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Supplementary Information

Ligand- free Cu-catalyzed odorless synthesis of unsymmetrical sulfides through cross-coupling reaction of aryl/benzyl/alkyl halides with aryl boronic acids/S_8 system as a thiolating agent in PEG

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Index

1. Experimental Sections

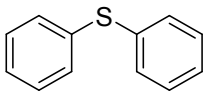
1.1. General information

All materials were obtained from commercial sources and used as received. ^{1}H NMR and ^{13}C NMR spectra were recorded on 250 or 400 MHz at ambient temperature with $CDCl_3$ as the solvent, Chemical shifts are reported in parts per million (ppm). Chemical shifts for protons are reported in parts per million downfield and are referenced to residual protium in the NMR solvent ($CHCl_3$ = δ 7.26). Chemical shifts for carbon are reported in parts per million downfield and are referenced to the carbon resonances of the solvent ($CDCl_3$ = δ 77.0). Data are represented as follows: chemical shift, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz), integration.

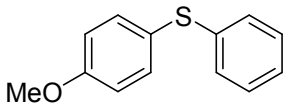
1.2. General procedure for the synthesis of unsymmetrical sulfides using aryl boronic acid and aryl/benzyl/alkyl halides:

A one-necked flask was charged with CuI (20-40 mg, 0.1-0.2 mmol), sodiuom hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), aryl/benzyl/alkyl halide (1 mmol), aryl boronic acid (1.1 mmol) and PEG200 (2 mL). The mixture was magnetically stirred and heated at 40-60 ºC for the appropriate reaction time (Table 2). After completion of the reaction, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. Evaporation of the solvent and purification by flash column chromatography on silica gel (EtOAc/*n*-hexane) gave the desired sulfides in 65-98% yields.

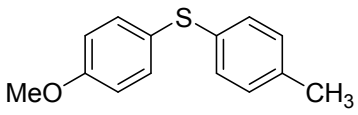
2. The Synthetic Procedure and Analytical Data for Table 2



A mixture of CuI (20 mg, 0.1 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), iodobenzene (1 mmol), phenyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 40 ^oC for 4.5 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. The mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 50:1) to afford the desired product diphenyl sulfide **(Table 2, entry1):**[1] ^1H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ= 7.2-7.3 ppm (m, 10 H); ^{13}C NMR (100MHz, $CDCl_3$, 25 °C, TMS): δ= 127.5, 129.8, 131.6, 135.9 ppm.

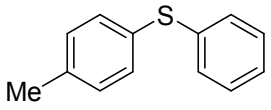


A mixture of CuI (20 mg, 0.1 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-iodoanisol (1 mmol), phenyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 ^oC for 9 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 30:1) to afford the desired product 4-methoxy diphenyl sulfide **(Table 2, entry 3):**[3] ^1H NMR (250 MHz, $CDCl_3$, 25 °C, TMS): δ= 4.2(s, 3H), 7.1-7.2 ppm (m, 5H), 7.3-7.6 (m, 4H); ^{13}C NMR (62.9 MHz, $CDCl_3$, 25 °C, TMS): δ= 55.3, 116.3, 126.8, 127.4, 128.5, 129.4, 137.4, 138.2, 158.2 ppm.

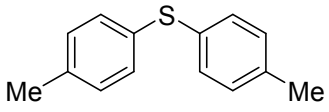


A mixture of CuI (30 mg, 0.15 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-iodoanisol (1 mmol), *p*-tolyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 ^oC for 11 h. After the reaction was

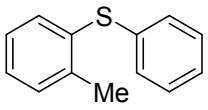
complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 30:1) to afford the desired product (4-methoy phenyl) *p*tolyl- sulfide **(Table 4, entry 4)**: [5] ^1H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ= 2.3 ppm (s, 3H), 3.8 (s, 3H), 6.91 (d, *J* = 8.4 Hz, 2H) , 7.11 (d, *J* = 7.6 Hz, 2H) 7.18, (d, *J* = 8.4 Hz, 2H) 7.41, (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C, TMS): δ= 21.0, 55.3, 114.9, 125.6, 129.4, 129.8, 134.4, 136.1, 159.5 ppm.



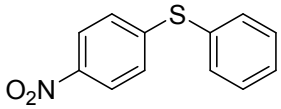
A mixture of CuI (20 mg, 0.1 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-methyliodobenzene (1 mmol), phenyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 50 °C for 9 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 40:1) to afford the desired product 4-methyl diphenyl sulfide **(Table 2, entry 5):**[3] ^1H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ= 2.5 ppm (s, 3H), 7.1-7.4(m, 9H);^{13}C NMR (100 MHz, $CDCl_3$, 25 °C, TMS): δ= 21.2, 126.1, 127.0, 127.7, 129.1, 129.4, 131.0, 133.6, 137.0 ppm.



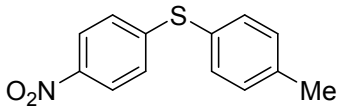
A mixture of CuI (30 mg, 0.15 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-methyliodobenzene (1 mmol), *p*-tolylboronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 °C for 10 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 40:1) to afford the desired product di-*p*-tolyl sulfide **(Table 2, entry 6)**:[1] ^1H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ= 2.25 ppm (s, 6H), 7.25 (d, *J*= 8, 4H), 7.34 (d, *J*= 8, 4H);^{13}C NMR (100 MHz, $CDCl_3$, 25 °C, TMS): δ= 21.0, 128.5, 129.8, 131.1, 137.4 ppm.



A mixture of CuI (20 mg, 0.10 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 2-methyliodobenzene (1 mmol), phenyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 ^oC for 12 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 40:1) to afford the desired product 2-methyl diphenyl sulfide **(Table 2, entry 7)**:[2] ^1H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ= 2.3 ppm (s, 3H), 7.1-7.4 (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C, TMS): δ= 20.6, 126.3, 127.9, 128.8, 129.6, 130.4, 131.0, 133.0, 135.7, 137.0, 139.9 ppm.

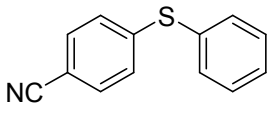


A mixture of CuI (20 mg, 0.10 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-nitroliodobenzene (1 mmol), phenyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 40 ^oC for 1.30 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 20:1) to afford the desired product 4-nitro diphenyl sulfide **(Table 2, entry 8):**[3] ^1H NMR (250 MHz, $CDCl_3$, 25 °C, TMS): δ= 7.0 ppm (d, 2H, *J*=7.5Hz), 7.3-7.4 (m, 5 H), 7.95 (d, 2H, *J*= 7.5 Hz); ^{13}C NMR (62.9 MHz, $CDCl_3$, 25 °C, TMS): δ= 124.0, 126.6, 129.6, 130.0, 130.4, 134.7, 145.3, 148.5 ppm.

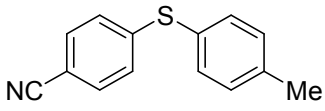


A mixture of CuI (20 mg, 0.10 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-nitroliodobenzene (1 mmol), *p*-tolylboronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 40 ^oC for 3 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was

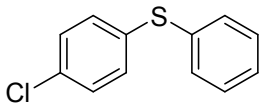
evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 20:1) to afford the desired product (4- nitro phenyl) *p*tolyl- sulfide **(Table 2, entry 9):**[5] ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ= 2.3 ppm (s, 3H), 7.14 (d, *J* =7.6 Hz, 2H) 7.29, (d, *J* = 7.6 Hz, 2H) 7.45, (d, *J* = 8 Hz, 2H) 8.06, (d, *J* = 8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C, TMS): δ= 21.3, 124.0 , 126.1, 126.4, 130.9, 135.1, 140.2, 145.1, 149.4 ppm.



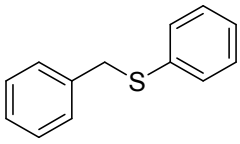
A mixture of CuI (20 mg, 0.10 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-nitryliodobenzene (1 mmol), phenyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 40 °C for 2.30 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 20:1) to afford the desired product 4-nitryl diphenyl sulfide **(Table 2, entry 6):**[4] ^{1}H NMR (250 MHz, $CDCl_3$, 25 °C, TMS): δ= 6.5-7.06 ppm (m, 5H), 7.31-7.9 (m, 4H); ^{13}C NMR (62.9 MHz, $CDCl_3$, 25 °C, TMS): δ= 108.5, 118.8, 127.3, 129.4, 129.9, 130.8 132.3, 132.8, 134.4, 145.7 ppm.



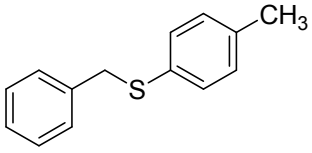
A mixture of CuI (20 mg, 0.10 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-nitrylliodobenzene (1 mmol), *p*-tolylboronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 40 °C for 4 h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 50:1) to afford the desired product (4nitryl phenyl) *p*tolyl- sulfide **(Table 2, entry 11)**:[3] ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ= 2.4 ppm (s, 3H), 7.13 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H) 7.43-7.49, (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C, TMS): δ= 21.3, 108.2, 118.9, 126.6, 130.7, 132.6, 133.4, 134.9, 140.0, 146.6 ppm.



A mixture of CuI (30 mg, 0.15 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 4-chloro bromobenzene (1 mmol), phenyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 ^oC for 21h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 40:1) to afford the desired product 4-chloro diphenyl sulfide **(Table 2, entry 20):**[3] ^1H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ= 7.1-7.4 ppm (m, 9H); ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C, TMS): δ= 125.6, 127.1, 127.4, 129.0, 131.0, 132.0, 135.7, 137.0 ppm.

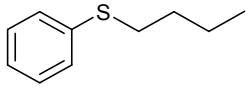


A mixture of CuI (40 mg, 0.2 mmol), sodiuom hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), benzyl bromide (1 mmol), phenyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 ^oC for 11h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (n-hexane /ethyl acetate 50:1) to afford the desired product Benzyl (phenyl) sulfide **(Table 2, entry 21):**[6] ^{1}HNMR (250 MHz, $CDCl_3$, 25 °C, TMS): δ= 4.01 ppm (s, 2H), 7.18-7.22 (m, 10H). ^{13}C NMR (62.9 MHz, $CDCl_3$, 25 °C, TMS): δ= 39.1, 126.5, 127.3, 127.5, 128.5, 128.9, 129.4, 129.8, 136.9, 137.7 ppm.

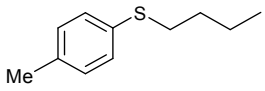


A mixture of CuI (40 mg, 0.2 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), benzyl bromide (1 mmol), *p*-tolyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 ^oC for 14h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was

evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 40:1) to afford the desired product benzyl (*p*-tolyl) sulfide **(Table 2, entry 22):**[6] ^{1}HNMR (250 MHz, $CDCl_3$, 25 °C, TMS): δ= 2.1 ppm (s, 3H), 3.94 (s, 2H), 6.96-6.92 (d, *J*= 8, 2H), 7.22-7.08 (m, 7H), ^{13}C NMR (62.9 MHz, $CDCl_3$, 25 °C, TMS): δ= 21.16, 39.79, 127.16, 128.51, 128.92, 129.10, 129.70, 130.71, 132.57, 136.58 ppm.



A mixture of CuI (40 mg, 0.2 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 1-bromobutane (1 mmol), boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 ^{o}C for 12h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 40:1) to afford the desired product butyl (phenyl) sulfide **(Table 2, entry 23):**[6] ^{1}HNMR (250 MHz, $CDCl_3$, 25 °C, TMS): δ=0.79-0.85 ppm (t, J= 7.5, 3H), 1.32-1.36 (m, 2H), 1.39-1.57 (m, 2H), 2.79-2.95 (t, J= 7.25, 3H), 7.01 -7.25 (m, 5H); ^{13}C NMR (62.9 MHz, $CDCl_3$, 25 °C, TMS): δ=13.67, 21.99, 31.23, 33.23, 125.6, 128.81, 128.81, 137.07 ppm.



A mixture of CuI (40 mg, 0.2 mmol), sodium hydroxide (160 mg, 4.0 mmol), S_8 (32 mg, 1 mmol), 1-bromobutane (1 mmol), *p*-tolyl boronic acid (1.1 mmol) and PEG200 (2 mL) in a one-necked flask was heated at 60 ^{o}C for 14h. After the reaction was complete, the reaction mixture was cooled to room temperature and H_2O (4 mL) was added. The product was extracted with EtOAc (3×4 mL) and dried over anhydrous Na_2SO_4. the mixture was evaporated under vacuum and purified by flash column chromatography (*n*-hexane /ethyl acetate 40:1) to afford the desired product hexyl (4-methoxyphenyl) sulfide **(Table 2, entry 24):**[6] ^{1}HNMR (250 MHz, $CDCl_3$, 25 °C, TMS): 0.83-0.78 (t, *J*= 7.25, 3H), 1.53-1.31 (m, 4H), 2.20 (s, 3H), 2.80-2.74 (t, *J*= 7.25, 2H), 6.99-6.96 (d, *J*= 8, 2H), 7.162-7.130 (d, *J*= 8, 2H); ^{13}C NMR (62.9 MHz, $CDCl_3$, 25 °C, TMS):, 13.69, 21.01, 21.97, 22.10, 31.37, 34.02, 129.62, 129.75, 133.22, 135.76.

References

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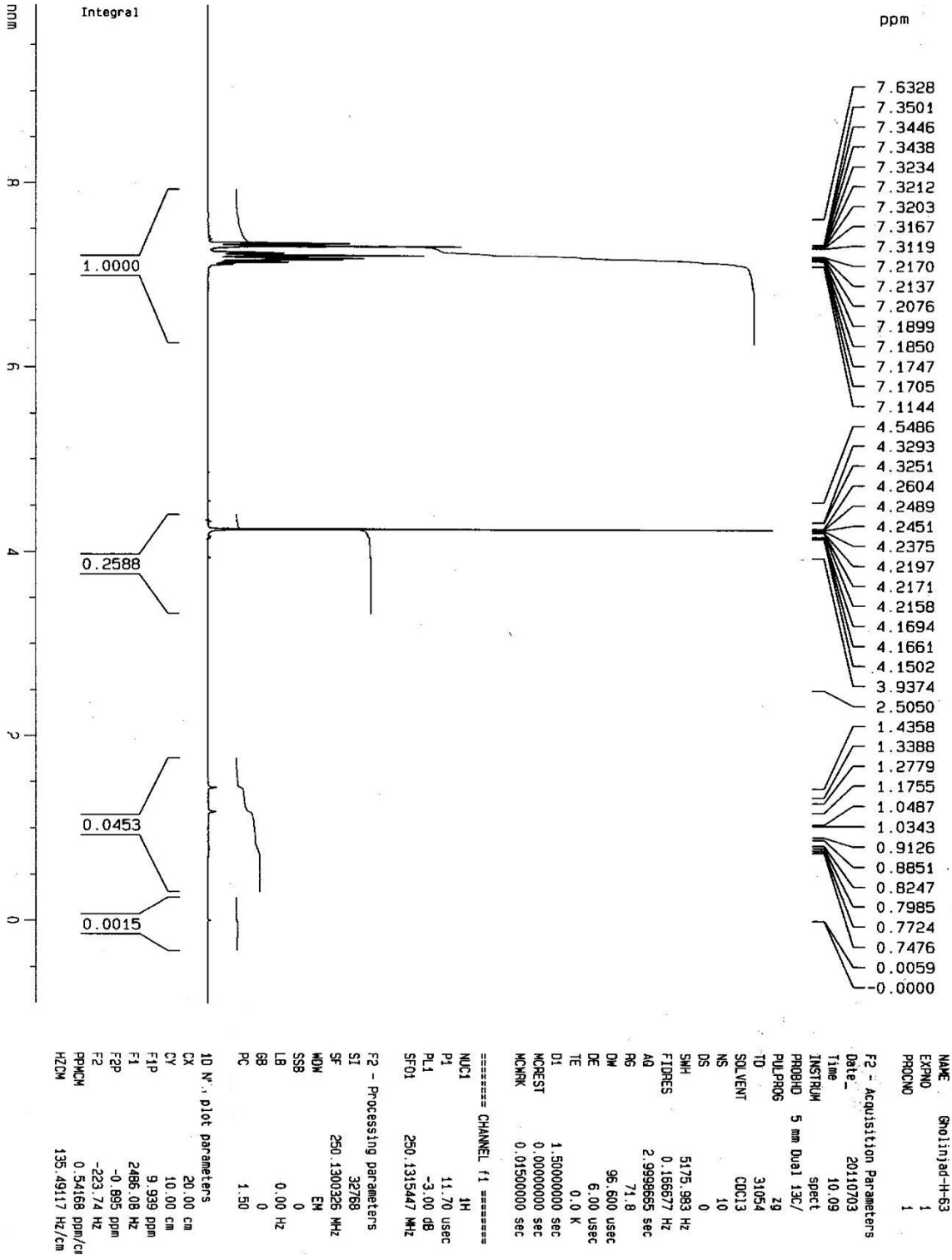
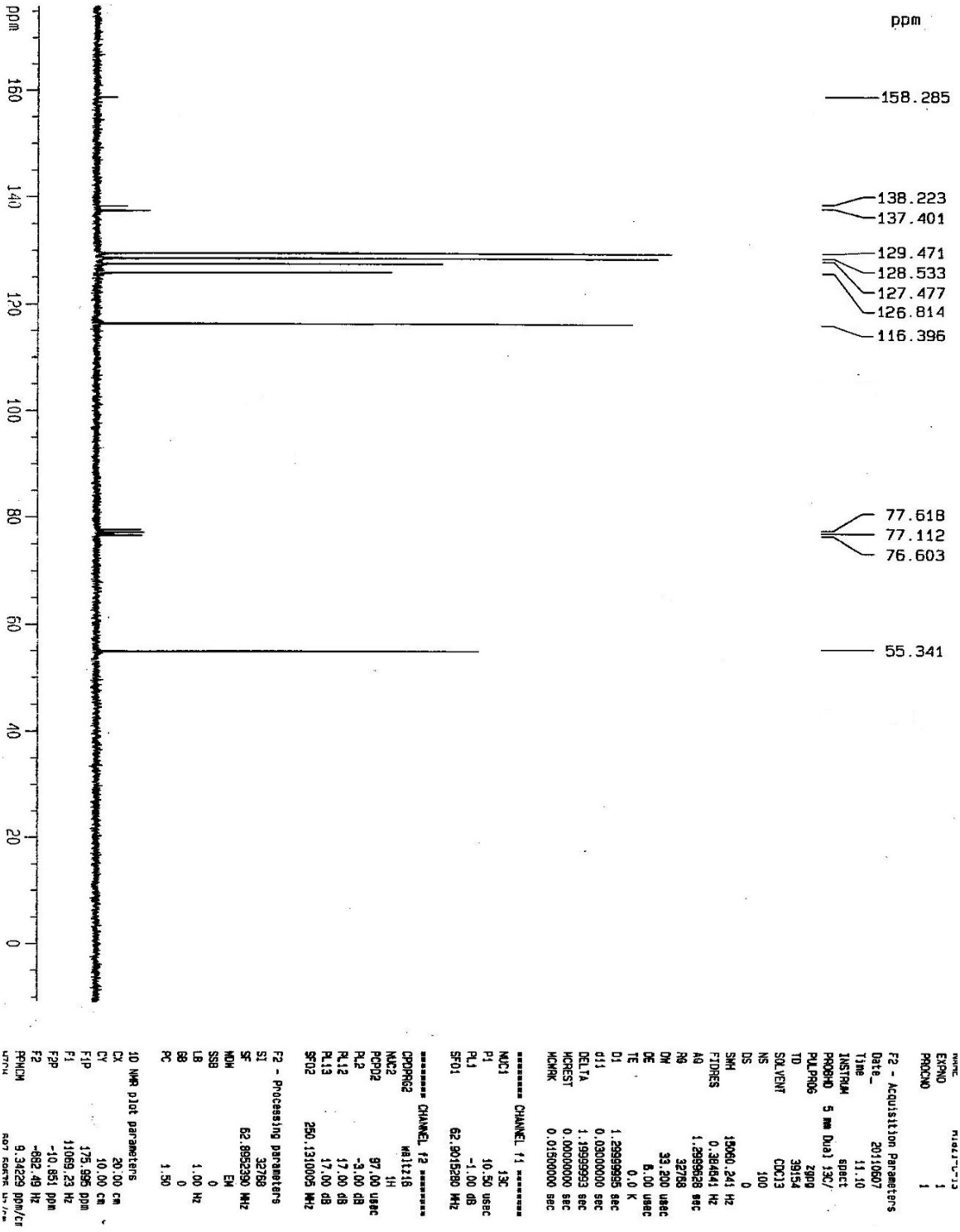
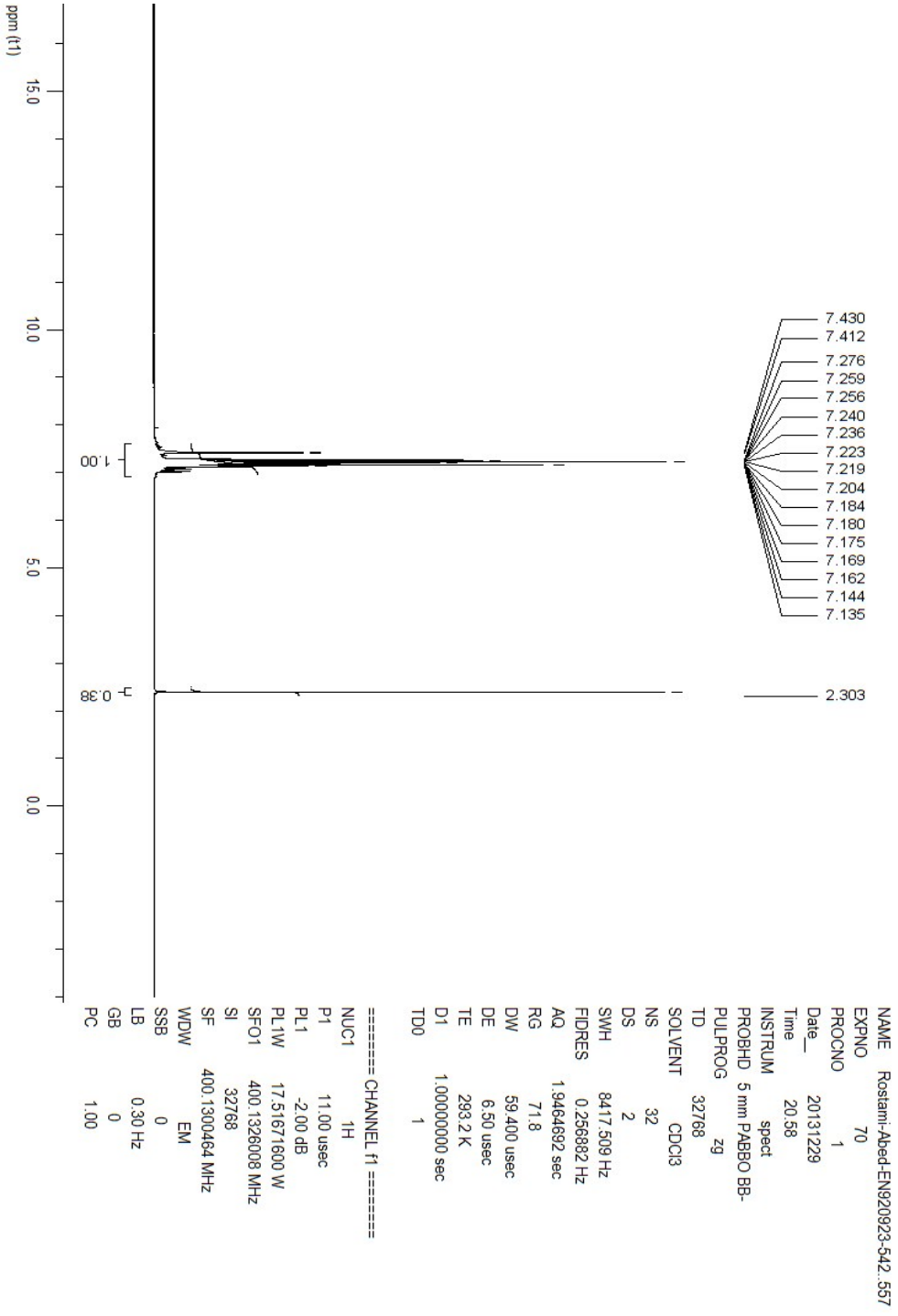
NMR Spectral: ^{1}H-NMR and ^{13}C-NMR



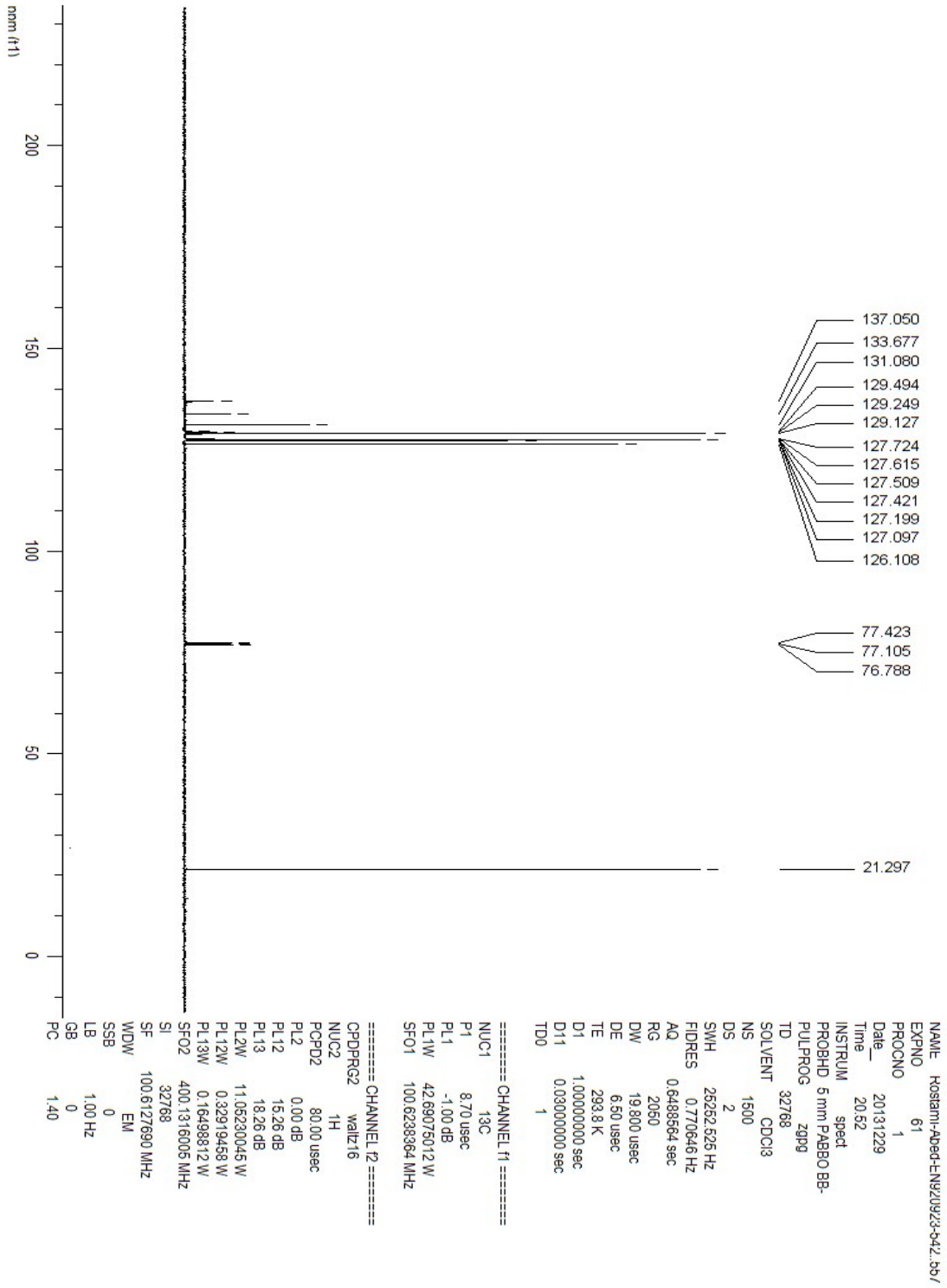
The ^{1}H-NMR spectrum of (4-Methoxy phenyl) sulfide (Table 2, entry 3)



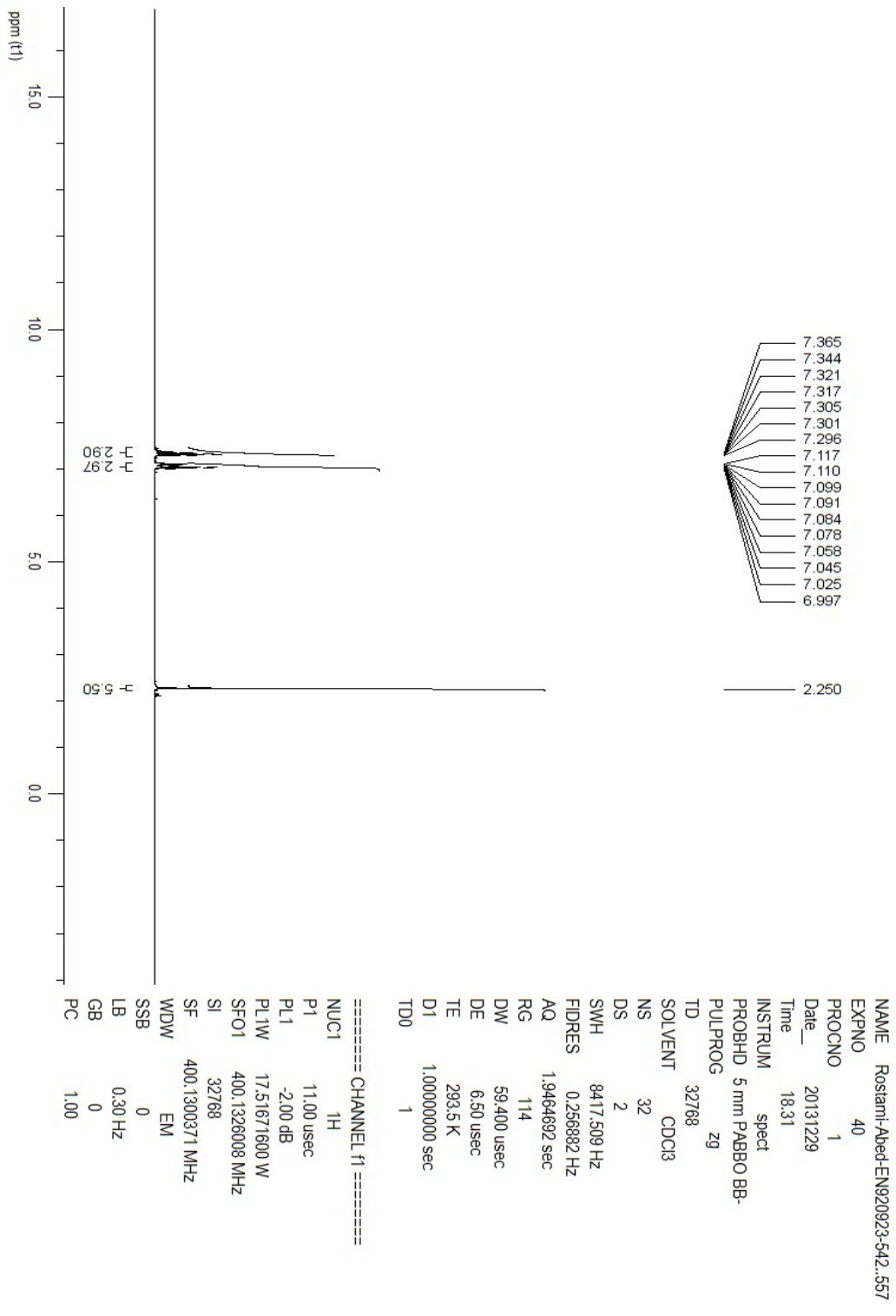
The ^{13}C-NMR spectrum (4-Methoxy phenyl) phenyl sulfide (Table 2, entry 3)



