Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Xinglin Yang[a,†], Gang Shan[a,†], Zimo Yang[a], Guiyi Huang[a], Guoqiang Dong[b], Chunquan Sheng[b,*], and Yu Rao[a,*]

a. MOE Key Laboratory of Protein Sciences, School of Pharmaceutical Sciences, Tsinghua University, Beijing100084, China. E-mail: yrao@mail.tsinghua.edu.cn

b. School of Pharmacy, Second Military Medical University, 325 Guohe Road, Shanghai 200433, China. E-mail: shengcq@smmu.edu.cn

† These authors contributed equally to this work.

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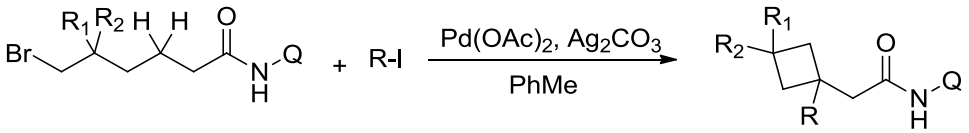
I. Materials and Methods

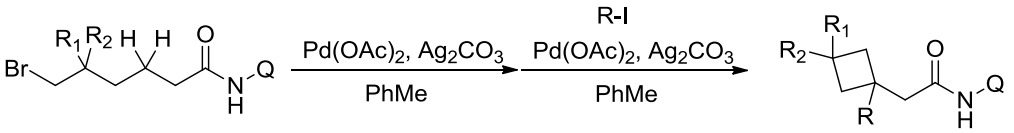
All commercial materials (Alfa Aesar, Aladdin, J&K Chemical LTD.) were used without further purification. All solvents were analytical grade. The ^{1}H-NMR and ^{13}C- NMR spectra were recorded on a Bruker 400 MHz spectrometer in $CDCl_3$ using TMS or solvent peak as a standard. All ^{13}C-NMR spectra were recorded with complete proton decoupling. Low-resolution mass spectral analyses were performed with a Waters AQUITY UPLC^{TM}/MS. High-resolution mass spectral analyses were performed with a Bruker Impact HD/QTOF. All reactions were carried out in oven-dried sealed tube. Analytical TLC was performed on Yantai Chemical Industry Research Institute silica gel 60 F254 plates and flash column chromatography was performed on Qingdao Haiyang Chemical Co. Ltd silica gel 60 (200-300 mesh). The rotavapor was BUCHI's Rotavapor R-3. Amides were easily synthesized from acyl chlorides and 8-aminoquinoline.

II. General procedure for cascade C(sp3)-H bond activation

General procedure I: Arylation and alkenylation

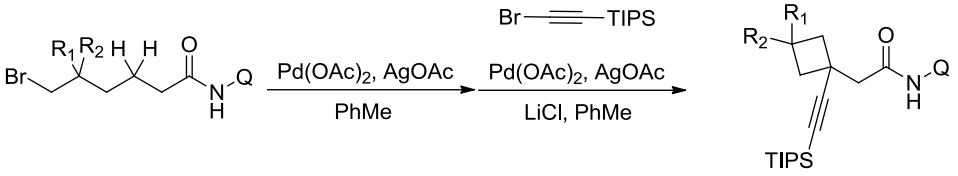


To a 15ml sealed-tube were added amide (1.0 equiv), aryl iodide or alkenyl iodide (2.0 equiv), $Pd(OAc)_2$ (0.01 equiv), Ag_2CO_3(2.0 equiv) and PhMe (2 ml). The tube was sealed and heated at 100 ^{o}C for 6-12h. The reaction was monitored by TLC (Petroleum ether: Ethyl acetate = 4:1) and LC-MS. The reaction mixture was cooled to RT, dichloromethane was added to dilute the reaction mixture and saturated aqueous $NaHCO_3$ was added to wash the reaction mixture. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.



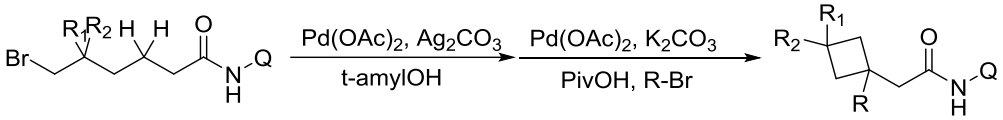
To a 15ml sealed-tube were added amide (1.0 equiv), $Pd(OAc)_2$ (0.01 equiv), Ag_2CO_3(1.0 equiv) and PhMe (2 ml). The tube was sealed and heated at 100 ^{o}C for 4h. Then aryl iodide or alkenyl iodide (2.0 equiv), $Pd(OAc)_2$ (0.01 equiv) and Ag_2CO_3(1.0 equiv) were added. The tube was sealed and heated at 100 ^{o}C. The reaction was monitored by TLC (Petroleum ether: Ethyl acetate = 4:1) and LC-MS. The reaction mixture was cooled to RT, dichloromethane was added to dilute the reaction mixture and saturated aqueous $NaHCO_3$ was added to wash the reaction mixture. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

General procedure II: Alkynylation



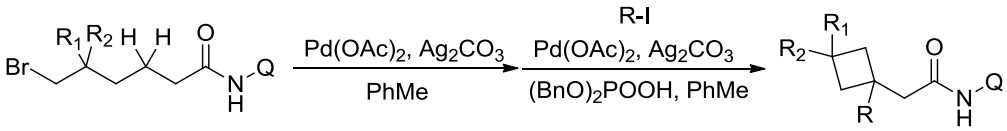
To a 15ml sealed-tube were added amide (1.0 equiv), $Pd(OAc)_2$ (0.01 equiv), AgOAc (2.0 equiv) and PhMe (2 ml). The tube was sealed and heated at 100 ^oC for 4h. Then (bromoethynyl)triisopropylsilane (2.0 equiv), $Pd(OAc)_2$ (0.01 equiv), AgOAc (2.0 equiv) and LiCl (1.0 equiv) were added. The tube was sealed and heated at 100 ^oC. The reaction was monitored by TLC (Petroleum ether: Ethyl acetate = 4:1) and LC-MS. The reaction mixture was cooled to RT, dichloromethane was added to dilute the reaction mixture and saturated aqueous $NaHCO_3$ was added to wash the reaction mixture. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

General procedure III: Allylation and benzylation



To a 15ml sealed-tube were added amide (1.0 equiv), $Pd(OAc)_2$ (0.01 equiv), Ag_2CO_3(2.0 equiv) and t-amylOH (2 ml). The tube was sealed and heated at 100 ^oC for 12 h. Then allyl bromide or benzyl bromide (2.0 equiv), $Pd(OAc)_2$ (0.01 equiv), PivOH (1.0 equiv) and K_2CO_3(2.0 equiv) were added. The tube was sealed and heated at 100 ^oC for another 12 h. The reaction was monitored by TLC (Petroleum ether: Ethyl acetate = 4:1) and LC-MS. The reaction mixture was cooled to RT, dichloromethane was added to dilute the reaction mixture and saturated aqueous $NaHCO_3$ was added to wash the reaction mixture. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

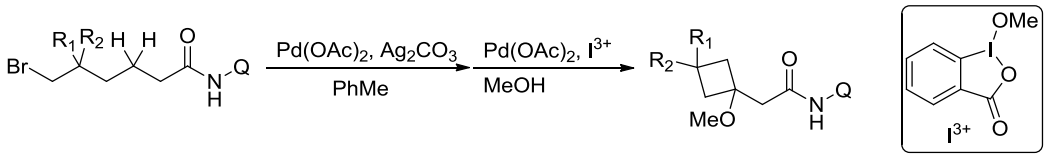
General procedure IV: Alkylation



To a 15ml sealed-tube were added amide (1.0 equiv), $Pd(OAc)_2$ (0.01 equiv), Ag_2CO_3(1.0 equiv**)** and PhMe (2 ml). The tube was sealed and heated at 100 ^oC for 4h. Then alkyl iodide (2.0 equiv**)**, $Pd(OAc)_2$ (0.01 equiv), $(BnO)_2POOH$ (0.3 equiv) and Ag_2CO_3(1.0 equiv) were added. The tube was sealed and heated at 100 ^oC. The reaction was monitored by TLC (Petroleum ether: Ethyl acetate = 4:1) and LC-MS. The reaction mixture was cooled to RT, dichloromethane was added to dilute the

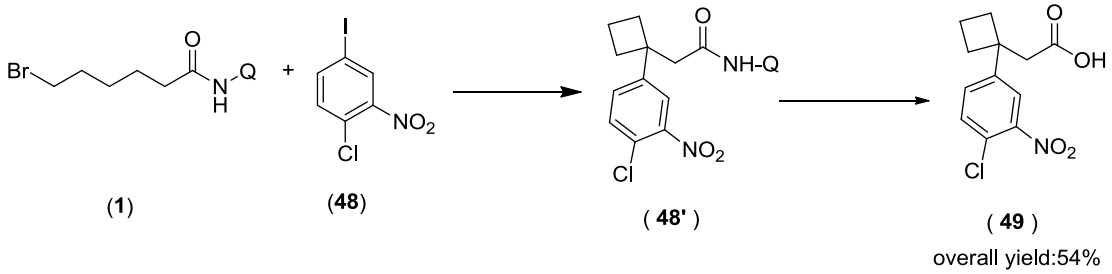
reaction mixture and saturated aqueous $NaHCO_3$ was added to wash the reaction mixture. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

General procedure V: Methoxylation



To a 15ml sealed-tube were added amide (1.0 equiv), $Pd(OAc)_2$ (0.01 equiv), Ag_2CO_3(1.0 equiv**)** and PhMe (1 ml). The tube was sealed and heated at 100 ^oC for 4h. Then I^{3+} (2.0 equiv**)**, $Pd(OAc)_2$ (0.01 equiv) and MeOH (5 ml) were added. The tube was sealed and heated at 100 ^oC. The reaction was monitored by TLC (Petroleum ether: Ethyl acetate = 4:1) and LC-MS. The reaction mixture was cooled to RT, dichloromethane was added to dilute the reaction mixture and saturated aqueous $NaHCO_3$ was added to wash the reaction mixture. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product.

III. General procedure for deprotection



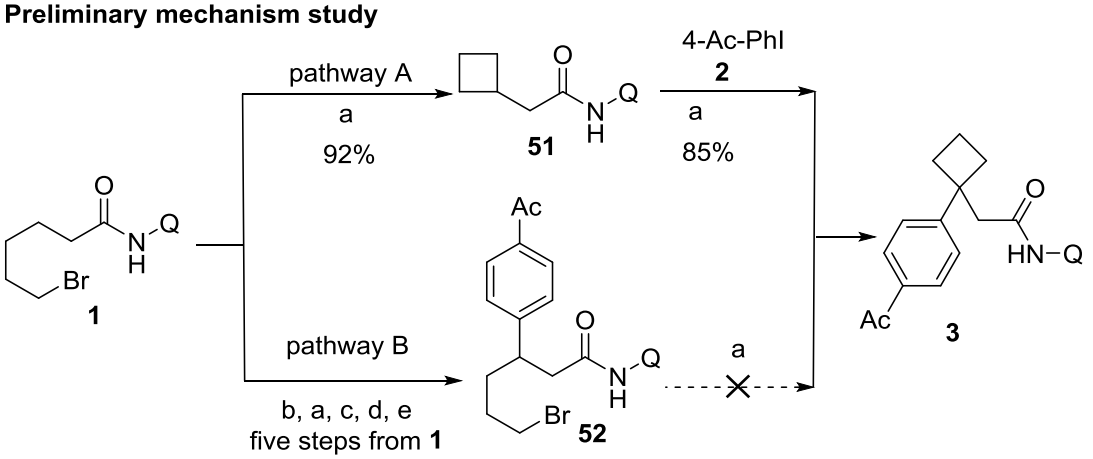
To a 15ml sealed-tube were added amide （**1**） (106mg, 0.33mmol), $Pd(OAc)_2$ (7 mg, 0.01 equiv), Ag_2CO_3(85mg, 1.0 equiv) and PhMe (2 ml). The tube was sealed and heated at 100 ^oC for 4 h. Then aryl iodide (**48**) (187 mg, 2.0 equiv), $Pd(OAc)_2$ (7mg, 0.01 equiv) and Ag_2CO_3(85mg, 1.0 equiv) were added. The tube was sealed and heated at 100 ^oC for 12 h. The reaction was monitored by TLC (Petroleum ether: Ethyl acetate = 4:1) and LC-MS. The reaction mixture was cooled to RT, dichloromethane was added to dilute the reaction mixture and saturated aqueous NaHCO3 was added to wash the reaction mixture. Then the organic layer was dried over anhydrous Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally, the residue was purified by silica gel column chromatography to give the desired product (**48'**) (98 mg) in 75% yield.

Compound **(48')** (98 mg, 0.25 mmol), Boc_2O (545 mg, 2.5 mmol), DMAP (95 mg, 0.78 mmol, 2 eq) were mixed with 3 mL of THF. The resulting mixture was heated at 50°C for 3h. Then, KOH (140 mg, 2.5 mmol) in 3ml H_2O and 3ml MeOH was added to

the reaction mixture at rt, stirred for 30 min. The reaction mixture was neutralized with HCl, extract with EtOAc, dried over anhydrous Na_2SO_4 , concentrated on rotavapor under reduced pressure and purified by silica gel column chromatography to give the corresponding acid **(49)** (48 mg) in 72% yield.

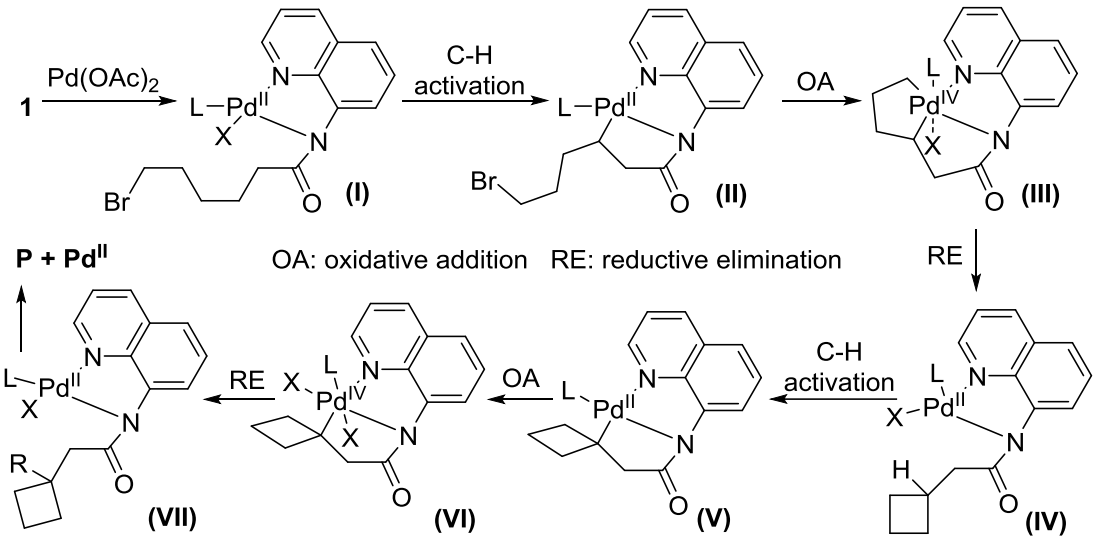
IV. Mechanism Study

Preliminary mechanism study



Conditions: (a) 10% $Pd(OAc)_2$, 1.0 equiv. Ag_2CO_3, PhMe, 100 ^oC ; (b) 2 equiv. AgOAc, DMF, 80 ^oC, 12 h; (c) NaOH, EtOH/H_2O, rt, 6 h; (d) 1.5 equiv. $PhSO_2Cl$, 1equiv.4-DMAP, 2 equiv. Et_3N, DCM, rt, 4 h; (e) 2 equiv. NaBr, DMF, 50 ^oC, 10 h

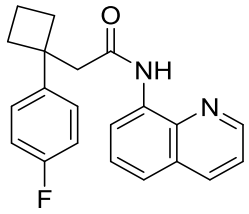
Plausible mechanism



The mechanistic study was conducted to confirm the proposed pathway A and exclude the possibility of another potential pathway B as shown in scheme. Specially, starting from 6-bromohexanamide **1**, cyclobutane **51** was isolated in 92% yield under optimal reaction conditions in the absence of ArI. This cyclobutane intermediate could react with 4-Ac-PhI to give corresponding product **3** in 85% yield under optimal reaction conditions. In contrast, another possible intermediate **52**, prepared through five steps from 6-bromohexanamide **1**， was totally inert under optimal reaction conditions. These results strongly favor pathway A, which involves a sequence of Pd(II)-catalyzed methylene C-H bond alkylation and methine C-H bond arylation.

Based on the above results, a plausible mechanism was proposed as followings. When $Pd(OAc)_2$ and 6-bromohexanamide **1** were mixed together, intermediate **(I)** formed through ligand exchanges, which could transform to bicyclic palladium complex **(II)** through C(sp^3)-H bond activation. Bicyclic intermediate **(II)** then transformed to tricyclic intermediate **(III)** through intramolecular oxidative addition. This Pd(IV) intermediate **(III)** could afford the cyclobutane intermediate **(IV)**. Then a second run of C(sp^3)-H bond acti-vation, oxidative addition and reductive elimination happened to give the final the quaternary carbon centered cyclobutane product.

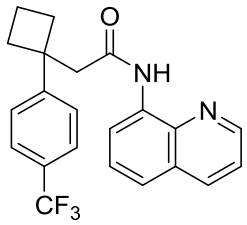
V. Data of products



2-(1-(4-Fluorophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (4)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 1-fluoro-4-iodobenzene (133 mg, 0.6 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3 (83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 ^oC for 13 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(4)** (68.1 mg) was isolated in 68% yield. Rf = 0.3(petroleum ether: ethyl acetate=10: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.20 (s, 1H), 8.65 - 8.62 (m, 2H), 8.08 (dd, *J* = 1.32 Hz, *J* = 8.24 Hz, 1H), 7.48 -7.36 (m, 3 H), 7.21 (dd, *J* = 3.12 Hz, *J* = 8.56 Hz, 2H), 6.88 (t, *J* = 1.32 Hz, 2H), 3.00 (s, 2H), 2.54 - 2.26 (m, 4H), 2.26 - 2.17 (m, 1H), 1.96 - 1.88 (m ,1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.6, 161.3 (d, *J* = 243.3 Hz), 148.0, 144.5 (d, *J* = 3.0 Hz), 138.2, 136.2, 134.4, 127.9, 127.5, 127.4 (d, *J* = 4.4 Hz), 121.6, 121.4, 116.3, 115.1 (d, J = 21.3 Hz), 51.0, 45.4, 33.3, 16.0; HRMS (ESI) calcd for $C_{21}H_{20}FN_2O$ [M+H]+: 335.15597, found 335.15711

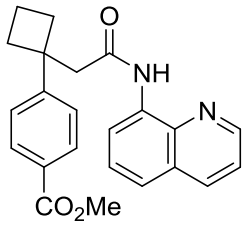


N-(quinolin-8-yl)-2-(1-(4-(trifluoromethyl)phenyl)cyclobutyl)acetamide (5)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 1-iodo-4-(trifluoromethyl)benzene (163 mg, 0.6 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3 (83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 ^oC for 13 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(5)** (83 mg) was isolated in 72% yield. Rf = 0.4(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.17 (s, 1H), 8.63 (dd, *J* = 1.48 Hz, *J* = 6.96 Hz, 1H), 8.57

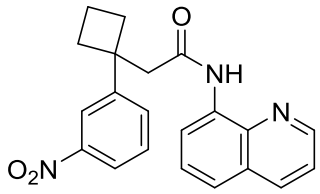
(d, *J* = 2.92 Hz, 1H), 8.08 (dd, *J* = 1.04 Hz, *J* = 8.24 Hz, 1H), 7.49-7.42 (m, 4H), 7.39-7.34 (m, 3H), 3.04 (s, 2H), 2.60-2.48 (m , 4H), 2.30-2.21 (m, 1H), 1.98-1.89 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.3, 153.0, 148.0, 138.2, 136.3, 134.2, 128.0, 127.9, 127.3, 126.3, 125.4 (q, *J* = 3.68 Hz), 124.4 (q, *J* = 270.26 Hz), 121.6, 121.5, 116.3, 50.6, 45.8, 33.1, 16.1; HRMS (ESI) calcd for $C_{22}H_{19}F_3N_2O$ [M+H]+: 385.15277, found 385.15445



methyl 4-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)benzoate (6)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 °C for 4h. Then methyl 4-iodobenzoate (157 mg, 0.6mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3 (83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 °C for 11 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(6)** (85 mg) was isolated in 76% yield.
Rf = 0.2(petroleum ether: ethyl acetate=5: 1)

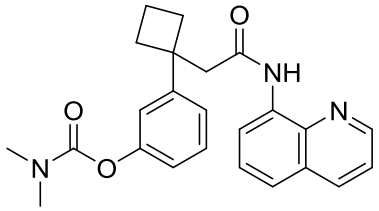
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.19 (s, 1H), 8.62 - 8.55 (m, 2H), 8.05 (d, *J* = 8.24 Hz, 1H), 7.88 -7.86 (m, 2 H), 7.46-7.39 (m, 2H), 7.35-7.30 (m, 3H), 3.82 (s, 3H), 3.03 (s, 2H), 2.56-2.52 (m, 4H), 2.27-2.16 (m, 1H), 1.96-1.90 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.3, 167.1, 154.2, 147.9, 138.2, 136.2, 134.2, 129.8, 127.8, 127.3, 125.9, 121.43, 121.41, 116.3, 51.9, 50.5, 45.9, 33.1, 16.1; HRMS (ESI) calcd for $C_{23}H_{22}N_2O_3$ [M+H]+: 375.17087, found 375.17165



2-(1-(3-nitrophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (7)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 °C for 4h. Then 1-iodo-3-nitrobenzene (149 mg, 0.6mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3 (83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 °C for 10 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(7)** (71 mg) was isolated in 65% yield.
Rf = 0.19(petroleum ether: ethyl acetate=5: 1)

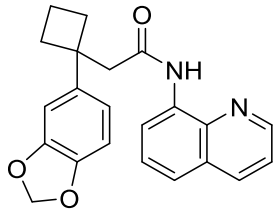
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.25 (s, 1H), 8.59 (d, *J* = 4.4 Hz, 2H), 8.14 (s, 1H), 8.74 (d, *J* = 8.1 Hz, 1H), 7.90 (d, *J* = 8.1 Hz, 1H), 7.58 (d, *J* = 7.7 Hz, 1H), 7.47-7.29 (m, 4H), 3.07 (s, 2H), 2.62-2.50 (m, 4H), 2.29-2.21 (m, 1H), 1.99-1.92 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 168.89, 151.0, 148.4, 148.0, 138.2, 136.3, 134.0, 132.5, 129.2, 127.9, 127.3, 121.7, 121.6, 121.1, 121.0, 116.4, 50.2, 45.7, 33.2, 16.1; HRMS (ESI) calcd for $C_{21}H_{19}N_3O_3$ [M+Na]+: 384.13241, found 384.13384



3-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)phenyl dimethylcarbamate (8)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 3-iodophenyl dimethylcarbamate (174 mg, 0.6mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3 (83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 ^oC for 10 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=10: 1). Finally, compound **(8)** (87 mg) was isolated in 72% yield. Rf = 0.2(petroleum ether: ethyl acetate=5: 1)

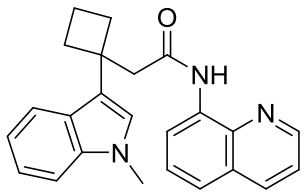
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.31 (s, 1H), 8.66 (d, *J* = 5.8 Hz, 2H), 8.10-8.07 (m, 1H), 7.49-7.36 (m, 3H), 7.17 (t, *J* = 7.8 Hz, 1H), 7.08 (d, *J* = 7.8 Hz, 1H), 7.02 (s, 1H), 6.85 (dd, *J* = 8.0 Hz, *J* = 1.2 Hz, 1H), 3.00-2.98 (m, 8H), 2.55-2.51 (m, 4H), 2.20-2.13 (m, 1H), 1.93-1.87 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.8, 155.0, 151.7, 150.5, 148.0, 138.4, 136.2, 134.5, 129.1, 127.9, 127.4, 122.8, 121.5, 121.3, 119.31, 119.25, 116.4, 50.8, 45.7, 36.8, 36.5, 33.1, 16.1; LRMS (ESI) calcd for $C_{24}H_{25}N_3O_3$ [M+H]+: 404.19, found 404.23



2-(1-(benzo[d][1,3]dioxol-5-yl)cyclobutyl)-N-(quinolin-8-yl)acetamide (9)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 5-iodobenzo[d][1,3]dioxole (145 mg, 0.6mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3 (83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 ^oC for 10 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(9)** (54 mg) was isolated in 50% yield.
Rf = 0.3(petroleum ether: ethyl acetate=5: 1)

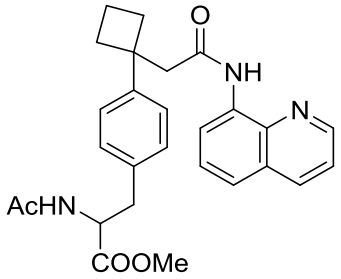
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.22 (s, 1H), 8.67-8.65 (m, 2H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.49-7.36 (m, 3H), 6.73-6.70 (m, 2H), 6.64 (d, *J* = 7.9 Hz, 1H), 5.74 (s, 2H), 2.97 (s, 2H), 2.49-2.47 (m, 4H), 2.23-2.12 (m, 1H), 1.95-1.89 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.8, 147.8, 147.7, 145.7, 142.9, 138.3, 136.2, 134.5, 127.9, 127.4, 121.5, 121.3, 118.9, 116.3, 108.2, 106.7, 100.8, 51.3, 45.8, 33.3, 15.9; LRMS (ESI) calcd for $C_{22}H_{20}N_2O_3$ [M+H]+: 361.15, found 361.32



2-(1-(1-methyl-1H-indol-3-yl)cyclobutyl)-N-(quinolin-8-yl)acetamide (10)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 3-iodo-1-methyl-1H-indole (154 mg, 0.6mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3 (83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 ^oC for 10 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(10)** (64 mg) was isolated in 58% yield. Rf = 0.15(petroleum ether: ethyl acetate=10: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.17 (s, 1H), 8.68 (d, *J* = 7.6 Hz, 1H), 8.40-8.39(m, 1H), 8.02 (dd, *J* = 8.2 Hz, *J* = 1.2 Hz, 1H), 7.66 (d, *J* = 7.9 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.32-7.23 (m, 3H), 7.14 (td, *J* = 7.9 Hz, *J* = 1.8 Hz, 1H), 6.89 (s, 1H), 3.49 (s, 3H), 3.25 (s, 2H), 2.62-2.58 (m, 4H), 2.24-2.17 (m, 1H), 2.10-2.02 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 170.5, 147.6, 138.3, 138.2, 136.1, 134.7, 127.8, 127.4, 126.6, 125.9, 121.6, 121.3, 121.1, 120.9, 120.2, 118.9, 116.2, 109.6, 50.0, 40.9, 33.6, 32.6, 17.1; LRMS (ESI) calcd for $C_{24}H_{23}N_3O$ [M+H]+: 370.18, found 370.64

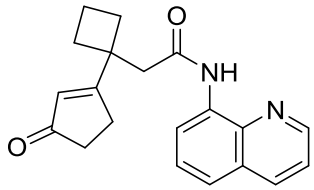


methyl

2-acetamido-3-(4-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)phenyl)propanoate (11)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then methyl 2-acetamido-3-(4-iodophenyl)propanoate (208 mg, 0.6mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3 (83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 ^oC for 10 h. After completion of the reaction, the residue was purified by silica gel column chromatography (DCM: ethyl acetate=100: 1). Finally, compound **(11)** (96 mg) was isolated in 70% yield. Rf = 0.5(DCM: MeOH=20: 1)

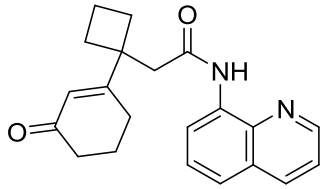
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.25 (s, 1H), 8.62-8.60 (m, 1H), 8.05 (d, *J* = 8.0 Hz, 1H), 7.42-7.34 (m, 3H), 7.16 (d, *J* = 7.6 Hz, 2H), 6.90 (d, *J* = 8.0 Hz, 2H), 5.92 (br, 1H), 4.72 (dd, *J* = 7.2 Hz, *J* = 14.4 Hz, 1H), 3.36 (s, 3H), 2.95-2.91 (m, 4H), 2.51-2.47 (m, 4H), 2.18-2.11 (m, 1H), 1.89-1.84 (m, 1H), 1.79 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 172.0, 169.7, 169.6, 147.9, 147.5, 138.2, 136.2, 134.3, 133.2, 129.1, 127.8, 127.3, 126.0, 121.5, 121.3, 116.2, 53.1, 52.1, 50.7, 45.4, 37.2, 33.3, 32.9, 22.9, 16.0; HRMS (ESI) calcd for $C_{27}H_{29}N_3O_4$ [M+Na]+: 482.20588, found 482.20841



2-(1-(3-oxocyclopent-1-en-1-yl)cyclobutyl)-N-(quinolin-8-yl)acetamide (12)

Following the general procedure I, substrate (48 mg, 0.20 mmol), Ag_2CO_3 (55 mg, 0.2 mmol), $Pd(OAc)_2$ (4.4 mg, 0.02 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 3-iodocyclopent-2-en-1-one (83 mg, 0.4 mmol), $Pd(OAc)_2$ (4.4 mg, 0.02 mmol) and Ag_2CO_3(55 mg, 0.2 mmol) were added. The tube was sealed and heated at 100 ^oC for 8h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=3: 1). Finally, compound **(12)** (35 mg) was isolated in 55% yield. Rf = 0.1(petroleum ether: ethyl acetate=3: 1)

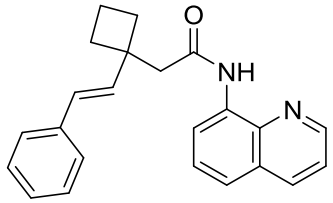
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.70 (s, 1H), 8.76-8.65 (m, 2H), 8.13 (d, *J* = 8.3 Hz, 1H), 7.49-7.26 (m, 3H), 6.13 (d, *J* = 1.4 Hz, 1H), 3.06 (s, 2H), 2.74-2.72 (m, 1H), 2.45-2.30 (m, 6H), 2.18-2.09 (m, 1H), 2.03-1.96 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 209.9, 186.4, 168.6, 148.3, 138.3, 136.4, 134.1, 129.0, 128.0, 127.3, 121.8, 116.6, 47.1, 44.3, 35.7, 32.5, 27.8, 15.9; LRMS (ESI) calcd for $C_{21}H_{22}N_2O_2$ [M+H]+: 321.15, found 321.23



2-(1-(3-oxocyclohex-1-en-1-yl)cyclobutyl)-N-(quinolin-8-yl)acetamide (13)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 3-iodocyclohex-2-en-1-one (133 mg, 0.6mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3(83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 ^oC for 12h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=3: 1). Finally, compound **(13)** (78 mg) was isolated in 78% yield. Rf = 0.1(petroleum ether: ethyl acetate=3: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.67 (s, 1H), 8.74-8.66 (m, 2H), 8.13 (d, *J* = 8.2 Hz, 1H), 7.51-7.41 (m, 3H), 6.00 (s, 1H), 2.96 (s, 2H), 2.38-2.25 (m, 8H), 2.17-2.08 (m, 1H), 2.00-1.93 (m, 2H), 1.90-1.84 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 200.0, 169.8, 168.8, 148.3, 138.4, 136.5, 134.3, 128.0, 127.5, 125.2, 121.8, 116.6, 47.2, 47.1, 37.6, 31.7, 25.8, 23.1, 15.3; LRMS (ESI) calcd for $C_{21}H_{22}N_2O_2$ [M+H]+: 335.17, found 335.60

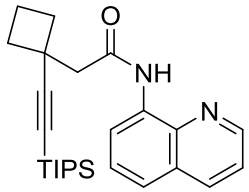


(E)-N-(quinolin-8-yl)-2-(1-styrylcyclobutyl)acetamide (14)

Following the general procedure I, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100

°C for 4h. Then (E)-(2-iodovinyl)benzene (138mg, 0.6mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and Ag_2CO_3(83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 °C for 12h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(14)** (62 mg) was isolated in 60% yield. Rf = 0.2(petroleum ether: ethyl acetate=15: 1)

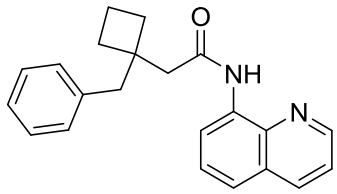
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.83 (s, 1H), 8.76 (d, *J* = 7.6 Hz, 1H), 8.45-8.44 (m, 1H), 8.10-8.08 (m, 1H), 7.52-7.45 (m, 2H), 7.38-7.33 (m, 3H), 7.25-7.16 (m, 3H), 6.56 (s, 2H), 3.12 (s, 2H), 2.36-2.24 (m, 4H), 2.09-1.96 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 170.0, 148.1, 138.5, 137.7, 136.3, 134.7, 131.7, 128.5, 128.3, 128.0, 127.5, 127.2, 126.5, 121.6, 121.4, 116.6, 49.4, 43.3, 32.5, 16.0; LRMS (ESI) calcd for $C_{21}H_{22}N_2O_2$ [M+H]+: 343.17, found 343.84



N-(quinolin-8-yl)-2-(1-((triisopropylsilyl)ethynyl)cyclobutyl)acetamide (15)

Following the general procedure III, substrate (72 mg, 0.30 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 °C for 4h. Then (bromoethynyl)triisopropylsilane (157 mg, 0.6 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol), AgOAc (100 mg, 0.6 mmol) and LiCl (13 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 °C for 8 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(15)** (64 mg) was isolated in 51% yield. Rf = 0.6(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.90 (s, 1H), 8.80-8.77 (m, 2H), 8.14 (d, *J* = 8.4 Hz, 1H), 7.53-7.41 (m, 3H), 2.85 (s, 2H), 2.49-2.37 (m, 4H), 2.25-2.14 (s, 1H), 2.06-1.96 (s, 1H), 0.88-0.81 (m, 21H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.3, 148.1, 138.6, 136.3, 134.7, 127.9, 127.4, 121.50, 121.47, 116.7, 113.8, 82.3, 48.6, 36.1, 35.3, 18.5, 17.0, 11.2; LRMS (ESI) calcd for $C_{27}H_{29}N_3O_4$ [M+H]+: 421.26, found 421.58

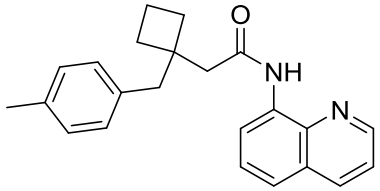


2-(1-benzylcyclobutyl)-N-(quinolin-8-yl)acetamide (16)

Following the genenal procedure III, amide (48 mg, 0.2 mmol), $Pd(OAc)_2$ (4.5 mg, 0.02 mmol), Ag_2CO_3(110 mg, 0.4 mmol) and t-amylOH (2 ml) were used. The tube was sealed and heated at 100 °C for 12 h. Then (bromomethyl)benzene (69 mg, 0.4 mmol**)**, $Pd(OAc)_2$ (4.5 mg, 0.02 mmol) and K_2CO_3(55 mg, 0.4 mmol) were added. The tube was sealed and heated at 100 °C for another 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(16)** (41 mg) was isolated in 62% yield. Rf = 0.4(petroleum ether: ethyl acetate=10: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.76 (s, 1H), 8.84-8.79 (m, 2H), 8.17 (d, *J* = 8.3 Hz, 1H), 7.58-7.44 (m, 3H), 7.29-7.22 (m, 5H), 3.04 (s, 2H), 2.63 (s, 2H), 2.21-2.11 (m, 4H), 1.99-1.88 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 170.8, 148.3, 139.4, 138.6, 136.5, 134.7, 130.4, 128.2,

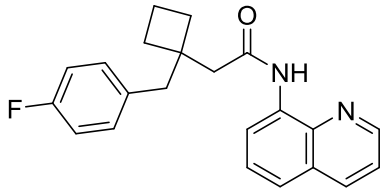
128.1, 127.6, 126.2, 121.7, 121.5, 116.6, 46.1, 44.6, 42.1, 32.0, 15.9; LRMS (ESI) calcd for $C_{22}H_{22}N_2O$ [M+H]+: 331.17, found 331.07



2-(1-(4-methylbenzyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (17)

Following the genenal procedure III, amide (72 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol), Ag_2CO_3(165 mg, 0.6 mmol) and t-amylOH (2 ml) were used. The tube was sealed and heated at 100 ^oC for 12 h. Then (bromomethyl)benzene (111 mg, 0.6 mmol**)**, $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and K_2CO_3(83 mg, 0.6 mmol) were added. The tube was sealed and heated at 100 ^oC for another 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(17)** (62 mg) was isolated in 60% yield. Rf = 0.4(petroleum ether: ethyl acetate=10: 1)

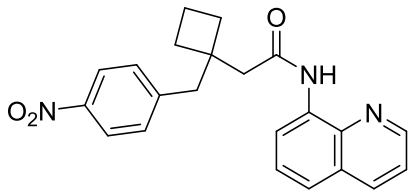
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.75 (s, 1H), 8.84-8.79 (m, 2H), 8.17 (dd, *J* = 8.2 Hz, *J* = 1.5 Hz, 1H), 7.57-7.44 (m, 3H), 7.18 (d, *J* = 7.9 Hz, 2H), 7.08 (d, *J* = 7.8 Hz, 2H), 2.99 (s, 2H), 2.63 (s, 2H), 2.32 (s, 3H), 2.17-2.08 (m, 4H), 2.02-1.85 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 170.8, 148.2, 138.5, 136.5, 136.2, 135.6, 134.8, 130.2, 128.9, 128.88, 128.6, 128.1, 127.6, 121.7, 121.4, 116.6, 46.1, 44.2, 42.0, 32.0, 21.2, 15.8; LRMS (ESI) calcd for $C_{23}H_{24}N_2O$ [M+H]+: 345.19, found 345.89



2-(1-(4-fluorobenzyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (18)

Following the genenal procedure III, amide (72 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol), Ag_2CO_3(165 mg, 0.6 mmol) and t-amylOH (2 ml) were used. The tube was sealed and heated at 100 ^oC for 12 h. Then 1-(bromomethyl)-4-(fluoromethyl)benzene (122 mg, 0.6 mmol**)**, $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and K_2CO_3(83 mg, 0.6 mmol) were added. The tube was sealed and heated at 100 ^oC for another 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(18)** (47 mg) was isolated in 45% yield. Rf = 0.3(petroleum ether: ethyl acetate=10: 1)

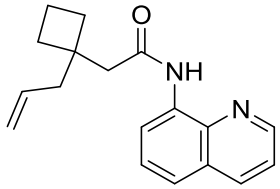
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.74 (s, 1H), 8.82-8.79 (m, 2H), 8.17 (d, *J* = 8.3 Hz, 1H), 7.57-7.45 (m, 3H), 7.26-7.23 (m, 2H), 6.95 (t, *J* = 8.7 Hz, 2H), 3.00 (s, 2H), 2.62 (s, 2H), 2.15-1.88 (m, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 170.6, 161.7 (d, *J* = 242.8 Hz), 148.3, 138.5, 136.5, 135.0 (d, *J* = 3.1 Hz), 134.6, 131.6 (d, *J* = 7.5 Hz), 128.1, 127.6, 121.8, 121.6, 116.6, 115.0 (d, *J* = 20.7 Hz), 45.9, 43.6, 42.0, 32.0, 15.8; LRMS (ESI) calcd for $C_{22}H_{21}FN_2O$ [M+H]+: 349.16, found 349.10



2-(1-(4-nitrobenzyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (19)

Following the genenal procedure III, amide (72 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol), Ag_2CO_3(165 mg, 0.6 mmol) and t-amylOH (2 ml) were used. The tube was sealed and heated at 100 ^oC for 12 h. Then 1-(bromomethyl)-4-(nitromethyl)benzene (138 mg, 0.6 mmol**)**, $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and K_2CO_3(83 mg, 0.6 mmol) were added. The tube was sealed and heated at 100 ^oC for another 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(19)** (56 mg) was isolated in 50% yield. Rf = 0.3(petroleum ether: ethyl acetate=10: 1)

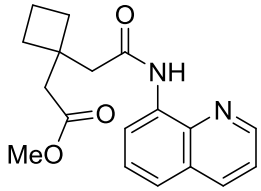
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.72 (s, 1H), 8.79-8.78 (m, 2H), 8.17 (dd, *J* = 8.3 Hz, *J* = 1.5 Hz, 1H), 8.10 (d, *J* = 8.7 Hz, 2H), 7.57-7.44 (m, 5H), 3.15 (s, 2H), 2.62 (s, 2H), 2.20-1.91 (m, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 170.1, 148.4, 147.4, 146.7, 138.4, 136.5, 134.4, 131.0, 128.1, 127.5, 123.5, 121.8, 121.8, 116.6, 45.6, 44.3, 42.0, 15.8; LRMS (ESI) calcd for $C_{22}H_{21}N_3O_3$ [M+H]+: 376.16, found 376.44



2-(1-allylcyclobutyl)-N-(quinolin-8-yl)acetamide (20)

Following the genenal procedure III, amide (72 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol), Ag_2CO_3(165 mg, 0.6 mmol) and t-amylOH (2 ml) were used. The tube was sealed and heated at 100 ^oC for 12 h. Then 3-bromoprop-1-ene (73 mg, 0.6 mmol**)**, $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and K_2CO_3(83 mg, 0.6 mmol) were added. The tube was sealed and heated at 100 ^oC for another 11 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=20: 1). Finally, compound **(20)** (38 mg) was isolated in 45% yield. Rf = 0.4(petroleum ether: ethyl acetate=10: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.76 (s, 1H), 8.80-8.78 (m, 2H), 8.15 (dd, *J* = 8.3 Hz, *J* = 1.5 Hz, 1H), 7.55-7.43 (m, 3H), 5.96-5.85 (m, 1H), 5.18-5.12 (m, 2H), 2.66 (s, 2H), 2.44 (d, *J* = 7.3 Hz, 1H), 2.17-2.13 (m, 2H), 2.04-1.94 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 170.6, 148.2, 138.5, 136.4, 135.0, 134.7, 128.1, 127.6, 121.7, 121.4, 117.9, 116.6, 48.1, 43.3, 40.8, 31.6, 15.7; LRMS (ESI) calcd for $C_{18}H_{20}N_2O$ [M+H]+: 281.16, found 281.47

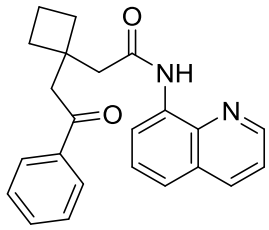


methyl 2-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (21)

Following the general procedure IV, substrate (24 mg, 0.1 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100

°C for 4h. Then methyl 2-iodoacetate (40 mg, 0.2 mmol), $(BnO)_2POOH$ (8 mg, 0.03 mmol) and Ag_2CO_3(28 mg, 0.1 mmol) were added. The tube was sealed and heated at 100 °C for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=5: 1). Finally, compound **(21)** (22 mg) was isolated in 72% yield. Rf = 0.1(petroleum ether: ethyl acetate=3: 1)

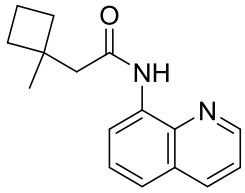
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.94 (s, 1H), 8.83-8.78 (m, 2H), 8.17 (dd, *J* = 8.3 Hz, *J* = 1.6 Hz, 1H), 7.54-7.45 (m, 3H), 3.72 (s, 3H), 2.93 (s, 2H), 2.80 (s, 2H), 2.27-2.20 (m, 2H), 2.13-1.99 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 172.4, 170.0, 148.0, 138.3, 136.2, 134.4, 127.8, 127.2, 121.5, 121.4, 116.4, 51.2, 46.0, 42.3, 39.1, 32.4, 15.6; LRMS (ESI) calcd for $C_{18}H_{20}N_2O_3$ [M+H]+: 313.15, found 313.72



2-(1-(2-oxo-2-phenylethyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (22)

Following the general procedure IV, substrate (72 mg, 0.3 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 °C for 4h. Then 2-iodo-1-phenylethan-1-one (148 mg, 0.6 mmol), $(BnO)_2POOH$ (24 mg, 0.09 mmol) and Ag_2CO_3(83 mg, 0.3 mmol) were added. The tube was sealed and heated at 100 °C for 6.5 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(22)** (45 mg) was isolated in 42% yield. Rf = 0.3(petroleum ether: ethyl acetate=10: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.80 (s, 1H), 8.73-8.65 (m, 2H), 8.11 (dd, *J* = 8.0 Hz, *J* = 1.2 Hz, 1H), 7.97 (d, *J* = 7.2 Hz, 2H), 7.51-7.36 (m, 6H), 3.52 (s, 2H), 3.09 (s, 2H), 2.31-2.24 (m, 2H), 2.15-1.98 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 200.0, 170.9, 148.2, 138.5, 137.9, 136.3, 134.6, 133.0, 128.6, 128.2, 128.0, 127.4, 121.6, 121.5, 116.5, 45.94, 45.88, 39.5, 33.4, 16.4; LRMS (ESI) calcd for $C_{23}H_{22}N_2O_2$ [M+H]+: 359.17, found 359.20

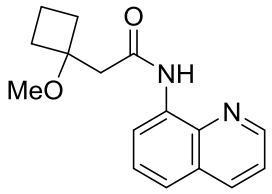


2-(1-methylcyclobutyl)-N-(quinolin-8-yl)acetamide (23)

Following the general procedure IV, substrate (48 mg, 0.20 mmol), Ag_2CO_3 (55 mg, 0.2 mmol), $Pd(OAc)_2$ (4.4 mg, 0.02 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 °C for 4h. Then iodomethane (57 mg, 0.4 mmol), $(BnO)_2POOH$ (17 mg, 0.06 mmol) and Ag_2CO_3(55 mg, 0.2 mmol) were added. The tube was sealed and heated at 100 °C for 4 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(23)** (24 mg) was isolated in 48% yield. Rf = 0.5(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.76 (s, 1H), 8.79 (s, 2H), 8.14 (d, *J* = 8.2 Hz, 1H), 7.54-7.42 (m, 3H), 2.64 (s, 2H), 2.21-2.15 (m, 2H), 2.01-1.87 (m, 4H), 1.34 (s, 3H); ^{13}C-NMR

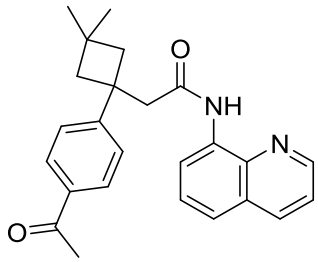
(100 MHz, $CDCl_3$) δ (ppm) 170.7, 148.2, 138.5, 136.5, 134.7, 128.1, 127.5, 121.7, 121.4, 116.5, 50.6, 37.9, 33.9, 26.4, 15.6; LRMS (ESI) calcd for $C_{16}H_{18}N_2O$ [M+H]+: 255.14, found 255.17



2-(1-methoxycyclobutyl)-N-(quinolin-8-yl)acetamide (24)

Following the general procedure IV, substrate (72 mg, 0.3 mmol), Ag_2CO_3 (83 mg, 0.3 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then I^{3+} (167 mg, 0.6 mmol), $Pd(OAc)_2$ (6.6 mg, 0.03 mmol) and MeOH (5 ml) were added. The tube was sealed and heated at 100 ^oC for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=7: 1). Finally, compound **(24)** (42 mg) was isolated in 52% yield. Rf = 0.25(petroleum ether: ethyl acetate=5: 1)

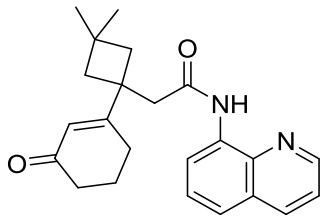
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 10.86 (s, 1H), 8.83-8.78 (m, 2H), 8.13 (d, *J* = 8.2 Hz, 1H), 7.54-7.41 (m, 3H), 3.47 (s, 3H), 2.93 (s, 2H), 2.44-2.36 (m, 2H), 2.06-2.00 (m, 2H), 1.81-1.70 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.6, 148.3, 139.0, 136.2, 135.2, 128.1, 127.5, 121.6, 121.5, 116.9, 78.2, 50.2, 44.5, 30.8, 12.3; LRMS (ESI) calcd for $C_{16}H_{18}N_2O_2$ [M+H]+: 271.14, found 271.19



2-(1-(4-acetylphenyl)-3,3-dimethylcyclobutyl)-N-(quinolin-8-yl)acetamide (25)

Following the general procedure I, substrate (27 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 1-(4-iodophenyl)ethan-1-one (49 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and Ag_2CO_3 (28 mg, 0.1 mmol) were added. The tube was sealed and heated at 100 ^oC for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(25)** (32 mg) was isolated in 83% yield. Rf = 0.2(petroleum ether: ethyl acetate=5: 1)

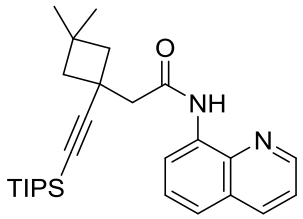
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.12 (s, 1H), 8.61-8.56 (m, 2H), 8.07 (dd, *J* = 8.3 Hz, *J* = 1.5 Hz, 1H), 7.78 (d, *J* = 8.4 Hz, 2H), 7.48-7.34 (m, 5H), 2.99 (s, 2H), 2.56-2.48 (m, 4H), 2.40 (s, 3H), 1.28 (s, 3H), 0.96 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 197.8, 169.1, 154.8, 147.9, 138.2, 136.3, 134.9, 134.3, 128.6, 127.8, 127.4, 126.8, 121.6, 121.5, 116.4, 54.2, 46.8, 38.7, 31.3, 30.8, 29.9, 26.6; HRMS (ESI) calcd for $C_{25}H_{26}N_2O_2$ [M+H]+: 387.20725, found 387.20834



2-(3,3-dimethyl-1-(3-oxocyclohex-1-en-1-yl)cyclobutyl)-N-(quinolin-8-yl)acetamide (26)

Following the general procedure I, substrate (27 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then 3-iodocyclohex-2-en-1-one (44 mg, 0.2mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and Ag_2CO_3(28 mg, 0.1 mmol) were added. The tube was sealed and heated at 100 ^oC for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=4: 1). Finally, compound **(26)** (25 mg) was isolated in 70% yield. Rf = 0.2(petroleum ether: ethyl acetate=3: 1)

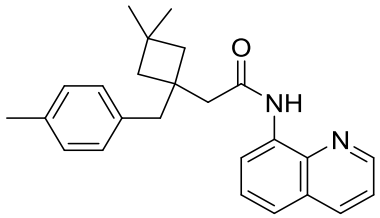
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.63 (s, 1H), 8.76-8.75 (m, 1H), 8.68-8.66 (m, 1H), 8.14 (d, *J* = 8.2 Hz, 1H), 7.50-7.26 (m, 3H), 6.13 (s, 1H), 2.94 (s, 2H), 2.41 (t, *J* = 5.8 Hz, 2H), 2.34 (t, *J* = 6.4 Hz, 2H), 2.29 (m, 4H), 2.01-1.95 (m, 2H), 1.25 (s, 3H), 1.08 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 197.8, 169.1, 154.8, 147.9, 138.2, 136.3, 134.9, 134.3, 128.6, 127.8, 127.4, 126.8, 121.6, 121.5, 116.4, 54.2, 46.8, 38.7, 31.3, 30.8, 29.9, 26.6; LRMS (ESI) calcd for $C_{23}H_{26}N_2O_2$ [M+H]+: 364.20, found 364.35



2-(3,3-dimethyl-1-((triisopropylsilyl)ethynyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (27)

Following the general procedure III, substrate (27 mg, 0.1 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then (bromoethynyl)triisopropylsilane (52 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), AgOAc (33 mg, 0.2 mmol) and LiCl (4 mg, 0.1 mmol) were added. The tube was sealed and heated at 100 ^oC for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(27)** (24 mg) was isolated in 53% yield. Rf = 0.6(petroleum ether: ethyl acetate=5: 1)

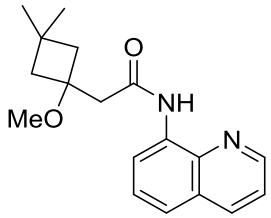
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.88 (s, 1H), 8.80-8.77 (m, 2H), 8.14 (dd, *J* = 8.2 Hz, *J* = 1.4 Hz, 1H), 7.53-7.41 (m, 3H), 2.82 (s, 2H), 2.36-2.26 (m, 4H), 1.39 (s, 3H), 1.14 (s, 3H), 0.86-0.85 (m, 21H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.2, 148.1, 138.7, 136.3, 134.8, 128.0, 127.5, 121.51, 121.46, 116.8, 115.1, 82.1, 51.1, 48.1, 31.7, 30.7, 30.1, 28.7, 18.6, 11.3; LRMS (ESI) calcd for $C_{28}H_{40}N_2OSi$ $[M+H]^+$: 449.29, found 449.17



2-(3,3-dimethyl-1-(4-methylbenzyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (28)

Following the genenal procedure III, amide (27 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), Ag_2CO_3(28 mg, 0.1 mmol) and t-amylOH (2 ml) were used. The tube was sealed and heated at 100 ^oC for 12 h. Then (bromomethyl)benzene (37 mg, 0.2 mmol**)**, $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and K_2CO_3(28 mg, 0.2 mmol) were added. The tube was sealed and heated at 100 ^oC for another 5.5 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(28)** (22 mg) was isolated in 60% yield. Rf = 0.7(petroleum ether: ethyl acetate=5: 1)

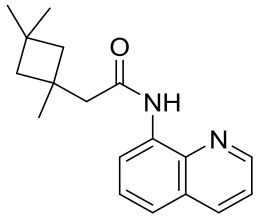
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.71 (s, 1H), 8.83-8.80 (m, 2H), 8.17 (d, *J* = 8.2 Hz, 1H), 7.57-7.44 (m, 3H), 8.15 (d, *J* = 7.8 Hz, 2H), 8.06 (d, *J* = 6.8 Hz, 2H), 3.02 (s, 2H), 2.65 (s, 2H), 2.31 (s, 3H), 2.11-1.99 (m, 4H), 1.17 (s, 3H), 1.10 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 170.8, 148.2, 138.6, 136.5, 136.3, 135.6, 134,8, 130.3, 128.9, 128.1, 127.6, 121.7, 121.4, 116.6, 47.8, 45.8, 44.9, 33.3, 32.1, 31.6, 28.3, 21.2; LRMS (ESI) calcd for $C_{25}H_{28}N_2O$ $[M+H]^+$: 373.22, found 373.57



2-(1-methoxy-3,3-dimethylcyclobutyl)-N-(quinolin-8-yl)acetamide (29)

Following the general procedure IV, substrate (27 mg, 0.1 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then I^{3+} (56 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and MeOH (5 ml) were added. The tube was sealed and heated at 100 ^oC for 20 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(29)** (16 mg) was isolated in 55% yield. Rf = 0.3(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 10.78 (s, 1H), 8.85-8.78 (m, 2H), 8.13 (dd, *J* = 8.4 Hz, *J* = 1.2 Hz, 1H), 7.54-7.41 (m, 3H), 3.43 (s, 3H), 2.93 (s, 2H), 2.22 (d, *J* = 13.6 Hz, 2H), 1.97 (d, *J* = 10.8 Hz, 2H), 1.22 (s, 3H), 1.19 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 169.4, 148.5, 139.1, 136.2, 135.2, 130.3, 128.6, 128.1, 127.5, 121.6, 117.0, 73.2, 49.9, 46.6, 43.8, 31.2, 30.1, 29.8, 26.8; LRMS (ESI) calcd for $C_{18}H_{22}N_2O_2$ $[M+H]^+$: 299.17, found 299.64

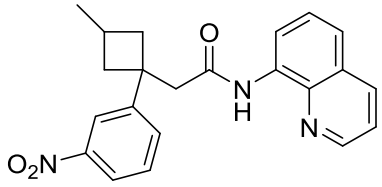


N-(quinolin-8-yl)-2-(1,3,3-trimethylcyclobutyl)acetamide (30)

Following the general procedure IV, substrate (27 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and 2 ml toluene were used. The tube was sealed and heated at 100 ^oC for 4h. Then iodomethane (28 mg, 0.2 mmol), $(BnO)_2POOH$ (8 mg, 0.03 mmol) and Ag_2CO_3(28 mg, 0.1 mmol) were added. The tube was sealed and heated at 100 ^oC for 12 h. After

completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=15: 1). Finally, compound **(30)** (20 mg) was isolated in 71% yield. Rf = 0.7(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.75 (s, 1H), 8.81-8.77 (m, 2H), 8.15 (dd, *J* = 8.3 Hz, *J* = 1.5 Hz, 1H), 7.55-7.43 (m, 3H), 2.66 (s, 2H), 2.09 (d, *J* = 12.6 Hz, 2H), 1.83 (d, *J* = 12.8 Hz, 2H), 1.36 (s, 3H), 1.19 (s, 3H), 1.17 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 207.1, 170.8, 148.3, 138.5, 136.5, 134.7, 128.1, 127.6, 121.7, 121.4, 116.5, 52.1, 46.9, 32.2, 31.6, 31.1, 29.4, 29.0, 28.0; LRMS (ESI) calcd for $C_{18}H_{22}N_2O$ $[M+H]^+$: 283.39, found 283.76



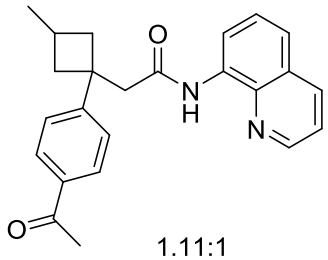
2.87:1

2-(3-methyl-1-(3-nitrophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (31)

Following the general procedure I, substrate (25 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 ^{o}C for 4h. Then 1-iodo-3-nitrobenzene (50 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and Ag_2CO_3 (28 mg, 0.1 mmol) were added. The tube was sealed and heated at 100 ^{o}C for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(31)** (25 mg) was isolated in 67% yield. Rf = 0.4(petroleum ether: ethyl acetate=5: 1)

major product

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.28 (s, 1H), 8.65-8.57 (m, 2H), 8.35 (t, *J* = 1.6 Hz, 1H), 8.12-8.08 (m, 1H), 7.97-7.95 (m, 1H), 7.53-7.28 (m, 4H), 3.01 (s, 2H), 2.84-2.79 (m, 2H), 2.32-2.19 (m, 3H), 1.18 (d, *J* = 6.4 Hz, 3H); LRMS (ESI) calcd for $C_{18}H_{22}N_2O_2$ $[M+H]^+$: 376.16, found 376.23



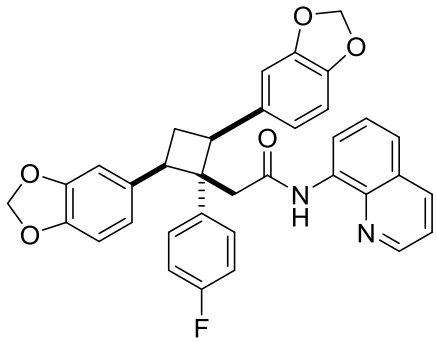
1.11:1

2-(1-(4-acetylphenyl)-3-methylcyclobutyl)-N-(quinolin-8-yl)acetamide (32)

Following the general procedure I, substrate (25 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 ^{o}C for 4h. Then 1-(4-iodophenyl)ethan-1-one (49 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol) and Ag_2CO_3 (28 mg, 0.1 mmol) were added. The tube was sealed and heated at 100 ^{o}C for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=8: 1). Finally, compound **(32)** (24 mg) was isolated in 65% yield. Rf = 0.2(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.24 (s, 0.47H), 9.16 (s, 0.53H), 8.63-8.53 (m, 2.02H), 8.10-8.06 (m, 1.06H), 7.85-7.76 (m, 2.08H), 7.54-7.28 (m, 4.32H), 3.01 (s, 1.08H), 2.98 (s, 1H),

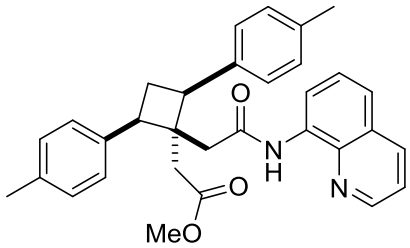
2.84-2.75 (m, 2.1H), 2.70-2.63 (m, 0.66H), 2.45 (s, 1.47H), 2.40 (s, 1.64H), 2.21-2.18 (m, 1.58H), 2.08-2.03 (m, 1.56H), 1.16 (d, *J* = 6.4 Hz, 1.55H), 1.08 (d, *J* = 6.4 Hz, 1.73H); LRMS (ESI) calcd for $C_{18}H_{22}N_2O_2$ $[M+H]^+$: 373.18, found 373.32



2-((1s,2R,4S)-2,4-bis(benzo[d][1,3]dioxol-5-yl)-1-(4-fluorophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (33)

Following the general procedure I, 2-(1-(4-fluorophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (33 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), 5-iodobenzo[d][1,3]dioxole (50 mg, 0.2 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 °C for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=5: 1). Finally, compound **(33)** (45 mg) was isolated in 78% yield. Rf = 0.3(petroleum ether: ethyl acetate=3: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 8.83 (s, 1H), 8.62 (dd, *J* = 4.2 Hz, *J* = 1.6 Hz, 1H), 8.23 (dd, *J* = 1.6 Hz, *J* = 7.4 Hz, 1H), 8.05 (dd, *J* = 1.6 Hz, *J* = 8.3 Hz, 1H), 7.60-7.57 (m, 2H), 7.39-7.28 (m, 3H), 7.20 (t, *J* = 8.7 Hz, 2H), 6.82-6.79 (m, 4H), 6.63 (d, *J* = 7.9 Hz, 2H), 5.67 (d, *J* = 1.5 Hz, 2H), 5.54 (d, *J* = 1.5 Hz, 2H), 3.93-3.88 (m, 2H), 2.92-2.84 (m, 1H), 2.71 (s, 2H), 2.47-2.40 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 168.7, 161.7 (d, *J* = 243.0 Hz), 147.7, 147.6, 146.5, 142.3 (d, *J* = 3.3 Hz), 138.2, 136.0, 134.6, 132.4, 127.76 (d, *J* = 5.9 Hz), 127.72, 127.3, 121.6, 121.4, 120.8, 116.3, 115.7 (d, *J* = 21.0 Hz), 109.1, 108.2, 100.9, 55.7, 46.1, 39.4, 25.1; HRMS (ESI) calcd for $C_{35}H_{27}FN_2O_5$ $[M+H]^+$: 575.19823, found 575.19980



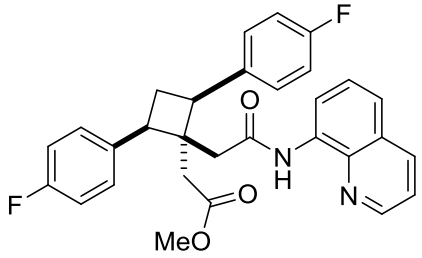
methyl 2-((1r,2R,4S)-1-(2-oxo-2-(quinolin-8-ylamino)ethyl)-2,4-di-p-tolylcyclobutyl)acetate (34)

Following the general procedure I, methyl 2-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (31 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), 1-iodo-4-methylbenzene (44 mg, 0.2 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 °C for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=5: 1). Finally, compound **(34)** (22 mg) was isolated in 45% yield.

