Electronic supplementary information for the manuscript

"Facile preparation of amine and amino acid adducts of [60]fullerene using chlorofullerene $C_{60}Cl_6$ as a precursor"

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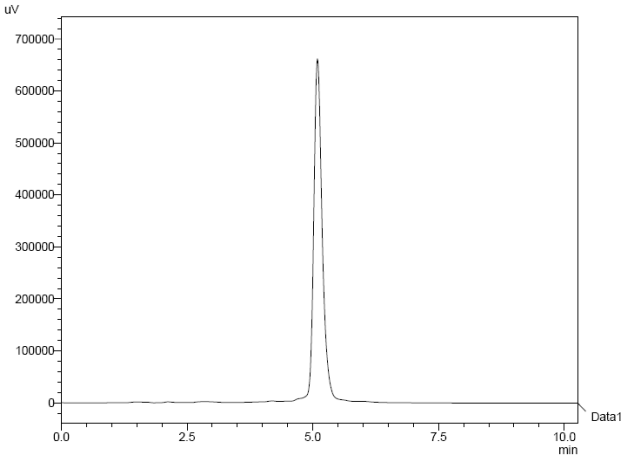

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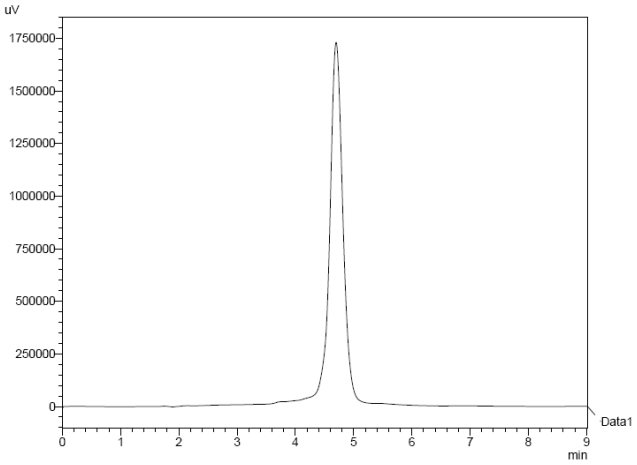

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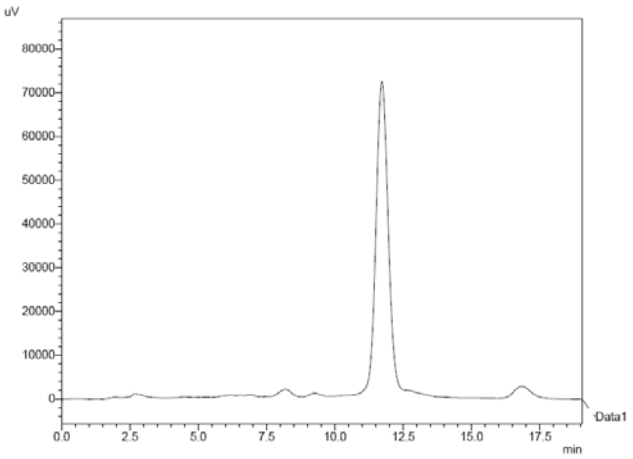


Fig. S1. HPLC profiles of compounds **1a** (a), **1b** (b) and **1f** (c) (Phenomenex Luna 5u C18(2) column, 150 x 4.6 mm, methanol/toluene 70/30 v/v, flow rate 1 mL min^{-1}).

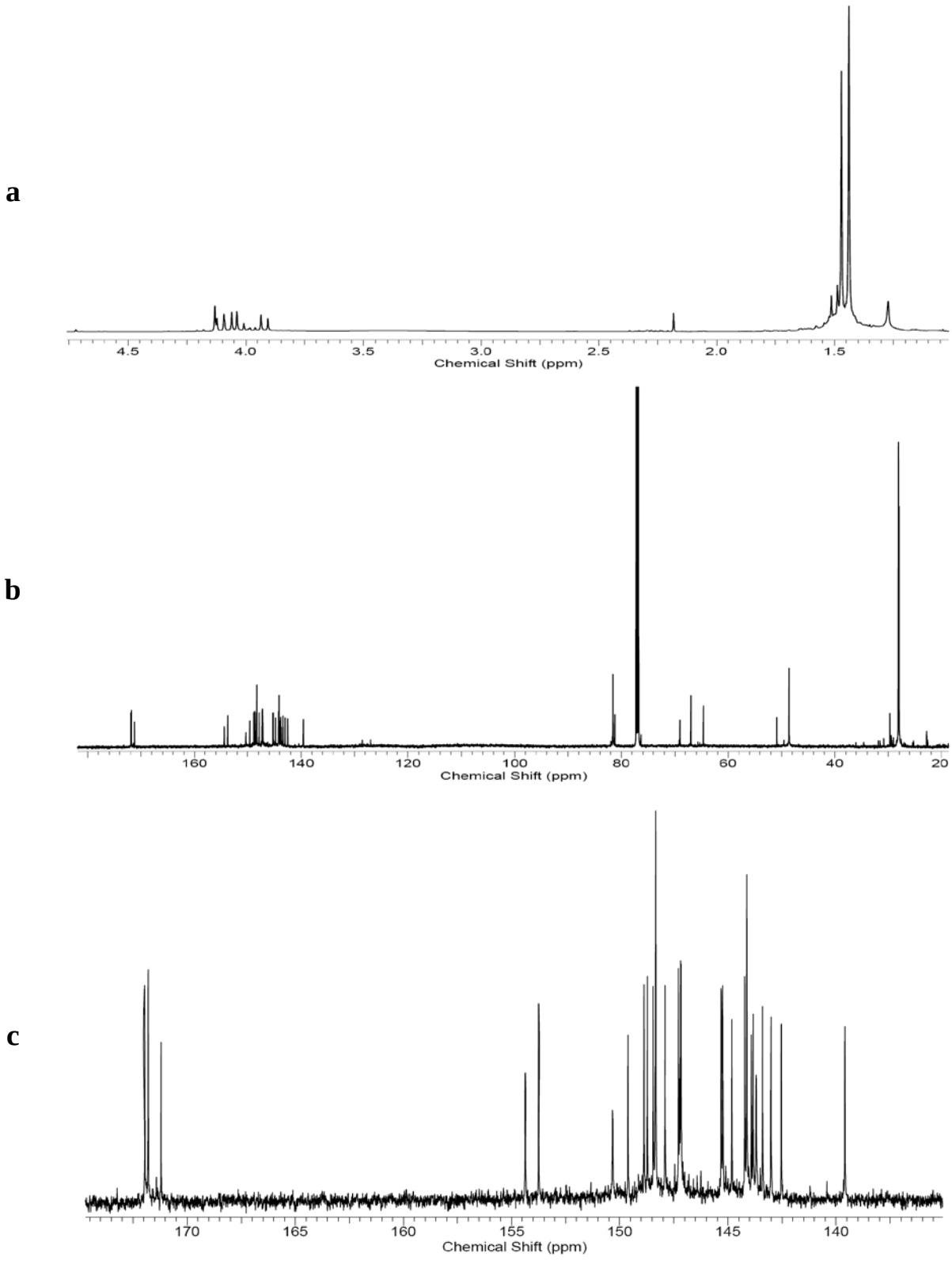


Fig. S2. ^{1}H (a), full ^{13}C (b) and low field ^{13}C (c) NMR spectra of **1a** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

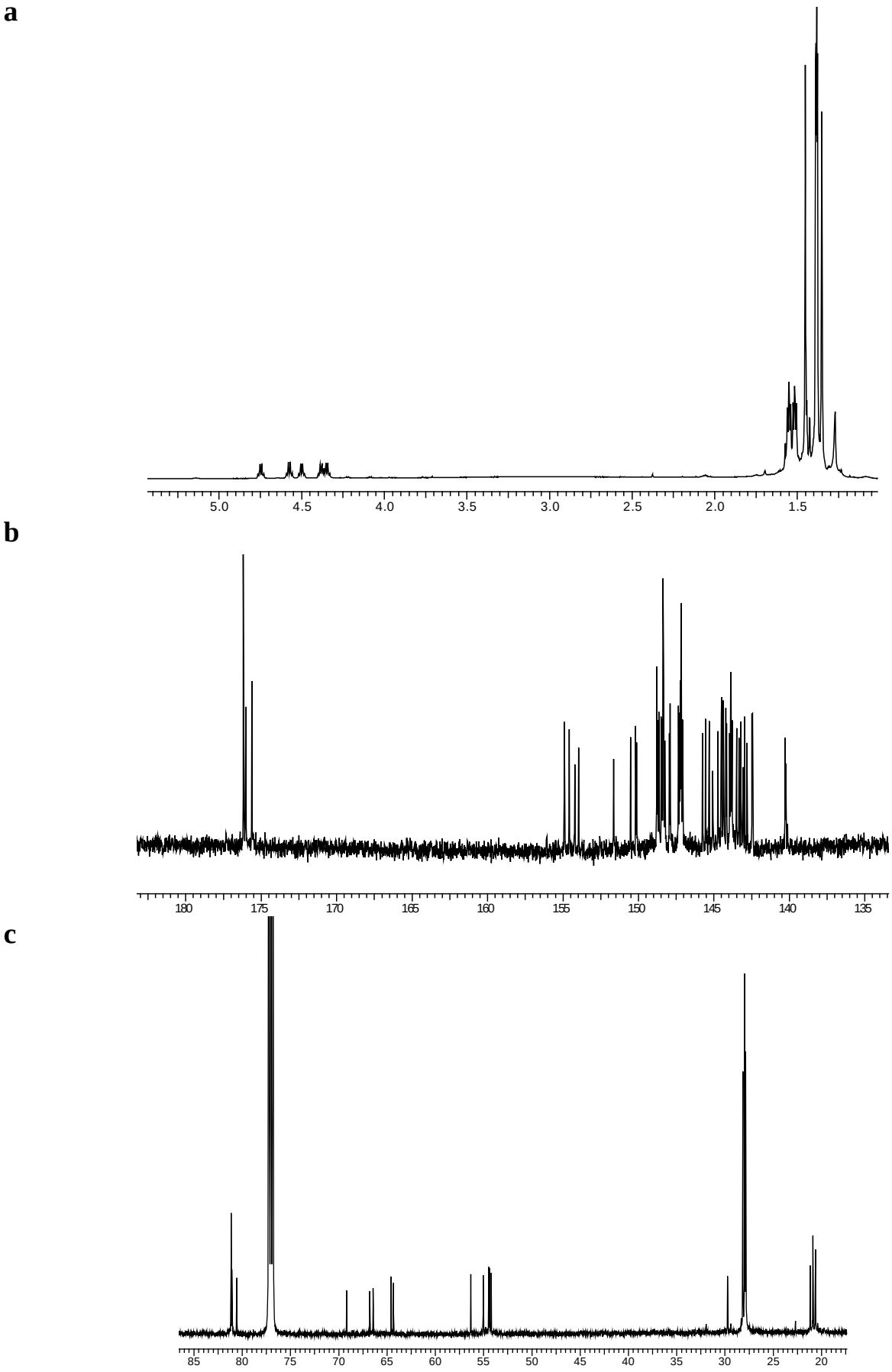


Fig. S3. ^{1}H (a), low field ^{13}C (b) and high field ^{13}C (c) NMR spectra of **1b** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

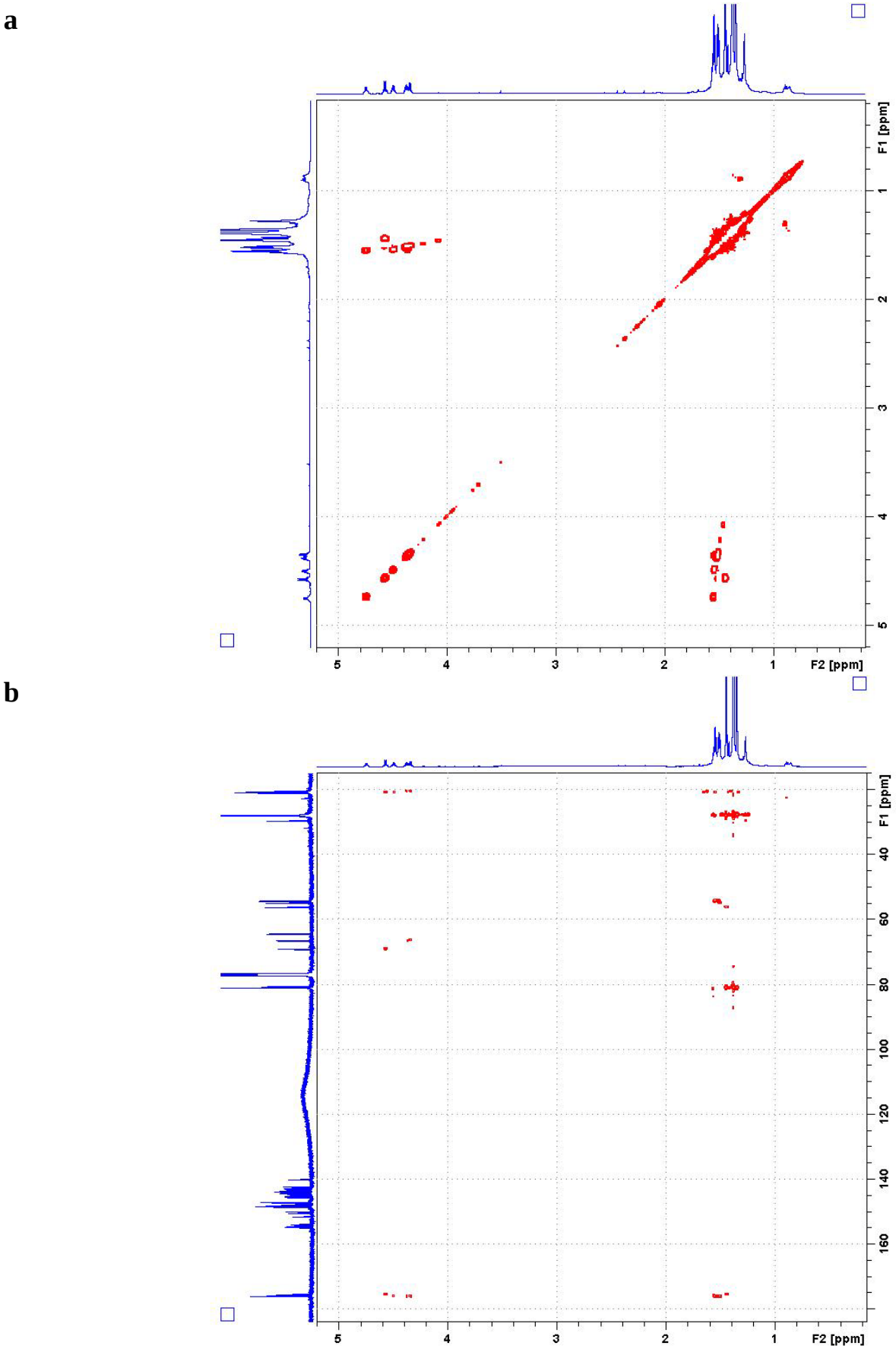
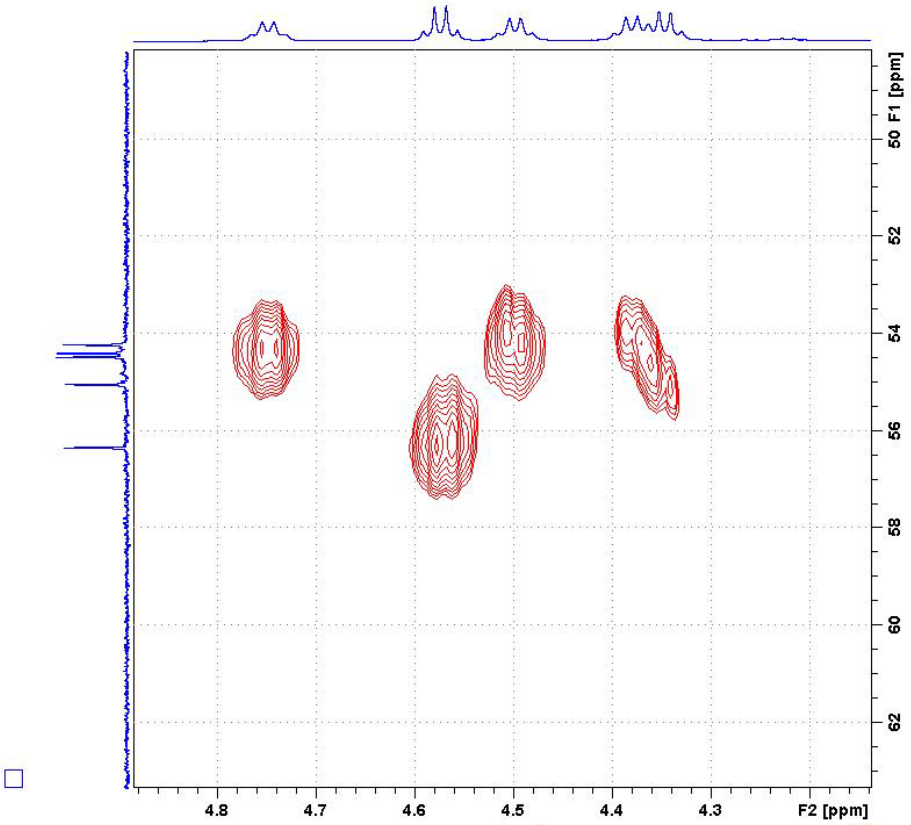


Fig. S4. H-H COSY (a) and H-C HMBC (b) NMR spectra of **1b**.

a



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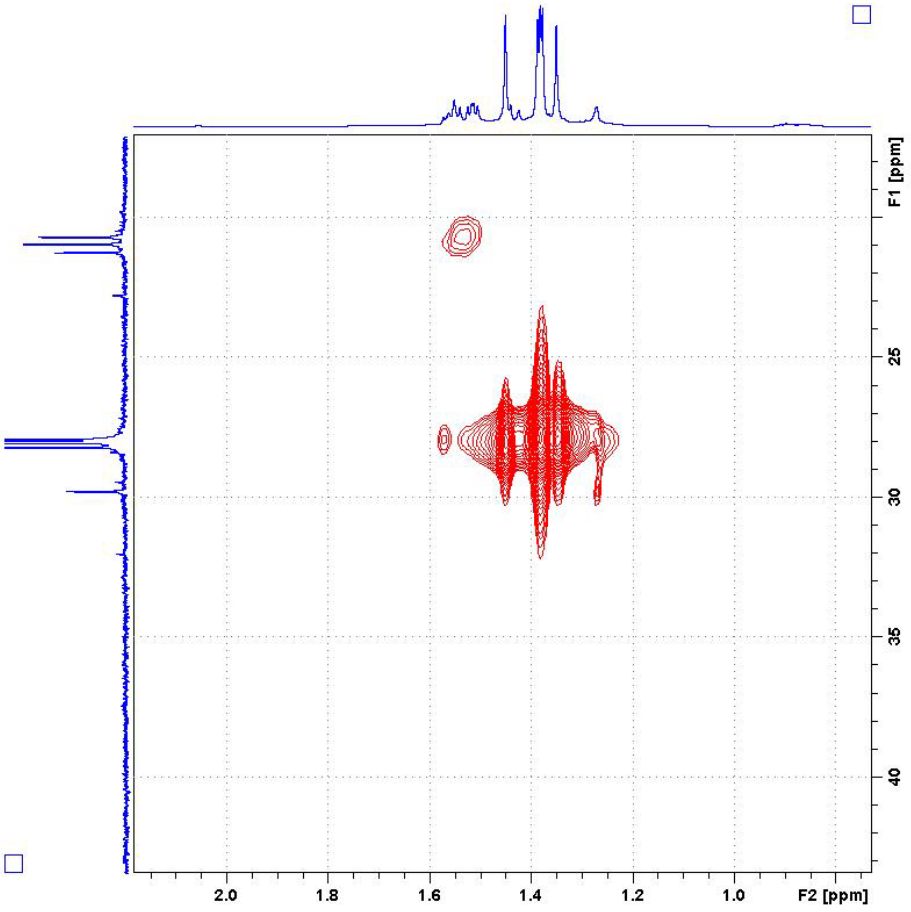


Fig. S5. Low-field H-C HMQC (a) and high-field H-C HMQC (b) NMR spectra of **1b**.

