Supporting Information for

Long-chain rhenium and technetium glucosamine conjugates

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N,N-Dibenzyl-6-aminohexanoic acid (2a). 6-Aminohexanoic acid (6.56 g, 50 mmol) was dissolved in H_2O (30 mL), and KOH (9.8 g, 750 mmol) was added. Once this had dissolved, ethanol (50 mL) was added, followed by benzyl bromide (11.95 mL, 100 mmol) which was added in a dropwise fashion. The resulting solution was stirred for 16 h at room temperature, followed by heating at reflux for 30 min. After cooling and evaporation to 50 % of the original volume, the solution was acidified with AcOH to pH 5, and an oily product formed, which extracted with chloroform (3 × 100 mL). The organic layers were combined, washed with brine (3 × 200 mL), and dried over anhydrous $MgSO_4$, which was then filtered out through a glass frit. The filtrate was reduced to dryness under reduced pressure. The oily product was purified via column chromatography (silica-5 % methanol: CH_2Cl_2) to isolate a colorless oil (13.2 g, 85 % yield). R_f = 0.41 (silica- 8 % methanol: CH_2Cl_2). ^{1}H NMR (MeOH-d_4, 300 MHz, δ): 10.66 (s, 1H), 7.42-7.25 (m, 10H), 3.76 (s, 4H), 2.54 (t, J = 15.06 Hz, 2H), 2.28 (t, J = 14.73Hz, 2H), 1.58 (m, 4H), 1.27 (m, 2H). ^{13}C NMR (CD_2Cl_2, 75 MHz, δ): 178.61, 136.31, 130.42, 129.08, 128.49, 57.94, 52.73, 35.29, 27.17, 25.73, 25.34. MS (ES^+, 100% MeOH): m/z = 312 ($M\text{-}H^+$). HR-MS (ES+ of $M\text{-}H^+$) m/z calcd for $C_{20}H_{26}NO_2$: 312.1964, found: 312.1968.

N,N-Dibenzyl-8-aminooctanoic acid (2b). The experimental procedure for **2b** is similar to that of **2a**. To a solution of 8-aminooctanoic acid (3.82 g, 24 mmol) in H_2O (30 mL), KOH (4.71 g, 84 mmol) and EtOH (30 mL) were added. Benzyl bromide (5.7 mL, 48 mmol) was added dropwise, and the resulting solution was stirred for 16 h at room temperature, followed by heating under reflux for 30 min. Following workup the oily product was purified via column chromatography (silica-5 %

methanol: CH_2Cl_2) to isolate a colorless oil (7.9 g, 70 % yield). R_f = 0.35 (silica- 5 % methanol: CH_2Cl_2). ^{1}H NMR (MeOH-d_4, 300 MHz, δ): 7.37-7.30 (m, 10H), 3.78(s, 4H), 2.56(t, J = 11.43Hz, 2H), 2.17(t, J = 12.57 Hz, 2H), 1.50(m, 4H), 1.17(m, 6H). ^{13}C NMR (MeOH-d_4, 75 MHz, δ): 179.21, 137.15, 131.02, 129.79, 129.29, 58.99, 53.70, 36.31, 30.30, 30.04, 27.95, 26.55, 26.44. MS (ES^+, 100% MeOH): *m/z* = 340 ($M\text{-}H^+$). HR-MS (ES^+ of $M\text{-}H^+$) *m/z* calcd for $C_{22}H_{30}NO_2$: 340.2277, found: 340.2281.

***N,N*-Dibenzyl-11-aminoundecanoic acid (2c).** This compound was prepared from 11-aminoundecanoic acid using a procedure similar to that for **2a**. To a solution of 11-aminoundecanoic acid (10.07 g, 50 mmol) in H_2O (100 mL), KOH (9.78 g, 174 mmol) and EtOH (100 mL) were added. Benzyl bromide (11.88 mL, 100 mmol) was added dropwise, and the resulting solution was stirred for 16 h at room temperature, followed by heating under reflux for 30 min. Following workup the oily product was purified via column chromatography (silica-5 % methanol: CH_2Cl_2) to isolate a colorless oil product (14.3 g, 75 % yield). R_f = 0.31 (silica- 5 % methanol: CH_2Cl_2). ^{1}H NMR (MeOH-d_4, 300 MHz, δ): 7.52-7.33(m, 10H), 3.95(s, 4H), 2.65(t, J = 15.63, 2H), 2.31(t, J = 14.40, 2H), 1.71(m, 2H), 1.59(m, 2H), 1.25(m, 2H), 1.18(m, 12H). ^{13}C NMR (MeOH-d_4, 75 MHz, δ): 179.29, 137.22, 131.10, 129.87, 129.36, 59.07, 53.78, 36.39, 30.38, 30.12, 28.02, 26.62, 26.52. MS (ES+, 100% MeOH) m/z = 382 ($M\text{-}H^+$). HR-MS (ES+ of $M\text{-}H^+$) *m/z* calcd for $C_{22}H_{30}NO_2$: 382.2746, found: 382.2758.

2-(6-(*N,N*-Dibenzylamino)-hexylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (3a). This compound was prepared via amide coupling of 1 and **2a**. **2a** (13.24 g, 42.5 mmol) was dissolved in DMF (150 mL). The resulting clear colorless

solution was cooled in an ice bath. EDC.HCl (N-(3-dimethylaminopropyl)-N'-ethylcarbodiimide hydrochloride) (5.216 g, 46.8 mmol), HOBt.H_2O (1-hydroxybenzotriazole hydrate) (7.17 g, 46.8 mmol) and DMAP (4-dimethylaminopyridine) (0.52 g, 4.2 mmol) were added sequentially to the DMF solution. The reaction mixture was stirred at 0 ºC for 30 min and to this solution was added 1,3,4,6-tetra-O-acetyl-β-D-glucosamine **1**, (16.31 g, 42.5 mmol) The ice-bath was removed and the clear colorless solution was stirred overnight for 18 h with slow warming to room temperature. Following consumption of the starting material as monitored by TLC (silica-15 % acetone: CH_2Cl_2), the solvent was removed under reduced pressure, leaving behind a pale white oily product. The oily product was dissolved in DCM (125 mL) and washed with saturated aqueous Na_2CO_3 solution (125 mL), 1M HCl (125 mL) and brine (3 x 125 mL). The organic layer was removed from the separatory funnel, dried over anhydrous $MgSO_4$, and filtered through a glass frit. The filtrate was reduced to dryness under reduced pressure. The oily product was purified via column chromatography (silica-15 % acetone: CH_2Cl_2) to isolate a white oily product (17.9 g, 66 % yield). R_f = 0.32 (silica- 15 % acetone: CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz, δ): 7.37-7.29(m, 10H), 5.68(d, J = 5.73 Hz, 1H), 5.38(m, 1H), 5.13(d, J = 3.42), 4.58(m, 2H), 4.13(d, J= 12.33 Hz, 1H), 3.78(1H), 3.53(s, 4H), 2.37(2H), 2.08(s, 3H), 2.05(s, 3H), 2.03(s, 3H), 2.01(s,3H), 1.98(m, 2H), 1.62-1.46 (m, 4H), 1.24(m, 2H). ^{13}C NMR (CD_2Cl_2, 75 MHz, δ):173.52, 171.45, 170.13, 170.03, 140.91, 129.48, 128.83, 127.43, 93.20, 73.63, 73.01, 68.67, 62.50, 58.99, 56.92, 53.61, 37.28, 27.48, 26.18, 21.38, 21.17. MS (ES^+, 100 % MeCN): *m/z* = 641 ($M-H^+$). HR-MS (ES^+ of $M-H^+$) *m/z* calcd for $C_{34}H_{45}N_2O_{10}$: 641.3074, found:

641.3099.

2-(8-(*N,N*-Dibenzylamino)-octylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (3b). This compound was prepared using a similar procedure to that for **3a**. **2b** (8.383 g, 24.69 mmol) was dissolved in DMF (130 mL). The resulting clear colorless solution was cooled in an ice bath, then EDC·HCl (5.216 g, 27.2 mmol), HOBt.H_2O (4.164 g, 27.2 mmol) and DMAP (0.30 g, 2.5 mmol) were added sequentially. The reaction mixture was stirred at 0 ºC for 30 min and to this solution was added **1** (19.48 g, 24.69 mmol) The ice-bath was removed and the clear colorless solution was stirred overnight for 18 h with slow warming to room temperature. The oily product was purified via column chromatography (silica-10 % acetone: CH_2Cl_2) to isolate a white oil (12.3 g, 74 % yield). R_f = 0.38 (silica- 10 % acetone: CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz, δ): 7.60-7.33(m, 10H), 5.68(d, J = 4.80 Hz, 1H), 5.42(m, 1H), 5.12(m, 1H), 4.28(m, 2H), 4.13(d, J = 12.45 Hz, 1H), 3.79(m, 1H), 3.53(s, 4H), 2.37(m, 2H), 2.08(s, 3H), 2.03(s, 3H), 2.02(s, 3H), 2.00(s, 3H), 1.62(m, 2H), 1.50(m, 4H), 1.20(m, 6H). ^{13}C NMR (DMSO-d_6, 75 MHz, δ): 172.52, 170.15, 169.58, 169.39, 168.90, 139.76, 128.58, 128.28, 126.88, 91.87, 72.29, 71.64, 68.23, 61.61, 57.70, 52.64, 51.87, 35.60, 28.64, 28.50, 26.65, 26.34, 25.35, 20.53, 20.44. MS (ES^+, 100 % MeCN): *m/z* = 669 ($M\text{-}H^+$). HR-MS (ES+ of $M\text{-}H^+$) *m/z* calcd for $C_{36}H_{49}N_2O_{10}$: 669.3387, found: 669.3395.

2-(11-(*N,N*-Dibenzylamino)-undecylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (3c). This compound was synthesized using a similar method to that for **3a**. **2c** (5.65 g, 14.8 mmol) was dissolved in DMF (100 mL). The resulting clear colourless solution was cooled in an ice bath, then EDC·HCl (3.13 g, 16.3 mmol),

$HOBt{\cdot}H_2O$ (2.50 g, 16.3 mmol) and DMAP (0.50 g, 4.0 mmol) were added sequentially. The reaction mixture was stirred at 0 ºC for 30 min and to this solution was added **1** (5.68 g, 14.8 mmol). The ice-bath was removed and the clear colorless solution was stirred overnight for 18 h after warming to room temperature. The oily product was purified via column chromatography (silica-10 % acetone: CH_2Cl_2) to isolated a white oil product (2.85 g, 55 % yield). R_f = 0.38 (silica- 10 % acetone: CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz, δ): 7.35-7.16(m, 10H), 5.70(d, J = 9.03 Hz, 1H), 5.49(m, 1H), 5.14(m, 1H), 4.27(m, 2H), 4.23(m, 1H), 3.78(m, 1H), 3.33(s, 4H), 2.38(t, J = 6.72 Hz, 2H), 2.09(s, 6H), 2.03(s, 3H), 2.02(s, 3H), 1.93(m, 2H), 1.65(m, 2H), 1.53(m, 4H), 1.22(m, 12H). ^{13}C NMR (CD_2Cl_2, 75 MHz, δ): 174.13, 171.28, 171.05, 170.12, 169.72, 140.77, 129.35, 128.72, 127.33, 92.99, 73.30, 69.27, 62.77, 58.88, 53.27, 37.27, 37.22, 30.30, 30.19, 30.13, 30.10, 29.87, 27.85, 27.61, 26.49, 21.25, 21.17, 21.09. MS (ES+, 100% MeCN): *m/z* = 711 ($M\text{-}H^+$). HR-MS (ES^+ of $M\text{-}H^+$) *m/z* calcd for $C_{39}H_{55}N_2O_{10}$: 711.3857, found: 711.3856.

2-(6-Aminohexylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (4a). **3a** (2.21 g, 3.44 mmol) was dissolved in 100 mL acetic acid. $Pd(OH)_2/C$ (0.80 g, 0.0071 mmol) was added in one portion and the flask was capped with a rubber septum and purged with H_2 from a balloon. The mixture was kept under a positive pressure of H_2 for 10 days. The absence of starting material was confirmed by TLC (silica-15 % acetone: CH_2Cl_2) and mass spectrometry ($[M\text{-}H^+]$ = 461). The suspension was filtered through a Celite plug (~1 cm in a small frit) giving a clear pale yellow solution. The solvent was removed under reduced pressure to yield a colorless oily **4a** (0.98 g, 62 % yield). **4a** is unstable, and was used directly in the next reaction.

2-(8-Aminooctylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (4b). This compound was prepared using a similar method to that for **4a**. **3b** (2.96 g, 4.42 mmol) was dissolved in 100 mL acetic acid. $Pd(OH)_2/C$ (0.80 g, 0.0057 mmol) was added in one portion and the flask was capped with a rubber septum and purged with H_2 from a balloon. The mixture was kept under a positive pressure of H_2 from the H_2 balloon for 14 days. The absence of starting material was confirmed by TLC (silica-15 % acetone: CH_2Cl_2) and mass spectrometry ($[M\text{-}H^+]$ = 489). The suspension was filtered through a Celite plug (~1 cm in a small frit) giving a clear pale yellow solution. The solvent was removed under reduced pressure to yield a colorless oily **4b** (1.40 g, 65 % yield). **4b** is unstable, and was used directly in the next reaction.

2-(11-Aminoundecylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (4c). This compound was prepared using a similar method to that for **4a**. **3c** (2.85 g, 4.02 mmol) was dissolved in 100 mL acetic acid. $Pd(OH)_2/C$ (1.00 g, 0.00713 mmol) was used. The reaction time is 18 days. MS: $[M\text{-}H^+]$ = 531. The solvent was removed under reduced pressure to yield a colorless oily **4c** (1.38 g, 65 % yield). **4c** is unstable, and was used directly in the next reaction.

2-((6-(*N*-Pyridin-2-ylmethyl)amino)-hexylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (5a). **4a** (0.98 g, 2.13 mmol) was dissolved straight away in dichloroethane (100 mL). Na_2CO_3 (14.6 g, 138 mmol) and 2-pyridinecarboxaldehyde (0.228 g, 2.13 mmol) were added sequentially. The cloudy yellow solution was stirred overnight for 18 h. $NaBH(OAc)_3$ (4.32 g, 20.4 mmol) was added in one portion after consumption of the starting material was confirmed by TLC and mass spectrometry, and the reaction mixture was stirred for an additional 16 h. The white solid was

removed via filtration through a glass frit. The resulting clear orange solution was concentrated under reduced pressure to give a yellow oily product. The oily product was dissolved in CH_2Cl_2 (50 mL) and washed with saturated aqueous Na_2CO_3 (50 mL), then brine (50 mL). The organic layer was dried over anhydrous $MgSO_4$, filtered and evaporated under reduced pressure to yield a yellow solid. ESI-MS: [M-H^+] = 561. **5a** was used directly in the next reaction.

