HRMS (Methode) [m/z]: 203.2 (ES+)

*Synthesis of L(Et-Et_2)H_2 (**1b**):*

Lithium aluminum hydride (380 mg, 10.0 mmol) was added slowly to a stirred solution of N^1,N^2-bis(2-(diethylamino)ethyl)oxalamide (1.15 g, 4.00 mmol) in dry THF (40 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C for three days, cooled down to room temperature and water (1 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 410 mg (41 %) of L(Et-Et_2)H_2 (**1b**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 2.74 (d, J = 0.8 Hz, 4H, CH_2CH_2), 2.67 (t, J = 6.3 Hz, 4H, NHCH_2CH_2), 2.56-2.47 (m, 12H, CH_2N(CH_2CH_3)$_2$), 1.98 (br, 2H, NH), 0.99 (t, J = 7.1 Hz, 12H, CH_2N(CH_2CH_3)$_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 52.8 (*C*H_2N(CH_2CH_3)$_2$), 49.7(NH*C*H_2CH_2), 47.7 (*C*H_2*C*H_2) , 47.2 (CH_2N(*C*H_2CH_3)$_2$), 11.9 (CH_2N(CH_2*C*H_3)$_2$).

HRMS (Methode) [m/z]: 259.3 (ES+)

*Synthesis of L(Et-^{i}Pr_2)H_2 (**1c**):*

Lithium aluminum hydride (760 mg, 20.0 mmol) was added slowly to a stirred solution of N^1,N^2-bis(2-(diisopropylamino)ethyl)oxalamide (2.74 g, 8.00 mmol) in dry THF (80 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C for three days, cooled down to room temperature and water (5 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 2.20 g (87 %) of L(Et-^{i}Pr_2)H_2 (**1c**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 2.91 (sept, J = 5.6 Hz, 4H, C*H*(CH_3)$_2$), 2.65 (s, 4H, CH2CH2), 2.53-2.46 (m, 8H, NHCH_2CH_2), 0.92 (d, J = 6.6 Hz, 24H, CH(CH_3)$_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 49.8 (NHCH_2*C*H_2), 49.7(NH*C*H_2CH_2), 48.1 (*C*H_2*C*H_2), 44.5 (C*H*(CH_3)$_2$), 20.8 (CH(CH_3)$_2$).

HRMS (Methode) [m/z]: 315.3 (ES+)

*Synthesis of L(Et-^{i}Bu$_2$)H$_2$ (**1d**):*

Lithium aluminum hydride (380 mg, 10.0 mmol) was added slowly to a stirred solution of N^1,N^2-bis(2-(diisobutylamino)ethyl)oxalamide (1.60 g, 4.00 mmol) in dry THF (80 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C for three days, cooled down to room temperature and water (3 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 1.10 g (74 %) of L(Et-^{i}Bu$_2$)H$_2$ (**1d**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 2.75 (s, 4H, CH_2CH_2), 2.63 (t, J = 5.7 Hz, 4H, NHCH_2CH_2), 2.46 (t, J = 5.7 Hz, 4H, NHCH_2CH_2), 2.06 (d, J = 7.2 Hz, 8H, CH_2CH(CH_3)$_2$), 1.69 (nonett, J = 6.9 Hz, 4H, CH_2CH(CH_3)$_2$), 0.86 (d, J = 6.7 Hz, 24H, CH_2CH(CH_3)$_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 64.4 (*C*H_2CH(CH_3)$_2$), 54.8 (NHCH_2*C*H_2), 49.5 (NH*C*H_2CH_2), 47.7 (*C*H_2*C*H_2), 26.6 (CH_2*C*H(CH_3)$_2$), 21.1 (CH_2CH(*C*H_3)$_2$).

HRMS (Methode) [m/z]: 371.4 (ES+)

*Synthesis of L(Bu-Et$_2$)H$_2$ (**1e**):*

Lithium aluminum hydride (380 mg, 10.0 mmol) was added slowly to a stirred solution of N^1,N^4-bis(2-(diethylamino)ethyl)succinamide (1.26 g, 4.00 mmol) in dry THF (40 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C for three days, cooled down to room temperature and water (1 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 520 mg (45 %) of L(Bu-Et$_2$)H$_2$ (**1e**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 2.67-2.62 (m, 8H), 2.55-2.44 (m, 12H), 2.01 (br, 2H, NH), 1.55-1.53 (m, 4H, CH_2CH_2CH_2CH_2), 0.99 (t, J = 7.1 Hz, CH_2CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 52.7 (*C*H_2N(CH_2CH_3)$_2$), 50.1 (NH*C*H_2CH_2), 47.7 (*C*H_2CH_2 CH_2*C*H_2), 47.3 (CH_2N(*C*H_2CH_3)$_2$), 28.1 (CH_2*C*H_2 *C*H_2CH_2), 12.0 (CH_2N(CH_2*C*H_3)$_2$).

HRMS (Methode) [m/z]: 287.3 (ES+)

*Synthesis of L(Xyl-Et$_2$)H$_2$ (**1f**):*

Sodium borohydride (1.70 g, 45.0 mmol) was added portion wise to a solution of bis[diethylaminoethyl]-N,N‘-m-xylylendiimine (4.95 g, 45.0 mmol) in EtOH (30 mL) at 0 °C followed by stirring of the reaction at 50 °C for three days. After the mixture was cooled to room temperature, water (10 mL) was added and the mixture was further stirred for 20 minutes before the majority of the solvent was removed in vacuum. The remaining solution was mixed with Et_2O (20 mL) and water (10 mL) and the aqueous phase was extracted with Et_2O (2x20 mL). The combined organic layers were dried over Na_2SO_4 followed by solvent removal to obtain 4.40 g (88 %) of L(Xyl-Et$_2$)H$_2$ (**1f**) as an yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.18-7.10 (m, 4H, C_6H_4), 3.70 (s, 4H, CH_2PhCH_2), 2.59 (t, J=6.0 Hz, 4H, $NHCH_2CH_2$), 2.47 (t, J=6.0 Hz, 4H, $NHCH_2CH_2$), 2.40 (q, J=7.1 Hz, 8H, NCH_2CH_3), 2.05 (br, 2H, NH), 0.91 (t, J=7.1 Hz, 12H, NCH_2CH_3).

$^{13}C\{^{1}H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 140.5 (2), 128.2 (1), 127.8 (4), 126.5 (3), 53.9 ($NHCH_2CH_2$), 52.5 (CH_2PhCH_2), 46.9 (NCH_2CH_3), 46.8 ($NHCH_2CH_2$), 11.7 (NCH_2CH_3).

HRMS (Methode) [m/z]: 335.317 (ES+)

*Synthesis of L(Xyl-^{i}Pr$_2$)H$_2$ (**1g**):*

Sodium borohydride (1.70 g, 45.0 mmol) was added portion wise to a solution of bis[diisopropylaminoethyl]-N,N‘-m-xylylendiimine (5.80 g, 15.0 mmol) in MeOH (50 mL) at room temperature followed by refluxing overnight. Afterwards, saturated NH_4Cl (aq.) (20 mL) solution and Et_2O (20 mL) were added to the mixture followed by extraction of the aqueous phase with Et_2O (2x20 mL). The combined organic layers were dried over $MgSO_4$ followed by solvent removal to obtain 4.26 g (73 %) of L(Xyl-^{i}Pr$_2$)H$_2$ (**1g**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.24-7.15 (m, 4H, C_6H_4), 3.76 (s, 4H, CH_2PhCH_2), 2.94 (sept., J=6.6 Hz, 4H, $CH(CH_3)_2$), 2.57 (br, 8H, $NHCH_2CH_2$), 1.90 (br, 2H, NH), 0.96 (d, J=6.6 Hz, 24H, $CH(CH_3)_2$).

$^{13}C\{^{1}H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 140.8 (2), 128.4 (1), 127.8 (4), 126.5 (3), 54.1 (CH_2PhCH_2), 48.8 ($NHCH_2CH_2$), 48.0 ($NCH(CH_3)_2$), 44.3 ($NHCH_2CH_2$), 21.9 ($NCH(CH_3)_2$).

HRMS (Methode) [m/z]: 391.380 (ES+)

*Synthesis of L(o-Ph-Et$_2$)H$_2$ (**1h**):*

Lithium aluminum hydride (4.70 g, 119 mmol) was added slowly to a stirred solution of N,N'-(1,2-phenylene)bis(2-(diethylamino)acetamide) (10.6 g, 34.0 mmol) in dry THF (160 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C overnight, cooled down to room temperature and water (9 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 7.90 g (76 %) of L(o-Ph-Et_2)H_2 (**1h**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 6.80-6.61 (m, 4H, C_6H_4), 4.08 (br, 2H, NH), 3.12 (t, J = 5.9 Hz, 4H, $NHCH_2CH_2$), 2.76 (t, J = 5.9 Hz, 4H, $NHCH_2CH_2$), 2.56 (q, J = 7.2 Hz, 8H, CH_2CH_3), 1.04 (t, J = 7.2 Hz, 12H, CH_2CH_3).

$^{13}C\{^{1}H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 137.6 (*i*-C_6H_4), 118.6 (*m*-C_6H_4), 110.9 (*o*-C_6H_4), 51.9 ($NHCH_2CH_2$), 46.8 (CH_2CH_3), 41.6 ($NHCH_2CH_2$), 12.0 (CH_2CH_3).

HRMS (Methode) [m/z]: 307.29 (ES+)

*Synthesis of L(o-Ph-Pip)H$_2$ (**1i**):*

Lithium aluminum hydride (1.90 g, 46.9 mmol) was added slowly to a stirred solution of N,N'-(1,2-phenylene)bis(2-(piperidin-1-yl)acetamide) (4.80 g, 13.4 mmol) in dry THF (100 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C overnight, cooled

down to room temperature and water (4 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 3.20 g (73 %) of L(o-Ph-Pip)H_2 (**1i**) as a pink solid.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 6.78-6.75 (m, 2H, *o*-C_6H_4), 6.66-6.62 (m, 2H, *m*-C_6H_4), 4.09 (br, 2H, NH), 3.16-3.15 (m, 4H, $NHCH_2CH_2$), 2.66 (t, J = 6.1 Hz, 4H, $NHCH_2CH_2$), 2.44 (br, 8H, α-pip), 1.63-1.55 (m, 8H, β-pip), 1.48-1.44 (m, 4H, γ-pip).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 137.5 (*i*-C_6H_4), 118.6 (*m*-C_6H_4), 111.0 (*o*-C_6H_4), 57.8 ($NHCH_2CH_2$), 54.6 (α-pip), 41.0 ($NHCH_2CH_2$), 26.2 (β-pip), 24.6 (γ-pip).

HRMS (Methode) [m/z]: 331.3 (ES+)

*Synthesis of L(m-Ph-Et_2)H_2 (**1j**):*

Lithium aluminum hydride (6.40 g, 160 mmol) was added slowly to a stirred solution of N,N'-(1,3-phenylene)bis(2-(diethylamino)acetamide) (15.0 g, 45.0 mmol) in dry THF (200 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C overnight, cooled down to room temperature and water (13 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 9.80 g (72 %) of L(m-Ph-Et_2)H_2 (**1j**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 6.98 (t, J = 7.9 Hz, 1H, 4), 6.03 (dd, J = 2.2 Hz, J = 7.9 Hz, 2H, 3), 5.94 (t, J = 2.2 Hz, 1H, 1), 4.21 (br, 2H, NH), 3.11 (dt, J = 5.9 Hz, 4H, $NHCH_2CH_2$), 2.67 (t, J = 5.9 Hz, 4H, $NHCH_2CH_2$), 2.55 (q, J = 7.1 Hz, 8H, CH_2CH_3), 1.02 (t, J = 7.1 Hz, 12H, CH_2CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 150.0 (2), 129.9 (4), 103.1 (3), 97.7 (1), 51.8 ($NHCH_2CH_2$), 46.8 (CH_2CH_3), 41.5 ($NHCH_2CH_2$), 11.9 (CH_2CH_3).

HRMS (Methode) [m/z]: 307.29 (ES+)

*Synthesis of L(m-Ph-Pip)H_2 (**1k**):*

Lithium aluminum hydride (1.00 g, 25.0 mmol) was added slowly to a stirred solution of N,N'-(1,3-phenylene)bis(2-(piperidin-1-yl)acetamide) (1.80 g, 5.00 mmol) in dry THF (50 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C overnight, cooled down to room temperature and water (2 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 930 mg (56 %) of L(m-Ph-Pip)H_2 (**1k**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 6.98 (t, J = 7.9Hz, 1H, 1), 6.03 (dd, J = 2.1 Hz, J = 7.9 Hz, 2H, 3), 5.94 (t, J = 2.1 Hz, 1H, 4), 4.24 (t, J = 5.3 Hz, 2H, NH), 3.14 (dt, J = 5.3 Hz, J = 6.0 Hz, 4H, $NHCH_2CH_2$), 2.56 (t, J = 6.0 Hz, 4H, $NHCH_2CH_2$), 2.39 (br, 8H, α-pip), 1.61-1.53 (m, 8H, β-pip), 1.47-1.43 (m, 4H,γ-pip).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 149.9 (2), 130.0 (4), 103.0 (3), 97.6 (1), 57.7 ($NHCH_2CH_2$), 54.5 (α-pip), 40.6 ($NHCH_2CH_2$), 26.1 (β-pip), 24.6 (γ-pip).

HRMS (Methode) [m/z]: 331.29 (ES+)

*Synthesis of L(o-Pyr-Et_2)H_2 (**1l**):*

Lithium aluminum hydride (390 mg, 10.3 mmol) was added slowly to a stirred solution of N,N'-(pyridine-2,6-diyl)bis(2-(diethylamino)acetamide) (1.00 g, 3.00 mmol) in dry THF (30 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C overnight, cooled down to room temperature and water (3 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 580 mg (63 %) of L(o-Pyr-Et_2)H_2 (**1l**) as a dark yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.21 (t, J = 7.8 Hz, 1H, *p*-C_6H_3N), 5.71 (d, J = 7.8 Hz, 2H, *m*-C_6H_3N), 4.73 (br, 2H, N*H*), 3.24 (dt, J = 6.2 Hz, 4H, $NHCH_2CH_2$), 2.63 (t, J = 6.2 Hz, $NHCH_2CH_2$), 2.54 (q, J = 7.0 Hz, 8H, CH_2CH_3), 1.01 (t, J = 7.0 Hz, CH_2CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 158.6 (*o*-C_6H_3N), 138.9 (*p*-C_6H_3N), 94.8 (*m*-C_6H_3N), 52.9 ($NHCH_2CH_2$), 46.8 (CH_2CH_3), 39.8 ($NHCH_2CH_2$), 11.8 (CH_2CH_3).

HRMS (Methode) [m/z]: 308.2 (ES+)

*Synthesis of L(o-Dpe-Et_2)H_2 (**1m**):*

Lithium aluminum hydride (133 mg, 3.50 mmol) was added slowly to a stirred solution of N,N'-(oxybis(2,1-phenylene))bis(2-(diethylamino)acetamide) (426 mg, 1.00 mmol) in dry THF (10 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C overnight, cooled down to room temperature and water (1 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 200 mg (51 %) of L(o-Dpe-Et_2)H_2 (**1m**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 6.99 (dt, J = 1,5 Hz, J = 7.7 Hz, 2H, *1*), 6.75-7.20 (m, 4H, *2+3*), 6.58 (dt, J = 1.5 Hz, J = 7.8 Hz, *4*), 4.74 (br, 2H, NH), 3.19 (q, J = 5.5 Hz, 4H, $NHCH_2CH_2$), 2.68 (t, J = 6.43 Hz, 4H, $NHCH_2CH_2$), 2.51 (q, J = 7.1 Hz, 8H, CH_2CH_3), 0.97 (t, J = 7.1 Hz, 12H, CH_2CH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 143.9 (C_6H_4), 140.3 (C_6H_4), 124.2 (C_6H_4), 117.4 (C_6H_4), 116.6 (C_6H_4), 111.3 (C_6H_4), 51.9 ($NHCH_2CH_2$), 47.0 (CH_2CH_3), 41.5 ($NHCH_2CH_2$), 12.0 (CH_2CH_3).

HRMS (Methode) [m/z]: 399.3 (ES+)

*Synthesis of L(o-Dpen-Et_2)H_2 (**1n**):*

Lithium aluminum hydride (332 mg, 8.75 mmol) was added slowly to a stirred solution of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diethylamino)acetamide) (1.00 g, 2.30 mmol) in dry THF (23 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C overnight, cooled down to room temperature and water (2 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 590 mg (63 %) of L(o-Dpen-Et_2)H_2 (**1n**) as a pale yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.18-7.09 (m, 4H, C_6H_4), 6.71-6.63 (m, 4H, C_6H_4), 4.58 (t, J = 4.8 Hz , 2H, NH), 3.20-3.13 (m, 4H, $NHCH_2CH_2$), 2.78 (s, 4H, CH_2CH_2), 2.74 (t, J = 5.8 Hz, 4H, $NHCH_2CH_2$), 2.58 (q, J = 7.2 Hz, 8H, CH_2CH_3), 1.05 (t, J = 7.2 Hz, 12H, CH_2CH_3).

$^{13}C\{^1H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 146.4 (C_6H_4), 129.4 (C_6H_4), 127.5 (C_6H_4), 126.3 (C_6H_4), 117.0 (C_6H_4), 110.6 (C_6H_4), 51.7 ($NHCH_2CH_2$), 46.7 (CH_2CH_3), 41.3 ($NHCH_2CH_2$), 31.2 (CH_2CH_2), 12.0 (CH_2CH_3).

HRMS (Methode) [m/z]: 411.3 (ES+)

*Synthesis of L(o-Dpen-^{i}Bu$_2$)H$_2$ (**1o**):*

Lithium aluminum hydride (771 mg, 20.3 mmol) was added slowly to a stirred solution of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diisobutylamino)acetamide) (3.20 g, 5.80 mmol) in dry THF (60 mL) at 0 °C followed by stirring for one hour at room temperature. The mixture was heated to 66 °C overnight, cooled down to room temperature and water (2 mL) was added followed by stirring for 30 minutes. The formed solid was filtered off, washed with THF and the solvent of the filtrate was removed at vacuum to obtain 1.70 g (57 %) of L(o-Dpen-^{i}Bu$_2$)H$_2$ (**1o**).

^1H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.15 (t, J = 7.6 Hz, 2H, C_6H_4), 6.99 (d, J = 7.4 Hz, 2H, C_6H_4), 6.68-6.63 (m, 4H, C_6H_4), 4.40 (br, 4H, NH), 3.14 (q, J = 4.9 Hz, 4H, $NHCH_2CH_2$), 2.84 (s, 4H, CH_2CH_2), 2.66 (t, J = 5.7 Hz, 4H, $NHCH_2CH_2$), 2.14 (d, J = 7.2 Hz, 8H, $CH_2CH(CH_3)_2$), 1.75 (nonett, 4H, $CH_2CH(CH_3)_2$), 0.88 (d, J = 6.5 Hz, 24H, $CH_2CH(CH_3)_2$).

$^{13}C\{^1H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 146.2 (C_6H_4), 128.6 (C_6H_4), 127.2 (C_6H_4), 125.7 (C_6H_4), 116.8 (C_6H_4), 110.2 (C_6H_4), 63.9 ($CH_2CH(CH_3)_2$), 54.4 ($NHCH_2CH_2$), 41.2 ($NHCH_2CH_2$), 29.2 (CH_2CH_2), 26.6 ($CH_2CH(CH_3)_2$), 21.1 ($CH_2CH(CH_3)_2$).

HRMS (Methode) [m/z]: 523.5 (ES+)

*Synthesis of L(Pip-Dmp)H$_2$ (**2a**):*

Lithium aluminum hydride (2.28 g, 60.0 mmol) was added slowly to a solution of 2,2'-(piperazine-1,4-diyl)bis(N-(2,6-dimethylphenyl)acetamide) (7.72 g, 60.0 mmol) in THF (50 mL) and the mixture was refluxed for four days. After cooling to room temperature water (40 mL) and NaOH (aq., 2M, 10 mL) were added and the suspension was filtered through a plug of celite. The solvent was removed and the crude product was recrystallized from THF to obtain 2.73 g (57 %) of L(Pip-Dmp)H$_2$ (**2a**).

^1H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.00 (d, J=7.4 Hz, 4H, *m*-C_6H_3), 6.81 (t, J=7.4 Hz, 2H, *p*-C_6H_3), 3.99 (br, 2H, N*H*), 3.11 (t, J= 5.9, 4H, $NHCH_2$), 2.63-2.53 (m, 12H, $CH_2N(CH_2)_2$), 2.32 (s, 12H, $PhCH_3$)

$^{13}C\{^1H\}$ NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 146.6 (*i*-C_6H_3), 128.9 (*m*-C_6H_3), 128.4 (*o*-C_6H_3), 121.3 (*p*-C_6H_3), 57.9 ($NHCH_2CH2N$), 53.2 ($N(C_2H_4)_2N$), 44.8 ($NHCH_2CH_2N$), 18.9 ($PhCH_3$)

HRMS (Methode) [m/z]: 381.302 (ES+)

*Sythesis of L(Et-Dmp)H$_2$ (**2b**):*

1,2-dibromoethane (1.00 g, 5.60 mmol) was slowly added to pure N^1-(2,6-dimethylphenyl)-N^2-methylethane-1,2-diamine (2.00 g, 11.2 mmol) at 130 °C and the mixture was stirred at this temperature for three hours. The formed sticky residue was dissolved in a mixture of EtOAc (70 mL) and water (70 mL) followed by separation of the organic phase. The crude product was recrystallized in EtOH (30 mL) at -20 °C to obtain 458 mg (21 %) of L(Et-Dmp)H$_2$ (**2b**) as white crystals.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.02 (d, J= 7.4 Hz, 4H, meta H), 6.83 (t, J= 7.4 Hz, 2H, para H), 3.91 (br, 2H, N*H*), 3.11 (t, J= 5.6 Hz, 4H, CH_2CH_2NHPh), 2.64-2.62 (m, 8H, $CH_2NCH_3CH_2$), 2.34 (s, 12H, $PhCH_3$), 2.32 (s, 6H, NCH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 146.7 (*i*-C_6H_3), 128.9 (*m*-C_6H_3), 128.7 (*o*-C_6H_3), 121.2 (*p*-C_6H_3), 58.1 (NCH_2CH_2NHPh), 56.2 (NCH_2CH_2N), 45.7 (NCH_2CH_2NPh), 42.2 (N*C*H3), 18.8 (Ph*C*H3).

HRMS (Methode) [m/z]: 383.316 (ES +)

*Sythesis of L(Et-Dipp)H_2 (**2c**):*

1,2-dibromoethane (3.70 g, 20.0 mmol) was slowly added to pure N^1-(2,6-diisopropylphenyl)-N^2-methylethane-1,2-diamine (9.40 g, 40.0 mmol) at 100 °C and the mixture was stirred at this temperature for three hours. The formed sticky residue was dissolved in a mixture of EtOAc (100 mL) and NaOH (aq.) (100 mL) followed by separation of the organic phase. Solvent concentration initiates crystal formation and 4.76 g (46 %) of L(Et-Dipp)H_2 (**2c**) could be obtained as white crystals.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.12-7.02 (m, 6H, C_6H_3), 3.33 (sept, J=6.8 Hz, 4H, $CH(CH_3)_2$), 2.98 (t, J= 2.98, 4H, NCH_2CH_2NPh), 2.68-2.64 (m, 8H, $CH_2N(CH_3)CH_2$), 2.33 (s, 6H, NCH_3), 1.25 (d, J=6.8Hz, 24H, $CH(CH_3)_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 143.9 (*i*-C_6H_3), 142.1 (*o*-C_6H_3), 123.6 (*m*-C_6H_3), 123.4 (*p*-C_6H_3), 58.5 (NCH_2CH_2NPh), 56.4 (NCH_2CH_2N), 49.1 (NCH_2CH_2NPh), 42.5 (NCH_3), 27.7 ($CH(CH_3)_2$), 24.4 ($CH(CH_3)_2$).

HRMS (Methode) [m/z]: 495.442 (ES+)

*Synthesis of L(Pr-Dmp)H_2 (**2d**):*

N^1-(2,6-dimethylphenyl)-N^2-methylethane-1,2-diamine (17.0 g, 95.0 mmol), 1,3-dibromopropane (8.90 g, 44.0 mmol) and potassium carbonate (27.0 g, 195 mmol) were suspended in EtOH (150 mL) and refluxed for three days. The solvent was removed and the residue was dissolved in a mixture of EtOAc (100 mL) and water (200 mL) followed by separation of the organic phase. The solvent of the organic phase was removed and HOAc (equal to the amount of unreacted N^1-(2,6-dimethylphenyl)-N^2-methylethane-1,2-diamine determined by ^{1}H-NMR analysis) as well as EtOAc (100 mL) and water (150 mL) were added to the mixture followed by stirring for one hour. The solvent of the organic phase was removed to obtain 15.2 g (87 %) of L(Pr-Dmp)H_2 (**2d**) as an oil.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.11 (d, *J*= 7.46Hz, 4H, *m*-C_6H_3), 6.92 (t, *J*=7.46Hz, 2H, *p*-C_6H_3), 3.83 (br, 2H, N*H*), 3.20 (t, *J*= 5.5Hz, 4H, $NHCH_2CH_2N$), 2.68 (t, *J*= 5.5, 4H, $NHCH_2CH_2N$), 2.60 (t, *J*=7.0, 4H, $CH_2CH_2CH_2$), 2.45 (s, 12H, $PhCH_3$), 2.37 (s, 6H, NCH_3), 1.87 (p, *J*= 7.0, 2H, $CH_2CH_2CH_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 146.4 (*i*-C_6H_3), 128.5 (*m*-C_6H_3), 128.1 (*o*-C_6H_3), 120.8 (*p*-C_6H_3), 57.4 ($NHCH_2CH_2N$), 55.9 ($CH_2CH_2CH_2$), 45.3 ($NHCH_2CH_2N$), 41.1(NCH_3), 25.1 ($CH_2CH_2CH_2$), 18.5 ($PhCH_3$).

HRMS (Methode) [m/z]: 397.333 (ES +)

*Sythesis of L(Bu-Dipp)H_2 (**2e**):*

1,3-dibromopropane (3.03 g, 15.0 mmol) was slowly added to pure N^1-(2,6-diisopropylphenyl)-N^2-methylethane-1,2-diamine (7.03 g, 30.0 mmol) at 100 °C and the mixture was stirred at this temperature for overnight. The formed solid residue was dissolved in a mixture of EtOAc (50 mL), NaOH (10 g) and water (50 mL) followed by extracting of the aqueous phase with EtOAc (2x50 mL). The combined organic phases were dried over Na_2SO_4 followed by solvent removal. The crude product was crystallized from EtOH (10 mL) at -16 °C to obtain 2.50 g (33 %) of L(Bu-Dipp)H_2 (**2e**) as white crystals.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.12-7.10 (m, 4H , *m*-C_6H_3), 7.06-7.03 (m, 2H, *p*-C_6H_3), 3.78 (br, 2H, N*H*), 3.35 (sept, J=6.8 Hz, 4H, C*H*$(CH_3)_2$), 2.99 (t, J= 5.6 Hz, 4H, $NHCH_2CH_2$), 2.62 (t, J=5.6 Hz, 4H, $NHCH_2CH_2$), 2.52-2.49 (m, 4H, $NCH_2CH_2CH_2N$), 2.29 (s, 6H, NCH_3), 1.79 (p, J=7.3 Hz, 2H, $NCH_2CH_2CH_2N$), 1.28-1.26 (d, J=6.8, 24H, $CH(CH_3)_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 144.0 (*i*-C_6H_3), 142.0 (*o*-C_6H_3), 123.6 (*m*-C_6H_3), 123.3 (*p*-C_6H_3), 58.2 ($NHCH_2CH_2$), 56.5 ($NHCH_2CH_2$), 49.1 ($NCH_2CH_2CH_2N$), 41.9 (NCH_3), 27.6 ($CH(CH_3)_2$), 25.9 ($NCH_2CH_2CH_2N$), 24.5 ($CH(CH_3)_2$).

HRMS (Methode) [m/z]: 509.458 (ES+)

*Synthesis of L(Xyl-Dmp)H_2 (**2f**):*

N^1-(2,6-dimethylphenyl)-N^2-methylethane-1,2-diamine (7.13 g, 40.0 mmol), m-xylylene dichloride (3.50 g, 20.0 mmol) and potassium carbonate (8.29 g, 60.0 mmol) were suspended in EtOH (100 mL) and refluxed for four days. The solvent was removed and the resulting residue was dissolved in a mixture of EtOAc (100 mL) and water (100 mL) followed by washing of the organic phase with saturated NaCl (aq.) (20 mL) solution. The organic phase was dried over Na_2SO_4 and the solvent was removed to obtain 8.70 g (95 %) of Synthesis of L(Xyl-Dmp)H_2 (**2f**) as an orange oil.

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.33-7.23 (m, 4H, Xylyl), 6.99 (d, J=7.52, 4H, *m*-C_6H_3), 6.79 (t, J=7.52, 2H, *p*-C_6H_3), 4.28 (br, 2H, N*H*), 3.59 (s, 4H, CH_2PhCH_2), 3.14 (t, J= 5.8Hz, 4H, NCH_2CH_2NHPh), 2.65 (t, J=5.8, 4H, NCH_2CH_2NHPh), 2.31 (s, 12H, $PhCH_3$), 2.21 (s, 6H, NCH_3).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 146.8 (*i*-C_6H_3), 138.9 (2), 129.8 (*m*-C_6H_3), 128.8 (1), 128.5 (*o*-C_6H_3), 128.3 (4), 127.8 (3), 121.1 (*p*-C_6H_3), 62.8 (CH_2PhCH2), 57.4 (NCH_2CH_2NHPh), 45.7 (NCH_2CH_2NHPh), 41.5 (NCH_3), 18.9 ($PhCH_3$).

HRMS (Methode) [m/z]: 459.348 (ES+)

*Synthesis of L(Xyl-Dipp)H_2 (**2g**):*

N^1-(2,6-diisopropylphenyl)-N^2-methylethane-1,2-diamine (9.40 g, 40.0 mmol), *m*-xylylene dichloride (3.50 g, 20.0 mmol) and potassium carbonate (8.29 g, 60.0 mmol) were suspended in EtOH (100 mL) and refluxed for four days. The solvent was removed and the resulting residue was dissolved in a mixture of EtOAc (100 mL) and water (100 mL) followed by washing of the organic phase with saturated NaCl (aq.) (20 mL) solution. The organic phase was dried over Na_2SO_4 and the solvent was removed to obtain 11.3 g (99 %) of L(Xyl-Dipp)H_2 (**2g**).

^{1}H NMR (400 MHz, $CDCl_3$): *δ(ppm)* = 7.32-7.03 (m, 10H, C_6H_3+C_6H_4), 3.60 (s, 2H, PhCH_2), 3.35 (sept, J=6.8 Hz, 6H, $CH(CH_3)_2$ + N*H*), 3.03 (t, J=5.6 Hz, 4H, NHCH_2CH_2), 2.69 (t, J=5.6Hz, 4H, $NHCH_2CH_2$), 2.21 (s, 6H, NCH_3), 1.24 (d, J=6.8Hz, 24H, $CH(CH_3)_2$).

^{13}C{^{1}H} NMR (101 MHz,$CDCl_3$): *δ (ppm)* = 144.0 (*i*-C_6H_3), 142.0 (*o*-C_6H_3),139.2 (2), 129.4 (1), 128.4 (4), 127.7 (3), 123.6 (*m*-C_6H_3), 123.3 (*p*-C_6H_3), 62.8 (PhCH2), 57.9 ($NHCH_2CH_2$), 49.0 ($NHCH_2CH_2$), 41.8 (NCH_3), 27.7 ($CH(CH_3)_2$), 24.5 ($CH(CH_3)_2$).

HRMS (Methode) [m/z]: 571.476 (ES+)

4. NMR and IR spectra

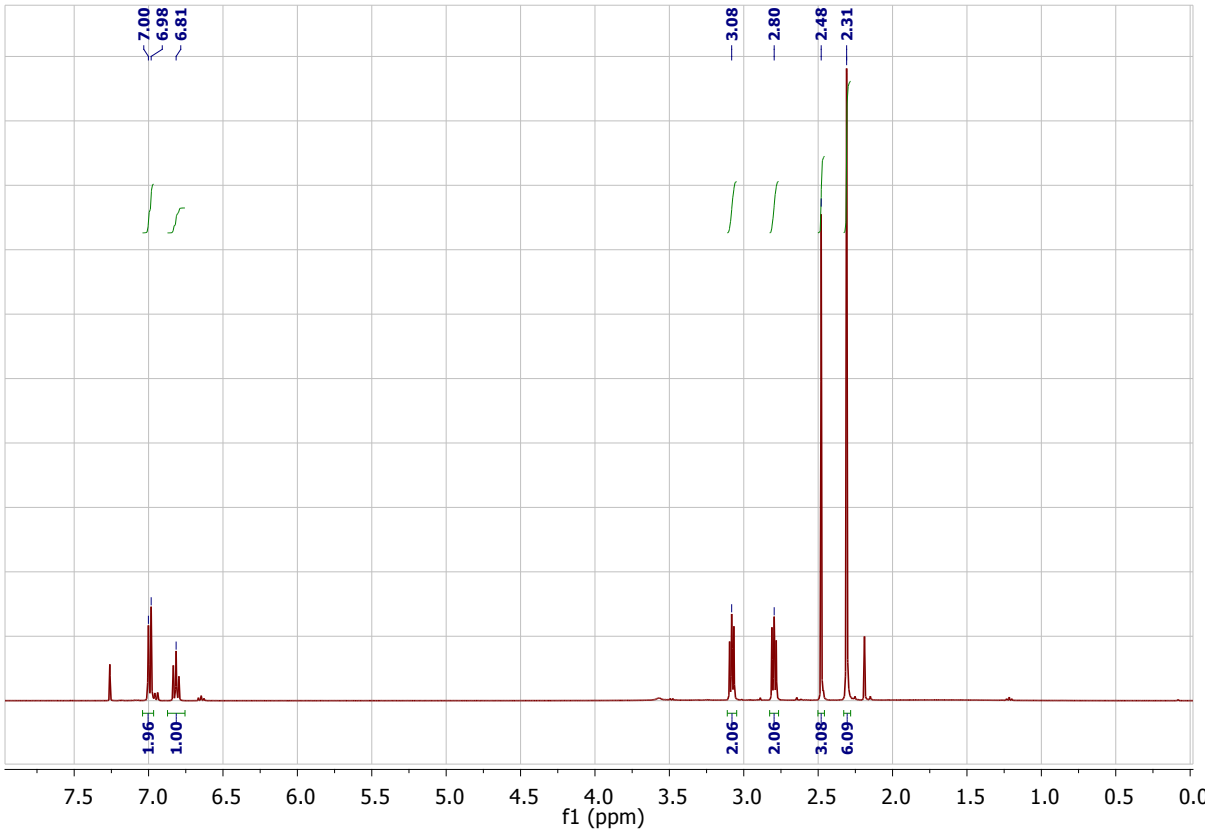

Figure S2 ^{1}H NMR spectrum (300 MHz) of N^1-(2,6-dimethylphenyl)-N^2-methylethane-1,2-diamine in $CDCl_3$ at 300 K.

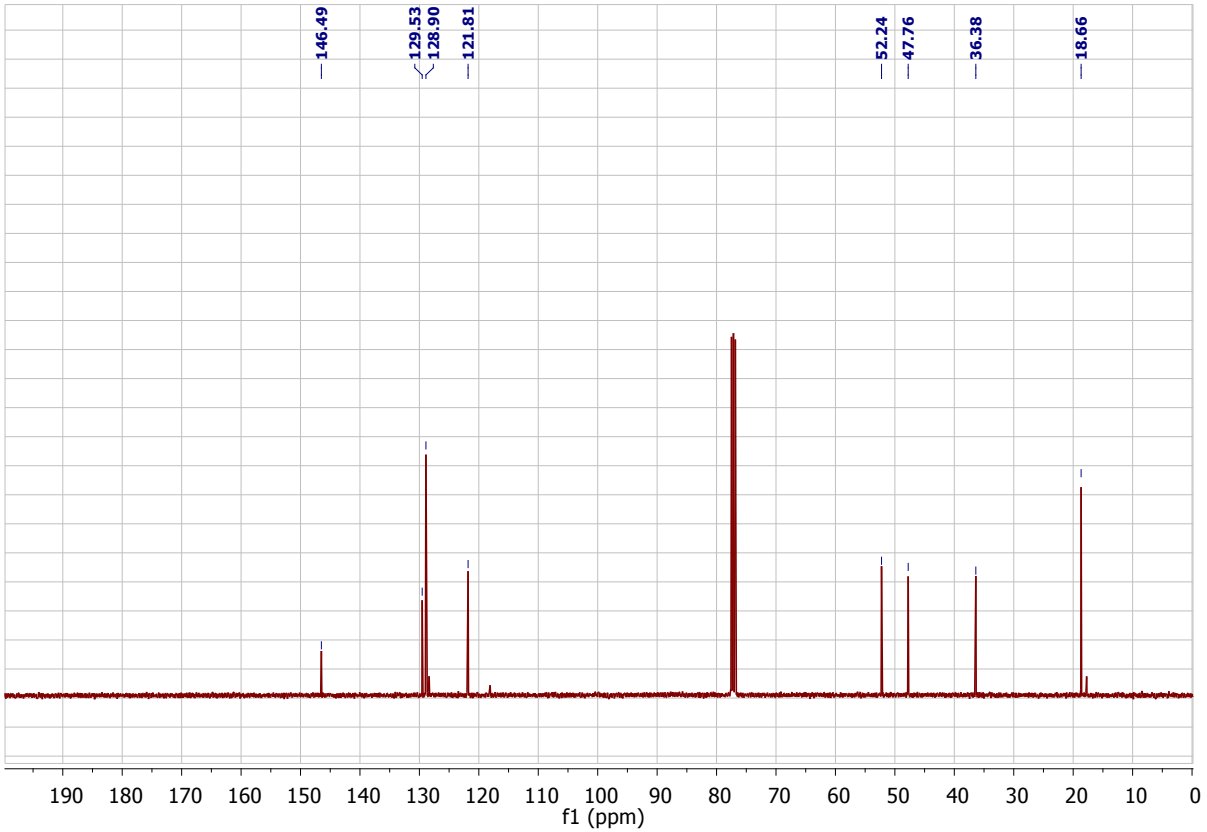

Figure S3 ^{13}C NMR spectrum (101 MHz) of N^1-(2,6-dimethylphenyl)-N^2-methylethane-1,2-diamine in $CDCl_3$ at 300 K.

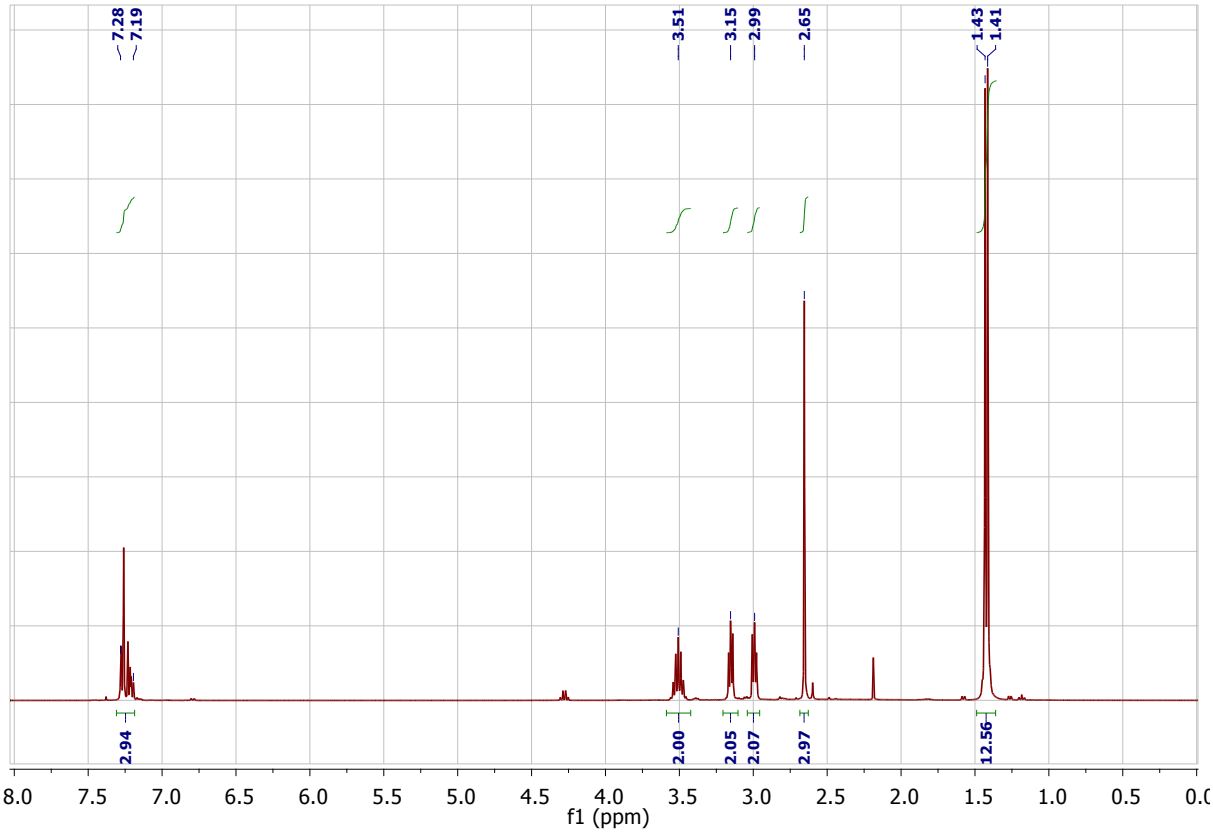

Figure S4 ^{1}H NMR spectrum (300 MHz) of N^1-(2,6-diisopropylphenyl)-N^2-methylethane-1,2-diamine in $CDCl_3$ at 300 K.

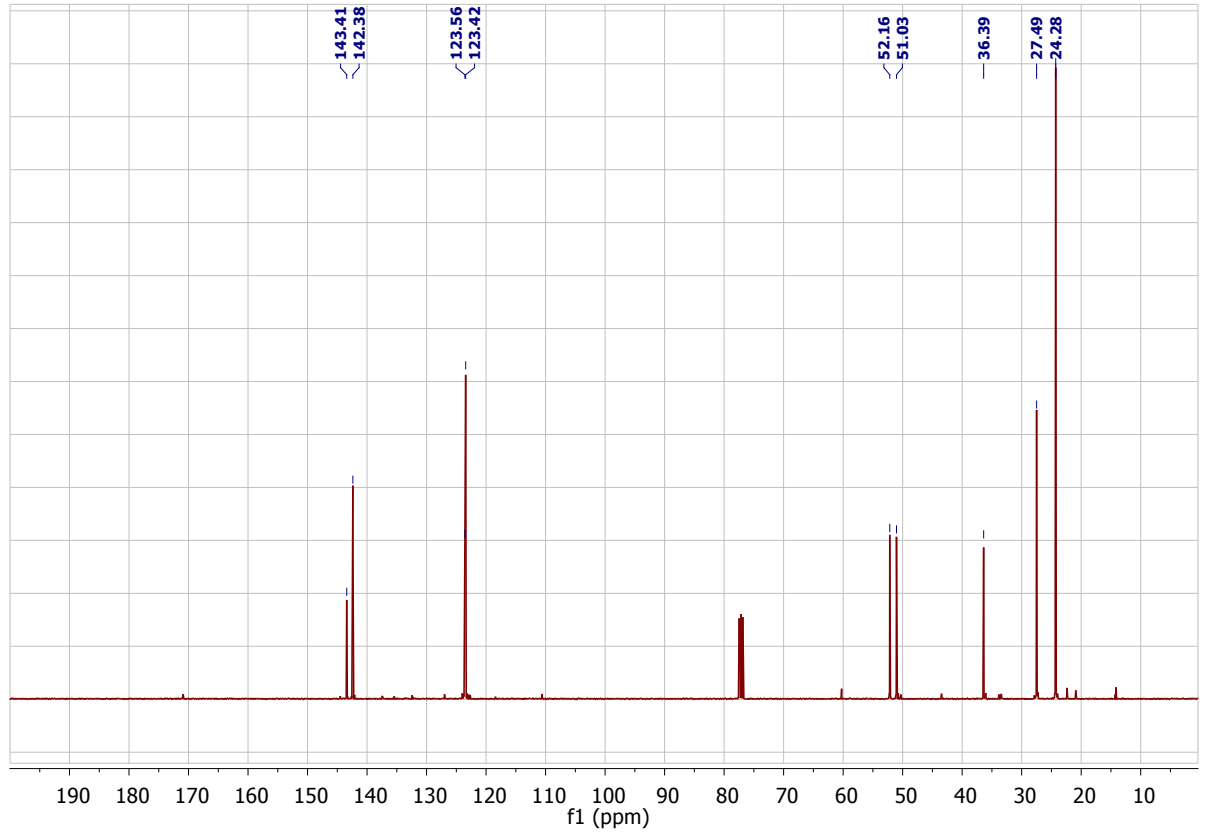

Figure S5 ^{13}C NMR spectrum (101 MHz) of N^1-(2,6-diisopropylphenyl)-N^2-methylethane-1,2-diamine in $CDCl_3$ at 300 K.

Figure S6 ^{1}H NMR spectrum (300 MHz) of Bis[diethylaminoethyl]-N,N'-m-xylylendiimine in $CDCl_3$ at 300 K.

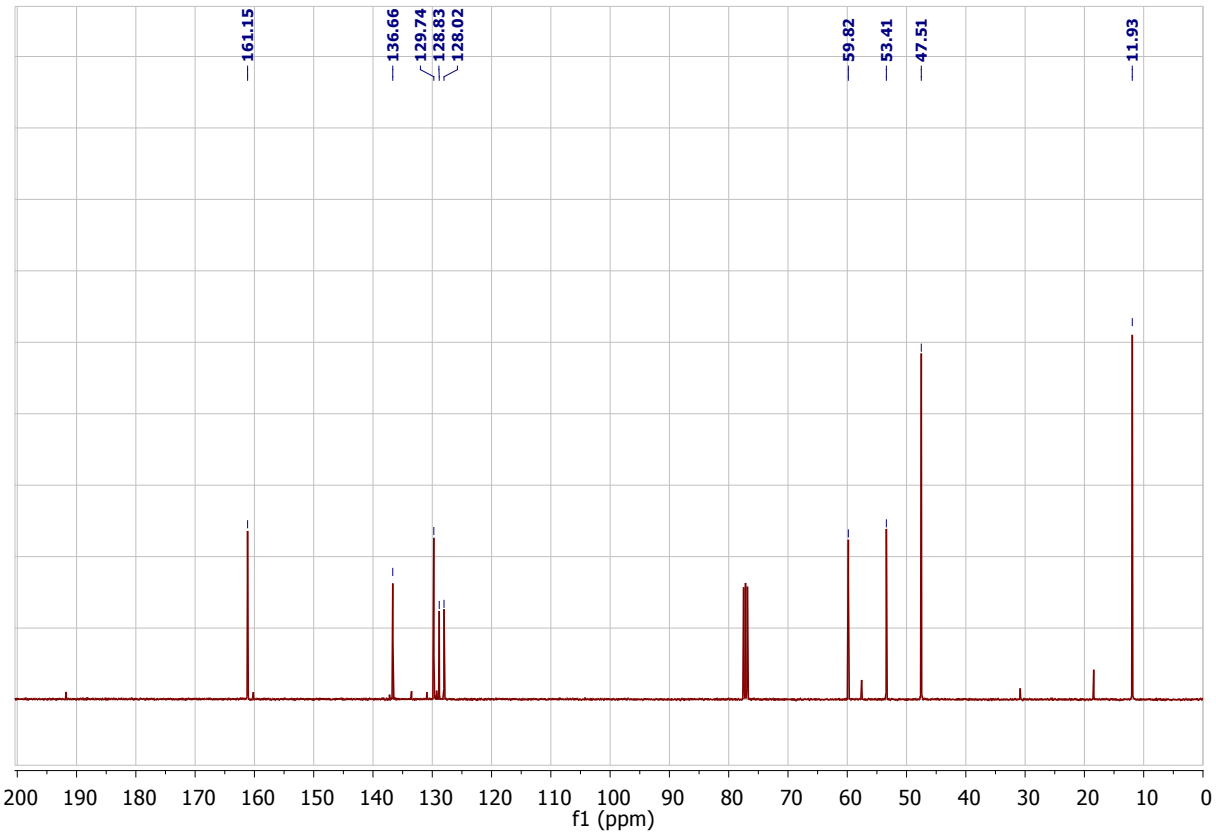

Figure S7 ^{13}C NMR spectrum (101 MHz) of Bis[diethylaminoethyl]-N,N'-m-xylylendiimine in $CDCl_3$ at 300 K.

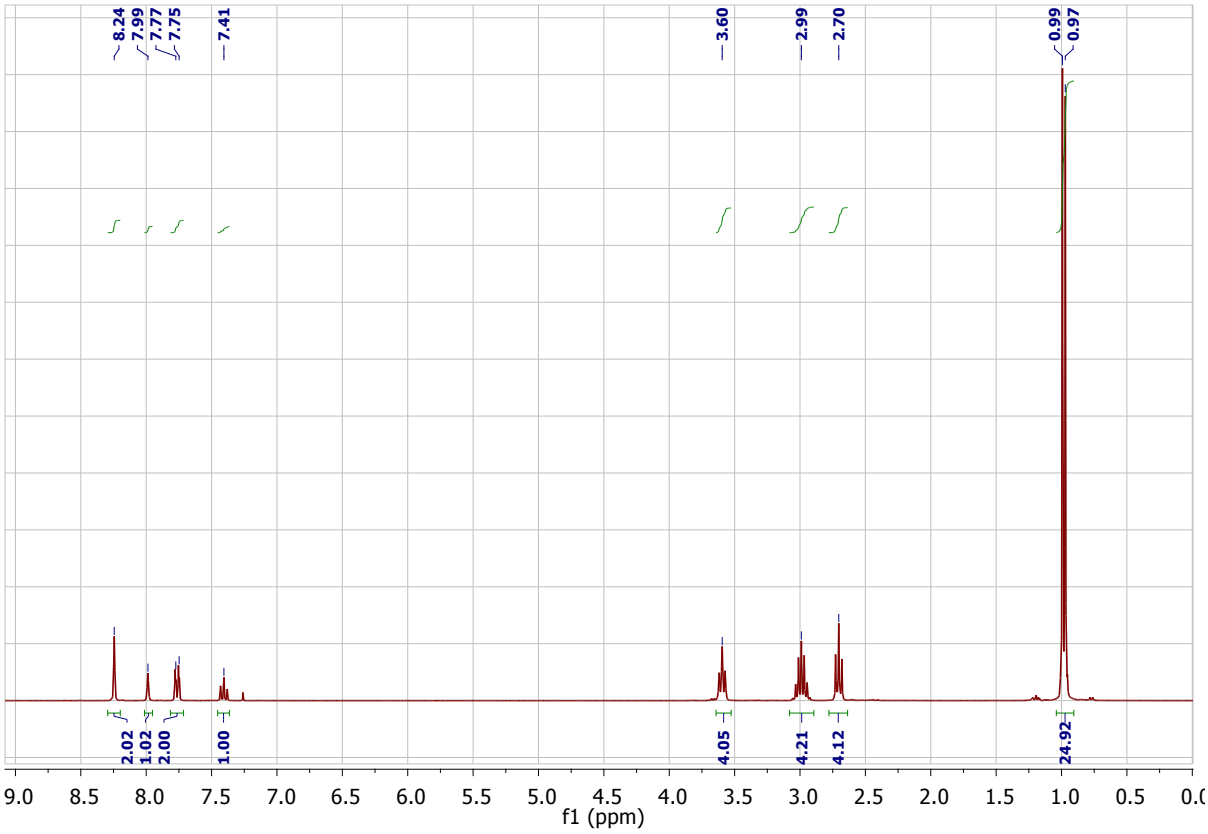

Figure S8 ^{1}H NMR spectrum (300 MHz) of Bis[diisopropylaminoethyl]-N,N'-m-xylylendiimine in $CDCl_3$ at 300 K.

