Palladium-Catalyzed Amidation-Hydrolysis Reaction of *gem*-Dihaloolefins: Efficient Synthesis of Homologated Carboxamides from Ketones

Wenchao Ye, Jun Mo, Tiankun Zhao and Bin Xu*

Department of Chemistry, Shanghai University, Shanghai 200444, China

Tel.: 86-21-66132065; Fax: 86-21-66132797.
Email: xubin@shu.edu.cn

Supporting Information

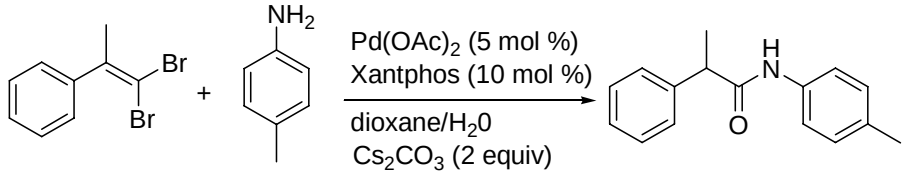
Table of Contents

(A) General Methods and Materials

All reagents, metal catalysts and solvents were obtained from commercial sources and were purified before use.[1] Chromatographic purifications were performed on silica gel (mesh 230-400) by the flash technique. The *gem*-dihaloolefins can be prepared from appropriate ketones according to the methods from literatures.[2-9] All melting points were taken on a Digital Melting Point Apparatus without correction. Infrared spectra were obtained using a FT-IR spectrometer. ^{1}H and ^{13}C NMR spectra were recorded at 500 and 125 MHz, respectively, with chemical shift values being reported in ppm relative to chloroform (δ = 7.26 ppm) or TMS (δ = 0.00 ppm) for ^{1}H NMR, and chloroform (δ = 77.16 ppm) for ^{13}C NMR. Mass spectra and high resolution mass spectra were recorded using an ES ion source unless stated otherwise. Elemental analyses were carried out on an elemental analyzer. Silica gel plate GF254 were used for thin layer chromatography (TLC) and silica gel H or 300-400 mesh were used for flash column chromatography. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise indicated. All the starting materials and products were satisfactorily characterized by ^{1}H and ^{13}C NMR, MS, and HRMS, element analysis and when possible, comparison of their ^{1}H NMR spectra has been made with available literature data and/or those of authentic samples**.** The following compounds 1-(1,1-dibromoprop-1-en-2-yl)benzene (**1a**) [60014-86-0],[5] 1-(1,1-dibromoprop-1-en-2-yl)-4-methylbenzene (**1b**) [452335-62-5],[6] 1-(1,1- dibromo-prop-1-en-2-yl)-4-methoxybenzene (**1c**) [95111-00-5],[6] 1-(1,1-dibromoprop-1-en-2-yl) -4-nitrobenzene (**1d**) [452335-63-6],[3] 1-(1,1-dibromoprop-1-en-2-yl)-4-chlorobenzene (**1e**) [127042-57-3],[6] (dibromomethylene)cyclopentane (**1h**) [103670-61-7],[7] (dibromo-methylene)cyclohexane (**1i**) [60014-85-9],[7] (2,2-dibromoethene- 1,1-diyl) dibenzene (**1j**) [2592-73-6],[8] 1-((*E*)-4,4-dibromo-3-methylbuta-1,3-dienyl) benzene (**1l**) [58898-15-0],[9]

1-(1,1-dichloroprop-1-en-2-yl)benzene (**1m**) [5264-26-6],[10] have all been reported previously.

(B) Screening for effect of amount of water



1a **2a** **3aa**

entry	dioxane: water	isolated yield (%)
1	3 : 1	76
2	5 : 1	85
.3	**7 : 1**	**91**
4	10 : 1	83
5	50 : 1	35

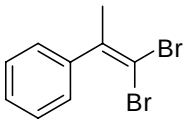
From Table 1, the best result was isolated in 91% yield when the solvent was dioxane:water = 7:1 (Entry 3). The yield decreased with the solvent ratio changed to 3:1 and 50:1 (Entries 1, 5).

(C) General procedure for the amidation-hydrolysis reaction

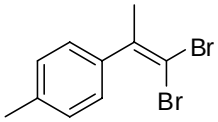
All reactions were carried out with standard Schlenk techniques under an nitrogen atmosphere. **2-phenyl-N-p-tolylpropanamide (3aa)**[14]: To an oven-dried, N_2-purged flask containing **2a** (107.2 mg, 1.0 mmol), $Pd(OAc)_2$ (5.6 mg, 0.025 mmol), Xantphos (28.9 mg, 0.05 mmol), and Cesium carbonate (325.8 mg, 1.0 mmol), was added a solution of **1a**

(138.0 mg, 0.5 mmol) in dioxane/H_2O (1.2 mL, dioxane/H_2O = 7:1). The reaction mixture was stirred under reflux for 3 h and monitored by TLC. Upon completion, the reaction was diluted by EtOAc (5ml) and quenched with aq. HCl (0.1 M, 10 mL). The aqueous layer was extracted with EtOAc (3 × 10 mL). The combined extract was washed with saturated $NaHCO_3$ aq. (10 mL) and dried over Na_2SO_4. After the solvent was removed under reduced pressure, the residue was quickly purified by column chromatography (Petroleum Ether/EtOAc = 8:1) to give **3aa** as a white solid (108.9 mg, 91%). M.p. 116-117 ^oC; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.42-7.34 (m, 4H), 7.34-7.27 (m, 1H), 7.30 (AA'of AA'BB', *J* = 8.5 Hz, 2H), 7.07 (BB'of AA'BB', *J* = 8.5 Hz, 2H), 7.12-7.05 (m, 1H), 3.70 (q, *J* = 7 Hz , 1H), 2.28 (s, 3H), 1.59 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.32, 141.15, 135.40, 133.99, 129.50, 129.25, 127.84, 127.65, 119.90, 48.16, 20.95, 18.71; IR (KBr, cm^{-1}): 3285, 3248, 1652, 1603, 1537, 818, 696; EI-MS *m/z* (%): 239 (73) [M^+], 133 (27), 107 (100), 106 (60), 105 (72).

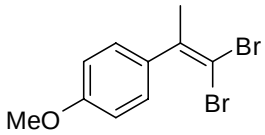
(D) Compounds characterized



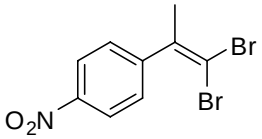
1-(1,1-dibromoprop-1-en-2-yl)benzene (1a)[5]: colorless oil. ^1H NMR ($CDCl_3$, 500 MHz): δ 7.40-7.34 (m, 2H), 7.34-7.29 (m, 1H), 7.25-7.21 (m, 2H), 2.21 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.22, 142.14, 128.53, 127.83, 127.53, 87.77, 26.30; IR (KBr, cm^{-1}): 3056, 3024, 2913, 822, 698; EI-MS *m/z* (%): 277 (48) [M^+H (^{81}Br, ^{79}Br)], 275 (88) [M^+H ($2\times^{79}Br$)], 116 (78), 115 (100).



1-(1,1-dibromoprop-1-en-2-yl)-4-methylbenzene (1b)[6]: colorless oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.18 (AA' of AA'BB', *J* = 8 Hz, 2H), 7.13 (BB' of AA'BB', *J* = 8 Hz, 2H), 2.36 (s, 3H), 2.20 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.17, 139.19, 137.66, 129.20, 127.46, 87.42, 26.31, 21.41; IR (KBr, cm^{-1}): 3023, 2918, 828, 805. EI-MS m/z (%): 292 (8) [M^+ (2×^{81}Br)], 290 (96) [M^+ (^{81}Br, ^{79}Br)], 289 (100) [M^+H (2×^{79}Br)], 130 (91), 129 (60), 115 (43).

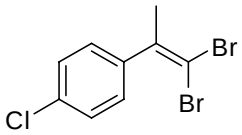


1-(1,1-dibromoprop-1-en-2-yl)-4-methoxybenzene (1c)[3]: purple solid. M.p. 43-44 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.21 (AA' of AA'BB', *J* = 8.5 Hz, 2H), 6.92 (BB' of AA'BB', *J* = 8.5 Hz, 2H), 3.85 (s, 3H), 2.23 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 159.01, 142.70, 134.20, 128.82, 113.73, 87.27, 55.27, 26.20; IR (KBr, cm^{-1}): 3425, 2954, 2909, 1508, 1241, 836. EI-MS *m/z* (%): 308 (36) [M^+ (2×^{81}Br)], 306 (72) [M^+ (^{81}Br, ^{79}Br)], 304 (37) [M^+ (2×^{79}Br)], 146 (100), 131 (26).

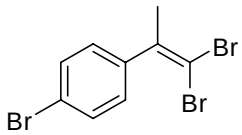


1-(1,1-dibromoprop-1-en-2-yl)-4-nitrobenzene (1d)[3]: yellow solid. M.p. 83-84 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.24 (AA' of AA'BB', *J* = 8.5 Hz, 2H), 7.42 (BB' of AA'BB', *J*

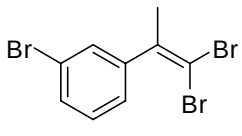
= 8.5 Hz, 2H), 2.22 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 148.65, 141.26, 128.77, 123.96, 89.78, 26.00; IR (KBr, cm^{-1}): 3436, 2913, 2845, 1514, 1343, 857. EI-MS *m/z* (%): 323 (33) [M^+ ($2\times^{81}$Br)], 321 (70) [M^+ (^{81}Br, ^{79}Br)], 319 (35) [M^+ ($2\times^{79}$Br)], 115 (100).



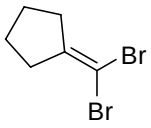
1-(1,1-dibromoprop-1-en-2-yl)-4-chlorobenzene (1e)[6]: yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.34 (AA' of AA'BB', *J* = 8.5 Hz, 2H), 7.17 (BB' of AA'BB', *J* = 8.5 Hz, 2H), 2.19 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.07, 140.46, 128.82, 88.46, 26.18; IR (KBr, cm^{-1}): 2915, 2847, 1488, 1016, 834, 540. EI-MS *m/z* (%): 311 (69) [M^+H (^{81}Br, ^{79}Br)], 309 (100) [M^+H ($2\times^{79}$Br)], 150 (65), 115 (87).



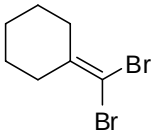
1-bromo-4-(1,1-dibromoprop-1-en-2-yl)benzene (1f): yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.49 (AA' of AA'BB', *J* = 8.5 Hz, 2H), 7.11 (BB' of AA'BB', *J* = 8.5 Hz, 2H), 2.18 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 142.07, 140.94, 131.76, 129.33, 121.93, 88.42, 26.13; IR (KBr, cm^{-1}): 3024, 2914, 2846, 1485, 833; EI-MS *m/z* (%): 355 (79) [M^+H (^{81}Br, ^{79}Br)], 354 (86) [M^+ (^{81}Br, ^{79}Br)], 353 (97) [M^+ ($2\times^{79}$Br)], 115 (100); EI-HRMS *m/z calcd.* for $C_9H_7Br_3$ 351.8098; *m/z* found: 351.8096.



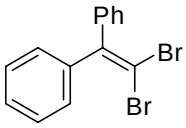
1-bromo-3-(1,1-dibromoprop-1-en-2-yl)benzene (1g): yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.50-7.45 (m, 1H), 7.42 (s, 1H), 7.25-7.15 (m, 1H), 7.15-7.05 (m, 1H), 2.22 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 143.92, 141.70, 130.83, 130.48, 130.06, 126.21, 122.33, 88.82, 26.06; IR (KBr, cm^{-1}): 3059, 2919, 2849, 1558, 826; EI-MS *m/z* (%): 356 (30) [M^+ ($2\times^{81}$Br, ^{79}Br)], 355 (77) [M^+H (^{81}Br, $2\times^{79}$Br)], 354 (73) [M^+ (^{81}Br, $2\times^{79}$Br)], 353 (80) [M^+H ($3\times^{79}$Br)], 115 (100); EI-HRMS *m/z calcd.* for $C_9H_7Br_3$ 351.8098; *m/z* found: 351.8086.



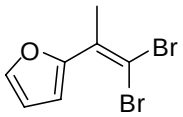
(dibromomethylene)cyclopentane (1h)[7]: colorless oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 2.36-2.28 (m, 4H), 1.84-1.76 (m, 4H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ 151.24, 78.45, 36.53, 27.36; IR (KBr, cm^{-1}): 2959, 2886, 2868, 804; EI-MS *m/z* (%): 242 (4) [M^+ ($2\times^{81}$Br)], 240 (63) [M^+ (^{81}Br, ^{79}Br)], 238 (32) [M^+ ($2\times^{79}$Br)], 198 (50), 161 (39), 159 (35), 79 (100), 77 (45).



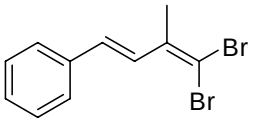
(dibromomethylene)cyclohexane (1i)[7]: colorless oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 2.42-2.35 (m, 4H), 1.61-1.50 (m, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 145.14, 81.94, 34.89, 26.93, 25.91; IR (KBr, cm^{-1}): 2974, 2931, 2854, 790; EI-MS *m/z* (%): 255 (27) [M^+H (^{81}Br, ^{79}Br)], 253 (52) [M^+H ($2\times^{79}$Br)], 211 (28), 93 (53), 68 (100).



(2,2-dibromoethene-1,1-diyl)dibenzene (1j)[8]**:** white solid. M.p. 78-80 ^{o}C (Lit.[12] 83.5 ^{o}C); ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.44-7.23 (m, 10H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 141.50, 128.87, 128.48, 128.12, 90.43; IR (KBr, cm^{-1}): 3438, 3054, 696, 594; EI-MS *m/z* (%): 340 (15) [M^+ ($2\times^{81}$Br)], 338 (31) [M^+ (^{81}Br, ^{79}Br)], 336 (17) [M^+ ($2\times^{79}$Br)], 178 (100).

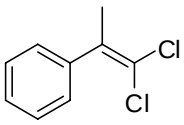


2-(1,1-dibromoprop-1-en-2-yl)furan (1k): green oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.47-7.41 (m, 1H), 6.93-6.90 (m, 1H), 6.48-6.42 (m, 1H), 2.26 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 151.48, 142.09, 131.68, 112.01, 111.44, 87.30, 22.69; IR (KBr, cm^{-1}): 3119, 2925, 2854, 824, 740; EI-MS *m/z* (%): 267 (54) [M^+H (^{81}Br, ^{79}Br)], 265 (100) [M^+H ($2\times^{79}$Br)], 106 (22), 78 (34). EI-HRMS *m/z calcd.* for $C_7H_6Br_2O$ 263.8785; *m/z* found: 263.8788.

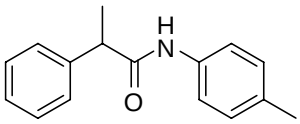


1-((*E*)-4,4-dibromo-3-methylbuta-1,3-dienyl)benzene (1l)[13]**:** green solid. M.p. 50-52 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.52-7.45 (m, 2H), 7.39-7.32 (m, 2H), 7.32-7.27 (m, 1H), 7.25 (d, J = 16 Hz, 1H), 6.75 (d, J = 16 Hz, 1H), 2.14 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 137.94, 136.88, 132.66, 128.91, 128.43, 127.34, 126.97, 92.65, 19.37; IR (KBr, cm^{-1}): 3429, 3945, 958, 815, 750, 688; EI-MS *m/z* (%): 304 (6) [M^+ ($2\times^{81}$Br)], 302 (12) [M^+ (^{81}Br,

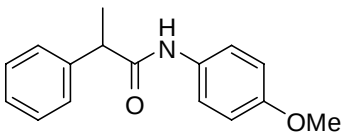
^{79}Br)], 300 (6) [M$^+$ (2×^{79}Br)], 142 (100), 141(39).



1-(1,1-dichloroprop-1-en-2-yl)benzene (1m)[10]**:** colorless oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.42-7.35 (m, 2H), 7.41-7.29 (m, 1H), 7.29-7.26 (m, 2H), 2.22 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 140.22, 135.85, 128.47, 127.90, 127.85, 117.13, 23.21; IR (KBr, cm^{-1}): 3057, 3025, 2918, 898, 697; EI-MS *m/z* (%): 190 (9) [M$^+$ (2×^{37}Cl)], 188 (45) [M$^+$ (^{37}Cl, ^{35}Cl)], 186 (74) [M$^+$ (2×^{35}Cl)], 115 (100), 78 (38).

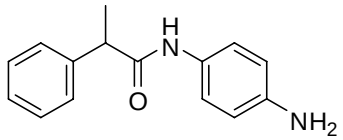


2-phenyl-N-p-tolylpropanamide (3aa)[14]: white solid M.p. 116-117 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.42-7.34 (m, 4H), 7.34-7.27 (m, 1H), 7.30 (AA'of AA'BB', *J* = 8.5 Hz, 2H), 7.07 (BB'of AA'BB', *J* = 8.5 Hz, 2H), 7.12-7.05 (m, 1H), 3.70 (q, *J* = 7 Hz , 1H), 2.28 (s, 3H), 1.59 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.32, 141.15, 135.40, 133.99, 129.50, 129.25, 127.84, 127.65, 119.90, 48.16, 20.95, 18.71; IR (KBr, cm^{-1}): 3285, 3248, 1652, 1603, 1537, 818, 696; EI-MS *m/z* (%): 239 (73) [M$^+$], 133 (27), 107 (100), 106 (60), 105 (72).

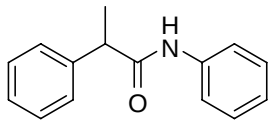


N-(4-methoxyphenyl)-2-phenylpropanamide (3ab)[15]: white solid. M.p. 117-118 ^{o}C; ^{1}H

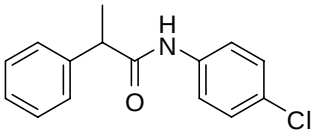
NMR ($CDCl_3$, 500 MHz): δ 7.41-7.34 (m, 5H), 7.32 (AA' of AA'BB', *J* = 9 Hz, 2H), 7.11 (bs, 1H), 6.79 (BB' of AA'BB', *J* = 9.5 Hz, 2H), 3.76 (s, 3H), 3.70 (q, *J* = 7 Hz ,1H), 1.59 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.23, 156.37, 141.10, 130.99, 129.12, 127.73, 127.52, 121.66, 114.04, 55.48, 47.90, 18.63; IR (KBr, cm^{-1}): 3289, 1655, 1514, 1237, 1173, 1029, 827; EI-MS *m/z* (%): 255 (78) [M^+], 123 (100), 105 (60).



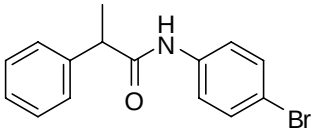
N-(4-aminophenyl)-2-phenylpropanamide (3ac)[16]: yellow solid. M.p. 76-78 ^oC; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.38-7.35 (m, 4H), 7.32-7.27 (m, 1H) , 7.19-7.15 (m, 2H), 7.02 (bs, 1H), 6.59-6.56 (m, 2H), 3.87 (q, *J* = 7 Hz, 1H), 3.55 (bs, 2H), 1.57 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.37, 143.34, 141.33, 129.27, 128.97, 127.71, 127.34, 122.04, 115.30, 47.64, 18.68; IR (KBr, cm^{-1}): 3441, 1652, 1514, 698, 515; EI-MS *m/z* (%): 240 (17) [M^+], 214 (83), 212 (82), 121 (41), 86 (65), 84 (100).



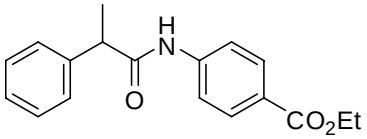
N,2-diphenylpropanamide (3ad) [17]**:** pale yellow solid. M.p. 132-134 ^oC (Lit.[17] 136 ^oC); ^1H NMR ($CDCl_3$, 500 MHz): δ 7.50-7.35 (m, 6H), 7.35-7.25 (m, 3H), 7.15-7.02 (m, 2H), 3.72 (q, *J* = 7 Hz, 1H), 1.61 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.39, 141.03, 137.95, 129.30, 129.04, 127.84, 127.73, 124.38, 119.79, 48.27, 18.68; IR (KBr, cm^{-1}): 3298, 3248, 1659, 1544, 755, 694; EI-MS *m/z* (%): 225 (68) [M^+], 106 (65), 105 (100), 93 (51), 77 (37).



N-(4-chlorophenyl)-2-phenylpropanamide (3ae)[16]: white solid. M.p. 132-133 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.43-7.28 (m, 7H), 7.26 (s, 1H), 7.21 (d, J = 9 Hz, 2H), 3.71 (q, J = 7 Hz, 1H), 1.59 (d, J = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.56, 140.80, 136.52, 129.32, 129.00, 127.80, 127.78, 121.15, 48.15, 18.66; IR (KBr, cm^{-1}): 3290, 3251, 1660, 1602, 1537, 1490, 1395, 828, 725; EI-MS *m/z* (%): 261 (13) [M^+ (^{37}Cl)], 259 (39) [M^+ (^{35}Cl)], 105 (100).

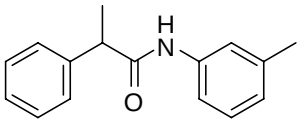


N-(4-bromophenyl)-2-phenylpropanamide (3af): yellow solid. M.p. 129-130 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.44-7.26 (m, 10H), 3.70 (q, J = 7 Hz, 1H), 1.58 (d, J = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.61, 140.80, 137.05, 131.95, 129.32, 127.81, 127.78, 121.51, 116.95, 48.16, 18.67; IR (KBr, cm^{-1}): 3443, 3293, 3253, 1661, 1534, 1489, 826, 725; EI-MS *m/z* (%): 305 (28) [M^+ (^{81}Br)], 303 (27) [M^+ (^{79}Br)], 105 (100); Anal. Calcd. for $C_{15}H_{14}BrNO$: C, 59.23; H, 4.64; N, 4.60. Found: C, 59.53; H, 4.68; N, 4.55.

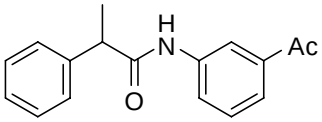


ethyl 4-(2-phenylpropanamido)benzoate (3ag): white solid. M.p. 102-103 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.95 (AA' of AA'BB', J = 7 Hz, 2H), 7.51 (BB' of AA'BB', J = 7 Hz, 2H), 7.45-7.25 (m, 6H), 4.33 (q, J = 7 Hz, 2H), 3.73 (q, J = 7 Hz, 1H), 1.59 (d, J = 7 Hz,

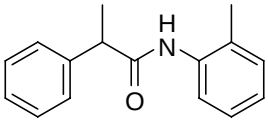
3H), 1.37 (t, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 173.02, 166.34, 142.31, 140.69, 130.68, 129.11, 127.61, 125.75, 118.96, 60.95, 48.04, 18.68, 14.35; IR (KBr, cm^{-1}): 3529, 3324, 2979, 1715, 1597, 1530, 1277, 770, 698; EI-MS *m/z* (%): 297 (46) [M^+], 296 (49), 165 (23), 132 (55) ,106 (53), 105 (100), 91 (20). HRMS (EI^+): m/z calcd for $C_{18}H_{19}NO_3$ 297.1365; found: 297.1368.



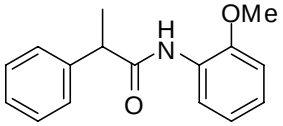
2-phenyl-N-m-tolylpropanamide (3ah)[18]: yellow solid. M.p. 99-100 ^{o}C; ^{1}HNMR ($CDCl_3$, 500MHz): δ 7.42-7.34 (m, 4H), 7.34-7.27 (m, 2H), 7.23-7.11 (m, 3H), 6.90-6.80 (m, 1H), 3.72 (q, *J* = 7 Hz, 1H), 2.29 (s, 3H), 1.60 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.46, 141.08, 138.96, 137.89, 129.23, 128.82, 127.80, 127.65, 125.15, 120.47, 116.88, 48.20, 21.54, 18.69; IR (KBr, cm^{-1}): 3298, 1656, 1612, 1551, 1489, 753, 693; EI-MS *m/z* (%): 239 (91) [M^+], 133 (36), 107 (100), 106 (82), 105 (100), 91 (45), 77 (36).



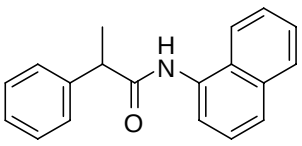
N-(3-acetylphenyl)-2-phenylpropanamide (3ai): yellow solid. M.p. 108-109 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.96 (s, 1H), 7.82 (d, *J* = 7.5 Hz, 1H), 7.72 (s, 1H), 7.63 (d, *J* = 7.5 Hz, 1H), 7.48-7.21 (m, 6H), 3.77 (q, *J* = 7 Hz, 1H), 2.55 (s, 3H), 1.60 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 198.23, 172.93, 140.80, 138.62, 137.69, 129.32, 129.25, 127.72, 124.52, 124.16, 119.30, 48.09, 26.79, 18.65; IR (KBr, cm^{-1}): 3442, 3333, 1691, 1670, 1545, 1486, 1300, 734, 691; EI-MS *m/z* (%): 267 (45) [M^+], 132 (33), 106 (47), 105 (100), 91 (24).



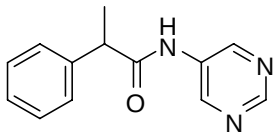
2-phenyl-N-o-tolylpropanamide (3aj)[18]**:** white solid. M.p. 82-83 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.88 (d, *J* = 8.5 Hz, 1H), 7.46-7.37 (m, 4H), 7.37-7.30 (m, 1H), 7.20-7.16 (m, 1H), 7.08 (d, *J* = 7 Hz, 1H), 7.03-6.97 (m, 1H), 6.86 (bs, 1H), 3.79 (q, *J* = 7 Hz, 1H), 1.88 (s, 3H), 1.65 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.31, 140.97, 135.72, 130.31, 129.28, 128.07, 127.90, 127.78, 126.78, 124.79, 122.06, 48.12, 18.01, 17.12; IR (KBr, cm^{-1}): 3288, 1651, 1526, 1454, 749, 694; EI-MS *m/z* (%): 239 (76) [M^+], 133 (31), 107 (88), 106 (70), 105 (100), 91 (40), 77 (32).



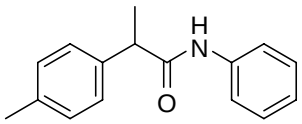
N-(2-methoxyphenyl)-2-phenylpropanamide (3ak)[18]: white solid. M.p. 98-99 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.38 (d, *J* = 8 Hz, 1H), 7.78 (bs, 1H), 7.50-7.20 (m, 5H), 7.10-6.90 (m, 2H), 6.85-6.70 (m, 1H), 3.76 (q, *J* = 7 Hz, 1H), 3.72 (s, 3H), 1.63 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.24, 147.94, 141.13, 129.08, 127.86, 127.50, 123.67, 121.20, 119.56, 110.05, 55.79, 48.55, 18.53; IR (KBr, cm^{-1}): 3370, 1664, 1537, 1459, 1258, 752, 617; EI-MS *m/z* (%): 255 (82) [M^+], 150 (28), 123 (100), 108 (28), 106 (29), 105 (68).



N-(naphthalen-1-yl)-2-phenylpropanamide (3al)[19]: purple solid. M.p. 134-135 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.92 (d, *J* = 7 Hz, 1H), 7.81 (d, *J* = 8 Hz, 1H), 7.64 (d, *J* = 8 Hz, 1H), 7.53 (bs, 1H), 7.51-7.33 (m, 8H), 7.32-7.23 (m, 1H), 3.91 (q, *J* = 7 Hz, 1H), 1.70 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.98, 141.21, 134.10, 132.28, 129.46, 128.80, 128.06, 127.91, 127.04, 126.28, 125.93, 125.80, 125.67, 120.41, 120.22, 48.17, 18.33; IR (KBr, cm^{-1}): 3435, 3246, 1653, 1534, 1499, 787, 699; EI-MS *m/z* (%): 275 (48) [M^+], 143 (100), 115 (18), 105 (46).

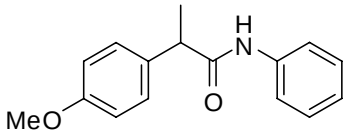


2-phenyl-N-(pyrimidin-5-yl)propanamide (3am): yellow solid. M.p. 108-110 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.89 (s, 2H), 8.86 (s, 1H), 8.02 (bs, 1H), 7.41-7.20 (m, 5H), 3.77 (q, *J* = 7 Hz, 1H), 1.57 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 173.05, 153.99, 147.67, 140.23, 129.34, 127.91, 127.64, 47.76, 18.56; IR (KBr, cm^{-1}): 3453, 2968, 2924, 2855, 1694, 1429, 698. EI-MS *m/z* (%): 227 (20) [M^+], 132 (15), 106 (18), 105 (100), 79 (15), 77 (14). EI-HRMS *m/z calcd. For* $C_{13}H_{13}N_3O$ 227.1059; *m/z* found: 227.1066.

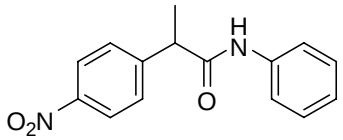


N-phenyl-2-p-tolylpropanamide (3bd)[20]: yellow solid. M.p. 129-130 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.45 (AA' of AA'BB', *J* = 8 Hz, 2H), 7.42 (bs, 1H), 7.35-7.23 (m, 4H), 7.19 (BB' of AA'BB', *J* = 7.5 Hz, 2H), 7.15-7.05 (m, 1H), 3.71 (q, *J* = 7 Hz, 1H), 2.37 (s, 3H), 1.58 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.83, 138.04, 137.99,

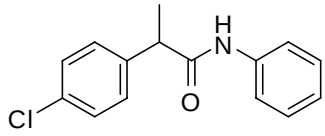
137.27, 129.85, 128.93, 127.65, 124.24, 119.86, 47.64, 21.15, 18.65; IR (KBr, cm^{-1}): 3443, 3248, 1656, 1598, 1544, 1441, 759, 692; EI-MS *m/z* (%): 239 (44) [M^+], 146 (25), 120 (52), 119 (100), 105 (25).



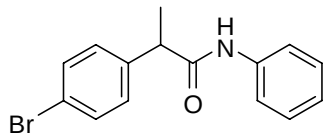
2-(4-methoxyphenyl)-N-phenylpropanamide (3cd)[21]: yellow solid. M.p. 110-112 ^oC; ^1H NMR ($CDCl_3$, 500 MHz): δ 7.42 (AA' of AA'BB', *J* = 8 Hz, 2H), 7.31-7.24 (m, 4H), 7.10-7.0 (m, 2H), 6.92 (BB' of AA'BB', *J* = 9 Hz, 2H), 3.82 (s, 3H), 3.68 (q, *J* = 7 Hz, 1H), 1.58 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.92, 159.03, 138.02, 132.99, 128.98, 128.87, 124.28, 119.80, 114.57, 55.40, 47.29, 18.73; IR (KBr, cm^{-1}): 3322, 3008, 1654, 1513, 1443, 1251, 758, 691; EI-MS *m/z* (%): 255 (20) [M^+], 135 (100), 105 (14), 77 (13).



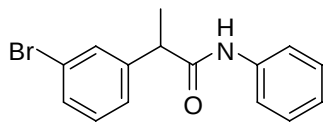
2-(4-nitrophenyl)-N-phenylpropanamide (3dd)[21]: yellow solid. M.p. 155-156 ^oC; ^1H NMR ($CDCl_3$, 500 MHz): δ 8.20 (AA' of AA'BB', *J* = 8.5 Hz, 2H), 7.57 (BB' of AA'BB', *J* = 8.5 Hz, 2H), 7.55-7.40 (m, 3H), 7.35-7.20 (m, 2H), 7.15-7.05 (m, 1H), 3.83 (q, *J* = 7 Hz, 1H), 1.63 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.84, 148.37, 147.21, 137.46, 129.07, 128.53, 124.81, 124.12, 120.02, 47.90, 19.03; IR (KBr, cm^{-1}): 3453, 3244, 3192, 1657, 1598, 1546, 1442, 759; EI-MS *m/z* (%): 270 (68) [M^+], 151 (95), 134 (24), 93 (100), 77 (36).



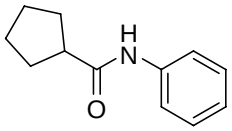
2-(4-chlorophenyl)-N-phenylpropanamide (3ed)[20]: yellow solid. M.p. 156-157 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.43 (AA' of AA'BB', J = 8 Hz, 2H), 7.40-7.25 (m, 4H), 7.34 (BB' of AA'BB', J = 8 Hz, 2H), 7.20-7.05 (m, 2H), 3.68 (q, J = 7 Hz, 1H), 1.57 (d, J = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.15, 139.52, 137.81, 133.39, 129.22, 129.07, 129.04, 124.57, 120.05, 47.40, 18.82; IR (KBr, cm^{-1}): 3453, 3244, 3192, 1657, 1598, 1546, 1442, 759; EI-MS *m/z* (%): 261 (26) [M^+ (^{37}Cl)], 259 (85) [M^+ (^{35}Cl)], 139 (100).



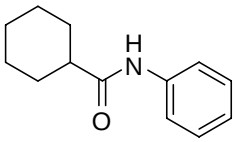
2-(4-bromophenyl)-N-phenylpropanamide (3fd): white solid. M.p. 127-129 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.51 (AA' of AA'BB', J = 8 Hz, 2H), 7.45 (BB' of AA'BB', J = 8 Hz, 2H), 7.38-7.23 (m, 4H), 7.19 (bs, 1H), 7.15-7.05 (m, 1H), 3.68 (q, J = 7 Hz, 1H), 1.59 (d, J = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.80, 140.05, 137.77, 132.31, 129.49, 129.10, 124.60, 121.61, 119.92, 47.69, 18.84; IR (KBr, cm^{-1}): 3451, 3246, 3192, 1658, 1598, 1545, 1440, 758; EI-MS *m/z* (%): 305 (23) [M^+ (^{81}Br)], 303 (24) [M^+ (^{79}Br)], 184 (26), 93 (100); Anal. Calcd. for $C_{15}H_{14}BrNO$: C, 59.23; H, 4.64; N, 4.60. Found: C, 58.83; H, 4.90; N, 4.32.



2-(3-bromophenyl)-N-phenylpropanamide (3gd): white solid. M.p. 128-130 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.52 (s, 1H), 7.48-7.41 (m, 3H), 7.34-7.26 (m, 3H), 7.26-7.20 (m, 1H), 7.15 (bs, 1H), 7.12-7.04 (m, 1H), 3.66 (q, *J* = 7 Hz, 1H), 1.57 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 171.67, 143.34, 137.78, 130.88, 130,78, 130.73, 129.09, 126.34, 124.61, 123.16, 120.01, 47.85, 18.86; IR (KBr, cm^{-1}): 3433, 3234, 3061, 1644, 1596, 1545, 1444, 755, 692; EI-MS *m/z* (%): 305 (28) [M^+ (^{81}Br)] , 303 (30) [M^+ (^{79}Br)], 229 (100), 194 (70), 149 (80), 105 (63), 93 (82). Anal. Calcd. for $C_{15}H_{14}BrNO$: C, 59.23; H, 4.64; N, 4.60. Found: C, 59.45; H, 4.68; N, 4.50.

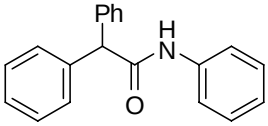


N-phenylcyclopentanecarboxamide (3hd): gray solid. M.p. 162-163 ^{o}C (Lit.[22] 160.1-161.2 ^{o}C); ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.60-7.45 (m, 2H), 7.35-7.30 (m, 2H), 7.17 (bs, 1H), 7.15-7.05 (m, 1H), 2.75-2.60 (m, 1H), 2.01-1.85 (m, 4H), 1.85-1.70 (m, 2H), 1.70-1.61 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 175.00, 138.35, 128.99, 124.09, 119.92, 46.88, 30.65, 26.11; IR (KBr, cm^{-1}): 3282, 3251, 2961, 1655, 1599, 1545, 1442, 1317, 1245, 757, 692; EI-MS *m/z* (%): 189 (21) [M^+], 93 (100), 69 (36).

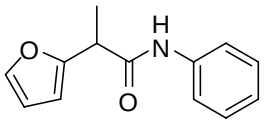


N-phenylcyclohexanecarboxamide (3id): white solid. M.p. 145-146 ^{o}C (Lit.[23] 149 ^{o}C); ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.60-7.45 (m, 2H), 7.40-7.25 (m, 2H), 7.22 (s, 1H), 7.15-7.0 (m, 1H), 2.29-2.17 (m, 1H), 2.01-1.90 (m, 2H), 1.90-1.77 (m, 2H), 1.77-1.66 (m,

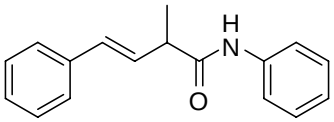
1H), 1.61-1.48 (m, 2H), 1.37-1.20 (m, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 174.52, 138.24, 129.12, 129.09, 124.19, 120.14, 119.87, 46.69, 29.80, 25.80; IR (KBr, cm^{-1}): 3449, 3242, 2930, 2850, 1659, 1598, 1440, 755, 689; EI-MS *m/z* (%): 203 (22) [M^+], 93 (100), 89 (29), 55 (23).



N,2,2-triphenylacetamide (3jd): yellow solid. M.p. 173-175 ^{o}C (Lit.[24] 177-179 ^{o}C); ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.55-7.45 (m, 2H), 7.45-7.20 (m, 13H), 7.15-7.05 (m, 1H), 5.09 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.34, 139.19, 137.77, 129.06, 129.05, 129.00, 127.59, 124.62, 119.97, 60.02; IR (KBr, cm^{-1}): 3445, 3305, 3201, 1656, 1549, 1442, 742, 691; EI-MS *m/z* (%): 287 (3) [M^+], 149 (100), 105 (38).



2-(furan-2-yl)-N-phenylpropanamide (3kd)[25]: yellow solid. M.p. 100-101 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.49-7.40 (m, 3H), 7.40 (bs, 1H), 7.32-7.27 (m, 2H), 7.11-7.07 (m, 1H), 6.40 (dd, *J* = 3, 2 Hz, 1H), 6.29 (d, *J* = 3 Hz, 1H), 3.83 (q, *J* = 7 Hz, 1H), 1.61 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.27, 153.82, 142.46, 137.84, 129.03, 124.46, 119.99, 110.81, 107.01, 41.88, 15.80; IR (KBr, cm^{-1}): 3256, 3200, 3139, 1660, 1602, 1548, 1440, 761, 738; EI-MS *m/z* (%): 215 (47) [M^+], 96 (89), 95 (100), 81 (49).



(*E*)-2-methyl-N,4-diphenylbut-3-enamide (3ld): white solid. M.p. 155-156 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.51 (d, *J* = 8 Hz, 2H), 7.45-7.30 (m, 7H), 7.28 (d, *J* = 16 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.63 (d, *J* = 16 Hz, 1H), 6.34 (dd, *J* = 16, 8 Hz, 1H), 3.31 (dq, *J* = 7 Hz, 1H), 1.46 (d, *J* = 7 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 172.07, 137.93, 136.52, 132.90, 129.20, 129.12, 128.84, 128.10, 126.54, 124.47, 119.90, 46.06, 17.42; IR (KBr, cm^{-1}): 3439, 3251, 3192, 3132, 1661, 1543, 1441, 757, 693; EI-MS *m/z* (%): 251 (16) [M^{+}], 132 (100), 131 (81), 117 (58), 91 (48), 77 (17). EI-HRMS *m/z calcd. For* $C_{17}H_{17}NO$ 251.1310; *m/z* found: 251.1316.

(E) References:

1. W. L. F. Armarego, C. L. L. Chai, *Purification of Laboratory Chemicals*, 5th ed. Elsevier, 2003.
2. R. Rabinowitz and R. Marcus, *J. Am. Chem. Soc*., 1962, **84**, 1312.
3. Y.-Q. Fang, O. Lifchits and M. Lautens, *Synlett*., 2008, **3**, 413.
4. E. J. Corey and P. L. Fuchs, *Tetrahedron Lett*., 1972, **36**, 3769.
5. Y. Okamoto, Y. Yoshikawa and T. Hayashi, *J. Organomet. Chem.* 1989, **359**, 143.
6. V. N. Korotchenko, A. V. Shastin, V. G. Nenajdenko and E. S. Balenkova, *J. Chem. Soc., Perkin Trans. 1*., 2002, **7**, 883.
7. V. N. Korotchenko, A. V. Shastin, V. G. Nenajdenko, E. S. Balenkova, *Org. Biomol. Chem*., 2003, **1**, 1906.
8. P. M. Donovan and L. T. Scott, *J. Am. Chem. Soc*., 2004, **126**, 3108.
9. P. Savignac and P. Coutrot, *Synthesis*, 1976, **3**, 197.

10. C.-T. Chien, C.-C. Tsai, C.-H. Tsai, T.-Y. Chang, P.-K. Tsai, Y.-C. Wang and T.-H. Yan, *J. Org. Chem*., 2006, **71**, 4324.

11. S. Chavda, E. Coulbeck, G. C. Coumbarides, M. Dingjan, J. Eames, S. Ghilagaber and Y. Yohannes, *Tetrahedron-Asymmetry*., 2006, **17**, 3386.

