Ultrasound accelerated synthesis of *O*-alkylated hydroximides under solvent- and metal-free conditions

Hongmei Jiang,[ab] Xiaoyue Tang,[c] Sihan Liu,[a] Lian Wang,[a] Haicheng Shen,[a] Jiankui Yang,[a] Huixian Wang,[a] and Qing-Wen Gui *[a]

[a]College of Science, Hunan Agricultural University, Changsha 410128, People's Republic of China

[b]State Key Laboratory of Chemo/Biosensing and Chemometrics, College of Chemical Engineering, Hunan University, Changsha 410082, P. R. China

[c]Department of Pharmaceutical Engineering, School of Chemistry and Chemical Engineering, South China University of Technology, Guangzhou 510640, People's Republic of China

E-mail: gqw1216@**hunau.edu.cn**, gqw1216@163.com

Table of Content:

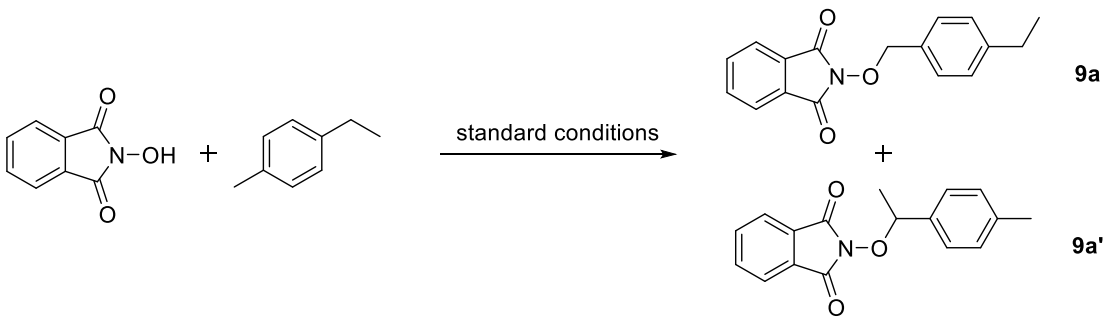
1. General Information

All reactions were carried out in anhydrous solvent and commercially available reagents were used as received unless otherwise stated. Analytical thin layer chromatography (TLC) was performed on precoated aluminium-backed silica gel 60 F_{254} plates (EMD Millipore, 200 μm thickness). TLC plates were visualized with ultraviolet light and treatment with $KMnO_4$ or vanillin stains followed by heating. Flash column chromatography was performed using Tsingtao silica gel (200-300). ^1H and ^{13}C NMR spectras were recorded on a Bruker Avance DRX-400 spectrometers; chemical shifts (δ) are given in ppm and calibrated using the signal of residual undeuterated solvent as internal reference ($CHCl_3$: δ_H =7.26 ppm and δ_C = 77.16 ppm). Data for ^1H NMR and ^{13}C NMR are reported as follows: chemical shift (δ, ppm), multiplicity, integration, and coupling constant (Hz).

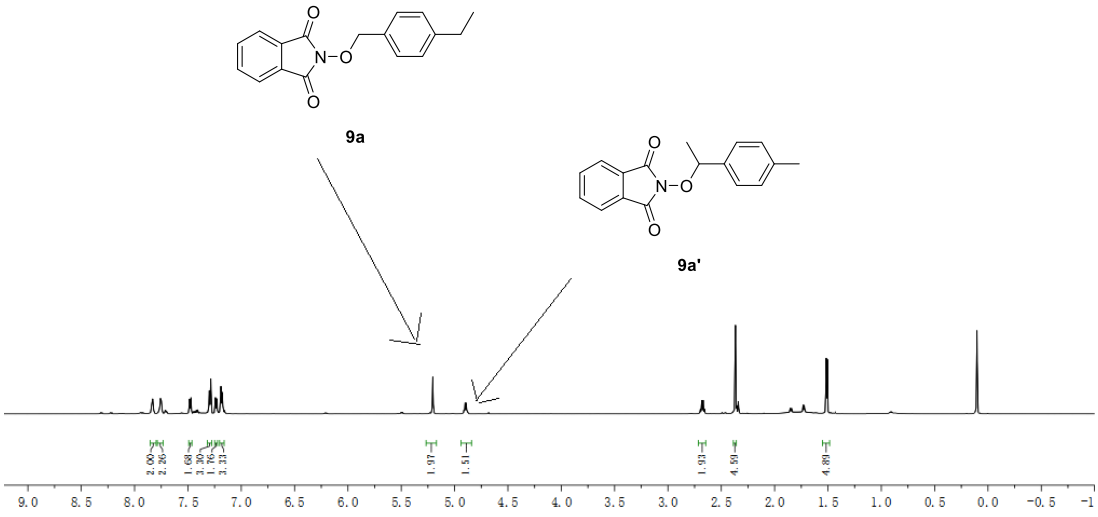
2. Experimental Section

General experimental procedures for 9a and 9a'

In a vial was placed NHPI (0.5 mmol), 4-ethyltoluene (2.5 mmol), TBAI (0.1 mmol) and TBHP (2.5 mmol), then the contents were reacted under ultrasound irradiation. Upon completion, the reaction mixture was quenched by addition of 10 mL of water. The aqueous layer was extracted three times with EtOAc (10 mL × 3), and the combine organic layers were dried over sodium sulfate, evaporated to dryness, and to afford the mixture of **9a** and **9a'**.

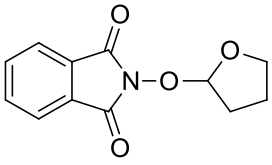


Scheme S1. The reaction of NHPI with 4-ethyltoluene



Scheme S2. The Crude 1H NMR of the mixture of **9a** and **9a'**

3. Characterization data of products 3a-h, 5a-t and 6

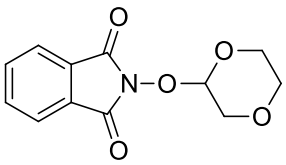


2-((tetrahydrofuran-2-yl)oxy)isoindoline-1,3-dione(3a)[1]

Color and State: White solid. m.p. 121-124 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.80 (m, 2H), 7.75-7.71 (m, 2H), 5.78 (d, *J* = 4.7 Hz, 1H), 4.37-4.32 (m, 1H), 4.03-3.98 (m, 1H), 2.33-2.19 (m, 2H), 2.15-2.06 (m, 1H), 1.99-1.90 (m, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 164.06, 134.43, 129.23, 123.56, 108.96, 69.28, 30.94, 22.68.

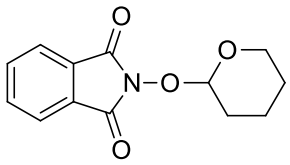


2-((1,4-dioxan-2-yl)oxy)isoindoline-1,3-dione(3b)[2]

Color and State: White solid. m.p. 185-187 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.81 (m, 2H), 7.76-7.73 (m, 2H), 5.22 (s, 1H), 4.83 (td, *J* = 12.0, 3.2 Hz, 1H), 4.14 (d, *J* = 12.0 Hz, 1H), 3.86 (d, *J* = 3.0 Hz, 1H), 3.81 (d, *J* = 2.4 Hz, 1H), 3.78 (d, *J* = 2.1 Hz, 1H), 3.53 (dd, *J* = 12.0, 2.4 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.67, 134.60, 129.08, 123.67, 99.62, 66.25, 66.05, 60.97.

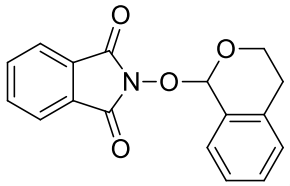


2-((tetrahydro-2H-pyran-2-yl)oxy)isoindoline-1,3-dione(3c)[2]

Color and State: White solid. m.p. 123-124 ºC

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58-7.82 (m, 2H), 7.77-7.73 (m, 2H), 5.43 (s, 1H), 4.53 (td, *J* = 11.4, 4.0 Hz, 1H), 3.68-3.65 (m, 1H), 2.12 (dd, *J* = 14.0, 1.5 Hz, 1H), 2.01-1.92 (m, 1H), 1.87 (dt, J = 13.3, 3.6 Hz, 1H), 1.71-1.69 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.89, 134.39, 129.38, 123.50, 103.21, 62.39, 27.86, 25.00, 17.76.

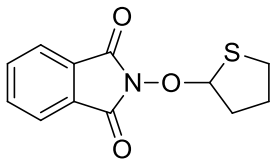


2-(isochroman-1-yloxy)isoindoline-1,3-dione(3d)[1]

Color and State: White solid. m.p. 131-133 ºC

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88-7.85 (m, 2H), 7.77-7.75 (m, 2H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.38-7.31 (m, 2H), 7.21 (d, *J* = 7.4 Hz, 1H), 6.20 (s, 1H), 4.70 (td, *J* = 12.0, 2.5 Hz, 1H), 4.05-4.01 (m, 1H), 3.15-3.06 (m, 1H), 2.73 (d, *J* = 16.0 Hz, 1H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 164.03, 134.99, 134.51, 129.70, 129.46, 129.32, 128.87, 128.59, 126.75, 123.63, 102.29, 59.73, 27.60.

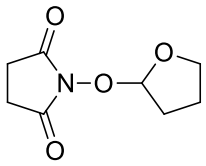


2-((tetrahydrothiophen-2-yl)oxy)isoindoline-1,3-dione(3e)[2]

Color and State: White solid. m.p. 119-121 ºC

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.78 (m, 2H), 7.75-7.71 (m, 2H), 6.09 (d, *J* = 4.0 Hz, 1H), 3.14-3.12 (m, 1H), 2.89-2.82 (m, 1H), 2.57 (dd, *J* = 13.8, 5.3 Hz, 1H), 2.41-2.29 (m, 1H), 2.25-2.17 (m, 1H), 2.02-1.92 (m, 1H).

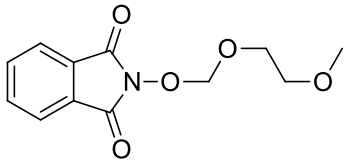
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.02, 134.51, 128.96, 123.60, 97.41, 37.17, 32.61, 27.23.



1-((tetrahydrofuran-2-yl)oxy)pyrrolidine-2,5-dione(3f)

Color and State: White solid. m.p. 133-135 ºC

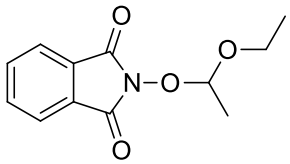
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.72 (d, *J* = 4.8 Hz, 1H), 4.25-4.20 (m, 1H), 3.97-3.92 (m, 1H), 2.70 (s, 4H), 2.24-2.14 (m, 2H), 2.08-2.01 (m, 1H), 1.93-1.84 (m, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 171.76, 107.84, 69.34, 30.94, 25.65, 22.60.
HRMS (ESI) calcd for $C_8H_{12}NO_4$ $[M+H]^+$: 186.0761, found 186.0770.



2-((2-methoxyethoxy)methoxy)isoindoline-1,3-dione(3g)[2]

Color and State: White solid. m.p. 80-82 ºC

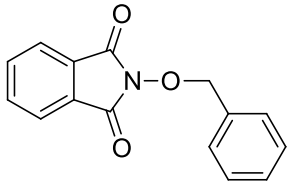
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.81 (m, 2H), 7.76-7.73 (m, 2H), 5.20 (s, 2H), 4.12 (t, *J* = 4.6 Hz, 2H), 3.62-3.58 (m, 2H), 3.38 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.76, 134.59, 129.14, 123.64, 100.19, 71.62, 69.16, 59.13.



2-(1-ethoxyethoxy)isoindoline-1,3-dione(3h)[2]

Color and State: White oil.

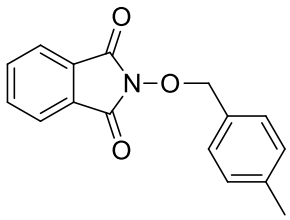
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.83 (m, 2H), 7.76-7.73 (m, 2H), 5.32-5.28 (m, 1H), 4.21-4.13 (m, 1H), 3.86-3.78 (m, 1H), 1.52 (d, *J* = 5.4 Hz, 3H), 1.23 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.54, 134.56, 129.30, 123.61, 106.57, 63.71, 19.05, 15.12.



2-(benzyloxy)isoindoline-1,3-dione(5a)[1]

Color and State: White solid. m.p. 144-146 ºC

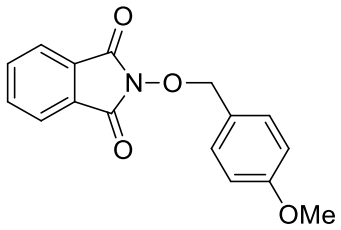
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.78 (m, 2H), 7.75-7.70 (m, 2H), 7.56-7.53 (m, 2H), 7.39-7.36 (m, 3H), 5.21 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.63, 134.57, 133.79, 130.03, 129.49, 128.97, 128.68, 123.63, 80.00.



2-((4-methylbenzyl)oxy)isoindoline-1,3-dione(5b)[1]

Color and State: White solid. m.p. 133-136 ºC

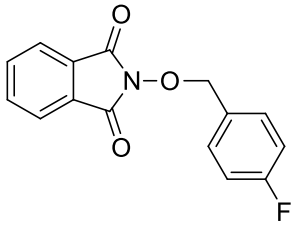
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.79 (m, 2H), 7.75-7.72 (m, 2H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 5.17 (s, 2H), 2.35 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.68, 139.44, 134.52, 130.82, 130.11, 129.38, 129.03, 123.61, 79.86, 21.48.



2-((4-methoxybenzyl)oxy)isoindoline-1,3-dione(5c)[4]

Color and State: White solid. m.p. 134-137 ºC

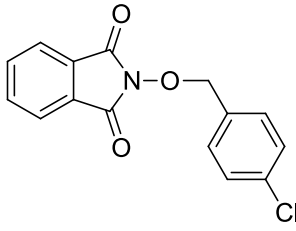
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.75 (m, 2H), 7.71-7.68 (m, 2H), 7.42 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 5.12 (s, 2H), 3.77 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.61, 160.47, 134.45, 131.70, 128.91, 125.88, 123.49, 113.95, 79.53, 55.29.



2-((4-fluorobenzyl)oxy)isoindoline-1,3-dione(5d)[4]

Color and State: White solid. m.p. 154-156 ºC

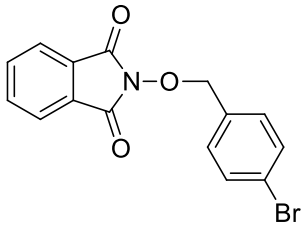
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.80 (m, 2H), 7.76-7.73 (m, 2H), 7.54-7.50 (m, 2H), 7.06 (t, *J* = 8.0 Hz, 2H), 5.17 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.33 (d, *J* = 195.0 Hz), 163.63, 134.65, 132.08 (d, *J* = 6.0 Hz), 129.78 (d, *J* = 2.0 Hz), 128.95, 123.69, 115.80 (d, *J* = 15.0 Hz), 79.15.