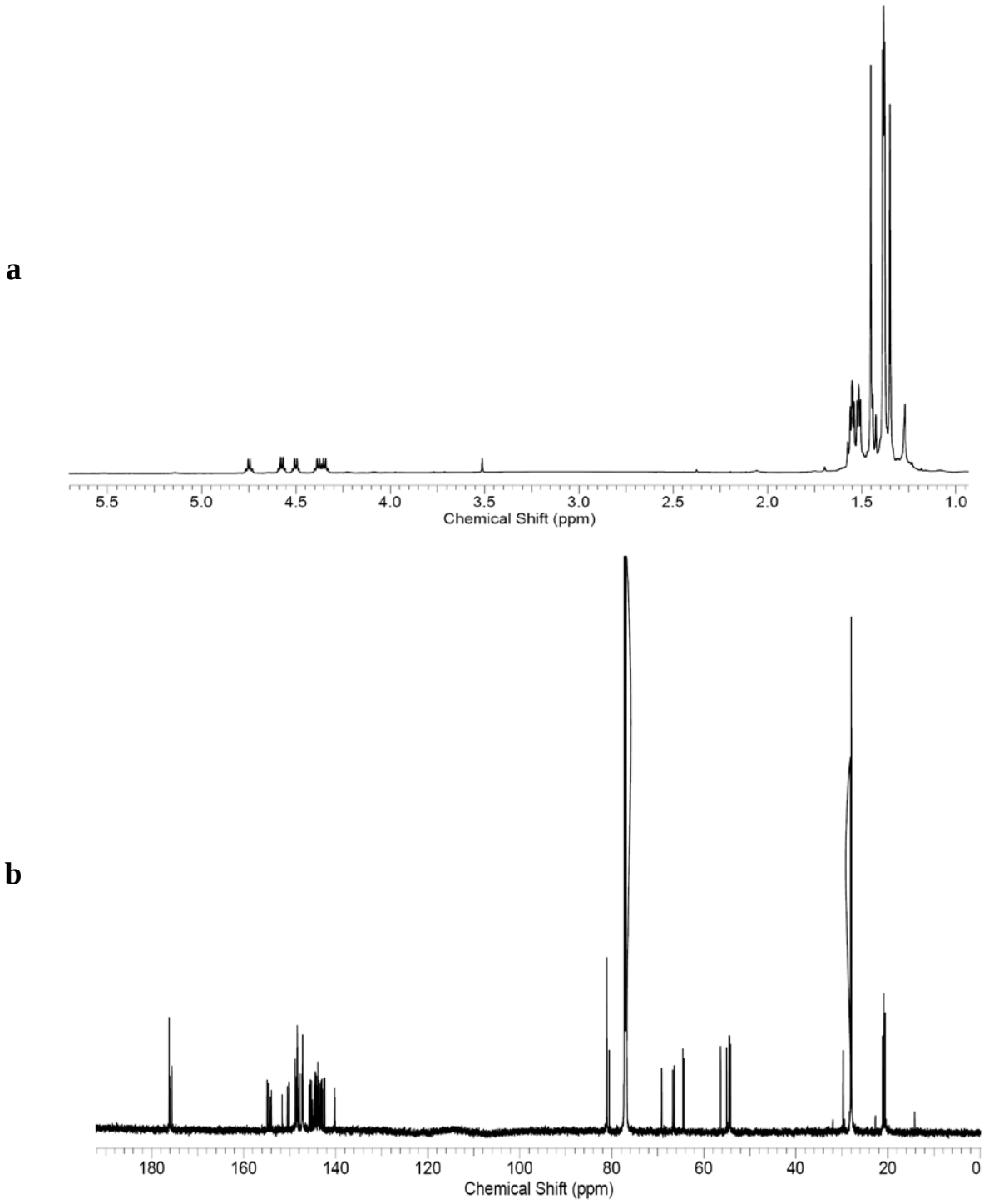


Fig. S6. ^{1}H (a) and full ^{13}C (b) NMR spectra of **1c** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

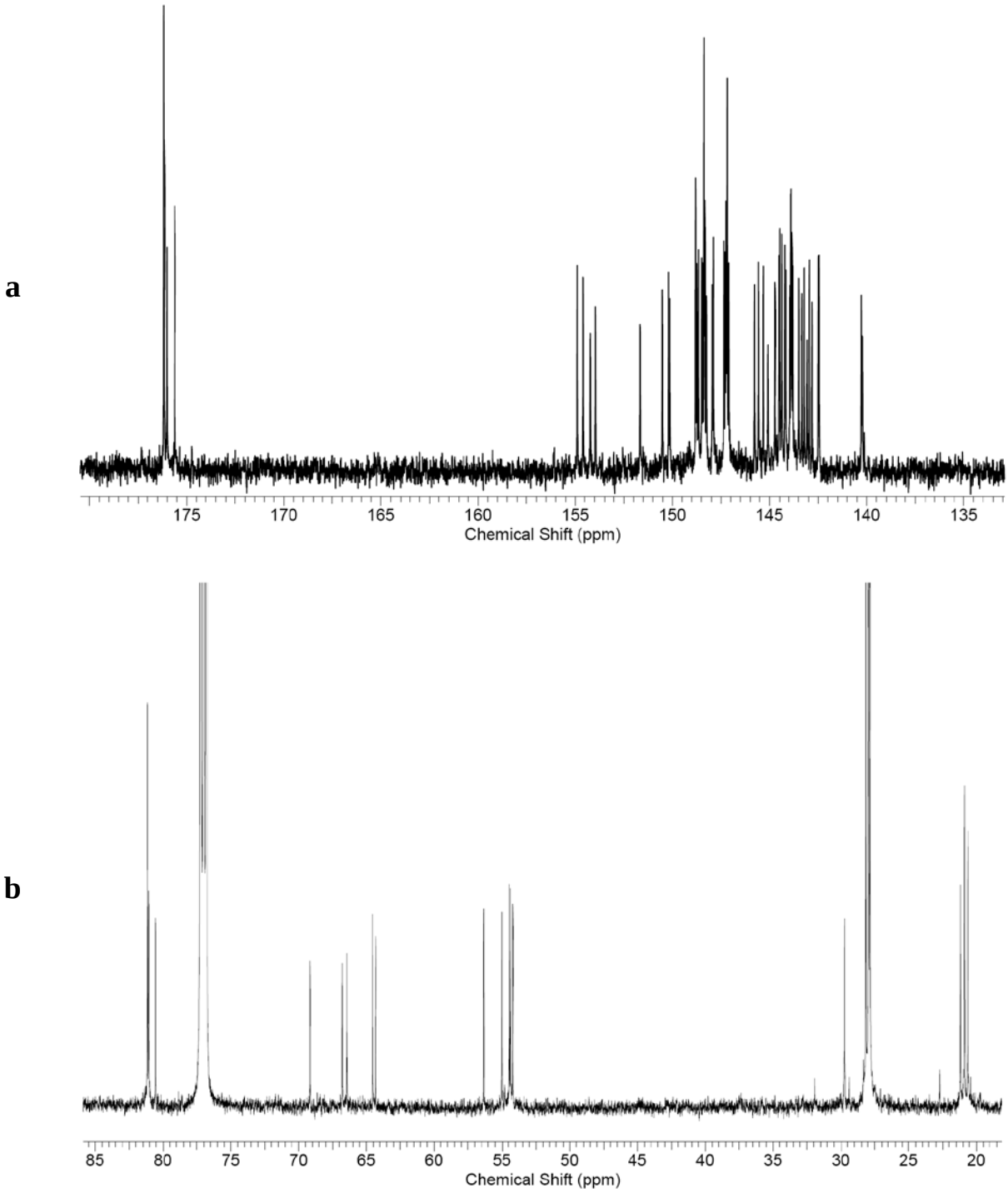


Fig. S7. Low field ^{13}C (a) and high field ^{13}C (b) NMR spectra of **1c** ($CDCl_3$, 150 MHz).

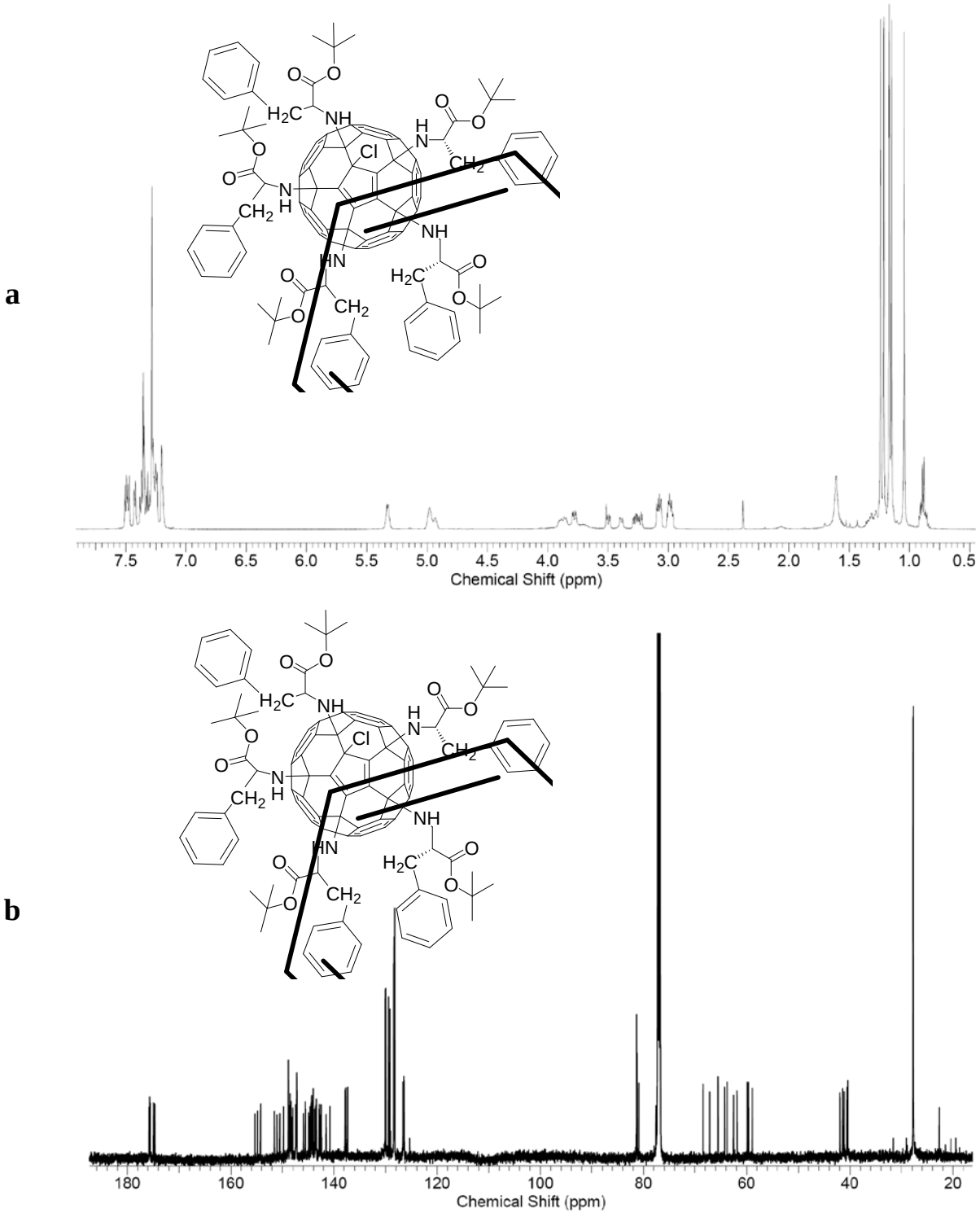


Fig. S8. ^{1}H (a) and full ^{13}C (b) NMR spectra of **1d** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

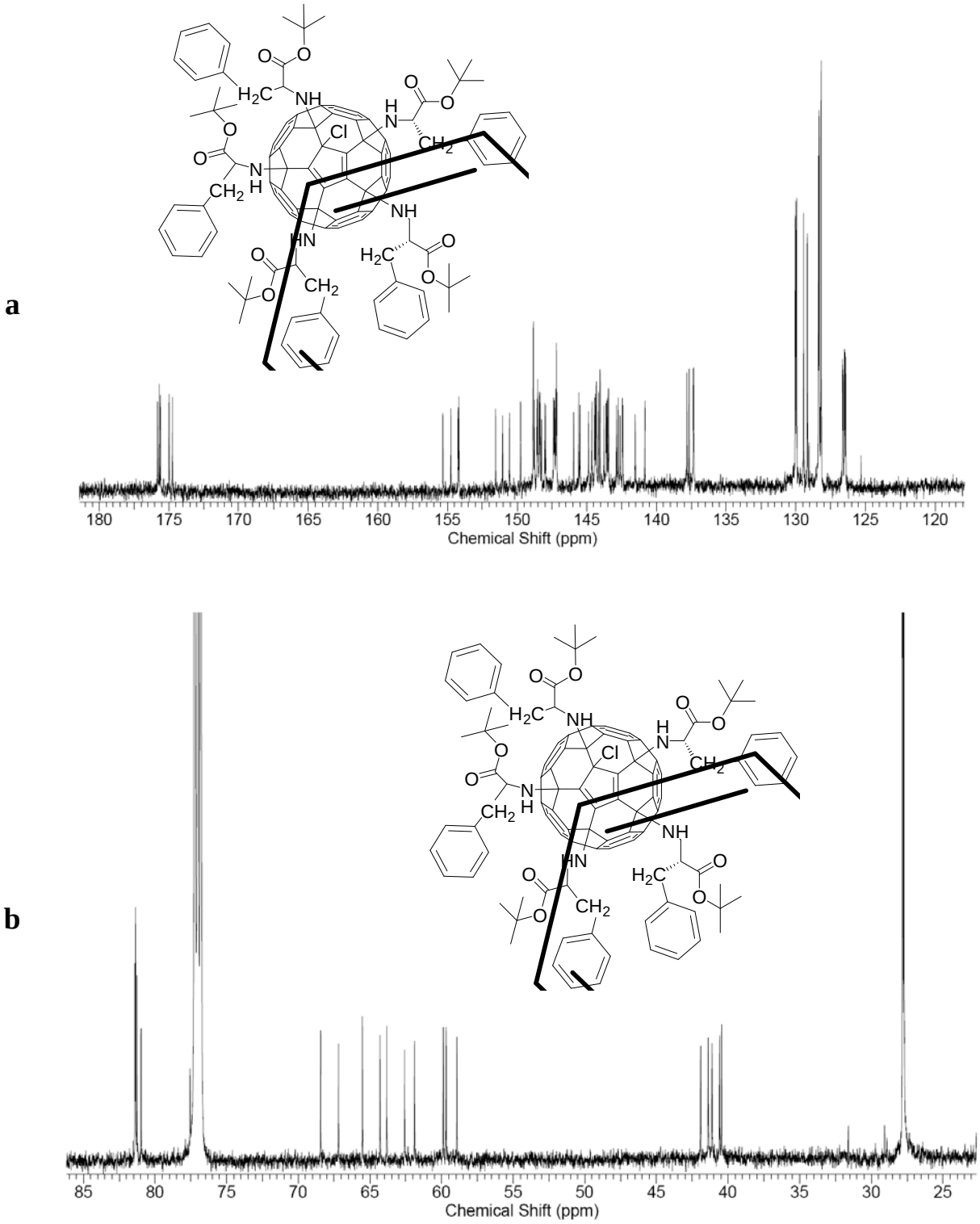
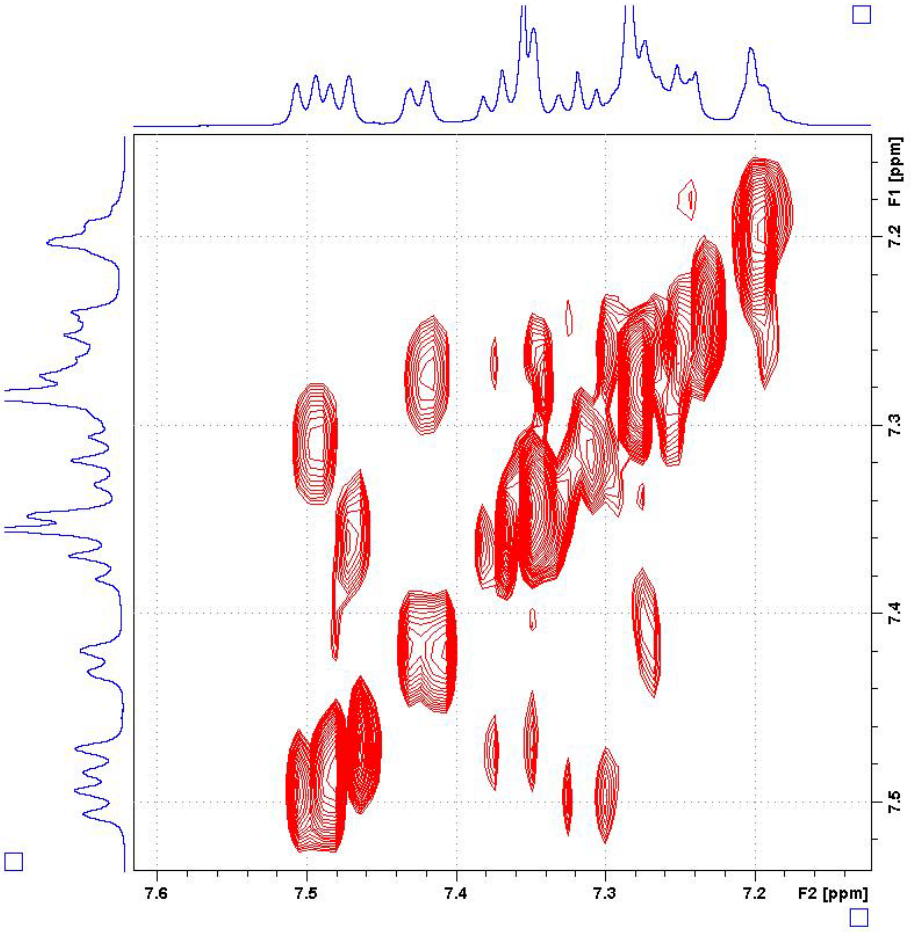