12. C. F. H. Allen and C. V. Wilson, *J. Org. Chem*., 1940, **5**, 146.

13. J. Uenishi, R. Kawahama, O. Yonemitsu and J. Tsuji, *J. Org. Chem*., 1998, **63**, 8965.

14. B. J. R. Nicolaus, L. Mariani , G. Gallo and E. Testa, *J. Org. Chem*., 1961, **26**, 2253.

15. P. J. Dunn, W. Hoffmann, Y. Kang, J. C. Mitchell and M. J. Snowden, *Org. Process Res. Dev.*, 2005, **9**, 956.

16. Q.-F. Yang, C. Olmsted and B. Borhan, *Org. Lett*., 2002, **4**, 3423.

17. I. Shiina and Y.-I. Kawakita, *Tetrahedron*, 2004, **60**, 4729.

18. M. H. Hyun, J.-J. Ryoo, Y. J. Cho and J. S. Jin, *J. Chromatogr*., *A* 1995, ***692***, 91.

19. G. Uray, N. M. Maier, K. S. Niederreiter and M. M. Spitaler, *J. Chromatogr*., *A* 1998, **799**, 67.

20. A. L. Wilds and A. L. Jr. Meader, *J. Org. Chem.*, 1948, **13**, 763.

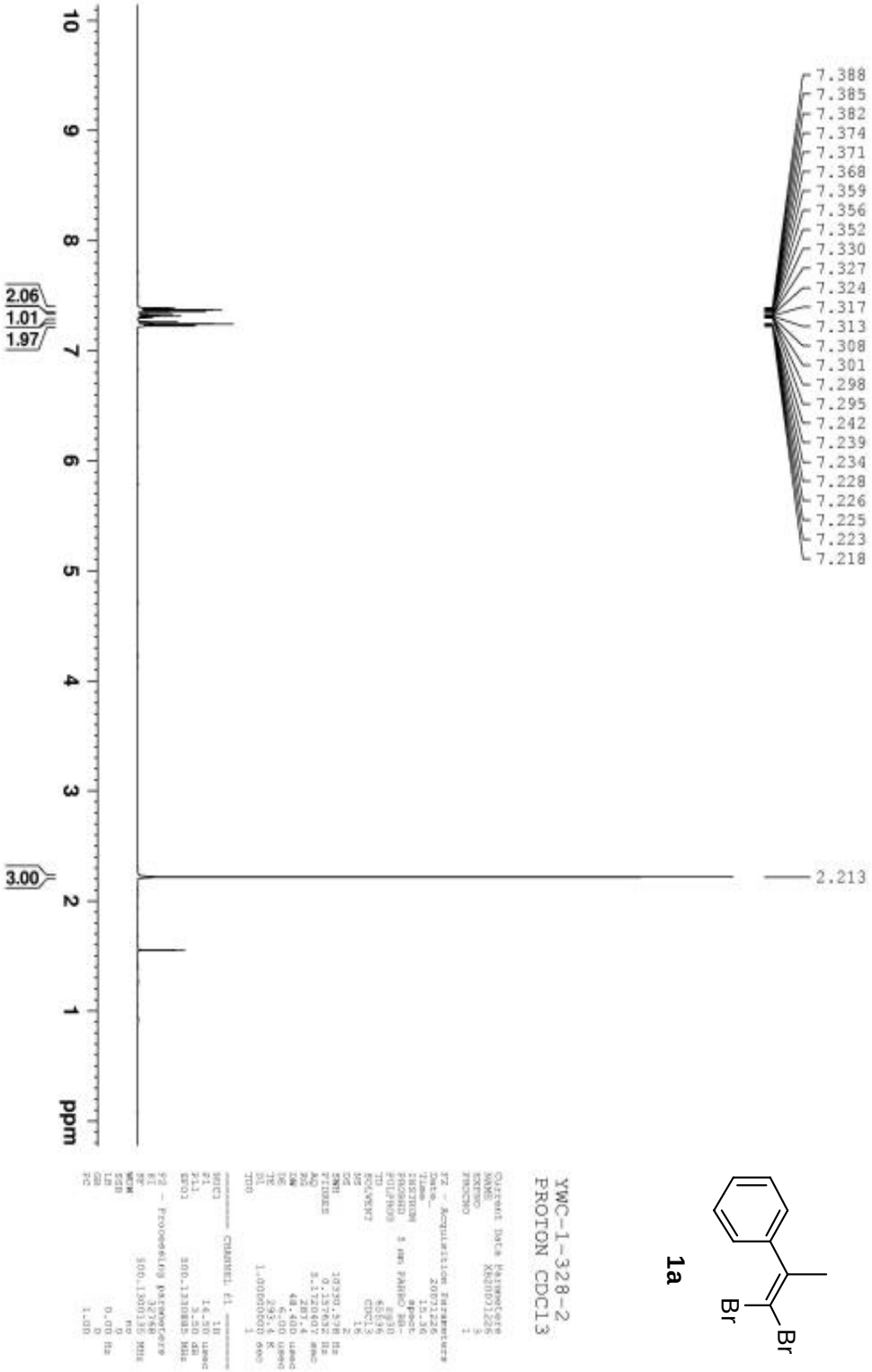
21. R. D. Kleene, *J. Am. Chem. Soc*., 1949, **71**, 2259.

22. R. C. Krug, L. W. Smith and C. E. Fry, *J. Am. Chem. Soc*., 1954, **76**, 3222.

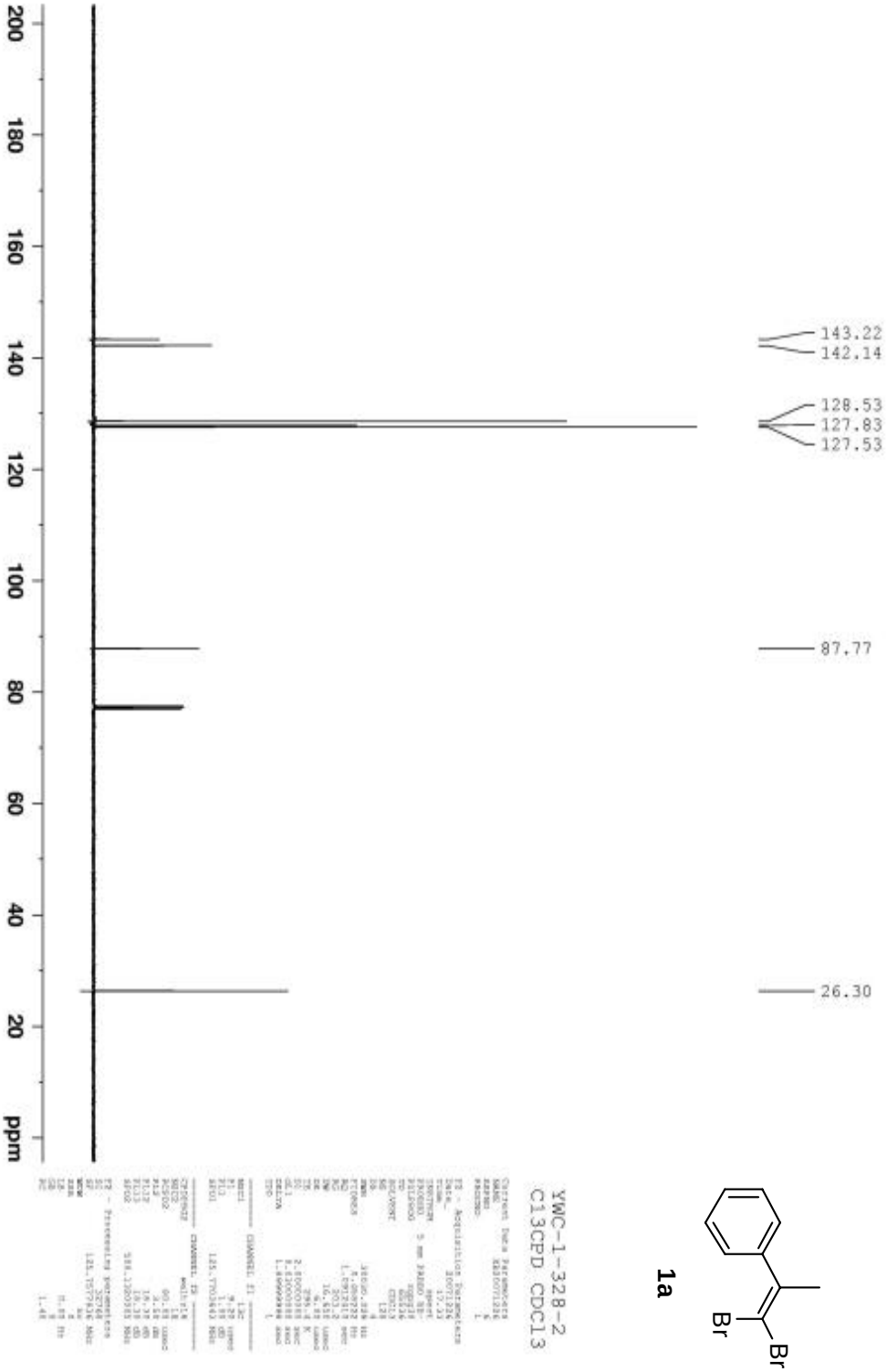
23. J. Vigier and A. Boucherle, *Bull. Soc. Chim. Fr*., 1965, **8**, 2339.

24. J. A. King and F. H. McMillan, *J. Am. Chem. Soc*., 1951, **73**, 4911.

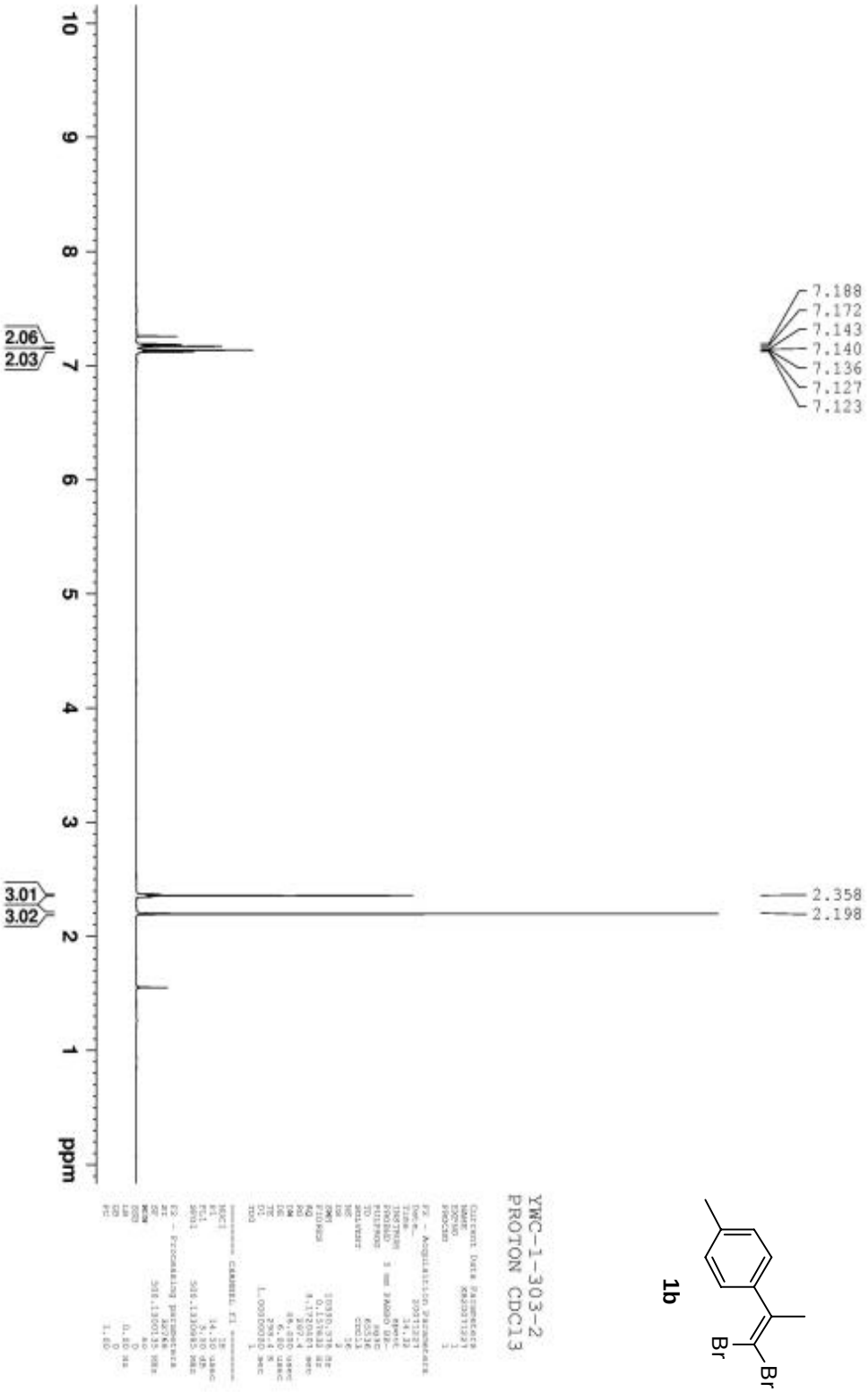
25. H. Hoberg, D. Guhl and P. Betz, *J. Organomet. Chem*., 1990, **387**, 233.


7.388
7.385
7.382
7.374
7.371
7.368
7.359
7.356
7.352
7.330
7.327
7.324
7.317
7.313
7.308
7.301
7.298
7.295
7.242
7.239
7.234
7.228
7.226
7.225
7.223
7.218
2.213
2.06
1.01
1.97
3.00
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-328-2
PROTON CDCl3
Br
Br

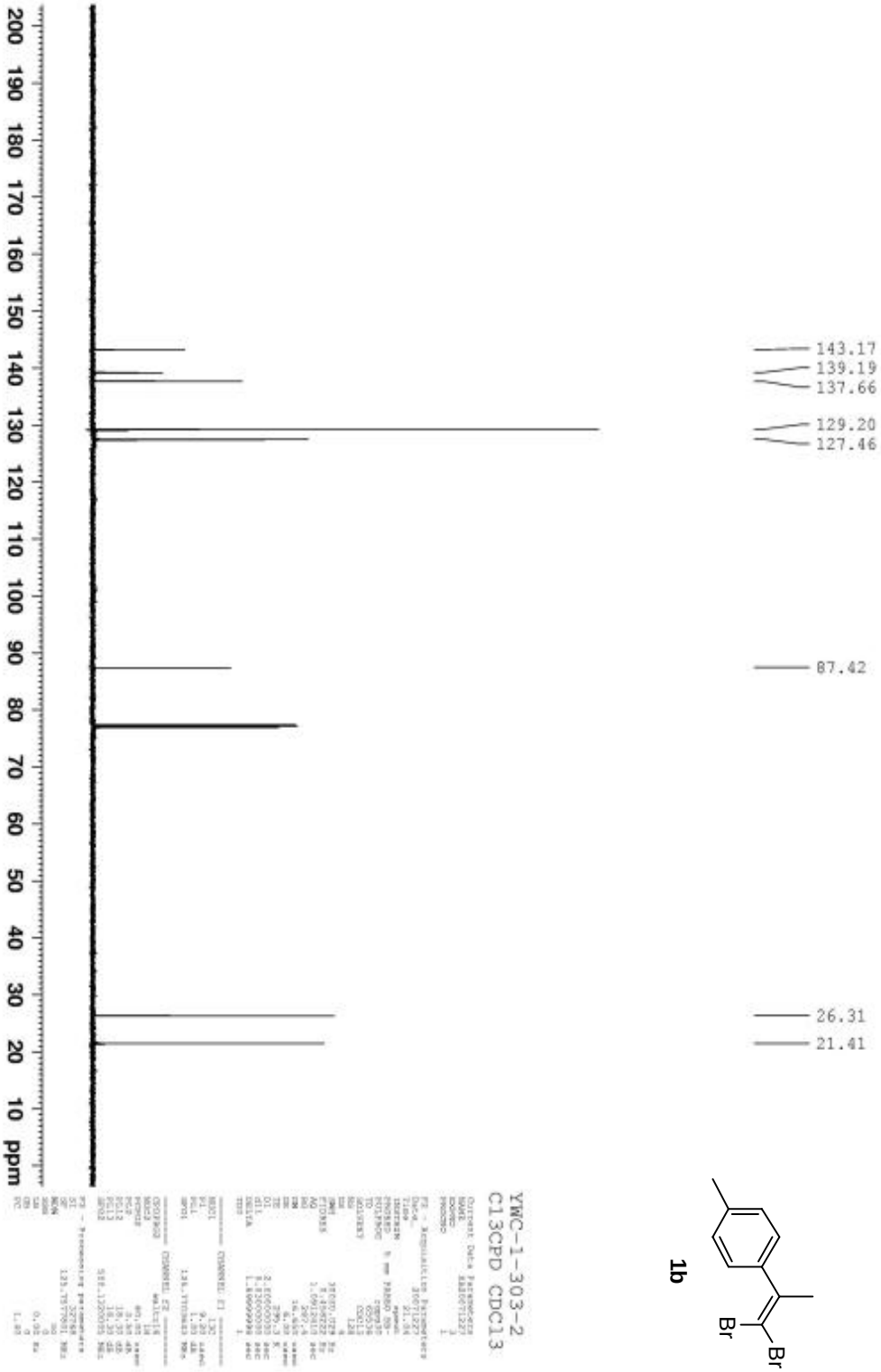
1a


200
180
160
140
120
100
80
60
40
20
ppm
143.22
142.14
128.53
127.83
127.53
87.77
26.30
YWC-1-328-2
C13CPD CDCl3
Br
Br

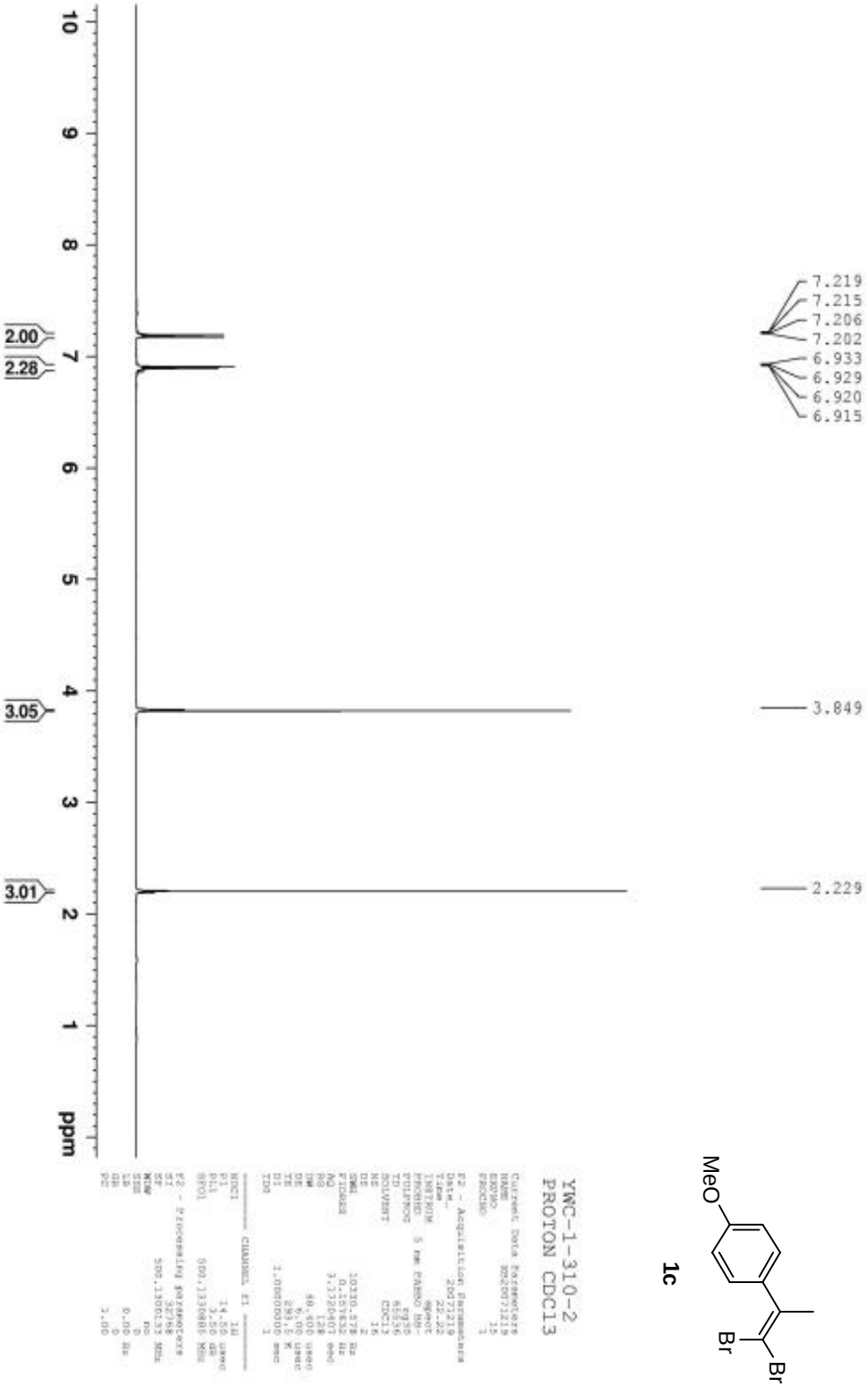
1a


7.188
7.172
7.143
7.140
7.136
7.127
7.123
2.358
2.198
2.06
2.03
3.01
3.02
ppm
YWC-1-303-2
PROTON CDC13
Br
Br

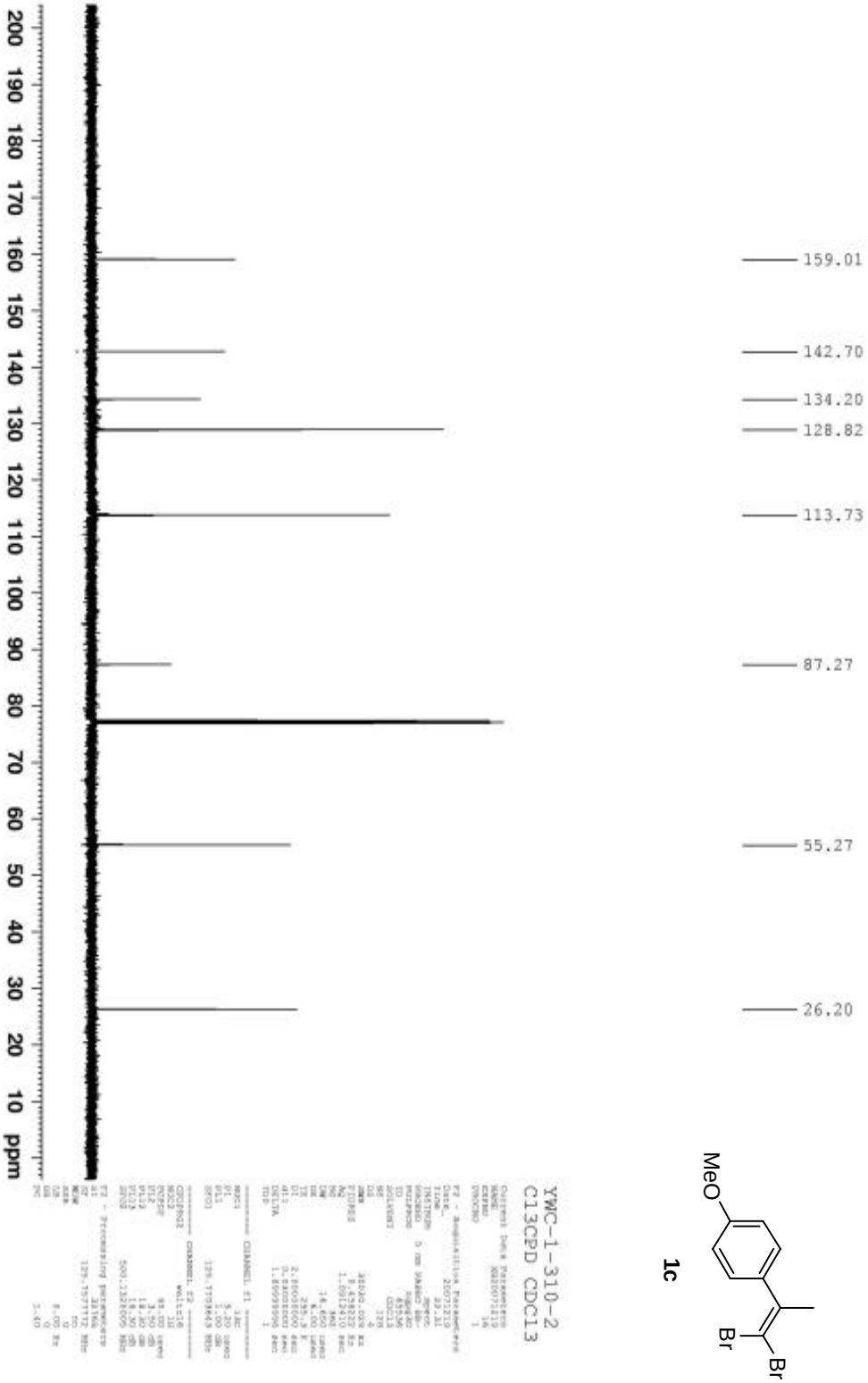
1b


YWC-1-303-2
C13CPD CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
143.17
139.19
137.66
129.20
127.46
87.42
26.31
21.41
Br
Br

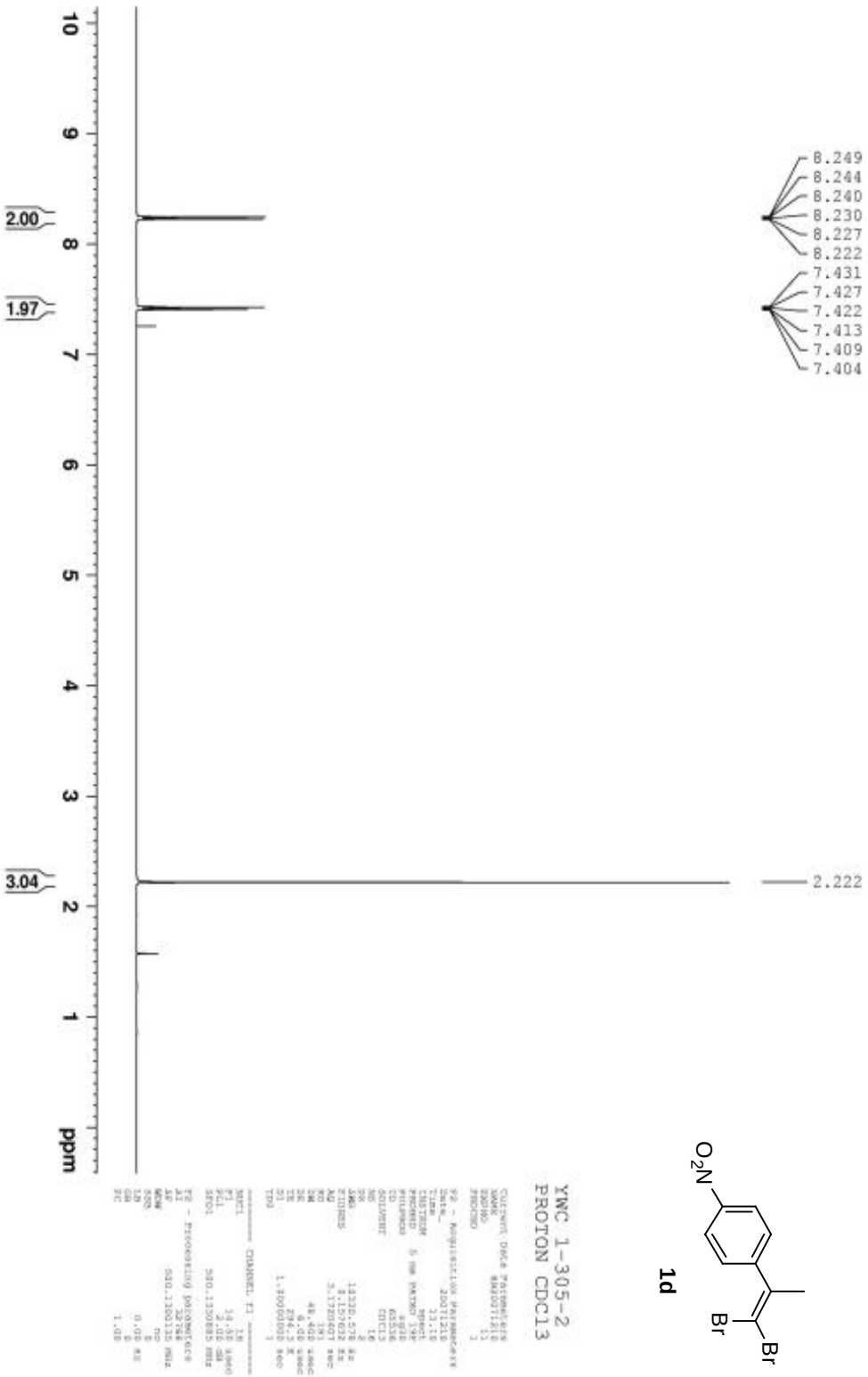
1b


7.219
7.215
7.206
7.202
6.933
6.929
6.920
6.915
3.849
2.229
2.00
2.28
3.05
3.01
ppm
YWC-1-310-2
PROTON CDC13
MeO
Br
Br

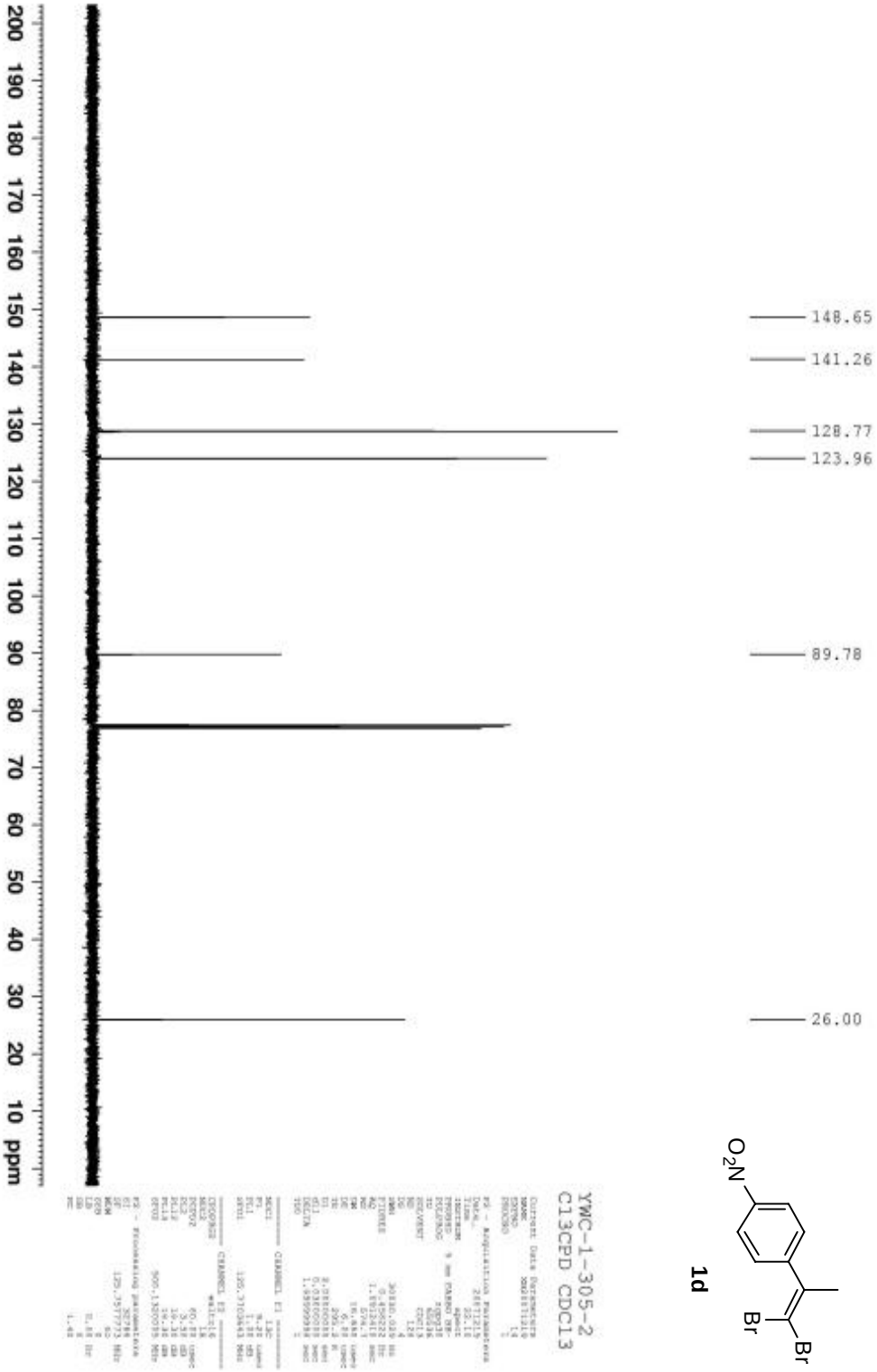
1c


159.01
142.70
134.20
128.82
113.73
87.27
55.27
26.20
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-310-2
C13CPD CDCl3
MeO
Br
Br

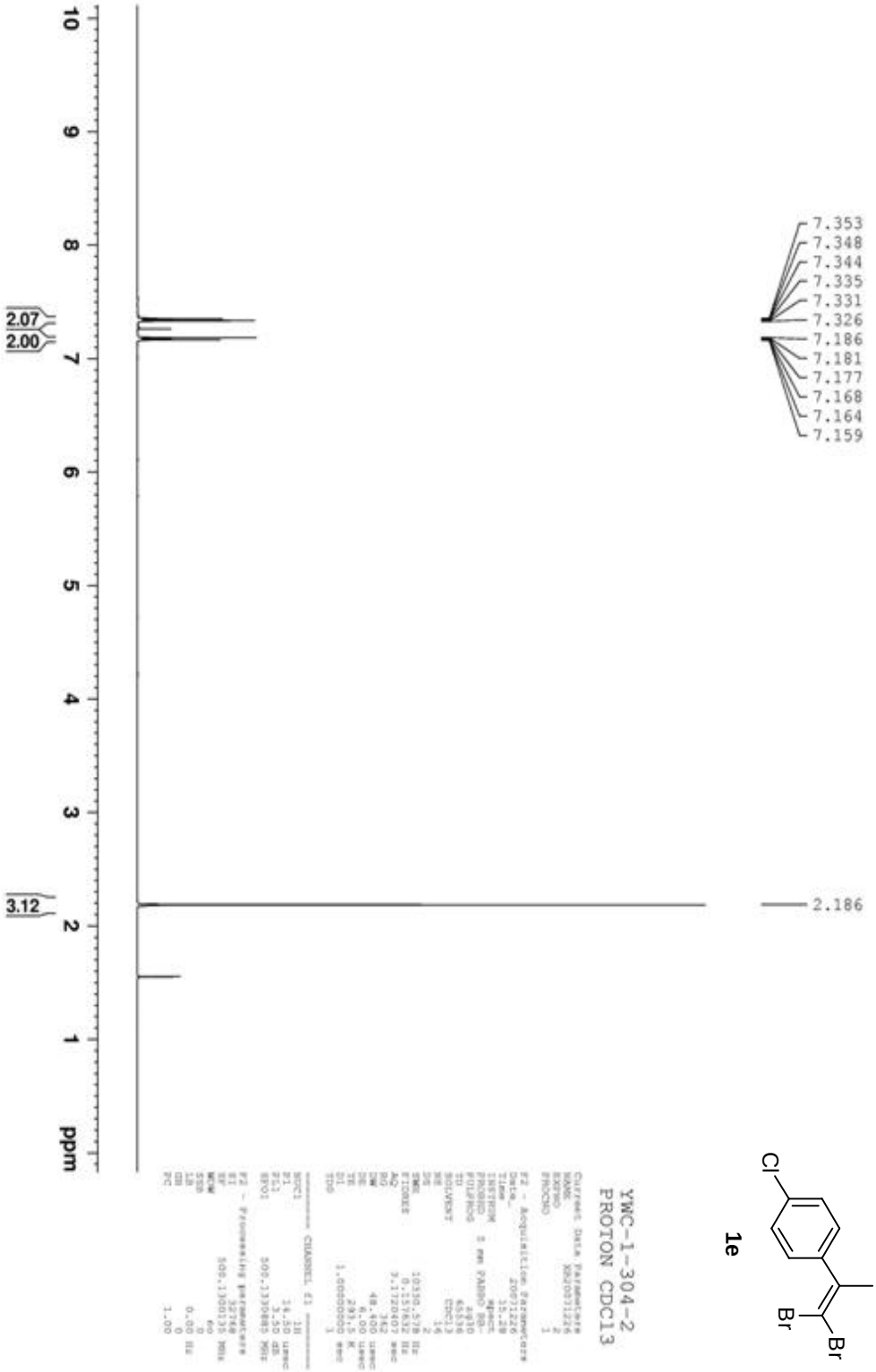
1c


8.249
8.244
8.240
8.230
8.227
8.222
7.431
7.427
7.422
7.413
7.409
7.404
2.222
2.00
1.97
3.04
10
9
8
7
6
5
4
3
2
1
ppm
YWC 1-305-2
PROTON CDC13
O2N
Br
Br

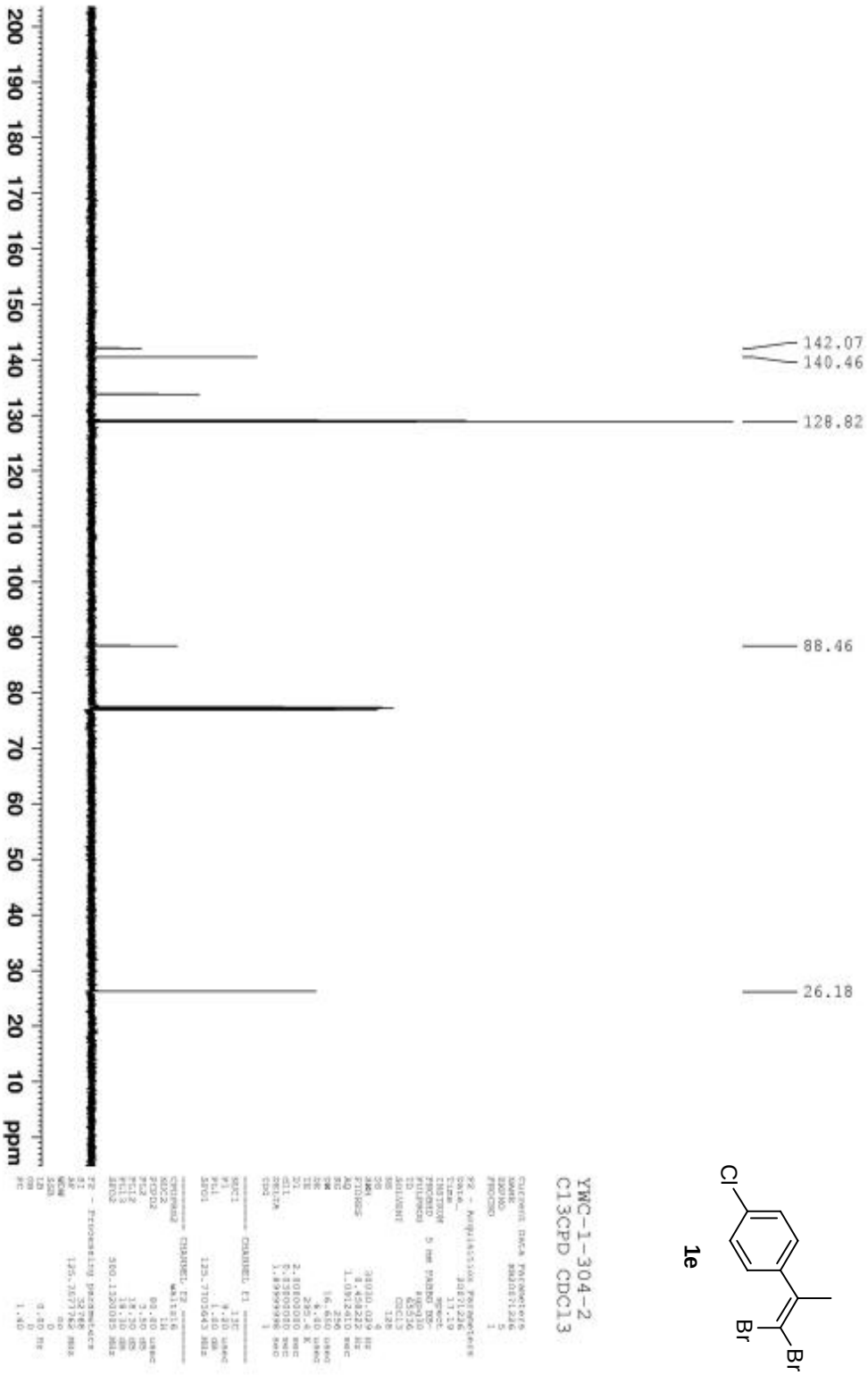
1d


148.65
141.26
128.77
123.96
89.78
26.00
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-305-2
C13CPD CDCl3
O2N
Br
Br

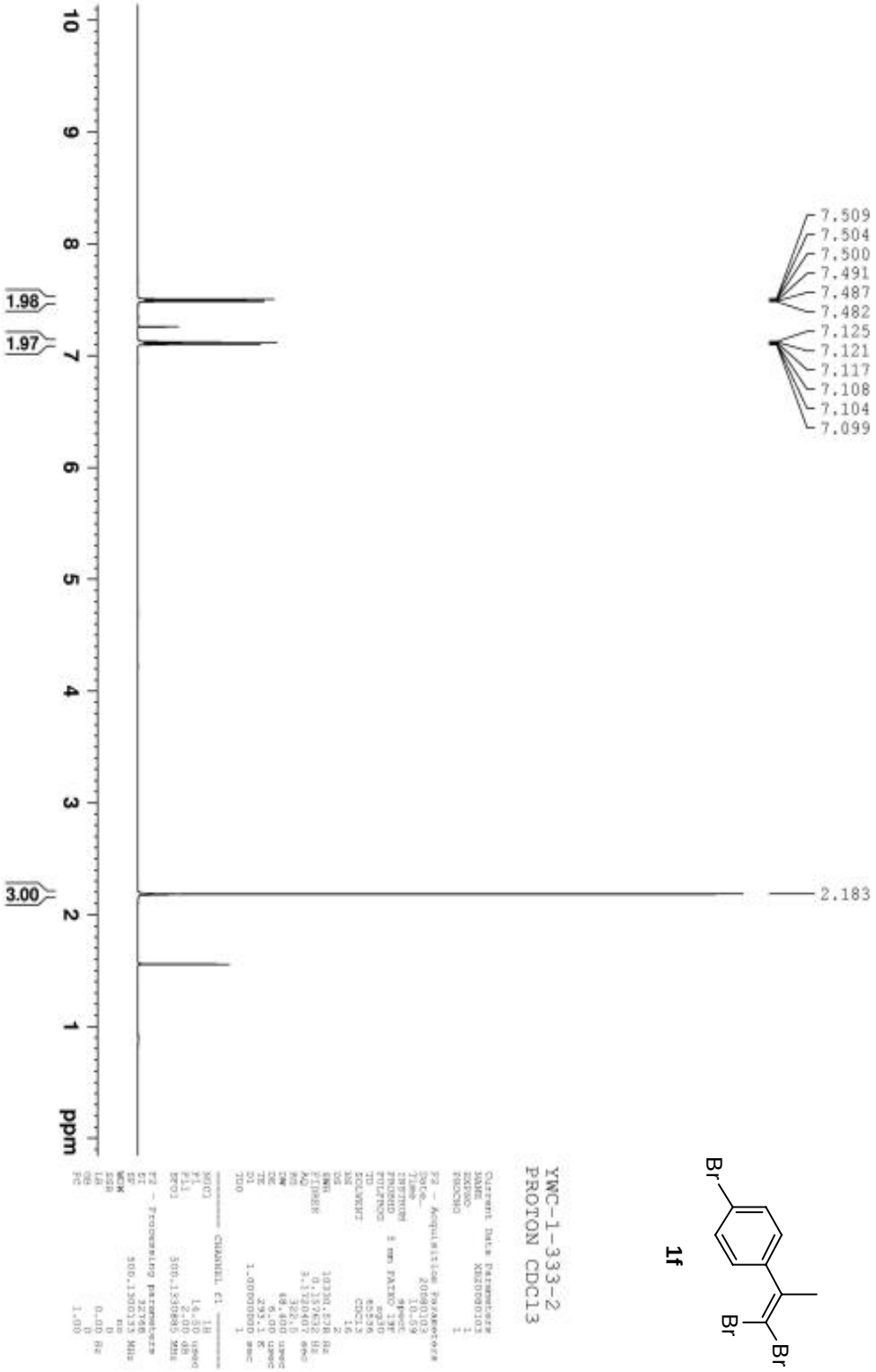
1d


7.353
7.348
7.344
7.335
7.331
7.326
7.186
7.181
7.177
7.168
7.164
7.159
2.186
2.07
2.00
3.12
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-304-2
PROTON CDCl3
Cl
Br
Br

