Supporting Information

An Efficient Synthesis of 2,5-Diimino-furans via Pd-Catalyzed Cyclization of Bromoacrylamides and Isocyanides

Huanfeng Jiang*, Meizhou Yin, Yibiao Li, Biufu Liu, Jinwu Zhao, Wanqing Wu

School of Chemistry and Chemical Engineering, South China University of Technology, Guangzhou 510640, China

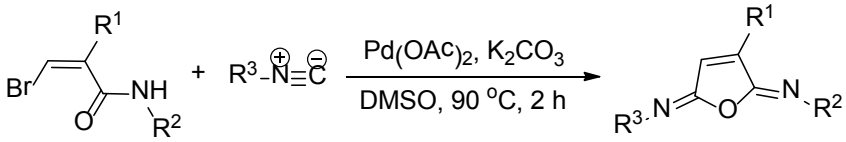
E-mail: jianghf@scut.edu.cn; Fax and Tel.: (+86) 20-87112906

Lists of Contents

General Information

All commercial materials and solvents were used as received without further purification unless otherwise noted. TLC was performed using commercially prepared 200-300 mesh aluminum oxide basic plates and visualization was effected at 254 nm. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ or Acetone-d_6 on BRUKER DRX-400 spectrometer at 298 K. The chemical shift of all ^{1}H and ^{13}C NMR spectra are referenced to the residual signal of $CDCl_3$ (δ 7.26 ppm for the ^{1}H NMR spectra and δ 77.0 ppm for the ^{13}C NMR spectra) or Acetone-d_6 (δ 2.05 ppm for the ^{1}H NMR spectra and δ 206.0 ppm for the ^{13}C NMR spectra). High-resolution mass spectra (ESI) were obtained with a LCMS-IT-TOF mass spectrometer. IR spectra were obtained as potassium bromide pellets or as liquid films between two potassium bromide pellets with a Brucker Vector 22 spectrometer. Melting points were measured with a BÜCHI B-545 melting point instrument and were uncorrected.

General Procedure for the Synthesis of 2,5-Diimino-furans



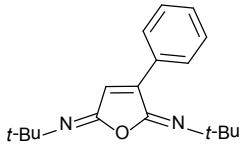
1 **2** **3**

A mixture of bromoacrylamide **1** (0.2 mmol), isocyanide **2** (0.24 mmol), $Pd(OAc)_2$ (5 mol %), K_2CO_3 (0.4 mmol) and DMSO (2 mL) were added successively and stirred at 90 ^{o}C for 2 h in a 10 mL tube. Upon completion, the reaction mixture was extracted with ethyl acetate (3×10 mL), and the organic layers were combined, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure, and the residue was separated by aluminum oxide basic preparative TLC (hexanes/EtOAc 10:1) to give the pure product **3**.

General Procedure for Maleamides

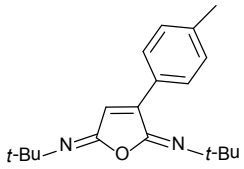
The corresponding 2,5-diimino-furans was stirred with silica gel in methylene dichloride at room temperature overnight. The resulting mixture was filtered and concentrated, and then the crude product was purified by column chromatography on silica gel using petroleum ether as eluent to provide the pure target product **4**.

Analysis Data for Compounds 3a-3x



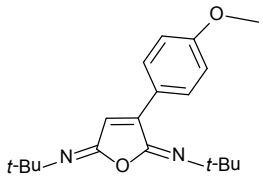
(E)-2,5-di-(*tert*-butylimino)-3-phenyl-furan (3a).

Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.88 (m, 2H), 7.39 (d, J = 4.4 Hz, 3H), 6.83 (s, 1H), 1.45 (s, 9H), 1.43 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.2, 149.9, 143.3, 130.2, 129.8, 128.7, 128.4, 127.4, 55.4, 55.0, 30.3,30.3; IR (KBr): ν_{max} = 2969, 1666, 1361, 1216, 1044, 809 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{18}H_{24}N_2O$ $[M + H]^+$ 285.1961, found 285.1959.



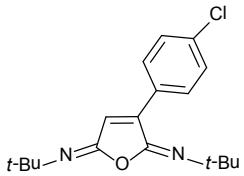
(E)-2,5-di-(*tert*-butylimino)-3-*p*-tolyl-furan (3b).

Yellow solid: m.p. 45-47 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 8 Hz, 2H), 7.19 (d, J = 7.6 Hz, 2H), 6.77 (s, 1H), 2.36 (s, 3H), 1.45 (s, 9H), 1.43 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.0, 150.0, 143.1, 139.8, 129.0, 128.5, 127.4, 126.5, 55.2, 54.8, 30.3, 30.3, 21.3; IR (KBr): ν_{max} = 2969, 1667, 1361, 1215, 1074, 818 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{19}H_{26}N_2O$ $[M + H]^+$ 299.2118, found 299.2123.



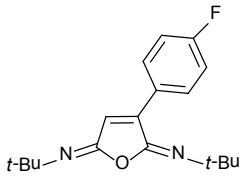
(E)-2,5-di-(*tert*-butylimino)-3-(4-methoxylphenyl)-furan (3c).

Yellow solid: m.p. 138-140 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.8 Hz, 2H), 6.88 (d, J = 8.8 Hz, 2H), 6.69 (s, 1H), 3.79 (s, 3H), 1.42 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.8, 152.1, 150.2, 142.5, 130.1, 125.2, 122.8, 113.7, 55.1, 54.7, 30.3,30.2; IR (KBr): ν_{max} = 2966, 2926, 1649, 1360, 1214, 1032, 892 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{19}H_{26}N_2O_2$ $[M + H]^+$ 315.2067 , found 315.2072.



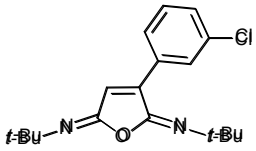
(E)-2,5-di-(*tert*-butylimino)-3-(4-chlorophenyl)-furan (3d).

Yellow solid: m.p. 125-128 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 8.8 Hz, 2H), 7.34 (d, J = 8.8 Hz, 2H), 6.78 (s, 1H), 1.42 (s, 9H), 1.41 (s, 9H); 13C NMR (100 MHz, $CDCl_3$) δ 151.5, 149.7, 141.8, 135.7, 129.8, 128.6, 128.5, 127.5, 55.3, 55.0, 30.2; IR (KBr): ν_{max} = 2969, 1666, 1488, 1361, 1214, 1073, 830 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{18}H_{23}ClN_2O$ $[M + H]^+$ 319.1572 , found 319.1574.



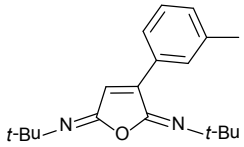
(E)-2,5-di-(*tert*-butylimino)-3-(4-fluorophenyl)-furan (3e).

Yellow solid: m.p. 131-135 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 – 7.88 (m, 2H), 7.06 (t, J = 8.8 Hz, 2H), 6.75 (s, 1H), 1.43 (s, 9H), 1.41 (s, 9H); 13C NMR (100 MHz, $CDCl_3$) δ 164.8, 162.3, 151.6, 145.9 (d, J=796.4 Hz), 130.6 (d, J = 8.3 Hz), 127.0(d, J = 1.5 Hz), 126.3 (d, J = 3.4 Hz), 115.37 (d, J=21.5 Hz), 55.3, 54.9, 30. 3; IR (KBr): ν_{max} = 2973, 1667, 1506, 1363, 1237, 836 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{18}H_{23}FN_2O$ $[M + H]^+$ 303.1867 , found 303.1860.



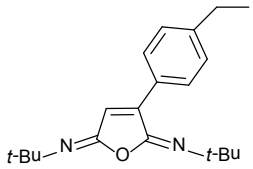
(E)-2,5-di-(*tert*-butylimino)-3-(3-chlorophenyl)-furan (3f).

Yellow solid: m.p. 128-131 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.22 (d, J = 0.8 Hz, 1H), 8.03 – 8.00 (m, 1H), 7.45 – 7.44 (m, 2H), 7.11 (s, 1H), 1.45 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, Acetone-d_6) δ 151.0, 150.8, 141.8, 134.5, 133.1, 130.7, 130.3, 129.9, 129.5, 127.9, 55.8, 55.5, 30.7, 30.6; IR (KBr): ν_{max} = 2970, 1666, 1361, 1213, 1075, 789 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{18}H_{23}ClN_2O$ $[M + H]^+$ 319.1572 , found 319.1574.



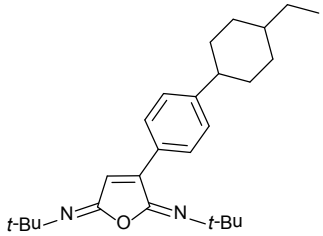
(E)-2,5-di-(*tert*-butylimino)-3-*m*-tolyl-furan (3g).

Brown solid: m.p. 57-60 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 7.89 (d, J = 7.6 Hz, 2H), 7.30 (t, J = 7.8 Hz, 1H), 7.24 (d, J = 7.6 Hz, 1H), 6.96 (s, 1H), 2.36 (s, 3H), 1.44 (s, 9H), 1.40 (s, 9H); ^{13}C NMR (100 MHz, Acetone-d_6) δ 151.4, 151.0, 143.5, 138.4, 131.0, 130.0, 128.8, 128.4, 126.7, 55.6, 55.2, 30.6, 30.5, 21.4; IR (KBr): ν_{max} = 2969, 1666, 1361, 1239, 1070, 834, 760 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{19}H_{26}N_2O$ $[M + H]^+$ 299.2118, found 299.2116.



(E)-2,5-di-(*tert*-butylimino)-3-(4-ethylphenyl)-furan (3h).

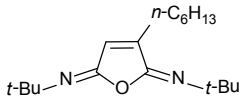
Yellowish solid: m.p. 53-55 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.00 (d, J = 8.4 Hz, 2H), 7.26 (d, J = 8.4 Hz, 2H), 6.92 (s, 1H), 2.69 – 2.63 (m, 2H), 1.44 (s, 9H), 1.40 (s, 9H), 1.23 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, Acetone-d_6) δ 151.5, 151.1, 146.8, 143.4, 129.6, 128.6, 128.5, 127.7, 55.6, 55.2, 41.2, 30.7, 30.6, 15.7; IR (KBr): ν_{max} = 2966, 1664, 1360, 1215, 1076, 877 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{26}H_{38}N_2O$ $[M + H]^+$ 313.2274, found 313.2279.



(E)-2,5-di-(*tert*-butylimino)-3-[4-(4-ethylcyclohexyl)-phenyl]-furan (3i).

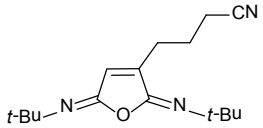
Yellow solid: m.p. 49-50 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.01 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 6.92 (s, 1H), 2.52 (t, J = 12.0 Hz, 1H), 1.88 (d, J = 10.4 Hz, 4H), 1.56 – 1.49 (m, 2H), 1.44 (s, 9H), 1.40 (s, 9H), 1.31 – 1.24 (m, 2H), 1.20 (t, J = 7.2 Hz, 1H), 1.12 – 1.03 (m, 2H), 0.91 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, Acetone-d_6) δ151. 5, 151.2, 150.4, 143.5, 129.6, 128.8, 127.8, 127.5, 55.6, 55.3, 45.2, 39.8, 34.8, 33.8, 30.7, 30.6, 11.7; IR (KBr): ν_{max} = 2966,

1666, 1360, 1215, 1073, 893, 830 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{26}H_{38}N_2O$ $[M + H]^+$ 395.3057, found 395.3059.



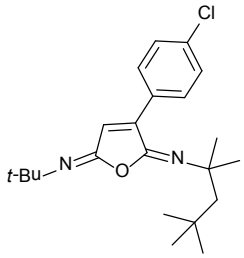
(E)-2,5-di-(*tert*-butylimino)-3-hexyl-furan (3j).

Colorless oil: ^1H NMR (400 MHz, $CDCl_3$) δ 6.28 (s, 1H), 2.33 (t, J = 7.5 Hz, 2H), 1.62 – 1.46 (m, 2H), 1.37 (s, 9H), 1.36 (s, 9H), 1.32 – 1.22 (m, 6H), 0.88 (t, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 152.2, 148.7 , 127.2, 54.6, 54.6, 31.5, 30.4, 30.3, 27.2, 25.8, 22.5, 14.0; IR (KBr): ν_{max} = 2968, 2869, 1666, 1362, 1233, 1013, 914, 874 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{18}H_{32}N_2O$ $[M + H]^+$ 293.2587, found 293.2586.



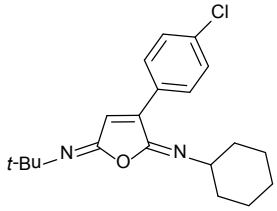
(E)-2,5-di-(*tert*-butylimino)-3-butyronitrile-furan (3k).

Brown solid: m.p. 89-93 ^oC; ^1H NMR (400 MHz, Acetone-d_6) δ 6.47 (t, J = 1.4 Hz, 1H), 2.57 – 2.49 (m, 4H), 2.02 – 1.94 (m, 2H), 1.37 (s, 9H), 1.35 (s, 9H); ^{13}C NMR (100 MHz, Acetone-d_6) δ152.7, 151.7, 147.0, 129.3, 120.2, 55.1, 55.1, 30.7, 30.6, 25.5, 24.2, 16.8; IR (KBr): ν_{max} = 2969, 2872, 2247, 1666, 1361, 1216, 1012 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{16}H_{25}N_3O$ $[M + H]^+$ 276.2070, found 276.2067.



(E)-2-(2,4,4-trimethylpentan-2-ylimino) -5-(*tert*-butylimino) -3-(4-chlorophenyl)-furan (3l).

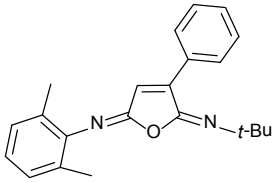
Yellow solid: m.p. 90-93 ^oC; ^1H NMR (400 MHz, Acetone-d_6) δ 8.15 (d, J = 8.8 Hz, 2H), 7.47 (d, J = 8.8 Hz, 2H), 7.06 (s, 1H), 1.87 (s, 2H), 1.50 (s, 6H), 1.41 (s, 9H), 0.99 (s, 9H); ^{13}C NMR (100 MHz, Acetone-d_6) δ151.2, 150.1, 142.1, 135.9, 131.2, 129.9, 129.2, 129.1, 59.5, 55.6, 55.3, 32.5, 32.0, 31.2, 30.6; IR (KBr): ν_{max} = 2966, 1646, 1345, 1293, 1170, 830 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{22}H_{31}ClN_2O$ $[M + H]^+$ 375.2198, found 375.2199.



(E)-2-cyclohexylimino -5-(*tert*-butylimino) -3-(4-chlorophenyl)-furan (3m).

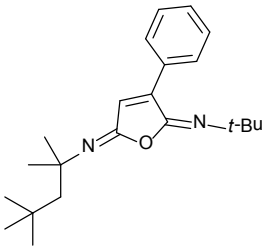
Yellow solid: m.p. 79-81 ^oC; ^1H NMR (400 MHz, Acetone-d_6) δ 8.17 (d, J = 8.8 Hz, 2H), 7.48 (d, J = 8.8 Hz, 2H), 7.12 (s, 1H), 2.81 (s, 1H), 1.86 – 1.80 (m, 4H), 1.63 – 1.40 (m, 6H), 1.38 (s, 9H); ^{13}C NMR (100 MHz, Acetone-d_6) δ153.5, 151.3, 141.3, 136.1, 131.1, 129.8, 129.7, 129.3,

58.0, 55.8, 34.5, 30.5, 26.5, 25.0; IR (KBr): ν_{max} = 2966, 1665, 1359, 1217, 1077, 769 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{20}H_{25}ClN_2O$ $[M + H]^+$ 345.1728, found 345.1725.



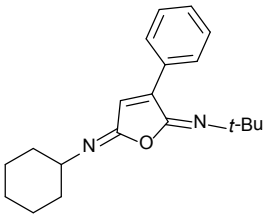
(E)-2-(*tert*-butylimino)-5-(2,6-dimethylphenylimino)-3-phenyl-furan (3n).

Brown oil; ^1H NMR (400 MHz, Acetone-d_6) δ 8.04 – 7.95 (m, 2H), 7.28 (d, J = 19.2, 4H), 6.89 (d, J = 7.2 Hz, 2H), 6.76 (t, J = 7.6 Hz, 1H), 1.95 (s, 6H), 1.06 (s, 9H); ^{13}C NMR (101 MHz, Acetone-d_6) δ 154.3, 150.4, 146.1, 146.1, 131.0, 130.8, 129.8, 129.1, 128.2, 127.72, 126.0, 124.2, 56.3, 30.3, 30.3, 18.3; IR (KBr): ν_{max} = 2925, 1669, 1383, 1222, 1078, 947, 768 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{22}H_{24}N_2O$ $[M + H]^+$ 333.1961, found 333.1960.



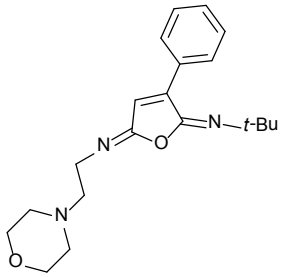
(E)-2-(*tert*-butylimino)-5-(2,4,4-trimethylpentan-2-ylimino)-3-phenyl-furan (3p).

Yellow solid: m.p. 78-82 ^oC; ^1H NMR (400 MHz, Acetone-d_6) δ 8.12 – 8.10 (m, 2H), 7.43 – 7.41 (m, 3H), 7.00 (s, 1H), 1.82 (s, 2H), 1.46 (s, 15H), 1.01 (s, 9H); ^{13}C NMR (101 MHz, Acetone-d_6) δ151.2, 150.5, 143.4, 131.2, 130.4, 129.6, 129.0, 128.7, 59.1, 55.7, 32.6, 32.0, 31.5, 30.7, 29.8; IR (KBr): ν_{max} = 2962, 1668, 1361, 1217, 1075, 942, 890, 769 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{22}H_{32}N_2O$ $[M + H]^+$ 341.2587, found 341.2585.



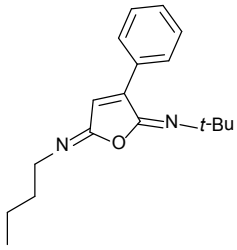
(E)-2-(*tert*-butylimino)-5-cyclohexylimino-3-phenyl-furan (3q).

White solid: ^1H NMR (400 MHz, Acetone-d_6) δ 8.11 – 8.09 (m, 2H), 7.43 – 7.42 (m, 3H), 7.07 (s, 1H), 3.86 – 3.72 (m, 1H), 1.82 – 1.78 (m, 4H), 1.51 – 1.36 (m, 15H); ^{13}C NMR (100 MHz, Acetone-d_6) δ153.6, 151.0, 144.4, 131.1, 130.4, 129.4, 128.9, 127.0, 57.8, 55.9, 34.5, 30.4, 26.3, 25.0; IR (KBr): ν_{max} = 2929, 2856, 1765, 1666, 1378, 1242, 1058, 893 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{20}H_{26}N_2O$ $[M + H]^+$ 311.2118, found 311.2115.



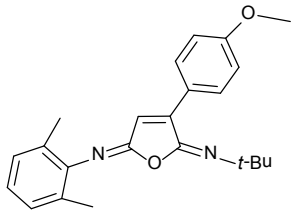
(E)-2-(*tert*-butylimino)-5-[(2-morpholinoethyl)imino]-3-phenyl-furan (3r).

White solid: m.p. 143-146 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 7.56 – 7.54 (m, 2H), 7.37 (d, J = 6.4 Hz, 3H), 6.29 (s, 1H), 3.61 (t, J = 4.4, 4H), 3.40 – 3.36 (m, 2H), 2.49 – 2.43 (m, 6H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, Acetone-d_6) δ 168.1, 165.1, 147.5, 137.5, 129.6, 129.3, 127.3, 121.3, 67.3, 58.3, 54.3, 51.9, 36.8, 28.8; IR (KBr): ν_{max} = 2991, 1764, 1376, 1242, 1056 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{20}H_{27}N_3O_2$ $[M + H]^+$ 342.2176, found 342.2168.



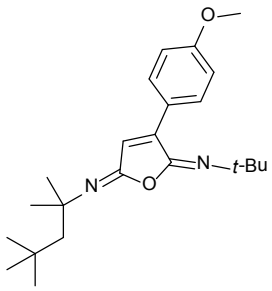
(E)-2-(*tert*-butylimino)-5-butylimino-3-phenyl-furan (3s).

Yellow solid: m.p. 61-63 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.12- 8.01 (m, 2H), 7.43 (t, J = 3.0, 3H), 7.09 (s, 1H), 3.57 (t, J = 6.8 Hz, 2H), 1.66 – 1.62 (m, 2H), 1.46 – 1.39 (m, 11H), 0.95 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, Acetone-d_6) δ 155.0, 151.0, 144.7, 131.2, 130.5, 129.5, 129.0, 127.0, 56.0, 49.0, 33.6,30.4, 21.2, 14.1; IR (KBr): ν_{max} = 2960, 2930, 2866, 1666, 1359, 1220, 1086, 952, 811 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{18}H_{24}N_2O$ $[M + H]^+$ 285.1961, found 285.1959.

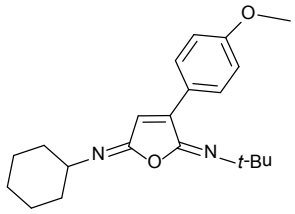


(E)-2-(*tert*-butylimino)-5-(2,6-dimethylphenylimino) -3-(4-methoxylphenyl)-furan (3t).

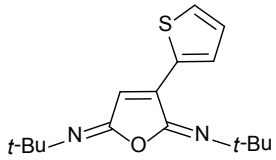
Brown solid: 54-57 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.22 (d, J = 8.8 Hz, 2H), 7.30 (s, 1H), 7.03 (t, J = 8.0 Hz, 4H), 6.90 (t, J = 7.4 Hz, 1H), 3.87 (s, 3H), 2.09 (s, 6H), 1.20 (s, 9H); ^{13}C NMR (101 MHz, Acetone-d_6) δ162.3, 154.4, 150.7, 146.2, 145.5, 131.4, 128.1, 127.7, 124.0, 123.4, 123.2, 114.5, 56.2, 55.6, 30.1, 18.2; IR (KBr): ν_{max} = 2984, 2828, 1766, 1667, 1380, 1244, 1063 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{23}H_{26}N_2O_2$ $[M + Na]^+$ 385.1886, found 385.1878.



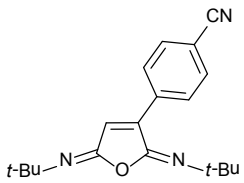
(E)-2-(*tert*-butylimino)-5-(2,4,4-trimethylpentan-2-ylimino)-3-(4-methoxylphenyl)-furan (3u). Yellow solid: m.p. 49-53 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 9.2 Hz, 2H), 6.92 (d, J = 8.8 Hz, 2H), 6.71 (s, 1H), 3.83 (s, 3H), 1.83 (s, 2H), 1.46 (s, 6H), 1.45 (s, 9H), 0.99 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.8, 151.0, 150.4, 142.4, 130.2, 125.4, 122.9, 113.8, 58.6, 55.3, 55.2, 54.8, 32.0, 31.7, 31.2, 30.4; IR (KBr): ν_{max} = 2959, 2361, 1667, 1509, 1243, 1059, 832 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{23}H_{34}N_2O_2$ $[M + H]^+$ 371.2693, found 371.2691.



(E)-2-(*tert*-butylimino)-5-cyclohexylimino-3-(4-methoxylphenyl)-furan (3v).
White solid: m.p. 126-128 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 8.8 Hz, 2H), 6.92 (d, J = 8.8 Hz, 2H), 6.76 (s, 1H), 3.83 (s, 3H), 3.77 – 3.65 (m, 1H), 1.86-1.79 (m, 4H), 1.68 – 1.58 (m, 2H), 1.49-1.44 (m, 2H), 1.42 (s, 9H), 1.27-1.24 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.0, 154.4, 150.3, 143.7, 130.2, 123.6, 122.8, 113.8, 57.3, 55.6, 55.3, 34.0, 30.2, 25.7, 24.8; IR (KBr): ν_{max} = 2928, 2855, 2362, 1663, 1509, 1251, 1179, 831 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{21}H_{28}N_2O_2$ $[M + H]^+$ 341.2224, found 341.2225.

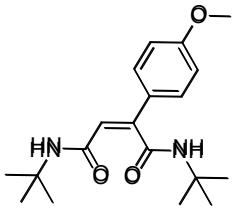


(E)-2-(*tert*-butylimino)-5-cyclohexylimino-3-(4-methoxylphenyl)-furan (3w).
Yellow oil; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.53-8.52 (m, 1H), 7.68-7.66 (m, 1H), 7.55-7.53 (m, 1H), 6.93 (s, 1H), 1.45 (s, 9H), 1.39 (s, 9H); ^{13}C NMR (100 MHz, Acetone-d_6) δ 151.58, 150.65, 138.21, 131.51, 128.42, 127.58, 126.30, 126.02, 55.20, 54.96, 30.23, 30.11; IR (KBr): ν_{max} = 2967, 2926, 1664, 1361, 1218, 1028, 803 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{16}H_{22}N_2OS$ $[M + H]^+$ 291.1526, found 291.1525.



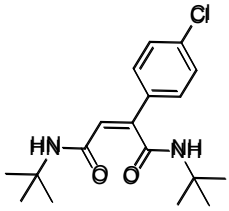
(E)-2,5-di-(*tert*-butylimino)-3-(4-cyanophenyl)-furan (3x).

White solid: m.p. 116-123 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 7.79 (d, J = 8.0 Hz, 2H), 7.70 (d, J = 7.2 Hz, 2H), 6.38 (s, 1H), 1.43 (s, 9H), 1.38 (s, 9H); ^{13}C NMR (100 MHz, Acetone-d_6) δ 164.42, 150.51, 142.51, 135.45, 133.08, 130.28, 128.22, 119.00, 112.78, 55.83, 52.03, 30.46, 28.78; IR (KBr): ν_{max} = 3856, 2924, 2369, 1655, 1361, 1220, 1095, 801 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{19}H_{23}N_3O$ $[M + H]^+$ 310.1914, found 310.1910.



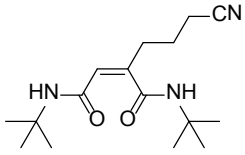
N^1-(*tert*-butyl)-N^4-(*tert*-butyl)-2-(4-methoxyphenyl)maleamide (4a).

White solid: m.p. 162-175 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, J = 8.8 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 6.83 (s 1H), 6.07 (s, 1H), 5.76 (s, 1H), 3.80 (s, 3H), 1.40 (s, 9H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.54, 165.52, 160.54, 142.91, 127.94, 127.91, 122.37, 114.30, 55.33, 52.29, 51.56, 28.64, 28.61; IR (KBr): ν_{max} = 3452, 2362, 2335, 1645, 1454, 1041, 675 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{19}H_{28}N_2O_3$ $[M + Na]^+$ 355.1992, found 355.1990.



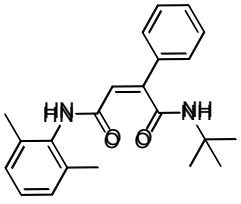
N^1-(*tert*-butyl)-N^4-(*tert*-butyl)-2-(4-chlorophenyl)maleamide (4b).

White solid: m.p. 189-200 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.30 (m, 4H), 6.69 (s, 1H), 6.11 (s, 1H), 5.92 (s, 1H), 1.39 (s, 9H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.66, 165.02, 142.31, 135.35, 134.21, 129.09, 127.80, 124.50, 52.40, 51.75, 28.60, 28.58; IR (KBr): ν_{max} = 3462, 2967, 2362, 2335, 1644, 1543, 1244, 1047, 675 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{18}H_{25}ClN_2O_2$ $[M + Na]^+$ 359.1497, found 359.1494.



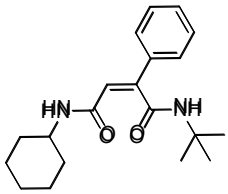
N^1-(*tert*-butyl)-N^4-(*tert*-butyl)-2-(butyronitrile)maleamide (4c).

White solid: m.p. 123-137 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (s, 1H), 6.17 (s, 1H), 5.80 (s, 1H), 2.41 – 2.34 (m, 4H), 1.83-1.76 (m, 2H), 1.36 (s, 9H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.89, 165.57, 143.54, 125.53, 119.30, 51.75, 34.31, 28.50, 28.49, 23.55, 16.37; IR (KBr): ν_{max} = 3318, 2970, 1660, 1538, 1454, 1224 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{16}H_{27}N_3O_2$ $[M + Na]^+$ 316.1995, found 316.1994.



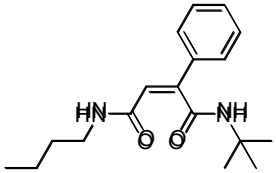
N^1-(*tert*-butyl)-N^4-(2,6-dimethylphenyl)-2-phenylmaleamide (4d).

White solid: m.p. 197-202 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (s, 1H), 7.49 – 7.48 (m, 2H), 7.39-7.38 (m, 3H), 7.07 – 7.05 (m, 3H), 6.39 (s, 1H), 5.75 (s, 1H), 2.24 (s, 6H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ168.11, 163.56, 145.47, 136.19, 135.07, 133.81, 129.60, 128.94, 128.10, 127.09, 127.06, 124.38, 52.50, 28.53, 18.66; IR (KBr): ν_{max} = 3456, 2361, 1640, 1244, 824 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{22}H_{26}N_2O_2$ $[M + H]^+$ 351.2067, found 351.2069.



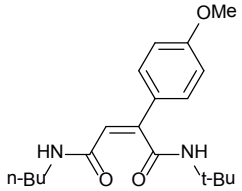
N^1-(*tert*-butyl)-N^4-cyclohexyl-2-phenylmaleamide (4e).

White solid: m.p. 181-186 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.43 (m, 2H), 7.35 – 7.34 (m, 3H), 6.96 (s, 1H), 6.21 (s, 1H), 5.88 (s, 1H), 3.84 – 3.77 (m, 1H), 1.93 – 1.89 (m, 2H), 1.72-1.69 (m, 2H), 1.60 – 1.54 (m, 2H), 1.39 (s, 9H), 1.25 – 1.19 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.13, 165.10, 144.02, 135.64, 129.37, 128.87, 126.57, 123.33, 52.36, 48.64, 32.79, 28.55, 25.49, 24.74; IR (KBr): ν_{max} = 3435, 2929, 2360, 1712, 1643, 1244, 883 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{20}H_{28}N_2O_2$ $[M + H]^+$ 329.2224, found 329.2225.



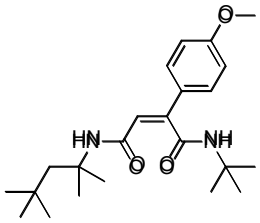
N^1-(*tert*-butyl)-N^4-butyl-2-phenylmaleamide (4f).

Brown oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.40 (m, 2H), 7.36 – 7.28 (m, 3H), 6.97 (s, 1H), 6.19 (s, 1H), 5.96 (s, 1H), 3.28-3.23 (m, 2H), 1.53-1.46 (m, 2H), 1.37 (s, 9H), 1.35-1.29 (m, 2H), 0.89 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ168.12, 165.76, 144.48, 135.37, 129.36, 128.82, 126.50, 122.76, 77.32, 77.00, 76.68, 52.30, 39.49, 31.35, 28.46, 20.10, 13.68; IR (KBr): ν_{max} = 3435, 3264, 2959, 1758, 1628, 1240, 1112, 617 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{18}H_{26}N_2O_2$ $[M + Na]^+$ 325.1886, found 325.1884.



N^1-(*tert*-butyl)-N^4-butyl-2-(4-methoxyphenyl)maleamide (4g).

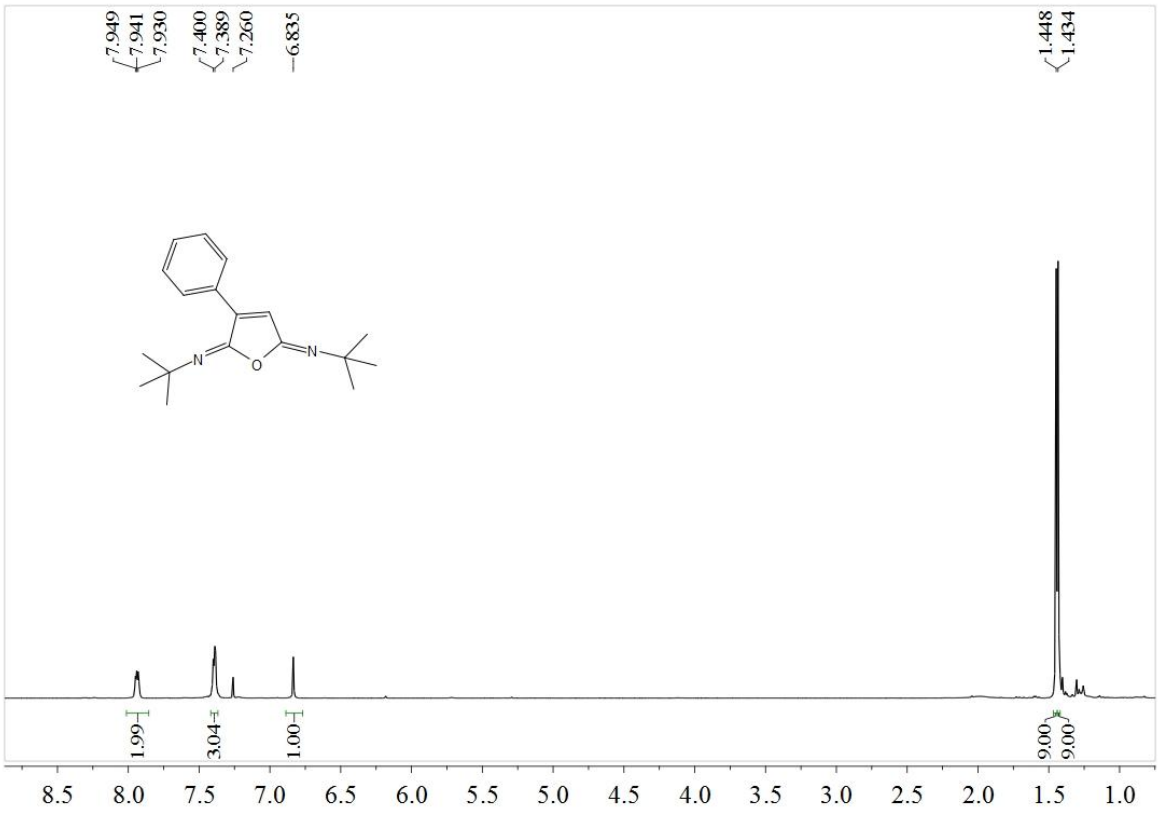
White solid: m.p. 156-158 °C; ^{1}H NMR (400 MHz, Acetone-d_6) δ 7.48 (d, J = 8.8 Hz, 2H), 7.22 (s, 1H), 6.92 (d, J = 9.2 Hz, 3H), 6.20 (s, 1H), 3.81 (s, 3H), 3.26 – 3.21 (m, 2H), 1.53 – 1.46 (dd, J = 14.6, 7.1 Hz, 2H), 1.44 (s, 9H), 1.40 – 1.34 (m, 2H), 0.91 (t, J = 7.4 Hz, 3H); ^{13}C NMR (100 MHz, Acetone-d_6) δ 168.5, 165.2, 161.2, 147.0, 129.7, 128.7, 119.2, 114.6, 55.5, 51.8, 39.4, 32.4, 28.8, 20.6, 13.9; IR (KBr): ν_{max} = 2931, 1645, 1512, 1251, 832 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{19}H_{28}N_2O_3$ $[M + Na]^+$ 355.1992, found 355.1990.

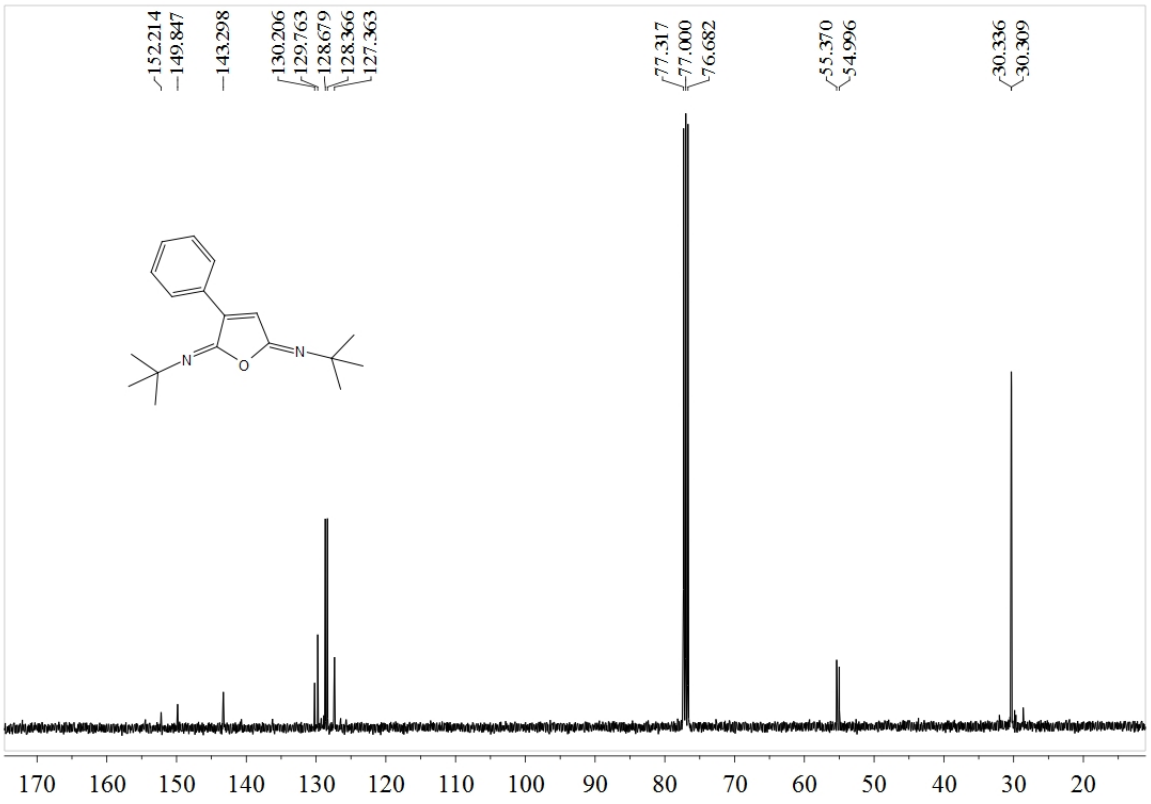


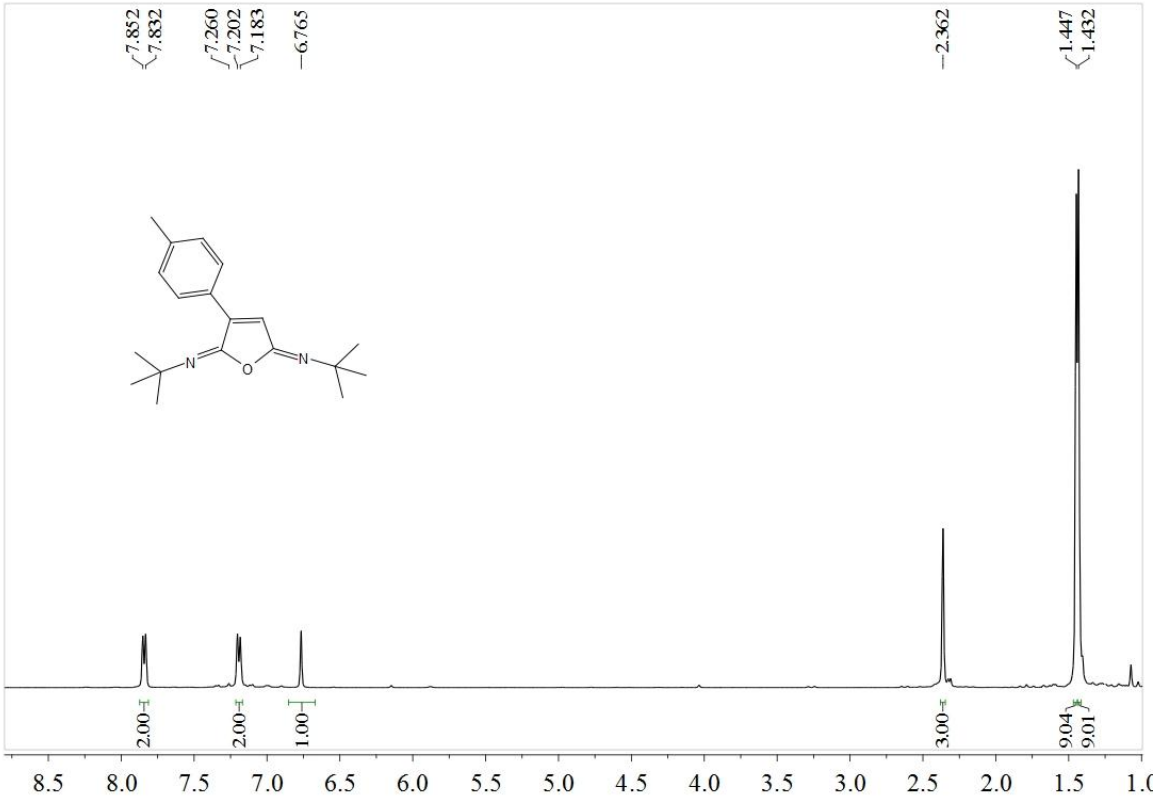
N^1-(*tert*-butyl)-N^4-(1,1,3,3-tetramethylbutyl)-2-(4-methoxyphenyl)maleamide (4h).

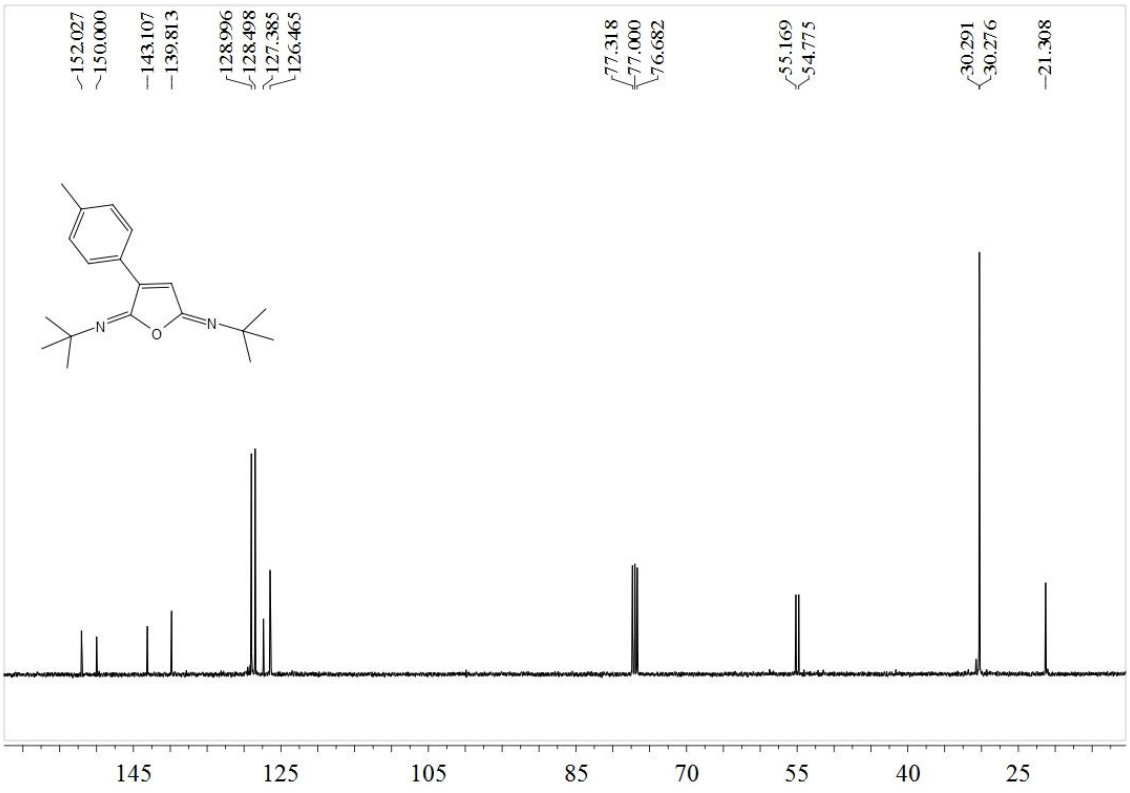
Brown oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, J = 8.8 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 6.83 (s, 1H), 6.06 (s, 1H), 5.82 (s, 1H), 3.79 (s, 3H), 1.79 (s, 2H), 1.42 (s, 6H), 1.40 (s, 9H), 0.99 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.91, 168.45, 165.44, 160.52, 143.07, 128.02, 122.59, 114.26, 55.63, 55.33, 52.30, 51.17, 31.67, 31.43, 29.03, 28.66; IR (KBr): ν_{max} = 3462, 2361, 1633, 1513, 1230, 760, 692 cm^{-1}; HRMS-ESI (m/z): calcd for. $C_{23}H_{36}N_2O_3$ $[M + H]^+$ 389.2799, found 389.2798.

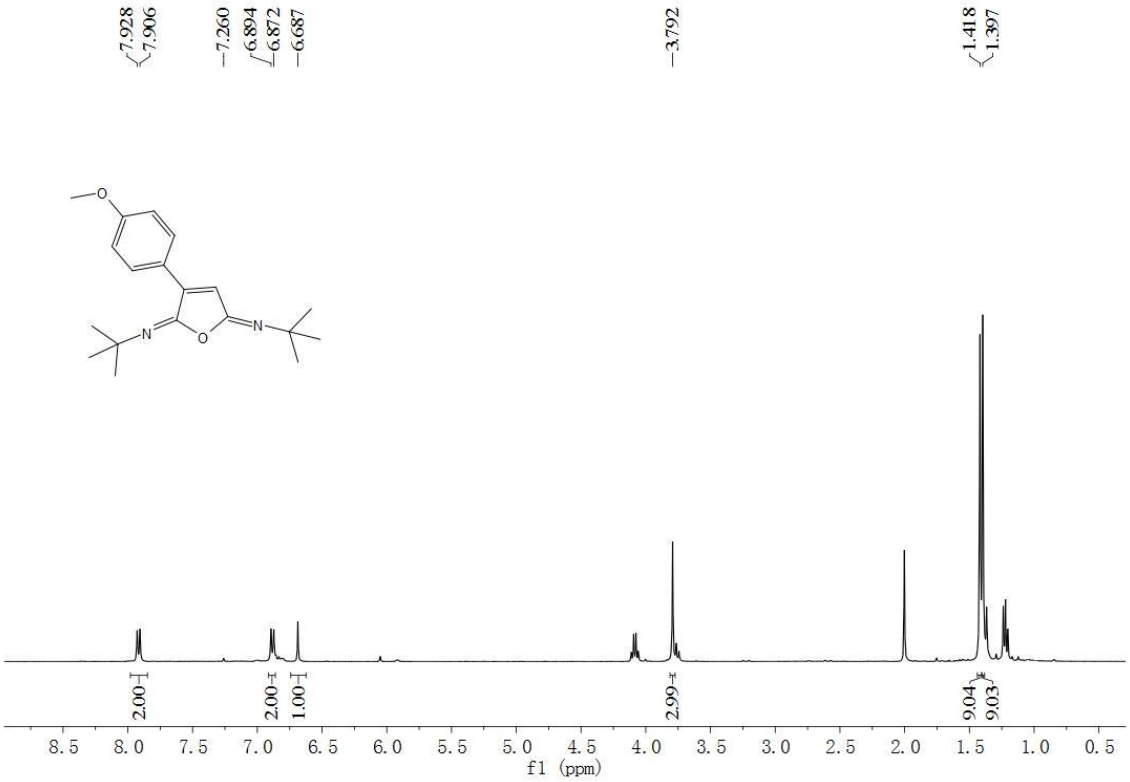
NMR Spectra for Compounds 3a-3x, 4a-4h

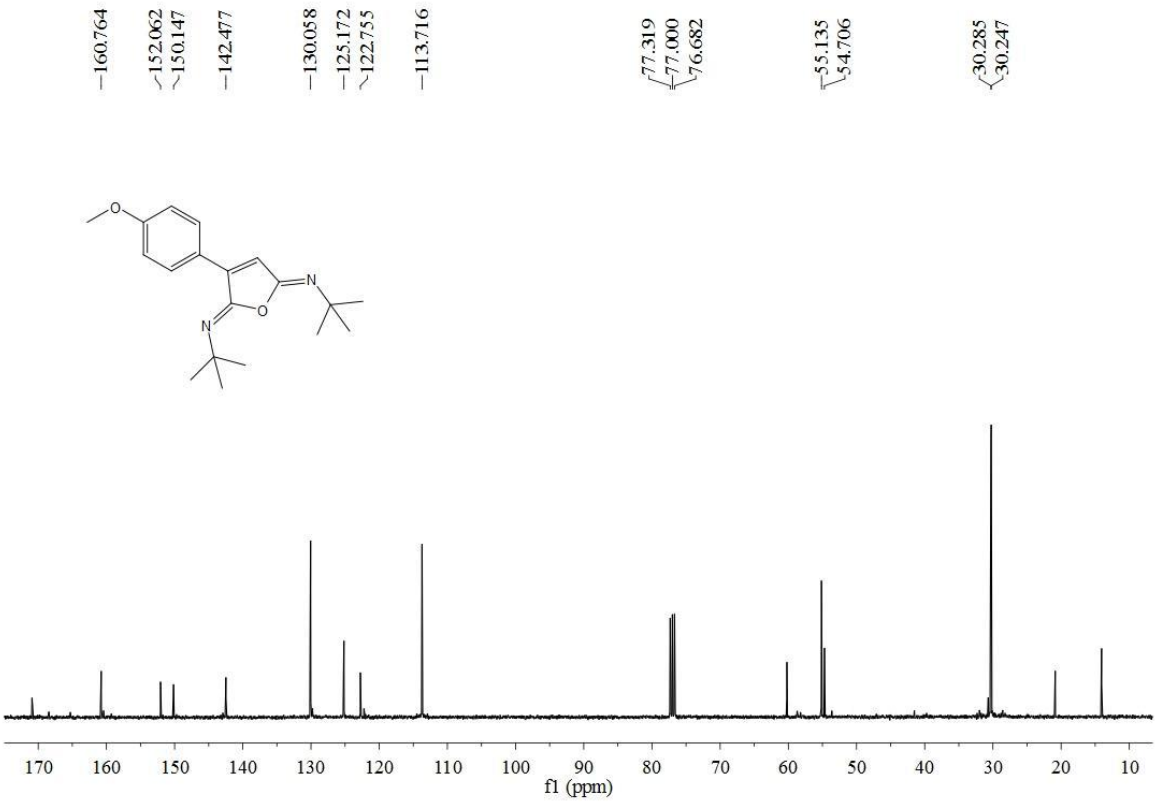


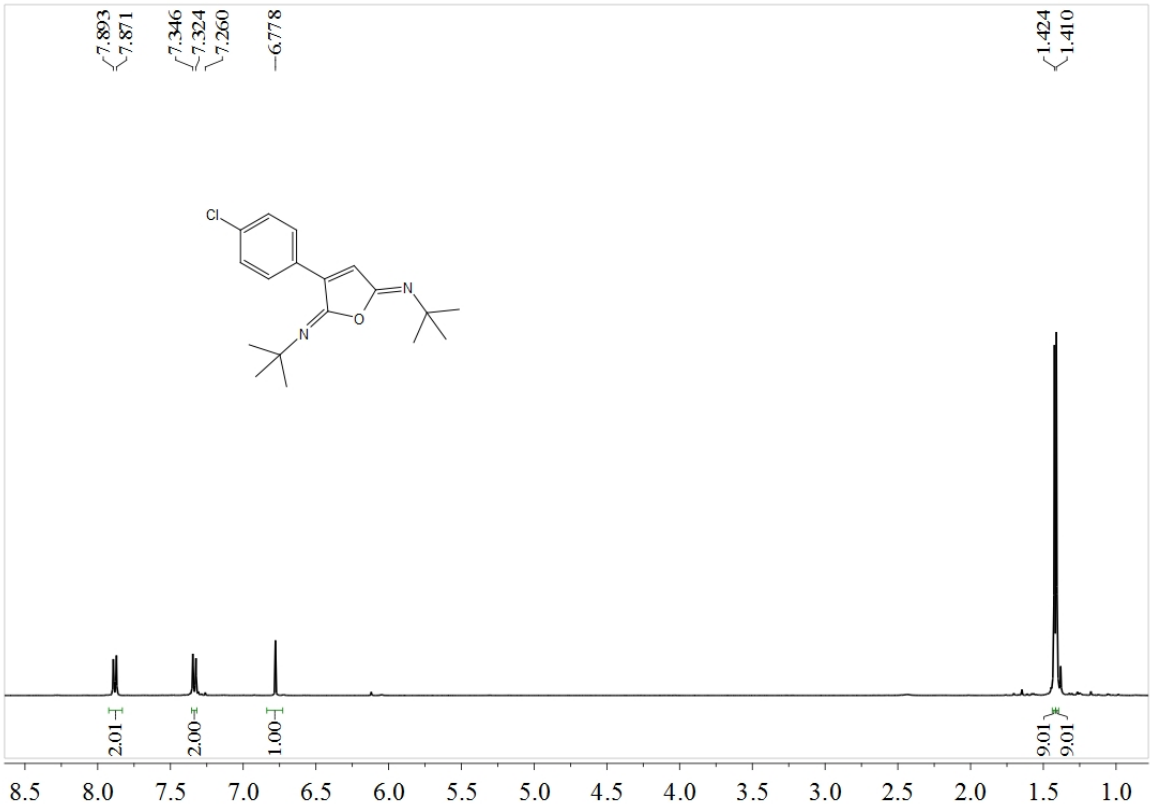


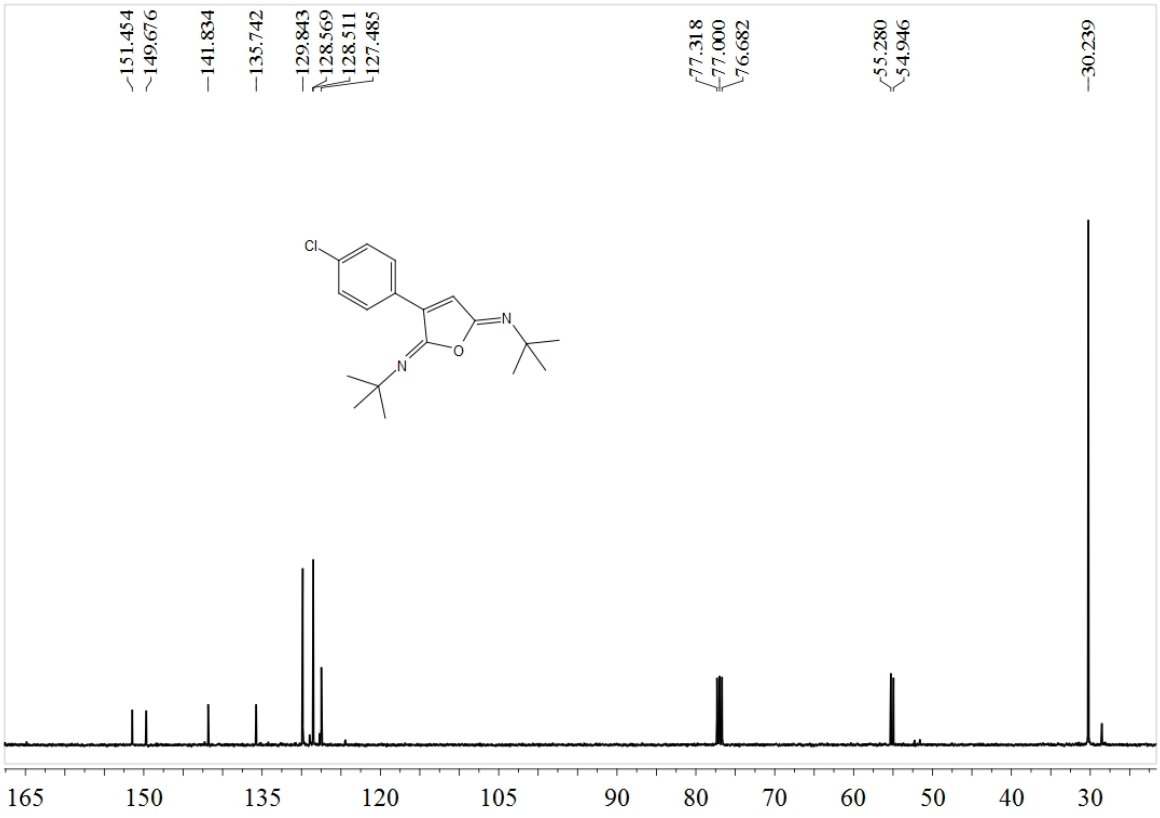

7.852
7.832
7.260
7.202
7.183
6.765
2.362
1.447
1.432
2.00
2.00
1.00
3.00
9.04
9.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
N
O
N

