Electronic Supplementary Information

I_2-mediated aerobic oxidative annulation of amidines with tertiary amines via C-H amination/C-N cleavage for the synthesis of 2,4-disubstitued 1,3,5-triazines

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Contents

General Information

Unless otherwise indicated, all commercial reagents and solvents were used without additional purification. ^{1}H-NMR spectra were recorded with a Bruker Ascend^{TM} 600 spectrometer. Chemical shifts (in ppm) were referenced to tetramethylsilane (δ = 0 ppm) in $CDCl_3$ as an internal standard. ^{13}C-NMR spectra were obtained by the same NMR spectrometer and were calibrated with $CDCl_3$ (δ = 77.00 ppm). HRMS (ESI) were recorded on a Water^{TM} Q-TOF Premier Mass Spectrometer. MS (ESI) was recorded by AB SCIEX QTRAP 5500 LC/MS/MS. Melting point was recorded on a Hanon MP430 Auto Melting Point System.

Experimental Procedure

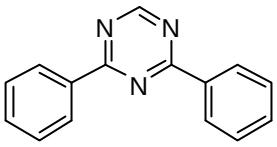
General Procedure for the Synthesis of Symmetrical 2,4-Disubstituted 1,3,5-Triazines

Amidines **1** (0.4 mmol), I_2 (0.4 mmol), Cs_2CO_3 (0.8 mmol) were added to a 10 mL Schlenk tube, followed by addition of DMSO (1.0 mL) and amines **2** (0.4 mmol). The mixture was stirred at 140 °C for 24 h. The solution was then cooled to r.t., quenched by a $Na_2S_2O_3$ aqueous solution and extracted with EtOAc (3×10 mL). The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 60:1) to afford the symmetrical 2,4-disubstituted 1,3,5-triazines **3**.

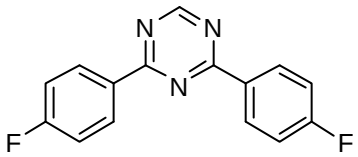
General Procedure for the Synthesis of Unsymmetrical 2,4-Disubstituted 1,3,5-Triazines

Amidines **1** (0.2 mmol), amidines **1'** (0.8 mmol), I_2 (0.4 mmol), Cs_2CO_3 (0.8 mmol) were added to a 10 mL Schlenk tube, followed by addition of DMSO (1.0 mL) and TMEDA (0.4 mmol). The mixture was stirred at 140 °C for 24 h. The solution was then cooled to r.t., quenched by a $Na_2S_2O_3$ aqueous solution and extracted with EtOAc (3×10 mL). The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 20:1) to afford the unsymmetrical 2,4-disubstituted 1,3,5-triazines **3**.

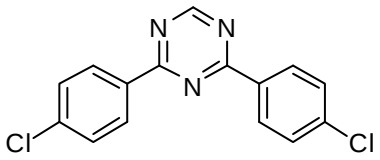
Characterization of Products



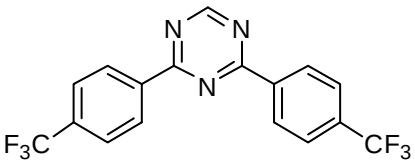
2,4-diphenyl-1,3,5-triazine (**3a**)[1]: 39.6 mg (85%); White solid; Mp: 74-75°C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.25 (s, 1H), 8.65-8.63 (m, 4H), 7.62-7.58 (m, 2H), 7.56-7.53 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 171.3, 166.7, 135.5, 132.8, 128.9, 128.7.



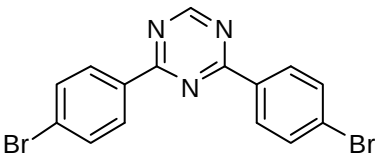
2,4-bis(4-fluorophenyl)-1,3,5-triazine (**3b**)[1]: 21.5 mg (40%); White solid; Mp: 155-156 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.20 (s, 1H), 8.67-8.62 (m, 4H), 7.25-7.20 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.3, 166.6, 166.0 (d, J = 252.6 Hz), 131.6 (d, J = 3.0 Hz), 131.3 (d, J = 9.4 Hz), 115.9 (d, J = 21.6 Hz).



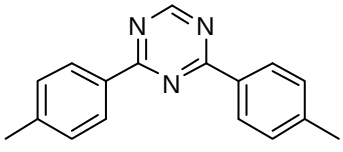
2,4-bis(4-chlorophenyl)-1,3,5-triazine (**3c**)[1]: 46.5 mg (77%); White solid; Mp: 191-192 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.23 (s, 1H), 8.58-8.55 (m, 4H), 7.53-7.50 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.5, 166.7, 139.4, 133.8, 130.2, 129.1.



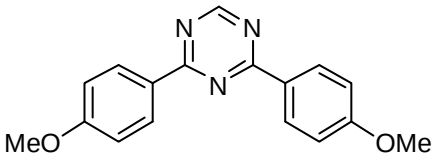
2,4-bis(4-(trifluoromethyl)phenyl)-1,3,5-triazine (**3d**)[1]: 35.4 mg (48%); White solid; Mp: 152-153 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.33 (s, 1H), 8.74 (d, J = 8.4 Hz, 4H), 7.81 (d, J = 7.8 Hz, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.4, 167.1, 138.5, 134.4 (q, J = 32.4 Hz), 129.2, 125.8 (q, J = 3.8 Hz), 123.8 (q, J = 271.0 Hz).



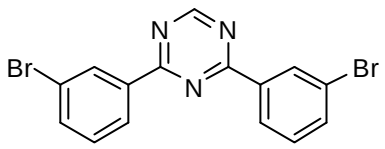
2,4-bis(4-bromophenyl)-1,3,5-triazine (**3e**)[1]: 41.4 mg (53%); White solid; Mp: 196-197 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.23 (s, 1H), 8.49 (d, J = 9.0 Hz, 4H), 7.68 (d, J = 9.0 Hz, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.7, 166.8, 134.3, 132.1, 130.4, 128.1.



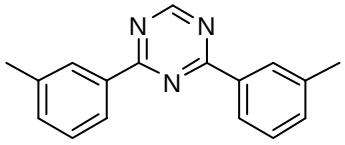
2,4-di-*p*-tolyl-1,3,5-triazine (**3f**)[1]: 42.3 mg (81%); White solid; Mp: 159-160 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.18 (s, 1H), 8.51 (d, J = 7.8 Hz, 4H), 7.33 (d, J = 8.4 Hz, 4H), 2.45 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 171.1, 166.5, 143.4, 132.9, 129.5, 128.8, 21.7.



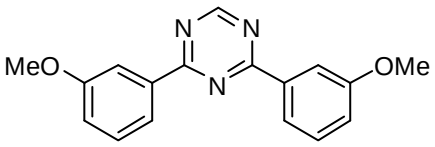
2,4-bis(4-methoxyphenyl)-1,3,5-triazine (**3g**)[1]: 39.2 mg (67%); White solid; Mp: 157-158 °C; δ 9.11 (s, 1H), 8.60-8.56 (m, 4H), 7.05-7.01 (m, 4H), 3.91 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.5, 166.2, 163.5, 130.8, 128.1, 114.0, 55.5.



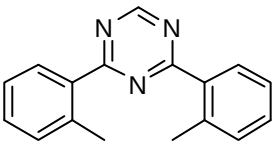
2,4-bis(3-bromophenyl)-1,3,5-triazine (**3h**)[1]: 43.0 mg (55%); White solid; Mp: 181-182 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.27 (s, 1H), 8.76 (t, J = 1.7 Hz, 2H), 8.57 (dt, J_1 = 7.8 Hz, J_2 = 1.1 Hz, 2H), 1.41 (dq, J_1 = 7.8 Hz, J_2 = 1.0 Hz, 2H), 1.31 (t, J = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.3, 166.9, 137.3, 135.9, 131.9, 130.3, 127.5, 123.1.



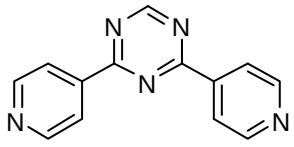
2,4-di-*m*-tolyl-1,3,5-triazine (**3i**)[1]: 41.8 mg (80%); White solid; Mp: 87-88 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.24 (s, 1H), 8.46-8.43 (m, 4H), 7.46-7.41 (m, 4H), 2.49 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 171.4, 166.5, 138.5, 135.5, 133.7, 129.4, 128.7, 126.1, 21.5.



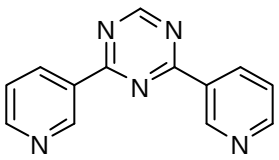
2,4-bis(3-methoxyphenyl)-1,3,5-triazine (**3j**)[1]: 34.0 mg (58%); White solid; Mp: 106-107 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.25 (s, 1H), 8.24 (t, J = 7.8 Hz, 2H), 8.18-8.16 (m, 2H), 7.46 (t, J = 7.8 Hz, 2H), 7.17-7.14 (m, 2H), 3. 94 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 171.1, 166.6, 160.0, 136.9, 129.8, 121.4, 119.2, 113.3, 55.4.



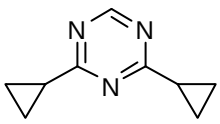
2,4-di-*o*-tolyl-1,3,5-triazine (**3k**)[1]: 27.1 mg (52%); Light yellow oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.32 (s, 1H), 8.14 (dd, J_1 = 7.7 Hz, J_2 = 1.1 Hz, 2H), 7.44-7.41 (m, 2H), 7.37-7.32 (m, 4H), 2.72 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 173.8, 165.7, 138.9, 135.5, 131.8, 131.2, 131.1, 126.1, 22.0.



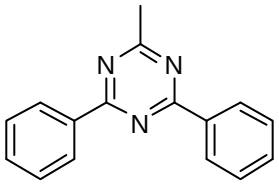
2,4-di(pyridin-4-yl)-1,3,5-triazine (**3m**)[1]: 14.6 mg (31%); White solid; Mp: 181-182 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.45 (s, 1H), 8.91 (d, J = 5.5 Hz, 4H), 8.47 (dd, J_1 = 4.6 Hz, J_2 = 1.4 Hz, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.5, 167.6, 150.8, 142.5, 122.2.



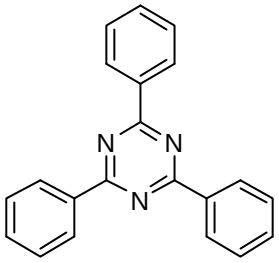
2,4-di(pyridin-3-yl)-1,3,5-triazine (**3n**)[1]: 21.2 mg (45%); White solid; Mp: 183-184 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.83 (q, J = 1.6 Hz, 2H), 9.34 (s, 1H), 8.89 (dt, J_1 = 7.9 Hz, J_2 = 1.9 Hz, 2H), 8.85 (dd, J_1 = 4.8 Hz, J_2 = 1.6 Hz, 2H), 7.54-7.51 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.2, 167.0, 153.4, 150.4, 136.3, 130.9, 123.7.



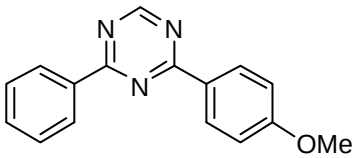
2,4-dicyclopropyl-1,3,5-triazine (**3o**)[1]: 6.4 mg (20%); Colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.70 (s, 1H), 2.11-2.06 (m, 2H), 1.22-1.19 (m, 4H), 1.15-1.10 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 179.6, 164.6, 17.8, 11.9.



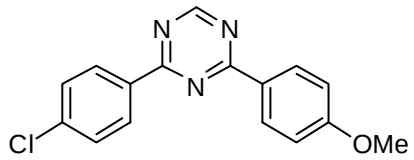
2-methyl-4,6-diphenyl-1,3,5-triazine (**3p**)[1]: 7.5 mg (15%); White solid; Mp: 106-107 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.65-8.62 (m, 4H), 7.59-7.56 (m 2H), 7.55-7.51 (m, 4H), 2.78 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 177.0, 171.2, 135.9, 132.5, 128.9, 128.6, 26.0.



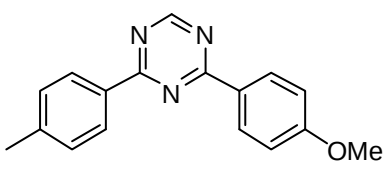
2,4,6-triphenyl-1,3,5-triazine (**3q**)[2]: 24.7 mg (40%); White solid; Mp: 232-233 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.80-8.77 (m, 6H), 7.64-7.56 (m, 9H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 171.6, 136.2, 132.5, 129.0, 128.6.



2-(4-methoxyphenyl)-4-phenyl-1,3,5-triazine (**3ga**)[1]: 12.1 mg (23%); White solid; Mp: 128-129 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.18 (s, 1H), 8.63-8.59 (m, 4H), 7.61-7.58 (m, 1H), 7.56-7.52 (m, 2H), 7.05-7.03 (m, 2H), 3.91 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 171.0, 170.8, 166.5, 163.6, 135.7, 132.6, 130.9, 128.8, 128.7, 128.0, 114.1, 55.5.

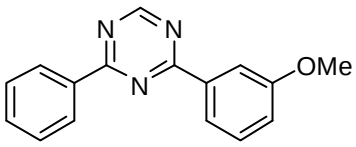


2-(4-chlorophenyl)-4-(4-methoxyphenyl)-1,3,5-triazine (**3gc**)[1]: 9.5 mg (16%); White solid; Mp: 168-169 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.16 (s, 1H), 8.59-8.54 (m, 4H), 7.52-7.49 (m, 2H), 7.05-7.02 (m, 2H), 3.91 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.9, 170.1, 166.5, 163.7, 139.0, 134.2, 130.9, 130.1, 129.0, 127.8, 114.1, 55.5.

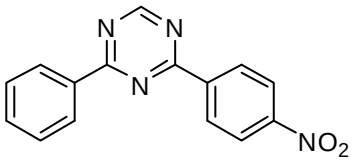


2-(4-methoxyphenyl)-4-(p-tolyl)-1,3,5-triazine (**3gf**): 12.2 mg (22%); White solid; Mp: 124-125 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.16 (s, 1H), 8.60 (dd, J_1 = 7.0 Hz,

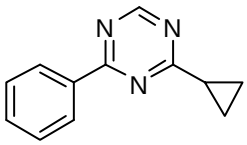
J_2 = 1.9 Hz, 2H), 8.51 (d, J = 8.0 Hz, 2H), 7.35 (d, J = 8.0 Hz, 2H), 7.05-7.03 (m, 2H), 3.91 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 170.9, 170.6, 166.1, 163.6, 143.5, 132.8, 130.9, 129.5, 128.9, 127.9, 114.1, 55.5, 21.7; HRMS (ESI): calcd for $C_{17}H_{16}N_3O$ $[M+H]^+$ 278.1288, found 278.1283.



2-(3-methoxyphenyl)-4-phenyl-1,3,5-triazine (**3ja**)[3]: 8.4 mg (16%); White solid; Mp: 82-83°C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.26 (s, 1H), 8.65-8.63 (m, 2H), 8.26 (d, J = 7.8 Hz, 1H), 8.19-8.17 (m, 1H), 7.63-7.58 (m, 1H), 7.57-7.54 (m, 2H), 7.47 (t, J = 7.8 Hz, 1H), 7.17-7.14 (m, 1H), 3.95 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 171.3, 171.1, 166.7, 160.0, 136.9, 135.5, 132.8, 129.8, 128.9, 128.8, 121.4, 119.2, 113.4, 55.5.

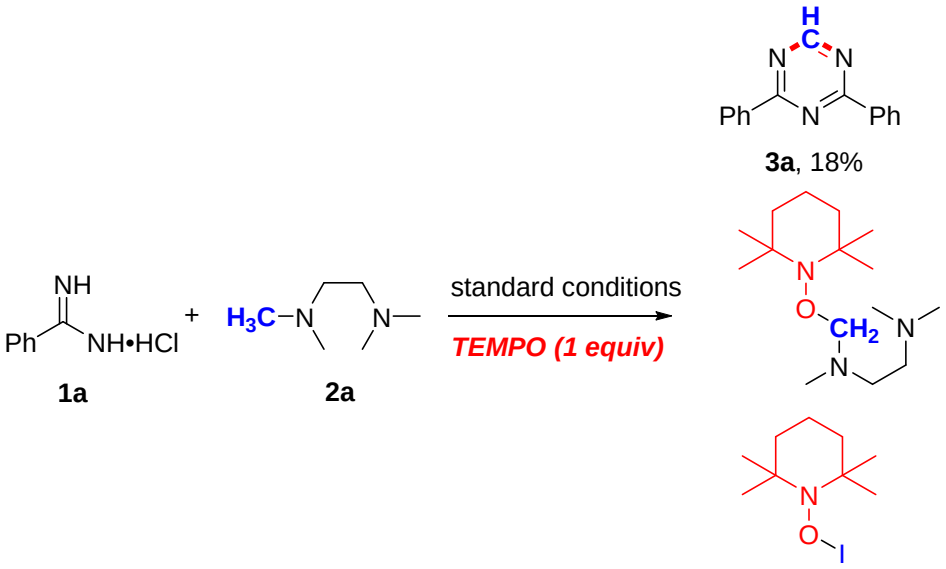


2-(4-nitrophenyl)-4-phenyl-1,3,5-triazine (**3pa**)[1]: 15.0 mg (27%); Light yellow solid; Mp: 165-166°C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.33 (s, 1H), 8.84-8.81 (m, 2H), 8.66-8.63 (m, 2H), 8.41-8.38 (m, 2H), 7.66-7.63 (m, 1H), 7.60-7.56 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 171.8, 169.5, 167.0, 150.5, 141.3, 134.9, 133.3, 129.8, 129.0, 128.9, 123.8.

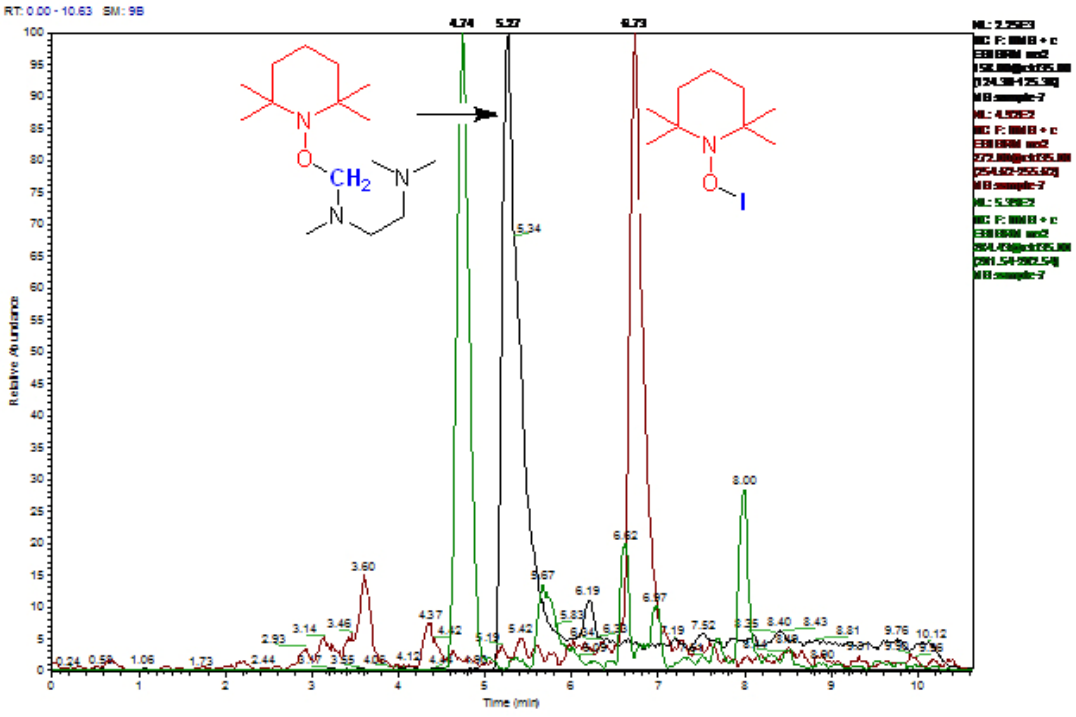


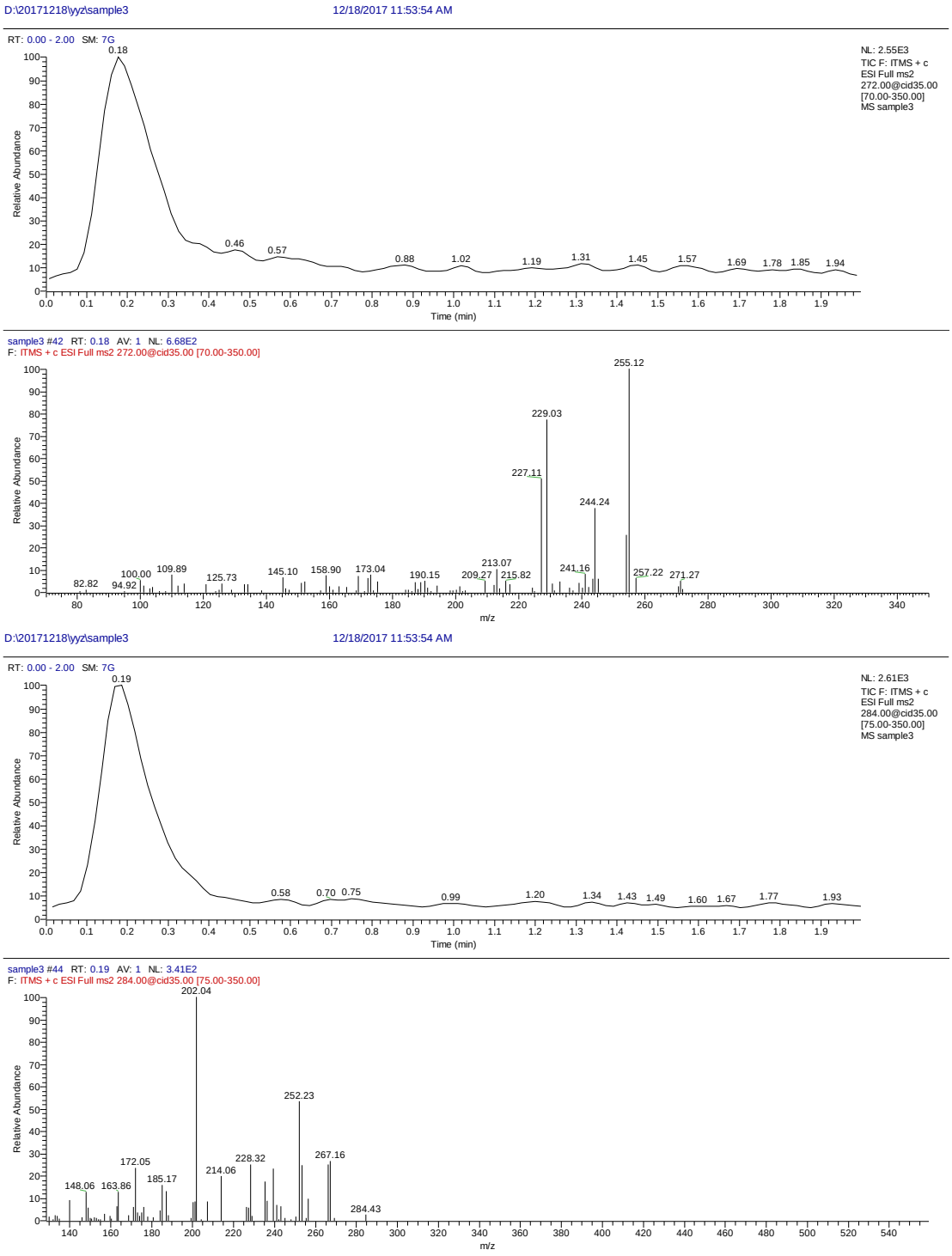
2-cyclopropyl-4-phenyl-1,3,5-triazine (**3oa**)[1]: 5.5 mg (14%); White solid; Mp: 55-56°C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.98 (s, 1H), 8.50-8.47 (m, 2H), 7.58-7.55 (m, 1H), 7.52-7.48 (m, 2H), 2.27-2.22 (m, 1H), 1.36-1.33 (m, 2H), 1.23-1.19 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 180.7, 170.4, 165.6, 135.4, 132.6, 128.7, 128.6, 18.2, 12.2.

Radical Trapping Experiment



1a (0.4 mmol), I_2 (0.4 mmol), TEMPO (0.4 mmol), Cs_2CO_3 (0.8 mmol) were added to a 10 mL Schlenk tube, followed by addition of DMSO (1.0 mL) and **2a** (0.4 mmol). The mixture was stirred at 140 °C for 24 h. The solution was then cooled to r.t., quenched by a $Na_2S_2O_3$ aqueous solution and extracted with EtOAc (3×10 mL). The combined organic layers were dried over Na_2SO_4, filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 60:1) to afford 2,4-diphenyl-1,3,5-triazine (**3a)** in 18% yield. Meanwhile, **t**race amount of residue was dissolved in CH_3OH/H_2O and analyzed by LC-MS.

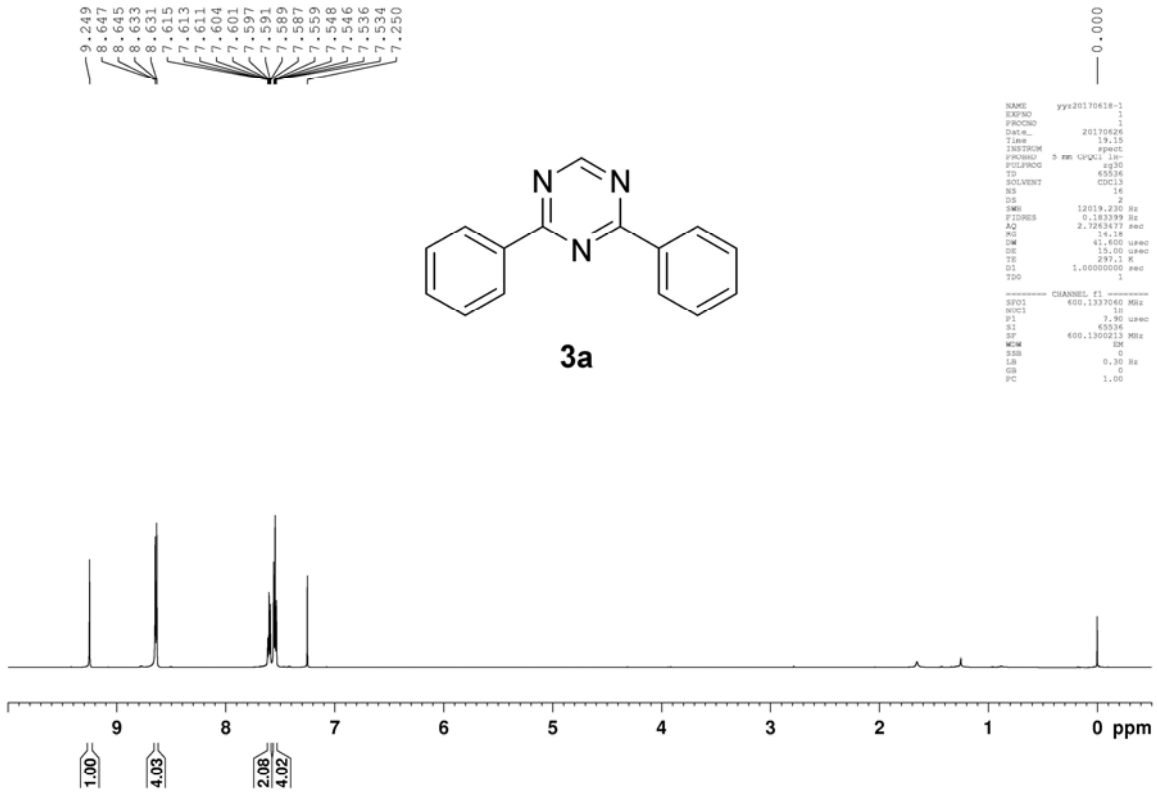


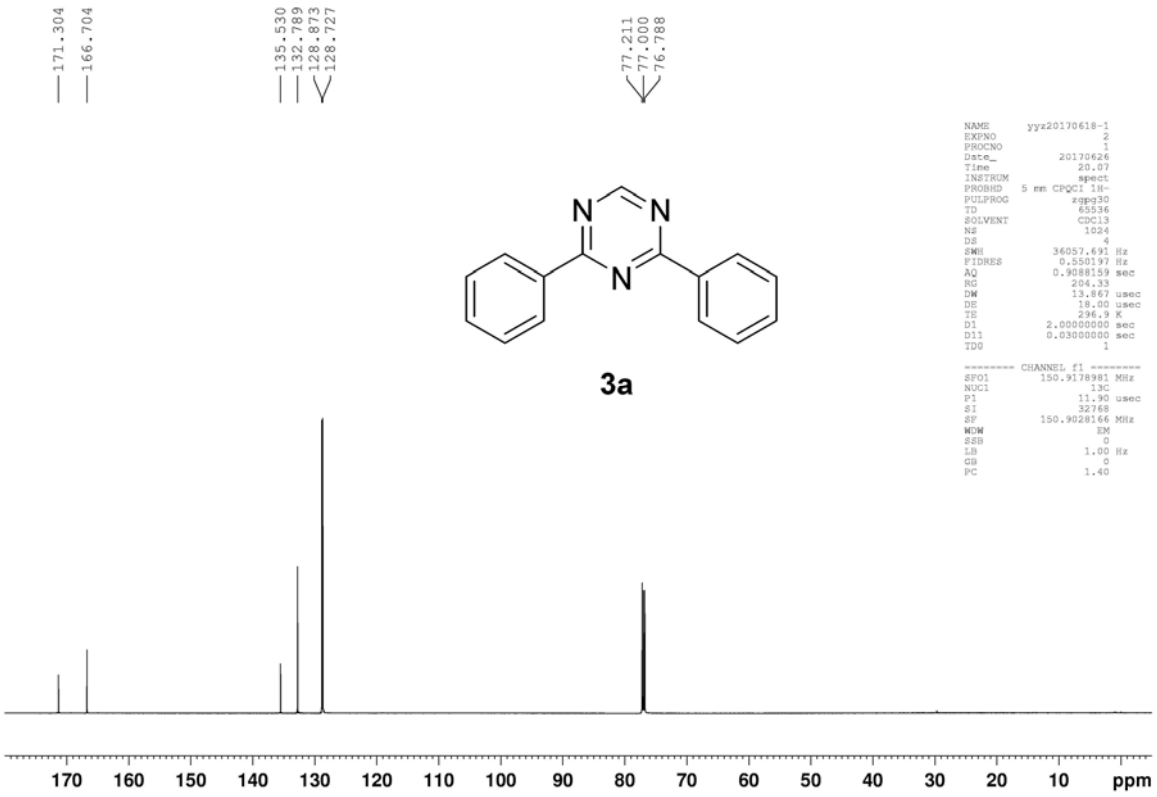


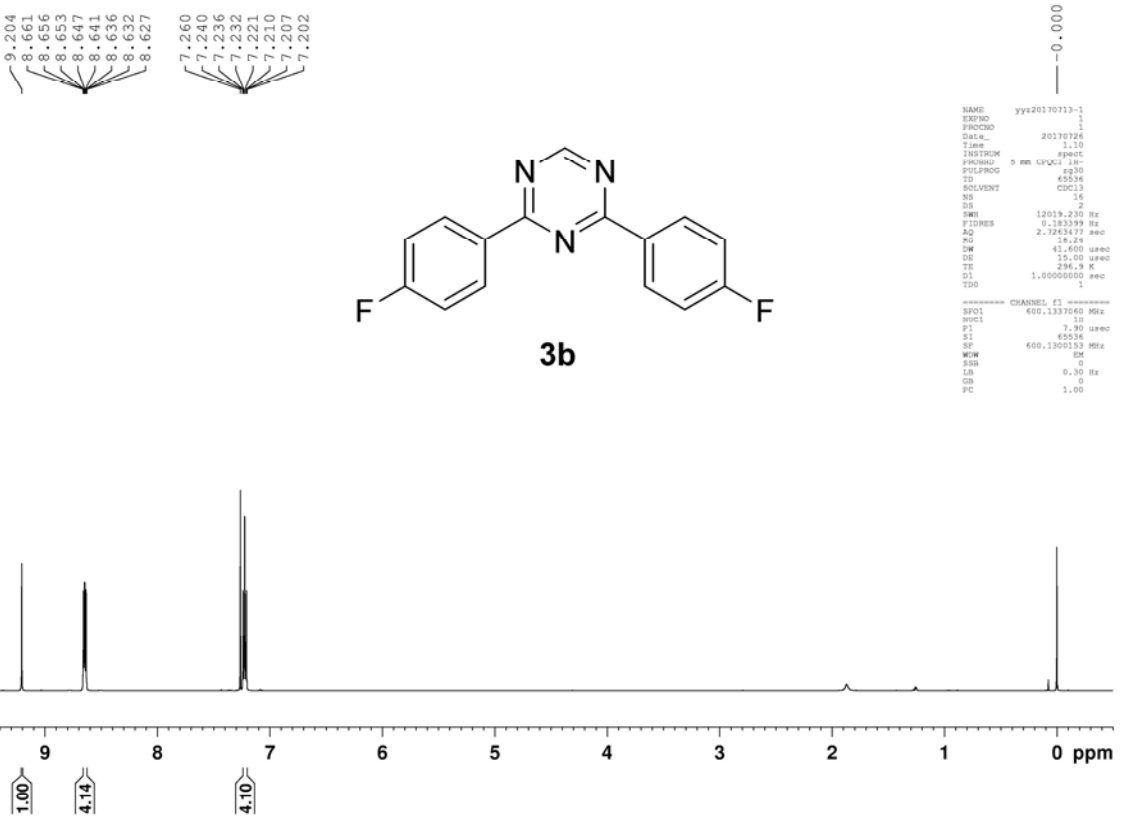
References

1. Y. Yan, Z. Li, H. Li, C. Cui, M. Shi, Y. Liu, *Org. Lett.* **2017**, *19*, 6228.
2. a) Q. You, F. Wang, C. Wu, T. Shi, D. Min, H. Chen, W. Zhang, *Org. Biomol. Chem.* **2015**, *13*, 6723; b) W. Guo, *Org. Biomol. Chem.* **2015**, *13*, 10285; c) A. R. Tiwari, B. M. Bhanage, *Green Chem.* **2016**, *18*, 144.
3. X. Xu, M. Zhang, H. Jiang, J. Zheng, Y. Li, *Org. Lett.* **2014**, *16*, 3540.

^{1}H NMR and ^{13}C NMR Spectra



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7.221
7.210
7.207
7.202
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3b
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8
7
6
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4
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2
1
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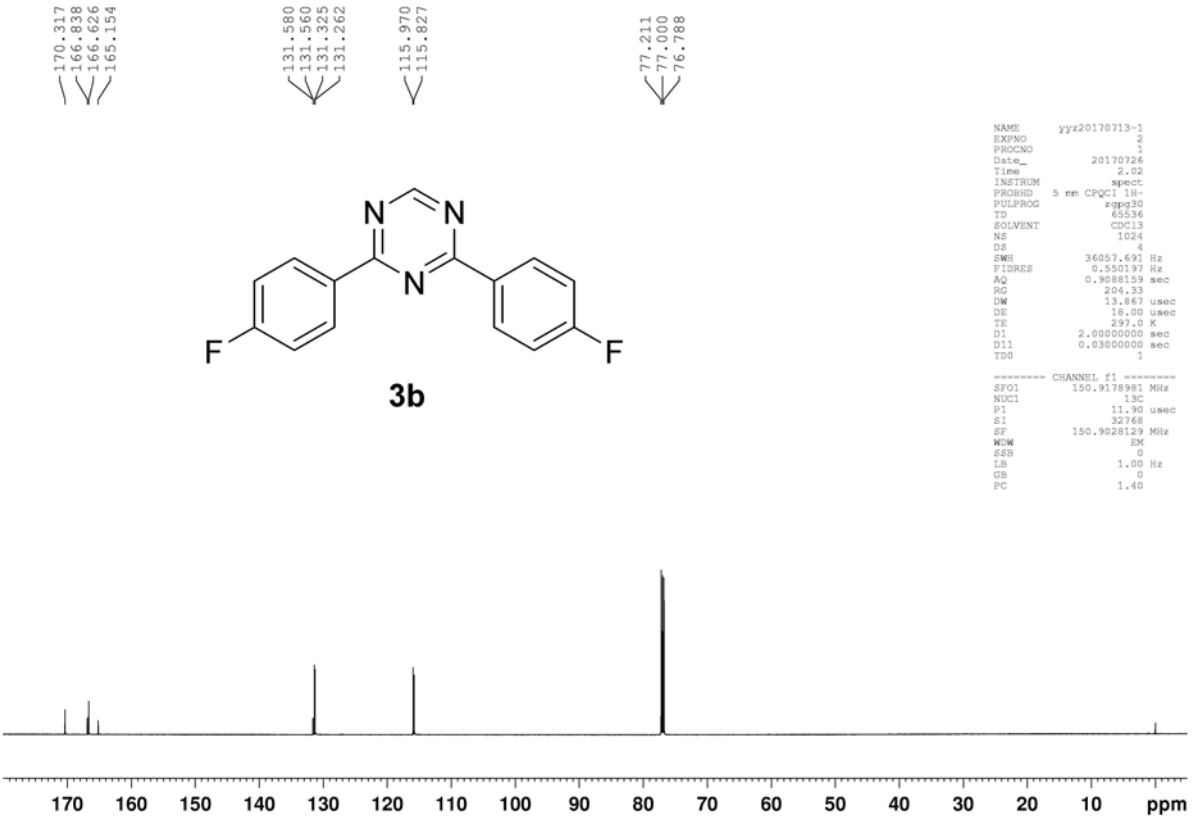
4.14
4.10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76.788
F
F
3b
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150
140
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110
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90
80
70
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30
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10
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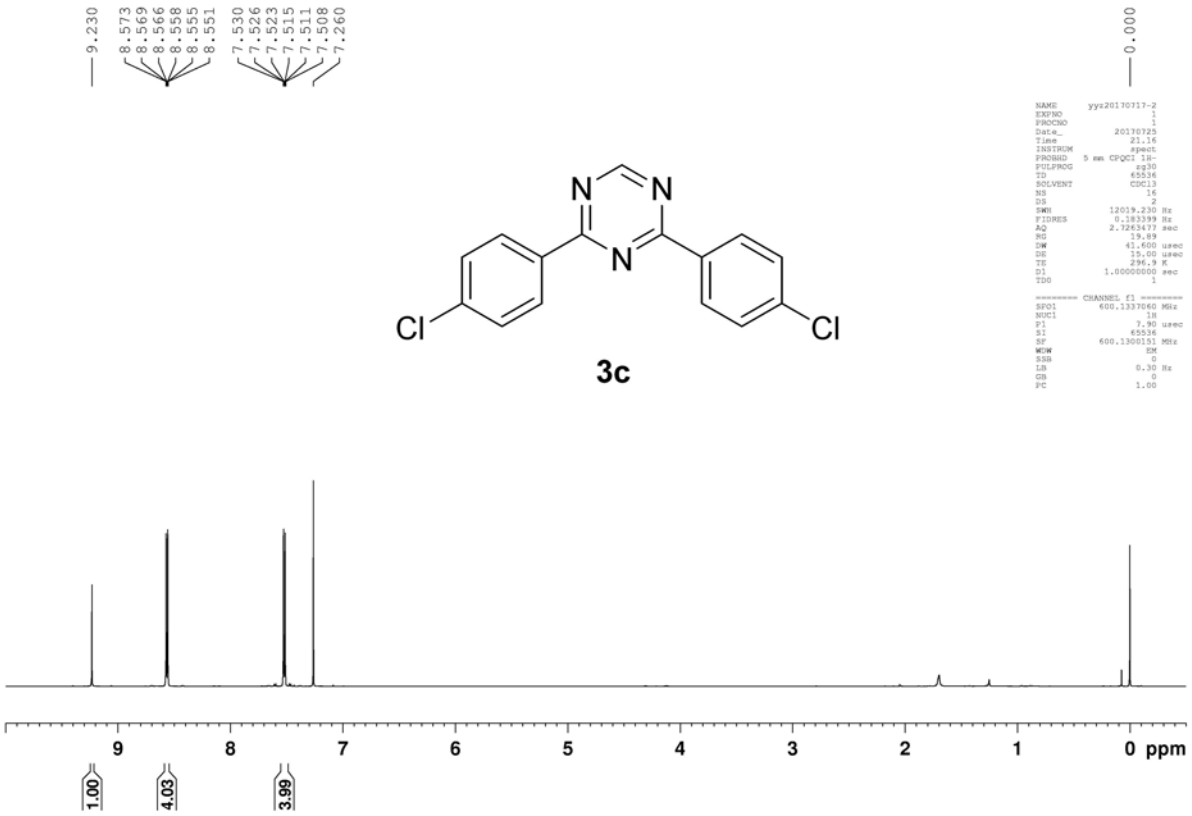
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7.508
7.260
0.000
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Cl
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6
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4
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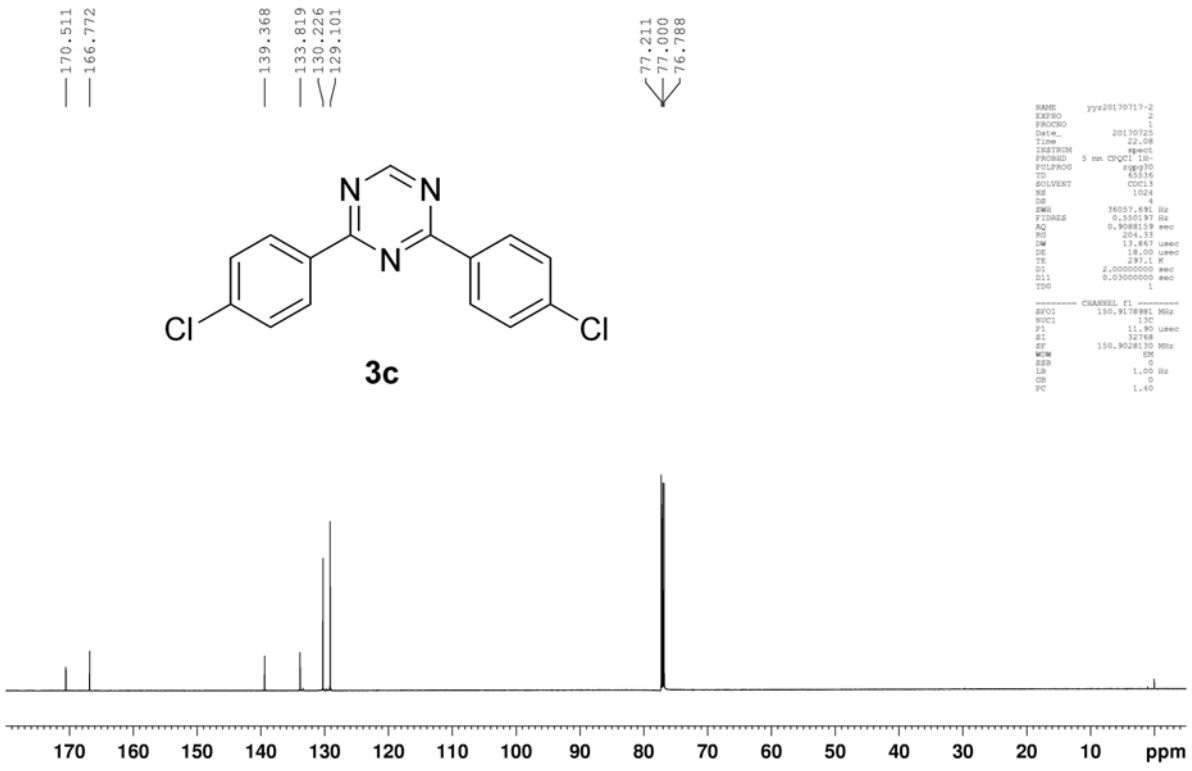
3.99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N
Cl
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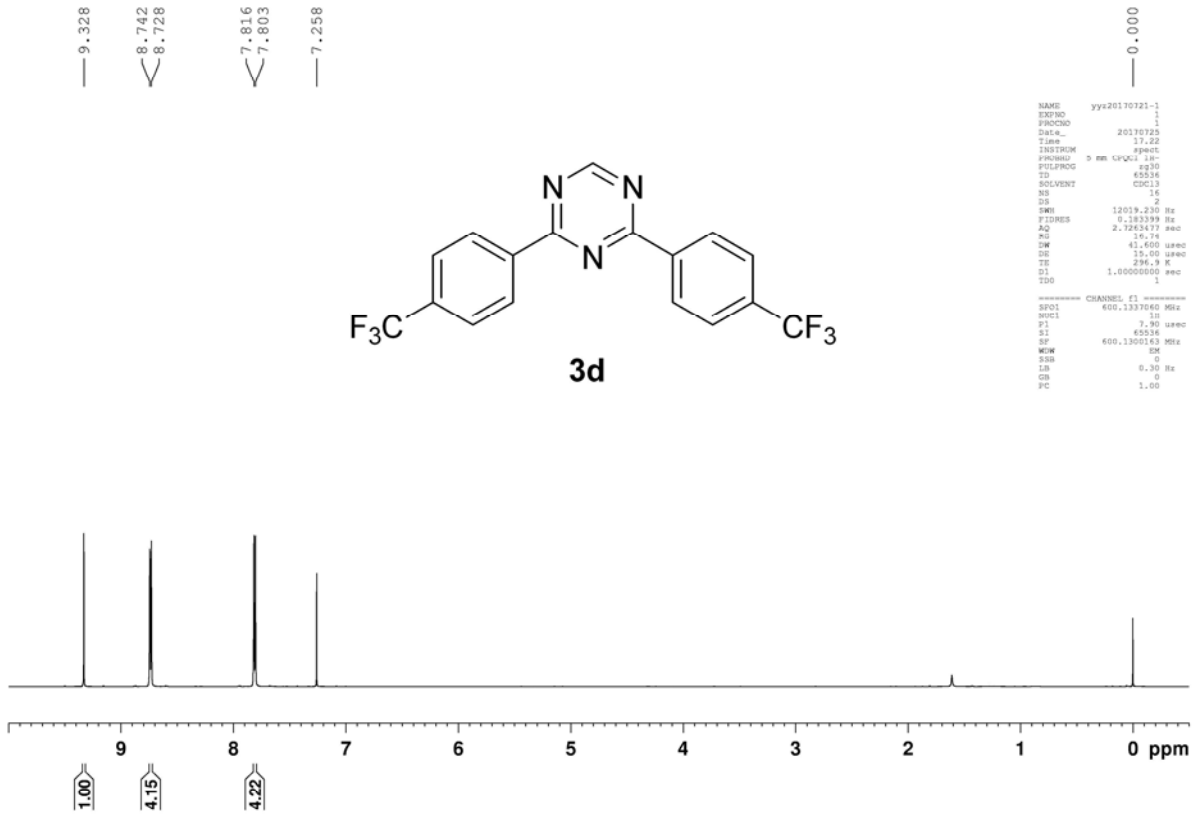