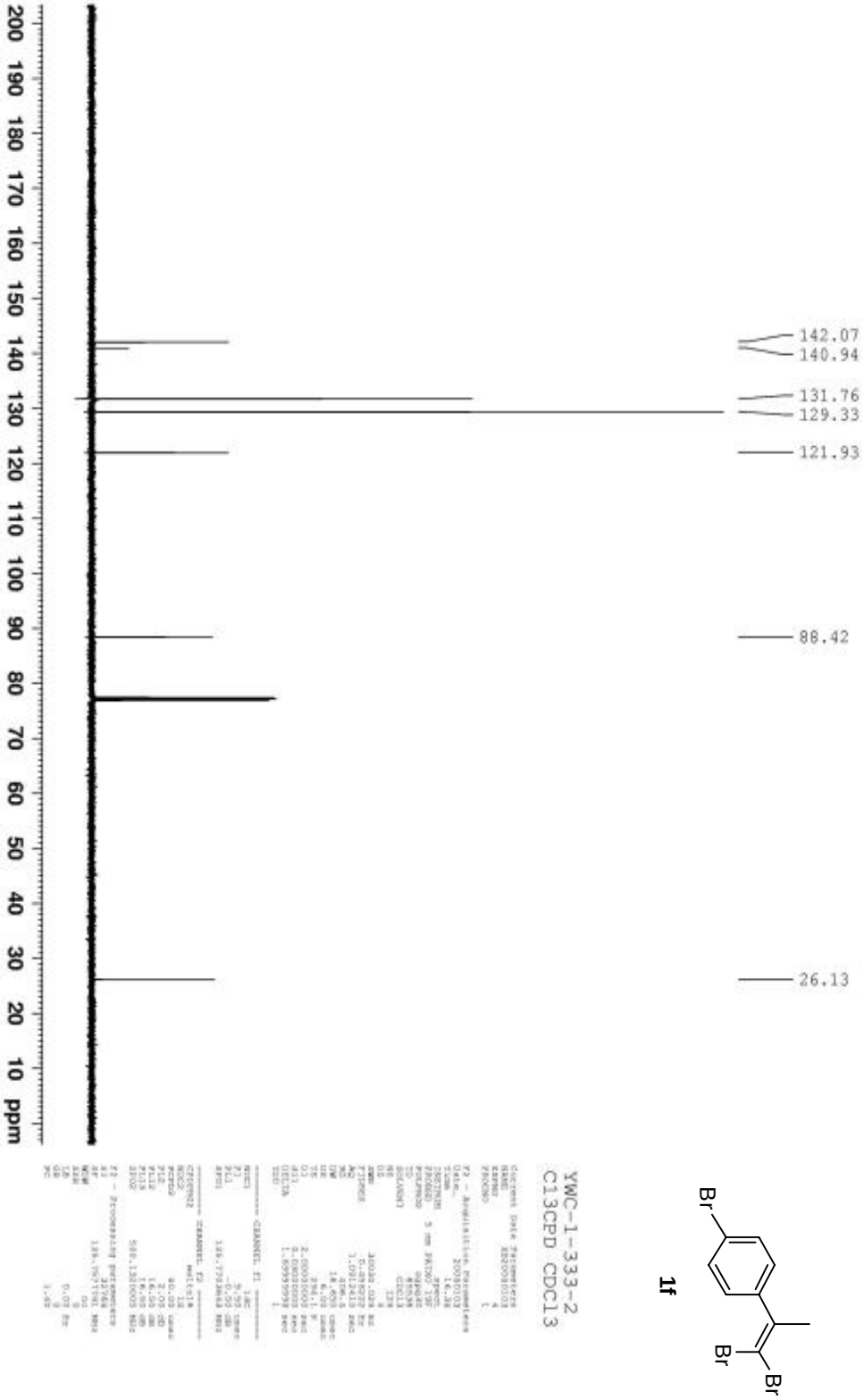
1e


142.07
140.46
128.82
88.46
26.18
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-304-2
C13CPD CDC13
Cl
Br
Br

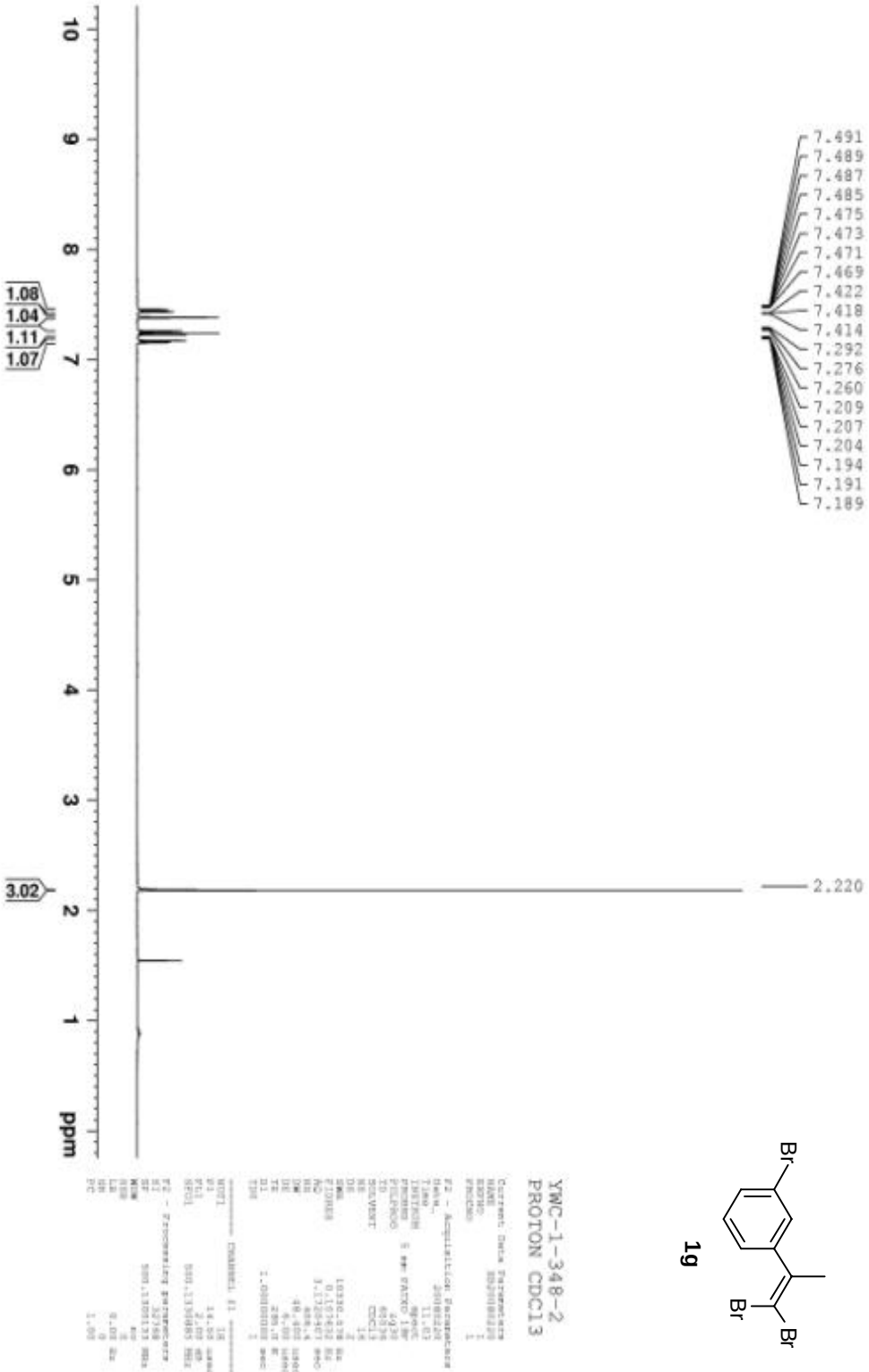
1e


7.509
7.504
7.500
7.491
7.487
7.482
7.125
7.121
7.117
7.108
7.104
7.099
2.183
1.98
1.97
3.00
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-333-2
PROTON CDC13
Br
Br
Br

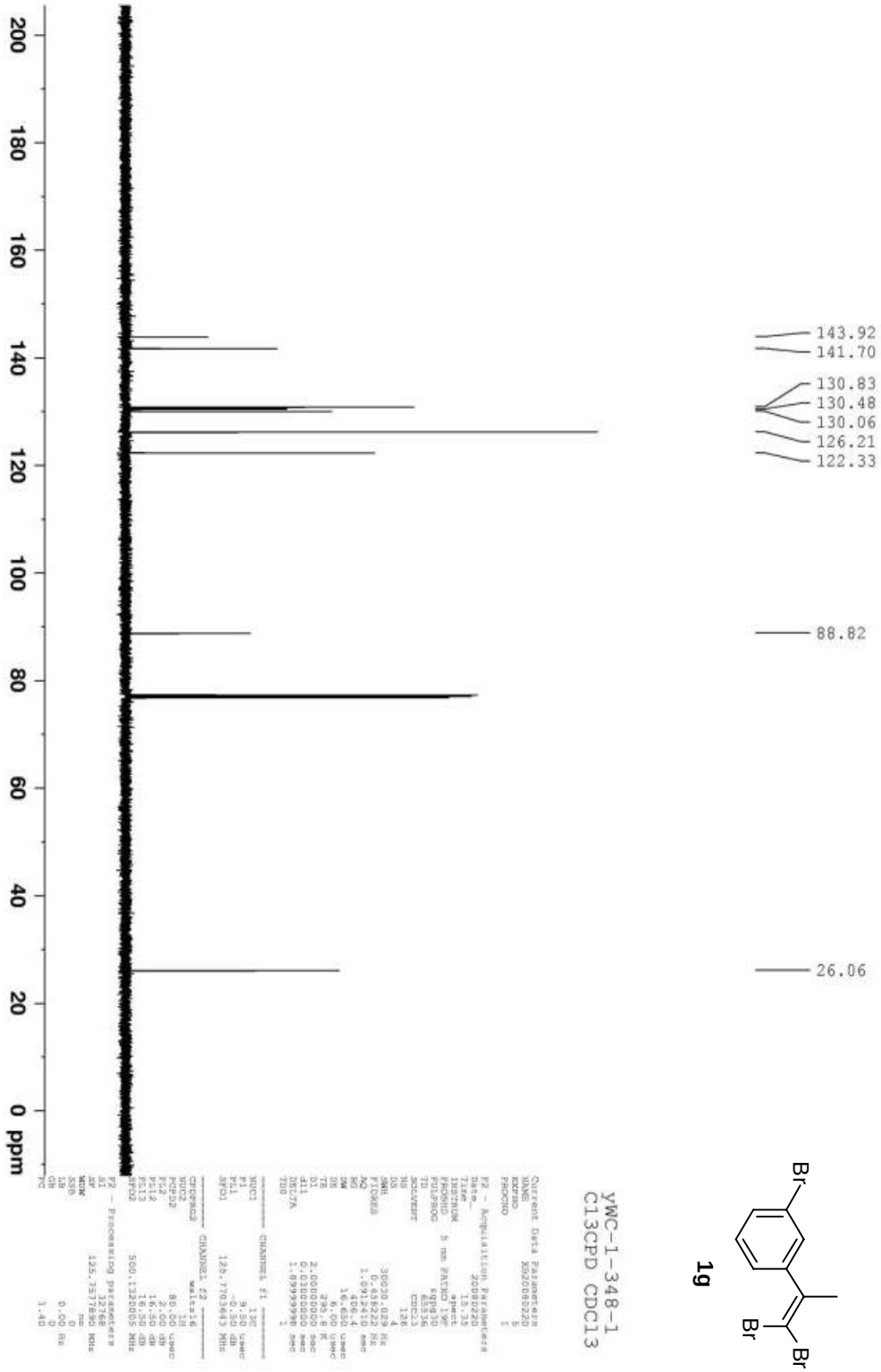
1f


142.07
140.94
131.76
129.33
121.93
88.42
26.13
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-333-2
C13CPD CDCl3
Br
Br
Br

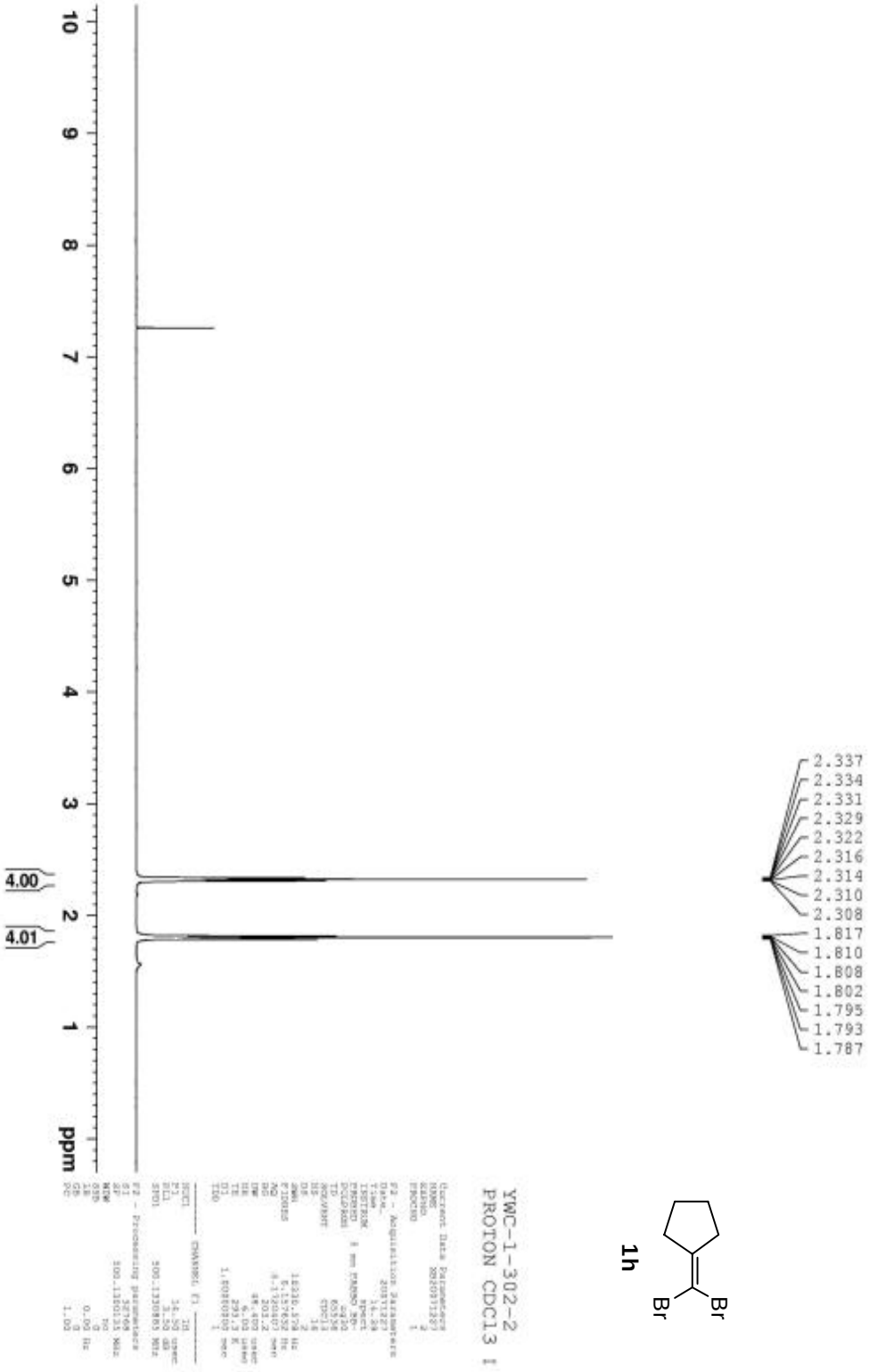
1f

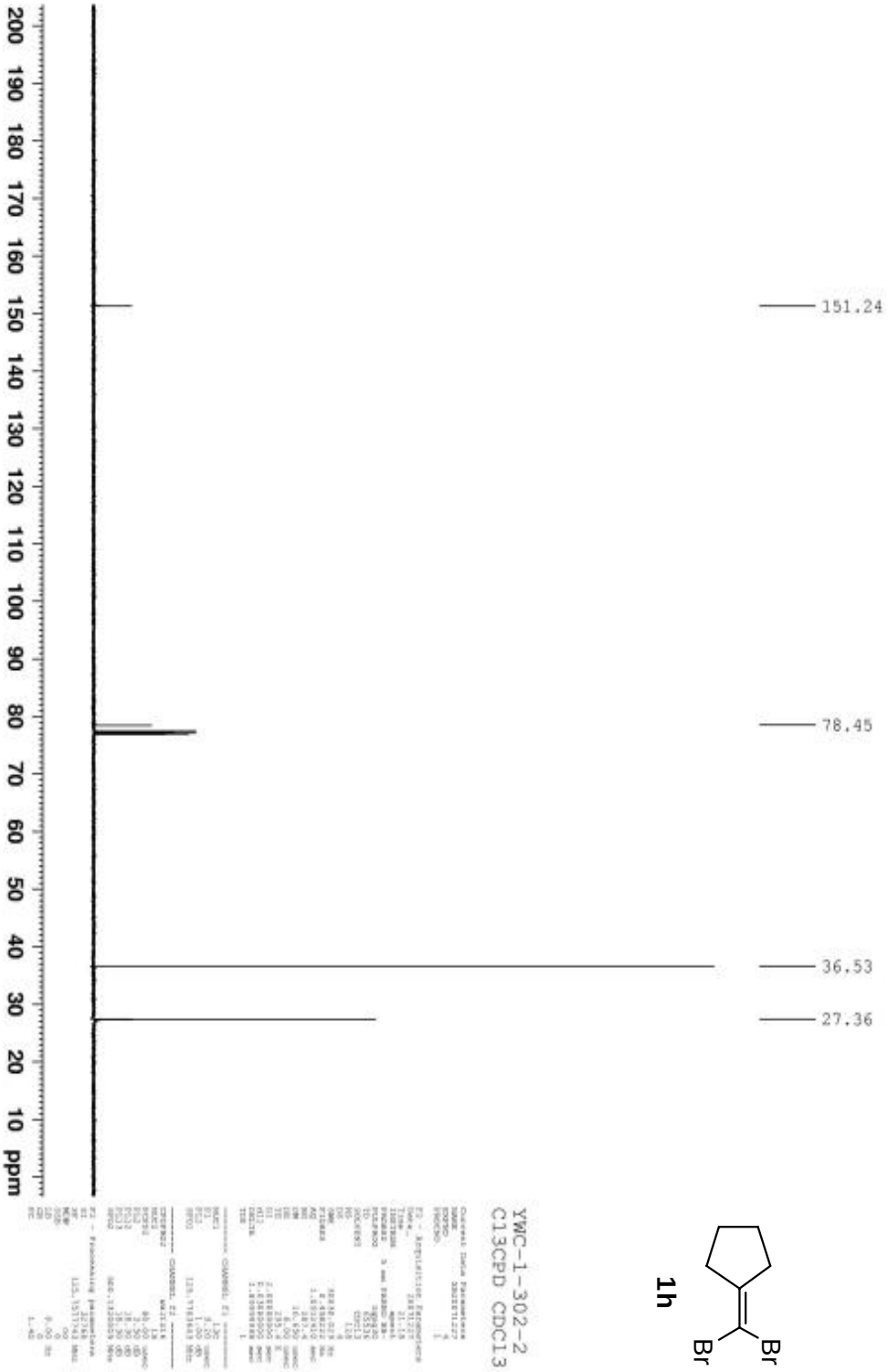

7.491
7.489
7.487
7.485
7.475
7.473
7.471
7.469
7.422
7.418
7.414
7.292
7.276
7.260
7.209
7.207
7.204
7.194
7.191
7.189
2.220
1.08
1.04
1.11
1.07
3.02
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-348-2
PROTON CDC13
Br
Br
Br

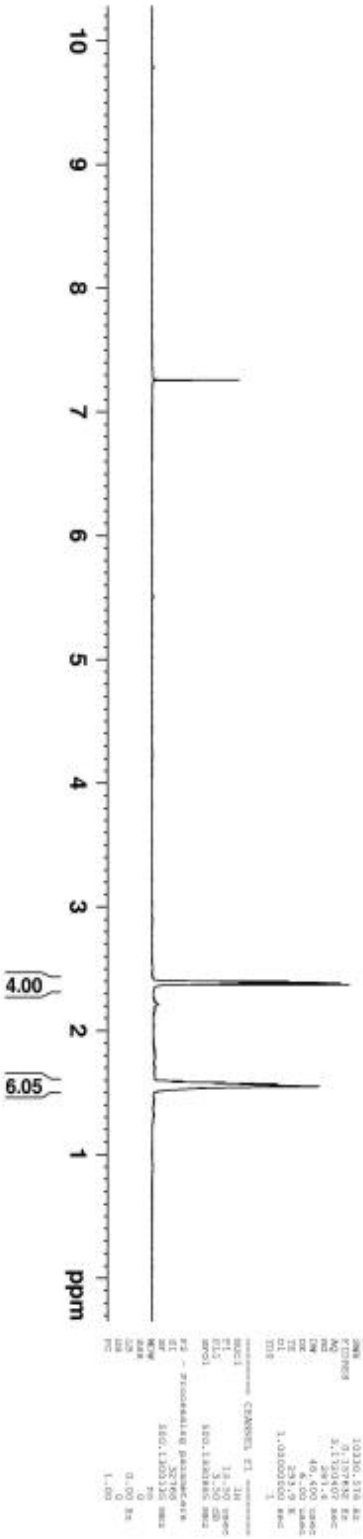
1g

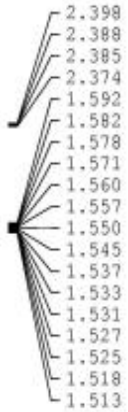

143.92
141.70
130.83
130.48
130.06
126.21
122.33
88.82
26.06
200
180
160
140
120
100
80
60
40
20
0 ppm
yWC-1-348-1
C13CPD CDC13
Br
Br
Br

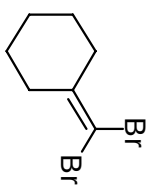
1g


4.00
4.01
ppm
2.337
2.334
2.331
2.329
2.322
2.316
2.314
2.310
2.308
1.817
1.810
1.808
1.802
1.795
1.793
1.787
Br
Br
1h

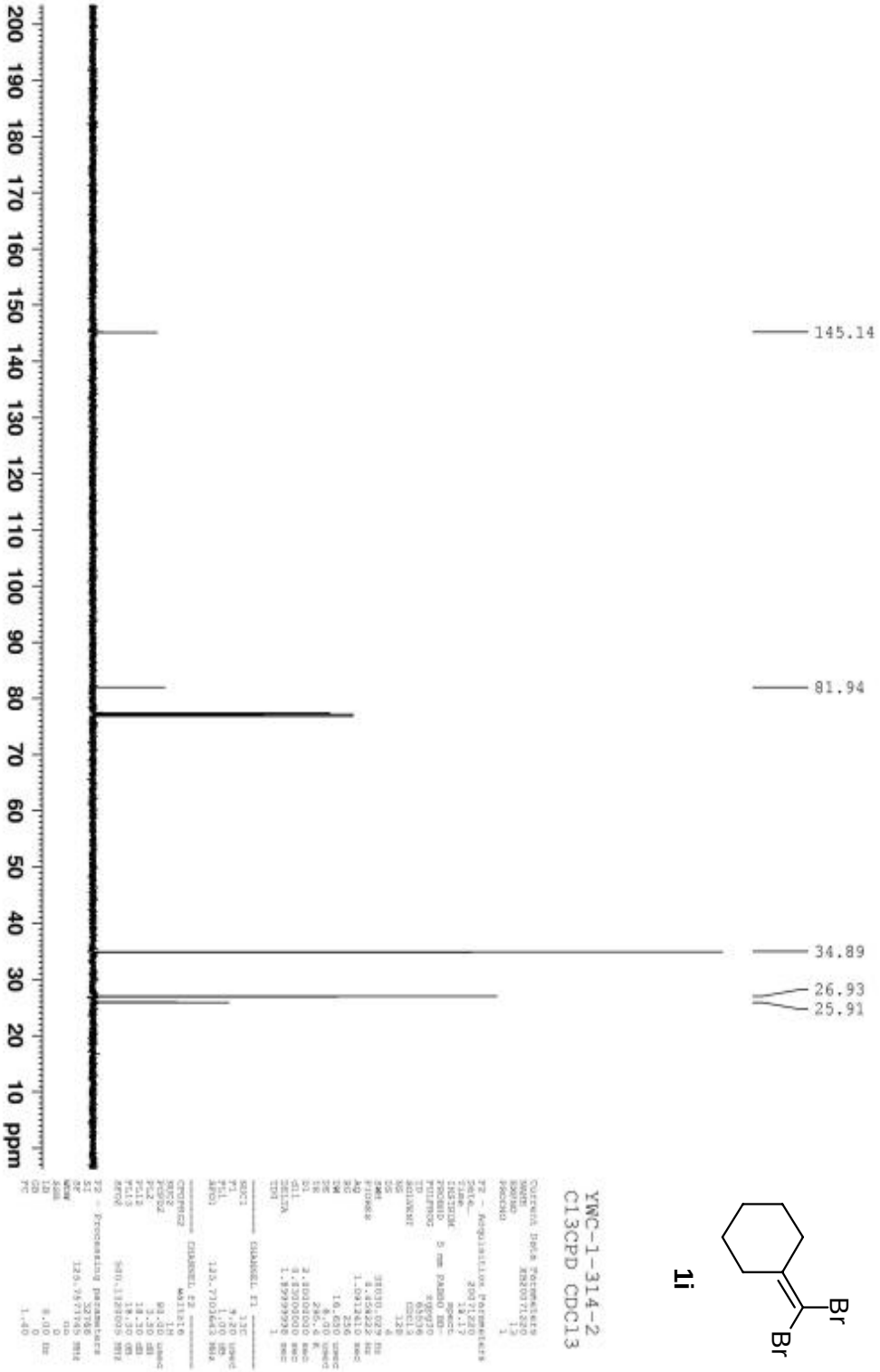

151.24
78.45
36.53
27.36
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-302-2
C13CPD CDC13
Br
Br
1h


4.00
6.05
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-314-2
PROTON CDCl3

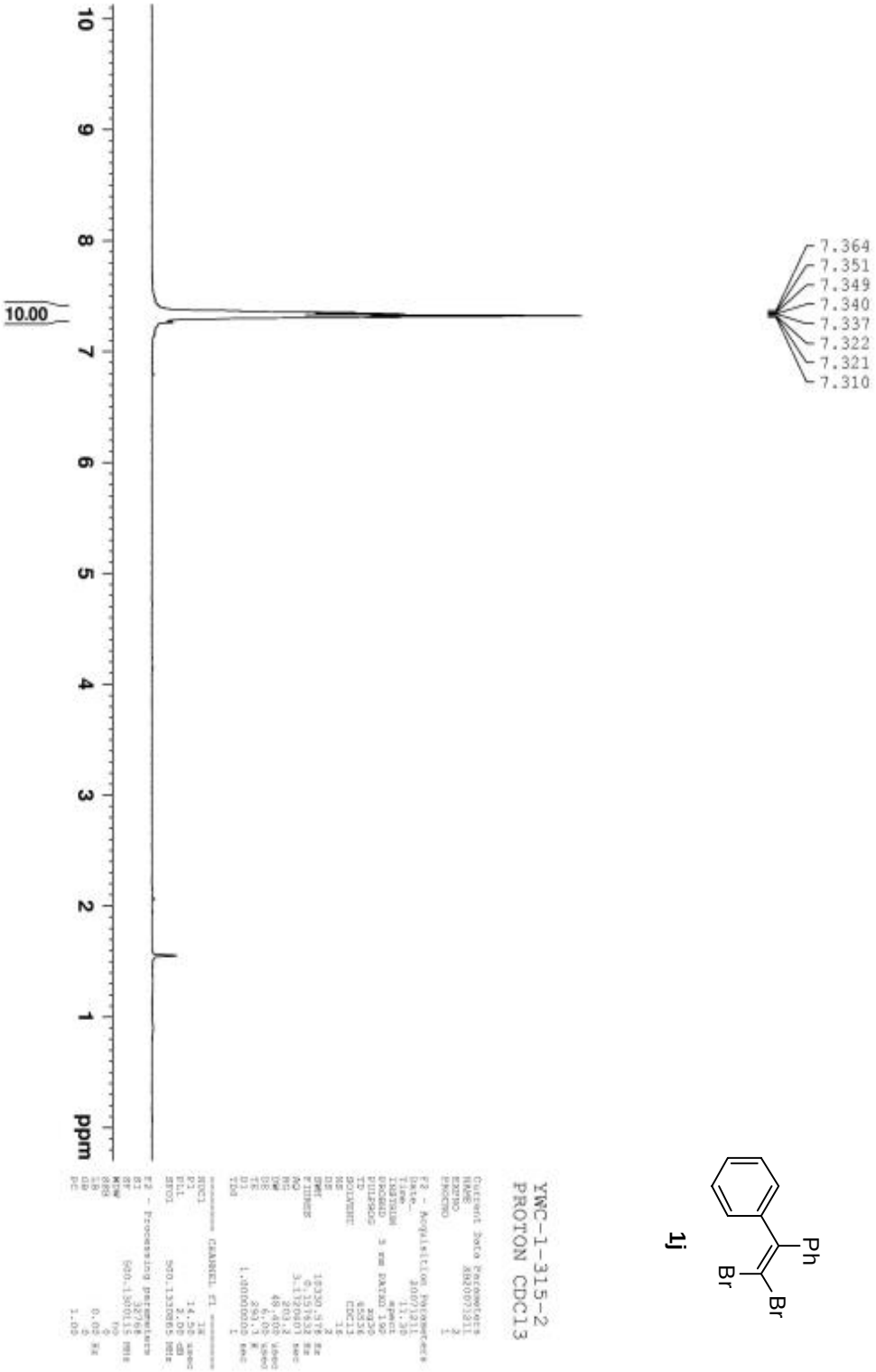

2.398
2.388
2.385
2.374
1.592
1.582
1.578
1.571
1.560
1.557
1.550
1.545
1.537
1.533
1.531
1.527
1.525
1.518
1.513


Br
Br

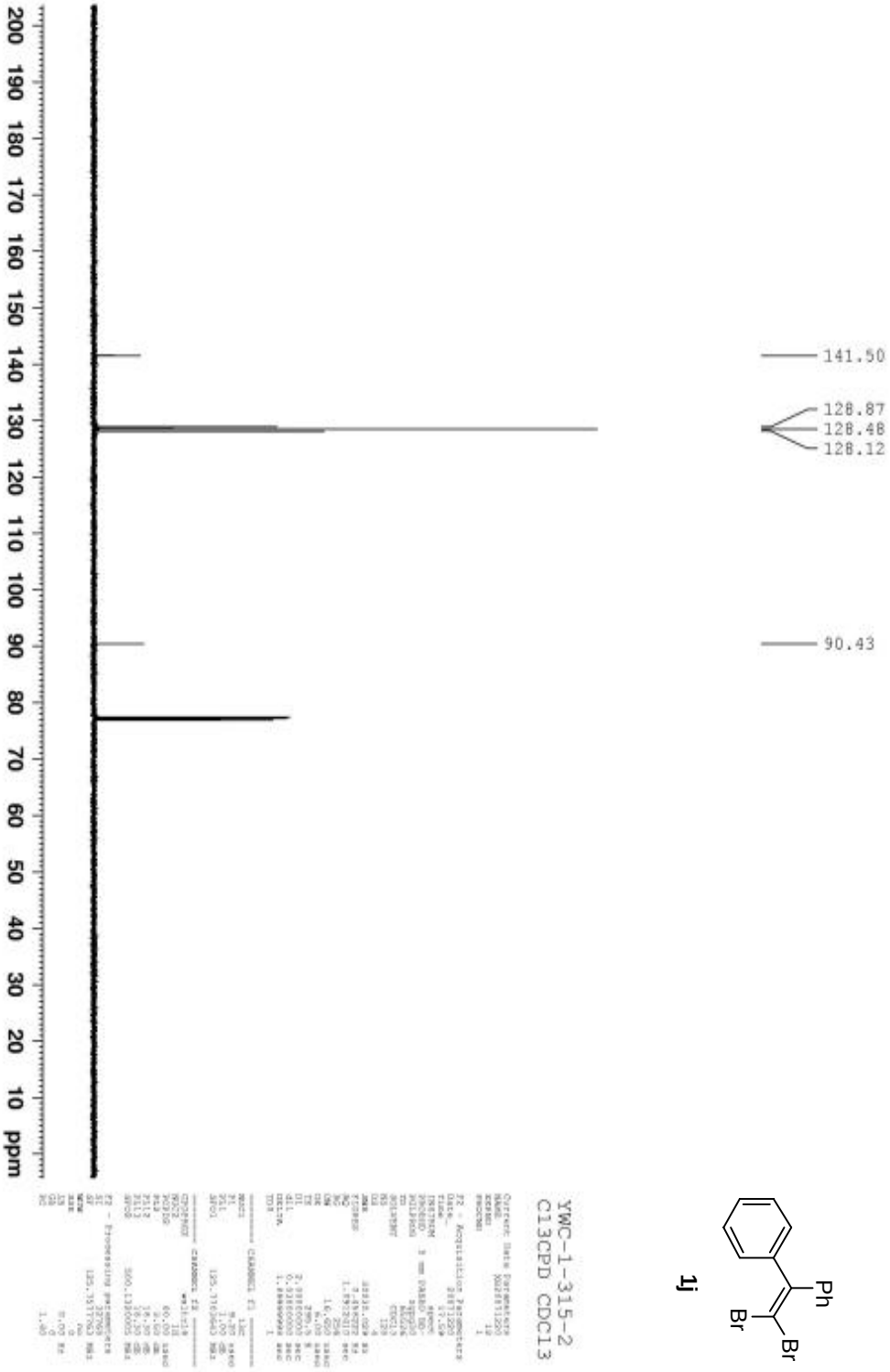
1i


145.14
81.94
34.89
26.93
25.91
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-314-2
C13CPD CDCl3
Br
Br

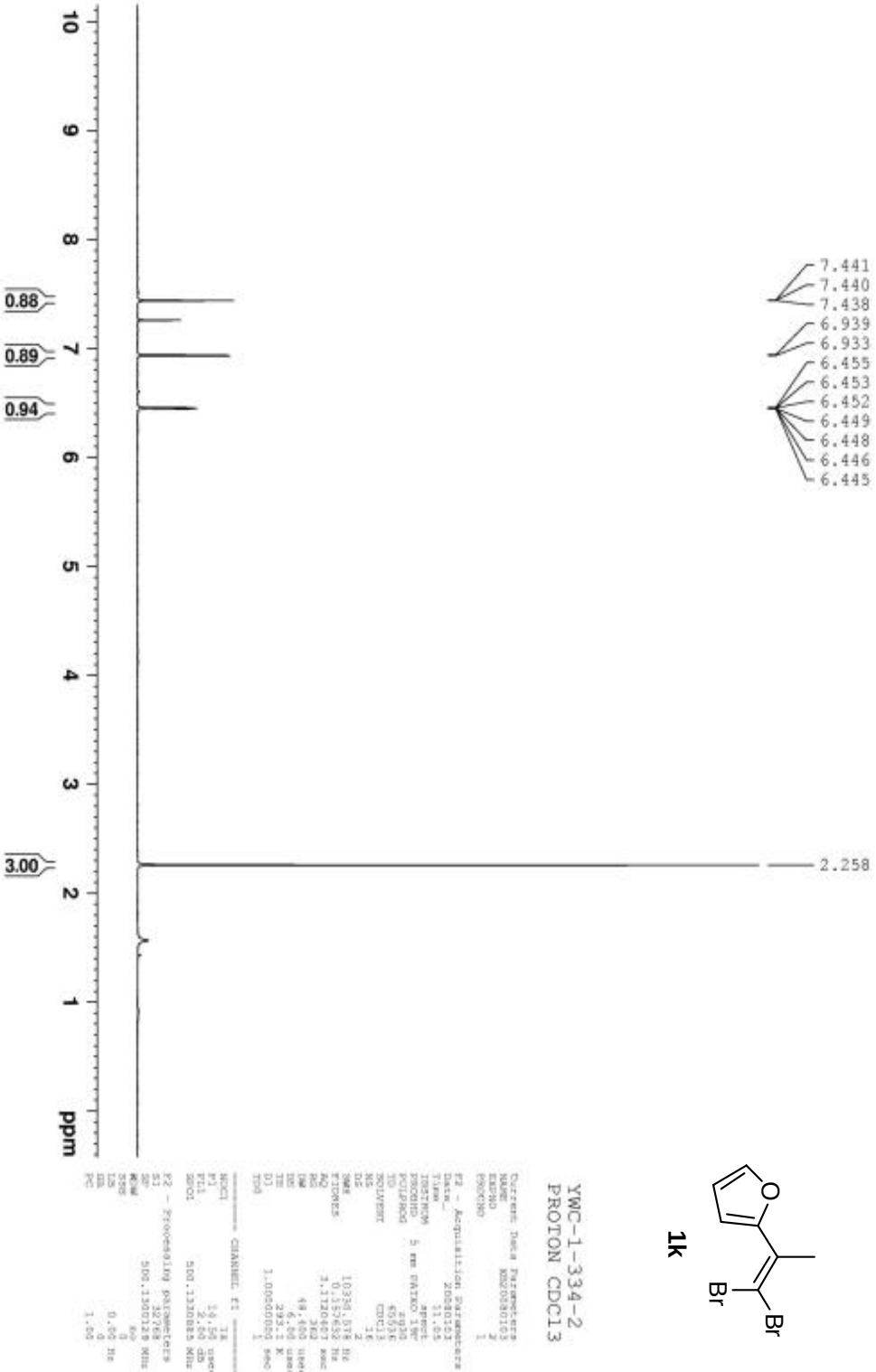
1i

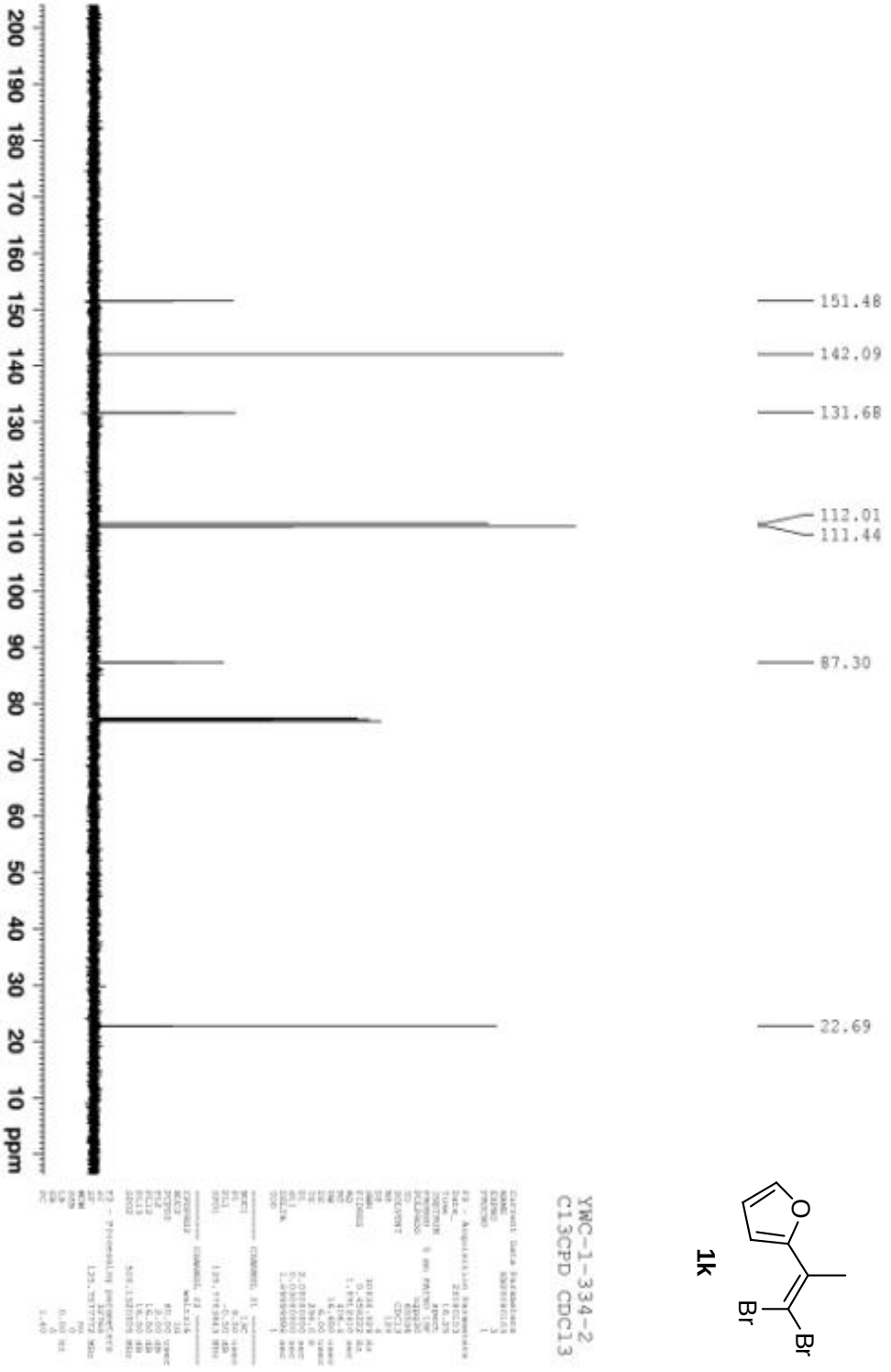

10.00
ppm
YWC-1-315-2
PROTON CDC13
7.364
7.351
7.349
7.340
7.337
7.322
7.321
7.310
Ph
Br
Br