2-((8-(*N*-Pyridin-2-ylmethyl)amino)-octylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (5b). The experimental procedure for **5b** is similar to that for **5a**. **4b** (1.40 g, 2.87 mmol), Na_2CO_3 (20.0 g, 189 mmol), 2-pyridinecarboxaldehyde (0.294 g, 2.74 mmol) and $NaBH(OAc)_3$ (4.32 g, 20.4 mmol) were added sequentially. ESI-MS: [M-H^+] = 580.

2-((11-(*N*-Pyridin-2-ylmethyl)amino)-undecylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (5c). The experimental procedure for 5c is similar to that for **5a**. **4c** (1.38 g, 2.61 mmol), Na_2CO_3 (14.0 g, 132 mmol), 2-pyridinecarboxaldehyde (0.280 g, 2.61 mol) and $NaBH(OAc)_3$ (5.60 g, 26.4 mmol) were added sequentially. ESI-MS: [M-H^+] = 621.

2-((6-*N*-(2-Hydroxybenzyl)-*N*-(pyridin-2-ylmethyl)amino)-hexylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (6a). 5a was dissolved directly in dichloroethane (50 mL). Salicylaldehyde (0.261 g, 2.13 mmol) and $NaBH(OAc)_3$ (4.32 g, 20.4 mmol) were added sequentially and the reaction mixture was stirred overnight for 20 h. Following consumption of the starting material as confirmed by TLC (silica-50 % MeCN: 50 % ethyl acetate) and mass spectrometry, the reaction mixture was reduced to dryness under reduced pressure. The residue was dissolved in

CH_2Cl_2 (25 mL) and washed with saturated aqueous Na_2CO_3 solution (25 mL) and then brine (25 mL). The organic layer was dried over anhydrous $MgSO_4$, and filtered; the solvent was removed under reduced pressure to give a yellow solid, which was purified using column chromatography (silica-20 % CH_2Cl_2, ethyl acetate) to give a yellow solid (1.02 g, 73 % over 2 steps). R_f = 0.33 (silica-20 % CH_2Cl_2, ethyl acetate). ^{1}H NMR (DMSO-d_6, 300 MHz, δ): 10.50(s, 1H), 8.49(s, 1H), 7.90(d, J = 6.84Hz, 1H), 7.76(t, J = 15.18Hz, 1H), 7.38(d, J = 6.25Hz, 1H), 7.26(t, J = 10.50Hz, 1H), 7.08(m, 2H), 6.70(m, 2H), 5.68(d, J = 6.27 Hz, 1H), 5.13(t, J = 17.25, 1H), 4.85(t, J = 16.32Hz, 1H), 4.17 (d, J = 12.66Hz, 2H), 3.97(m, 2H), 3.72(s, 2H), 3.64(s, 1H), 2.16(m, 2H), 1.98(s, 3H), 1.97(s, 3H), 1.95(s, 6H), 1.83(t, J = 2.61Hz, 2H), 1.43(m, 2H), 1.31(m, 2H), 1.05(m, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz, δ): 172.22, 169.92, 169.38, 169.17, 168.68, 158.11, 156.80, 148.78, 136.68, 129.25, 128.07, 123.02, 122.25, 118.57, 115.32, 91.63, 72.05, 71.41, 67.98, 61.39, 58.58, 55.11, 52.62, 51.62, 35.31, 25.97, 25.53, 24.95, 20.37, 20.31, 20.19. MS (ES+, 100 % MeCN): *m/z* = 658 (M-H$^+$). HR-MS (ES+ of M-H$^+$) *m/z* calcd for $C_{33}H_{44}N_3O_{11}$: 658.2976, found: 658.2990.

2-((8-*N*-(2-Hydroxybenzyl)-*N*-(pyridin-2-ylmethyl)amino)-octylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (6b). The experimental procedure for **6b** is similar to that for **6a**. **5b** was dissolved in dichloroethane (20 mL), and salicylaldehyde (0.335 g, 2.74 mmol) and $NaBH(OAc)_3$ (4.30 g, 20.4 mmol) were added sequentially and reacted overnight at room temperature. Following workup the yellow solid was purified using column chromatography (silica-50 % MeCN:50 % ethyl acetate eluent) to give a yellow solid (1.27 g, 64 % over 2 steps). R_f = 0.45

(silice-50 % MeCN: 50 % ethyl acetate). ^{1}H NMR (DMSO-d_6, 300 MHz, δ). NMR data: 10.52(s, 1H), 8.52(s, 1H), 7.90(d, J = 5.13Hz, 1H), 7.79(m, 1H), 7.39 (t, J = 12.12 Hz, 1H), 7.29(m, 1H), 7.11(m, 2H), 6.72(m, 2H), 5.70(dd, J = 13.14, J = 4.47Hz, 1H), 5.17(m, 1H), 4.88(m, 1H), 4.20(m, 2H), 4.00(s, 2H), 3.74(s, 2H), 2.42(t, J = 13.71Hz, 2H), 2.01(s, 3H), 1.99(s, 3H), 1.97(s, 3H), 1.96(s, 3H), 1.89(t, J = 12.33Hz, 2H), 1.44(m, 2H), 1.35(m, 2H), 1.10(m, 6H). ^{13}C NMR (DMSO-d_6, 100 MHz, δ): 172.29, 169.92, 169.36, 169.16, 168.67, 158.14, 156.82, 148.75, 136.65, 129.25, 128.05, 123.27, 123.02, 122.23, 118.54, 115.31, 91.64, 72.05, 71.41, 68.00, 61.37, 58.60, 55.57, 52. 57, 51.64, 35.35, 28.32, 28.17, 26.32, 25.69, 25.08, 20.39, 20.30, 20.21. MS (ES^+, 100 % MeCN): *m/z* = 708 ($M\text{-}Na^+$). HR-MS (ES^+ of $M\text{-}Na^+$) *m/z* calcd for $C_{35}H_{47}N_3O_{11}Na$: 708.3108, found: 708.3107.

2-((11-*N*-(2-Hydroxybenzyl)-*N*-(pyridin-2-ylmethyl)amino)-undecylamido)-2-deoxy-1,3,4,6-tetra-*O*-acetyl-*D*-β-glucopyranose (6c). The experimental procedure for **6c** is similar to that for **6a**. **5c**, salicylaldehyde (0.319 g, 2.61 mmol) and $NaBH(OAc)_3$ (5.00 g, 23.6 mmol) were added sequentially to a round bottom flask with dichloroethane (20 mL). The resulting mixture was stirred overnight at room temperature, then worked up and purified by column chromatography (50 % MeCN: 50 % ethyl acetate) as above to give a yellow solid (1.24 g, 67 % over 2 steps). R_f = 0.48 (silica-50 % MeCN: 50 % ethyl acetate). ^{1}H NMR (DMSO-d_6, 300 MHz, δ): 10.53(s, 1H), 8.51(s, 1H), 7.94(d, J = 9.03Hz, 1H), 7.78(m, 1H), 7.74(m, 1H), 7.38(t, J = 12.45 Hz, 1H), 7.26(m, 1H), 7.10(m, 2H), 6.72(m, 2H), 5.69(m, 1H), 5.17(m, 1H), 4.88(m, 1H), 4.2(m, 1H), 3.96(m, 3H), 3.74(s, 2H), 3.66(s, 2H), 2.42(t, J = 10.29Hz, 2H), 2.00(s, 3H), 1.98(s, 3H), 1.97(s, 3H), 1.95(s, 3H), 1.89(t, J = 12.78 Hz, 2H),

1.40(m, 4H), 1.13(m, 12H). ^{13}C NMR (DMSO-d_6, 75 MHz, δ): 172.17, 169.77, 169.22, 169.01, 168.53, 158.01, 156.70, 148.60, 136.50, 129.10, 127.91, 123.14, 122.88, 122.09, 118.38, 115.18, 91.52, 71.93, 71.27, 67.87, 61.24, 58.50, 55.17, 52.45, 51.51, 35.24, 28.65, 28.60, 28.48, 28.40, 28.10, 26.27, 25.52, 25.00, 20.25, 20.16, 20.08. MS (ES+, 100 % MeCN): *m/z* = 708 (M-H$^+$). HR-MS (ES+ of M-H$^+$) *m/z* calcd for $C_{38}H_{54}N_3O_{11}$: 728.3758, found: 728.3774.

2-((6-*N*-(2-Hydroxybenzyl)-*N*-(pyridin-2-ylmethyl)amino)-hexylamido)-2-deoxy-*D*-glucopyranose (7a). 6a (0.906 g, 1.38 mmol) was dissolved in MeOH (5 mL). NaOMe (900 mg, 1.37 mmol) was added in one portion and the solution stirred for 6 h. Following consumption of the starting material by TLC (silica-15 % MeOH, CH_2Cl_2) and mass spectrometry, the solvent was removed under reduced pressure to yield a pale white solid. The solid was purified using silica gel chromatography with 15 % MeOH: 85 % CH_2Cl_2 as the eluent to yield a pale yellow solid 7a (560 mg, 83 % yield). R_f = 0. 40 (silica- 15% MeOH: 85% CH_2Cl_2). ^{1}H NMR (DMSO-d_6, 300 MHz, δ): 8.54(d, J = 4.23 Hz, 1H), 7.79(t, J = 14.49 Hz, 1H), 7.50(d, J = 7.65 Hz, 1H), 7.29(t, J = 12.21 Hz, 1H), 7.11(dd, J = 22.83 and 7.32 Hz, 2H), 6.73(t, J = 15.18 Hz, 2H), 4.91(d, J = 2.85 Hz, 1H-β), 4.43(d, J = 7.65 Hz, 1H-α), 3.75(s, 2H), 3.67(s, 2H), 3.59(m, 2H), 3.50-3.31(m, 3H), 3.10(m, 1H), 2.42(t, J = 13.68 Hz, 2H), 2.05(m, 2H), 1.48(m, 2H), 1.38(m, 2H), 1.15(m, 2H). ^{13}C NMR (DMSO-d_6, 75 MHz, δ): 172.80,158.78, 157.50, 149.45, 137.52, 129.91, 128.74, 123.91, 123.72, 122.93, 119.26, 116.00, 96.18, 91.18, 72.64, 71.75, 71.03, 61.73, 59.23, 55.78, 54.79, 53.38, 35.71, 26.99, 26.22, 25.66. MS (ES+, 100 % MeOH): *m/z* = 490 (M-H$^+$). HR-MS (ES$^+$ of M-H$^+$) *m/z* calcd for $C_{25}H_{36}N_3O_7$: 490.2553, found: 490.2548.

2-((8-*N*-(2-Hydroxybenzyl)-*N*-(pyridin-2-ylmethyl)amino)-octylamido)-2-deoxy-*D*-glucopyranose (7b). 6b (0.871 g, 1.27 mmol) was dissolved in 5 mL MeOH. NaOMe (868 mg, 1.27 mmol) was added in one portion and the solution stirred for 4 h. Following consumption of the starting material by TLC (silica-12 % MeOH: CH_2Cl_2) and mass spectrometry, the solvent was removed under reduced pressure to yield a pale white solid. The solid was purified using silica gel chromatography with 12 % MeOH: 88 % CH_2Cl_2 as the eluent to yield a pale yellow solid 7b (420 mg, 64 % yield). R_f = 0. 38 (silica-12 % MeOH: 88 % CH_2Cl_2). ^{1}H NMR (DMSO-d_6, 300 MHz, δ): 8.52(d, J = 3.90 Hz, 1H), 7.79(t, J = 15.75Hz, 1H), 7.41(d, J = 7.77 Hz, 1H), 7.28(t, J = 11.97 Hz, 1H), 7.10(dd, J = 20.19 and 6.6Hz, 2H), 6.73(t, J = 14.52Hz, 2H), 4.90(d, J = 2.4 Hz, 1H-β), 4.40(d, J = 5.13Hz, 1H-α), 3.74(s, 2H), 3.66(s, 2H), 3.57(m, 2H), 3.47(m, 2H), 3.16(m, 3H), 2.42(t, J = 13.02 Hz, 2H), 2.05(t, J = 14.04Hz, 2H), 1.44(m, 4H), 1.13(m, 6H). ^{13}C NMR (DMSO-d_6, 75 MHz, δ): 172.23, 158.14, 156.85, 148.78, 129.27, 128.09, 123.26, 123.06, 122.27, 118.57, 115.34, 95.52, 90.53, 71.09, 70.35, 61.06, 58.62, 55.27, 54.13, 52.65, 35.17, 28.60, 28.49, 26.42, 25.68, 25.18. MS (ES+, 100 % MeOH): *m/z* = 518 (M-H$^+$). HR-MS (ES$^+$ of M-H$^+$) *m/z* calcd for $C_{27}H_{40}N_3O_7$: 518.2866, found: 518.2870.

2-((11-*N*-(2-Hydroxybenzyl)-*N*-(pyridin-2-ylmethyl)amino)-undecylamido)-2-deoxy-*D*-glucopyranose (7c). 6c (0.779 g, 1.07 mmol) was dissolved in MeOH (5 mL). NaOMe (779 mg, 1.07 mmol) was added in one portion and the solution stirred for 10 h. Following consumption of the starting material by TLC (silica-12 % MeOH, CH_2Cl_2) and mass spectrometry, the solvent was removed under reduced pressure to yield a pale white solid. The solid was purified using silica gel chromatography with

12 % MeOH: 88 % CH_2Cl_2 as the eluent to yield a pale yellow solid **7c** (435 mg, 73 % yield). R_f = 0.30 (silica-12 % MeOH: 88 % CH_2Cl_2). ^{1}H NMR (MeOH-d_4, 300 MHz, δ): 8.51(d, J = 3.50 Hz, 1H), 7.82(t, J = 14.25 Hz, 1H), 7.46(d, J = 6.75 Hz, 1H), 7.33(bs, 1H), 7.13(m, 2H), 6.76(m, 2H), 5.10(s, 1H-β), 4.57(d, J = 7.89, 1H-α), 3.81(s, 2H), 3.80(m, 2H), 3.79(s, 2H), 3.71(m, 3H), 3.35(m, 1H), 2.55(t, J = 12.33 Hz, 2H), 2.24(t, J = 9.36 Hz, 2H), 1.58(m, 4H), 1.21(m, 12H). ^{13}C NMR (MeOH-d_4, 75 MHz, δ): 176.82, 159.16,158.49, 149.93, 138.81, 130.58, 129.95, 125.32, 124.16, 123.94, 120.58, 116.87, 97.31, 92.91, 73.19, 72.98, 72.70, 72.42, 62.96, 60.38, 58.21, 55.93, 54.83, 37.18, 30.60, 30.37, 28.24, 27.35, 27.11. MS (ES+, 100 % MeOH): *m/z* = 560 (M-H$^+$). HR-MS (ES$^+$ of M-H$^+$) *m/z* calcd for $C_{30}H_{46}N_3O_7$: 560.3336, found: 560.3335.