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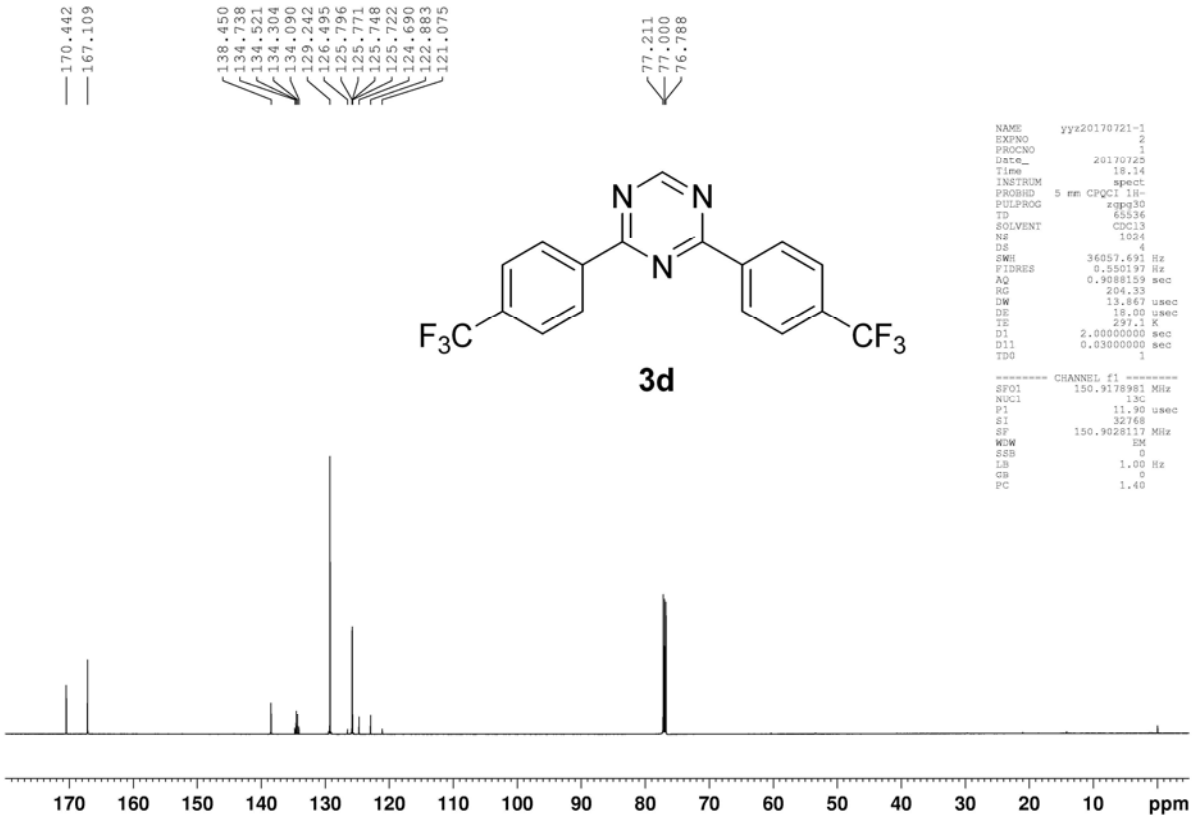
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77.000
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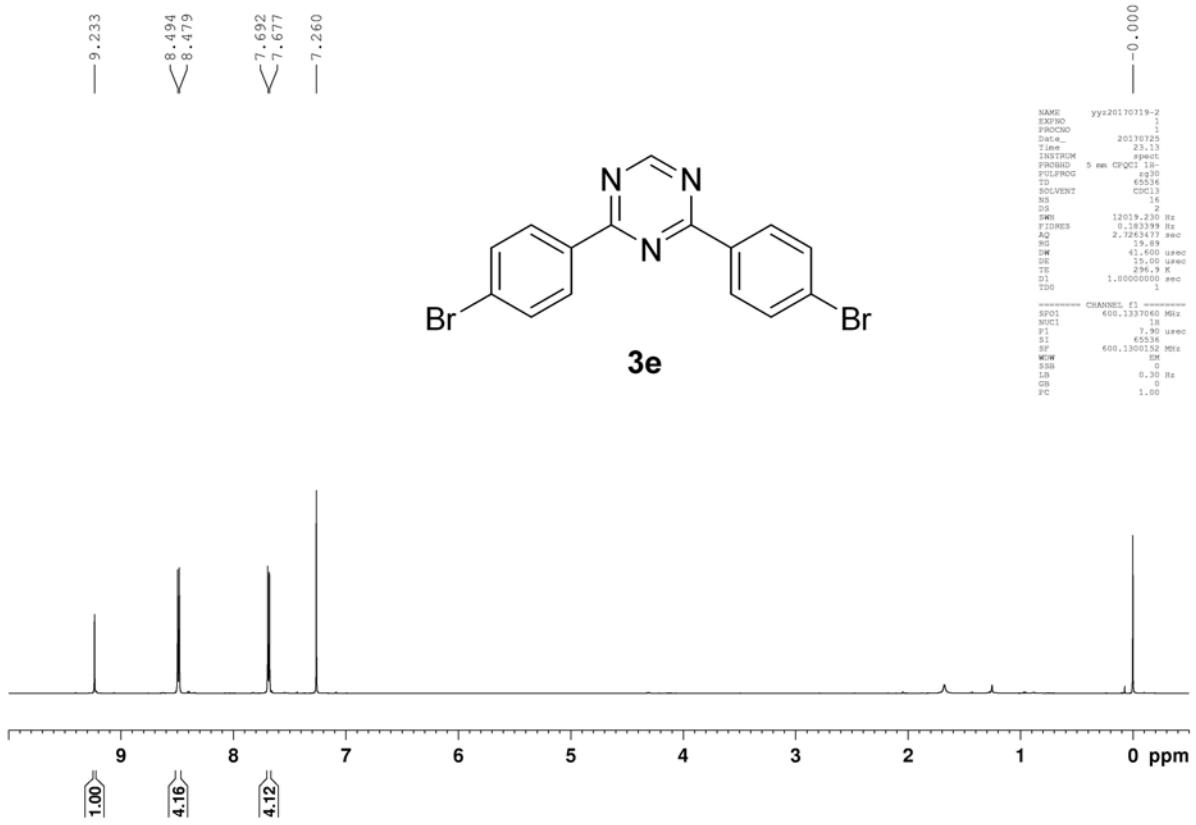
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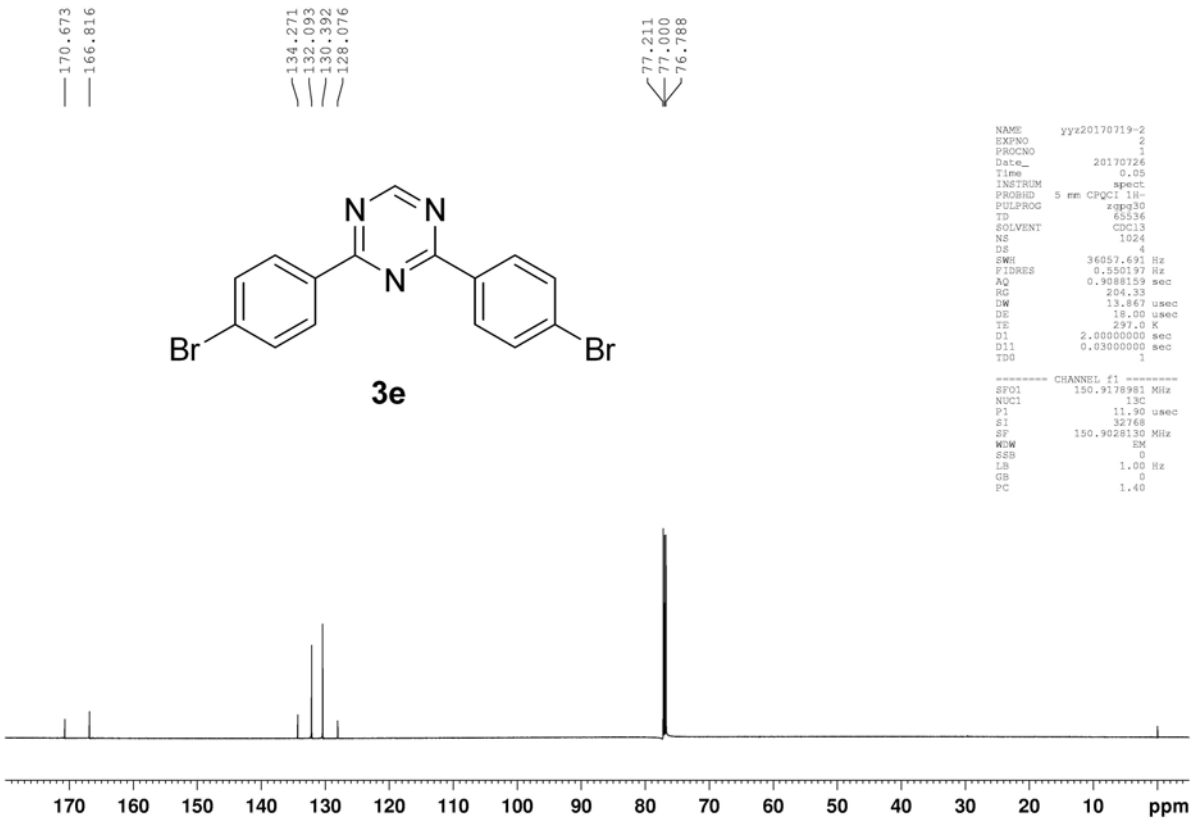
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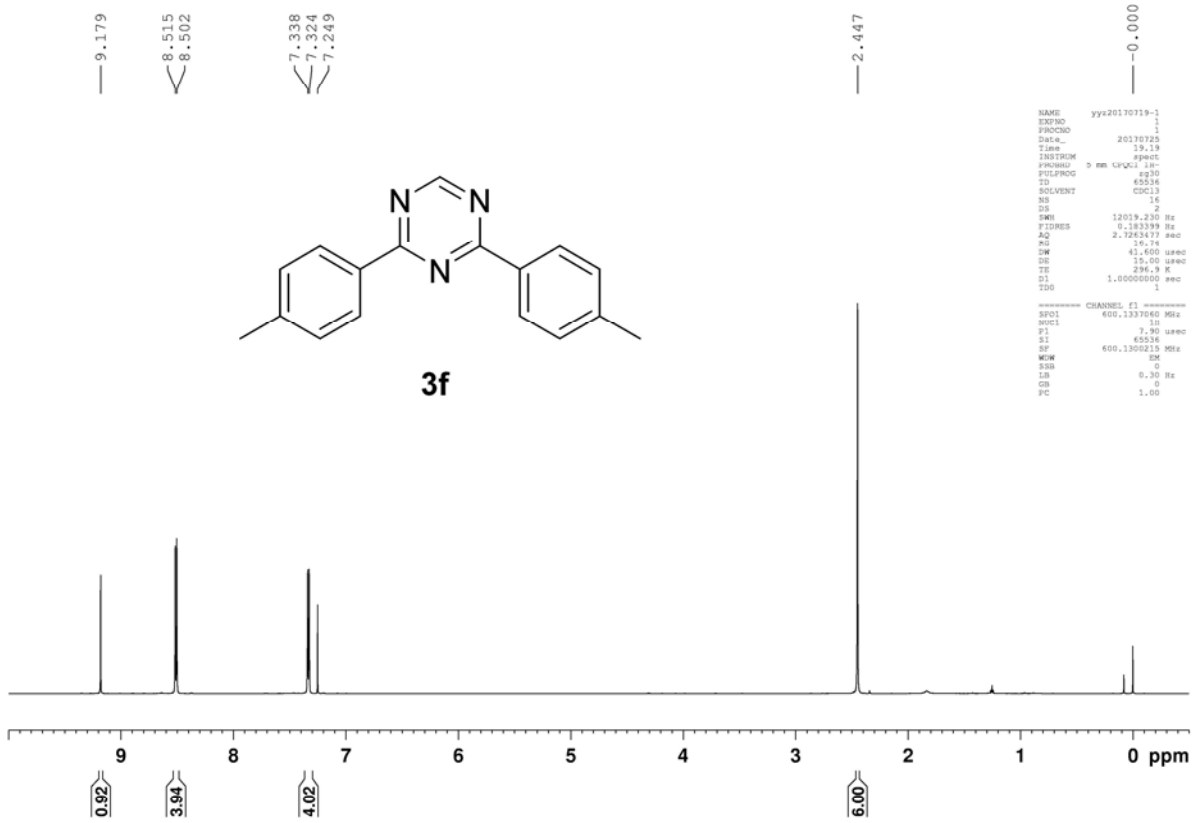
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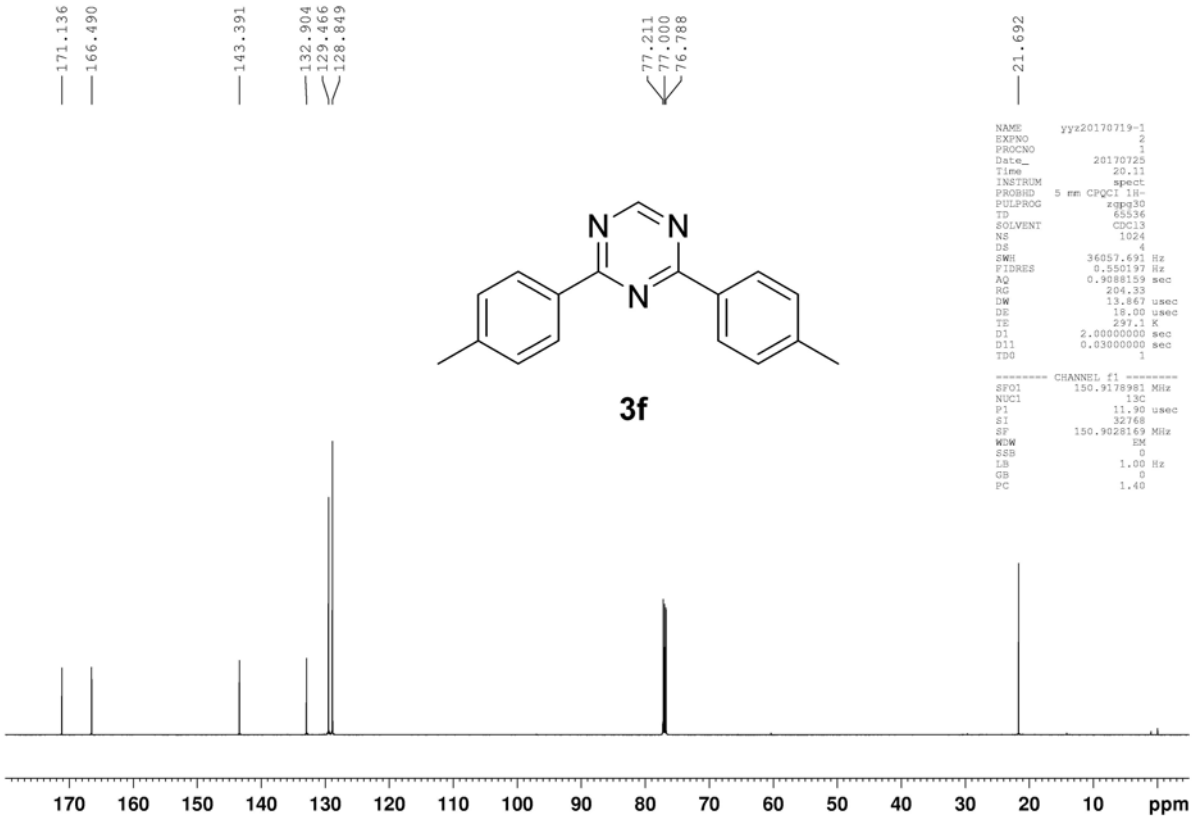
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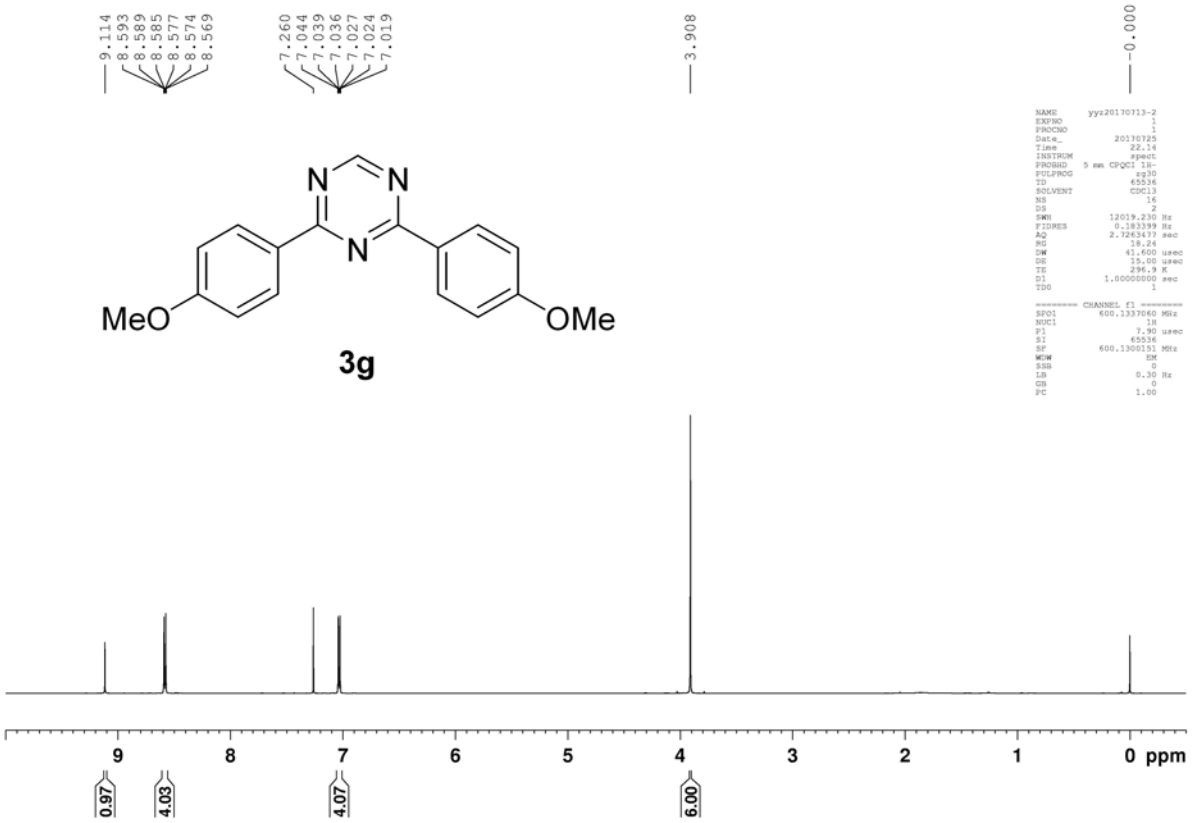
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0.000
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3
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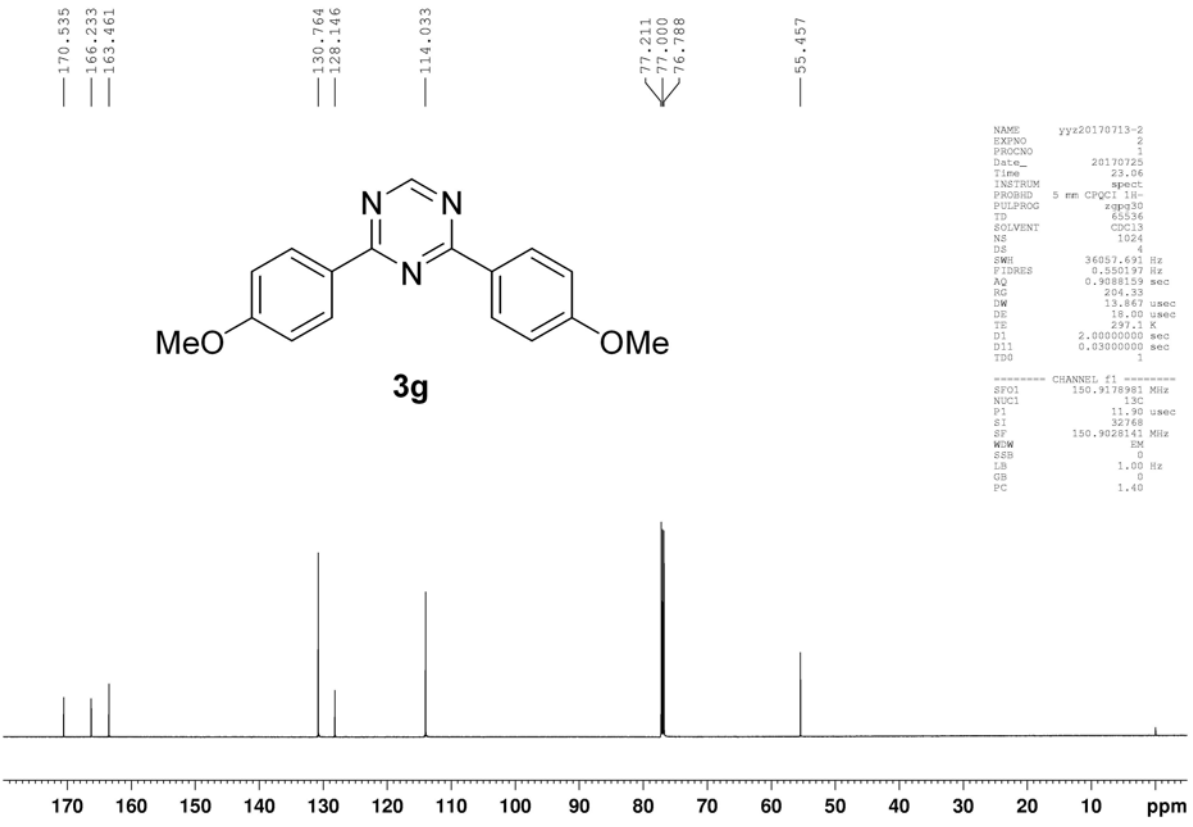
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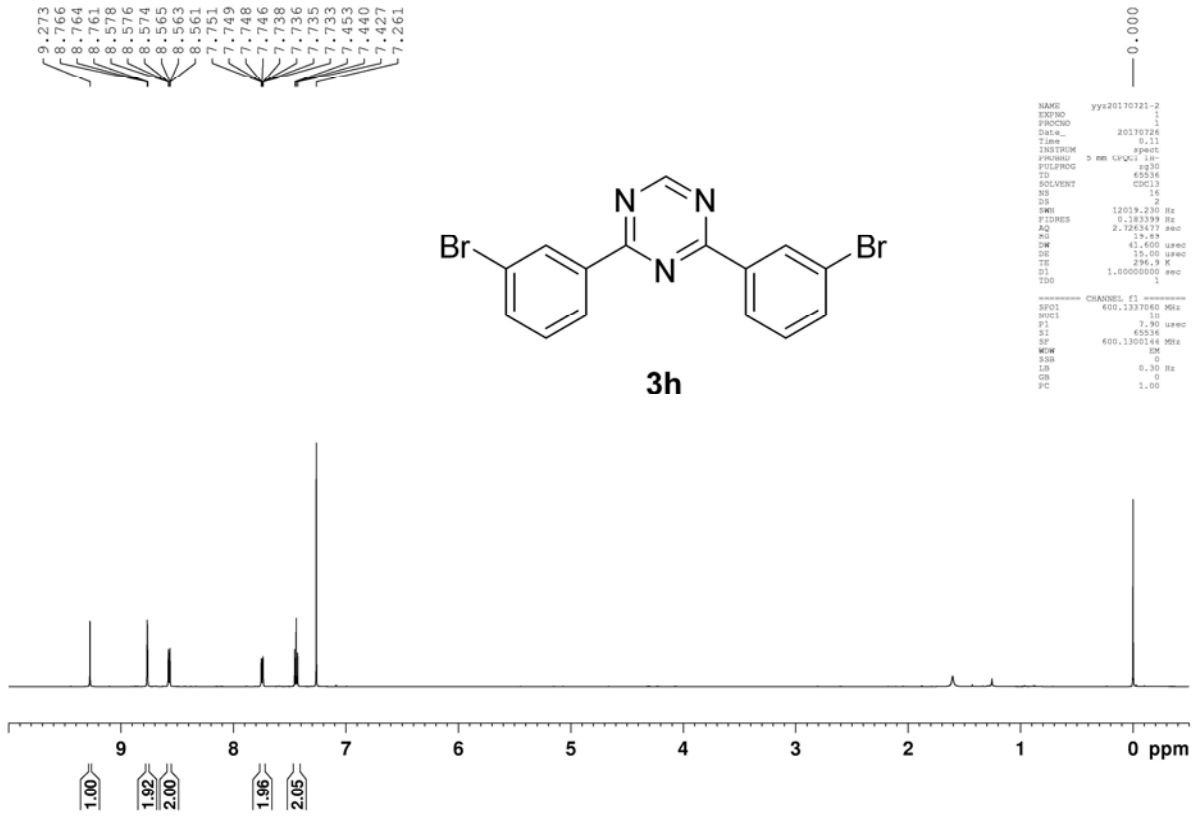
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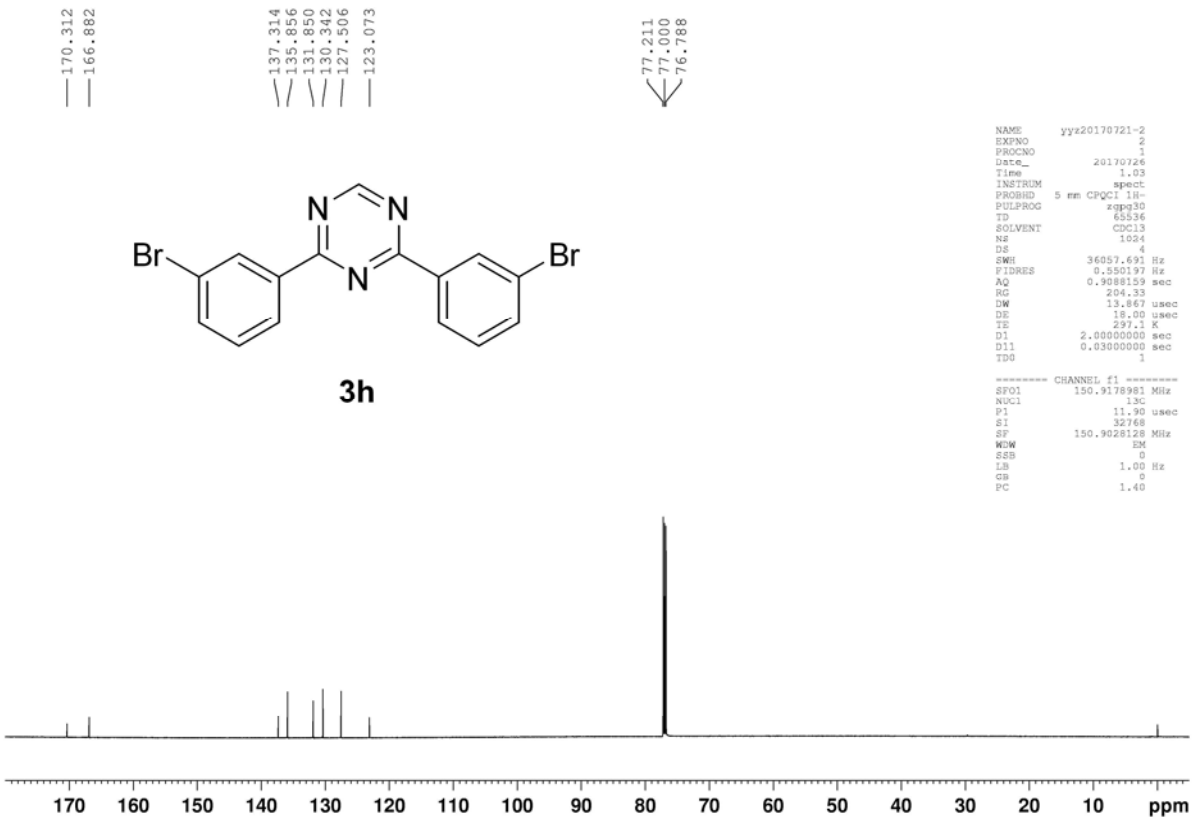
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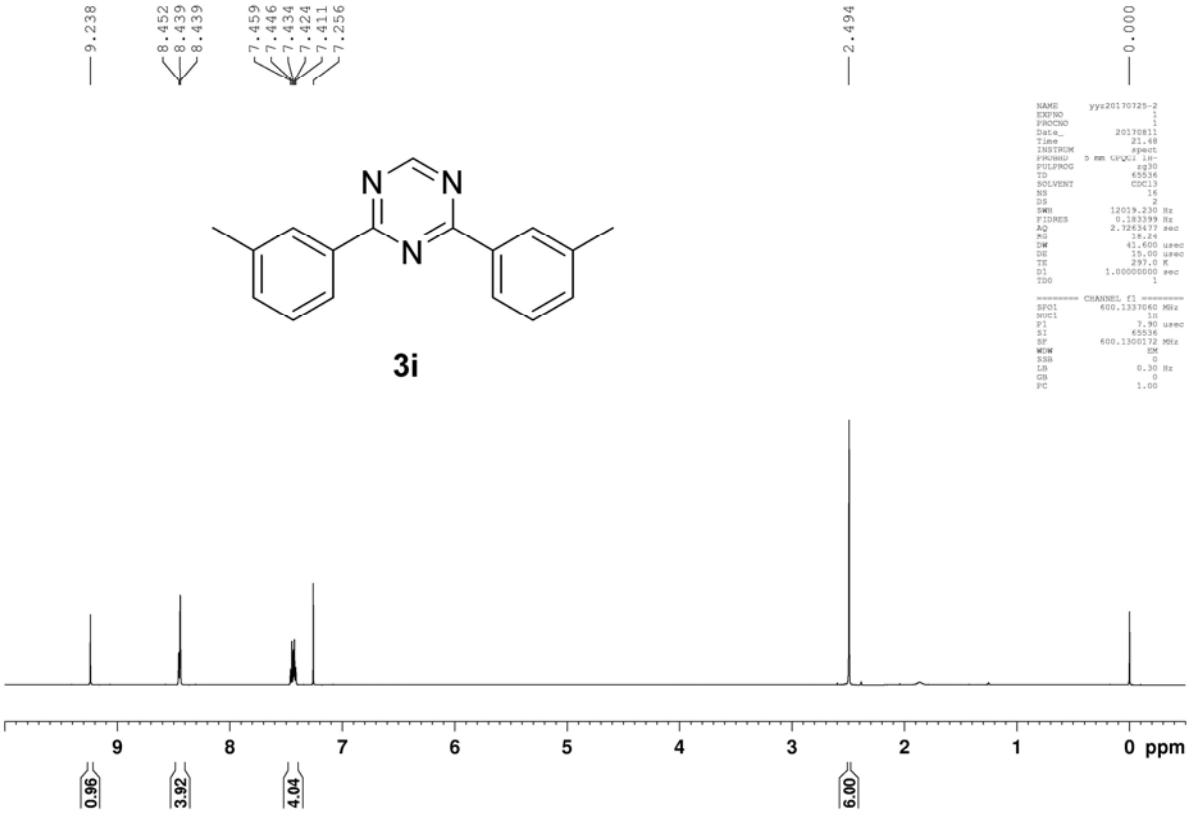
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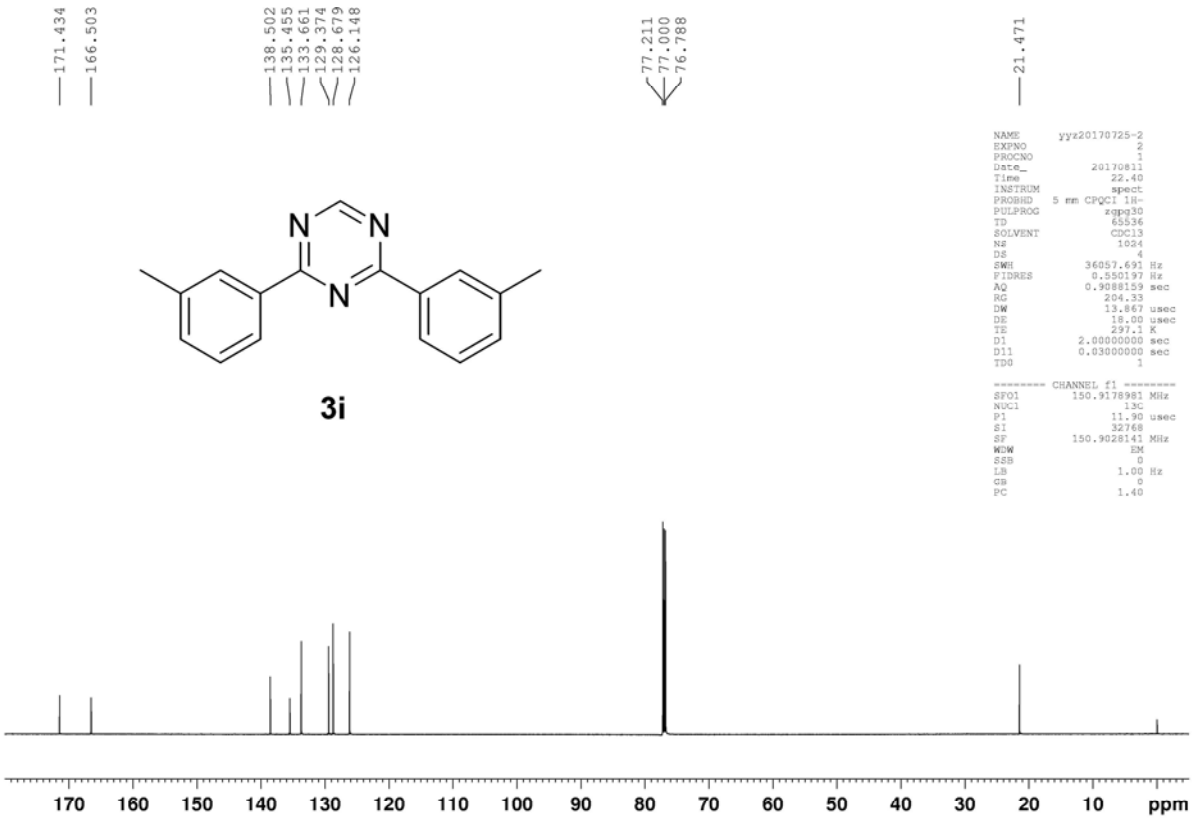
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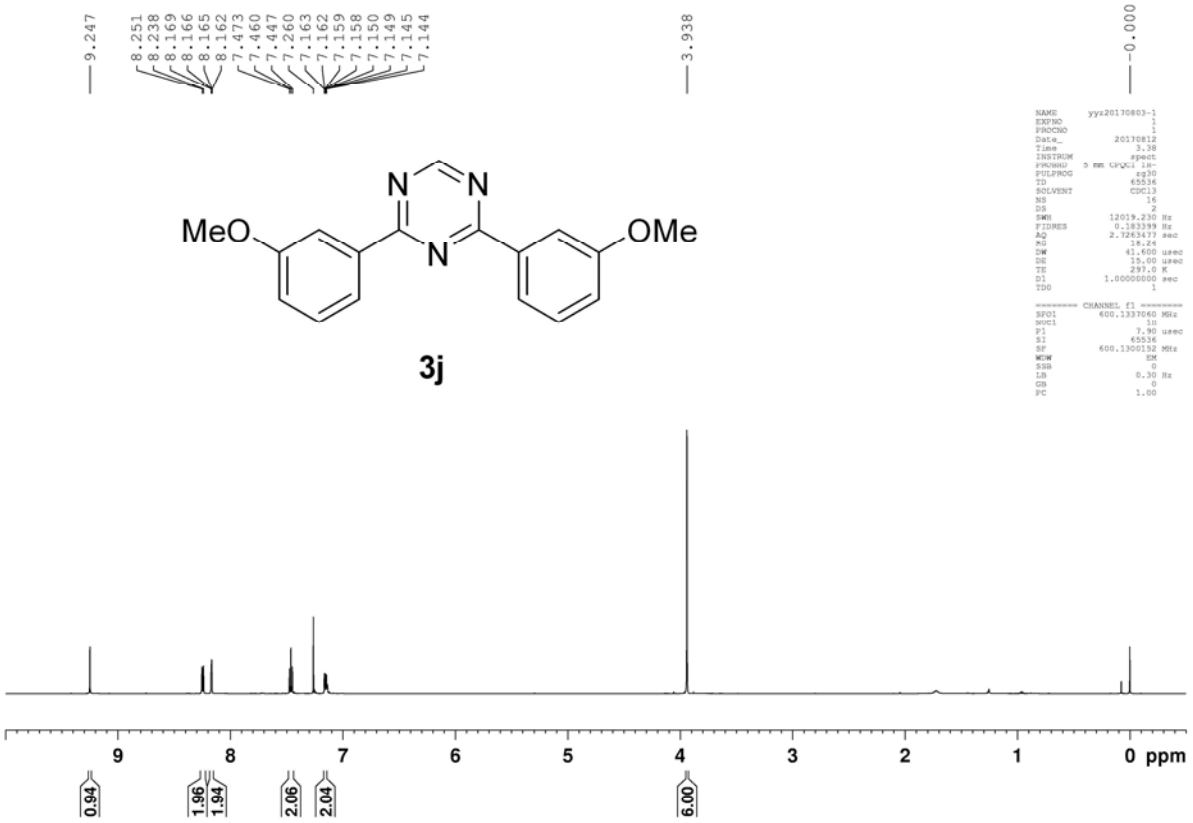
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OMe
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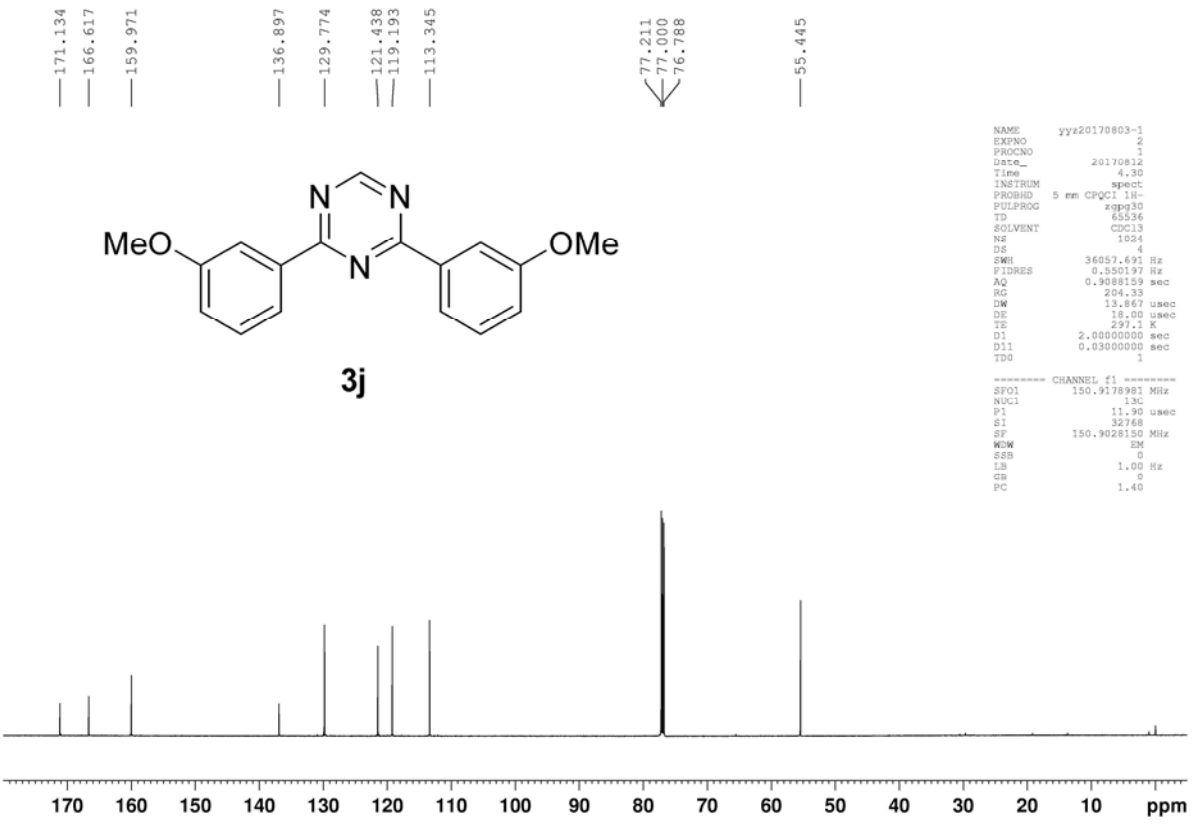
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N
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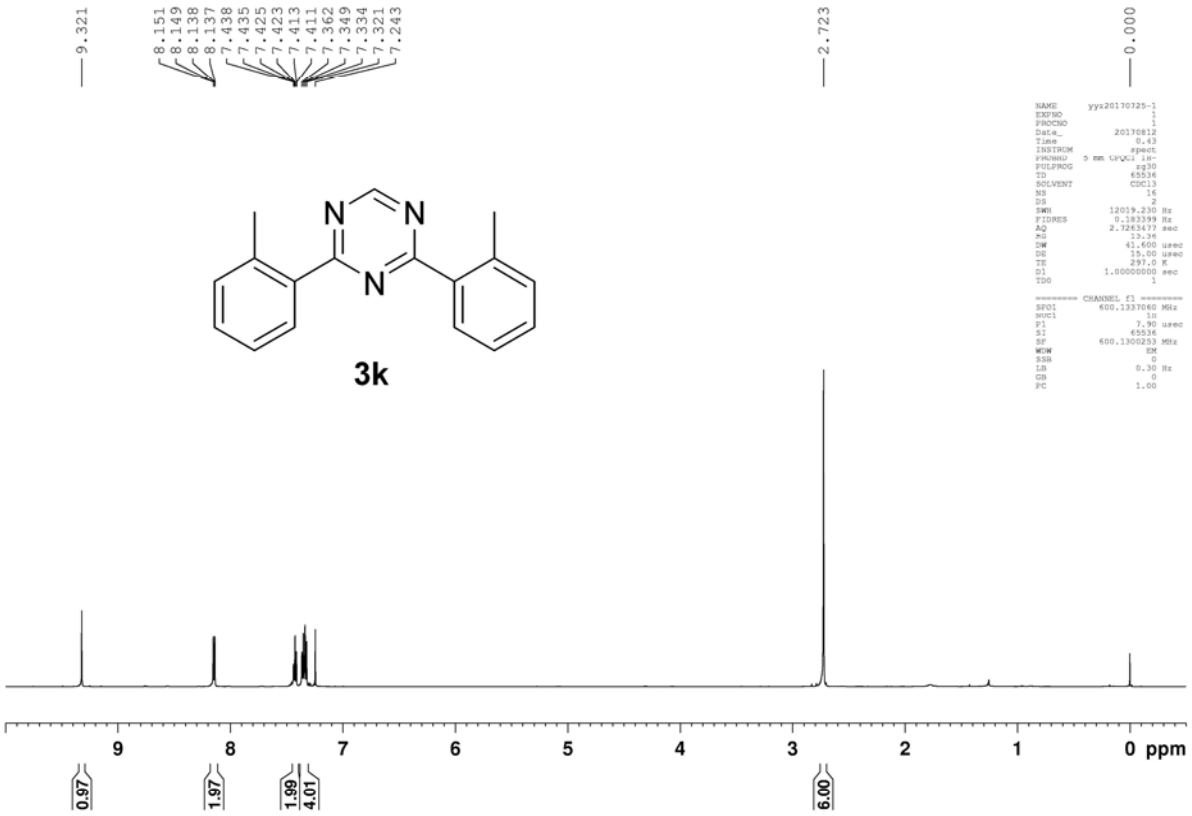
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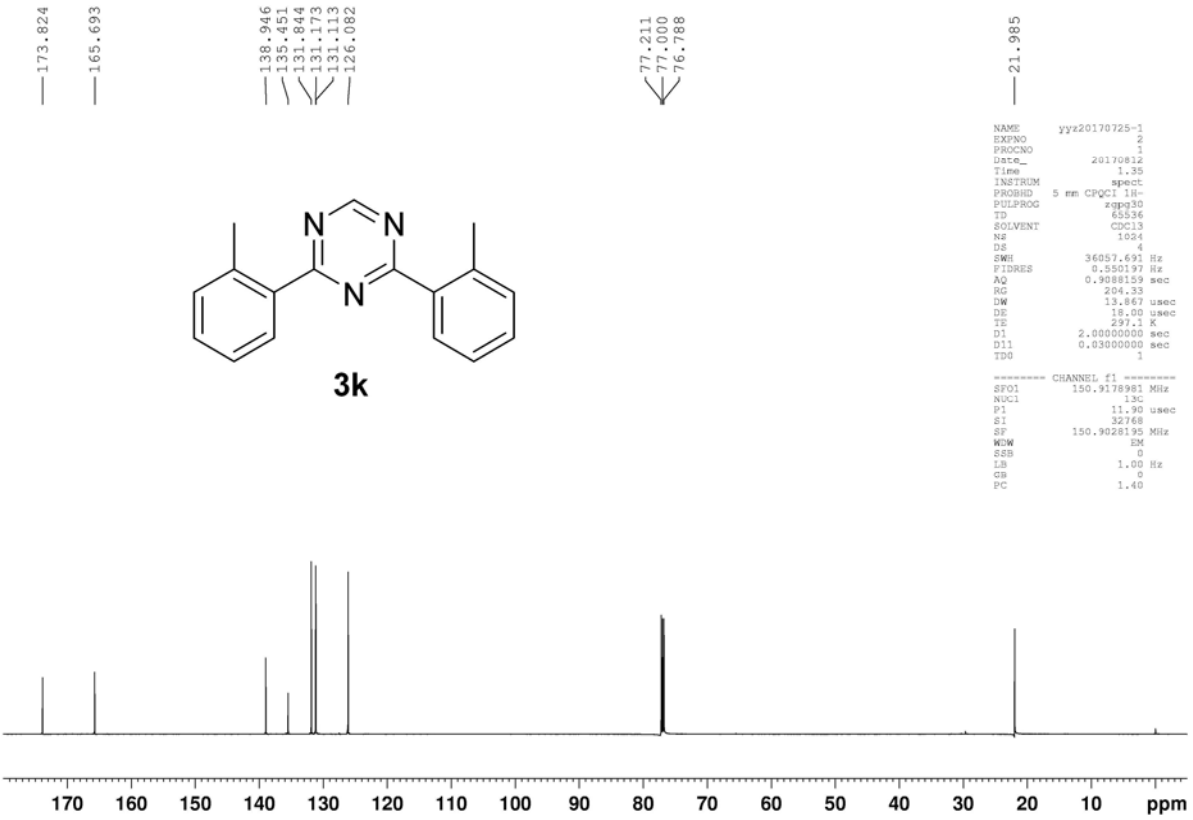
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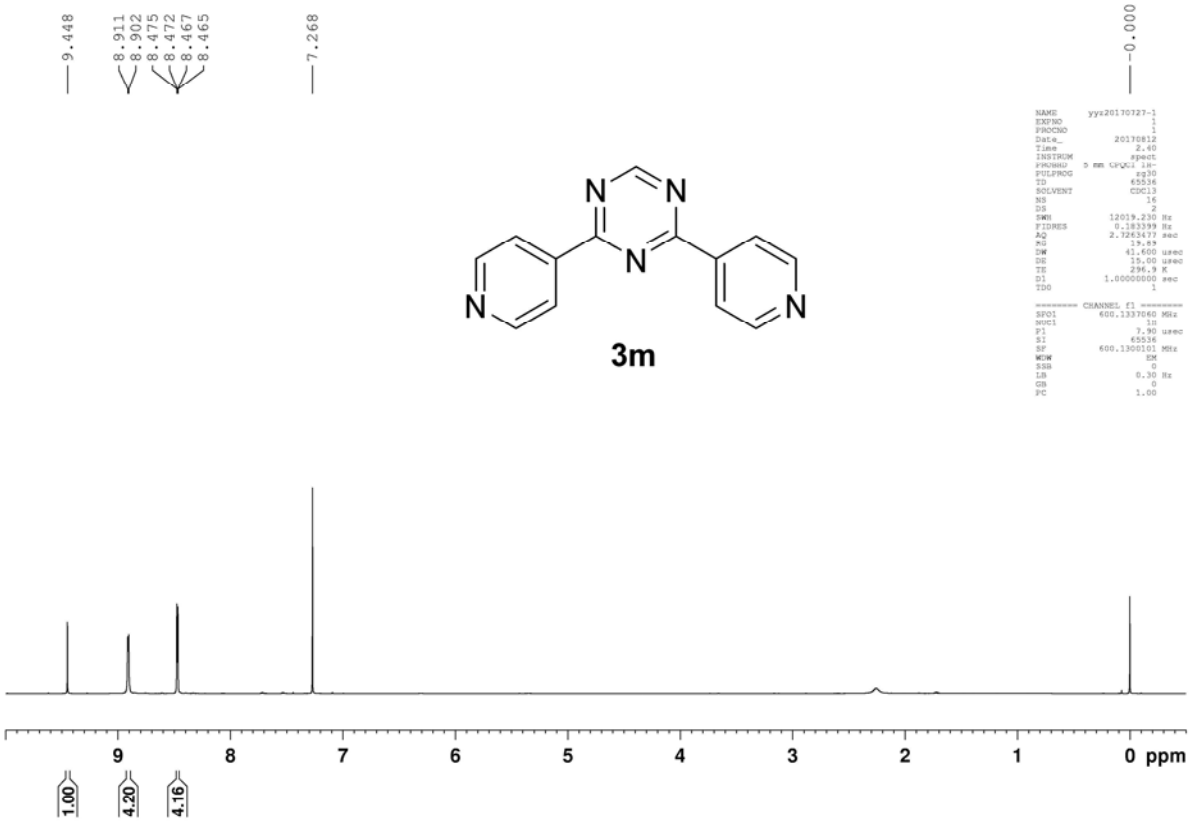
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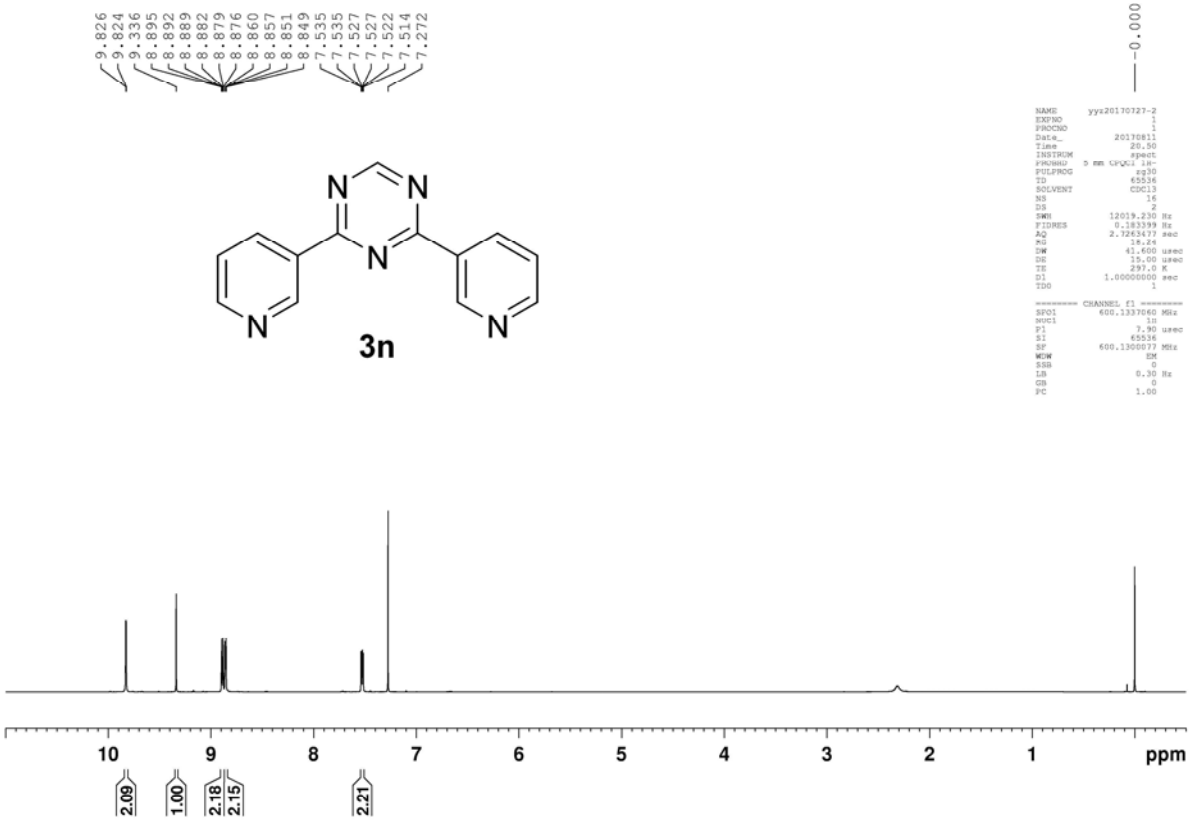
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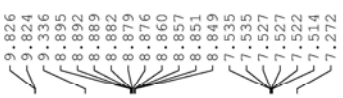
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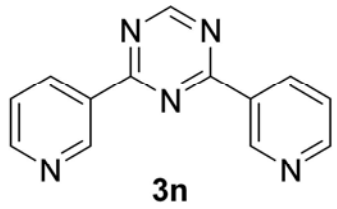
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DE 15.00 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
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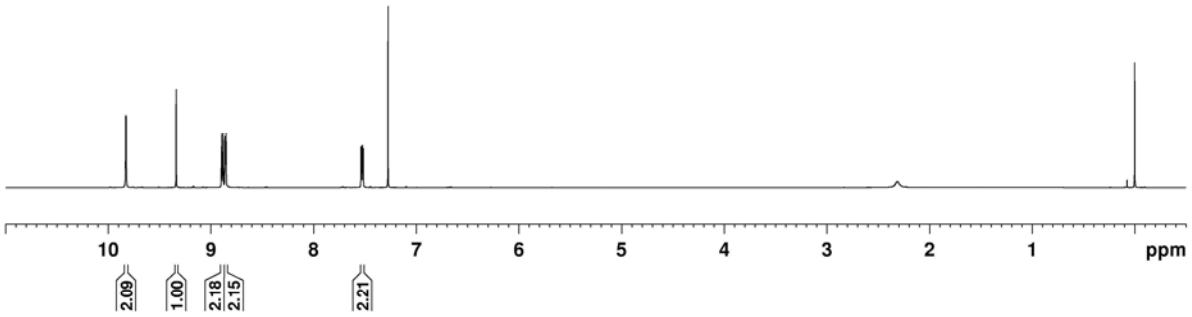
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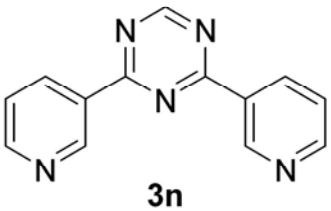
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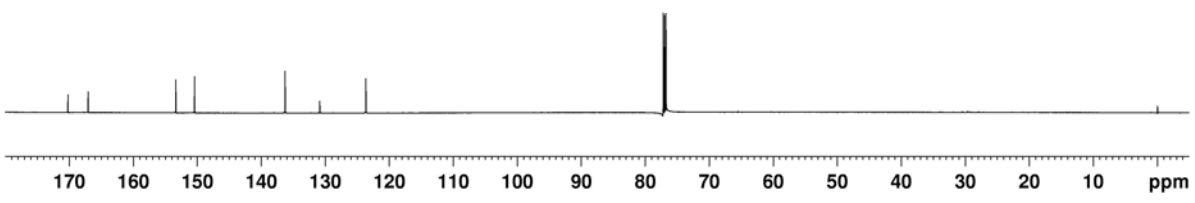
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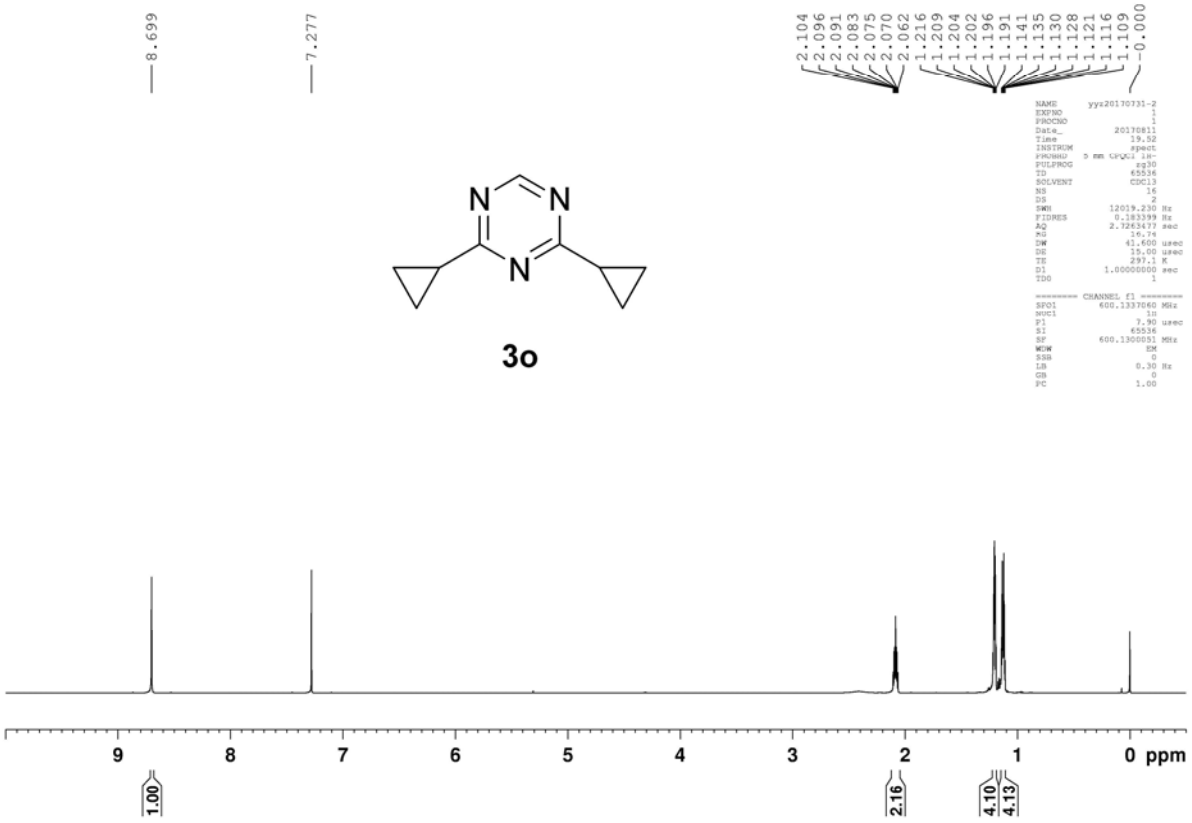
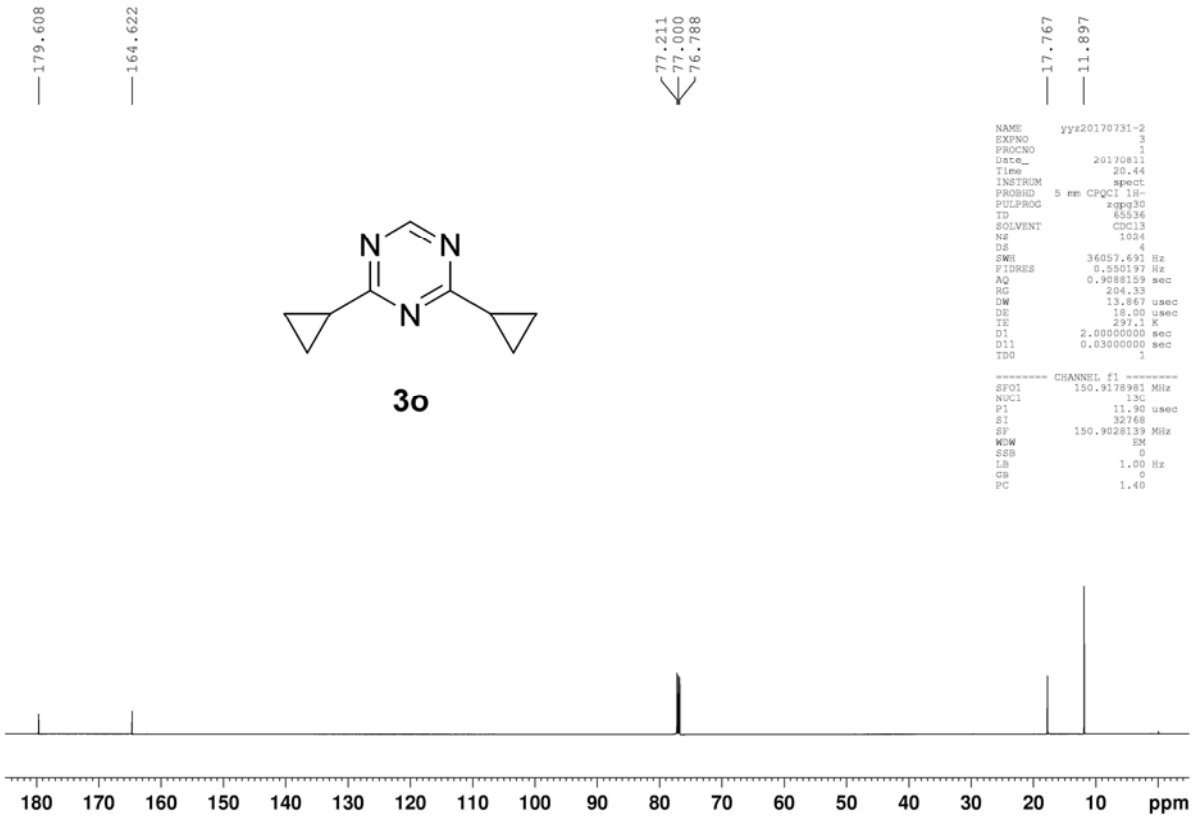
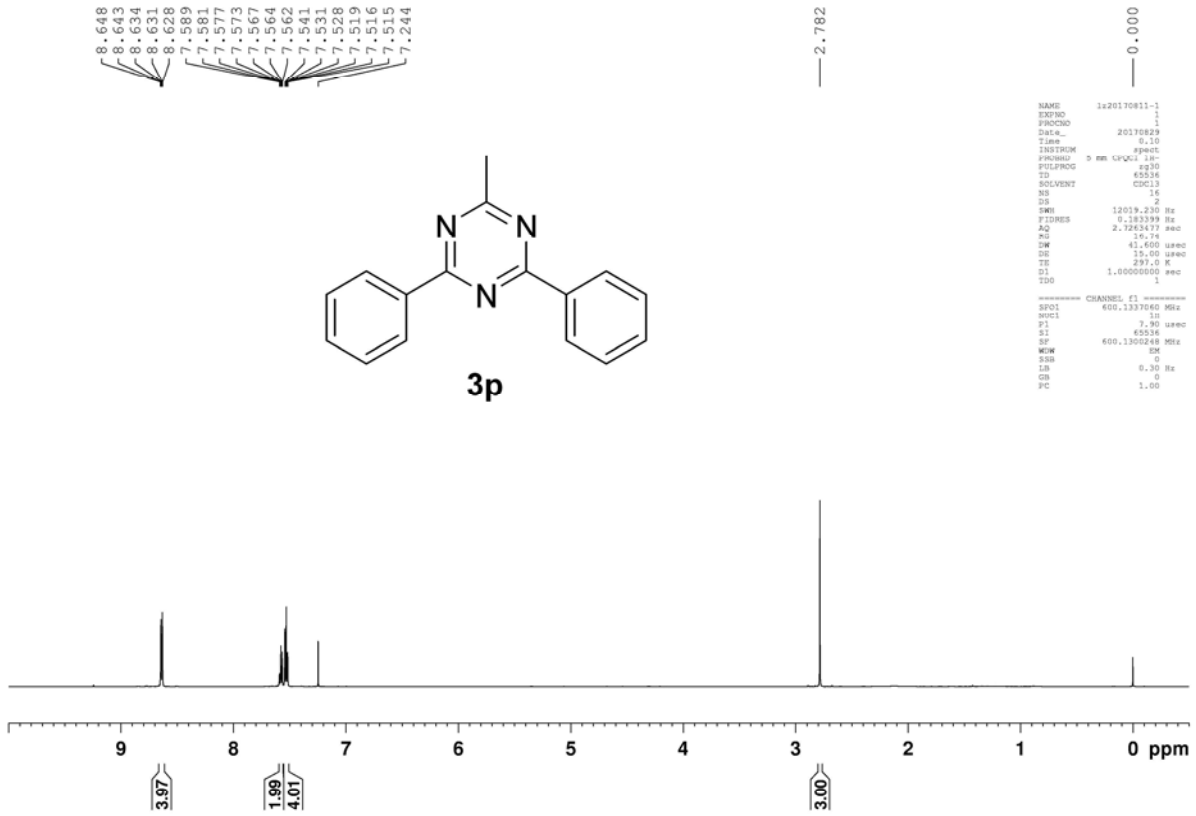
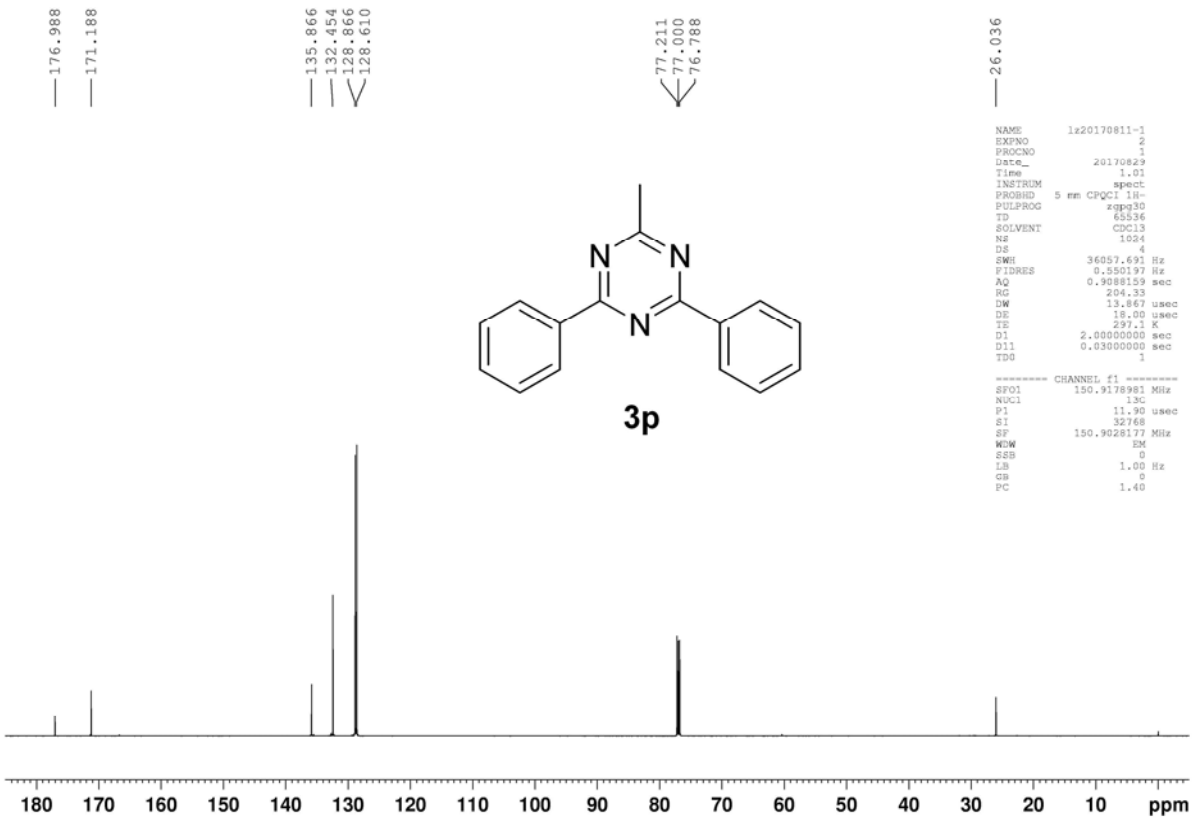
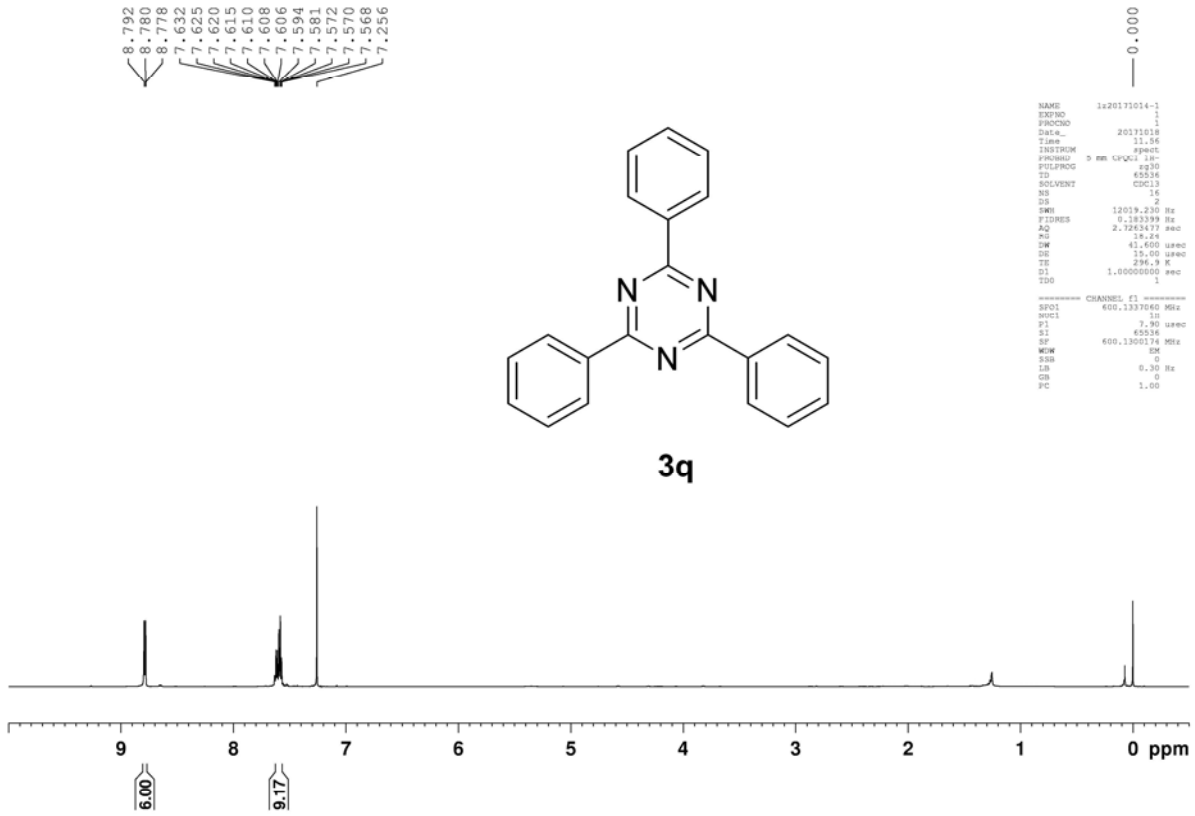
10
ppm

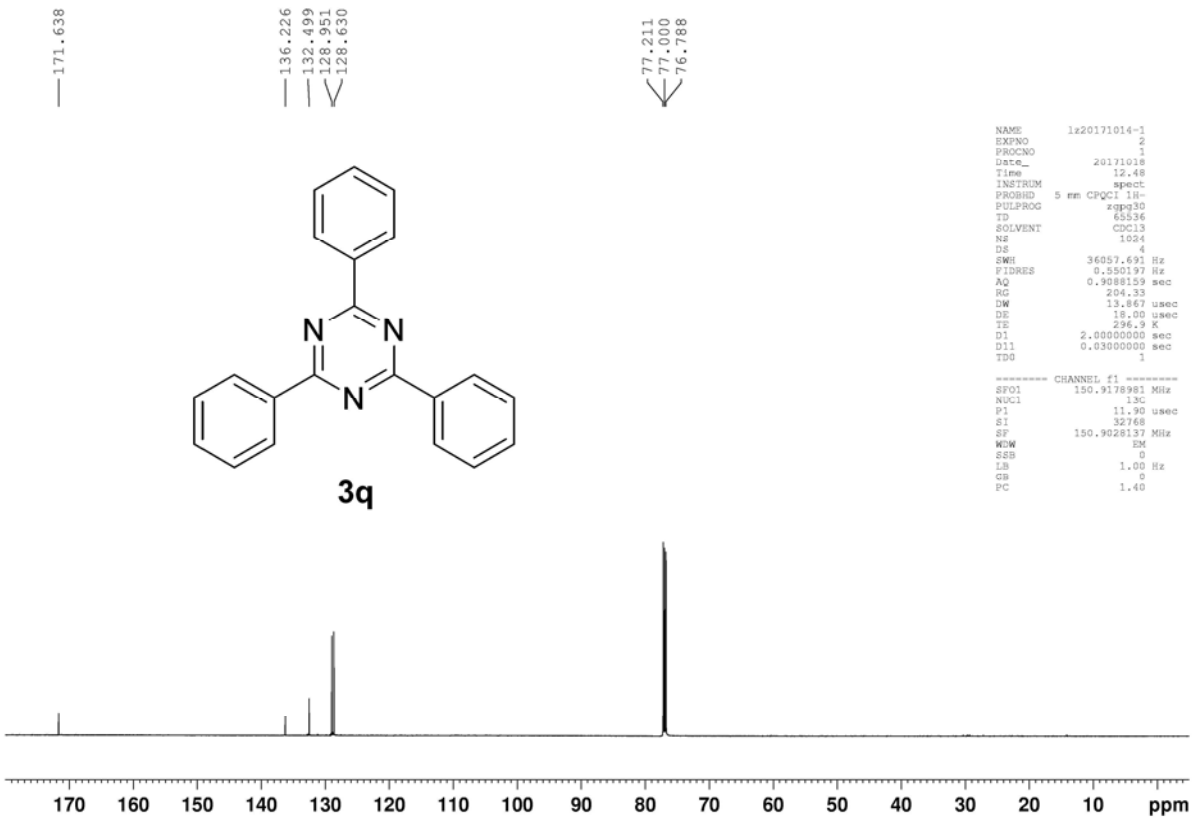

8.699
7.277
2.104
2.096
2.091
2.083
2.075
2.070
2.062
1.216
1.209
1.204
1.202
1.196
1.191
1.141
1.135
1.130
1.128
1.121
1.116
1.109
0.000
3o
9
8
7
6
5
4
3
2
1
0 ppm
1.00
2.16
4.10
4.13

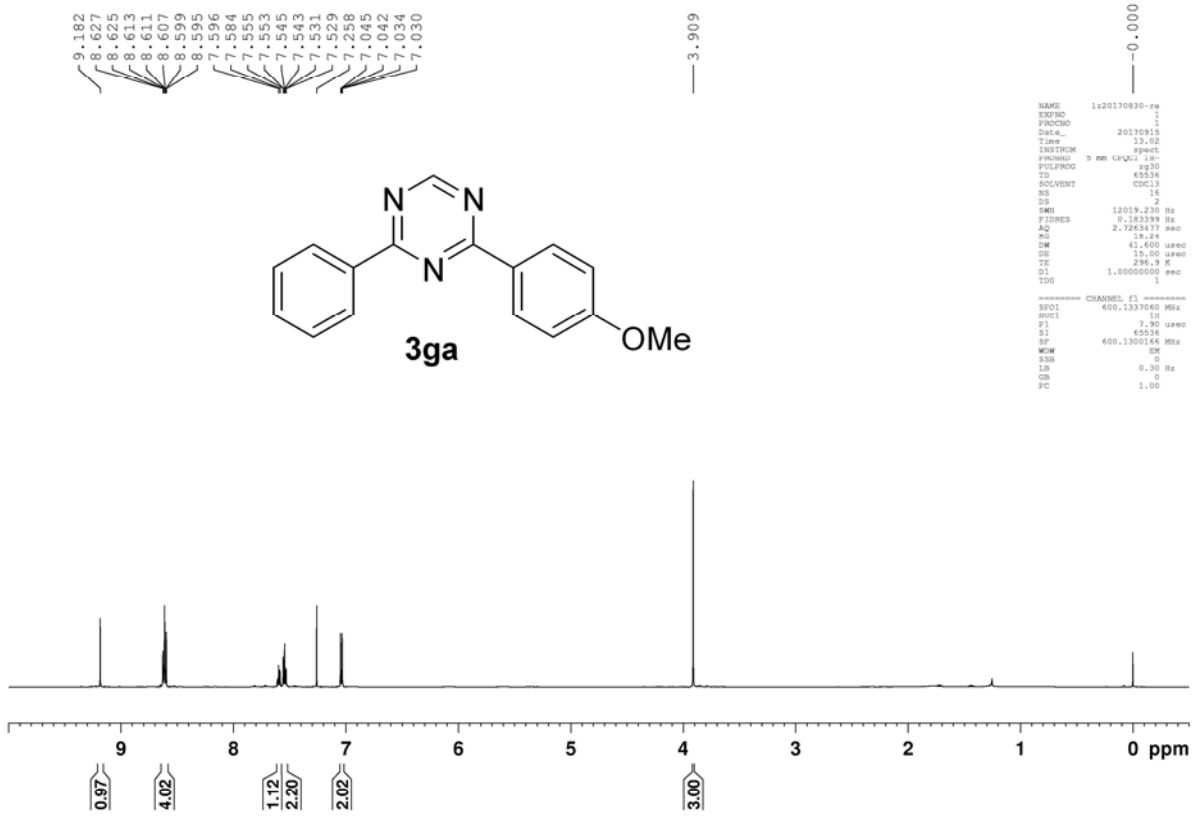

179.608
164.622
77.211
77.000
76.788
17.767
11.897
3o
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

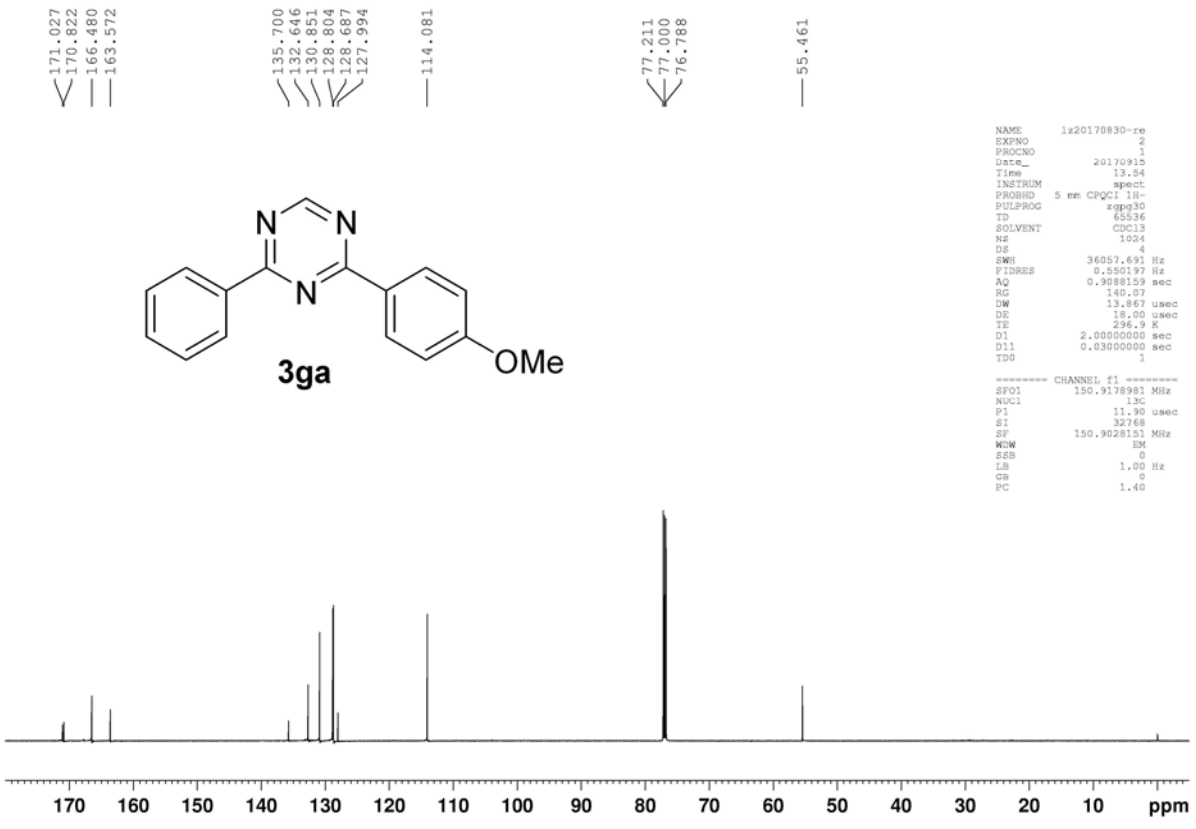

8.648
8.643
8.634
8.631
8.628
7.589
7.581
7.577
7.573
7.567
7.564
7.562
7.541
7.531
7.528
7.519
7.516
7.515
7.244
2.782
0.000
3p
9
8
7
6
5
4
3
2
1
0 ppm
3.97
1.99
4.01
3.00


176.988
171.188
135.866
132.454
128.866
128.610
77.211
77.000
76.788
26.036
3p
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.792
8.780
8.778
7.632
7.625
7.620
7.615
7.610
7.608
7.606
7.594
7.581
7.572
7.570
7.568
7.256
0.000
N
N
N
3q
9
8
7
6
5
4
3
2
1
0 ppm
6.00
9.17


171.638
136.226
132.499
128.951
128.630
77.211
77.000
76.788
N
N
N
3q
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


9.182
8.627
8.625
8.613
8.611
8.607
8.599
8.595
7.596
7.584
7.555
7.553
7.545
7.543
7.531
7.529
7.258
7.045
7.042
7.034
7.030
3.909
0.000
N
N
N
OMe
3ga
NAME 1z20170830-re
EXPNO 1
PROCNO 1
Date_ 20170915
Time 13.02
INSTRUM spect
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 18.24
DW 41.600 usec
DE 15.00 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 7.90 usec
SI 65536
SF 600.1300166 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0 ppm
0.97
4.02
1.12
2.20
2.02
3.00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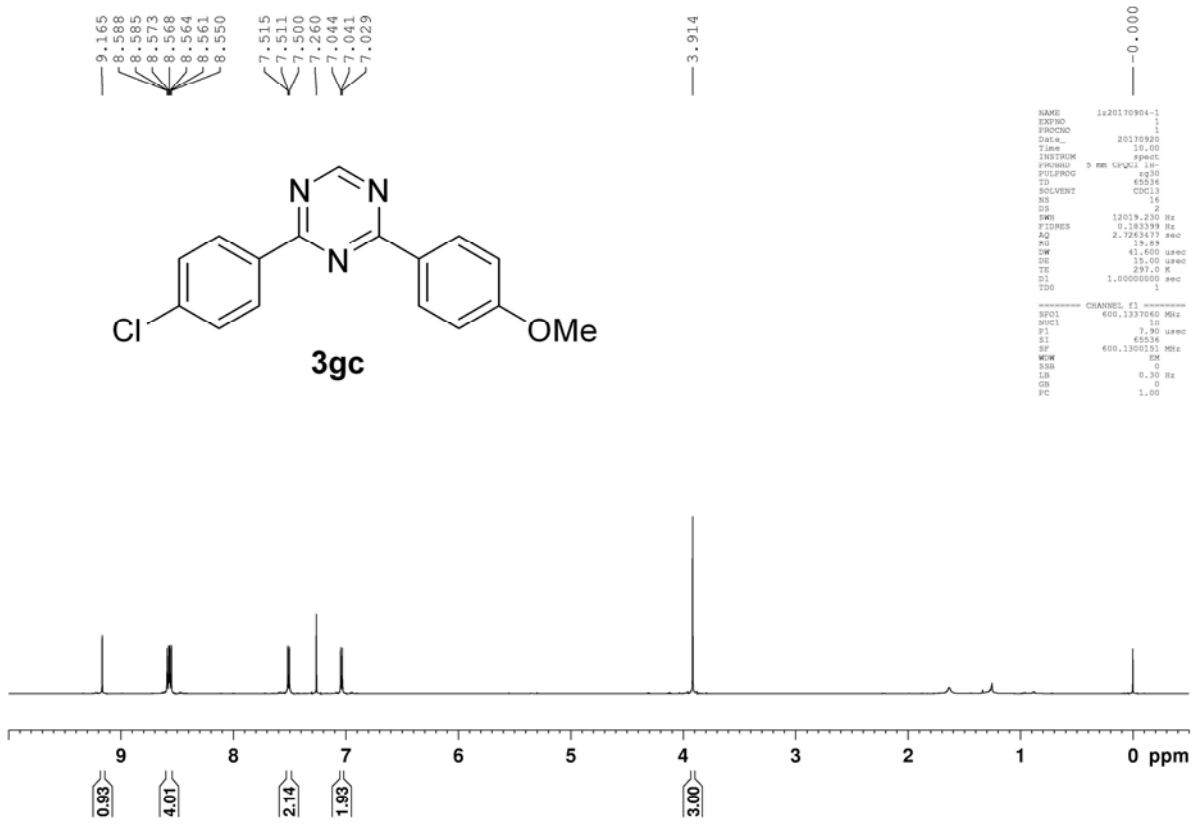
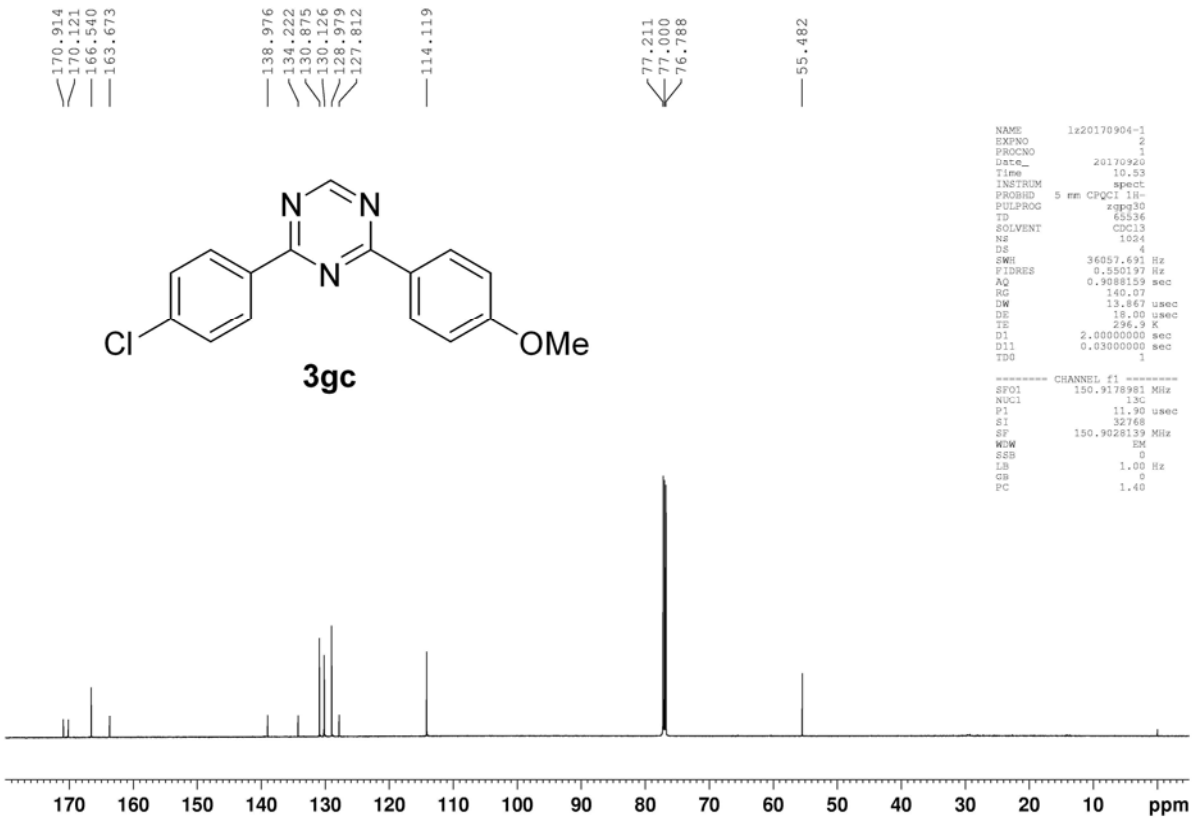
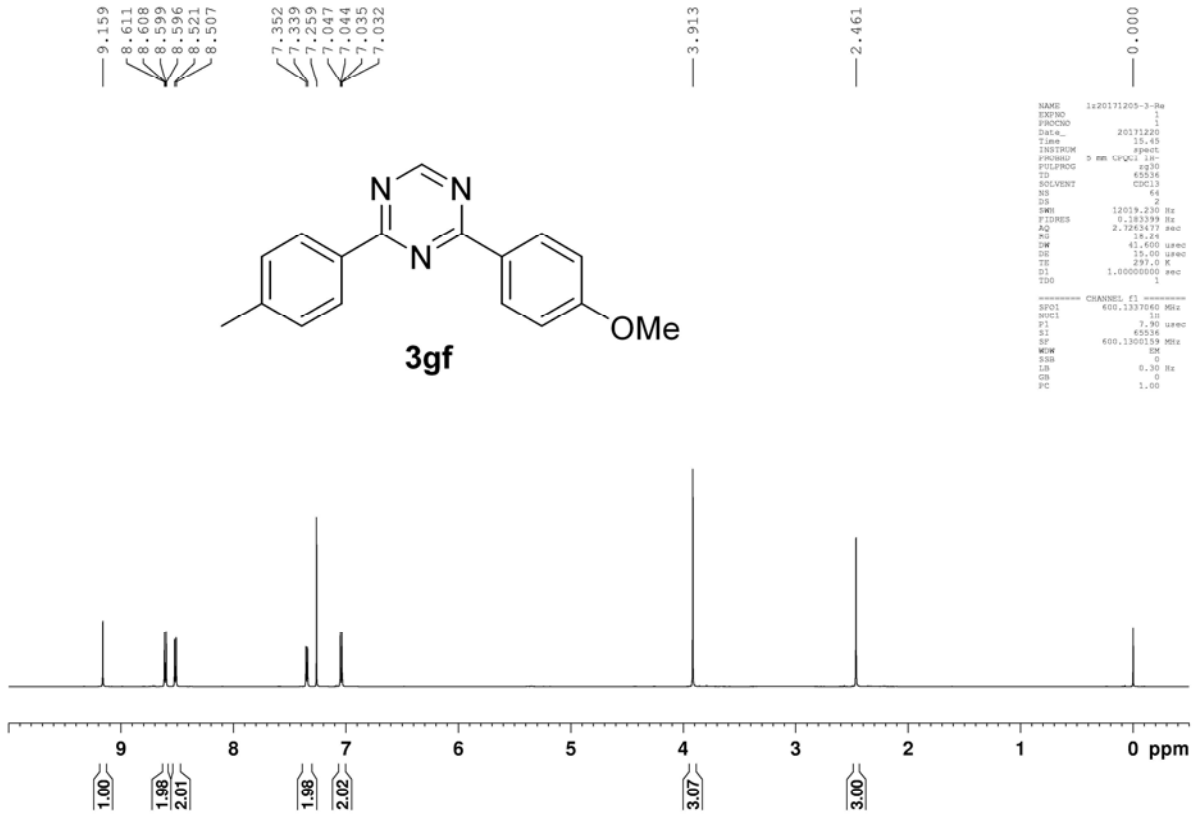
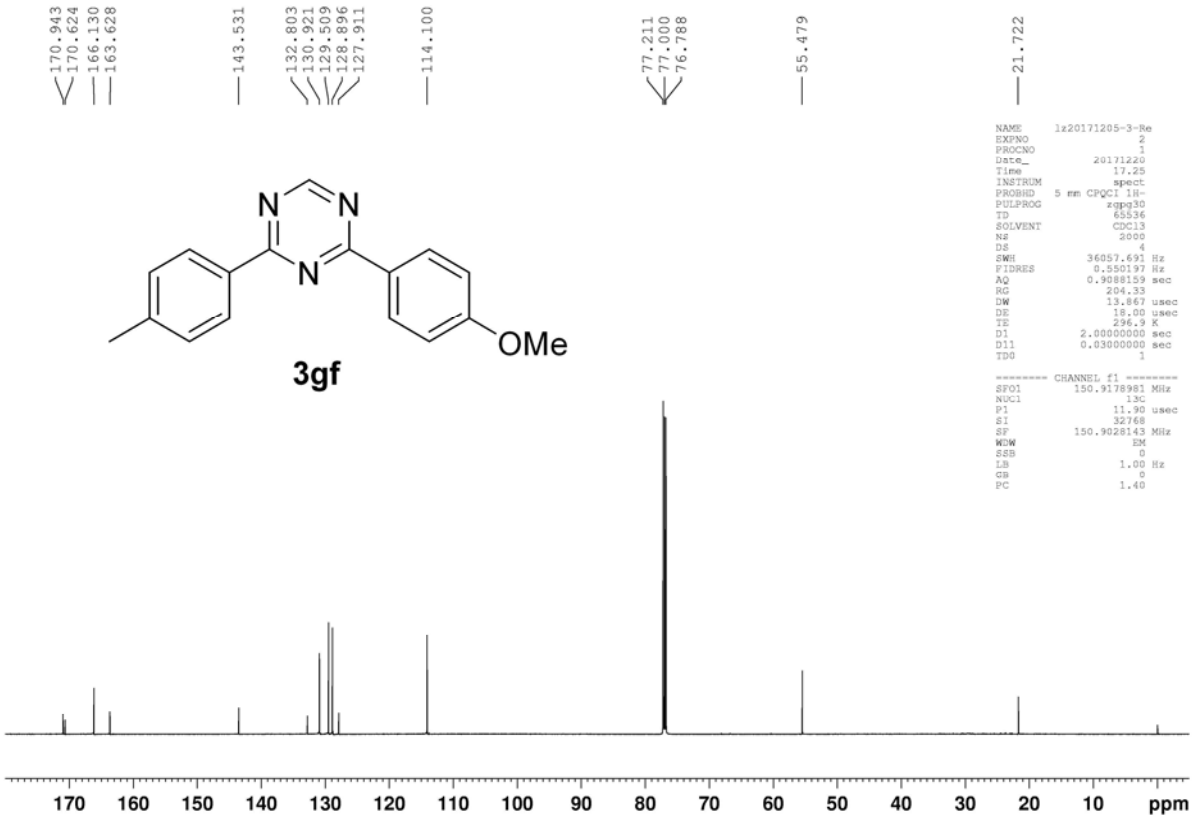
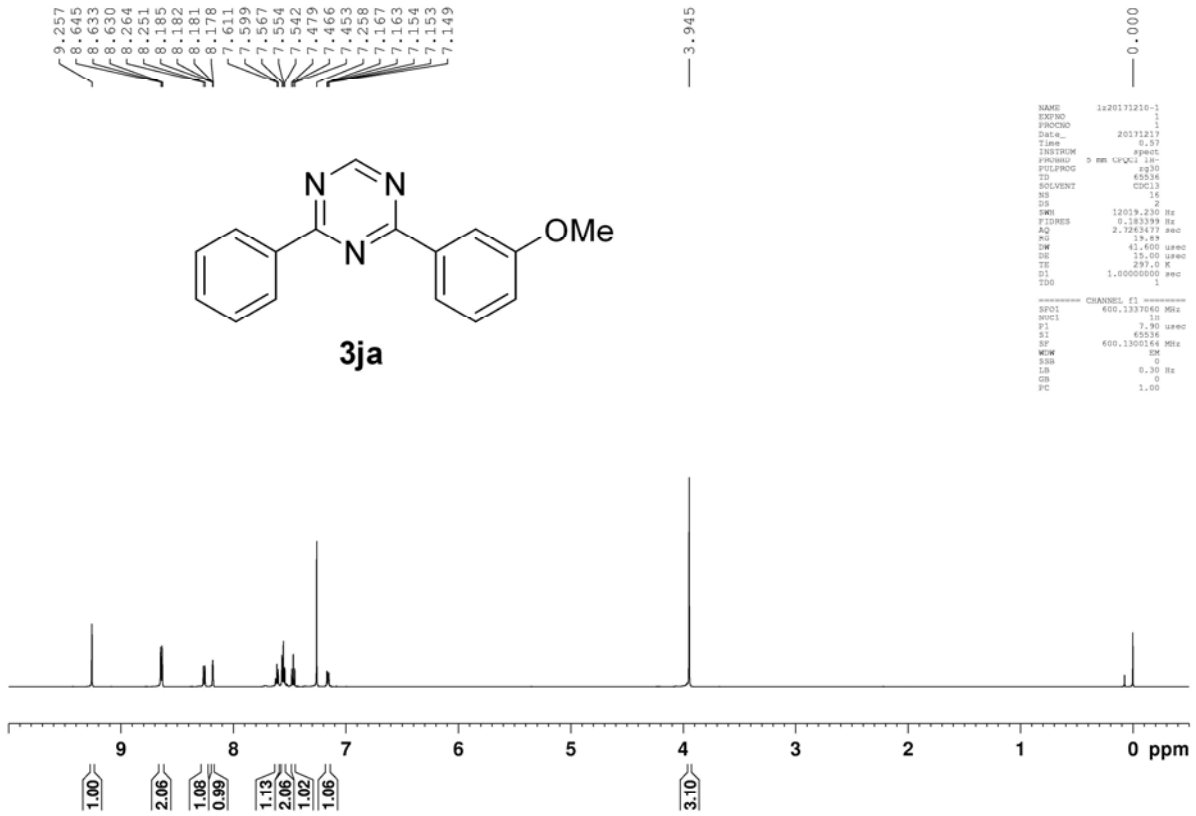
170.822
166.480
163.572
135.700
132.646
130.851
128.804
128.687
127.994
114.081
77.211
77.000
76.788
55.461
N
N
N
OMe
3ga
NAME 1z20170830-re
EXPNO 2
PROCNO 1
Date_ 20170915
Time 13.54
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 140.07
DW 13.867 usec
DE 18.00 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 11.90 usec
SI 32768
SF 150.9028151 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

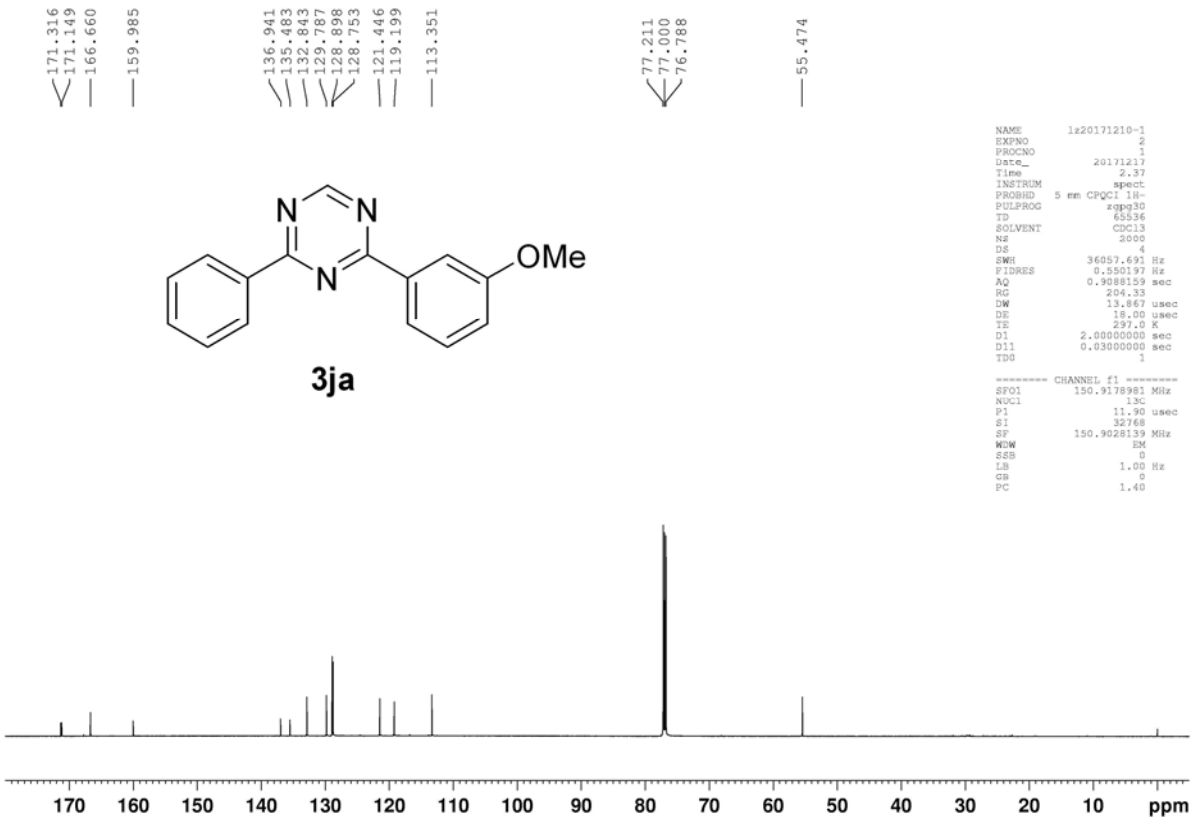

9.165
8.588
8.585
8.573
8.568
8.564
8.561
8.550
7.515
7.511
7.500
7.260
7.044
7.041
7.029
3.914
0.000
Cl
OMe
3gc
9
8
7
6
5
4
3
2
1
0 ppm
0.93
4.01
2.14
1.93
3.00

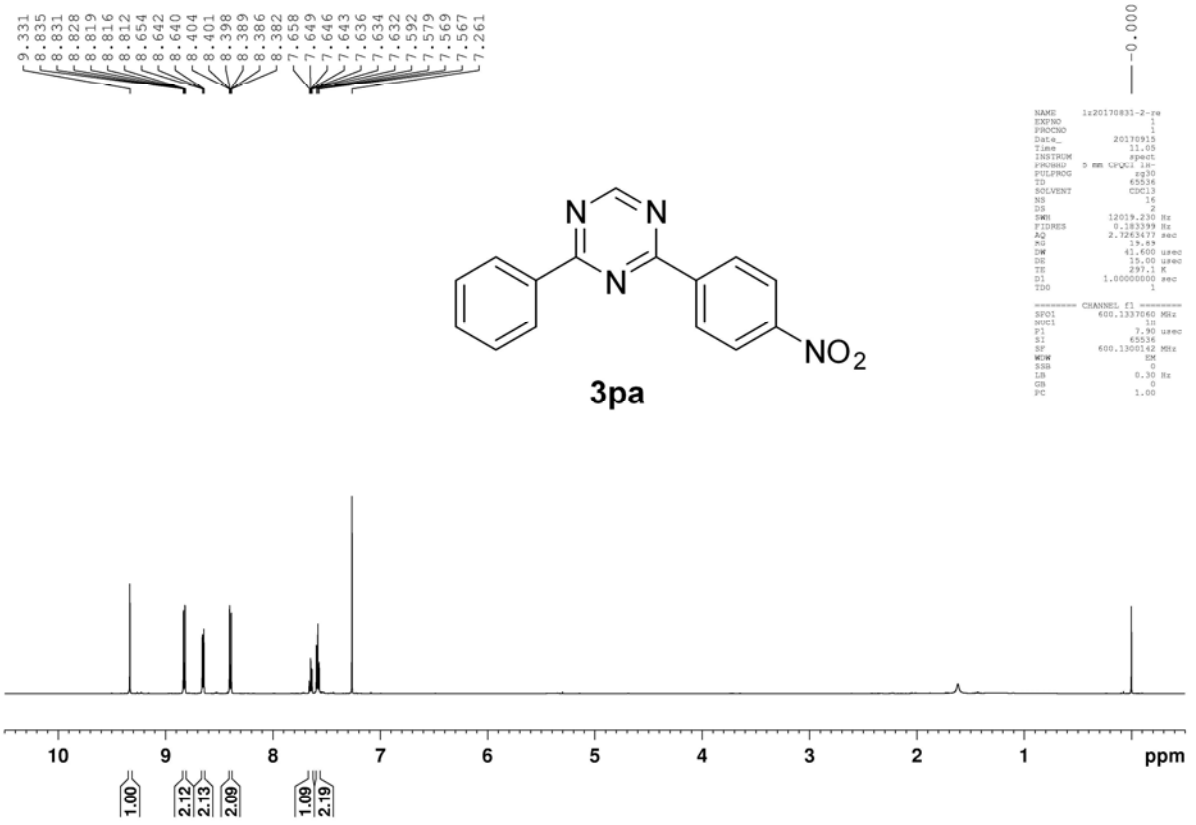

170.914
170.121
166.540
163.673
138.976
134.222
130.875
130.126
128.979
127.812
114.119
77.211
77.000
76.788
55.482
Cl
OMe
3gc
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


9.159
8.611
8.608
8.599
8.596
8.521
8.507
7.352
7.339
7.259
7.047
7.044
7.035
7.032
3.913
2.461
0.000
N
N
N
OMe
3gf
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.98
2.01
1.98
2.02
3.07
3.00


170.943
170.624
166.130
163.628
143.531
132.803
130.921
129.509
128.896
127.911
114.100
77.211
77.000
76.788
55.479
21.722
N
N
N
OMe
3gf
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


9.257
8.645
8.633
8.630
8.264
8.251
8.185
8.182
8.181
8.178
7.611
7.599
7.567
7.554
7.542
7.479
7.466
7.453
7.258
7.167
7.163
7.154
7.153
7.149
3.945
0.000
OMe
3ja
1.00
2.06
1.08
0.99
1.13
2.06
1.02
1.06
3.10
9
8
7
6
5
4
3
2
1
0 ppm


171.316
171.149
166.660
159.985
136.941
135.483
132.843
129.787
128.898
128.753
121.446
119.199
113.351
77.211
77.000
76.788
55.474
OMe
3ja
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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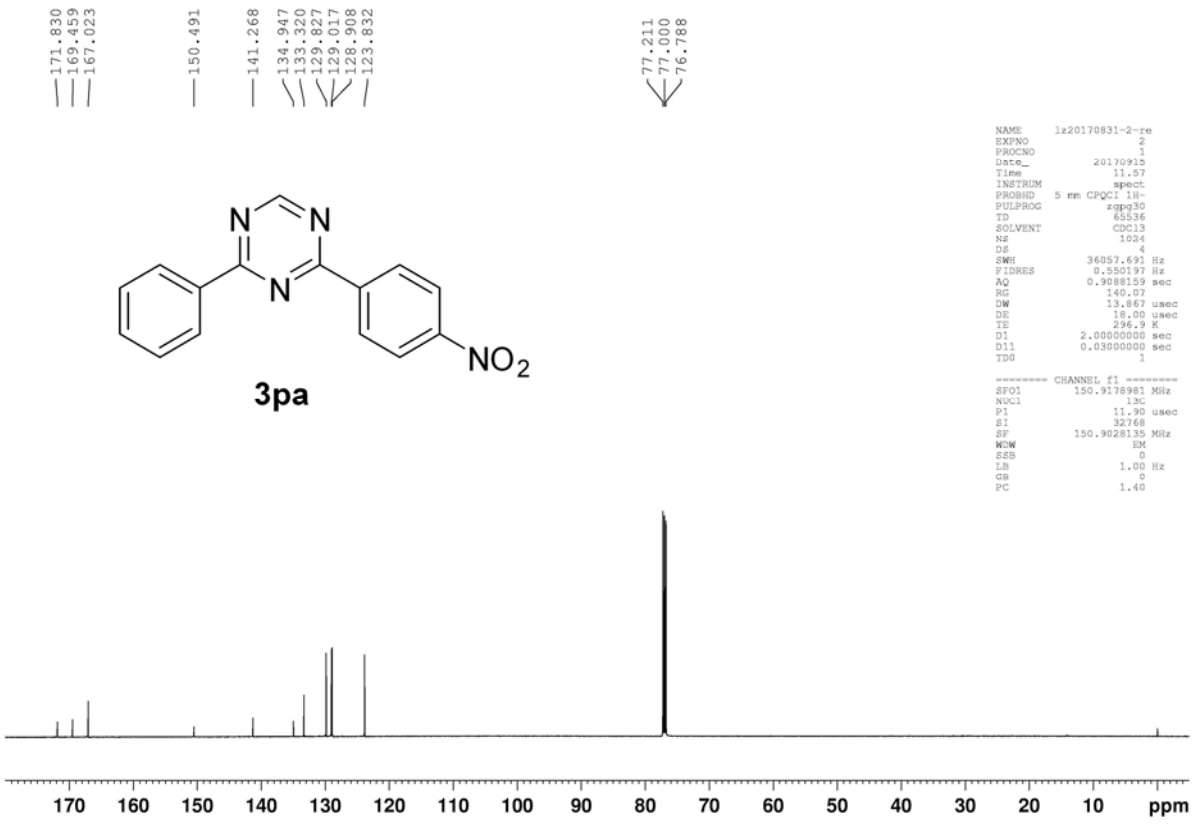
8.831
8.828
8.819
8.816
8.812
8.654
8.642
8.640
8.404
8.401
8.398
8.389
8.386
8.382
7.658
7.649
7.646
7.643
7.636
7.634
7.632
7.592
7.579
7.569
7.567
7.261
-0.000
NAME 1z20170831-2-re
EXPNO 1
PROCNO 1
Date_ 20170915
Time 11.05
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 19.69
DW 41.600 usec
DE 15.00 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 600.1337060 MHz
NUC1 1H
P1 7.90 usec
SI 65536
SF 600.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
N
N
NO2
3pa
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.12
2.13
2.09
1.09
2.19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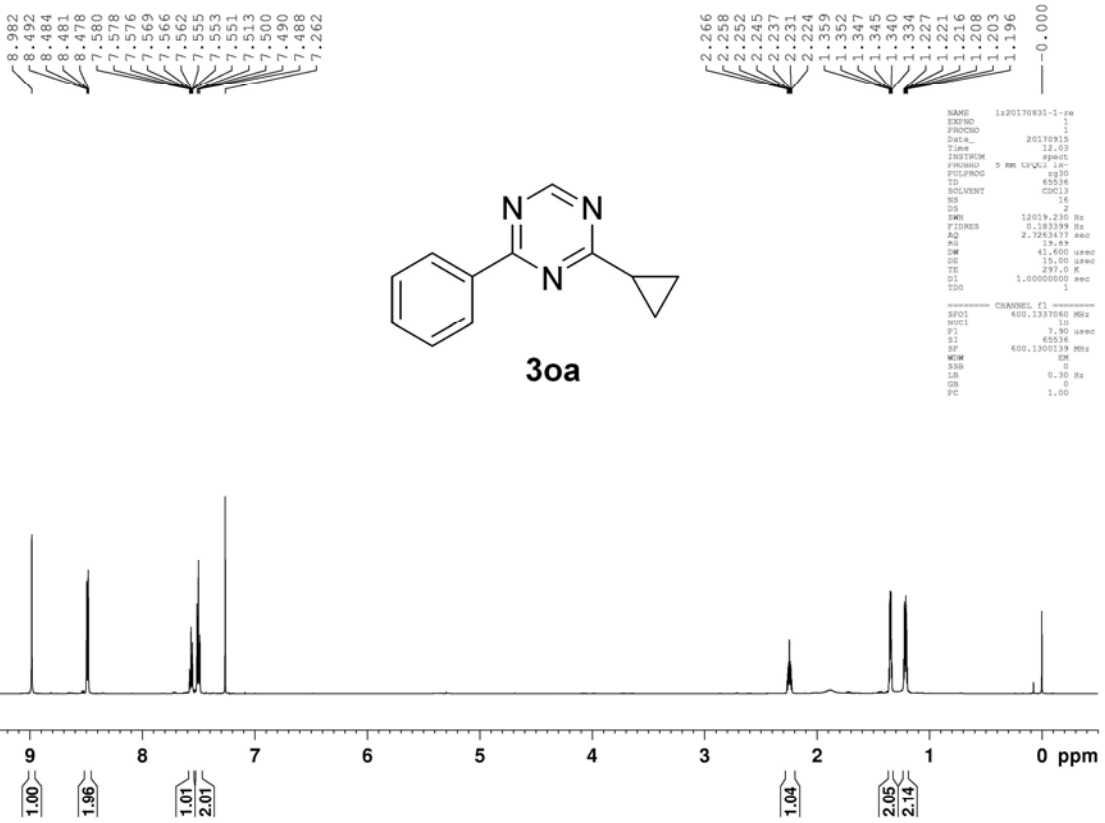
167.023
150.491
141.268
134.947
133.320
129.827
129.017
128.908
123.832
77.211
77.000
76.788
NAME 1z20170831-2-re
EXPNO 2
PROCNO 1
Date_ 20170915
Time 11.57
INSTRUM spect
PROBHD 5 mm CPQCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 140.07
DW 13.867 usec
DE 18.00 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 11.90 usec
SI 32768
SF 150.9028135 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
N
N
NO2
3pa
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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8.481
8.478
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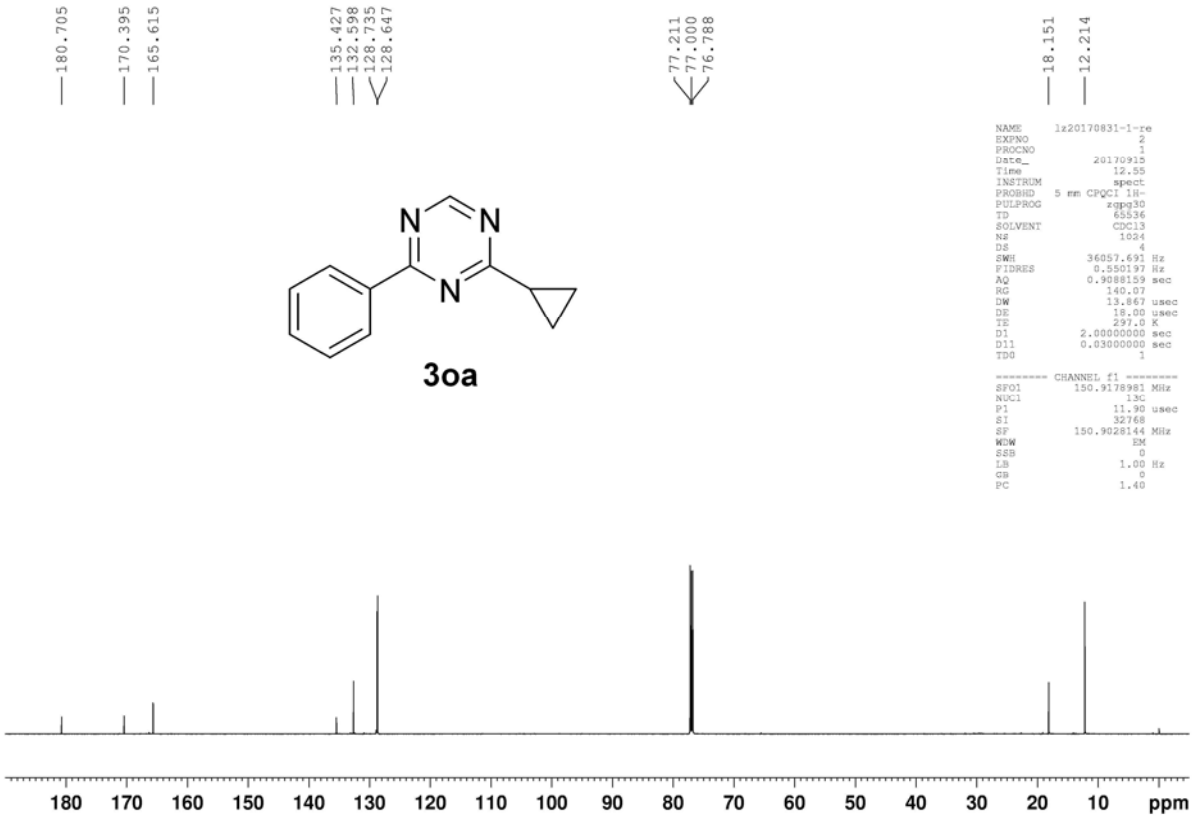
7.578
7.576
7.569
7.566
7.562
7.555
7.553
7.551
7.513
7.500
7.490
7.488
7.262
2.266
2.258
2.252
2.245
2.237
2.231
2.224
1.359
1.352
1.347
1.345
1.340
1.334
1.227
1.221
1.216
1.208
1.203
1.196
0.000
3oa
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.96
1.01
2.01
1.04
2.05
2.14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170.395
165.615
135.427
132.598
128.735
128.647
77.211
77.000
76.788
18.151
12.214
3oa
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm