

β -Amino Acid Derived Gemini Surfactants from Diformylfuran (DFF) with Particularly Low Critical Micelle Concentration (CMC)

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Supplementary information

General considerations

2,5-diformylfuran was synthesised according to an already reported procedure.¹ All reagents were provided by Fischer Scientifique or accros chemicals and used without purification. Solvent were used as received. Raney Nickel (activated, 50% in water) was stored for 1 year and not reactivated before use.

All intermediary compounds were characterised by ¹H and ¹³C NMR without purification. Final products were dried for 72 h at 40 °C under vacuum (0.1 mbar) before characterisation (¹H, ¹³C, IR, HRMS).

¹H and ¹³C NMR analysis were performed using a 250 MHz Bruker spectrometer. NOE spectra were performed using a 500 MHz Advance Bruker apparatus. Surface tension measurements were determined by AgroIndustrie recherches et Développements.

Synthesis of imines

The amine (2 equivalents, 16 mmol) is added to a stirred solution of DFF (1 g, 8 mmol) in ethanol (10 mL). The mixture is stirred for 4 h and diluted with water. The product is extracted with ethyl acetate (3*50 mL). The organic fractions are dried with MgSO₄, filtered and concentrated under vacuum.

Synthesis of amines

The crude imines are dissolved in ethanol (10 mL/g) and NaBH₄ (610 mg, 16 mmol) is added at 0 °C (ice bath). After the addition of NaBH₄, the ice bath is removed and the mixture is stirred 4h. Hydride excess is quenched by water addition and the product is extracted with ethyl acetate (3*50 mL). The organic fraction are dried with MgSO₄, filtered and concentrated under vacuum.

Reduction of furanic ring

The crude amines are dissolved in ethanol (10 mL/g) and Raney Nickel is added (1.7 mL of a 50% water solution, washed with 3*5 mL of ethanol). The Parr reactor is sealed and pressurised with

¹ Q. Girka, B. Estrine, N. Hoffmann J. Le Bras, S. Marinković, J. Muzart, *React. Chem. Eng.*, **2016**, 1, 176-182.

hydrogen (15 bars, 50 °C). The mixture is stirred overnight. After completion, the mixture is cooled to room temperature and hydrogen is carefully evacuated. Raney Nickel is filtered off and the product is obtained by vacuum evaporation of ethanol.

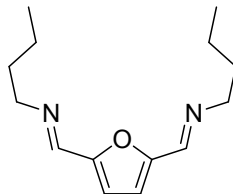
Synthesis of benzyl esters

The crude tetrahydrofuranic compounds are dissolved in ethanol (10 mL/g). The solution is cooled down to 5 °C and benzylacrylate is added (2.2 equivalents). The solution is stirred for 24 h and then concentrated under vacuum. The esters are purified using silica gel chromatographic column with petroleum ether/ethyl acetate (95/5 to 75/25).

Deprotection of benzyl esters

The pure benzyl esters are dissolved in ethanol (10 mL/g) in the presence of Pd/C (5 mol%). The mixture is stirred 12 h under hydrogen (balloon) and the catalyst is filtered off. Vacuum elimination of the solvent yield the final product. Before the determination of the activity of these compounds, they are purified using silica gel chromatography with petroleum ether/methanol (80/20 to 100% methanol) as eluent.

N,N'-(furan-2,5-diylbis(methylene))bis(butan-1-imine) (Ia) :

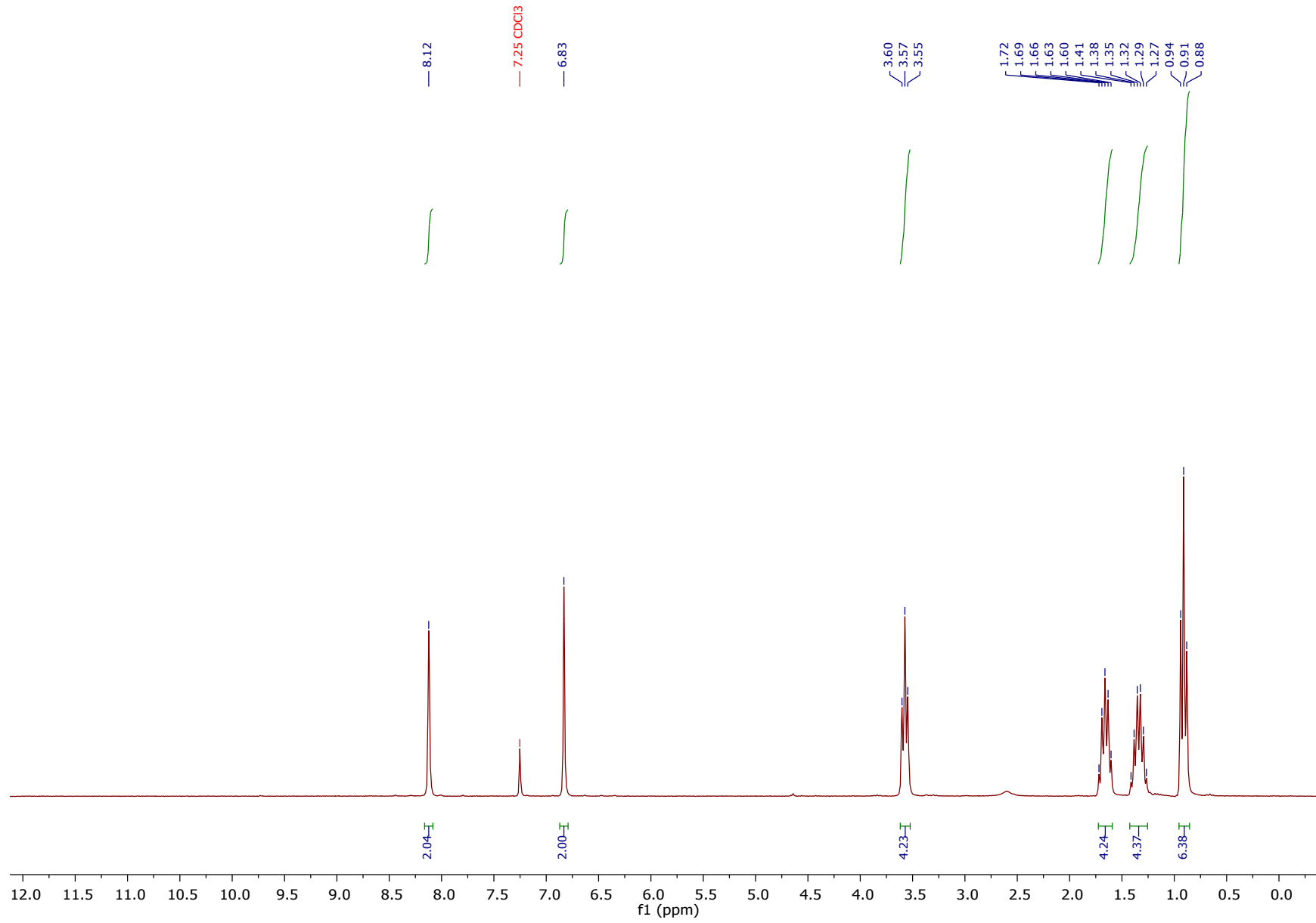


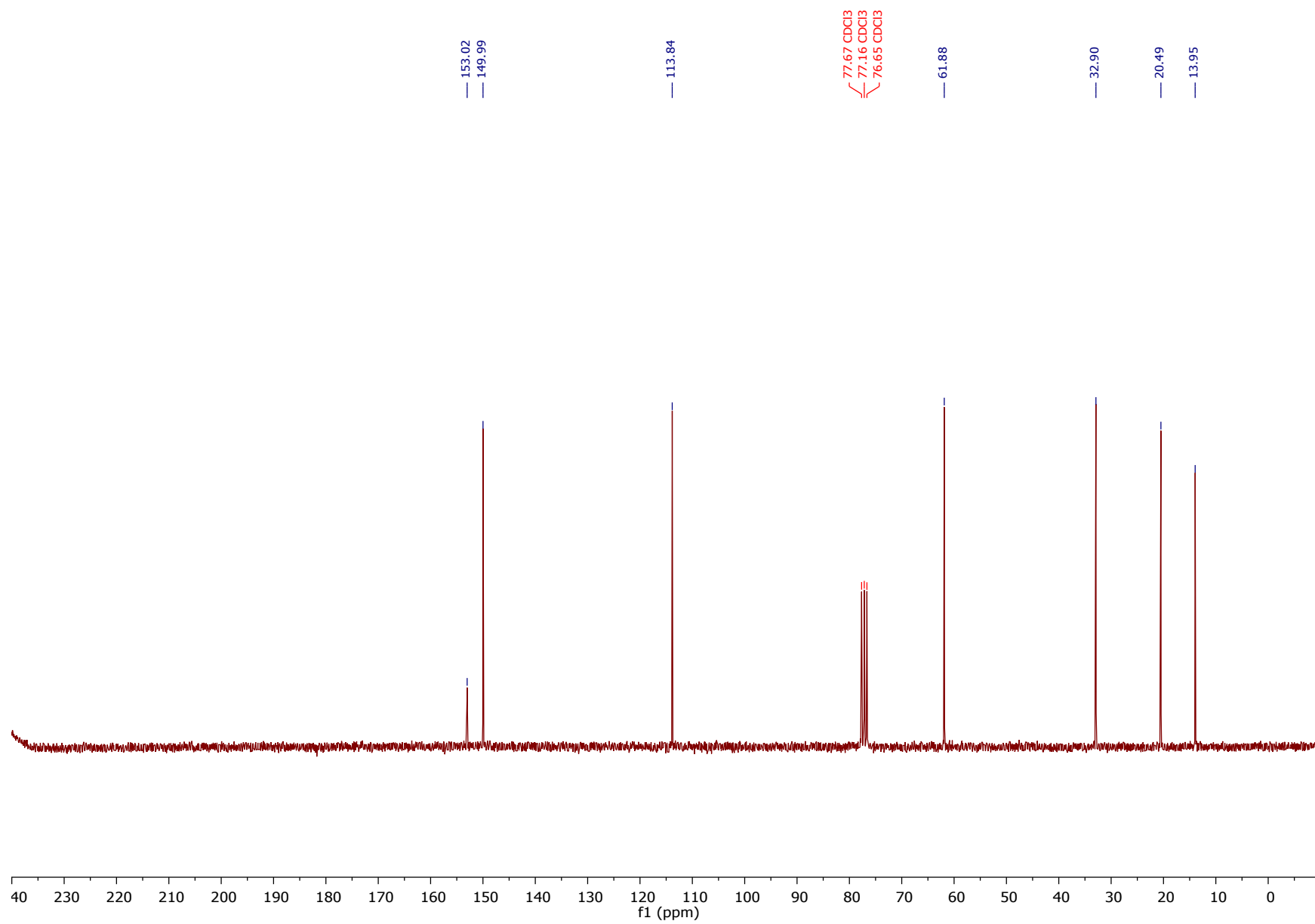
Compound **Ia** is synthesised using *n*-butylamine.

Yield: 98%, yellow viscous oil.

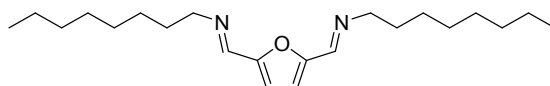
NMR ¹H (250 MHz, CDCl₃) δ: . 8.12 (s, 2H), 6.83 (s, 2H), 3.57 (t, *J* = 7 Hz, 4H), 1.66 (p, *J* = 7.0 Hz, 4H), 1.34 (h, *J* = 7 Hz, 4H), 0.91 (t, *J* = 7 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 153.0, 150.0, 113.9, 77.7, 77.2, 76.7, 61.9, 32.9, 20.5, 14.0.





N,N'-(furan-2,5-diylbis(methylene))bis(octan-1-imine) (Ib) :

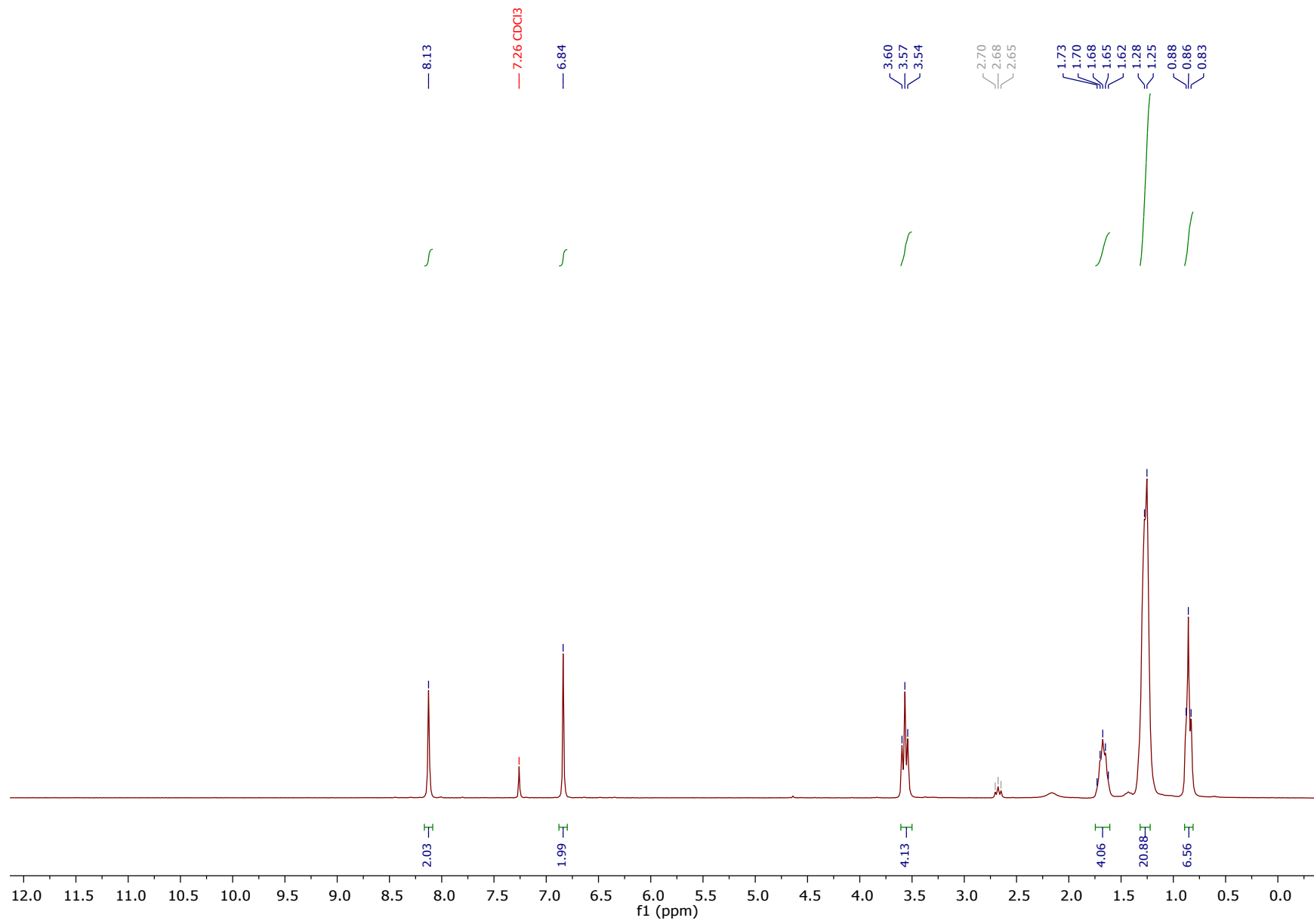


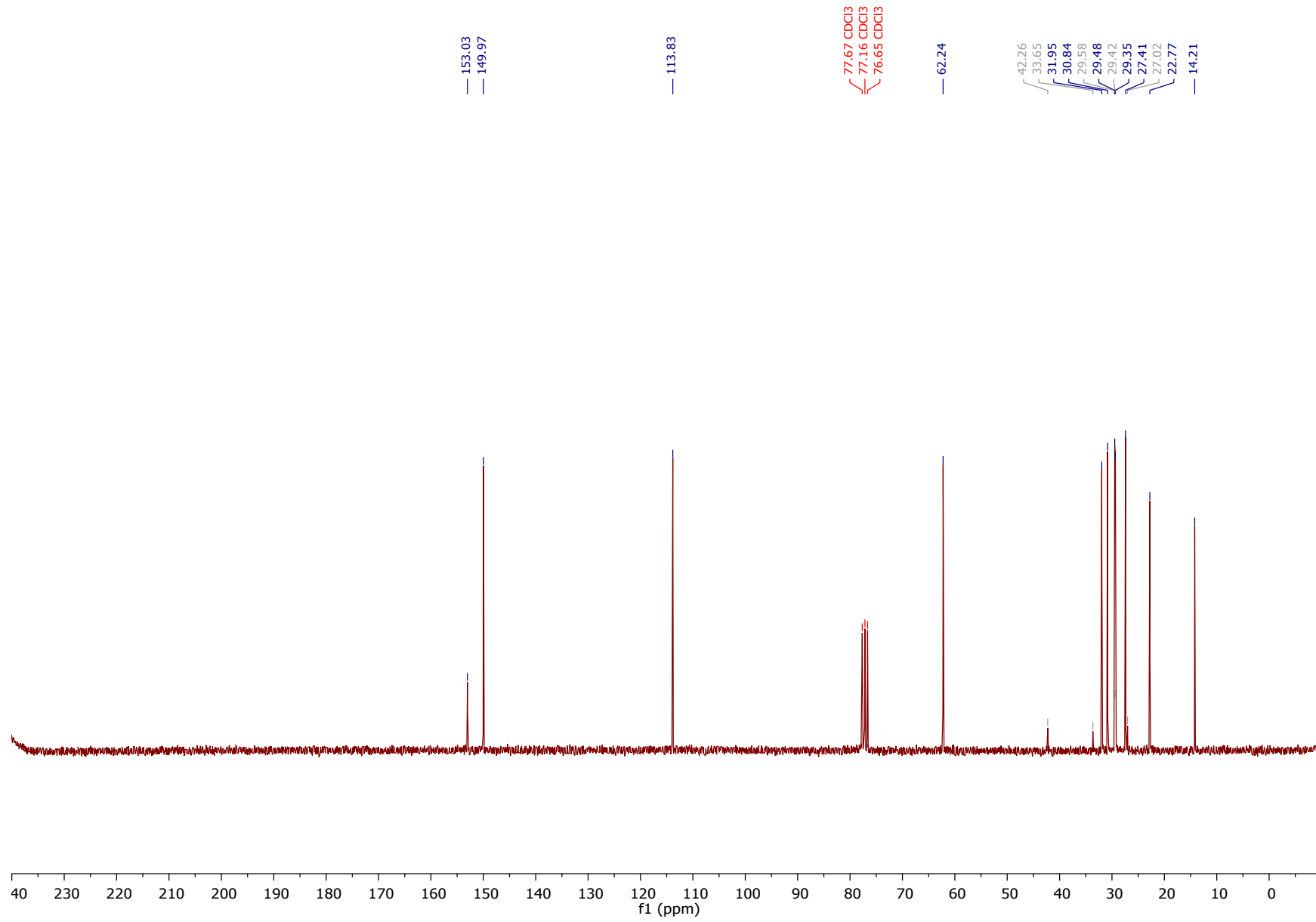
Compound **Ib** is synthesised using octylamine.

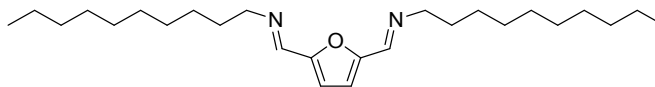
Yield: 96 %, yellow viscous oil.

NMR ¹H (250 MHz, CDCl₃) δ: 8.13 (s, 2H), 6.84 (s, 2H), 3.57 (t, *J* = 7 Hz, 4H), 1.66 (m, 4H), 1.53 – 1.02 (m, 20H), 0.85 (t, *J* = 6 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 153.0, 150.0, 113.8, 77.7, 77.2, 76.7, 62.2, 42.3, 33.7, 32.0, 30.8, 29.6, 29.5, 29.4, 29.4, 27.4, 27.0, 22.8, 14.2.





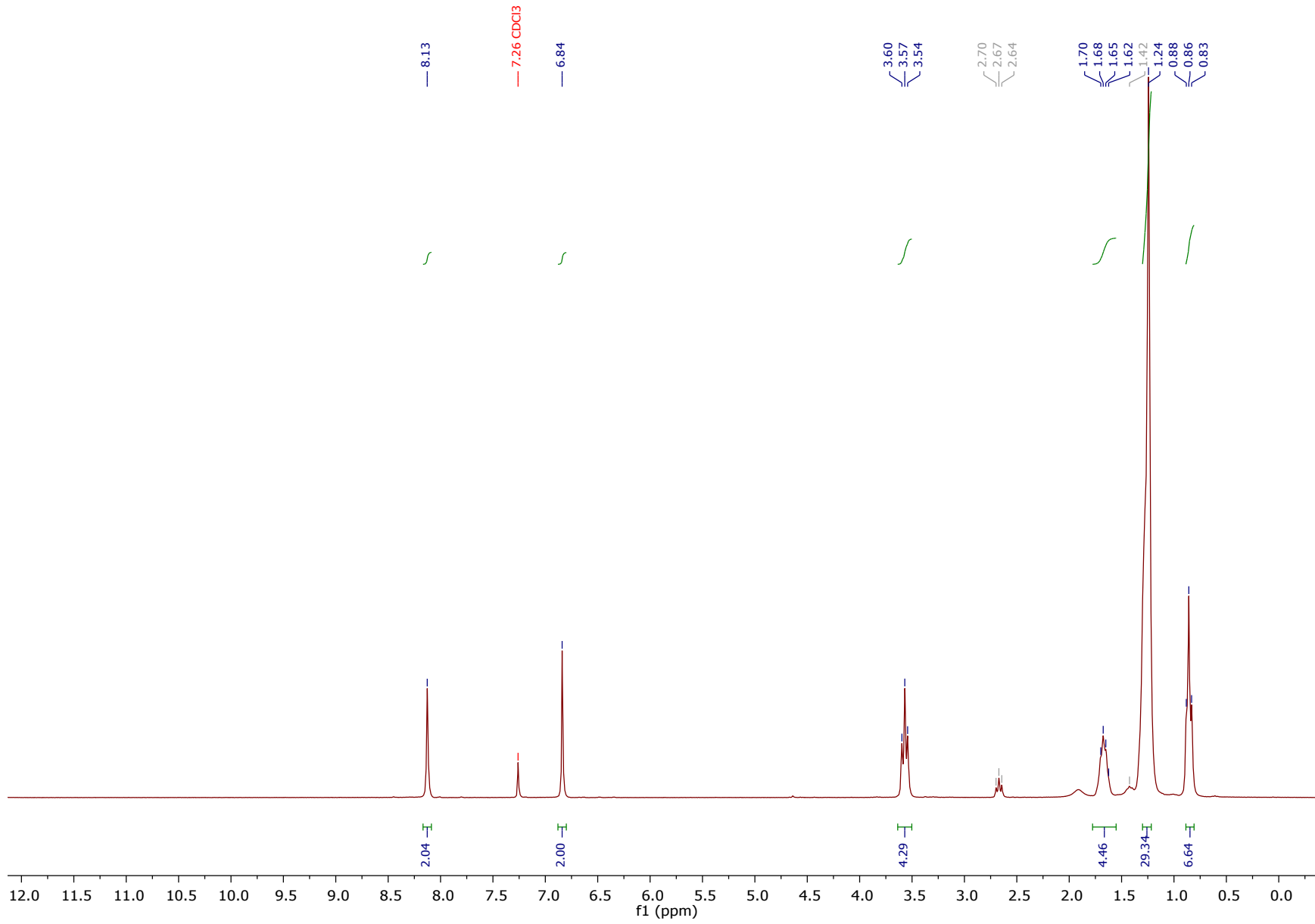
N,N'-(furan-2,5-diylbis(methylene))bis(decane-1-imine) (Ic) :