6-Amino-hexylamino-*N*-(2-deoxy-β-*D*-glucopyranose) (10a). Compound **3a** (0.717 g, 1.12 mmol) was dissolved in methanol (15 mL). NaOMe (0.907 g, 16.8 mmol) was added, and the reaction mixture stirred at room temperature for 2 h. Amberlite resin was added and the heterogeneous mixture stirred for 15 min before being filtered. The crude reaction mixture was purified by semi-preparative HPLC to give a pale brown oil **9a**, and ESI-MS ([M-Na$^+$] = 495) was used to confirm the nature of the oily product that resulted (0.18 g, 32 % yield). The material obtained from the HPLC was dissolved in glacial acetic acid (5 mL) and added to a hydrogenation bomb with $Pd(OH)_2$ (0.18 g, 0.26 mmol). The bomb was sealed and purged with H_2 three times before being filled with H_2 to a pressure of 200 psi and the reaction mixture in the bomb stirred at room temperature for five days. The mixture was filtered through Celite, washed with glacial acetic acid (10 mL) and evacuated on a rotary evaporator

before being purified by semi-preparative HPLC. ESI-MS ([M-H^+] = 293) was used to confirm the nature of the oily product that resulted. The amount of product was estimated based on a comparison of the integration of the anomeric hydrogens with the CH_3 hydrogens of acetic acid.

8-Amino-octylamino-*N*-(2-deoxy-*D*-β-glucopyranose) (10b). Compound **3b** (0.256 g, 0.383 mmol) was dissolved in methanol (10 mL). NaOMe (0.207 g, 3.83 mmol) was added, and the reaction mixture stirred at room temperature for 4 h. The reaction was quenched by stirring with Amberlite ion exchange resin for 10 min, filtered and purified by semi-preparative HPLC to give **9b**, and ESI-MS ([M-H^+] = 501) was used to confirm the nature of the oily product that resulted (0.090 g, 47 % yield). This product was transferred to a hydrogenation bomb with glacial acetic acid (5 mL) and $Pd(OH)_2$ (0.18 g, 0.26 mmol). The system was sealed and purged three times with hydrogen before being filled to a pressure of 200 psi and then stirred at room temperature for five days. The mixture was filtered through Celite, washed with glacial acetic acid (10 mL) and evacuated on a rotary evaporator. ESI-MS ([M-H^+] = 321) was used to confirm the nature of the oily product that resulted. The amount of product was estimated based on a comparison of the integration of the anomeric hydrogens with the hydrogens of acetic acid.

Tricarbonyl{6-amino-hexylamido-*N*-(2-amino-2-deoxy-D-glucopyranose)cyclopentadienyl carboxamide}rhenium(I) (11a). Tricarbonyl(cyclopentadienyl carboxylic acid)rhenium (0.074 g, 0.20 mmol) and N,N'-dicyclohexylcarbodiimide (DCC) (0.041 g, 0.20 mmol) were dissolved in dry dichloromethane (10 mL). Activated 4 Å molecular sieves were added, the flask was

purged with argon and then stirred at room temperature for four hours. **10a** (~0.009 g, 0.030 mmol, used as a crude oil from debenzylation reaction above) and diisopropylethylamine (1.0 mL, 5.7 mmol) were stirred in dimethylformamide (6 mL) for 10 min, before addition to the activated rhenium precursor solution. The resulting solution was stirred under inert atmosphere at room temperature for 40 h. The reaction mixture was filtered, washed with dimethylformamide (5 mL), reduced on a rotary evaporator, taken up in dimethylsulfoxide (7 mL) and the resulting precipitate filtered off. The solution was purified by semi-preparative HPLC, and the solvent removed *in vacuo* to give a pale brown oil (0.0080 g, 41 % yield). ^{1}H NMR (MeOH-d_4, 400 MHz, δ): 6.16 (dd, ^{3}J = 2.3 Hz, ^{3}J = 2.2 Hz, 2H), 5.57 (dd, ^{3}J = 2.4 Hz, ^{3}J = 2.2 Hz, 2H), 5.10 (d, ^{3}J = 3.6 Hz, 0.8 H, *H1β*), 4.59 (d, ^{3}J = 8.4 Hz, 0.2 H, *H1α*), 3.82, 3.70, 3.31, 3.17 (m, 8H), 2.27 (t, ^{3}J = 7.6 Hz, ^{3}J = 7.6 Hz, 2H), 1.57, 1.35 (m, 8H). ^{13}C NMR (MeOH-d_4, 100 MHz, δ): 194.36, 175.24, 166.38, 97.22, 92.76, 93.92, 87.89, 86.63, 78.20, 78.05, 73.25, 72.75, 70.84, 63.01, 58.86, 55.99, 40.63, 40.60, 37.03, 32.11, 30.24, 27.62, 27.18, 26.73. IR ν_{max} (cm^{-1}): 3305 (m, br), 2931 (w), 2360 (m), 2341 (m), 2024 (s), 1921 (s), 1675 (m), 1639 (m), 1553 (m), 1203 (m), 1136 (m). HR-MS (ES+ of MNa^{+}): m/z calcd for $C_{21}H_{27}N_2O_{10}{}^{185}Re$: 675.1093, found: 675.1086.

Tricarbonyl{8-amino-octylamido-*N*-(2-amino-2-deoxy-D-glucopyranose)cyclopentadienyl carboxamide}rhenium(I) (11b). Tricarbonyl(cyclopentadienyl carboxylic acid)rhenium (0.085 g, 0.22 mmol) and dicyclohexylcarbodiimide (DCC) (0.052 g, 0.25 mmol) were dissolved in dry dichloromethane (5 mL). The flask was purged with argon and the resulting solution

stirred at room temperature for 2.5 h. **10b** (0.25 g, 0.23 mmol, as estimated from crude ^{1}H NMR studies of debenzylation reaction shown above) and diisopropylethylamine (0.74 mL, 4.5 mmol) were stirred in dimethylformamide (5 mL) for 10 min, before addition to the activated rhenium precursor. The resulting reaction mixture was stirred under inert atmosphere at room temperature for 40 h, then the volume reduced on a rotary evaporator and the compound purified by semi-preparative HPLC. The solvent was removed *in vacuo* to give a pale brown oil (0.076 g, 50 % yield). ^{1}H NMR (MeOH-d_4, 400 MHz, δ): 6.16 (s, 2H), 5.57 (s, 2H), 5.10 (d, ^{3}J = 3.2 Hz, 0.75 H, *H1β*), 4.59 (d, ^{3}J = 8.4 Hz, 0.25 H, *H1α*), 3.73, 3.24 (m, 8H), 2.25 (dd, ^{3}J = 7.4 Hz, ^{3}J = 7.4 Hz, 2H), 1.84 (m, 4H), 1.39 (m, 6H). ^{13}C NMR (MeOH-d_4, 100 MHz, δ): 193.80, 176.33, 164.33, 96.78, 92.20, 95.63, 87.39, 86.07, 77.60, 75.64, 72.68, 72.19, 72.16, 71.88, 62.43, 58.27, 55.41, 40.28, 40.15, 36.96, 36.60, 29.91, 29.71, 29.65, 29.62, 29.38, 27.35, 26.48. IR ν_{max} (cm^{-1}): 3037 (w, br), 2934 (w), 2360 (s), 2341 (s), 2024 (s), 1921 (s), 1672 (m), 1636 (m), 1557 (w), 1201 (w), 1134 (w). HR-MS (ES+ of MNa^{+}): m/z calcd for $C_{23}H_{31}N_2O_{10}{}^{187}Re$: 705.1434, found: 705.1420.

Tricarbonyl{6-amino-hexylamido-*N*-(1,3,4,6-tetra-O-acetyl-2-amino-2-deoxy-β-D-glucopyranose)cyclopentadienyl carboxamide}rhenium(I) (12a). Tricarbonyl(cyclopentadienyl carboxylic acid)rhenium (0.058 g, 0.15 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDC) (0.044 g, 0.23 mmol) were dissolved in dry dichloromethane (5 mL). The flask was purged with argon for ten minutes and the resulting solution stirred at room temperature for three hours. 6-Amino-hexylamino-1,3,4,6-tetra-*O*-acetyl-2-deoxy-*D*-β-glucopyranose (**4a**)

(0.070 g, 0.090 mmol) in dry dichloromethane (3 mL) was added to the reaction mixture, followed by diisopropylethylamine (0.18 mL, 1.0 mmol). The resulting solution was stirred under inert atmosphere at room temperature for 24 h. The reaction mixture was washed twice with water (10 mL), once with brine (10 mL), and dried over $MgSO_4$. The drying agent was filtered out and the solution volume reduced on the rotary evaporator before purification by column chromatography on silica gel with 2 % methanol in dichloromethane as eluent. The solvent was removed *in vacuo* to give a colourless oil (17 mg, 14 % yield). ^{1}H NMR (DMSO-d_6, 400 MHz, δ): 8.17 (dd, ^3J = 5.6 Hz, ^3J = 5.6 Hz, 1H), 7.92 (d, ^3J = 9.2 Hz, 1H), 6.25 (dd, ^3J = 2.4 Hz, ^3J = 2.0 Hz, 2H), 5.73 (d, ^3J = 7.2 Hz, 1H), 5.70 (dd, ^3J = 2.4 Hz, ^3J = 1.6 Hz, 2H), 5.18 (dd, ^3J = 10.0 Hz, ^3J = 10.0 Hz, 1H), 4.88 (dd, ^3J = 10.0 Hz, ^3J = 10.0 Hz, 1H), 4.19 (dd, ^3J = 12.8 Hz, ^3J = 4.8 Hz, 1H), 3.96 (m, 3H), 3.09 (m, 2H), 2.03, 2.00, 1.97, 1.94 (s, 3H), 1.42, (m, 4H) 1.23 (m, 2H). ^{13}C NMR (DMSO-d_6, 400 MHz, δ): 194.06, 172.32, 169.96, 169.45, 169.20, 168.75, 161.11, 96.15, 91.71, 87.05, 86.14, 72.13, 71.50, 68.10, 61.46, 51.77, 38.51, 35.39, 28.66, 25.69, 24.95, 20.43, 20.35, 20.26. IR ν_{max} (cm^{-1}): 3305 (w, br), 2936 (w), 2359 (w), 2023 (s), 1916 (s), 1747 (s), 1643 (m), 1544 (m), 1388 (m), 1213 (s), 1034 (s), 737 (m), 596 (m), 511 (m). HR-MS (ES+ of MNa^+): m/z calcd for $C_{29}H_{35}N_2O_{14}{}^{187}Re$: 845.1544, found: 845.1567.

Tricarbonyl{8-amino-octylamido-*N*-(1,3,4,6-tetra-O-acetyl-2-amino-2-deoxy-β-D-glucopyranose)cyclopentadienyl carboxamide}rhenium(I) (12b). Tricarbonyl(cyclopentadienyl carboxylic acid)rhenium (0.024 g, 0.063 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDC) (0.015 g, 0.076

mmol) were dissolved in dry dichloromethane (5 mL). The flask was purged with argon and the resulting solution stirred at room temperature for four hours. 8-Amino-octylamido-1,3,4,6-tetra-*O*-acetyl-2-deoxy-*D*-β-glucopyranose (**4b**) (0.070 g, 0.090 mmol) and diisopropylethylamine (0.18 mL, 1.0 mmol) were stirred in dry dichloromethane (2 mL) for 10 min, and then added to the activated acid solution. The resulting solution was stirred under inert atmosphere at room temperature overnight. Once TLC had confirmed the consumption of starting material, the solution was washed twice with water (7 mL), once with brine (10 mL), and dried over $MgSO_4$. The drying agent was filtered out and the solution volume reduced on the rotary evaporator before purification by column chromatography on silica gel with 1.5 % methanol in dichloromethane as eluent. The solvent was removed *in vacuo* to give the product as a colourless oil (2 mg, 5 % yield). ^{1}H NMR (MeOH-d_4, 400 MHz, δ): 6.16 (dd, ^3J = 2.3 Hz, ^3J = 2.0 Hz, 2H), 5.78 (d, ^3J = 8.6 Hz, 1H), 5.57 (dd, ^3J = 2.4 Hz, ^3J = 2.4 Hz, 2H), 5.28 (dd, ^3J = 10.6 Hz, ^3J = 9.4 Hz, 1H), 5.02 (dd, ^3J = 9.4 Hz, ^3J = 10.2 Hz, 1H), 4.28 (dd, ^3J = 12.5 Hz, ^3J = 4.7 Hz, 1H), 4.10 (dd, ^3J = 12.5 Hz, ^3J = 2.4 Hz, 1H), 4.06 (d, ^3J = 9.0 Hz, 1H), 3.91 (ddd, ^3J = 4.7 Hz, ^3J = 2.3 Hz, ^3J = 10.2 Hz, 1H), 3.26 (dd, ^3J = 6.6 Hz, ^3J = 6.6 Hz, 2H), 2.13 (dd, ^3J = 7.3 Hz, ^3J = 7.3 Hz, 2H), 2.07, 2.05, 2.01, 1.98 (s, 12H), 1.55, (m, 4H), 1.29 (m, 6H). ^{13}C NMR (MeOH-d_4, 100 MHz, δ): 194.34, 176.65, 172.45, 171.82, 171.37, 170.74, 164.86, 96.23, 93.51, 87.94, 87.91, 86.62, 86.59, 73.97, 73.91, 69.94, 63.10, 54.19, 40.64, 37.26, 30.51, 30.13, 30.12, 27.85, 26.90, 20.82, 20.79, 20.70. IR ν_{max} (cm^{-1}): 3308 (w, br), 2931 (m), 2360 (s), 2341 (s), 2023 (s), 1920 (s), 1750 (s), 1646 (m), 1545 (m), 1218 (s), 1038 (m). HR-MS (ES+ of MNa^+): m/z calcd for $C_{31}H_{39}N_2O_{14}{}^{187}Re$:

873.1857, found: 873.1875.

