Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Diversified facile synthesis of benzimidazoles, quinazolin-4(*3H*)-ones and 1,4-benzodiazepine-2,5-diones via palladium-catalyzed transfer hydrogenation/condensation cascade of nitroarenes under microwave irradiation

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Supporting Information

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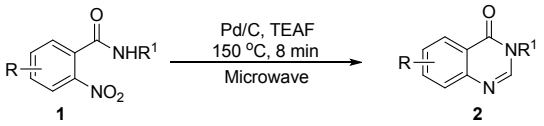
1. General experimental details

o-nitrobenzamides (**1**)and 2-nitrobenzoyl-α-amino acid methyl esters (**5**) were prepared according to the reported method,[1] other chemicals were purchased from Aladdin. All the reactions and manipulations were performed in atmosphere. All reactions were monitored by TLC with silica gel-coated plates. NMR spectra were recorded on BRUKER DRX 400 or 500 spectrometers. Chemical shifts are reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (*J*) were reported in Hz and referred to apparent peak multiplications. High resolution mass spectra (HRMS) were recorded on Bruker microTOF mass instrument (ESI). IR spectra were recorded on BRUKER TENSOR 27 spectrometers.Biotage356007 was used as the microwave reactor for the palladium-catalyzed transfer hydrogenaation/condensation cascade.

2. Synthesis of Benzimidazoles, Quinazolin-4(*3H*)-ones and 1, 4-Benzodiazepine-2, 5-diones

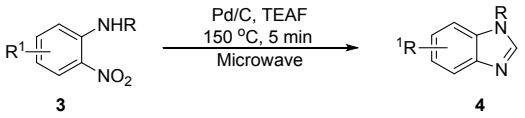
2.1 Synthesis of Quinazolin-4(*3H*)-ones (**2**)



1 **2**

General procedure: 1.0 mmol *o*-nitrobenzamides(**1**)was dissolved in 0.8 g of TEAF (4.0 mmol, 4.0 equiv), 5.5 mg of 10% Pd/C (0.5mol%) was added. The suspension was then heated by microwave at 150 °C for 8 min. The completion of reaction was monitored by TLC. The reaction mixture was mixed with 15 mL of methanol and filtered through a Celite pad to remove the catalyst. Methanol was evaporated and saturated aqueous sodium bicarbonate solution (10 mL) was added to the residue. After gas evolution, the solution was extracted with EtOAc (15 mL X 3) and the combined organic layer was dried over Na_2SO_4. Evaporation of EtOAc gave crystalline product **2**in most cases. Otherwise flash chromatography was applied.

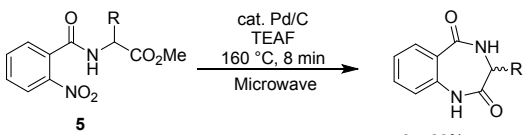
2.2 Synthesis of Benzimidazoles (**4**)



3 **4**

General procedure: 1.0 mmol *o*-nitroaniline (**3**) was dissolved in 0.8 g of TEAF (4.0 mmol, 4.0 equiv), 5.5 mg of 10% Pd/C (0.5mol%) was added. The suspension was then heated by microwave at 150 °C for 5 min. The completion of reaction was monitored by TLC. The reaction mixture was mixed with 15 mL of methanol and filtered through a Celite pad to remove the catalyst. Methanol was evaporated and saturated aqueous sodium bicarbonate solution (10 mL) was added to the residue. After gas evolution, the solution was extracted with EtOAc (15 mL X 3) and dried over Na_2SO_4. Evaporation of EtOAc gave crystalline product in most cases.Otherwise flash chromatography was applied.

2.3 Synthesis of 1,4-benzodiazepine-2,5-diones (**6**)



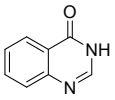
5
R = H, **5a**
R = Bn, **5b**

6a, 68%
6b, 98% (configuration not identified)

General procedure: 1.0 mmol 2-nitrobenzoyl-α-amino acid methyl esters (**5**) was dissolved in 0.8 g of TEAF

(4.0 mmol, 4.0 equiv), 5.5 mg of 10% Pd/C (0.5 mol%) was added. The suspension was then heated by microwave at 160 °C for 8 min. The completion of reaction was monitored by TLC. The reaction mixture was mixed with 15 mL of methanol and filtered through a Celite pad to remove the catalyst. Methanol was evaporated and saturated aqueous sodium bicarbonate solution (10 mL) was added to the residue. After gas evolution, the solution was extracted with EtOAc (15 mL X 3) and dried over Na_2SO_4. Evaporation of EtOAc gave crystalline product in most cases. Otherwise flash chromatography was applied.

3. Experimental characterization data for products



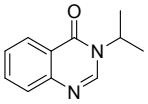
2a

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

All the characterization data are consistent with the previous report.[2]

^{1}H-NMR (500 MHz, DMSO-d_6): δ (ppm) 7.53 (t, 1H, *J* = 8.0Hz), 7.67 (d, 1H, *J* = 8.0Hz), 7.82 (dt, 1H, *J* = 8.0, 1.5Hz), 8.09 (s, 1H), 8.12 (t, 1H, *J* = 8.0, 1.5Hz), 12.24 (brs, 1H);

HR-MS (ESI) calcd for $C_8H_7N_2O$ $[M + H]^+$147.0553,found 147.0553.



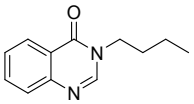
2b

3-isopropyl-quinazolin-4(*3H*)-one (**2b**)

All the characterization data are consistent with the previous report.[3]

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.32 (d, *J* = 8.0 Hz, 1H), 8.13 (s, 1H), 7.78–7.69 (m, 2H), 7.50 (dd, *J* = 6.4, 1.6 Hz, 1 H), 5.21 (m, 1H), 1.50 (d, *J* = 6.8 Hz, 6H);

HR-MS (ESI) calcd for $C_{11}H_{13}N_2O$ $[M + H]^+$189.1028, found 189.1030.



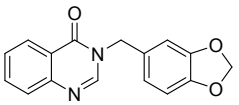
2c

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

All the characterization data are consistent with the previous report.[4]

^{1}H-NMR (500 MHz, DMSO-d_6): δ (ppm) 8.40 (s, 1H), 8.16 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.82 (ddd, *J* = 8.5, 7.0, 1.5 Hz, 1H), 7.68 (dd,*J* = 8.0, 1.0 Hz, 1H), 7.55 (ddd, *J* = 8.5, 7.0, 1.0 Hz, 1H), 3.98 (t, *J* = 7.0 Hz, 2H), 1.73-1.62 (m, 2H), 1.31 (q, *J* = 7.5 Hz, 2H), 0.91 (t, *J* = 7.5 Hz, 3H);

HR-MS (ESI) calcd for $C_{12}H_{15}N_2O$ $[M + H]^+$ 203.1179, found 203.1179.



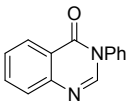
2d

3-(benzo[d][1,3]dioxol-5-ylmethyl)quinazolin-4(*3H*)-one (**2d)**

All the characterization data are consistent with the previous report.[5]

^{1}H-NMR (500 MHz, DMSO-d_6): δ (ppm) 8.56 (s, 1H), 8.16 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.83 (ddd, *J* = 8.5, 7.0, 1.5 Hz,1H), 7.72-7.65 (m, 1H), 7.60-7.48 (m, 1H), 7.02 (d, *J* = 1.5 Hz, 1H) 6.93-6.82 (m, 2H), 5.98 (s, 2H), 5.09 (s, 2H);

HR-MS (ESI) calcd for $C_{16}H_{13}N_2O_3[M+H]^+$ 281.0921, found281.0922.



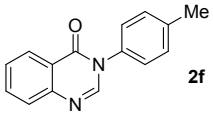
2e

3-Phenylquinazolin-4(*3H*)-one (**2e**)

All the characterization data are consistent with the previous report.[4]

^{1}H-NMR (400 MHz,DMSO-d_6): δ (ppm) 8.36 (s, 1H), 8.22 (dd, *J*= 8.0, 0.8 Hz, 1H), 7.88-7.92 (m, 1H),7.76 (d, *J*= 8.0 Hz,1H),7.52-7.63 (m, 6H);

HR-MS (ESI) calcd for $C_{14}H_{11}N_2O[M+H]^+$223.0871, found223.0869.



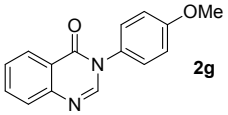
2f

3-*p*-tolylquinazolin-4(*3H*)-one (**2f**)

All the characterization data are consistent with the previous report.[4]

^{1}H-NMR (400 MHz,DMSO-d_6): 8.32 (s, 1H), 8.20 (dd, *J*= 8.0, 0.8 Hz, 1H), 7.86–7.90 (m, 1H),7.74 (d, *J*= 8.0 Hz,1H), 7.57–7.61 (m, 1H),7.42 (d, *J*= 8.0 Hz, 2H),7.36 (d, *J*= 8.0 Hz, 1H), 2.39 (s, 3H);

HR-MS (ESI) calcd for $C_{15}H_{13}N_2O[M+H]^+$237.1028, found 237.1030.



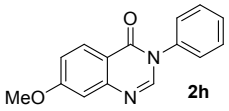
2g

3-(4-Methoxyphenyl)quinazolin-4(*3H*)-one (**2g**)

All the characterization data are consistent with the previous report.[4]

^{1}H-NMR (DMSO-d_6, 400 MHz): 8.31 (s, 1H), 8.20 (d, *J*= 7.6 Hz, 1H), 7.86-7.90 (m, 1H), 7.74(d, *J*= 8.0 Hz, 1H),7.58-7.62 (m, 1H),7.47 (d, *J*= 8.8 Hz, 2H), 7.10 (d,*J*= 8.8 Hz, 2H),3.84 (s, 3H);

HR-MS (ESI) calcd for $C_{14}H_{11}N_2O_2[M+H]^+$253.0977, found253.0979.



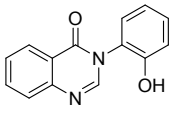
2h

7-methoxy-3-phenylquinazolin-4(*3H*)-one (**2h**)

^{1}H-NMR (500 MHz, $CDCl_3$): δ (ppm) 8.33 (d, *J* = 9.0 Hz, 1H), 8.17 (s, 1H), 7.58-7.64 (m, 2H), 7.54-7.58 (m, 1H), 7.49 (dd, *J* = 7.5, 2.0 Hz,2H), 7.22 (d,*J* = 2.5 Hz, 1H), 7.18 (dd, *J* = 9.0, 2.5 Hz, 1H),4.02 (s, 3H);

^{13}C-NMR (125 MHz, $CDCl_3$): δ (ppm) 164.7, 160.3, 150.1, 146.8, 137.5, 129.6, 129.2, 129.0, 128.7, 127.0, 125.2, 117.4, 115.7, 108.5, 55.72;

HR-MS (ESI)for $C_{15}H_{12}N_2O_2$ [M +H]253.0971,found 253.0975.



2i

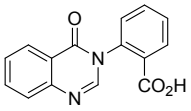
3-(2-hydroxyphenyl)quinazolin-4(*3H*)-one (**2i**)

^{1}H-NMR (500 MHz, $CDCl_3$): δ(ppm) 8.29 (d, *J* = 8.0 Hz, 1H), 8.07(s, 1H), 7.70-7.81 (m, 2H), 7.50 (t, *J* = 7.5 Hz,

1H), 7.19-7.26 (m, 2H), 7.03 (d, *J* = 8.0 Hz, 1H), 6.98 (d, *J* = 7.5 Hz, 1H),

^{13}C-NMR (125 MHz, $CDCl_3$): δ(ppm) 161.5, 152.3, 147.4, 147.0, 134.9, 130.8, 127.9, 127.8, 127.2, 127.1, 125.4, 121.8, 120.9, 118.8;

HR-MS (ESI) for $C_{14}H_{10}N_2O_2$ [M + H]239.0815,found 239.0824.



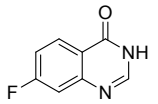
2j

2-[4-Oxoquinazolin-3(*4H*)-yl]benzoic Acid (**2j**)

All the characterization data are consistent with the previous report.[6]

^{1}H-NMR (500 MHz, DMSO-d_6): δ (ppm) 7.38 (dd, *J* = 8.0, 1.0 Hz, 1 H), 7.45-7.48 (m, 1 H), 7.56 (dt, *J* = 7.5,1.5 Hz, 1H), 7.66-7.69 (m, 2H), 7.73-7.76 (m, 1H), 8.01 (s, 1H), 8.09 (dd, *J* = 8.0, 1.5 Hz, 1 H), 8.17 (dd, *J* = 8.0, 1.5 Hz, 1H);

HR-MS (ESI) for $C_{15}H_{11}N_2O_3$[M + H] 267.0772,found 267.0775.



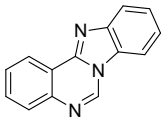
2k

7-fluoroquinazolin-4(*3H*)-one (**2k**)

All the characterization data are consistent with the previous report.[7]

^{1}H NMR (500 MHz, methanol-d_4) δ (ppm) 8.30 (dd, *J* = 8.5, 6.0 Hz, 1H), 8.13(s, 1H), 7.31-7.42 (m, 2H)

HR-MS (ESI) calcd for$C_8H_6FN_2O$[M + H]$^+$ 165.0459, found 165.0459.



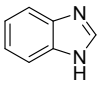
2l

Benzimidazo[l ,2-c]quinazoline (**2l**)

All the characterization data are consistent with the previous report.[8]

^{1}H-NMR(500 MHz, DMSO-d_6): δ (ppm) 9.05 (s, 1H), 8.65 (dd, *J*= 6.5, 1.5 Hz,1H), 8.10-7.10 (m, 7H);

HR-MS (ESI) calcd for $C_{14}H_{10}N_3$[M + H]$^+$220.0875, found 220.0877.



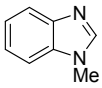
4a

1H-benzo[d]imidazole (**4a**)

All the characterization data are consistent with the previous report.[9]

^{1}H-NMR (DMSO-d_6, 500MHz) δ (ppm) 7.18 (dd, *J* = 6.0, 3.0 Hz, 2H), 7.59 (dd, *J* = 6.0, 3.0 Hz, 2H), 8.22 (s, 1H);

ESI-MS: *m/z* = 119.3 [M + H]$^+$.



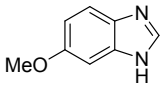
4b

1-Methyl-1H-benzo[d]imidazole (**4b**)

All the characterization data are consistent with the previous report.[10]

^{1}H-NMR (DMSO-d_6, 500MHz) δ (ppm) 8.26 (s, 1H),7.73 (d, J = 8.0 Hz, 1H), 7.62 (d, J = 8.0 Hz, 1H), 7.34 (t, J = 7.5 Hz, 1H), 7.31-7.23 (m, 1H), 3.89 (s, 3H);

HR-MS (ESI) calcd for $C_8H_9N_2$[M + H]$^+$133.0766, found 133.0764.



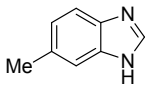
4c

6-methoxy-1H-benzo[d]imidazole (**4c**)

All the characterization data are consistent with the previous report.[11]

^{1}H-NMR (500MHz,$CDCl_3$) δ (ppm) 9.70 (brs, 1H), 7.64 (d, J = 9.0 Hz, 1H), 8.16 (s, 1H), 7.18 (d, J = 2.5 Hz, 1H), 7.02 (dt, J = 9.0, 2.0 Hz, 1H), 3.89 (s, 3H);

HR-MS (ESI) calcd for $C_8H_9ON_2$ [M + H]$^+$149.0715, found 149.0719.



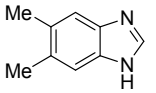
4d

6-methyl-1H-benzo[d]imidazole (**4d**)

All the characterization data are consistent with the previous report.[11]

^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 11.50 (s, 1H), 8.15 (s, 1H), 7.60 (d, J = 7.0 Hz, 1 H), 7.48 (s, 1H); 7.14 (d, J = 7.0 Hz, 1H), 2.49 (s, 3H);

HR-MS (ESI) calcd for $C_8H_9N_2$ [M + H]$^+$ 133.0760, found 133.0760.



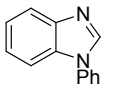
4e

5,6-dimethyl-1H-benzo[d]imidazole (**4e**)

All the characterization data are consistent with the previous report.[12]

^{1}H NMR (500 MHz, methanol-d_4): δ (ppm) 7.87 (s, 1H), 7.21 (s, 2H), 2.20 (s, 6H);

HR-MS (ESI) calcd for $C_9H_{11}N_2$[M + H]$^+$147.0917, found 147.0917.



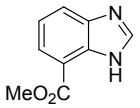
4f

1-phenyl-1H-benzo[d]imidazole (**4f**)

All the characterization data are consistent with the previous report.[13]

^{1}H-NMR(500 MHz,$CDCl_3$): δ (ppm) 8.13 (s, 1H), 7.88−7.90 (m, 1H),7.46−7.60 (m, 6H),7.33−7.36 (m, 2H);

HR-MS (ESI) calcd for $C_{13}H_{11}N_2$ [M + H]$^+$195.0922, found 195.0924.



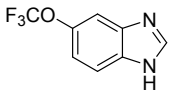
4g

Methyl-1H-benzo[d]imidazole-7-carboxylate (**4g**)

All the characterization data are consistent with the previous report.[14]

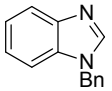
^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 8.26 (brs, 1H), 8.23 (s, 1H), 8.05 (d, J = 8.0 Hz, 1H), 7.96 (d, J = 7.5 Hz, 1H),

7.35 (t, *J* = 6.5 Hz, 1H), 4.00 (s, 3H);
HR-MS (ESI) calcd for $C_9H_9N_2O_2$ $[M + H]^+$177.0658, found 177.0660.



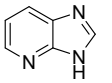
4h

5-(trifluoromethoxy)-1H-benzo[d]imidazole (**4h**)
All the characterization data are consistent with the previous report.[12]
^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 8.27 (s, 1H), 7.66 (d, *J* = 9.0 Hz, 1H), 7.56 (s, 1H), 7.20 (dd, *J* = 9.0, 2.0 Hz, 1H);
HR-MS (ESI) calcd for $C_8H_5F_3N_2O$203.0427, found 203.0427.



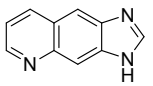
4i

1-Benzyl-1H-benzo[d]imidazole (**4i**)
All the characterization data are consistent with the previous report.[15]
^{1}H-NMR (500 MHz, $CDCl_3$): δ (ppm) 7.95 (s, 1H), 7.83 (d, *J*= 7.5 Hz, 1H), 7.36-7.24 (m, 6H), 7.19 (d, *J*=7.0 Hz, 2H), 5.37 (s, 2H);
HR-MS (ESI) calcd for $C_{14}H_{13}N_2$ $[M + H]^+$209.1077, found 209.1079.



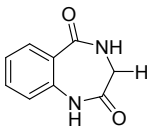
4j

3H-imidazo[4,5-b]pyridine (**4j**)
All the characterization data are consistent with the previous report.[16]
^{1}H-NMR (400MHz,$CDCl_3$) δ (ppm) 8.44 (dd, *J* = 4.8, 1.4 Hz, 1H), 8.32 (s, 1H), 8.11 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.21-7.30 (m, 1H);
HR-MS (ESI) calcd for $C_6H_6N_3[M + H]^+$ 120.0556, found 120.0557.



4k

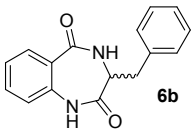
3H-imidazo[4,5-g]quinoline (**4k**)
All the characterization data are consistent with the previous report.[14]
^{1}H-NMR (400 MHz, DMSO-d_6): δ (ppm) 8.86–8.85 (dd, *J* = 4.0, 1.6 Hz, 1H), 8.79 (br s, 1H), 8.38 (brs, 1 H), 7.97 (brd, *J* = 8.8 Hz, 1H), 7.81 (d, *J* = 8.8 Hz, 1H), 7.61 (dd, *J* = 8.0, 4.0 Hz, 1H);
HR-MS (ESI) calcd for $C_{10}H_8N_3$ $[M + H]^+$ 170.0713, found 170.0713.