2-((4-chlorobenzyl)oxy)isoindolin-1,3-dione(5e)[4]

Color and State: White solid. m.p. 132-135 ºC

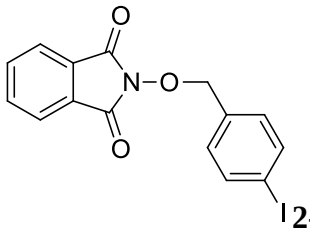
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.80 (m, 2H), 7.76-7.73 (m, 2H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 5.18 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.42, 135.32, 134.50, 132.19, 131.13, 128.77, 124.06, 123.54, 78.90.



2-((4-bromobenzyl)oxy)isoindoline-1,3-dione(5f)[4]

Color and State: White solid. m.p. 134-136 °C

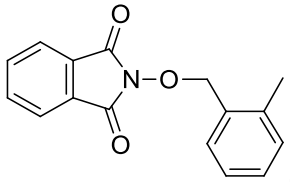
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.77 (m, 2H), 7.75-7.71 (m, 2H), 7.49 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 8.2 Hz, 2H), 5.15 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.51, 134.61, 132.06, 131.86, 131.48, 128.95, 123.69, 123.65, 79.07.



2-((4-iodobenzyl)oxy)isoindoline-1,3-dione(5g)

Color and State: White soild. m.p. 133-135 °C

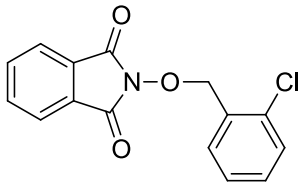
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.76 (m, 2H), 7.73-7.70 (m, 3H), 7.68 (s, 1H), 7.23 (d, *J* = 4.0 Hz, 2H), 5.12 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.59, 137.88, 134.68, 133.49, 131.65, 128.94, 123.73, 95.65, 79.22.
HRMS (ESI) calcd for $C_{15}H_{11}NO_3$ $[M+H]^+$: 379.9778, found 379.9782.



2-((2-methylbenzyl)oxy)isoindoline-1,3-dione(5h)[3]

Color and State: White solid. m.p. 129-131°C

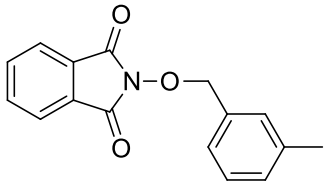
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74-7.70 (m, 2H), 7.76-7.63 (m, 2H), 7.28 (t, *J* = 7.0 Hz, 1H), 7.22-7.15 (m, 2H), 7.07 (t, *J* = 7.0 Hz, 1H), 5.16 (s, 2H), 2.52 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.62, 139.23, 134.51, 131.79, 131.42, 130.65, 129.86, 129.00, 125.96, 123.55, 78.16, 19.13.



2-((2-chlorobenzyl)oxy)isoindoline-1,3-dione(5i)[3]

Color and State: White solid. m.p. 141-143 °C

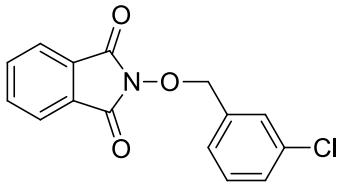
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.80 (m, 2H), 7.75-7.73 (m, 2H), 7.63 (dd, *J* = 7.0, 2.0 Hz, 1H), 7.39 (dd, *J* = 7.2, 1.8 Hz, 1H), 7.32-7.28 (m, 2H), 5.37 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.48, 134.98, 134.60, 132.60, 131.91, 130.70, 129.73, 128.97, 127.13, 123.67, 76.56.



2-((3-methylbenzyl)oxy)isoindoline-1,3-dione(5j)[3]

Color and State: White solid. m.p. 130-133 °C

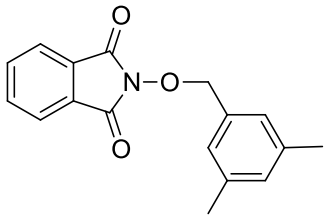
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76-7.72 (m, 2H), 7.68-7.64 (m, 2H), 7.29 (s, 1H), 7.25 (d, J = 8.0 Hz, 1H), 7.19 (t, J = 8.0 Hz, 1H), 7.11 (d, *J* = 8.0 Hz, 1H), 5.10 (s, 2H), 2.29 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.62, 138.40, 134.54, 133.67, 130.70, 130.22, 129.02, 128.56, 127.03, 123.61, 80.11, 21.42.



2-((3-chlorobenzyl)oxy)isoindoline-1,3-dione(5k)

Color and State: White solid. m.p. 104-106 °C

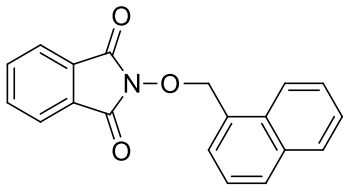
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.81 (m, 2H), 7.77-7.76 (m, 2H), 7.55 (s, 1H), 7.45 (d, *J* = 7.2 Hz, 1H), 7.37-7.32 (m, 2H), 5.18 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.56, 135.80, 134.70, 134.55, 130.01, 129.89, 129.61, 124.54, 120.12, 108.84, 82.75.
HRMS (ESI) calcd for $C_{15}H_{11}ClNO_3$ $[M+H]^+$: 288.0422, found 288.0428.



2-((3,5-dimethylbenzyl)oxy)isoindoline-1,3-dione(5l)[5]

Color and State: White solid. m.p. 132-135 °C

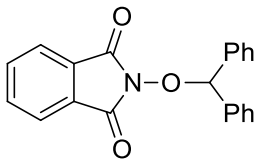
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.80 (m, 2H), 7.54-7.52 (m, 2H), 7.15 (s, 2H), 7.00 (s, 1H), 5.13 (s, 2H), 2.32 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.58, 138.23, 134.48, 133.58, 131.04, 129.09, 127.71, 123.55, 80.18, 21.24.
HRMS (ESI) calcd for $C_{17}H_{16}NO_3$ $[M+H]^+$: 282.1125, found 282.1129.



2-(naphthalen-1-ylmethoxy)isoindoline-1,3-dione(5m)[1]

Color and State: White solid. m.p. 127-130 °C

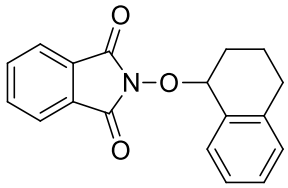
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.64 (d, *J* = 8.4 Hz, 1H), 7.90 (t, *J* = 8.0 Hz, 2H), 7.84-7.81 (m, 2H), 7.76-7.72 (m, 2H), 7.68 (t, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 7.0 Hz, 1H), 7.55 (t, *J* = 7.5 Hz , 1H), 7.44 (t, *J* = 8.0 Hz, 1H), 5.65 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.67, 134.60, 133.82, 132.61, 130.69, 129.72, 129.57, 128.59, 127.12, 126.31, 125.16, 124.62, 123.65, 78.25.



2-(benzhydryloxy)isoindoline-1,3-dione(5n)[1]

Color and State: White solid. m.p. 165-167 °C

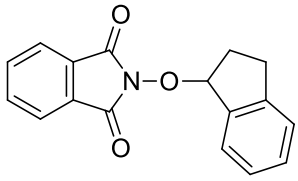
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64-7.61 (m, 2H), 7.59-7.55 (m, 2H), 7.46 (d, *J* = 7.4 Hz, 4H), 7.28-7.24 (m, 6H), 6.44(s, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.78, 137.91, 134.41, 128.91, 128.82, 128.45, 128.41, 123.47, 89.72.



2-((1,2,3,4-tetrahydronaphthalen-1-yl)oxy)isoindoline-1,3-dione(5o)[1]

Color and State: White solid. m.p. 103-107 °C

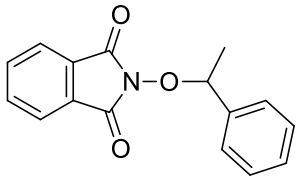
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.78 (m, 2H), 7.73-7.69 (m, 2H), 7.66 (d, *J* = 7.6 Hz, 1H), 7.23 (d, *J* = 7.2 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 7.13 (d, *J* = 7.2 Hz, 1H), 5.28 (t, *J* = 4.0 Hz, 1H), 2.91 (dt, *J* = 16.6, 4.5 Hz, 1H), 2.76-2.67 (m, 1H), 2.36-2.27 (m, 2H), 1.86-1.77 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.43, 139.03, 134.52, 131.93, 131.56, 129.19, 129.04, 123.56, 83.29, 29.12, 27.49, 17.84.



2-((2,3-dihydro-1H-inden-1-yl)oxy)isoindoline-1,3-dione(5p)[1]

Color and State: White solid. m.p. 96-98 °C

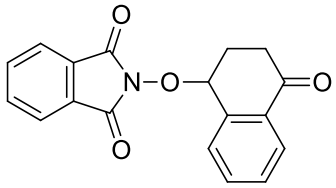
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.80 (m, 2H), 7.76-7.72 (m, 2H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 4.0 Hz, 2H), 7.22-7.18 (m, 1H), 5.79 (d, *J* = 6.2 Hz, 1H), 3.40-3.32 (m, 1H), 2.94-2.87 (m, 1H), 2.58-2.53 (m, 1H), 2.39-2.30 (m, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 164.21, 146.07, 138.92, 134.52, 130.06, 129.03, 126.62, 126.41, 125.17, 123.58, 91.82, 31.69, 30.39.



2-(1-phenylethoxy)isoindolie-1,3-dione(5q)[1]

Color and State: White solid. m.p. 93-95 °C

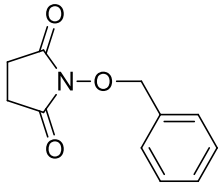
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76-7.74 (m, 2H), 7.70-7.67 (m, 2H), 7.51 (dd, *J* = 8.0, 2.0 Hz, 2H), 7.36-7.30 (m, 3H), 5.52-5.47 (m, 1H), 1.72 (d, *J* = 6.6 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 163.97, 139.10, 134.44, 129.13, 128.94, 128.50, 127.77, 123.52, 85.28, 20.59.



2-((4-oxo-1,2,3,4-tetrahydronaphthalen-1-yl)oxy)isoindoline-1,3-dione(5r)

Color and State: White solid. m.p. 126-128 °C

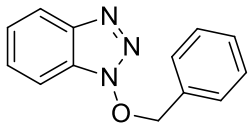
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.0 Hz, 1H), 7.74-7.72 (m, 2H), 7.67-7.64 (m, 2H), 7.43 (td, *J* = 7.6, 1.4 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 7.8 Hz, 1H), 4.88-4.86 (m, 1H), 3.27-3.22 (m, 1H), 2.95-2.90 (m, 1H), 2.53-2.42 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 192.35, 163.36, 143.33, 134.58, 134.18, 131.32, 128.88, 128.73, 128.00, 127.11, 123.68, 84.47, 28.32, 25.86.
HRMS (ESI) calcd for $C_{18}H_{14}NO_4$ $[M+H]^+$: 308.0917, found 308.0925.



1-(benzyloxy)pyrrolidine-2,5-dione(5s)[4]

Color and State: White solid. m.p. 160-163 °C

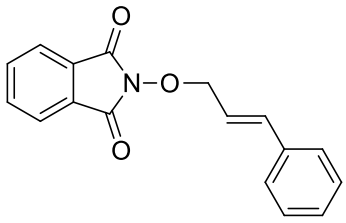
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.47 (m, 2H), 7.38-7.36 (m, 3H), 5.11 (s, 2H), 2.63 (s, 4H).
^{13}C NMR (101 MHz, $CDCl_3$) δ171.14, 133.53, 130.00, 129.52, 128.66, 78.73, 25.55.



1-(benzyloxy)-1H-benzo[d][1,2,3]triazole(5t)[6]

Color and State: White solid. m.p. 180-183 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.0 Hz, 1H), 7.37-7.28 (m, 7H), 7.17 (d, *J* = 8.0 Hz, 1H), 5.52 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 143.40, 133.22, 130.12, 129.91, 128.95, 128.08, 127.89, 124.54, 120.12, 108.84, 82.75.



2-(cinnamyloxy)isoindoline-1,3-dione(6)[1]

Color and State: White solid. m.p. 118-120^{o}C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74-7.71(m, 2H), 7.64-7.61 (m, 2H), 7.29 (d, *J* = 7.8 Hz, 2H), 7.22 (t, *J* = 7.6 Hz, 2H), 7.17 (d, *J* = 7.0 Hz, 1H), 6.58 (d, *J* = 16.0 Hz, 1H), 6.41-6.36 (m, 1H), 4.78 (d, *J* = 7.0 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.93, 137.60, 135.88, 134.55, 128.91, 128.72, 128.54, 127.01, 123.63, 122.11, 78.74.

