Supporting Information

Copper-Catalyzed Tandem *N*-Arylation/Condensation: Synthesis of Quinazolin-4(3*H*)-ones from 2-Halobenzonitrile and Amides

Hongxin Chai [a], Jiarong Li [a], Liupan Yang[b], Hongyan Lu [a], Zhang Qi [a] and Daxin Shi [a,*]

[a] School of Chemical Engineering and Environment, Beijing Institute of Technology, Beijing 100081, China.

[b] Jiangsu Hengrui Medicine Co., Ltd., No.7 Kunlunshan Road, Lianyungang Eco & Tech Development Zone, Jiangsu Province 222047, China.

Fax: +86(10)68918012; E-mail: shidaxin@bit.edu.cn.

Content

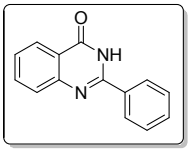
General information

Unless otherwise noted, all chemcials were purchased from commercial suppliers and were used without further purification. All experiments were monitored by thin layer chromatography (TLC) and visualized under 254 nm UV light. Column chromatography was performed on Silicycle silica gel (200-300 mesh). Melting points were determined using XT4 micro-scope melting point apparatus. Infrared spectra was recorded on a Perkin Elmer FT-IR spectrophotometer with KBr pellets. ^{1}H and ^{13}C NMR spectra were recorded at a Varian mercury-plus 400 spectrometer in DMSO-d_6 with TMS as the internal standard. ESI-MS was carried out on a Bruker APE XII FT-ICR using ESI ionization. HR-MS was recorded on a APEX IV FT-ICR Mass Spectrometer (Bruker, U.S.A.).

General experimental procedure for the synthesis of quinazolin-4(3*H*)-ones

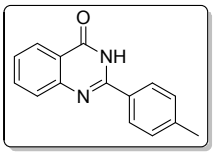
A 25 ml vial equipped with a magnetic stirring bar was charged with 2-halobenzonitrile (1.0 mmol), amides (1.0 mmol), CuI (5 % mmol), NaOH (1.2 mmol), NMP (5.0 ml). The reaction proceeded under an air atmosphere and heated for 12 h at 120 ℃. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to room temperature and diluted in ethyl acetate and washed with water. The aqueous phase was extracted twice with ethyl acetate. The organic layers were combined, dried over anhydrous Na_2SO_4, and the solvent was removed under reduced pressure and the crude product was purified by column chromatography or recrystallization using petroleum ether/EtOAc to provide the analytically pure product **3**.

2-phenylquinazolin-4(3*H*)-one (3a)



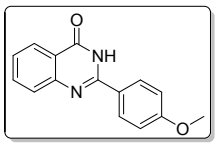
White crystal; m.p. 240-242℃ (Ref.[1] 234-236℃); IR (KBr, *v*, cm^{-1}): 3194, 3167, 3061, 1668, 1602, 1481; ^1H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.57 (s, 1H), 8.20-8.16 (m, 3H), 7.85 (t, *J* = 15.2 Hz, 1H), 7.75 (d, *J* = 7.2 Hz, 6H), 7.62-7.52 (m, 4H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.25, 152.30, 148.75, 134.59, 132.71, 131.38, 128.60 (2C), 127.76 (2C), 127.50, 126.58, 125.84, 120.98; ESI-MS (*m/z*) = 222.9 ($[M+H]^+$).

2-(p-tolyl)quinazolin-4(3*H*)-one (3b)



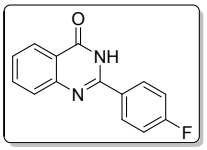
Light yellow crystal; m.p. 250-252℃ (Ref.[2] 256-257℃); IR (KBr, *v*, cm^{-1}): 3175, 3132, 3063, 1663, 1601, 1485; ^1H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.49 (s, 1H), 8.16-8.09 (m, 3H), 7.83 (t, *J* = 13.2 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.52 (t, *J* = 13.2 Hz, 1H); 7.36 (d, *J* = 7.6 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.72, 152.67, 149.30, 135.02, 130.35, 129.64 (2C), 128.14 (2C), 127.88, 126.85, 126.30, 121.37, 21.45; ESI-MS (*m/z*) = 237.5 ($[M+H]^+$).

2-(4-methoxyphenyl)quinazolin-4(3*H*)-one (3c)



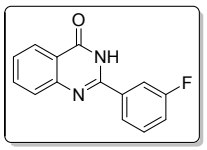
Light yellow crystal; m.p. 258-259℃ (Ref.[2] 250-251℃); IR (KBr, *v*, cm^{-1}): 3176, 3133, 3066, 1677, 1601, 1483; ^1H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.42 (s, 1H), 8.19 (d, *J* = 8.4 Hz, 2H), 8.13 (d, *J* = 8.0 Hz, 1H), 7.82 (t, *J* = 15.2 Hz, 1H), 7.70 (d, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 15.2 Hz, 1H), 7.09 (d, *J* = 8.4 Hz, 2H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.77, 162.33, 152.32, 149.40, 135.00, 129.92 (2C), 127.75, 126.59, 125.26, 121.15, 114.46 (2C), 55.92; ESI-MS (*m/z*) = 253.1 ($[M+H]^+$).

2-(4-fluorophenyl)quinazolin-4(3*H*)-one (3d)



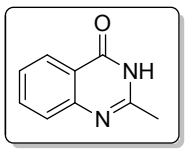
Yellow solid; m.p. 262-265℃ (Ref.[1] 284-287℃); IR (KBr, *v*, cm^{-1}): 3174, 3132, 3049, 1681, 1611, 1484; ^1H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.59 (s, 1H), 8.27-8.23 (m, 2H), 8.15 (d, *J* = 8.0 Hz, 1H), 7.84 (t, *J* = 5.2 Hz, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.53 (t, *J* = 14.8 Hz, 1H), 7.40 (t, *J* = 16.8 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 164.51 (d, *J* = 198.4 Hz), 162.67, 151.84, 149.12, 135.11, 130.85 (d, *J* = 7.1 Hz), 129.70 (d, *J* = 2.1 Hz), 127.93, 127.09, 126.32, 121.35, 116.10 (d, *J* = 17.4 Hz); ESI-MS (*m/z*) = 241.0 ($[M+H]^+$).

2-(3-fluorophenyl)quinazolin-4(3*H*)-one (3e)



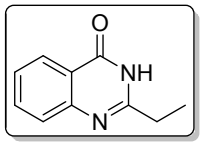
Light yellow crystal; m.p. 261-263℃ (Ref.[3] 267℃); IR (KBr, *v*, cm^{-1}): 3192, 3161, 3055, 1682, 1608, 1480; ^1H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.62 (s, 1H), 8.17 (d, *J* = 7.6 Hz, 1H), 8.07-8.00 (m, 2H), 7.86 (t, *J* = 14.0 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 1H), 7.63-7.53 (m, 2H), 7.47-7.43 (m, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.58, 162.55 (d, *J* = 193.9 Hz), 151.48, 148.92, 135.47 (d, *J* = 6.3 Hz), 135.17, 131.21 (d, *J* = 6.5 Hz), 128.07, 127.39, 126.34, 124.40 (d, *J* = 1.9 Hz), 121.59, 118.73 (d, *J* = 16.7 Hz), 115.00 (d, *J* = 18.9 Hz); ESI-MS (*m/z*) = 241.0 ($[M+H]^+$).

2-methylquinazolin-4(3*H*)-one (3f)[4]



White crystal; m.p. 236-237℃; IR (KBr, *v*, cm^{-1}): 3170, 3120, 2869, 1681, 1615, 1468; ^1H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.20 (s, 1H), 8.06 (d, *J* = 8.0 Hz, 1H), 7.77-7.73 (m, 1H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.44 (t, *J* = 14.8 Hz, 1H), 2.34 (s, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 161.69, 154.25, 148.98, 134.27, 126.58, 125.85, 125.67, 120.61, 21.42; ESI-MS (*m/z*) = 161.0 ($[M+H]^+$).

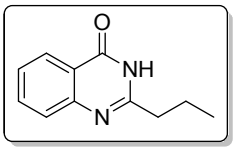
2-ethylquinazolin-4(3*H*)-one (3g)



White crystal; m.p. 230-232℃ (Ref.[5] 229-231℃); IR (KBr, *v*, cm^{-1}): 3169, 3111, 2980, 1679, 1620, 1468; ^1H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.16 (s, 1H), 8.07 (d, *J* = 8.0 Hz, 1H), 7.79-7.75 (m, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 14.4 Hz, 1H), 2.65-2.59 (m, 2H), 1.24 (t, *J* = 14.8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.26, 158.77, 149.43, 134.69,

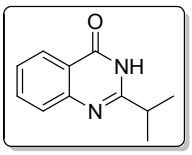
127.27, 126.35, 126.14, 121.30, 28.30, 11.73; ESI-MS (*m/z*) = 175.0 ([M+H]$^+$).

2-propylquinazolin-4(3*H*)-one (3h)



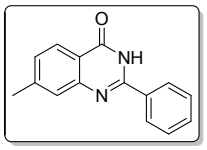
White crystal; m.p. 206-207℃ (Ref.[5] 200-202℃); IR (KBr, *v*, cm^{-1}): 3174, 3132, 3049, 1681, 1611, 1484; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.18 (s, 1H), 8.07 (d, *J* = 7.6 Hz, 1H), 7.76 (t, *J* = 14.8 Hz, 1H), 7.59 (d, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 15.2 Hz, 1H), 2.56 (t, *J* = 14.8 Hz, 2H), 1.74 (d, *J* = 15.2 Hz, 2H), 0.94-0.91 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.28, 157.76, 149.42, 134.71, 127.26, 126.36, 126.13, 121.26, 36.81, 20.66, 13.96; ESI-MS (*m/z*) = 189.4 ([M+H]$^+$).

2-isopropylquinazolin-4(3*H*)-one (3i)



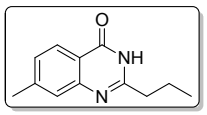
White crystal; m.p. 234-236℃ (Ref.[5] 225-228℃); IR (KBr, *v*, cm^{-1}): 3169, 3125, 2969, 1682, 1621, 1471; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.15 (s, 1H), 8.08 (d, *J* = 8.8 Hz, 1H), 7.77 (t, *J* = 14.8 Hz, 1H), 7.61 (d, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 15.6 Hz, 1H), 2.92-2.85 (m, 1H), 1.26 (d, *J* = 7.6 Hz, 6H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.41, 162.03, 149.35, 134.71, 127.4, 126.43, 126.13, 121.40, 20.83 (2C); ESI-MS (*m/z*) = 189.4 ([M+H]$^+$).

7-methyl-2-phenylquinazolin-4(3*H*)-one (3j)



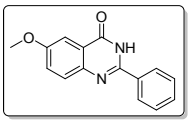
Light yellow solid; m.p. 248-250℃ (Ref.[6] 240-241℃); IR (KBr, *v*, cm^{-1}): 3129, 3055, 3035, 1670, 1605, 1457; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.45 (s, 1H), 8.17 (d, *J* = 7.2 Hz, 2H), 8.03 (d, *J* = 7.6 Hz, 1H), 7.57 (d, *J* = 10.0 Hz, 4H), 7.35 (d, *J* = 8.8 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.57, 152.77, 149.32, 145.53, 133.24, 131.80, 129.64, 129.06, 128.48, 128.17, 128.08, 127.62, 126.17, 119.06, 21.83; ESI-MS (*m/z*) = 237.1 ([M+H]$^+$).

7-methyl-2-propylquinazolin-4(3*H*)-one (3k)



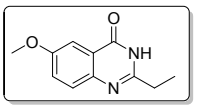
White crystal; m.p. 226-228℃ (Ref.[7] 232-234℃); IR (KBr, *v*, cm^{-1}): 3171, 3129, 3043, 1675, 1615, 1461; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.07 (s, 1H), 7.96 (d, *J* = 7.6 Hz, 1H), 7.41 (s, 1H), 7.28 (d, *J* = 7.2 Hz, 1H), 2.56 (t, *J* = 14.4 Hz, 2H), 2.43 (s, 3H) 1.76-1.69 (m, 2H), 0.93 (t, *J* = 14.4 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.17, 157.75, 145.12, 127.76, 125.99, 118.86, 36.77, 21.79, 20.63, 13.94; ESI-MS (*m/z*) = 203.1 ([M+H]$^+$).

6-methoxy-2-phenylquinazolin-4(3*H*)-one (3l)



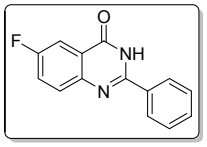
Yellow solid; m.p. 246-248℃ (Ref.[2] 247-248℃); IR (KBr, *v*, cm^{-1}): 3161, 3055, 3028, 1672, 1492; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.51 (s, 1H), 8.16(d, *J* = 6.8 Hz, 2H), 7.70 (d, *J* = 8.0 Hz, 2H), 7.59-7.52 (m, 4H), 7.46-7.43 (m, 1H), 3.86 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.50, 158.22, 150.55, 143.68, 133.27, 131.50, 130.17, 129.70, 129.04, 127.96, 124.57, 106.35, 56.12; ESI-MS (*m/z*) = 253.1 ([M+H]$^+$).

2-ethyl-6-methoxyquinazolin-4(3*H*)-one (3m)



Light yellow solid; m.p. 241-243℃; IR (KBr, *v*, cm^{-1}): 3162, 3014, 1669, 1624, 1488; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.11 (s, 1H), 7.54 (d, *J* = 9.6 Hz, 1H), 7.46 (d, *J* = 2.4 Hz, 1H), 7.37 (t, *J* = 9.2 Hz, 1H), 3.85 (s, 3H), 2.61-2.56 (m, 2H), 1.23 (t, *J* = 14.8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.09, 157.64, 156.36, 143.90, 128.91, 124.08, 122.00, 106.24, 55.99, 28.10, 11.75; HR-ESI ([M+H]$^+$) *m/z* calcd for $C_{11}H_{13}N_2O_2$ 205.09715, found 205.09710.

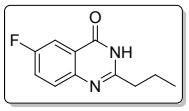
6-fluoro-2-phenylquinazolin-4(3*H*)-one (3n)



Yellow solid; m.p. 279-281℃ (Ref.[8] > 280℃); IR (KBr, *v*, cm^{-1}): 3163, 3104, 3036, 1662, 1605, 1485; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.67 (s, 1H), 8.17 (d, *J* = 6.4 Hz, 2H), 7.81 (t, *J* = 13.2 Hz, 2H), 7.75-7.70 (m, 1H), 7.61-7.53 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.13 (d, *J* = 2.6 Hz), 160.46 (d, = 195.2 Hz), 152.32, 146.10, 133.02, 131.89, 130.80 (d, *J* = 6.6 Hz), 129.08, 128.21, 123.55 (d, *J* = 19.0 Hz), 122.68 (d, *J* = 6.6 Hz), 110.98 (d, *J* = 18.6 Hz); ESI-MS (*m/z*) = 241.0

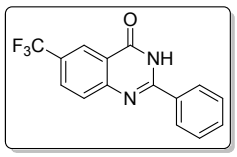
([M+H]$^+$).

6-fluoro-2-propylquinazolin-4(3*H*)-one (3o)[9]



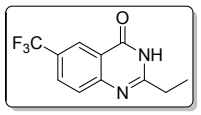
White crystal; m.p. 240-242℃; IR (KBr, *v*, cm^{-1}): 3184, 3108, 3035, 1670, 1605, 1457; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.29 (s, 1H), 7.73 (t, *J* = 10.4 Hz, 1H), 7.65 (t, *J* = 9.6 Hz, 2H), 2.57 (t, *J* = 15.6 Hz, 2H), 1.76-1.70 (m, 2H), 0.92 (t, *J* = 14.8 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 161.65 (d, *J* = 2.6 Hz), 160.03 (d, *J* = 194.3 Hz), 157.20 (d, *J* = 1.2 Hz), 146.26, 130.09(d, *J* = 6.6 Hz), 123.06 (d, *J* = 19.0 Hz), 122.40 (d, *J* = 6.6 Hz), 110.66 (d, *J* = 18.4 Hz), 36.70, 20.58, 13.90; ESI-MS (*m/z*) = 207.0 ([M+H]$^+$).

2-phenyl-6-(trifluoromethyl)quinazolin-4(3*H*)-one (3p)



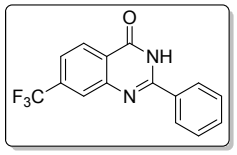
White solid; m.p. > 300℃; IR (KBr, *v*, cm^{-1}): 3167, 3108, 3052, 3028, 1671, 1626, 1602, 1317; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.88 (s, 1H), 8.39 (s, 1H), 8.21 (d, *J* = 7.2 Hz, 2H), 8.14 (t, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 8.4 Hz, 1H), 7.63-7.56 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.14, 155.16, 151.75, 132.75, 132.42, 130.98, 129.44, 129.15, 128.55, 126.88 (q, *J* = 26.0 Hz), 124.35 (q, *J* = 216.4 Hz), 123.78 (q, *J* = 3.1 Hz), 121.53; HR-ESI ([M+H]$^+$) *m/z* calcd for $C_{15}H_{10}F_3N_2O$ 291.07397; found 291.07378.

2-ethyl-6-(trifluoromethyl)quinazolin-4(3H)-one (3q)



White solid; m.p. 260-261℃; IR (KBr, *v*, cm^{-1}): 3172, 3111, 3047, 3019，1689, 1614, 1319; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.50 (s, 1H), 8.31 (s, 1H), 8.06-8.04 (m, 1H), 7.77 (d, *J* = 8.4 Hz, 1H), 2.69-2.63 (m, 2H), 1.25 (t, *J* = 15.6 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 161.57, 161.55, 151.91, 130.63, 128.74, 126.39 (q, *J* = 25.9 Hz), 123.55 (q, *J* = 3.4 Hz), 123.34 (q, *J* = 216.1 Hz), 121.32, 28.44, 11.50; HR-ESI ([M+H]$^+$) *m/z* calcd for $C_{11}H_{10}F_3N_2O$ 243.07397; found 243.07412.

2-phenyl-7-(trifluoromethyl)quinazolin-4(3*H*)-one (3r) [9]

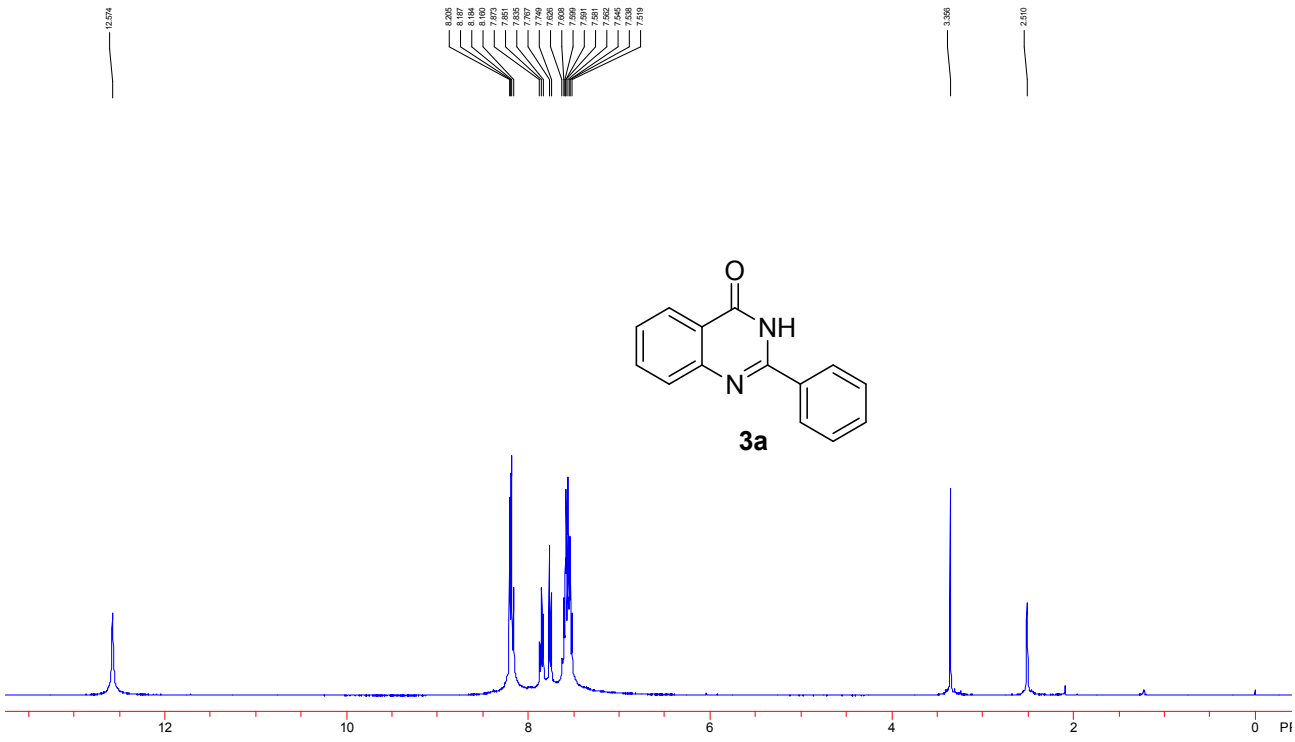


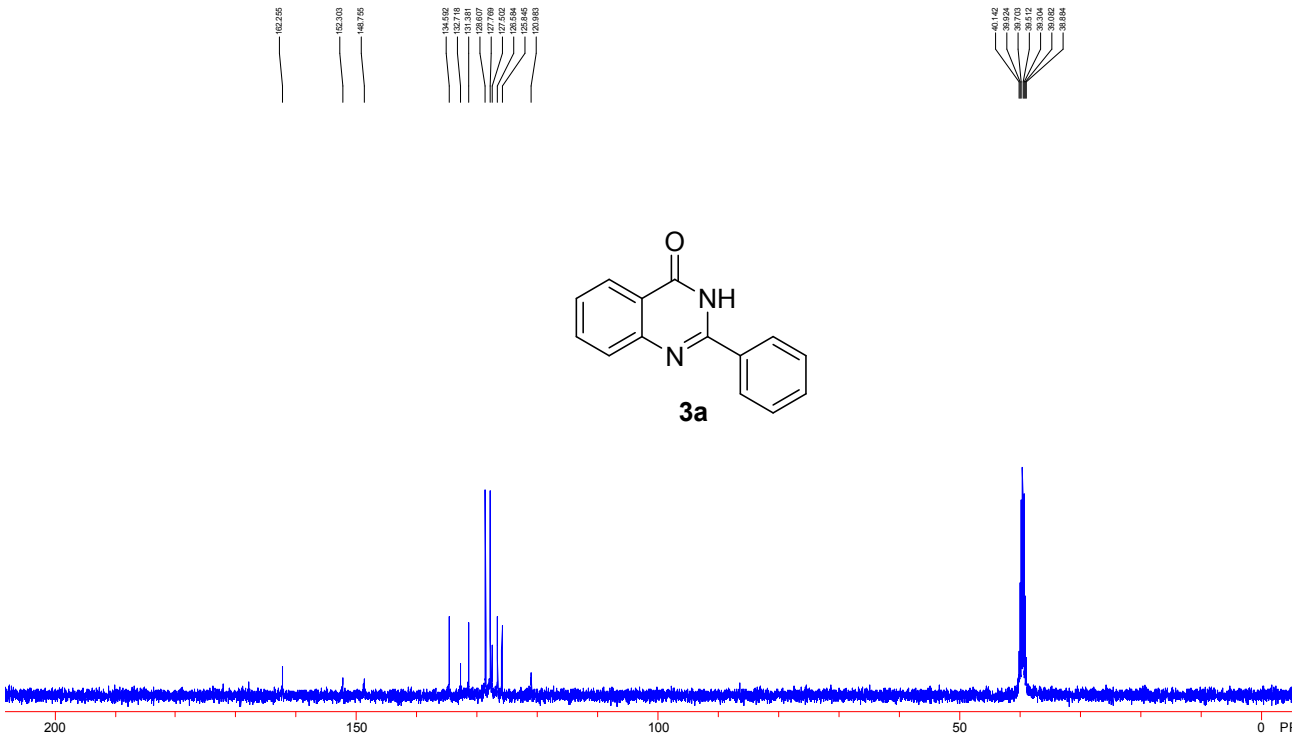
Light yellow solid; m.p. 194-196℃; IR (KBr, *v*, cm^{-1}): 3206, 3160, 3095, 1670, 1607, 1492; ^{1}H NMR (400 MHz, DMSO-d_6) (δ, ppm): 12.84 (s, 1H), 8.33 (t, *J* = 24.8 Hz, 1H), 8.20 (d, *J* = 7.6 Hz, 2H), 8.04 (s, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 7.62-7.57 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) (δ, ppm): 162.03, 154.40, 149.27, 134.70 (q, *J* = 25.5 Hz), 132.74, 132.28, 129.12 (2C), 128.29 (2C), 128.15, 125.05 (q, *J* = 2.9 Hz), 123.98 (q, *J* = 206.9 Hz), 122.95, 122.63; ESI-MS (*m/z*) = 291.1 ([M+H]$^+$).

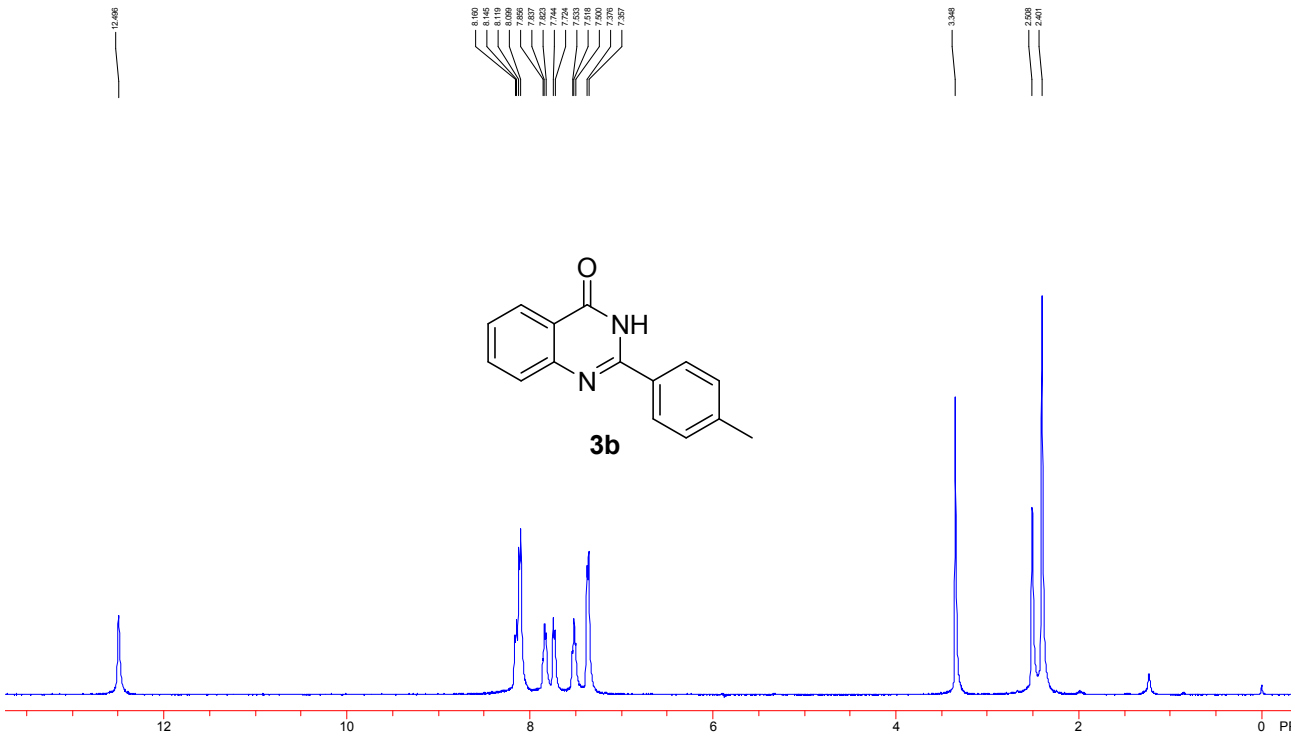
References

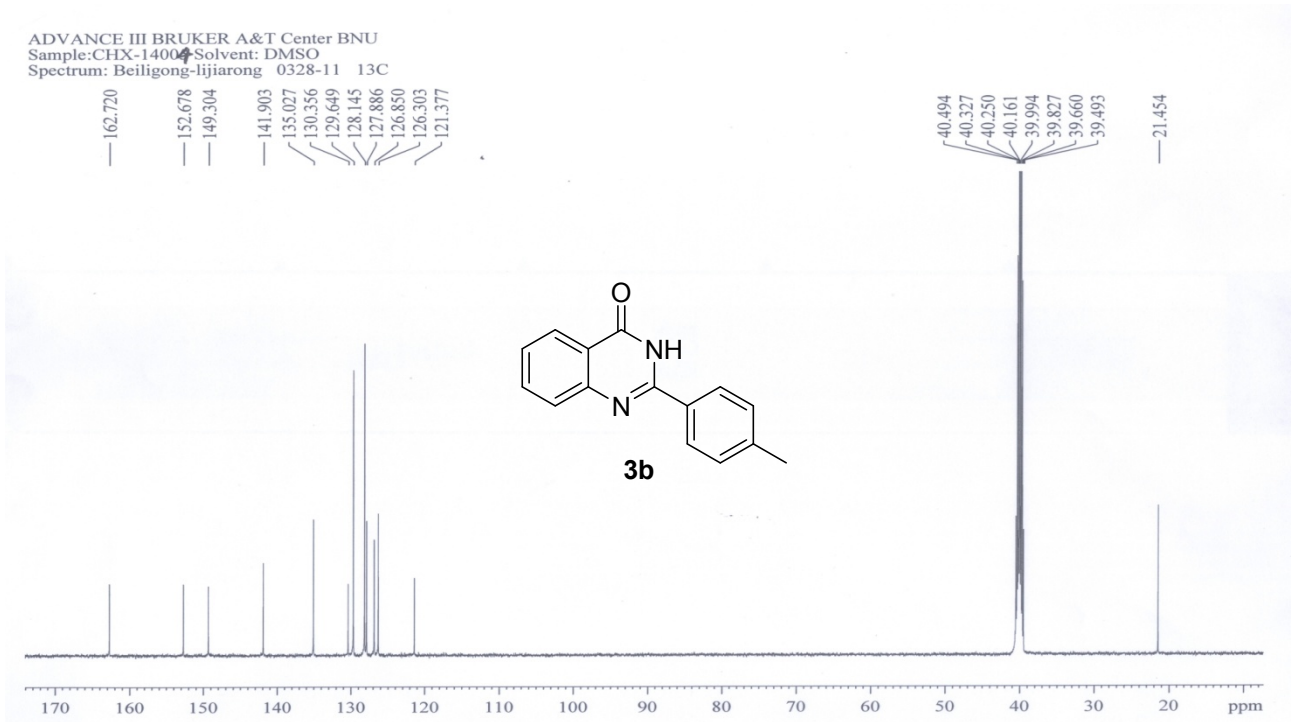
1. D. Zhao, T. Wang and J. X. Li, *Chem. Commun.*, 2014, **50**, 6471.
2. L. X. Wang, J. F. Xiang and Y. L. Tang, *Eur. J. Org. Chem.*, 2014, **2014**, 2682.
3. J. X. Chen, D. Z. Wu, F. He, M. C. Liu, H. Y. Wu, J. C. Ding, W. K. Su, *Tetrahedron Lett.*, 2008, **49**, 3814.
4. B. Ma, Y. Wang, J. Peng and Q. Zhu, *J. Org. Chem.*, 2011, **76**, 6362.
5. W. Xu and H. Fu, *J. Org. Chem.*, 2011, **76**, 3846.
6. W. Xu, Y. Jin, H. Liu, Y. Jiang and H. Fu, *Org. lett.*, 2011, **13**, 1274.
7. C. E. Liu, Q. Y. Yu, J. H. Tang, J. R. Li, *Chin. J. Org. Chem.*, 2012, **3**, 532.
8. M. J. Hour, J. S. Yang, T. L. Chen, K. T. Chen, S. C. Kuo, J. G. Chung, C. C. Lu, C. Y. Chen, Y. H. Chuang, *Eur. J. Med. Chem.*, 2011, **46**, 2709.
9. F. Jens, Z. Hans-Joachim, H. Ines, W. Lothar, M. Thomas, B. Marco, V. K. D. Pascal, H. H. Isolde, R. C. Hugh, D. Jan, PCT Int. Appl. (2012), WO 2012028578 A1 20120308.

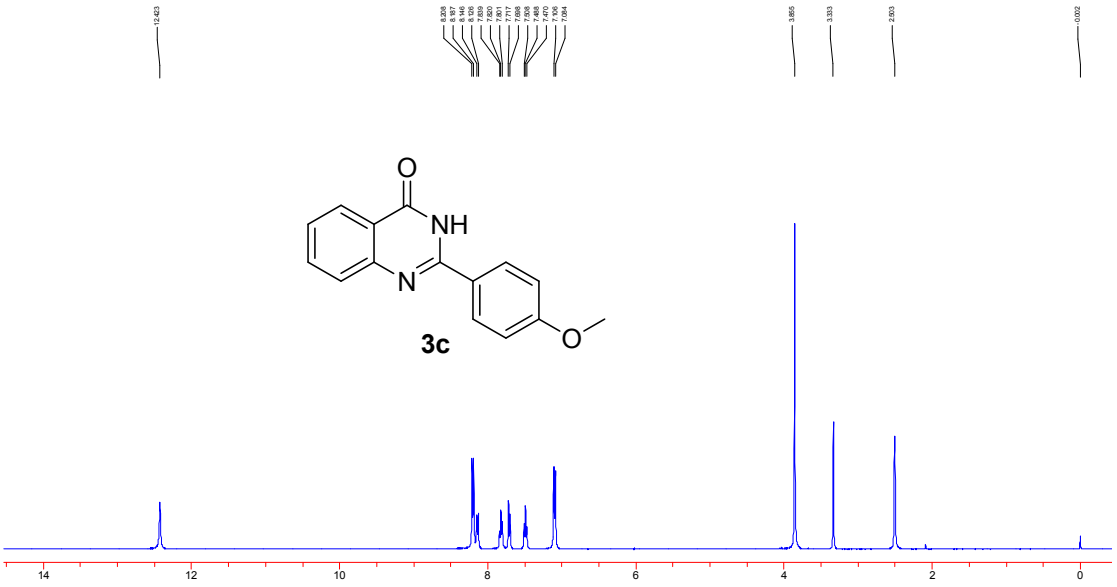
The ^{1}H-NMR and^{13}C-NMR Spectra of products

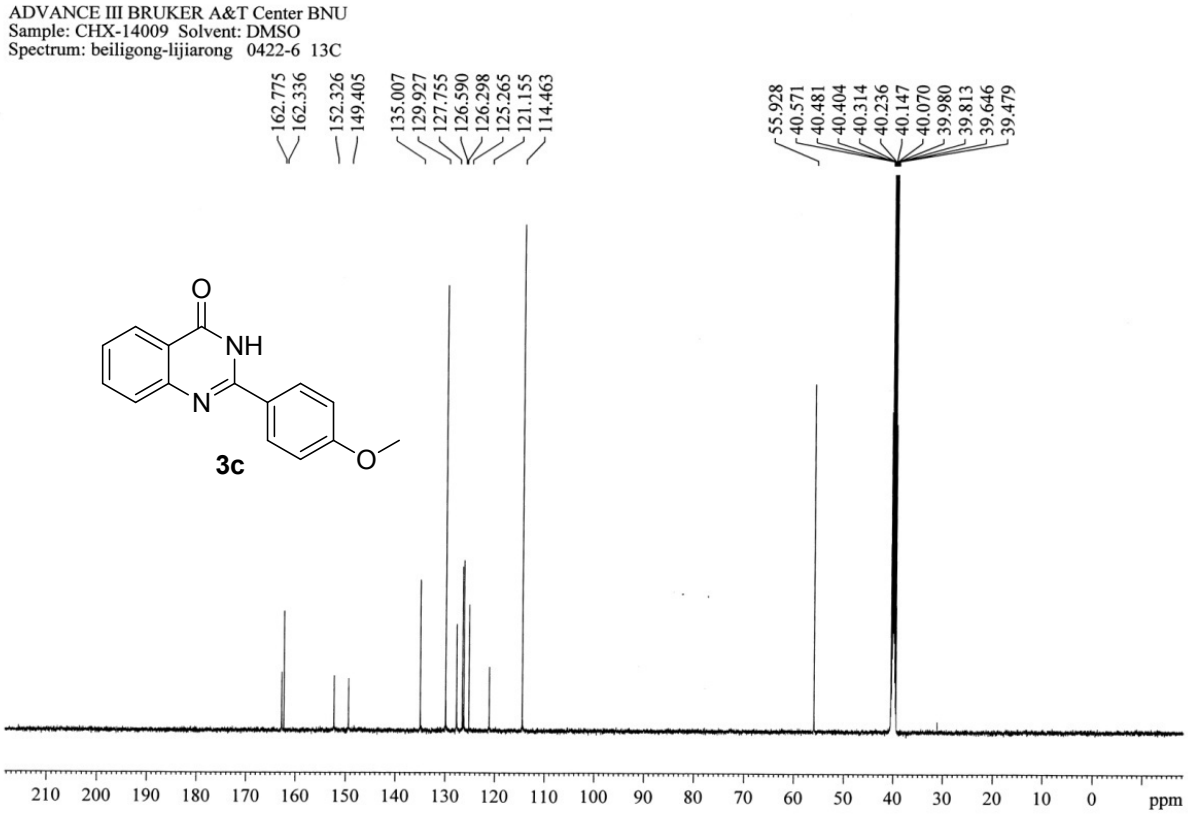


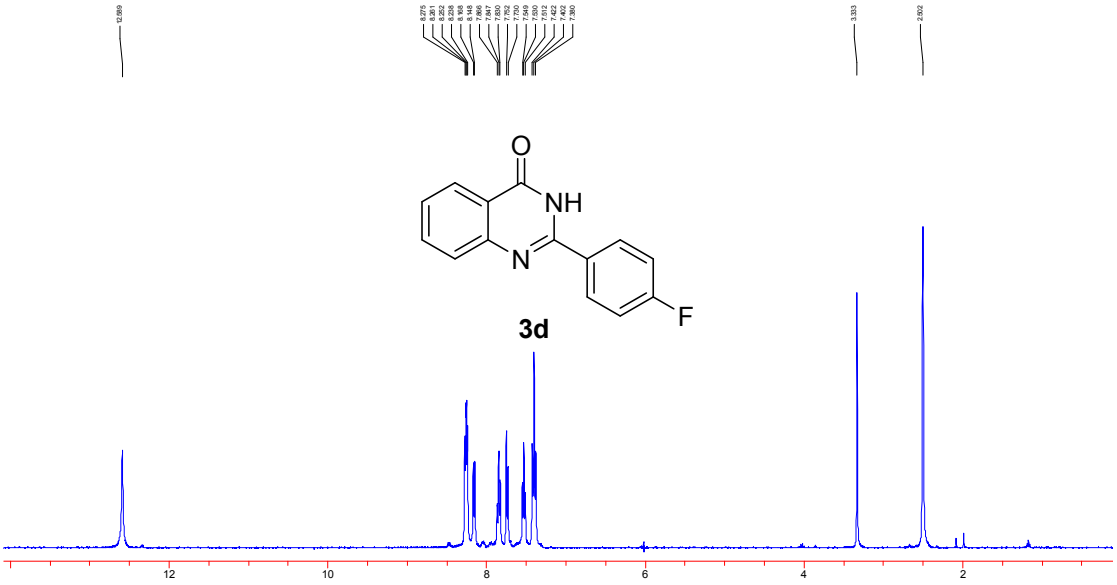


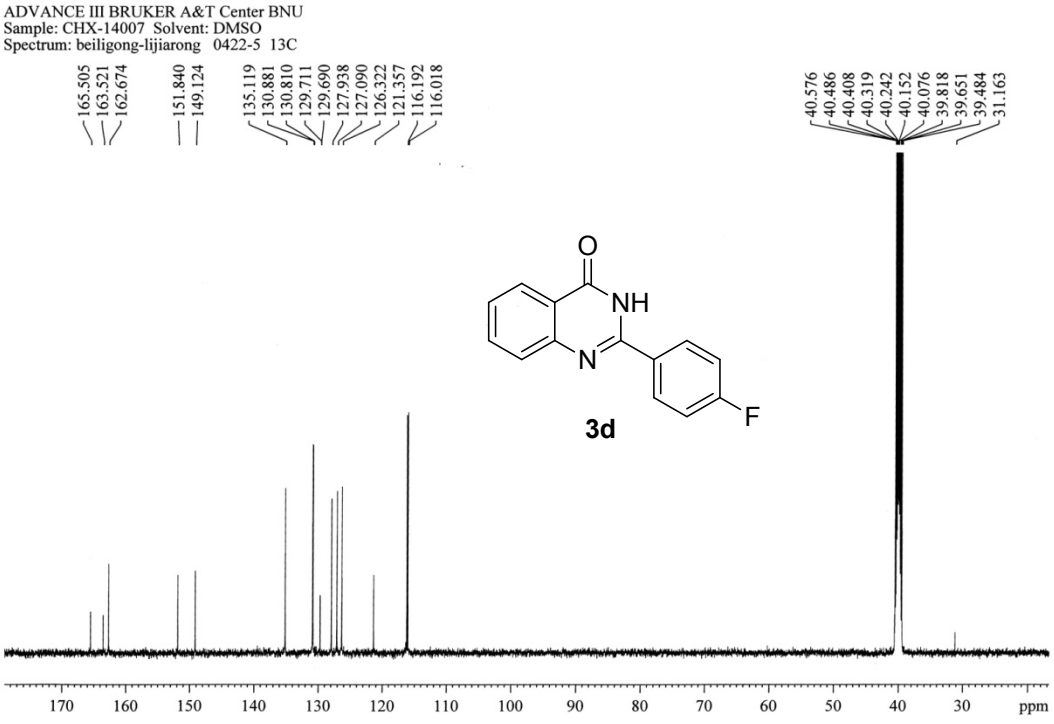

12.466
8.160
8.145
8.119
8.099
8.095
7.857
7.823
7.744
7.724
7.553
7.518
7.500
7.376
7.357
3.348
2.508
2.401
O
NH
N
3b
12
10
8
6
4
2
0
PI

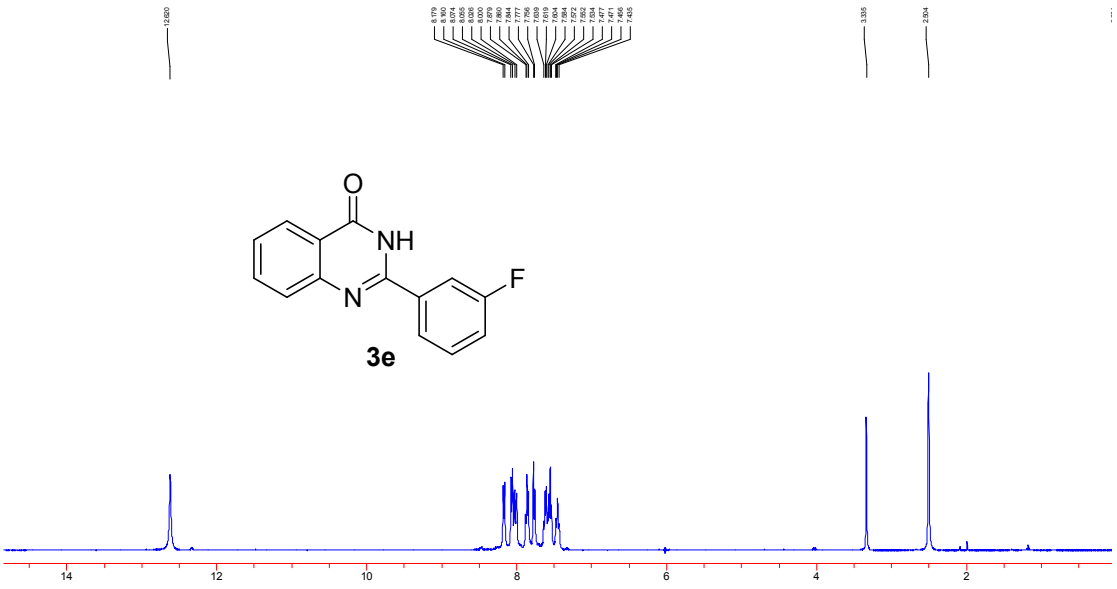

ADVANCE III BRUKER A&T Center BNU
Sample:CHX-14004 Solvent: DMSO
Spectrum: Beiligong-lijiarong 0328-11 13C
162.720
152.678
149.304
141.903
135.027
130.356
129.649
128.145
127.886
126.850
126.303
121.377
40.494
40.327
40.250
40.161
39.994
39.827
39.660
39.493
21.454
O
NH
N
3b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

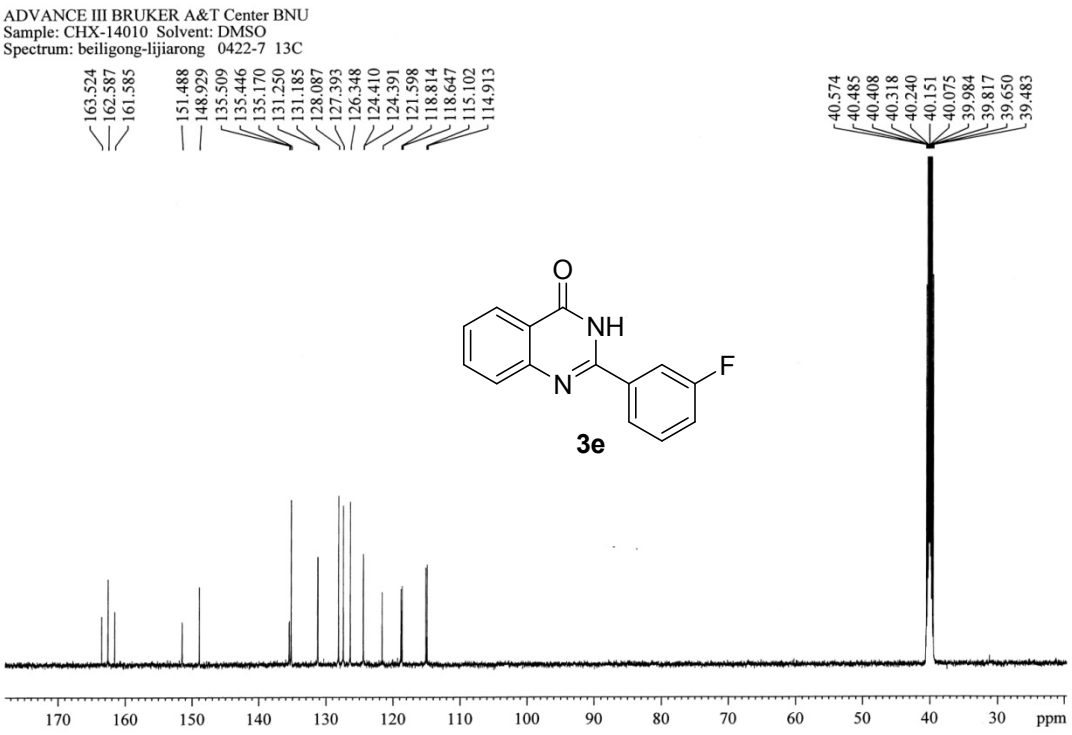

12.423
8.206
8.187
8.146
8.126
7.839
7.820
7.801
7.777
7.758
7.506
7.486
7.470
7.084
3.865
3.333
2.503
0.002
O
NH
N
O
3c
14
12
10
8
6
4
2
0

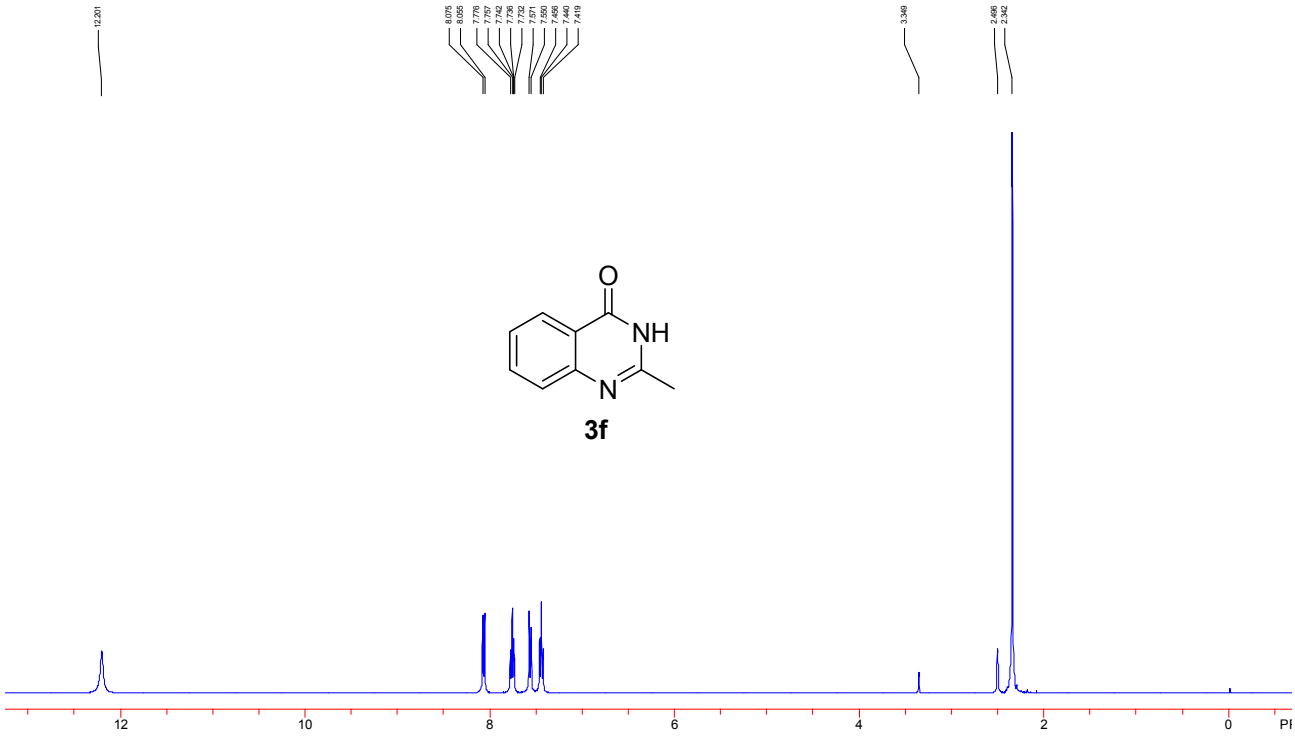

ADVANCE III BRUKER A&T Center BNU
Sample: CHX-14009 Solvent: DMSO
Spectrum: beiligong-lijiarong 0422-6 13C
162.775
162.336
152.326
149.405
135.007
129.927
127.755
126.590
126.298
125.265
121.155
114.463
55.928
40.571
40.481
40.404
40.314
40.236
40.147
40.070
39.980
39.813
39.646
39.479
O
NH
N
O
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


12.889
8.275
8.263
8.252
8.228
8.148
7.966
7.944
7.830
7.792
7.770
7.549
7.530
7.512
7.422
7.400
3.333
2.502
O
NH
N
F
3d
12
10
8
6
4
2

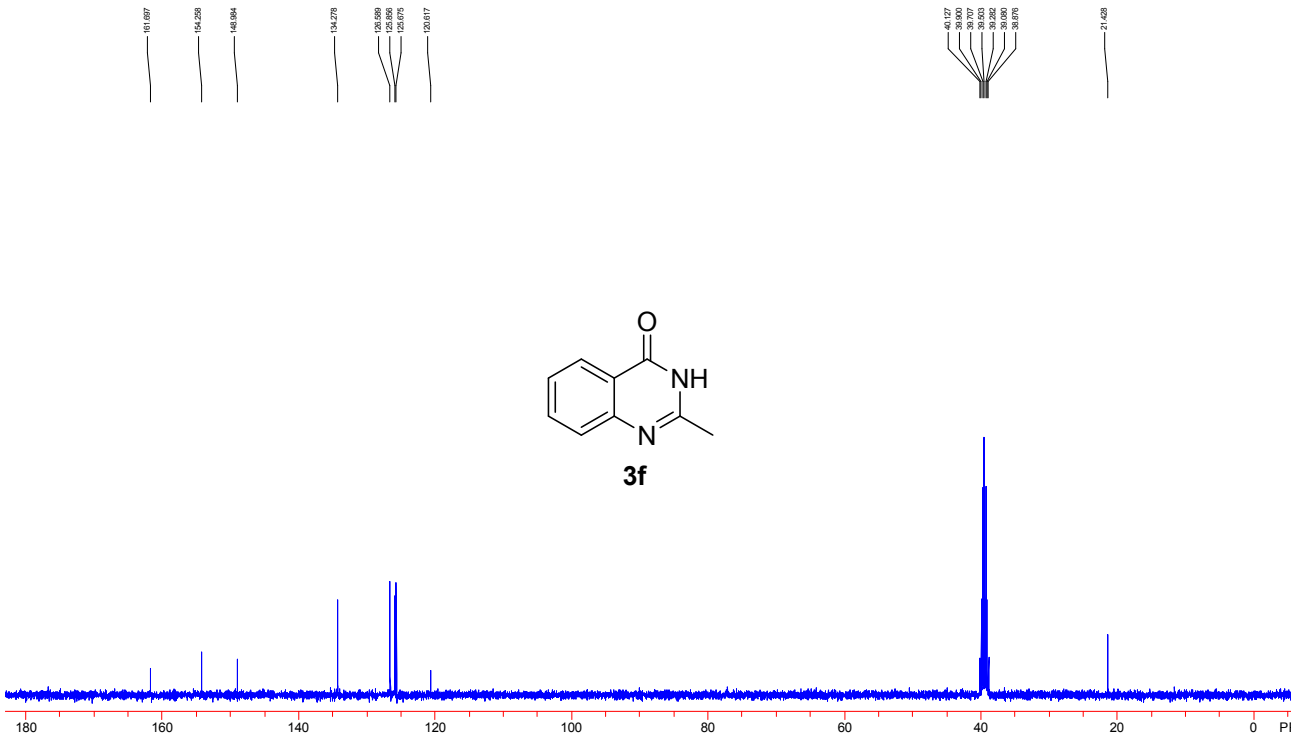

ADVANCE III BRUKER A&T Center BNU
Sample: CHX-14007 Solvent: DMSO
Spectrum: beiligong-lijiarong 0422-5 13C
165.505
163.521
162.674
151.840
149.124
135.119
130.881
130.810
129.711
129.690
127.938
127.090
126.322
121.357
116.192
116.018
40.576
40.486
40.408
40.319
40.242
40.152
40.076
39.818
39.651
39.484
31.163
O
NH
N
F
3d
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


O
NH
N
F
3e
14
12
10
8
6
4
2

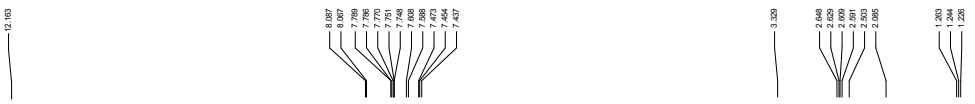

ADVANCE III BRUKER A&T Center BNU
Sample: CHX-14010 Solvent: DMSO
Spectrum: beiligong-lijiarong 0422-7 13C
163.524
162.587
161.585
151.488
148.929
135.509
135.446
135.170
131.250
131.185
128.087
127.393
126.348
124.410
124.391
121.598
118.814
118.647
115.102
114.913
40.574
40.485
40.408
40.318
40.240
40.151
40.075
39.984
39.817
39.650
39.483
O
NH
N
F
3e
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

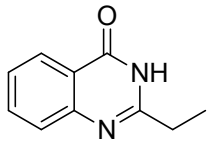

12.201
8.075
8.055
7.776
7.757
7.742
7.736
7.722
7.571
7.550
7.466
7.440
7.419
3.349
2.496
2.342
O
NH
N
12
10
8
6
4
2
0
PI

3f

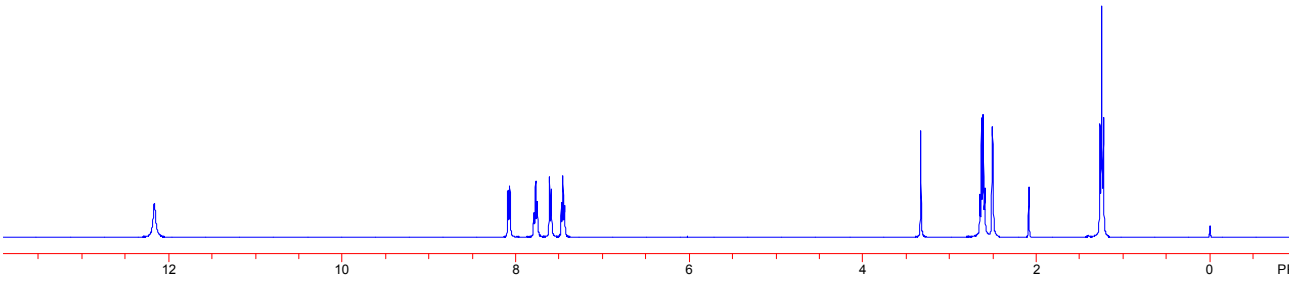

161.697
154.268
148.984
134.278
126.599
125.866
125.675
120.617
40.127
39.930
39.727
39.523
39.292
39.080
38.876
21.428
O
NH
N
180
160
140
120
100
80
60
40
20
0
PI

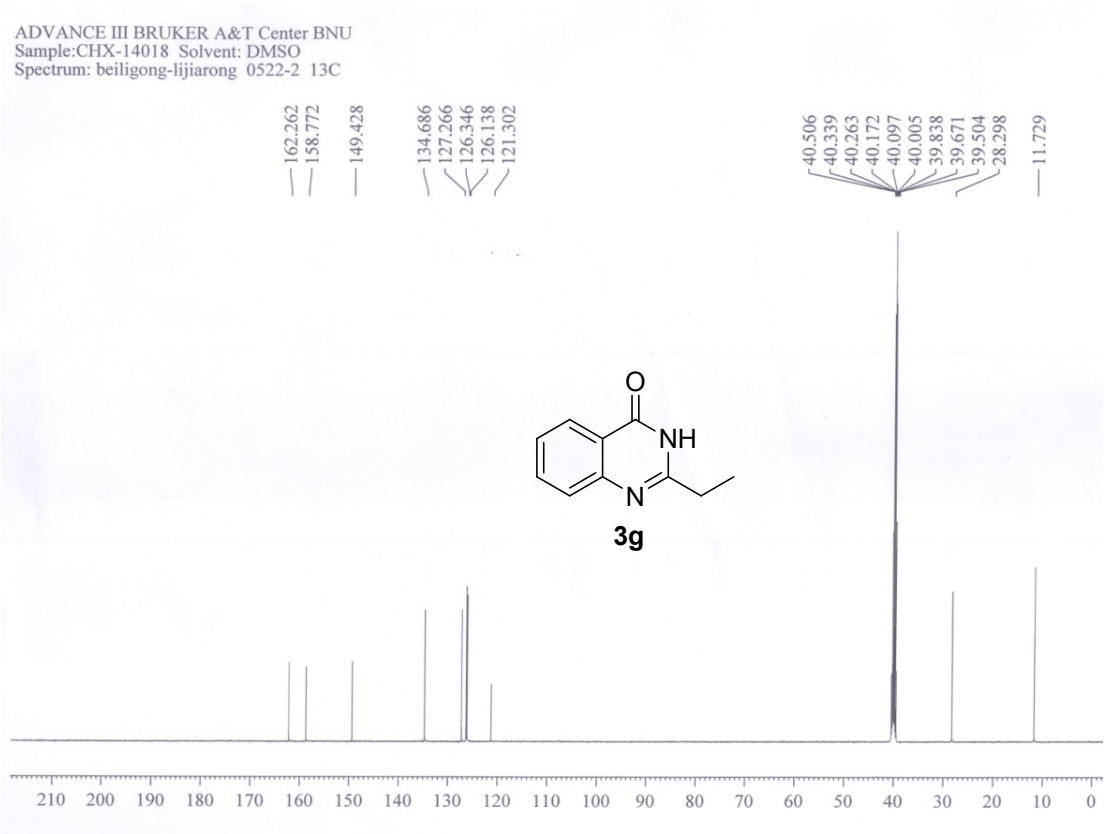
3f


12.163
8.087
8.067
7.789
7.786
7.770
7.751
7.748
7.608
7.588
7.473
7.454
7.437
3.329
2.648
2.629
2.609
2.591
2.503
2.085
1.263
1.244
1.226