Rf = 0.45(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 8.78 (dd, *J* = 4.2 Hz, *J* = 1.7 Hz, 1H), 8.61 (s, 1H), 8.32 (dd,

J = 6.1 Hz, *J* = 2.9 Hz, 1H), 8.09 (dd, *J* = 8.2 Hz, *J* = 1.7 Hz, 1H), 7.44-7.37 (m, 3H), 7.22 (d, *J* = 8.0 Hz, 4H), 6.90 (d, *J* = 7.8 Hz, 4H), 3.79 (s, 3H), 3.77-3.72 (m, 2H), 3.40 (s, 2H), 2.92-2.84 (m, 1H), 2.41-2.34 (m, 1H), 2.17 (s, 2H), 1.77 (s, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 173.1, 169.7, 147.6, 137.9, 136.2, 136.15, 134.8, 129.1, 127.9, 127.8, 127.4, 121.4, 120.9, 116.0, 51.6, 50.5, 43.8, 38.4, 23.1, 20.5; LRMS (ESI) calcd for $C_{32}H_{32}N_2O_3$ $[M+H]^+$: 493.24, found 493.63

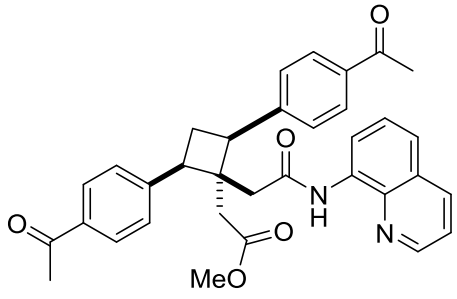


methyl 2-((1r,2R,4S)-2,4-bis(4-fluorophenyl)-1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (35)

Following the general procedure I, methyl 2-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (31 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), 1-fluoro-4-iodobenzene (44 mg, 0.2 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 °C for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (toluene: ethyl acetate=60: 1). Finally, compound **(35)** (16 mg) was isolated in 32% yield.

Rf = 0.36(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 8.76-8.75 (m, 2H), 8.31 (d, *J* = 6.1 Hz, 1H), 8.12-8.10 (m, 1H), 7.44-7.38 (m, 3H), 7.31-7.28 (m, 4H), 6.82 (t, *J* = 8.6 Hz, 4H), 3.79 (s, 3H), 3.74 (t, *J* = 10.4 Hz, 2H), 3.39 (s, 2H), 2.85-2.76 (m, 1H), 2.46-2.40 (m, 1H), 2.20 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 172.9, 169.2, 161.7 (d, *J* = 245.3 Hz), 147.8, 137.9, 136.4, 134.7 (d, *J* = 3.0 Hz), 134.3, 129.5 (d, *J* = 7.8 Hz), 127.9, 127.3, 121.5, 121.4, 116.1, 115.2 (d, *J* = 21.1 Hz), 51.7, 50.4, 43.7, 43.6, 37.9, 24.0; LRMS (ESI) calcd for $C_{30}H_{26}F_2N_2O_3$ $[M+H]^+$: 501.19, found 501.57



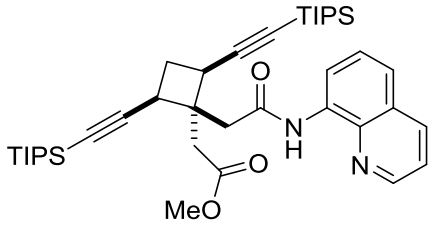
methyl 2-((1r,2R,4S)-2,4-bis(4-acetylphenyl)-1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (36)

Following the general procedure I, methyl 2-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (31 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), 1-(4-iodophenyl)ethan-1-one (44 mg, 0.3 mmol) and 1 ml toluene were used. The tube was sealed and heated at 100 °C for 16 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl

acetate=3: 1). Finally, **(36)** (22 mg) was isolated in 40% yield.

Rf = 0.2(DCM: ethyl acetate=10: 1)

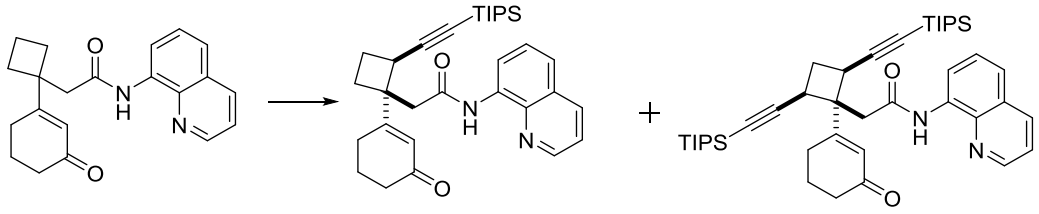
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 8.75-8.74 (m, 1H), 8.69 (s, 1H), 8.29-8.26 (m, 1H), 8.06 (dd, *J* = 8.2 Hz, *J* = 1.6 Hz, 1H), 7.71 (d, *J* = 8.2 Hz, 4H), 7.45-7.41 (m, 5H), 7.35-7.34 (m, 2H), 3.92-3.87 (m, 2H), 3.82 (s, 3H), 3.00 (dd, *J* = 22.0 Hz, *J* = 11.0 Hz, 1H), 2.53-2.46 (m, 1H), 2.17 (s, 2H), 2.15 (s, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 197.1, 172.7, 168.8, 148.1, 144.5, 136.3, 135.4, 134.4, 128.5, 128.2, 127.9, 127.3, 121.7, 121.4, 115.8, 51.9, 51.0, 44.2, 43.6, 38.3, 26.2, 23.1; LRMS (ESI) calcd for $C_{34}H_{32}N_2O_5$ $[M+H]^+$: 549.23, found 548.67



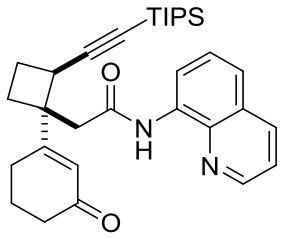
methyl 2-((1r,2R,4S)-1-(2-oxo-2-(quinolin-8-ylamino)ethyl)-2,4-bis((triisopropylsilyl)ethynyl)cyclobutyl)acetate (37)

Following the general procedure III, methyl 2-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (31 mg, 0.10 mmol), (bromoethynyl)triisopropylsilane (52 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), AgOAc (33 mg, 0.2 mmol), LiCl (4 mg, 0.1 mmol) and toluene (2 mL) were added. The tube was sealed and heated at 100 °C for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=10: 1). Finally, compound **(37)** (30 mg) was isolated in 45% yield. Rf = 0.6(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.89 (s, 1H), 8.79-8.70 (m, 3H), 8.14 (dd, *J* = 8.2 Hz, *J* = 1.5 Hz, 1H), 7.50-7.41 (m, 3H), 3.65 (s, 3H), 3.36 (s, 2H), 2.23 (s, 2H), 3.18-3.14 (m, 2H), 2.86-2.78 (m, 1H), 2.16-2.10 (m, 1H), 1.00-0.98 (m, 42H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 171.8, 169.2, 148.0, 138.4, 136.2, 134.9, 127.9, 127.4, 121.5, 129.9, 116.4, 108.1, 85.7, 51.6, 44.0, 42.9, 39.1, 33.5, 32.7, 18.7, 11.4; LRMS (ESI) calcd for $C_{40}H_{60}N_2O_3Si_2$ $[M+H]^+$: 673.41, found 673.36



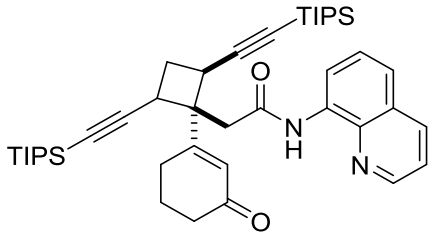
Following the general procedure III, 2-(1-(3-oxocyclohex-1-en-1-yl)cyclobutyl)-N-(quinolin-8-yl)acetamide (33 mg, 0.10 mmol), (bromoethynyl)triisopropylsilane (52 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), AgOAc (33 mg, 0.2 mmol), LiCl (4 mg, 0.1 mmol) and toluene (2 mL) were added. The tube was sealed and heated at 100 °C for 12 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=5: 1). Finally, compound **(38a)** (5 mg) and compound **(38b)** (35 mg) was isolated in 10% and 50% yield.



2-((1S,2R)-1-(3-oxocyclohex-1-en-1-yl)-2-((triisopropylsilyl)ethynyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (38a)

Rf = 0.2(petroleum ether: ethyl acetate=5: 1)

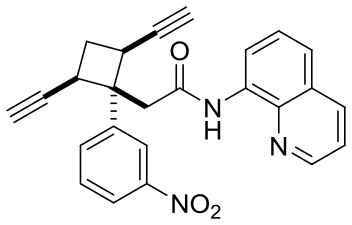
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.71 (s, 1H), 8.75-8.66 (m, 2H), 8.14 (dd, *J* = 8.4 Hz, *J* = 1.6 Hz, 1H), 7.50-7.42 (m, 3H), 5.96 (s, 1H), 3.20-3.05 (m, 3H), 2.51-2.22 (m, 8H), 2.11-2.07 (m, 1H), 1.96-1.94 (m, 1H), 1.12-1.08 (m, 25H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 199.7, 168.9, 168.6, 148.3, 138.4, 136.5, 134.3, 128.1, 127.5, 125.4, 121.8, 116.7, 107.0, 86.0, 49.9, 43.3, 37.8, 36.7, 27.2, 25.6, 23.8, 18.8, 11.5; HRMS (ESI) calcd for $C_{32}H_{42}N_2O_2Si$ $[M+H]^+$: 515.30938, found 515.31020



2-((1R,2R)-1-(3-oxocyclohex-1-en-1-yl)-2,4-bis((triisopropylsilyl)ethynyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (38b)

Rf = 0.5(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.84 (s, 1H), 8.75-8.72 (m, 2H), 8.11 (d, *J* = 8.2 Hz, 1H), 7.46-7.39 (m, 3H), 6.28 (s, 1H), 3.42 (s, 2H), 3.23 (t, *J* = 8.9 Hz, 2H), 2.64-2.61 (m, 1H), 2.50-2.47 (m, 2H), 2.41-2.38 (m, 2H), 2.22-2.20 (m, 1H), 2.07-2.02 (m, 2H), 0.96-0.91 (m, 42H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 199.7, 167.8, 167.6, 148.3, 138.7, 136.2, 134.9, 127.9, 127.2, 125.3, 121.6, 121.4, 117.0, 105.9, 86.6, 52.8, 40.3, 37.8, 33.1, 32.4, 26.1, 23.0, 18.7, 18.6, 11.8, 11.4; LRMS (ESI) calcd for $C_{43}H_{62}N_2O_2Si_2$ $[M+H]^+$: 695.43, found 695.41

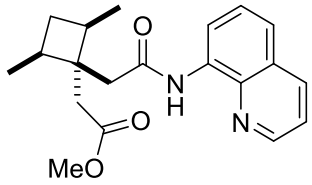


2-((1R,2R)-1-(3-nitrophenyl)-2,4-bis((triisopropylsilyl)ethynyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (39)

Following the general procedure III, 2-(1-(3-nitrophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (36 mg, 0.10 mmol), (bromoethynyl)triisopropylsilane (52 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), AgOAc (33 mg, 0.2 mmol), LiCl (4 mg, 0.1 mmol) and toluene (2 mL) were added. The tube was sealed and heated at 100 °C for 10 h. After completion of the reaction, the residue was treated with TBAF in THF at room temperature for an hour and purified by silica gel column chromatography (petroleum ether: ethyl acetate=20: 1). Finally, compound **(39)** (20 mg) was

isolated in 48% yield.

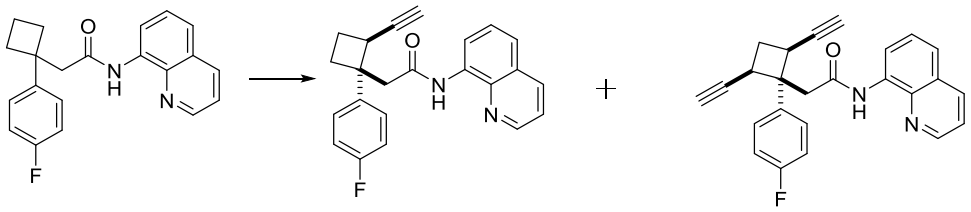
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.90 (s, 1H), 8.78-8.77 (d, 1H), 8.49-8.47 (d, 1H), 8.23 (s, 1H), 8.13 (d, *J* = 8.4 Hz, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 7.9 Hz, 1H), 7.51-7.39 (m, 4H), 3.51 (s, 2H), 3.42-3.36 (m, 2H), 2.76-2.69 (m, 1H), 2.48-2.40 (m, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 167.92, 149.05, 148.22, 148.01, 138.41, 136.38, 134.37, 132.15, 129.13, 127.98, 127.41, 121.86, 121.60, 121.47, 120.68, 116.75, 82.32, 74.38, 51.31, 42.56, 33.54, 31.88; LRMS (ESI) calcd for $C_{25}H_{19}N_3O_3$ $[M+H]^+$: 410.14, found 410.09



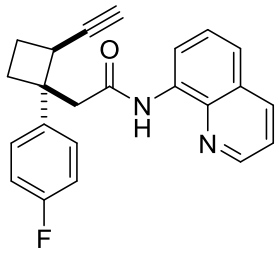
methyl 2-((1r,2R,4S)-2,4-dimethyl-1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (40)

Following the general procedure IV, methyl 2-(1-(2-oxo-2-(quinolin-8-ylamino)ethyl)cyclobutyl)acetate (31 mg, 0.10 mmol), Ag_2CO_3 (28 mg, 0.1 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), iodomethane (28 mg, 0.2 mmol), $(BnO)_2POOH$ (8 mg, 0.03 mmol) and MeOH (2mL) were added. The tube was sealed and heated at 140 ^{o}C for 14 h. After completion of the reaction, the residue was purified by silica gel column chromatography (petroleum ether: ethyl acetate=7: 1). Finally, compound **(43)** (19 mg, white solid) was isolated in 55% yield. Rf = 0.4(petroleum ether: ethyl acetate=5: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.88 (s, 1H), 8.83-8.82 (m, 1H), 8.76-8.75 (m, 1H), 8.15 (d, *J* = 7.6 Hz, 1H), 7.51-7.43 (m, 3H), 3.63 (s, 3H), 2.97 (s, 2H), 2.91 (s, 2H), 2.28-2.17 (m, 3H), 1.25-1.21 (m, 1H), 1.09 (d, *J* = 6.8 Hz, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 173.2, 171.1, 148.2, 138.5, 136.5, 134.9, 128.1, 127.5, 121.7, 121.3, 116.5, 51.3, 44.7, 43.6, 37.7, 35.2, 33.2, 17.1; LRMS (ESI) calcd for $C_{20}H_{24}N_2O_3$ $[M+H]^+$: 341.18, found 341.65

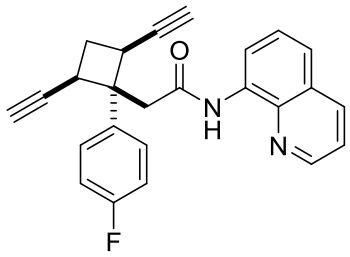


Following the general procedure III, 2-(1-(4-fluorophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (33 mg, 0.10 mmol), (bromoethynyl)triisopropylsilane (52 mg, 0.2 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), AgOAc (33 mg, 0.2 mmol), LiCl (4 mg, 0.1 mmol) and toluene (2 mL) were added. The tube was sealed and heated at 100 ^{o}C for 12 h. After completion of the reaction, the residue was treated with TBAF in THF at room temperature for an hour and purified by silica gel column chromatography (petroleum ether: ethyl acetate=20: 1). Finally, compound **(41a)** (4 mg) and compound **(41b)** (15 mg)was isolated in 11% and 40% yield.



2-((1S,2S)-2-ethynyl-1-(4-fluorophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (41a)

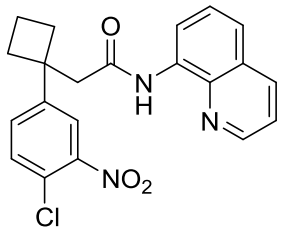
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.83 (s, 1H), 8.75-8.74 (d, 1H), 8.54-8.52 (dd, 1H), 8.13-8.10 (d, 1H), 7.44-7.35 (m, 5H), 7.02-6.98 (t, *J* = 8.0 Hz, 2H), 3.45 (s, 2H), 3.38-3.33 (m, 2H), 2.71-2.64 (m, 1H), 2.43-2.38 (m, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 168.49, 162.86, 160.43, 147.86, 142.69, 142.65, 138.58, 136.35, 134.73, 128.03, 127.53, 127.37, 127.29, 121.55, 121.32, 116.94, 115.41, 115.20, 83.07, 74.01, 51.25, 43.22, 33.87, 31.85; LRMS (ESI) calcd for $C_{23}H_{19}FN_2O$ $[M+H]^+$: 359.15, found 359.56



2-((1s,2R,4S)-2,4-diethynyl-1-(4-fluoropheny)cyclobutyl)-N-(quinolin-8-yl)acetamide (41b)

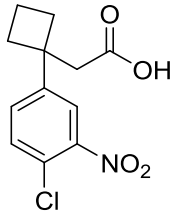
Rf = 0.6(petroleum ether: ethyl acetate=20: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.22 (s, 1H), 8.62-8.61 (m, 2H), 8.10-8.08 (dd, *J* = 8.4 Hz, *J* = 1.2 Hz 1H), 7.48-7.37 (m, 3H), 7.27-7.23 (m, 2H), 6.89-6.85 (t, *J* = 8.8 Hz, 2H), 3.35-3.27 (m, 2H), 3.02 (d, 1H), 2.79-2.74 (m, 1H), 2.47-2.28 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 167.92, 149.05, 148.22, 148.01, 138.41, 136.38, 134.37, 132.15, 129.13, 127.98, 127.41, 121.86, 121.60, 121.47, 120.68, 116.75, 82.32, 74.38, 51.31, 42.56, 33.54, 31.88; LRMS (ESI) calcd for $C_{25}H_{19}FN_2O$ $[M+H]^+$: 383.15, found 383.49



2-(1-(4-chloro-3-nitrophenyl)cyclobutyl)-N-(quinolin-8-yl)acetamide (42')

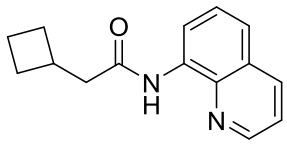
^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.23 (s, 1H), 8.65-8.57 (m, 2H), 8.09 (dd, *J* = 1.1 Hz, *J* = 8.2 Hz, 1H), 7.76 (d, *J* = 2.0 Hz, 1H), 7.46-7.36 (m, 4H), 7.28 (d, *J* = 8.3 Hz, 1H), 3.04 (s, 2H), 2.59-2.44 (m, 4H), 2.29-2.18 (m, 1H), 1.99-1.90 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 168.6, 149.5, 148.3, 138.1, 136.4, 133.9, 131.6, 131.2, 127.9, 127.3, 124.4, 123.3, 121.8, 121.7, 116.5, 50.0, 45.3, 33.1, 16.0; LRMS (ESI) calcd for $C_{21}H_{18}ClN_3O_3$ $[M+H]^+$: 396.10, found 396.03



2-(1-(4-chloro-3-nitrophenyl)cyclobutyl)acetic acid (42)

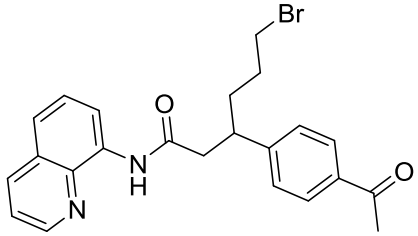
Rf = 0.36(petroleum ether: ethyl acetate=3: 1)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 10.16 (br, 1H), 7.69 (d, *J* = 1.8 Hz, 1H), 7.45 (d, *J* = 8.3 Hz, 1H), 7.35 (dd, *J* = 1.8 Hz, *J* = 8.3 Hz, 1H), 2.87 (s, 2H), 2.43-2.37 (m, 4H), 2.16-2.04 (m, 1H), 1.93-1.88 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 177.0, 149.0, 147.7, 131.6, 131.2, 124.5, 123.5, 45.6, 44.0, 33.1, 15.9; LRMS (ESI) calcd for $C_{12}H_{12}ClNO_4$ $[M-H]^-$: 268.05, found 268.12



2-cyclobutyl-N-(quinolin-8-yl)acetamide (44)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.78 (s, 1H), 8.81-8.76 (m, 2H), 8.16-8.12 (m, 1H), 7.54-7.41 (m, 3H), 2.94-2.85 (m, 1H), 2.68-2.65 (m, 2H), 2.29-2.22 (m, 2H), 2.00-1.85 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 171.1, 148.3, 138.5, 136.5, 134.7, 128.0, 127.6, 121.7, 121.4, 116.5, 45.4, 32.9, 28.6, 18.8; HRMS (ESI) calcd for $C_{21}H_{18}ClN_3O_3$ $[M+Na]^+$: 263.11603, found 263.11644



3-(4-acetylphenyl)-6-bromo-N-(quinolin-8-yl)hexanamide (45)

^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.67 (s, 1H), 8.75-8.67 (m, 2H), 8.14 (dd, *J* = 2.0 Hz, *J* = 8.4 Hz, 1H), 7.89 (dd, *J* = 1.6 Hz, *J* = 6.8 Hz, 2H), 7.50-7.38 (m, 5H), 3.50-3.33 (m, 3H), 2.94-2.83 (m, 2H), 2.54 (s, 3H), 2.04-1.92 (m, 1H), 1.91-1.83 (m, 2H), 1.78-1.63 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 197.9, 169.5, 149.3, 148.2, 138.3, 136.5, 136.0, 134.3, 129.1, 128.0, 127.9, 127.5, 121.8, 116.6, 45.4, 42.1, 34.5, 33.5, 30.8, 26.7; LRMS (ESI) calcd for $C_{21}H_{18}ClN_3O_3$ $[M+H]^+$: 396.10, found 396.03

Crystal Structure of $C_{25}H_{19}FN_2O$

The low temperature [173.0(1)°K] single-crystal X-ray experiments were performed on a SuperNova diffractometer with Cu K_α radiation. Unit cell was obtained and refined by 9484 reflections with $4.6° < \theta < 76.8°$. No decay was observed in data collection. Raw intensities were corrected for Lorentz and polarization effects, and for absorption by empirical method. Direct phase determination yielded the positions of all non-hydrogen atoms. All non-hydrogen atoms were subjected to anisotropic refinement. The hydrogen atoms of carbon atoms were generated geometrically with C-H bonds of 0.93-0.98 Å according to criteria described in the SHELXTL manual (Bruker, 1997). The hydrogen atoms of nitrogen atoms were found on the difference Fourier map. They were included in the refinement with $U_{iso}(H) = 1.2U_{eq}$ of their parent atoms. The final full-matric least-square refinement on F^2 converged with $R1$ = 0.0627 and $wR2$ = 0.1395 for 7104 observed reflections [$I \geq 2\sigma(I)$]. The final difference electron density map shows no features. Details of crystal parameters, data collection and structure refinement are given in Table 1.

Data collection was controlled by CrysAlis^{Pro} (Rigaku, 2016). Computations were performed using the SHELXTL NT ver. 5.10 program package (Bruker, 1997) on an IBM PC 586 computer. Analytic expressions of atomic scattering factors were employed, and anomalous dispersion corrections were incorporated (*International Tables for X-ray Crystallography*, 1989). Crystal drawings were produced with XP (Bruker, 1997).

References

Bruker. (1997) SHELXTL. Structure Determination Programs, Version 5.10, Bruker AXS Inc.,6300 Enterprise Lane, Madison, WI 53719-1173, USA.

International Tables for X-ray Crystallography: (1989) Vol. C (Kluwer Academic Publishers, Dordrecht) Tables 4.2.6.8 and 6.1.1.4.

Rigaku. (2016) CrysAlis^{Pro}, Data Collection and Process Software for Rigaku Oxford Diffraction X-ray Diffractometer, Version 5.4, February, 2016. Rigaku Corporation, 9009, New Trails Drive, The Woodlands, TX77381, USA.

Table 1. Details of Data Collection, Processing and Structure Refinement

Sample code	**RaoY-2**
Molecular formula	$C_{25}H_{19}FN_2O$
Molecular weight	382.42
Color and habit	colorless prism
Crystal size	0.1 × 0.3 × 0.3 mm
Crystal system	triclinic
Space group	$P\bar{1}$ (No. 2)
Unit cell parameters	a = 7.2657(2) Å α = 110.971(3)° b = 15.2058(5) Å β = 99.332(3)° c = 19.3128(8) Å γ = 95.705(2)° V = 1937.08(12) Å^3 Z = 4 F(000) = 800
Density (calcd)	1.311 g/cm^3
Diffractometer	SuperNova, Dual, Cu at zero, AtlasS2
Radiation	Cu K_α, λ = 1.54178 Å
Temperature	173.0(1)°K
Scan type	ω-scan
Data collection range	$-8 < h < 9$, $-15 < k < 19$, $-24 < l < 22$; θ_{max} = 76.9°
Reflections measured	Total: 17921 Unique (n): 8027 Observed [I ≥ 2σ(I)]: 7104
Absorption coefficient	0.704 mm^{-1}
Minimum and maximum transmission	0.790, 1.000
No. of variables, p	541
Weighting scheme	$w = \frac{1}{\sigma^2(F_o^2) + (0.001P)^2 + 3.5P}$ $P = (F_o^2 + 2F_c^2)/3$
$R1 = \frac{\Sigma\|\|F_o\|-\|F_c\|\|}{\Sigma\|F_o\|}$ (for all reflections)	0.0691 0.0627 (for observed data)
$wR2 = \sqrt{\frac{\Sigma[w(F_o^2 - F_c^2)^2]}{\Sigma w(F_o^2)^2}}$ (for all reflections)	0.1424 0.1395 (for observed data)
Goof = S = $\sqrt{\frac{\Sigma[w(F_o^2 - F_c^2)^2]}{n - p}}$	1.056
Largest and mean Δ/σ	0.000, 0.000
Residual extrema in final difference map	-0.258 to 0.348 e Å^{-3}

Table 2. Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

Atoms	*x*	*y*	*z*	$U_{eq.}$
F(1)	1.0163(3)	0.86393(12)	0.73545(9)	0.0504(5)
F(2)	0.2321(3)	0.13801(13)	-0.21801(10)	0.0555(5)
O(1)	0.3112(2)	0.62529(13)	0.33973(10)	0.0332(4)
O(2)	-0.0474(3)	0.28119(15)	0.17231(11)	0.0409(5)
N(1)	0.5247(3)	0.86894(15)	0.59157(13)	0.0336(5)
N(2)	0.4724(3)	0.73466(14)	0.45386(12)	0.0270(4)
N(3)	-0.1005(3)	0.28172(15)	-0.08086(12)	0.0311(5)
N(4)	-0.0072(3)	0.27658(15)	0.05707(12)	0.0267(4)
C(1)	0.4371(3)	0.64740(16)	0.39640(13)	0.0240(5)
C(2)	0.5550(3)	0.57489(15)	0.40828(13)	0.0235(5)
C(3)	0.7724(3)	0.60271(15)	0.43274(13)	0.0210(4)
C(4)	0.8391(3)	0.67431(16)	0.51354(13)	0.0220(4)
C(5)	0.9373(3)	0.76535(17)	0.53081(14)	0.0278(5)
C(6)	0.9981(4)	0.82944(18)	0.60577(15)	0.0330(6)
C(7)	0.9591(4)	0.80053(18)	0.66233(14)	0.0327(6)
C(8)	0.8644(4)	0.71159(18)	0.64870(14)	0.0307(5)
C(9)	0.8034(3)	0.64870(16)	0.57331(13)	0.0250(5)
C(10)	0.8692(3)	0.62022(17)	0.37091(13)	0.0257(5)
C(11)	0.8787(4)	0.51226(17)	0.33596(14)	0.0288(5)
C(12)	0.8617(3)	0.50798(16)	0.41375(13)	0.0244(5)
C(13)	0.7776(4)	0.66560(18)	0.32305(14)	0.0315(5)
C(14)	0.7051(5)	0.7016(2)	0.28366(17)	0.0456(7)
C(15)	0.7562(3)	0.42163(17)	0.41411(14)	0.0272(5)
C(16)	0.6701(4)	0.34981(18)	0.41152(16)	0.0333(6)
C(17)	0.3778(3)	0.81227(16)	0.45977(14)	0.0267(5)
C(18)	0.2611(4)	0.82328(19)	0.40132(16)	0.0330(6)
C(19)	0.1822(4)	0.9075(2)	0.41465(19)	0.0405(7)
C(20)	0.2176(4)	0.97878(19)	0.4842(2)	0.0425(7)
C(21)	0.3335(4)	0.96884(18)	0.54651(18)	0.0358(6)
C(22)	0.3744(4)	1.03807(19)	0.6216(2)	0.0467(8)
C(23)	0.4844(5)	1.0215(2)	0.67808(19)	0.0485(8)
C(24)	0.5580(5)	0.9362(2)	0.66085(17)	0.0429(7)
C(25)	0.4138(4)	0.88510(17)	0.53473(15)	0.0293(5)
C(26)	0.0222(3)	0.24841(17)	0.11711(14)	0.0270(5)
C(27)	0.1394(3)	0.16968(17)	0.10997(14)	0.0270(5)
C(28)	0.3334(3)	0.18025(16)	0.08829(14)	0.0255(5)
C(29)	0.3163(3)	0.16981(16)	0.00648(14)	0.0263(5)
C(30)	0.3896(4)	0.24292(18)	-0.01401(15)	0.0315(5)
C(31)	0.3649(4)	0.23230(19)	-0.08939(16)	0.0368(6)

(Table 2. continued)

Atoms	*x*	*y*	*z*	$U_{eq.}$
C(32)	0.2645(4)	0.1478(2)	-0.14340(15)	0.0366(6)
C(33)	0.1929(4)	0.07291(19)	-0.12679(16)	0.0407(6)
C(34)	0.2201(4)	0.08490(18)	-0.05085(16)	0.0351(6)
C(35)	0.4807(3)	0.26542(17)	0.15033(15)	0.0299(5)
C(36)	0.5342(4)	0.1976(2)	0.19127(17)	0.0382(6)
C(37)	0.4577(4)	0.11317(18)	0.11385(16)	0.0340(6)
C(38)	0.4176(4)	0.35457(18)	0.18924(15)	0.0328(6)
C(39)	0.3617(4)	0.4265(2)	0.21814(16)	0.0391(6)
C(40)	0.3653(4)	0.02150(19)	0.11015(17)	0.0377(6)
C(41)	0.2901(5)	-0.0532(2)	0.1056(2)	0.0452(7)
C(42)	-0.0978(3)	0.35159(16)	0.05170(14)	0.0250(5)
C(43)	-0.1414(4)	0.42200(18)	0.11185(15)	0.0311(5)
C(44)	-0.2245(4)	0.49701(19)	0.10097(17)	0.0378(6)
C(45)	-0.2644(4)	0.50234(18)	0.03141(17)	0.0357(6)
C(46)	-0.2251(3)	0.43013(17)	-0.03224(15)	0.0298(5)
C(47)	-0.2651(4)	0.4294(2)	-0.10654(17)	0.0373(6)
C(48)	-0.2221(4)	0.3576(2)	-0.16462(17)	0.0415(7)
C(49)	-0.1400(4)	0.2853(2)	-0.14903(16)	0.0390(6)
C(50)	-0.1420(3)	0.35411(16)	-0.02246(14)	0.0256(5)

*$U_{eq.}$ defined as one third of the trace of the orthogonalized **U** tensor.

Table 3. Bond lengths (Å) and bond angles (°)

F(1)-C(7)	1.361(3)	F(2)-C(32)	1.372(3)
O(1)-C(1)	1.224(3)	O(2)-C(26)	1.219(3)
N(1)-C(24)	1.327(3)	N(3)-C(49)	1.323(3)
N(1)-C(25)	1.364(4)	N(3)-C(50)	1.365(3)
N(2)-C(1)	1.358(3)	N(4)-C(26)	1.366(3)
N(2)-C(17)	1.403(3)	N(4)-C(42)	1.399(3)
C(1)-C(2)	1.519(3)	C(26)-C(27)	1.517(3)
C(2)-C(3)	1.543(3)	C(27)-C(28)	1.543(3)
C(3)-C(4)	1.513(3)	C(28)-C(29)	1.513(3)
C(3)-C(10)	1.567(3)	C(28)-C(35)	1.567(3)
C(3)-C(12)	1.586(3)	C(28)-C(37)	1.581(3)
C(4)-C(5)	1.390(3)	C(29)-C(34)	1.388(3)
C(4)-C(9)	1.397(3)	C(29)-C(30)	1.394(3)
C(5)-C(6)	1.392(3)	C(30)-C(31)	1.385(4)
C(6)-C(7)	1.373(4)	C(31)-C(32)	1.371(4)
C(7)-C(8)	1.369(4)	C(32)-C(33)	1.367(4)
C(8)-C(9)	1.393(3)	C(33)-C(34)	1.390(4)
C(10)-C(13)	1.457(3)	C(35)-C(38)	1.456(4)
C(10)-C(11)	1.549(3)	C(35)-C(36)	1.551(4)
C(11)-C(12)	1.551(3)	C(36)-C(37)	1.551(4)
C(12)-C(15)	1.456(3)	C(37)-C(40)	1.458(4)
C(13)-C(14)	1.175(4)	C(38)-C(39)	1.182(4)
C(15)-C(16)	1.184(4)	C(40)-C(41)	1.177(4)
C(17)-C(18)	1.369(4)	C(42)-C(43)	1.376(3)
C(17)-C(25)	1.436(3)	C(42)-C(50)	1.431(3)
C(18)-C(19)	1.412(4)	C(43)-C(44)	1.406(4)
C(19)-C(20)	1.355(5)	C(44)-C(45)	1.361(4)
C(20)-C(21)	1.416(4)	C(45)-C(46)	1.417(4)
C(21)-C(25)	1.413(3)	C(46)-C(50)	1.412(3)
C(21)-C(22)	1.416(4)	C(46)-C(47)	1.413(4)
C(22)-C(23)	1.359(5)	C(47)-C(48)	1.361(4)
C(23)-C(24)	1.403(4)	C(48)-C(49)	1.403(4)
C(24)-N(1)-C(25)	117.5(2)	C(49)-N(3)-C(50)	117.1(2)
C(1)-N(2)-C(17)	128.0(2)	C(26)-N(4)-C(42)	127.5(2)
O(1)-C(1)-N(2)	122.9(2)	O(2)-C(26)-N(4)	122.6(2)
O(1)-C(1)-C(2)	120.9(2)	O(2)-C(26)-C(27)	121.1(2)
N(2)-C(1)-C(2)	116.1(2)	N(4)-C(26)-C(27)	116.2(2)
C(1)-C(2)-C(3)	119.65(18)	C(26)-C(27)-C(28)	119.09(19)
C(4)-C(3)-C(2)	113.08(18)	C(29)-C(28)-C(27)	112.8(2)
C(4)-C(3)-C(10)	117.36(18)	C(29)-C(28)-C(35)	117.6(2)

(Table 3. continued)

C(2)-C(3)-C(10)	113.82(19)	C(27)-C(28)-C(35)	112.9(2)
C(4)-C(3)-C(12)	115.02(18)	C(29)-C(28)-C(37)	115.7(2)
C(2)-C(3)-C(12)	108.70(17)	C(27)-C(28)-C(37)	109.09(19)
C(10)-C(3)-C(12)	85.82(16)	C(35)-C(28)-C(37)	85.91(18)
C(5)-C(4)-C(9)	118.3(2)	C(34)-C(29)-C(30)	118.2(2)
C(5)-C(4)-C(3)	122.2(2)	C(34)-C(29)-C(28)	119.0(2)
C(9)-C(4)-C(3)	119.5(2)	C(30)-C(29)-C(28)	122.7(2)
C(4)-C(5)-C(6)	120.9(2)	C(31)-C(30)-C(29)	121.2(2)
C(7)-C(6)-C(5)	118.4(2)	C(32)-C(31)-C(30)	118.0(2)
F(1)-C(7)-C(8)	118.5(2)	C(33)-C(32)-C(31)	123.4(3)
F(1)-C(7)-C(6)	118.2(2)	C(33)-C(32)-F(2)	118.1(2)
C(8)-C(7)-C(6)	123.3(2)	C(31)-C(32)-F(2)	118.5(3)
C(7)-C(8)-C(9)	117.5(2)	C(32)-C(33)-C(34)	117.7(2)
C(8)-C(9)-C(4)	121.7(2)	C(29)-C(34)-C(33)	121.5(2)
C(13)-C(10)-C(11)	119.4(2)	C(38)-C(35)-C(36)	121.4(2)
C(13)-C(10)-C(3)	120.2(2)	C(38)-C(35)-C(28)	119.1(2)
C(11)-C(10)-C(3)	89.62(17)	C(36)-C(35)-C(28)	88.83(18)
C(10)-C(11)-C(12)	87.64(17)	C(35)-C(36)-C(37)	87.5(2)
C(15)-C(12)-C(11)	118.1(2)	C(40)-C(37)-C(36)	120.8(3)
C(15)-C(12)-C(3)	119.92(19)	C(40)-C(37)-C(28)	119.1(2)
C(11)-C(12)-C(3)	88.84(17)	C(36)-C(37)-C(28)	88.32(18)
C(14)-C(13)-C(10)	179.0(3)	C(39)-C(38)-C(35)	177.3(3)
C(16)-C(15)-C(12)	177.5(3)	C(41)-C(40)-C(37)	178.7(3)
C(18)-C(17)-N(2)	125.7(2)	C(43)-C(42)-N(4)	124.8(2)
C(18)-C(17)-C(25)	119.7(2)	C(43)-C(42)-C(50)	119.4(2)
N(2)-C(17)-C(25)	114.6(2)	N(4)-C(42)-C(50)	115.8(2)
C(17)-C(18)-C(19)	119.8(3)	C(42)-C(43)-C(44)	120.4(2)
C(20)-C(19)-C(18)	122.0(3)	C(45)-C(44)-C(43)	121.5(2)
C(19)-C(20)-C(21)	119.8(2)	C(44)-C(45)-C(46)	119.8(2)
C(25)-C(21)-C(22)	116.8(3)	C(50)-C(46)-C(47)	117.1(2)
C(25)-C(21)-C(20)	119.3(3)	C(50)-C(46)-C(45)	119.5(2)
C(22)-C(21)-C(20)	124.0(3)	C(47)-C(46)-C(45)	123.4(2)
C(23)-C(22)-C(21)	119.8(3)	C(48)-C(47)-C(46)	119.8(2)
C(22)-C(23)-C(24)	119.3(3)	C(47)-C(48)-C(49)	118.7(3)
N(1)-C(24)-C(23)	123.4(3)	N(3)-C(49)-C(48)	124.3(3)
N(1)-C(25)-C(21)	123.2(2)	N(3)-C(50)-C(46)	123.0(2)
N(1)-C(25)-C(17)	117.4(2)	N(3)-C(50)-C(42)	117.5(2)
C(21)-C(25)-C(17)	119.4(2)	C(46)-C(50)-C(42)	119.4(2)
Hydrogen bondings			
H(2)···N(1)	2.19(3)	H(4)··N(3)	2.28(3)
N(2)-H(2)···N(1)	111(2)	N(4)-H(4)··N(3)	108(2)

Table 4. Anisotropic thermal parameters* (Å^2)

Atoms	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
F(1)	0.0717(12)	0.0373(9)	0.0229(8)	-0.0044(7)	0.0018(8)	-0.0036(8)
F(2)	0.0831(14)	0.0547(11)	0.0281(9)	0.0104(8)	0.0223(9)	0.0126(10)
O(1)	0.0302(9)	0.0330(9)	0.0303(9)	0.0063(7)	0.0006(7)	0.0102(7)
O(2)	0.0420(11)	0.0574(12)	0.0383(11)	0.0265(10)	0.0214(9)	0.0228(10)
N(1)	0.0417(12)	0.0253(10)	0.0317(12)	0.0062(9)	0.0141(9)	0.0038(9)
N(2)	0.0307(11)	0.0236(10)	0.0248(10)	0.0065(8)	0.0050(8)	0.0082(8)
N(3)	0.0379(12)	0.0272(10)	0.0276(11)	0.0092(9)	0.0071(9)	0.0071(9)
N(4)	0.0302(10)	0.0282(10)	0.0216(10)	0.0076(8)	0.0070(8)	0.0097(8)
C(1)	0.0228(11)	0.0246(11)	0.0251(11)	0.0082(9)	0.0093(9)	0.0042(9)
C(2)	0.0234(11)	0.0201(10)	0.0248(11)	0.0054(9)	0.0061(9)	0.0036(9)
C(3)	0.0215(10)	0.0198(10)	0.0226(11)	0.0079(9)	0.0073(8)	0.0037(8)
C(4)	0.0214(10)	0.0214(10)	0.0216(11)	0.0061(9)	0.0044(8)	0.0047(8)
C(5)	0.0302(12)	0.0250(11)	0.0281(12)	0.0093(10)	0.0094(10)	0.0023(9)
C(6)	0.0351(13)	0.0232(12)	0.0332(14)	0.0044(10)	0.0060(11)	-0.0031(10)
C(7)	0.0382(14)	0.0285(12)	0.0202(12)	-0.0011(10)	0.0011(10)	0.0032(10)
C(8)	0.0391(14)	0.0302(12)	0.0231(12)	0.0088(10)	0.0088(10)	0.0084(11)
C(9)	0.0276(12)	0.0216(11)	0.0244(11)	0.0073(9)	0.0057(9)	0.0029(9)
C(10)	0.0264(11)	0.0263(11)	0.0237(11)	0.0075(9)	0.0090(9)	0.0024(9)
C(11)	0.0308(12)	0.0290(12)	0.0261(12)	0.0070(10)	0.0114(10)	0.0080(10)
C(12)	0.0238(11)	0.0232(11)	0.0245(11)	0.0066(9)	0.0053(9)	0.0062(9)
C(13)	0.0413(14)	0.0283(12)	0.0268(12)	0.0103(10)	0.0140(11)	0.0047(11)
C(14)	0.068(2)	0.0403(16)	0.0319(15)	0.0165(13)	0.0099(14)	0.0143(15)
C(15)	0.0293(12)	0.0256(12)	0.0268(12)	0.0078(9)	0.0073(9)	0.0117(10)
C(16)	0.0353(14)	0.0243(12)	0.0396(15)	0.0104(11)	0.0097(11)	0.0056(10)
C(17)	0.0249(11)	0.0219(11)	0.0363(13)	0.0112(10)	0.0136(10)	0.0046(9)
C(18)	0.0286(12)	0.0339(13)	0.0440(15)	0.0202(12)	0.0141(11)	0.0085(10)
C(19)	0.0293(13)	0.0414(15)	0.064(2)	0.0314(15)	0.0164(13)	0.0126(12)
C(20)	0.0320(14)	0.0284(13)	0.080(2)	0.0271(14)	0.0281(14)	0.0142(11)
C(21)	0.0327(13)	0.0242(12)	0.0571(18)	0.0150(12)	0.0272(12)	0.0074(10)
C(22)	0.0472(17)	0.0231(13)	0.072(2)	0.0094(13)	0.0369(16)	0.0092(12)
C(23)	0.0554(19)	0.0315(14)	0.0493(18)	-0.0014(13)	0.0284(15)	0.0018(13)
C(24)	0.0530(18)	0.0321(14)	0.0363(15)	0.0036(12)	0.0159(13)	-0.0002(12)
C(25)	0.0293(12)	0.0232(11)	0.0395(14)	0.0118(10)	0.0186(10)	0.0046(9)
C(26)	0.0211(11)	0.0319(12)	0.0283(12)	0.0126(10)	0.0055(9)	0.0018(9)
C(27)	0.0243(11)	0.0281(12)	0.0309(12)	0.0141(10)	0.0063(9)	0.0029(9)
C(28)	0.0238(11)	0.0213(11)	0.0321(13)	0.0105(9)	0.0062(9)	0.0048(9)
C(29)	0.0239(11)	0.0226(11)	0.0318(13)	0.0077(10)	0.0097(9)	0.0057(9)
C(30)	0.0335(13)	0.0242(11)	0.0348(14)	0.0089(10)	0.0101(11)	0.0010(10)
C(31)	0.0428(15)	0.0304(13)	0.0410(15)	0.0145(11)	0.0179(12)	0.0055(11)

(Table 4. continued)

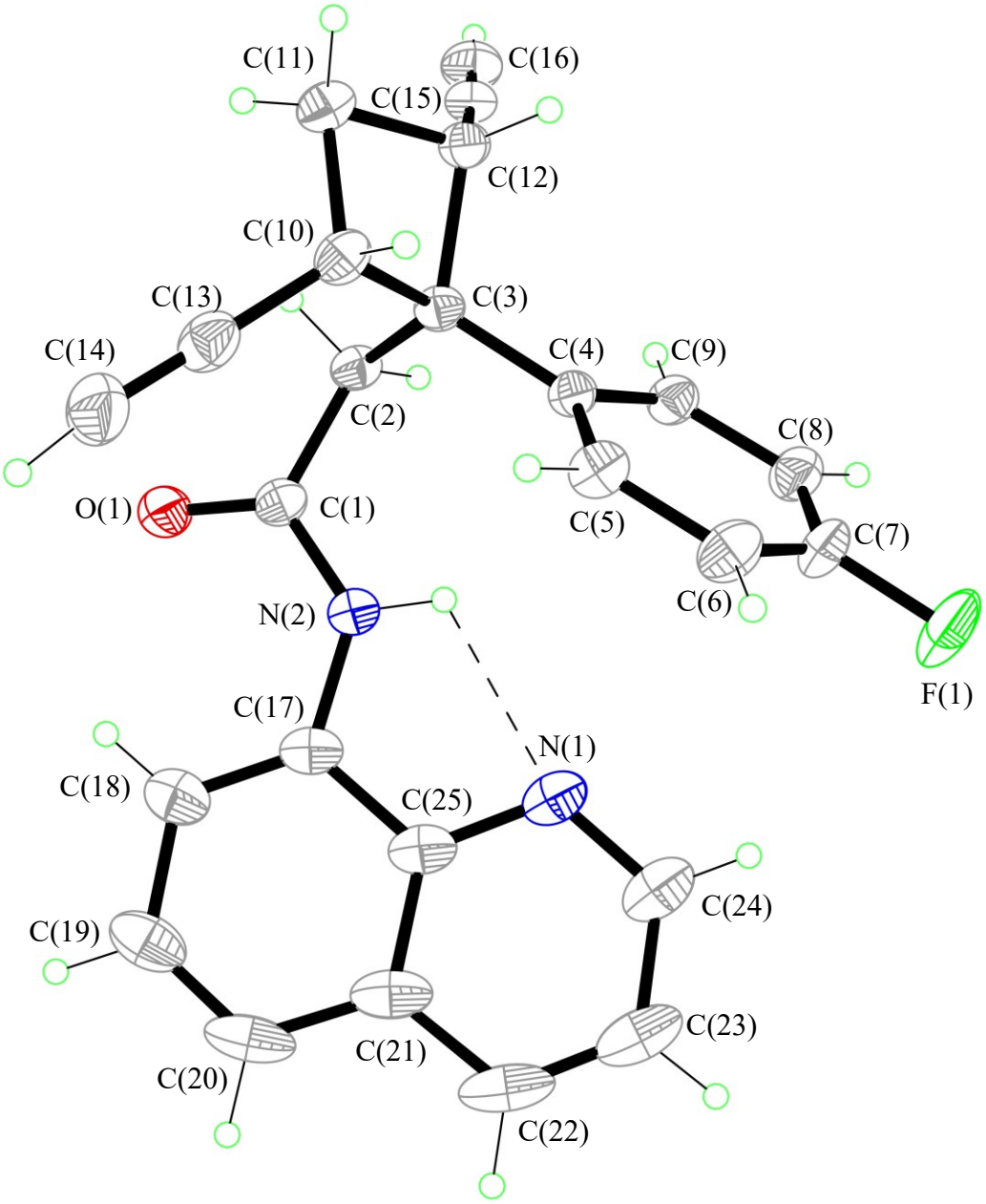
Atoms	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(32)	0.0468(16)	0.0364(14)	0.0269(13)	0.0076(11)	0.0168(11)	0.0116(12)
C(33)	0.0521(17)	0.0280(13)	0.0329(14)	0.0001(11)	0.0125(12)	0.0040(12)
C(34)	0.0415(15)	0.0228(12)	0.0381(15)	0.0074(11)	0.0126(12)	0.0013(10)
C(35)	0.0258(12)	0.0270(12)	0.0346(13)	0.0118(10)	0.0019(10)	0.0015(10)
C(36)	0.0314(13)	0.0382(14)	0.0436(16)	0.0193(12)	-0.0034(11)	0.0038(11)
C(37)	0.0280(12)	0.0298(13)	0.0487(16)	0.0186(12)	0.0085(11)	0.0103(10)
C(38)	0.0336(13)	0.0308(13)	0.0289(13)	0.0108(11)	-0.0021(10)	-0.0010(11)
C(39)	0.0468(16)	0.0309(14)	0.0307(14)	0.0057(11)	-0.0021(12)	0.0065(12)
C(40)	0.0357(14)	0.0325(14)	0.0491(17)	0.0187(12)	0.0079(12)	0.0145(11)
C(41)	0.0459(17)	0.0318(15)	0.063(2)	0.0241(14)	0.0084(14)	0.0096(13)
C(42)	0.0202(11)	0.0233(11)	0.0289(12)	0.0077(9)	0.0045(9)	0.0015(9)
C(43)	0.0293(12)	0.0308(12)	0.0287(13)	0.0059(10)	0.0062(10)	0.0059(10)
C(44)	0.0345(14)	0.0307(13)	0.0416(15)	0.0032(11)	0.0112(12)	0.0102(11)
C(45)	0.0312(13)	0.0247(12)	0.0509(17)	0.0130(11)	0.0077(12)	0.0103(10)
C(46)	0.0221(11)	0.0258(12)	0.0401(14)	0.0132(10)	0.0030(10)	0.0010(9)
C(47)	0.0334(14)	0.0361(14)	0.0474(16)	0.0249(13)	0.0017(12)	0.0056(11)
C(48)	0.0457(16)	0.0460(16)	0.0342(15)	0.0217(13)	0.0013(12)	0.0010(13)
C(49)	0.0493(16)	0.0375(14)	0.0272(13)	0.0098(11)	0.0061(12)	0.0071(12)
C(50)	0.0213(11)	0.0221(11)	0.0302(12)	0.0080(9)	0.0030(9)	0.0007(9)

*The exponent takes the form: $-2\pi^2\Sigma\Sigma U_{ij}h_ih_j\mathbf{a}_i^*\mathbf{a}_j^*$