6a

3,4-Dihydro-1H-benzo[e][1,4]diazepine-2,5-dione (**6a**)
All the characterization data are consistent with the previous report.[17]

^{1}H NMR (500 MHz, DMSO-d_6) δ(ppm) 3.59 (d, J = 5.5 Hz, 2H), 7.11 (d, J = 8.0 Hz, 1H), 7.22 (t, J = 8.0 Hz, 1H), 7.51 (dt, J = 8.0, 1.5Hz, 1H), 7.76 (t, J = 8.0 Hz, 1H), 8.53 (t, J = 5.5 Hz, 1H), 10.34 (s, 1H);
ESI-MS: m/z = 177.1 $[M + H]^+$.



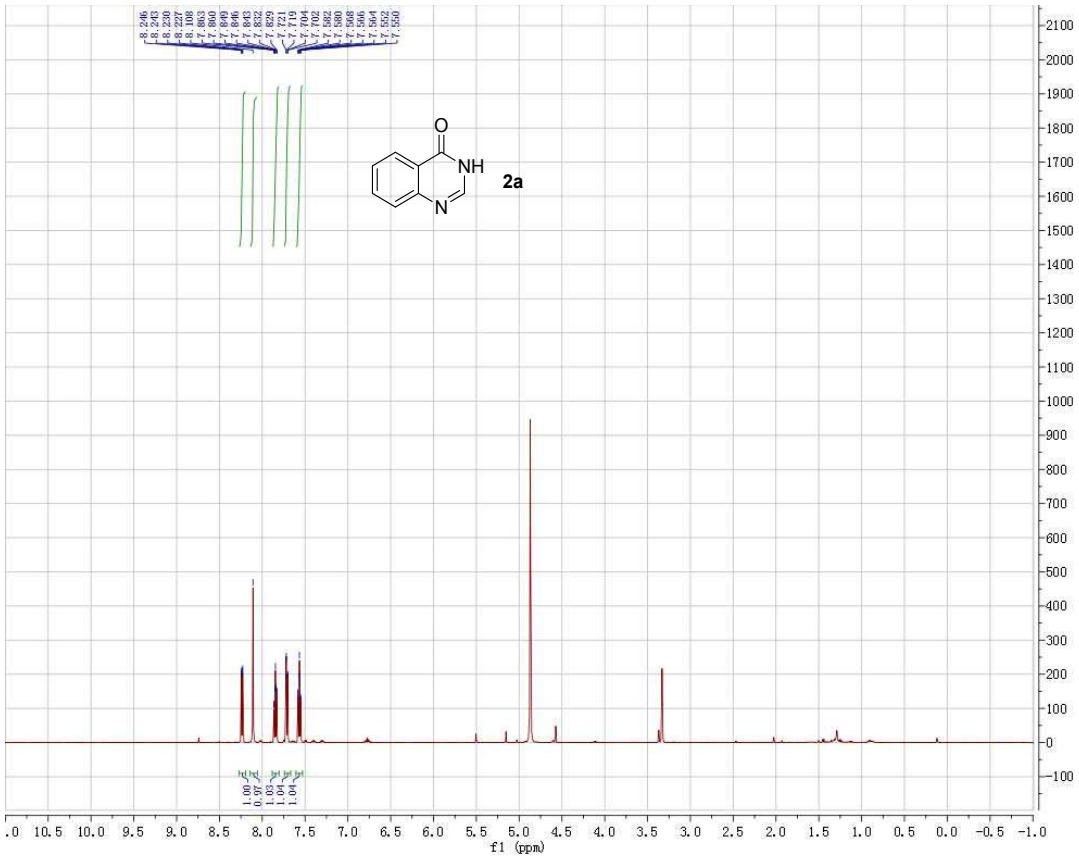
6b

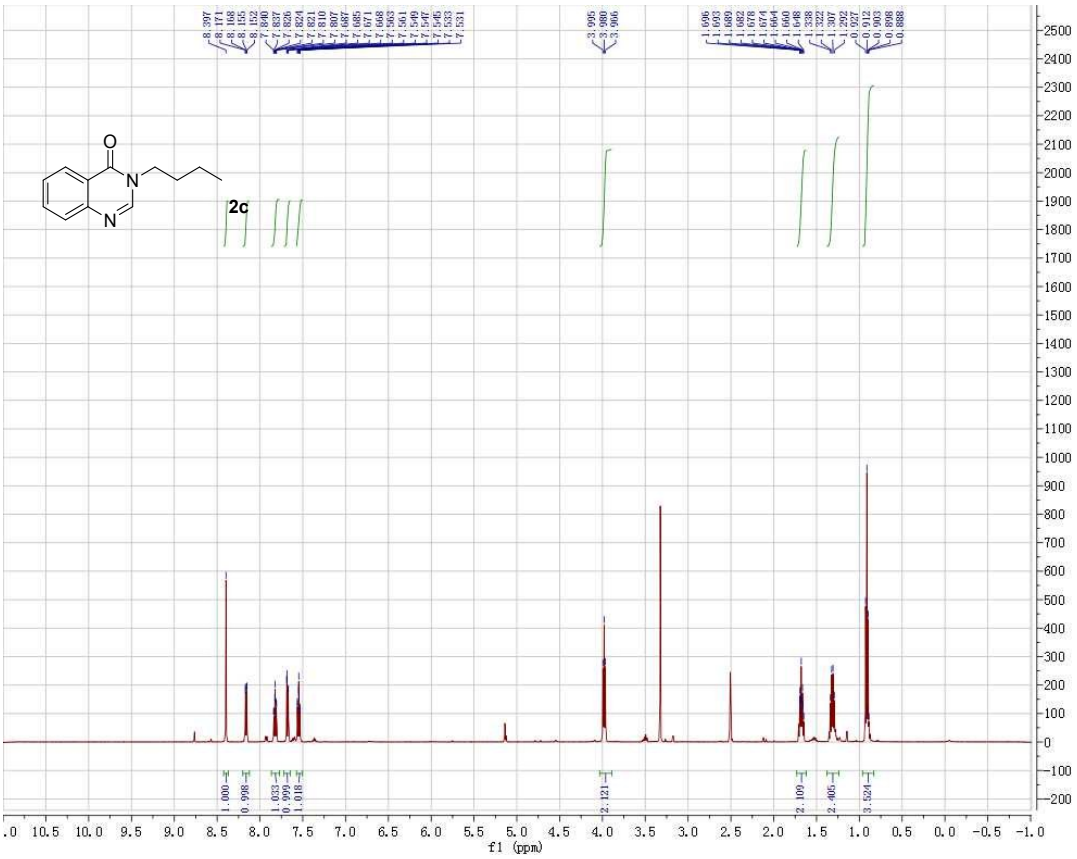
3-benzyl-3,4-dihydro-1H-benzo[e][1,4]diazepine-2,5-dione(**6b**)
All the characterization data are consistent with the previous report.[18]
^{1}H-NMR (500 MHz,DMSO-d_6): δ (ppm) 2.83 (dd,J = 14.0, 5.0 Hz, 1H), 3.11 (dd, J = 14. 0, 9.5 Hz, 1H), 3.87 (m, 1H), 7.08-7.65 (m, 9H), 8.50 (s, 1H), 10.40 (s, 1H);
ESI-MS: m/z = 267.1 $[M + H]^+$.

4. References:

1. (a) F.Zhang, X.Zhai, L.Chen, J.Qi, B.Cui, Y.Gu and P.Gong, *Chinese Chem. Lett.*, **2010**, *22*, 1277.
(b)H.Kakuta, X. Zheng, H.Oda,S.Harada, Y. Sugimoto, K. Sasaki and A.Tai,*J. Med. Chem.*,**2008**, *51*, 2400.
2. S. K.Kundu, M. P. D. Mahindaratne, M. V. Quintero, A. Bao and G. R. Negrete, *ARKIVOC***2008**, *2*, 33.
3. A. V. Lygin and A. de Meijere,*Org. Lett.*, **2009**, *11*, 389.
4. S. Wang, K. Yang, C. Yao and X. Wang,*Synthetic Commun.*, **2012**, *42*, 341.
5.D.Ravier, J. P. Dupin, F. Casadebaig, G. Hou, M. Boisseau and H. Bernard, *Pharmazie*, **1992**, *47*, 91.
6. G.Aridossand K. K. Laali, *Eur. J. Org. Chem.*,**2011**,*15*, 2827.
7.J. Wu, W. Chen, G. Xia, J. Zhang, J. Shao, B. Tan, C. Zhang, W. Yu, Q.Weng, H. Liu, M. Hu, H. Deng, Y.Hao, J. Shen and Y. Yu,*ACS Med. Chem. Lett.*, **2013**, *4*, 974.
8. M. S. Khajavi, K. Rad-moghadam and H.Hazarkhani,*Synthetic Commun.*, **1999**, *29*, 2617.
9. C. Fortenberry, B. Nammalwar and R. A. Bunce, *Org. Prep.Proced.Int.*, **2013**, *45*, 57.
10. T. H. Graham, W. Liu and D Shen, *Org. Lett.*, **2011**, *13*, 6232.
11. H. Alinezhadab, F.Salehianaand P Biparva, *Synthetic Commun.*,**2012**, *42*, 102.
12.B. Yu , H. Zhang , Y. Zhao , S. Chen , J. Xu , C. Huang and Z. Liu, *Green Chemistry*, **2013**, 15, 95.
13. P. Suresh and K.Pitchumani, *J. Org. Chem.*, **2008**, *73*, 9121.
14. E. J. Hanan, B. K. Chan, A. A. Estrada, D. G. Shore and J. P. Lyssikatos, *Syn. Lett*.,**2010**, *18*, 2759.
15. M. J. Ohlmeyer, A. C. Bohnstedt and H.Park, *U.S. Pat. Appl. Publ*. 20080287468.
16. H. M. Möller, M. C. Baier, S.Mecking, E. P. Talsi and K. P. Bryliakov, *Chem. Eur. J.*,**2012**, *18*, 857.
17. J. L. Vrijdag , A. V.Bogaert and W. M. De Borggraeve, *Org. Lett.,***2013**, *15*, 1052.
18. M. Akssira, M. Boumzebra, H. Kasmi, A. Dahdouh, M. L. Roumestant and P. Viallefont, *Tetrahedron*, **1994**, *50*, 9051.

