Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supplementary Information for

Triflic acid-mediated Phenylation of *N*-Acylaminoalkyl Diethylacetals and *N*-Acyl-2-phenyl Cyclic Amides

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CONTENTS

General procedure for the diphenylation reaction.

Triflic acid (10 fold excess) was added to a stirred solution of the amide or sulfonamide (2 mmol) in benzene (20 ml) and the reaction mixture heat under reflux until TLC showed no starting amide remaining. The reaction mixture was cooled to room temperature, water (50 ml) was added and the mixture basified with an excess of solid K_2CO_3. The product was extracted into DCM (2 x 50 ml), dried ($MgSO_4$), concentrated *in vacuo* and the product purified by column chromatography on SiO_2.

Reaction of 1 with benzene.

Following the general procedure, **1** (King, F.D.; Aliev, A. E.; Caddick, S.; Copley, R.C. *Org. Biomol. Chem*., **2009**, *7*, 3561) was heated under reflux for 1h. Purification, initially eluting with DCM gave **3** (50% yield), mp 120-121^{o}C (EtOAc/petroleum ether). ^{1}H-NMR (600 MHz) δ = 1.38 – 1.45 (2H, m), 1.96 – 2.02 (2H, m), 3.23 (2H, q, *J* = 7.0 Hz), 3.54 (2H, s), 3.86 (1H, t, *J* = 7.8 Hz), 5.52 (1H, brs), 7.15 – 7.40 (15H, m); ^{13}C-NMR + DEPT (150 MHz) δ = 28.2 (CH_2), 32.9 (CH_2), 39.5 (CH_2), 44.0 (CH_2), 51.0 (CH), 126.4 (CH), 127.5 (CH), 127.9 (CH), 128.6 (CH), 129.2 (CH), 129.6 (CH), 135.2 (C), 144.8 (C), 171.1 (C), FT-IR (neat) 3269, 2935, 1638, 1556, 1494, 1454, 1435, 1344, 751, 716, 695 cm^{-1}, LRMS (EI) 343, 167, 92, 91, HRMS calcd for $C_{24}H_{25}NO$ 343.1931, found 343.1920. Further elution with DCM + 2% MeOH gave **2** (30% yield), identical to that previously reported (King, F.D.; Aliev, A. E.; Caddick, S.; Copley, R.C. *Org. Biomol. Chem*., **2009**, *7*, 3561).

The following amides were prepared following the general procedure:

2-(4-Chlorophenyl)-N-(4,4-diphenylbutyl)acetamide 6 from 5.

Following the general procedure, 2-(4-chlorophenyl)-N-(4,4-diethoxybutyl)-acetamide **5** (King, F.D.; Aliev, A. E.; Caddick, S.; Copley, R.C. *Org. Biomol. Chem*., **2009**, *7*, 3561) (0.62g, 2mmol) was heated under reflux for 1h. Purification by column chromatography, eluting with DCM gave **6** (0.68g, 70% yield), identical to that prepared before and described in the paper.

2-(4-Nitrophenyl)-N-(4,4-diphenylbutyl)acetamide 8 from 4c.

A solution of 2-(4-nitrophenyl)-N-(4,4-diethoxybutyl)-acetamide (King, F.D.; Aliev, A. E.; Caddick, S.; Copley, R.C. *Org. Biomol. Chem*., **2009**, *7*, 3561) (3.2g, 10mmol) in THF (100 ml) and 2M HCl (20 ml) was stirred at room temperature for 2h, until all the starting material had been consumed (TLC). Water (50 ml) was added and the THF removed *in vacuo*. The 2-(4-Nitrophenyl)-1-(2-hydroxypyrrolidin-1-yl)ethanone **4c** formed as a pale yellow solid, which was collected and dried (2.5g, ~100% yield), pure by TLC and NMR showed it to be in equilibrium with the amido-aldehyde (~5%), and was used without further purification; ^{1}H-NMR (500 MHz) δ = 1.80 – 2.15 (4H, m), 3.41 – 3.50 (1H, m), 3.55 – 3.68 (1H, m), 3.74 (2H, s), 4.10 (1H, brs), 5.65 (1H, dd, *J* = 2.7, 6.1 Hz) 7.46 (2H, d, *J* = 8.2 Hz), 8.19 (2H, d, *J* = 8.2 Hz); ^{13}C-NMR + DEPT (150 MHz) δ = 23.2 (CH_2), 32.1 (CH_2), 41.6 (CH_2), 46.9 (CH_2), 82.1 (CH), 123.8 (CH), 130.3 (CH), 141.7 (C), 147.2 (C), 169.9 (C); FT-IR (neat) 3356, 1608, 1514, 1444, 1421, 1343, 1318, 1263, 1172, 1154, 1106, 1040, 1016, 963, 859, 820, 727 cm^{-1}. Following the general procedure, **4c** (1g, 4mmol) was reacted for 1h to give **8**, purified by column chromatography, eluting with DCM (1.2g, 78% yield), mpt 100-102^{o}C (EtOAc/petroleum ether). ^{1}H-NMR (600 MHz) δ = 1.40 – 1.48 (2H, m), 1.98 – 2.05 (2H, m), 3.25 (2H, q, *J* = 7.0 Hz), 3.53 (2H, s), 3.87 (1H, t, *J* = 7.8 Hz), 6.20 (1H, t, *J* = 5.5 Hz), 7.127.30 (10H, m), 7.38 (2H, d, J = 8.6 Hz), 8.10 (2H, d, J = 8.6 Hz); ^{13}C-NMR + DEPT (150 MHz) δ = 28.2 (CH_2), 32.9 (CH_2), 39.8 (CH_2), 43.2 (CH_2), 51.0 (CH), 123.9 (CH), 126.5 (CH), 127.9 (CH), 128.7 (CH), 130.3 (CH), 143.1 (C), 144.8 (C), 147.0 (C), 169.5 (C), FT-IR (neat) 3281, 1637, 1557,

1516, 1493, 1345, 1254, 1110, 1030, 747, 697 cm^{-1}, LRMS (EI) 388, 221, 167, 137, 106, 91; HRMS calcd for $C_{24}H_{24}N_2O_3$ 388.1781, found 388.1777.

2-(4-Nitrophenyl)-N-(4,4-diphenylbutyl)acetamide 8 from 7.

Following the general procedure, **7** (King, F.D.; Aliev, A.E.; Caddick, S.; Copley, R.C. *Org. Biomol. Chem*., **2009**, *7*, 3561) (0.32g, 1mmol) was reacted for 1h to give **8** (0.27g, 68% yield), identical to that obtained previously..

2-(4-Chlorophenyl)-N-(5,5-diphenylpentyl)acetamide 16 from 18a.

Following the procedure described for **20**, 2-phenylpiperidine (0.5g, 3.1mmol) was converted to 2-(4-chlorophenyl)-1-(2-phenylpiperidin-1-yl) ethanone **18a** (0.72g, 75% yield). ^{1}H-NMR (500 MHz) δ = 1.3–1.9 (5H, brm), 2.25–2.45 (1H, brm), 2.59–2.75 (0.5H, brm), 2.88–3.02 (0.5H, brm), 3.60–3.90 (2.5H, brm), 4.53–4.67 (0.5H, brm), 5.03–5.20 (0.5H, brm), 5.93–6.08 (0.5H, brm), 7.09–7.43 (9H, brm, including 7.17, 2H, d, *J* = 7.5 Hz and 7.27, 2H, d, *J* = 7.5 Hz), ^{13}C-NMR + DEPT (125 MHz) δ = 19.5 (CH_2), 25.2 (CH_2), 26.2 (CH_2), 27.0 (CH_2), 29.0 (CH_2), 38.4 (CH_2), 40.3 (CH_2), 40.7 (CH_2), 42.8 (CH_2), 50.8 (CH), 56.0 (CH), 126.3 (CH), 126.7 (CH), 127.1 (CH), 128.9 (CH), 130.2 (CH), 132.7 (C), 133.9 (C), 139.1 (C), 169.8 (C), 170.5 (C), FT-IR (neat) 2938, 1631, 1491, 1428, 1090, 1013, 806, 735, 696 cm^{-1}, LRMS (EI) 313, 188, 91; HRMS calcd for $C_{19}H_{20}ClNO$ 313.1228, found 313.1232. Following the general procedure, **18a** was converted to **16**, purified by elution with DCM (95% yield), identical to that previously described.

Attempted preparation of 2-(4-Chlorophenyl)-N-(6,6-diphenylhexyl)acetamide 19b.

By the method described for **20**, a solution of the 2-phenylperhydroazepine (0.62g, 3.5mmol) (Healy, M. A. M.; Smith, S. A.; Stemp, G. *Syn. Commun*. **1995**, *25*, 3789) was converted to 2-(4-Chlorophenyl)-1-(2-phenylperhydroazepin-1-yl)ethanone **18b** (1.0g, 85% yield), isolated as an oil. ^{1}H-NMR (500 MHz) δ = Two rotamers: 1.05 – 2.00 (5.2H, m), 2.30 – 2.40 (0.8H, tt, *J* = 8.5, 15.5 Hz), 2.89 (0.6H, t, *J* = 13.2 Hz), 3.23 (0.4H, t, *J* = 13.9 Hz), 3.50 (1.2H, s), 3.65 – 3.80 (1.2H, m), 4.49 (0.6H, d, *J* = 13.5 Hz), 4.76 (0.6H, dd, *J* = 5.9, 11.2 Hz), 5.54 (0.4H, dd, *J* = 6.0, 11.8 Hz), 7.02 – 7.40 (9H, m); ^{13}C-NMR + DEPT (125 MHz) δ = Rotamer A: 25.9 (CH_2), 29.1 (CH_2), 29.5 (CH_2), 37.2 (CH_2), 40.5 (CH_2), 42.7 (CH_2), 61.5 (CH), 125.3 (CH), 126.9 (C), 128.8 (CH), 129.0 (CH), 130.1 (CH), 132.6 (C), 133.8 (C), 142.9 (C), Rotamer B, 25.7 (CH_2), 29.4 (CH_2), 30.9 (CH_2), 40.3 (CH_2), 44.3 (CH_2), 57.8 (CH), 126.0 (CH), 127.2 (CH), 128.5 (CH), 128.6 (C), 128.7 (CH), 130.5 (C), 132.7 (C), 133.8 (C), 143.0 (C), LRMS (EI) 327, 202, 125, 91; HRMS calcd for $C_{20}H_{22}ClNO$ 327.1384, found 327.1390. Following the general procedure, **18b** (0.6g, 1.6mmol) was heated under reflux for 4h for complete consumption of **18b**. Purification on a silica column, eluting with DCM gave an inseparable mixture of compounds (0.48g) as an oil. HRMS calcd for $C_{26}H_{28}ClNO$ 405.1854, found 405.1858; calcd for $C_{20}H_{24}ClNO$ 329.1541, found 329.1545.

Preparation of 2-(4-Chlorophenyl)-1-(4-methyl-2-phenylpiperazin-1-yl)ethanone 18c.

Following the procedure described for **20**, 4-methyl-2-phenylpiperazine (0.52g, 3.0mmol) was converted to 2-(4-chlorophenyl)-1-(4-methyl-2-phenylpiperazin-1-yl)ethan-one **18c**, isolated as an oil (0.85g, 88% yield). ^{1}H-NMR (400 MHz, 333K) δ = 1.96 (1H, dt, *J* = 2.8, 11.7 Hz), 2.26 (3H, s), 2.30 (1H, dd, *J* = 4.3, 12.0 Hz), 2.74 (1H, ddt, *J* = 1.8, 2.6, 11.1), 3.15 (1H, brt, *J* = 12.1 Hz), 3.32 (1H, dt, *J* = 1.6, 12.0 Hz), 3.72 (1H, d, *J* = 15.5 Hz), 3.76 (1H, d, *J* = 15.5 Hz), 3.92 (1H, brm), 5.58 (1H, brm), 7.19 (2H, d, *J* = 8.3 Hz), 7.22 – 7.36 (5H, m), 7.40-7.50 (2H, m); ^{13}C-NMR (100 MHz, 333K) δ = 40.1, 41.0, 46.0, 52.9, 55.1, 57.3, 127.1, 127.5, 128.4, 128.7, 130.1, 132.8, 133.7, 139.6, 169.4, FT-IR (neat) 2792, 1634, 1492, 1627, 1296, 1132, 1090, 1016, 808, 782, 722, 697 cm^{-1}, LRMS (EI) 328, 175, 146, 104, 91; HRMS calcd for $C_{19}H_{21}ClN_2O$ 328.1337, found

328.1329. Heating a solution of **18c** (0.5g, 1.5 mmol) in benzene (15 ml) and triflic acid (1.5 ml) under reflux for 4h showed only starting material by TLC.

Preparation of 1 {4-[2(4-chlorophenyl)acety]-3-phenylpiperazin-1-yl}-2-(4-nitrophenyl)-ethanone 18d.

A solution of 2-phenylpiperazine (1.6g, 10mmol) and 4-nitrophenylacetic acid O-N-hydroxysuccinimide ester (2.8g, 10 mmol) in DCM (100 ml) was stirred at ambient temperatures overnight. The reaction mixture was concentrated *in vacuo* and the residue treated with EtOAc (150 ml), washed with 2M NaOH (2 x 50 ml), brine (50 ml) and dried (K_2CO_3). Filtration and concentration *in vacuo* gave the 2-(4-nitrophenyl)-1-(3-phenylpiperazin-1-yl)ethanone as a pale yellow solid (2.8g, 85% yield), mpt 108-110^oC (EtOAc/petroleum ether). ^{1}H-NMR (500 MHz) rotamer mixture δ = 1.98 (brs, 1H), 2.63 (0.4H, t, *J* = 12.5 Hz), 2.69 – 2.90 (1.6H, m), 3.00 – 3.19 (1.4H, m), 3.27 (0.4H, t, *J* = 12 Hz), 3.47 (0.6H, d, *J* = 10.5 Hz), 3.61 – 3.87 (3.6H, m), 4.61 (2H, d, *J* = 11 Hz), 7.16 – 7.50 (7H, m), 8.20 (2H, d, *J* = 8Hz); ^{13}C-NMR + DEPT (125 MHz) δ = 40.4, 42.2, 45.9, 46.4, 46.5, 49.2, 53.7, 60.1, 61.1, 123.9, 126.9, 128.0, 128.3, 128.6, 128.8, 130.0, 140.5, 140.9, 142.8, 147.0, 167.8, FT-IR (neat) 3310, 2848, 1650, 1510, 1435, 1417, 1338, 1237, 1223, 1143, 1100, 1047, 816, 765, 740, 709, 700 cm^{-1}; LRMS (EI) 325, 282, 220, 161, 132, 118, 104, 91; HRMS calcd for $C_{18}H_{19}N_3O_3$ 325.1421, found 325.1425. Structural assignment was made from the NOESY spectrum which showed no NOE between the $ArCH_2CO$ and 3-CHPh protons, but did between the $ArCH_2CO$ and the 2-CH and 6-CH protons.