Fig. S9. Low field ^{13}C (a) and high field ^{13}C (b) NMR spectra of **1d** ($CDCl_3$, 150 MHz).

a



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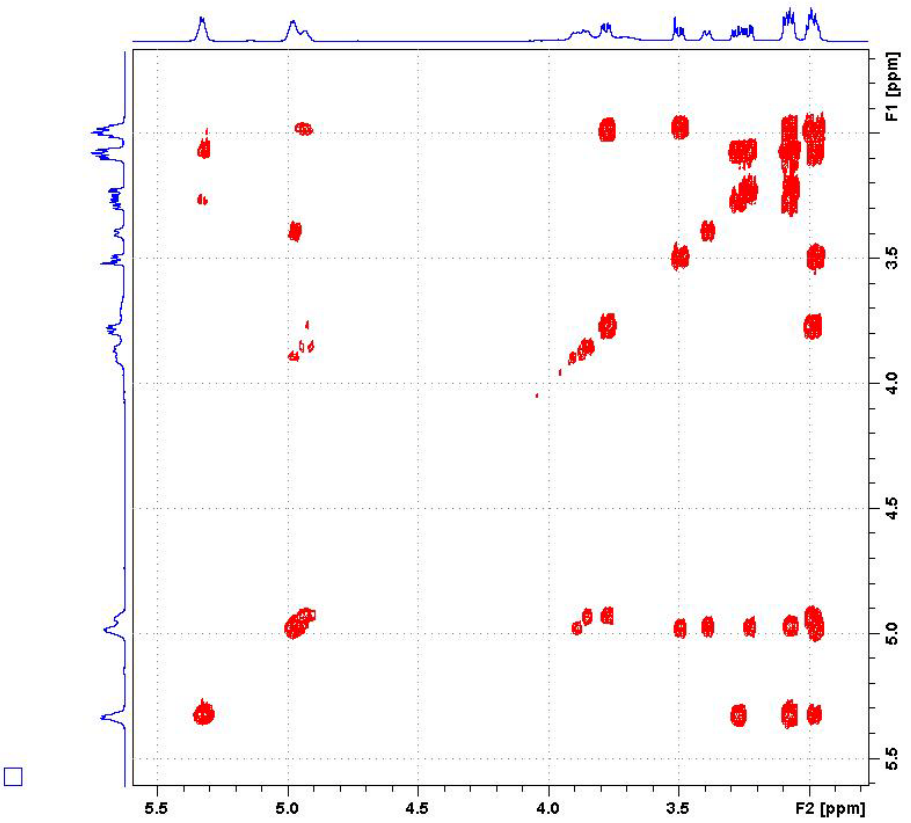
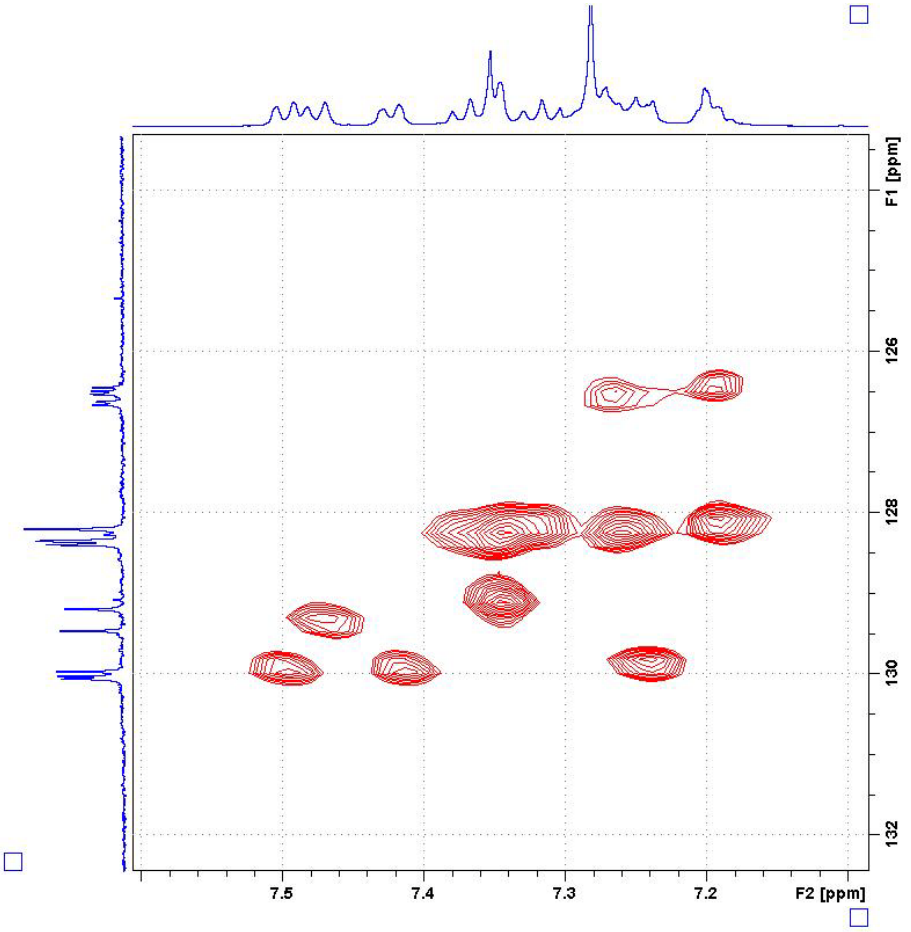


Fig. S10. Low-field H-H COSY (a) and high-field H-H COSY (b) NMR spectra of **1d**.

a



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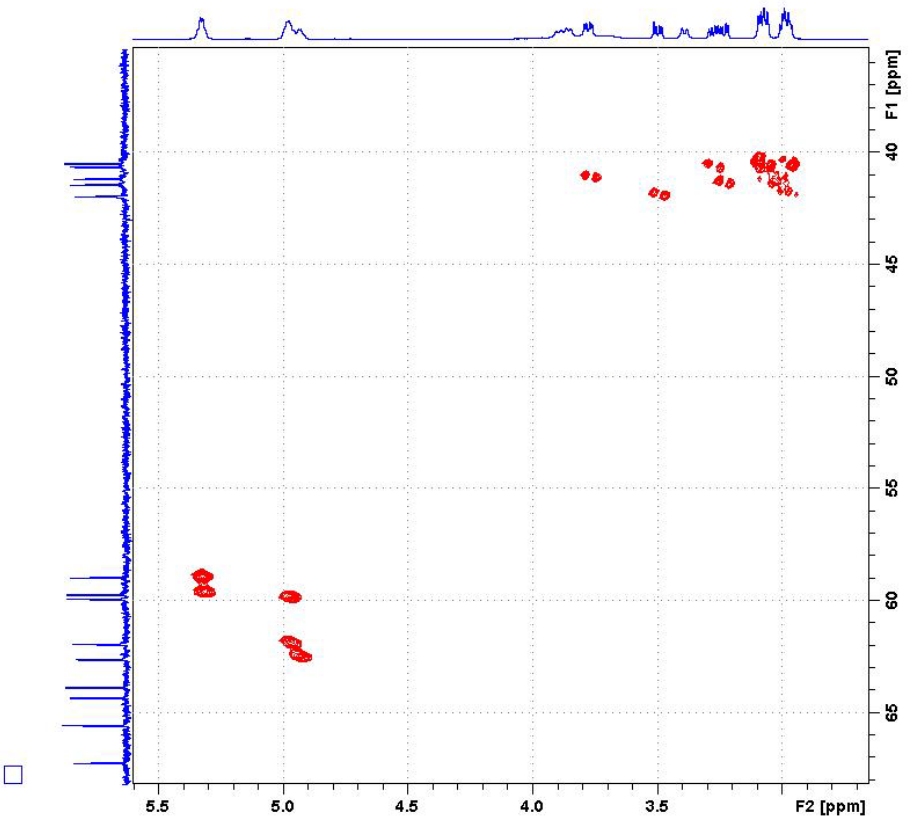


Fig. S11. Low-field H-C HMQC (a) and high-field H-C HMQC (b) NMR spectra of **1d**.

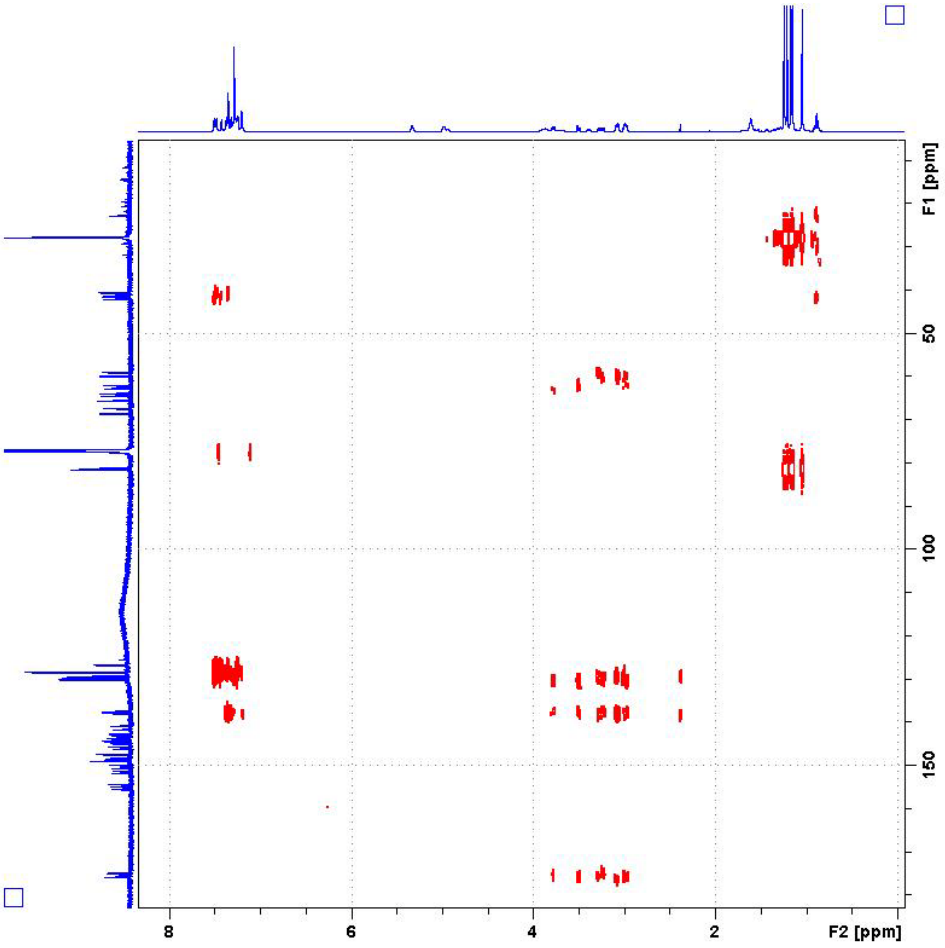


Fig. S12. H-C HMBC NMR spectrum of **1d**.

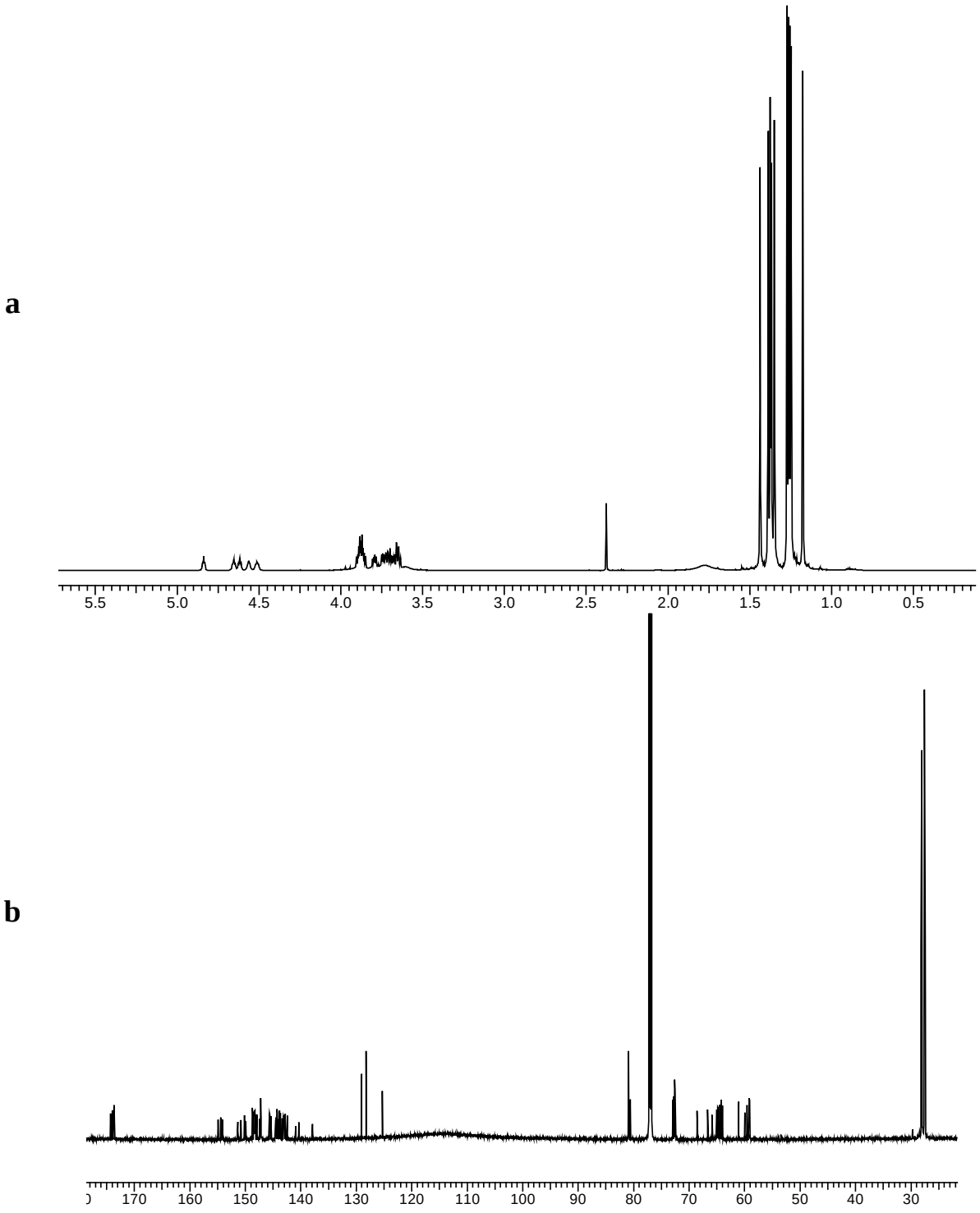


Fig. S13. ^{1}H (a) and full ^{13}C (b) NMR spectra of **1e** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

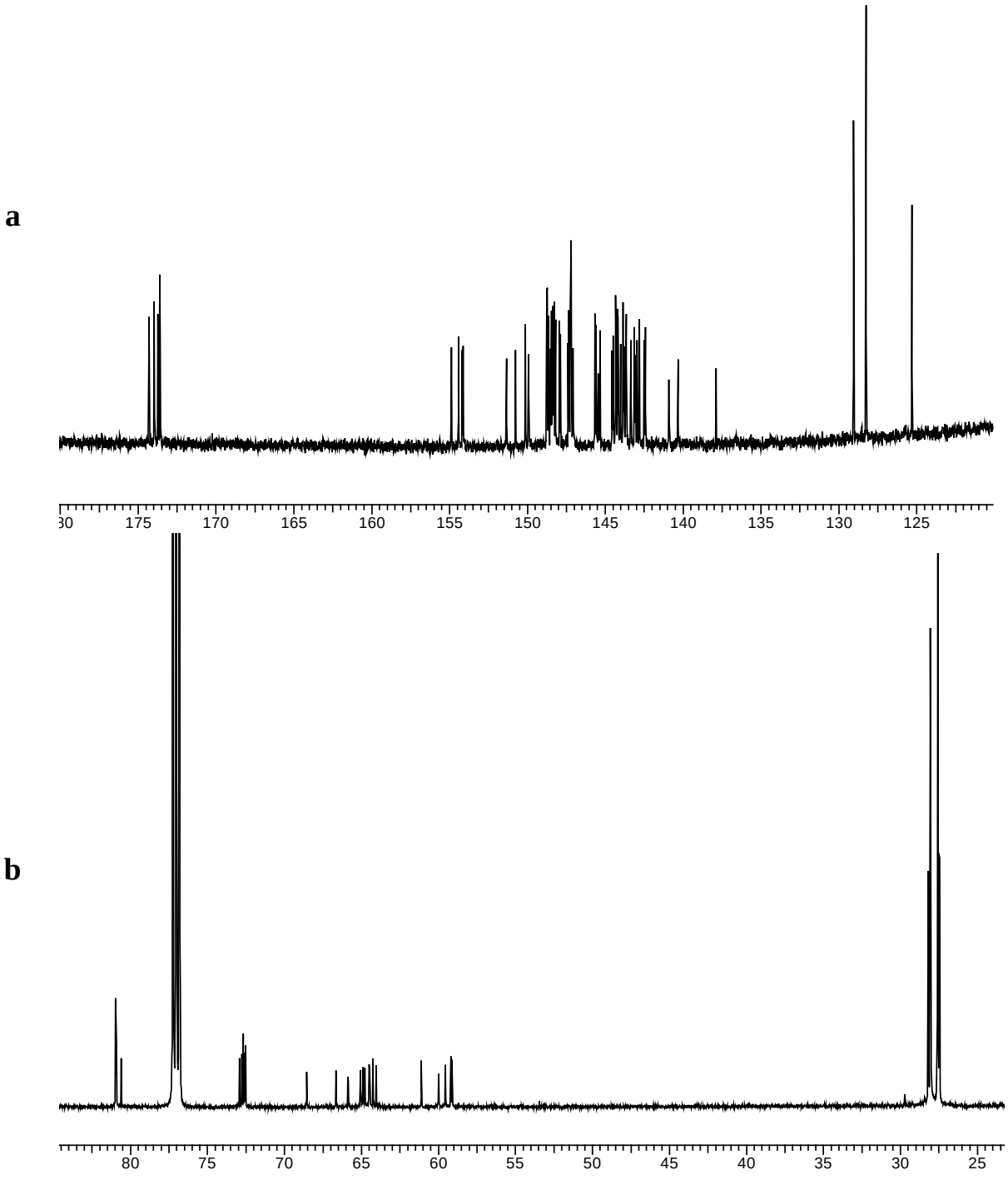


Fig. S14. Low field ^{13}C (a) and high field ^{13}C (b) NMR spectra of **1e** ($CDCl_3$, 150 MHz).

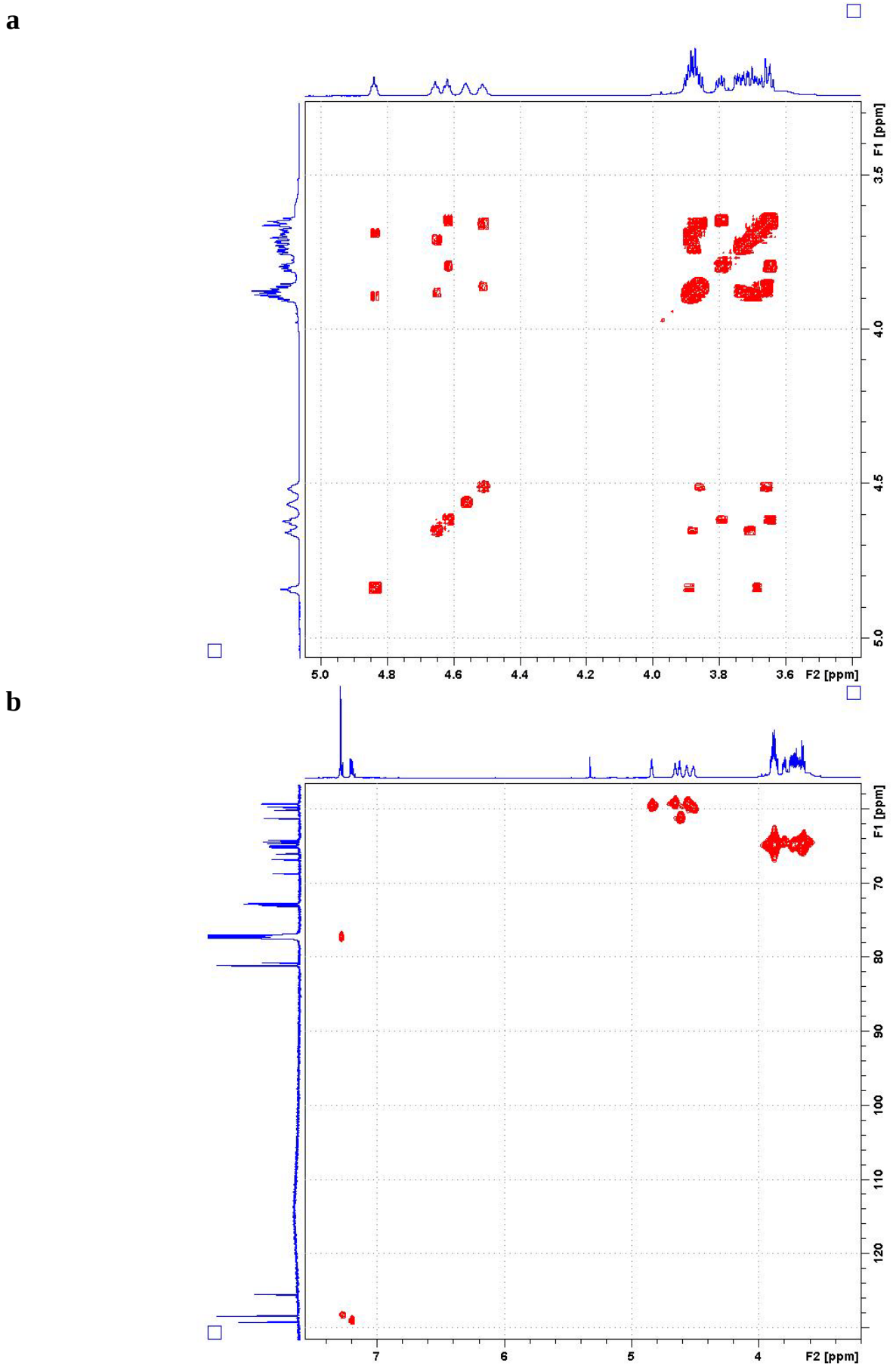


Fig. S15. H-H COSY (a) and H-C HMQC (b) NMR spectra of **1e**.

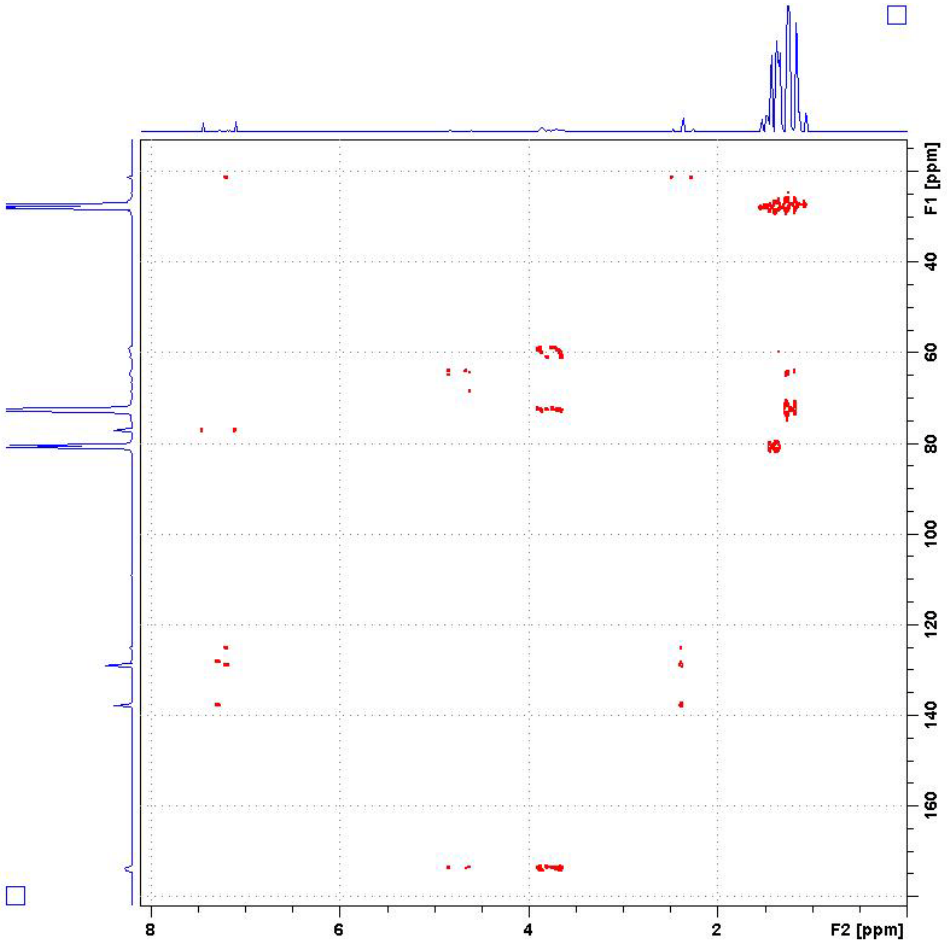


Fig. S16. H-C HMBC NMR spectrum of **1e**.

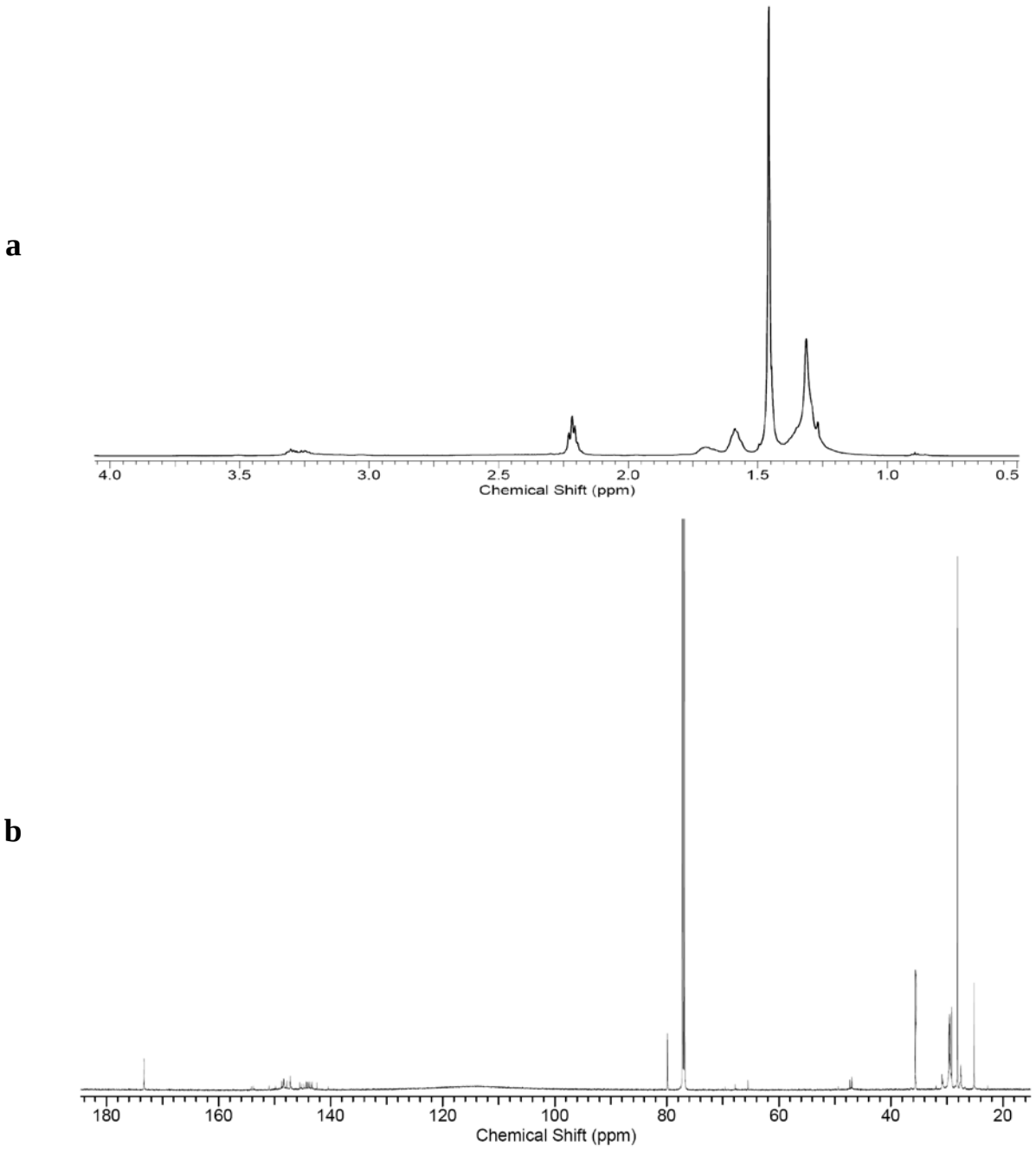


Fig. S17. ^{1}H (a) and full ^{13}C (b) NMR spectra of **1f** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

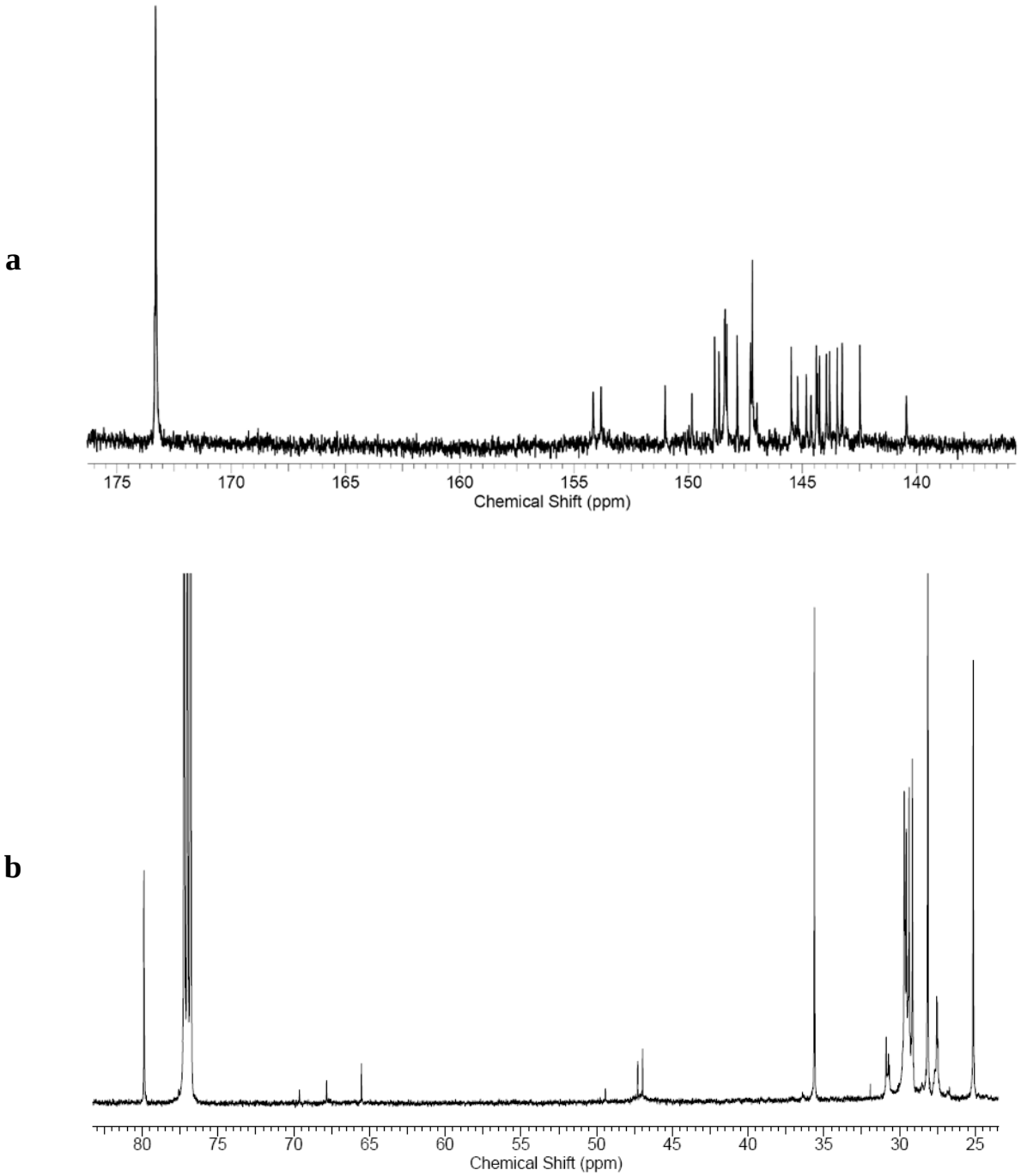


Fig. S18. Low field ^{13}C (a) and high field ^{13}C (b) NMR spectra of **1f** ($CDCl_3$, 150 MHz).

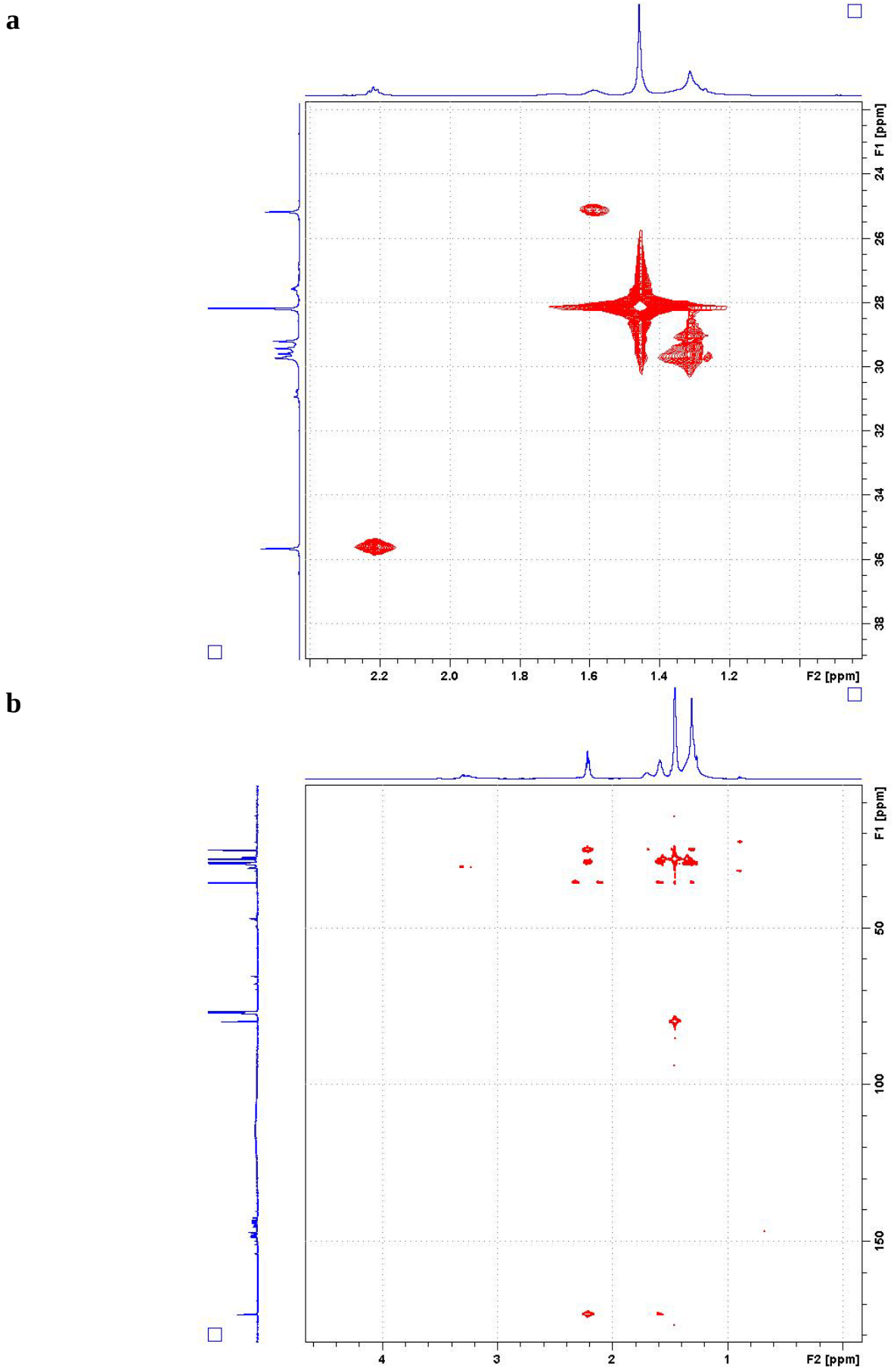


Fig. S19. H-C HMQC (a) and H-C HMBC (b) NMR spectra of **1f**.