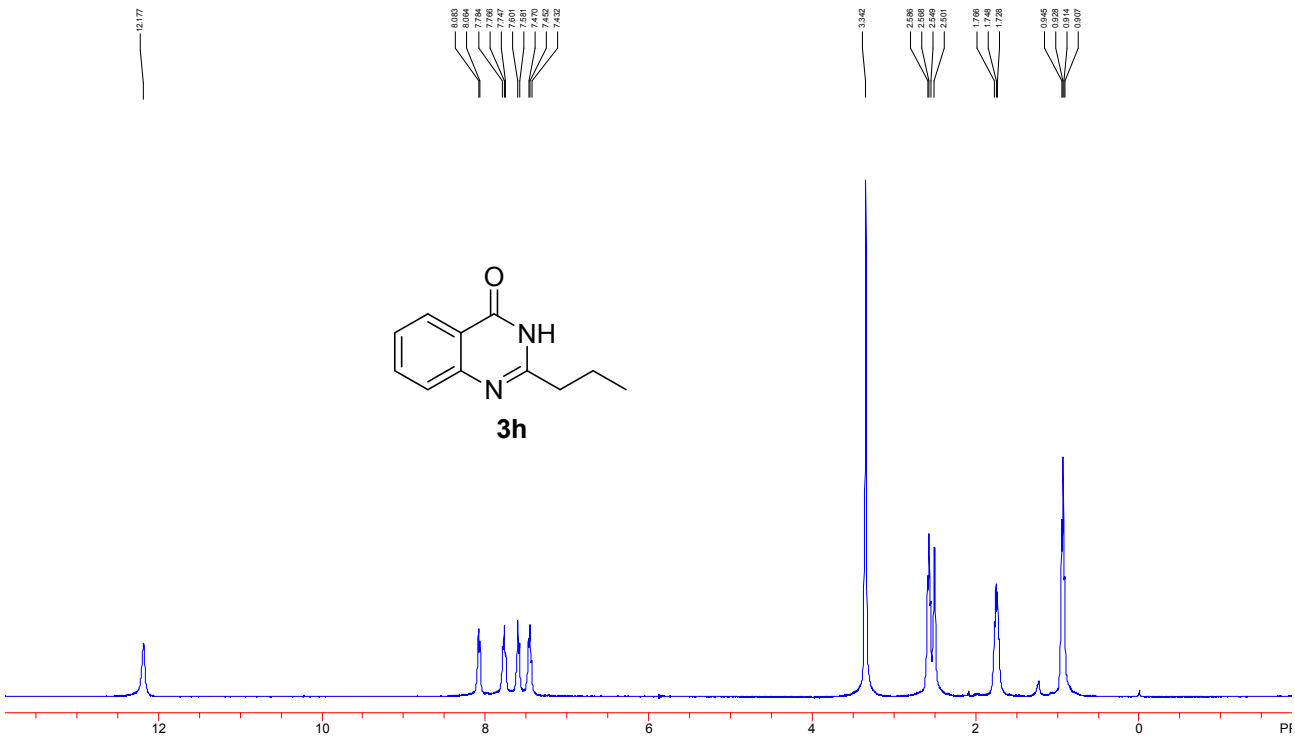

O
NH
N

3g

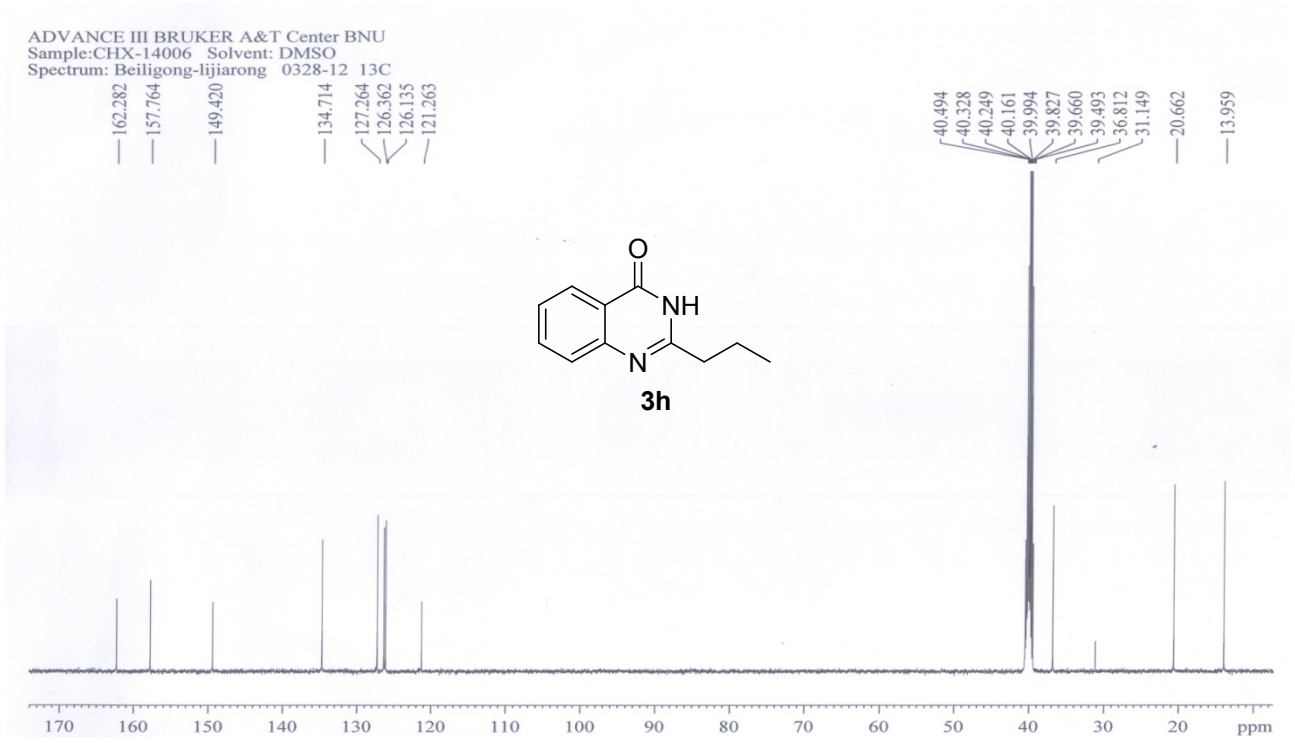

12
10
8
6
4
2
0
PI


ADVANCE III BRUKER A&T Center BNU
Sample:CHX-14018 Solvent: DMSO
Spectrum: beiligong-lijiarong 0522-2 13C
162.262
158.772
149.428
134.686
127.266
126.346
126.138
121.302
40.506
40.339
40.263
40.172
40.097
40.005
39.838
39.671
39.504
28.298
11.729
O
NH
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

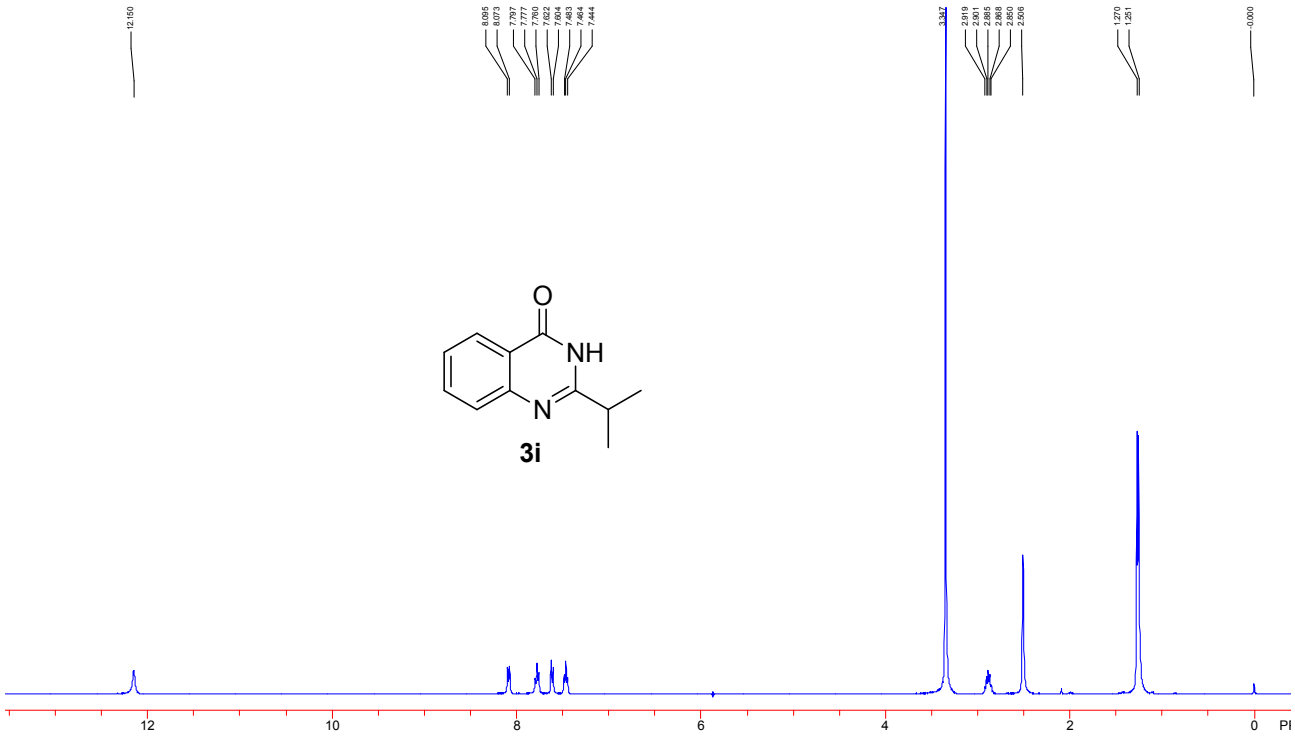
3g

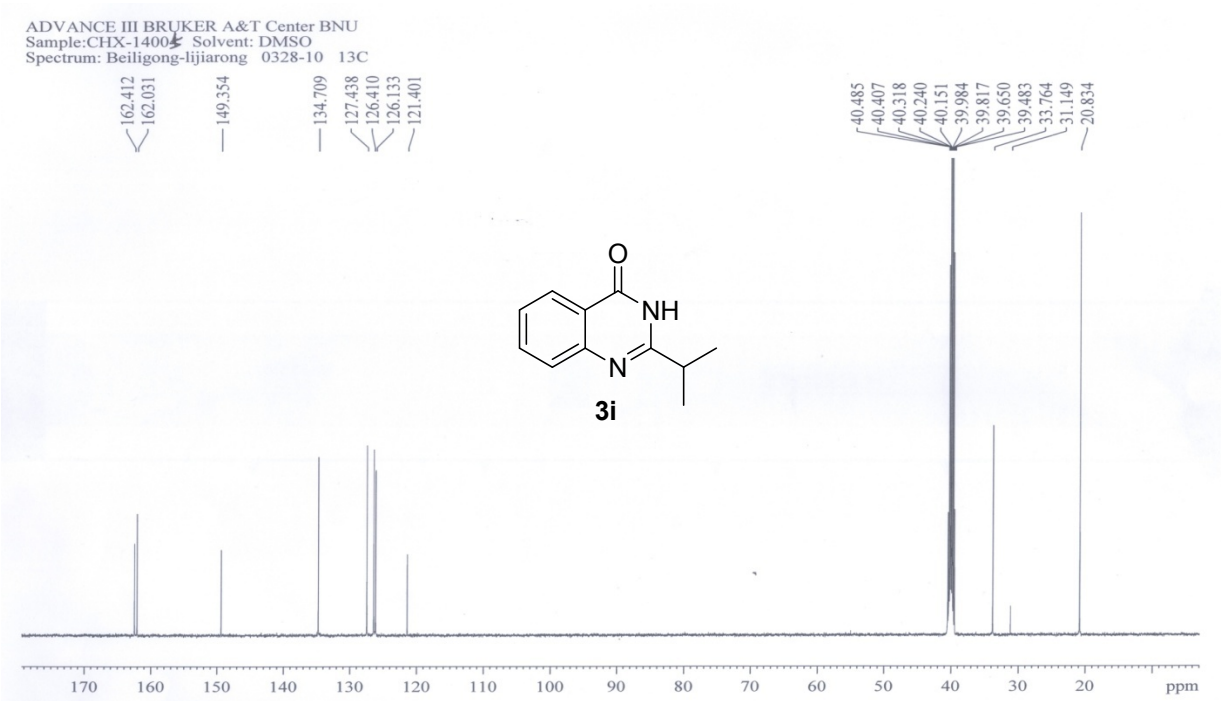

12.177
8.083
8.064
7.794
7.766
7.747
7.601
7.581
7.470
7.452
7.432
3.342
2.586
2.568
2.549
2.531
1.766
1.748
1.728
0.945
0.926
0.914
0.907
O
NH
N
12
10
8
6
4
2
0

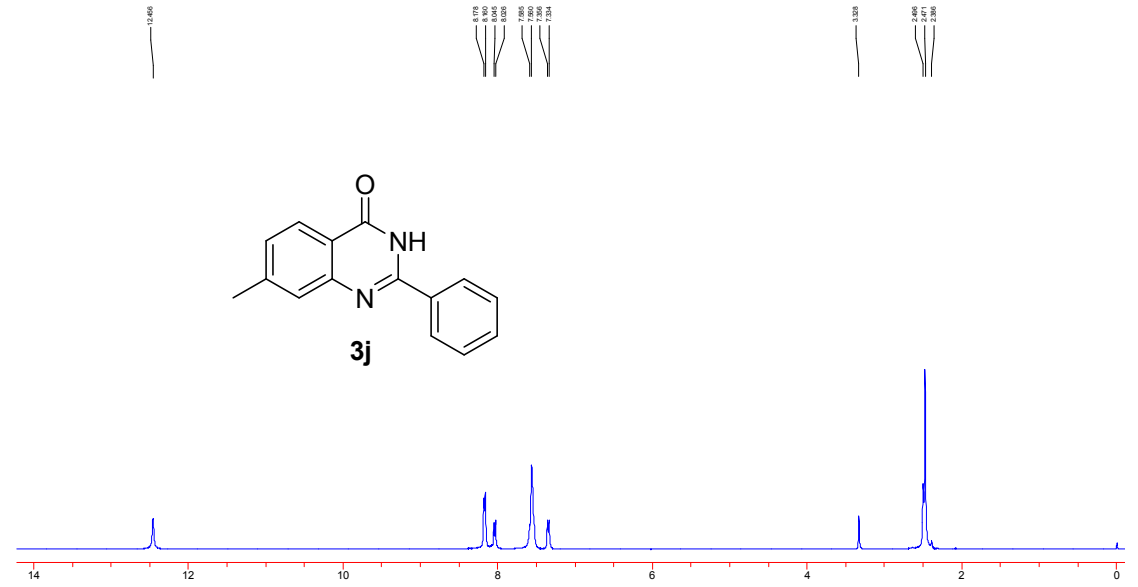
3h

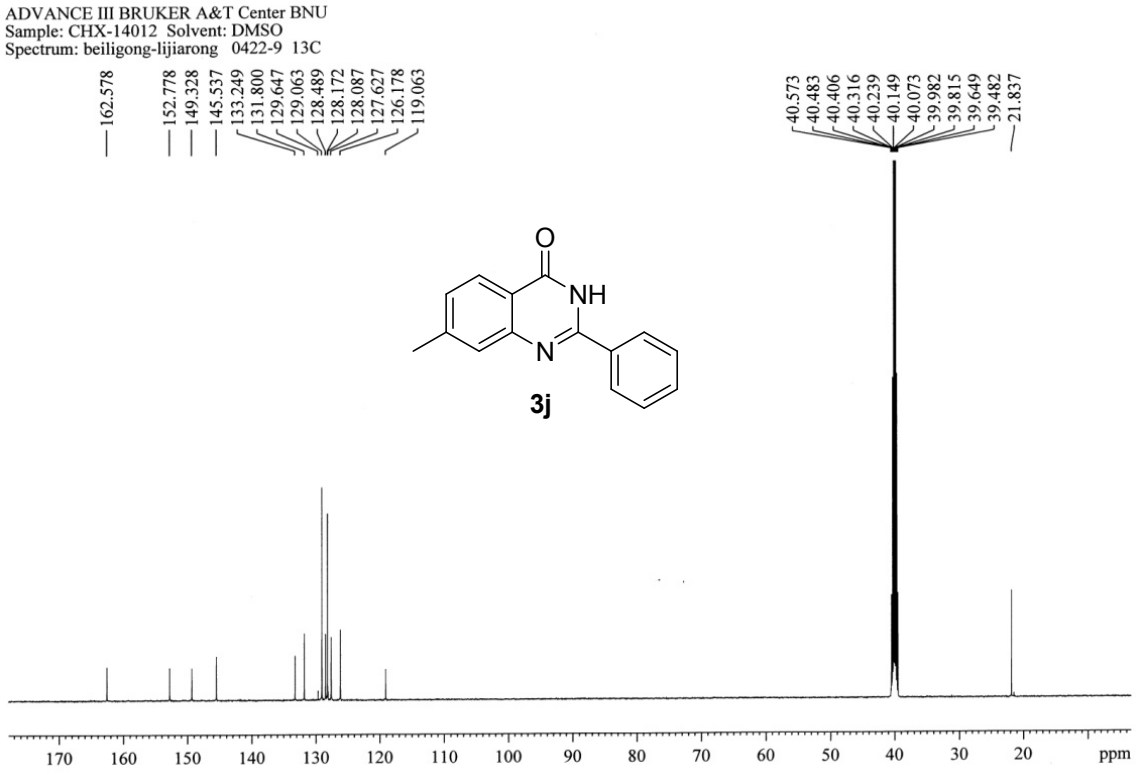

ADVANCE III BRUKER A&T Center BNU
Sample:CHX-14006 Solvent: DMSO
Spectrum: Beiligong-lijiarong 0328-12 13C
162.282
157.764
149.420
134.714
127.264
126.362
126.135
121.263
40.494
40.328
40.249
40.161
39.994
39.827
39.660
39.493
36.812
31.149
20.662
13.959
O
NH
N
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