5. Selected spectra

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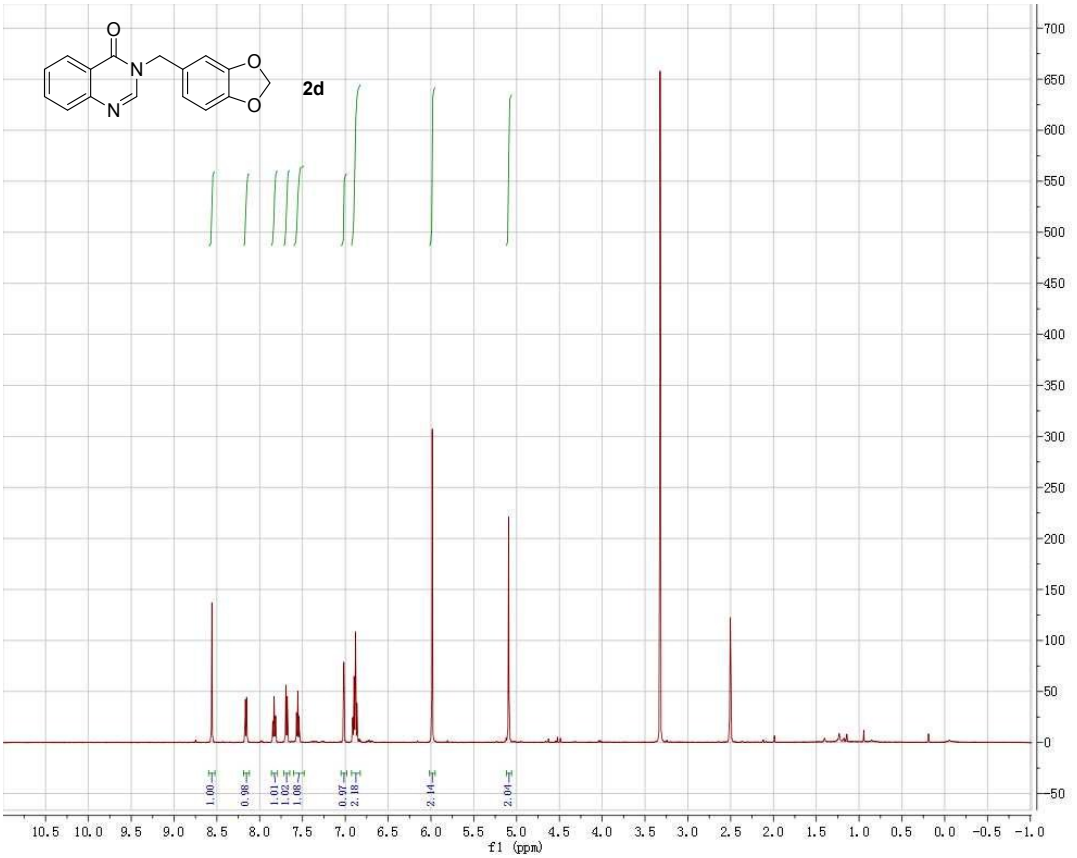
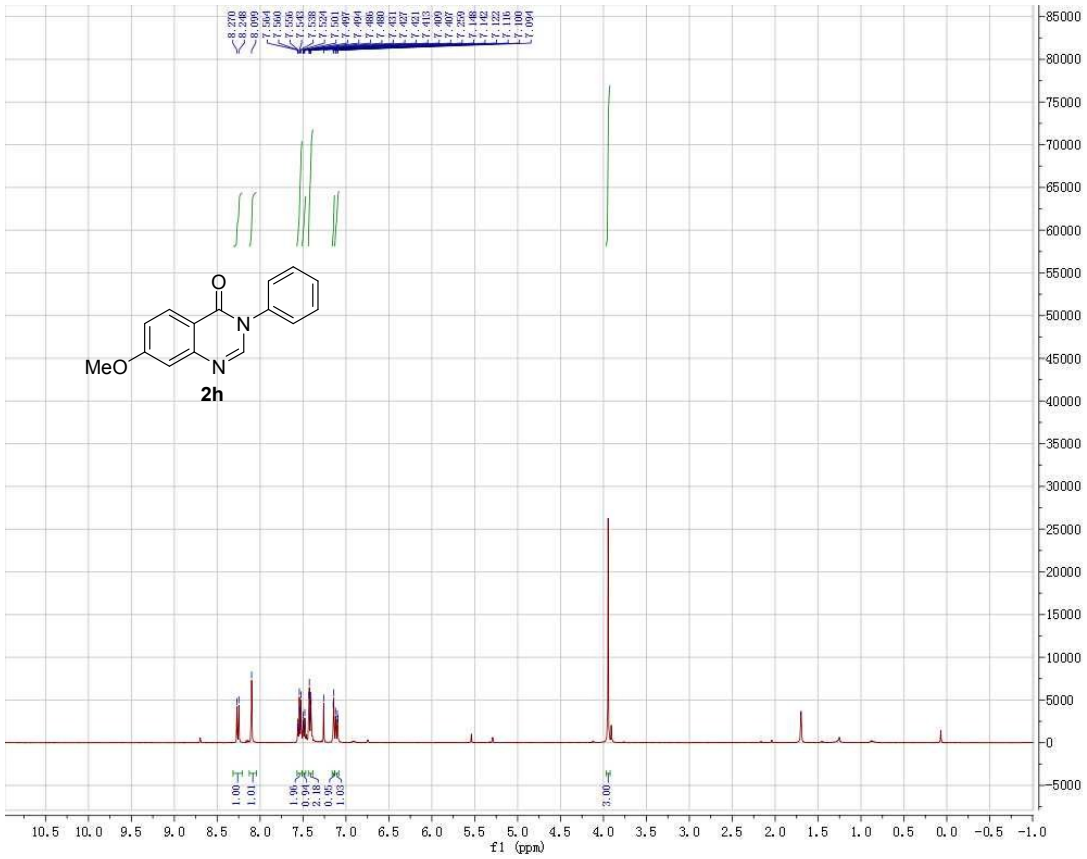

2d
f1 (ppm)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2h
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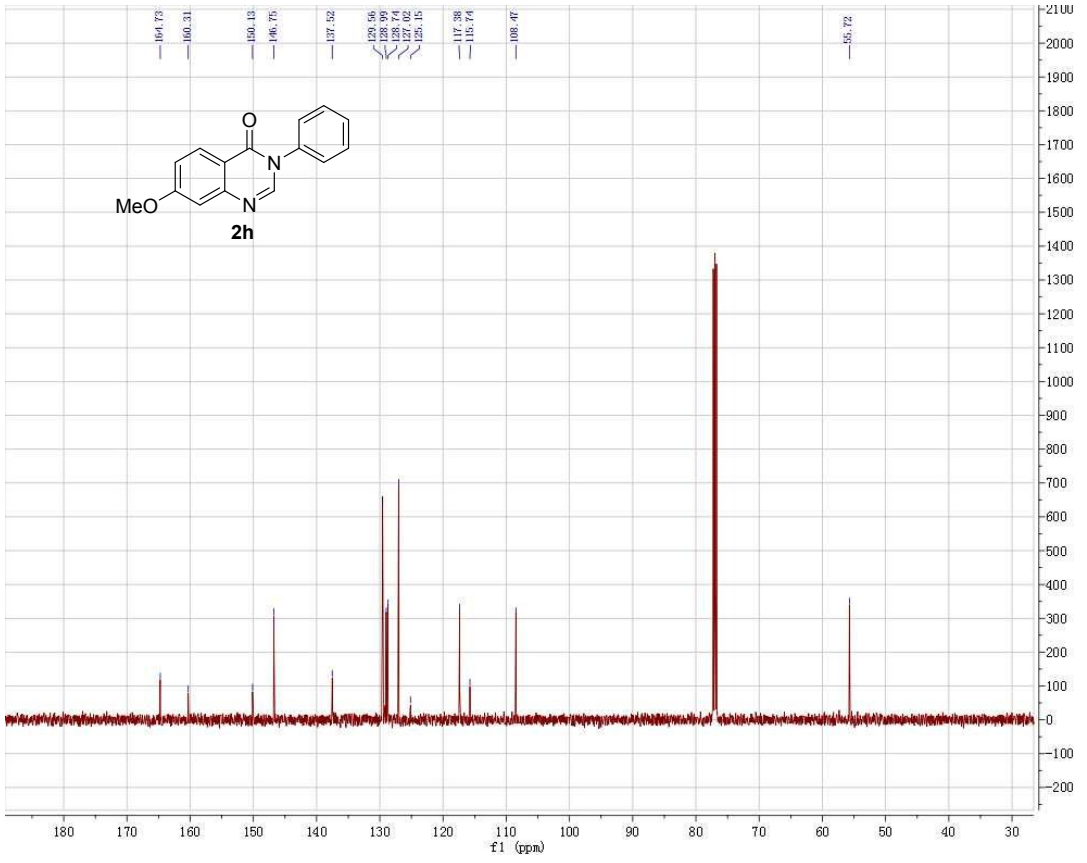
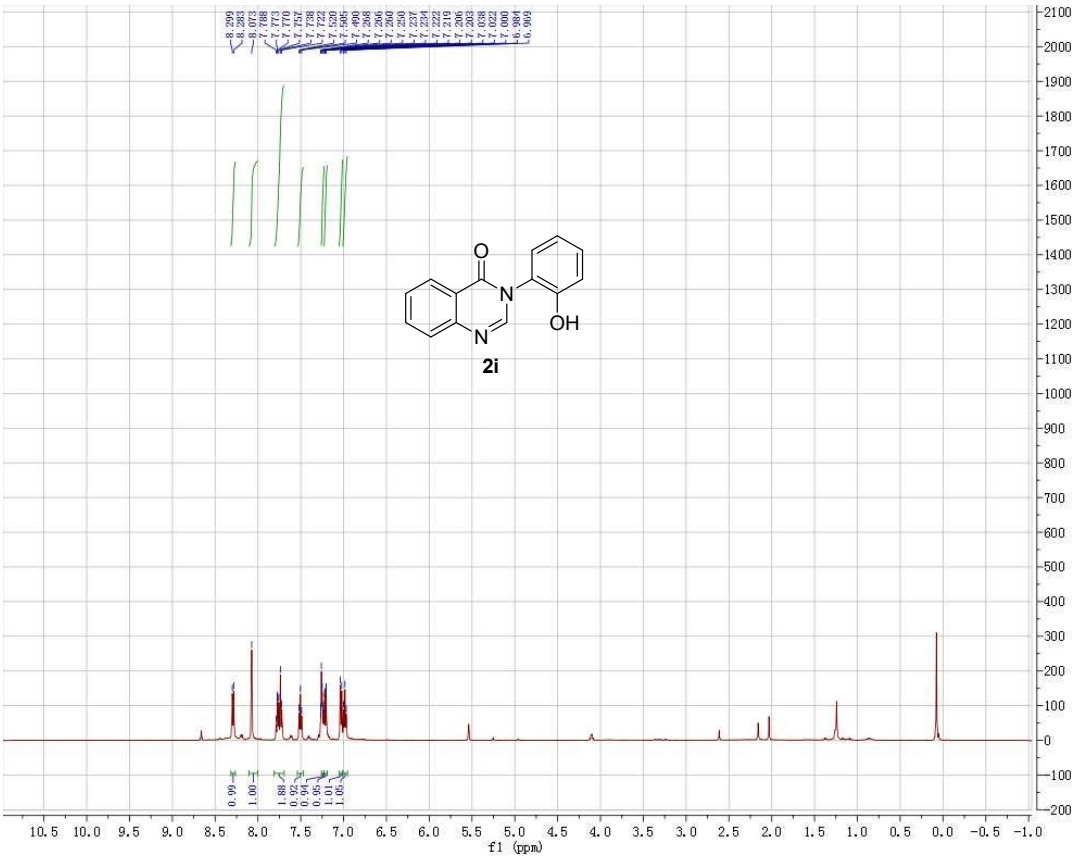

MeO
2h
f1 (ppm)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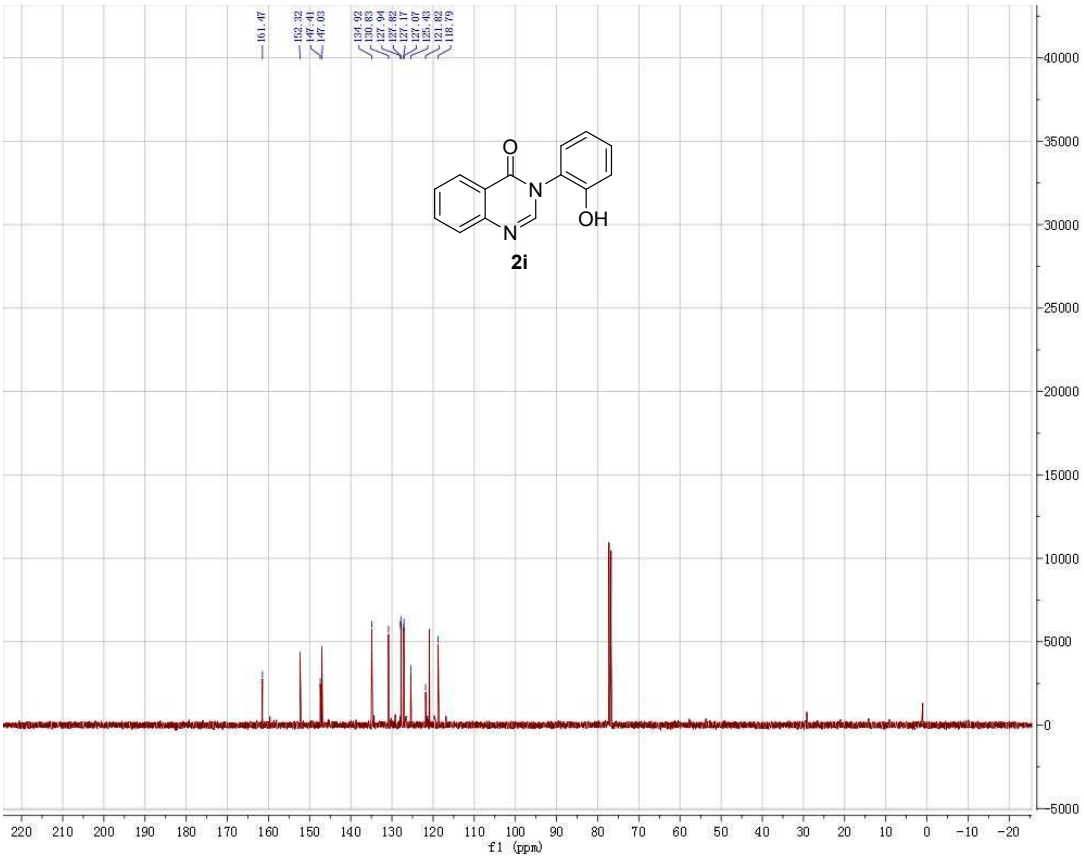
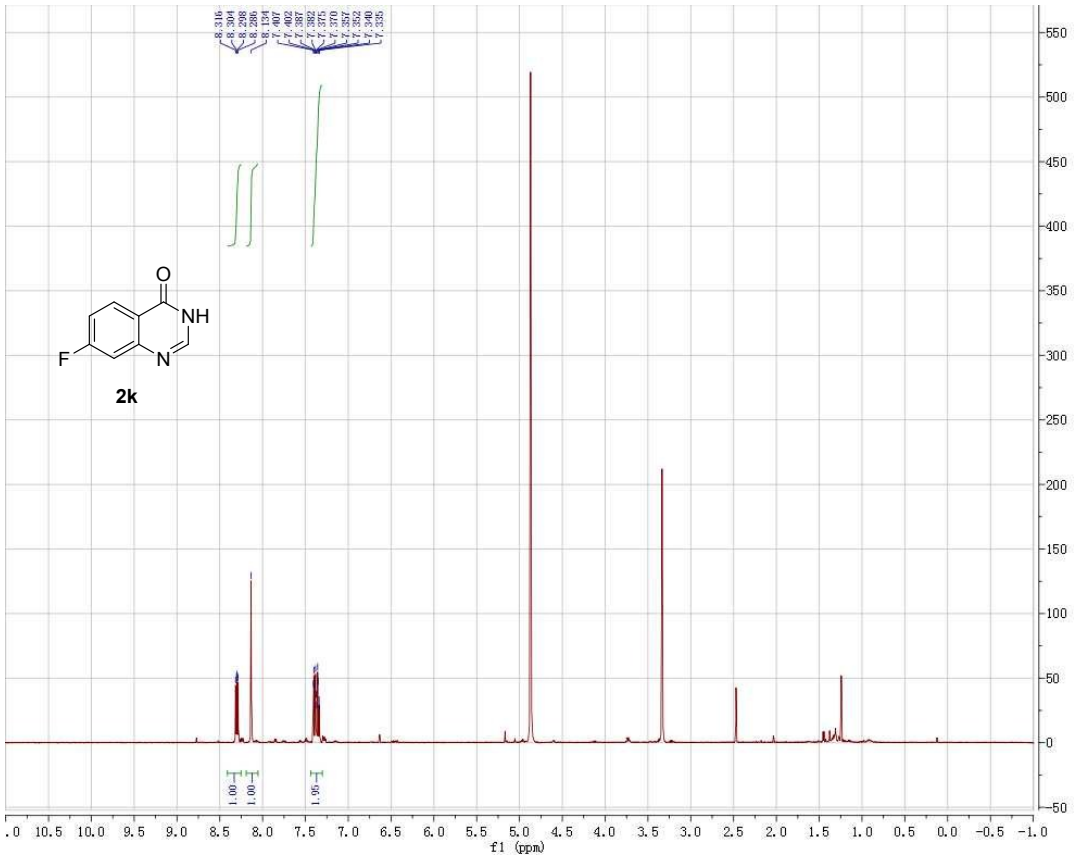
8.283
8.073
7.788
7.771
7.770
7.757
7.738
7.722
7.520
7.505
7.490
7.368
7.366
7.360
7.359
7.237
7.234
7.222
7.219
7.208
7.205
7.038
7.022
7.000
6.984
6.969
O
N
N
OH
0.99
1.00
1.88
0.92
0.94
0.95
1.01
1.05
f1 (ppm)

2i


161.47
152.32
147.41
147.03
134.92
130.83
127.94
127.62
127.17
127.07
125.45
121.82
118.79
O
N
N
OH
f1 (ppm)

2i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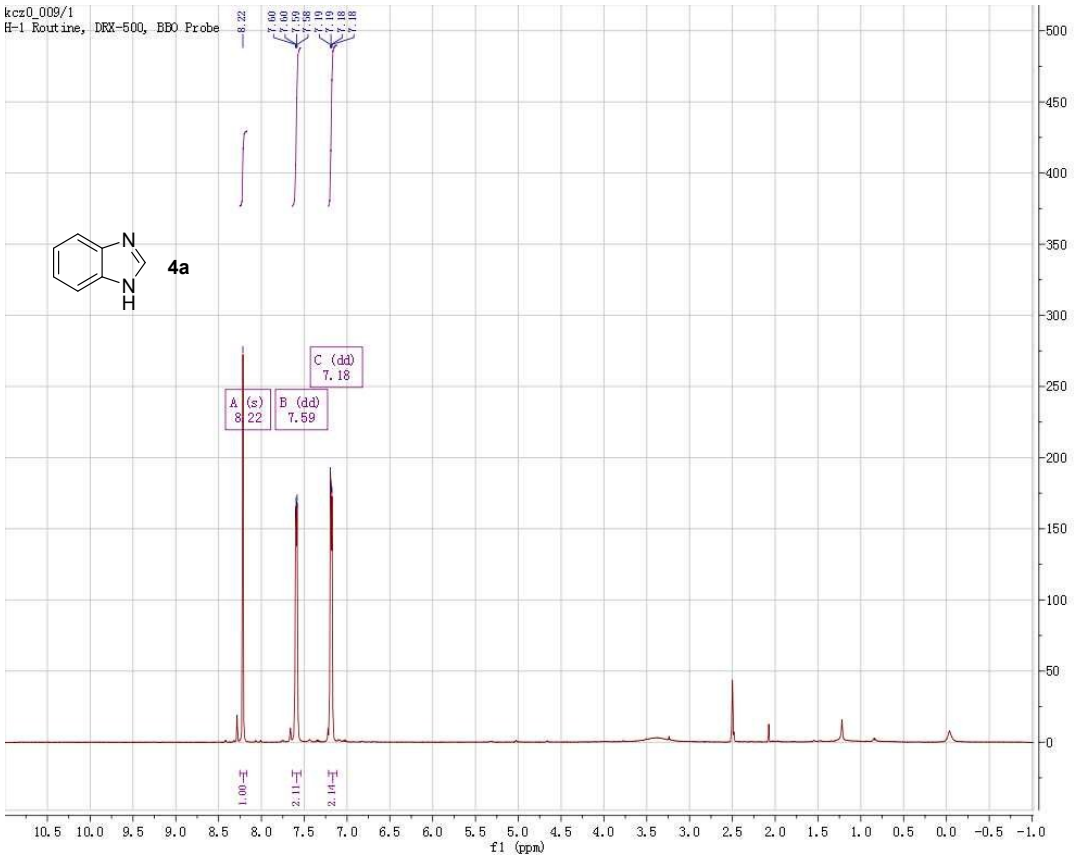
NH
F
N
2k
f1 (ppm)


kcz0_009/1
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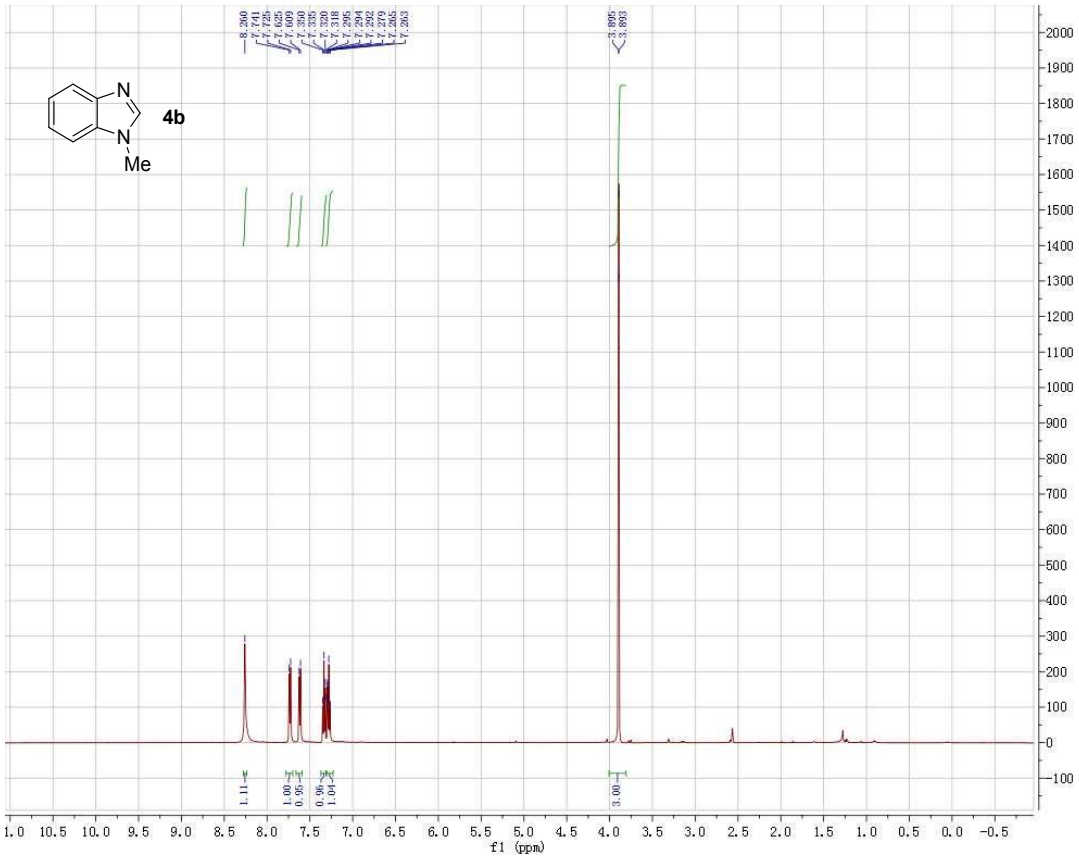
N
H
4a
C (dd)
7.18
A (s)
8.22
B (dd)
7.59
f1 (ppm)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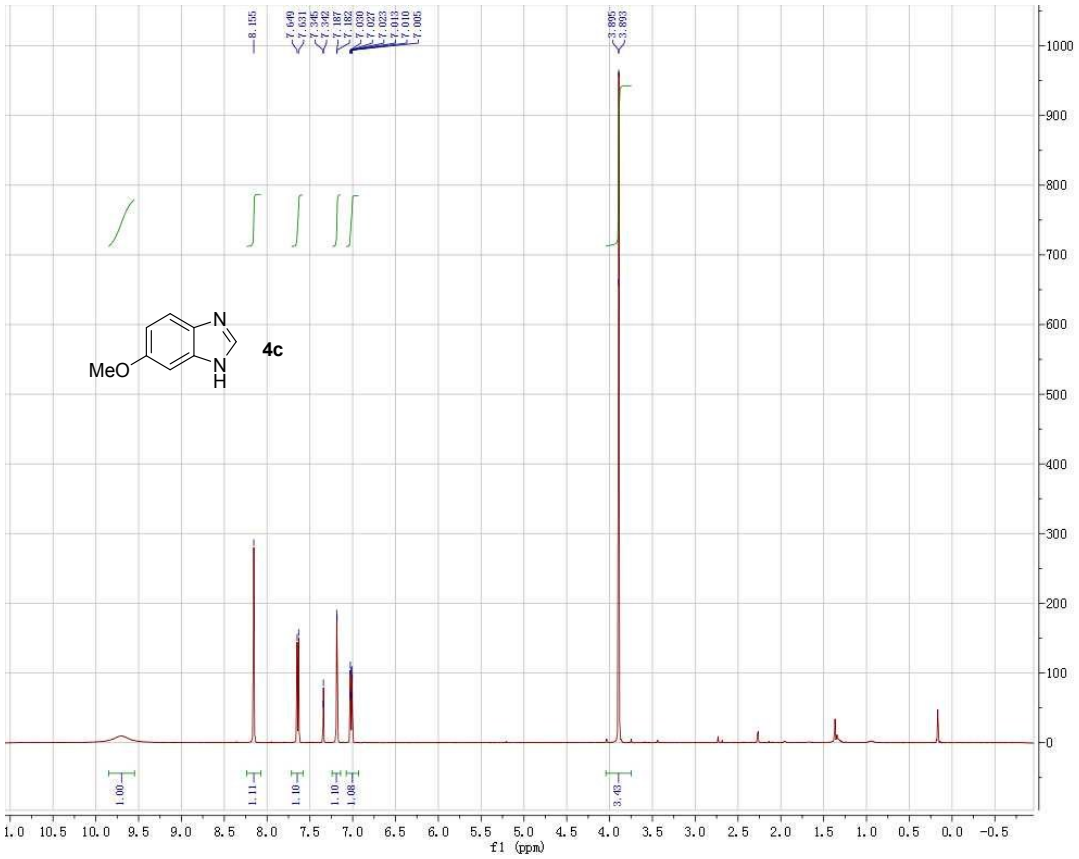
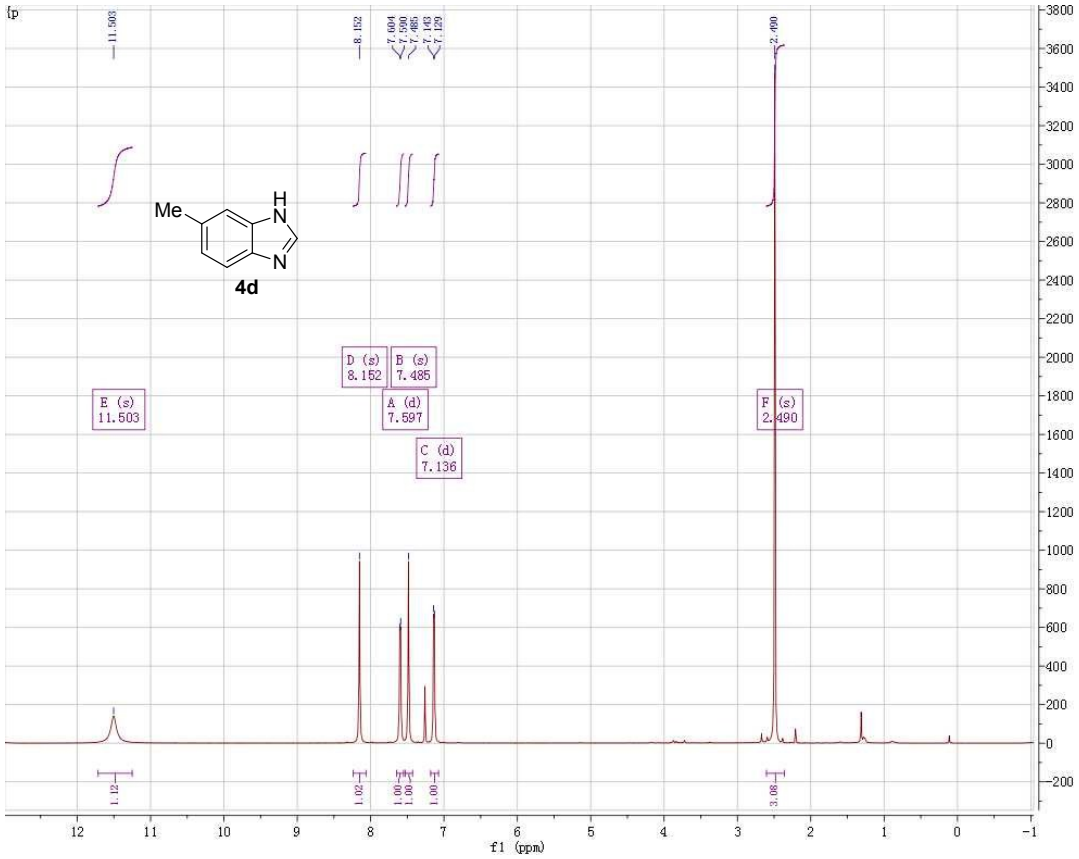
N
N
Me
f1 (ppm)