2-*N*-(6-Amino-hexylamido-1,3,4,6-tetra-O-acetyl-2-amino-2-deoxy-D-glucopyranose)- carboxamide-1-ferrocene (13a). Ferrocenecarboxylic acid (0.359 g, 1.56 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDC) (0.329 g, 1.72 mmol) were dissolved in dry dichloromethane (10 mL). The flask was purged with argon and the resulting mixture stirred at room temperature for six hours. 6-Amino-hexylamido-1,3,4,6-tetra-*O*-acetyl-2-deoxy-*D*-β-glucopyranose (**4a**) (amount of debenzylated product quantified by ^{1}H NMR, 1.40 mmol) and diisopropylethylamine (2.8 mL, 16 mmol) were stirred in dry dichloromethane (3 mL) for 10 min, before addition via syringe to the activated ferrocene. The resulting dark red-brown solution was stirred under inert atmosphere at room temperature for 40 h. The solution was washed twice with water (2 x 10 mL), once with brine (15 mL), and dried over $MgSO_4$. The drying agent was filtered out and the solution volume reduced on the rotary evaporator before purification by column chromatography on silica gel with 1 % methanol in dichloromethane followed by 2.5 % methanol in dichloromethane as eluent. The solvent was removed *in vacuo* to give an orange oil (0.124 g, 12 % yield). ^{1}H NMR ($CDCl_3$, 400 MHz, δ): 6.05 (s, 1H), 5.93 (d, ^{3}J = 9.0 Hz, 1H), 5.75 (d, ^{3}J = 9.0 Hz, 1H), 5.21 (dd, ^{3}J = 9.4 Hz, ^{3}J = 9.4 Hz, 1H), 5.14 (dd, ^{3}J = 9.4 Hz, ^{3}J = 9.0 Hz, 1H), 4.70 (dd, ^{3}J = 2.0 Hz, ^{3}J = 2.0 Hz, 2H), 4.35 (dd, ^{3}J = 2.0 Hz, ^{3}J = 2.0 Hz, 2H), 4.28 (m, 2H), 4.22 (s, 5H), 4.13 (dd, ^{3}J = 1.9 Hz, ^{3}J = 12.4 Hz, 1H), 3.80 (ddd, ^{3}J = 9.8 Hz, ^{3}J = 4.8 Hz, ^{3}J = 2.3 Hz, 1H), 3.37 (dd, ^{3}J = 6.6 Hz, ^{3}J = 6.2 Hz, 2H), 2.17 (dd, ^{3}J = 7.4 Hz, ^{3}J = 7.4 Hz, 2H), 2.10, 2.10, 2.04, 2.03 (s, 12H), 1.61 (m, 4H), 1.38 (m, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ): 173.00, 170.93,

170.91, 169.38 169.20, 99.91, 92.51, 76.63, 72.82, 72.76, 70.32, 69.95, 68.04, 67.85, 61.60, 52.82, 38.92, 36.10, 29.16, 25.91, 24.56, 20.84, 20.65, 20.51. IR ν_{max} (cm^{-1}): 3295 (w), 2934 (w), 1740 (s), 1633 (s), 1538 (s), 1366 (m), 1222 (m), 1035 (s). HR-MS (ES+ of MNa^+): m/z calcd for $C_{31}H_{40}{}^{56}FeN_2O_{11}$: 695.1879, found: 695.1895.

2-*N*-(8-Amino-octylamido-1,3,4,6-tetra-O-acetyl-2-amino-2-deoxy-D-glucopyranose)- carboxamide -1-ferrocene (13b). Ferrocenecarboxylic acid (0.050 g, 0.22 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDC) (0.046 g, 0.24 mmol) were dissolved in dry dichloromethane (8 mL). The flask was purged with argon and the resulting mixture stirred at room temperature for four hours. 8-Amino-octylamido-1,3,4,6-tetra-*O*-acetyl-2-deoxy-*D*-β-glucopyranose (**4b**) (0.116 g, 0.240 mmol) and diisopropylethylamine (0.42 mL, 2.4 mmol) were stirred in dry dichloromethane (5 mL) for 10 min, before addition to the activated acid mixture. The resulting solution was stirred under inert atmosphere at room temperature overnight. Once TLC had confirmed the consumption of starting material the solution was washed twice with water (20 mL), once with brine (20 mL), and dried over $MgSO_4$. The drying agent was filtered out and the solution reduced on the rotary evaporator before purification by column chromatography on silica gel with 1.5 % methanol in dichloromethane as eluent. The solvent volume was removed *in vacuo* to give an orange oil (0.022 g, 15 % yield). ^{1}H NMR ($CDCl_3$, 400 MHz, δ): 5.94 (d, ^3J = 9.4 Hz, 1H), 5.84 (dd, ^3J = 8.6 Hz, ^3J = 8.6 Hz, 1H), 5.71 (d, ^3J = 8.6 Hz, 1H), 5.19 (dd, ^3J = 9.4 Hz, ^3J = 9.4 Hz, 1H), 5.12 (dd, ^3J = 9.4 Hz, ^3J = 9.4 Hz, 1H), 4.68 (d, ^3J = 2.0 Hz, 2H), 4.34 (d, ^3J = 2.0 Hz, 2H), 4.26 (m, 2H), 4.20 (s, 5H), 4.11 (dd, ^3J = 2.4 Hz, ^3J = 12.5 Hz, 1H), 3.77 (ddd, ^3J = 9.8 Hz, ^3J = 4.7 Hz, ^3J = 2.3 Hz, 1H), 3.36 (dd, ^3J = 6.4 Hz, ^3J = 7.0 Hz, 2H),

2.13 (d, ^{3}J = 7.4 Hz, 7.4 Hz, 2H), 2.10, 2.09, 2.03, 2.02 (s, 12H), 1.57 (m, 4H), 1.32 (m, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ): 173.39, 171.18, 170.88, 170.51, 169.66 169.49, 92.84, 76.65, 73.05, 72.81, 70.56, 69.94, 68.29, 68.13, 61.90, 52.97, 39.50, 36.60, 29.96, 28.76, 28.61, 26.58, 25.46, 21.12, 20.93, 20.89. IR ν_{max} (cm^{-1}): 3292 (w), 2939 (w), 2360 (s), 2341 (s), 1750 (s), 1630 (m), 1541 (m), 1218 (s), 1038 (m). HR-MS (ES+ of MNa^{+}): m/z calcd for $C_{33}H_{44}{}^{56}FeN_2O_{11}$: 723.2192, found: 723.2187.

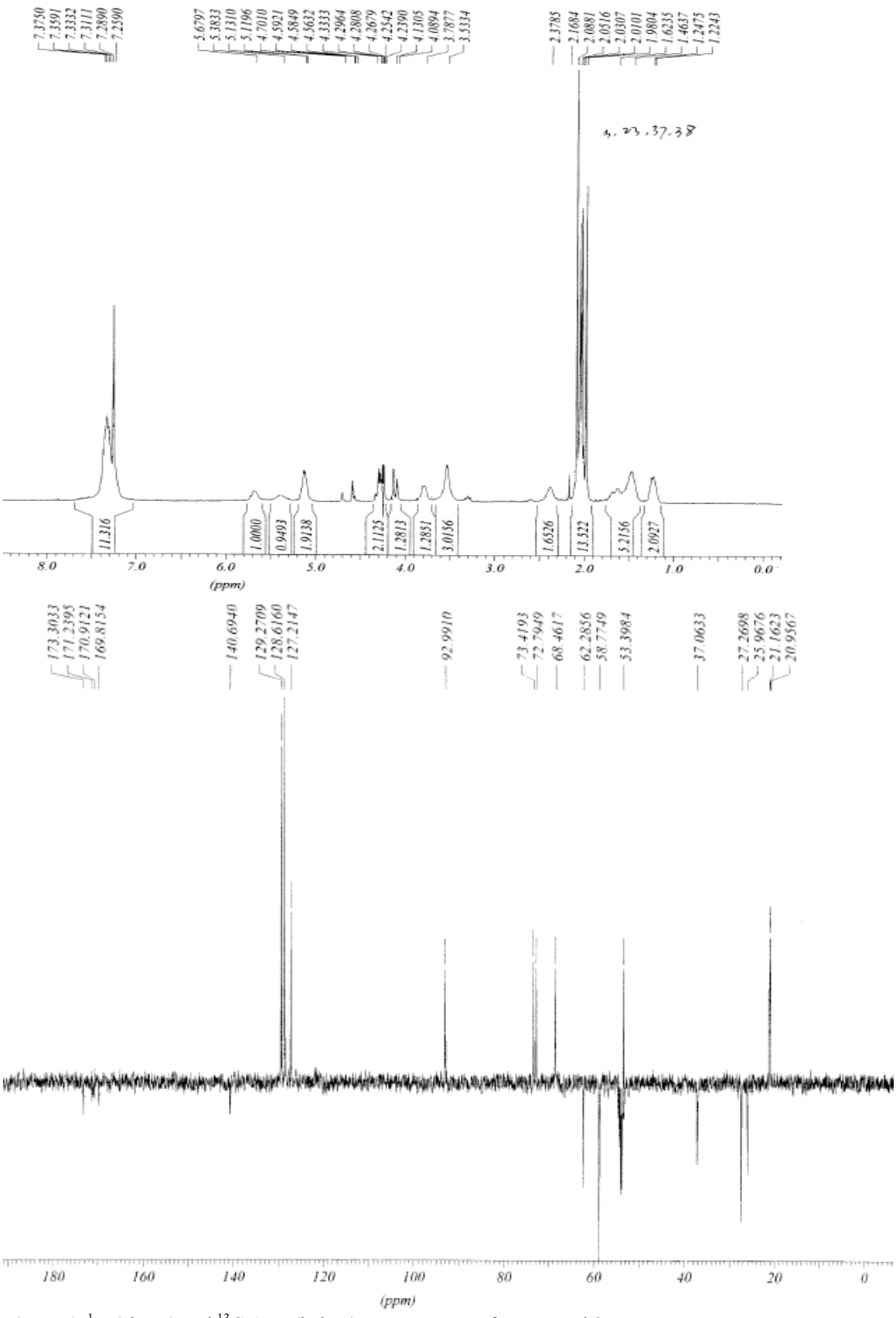

Figure 1. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **3a**.

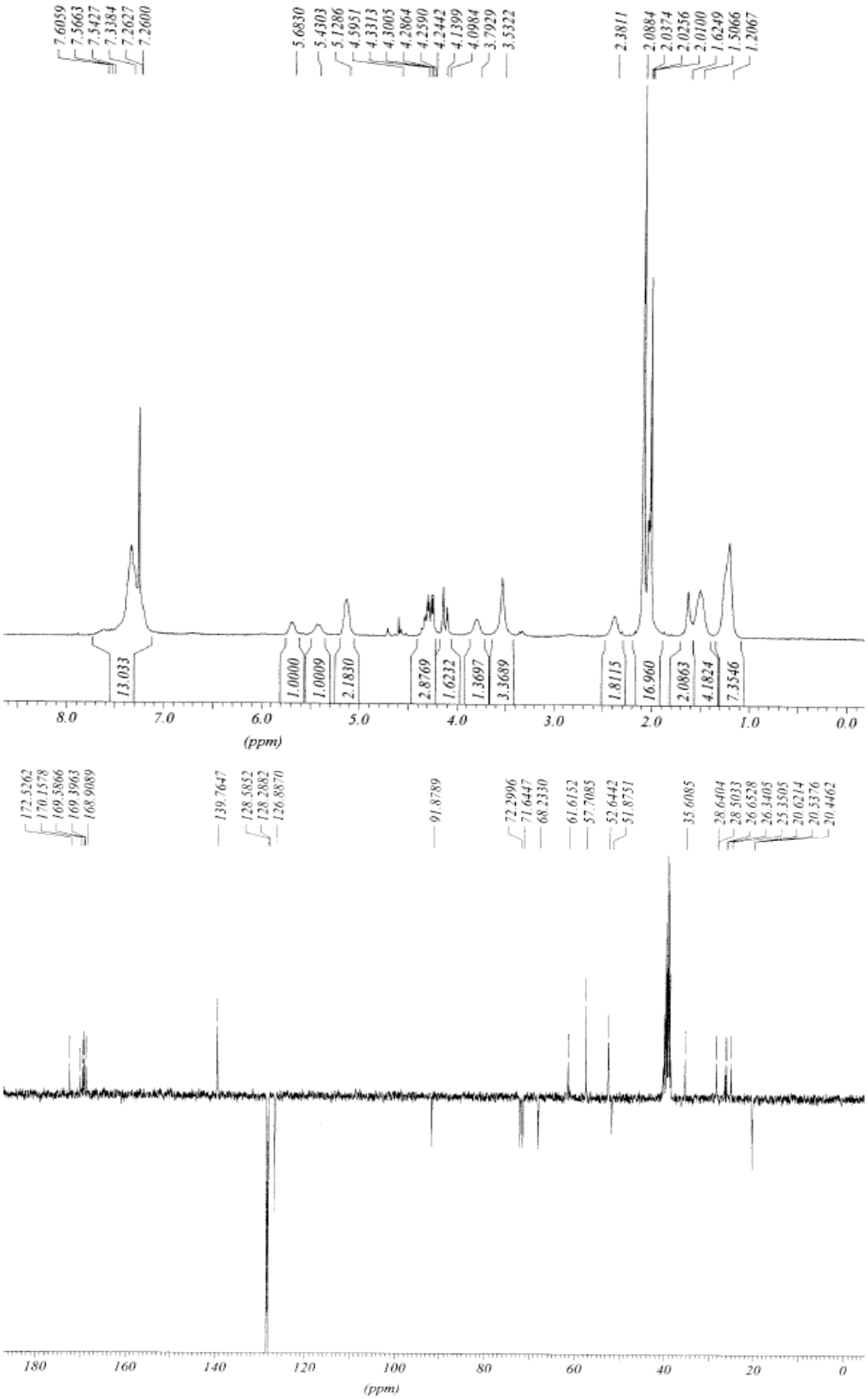

Figure 2. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **3b**.