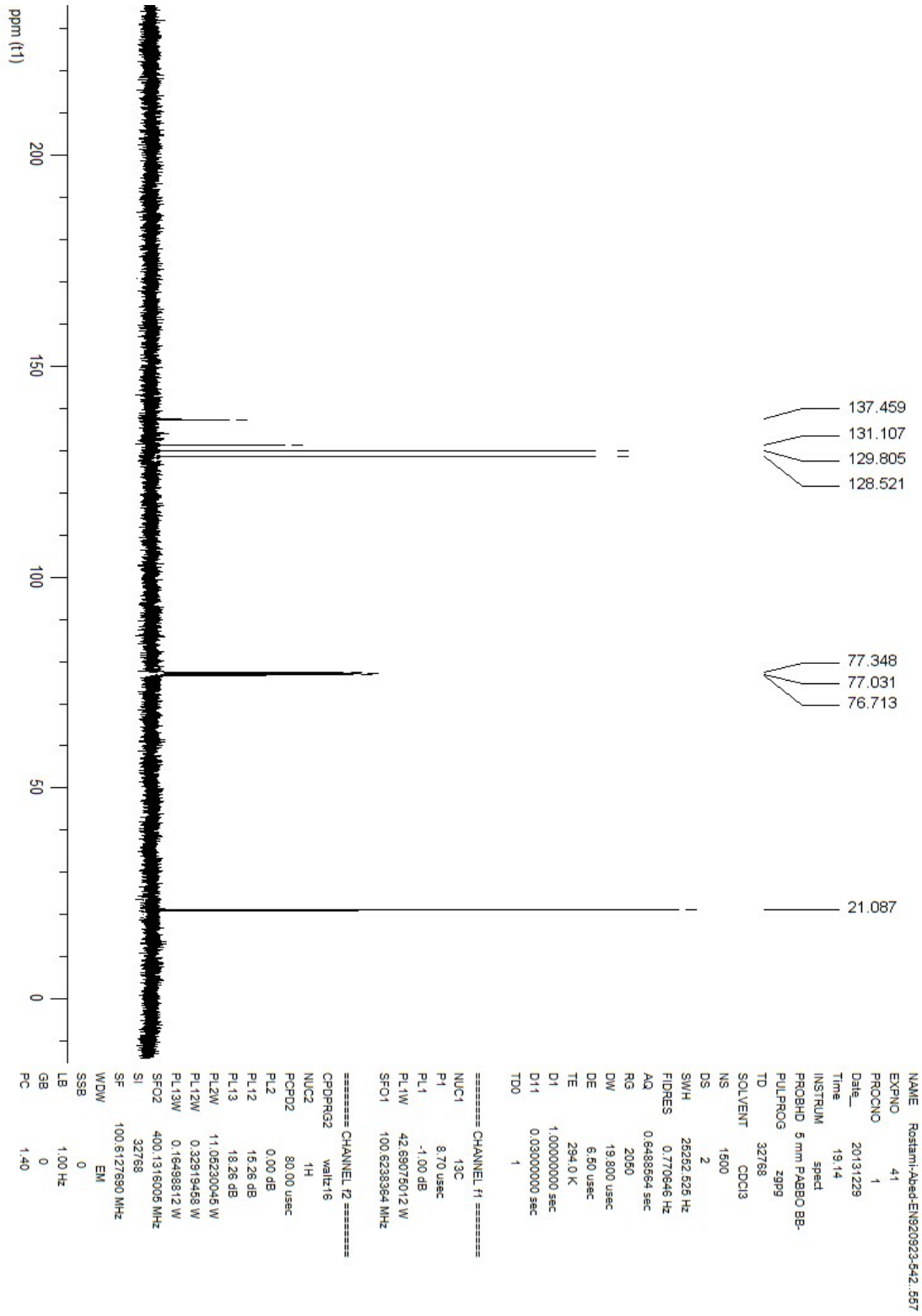
The ^{1}H-NMR spectrum of (4-Methyl phenyl) phenyl sulfide (Table 2, entry 4)



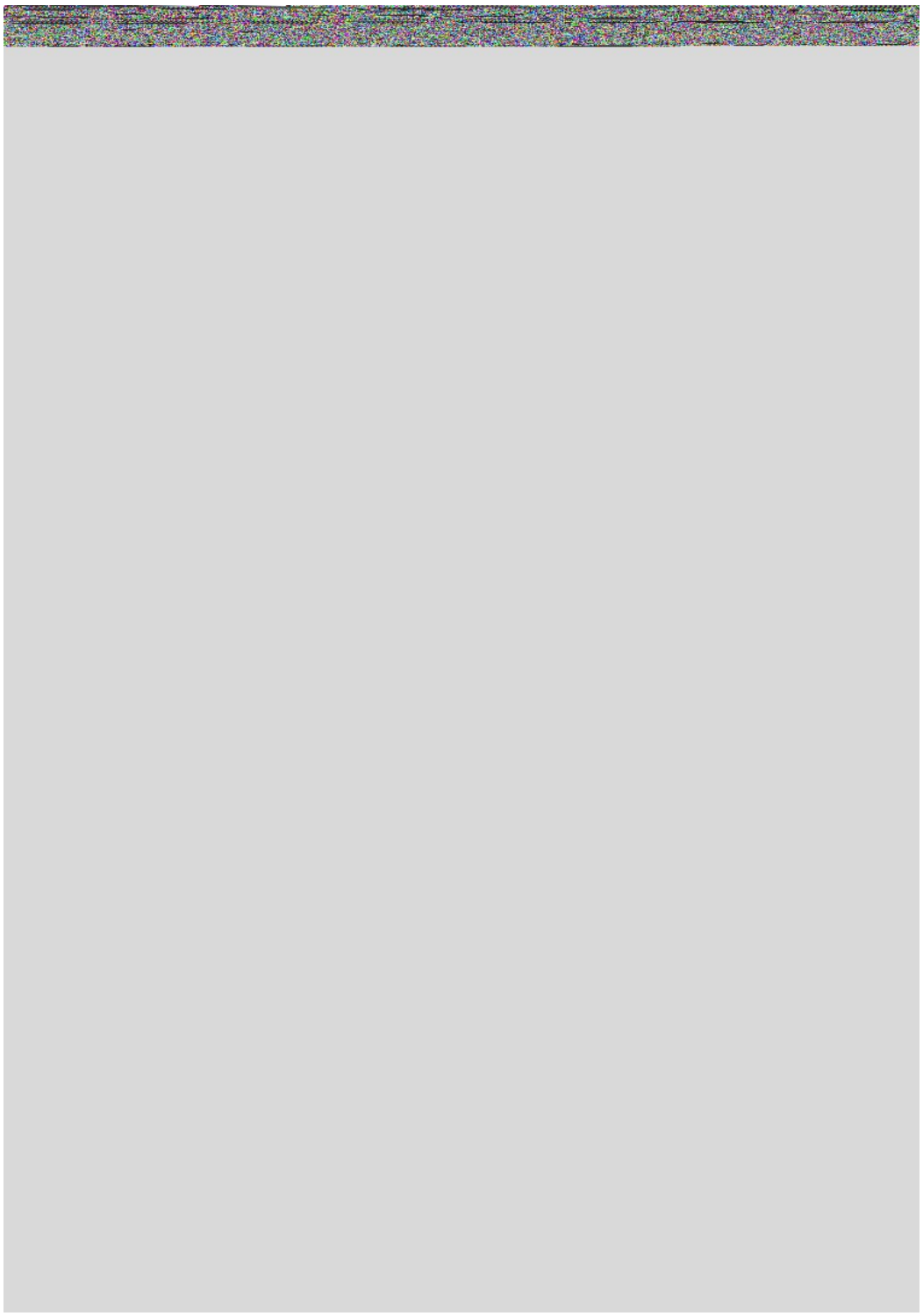
The ^{13}C-NMR spectrum of (4-Methyl phenyl) phenyl sulfide (Table 2, entry 4)



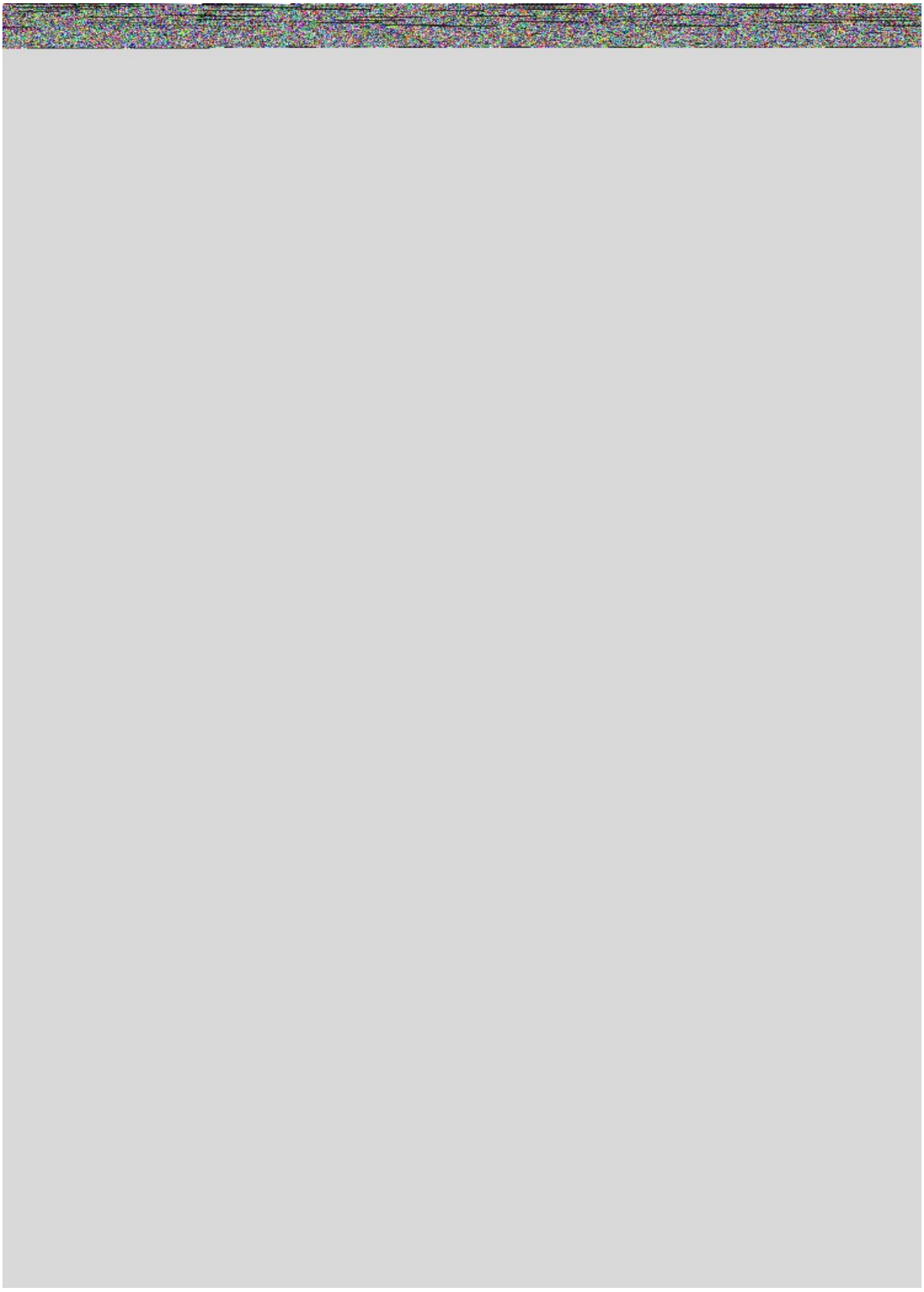
The ^{1}H-NMR spectrum of Di-*p*-tolyl sulfide (Table 6, entry 3)