1j

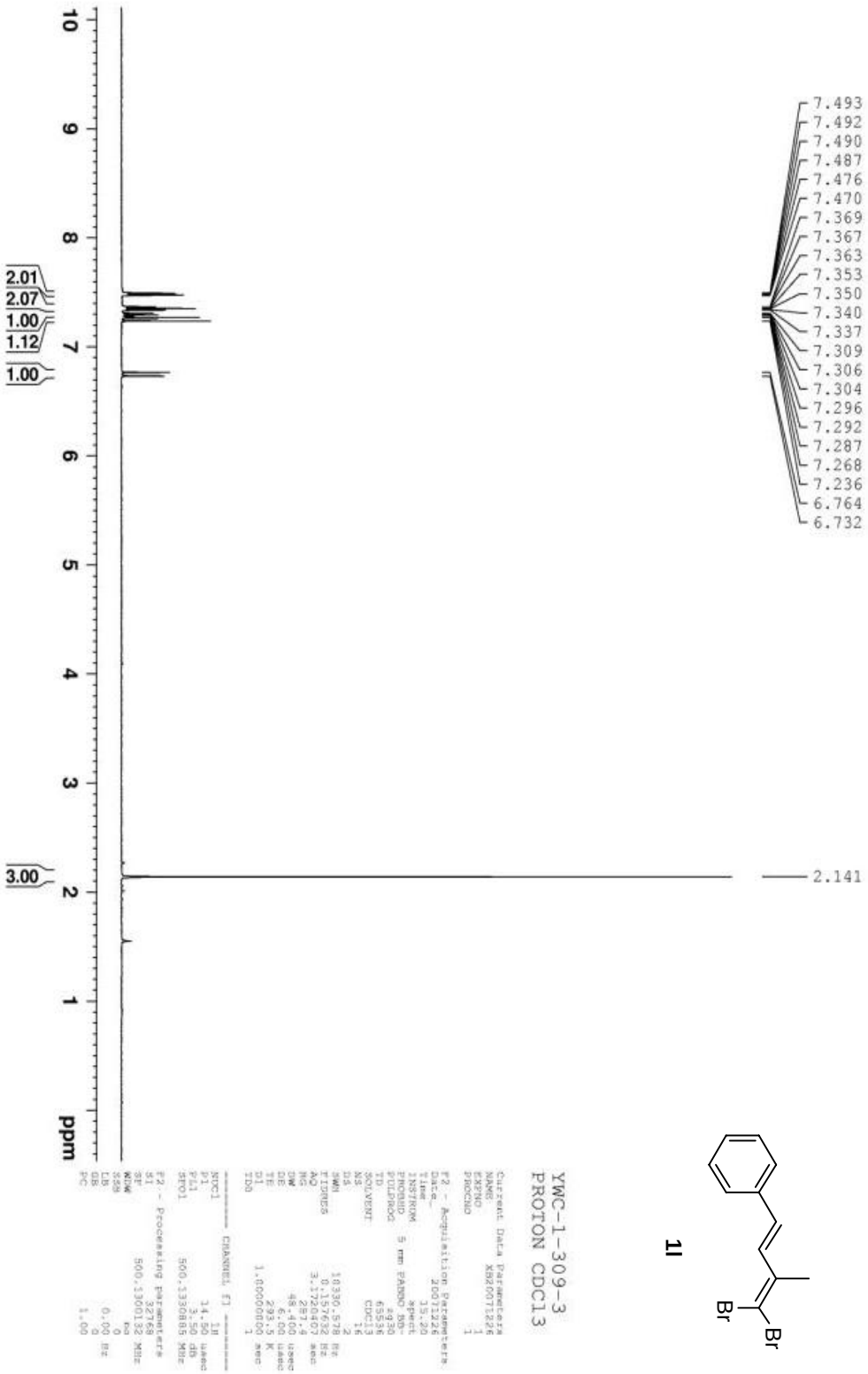

141.50
128.87
128.48
128.12
90.43
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-315-2
C13CPD CDCl3
Ph
Br
Br

1j

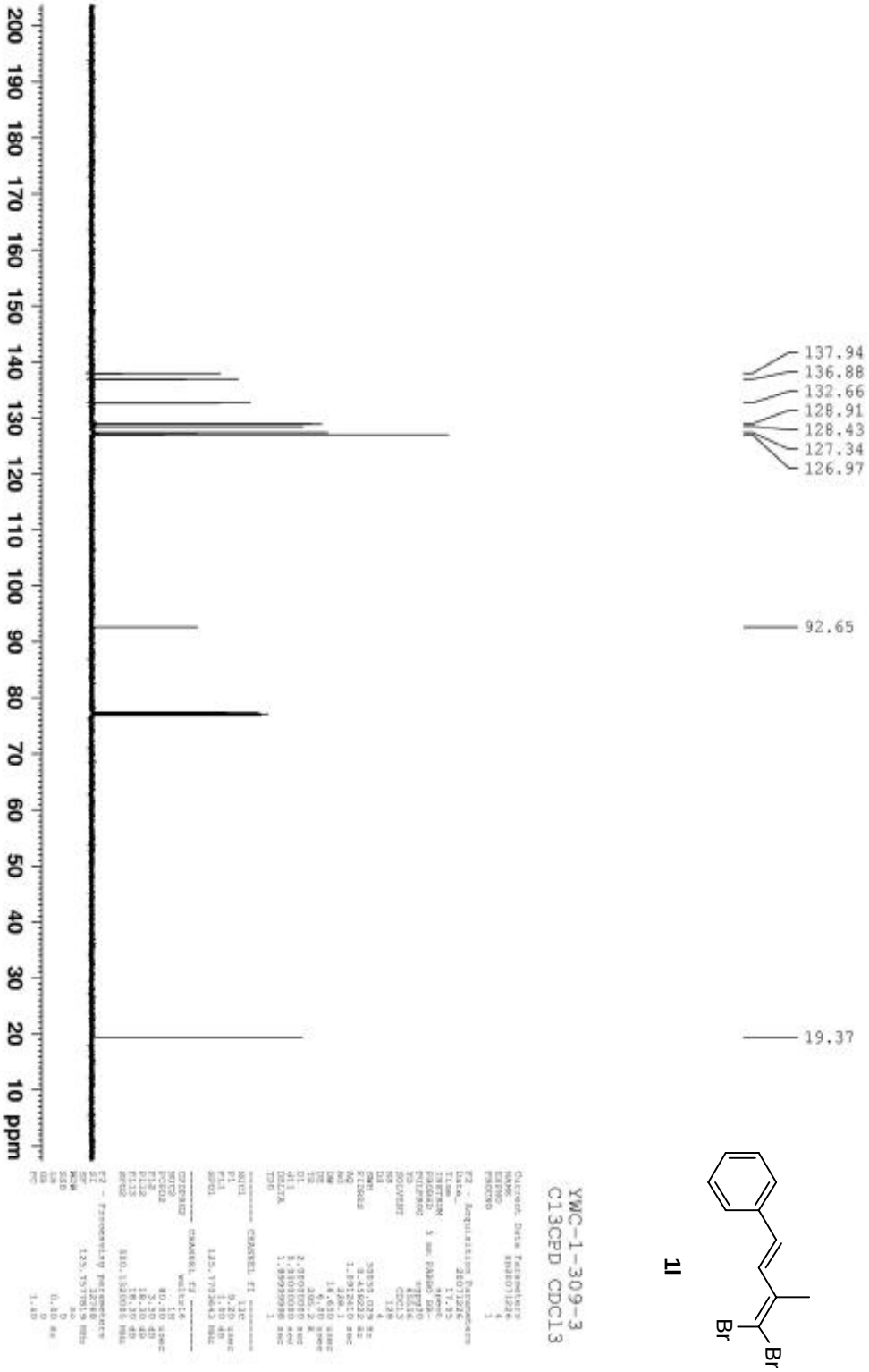

7.441
7.440
7.438
6.939
6.933
6.455
6.453
6.452
6.449
6.448
6.446
6.445
2.258
0.88
0.89
0.94
3.00
ppm
YWC-1-334-2
PROTON CDC13
1k
O
Br
Br


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
151.48
142.09
131.68
112.01
111.44
87.30
22.69
YWC-1-334-2
C13CPD CDCl3
O
Br
Br

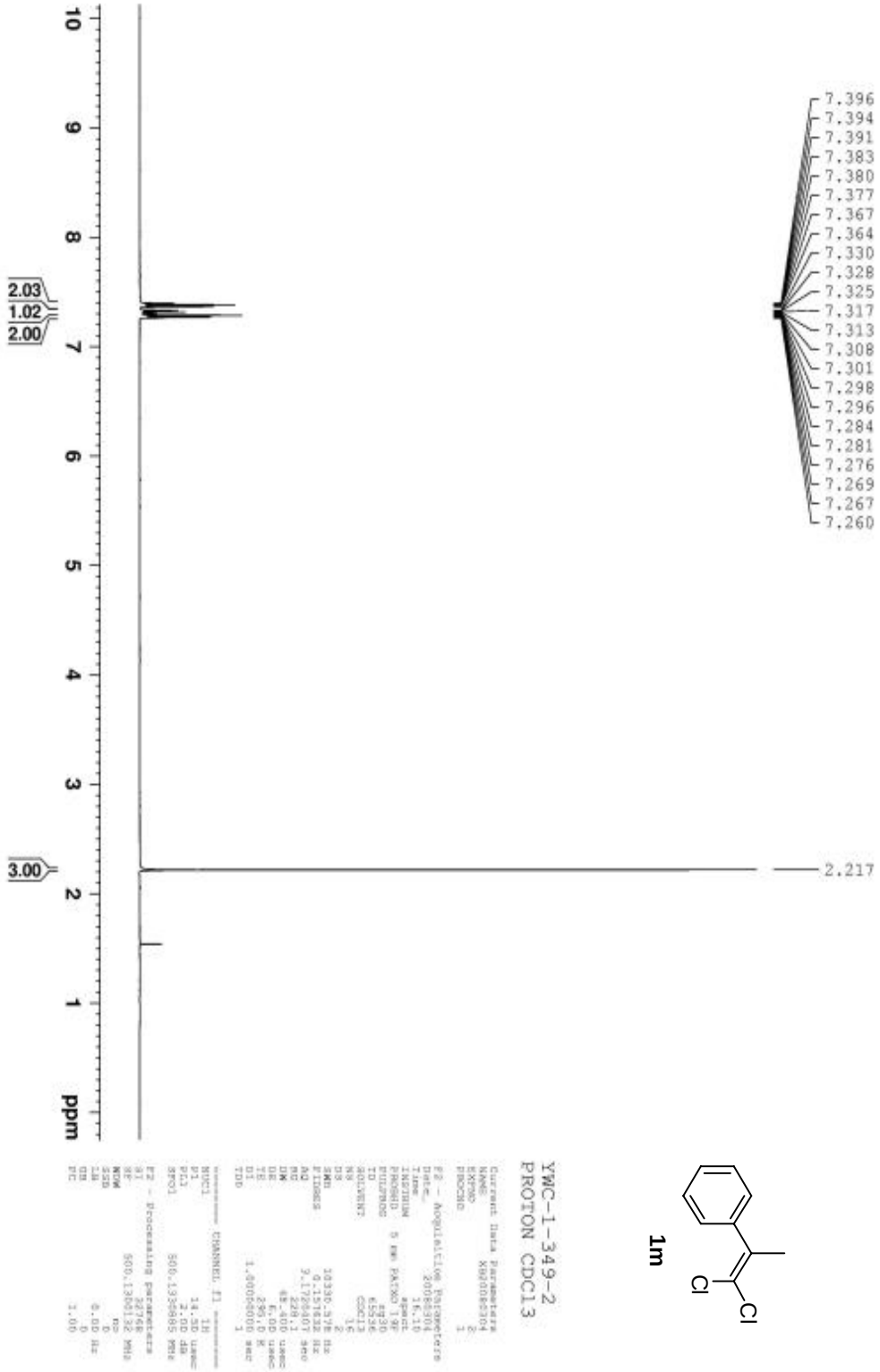
1k

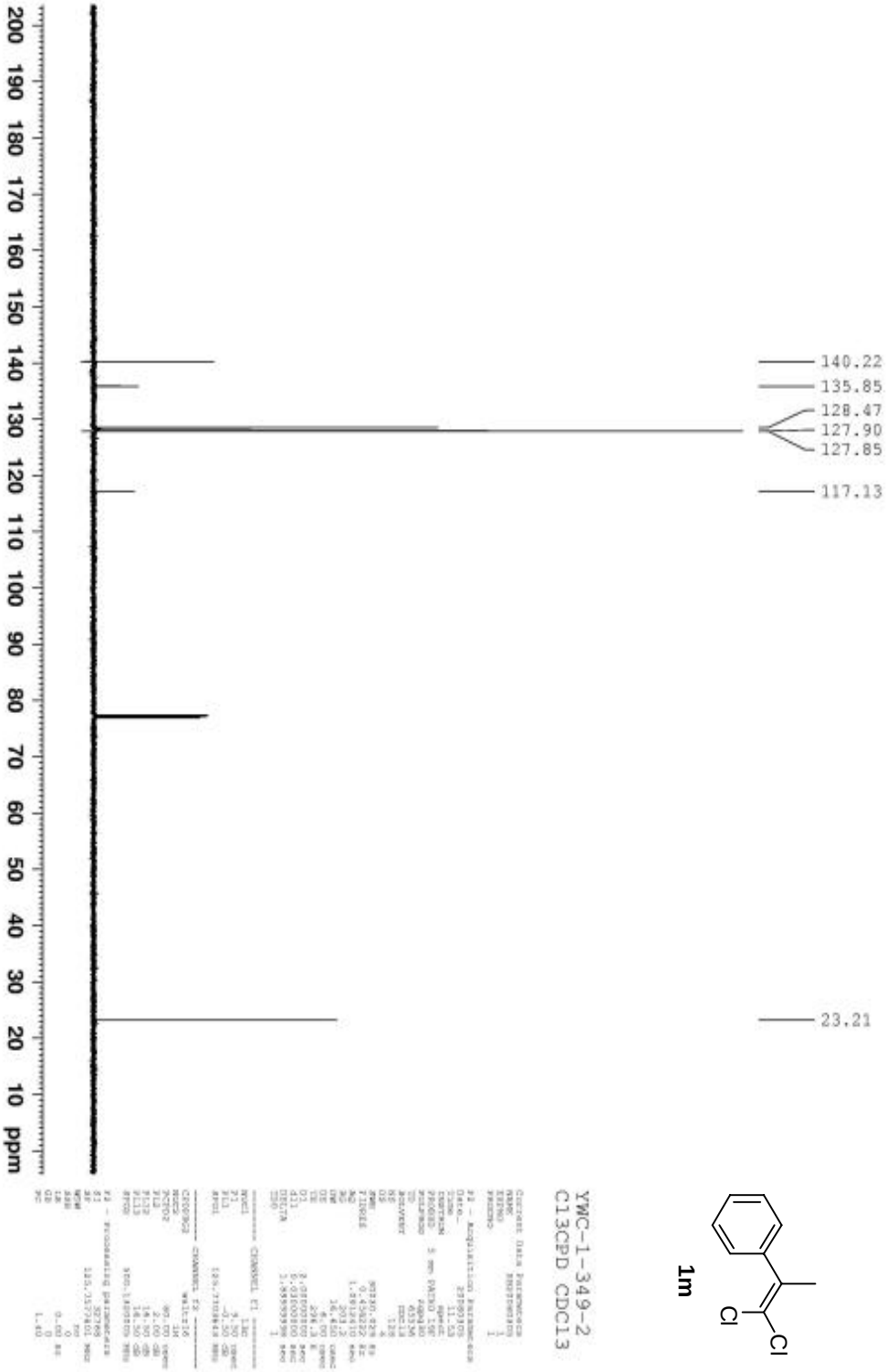

7.493
7.492
7.490
7.487
7.476
7.470
7.369
7.367
7.363
7.353
7.350
7.340
7.337
7.309
7.306
7.304
7.296
7.292
7.287
7.268
7.236
6.764
6.732
2.141
2.01
2.07
1.00
1.12
1.00
3.00
ppm
YWC-1-309-3
PROTON CDCl3
Br
Br

1l

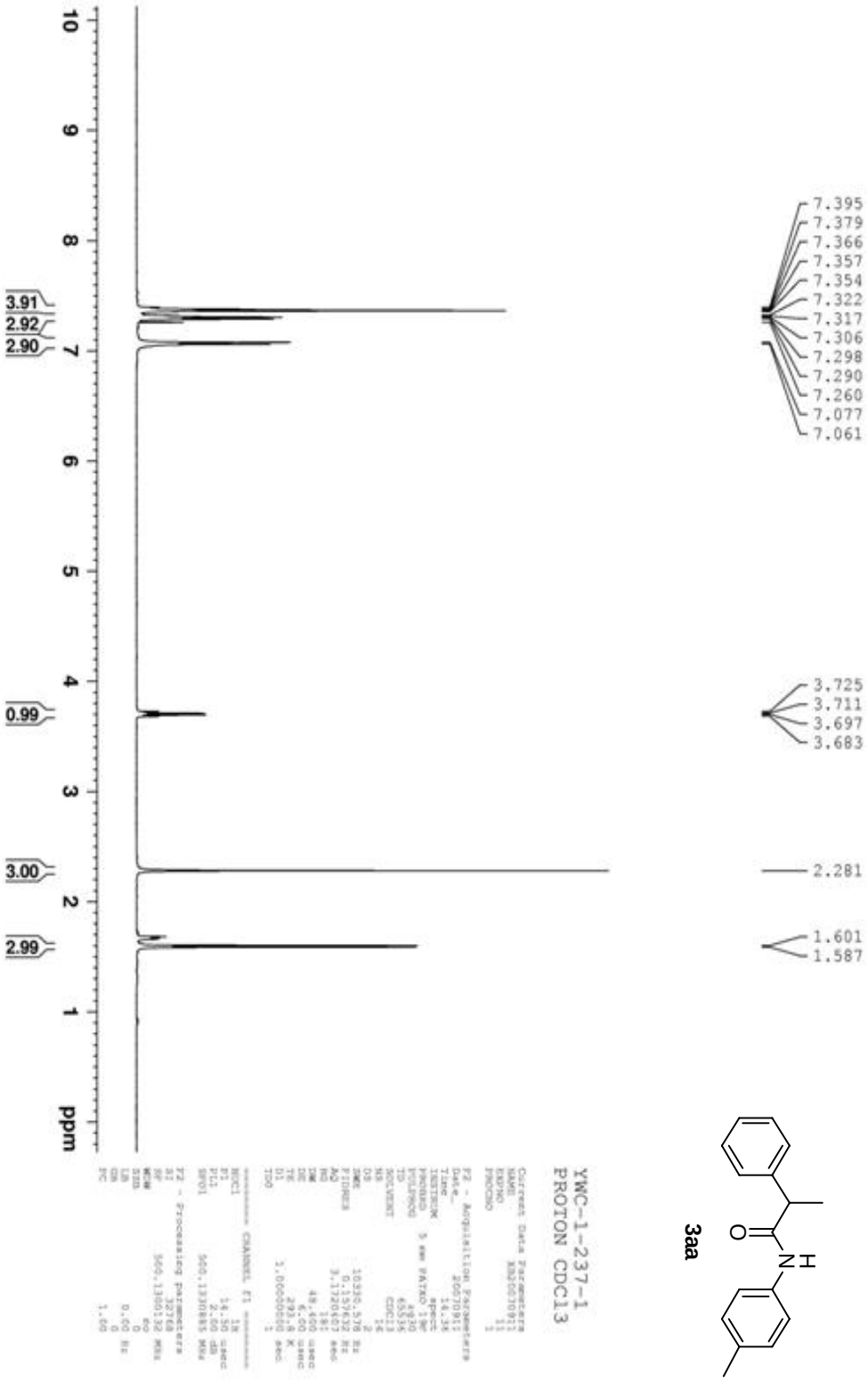

137.94
136.88
132.66
128.91
128.43
127.34
126.97
92.65
19.37
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-309-3
C13CPD CDCl3
Br
Br

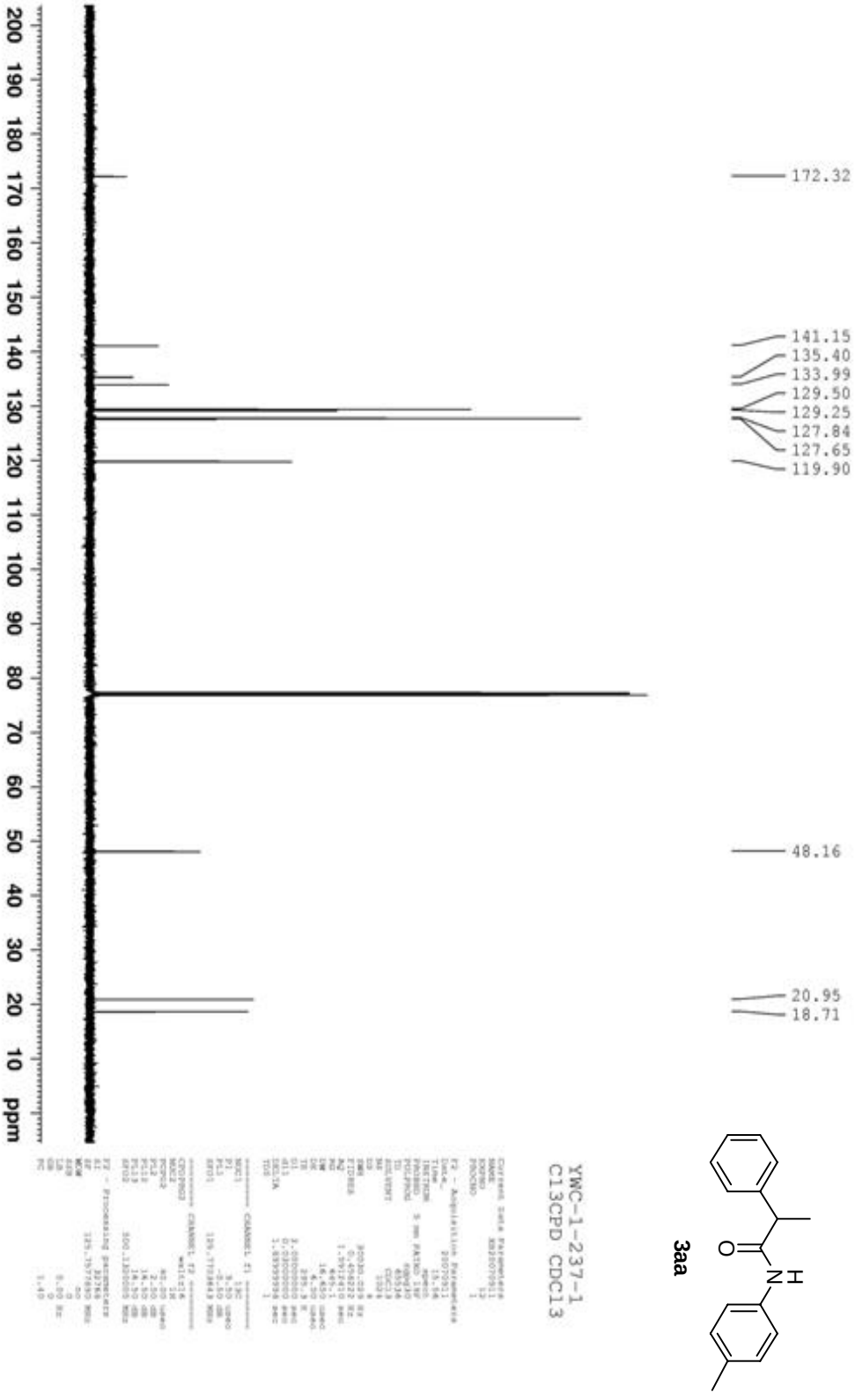
1l

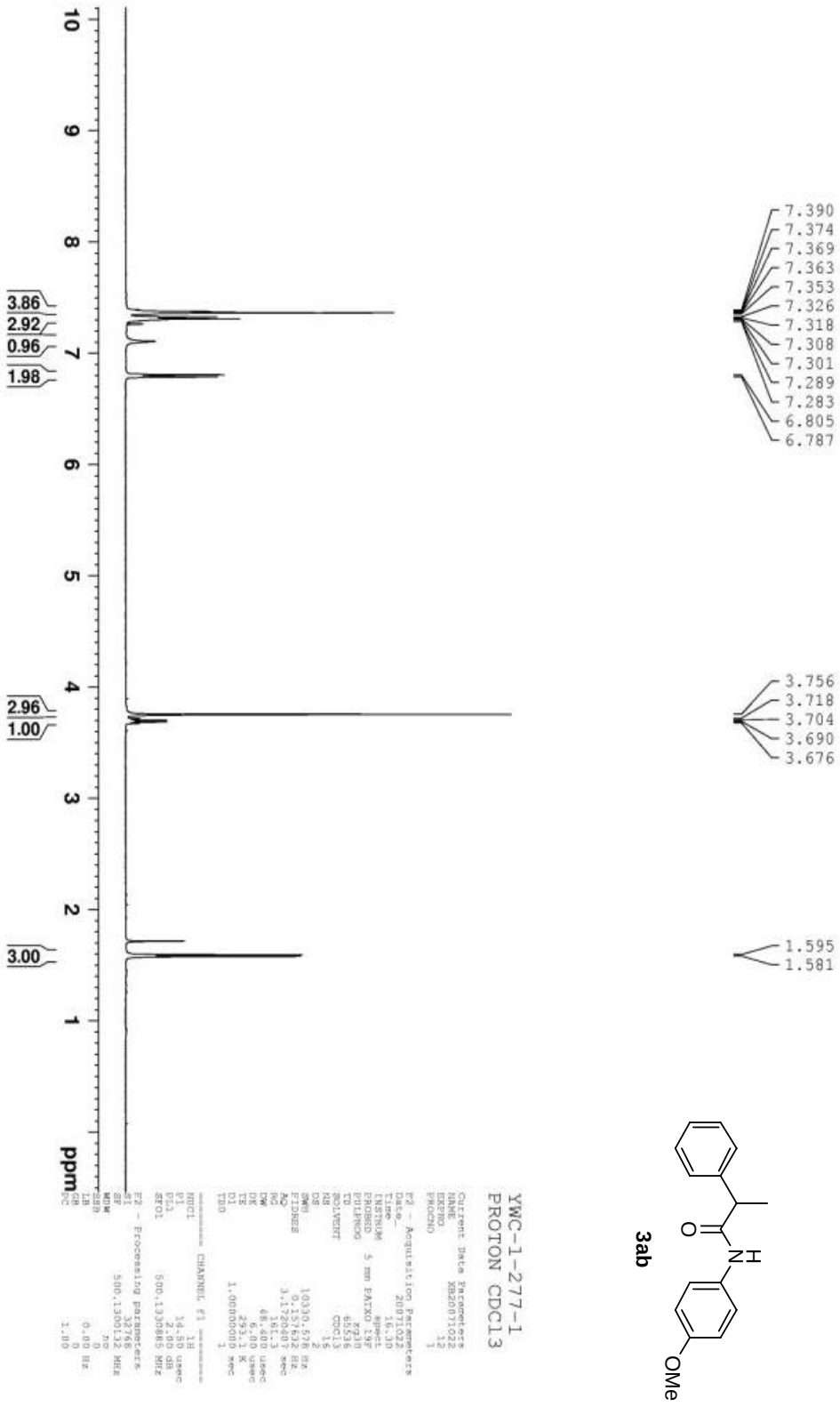

7.396
7.394
7.391
7.383
7.380
7.377
7.367
7.364
7.330
7.328
7.325
7.317
7.313
7.308
7.301
7.298
7.296
7.284
7.281
7.276
7.269
7.267
7.260
2.217
2.03
1.02
2.00
3.00
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-349-2
PROTON CDCl3
1m
Cl
Cl

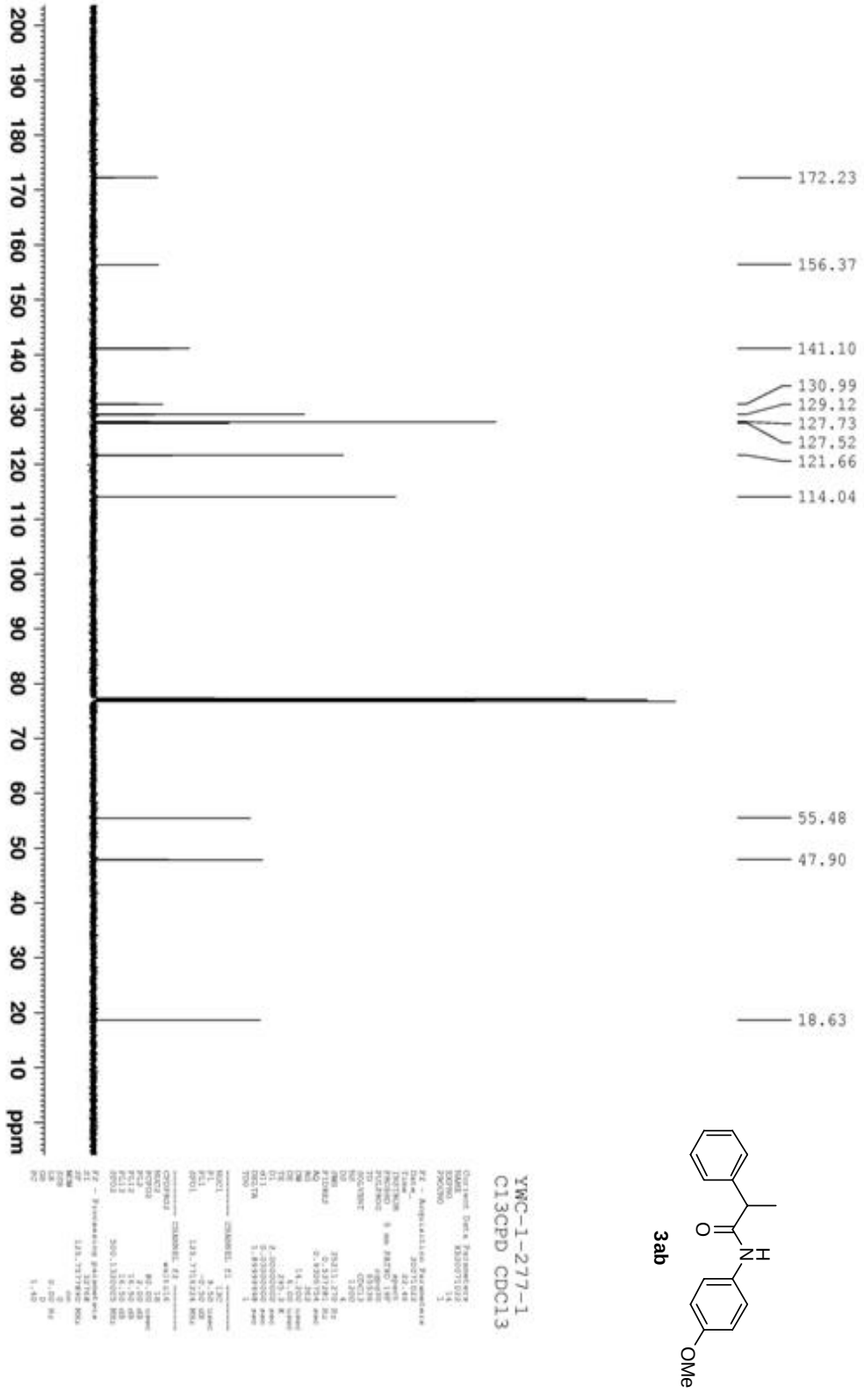

140.22
135.85
128.47
127.90
127.85
117.13
23.21
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-349-2
C13CPD CDCl3
Cl
Cl

1m

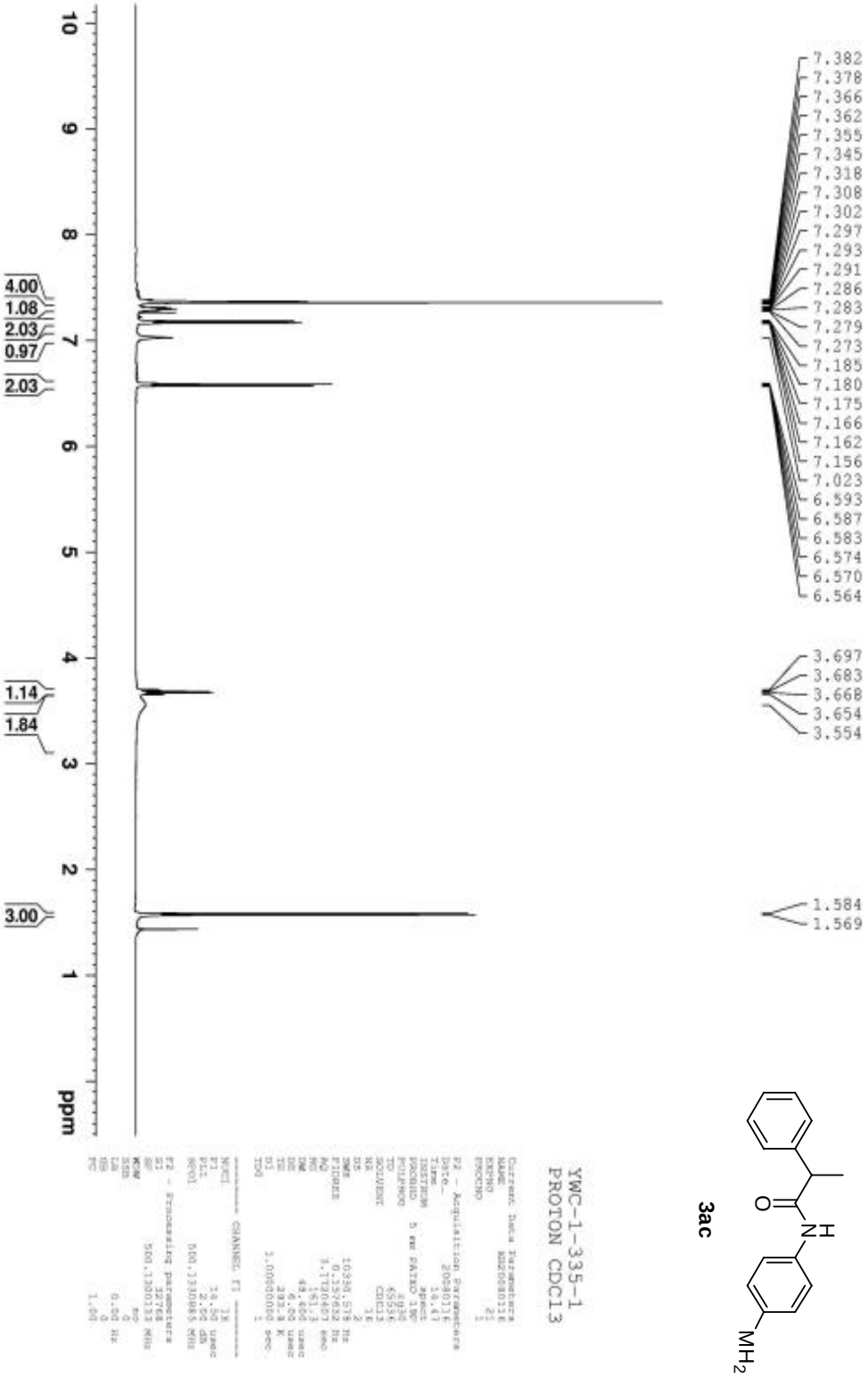

7.395
7.379
7.366
7.357
7.354
7.322
7.317
7.306
7.298
7.290
7.260
7.077
7.061
3.725
3.711
3.697
3.683
2.281
1.601
1.587
3.91
2.92
2.90
0.99
3.00
2.99
ppm
YWC-1-237-1
PROTON CDCl3
3aa

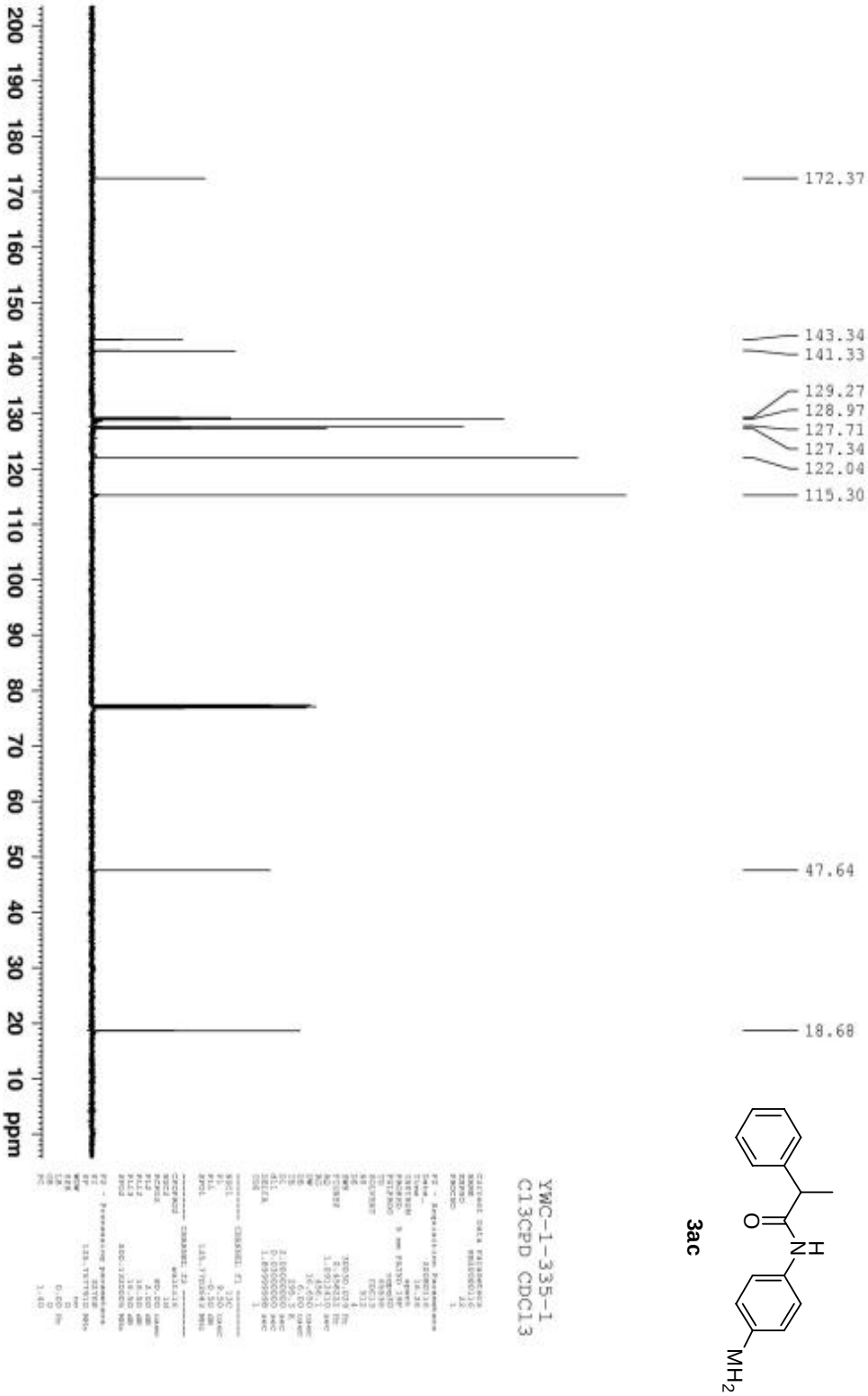

172.32
141.15
135.40
133.99
129.50
129.25
127.84
127.65
119.90
48.16
20.95
18.71
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-237-1
C13CPD CDCl3
3aa