4. Reference

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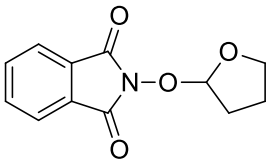
5. H and C NMR spectra of products



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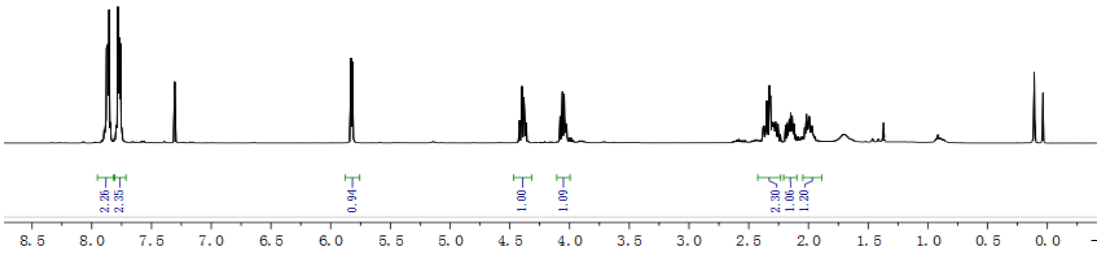

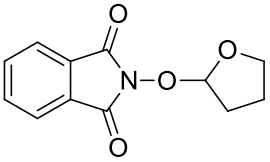

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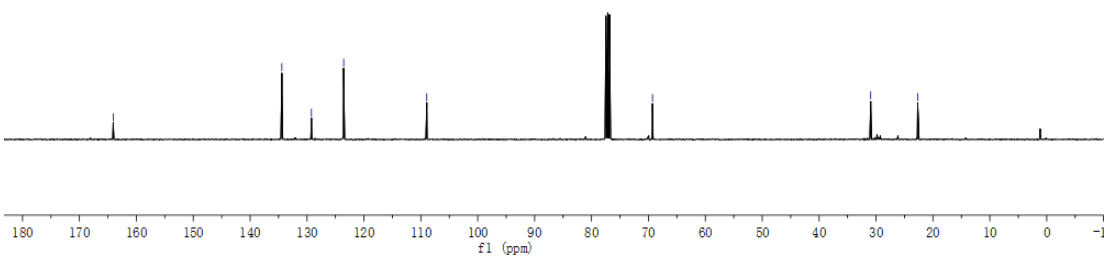

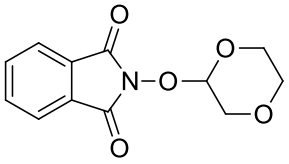

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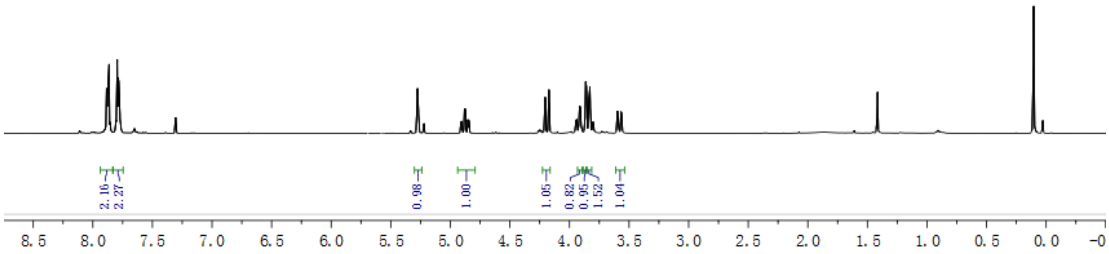
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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0.0


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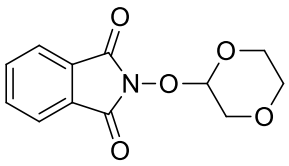
129.08
123.67
99.62
66.25
66.05
60.97



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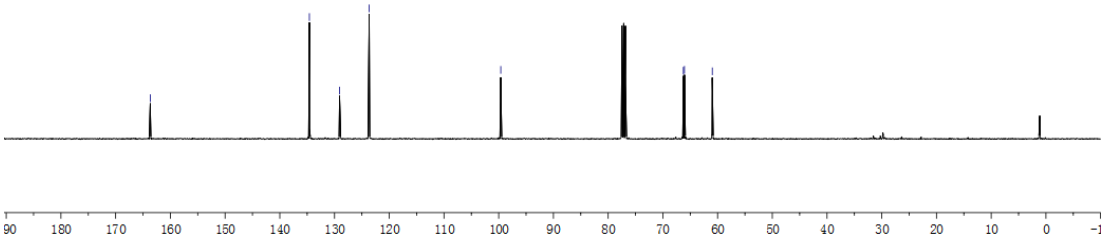
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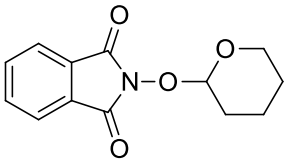
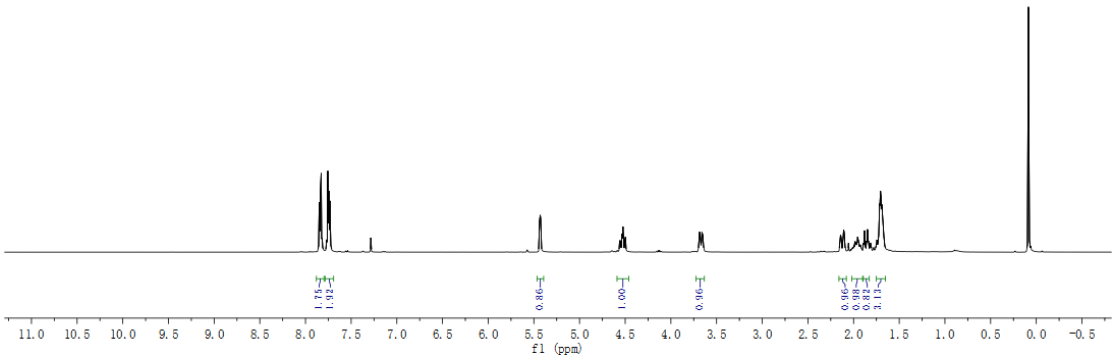
10
0



3c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1.00
0.96
0.96
0.83
3.13
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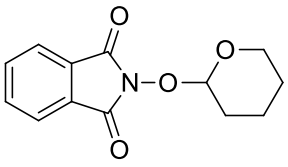
25.00
17.76



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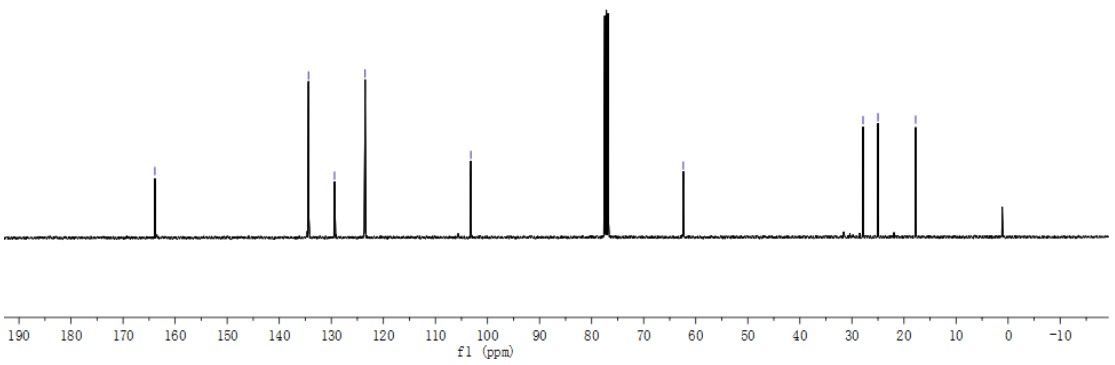
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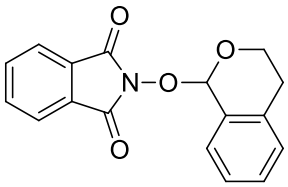
f1 (ppm)



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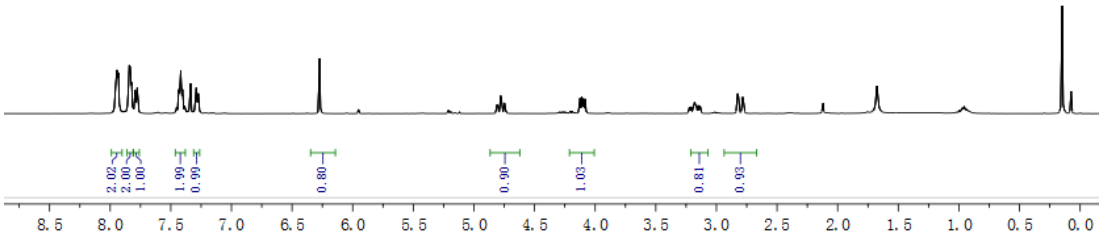
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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


164.03
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126.75
123.63
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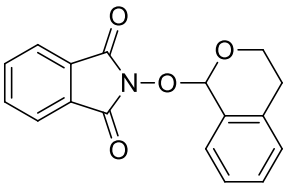
27.60



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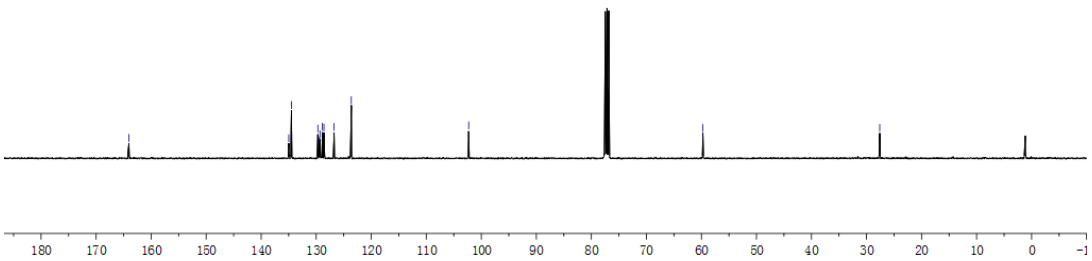
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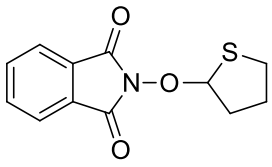
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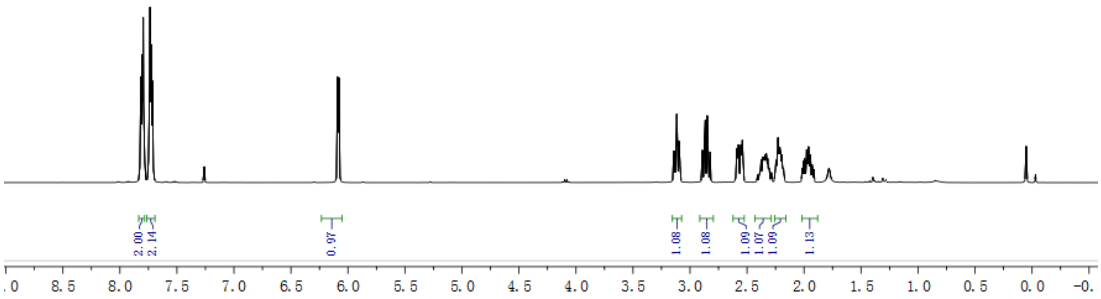
O
S
O

3e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6.5
6.0
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4.0
3.5
3.0
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2.0
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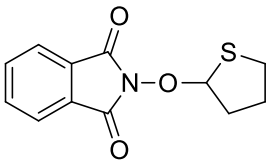
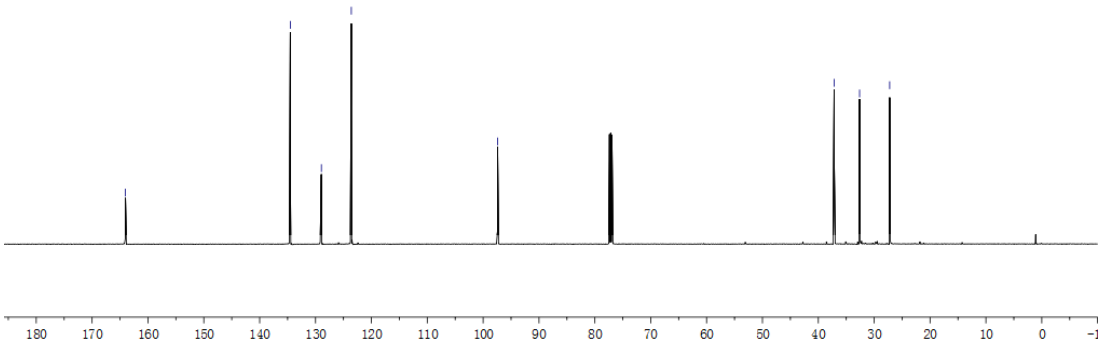

O
N
O
S
O

3e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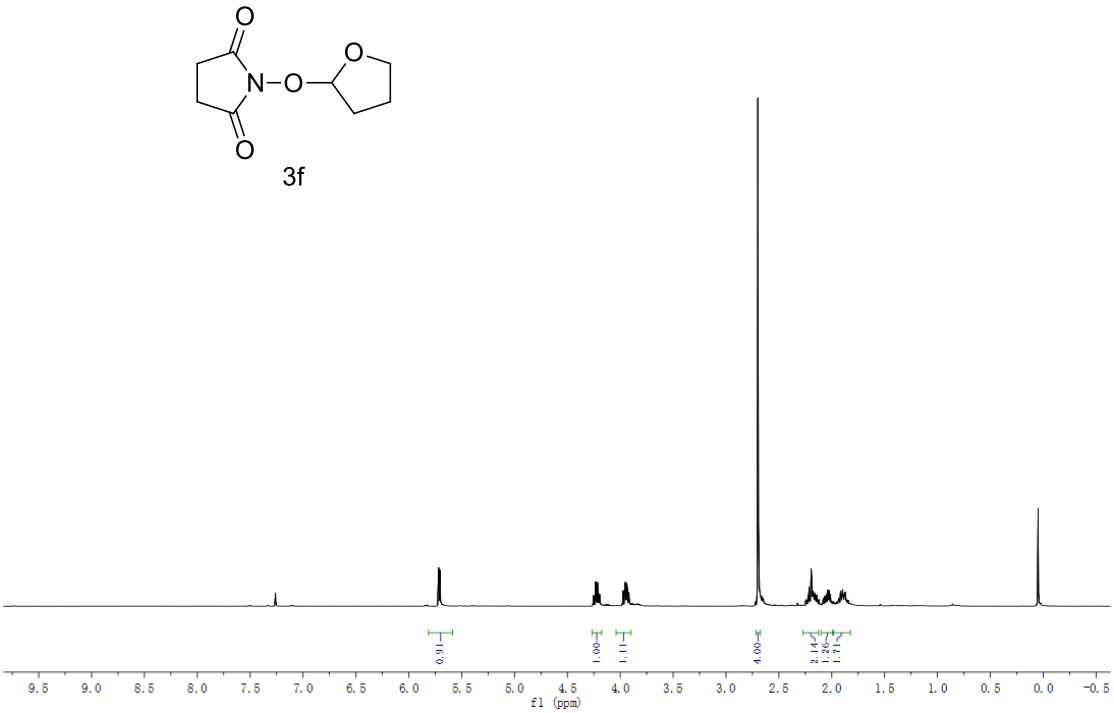
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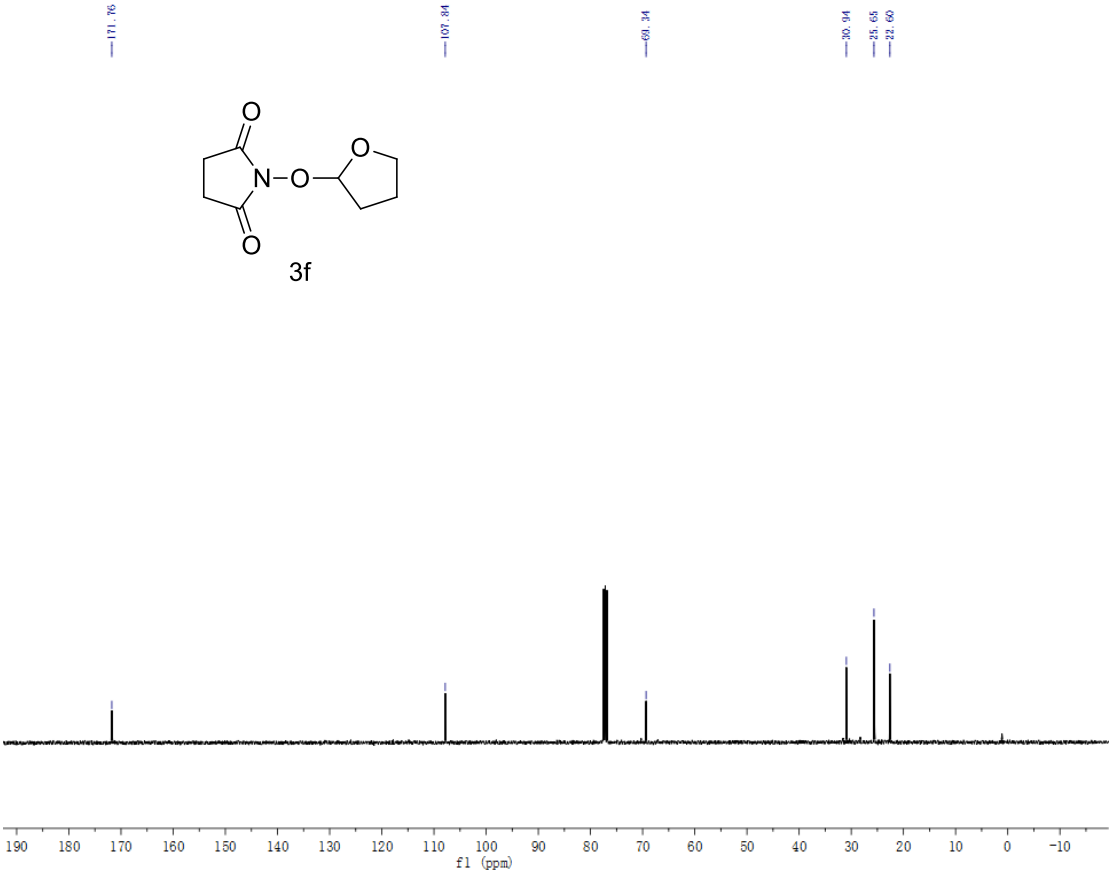
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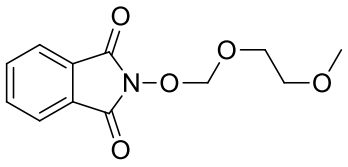
f1 (ppm)