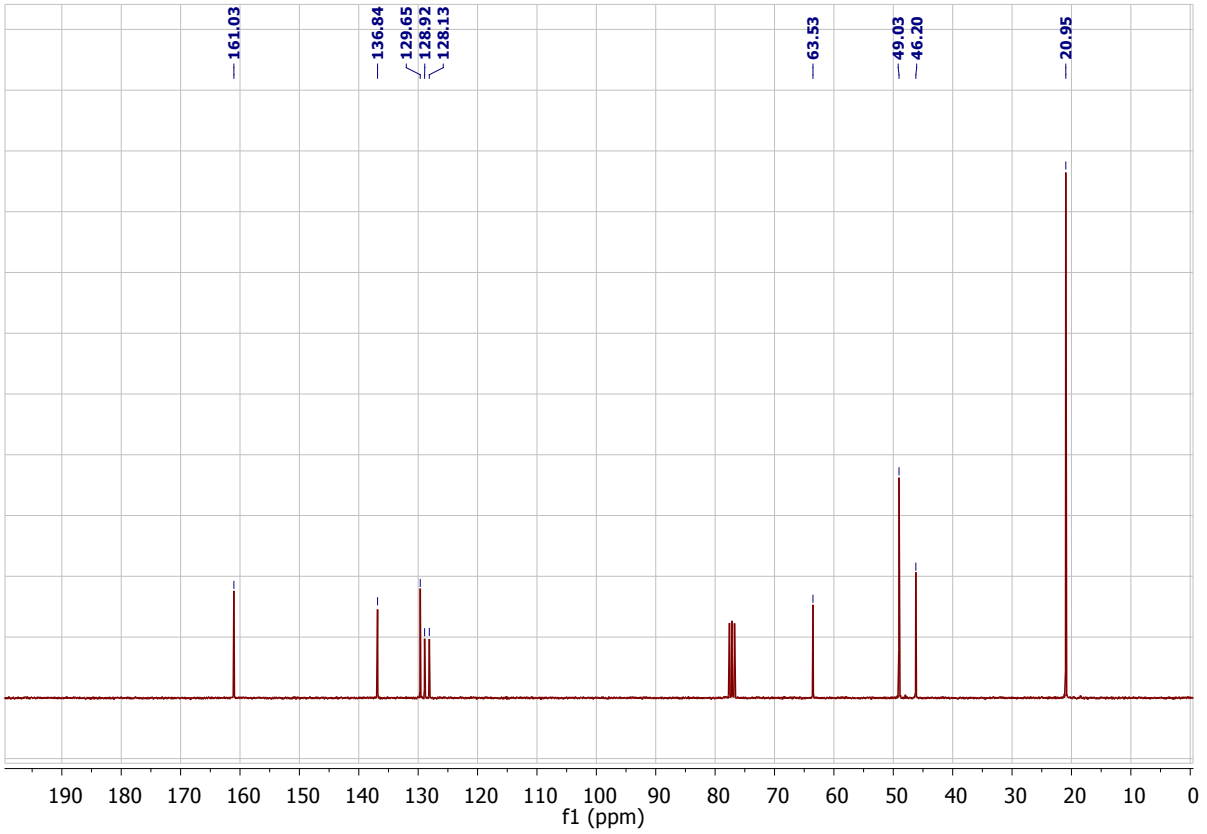

Figure S9 ^{13}C NMR spectrum (101 MHz) of Bis[diisopropylaminoethyl]-N,N'-m-xylylendiimine in $CDCl_3$ at 300 K.

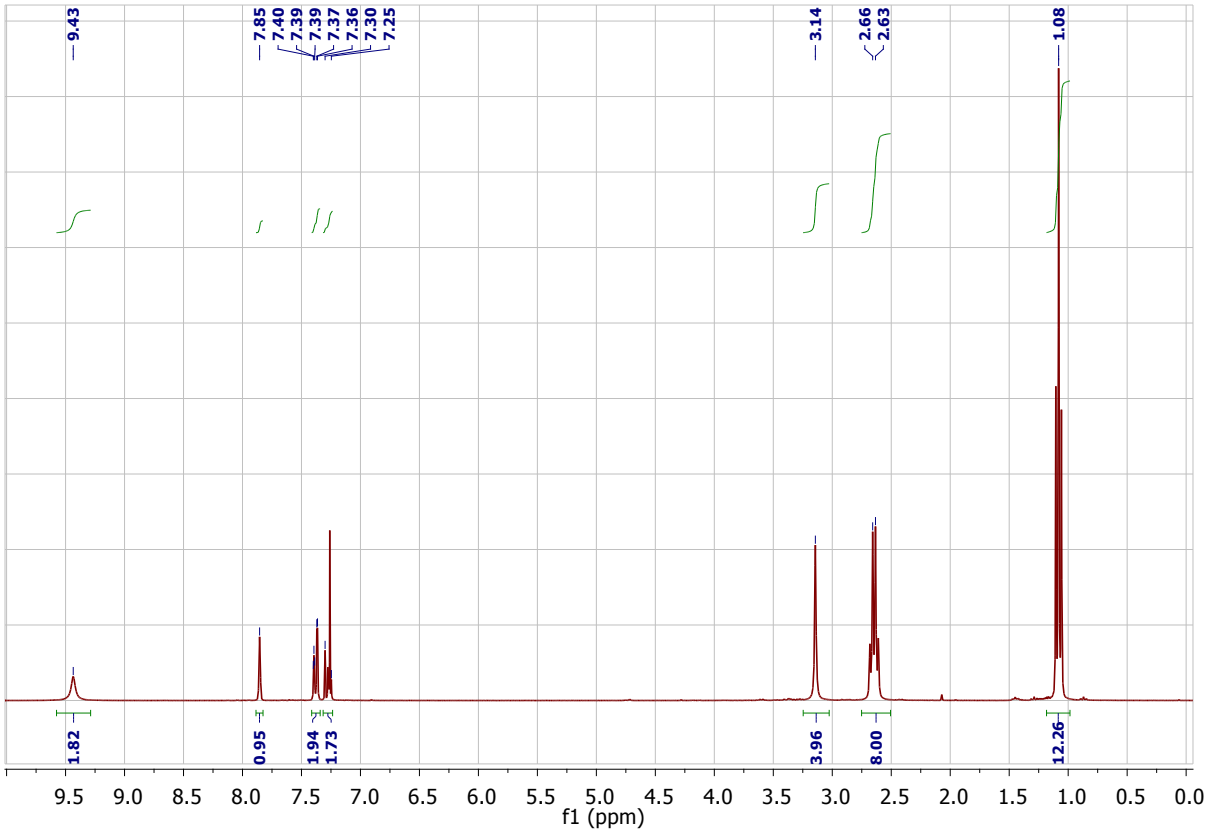

Figure S10 ^{1}H NMR spectrum (300 MHz) of N,N'-(1,3-phenylene)bis(2-(diethylamino)acetamide) in $CDCl_3$ at 300 K.

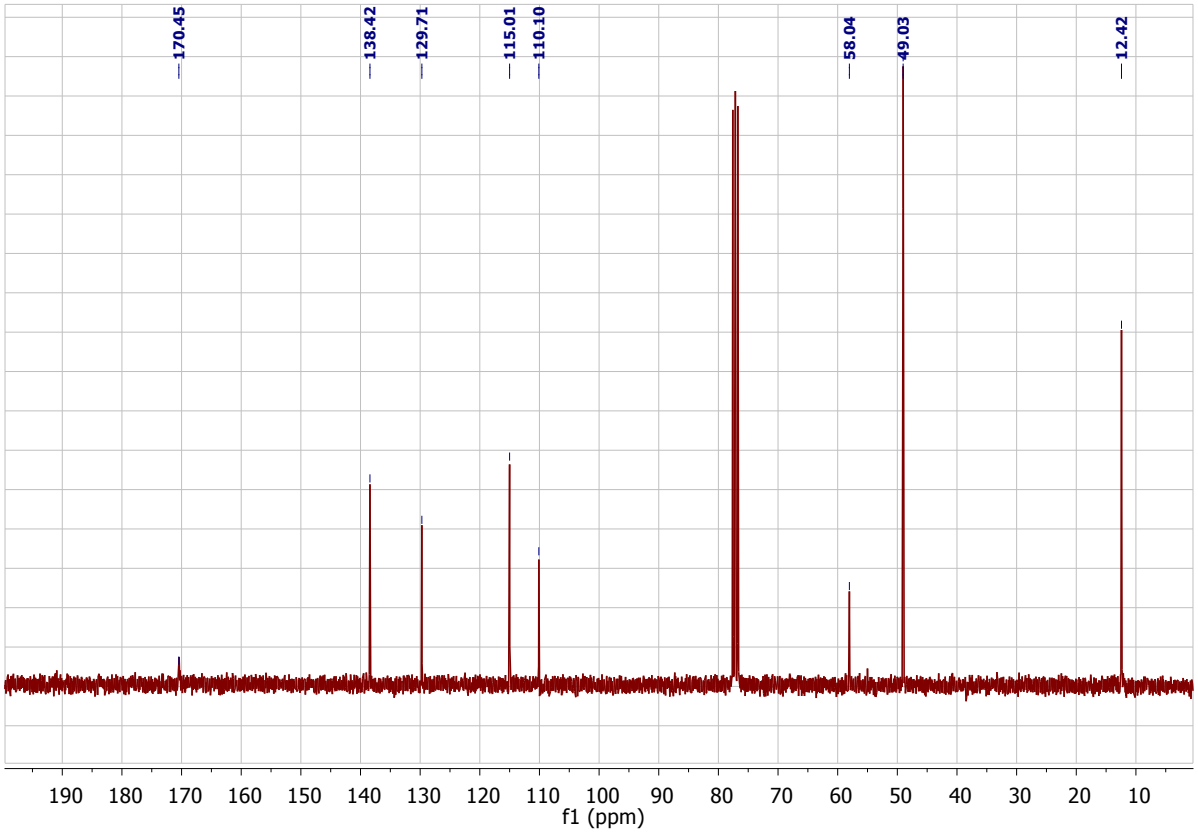

Figure S11 ^{13}C NMR spectrum (101 MHz) of N,N'-(1,3-phenylene)bis(2-(diethylamino)acetamide) in $CDCl_3$ at 300 K.

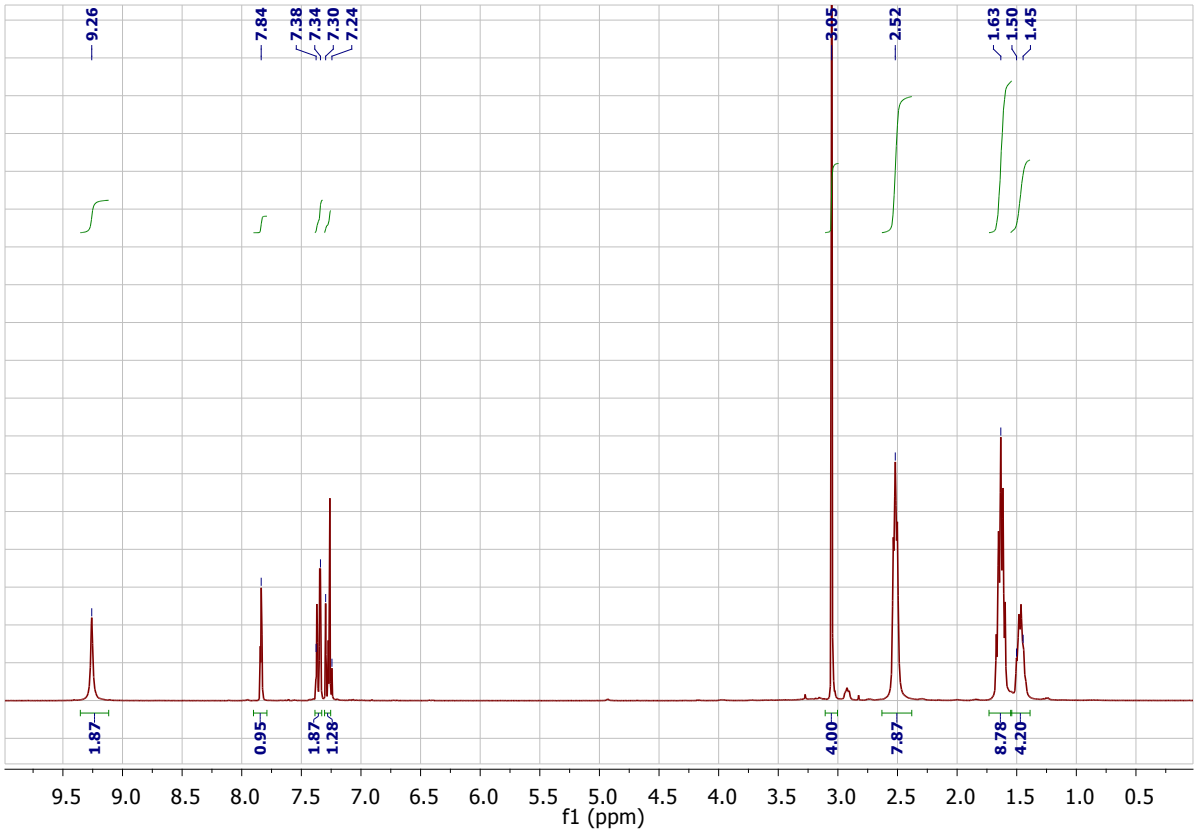

Figure S12 ^{1}H NMR spectrum (300 MHz) of N,N'-(1,3-phenylene)bis(2-(piperidin-1-yl)acetamide) in $CDCl_3$ at 300 K.

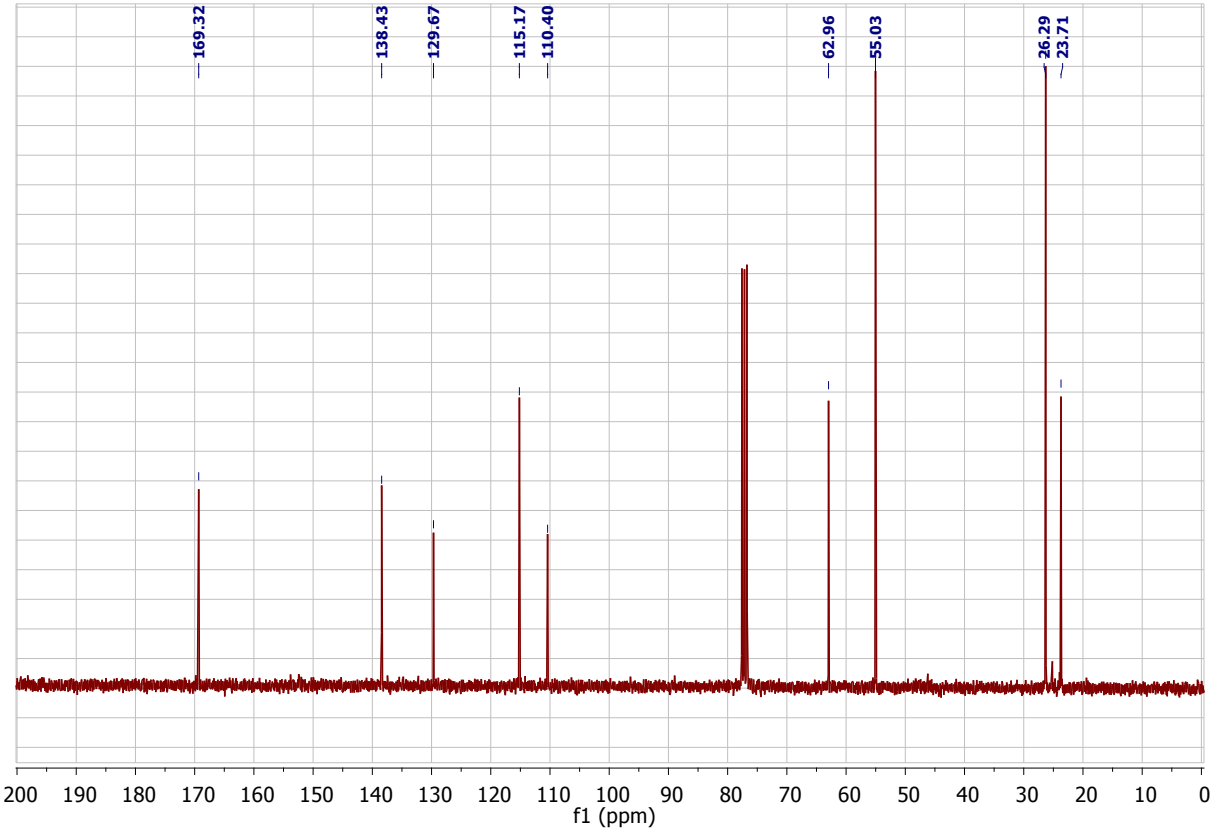

Figure S13 ^{13}C NMR spectrum (101 MHz) of N,N'-(1,3-phenylene)bis(2-(piperidin-1-yl)acetamide) in $CDCl_3$ at 300 K.

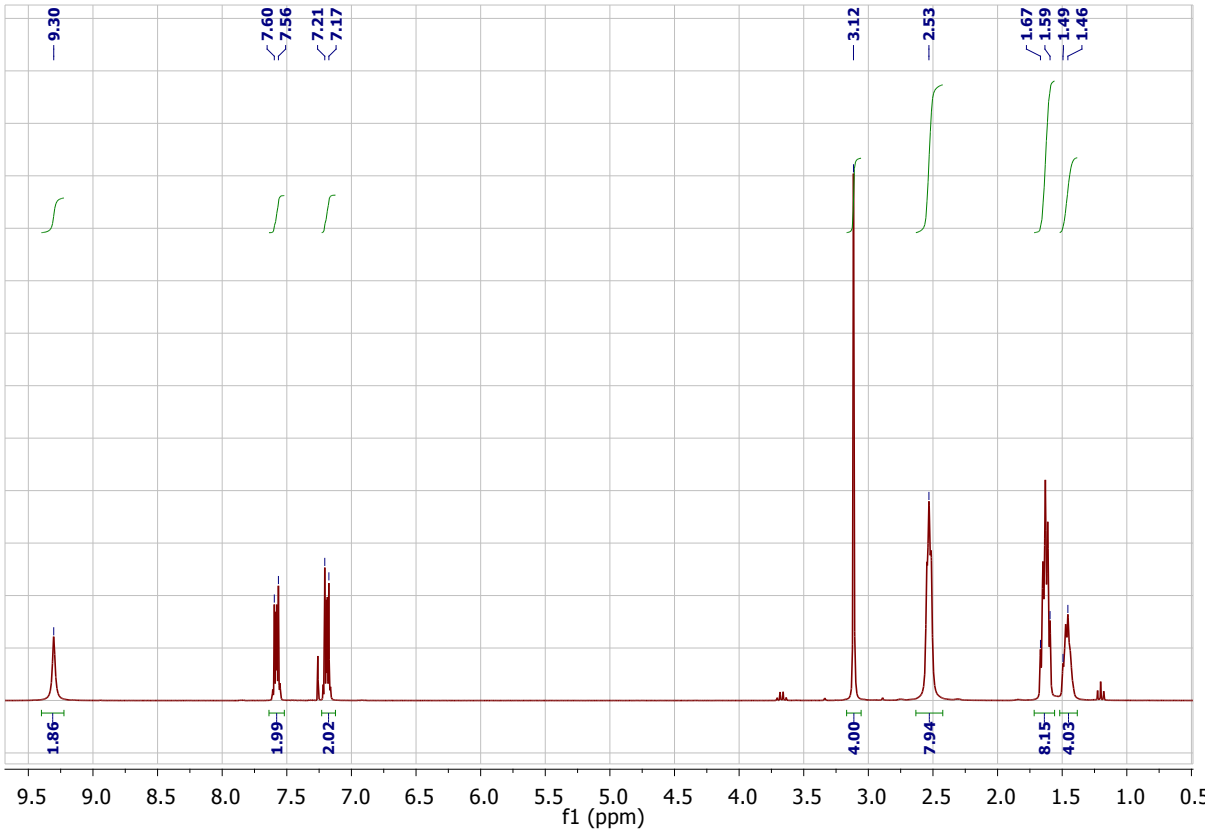

Figure S14 ^{1}H NMR spectrum (300 MHz) of N,N'-(1,2-phenylene)bis(2-(piperidin-1-yl)acetamide) in $CDCl_3$ at 300 K.

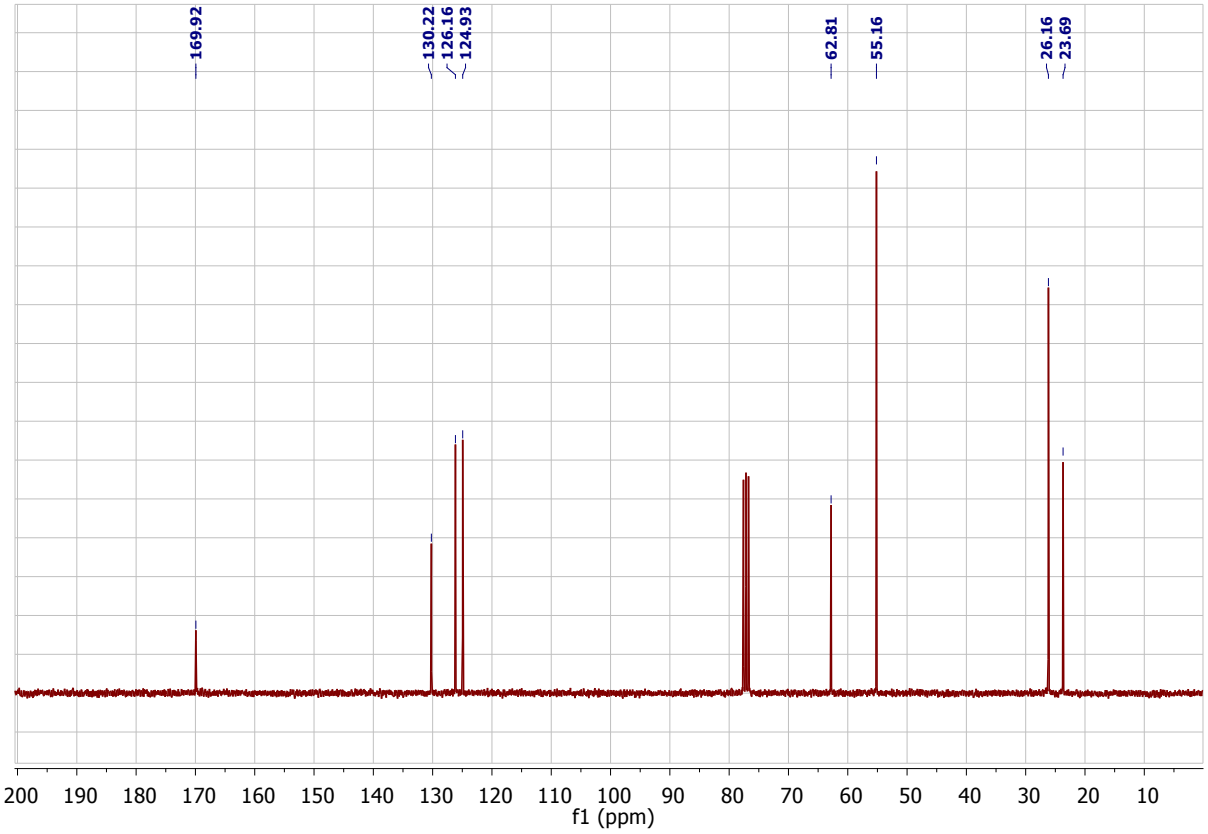

Figure S15 ^{13}C NMR spectrum (101 MHz) of N,N'-(1,2-phenylene)bis(2-(piperidin-1-yl)acetamide) in $CDCl_3$ at 300 K.

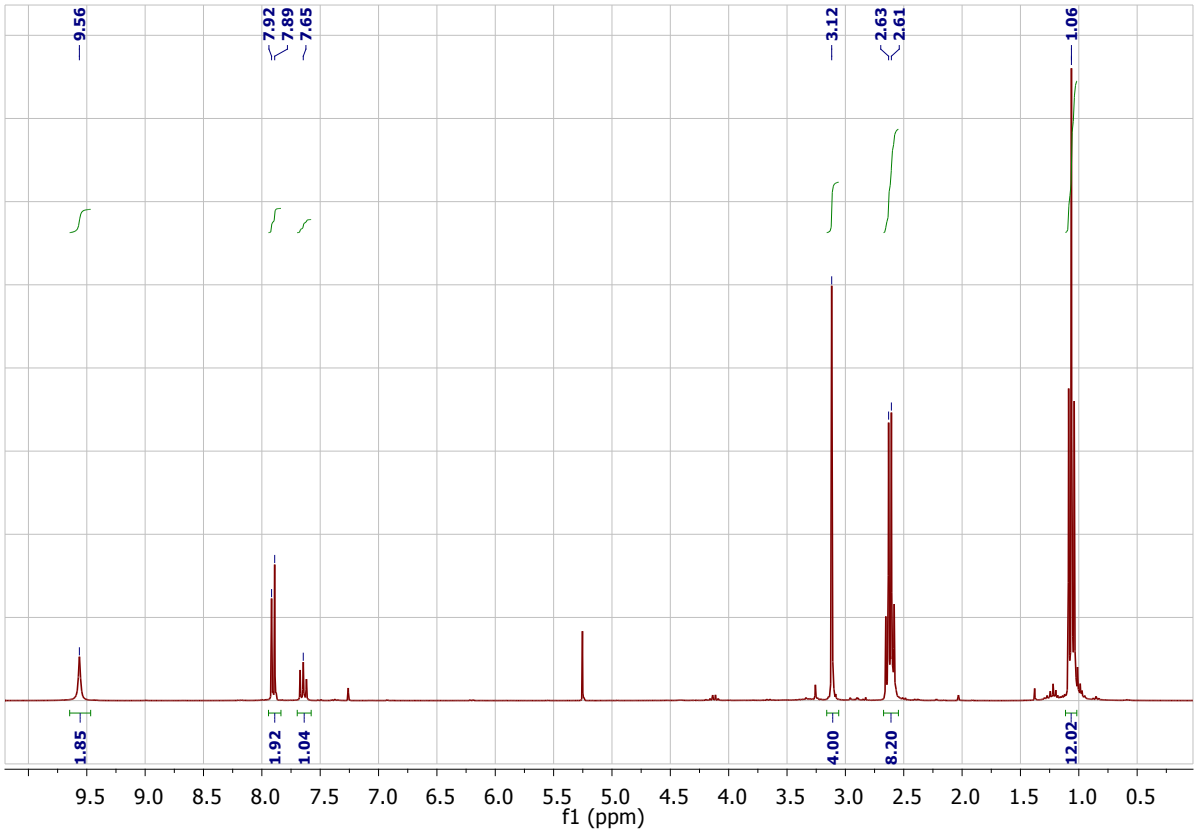

Figure S16 ^{1}H NMR spectrum (300 MHz) of N,N'-(pyridine-2,6-diyl)bis(2-(diethylamino)acetamide) in $CDCl_3$ at 300 K.

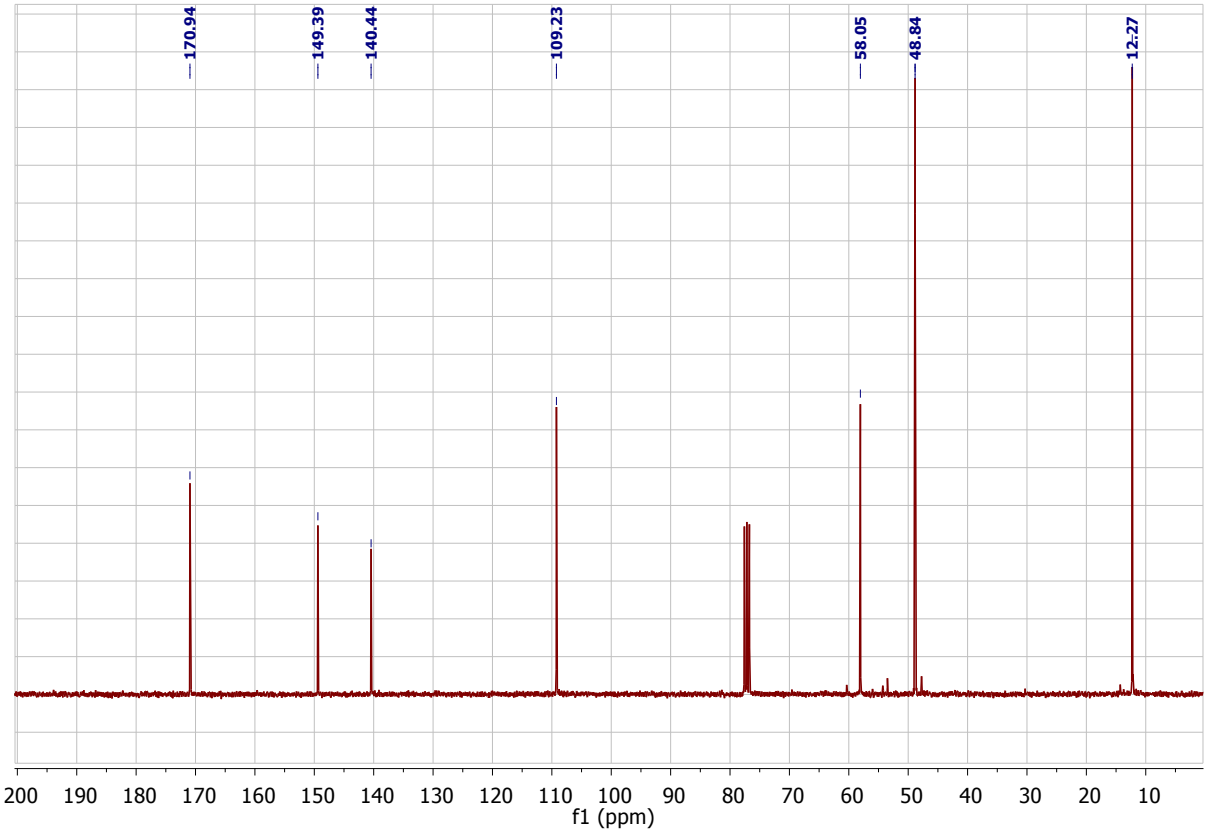

Figure S17 ^{13}C NMR spectrum (101 MHz) of N,N'-(pyridine-2,6-diyl)bis(2-(diethylamino)acetamide) in $CDCl_3$ at 300 K.

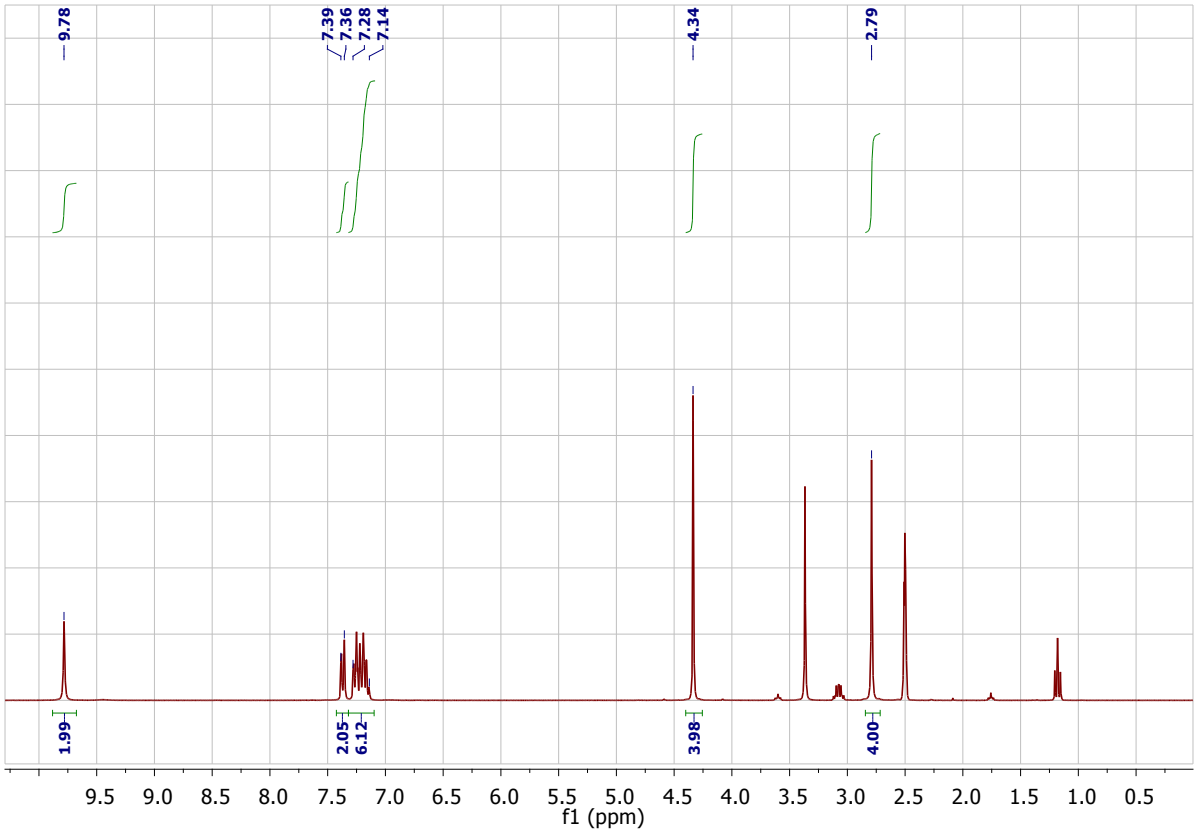

Figure S18 ^{1}H NMR spectrum (300 MHz) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-chloroacetamide) in DMSO-d_6 at 300 K.

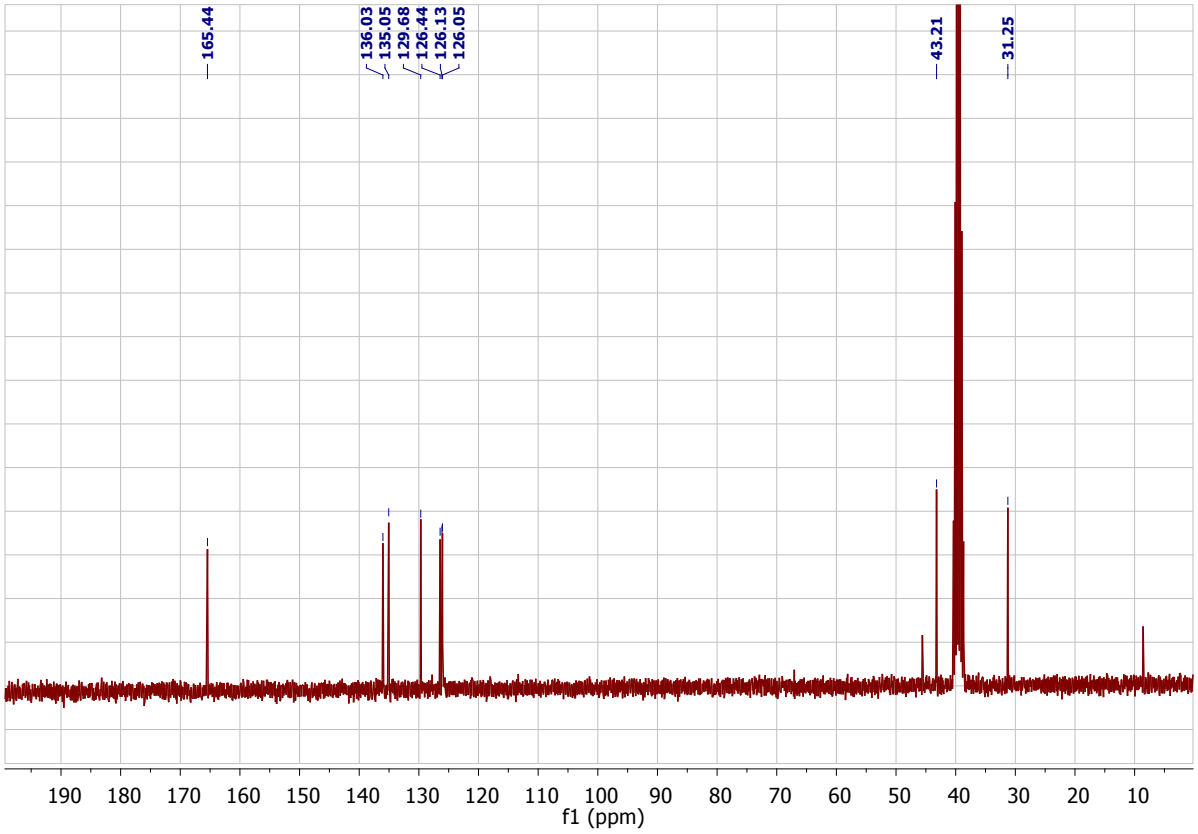

Figure S19 ^{13}C NMR spectrum (101 MHz) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-chloroacetamide) in DMSO-d_6 at 300 K.

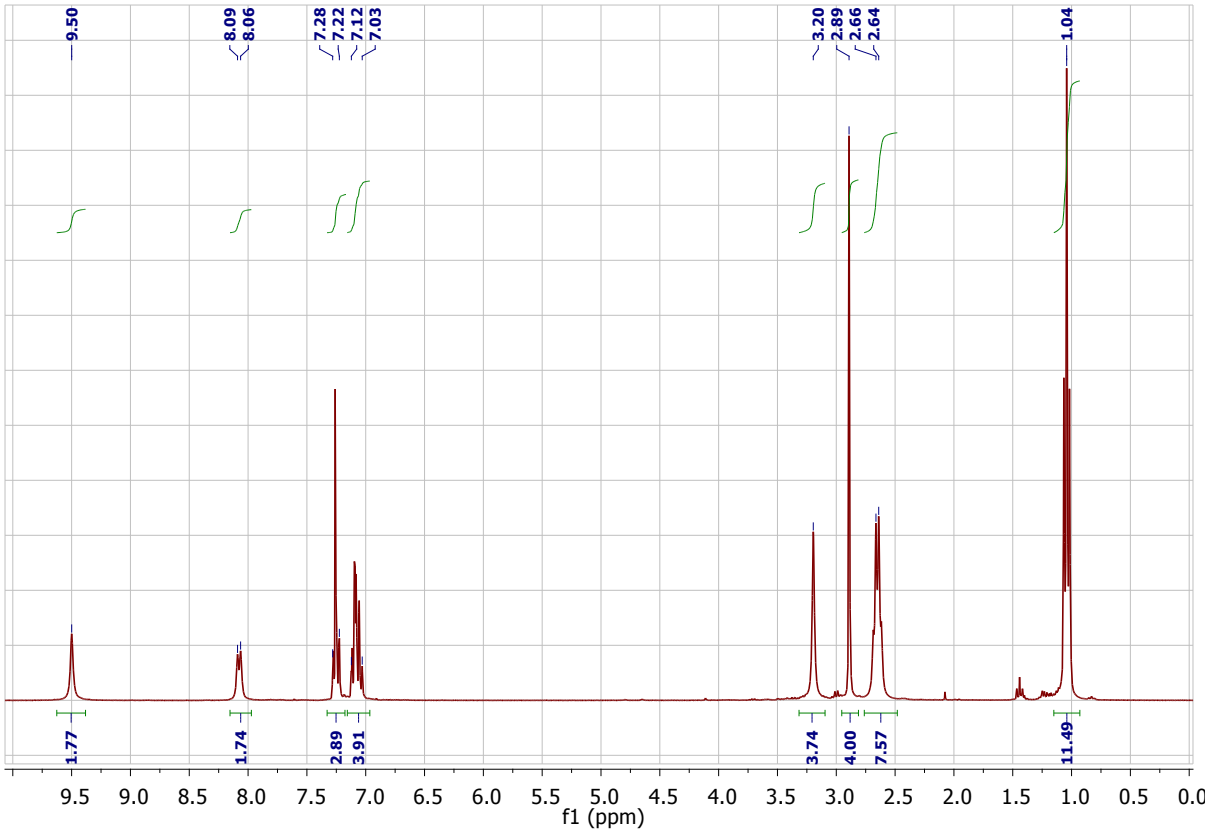

Figure S20 ^{1}H NMR spectrum (300 MHz) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diethylamino)acetamide) in $CDCl_3$ at 300 K.

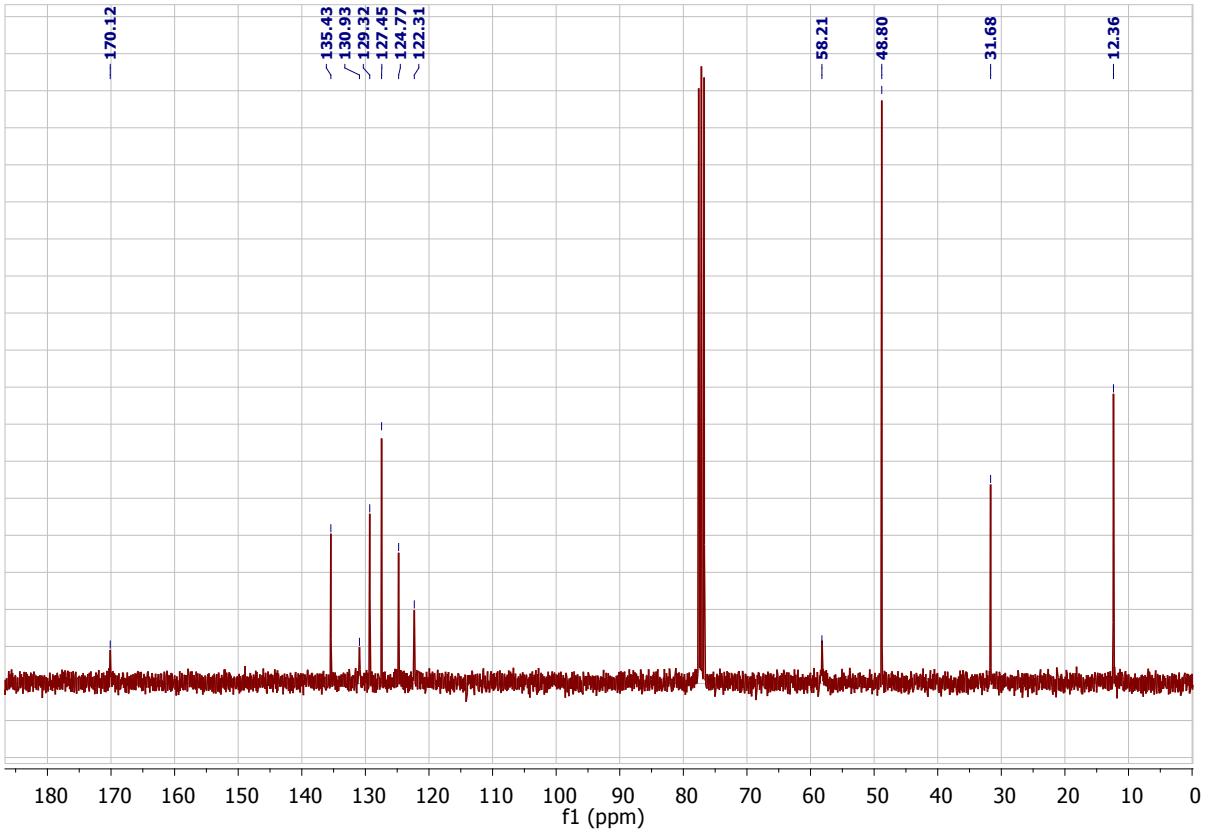

Figure S21 ^{13}C NMR spectrum (101 MHz) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diethylamino)acetamide) in $CDCl_3$ at 300 K.

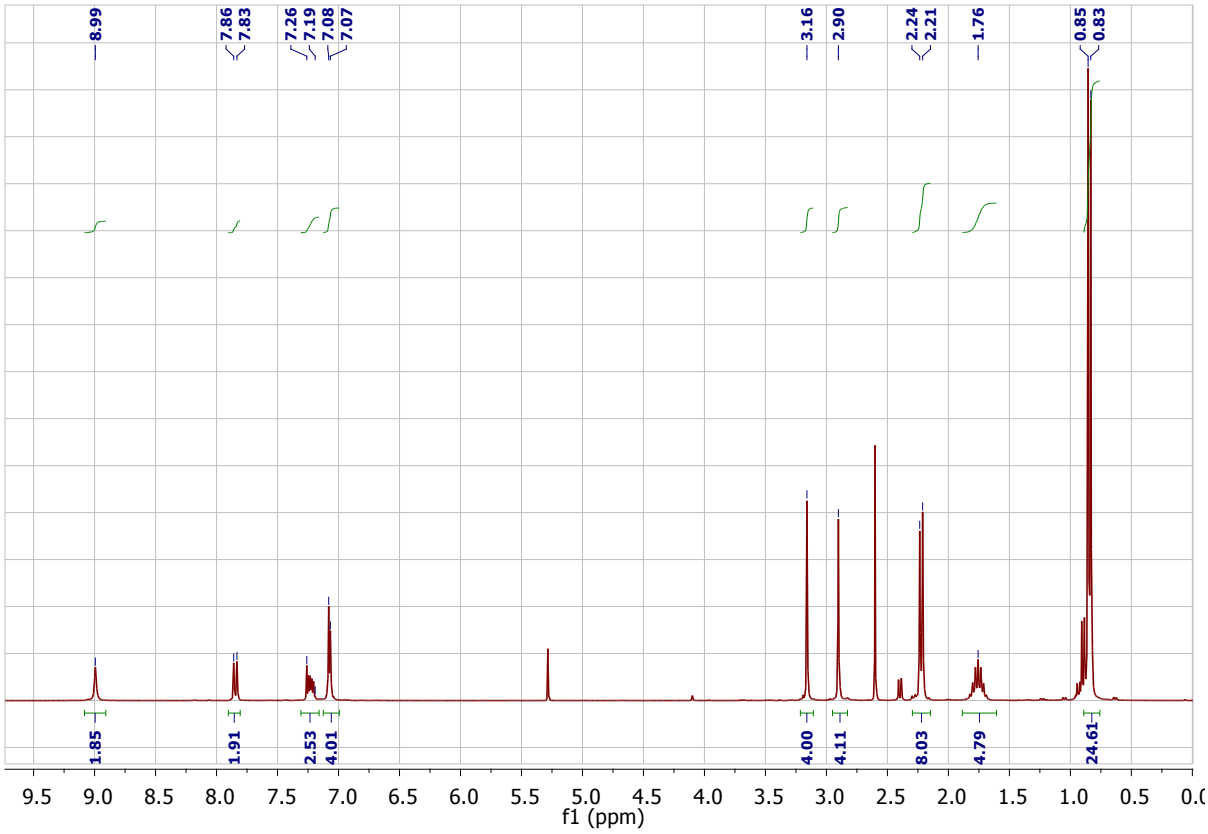

Figure S22 ^{1}H NMR spectrum (300 MHz) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diisobutylamino)acetamide) in $CDCl_3$ at 300 K.

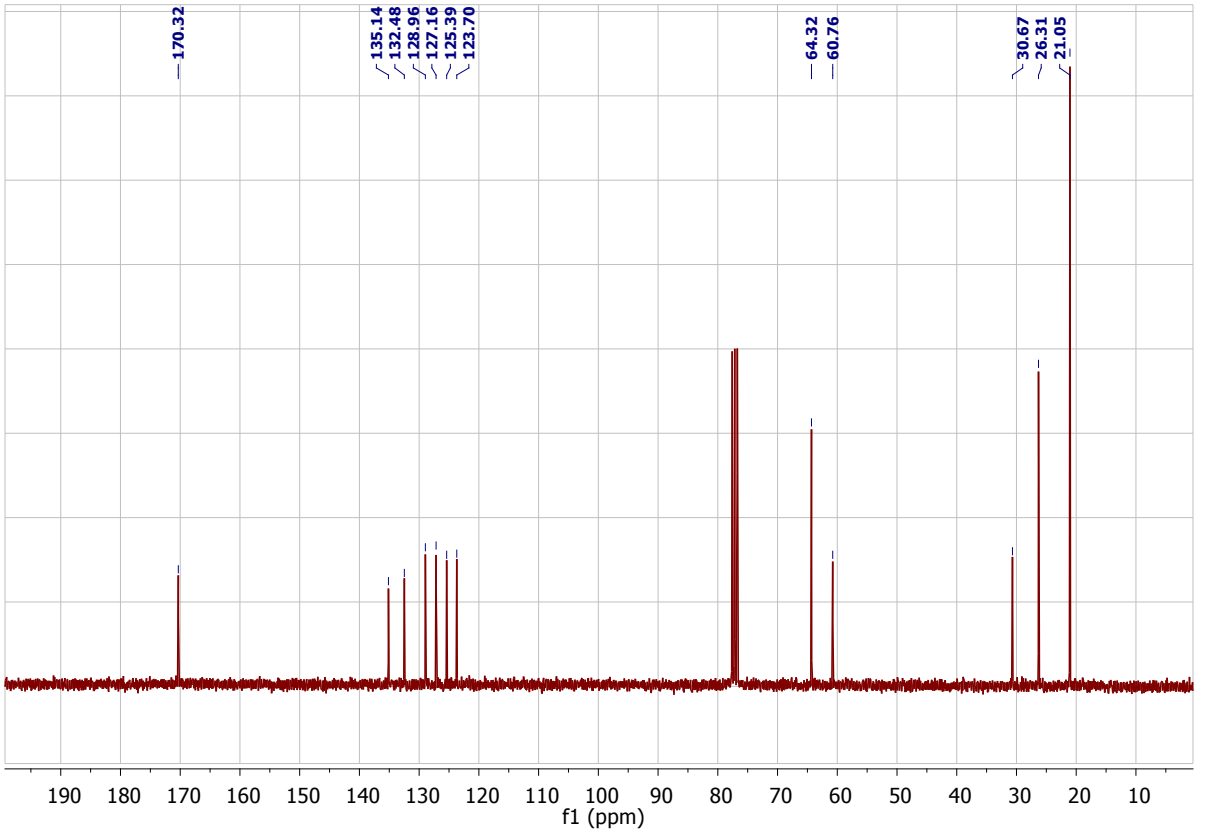

Figure S23 ^{13}C NMR spectrum (101 MHz) of N,N'-(ethane-1,2-diylbis(2,1-phenylene))bis(2-(diisobutylamino)acetamide) in $CDCl_3$ at 300 K.

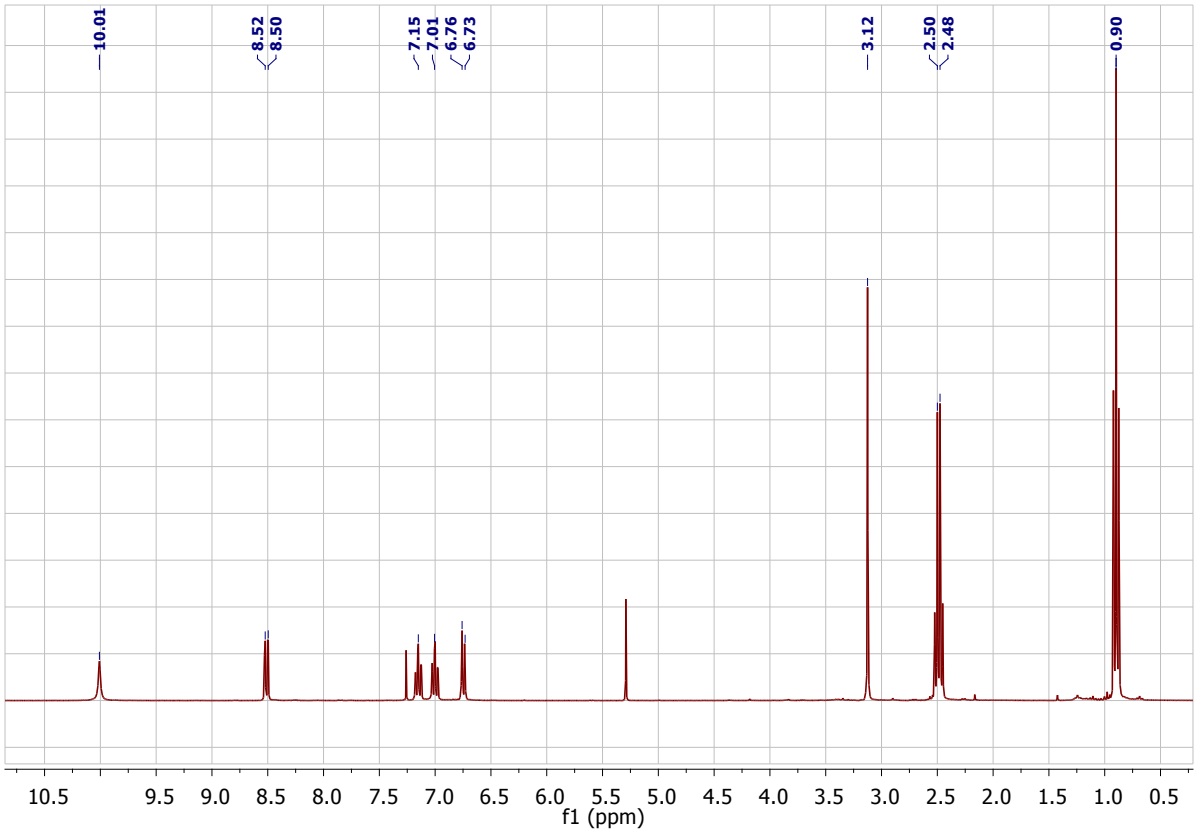

Figure S24 ^{1}H NMR spectrum (300 MHz) of N,N'-(oxybis(2,1-phenylene))bis(2-(diethylamino)acetamide) in $CDCl_3$ at 300 K.