Table 5. Coordinates and isotropic temperature factors* (Å^2) for H atoms

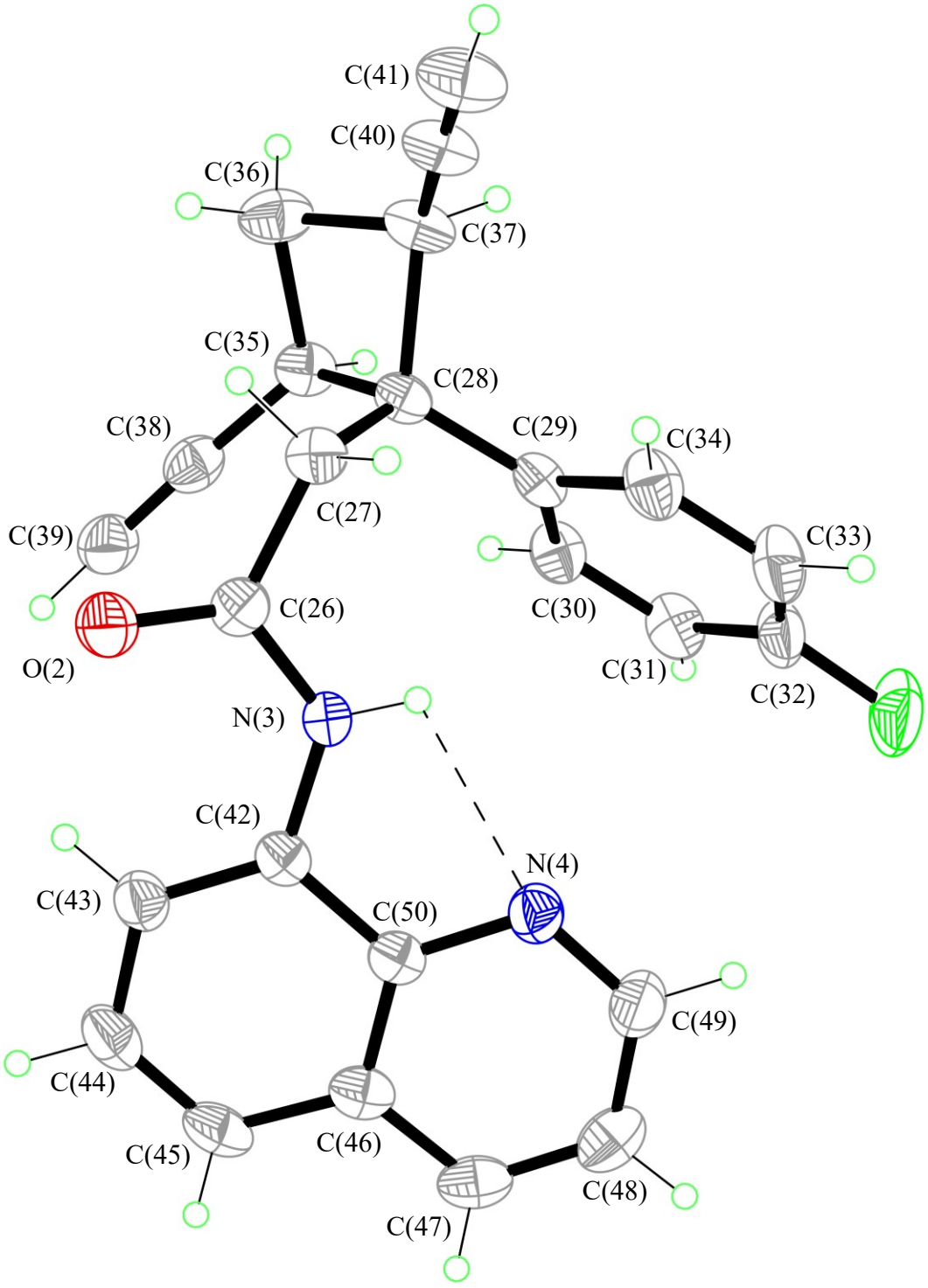
Atoms	*x*	*y*	*z*	$U_{eq.}$
H(2)	0.554(4)	0.743(2)	0.4963(18)	0.032
H(4)	0.037(4)	0.246(2)	0.0185(17)	0.032
H(2A)	0.5261	0.5191	0.3612	0.028
H(2B)	0.5111	0.5551	0.4461	0.028
H(5)	0.9627	0.7837	0.4918	0.033
H(6)	1.0635	0.8904	0.6173	0.040
H(8)	0.8415	0.6938	0.6883	0.037
H(9)	0.7372	0.5881	0.5625	0.030
H(10)	0.9978	0.6557	0.3952	0.031
H(11A)	0.9981	0.4988	0.3218	0.035
H(11B)	0.7723	0.4750	0.2949	0.035
H(12)	0.9883	0.5220	0.4460	0.029
H(14)	0.646(5)	0.734(2)	0.258(2)	0.055
H(16)	0.608(4)	0.292(2)	0.4095(18)	0.040
H(18)	0.2339	0.7754	0.3530	0.040
H(19)	0.1035	0.9143	0.3745	0.049
H(20)	0.1660	1.0341	0.4911	0.051
H(22)	0.3261	1.0948	0.6322	0.056
H(23)	0.5106	1.0663	0.7277	0.058
H(24)	0.6341	0.9262	0.7000	0.051
H(27A)	0.0632	0.1108	0.0724	0.032
H(27B)	0.1606	0.1617	0.1581	0.032
H(30)	0.4564	0.2999	0.0236	0.038
H(31)	0.4149	0.2810	-0.1029	0.044
H(33)	0.1282	0.0159	-0.1650	0.049
H(34)	0.1729	0.0349	-0.0381	0.042
H(35)	0.5861	0.2794	0.1279	0.036
H(36A)	0.4630	0.1982	0.2298	0.046
H(36B)	0.6690	0.2050	0.2106	0.046
H(37)	0.5592	0.1024	0.0855	0.041
H(39)	0.317(5)	0.484(2)	0.2404(19)	0.047
H(41)	0.231(5)	-0.115(3)	0.101(2)	0.054
H(43)	-0.1158	0.4200	0.1600	0.037
H(44)	-0.2529	0.5441	0.1423	0.045
H(45)	-0.3172	0.5532	0.0257	0.043
H(47)	-0.3206	0.4778	-0.1157	0.045
H(48)	-0.2467	0.3566	-0.2137	0.050
H(49)	-0.1115	0.2366	-0.1892	0.047

*The exponent takes the form: $-8\pi^2 U\sin^2\theta/\lambda^2$



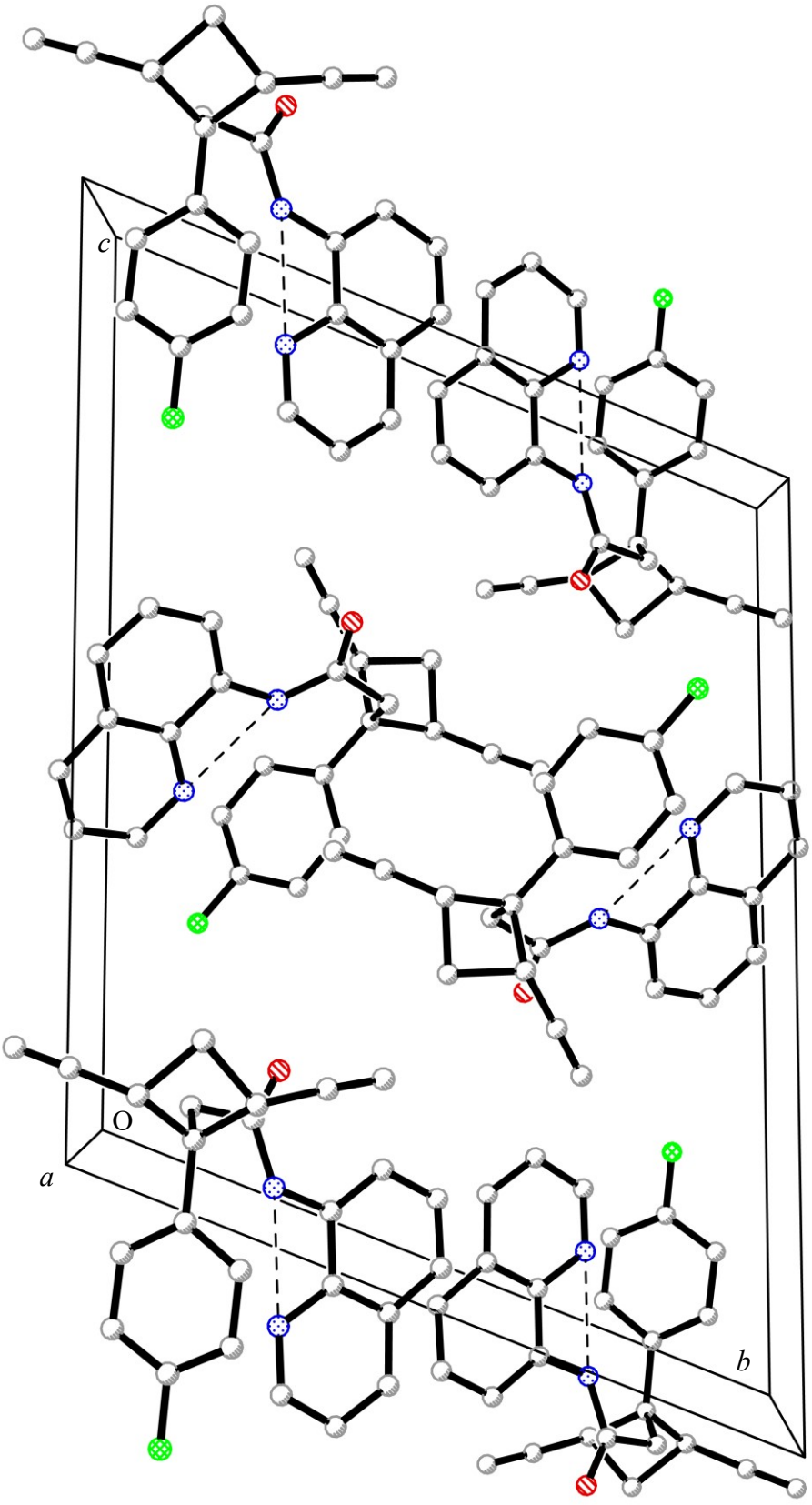
Molecule **I**

ORTEP drawing of $C_{25}H_{19}FN_2O$ with 50% probability ellipsoids, showing the atomic numbering scheme.

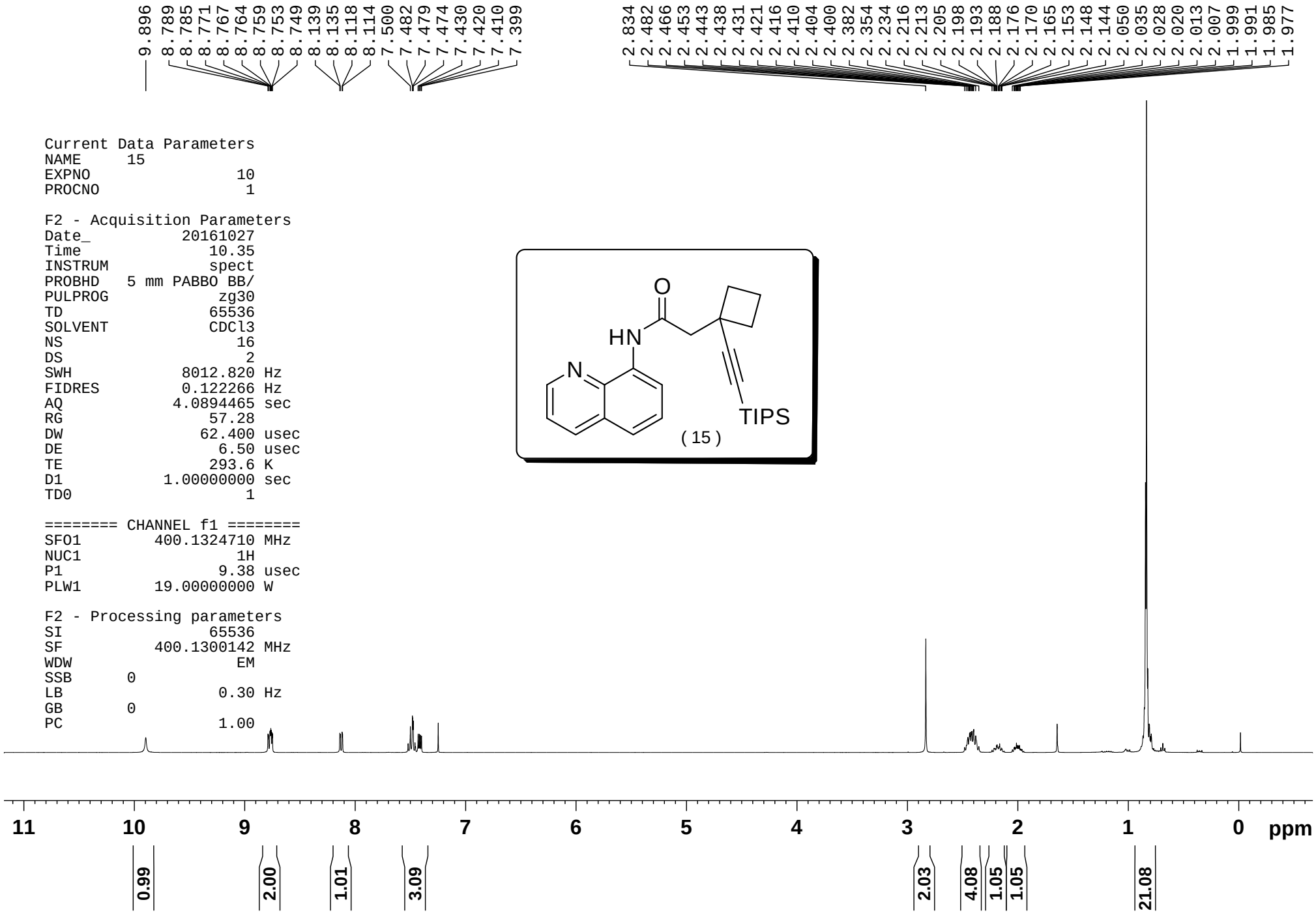


Molecule **II**

ORTEP drawing of $C_{25}H_{19}FN_2O$ with 50% probability ellipsoids, showing the atomic numbering scheme.



A packing view along the *a* direction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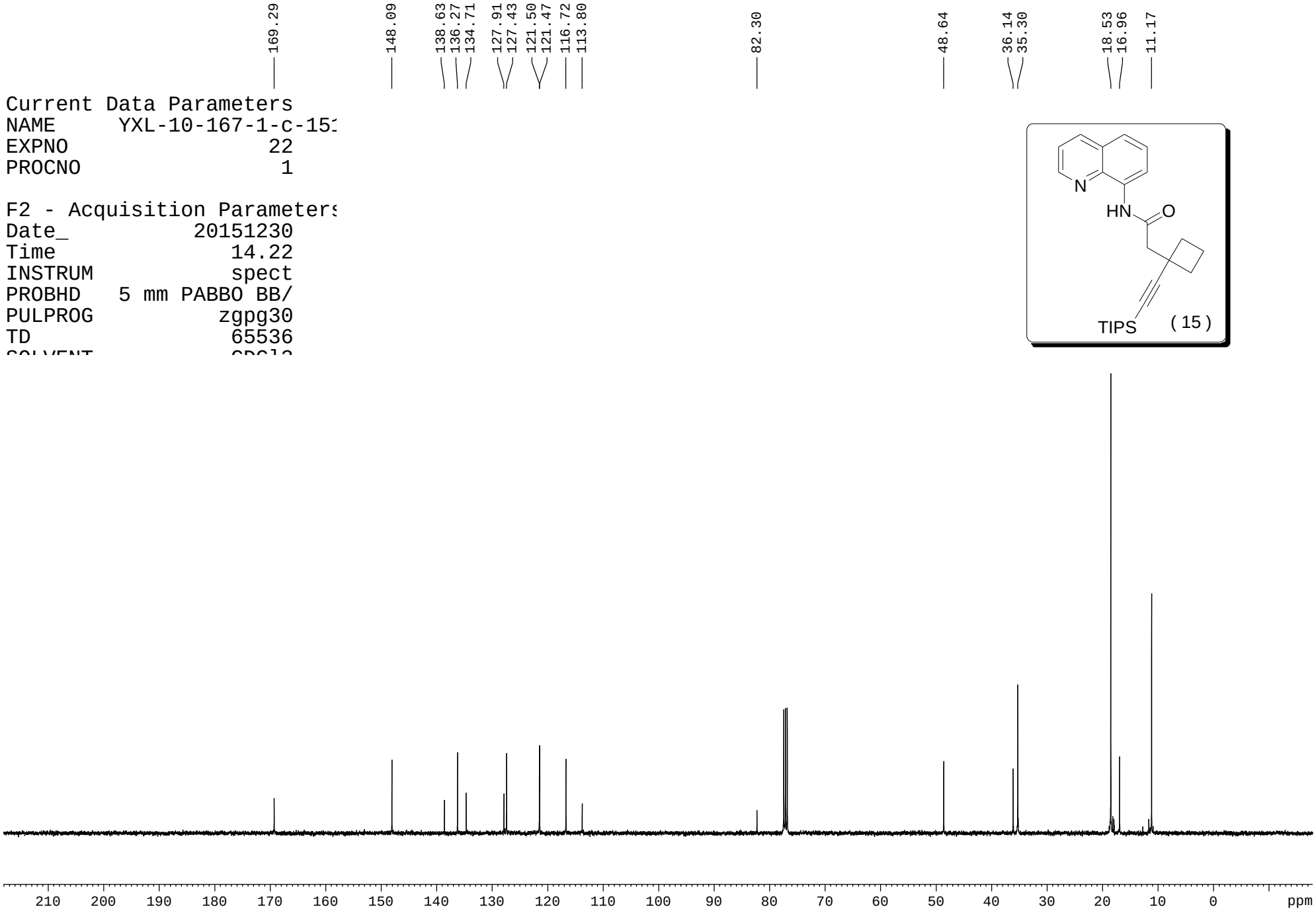
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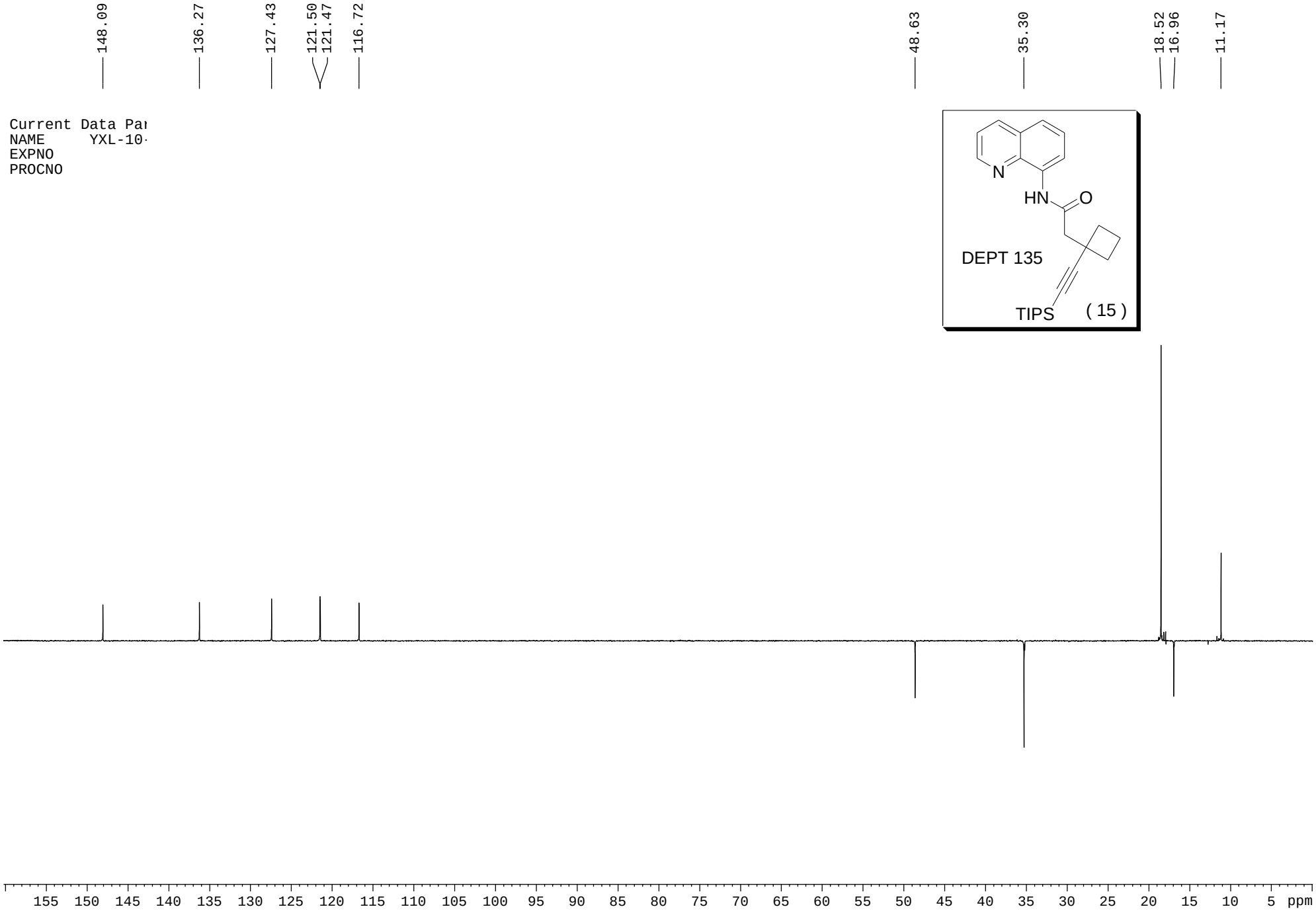
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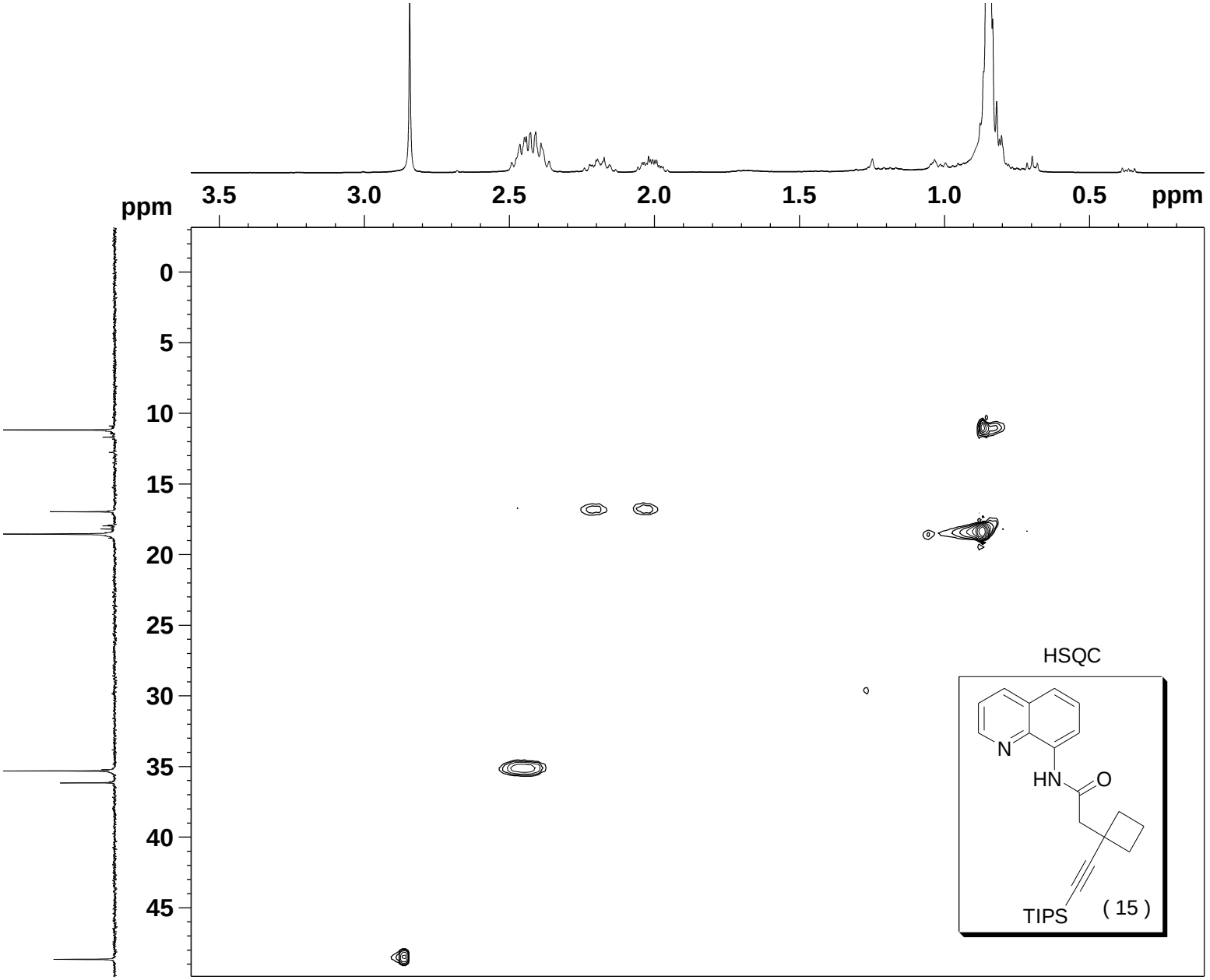
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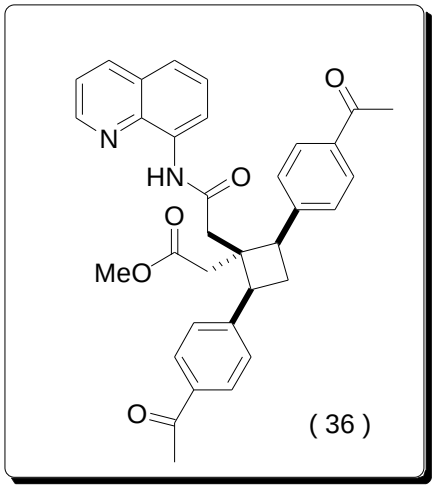

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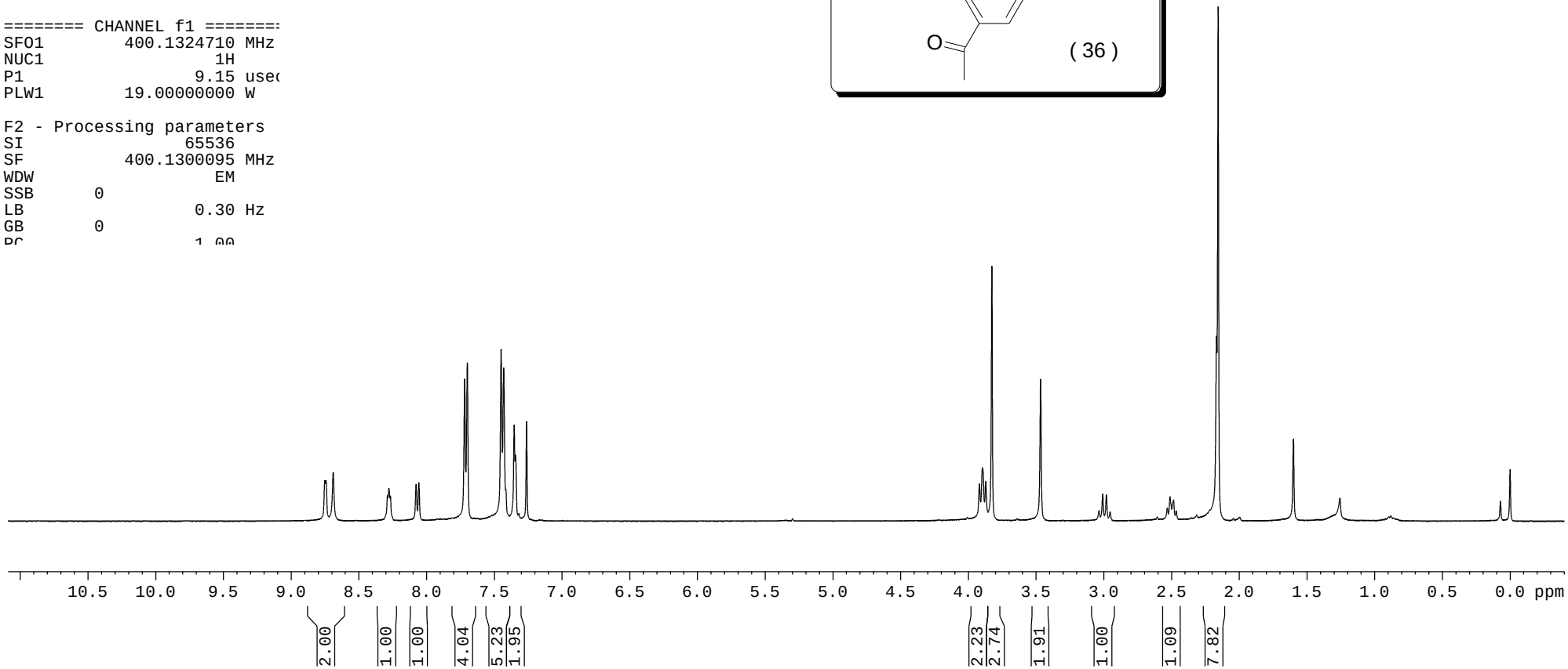

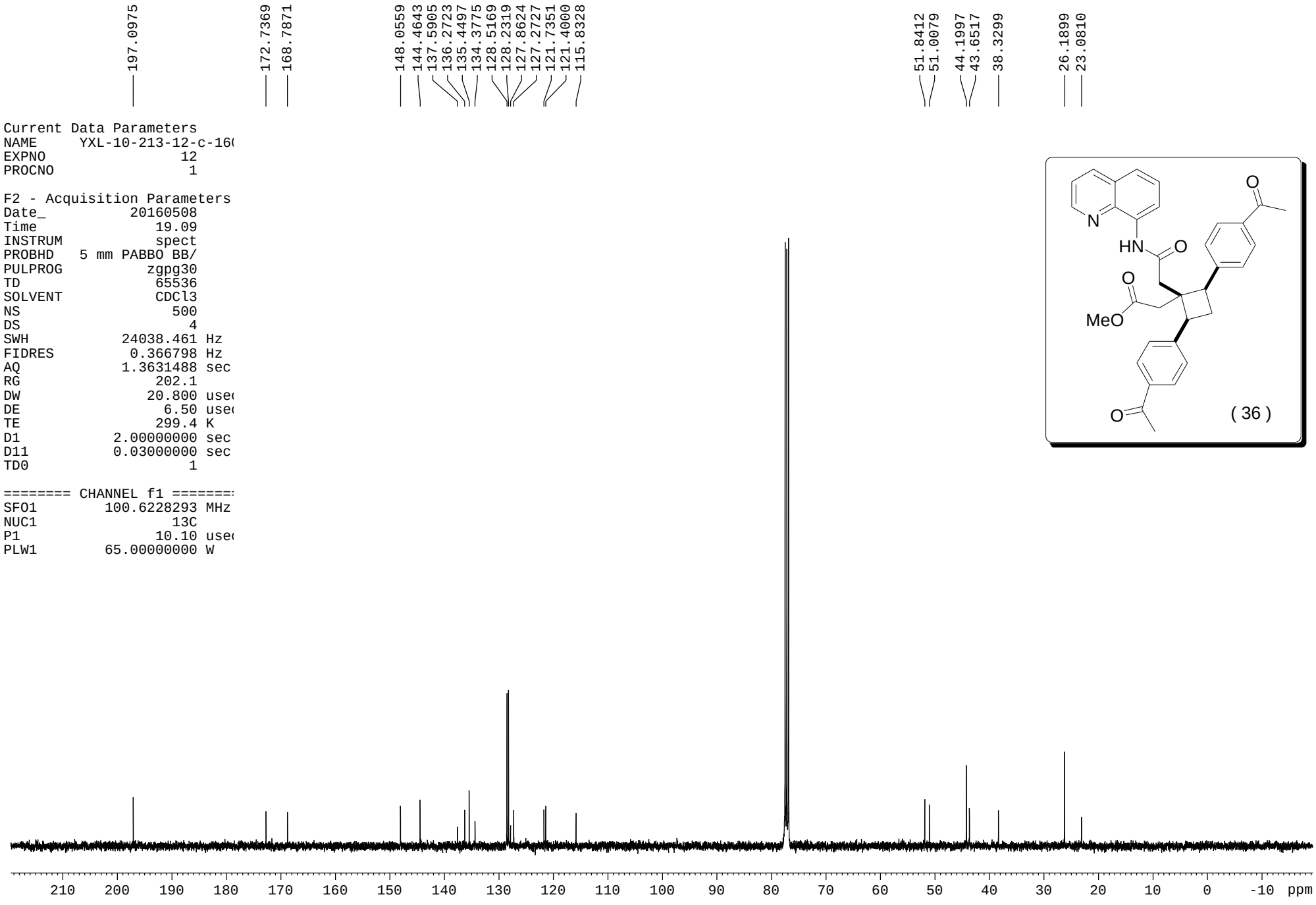
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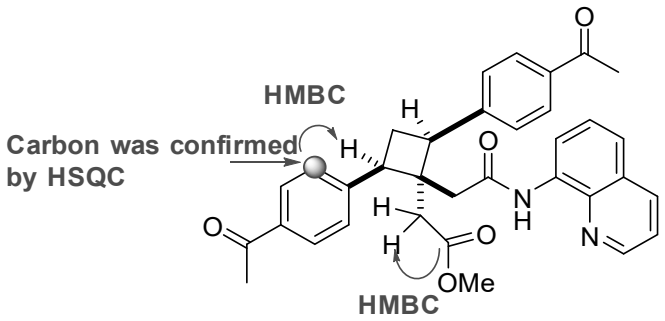
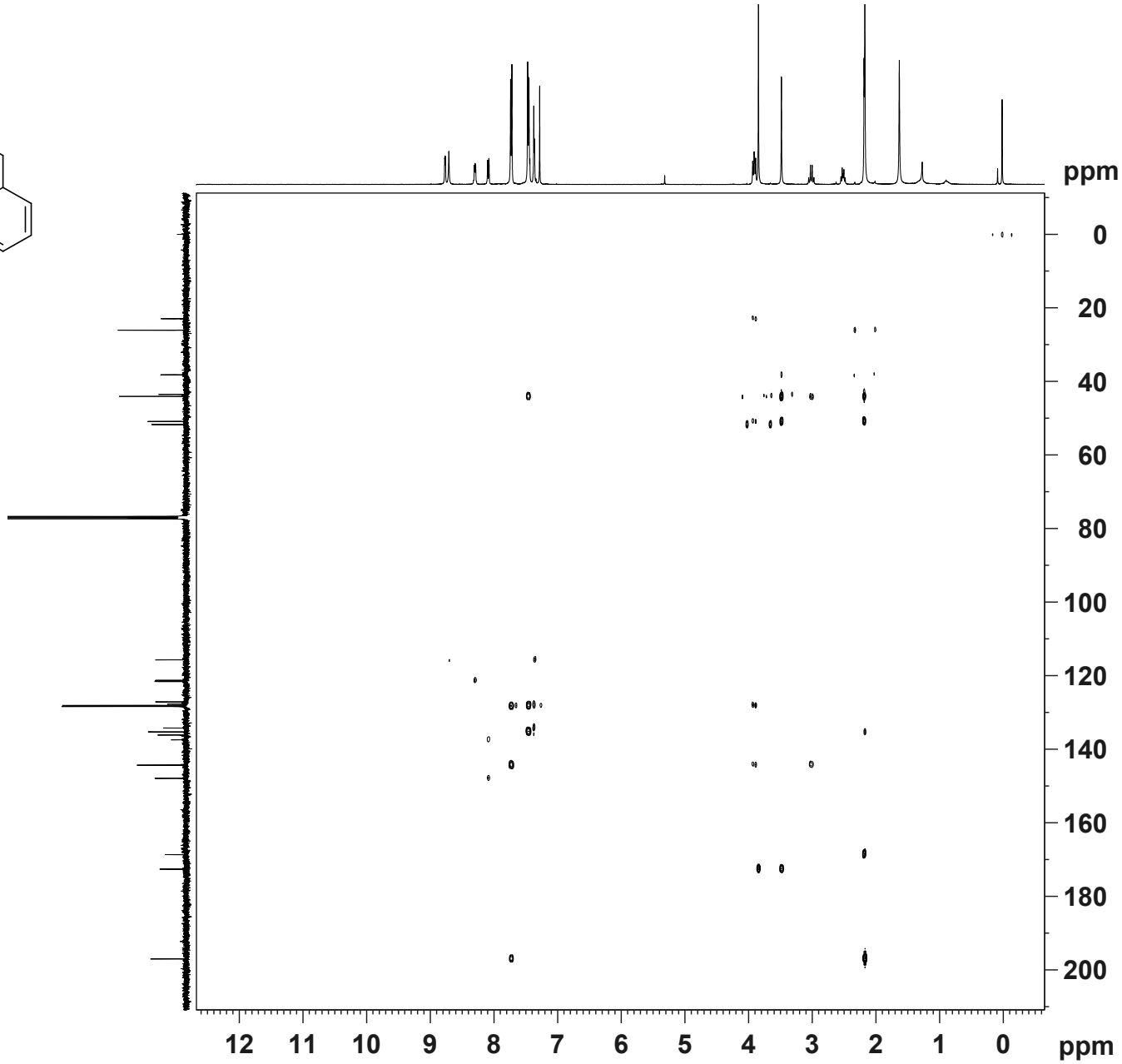
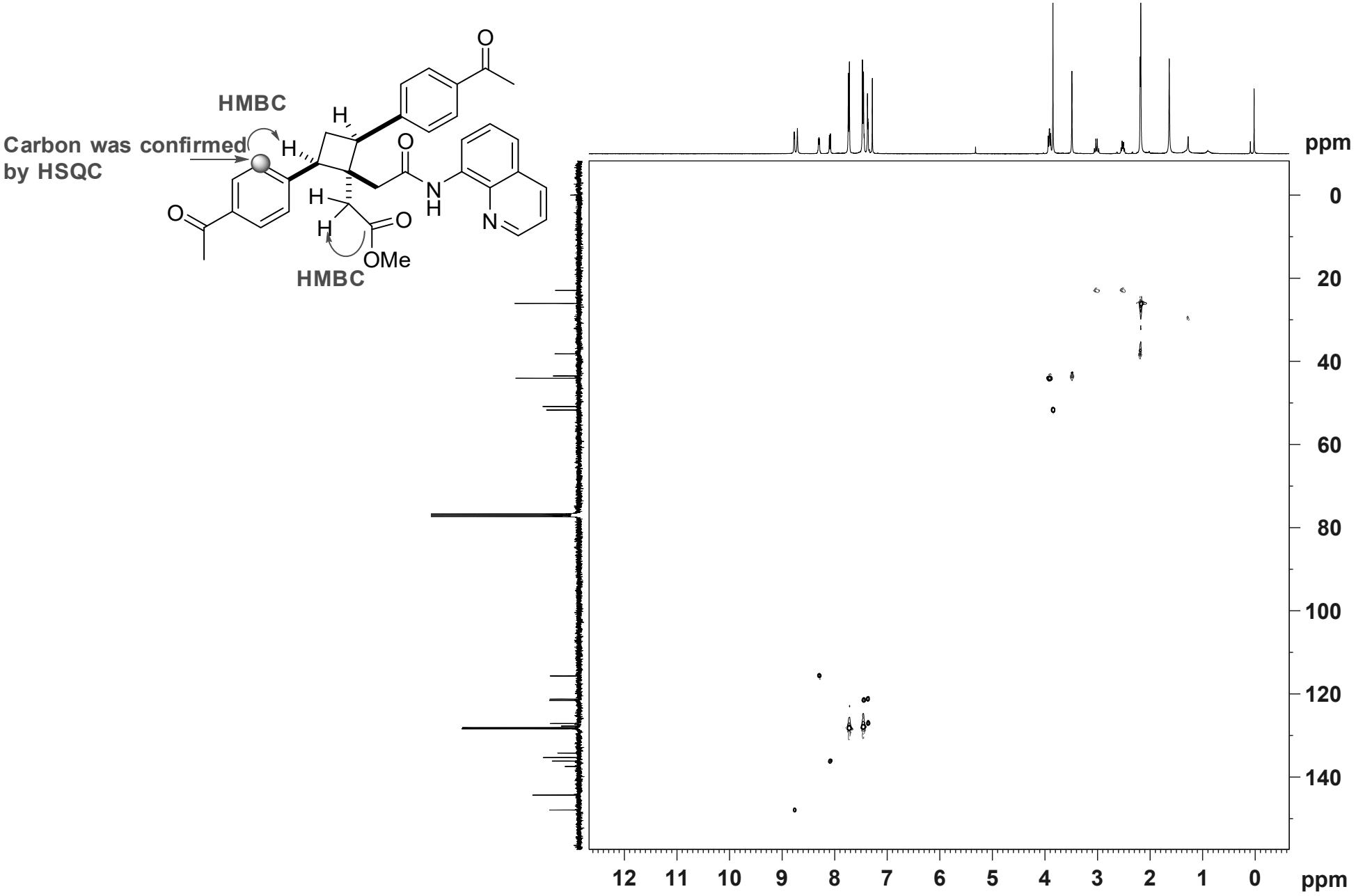
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.10 use(
PLW1 65.00000000 W
N
HN
O
O
O
MeO
O
O
(36)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

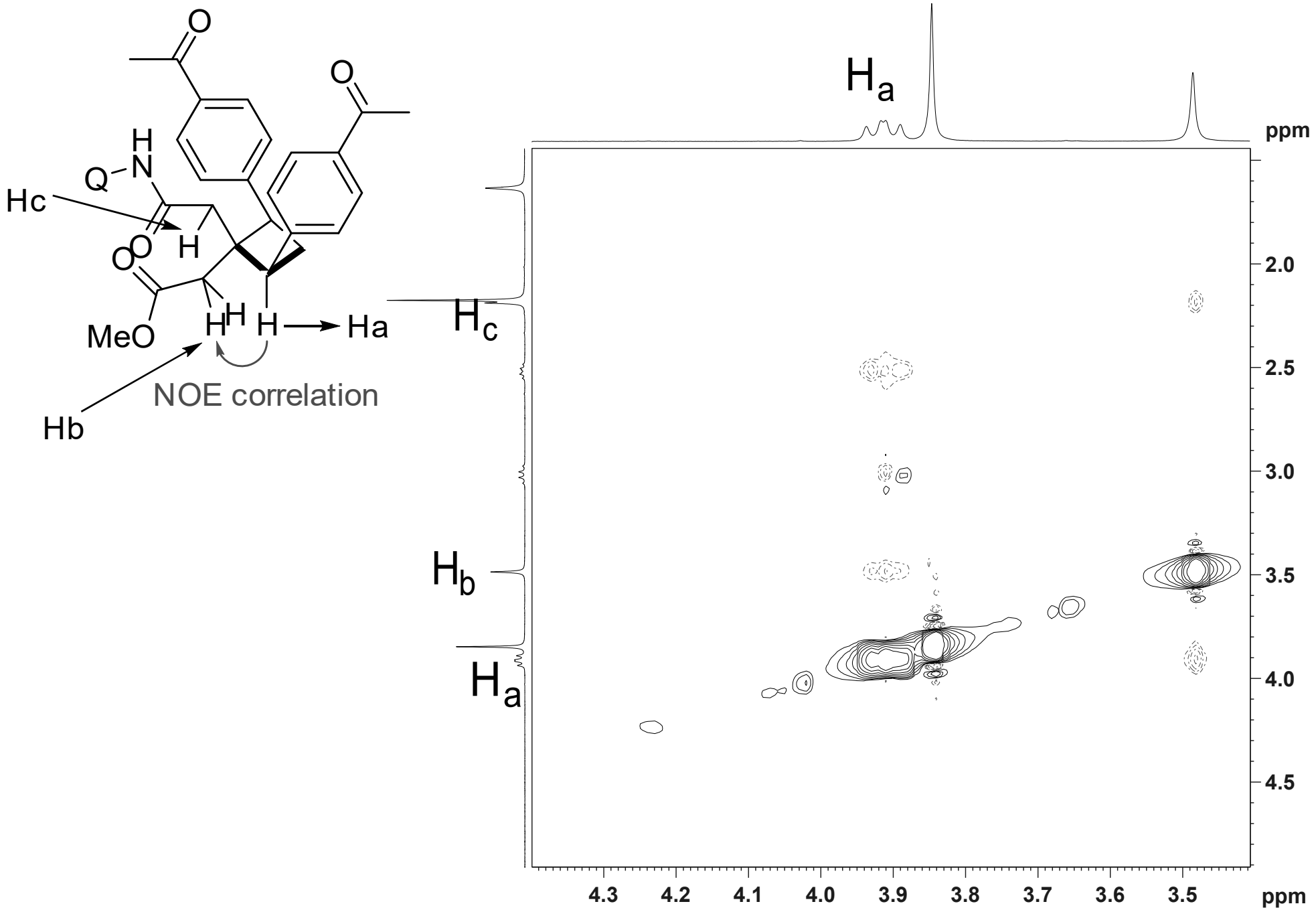
HMBCGPND CDCl3 {D:\NMR_DATA} RY 25





HSQCEDETGP CDCl3 {D:\NMR_DATA} RY 25



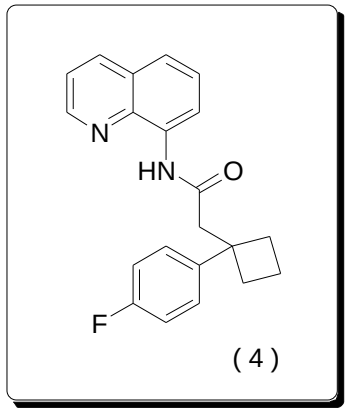

O
O
H
N
Q
Hc
O
O
H
MeO
H
H
H
Ha
Hb
NOE correlation
Ha
ppm
Hc
Hb
Ha
2.0
2.5
3.0
3.5
4.0
4.5
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
ppm

Current Data Parameters
NAME YXL-10-217-2-H-166
EXPNO 20
PROCNO 1

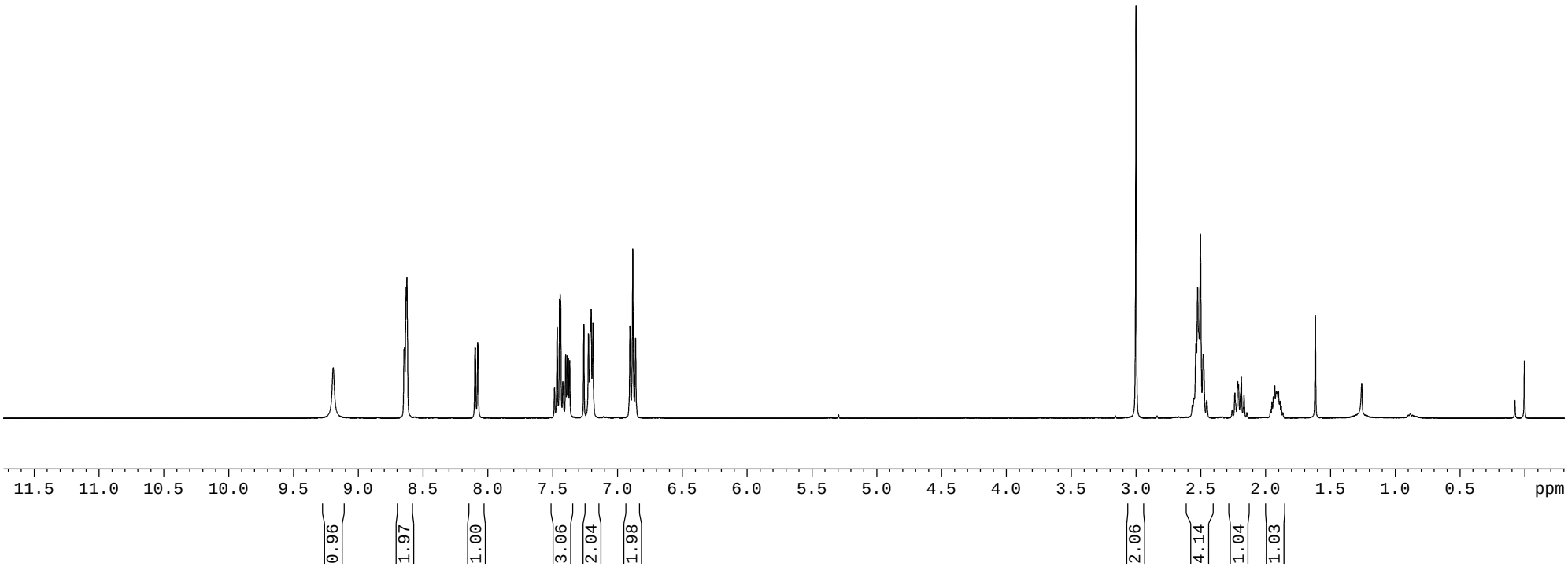
F2 - Acquisition Parameters
Date_ 20160508
Time 16.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 89.81
DW 62.400 use
DE 6.50 use
TE 298.5 K
D1 1.00000000 sec

9.1939 8.6470 8.6437 8.6304 8.6253 8.0996 8.0970 8.0790 8.0763 7.4659 7.4472 7.4430 7.4391 7.4222 7.4005 7.3900 7.3799 7.3694 7.2601 7.2246 7.2109 7.2034 7.1898 6.9043 6.8825 6.8608

2.9995 2.5651 2.5531 2.5369 2.5240 2.5161 2.5027 2.4816 2.4540 2.2371 2.2151 2.2092 2.1871 2.1660 1.9502 1.9401 1.9291 1.9173 1.9117 1.9081 1.9005 1.8890 1.8789 1.6162

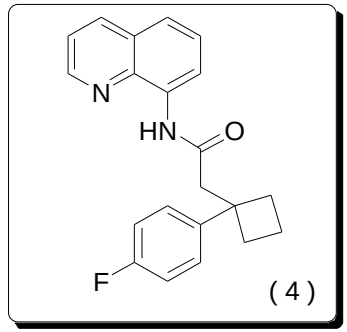


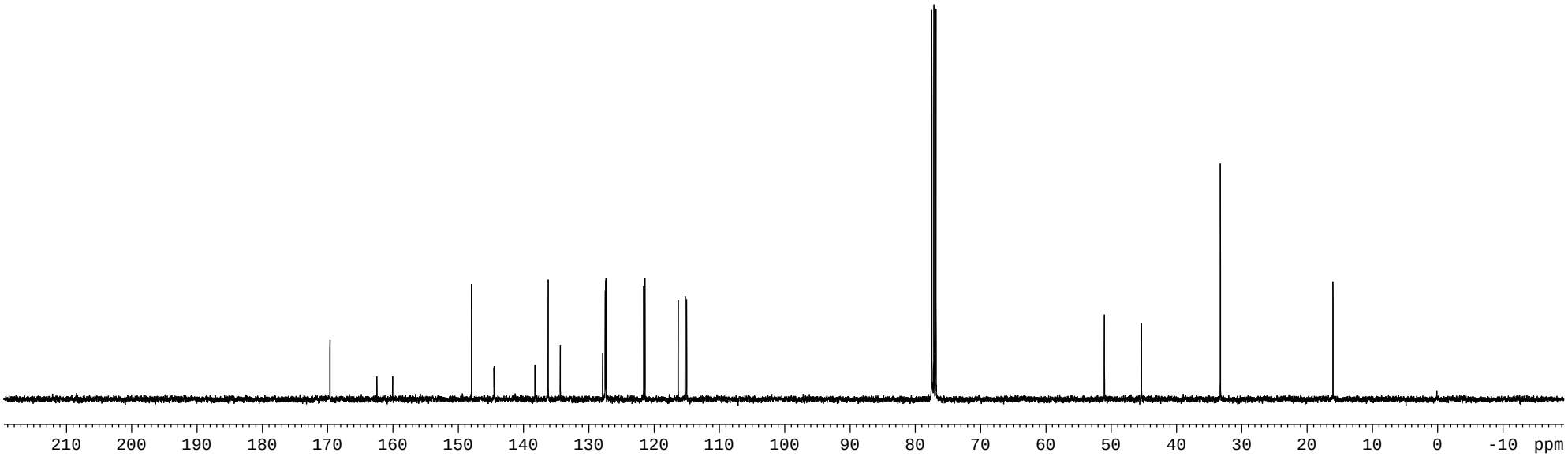
(4)

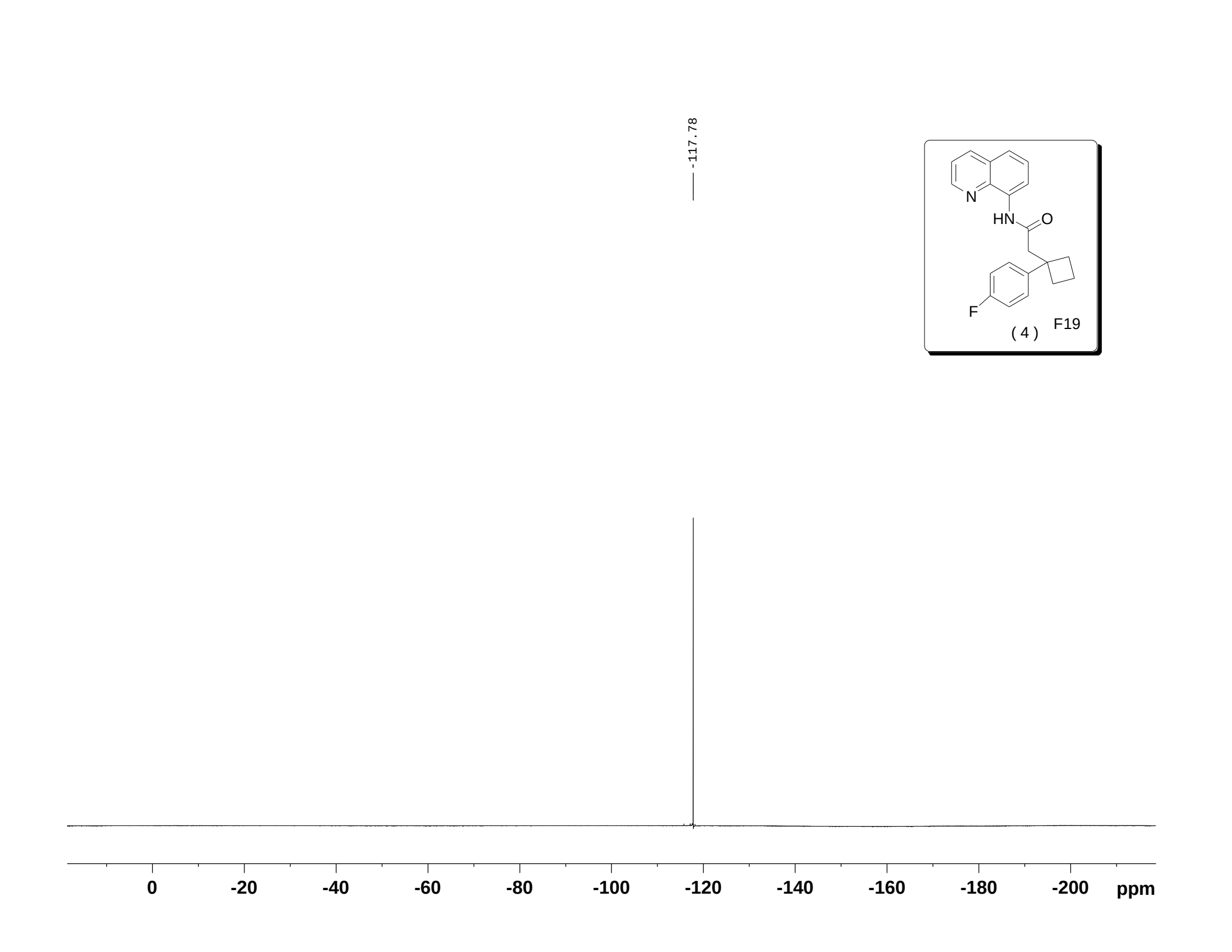


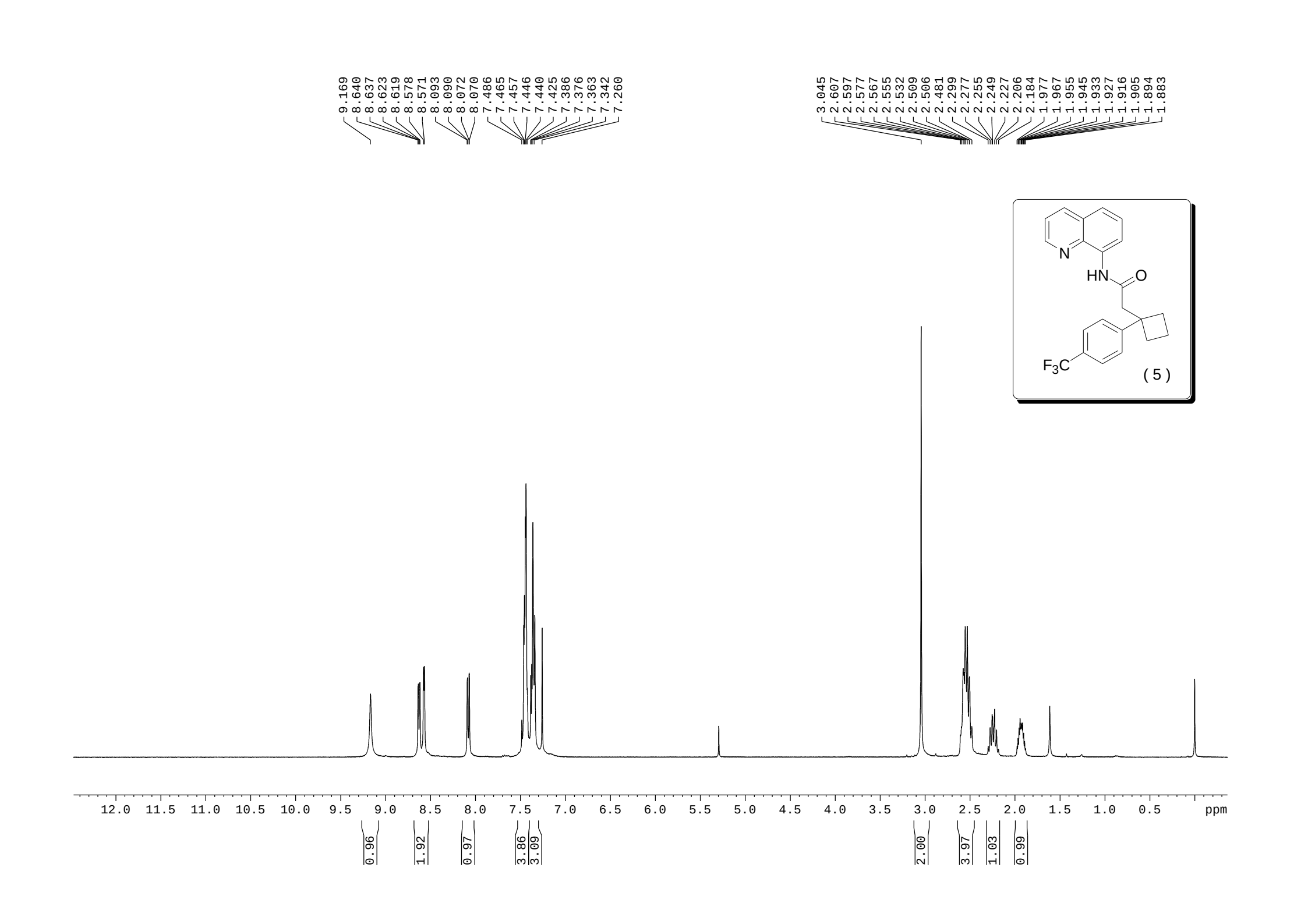
```
Current Data Parameters
NAME      YXL-10-217-1-C-16(
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160506
Time               22.16
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  300
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                202.1
DW               20.800 use
DE                6.50 use
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1
```