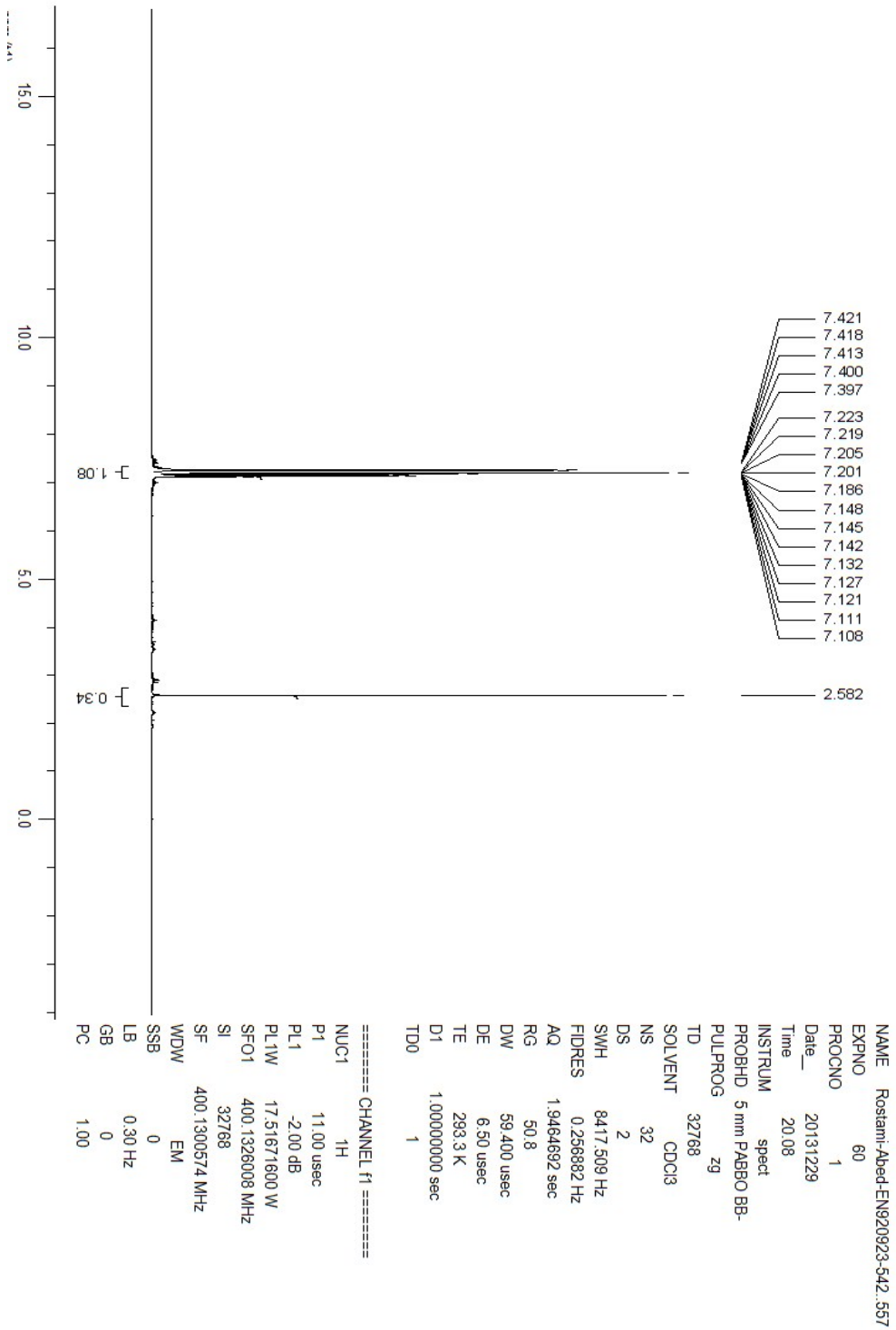
The ^{13}C-NMR spectrum of Di-*p*-tolyl sulfide (Table 6, entry 3)



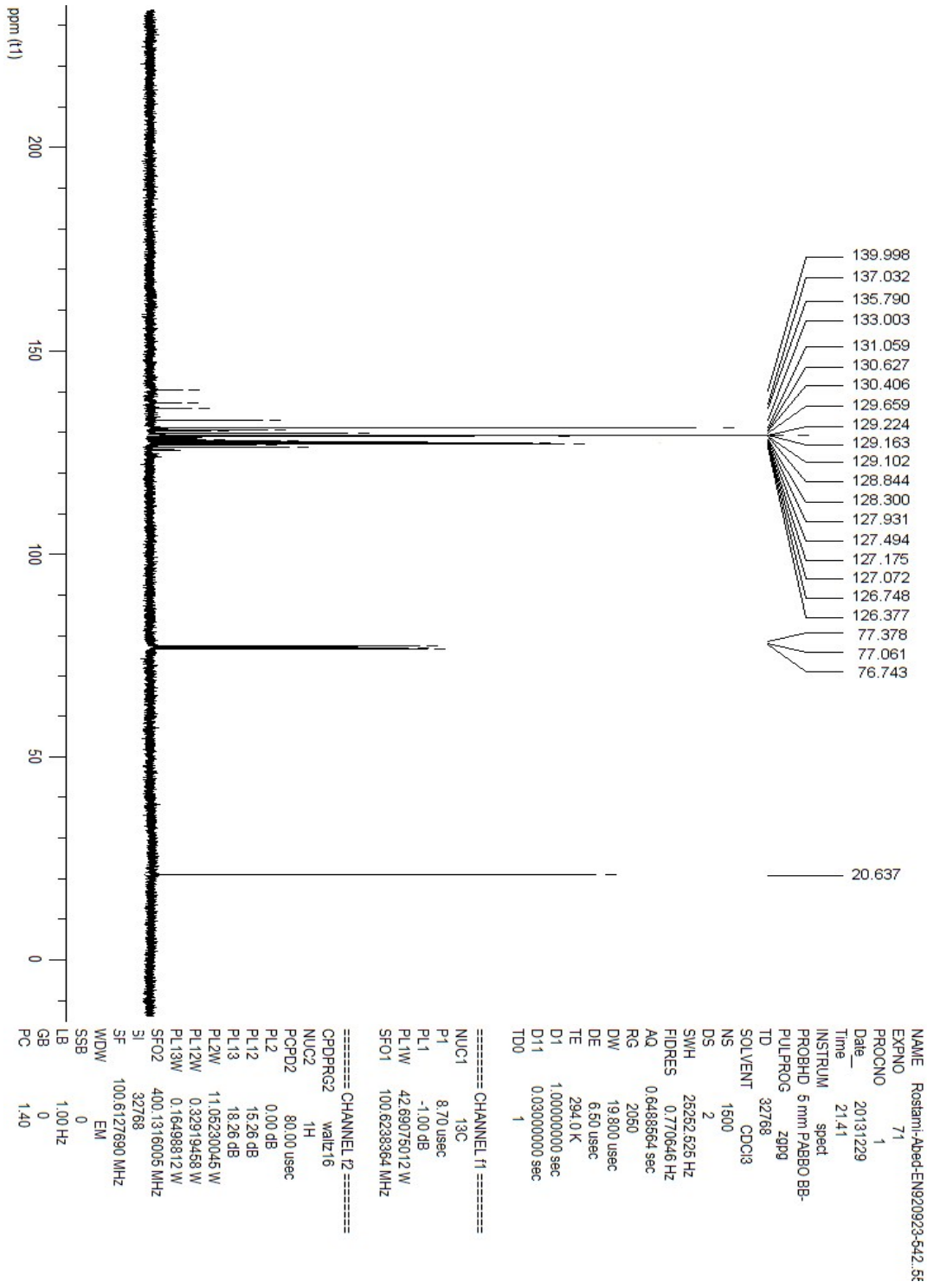
The ^{1}H-NMR spectrum of Di-*p*-tolyl sulfide (Table 6, entry 6)



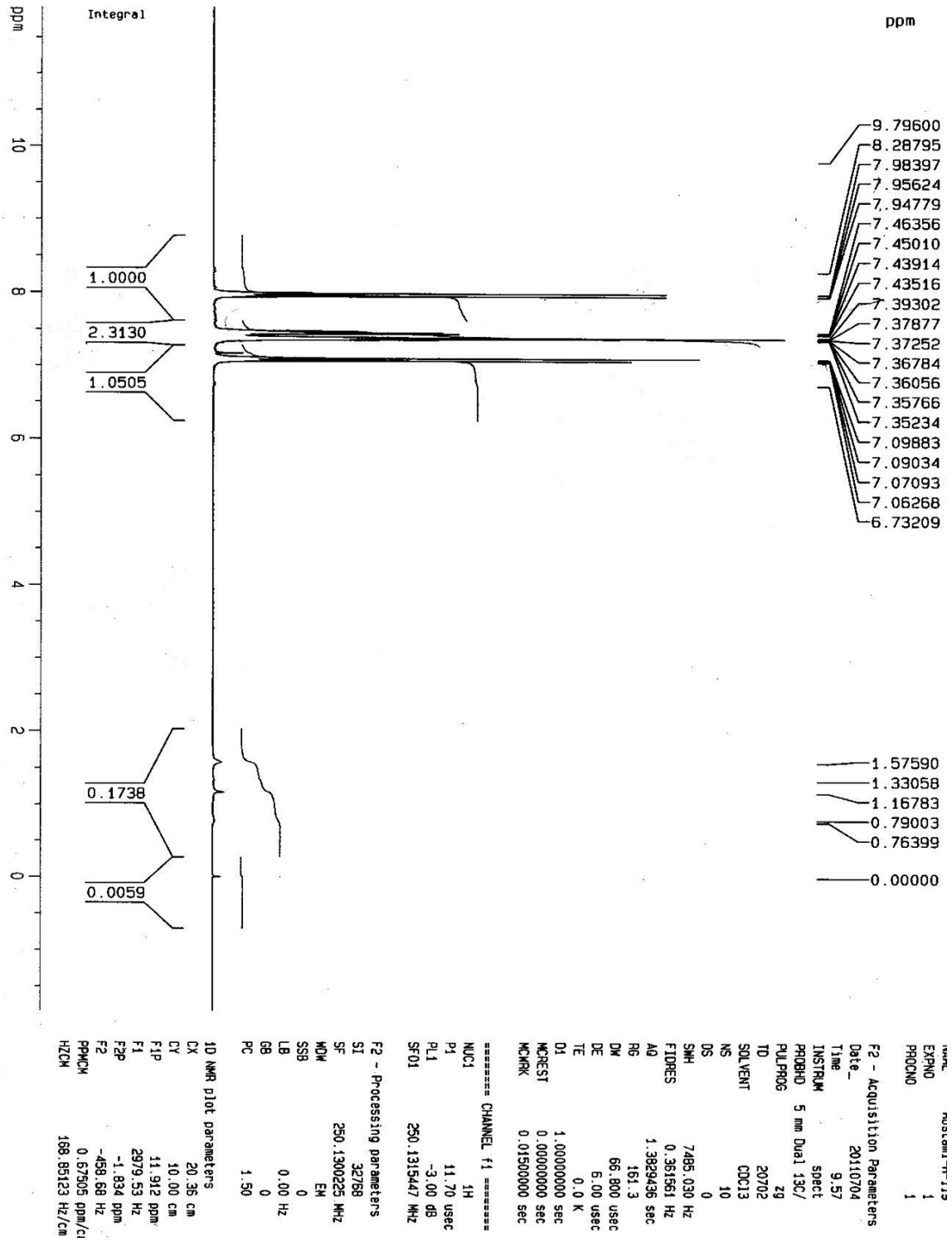
The ^{13}C-NMR spectrum of Di-*p*-tolyl sulfide (Table 2, entry 6)



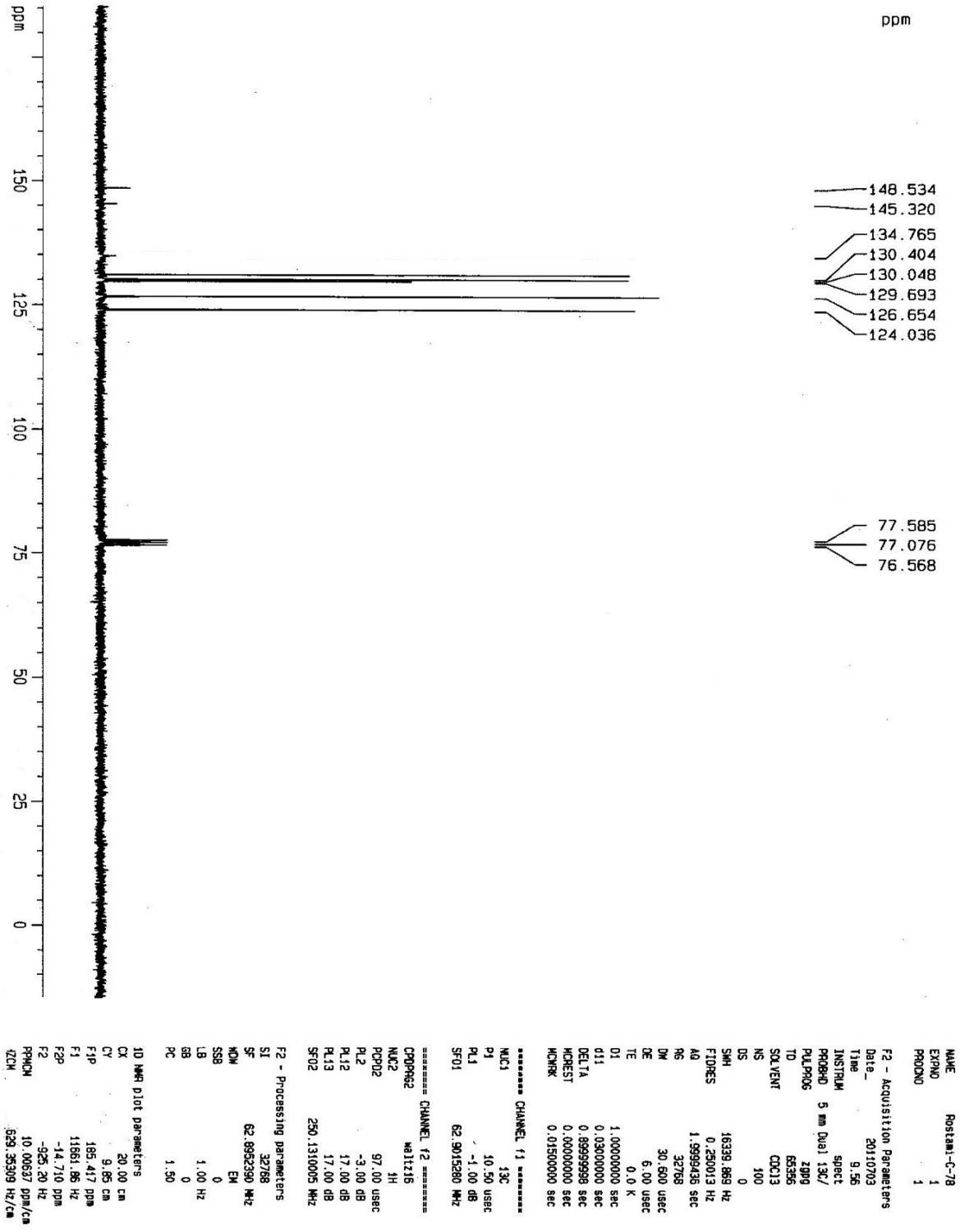
The ^{1}H-NMR spectrum of (2-methyl phenyl) phenyl sulfide (Table 2, entry 7)