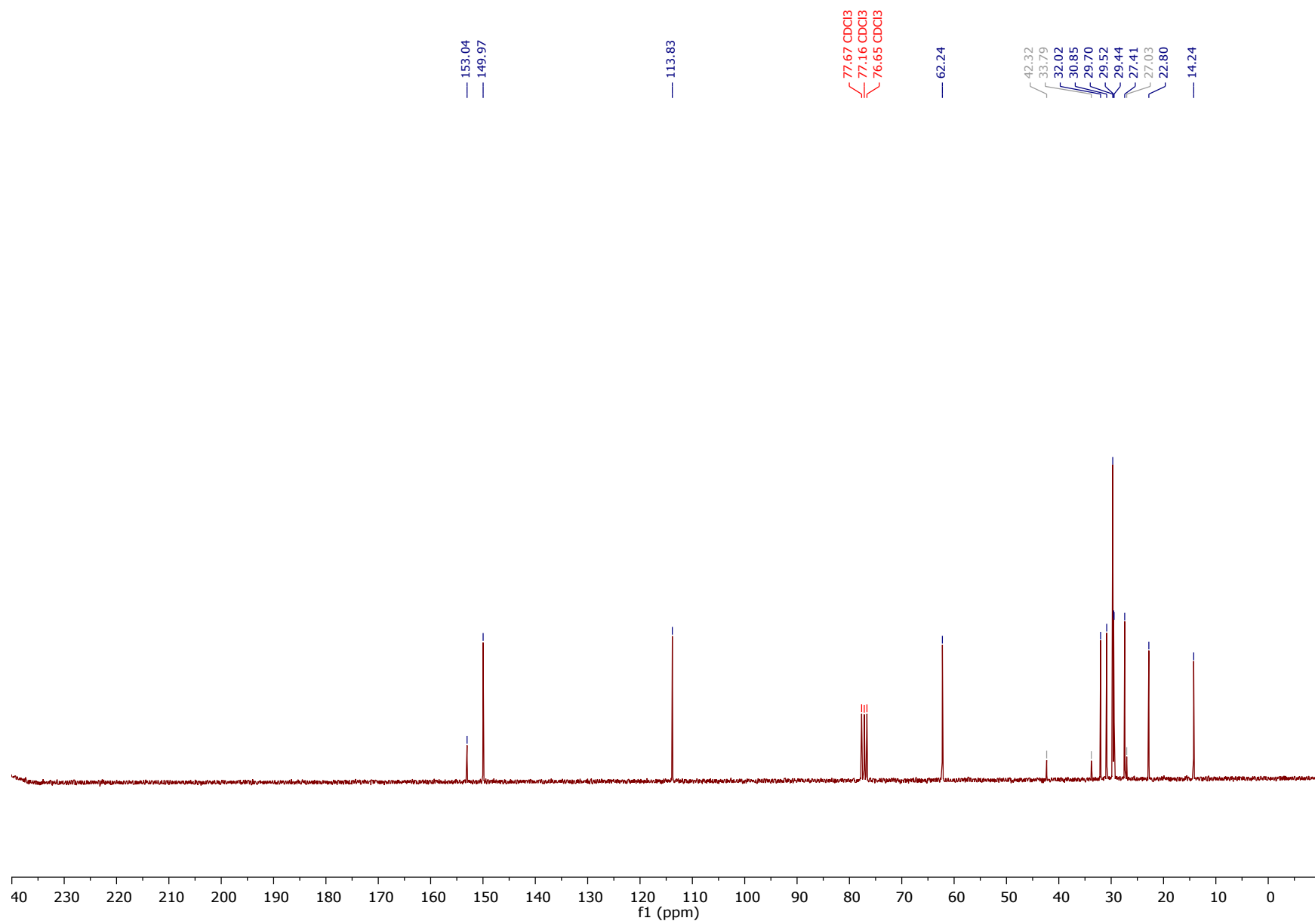
Compound **Ic** is synthesised using decylamine.

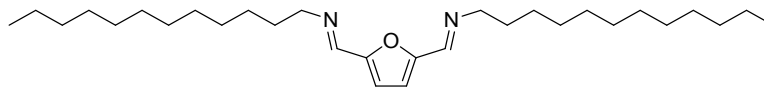
Yield: 99%, white solid.

NMR ¹H (250 MHz, CDCl₃) δ: 8.13 (s, 2H), 6.84 (s, 2H), 3.57 (t, *J* = 7 Hz, 4H), 1.68 (m, 4H), 1.24 (s, 28H), 0.86 (t, *J* = 6 Hz, 6H)

NMR ¹³C (63 MHz, CDCl₃) δ: 153.0, 150.0, 113.8, 77.7, 77.2, 76.7, 62.2, 42.3, 33.8, 32.0, 30.9, 29.7, 29.5, 29.4, 27.4, 27.0, 22.8, 14.2.





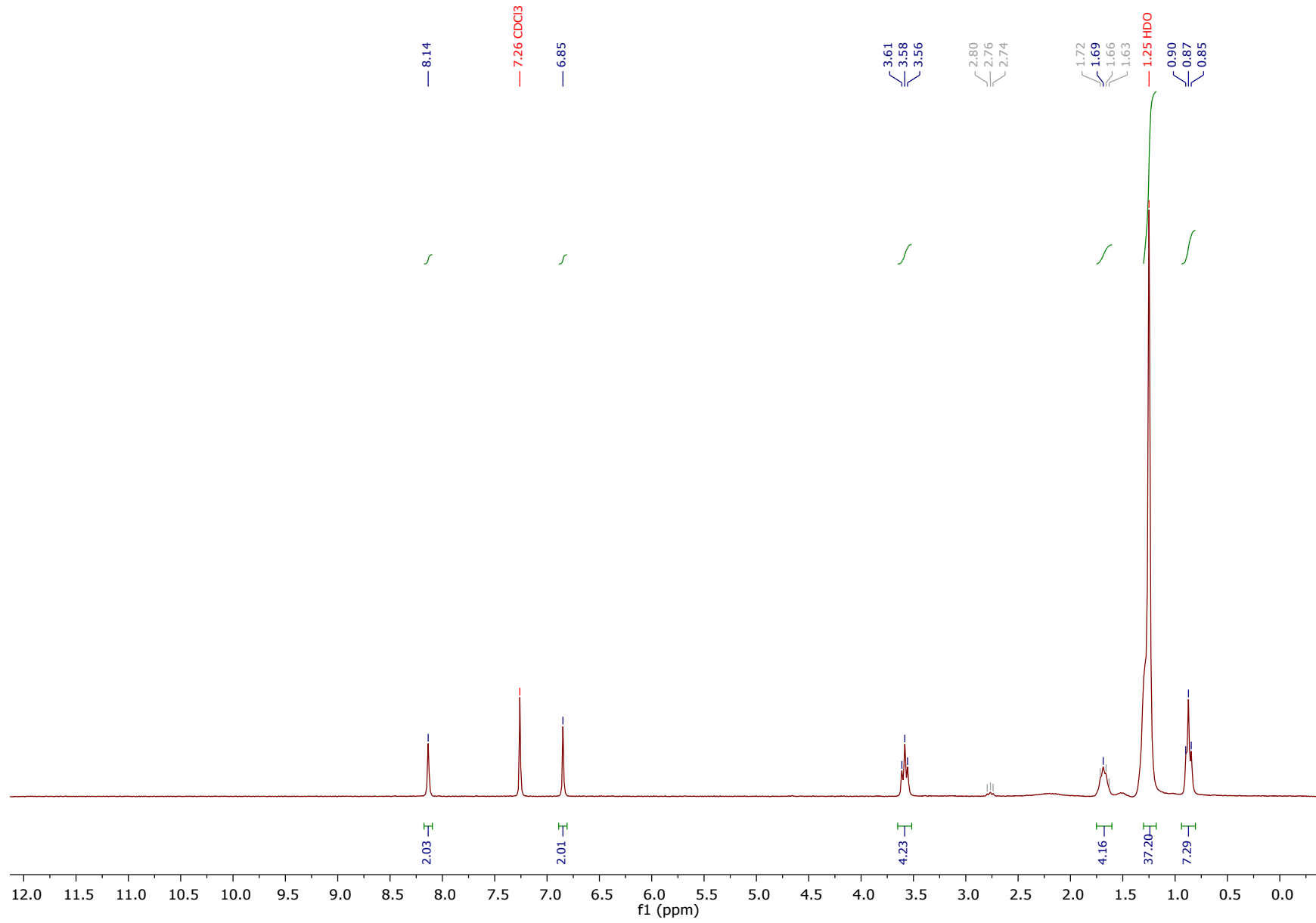
N,N'-(furan-2,5-diylbis(methylene))bis(dodecan-1-imine) (Id) :

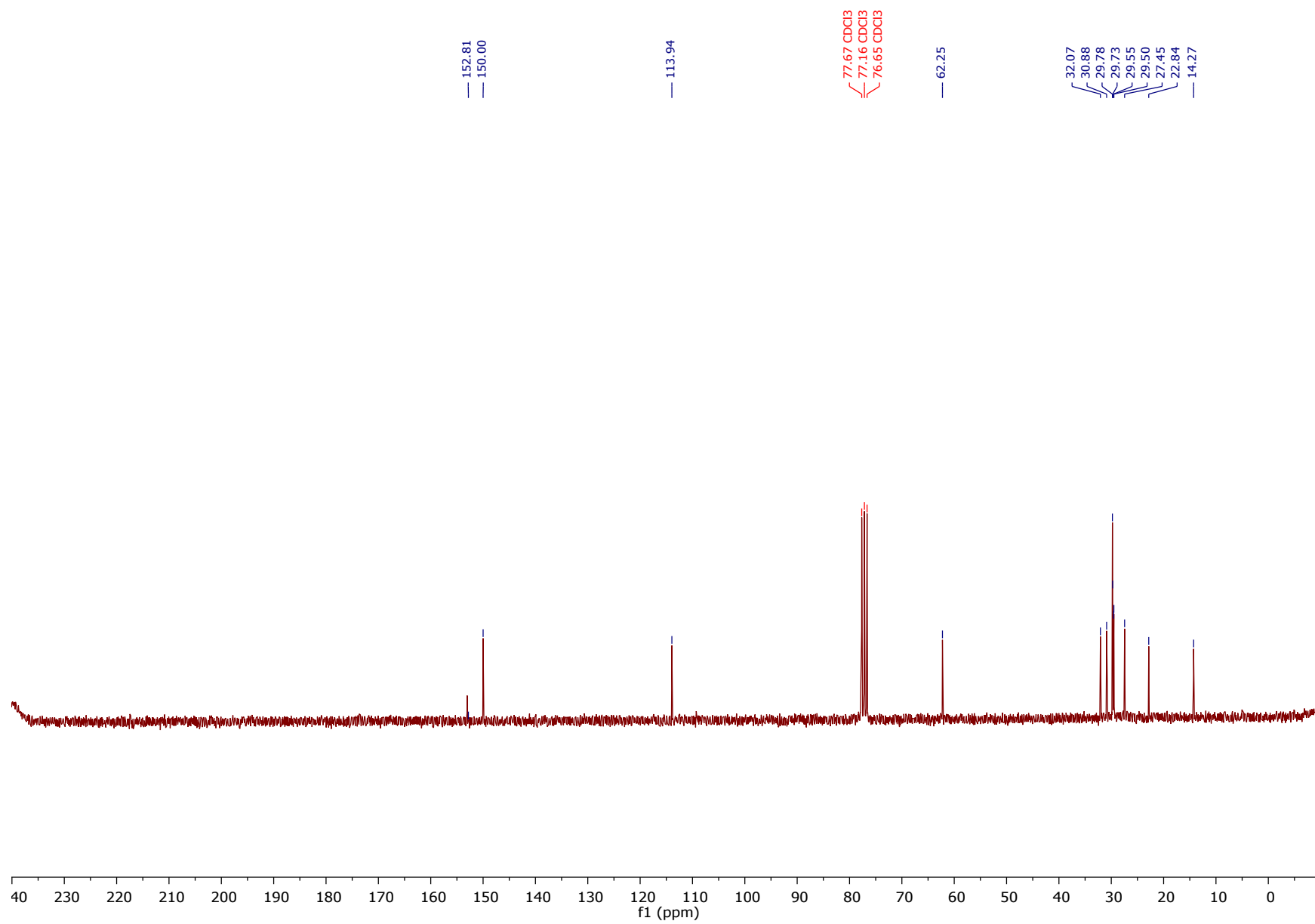
The compound **Id** is synthesised using dodecylamine.

Yield: 98%, white solid.

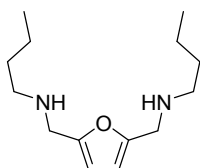
NMR ¹H (250 MHz, CDCl₃) δ: 8.14 (s, 2H), 6.85 (s, 2H), 3.58 (t, *J* = 7 Hz, 4H), 1.75 – 1.60 (m, 4H), 1.25 (s, 36H), 0.87 (t, *J* = 7 Hz, 6H)

NMR ¹³C (63 MHz, CDCl₃) δ: 152.8, 150.0, 113.9, 77.7, 77.2, 76.7, 62.3, 32.1, 30.9, 29.8, 29.7, 29.6, 29.5, 27.5, 22.8, 14.3.





N,N'-(furan-2,5-diylbis(methylene))bis(butan-1-amine) (IIa) :

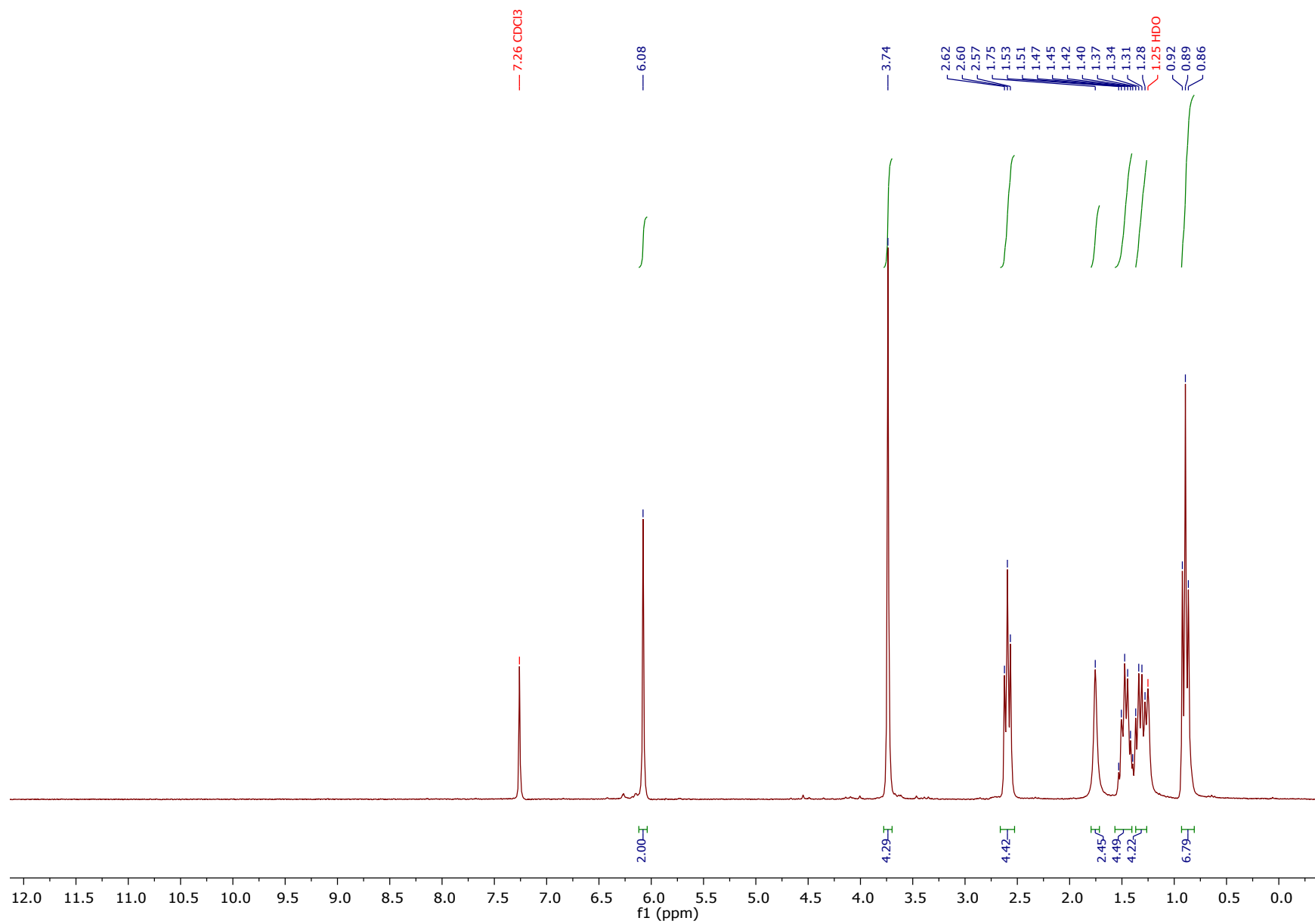


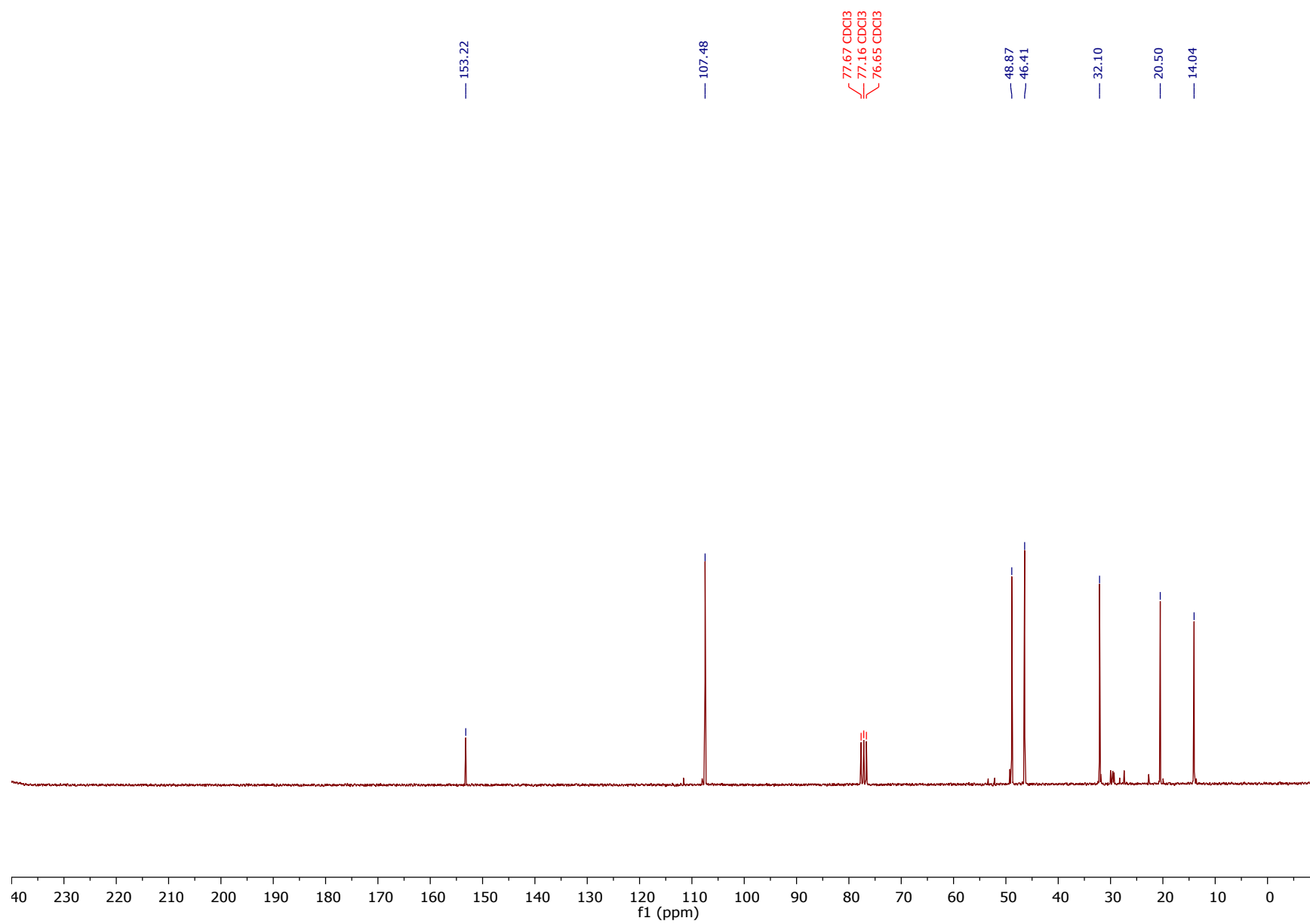
Compound **IIa** is synthesised using *n*-butylamine.

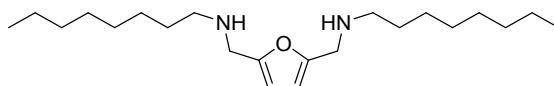
Yield: 98%, yellow viscous oil.

NMR ¹H (250 MHz, CDCl₃) δ: 6.08 (s, 2H), 3.74 (s, 4H), 2.59 (t, *J* = 7.0 Hz, 4H), 1.75 (br s, 2H), 1.57 – 1.36 (m, 4H), 1.36-1,22 (m, 4H), 0.89 (t, *J* = 7.0 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 153.1, 107.4, 48.8, 46.3, 32.0, 20.4, 13.9.





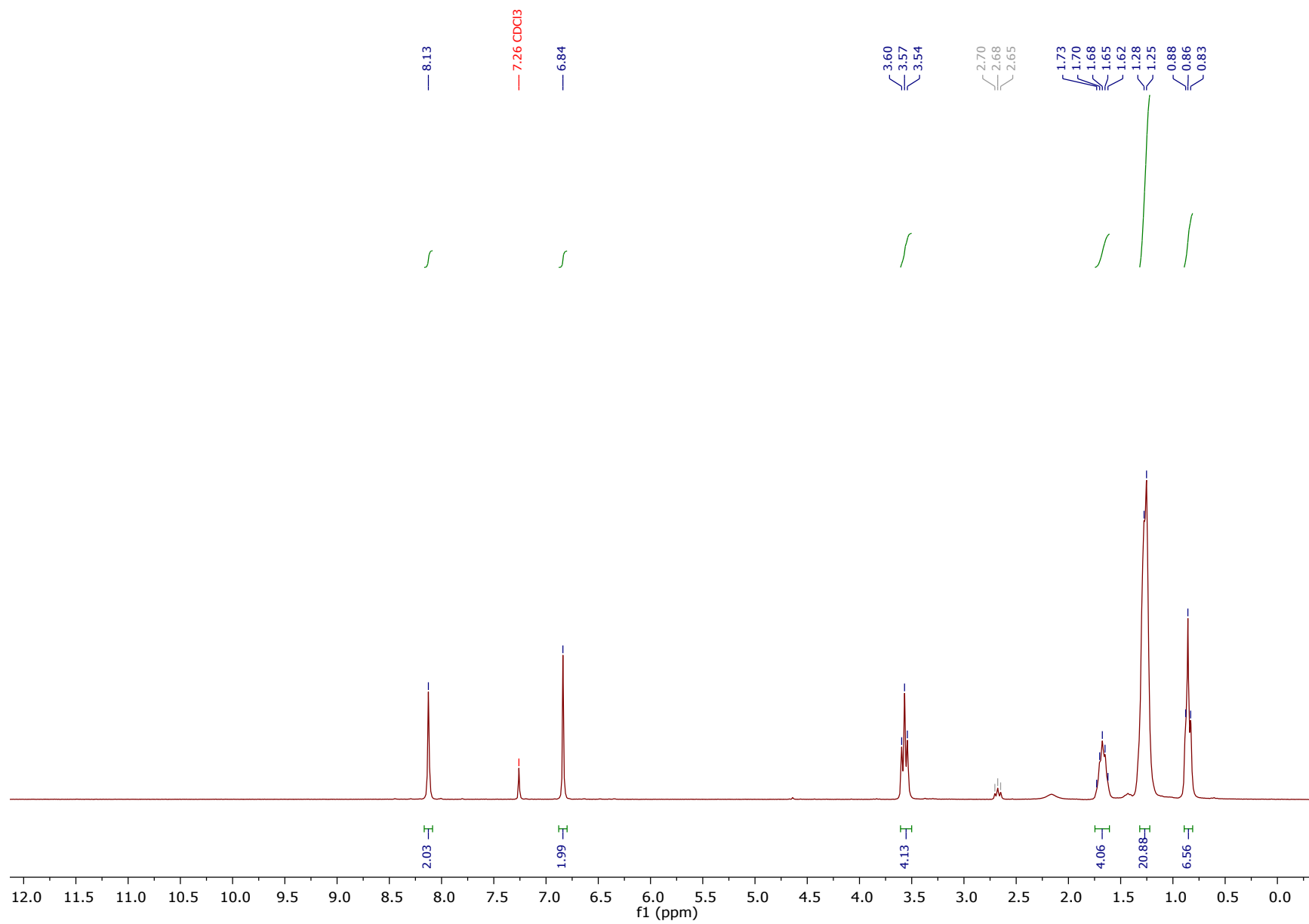
N,N'-(furan-2,5-diylbis(methylene))bis(octan-1-amine) (IIb) :

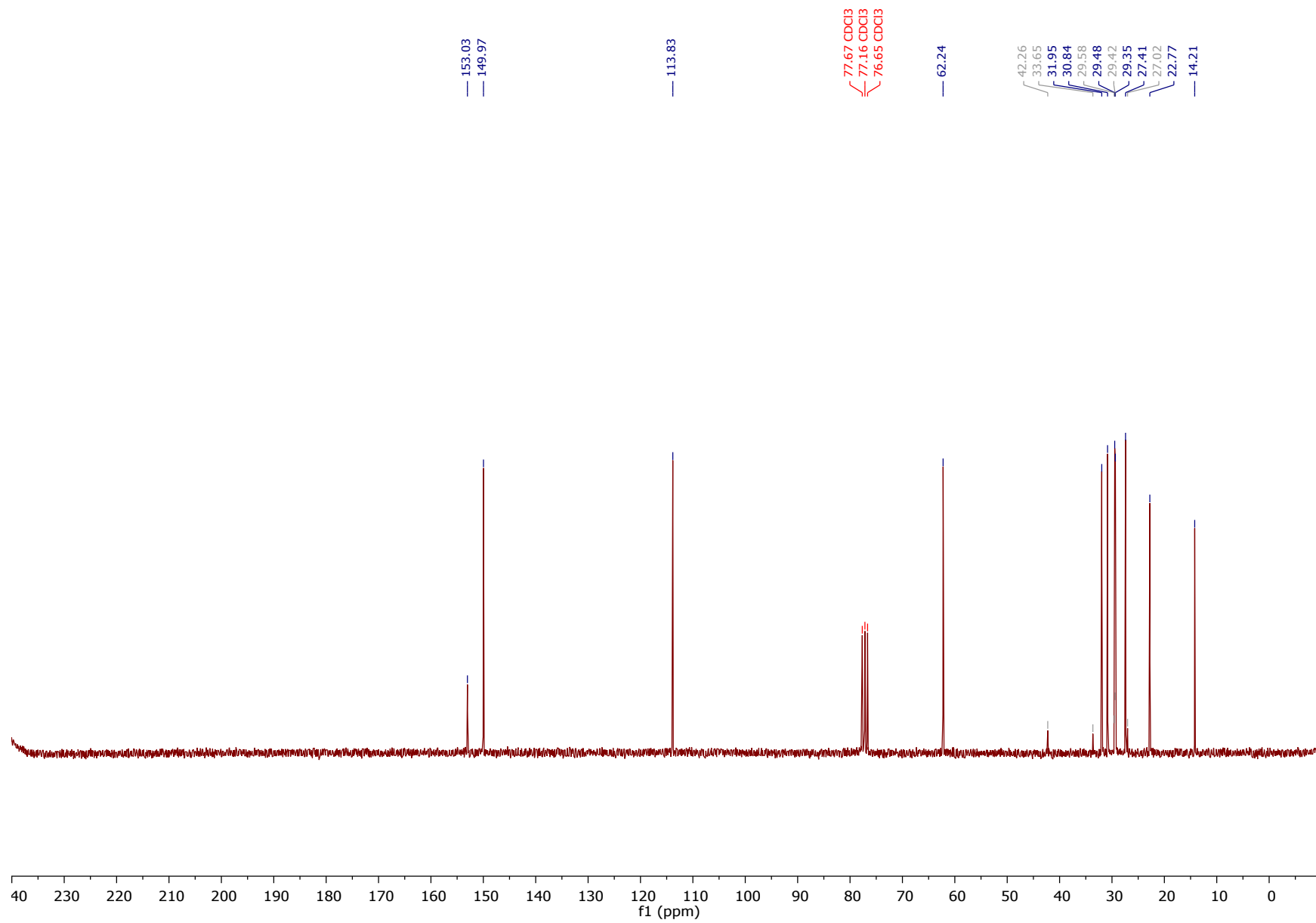
Compound **IIb** is synthesised using octylamine.

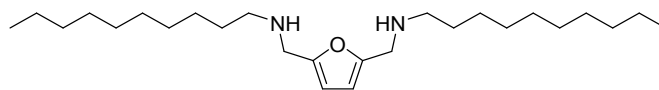
Yield: 96 %, yellow viscous oil.

NMR ¹H (250 MHz, CDCl₃) δ: 6.07 (s, 2H), 3.73 (s, 4H), 2.58 (t, *J* = 7.0 Hz, 4H), 1.72 (br s, 2H), 1.54 – 1.39 (m, 4H), 1.26 (br s, 20H), 0.86 (t, *J* = 6.0 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 153.4, 107.5, 49.4, 46.6, 32.0, 30.1, 29.7, 29.4, 27.5, 22.8, 14.2.





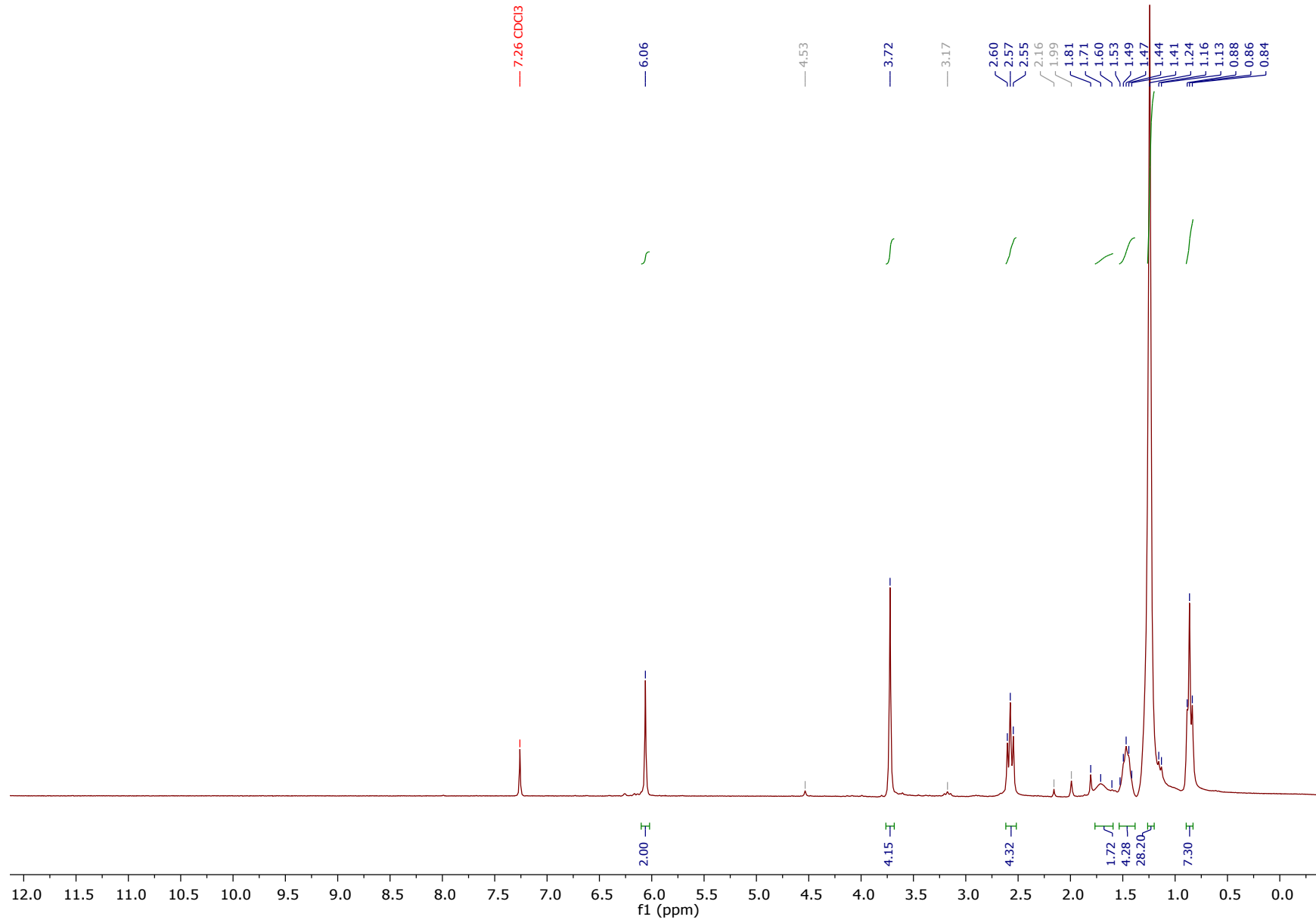
N,N'-(furan-2,5-diylbis(methylene))bis(decane-1-amine) (IIc) :

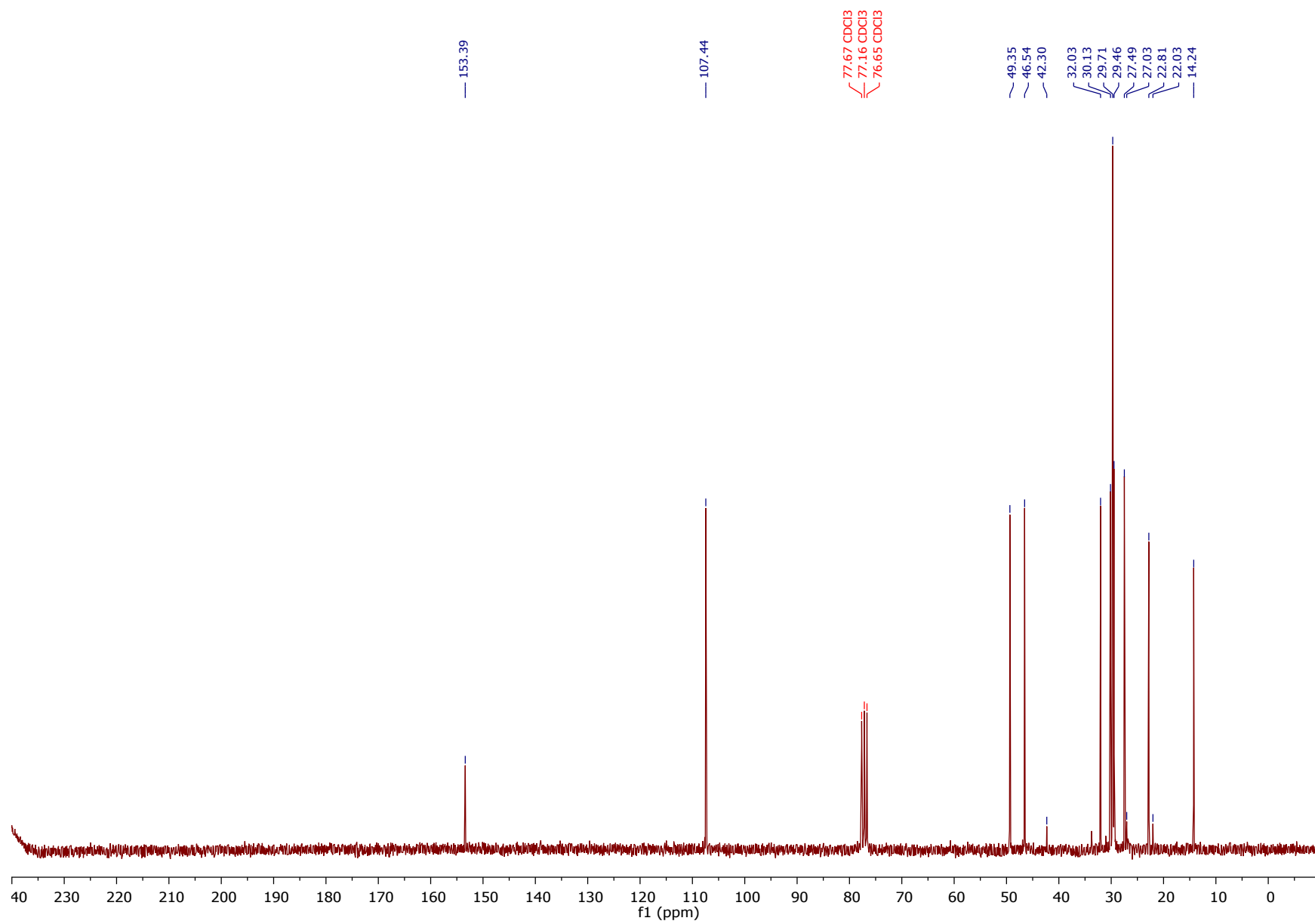
Compound **IIc** is synthesised using decylamine.

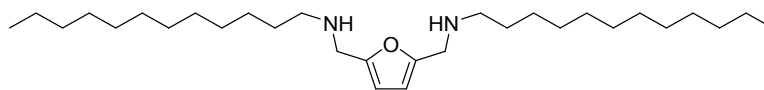
Yield: 99%, white solid.

NMR ¹H (250 MHz, CDCl₃) δ: 6.06 (s, 2H), 3.72 (s, 4H), 2.57 (t, *J* = 7.0 Hz, 4H), 1.71 (br s, 2H), 1.53 – 1.37 (m, 4H), 1.24 (br s, 28H), 0.86 (t, *J* = 6.0 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 153.4, 107.4, 49.4, 46.5, 42.3, 32.0, 30.1, 29.7, 29.5, 27.5, 27.0, 22.8, 22.0, 14.2.





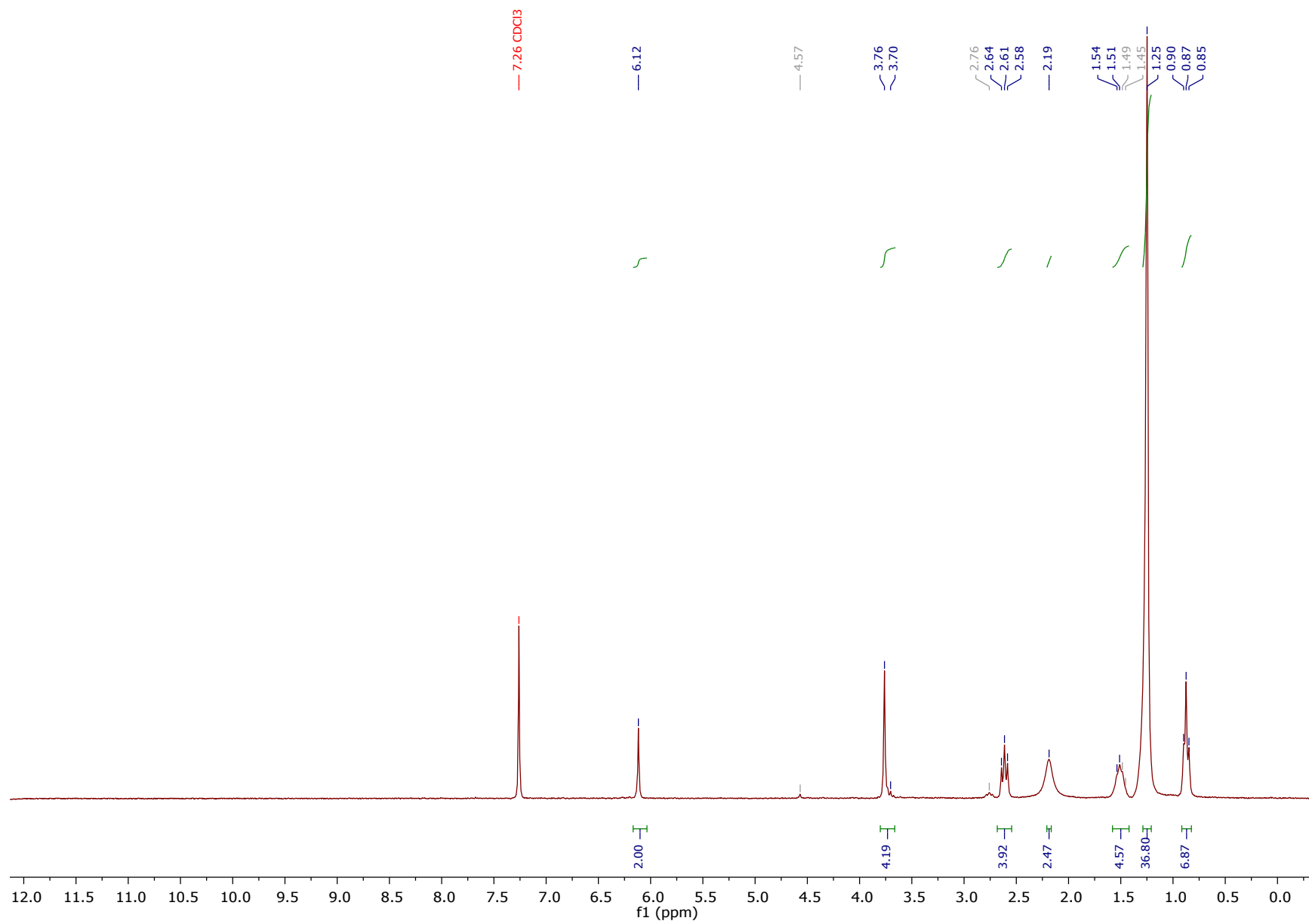
N,N'-(furan-2,5-diylbis(methylene))bis(dodecan-1-amine) (IId) :

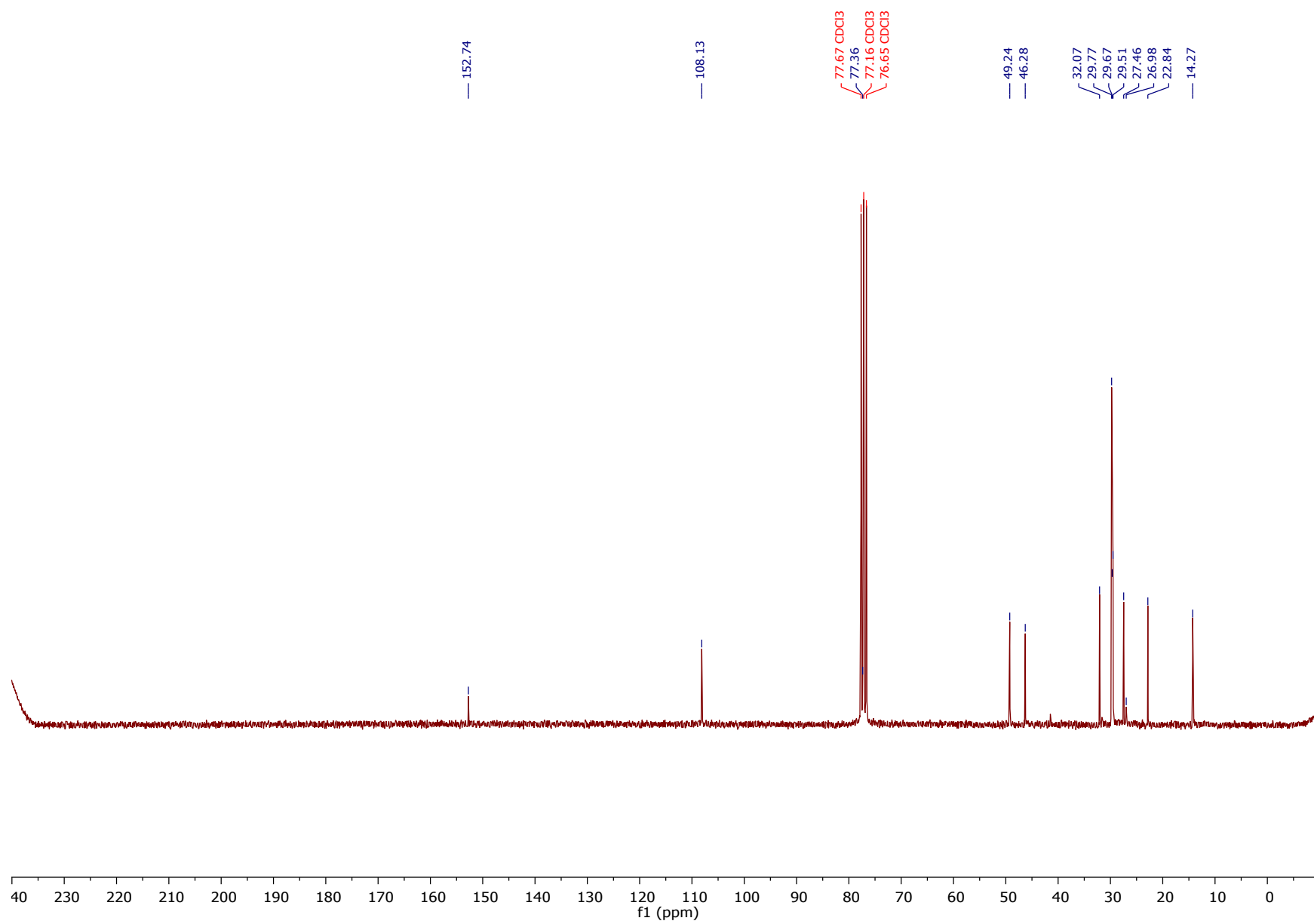
The compound **IId** is synthesised using dodecylamine.

Yield: 98%, white solid.

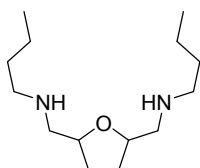
NMR ¹H (250 MHz, CDCl₃) δ: 6.12 (s, 2H), 3.76 (s, 4H), 2.61 (t, *J* = 7.2 Hz, 4H), 2.19 (br s, 2H), 1.57 – 1.44 (m, 4H), 1.25 (br s, 36H), 0.87 (t, *J* = 6.4 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 152.7, 108.1, 49.2, 46.3, 32.1, 29.8, 29.7, 29.5, 27.5, 27.0, 22.8, 14.3.