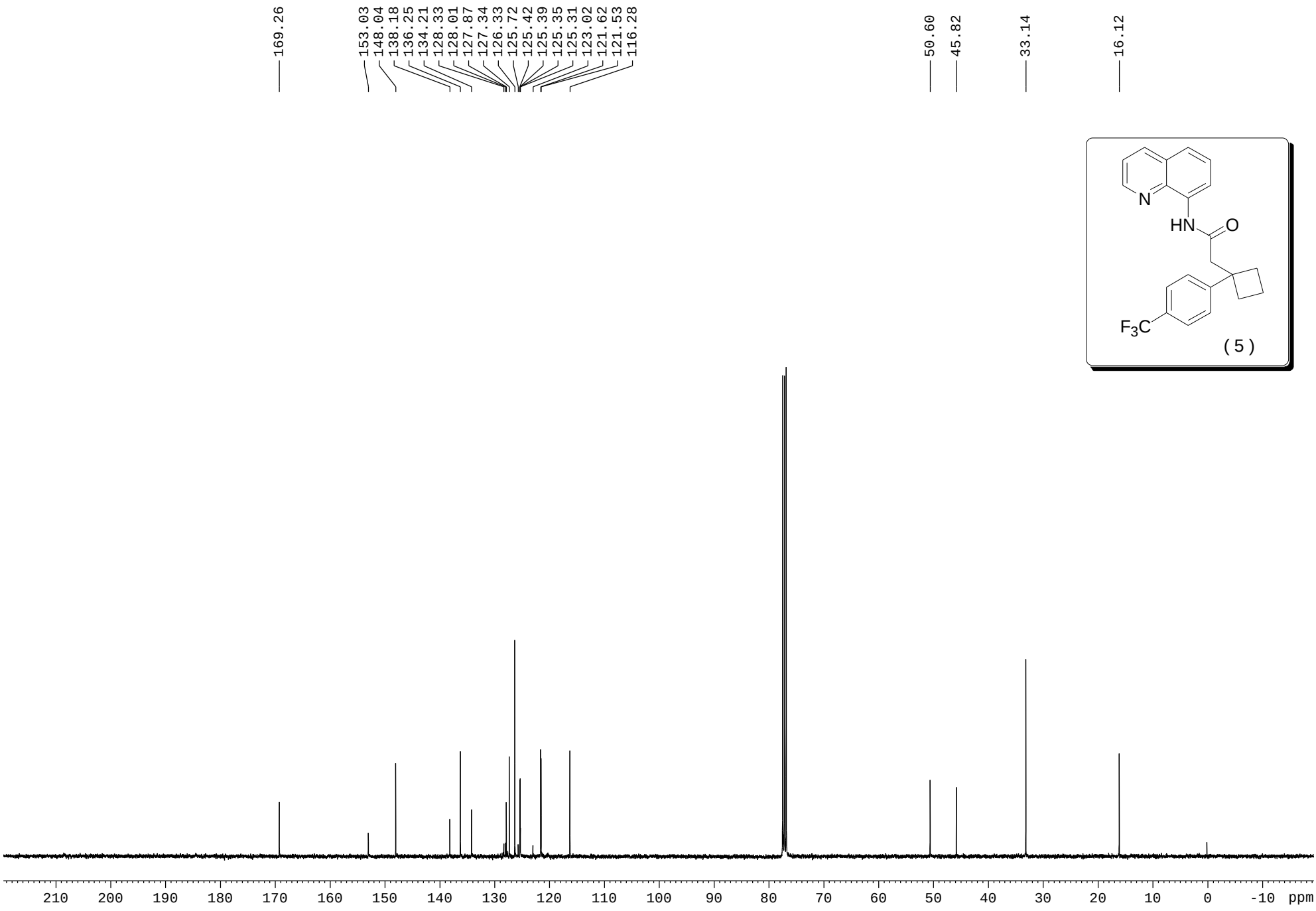
169.6488 162.4733 160.0521 147.9641 144.5216 144.4919 138.2542 136.2310 134.3719 127.8869 127.5017 127.4222 127.3809 121.5958 121.4075 116.3222 115.2353 115.0244 51.0415 45.3586 33.2783 16.0327

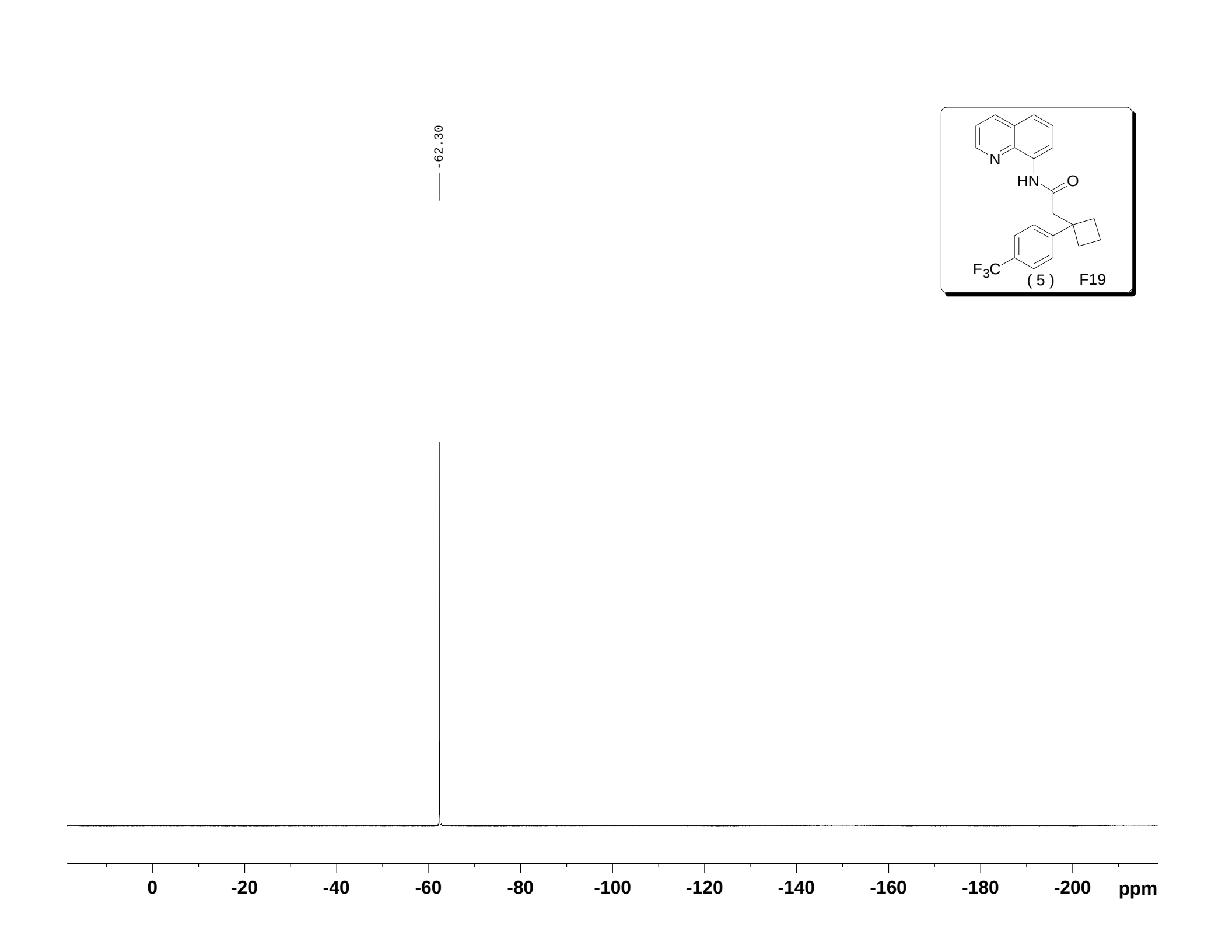


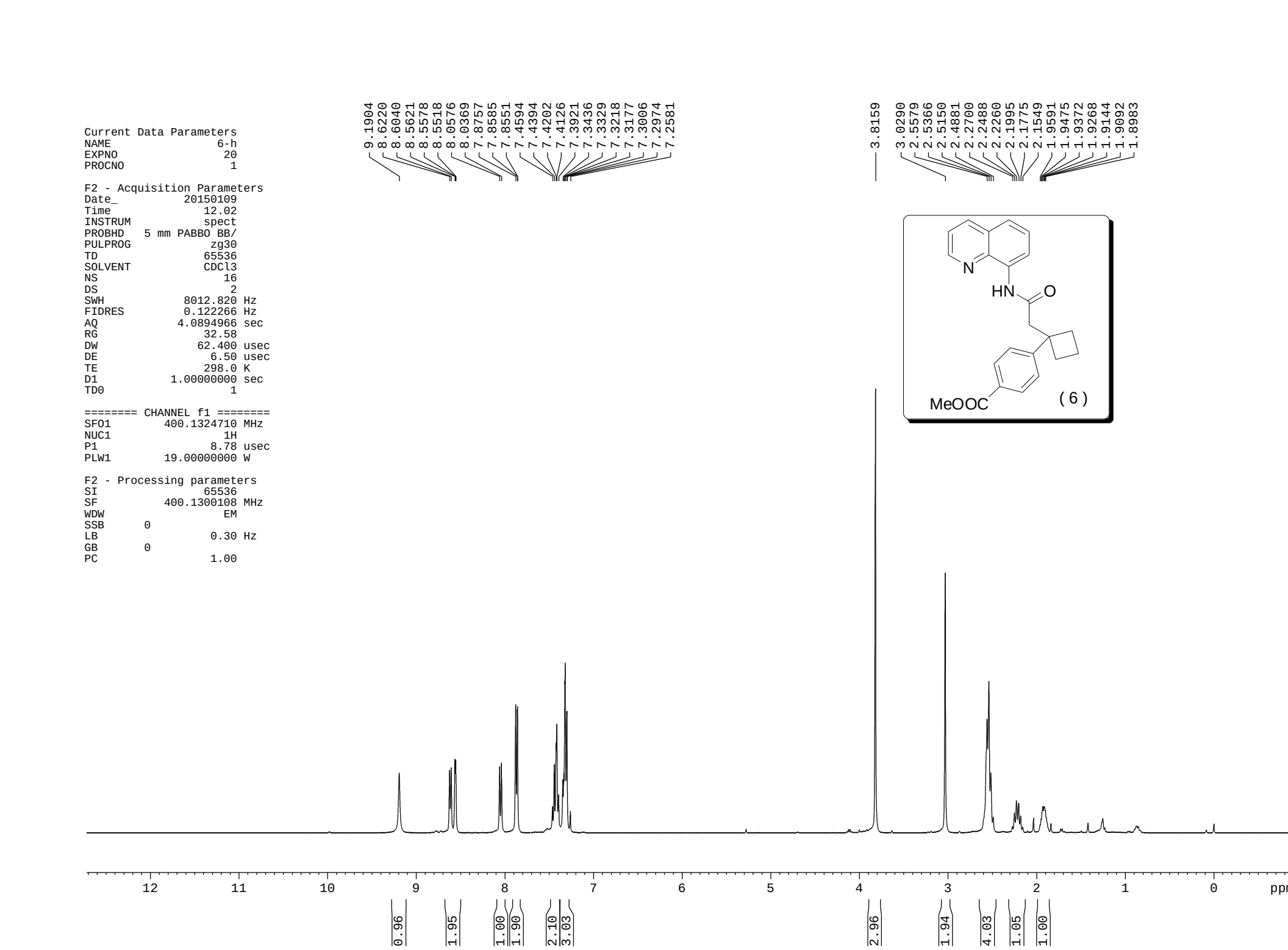



-117.78
N
HN
O
F
(4)
F19
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm


9.169
8.640
8.637
8.623
8.619
8.578
8.571
8.093
8.090
8.072
8.070
7.486
7.465
7.457
7.446
7.440
7.425
7.386
7.376
7.363
7.342
7.260
3.045
2.607
2.597
2.577
2.567
2.555
2.532
2.509
2.506
2.481
2.299
2.277
2.255
2.249
2.227
2.206
2.184
1.977
1.967
1.955
1.945
1.933
1.927
1.916
1.905
1.894
1.883
N
HN
O
F3C
(5)
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.96
1.92
0.97
3.86
3.09
2.00
3.97
1.03
0.99

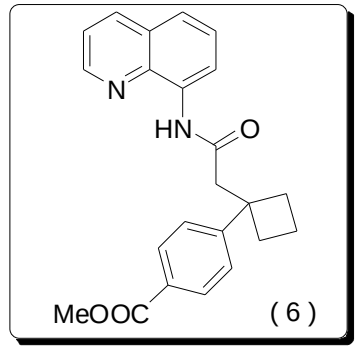

169.26
153.03
148.04
138.18
136.25
134.21
128.33
128.01
127.87
127.34
126.33
125.72
125.42
125.39
125.35
125.31
123.02
121.62
121.53
116.28
50.60
45.82
33.14
16.12
N
HN
O
F3C
(5)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

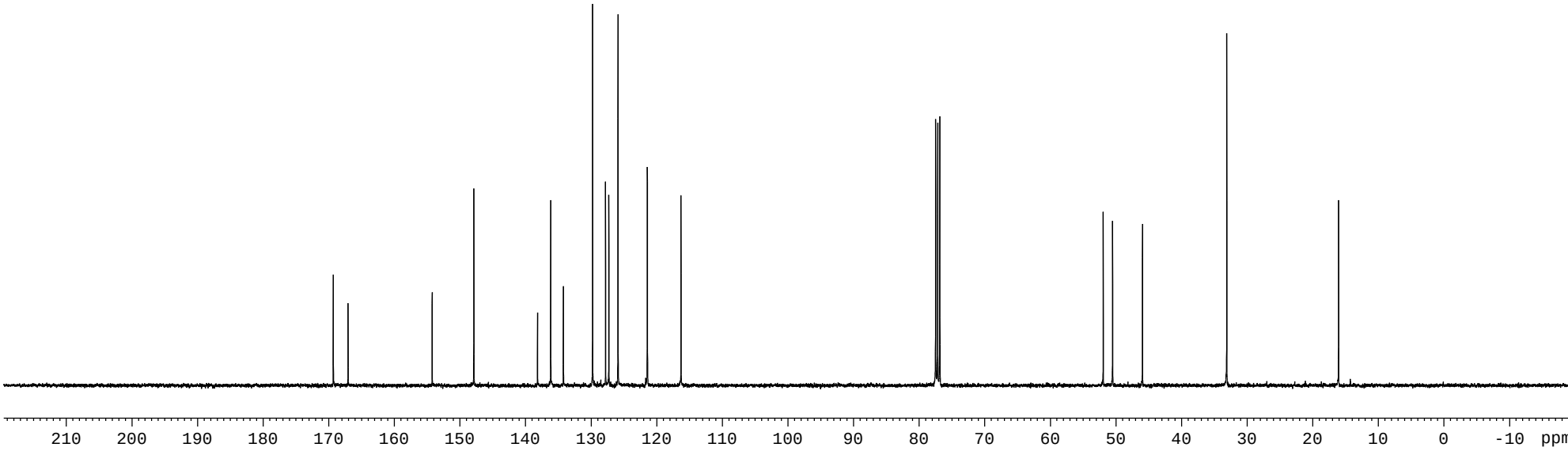

-62.30
N
HN
O
F3C
(5)
F19
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
ppm

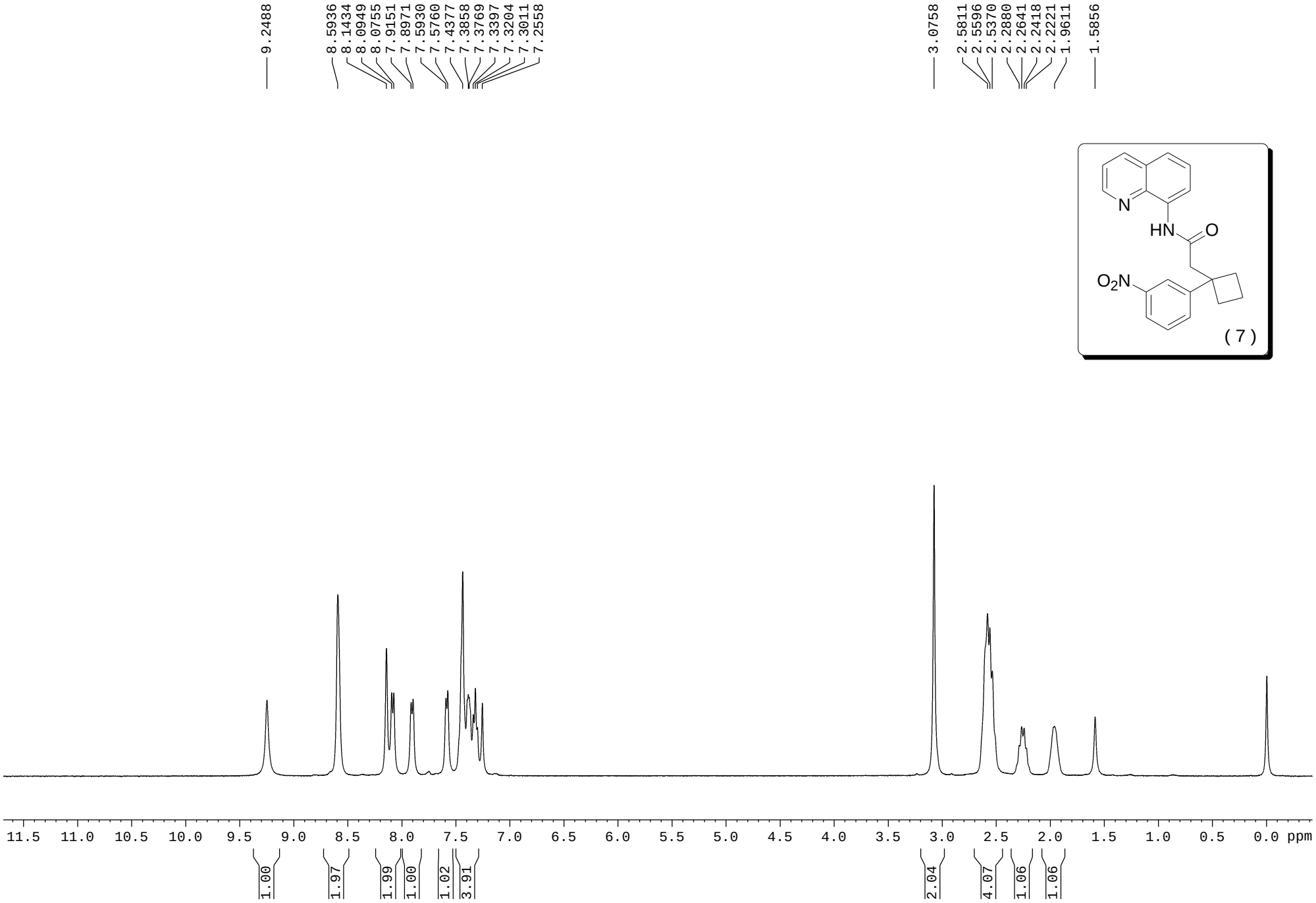

9.1904
8.6220
8.6040
8.5621
8.5578
8.5518
8.0576
8.0369
7.8757
7.8585
7.8551
7.4594
7.4394
7.4202
7.4126
7.3921
7.3436
7.3329
7.3218
7.3177
7.3006
7.2974
7.2581
3.8159
3.0290
2.5579
2.5366
2.5150
2.4881
2.2700
2.2488
2.2260
2.1995
2.1775
2.1549
1.9591
1.9475
1.9372
1.9268
1.9144
1.9092
1.8983
Current Data Parameters
NAME 6-h
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150109
Time 12.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 32.58
DW 62.400 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 8.78 usec
PLW1 19.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300108 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
HN
O
MeOOC
(6)
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96
1.95
1.00
1.90
2.10
3.03
2.96
1.94
4.03
1.05
1.00

```
Current Data Parameters
NAME                  6-c
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150110
Time                23.48
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
```

169.3142
167.0637
154.2400
147.8937
138.1802
136.1704
134.2372
129.7892
127.8063
127.3099
125.9120
121.4488
121.4293
116.3085
51.9459
50.5129
45.9567
33.1089
16.0620



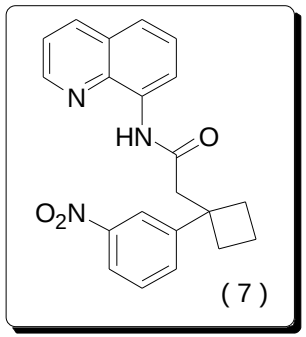



9.2488
8.5936
8.1434
8.0949
8.0755
7.9151
7.8971
7.5930
7.5760
7.4377
7.3858
7.3769
7.3397
7.3204
7.3011
7.2558
3.0758
2.5811
2.5596
2.5370
2.2880
2.2641
2.2418
2.2221
1.9611
1.5856
N
HN
O
O2N
(7)
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.97
1.99
1.00
1.02
3.91
2.04
4.07
1.06
1.06

Current Data Parameters
NAME YXL-3-313-C-20160
EXPNO 22
PROCNO 1

F2 - Acquisition Parameter
Date_ 20160508
Time 11.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 210
DS 4
SWH 24038.461 Hz

168.9136
151.0367
148.5122
148.0617
138.2059
136.3482
134.0755
132.4881
129.1982
127.8991
127.3848
121.6733
121.6317
121.1314
121.0785
116.4393
50.2625
45.7152
33.2751
16.1603



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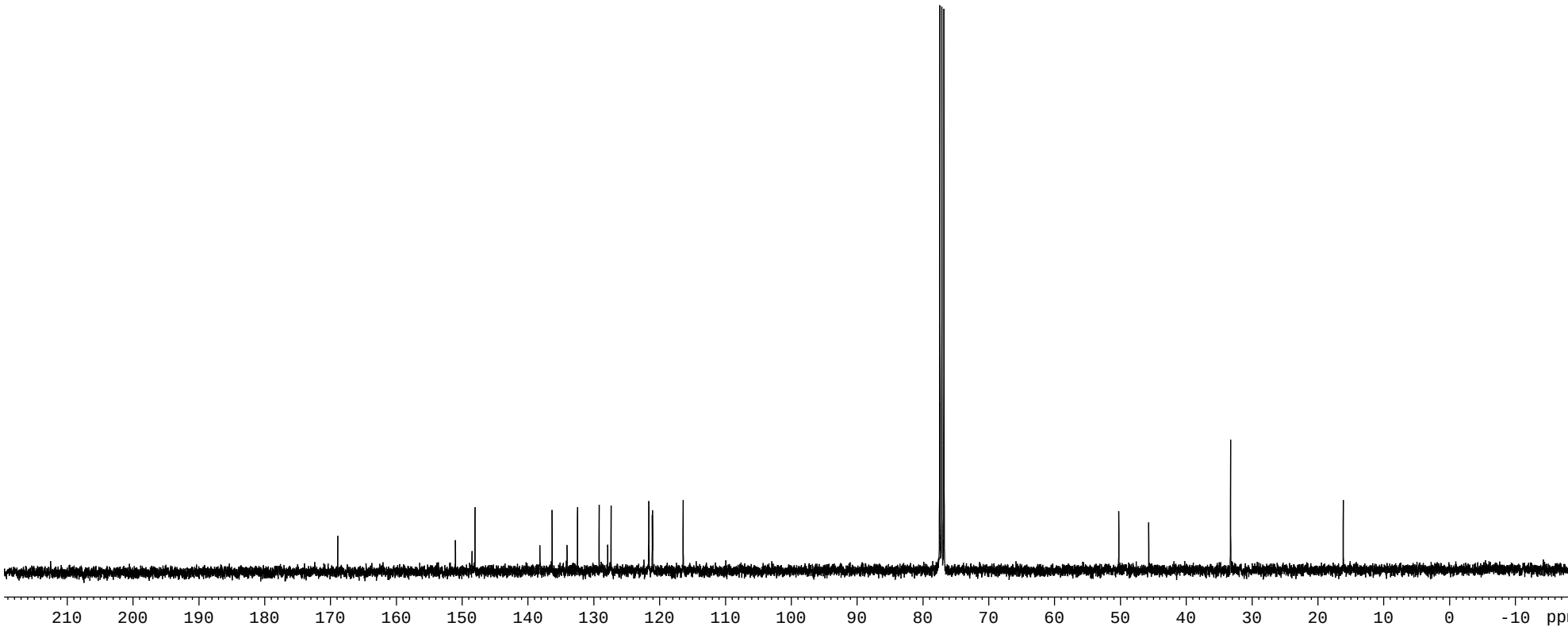
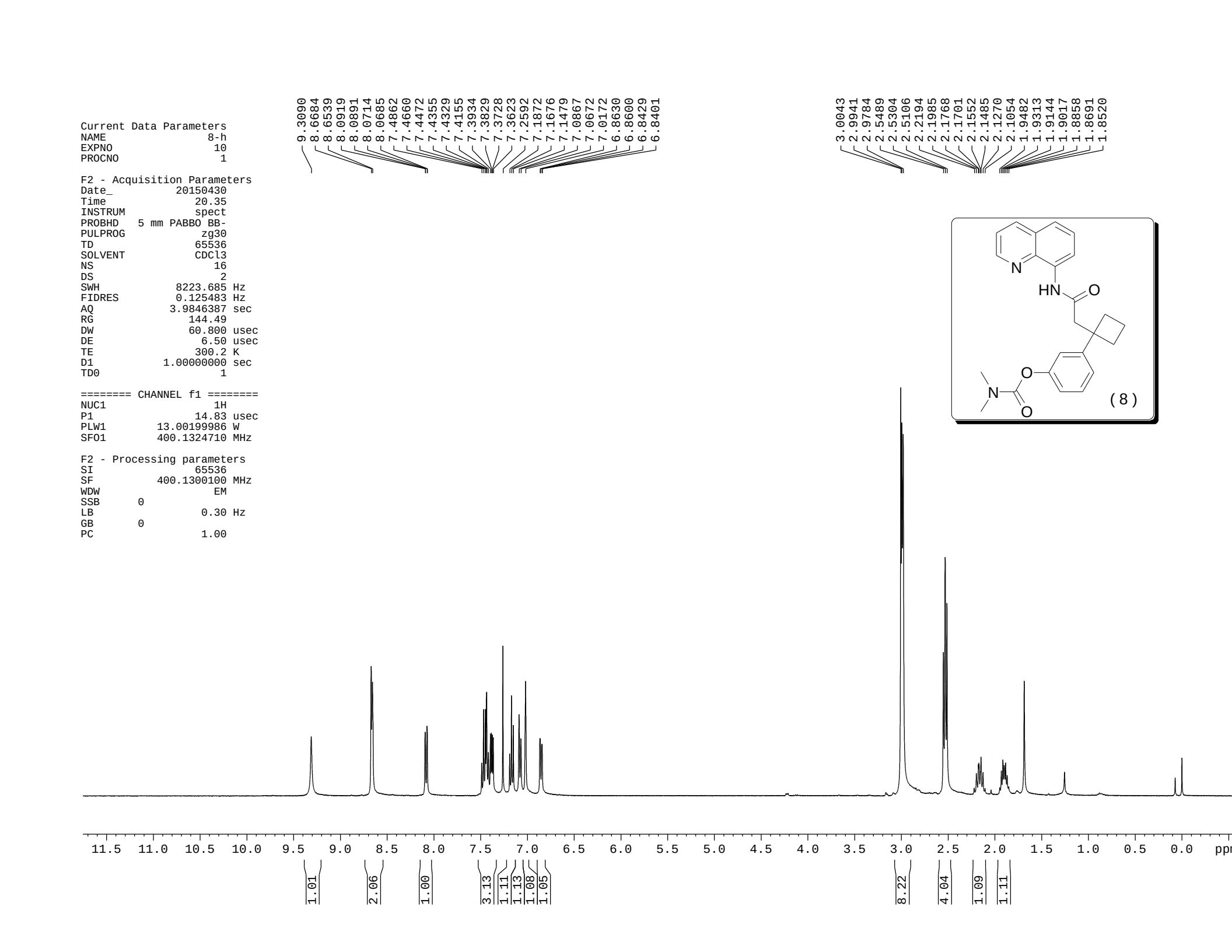
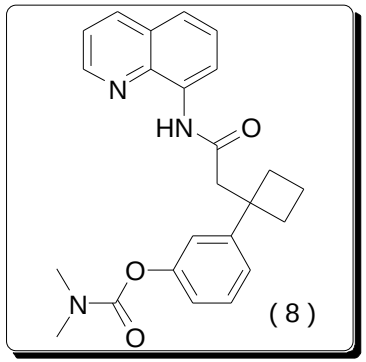



9.3090
8.6684
8.6539
8.0919
8.0891
8.0714
8.0685
7.4862
7.4660
7.4472
7.4355
7.4329
7.4155
7.3934
7.3829
7.3728
7.3623
7.2592
7.1872
7.1676
7.1479
7.0867
7.0672
7.0172
6.8630
6.8600
6.8429
6.8401
3.0043
2.9941
2.9784
2.5489
2.5304
2.5106
2.2194
2.1985
2.1768
2.1701
2.1552
2.1485
2.1270
2.1054
1.9482
1.9313
1.9144
1.9017
1.8858
1.8691
1.8520
Current Data Parameters
NAME 8-h
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150430
Time 20.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 144.49
DW 60.800 usec
DE 6.50 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
HN
O
O
N
O
O
(8)
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.01
2.06
1.00
3.13
1.11
1.13
1.08
1.05
8.22
4.04
1.09
1.11

```
Current Data Parameters
NAME                  8-c
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150501
Time               10.32
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
```

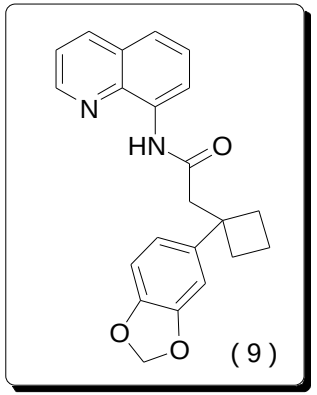
169.7552
154.9722
151.7303
150.4734
148.0345
138.4038
136.1493
134.4956
129.1087
127.8877
127.3811
122.7559
121.5331
121.3259
119.3118
119.2529
116.4270
50.7984
45.6733
36.7780
36.5110
33.0760
16.0658

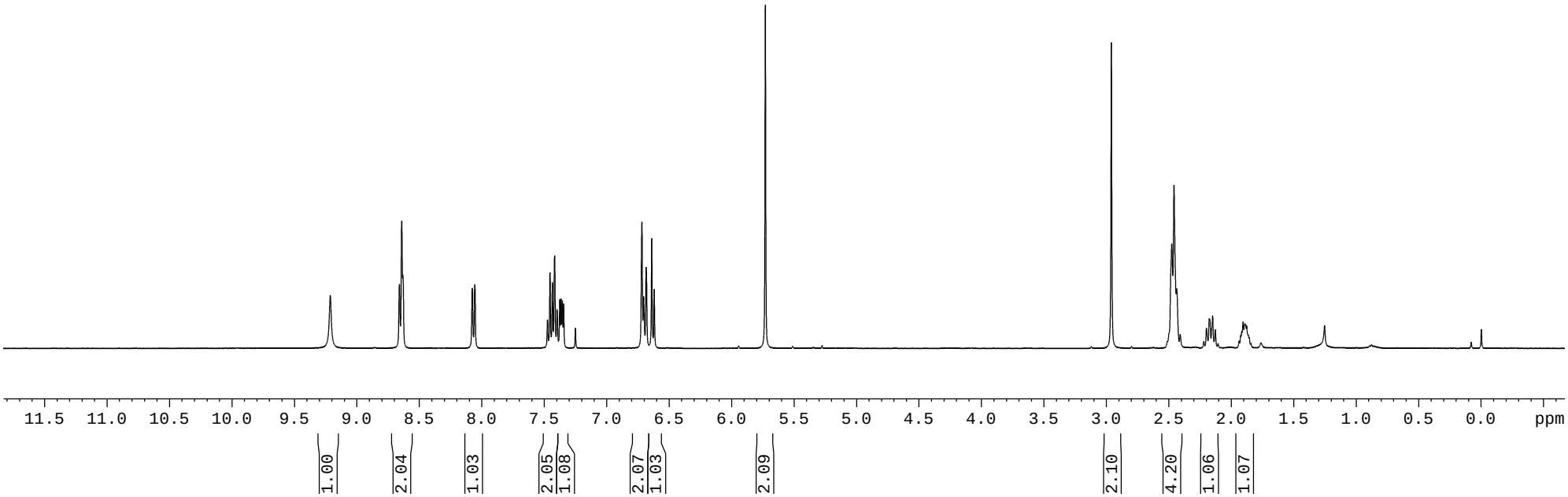


```
Current Data Parameters
NAME      YXL-4-61-H-160
EXPNO                 20
PROCNO                 1

F2 - Acquisition Parame
Date_           20160507
Time               16.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820
FIDRES          0.122266
AQ              4.0894465
RG                 32.58
DW                 62.400
DE                   6.50
TE                  297.9
D1            1.00000000
TD0                    1

======== CHANNEL f1 ===
SFO1         400.1324710
NUC1                1H
P1                  9.15
PLW1         19.00000000

F2 - Processing paramet
SI                 65536
```

9.2136 | 8.6600 | 8.6412 | 8.6327 | 8.0768 | 8.0565 | 7.4745 | 7.4544 | 7.4354 | 7.4173 | 7.3979 | 7.3761 | 7.3657 | 7.3556 | 7.3451 | 6.7182 | 6.7051 | 6.6846 | 6.6397 | 6.6199

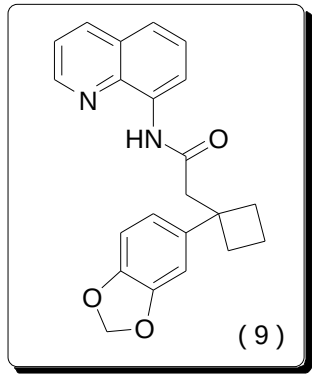
2.9598 | 2.4762 | 2.4582 | 2.4371 | 2.4088 | 2.1984 | 2.1761 | 2.1712 | 2.1489 | 2.1275 | 1.9244 | 1.9160 | 1.9049 | 1.8949 | 1.8890 | 1.8842 | 1.8761

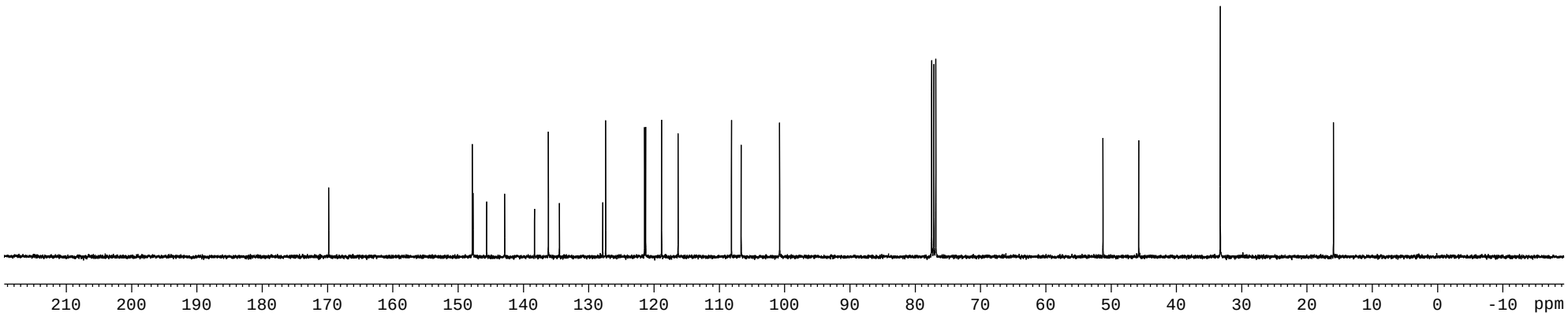


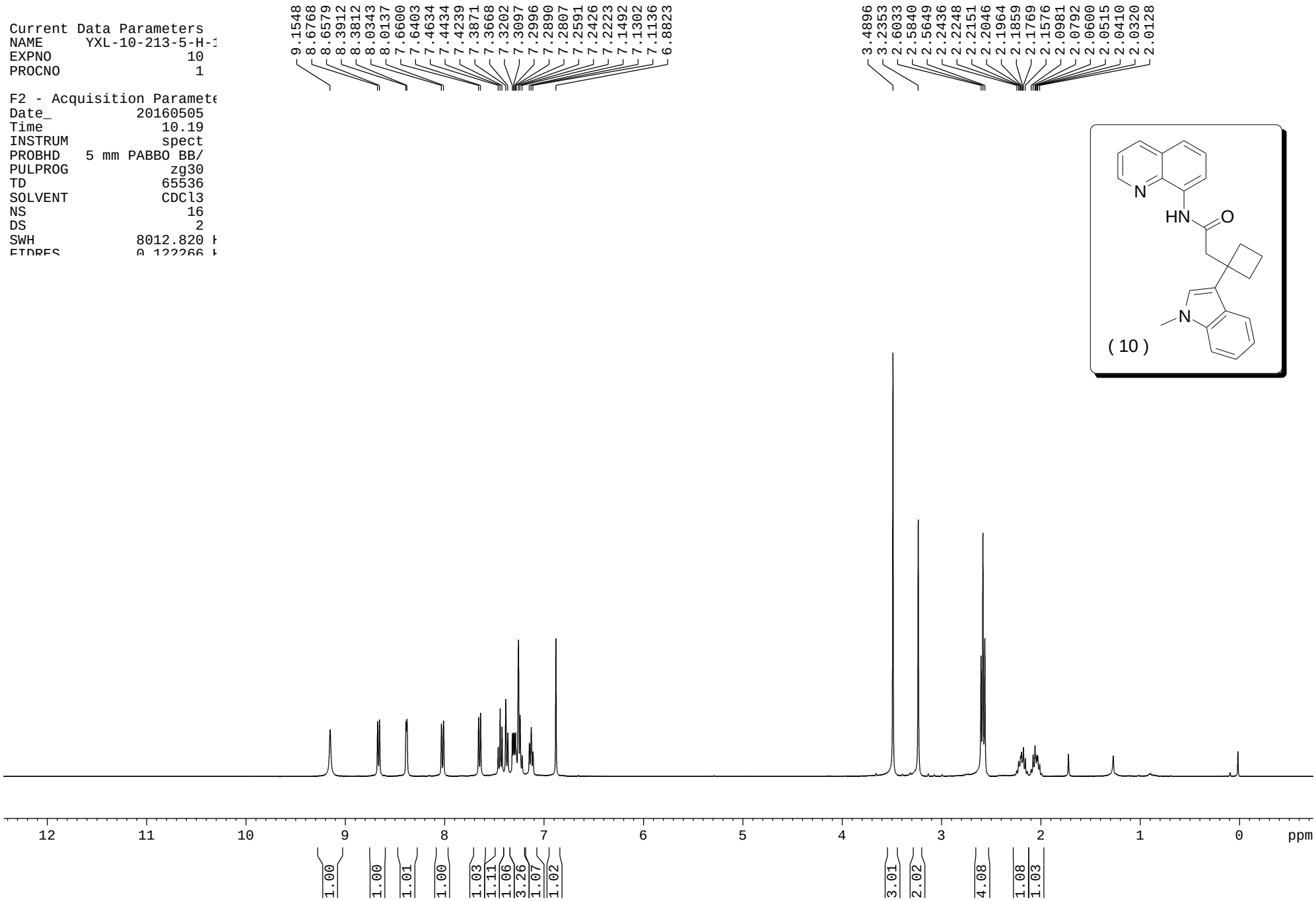


```
Current Data Parameters
NAME      YXL-4-61-C-16(
EXPNO                 1(
PROCNO                1

F2 - Acquisition Parame
Date_           2016050
Time               20.4(
INSTRUM            spec(
PROBHD   5 mm PABBO BB/
PULPROG            zgpg3(
TD                 6553(
SOLVENT            CDCl3
NS                   21(
DS                    4
SWH            24038.46
FIDRES          0.36679
AQ             1.363148
RG                 202.1
```

169.8134
147.8096
147.6896
145.6244
142.8649
138.2926
136.1702
134.4817
127.8387
127.3981
127.4399
121.2657
118.8234
116.3062
108.1271
106.6435
100.7489
51.2249
45.7504
33.2609
29.8011
15.9206

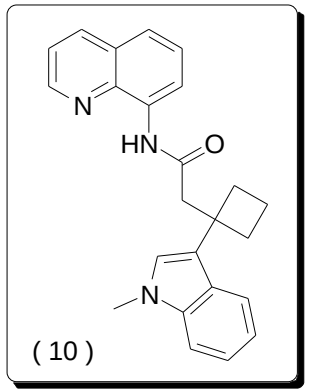




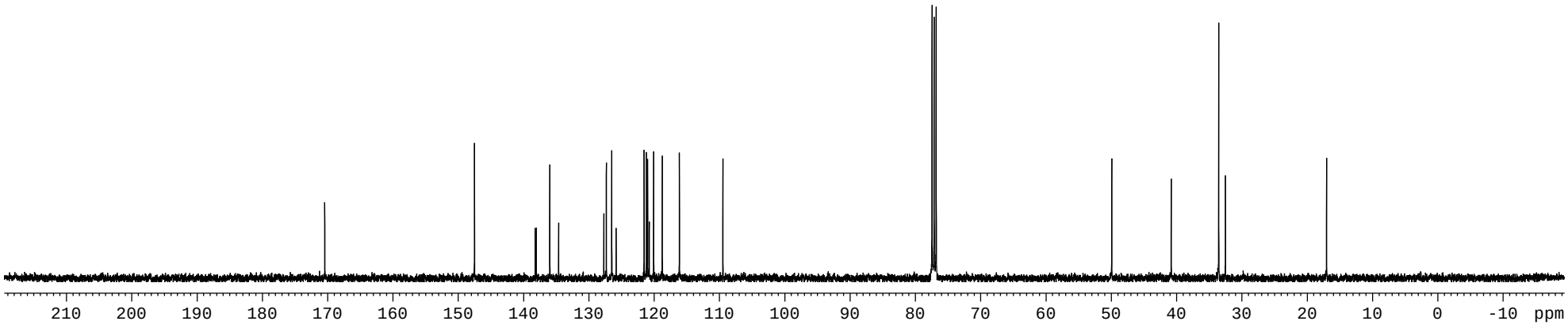

Current Data Parameters
NAME YXL-10-213-5-H-1
EXPNO 10
PROCNO 1
F2 - Acquisition Paramete
Date_ 20160505
Time 10.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 H
9.1548
8.6768
8.6579
8.3912
8.3812
8.0343
8.0137
7.6600
7.6403
7.4634
7.4434
7.4239
7.3871
7.3668
7.3202
7.3097
7.2996
7.2890
7.2807
7.2591
7.2426
7.2223
7.1492
7.1302
7.1136
6.8823
3.4896
3.2353
2.6033
2.5840
2.5649
2.2436
2.2248
2.2151
2.2046
2.1964
2.1859
2.1769
2.1576
2.0981
2.0792
2.0600
2.0515
2.0410
2.0320
2.0128
N
HN
O
N
(10)
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.00
1.01
1.00
1.03
1.11
1.06
3.26
1.07
1.02
3.01
2.02
4.08
1.08
1.03

```
Current Data Parameters
NAME      YXL-10-213-5-C-16
EXPNO                   11
PROCNO                   1

F2 - Acquisition Parameter
Date_           20160505
Time               10.25
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
```

170.4676
147.5442
138.2566
138.0785
136.0118
134.6510
127.7280
127.3500
126.5168
125.8578
121.5735
121.2221
121.0206
120.7799
120.1058
118.7856
116.1643
109.5012
49.9521
40.8466
33.5681
32.5451
17.0564

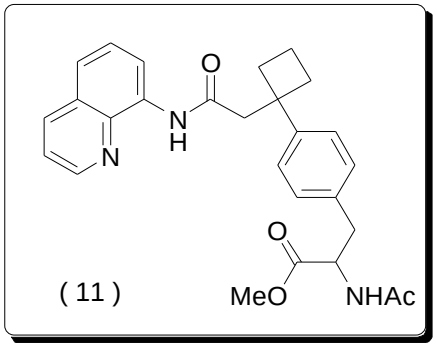


(10)



```
Current Data Parameters
NAME      YXL-10-213-6-H-
EXPNO                 10
PROCNO                 1

F2 - Acquisition Paramet
Date_           20160505
Time               16.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820
```

9.2561 8.6330 8.6186 8.0792 8.0587 7.4374 7.4179 7.3951 7.3837 7.3731 7.3630 7.3526 7.1812 7.1615 6.9199 6.9003 5.8519 5.8328 4.7620 4.7474 4.7290 4.7144 3.5796 2.9750 2.9673 2.9421 2.9283 2.5217 2.5026



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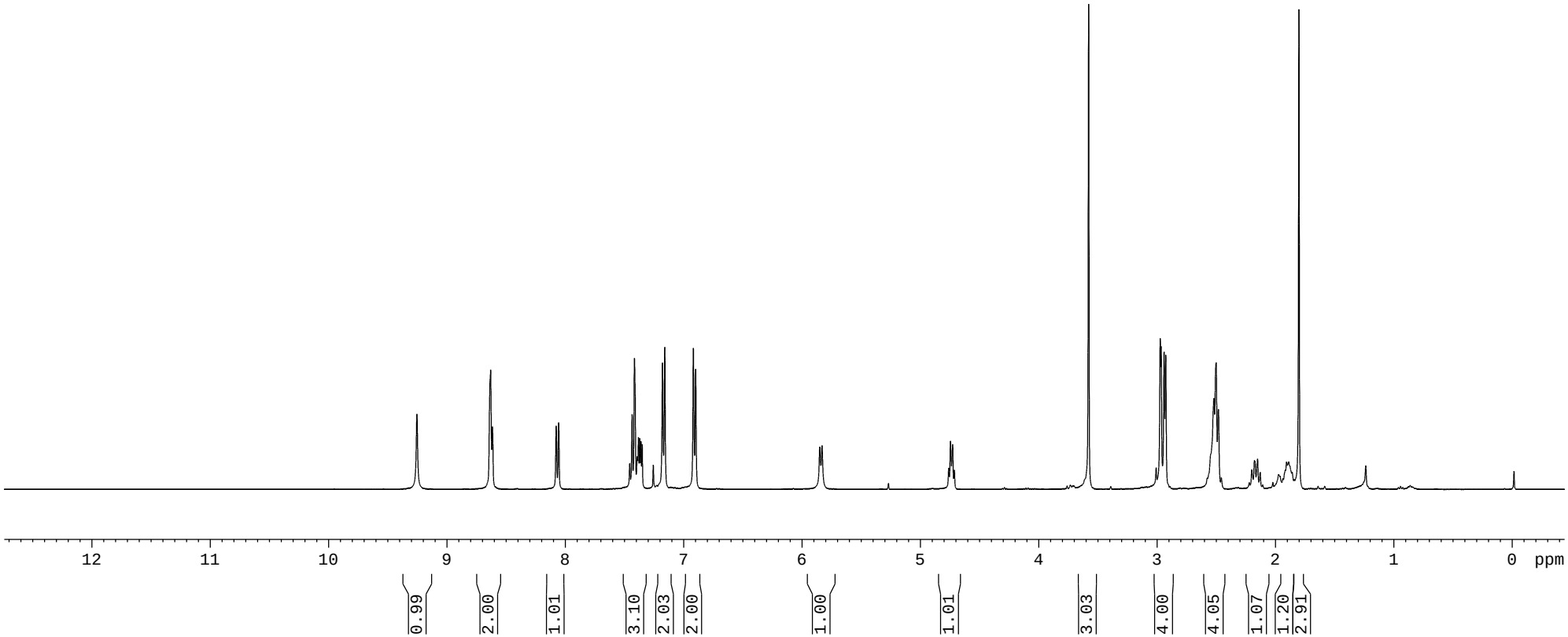
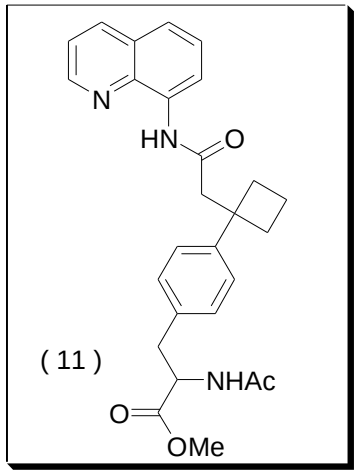
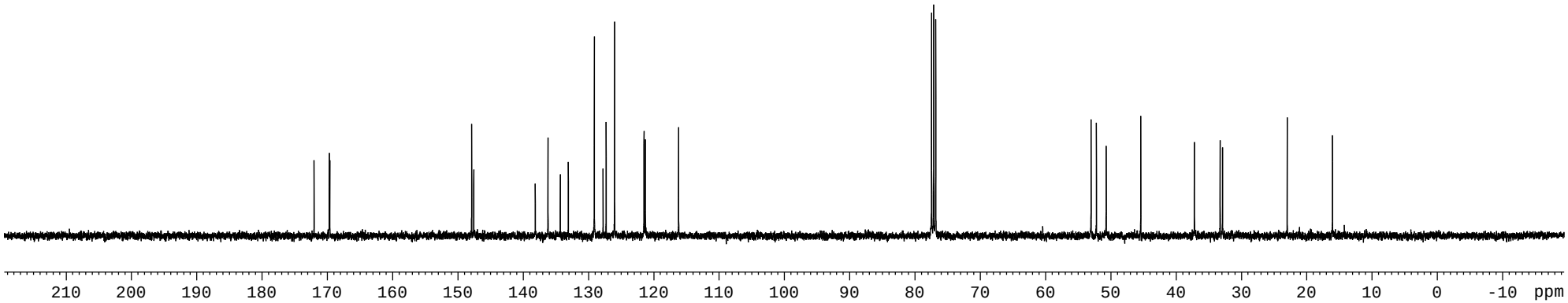


Current Data Pa
NAME YXL-10
EXPNO
PROCNO

F2 - Acquisitio
Date_
Time

172.06
169.73
169.64
147.92
147.60
138.17
136.22
134.34
133.11
129.13
127.80
127.32
126.03
121.52
121.31
116.22
77.47
77.15
76.84
53.05
52.23
50.73
45.44
37.22
33.28
32.91
23.00
16.07