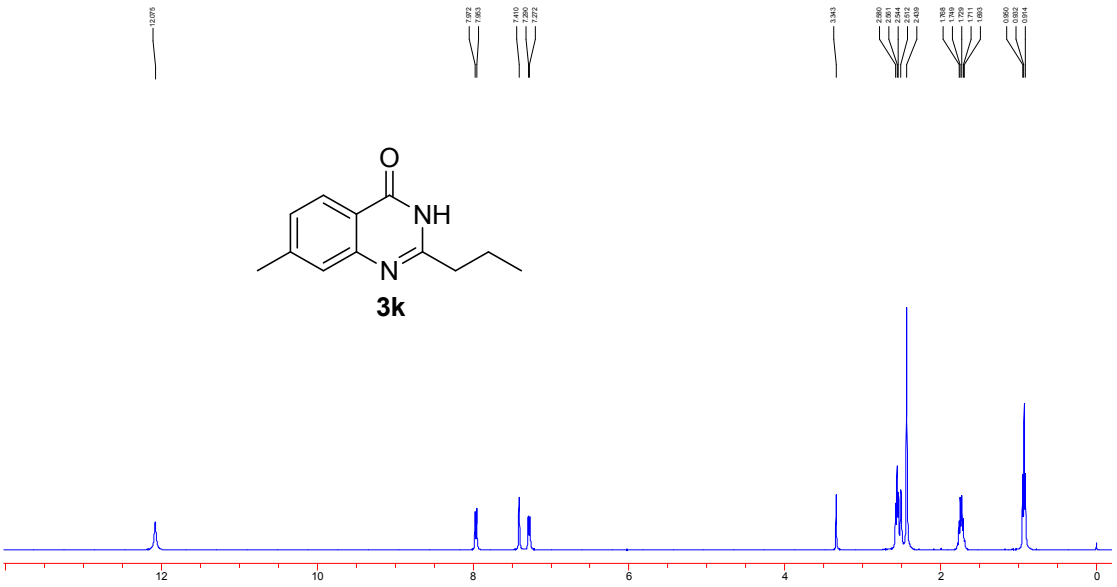
3h

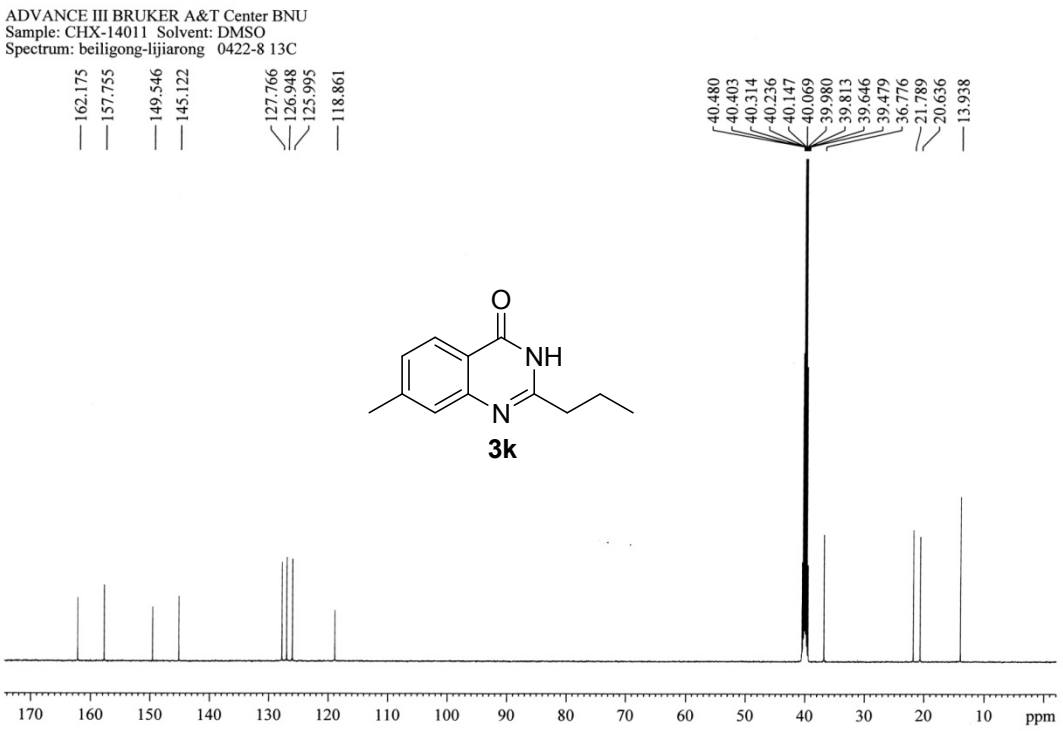

12.150
8.095
8.073
7.797
7.777
7.769
7.622
7.604
7.483
7.464
7.444
3.347
2.919
2.901
2.885
2.868
2.850
2.506
1.270
1.251
-0.000
O
NH
N
3i
12
10
8
6
4
2
0
PI

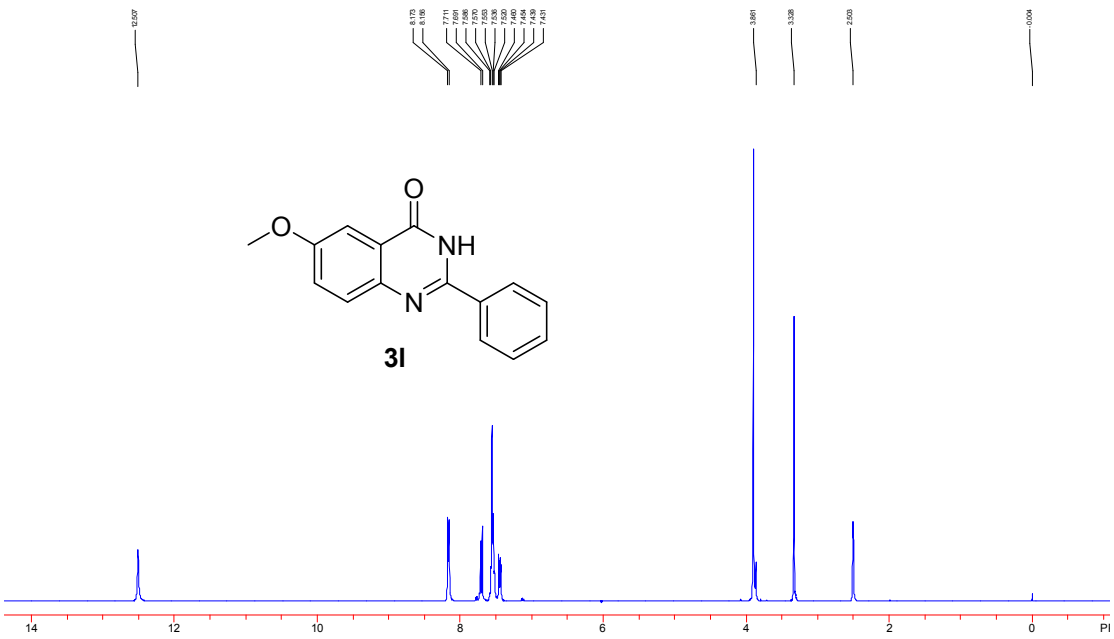

ADVANCE III BRUKER A&T Center BNU
Sample:CHX-1400 Solvent: DMSO
Spectrum: Beiligong-lijiarong 0328-10 13C
162.412
162.031
149.354
134.709
127.438
126.410
126.133
121.401
40.485
40.407
40.318
40.240
40.151
39.984
39.817
39.650
39.483
33.764
31.149
20.834
O
NH
N
3i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

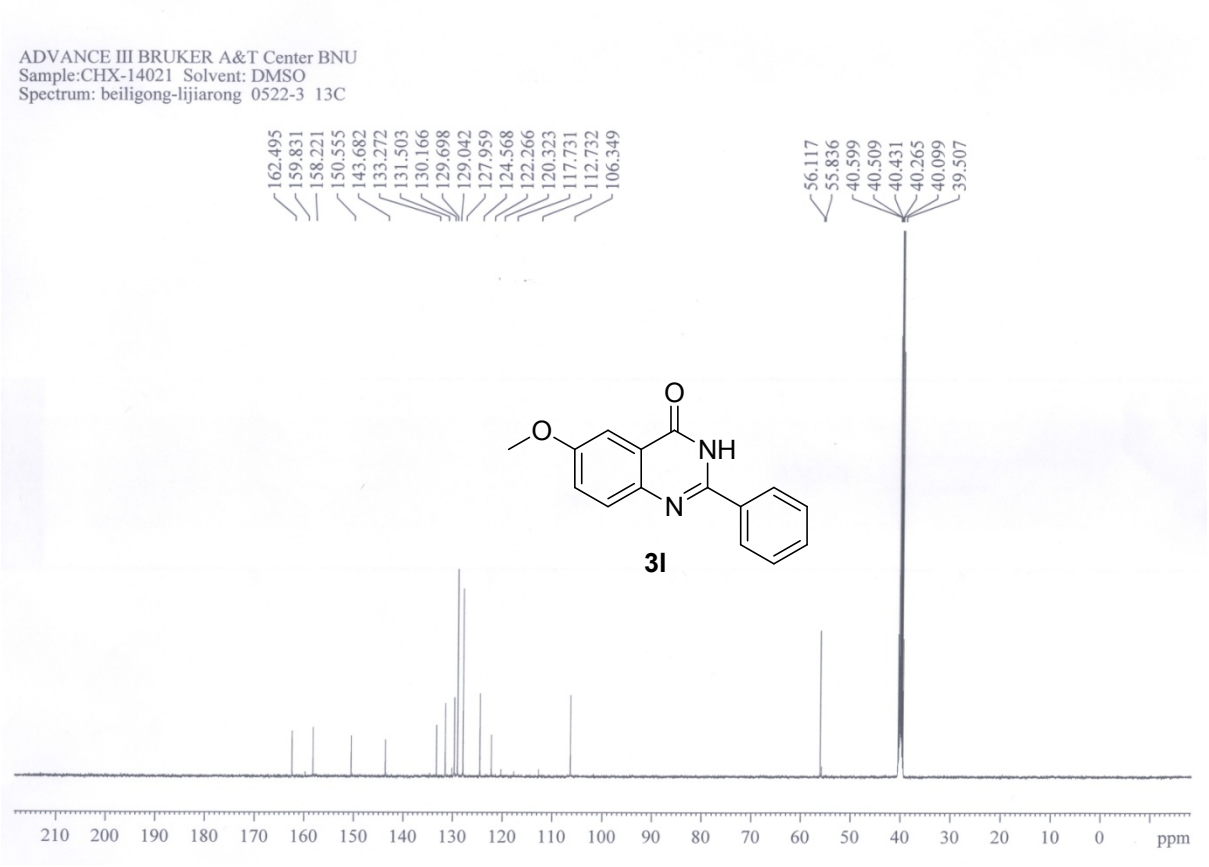

12.466
8.178
8.160
8.045
8.026
7.895
7.892
7.350
7.334
3.328
2.496
2.491
2.487
2.360
O
NH
N
3j
14
12
10
8
6
4
2
0

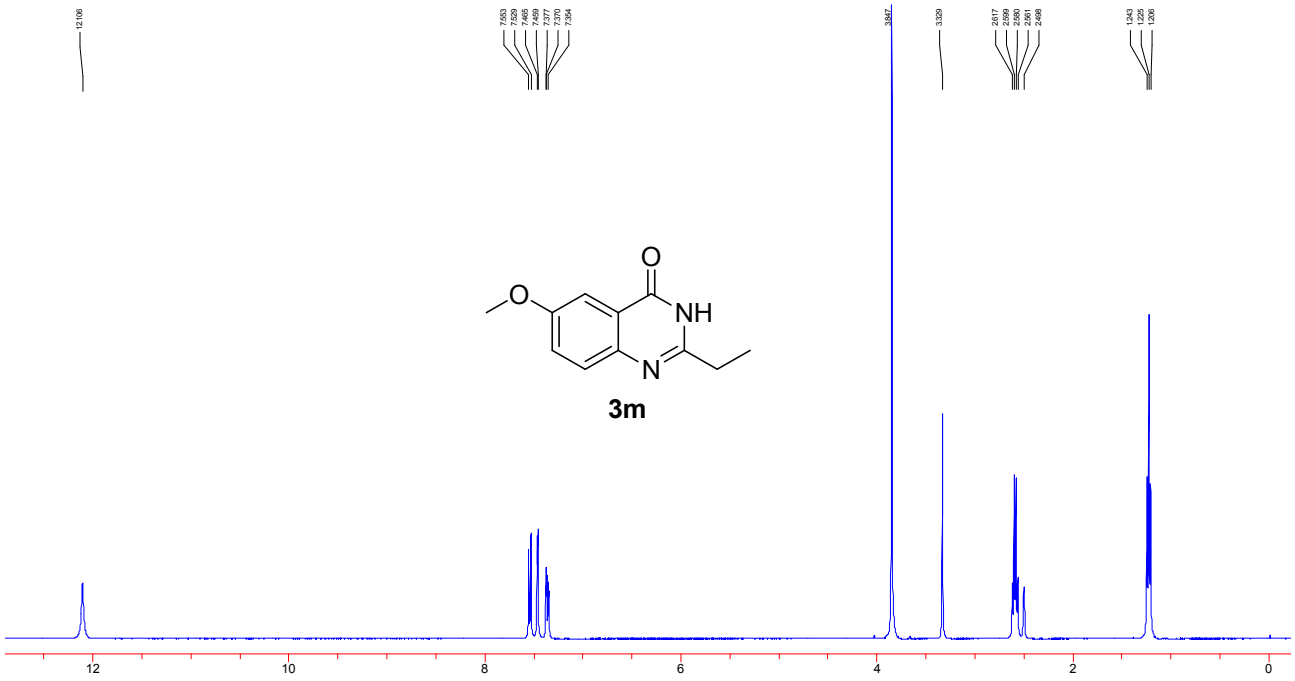

ADVANCE III BRUKER A&T Center BNU
Sample: CHX-14012 Solvent: DMSO
Spectrum: beiligong-lijiarong 0422-9 13C
162.578
152.778
149.328
145.537
133.249
131.800
129.647
129.063
128.489
128.172
128.087
127.627
126.178
119.063
40.573
40.483
40.406
40.316
40.239
40.149
40.073
39.982
39.815
39.649
39.482
21.837
O
NH
N
3j
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


12.075
7.872
7.863
7.410
7.290
7.272
3.343
2.890
2.881
2.544
2.534
2.459
1.768
1.758
1.729
1.711
1.695
0.950
0.932
0.914
O
NH
N
3k
12
10
8
6
4
2
0

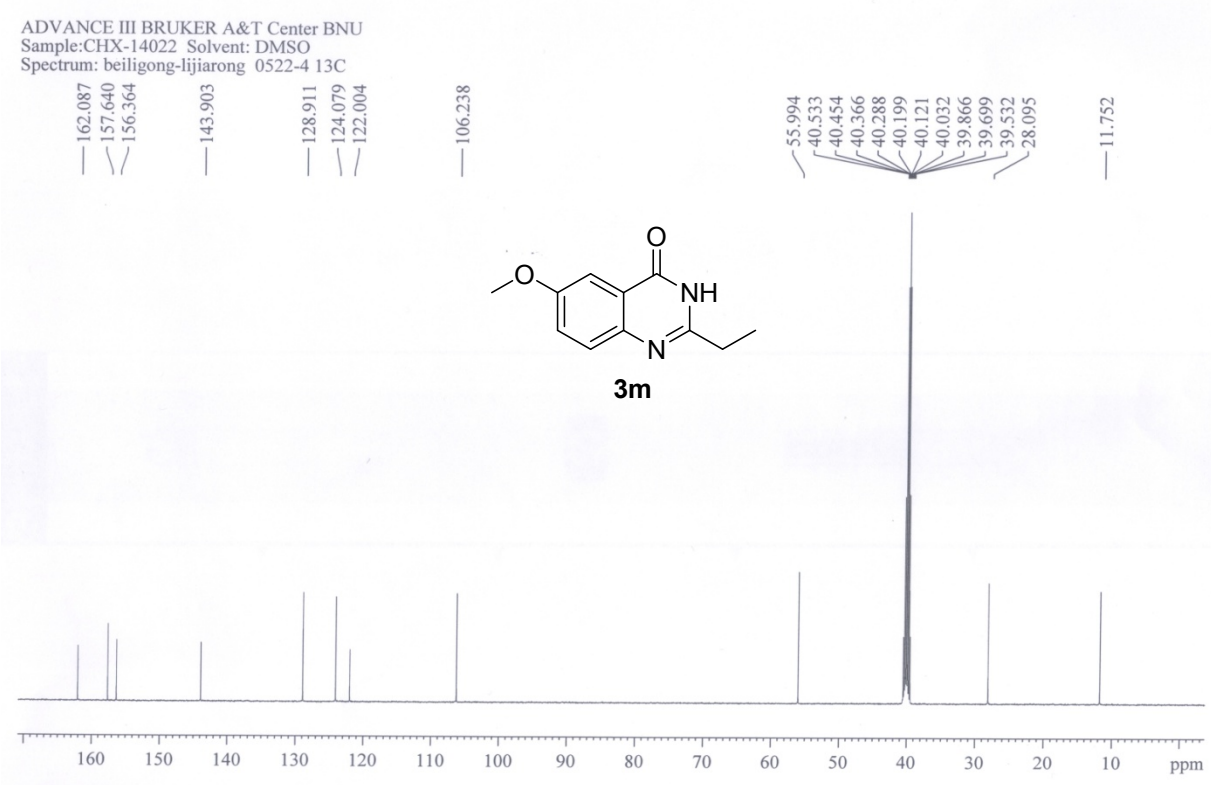

ADVANCE III BRUKER A&T Center BNU
Sample: CHX-14011 Solvent: DMSO
Spectrum: beiligong-lijiarong 0422-8 13C
162.175
157.755
149.546
145.122
127.766
126.948
125.995
118.861
40.480
40.403
40.314
40.236
40.147
40.069
39.980
39.813
39.646
39.479
36.776
21.789
20.636
13.938
O
NH
N
3k
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