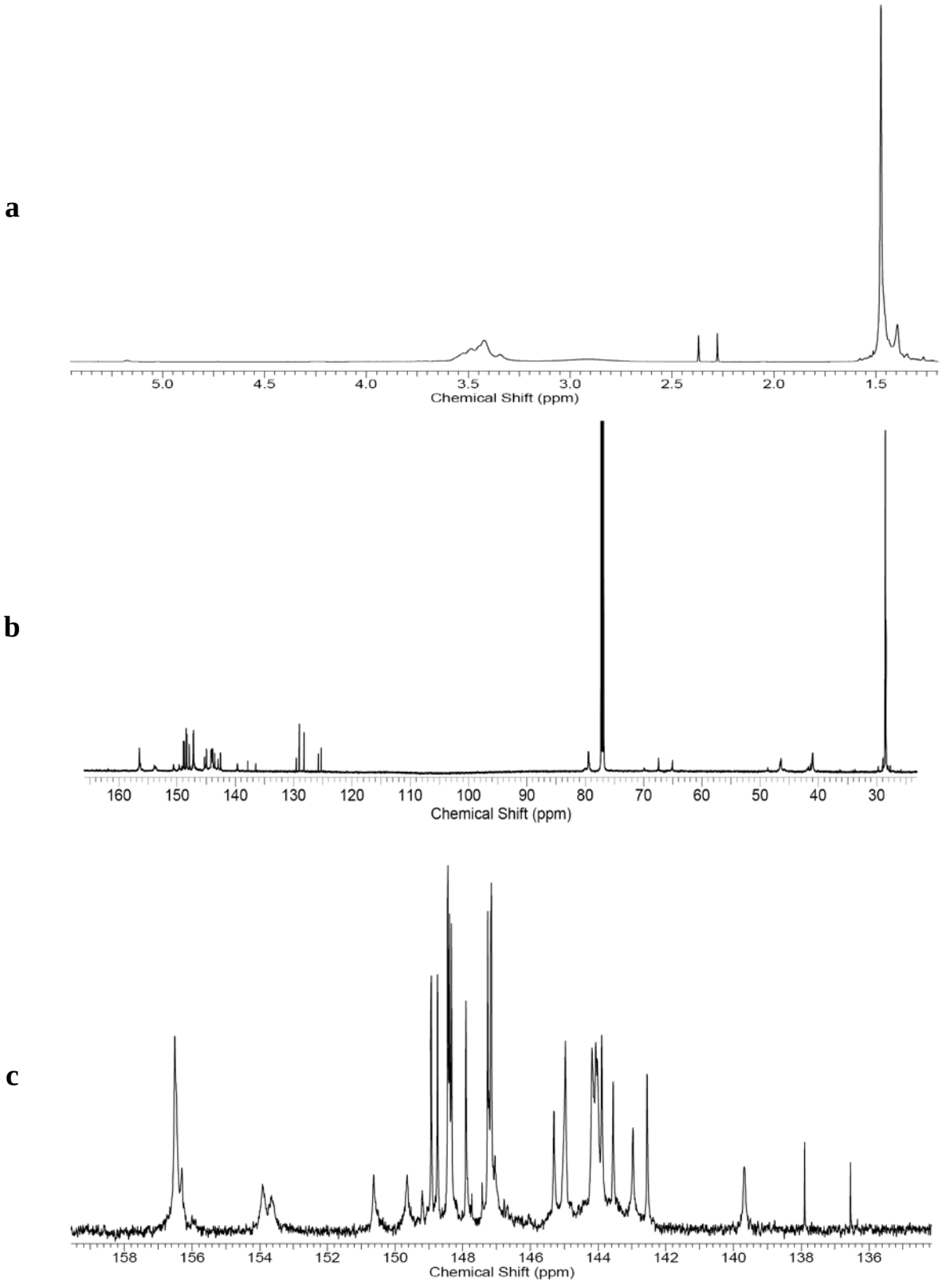


Fig. S20. ^{1}H (a), full ^{13}C (b) and low field ^{13}C (c) NMR spectra of **1g** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

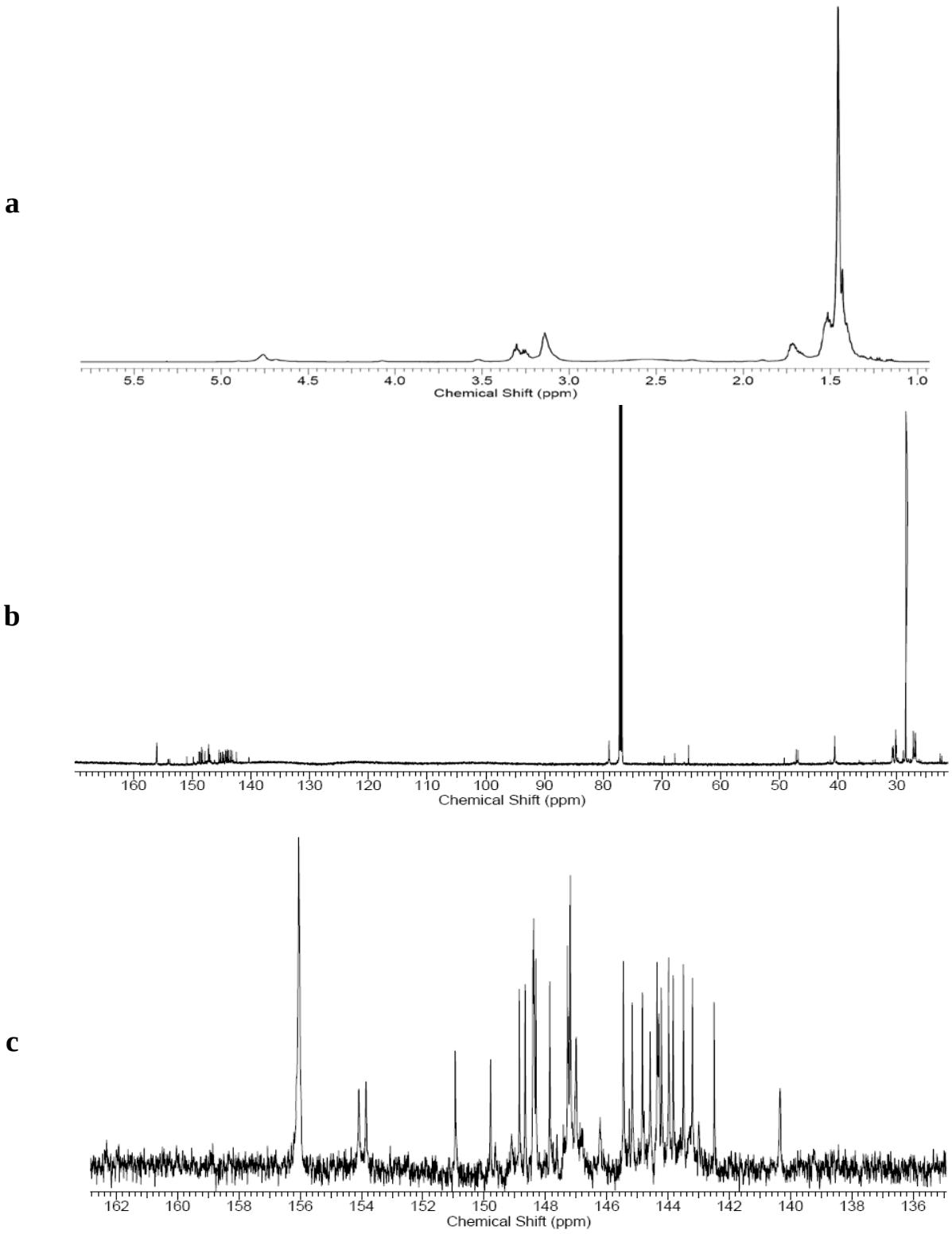


Fig. S21. ^{1}H (a), full ^{13}C (b) and low field ^{13}C (c) NMR spectra of **1h** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

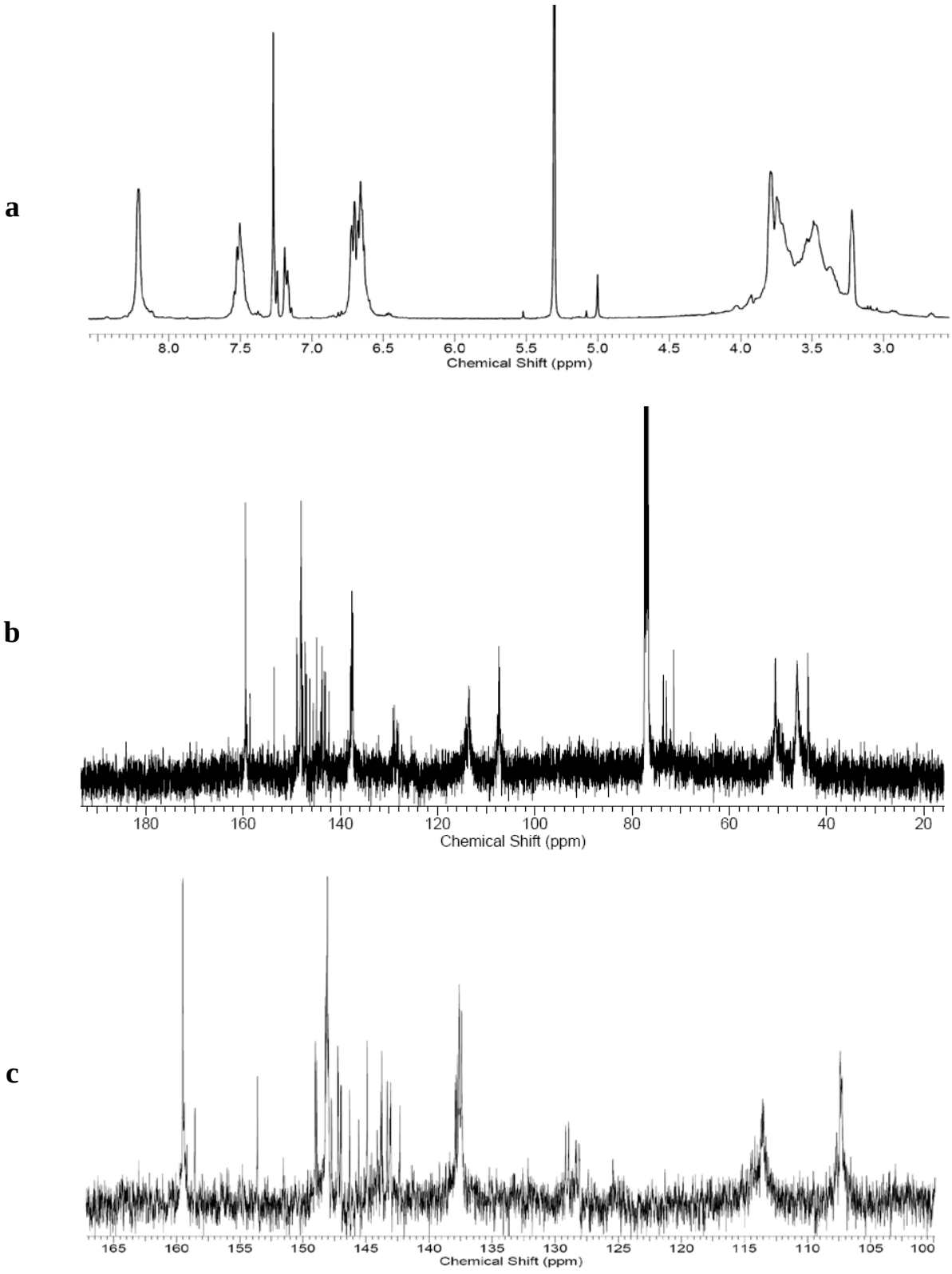


Fig. S22. ^{1}H (a), full ^{13}C (b) and low field ^{13}C (c) NMR spectra of **1i** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

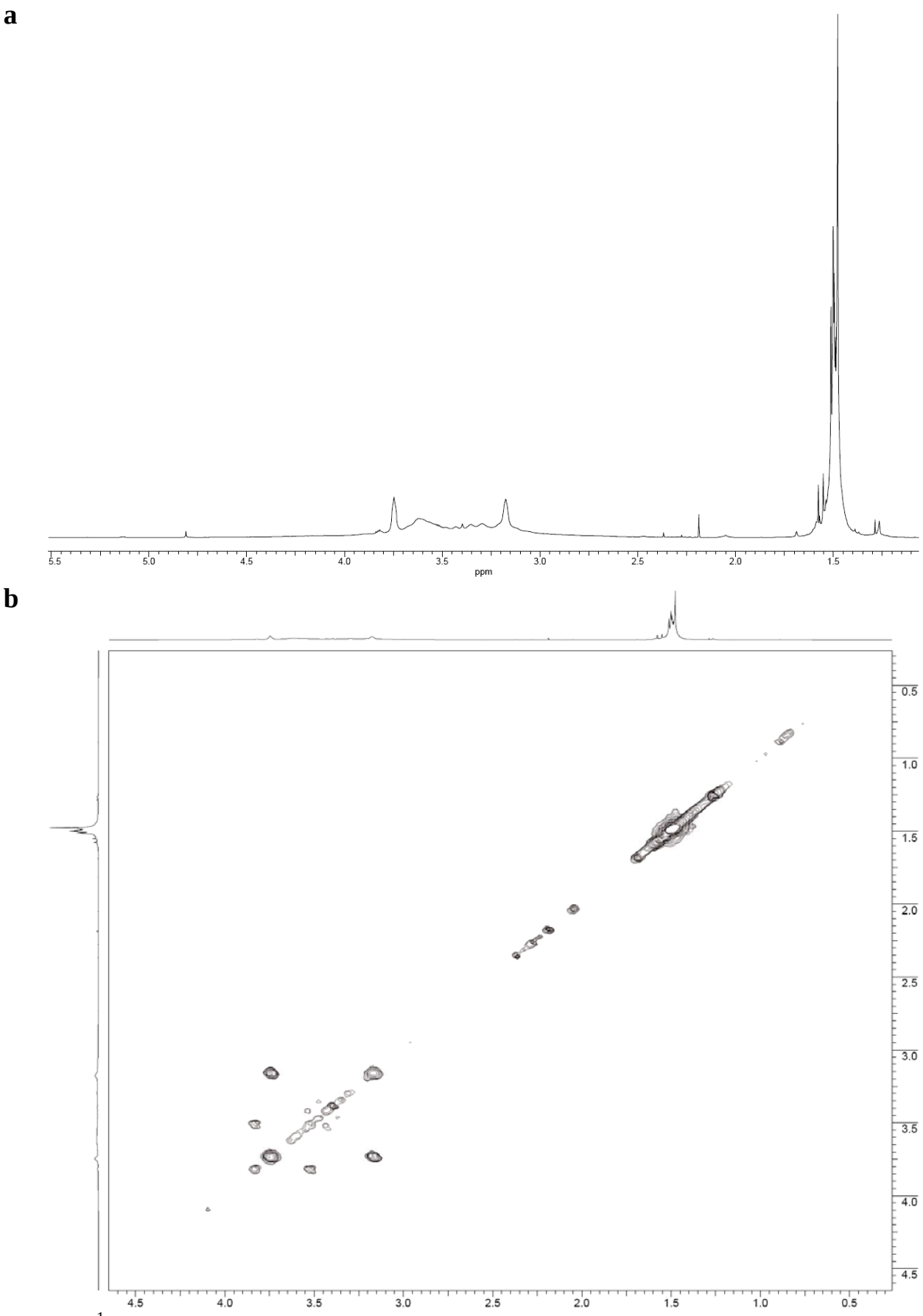


Fig. S23. ^{1}H (a) and H-H COSY (b) NMR spectra of **1j** ($CDCl_3$, 600 MHz)

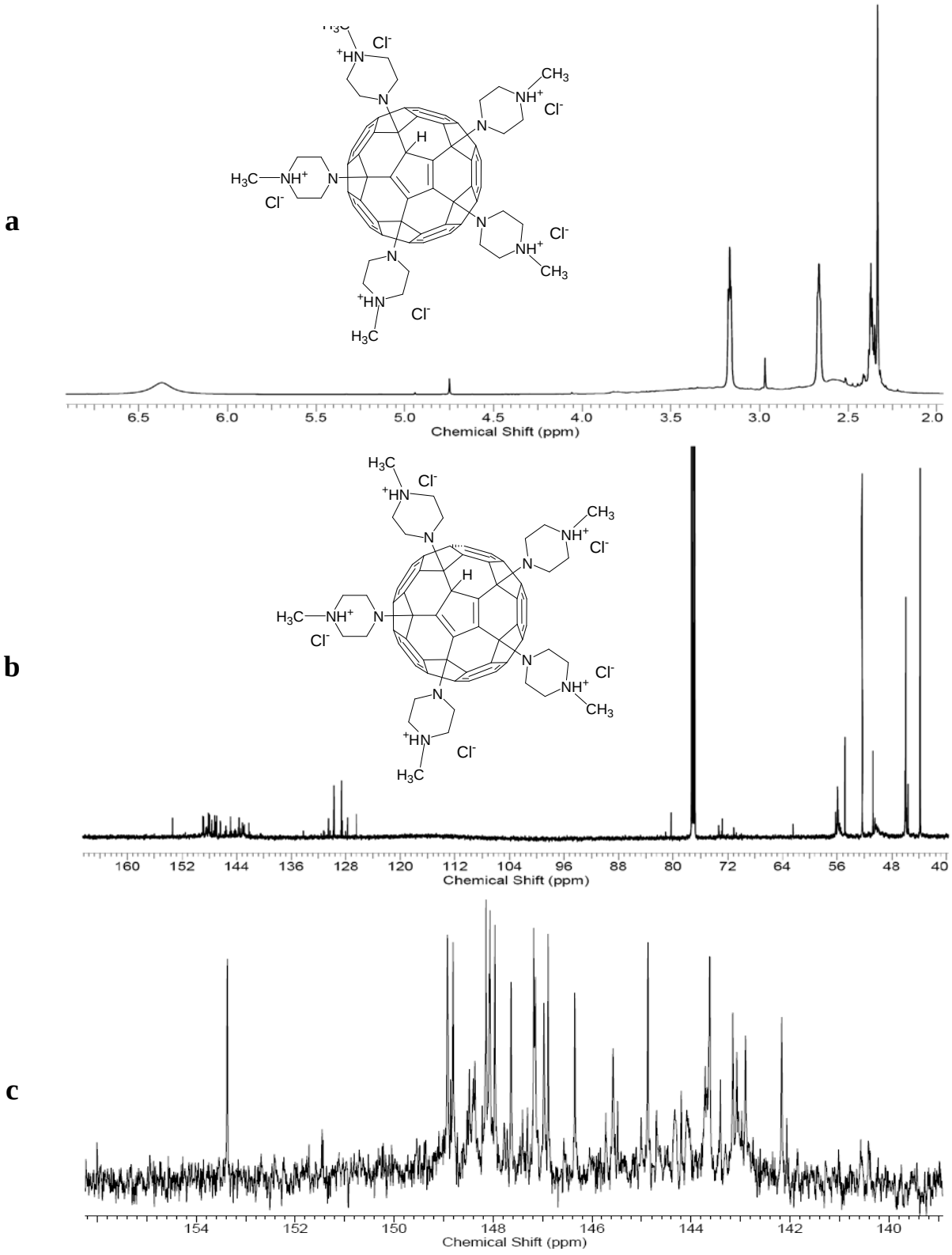
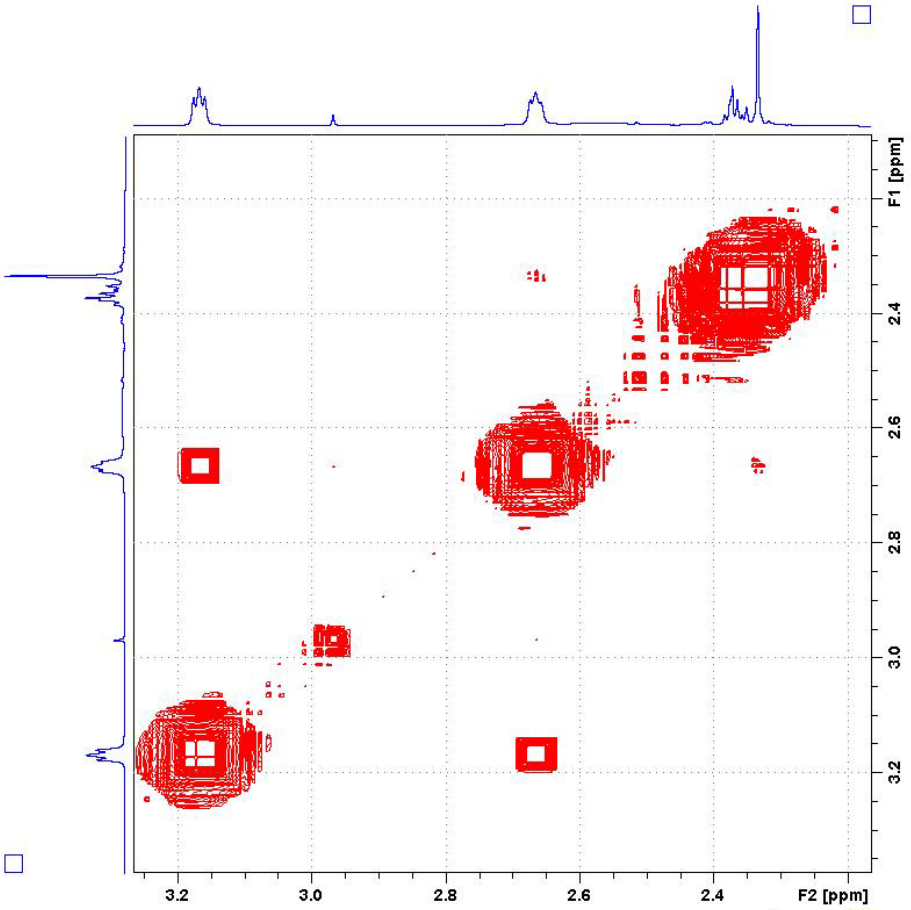


Fig. S24. ^{1}H (a), full ^{13}C (b) and low field ^{13}C (c) NMR spectra of **1k** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

a



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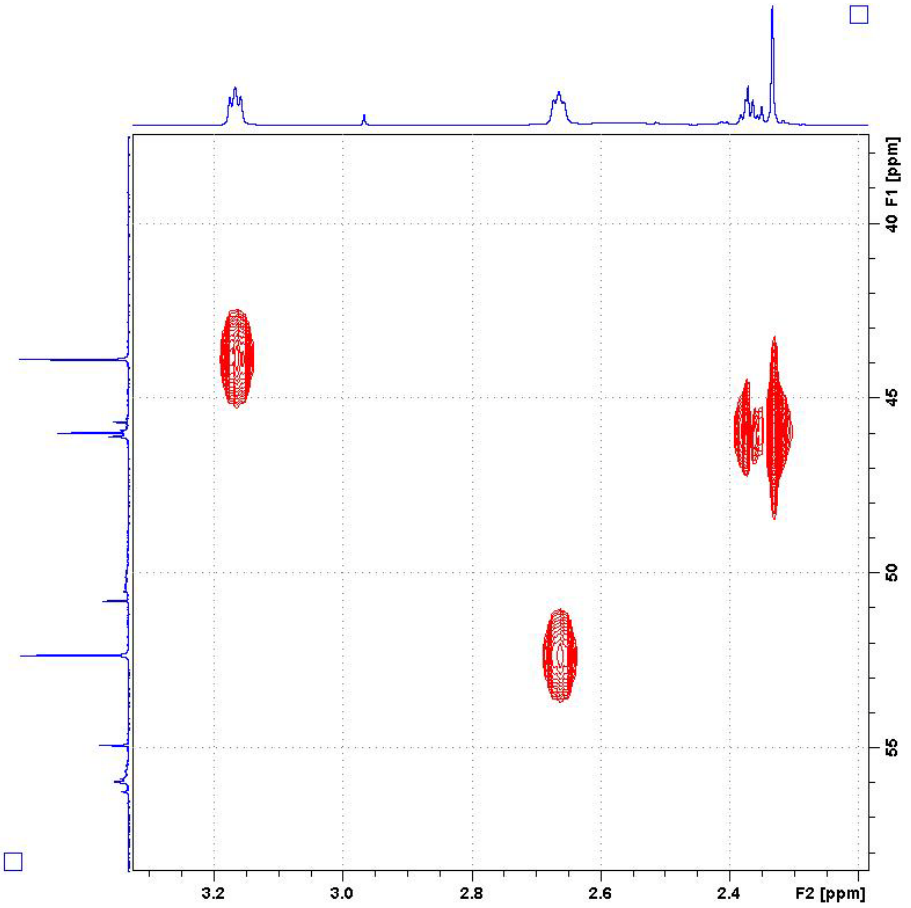

Fig. S25. H-H COSY (a) and H-C HMQC (b) NMR spectra of **1k** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

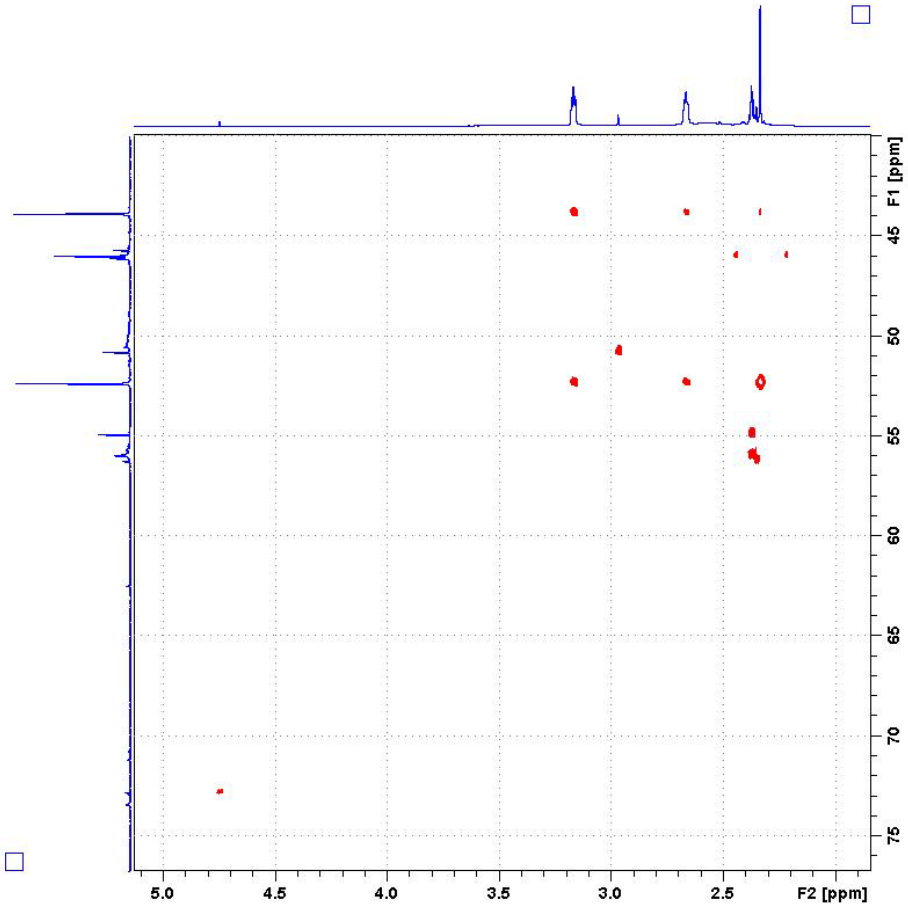

Fig. S26. H-C HMBC NMR spectrum of **1k**.

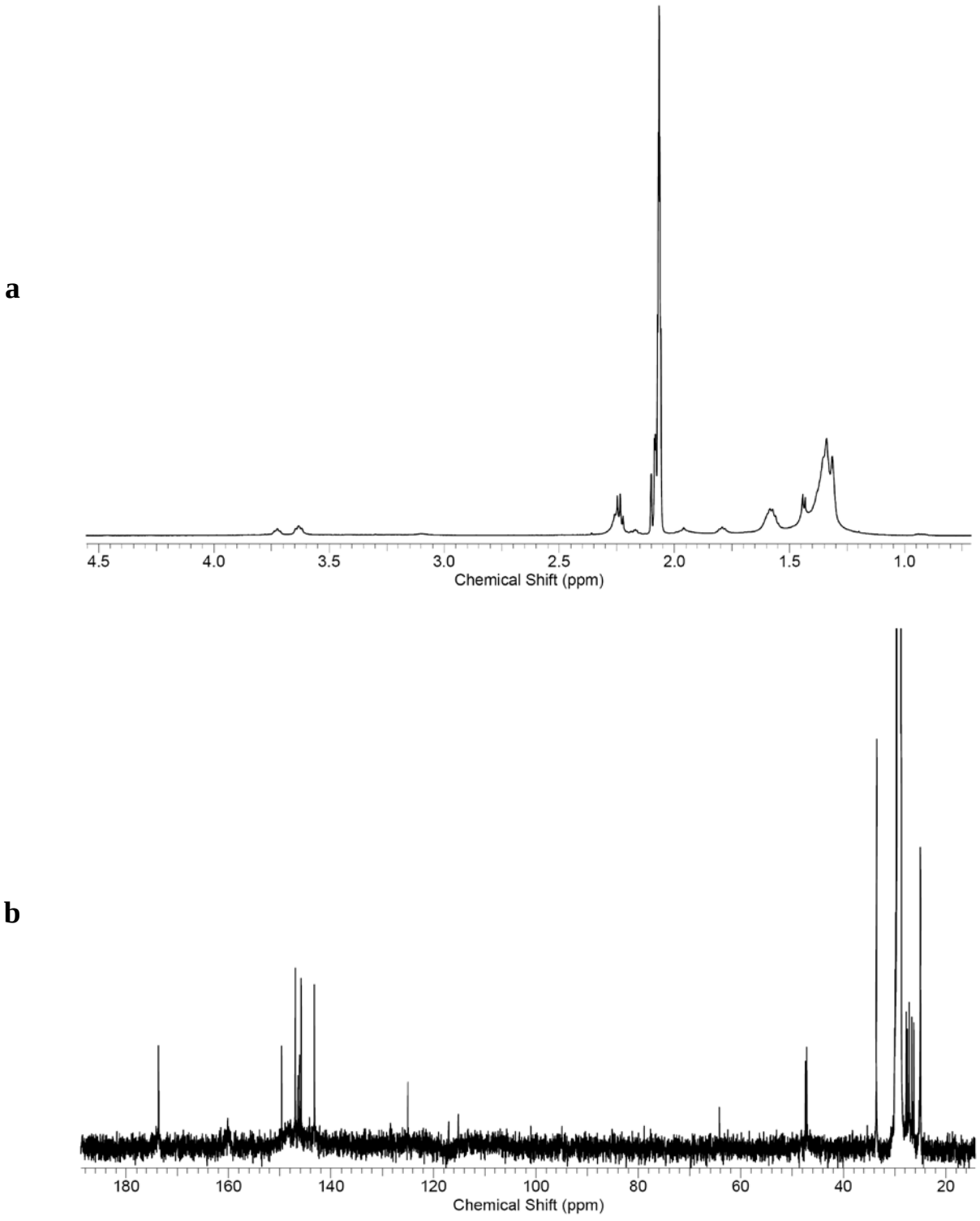

Fig. S27. ^{1}H (a) and full ^{13}C (b) NMR spectra of **1l** ($CDCl_3$, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR).

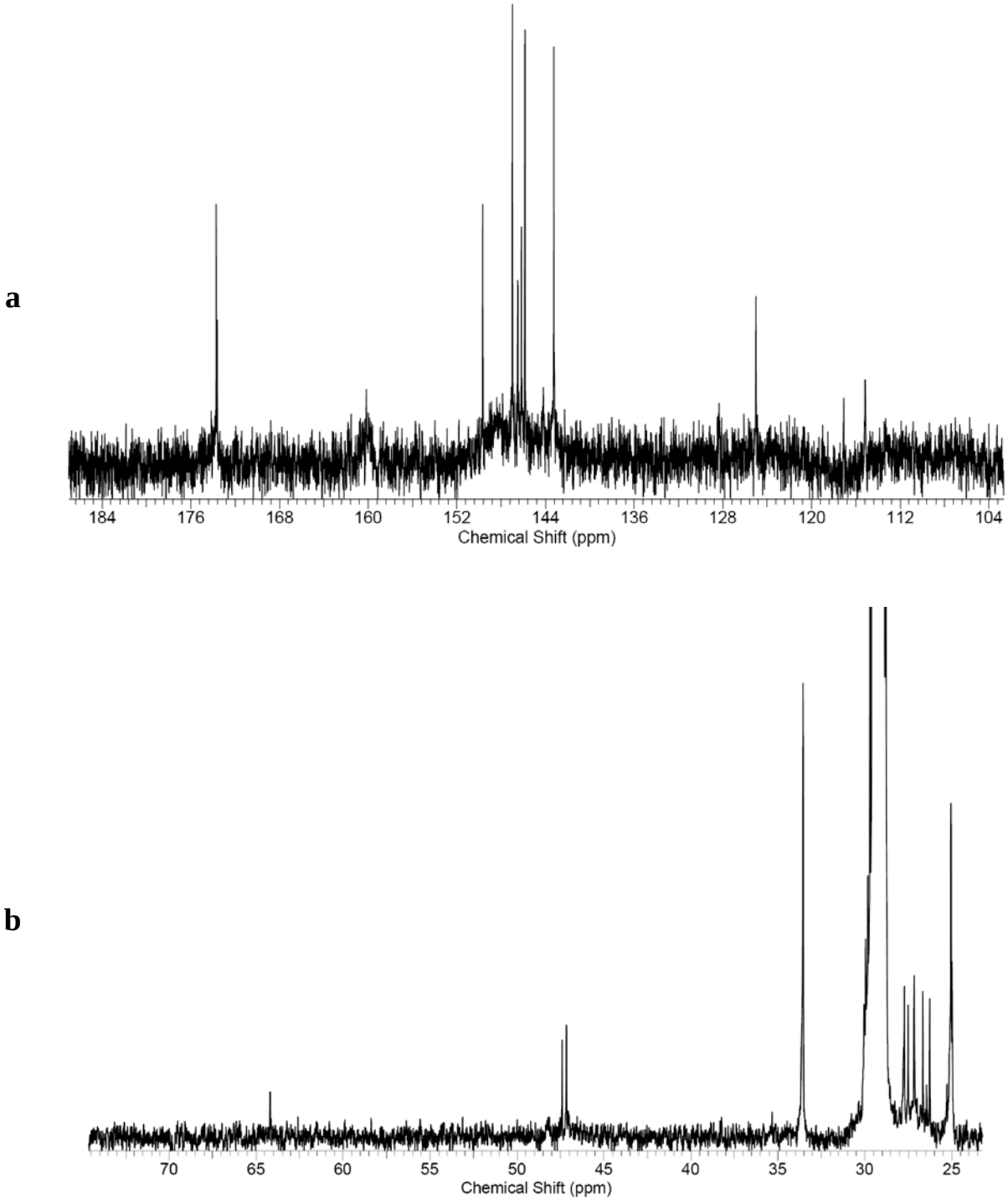

Fig. S28. Low field ^{13}C (a) and high field ^{13}C (b) NMR spectra of **1l** ($CDCl_3$, 150 MHz).