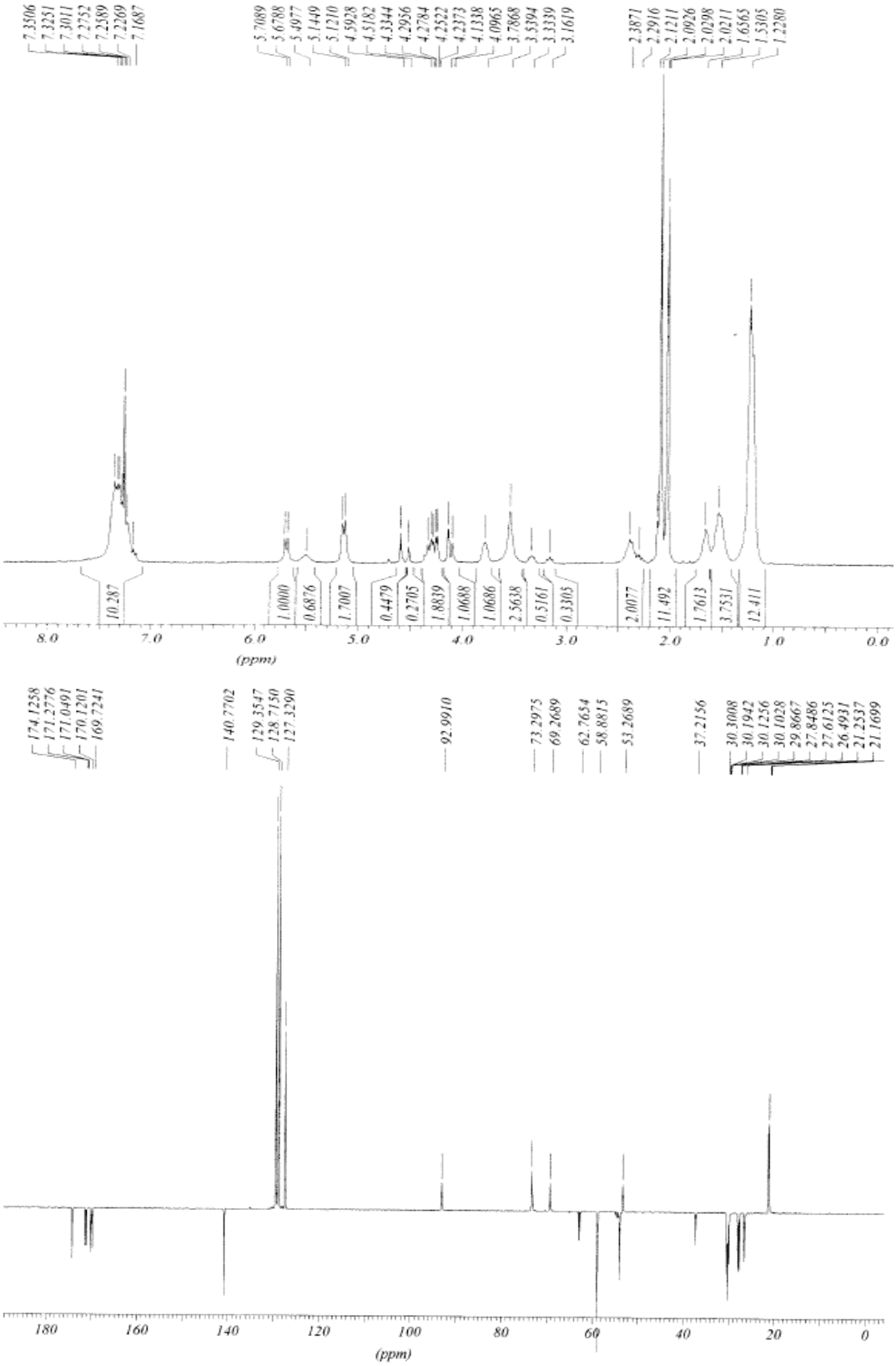

Figure 3. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **3c**.

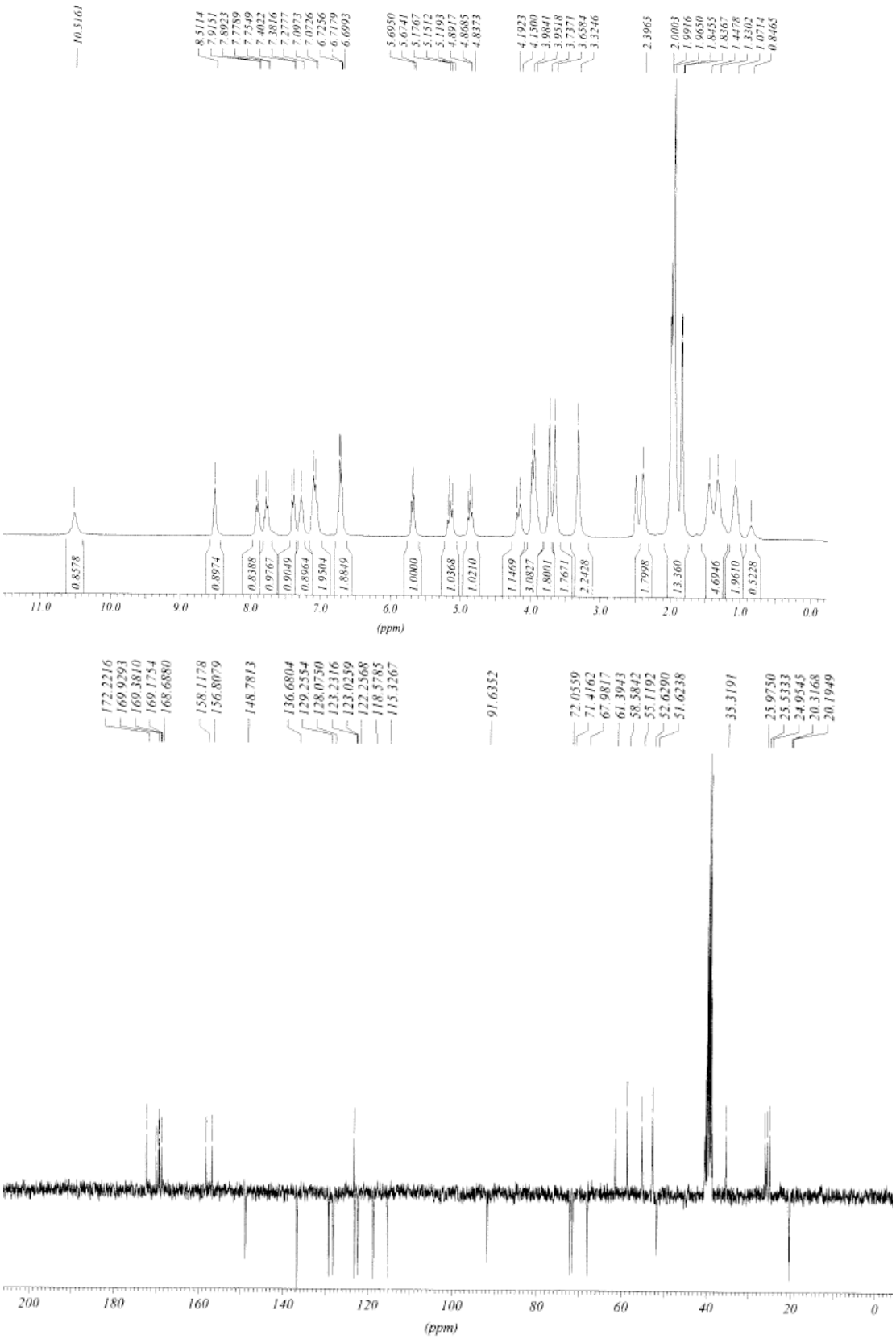

Figure 4. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **6a**.

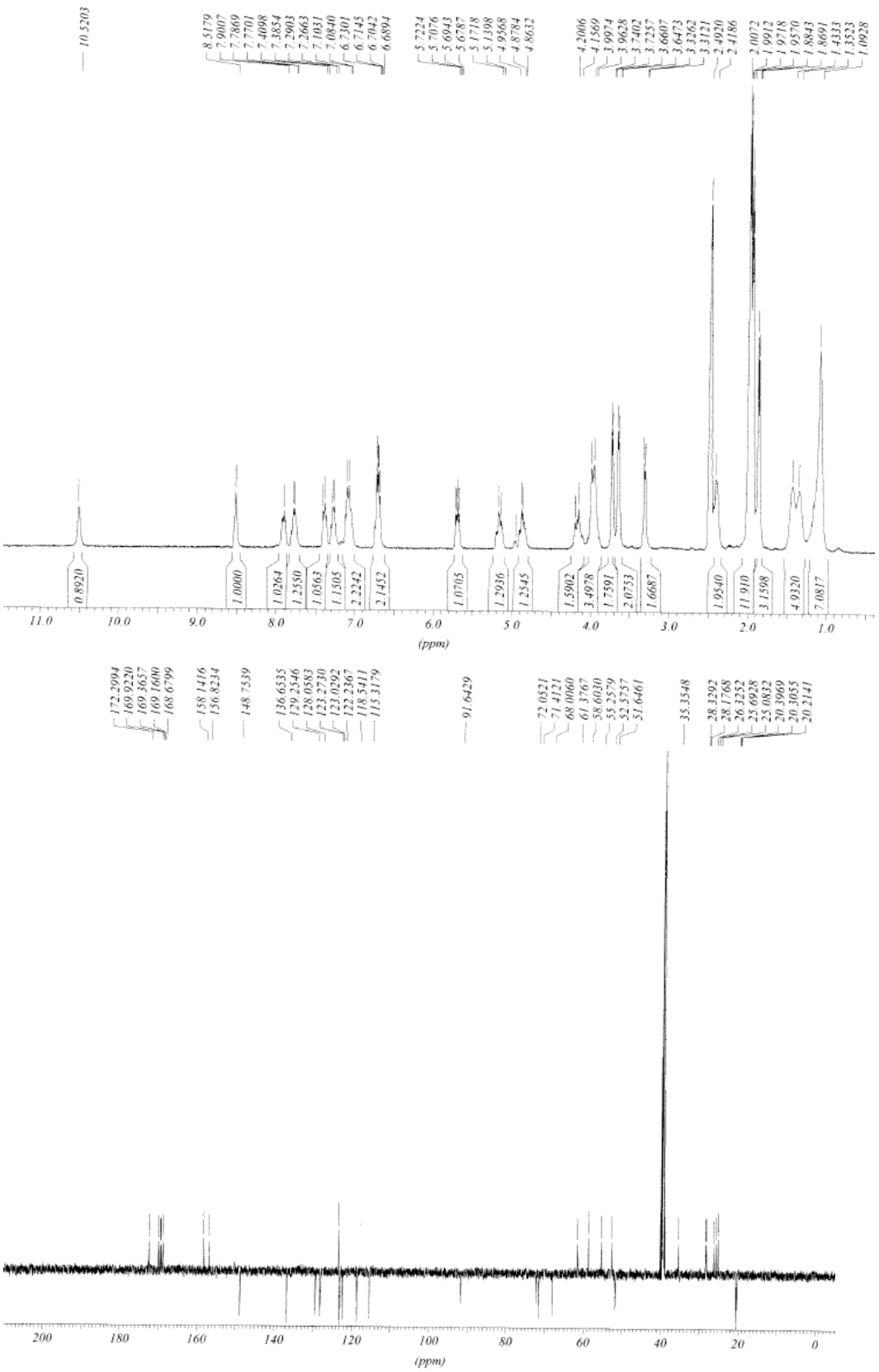

Figure 5. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **6b**.

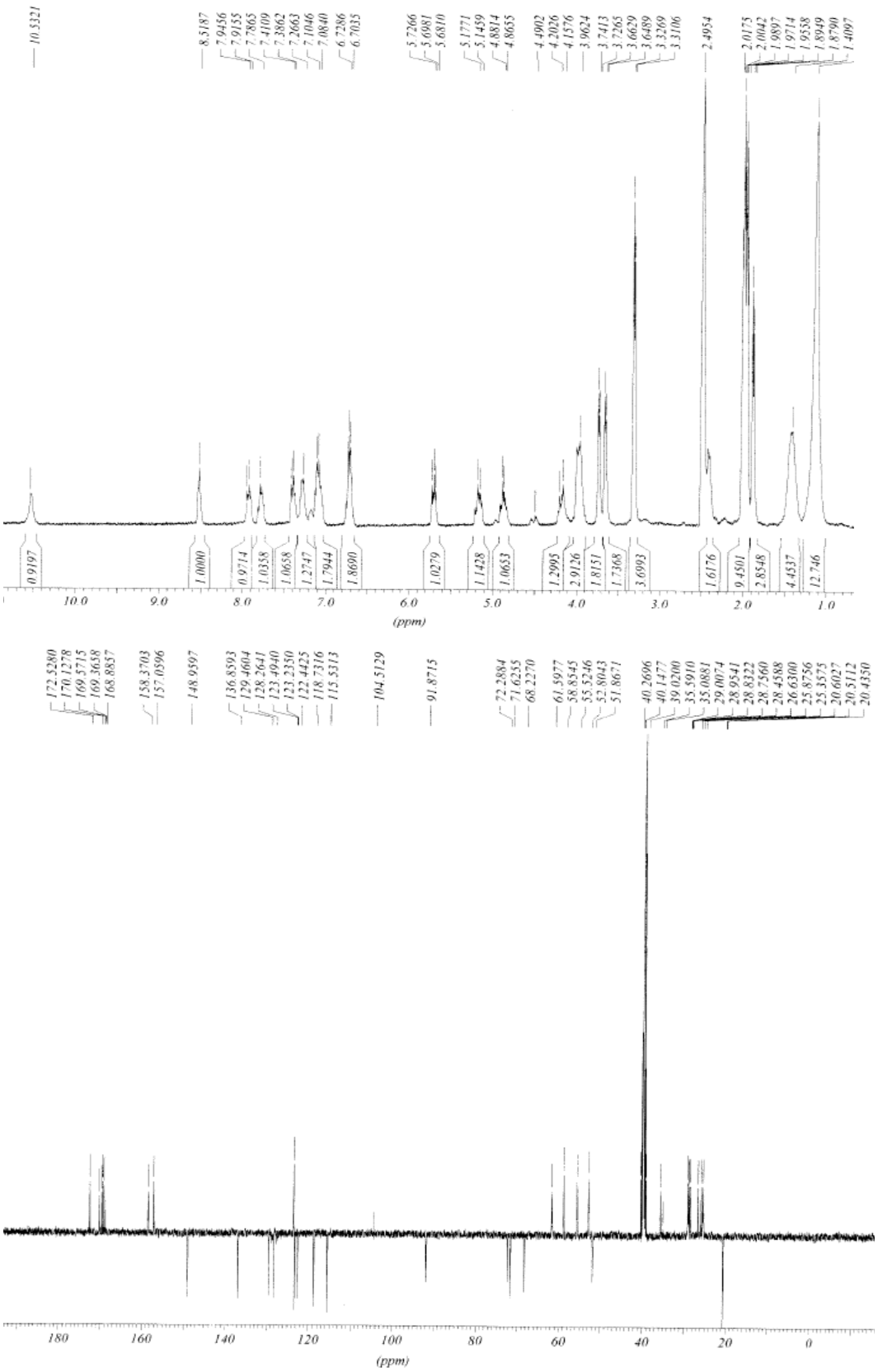

Figure 6. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **6c**.

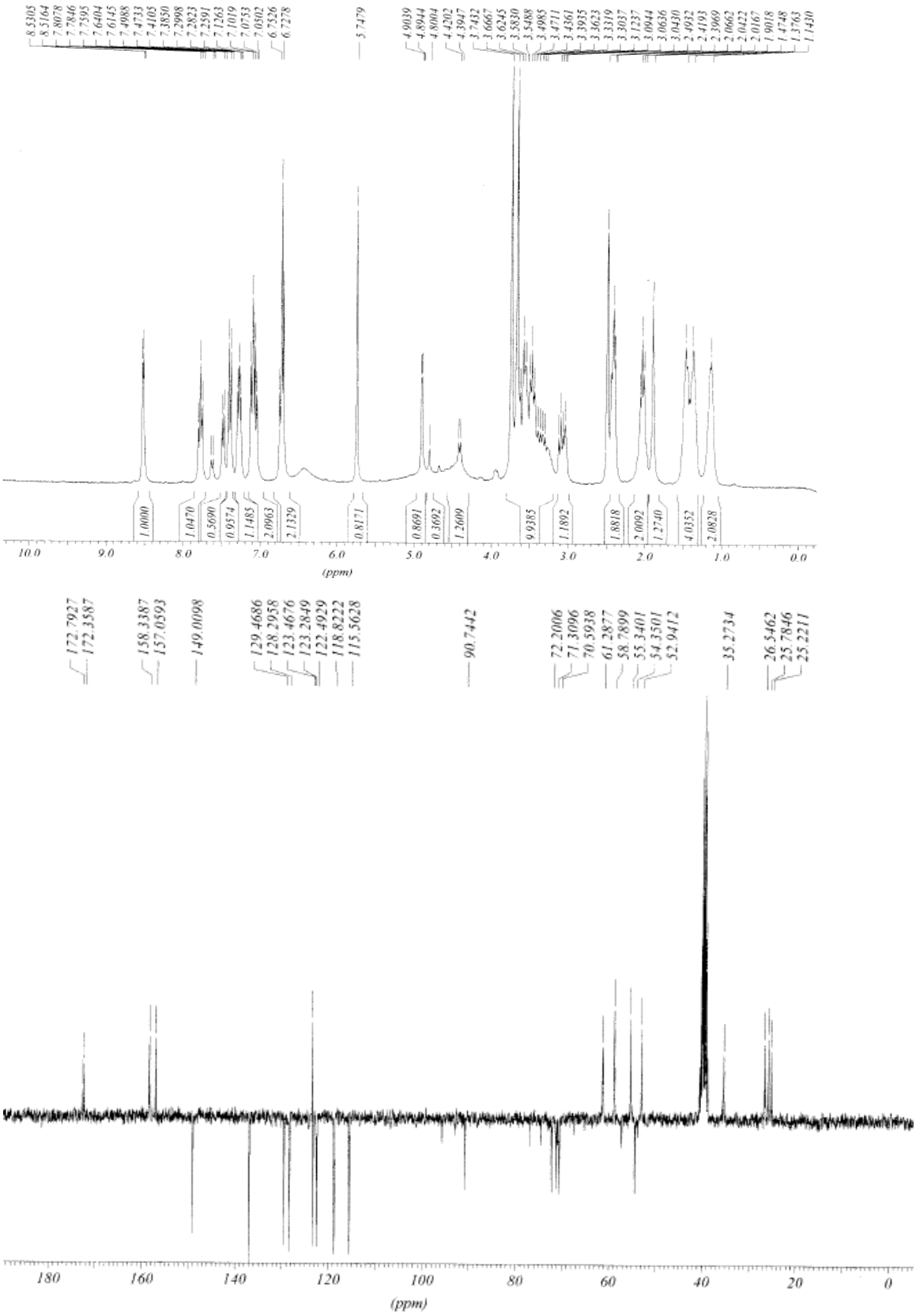

Figure 7. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **7a**.

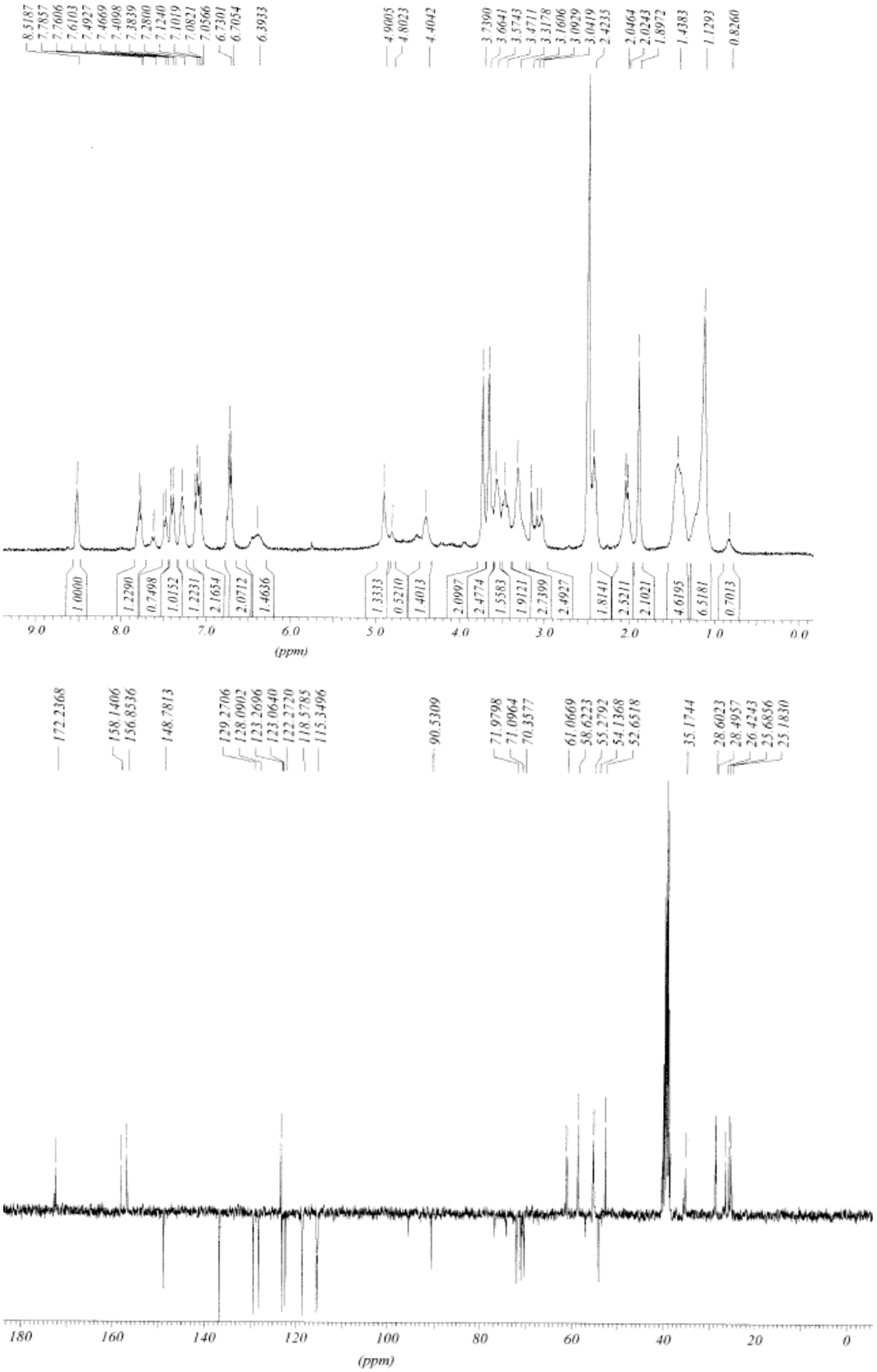

Figure 8. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **7b**.

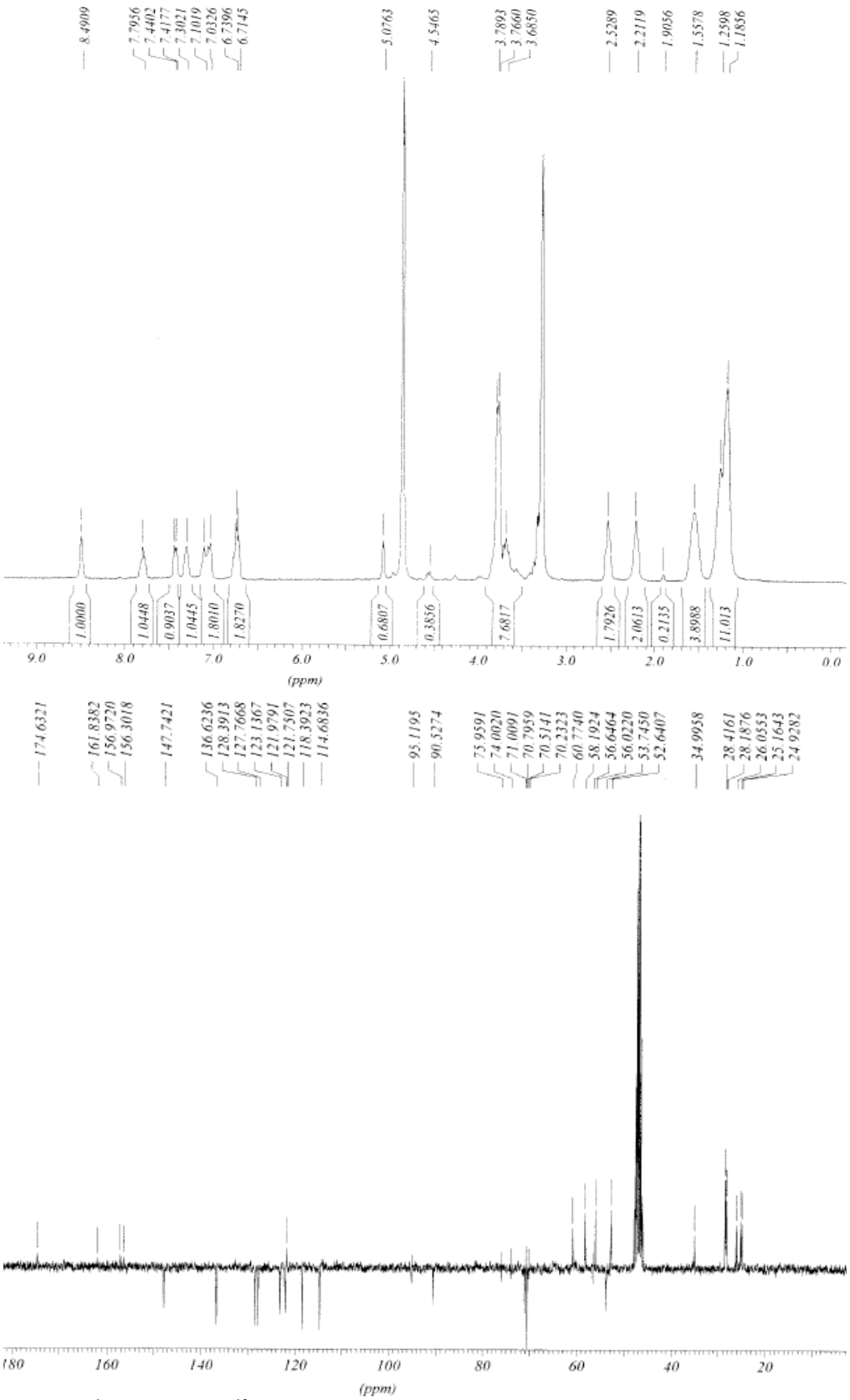

Figure 9. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **7c**.

Figure 10. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **8a**.

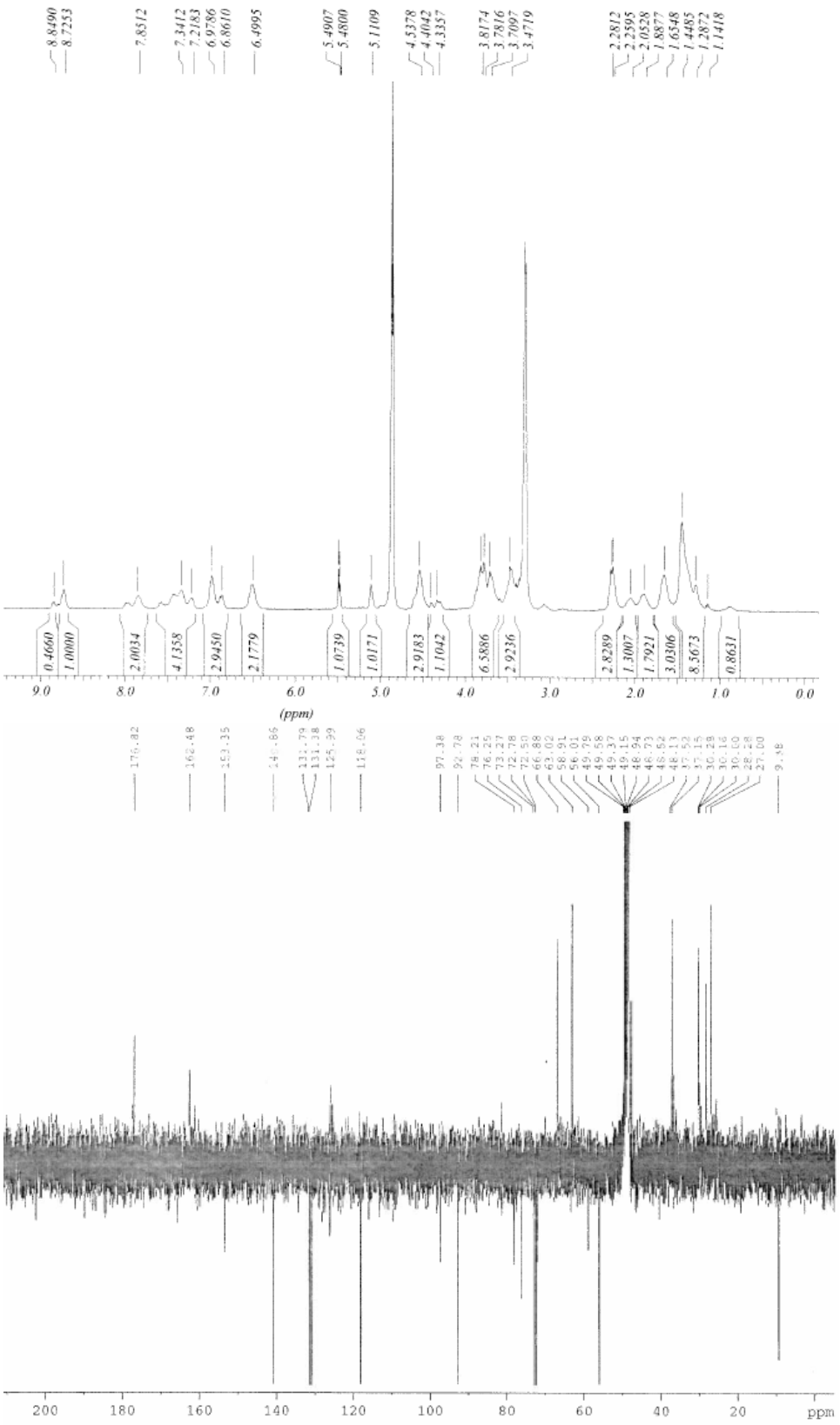

Figure 11. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **8b**.

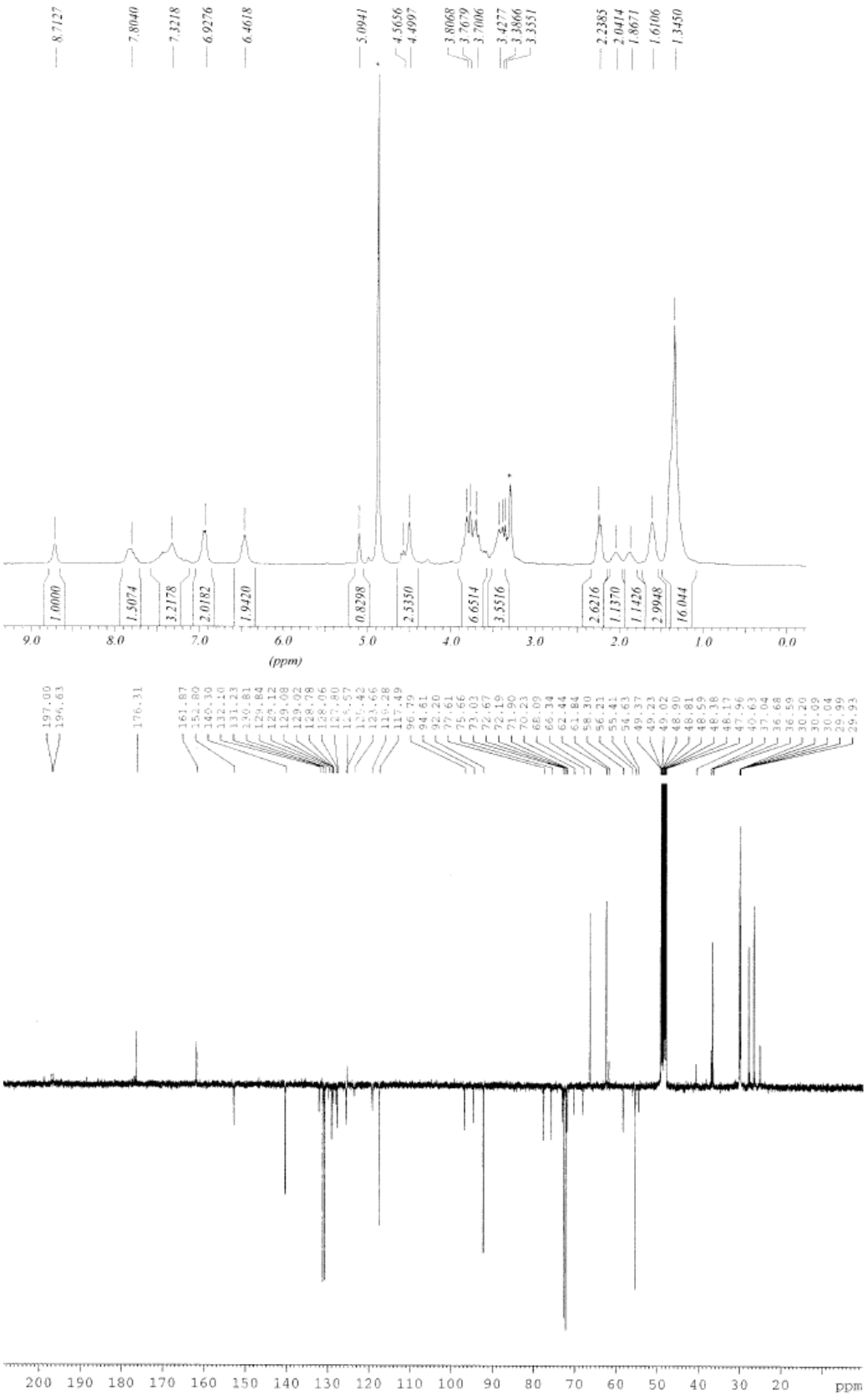

Figure 12. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **8c**.

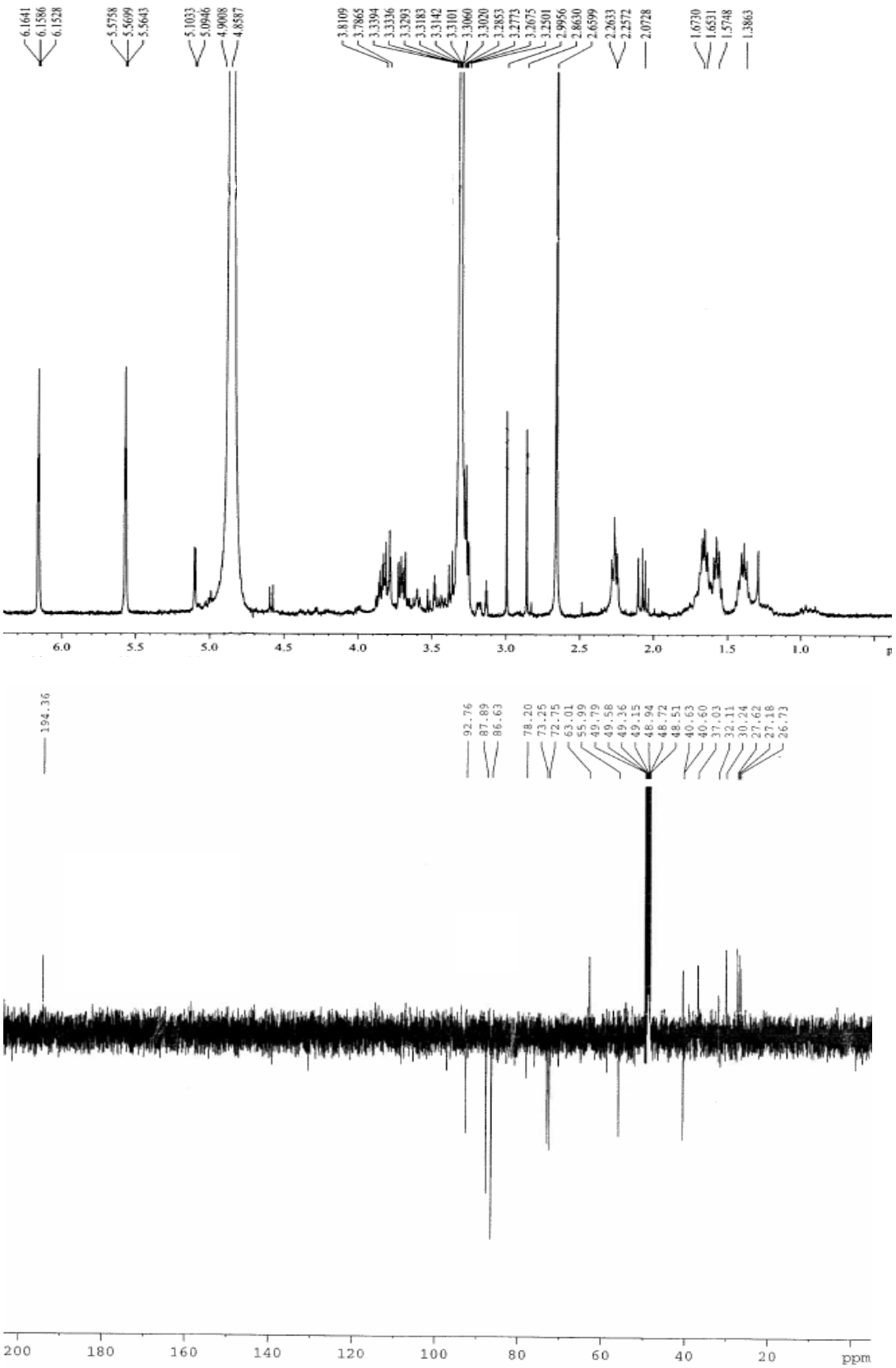

Figure 13. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **11a**.

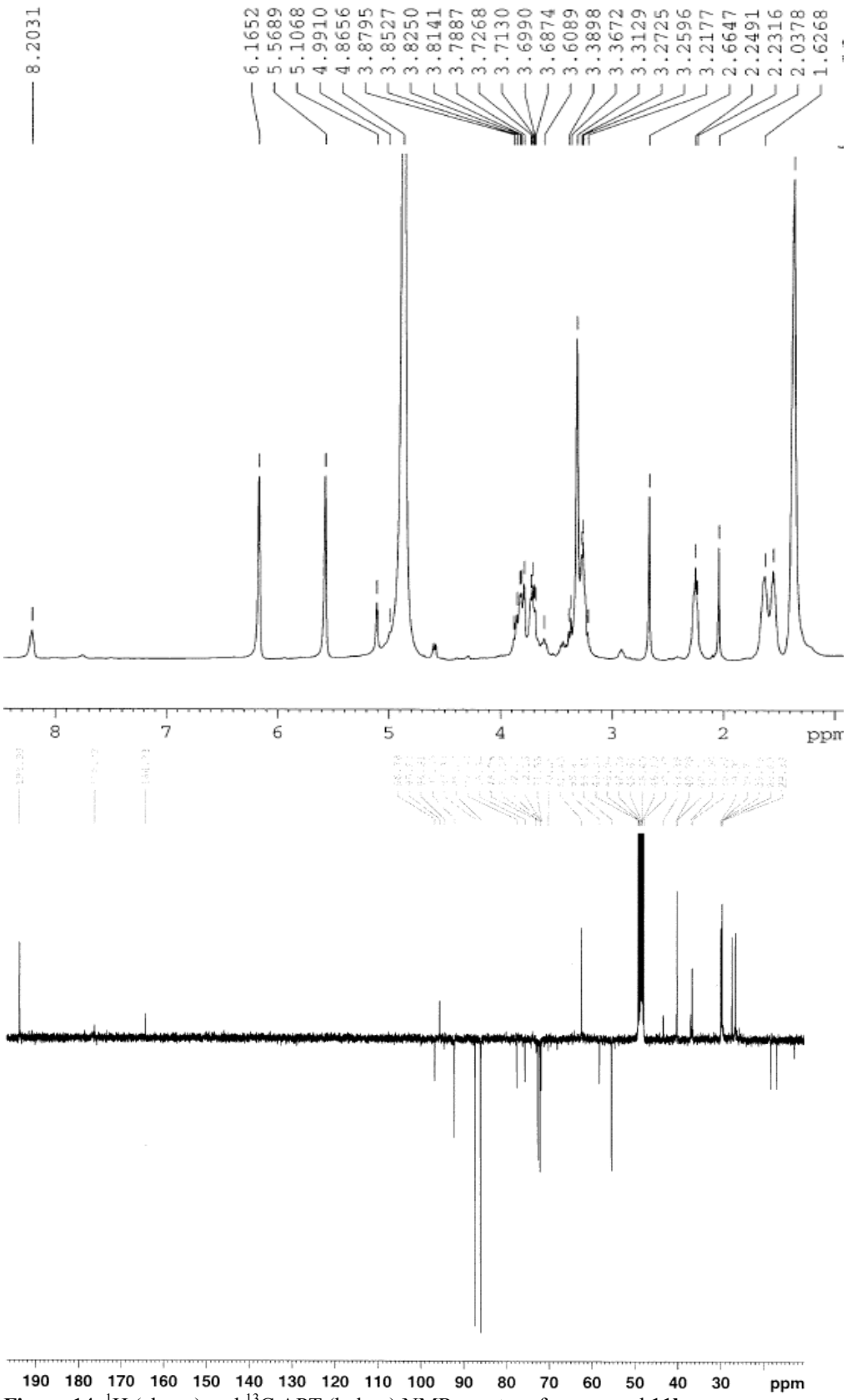

Figure 14. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **11b**.

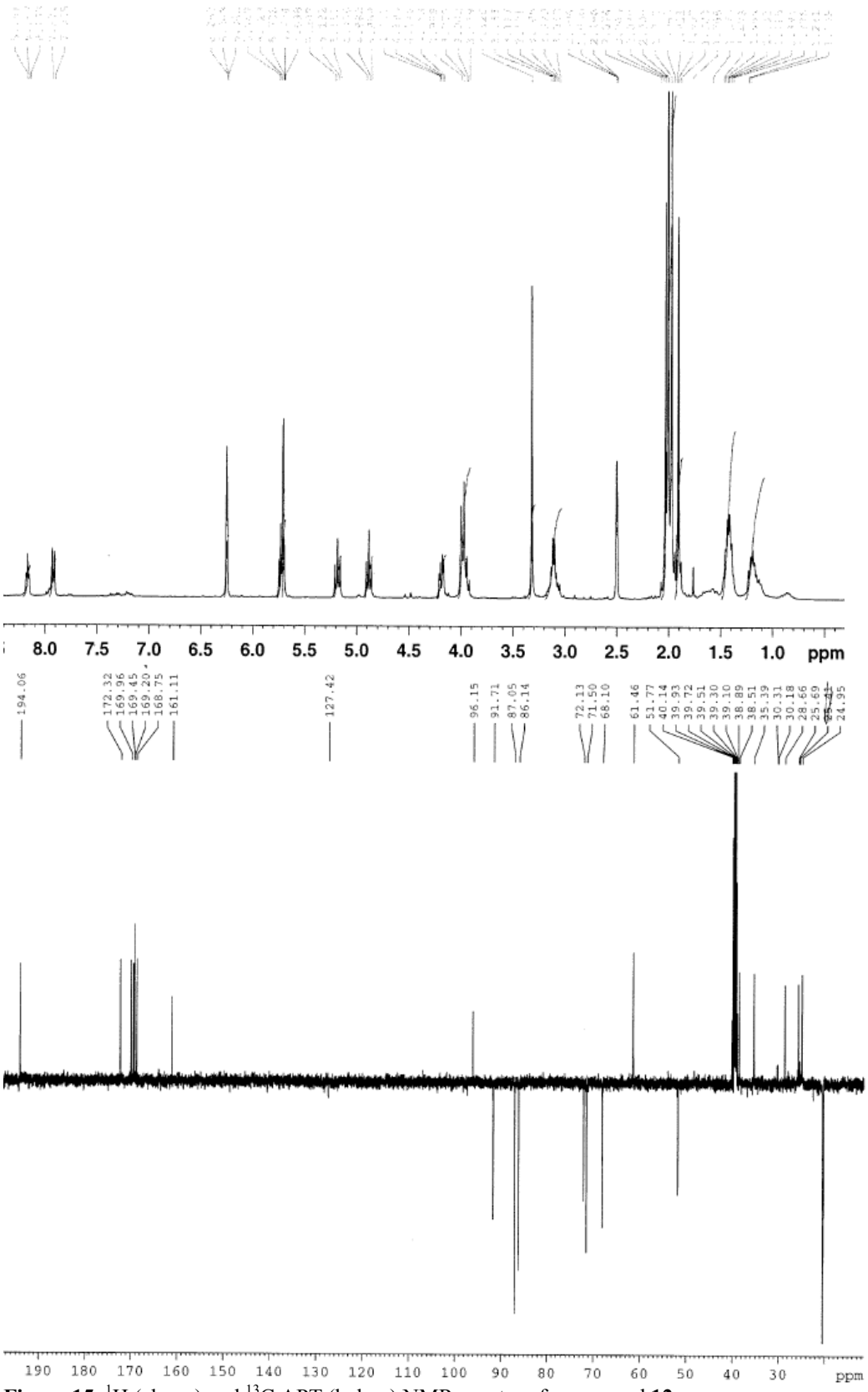

Figure 15. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **12a**.

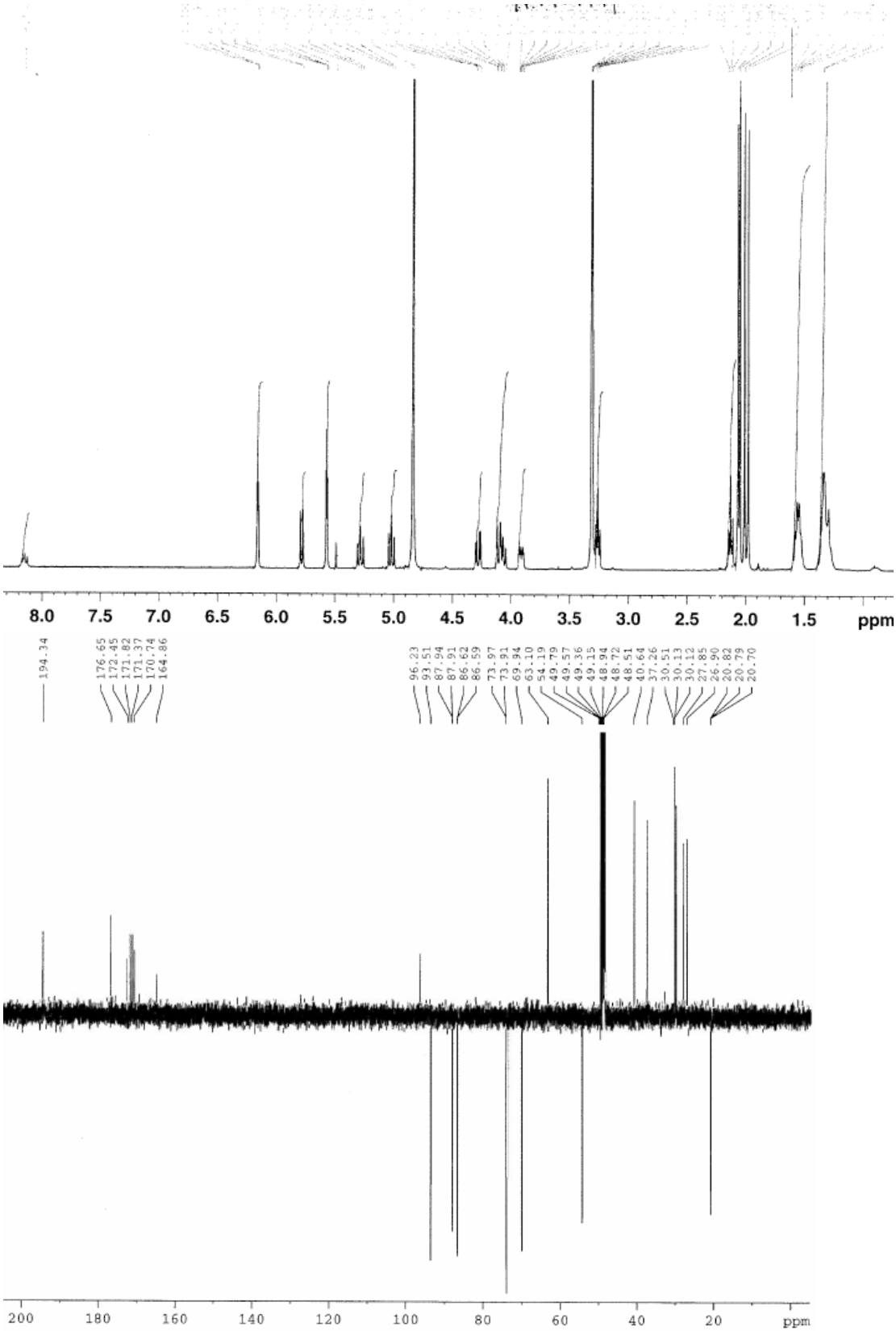

Figure 16. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **12b** .

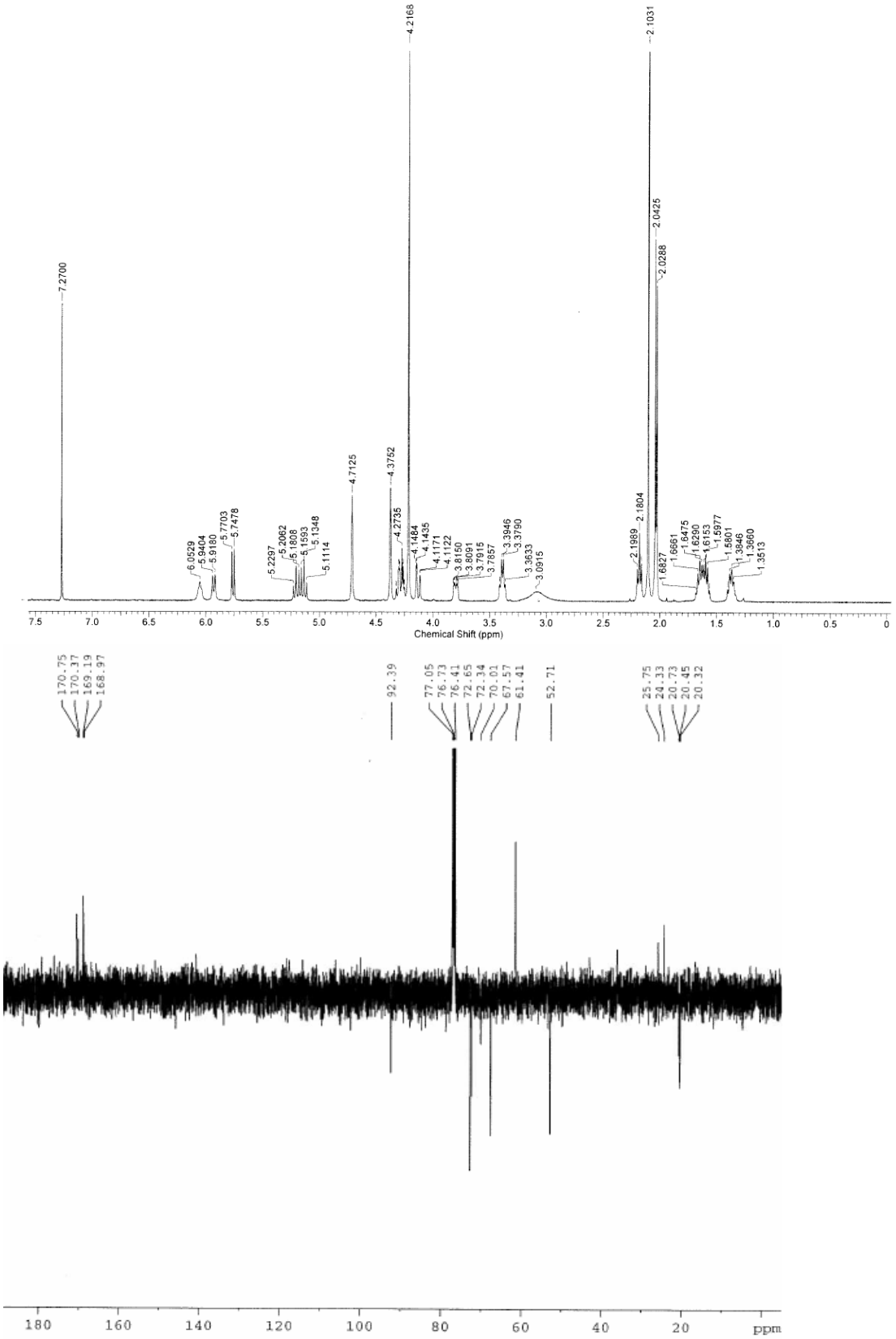

Figure 17. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **13a**.

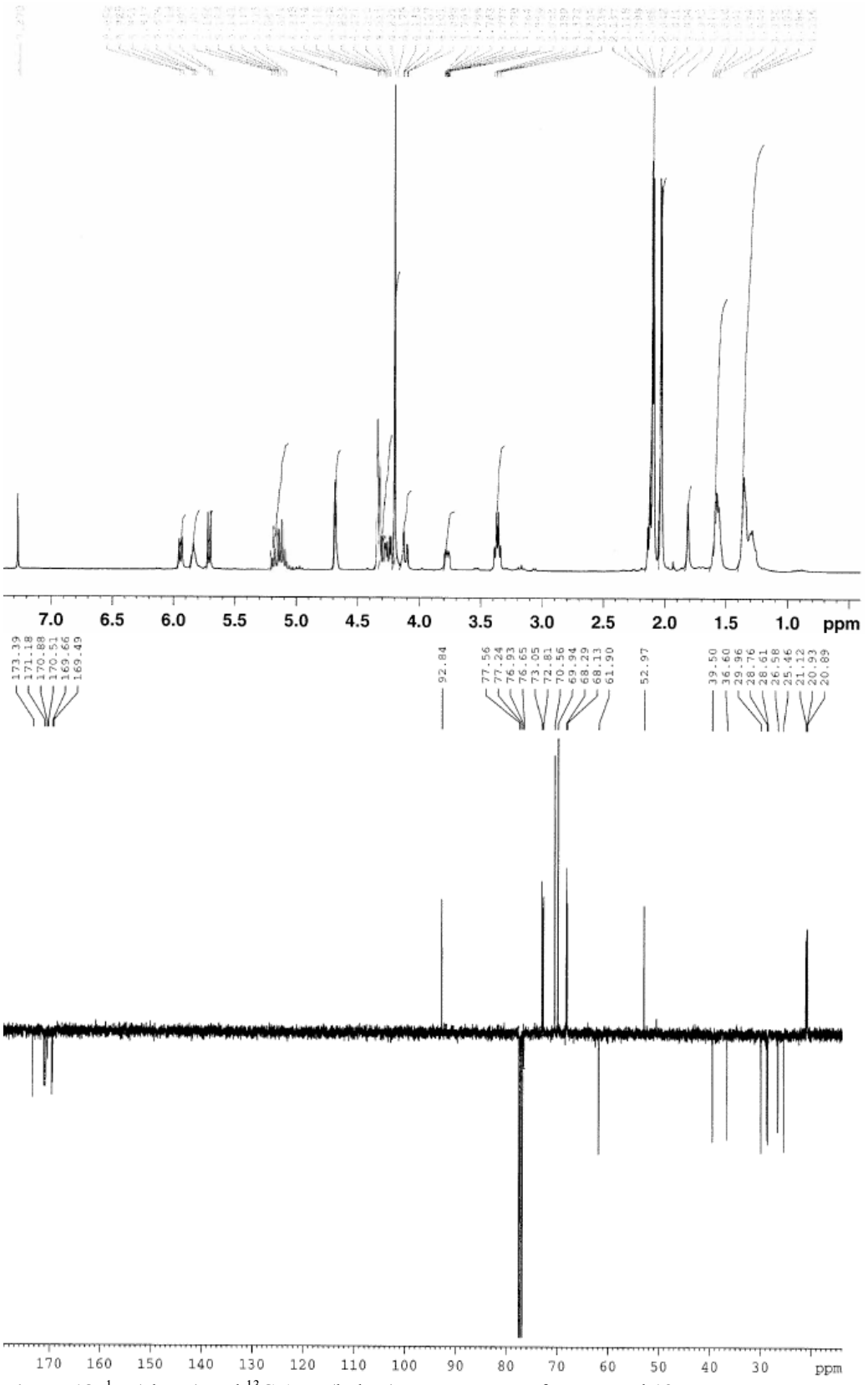

Figure 18. ^{1}H (above) and ^{13}C APT (below) NMR spectra of compound **13b**.