Following the general procedure described for **20**, 2-(4-nitrophenyl)-1-(3-phenylpiperazin-1-yl)ethanone (0.98g, 3.0 mmol) was converted to **18d**, purified by column chromatography on SiO_2, eluting with 2% MeOH/DCM and isolated as a solid, m.pt 63-65^oC (1.3g, 90% yield). ^{1}H-NMR (400 MHz, d^6-DMSO, 373K) δ = 3.21 – 3.37 (2H, brm), 3.48 – 3.90 (6H ,m), 4.04 – 4.14 (1H, brm), 4.35 – 4.55 (1H, brm), 5.51 (1H, brs), 7.20 – 7.38 (11H, m), 8.06 (2H, d, J = 8.5 Hz); ^{1}H-NMR (600 MHz, d^6-DMSO, 298 K) δ = 37.2, 37.5, 38.6, 38.9, 40.1, 41.1, 41.2, 42.1, 42.5, 43.3, 44.7, 45.1, 47.5, 48.3, 51.7, 53.5, 56.1, 56.8, 65.0, 123.2, 126.2, 126.4, 127.0, 127.4, 128.2, 128.4, 128.7, 128.9, 130.4, 130.7, 130.9, 131.2, 134.7, 134.8, 138.4, 139.3, 143.9, 146.1, 146.2, 168.6, 169.5, 169.8, 170.0, FT-IR (neat) 1638, 1515, 1413, 1344, 1089, 1016, 857, 810, 732, 698 cm^{-1}, LRMS no molecular ion could be detected, (EI) and (CI), 267, 167, 100. Heating a solution of **18d** (0.75g, 1.5 mmol) in benzene (15 ml) and triflic acid (1.5 ml) for 4h showed only starting material by TLC.

Attempted preparation of 2-(4-chlorophenyl)-N-[2-(2,2-diphenylethoxy)ethyl]acetamide 19e.

Following the procedure described for **20**, 2-phenylmorpholine (Alexander, R.; Balasundaram, A.; Batchelor, M.; Brookings, D.; Crepy, K.; Kulisa, C.; Turner, J.; Whitcombe, I.; Crabbe, T.; Gill, A.; Hutchinson, G.; Merriman, M.; Deltent, M-F.; Driessens, F.; Harris, S.; Mistry, P.; Parton, T.; Wright, S. *Bioorg. Med. Chem. Lett.* **2008,** *18*, 4326) (0.98g, 6.0 mmol) was converted to **18e**, purified by column chromatography on SiO_2, eluting with a solvent gradient, starting with 1:1 petroleum ether/DCM to 1% MeOH/DCM (1.27g, 67% yield). ^{1}H-NMR (400 MHz, 333K) δ = 3.21 (1H, brt, *J* = 11.2 Hz), 3.43 (1H, brt, *J* = 11.7 Hz), 3.62 – 3.90 (5H, m), 4.37 (1H, d, *J* = 12.1 Hz), 5.41 (1H, brm), 7.16 (2H, d, *J* = 8.4 Hz), 7.22 – 7.48 (7H, m); ^{13}C-NMR + DEPT (100 MHz, 333K) δ = 39.9 (CH_2), 41.0 (CH_2), 53.1 (CH), 66.8 (CH_2), 68.9 (CH_2), 127.5 (CH), 127.6 (CH), 128.6 (CH), 128.8 (CH), 130.2 (CH), 132.8 (C), 133.5 (C), 138.6 (C), 169.5 (C), FT-IR (neat) 2857, 1635, 1492, 1420, 1278, 1223, 1118, 1091, 1052, 1029, 1015, 807, 727, 697 cm^{-1}, LRMS (EI) 315, 190, 162, 132, 125, 104; HRMS calcd for $C_{18}H_{18}ClNO_2$ 315.1020, found 315.1012. Reaction of **18e** (0.43g, 1.4 mmol) following the general procedure, heating under reflux for 6h showed no starting material by TLC and gave a mixture of products which were separated by column chromatography on SiO_2, initially eluting with DCM to give 1,1,2-triphenylethane (0.27g, 75% yield), ^{1}H-NMR (500 MHz) δ = 3.40 (2H, d, *J* = 7.8 Hz), 4.27 (1H, d, J = 7.8 Hz), 7.04 (2H, dd, *J* = 1.5, 8.3 Hz), 7.11 – 7.30 (13H, m); ^{13}C-NMR + DEPT (125 MHz) δ = 42.2 (CH_2), 53.2 (CH), 126.0 (CH), 126.3 (CH), 128.2 (CH), 128.5 (CH), 129.2 (CH), 140.4 (C), 144.6 (C), identical to the literature report (Fessard, T.C., Motoyoshi, H., Carreira, E.M. *Angew. Chemie (Int. Edn.)* **2007**, *46*, 2078) mpt 50-52^oC (MeOH) (lit. 52-53^oC Alexander, L.L., Fuson, R.C. *J. Amer. Chem. Soc*, **1936,** *58*, 1745). Further elution with

0.5% MeOH/DCM gave a mixture of unidentified material and finally elution with 4% MeOH/DCM gave 2-(4-chlorophenyl)-N-(2-hydroxyethyl)acetamide (0.05g, 17% yield) ^{1}H-NMR (500 MHz) δ = 2.59 (1H, brs), 3.38 (2H, q, *J* = 5.5 Hz), 3.54 (2H, s), 3.67 (2H, t, *J* = 5.5 Hz), 7.20 (2H, d, *J* = 8.5 Hz), 7.32 (2H, d, *J* = 8.5 Hz), ^{13}C-NMR + DEPT (125 MHz) δ = 42.7 (CH_2), 42.9 (CH_2), 62.3 (CH_2), 129.2 (CH), 130.8 (CH_2), 133.2 (C), 133.5 (C), 171.8 (C).

Attempted preparation of 2-(4-chlorophenyl)-N-[2-(2,2-diphenylethylsulfanyl)-ethyl]acetamide 19f.

Following the procedure described for **20**, 3-phenylthiomorpholine (Ziakas, G.N., Rekka, E.A., Gavalas, A.M., Eleftheriou, P.T., Kourounakis, P.N. *Bioorg. Med. Chem*. **2006**, *14*, 5616) (0.9g, 5.0 mmol) was converted to **18f**, purified by column chromatography on SiO_2, eluting with a solvent gradient, starting with 1:1 petroleum ether/DCM to DCM (1.3g, 70% yield). ^{1}H-NMR (400 MHz, 333K) δ = 2.45 (1H, d, *J* = 13 Hz), 2.71 (1H, t, *J* = 10.5 Hz), 3.09 (2H, d, *J* = 3.5 Hz), 3.21 (1H, t, *J* = 12.0 Hz), 3.73 (1H, d, *J* = 15 Hz), 3.78 (1H, d, *J* = 15 Hz), 4.10 – 4.50 (1H, brm), 5.50 – 6.00 (1H, brm), 7.21 (2H, d, *J* = 8.5 Hz), 7.25 – 7.42 (7H, m); ^{13}C-NMR (125 MHz, 300K) δ = 27.0, 27.5, 40.7, 50.1, 126.5, 127.4, 129.1, 130.2, 133.0, 133.3, 137.9; LRMS (EI) 331, 179, 125, 104; HRMS calcd for $C_{18}H_{18}ClNOS$ 331.0792, found 331.0783. Following the general procedure, heating a solution of **18f** (0.7g, 2.1 mmol) under reflux for 1h showed no starting material by TLC and gave a mixture of products which were separated by column chromatography on SiO_2, initially eluting with DCM to give 1,1,2-triphenylethane (0.4g, 75% yield), identical to that isolated from the reaction of **18e**. Further elution with more polar solvents gave mixtures of unidentified compounds.

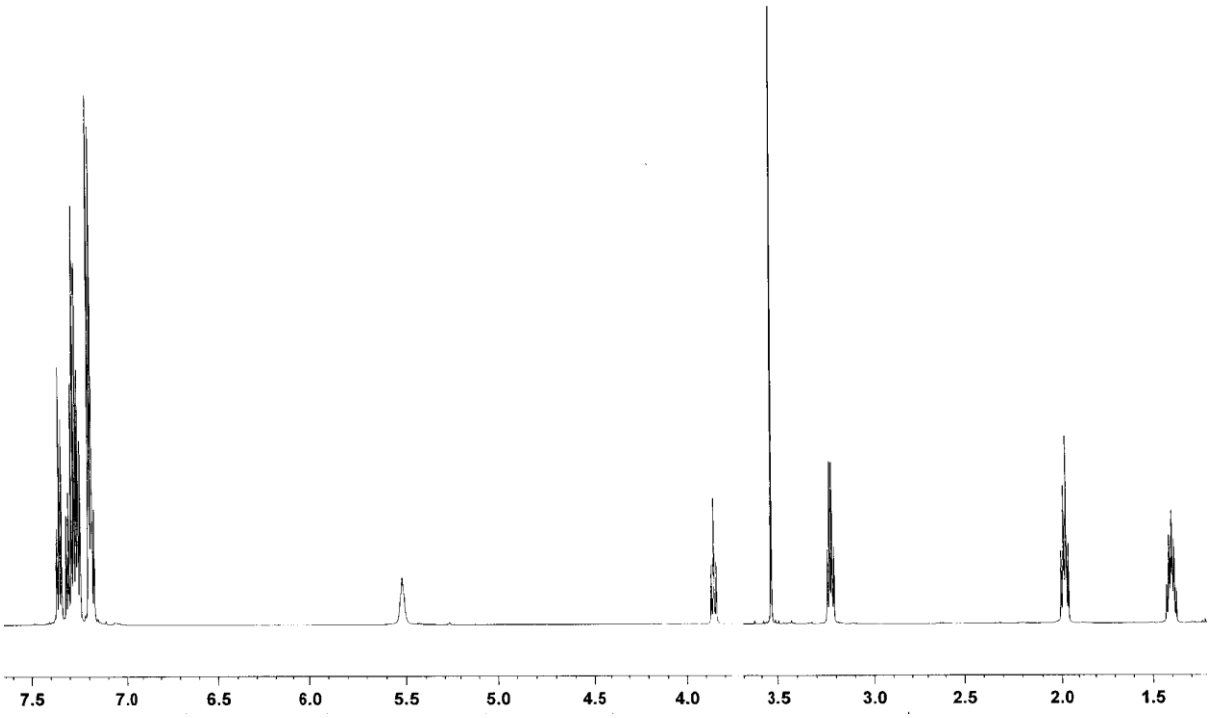
N-(4,4-Diphenylbutyl)-4-methylbenzenesulfonamide 12 from 22.

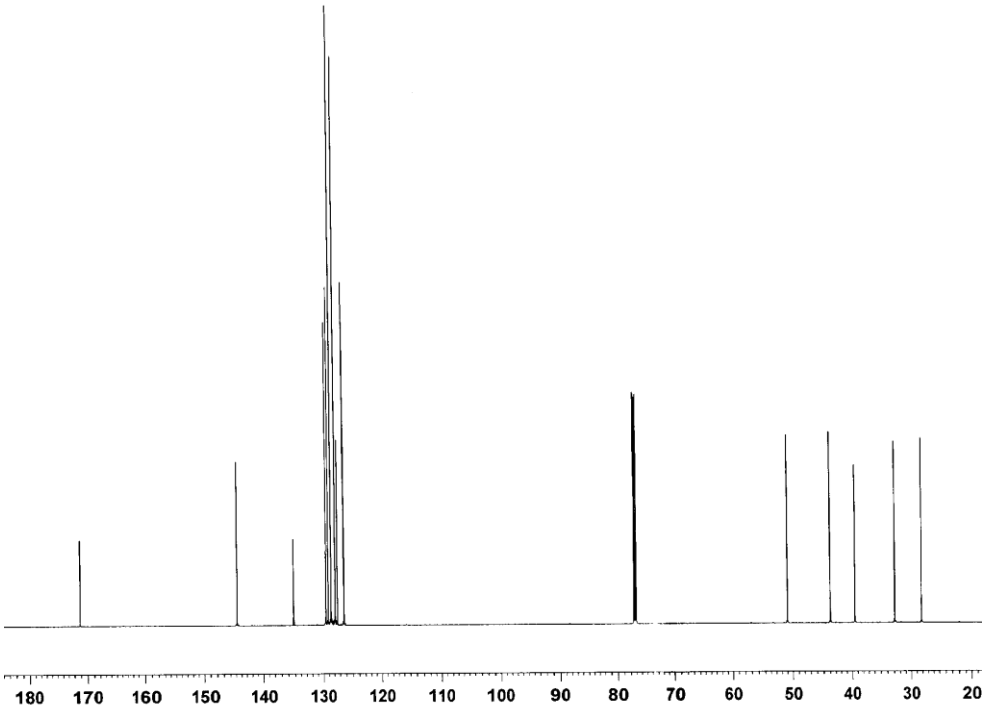
Following the procedure described for **20**, 2-phenylpyrrolidine (0.3g, 2mmol) was reacted with tosyl chloride (0.38g, 2mmol) and triethylamine (0.5ml, 2.5mmol) to give N-(p-methylphenylsulfonyl-2-phenylpyrrolidine **22**, (0.6g, ~100% yield) as a white solid, m.pt. 97-99^{o}C, pure by NMR and TLC and used without further purification; ^{1}H-NMR (500 MHz) δ = 1.60 – 1.70 (1H, m), 1.77 – 1.90 (2H, m), 1.93 – 2.03 (1H, m), 2.42 (3H, s), 3.38 – 48 (1H, m), 3.58 – 3.64 (1H, m), 4.78 (1H, dd, *J* = 4.0, 8.2 Hz), 7.18 – 7.32 (7H, m), 7.67 (2H, d, *J* = 8.2 Hz); ^{13}C-NMR + DEPT (125 MHz) δ = 21.6 (CH3), 24.0 (CH2), 35.8 (CH2), 49.4 (CH2), 63.3 (CH), 126.2 (CH), 127.1 (CH), 127.6 (CH), 128.4 (CH), 129.6 (CH), 135.2 (C), 143.1 (C), 143.4 (C); FT-IR (neat) 1449, 1330, 1155, 1091, 999, 815, 743, 700, 666 cm^{-1}. Following the procedure, **22** (0.3g, 1mmol), with heating under reflux for 1h, was converted into **22**, purified on a silica column, eluting with DCM (0.35g, 92% yield), identical to that previously prepared.

Preparation of 12 and 27 from 26.

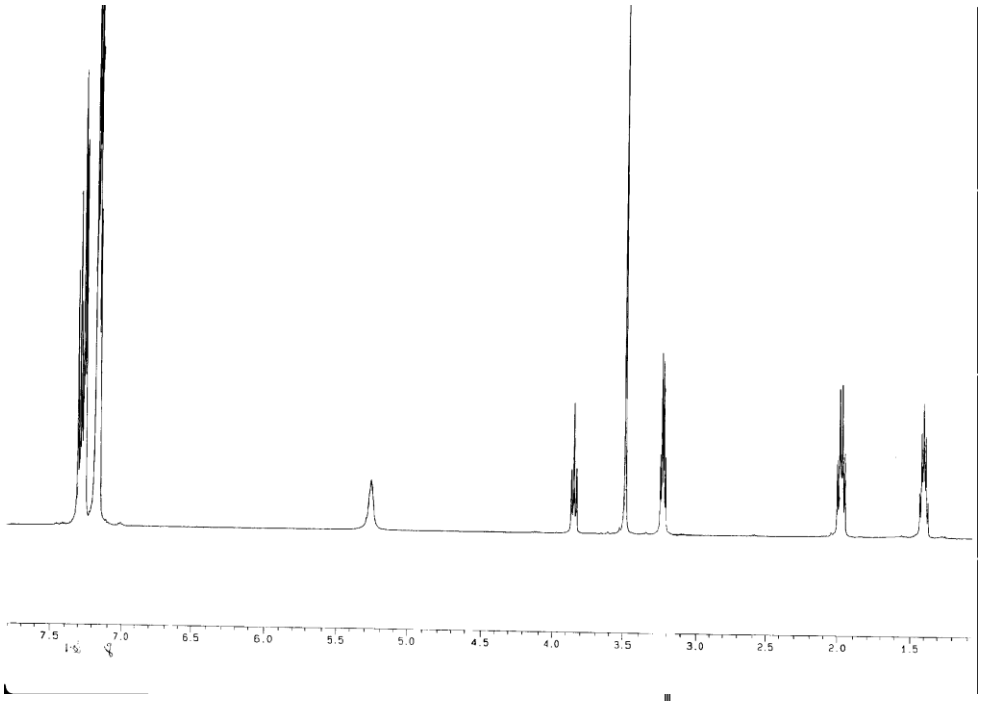
Following the general procedure, a solution of **26** (0.48g, 2 mmol) was stirred and heated under reflux in benzene (30 ml) and triflic acid (4 ml, 20 mmol) for 1h, until no **26** remained by TLC. Work-up and purification by column chromatography on silica, eluting with 20% petroleum ether/DCM gave **12** (0.26g, 35% yield), identical to that prepared before. Further elution with DCM gave **27** as an oil (0.13g, 30% yield),

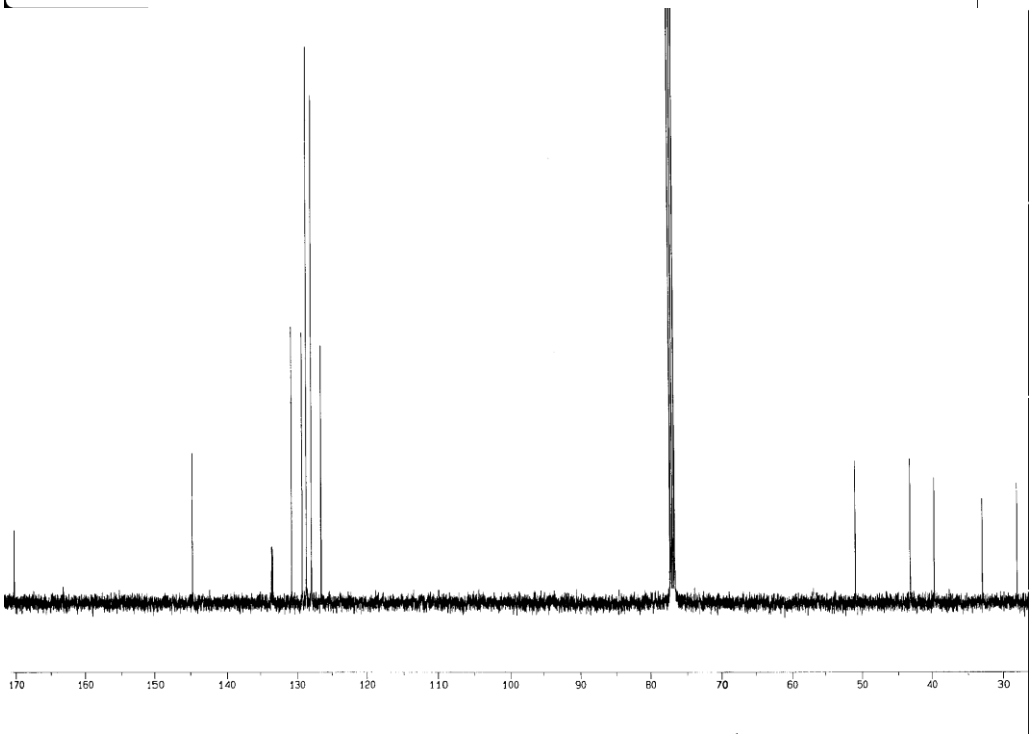
^{1}H and ^{13}C NMR spectra for **3**



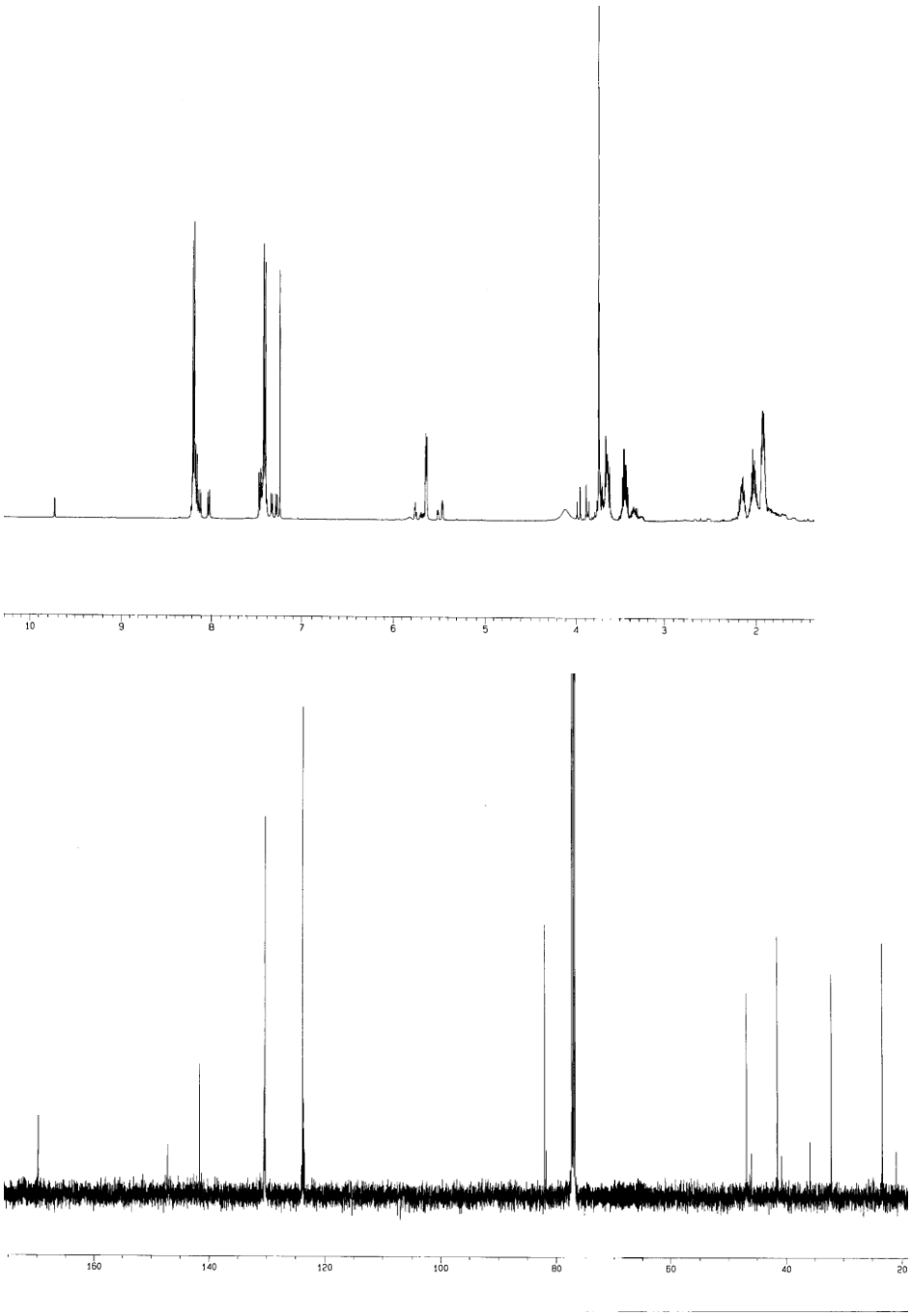


^{1}H and ^{13}C NMR spectra for **6**

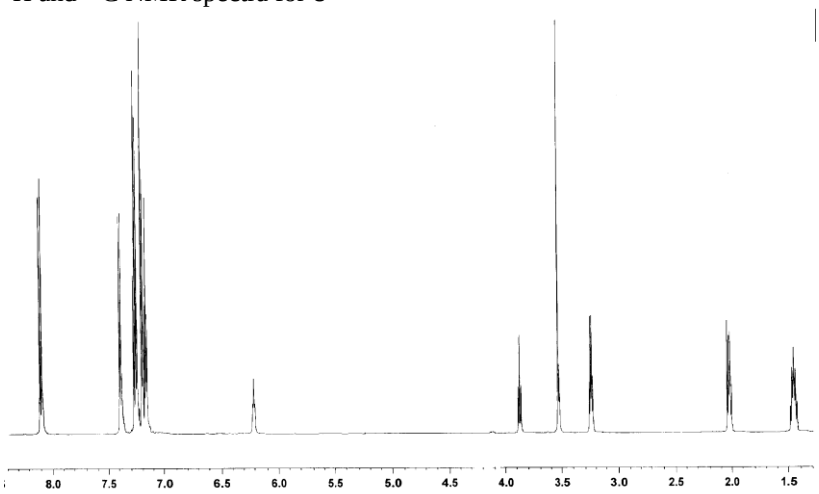


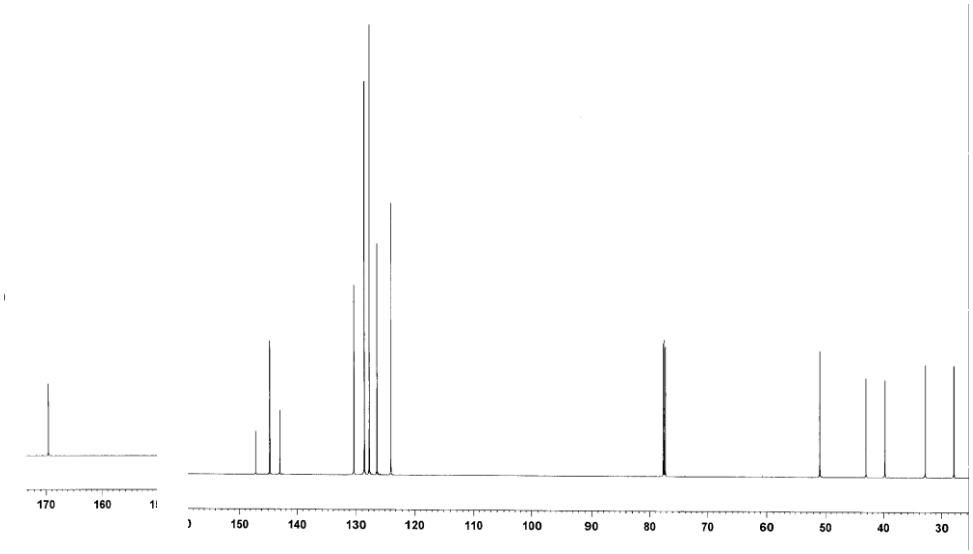


^{1}H and ^{13}C NMR spectra for **4c**

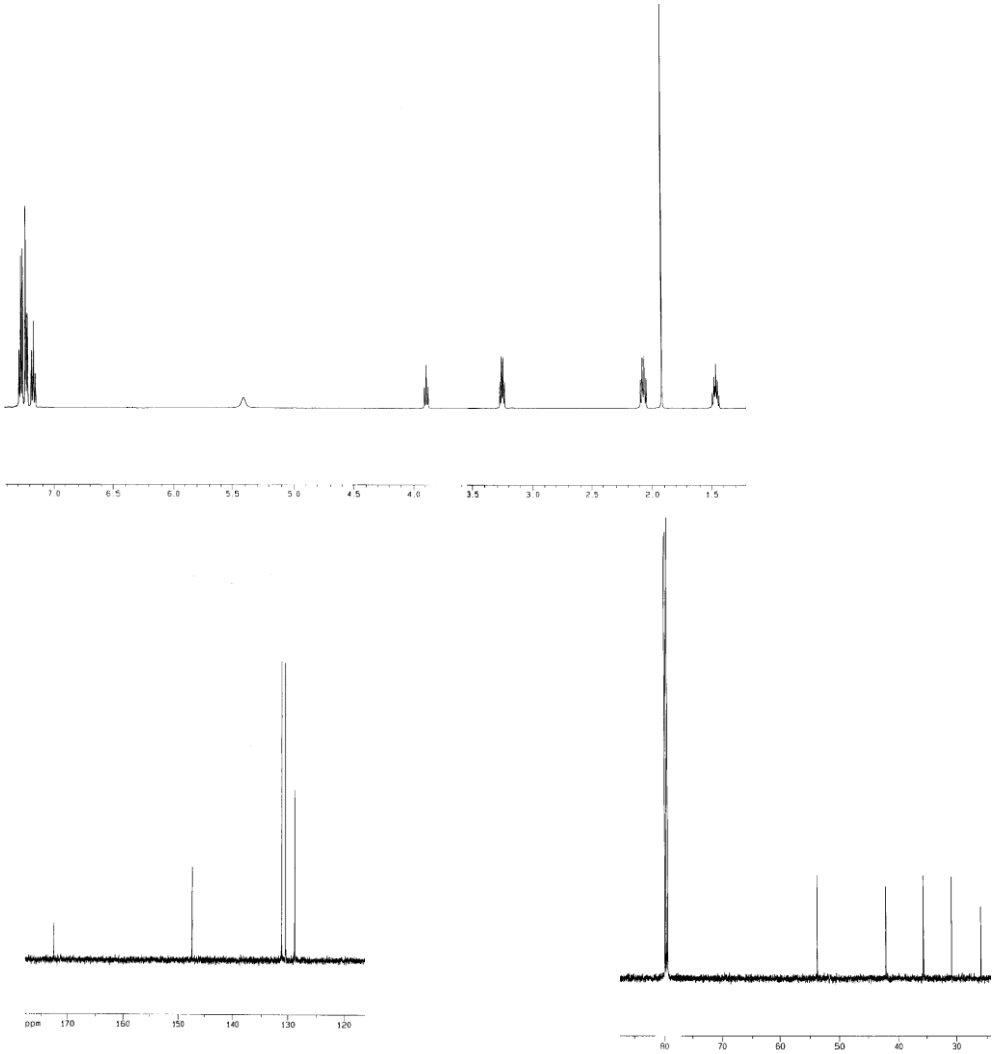


^{1}H and ^{13}C NMR spectra for **8**

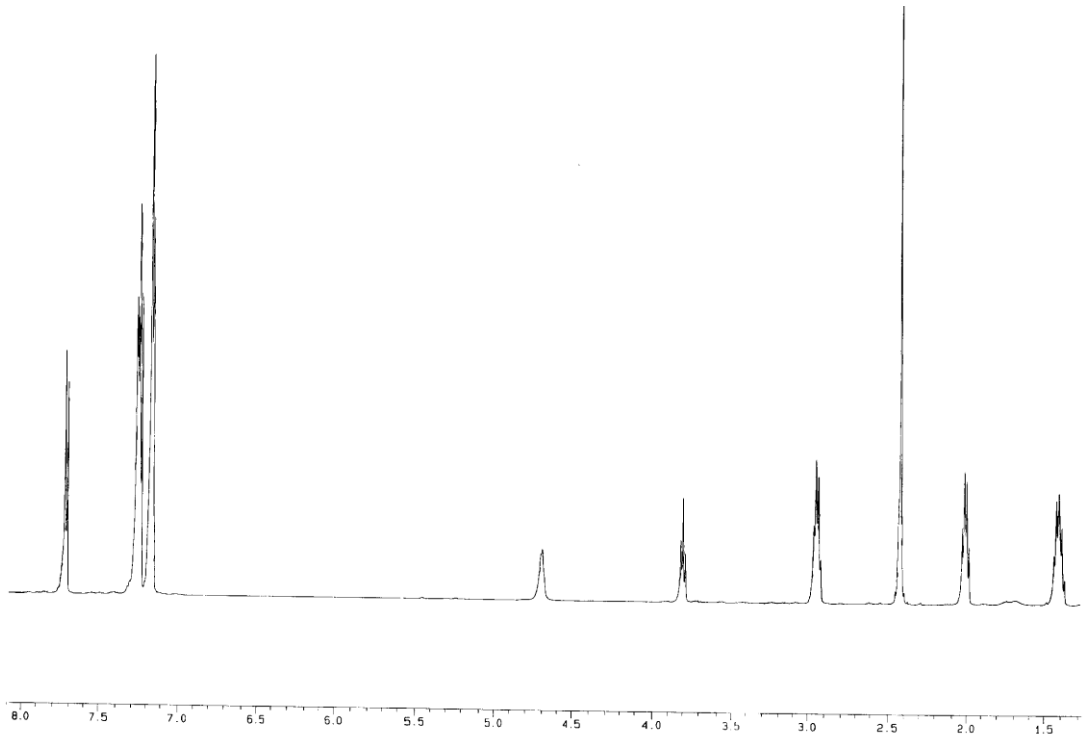


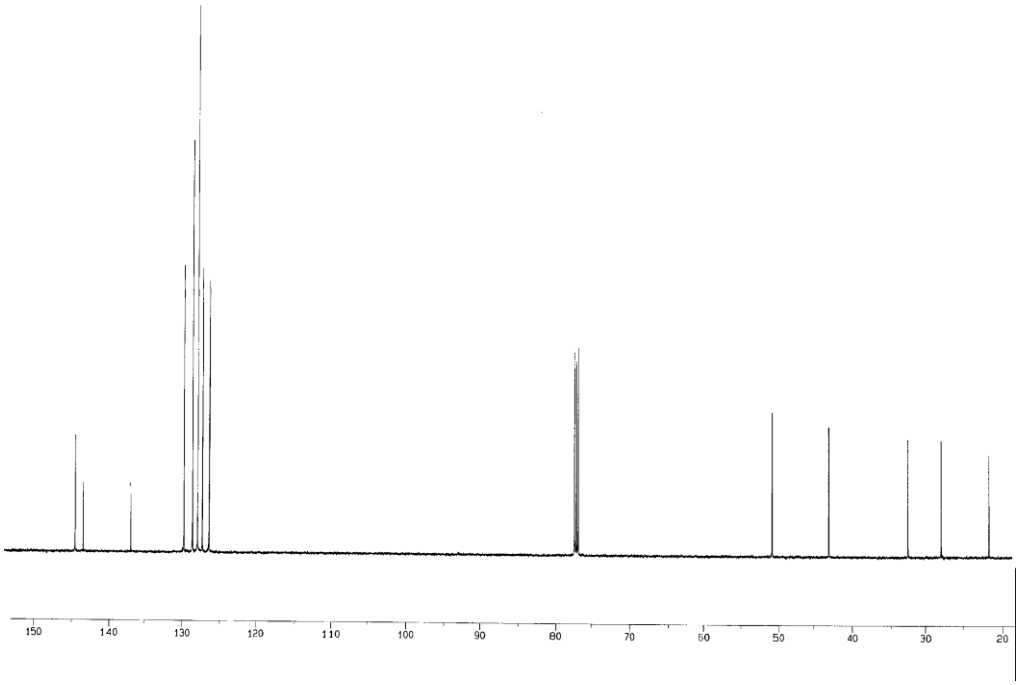


^{1}H and ^{13}C NMR spectra for **10**