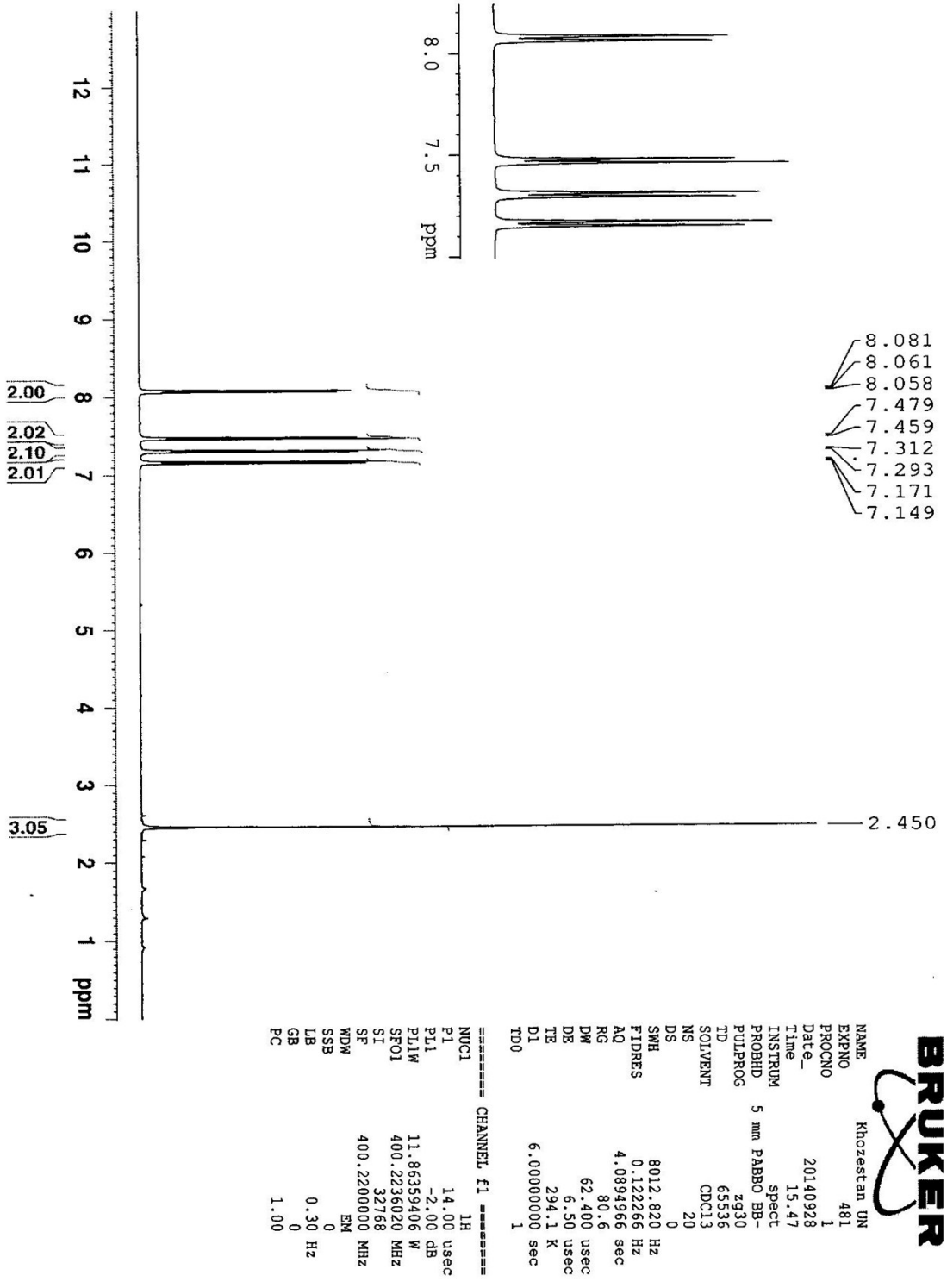
The ^{13}C-NMR spectrum of (2-methyl phenyl) phenyl sulfide (Table 2, entry 7)



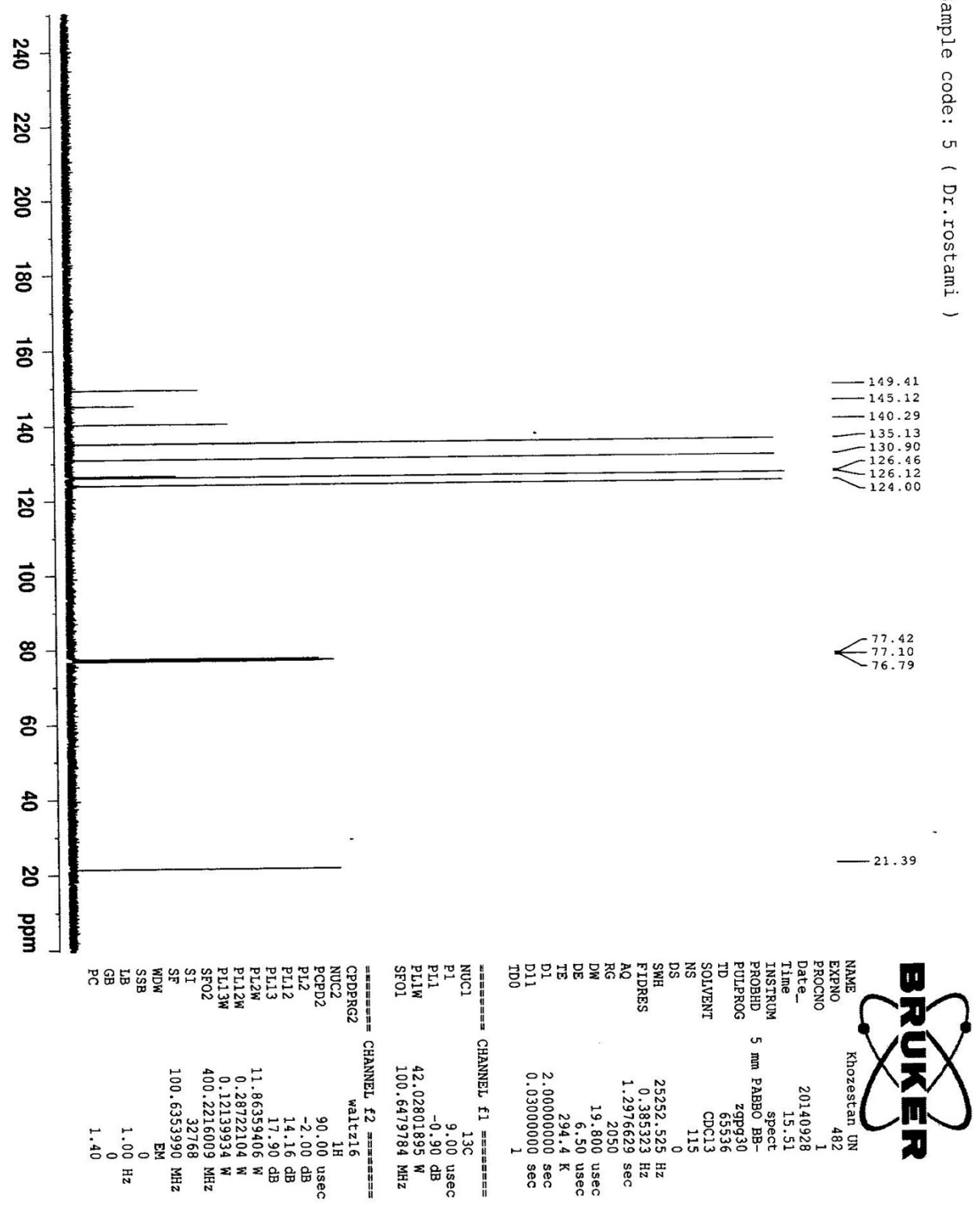
The ^{1}H-NMR spectrum of (4-Nitro phenyl) phenyl sulfide (Table 2, entry 8)



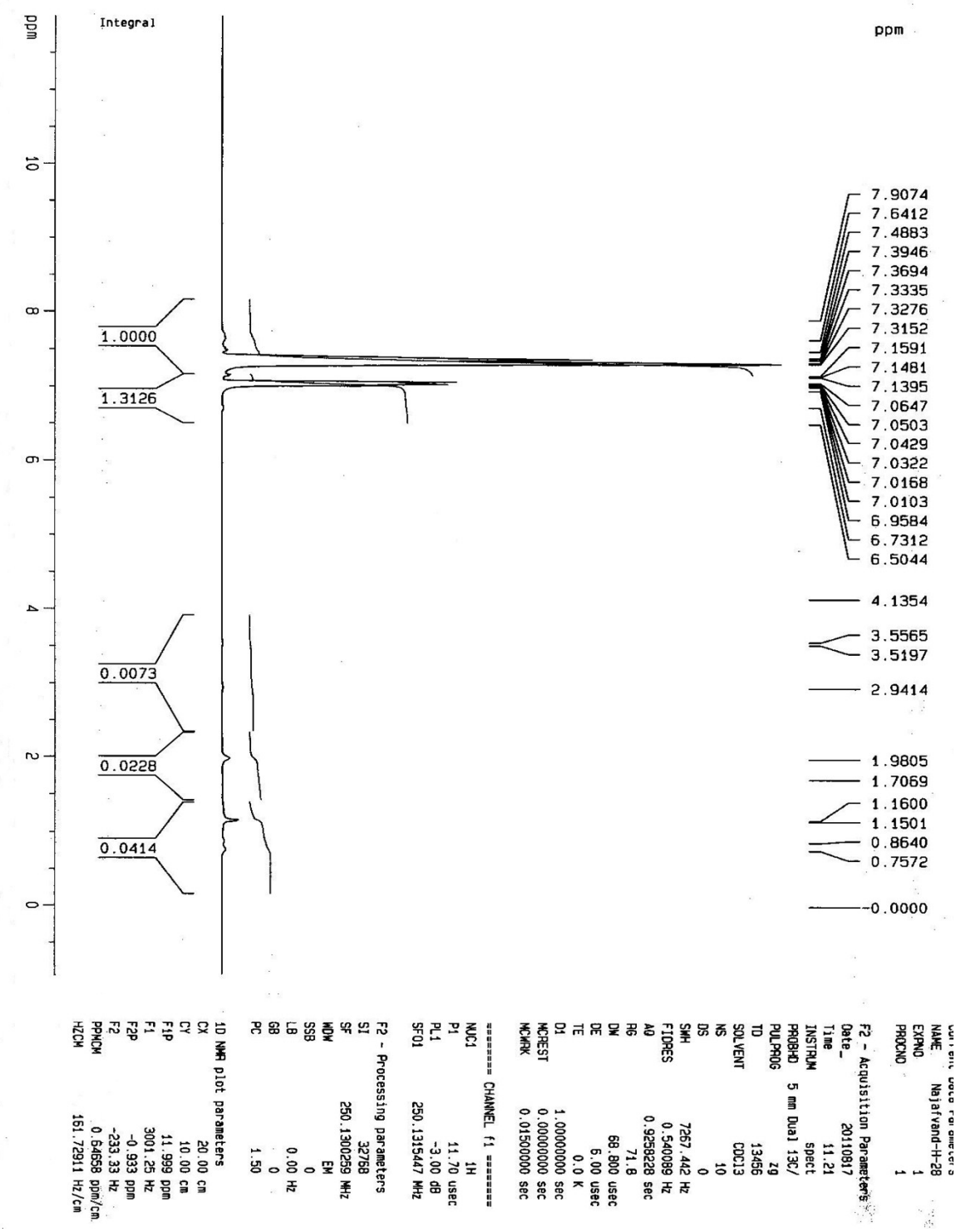
The ^{13}C-NMR spectrum of (4-Nitro phenyl) phenyl sulfide (Table 2, entry 8)



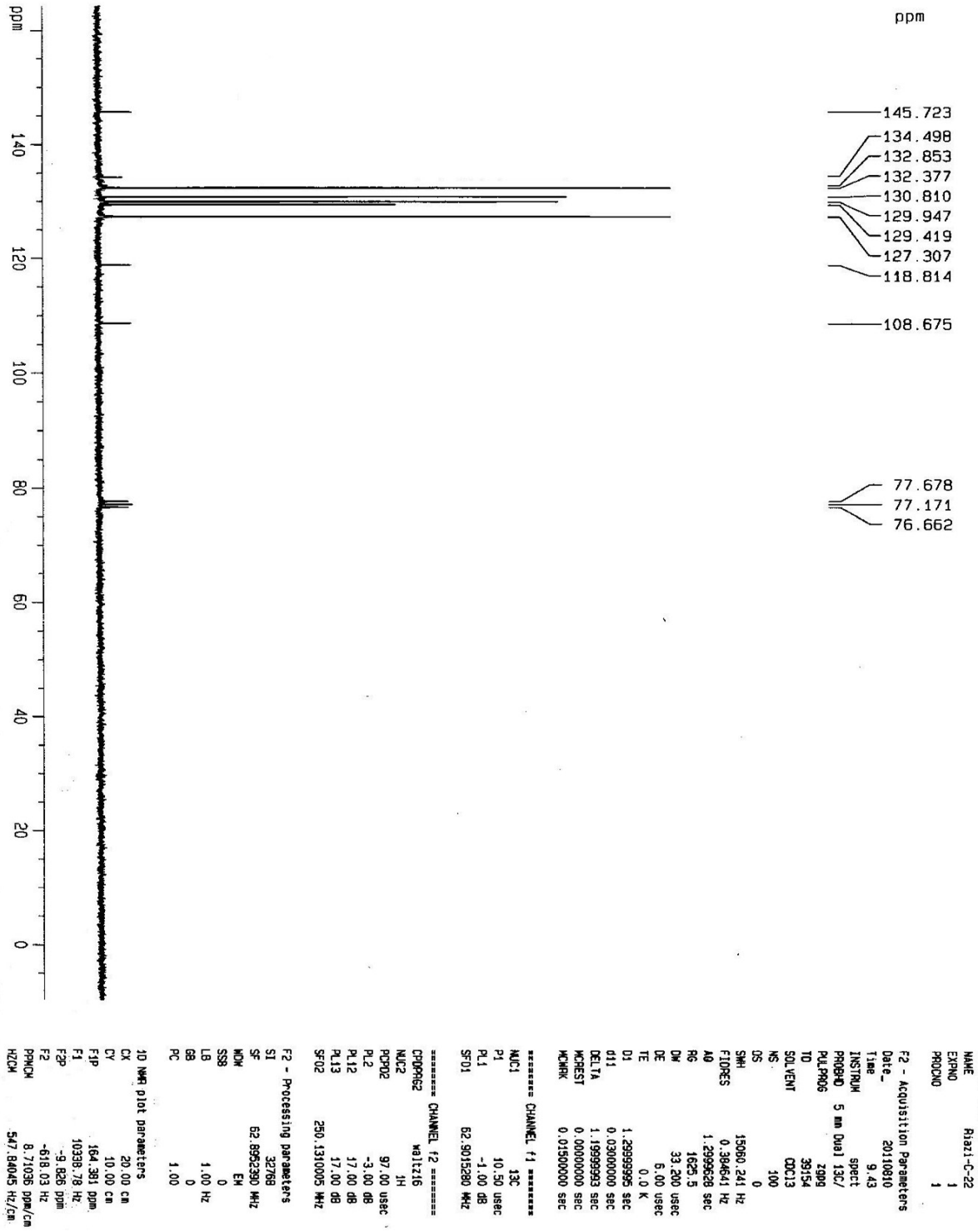
The ^{1}H-NMR spectrum of (4-Nitro phenyl *p*lylot- sulfide (Table 2, entry 9)