12.507
8.173
8.156
7.711
7.691
7.686
7.673
7.655
7.538
7.520
7.480
7.464
7.439
7.421
3.861
3.328
2.505
1.004
O
NH
N
O
3l
14
12
10
8
6
4
2
0
PI

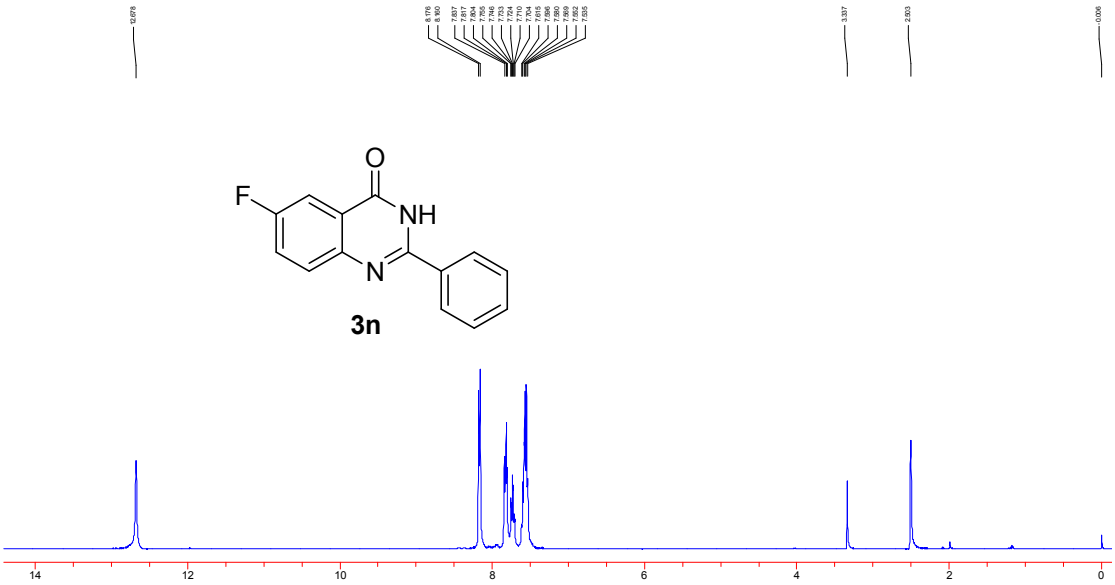

ADVANCE III BRUKER A&T Center BNU
Sample:CHX-14021 Solvent: DMSO
Spectrum: beiligong-lijiarong 0522-3 13C
162.495
159.831
158.221
150.555
143.682
133.272
131.503
130.166
129.698
129.042
127.959
124.568
122.266
120.323
117.731
112.732
106.349
56.117
55.836
40.599
40.509
40.431
40.265
40.099
39.507
O
NH
N
O
3l
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

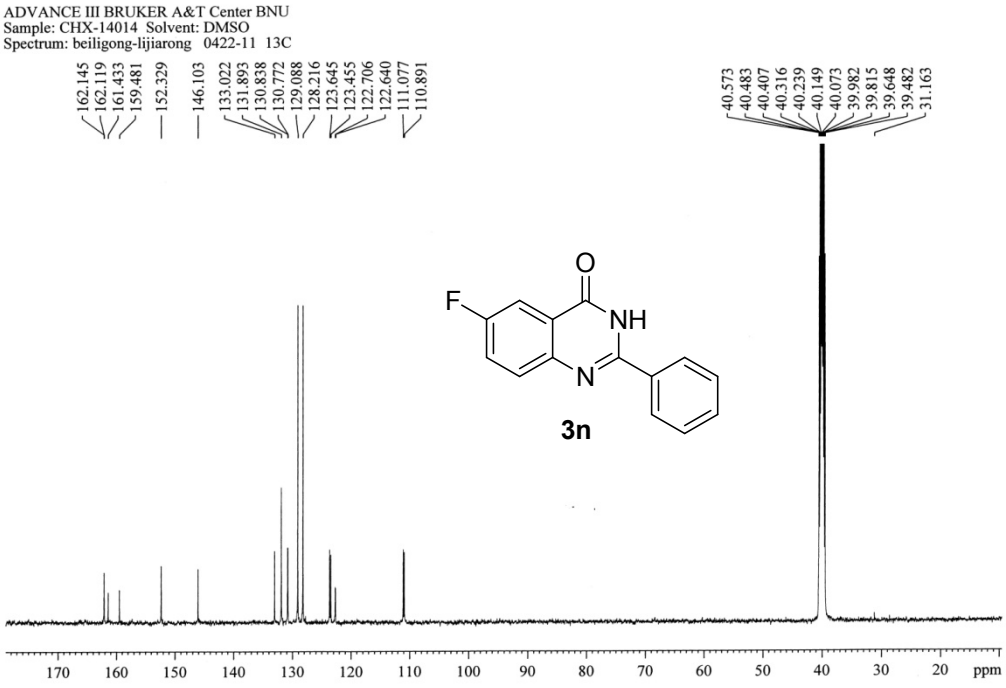

12.106
7.553
7.529
7.465
7.449
7.377
7.370
7.354
3.847
3.329
2.617
2.599
2.580
2.561
2.448
1.243
1.225
1.206
O
O
NH
N
12
10
8
6
4
2
0

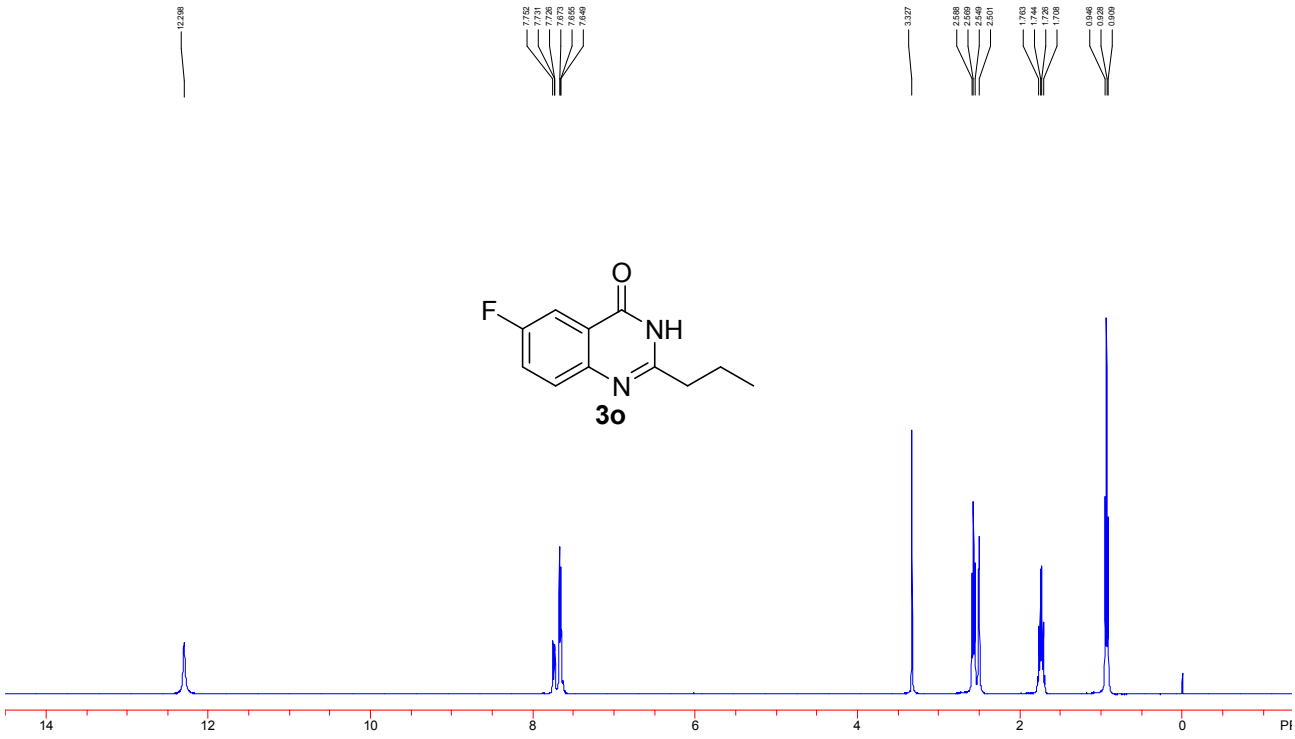
3m

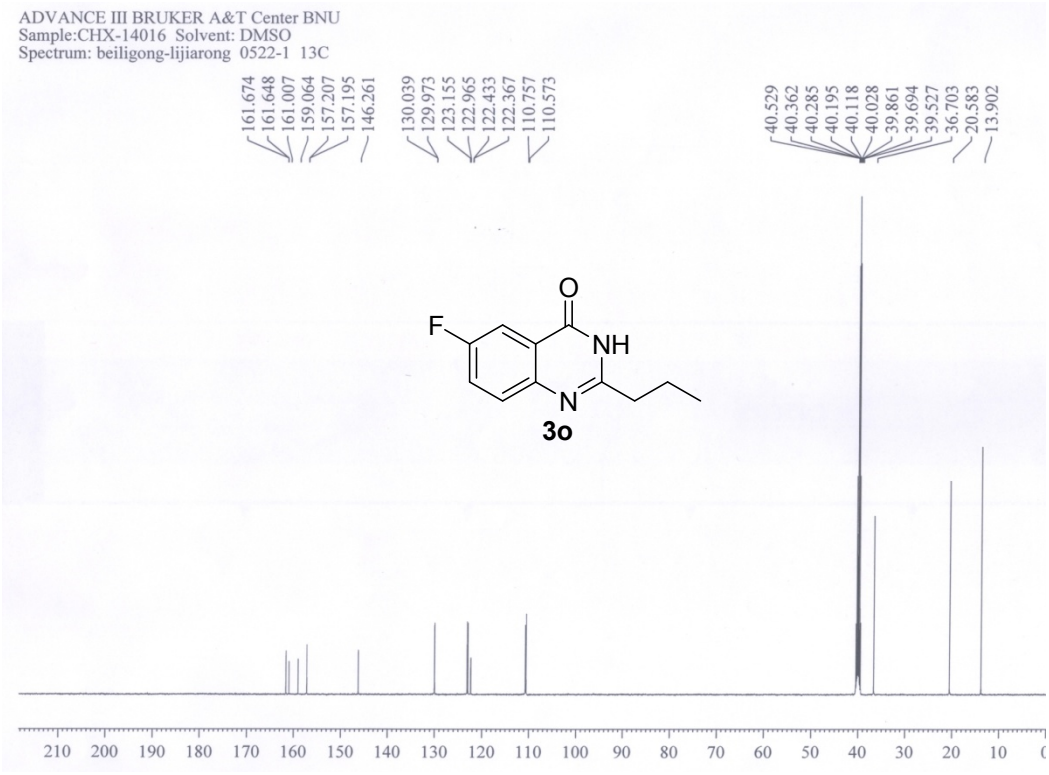

ADVANCE III BRUKER A&T Center BNU
Sample:CHX-14022 Solvent: DMSO
Spectrum: beiligong-lijiarong 0522-4 13C
162.087
157.640
156.364
143.903
128.911
124.079
122.004
106.238
55.994
40.533
40.454
40.366
40.288
40.199
40.121
40.032
39.866
39.699
39.532
28.095
11.752
O
O
NH
N
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

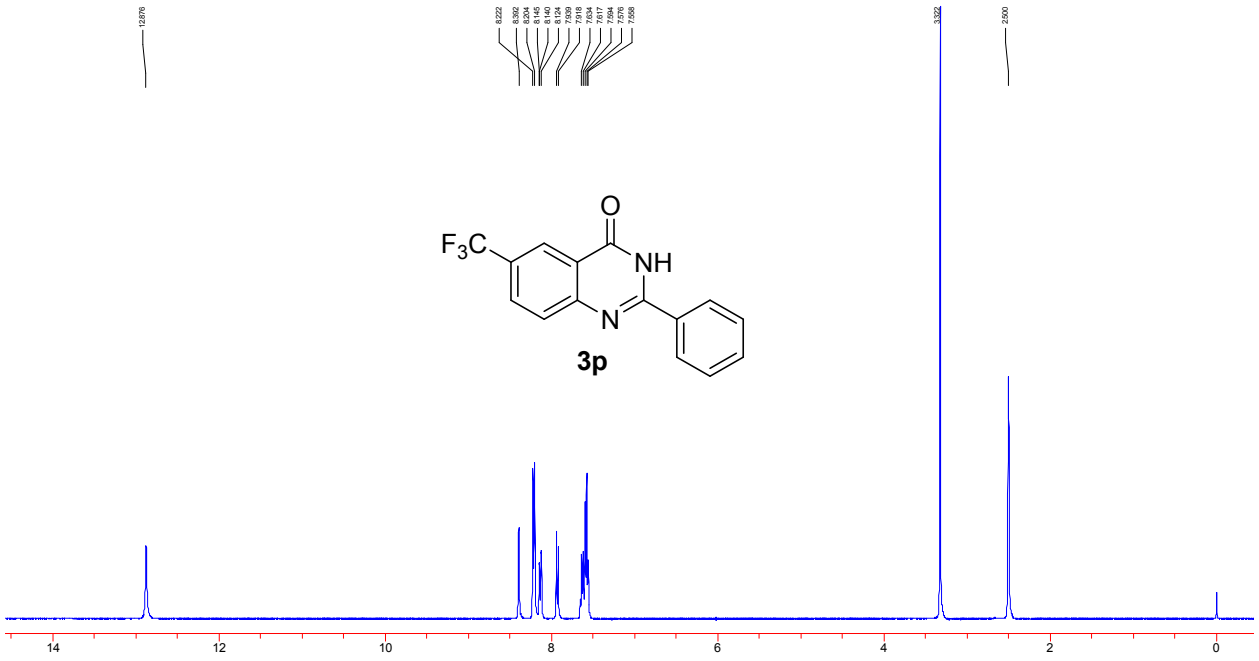
3m

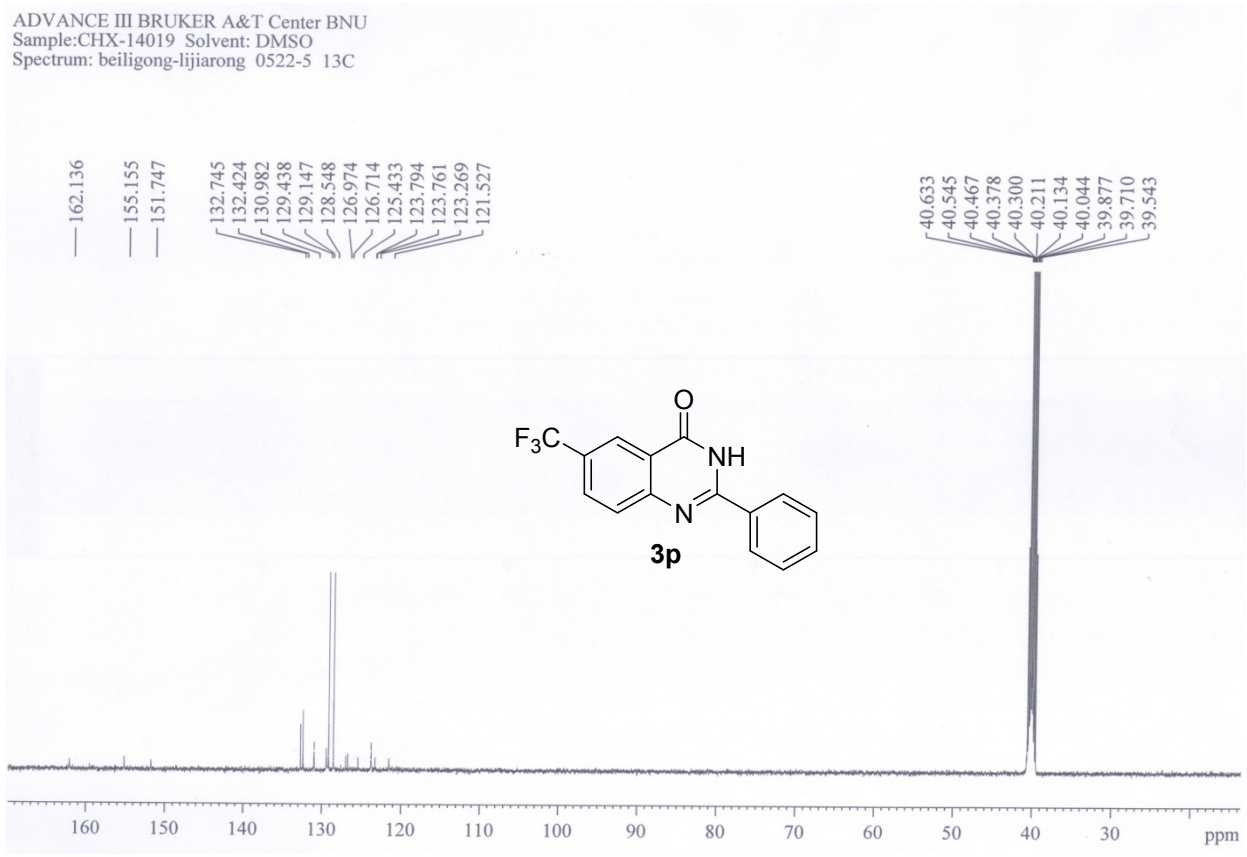

12.878
8.176
8.160
7.857
7.851
7.844
7.765
7.748
7.733
7.724
7.710
7.701
7.694
7.615
7.586
7.580
7.569
7.562
7.555
3.337
2.503
0.006
O
F
NH
N
3n
14
12
10
8
6
4
2
0

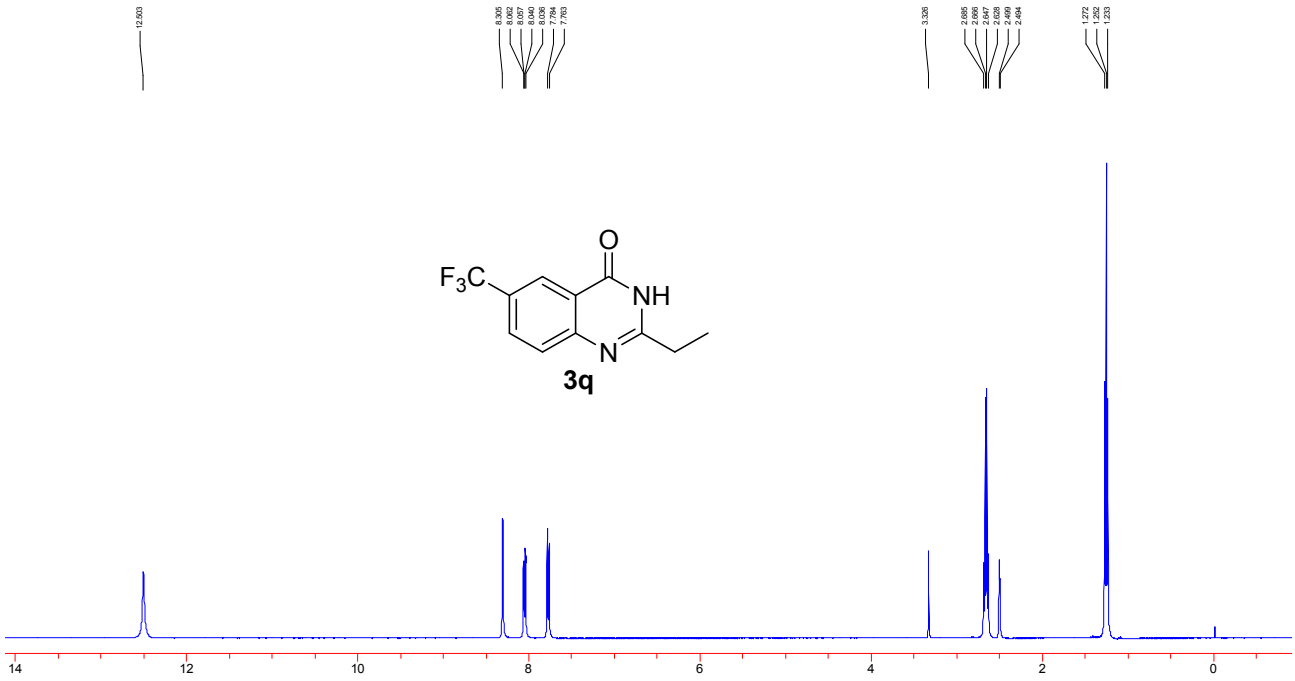

ADVANCE III BRUKER A&T Center BNU
Sample: CHX-14014 Solvent: DMSO
Spectrum: beiligong-lijiarong 0422-11 13C
162.145
162.119
161.433
159.481
152.329
146.103
133.022
131.893
130.838
130.772
129.088
128.216
123.645
123.455
122.706
122.640
111.077
110.891
40.573
40.483
40.407
40.316
40.239
40.149
40.073
39.982
39.815
39.648
39.482
31.163
O
F
NH
N
3n
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

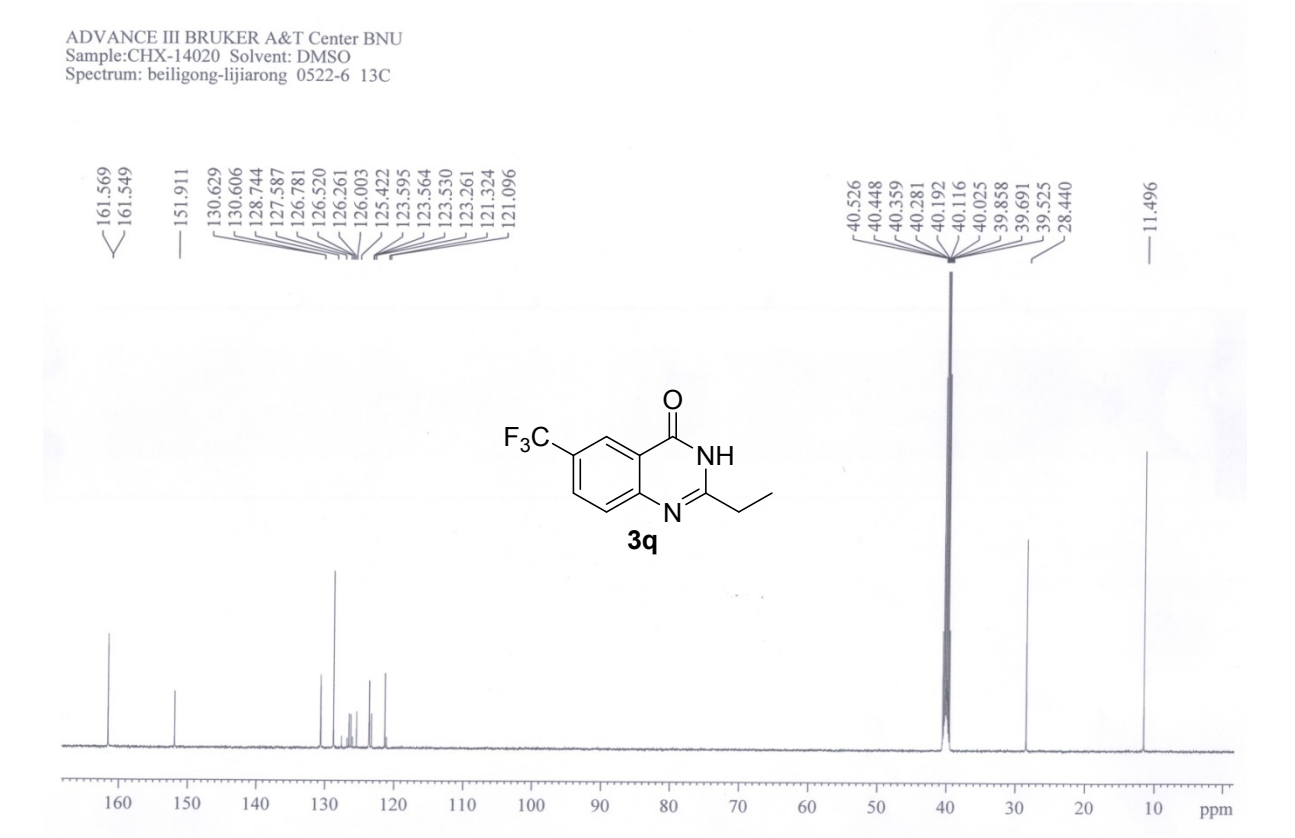

12.298
7.752
7.731
7.726
7.673
7.655
7.649
3.327
2.588
2.569
2.549
2.501
1.763
1.744
1.726
1.708
0.946
0.928
0.909
O
F
NH
N
3o
14
12
10
8
6
4
2
0
PI

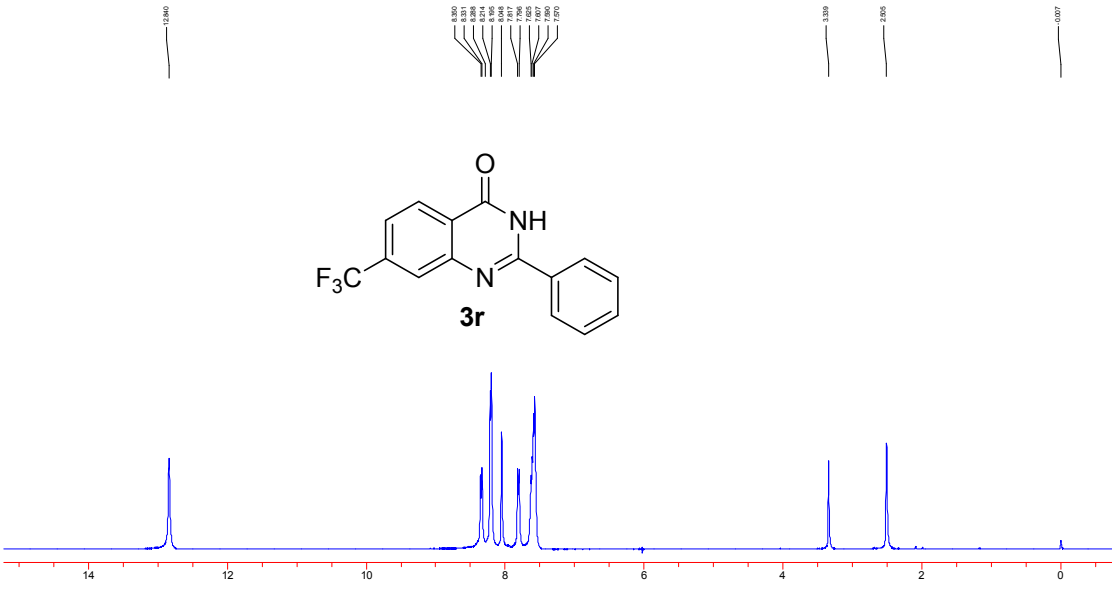

ADVANCE III BRUKER A&T Center BNU
Sample:CHX-14016 Solvent: DMSO
Spectrum: beiligong-lijiarong 0522-1 13C
161.674
161.648
161.007
159.064
157.207
157.195
146.261
130.039
129.973
123.155
122.965
122.433
122.367
110.757
110.573
40.529
40.362
40.285
40.195
40.118
40.028
39.861
39.694
39.527
36.703
20.583
13.902
O
F
NH
N
3o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

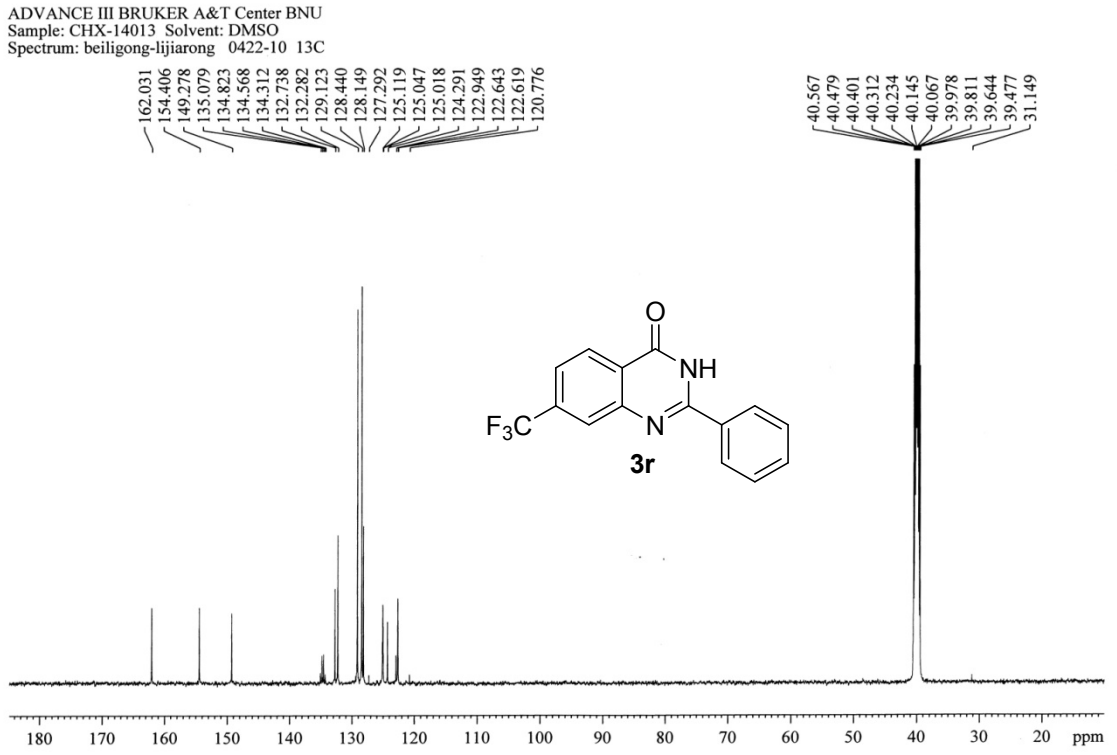

12.676
8.222
8.202
8.204
8.145
8.140
8.124
7.939
7.936
7.634
7.617
7.594
7.578
7.558
3.322
2.500
O
F3C
NH
N
3p
14
12
10
8
6
4
2
0


ADVANCE III BRUKER A&T Center BNU
Sample:CHX-14019 Solvent: DMSO
Spectrum: beiligong-lijiarong 0522-5 13C
162.136
155.155
151.747
132.745
132.424
130.982
129.438
129.147
128.548
126.974
126.714
125.433
123.794
123.761
123.269
121.527
40.633
40.545
40.467
40.378
40.300
40.211
40.134
40.044
39.877
39.710
39.543
O
F3C
NH
N
3p
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


12.503
8.309
8.092
8.057
8.040
8.036
7.784
7.763
3.326
2.685
2.666
2.647
2.628
2.499
2.494
1.272
1.253
1.233
O
F3C
NH
N
3q
14
12
10
8
6
4
2
0


ADVANCE III BRUKER A&T Center BNU
Sample:CHX-14020 Solvent: DMSO
Spectrum: beiligong-lijiarong 0522-6 13C
161.569
161.549
151.911
130.629
130.606
128.744
127.587
126.781
126.520
126.261
126.003
125.422
123.595
123.564
123.530
123.261
121.324
121.096
40.526
40.448
40.359
40.281
40.192
40.116
40.025
39.858
39.691
39.525
28.440
11.496
O
F3C
NH
N
3q
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


12.840
8.300
8.301
8.298
8.214
8.195
8.193
7.617
7.615
7.605
7.603
7.601
7.600
7.590
7.588
3.339
2.505
0.007
O
NH
F3C
N
3r
14
12
10
8
6
4
2
0


ADVANCE III BRUKER A&T Center BNU
Sample: CHX-14013 Solvent: DMSO
Spectrum: beiligong-lijiarong 0422-10 13C
162.031
154.406
149.278
135.079
134.823
134.568
134.312
132.738
132.282
129.123
128.440
128.149
127.292
125.119
125.047
125.018
124.291
122.949
122.643
122.619
120.776
40.567
40.479
40.401
40.312
40.234
40.145
40.067
39.978
39.811
39.644
39.477
31.149
O
NH
F3C
N
3r
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm