Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.
This journal is © The Royal Society of Chemistry 2020

Visible-Light Induced Copper(I)-Catalyzed Oxidative Cyclization of *o*-Aminobenzamides with Methanol and Ethanol *via* HAT

*Mandapati Bhargava Reddy, Kesavan Prasanth, Ramasamy Anandhan**

Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India.

E-mail: ananthanramasamy@gmail.com; Tel: +91 044 22202881.

1. General experimental details

The ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on Bruker spectrometers 300 MHz NMR spectrometer (300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR) respectively with TMS as an internal standard. Mass spectra were recorded on Xevo G2S Q-TOF spectrometer. TLC was performed on using Merck pre-coated TLC plates (Merck 60 F_{254}) and detected under UV light. Column chromatography was carried out with silica gel (100-200 mesh). Reagents and solvents were purified as per standard procedures. Absorption spectra were recorded using Shimadzu UV-1601 UV-VIS spectrophotometer in MeOH solvent.

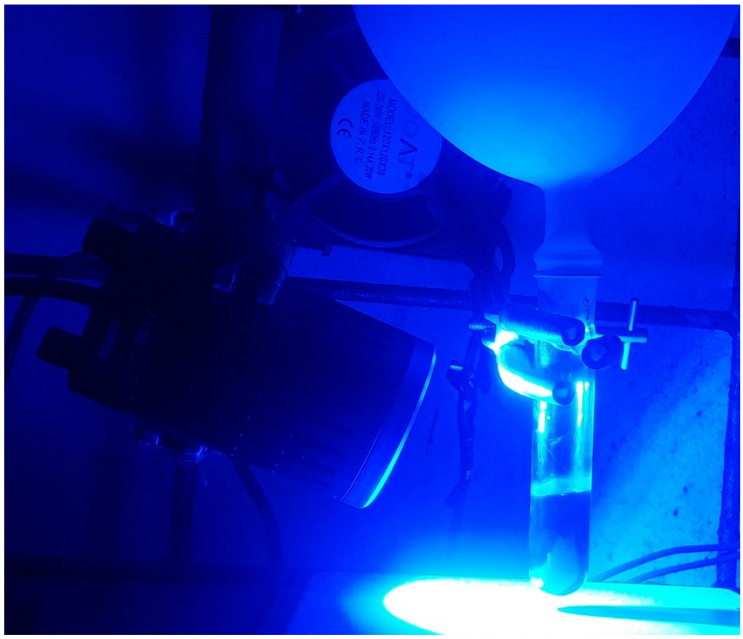


Figure I. Reaction setup for quinazolinone synthesis.

2. EPR measurements

EPR spectra were recorded at room temperature on a JEOL Model JES FA200 instrument (X band, 9.8 GHz). Under standard reaction conditions the EPR signals shown in Figure S1 is corresponding to Cu(II)OO-H. This result implies that superoxide anion radical was formed in the reaction solution. No superoxide EPR signal was observed in the absence of CuI under standard conditions (Figures S2).

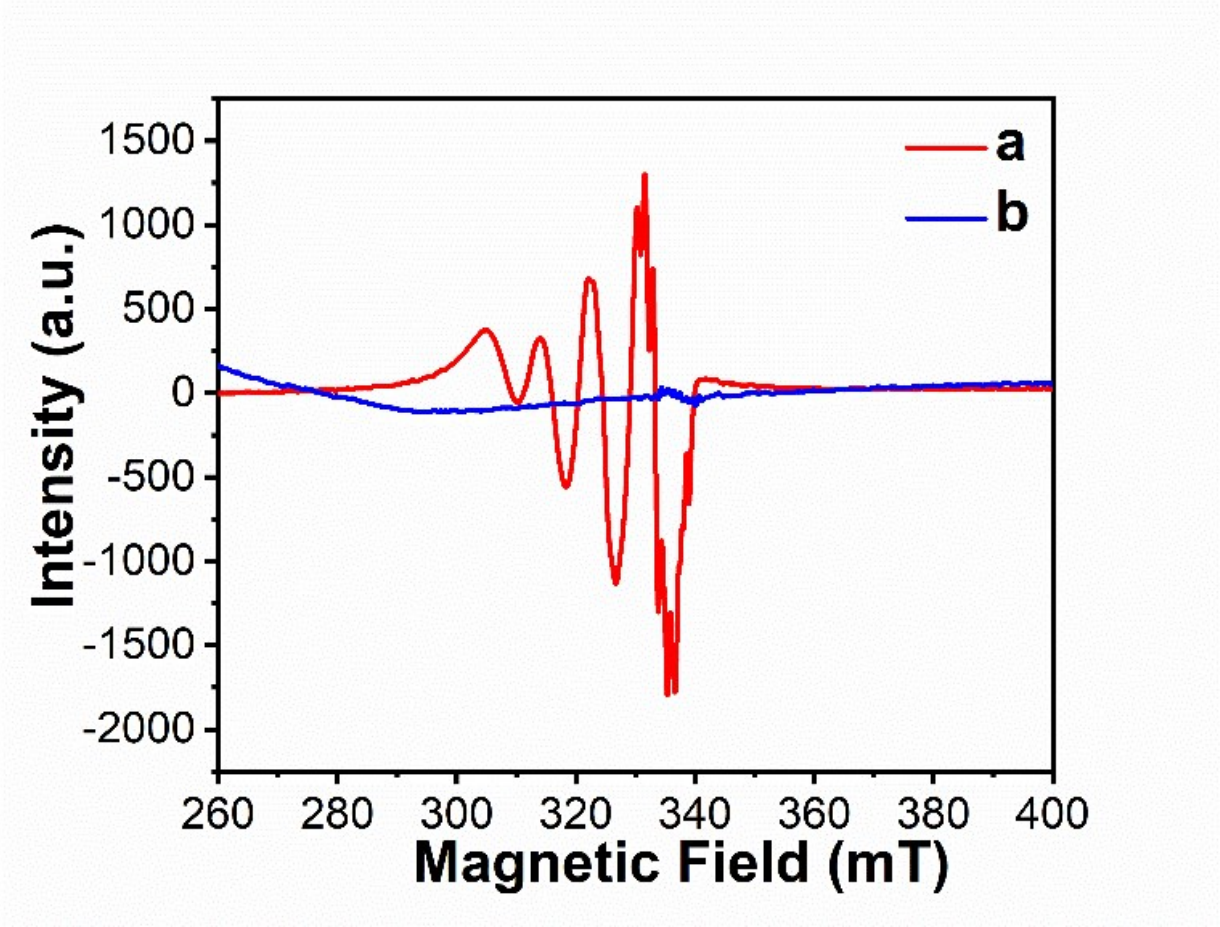
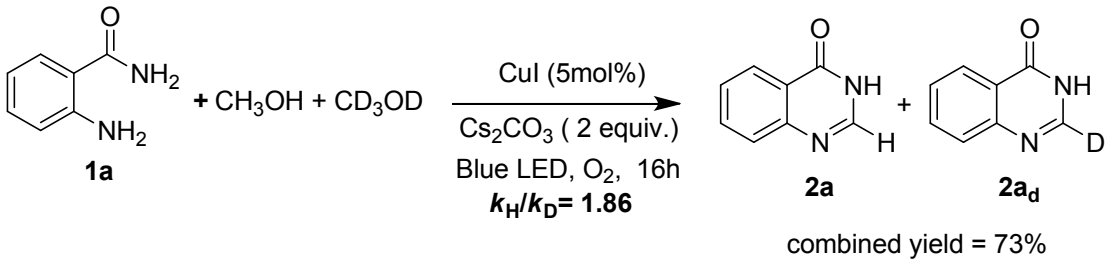
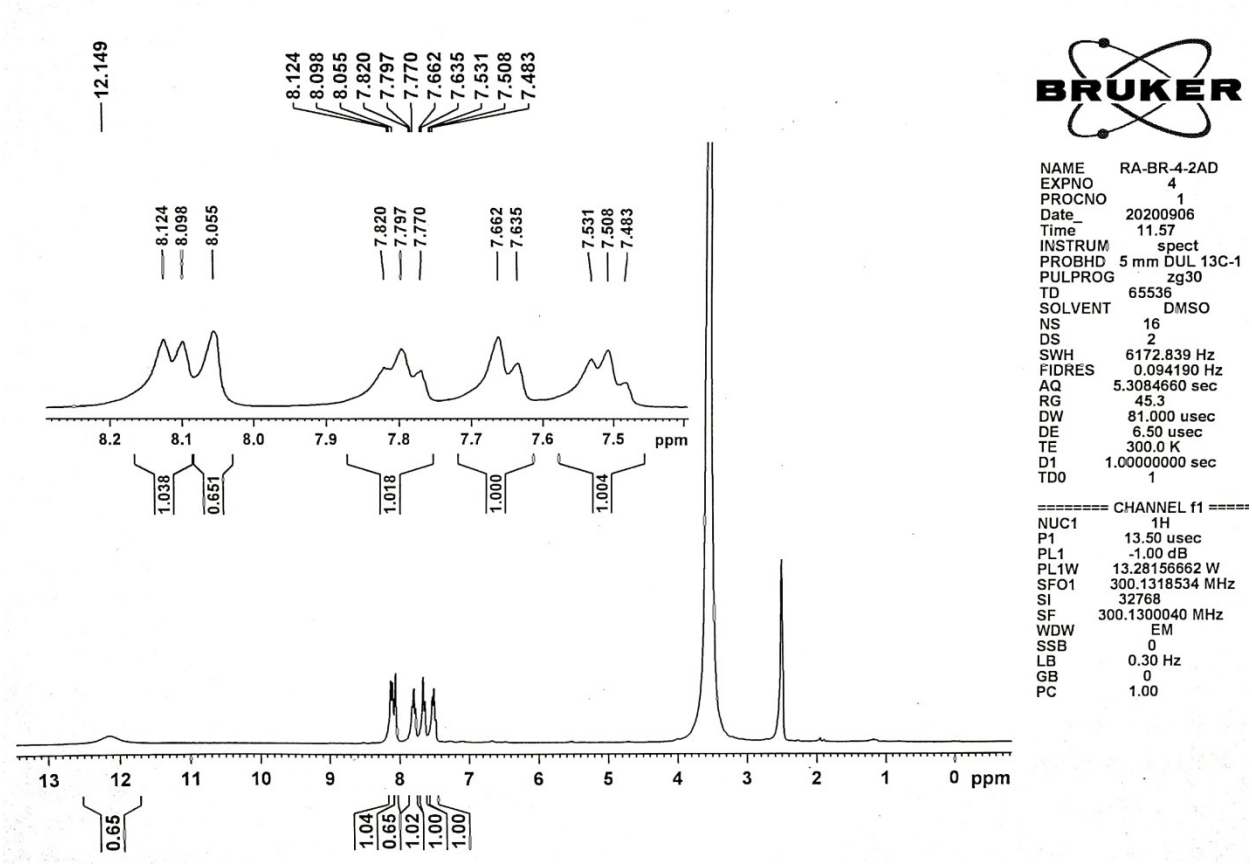


Figure S1: EPR spectra of the reaction mixture under blue LED: (a) **1a** (0.07 mmol), CuI (5 mol%) and Cs_2CO_3 (0.14 mmol) in methanol (1 mL). The reaction mixture was irradiated with blue LED at room temperature under an oxygen atmosphere (1 atm) for 30 minutes; (b) in the absence of CuI.

3. Kinetic isotopic effect



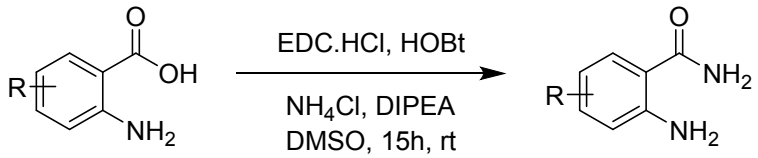
Reaction tube was charged with 2-aminobenzamide **1a** (0.36 mmol), Cs_2CO_3 (0.72 mmol) and CuI (5 mol%) in a mixture of CH_3OH and CD_3OD (1:1; 2 mL). The reaction mixture was stirred under blue light for 16 hours. After the reaction completion, mixture was passed through celite and solvent was evaporated under reduced pressure. The crude was purified by column chromatography using EtOAc/Hexane (4: 6). The kinetic isotopic effect was determined by ^{1}H NMR measurement (See the following ^{1}H NMR spectrum).

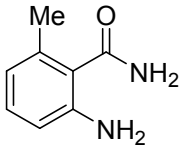


4. Experimental procedures and characterizations of substrates

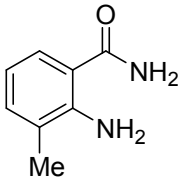
4.1 Synthesis of substrates 2-aminobenzamides[1]

A mixture of substituted acid (1 equiv.), EDC•HCl (1.5 equiv), HOBt (1.65 equiv.), NH_4Cl (3.25 equiv.) and DIPEA (6.5 equiv.) in DMSO was stirred at room temperature for 15 h. The mixture was extracted with EtOAc three times, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude was purified by column chromatography using EtOAc/Hexane (4:6) as eluent to furnish the 2-aminobenzamides.

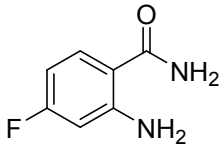




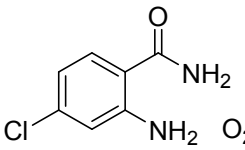
1b, 50%
White solid



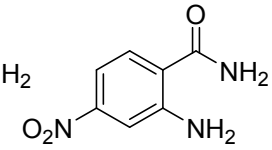
1c, 55%
White solid



1d, 68%
White solid



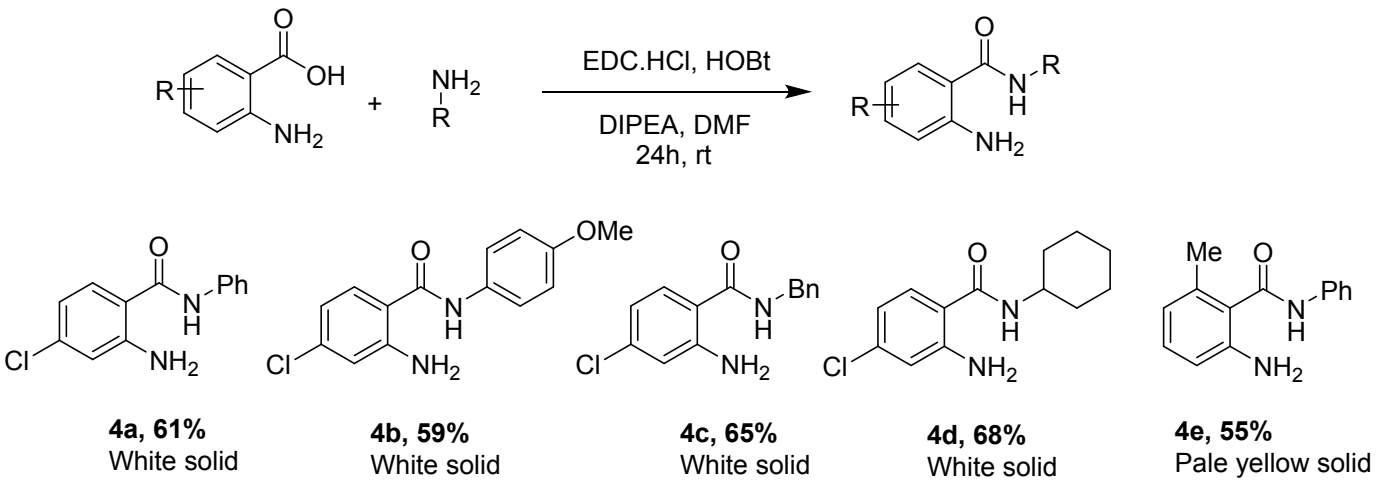
1e, 71%
White solid



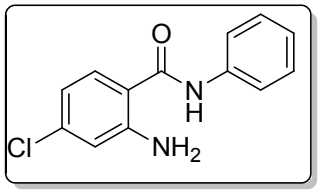
1g, 85%
Yellow solid

4.2 Synthesis of substrates 2-amino-N-phenylbenzamide[1]

A mixture of substituted acid (1 equiv.), EDC•HCl (1.2 equiv), HOBt (1.25 equiv.), aniline (1 equiv.) and DIPEA (2.5 equiv.) in DMF was stirred at room temperature for 24 h. The mixture was extracted with EtOAc three times, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography using EtOAc/Hexane (1:9) as eluent to furnish the 2-aminobenzamides.



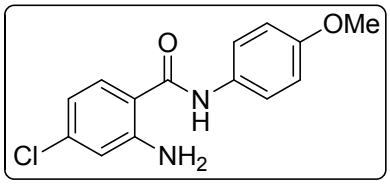
2-amino-4-chloro-N-phenylbenzamide 4a



Yield 61%, white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 5.65 (br s, 2H), 6.67-6.73 (m, 2H), 7.18 (t, *J*= 7.5 Hz, 1H), 7.36-7.42 (m, 3H), 7.58 (d, *J*= 7.8 Hz, 2H), 7.71 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 114.4, 116.8, 116.9, 120.6, 124.7, 128.3, 129.1, 137.5, 138.6, 150.0, 166.8.

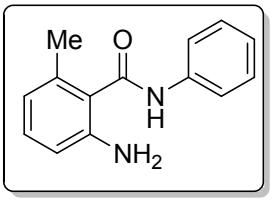
2-amino-4-chloro-N-(4-methoxyphenyl)benzamide 4b



Yield 59%, white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 3.82 (s, 3H), 5.62 (br s, 2H), 6.65-6.71 (m, 2H), 6.92 (d, *J*= 8.7 Hz, 2H), 7.38 (d, *J*= 8.4 Hz, 1H), 7.45 (d, *J*= 8.7 Hz, 2H), 7.61 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 55.5, 114.4, 114.7, 116.8, 116.9, 122.7, 128.3, 130.7, 138.4, 150.0, 157.0, 166.8.

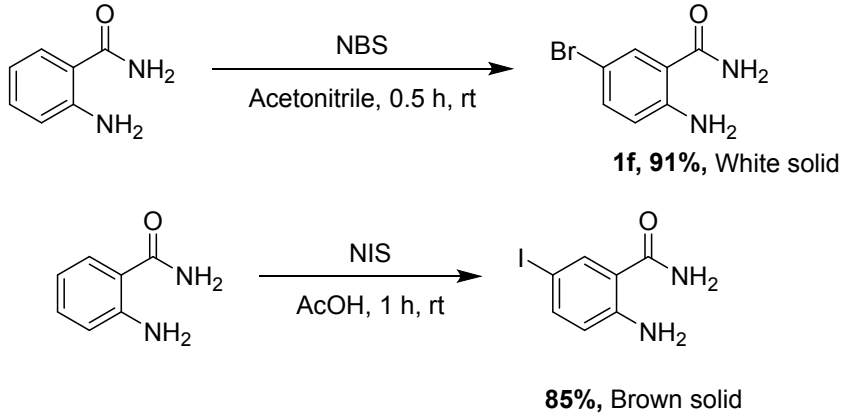
2-amino-6-methyl-N-phenylbenzamide 4e



Yield 55%, pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 2.40 (s, 3H), 4.16 (br s, 2H), 6.56 (d, *J*= 7.8 Hz , 1H), 6.63 (d, *J*= 7.5 Hz , 1H), 7.09 (t, *J*= 7.8 Hz, 1H), 7.17 (t, *J*= 7.2 Hz, 1H), 7.34-7.39 (m, 2H), 7.61 (d, *J*= 7.8 Hz, 2H), 7.74 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 20.8, 114.0, 120.0, 120.4, 122.9, 124.7, 129.1, 130.3, 135.5, 137.7, 144.7, 167.6.

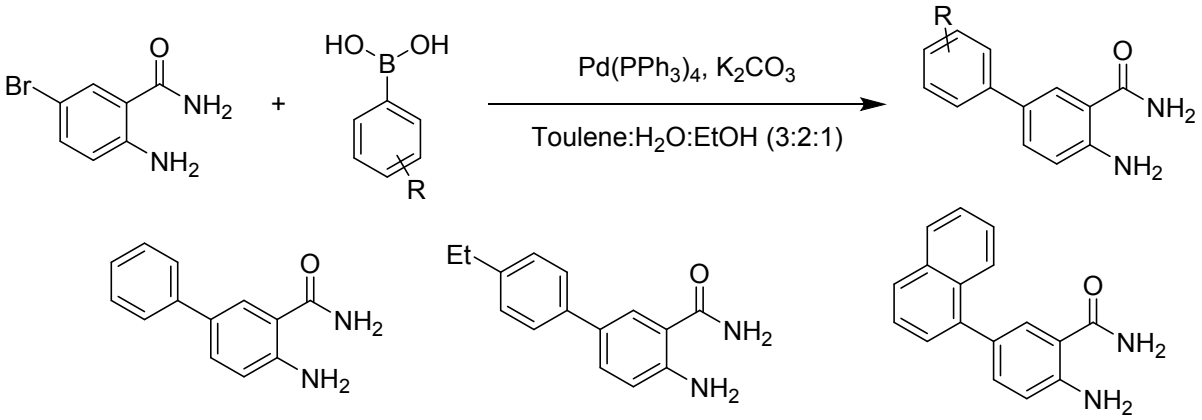
4.3 Bromination and Iodination of 2-aminobenzamides[2]



A stirred solution of 2-aminobenzamide (1 equiv.) in acetonitrile at room temperature was treated with NBS/NIS (1.05 equiv.). The mixture was stirred at room temperature and quenched with ice-cold water. The mixture was extracted with EtOAc two times, and the combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography using EtOAc/Hexane (4:6) as eluent to furnish the substituted 2-aminobenzamides.

4.4 General procedure for Suzuki coupling [3]

A flask fitted with a stirrer bar was charged with 2-amino-5bromobenzamide (1.0 equiv.), phenyl boronic acid (2.6 equiv.) and potassium carbonate (9.0 equiv.) under nitrogen. Tetrakis(triphenylphosphine)palladium (10 mol %) was then added under positive pressure of nitrogen prior to addition of degassed toluene , water and ethanol (3:2:1) in succession. The orange solution was then heated at reflux with stirring overnight. After cooling, DCM (25 mL) and saturated ammonium chloride (25 mL) were added and the layers separated. The aqueous phase was extracted with further DCM (2 x 25 mL) and the combined organics washed with saturated sodium hydrogen carbonate (25 mL) and water (25 mL) and concentrated under reduced pressure. The crude product was purified by column chromatography using EtOAc/Hexane (4:6) as eluent to furnish the substituted 2-amino-5-phenylbenzamide.

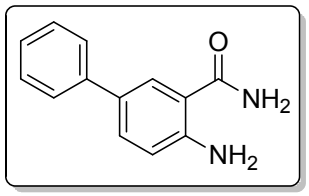


1h, 81%
White solid

1i, 75%
White solid

1j, 70%
Light brown solid

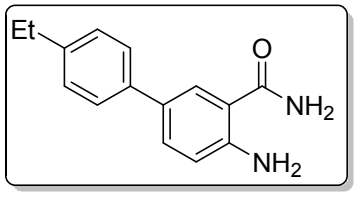
4-amino-[1,1'-biphenyl]-3-carboxamide 1h



Yield 81%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 6.69 (br s, 2H), 6.80 (d, *J*= 8.4 Hz, 1H), 7.13 (br s, 1H), 7.24 (t, *J*= 7.2 Hz, 1H), 7.39 (t, *J*= 7.8 Hz, 2H), 7.51 (d, *J*= 8.7 Hz, 1H), 7.65 (d, *J*= 7.8 Hz, 2H), 7.81 (s, 1H), 7.98 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 113.9, 116.9, 125.5, 125.9, 126.3, 126.7, 128.6, 130.1, 140.0, 149.6, 171.2.

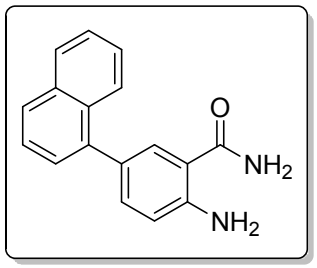
4-amino-4'-ethyl-[1,1'-biphenyl]-3-carboxamide 1i



Yield 81%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 1.27 (t, *J*= 7.5 Hz, 3H), 2.60 (q, *J*= 7.5 Hz, 7.5 Hz, 2H), 6.64 (br s, 2H), 6.78 (d, *J*= 8.4 Hz, 1H), 7.12 (br s, 1H), 7.23 (d, *J*= 7.5 Hz, 2H), 7.31 (d, *J*= 7.8 Hz, 1H), 7.47 (d, *J*= 8.4 Hz, 1H), 7.56 (d, *J*= 7.8 Hz, 2H), 7.99 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 15.6, 27.7, 113.9, 116.9, 125.5, 126.4, 126.5, 128.0, 129.9, 137.4, 141.4, 149.3, 171.3.

2-amino-5-(naphthalen-1-yl)benzamide 1j



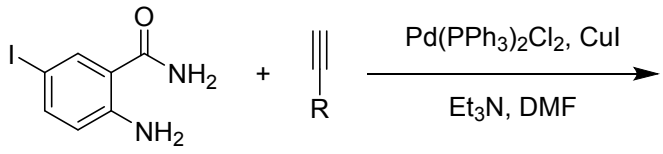
Yield 81%, white solid.

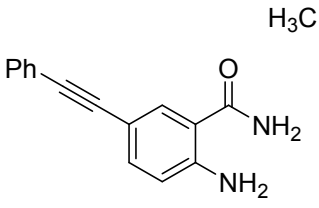
^{1}H NMR (300 MHz, DMSO-d_6): δ_H 6.77 (br s, 2H), 6.85 (d, *J*= 8.4 Hz, 1H), 7.20 (br s, 1H), 7.43-7.53 (m, 2H), 7.68 (d, *J*= 8.4 Hz, 1H), 7.88-7.96 (m, 4H), 8.04 (s, 2H), 7.81 (s, 1H); ^{13}C

NMR (75 MHz, DMSO-d_6): δ_C 113.9, 117.0, 123.2, 124.6, 125.3, 15.9, 126.1, 127.0, 127.4, 127.7, 128.0, 130.3, 131.5, 133.4, 137.3, 149.8, 171.2.

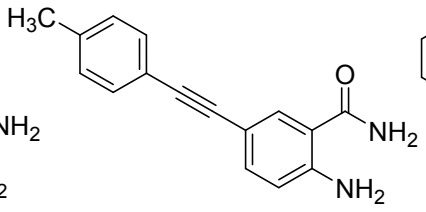
4.5 General procedure for Sonagashira coupling [4]

2-Amino-5-iodobenzamide (1 equiv.), $Pd(PPh_3)_2Cl_2$ (10 mol%) and CuI (10 mol%) were taken in a 25 mL RB under nitrogen atmosphere. DMF and Et_3N (5 mL) were added to the reaction mixture. The resulting reaction mixture were stirred at room temperature for 12 h. After disappearance of the reactant (monitored by TLC), then added 50 mL water to the mixture, extracted with $CHCl_3$ (3 × 30 mL). The combined organic layers were washed with brine (30 mL), dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel afforded the corresponding coupling product.

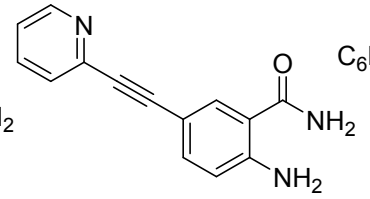




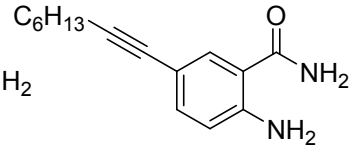
1k, 86%
Pale yellow solid



1l, 85%
Pale yellow solid



1m, 72%
Brown solid

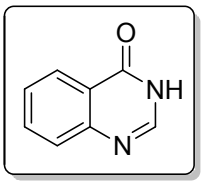


1n, 83%
Yellow liquid

5. General procedure for quinazolinones

Reaction tube was charged with 2-aminobenzamide (0.36 mmol), Cs_2CO_3 (0.72 mmol) and CuI (5 mol%) in alcohol (2 mL). The reaction mixture was stirred under blue light for 24-36 hours. After the reaction completion, mixture was passed through celite and solvent was evaporated under reduced pressure. The crude was purified by column chromatography using EtOAc/Hexane (4: 6) as eluent to furnish the afforded quinazolinone compounds.

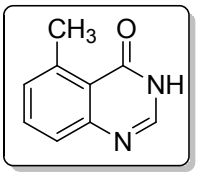
quinazolin-4(3H)-one 2a[5]:



Yield 88%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.52 (t, *J*= 7.2 Hz, 1H), 7.65-7.68 (m, 1H), 7.81 (t, *J*= 7.2 Hz, 1H), 8.10 (s, 1H), 8.13 (d, *J*= 8.1 Hz, 1H), 12.25 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 122.5, 125.7, 126.7, 127.1, 134.2, 145.3, 148.7, 160.7.

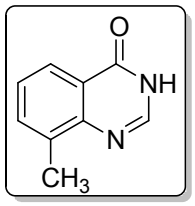
5-methylquinazolin-4(3H)-one 2b:



Yield 85%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.78 (s, 3H), 7.25 (d, *J*= 7.2 Hz, 1H), 7.47 (d, *J*= 8.1 Hz, 1H), 7.62 (t, *J*= 7.8 Hz, 1H), 8.00 (s, 1H), 12.02 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 22.4, 120.9, 125.3, 128.9, 133.2, 139.9, 145.0, 150.3, 161.5.

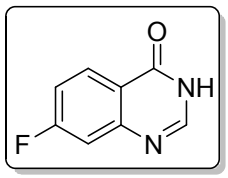
8-methylquinazolin-4(3H)-one 2c:



Yield 80%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.52 (s, 3H), 7.38 (t, *J*= 7.8 Hz, 1H), 7.65 (d, *J*= 6.9 Hz, 1H), 7.96 (d, *J*= 7.8 Hz, 1H), 8.10 (s, 1H), 12.23 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 17.18, 122.5, 123.4, 126.1, 134.6, 135.2, 144.3, 147.1, 160.9.

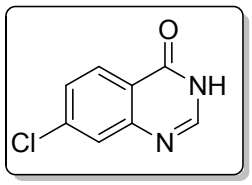
7-fluoroquinazolin-4(3H)-one 2d[6]:



Yield 88%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.36-7.47 (m, 2H), 8.15 (s, 1H), 8.17-8.22 (m, 1H), 12.35 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 112.1, 112.4, 115.0, 115.3, 119.6, 128.8, 129.0, 146.7, 150.8, 150.9, 159.9, 163.8, 167.1.

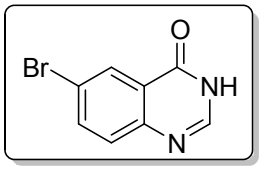
7-chloroquinazolin-4(3H)-one 2e[7]:



Yield 85%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.56 (d, *J*= 8.1 Hz, 1H), 7.12 (s, 1H), 8.12 (d, *J*= 8.7 Hz, 1H), 8.16 (s, 1H), 12.42 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 121.3, 126.2, 126.9, 127.8, 138.8, 146.8, 149.7, 160.1.