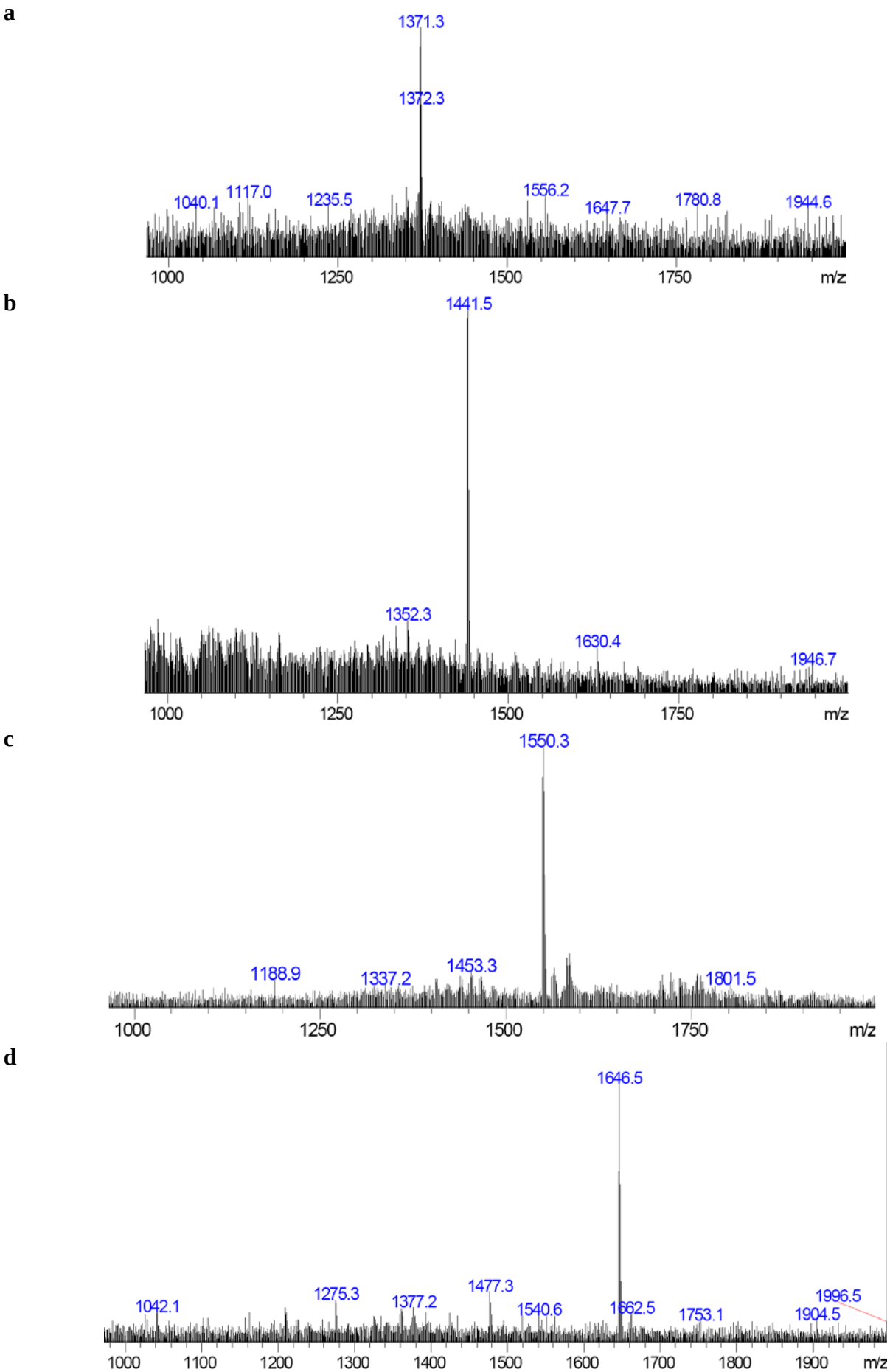

Fig. S29. ESI-MS spectra of **1a** (a, [**1a**-Cl]$^-$, calculated m/z 1371.4), **1c** (b, [**1b**-Cl]$^-$, calculated m/z 1441.5), **1g** (c, [**1g**-H]$^-$, calculated m/z 1550.5), **1j** (d, [**1j**-H]$^-$, calculated m/z 1646.6)

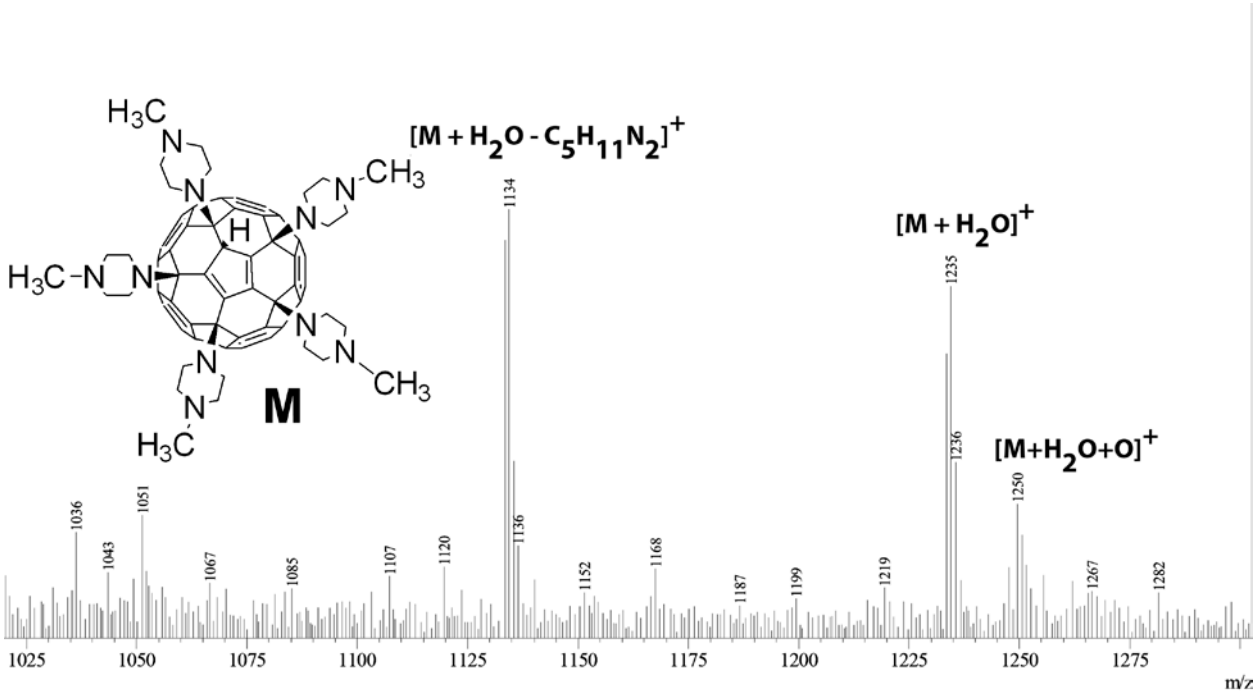

Fig. S30. ESI-MS spectrum of **1k**.

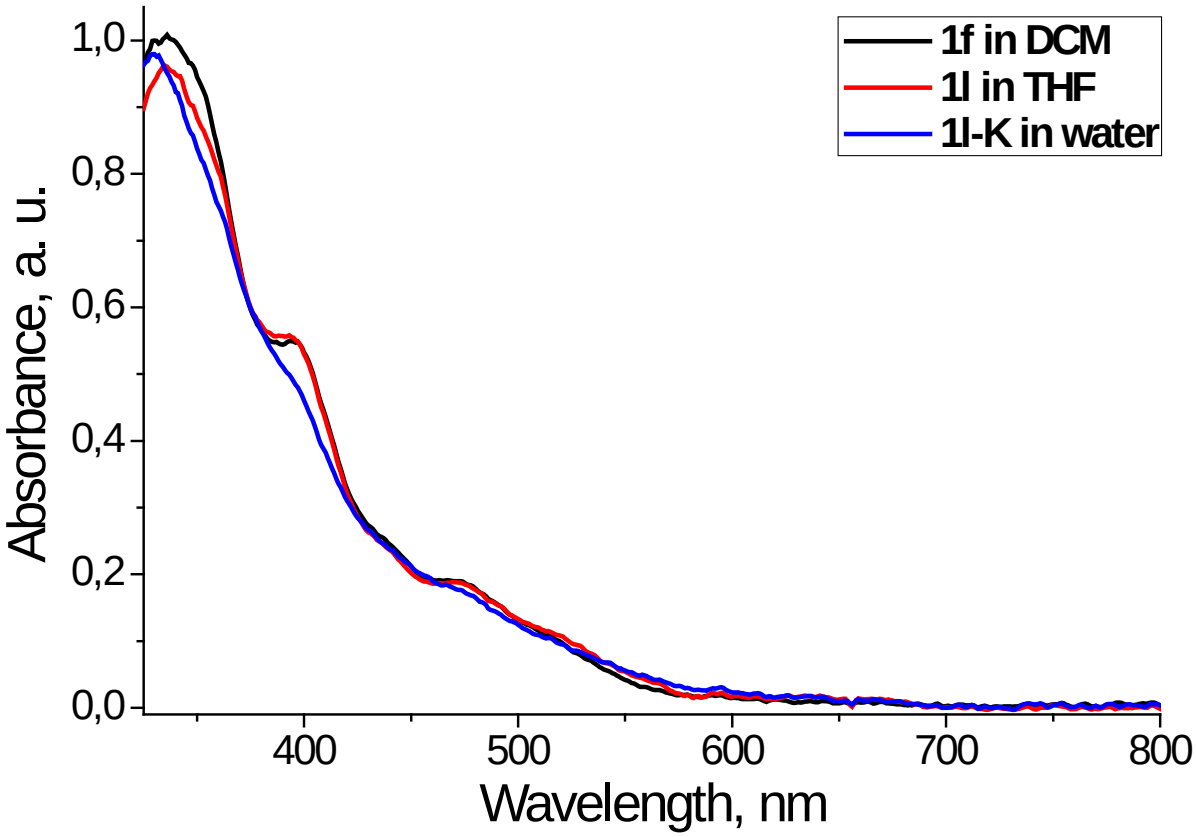

Fig. S31. The absorption spectra of the compound **1f** in low polar solvent (dichloromethane), the acid **1l** in polar solvent (tetrahydrofurane) and the salt **1l-K** in water.

X-ray crystallography for 1d·$CHCl_3$

Data collection for single crystal of **1d**·$CHCl_3$ (0.04 × 0.02 × 0.01 mm^3) was carried out with a MAR225 CCD detector at 100 K using synchrotron radiation at the BESSY storage ring, BL 14.2 (λ = 0.9050 Å, PSF of the Free University of Berlin, Germany). The structure was solved using direct methods (SHELXS97) and anisotropically refined against $|F^2|$ with SHELXL97. Absorption correction was not applied. All hydrogen atoms were placed into geometrically calculated positions and refined in the riding mode. Crystal data for **1d**·$CHCl_3$: M = 1976.84, monoclinic, $P2_1$, a = 27.853(1), b = 29.022(1), c = 11.599(1) Å, β = 90.132(5)°, V = 9376.0(6) $Å^3$, Z = 4, D_{calc} = 1.400 g cm^{-3}. Anisotropic refinement with 38574 reflections and 2710 parameters yielded a conventional R_1 = 0.082 for 35863 reflections with $I > 2\sigma(I)$ and wR_2 = 0.199 for all reflections. Asymmetric unit contains two independent **1d** molecules (1 and 2) and two chloroform molecules. Both **1d** molecules exhibit the disorder of Cl atoms attached to the central pentagon on the fullerene cage. Obviously, the disorder occurs due to the approximate five-fold symmetry of the $C_{60}(NH\text{-}CH(CH_2Ph)COOBu^t)_5Cl$ molecule which results in an overlap of the main molecule and the molecule(s) rotated by 72° around the pseudo five-fold axis. Therefore, the Cl atom in the molecule 1 is formally disordered between two positions, whereas disordering of Cl atom between three positions was present in the molecule 2. One of the solvated chloroform molecules is also disordered. For more details see CCDC 861958.

Experimental procedures used for the preparation of 1a-l

Chlorofullerene $C_{60}Cl_6$ (0.2-1.0 g) was dissolved in dry toluene (200-500 mL, distilled over sodium and stored with Na turnings prior using) in an inert atmosphere at room temperature. A small excess (7 eq.) of either a hydrochloride salt of amine or amino acid ester or a toluene solution of corresponding amino compound mixed with 4-5 fold excess of glacial acetic acid was added. Then a finely ground anhydrous potassium carbonate was added in a great excess (1-5 g). The resulting mixture was vigorously stirred at room temperature until TLC or HPLC analysis showed complete disappearance of the pristine $C_{60}Cl_6$ and the formation of corresponding aminofullerenes. The reactions with acetate salts of amines were very fast (2-15 min), while the crystalline hydrochlorides reacted slowly and required 2-5 days to complete the synthesis.

After the completion of the reaction insoluble byproducts and excess of potassium carbonate were removed by filtration and the filtrate was poured on the top of the silica gel chromatography column (2x30 cm, silica 40-60 μm, 60 Å). Elution with the toluene/ethyl acetate (1-20%) or toluene/methanol (0.3-1.0%) mixtures produced bright orange-red solutions of the title aminofullerenes **1a-j**.

For the deprotection of BOC- or Bu^t-protected solubilizing groups compounds **1f-1h** and **1j** (0.1-0.5 g) were dissolved in anhydrous CH_2Cl_2 (50-100 ml) and treated with a large excess of trifluoroacetic acid (10 ml). Resulting solution was stirred for 15 min at room temperature and then concentrated at the rotary evaporator. The obtained residues of carboxylic acid **1l** or trifluoroacetes of the amines obtained from **1g**, **1h** and **1j** were washed with diethyl ether and dried in air.

Selected spectroscopy data:

1a. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.40-1.55 (m, 45H), 3.89-4.20 (m, 10H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 28.04 (COOC(*C*H_3)$_3$), 28.09 (COOC(*C*H_3)$_3$), 28.13 (COOC(*C*H_3)$_3$), 48.65 (*C*H_2), 49.57 (*C*H_2), 50.85 (*C*H_2), 64.61 (sp^3 fullerene cage), 67.00 (sp^3 fullerene cage), 69.08 (sp^3 fullerene cage), 76.36 (*C*-Cl), 81.24 (COO*C*(CH$_3$)$_3$), 81.55 (COO*C*(CH$_3$)$_3$), 81.58 (COO*C*(CH$_3$)$_3$), 139.59, 142.53, 143.00, 143.40, 143.69, 143.83, 143.92, 144.13, 144.22, 144.82, 145.23, 145.31, 147.17, 147.19, 147.23, 147.29, 147.91, 148.34, 148.37, 148.45, 148.72, 148.88, 149.62, 150.33, 153.75, 154.37, 171.20 (*C*OOC(CH$_3$)$_3$), 171.80 (*C*OOC(CH$_3$)$_3$), 171.96 (*C*OOC(CH$_3$)$_3$).

FT-IR (KBr pellet, υ, cm^{-1}): 534 (M), 557 (M), 668 (M), 753 (M), 849 (M), 937 (M), 956 (M), 977 (M), 1036 (M), 1157 (VS), 1240 (S), 1282 (M), 1356 (M), 1368 (S), 1392 (M), 1433 (M), 1457 (M), 1732 (S), 2930 (M), 2977 (M).

1b. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.35 (s, 9H), 1.37 (s, 9H), 1.38 (s, 9H), 1.39 (s, 9H), 1.45 (s, 9H), 1.50 -1.57 (m, 15H), 4.32-4.78 (m, 5H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 20.62 (CH_3), 20.64 (CH_3), 20.89 (CH_3), 21.19 (CH_3), 27.85 (COOC(CH_3)$_3$), 27.94 (COOC(CH_3)$_3$), 27.96 (COOC(CH_3)$_3$), 27.97 (COOC(CH_3)$_3$), 54.21 (CH), 54.38 (CH), 54.46 (CH), 55.03 (CH), 56.33 (CH), 64.34 (sp^3 fullerene cage), 64.56 (sp^3 fullerene cage), 66.44 (sp^3 fullerene cage), 66.78 (sp^3 fullerene cage), 69.18 (sp^3 fullerene cage), 76.6 (C-Cl), 80.57 (COOC(CH_3)$_3$), 81.04 (COOC(CH_3)$_3$), 81.06 (COOC(CH_3)$_3$), 81.14 (COOC(CH_3)$_3$), 140.23, 140.28, 142.45, 142.48, 142.80, 142.96, 143.08, 143.22, 143.35, 143.50, 143.79, 143.85, 143.89, 143.95, 144.16, 144.20, 144.37, 144.38, 144.48, 144.52, 144.73, 145.08, 145.32, 145.56, 145.76, 147.09, 147.17, 147.21, 147.24, 147.28, 147.35, 147.90, 147.95, 148.24, 148.34, 148.37, 148.44, 148.49, 148.65, 148.73, 148.79, 150.13, 150.19, 150.53, 151.65, 153.97, 154.23, 154.59, 154.91, 175.61 (COOC(CH_3)$_3$), 176.02 (COOC(CH_3)$_3$), 176.17 (COOC(CH_3)$_3$).

FT-IR (KBr pellet, υ, cm^{-1}): 469 (W), 538 (W), 563 (W), 668 (W), 748 (W), 849 (W), 882 (VW), 1036 (W), 1066 (W), 1152 (S), 1220 (W), 1252 (W), 1300 (W), 1338 (W), 1368 (M), 1392 (W), 1456 (W), 1474 (W), 1636 (M), 1726 (M), 2853 (W), 2926 (M), 2975 (M), 3432 (VS).

1c. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.35 (s, 9H), 1.37 (s, 9H), 1.38 (s, 9H), 1.39 (s, 9H), 1.45 (s, 9H), 1.50 -1.57 (m, 15H), 4.32-4.78 (m, 5H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 20.62 (CH_3), 20.64 (CH_3), 20.88 (CH_3), 21.18 (CH_3), 27.86 (COOC(CH_3)$_3$), 27.94 (COOC(CH_3)$_3$), 27.96 (COOC(CH_3)$_3$), 27.97 (COOC(CH_3)$_3$), 54.21 (CH), 54.38 (CH), 54.47 (CH), 55.03 (CH), 56.33 (CH), 64.34 (sp^3 fullerene cage), 64.57 (sp^3 fullerene cage), 66.45 (sp^3 fullerene cage), 66.79 (sp^3 fullerene cage), 69.17 (sp^3 fullerene cage), 76.6 (C-Cl), 80.55 (COOC(CH_3)$_3$), 81.02 (COOC(CH_3)$_3$), 81.06 (COOC(CH_3)$_3$), 81.13 (COOC(CH_3)$_3$), 140.25, 140.29, 142.45, 142.48, 142.80, 142.96, 143.10, 143.22, 143.35, 143.50, 143.79, 143.85, 143.90, 143.95, 144.16, 144.20, 144.36, 144.38, 144.48, 144.51, 144.73, 145.09, 145.32, 145.56, 145.76, 147.10, 147.17, 147.21, 147.24, 147.28, 147.35, 147.90, 147.95, 148.25, 148.33, 148.37, 148.44, 148.49, 148.65, 148.73, 148.80, 150.11, 150.20, 150.53, 151.66, 153.98, 154.23, 154.59, 154.92, 175.59 (COOC(CH_3)$_3$), 175.99 (COOC(CH_3)$_3$), 176.15 (COOC(CH_3)$_3$).

FT-IR (KBr pellet, υ, cm^{-1}): 438 (VW), 476 (VW), 538 (M), 564 (W), 592 (VW), 652 (W), 682 (VW), 748 (W), 772 (W), 798 (W), 848 (M), 882 (W), 1066 (M), 1084 (M), 1152 (VS), 1220 (M), 1252 (M), 1300 (M), 1328 (W), 1368 (S), 1392 (M), 1456 (M), 1478 (M), 1632 (W), 1726 (S), 2868 (W), 2930 (M), 2974 (M), 3314 (W), 3420 (W).

1d. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.04 (s, 9H), 1.15 (s, 9H), 1.17 (s, 9H), 1.21 (s, 9H), 1.24 (s, 9H), 2.94-3.80 (m, 10H), 4.90-5.35 (m, 5H), 7.19-7.52 (m, 25H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 27.73 (COOC(CH_3)$_3$), 27.74 (COOC(CH_3)$_3$), 27.80 (COOC(CH_3)$_3$), 27.82 (COOC(CH_3)$_3$), 27.84 (COOC(CH_3)$_3$), 40.45 (CH_2Ph), 40.59 (CH_2Ph), 41.13 (CH_2Ph), 41.39 (CH_2Ph), 41.92 (CH_2Ph), 58.93, 59.69, 59.88, 61.89, 62.58, 63.83, 64.29, 65.52, 67.20, 68.45, 77.56 (*C*-Cl), 80.98 (COO*C*(CH_3)$_3$), 81.27 (COO*C*(CH_3)$_3$), 81.36 (COO*C*(CH_3)$_3$), 81.40 (COO*C*(CH_3)$_3$), 126.42, 126.47, 126.50, 126.61, 126.64, 128.18, 128.33, 128.37, 129.17, 129.44, 129.95, 130.00, 130.04, 137.32, 137.35, 137.38, 137.66, 137.81, 140.83, 141.53, 142.41, 142.45, 142.62, 142.75, 142.86, 143.44, 143.52, 143.58, 143.62, 143.77, 144.06, 144.17, 144.18, 144.31, 144.32, 144.39, 144.44, 144.64, 144.88, 145.49, 145.58, 145.95, 147.15, 147.18, 147.23, 147.30, 147.34, 147.38, 147.95, 148.01, 148.25, 148.34, 148.37, 148.42, 148.52, 148.53, 148.58, 148.75, 148.83, 149.76, 150.56, 151.05, 151.55, 154.18, 154.23, 154.76, 155.33, 174.74 (*C*OOC(CH_3)$_3$), 174.98 (*C*OOC(CH_3)$_3$), 175.60 (*C*OOC(CH_3)$_3$), 175.69 (*C*OOC(CH_3)$_3$), 175.81 (*C*OOC(CH_3)$_3$).

FT-IR (KBr pellet, υ, cm^{-1}): 467 (VW), 503 (W), 536 (W), 564 (W), 592 (W), 622 (W), 668 (W), 698 (M), 738 (M), 798 (W), 845 (W), 911 (VW), 984 (VW), 1031 (W), 1077 (W), 1151 (VS), 1223 (M), 1251 (M), 1342 (W), 1368 (M), 1392 (W), 1420 (W), 1456 (M), 1475 (W), 1496 (W), 1522 (VW), 1540 (VW), 1559 (VW), 1605 (W), 1636 (W), 1722 (S), 2930 (M), 2975 (M), 3002 (W), 3028 (W), 3062 (W), 3086 (W), 3433 (M).

1e. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.18 (s, 9H), 1.25 (s, 9H), 1.26 (s, 9H), 1.27 (s, 9H), 1.28 (s, 9H), 1.35 (s, 9H), 1.37 (s, 9H), 1.38 (s, 9H), 1.39 (s, 9H), 1.44 (s, 9H), 3.60-3.95 (m, 10H), 4.51 (s, 1H), 4.56 (s, 1H), 4.62 (s, 1H), 4.65 (s, 1H), 4.84 (s, 1H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 27.46 (COOC(CH_3)$_3$), 27.50 (COOC(CH_3)$_3$), 27.56 (COOC(CH_3)$_3$), 27.59 (COOC(CH_3)$_3$), 28.03 (COOC(CH_3)$_3$), 28.07 (COOC(CH_3)$_3$), 28.19 (COOC(CH_3)$_3$), 59.11, 59.19, 59.55, 59.99, 61.11, 64.05, 64.26, 64.47, 64.50, 64.79, 64.91, 65.06, 65.88, 66.67, 68.56, 72.54, 72.59, 72.68, 72.76, 72.93, 77.12 (*C*-Cl), 80.60 (COO*C*(CH_3)$_3$), 80.93 (COO*C*(CH_3)$_3$), 80.96 (COO*C*(CH_3)$_3$), 140.34, 140.92, 142.44, 142.50, 142.82, 142.96, 143.08, 143.13, 143.37, 143.66, 143.70, 143.82, 143.86, 143.87, 144.03, 144.18, 144.23, 144.31, 144.34, 144.51, 144.58, 145.35, 145.45, 145.60, 145.68, 147.08, 147.19, 147.21, 147.27, 147.34, 147.40, 147.90, 147.96, 148.20, 148.29, 148.34, 148.38, 148.40, 148.47, 148.53, 148.65, 148.75, 148.77, 149.93, 150.13, 150.78, 151.35, 154.13, 154.20, 154.43, 154.90, 173.60 (*C*OOC(CH_3)$_3$), 173.63 (*C*OOC(CH_3)$_3$), 173.74 (*C*OOC(CH_3)$_3$), 173.97(*C*OOC(CH_3)$_3$), 174.31 (*C*OOC(CH_3)$_3$).

FT-IR (KBr pellet, υ, cm^{-1}): 459 (W), 482 (W), 523 (W), 544 (M), 564 (W), 613 (W), 668 (W), 694 (W), 751 (W), 764 (W), 788 (W), 816 (W), 850 (M), 880 (W), 983 (VW), 1021 (W), 1055 (M), 1096 (S), 1155 (VS), 1197 (S), 1235 (M), 1247 (M), 1290 (W), 1339 (W), 1365 (S),

1391 (M), 1419 (W), 1462 (M), 1473 (M), 1636 (W), 1732 (S), 2871 (M), 2932 (M), 2975 (S), 3436 (M).

1f. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.31 (m, 60H), 1.45 (m, 10H), 1.46 (s, 45H), 1.59 (m, 10H), 1.70 (m, 10H), 2.22 (t, 10H), 3.28 (m, 5H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 25.13, 27.52, 27.54, 28.14, 29.16, 29.38, 29.52, 29.55, 29.63, 29.67, 29.68, 35.63, 46.97, 47.30, 65.52, 67.84, 69.62, 77.59, 79.89, 140.45, 142.49, 143.27, 143.49, 143.82, 143.95, 144.25, 144.35, 144.4, 144.63, 144.83, 145.21, 145.49, 146.99, 147.2, 147.26, 147.29, 147.86, 148.31, 148.36, 148.39, 148.41, 148.66, 148.85, 149.84, 151.02, 153.82, 154.16, 173.3, 173.35.

FT-IR (KBr pellet, υ, cm^{-1}): 420 (W), 465 (W), 529 (M), 536 (M), 582 (W), 651 (W), 668 (M), 722 (M), 754 (W), 847 (M), 919 (W), 950 (W), 1039 (M), 1112 (M), 1153 (VS), 1255 (M), 1318 (M), 1366 (S), 1391 (M), 1419 (M), 1457 (M), 1540 (W), 1559 (W), 1636 (M), 1653 (M), 1730 (VS), 1772 (W), 2853 (S), 2926 (VS), 2975 (M), 3434 (S).

1g. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.37-1.50 (m, 45H), 3.30-3.60 (m, 20H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 28.47 (*C*H_3), 28.53 (*C*H_3), 41.00 (*C*H_2), 41.35 (*C*H_2), 41.69 (*C*H_2), 46.41 (*C*H_2), 46.57 (*C*H_2), 48.68 (*C*H_2), 65.05 (sp^3 fullerene cage), 67.43 (sp^3 fullerene cage), 69.92 (sp^3 fullerene cage), 79.29 (COO*C*$(CH_3)_3$), 79.41 (COO*C*$(CH_3)_3$), 79.46 (COO*C*$(CH_3)_3$), 136.54, 137.89, 139.69, 142.56, 142.97, 143.56, 143.89, 144.02, 144.07, 144.18, 144.97, 145.31, 147.16, 147.17, 147.27, 147.90, 148.33, 148.39, 148.44, 148.75, 148.93, 149.65, 150.64, 153.65, 153.91, 156.30 (*C*OOC$(CH_3)_3$), 156.46 (*C*OOC$(CH_3)_3$), 156.51 (*C*OOC$(CH_3)_3$).

FT-IR (KBr pellet, υ, cm^{-1}): 465 (W), 539 (M), 584 (M), 668 (W), 730 (W), 763 (W), 780 (M), 859 (W), 998 (M), 1043 (M), 1169 (VS), 1251 (S), 1271 (S), 1341 (M), 1366 (S), 1392 (S), 1419 (M), 1456 (S), 1479 (M), 1508 (S), 1636 (M), 1697 (VS), 2930 (M), 2976 (S), 3427 (S).

1h. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.37-1.58 (m, 75H), 1.72 (m, 10H), 3.14 (m, 10H), 3.28 (m, 10H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 28.47 (*C*H_3), 26.83 (*C*H_2), 26.96 (*C*H_2), 27.11 (*C*H_2), 27.17 (*C*H_2), 27.20 (*C*H_2), 30.09 (*C*H_2), 30.16 (*C*H_2), 30.62 (*C*H_2), 30.80 (*C*H_2), 40.59 (*C*H_2), 46.83 (*C*H_2), 47.14 (*C*H_2), 49.18 (*C*H_2), 65.48 (sp^3 fullerene cage), 67.82 (sp^3 fullerene cage), 69.61 (sp^3 fullerene cage), 79.00 (COO*C*$(CH_3)_3$), 140.34, 142.49, 143.21, 143.50, 143.83, 143.98, 144.22, 144.29, 144.36, 144.59, 144.83, 145.17, 145.45, 146.21, 147.00, 147.19, 147.25, 147.28, 147.86, 148.31, 148.38, 148.41, 148.66, 148.85, 149.79, 150.94, 153.85, 154.09, 156.05 (*C*OOC$(CH_3)_3$).

FT-IR (KBr pellet, υ, cm^{-1}): 464 (W), 537 (M), 558 (M), 582 (M), 648 (W), 668 (W), 730 (W), 763 (W), 780 (M), 865 (W), 924 (W), 1016 (M), 1042 (M), 1171 (VS), 1250 (S), 1272 (S),

1365 (S), 1391 (M), 1457 (S), 1519 (S), 1694 (VS), 2715 (M), 2857 (S), 2930 (S), 2974 (S), 3374 (S).

1i. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 3.20-3.90 (m, 40H), 6.67 (t, 5H), 6.72 (d, 5H), 7.52 (m, 5H), 8.23 (d, 5H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 43.84, 46.07, 46.17, 50.54, 71.44, 72.99, 73.59, 107.42, 113.54, 128.12, 128.93, 137.43, 137.60, 137.75, 137.87, 137.93, 142.33, 143.05, 143.13, 143.31, 143.68, 143.75, 143.83, 144.10, 144.91, 145.59, 146.30, 146.96, 147.02, 147.18, 147.22, 147.73, 147.97, 148.05, 148.14, 148.23, 148.90, 149.01, 153.61, 158.56, 159.51.

FT-IR (KBr pellet, υ, cm^{-1}): 424 (W), 462 (W), 529 (W), 668 (W), 732 (W), 773 (M), 846 (VW), 942 (W), 980 (M), 1003 (W), 1057 (W), 1098 (W), 1128 (W), 1156 (W), 1246 (M), 1267 (W), 1278 (W), 1312 (W), 1337 (W), 1383 (W), 1437 (S), 1482 (S), 1560 (W), 1595 (S), 1636 (M), 2834 (M), 2923 (M), 3439 (VS).

1j. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 1.40-1.60 (m, 45H), 3.10-3.90 (m, 40H).

FT-IR (KBr pellet, υ, cm^{-1}): 420 (W), 464 (W), 536 (W), 547 (W), 582 (W), 664 (W), 698 (W), 770 (W), 829 (W), 869 (W), 934 (VW), 1000 (S), 1036 (W), 1170 (S), 1252 (S), 1262 (S), 1285 (S), 1298 (M), 1366 (S), 1394 (S), 1420 (S), 1456 (S), 1477 (M), 1701 (VS), 2819 (W), 2856 (M), 2931 (M), 2975 (M), 3436 (M).

1k. ^{1}H NMR (600 MHz, $CDCl_3$, δ, ppm): 2.37 (m, 15H), 2.67 (m, 20H), 3.17 (m, 20H), 4.75 (s, 1H), 6.37 (m, 20H).

^{13}C NMR (150 MHz, $CDCl_3$, δ, ppm): 43.85, 45.95, 46.06, 50.75, 52.32, 54.88, 71.14, 72.79, 73.38, 80.35, 130.52, 131.11, 131.28, 134.21, 142.06, 142.17, 142.9, 143.08, 143.16, 143.4, 143.62, 143.71, 144.87, 145.57, 146.35, 146.88, 146.97, 147.15, 147.18, 147.64, 147.96, 148.06, 148.15, 148.81, 148.86, 148.92, 153.37.

FT-IR (KBr pellet, υ, cm^{-1}): 474 (M), 525 (M), 548 (M), 585 (M), 837 (VW), 918 (VW), 981 (M), 1025 (M), 1057 (W), 1114 (M), 1144 (W), 1187 (M), 1280 (W), 1303 (W), 1319 (W), 1374 (W), 1458 (M), 1636 (M), 2476 (W), 2601 (M), 2689 (M), 2833 (M), 2955 (M), 3429 (VS).

Elemental analysis: calculated C, 72.94; H, 4.39; N, 10.01; Cl, 12.66. Found: C, 72.97; H, 4.47; N, 9.94, Cl, 12.84.

1l. ^{1}H NMR (600 MHz, acetone-D6, δ, ppm): 1.34 (m, 70H), 1.58 (m, 10H), 2.09 (m, 10H), 2.24 (m, 10H), 3.68 (m, 5H).

^{13}C NMR (150 MHz, acetone-D6, δ, ppm): 26.66, 27.16, 27.51, 27.72, 29.7, 29.82, 29.95, 33.55, 47.16, 47.40, 64.19, 125.02, 143.25, 145.87, 146.18, 146.5, 146.99, 149.67, 173.71.

FT-IR (KBr pellet, υ, cm^{-1}): 439 (W), 490 (W), 536 (M), 563 (W), 582 (W), 626 (W), 663 (W), 721 (M), 798 (M), 837 (M), 925 (M), 978 (M), 1000 (M), 1038 (W), 1093 (M), 1140 (S),

1199 (VS), 1277 (M), 1410 (M), 1434 (M), 1463 (M), 1531 (W), 1674 (S), 1682 (S), 1705 (VS), 2852 (S), 2925 (VS).

Preparation of water-soluble salts 1l-K and 1l-Na

Compound **1l** was dissolved in a highly polar organic solvent (THF, acetone, methanol or ethanol) and mixed with equivalent amount of 1M aqueous potassium carbonate or sodium carbonate solution. The solutions were concentrated at the rotary evaporator; the obtained residues were washed with ethanol or acetone and dried in air. The brown powders of **1l-K** and **1l-Na** were obtained in 90-95% yields.

1l-K. Elemental analysis: calculated C, 72.18; H, 5.58; N, 3.66. Found: C, 72.15; H, 5.65; N, 3.50.

FT-IR (KBr pellet, υ, cm^{-1}): 474 (W), 622 (W), 648 (M), 703 (M), 752 (W), 832 (M), 880 (W), 921 (W), 986 (M), 1008 (M), 1059 (M), 1162 (W), 1404 (VS), 1571 (VS), 1631 (S), 1647 (S), 1750 (M), 2852 (S), 2924 (S), 3414 (S).

Virological assays

Activity of the compound **1l-K** against two herpes viruses (*Herpes Simplex* (HSV) and human cytomegalovirus (CMV)) was investigated using standard *in vitro* assays. HSV-sensitive Vero cells and CMV-sensitive human embryo lung fibroblasts (HELF) were used to evaluate the **1l-K** cytotoxicity. The concentration of the compound reducing the viability of the cells by 50% within 3 days after its addition to the culture medium (CC_{50}) was >2500 ug/ml for Vero cells and >1000 ug/ml for HELF. These results show that **1l-K** has low cytotoxicity with respect to the Vero and HELF cells. Antiviral activity was evaluated by monitoring the ability of the compound to inhibit a plaque formation (plaque reduction test) in the cells infected with corresponding viruses. The effective concentration of **1l-K** providing the 50% inhibition of the plaque-forming activity (EC_{50}) was 0.5 ug/ml for HSV and 72 ug/ml for CMV. Selectivity indices (SI) calculated as CC_{50}/EC_{50} were >5000 for HSV and >14 for CMV. These data indicate that **1l-K** shows strong antiviral effect with respect to the both herpes viruses. The efficiency of the antiviral action varies from exceptionally high for HSV to moderate for CMV.

A detailed description of the general experimental procedures (citotoxicity and antiviral assays) is provided in the previous publications:

1. R. A. Kotelnikova, G. N. Bogdanov, E. C. Frog, A. I. Kotelnikov, V. N. Shtolko, V. S. Romanova, S. M. Andreev, A. A. Kushch, N. E. Fedorova, A. A. Medzhidova, G. G. Miller, *J. Nanopart. Res.*, 2003, **5**, 561.

2. M. V. Pavlova, N. E. Fedorova, A. V. Serbin, Iu. A. Egorov, E. N. Karaseva, E. V. Klimova, A. A. Kushch, *Antibiot Khimioter.*, 2008, **53**, 8 [Article in Russian].

Table S1. Agarose gel electrophoresis for **1j** and **1k-K**

Compound name	Mobility in electric field (the cathode is at the left, the anode - on the right)	ζ, mV
1k		+ 20,5
1l-K		– 119