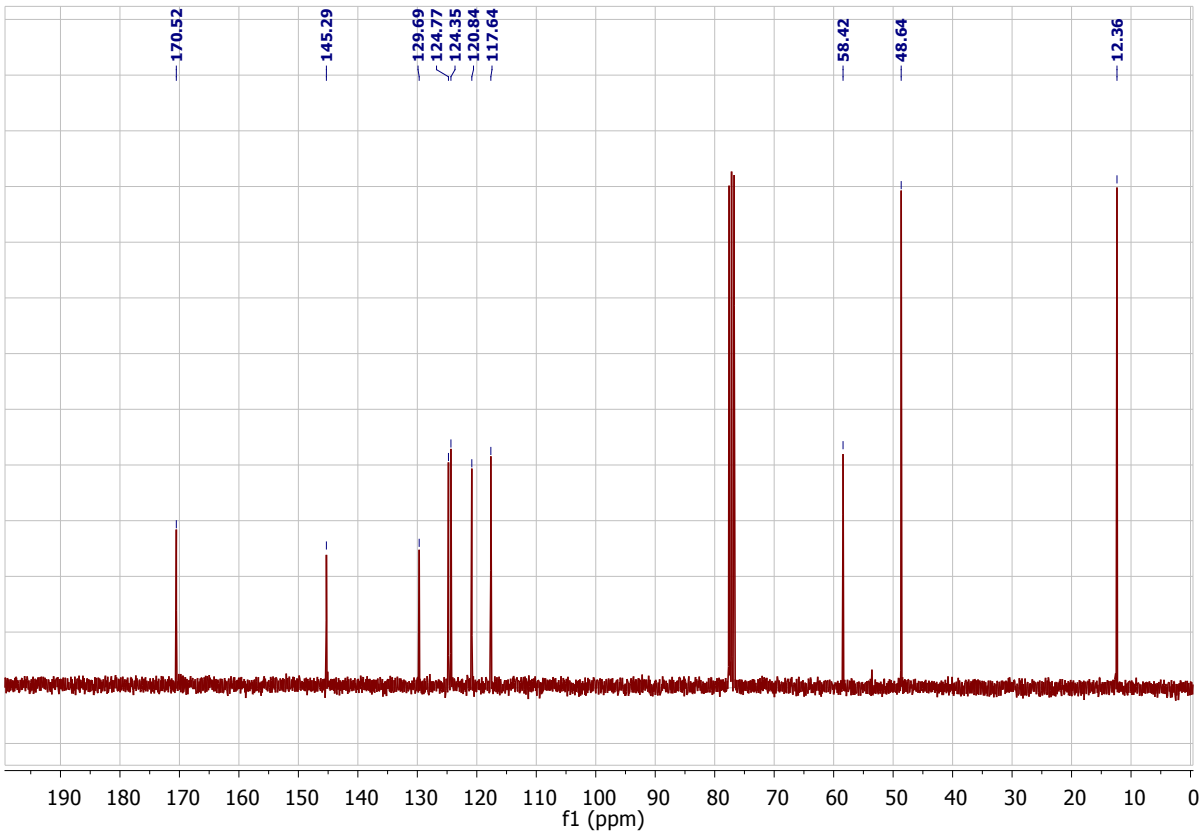

Figure S25 ^{13}C NMR spectrum (101 MHz) of N,N'-(oxybis(2,1-phenylene))bis(2-(diethylamino)acetamide) in $CDCl_3$ at 300 K.

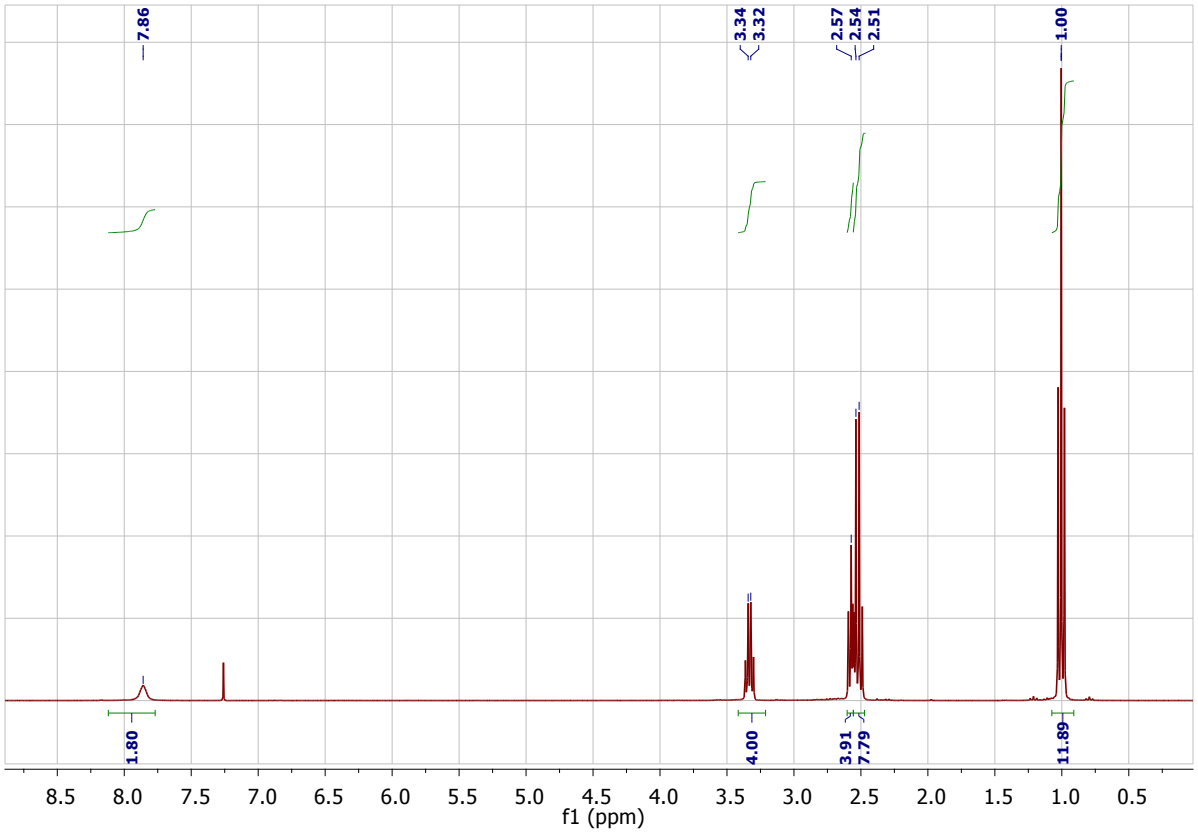

Figure S26 ^{1}H NMR spectrum (300 MHz) of N^1,N^2-bis(2-(diethylamino)ethyl)oxalamide in $CDCl_3$ at 300 K.

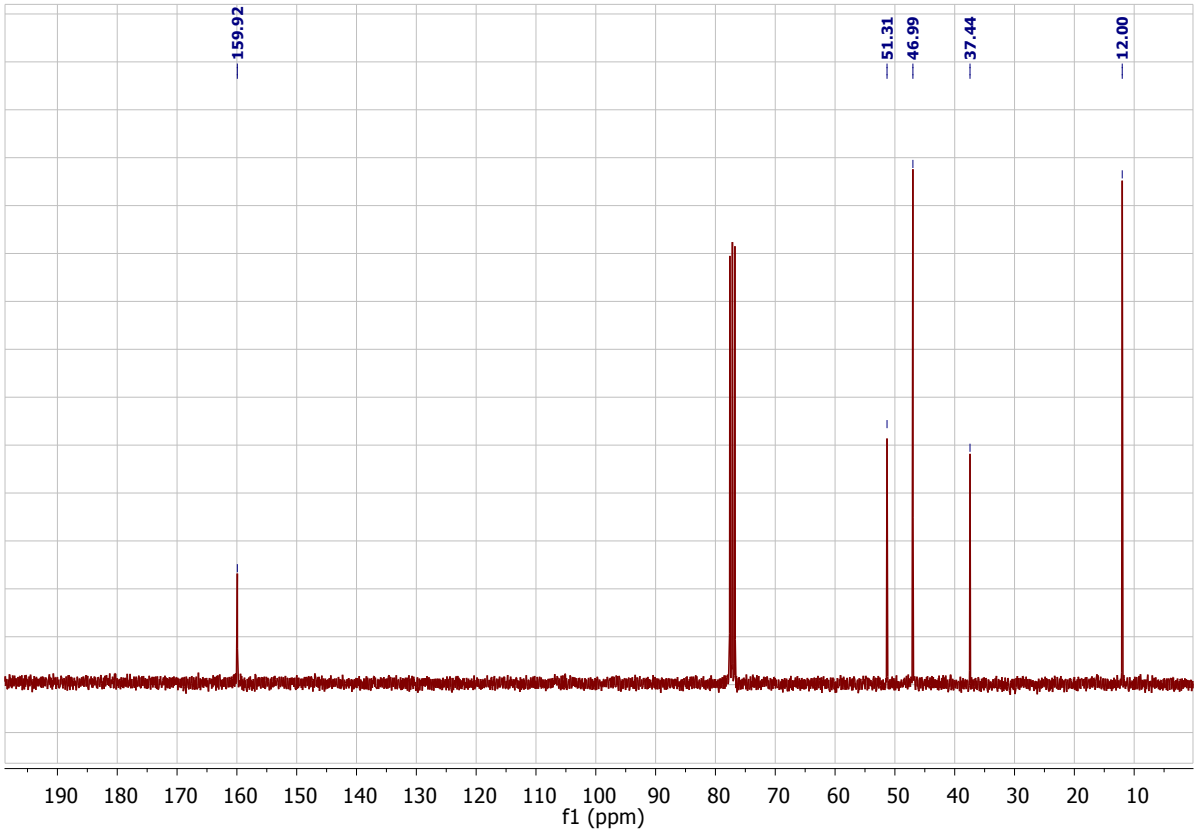

Figure S27 ^{13}C NMR spectrum (101 MHz) of N^1,N^2-bis(2-(diethylamino)ethyl)oxalamide in $CDCl_3$ at 300 K.

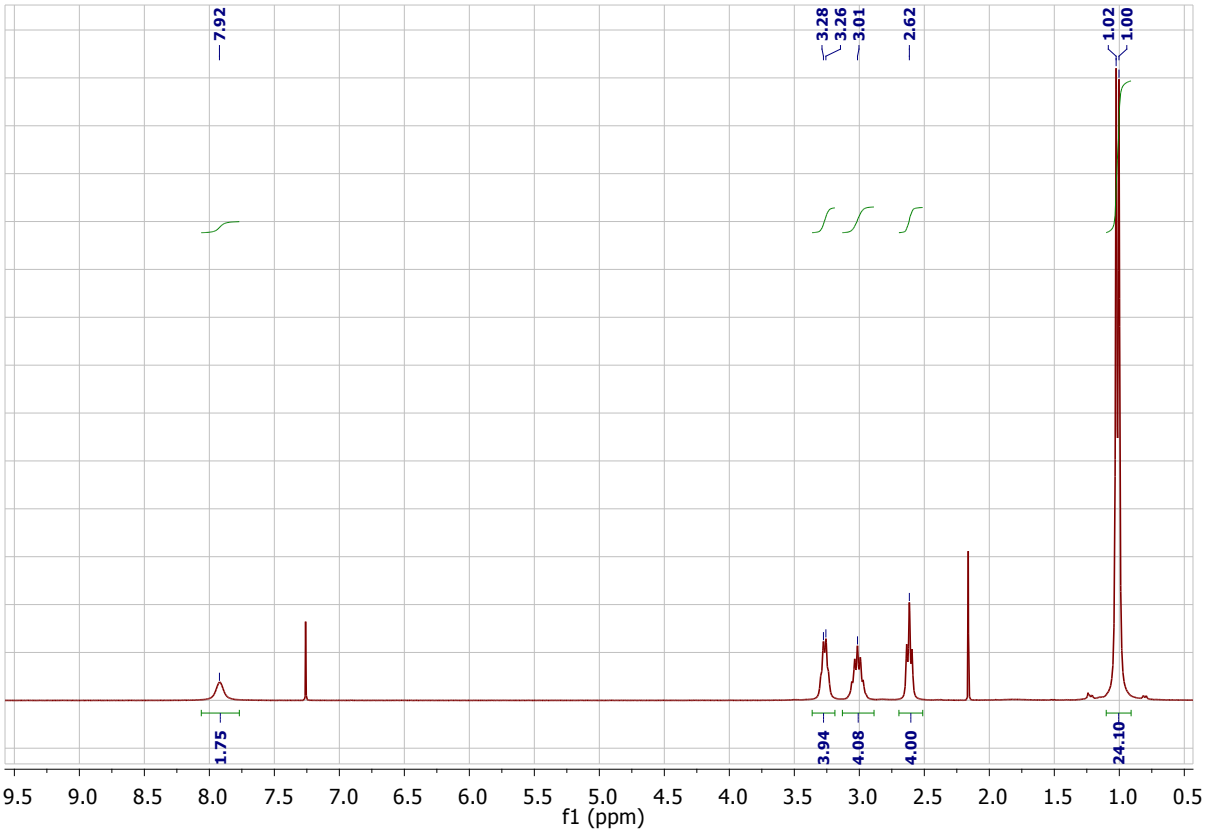

Figure S28 ^{1}H NMR spectrum (300 MHz) of N^1,N^2-bis(2-(diisopropylamino)ethyl)oxalamide in $CDCl_3$ at 300 K.

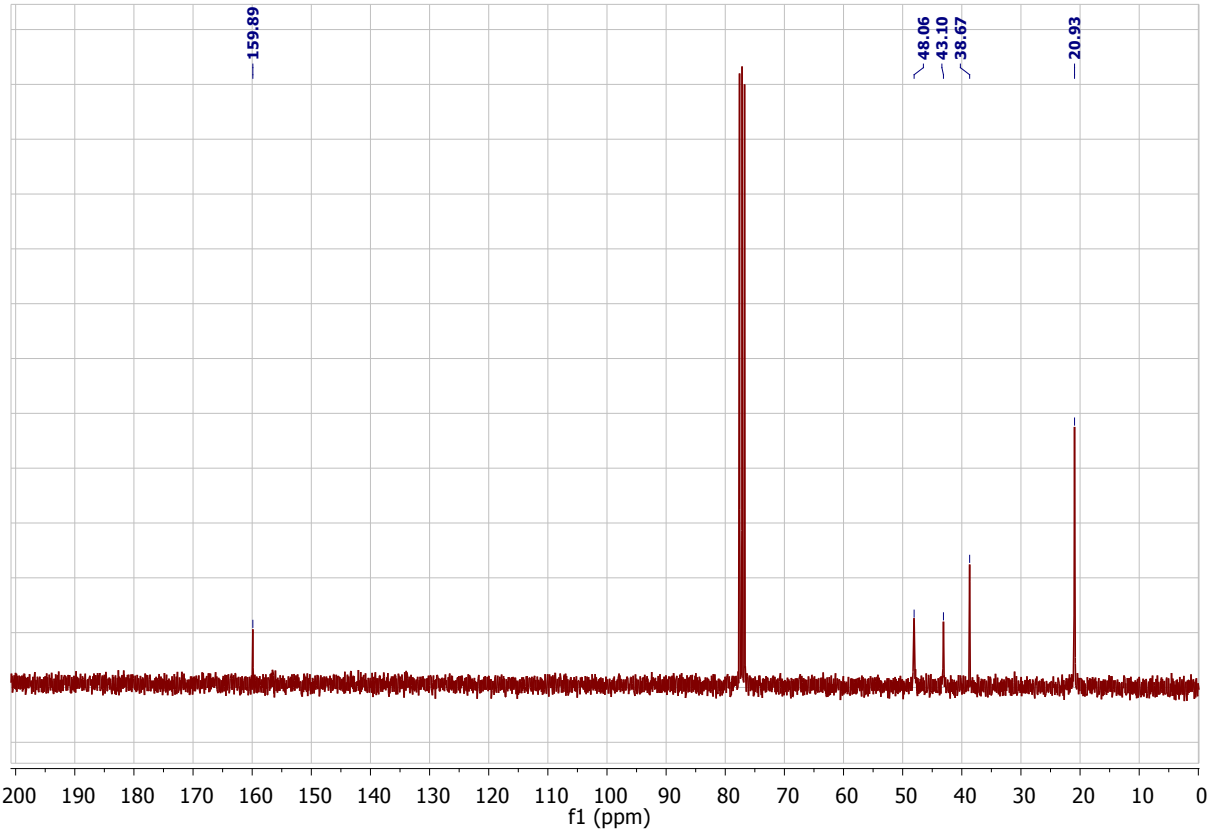

Figure S29 ^{13}C NMR spectrum (101 MHz) of N^1,N^2-bis(2-(diisopropylamino)ethyl)oxalamide in $CDCl_3$ at 300 K.

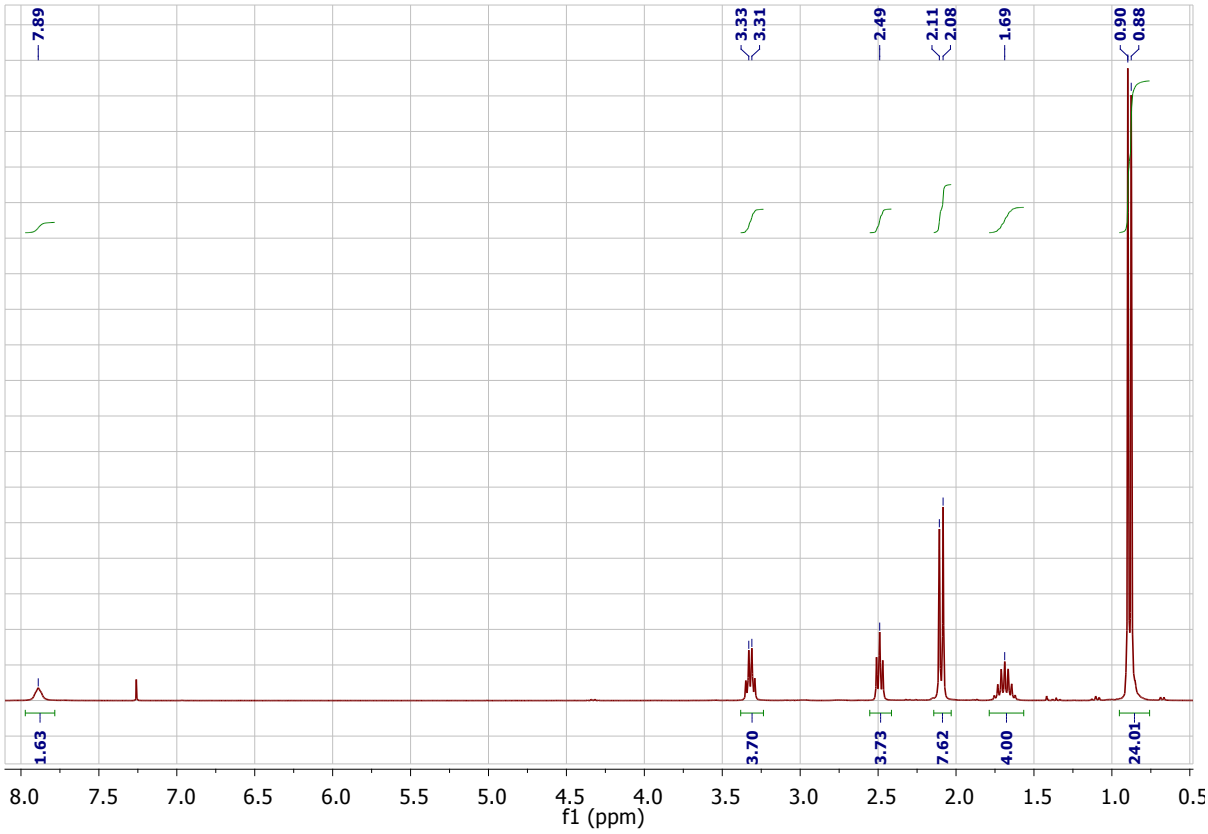

Figure S30 ^{1}H NMR spectrum (300 MHz) of N^1,N^2-bis(2-(diisobutylamino)ethyl)oxalamide in $CDCl_3$ at 300 K.

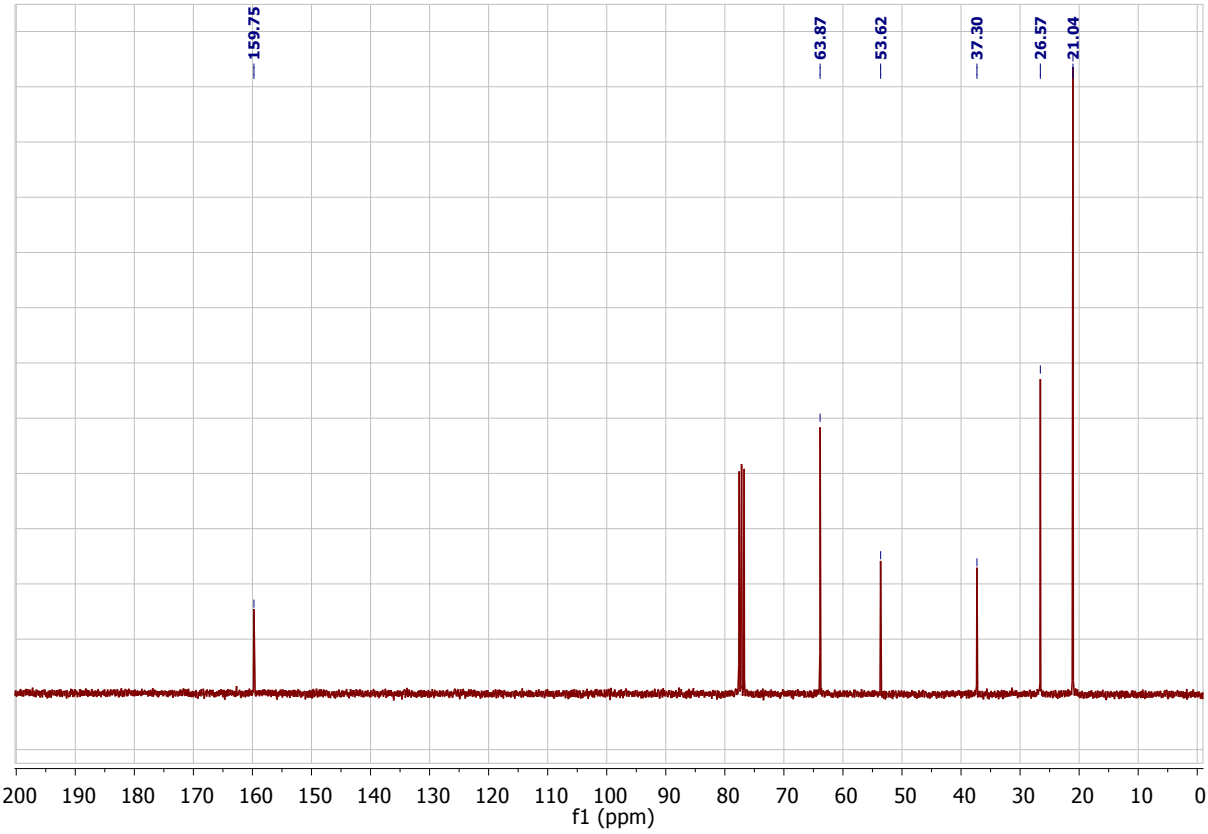

Figure S31 ^{13}C NMR spectrum (101 MHz) of N^1,N^2-bis(2-(diisobutylamino)ethyl)oxalamide in $CDCl_3$ at 300 K.

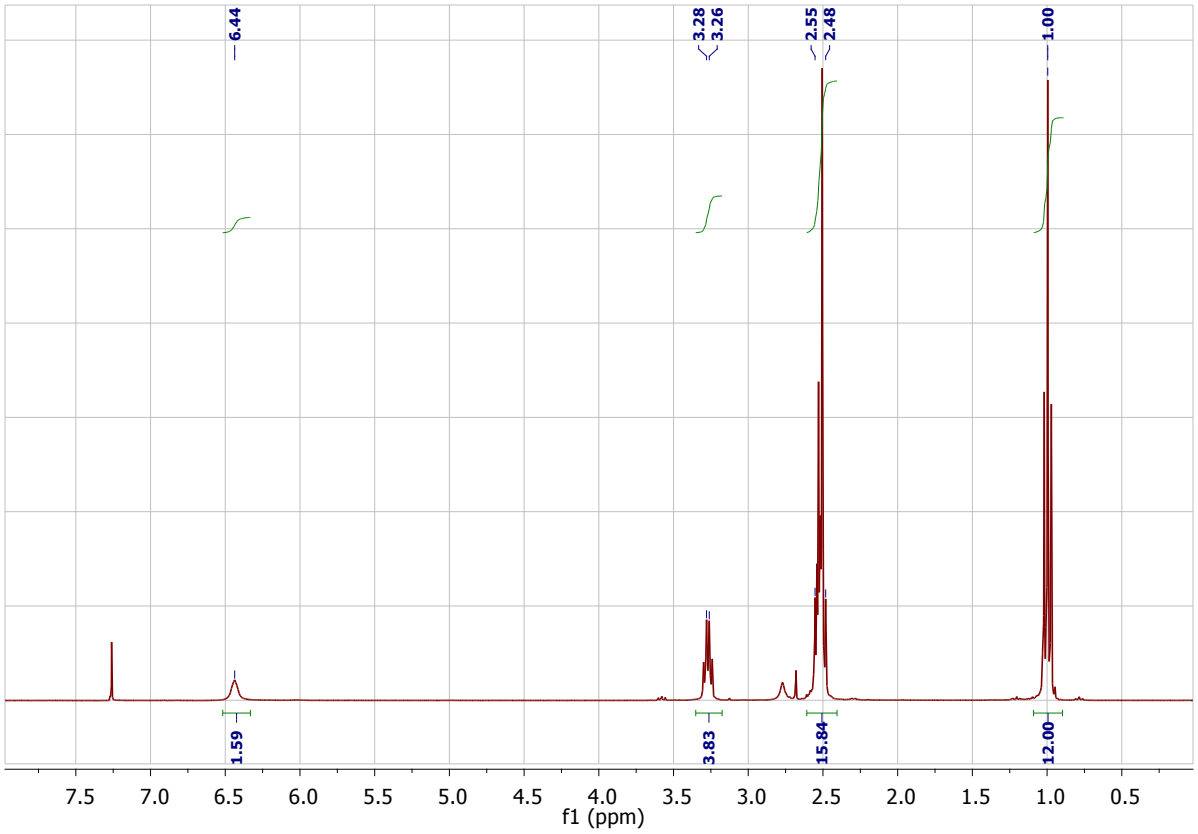

Figure S32 ^{1}H NMR spectrum (300 MHz) of N^1,N^4-bis(2-(diethylamino)ethyl)succinamide in $CDCl_3$ at 300 K.

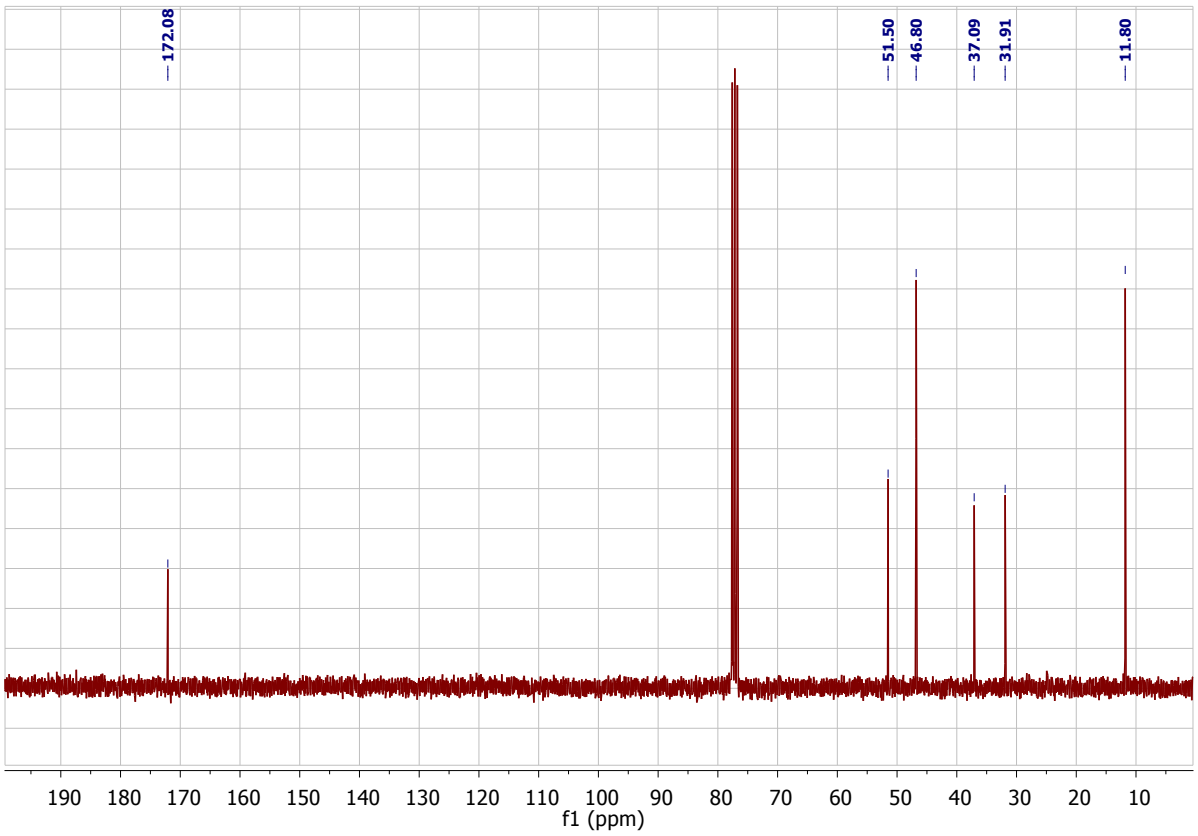

Figure S33 ^{13}C NMR spectrum (101 MHz) of N^1,N^4-bis(2-(diethylamino)ethyl)succinamide in $CDCl_3$ at 300 K.

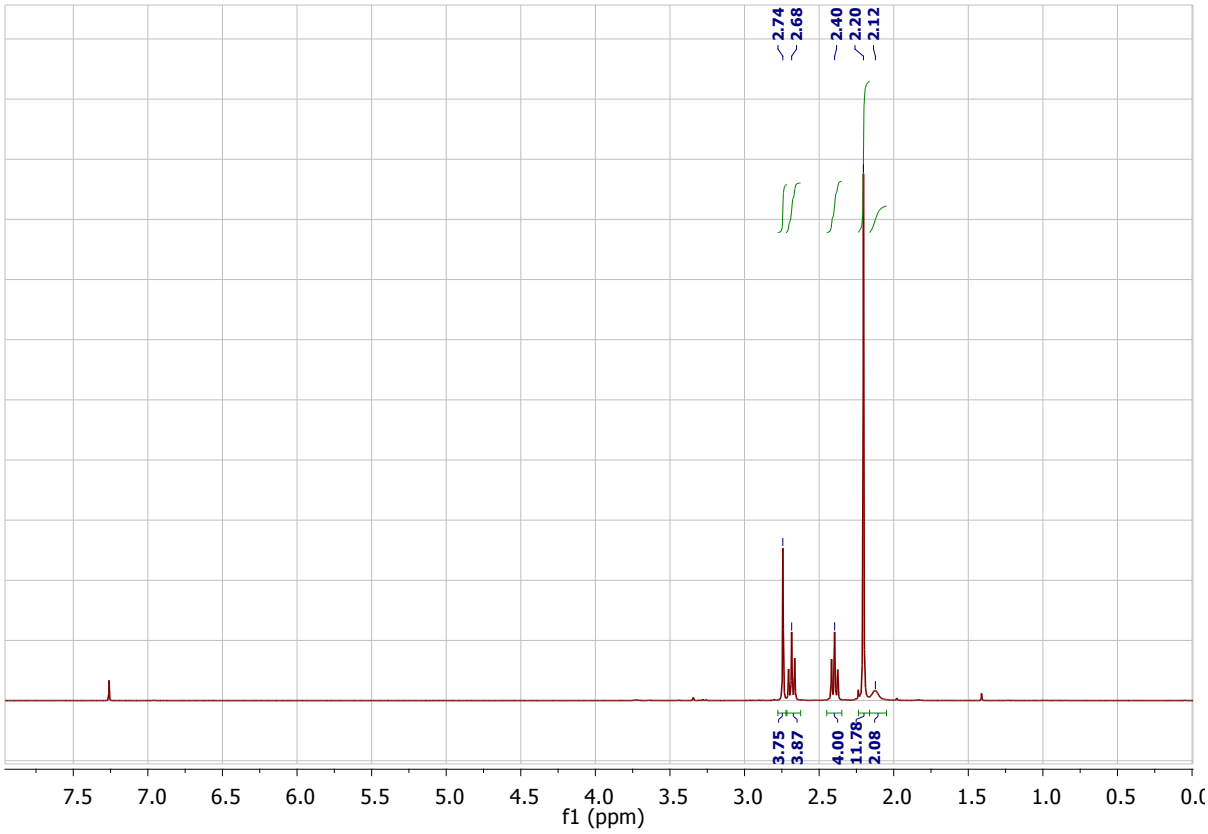

Figure S34 ^{1}H NMR spectrum (400 MHz) of 1a in $CDCl_3$ at 300 K.

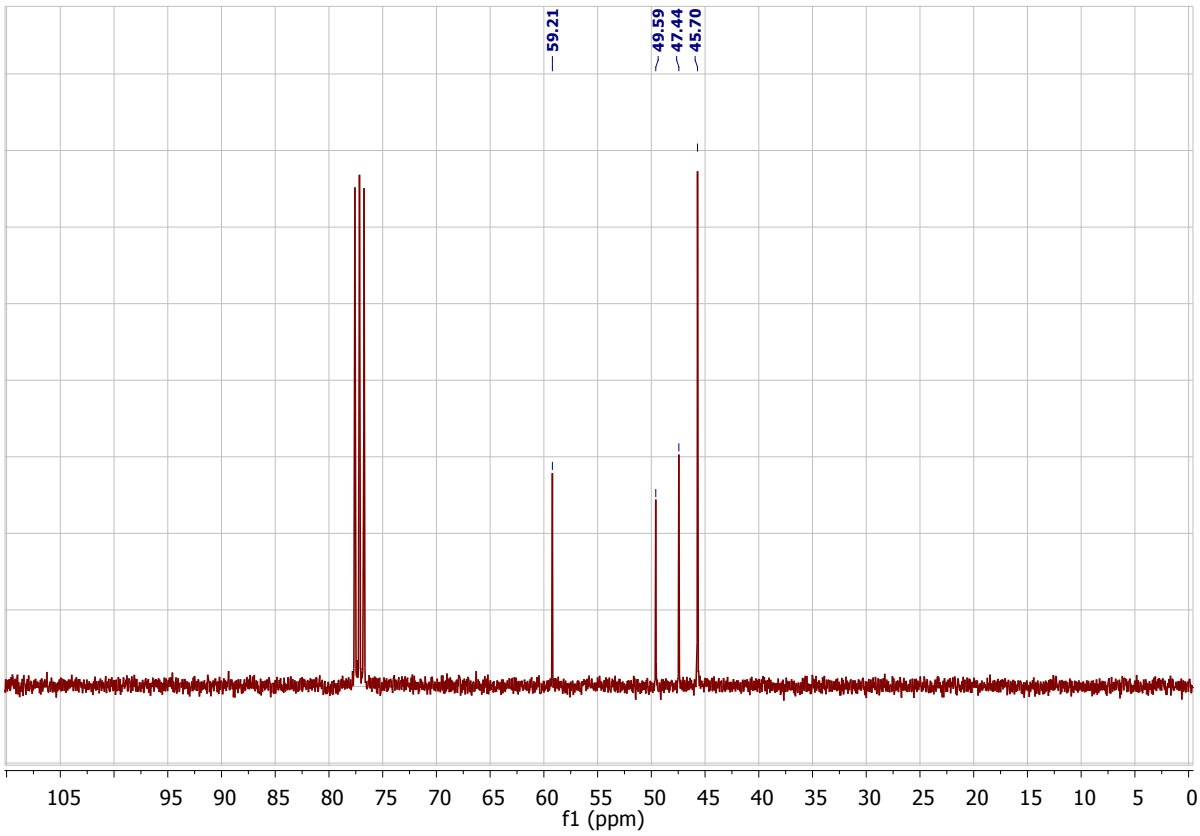

Figure S35 ^{13}C NMR spectrum (101 MHz) of 1a in $CDCl_3$ at 300 K.

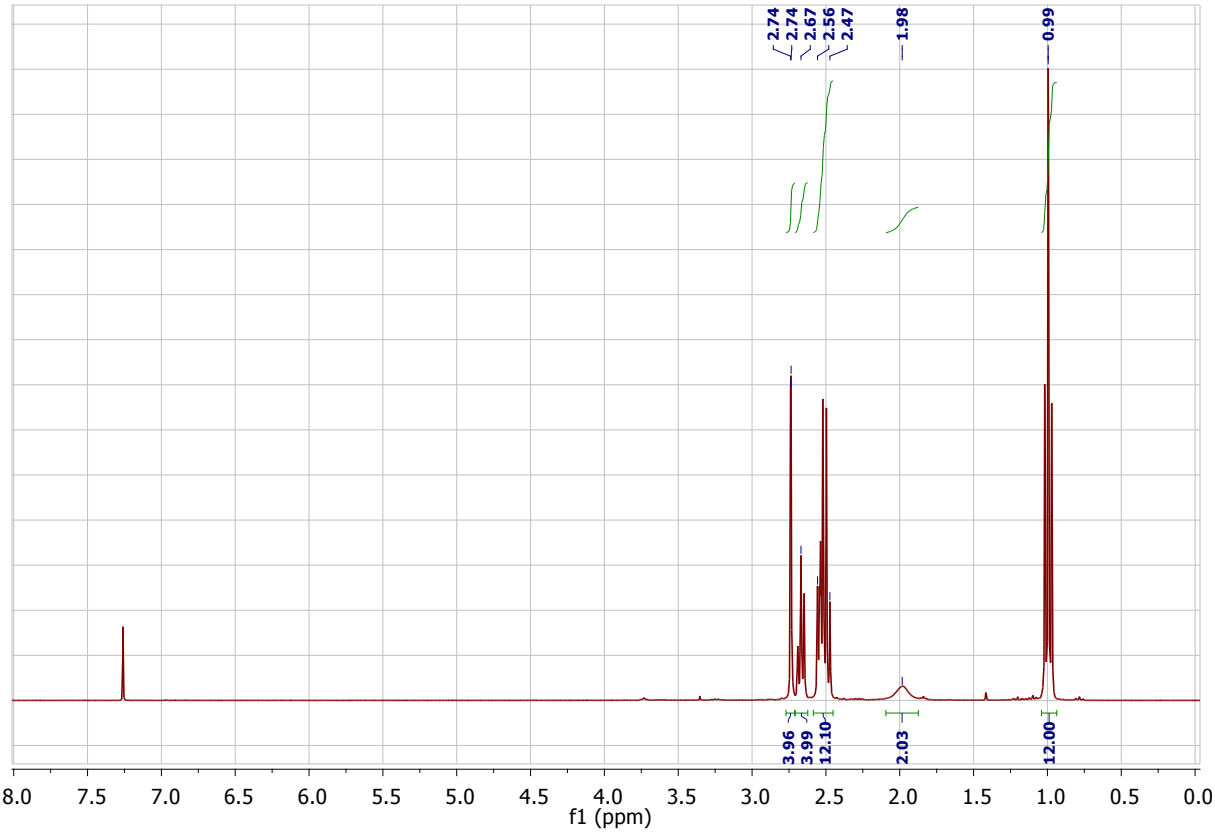

Figure S36 ^{1}H NMR spectrum (400 MHz) of 1b in $CDCl_3$ at 300 K.

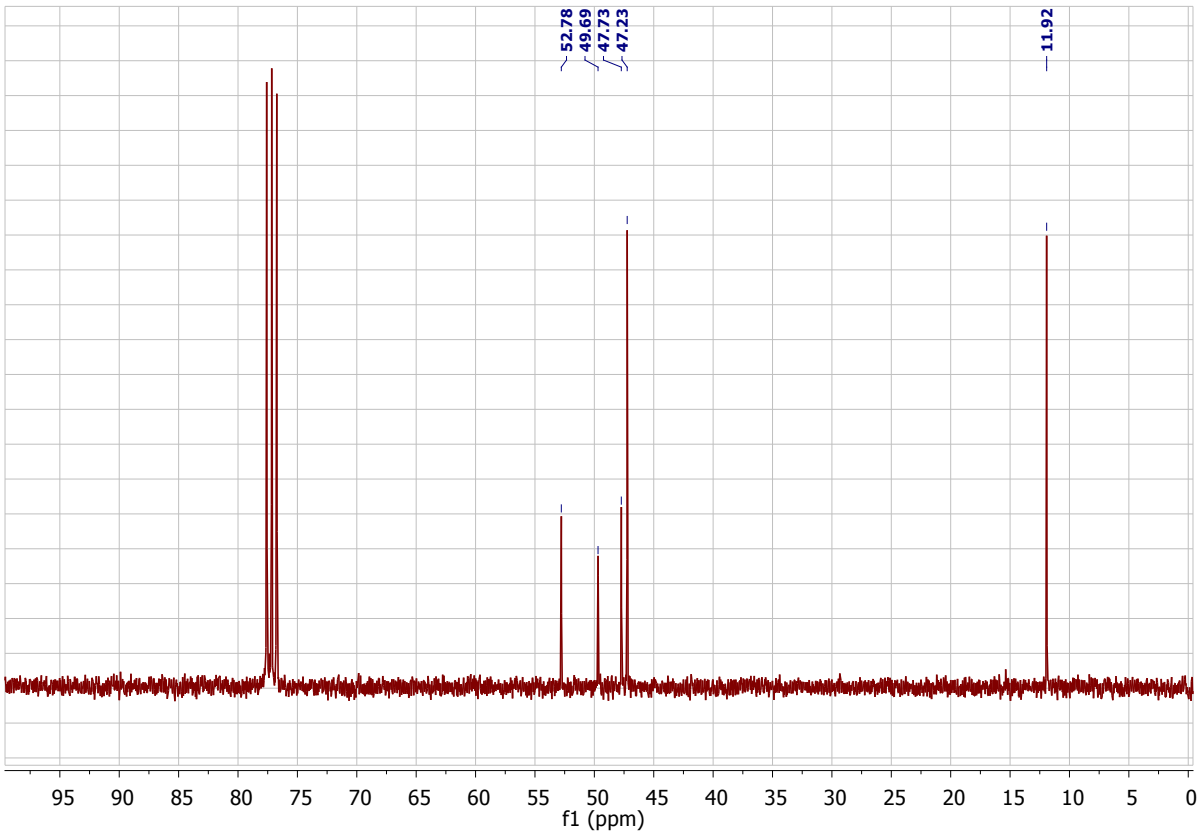

Figure S37 ^{13}C NMR spectrum (101 MHz) of 1b in $CDCl_3$ at 300 K.

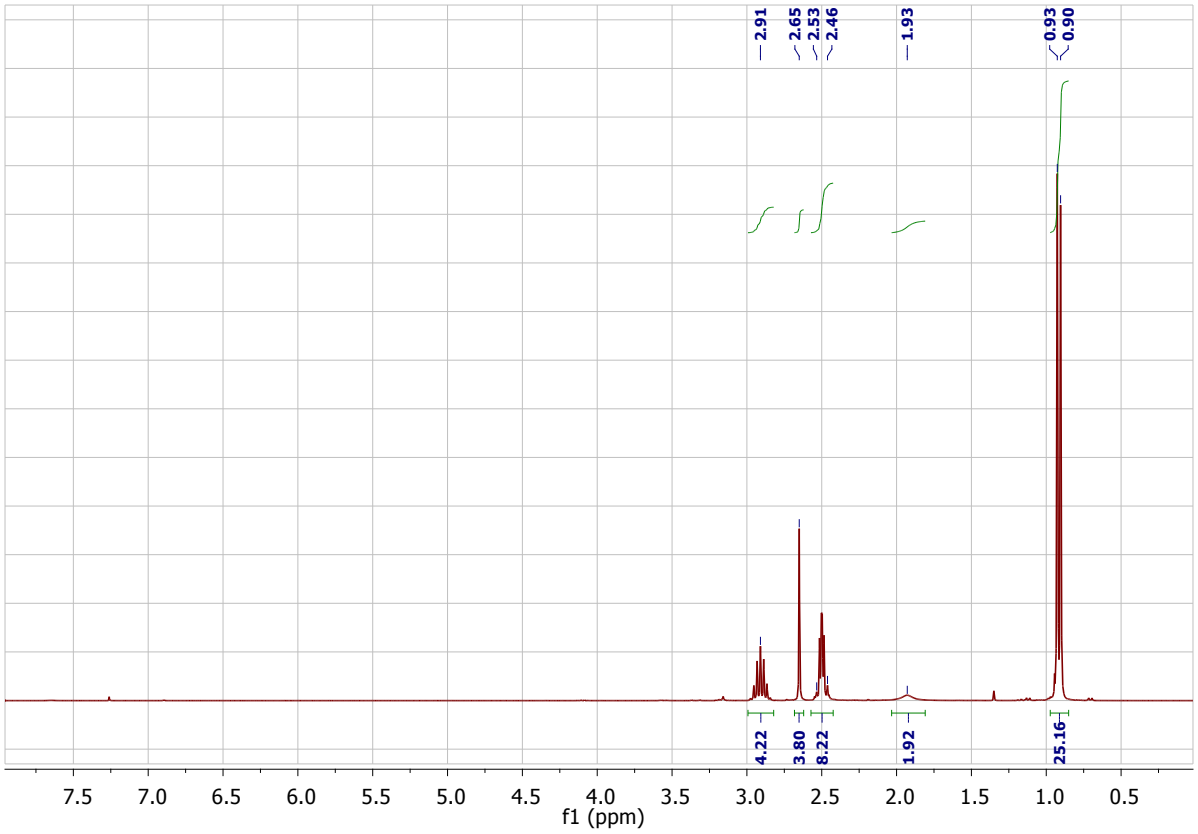

Figure S38 ^{1}H NMR spectrum (400 MHz) of 1c in $CDCl_3$ at 300 K.

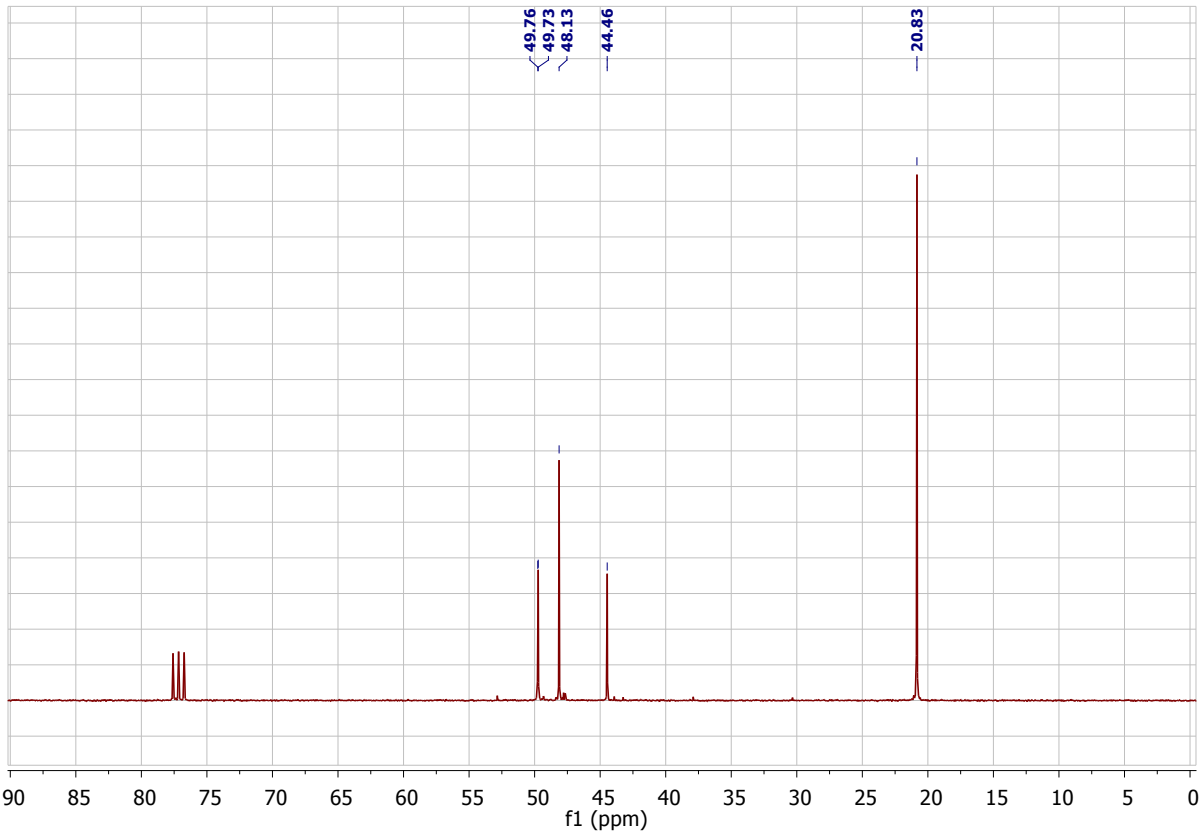

Figure S39 ^{13}C NMR spectrum (101 MHz) of 1c in $CDCl_3$ at 300 K.

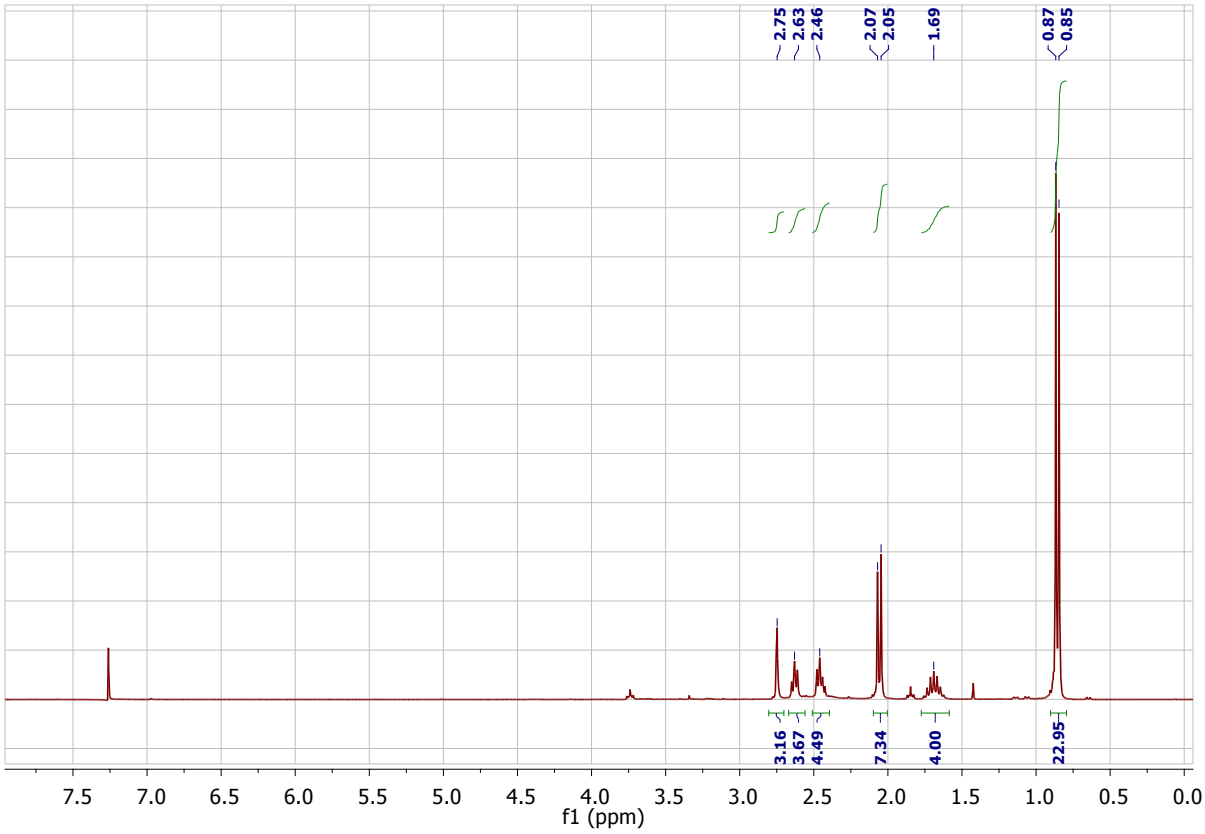

Figure S40 ^{1}H NMR spectrum (400 MHz) of 1d in $CDCl_3$ at 300 K.