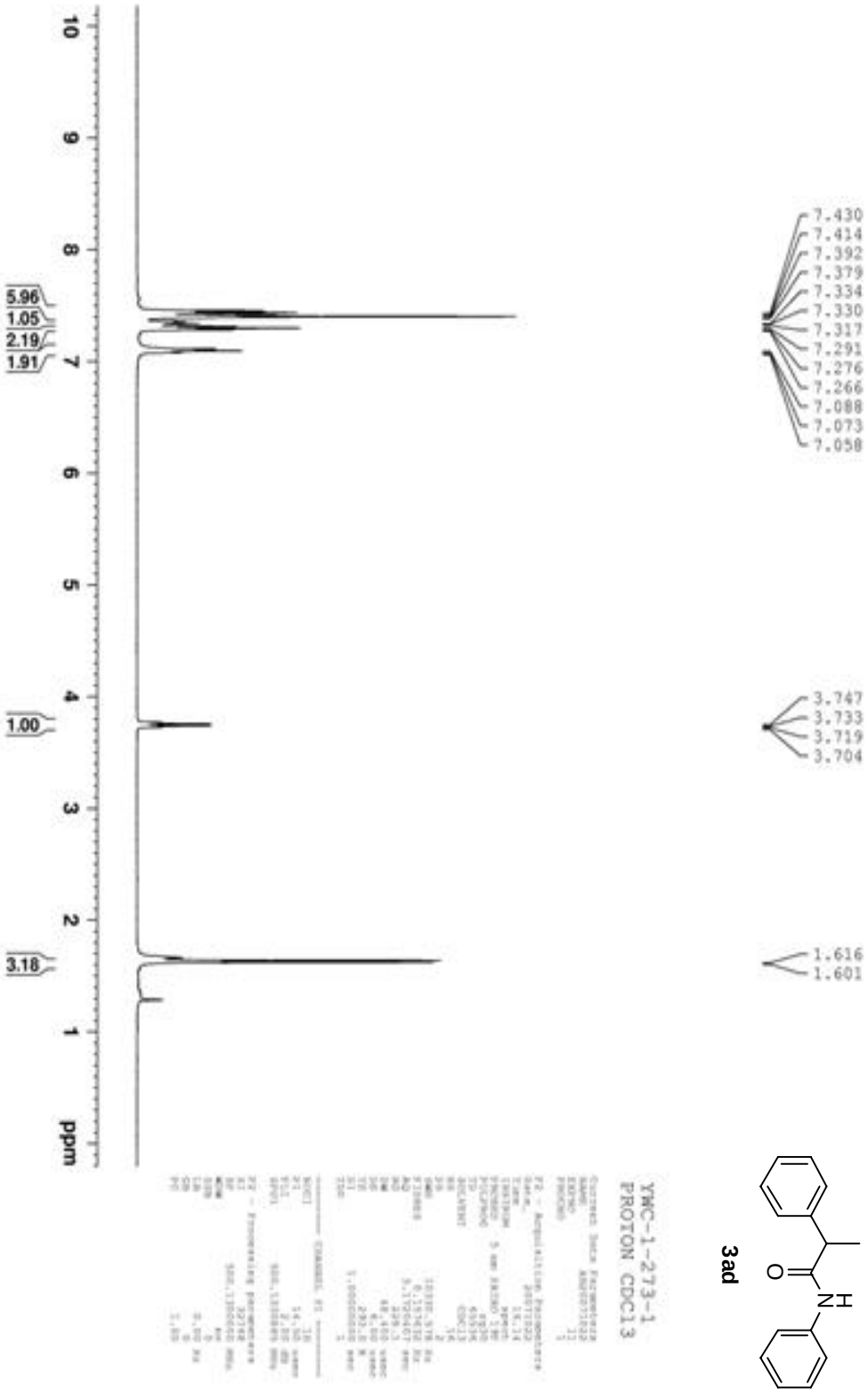

7.390
7.374
7.369
7.363
7.353
7.326
7.318
7.308
7.301
7.289
7.283
6.805
6.787
3.756
3.718
3.704
3.690
3.676
1.595
1.581
3.86
2.92
0.96
1.98
2.96
1.00
3.00
ppm
YWC-1-277-1
PROTON CDC13
3ab
O
H
N
OMe

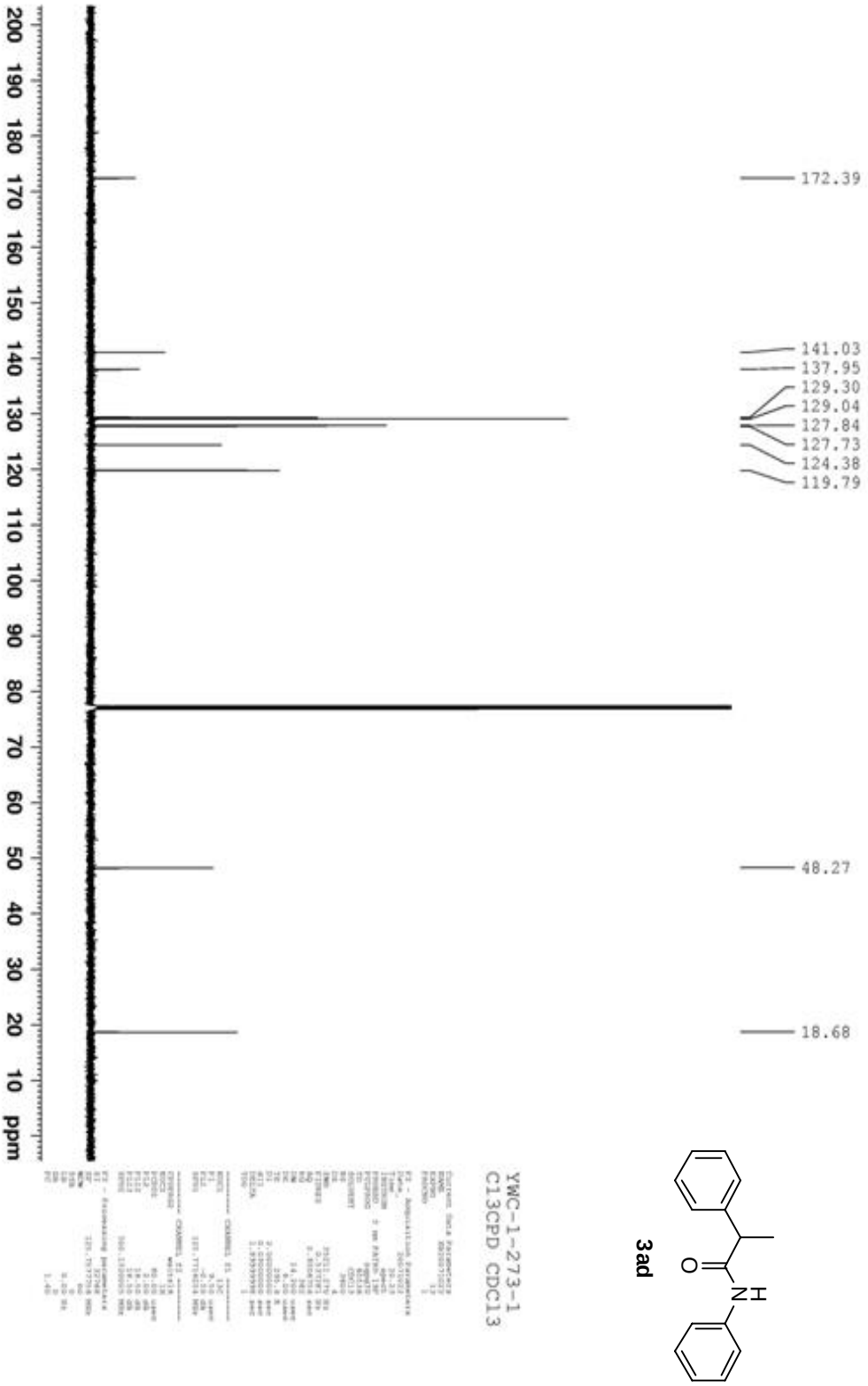

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
172.23
156.37
141.10
130.99
129.12
127.73
127.52
121.66
114.04
55.48
47.90
18.63
YWC-1-277-1
C13CPD CDCl3
O
H
N
OMe

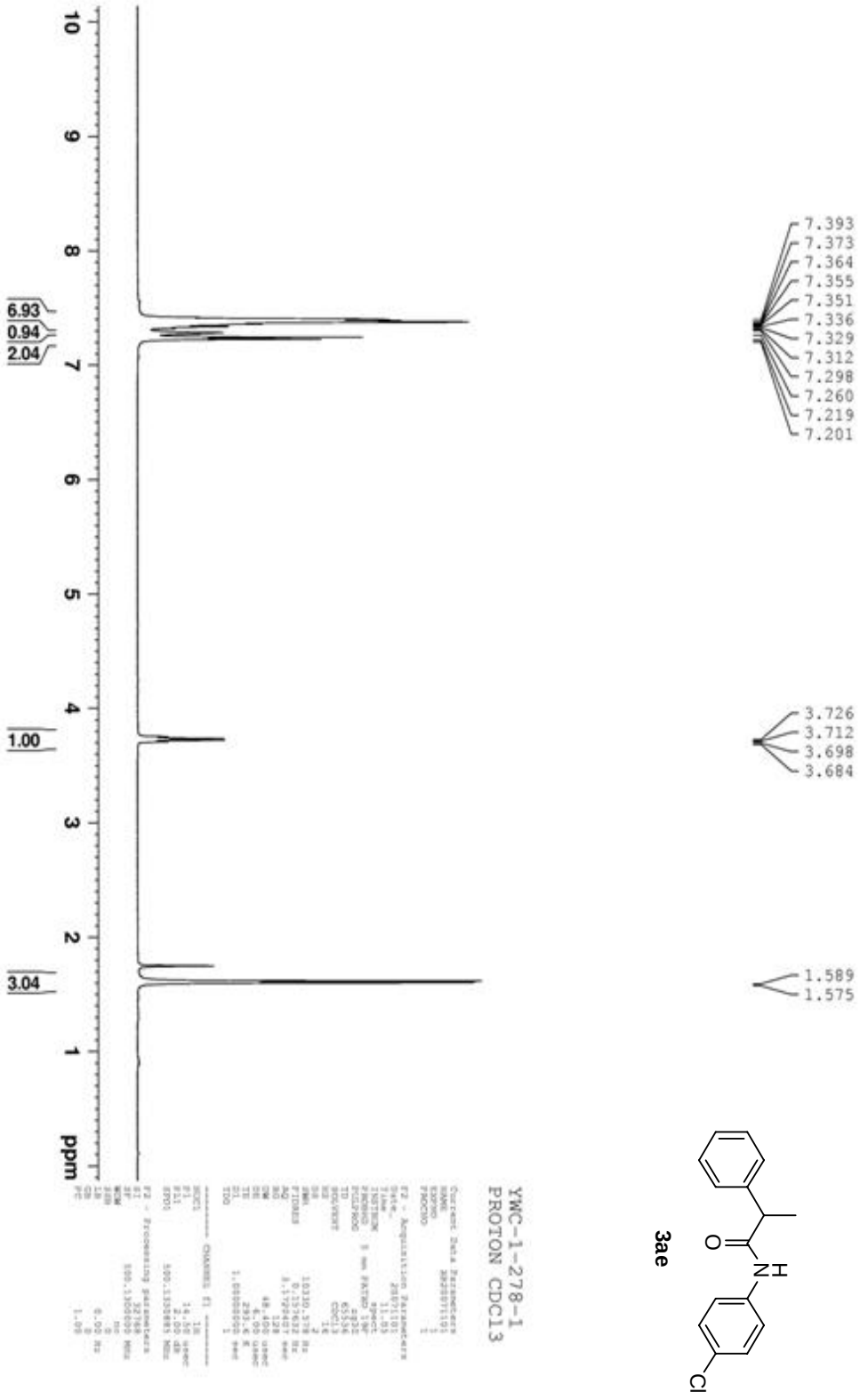
3ab

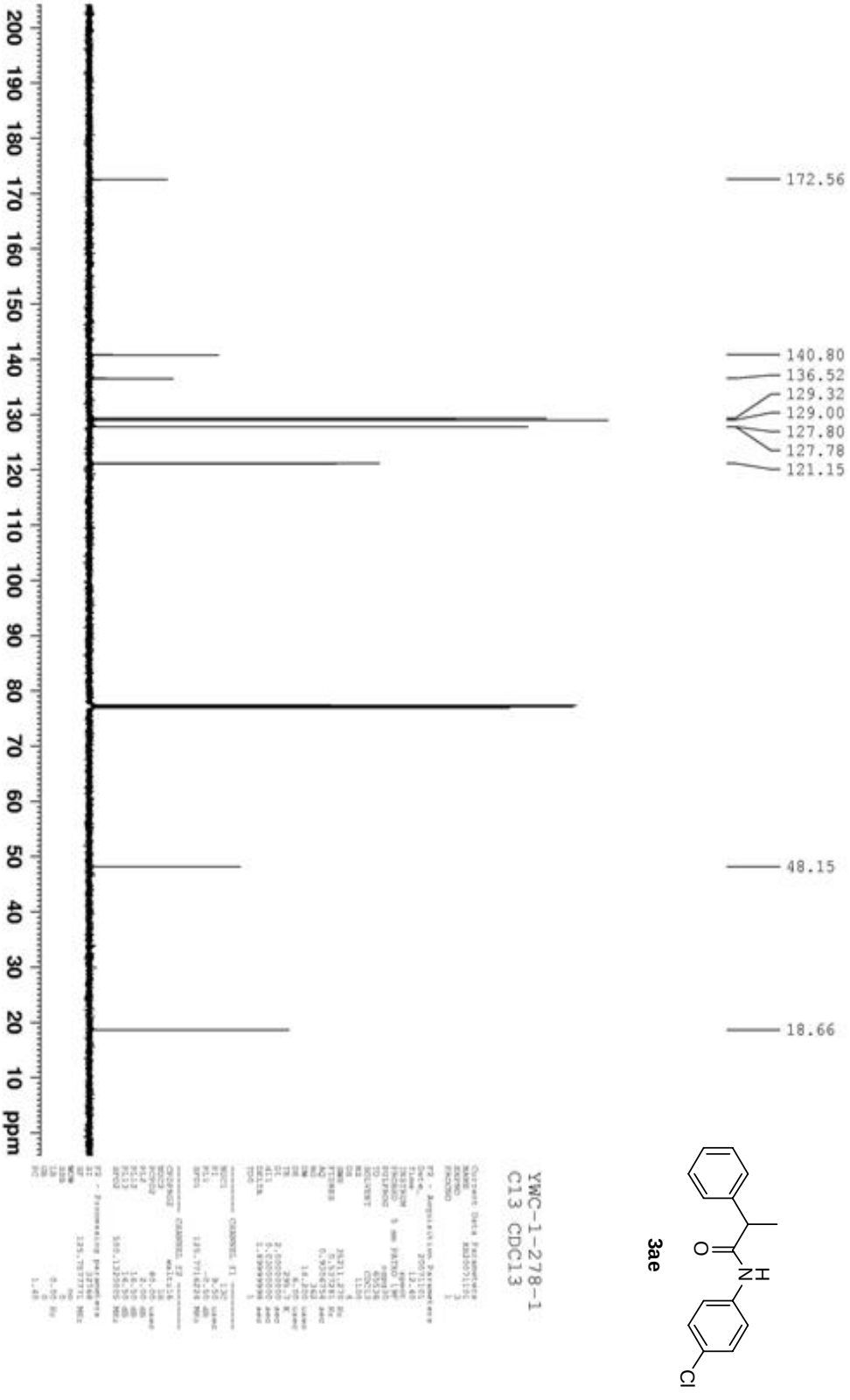

7.382
7.378
7.366
7.362
7.355
7.345
7.318
7.308
7.302
7.297
7.293
7.291
7.286
7.283
7.279
7.273
7.185
7.180
7.175
7.166
7.162
7.156
7.023
6.593
6.587
6.583
6.574
6.570
6.564
3.697
3.683
3.668
3.654
3.554
1.584
1.569
4.00
1.08
2.03
0.97
2.03
1.14
1.84
3.00
ppm
YWC-1-335-1
PROTON CDC13
3ac
O
N
H
NH2


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
172.37
143.34
141.33
129.27
128.97
127.71
127.34
122.04
115.30
47.64
18.68
YWC-1-335-1
C13CPD CDCl3
3ac
O
N
H
NH2

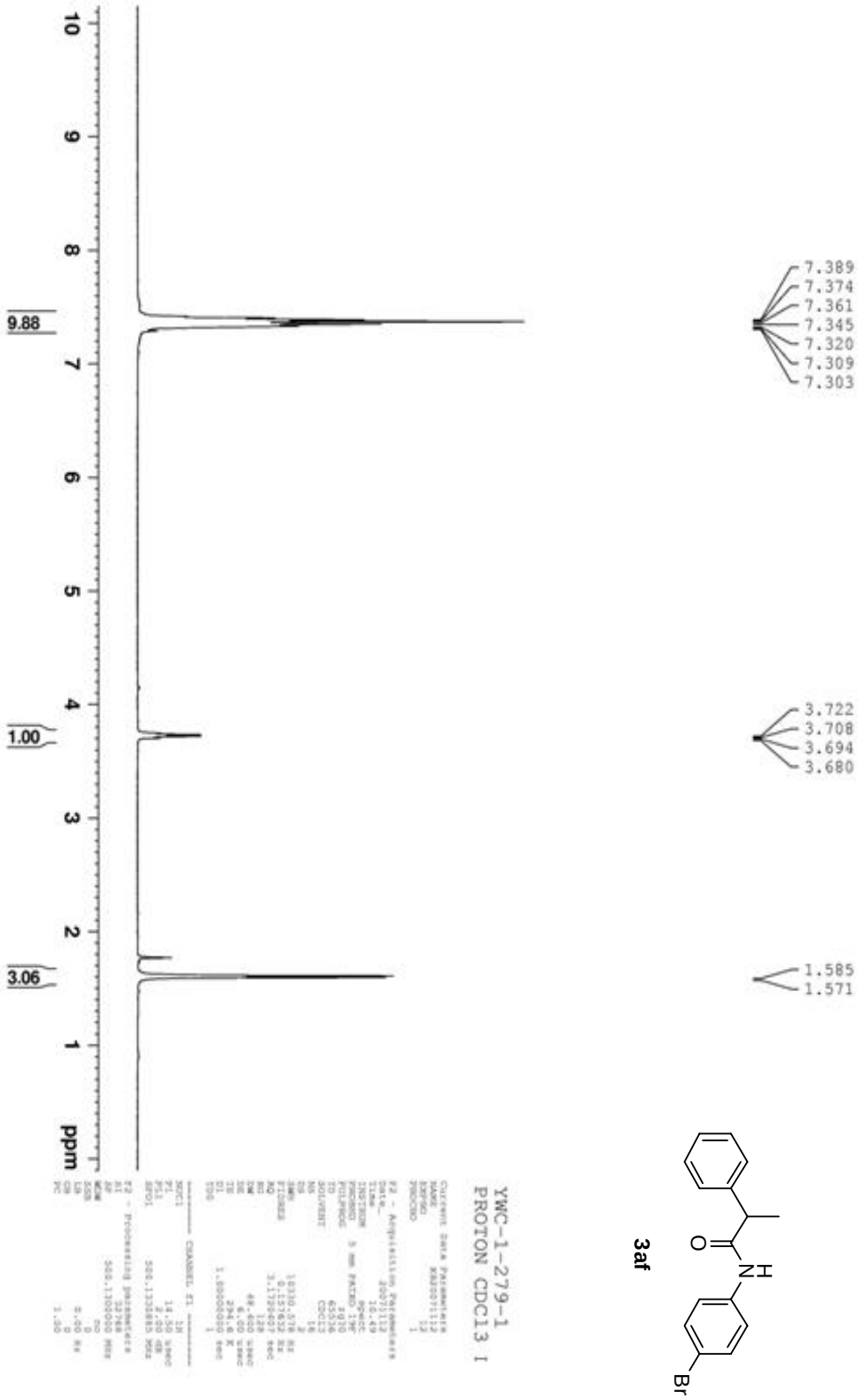

7.430
7.414
7.392
7.379
7.334
7.330
7.317
7.291
7.276
7.266
7.088
7.073
7.058
3.747
3.733
3.719
3.704
1.616
1.601
5.96
1.05
2.19
1.91
1.00
3.18
ppm
YWC-1-273-1
PROTON CDCl3
3ad


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
172.39
141.03
137.95
129.30
129.04
127.84
127.73
124.38
119.79
48.27
18.68
YWC-1-273-1
C13CPD CDCl3
3ad
O
N
H

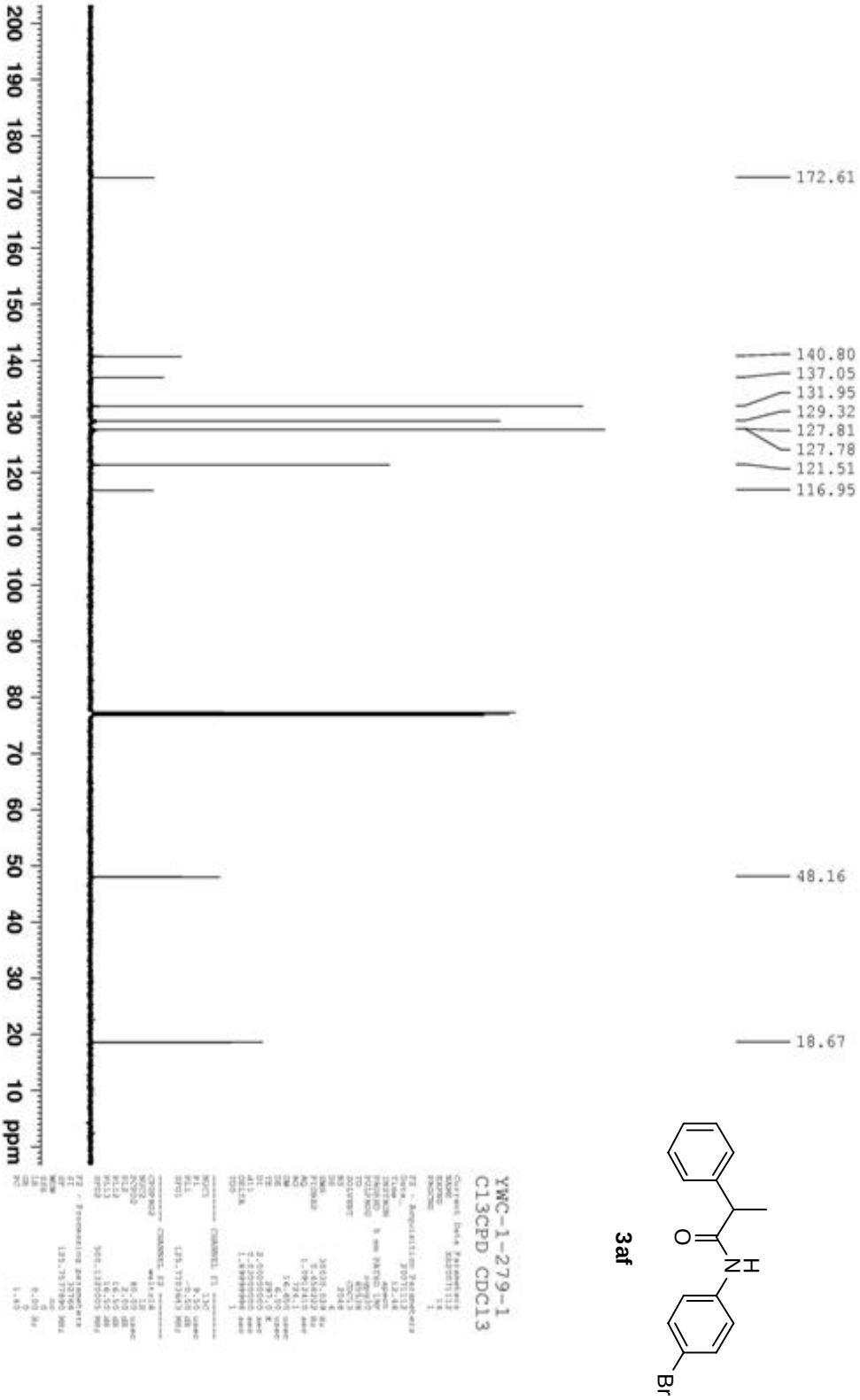

YWC-1-278-1
PROTON CDC13
6.93
0.94
2.04
1.00
3.04
ppm
7.393
7.373
7.364
7.355
7.351
7.336
7.329
7.312
7.298
7.260
7.219
7.201
3.726
3.712
3.698
3.684
1.589
1.575
O
N
H
Cl
3ae


172.56
140.80
136.52
129.32
129.00
127.80
127.78
121.15
48.15
18.66
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-278-1
C13 CDCl3
O
N
H
Cl

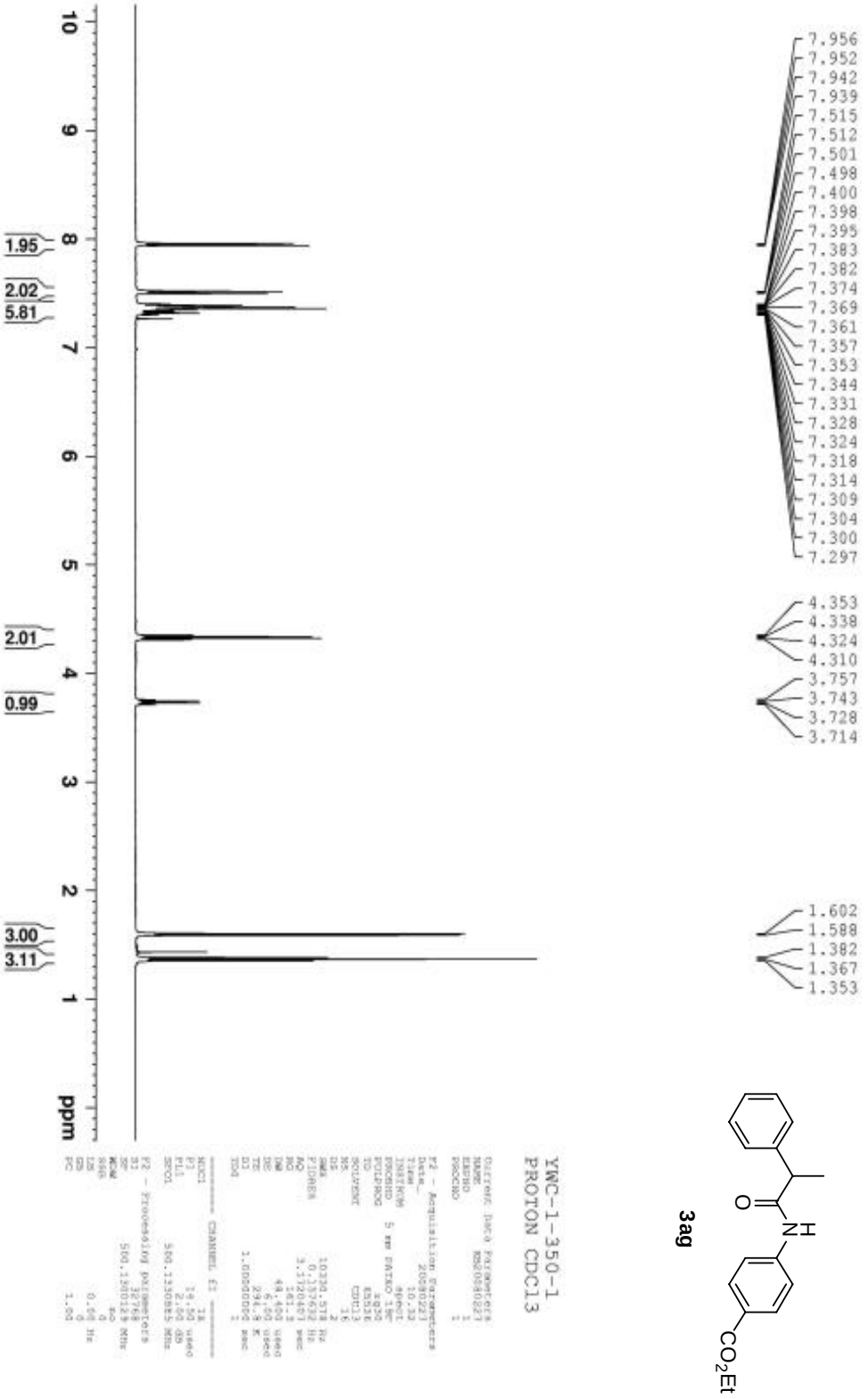
3ae


7.389
7.374
7.361
7.345
7.320
7.309
7.303
3.722
3.708
3.694
3.680
1.585
1.571
9.88
1.00
3.06
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-279-1
PROTON CDC13 I
O
N
H
Br

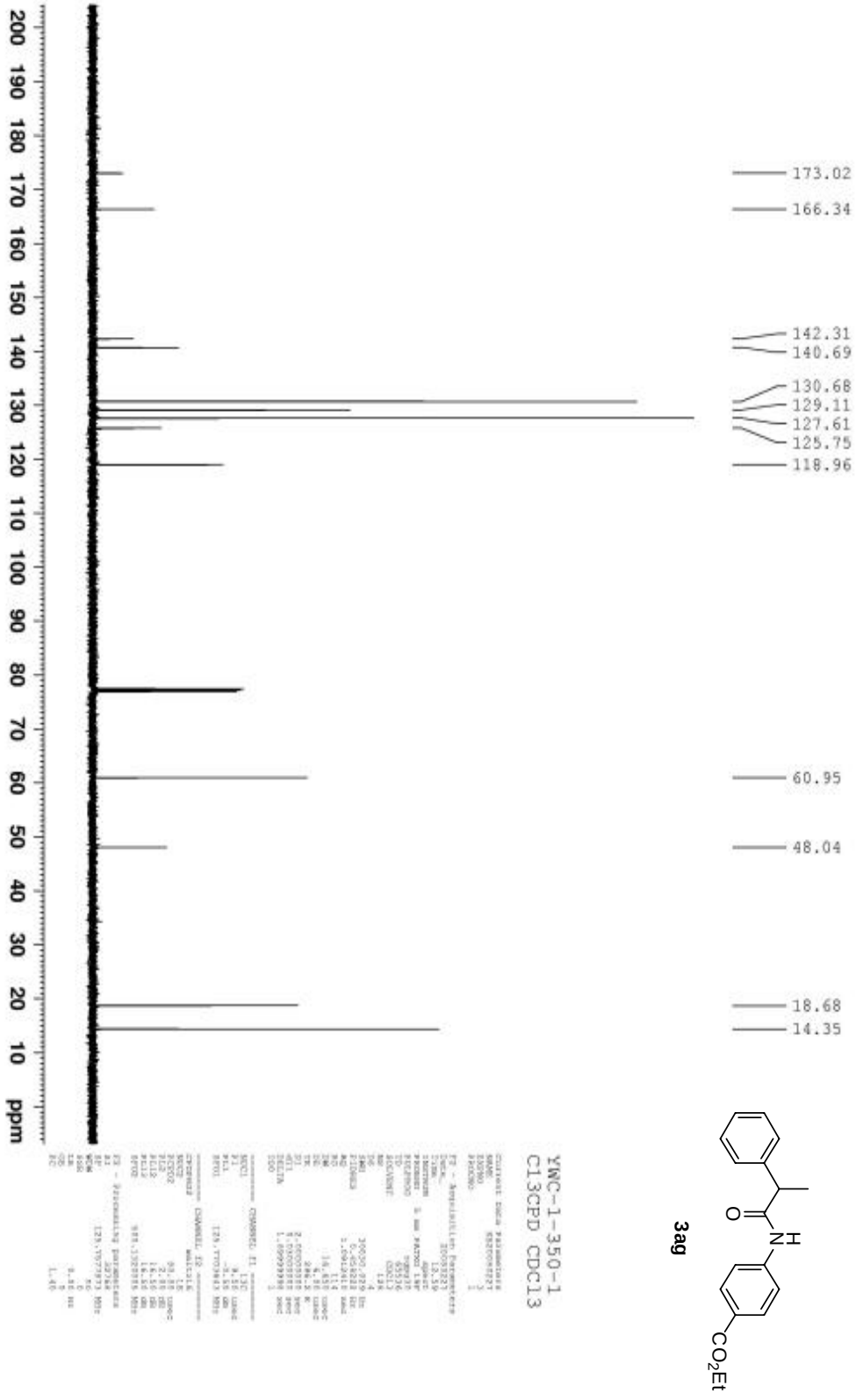
3af


172.61
140.80
137.05
131.95
129.32
127.81
127.78
121.51
116.95
48.16
18.67
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-279-1
C13CPD CDC13
O
N
H
Br

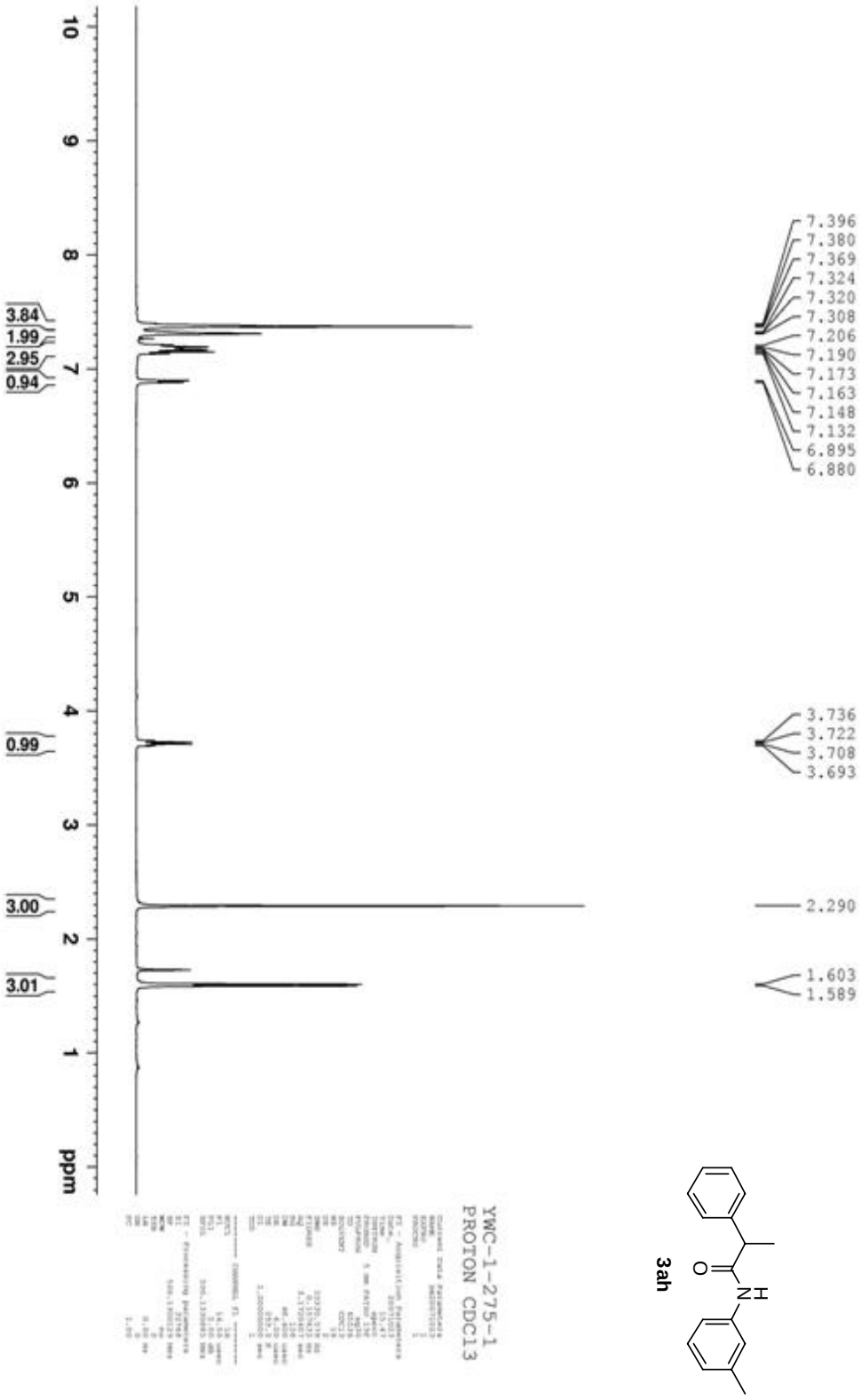
3af

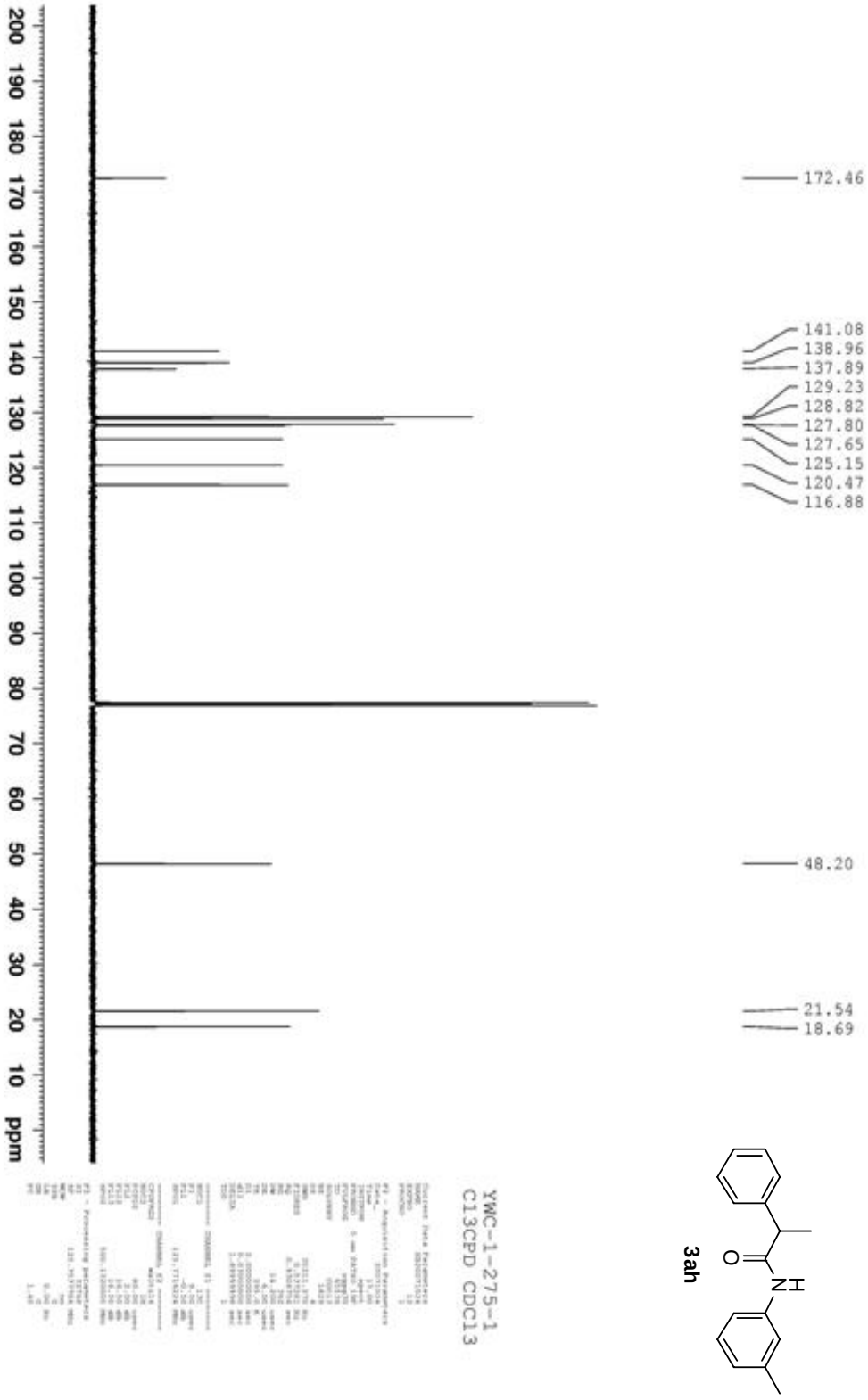

7.956
7.952
7.942
7.939
7.515
7.512
7.501
7.498
7.400
7.398
7.395
7.383
7.382
7.374
7.369
7.361
7.357
7.353
7.344
7.331
7.328
7.324
7.318
7.314
7.309
7.304
7.300
7.297
4.353
4.338
4.324
4.310
3.757
3.743
3.728
3.714
1.602
1.588
1.382
1.367
1.353
1.95
2.02
5.81
2.01
0.99
3.00
3.11
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-350-1
PROTON CDC13
O
N
H
CO2Et

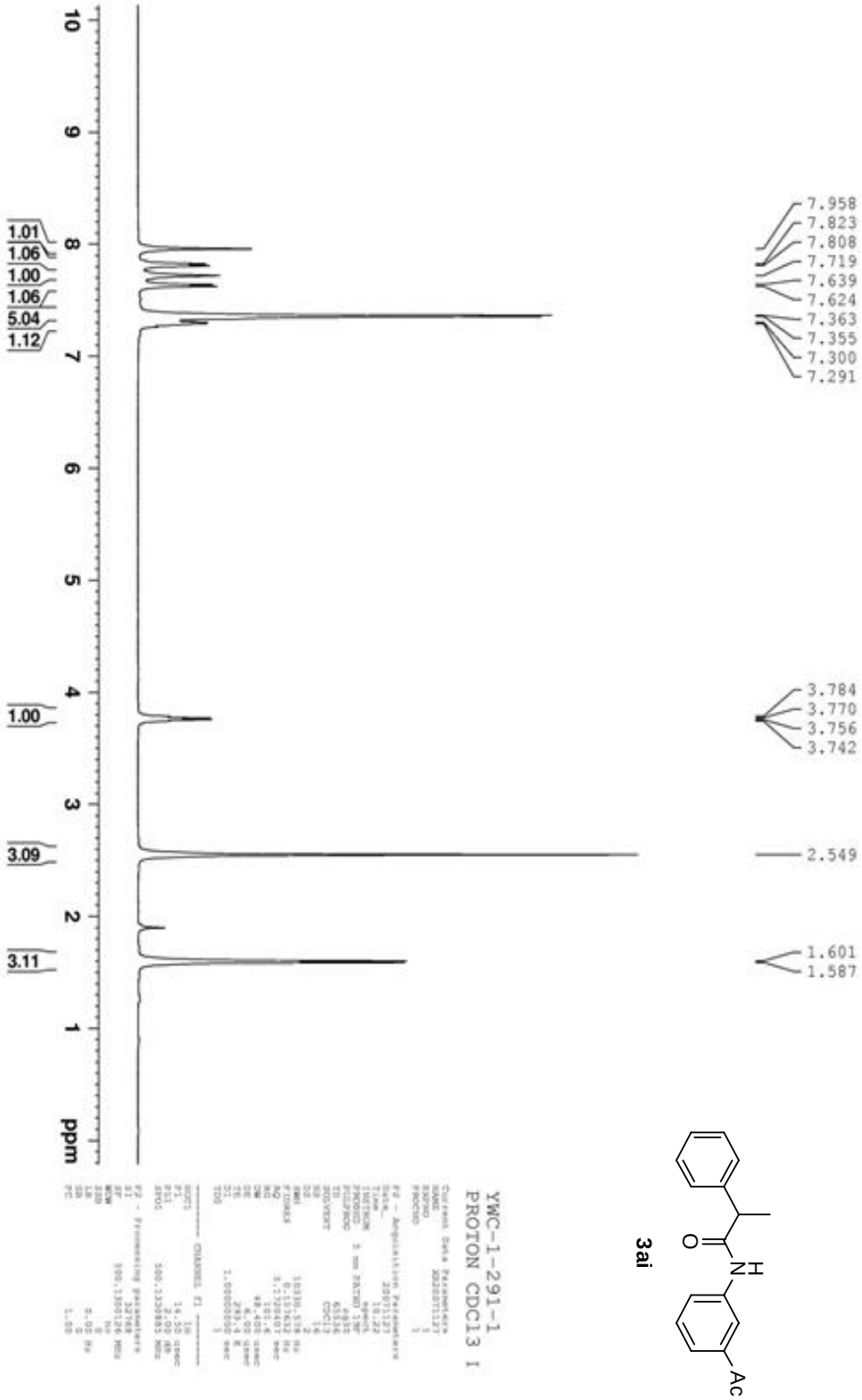
3ag


YWC-1-350-1
C13CPD CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
173.02
166.34
142.31
140.69
130.68
129.11
127.61
125.75
118.96
60.95
48.04
18.68
14.35
O
N
H
CO2Et

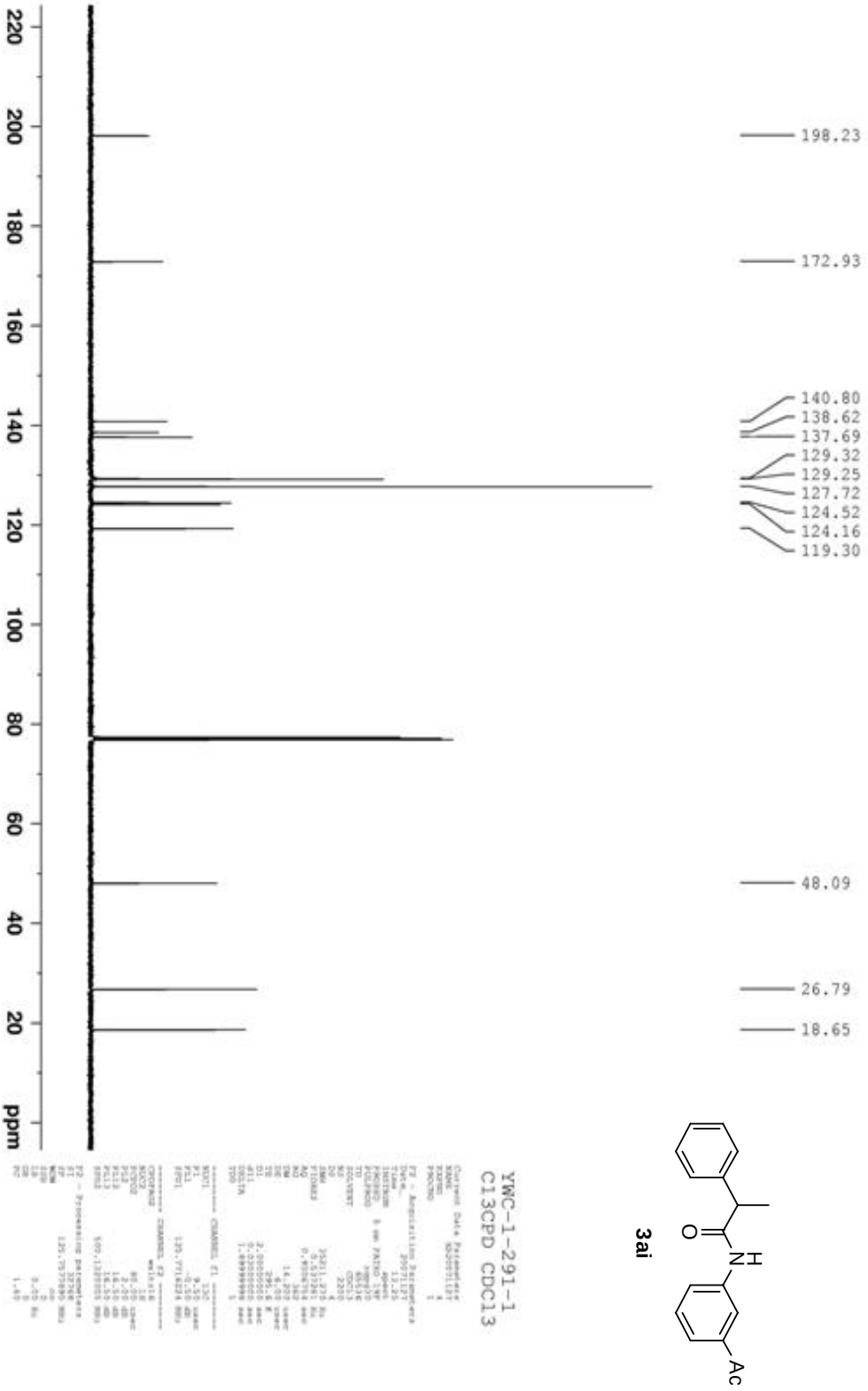
3ag


7.396
7.380
7.369
7.324
7.320
7.308
7.206
7.190
7.173
7.163
7.148
7.132
6.895
6.880
3.736
3.722
3.708
3.693
2.290
1.603
1.589
3.84
1.99
2.95
0.94
0.99
3.00
3.01
ppm
YWC-1-275-1
PROTON CDCl3
3ah

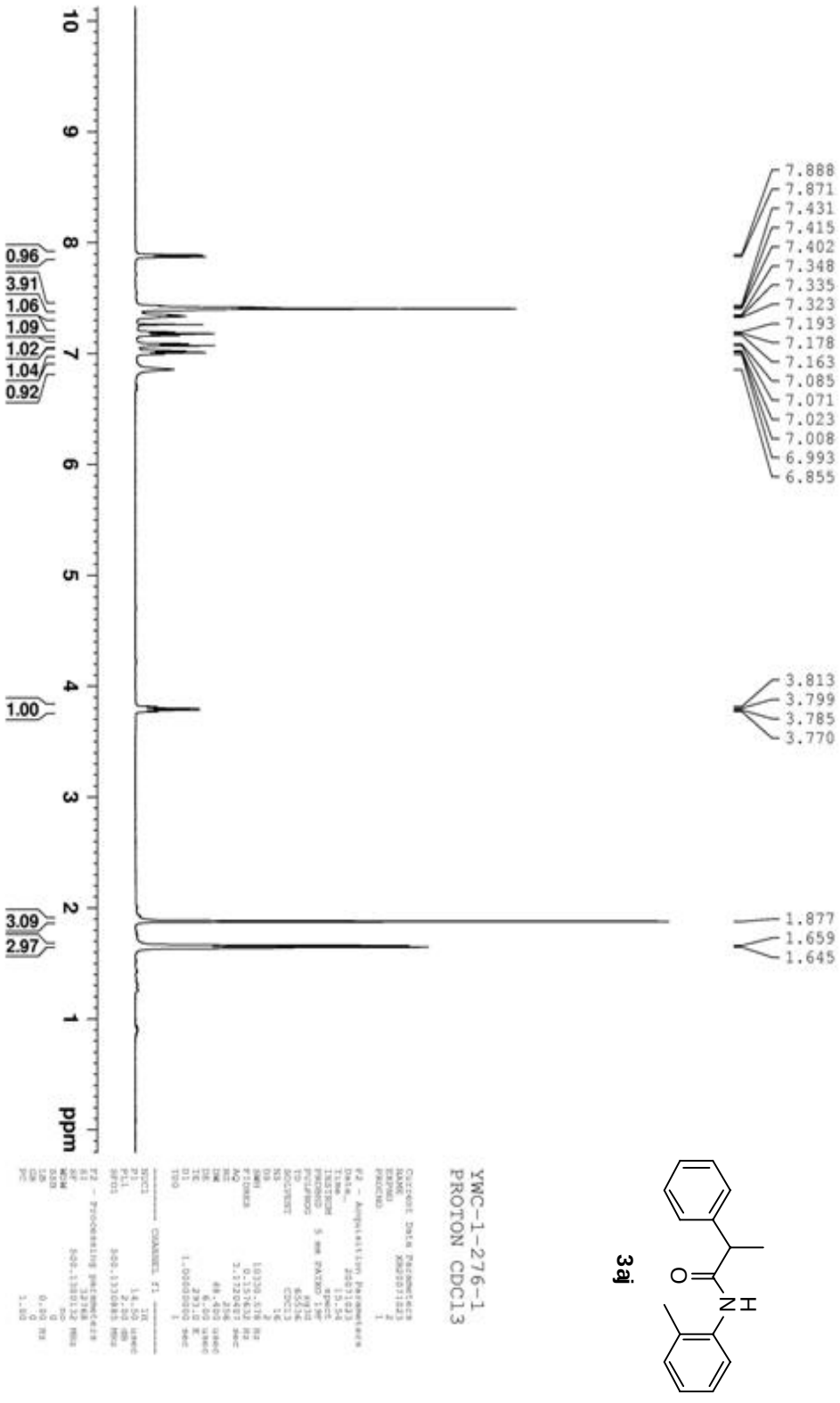

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
172.46
141.08
138.96
137.89
129.23
128.82
127.80
127.65
125.15
120.47
116.88
48.20
21.54
18.69
YWC-1-275-1
C13CPD CDCl3
O
N
H
3ah

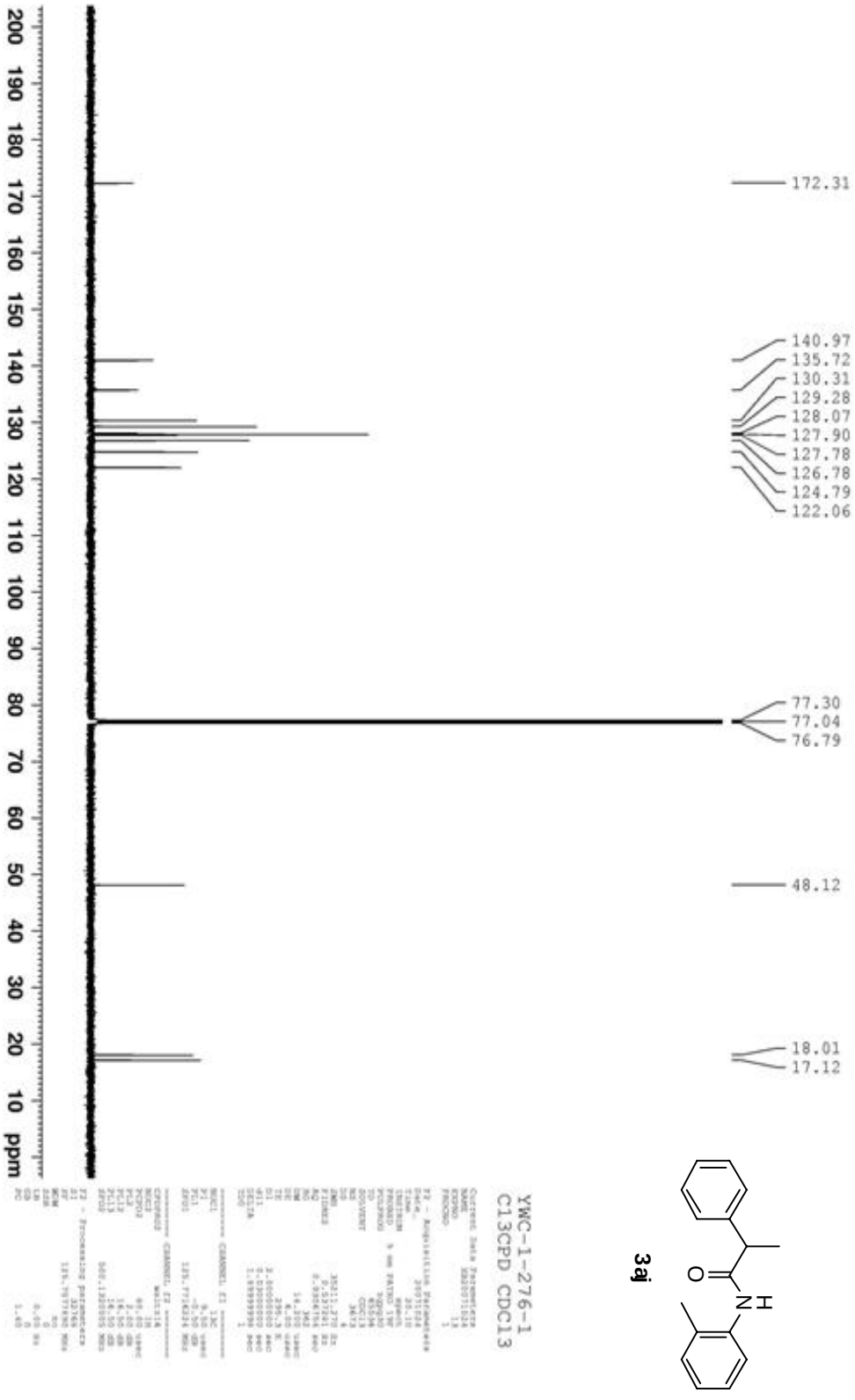

7.958
7.823
7.808
7.719
7.639
7.624
7.363
7.355
7.300
7.291
3.784
3.770
3.756
3.742
2.549
1.601
1.587
1.01
1.06
1.00
1.06
5.04
1.12
1.00
3.09
3.11
ppm
YWC-1-291-1
PROTON CDC13 1
O
N
H
Ac

3ai

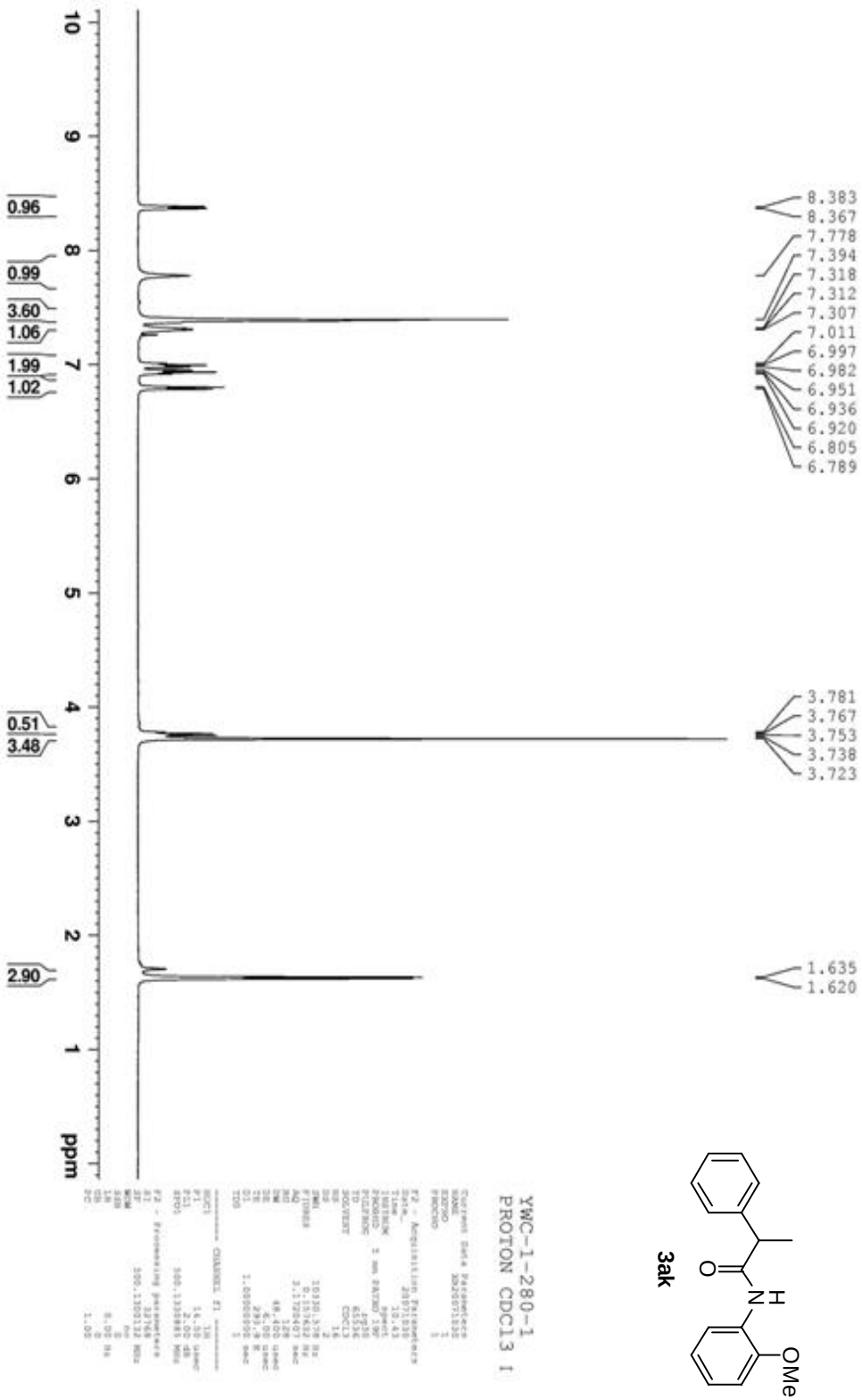

198.23
172.93
140.80
138.62
137.69
129.32
129.25
127.72
124.52
124.16
119.30
48.09
26.79
18.65
ppm
YWC-1-291-1
C13CPD CDCl3
O
N
H
Ac

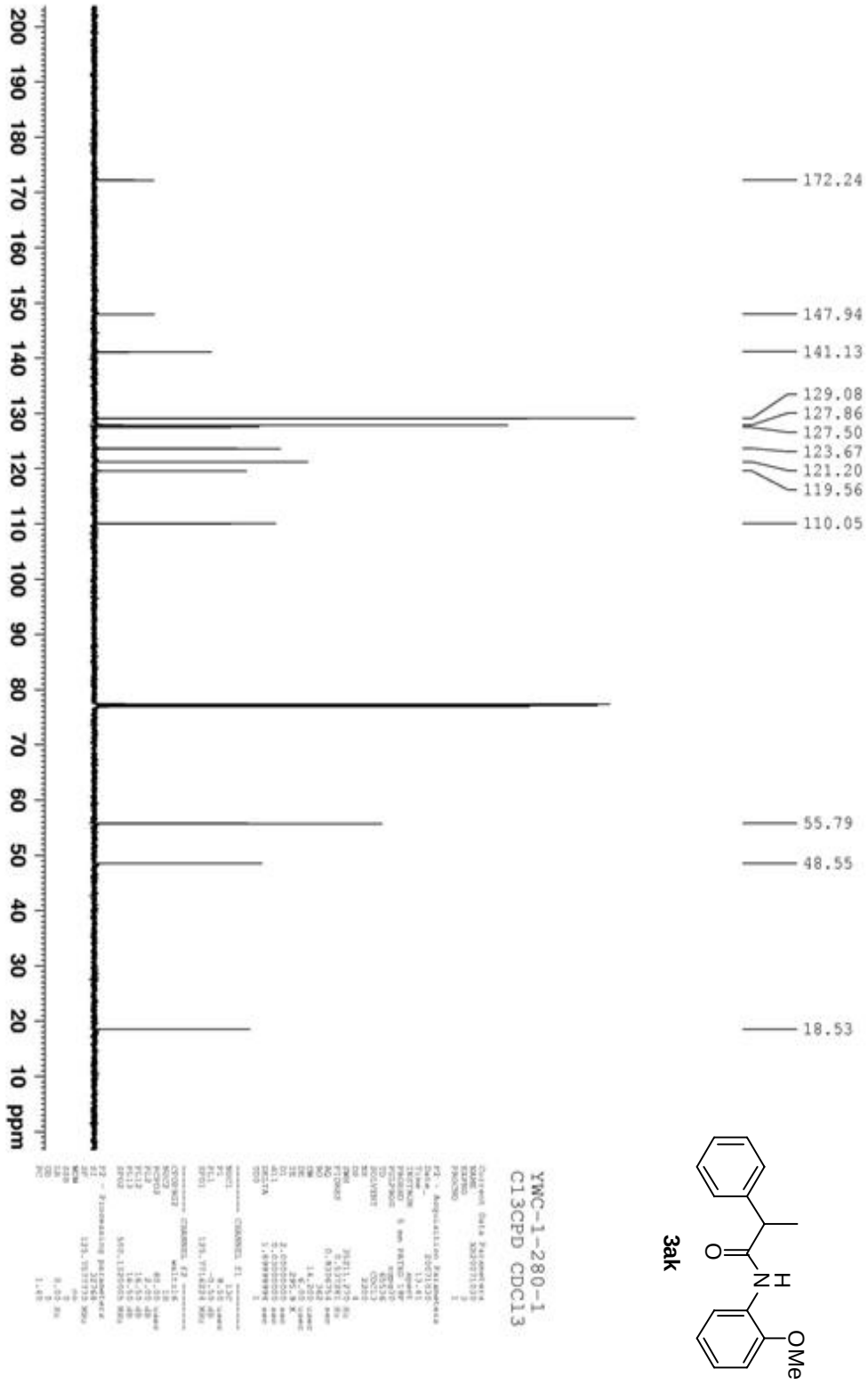
3ai


7.888
7.871
7.431
7.415
7.402
7.348
7.335
7.323
7.193
7.178
7.163
7.085
7.071
7.023
7.008
6.993
6.855
3.813
3.799
3.785
3.770
1.877
1.659
1.645
0.96
3.91
1.06
1.09
1.02
1.04
0.92
1.00
3.09
2.97
10
9
8
7
6
5
4
3
2
1
ppm
YWC-1-276-1
PROTON CDCl3
3aj

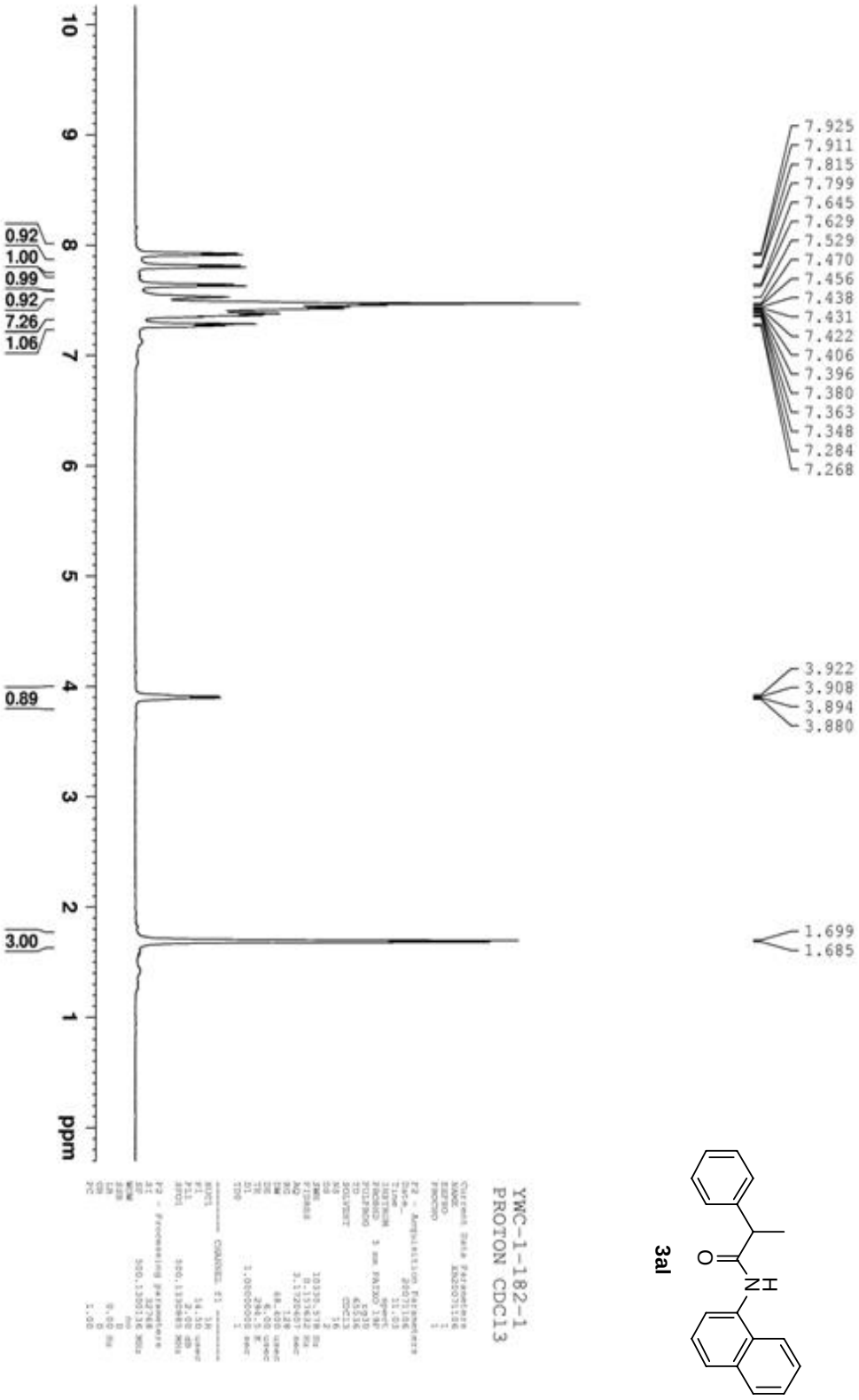

172.31
140.97
135.72
130.31
129.28
128.07
127.90
127.78
126.78
124.79
122.06
77.30
77.04
76.79
48.12
18.01
17.12
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-276-1
C13CPD CDCl3
O
N
H

3aj

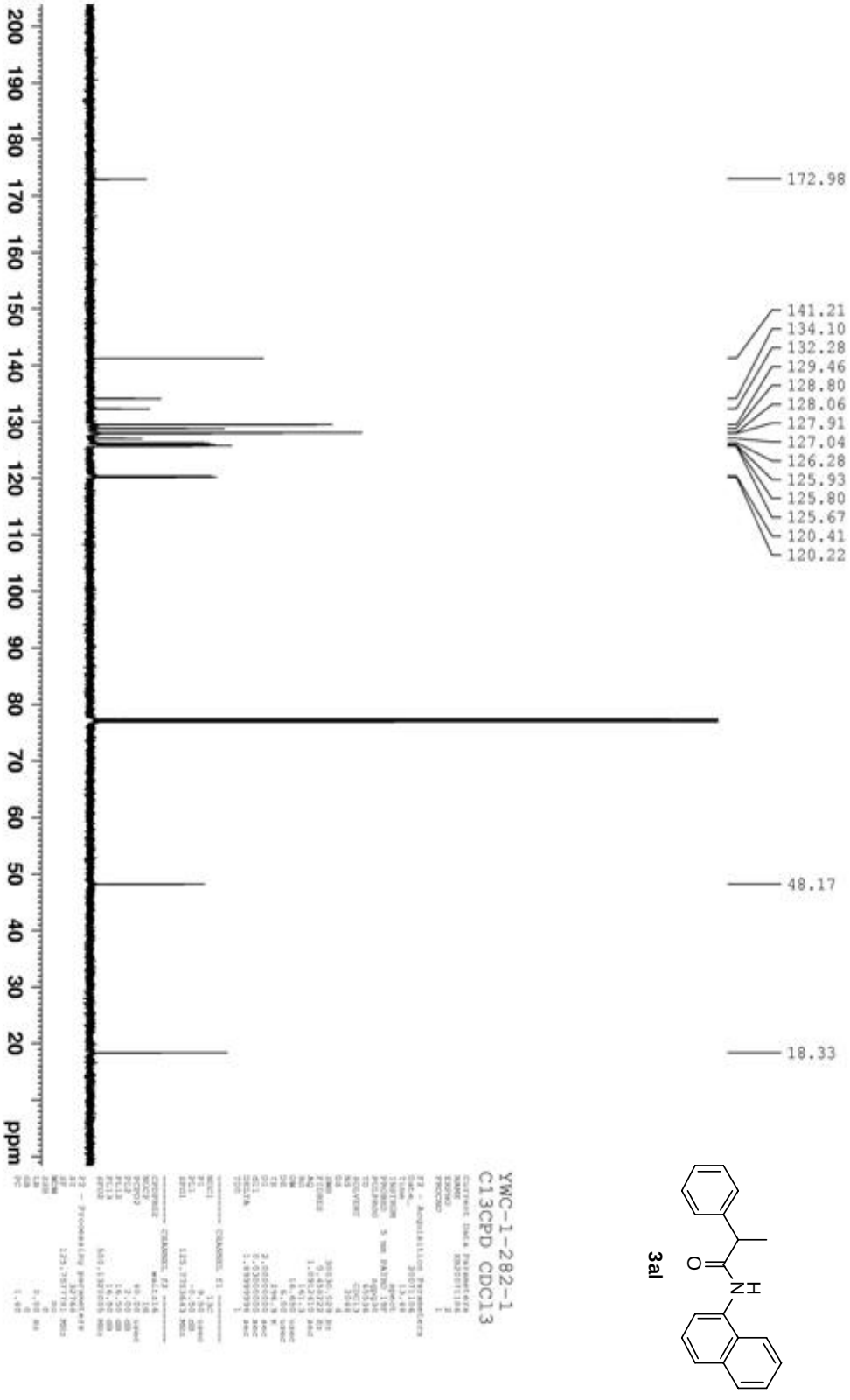

YWC-1-280-1
PROTON CDCl3 1
8.383
8.367
7.778
7.394
7.318
7.312
7.307
7.011
6.997
6.982
6.951
6.936
6.920
6.805
6.789
3.781
3.767
3.753
3.738
3.723
1.635
1.620
0.96
0.99
3.60
1.06
1.99
1.02
0.51
3.48
2.90
ppm
3ak
O
H
N
OMe

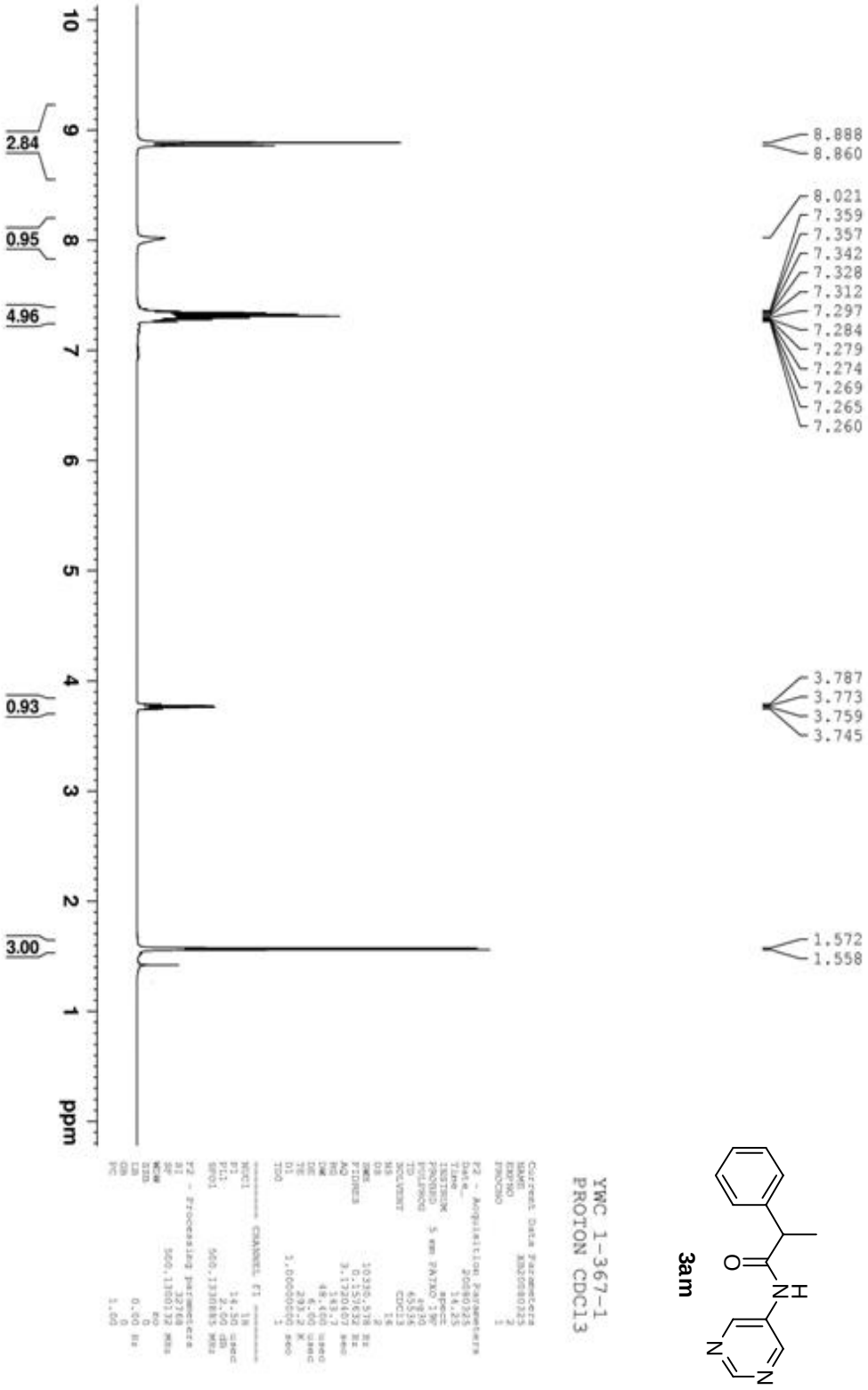

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
172.24
147.94
141.13
129.08
127.86
127.50
123.67
121.20
119.56
110.05
55.79
48.55
18.53
YWC-1-280-1
C13CPD CDCl3
O
N
H
OMe

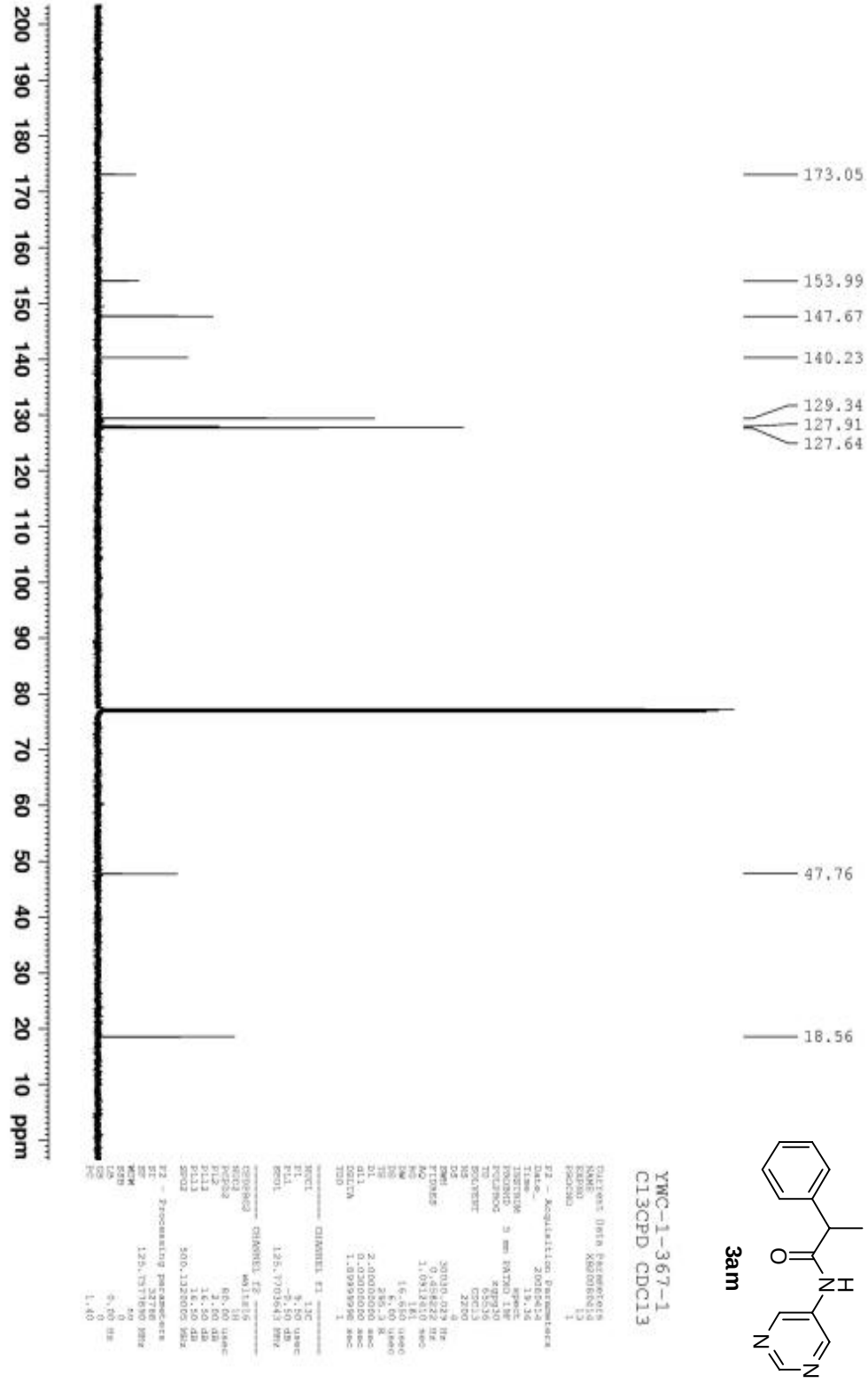
3ak

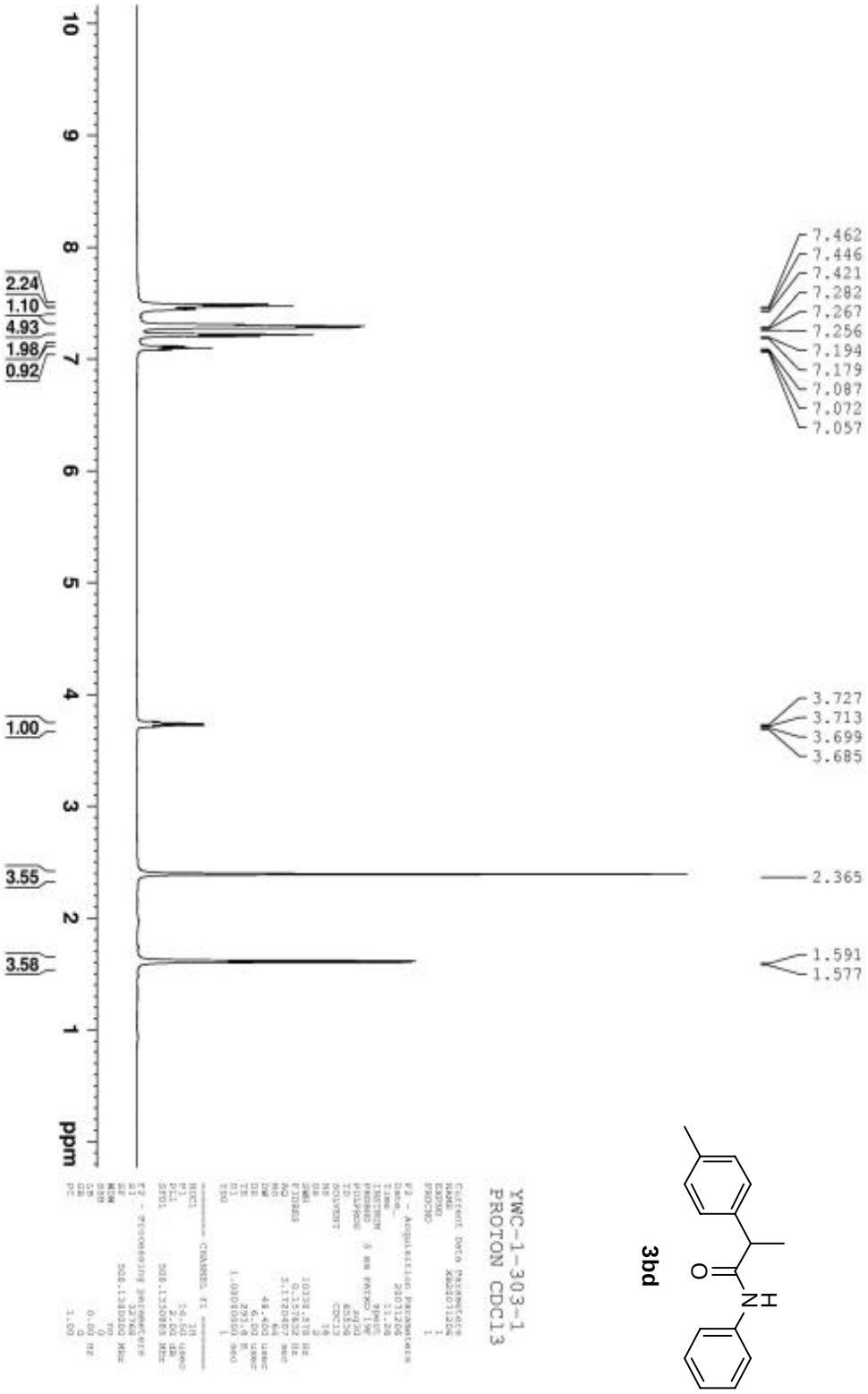

YWC-1-182-1
PROTON CDCl3
ppm
0.92
1.00
0.99
0.92
7.26
1.06
0.89
3.00
7.925
7.911
7.815
7.799
7.645
7.629
7.529
7.470
7.456
7.438
7.431
7.422
7.406
7.396
7.380
7.363
7.348
7.284
7.268
3.922
3.908
3.894
3.880
1.699
1.685