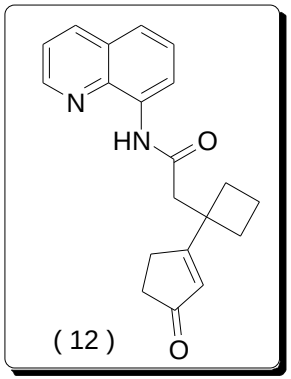


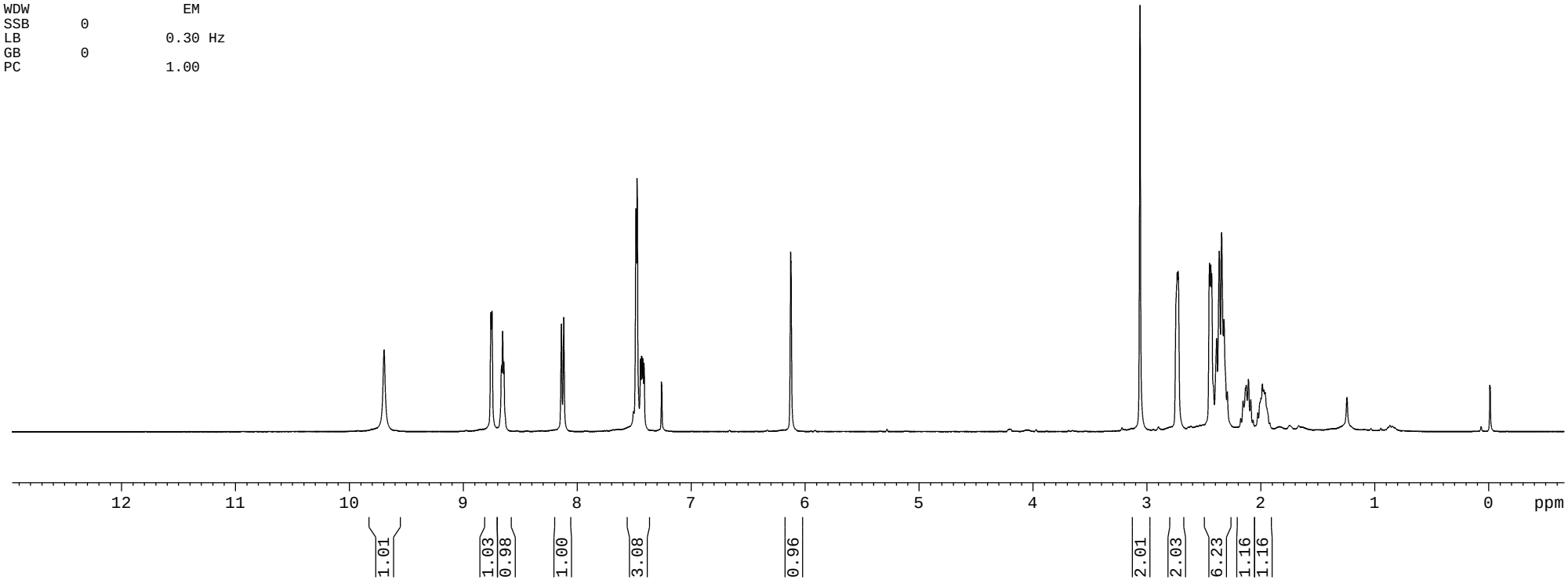


```
Current Data Parameters
NAME                 19-h
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150106
Time               14.45
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                 32.58
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  8.78 usec
PLW1         19.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300109 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

9.6968 8.7593 8.7533 8.7490 8.6653 8.6560 8.6463 8.1401 8.1194 7.4858 7.4762 7.4741 7.4467 7.4441 7.4363 7.4336 7.4262 7.4237 7.4156 7.2620 7.2594 6.1276 6.1242

3.0622 3.0605 2.7346 2.7269 2.7231 2.4508 2.4433 2.4386 2.4319 2.3882 2.3660 2.3442 2.3244 2.2945 2.1760 2.1567 2.1337 2.1283 2.1099 2.0884 2.0700 2.0273 2.0094 1.9992 1.9869 1.9752 1.9616 1.9231

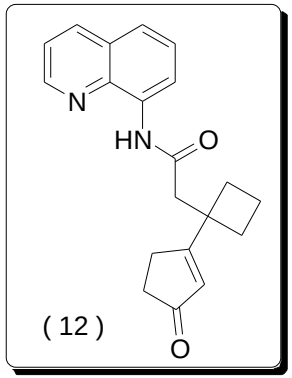




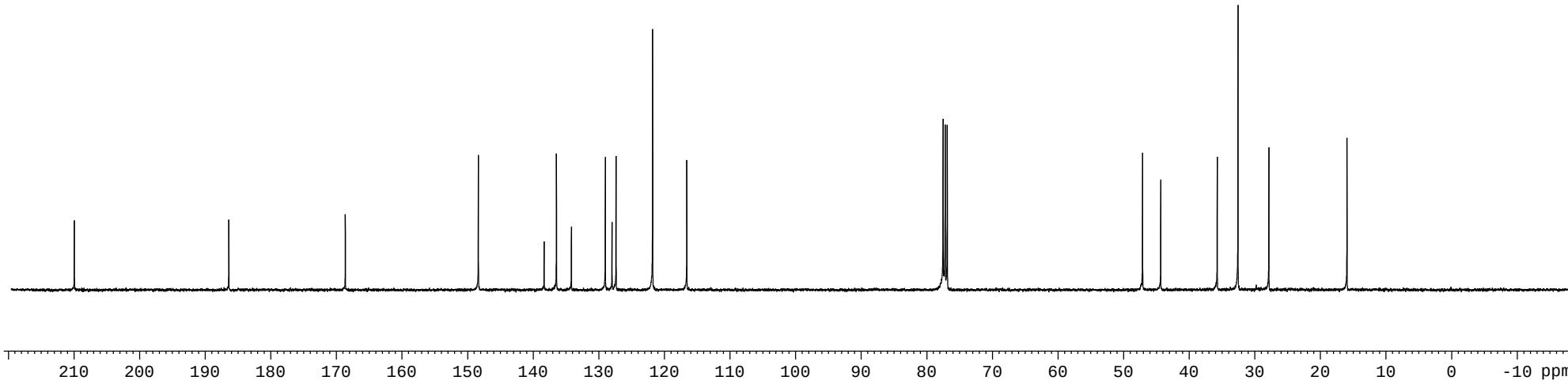
209.9146
186.3703
168.6084
148.3268
138.3021
136.4265
134.1442
128.9709
127.9524
127.3378
121.7648
116.5544
47.0707
44.3075
35.6921
32.5314
27.8227
15.9153

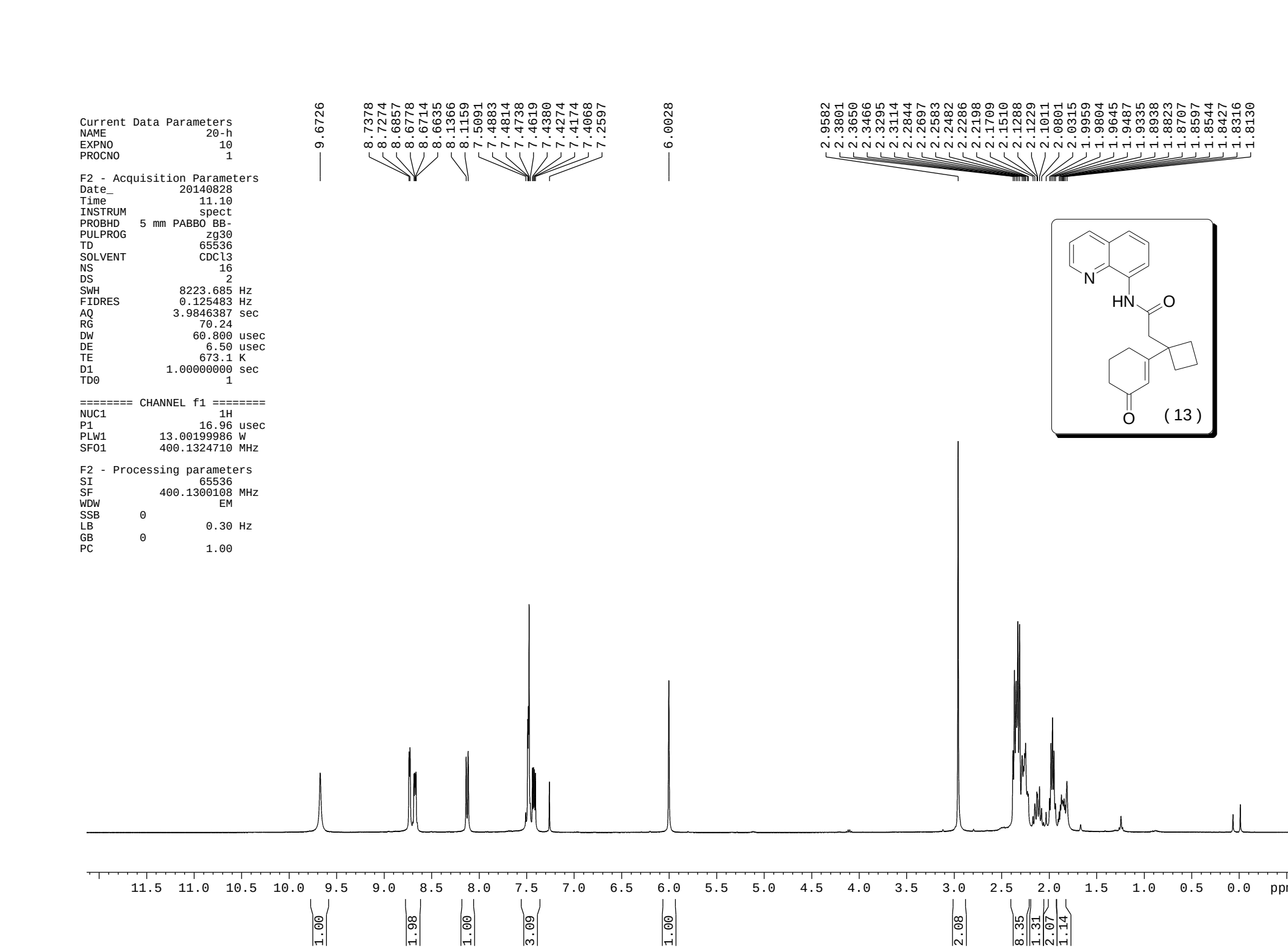
Current Data Parameters
NAME 19-c
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141209
Time 0.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3



(12)

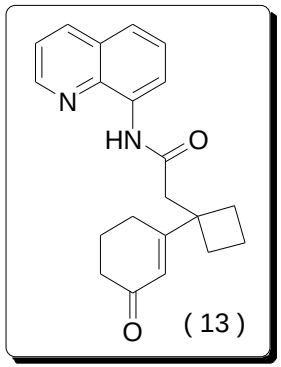



Current Data Parameters
NAME 20-h
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140828
Time 11.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 70.24
DW 60.800 usec
DE 6.50 usec
TE 673.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 16.96 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300108 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.6726
8.7378
8.7274
8.6857
8.6778
8.6714
8.6635
8.1366
8.1159
7.5091
7.4883
7.4814
7.4738
7.4619
7.4380
7.4274
7.4174
7.4068
7.2597
6.0028
2.9582
2.3801
2.3650
2.3466
2.3295
2.3114
2.2844
2.2697
2.2583
2.2482
2.2286
2.2198
2.1709
2.1510
2.1288
2.1229
2.1011
2.0801
2.0315
1.9959
1.9804
1.9645
1.9487
1.9335
1.8938
1.8823
1.8707
1.8597
1.8544
1.8427
1.8316
1.8130
N
HN
O
O
(13)
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.98
1.00
3.09
1.00
2.08
8.35
1.31
2.07
1.14

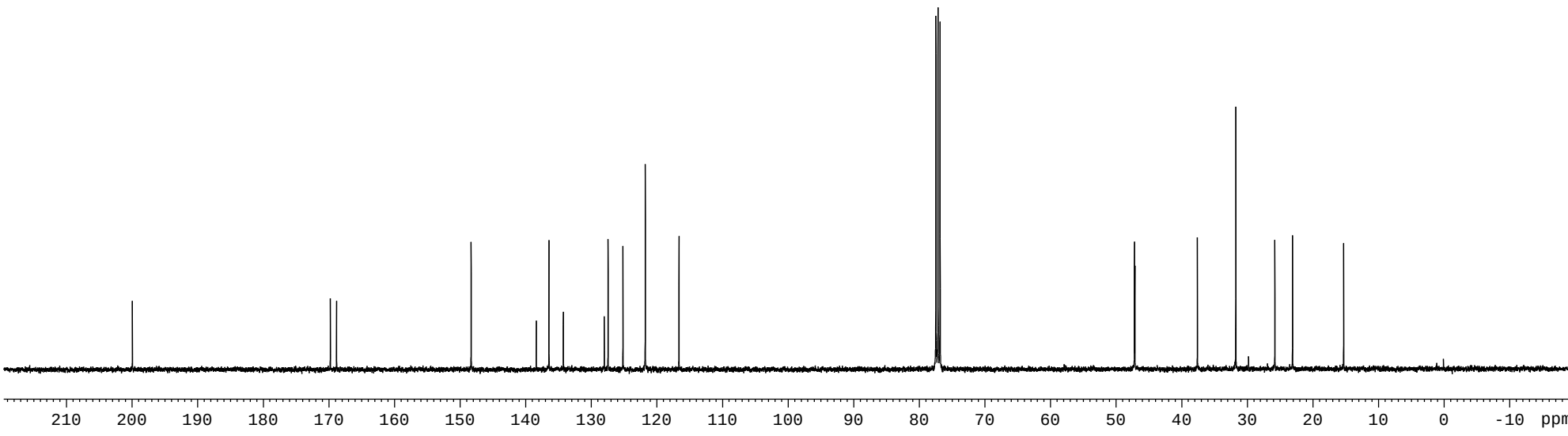
199.9660
169.7937
168.8396
148.3267
138.3984
136.4653
134.2719
128.0340
127.4518
125.1881
121.7826
116.6405
77.4771
77.1589
76.8417
47.2138
47.1406
37.6181
31.7621
25.8060
23.1153
15.3230

Current Data Parameters
NAME 20-c
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150103
Time 11.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3



(13)

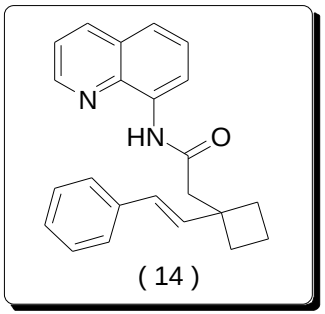


```
Current Data Parameters
NAME     YXL-10-213-1-H-201
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160519
Time               17.53
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    31
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 209.25
DW                 60.800 use
DE                  6.50 use
TE                 299.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                   1H
P1                  14.80 use
PLW1         13.00199986 W
SFO1         400.1324710 MHz
```

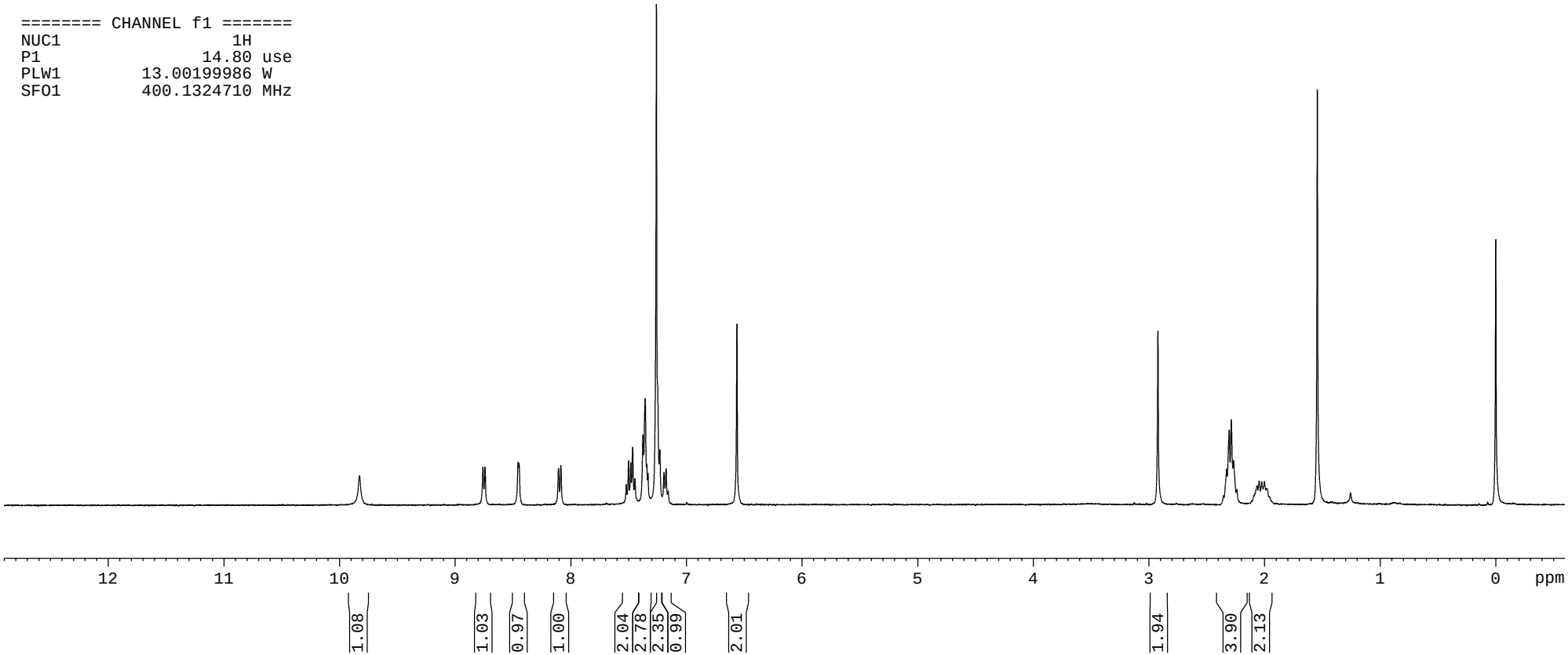
9.8270

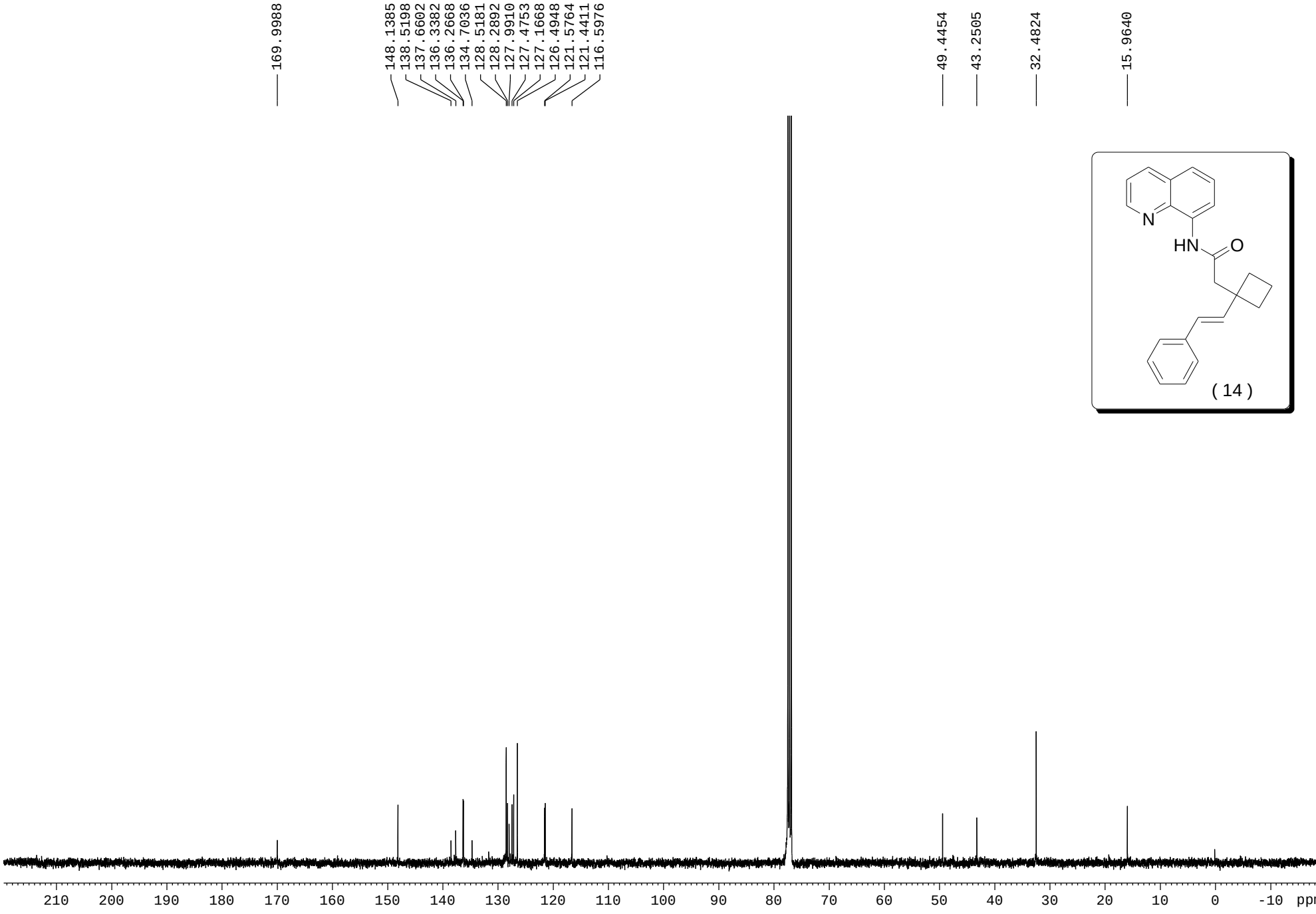
8.7600 8.7420 8.4555 8.4476 8.1067 8.0861 7.5203 7.5002 7.4812 7.4655 7.4457 7.3755 7.3567 7.3425 7.3321 7.2595 7.2298 7.1936 7.1756 7.1572 6.5630

2.9216 2.3565 2.3277 2.3056 2.2875 2.2666 2.2394 2.0930 2.0638 2.0461 2.0242 2.0011 1.9784



(14)

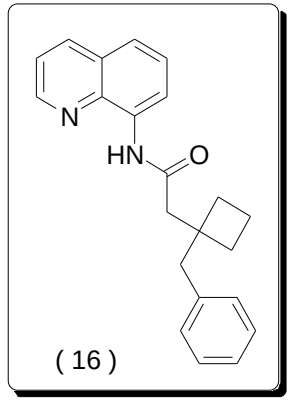


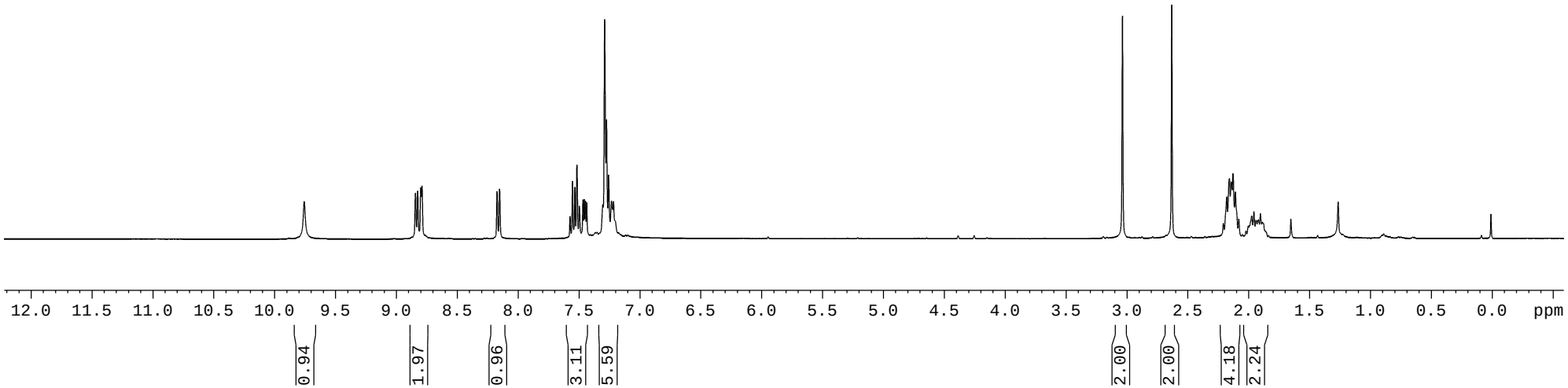

169.9988
148.1385
138.5198
137.6602
136.3382
136.2668
134.7036
128.5181
128.2892
127.9910
127.4753
127.1668
126.4948
121.5764
121.4411
116.5976
49.4454
43.2505
32.4824
15.9640
N
HN
O
(14)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm

Current Data Parameters
NAME YXL-10-213-10-H-2
EXPNO 31
PROCNO 1

F2 - Acquisition Parameter
Date_ 20160505
Time 20.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz

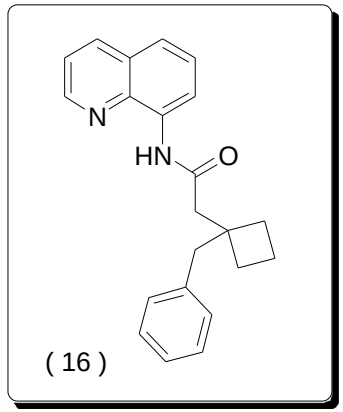
9.7593
8.8456 8.8273 8.8020 8.7921 8.1751 8.1546 7.5550 7.5359 7.5193 7.4691 7.4586 7.4485 7.4380 7.3067 7.2913 7.2761 7.2592 7.2340 7.2182
3.0376 2.6327 2.2088 2.1808 2.1597 2.1423 2.1300 2.1099 2.0822 1.9946 1.9768 1.9575 1.9424 1.9276 1.9199 1.9046 1.8898 1.8828

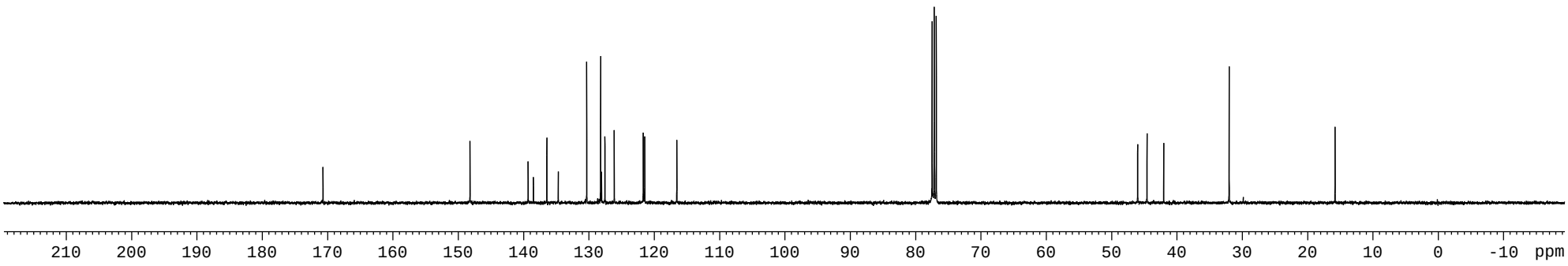


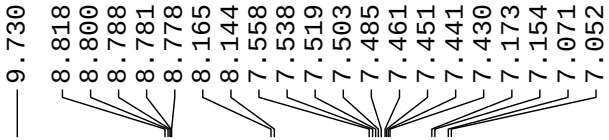


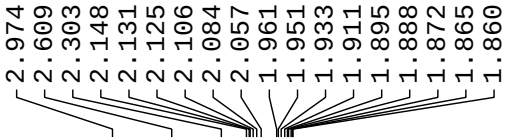
```
Current Data Parameters
NAME     YXL-10-213-10-C-16(
EXPNO                  21
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160506
Time                6.44
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   330
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                 202.1
DW                20.800 usec
DE                 6.50 usec
TE                295.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                10.10 usec
PLW1         65.00000000 W
```

170.7197
148.2199
139.3209
138.5036
136.4494
134.6991
130.3466
128.2162
128.0727
127.5580
126.1388
121.6980
121.4701
116.5551
46.0091
44.5777
42.0188
31.9989
15.7919

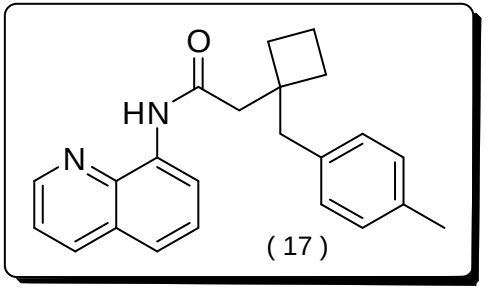


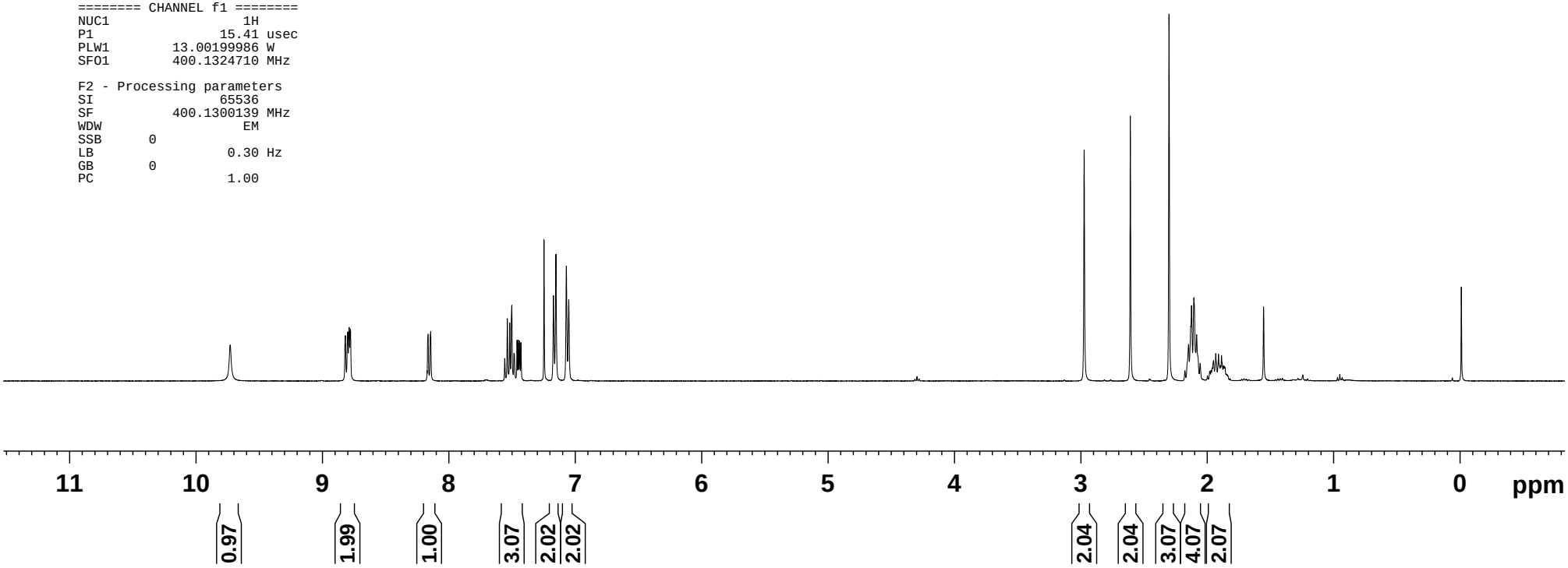







```
Current Data Parameters
NAME     YZM-Purify-17-H-20161209
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20161210
Time               5.23
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                184.16
DW                60.800 usec
DE                 6.50 usec
TE                299.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.41 usec
PLW1        13.00199986 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300139 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

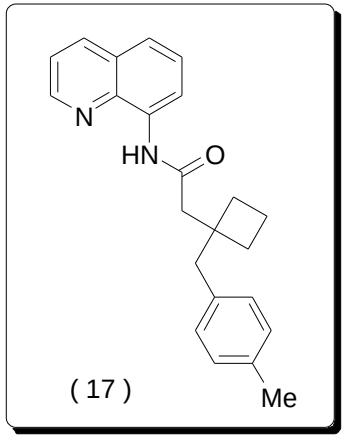




Current Data Parameters
NAME 13-c
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150115
Time 0.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 750

170.7898
148.2108
138.5415
136.4580
136.2007
135.5940
134.7574
130.2435
128.9344
128.8858
128.5929
128.0999
127.5831
121.6948
121.4513
116.5811
77.4842
77.1668
76.8493
46.1027
44.1996
42.0498
31.9927
21.1665
15.7989



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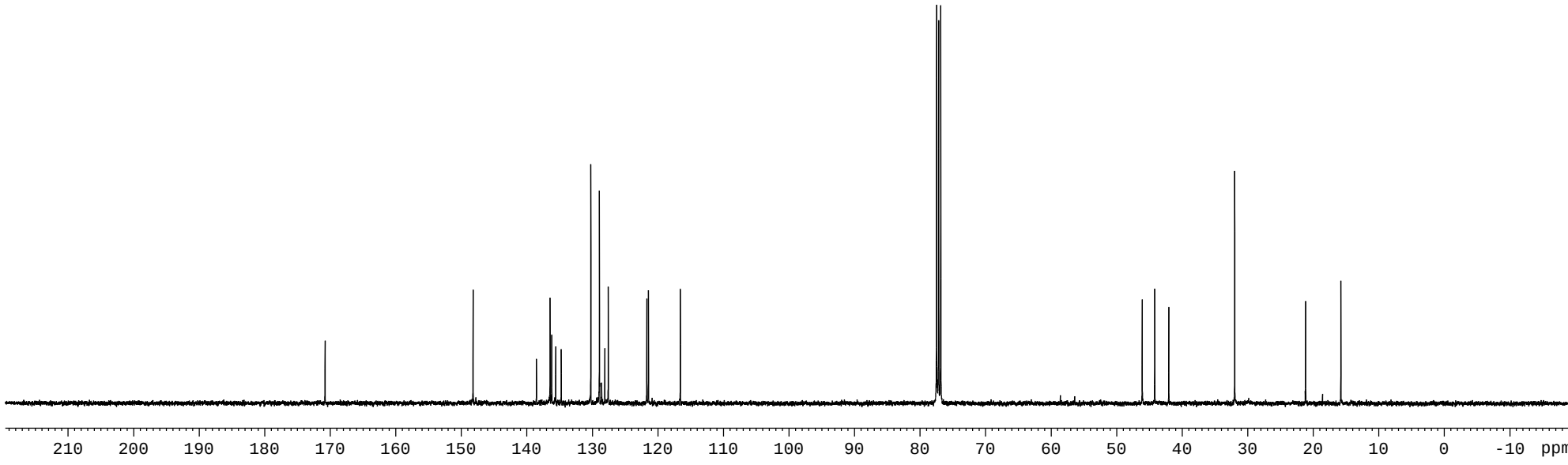
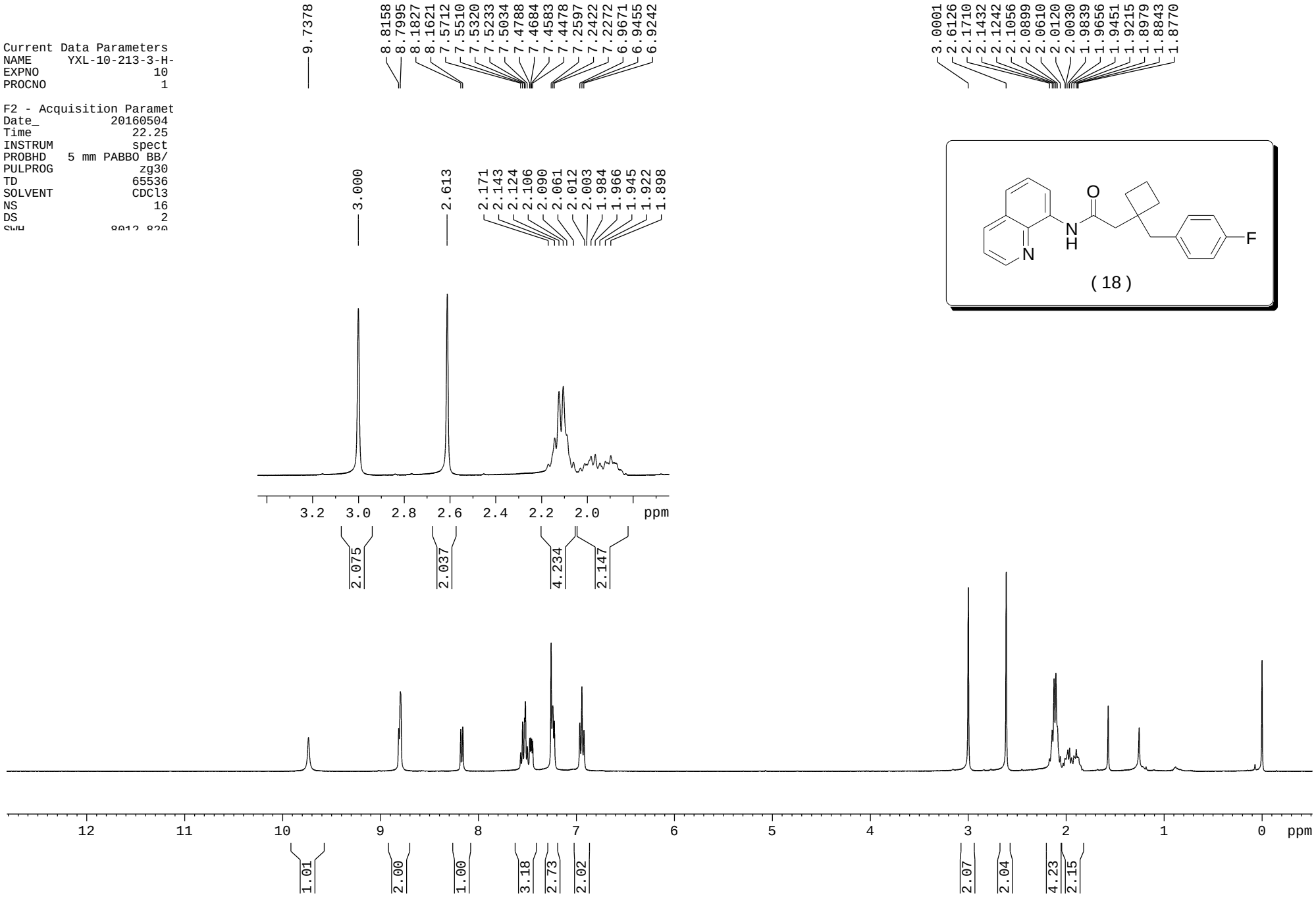
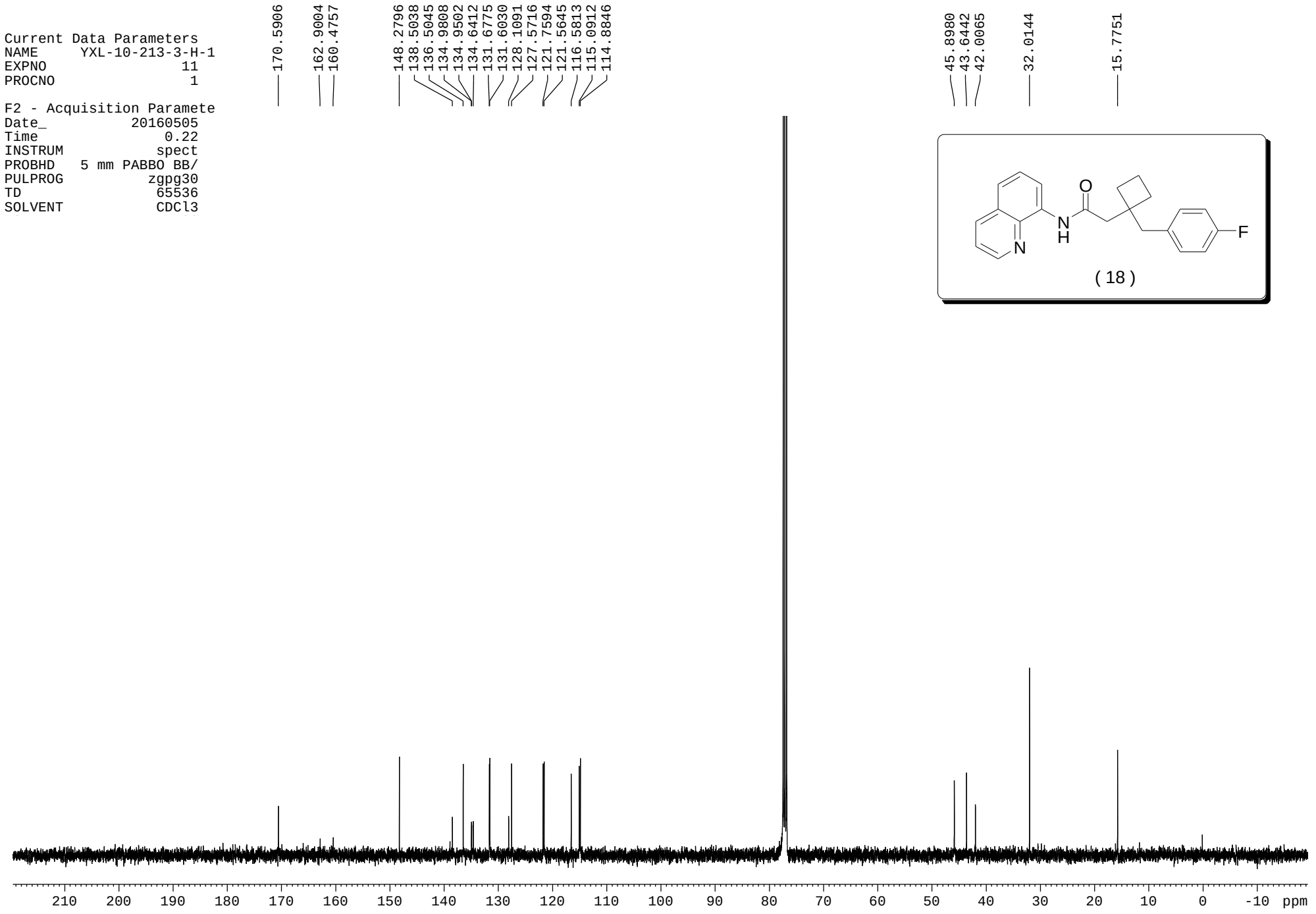
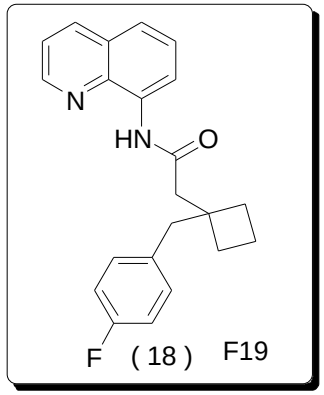


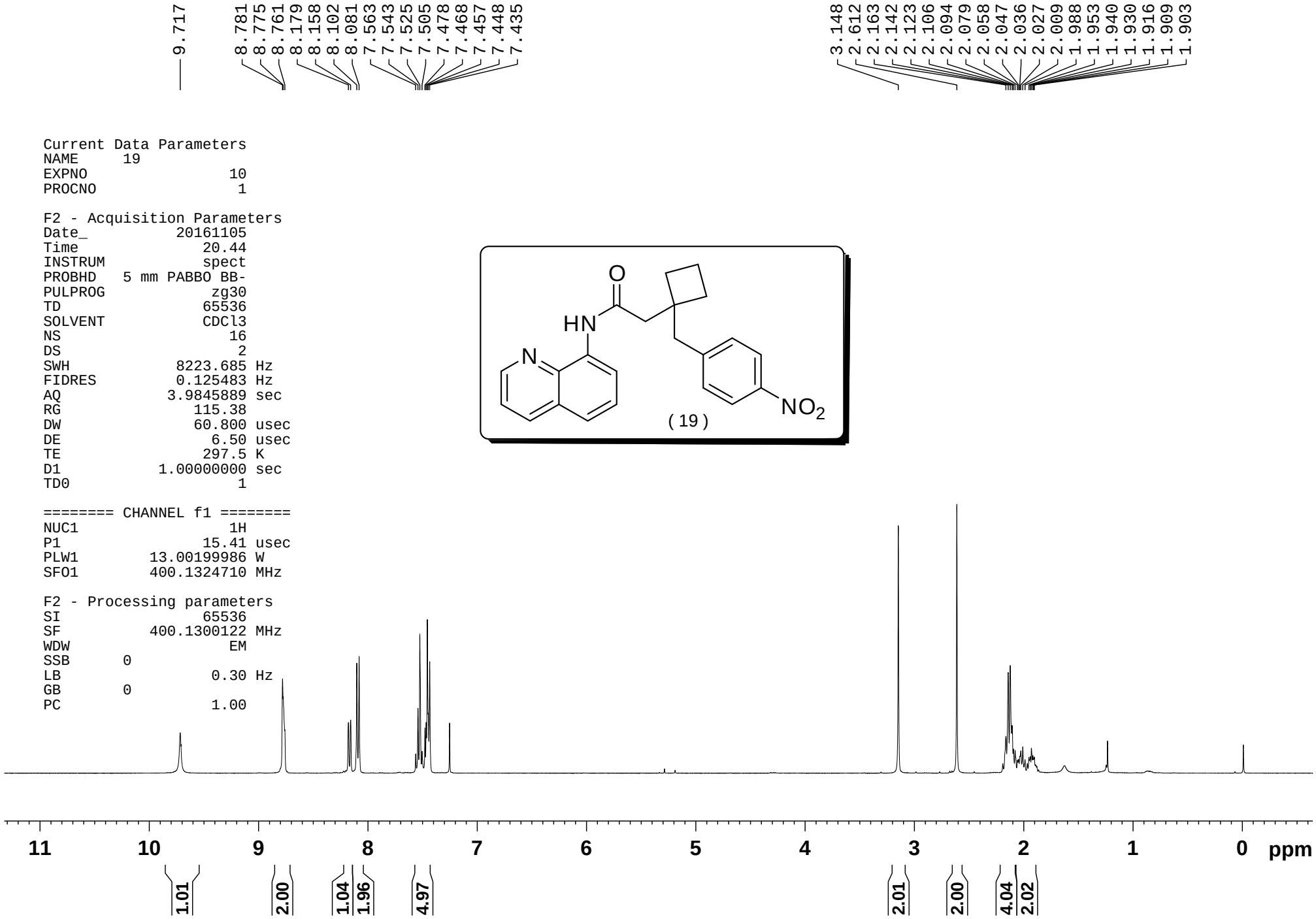

Current Data Parameters
NAME YXL-10-213-3-H-
EXPNO 10
PROCNO 1
F2 - Acquisition Paramet
Date_ 20160504
Time 22.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
9.7378
8.8158 8.7995 8.1827 8.1621 7.5712 7.5510 7.5320 7.5233 7.5034 7.4788 7.4684 7.4583 7.4478 7.2597 7.2422 7.2272 6.9671 6.9455 6.9242
3.0001 2.6126 2.1710 2.1432 2.1242 2.1056 2.0899 2.0610 2.0120 2.0030 1.9839 1.9656 1.9451 1.9215 1.8979 1.8843 1.8770
3.000 2.613
2.171 2.143 2.124 2.106 2.090 2.061 2.012 2.003 1.984 1.966 1.945 1.922 1.898
O
N
H
N
F
(18)
3.2 3.0 2.8 2.6 2.4 2.2 2.0 ppm
2.075 2.037 4.234 2.147
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.01 2.00 1.00 3.18 2.73 2.02 2.07 2.04 4.23 2.15


Current Data Parameters
NAME YXL-10-213-3-H-1
EXPNO 11
PROCNO 1
F2 - Acquisition Paramete
Date_ 20160505
Time 0.22
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
170.5906
162.9004
160.4757
148.2796
138.5038
136.5045
134.9808
134.9502
134.6412
131.6775
131.6030
128.1091
127.5716
121.7594
121.5645
116.5813
115.0912
114.8846
45.8980
43.6442
42.0065
32.0144
15.7751
O
N
H
N
F
(18)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm



-117.50

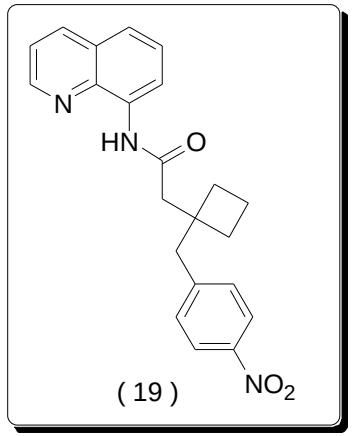
0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm


9.717
8.781
8.775
8.761
8.179
8.158
8.102
8.081
7.563
7.543
7.525
7.505
7.478
7.468
7.457
7.448
7.435
3.148
2.612
2.163
2.142
2.123
2.106
2.094
2.079
2.058
2.047
2.036
2.027
2.009
1.988
1.953
1.940
1.930
1.916
1.909
1.903
Current Data Parameters
NAME 19
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161105
Time 20.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 115.38
DW 60.800 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.41 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
HN
N
NO2
(19)
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
2.00
1.04
1.96
4.97
2.01
2.00
4.04
2.02

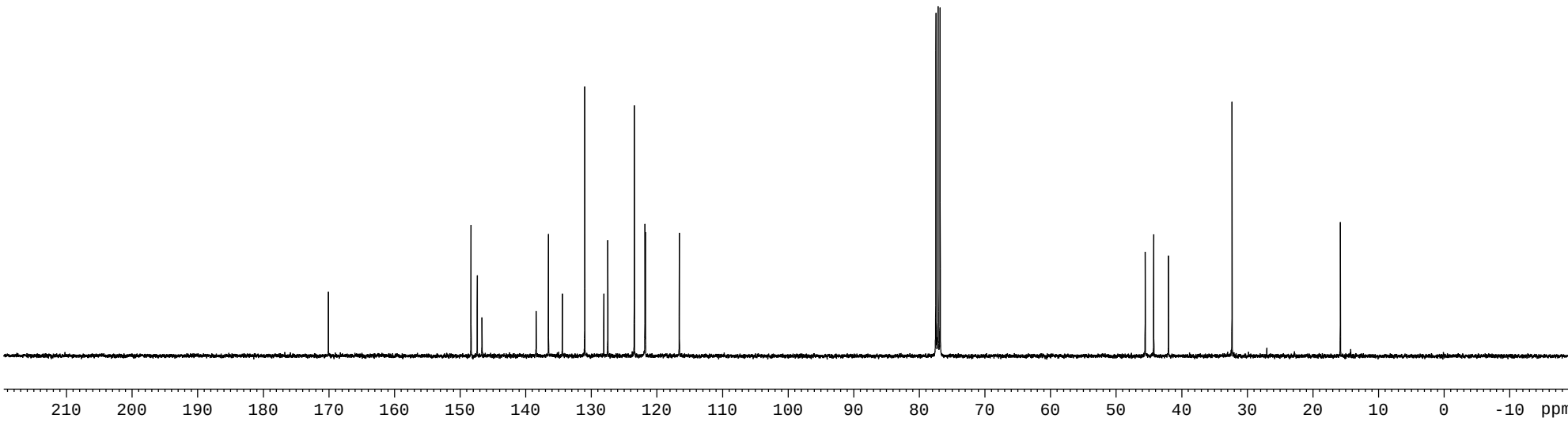
Current Data Parameters
NAME 15-c
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150114
Time 23.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 750

170.0908
148.3446
147.3972
146.6788
138.4237
136.5387
134.4252
131.0057
128.1030
127.4949
123.4507
121.8329
121.7595
116.5917
45.5791
44.2777
42.0186
32.3394
15.8249



(19)



```
Current Data Parameters
NAME     YXL-10-213-2-2-H
EXPNO                 12
PROCNO                 1

F2 - Acquisition Paramete
Date_           20160504
Time               22.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 H
FIDRES          0.122266 H
```

9.7627

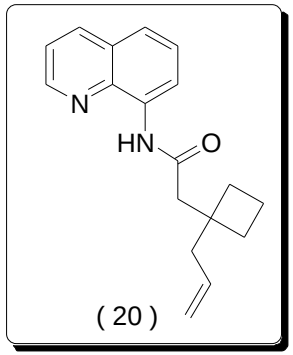
8.7961 8.7825 8.7792

8.1615 8.1410 7.5471 7.5266 7.5082 7.4973 7.4770 7.4609 7.4508 7.4407 7.4302 7.2596

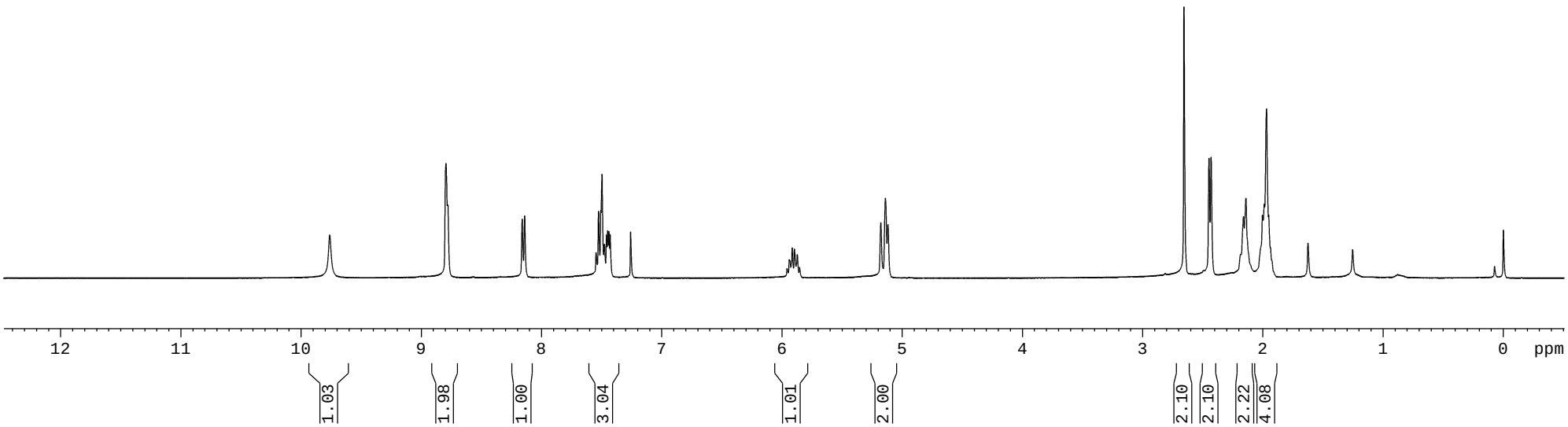
5.9399 5.9340 5.9153 5.8971 5.8724

5.1784 5.1399 5.1195

2.6562 2.4480 2.4302 2.1613 2.1407 2.0031 1.9882 1.9693 1.9513 1.9358

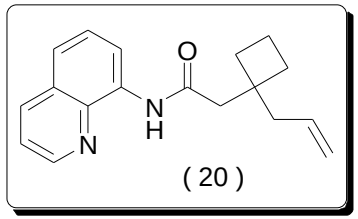


(20)

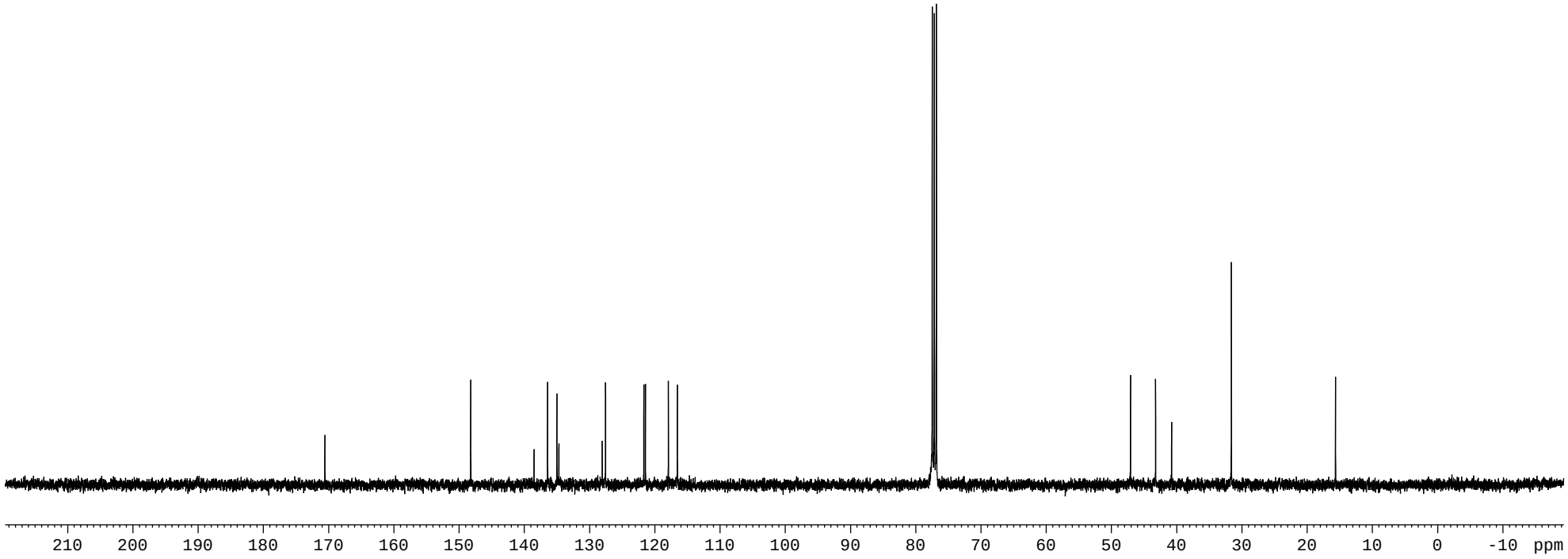


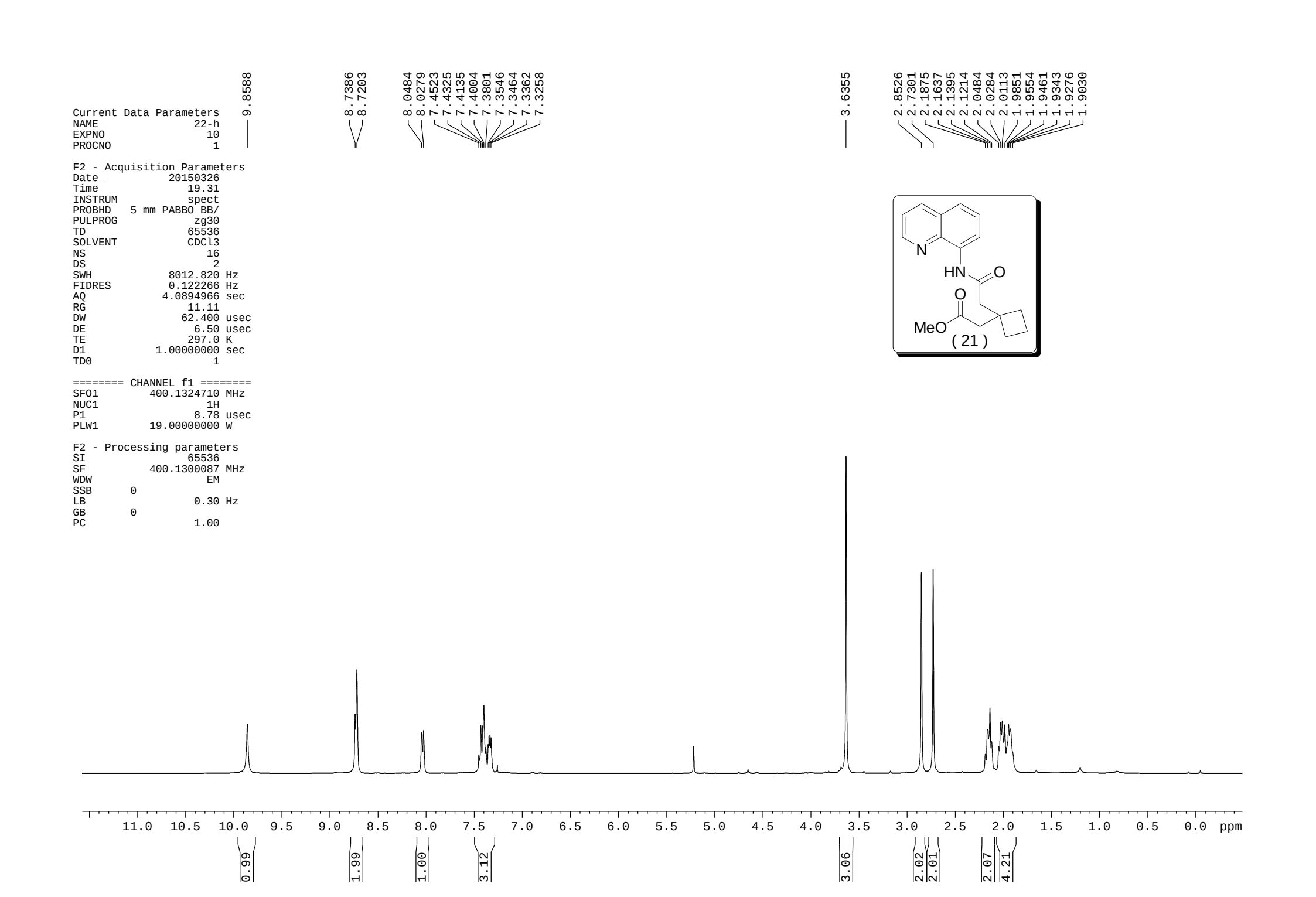
```
Current Data Parameters
NAME     YXL-10-213-2-2-C
EXPNO                  13
PROCNO                  1

F2 - Acquisition Paramete
Date_           20160504
Time               23.33
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   250
DS                     4
SWH           24038.461 H
```

170.5755
148.2173
138.5115
136.4410
135.0003
134.7080
128.0671
127.5594
121.6820
121.4410
117.9266
116.5391
47.0800
43.2385
40.7754
31.6225
15.6541



(20)