4c
MeO
N
N
H
f1 (ppm)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7.604
7.597
7.485
7.143
7.129
2.490
Me
H
N
N
4d
D (s)
8.152
B (s)
7.485
E (s)
11.503
A (d)
7.597
C (d)
7.136
F (s)
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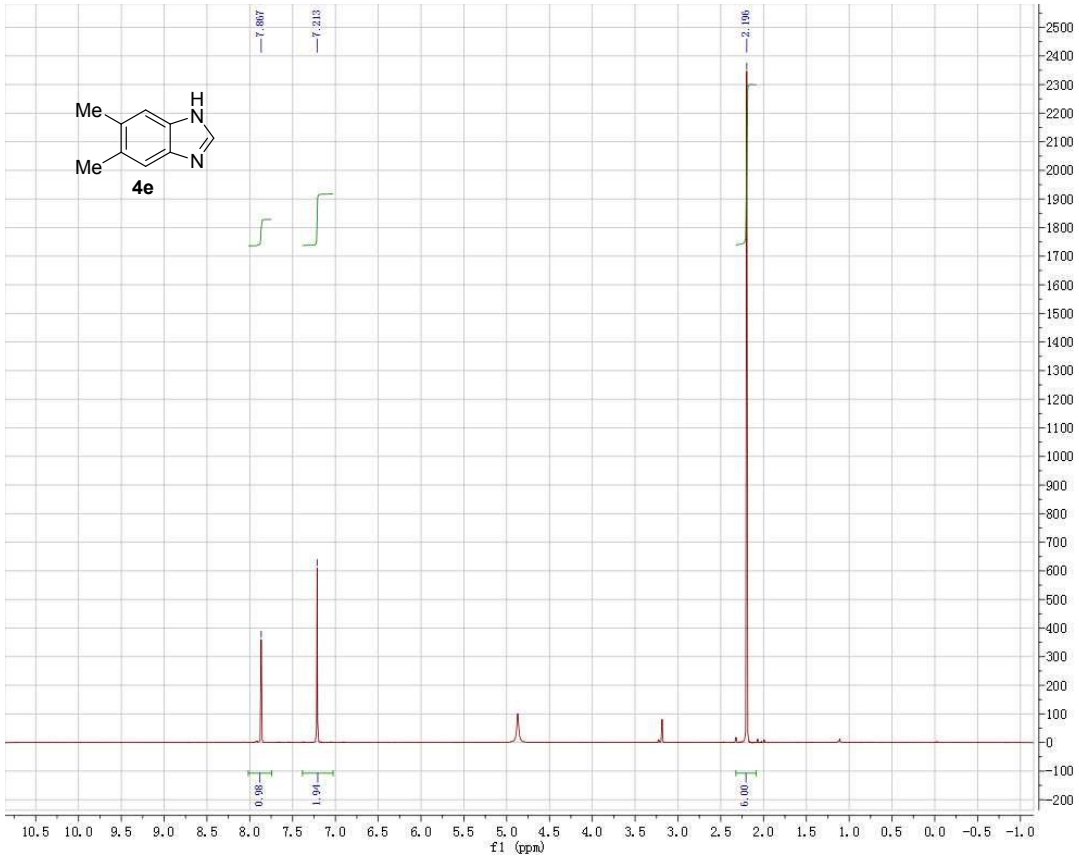
1.12
1.02
1.00
1.00
1.00
3.08
f1 (ppm)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7.213
2.196
Me
Me
H
N
N
4e
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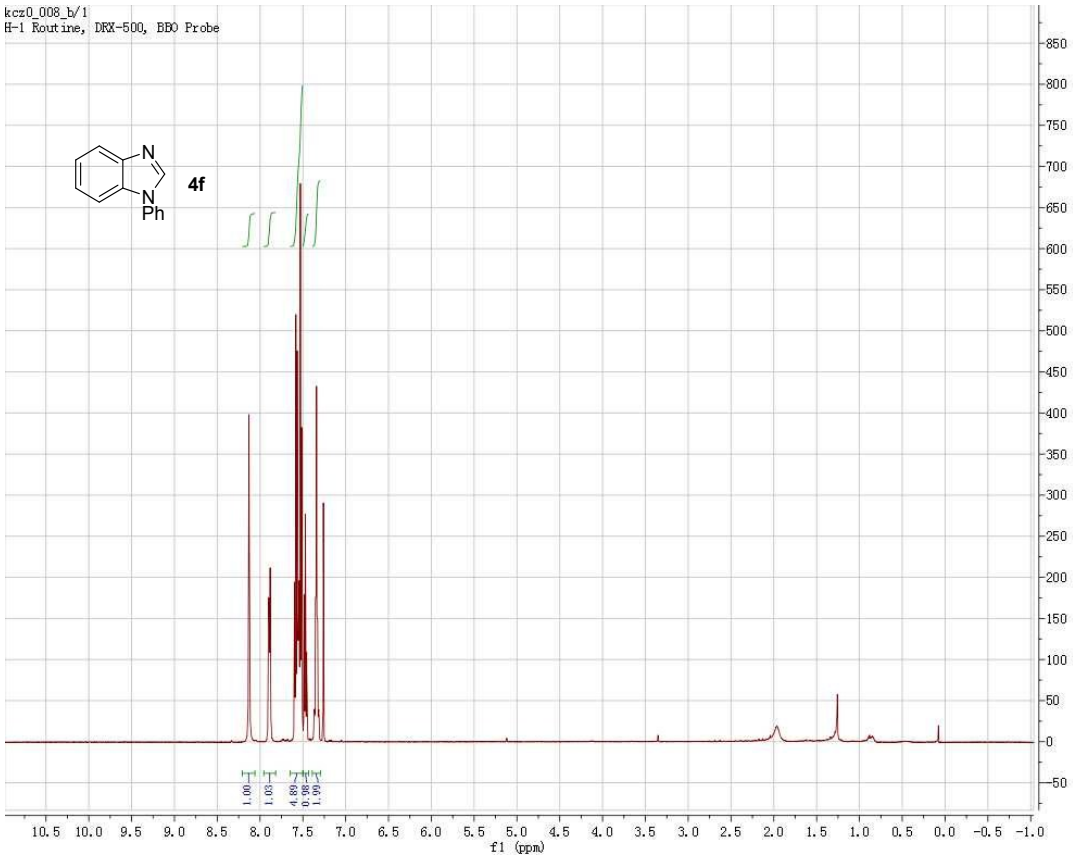
6.00
f1 (ppm)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N
Ph
4f
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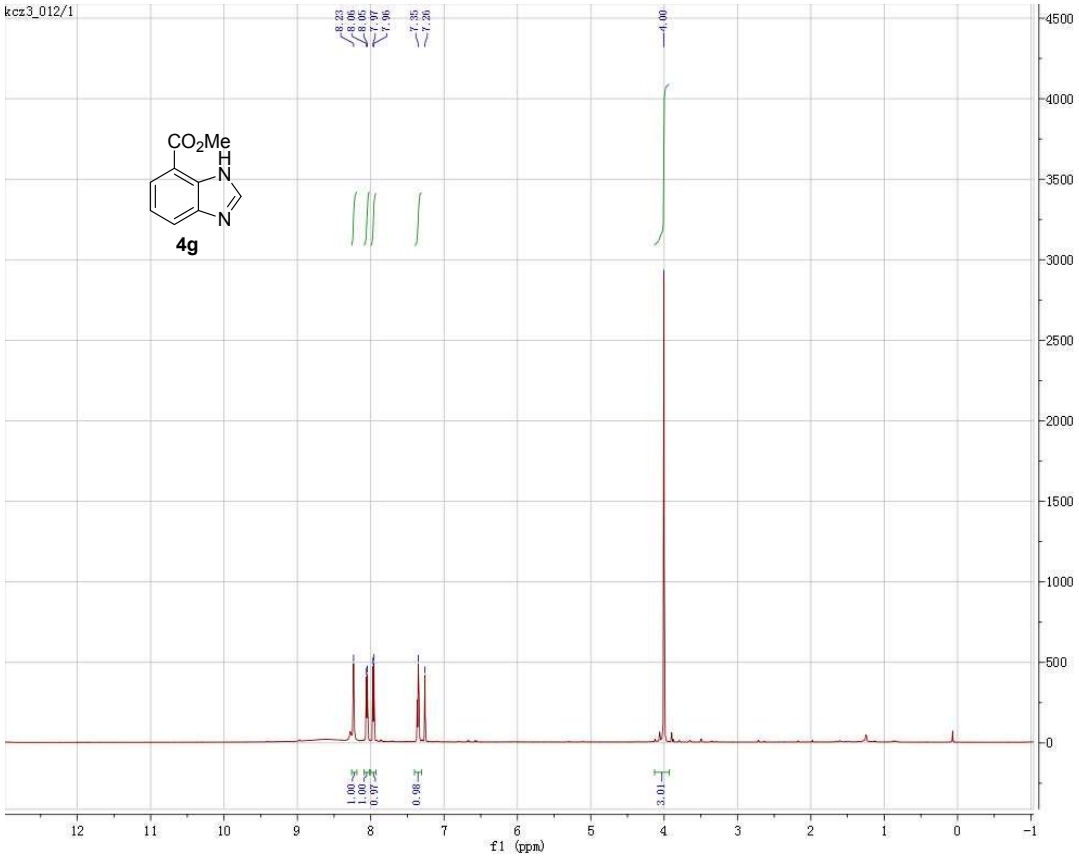
1.99
f1 (ppm)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8.05
7.97
7.96
7.35
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CO2Me
H
N
N
4g
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1.00