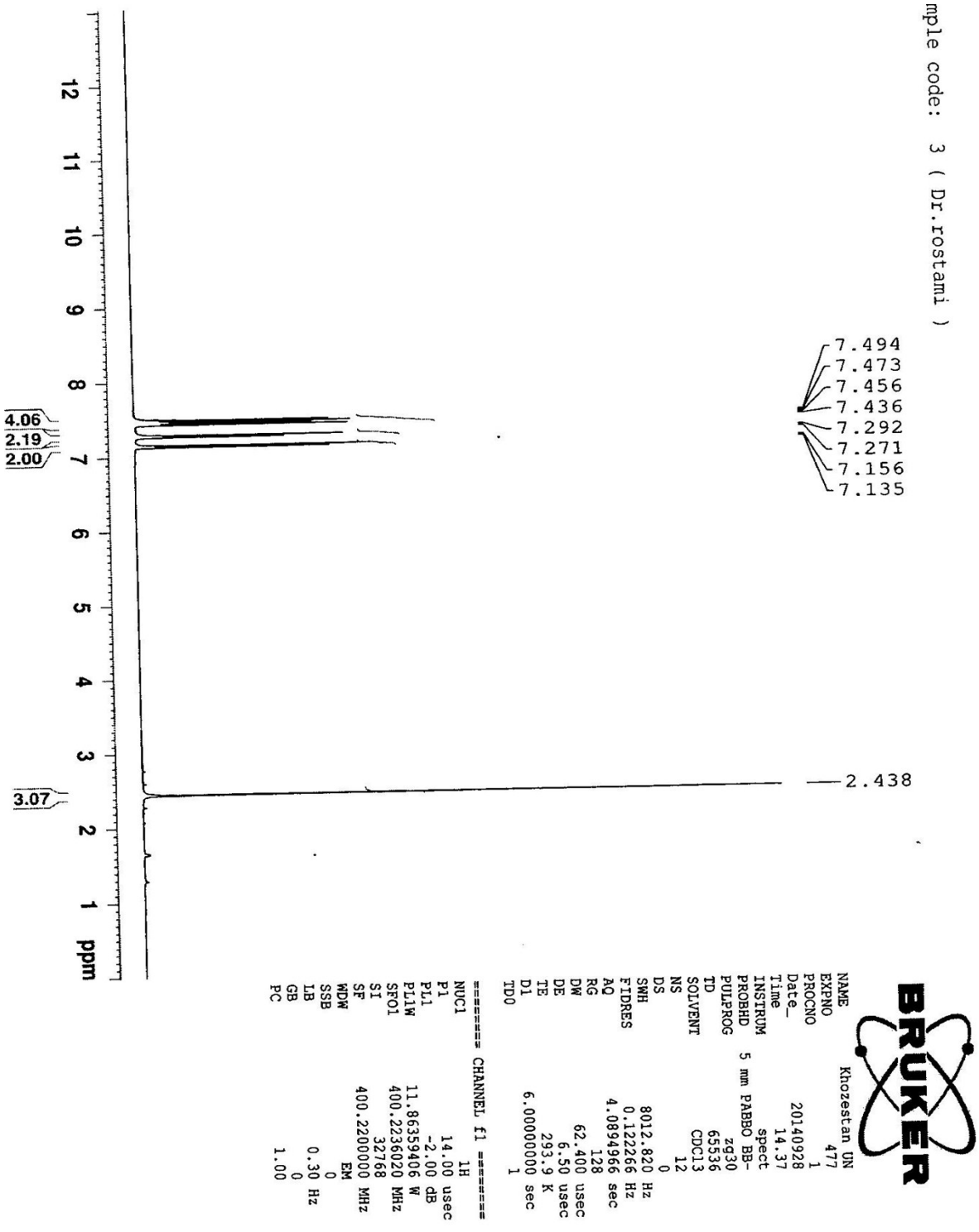
The ^{13}C-NMR spectrum of (4-Nitro phenyl) *p*tolyl- sulfide (Table 2, entry 9)



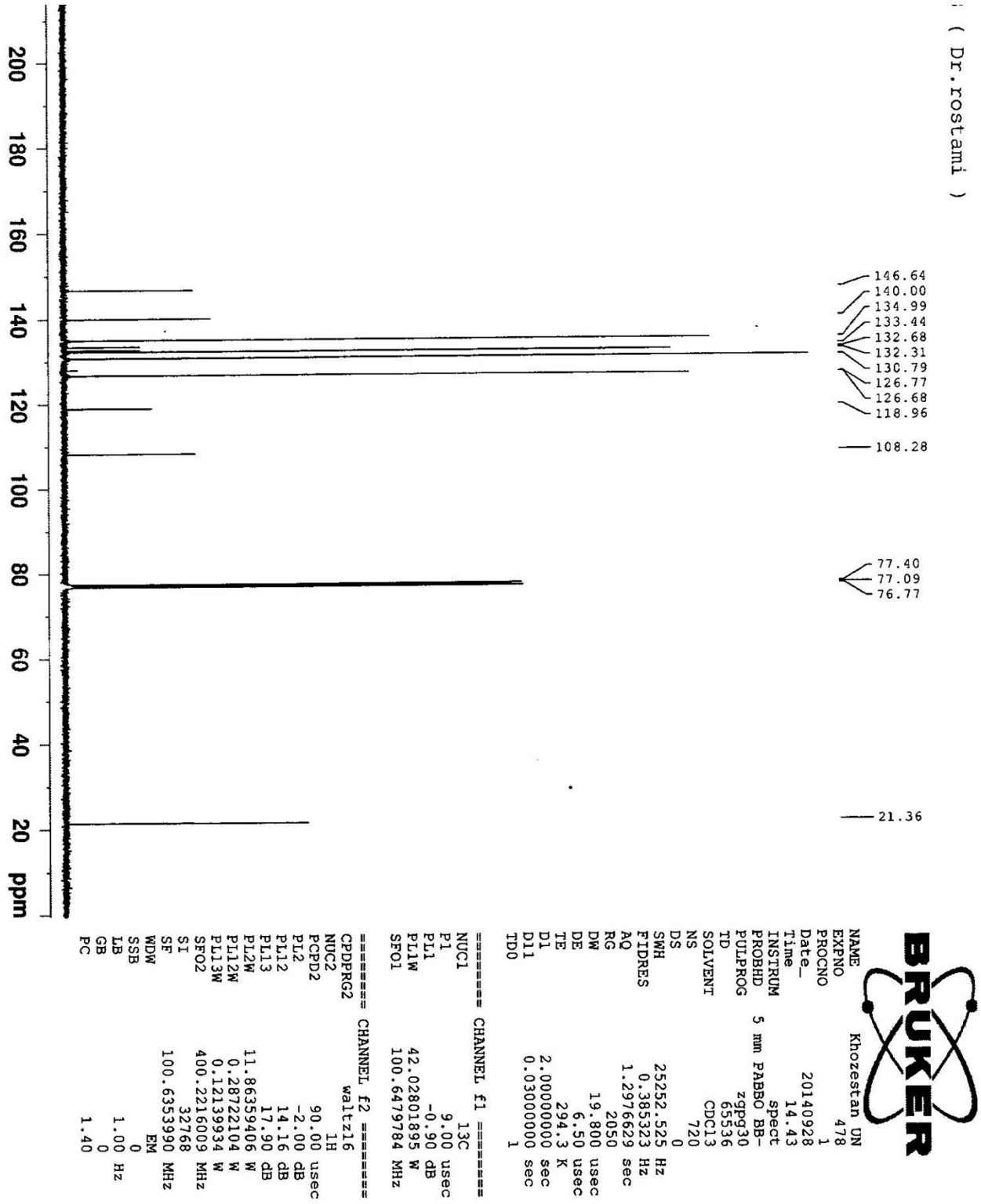
The ^{1}HNMR spectrum of (4-Nitryl phenyl) phenyl sulfide (Table 2, entry 10)



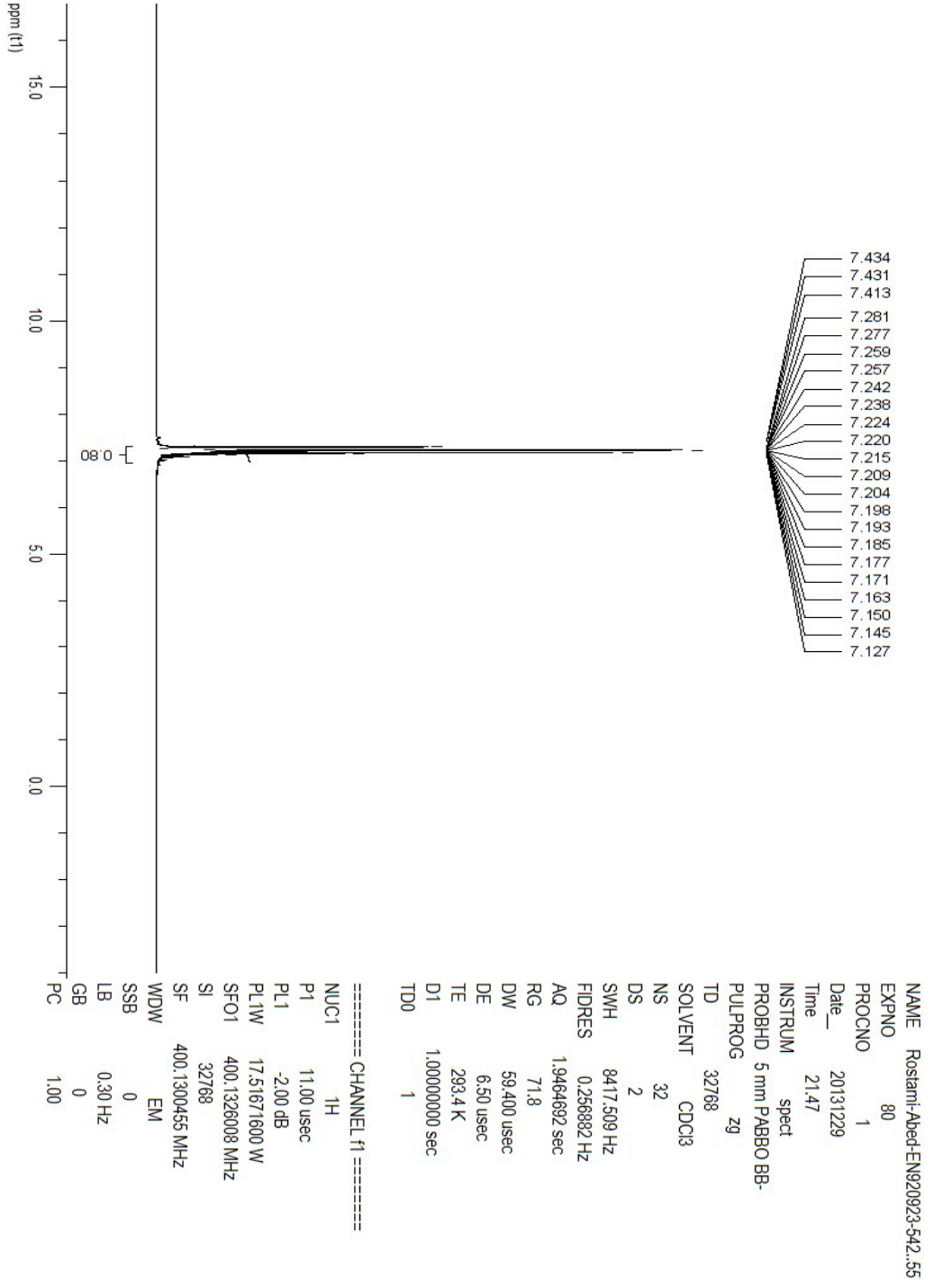
The ^{13}C-NMR spectrum of (4-nitryl phenyl) phenyl sulfide (Table 2, entry 10)



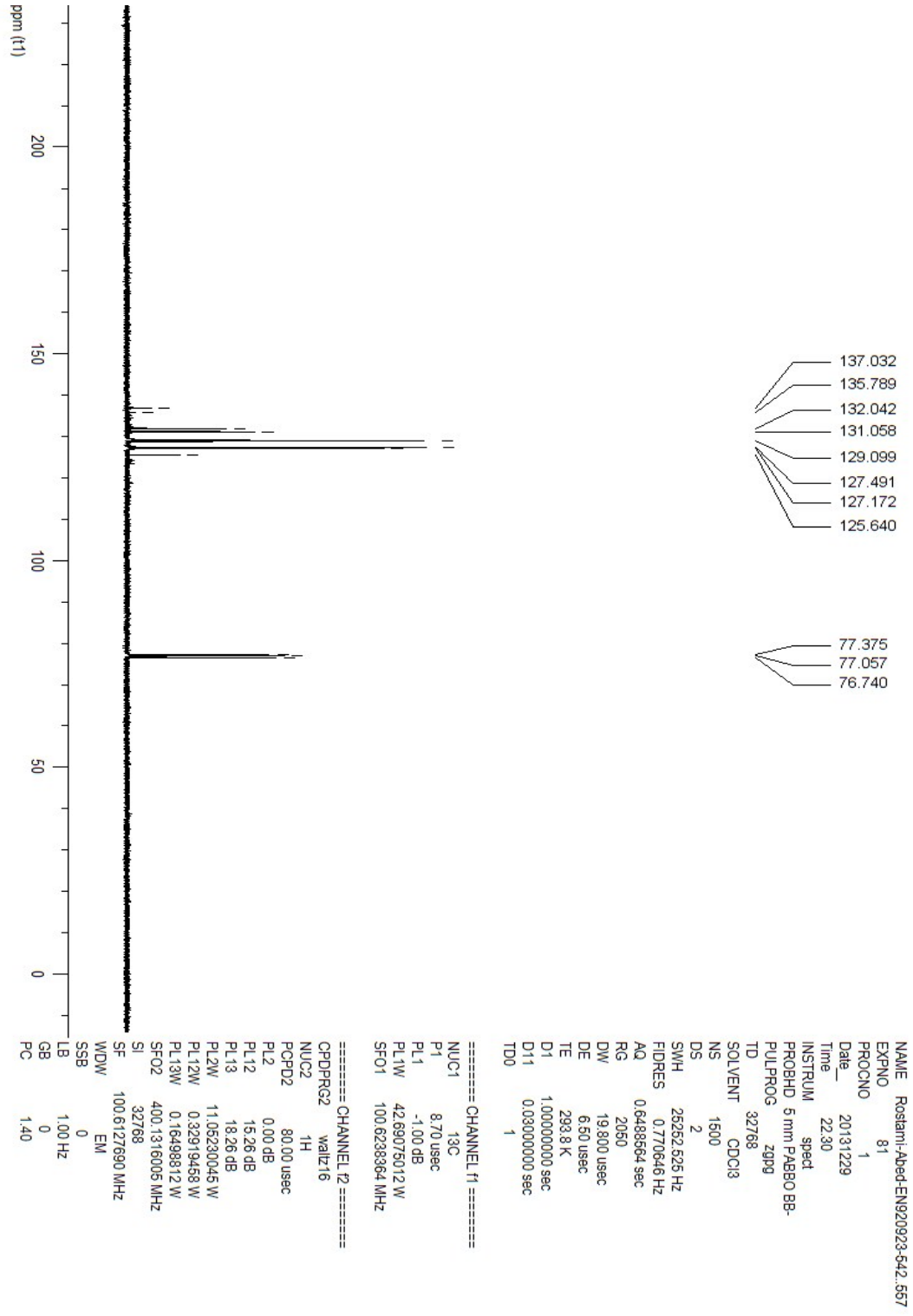
The ^{1}H-NMR spectrum of (4-Nitryl phenyl) *p*tolyl- sulfide (Table 2, entry 11)