3al

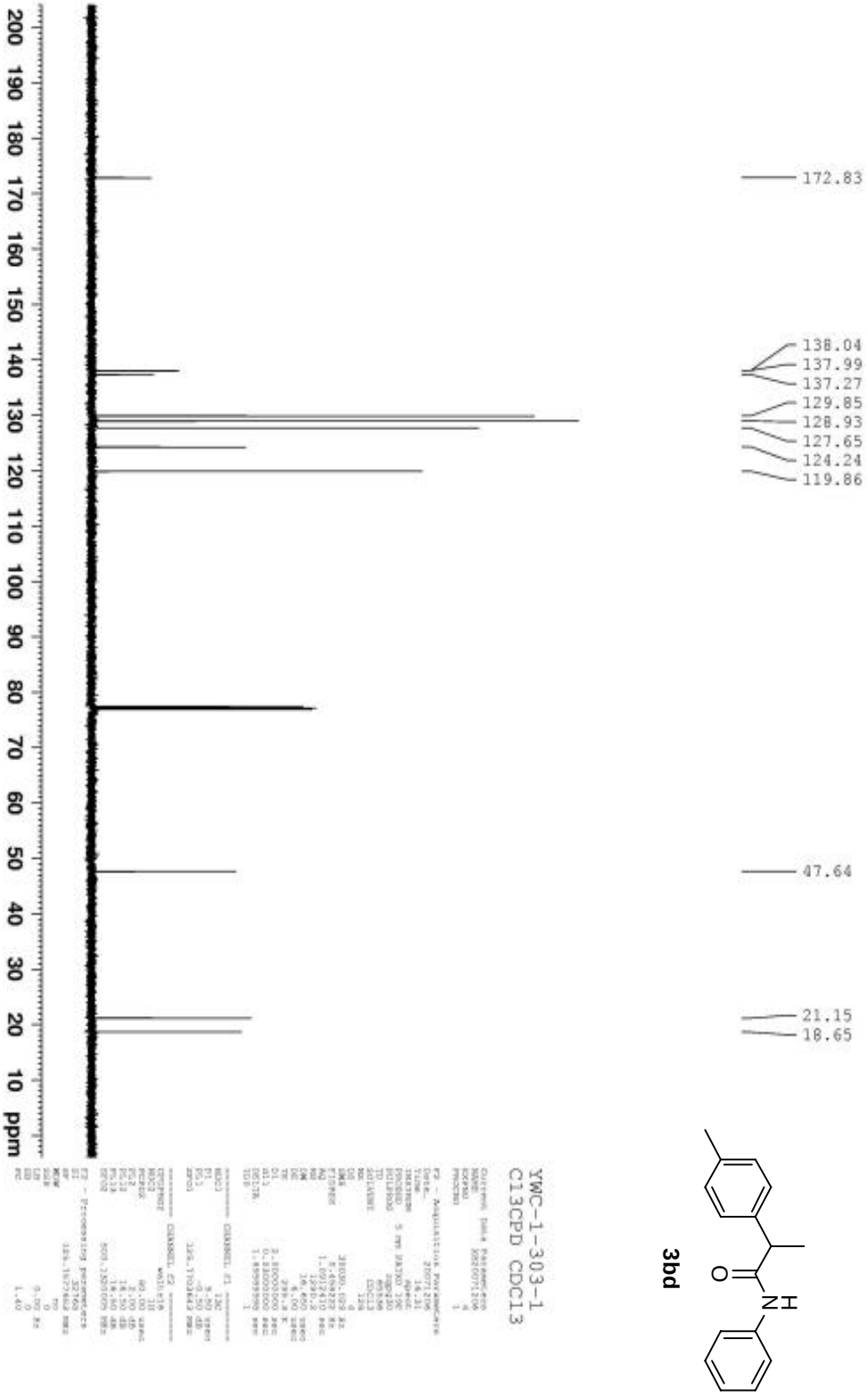

172.98
141.21
134.10
132.28
129.46
128.80
128.06
127.91
127.04
126.28
125.93
125.80
125.67
120.41
120.22
48.17
18.33
ppm
YWC-1-282-1
C13CPD CDCl3
3al


8.888
8.860
8.021
7.359
7.357
7.342
7.328
7.312
7.297
7.284
7.279
7.274
7.269
7.265
7.260
3.787
3.773
3.759
3.745
1.572
1.558
2.84
0.95
4.96
0.93
3.00
ppm
YWC 1-367-1
PROTON CDCl3
3am

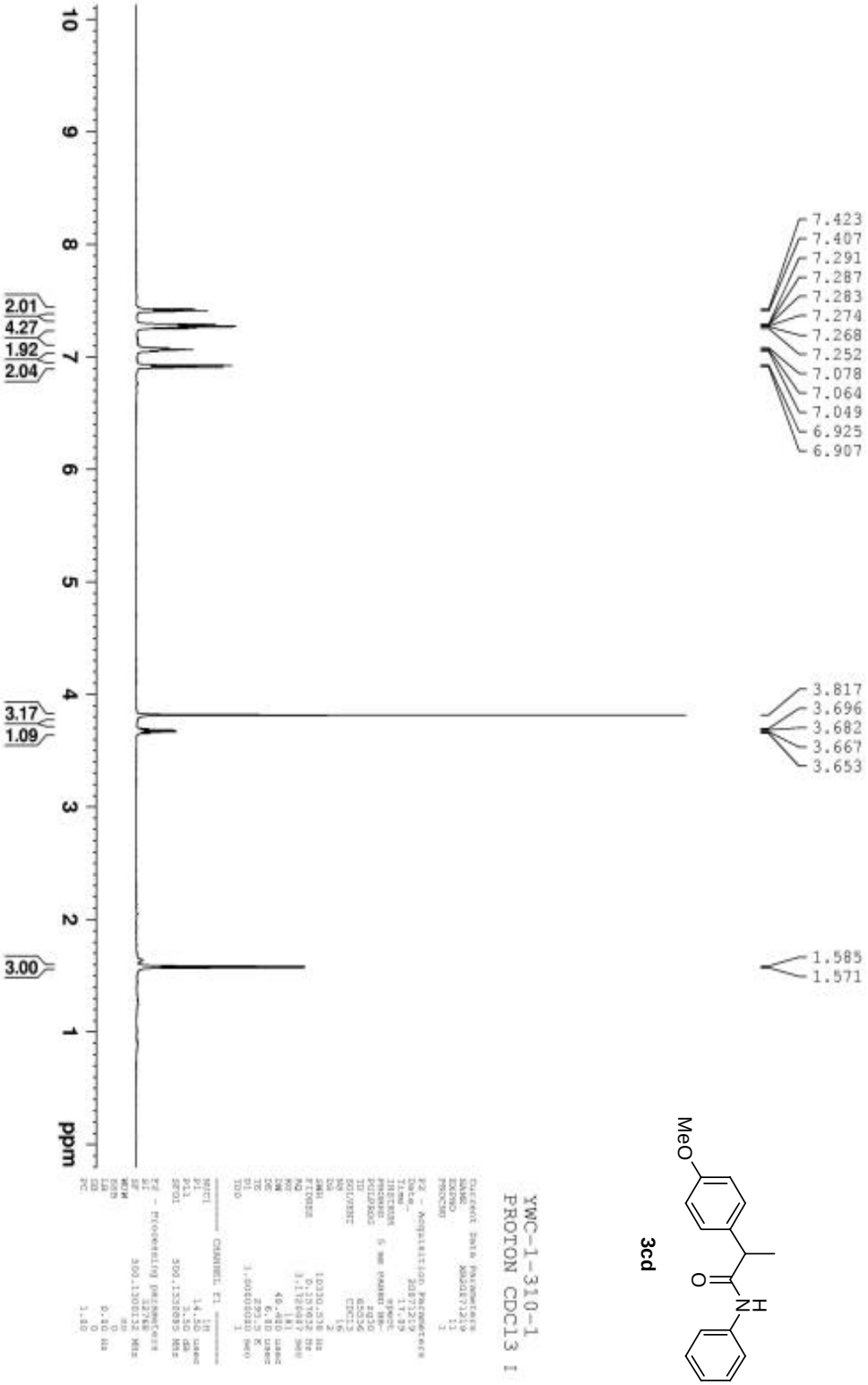

173.05
153.99
147.67
140.23
129.34
127.91
127.64
47.76
18.56
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-367-1
C13CPD CDC13
3am


YWC-1-303-1
PROTON CDCl3
ppm
10
9
8
7
6
5
4
3
2
1
2.24
1.10
4.93
1.98
0.92
1.00
3.55
3.58
7.462
7.446
7.421
7.282
7.267
7.256
7.194
7.179
7.087
7.072
7.057
3.727
3.713
3.699
3.685
2.365
1.591
1.577

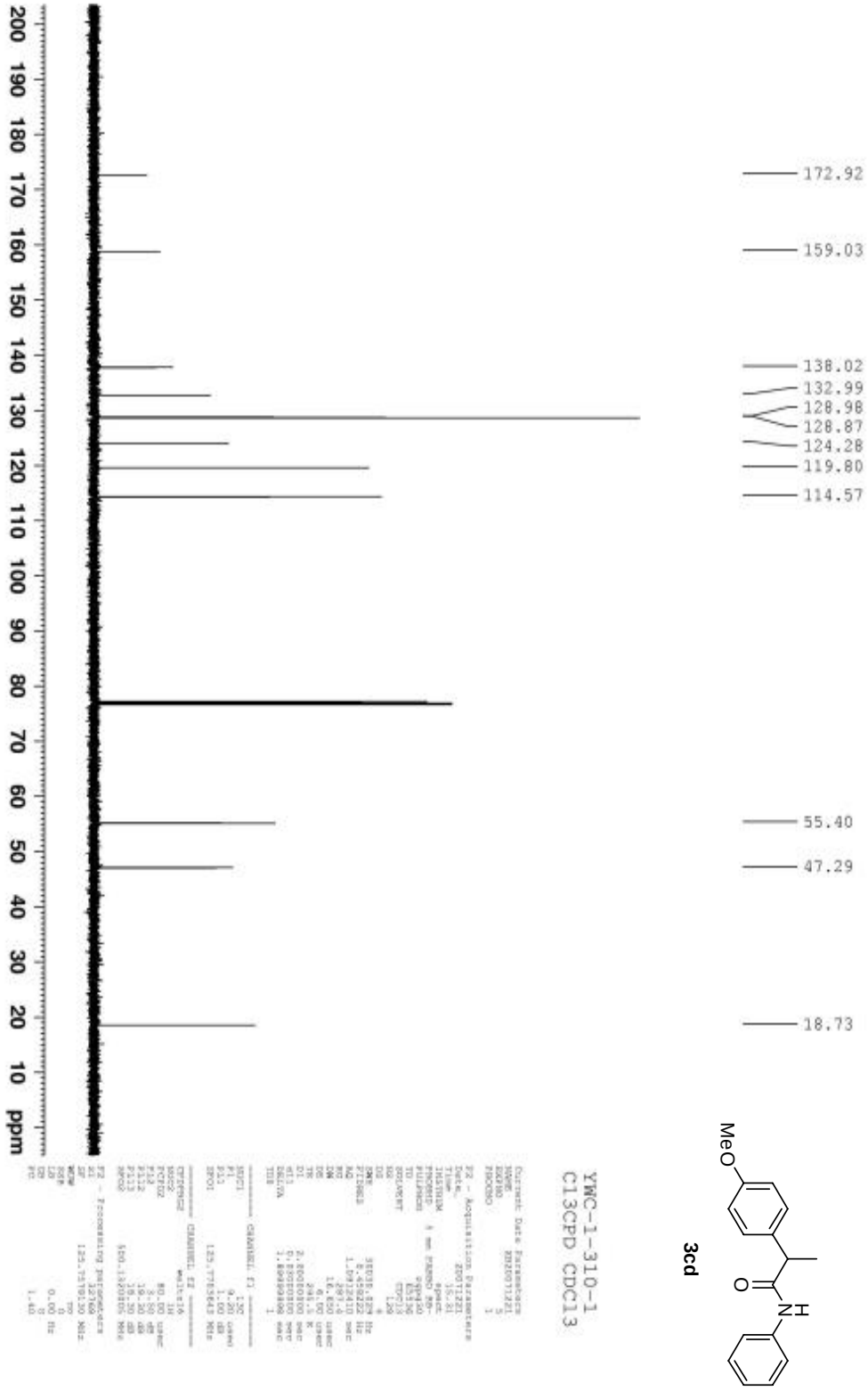
3bd


172.83
138.04
137.99
137.27
129.85
128.93
127.65
124.24
119.86
47.64
21.15
18.65
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-303-1
C13CPD CDCl3
O
N
H

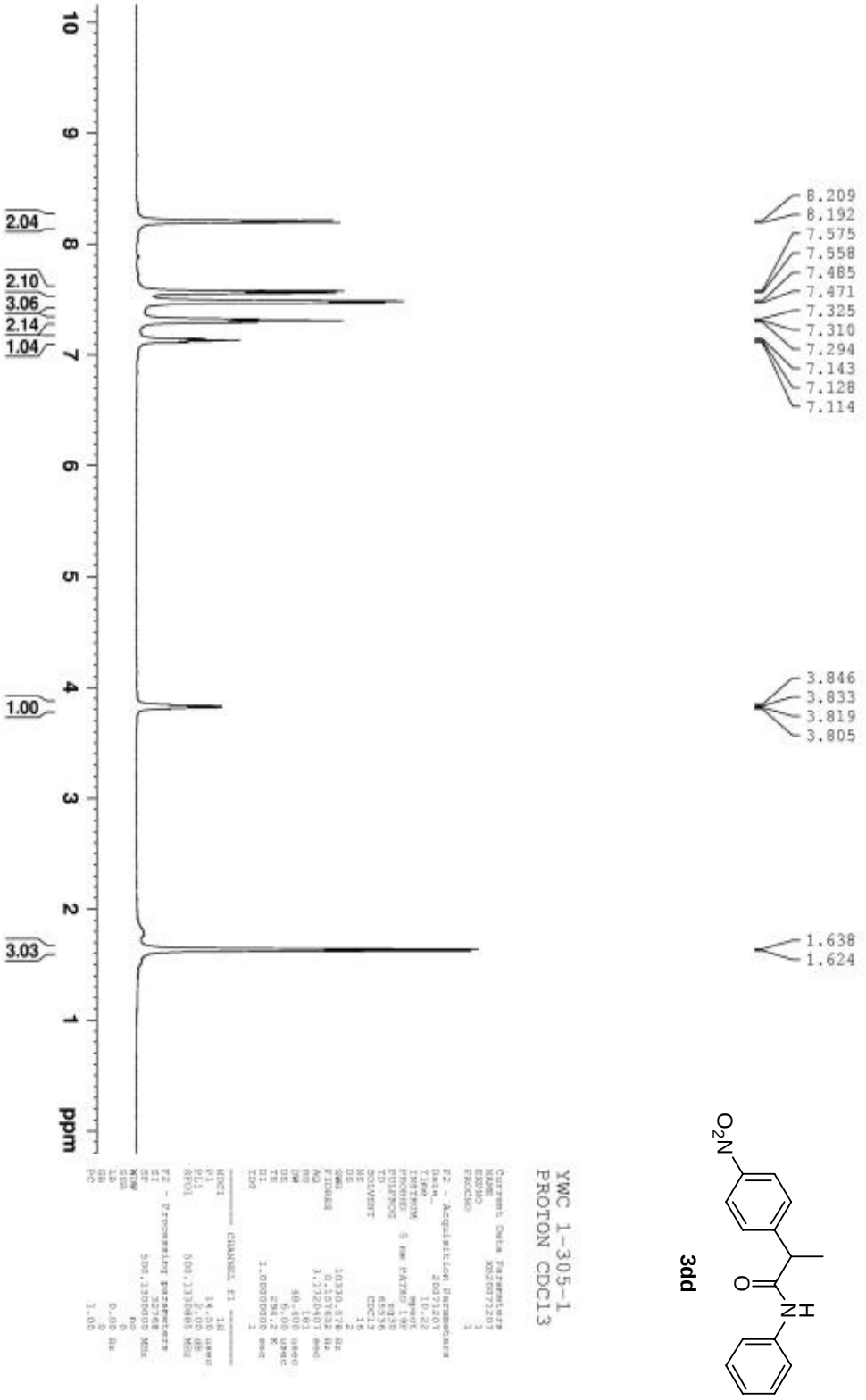
3bd


YWC-1-310-1
PROTON CDCl3 I
ppm
10
9
8
7
6
5
4
3
2
1
2.01
4.27
1.92
2.04
3.17
1.09
3.00
7.423
7.407
7.291
7.287
7.283
7.274
7.268
7.252
7.078
7.064
7.049
6.925
6.907
3.817
3.696
3.682
3.667
3.653
1.585
1.571
MeO
O
N
H

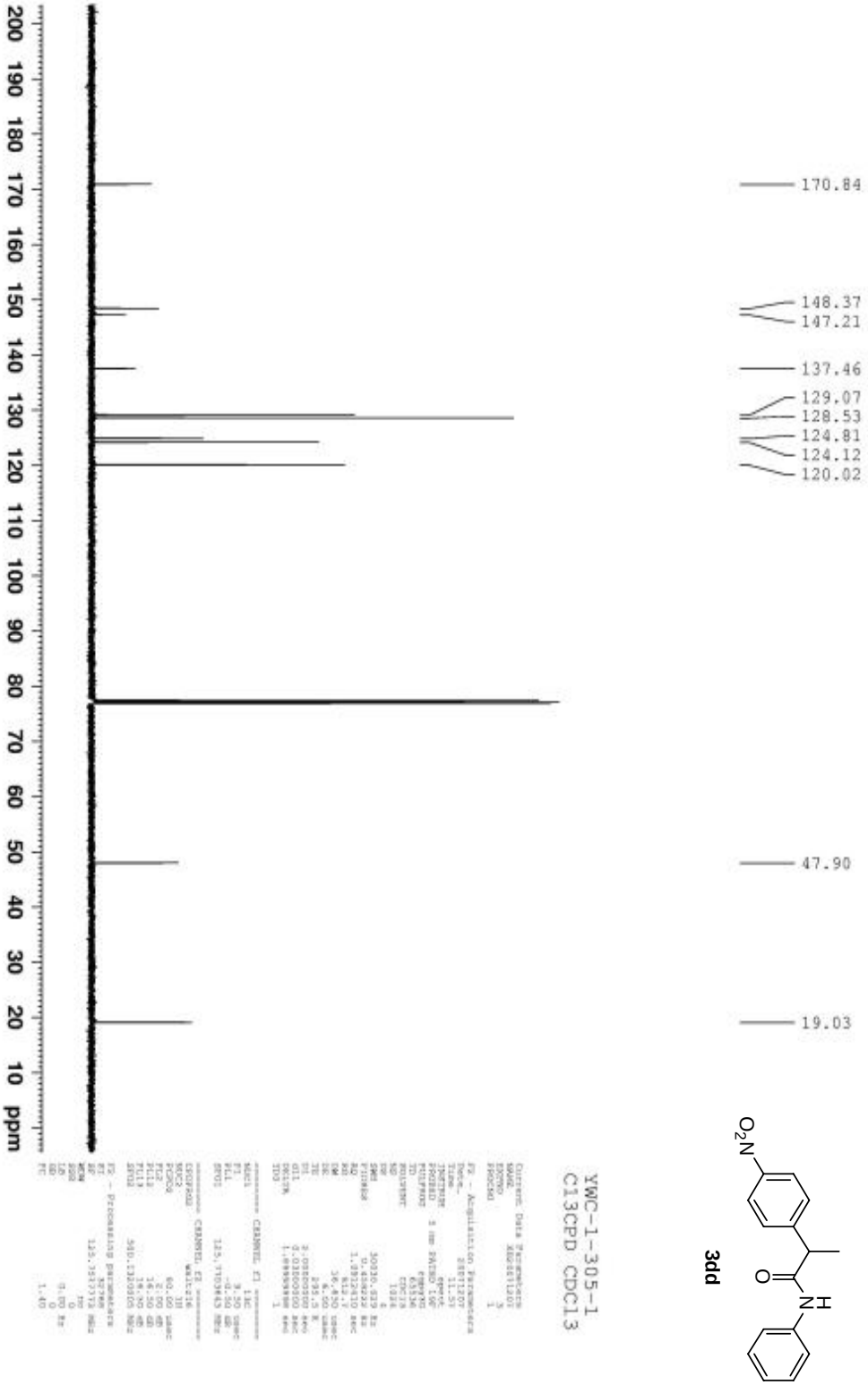
3cd


172.92
159.03
138.02
132.99
128.98
128.87
124.28
119.80
114.57
55.40
47.29
18.73
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-310-1
C13CPD CDCl3
MeO
O
N
H

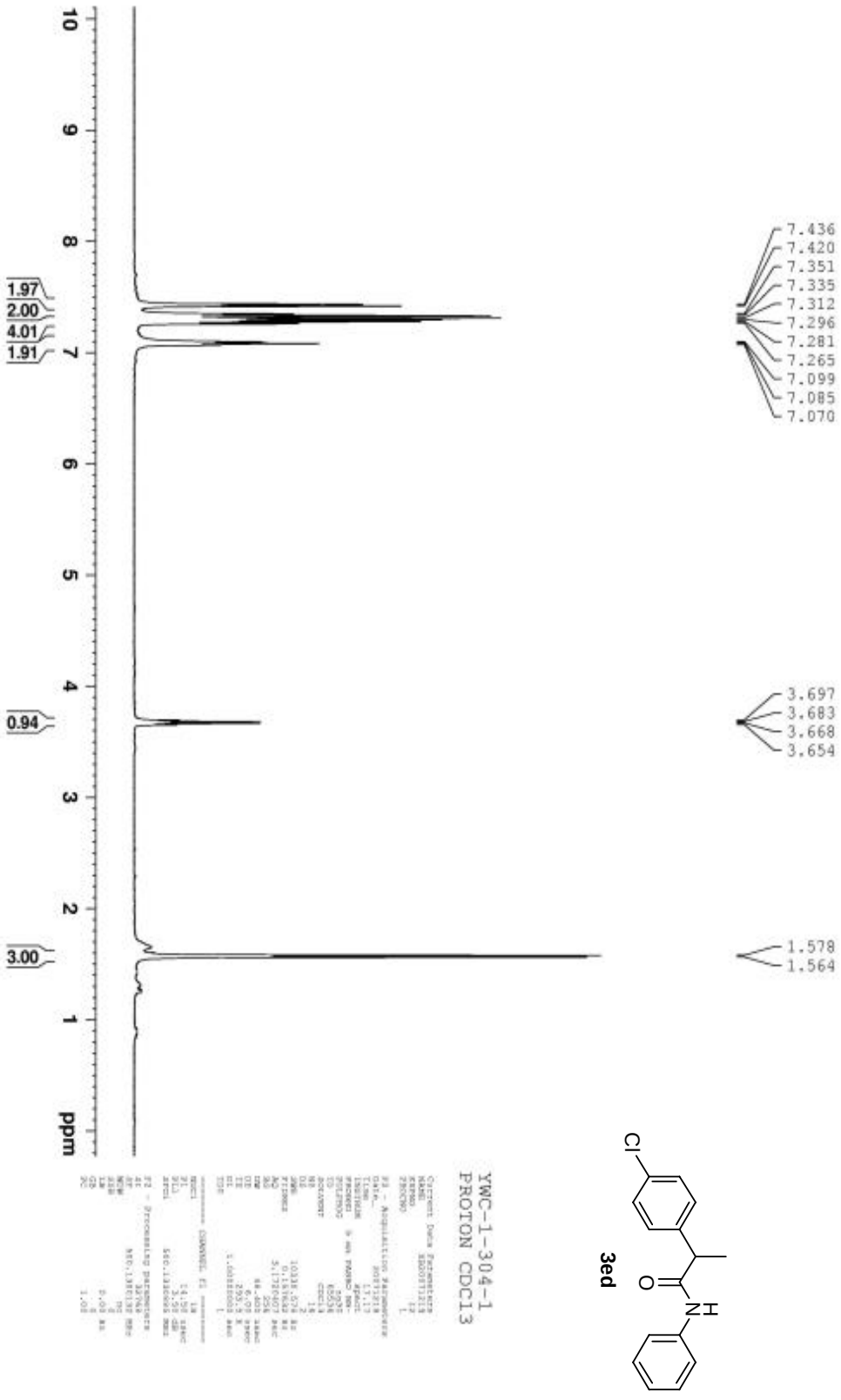
3cd

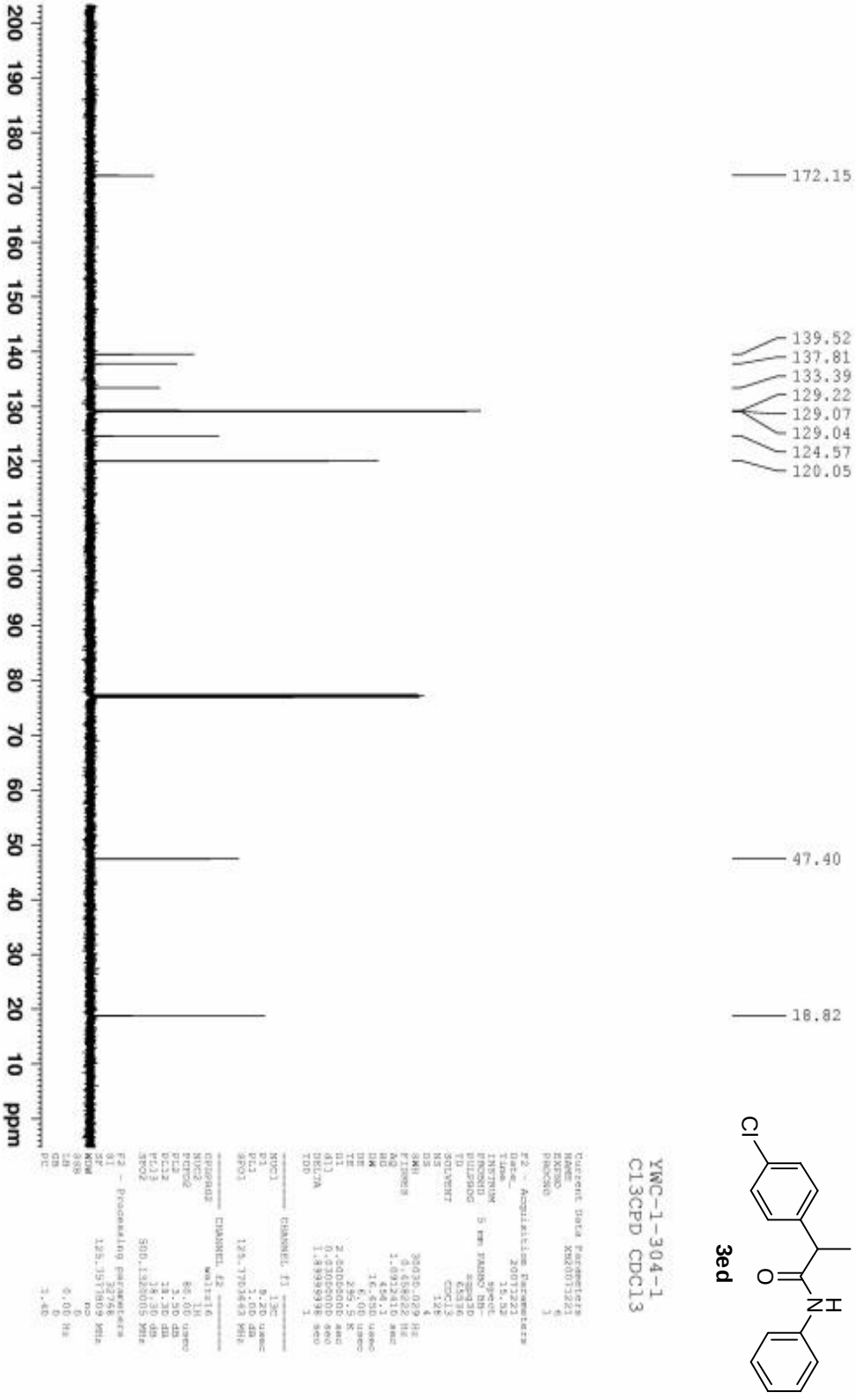

8.209
8.192
7.575
7.558
7.485
7.471
7.325
7.310
7.294
7.143
7.128
7.114
3.846
3.833
3.819
3.805
1.638
1.624
2.04
2.10
3.06
2.14
1.04
1.00
3.03
ppm
YWC 1-305-1
PROTON CDC13
O2N
O
N
H

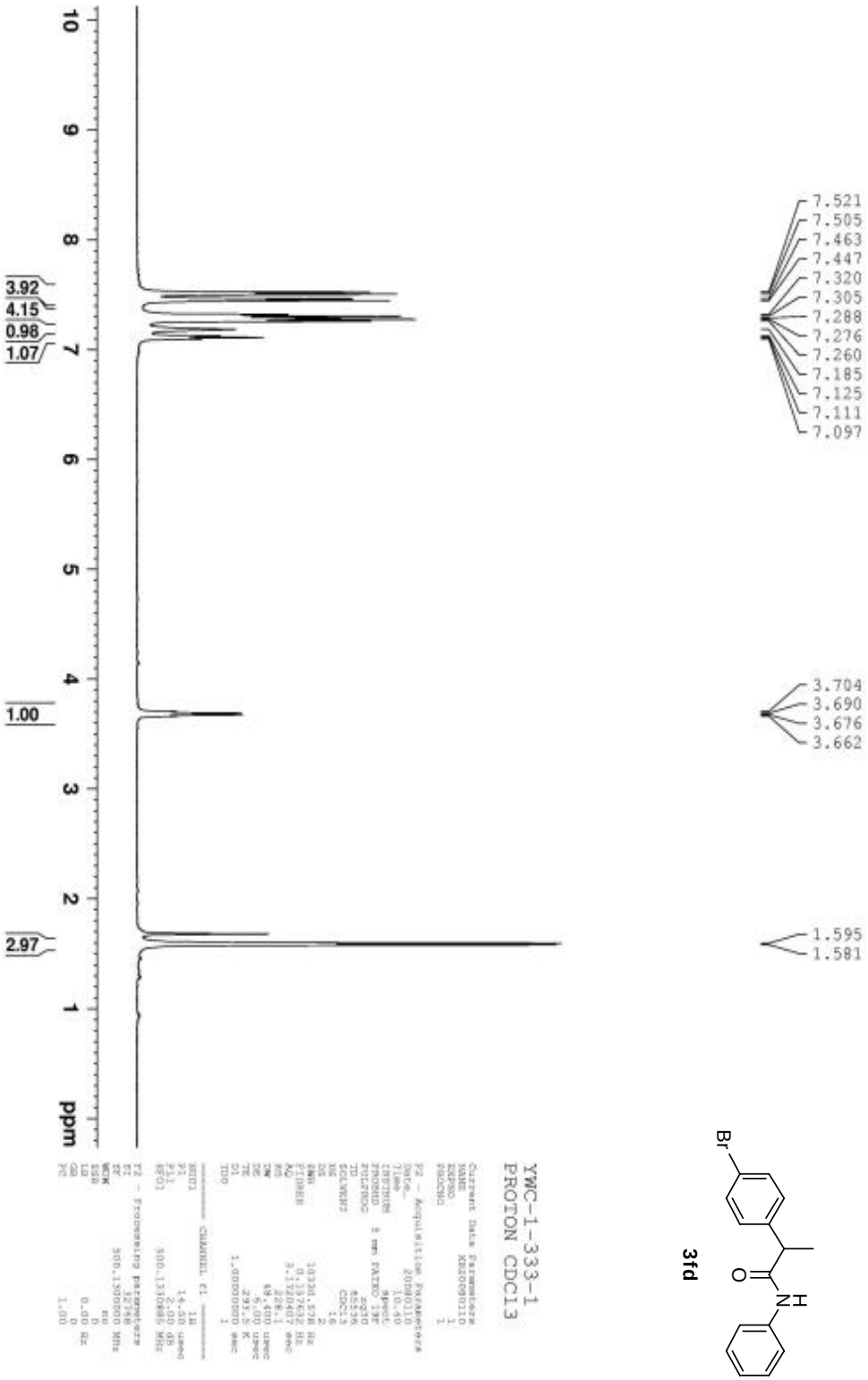
3dd


YWC-1-305-1
C13CPD CDCl3
170.84
148.37
147.21
137.46
129.07
128.53
124.81
124.12
120.02
47.90
19.03
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

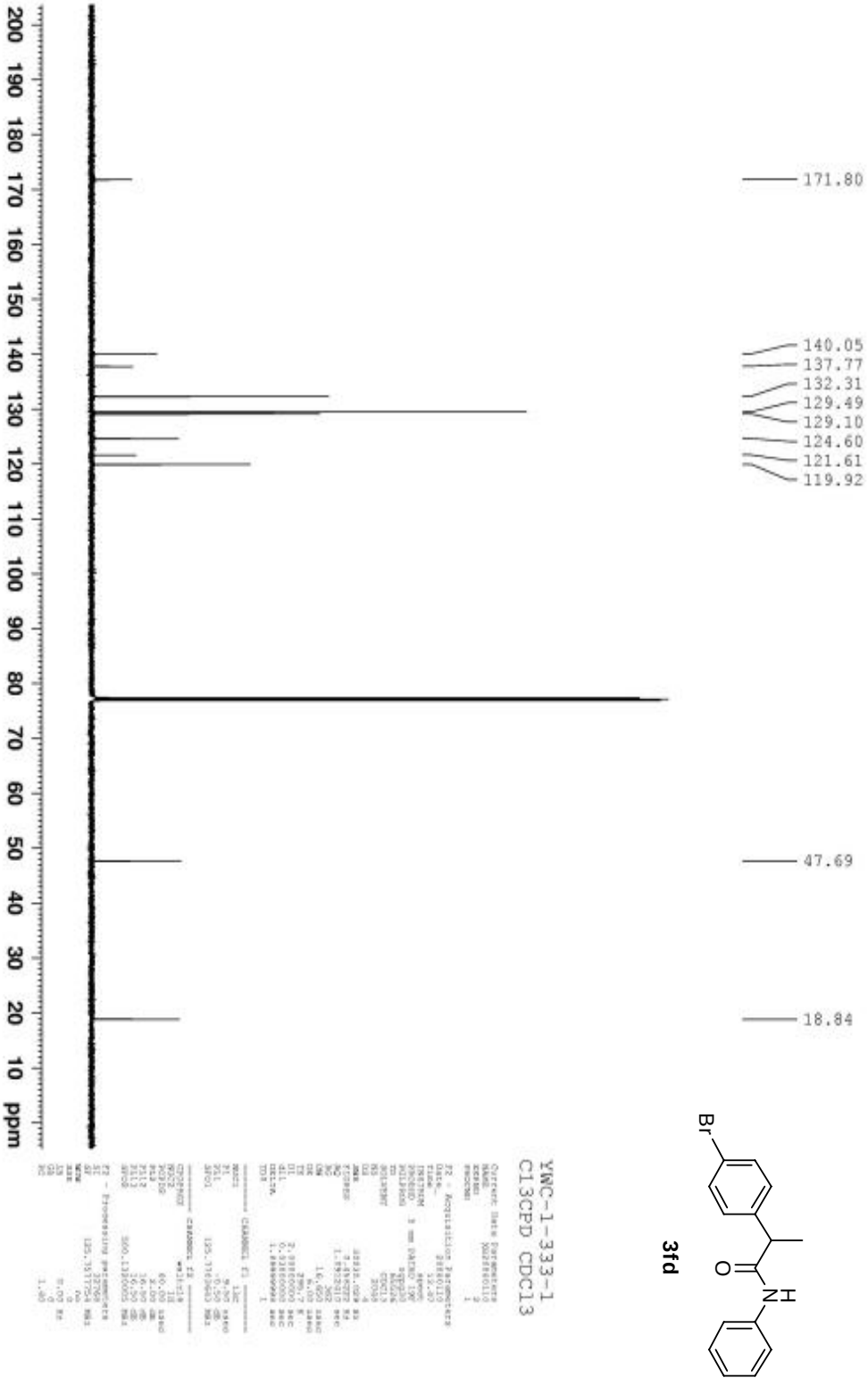
3dd


YWC-1-304-1
PROTON CDCl3
ppm
1.97
2.00
4.01
1.91
0.94
3.00
7.436
7.420
7.351
7.335
7.312
7.296
7.281
7.265
7.099
7.085
7.070
3.697
3.683
3.668
3.654
1.578
1.564
Cl
O
N
H
3ed

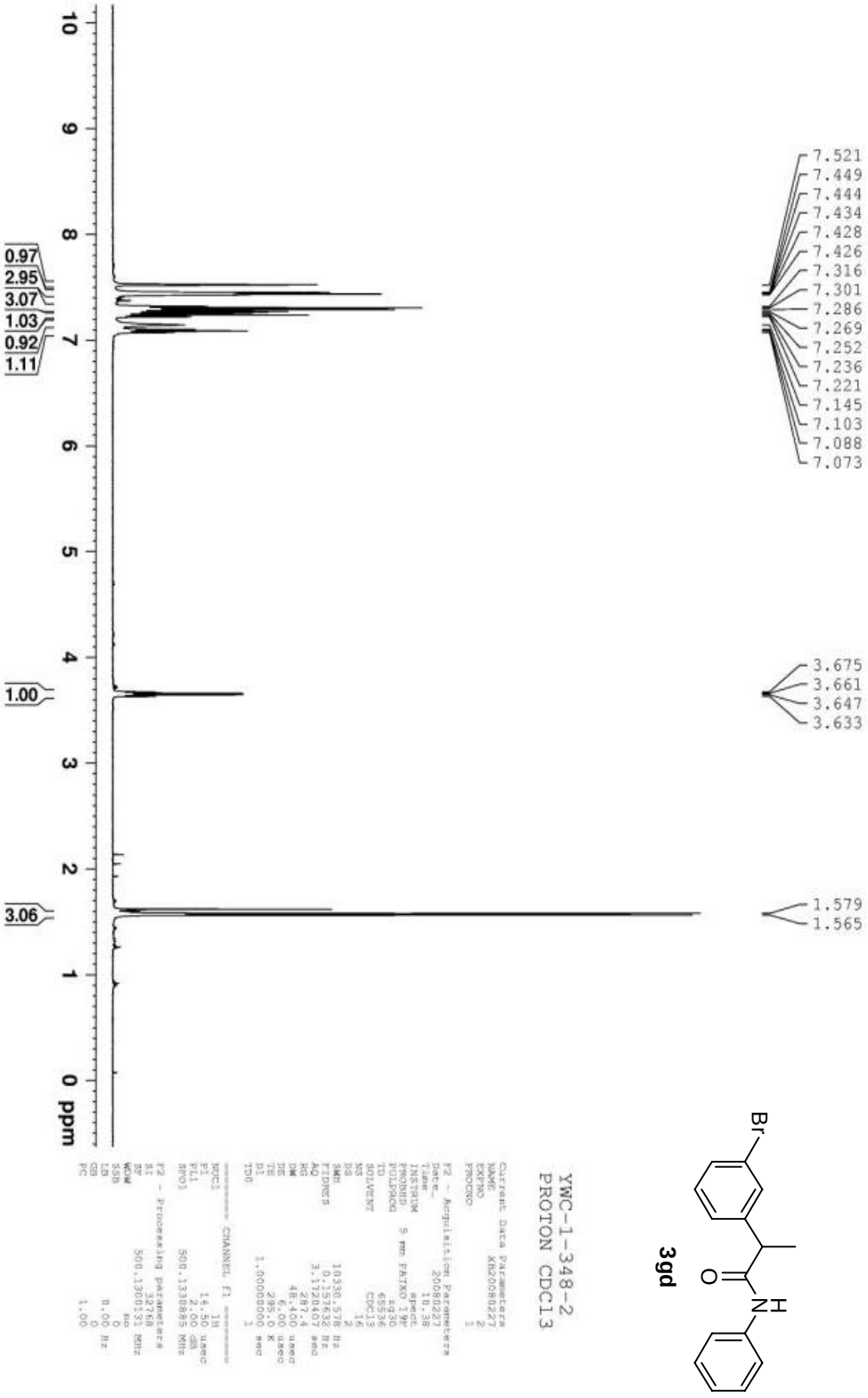

YWC-1-304-1
C13CPD CDCl3
172.15
139.52
137.81
133.39
129.22
129.07
129.04
124.57
120.05
47.40
18.82
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Cl
O
N
H
3ed

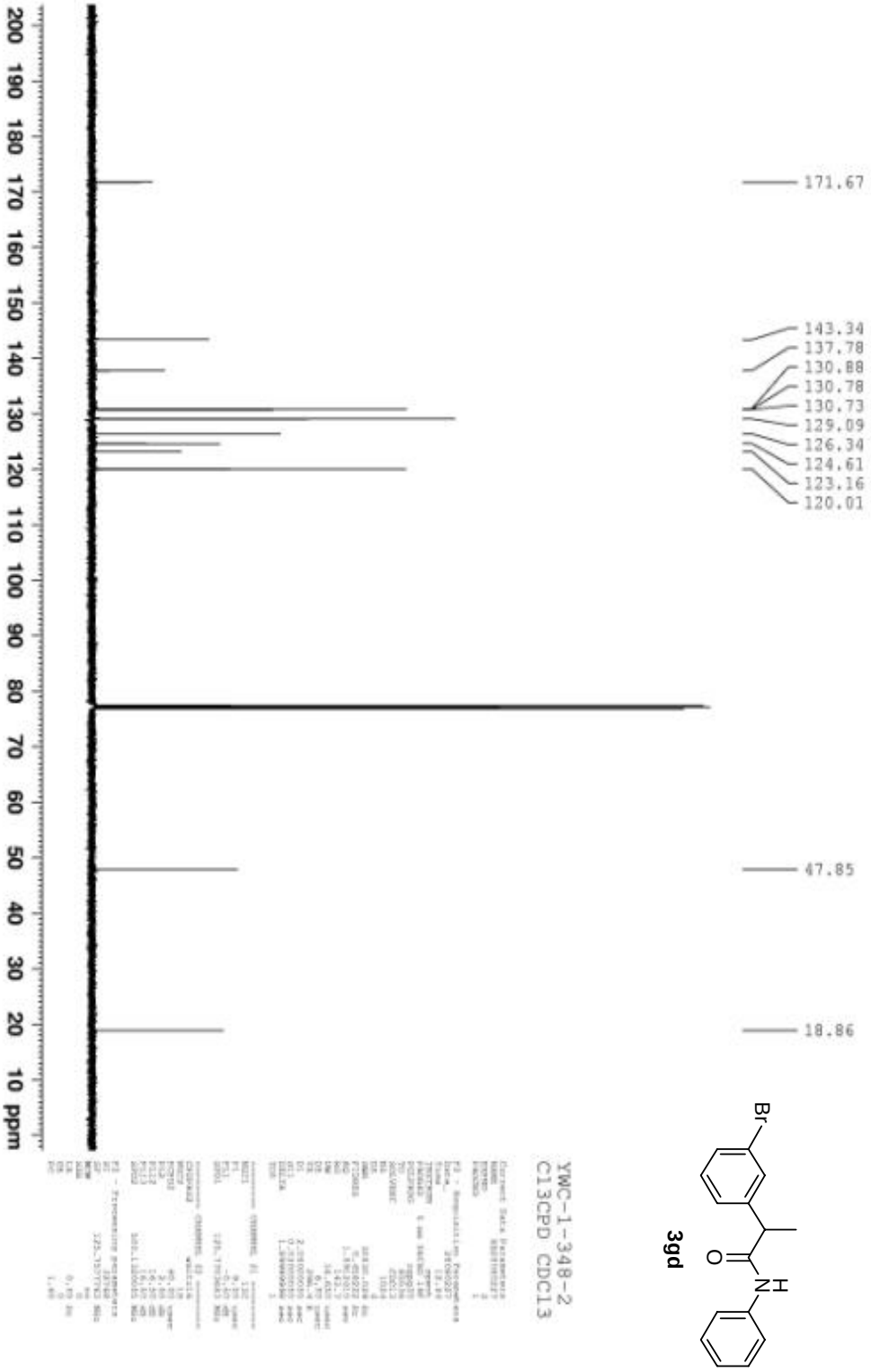

7.521
7.505
7.463
7.447
7.320
7.305
7.288
7.276
7.260
7.185
7.125
7.111
7.097
3.704
3.690
3.676
3.662
1.595
1.581
3.92
4.15
0.98
1.07
1.00
2.97
ppm
YWC-1-333-1
PROTON CDC13
Br
O
H
N

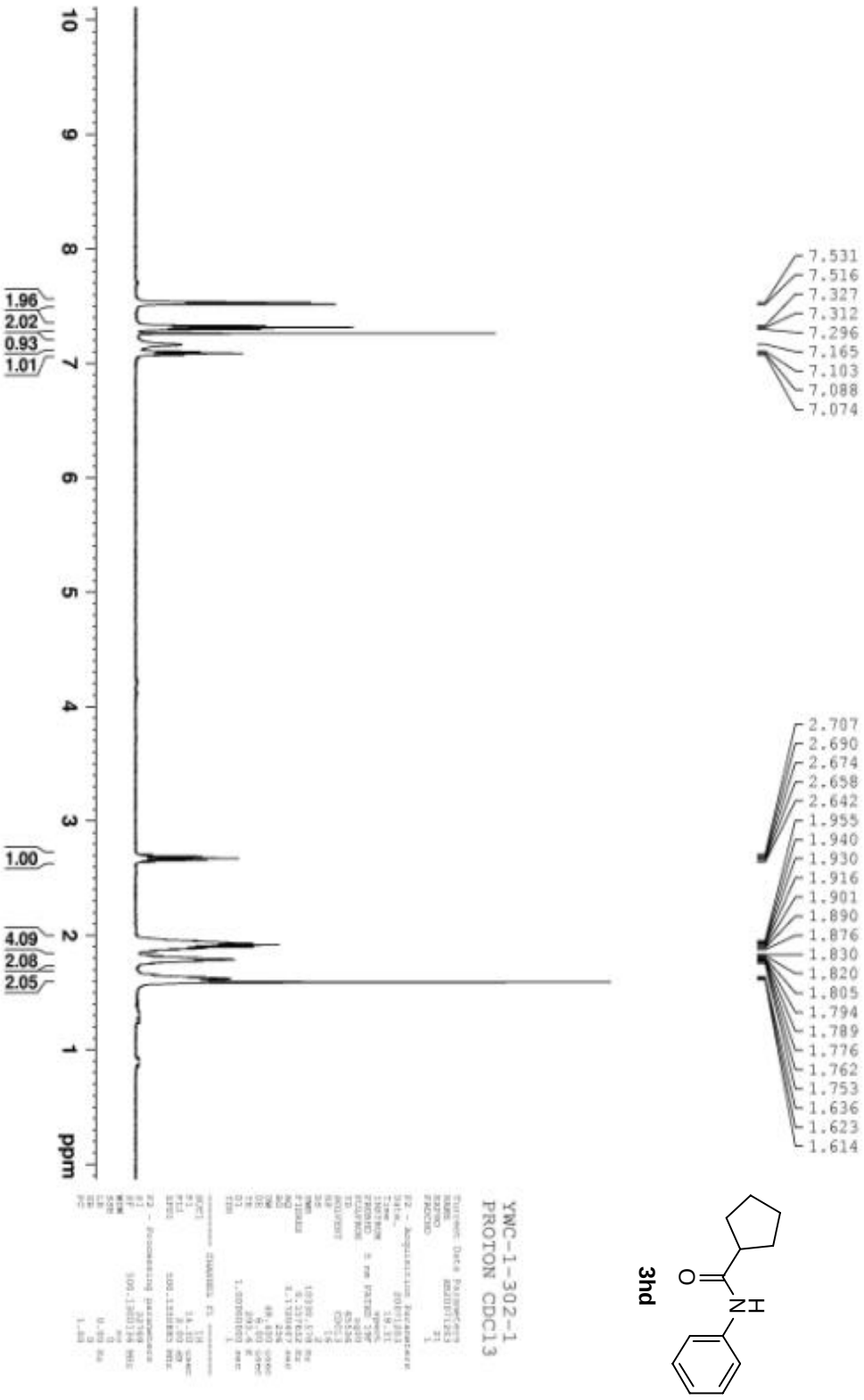
3fd

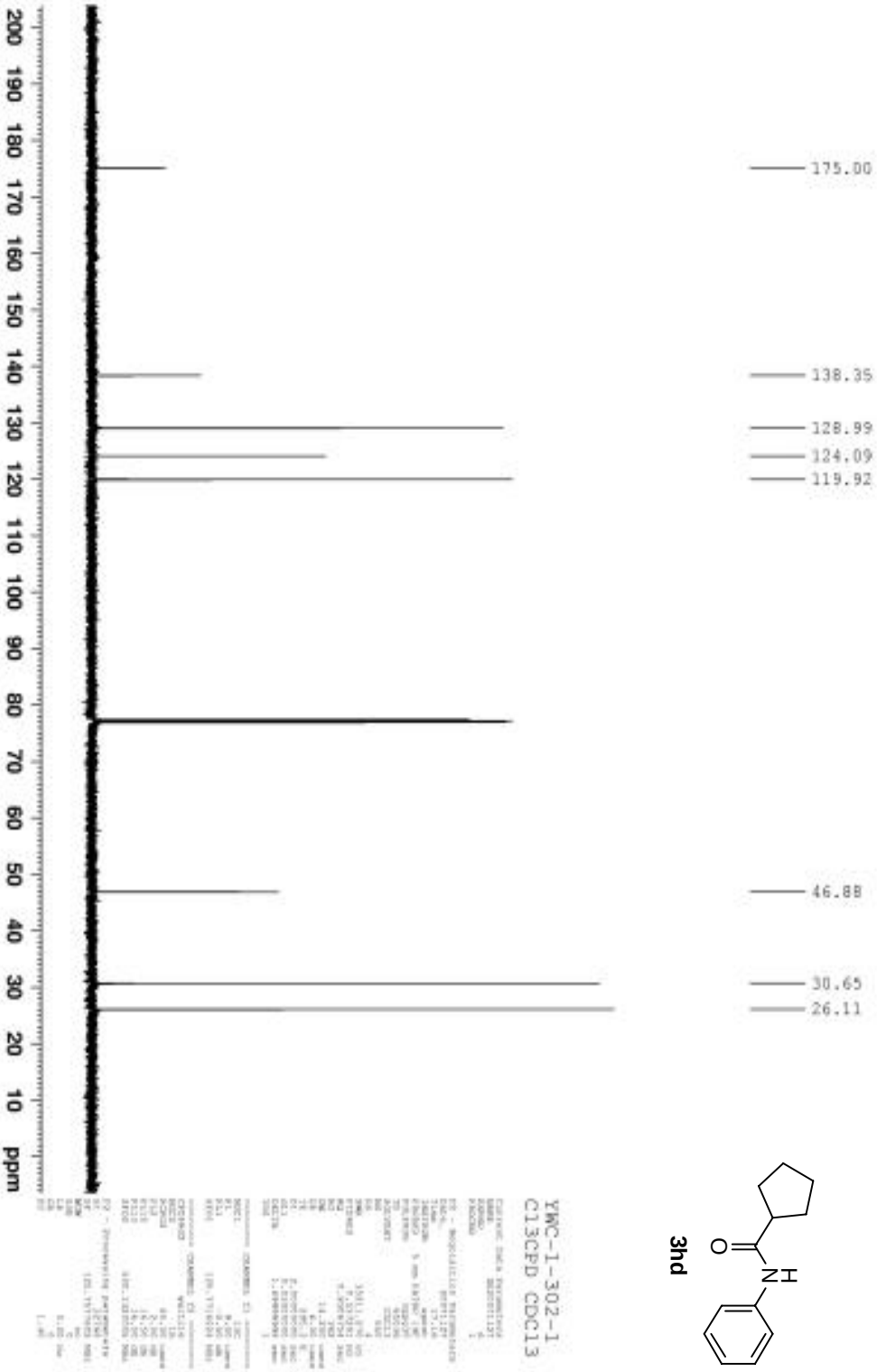

171.80
140.05
137.77
132.31
129.49
129.10
124.60
121.61
119.92
47.69
18.84
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-333-1
C13CPD CDCl3
Br
O
N
H

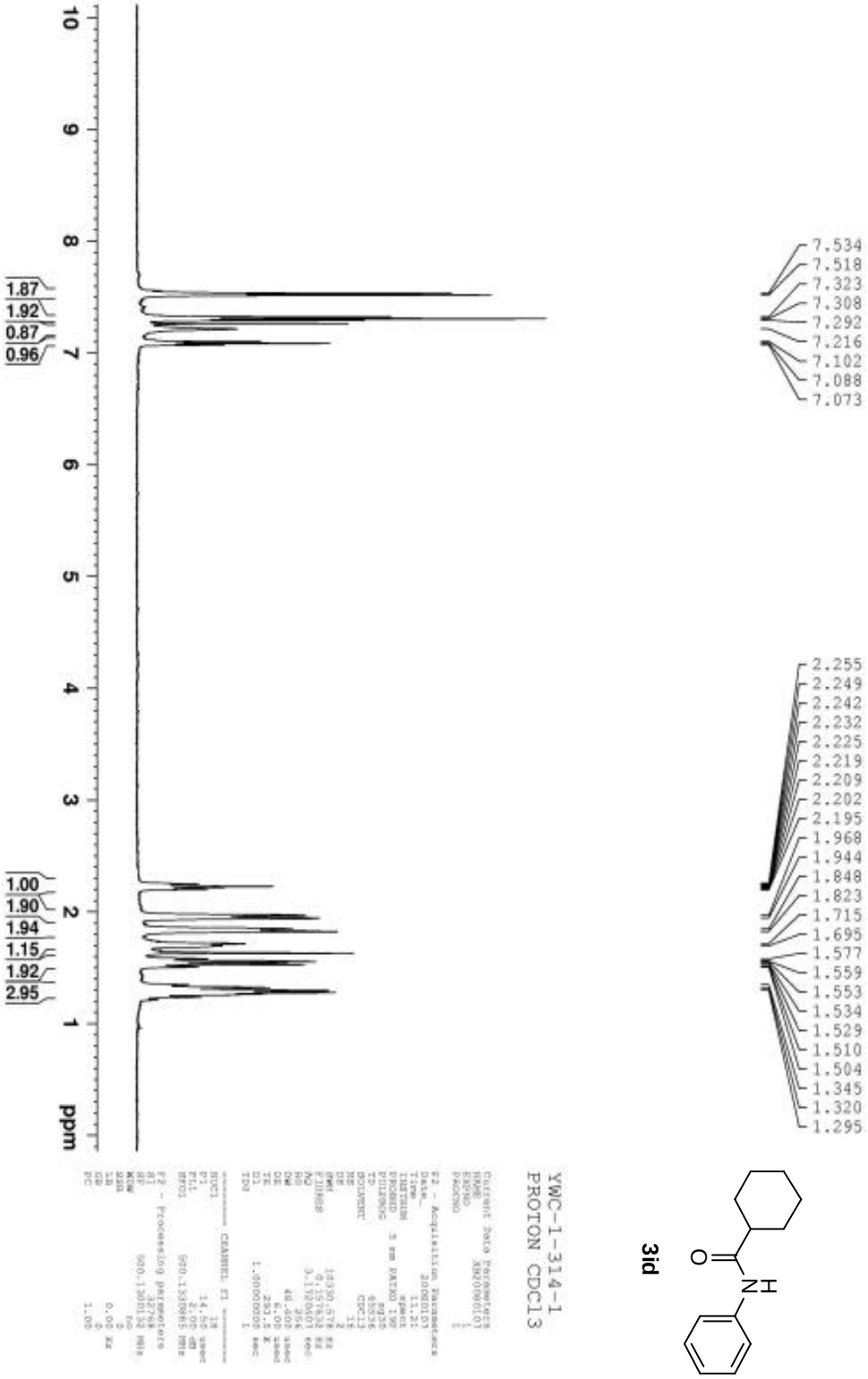
3fd


7.521
7.449
7.444
7.434
7.428
7.426
7.316
7.301
7.286
7.269
7.252
7.236
7.221
7.145
7.103
7.088
7.073
3.675
3.661
3.647
3.633
1.579
1.565
0.97
2.95
3.07
1.03
0.92
1.11
1.00
3.06
10 9 8 7 6 5 4 3 2 1 0 ppm
YWC-1-348-2
PROTON CDC13
Br
O
H
N
3gd

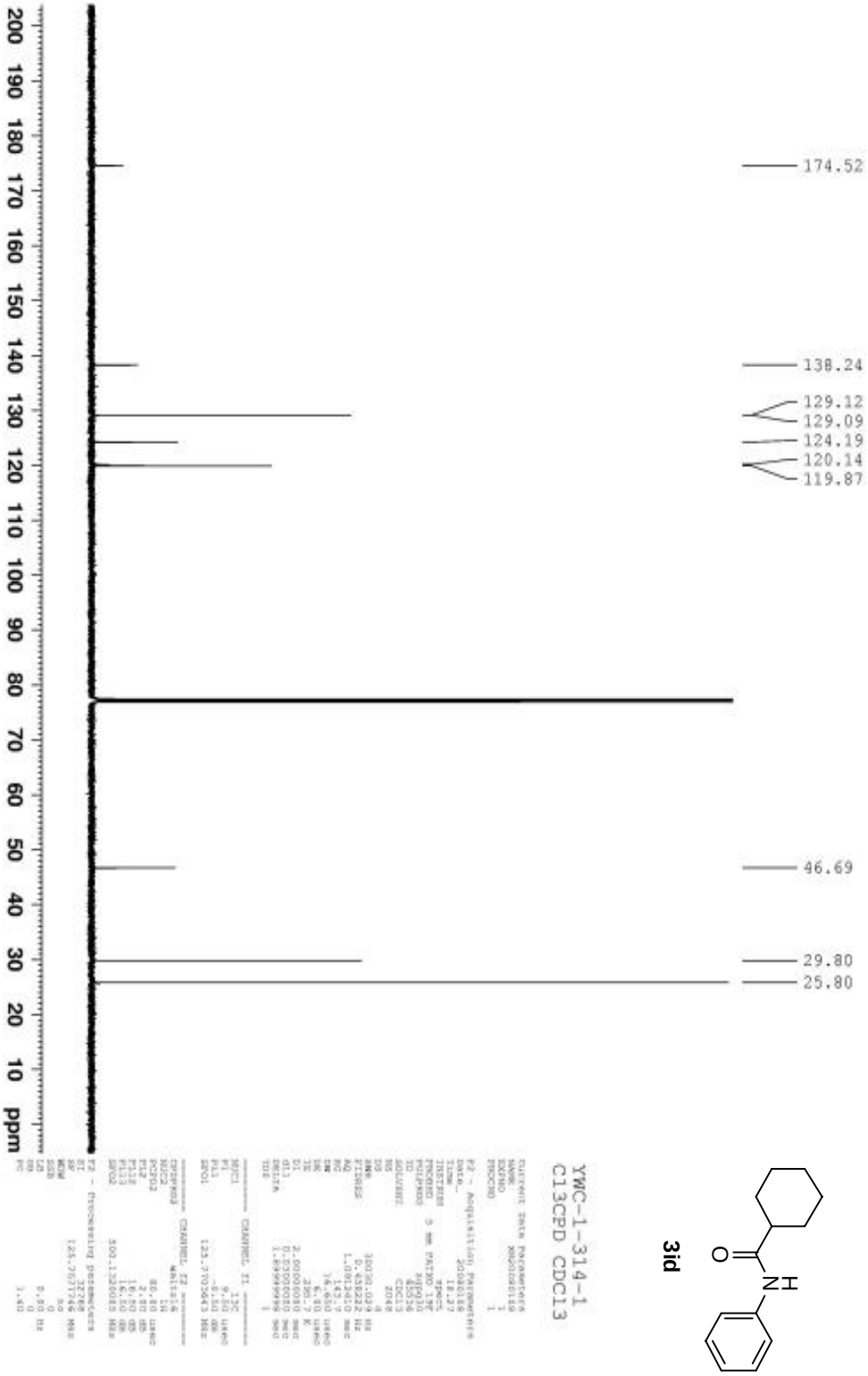

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
171.67
143.34
137.78
130.88
130.78
130.73
129.09
126.34
124.61
123.16
120.01
47.85
18.86
YWC-1-348-2
C13CPD CDCl3
Br
O
H
N
3gd

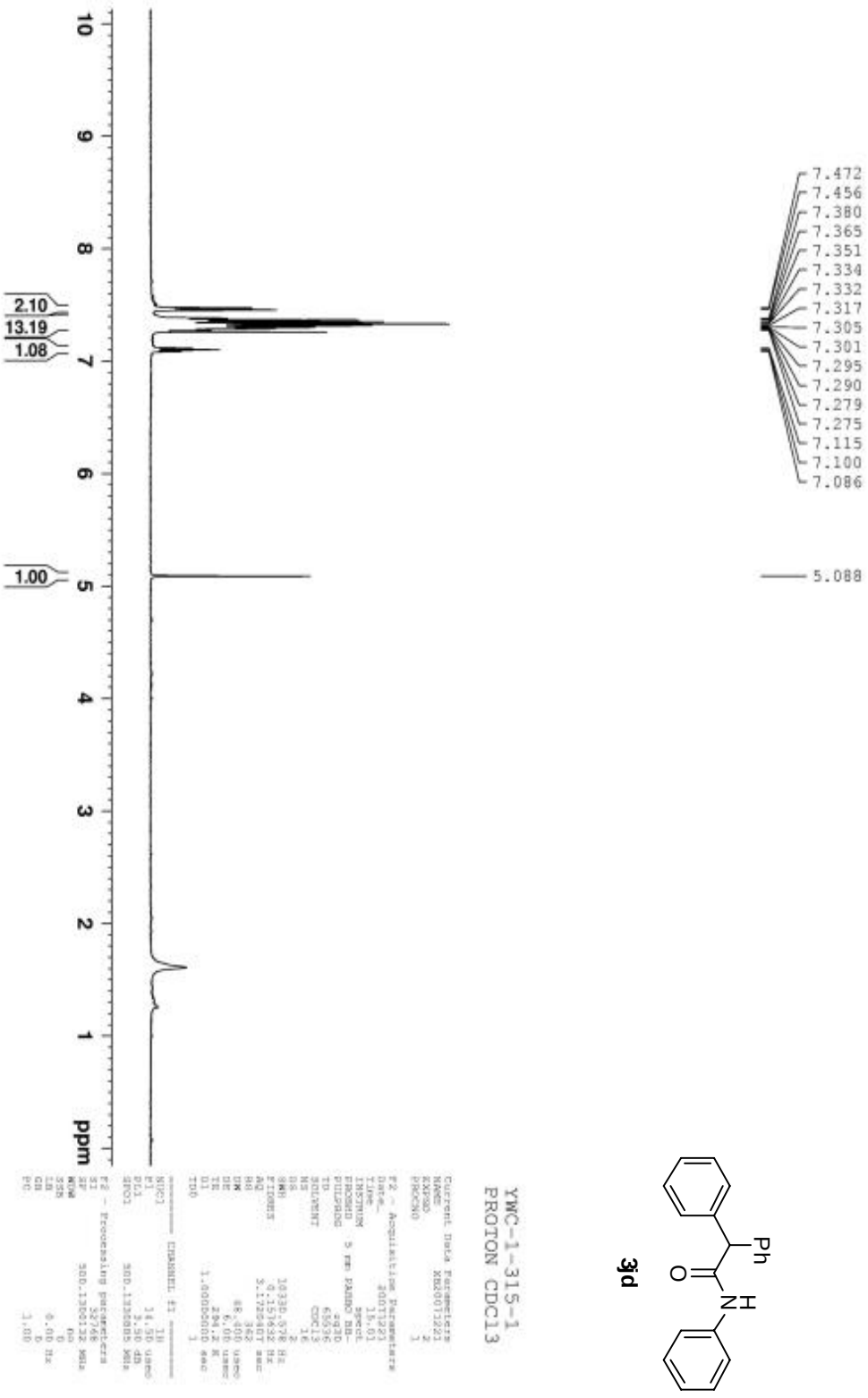

YNC-1-302-1
PROTON CDCl3
ppm
1.96
2.02
0.93
1.01
1.00
4.09
2.08
2.05
7.531
7.516
7.327
7.312
7.296
7.165
7.103
7.088
7.074
2.707
2.690
2.674
2.658
2.642
1.955
1.940
1.930
1.916
1.901
1.890
1.876
1.830
1.820
1.805
1.794
1.789
1.776
1.762
1.753
1.636
1.623
1.614
3hd

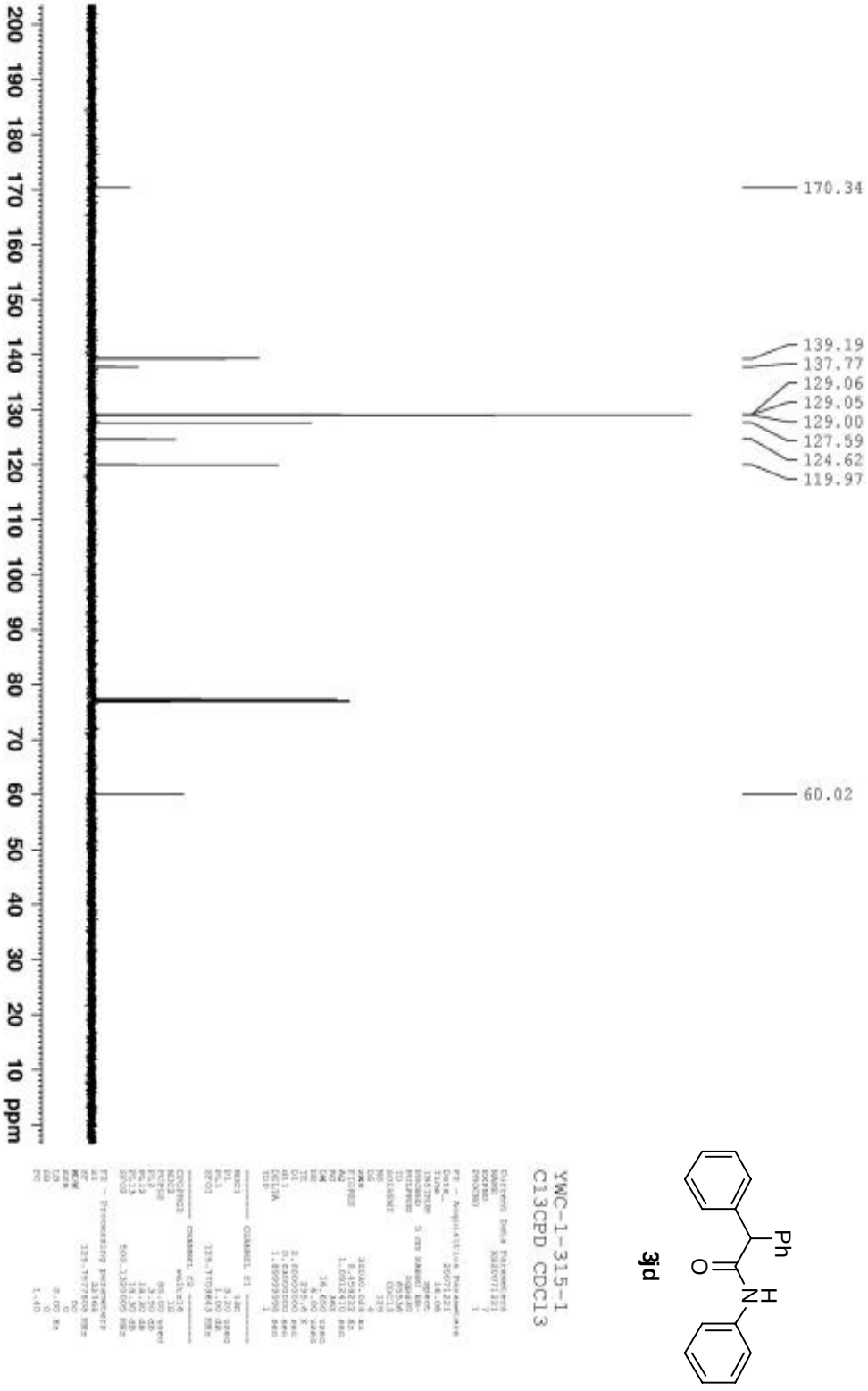

175.00
138.35
128.99
124.09
119.92
46.88
30.65
26.11
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-302-1
C13CPD CDCl3
3hd


7.534
7.518
7.323
7.308
7.292
7.216
7.102
7.088
7.073
2.255
2.249
2.242
2.232
2.225
2.219
2.209
2.202
2.195
1.968
1.944
1.848
1.823
1.715
1.695
1.577
1.559
1.553
1.534
1.529
1.510
1.504
1.345
1.320
1.295
1.87
1.92
0.87
0.96
1.00
1.90
1.94
1.15
1.92
2.95
ppm
YWC-1-314-1
PROTON CDCl3
O
N
H

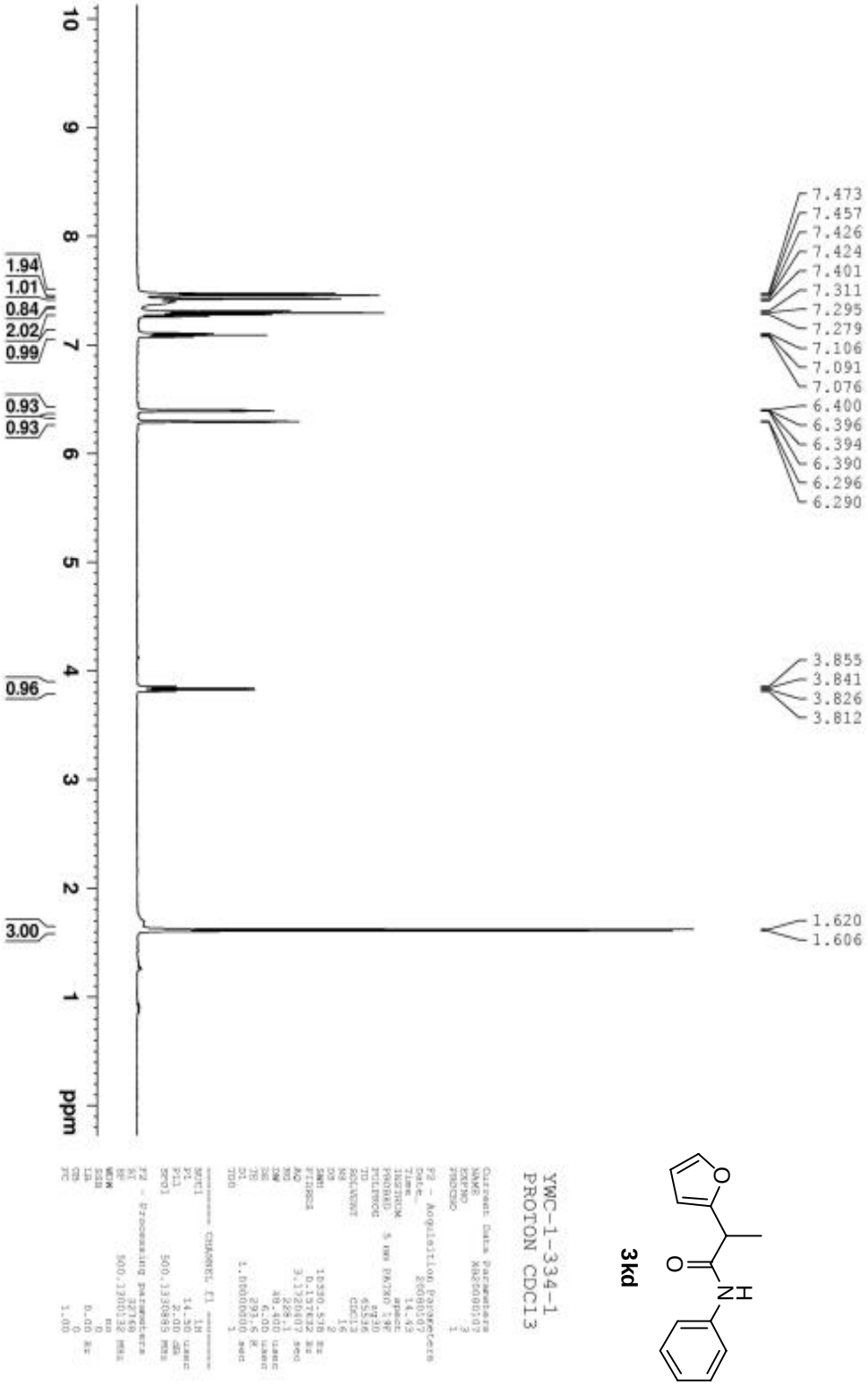
3id

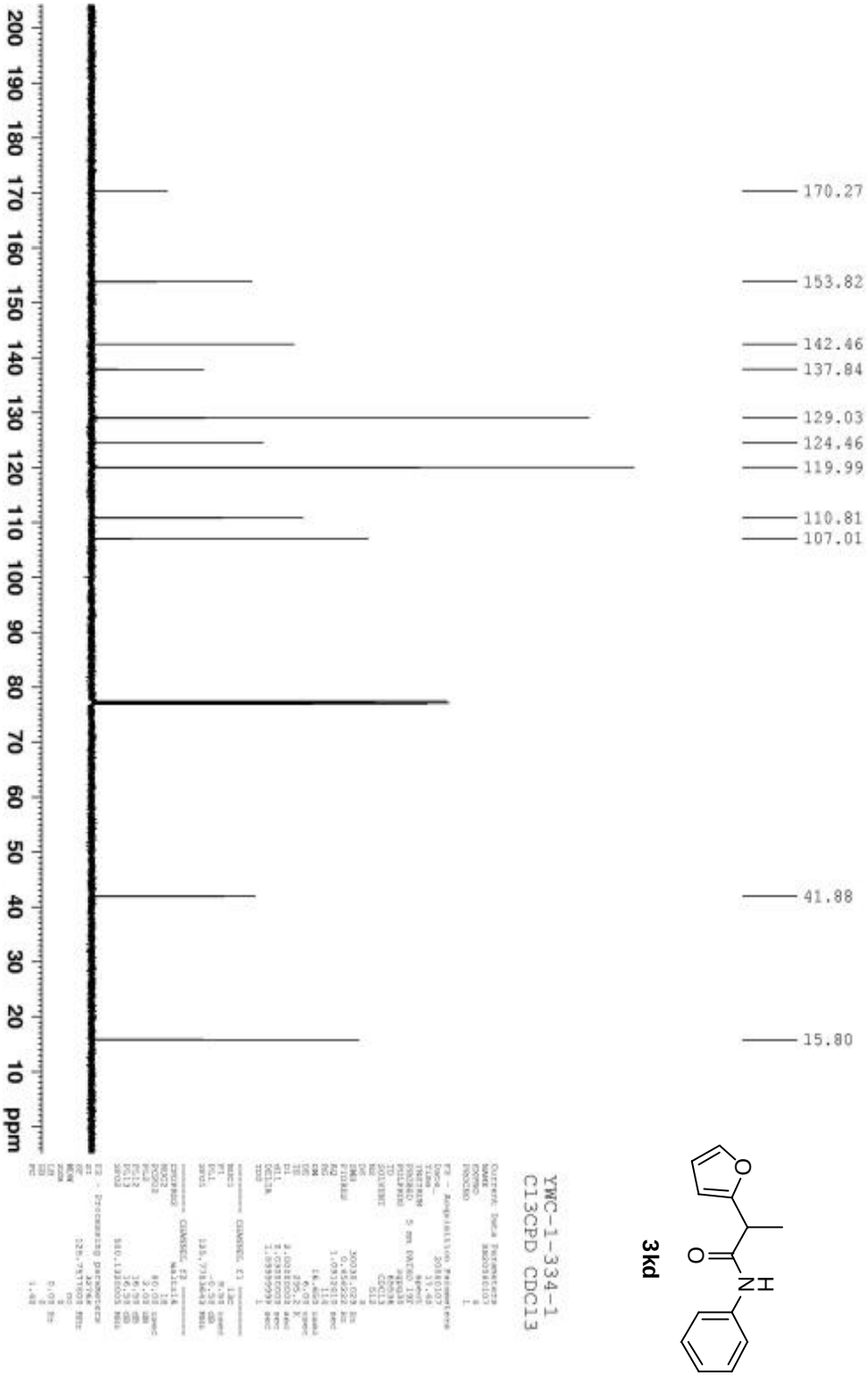

174.52
138.24
129.12
129.09
124.19
120.14
119.87
46.69
29.80
25.80
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-314-1
C13CPD CDCl3
3id
O
N
H


7.472
7.456
7.380
7.365
7.351
7.334
7.332
7.317
7.305
7.301
7.295
7.290
7.279
7.275
7.115
7.100
7.086
5.088
2.10
13.19
1.08
1.00
ppm
YWC-1-315-1
PROTON CDC13
3jd
Ph
O
H
N


170.34
139.19
137.77
129.06
129.05
129.00
127.59
124.62
119.97
60.02
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-315-1
C13CPD CDCl3
Ph
O
N
H

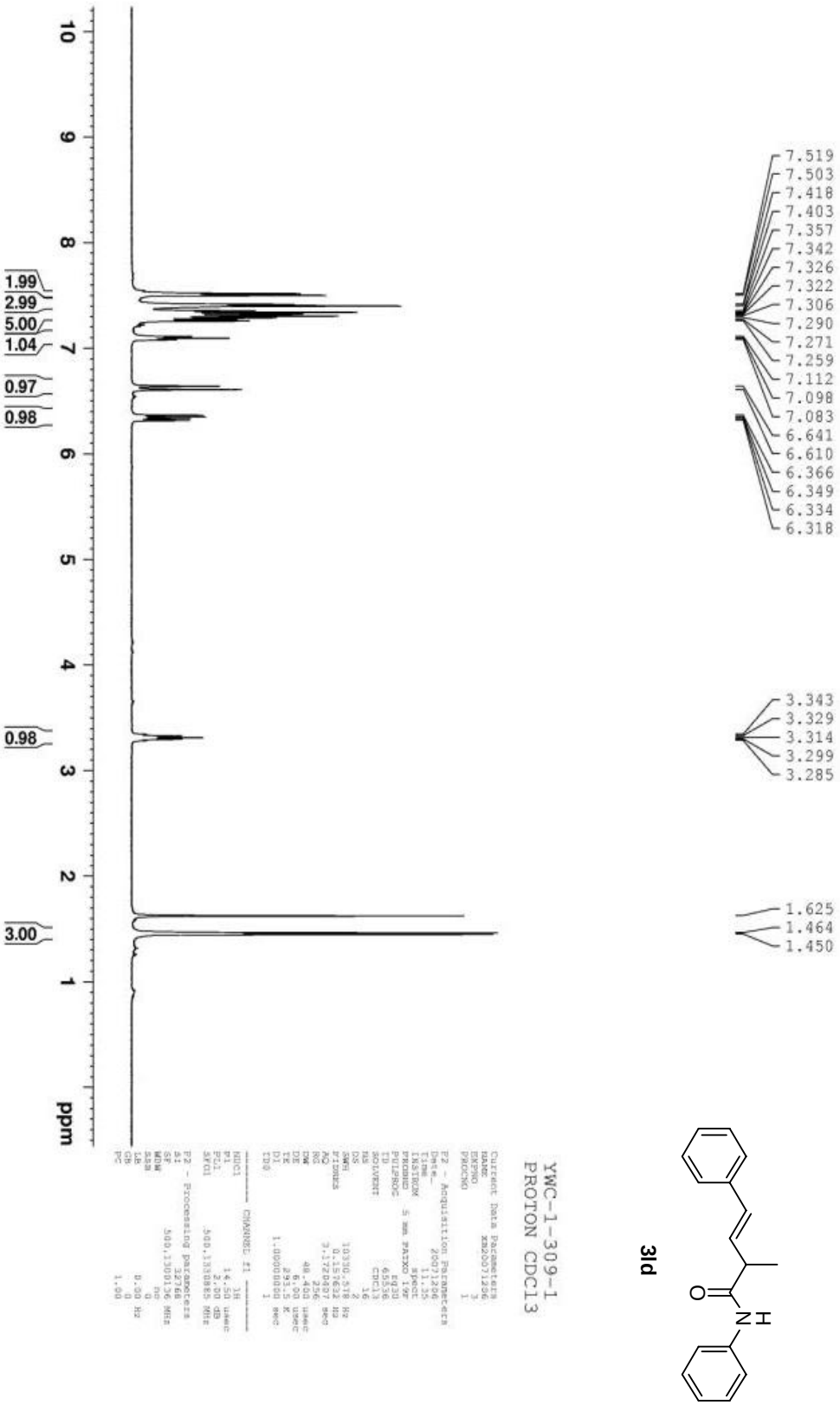
3jd


7.473
7.457
7.426
7.424
7.401
7.311
7.295
7.279
7.106
7.091
7.076
6.400
6.396
6.394
6.390
6.296
6.290
3.855
3.841
3.826
3.812
1.620
1.606
1.94
1.01
0.84
2.02
0.99
0.93
0.93
0.96
3.00
ppm
YWC-1-334-1
PROTON CDC13
3kd

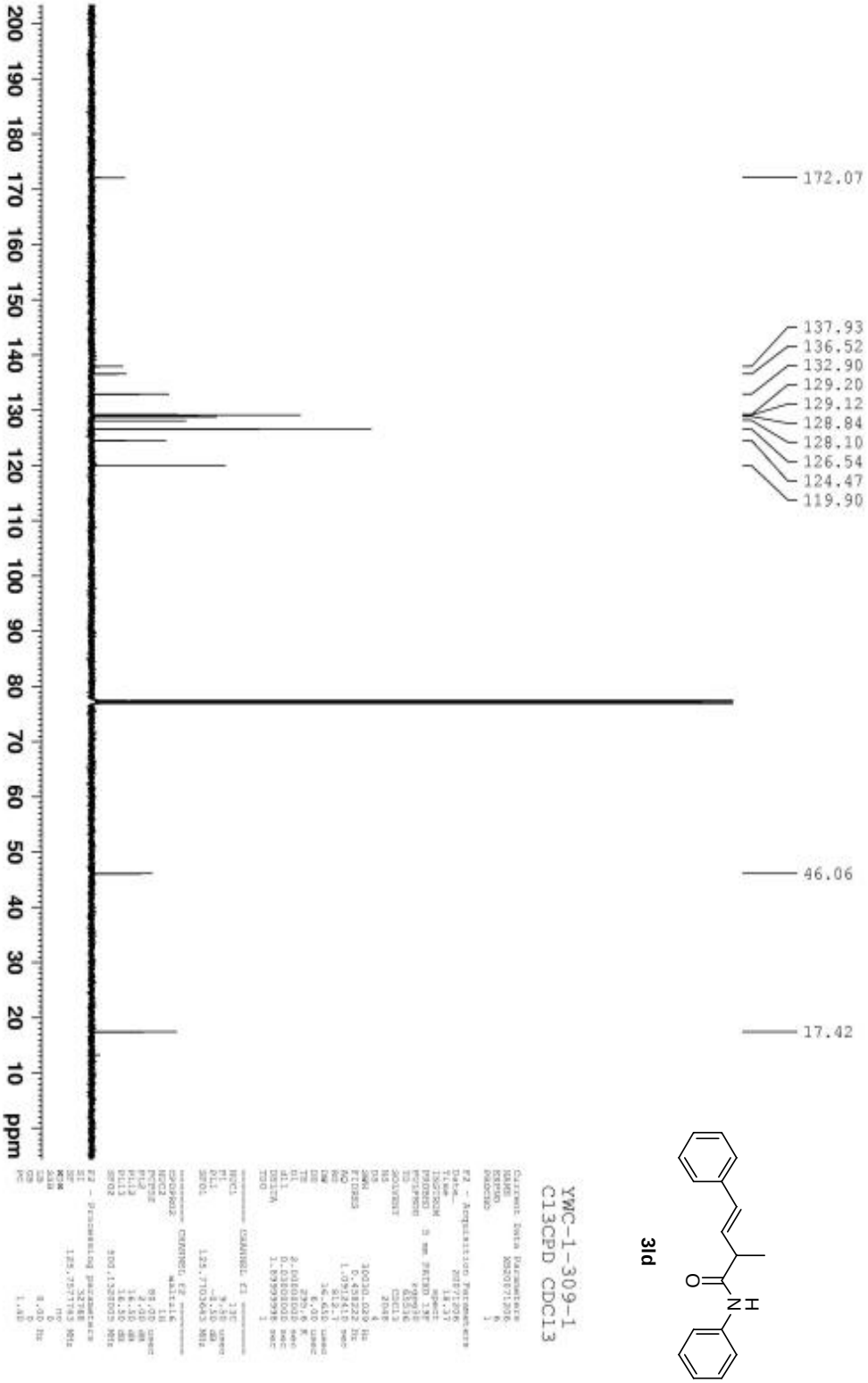

170.27
153.82
142.46
137.84
129.03
124.46
119.99
110.81
107.01
41.88
15.80
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-334-1
C13CPD CDCl3

O
O
NH

3kd


YWC-1-309-1
PROTON CDC13
7.519
7.503
7.418
7.403
7.357
7.342
7.326
7.322
7.306
7.290
7.271
7.259
7.112
7.098
7.083
6.641
6.610
6.366
6.349
6.334
6.318
3.343
3.329
3.314
3.299
3.285
1.625
1.464
1.450
1.99
2.99
5.00
1.04
0.97
0.98
0.98
3.00
ppm

3ld


172.07
137.93
136.52
132.90
129.20
129.12
128.84
128.10
126.54
124.47
119.90
46.06
17.42
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
YWC-1-309-1
C13CPD CDCl3
3ld