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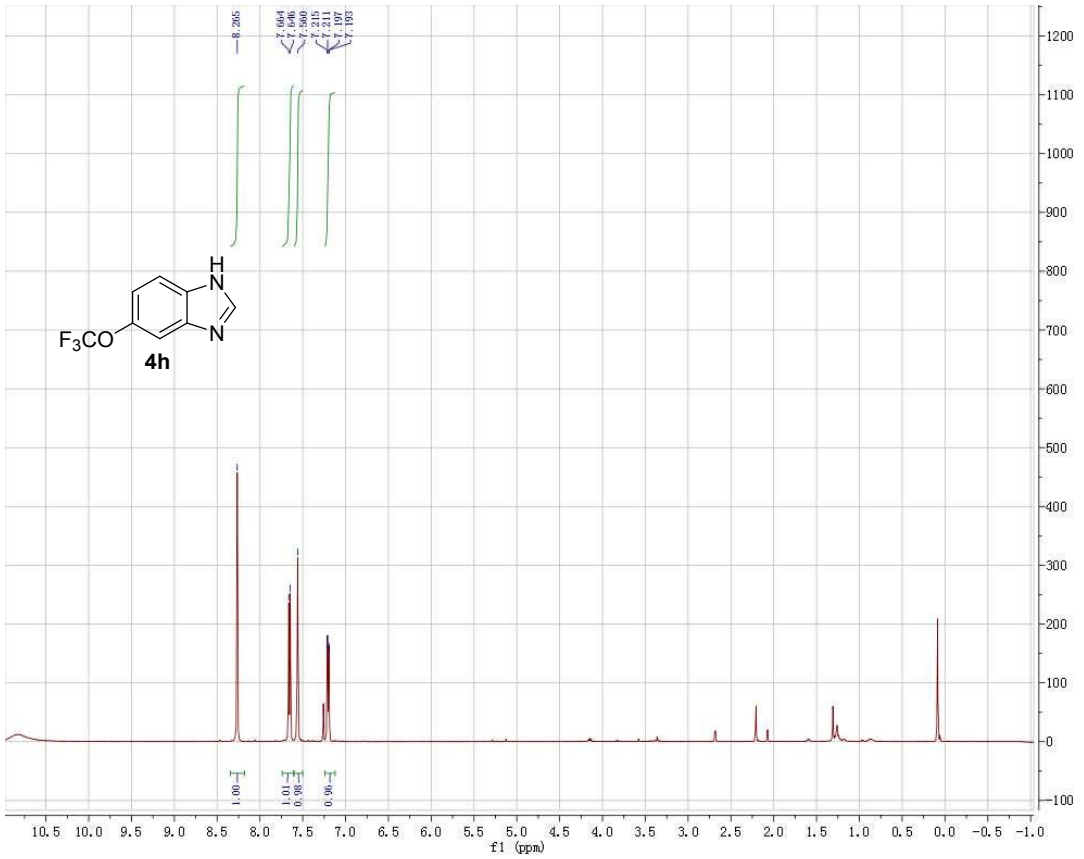
3.01
f1 (ppm)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N
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1.01
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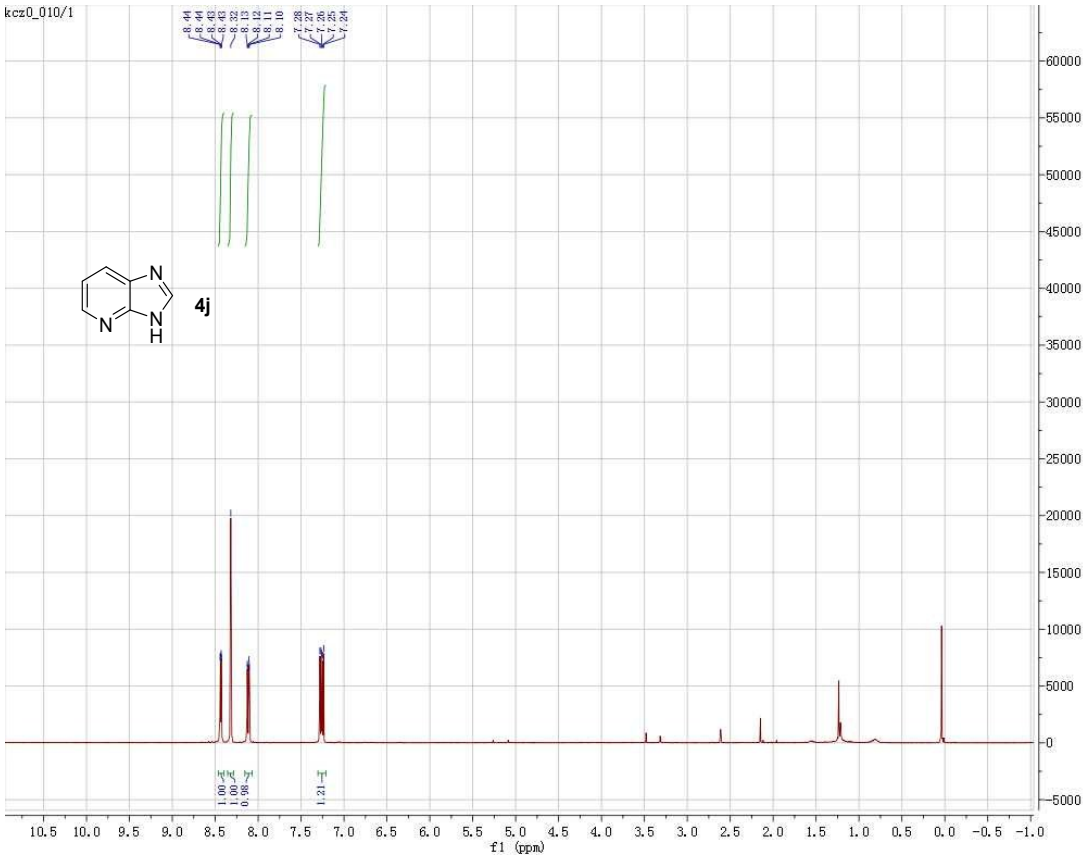
f1 (ppm)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8.43
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8.13
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8.11
8.10
7.28
7.27
7.26
7.25
7.24
N
N
N
H
4j
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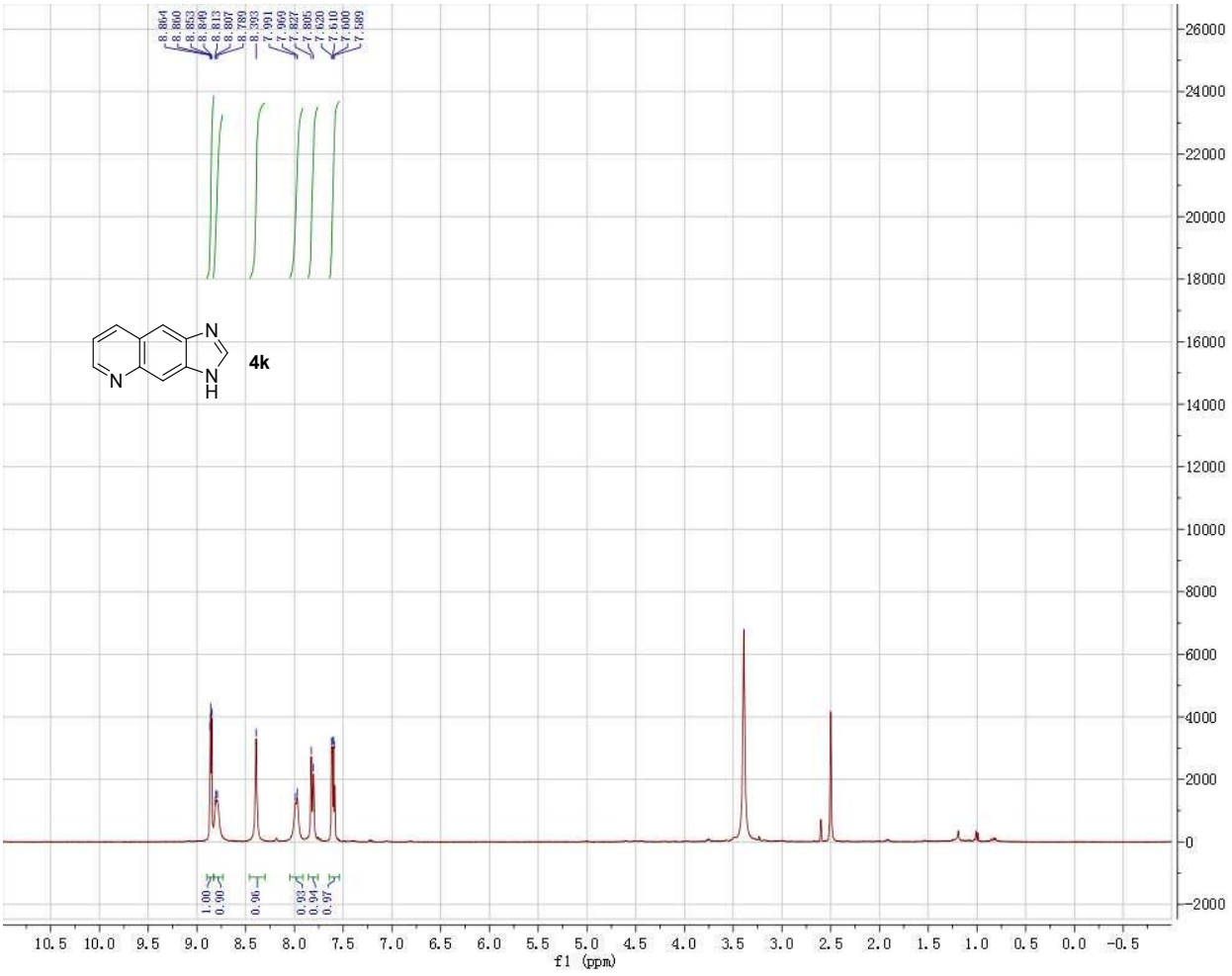
f1 (ppm)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7.969
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7.805
7.620
7.610
7.600
7.589
4k
1.00
0.90
0.90
0.93
0.94
0.97
f1 (ppm)