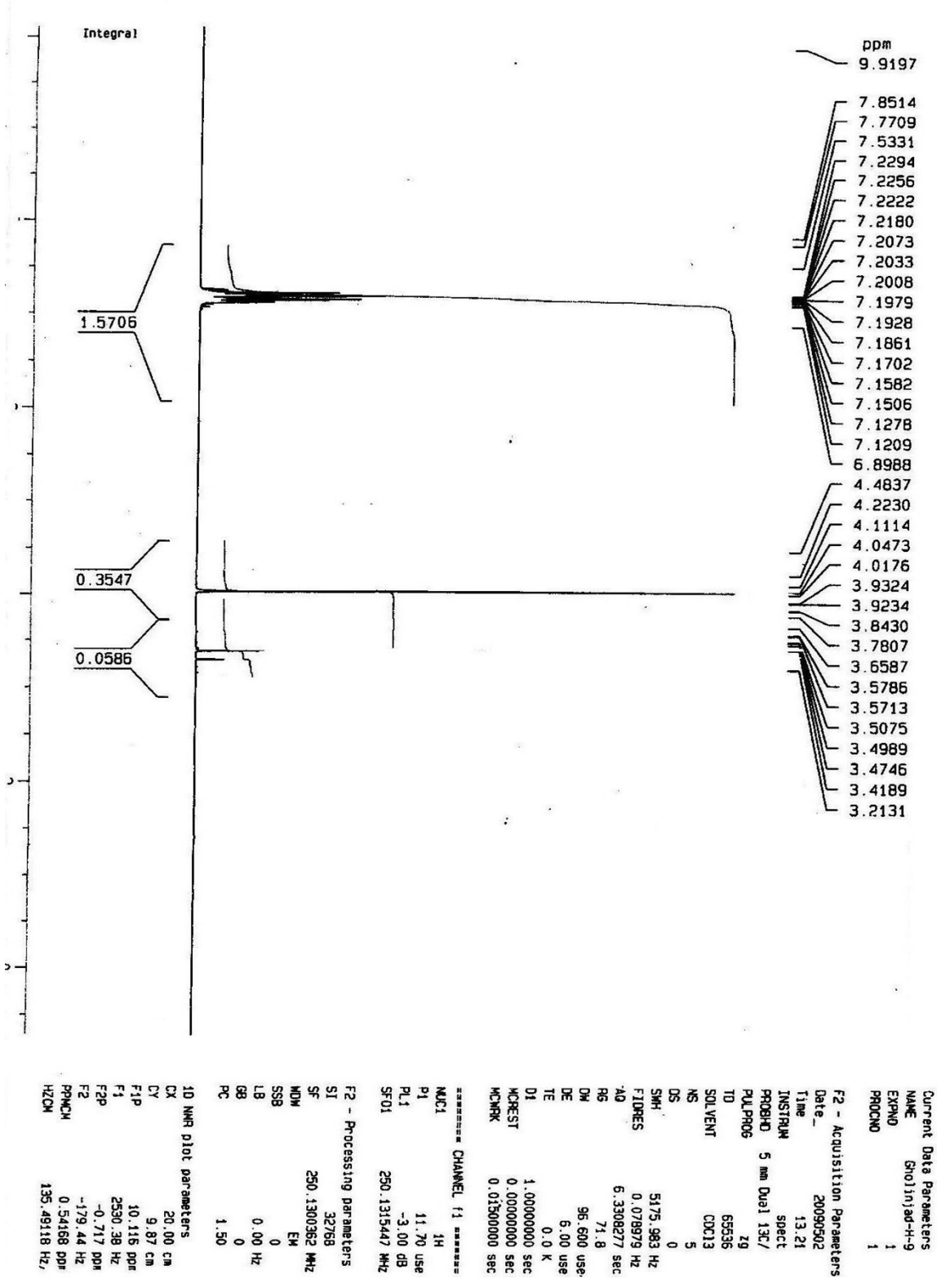
The ^{13}C-NMR spectrum of (4-Nitryl phenyl) *p*tolyl- sulfide (Table 2, entry 11)



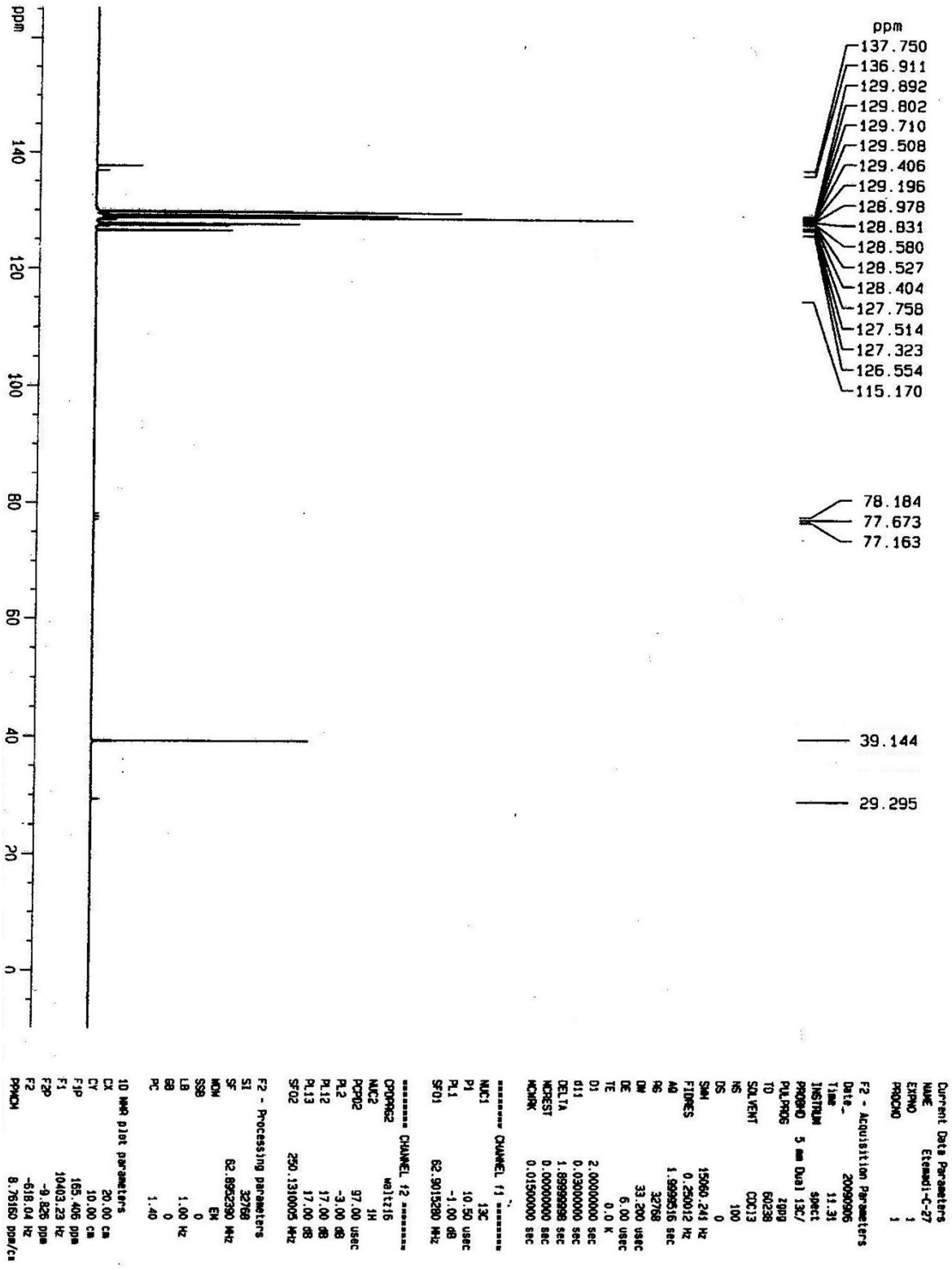
The ^{1}H-NMR spectrum of (4-Chloro phenyl) phenyl sulfide (Table 2, entry 20)



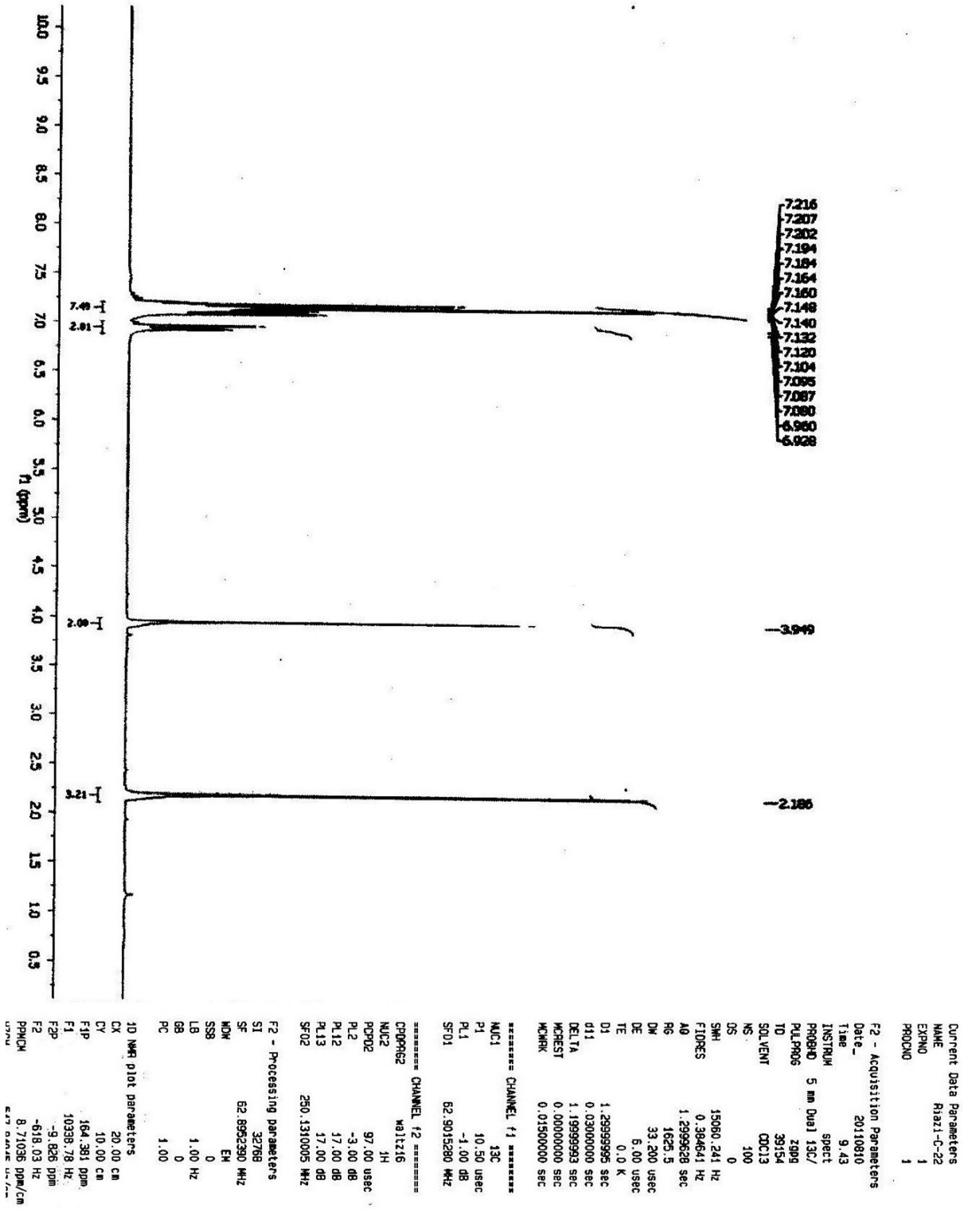
The ^{13}C-NMR spectrum of (4-Chloro phenyl) phenyl sulfide (Table 2, entry 20)



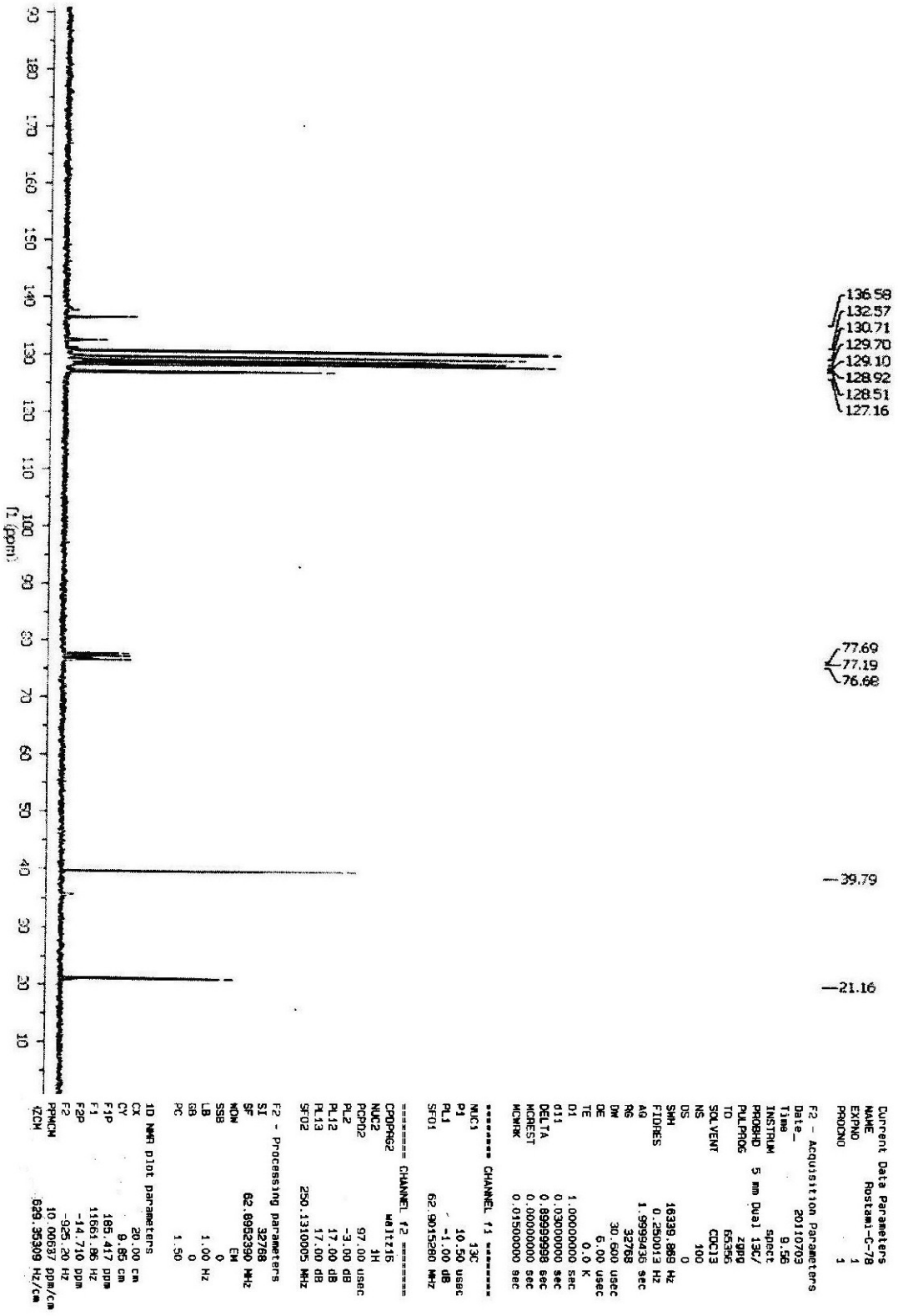
The ^{1}H-NMR spectrum of Benzyl (phenyl) sulfide (Table 2, entry 21)



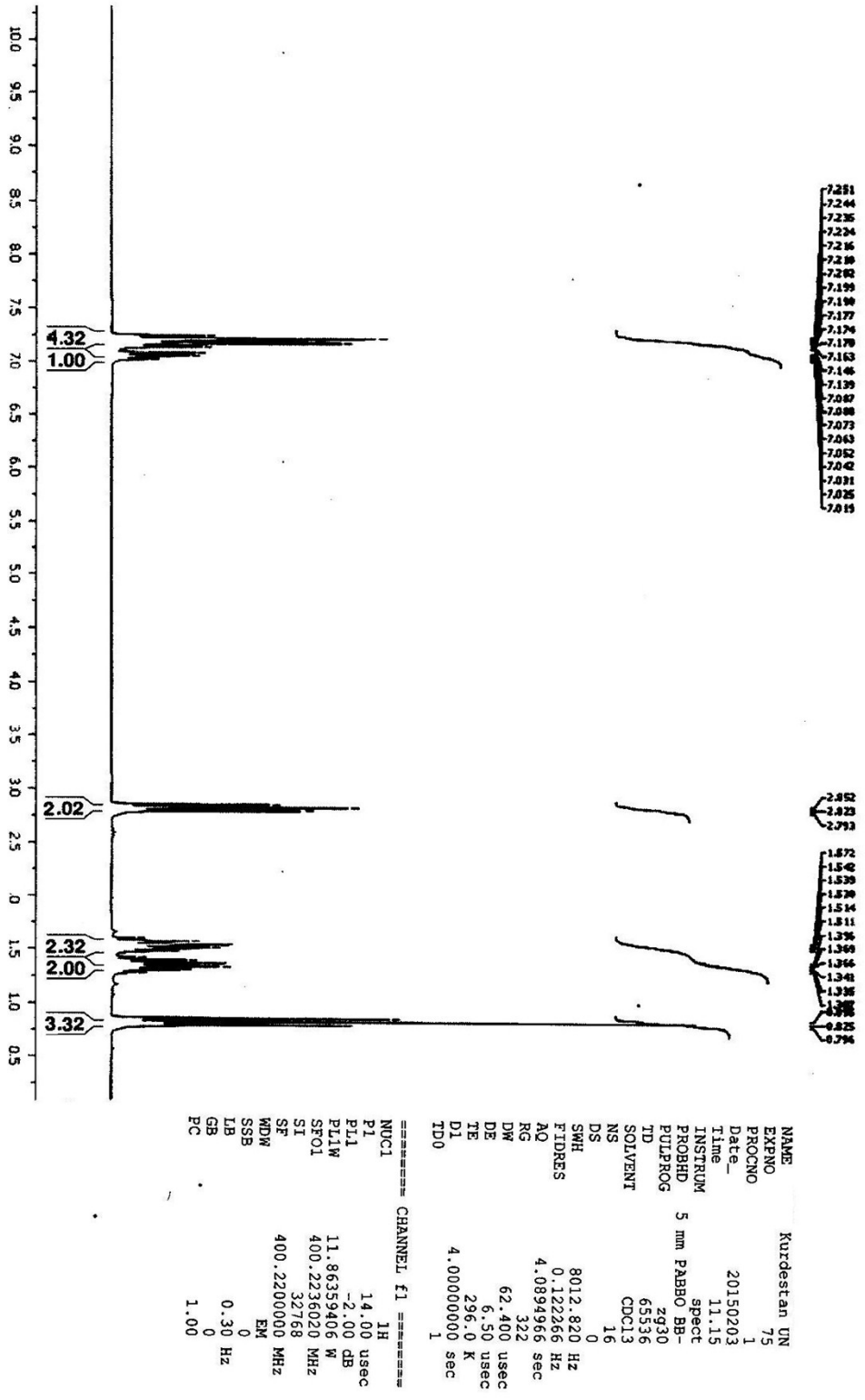
The ^{13}C-NMR spectrum of Benzyl (phenyl) sulfide (Table 2, entry 21)



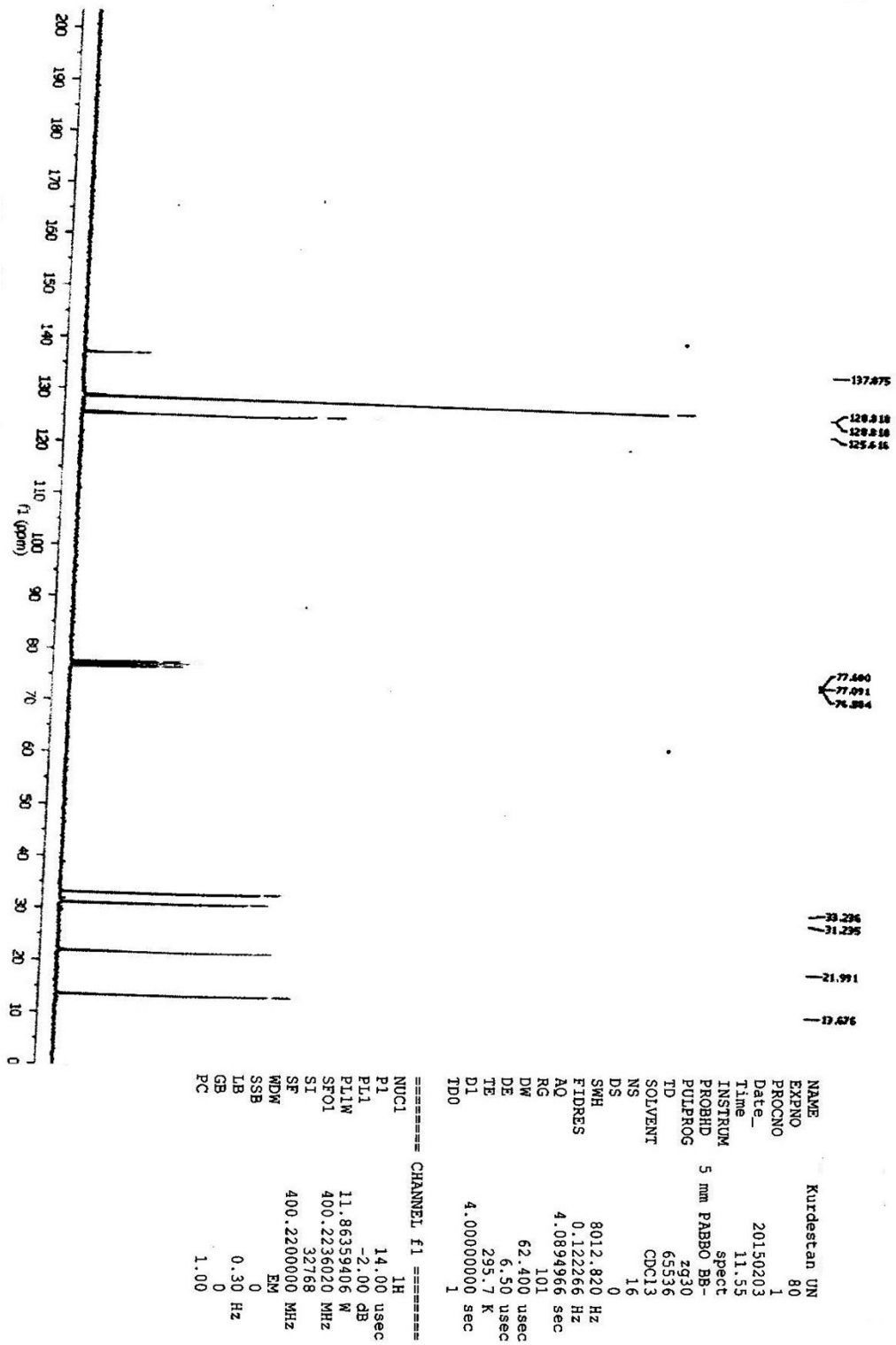
The ^{1}H-NMR spectrum of Benzyl (*p*-tolyl) sulfide (Table 2, entry 22)



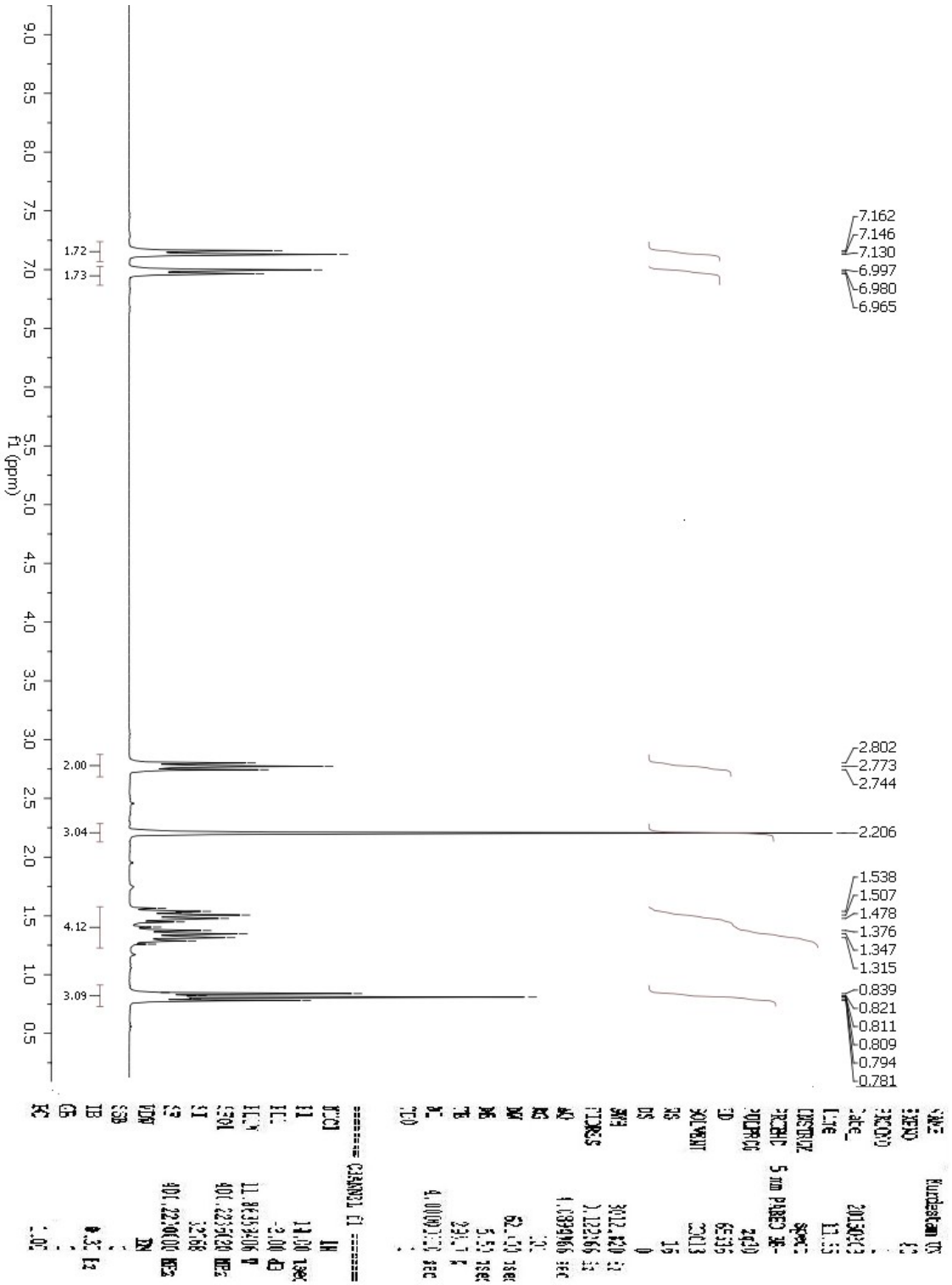
The ^{13}C-NMR spectrum of Benzyl (*p*-tolyl) sulfide (Table 2, entry 22)



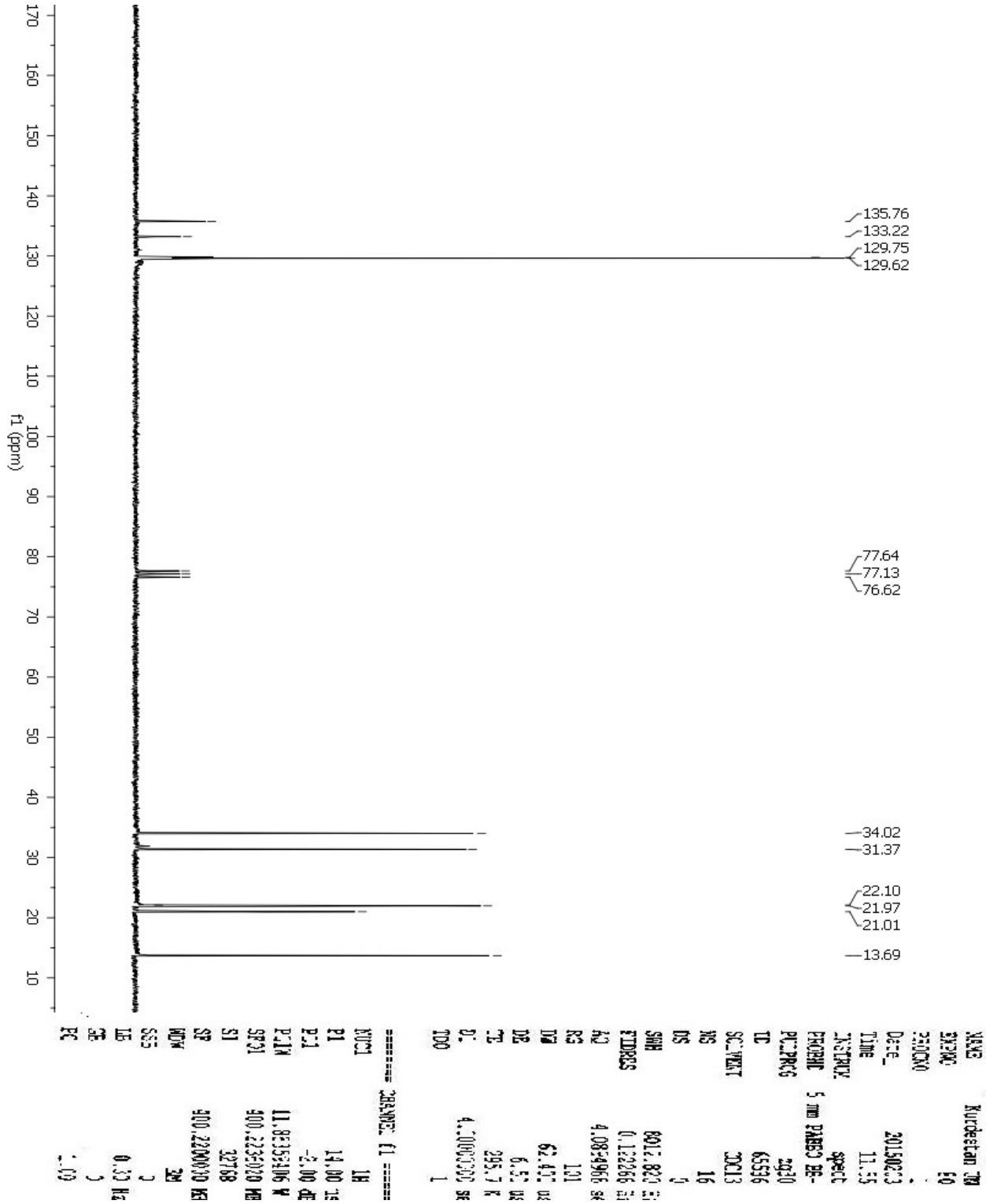
The ^{1}H-NMR spectrum of Butyl phenyl sulfide (Table 2, entry 23)



The ^{13}C-NMR spectrum of Butyl phenyl sulfide (Table 2, entry 23)



The ^{1}H-NMR spectrum of Butyl (4-methyl phenyl) sulfide (Table 2, entry 24)



The ^{13}C-NMR spectrum of Butyl (4-methyl phenyl) sulfide (Table 2, entry 24)