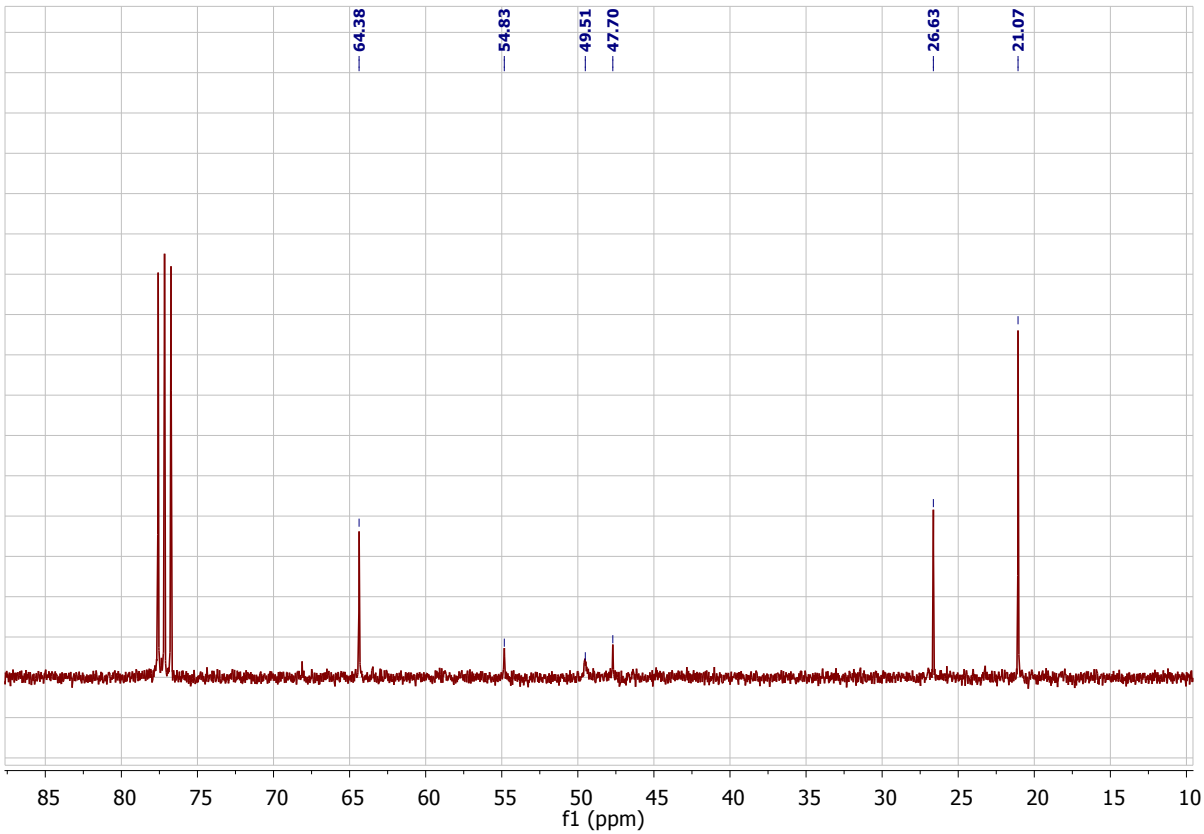

Figure S41 ^{13}C NMR spectrum (101 MHz) of 1d in $CDCl_3$ at 300 K.

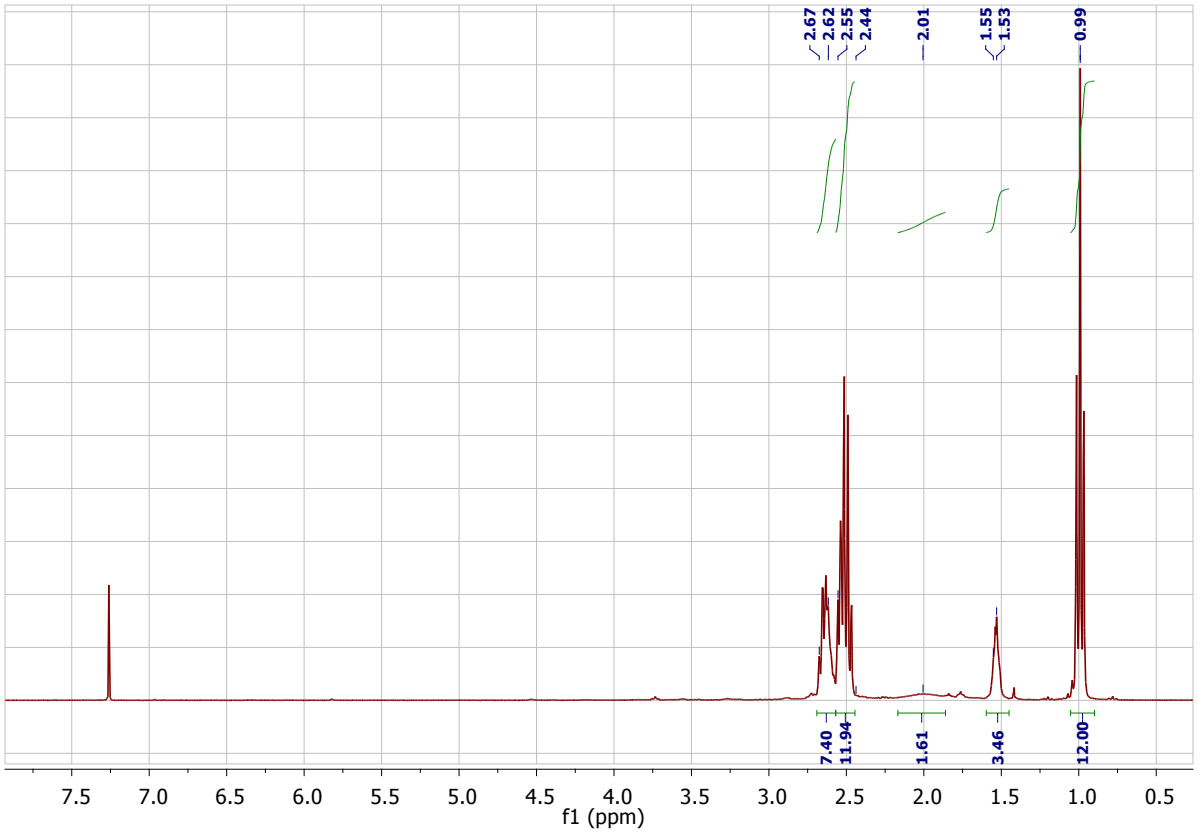

Figure S42 ^{1}H NMR spectrum (400 MHz) of 1e in $CDCl_3$ at 300 K.

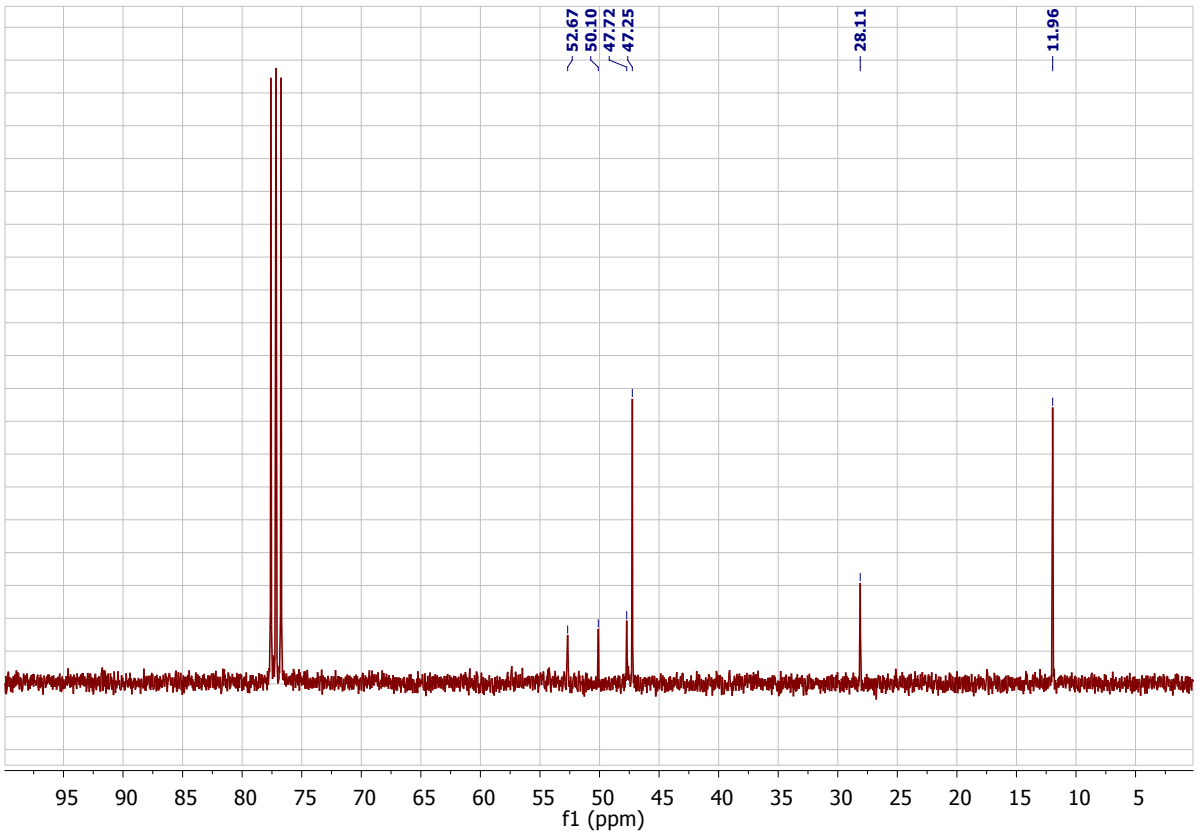

Figure S43 ^{13}C NMR spectrum (101 MHz) of 1e in $CDCl_3$ at 300 K.

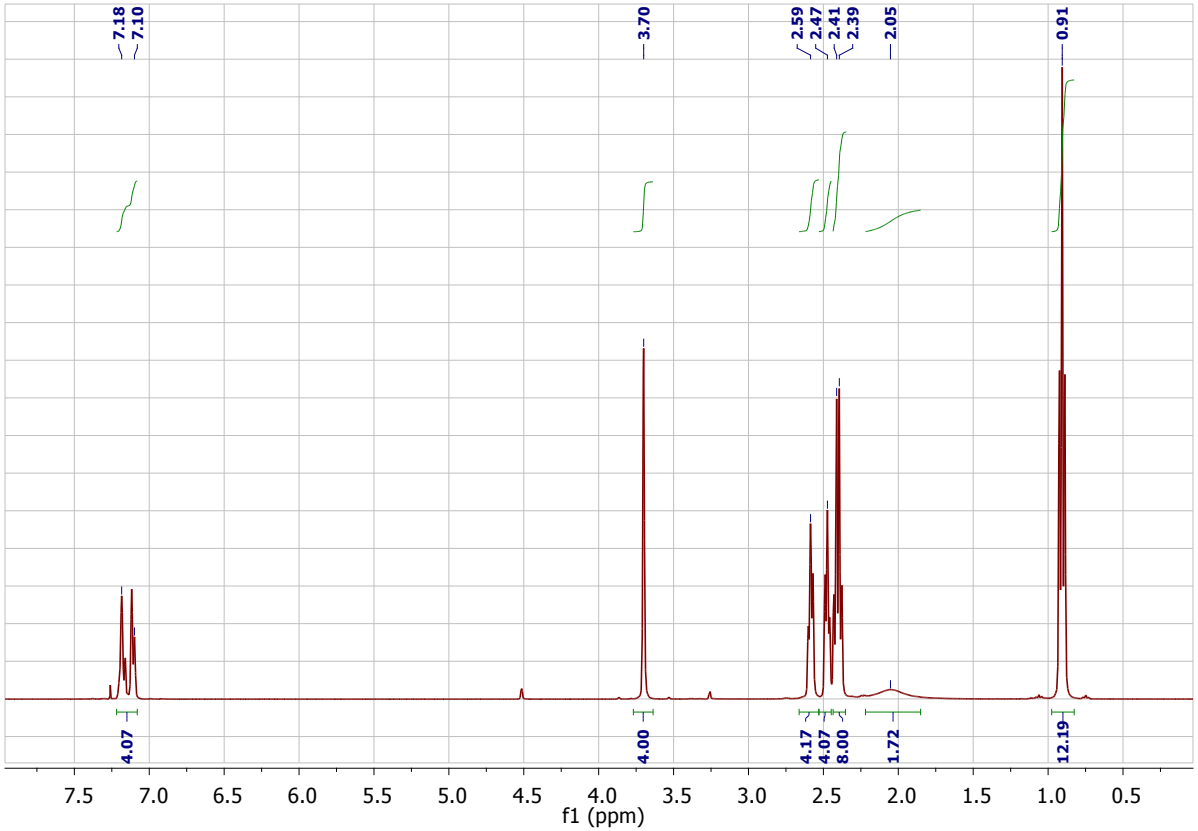

Figure S44 ^{1}H NMR spectrum (400 MHz) of 1f in $CDCl_3$ at 300 K.

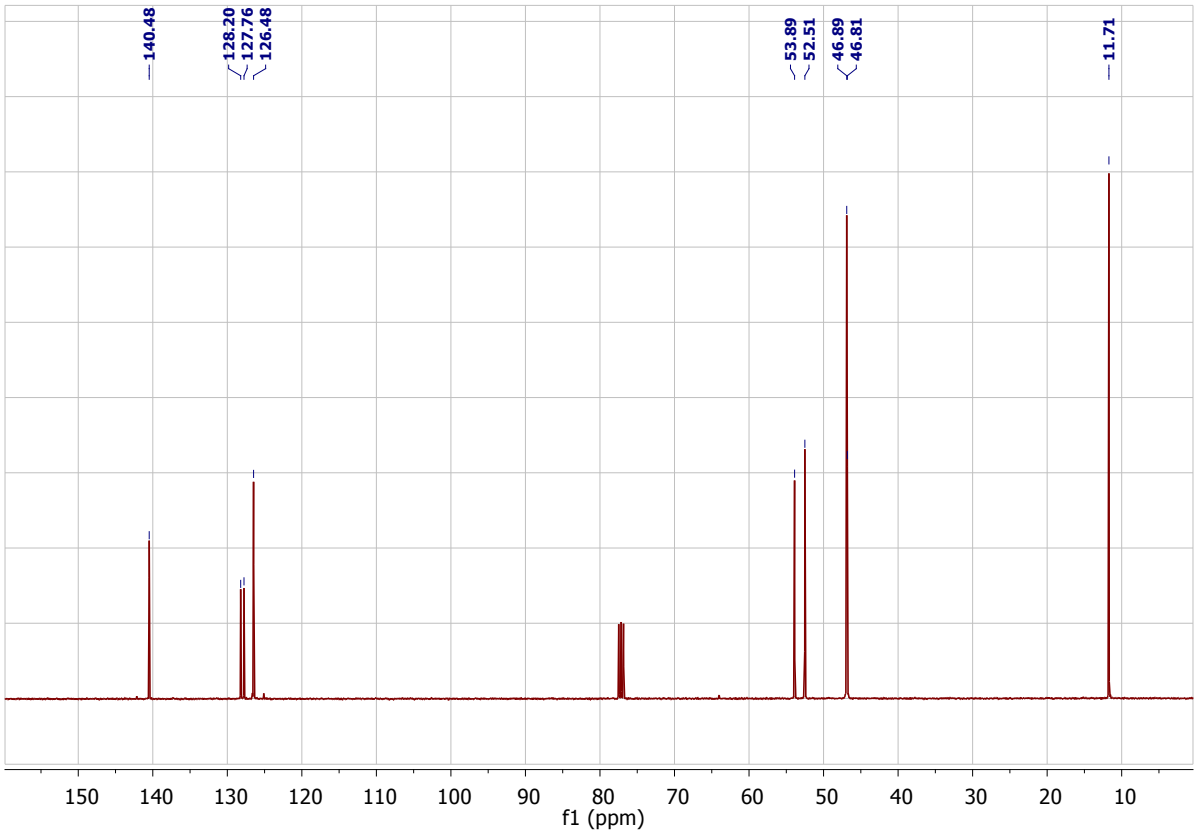

Figure S45 ^{13}C NMR spectrum (101 MHz) of 1f in $CDCl_3$ at 300 K.

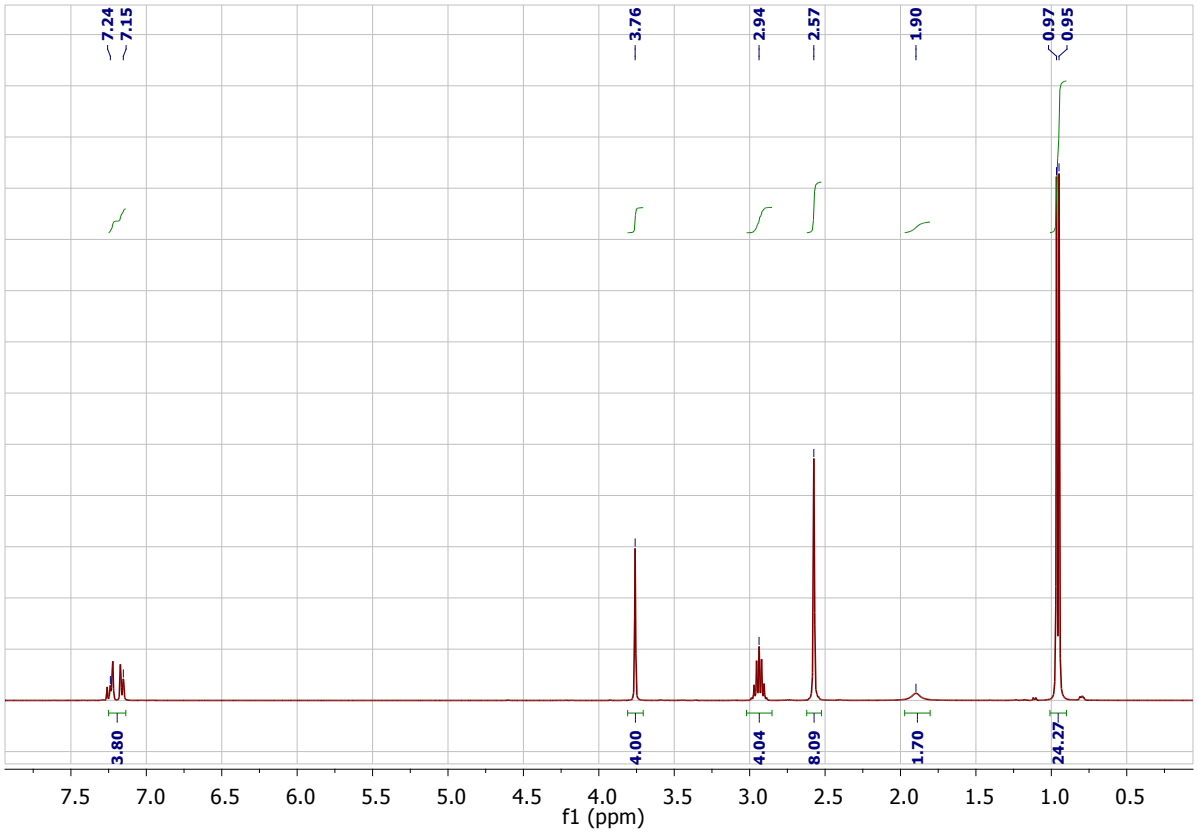

Figure S46 ^{1}H NMR spectrum (400 MHz) of 1g in $CDCl_3$ at 300 K.

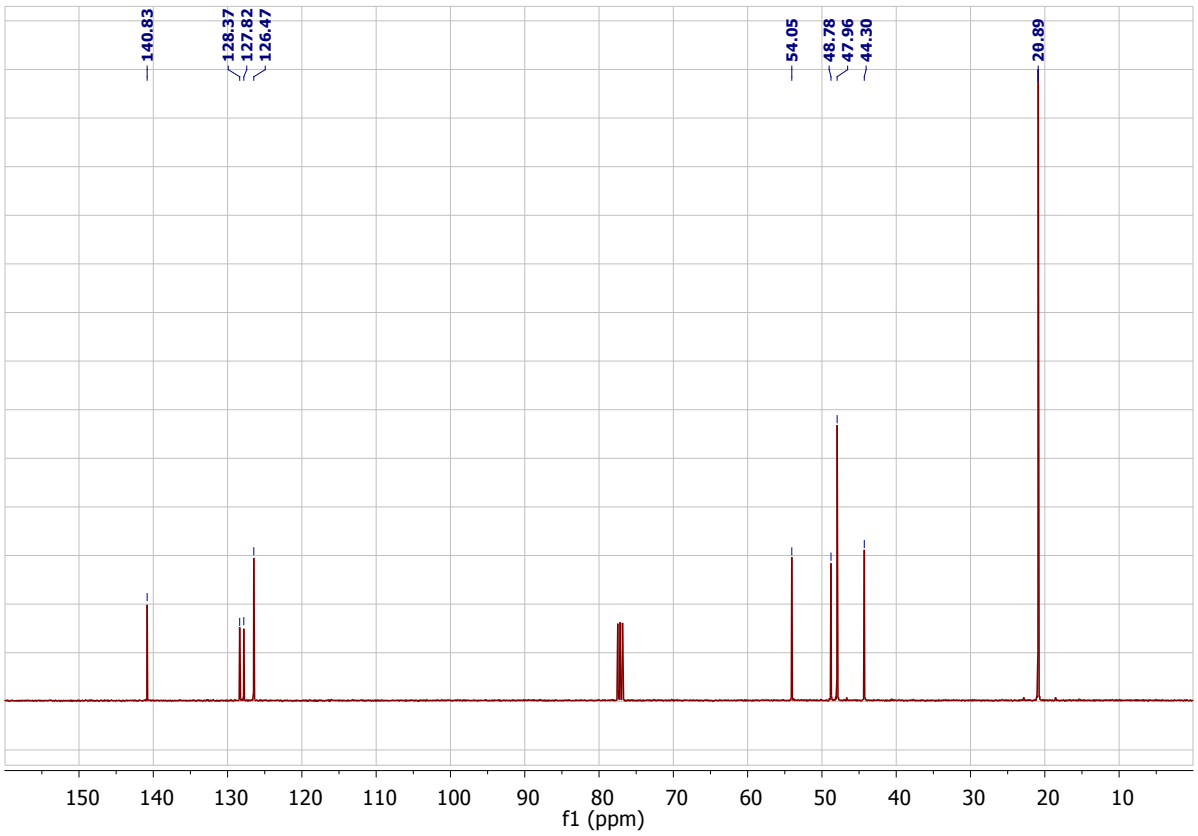

Figure S47 ^{13}C NMR spectrum (101 MHz) of 1g in $CDCl_3$ at 300 K.

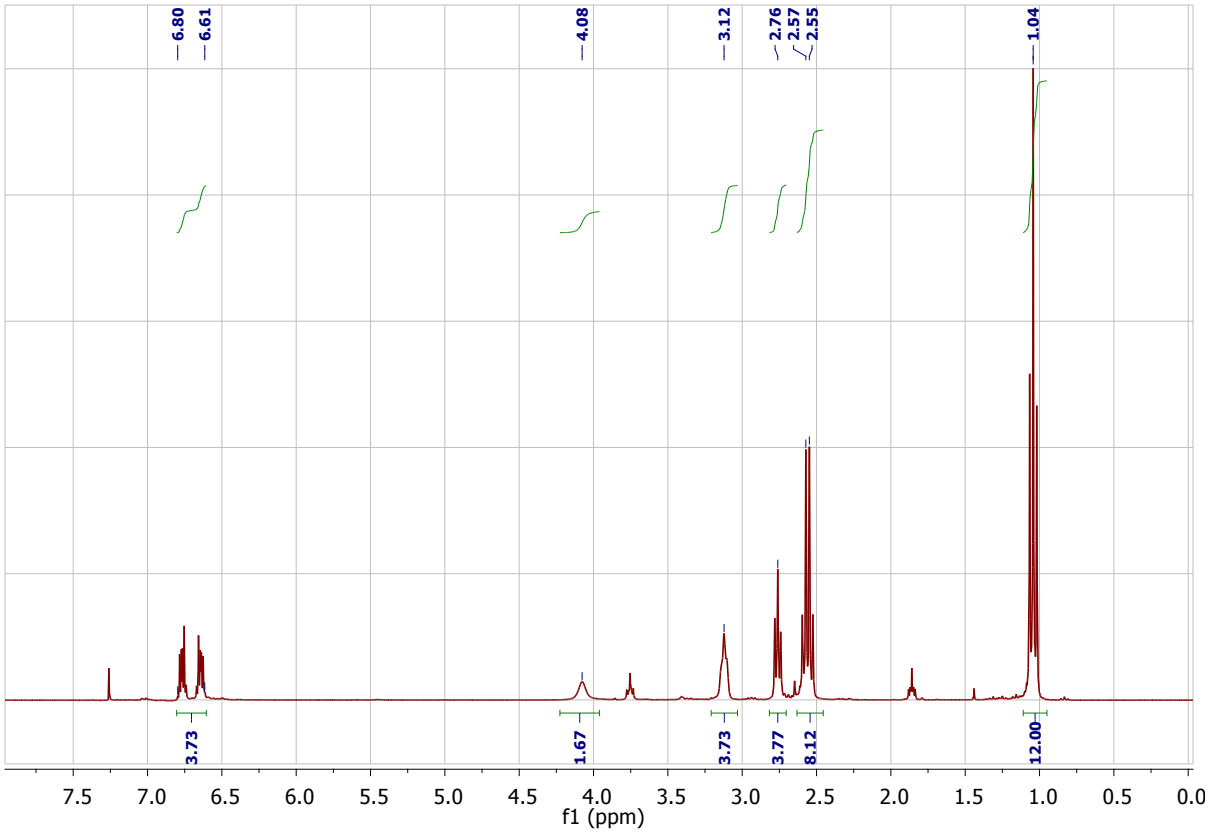

Figure S48 ^{1}H NMR spectrum (400 MHz) of 1h in $CDCl_3$ at 300 K.

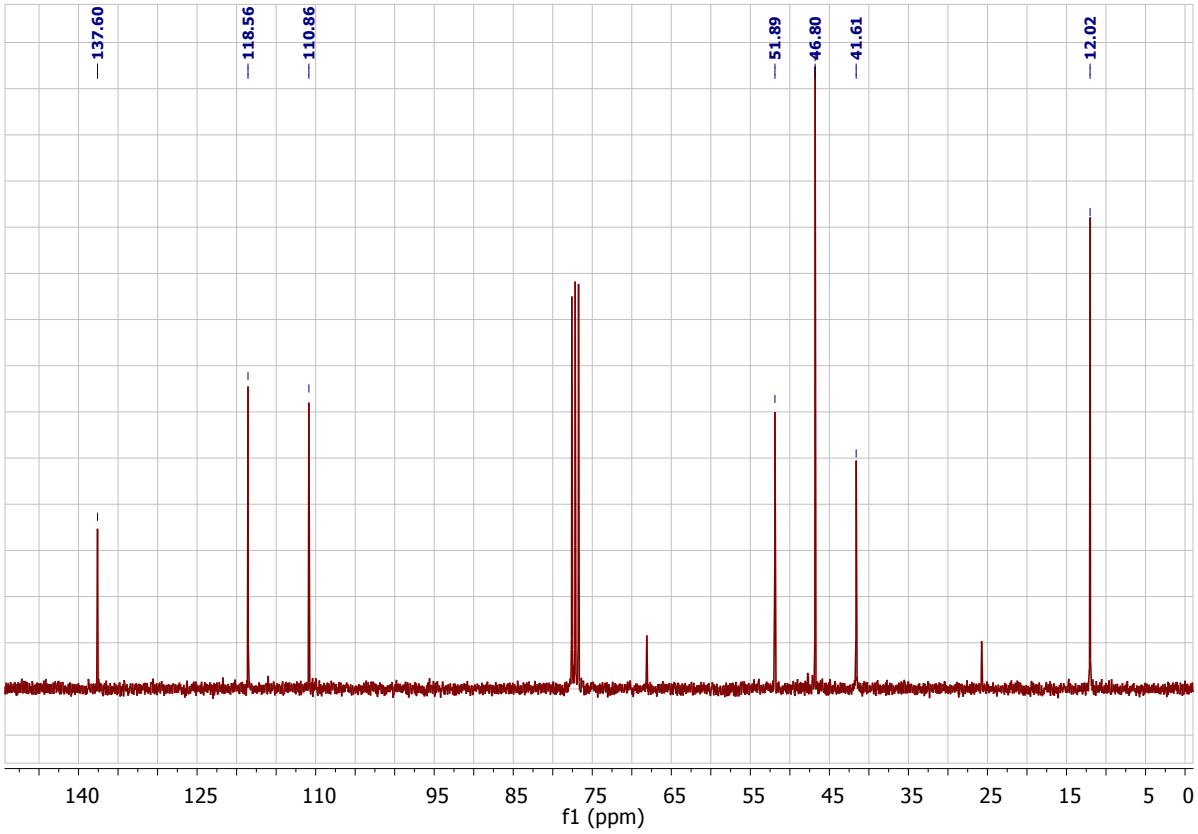

Figure S49 ^{13}C NMR spectrum (101 MHz) of 1h in $CDCl_3$ at 300 K.

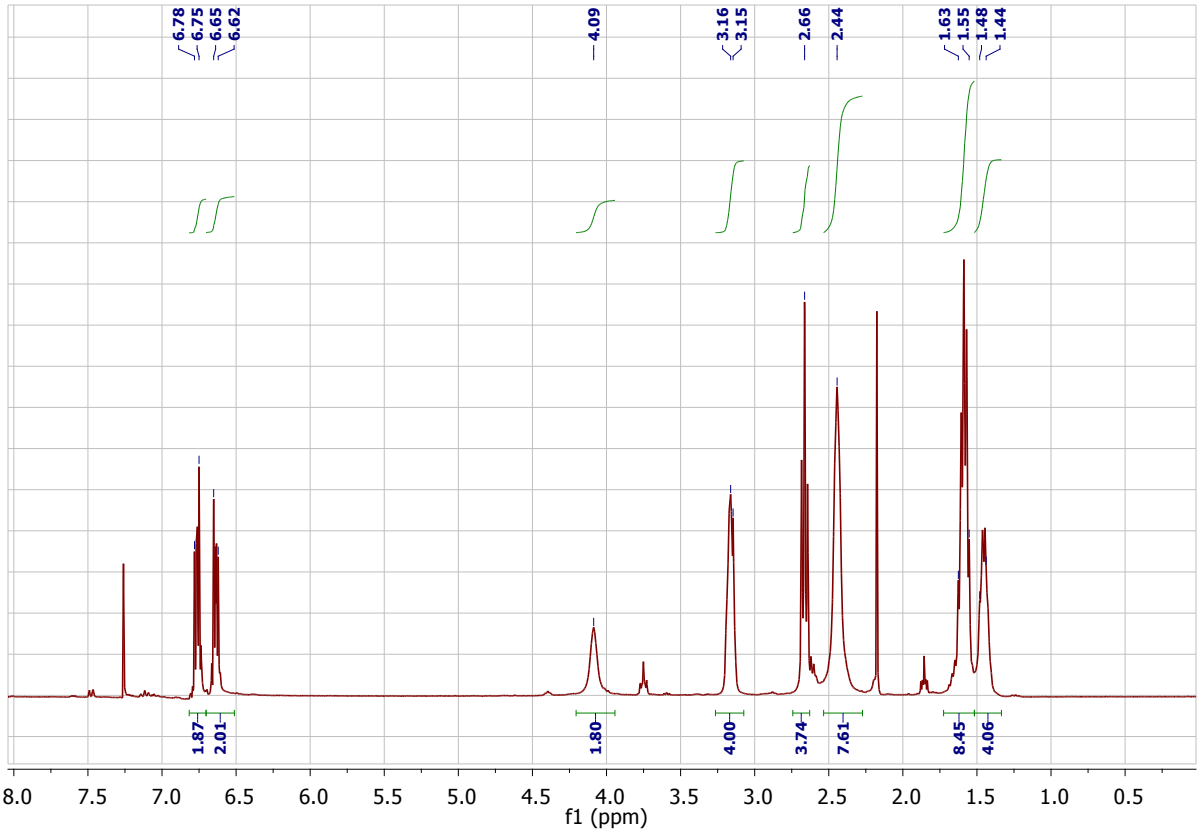

Figure S50 ^{1}H NMR spectrum (400 MHz) of 1i in $CDCl_3$ at 300 K.

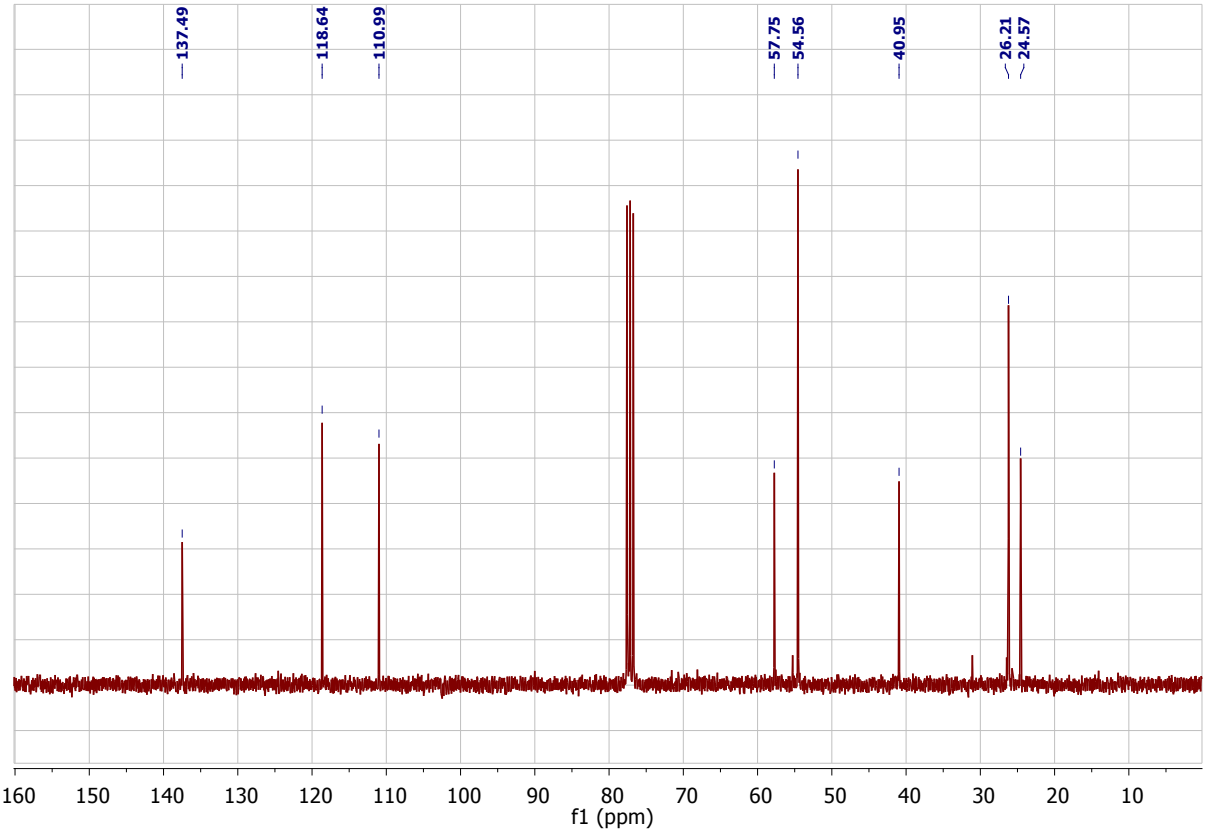

Figure S51 ^{13}C NMR spectrum (101 MHz) of 1i in $CDCl_3$ at 300 K.

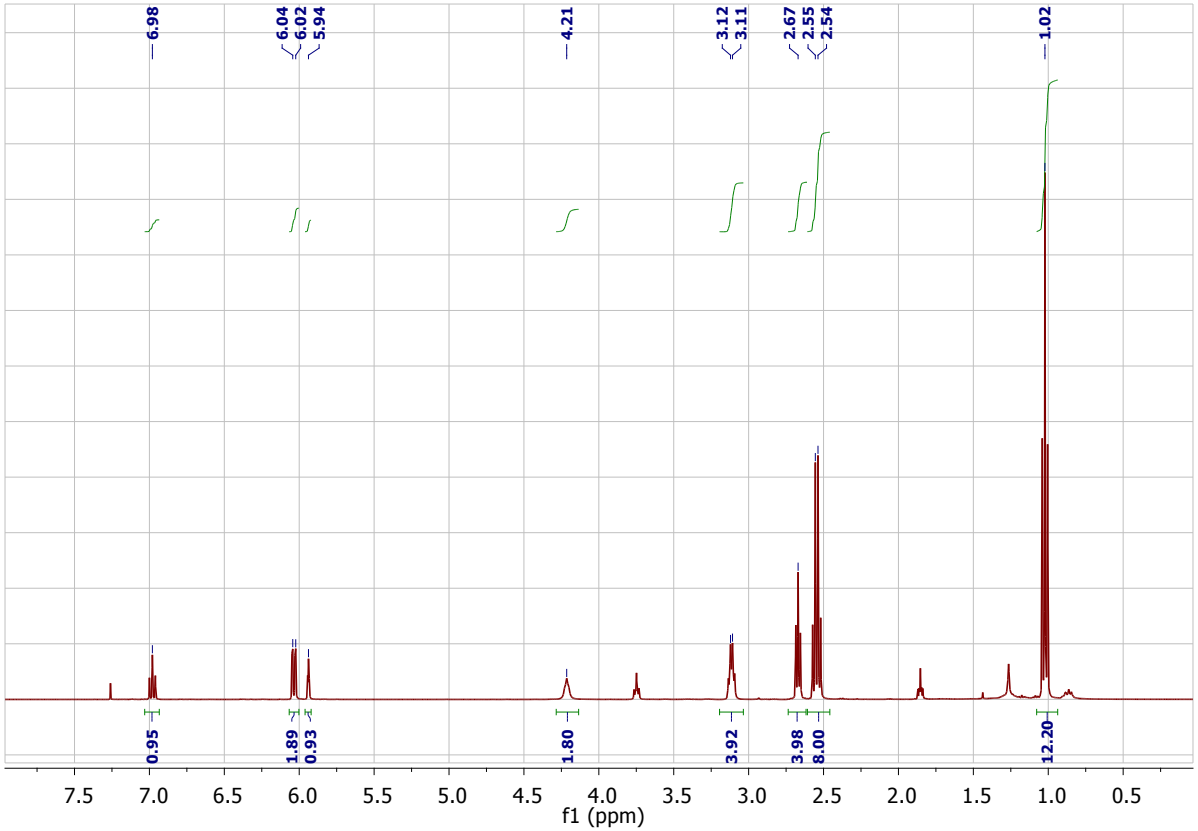

Figure S52 ^{1}H NMR spectrum (400 MHz) of 1j in $CDCl_3$ at 300 K.

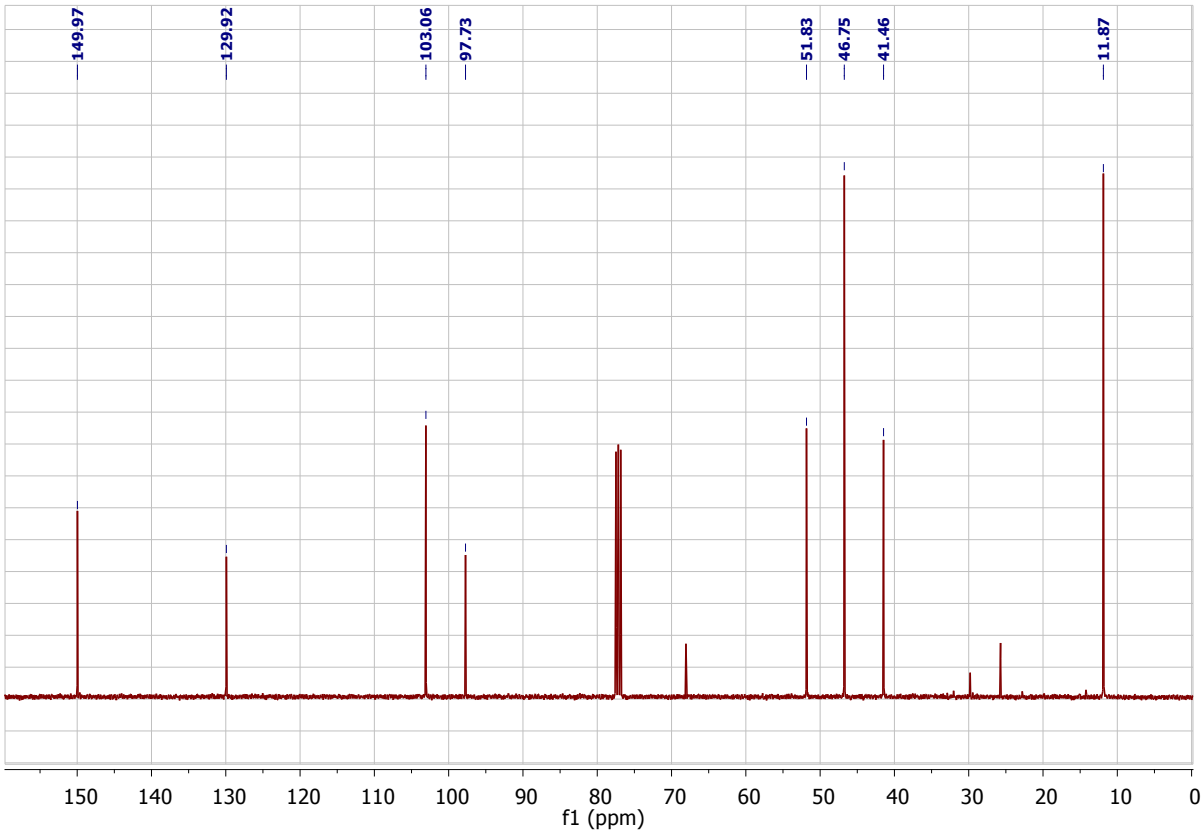

Figure S53 ^{13}C NMR spectrum (101 MHz) of 1j in $CDCl_3$ at 300 K.

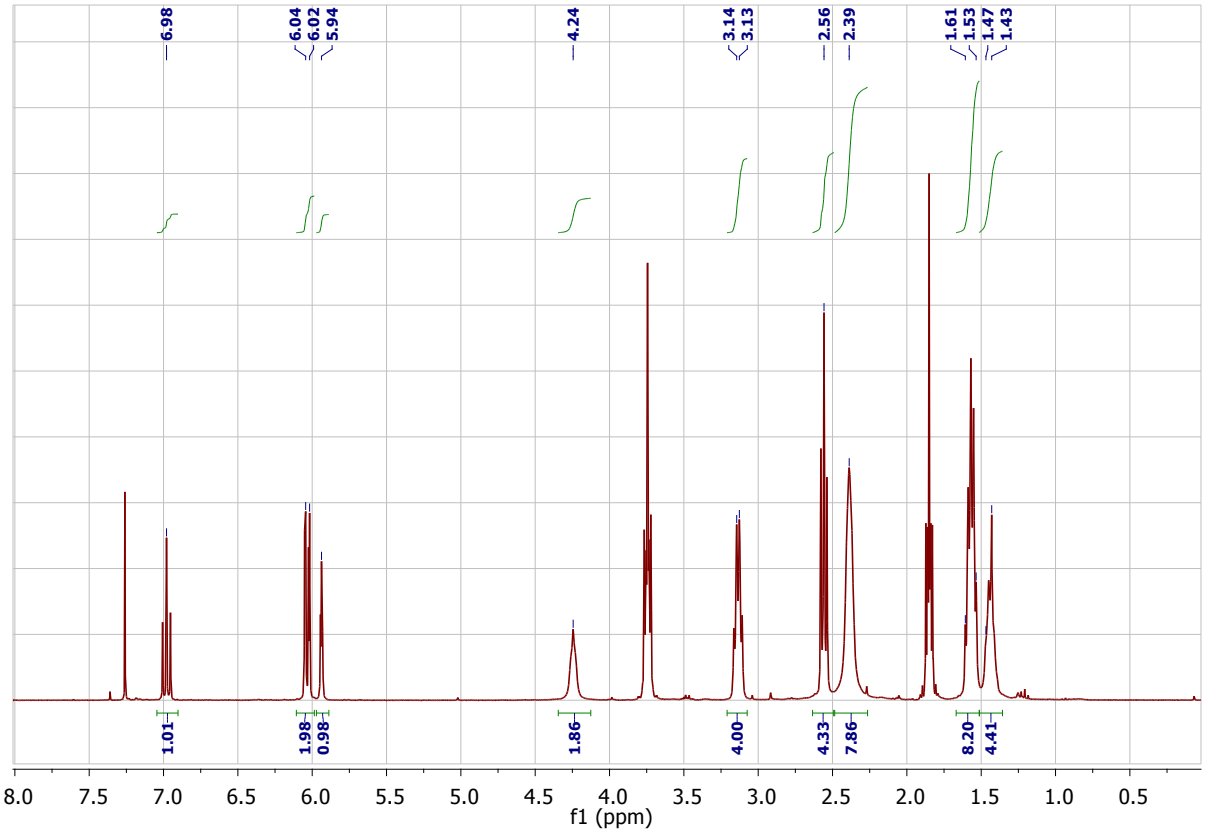

Figure S54 ^{1}H NMR spectrum (400 MHz) of 1k in $CDCl_3$ at 300 K.

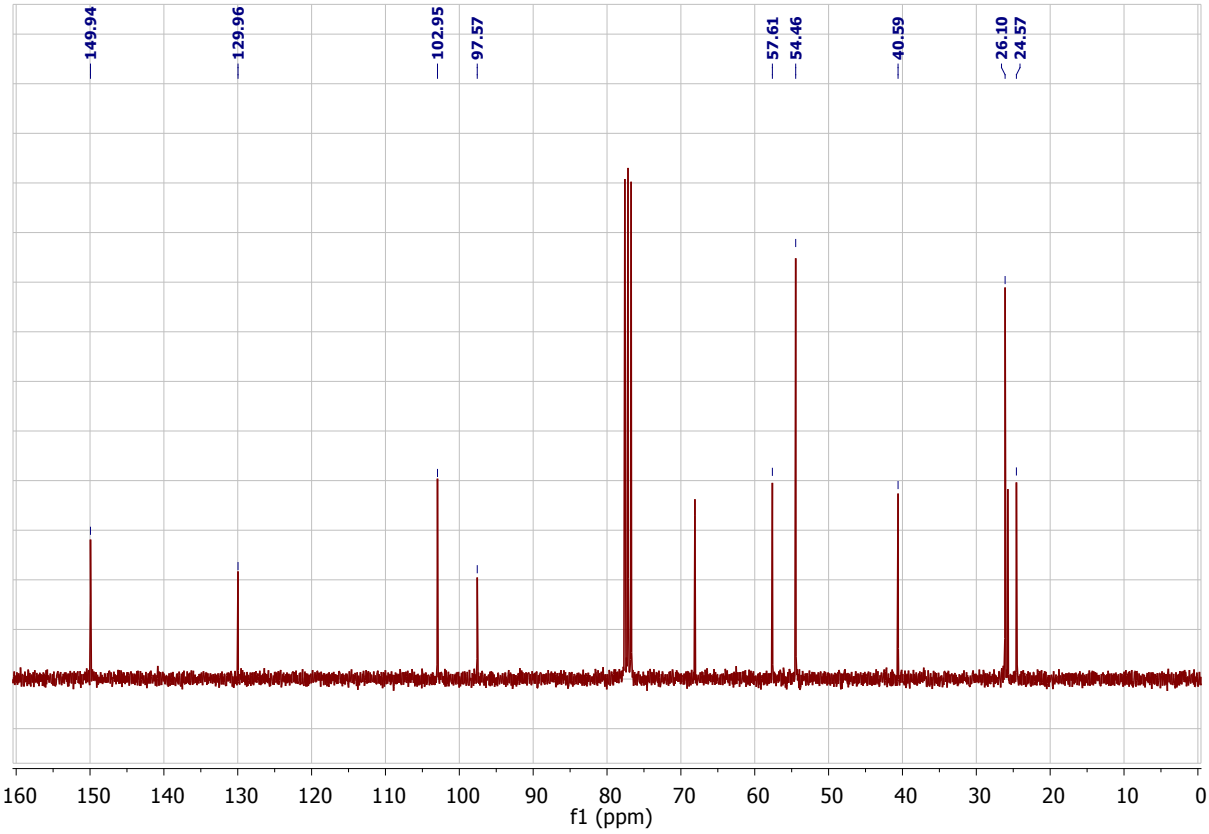

Figure S55 ^{13}C NMR spectrum (101 MHz) of 1k in $CDCl_3$ at 300 K.

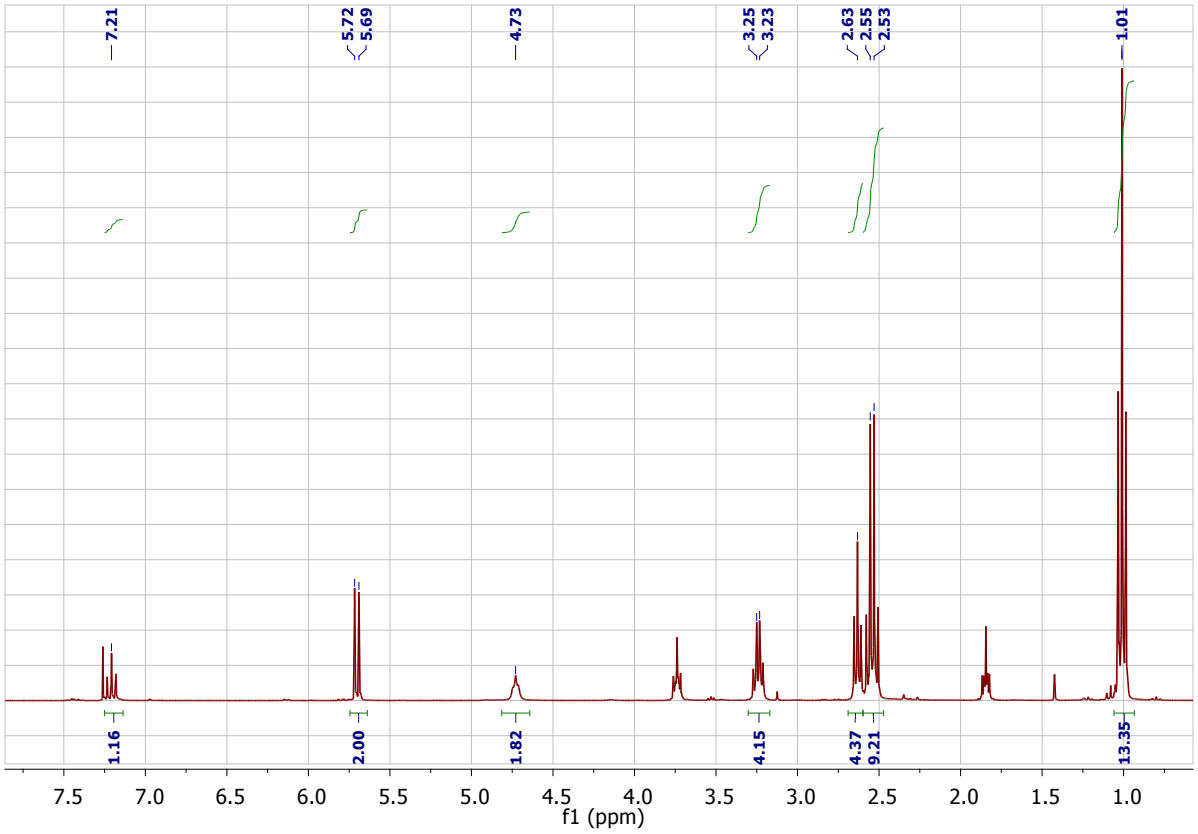

Figure S56 ^{1}H NMR spectrum (400 MHz) of 1l in $CDCl_3$ at 300 K.

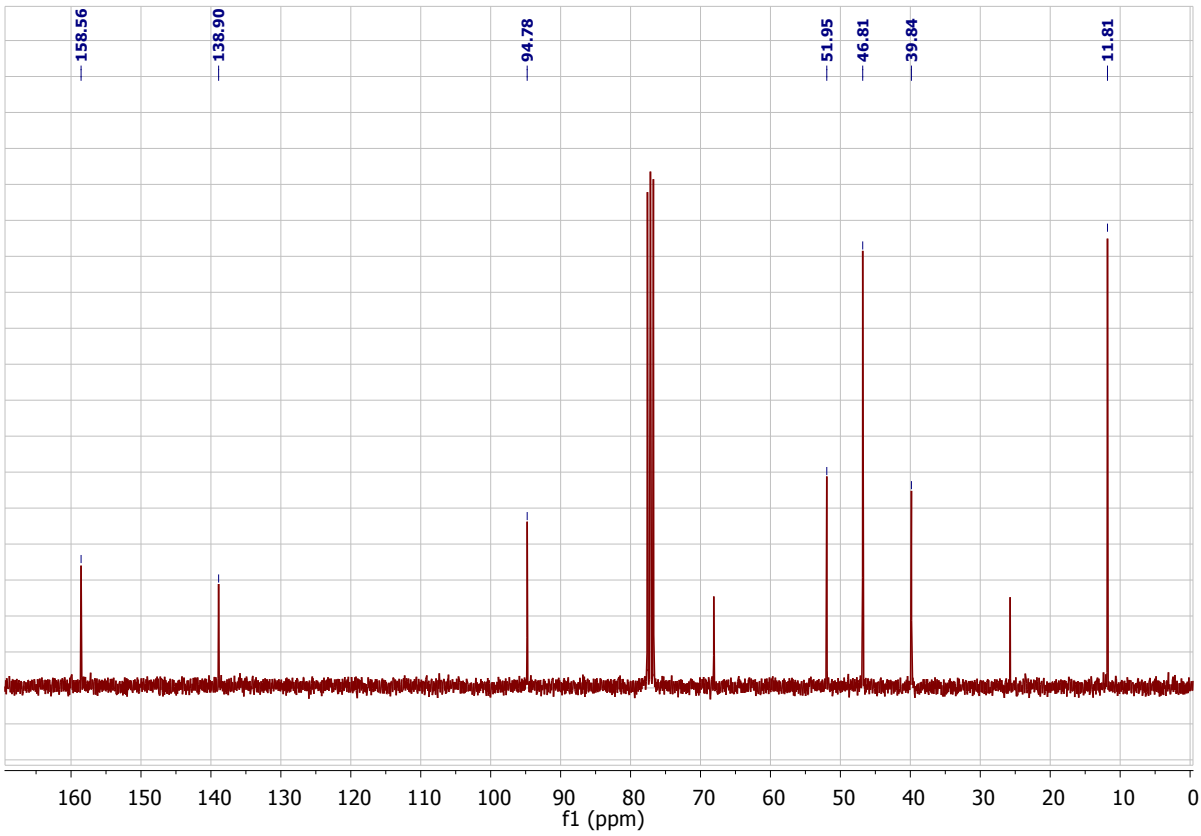

Figure S57 ^{13}C NMR spectrum (101 MHz) of 1l in $CDCl_3$ at 300 K.

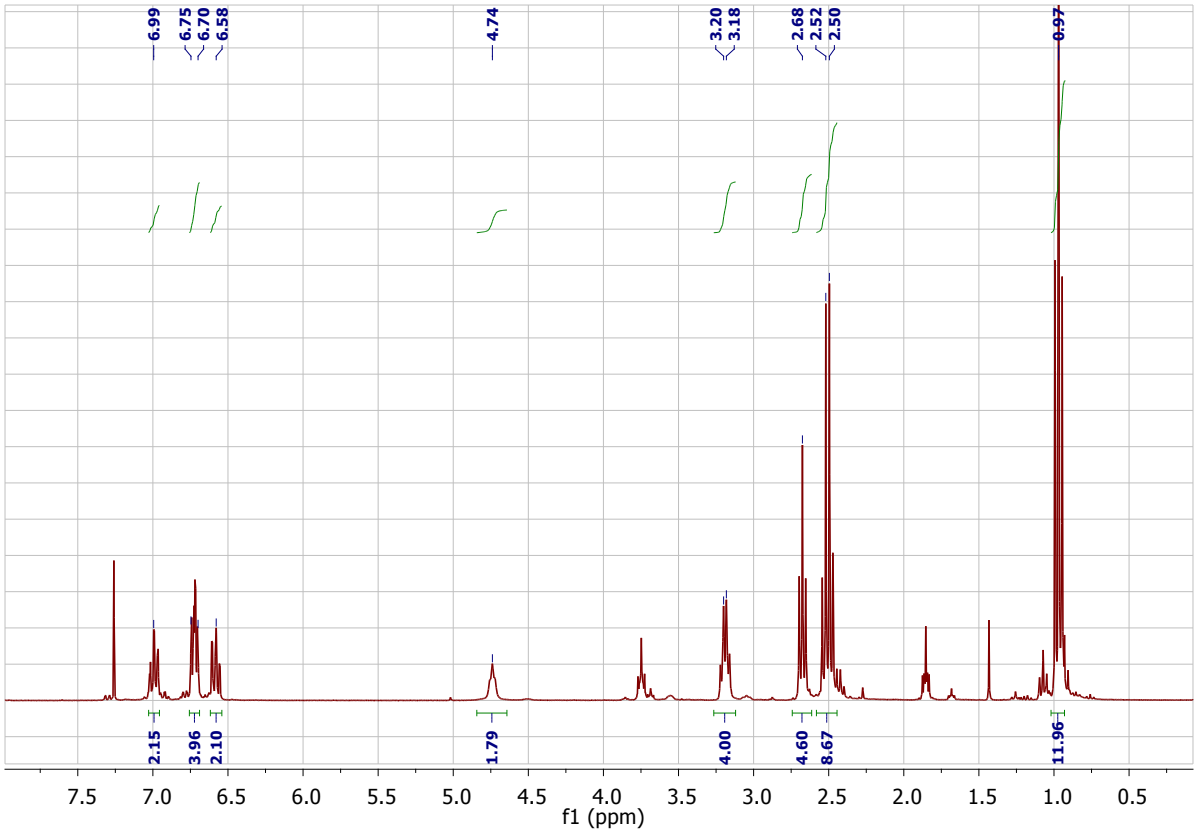

Figure S58 ^{1}H NMR spectrum (400 MHz) of 1m in $CDCl_3$ at 300 K.

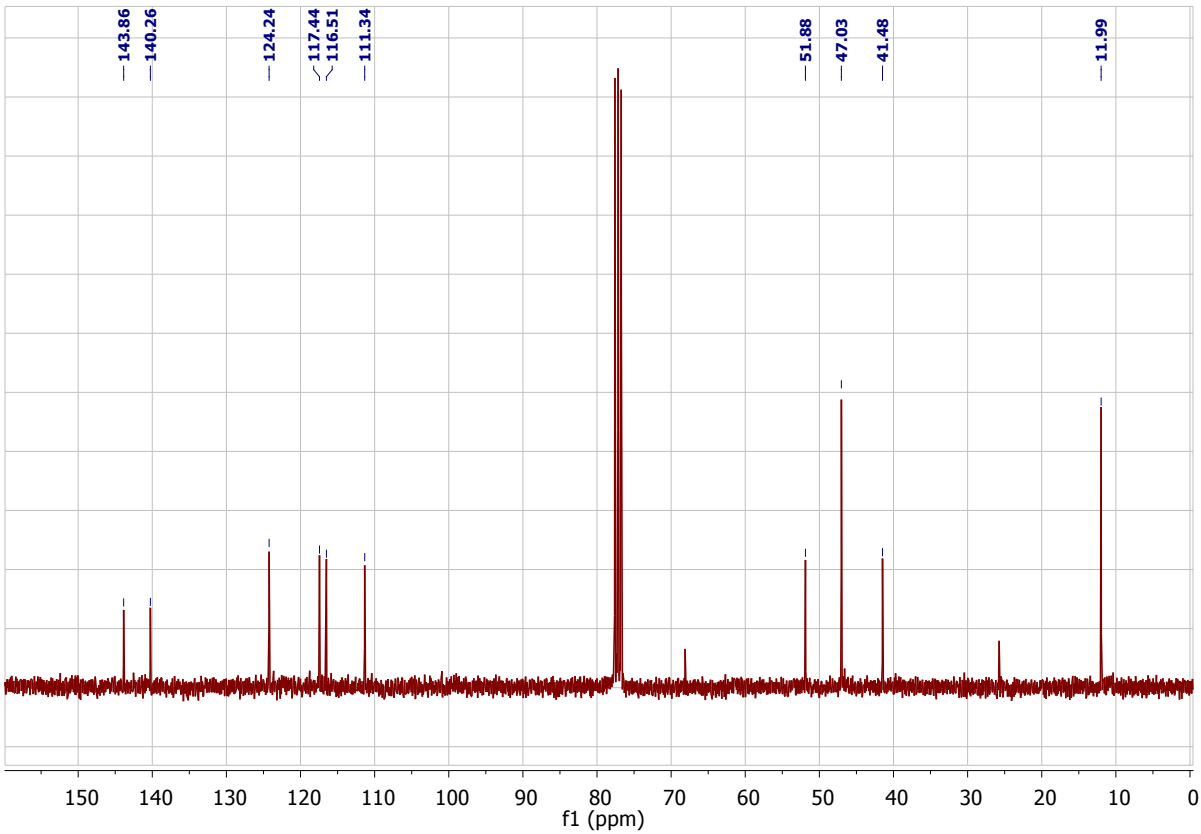

Figure S59 ^{13}C NMR spectrum (101 MHz) of 1m in $CDCl_3$ at 300 K.

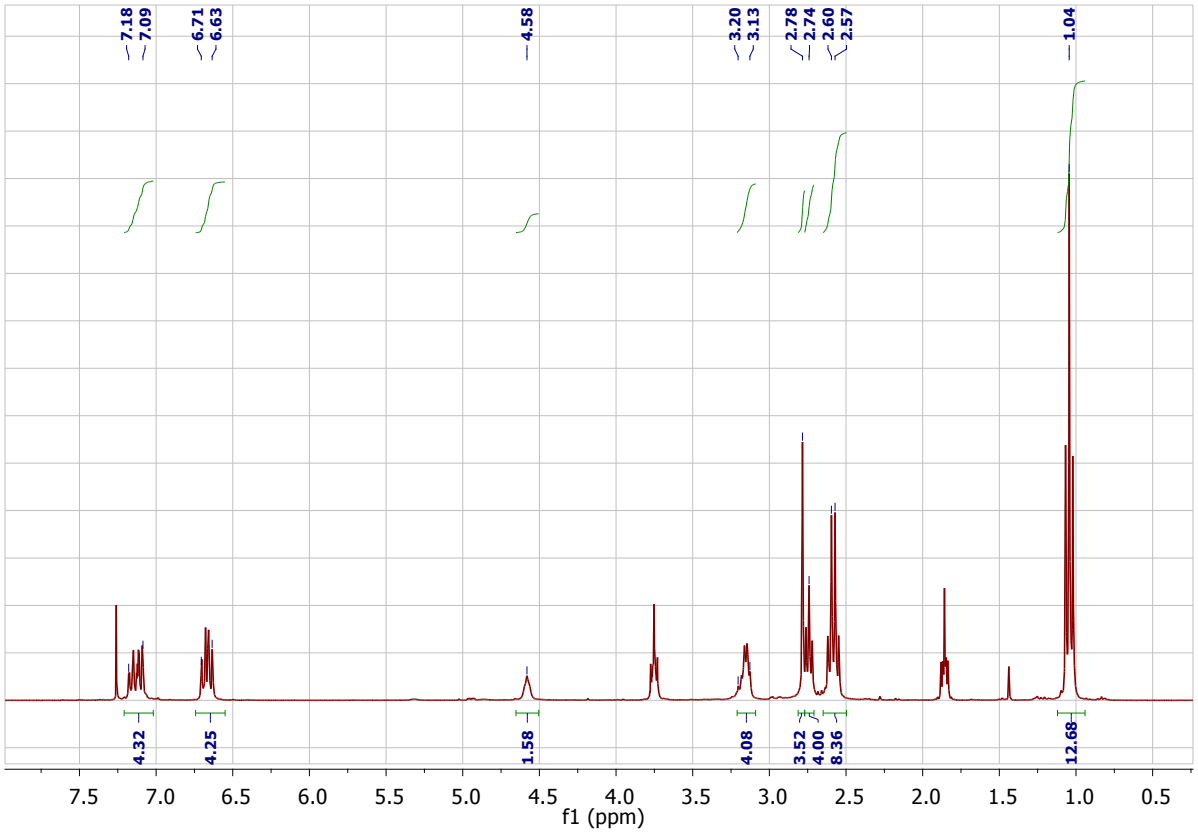

Figure S60 ^{1}H NMR spectrum (400 MHz) of 1n in $CDCl_3$ at 300 K.

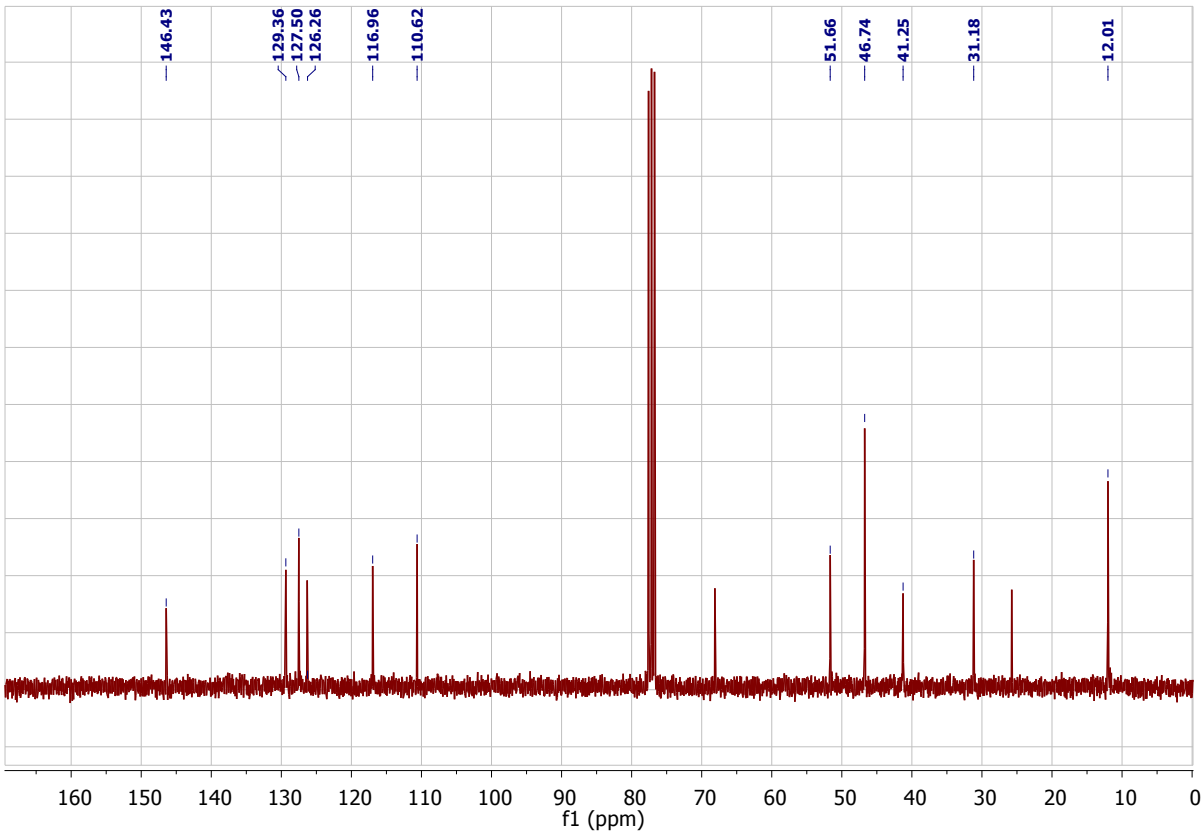

Figure S61 ^{13}C NMR spectrum (101 MHz) of 1n in $CDCl_3$ at 300 K.

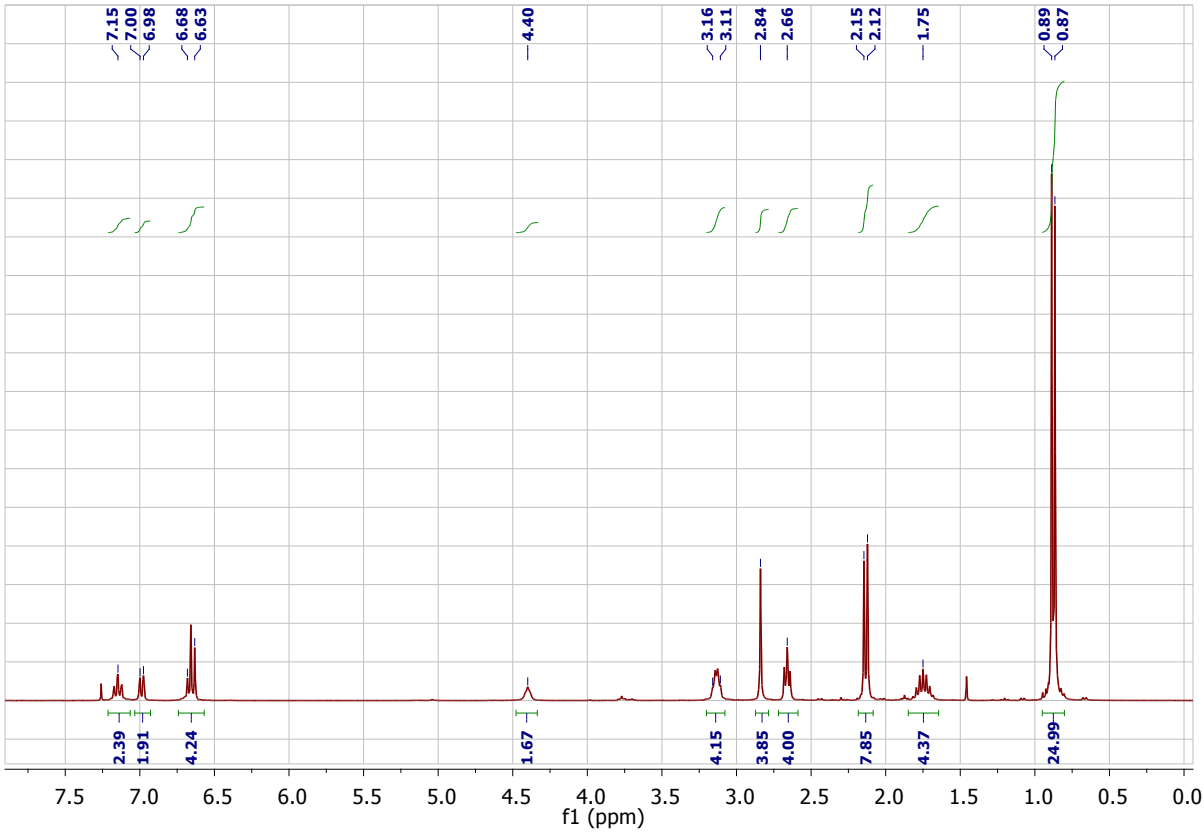

Figure S62 ^{1}H NMR spectrum (400 MHz) of 1o in $CDCl_3$ at 300 K.

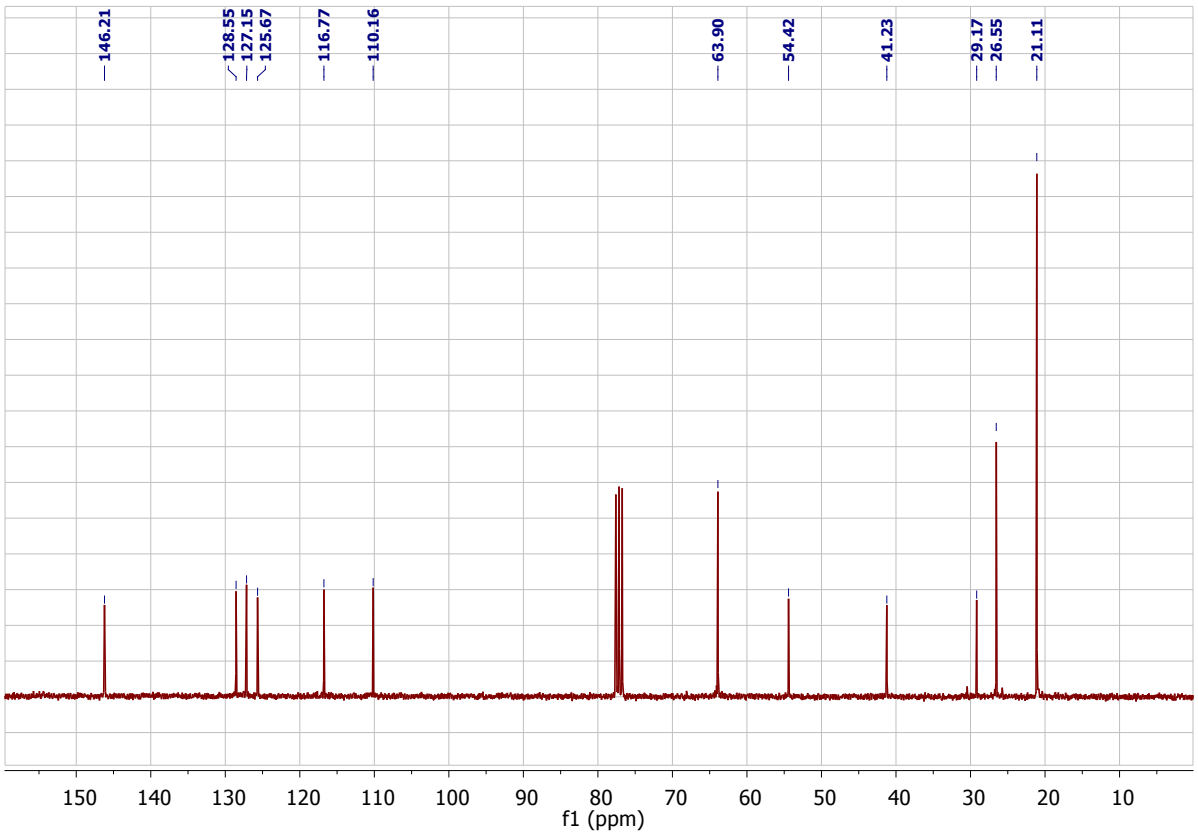

Figure S63 ^{13}C NMR spectrum (101 MHz) of 1o in $CDCl_3$ at 300 K.

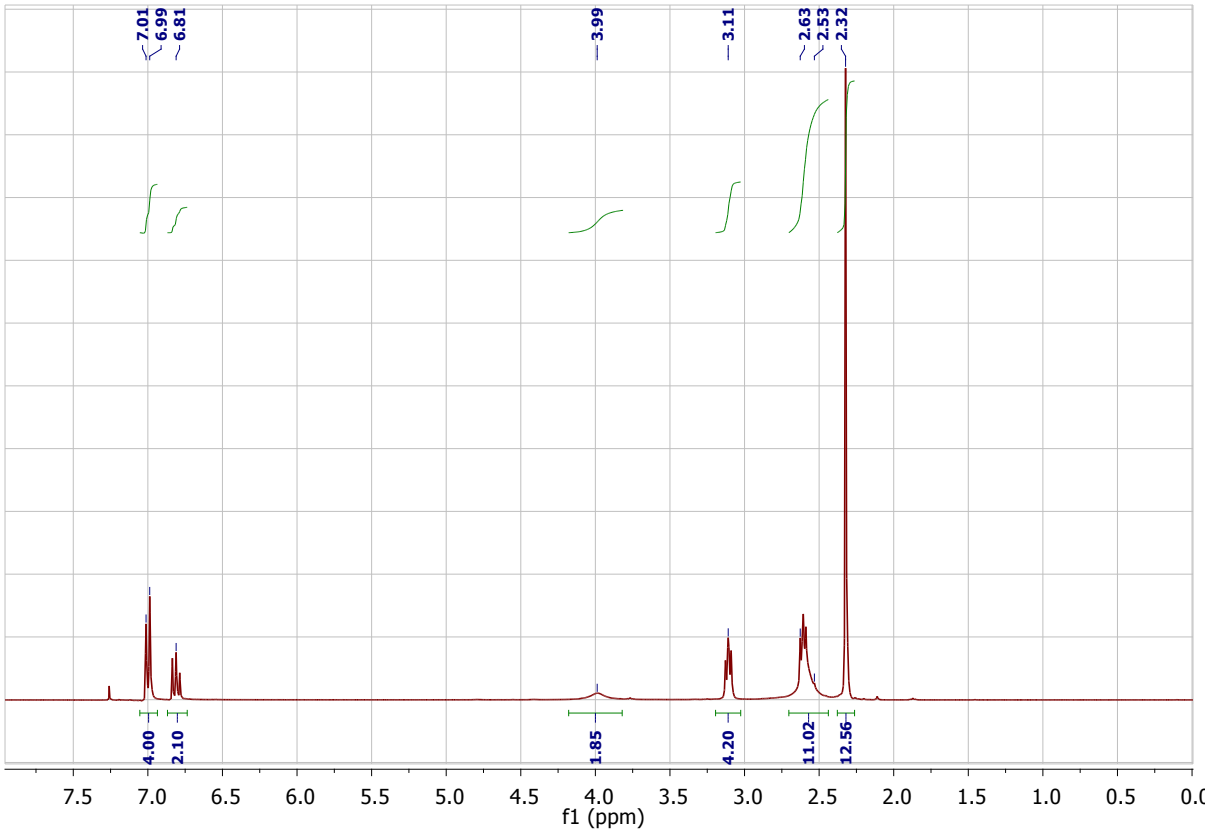

Figure S64 ^{1}H NMR spectrum (400 MHz) of 2a in $CDCl_3$ at 300 K.

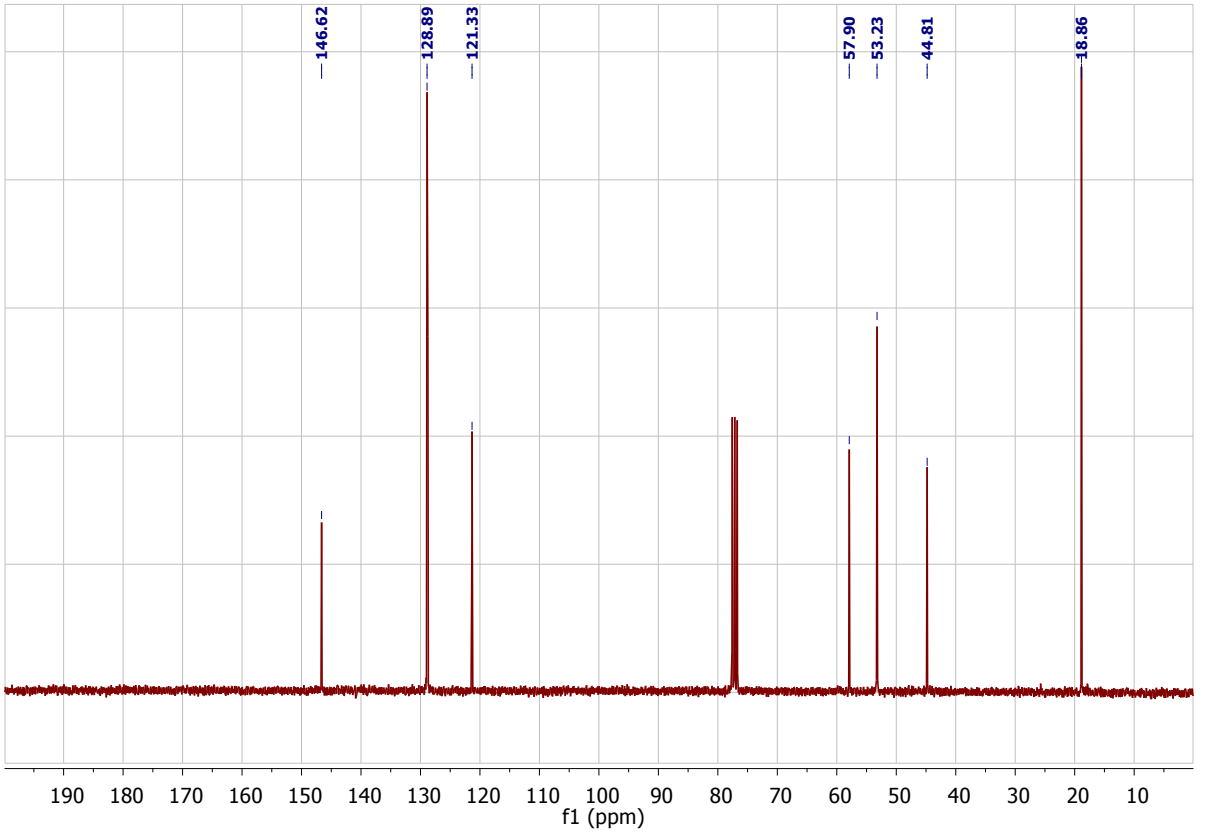

Figure S65 ^{13}C NMR spectrum (101 MHz) of 2a in $CDCl_3$ at 300 K.

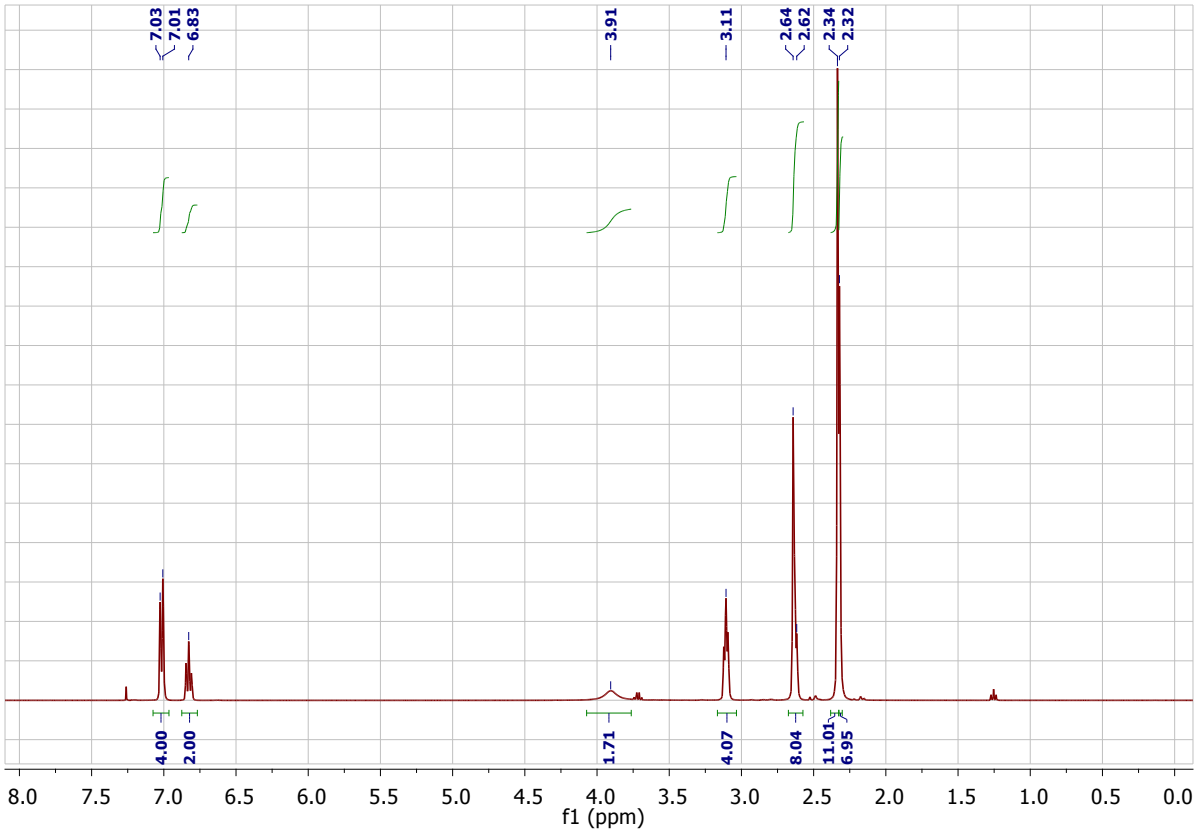

Figure S66 ^{1}H NMR spectrum (400 MHz) of 2b in $CDCl_3$ at 300 K.

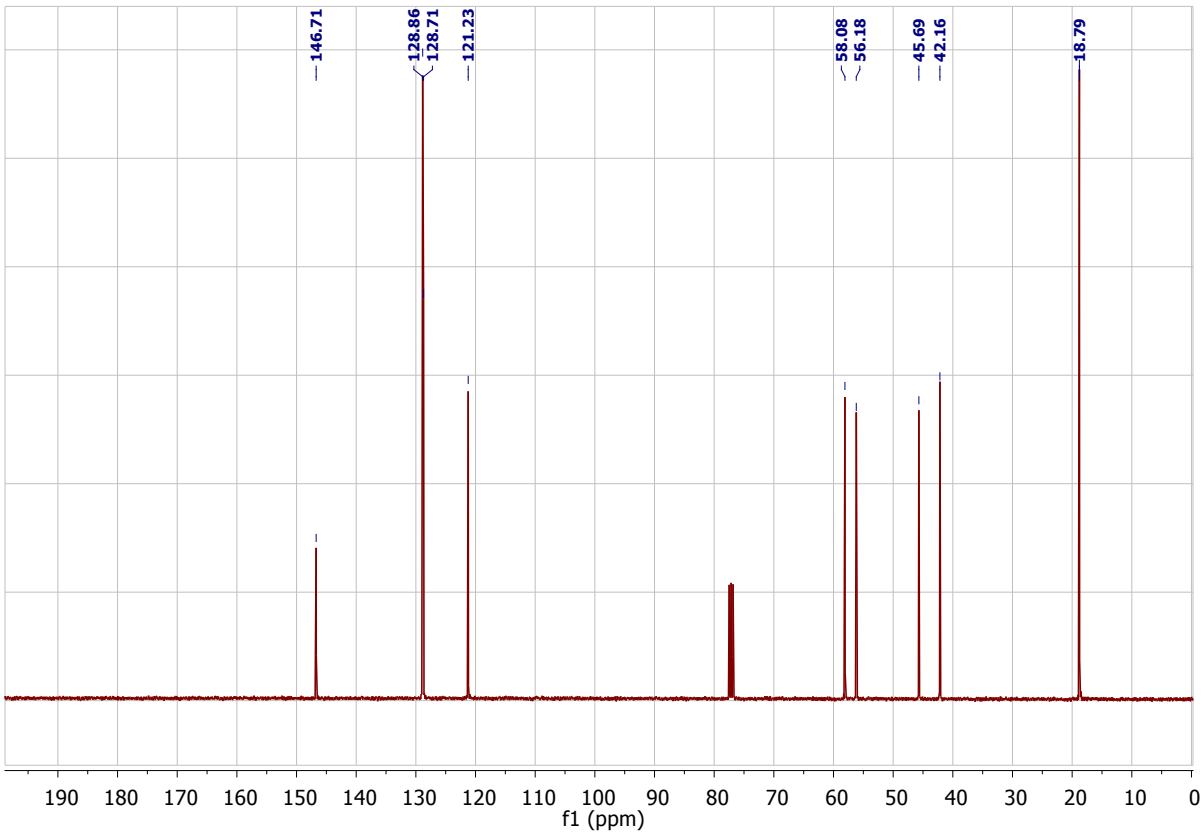

Figure S67 ^{13}C NMR spectrum (101 MHz) of 2b in $CDCl_3$ at 300 K.

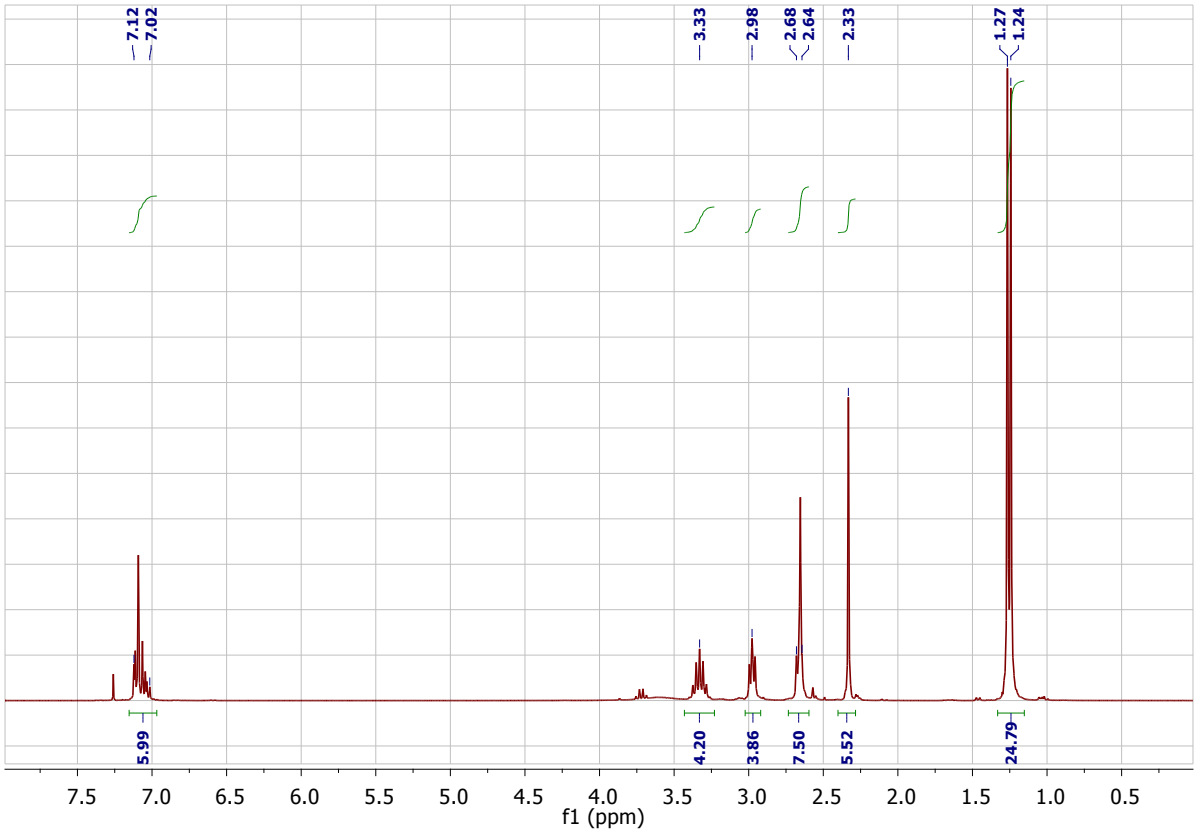

Figure S68 ^{1}H NMR spectrum (400 MHz) of 2c in $CDCl_3$ at 300 K.

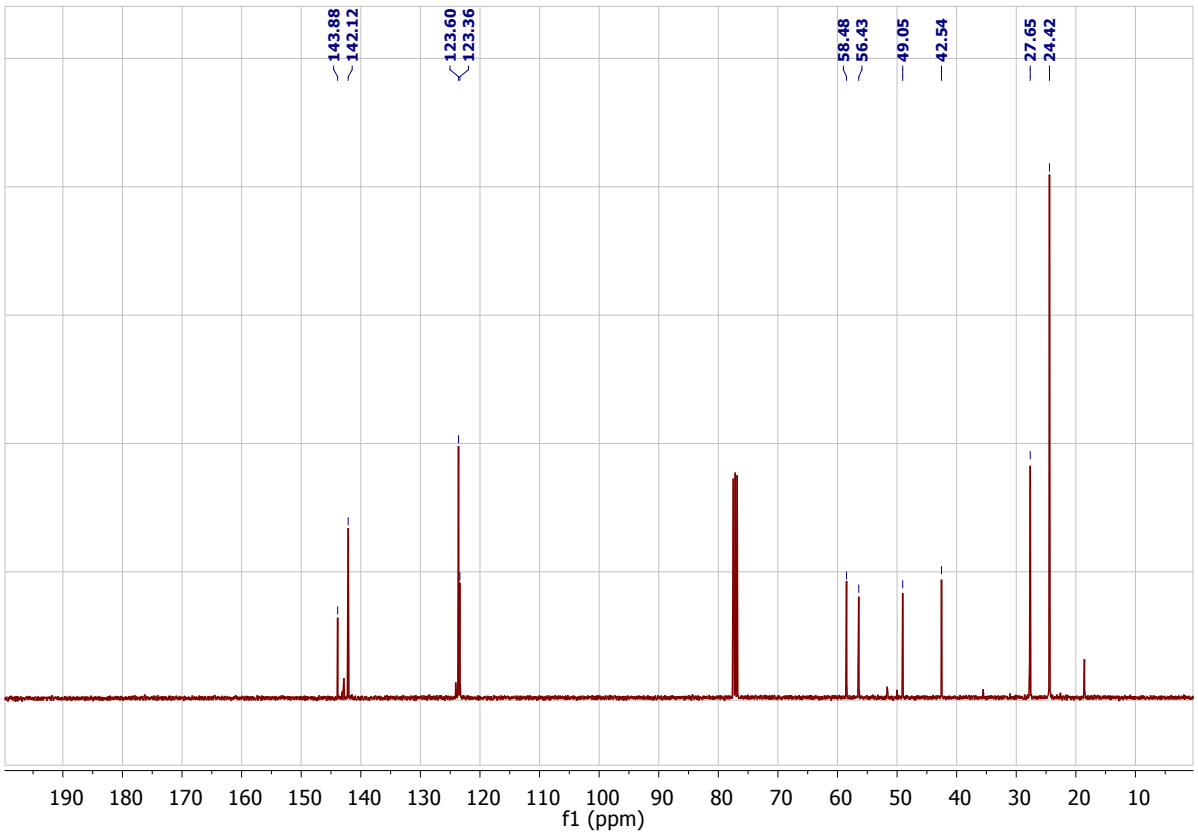

Figure S69 ^{13}C NMR spectrum (101 MHz) of 2c in $CDCl_3$ at 300 K.

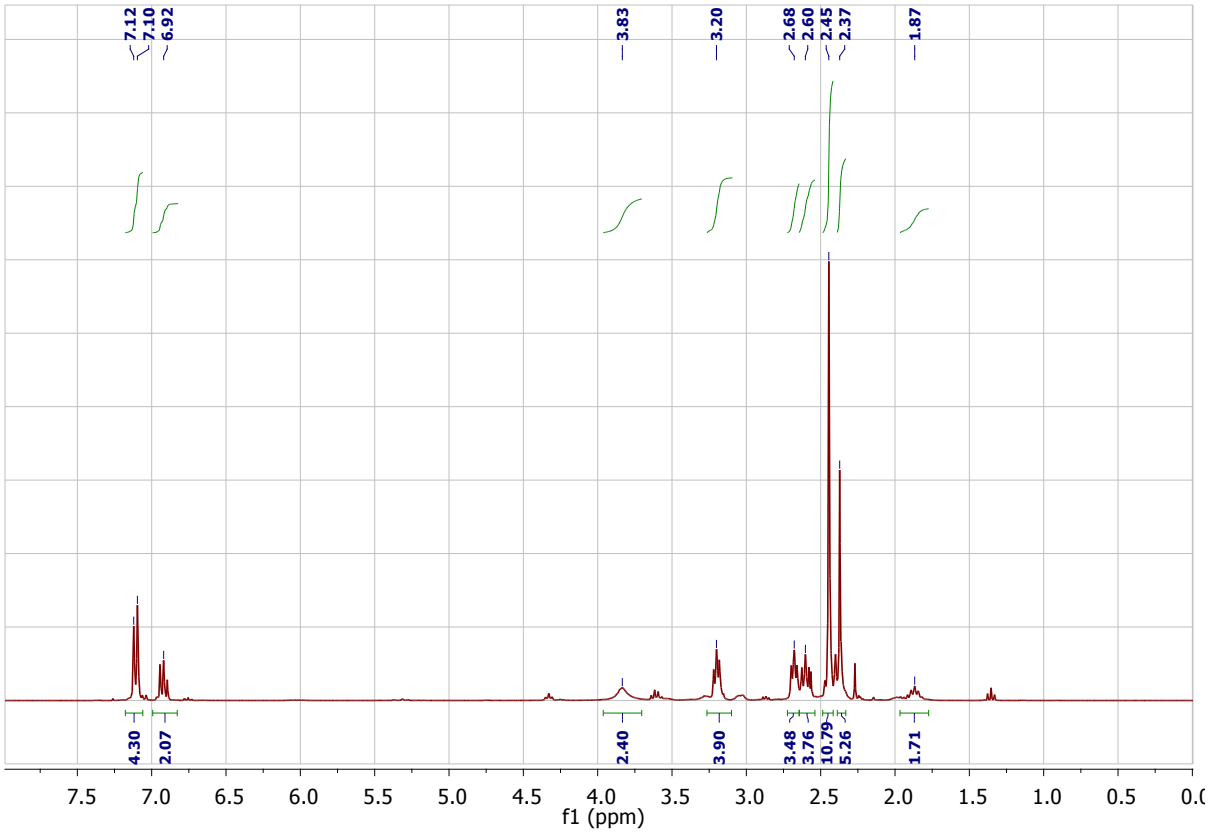

Figure S70 ^{1}H NMR spectrum (400 MHz) of 2d in $CDCl_3$ at 300 K.

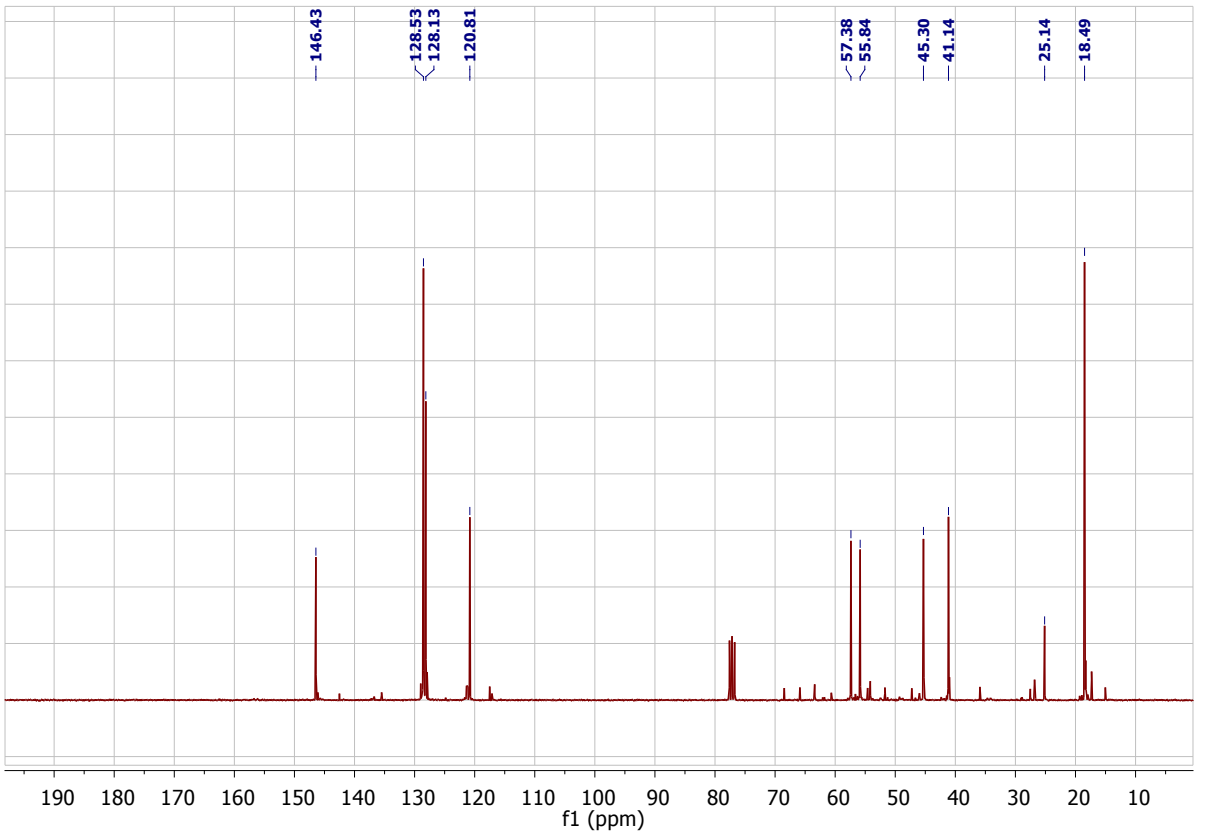

Figure S71 ^{13}C NMR spectrum (101 MHz) of 2d in $CDCl_3$ at 300 K.

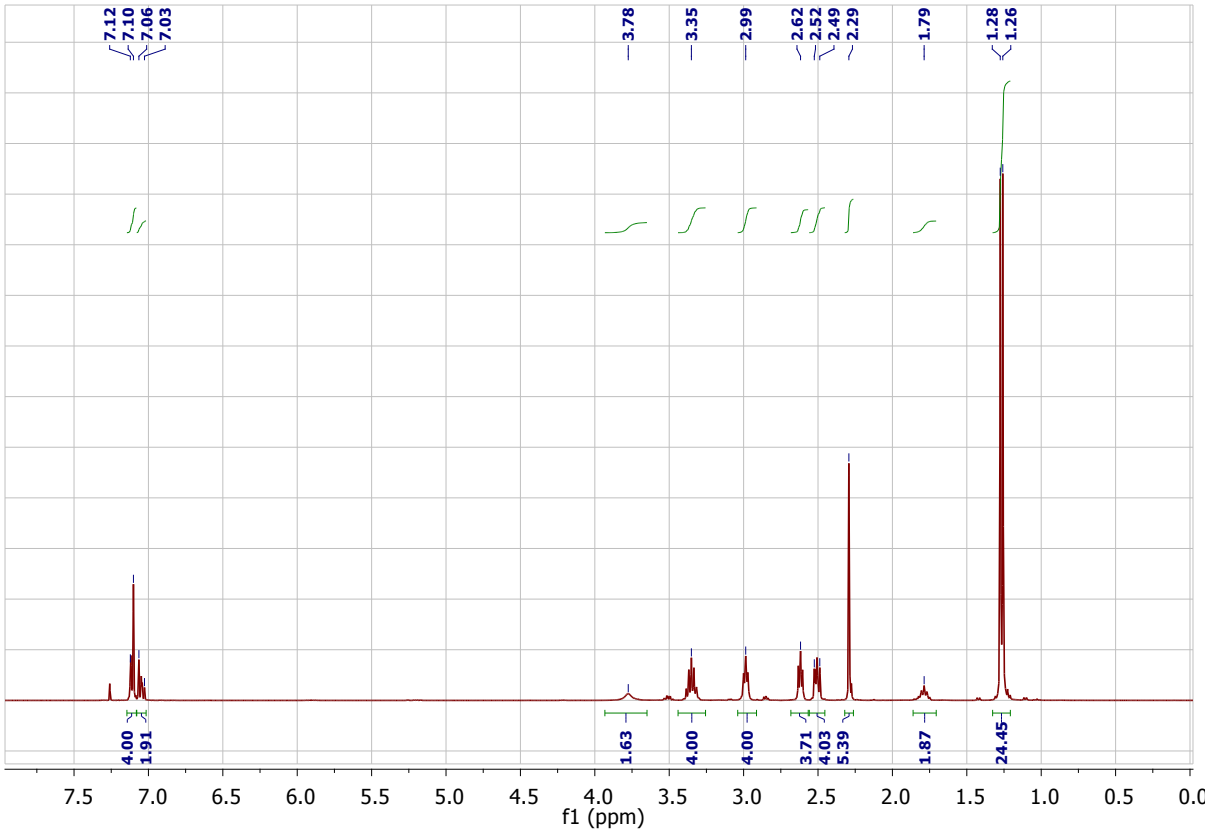

Figure S72 ^{1}H NMR spectrum (400 MHz) of 2e in $CDCl_3$ at 300 K.

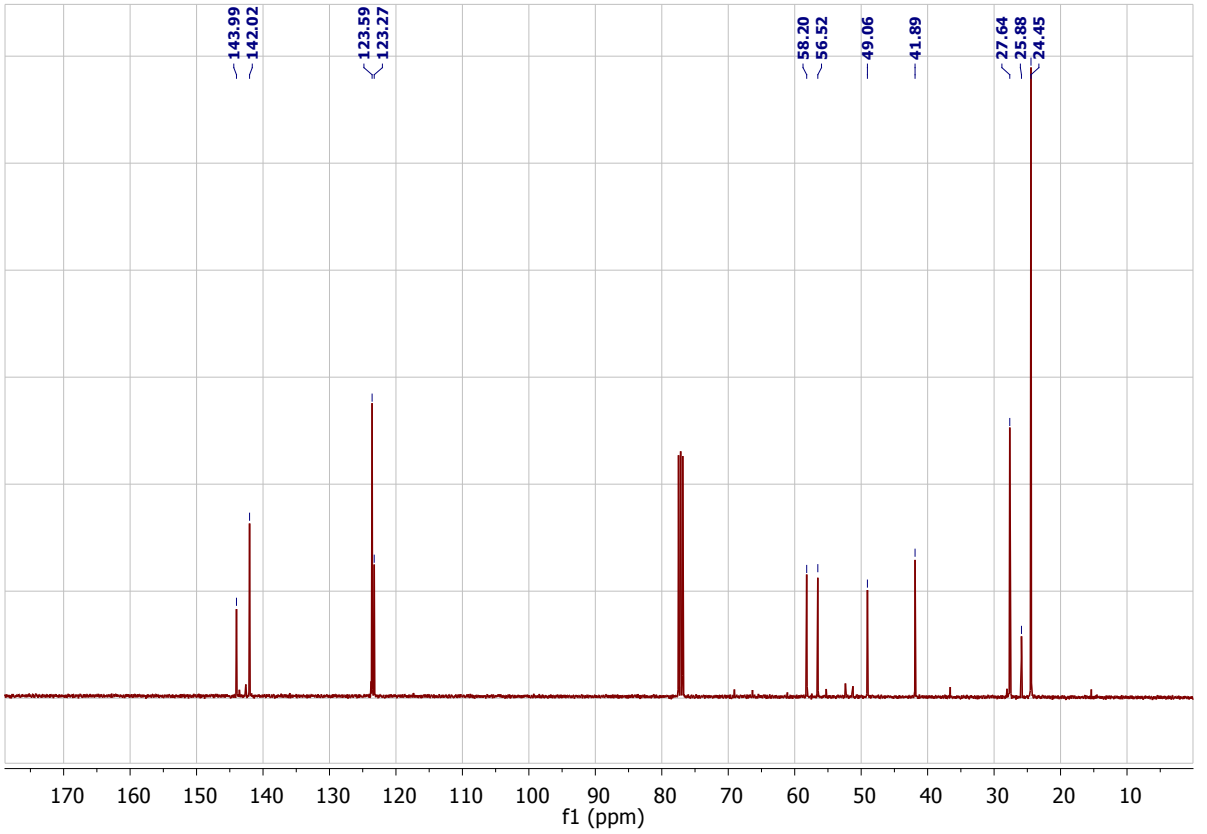

Figure S73 ^{13}C NMR spectrum (101 MHz) of 2e in $CDCl_3$ at 300 K.

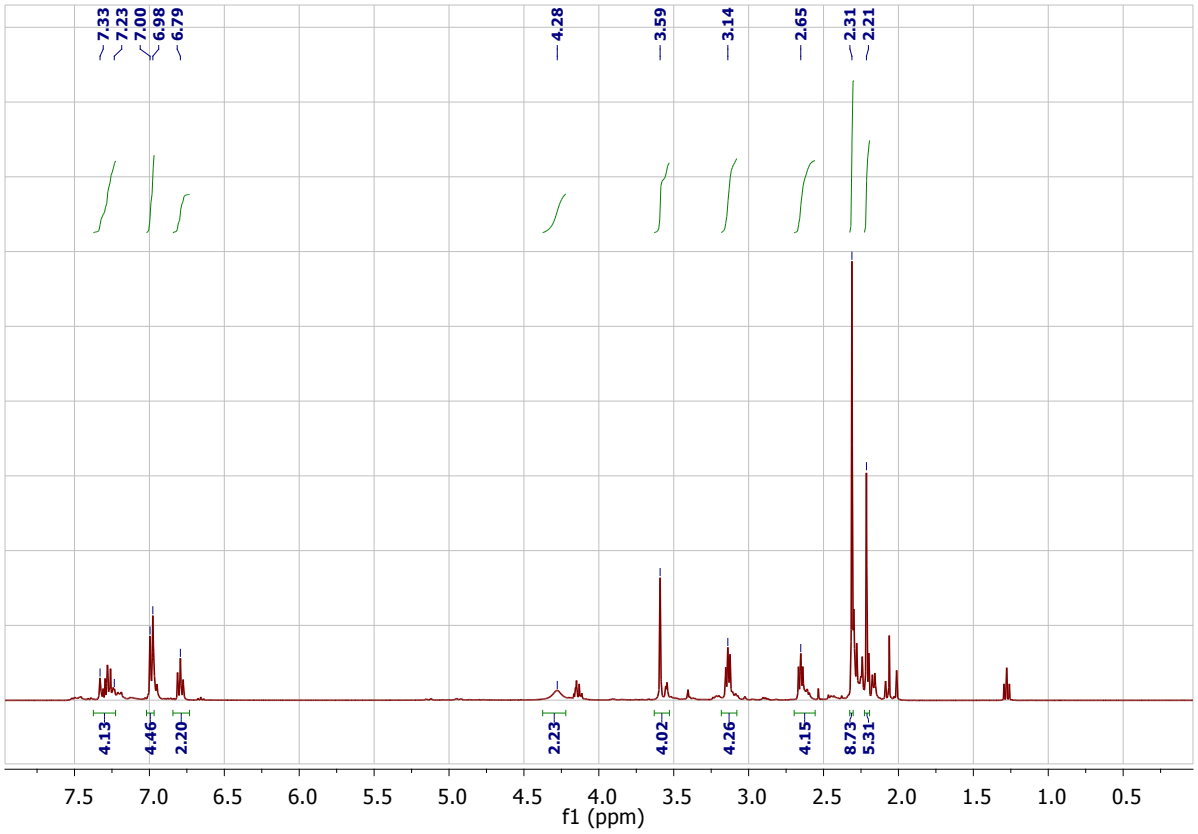

Figure S74 ^{1}H NMR spectrum (400 MHz) of 2f in $CDCl_3$ at 300 K.

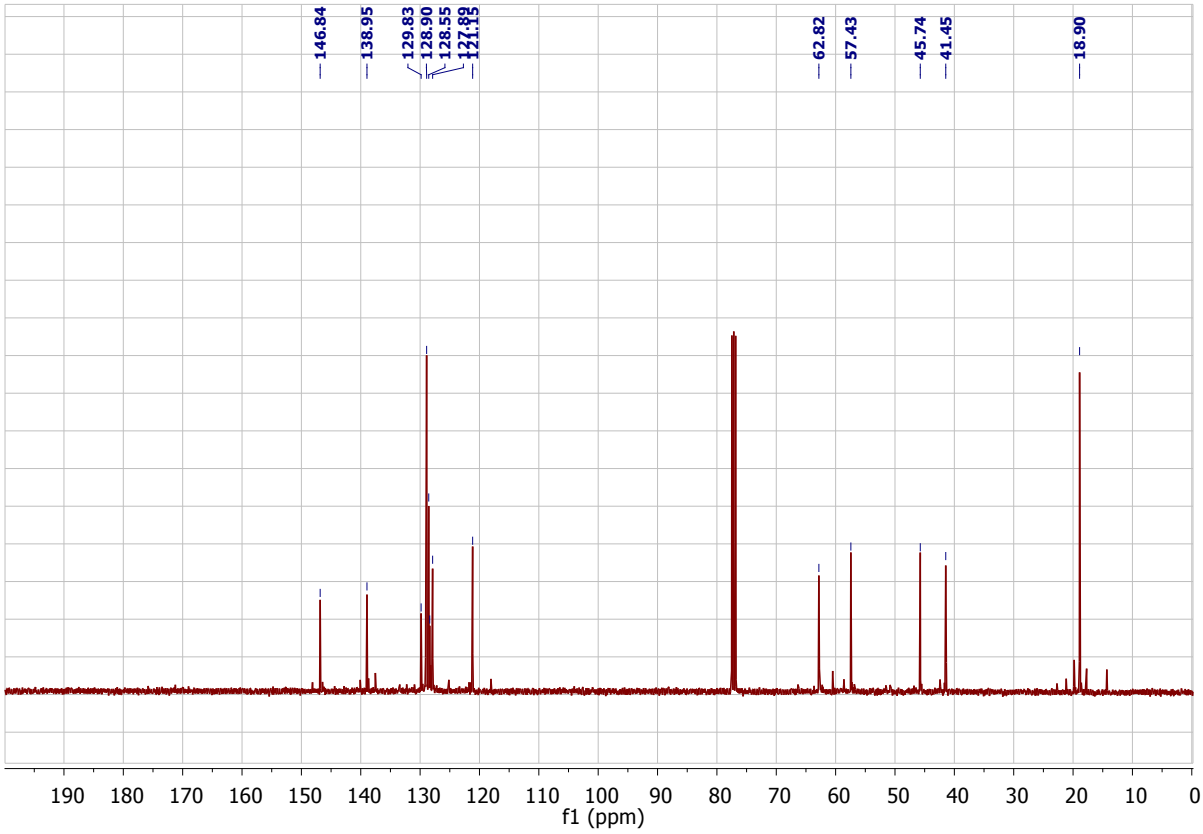

Figure S75 ^{13}C NMR spectrum (101 MHz) of 2f in $CDCl_3$ at 300 K.

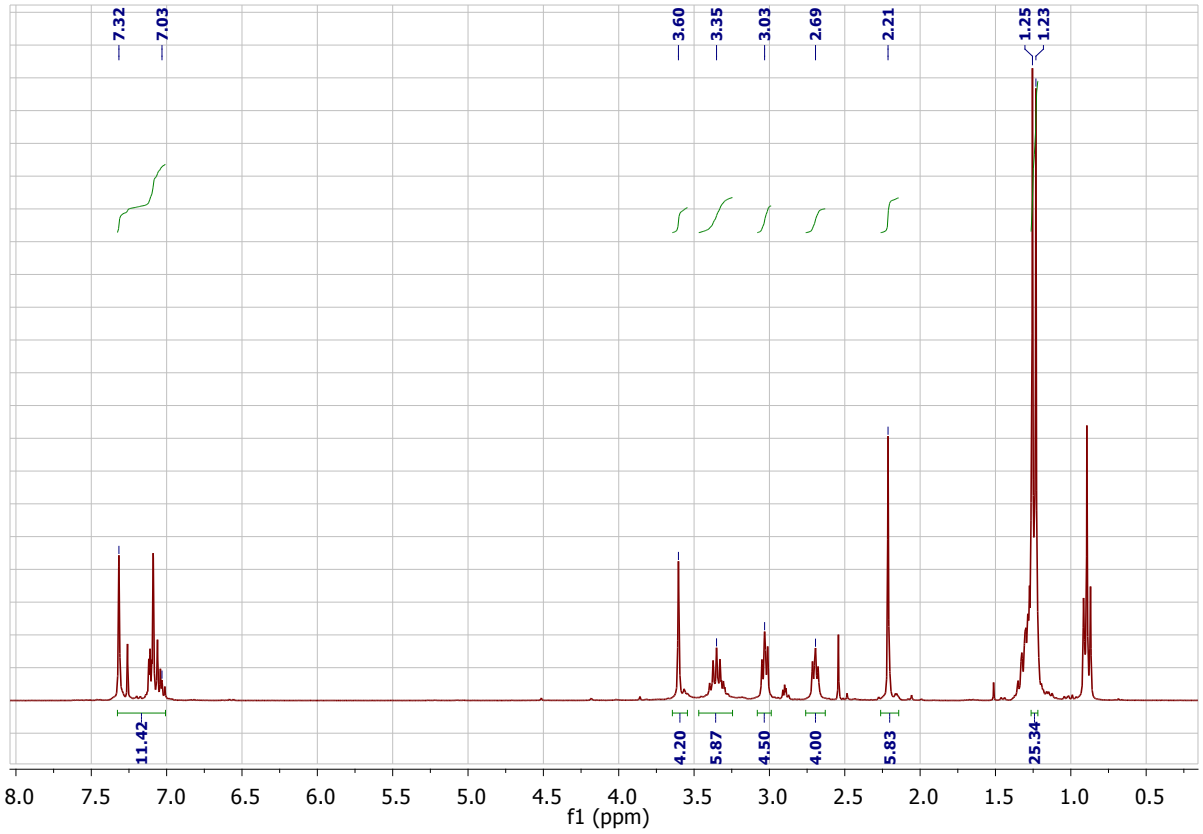

Figure S76 ^{1}H NMR spectrum (400 MHz) of 2g in $CDCl_3$ at 300 K including impurities of *n*-pentane.

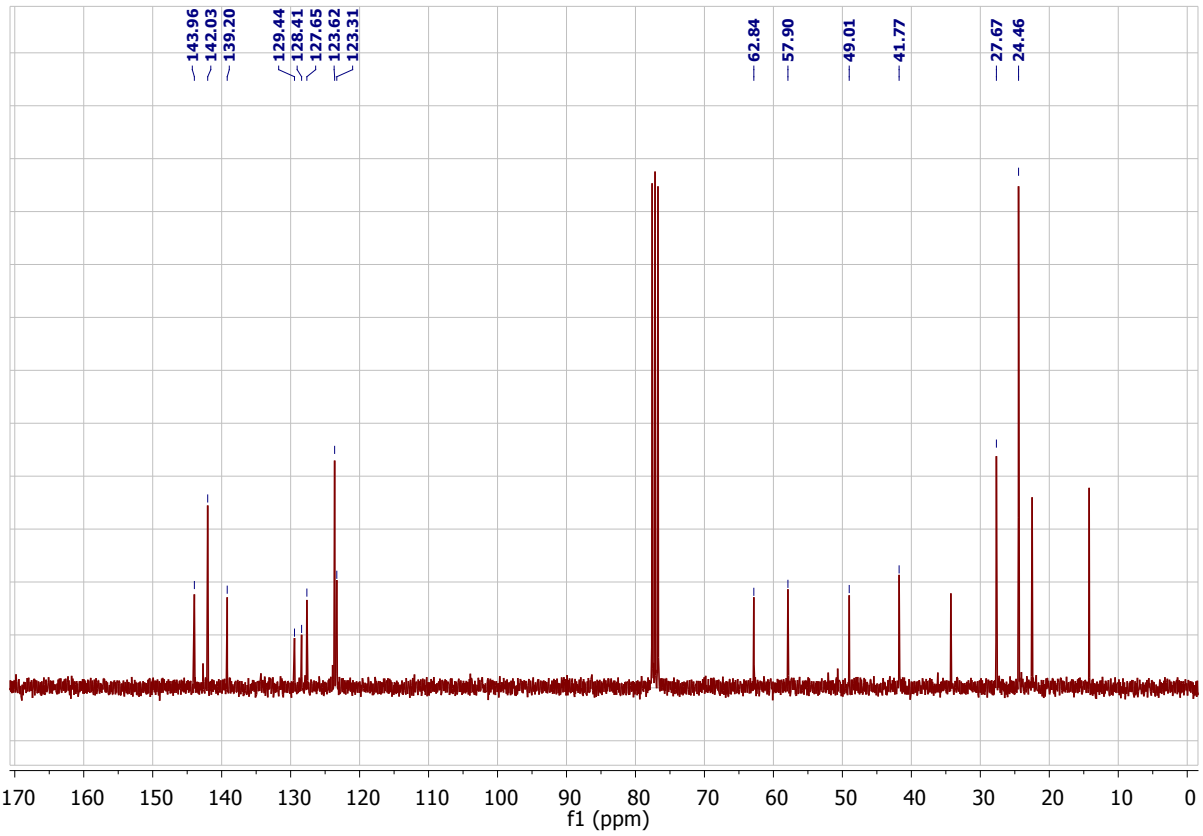

Figure S77 ^{13}C NMR spectrum (101 MHz) of 2g in $CDCl_3$ at 300 K containing impurities of *n*-pentane.

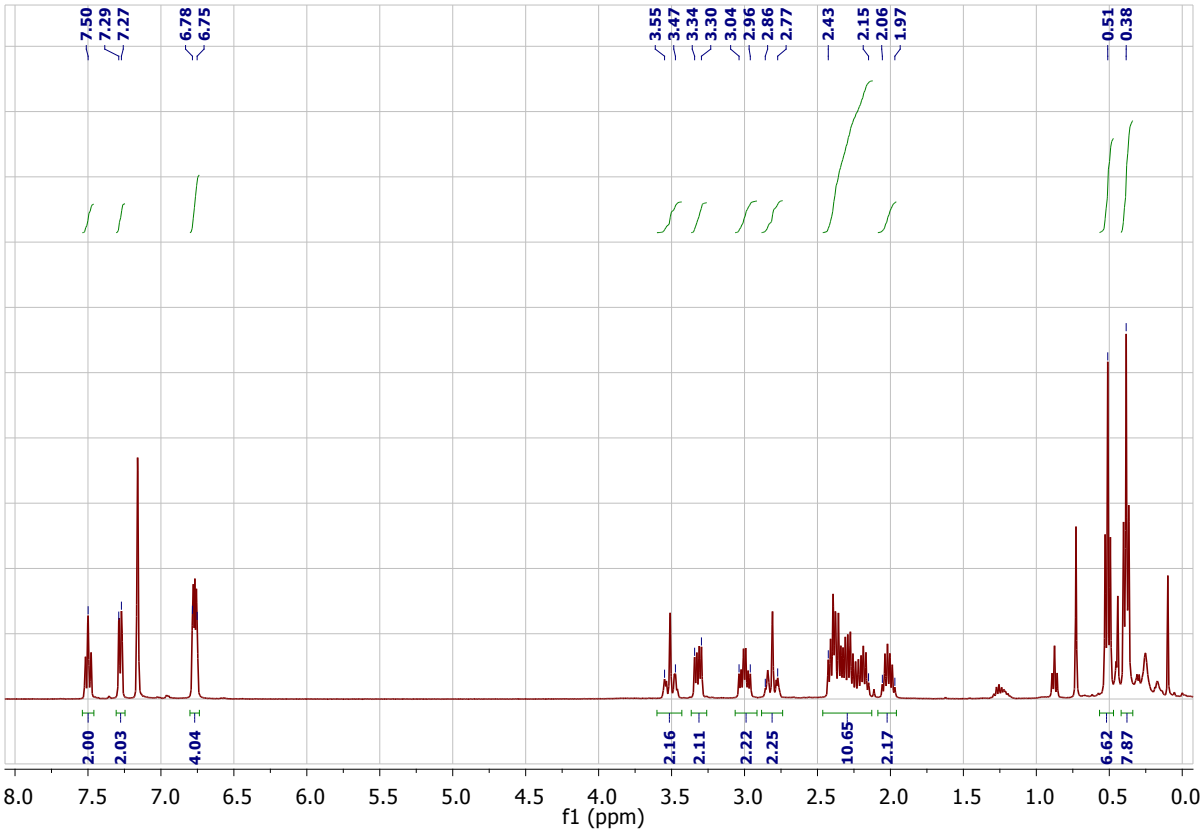

Figure S78 ^{1}H NMR spectrum (400 MHz) of 1nMg in C_6D_6 at 300 K.

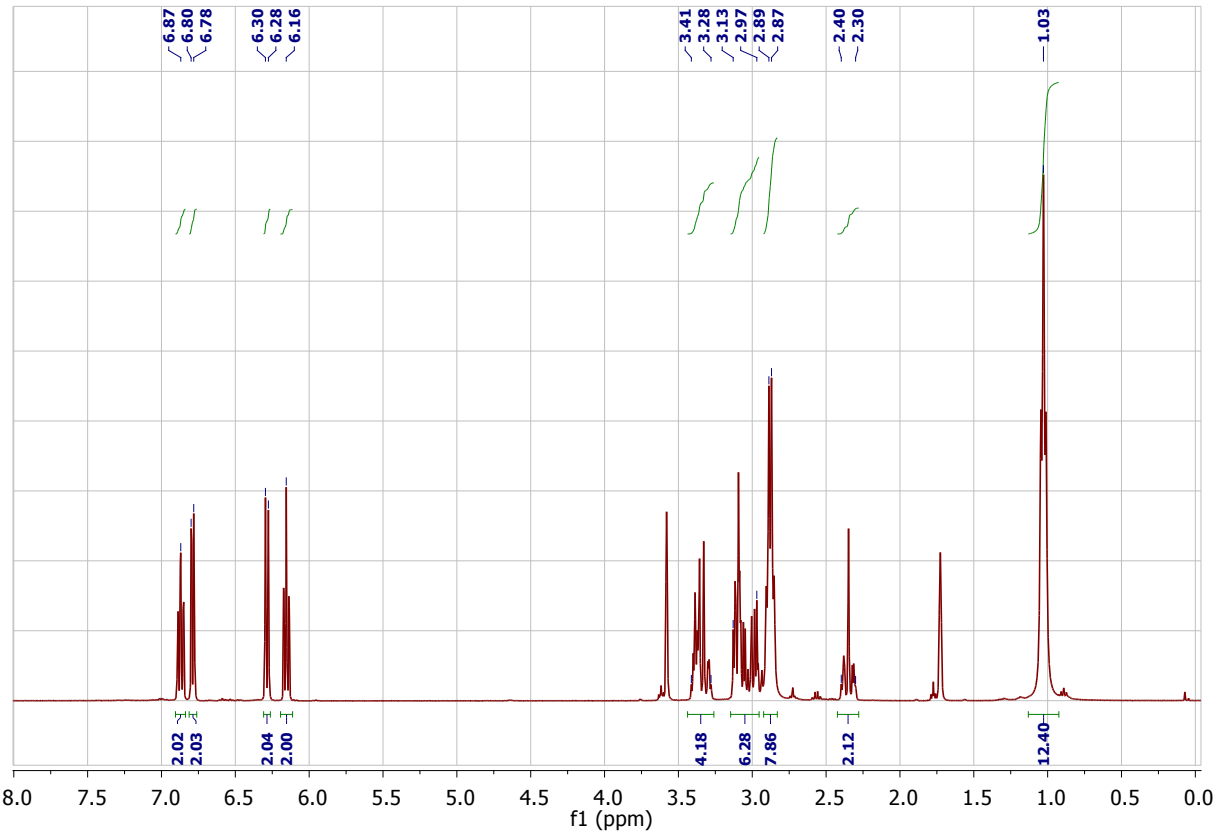

Figure S79 ^{1}H NMR spectrum (400 MHz) of 1nMg in THF-d_8 at 300 K.

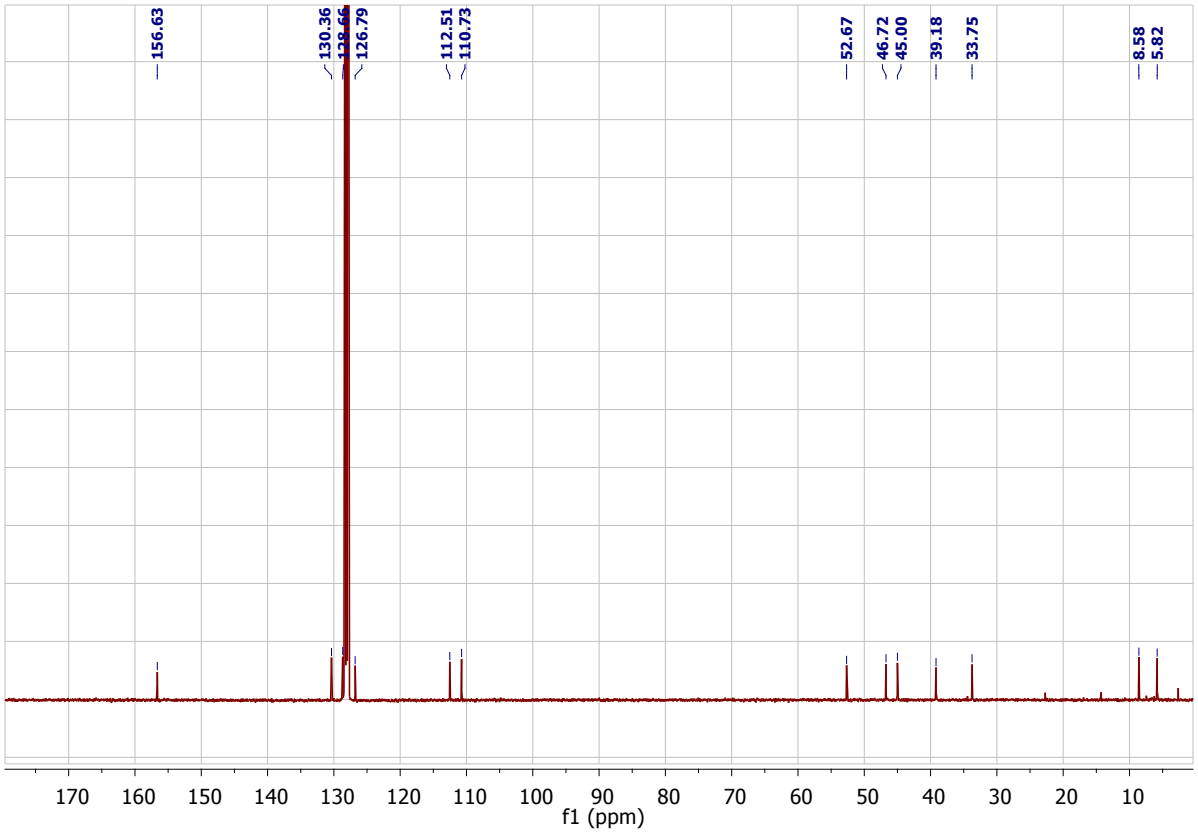

Figure S80 ^{13}C NMR spectrum (101 MHz) of 1nMg in C_6D_6 at 300 K.

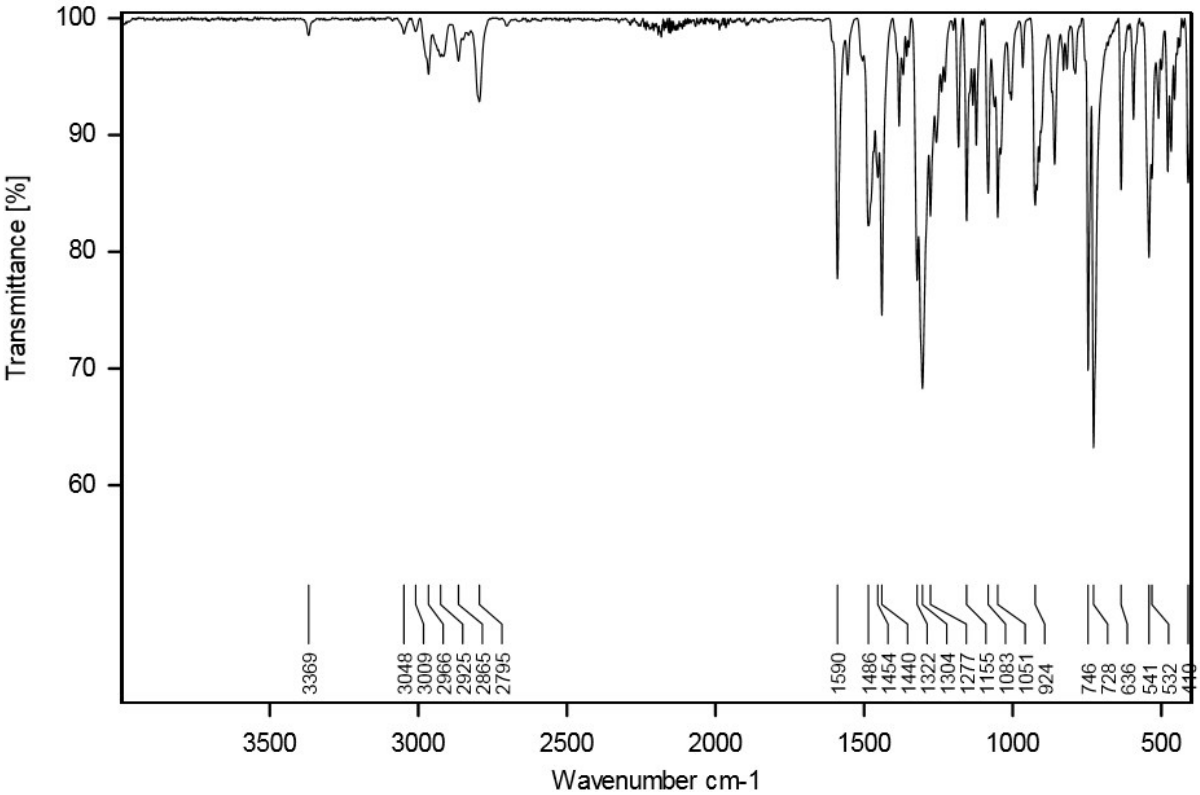

Figure S81 ATR-IR spectrum of 1nMg.

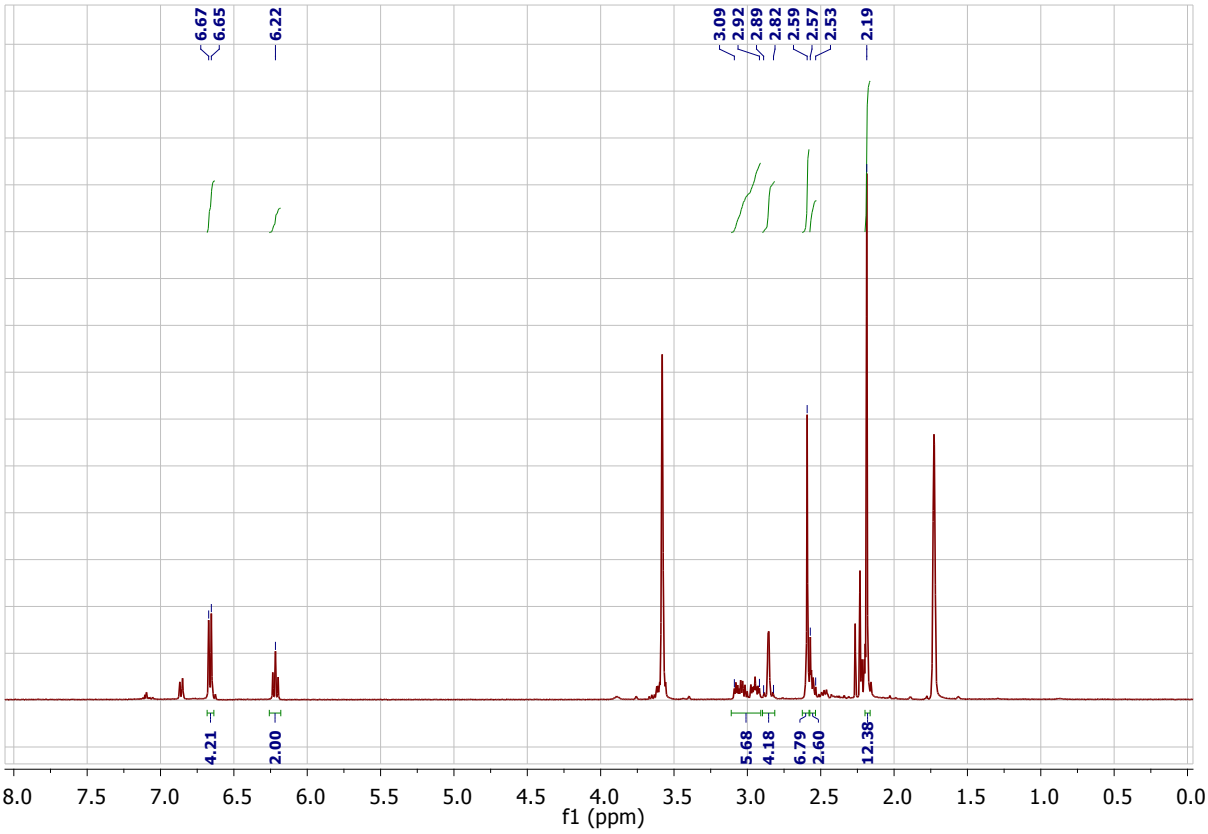

Figure S82 ^{1}H NMR spectrum (400 MHz) of 2bMg in THF-d_8 at 300 K.

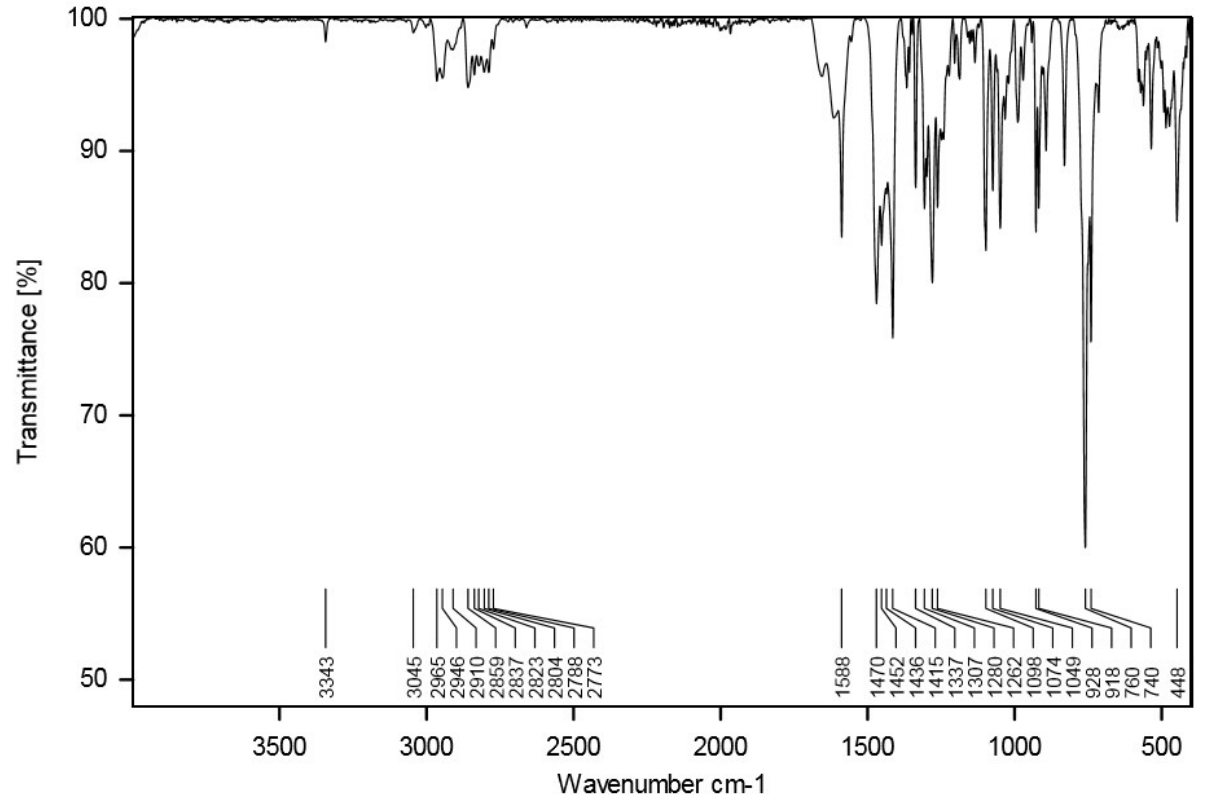

Figure S83 ATR-IR spectrum of 2bMg.

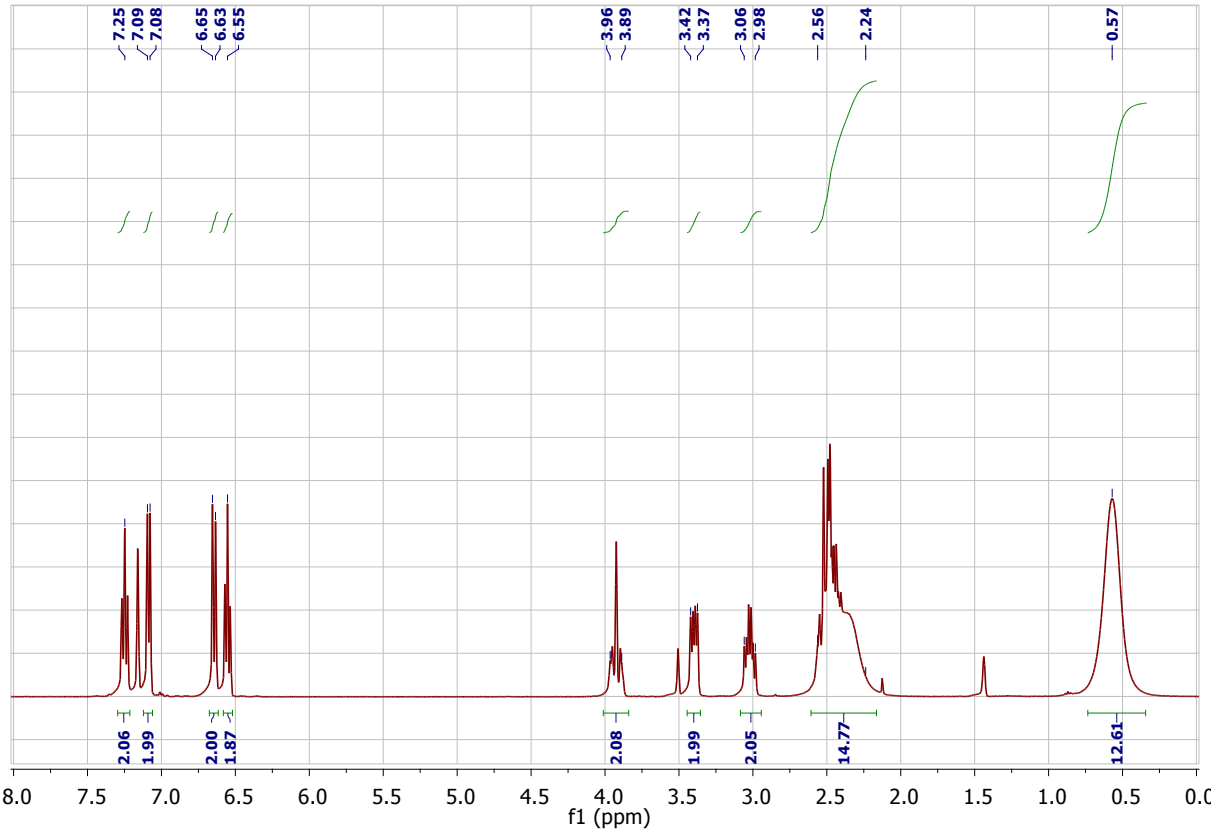

Figure S84 ^{1}H NMR spectrum (400 MHz) of 1nZn in C_6D_6+THF-d_8 at 300 K.

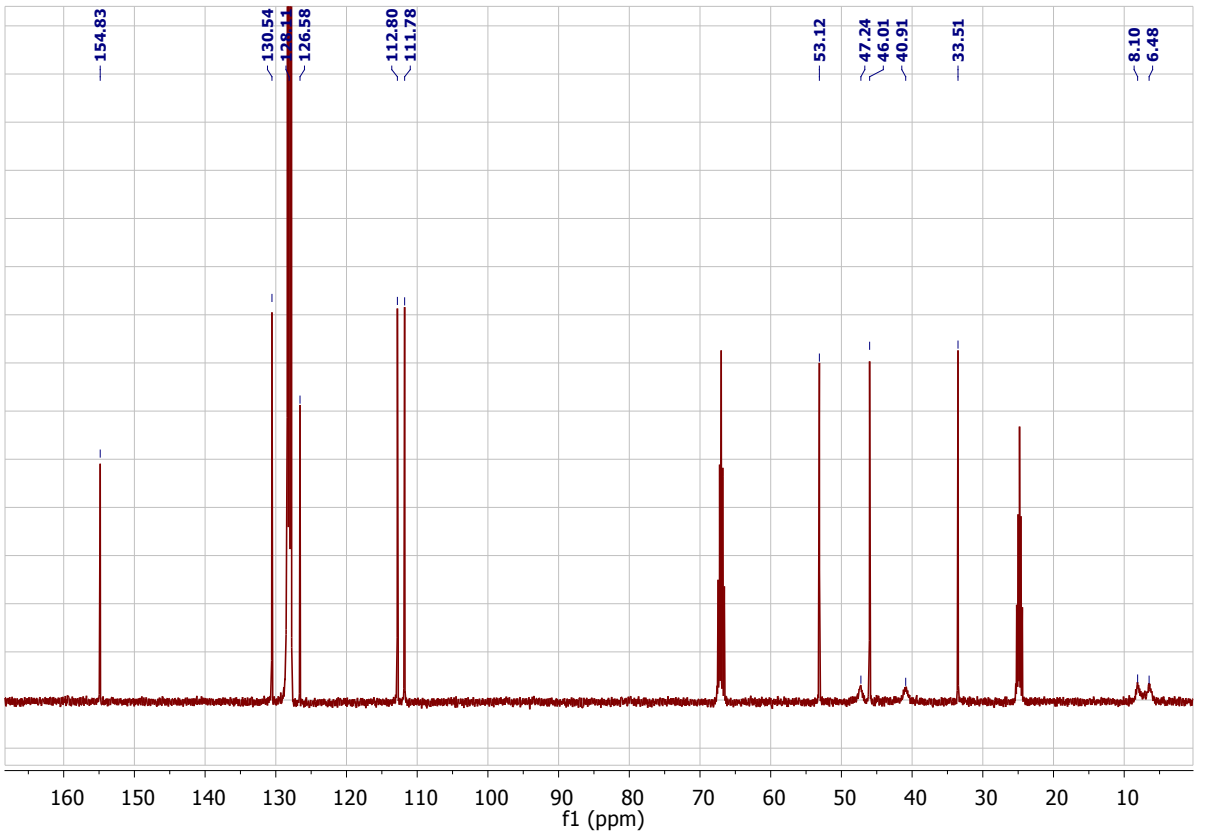

Figure S85 ^{13}C NMR spectrum (101 MHz) of 1nZn in C_6D_6+THF-d_8 at 300 K.

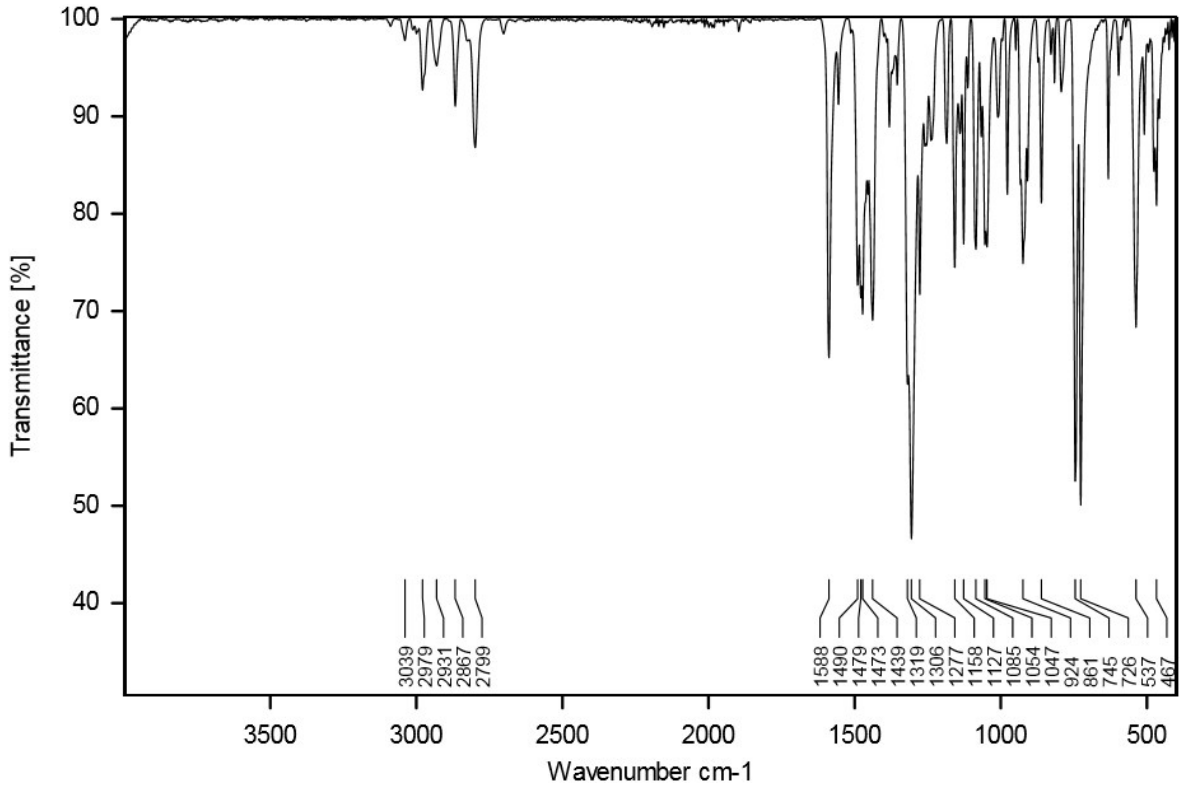

Figure S86 ATR-IR spectrum of 1nZn.

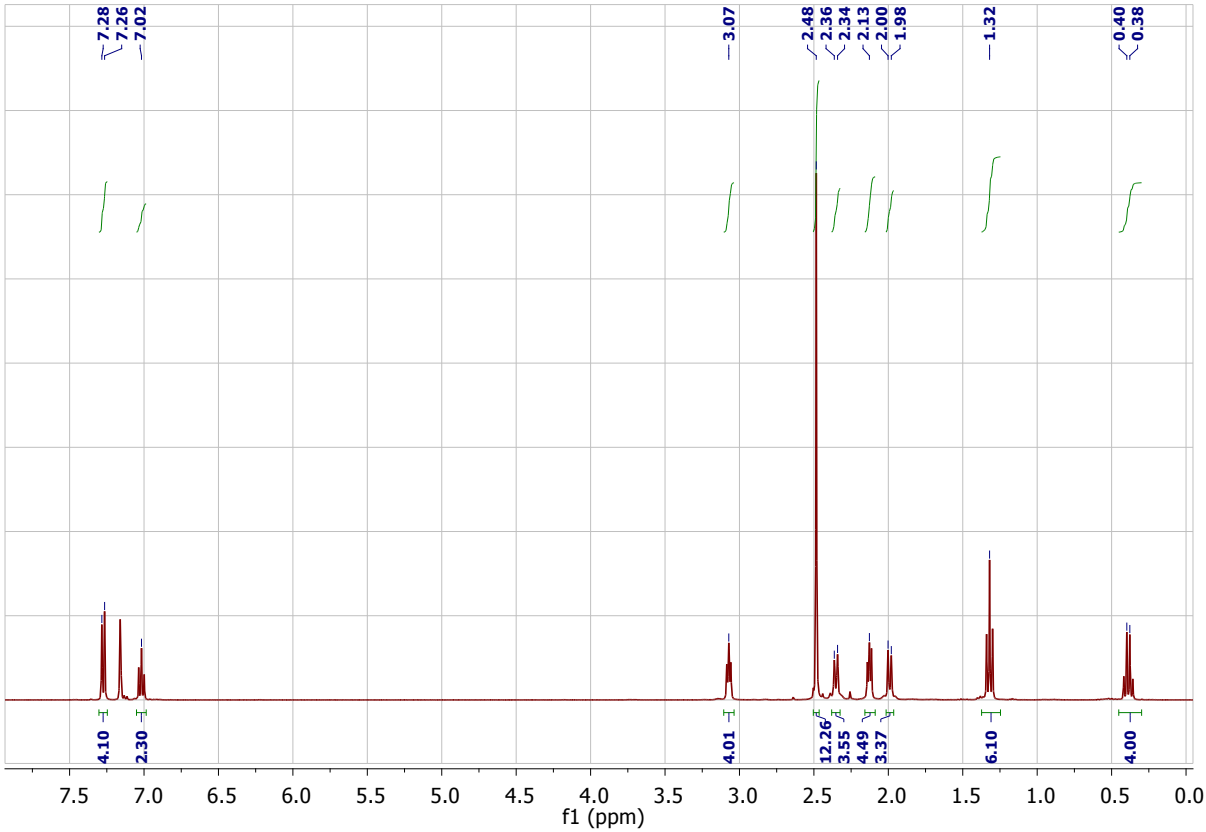

Figure S87 ^{1}H NMR spectrum (400 MHz) of 2aZnEt in C_6D_6 at 300 K.

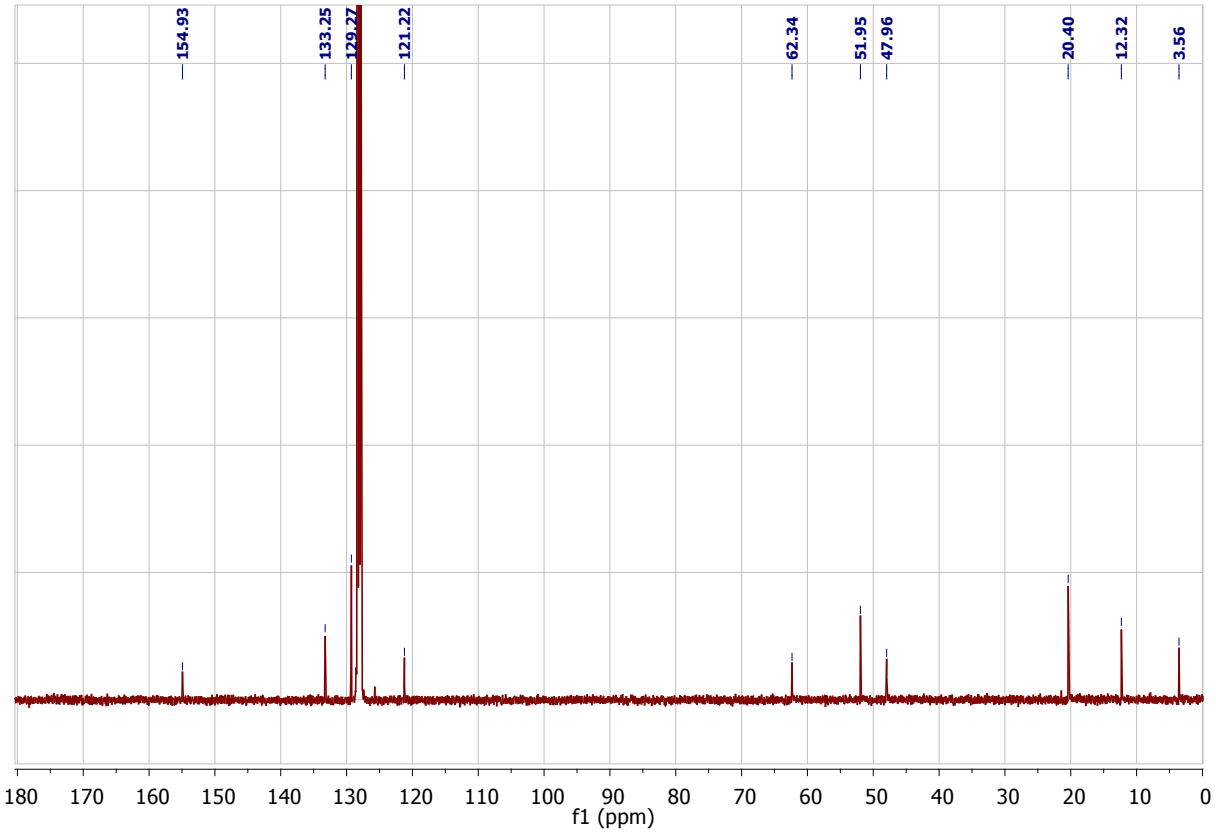

Figure S88 ^{13}C NMR spectrum (101 MHz) of 2aZnEt in C_6D_6 at 300 K.

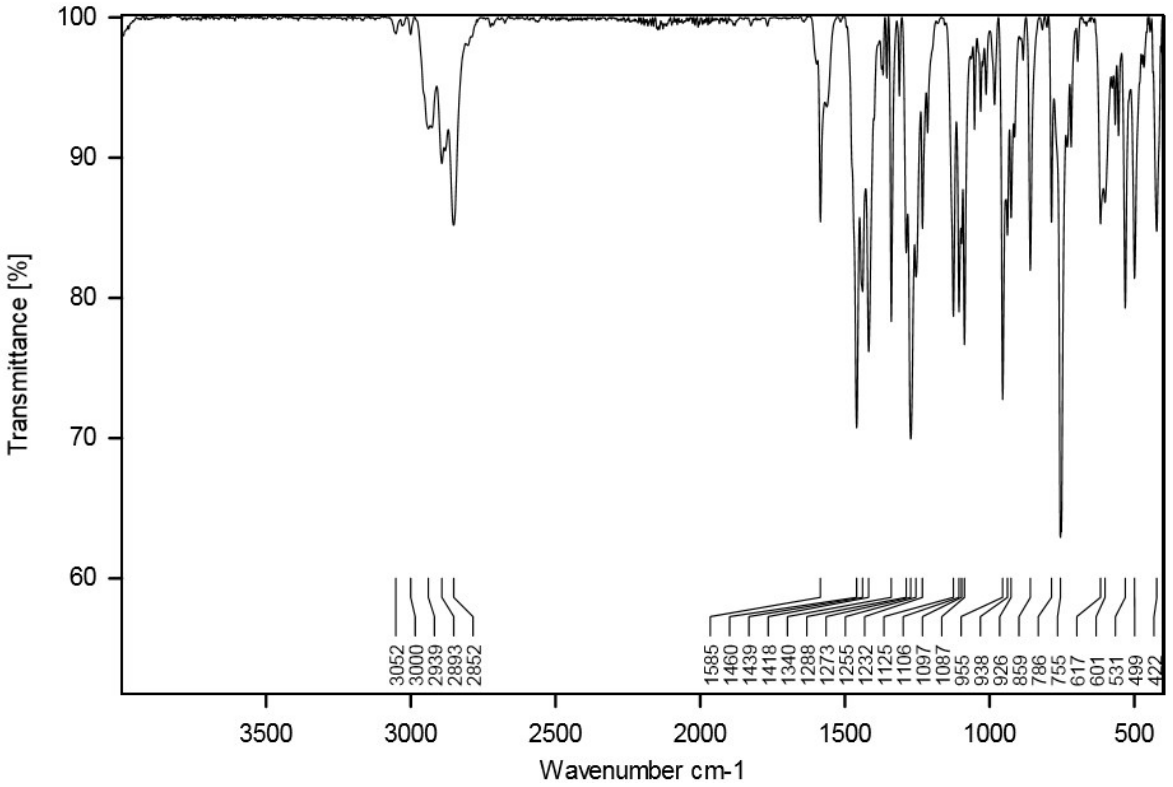

Figure S89 ATR-IR spectrum of 2aZnEt.

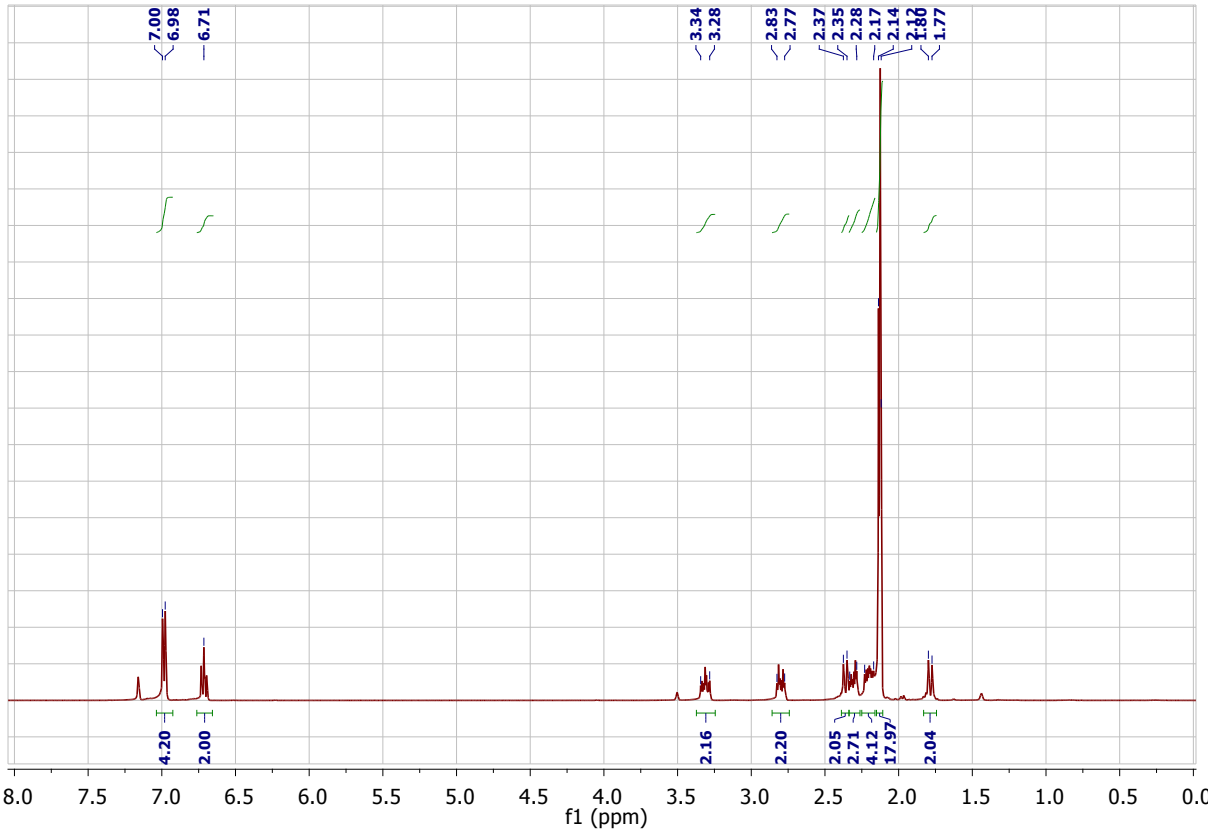

Figure S90 ^{1}H NMR spectrum (400 MHz) of 2bZn in C_6D_6+THF-d_8 at 300 K.

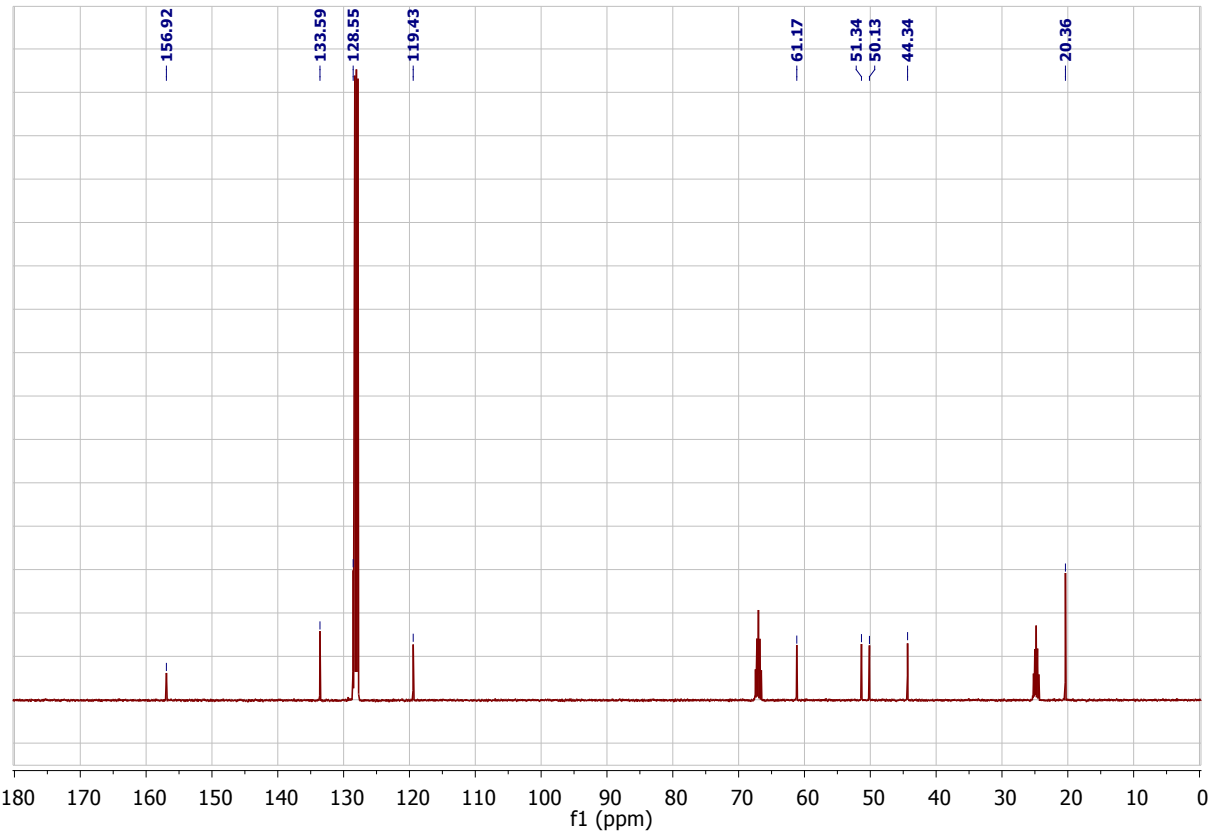

Figure S91 ^{13}C NMR spectrum (101 MHz) of 2bZn in C_6D_6+THF-d_8 at 300 K.

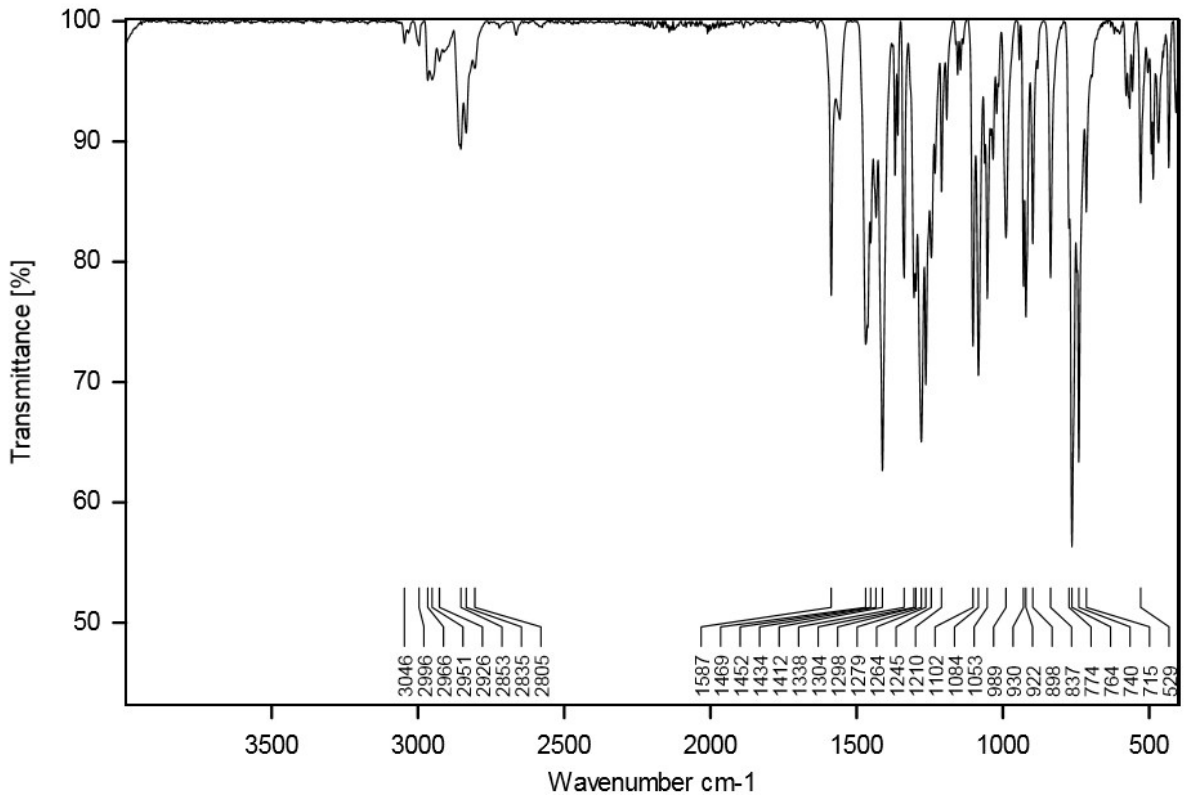

Figure S92 ATR-IR spectrum of 2bZn.

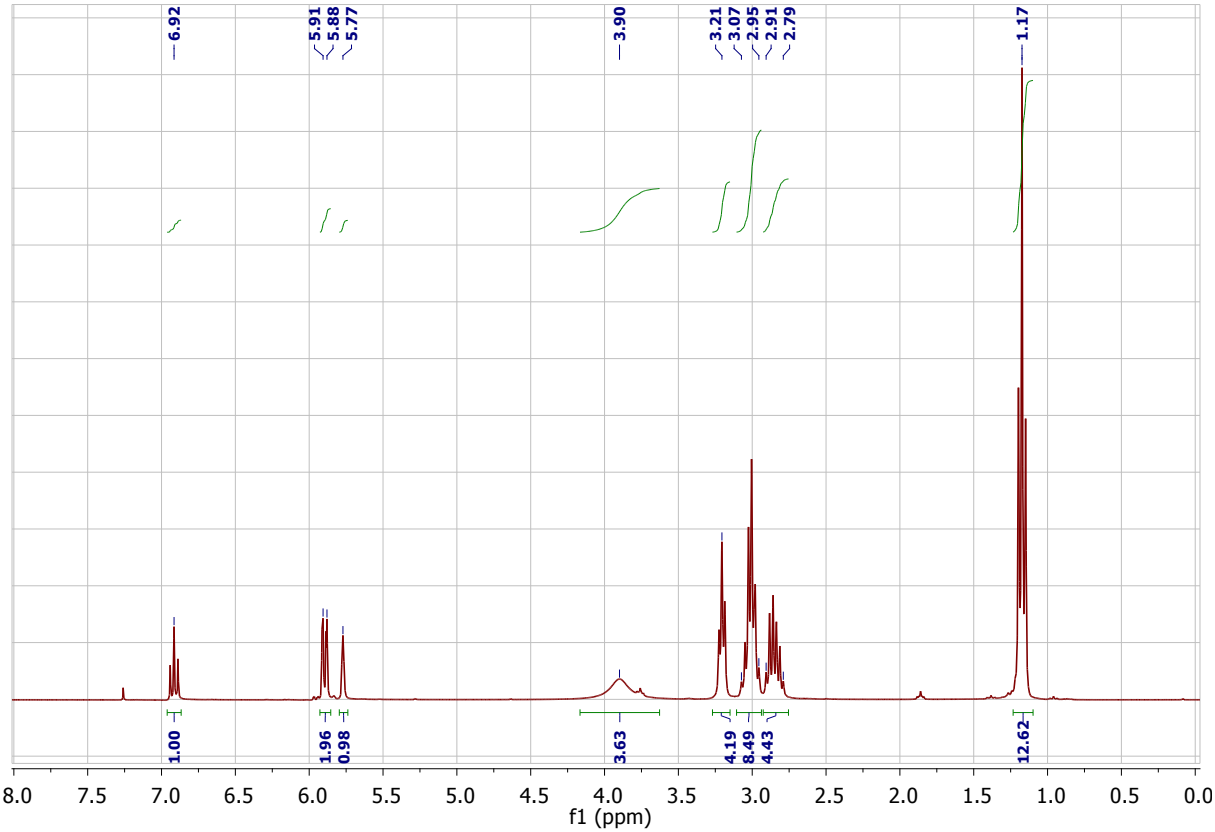

Figure S93 ^{1}H NMR spectrum (400 MHz) of 1jAlH_2 in $CDCl_3$ at 300 K.

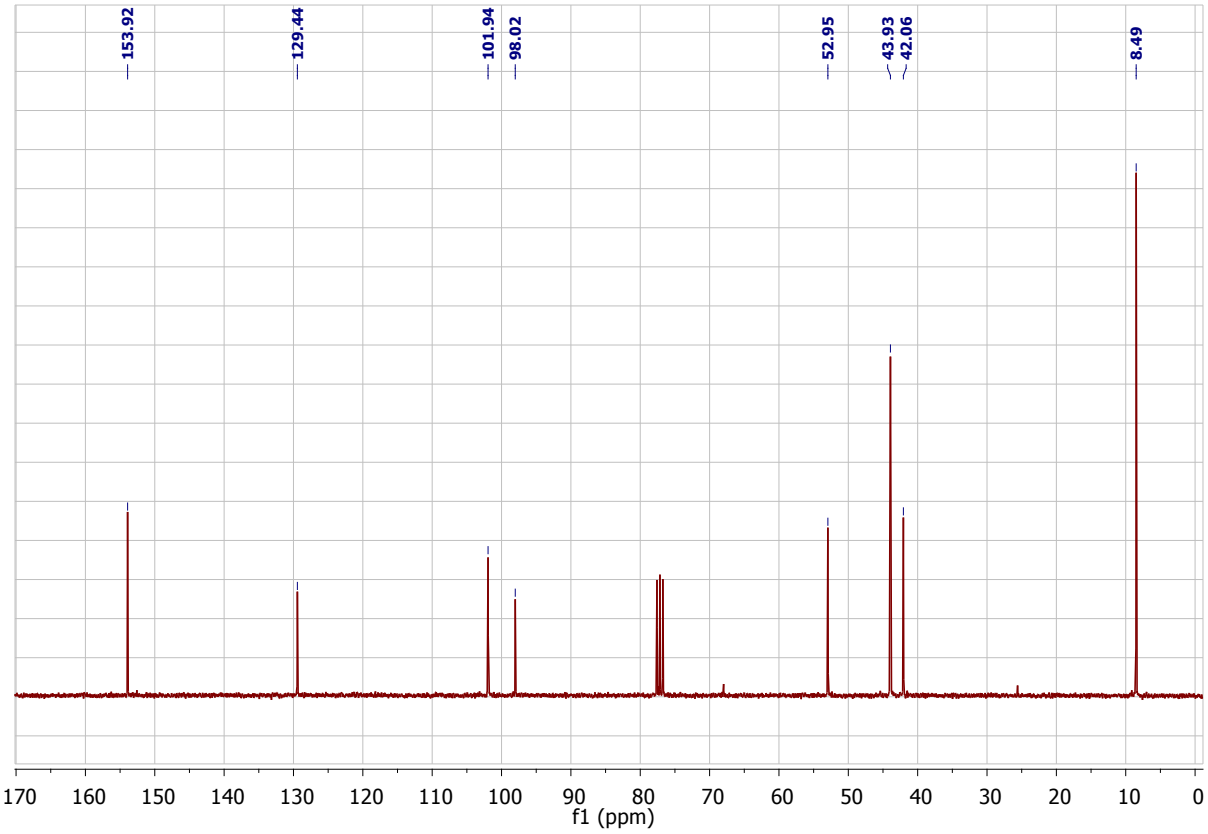

Figure S94 ^{13}C NMR spectrum (101 MHz) of $1jAlH_2$ in $CDCl_3$ at 300 K.

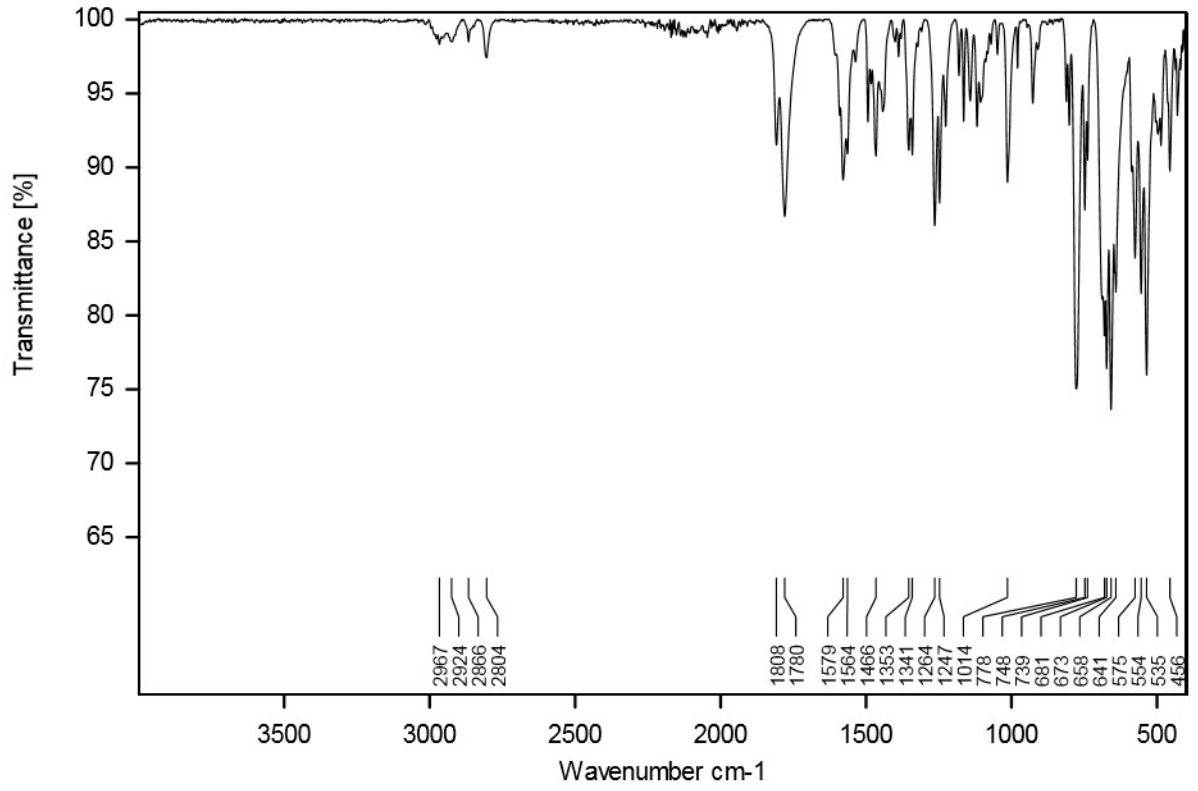

Figure S95 ATR-IR spectrum of $1jAlH_2$.

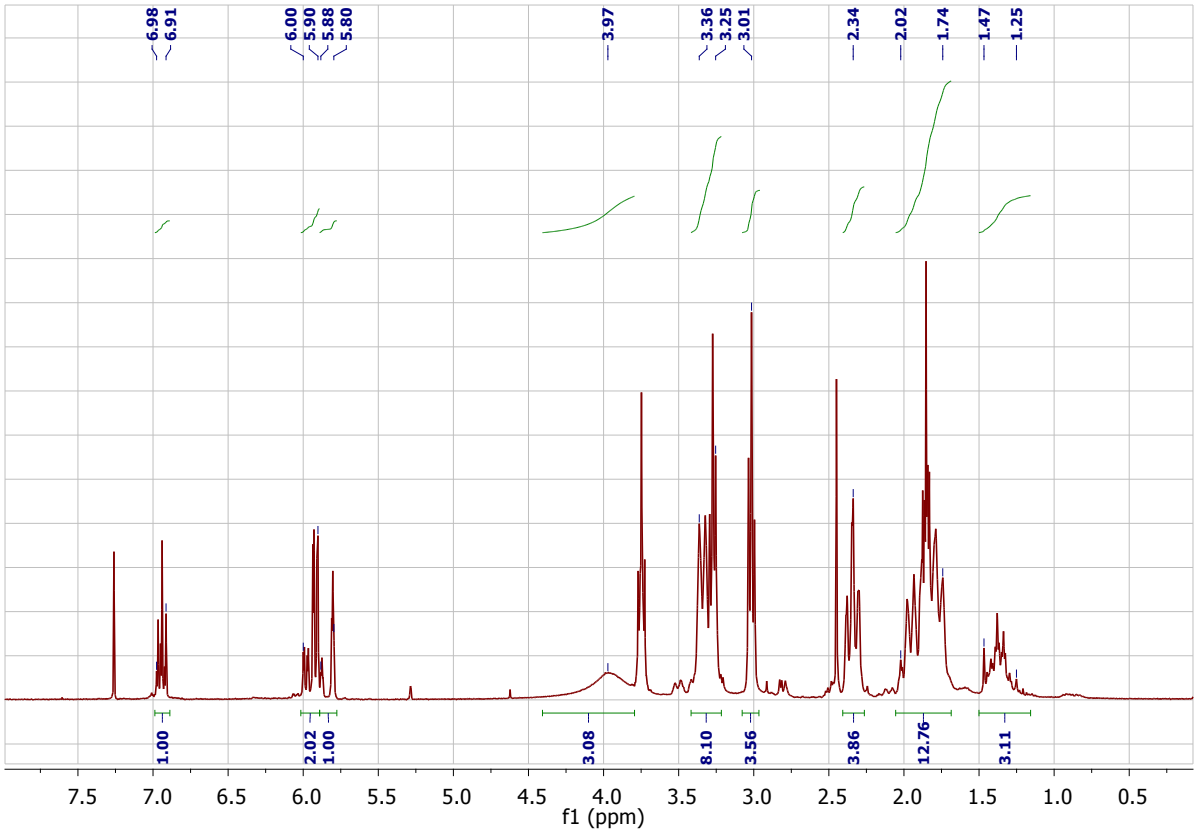

Figure S96 ^{1}H NMR spectrum (400 MHz) of 1kAlH$_2$ in CDCl$_3$ at 300 K.

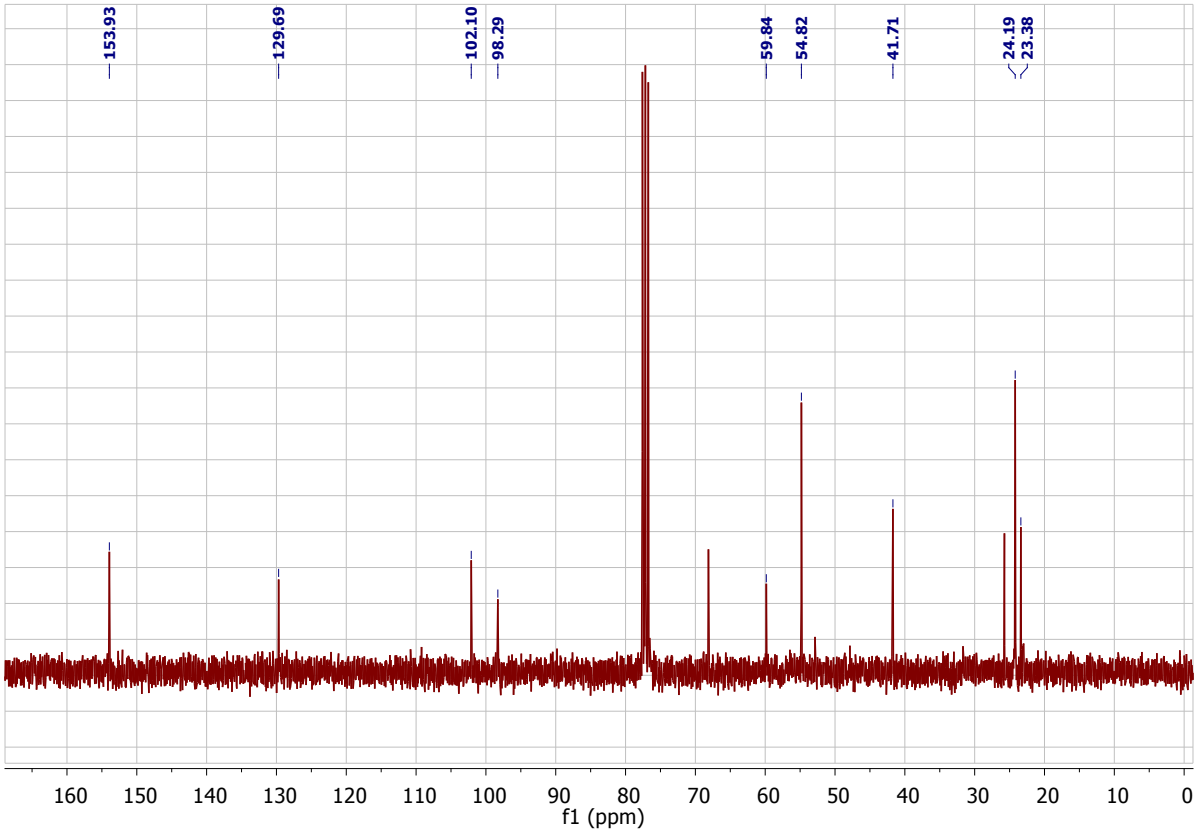

Figure S97 ^{13}C NMR spectrum (101 MHz) of 1kAlH$_2$ in CDCl$_3$ at 300 K.

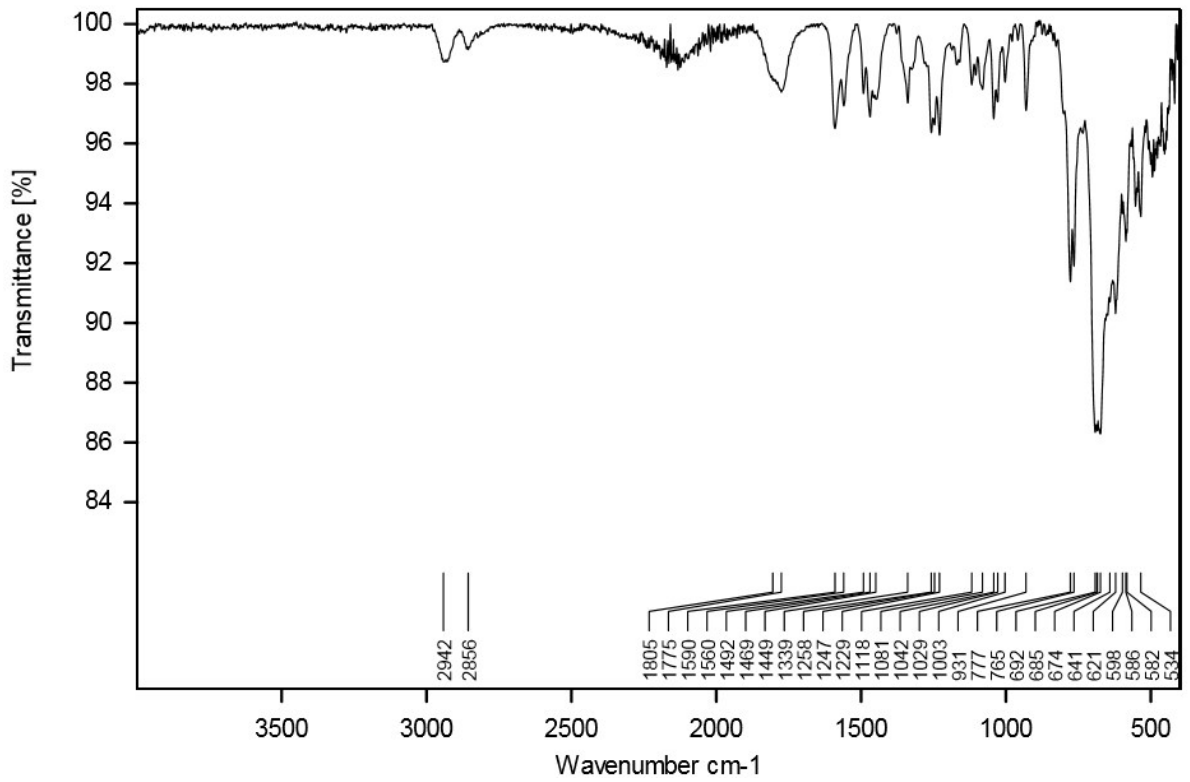

Figure S 98 ATR-IR spectrum of 1kAlH$_2$.

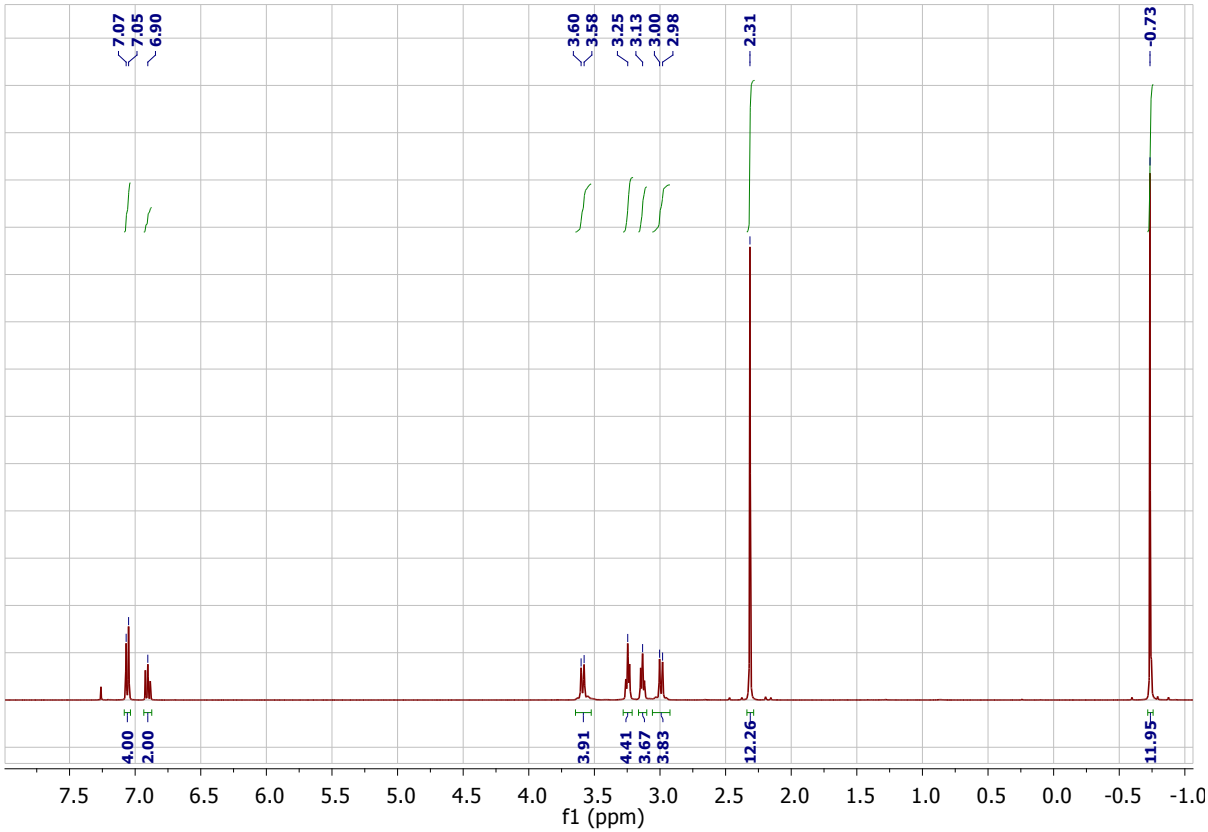

Figure S99 ^{1}H NMR spectrum (400 MHz) of 2aAlMe$_2$ in CDCl$_3$ at 300 K.

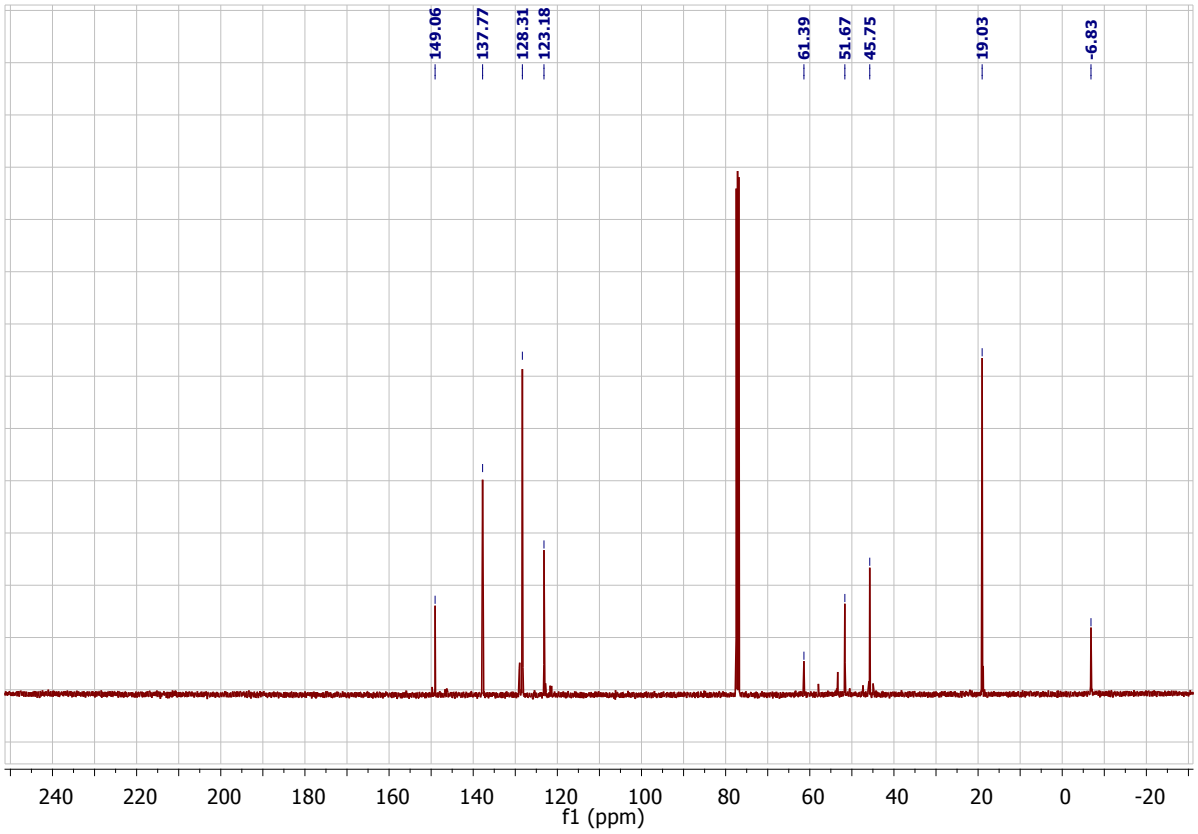

Figure S100 ^{13}C NMR spectrum (101 MHz) of 2aAlMe$_2$ in CDCl$_3$ at 300 K.

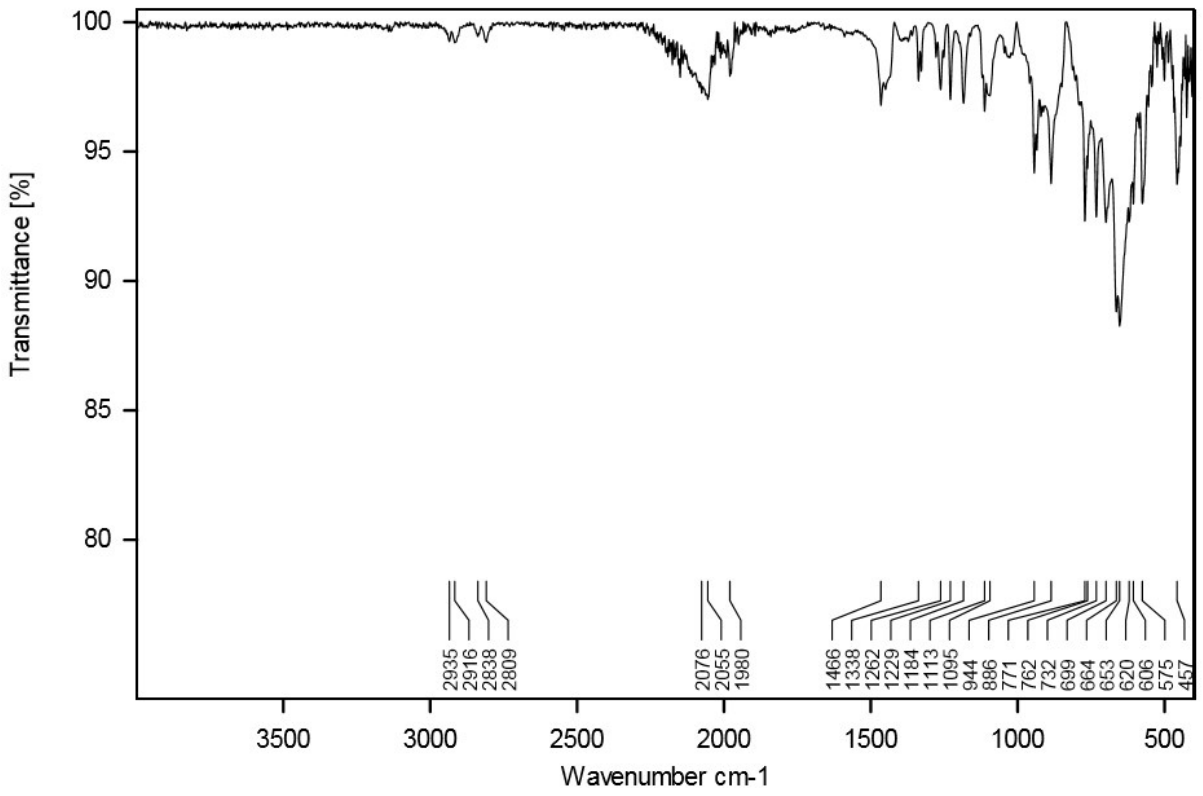

Figure S101 ATR-IR spectrum of 2aAlMe$_2$.

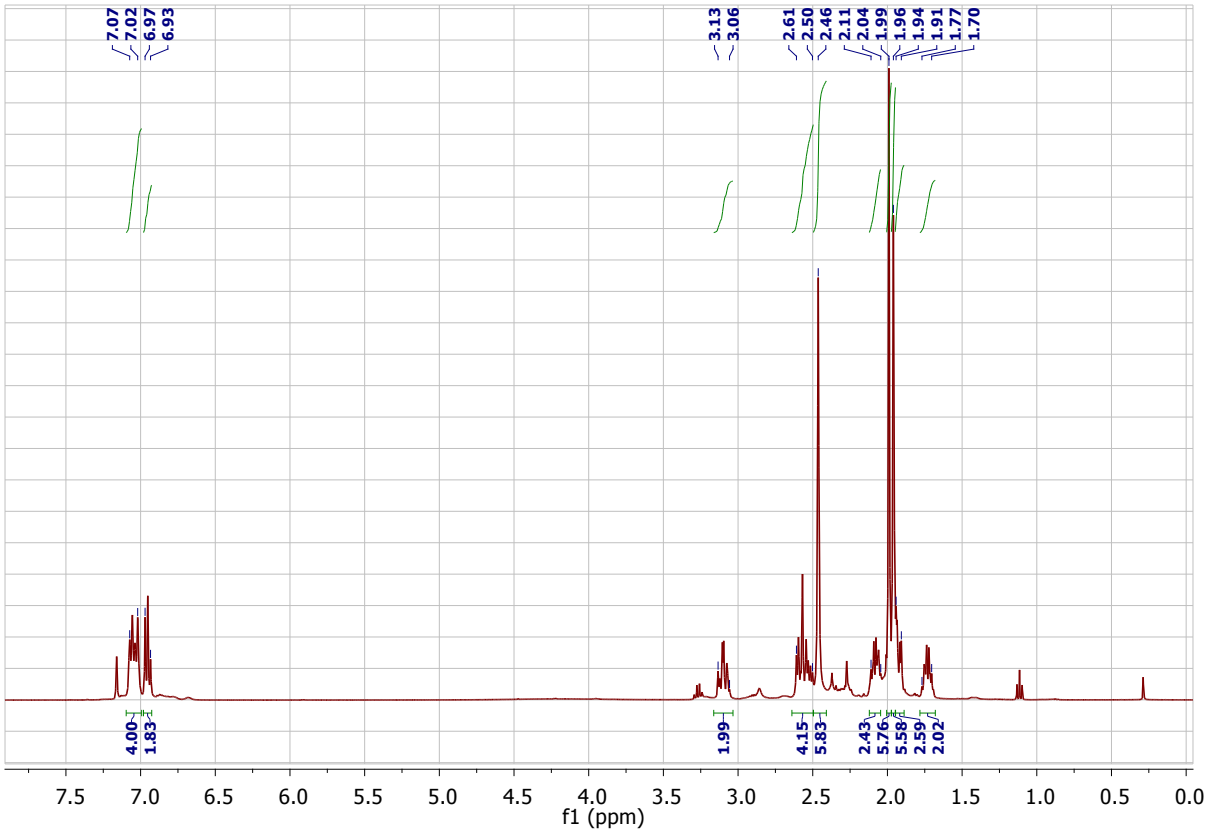

Figure S102 ^{1}H NMR spectrum (400 MHz) of 2bAlH in C_6D_6 at 300 K.

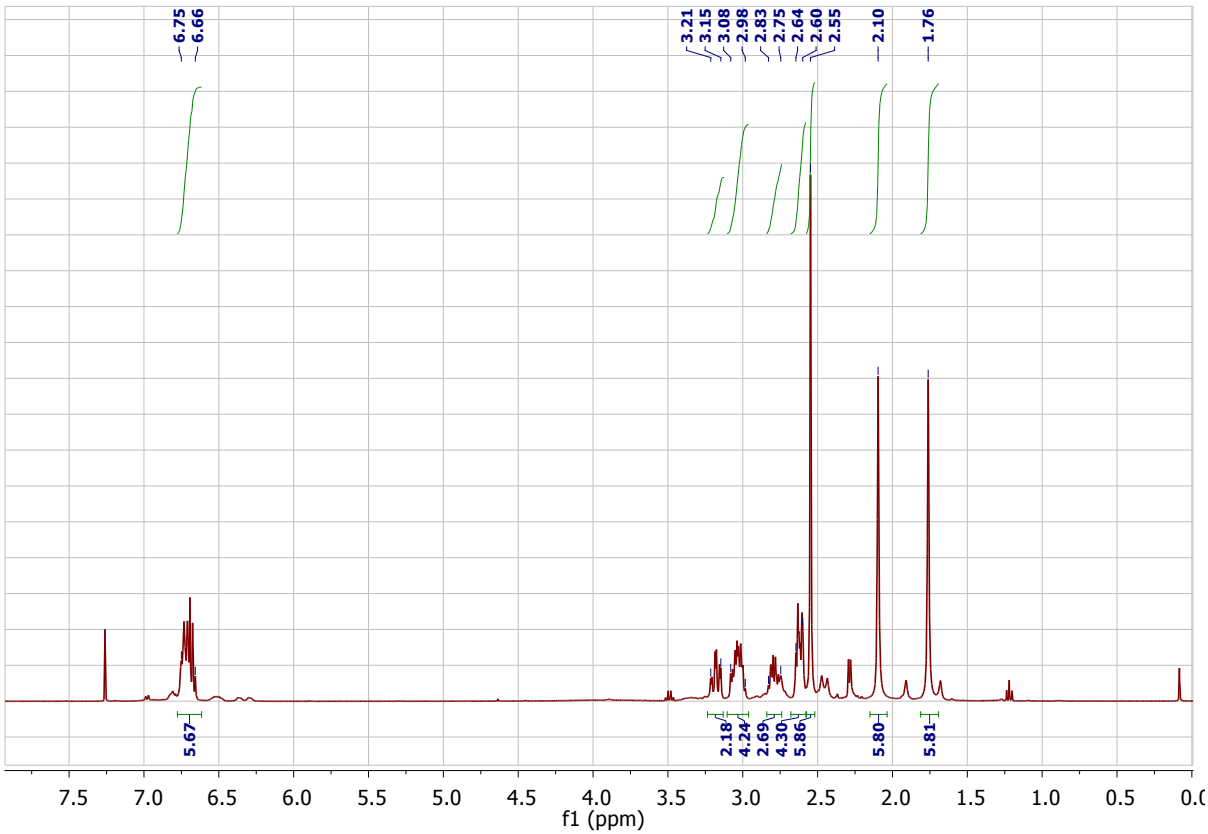

Figure S103 ^{1}H NMR spectrum (400 MHz) of 2bAlH in $CDCl_3$ at 300 K.

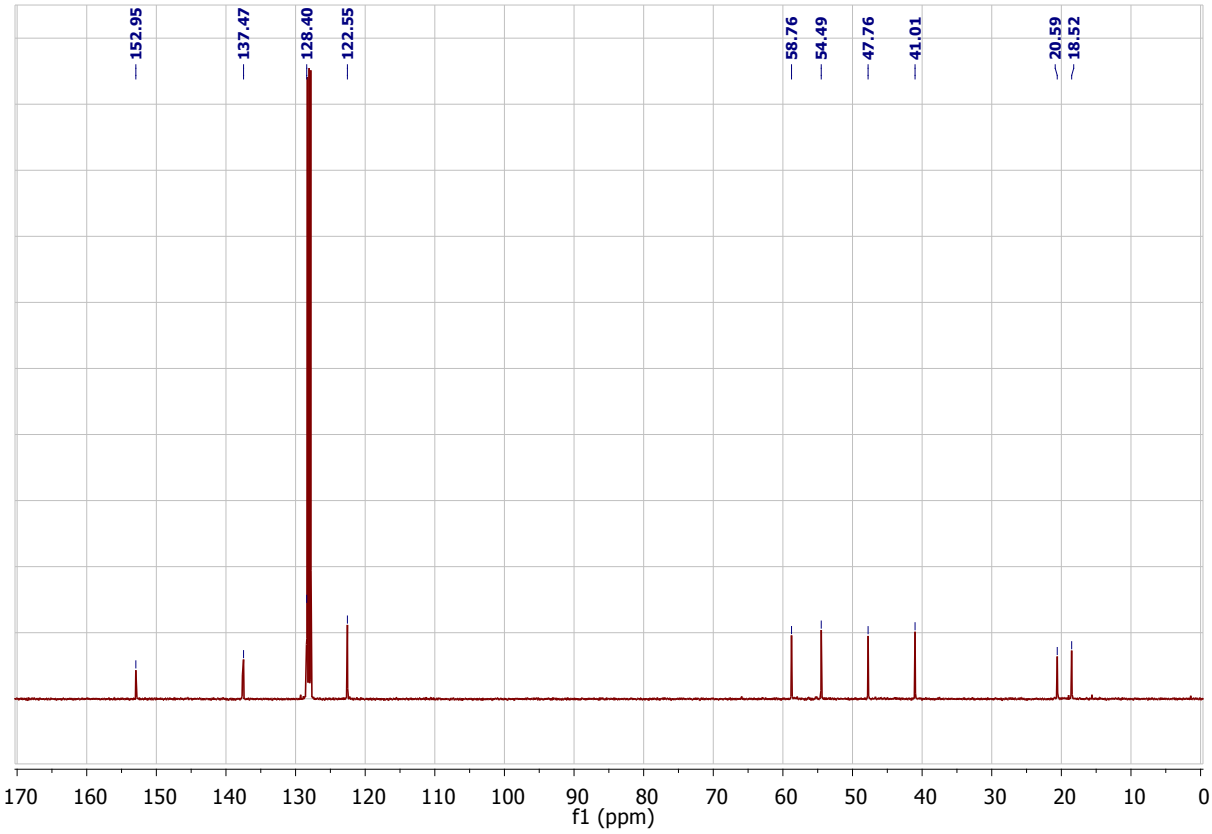

Figure S104 ^{13}C NMR spectrum (101 MHz) of 2bAlH in C_6D_6 at 300 K.

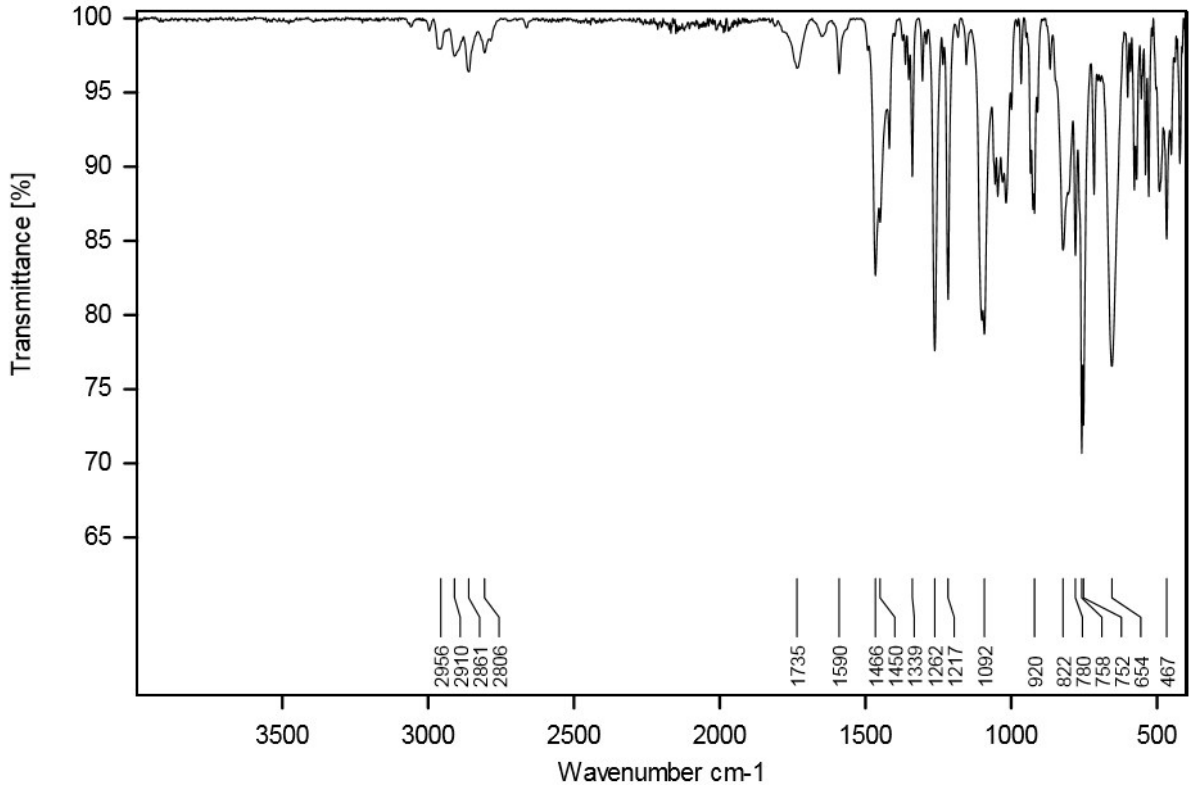

Figure S105 ATR-IR spectrum of 2bAlH.

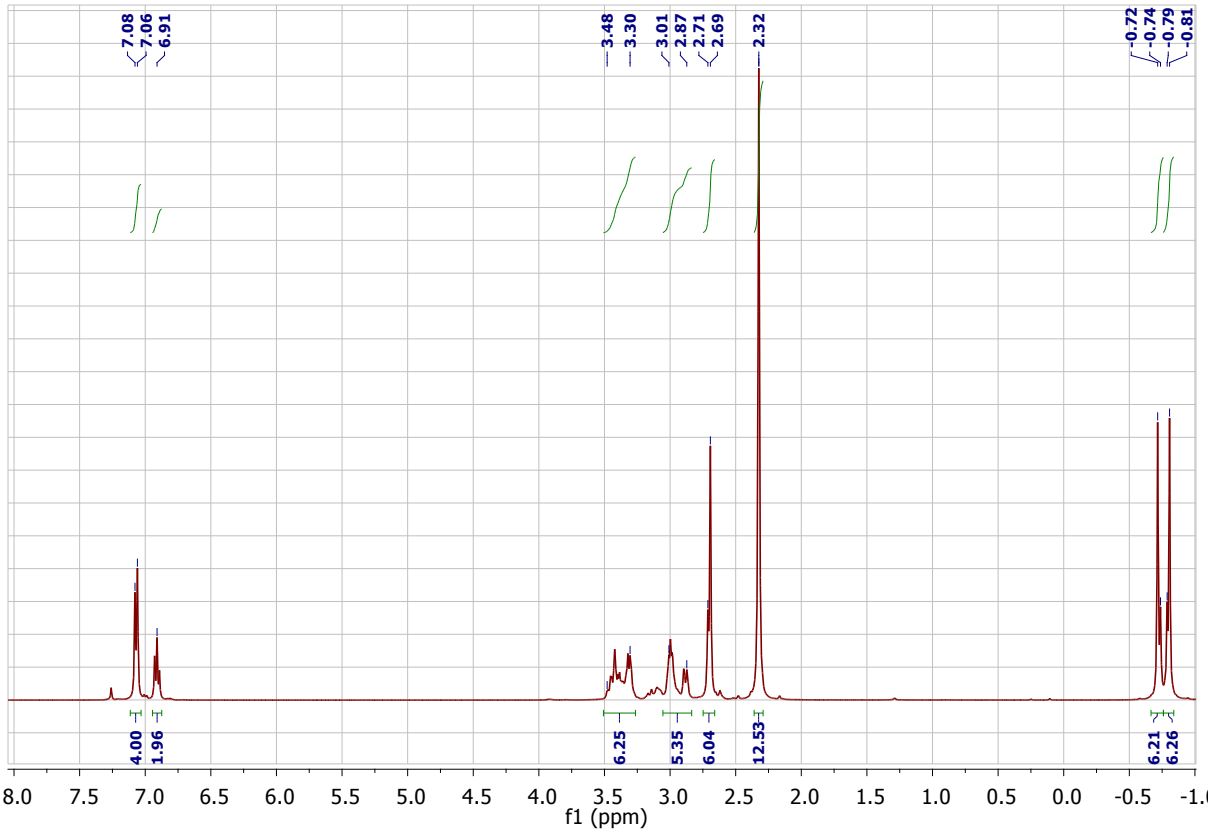

Figure S106 ^{1}H NMR spectrum (400 MHz) of 2bAlMe$_2$ in CDCl$_3$ at 300 K.

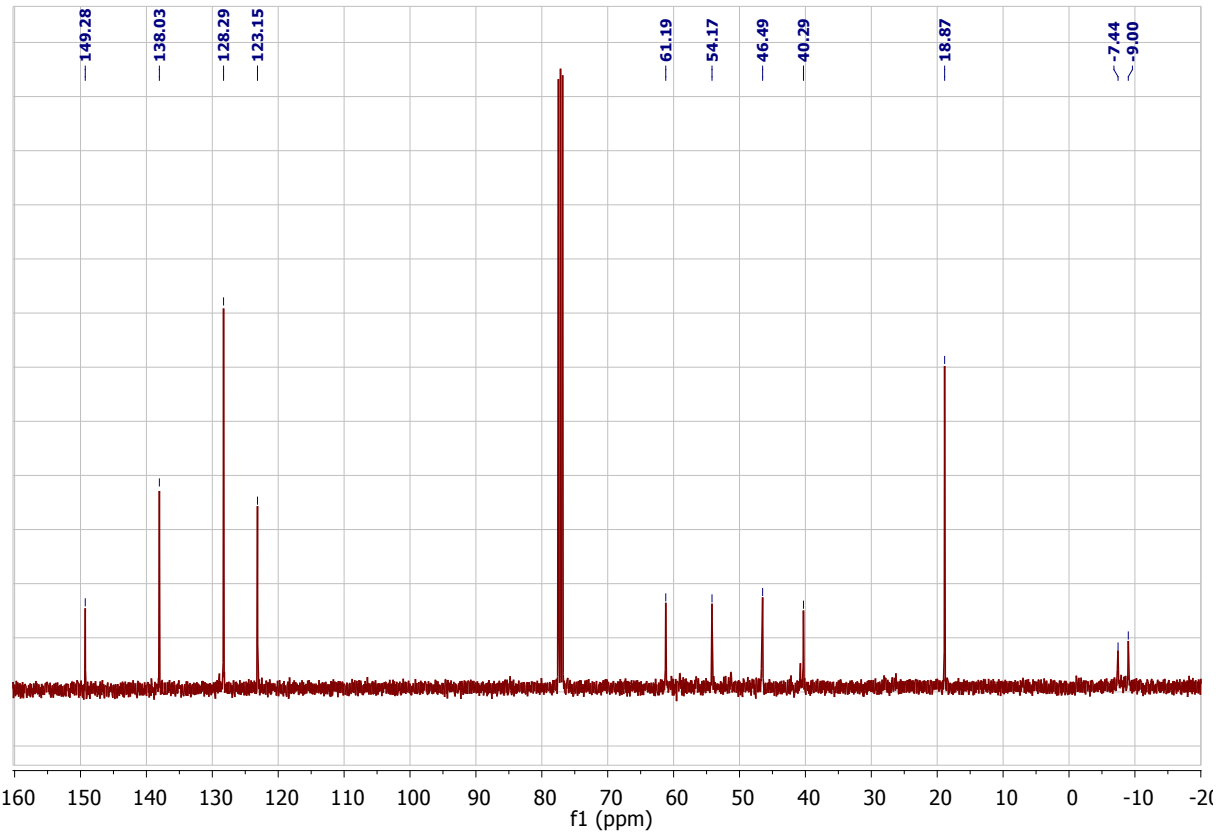

Figure S107 ^{13}C NMR spectrum (101 MHz) of 2bAlMe$_2$ in CDCl$_3$ at 300 K.

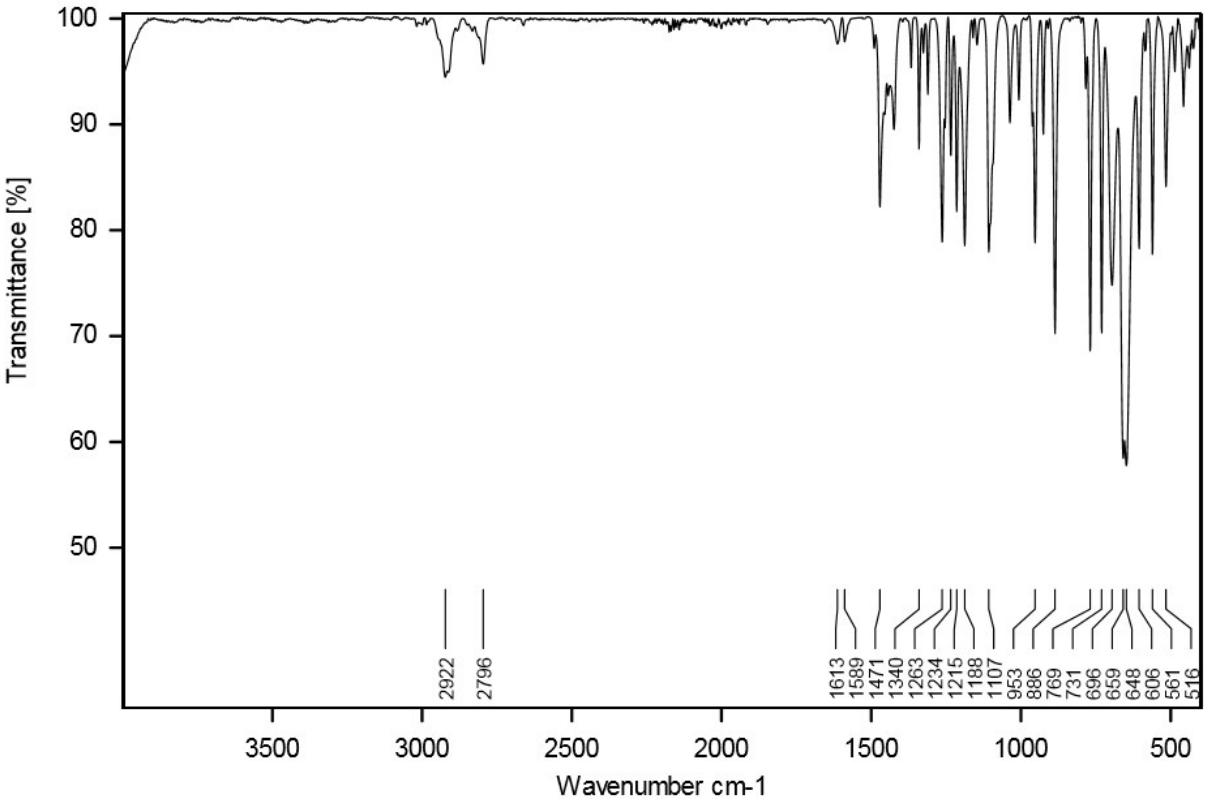

Figure S108 ATR-IR spectrum of 2bAlMe$_2$.

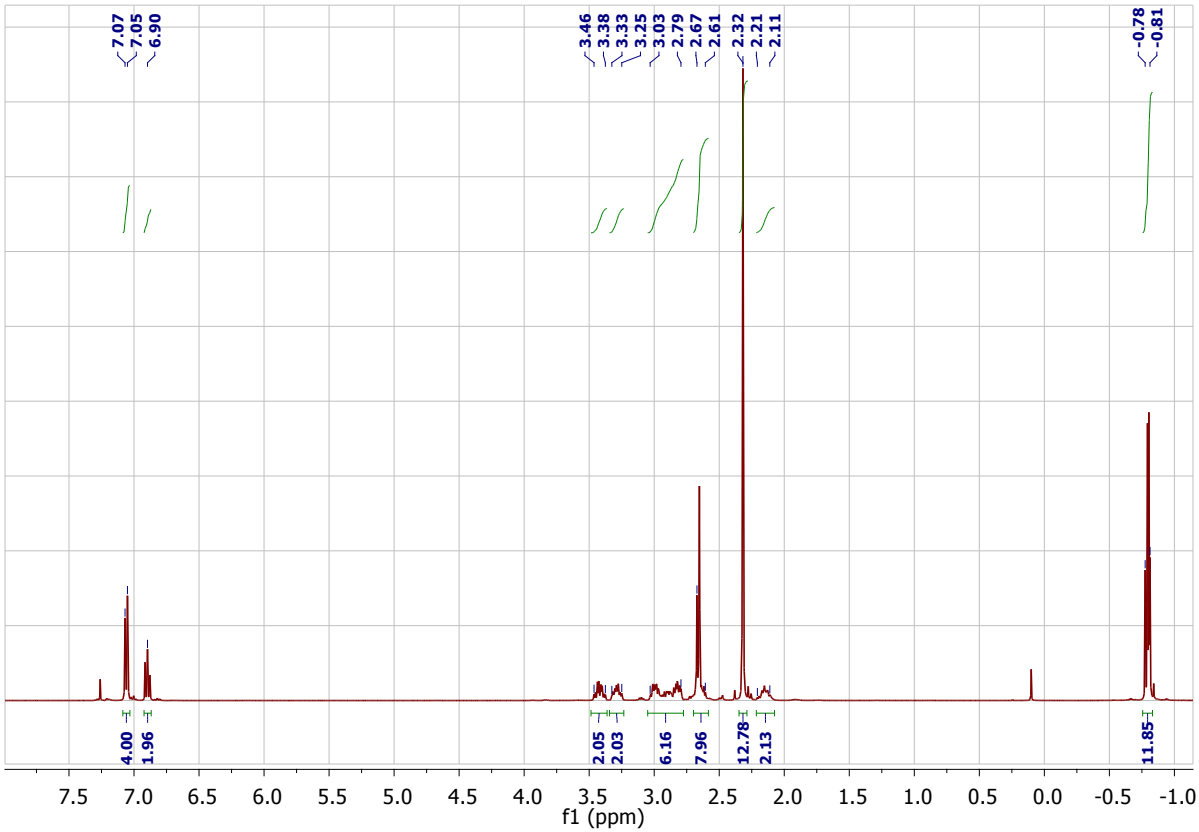

Figure S109 ^{1}H NMR spectrum (400 MHz) of 2dAlMe$_2$ in CDCl$_3$ at 300 K.

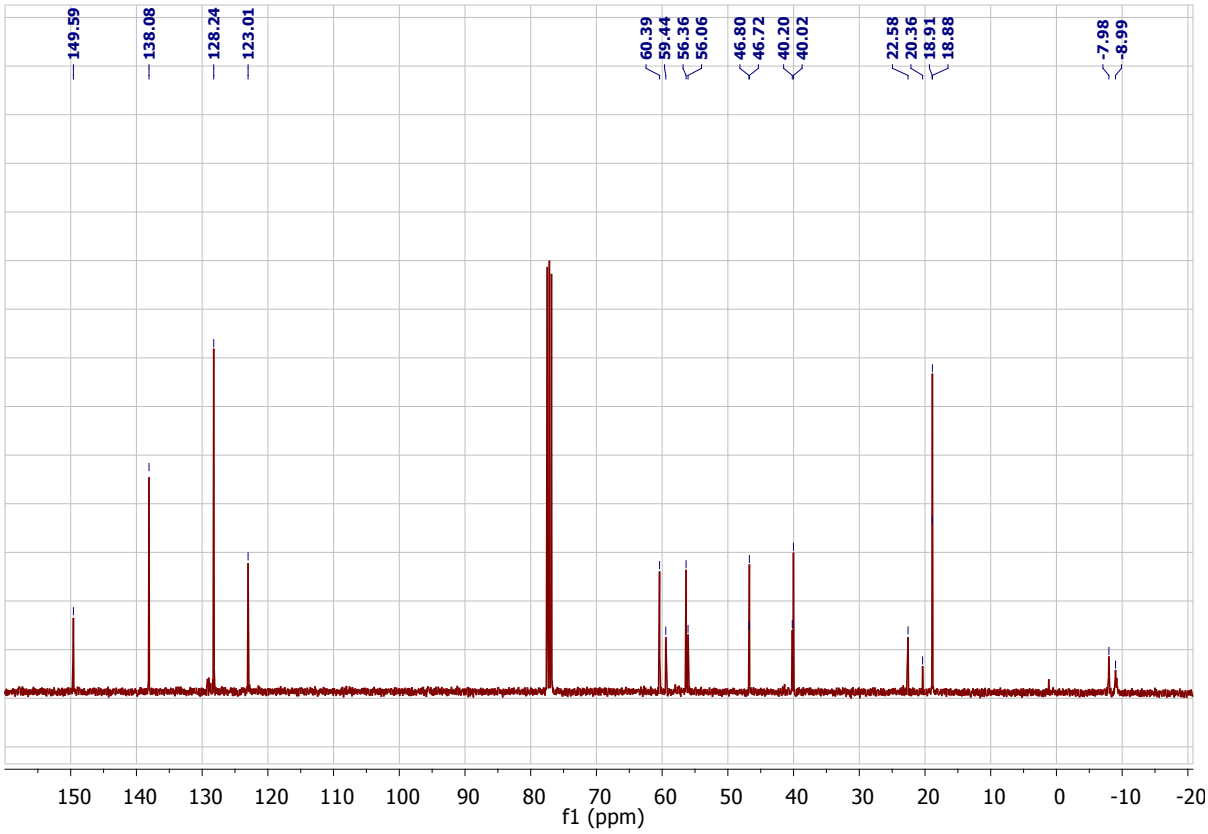

Figure S110 ^{13}C NMR spectrum (101 MHz) of 2dAlMe$_2$ in CDCl$_3$ at 300 K.

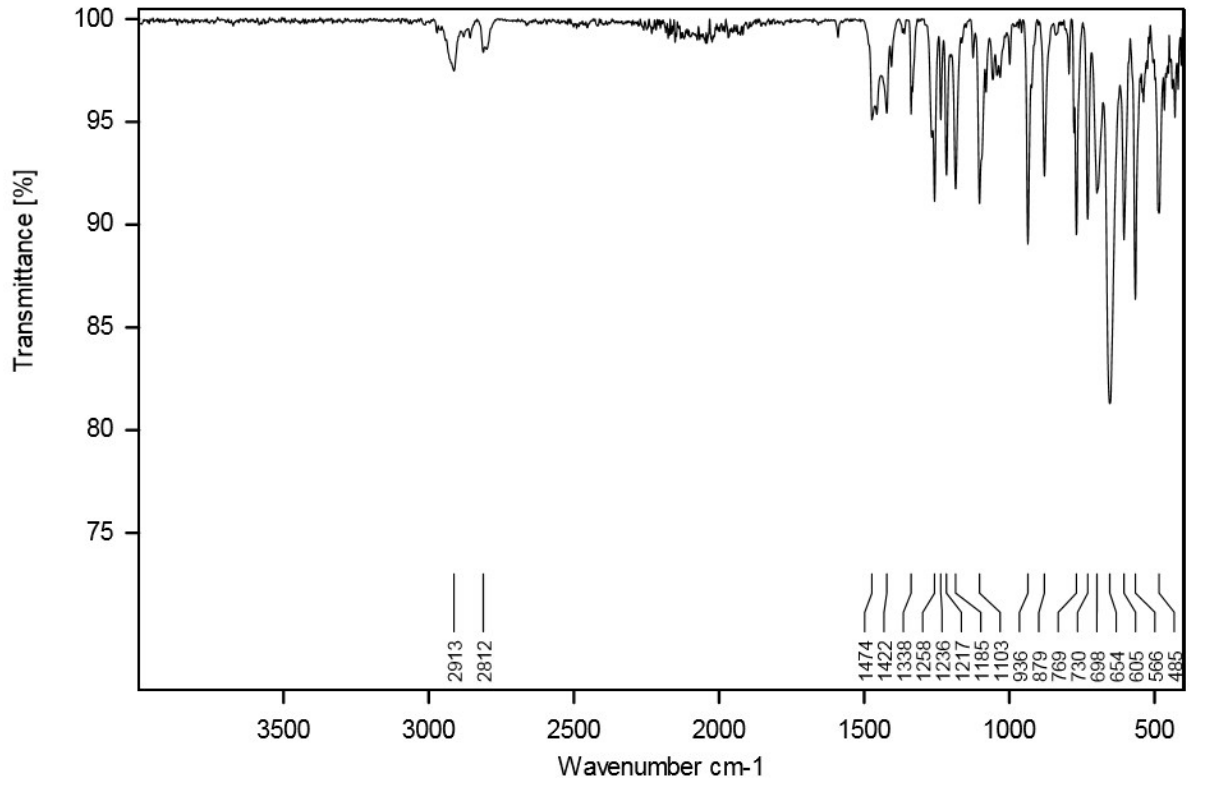

Figure S111 ATR-IR spectrum of 2dAlMe$_2$.

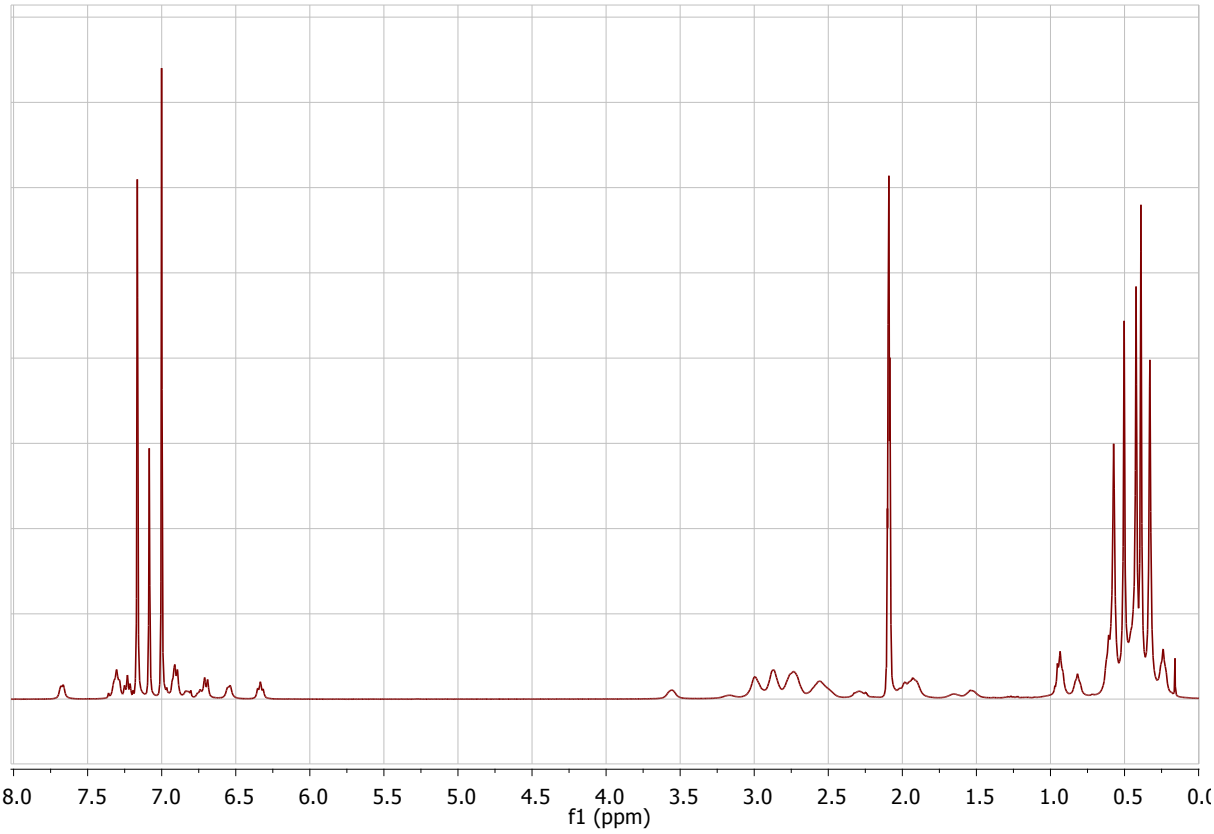

Figure S112 ^{1}H NMR spectrum (400 MHz) of 1mSn in toluene-d_8 at 193 K.

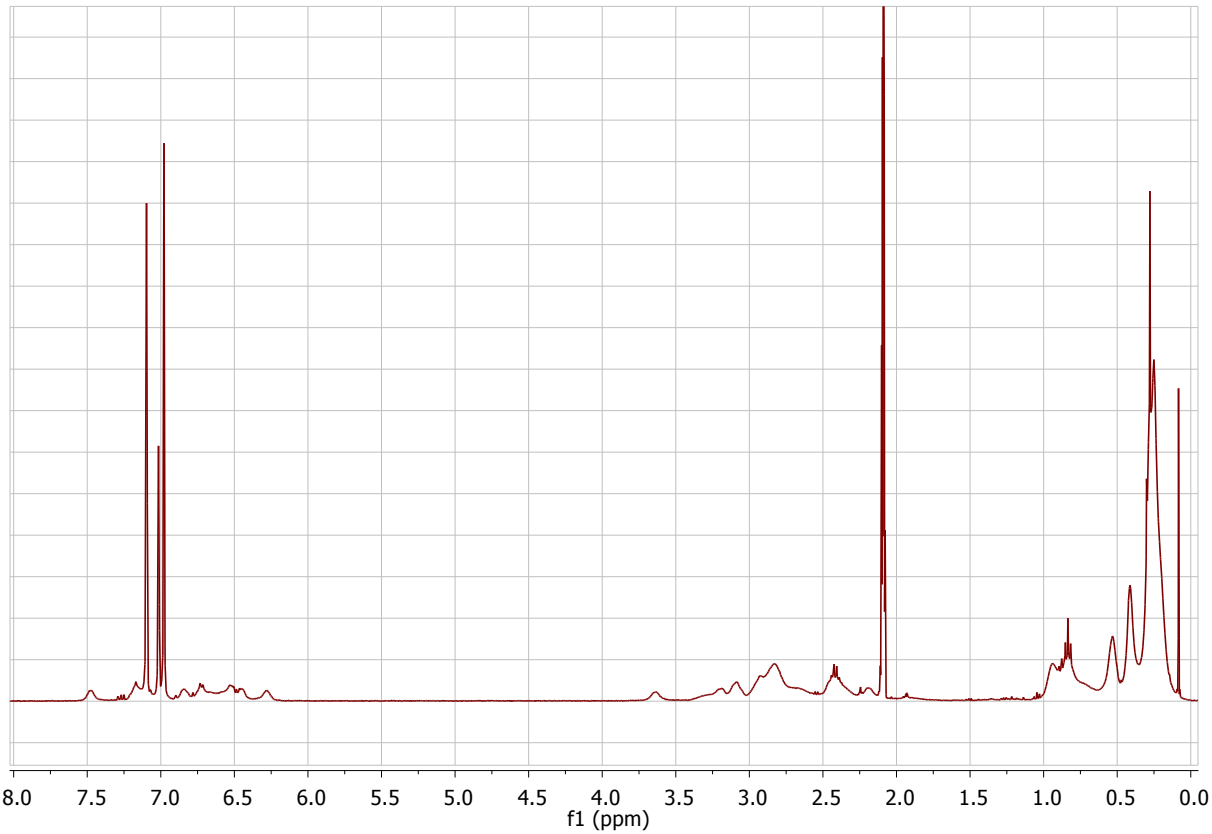

Figure S113 ^{1}H NMR spectrum (400 MHz) of 1mSn in toluene-d_8 at 300 K.

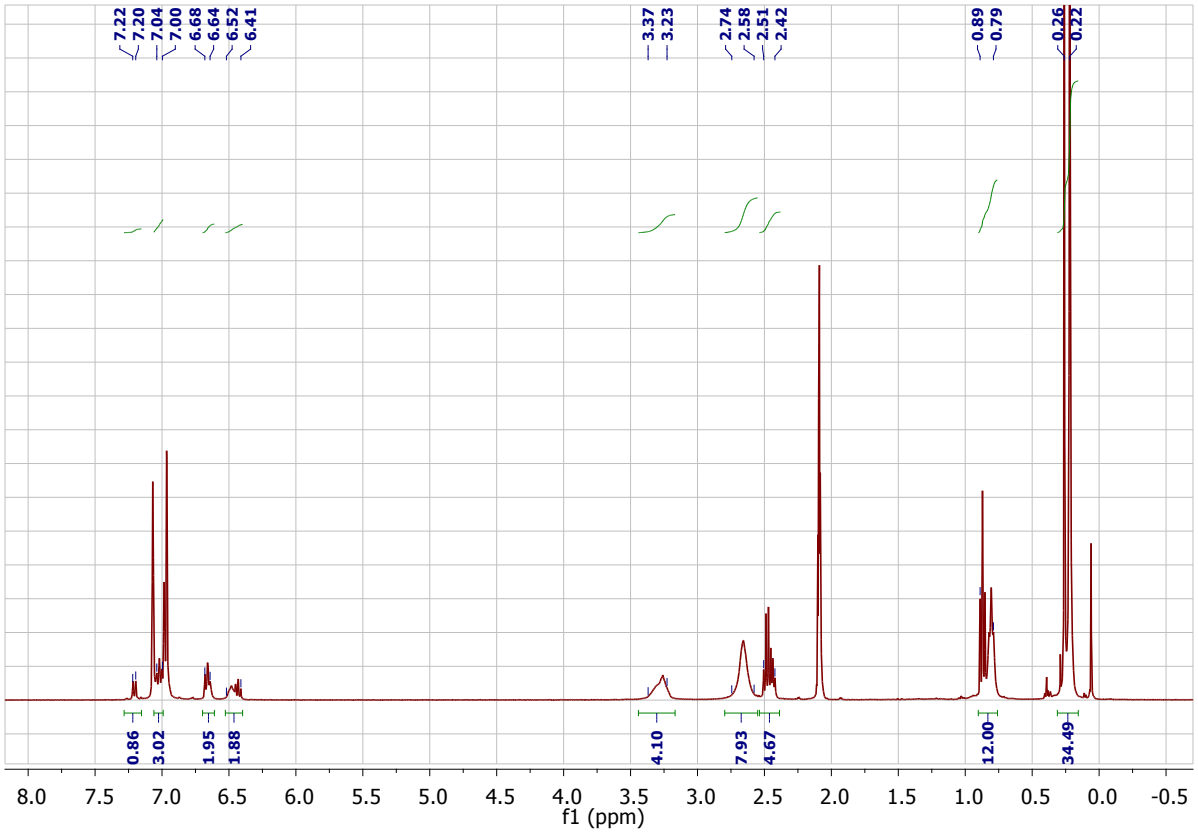

Figure S114 ^{1}H NMR spectrum (400 MHz) of 1mSn in toluene-d_8 at 353 K.

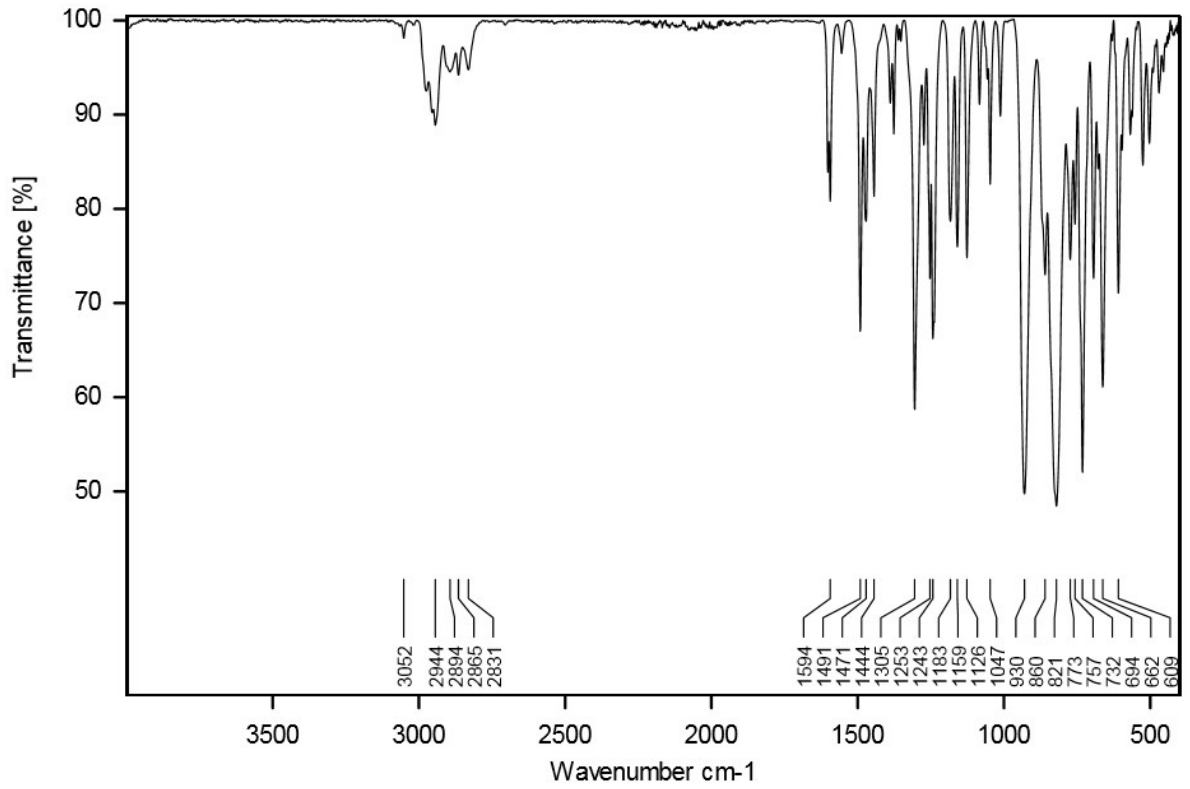

Figure S115 ATR-IR spectrum of 1mSn.

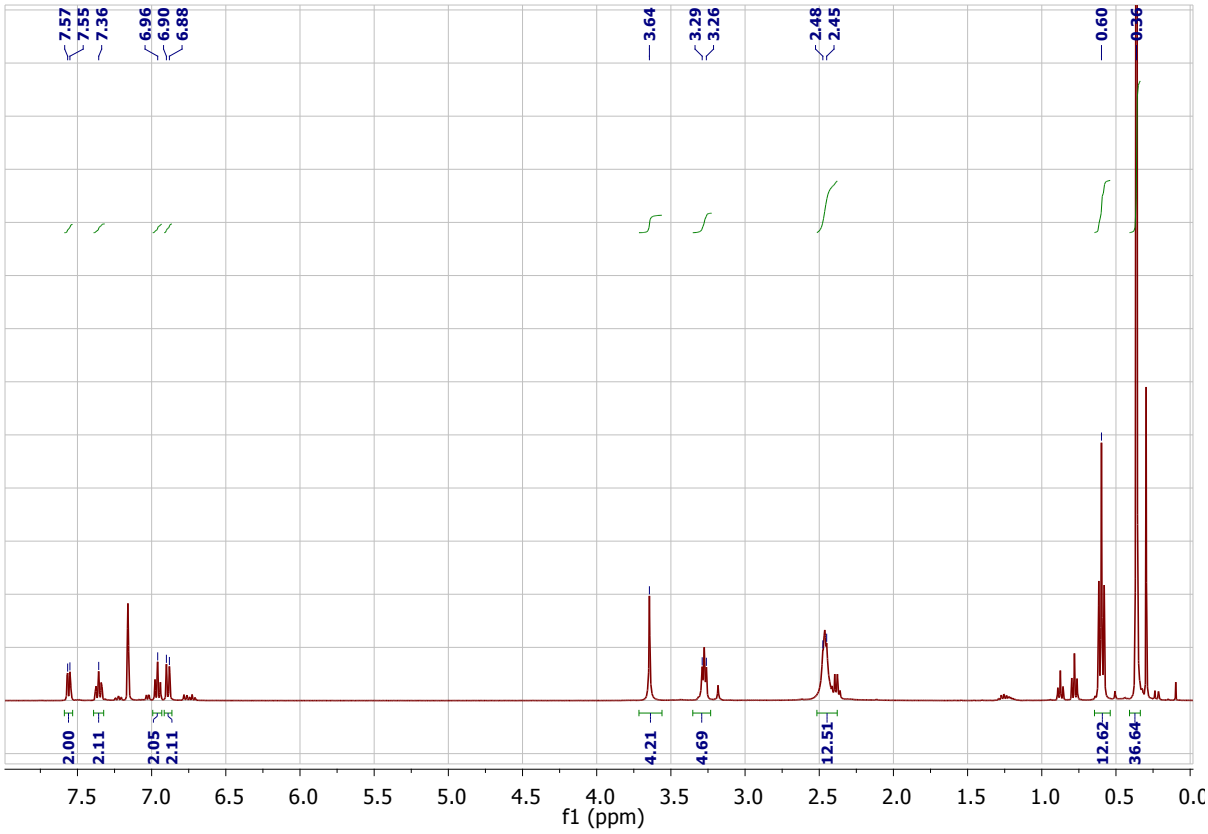

Figure S116 ^{1}H NMR spectrum (400 MHz) of 1nSn in C_6D_6 at 300 K.

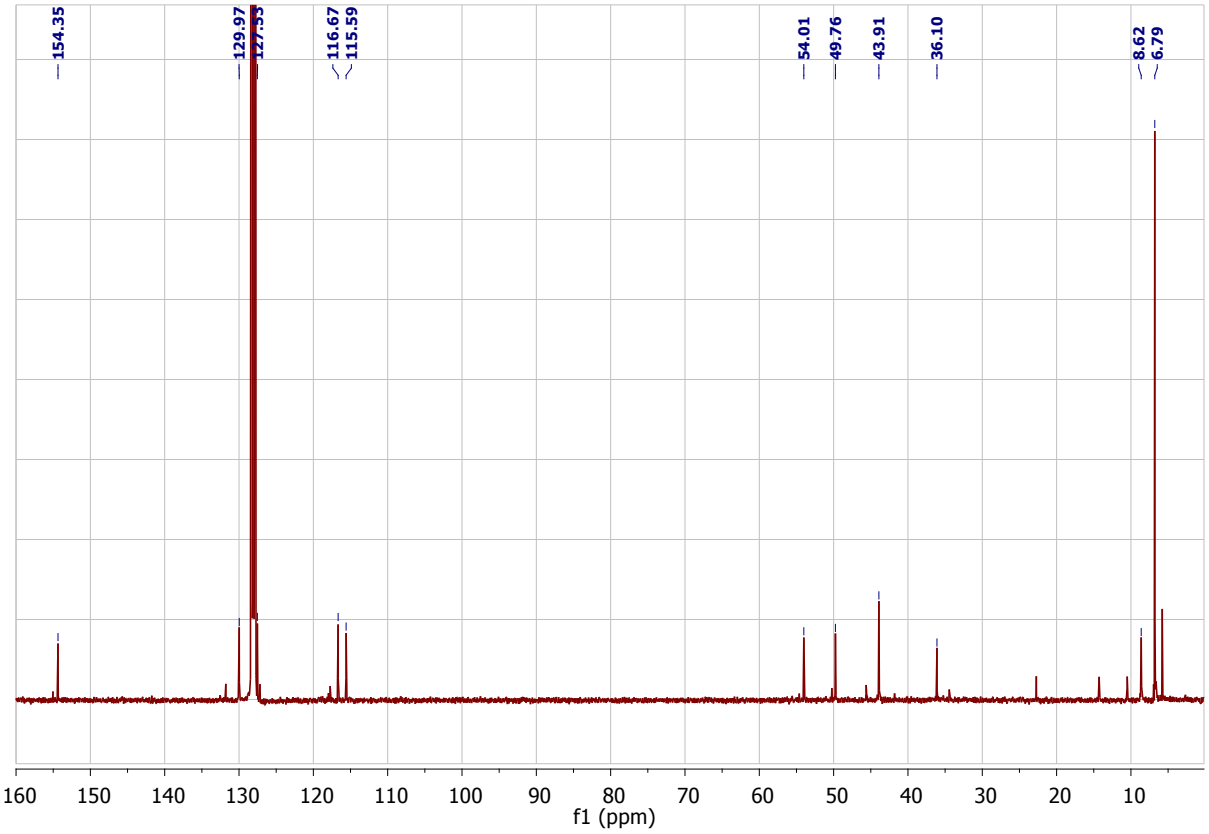

Figure S117 ^{13}C NMR spectrum (101 MHz) of 1nSn in C_6D_6 at 300 K.

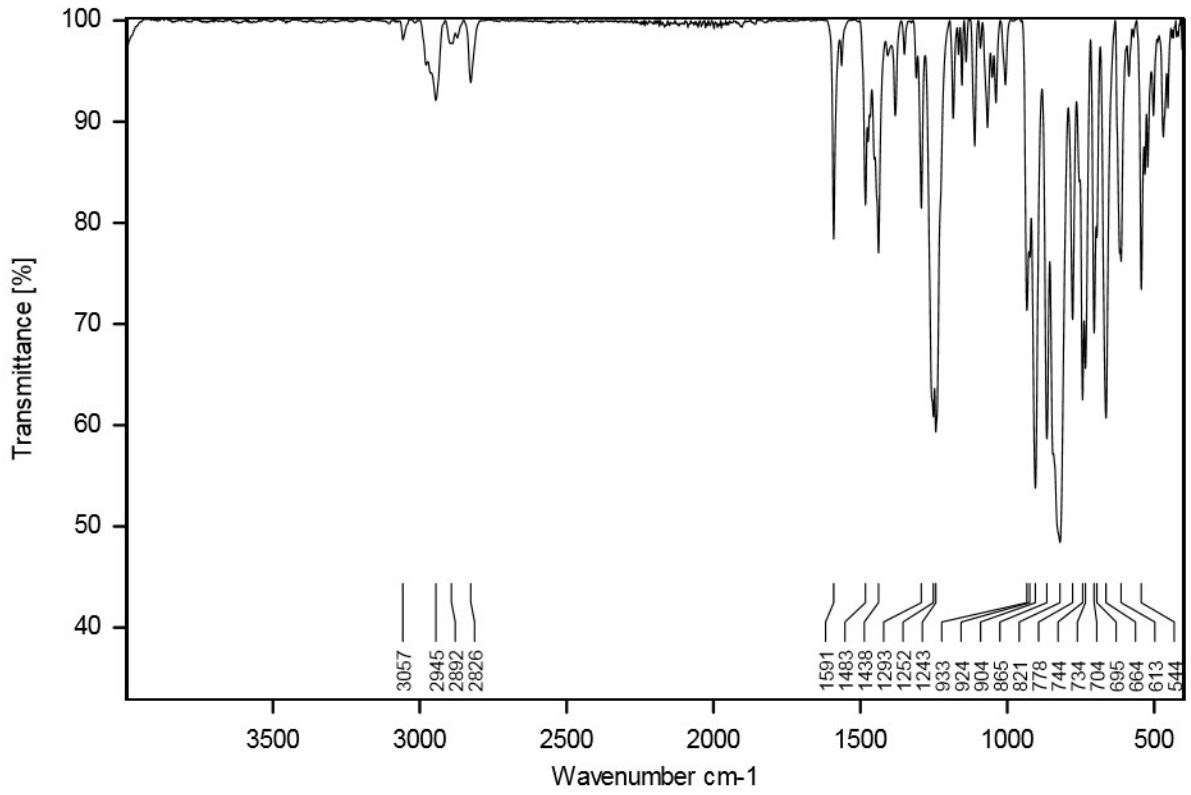

Figure S118 ATR-IR spectrum of 1nSn.

5. X-Ray data

Table S1: Crystal data and refinement details for the X-ray structure determinations.

Compound	**1jAlH2**	**1kAlH2**	**1mSnHMDS**	**1nMg**	**1nSnHMDS**
formula	$C_{18}H_{36}Al_2N_4$	$C_{22}H_{40}Al_2N_4O_{0.50}$	$C_{36}H_{72}N_6OSi_4Sn_2$	$C_{26}H_{40}MgN_4$	$C_{38}H_{76}N_6Si_4Sn_2$
fw ($g \cdot mol^{-1}$)	362.47	422.54	954.73	432.93	966.78
T/°C	-140(2)	-150(2)	-150(2)	-150(2)	-150(2)
crystal system	monoclinic	monoclinic	orthorhombic	orthorhombic	monoclinic
space group	P 2_1/c	P 2_1/c	P 2_1 2_1 2_1	P n a 2_1	P 2_1/n
a/ Å	13.2404(3)	12.0676(3)	13.8445(2)	18.2567(3)	13.02620(10)
b/ Å	14.7403(3)	13.4021(3)	17.9944(2)	11.3141(2)	16.2087(2)
c/ Å	12.6651(4)	15.1427(3)	18.8137(2)	11.7597(2)	33.3195(3)
α/°	90	90	90	90	90
β/°	117.329(4)	95.948(2)	90	90	96.394(1)
γ/°	90	90	90	90	90
V/Å^3	2195.92(12)	2435.86(10)	4686.93(10)	2429.06(7)	6991.24(12)
Z	4	4	4	4	6
ρ ($g \cdot cm^{-3}$)	1.096	1.152	1.353	1.184	1.378
μ (cm^{-1})	12.34	11.97	72.95	7.68	97.48
measured data	12578	14061	30878	15495	46347
data with I > 2σ(I)	3807	4327	9096	4244	12581
unique data (R_{int})	4365/0.0263	4836/0.0281	9256/0.0282	4291/0.0202	13491/0.0293
wR_2 (all data, on F^2)[a]	0.0976	0.1107	0.0431	0.0643	0.0559
R_1 ($I > 2\sigma(I)$) [a]	0.0336	0.0403	0.0178	0.0242	0.0224
S [b]	1.034	1.067	1.040	1.037	1.025
Res. dens./e·Å^{-3}	0.221/-0.186	0.243/-0.271	0.385/-0.291	0.175/-0.139	1.151/-0.596
Flack-parameter	-	-	0.004(2)	0.009(15)	-
absorpt method	gaussian	gaussian	multi-scan	multi-scan	multi-scan
absorpt corr $T_{min}/_{max}$	0.951/0.977	0.965/0.975	0.575/1.000	0.955/1.000	0.378/1.000
CCDC No.	2026438	2026439	2026440	2026441	2026442

cont. Table S1: Crystal data and refinement details for the X-ray structure determinations.

Compound	**1nZn**	**2a**	**2aAlMe2**	**2aZnEt**	**2b**
formula	$C_{26}H_{40}N_4Zn$	$C_{24}H_{36}N_4$	$C_{28}H_{46}Al_2N_4$	$C_{28}H_{44}N_4Zn_2$	$C_{24}H_{38}N_4$
fw ($g \cdot mol^{-1}$)	473.99	380.57	492.65	567.41	382.58
°C	-150(2)	-150(2)	-150(2)	-150(2)	-150(2)
crystal system	monoclinic	monoclinic	monoclinic	triclinic	monoclinic
space group	P $2_1/c$	P $2_1/c$	P $2_1/n$	P $\bar{1}$	P $2_1/c$
a/ Å	17.1511(1)	8.5472(2)	12.2153(3)	9.2946(3)	8.05464(11)
b/ Å	21.9866(2)	18.5720(4)	8.4890(3)	10.3097(3)	18.54236(19)
c/ Å	12.9305(1)	14.0121(3)	13.6679(5)	15.5461(6)	8.27176(10)
α/°	90	90	90	104.387(3)	90
β/°	91.116(1)	98.247(2)	92.201(2)	90.487(3)	114.3273(16)
γ/°	90	90	90	109.209(3)	90
V/Å^3	4875.09(6)	2201.26(9)	1416.26(8)	1355.99(8)	1125.71(3)
Z	8	4	2	2	2
ρ ($g \cdot cm^{-3}$)	1.292	1.148	1.155	1.390	1.129
μ (cm^{-1})	11.17	5.22	10.84	23.33	5.11
measured data	32011	12964	8174	17904	34806
data with I > 2σ(I)	8895	3268	2443	4628	2115
unique data (R_{int})	9646/0.0276	3870/0.0381	2807/0.0331	5320/0.0322	2280/0.0381
wR_2 (all data, on F^2)[a)]	0.0778	0.1050	0.0962	0.0706	0.0918
R_1 ($I > 2\sigma(I)$) [a)]	0.0299	0.0402	0.0364	0.0277	0.0339
S [b)]	1.027	1.040	1.025	1.044	1.031
Res. dens./e·Å^{-3}	0.315/-0.390	0.204/-0.188	0.284/-0.184	0.342/-0.447	0.220/-0.182
absorpt method	multi-scan	Gaussian	multi-scan	multi-scan	gaussian
absorpt corr $T_{min}/_{max}$	0.648/1.000	0.878/0.956	0.682/1.000	0.767/1.000	0.866/0.975
CCDC No.	2026443	2026444	2026430	2026431	2026432

cont. Table S1: Crystal data and refinement details for the X-ray structure determinations.

Compound	**2bAlH**	**2bAlMe2**	**2bMg**	**2bZn**	**2dAlMe2**
formula	$C_{24}H_{37}AlN_4$	$C_{28}H_{48}Al_2N_4$	$C_{24}H_{36}MgN_4$	$C_{24}H_{36}N_4Zn$	$C_{29}H_{50}Al_2N_4$
fw ($g \cdot mol^{-1}$)	408.55	494.66	404.88	445.94	508.69
°C	-150(2)	-150(2)	-150(2)	-150(2)	-150(2)
crystal system	monoclinic	monoclinic	monoclinic	monoclinic	monoclinic
space group	P 2_1/n	P 2_1/c	P 2_1/c	P 2_1/n	P 2_1/c
a/ Å	9.7448(2)	9.4698(3)	8.7239(1)	8.66060(10)	31.9265(7)
b/ Å	13.2241(2)	12.9516(5)	15.0640(2)	15.1890(2)	7.4319(2)
c/ Å	18.4085(3)	12.8812(4)	17.5875(3)	17.3370(2)	13.1546(3)
α/°	90	90	90	90	90
β/°	103.626(2)	107.433(4)	104.291(2)	102.0200(10)	98.015(2)
γ/°	90	90	90	90	90
V/Å^3	2305.46(7)	1507.30(9)	2239.77(6)	2230.61(5)	3090.76(13)
Z	4	2	4	4	4
ρ ($g \cdot cm^{-3}$)	1.177	1.090	1.201	1.328	1.093
μ (cm^{-1})	8.84	10.19	8.01	11.97	10.06
measured data	20227	7451	14829	14690	11163
data with I > 2σ(I)	3971	2496	4054	4082	4823
unique data (R_{int})	4615/0.0252	2659/0.0262	4369/0.0205	4364/0.0180	5946/0.0259
wR_2 (all data, on F^2)[a)]	0.0956	0.1144	0.0875	0.0712	0.1294
R_1 ($I > 2\sigma(I)$) [a)]	0.0368	0.0460	0.0317	0.0253	0.0448
S [b)]	1.045	1.146	1.027	1.049	1.038
Res. dens./e·Å^{-3}	0.236/-0.238	0.226/-0.242	0.227/-0.186	0.310/-0.263	0.394/-0.241
absorpt method	gaussian	analytical	multi-scan	multi-scan	gaussian
absorpt corr $T_{min}/_{max}$	0.932/0.970	0.840/0.922	0.937/1.000	0.866/1.000	0.963/0.992
CCDC No.	2026433	2026434	2026435	2026436	2026437

[a)] Definition of the *R* indices: $R_1 = (\Sigma \,||F_o| - |F_c||)/\Sigma |F_o|$;

$wR_2 = \{\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$ with $w^{-1} = \sigma^2(F_o^2) + (aP)^2+bP$; $P = [2F_c^2 + Max(F_O^2]/3$;

[b)] $s = \{\Sigma[w(F_o^2-F_c^2)^2]/(N_o-N_p)\}^{1/2}$.

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