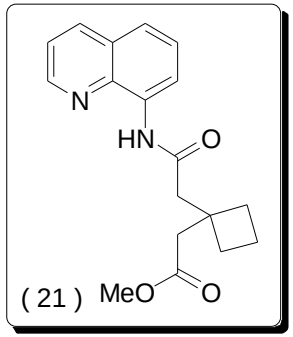



9.8588
8.7386
8.7203
8.0484
8.0279
7.4523
7.4325
7.4135
7.4004
7.3801
7.3546
7.3464
7.3362
7.3258
3.6355
2.8526
2.7301
2.1875
2.1637
2.1395
2.1214
2.0484
2.0284
2.0113
1.9851
1.9554
1.9461
1.9343
1.9276
1.9030
Current Data Parameters
NAME 22-h
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150326
Time 19.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 11.11
DW 62.400 usec
DE 6.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 8.78 usec
PLW1 19.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
HN
O
O
MeO
(21)
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.99
1.99
1.00
3.12
3.06
2.02
2.01
2.07
4.21

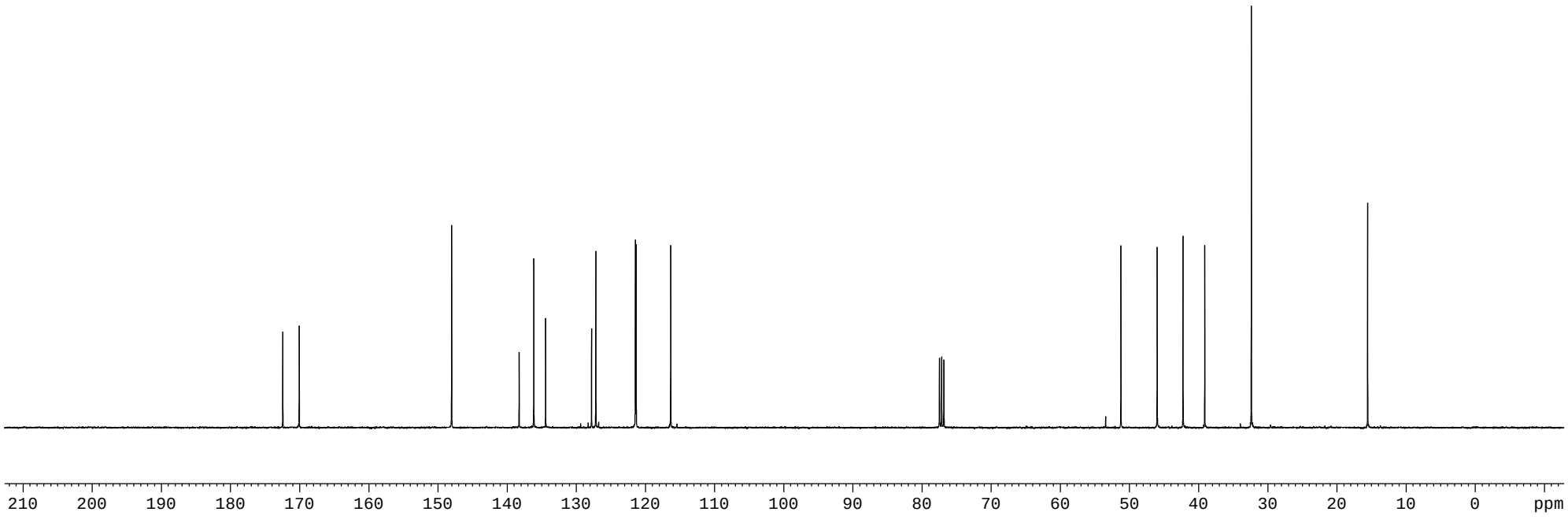
Current Data Parameters
NAME 22-c
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150326
Time 19.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200

172.4171
170.0402
148.0379
138.2809
136.1498
134.4393
127.8044
127.1773
121.4555
121.3485
116.3489
51.2395
45.9965
42.2539
39.1199
32.3753
15.5482

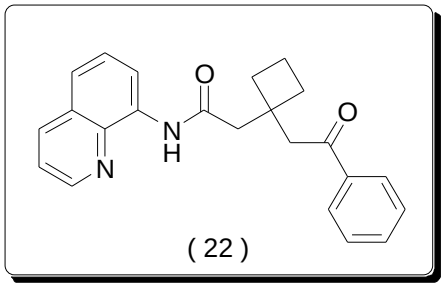


(21)

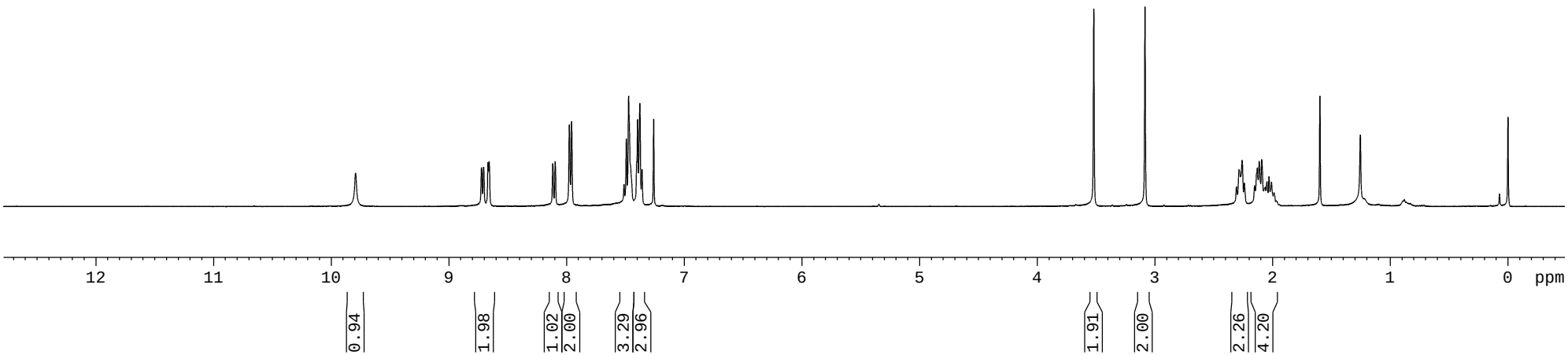


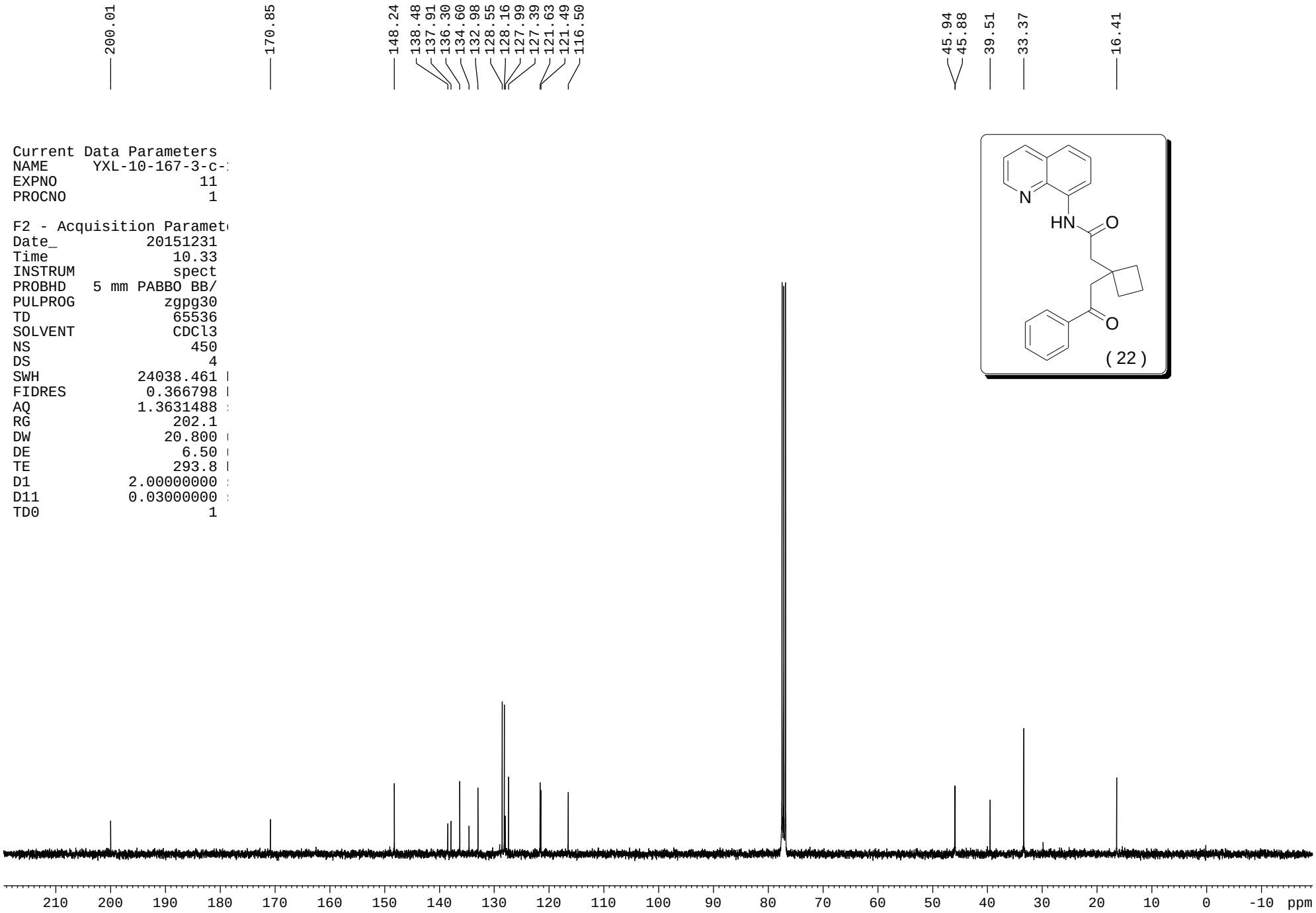
```
Current Data Parameters
NAME      YXL-10-213-9-2-H
EXPNO                  11
PROCNO                  1

F2 - Acquisition Paramete
Date_           20160505
Time               20.51
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 H
FIDRES          0.122266 H
```

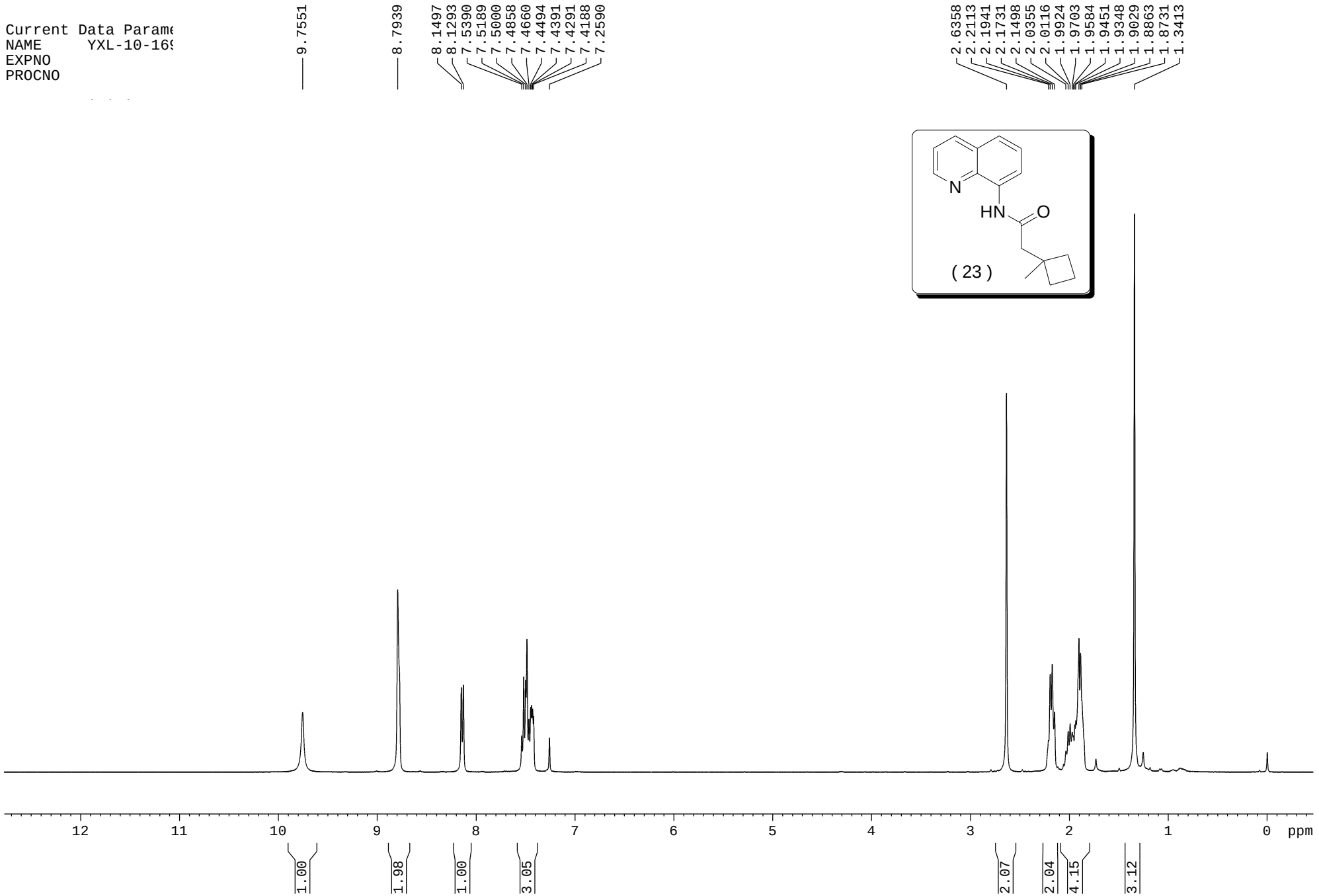
9.7938 | 8.7236 8.7058 8.6679 8.6578 8.1174 8.0969 7.9769 7.9575 7.5120 7.4920 7.4730 7.4042 7.3964 7.3774 7.3592 7.2596 | 3.5193 | 3.0838 2.3072 2.2848 2.2584 2.2400 2.1525 2.1299 2.1132 2.0921 2.0629 2.0477 2.0300 2.0087 1.9874

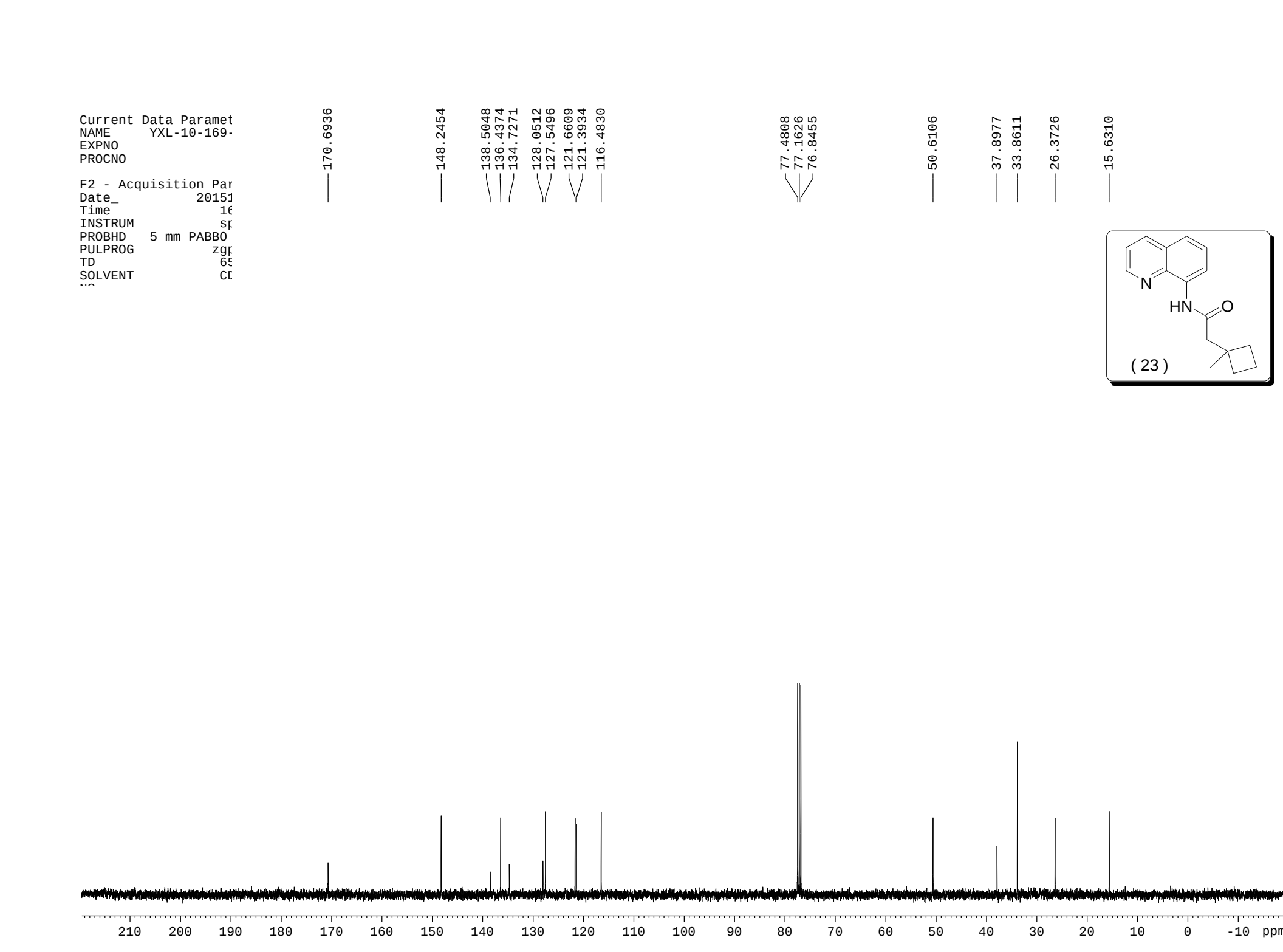


(22)

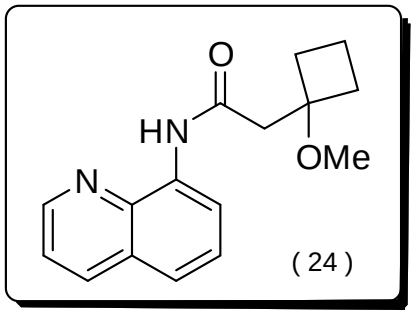


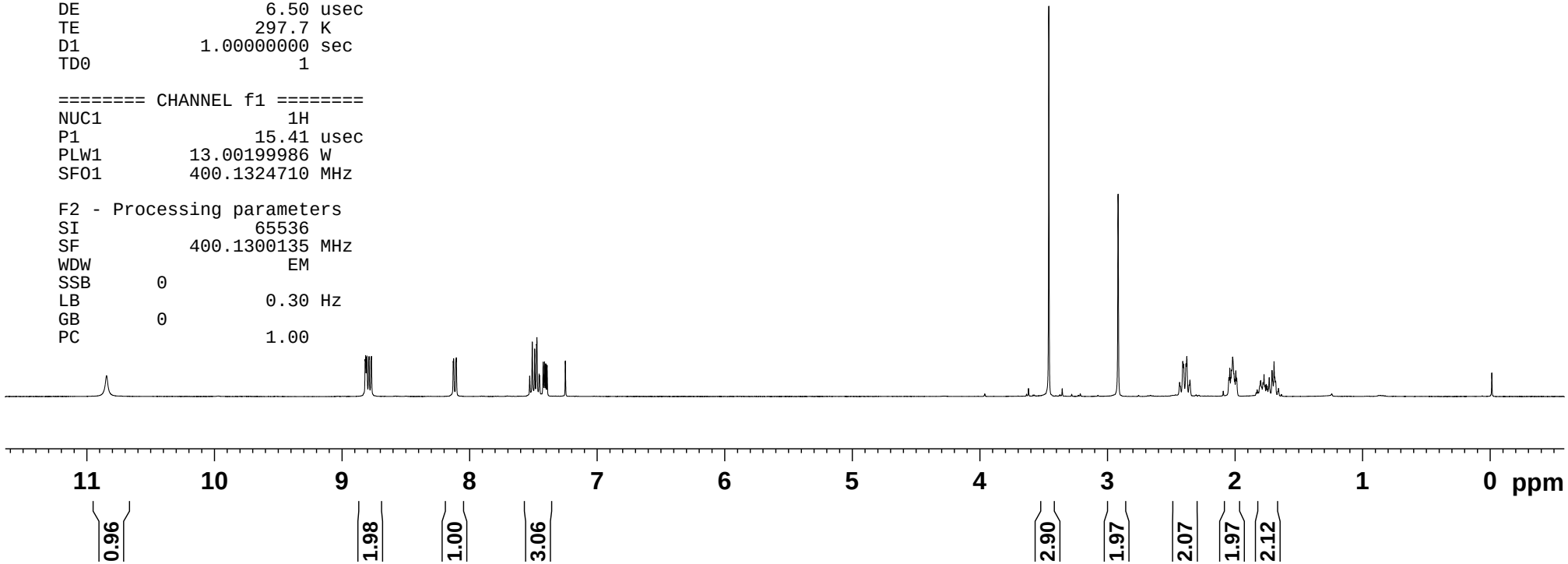

200.01
170.85
148.24
138.48
137.91
136.30
134.60
132.98
128.55
128.16
127.99
127.39
121.63
121.49
116.50
45.94
45.88
39.51
33.37
16.41
Current Data Parameters
NAME YXL-10-167-3-c-
EXPNO 11
PROCNO 1
F2 - Acquisition Paramet
Date_ 20151231
Time 10.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 450
DS 4
SWH 24038.461
FIDRES 0.366798
AQ 1.3631488
RG 202.1
DW 20.800
DE 6.50
TE 293.8
D1 2.00000000
D11 0.03000000
TD0 1
N
HN
O
O
(22)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm


Current Data Parame
NAME YXL-10-16
EXPNO
PROCNO
9.7551
8.7939
8.1497
8.1293
7.5390
7.5189
7.5000
7.4858
7.4660
7.4494
7.4391
7.4291
7.4188
7.2590
2.6358
2.2113
2.1941
2.1731
2.1498
2.0355
2.0116
1.9924
1.9703
1.9584
1.9451
1.9348
1.9029
1.8863
1.8731
1.3413
N
HN
O
(23)
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
1.98
1.00
3.05
2.07
2.04
4.15
3.12


Current Data Paramet
NAME YXL-10-169-
EXPNO
PROCNO
F2 - Acquisition Par
Date_ 20151
Time 16
INSTRUM sp
PROBHD 5 mm PABBO
PULPROG zgp
TD 65
SOLVENT CD
170.6936
148.2454
138.5048
136.4374
134.7271
128.0512
127.5496
121.6609
121.3934
116.4830
77.4808
77.1626
76.8455
50.6106
37.8977
33.8611
26.3726
15.6310
N
HN
O
(23)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm


```
Current Data Parameters
NAME     24
EXPNO                11
PROCNO                1

F2 - Acquisition Parameters
Date_           20161028
Time               21.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9845889 sec
RG                115.38
DW                60.800 usec
DE                 6.50 usec
TE                297.7 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                15.41 usec
PLW1        13.00199986 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300135 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

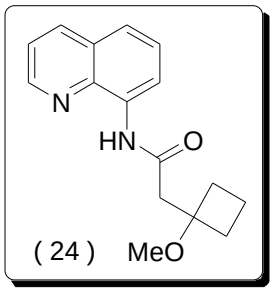


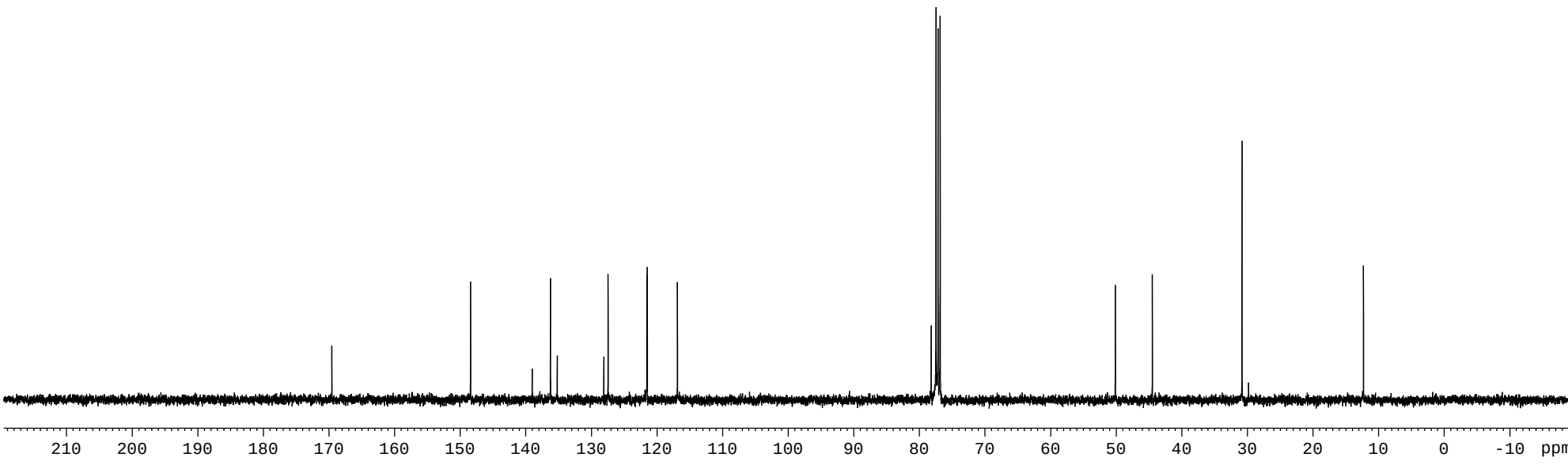


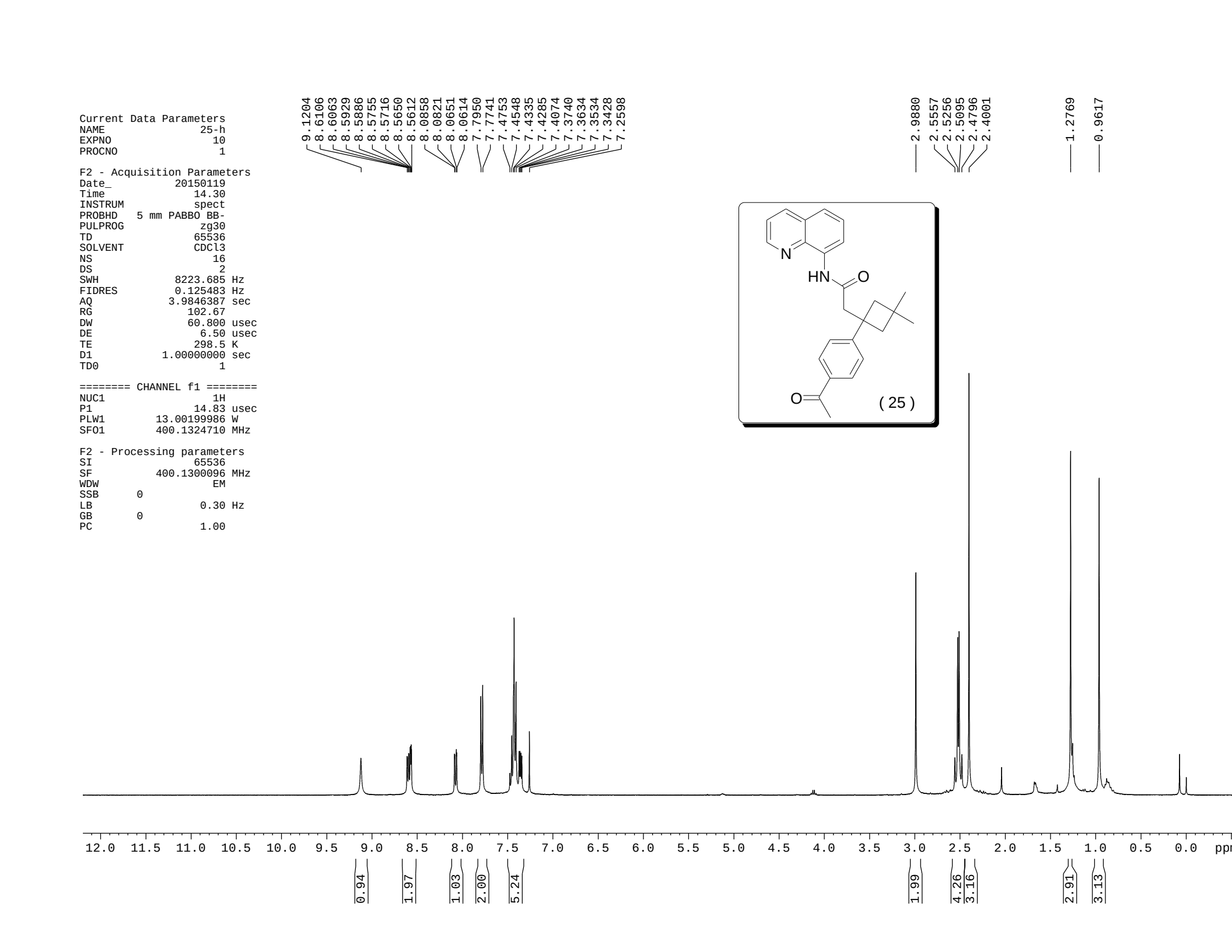
Current Data Parameters
NAME 24-c
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140910
Time 8.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200

169.5771
148.4256
139.0238
136.2352
135.2061
128.1085
127.4639
121.5500
121.4790
116.8859
78.2061
77.4757
77.1581
76.8405
50.1394
44.4965
30.8192
12.3055

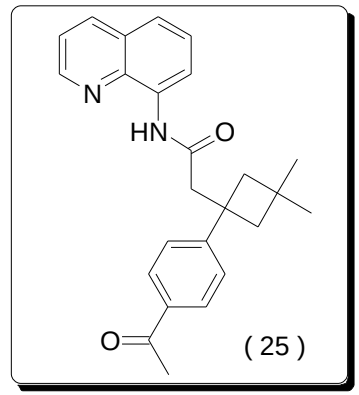


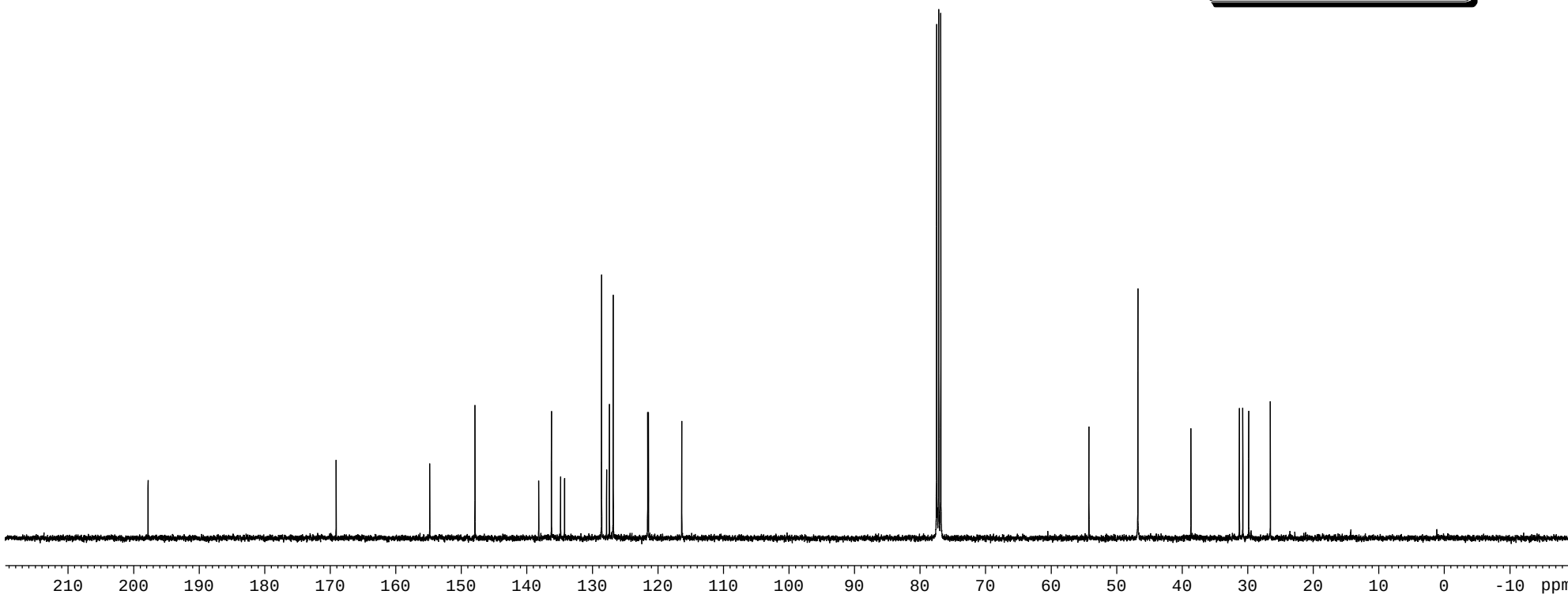



Current Data Parameters
NAME 25-h
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150119
Time 14.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 102.67
DW 60.800 usec
DE 6.50 usec
TE 298.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.1204
8.6106
8.6063
8.5929
8.5886
8.5755
8.5716
8.5650
8.5612
8.0858
8.0821
8.0651
8.0614
7.7950
7.7741
7.4753
7.4548
7.4335
7.4285
7.4074
7.3740
7.3634
7.3534
7.3428
7.2598
2.9880
2.5557
2.5256
2.5095
2.4796
2.4001
1.2769
0.9617
N
HN
O
O
(25)
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.94
1.97
1.03
2.00
5.24
1.99
4.26
3.16
2.91
3.13

197.8068
169.1115
154.8300
147.9312
138.1987
136.2505
134.8476
134.2496
128.6022
127.8366
127.4066
126.8264
121.5693
121.4556
116.3641
77.4785
77.1614
76.8433
54.2336
46.7651
38.6742
31.2770
30.7554
29.8649
26.5553

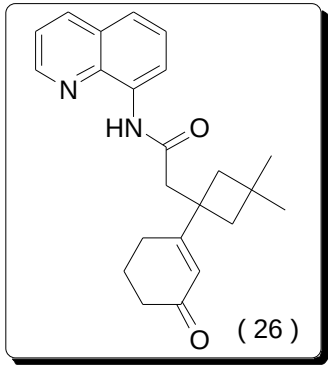
```
Current Data Parameters
NAME                 25-c
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150120
Time                6.26
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
```

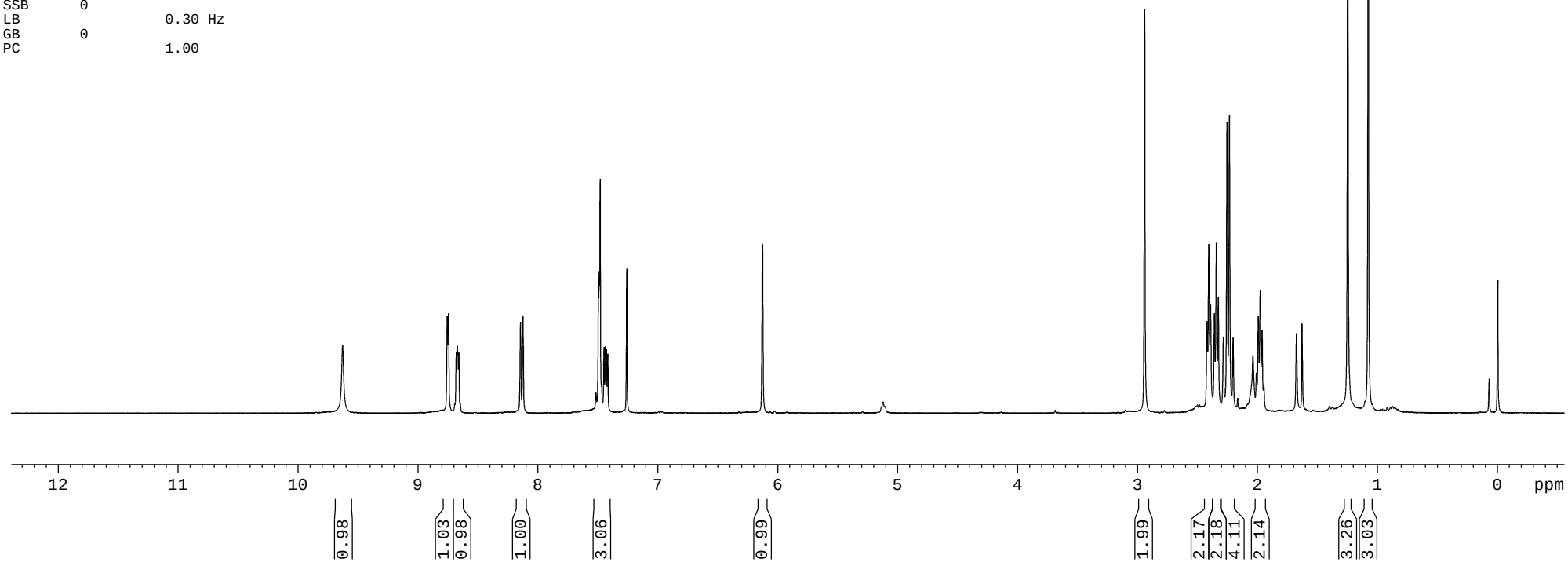


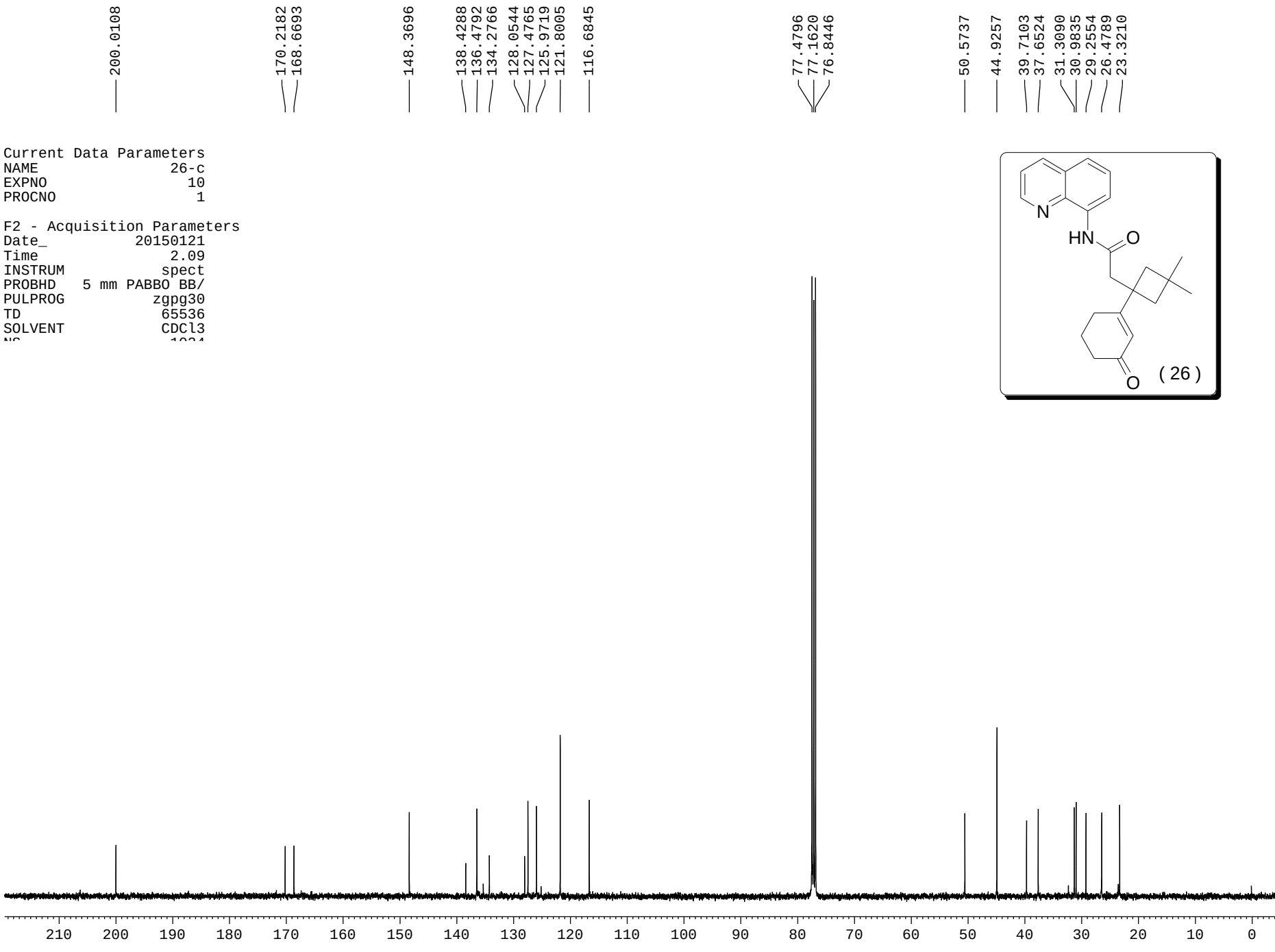


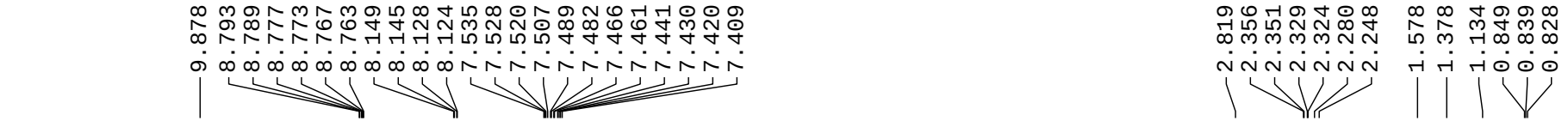
```
Current Data Parameters
NAME                 26-h
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150120
Time               17.19
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                 80.88
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                  8.78 usec
PLW1         19.00000000 W

F2 - Processing parameters
SI                 65536
SF            400.1300108 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

9.6273
8.7568 8.7534 8.7464 8.6810 8.6727 8.6669 8.6589
8.1457 8.1251
7.4946 7.4882 7.4829 7.4805 7.4485 7.4379 7.4279 7.4173 7.2613 7.2587
6.1269
2.9403 2.4204 2.4058 2.3910 2.3587 2.3428 2.3261 2.2846 2.2534 2.2336 2.2031 2.0377 2.0081 1.9928 1.9770 1.9616 1.2461 1.0757

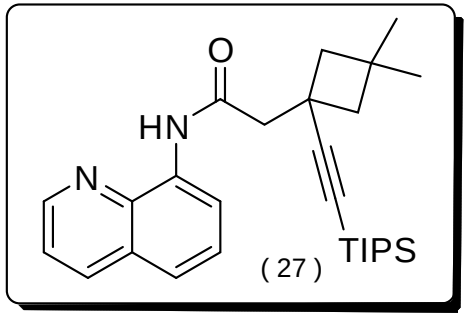


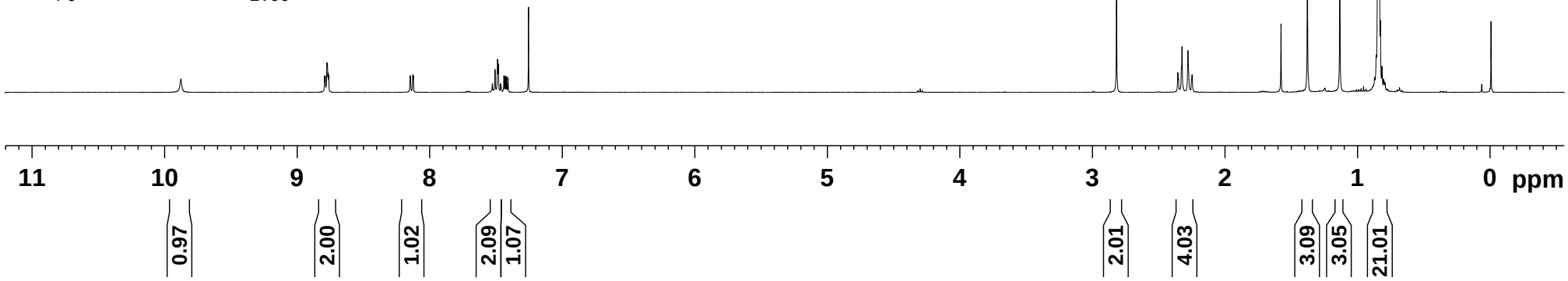



200.0108
170.2182
168.6693
148.3696
138.4288
136.4792
134.2766
128.0544
127.4765
125.9719
121.8005
116.6845
77.4796
77.1620
76.8446
50.5737
44.9257
39.7103
37.6524
31.3090
30.9835
29.2554
26.4789
23.3210
Current Data Parameters
NAME 26-c
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150121
Time 2.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
N
HN
O
O
(26)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm



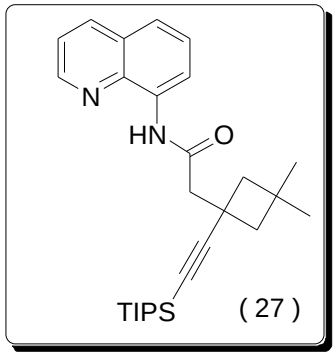

```
Current Data Parameters
NAME     27
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20161029
Time              12.52
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ            4.0894465 sec
RG                99.51
DW               62.400 usec
DE                6.50 usec
TE               293.3 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                9.38 usec
PLW1        19.00000000 W

F2 - Processing parameters
SI                65536
SF          400.1300114 MHz
WDW                 EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```

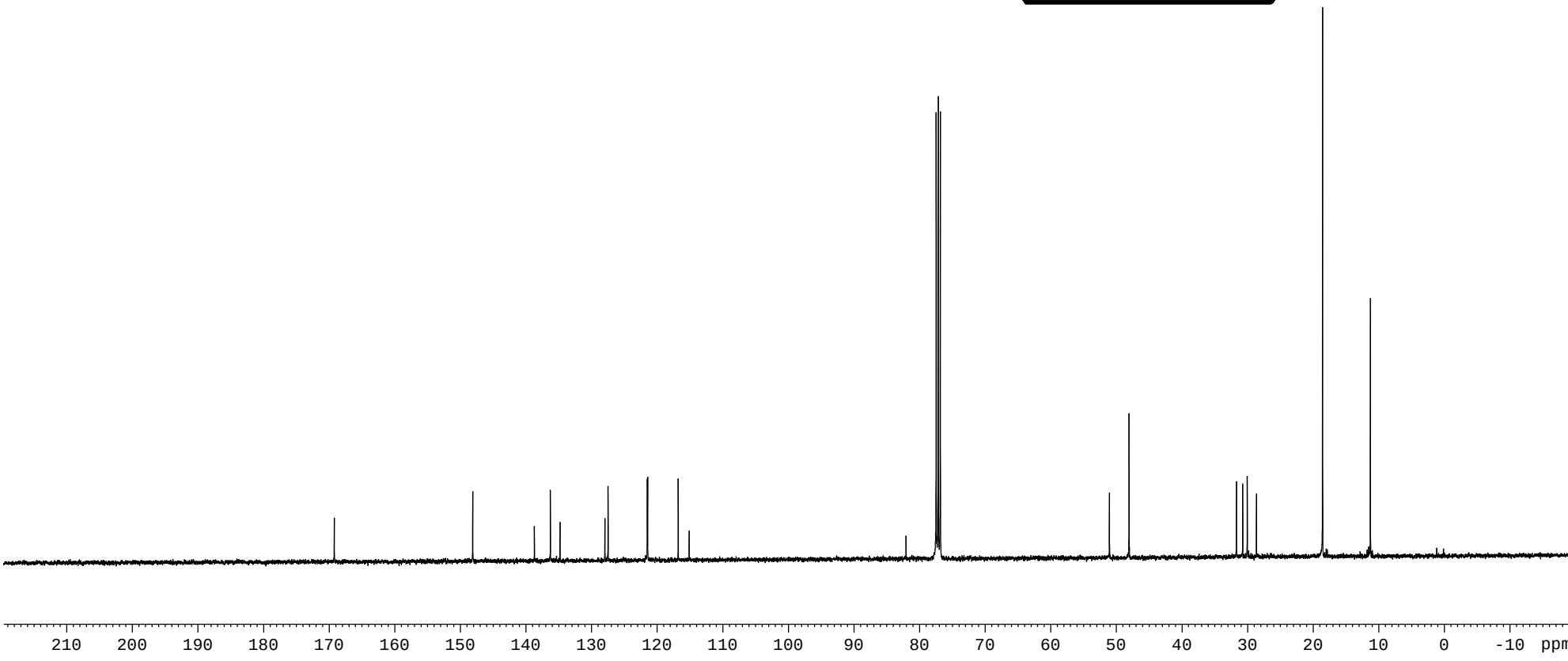




```
Current Data Parameters
NAME                  27-c
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150124
Time                0.02
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
```

169.2229
148.1235
138.7293
136.2924
134.8081
127.9679
127.4960
121.5156
121.4572
116.8046
115.1336
82.0803
77.4778
77.3660
77.1608
76.8428
51.0635
48.0786
31.6956
30.7464
30.0593
28.6522
18.5696
11.2788

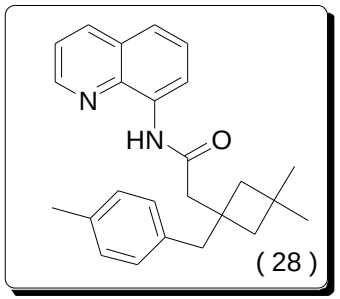


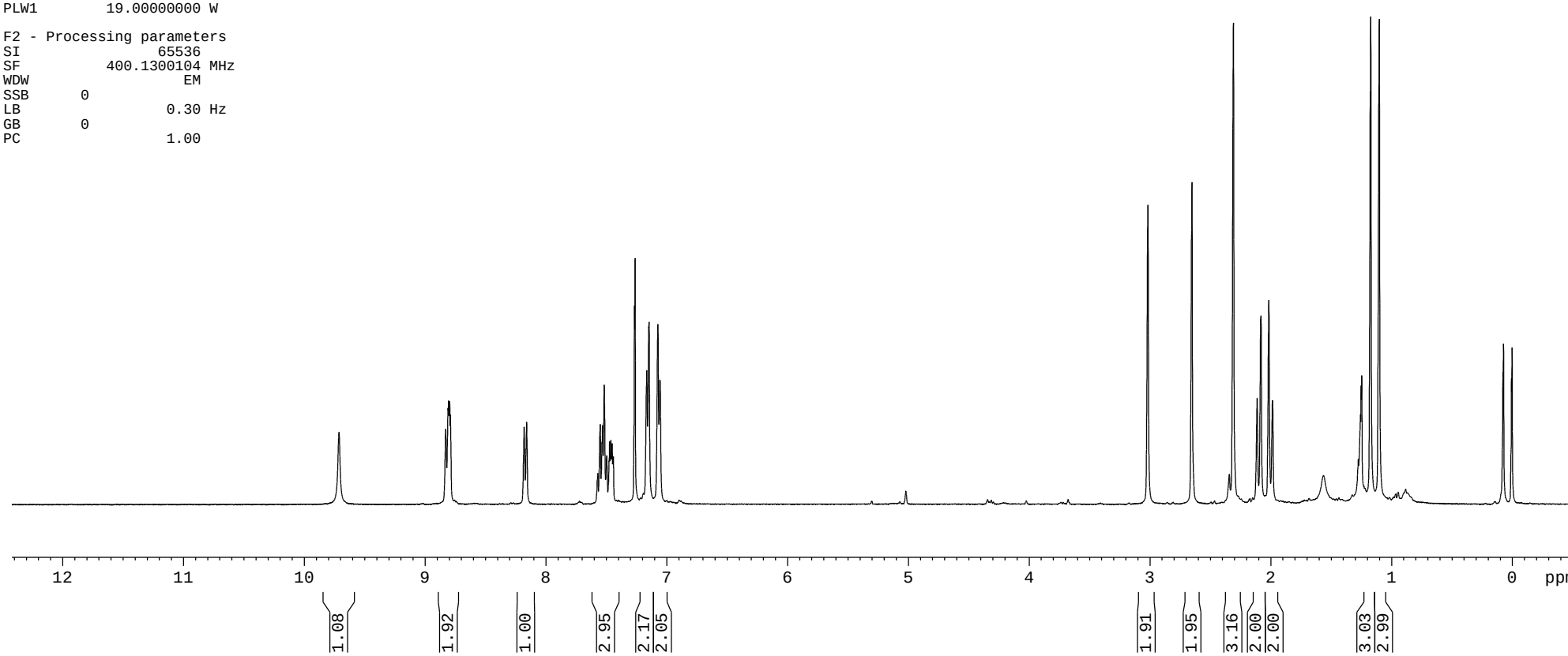


```
Current Data Parameters
NAME                 28-h
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150120
Time               17.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894966 sec
RG                 112.9
DW                62.400 usec
DE                 6.50 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                1H
P1                  8.78 usec
PLW1         19.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300104 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

9.7104 8.8271 8.8080 8.8044 8.7990 8.7940 8.7887 8.1780 8.1575 7.5679 7.5479 7.5335 7.5286 7.5153 7.4944 7.4700 7.4651 7.4596 7.4551 7.4494 7.4445 7.4392 7.2686 7.2648 7.2595 7.1629 7.1435 7.0706 7.0538

3.0161 2.6517 2.3086 2.1122 2.0803 2.0151 1.9854 1.1719 1.1013

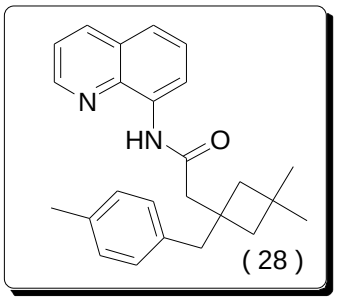




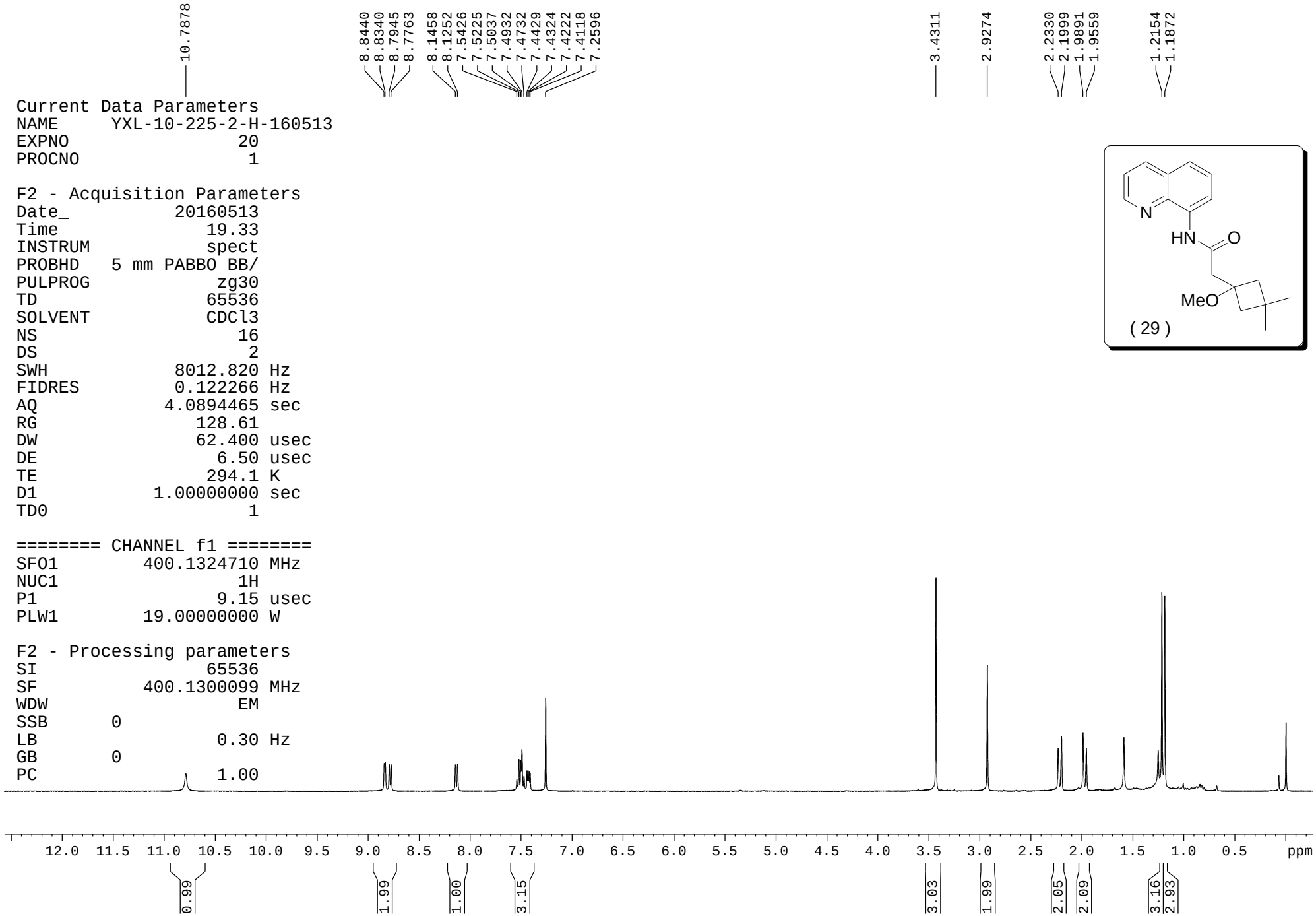
Current Data Parameters
NAME 28-c
EXPNO 10
PROCNO 1