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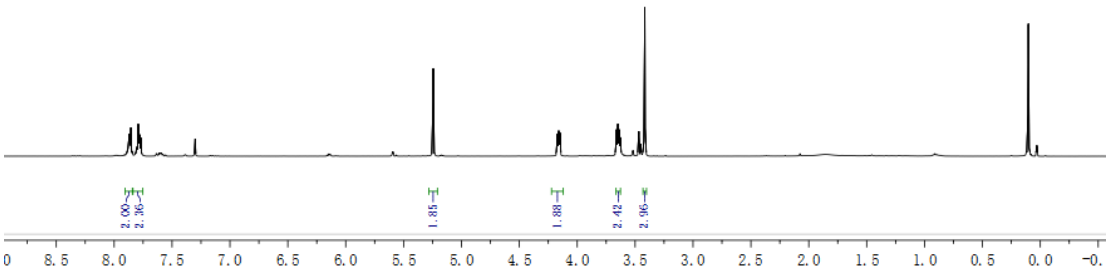
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4.0
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2.0
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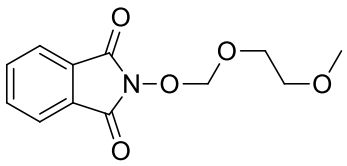
69.16
59.13



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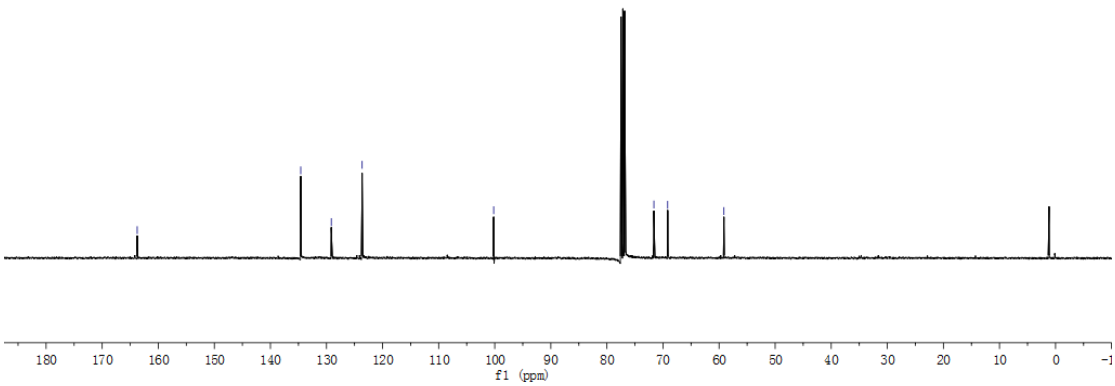
180
170
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110
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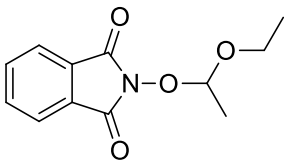
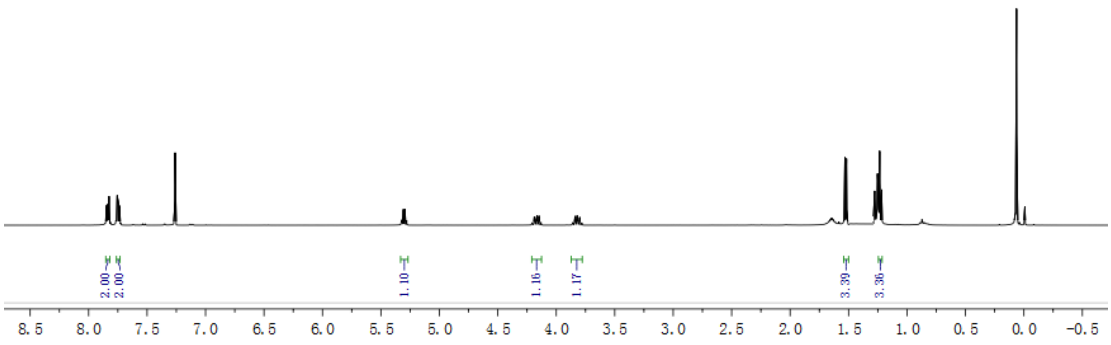
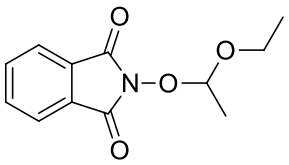
70
60
50
40
30
20
10
0
f1 (ppm)



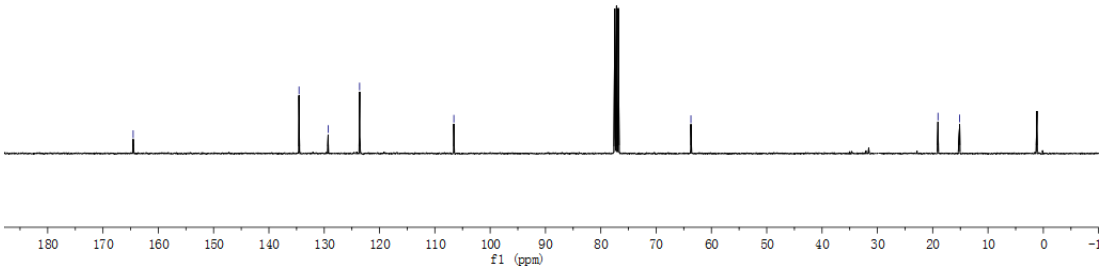
3h

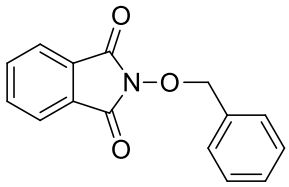

2.00
2.00
1.10
1.16
1.17
3.39
3.36
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5


164.54
134.56
129.39
123.61
106.57
63.71
19.05
15.12

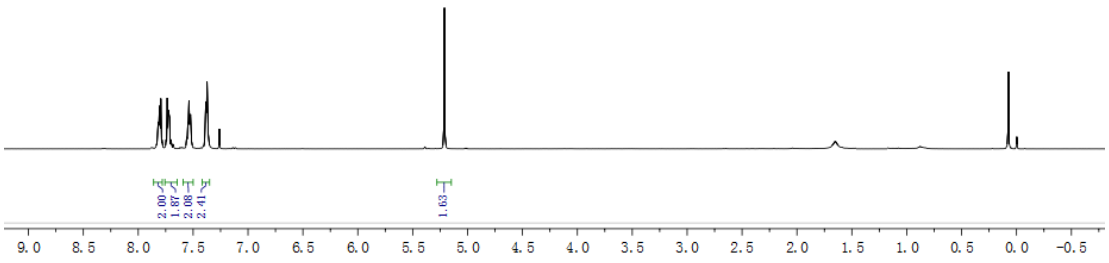


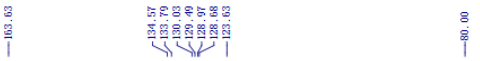
3h

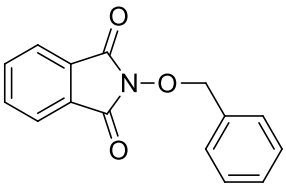

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



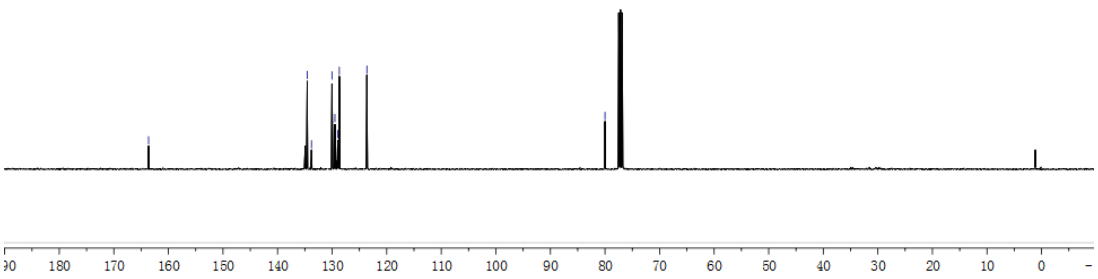
5a


2.00
1.87
2.08
2.41
1.63
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 -0.5


163.63
134.57
133.79
130.03
129.49
128.97
128.68
123.63
80.00



5a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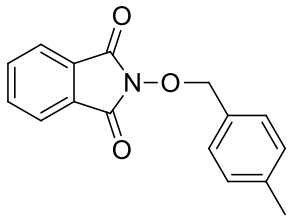
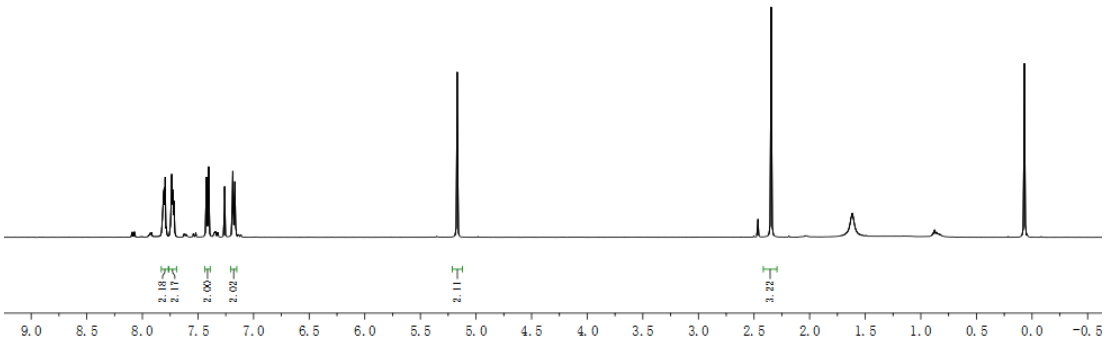
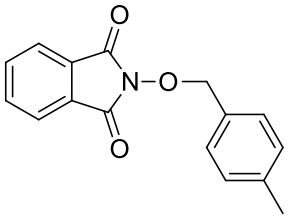


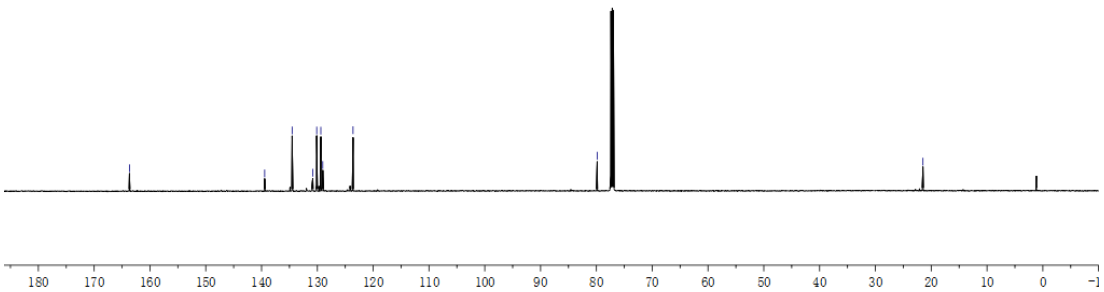
5b

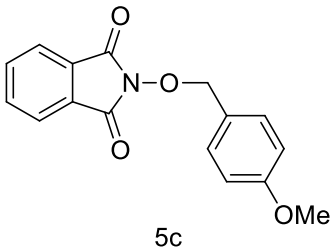

2.18
2.17
2.00
2.02
2.11
3.22
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5


163.68
139.44
134.52
130.32
130.11
129.38
129.03
123.61
79.86
21.48

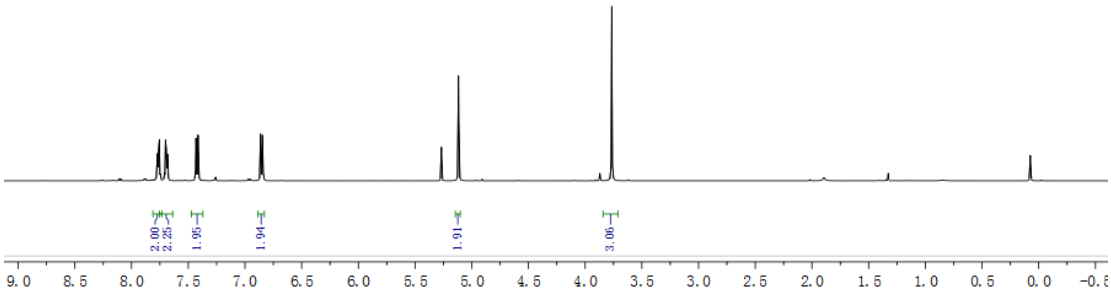


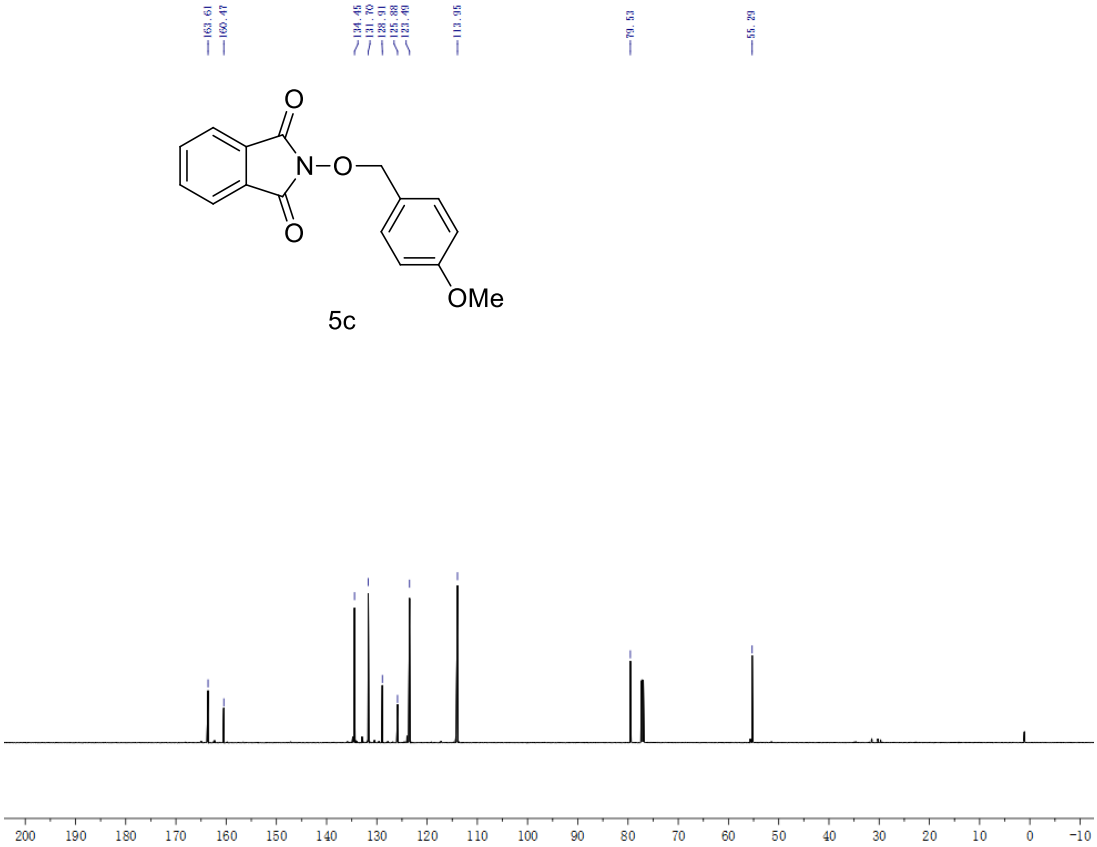
5b


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1

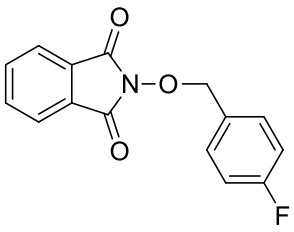

O
N–O
O
OMe

5c

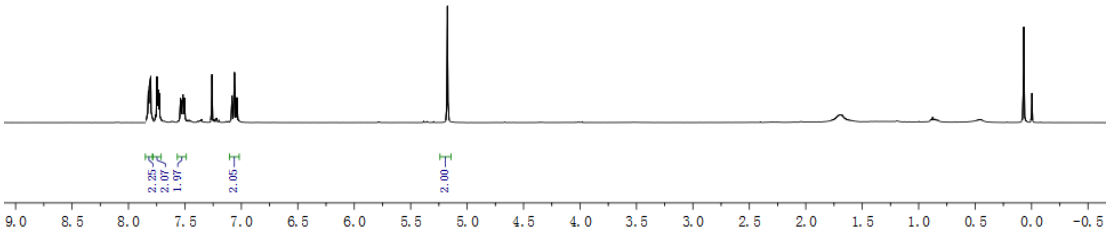

2.00
2.25
1.95
1.94
1.91
3.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

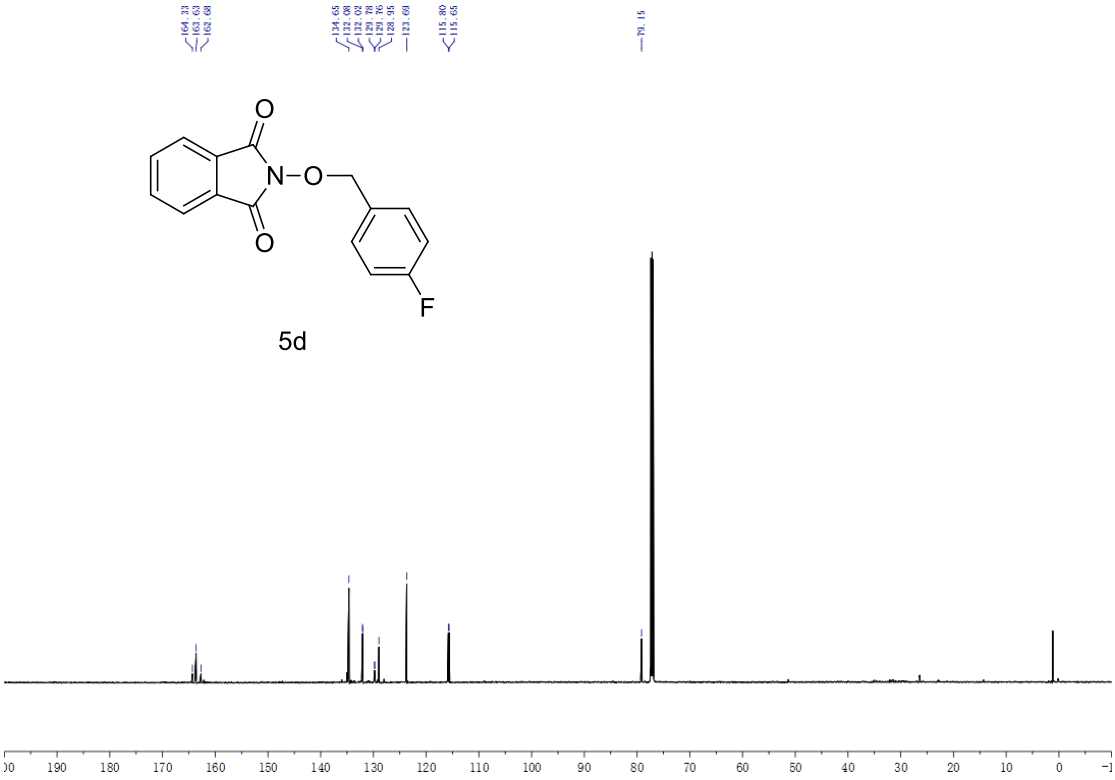

163.61
160.47
134.45
131.70
128.91
123.88
121.40
113.95
79.53
55.29
O
N–O
O
OMe
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

5c


O
N
O
O
F

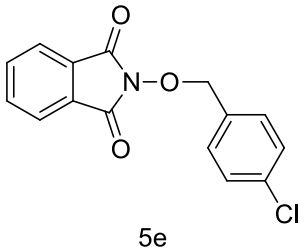
5d


2.25
2.07
1.97
2.05
2.00
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
0.0
-0.5

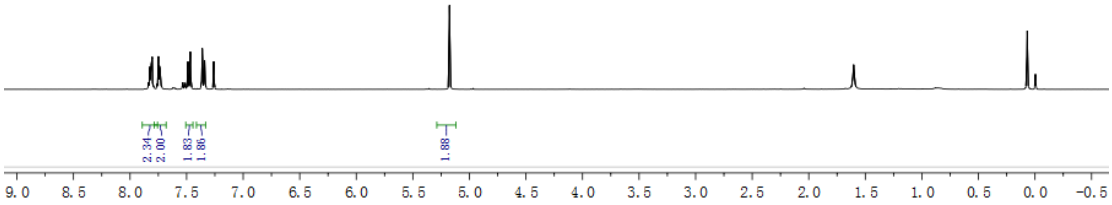

164.33
163.63
162.68
134.65
132.08
132.02
129.78
129.76
128.95
123.69
115.80
115.65
79.15
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

O
N
O
O
F

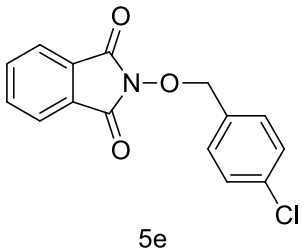
5d


O
N
O
O
Cl

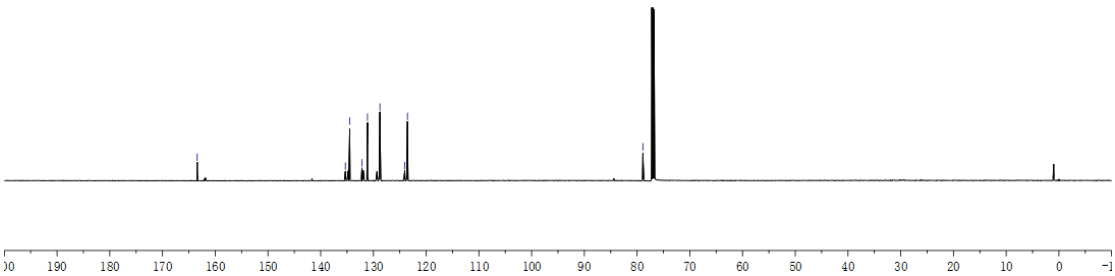
5e

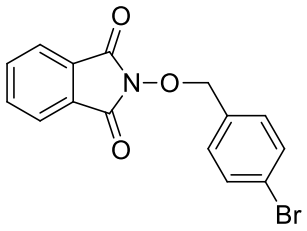

2.34
2.00
1.83
1.86
1.88
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


163.42
135.32
134.50
132.19
131.13
128.77
124.06
123.54
78.90

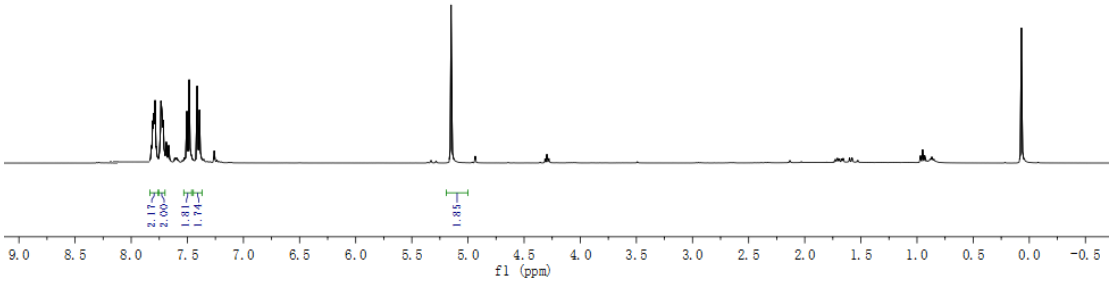

O
N
O
O
Cl

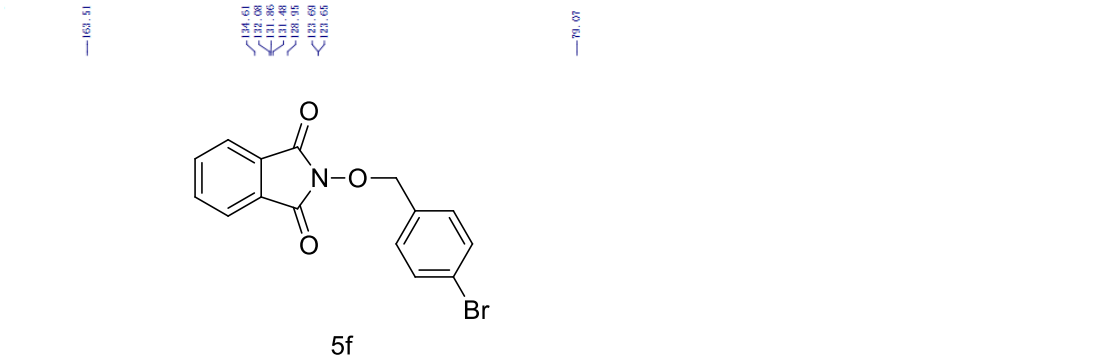
5e


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


O
N
O
O
Br

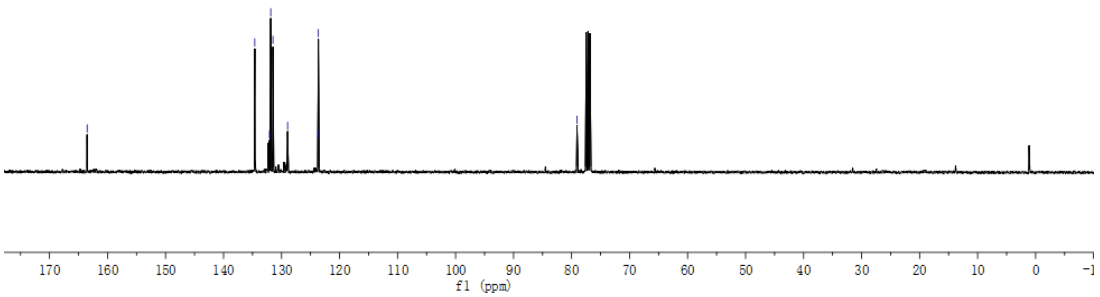
5f

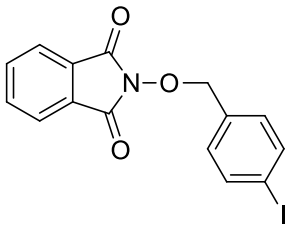

2.17
2.00
1.81
1.74
1.85
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


163.51
134.61
132.08
131.86
131.48
128.95
123.69
123.65
79.07

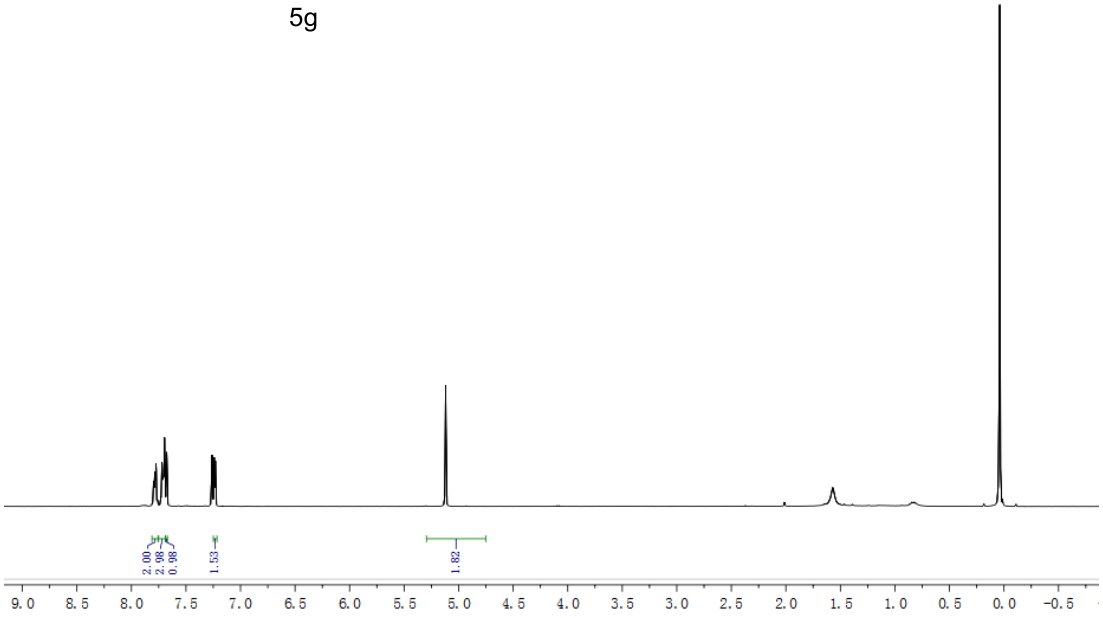
O
N
O
O
Br

5f

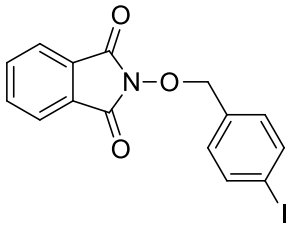

170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



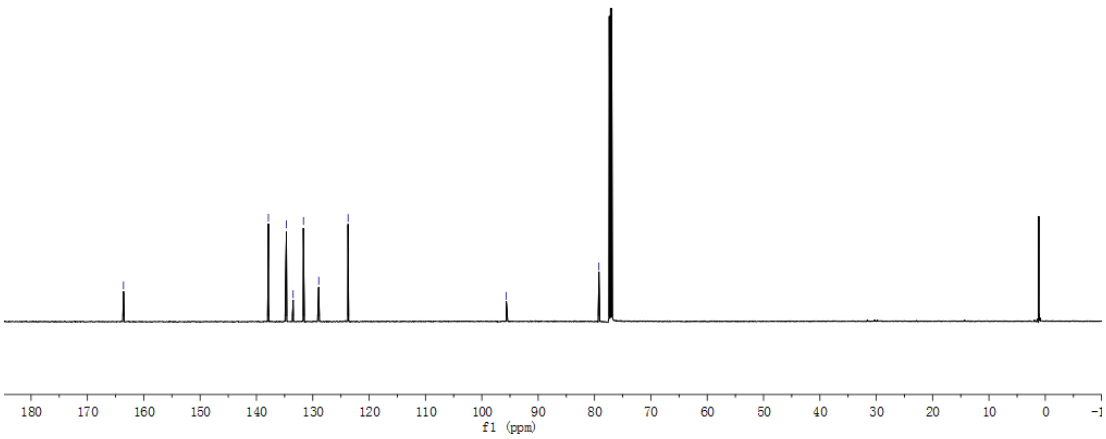
5g

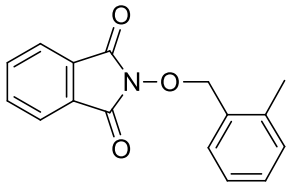

2.00
2.98
0.98
1.53
1.82
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5


163.59
137.88
134.68
133.49
131.65
128.94
123.73
95.65
79.22

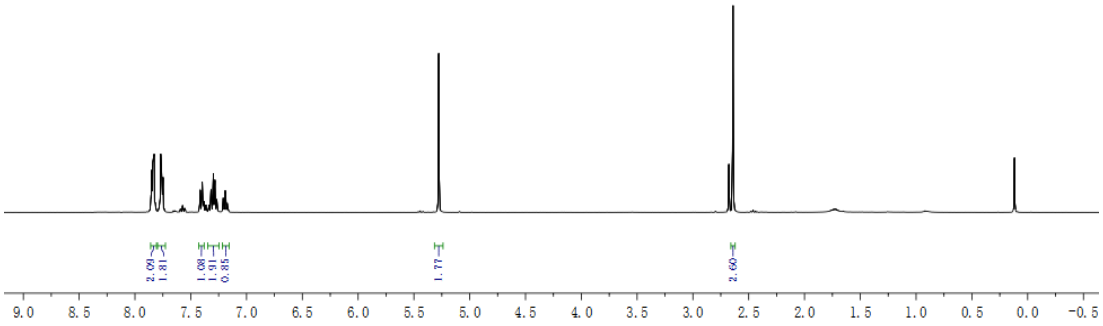


5g

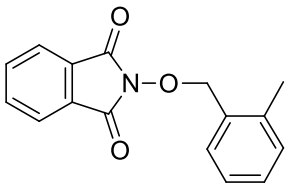

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)



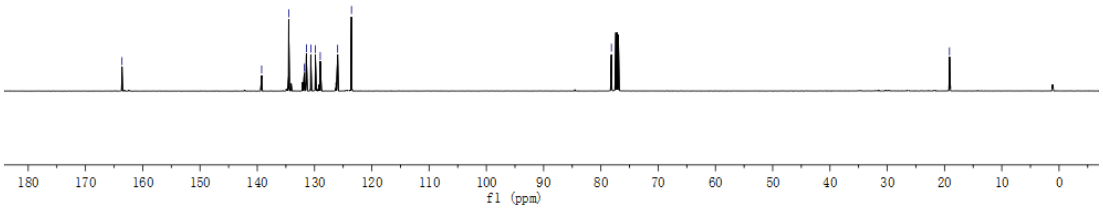
5h

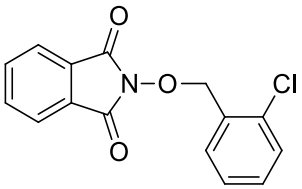

2.09
1.81
1.08
1.01
0.85
1.77
2.60
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5


163.62
139.23
134.51
131.79
131.42
130.65
129.86
129.05
125.96
123.55
78.16
19.13

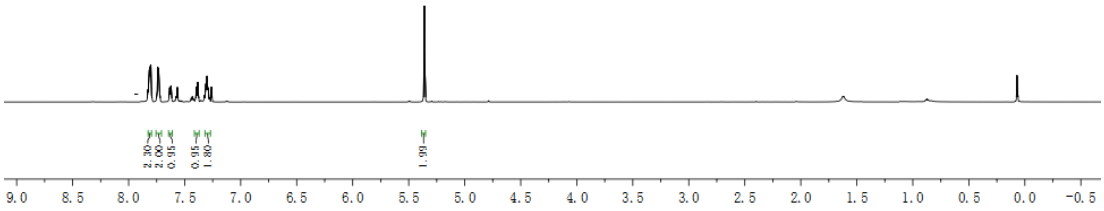


5h

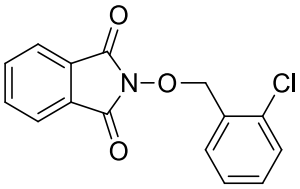

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
N–O
Cl
O

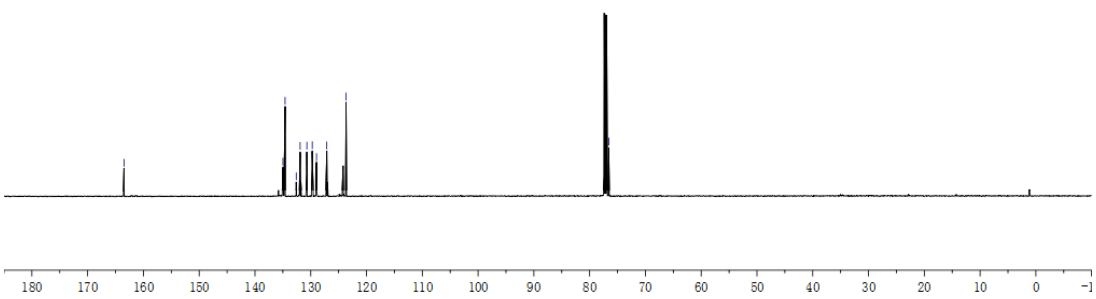
5i

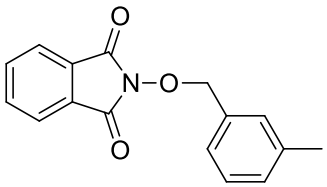

2.30
2.00
0.95
0.95
1.80
1.99
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


163.48
134.98
134.60
132.60
131.91
130.70
129.73
129.37
128.05
127.13
123.67
76.56

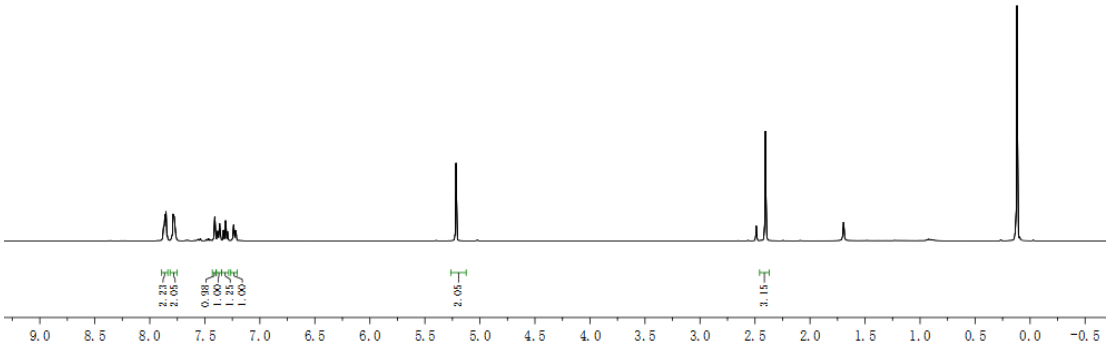

O
N–O
Cl
O

5i

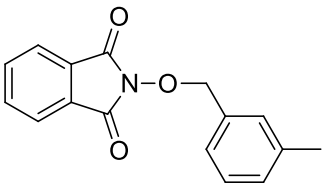

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1



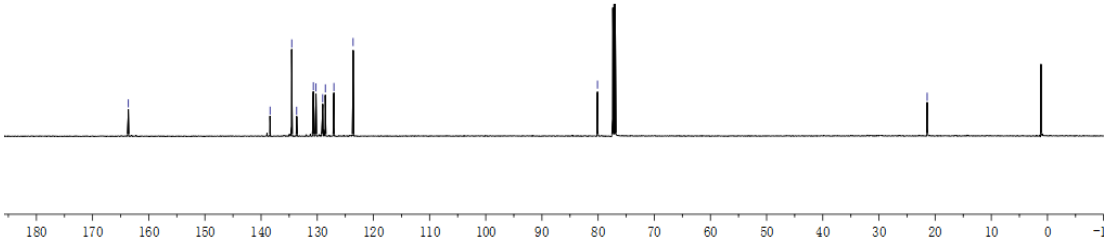
5j

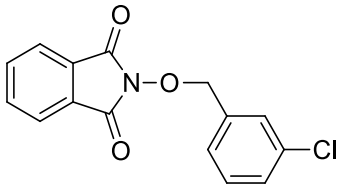

2.23
2.05
0.98
1.00
1.25
1.00
2.05
3.15
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5


163.62
138.40
134.54
133.67
130.70
130.22
129.02
128.56
127.03
123.61
80.11
21.42

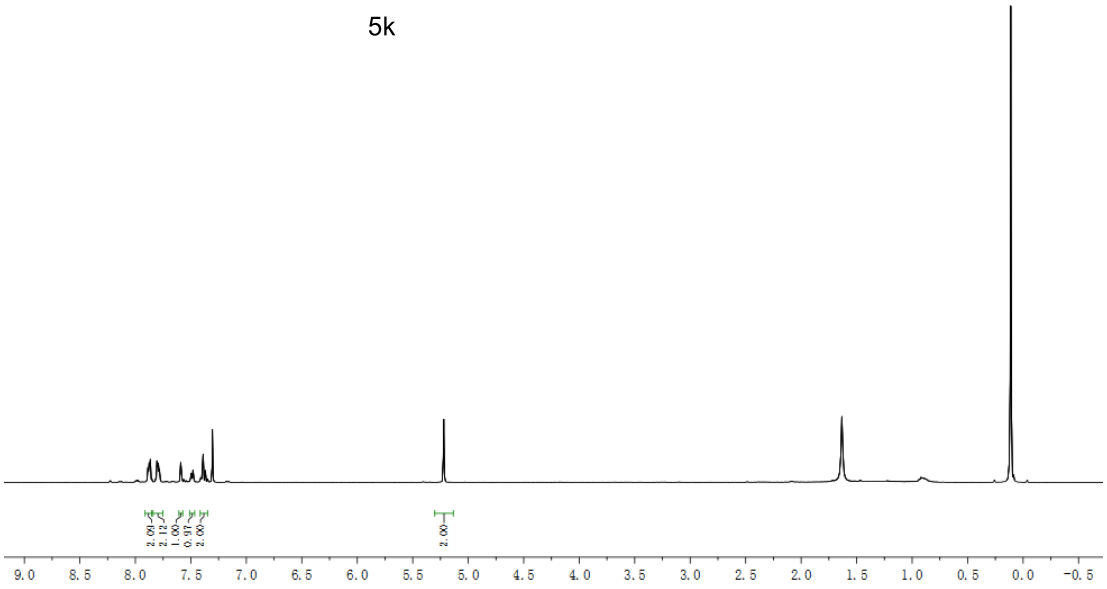


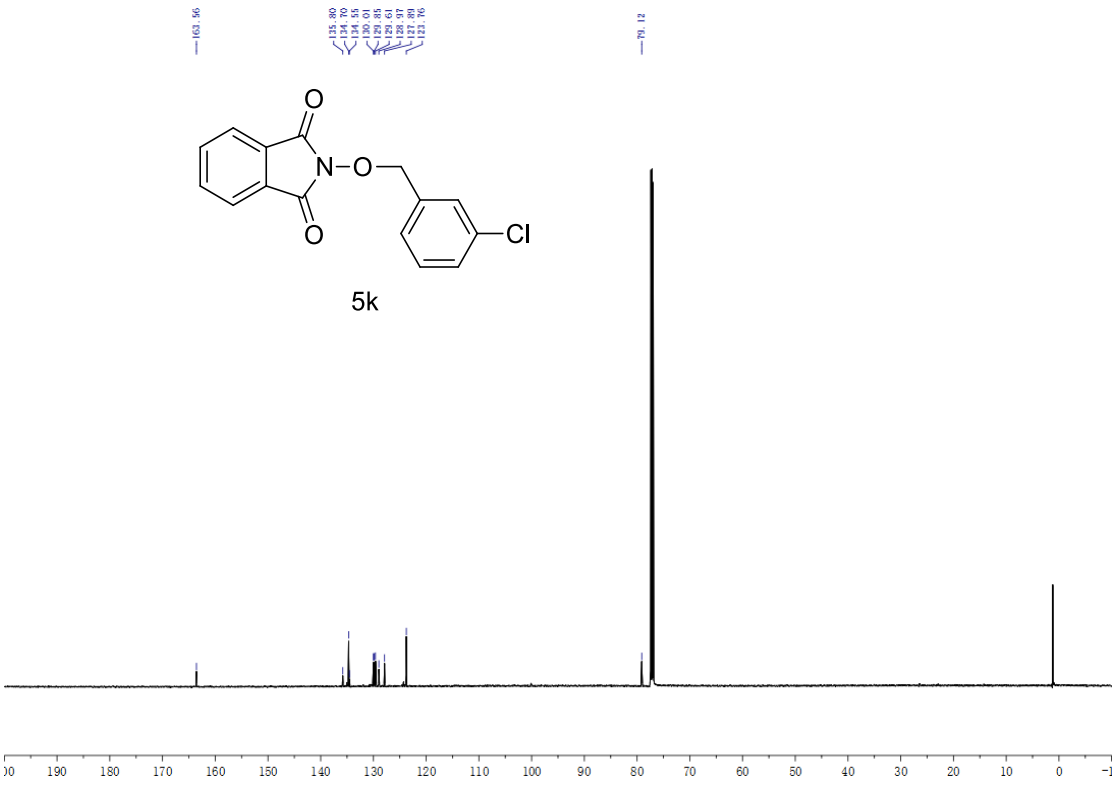
5j


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1


O
N
O
O
Cl

5k


2.00
2.12
1.00
0.97
2.00
2.00
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
0.0
-0.5


163.56
135.80
134.70
134.55
130.61
129.85
129.61
128.97
127.89
123.76
79.12
O
N
O
O
Cl
190
180
170
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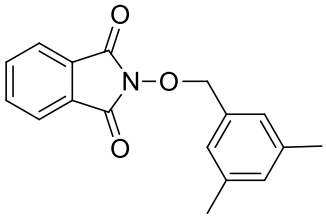
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

5k



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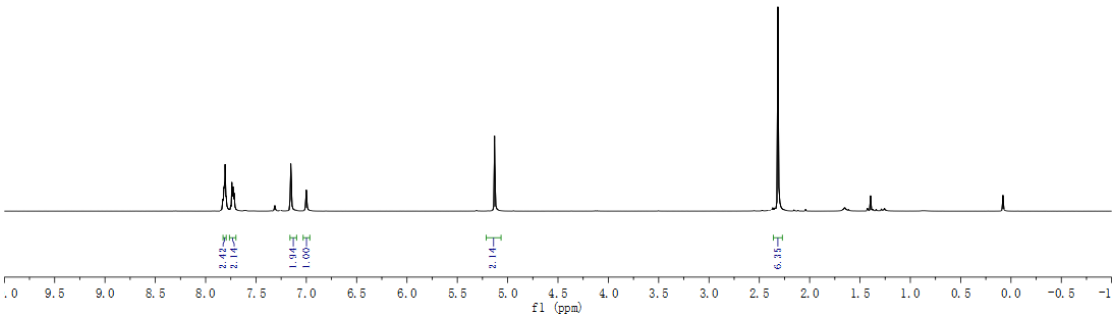
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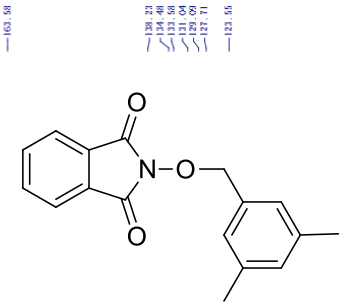
2.14
1.94
1.00
2.14
6.35
f1 (ppm)



5l

163.58
138.23
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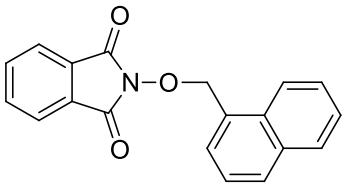
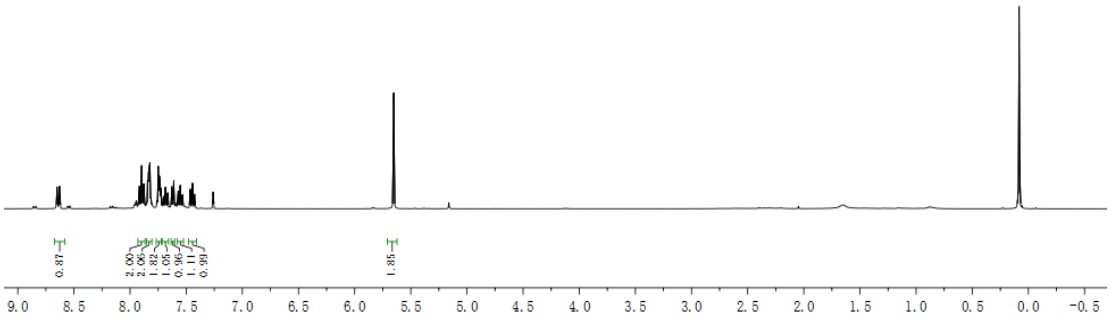
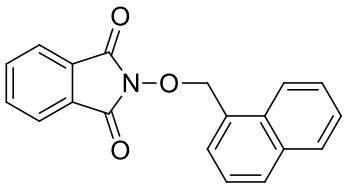
133.58
131.04
129.69
127.71
123.55
80.18
21.24
f1 (ppm)



5m


0.87
2.00
2.06
1.82
1.05
0.96
1.11
0.99
1.85
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
-0.5


163.67
134.60
133.82
132.61
130.69
129.72
129.57
129.06
128.55
127.12
126.31
125.16
124.62
123.65
78.25



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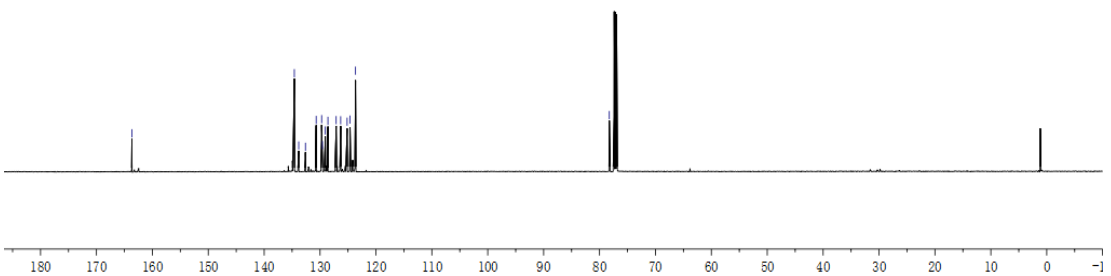
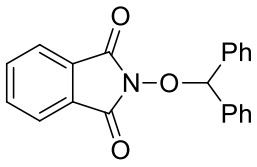
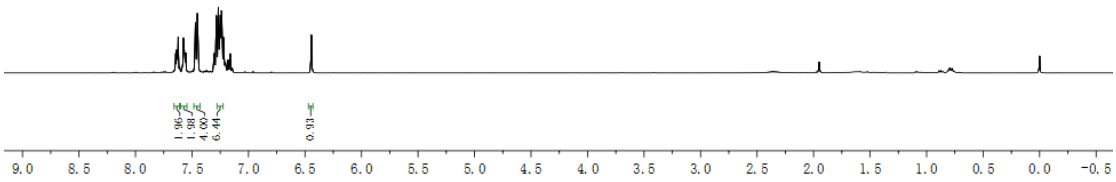
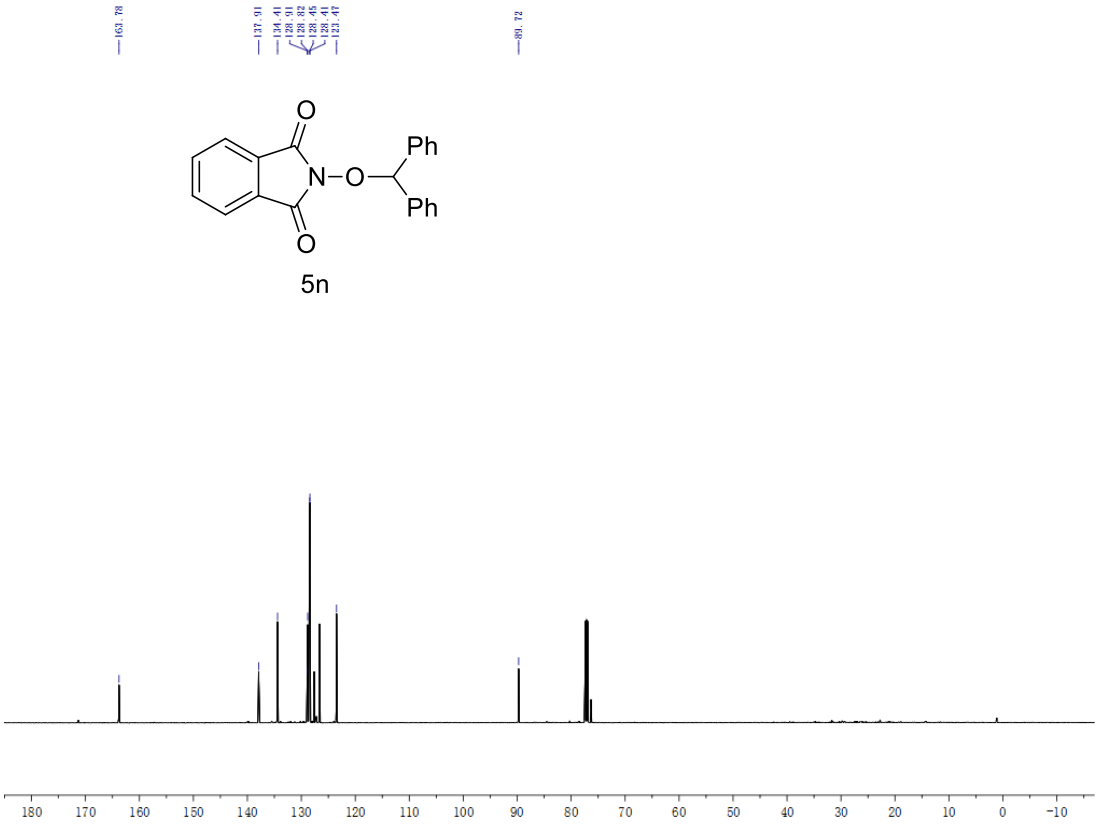

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

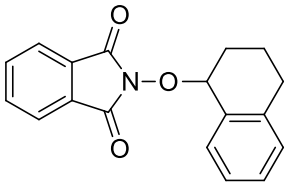

O
N
O
Ph
Ph
O

5n

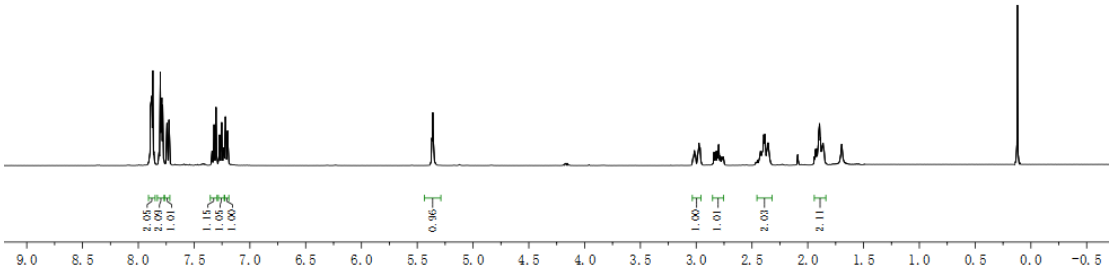

1.96
1.98
4.00
6.44
0.93
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


163.78
137.91
134.41
128.91
128.82
128.45
128.44
123.47
89.72
O
N
O
Ph
Ph
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

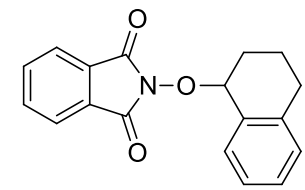
5n



5o


2.05
2.09
1.01
1.15
1.05
1.00
0.96
1.00
1.01
2.03
2.11
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 -0.5


164.43
139.03
134.52
131.93
131.56
129.19
129.16
126.04
123.96
83.29
29.12
27.49
17.84



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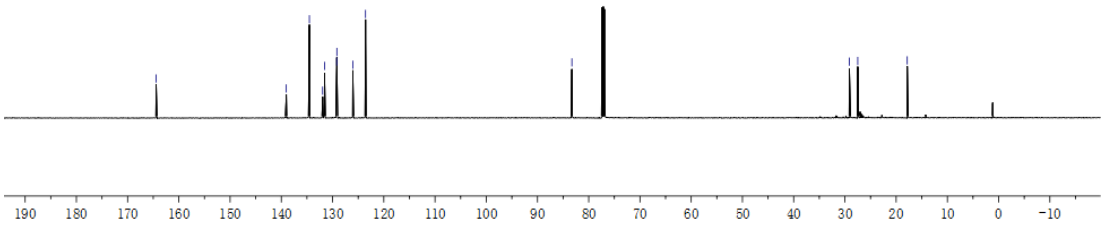
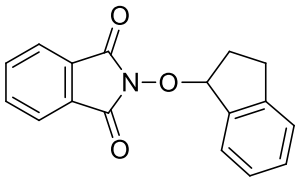
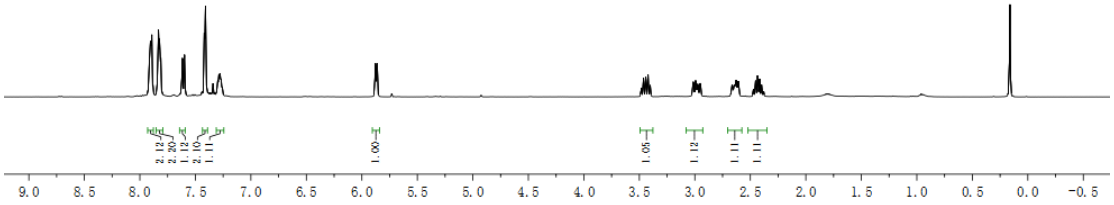
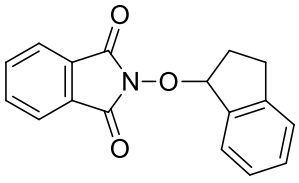

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10



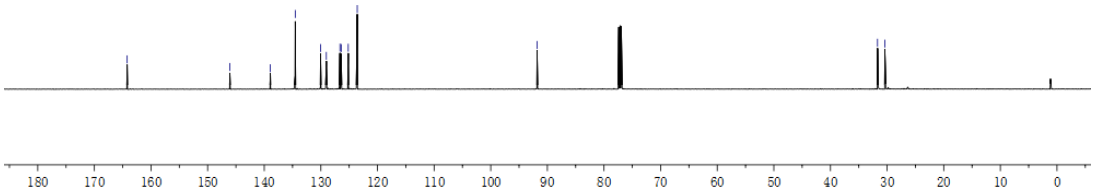
5p

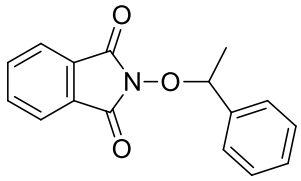

2.12
2.20
1.12
2.05
2.11
1.00
1.05
1.12
1.11
1.11
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


164.21
146.07
139.92
134.52
130.06
129.03
126.62
126.41
125.17
123.58
91.82
31.69
30.39

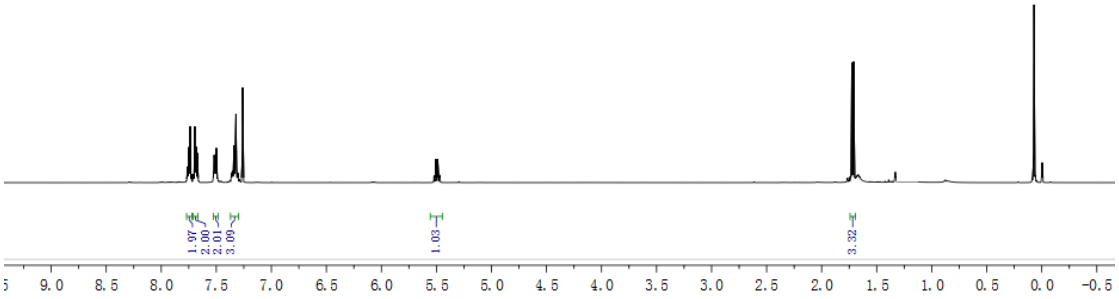


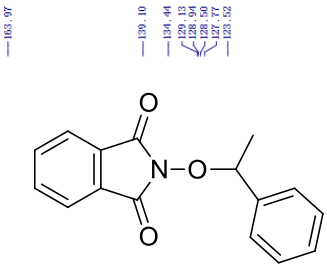
5p


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

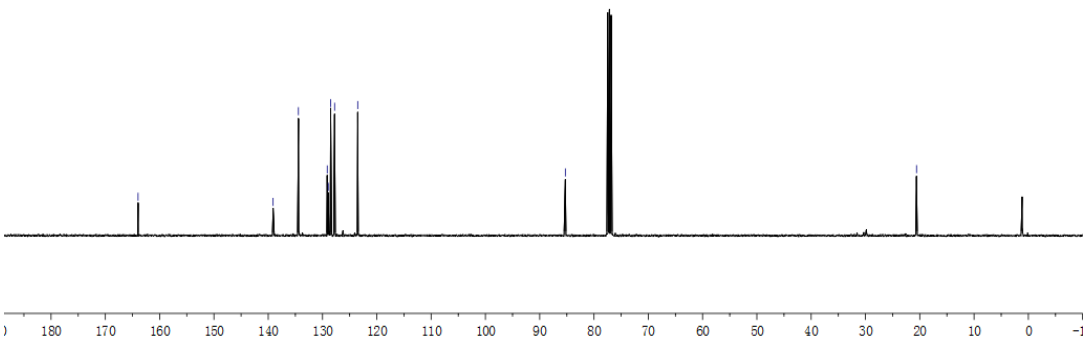


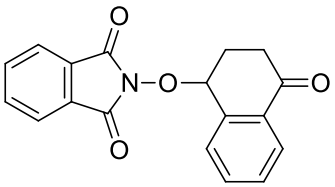
5q


1.97
2.00
2.01
3.09
1.03
3.32
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
-0.5

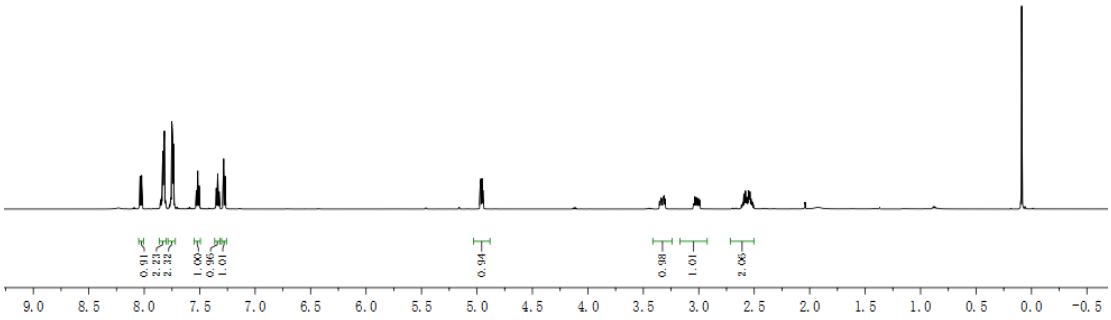


5q

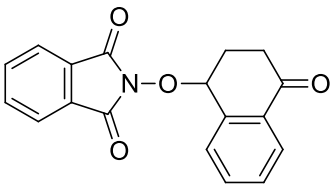

180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0



5r


0.91
2.23
2.12
1.00
0.96
1.01
0.94
0.88
1.01
2.06
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
0.5
0.0
-0.5


192.35
163.36
143.33
134.58
134.18
131.32
130.88
128.73
128.00
127.11
123.68
84.47
28.32
25.86



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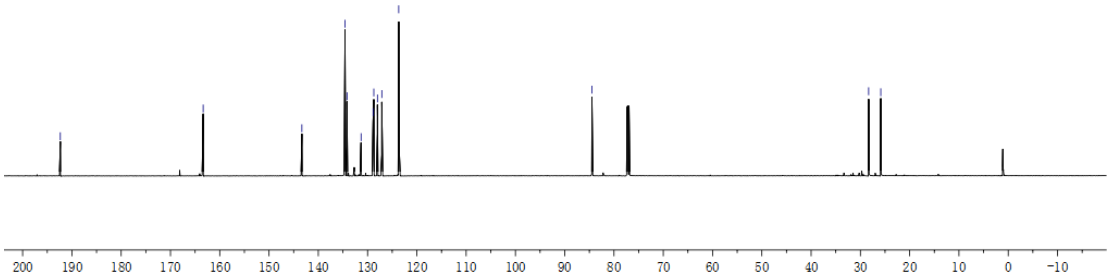
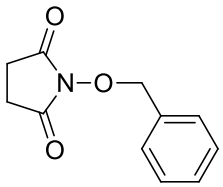
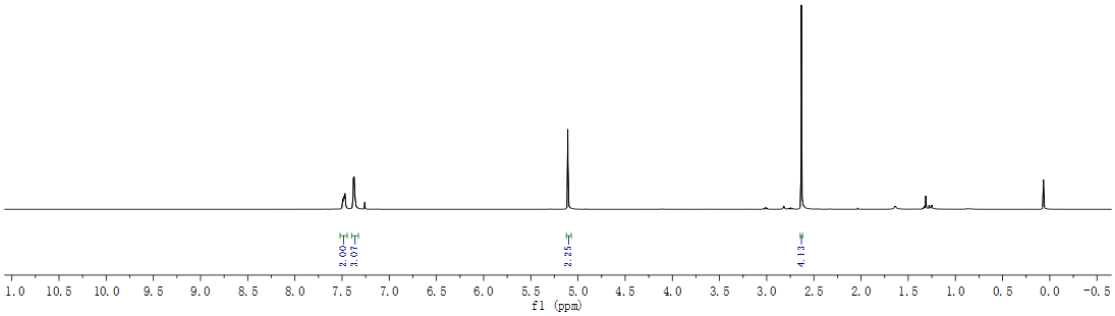
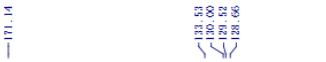
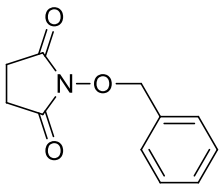

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10



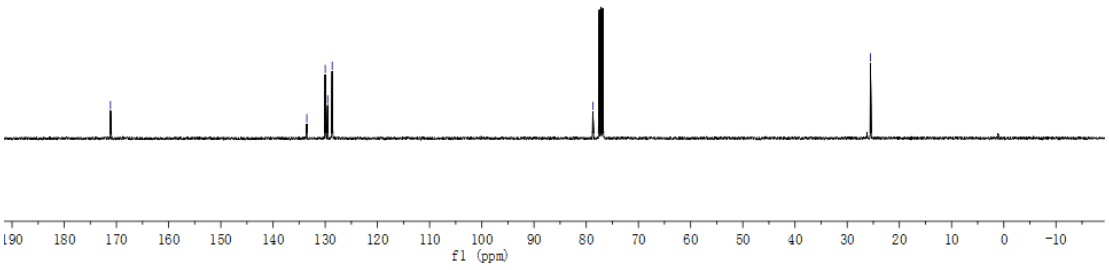
5s

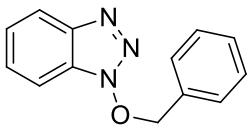

2.09
3.07
2.25
4.13
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


171.14
133.53
130.00
129.52
128.66
78.73
25.55

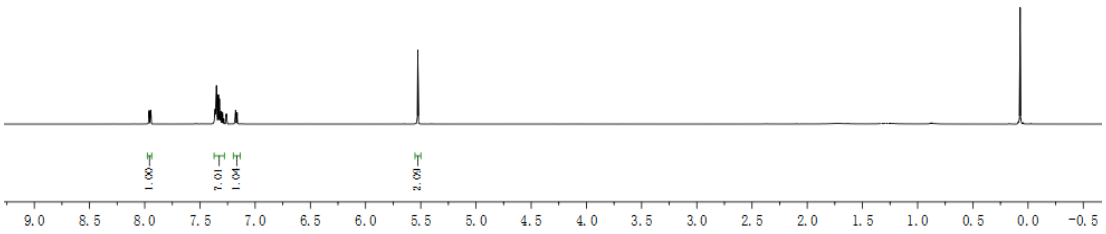


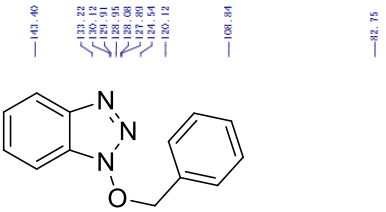
5s


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

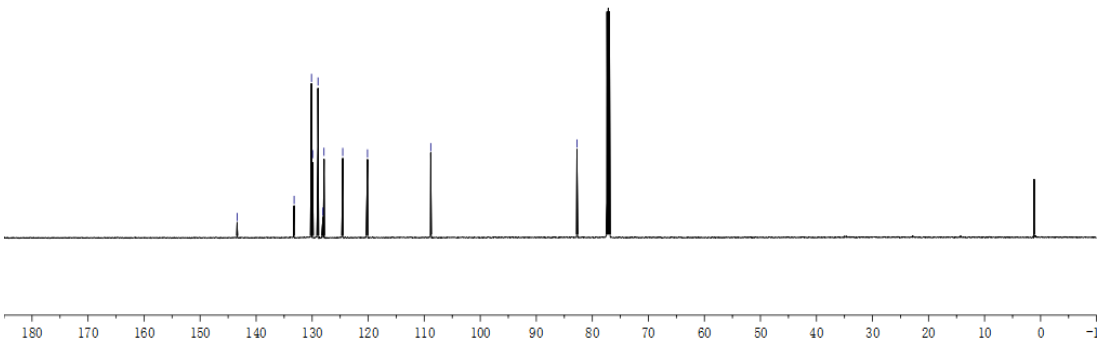


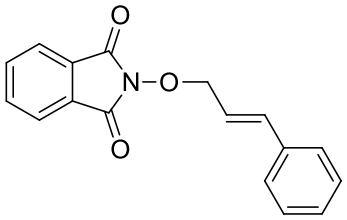
5t


1.00
7.01
1.04
2.09
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 -0.5

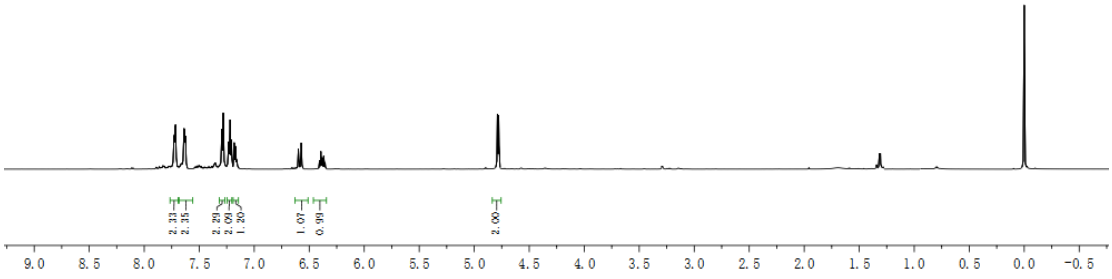


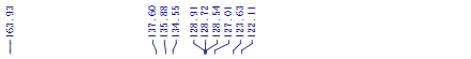
5t

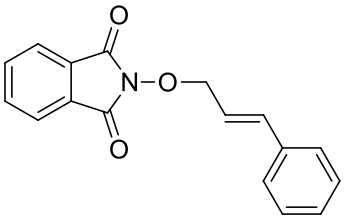

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1



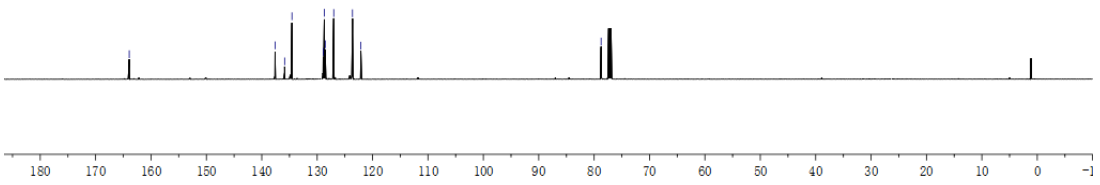
6


2.33
2.35
2.29
2.05
1.20
1.07
0.99
2.00
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 0.0 -0.5


163.93
137.60
135.88
134.55
128.91
128.72
128.54
127.01
123.63
122.11
78.74



6


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1