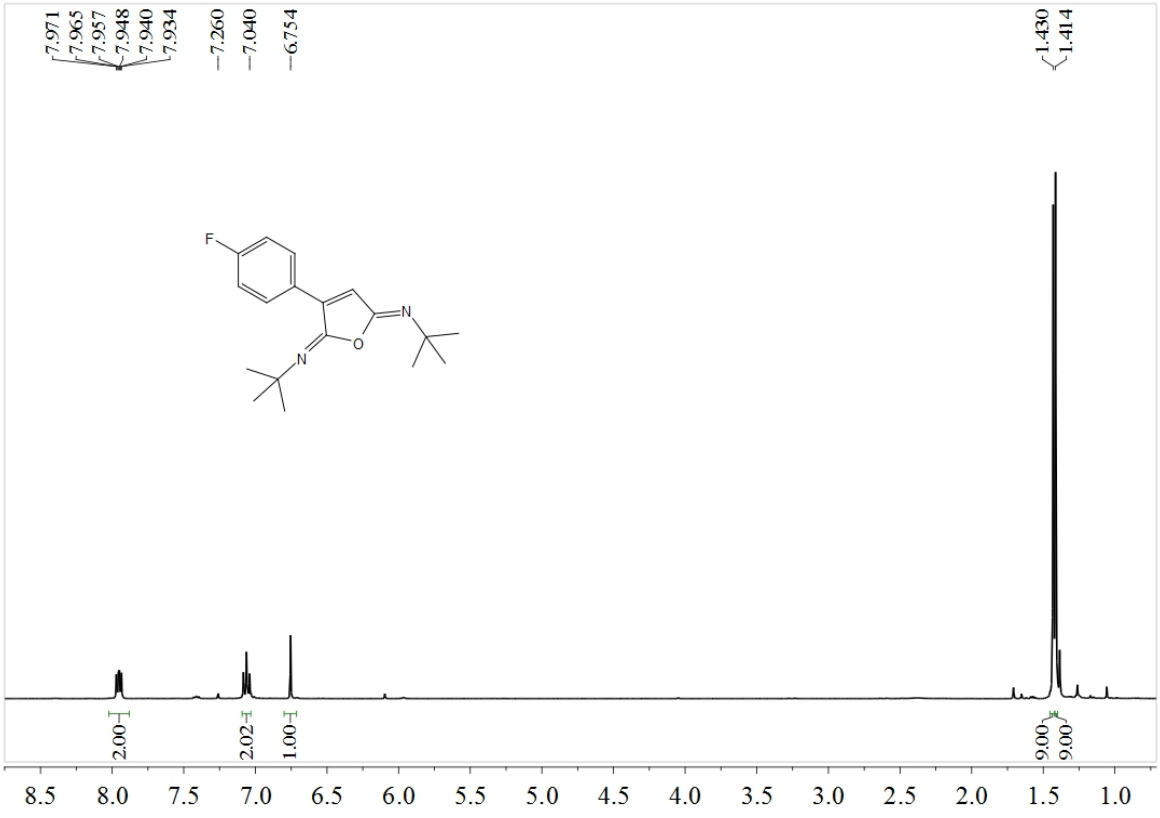

152.027
150.000
143.107
139.813
128.996
128.498
127.385
126.465
77.318
77.000
76.682
55.169
54.775
30.291
30.276
21.308
145
125
105
85
70
55
40
25
N
O
N

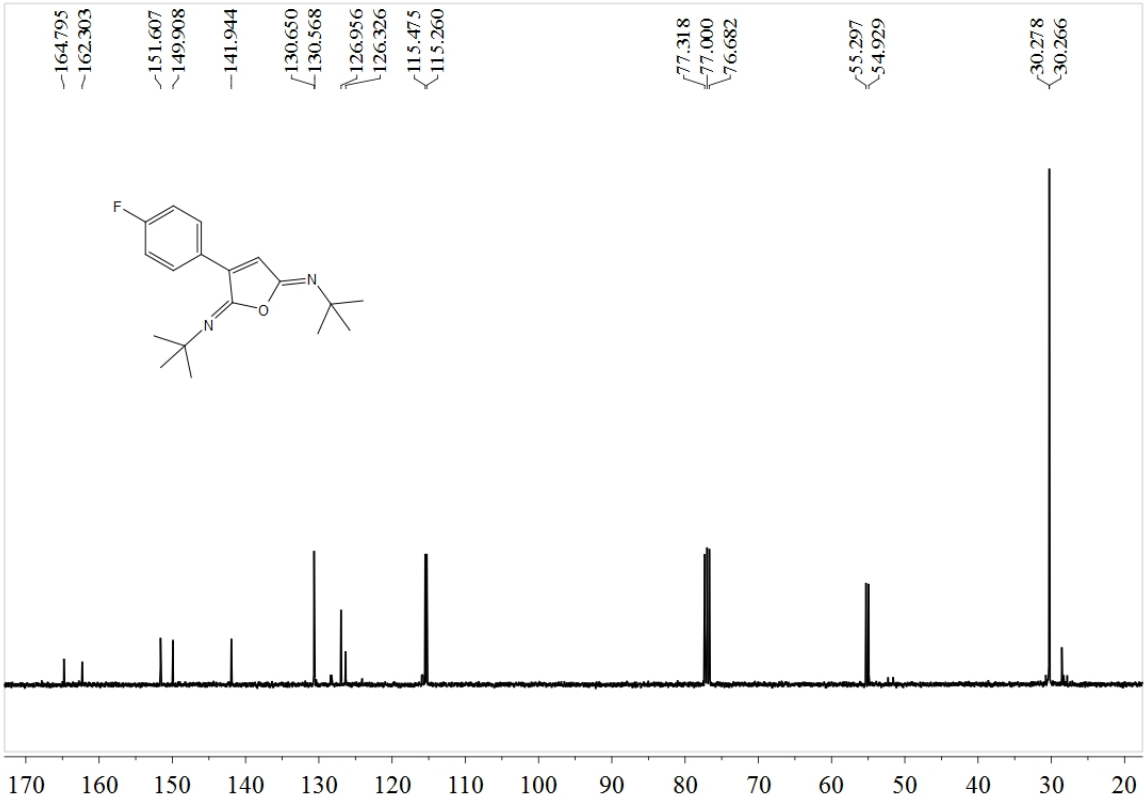

7.928
7.906
7.260
6.894
6.872
6.687
3.792
1.418
1.397
2.00
2.00
1.00
2.99
9.04
9.03
f1 (ppm)

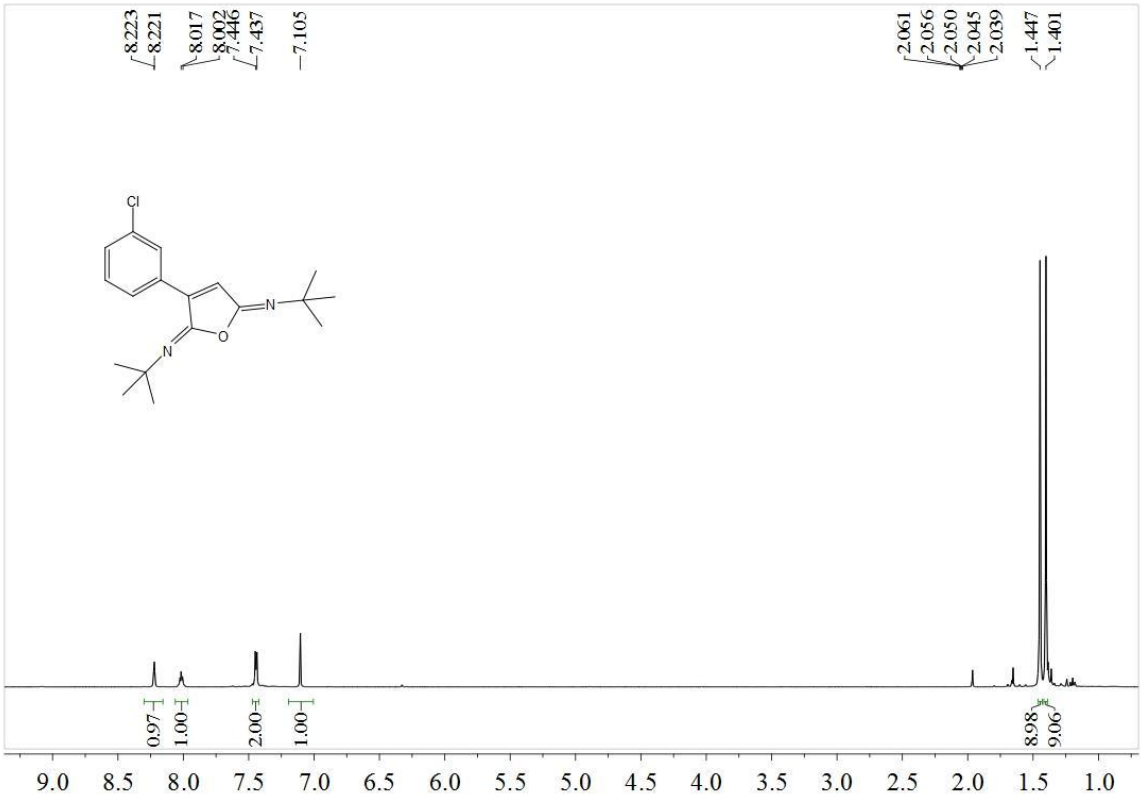

160.764
152.062
150.147
142.477
130.058
125.172
122.755
113.716
77.319
77.000
76.682
55.135
54.706
30.285
30.247
f1 (ppm)

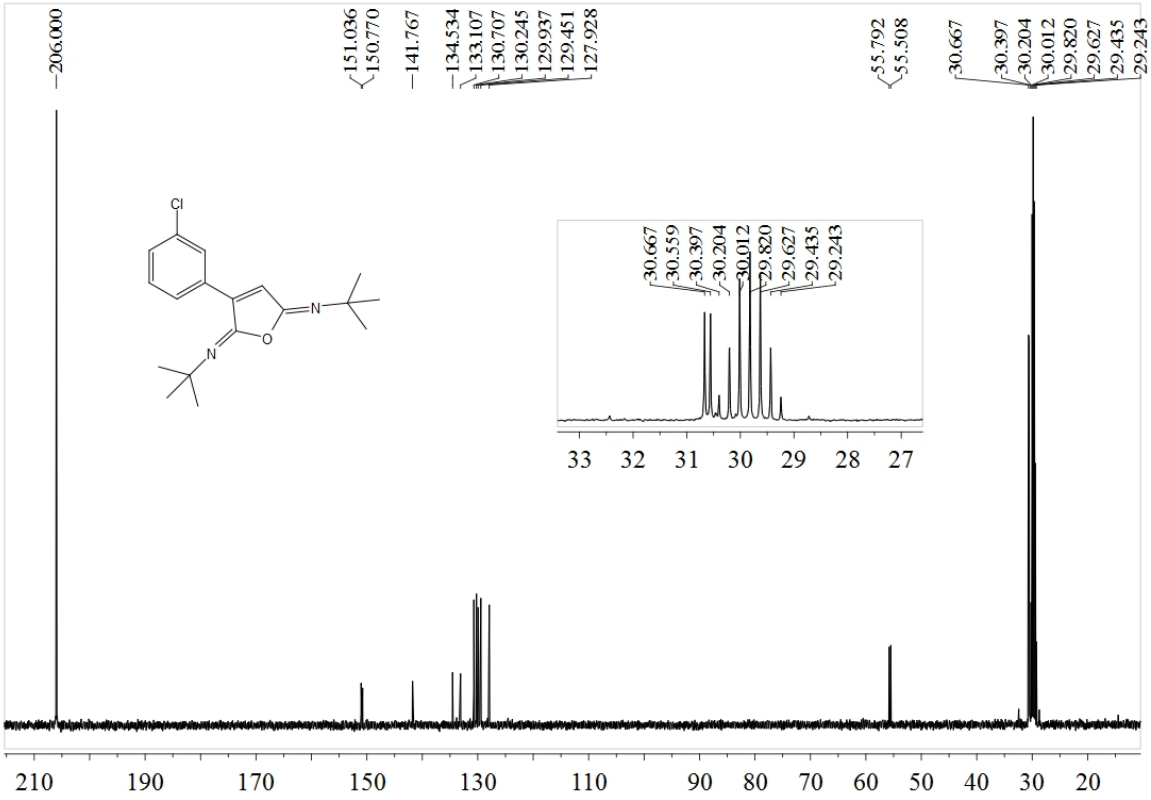

7.893
7.871
7.346
7.324
7.260
6.778
1.424
1.410
Cl
N
O
N
2.01
2.00
1.00
9.01
9.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

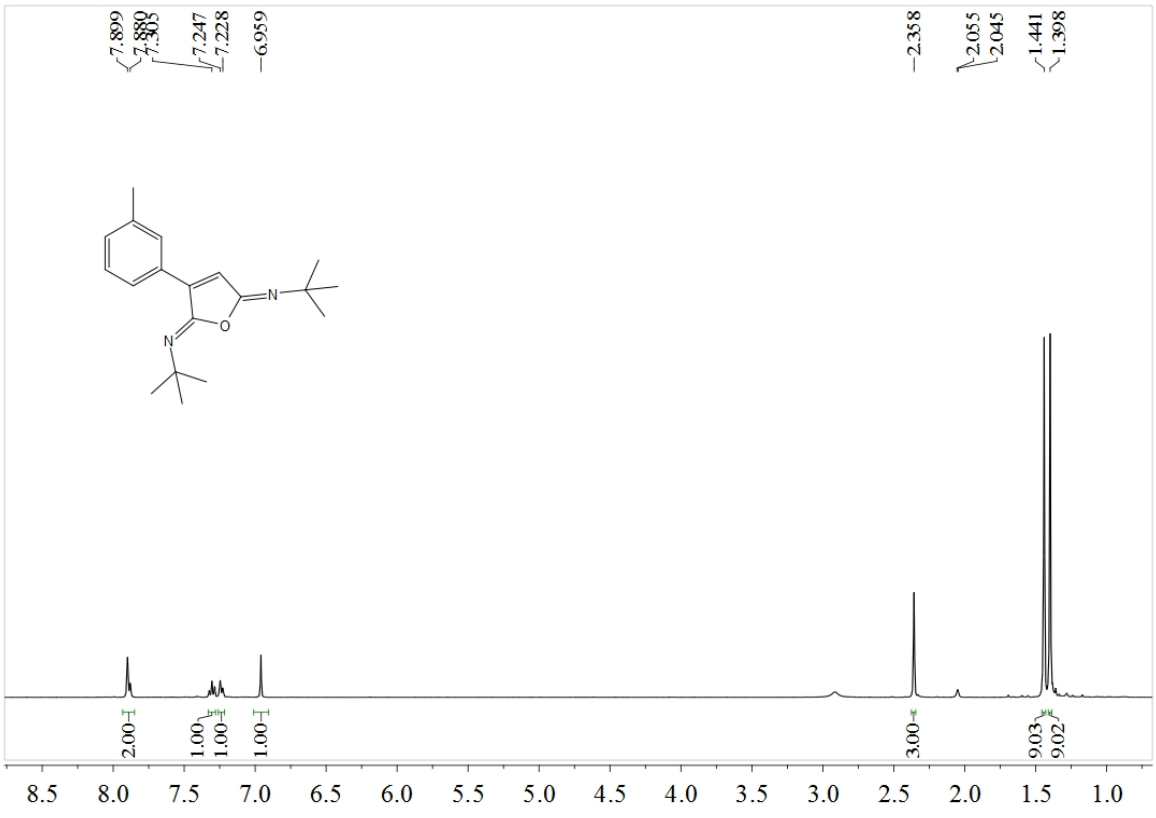

151.454
149.676
141.834
135.742
129.843
128.569
128.511
127.485
77.318
77.000
76.682
55.280
54.946
30.239
Cl
N
O
N
165
150
135
120
105
90
80
70
60
50
40
30

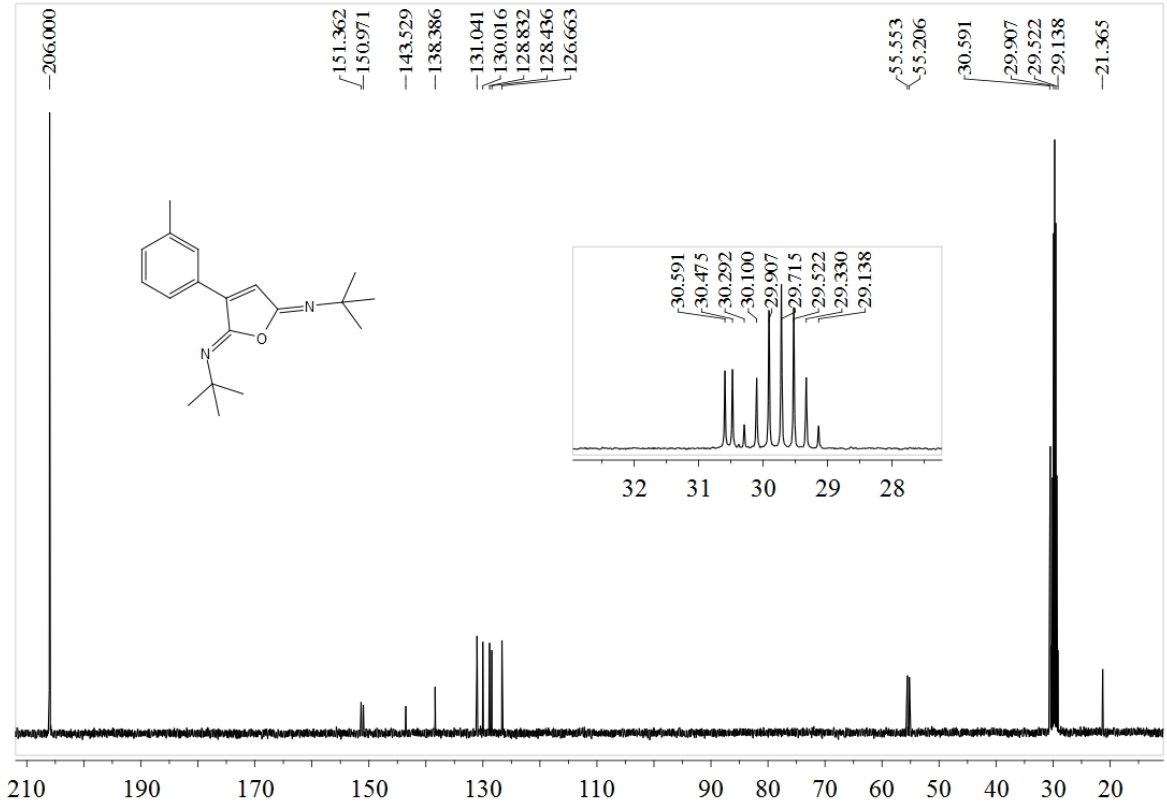


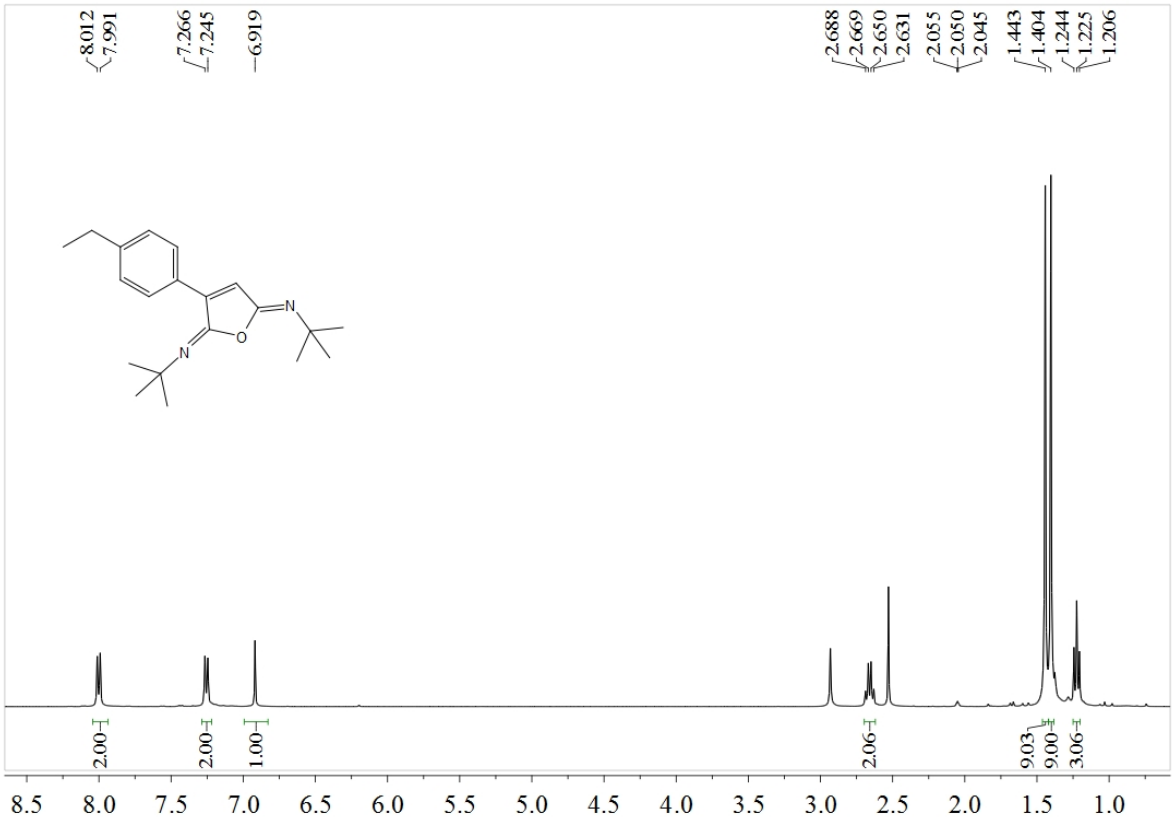


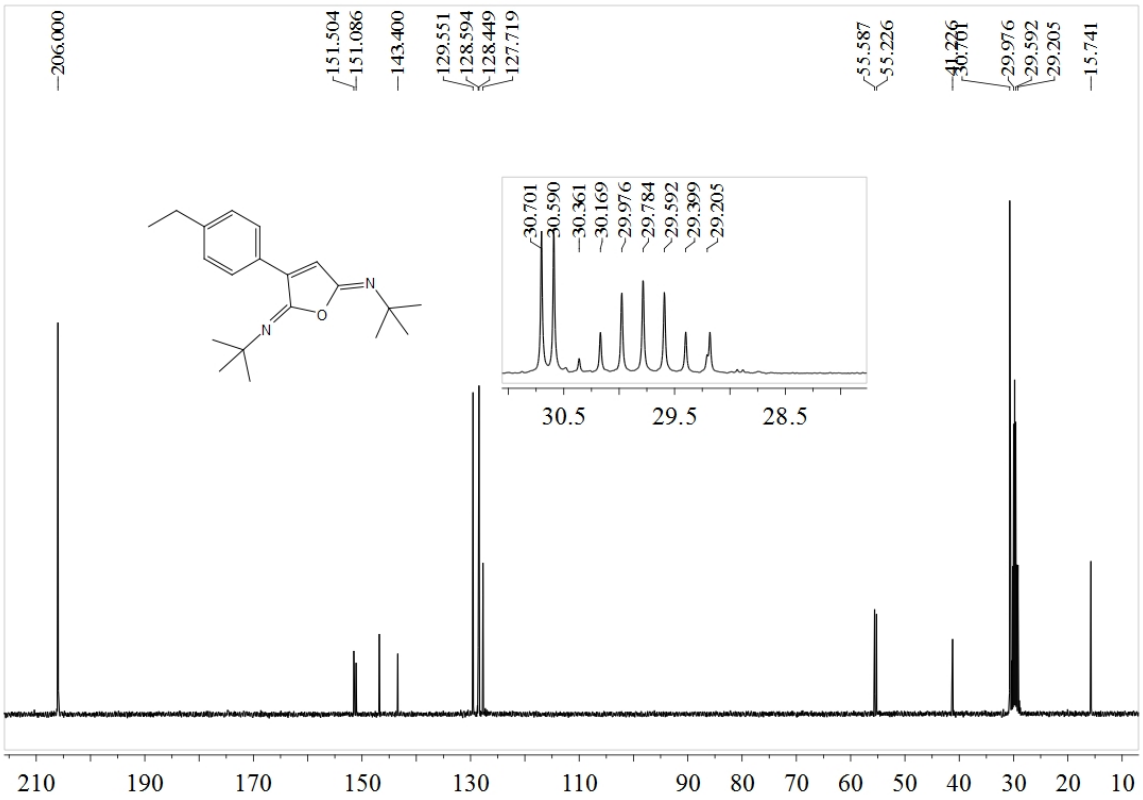

8.223
8.221
8.017
8.002
7.446
7.437
7.105
2.061
2.056
2.050
2.045
2.039
1.447
1.401
Cl
N
O
N
0.97
1.00
2.00
1.00
8.98
9.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0

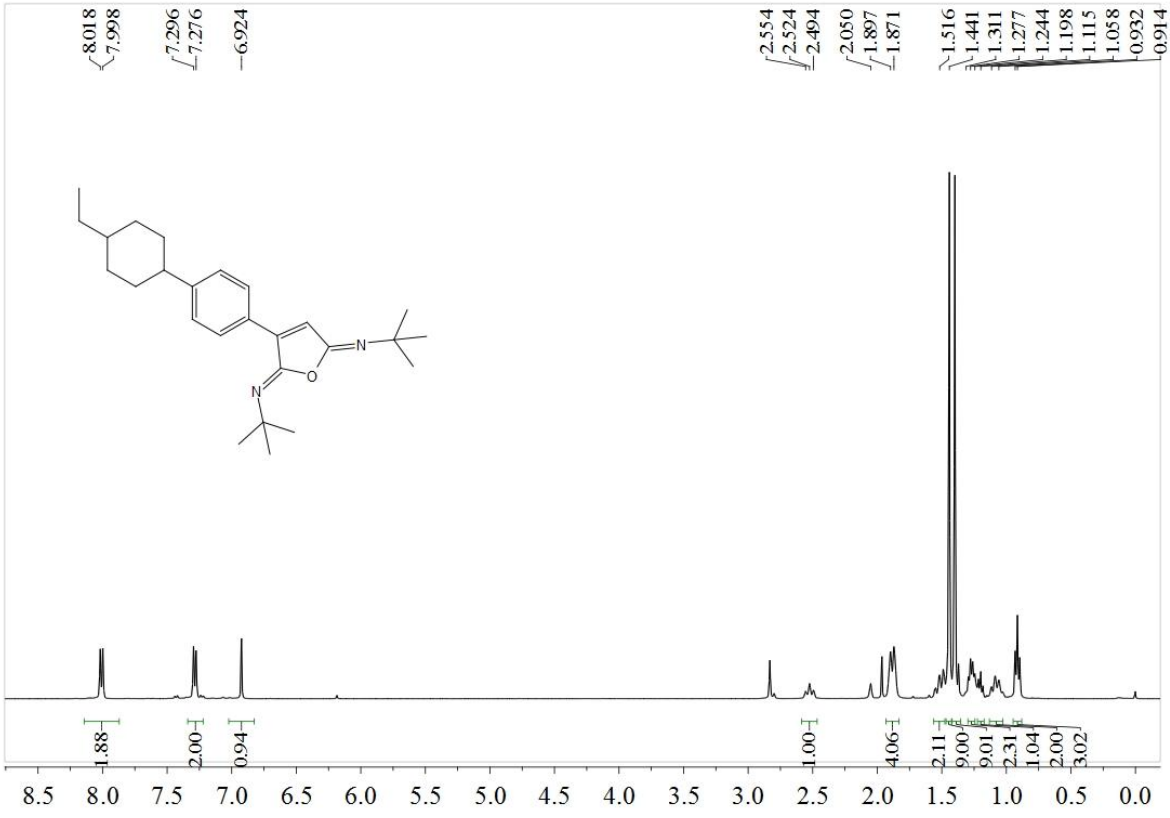

206.000
151.036
150.770
141.767
134.534
133.107
130.707
130.245
129.937
129.451
127.928
55.792
55.508
30.667
30.397
30.204
30.012
29.820
29.627
29.435
29.243
Cl
N
O
N
30.667
30.559
30.397
30.204
30.012
29.820
29.627
29.435
29.243
33 32 31 30 29 28 27
210 190 170 150 130 110 90 80 70 60 50 40 30 20

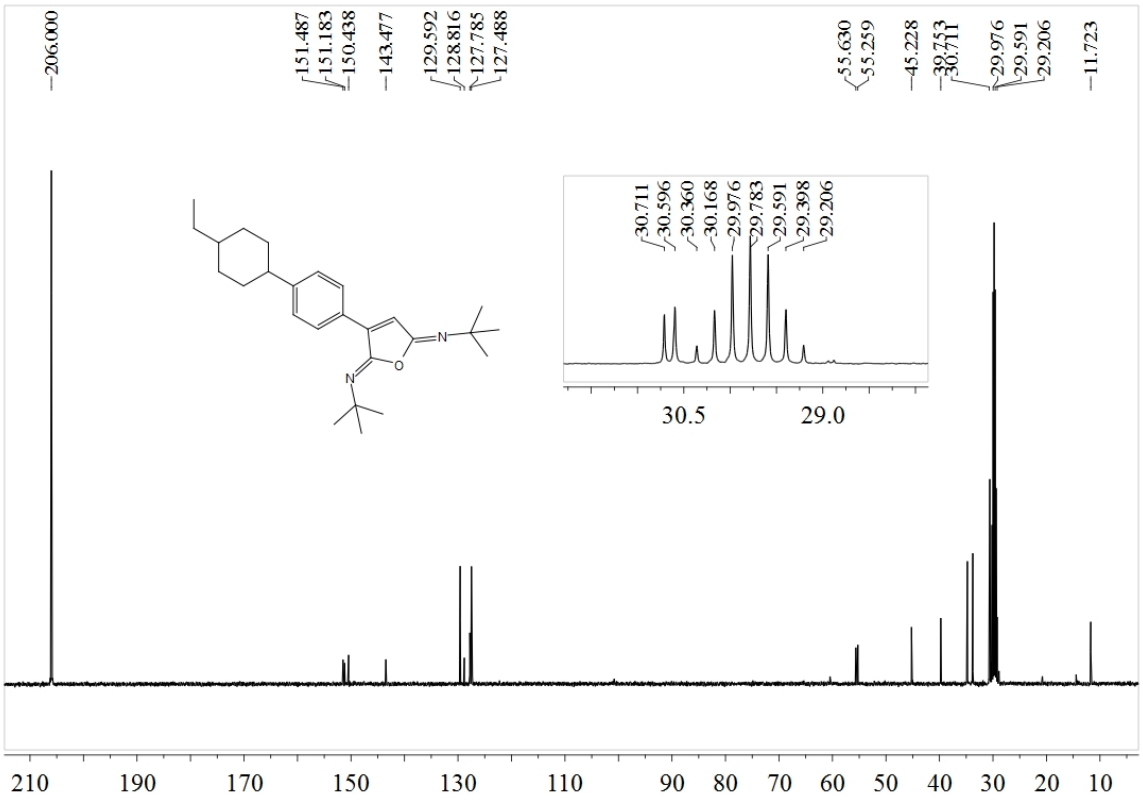

7.899
7.880
7.303
7.247
7.228
6.959
2.358
2.055
2.045
1.441
1.398
2.00
1.00
1.00
1.00
3.00
9.03
9.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

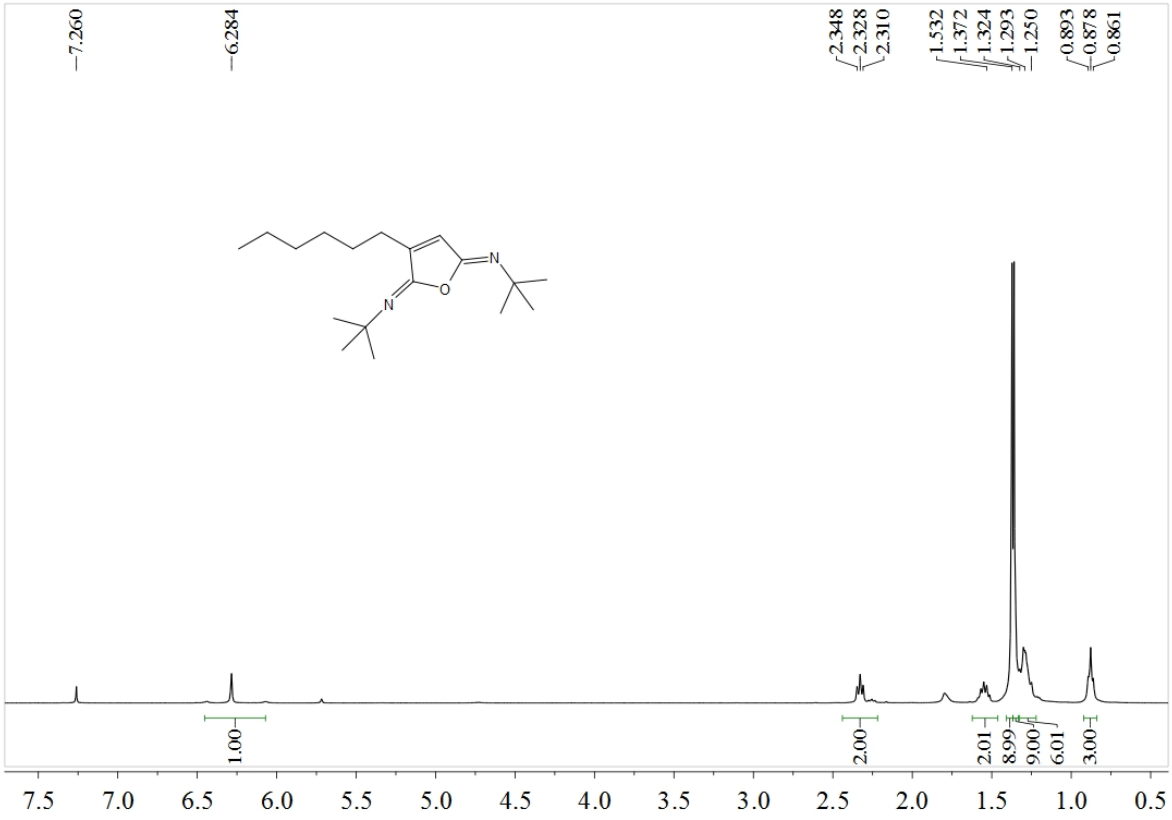

206.000
151.362
150.971
143.529
138.386
131.041
130.016
128.832
128.436
126.663
55.553
55.206
30.591
29.907
29.522
29.138
21.365
30.591
30.475
30.292
30.100
29.907
29.715
29.522
29.330
29.138
32
31
30
29
28
210
190
170
150
130
110
90
80
70
60
50
40
30
20

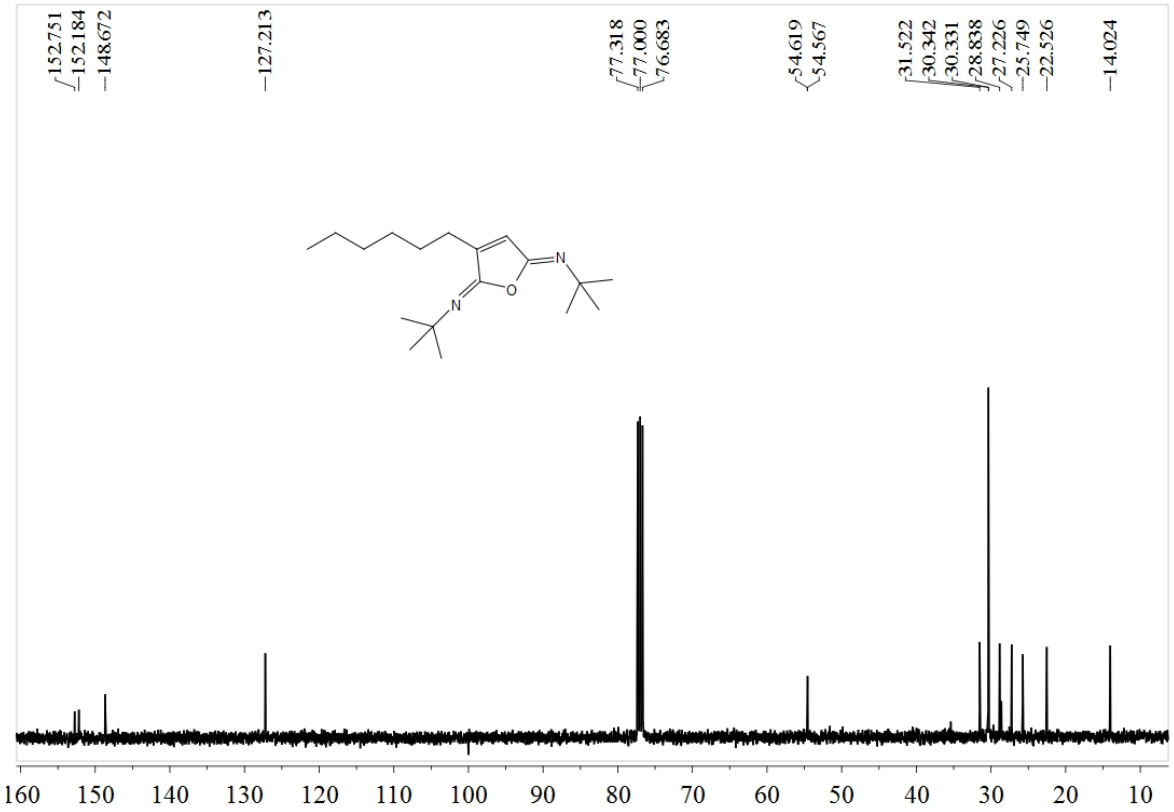

8.012
7.991
7.266
7.245
6.919
2.688
2.669
2.650
2.631
2.055
2.050
2.045
1.443
1.404
1.244
1.225
1.206
2.00
2.00
1.00
2.06
9.03
9.00
3.06
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

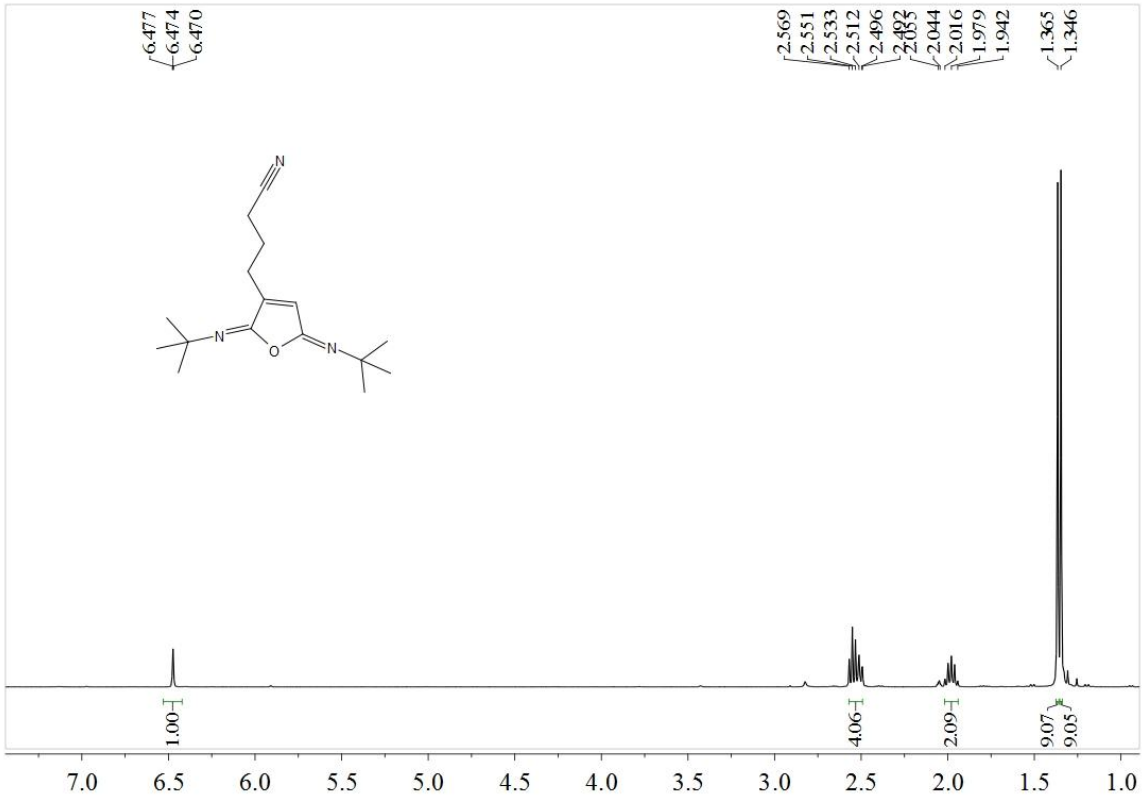

206.000
151.504
151.086
143.400
129.551
128.594
128.449
127.719
55.587
55.226
41.226
30.701
29.976
29.592
29.205
15.741
30.701
30.590
30.361
30.169
29.976
29.784
29.592
29.399
29.205
30.5
29.5
28.5
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10

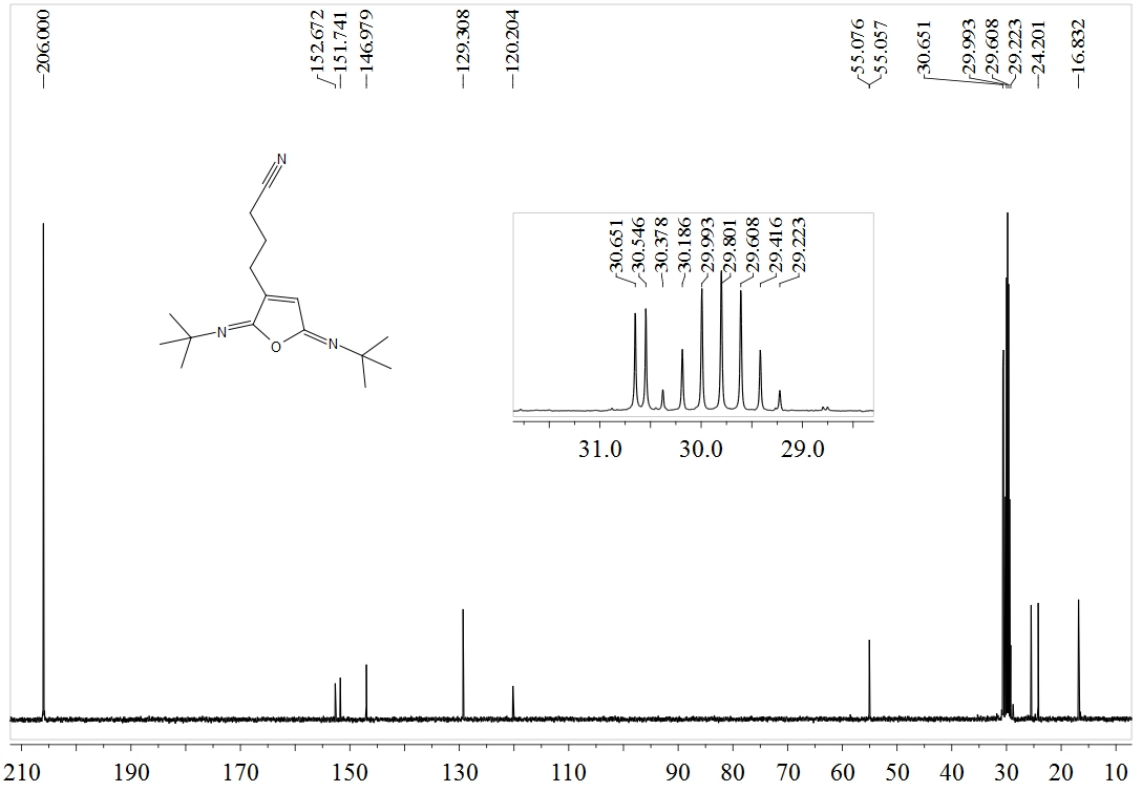

8.018
7.998
7.296
7.276
6.924
2.554
2.524
2.494
2.050
1.897
1.871
1.516
1.441
1.311
1.277
1.244
1.198
1.115
1.058
0.932
0.914
1.88
2.00
0.94
1.00
4.06
2.11
9.00
9.01
2.31
1.04
2.00
3.02
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

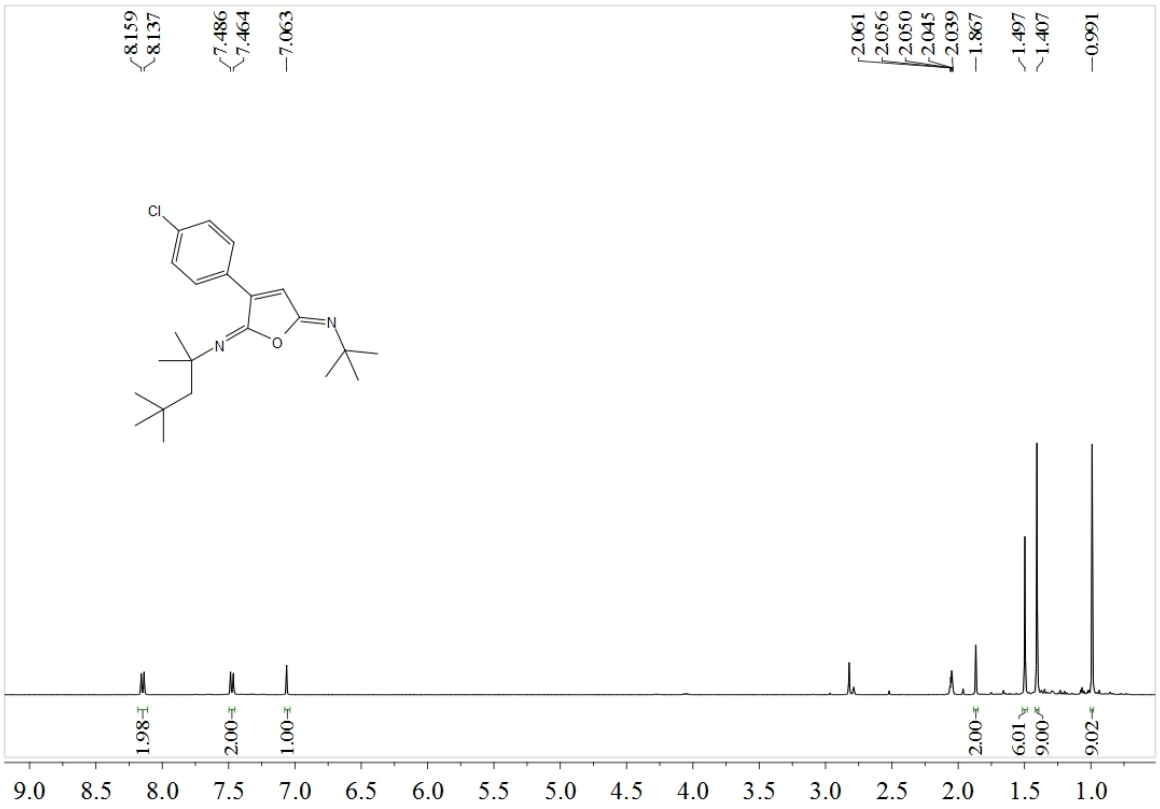

206.000
151.487
151.183
150.438
143.477
129.592
128.816
127.785
127.488
55.630
55.259
45.228
39.753
30.711
29.976
29.591
29.206
11.723
30.711
30.596
30.360
30.168
29.976
29.783
29.591
29.398
29.206
30.5
29.0
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10

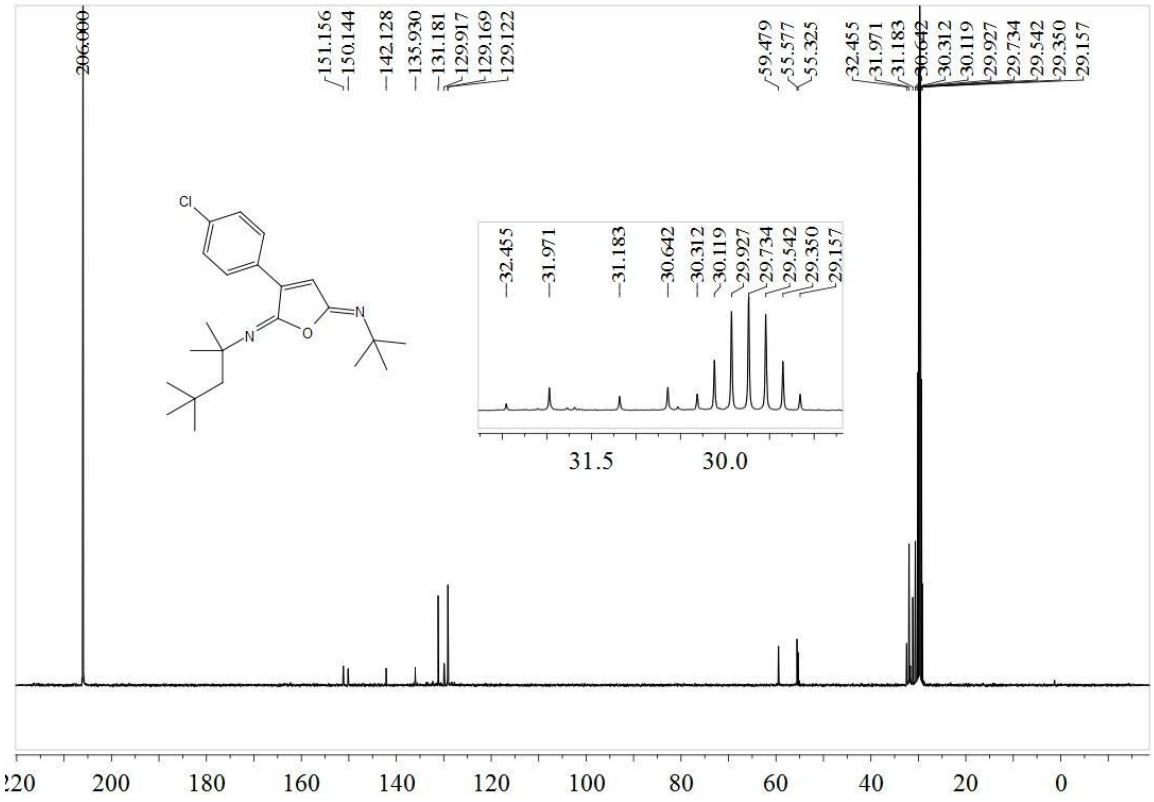

7.260
6.284
2.348
2.328
2.310
1.532
1.372
1.324
1.293
1.250
0.893
0.878
0.861
1.00
2.00
2.01
8.99
9.00
6.01
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

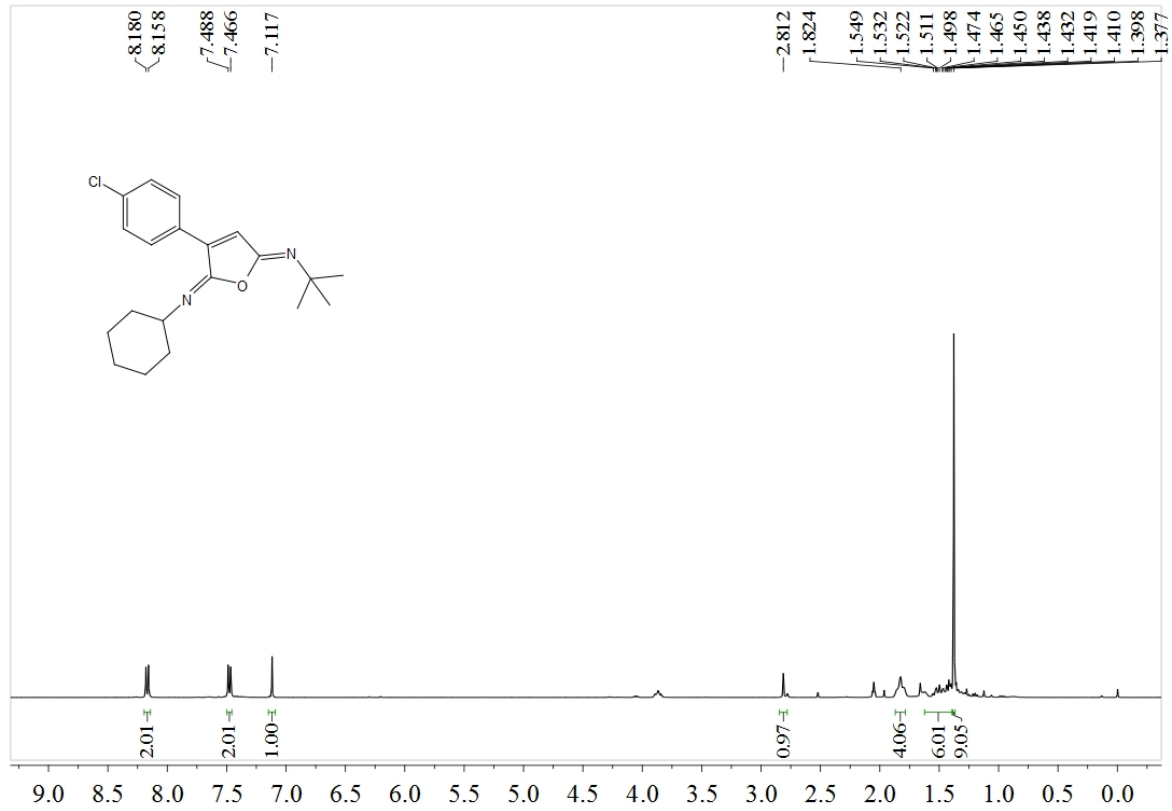

152.751
152.184
148.672
127.213
77.318
77.000
76.683
54.619
54.567
31.522
30.342
30.331
28.838
27.226
25.749
22.526
14.024
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

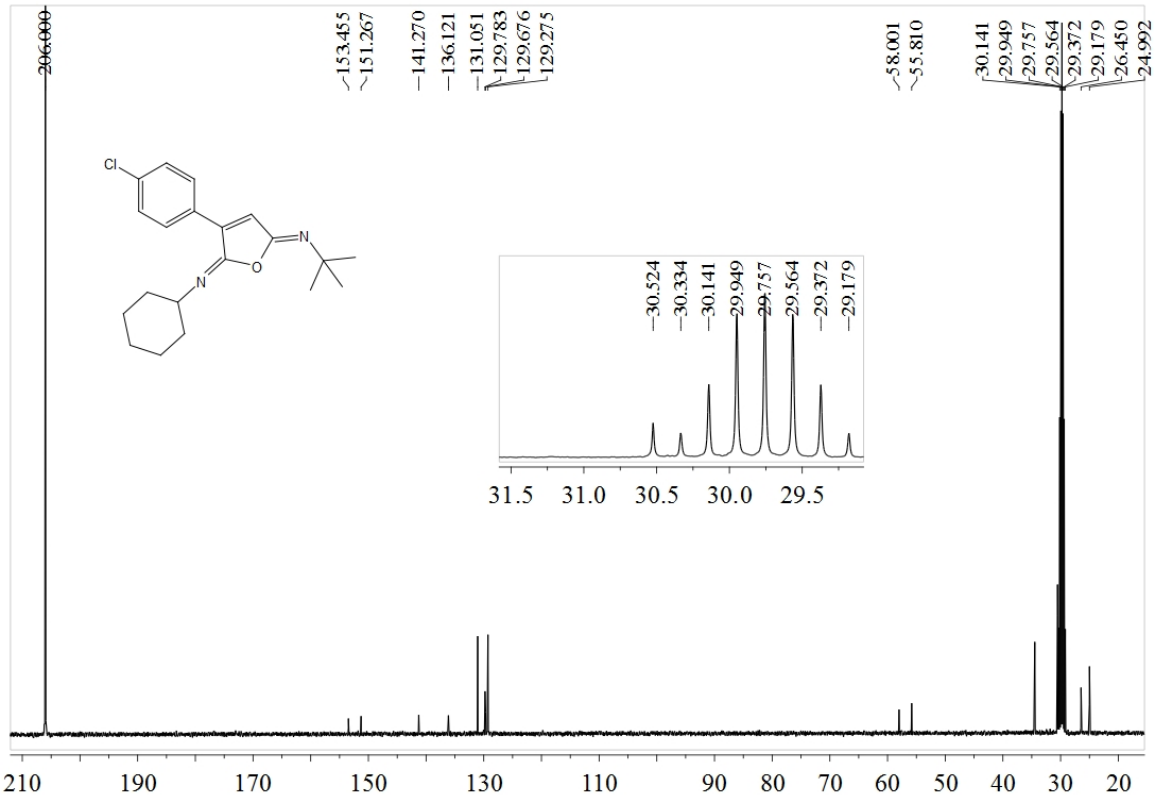

6.477
6.474
6.470
2.569
2.551
2.533
2.512
2.496
2.402
2.055
2.044
2.016
1.979
1.942
1.365
1.346
N
N
O
N
1.00
4.06
2.09
9.07
9.05
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

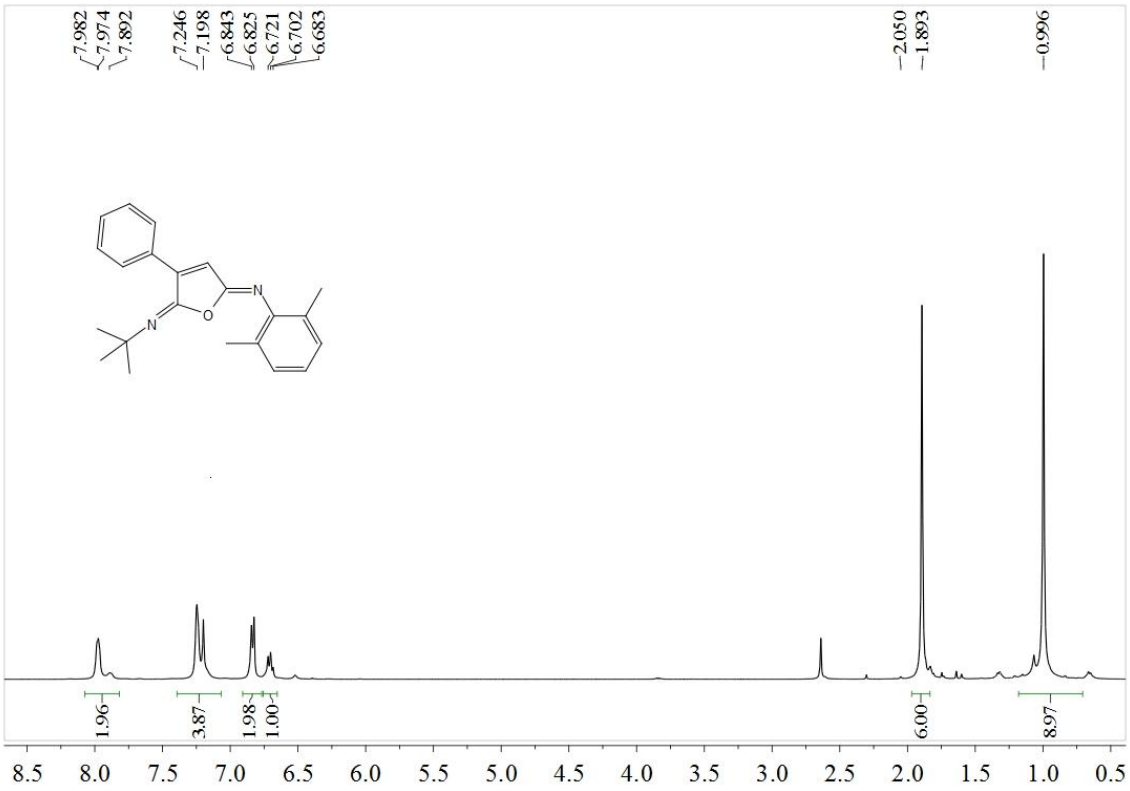

206.000
152.672
151.741
146.979
129.308
120.204
55.076
55.057
30.651
29.993
29.608
29.223
24.201
16.832
N
N
O
N
30.651
30.546
30.378
30.186
29.993
29.801
29.608
29.416
29.223
31.0
30.0
29.0
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10

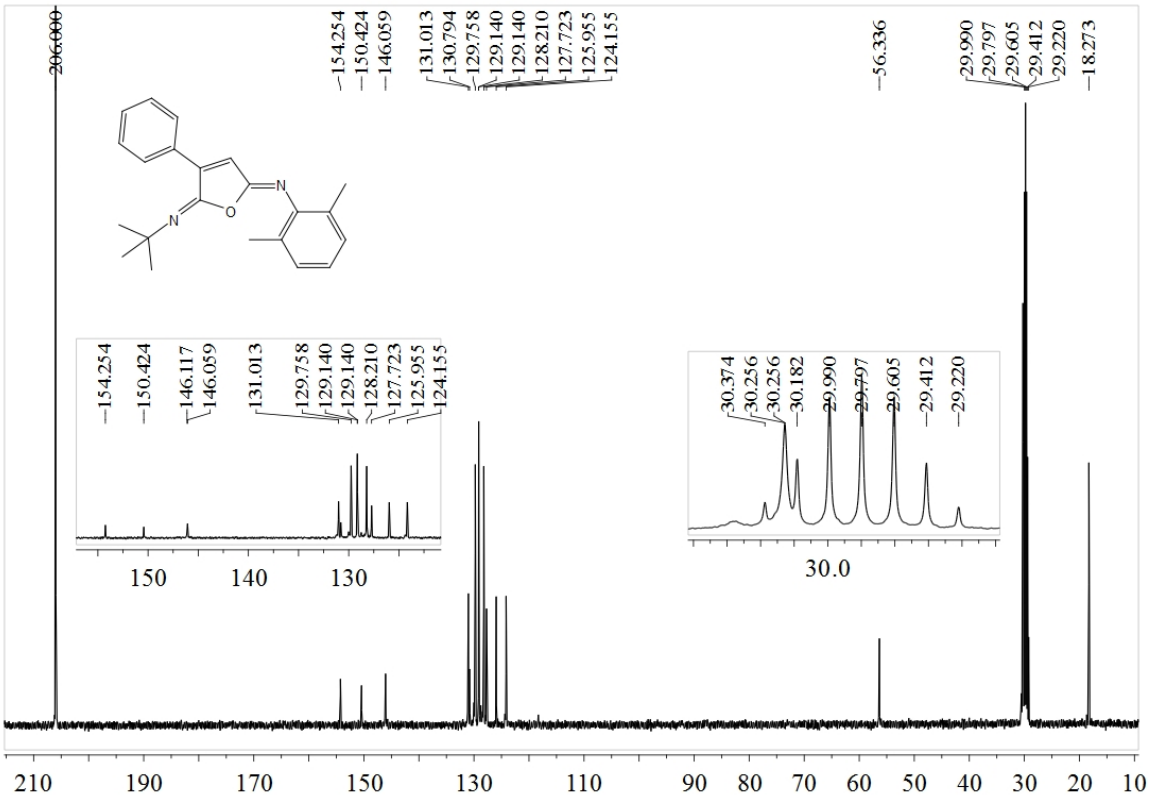

8.159
8.137
7.486
7.464
7.063
2.061
2.056
2.050
2.045
2.039
1.867
1.497
1.407
0.991
Cl
N
O
N
1.98
2.00
1.00
2.00
6.01
9.00
9.02
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0

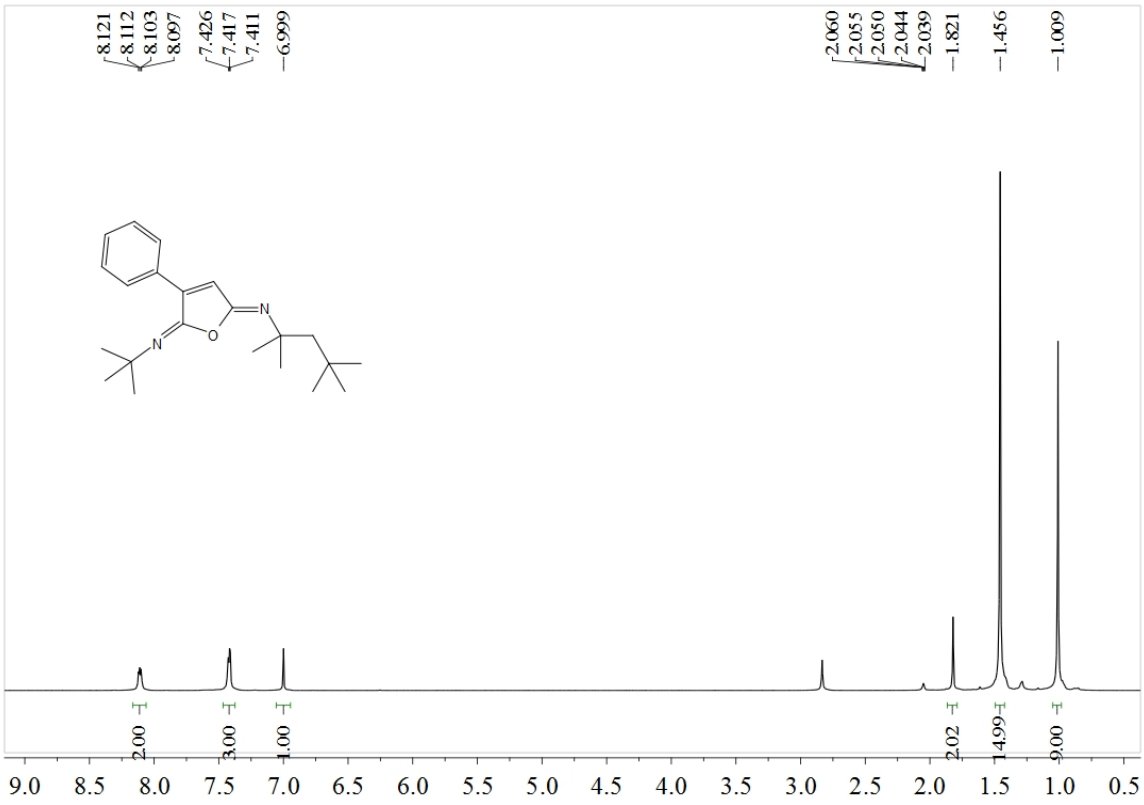

206.000
151.156
150.144
142.128
135.930
131.181
129.917
129.169
129.122
59.479
55.577
55.325
32.455
31.971
31.183
30.642
30.312
30.119
29.927
29.734
29.542
29.350
29.157
Cl
N
O
N
31.5
30.0
220 200 180 160 140 120 100 80 60 40 20 0

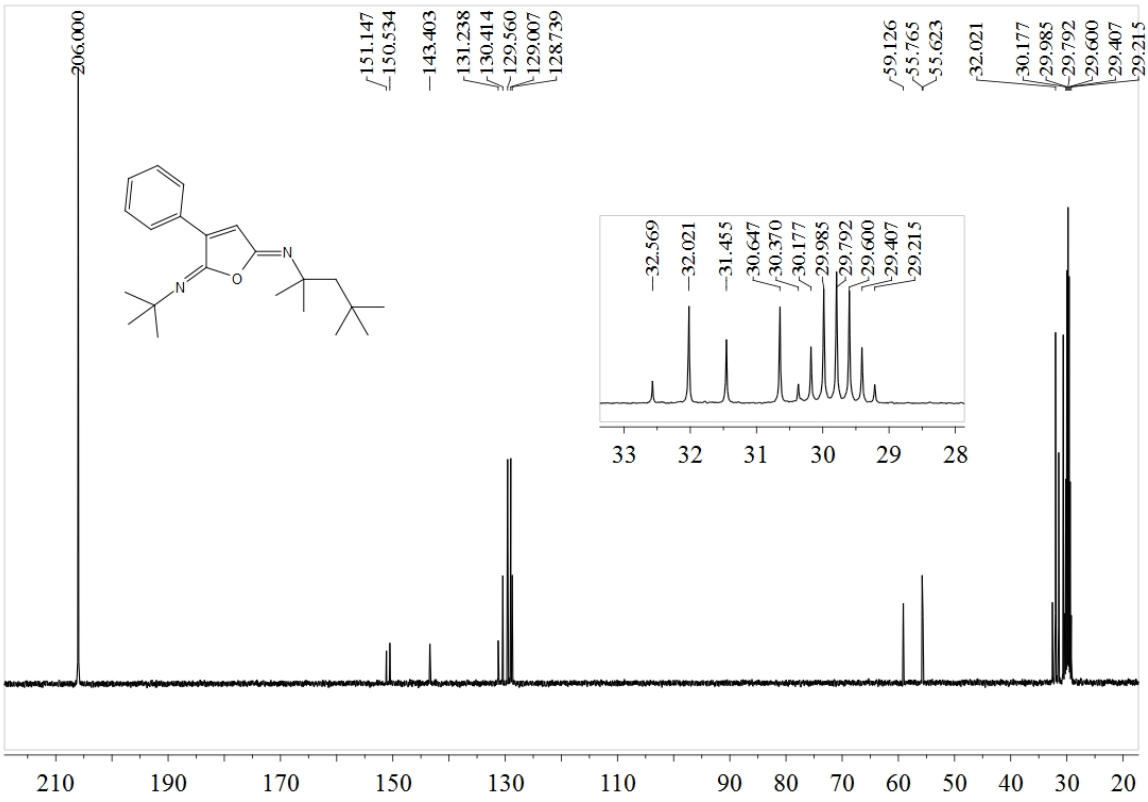

8.180
8.158
7.488
7.466
7.117
2.812
1.824
1.549
1.532
1.522
1.511
1.498
1.474
1.465
1.450
1.438
1.432
1.419
1.410
1.398
1.377
Cl
N
O
N
2.01
2.01
1.00
0.97
4.06
6.01
9.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

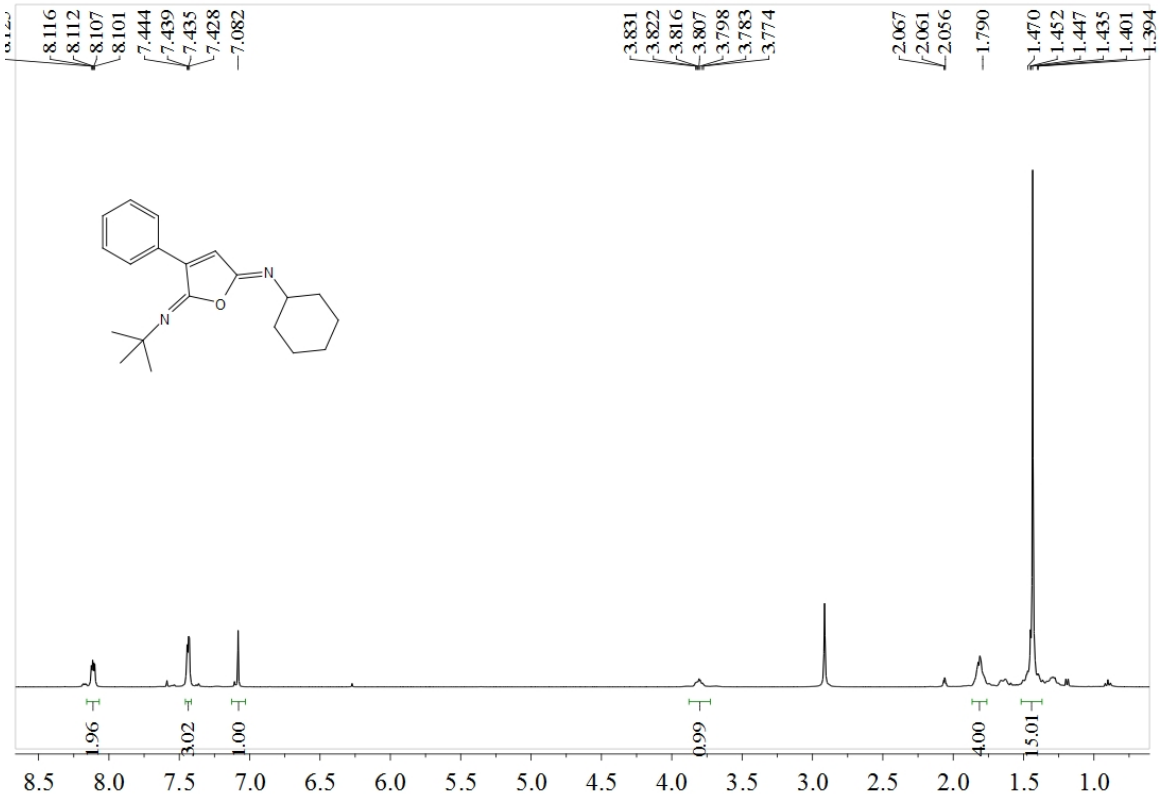

206.800
153.455
151.267
141.270
136.121
131.051
129.783
129.676
129.275
58.001
55.810
30.141
29.949
29.757
29.564
29.372
29.179
26.450
24.992
Cl
N
O
N
30.524
30.334
30.141
29.949
29.757
29.564
29.372
29.179
31.5 31.0 30.5 30.0 29.5
210 190 170 150 130 110 90 80 70 60 50 40 30 20

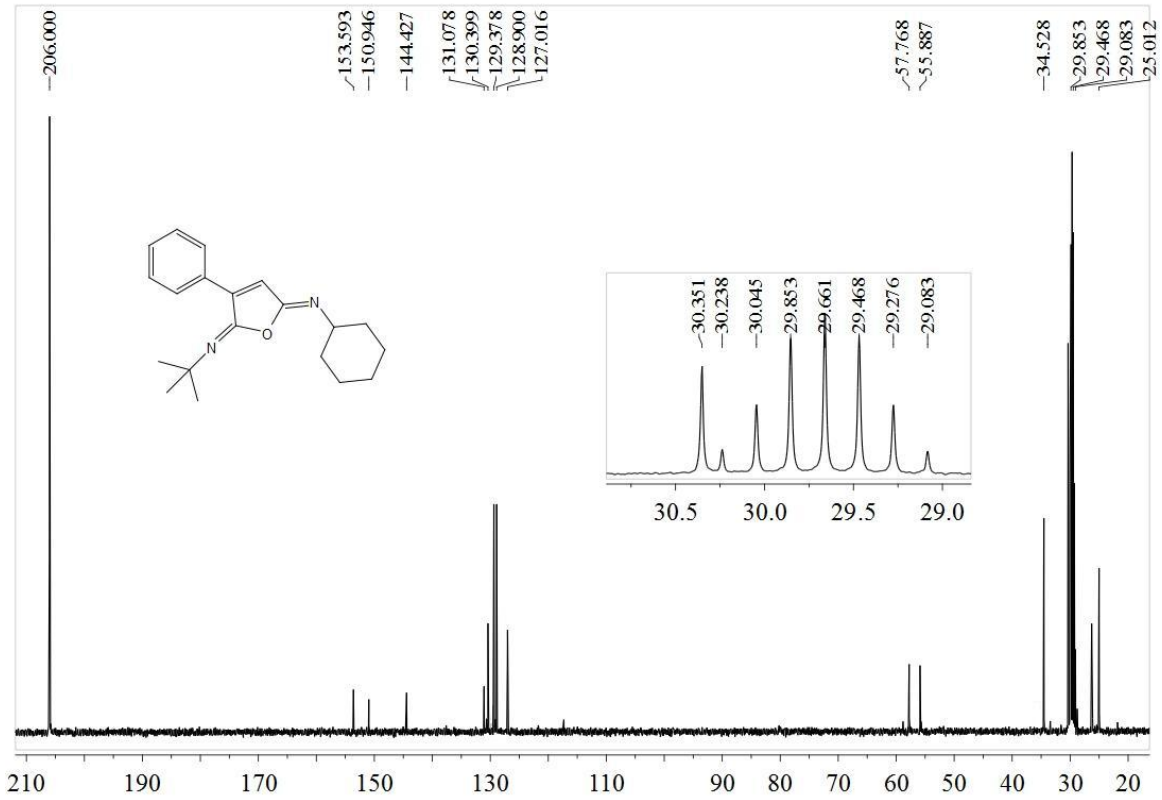

7.982
7.974
7.892
7.246
7.198
6.843
6.825
6.721
6.702
6.683
2.050
1.893
0.996
1.96
3.87
1.98
1.00
6.00
8.97
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

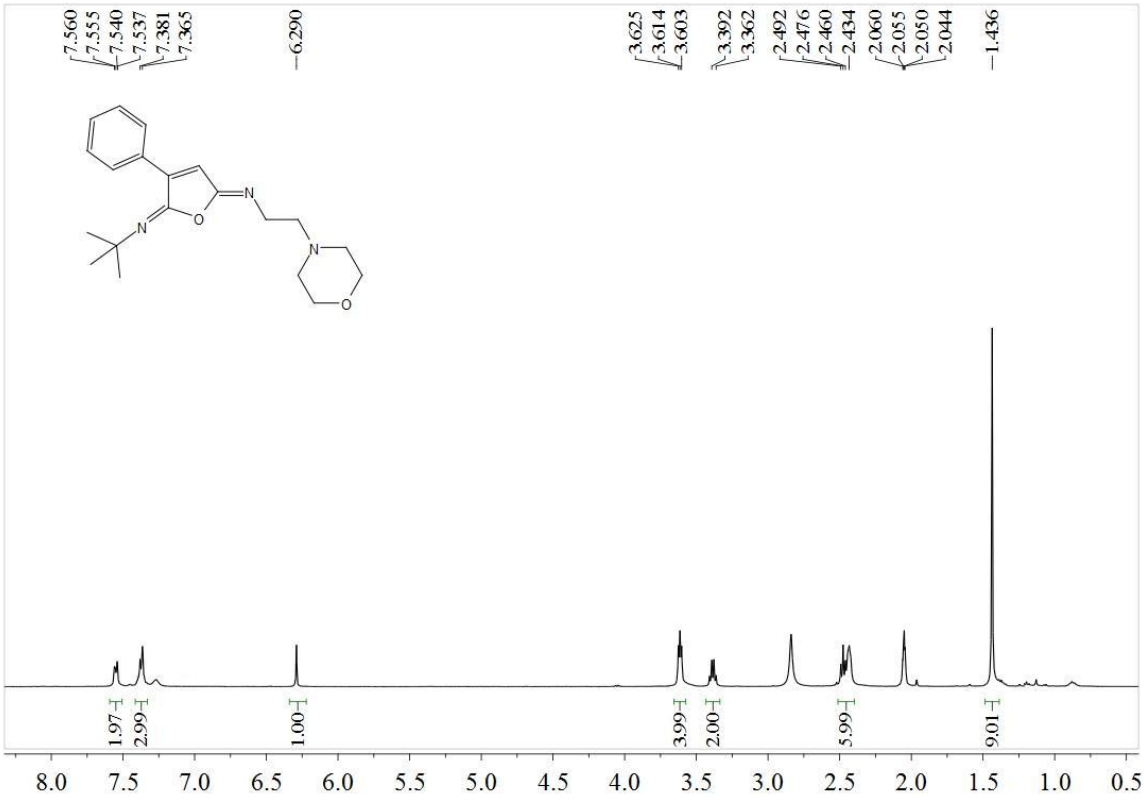

206.000
154.254
150.424
146.059
131.013
130.794
129.758
129.140
129.140
128.210
127.723
125.955
124.155
56.336
29.990
29.797
29.605
29.412
29.220
18.273
154.254
150.424
146.117
146.059
131.013
129.758
129.140
129.140
128.210
127.723
125.955
124.155
150
140
130
30.374
30.256
30.256
30.182
29.990
29.797
29.605
29.412
29.220
30.0
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10

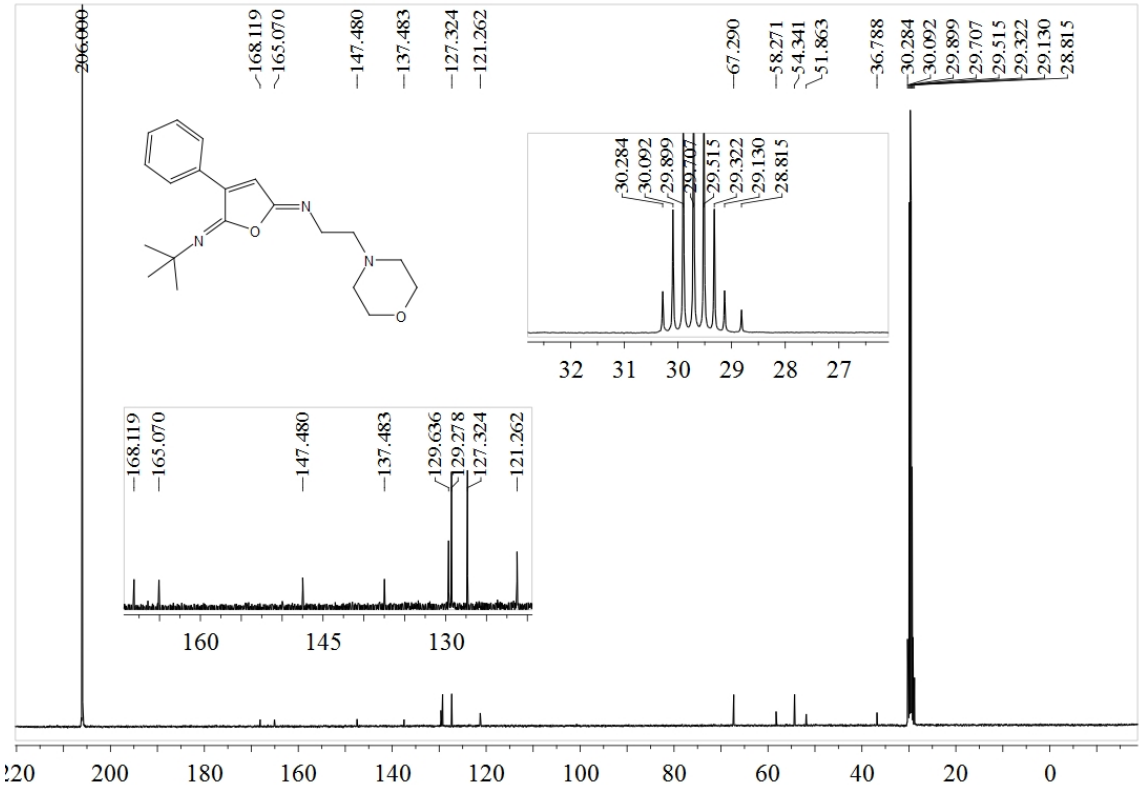

8.121
8.112
8.103
8.097
7.426
7.417
7.411
6.999
2.060
2.055
2.050
2.044
2.039
1.821
1.456
1.009
2.00
3.00
1.00
2.02
14.99
9.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

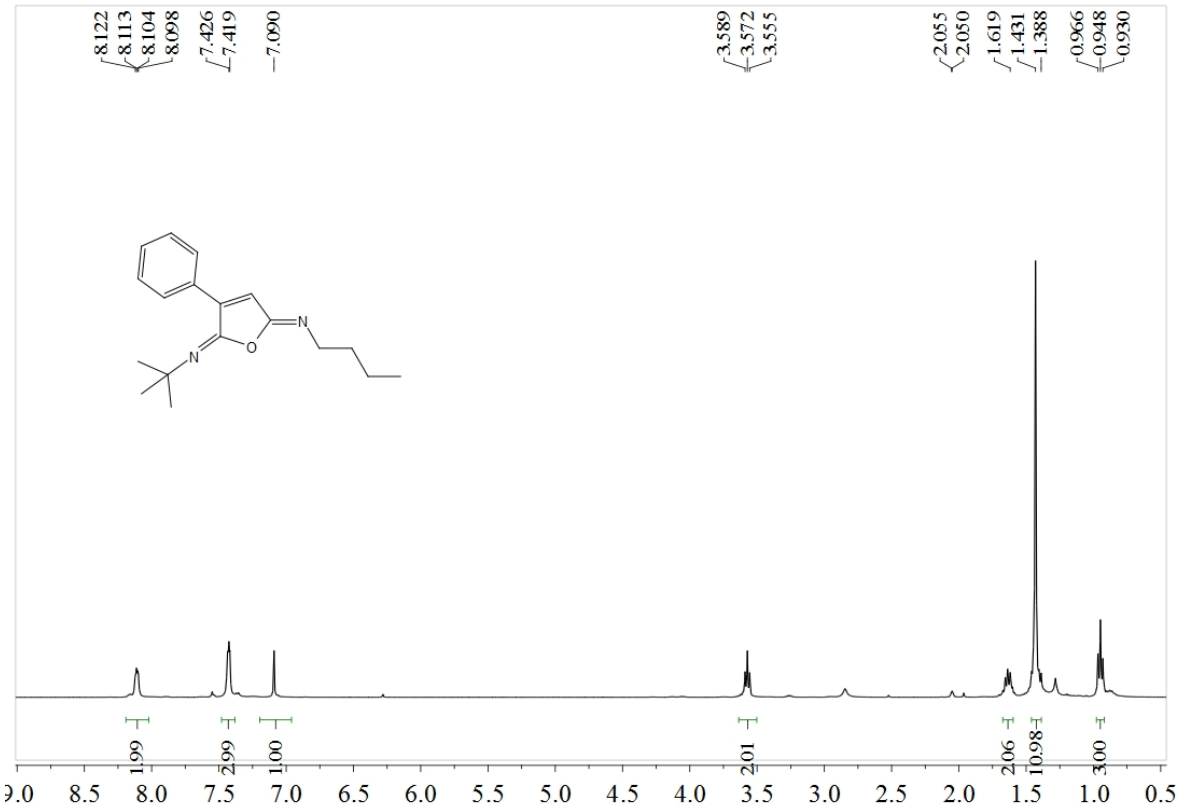

206.000
151.147
150.534
143.403
131.238
130.414
129.560
129.007
128.739
59.126
55.765
55.623
32.021
30.177
29.985
29.792
29.600
29.407
29.215
32.569
32.021
31.455
30.647
30.370
30.177
29.985
29.792
29.600
29.407
29.215
33 32 31 30 29 28
210 190 170 150 130 110 90 80 70 60 50 40 30 20

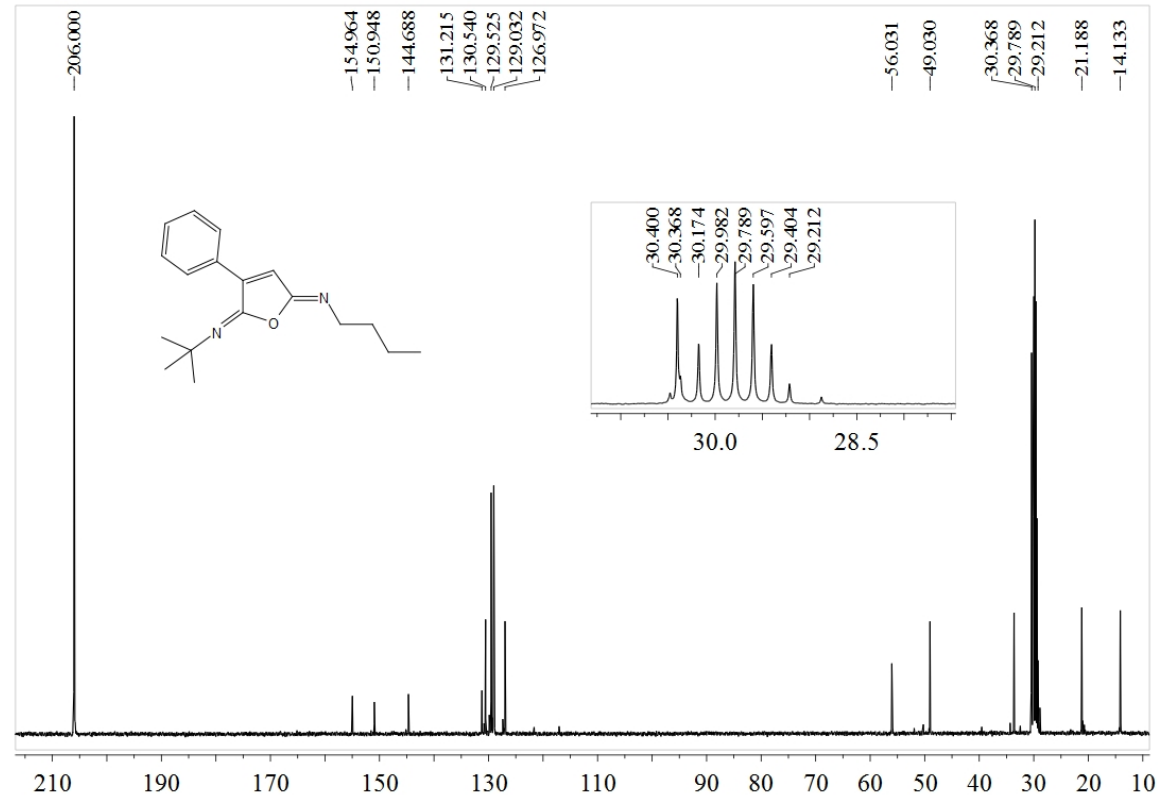

8.116
8.112
8.107
8.101
7.444
7.439
7.435
7.428
7.082
3.831
3.822
3.816
3.807
3.798
3.783
3.774
2.067
2.061
2.056
1.790
1.470
1.452
1.447
1.435
1.401
1.394
1.96
3.02
1.00
0.99
4.00
15.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

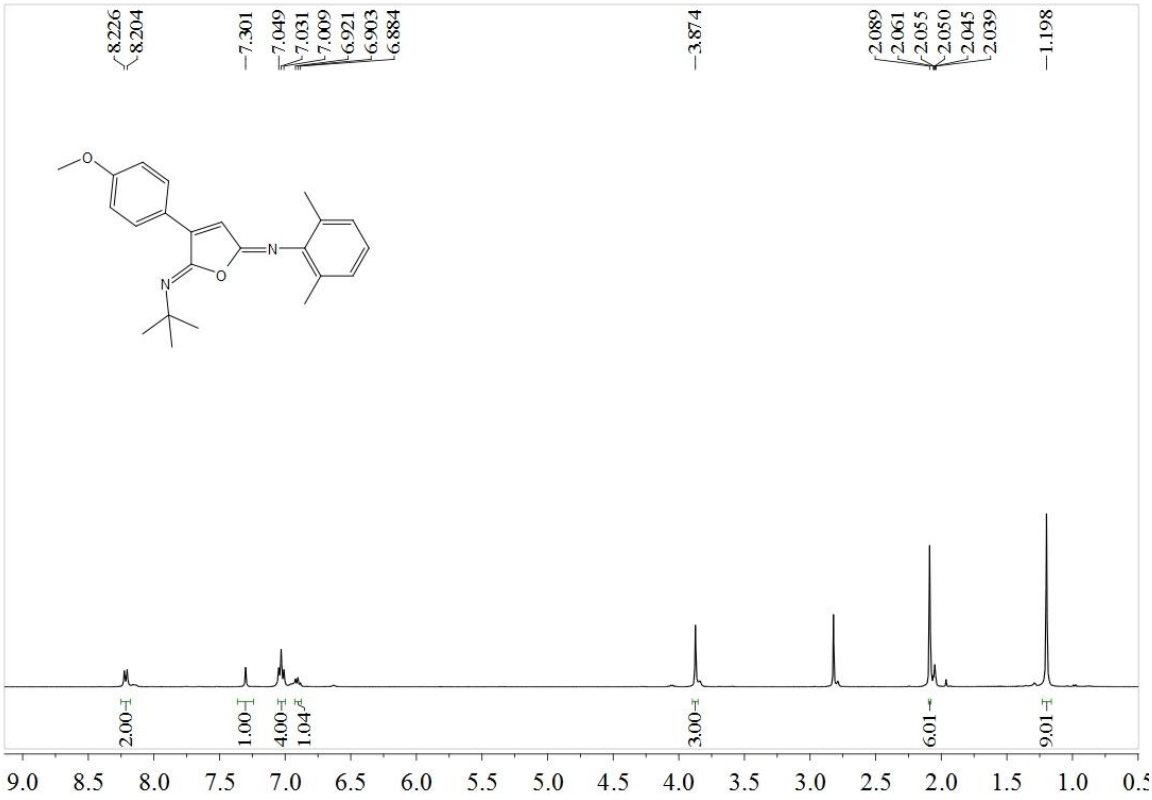

206.000
153.593
150.946
144.427
131.078
130.399
129.378
128.900
127.016
57.768
55.887
34.528
29.853
29.468
29.083
25.012
30.351
30.238
30.045
29.853
29.661
29.468
29.276
29.083
30.5
30.0
29.5
29.0
210
190
170
150
130
110
90
80
70
60
50
40
30
20

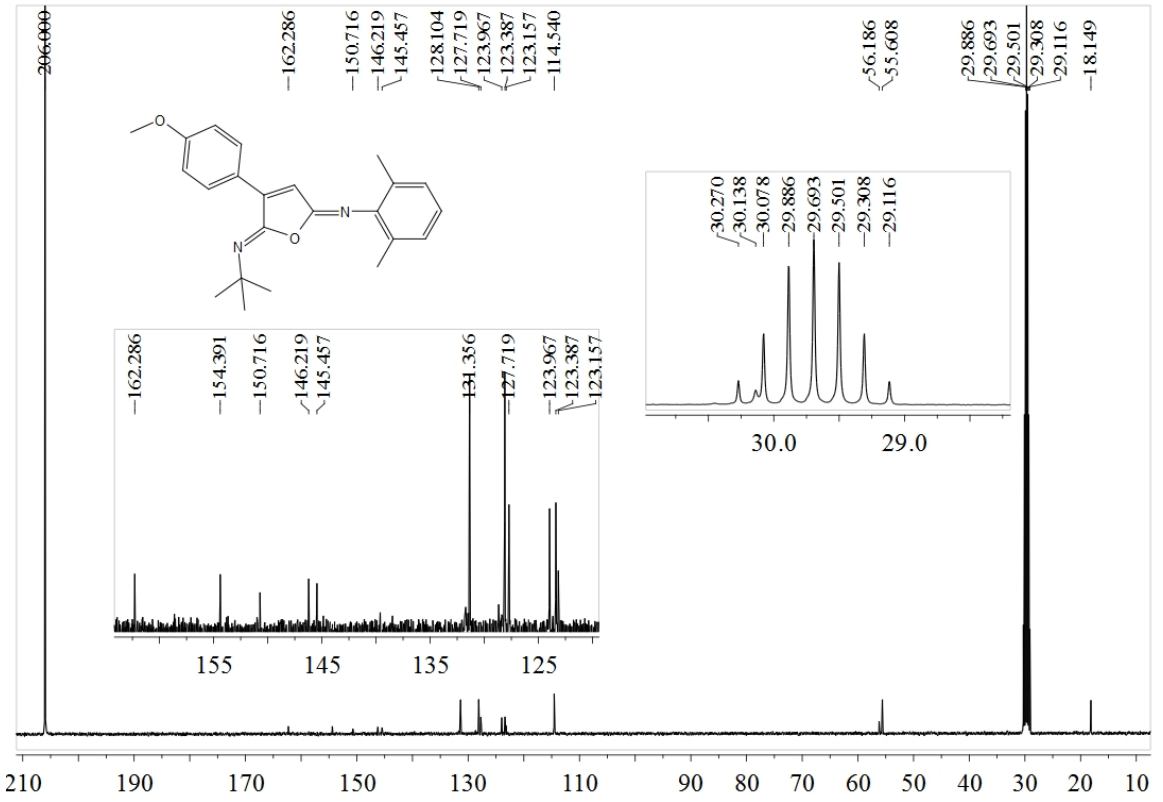

7.560
7.555
7.540
7.537
7.381
7.365
6.290
3.625
3.614
3.603
3.392
3.362
2.492
2.476
2.460
2.434
2.060
2.055
2.050
2.044
1.436
1.97
2.99
1.00
3.99
2.00
5.99
9.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

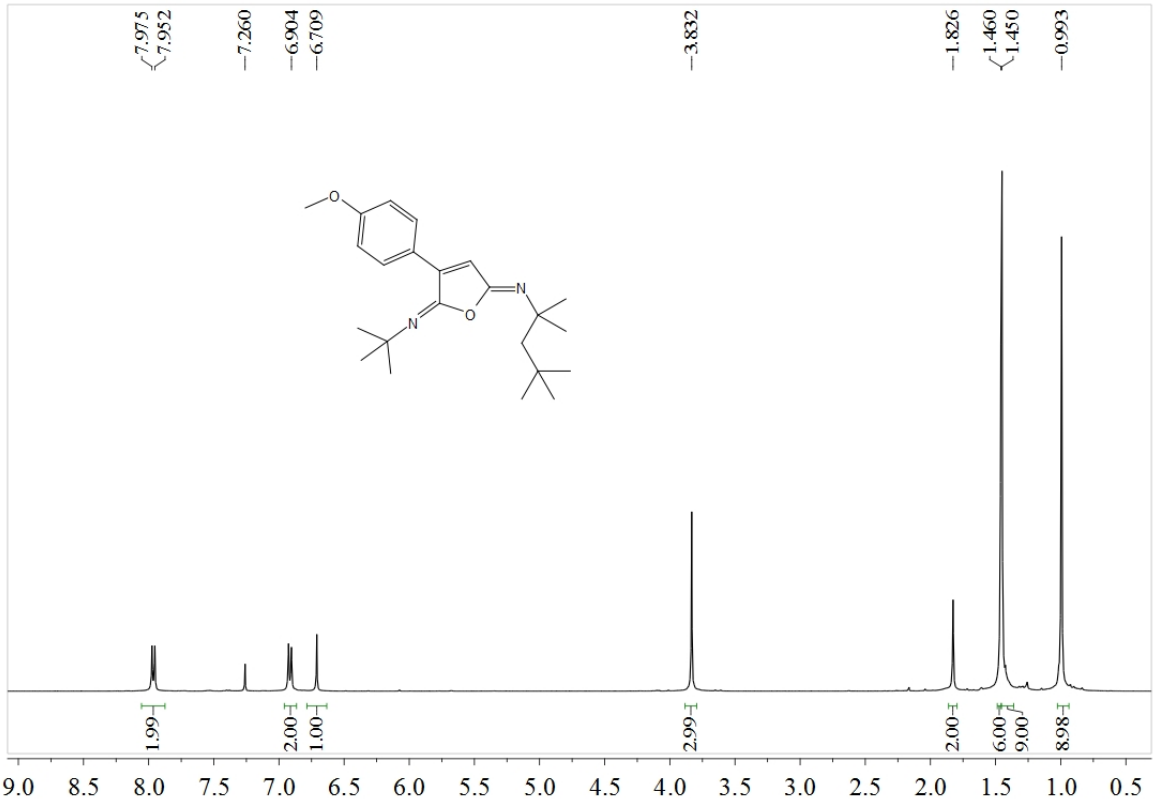

206.000
168.119
165.070
147.480
137.483
127.324
121.262
67.290
58.271
54.341
51.863
36.788
30.284
30.092
29.899
29.707
29.515
29.322
29.130
28.815
32
31
30
29
28
27
129.636
29.278
160
145
130
220
200
180
160
140
120
100
80
60
40
20
0

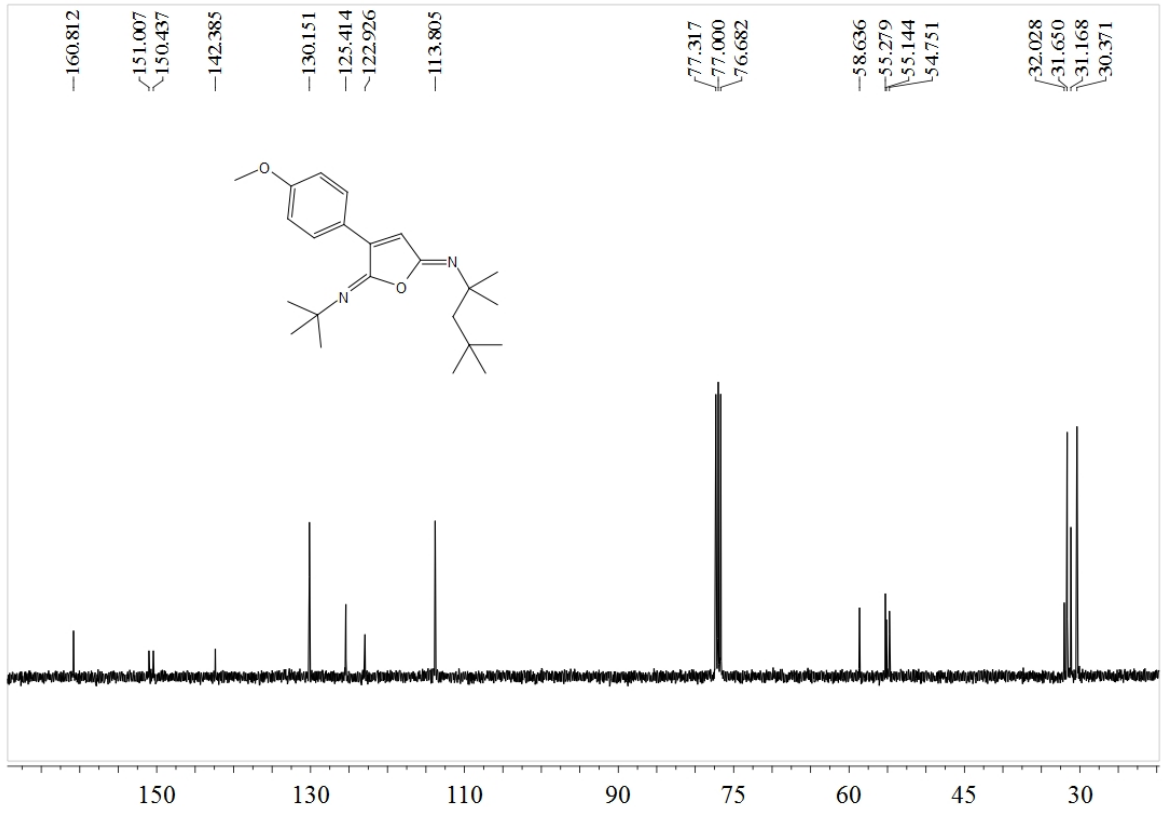

8.122
8.113
8.104
8.098
7.426
7.419
7.090
3.589
3.572
3.555
2.055
2.050
1.619
1.431
1.388
0.966
0.948
0.930
1.99
2.99
1.00
2.01
2.06
10.98
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

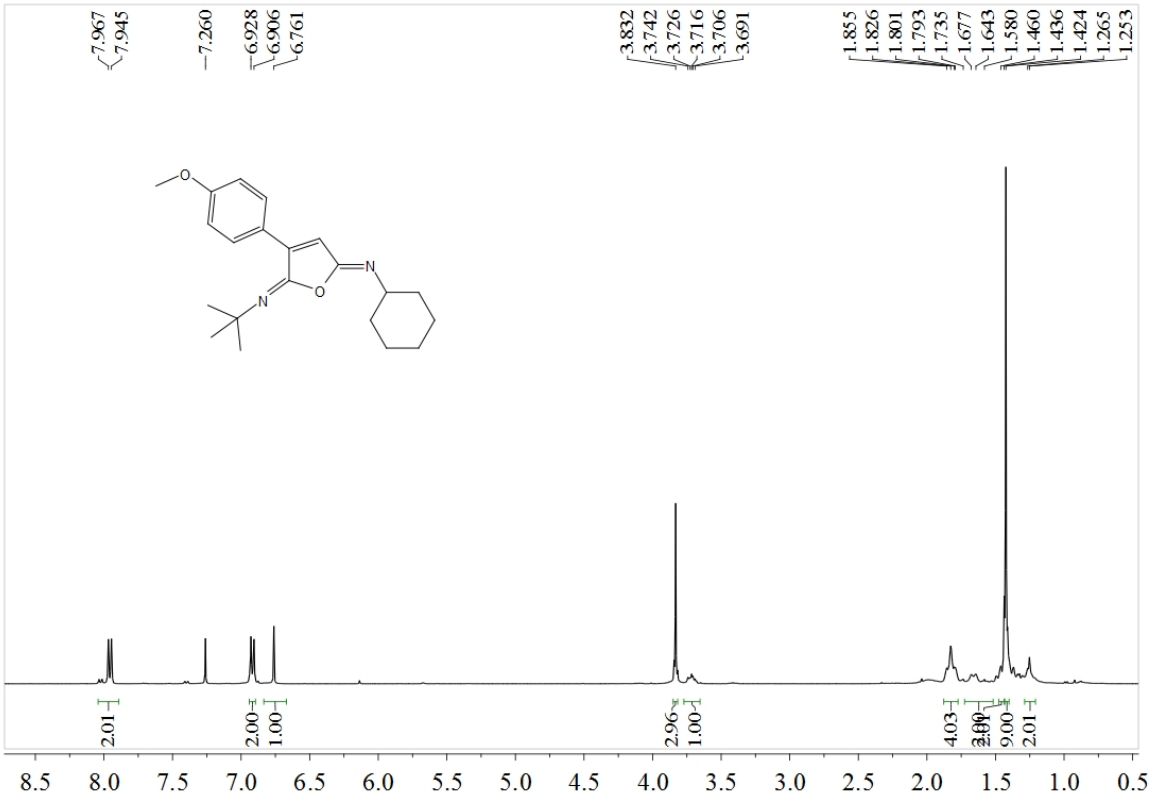

206.000
154.964
150.948
144.688
131.215
130.540
129.525
129.032
126.972
56.031
49.030
30.368
29.789
29.212
21.188
14.133
30.400
30.368
30.174
29.982
29.789
29.597
29.404
29.212
30.0
28.5
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10

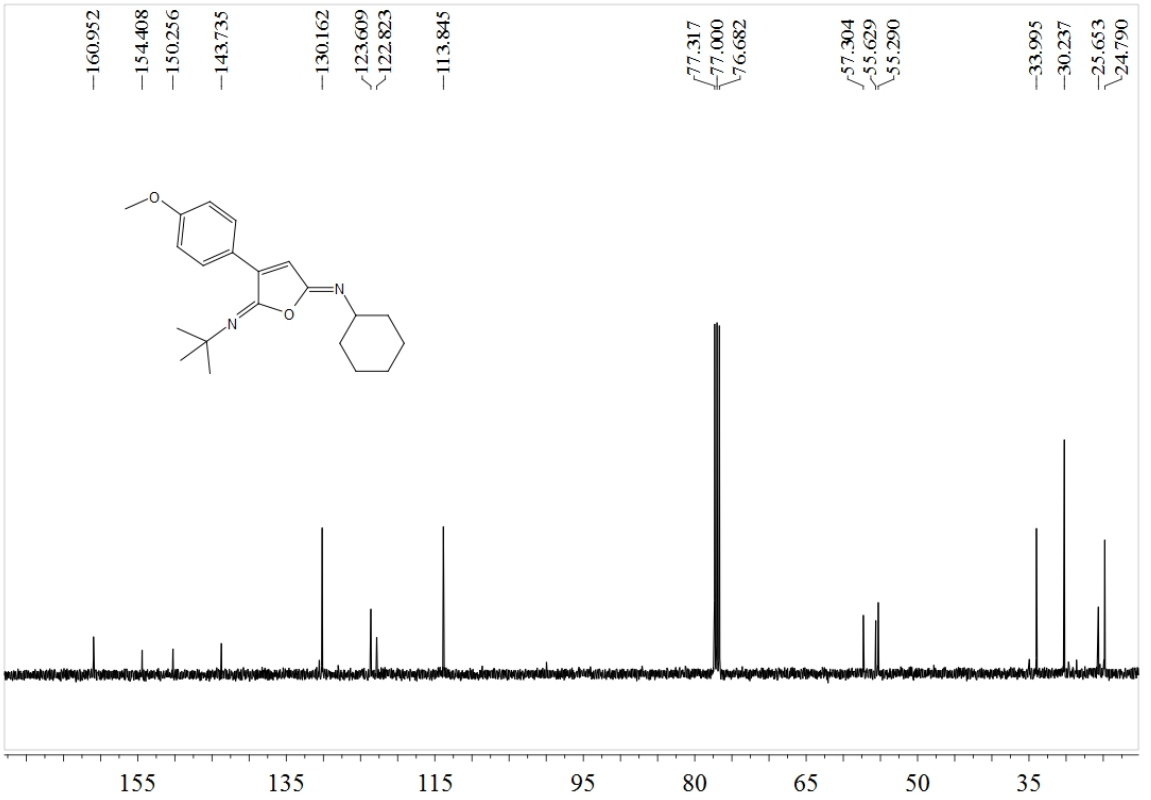

8.226
8.204
7.301
7.049
7.031
7.009
6.921
6.903
6.884
3.874
2.089
2.061
2.055
2.050
2.045
2.039
1.198
2.00
1.00
4.00
1.04
3.00
6.01
9.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

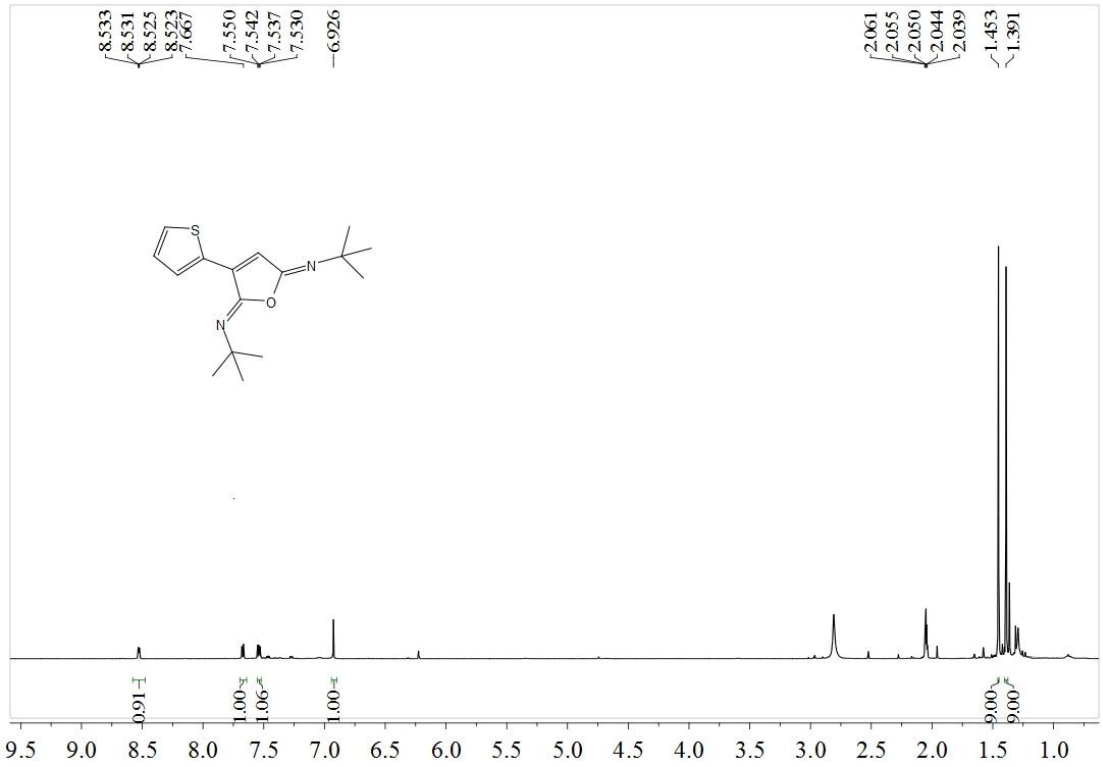

206.000
162.286
150.716
146.219
145.457
128.104
127.719
123.967
123.387
123.157
114.540
56.186
55.608
29.886
29.693
29.501
29.308
29.116
18.149
30.270
30.138
30.078
29.886
29.693
29.501
29.308
29.116
30.0
29.0
154.391
131.356
155
145
135
125
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10

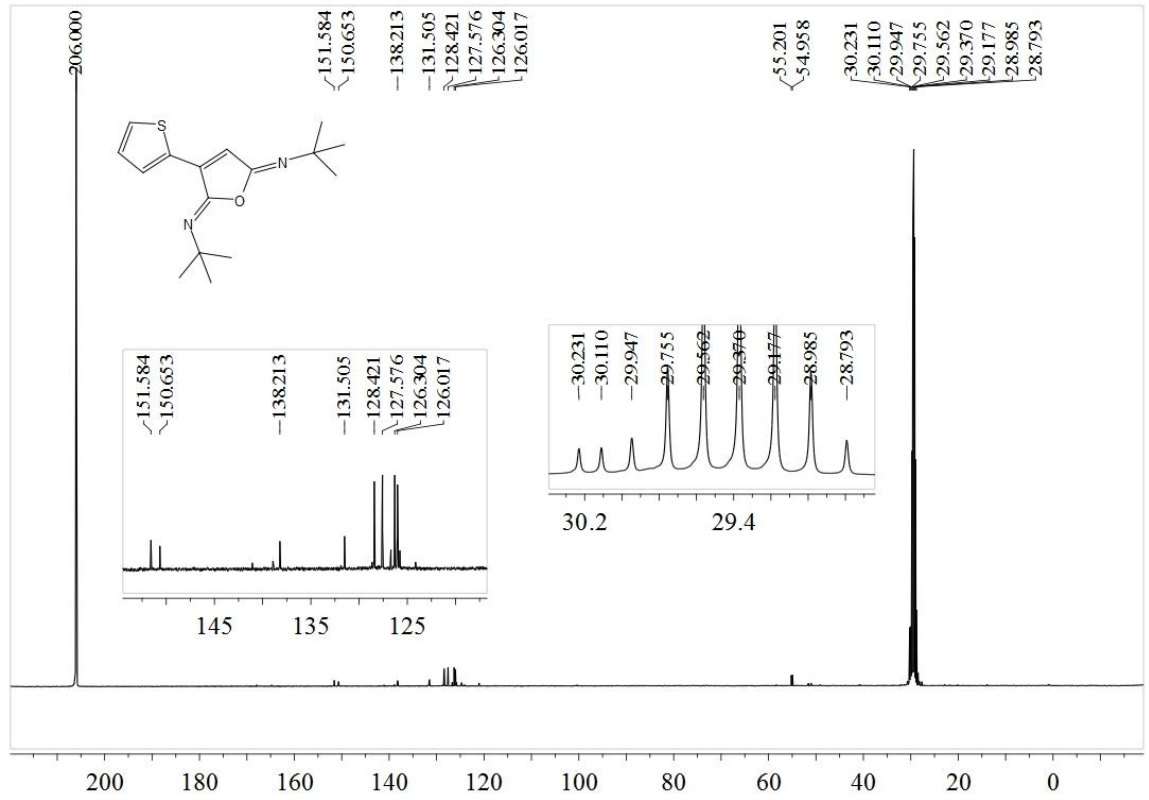

7.975
7.952
7.260
6.904
6.709
3.832
1.826
1.460
1.450
0.993
1.99
2.00
1.00
2.99
2.00
6.00
9.00
8.98
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

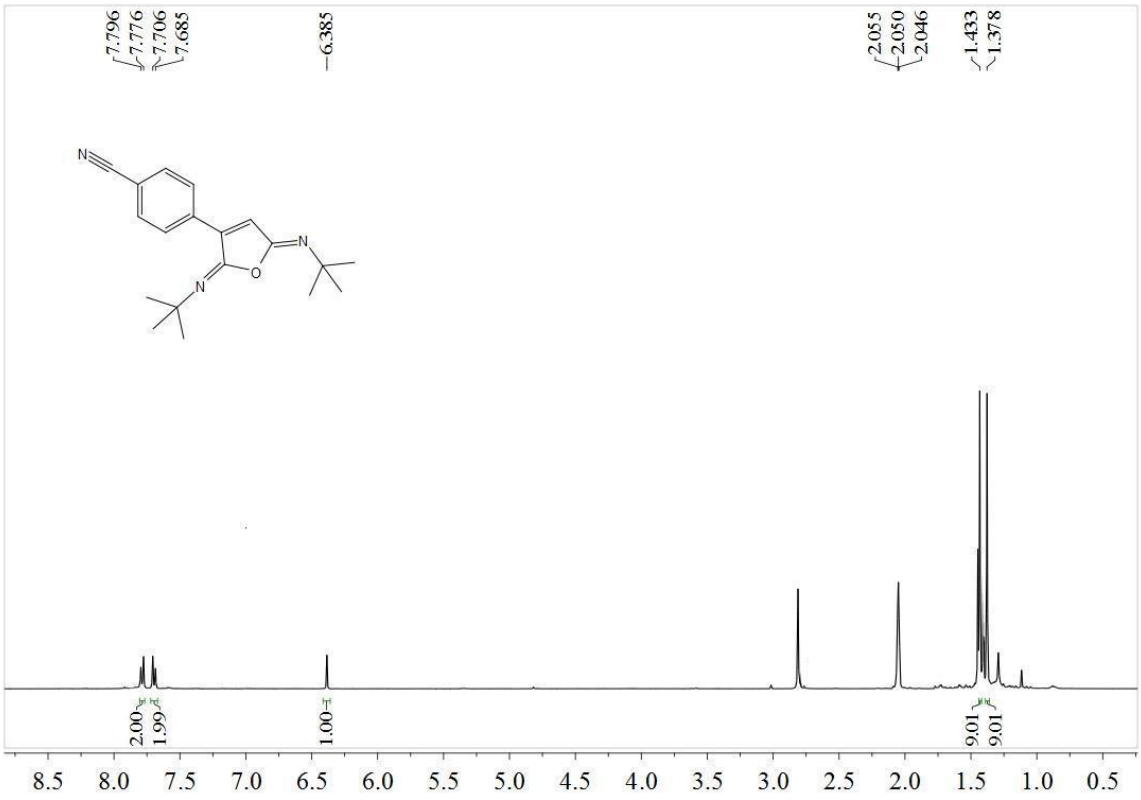

160.812
151.007
150.437
142.385
130.151
125.414
122.926
113.805
77.317
77.000
76.682
58.636
55.279
55.144
54.751
32.028
31.650
31.168
30.371
150 130 110 90 75 60 45 30

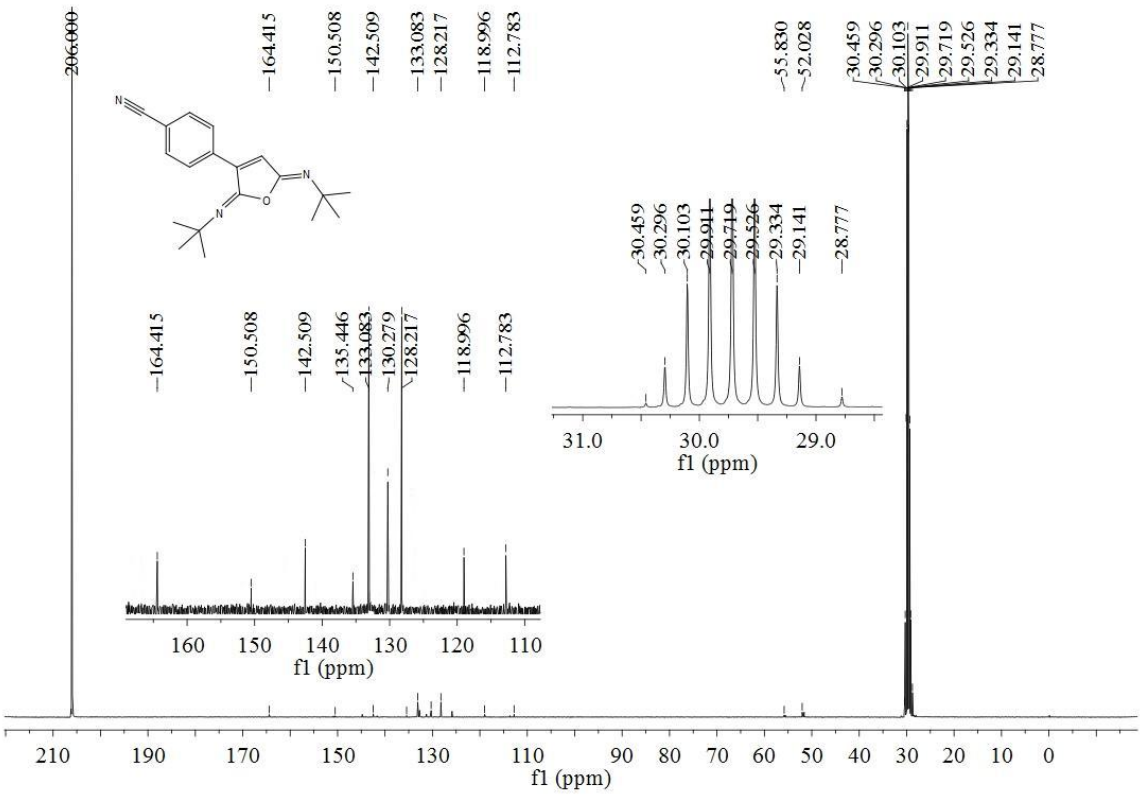

7.967
7.945
7.260
6.928
6.906
6.761
3.832
3.742
3.726
3.716
3.706
3.691
1.855
1.826
1.801
1.793
1.735
1.677
1.643
1.580
1.460
1.436
1.424
1.265
1.253
2.01
2.00
1.00
2.96
1.00
4.03
9.00
2.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

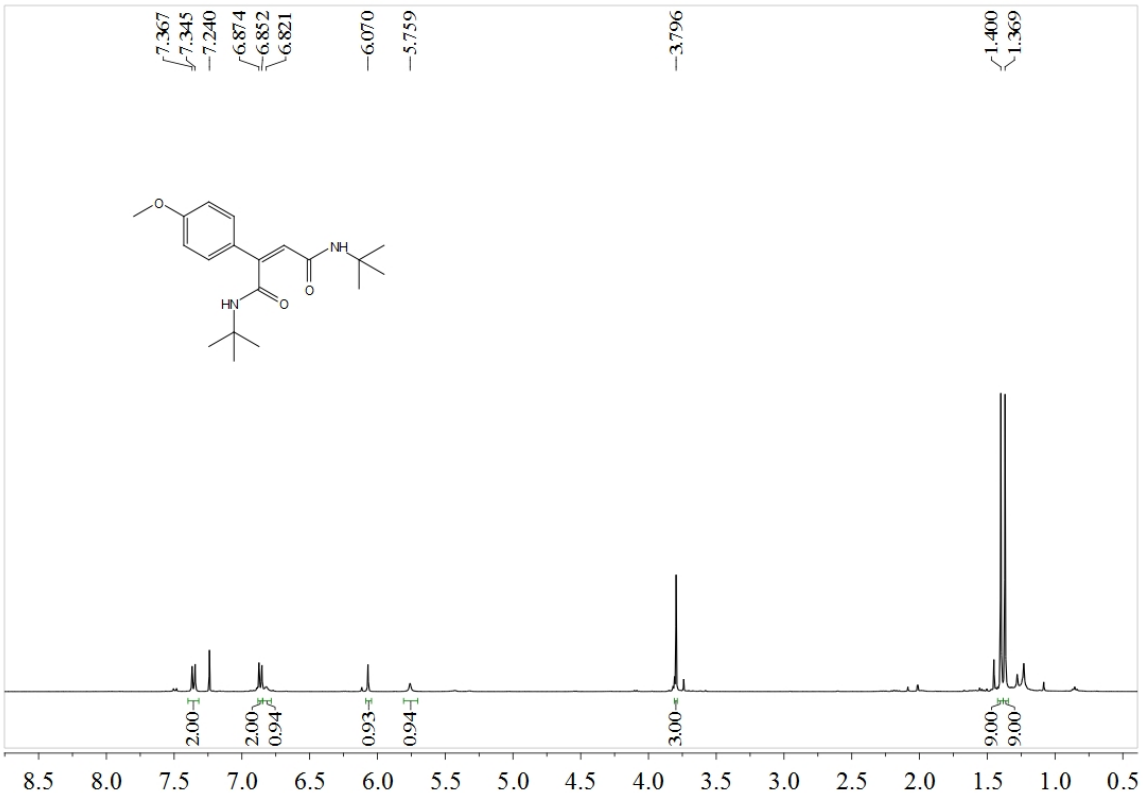

160.952
154.408
150.256
143.735
130.162
123.609
122.823
113.845
77.317
77.000
76.682
57.304
55.629
55.290
33.995
30.237
25.653
24.790
155
135
115
95
80
65
50
35

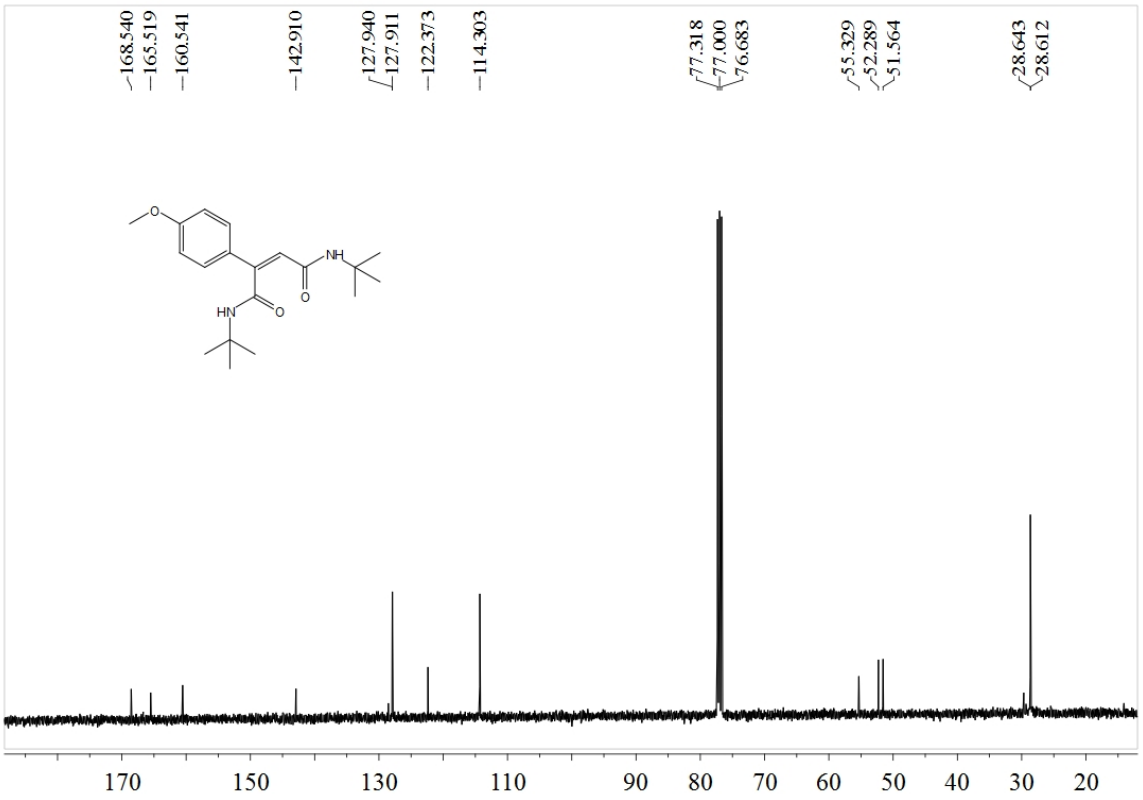

8.533
8.531
8.525
8.523
7.667
7.550
7.542
7.537
7.530
6.926
2.061
2.055
2.050
2.044
2.039
1.453
1.391
0.91
1.00
1.06
1.00
9.00
9.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0

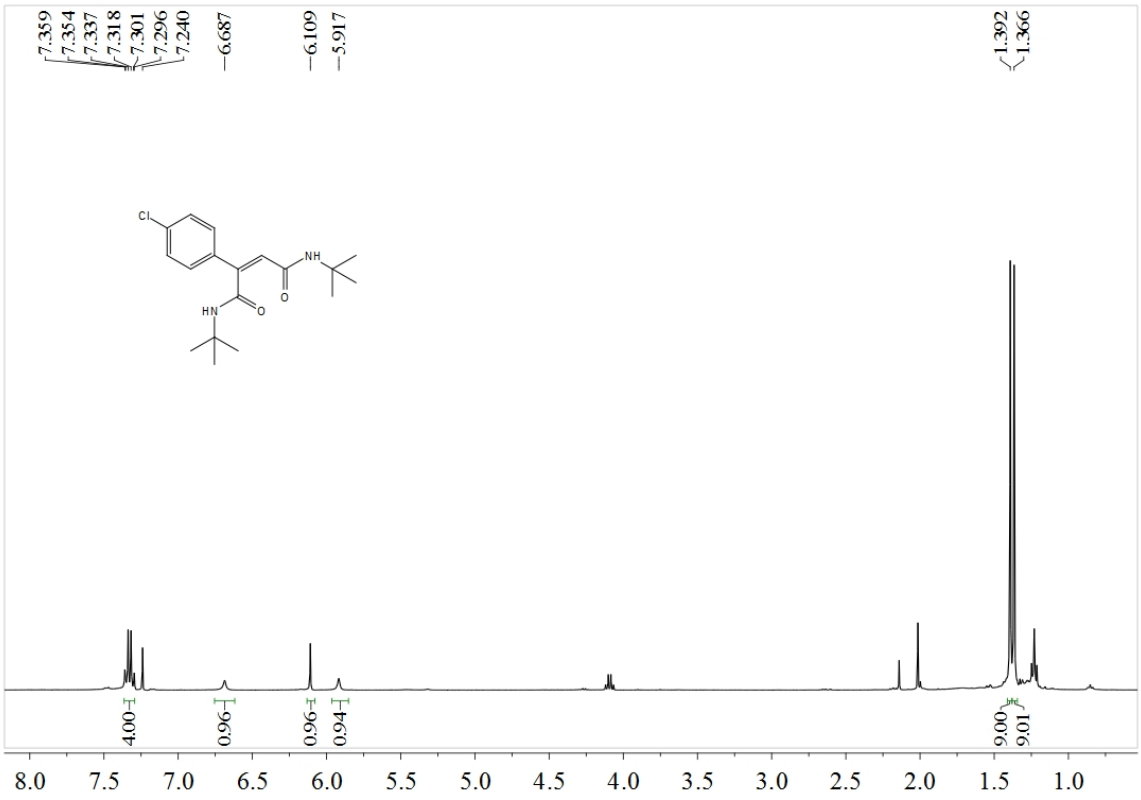

206.000
151.584
150.653
138.213
131.505
128.421
127.576
126.304
126.017
55.201
54.958
30.231
30.110
29.947
29.755
29.562
29.370
29.177
28.985
28.793
145 135 125
30.2 29.4
200 180 160 140 120 100 80 60 40 20 0

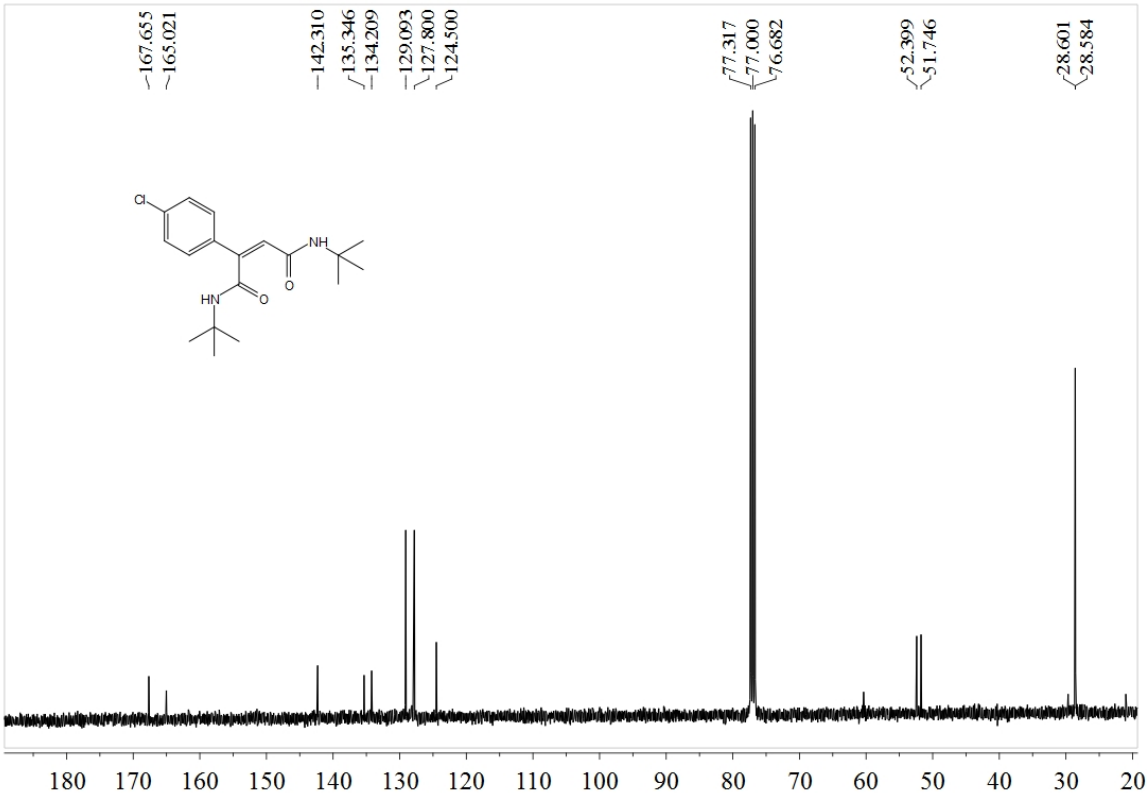

7.796
7.776
7.706
7.685
6.385
2.055
2.050
2.046
1.433
1.378
2.00
1.99
1.00
9.01
9.01
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

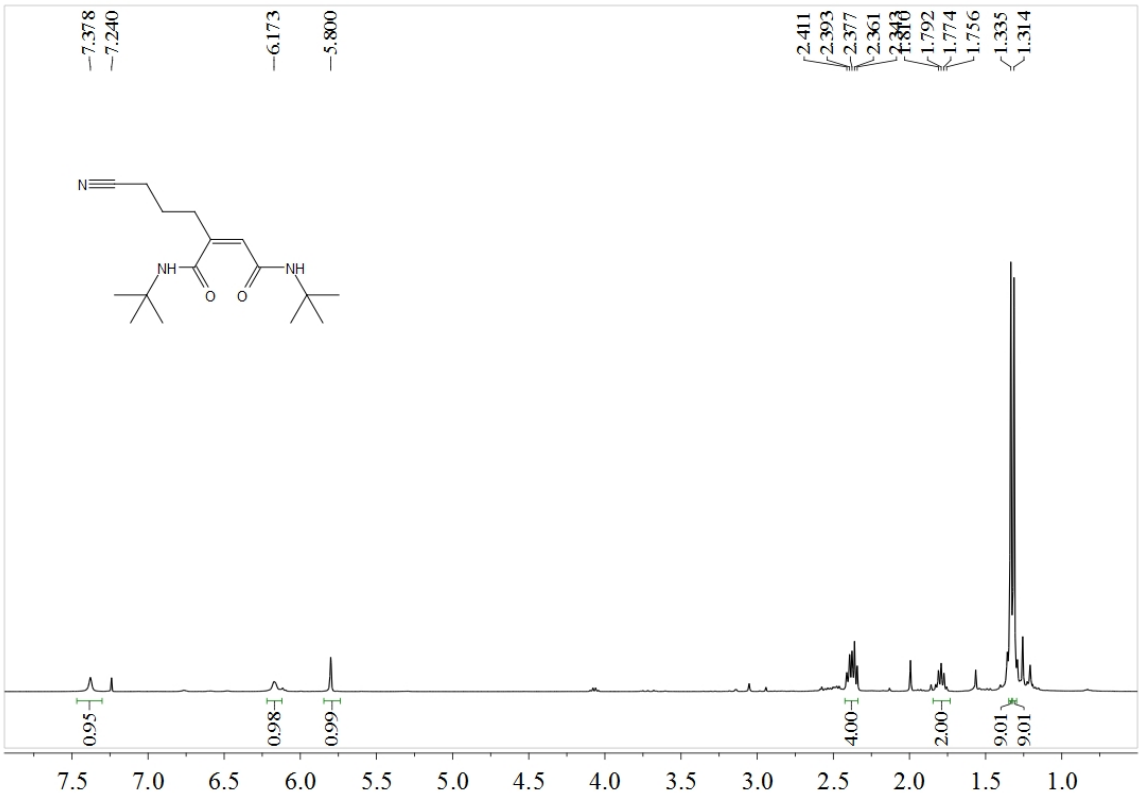

206.000
164.415
150.508
142.509
133.083
128.217
118.996
112.783
55.830
52.028
30.459
30.296
30.103
29.911
29.719
29.526
29.334
29.141
28.777
31.0 30.0 29.0
f1 (ppm)
160 150 140 130 120 110
f1 (ppm)
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

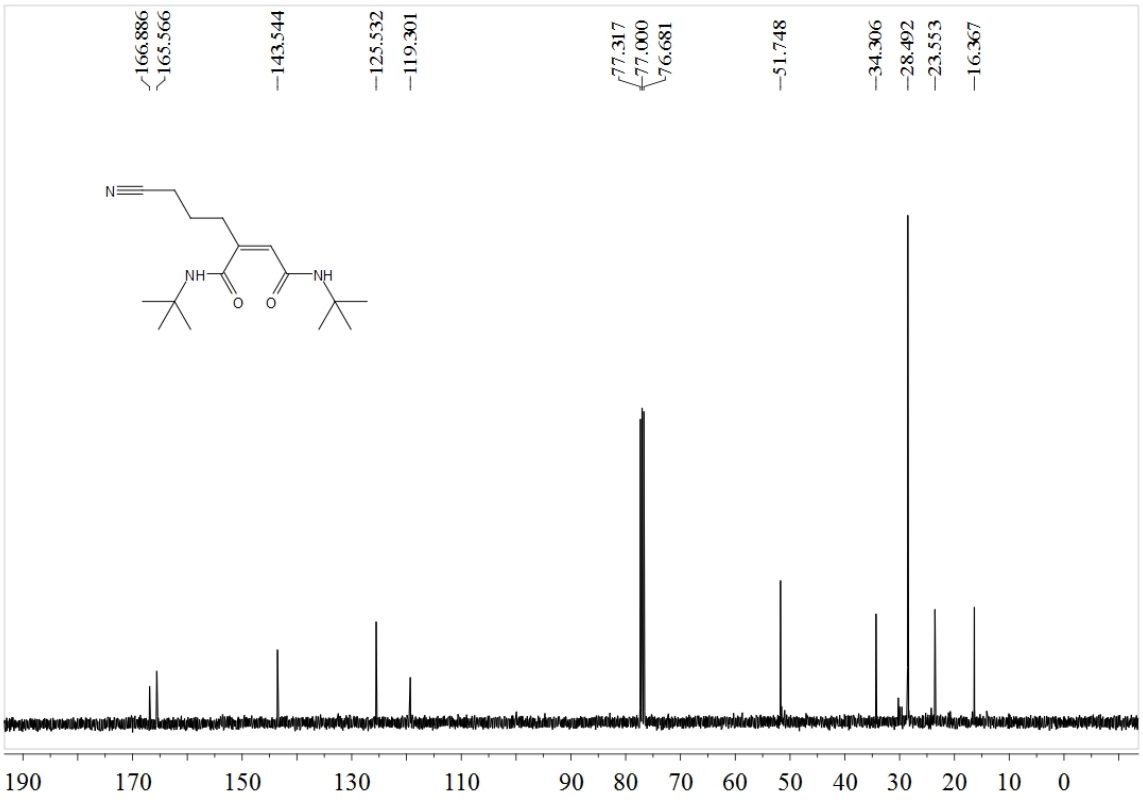

7.367
7.345
7.240
6.874
6.852
6.821
6.070
5.759
3.796
1.400
1.369
O
NH
HN
O
O
2.00
2.00
0.94
0.93
0.94
3.00
9.00
9.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

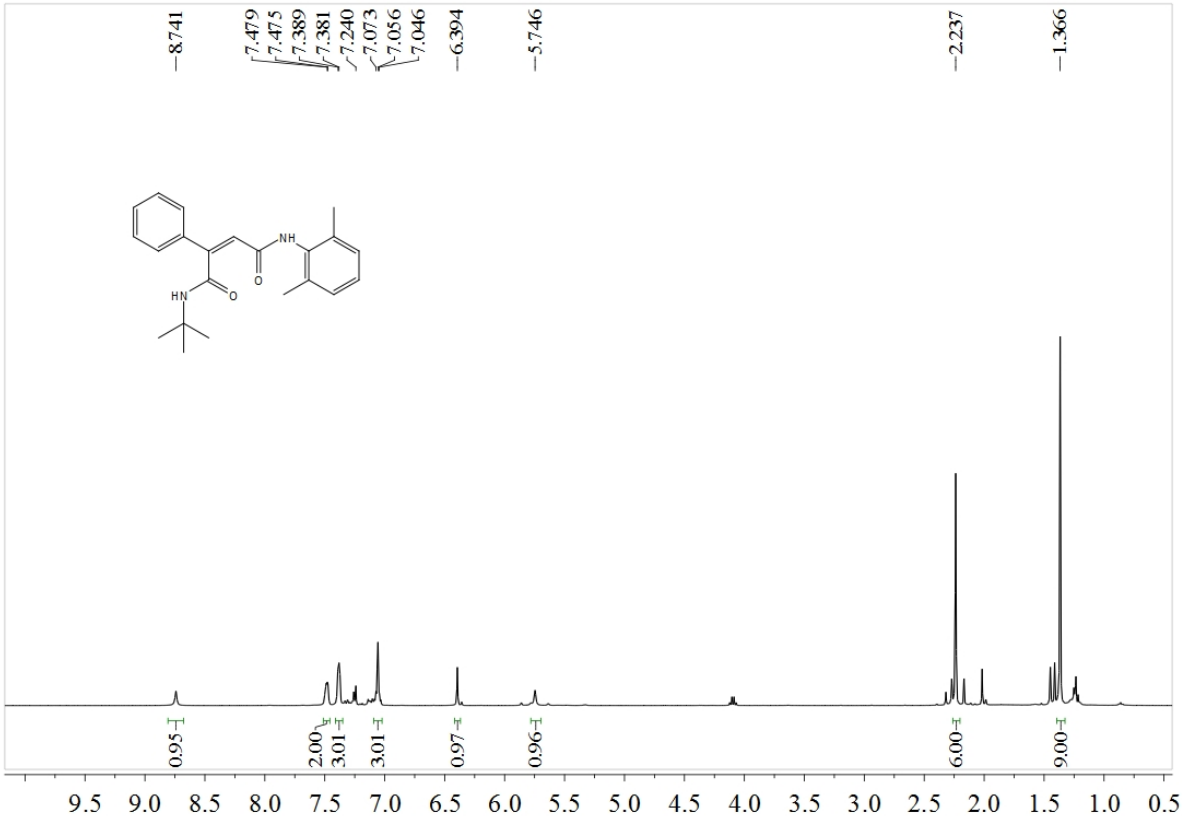

168.540
165.519
160.541
142.910
127.940
127.911
122.373
114.303
77.318
77.000
76.683
55.329
52.289
51.564
28.643
28.612
O
NH
HN
O
O
170
150
130
110
90
80
70
60
50
40
30
20

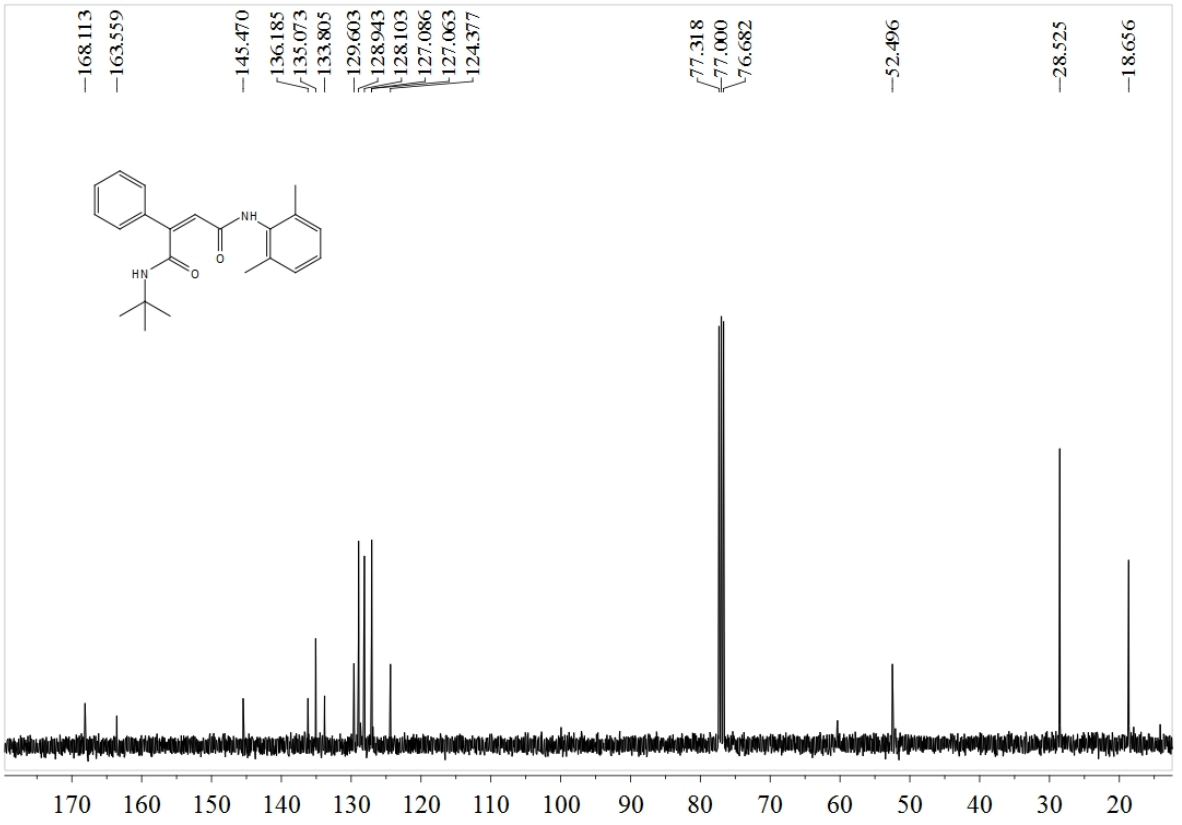

7.359
7.354
7.337
7.318
7.301
7.296
7.240
6.687
6.109
5.917
1.392
1.366
Cl
NH
HN
O
4.00
0.96
0.96
0.94
9.00
9.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

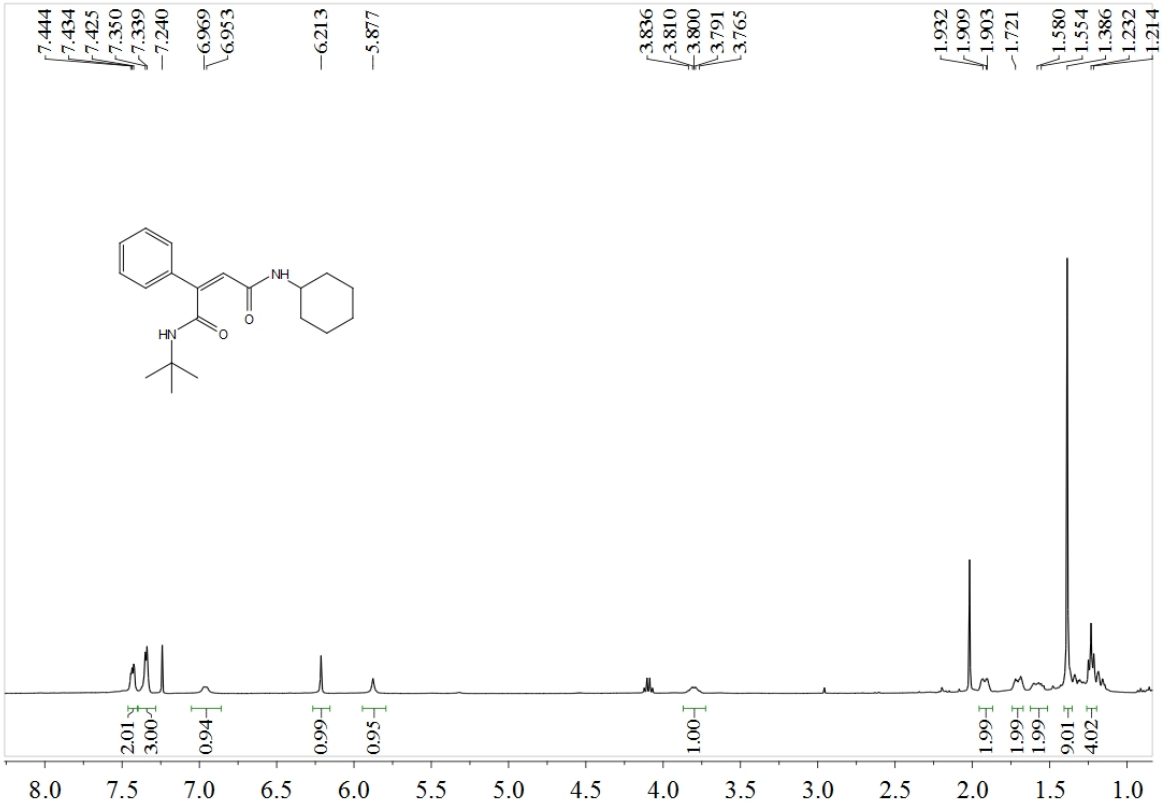

167.655
165.021
142.310
135.346
134.209
129.093
127.800
124.500
77.317
77.000
76.682
52.399
51.746
28.601
28.584
Cl
NH
HN
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

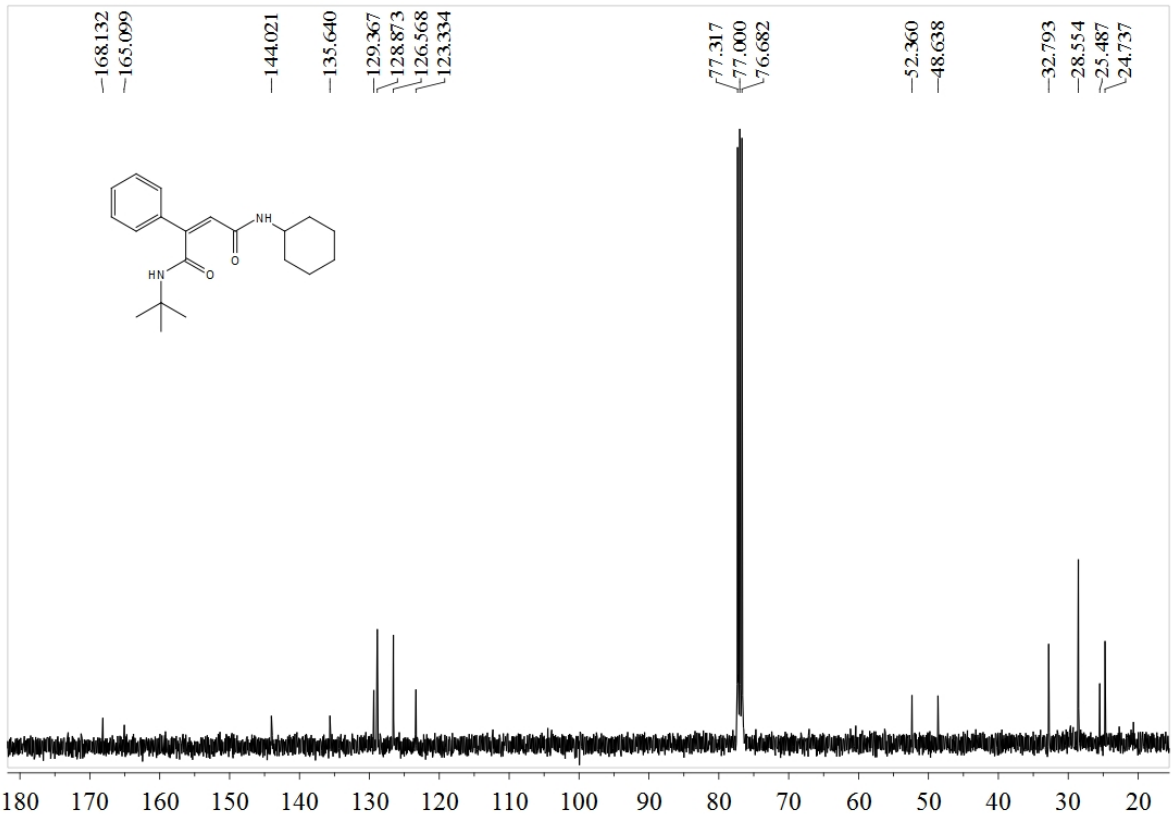

7.378
7.240
6.173
5.800
2.411
2.393
2.377
2.361
1.792
1.774
1.756
1.335
1.314
N
NH
O
O
NH
0.95
0.98
0.99
4.00
2.00
9.01
9.01
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

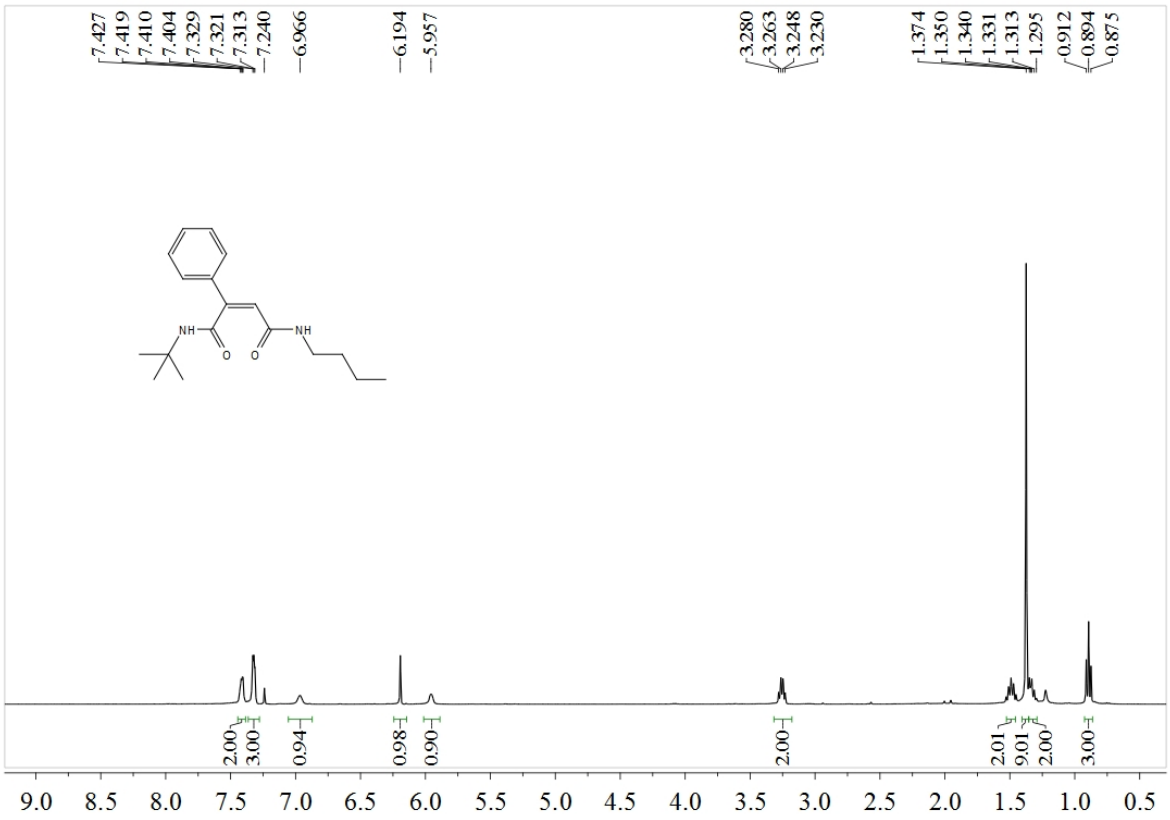

166.886
165.566
143.544
125.532
119.301
77.317
77.000
76.681
51.748
34.306
28.492
23.553
16.367
N
NH
O
O
NH
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

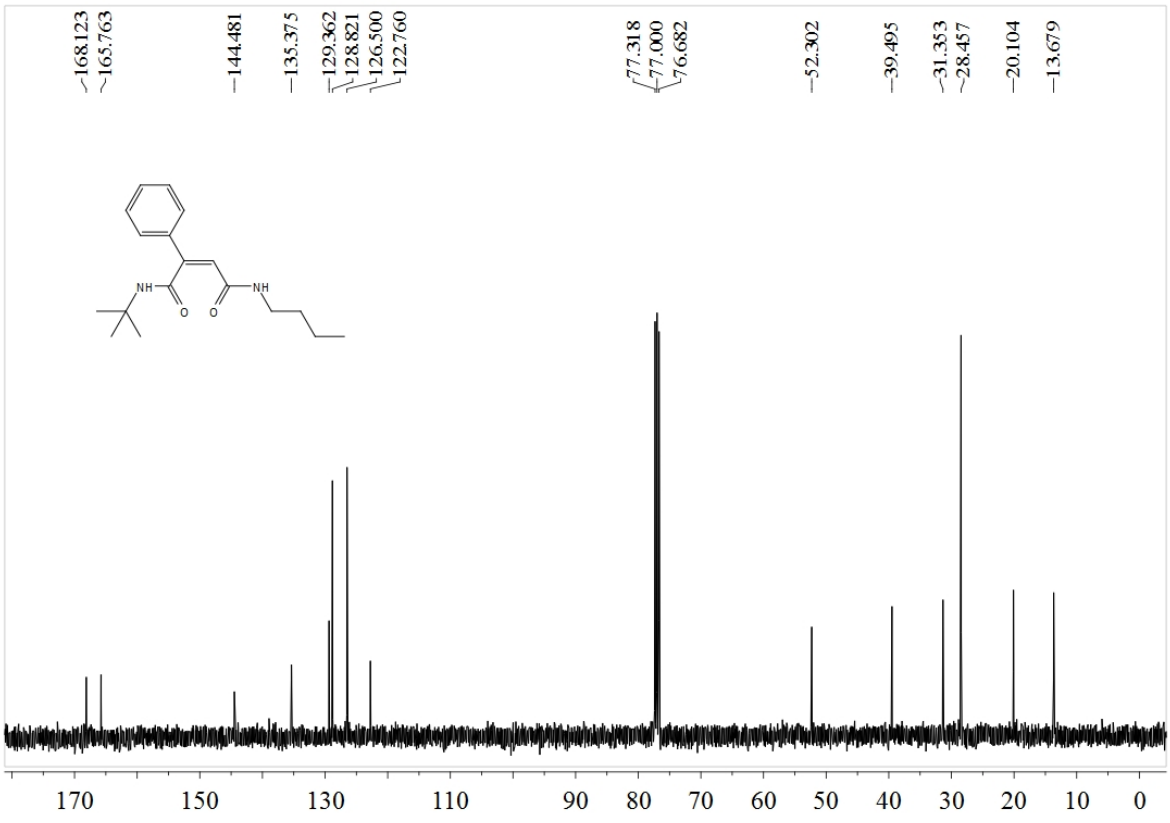

8.741
7.479
7.475
7.389
7.381
7.240
7.073
7.056
7.046
6.394
5.746
2.237
1.366
NH
O
HN
O
0.95
2.00
3.01
3.01
0.97
0.96
6.00
9.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

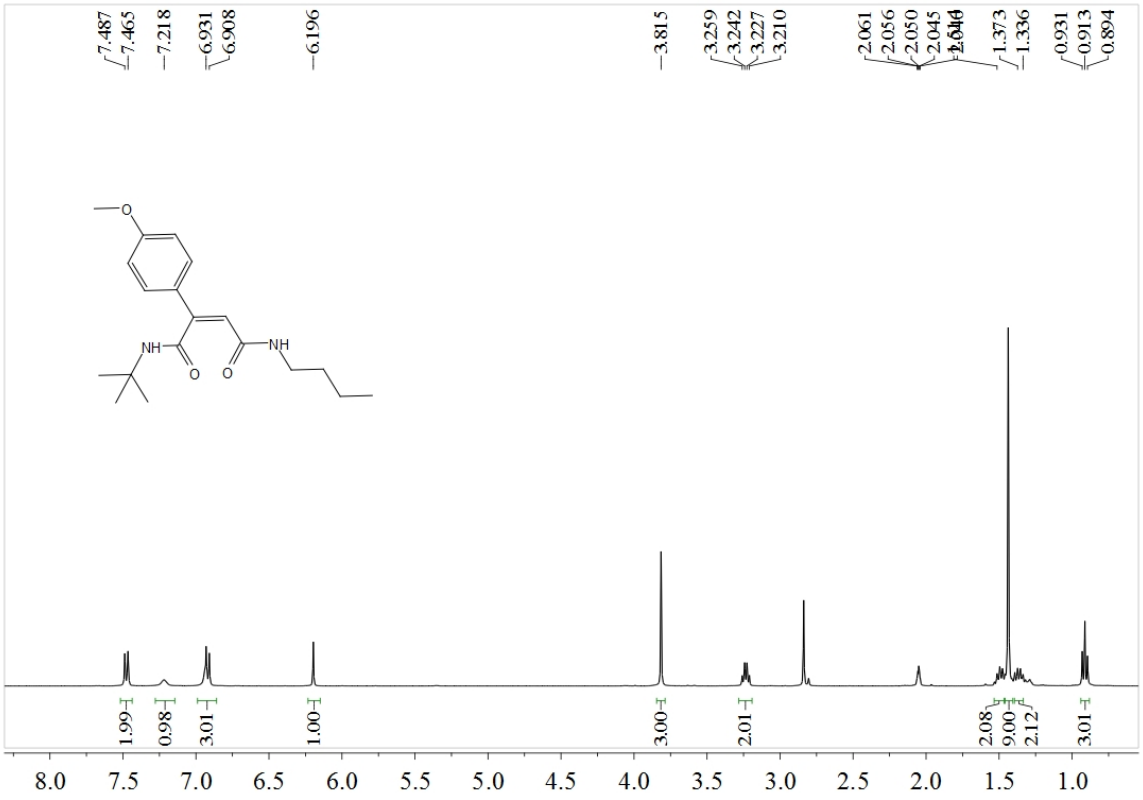

168.113
163.559
145.470
136.185
135.073
133.805
129.603
128.943
128.103
127.086
127.063
124.377
77.318
77.000
76.682
52.496
28.525
18.656
NH
O
HN
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20

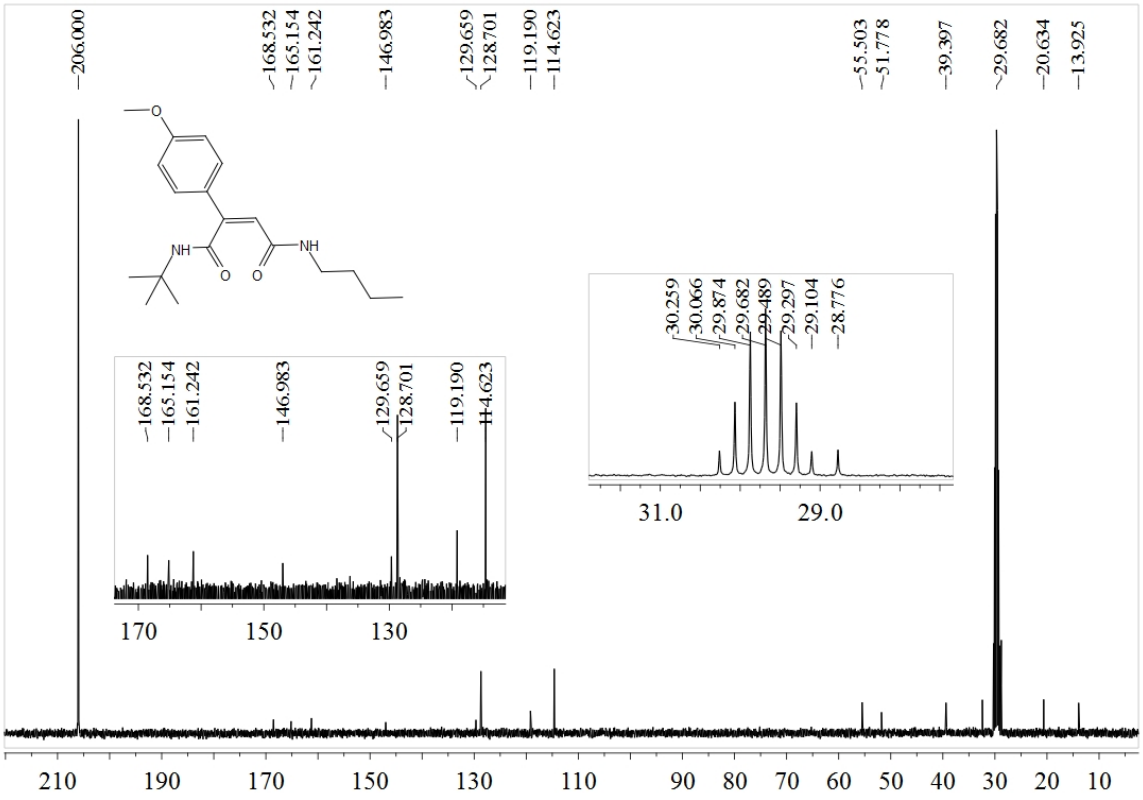

7.444
7.434
7.425
7.350
7.339
7.240
6.969
6.953
6.213
5.877
3.836
3.810
3.800
3.791
3.765
1.932
1.909
1.903
1.721
1.580
1.554
1.386
1.232
1.214
NH
HN
O
O
2.01
3.00
0.94
0.99
0.95
1.00
1.99
1.99
1.99
9.01
4.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0

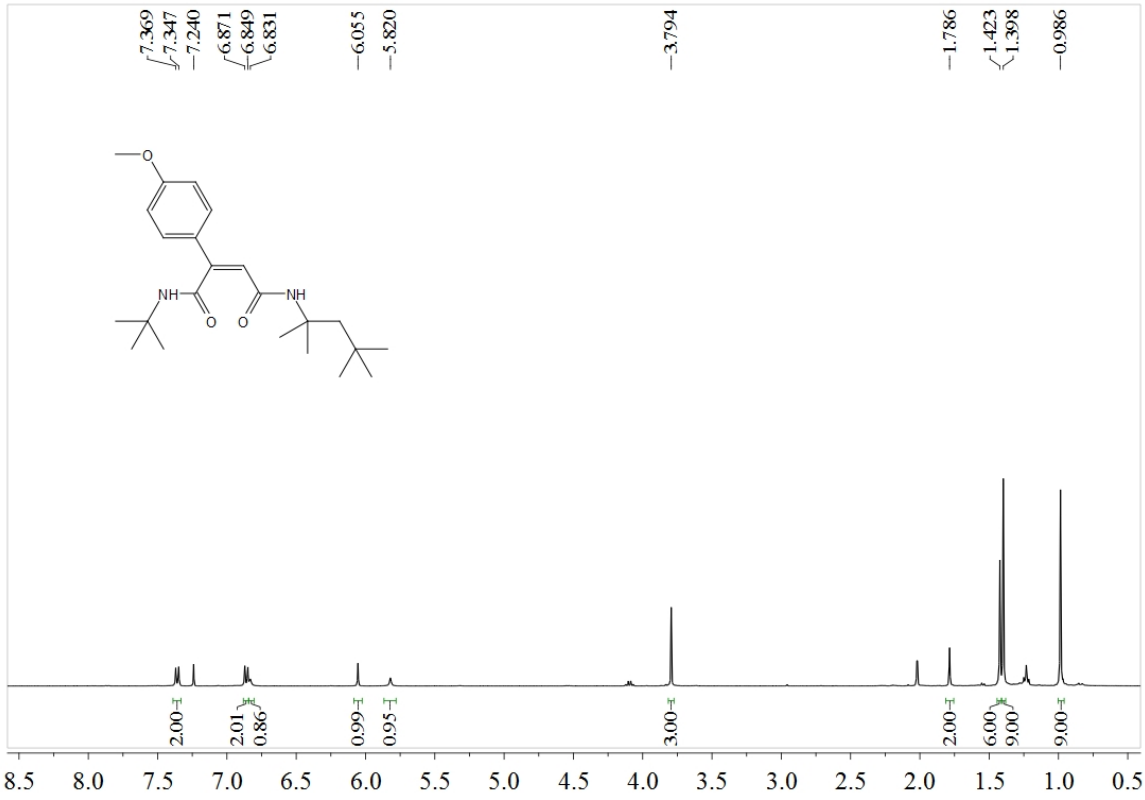

168.132
165.099
144.021
135.640
129.367
128.873
126.568
123.334
77.317
77.000
76.682
52.360
48.638
32.793
28.554
25.487
24.737
NH
HN
O
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20

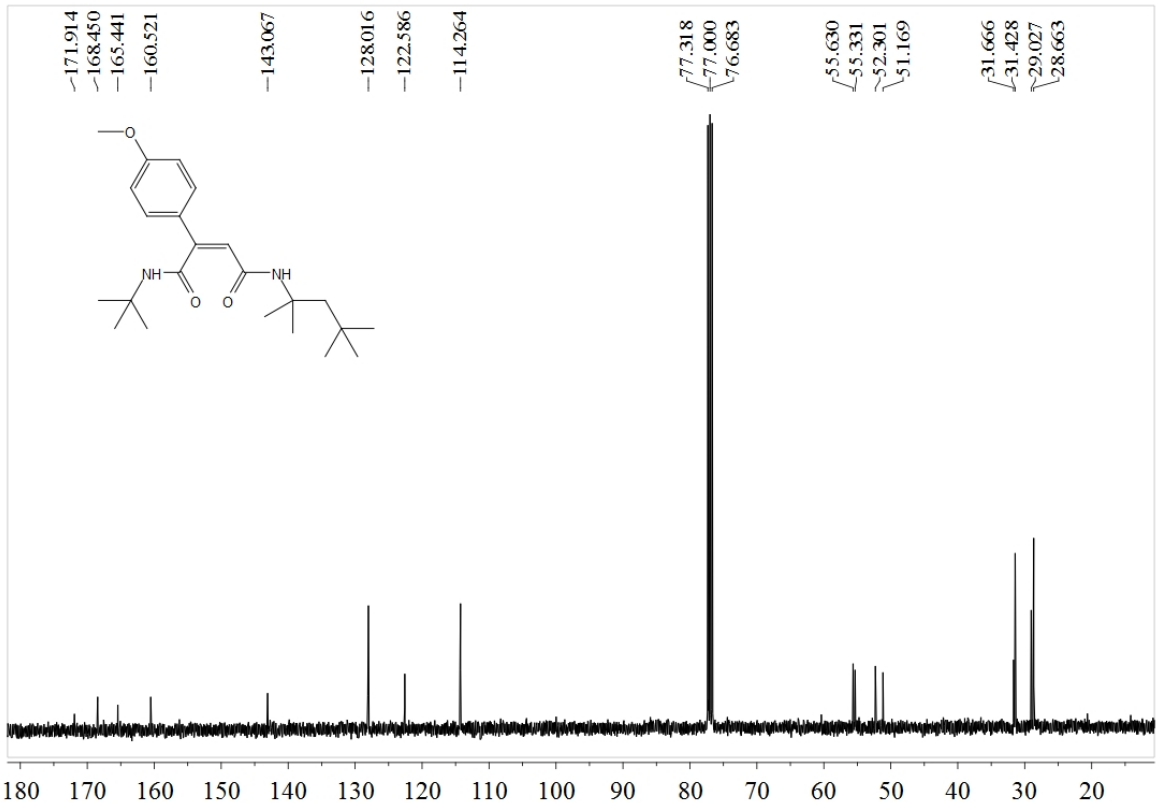

7.427
7.419
7.410
7.404
7.329
7.321
7.313
7.240
6.966
6.194
5.957
3.280
3.263
3.248
3.230
1.374
1.350
1.340
1.331
1.313
1.295
0.912
0.894
0.875
NH
O
O
NH
2.00
3.00
0.94
0.98
0.90
2.00
2.01
9.01
2.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5


168.123
165.763
144.481
135.375
129.362
128.821
126.500
122.760
77.318
77.000
76.682
52.302
39.495
31.353
28.457
20.104
13.679
NH
O
O
NH
170 150 130 110 90 80 70 60 50 40 30 20 10 0


7.487
7.465
7.218
6.931
6.908
6.196
3.815
3.259
3.242
3.227
3.210
2.061
2.056
2.050
2.045
1.373
1.336
0.931
0.913
0.894
O
NH
O
O
NH
1.99
0.98
3.01
1.00
3.00
2.01
2.08
9.00
2.12
3.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0


206.000
168.532
165.154
161.242
146.983
129.659
128.701
119.190
114.623
55.503
51.778
39.397
29.682
20.634
13.925
O
NH
O
O
NH
30.259
30.066
29.874
29.682
29.489
29.297
29.104
28.776
31.0
29.0
168.532
165.154
161.242
146.983
129.659
128.701
119.190
114.623
170
150
130
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10


7.369
7.347
7.240
6.871
6.849
6.831
6.055
5.820
3.794
1.786
1.423
1.398
0.986
O
NH
O
O
NH
2.00
2.01
0.86
0.99
0.95
3.00
2.00
6.00
9.00
9.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5


171.914
168.450
165.441
160.521
143.067
128.016
122.586
114.264
77.318
77.000
76.683
55.630
55.331
52.301
51.169
31.666
31.428
29.027
28.663
O
NH
O
O
NH
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20