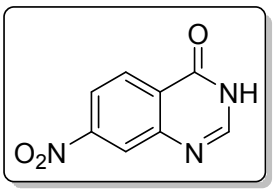
6-bromoquinazolin-4(3H)-one 2f[7]:



Yield 80%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.62 (d, *J*= 8.7 Hz, 1H), 7.93-7.97 (m, 1H), 8.13 (s, 1H), 8.18 (d, *J*= 2.1 Hz, 1H), 12.45 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 119.3, 123.9, 127.9, 128.9, 136.5, 144.7, 147.3, 159.7, 178.4; HRMS: $(M+H)^+$ calculated for $C_8H_6BrN_2O$: 224.9663, Found: 224.9664.

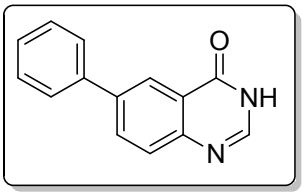
7-nitroquinazolin-4(3H)-one 2g:



Yield 77%, pale yellow solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 8.21-8.26 (m, 2H), 8.32-8.38 (m, 2H), 12.59 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 120.0, 121.9, 126.8, 128.0, 147.4, 149.0, 150.9, 159.5.

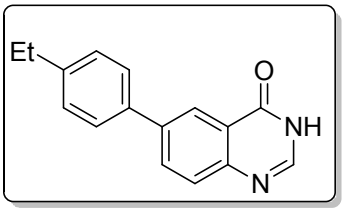
6-phenylquinazolin-4(3H)-one 2h:



Yield 75%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.43 (t, *J*= 7.2 Hz, 1H), 7.52 (t, *J*= 7.5 Hz, 2H), 7.77 (d, *J*= 7.8 Hz, 3H), 8.12-8.14 (m, 2H), 8.36 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 122.9, 123.1, 126.7, 127.8, 129.1, 132.7, 138.3, 138.7, 145.4, 148.0, 160.8.

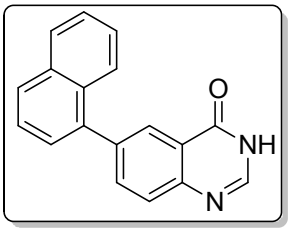
6-(4-ethylphenyl)quinazolin-4(3H)-one 2i:



Yield 75%, pale yellow solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 1.21 (t, *J*= 7.2 Hz, 3H), 2.65 (q, *J*= 7.2 Hz, 7.5 Hz, 2H), 7.34 (d, *J*= 7.8 Hz, 2H), 7.68 (d, *J*= 7.8 Hz, 2H), 7.74 (d, *J*= 8.4 Hz, 1H), 8.10-8.13 (m, 2H), 8.32 (s, 1H), 12.31 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 15.4, 27.7, 122.7, 122.8, 126.6, 127.8, 128.5, 132.6, 136.1, 138.3, 143.6, 145.2, 147.8, 160.7.

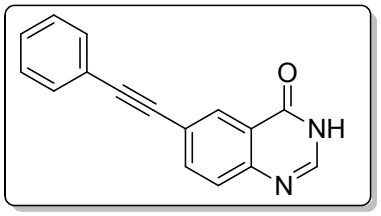
6-(naphthalen-1-yl)quinazolin-4(3H)-one 2j:



Yield 70%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.52-7.59 (m, 2H), 7.80-7.82 (m, 1H), 7.95 (t, *J*= 8.7 Hz, 2H), 8.06 (d, *J*= 8.4 Hz , 2H), 8.12-8.15 (m, 1H), 8.30 (d, *J*= 7.5 Hz, 1H), 8.35 (s, 1H), 8.51 (s, 1H), 12.34 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 123.4, 124.8, 125.6, 126.4, 126.5, 127.4, 128.0, 128.3, 128.7, 132.3, 132.9, 133.3, 136.0, 138.2, 145.5, 148.1, 160.8.

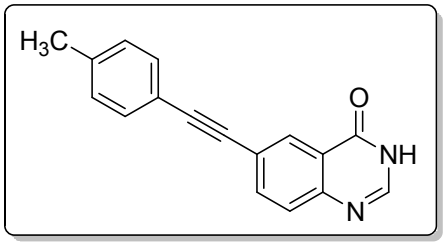
6-(phenylethynyl)quinazolin-4(3H)-one 2k:



Yield 75%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.45 (d, *J*= 2.7 Hz, 2H), 7.60 (d, *J*= 3 Hz , 2H), 7.70 (d, *J*= 8.4 Hz, 1H), 7.93 (d, *J*= 8.4 Hz, 1H), 8.13 (s, 1H), 8.22 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 88.3, 90.4, 120.4, 121.8, 122.7, 127.8, 128.7, 129.0, 131.4, 136.5, 146.2, 148.4, 160.0; HRMS: $(M+2H)^+$ calculated for $C_{16}H_{12}N_2O$: 248.0949, Found: 248.0959.

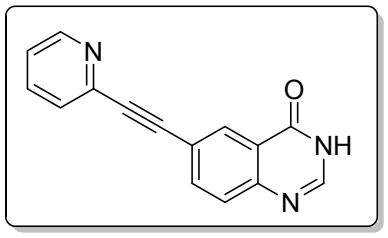
6-(p-tolylethynyl)quinazolin-4(3H)-one 2l:



Yield 70%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.35 (s, 3H), 7.26 (d, *J*= 7.8 Hz, 2H), 7.50 (d, *J*= 7.8 Hz , 2H), 7.69 (d, *J*= 8.4 Hz, 1H), 7.92 (d, *J*= 8.4 Hz, 1H), 8.14 (s, 1H), 8.21 (s, 1H), 12.41 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 21.03, 87.7, 90.7, 118.8, 120.6, 122.8, 127.8, 128.5, 129.3, 131.3, 136.5, 138.8, 146.1, 148.4, 160.0.

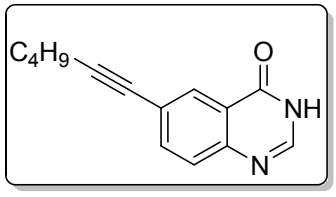
6-(pyridin-2-ylethynyl)quinazolin-4(3H)-one 2m:



Yield 65%, brown solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.43-7.47 (m, 1H), 7.73 (d, *J*= 8.4 Hz, 2H), 7.86-7.91 (m, 1H), 7.97-8.01 (m, 1H), 8.17 (s, 1H), 8.28 (s, 1H), 8.64 (d, *J*= 4.5 Hz, 1H), 12.47 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 87.2, 89.9, 119.5, 122.9, 123.7, 127.5, 128.0, 129.2, 136.7, 1368, 141.9, 146.5, 149.0, 150.1, 159.9.

6-(hex-1-yn-1-yl)quinazolin-4(3H)-one 2n:

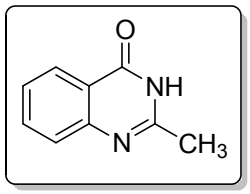


Yield 75%, pale yellow solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 0.88 (t, *J*= 7.1 Hz, 3H), 1.22-1.29 (m, 4H), 1.31-1.42 (m, 2H), 1.54-1.56 (m, 2H), 2.45 (t, *J*= 8.4 Hz, 2H), 7.63 (d, *J*= 8.4 Hz, 1H), 7.74-7.77 (m, 1H), 8.03

(s, 1H), 8.11 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 13.8, 18.6, 21.9, 27.9, 27.9, 28.0, 30.7, 79.6, 92.2, 121.5, 122.6, 127.6, 128.2, 136.6, 145.8, 147.9, 160.0.

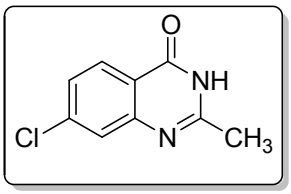
2-methylquinazolin-4(3H)-one 3a[5]:



Yield 65%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.35 (s, 3H), 7.45 (t, *J*= 7.2 Hz, 1H), 7.57 (d, *J*= 8.1 Hz, 1H) 7.74-7.80 (m, 1H), 8.08 (d, *J*= 7.8 Hz, 1H), 12.21 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 21.3, 120.5, 125.6, 125.8, 126.5, 134.2, 148.9, 154.2, 161.6; HRMS: $(M+H)^+$ calculated for $C_9H_9N_2O$: 161.0714, Found: 161.0728.

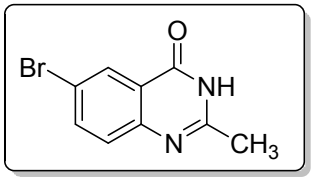
7-chloro-2-methylquinazolin-4(3H)-one 3b[8]:



Yield 55%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.35 (s, 3H), 7.48 (t, *J*= 6.9 Hz, 1H), 7.61 (d, *J*= 1.5 Hz, 1H) 8.06 (d, *J*= 8.4 Hz, 1H), 12.34 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 21.4, 119.4, 125.6, 126.1, 127.7, 138.8, 150.0, 156.0, 161.0.

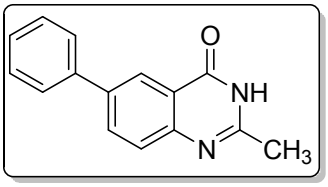
6-bromo-2-methylquinazolin-4(3H)-one 3c[8]:



Yield 60%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.34 (s, 3H), 7.52 (d, *J*= 8.4 Hz, 1H), 7.91 (d, *J*= 8.7 Hz, 1H) 8.13 (s, 1H), 12.39 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 21.4, 118.1, 122.2, 127.7, 128.9, 137.0, 147.8, 155.0, 160.5.

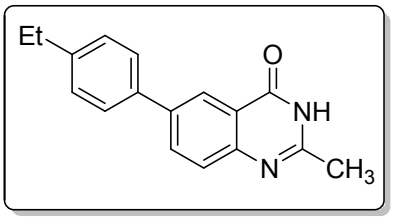
2-methyl-6-phenylquinazolin-4(3H)-one 3e:



Yield 55%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.37 (s, 3H), 7.41-7.49 (m, 3H), 7.63-7.74 (m, 3H), 8.07 (d, *J*= 7.8 Hz, 1H), 8.28 (s, 1H), 12.28 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 21.4, 120.8, 122.9, 126.6, 127.2, 127.7, 129.1, 132.7, 137.5, 138.8, 148.2, 154.3, 161.7.

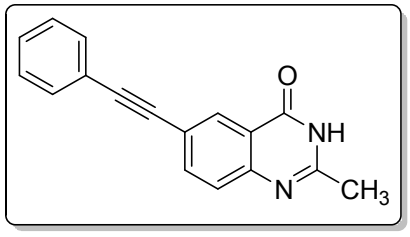
6-(4-ethylphenyl)-2-methylquinazolin-4(3H)-one 3f:



Yield 55%, pale yellow solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 1.22 (t, *J*= 7.5 Hz, 3H), 2.38 (s, 3H), 2.66 (q, *J*= 7.5 Hz, 7.5 Hz, 2H), 7.32-7.34 (m, 2H), 7.65-7.67 (m, 3H), 8.04-8.11 (m, 1H), 8.28 (s, 1H), 12.25 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 15.4, 21.4, 27.7, 120.9, 122.5, 126.5, 127.2, 128.4, 132.5, 136.2, 137.4, 143.4, 148.0, 154.1, 161.7; (M+Na) calculated for $C_{17}H_{16}N_2ONa$: 287.1160, Found: 287.1159.

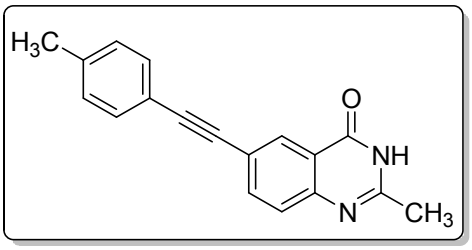
2-methyl-6-(phenylethynyl)quinazolin-4(3H)-one 3g:



Yield 60%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.37 (s, 3H), 7.45 (t, *J*= 3 Hz, 3H), 7.58-7.62 (m, 3H), 7.87-7.90 (m, 1H), 8.17 (s, 1H), 12.37 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 21.4, 88.5, 90.1, 119.4, 120.8, 122.0, 127.1, 128.6, 129.7, 128.9, 131.4, 136.4, 148.7, 155.3, 160.9.

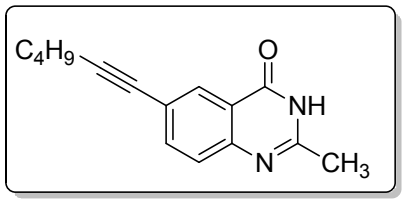
2-methyl-6-(p-tolylethynyl)quinazolin-4(3H)-one 3h:



Yield 55%, yellow solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 2.35 (s, 3H), 2.37 (s, 3H), 7.25-7.27 (m, 3H), 7.48-7.50 (m, 3H), 7.59 (d, *J*= 8.1 Hz, 1H), 7.87 (d, *J*= 8.4 Hz, 1H), 8.15 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 21.0, 21.4, 87.8, 90.4, 118.9, 119.8, 120.7, 127.1, 128.4, 129.4, 131.3, 136.6, 138.8, 148.5, 155.4, 161.0.

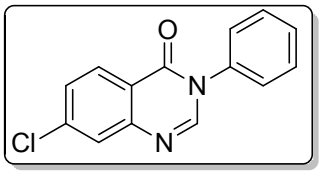
6-(hex-1-yn-1-yl)-2-methylquinazolin-4(3H)-one 3i:



Yield 60%, yellow solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 0.87 (t, *J*= 7.1 Hz, 3H), 1.28-1.31 (m, 4H), 1.37-1.43 (m, 2H), 1.50-1.57 (m, 2H), 2.34 (s, 3H), 2.43 (t, *J*= 8.4 Hz, 2H), 7.50 (d, *J*= 8.4 Hz, 1H), 7.68-7.71 (m, 1H), 7.98 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 13.8, 18.6, 21.4, 21.9, 27.9, 28.0, 30.7, 79.7, 91.7, 120.6, 120.7, 126.9, 128.1, 136.5, 148.1, 154.9, 160.9.

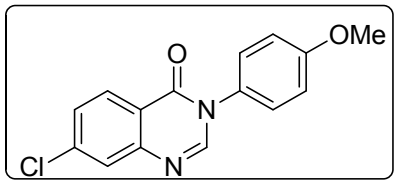
7-chloro-3-phenylquinazolin-4(3H)-one 5a:



Yield 91%, white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 7.44-7.50 (m, 2H), 7.55-7.57 (m, 4H), 7.78 (s, 1H), 8.14 (s, 1H), 8.31 (d, *J*= 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 120.8, 126.9, 127.2, 128.2, 128.6, 129.3, 129.7, 137.2, 140.8, 147.2, 148.8, 160.1.

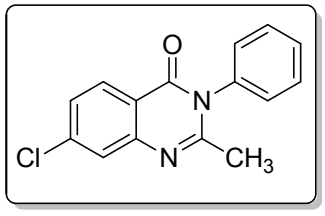
7-chloro-3-(4-methoxyphenyl)quinazolin-4(3H)-one 5b:



Yield 87%, white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 3.89 (s, 3H), 7.06 (d, *J*= 6.9 Hz, 2H), 7.34 (d, *J*= 6.9 Hz, 2H), 7.51 (d, *J*= 8.4 Hz, 1H), 7.77 (s, 1H), 8.12 (s, 1H), 8.30 (d, *J*= 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 55.6, 115.0, 121.0, 127.2, 128.1, 128.2, 128.6, 130., 140.8, 147.6, 149.0, 160.2, 160.4.

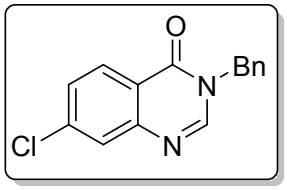
7-chloro-2-methyl-3-phenylquinazolin-4(3H)-one 5c:



Yield 75%, brown solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 2.25 (s, 3H), 7.27 (d, *J*= 6.6 Hz, 2H), 7.41-7.44 (m, 1H), 7.53-7.61 (m, 3H), 7.69 (s, 1H), 8.20 (d, *J*= 8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 24.4, 119.2, 126.4, 127.2, 127.9, 128.5, 129.4, 130.0, 137.4, 140.7, 148.4, 155.6, 161.6.

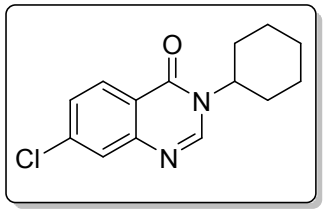
3-benzyl-7-chloroquinazolin-4(3H)-one 5d[9]:



Yield 77%, white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 5.19 (s, 2H), 7.34-7.38 (m, 5H), 7.47 (d, *J*= 8.4 Hz, 1H), 7.72 (s, 1H), 8.12 (s, 1H), 8.28 (d, *J*= 8.4 Hz, 1H) ; ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 49.7, 127.2, 128.0, 128.1, 128.4, 129.1, 129.2, 135.5, 140.6, 147.3, 149.3, 160.5.

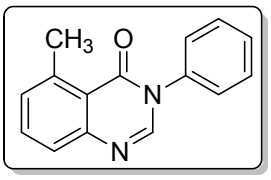
7-chloro-3-cyclohexylquinazolin-4(3H)-one 5e[9]:



Yield 86%, white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 1.22-1.23 (m, 1H), 1.52-1.72 (m, 4H), 1.79-1.83 (m, 1H), 1.95-2.04 (m, 4H), 4.74-4.81 (m, 1H), 7.44 (d, *J*= 8.4 Hz, 1H), 7.70 (s, 1H), 8.12 (s, 1H), 8.24 (d, *J*= 8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 25.3, 25.9, 32.6, 53.9, 120.6, 126.9, 127.7, 128.5, 140.3, 145.1, 148.6, 160.1.

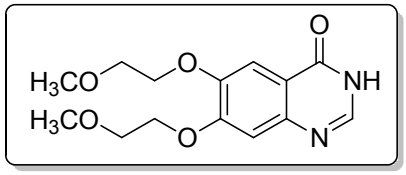
5-methyl-3-phenylquinazolin-4(3H)-one 5f:



Yield 85%, pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 2.89 (s, 3H), 7.30-7.32 (m, 1H), 7.43 (d, *J*= 6.9 Hz, 2H), 7.41-7.65 (m, 5H), 8.08 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 23.3, 120.8, 125.8, 127.2, 129.0, 129.6, 130.3, 133.7, 137.6, 141.8, 145.8, 149.6, 161.4.

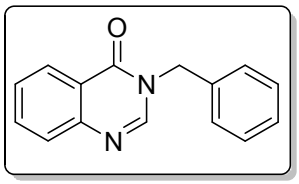
6,7-bis(2-methoxyethoxy)quinazolin-4(3H)-one 6a[7]:



Yield 72%, pale yellow solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 3.34 (s, 6H), 3.69-3.74 (m, 4H), 4.20 (t, *J*= 4.8 Hz , 2H), 4.25 (t, *J*= 4.8 Hz, 2H), 7.15 (s, 1H), 7.48 (s, 1H), 7.94 (s, 1H), 11.9 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 28.2, 58.3, 68.2, 68.3, 70.1, 70.2, 107.1, 109.4, 115.8, 143.8, 144.9, 1479, 154.0, 159.9.

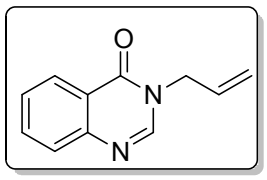
3-benzylquinazolin-4(3H)-one 7:



Yield 85%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 5.23 (s, 2H), 7.30-7.38 (m, 5H), 7.57 (t, *J*= 7.5 Hz, 1H), 7.72 (d, *J*= 8.1 Hz, 1H), 7.85 (t, *J*= 7.5 Hz, 1H), 8.18 (d, *J*= 7.8 Hz, 1H), 8.60 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 48.8, 121.6, 126.0, 127.1, 127.2, 127.6, 128.6, 134.3, 136.7, 147.8, 147.9, 160.1.

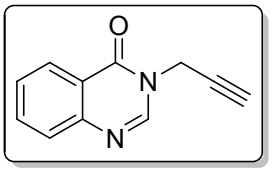
3-allylquinazolin-4(3H)-one 8:



Yield 85%, white solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 4.54-4.56 (m, 2H), 5.15-5.23 (m, 2H), 5.86-5.95 (m, 1H), 7.41-7.44 (m, 1H), 7.60-7.69 (m, 2H), 7.93 (s, 1H), 8.22 (d, *J*= 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 47.3, 117.8, 121.1, 125.7, 126.2, 126.4, 130.8, 133.2, 145.2, 147.0, 159.7.

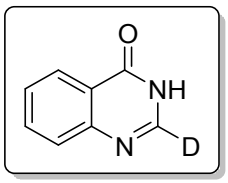
3-(prop-2-yn-1-yl)quinazolin-4(3H)-one 9:



Yield 80%, pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$): δ_H 2.51 (t, *J*= 2.4 Hz, 1H), 4.83 (d, *J*= 2.4 Hz, 2H), 7.49-7.55 (m, 1H), 7.71-7.81 (m, 2H), 8.30-8.33(m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ_C 35.1, 75.1, 76.4, 121.7, 126.8, 1275, 127.6, 134.5, 145.0, 147.9, 160.3.

Quinazolin-4(3H)-one-2-*d* 2a$_d$[10]:



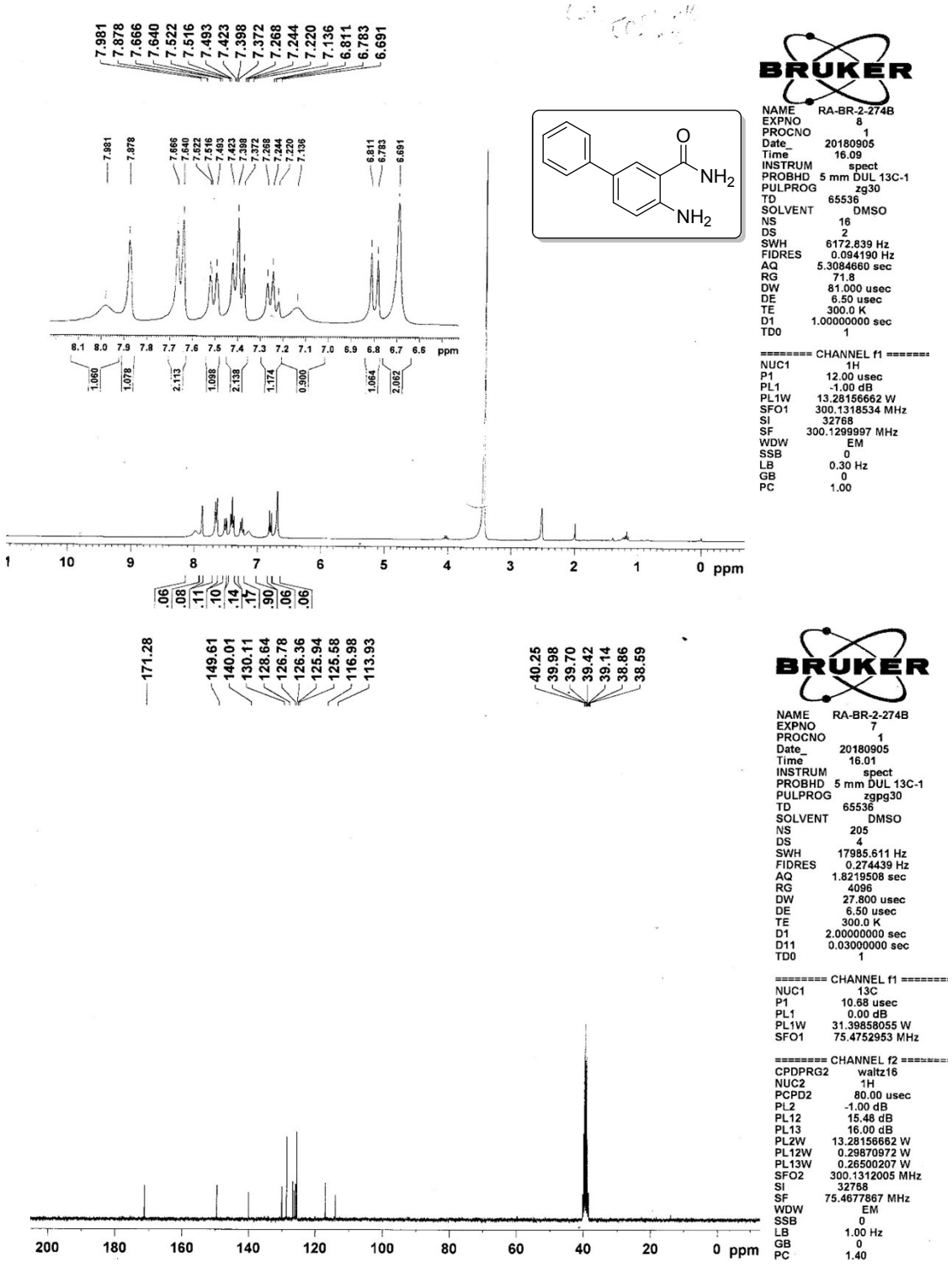
Yield 41%, white solid.

^{1}H NMR (300 MHz, DMSO-d_6): δ_H 7.52 (t, *J*= 7.8 Hz, 1H), 7.66 (d, *J*= 8.1 Hz, 1H), 7.81 (t, *J*= 6.9 Hz, 1H), 8.12 (d, *J*= 7.8 Hz, 1H), 12.25 (br s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ_C 122.6, 125.8, 127.1, 127.2, 134.4, 148.7, 160.8.

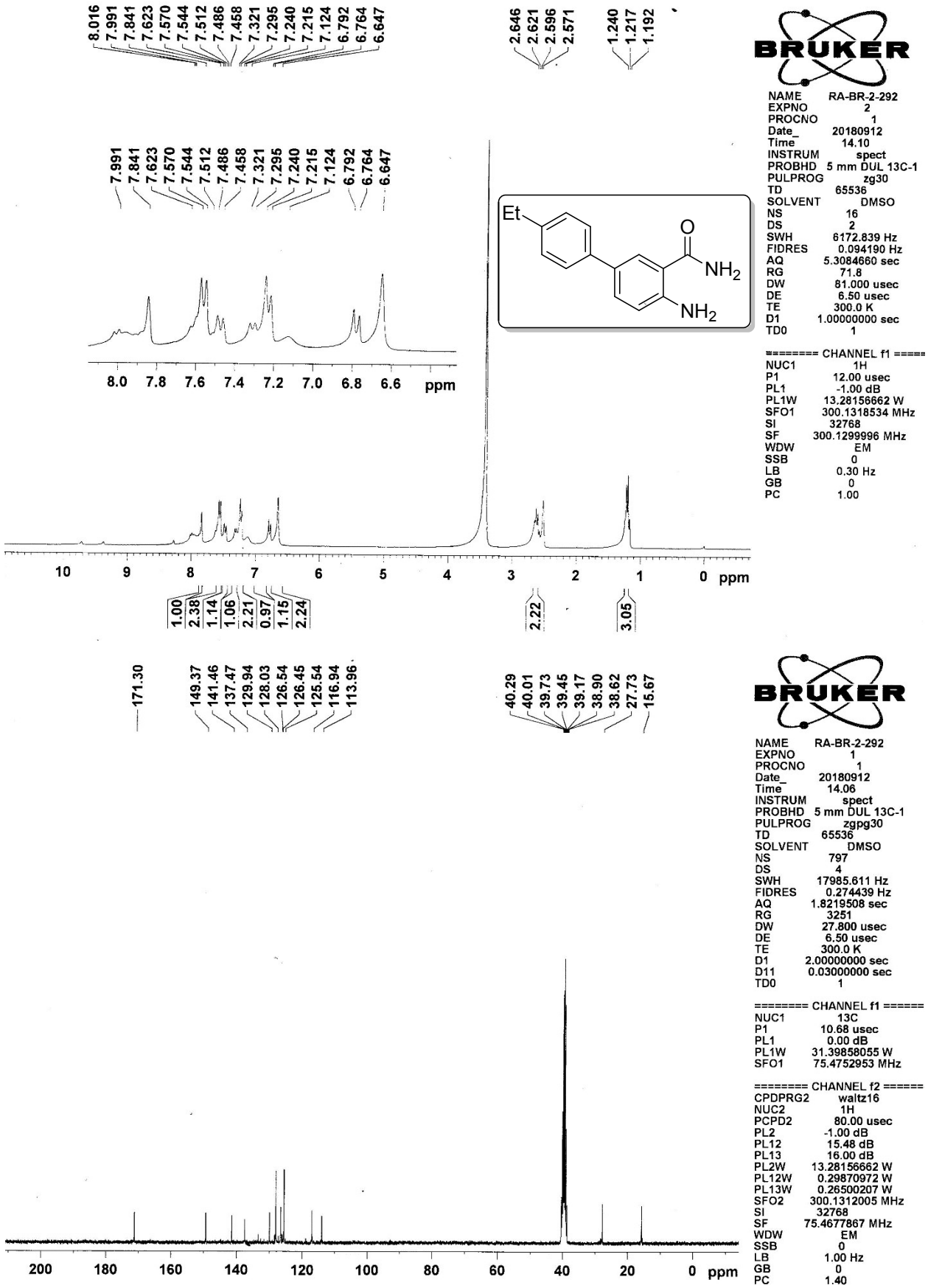
6. References:

1. H. Zhang, H. Liu, X. Luo, Y. Wang, Y. Liu, H. Jin, Z. Liu, W. Yang, P. Yu, L. Zhang and L. Zhang, *j.ejmech.,* 2018, **125**, 235.
2. H. K. Paumo, M. J. Mphahlele, L. Rhyman and P. Ramasami, *Tetrahedron*, 2016, **72**, 123.
3. J. Latham, J-M Henry, H. H. Sharif, B. R. K. Menon, S. A. Shepherd, M. F. Greaney and J. Micklefield, *Nature Communications*, 2016, **7**, 11873.
4. Y. P. Zhu, Z. Fei, M. C. Liu, F. C. Jia and A. X. Wu, *Org. Lett.,* 2013, **15**, 378.
5. Z. Zhang, M. Wang, C. Zhang, Z. Zhang, J. Lu and F. Wang, *Chem. Commun.,* 2015, **51**, 9205.
6. S. Kerdphon, P, Sanghong, J. Chatwichien, V. Choommongkol, P. Rithchumpon, T. Singh and P. Meepowpan, *Eur. J. Org. Chem.,* 2020, **18**, 2730.
7. F. Li, L. Lu and P. Liu, *Org. Lett.,* 2016, **18**, 2580.
8. J. Sun, T. Tao, D. Xu, H. Cao, Q. Kong, X. Wang, Y. Liu, J. Zhao, Y. Wang and Y. Pan, *Tetrahedron Lett.,* 2018, **59**, 2099.
9. D. Shi, C. Shi, J. Wang, L. Rong, Q. Zhuang and X. Wang, *J. Heterocyclic Chem.,* 2005, **42**, 173.
10. B. C. Roy, S. A. Samim, D. Panja and S. Kundu, *Catal. Sci. Technol.,* 2019, **9**, 6002.

7. Spectral data:



^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 1h



BRUKER

NAME RA-BR-2-292
EXPNO 2
PROCNO 1
Date_ 20180912
Time 14.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 71.8
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299996 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

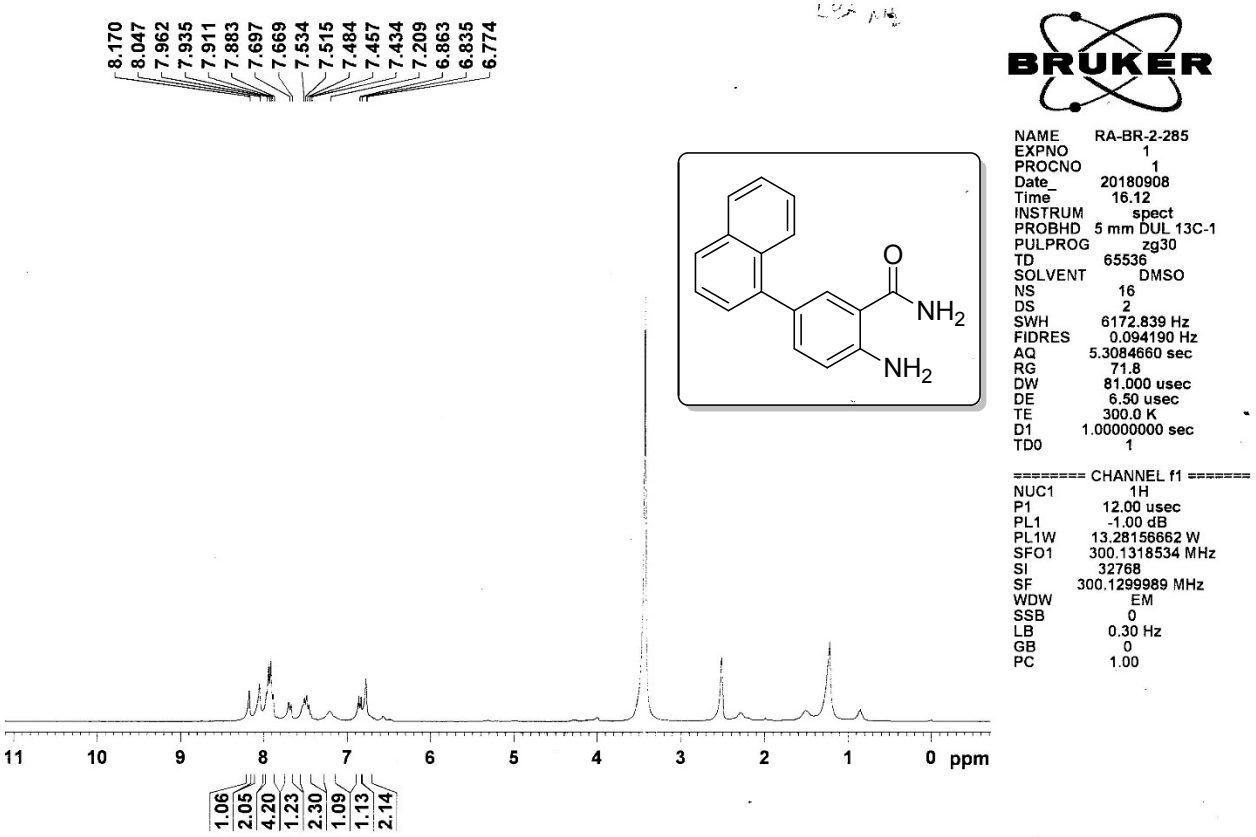
BRUKER

NAME RA-BR-2-292
EXPNO 1
PROCNO 1
Date_ 20180912
Time 14.06
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 797
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 3251
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

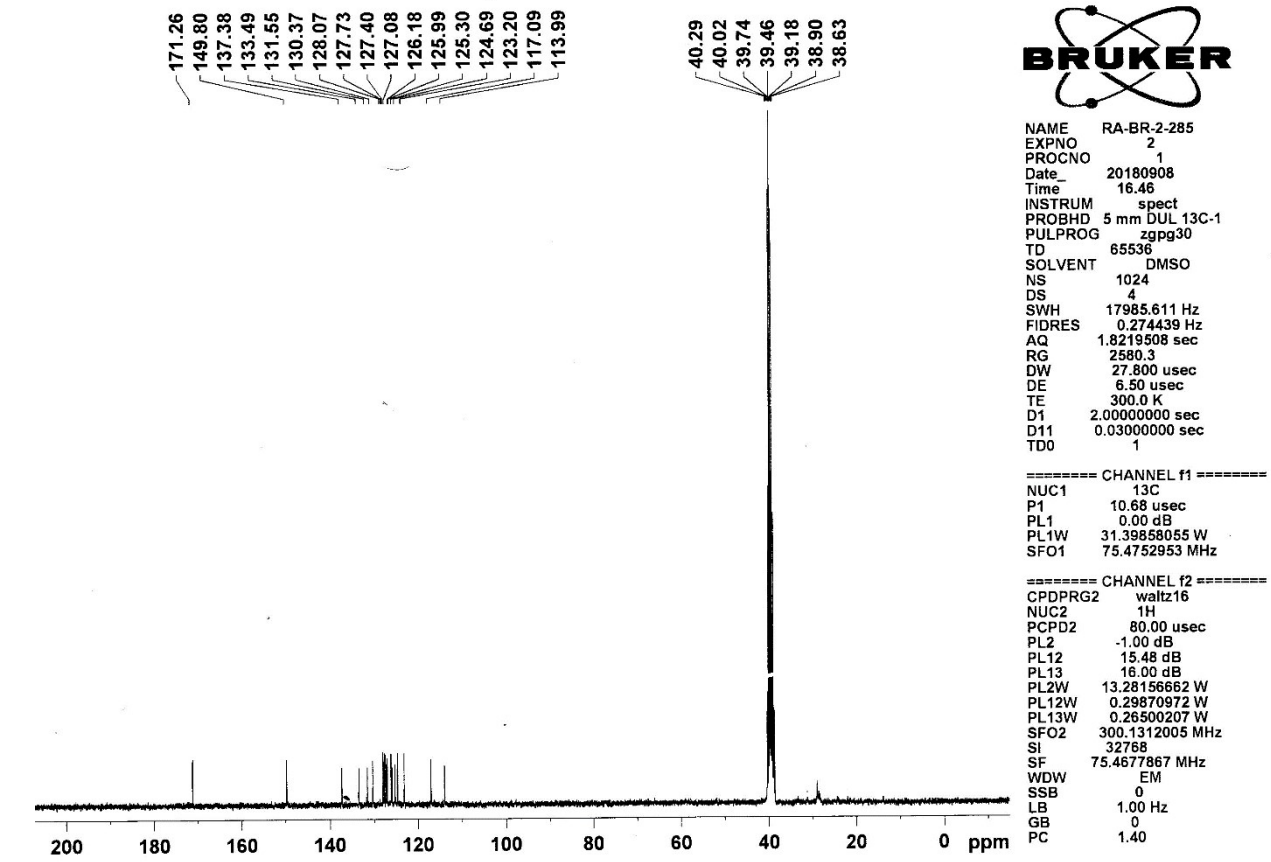
======== CHANNEL f1 ======
NUC1 13C
P1 10.68 usec
PL1 0.00 dB
PL1W 31.39858055 W
SFO1 75.4752953 MHz

======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.29870972 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 1i

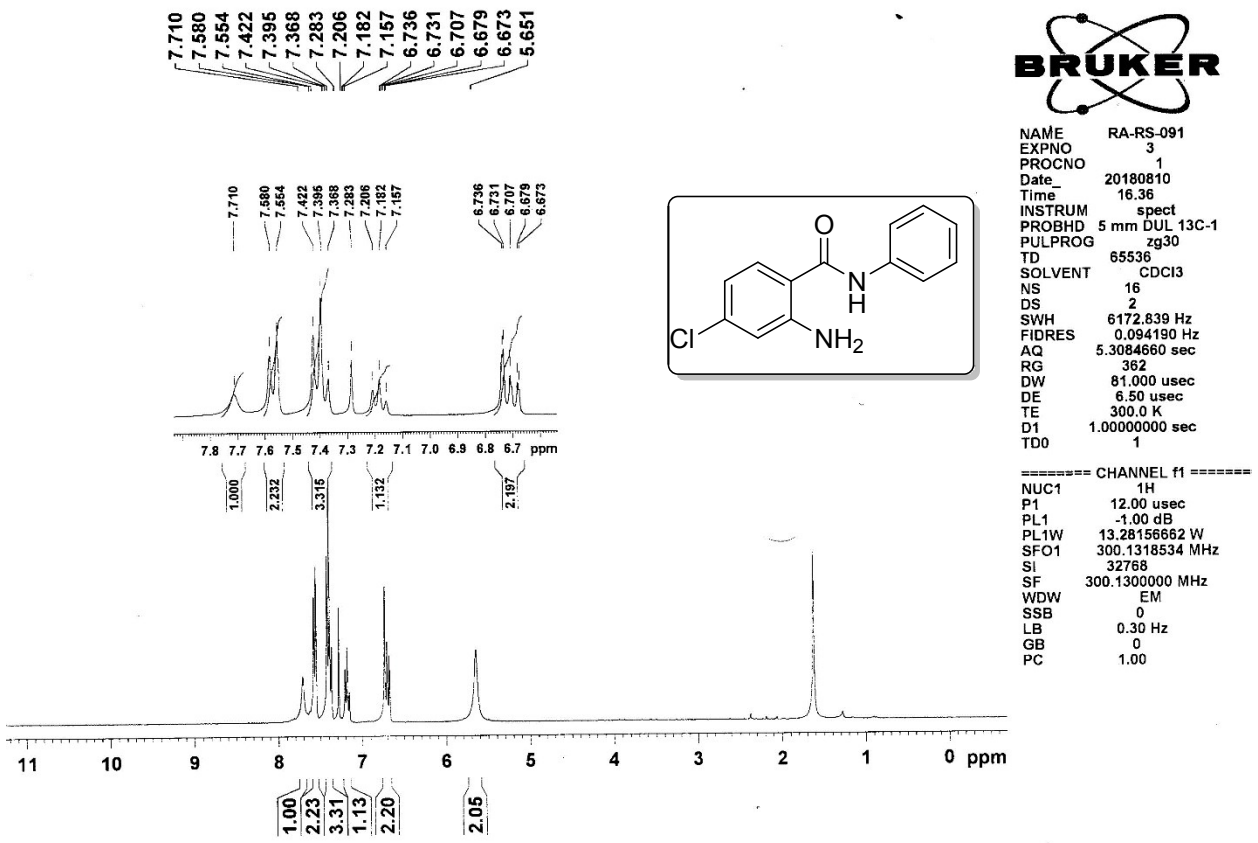


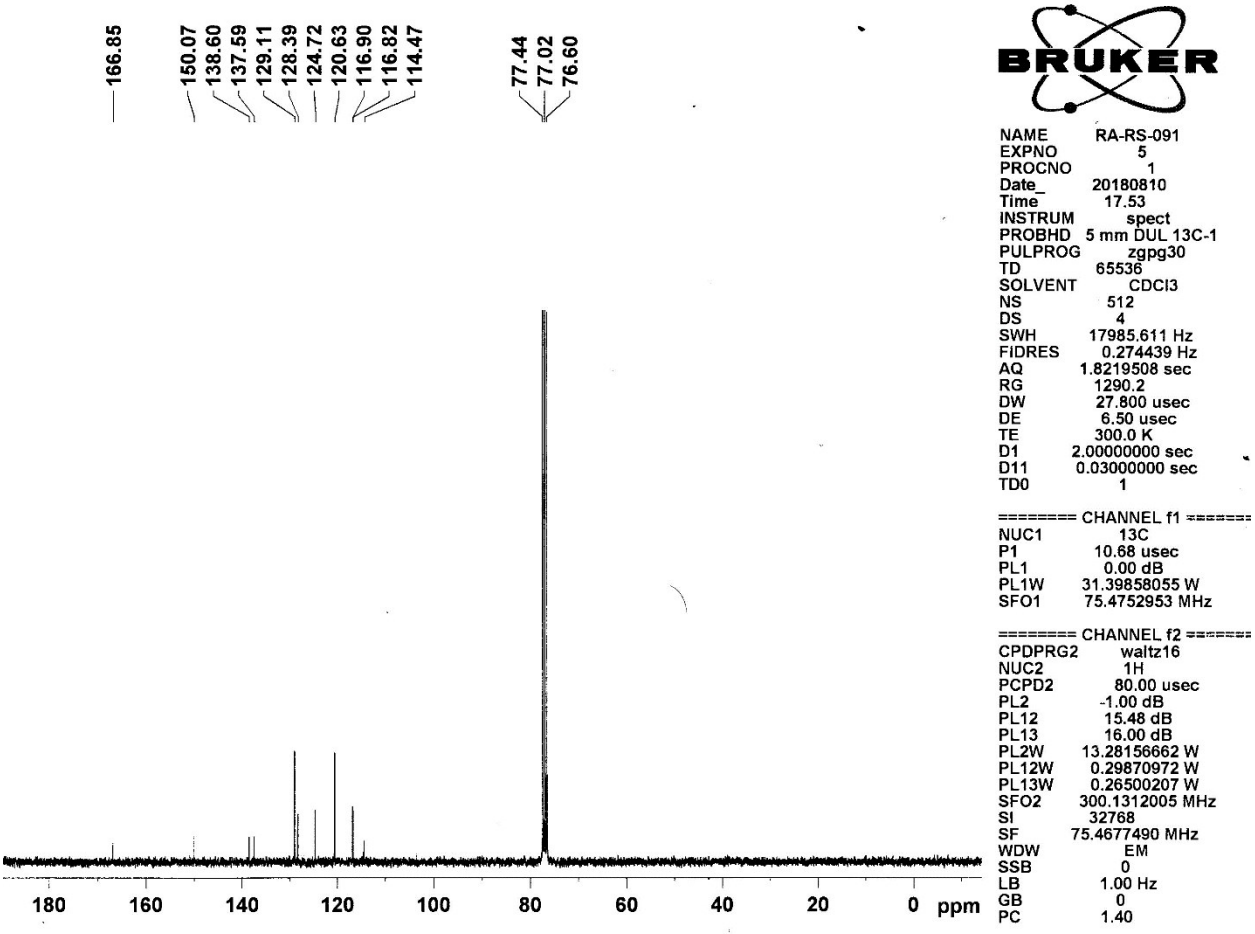

```
NAME        RA-BR-2-285
EXPNO       1
PROCNO      1
Date_       20180908
Time        16.12
INSTRUM     spect
PROBHD      5 mm DUL 13C-1
PULPROG     zg30
TD          65536
SOLVENT     DMSO
NS          16
DS          2
SWH         6172.839 Hz
FIDRES      0.094190 Hz
AQ          5.3084660 sec
RG          71.8
DW          81.000 usec
DE          6.50 usec
TE          300.0 K
D1          1.00000000 sec
TD0         1

======== CHANNEL f1 ========
NUC1        1H
P1          12.00 usec
PL1         -1.00 dB
PL1W        13.28156662 W
SFO1        300.1318534 MHz
SI          32768
SF          300.1299989 MHz
WDW         EM
SSB         0
LB          0.30 Hz
GB          0
PC          1.00
```



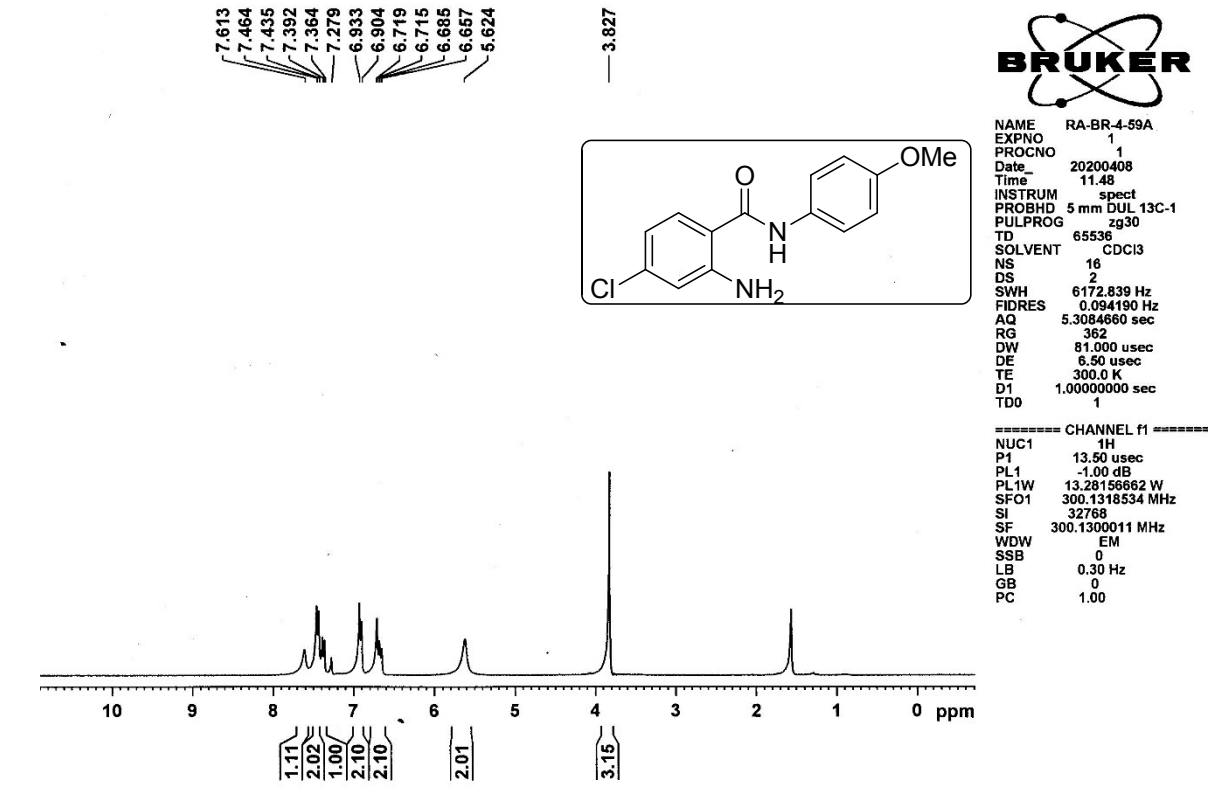

```
NAME        RA-BR-2-285
EXPNO       2
PROCNO      1
Date_       20180908
Time        16.46
INSTRUM     spect
PROBHD      5 mm DUL 13C-1
PULPROG     zgpg30
TD          65536
SOLVENT     DMSO
NS          1024
DS          4
SWH         17985.611 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG          2580.3
DW          27.800 usec
DE          6.50 usec
TE          300.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

======== CHANNEL f1 ========
NUC1        13C
P1          10.68 usec
PL1         0.00 dB
PL1W        31.39858055 W
SFO1        75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2     waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -1.00 dB
PL12        15.48 dB
PL13        16.00 dB
PL2W        13.28156662 W
PL12W       0.29870972 W
PL13W       0.26500207 W
SFO2        300.1312005 MHz
SI          32768
SF          75.4677867 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
```

^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 1j





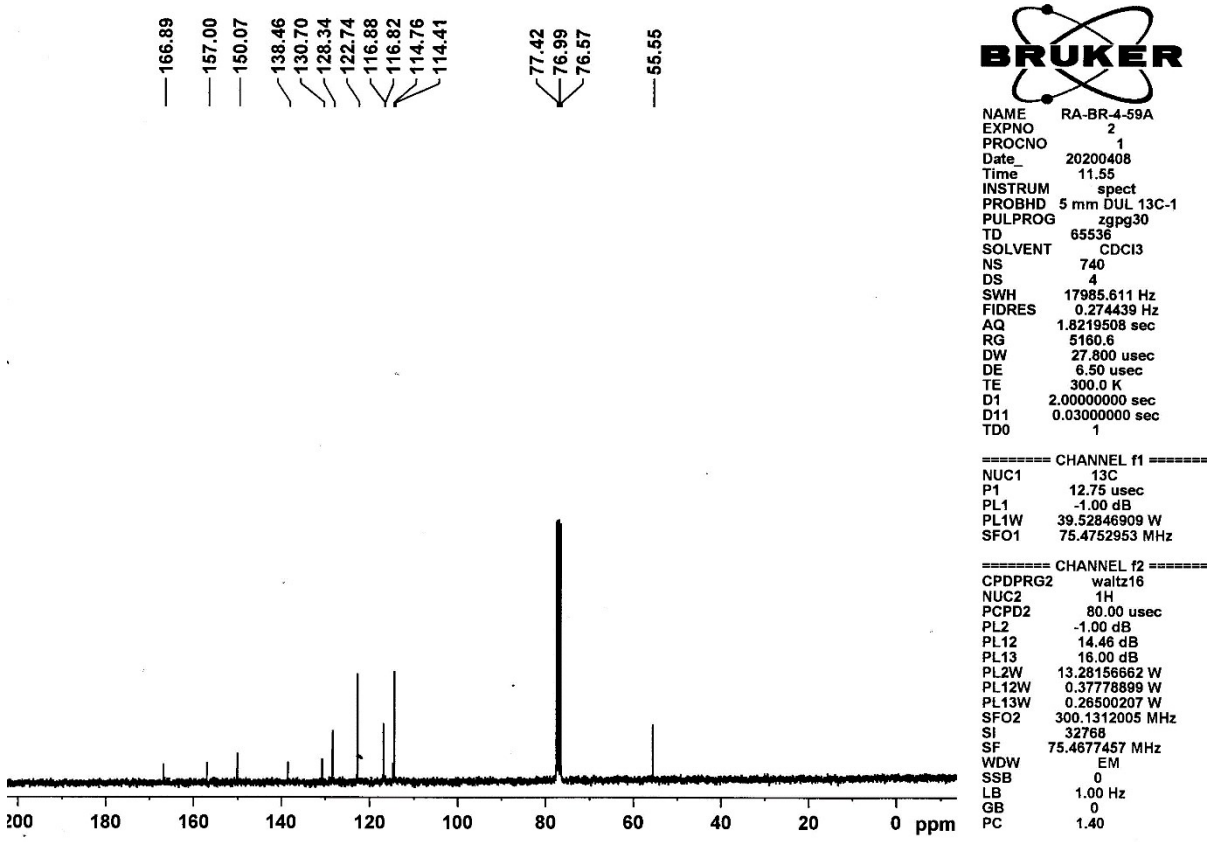
^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 4a



BRUKER

NAME RA-BR-4-59A
EXPNO 1
PROCNO 1
Date_ 20200408
Time 11.48
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 362
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 =======
NUC1 1H
P1 13.50 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300011 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



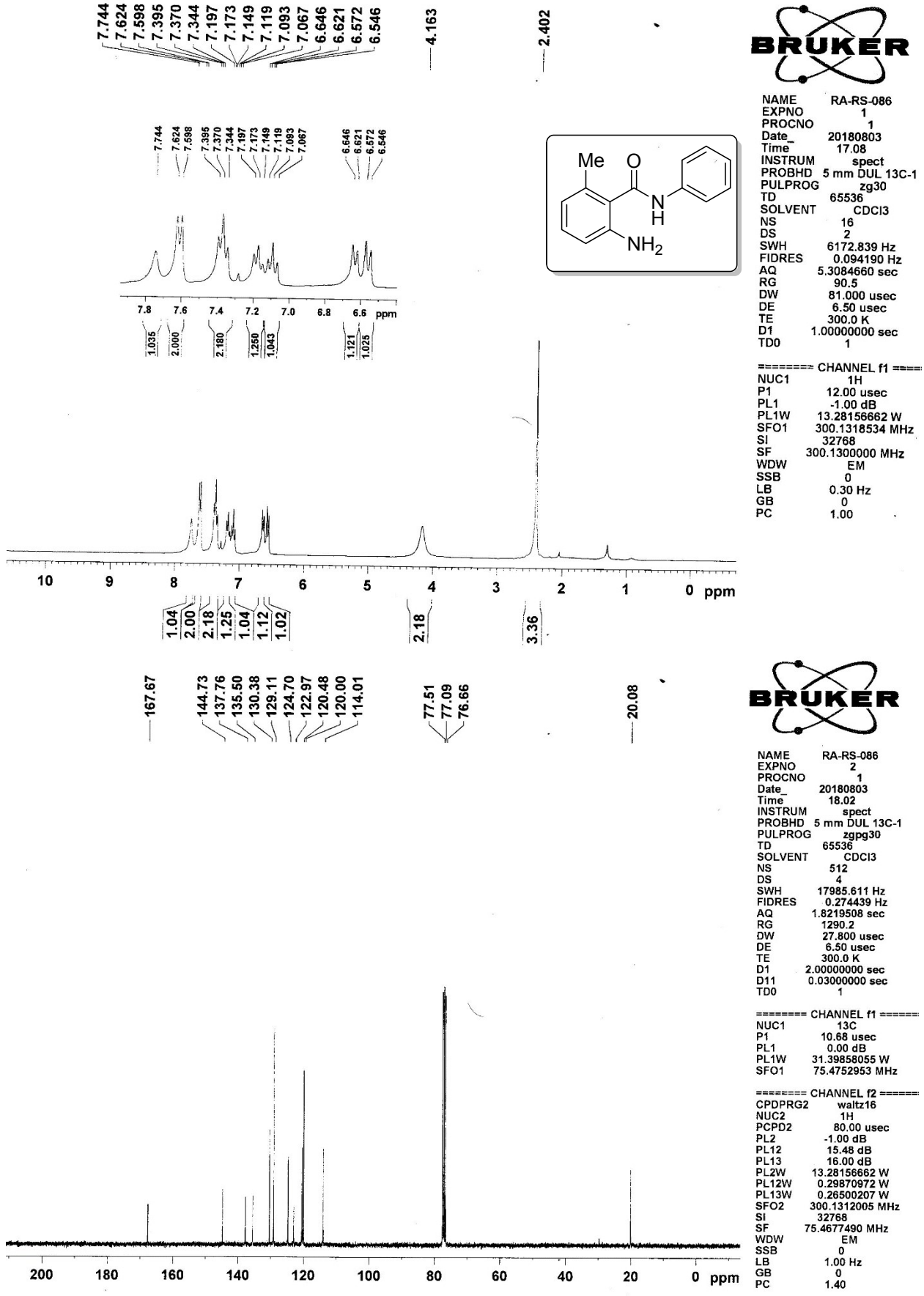
BRUKER

NAME RA-BR-4-59A
EXPNO 2
PROCNO 1
Date_ 20200408
Time 11.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 740
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 5160.6
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 =======
NUC1 13C
P1 12.75 usec
PL1 -1.00 dB
PL1W 39.52846909 W
SFO1 75.4752953 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.46 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.37778899 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677457 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 4b



BRUKER

NAME RA-RS-086
EXPNO 1
PROCNO 1
Date_ 20180803
Time 17.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 90.5
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

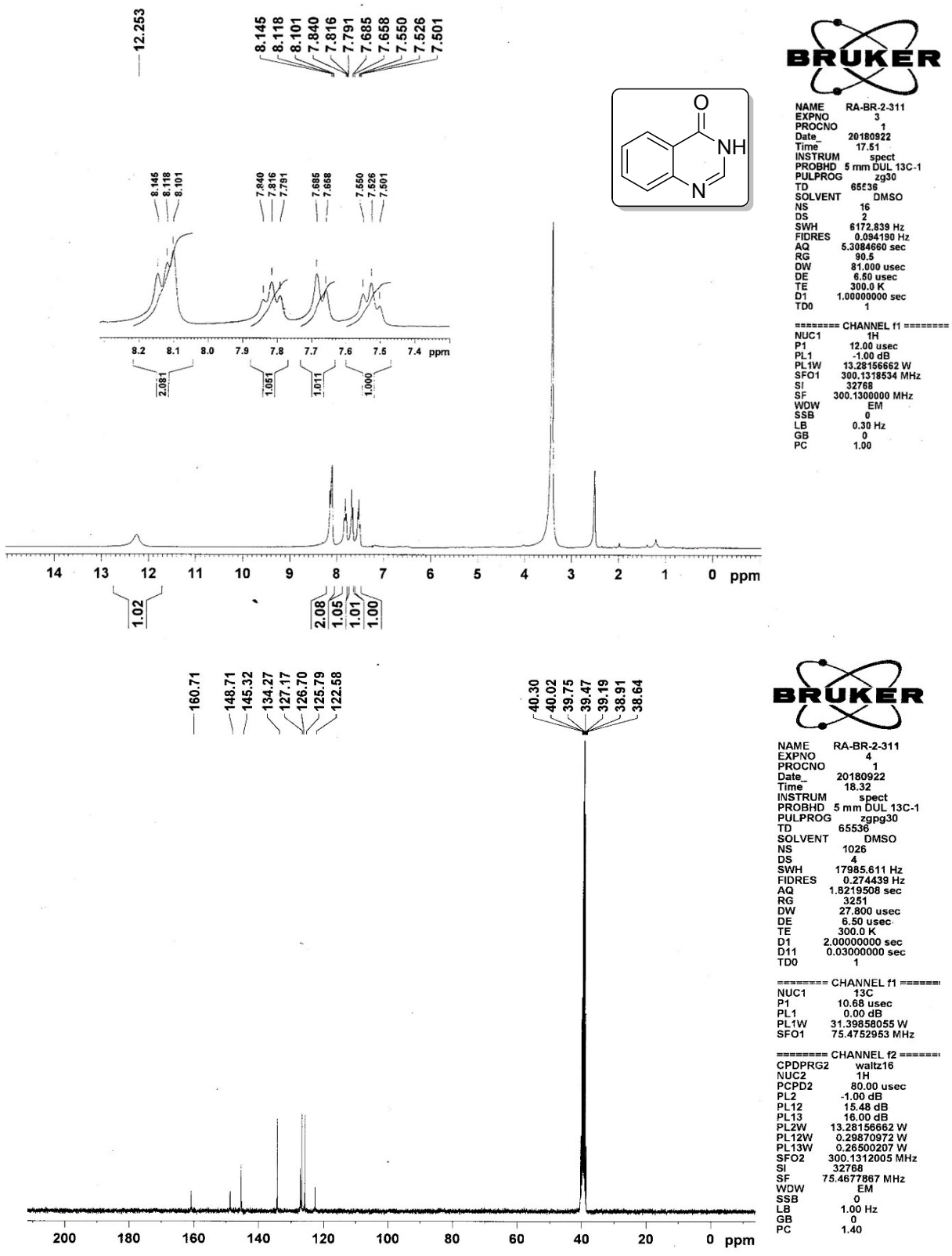
BRUKER

NAME RA-RS-086
EXPNO 2
PROCNO 1
Date_ 20180803
Time 18.02
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1290.2
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

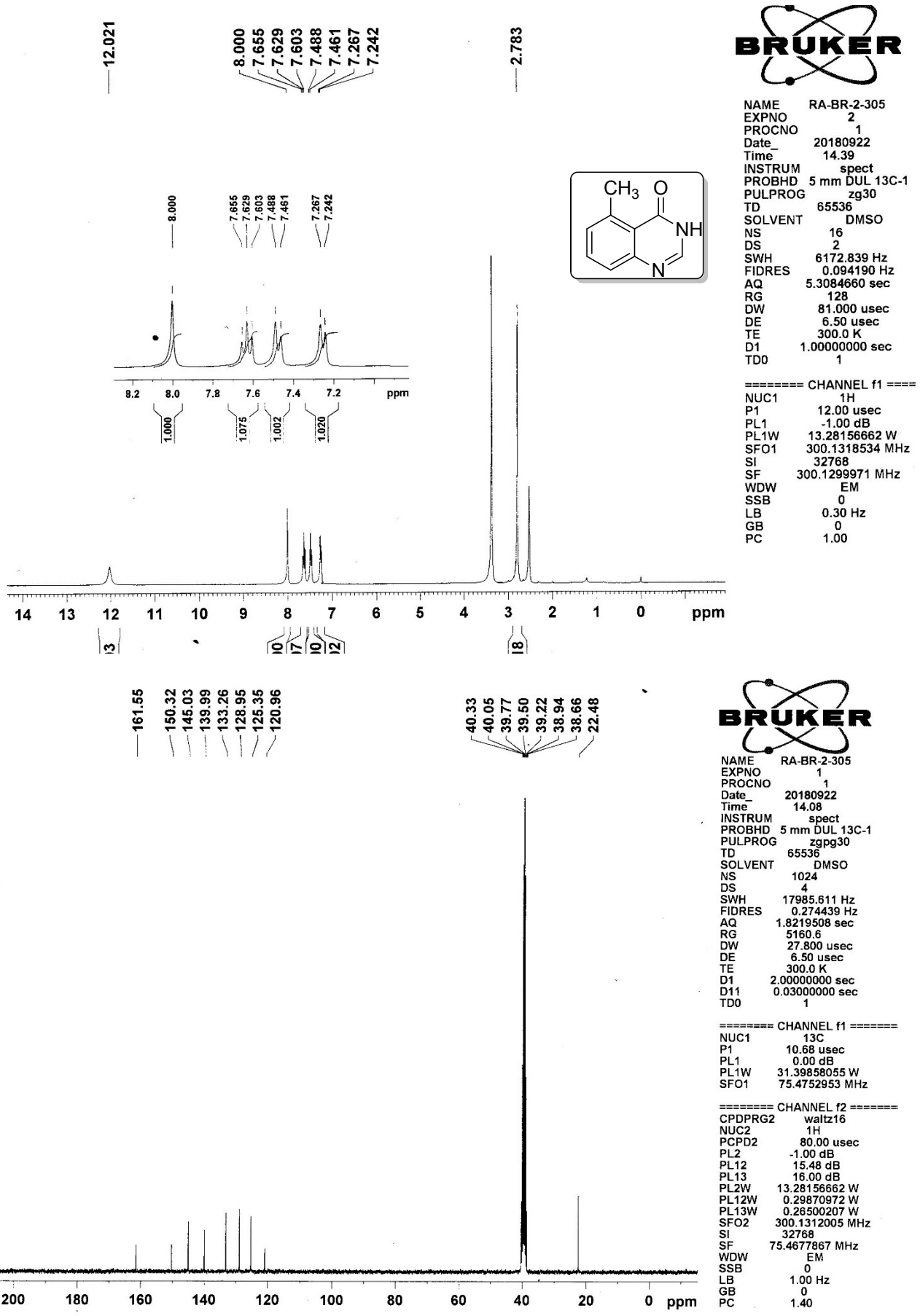
======== CHANNEL f1 ========
NUC1 13C
P1 10.68 usec
PL1 0.00 dB
PL1W 31.39858055 W
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.29870972 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

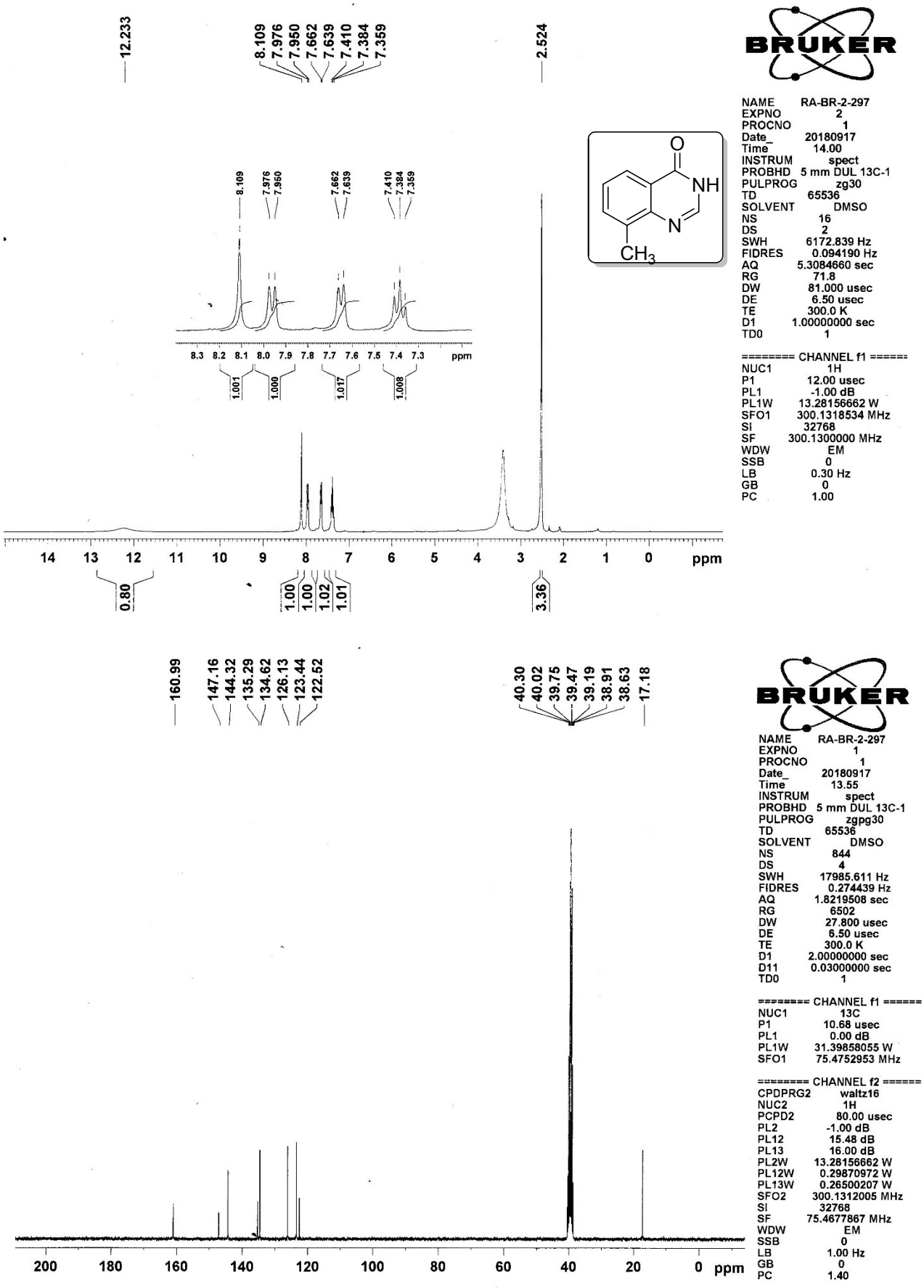
^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 4e



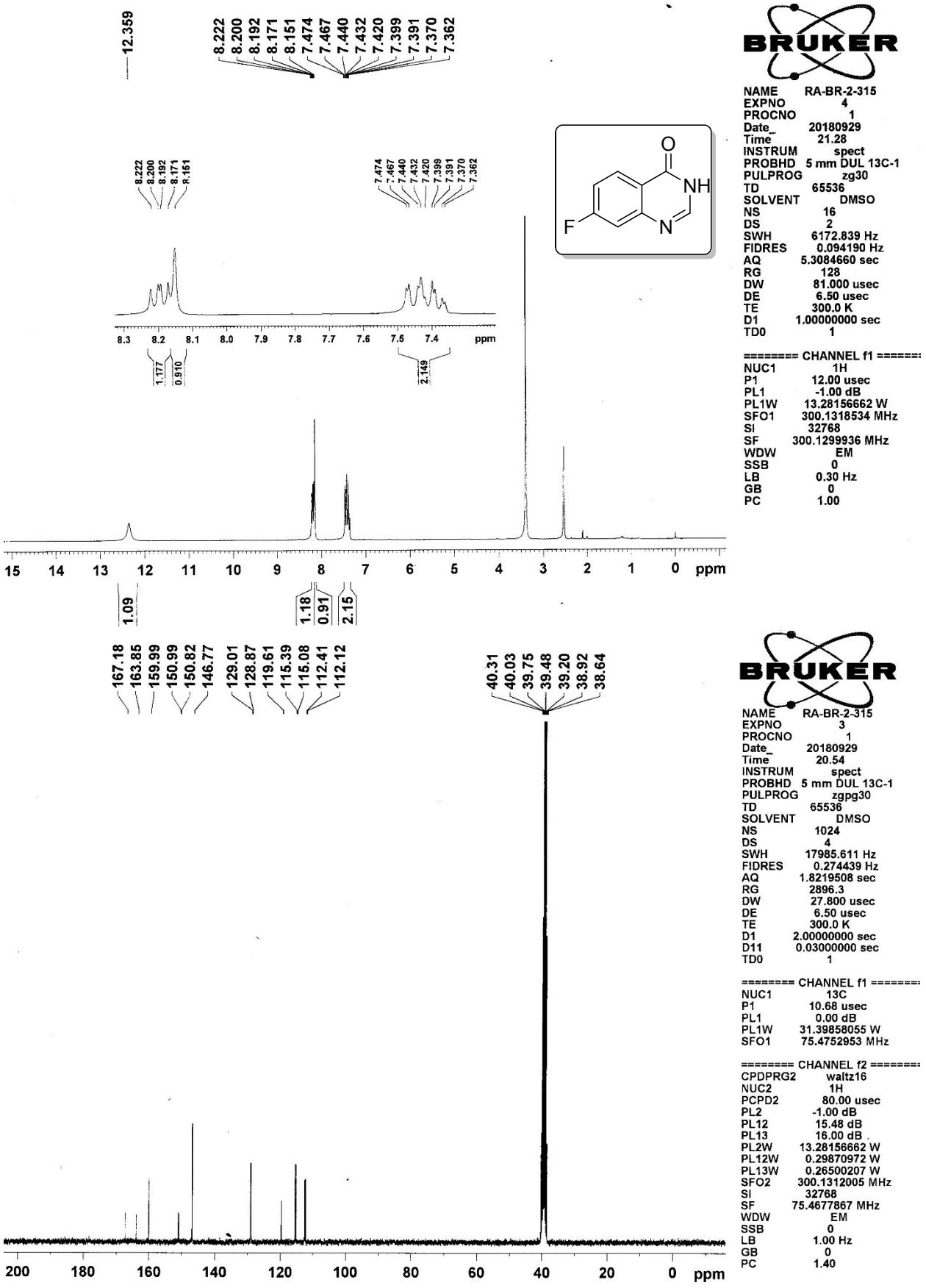
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2a



^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2b



^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2c



BRUKER

NAME RA-BR-2-315
EXPNO 4
PROCNO 1
Date_ 20180929
Time 21.28
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299936 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

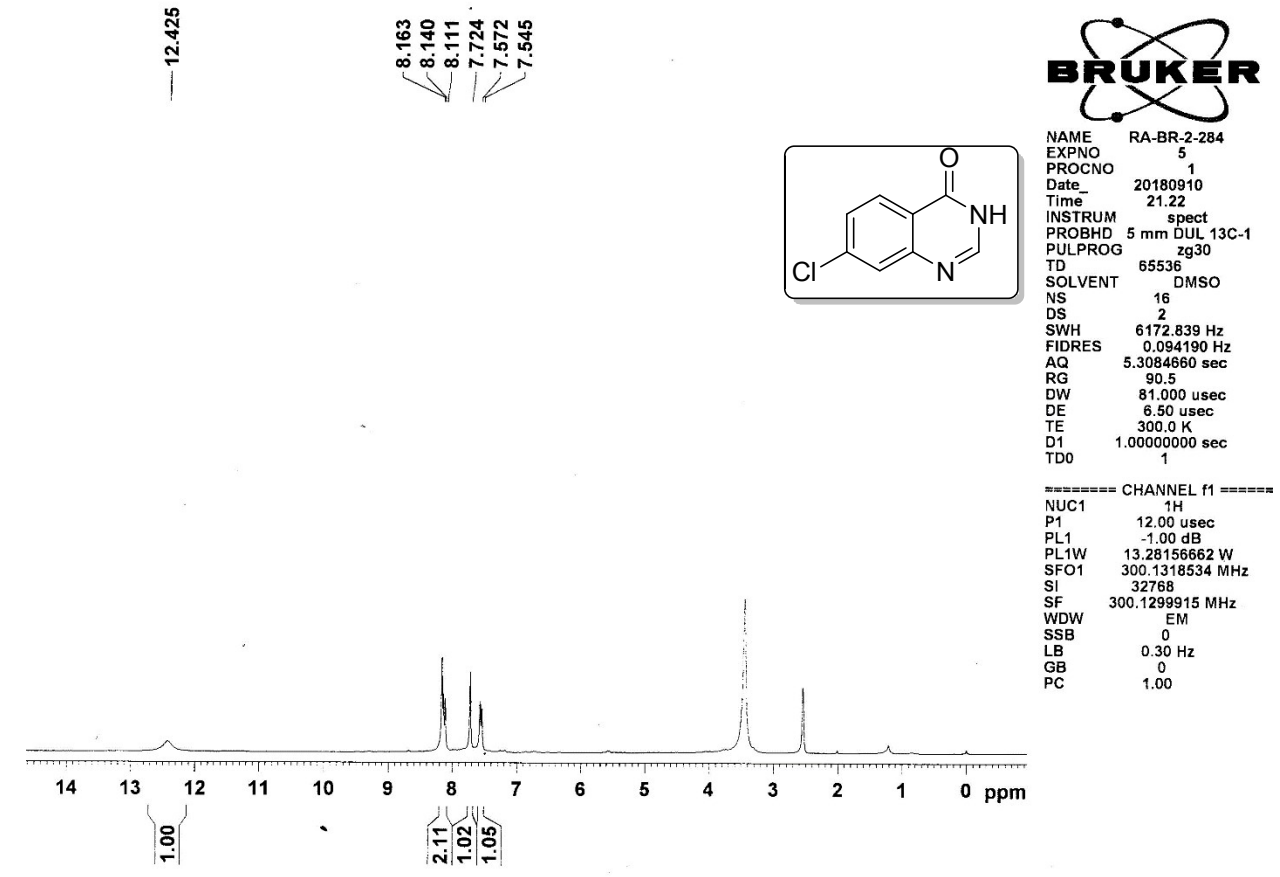
BRUKER

NAME RA-BR-2-315
EXPNO 3
PROCNO 1
Date_ 20180929
Time 20.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2896.3
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.68 usec
PL1 0.00 dB
PL1W 31.39858055 W
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.29870972 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

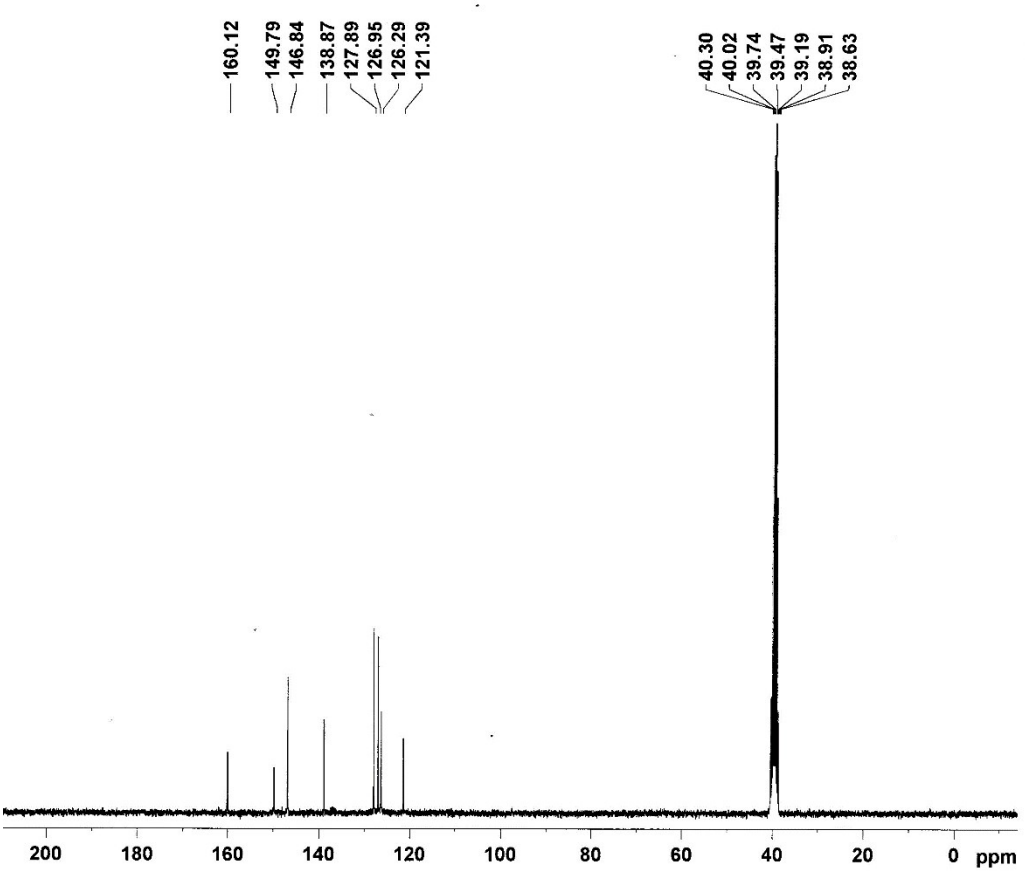
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2d



BRUKER

NAME RA-BR-2-284
EXPNO 5
PROCNO 1
Date_ 20180910
Time 21.22
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 90.5
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1299915 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



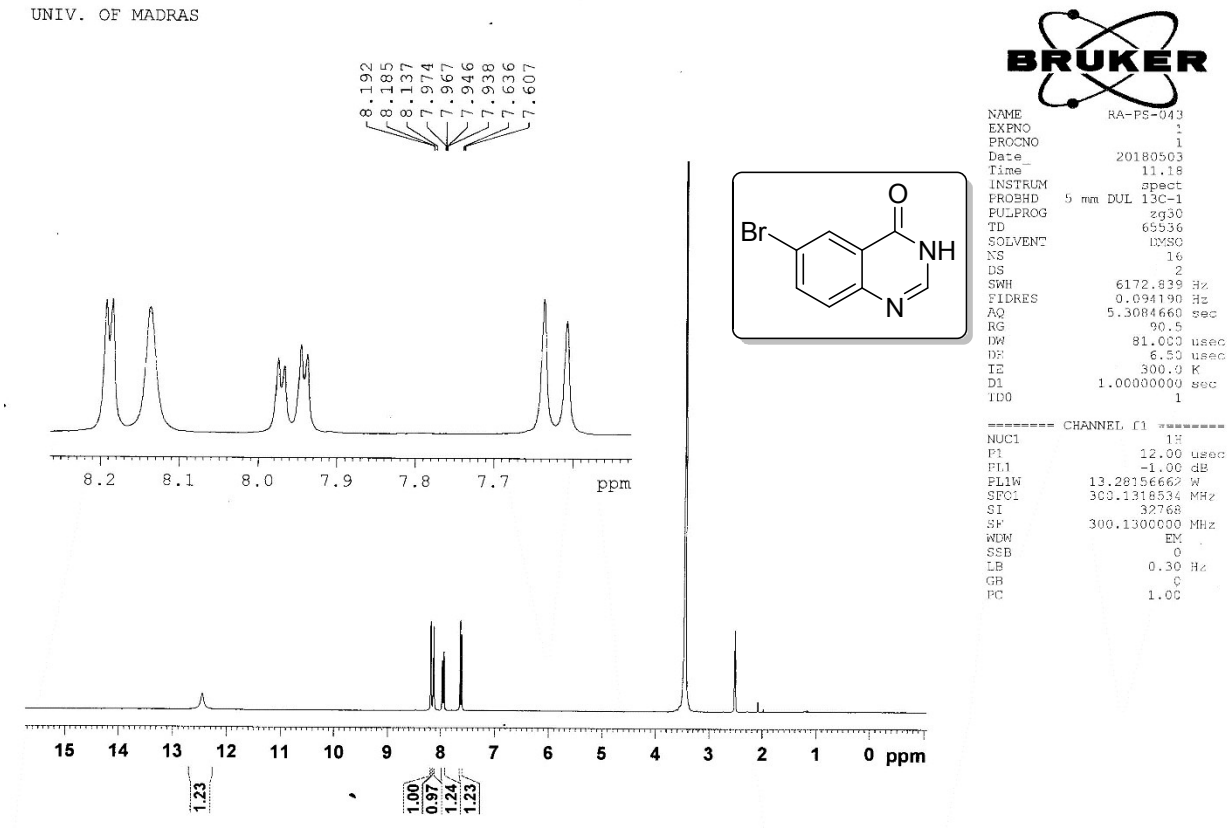
BRUKER

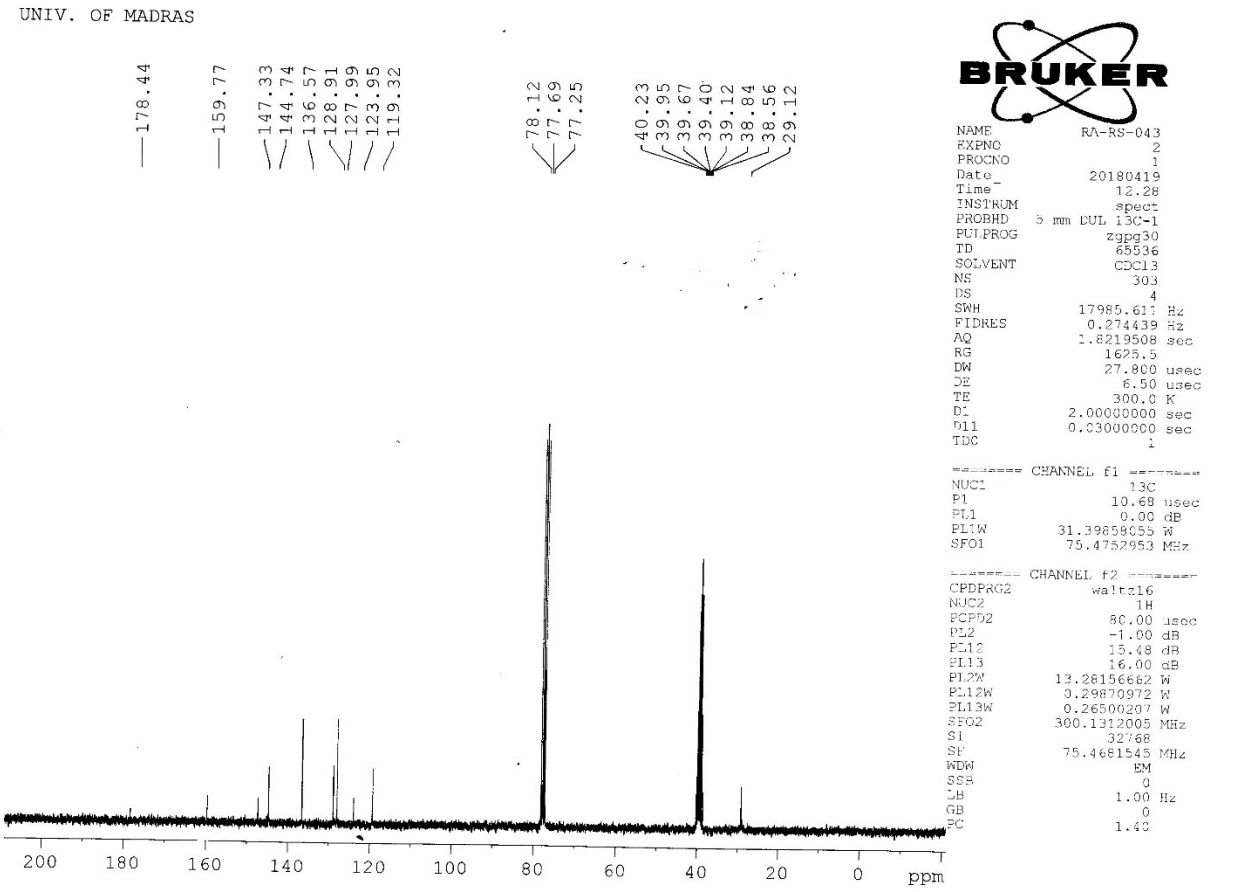
NAME RA-BR-2-284
EXPNO 6
PROCNO 1
Date_ 20180910
Time 21.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 842
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 4096
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ====
NUC1 13C
P1 10.68 usec
PL1 0.00 dB
PL1W 31.39858055 W
SFO1 75.4752953 MHz

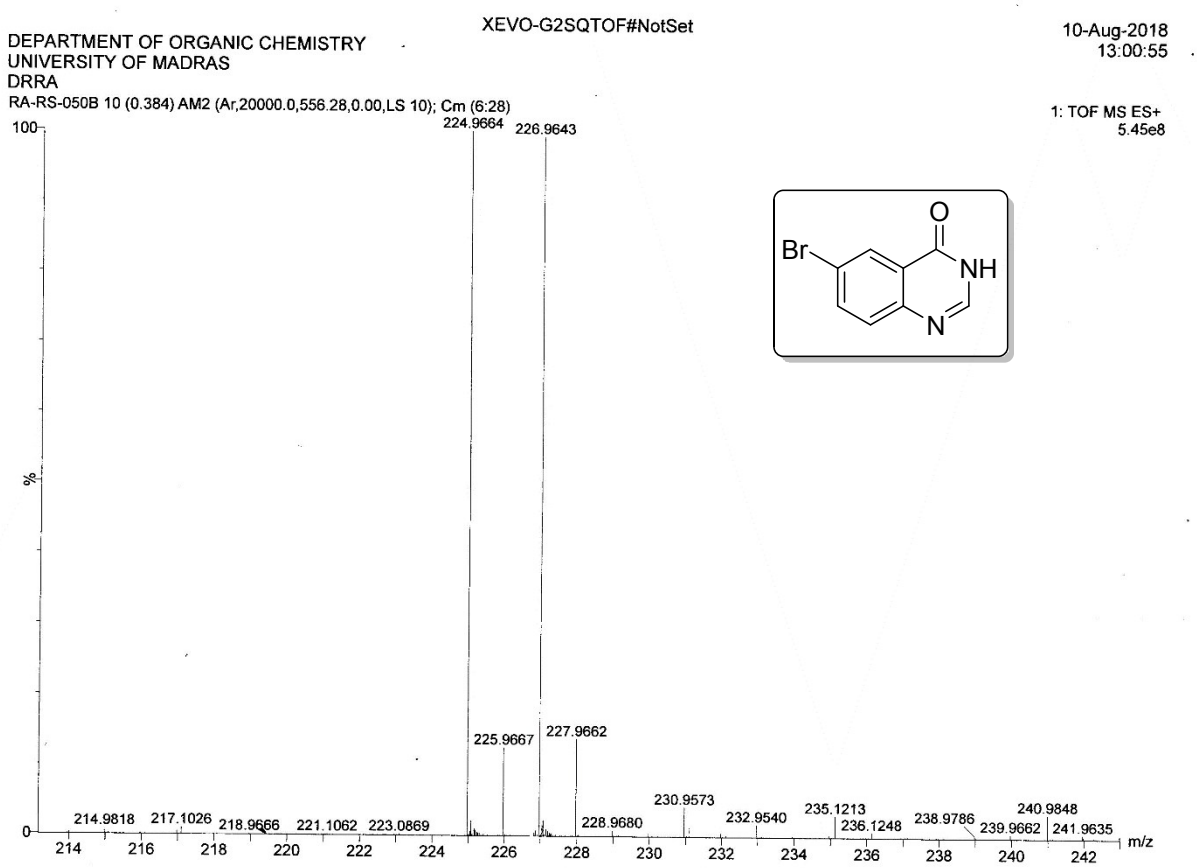
======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.29870972 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2e

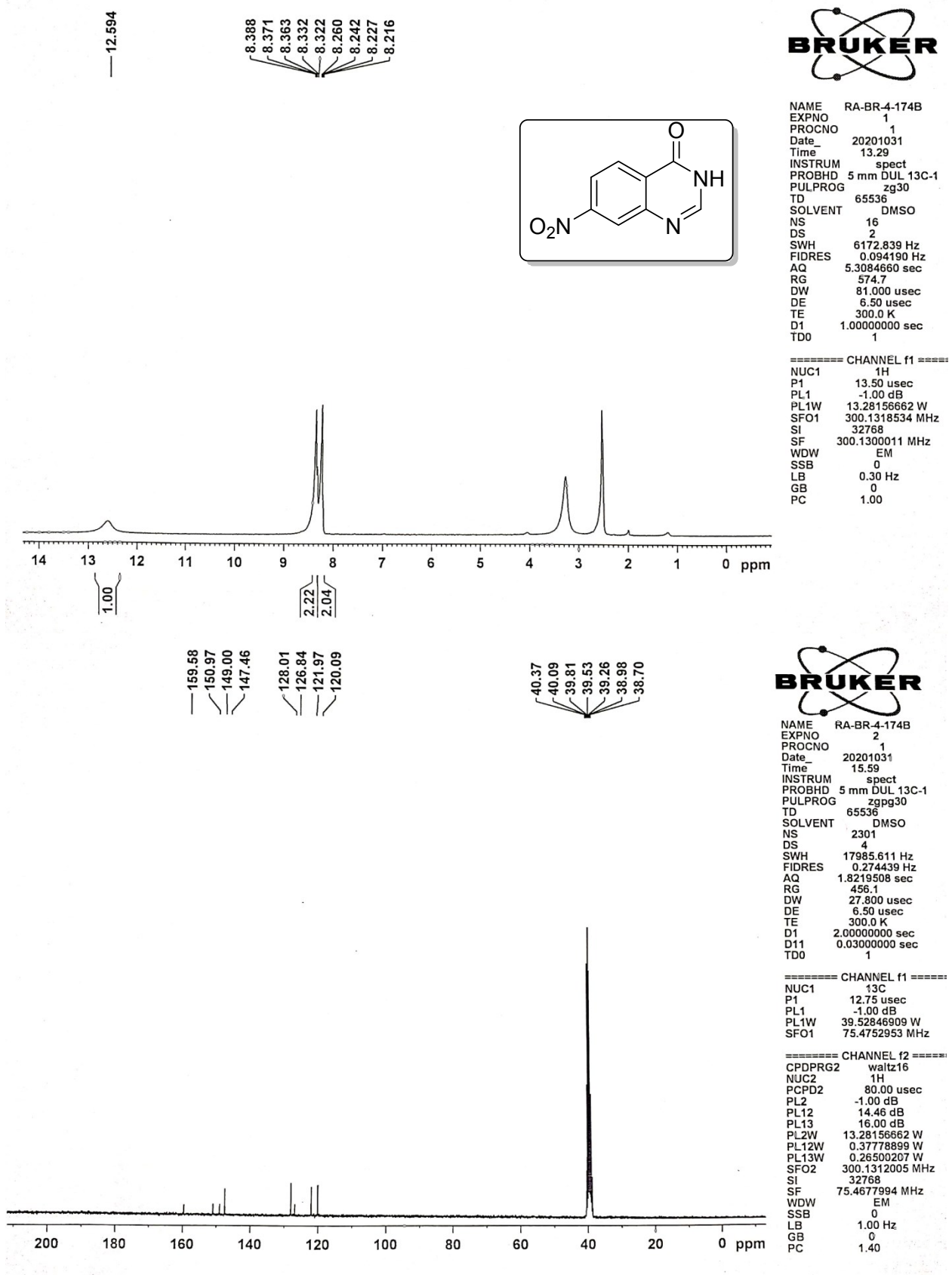




^{1}H & ^{13}C NMR (DMSO-d_6) spectra of compound 2f



HRMS spectrum of compound 2f



BRUKER

NAME	RA-BR-4-174B
EXPNO	1
PROCNO	1
Date_	20201031
Time	13.29
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	574.7
DW	81.000 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ======

NUC1	1H
P1	13.50 usec
PL1	-1.00 dB
PL1W	13.28156662 W
SFO1	300.1318534 MHz
SI	32768
SF	300.1300011 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

BRUKER

NAME	RA-BR-4-174B
EXPNO	2
PROCNO	1
Date_	20201031
Time	15.59
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zgpg30
TD	65536
SOLVENT	DMSO
NS	2301
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	456.1
DW	27.800 usec
DE	6.50 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

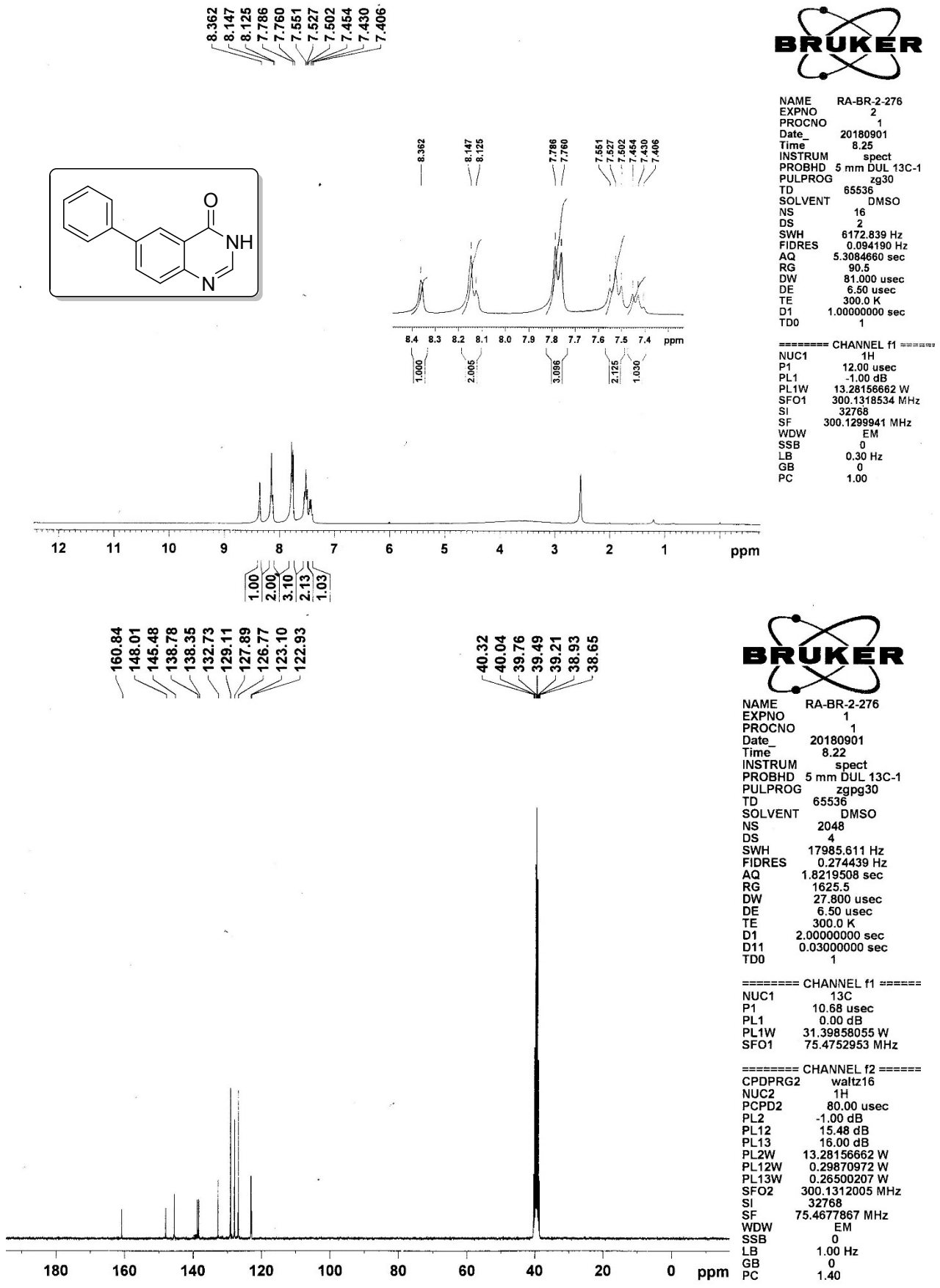
======== CHANNEL f1 ======

NUC1	13C
P1	12.75 usec
PL1	-1.00 dB
PL1W	39.52846909 W
SFO1	75.4752953 MHz

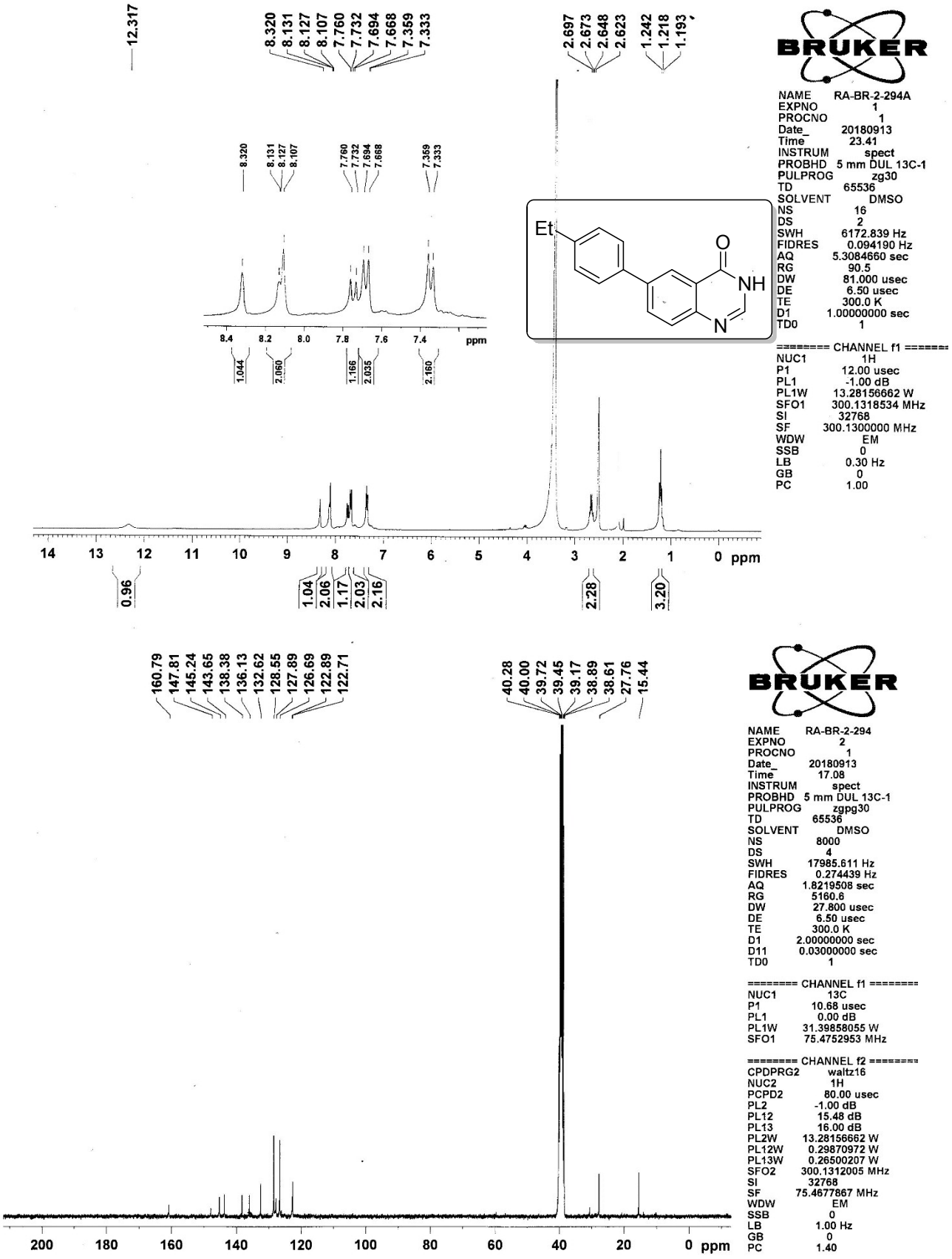
======== CHANNEL f2 ======

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.46 dB
PL13	16.00 dB
PL2W	13.28156662 W
PL12W	0.37778899 W
PL13W	0.26500207 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677994 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

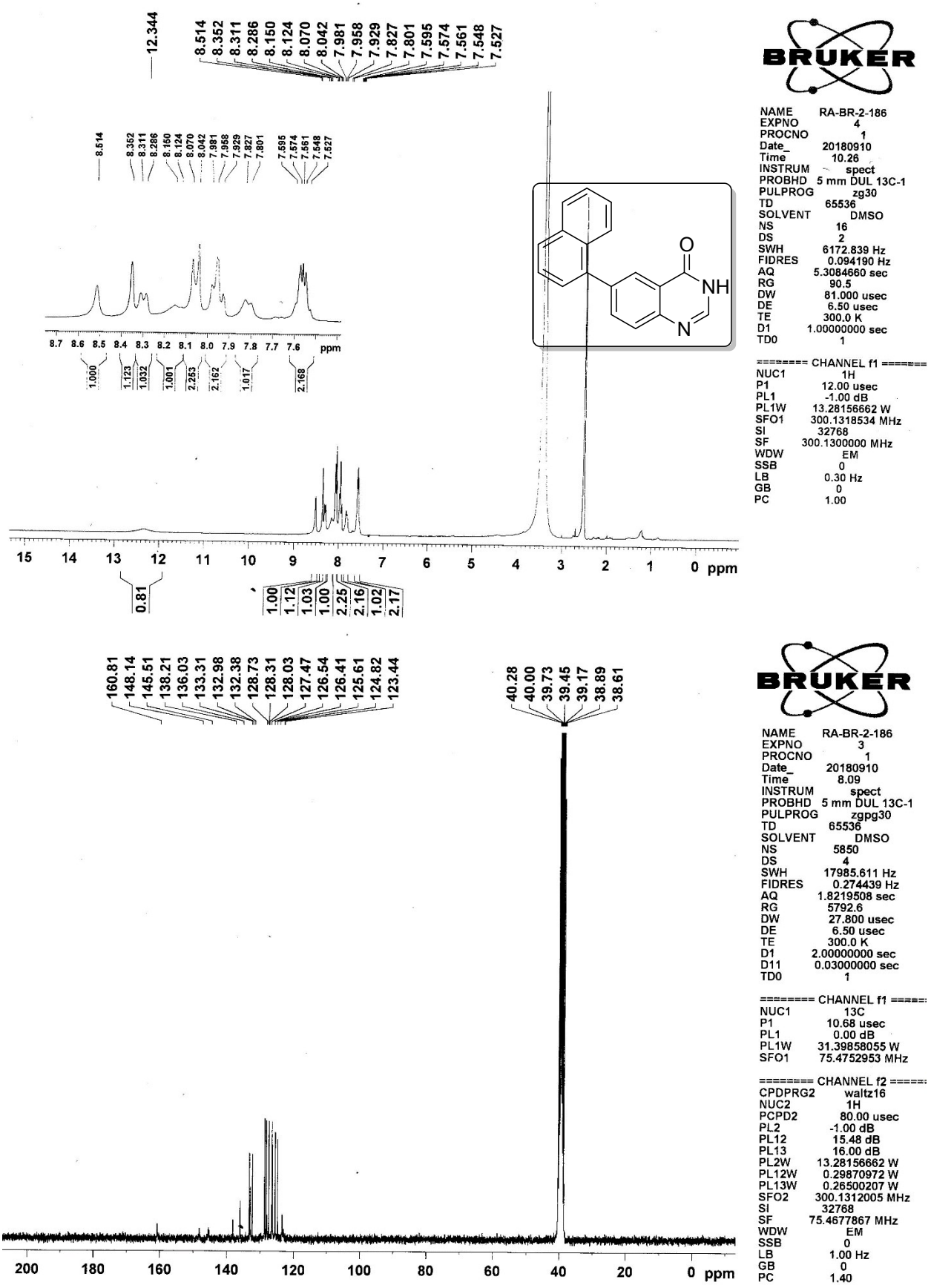
^{1}H & ^{13}C NMR (DMSO-d_6) spectra of compound 2g



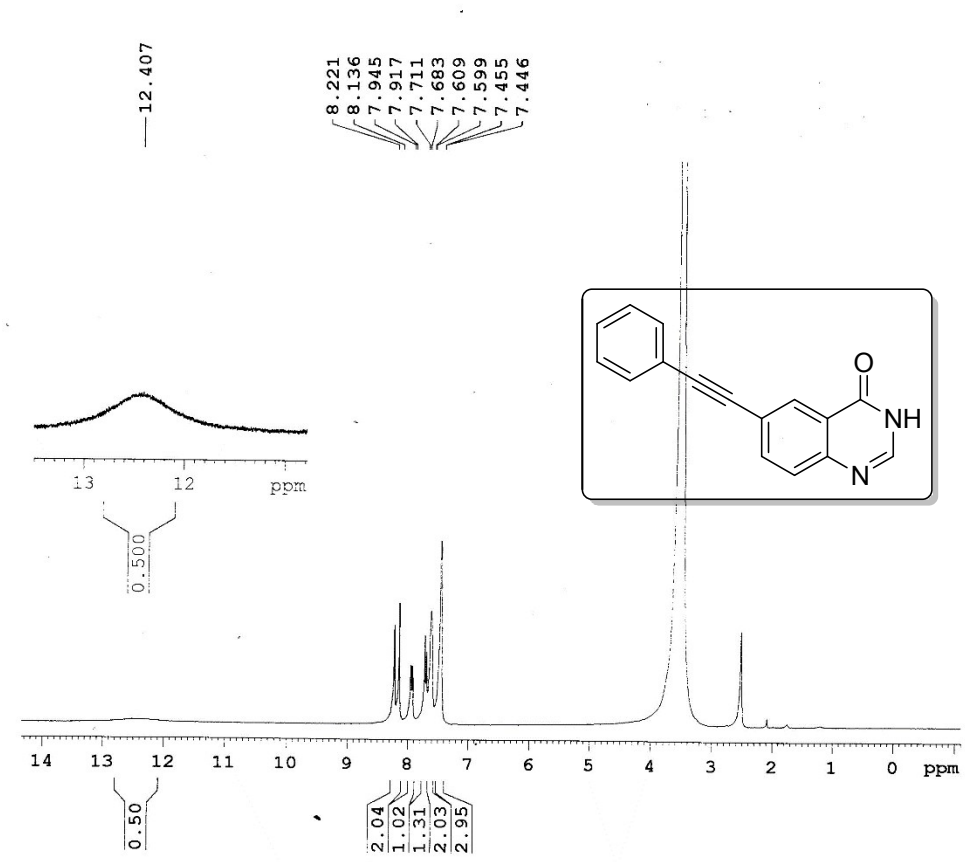
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2h



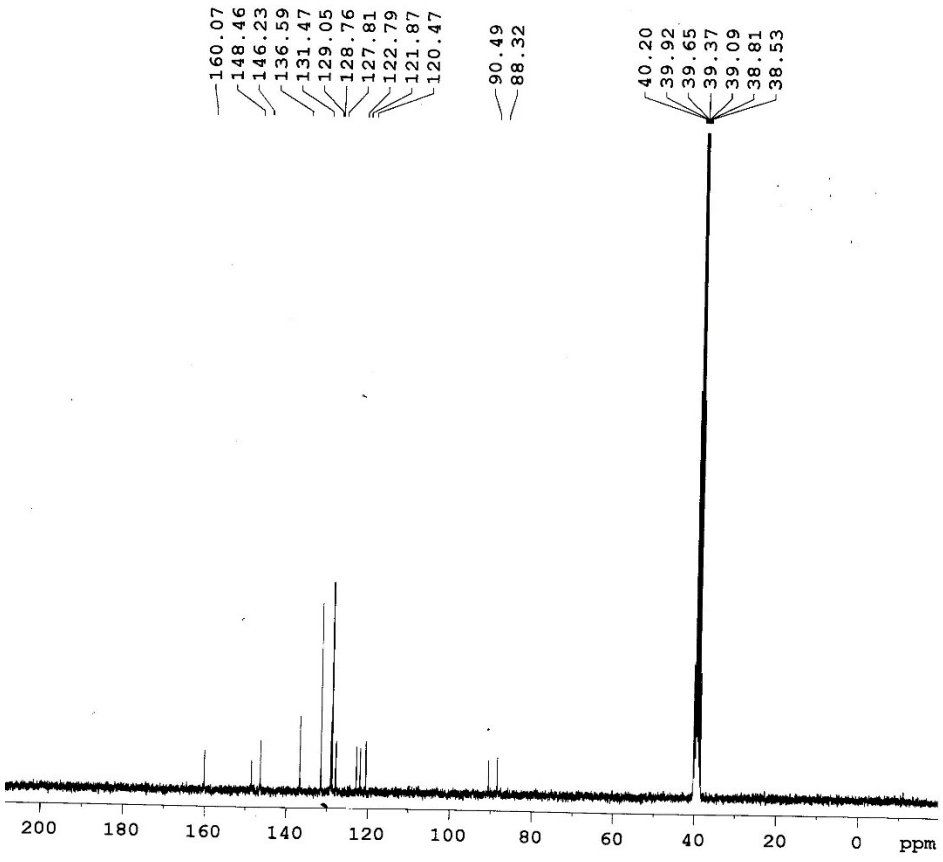
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2i



^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2j

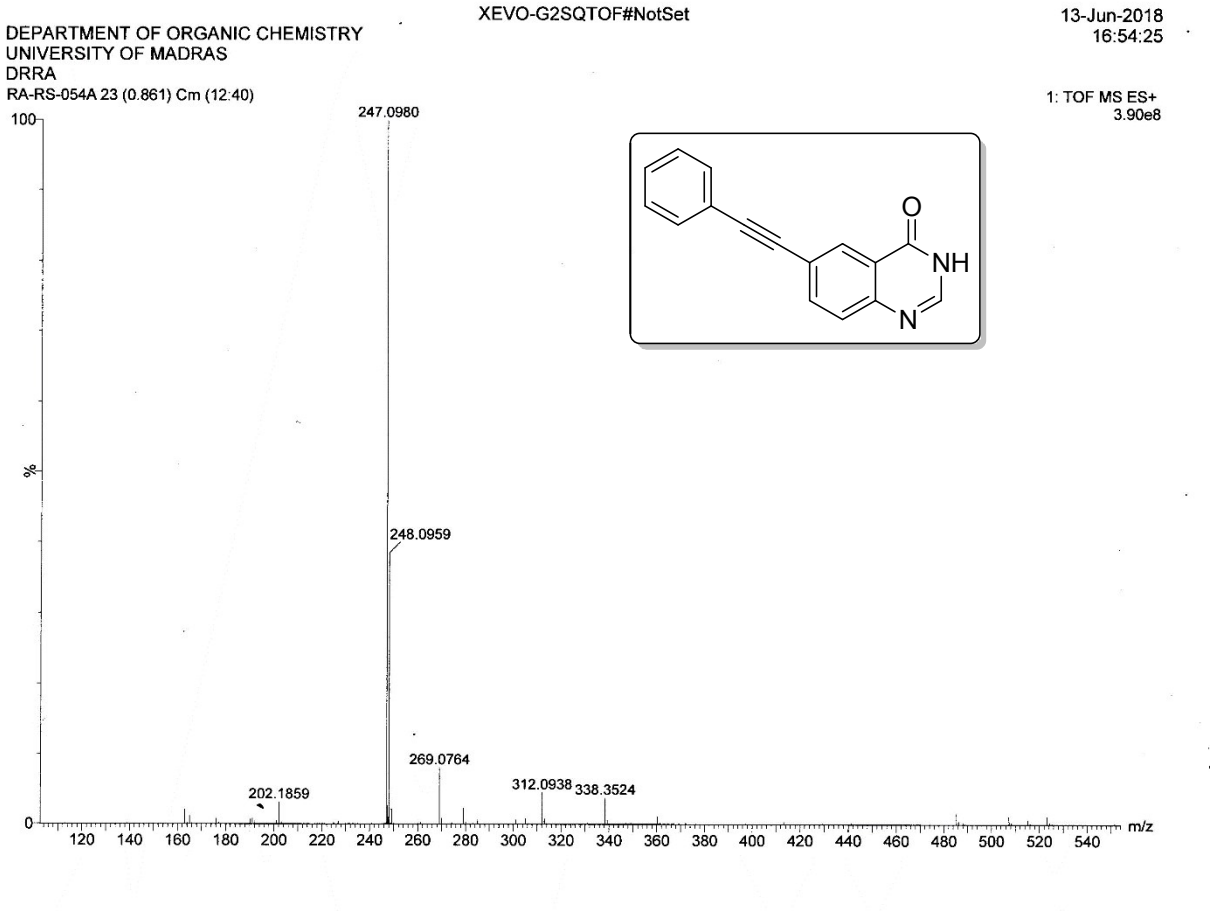



```
NAME            RA-RS-054A
EXPNO                    1
PROCNO                   1
Date_             20180522
Time                 23.03
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH              6172.839 Hz
FIDRES           0.094190 Hz
AQ               5.3084660 sec
RG                     64
DW                 81.000 usec
DE                  6.50 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 12.00 usec
PL1                -1.00 dB
PL1W        13.28156662 W
SFO1        300.1318534 MHz
SI                  32768
SF          300.1300000 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

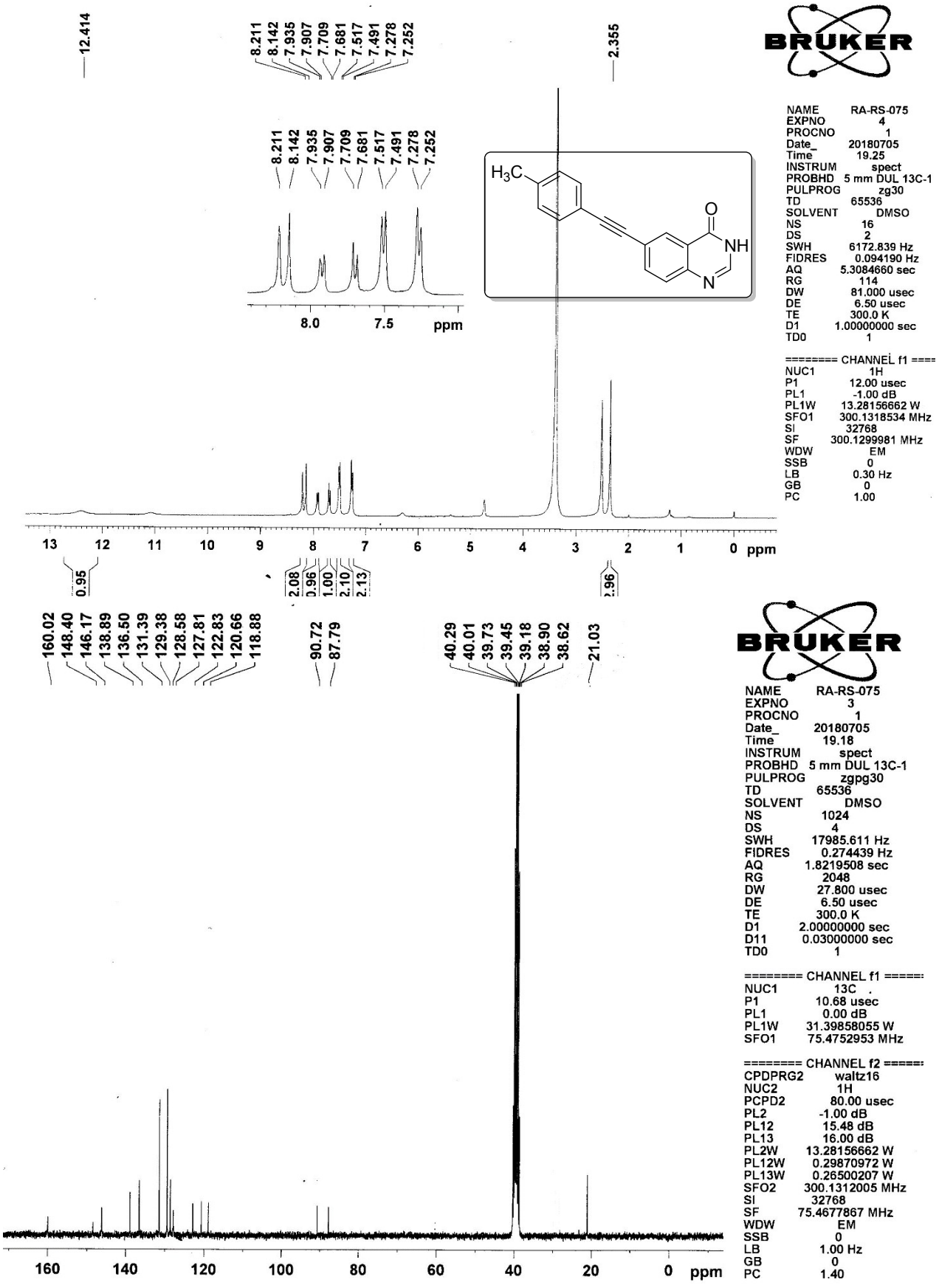



```
NAME            RA-RS-054 A
EXPNO                    2
PROCNO                   1
Date_             20180523
Time                 14.59
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                    783
DS                      4
SWH             17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                  1625.5
DW                 27.800 usec
DE                  6.50 usec
TE                 300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                 10.68 usec
PL1                 0.00 dB
PL1W        31.39858055 W
SFO1        75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               15.48 dB
PL13               18.00 dB
PL2W        13.28156662 W
PL12W        0.29870972 W
PL13W        0.26500207 W
SFO2        300.1312005 MHz
SI                  32768
SF          75.4677867 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```

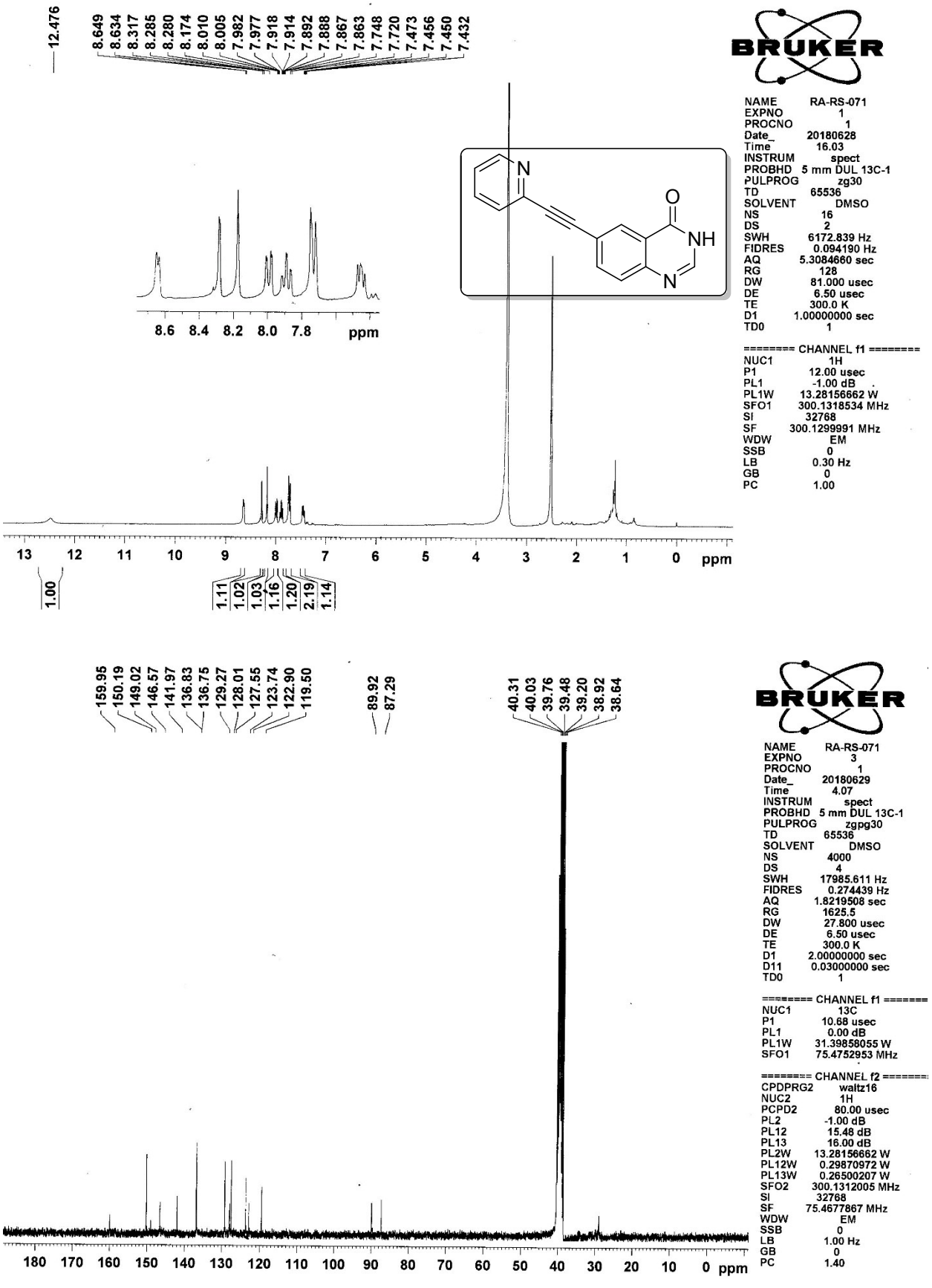
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2k



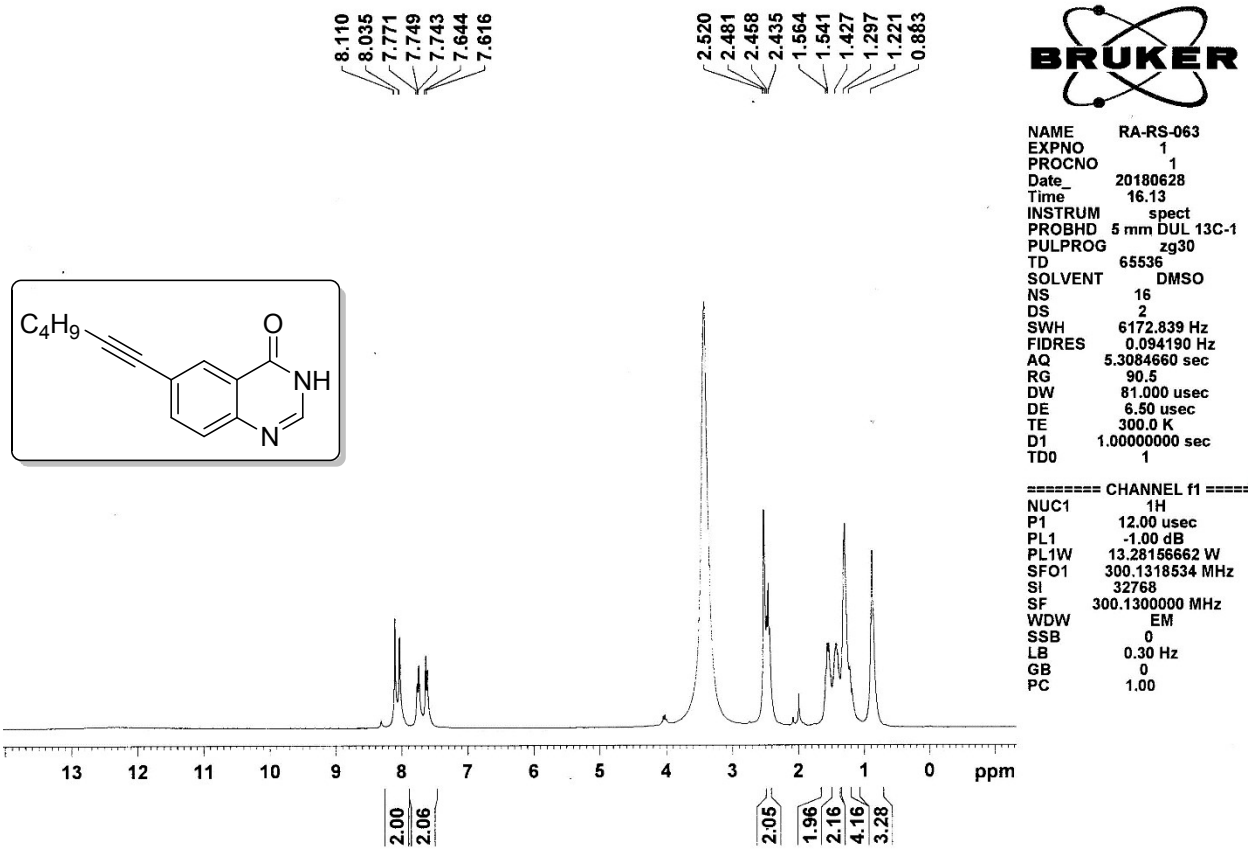
HRMS spectrum of compound 2k

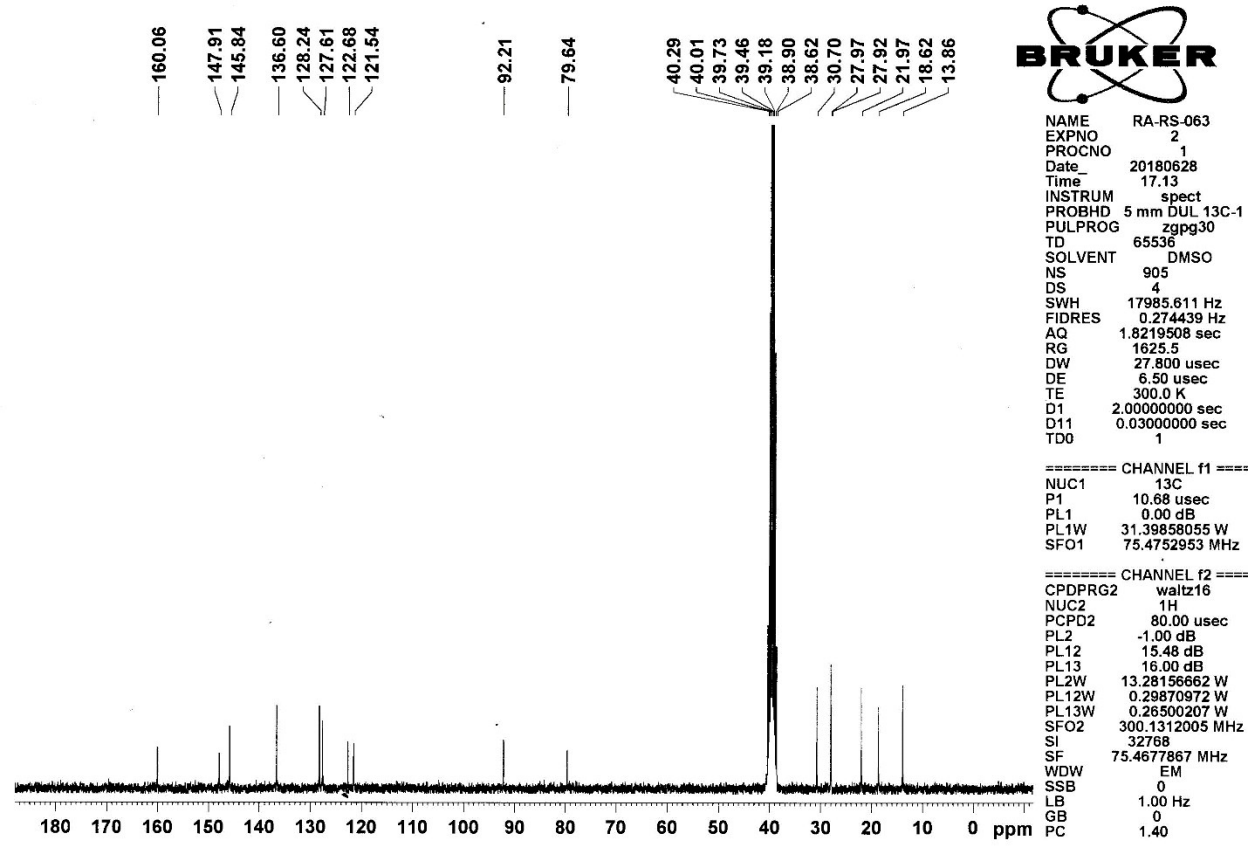


^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2l

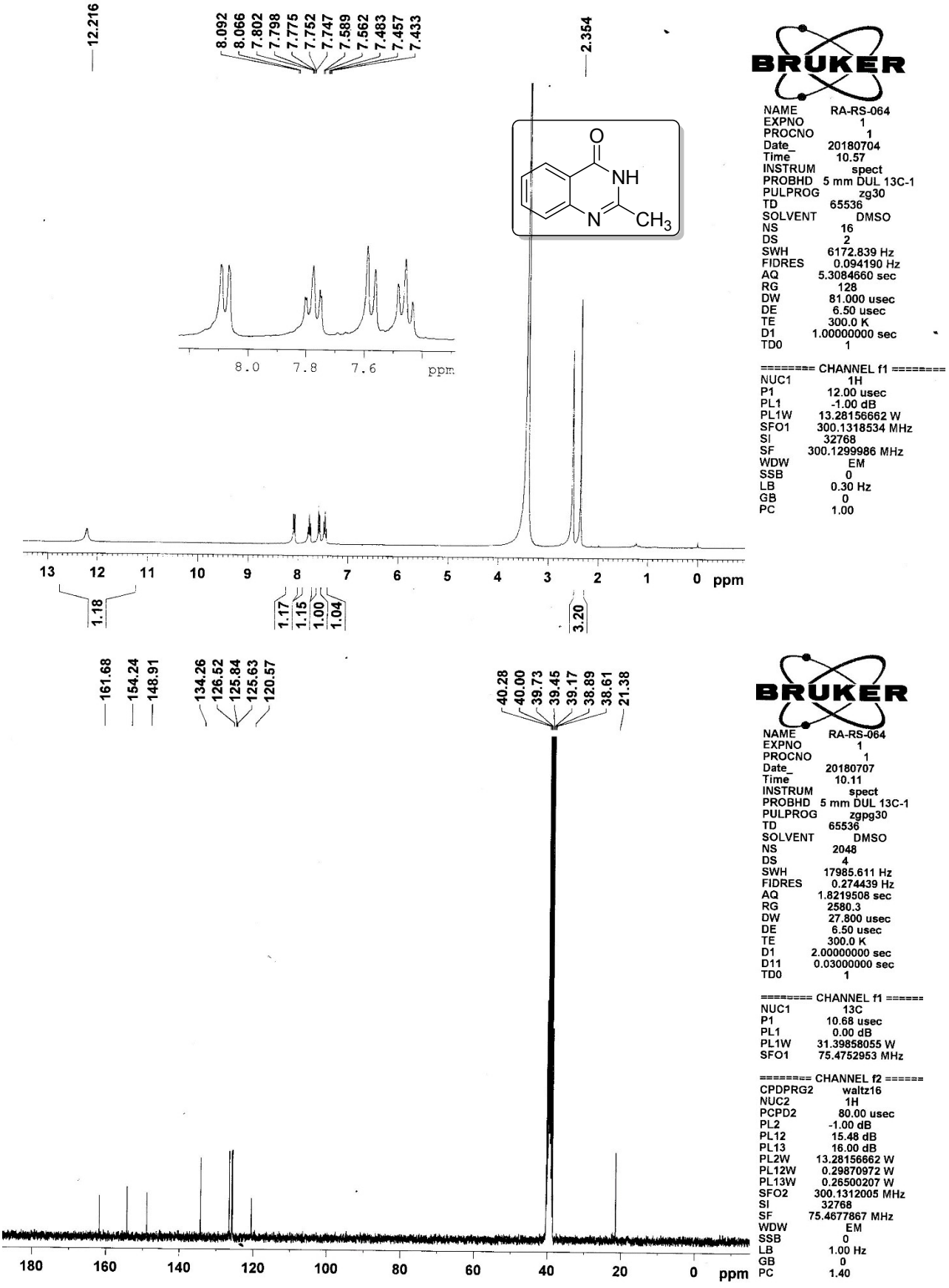


^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2m

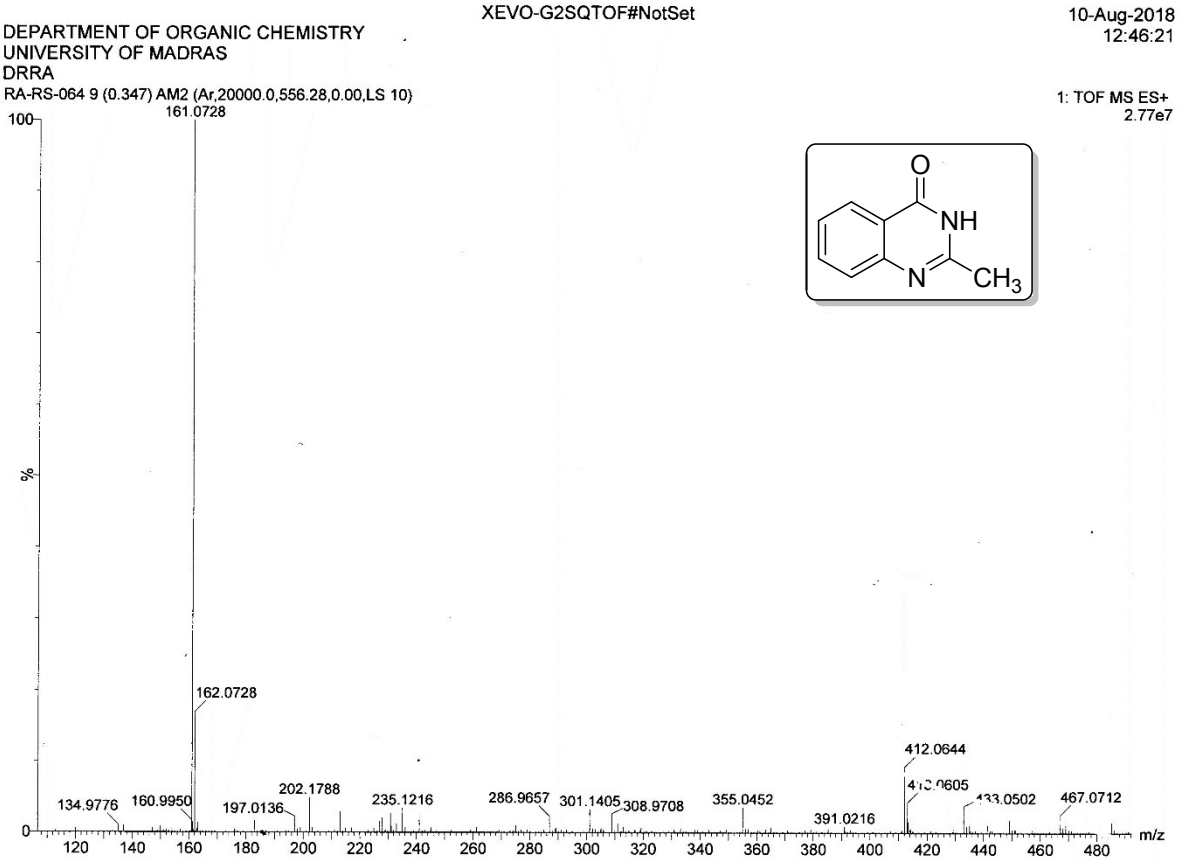




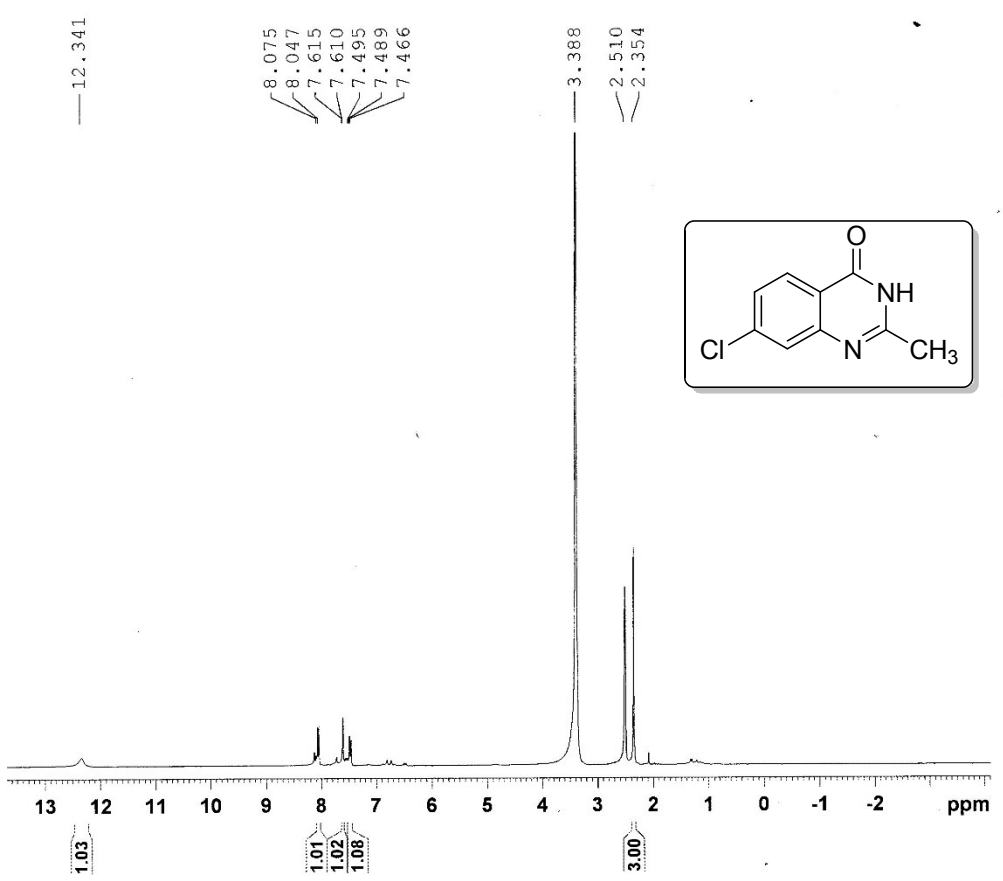
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 2n



^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 3a



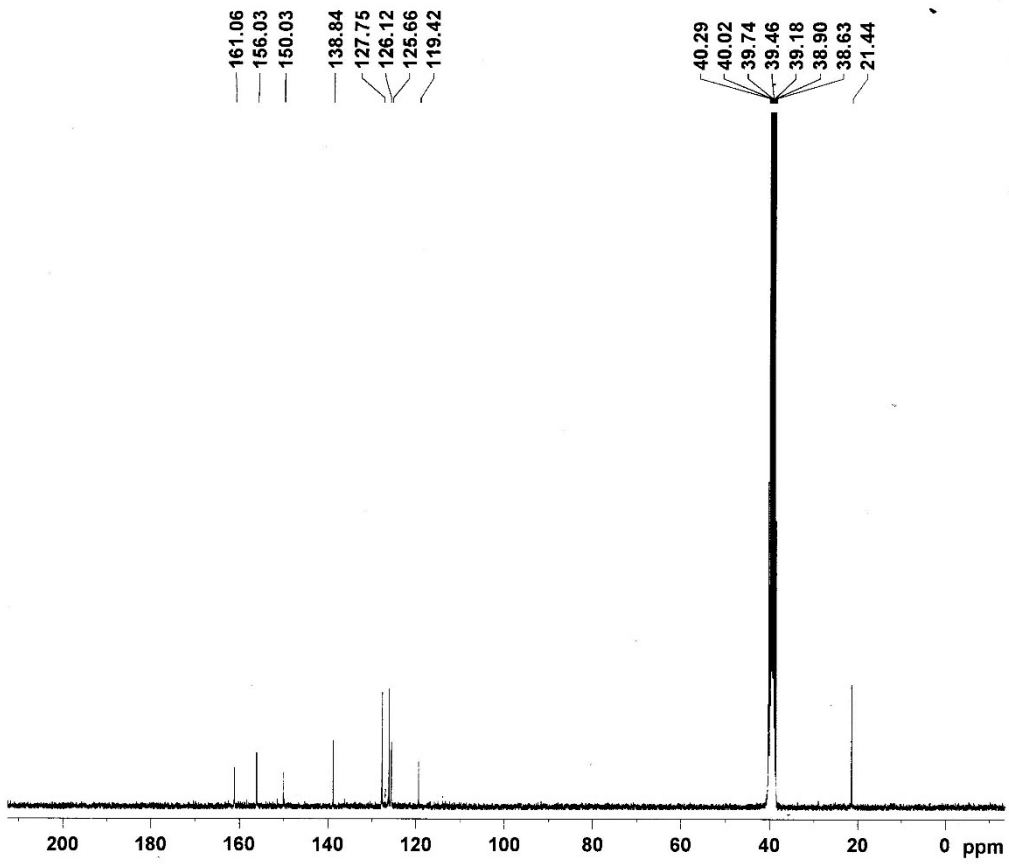
HRMS spectrum of compound 3a



BRUKER

NAME RA-BR-2-321
EXPNO 4
PROCNO 1
Date_ 20180930
Time 6.28
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 128
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



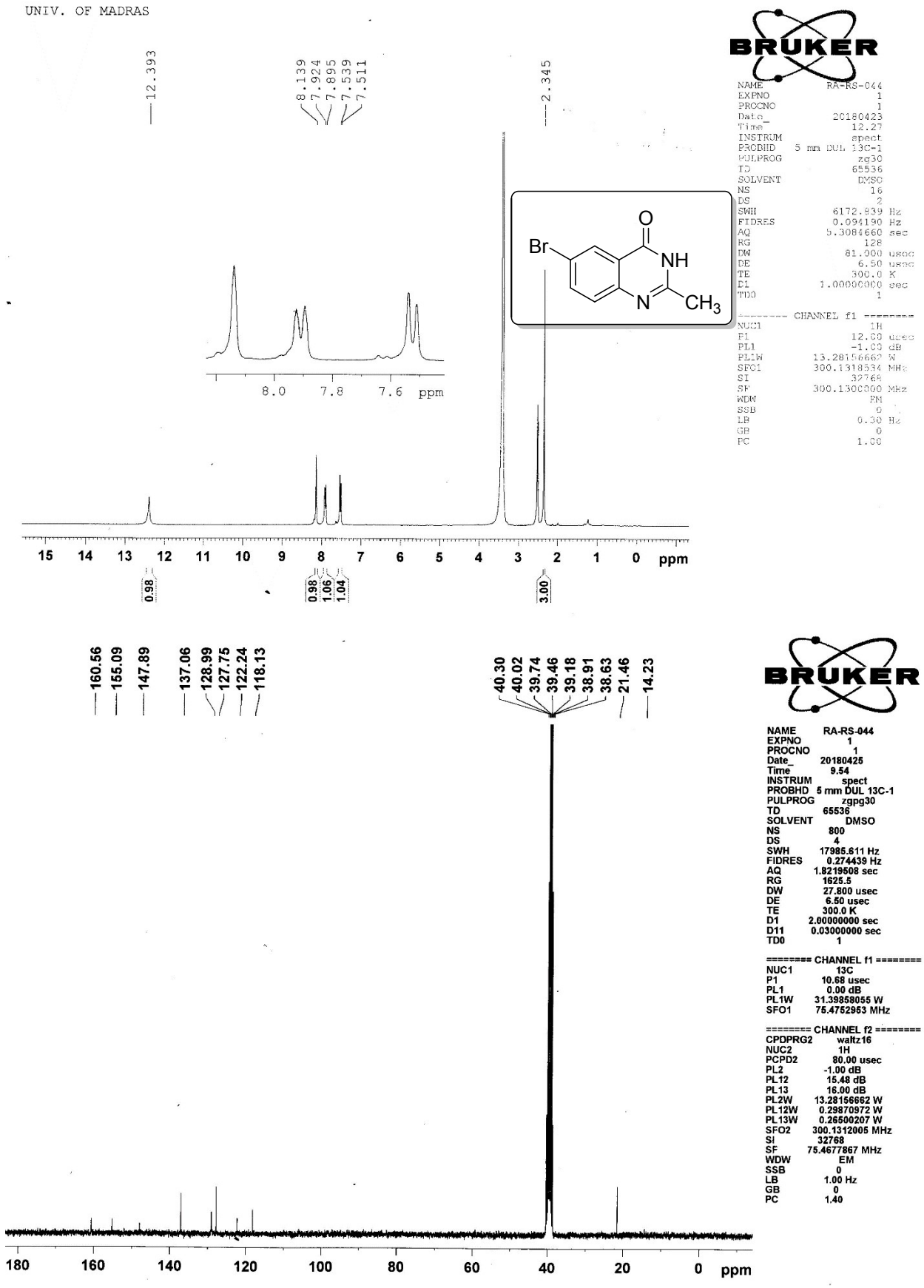
BRUKER

NAME RA-BR-2-321
EXPNO 3
PROCNO 1
Date_ 20180930
Time 6.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 7000
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2896.3
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

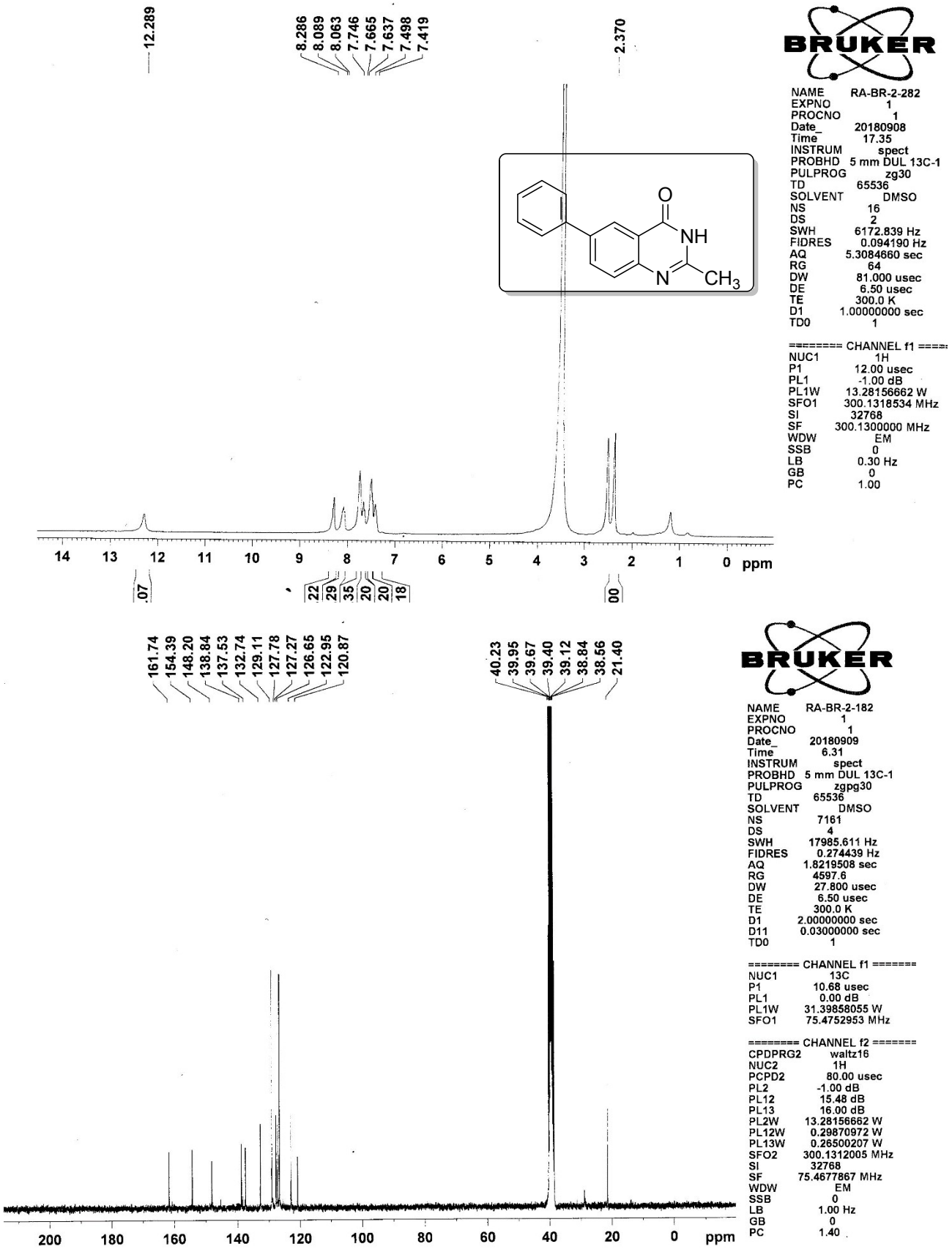
======== CHANNEL f1 ======
NUC1 13C
P1 10.68 usec
PL1 0.00 dB
PL1W 31.39858055 W
SFO1 75.4752953 MHz

======== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.29870972 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

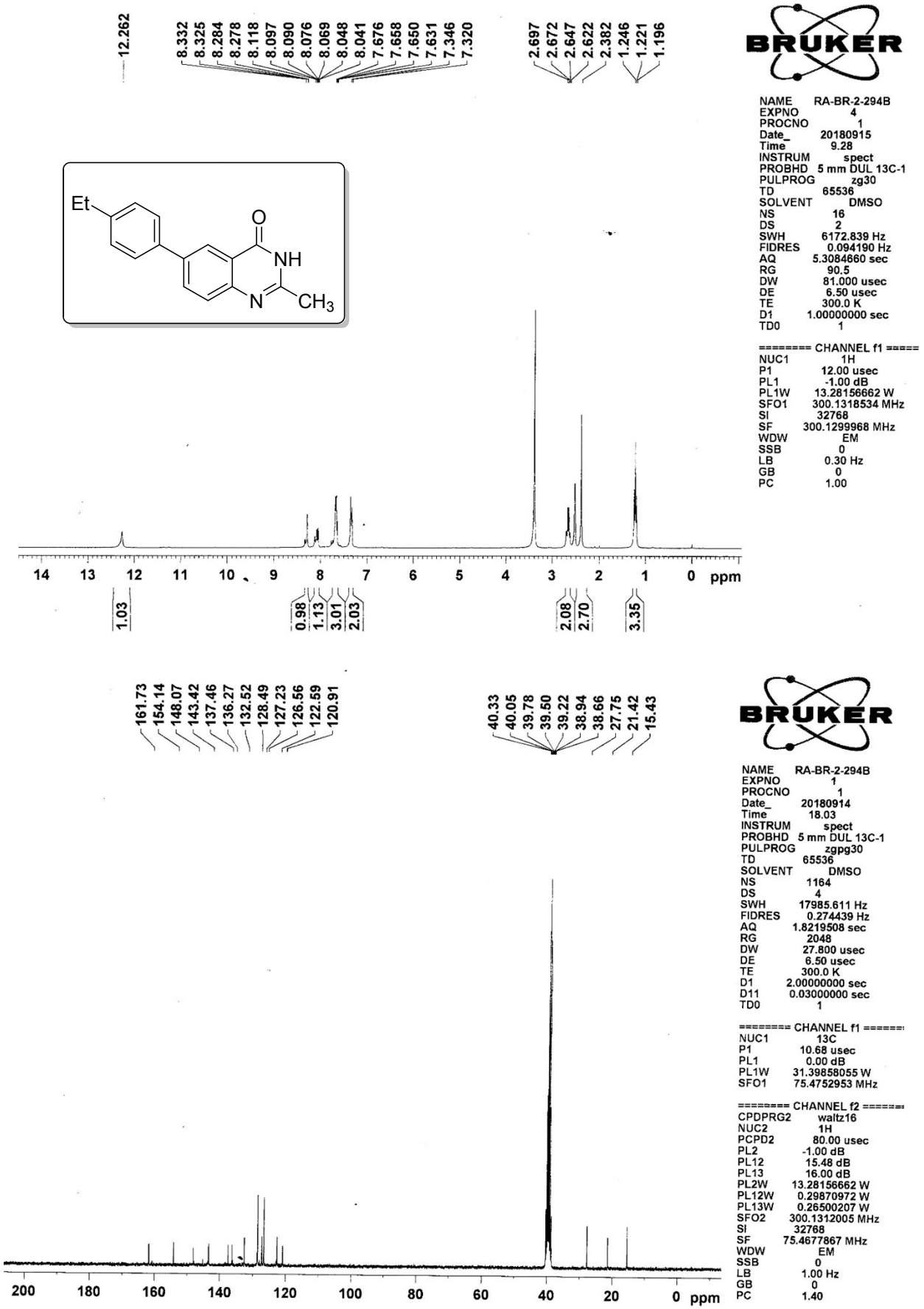
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 3b



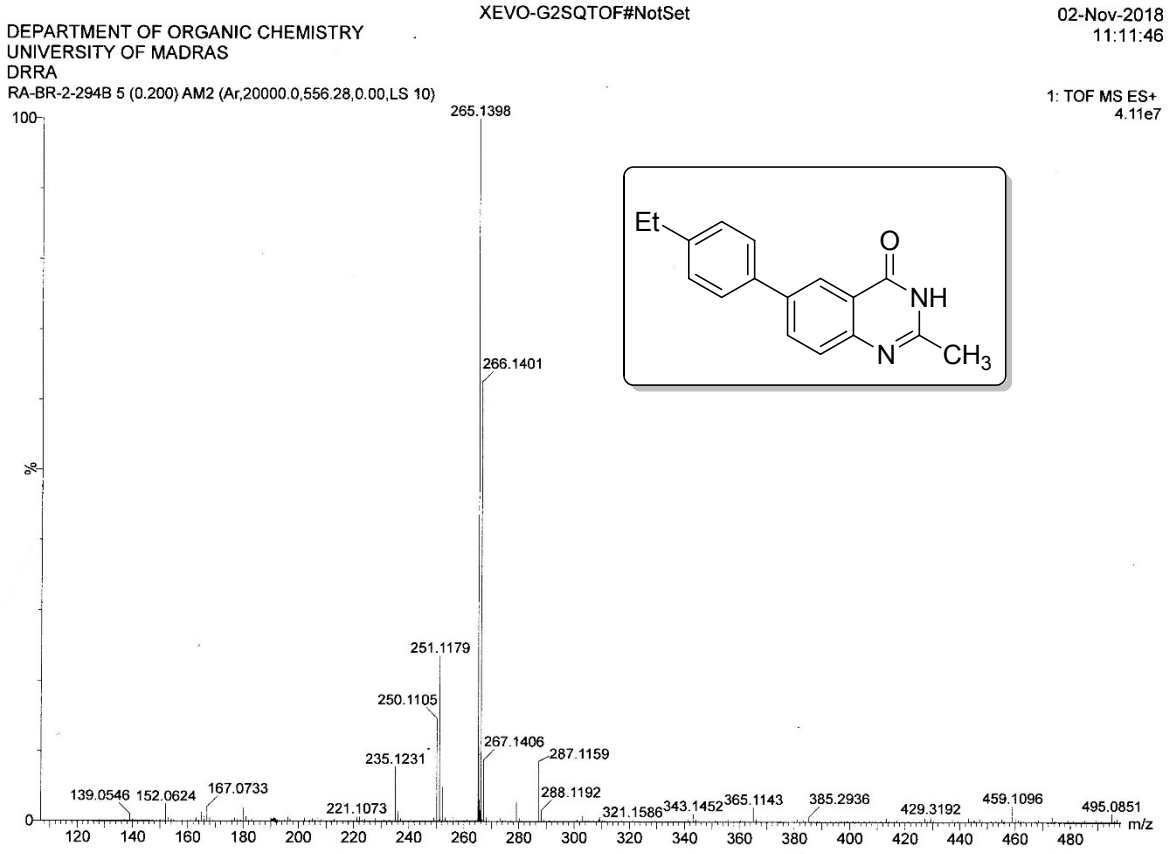
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 3c



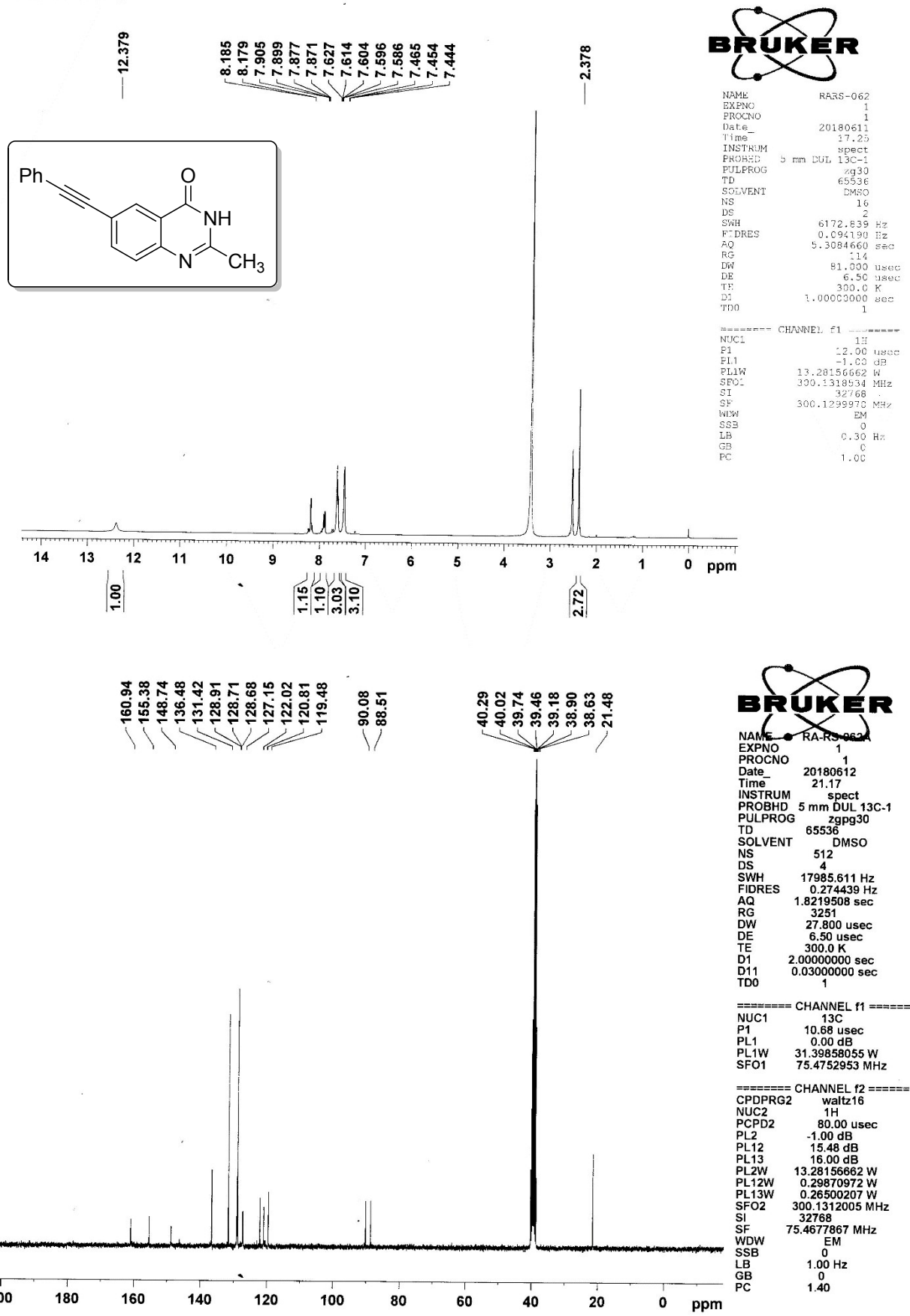
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 3e



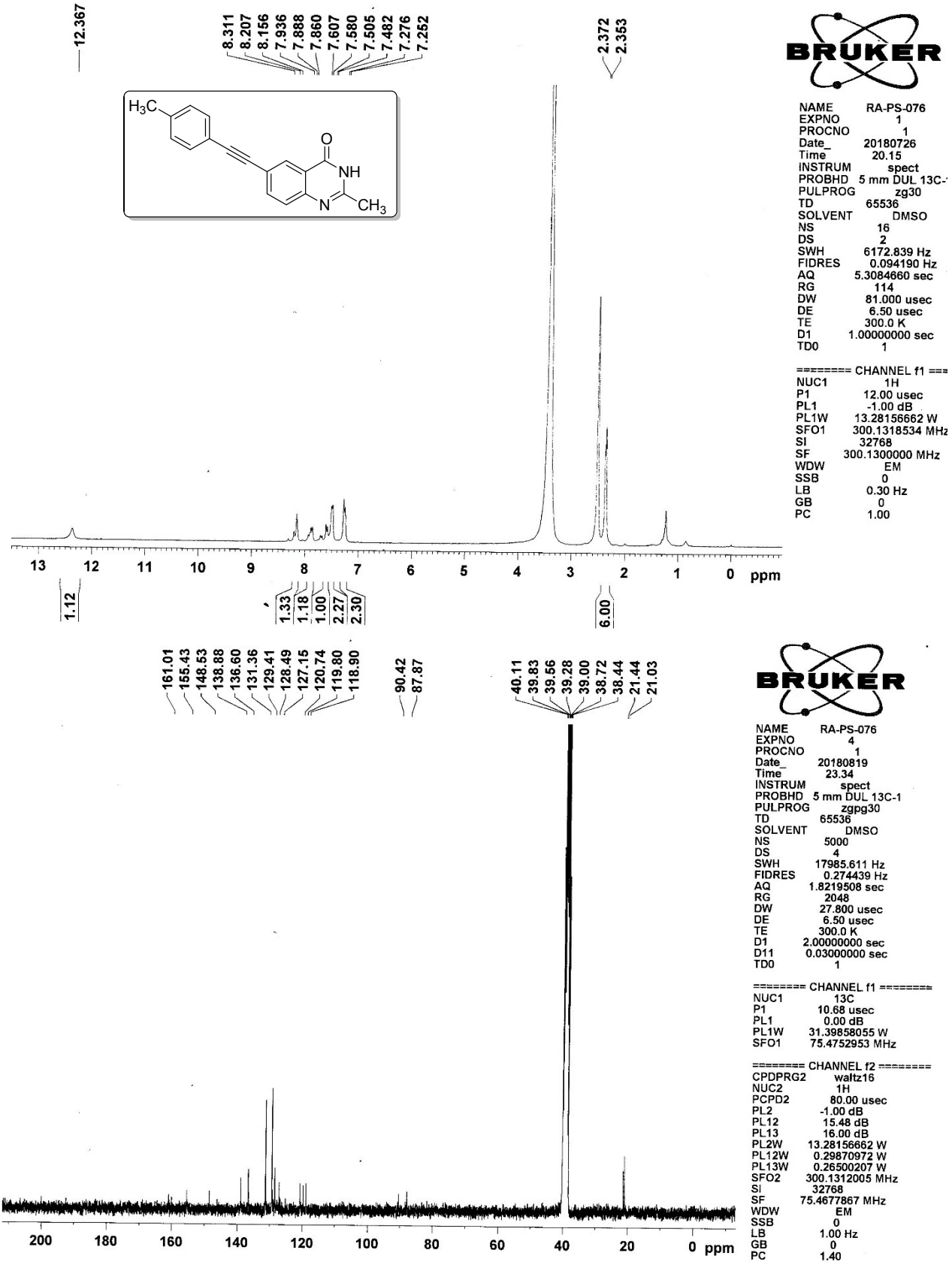
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 3f



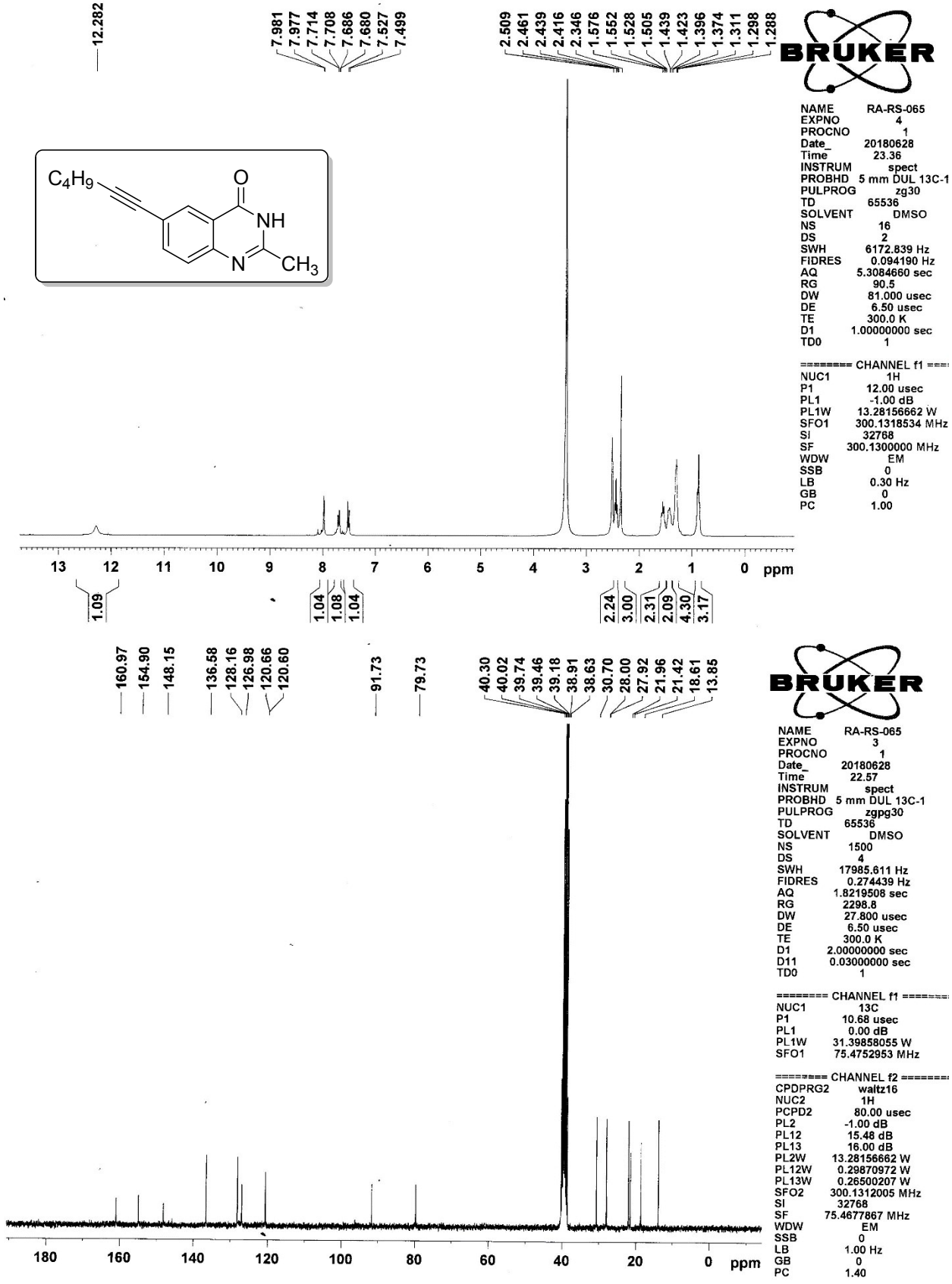
HRMS spectrum of compound 3f



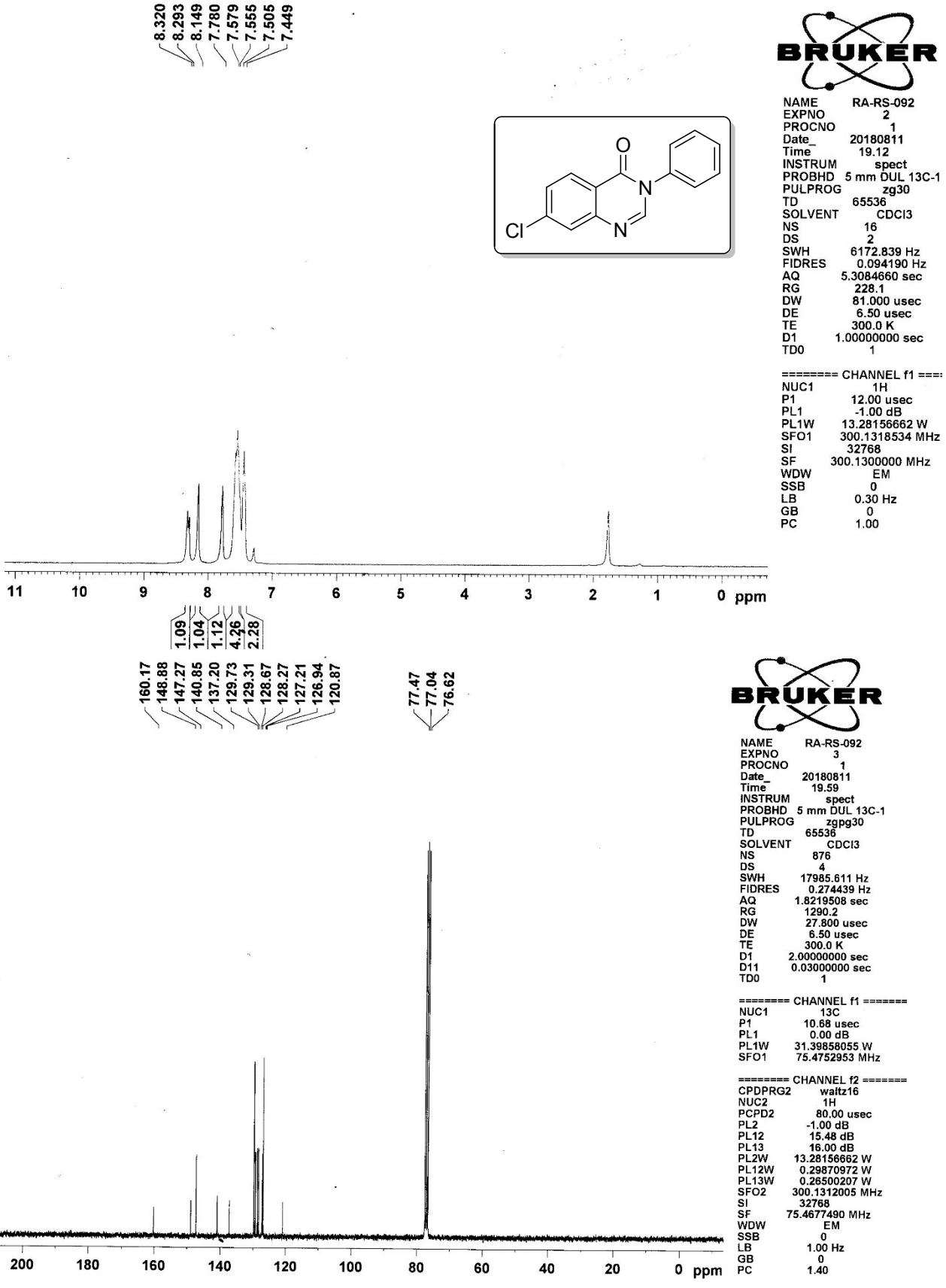
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 3g



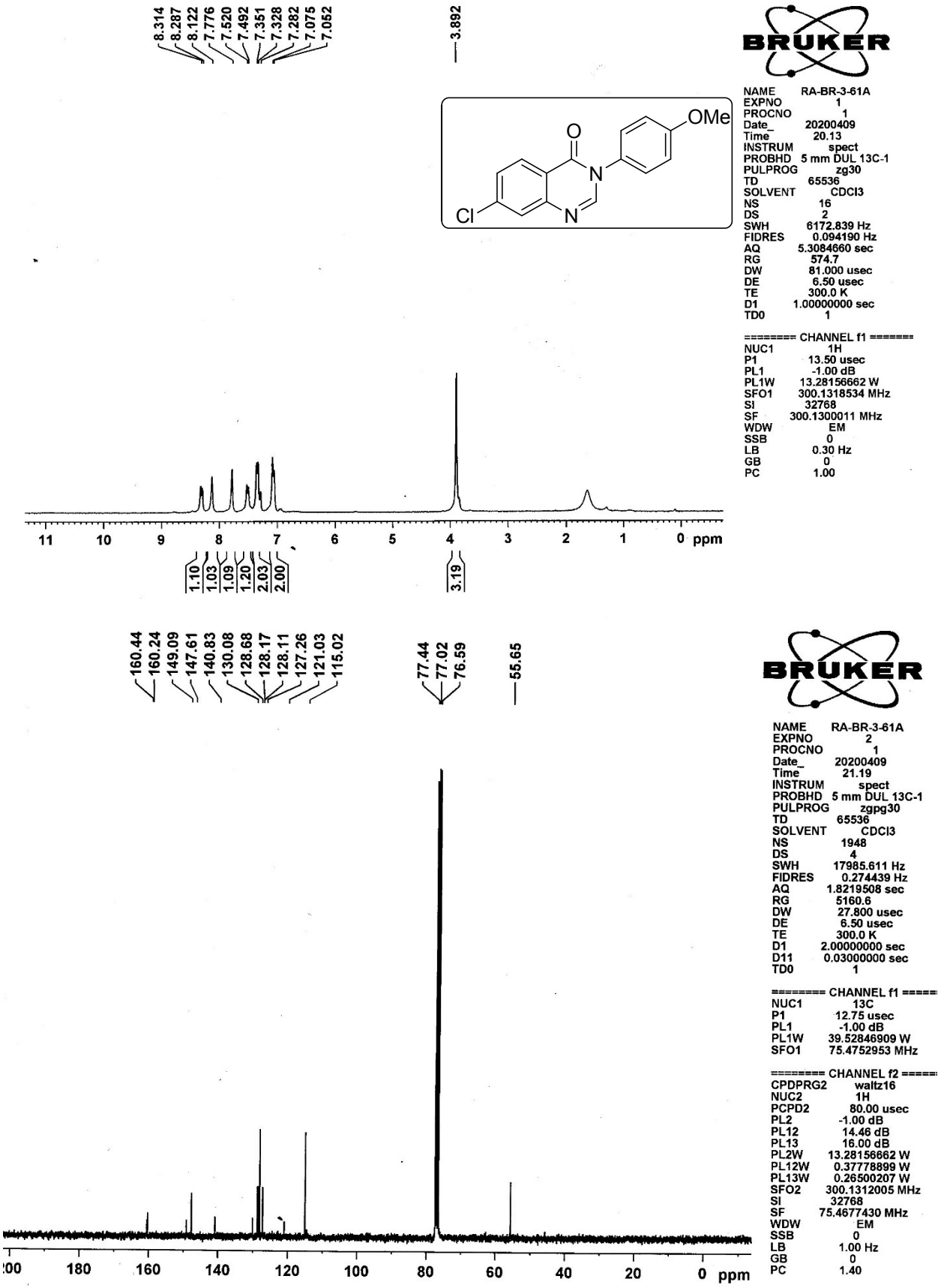
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 3h



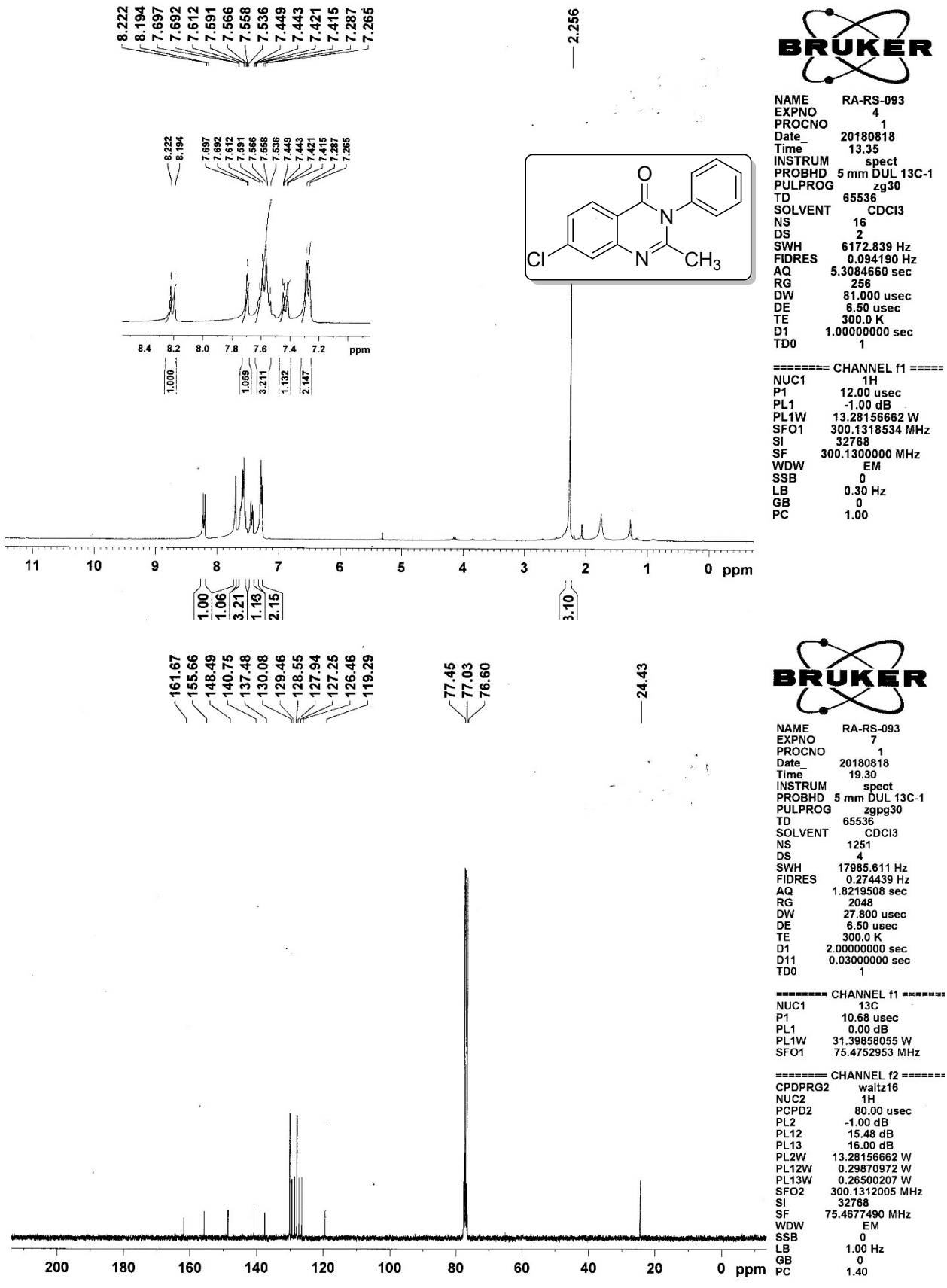
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 3i



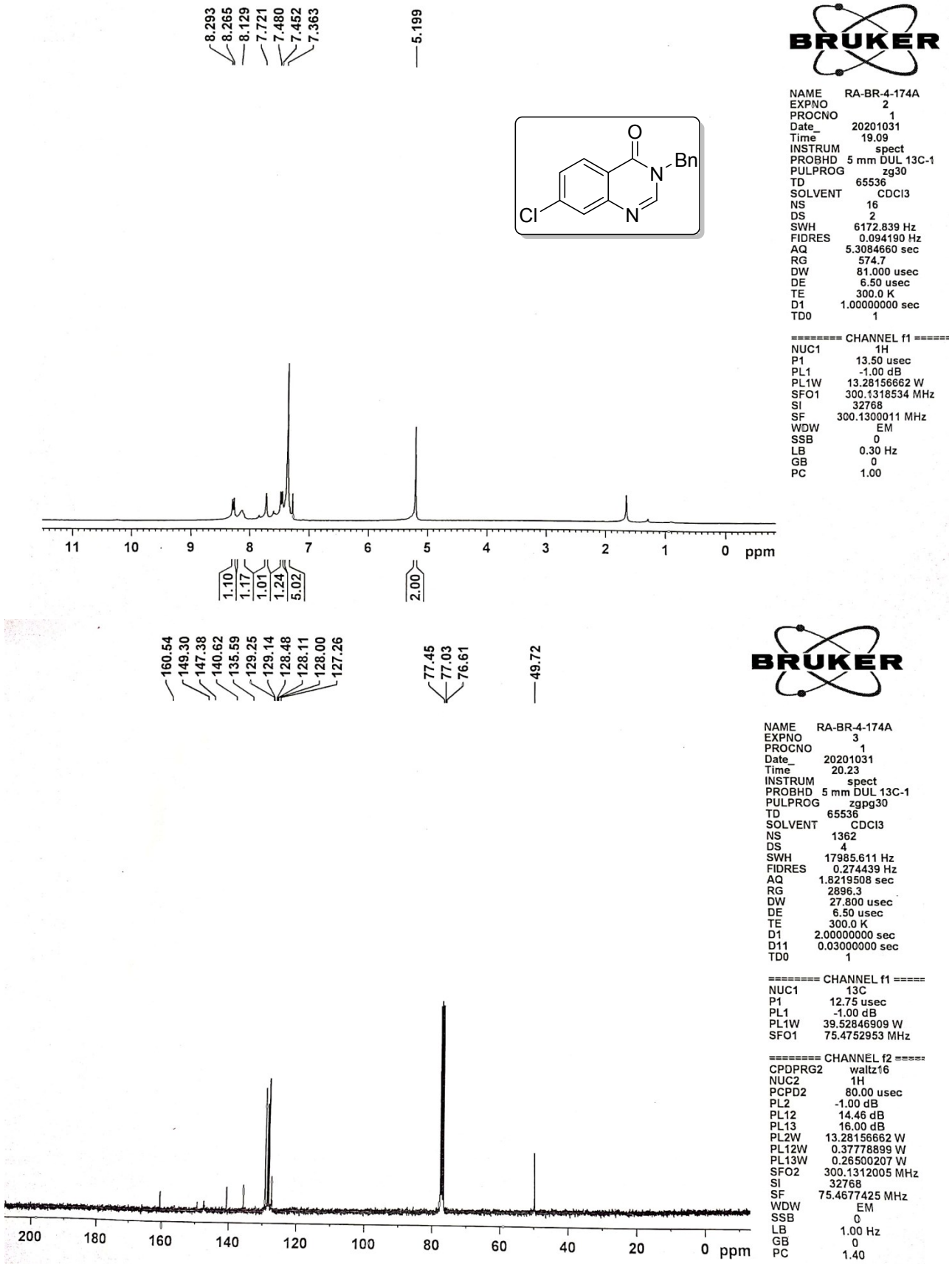
^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 5a



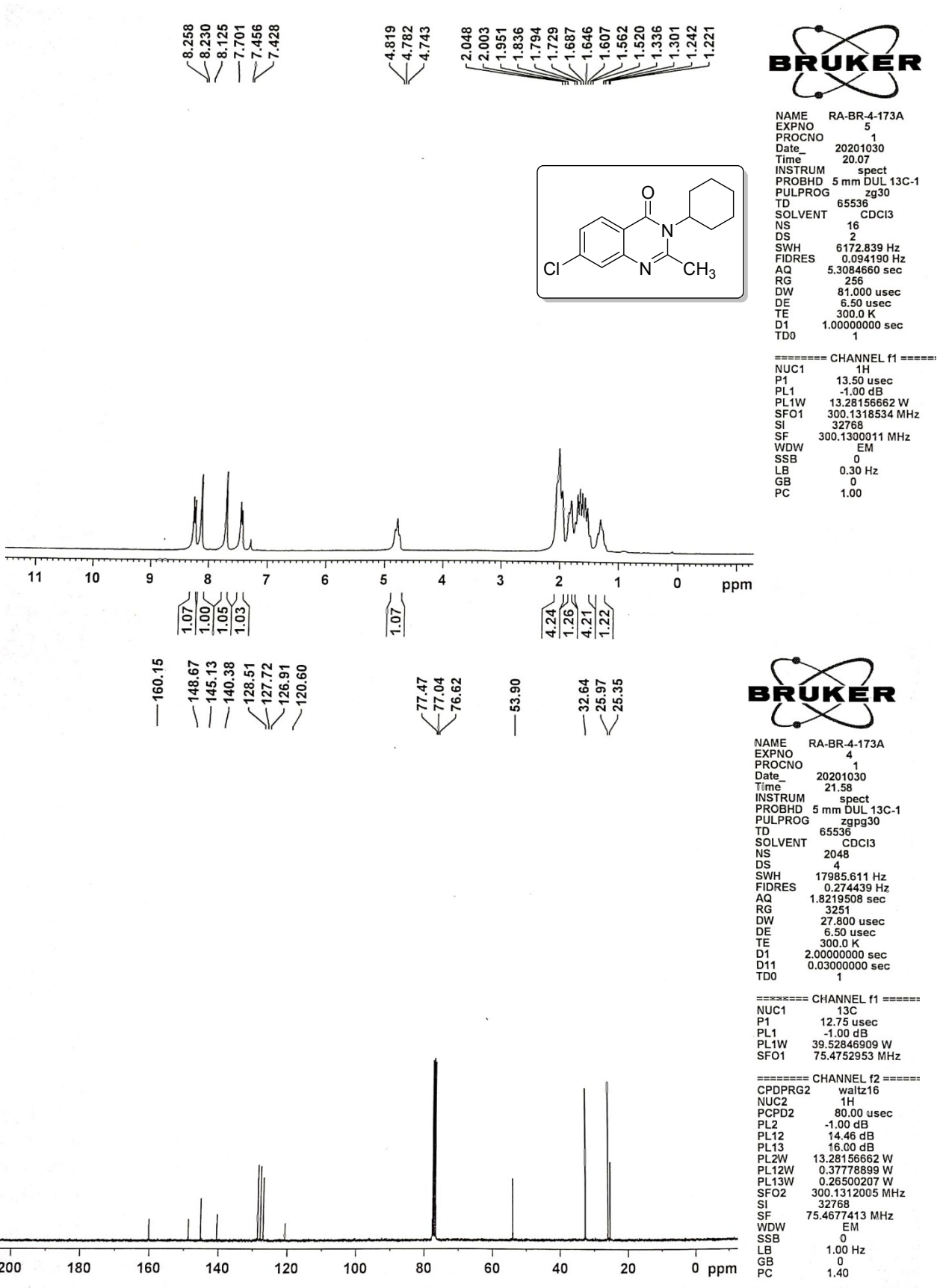
^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 5b



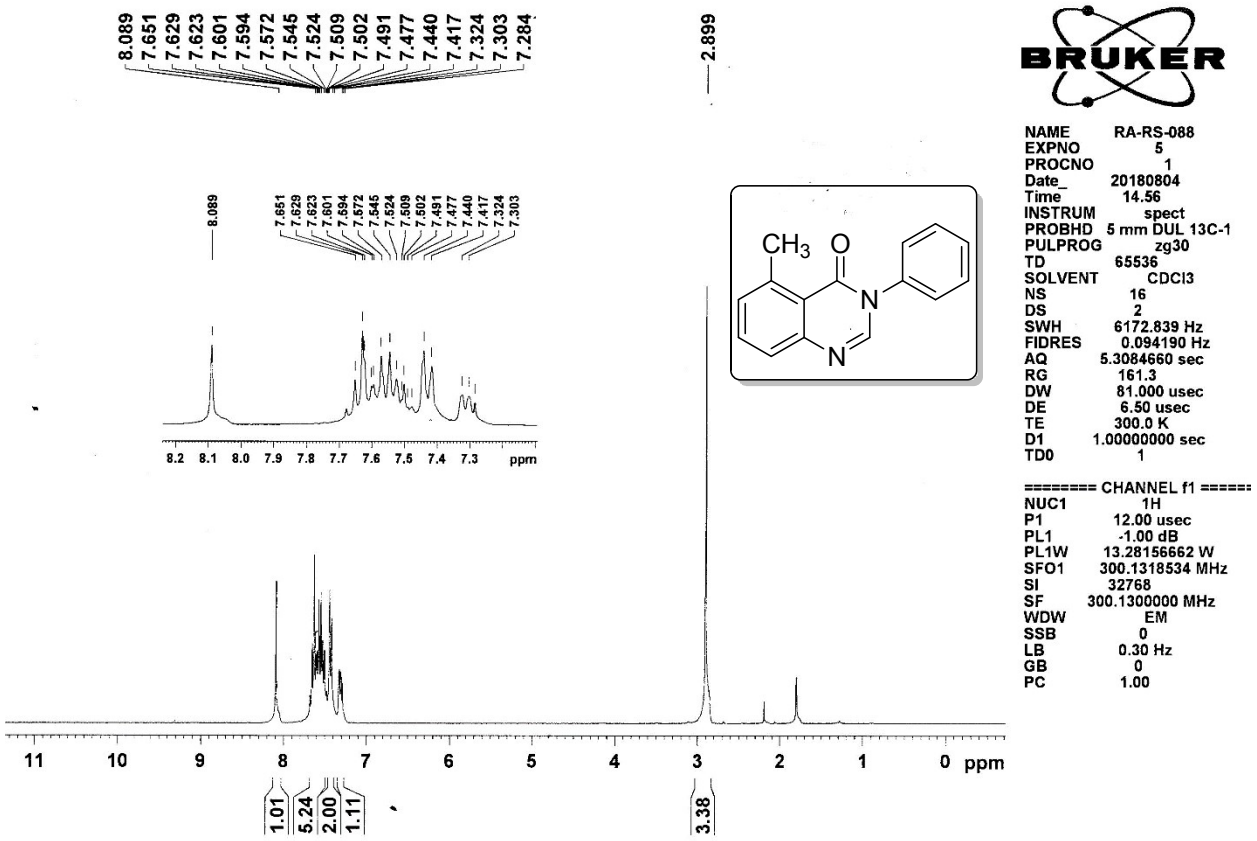
^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 5c



^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 5d



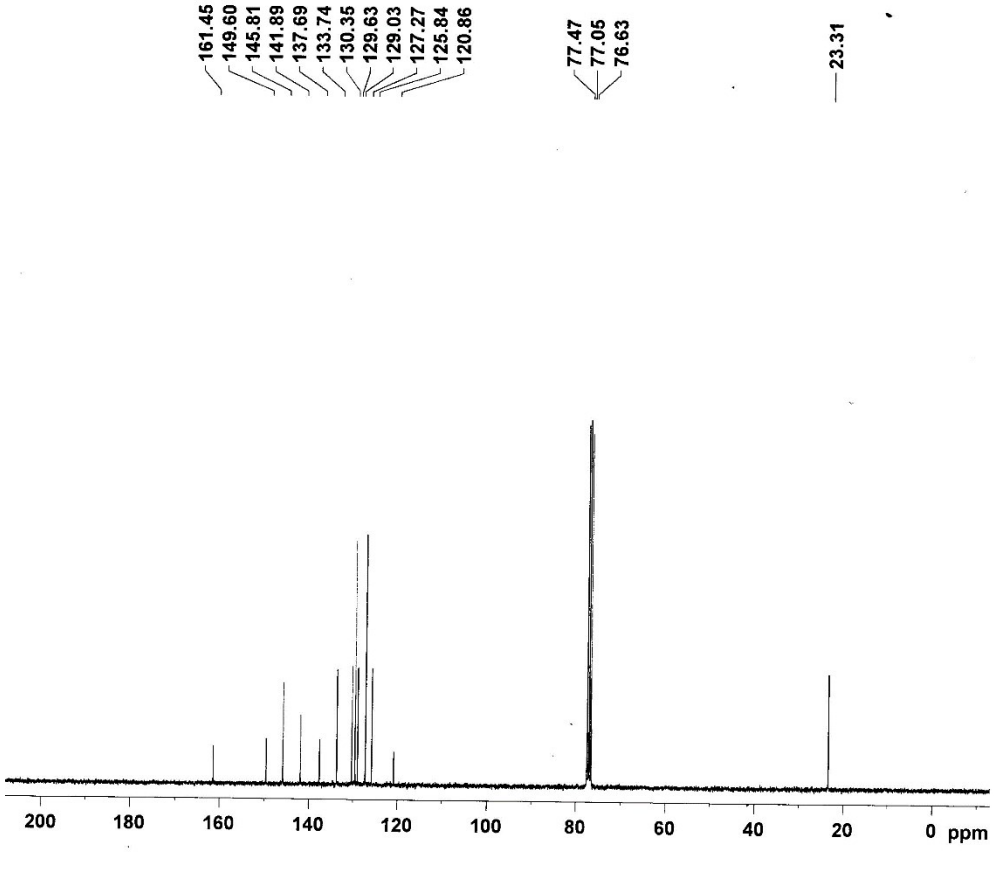
^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 5e



BRUKER

NAME RA-RS-088
EXPNO 5
PROCNO 1
Date_ 20180804
Time 14.56
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



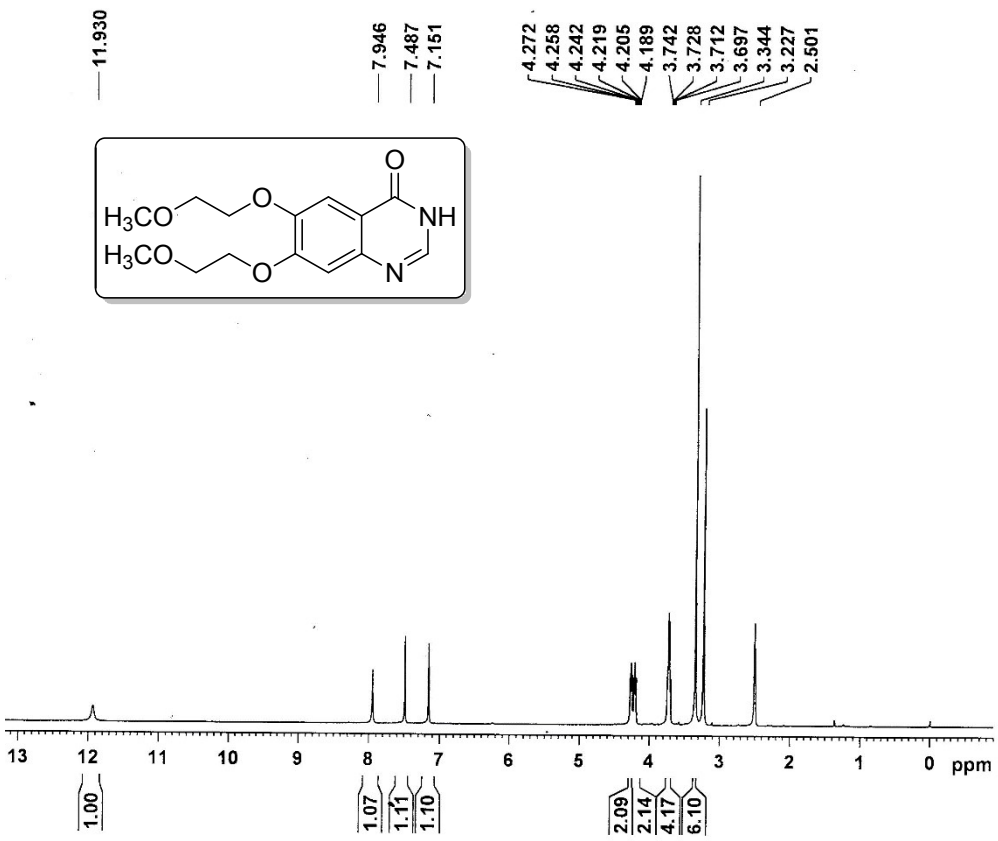
BRUKER

NAME RA-RS-088
EXPNO 1
PROCNO 1
Date_ 20180804
Time 14.54
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 =======
NUC1 13C
P1 10.68 usec
PL1 0.00 dB
PL1W 31.39858055 W
SFO1 75.4752953 MHz

======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.29870972 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

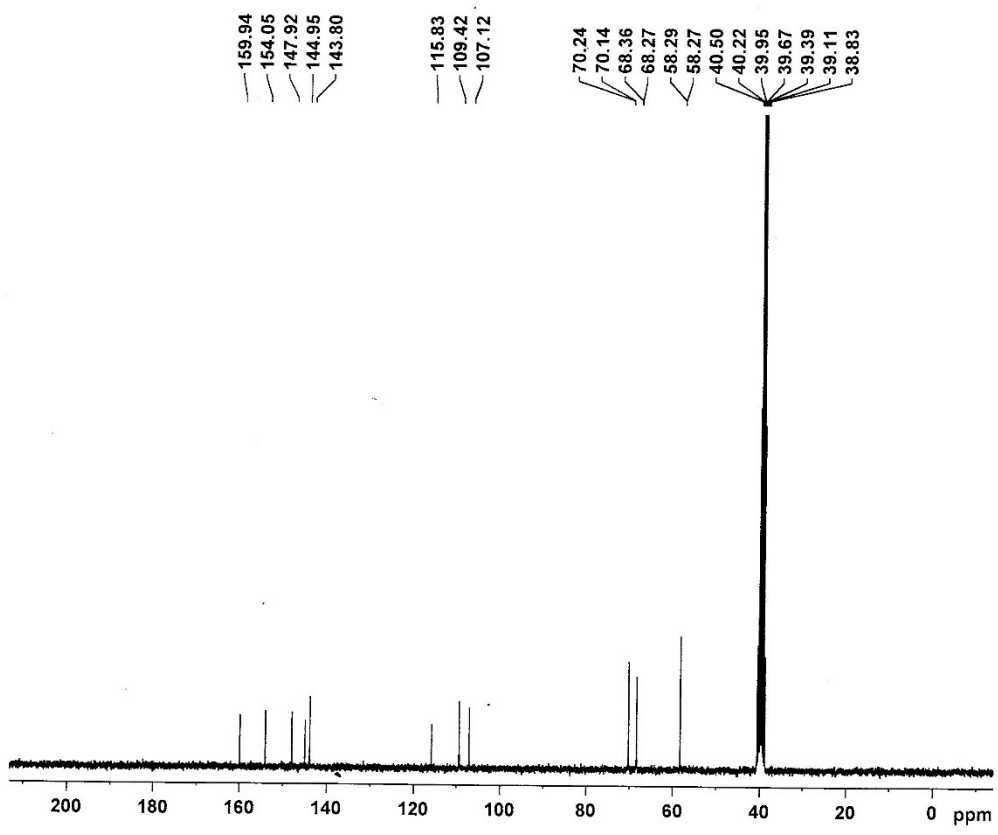
^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 5f



BRUKER

NAME RA-BR-3-80
EXPNO 5
PROCNO 1
Date_ 20190306
Time 15.13
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 161.3
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300025 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



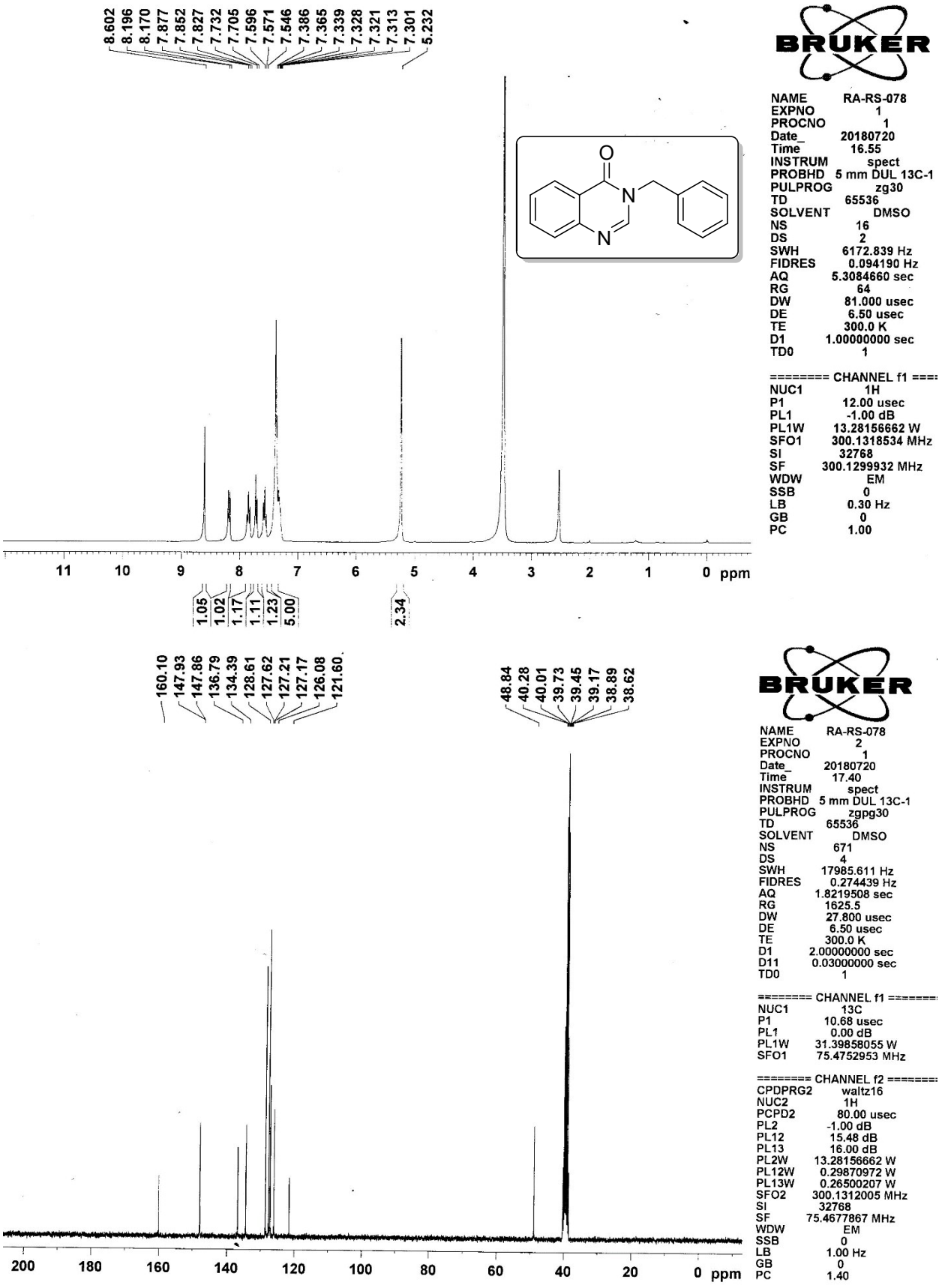
BRUKER

NAME RA-BR-3-80
EXPNO 4
PROCNO 1
Date_ 20190306
Time 15.02
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 3649.1
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

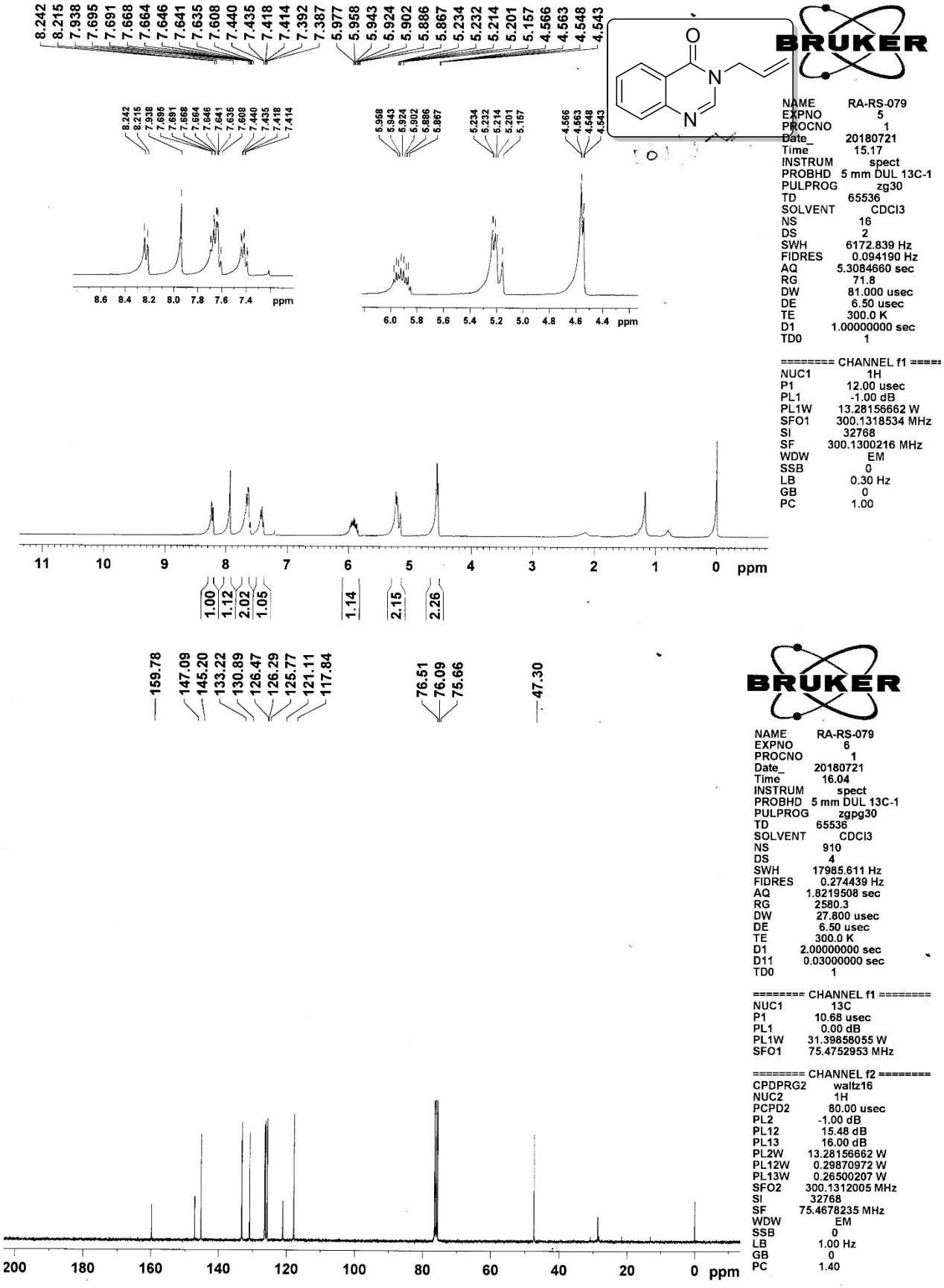
======== CHANNEL f1 ========
NUC1 13C
P1 10.68 usec
PL1 0.00 dB
PL1W 31.39858055 W
SFO1 75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.29870972 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

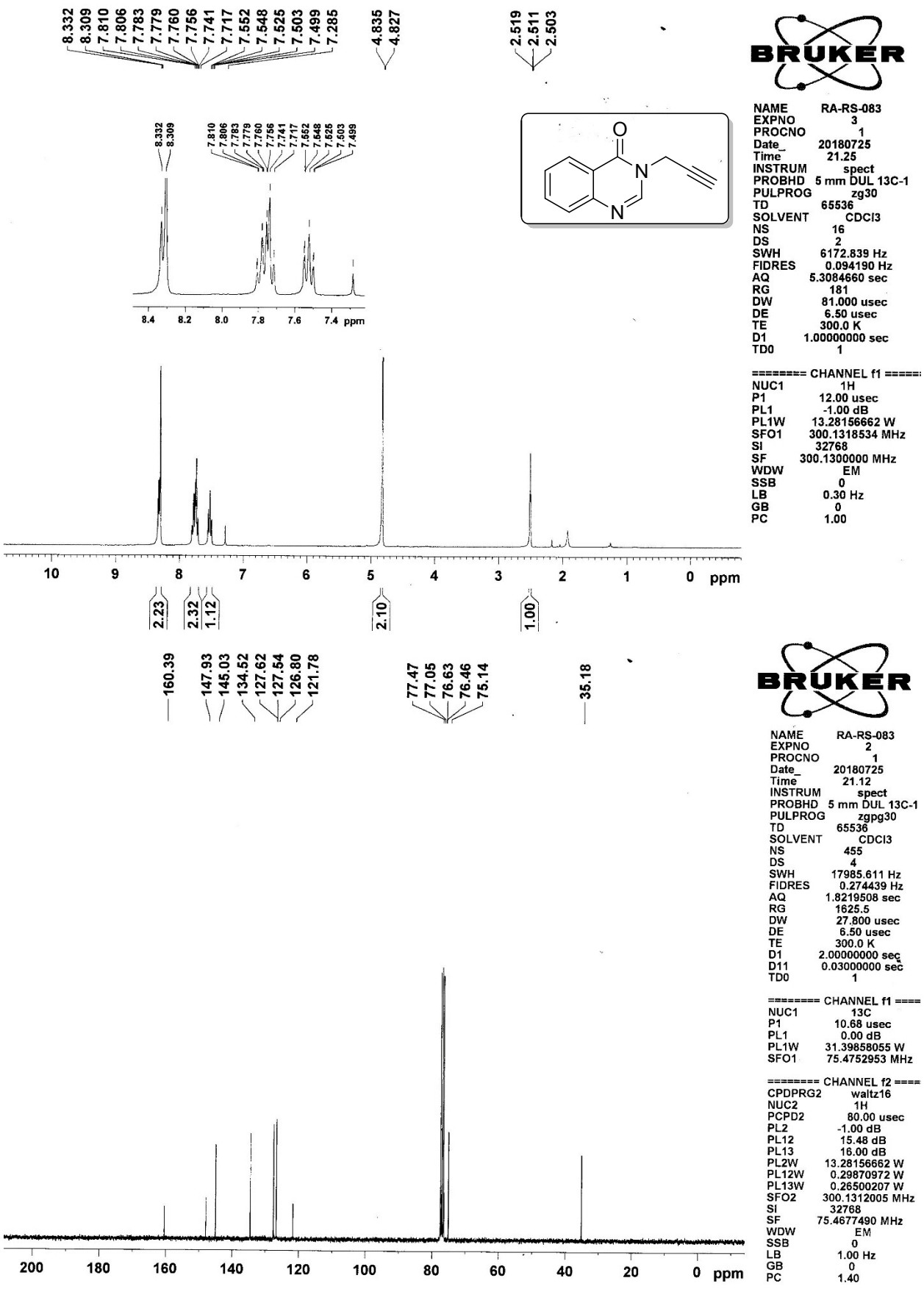
^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 6a



^{1}H & ^{13}C NMR (DMSO-d$_6$) spectra of compound 10



^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 11



BRUKER

NAME RA-RS-083
EXPNO 3
PROCNO 1
Date_ 20180725
Time 21.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 6172.839 Hz
FIDRES 0.094190 Hz
AQ 5.3084660 sec
RG 181
DW 81.000 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ======
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
PL1W 13.28156662 W
SFO1 300.1318534 MHz
SI 32768
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

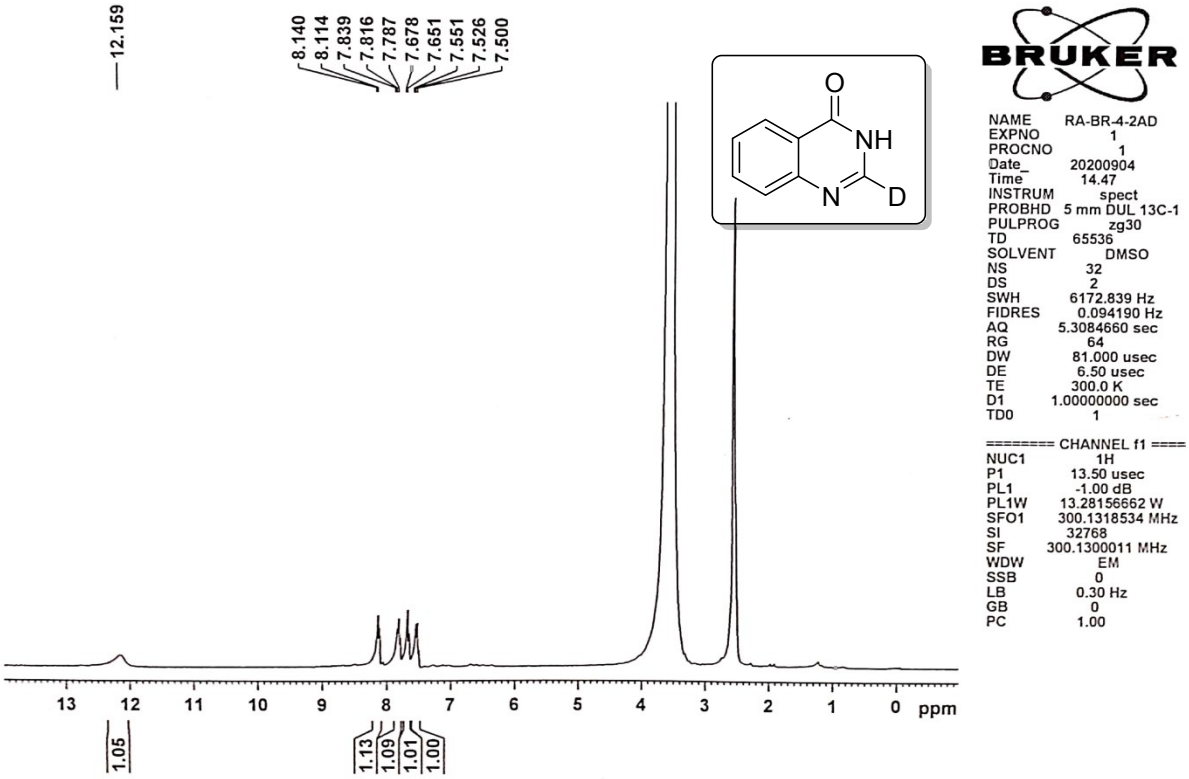
BRUKER

NAME RA-RS-083
EXPNO 2
PROCNO 1
Date_ 20180725
Time 21.12
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 455
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1625.5
DW 27.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ====
NUC1 13C
P1 10.68 usec
PL1 0.00 dB
PL1W 31.39858055 W
SFO1 75.4752953 MHz

======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 15.48 dB
PL13 16.00 dB
PL2W 13.28156662 W
PL12W 0.29870972 W
PL13W 0.26500207 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{1}H & ^{13}C NMR ($CDCl_3$) spectra of compound 12



^{1}H & ^{13}C NMR (DMSO-d_6) spectra of compound 2a$_d$