N,N'-((tetrahydrofuran-2,5-diyl)bis(methylene))bis(butan-1-amine) (2a) :

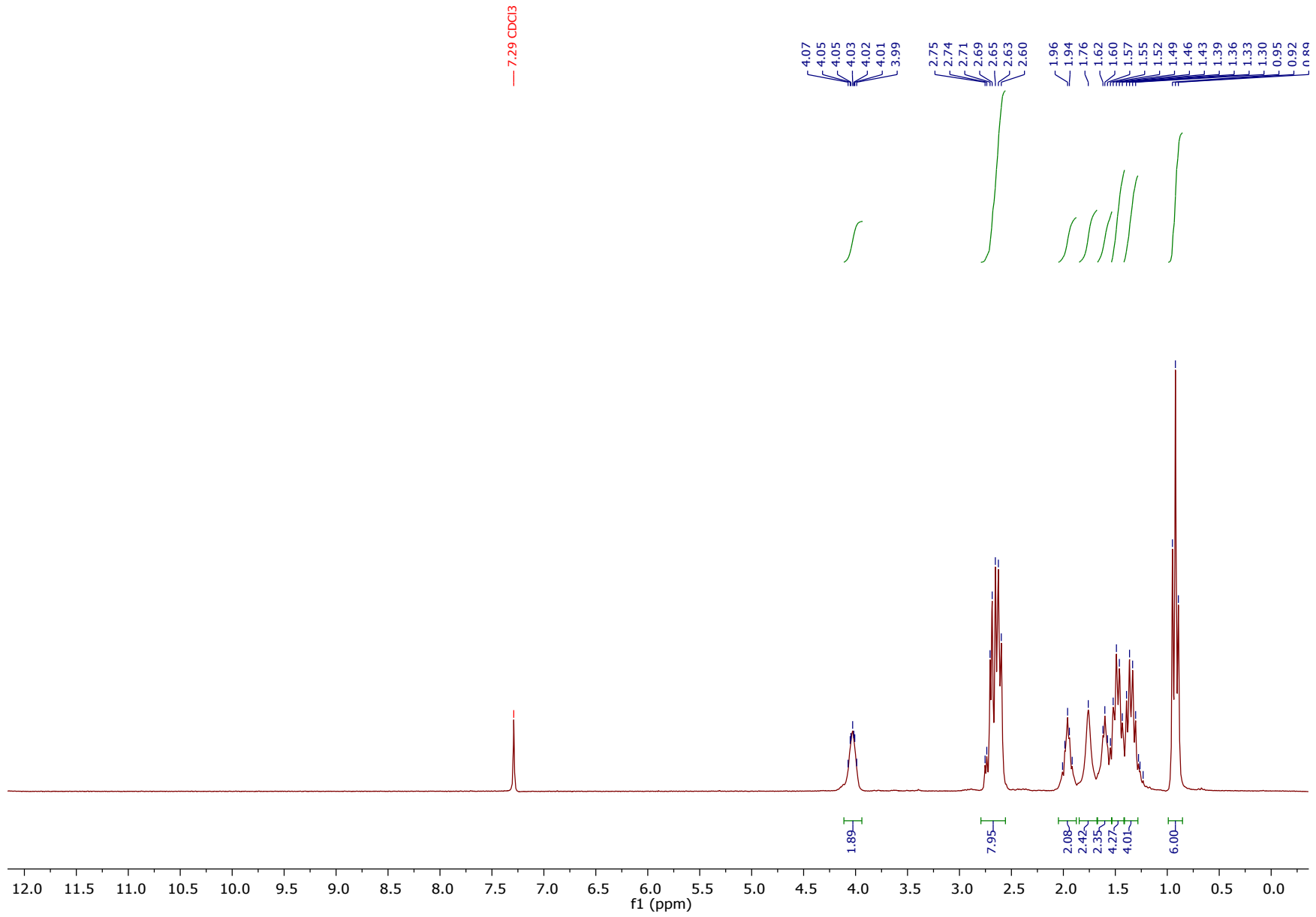


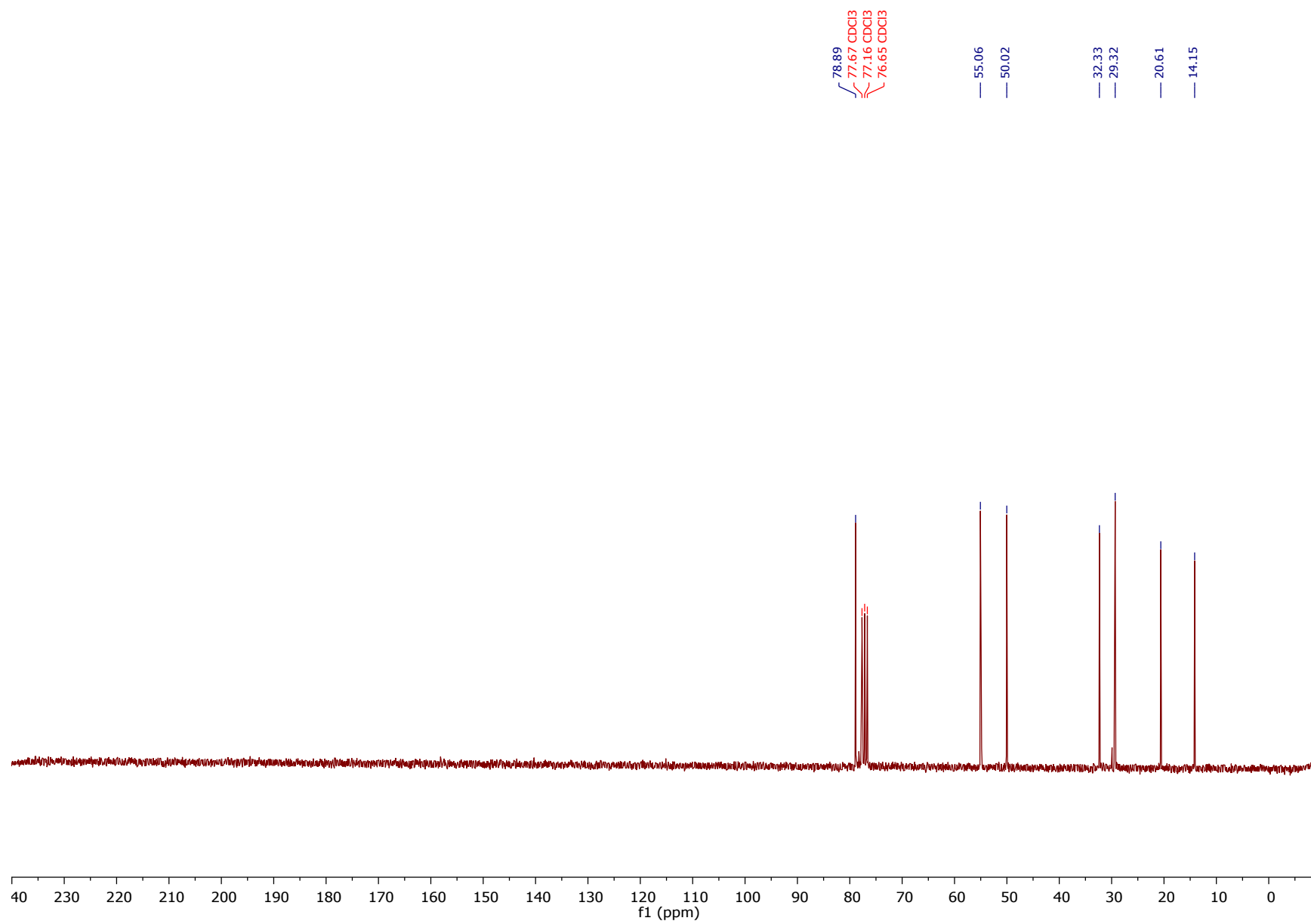
Compound **2a** is synthesised using *n*-butylamine.

Yield: 98%, yellow viscous oil.

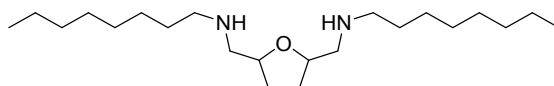
NMR ¹H (250 MHz, CDCl₃) δ: . 4.11 – 3.94 (m, 2H), 2.79 – 2.56 (m, 8H), 1.96 (p, *J* = 7.0, 6.0 Hz, 2H), 1.76 (br s, 2H), 1.67 – 1.53 (m, 2H), 1.48 (m, 4H), 1.35 (m, 4H), 0.92 (t, *J* = 7.0 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 78.9, 55.1, 50.0, 32.3, 29.3, 20.6, 14.2.





N,N'-((tetrahydrofuran-2,5-diyl)bis(methylene))bis(octan-1-amine) (2b) :

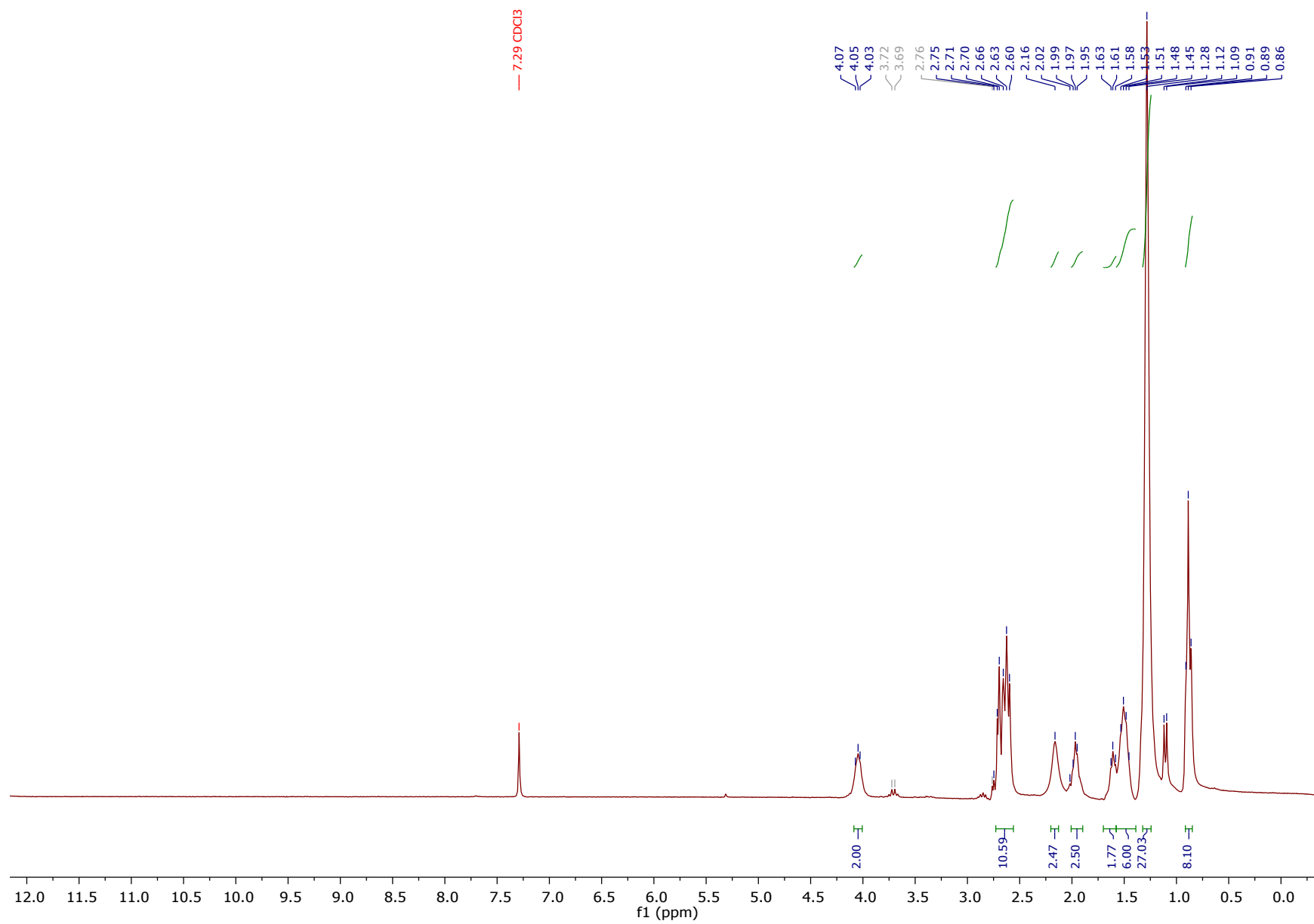


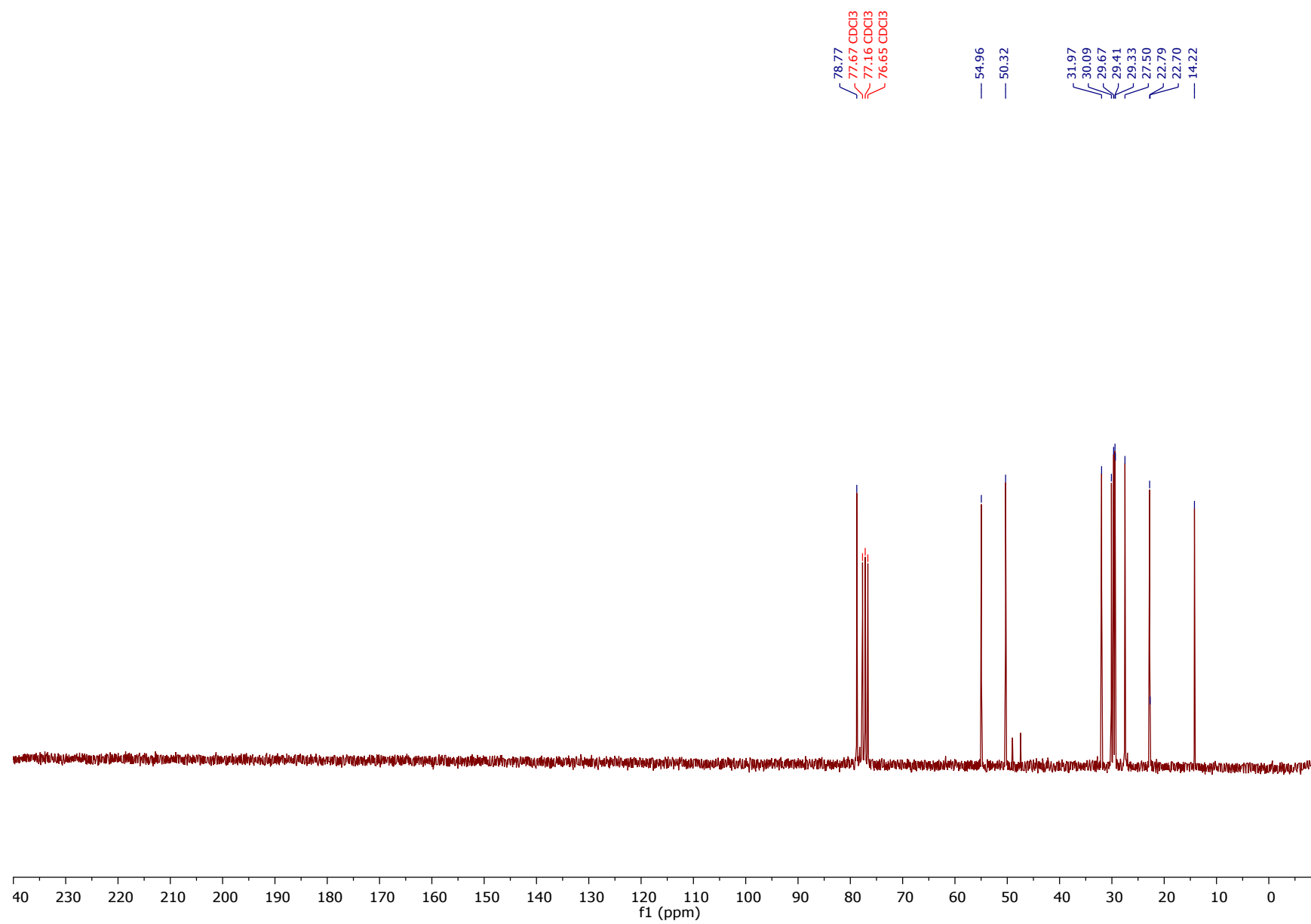
Compound **2b** is synthesised using octylamine.

Yield: 96 %, yellow viscous oil.

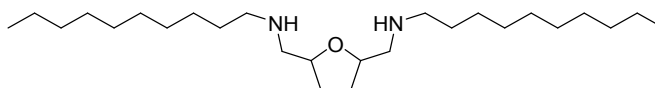
NMR ^1H (250 MHz, CDCl_3) δ : δ 4.15 – 3.96 (m, 2H), 2.77 – 2.57 (m, 10H), 2.16 (br s, 2H), 2.03 – 1.89 (m, 2H), 1.65 – 1.54 (m, 2H), 1.54 – 1.40 (m, 6H), 1.28 (br s, 28H), 0.89 (t, $J = 6.0$ Hz, 6H).

NMR ^{13}C (63 MHz, CDCl_3) δ : 78.8, 55.0, 50.3, 32.0, 30.1, 29.7, 29.4, 29.3, 27.5, 22.8, 22.7, 14.2.





N,N'-((tetrahydrofuran-2,5-diyl)bis(methylene))bis(decane-1-amine) (2c) :



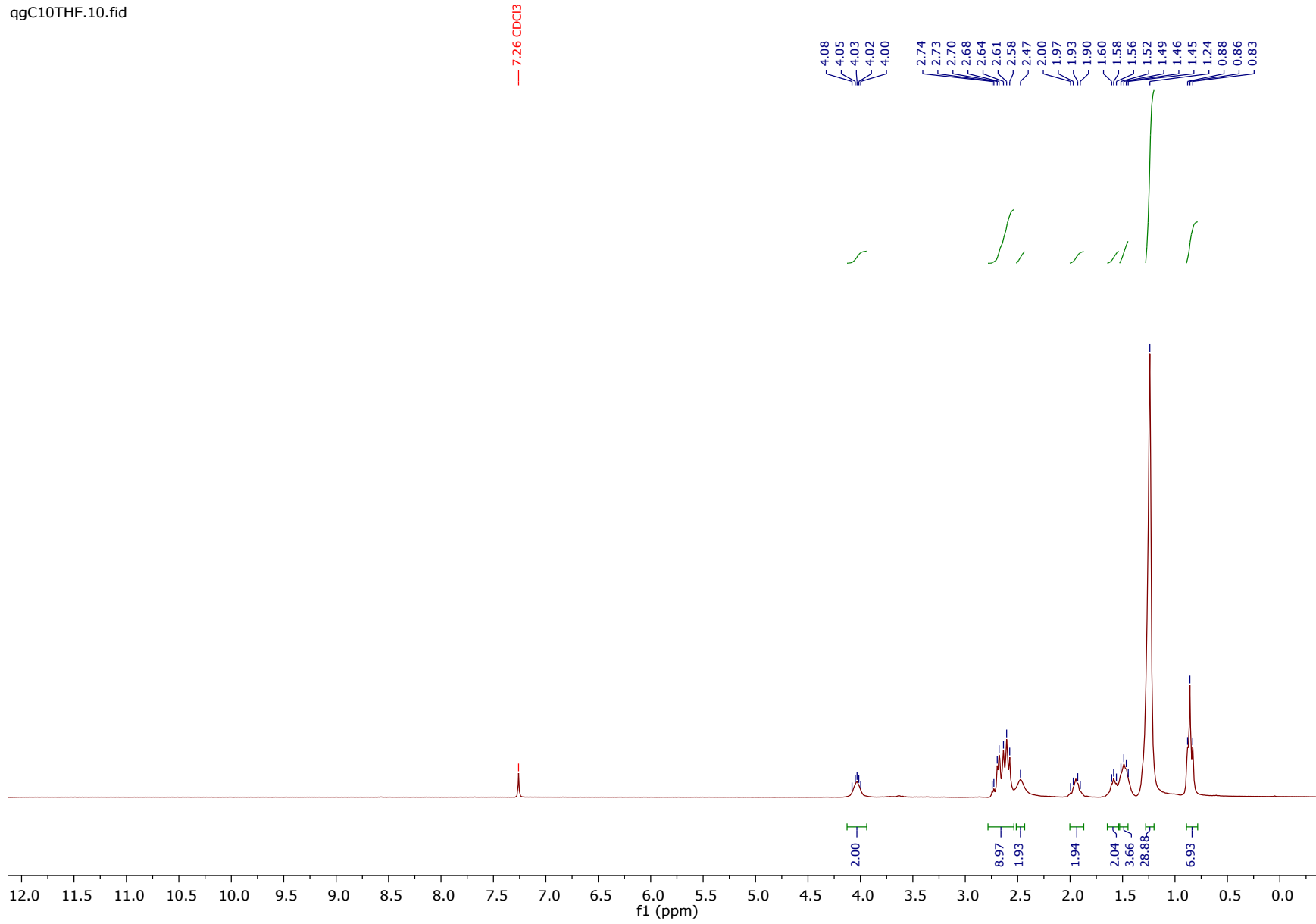
Compound **2c** is synthesised using decylamine.

Yield: 99%, white solid.

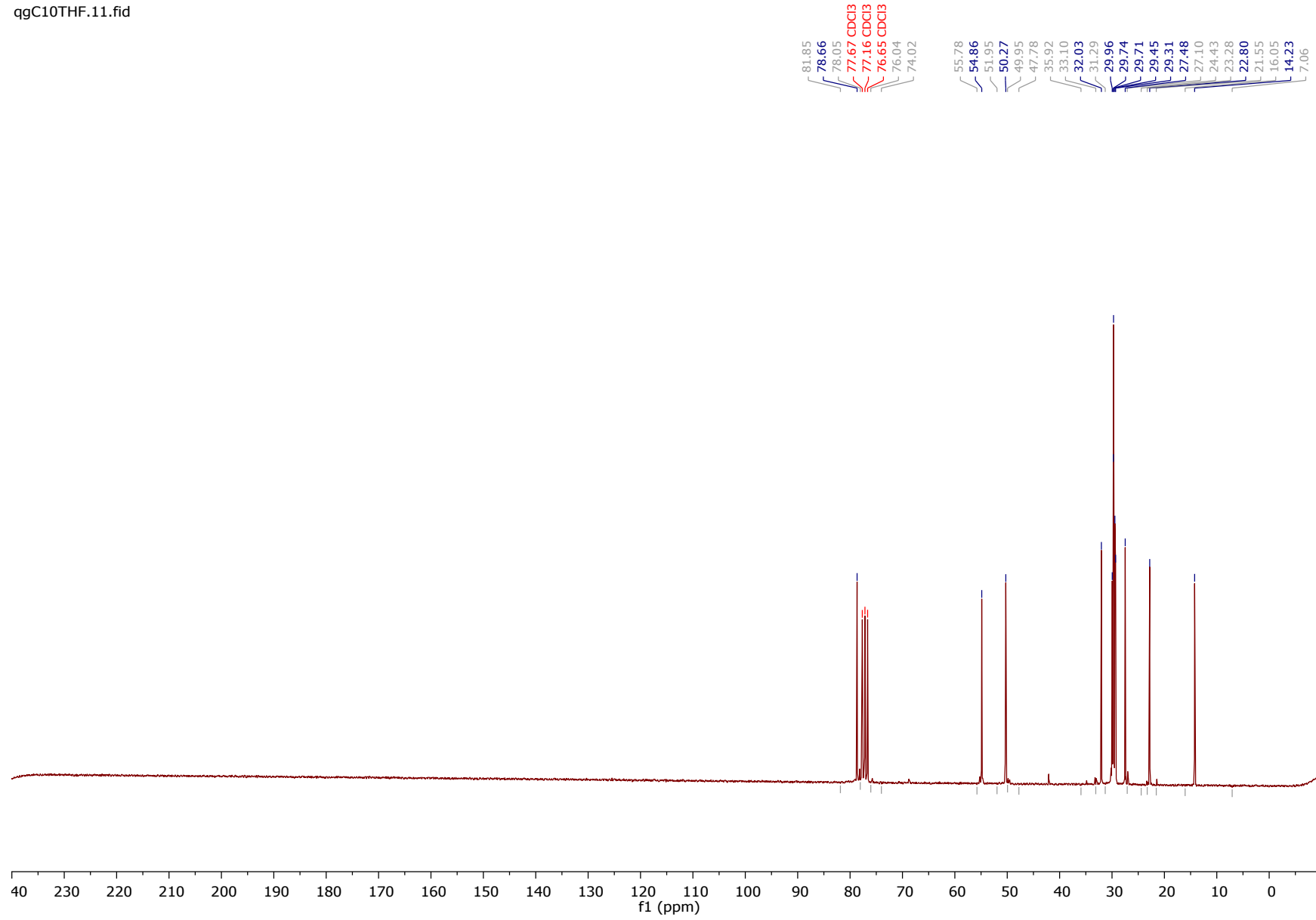
NMR ^1H (250 MHz, CDCl_3) δ : 4.11 – 3.93 (m, 2H), 2.75 – 2.54 (m, 8H), 2.47 (br s, 2H), 2.05 – 1.86 (m, 2H), 1.66 – 1.55 (m, 2H), 1.54 – 1.40 (m, 4H), 1.24 (br s, 28H), 0.86 (t, $J = 6.0$ Hz, 6H).

NMR ^{13}C (63 MHz, CDCl_3) δ : 78.7, 54.9, 50.3, 32.0, 30.0, 29.7, 29.7, 29.5, 29.3, 27.5, 22.8, 14.2.

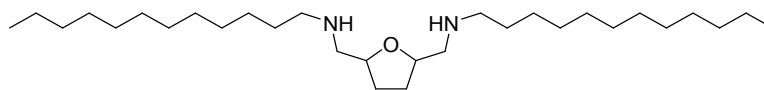
qgC10THF.10.fid



qgC10THF.11.fid



N,N'-((tetrahydrofuran-2,5-diyl)bis(methylene))bis(dodecan-1-amine) (2d) :

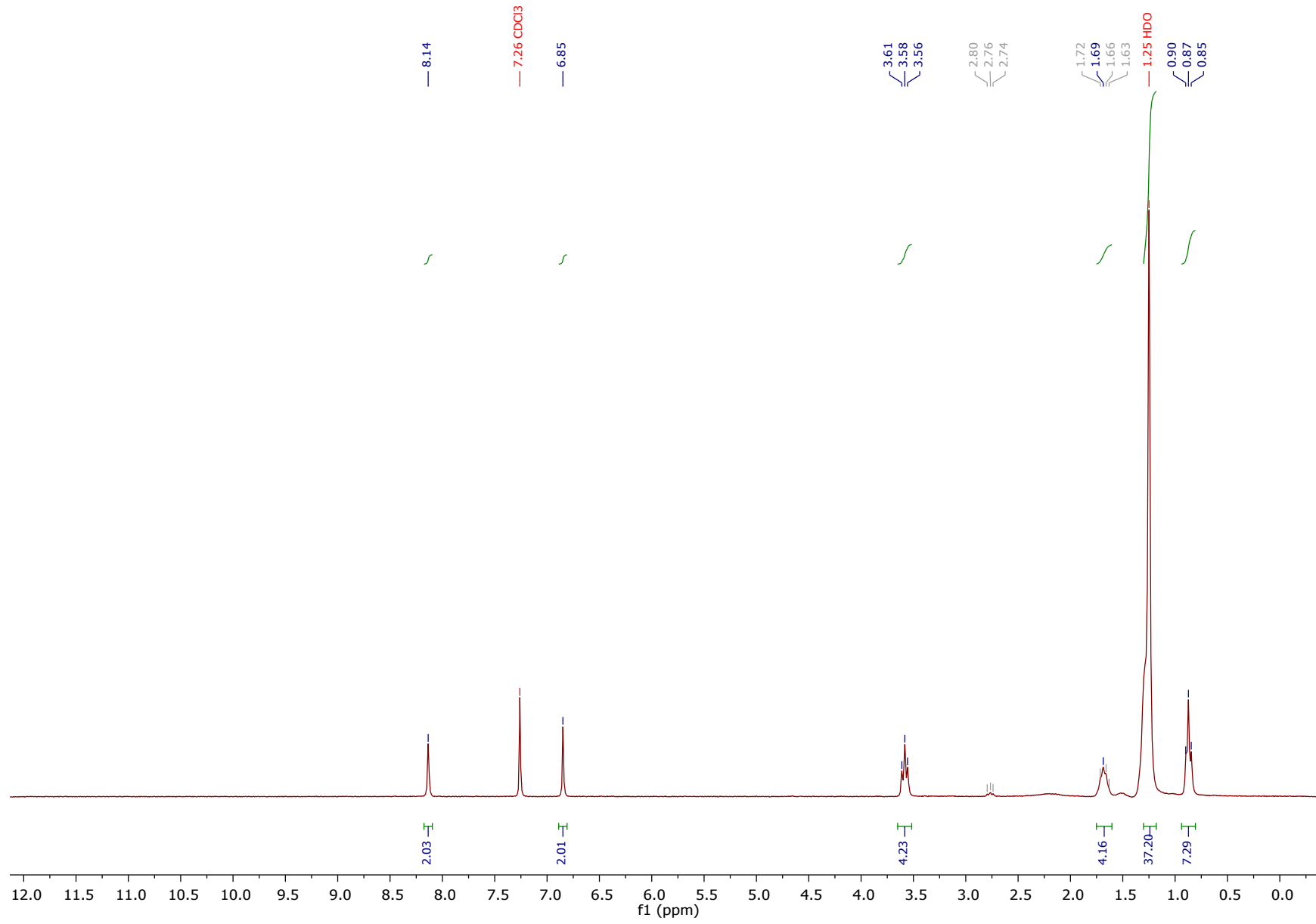


The compound **2d** is synthesised using dodecylamine.

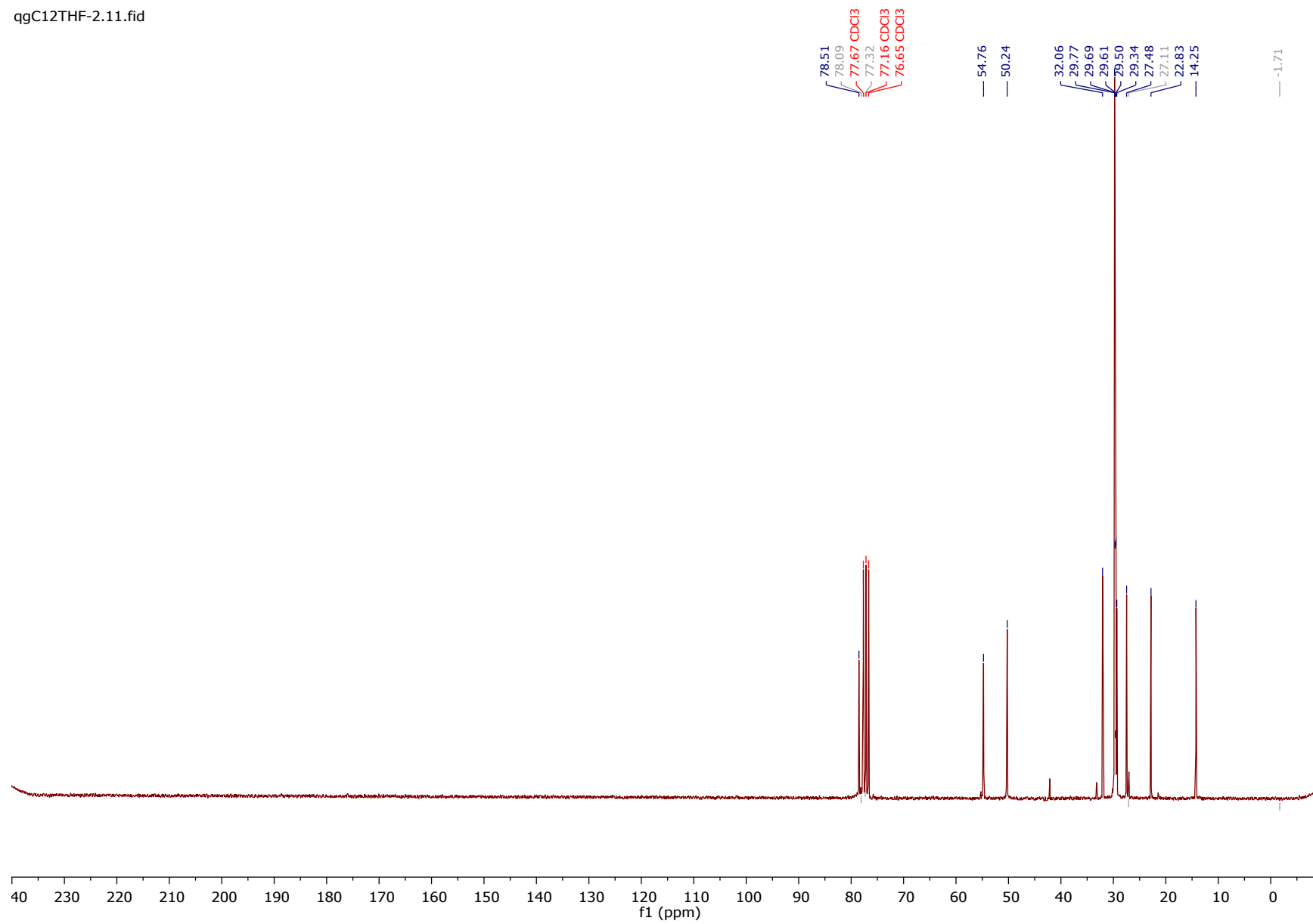
Yield: 98%, white solid.

NMR ¹H (250 MHz, CDCl₃) δ: 4.14 – 4.01 (m, 2H), 2.90 (br s, 2H) 2.78 – 2.58 (m, 8H), 2.03 – 1.89 (m, 2H), 1.66 – 1.57 (m, 2H), 1.56 – 1.41 (m, 4H), 1.24 (br s, 36H), 0.86 (t, *J* = 6.0 Hz, 6H).

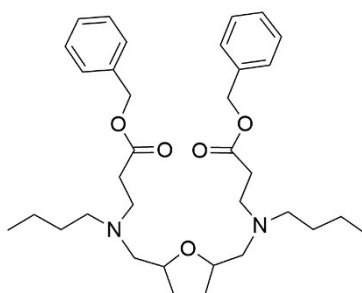
NMR ¹³C (63 MHz, CDCl₃) δ: 78.5, 54.8, 50.2, 32.1, 29.8, 29.7, 29.6, 29.5, 29.3, 27.5, 22.8, 14.3.



qgC12THF-2.11.fid



dibenzyl 3,3'-(((tetrahydrofuran-2,5-diyl)bis(methylene))bis(butylazanediy)) dipropionate
(3a) :



Compound **3a** is synthesised using *n*-butylamine.

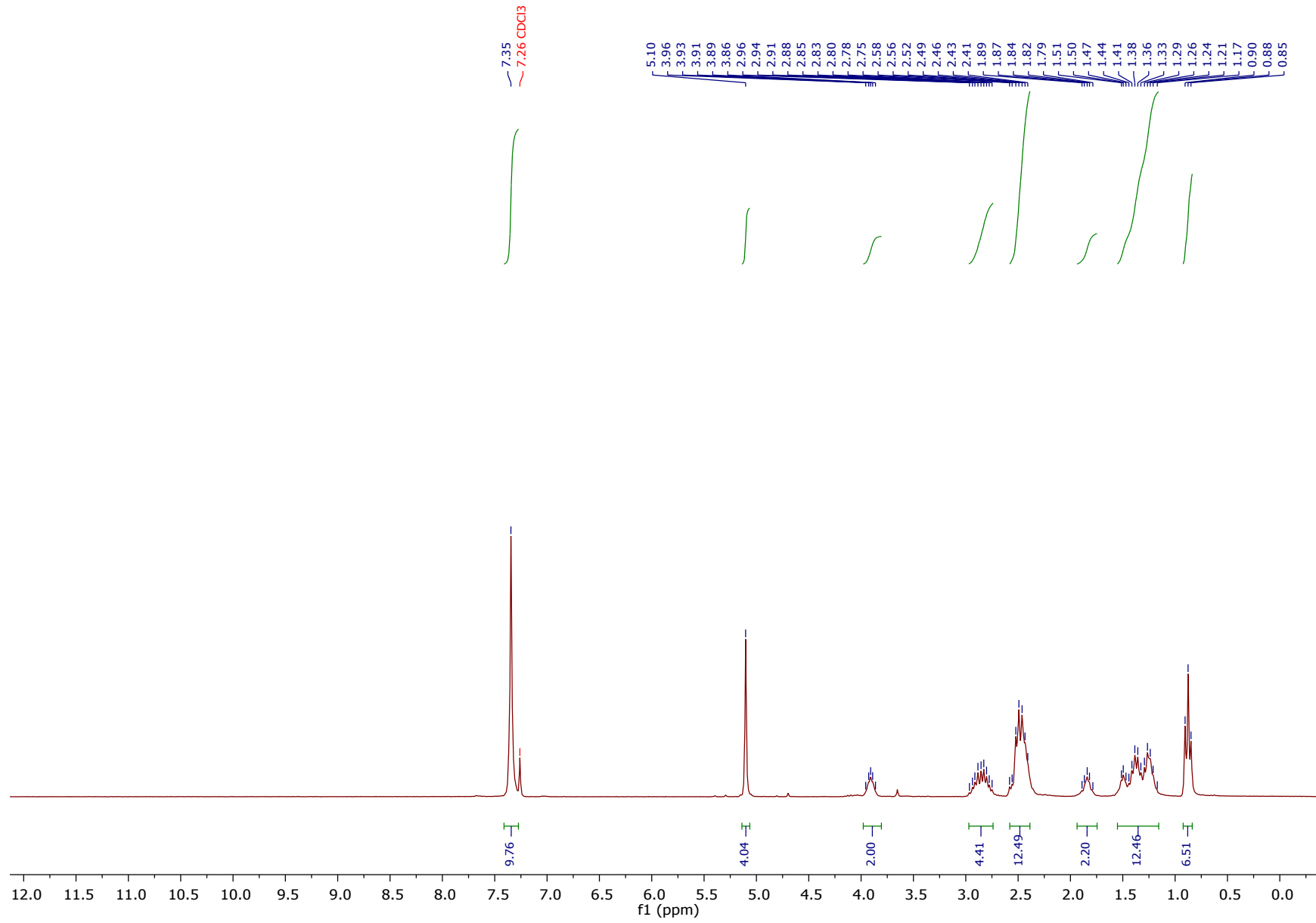
Yield: 78%, yellow highly viscous oil.

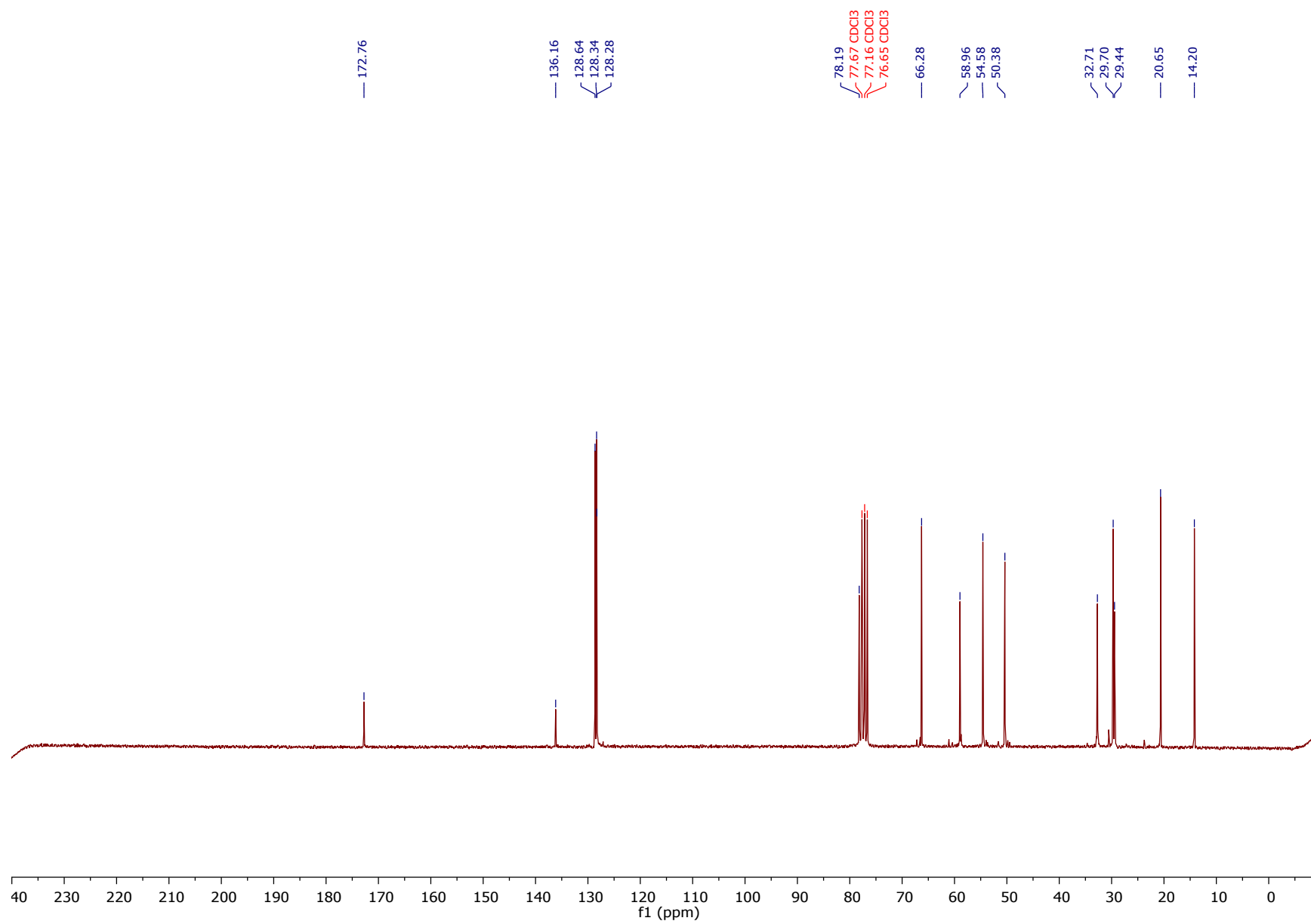
NMR ¹H (250 MHz, CDCl₃) δ: 7.35 (s, 10H), 5.10 (s, 4H), 3.95 – 3.81 (m, 2H), 3.0 – 2.69 (m, 4H), 2.59 – 2.27 (m, 12H), 1.90 – 1.74 (m, 2H), 1.57 – 1.13 (m, 10H), 0.86 (t, *J* = 7 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 172.8, 136.2, 128.6, 128.3, 128.3, 78.2, 66.3, 59.0, 54.6, 50.4, 32.7, 29.7, 29.4, 20.7, 14.2.

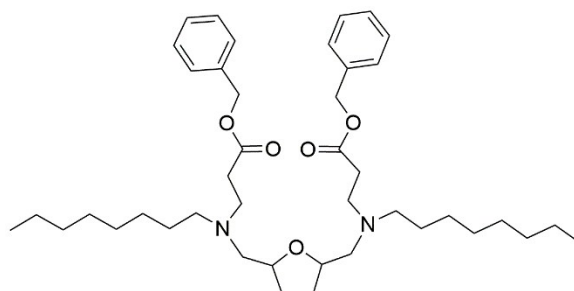
IR (cm⁻¹): 3443, 3064, 3033, 2955, 2928, 2866 2815, 2361, 2336, 1736, 1625, 1498, 1456, 1377, 1347, 1313, 1255, 1184, 1070, 1005, 909, 745, 699, 429, 404.

HRMS (ESI-TOF): *m/z*: [M + H]⁺ Calcd for C₃₄H₅₁N₂O₅ 567.3792; Found 567.3787





dibenzyl 3,3'-(((tetrahydrofuran-2,5-diyl)bis(methylene))bis(octylazanediyl)) dipropionate (3b) :



Compound **3b** is synthesised using octylamine.

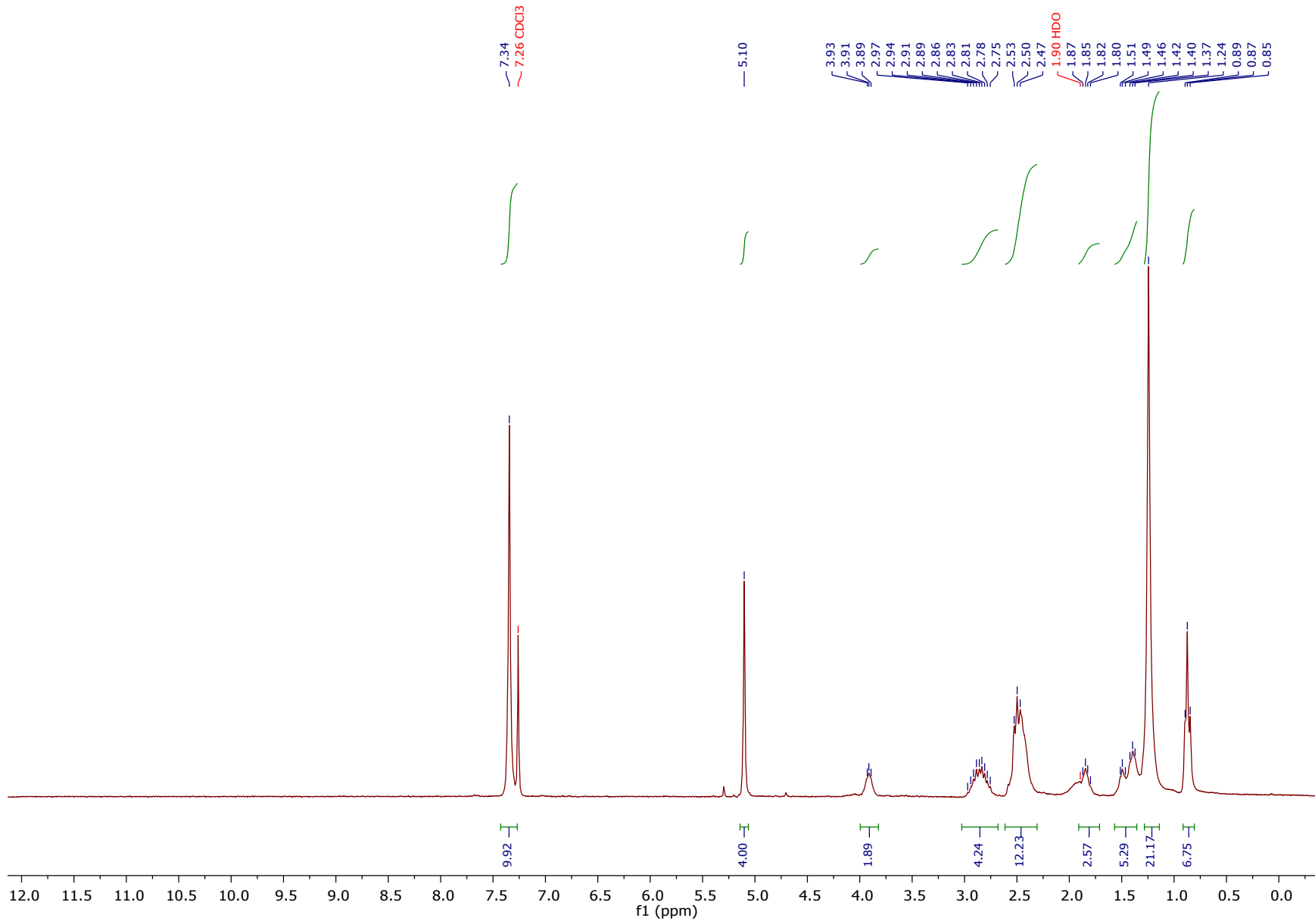
Yield: 83 %, yellow highly viscous oil.

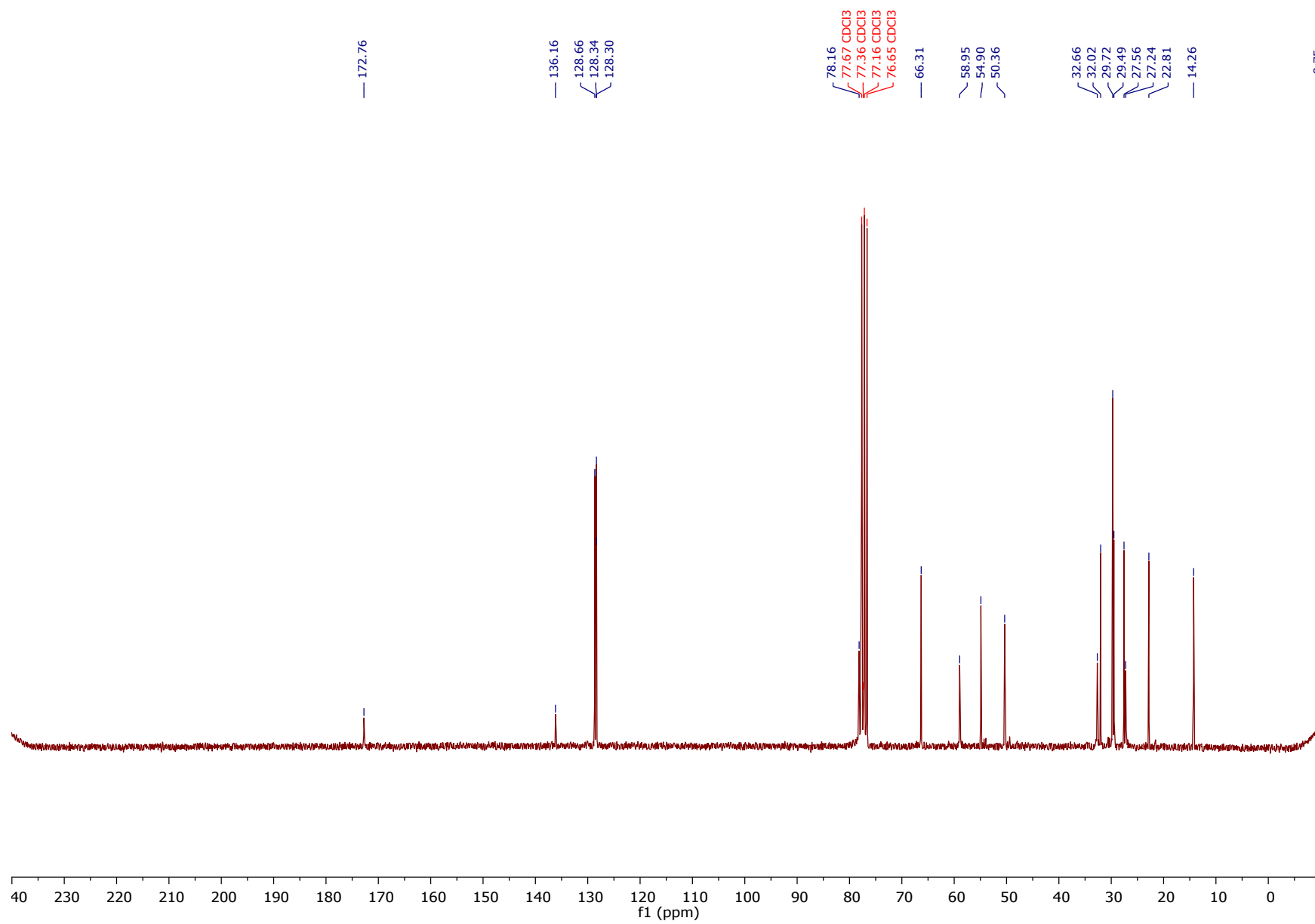
NMR ¹H (250 MHz, CDCl₃) δ: 7.34 (s, 10H), 5.10 (s, 4H), 4.00 – 3.86 (m, 2H), 2.99 – 2.74 (m, 4H), 3.60 – 2.37 (m, 12H), 1.98 – 1.77 (m, 2H), 1.66 – 1.18 (m, 26H), 0.87 (t, *J* = 7 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 172.8, 136.2, 128.7, 128.3, 128.3, 78.2, 66.3, 59.0, 54.9, 50.4, 32.7, 32.0, 29.7, 29.5, 27.6, 27.2, 22.8, 14.3.

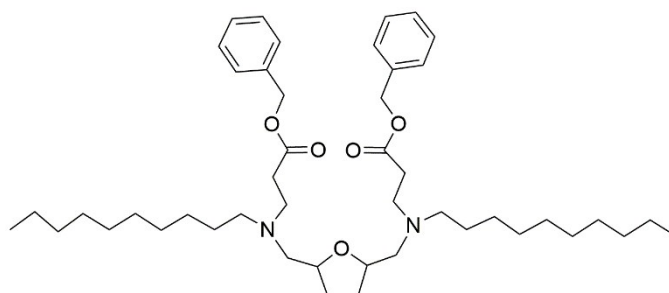
IR (cm⁻¹): 3440, 3065, 3033, 2926, 2854, 1737, 1624, 1498, 1456, 1377, 1348, 1312, 1254, 1170, 1066, 1005, 744, 697, 436.

HRMS (ESI-TOF): m/z: [M + H]⁺ Calcd for C₄₂H₆₇N₂O₅ 679.5044; Found 679.5057.





dibenzyl 3,3'-(((tetrahydrofuran-2,5-diyl)bis(methylene))bis(decylazanediy))dipropionate
(3c) :



Compound **3c** is synthesised using decylamine.

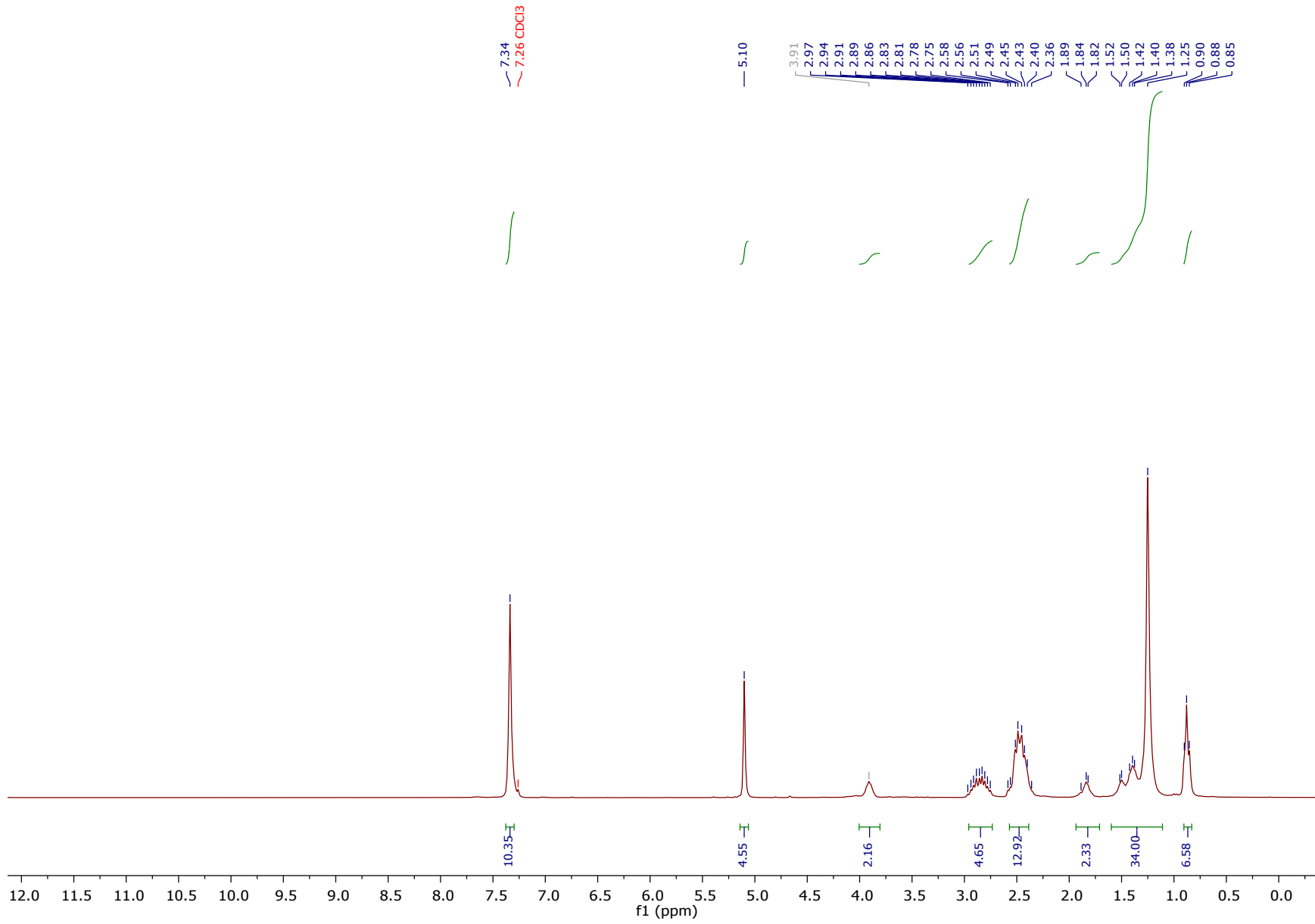
Yield: 79%, colourless gum.

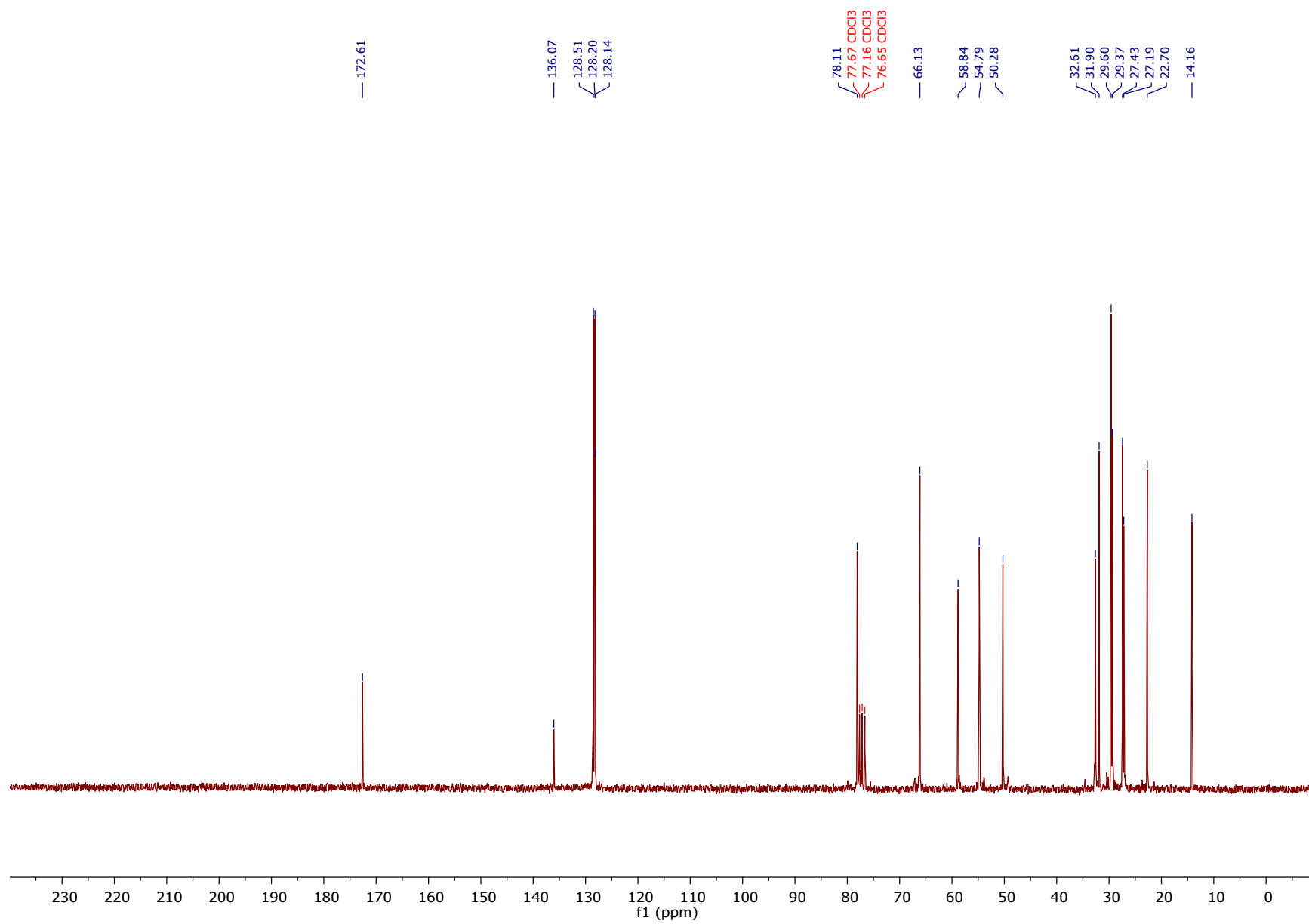
NMR ¹H (250 MHz, CDCl₃) δ: 7.35 (s, 10H), 5.10 (s, 4H), 4.00 – 3.83 (m, 2H), 2.98 – 2.66 (m, 4H), 2.60 – 2.29 (m, 12H), 1.94 – 1.68 (m, 2H), 1.56 – 1.12 (m, 34H), 0.87 (t, *J* = 7,0 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 172.6, 136.1, 128.5, 128.2, 128.1, 78.11, 66.13, 58.8, 54.8, 50.3, 32.6, 31.9, 29.6, 29.4, 27.4, 27.2, 22.7, 14.2.

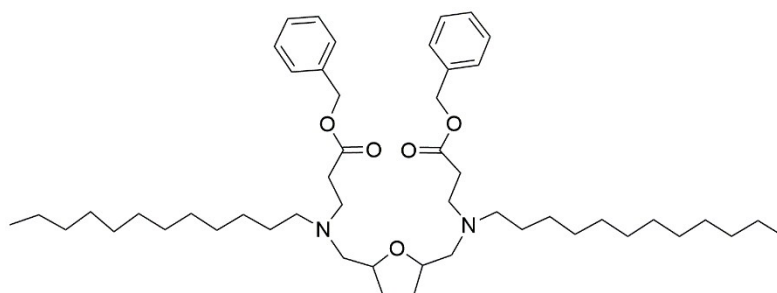
IR (cm⁻¹): 3442, 3064, 3033, 2925, 2854, 1738, 1616, 1498, 1460, 1377, 1348, 1311, 1256, 1169, 1067, 1005, 909, 743, 697, 671, 442, 404.

HRMS (ESI-TOF): *m/z*: [M + H]⁺ Calcd for C₄₆H₇₅N₂O₅ 735.5670; Found 735.5668.





**dibenzyl 3,3'-(((tetrahydrofuran-2,5-diyl)bis(methylene))bis
(dodecylazanediy))dipropionate (3d) :**



The compound **3d** is synthesised using dodecylamine.

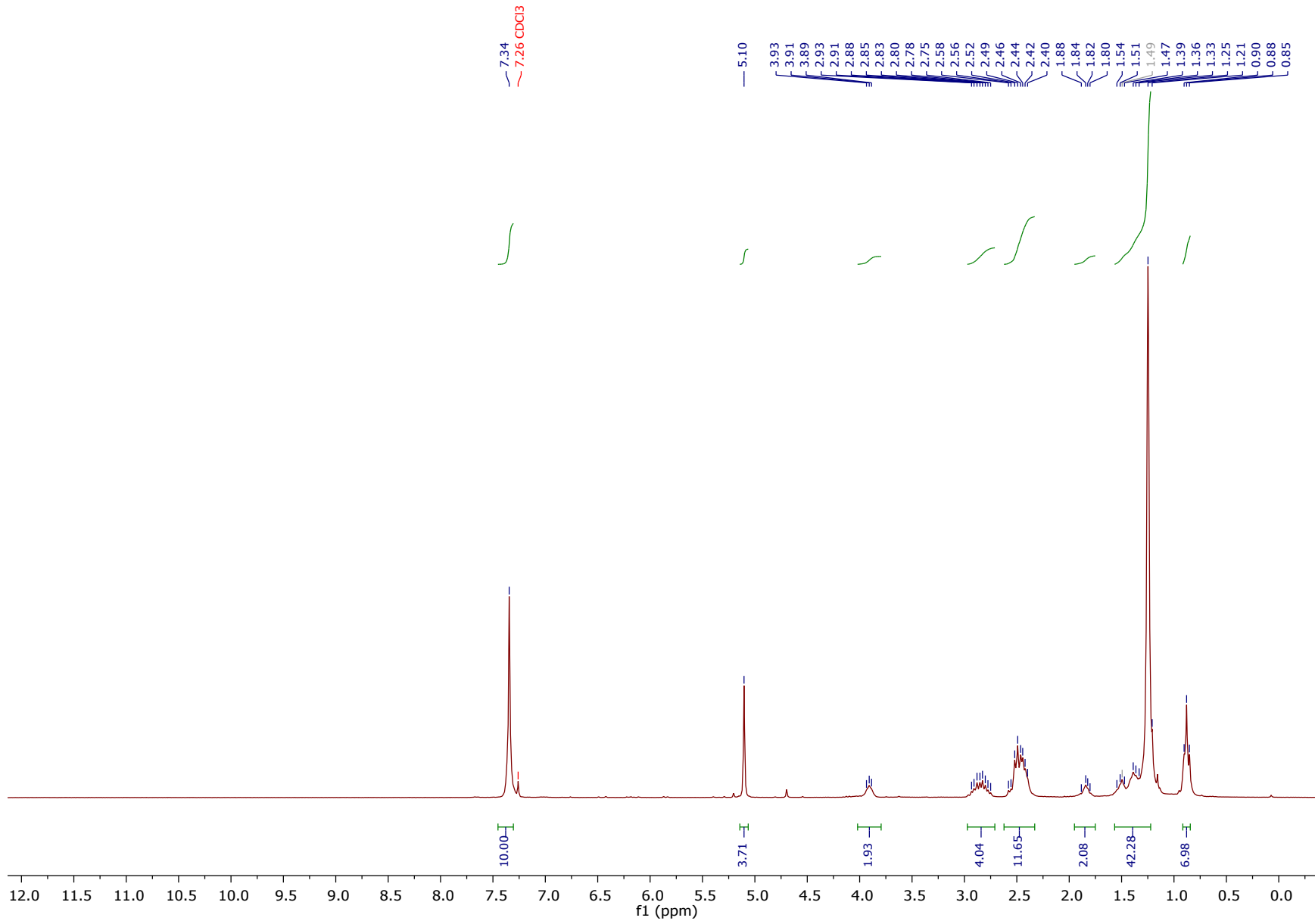
Yield: 77%, colourless gum.

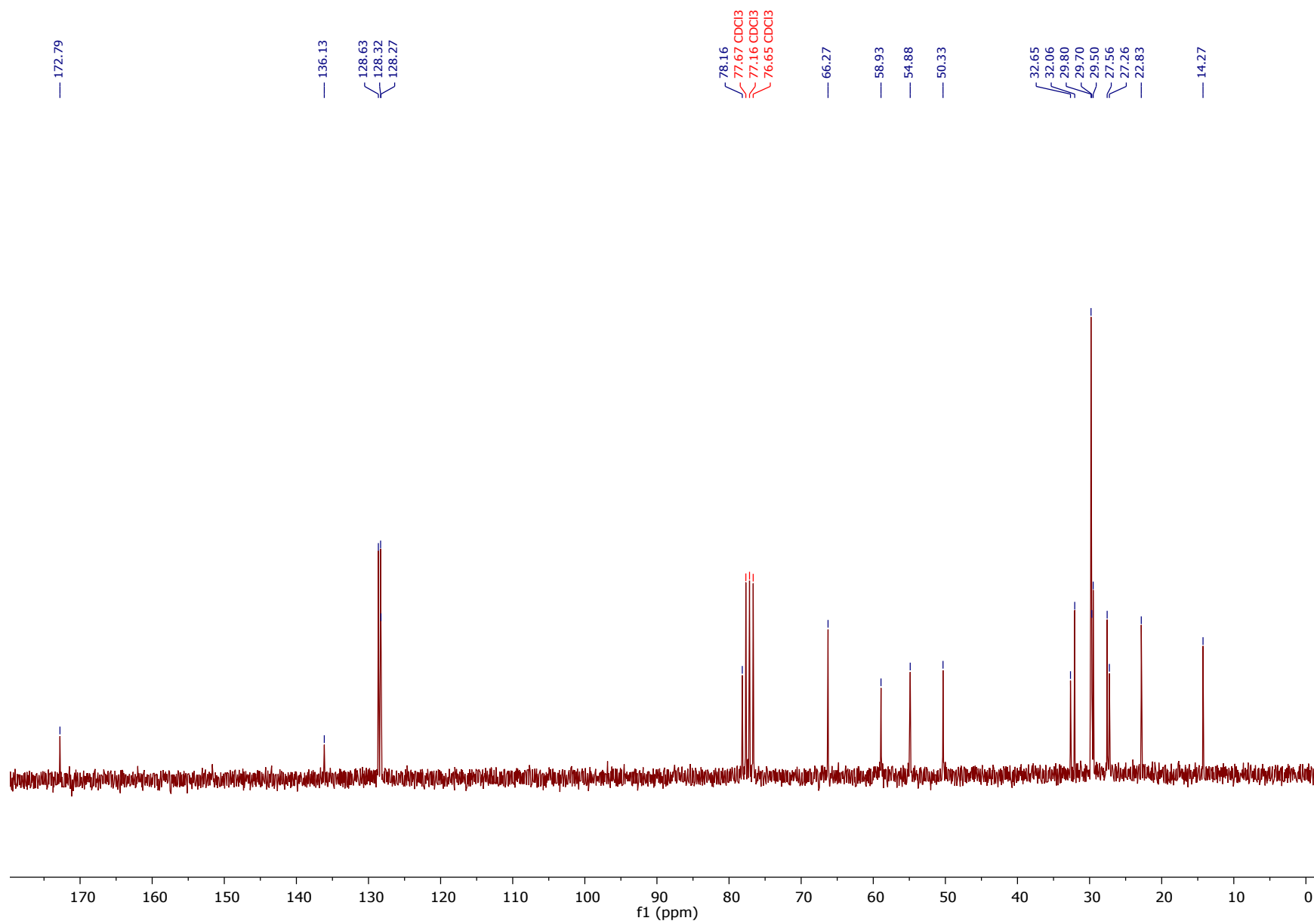
NMR ¹H (250 MHz, CDCl₃) δ: 7,35 (s, 10H), 5,10 (s, 4H), 3,98 – 3,84 (m, 2H), 2,98 – 2,72 (m, 4H), 2,58 – 2,39 (m, 12H), 1,93 – 1,72 (m, 2H), 1,55 – 1,13 (m, 42H), 0,88 (t, *J* = 6,0 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 172.8, 136.1, 128.6, 128.3 128.3, 78.2, 66.3, 59.0, 54.9, 50.3, 32.7, 32.1, 29.8, 29.7, 29.5, 27.6, 27.3, 22.8, 14.3

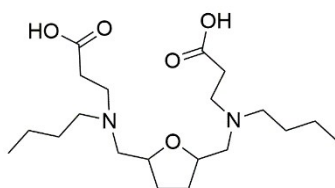
IR (cm⁻¹): 3442, 3090, 3063, 3034, 2924, 2853, 1738, 1630, 1498, 1460, 1376, 1314, 1254, 1167, 1069, 1005, 910, 825, 743, 698, 444, 404.

HRMS (ESI-TOF): *m/z*: [M + H]⁺ Calcd for C₅₀H₈₃N₂O₅ 791.6297; Found 791.6295.





3,3'-(((tetrahydrofuran-2,5-diyl)bis(methylene))bis(butylazanediyl))dipropionic acid (4a) :



The compound **4a** is synthesised using butylamine.

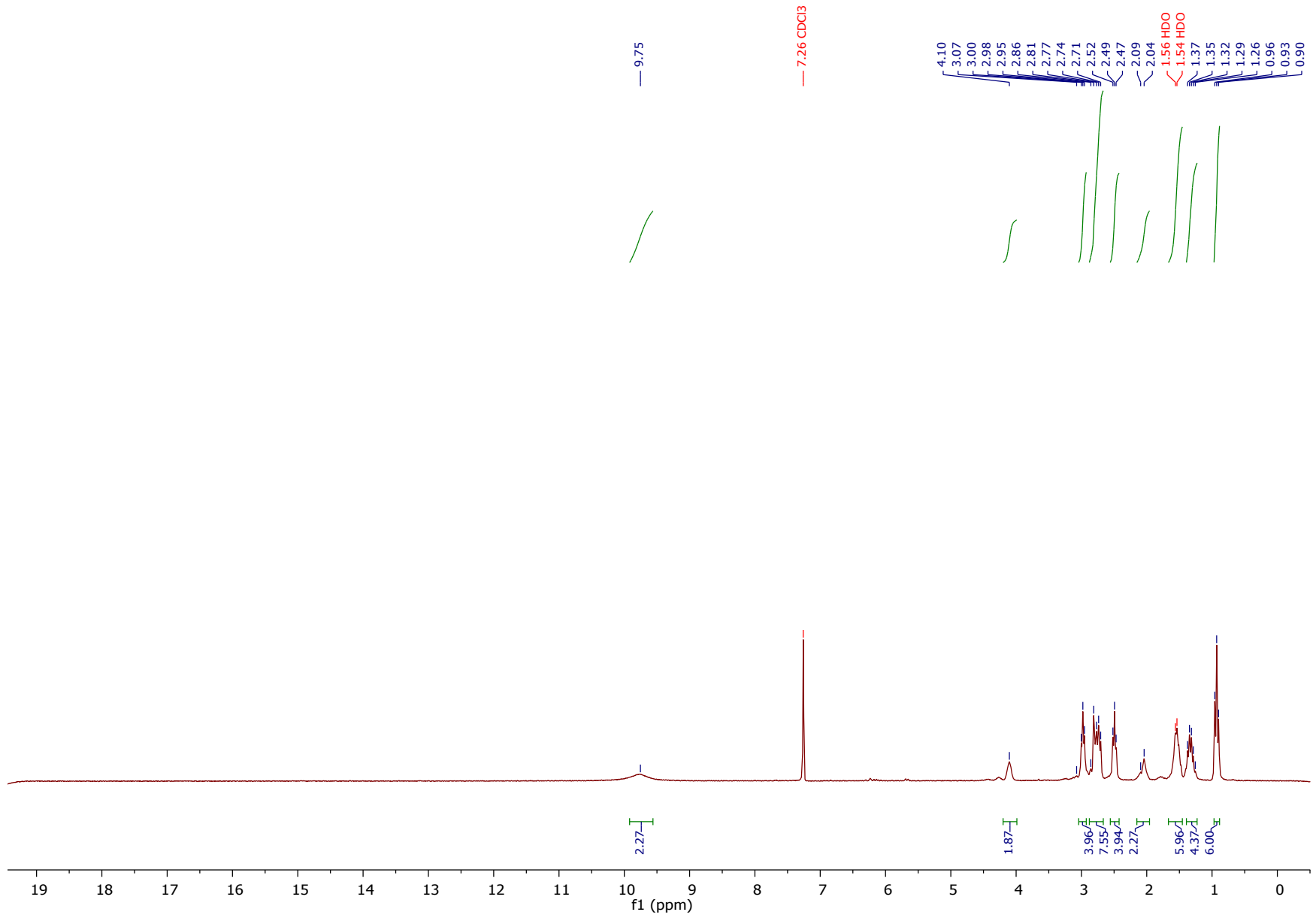
Yield: 39%, yellow highly viscous oil.

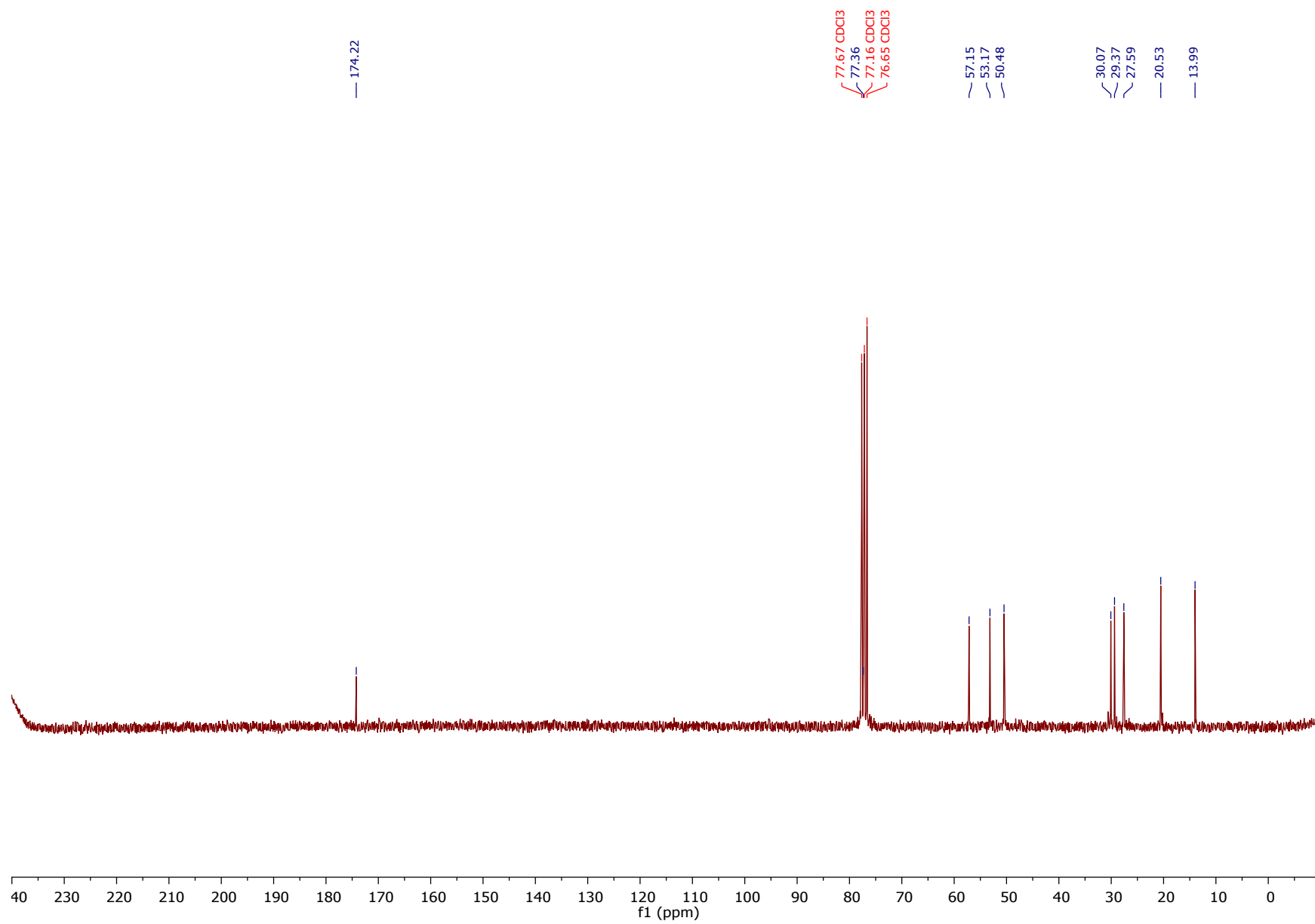
NMR ^1H (250 MHz, CDCl_3) δ : 9.83 (s, 2H), 4.17 – 4.01 (m, 2H), 3.01 (t, $J = 6$ Hz, 4H), 2.88 – 2.71 (m, 8H), 2.52 (t, $J = 6$ Hz, 4H), 2.16-1.97 (m, 2H), 1.68 – 1.48 (m, 6H), 1.47 – 1.24 (m, 4H), 0.93 (t, $J = 7$ Hz, 6H).

NMR ^{13}C (63 MHz, CDCl_3) δ : 174.2, 77.4, 57.2, 53.2, 50.5, 30.1, 29.4, 27.6, 20.5, 14.0.

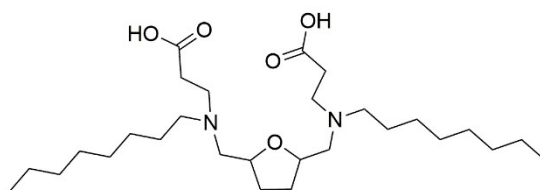
IR (cm^{-1}): 3412, 2955, 2926, 2855, 280, 2736, 2683, 2603, 1713, 1591, 1464, 1384, 1310, 1205, 1146, 1070, 905, 807, 658.

HRMS (ESI-TOF): m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{20}\text{H}_{39}\text{N}_2\text{O}_5$ 387.2853; Found 387.2827





3,3'-(((tetrahydrofuran-2,5-diyl)bis(methylene))bis(acylazanediy))dipropionic acid (4b**) :**



The compound **4b** is synthesised using octylamine.

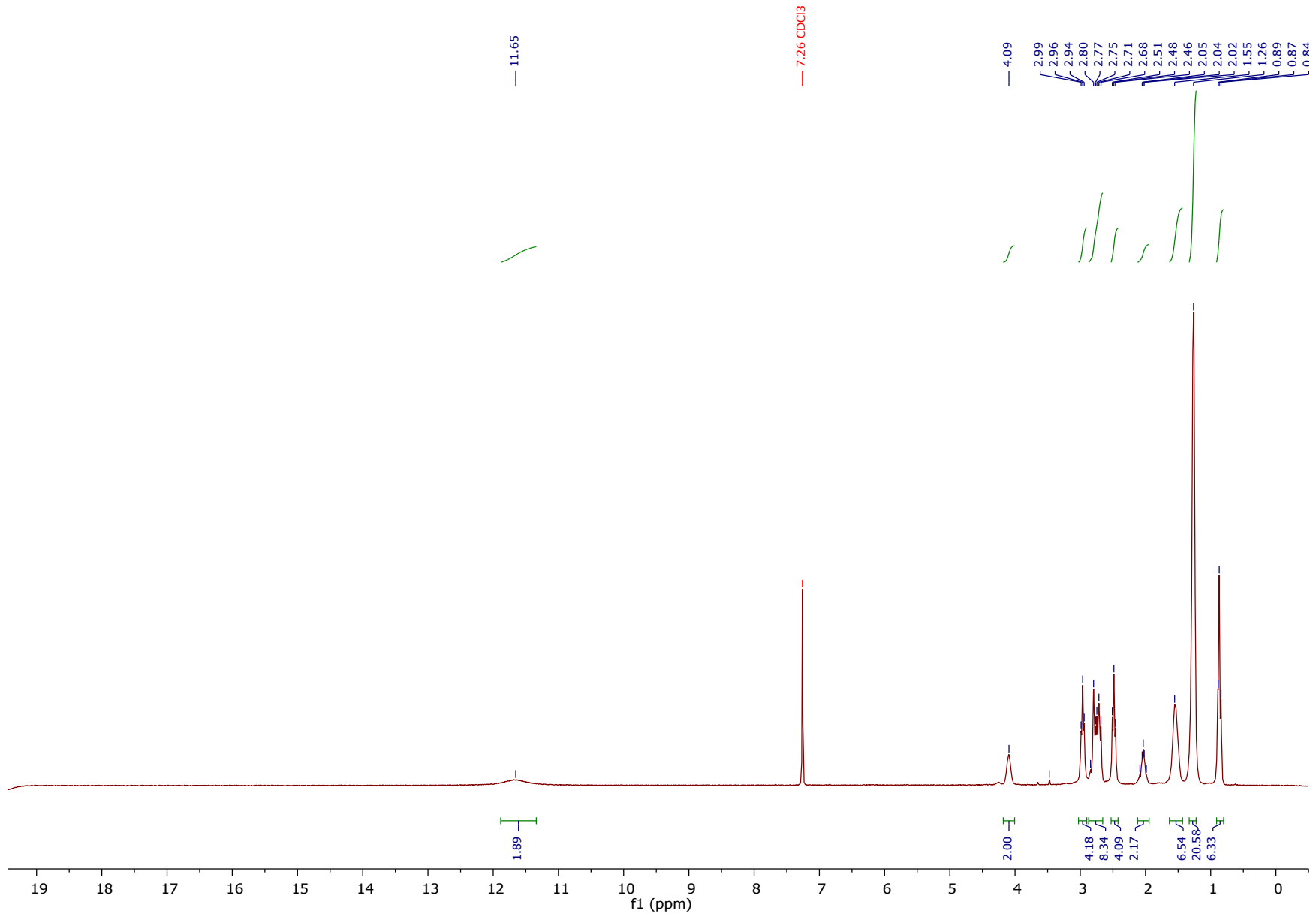
Yield: 58 %, slightly yellow highly viscous oil.

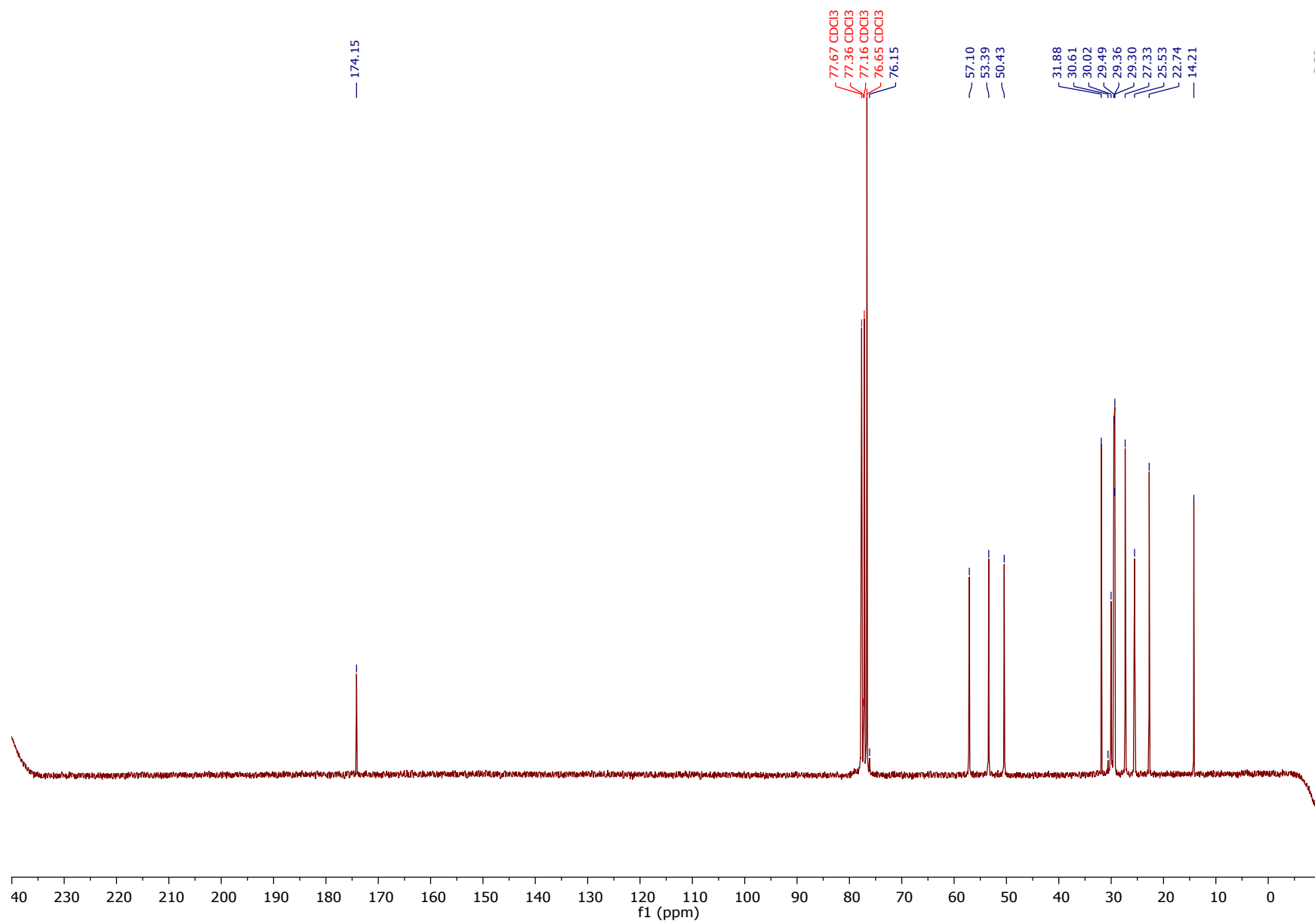
NMR ¹H (250 MHz, CDCl₃) δ: 11.65 (s, 2H), 4.19 – 4.06 (s, 2H), 2.96 (t, *J* = 6 Hz, 4H), 2.86 – 2.66 (m, 8H), 2.48 (t, *J* = 6 Hz, 4H), 2.19 – 1.96 (m, 2H), 1.68 – 1.45 (m, 6H), 1.40 – 1.18 (m, 20H), 0.87 (t, *J* = 6 Hz, 6H).

NMR ¹³C: (63 MHz, CDCl₃) δ: 174.2, 76.2, 57.1, 53.4, 50.4, 31.9, 30.6, 30.0, 29.5, 29.4, 29.3, 27.3, 25.5, 22.7, 14.2.

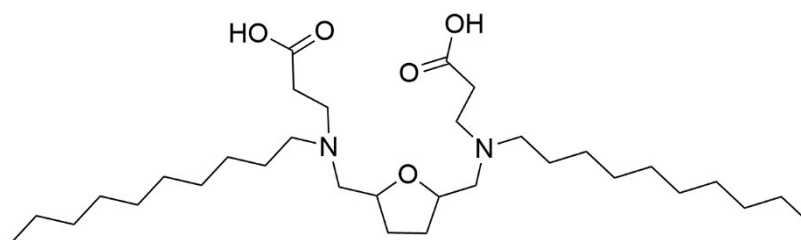
IR (cm⁻¹): 3410, 2954, 2926, 2855, 2803, 2729, 2584, 1956, 1714, 1590, 1463, 1381, 1302, 1205, 1072, 906, 804, 721, 665.

HRMS (ESI-TOF): *m/z*: [M + H]⁺ Calcd for C₂₈H₅₅N₂O₅ 499.4105; Found 499.4105





3,3'-(((tetrahydrofuan-2,5-diyl)bis(methylene))bis(decylazanediy))dipropionic acid (4c) :



The compound **4c** is synthesised using décylamine.

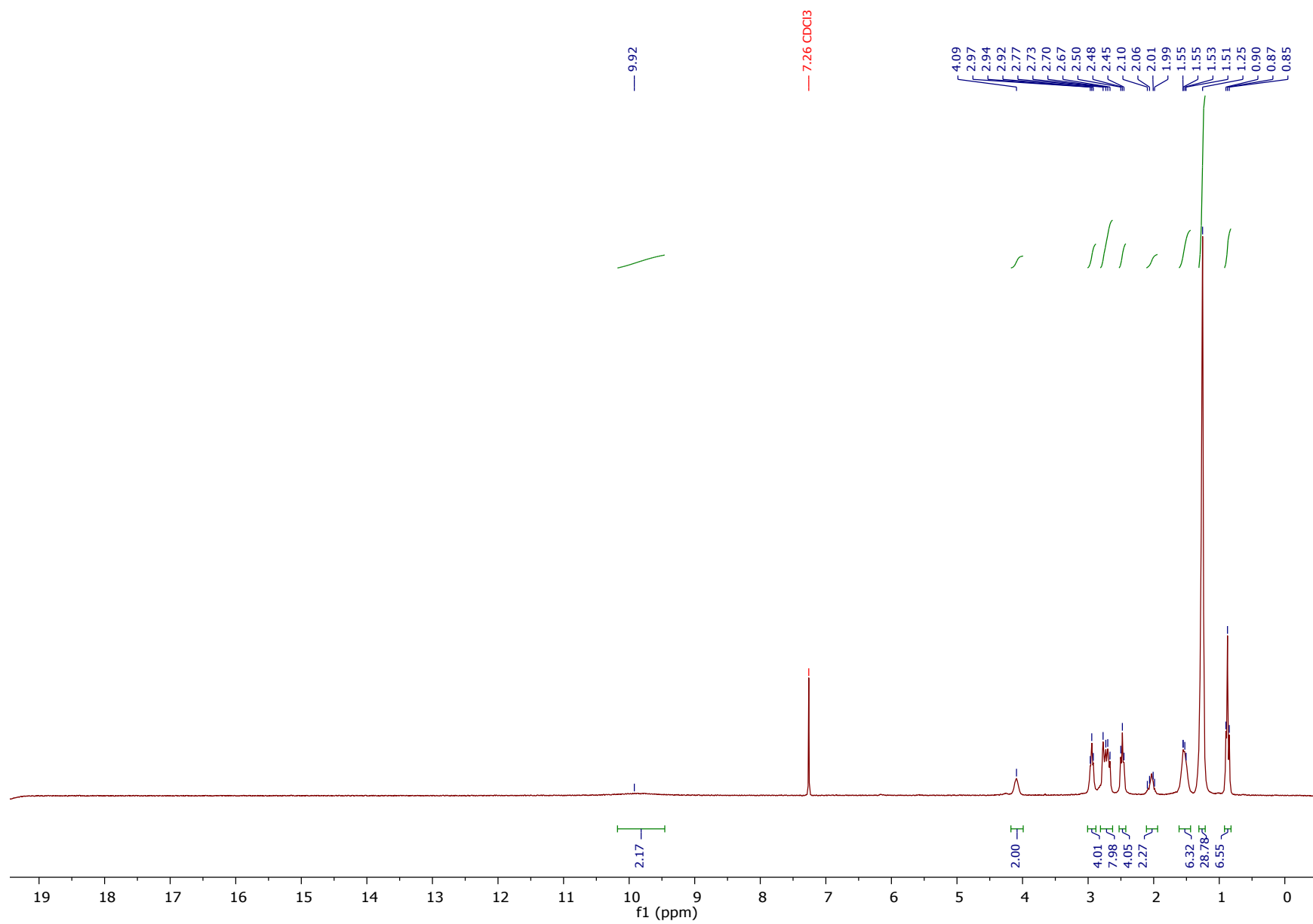
Yield: 48%, colourless gum.

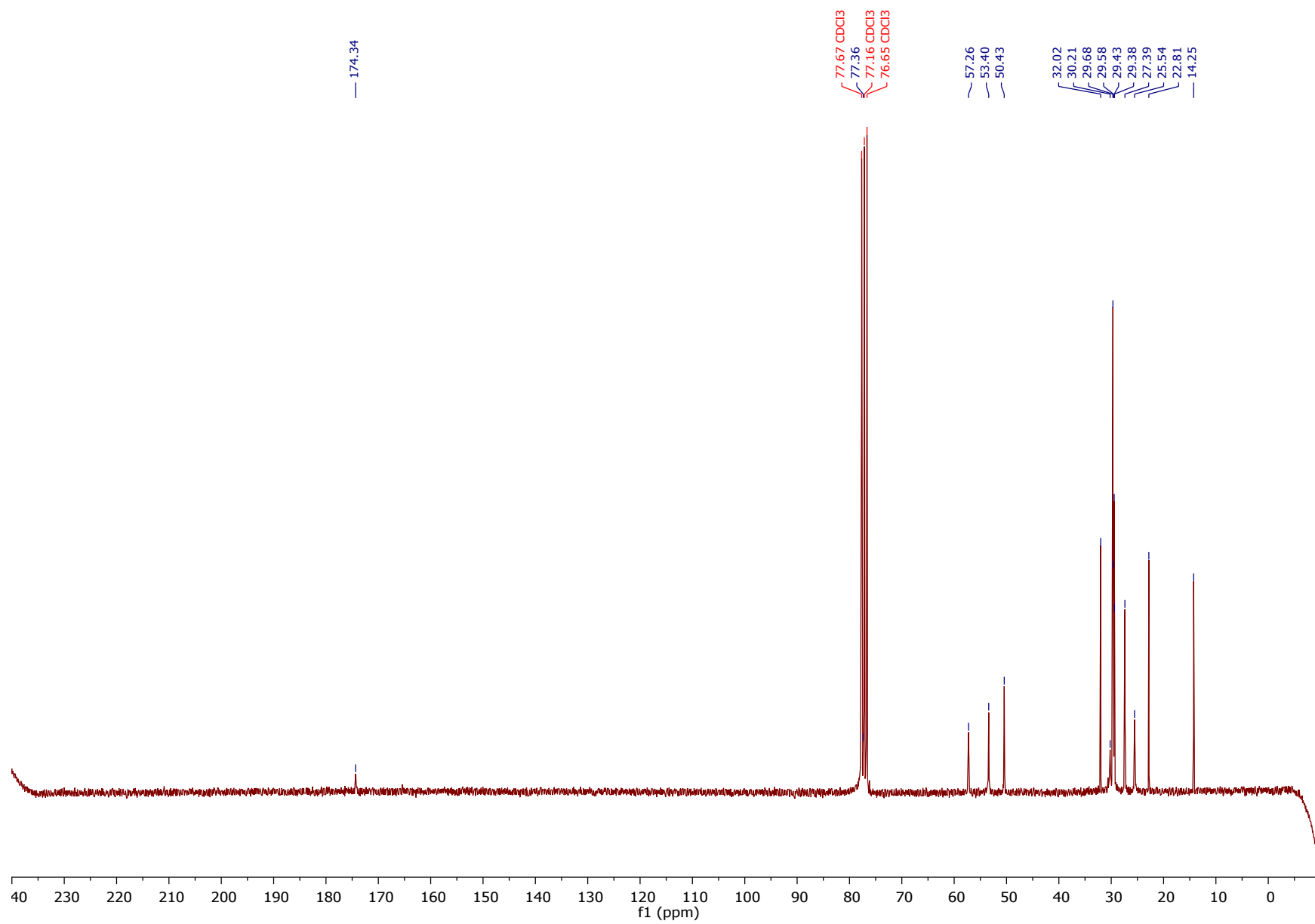
NMR ¹H (250 MHz, CDCl₃) δ: 9.95 (s, 2H), 4.19 – 4.03 (m, 2H), 2.94 (t, *J* = 6 Hz, 4H), 2.66-2.84 (m, 8H), 2.48 (t, *J* = 6 Hz, 4H), 2.16 – 1.98 (m, 2H), 1.66 – 1.45 (m, 6H), 1.25 (m, 28H), 0.90 (t, *J* = 7 Hz, 6H).

NMR ¹³C (63 MHz, CDCl₃) δ: 174.3, 77.4, 57.3, 53.4, 50.4, 32.0, 30.2, 29.7, 29.6, 29.4, 29.4, 27.4, 25.5, 22.8, 14.3.

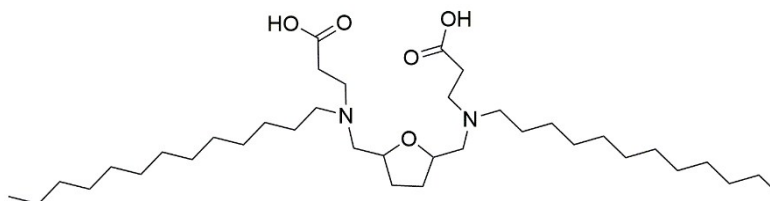
IR (cm⁻¹): 3397, 2955, 2925, 2807, 2495, 1956, 1710, 1597, 1463, 1398, 1308, 1208, 1070, 906, 805, 721, 665.

HRMS (ESI-TOF): *m/z*: [M + H]⁺ Calcd for C₃₂H₆₃N₂O₅ 555.4731; Found 555.4740





3,3'-(((tetrahydrofuan-2,5-diyl)bis(methylene))bis(dodecylazenediyl))dipropionic acid (4d) :



The compound **4d** is synthesised using dodecylamine.

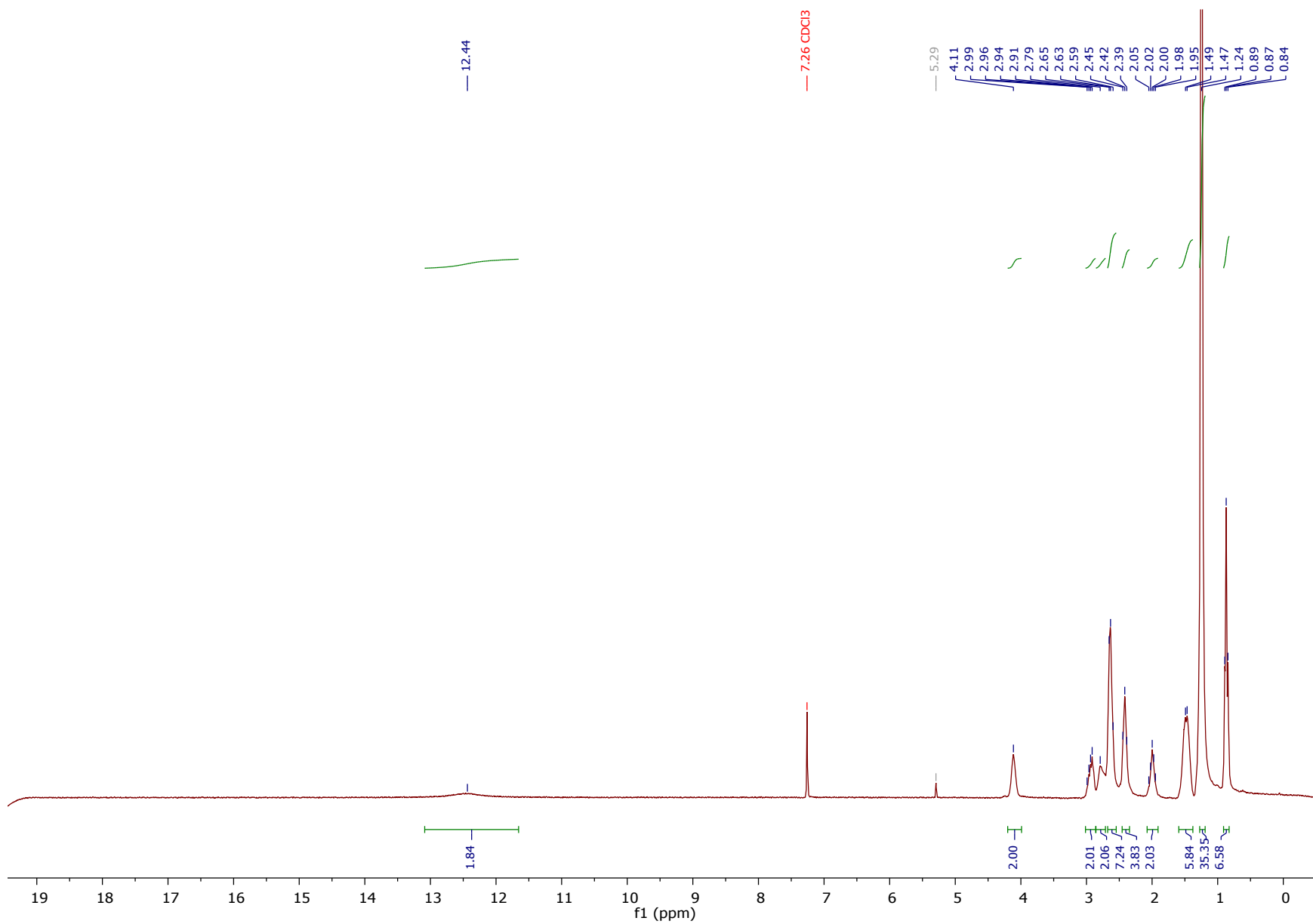
Yield: 55%, colourless gum.

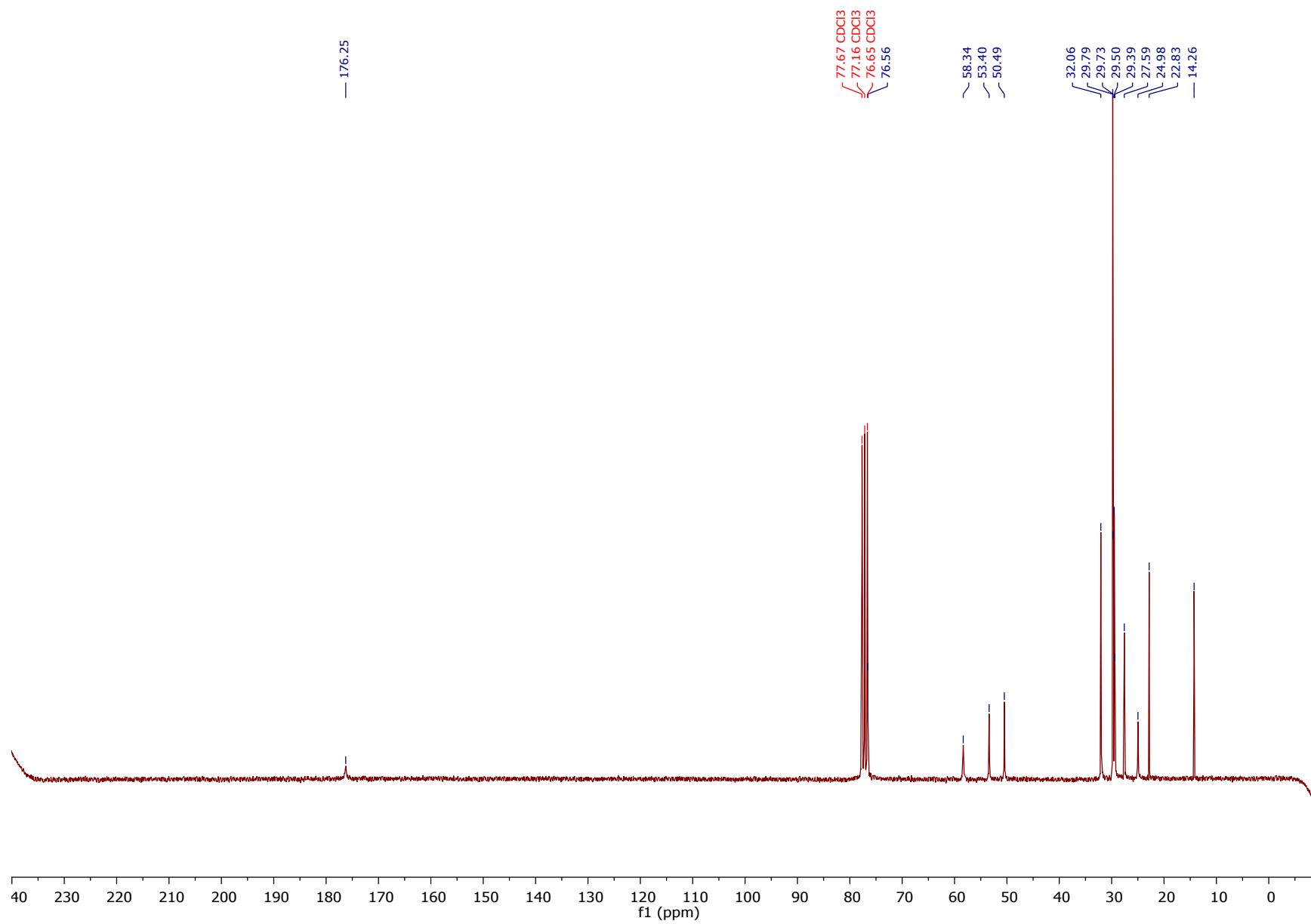
NMR ^1H (250 MHz, CDCl_3) δ : 12.44 (s, 2H), 4.22 – 4.08 (m, 2H), 3.04 – 2.89 (m, 2H), 2.88 – 2.75 (m, 2H), 2.74 – 2.52 (m, 8H), 2.50 – 2.37 (m, 4H), 2.12-1.91 (m, 2H), 1.63 – 1.41 (m, 6H), 1.38 – 1.18 (m, 34H), 0.96 (t, $J = 6,5$ Hz, 6H).

NMR ^{13}C (63 MHz, CDCl_3) δ : 176.3, 76.6, 58.3, 53.4, 50.5, 32.1, 29.8, 29.7, 29.5, 29.4, 27.6, 25.0, 22.8, 14.3.

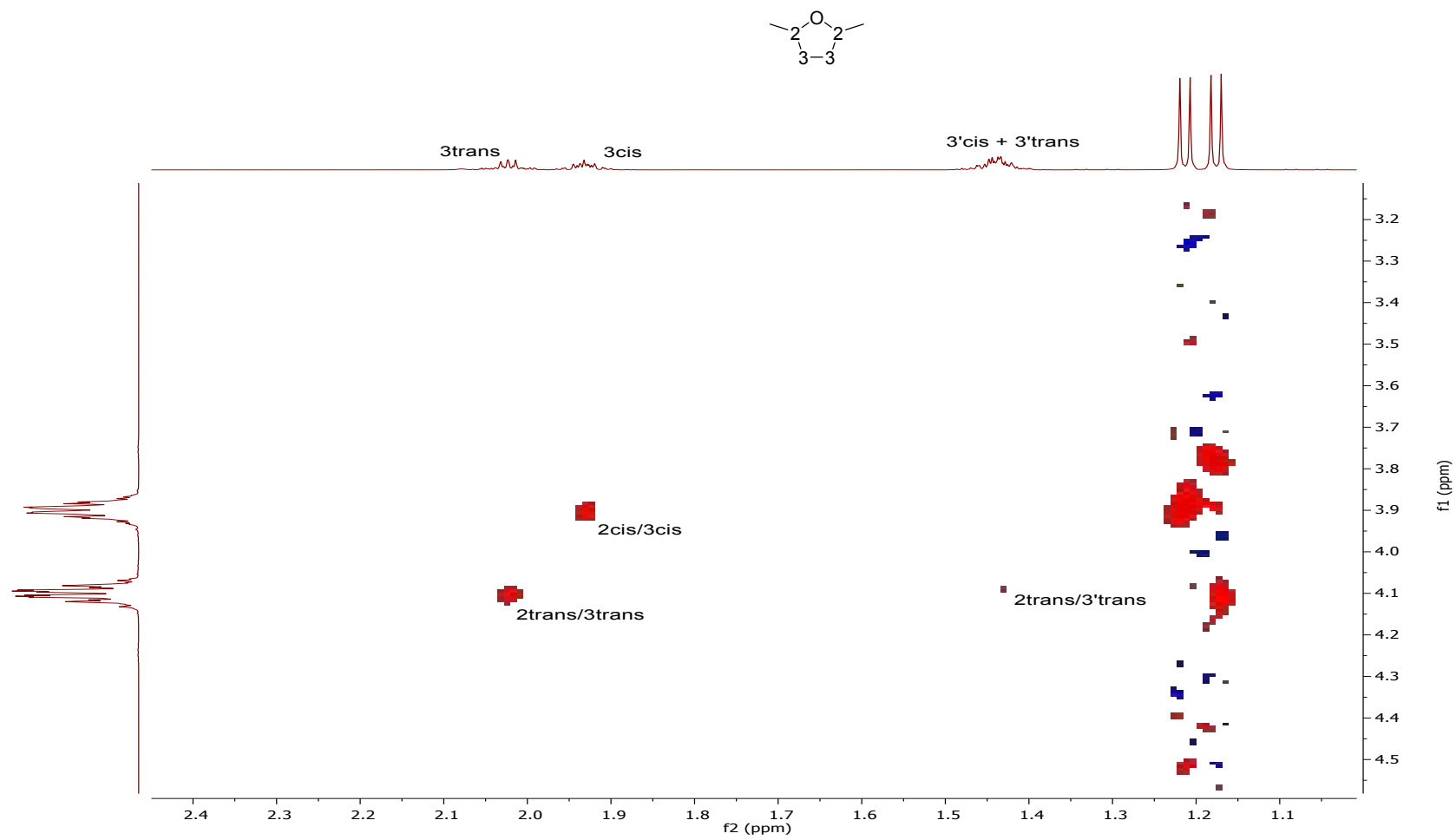
IR (cm^{-1}): 3410, 2959, 2935, 2871, 2488, 1962, 1713, 1597, 1463, 1382, 1302, 1242, 1206, 1074, 948, 906, 868, 805, 754, 663.

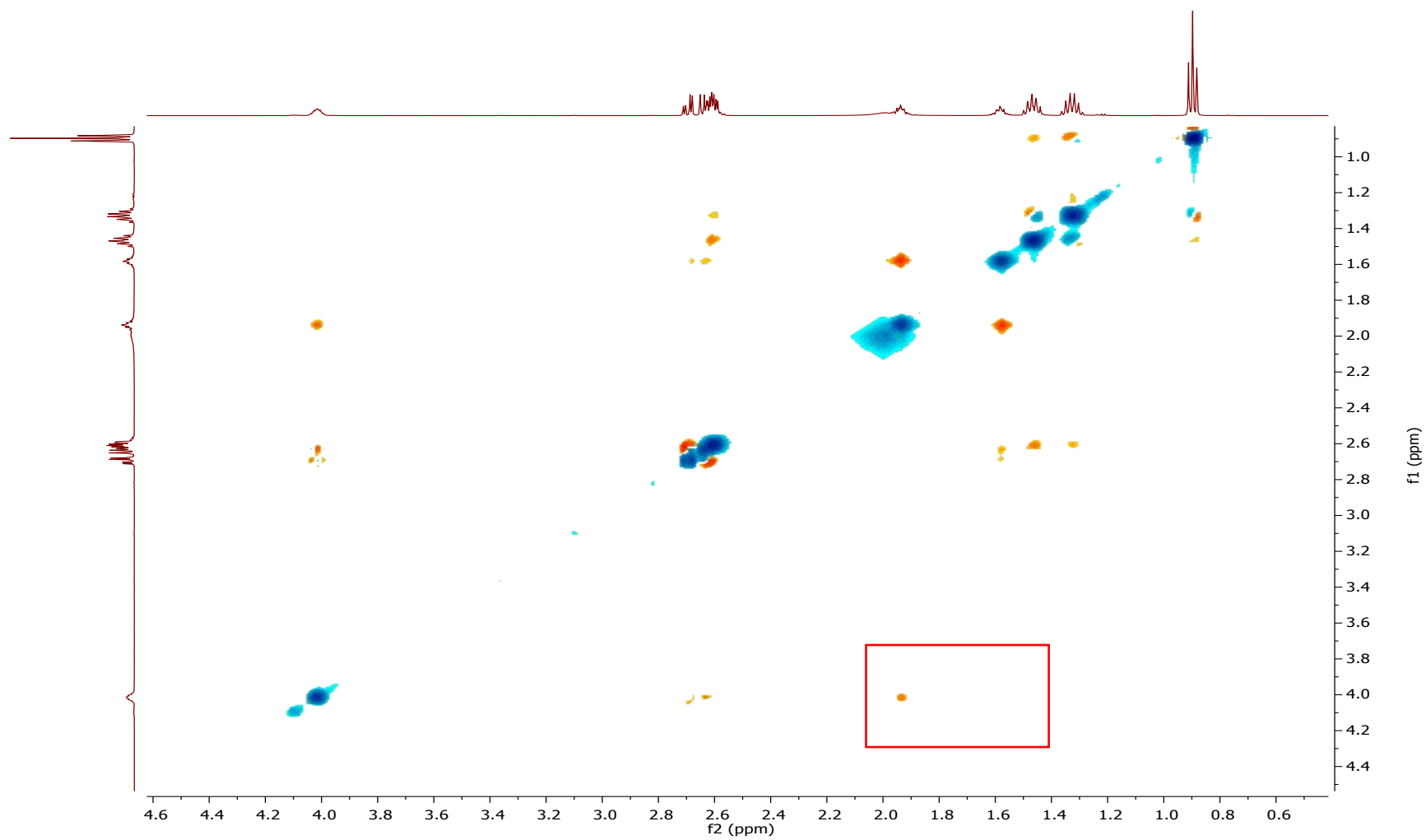
HRMS (ESI-TOF): m/z: $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{36}\text{H}_{70}\text{N}_2\text{O}_5\text{Na}$ 633.5177; Found 633.5182





NOE spectra of cis/trans 2,5-dimethylfuran (zoom)



NOE spectra of compound **2a**

Determination of surface tensions and critical micellar concentrations

Surface tensions (γ) of aqueous surfactant solutions were recorded by a Wilhelmy-type surface balance (Krüss K100MK2) equipped with a dosimeter (700 dosino, Metrohm). CMC values were determined by the cross-section of both extreme tangential straight lines to the curves and adjusted if a clear break of γ -logC (C is mass concentration) curves appeared. Measurements were conducted at 25 °C and pH was specified in figures.

Determination of wetting power

Wetting power was investigated following the Draves test (AFNOR NTF 73-406 standard, 1 g L⁻¹ at 25 °C). Wetting power is defined as the required time to observe a cotton sink when this latter is immersed in a surfactant solution. The given value is average value obtained after three replicates.

Determination of antifungal properties

The antifungal activity was investigated against *Fusarium graminearum*. The tests were carried out by insemination of 30 microliters of a solution containing *F. graminearum* (2.10⁴spores/mL) mixed with the desired test concentration of gemini surfactant in Petri dishes containing PDA. The plates were sealed and incubated in the dark at 22°C. Control samples with *F. Graminearum* in PDA and distilled water were subjected to the same treatments. The effectiveness of the treatments was evaluated by measuring the average diametric growth of the colonies after 96 hours after the inoculum. The percentage of inhibition was calculated according to Wang et al.²

Biodegradation of DFF:

Biodegradation of DFF has been determined following the OECD 301F standard.³

Product	Latency (< 10%)	% biodegradation at 10 days after attained 10%	% biodegradation after 28 days
commercial DFF ⁴	11 days	not easy biodegradable 35 ± 32 %	easy biodegradable 66 ± 4 %
synthesized DFF ⁵	11 days	easy biodegradable 62 ± 5 %	easy biodegradable 66.5 ± 4 %

² Wang, S.Y., P.F. Chen, S.T. Chang, *Bioresour. Technol.* **2005**, *96*, 813-818.

³ OECD Test Guideline 301F, 1992, <http://www.oecd.org/dataoecd/17/16/1948209.pdf>

⁴ Purchased from Sigma Aldrich

⁵ a) C. Laugel, B. Estrine, J. Le Bras, N. Hoffmann, S. Marinkovic, J. Muzart, *ChemCatChem.* 2014, **6**, 1195-1198.

b) Q. Girka, B. Estrine, N. Hoffmann J. Le Bras, S. Marinković, J. Muzart, *React. Chem. Eng.*, **2016**, *1*, 176-182.