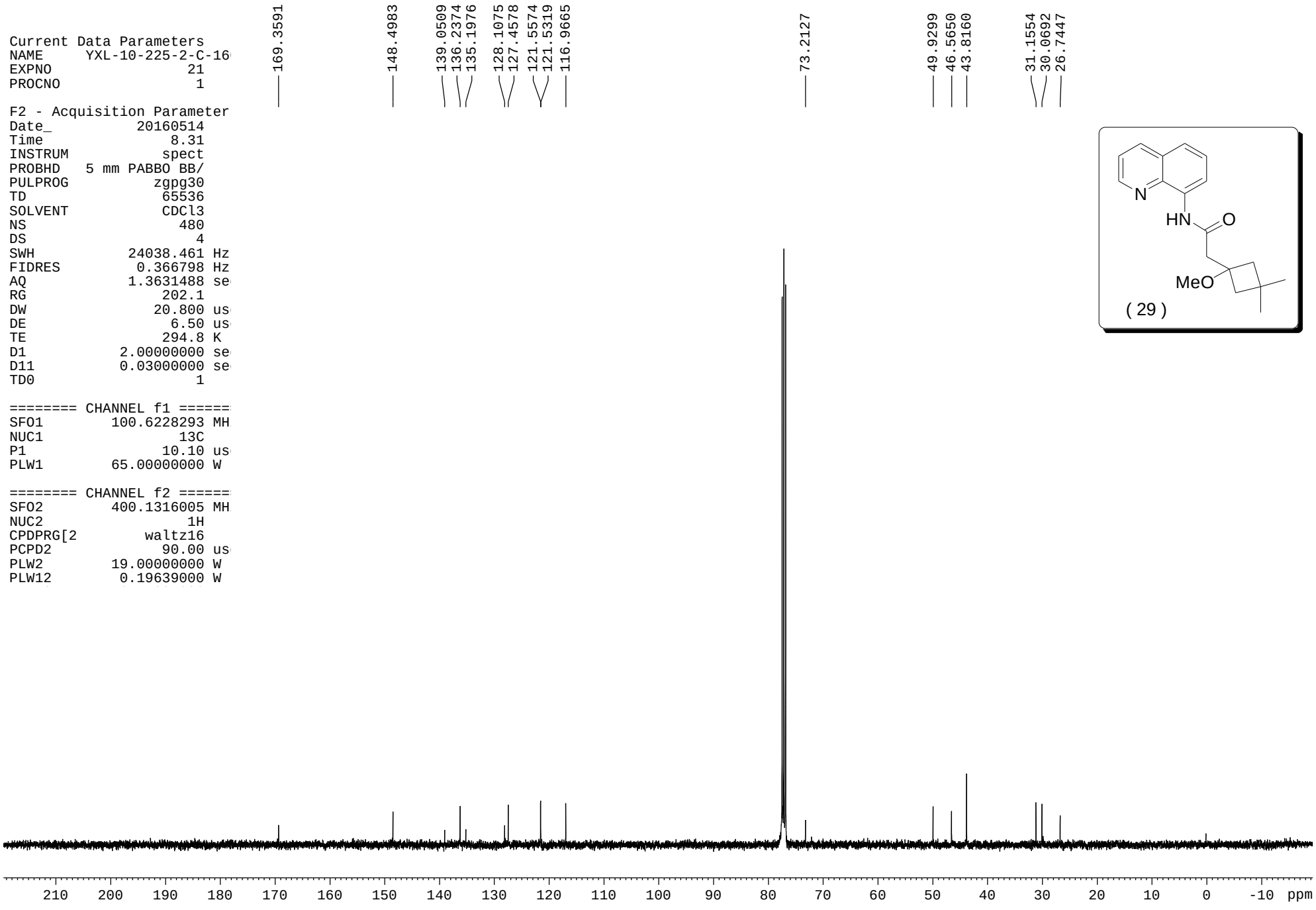
F2 - Acquisition Parameters
Date_ 20150121
Time 8.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3

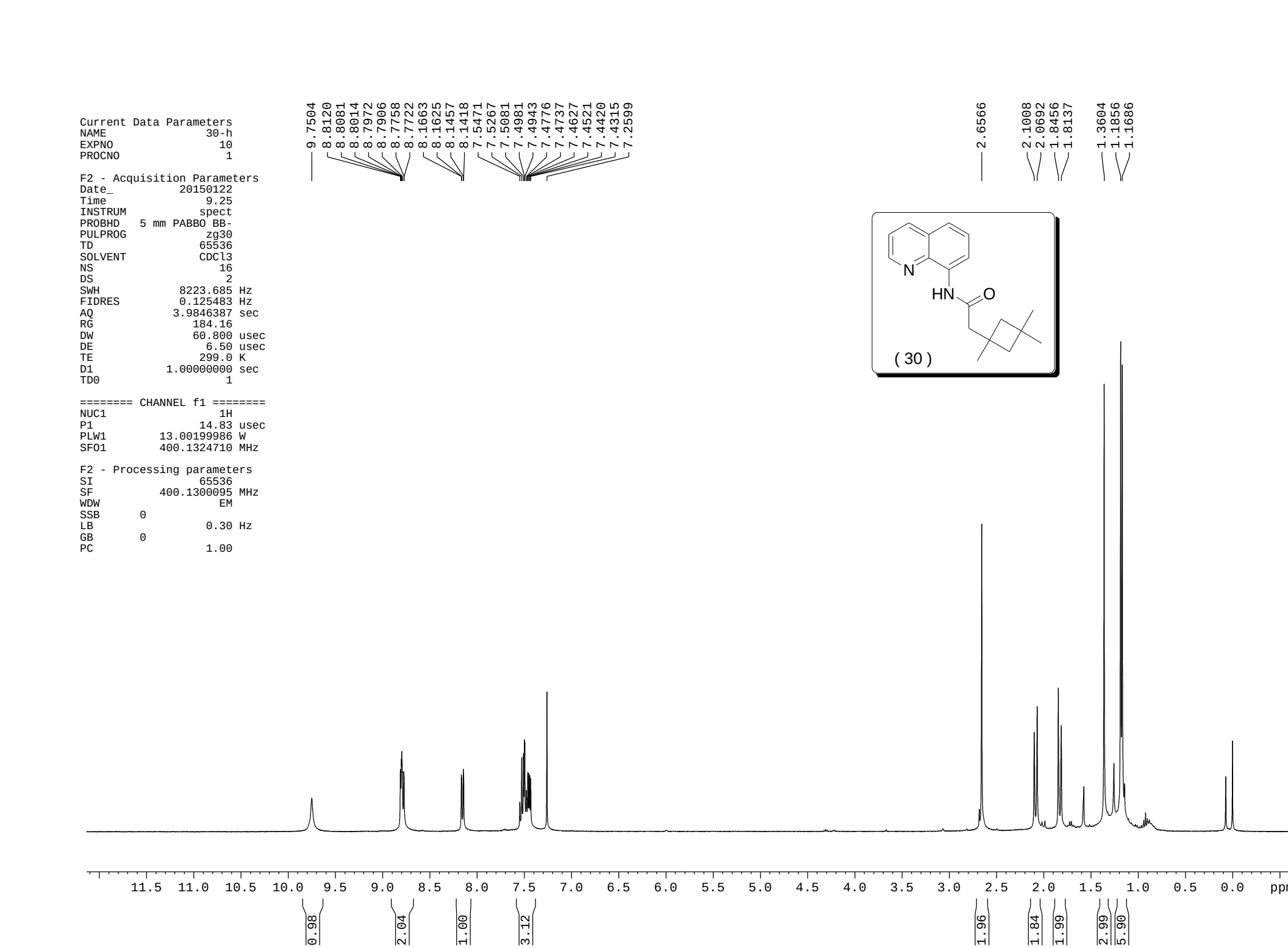
170.7536
148.2214
138.5555
136.4655
136.2499
135.6377
134.7538
130.3239
128.9215
128.1088
127.6086
121.6976
121.4384
116.6087
47.7988
45.8222
44.9469
33.3157
32.0506
31.6057
28.3381
21.1765

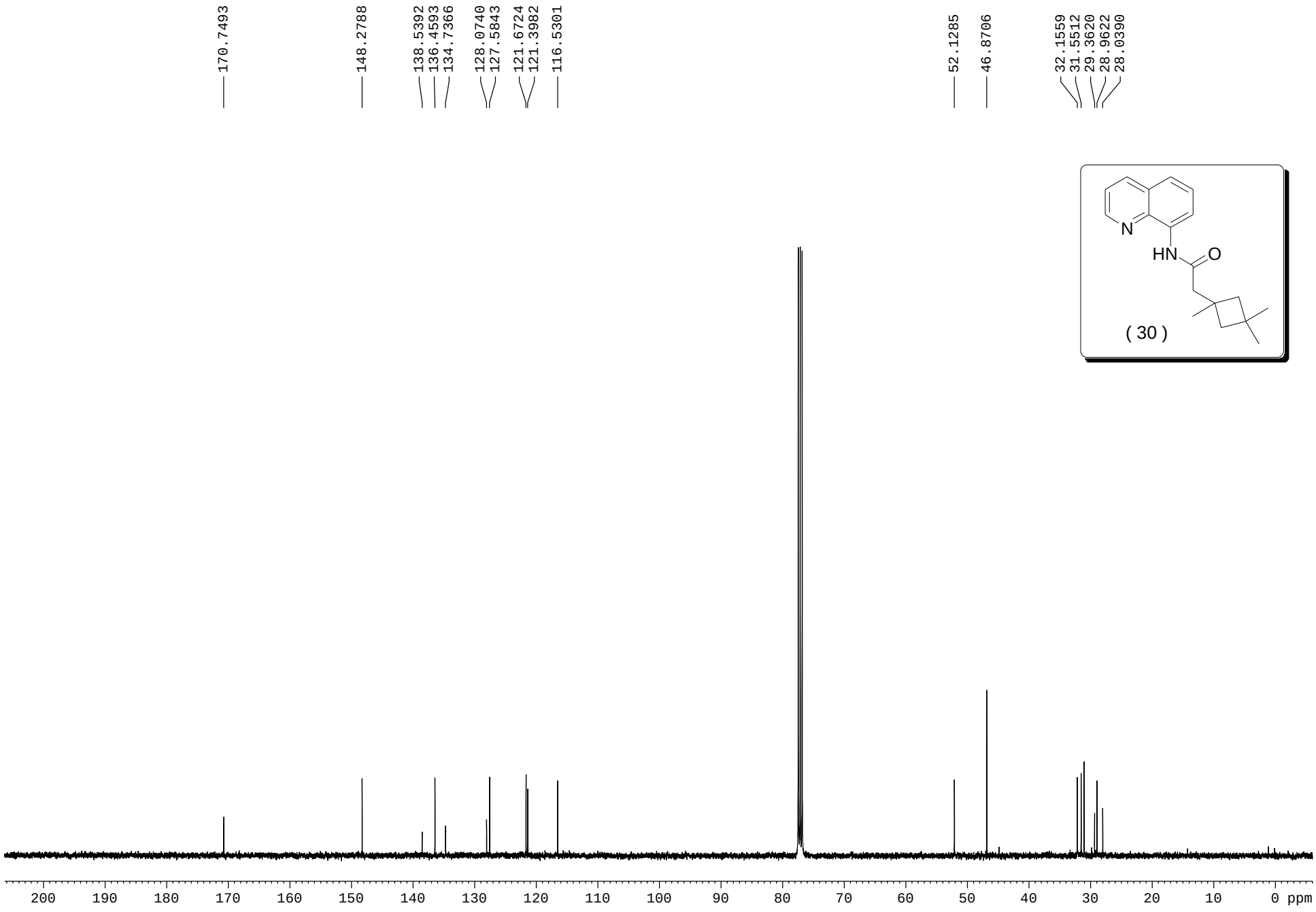


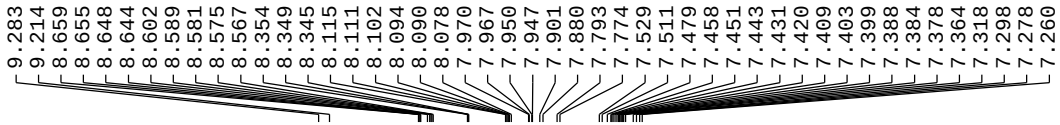
(28)

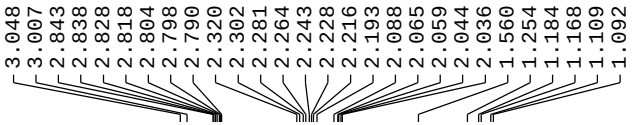

10.7878
8.8440
8.8340
8.7945
8.7763
8.1458
8.1252
7.5426
7.5225
7.5037
7.4932
7.4732
7.4429
7.4324
7.4222
7.4118
7.2596
3.4311
2.9274
2.2330
2.1999
1.9891
1.9559
1.2154
1.1872
Current Data Parameters
NAME YXL-10-225-2-H-160513
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160513
Time 19.33
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 128.61
DW 62.400 usec
DE 6.50 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.15 usec
PLW1 19.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
HN
O
MeO
(29)
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.99
1.99
1.00
3.15
3.03
1.99
2.05
2.09
3.16
2.93


169.3591
148.4983
139.0509
136.2374
135.1976
128.1075
127.4578
121.5574
121.5319
116.9665
73.2127
49.9299
46.5650
43.8160
31.1554
30.0692
26.7447
Current Data Parameters
NAME YXL-10-225-2-C-16
EXPNO 21
PROCNO 1
F2 - Acquisition Parameter
Date_ 20160514
Time 8.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 480
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 se
RG 202.1
DW 20.800 us
DE 6.50 us
TE 294.8 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1
======== CHANNEL f1 ======
SFO1 100.6228293 MH
NUC1 13C
P1 10.10 us
PLW1 65.00000000 W
======== CHANNEL f2 ======
SFO2 400.1316005 MH
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 us
PLW2 19.00000000 W
PLW12 0.19639000 W
N
HN
O
MeO
(29)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 ppm

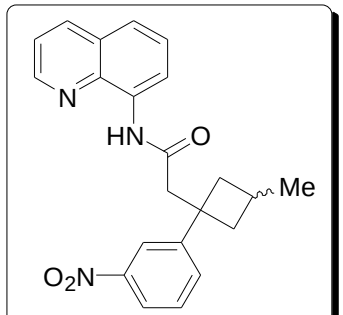

Current Data Parameters
NAME 30-h
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150122
Time 9.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 184.16
DW 60.800 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.7504
8.8120
8.8081
8.8014
8.7972
8.7906
8.7758
8.7722
8.1663
8.1625
8.1457
8.1418
7.5471
7.5267
7.5081
7.4981
7.4943
7.4776
7.4737
7.4627
7.4521
7.4420
7.4315
7.2599
2.6566
2.1008
2.0692
1.8456
1.8137
1.3604
1.1856
1.1686
N
HN
O
(30)
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
2.04
1.00
3.12
1.96
1.84
1.99
2.99
5.90

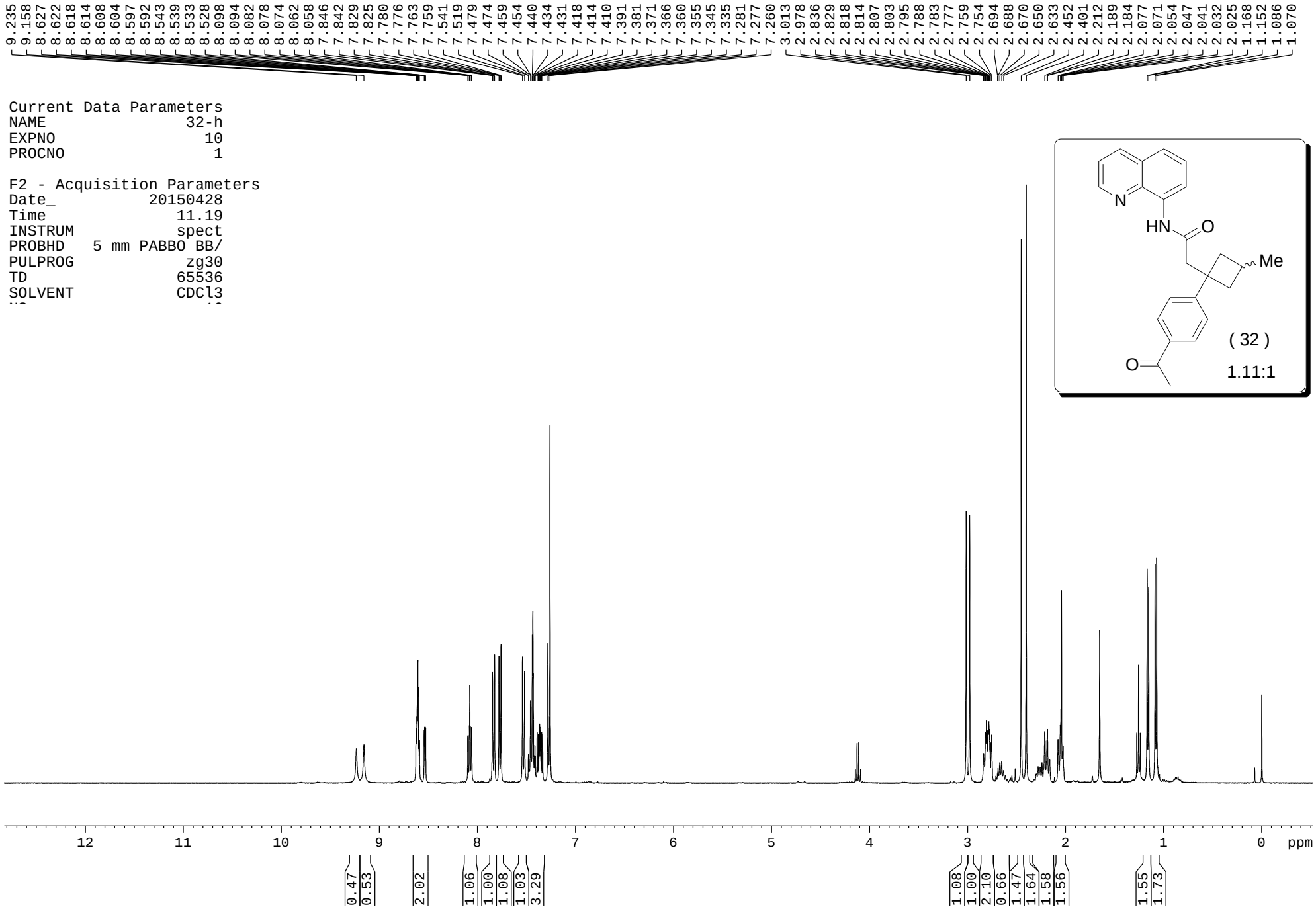

170.7493
148.2788
138.5392
136.4593
134.7366
128.0740
127.5843
121.6724
121.3982
116.5301
52.1285
46.8706
32.1559
31.5512
29.3620
28.9622
28.0390
N
HN
O
(30)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm






```
Current Data Parameters
NAME                 31-h
EXPNO                  40
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150520
Time               11.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
```




9.235 9.158 8.627 8.622 8.618 8.614 8.608 8.604 8.597 8.592 8.543 8.539 8.533 8.528 8.098 8.094 8.082 8.078 8.074 8.062 8.058 7.846 7.842 7.829 7.825 7.780 7.776 7.763 7.759 7.541 7.519 7.479 7.474 7.459 7.454 7.440 7.434 7.431 7.418 7.414 7.410 7.391 7.381 7.371 7.366 7.360 7.355 7.345 7.335 7.281 7.277 7.260 3.013 2.978 2.836 2.829 2.818 2.814 2.807 2.803 2.795 2.788 2.783 2.777 2.759 2.754 2.694 2.688 2.670 2.650 2.633 2.452 2.401 2.212 2.189 2.184 2.077 2.071 2.054 2.047 2.041 2.032 2.025 1.168 1.152 1.086 1.070
Current Data Parameters
NAME 32-h
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150428
Time 11.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
N
HN
O
Me
O
(32)
1.11:1
12 11 10 9 8 7 6 5 4 3 2 1 0 ppm
0.47 0.53 2.02 1.06 1.00 1.08 1.03 3.29 1.08 1.00 2.10 0.66 1.47 1.64 1.58 1.56 1.55 1.73

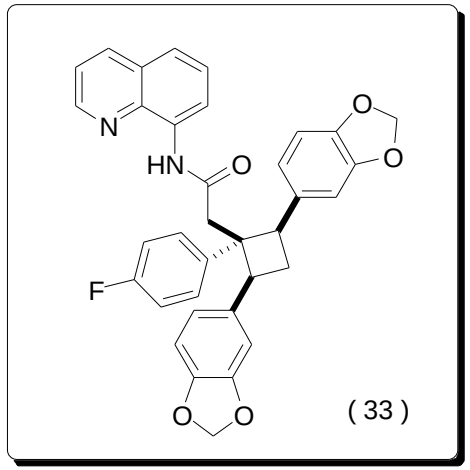
```
Current Data Parameters
NAME      YXL-10-213-8-H-160
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20160508
Time               16.02
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
```

8.8290 8.6178 8.6095 8.2344 8.2162 8.0563 8.0359 7.6029 7.5893 7.5819 7.5686 7.3873 7.3768 7.3667 7.3558 7.3297 7.3150 7.2960 7.2758 7.2588 7.2182 7.1968 7.1754 6.8199 6.7994 6.7840 6.6325 6.6128 5.6758 5.5418

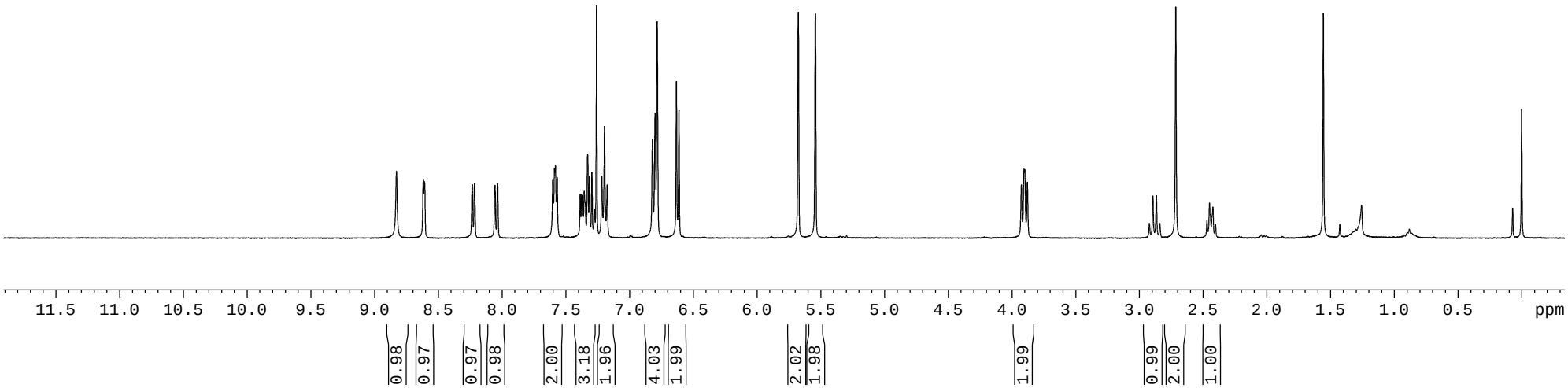
3.9260 3.9056 3.8993 3.8790

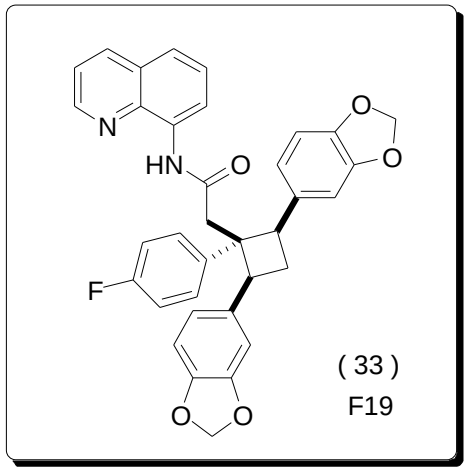
2.9219 2.8943 2.8669 2.8391 2.7147 2.4698 2.4500 2.4233 2.4034

1.5567



(33)



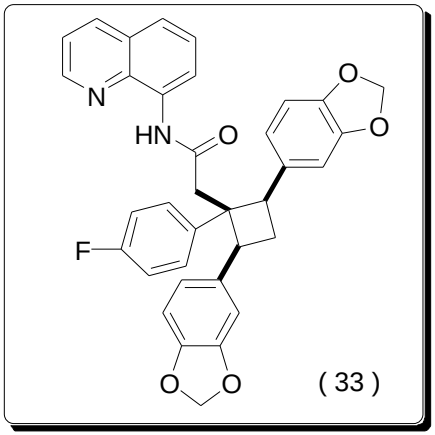


-116.94

0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

```
Current Data Parameters
NAME     YXL-10-213-8-c-160508
EXPNO                  13
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160509
Time                5.54
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1000
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                 202.1
DW                20.800 usec
DE                 6.50 usec
TE                299.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                 10.10 usec
PLW1         65.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         19.00000000 W
PLW12         0.19639000 W
PLW13         0.15907000 W

F2 - Processing parameters
SI                 32768
SF           100.6127549 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```

168.6793, 162.9402, 160.5563, 147.6505, 147.5762, 146.5304, 142.3374, 138.1748, 136.0524, 134.5346, 132.4274, 127.7917, 127.7165, 127.2514, 121.5907, 121.3693, 120.8104, 116.2935, 115.8566, 115.6484, 109.0972, 108.1920, 100.9176, 55.6770, 46.0328, 39.3878, 25.0906

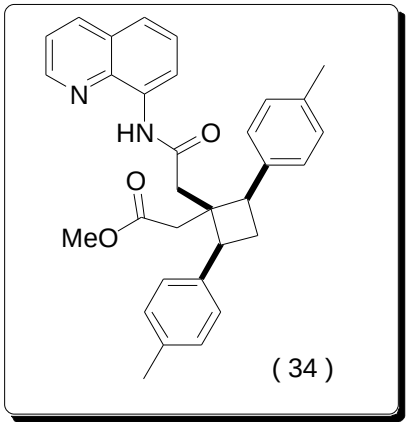


(33)

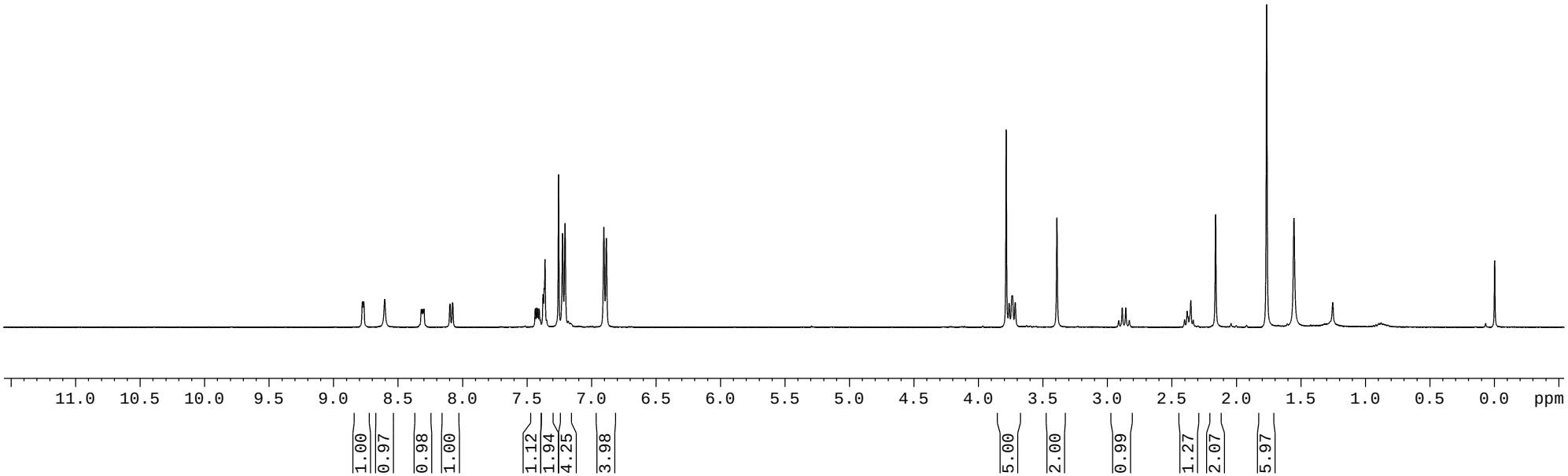
```
Current Data Parameter
NAME      YXL-4-289-H-1
EXPNO                  1
PROCNO                 1

F2 - Acquisition Param
Date_           2016051
Time               10.5
INSTRUM            spec
PROBHD   5 mm PABBO BE
PULPROG             zg3
TD                 6553
SOLVENT            CDCl
NS                    1
DS
SWH             8012.82
FIDRES          0.12226
AQ              4.089446
RG                143.9
```

8.7768 8.7687 8.6055 8.3223 8.3149 8.3072 8.3001 8.0984 8.0793 7.4380 7.4275 7.4174 7.4069 7.3985 7.3773 7.3688 7.3622 7.3492 7.2570 7.2262 7.2066 7.1816 6.9060 6.8867

3.7859 3.7631 3.7418 3.7372 3.7162 3.3925 2.9135 2.8862 2.8587 2.8313 2.4028 2.3827 2.3546 2.3356 2.1620 1.7660 1.5544



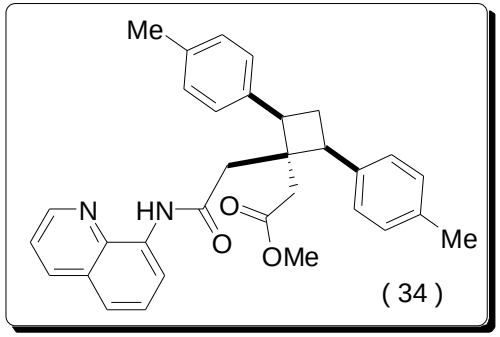
(34)



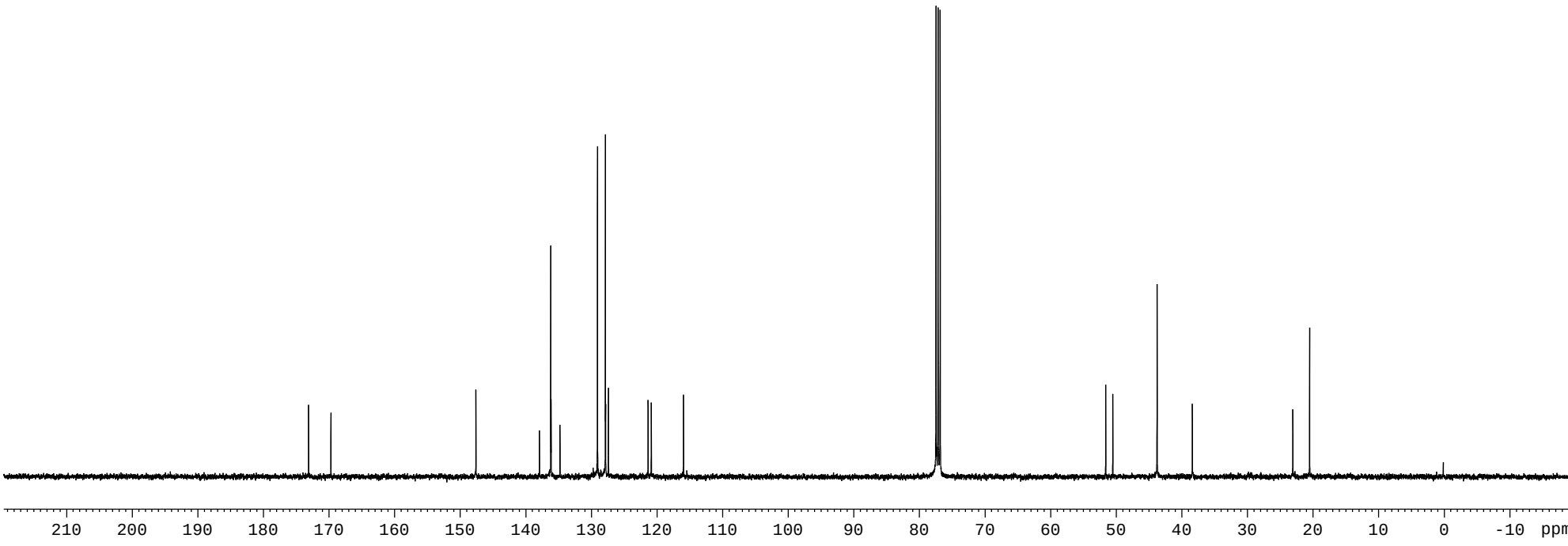
Current Data Parameters
NAME 37-c
EXPNO 10
PROCNO 1

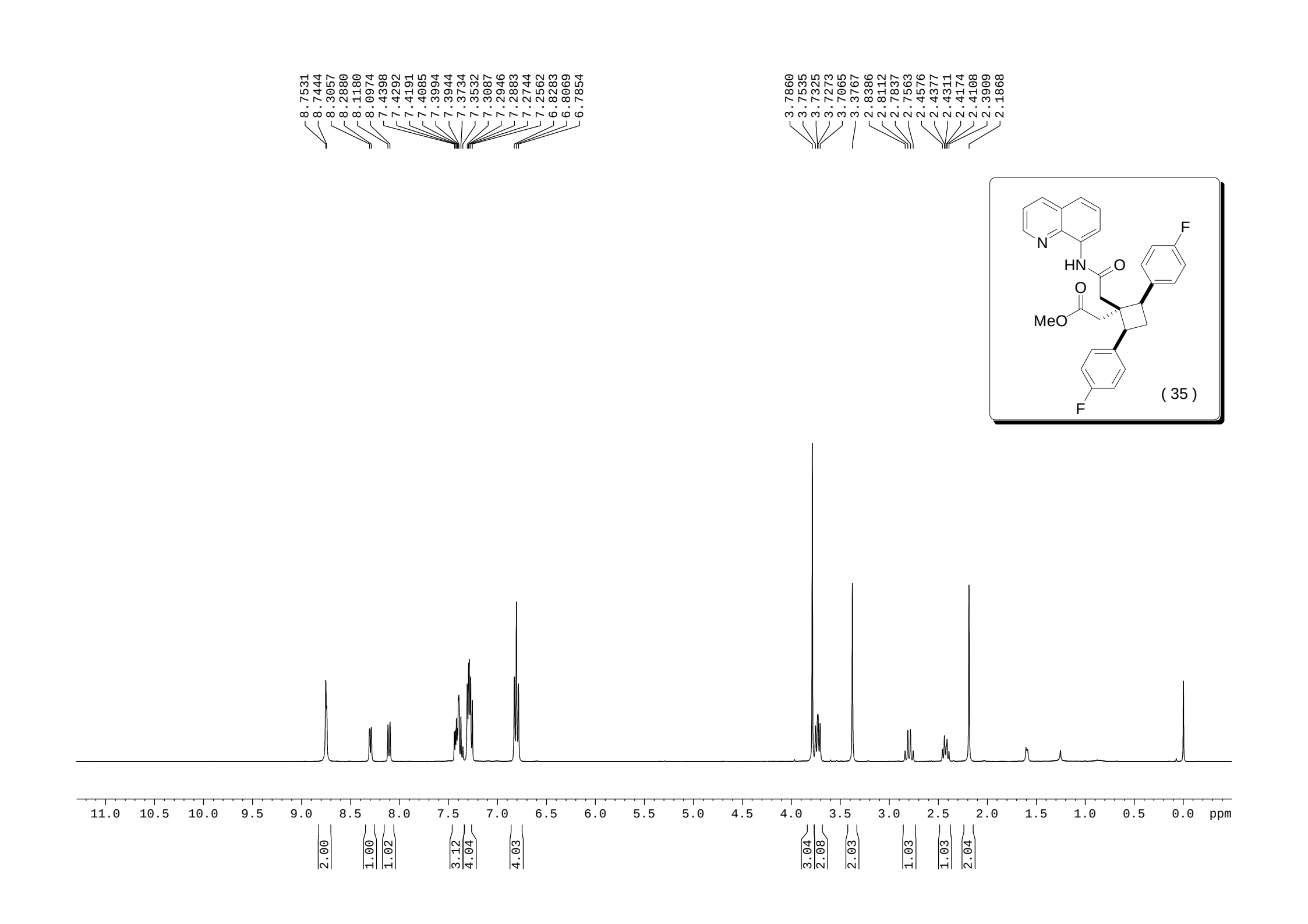
F2 - Acquisition Parameters
Date_ 20150506
Time 2.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024

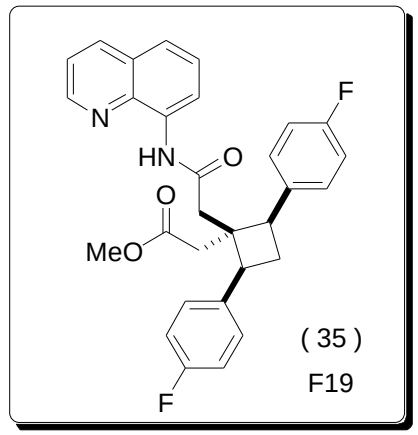
173.1206
169.7158
147.6148
137.9322
136.2049
136.1518
134.7967
129.0824
127.8703
127.8345
127.4112
121.3642
120.8838
115.9722
77.4795
77.1615
76.8444
51.6120
50.5305
43.7789
38.3985
23.0805
20.5256



(34)




8.7531
8.7444
8.3057
8.2880
8.1180
8.0974
7.4398
7.4292
7.4191
7.4085
7.3994
7.3944
7.3734
7.3532
7.3087
7.2946
7.2883
7.2744
7.2562
6.8283
6.8069
6.7854
3.7860
3.7535
3.7325
3.7273
3.7065
3.3767
2.8386
2.8112
2.7837
2.7563
2.4576
2.4377
2.4311
2.4174
2.4108
2.3909
2.1868
N
HN
O
O
MeO
F
F
(35)
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
2.00
1.00
1.02
3.12
4.04
4.03
3.04
2.08
2.03
1.03
1.03
2.04

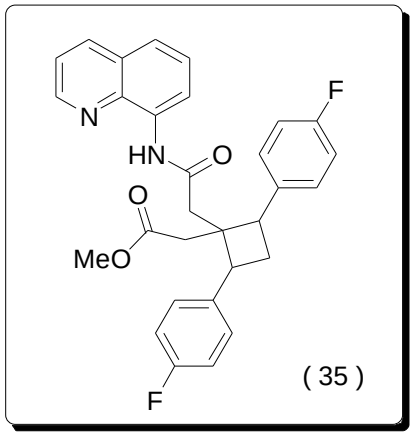


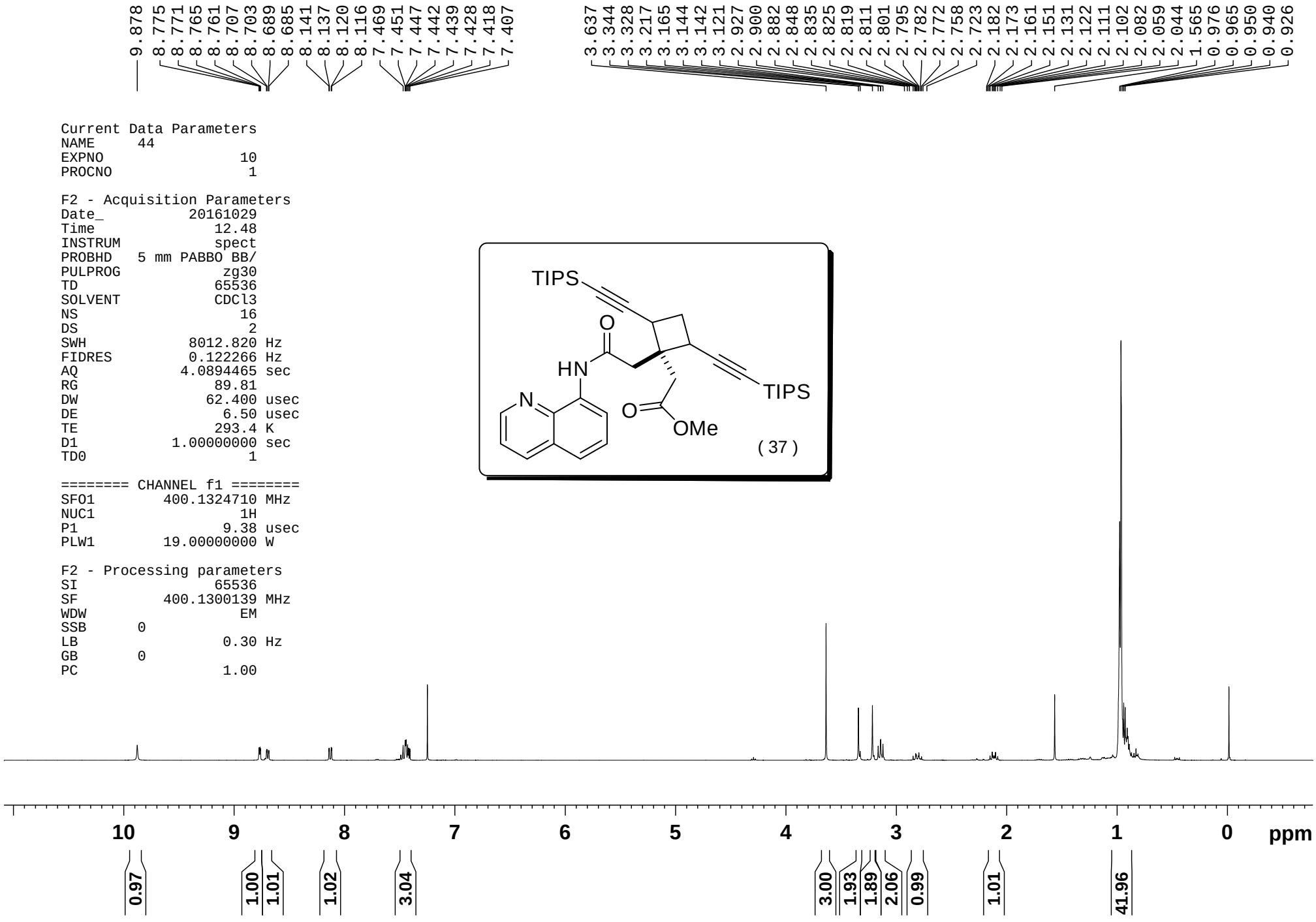
-116.62

0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

```
Current Data Parameters
NAME      YXL-4-303-C-160
EXPNO                 10
PROCNO                 1

F2 - Acquisition Paramet
Date_           20160508
Time               18.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  580
DS                    4
SWH           24038.461
FIDRES         0.366798
AQ            1.3631488
RG                202.1
DW                20.800
DE                 6.50
TE                299.3
D1           2.00000000
D11          0.03000000
TD0                   1

======== CHANNEL f1 ====
SFO1        100.6228293
NUC1               13C
P1                10.10
PLW1        65.00000000

======== CHANNEL f2 ====
SFO2        400.1316005
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00
PLW2        19.00000000
```

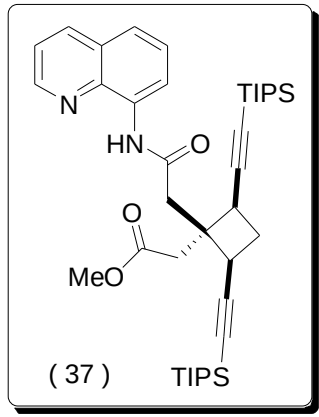
172.8766 169.1870 162.9400 160.5040 147.7788 137.9429 136.4046 134.7654 134.7352 134.3421 129.5530 129.4744 127.9381 127.2762 121.5276 121.3900 116.0616 115.3119 115.1007 51.7172 50.4116 43.7225 43.5971 37.9744 24.0094



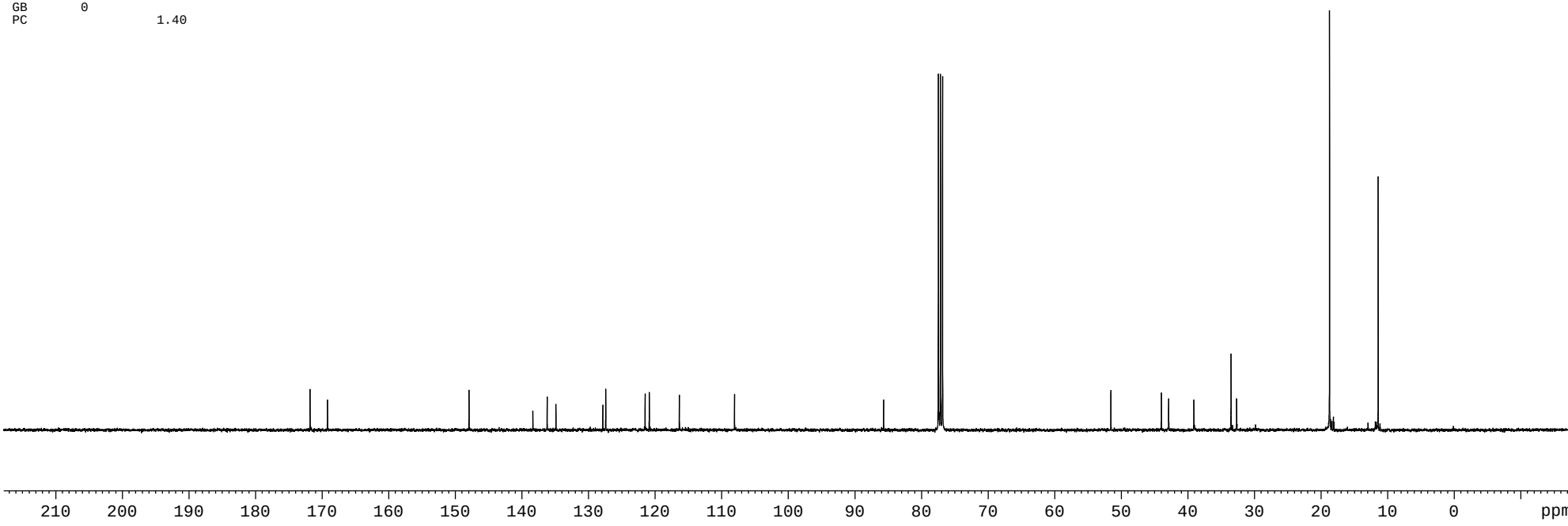

9.878
8.775
8.771
8.765
8.761
8.707
8.703
8.689
8.685
8.141
8.137
8.120
8.116
7.469
7.451
7.447
7.442
7.439
7.428
7.418
7.407
3.637
3.344
3.328
3.217
3.165
3.144
3.142
3.121
2.927
2.900
2.882
2.848
2.835
2.825
2.819
2.811
2.801
2.795
2.782
2.772
2.758
2.723
2.182
2.173
2.161
2.151
2.131
2.122
2.111
2.102
2.082
2.059
2.044
1.565
0.976
0.965
0.950
0.940
0.926
Current Data Parameters
NAME 44
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161029
Time 12.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 89.81
DW 62.400 usec
DE 6.50 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.38 usec
PLW1 19.00000000 W
F2 - Processing parameters
SI 65536
SF 400.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TIPS
O
HN
N
O
OMe
TIPS
(37)
10
9
8
7
6
5
4
3
2
1
0
ppm
0.97
1.00
1.01
1.02
3.04
3.00
1.93
1.89
2.06
0.99
1.01
41.96

```
Current Data Parameters
NAME                 41-c
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150331
Time                7.34
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   950
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                 202.1
DW                20.800 usec
DE                 6.50 usec
TE                297.5 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1
NUC1                 13C
P1                  9.85 usec
PLW1         65.00000000 W
SFO1         100.6228293 MHz
CPDPRG2
NUC2                   1H
PLW2         19.00000000 W
PLW12         0.18082000 W
PLW13         0.14647000 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127551 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

171.79
169.15
147.95
138.37
136.21
134.90
127.86
127.43
121.51
120.88
116.37
108.09
85.69
51.57
43.98
42.89
39.11
33.53
32.69
18.73
11.43



(37)

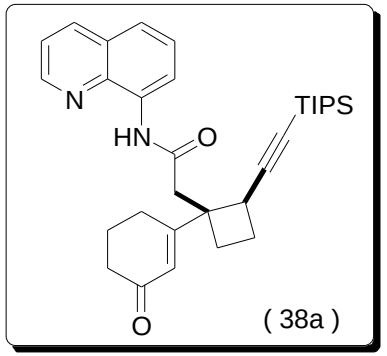


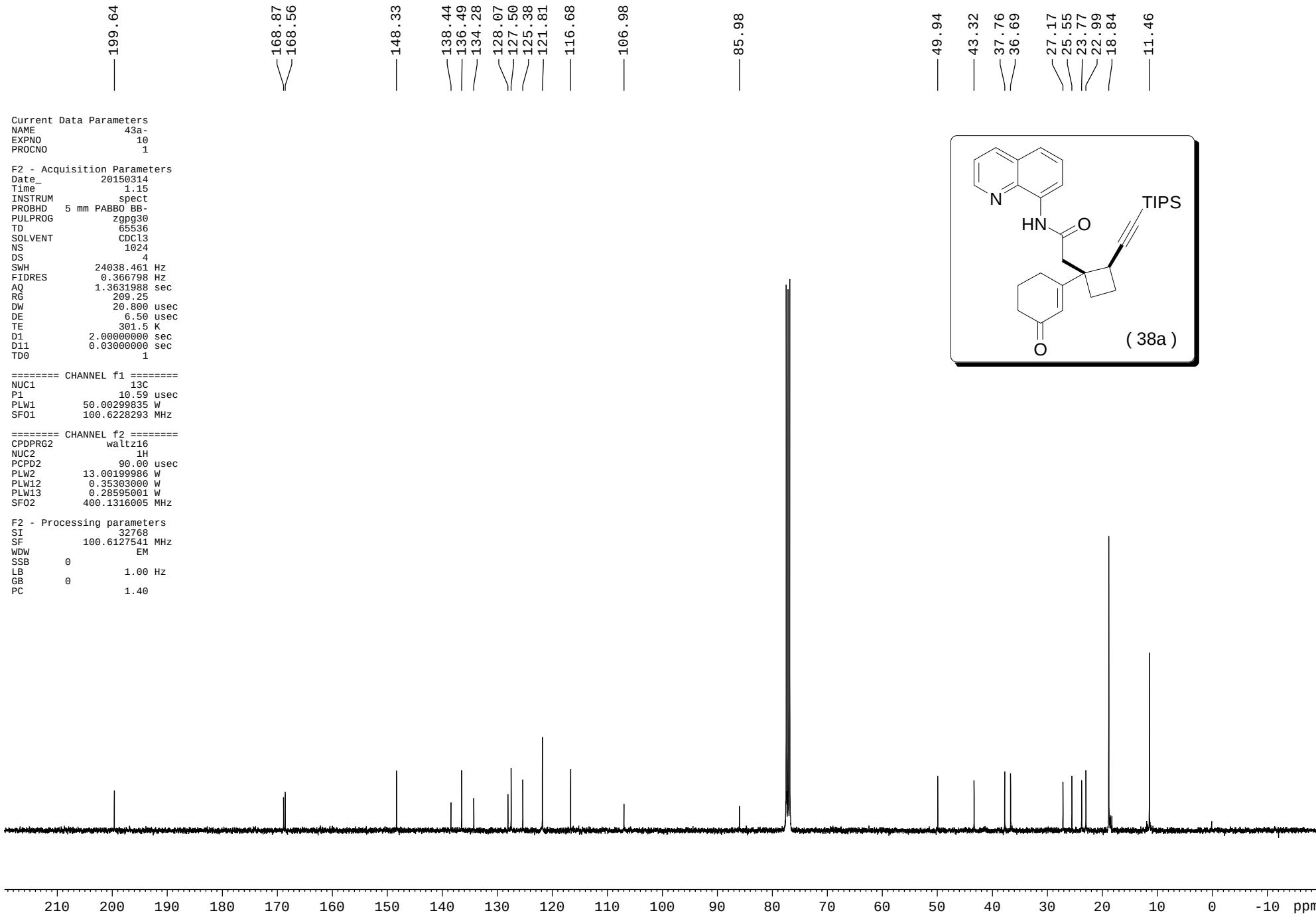
Current Data Parameters
NAME 43a-h
EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150306
Time 12.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536

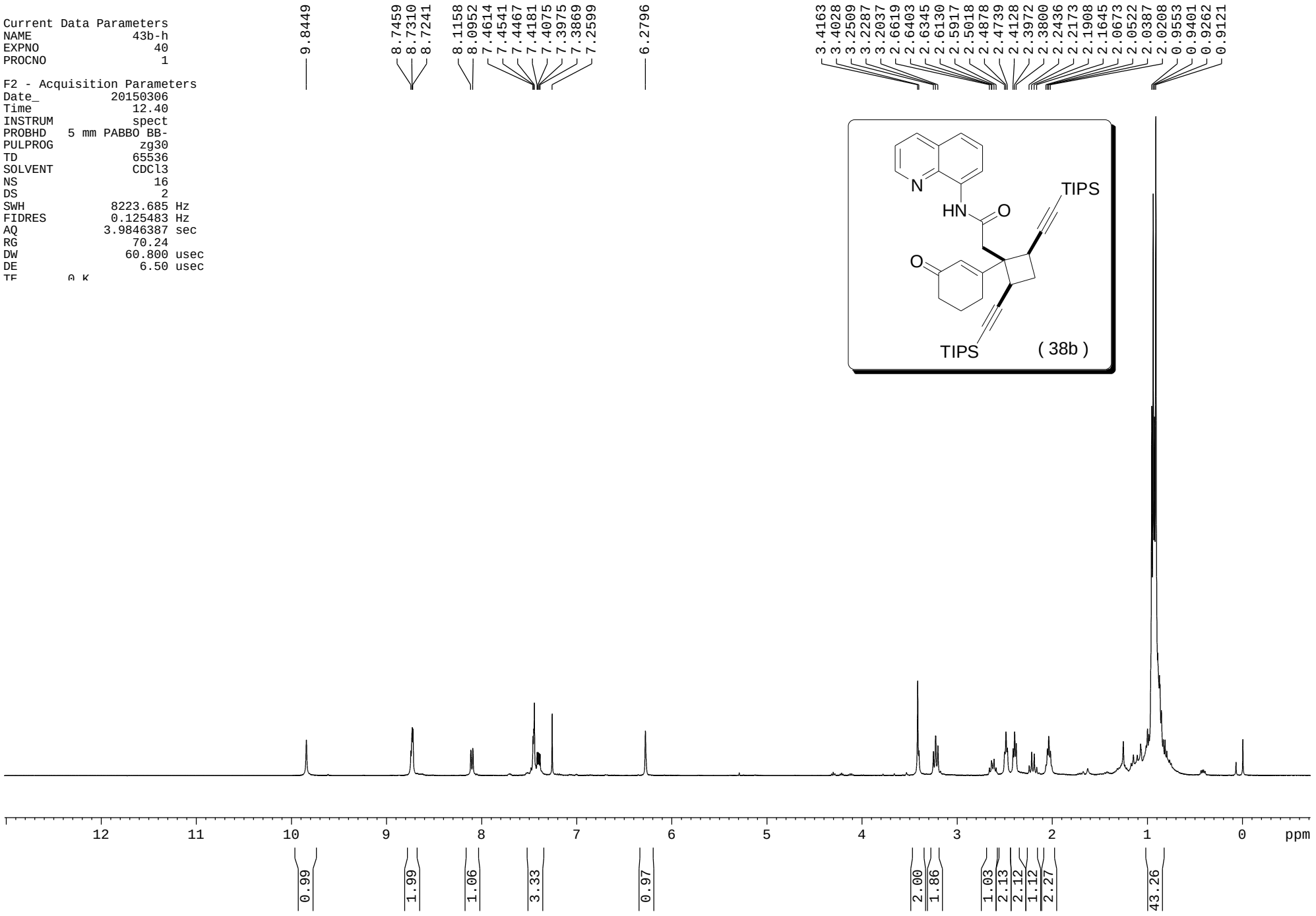
9.7134 8.7498 8.7461 8.7394 8.7356 8.6864 8.6785 8.6720 8.6641 8.1526 8.1489 8.1320 8.1283 7.5224 7.5017 7.4950 7.4872 7.4747 7.4527 7.4422 7.4321 7.4215 7.2598