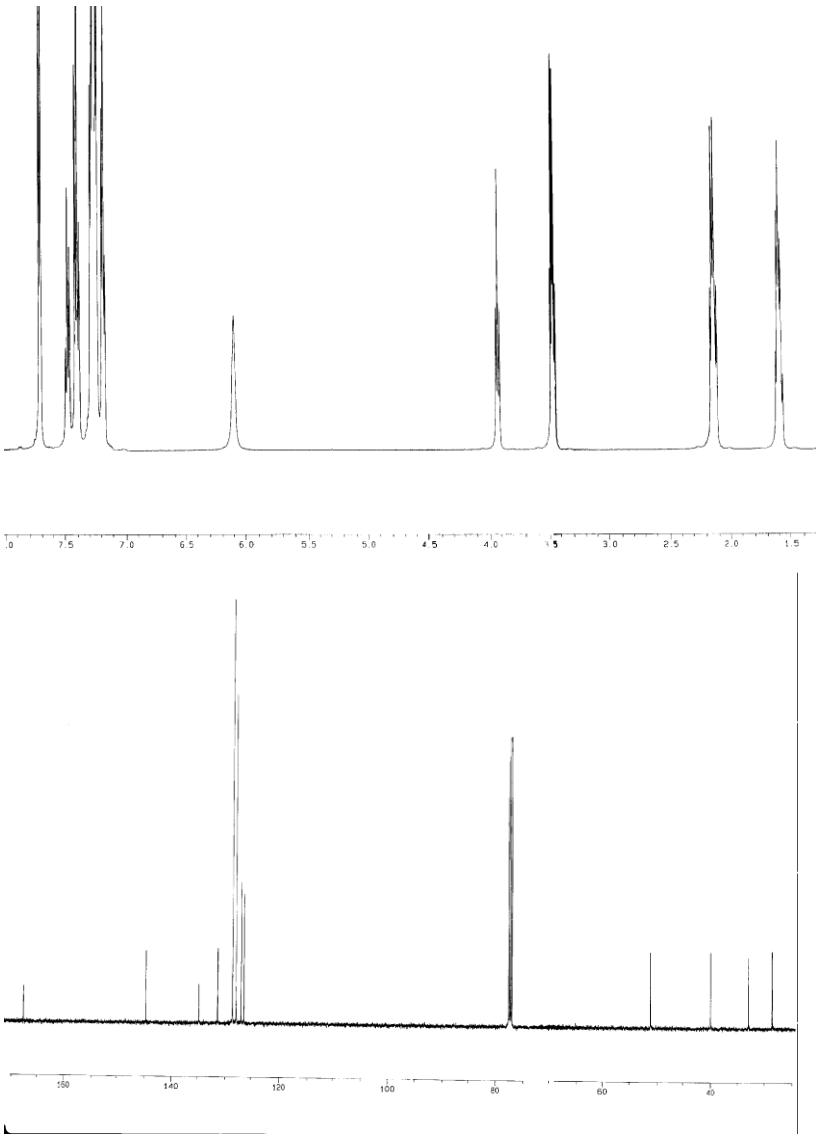


^{1}H and ^{13}C NMR spectra for **12**

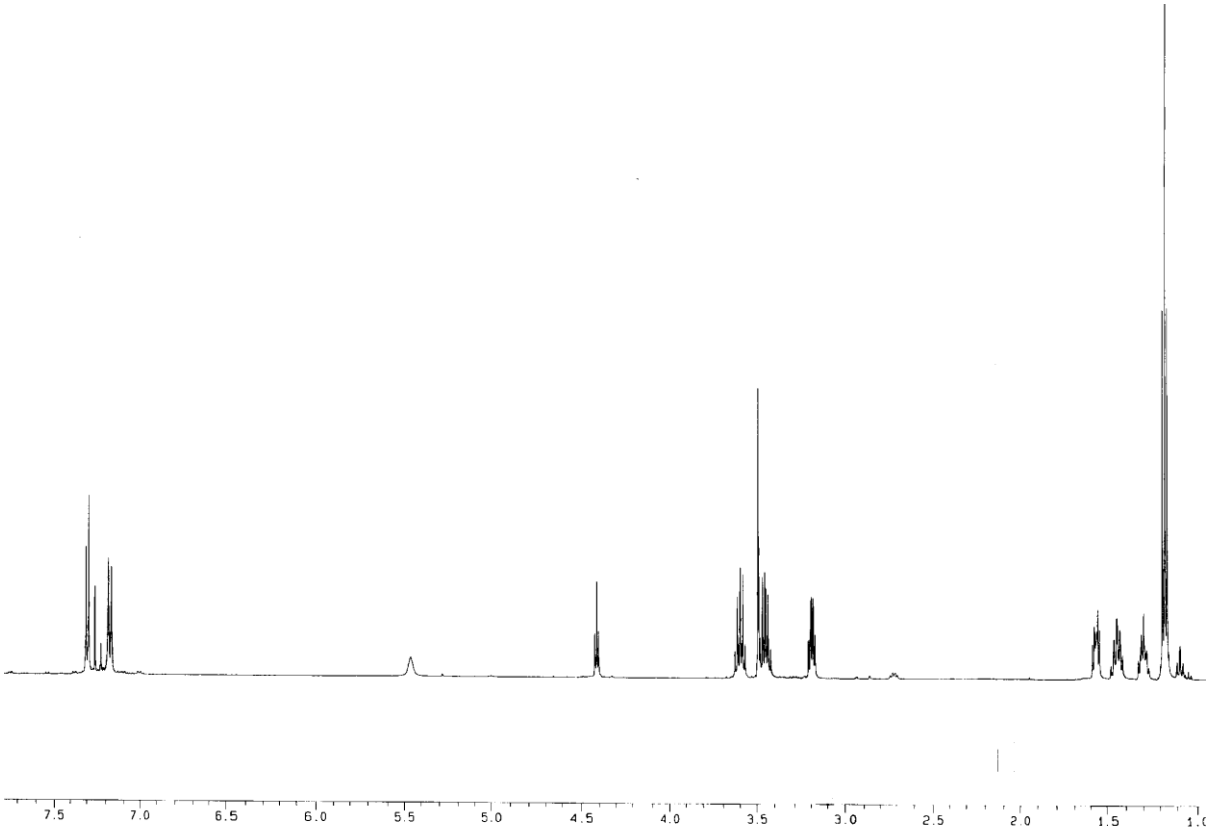


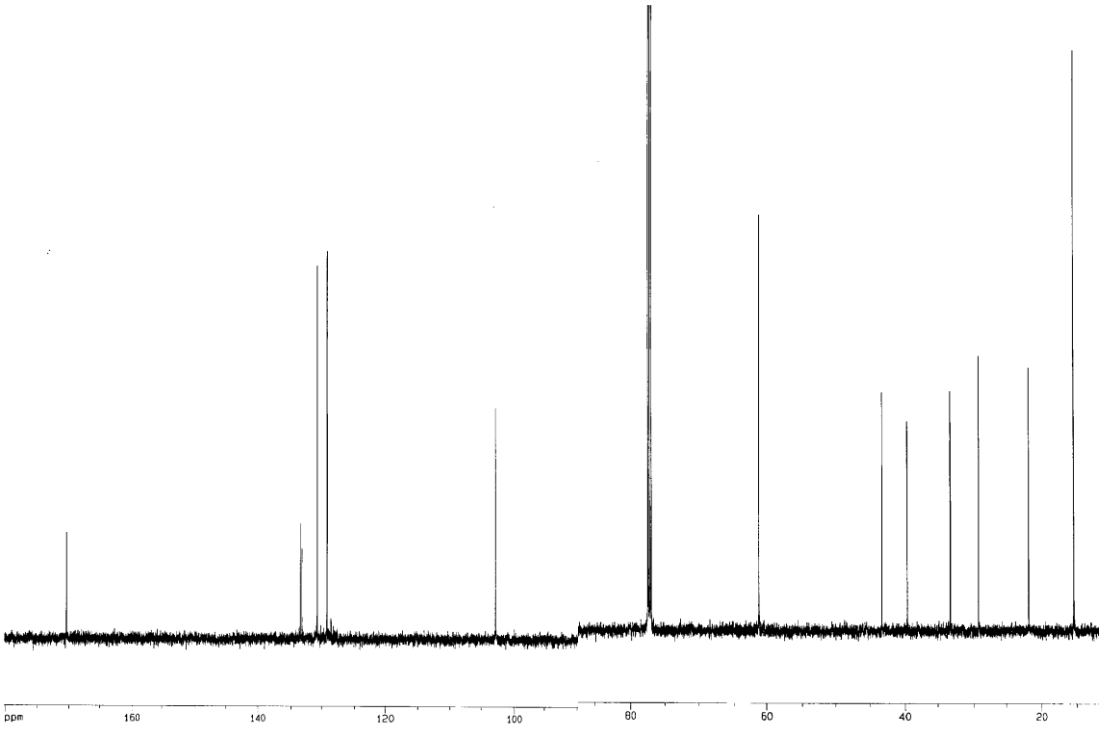


^{1}H and ^{13}C NMR spectra for **14**

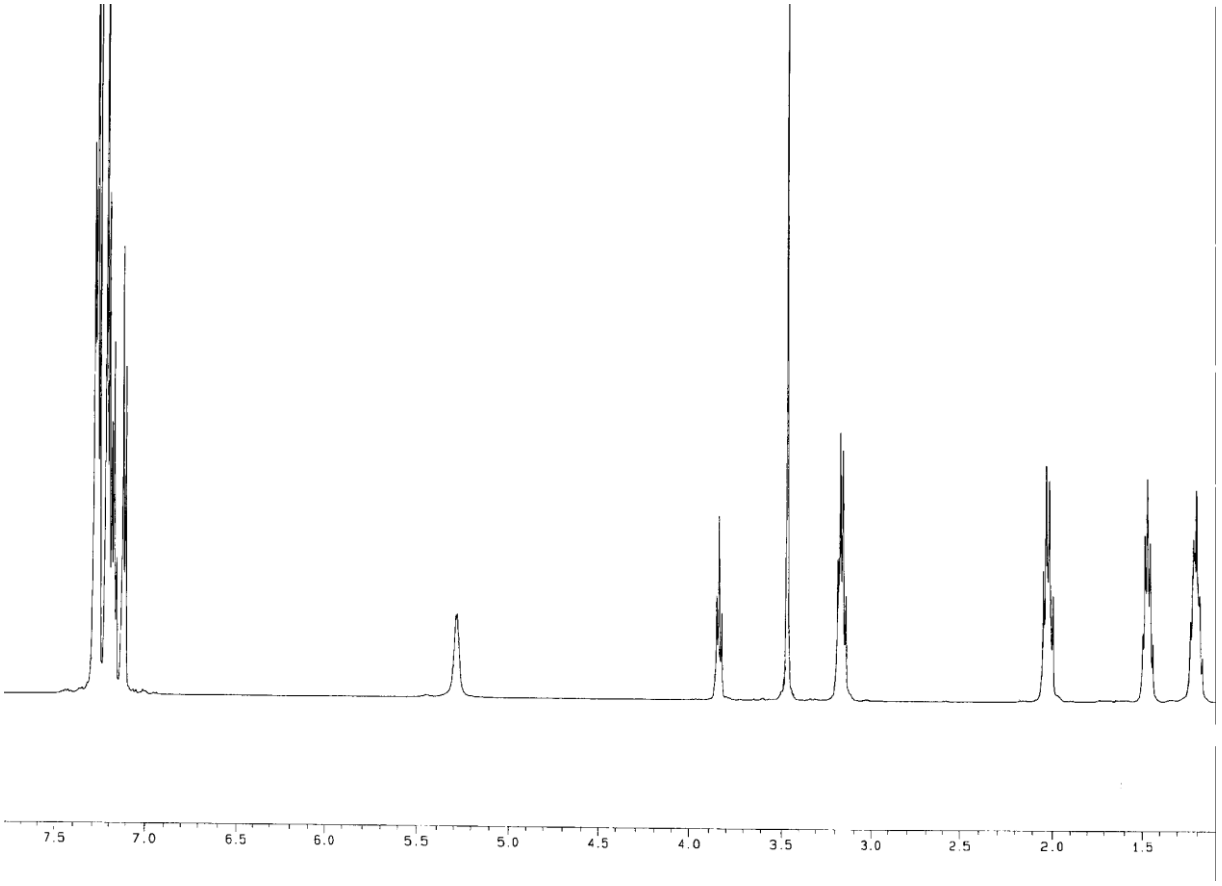


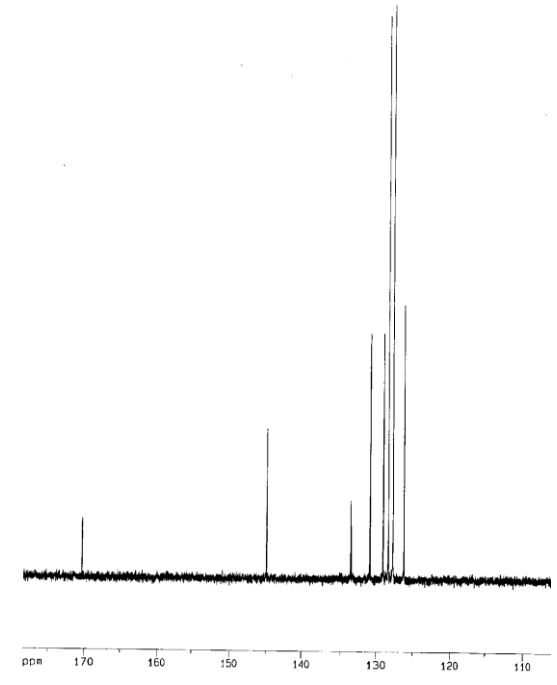
^{1}H and ^{13}C NMR spectra for **15**

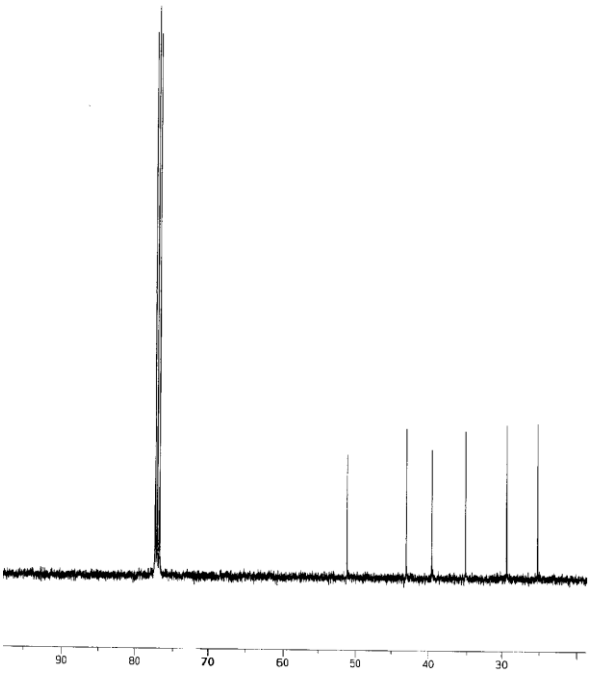




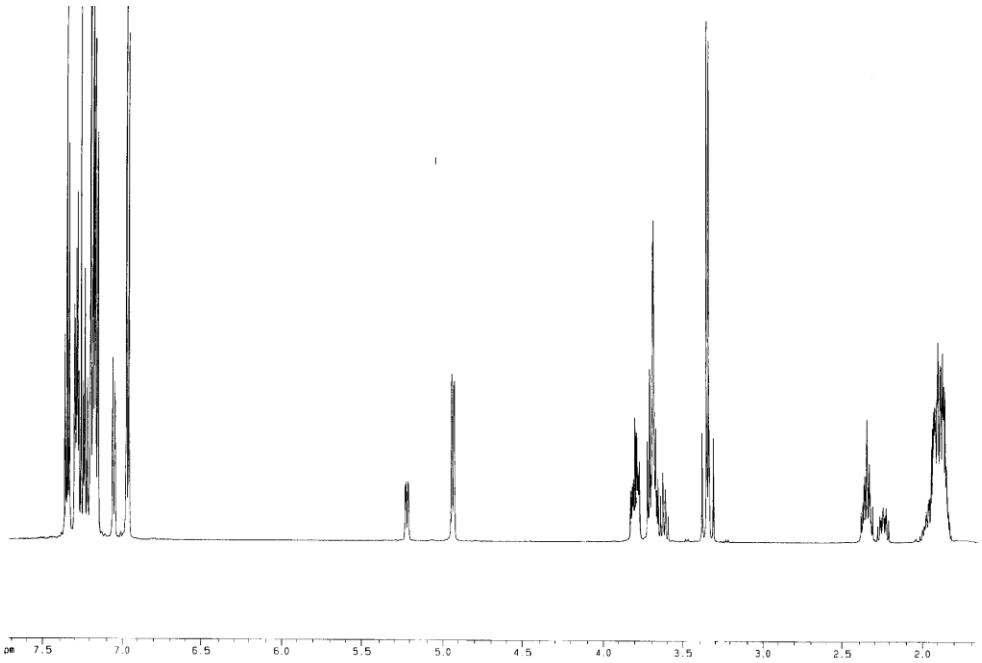
^{1}H and ^{13}C NMR spectra for **16**

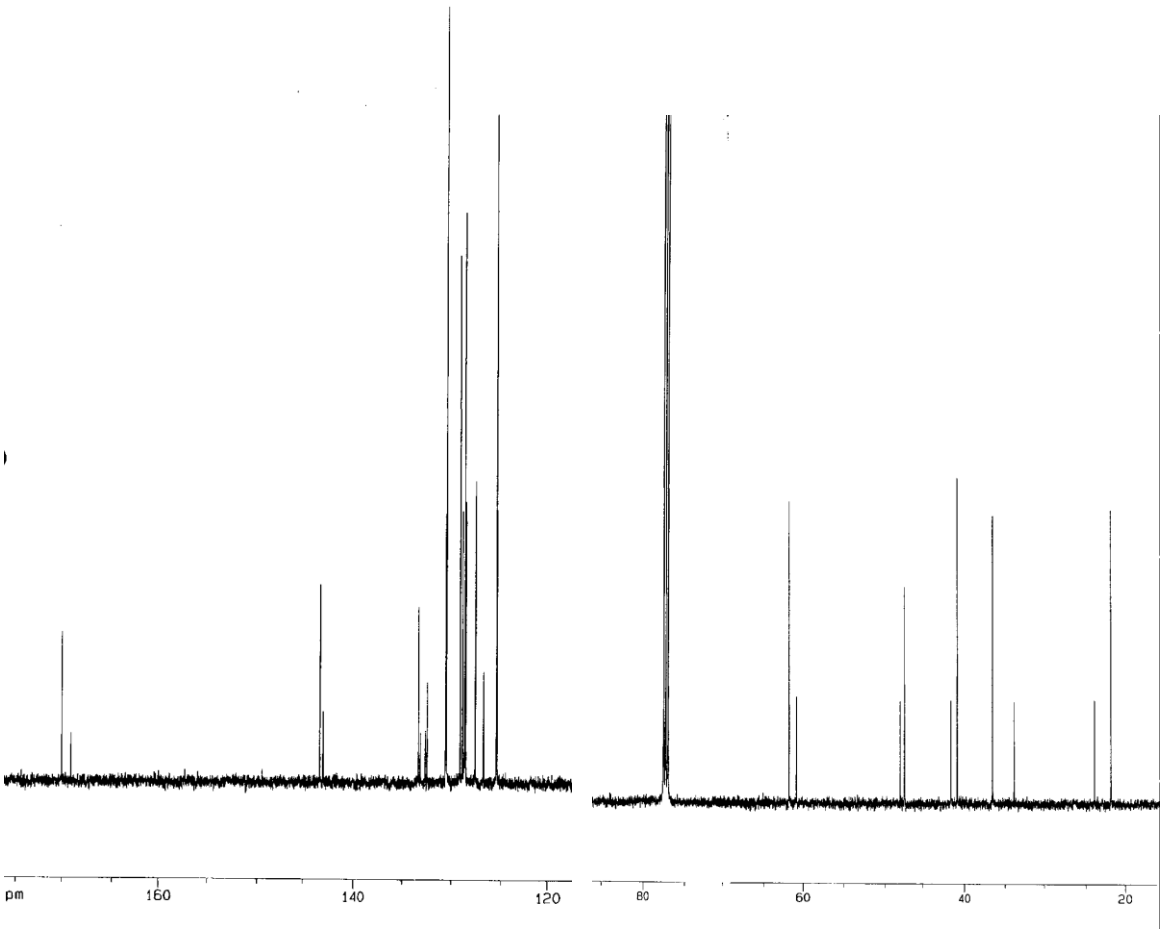




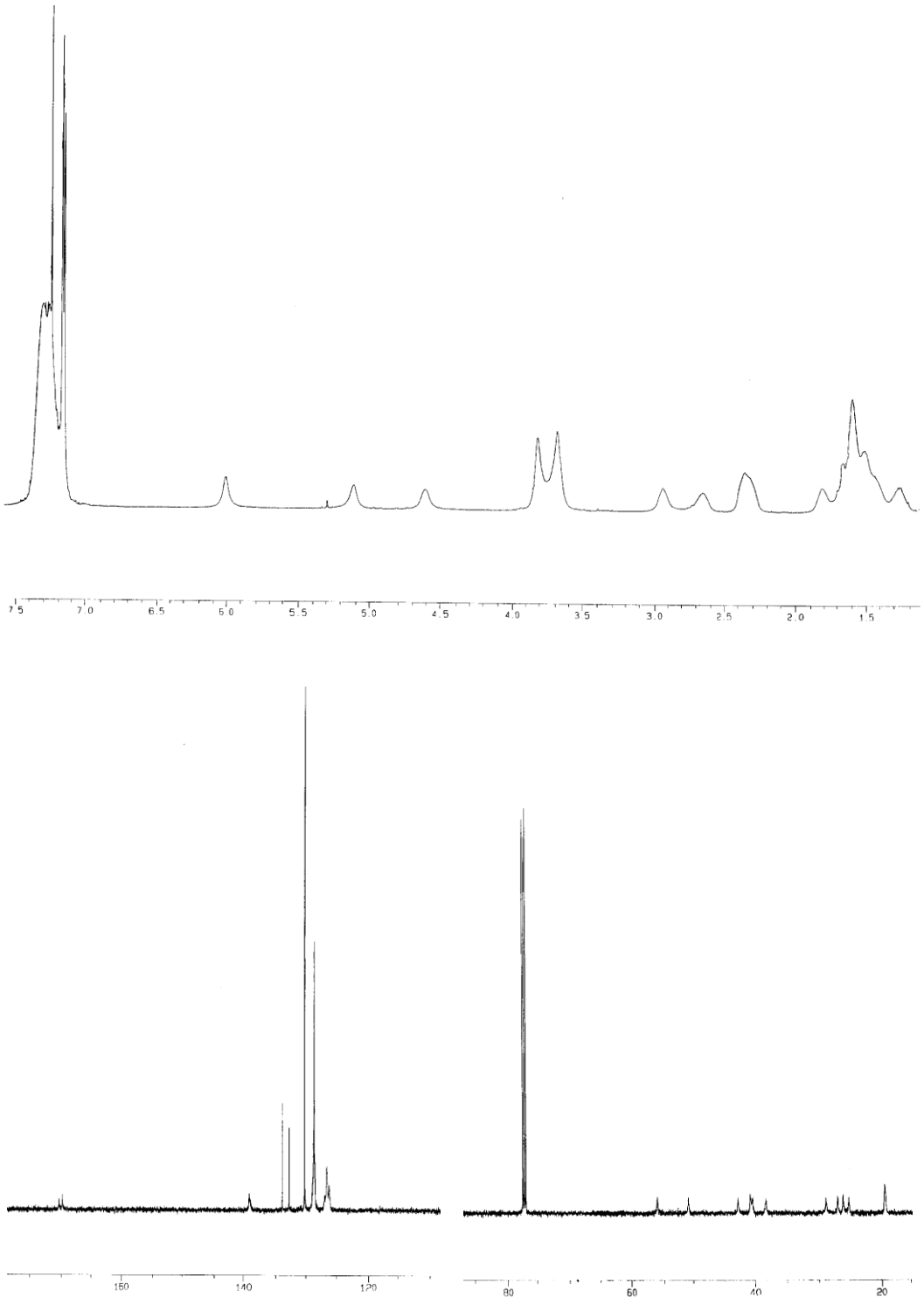


^{1}H and ^{13}C NMR spectra for **17**

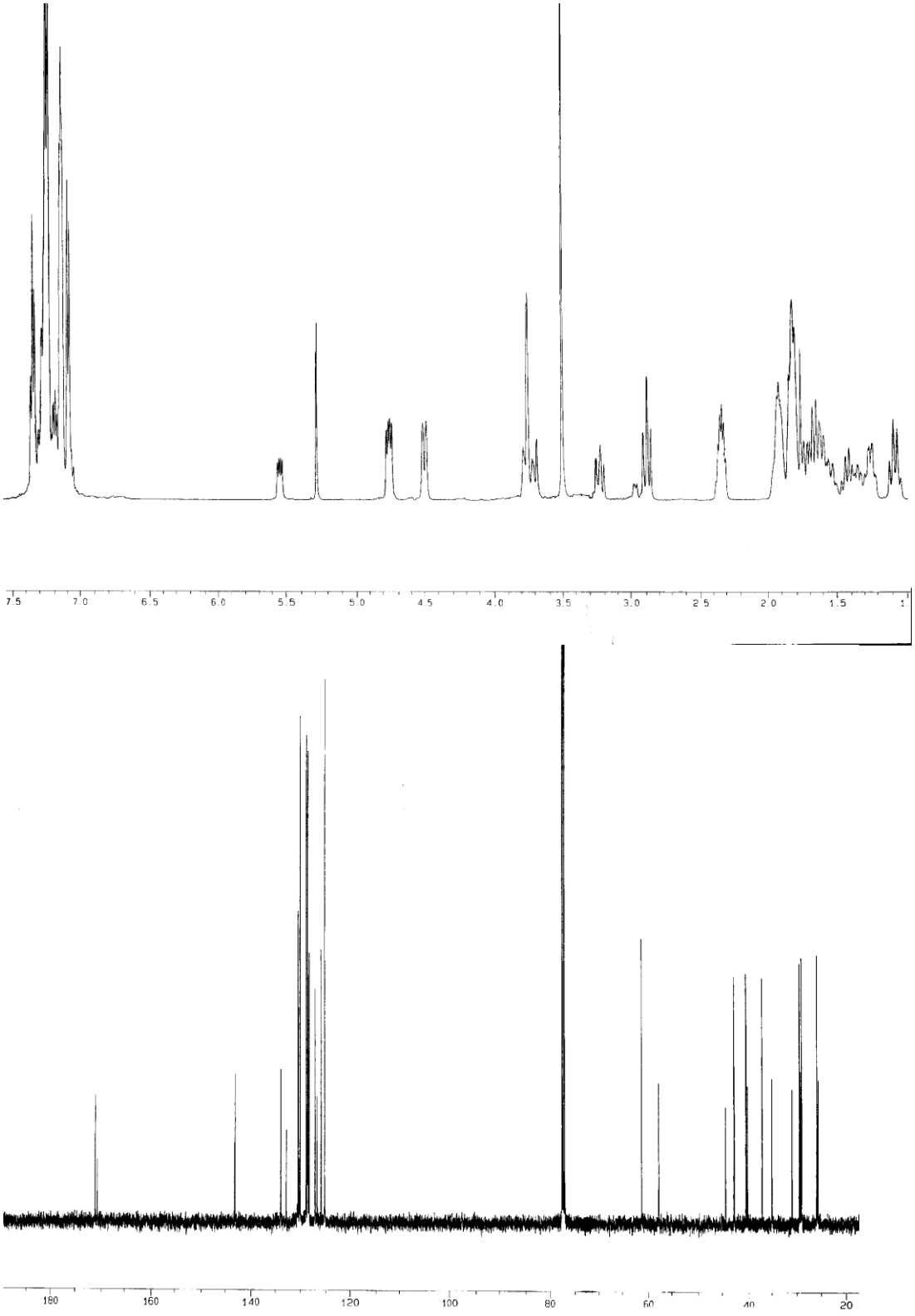




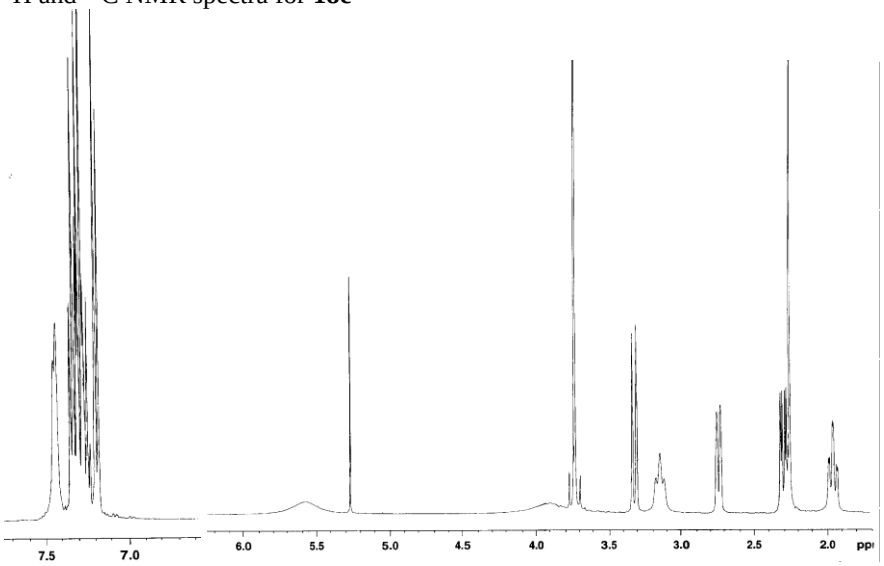
^{1}H and ^{13}C NMR spectra for **18a**

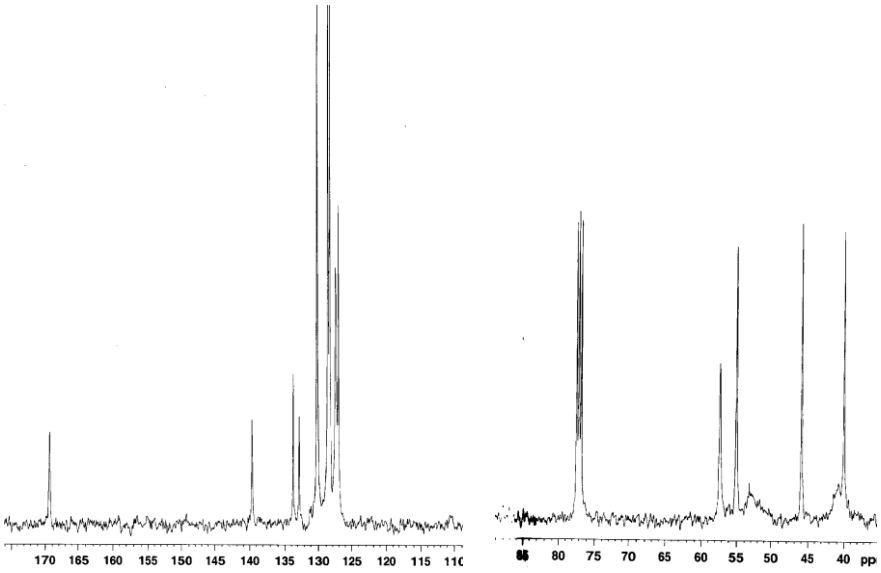


^{1}H and ^{13}C NMR spectra for **18b**

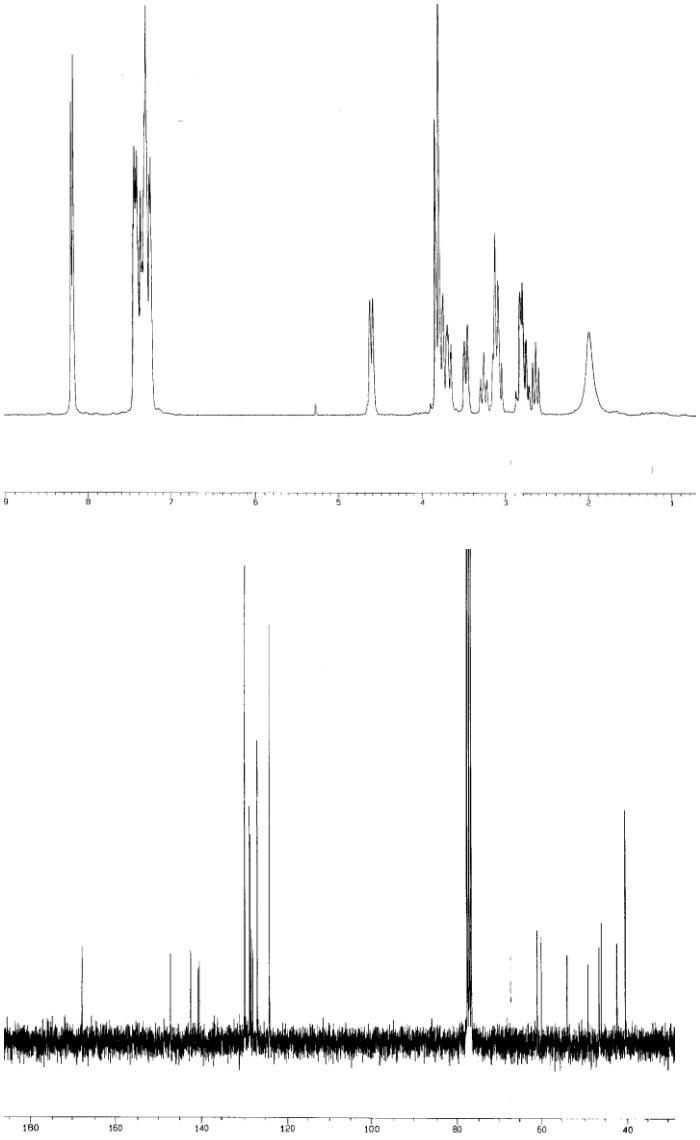


^{1}H and ^{13}C NMR spectra for **18c**

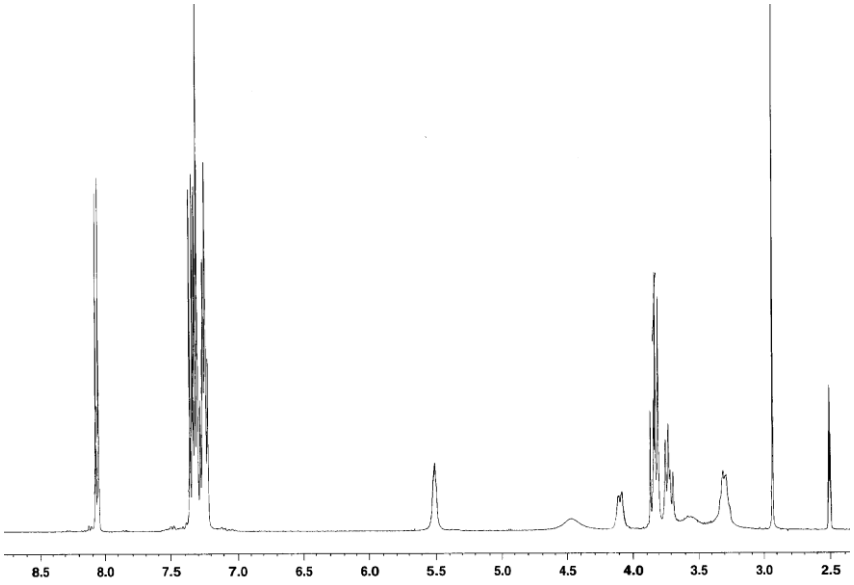


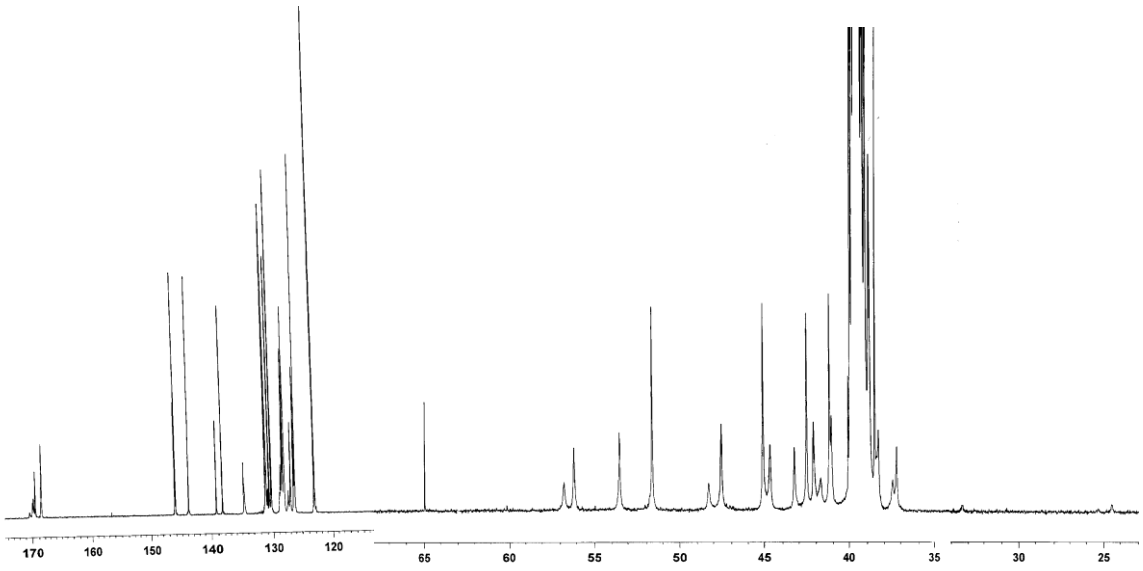


^{1}H and ^{13}C NMR spectra for 2-(4-nitrophenyl)-1-(3-phenylpiperazin-1-yl)ethanone

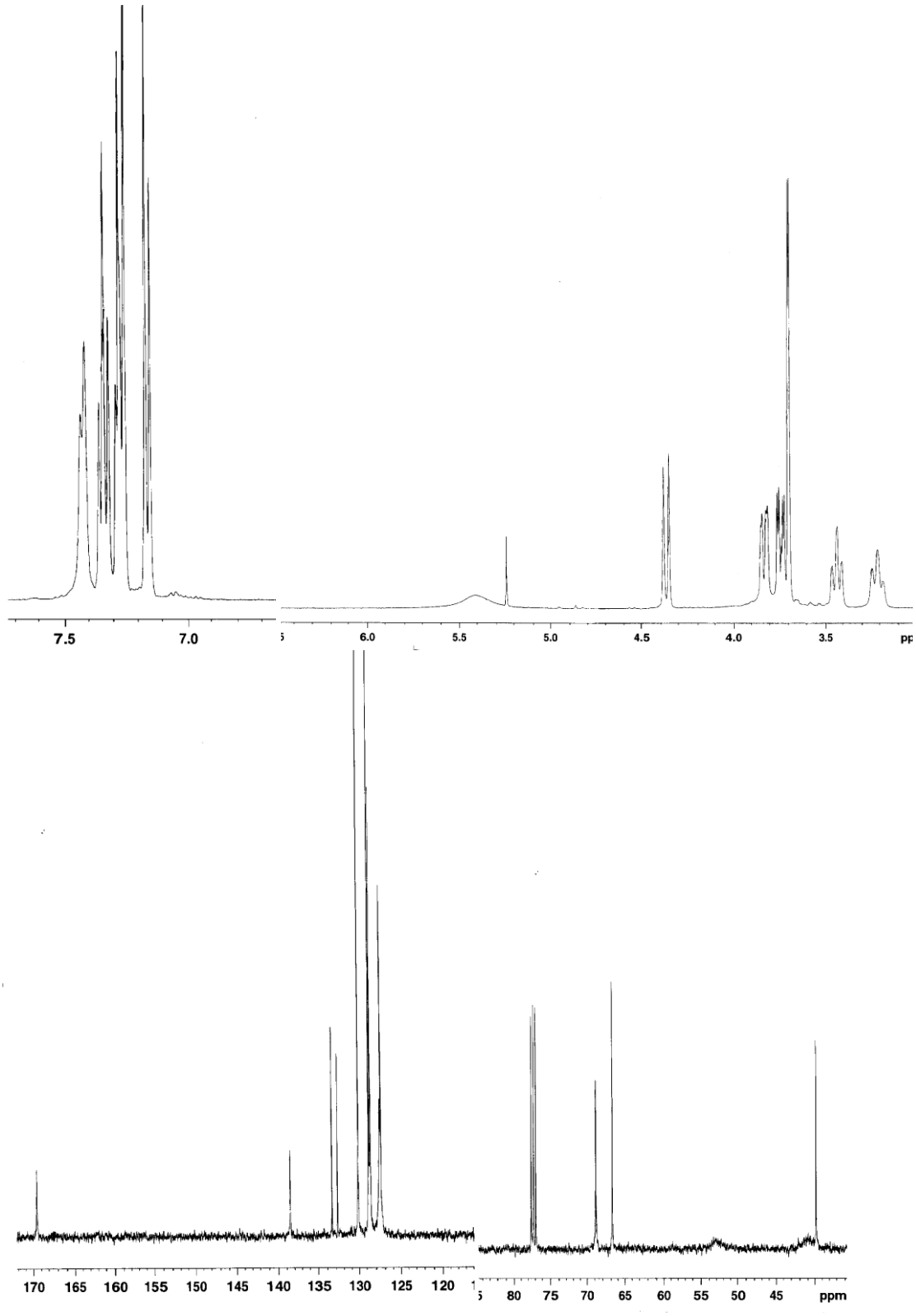


^{1}H and ^{13}C NMR spectra for **18d**

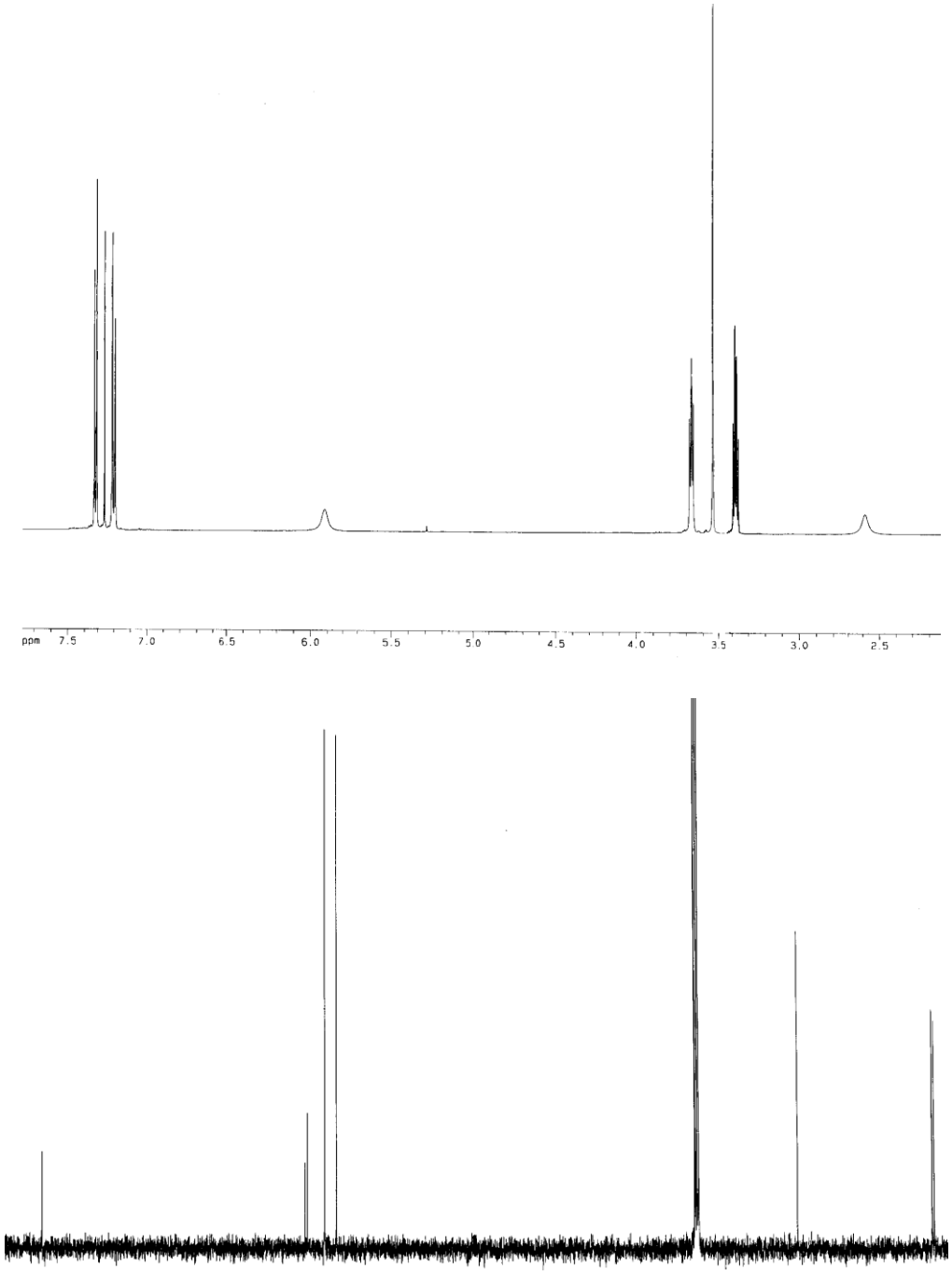




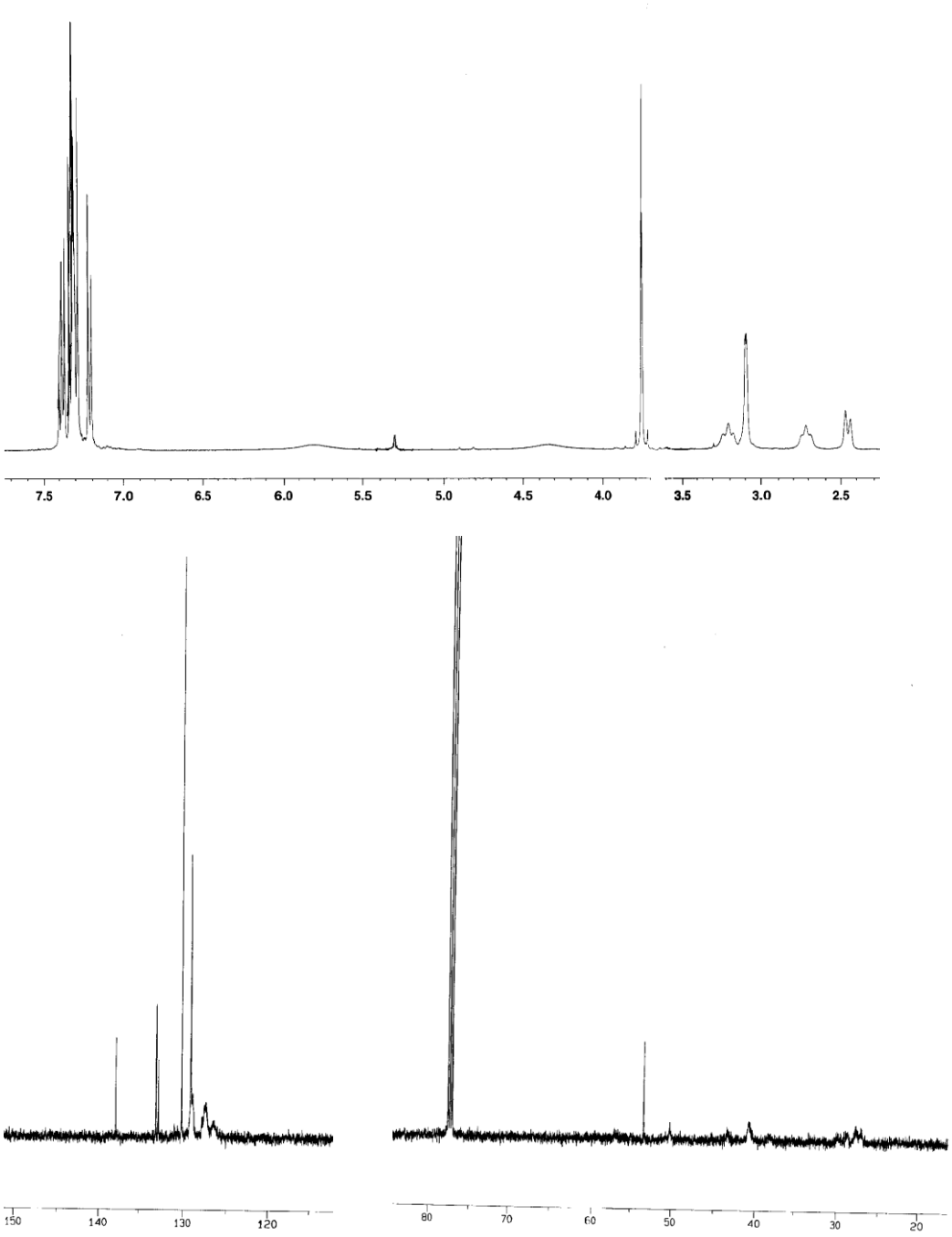
^{1}H and ^{13}C NMR spectra for **18e**



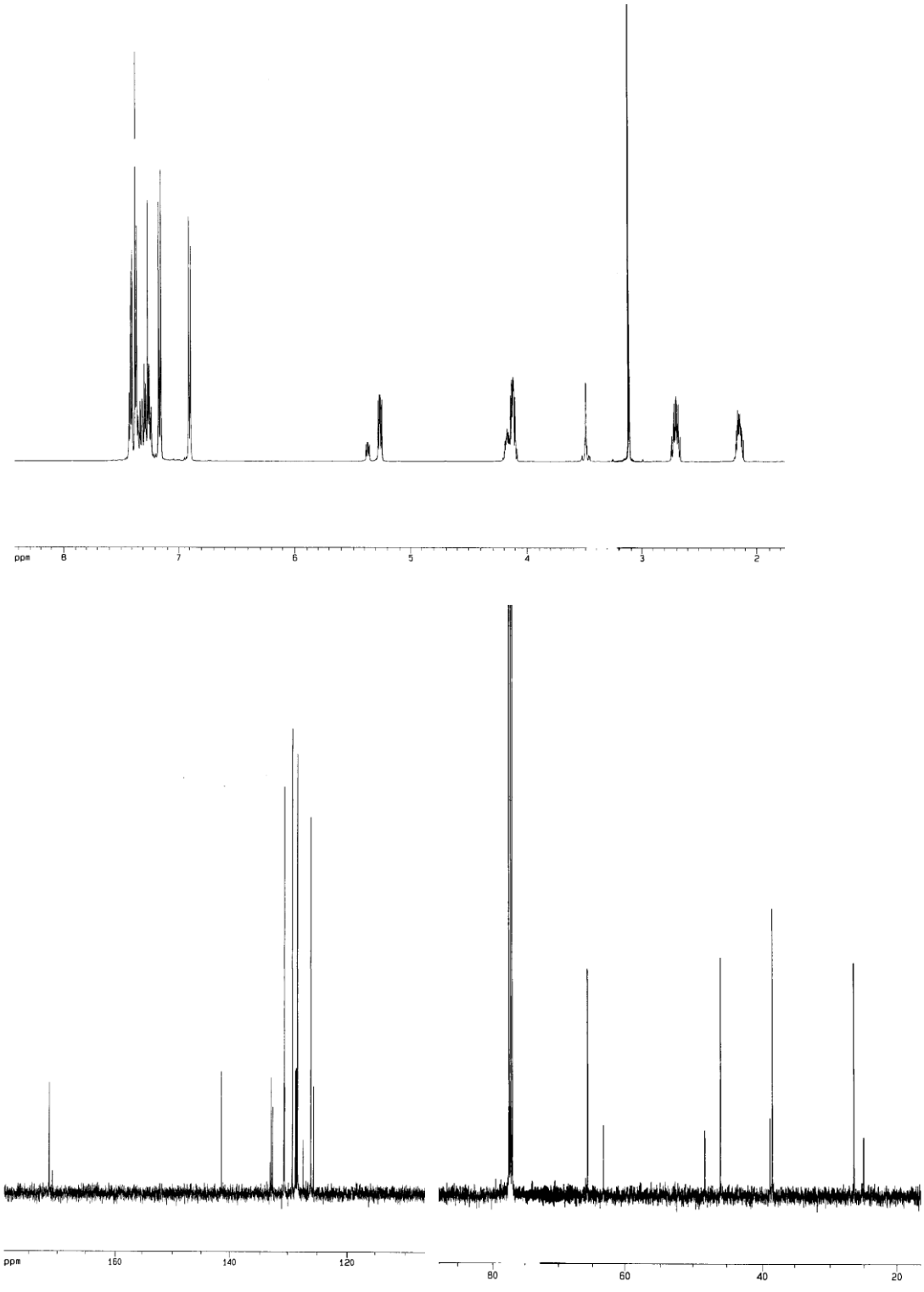
^{1}H and ^{13}C NMR spectra for 1,1,2-triphenylethane



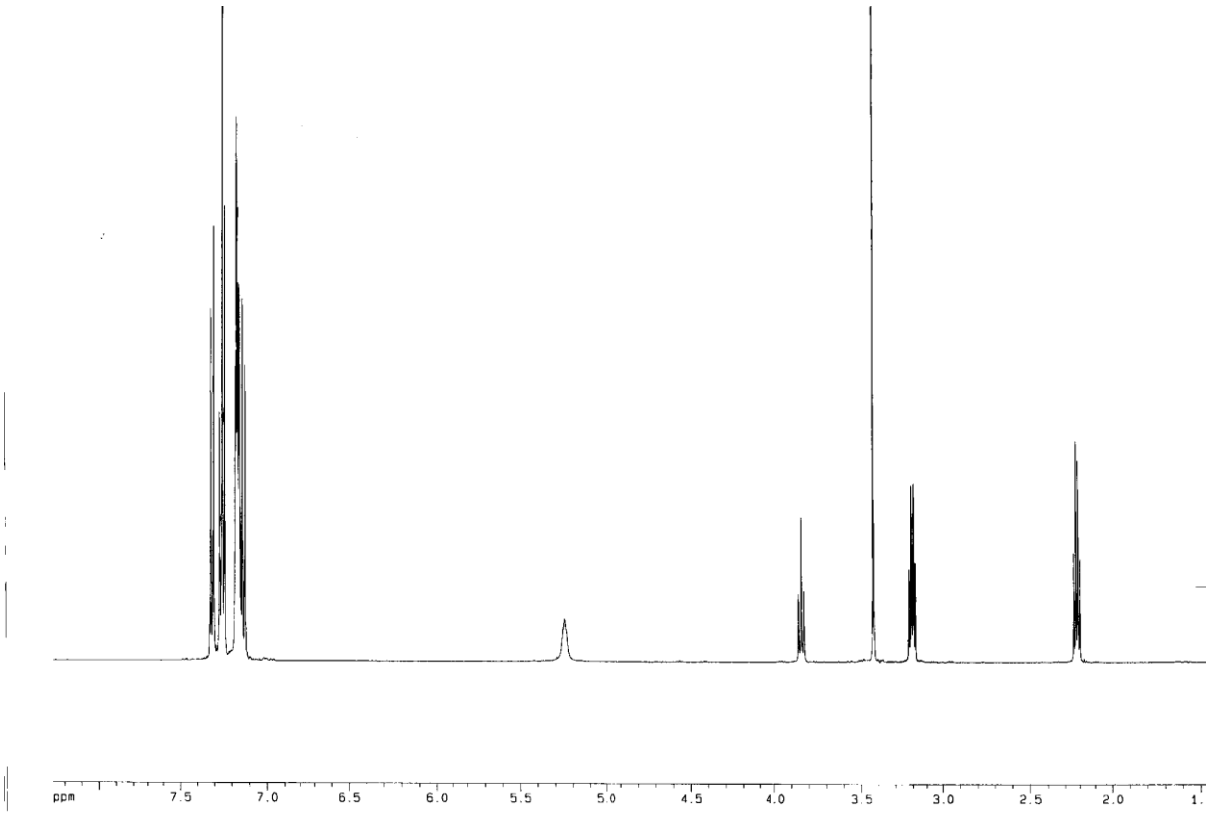
^{1}H and ^{13}C NMR spectra for **18f**

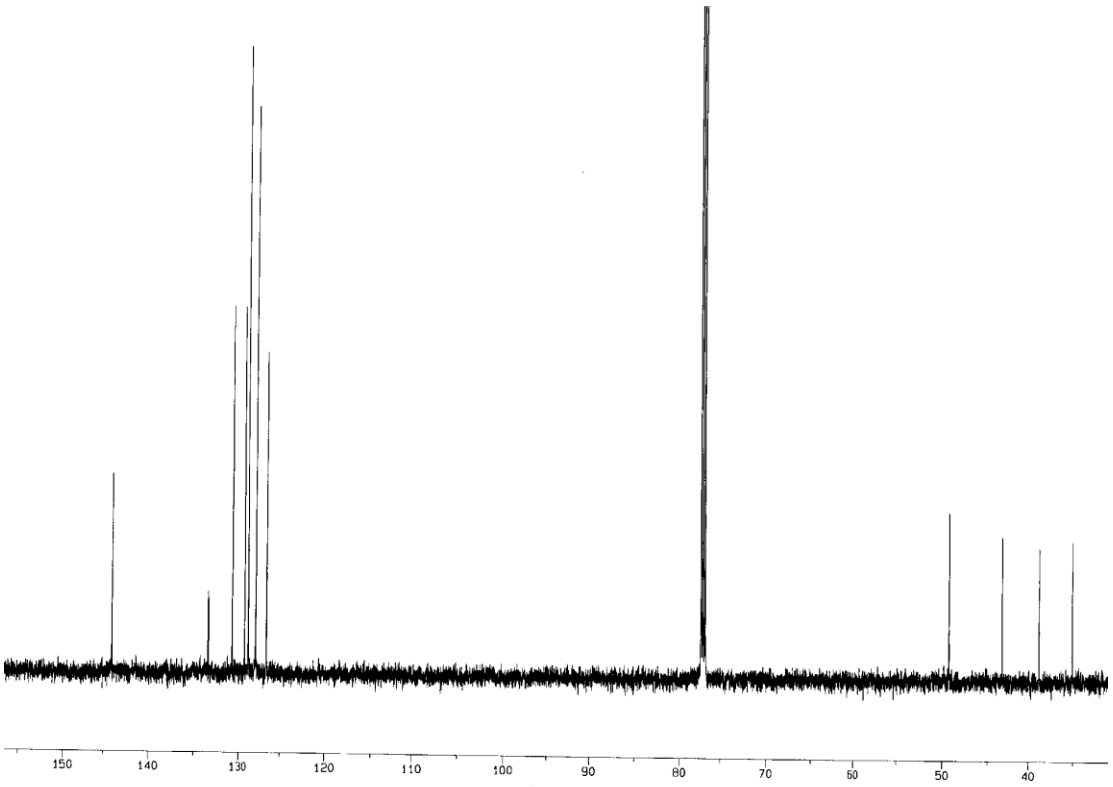


^{1}H and ^{13}C NMR spectra for **20**

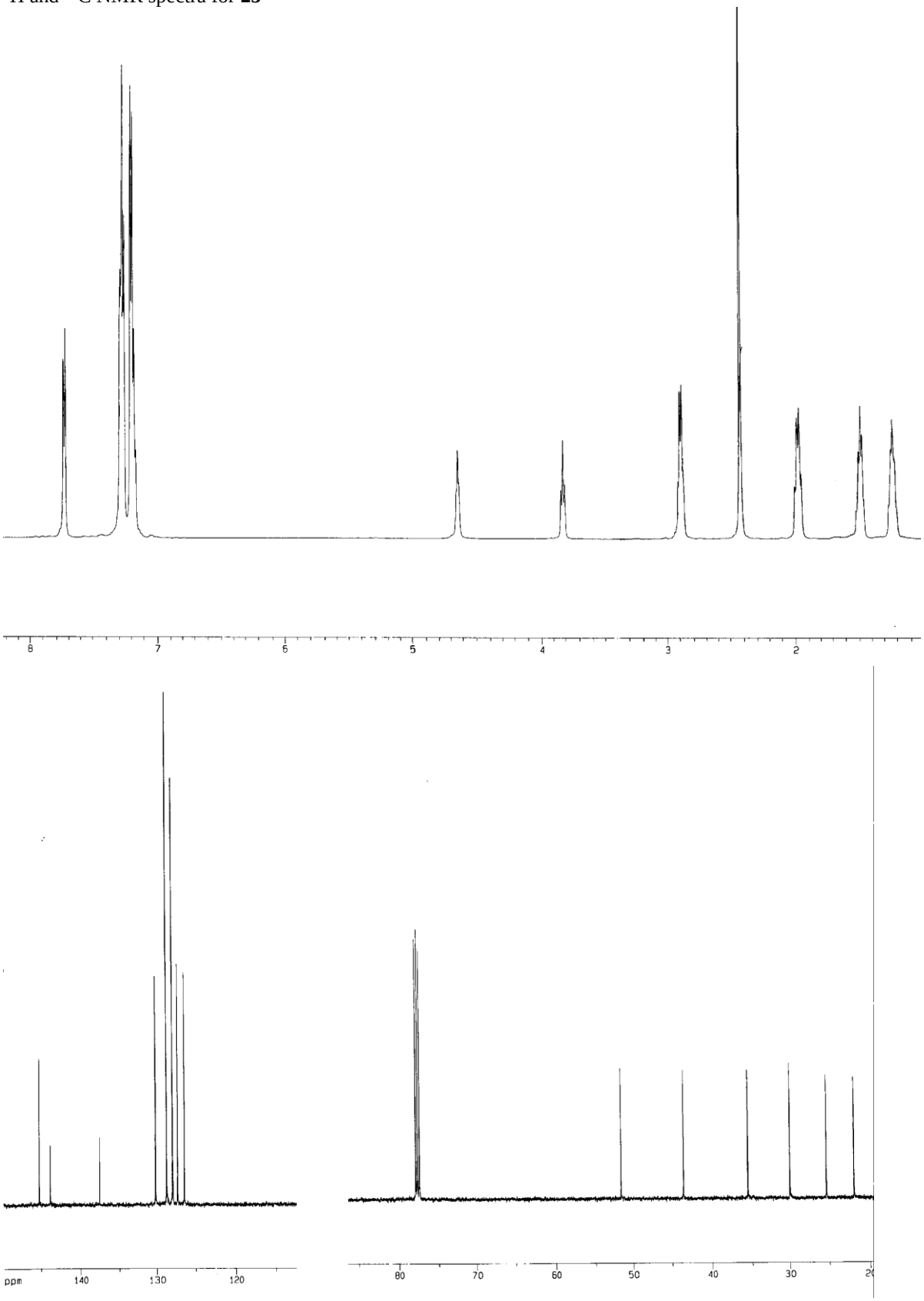


^{1}H and ^{13}C NMR spectra for **21**

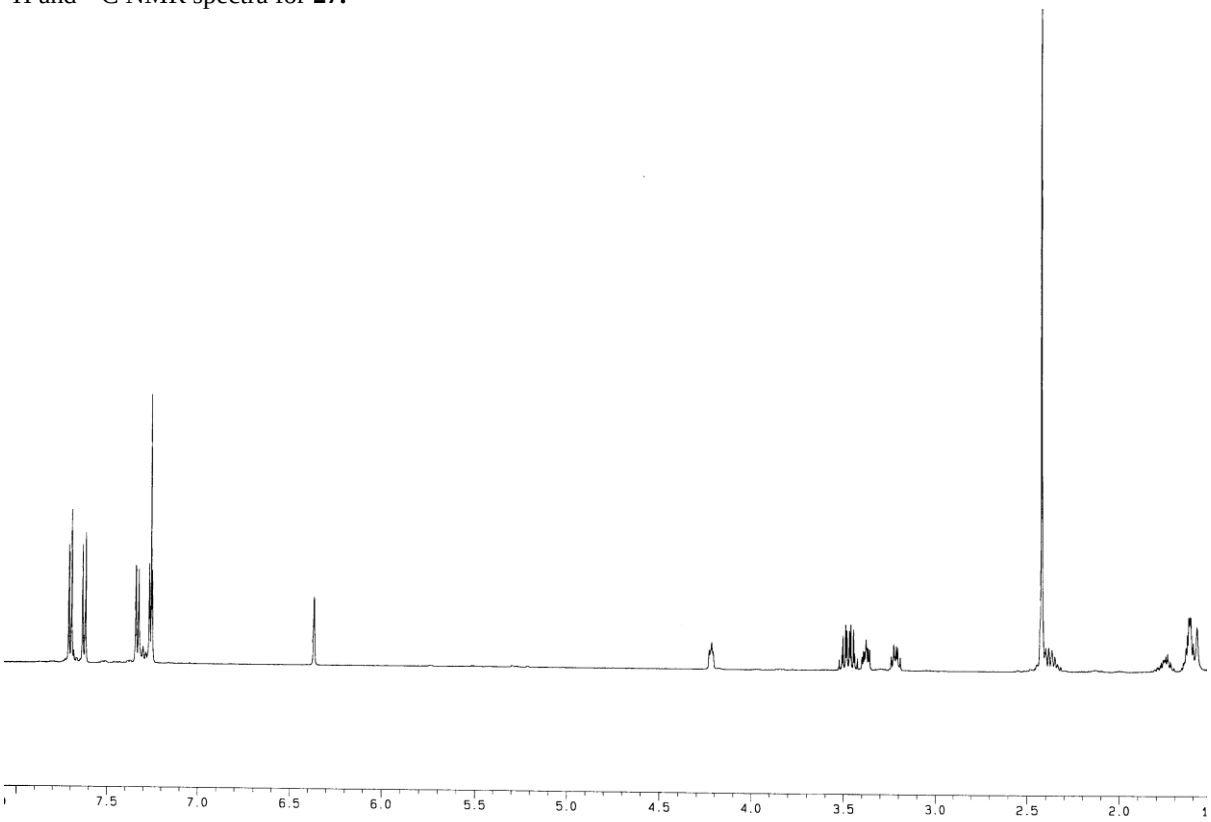


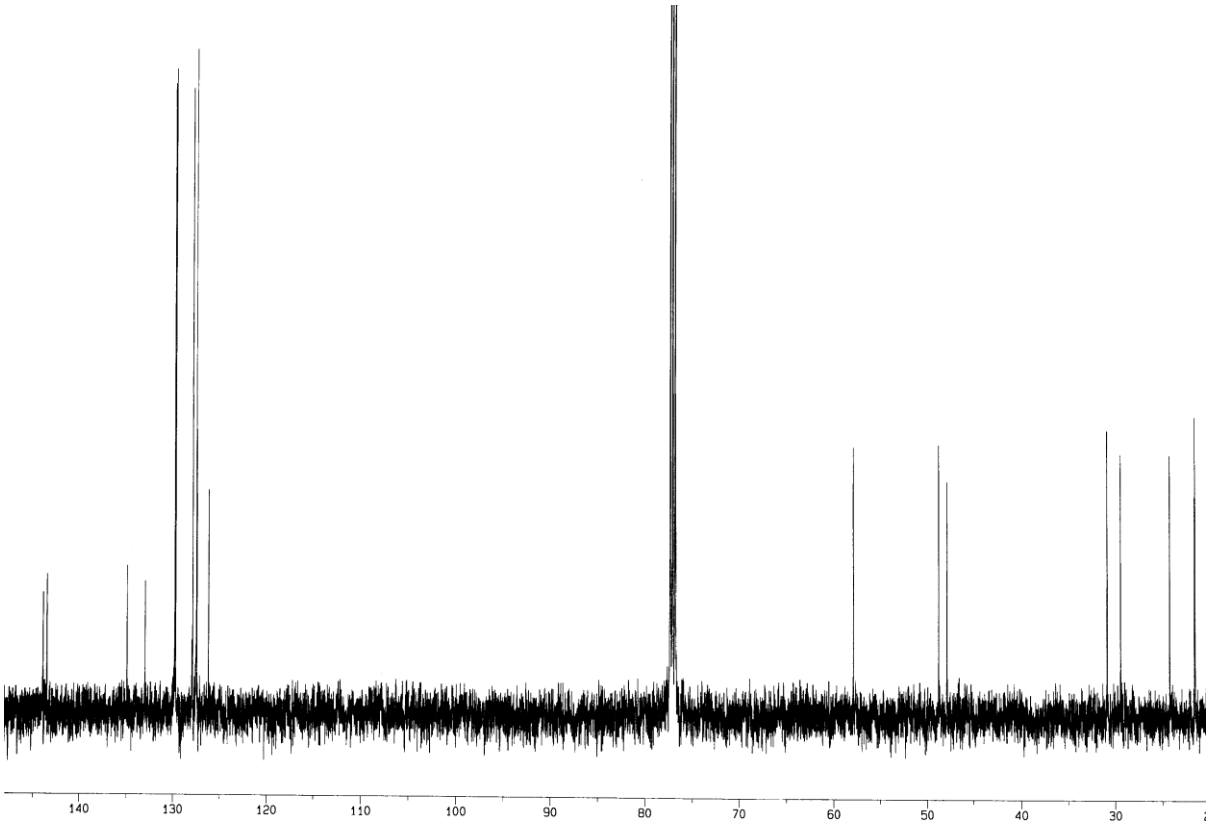


^{1}H and ^{13}C NMR spectra for **25**



^{1}H and ^{13}C NMR spectra for **27.**





^{1}H and ^{13}C NMR spectra for the mixture of **28** and **29**.