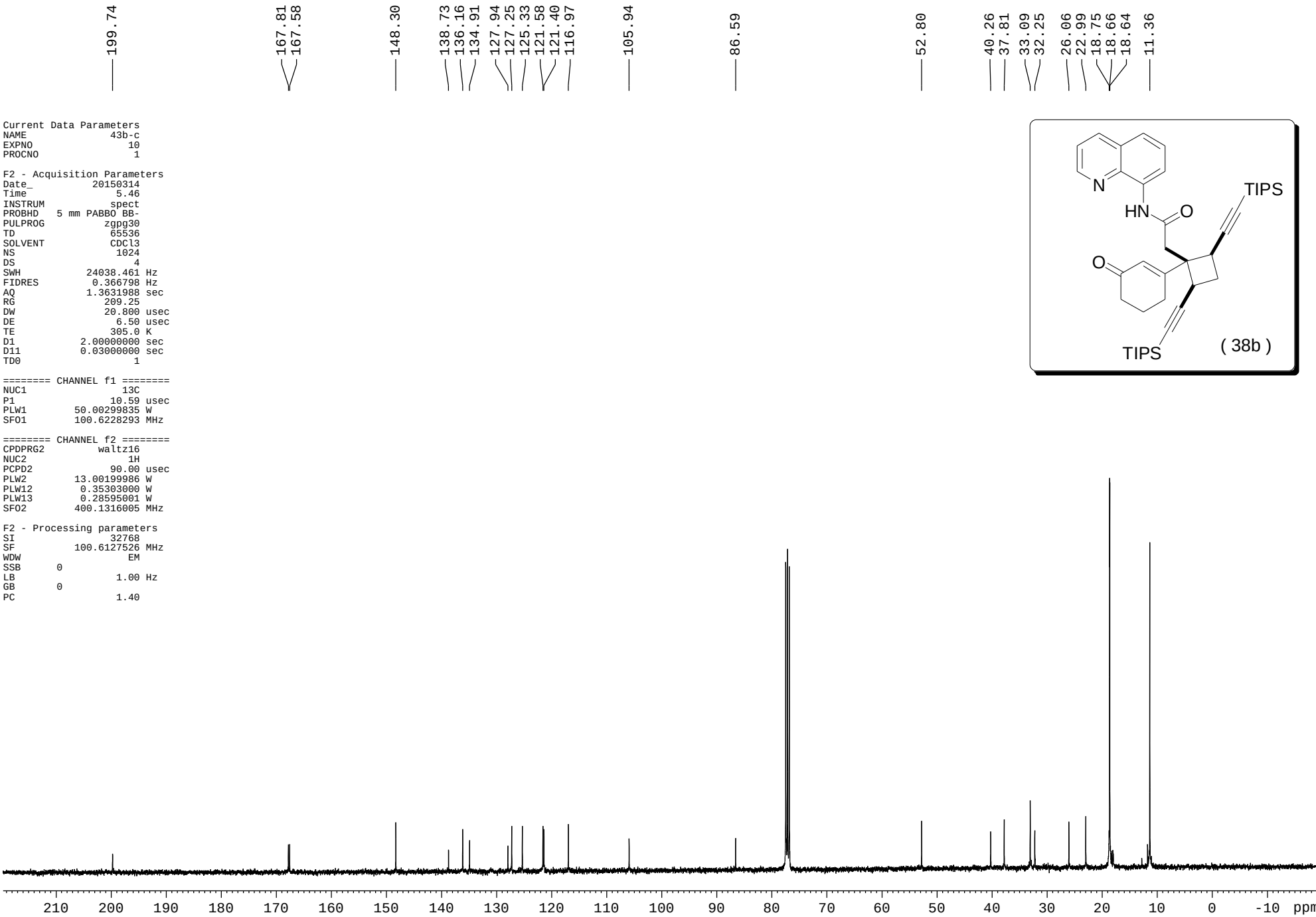
5.9559 3.2053 3.1931 3.1694 3.1501 3.1280 3.0839 3.0478 2.5584 2.5492 2.5256 2.5134 2.5075 2.4960 2.4734 2.4608 2.4483 2.4299 2.4159 2.4031 2.3917 2.3704 2.3532 2.3376 2.3019 2.2688 2.2596 2.2463 2.2296 2.2208 2.1993 2.1702 2.1205 2.1059 2.0873 2.0732 2.0600 2.0452 1.9877 1.9681 1.9551 1.9488 1.9368

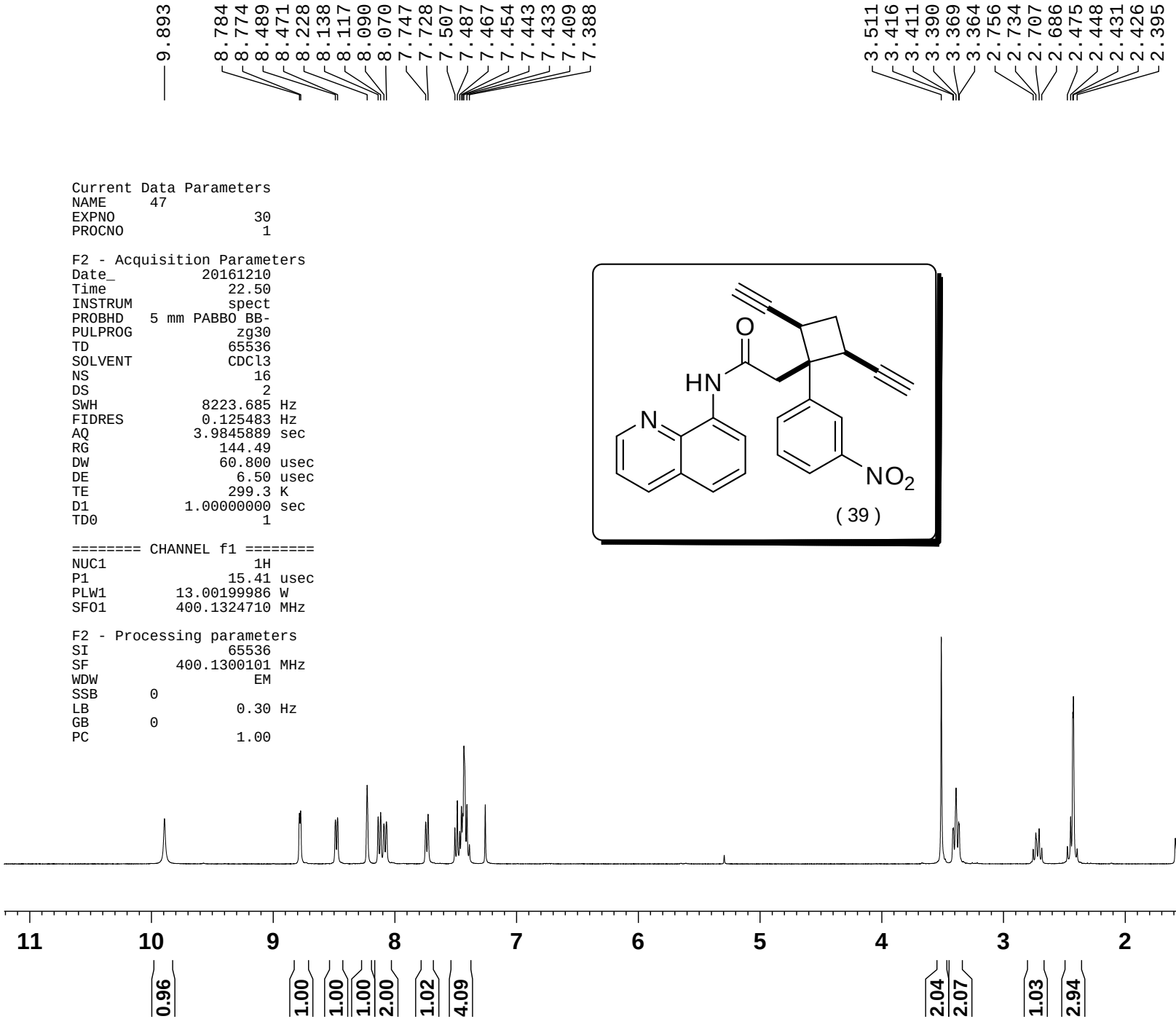


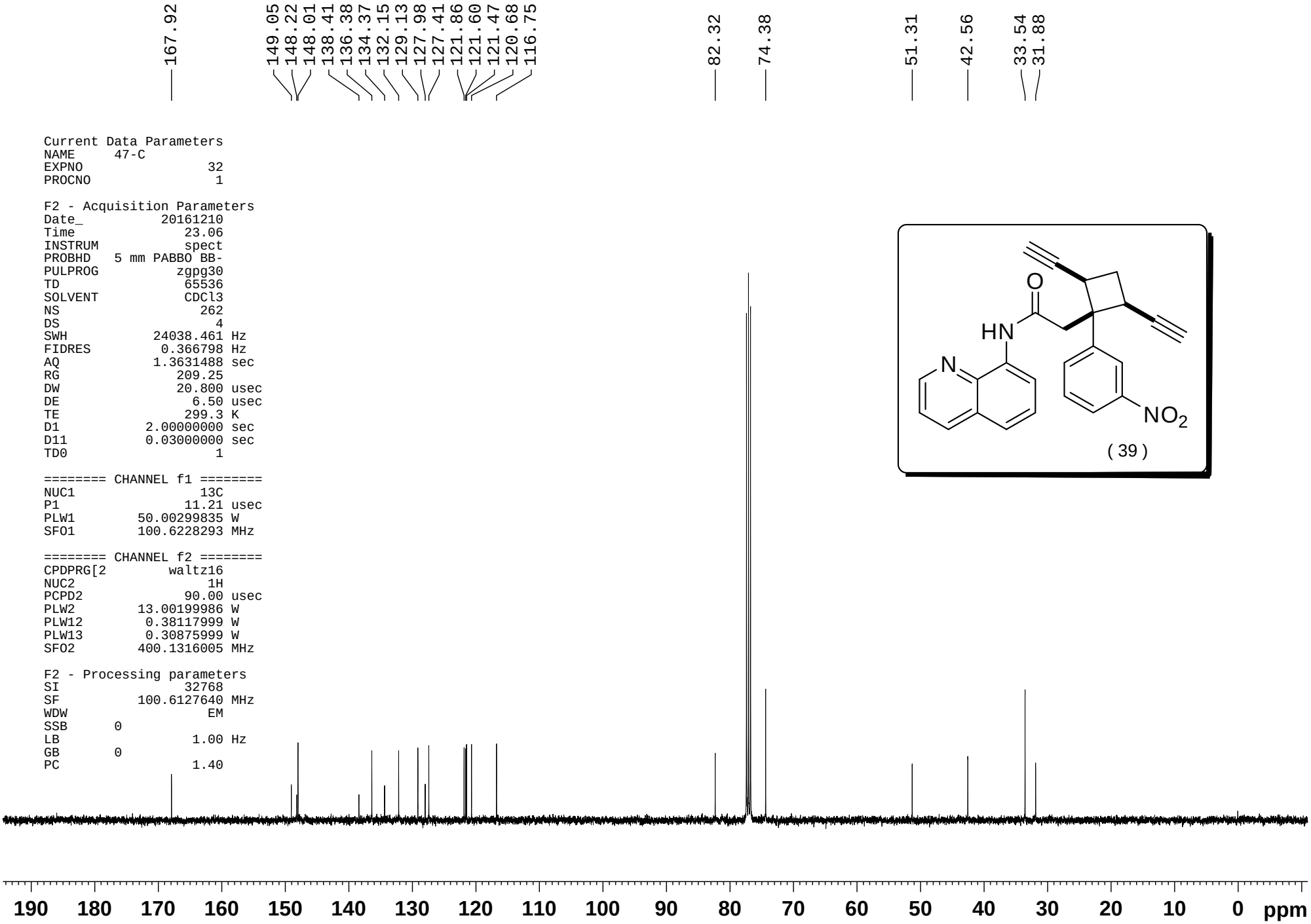



```
Current Data Parameters
NAME                  43a-
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150314
Time                 1.15
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                 209.25
DW                 20.800 usec
DE                  6.50 usec
TE                 301.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.59 usec
PLW1        50.00299835 W
SFO1        100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2               90.00 usec
PLW2        13.00199986 W
PLW12        0.35303000 W
PLW13        0.28595001 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127541 MHz
WDW                   EM
SSB      0
LB                 1.00 Hz
GB       0
PC                 1.40
```


Current Data Parameters
NAME 43b-h
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150306
Time 12.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 70.24
DW 60.800 usec
DE 6.50 usec
9.8449
8.7459
8.7310
8.7241
8.1158
8.0952
7.4614
7.4541
7.4467
7.4181
7.4075
7.3975
7.3869
7.2599
6.2796
3.4163
3.4028
3.2509
3.2287
3.2037
2.6619
2.6403
2.6345
2.6130
2.5917
2.5018
2.4878
2.4739
2.4128
2.3972
2.3800
2.2436
2.2173
2.1908
2.1645
2.0673
2.0522
2.0387
2.0208
0.9553
0.9401
0.9262
0.9121
N
HN
O
O
TIPS
TIPS
(38b)
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.99
1.99
1.06
3.33
0.97
2.00
1.86
1.03
2.13
2.12
1.12
2.27
43.26

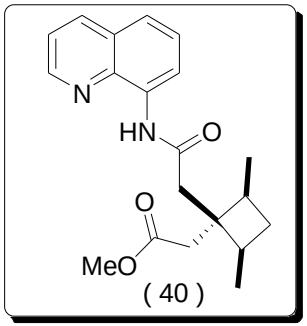

199.74
167.81
167.58
148.30
138.73
136.16
134.91
127.94
127.25
125.33
121.58
121.40
116.97
105.94
86.59
52.80
40.26
37.81
33.09
32.25
26.06
22.99
18.75
18.66
18.64
11.36
Current Data Parameters
NAME 43b-c
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150314
Time 5.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 209.25
DW 20.800 usec
DE 6.50 usec
TE 305.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.59 usec
PLW1 50.00299835 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 13.00199986 W
PLW12 0.35303000 W
PLW13 0.28595001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127526 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
HN
O
O
TIPS
TIPS
(38b)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm


9.893
8.784
8.774
8.489
8.471
8.228
8.138
8.117
8.090
8.070
7.747
7.728
7.507
7.487
7.467
7.454
7.443
7.433
7.409
7.388
3.511
3.416
3.411
3.390
3.369
3.364
2.756
2.734
2.707
2.686
2.475
2.448
2.431
2.426
2.395
Current Data Parameters
NAME 47
EXPNO 30
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161210
Time 22.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 144.49
DW 60.800 usec
DE 6.50 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.41 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300101 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
HN
N
NO2
(39)
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.96
1.00
1.00
1.00
2.00
1.02
4.09
2.04
2.07
1.03
2.94

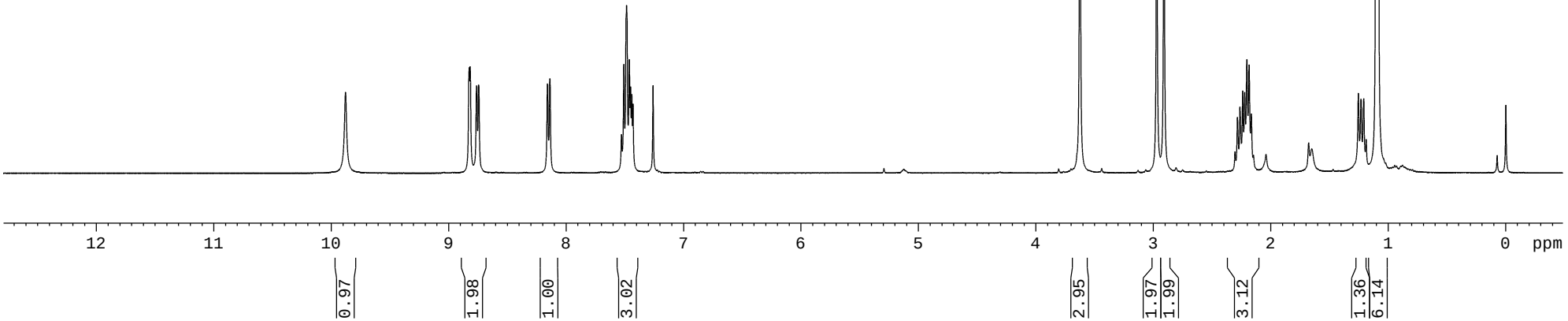

167.92
149.05
148.22
148.01
138.41
136.38
134.37
132.15
129.13
127.98
127.41
121.86
121.60
121.47
120.68
116.75
82.32
74.38
51.31
42.56
33.54
31.88
Current Data Parameters
NAME 47-C
EXPNO 32
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161210
Time 23.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 262
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 209.25
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.21 usec
PLW1 50.00299835 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 13.00199986 W
PLW12 0.38117999 W
PLW13 0.30875999 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127640 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
HN
N
NO2
(39)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

```
Current Data Parameters
NAME                 40-h
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150512
Time               21.37
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894966 sec
RG                 60.76
DW                62.400 usec
DE                 6.50 usec
TE                297.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                1H
P1                 8.78 usec
PLW1         19.00000000 W

F2 - Processing parameters
SI                 65536
SF           400.1300096 MHz
WDW                   EM
SSB      0
LB                 0.30 Hz
GB       0
PC                 1.00
```

9.8791
8.8256 8.8184 8.7616 8.7455
8.1583 8.1390 7.5290 7.5087 7.4855 7.4619 7.4514 7.4412 7.4308 7.2601
3.6248
2.9707 2.9090 2.2834 2.2624 2.2383 2.2214 2.2029 2.1840 2.1651
1.2538 1.2311 1.2075 1.1871 1.1024 1.0855

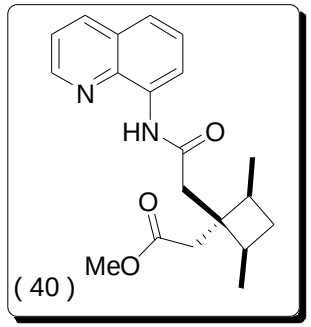


(40)

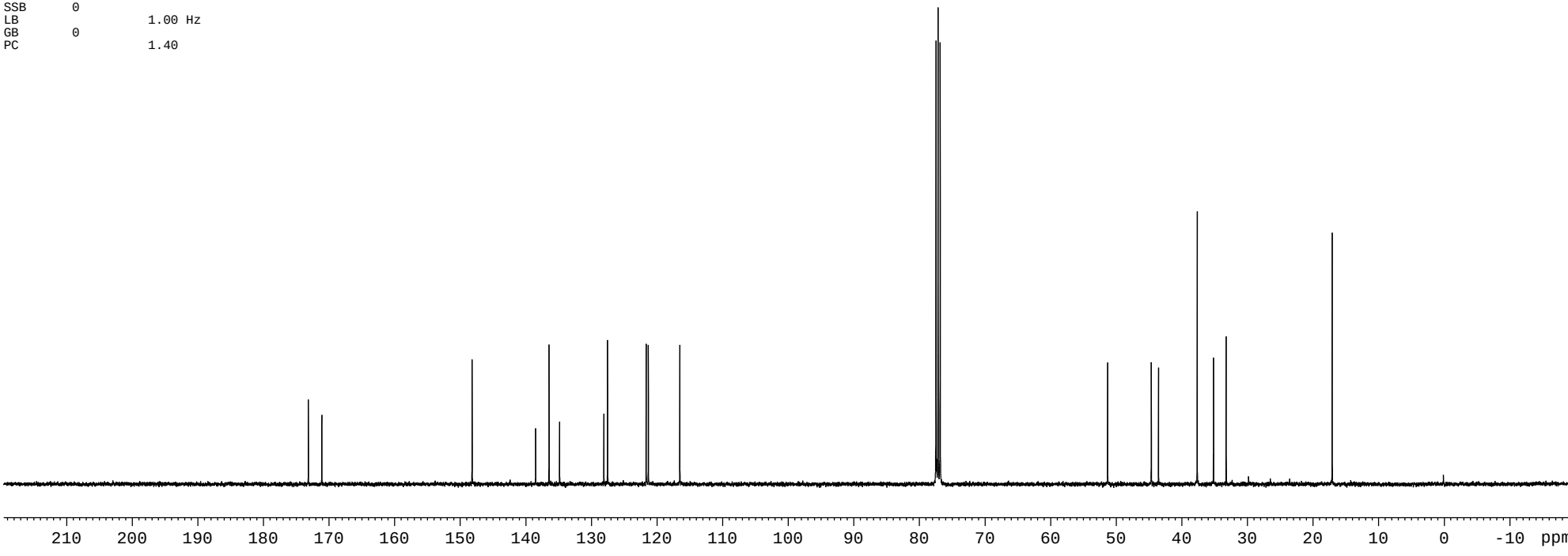


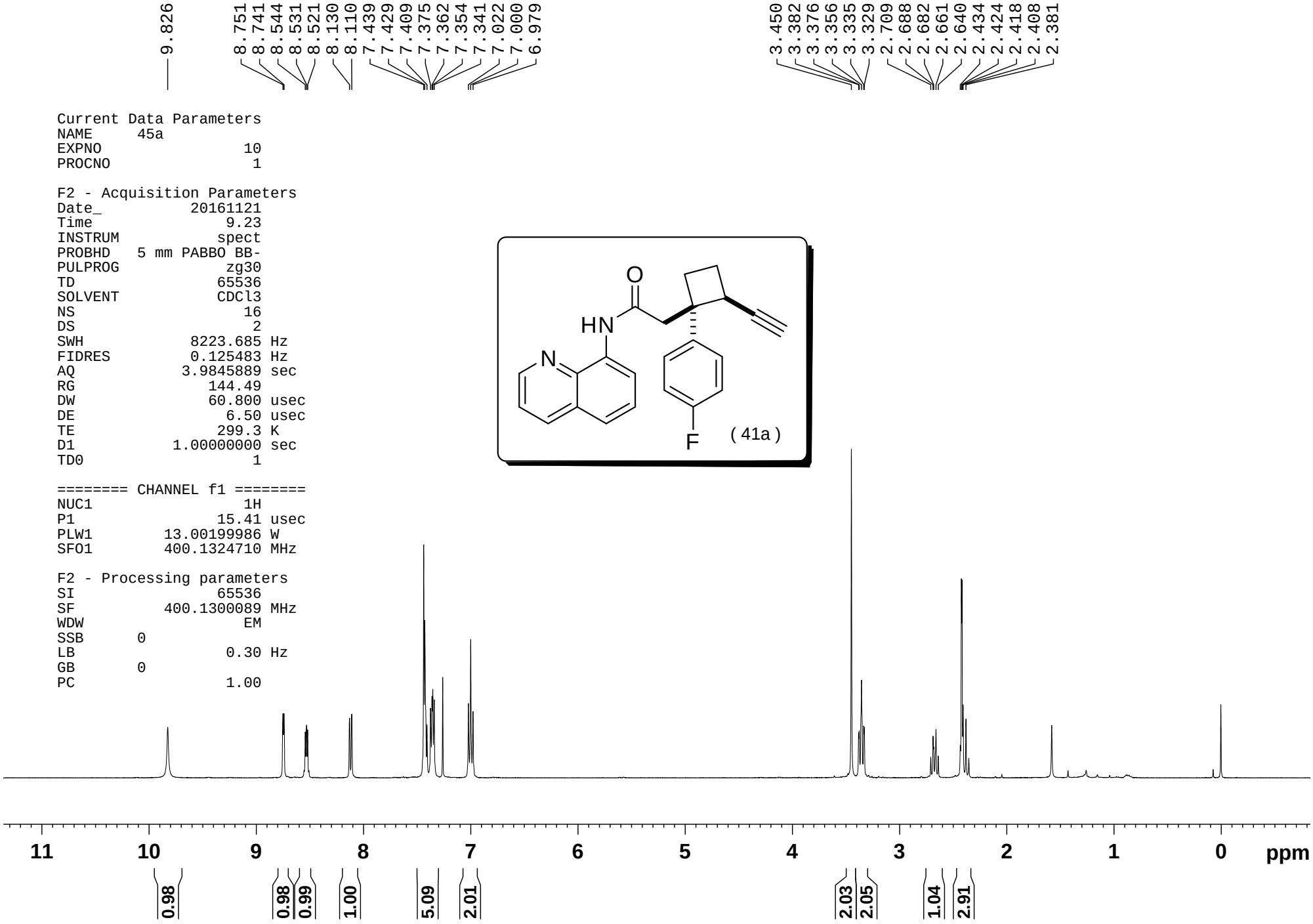
```
Current Data Parameters
NAME                 40-c
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_           20150515
Time                6.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                 202.1
DW                20.800 usec
DE                 6.50 usec
TE                297.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1
NUC1                13C
P1                  9.85 usec
PLW1         65.00000000 W
SFO1        100.6228293 MHz
CPDPRG2
NUC2                   1H
PLW2         19.00000000 W
PLW12         0.18082000 W
PLW13         0.14647000 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127561 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

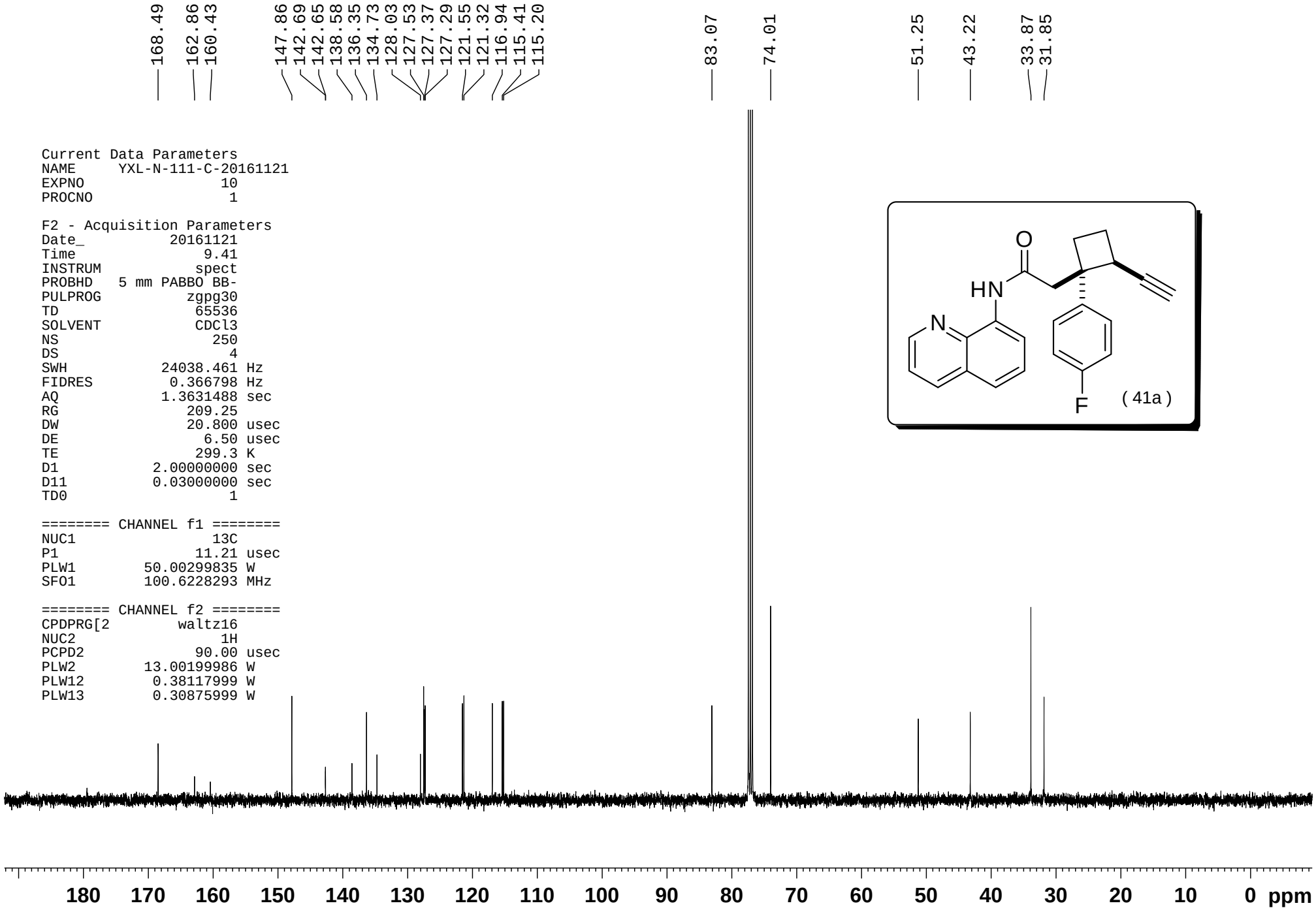
173.16
171.11
148.20
138.52
136.47
134.86
128.11
127.54
121.67
121.33
116.53
77.48
77.16
76.84
51.32
44.66
43.56
37.66
35.18
33.25
17.08



(40)



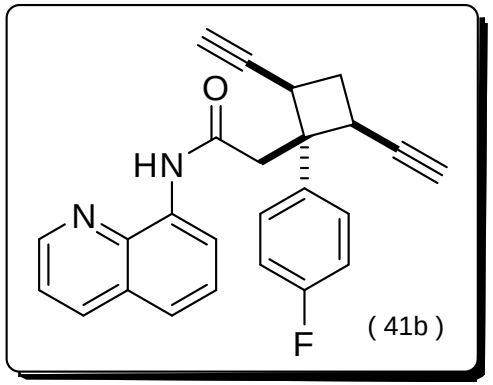

9.826
8.751
8.741
8.544
8.531
8.521
8.130
8.110
7.439
7.429
7.409
7.375
7.362
7.354
7.341
7.022
7.000
6.979
3.450
3.382
3.376
3.356
3.335
3.329
2.709
2.688
2.682
2.661
2.640
2.434
2.424
2.418
2.408
2.381
Current Data Parameters
NAME 45a
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161121
Time 9.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 144.49
DW 60.800 usec
DE 6.50 usec
TE 299.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.41 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
HN
N
F
(41a)
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.98
0.98
0.99
1.00
5.09
2.01
2.03
2.05
1.04
2.91


168.49
162.86
160.43
147.86
142.69
142.65
138.58
136.35
134.73
128.03
127.53
127.37
127.29
121.55
121.32
116.94
115.41
115.20
83.07
74.01
51.25
43.22
33.87
31.85
Current Data Parameters
NAME YXL-N-111-C-20161121
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161121
Time 9.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 250
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 209.25
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.21 usec
PLW1 50.00299835 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 13.00199986 W
PLW12 0.38117999 W
PLW13 0.30875999 W
O
HN
N
F
(41a)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

9.224 8.622 8.609 8.605 8.099 8.096 8.078 8.075 7.481 7.460 7.443 7.423 7.402 7.391 7.381 7.370 7.267 7.257 7.246 7.232 6.893 6.871 6.849

3.354 3.349 3.326 3.307 3.272 3.033 2.999 2.791 2.782 2.770 2.763 2.756 2.742 2.736 2.467 2.442 2.436 2.430 2.418 2.392 2.381 2.355 2.330 2.323 2.308 2.301 2.285 2.277

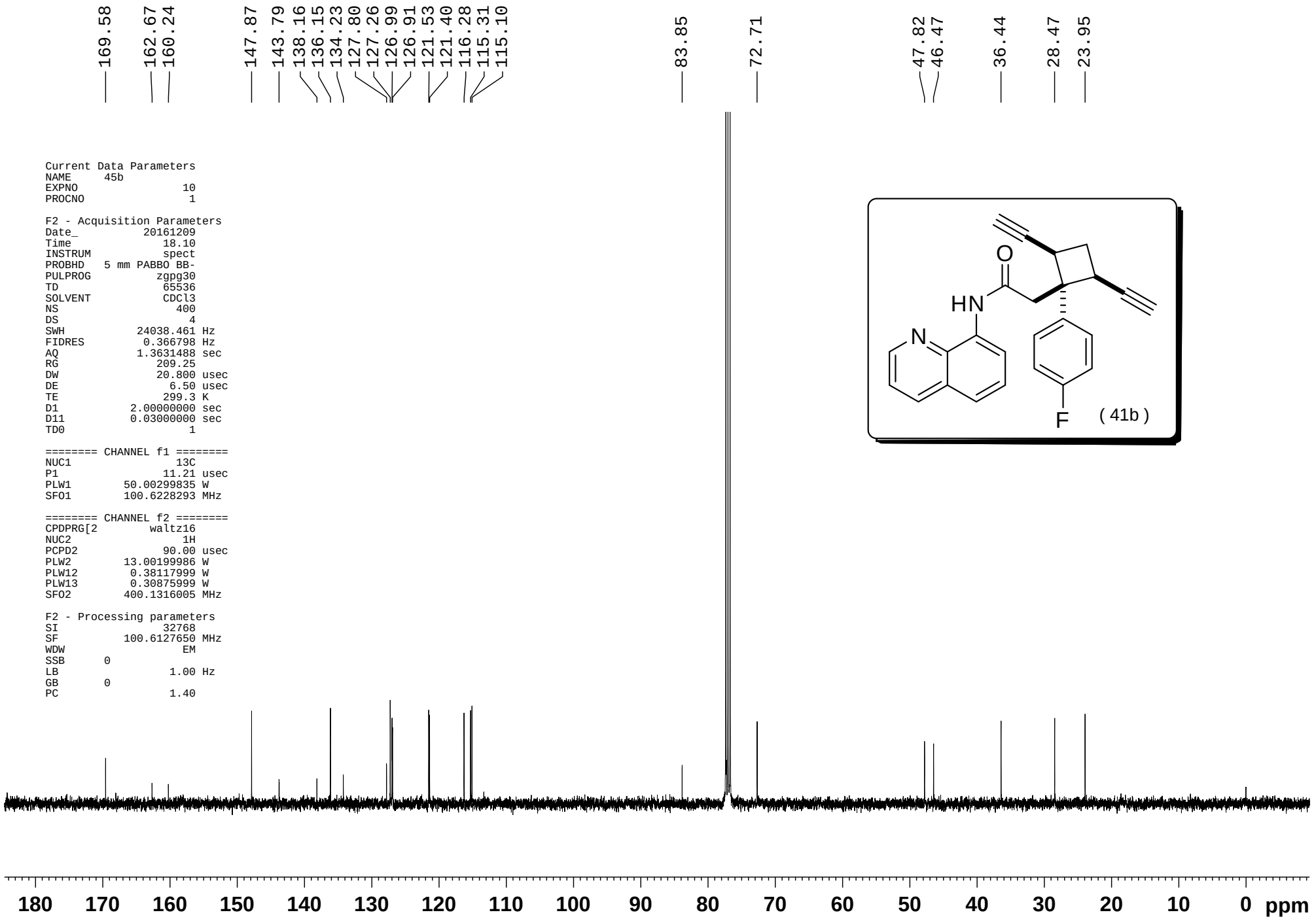
```
Current Data Parameters
NAME     45b
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20161209
Time               5.49
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ            3.9845889 sec
RG              209.25
DW              60.800 usec
DE               6.50 usec
TE              299.3 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                15.41 usec
PLW1        13.00199986 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                65536
SF          400.1300104 MHz
WDW                  EM
SSB      0
LB                0.30 Hz
GB       0
PC                1.00
```



(41b)

10 9 8 7 6 5 4 3 2 1 0 ppm

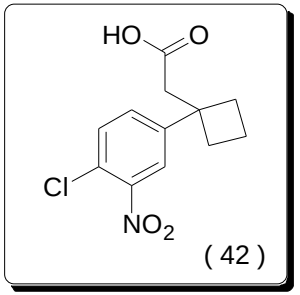
0.98 2.00 1.00 3.06 2.81 2.01 2.04 1.02 1.03 2.05 2.04


169.58
162.67
160.24
147.87
143.79
138.16
136.15
134.23
127.80
127.26
126.99
126.91
121.53
121.40
116.28
115.31
115.10
83.85
72.71
47.82
46.47
36.44
28.47
23.95
Current Data Parameters
NAME 45b
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20161209
Time 18.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 209.25
DW 20.800 usec
DE 6.50 usec
TE 299.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 11.21 usec
PLW1 50.00299835 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 13.00199986 W
PLW12 0.38117999 W
PLW13 0.30875999 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127650 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
O
HN
N
F
(41b)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

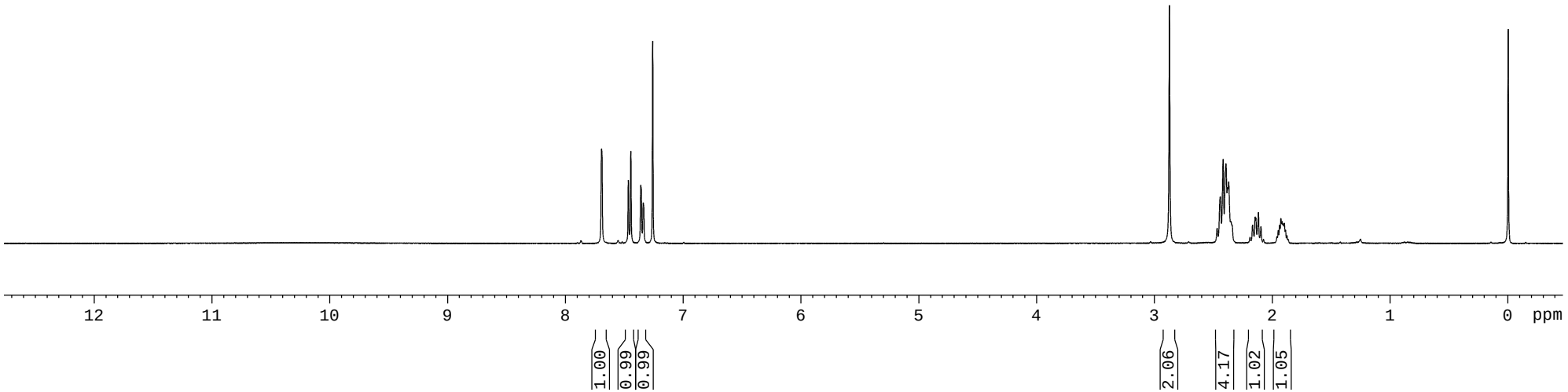
```
Current Data Paramete
NAME      YXL-Acid-H-1
EXPNO
PROCNO

F2 - Acquisition Para
Date_           201605
Time               10.
INSTRUM             spe
PROBHD   5 mm PABBO B
PULPROG             zg
TD                 655
SOLVENT            CDC
NS
DS
SWH             8012.8
FIDRES          0.1222
AQ             4.08944
RG                143.
DW                62.4
DE                  6.
TE                293
D1           1.000000
TD0

======== CHANNEL f1 =
```

7.6936 7.4662 7.4453 7.3603 7.3396

2.8727 2.4682 2.4412 2.4170 2.3932 2.3793 2.3696 2.3507 2.3414 2.1886 2.1675 2.1456 2.1392 2.1172 2.0958 1.9599 1.9492 1.9378 1.9266 1.9168 1.9085 1.8975 1.8870 1.8753



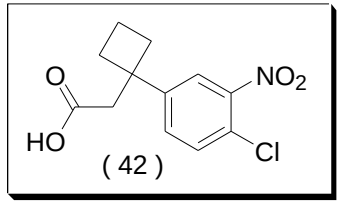
(42)



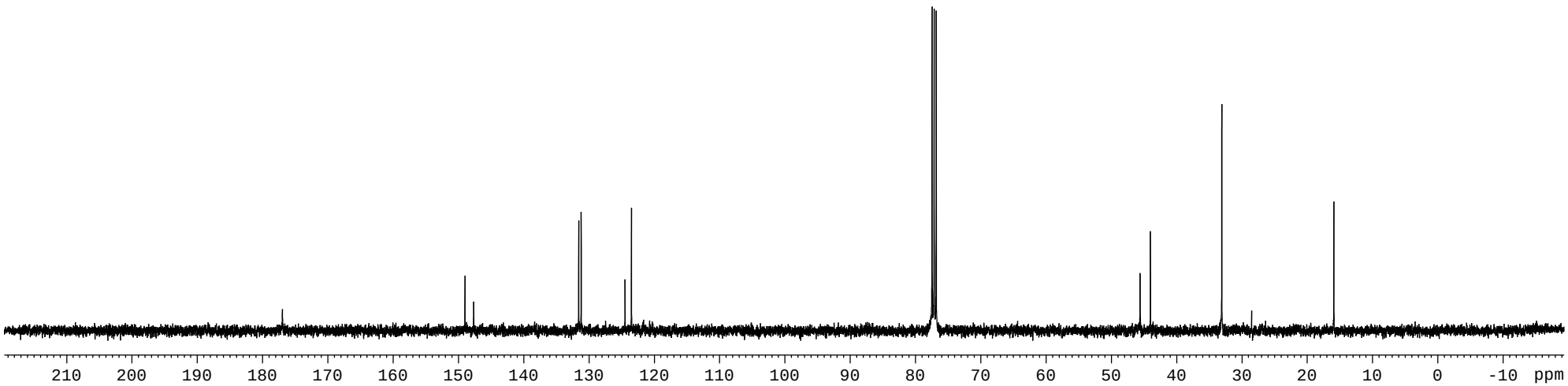
Current Data Para
NAME YXL-10-1
EXPNO
PROCNO

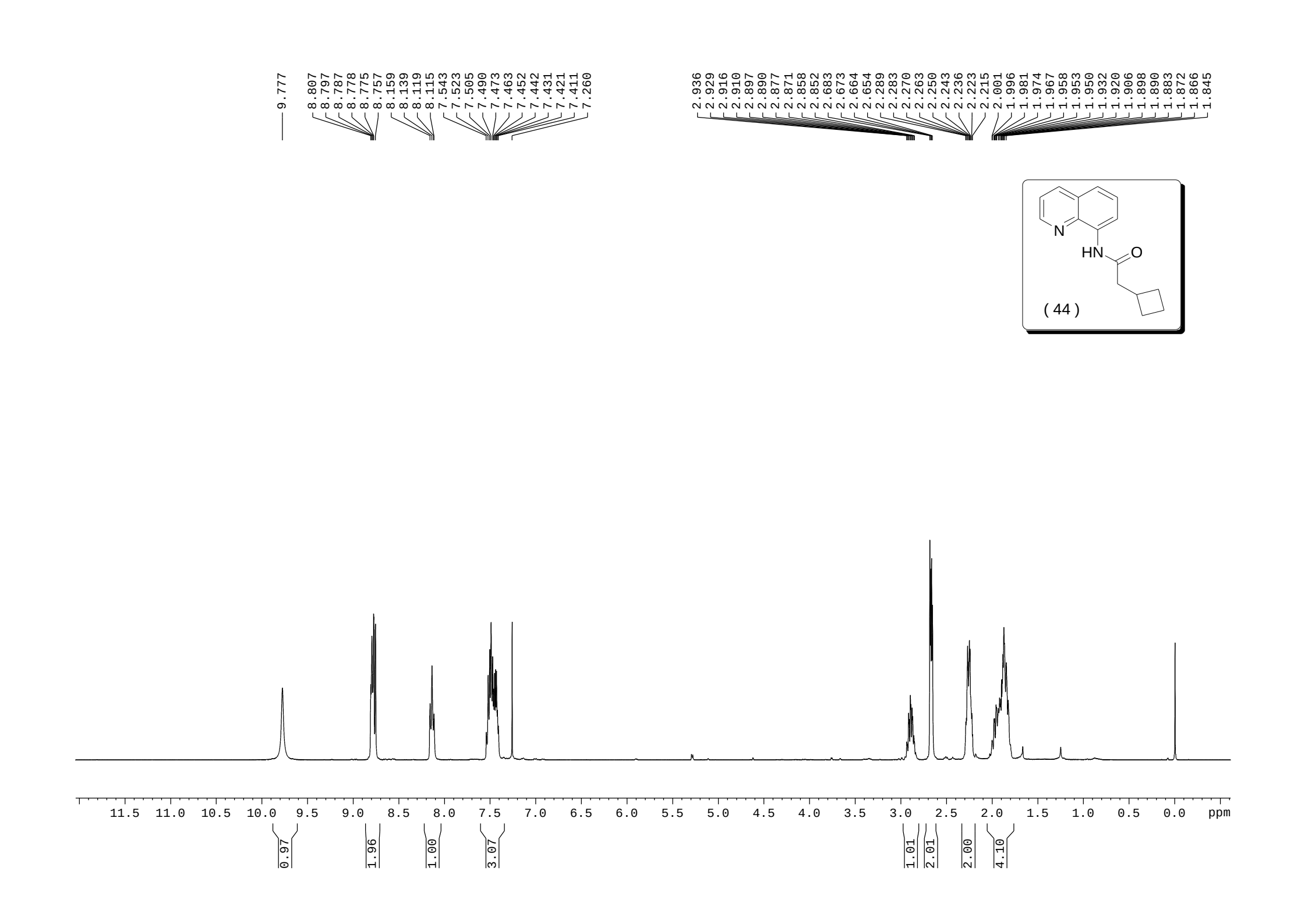
F2 - Acquisition
Date_ 2(
Time
INSTRUM
PROBHD 5 mm PAE

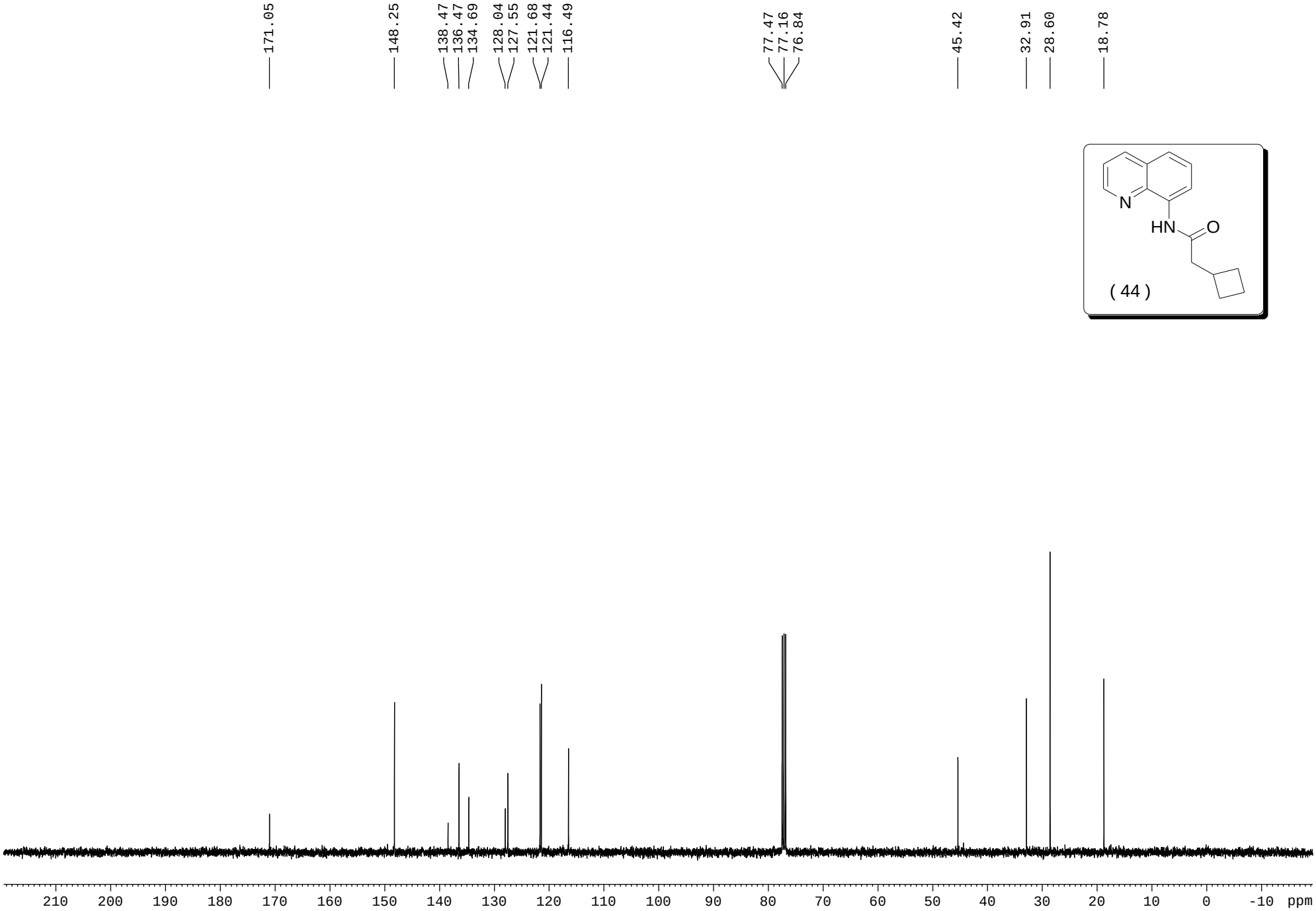
176.98
149.02
147.69
131.60
131.22
124.52
123.52
45.61
44.03
33.10
15.93

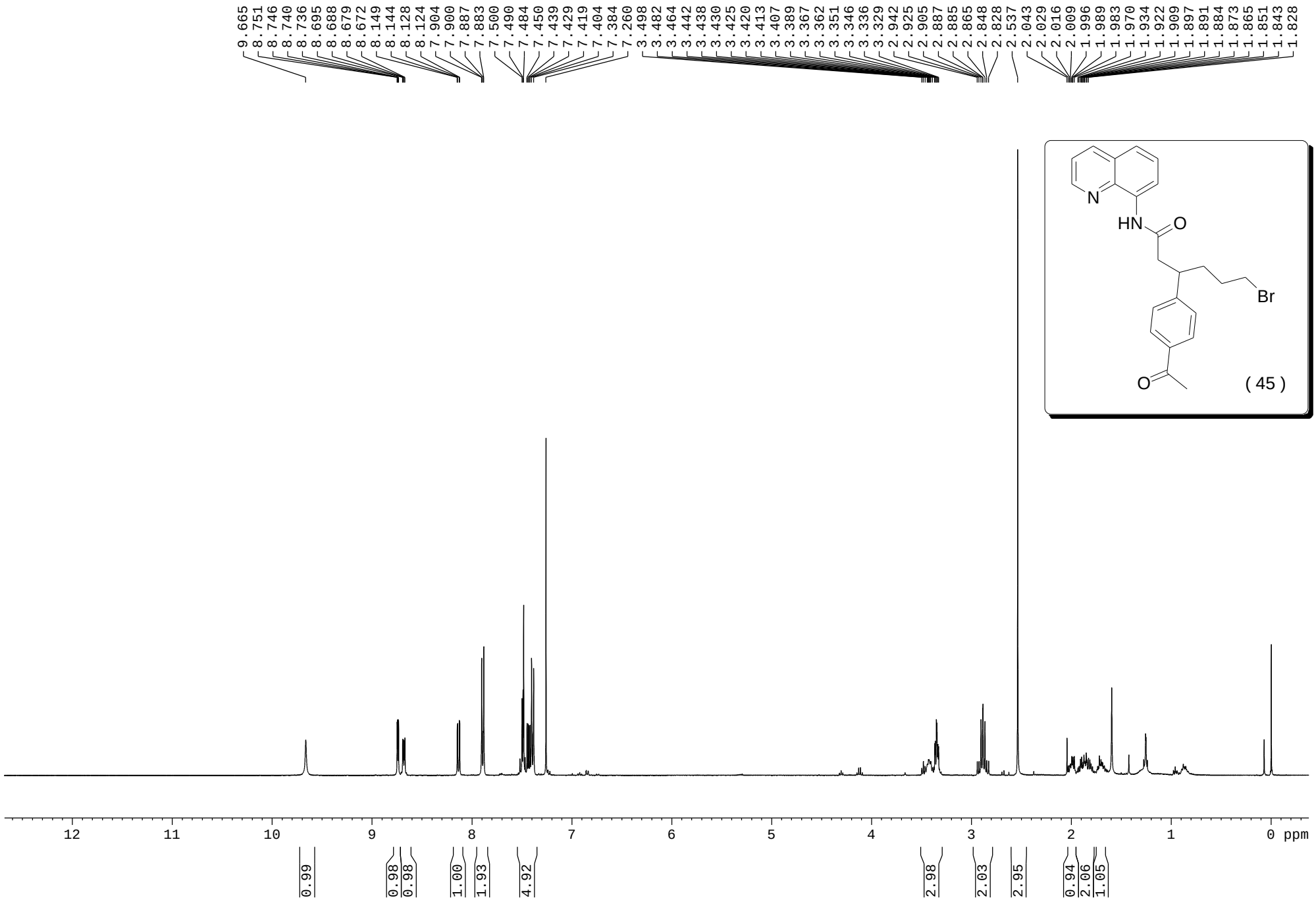


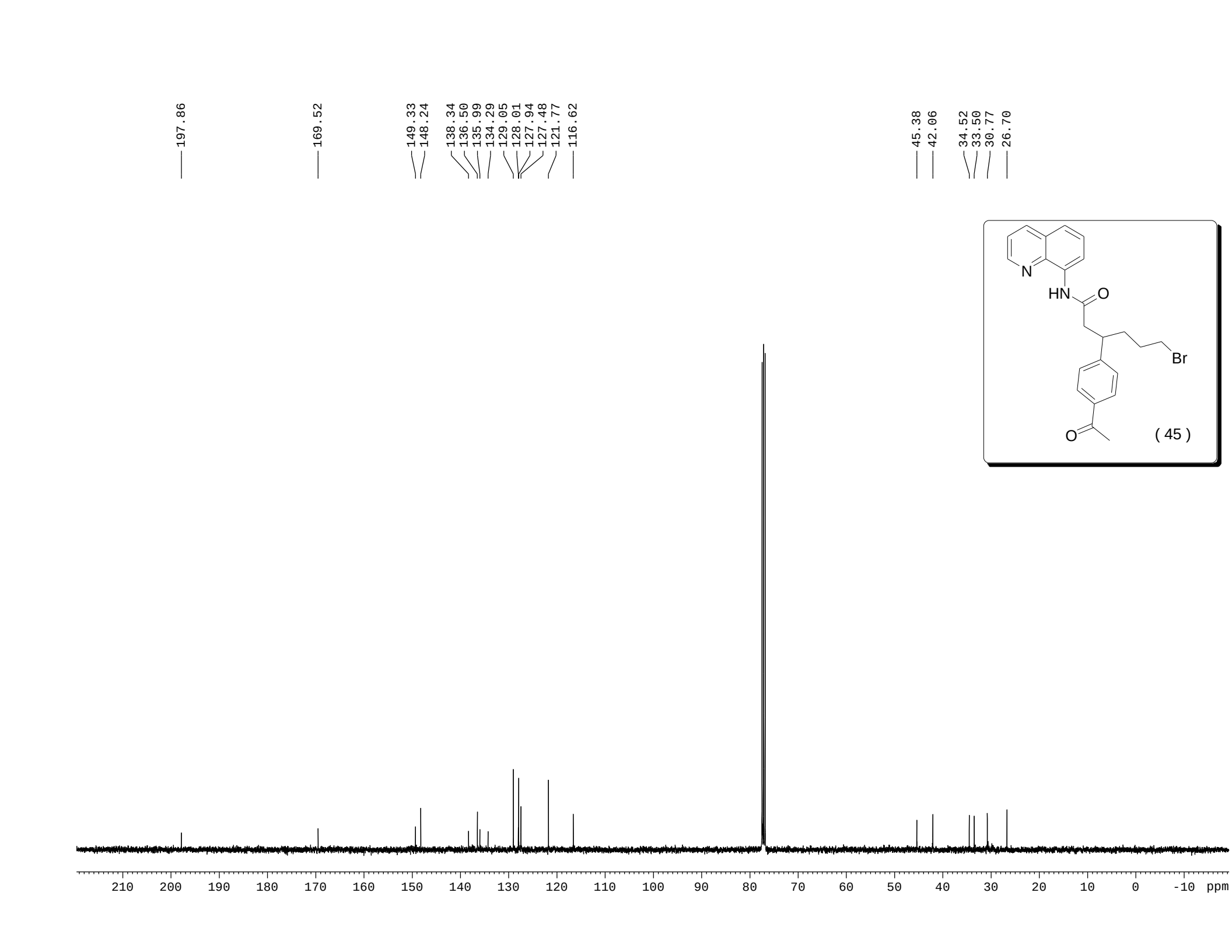
(42)




9.777
8.807
8.797
8.787
8.778
8.775
8.757
8.159
8.139
8.119
8.115
7.543
7.523
7.505
7.490
7.473
7.463
7.452
7.442
7.431
7.421
7.411
7.260
2.936
2.929
2.916
2.910
2.897
2.890
2.877
2.871
2.858
2.852
2.683
2.673
2.664
2.654
2.289
2.283
2.270
2.263
2.250
2.243
2.236
2.223
2.215
2.001
1.996
1.981
1.974
1.967
1.958
1.953
1.950
1.932
1.920
1.906
1.898
1.890
1.883
1.872
1.866
1.845
N
HN
O
(44)
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.97
1.96
1.00
3.07
1.01
2.01
2.00
4.10


171.05
148.25
138.47
136.47
134.69
128.04
127.55
121.68
121.44
116.49
77.47
77.16
76.84
45.42
32.91
28.60
18.78
N
HN
O
(44)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm


9.665
8.751
8.746
8.740
8.736
8.695
8.688
8.679
8.672
8.149
8.144
8.128
8.124
7.904
7.900
7.887
7.883
7.500
7.490
7.484
7.450
7.439
7.429
7.419
7.404
7.384
7.260
3.498
3.482
3.464
3.442
3.438
3.430
3.425
3.420
3.413
3.407
3.389
3.367
3.362
3.351
3.346
3.336
3.329
2.942
2.925
2.905
2.887
2.885
2.865
2.848
2.828
2.537
2.043
2.029
2.016
2.009
1.996
1.989
1.983
1.970
1.934
1.922
1.909
1.897
1.891
1.884
1.873
1.865
1.851
1.843
1.828
N
HN
O
Br
O
(45)
12
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.99
0.98
0.98
1.00
1.93
4.92
2.98
2.03
2.95
0.94
2.06
1.05


197.86
169.52
149.33
148.24
138.34
136.50
135.99
134.29
129.05
128.01
127.94
127.48
121.77
116.62
45.38
42.06
34.52
33.50
30.77
26.70
N
HN
O
Br
O
(45)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
ppm