

Supporting Information for

Synthesis of trisubstituted hydroxylamines by visible light-promoted multicomponent reaction

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1. General

All reactions involving air- or moisture-sensitive reagents or intermediates were carried out in pre-heated glassware under an argon atmosphere using standard Schlenk techniques. THF was freshly distilled from Na under argon. All other solvents and reagents were purified according to standard procedures or were used as received from chemical suppliers. The starting materials were synthesized according to literature procedures. The light employed in this work was bought from GeAo Chemical: model H106062, 24 W blue LEDs. All reactions involving heating are carried out in an oil bath.

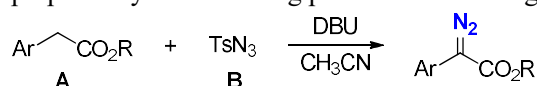
Chromatography: Analytical thin layer chromatography was performed using Qingdao Puke Parting Materials Co. silica gel plates (Silica gel 60 F254). Visualisation was by ultraviolet fluorescence ($\lambda = 254$ nm) and/or staining with phosphomolybdic acid or potassium permanganate (KMnO₄). Flash column chromatography was performed using 200-300 mesh silica gel.

¹H NMR and **¹³C NMR** spectra were recorded on a JEOL JNM ECZ000R at 300 K. Spectra were calibrated relative to solvent's residual proton and carbon chemical shift: CHCl₃ ($\delta = 7.26$ for ¹H NMR and $\delta = 77.0$ for ¹³C NMR). Data are reported as follows: chemical shift δ /ppm, integration (¹H only), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet or combinations thereof; ¹³C signals are singlets unless otherwise stated), coupling constants *J* in Hz, assignment.

High Resolution Mass Spectrometry (HRMS): All were recorded on LTQ Orbitrap XL using a positive electrospray ionization (ESI⁺). Measured values are reported to 4 decimal places of the calculated value. The calculated values are based on the most abundant isotope.

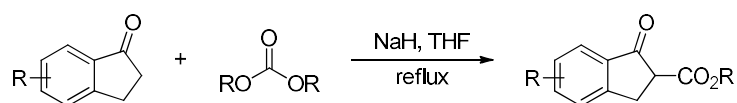
2. Preparation of Starting Materials

The aryldiazoacetates are prepared by the following procedure according to literature reports.¹⁻⁴



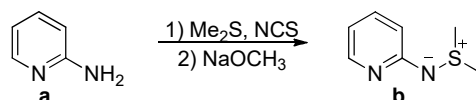
To a mixture of esters **A** (1.0 equiv.) and *p*-toluenesulfonyl azide (TsN₃) **B** (1.2 equiv.) in anhydrous acetonitrile (3 mL/mmol), DBU (1.4 equiv.) was added. The reaction mixture was stirred at room temperature for overnight. Upon the complete consumption of the starting materials, the reaction mixture was diluted with appropriate water, followed by extraction with ethyl acetate. After washing with 10% NH₄Cl solution and brine, the combined organic extracts were dried over Na₂SO₄ and concentrated by rotary evaporation. The residue was purified by flash chromatography (petroleum ether/ethyl acetate = 60/1 to 30/1) to afford the aryldiazoacetate.

The β -keto ester are prepared by the following procedure according to literature reports.⁵

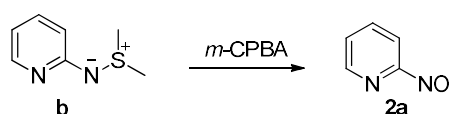


A solution of ketone (1.0 equiv.) in THF (1 L/mol) was added dropwise to a stirred solution of dialkyl carbonate (5.0-10 equiv.) in THF (2 L/mol) containing NaH (60% dispersion in mineral oil, 2.1 equiv.) under a nitrogen atmosphere. The mixture was heated to reflux (4 h), cooled in an ice-bath and then acidified with 1 mol/L hydrochloric acid (1.5 L/mol). The residue was then extracted with Et₂O (3 x 5 L/mol). The combined organic layers were dried over MgSO₄, filtered and the solvent was removed in vacuo. The crude product was purified by column chromatography to give the β-keto ester.

The 2-nitrosopyridine are prepared by the following procedure according to literature reports.⁶



To a solution of 9.40 g (0.10 mol) of 2-aminopyridine **a** and 6.80 g (8.0 mL, 0.11 mol) of dimethyl sulfide in 100 mL of DCM was added dropwise, over a period of 1 h, 13.3 g (0.10 mol) of *N*-chlorosuccinimide in 250 mL of DCM, while the temperature was maintained at -20 °C. After the addition was complete, the reaction mixture was stirred at -20 °C for 1 h and then for an additional hour at room temperature. A solution of sodium methoxide in methanol (from 4.05 g, 0.17 mol, of sodium and 75 mL of methanol) was then added, the mixture stirred for 10 min, 150 mL of water added, and stirring continued for 4 h. The organic layer was separated, and the aqueous layer was extracted with two 50-mL portions of methylene chloride. The combined organic extracts were washed with 50 mL of water, dried, and evaporated to give a thick gum which solidified upon being chilled; yield 11 g (71%). Recrystallization from diethyl ether gave a cream-colored solid.



To a solution of 20.1 g (0.119 mol, 80-90%) of m-chloroperbenzoic acid in 500 mL of dry DCM,

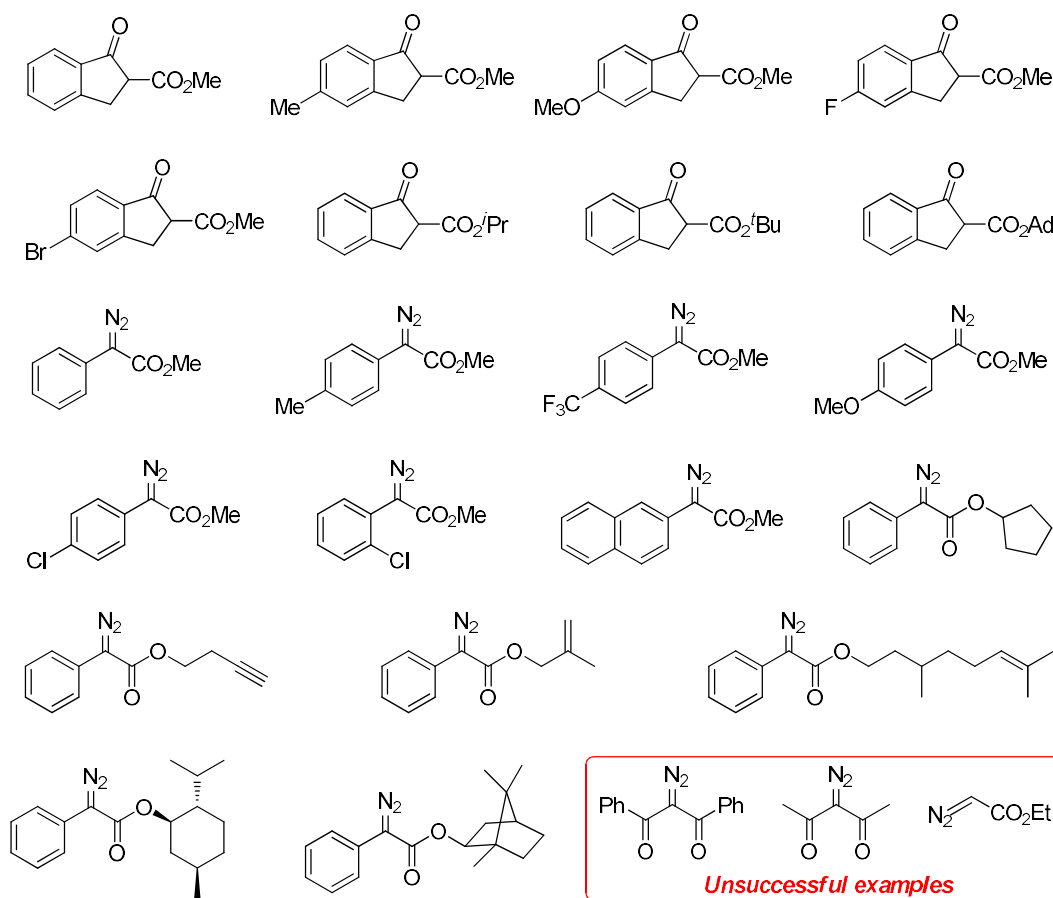
cooled to 0 °C, was added, all at once, a solution of 10.9 g (0.07 mol) of **b** in 100 mL of DCM. The mixture was stirred at 0-5 °C for 90 min, 3-4 mL of dimethyl sulfide added, and stirring continued for an additional 30 min. To the reaction mixture was then added 500 mL of a saturated aqueous solution of sodium carbonate, the layers were separated, and the green organic layer was washed with water and dried (Na₂SO₄). Evaporation of the dried extracts gave a light tan solid which was recrystallized from ethanol to give product as a light yellow solid.

Reference:

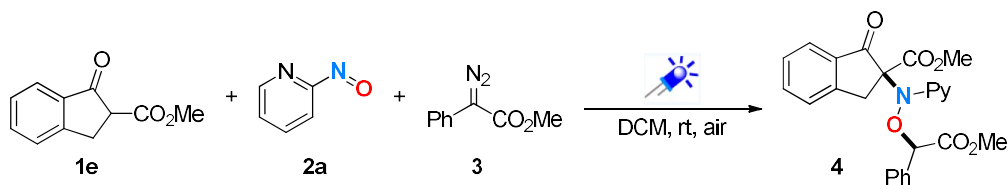
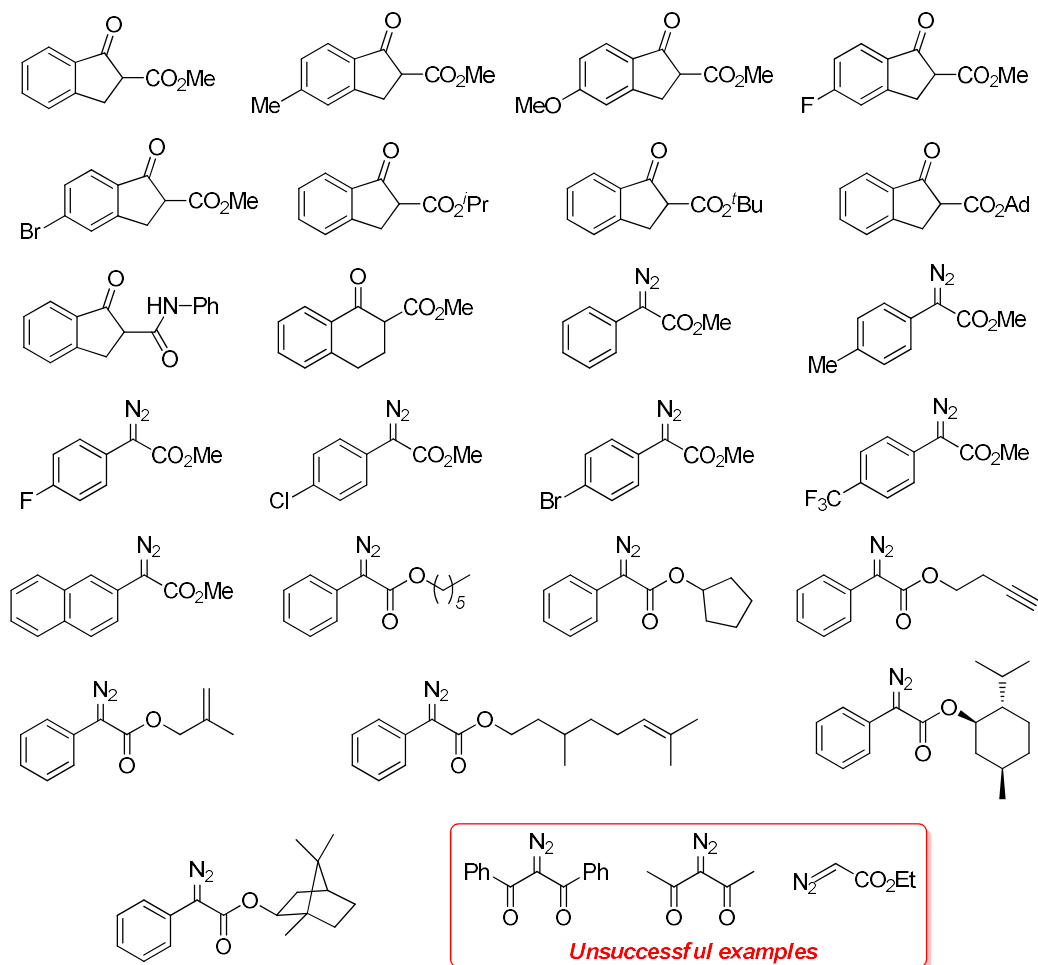
1. E. N. Bess, D. M. Guptill, H. M. L. Davies, M. S. Sigman, *Chem. Sci.* **2015**, 6, 3057.
2. X. Cheng, B.-G. Cai, H. Mao, J. Lu, L. Li, K. Wang, J. Xuan, *Org. Lett.* **2021**, 23, 4109.
3. C. Ye, B.-G. Cai, J. Lu, X. Cheng, L. Li, Z.-W. Pan, J. Xuan, *J. Org. Chem.* **2021**, 86, 1012.
4. B.-G. Cai, S.-S. Luo, L. Li, L. Li, J. Xuan, W.-J. Xiao, *CCS Chem.* **2020**, 2, 2764.
5. S. Jones, P. Zhao, *Tetrahedron: Asymmetry.* **2014**, 25, 238.
6. W. Lin, A. Gupta, K. H. Kim, D. Mendel, M. J. Miller, *Org. Lett.* **2009**, 11, 449.

3. General Procedure and Spectral Data of Products

Scope of three component reaction in DCM

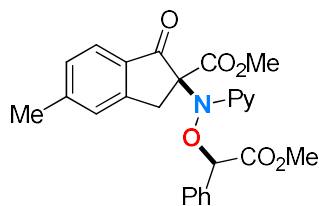


Scope of four component reaction in THF



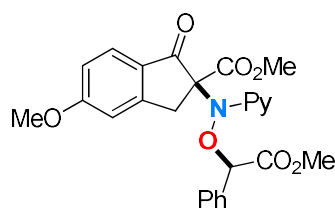
General procedure (GPI): To a 10 mL Schlenk flask equipped with a magnetic stir bar was added **1e** (0.1 mmol), **2a** (0.1 mmol), **3** (0.15 mmol), dry DCM (1.0 mL). The resulting mixture was stirred at a distance of ~3 cm from a 24 w blue LED at room temperature for 12 h. The solvent was removed by vacuum and the crude product was purified by flash chromatography on silica gel silica: 200~300; eluant: petroleum ether/ethyl acetate (20:1~5:1) to provide pure product **4** as a yellow oil in 91% yield (40.6 mg, d.r. = 1:1). ¹H NMR (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.10 – 7.95 (m, 1H), 7.63 – 7.18 (m, 11H), 6.92 – 6.72 (m, 1H), 6.04 (s, 0.5 H), 5.98 (s, 0.5 H), 4.06 (dd, *J* = 39.1, 17.1 Hz, 1H), 3.77 – 3.57 (m, 6H), 3.20 (dd, *J* = 48.0, 17.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃, 300 K): δ (ppm) = 196.4, 195.3, 171.1, 169.9, 167.6, 167.2, 160.8, 153.3, 152.7, 145.9, 145.7, 137.6, 137.4, 135.7, 135.6, 135.2, 134.9, 134.4, 134.4, 129.1, 128.8, 128.7, 128.3, 128.2, 127.7, 127.5, 127.3, 126.3, 125.9, 124.9, 124.8, 118.6, 118.1, 113.8, 112.7, 87.4, 86.3, 81.3, 81.2, 53.2, 53.1, 52.1, 52.0, 34.9, 34.7. HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₂₅H₂₃N₂O₆: 447.1551; Found: 447.1544.

Methyl 2-((2-methoxy-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-5-methyl-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (6)



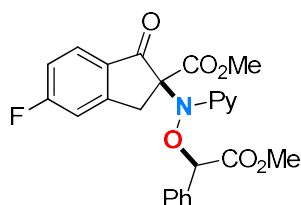
According to *GPI* with β -keto ester (20.4 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **6** and as a yellow oil in 74% yield (34.1 mg, d.r. = 1:1). ¹H NMR (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.05 (d, J = 5.0 Hz, 1H), 7.69 – 7.03 (m, 10H), 6.91 – 6.73 (m, 1H), 6.05 (s, 0.5 H), 5.98 (s, 0.5 H), 4.00 (dd, J = 46.9, 17.1 Hz, 1H), 3.78 – 3.58 (m, 6H), 3.14 (dd, J = 55.8, 17.1 Hz, 1H), 2.40 (d, J = 28.2 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃, 300 K): δ (ppm) = 196.1, 195.1, 171.4, 170.1, 167.9, 167.6, 161.1, 154.2, 153.4, 147.3, 146.9, 146.1, 146.0, 137.8, 137.6, 136.0, 135.1, 132.3, 132.3, 129.2, 129.1, 129.0, 128.9, 128.8, 128.5, 128.4, 128.0, 126.9, 126.5, 125.0, 124.9, 118.7, 118.2, 114.0, 112.8, 87.6, 86.4, 81.8, 81.6, 53.4, 53.3, 52.3, 52.1, 34.9, 34.7, 22.4, 22.2. HRMS (ESI) m/z : [M+H]⁺ Calcd for C₂₆H₂₅N₂O₆: 461.1707; Found: 461.1696.

Methyl 5-methoxy-2-((2-methoxy-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (7)



According to *GPI* with β -keto ester (22.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **7** and as a yellow oil in 63% yield (30.2 mg, d.r. = 1:1). ¹H NMR (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.12 – 8.03 (m, 1H), 7.73 – 7.21 (m, 8H), 6.94 – 6.74 (m, 3H), 6.06 (s, 0.5H), 5.98 (s, 0.5H), 4.07 (d, J = 17.0 Hz, 1H), 3.88 – 3.60 (m, 9H), 3.13 (dd, J = 59.1, 17.2 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃, 300 K): δ (ppm) = 194.6, 193.7, 171.3, 170.0, 167.8, 167.7, 166.1, 165.9, 161.0, 156.6, 156.2, 146.0, 145.9, 137.6, 137.4, 135.9, 134.9, 129.1, 128.8, 128.7, 128.3, 127.8, 127.6, 126.8, 126.7, 118.6, 118.0, 116.0, 115.8, 114.0, 112.7, 109.3, 108.9, 87.5, 86.1, 81.7, 81.4, 55.6, 55.6, 53.2, 53.1, 52.1, 52.0, 35.0, 34.8. HRMS (ESI) m/z : [M+H]⁺ Calcd for C₂₆H₂₅N₂O₇: 477.1656; Found: 477.1637.

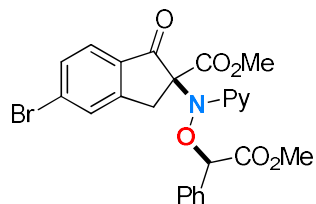
Methyl 5-fluoro-2-((2-methoxy-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (8)



According to *GPI* with β -keto ester (20.8 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **8** and as a yellow oil in 82% yield (38.0 mg, d.r. = 1:1). ¹H NMR (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.03 (s, 1H), 7.81 – 7.23 (m, 8H), 7.13 – 6.76 (m, 3H), 6.01 (s, 0.5H), 5.97 (s, 0.5H), 4.06 (dd, J = 37.7, 17.4 Hz, 1H), 3.68 (dd, J = 35.1, 29.3 Hz, 6H), 3.19 (dd, J = 47.3, 17.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃, 300 K): δ (ppm) = 194.7, 171.1, 169.9, 168.9, 168.6, 167.4, 167.1, 166.0, 160.7, 156.4, 155.7, 145.9, 145.7, 137.8, 137.5, 135.7, 134.9, 130.8, 129.1, 128.8, 128.7, 128.5, 128.3, 128.1, 127.6, 127.4, 127.3, 127.2, 127.1, 126.5, 118.8, 118.3, 116.1, 115.9, 115.8, 115.6, 113.9, 113.2, 113.0, 112.8, 112.7, 112.6,

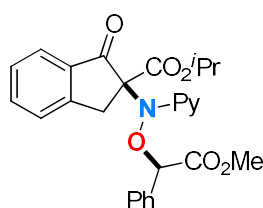
87.4, 86.4, 81.4, 81.3, 53.3, 53.2, 52.1, 52.0, 34.8, 34.6. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{25}H_{22}FN_2O_6$: 465.1456; Found: 465.1430.

Methyl 5-bromo-2-((2-methoxy-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (9)



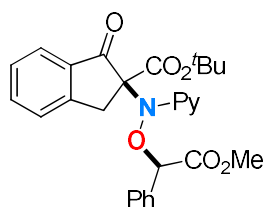
According to *GPI* with β -keto ester (26.8 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **9** and as a yellow oil in 72% yield (37.6 mg, d.r. = 1:1). **¹H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.00 (d, J = 18.9 Hz, 1H), 7.65 – 7.26 (m, 10H), 6.82 (d, J = 30.7 Hz, 1H), 6.01 (s, 1H), 4.05 (dd, J = 33.7, 17.4 Hz, 1H), 3.76 – 3.59 (m, 6H), 3.19 (dd, J = 46.1, 17.4 Hz, 1H). **¹³C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 171.0, 169.9, 167.3, 166.9, 160.6, 154.7, 154.0, 145.9, 145.6, 137.8, 137.5, 135.6, 135.0, 133.4, 133.3, 131.2, 130.9, 129.6, 129.2, 129.1, 128.9, 128.7, 128.4, 128.0, 127.6, 126.0, 125.9, 118.8, 118.3, 113.8, 112.8, 87.4, 86.6, 81.1, 77.3, 53.3, 53.2, 52.1, 52.1, 34.5, 34.4. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{25}H_{22}BrN_2O_6$: 525.0656; Found: 525.0625.

Isopropyl 2-((2-methoxy-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (10)



According to *GPI* with β -keto ester (21.8 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **10** and as a yellow oil in 75% yield (35.4 mg, d.r. = 1:1). **¹H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.02 (s, 1H), 7.81 – 7.22 (m, 11H), 6.80 (d, J = 45.2 Hz, 1H), 6.07 (s, 0.5H), 6.03 (s, 0.5H), 5.07 – 4.94 (m, 1H), 4.05 (dd, J = 36.7, 17.2 Hz, 1H), 3.63 (d, J = 29.2 Hz, 3H), 3.18 (dd, J = 35.2, 17.2 Hz, 1H), 1.27 (d, J = 7.7 Hz, 3H), 1.12 – 1.06 (m, 3H). **¹³C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 171.1, 169.9, 166.3, 166.0, 160.7, 153.4, 152.8, 145.6, 145.4, 137.4, 137.0, 135.5, 135.4, 134.9, 134.6, 134.4, 129.0, 128.6, 128.5, 128.2, 127.6, 127.3, 127.1, 126.1, 125.7, 124.7, 124.6, 118.4, 117.8, 113.7, 112.5, 86.8, 86.2, 81.2, 81.2, 69.5, 69.4, 52.0, 51.9, 35.1, 35.0, 21.3, 21.3, 20.9. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{27}H_{27}N_2O_6$: 475.1864; Found: 475.1840.

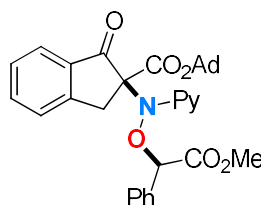
Tert-butyl 2-((2-methoxy-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (11)



According to *GPI* with β -keto ester (23.2 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **11** and as a yellow oil in 78% yield (38.3 mg, d.r. = 1:1). **¹H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.02 (s, 1H), 7.80 – 7.24 (m, 11H), 6.79 (d, J = 43.2 Hz, 1H), 6.11 (s, 0.5H), 6.04 (s, 0.5H), 4.01 (dd, J = 42.6, 17.1 Hz, 1H), 3.63 (d, J = 18.4 Hz, 3H), 3.13 (dd, J = 33.1, 17.2 Hz, 1H), 1.38 (s, 9H). **¹³C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 196.7, 171.2, 170.0, 165.7, 165.3, 161.0, 160.9, 153.6, 152.9, 145.7, 145.5, 137.3, 137.1, 135.5, 135.4, 135.0,

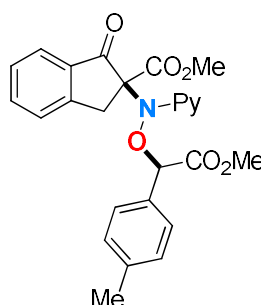
134.9, 134.6, 129.0, 128.8, 128.7, 128.6, 128.4, 127.8, 127.3, 127.1, 126.1, 125.7, 124.7, 124.6, 118.3, 117.8, 113.7, 112.4, 86.8, 86.3, 82.0, 81.9, 81.8, 52.0, 51.9, 35.4, 35.3, 27.5. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{28}H_{29}N_2O_6$: 489.2020; Found: 489.1994.

Adamantan-1-yl 2-((2-methoxy-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (12)



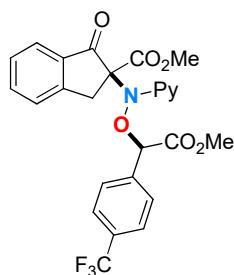
According to *GPI* with β -keto ester (31.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **12** and as a yellow oil in 89% yield (50.1 mg, d.r. = 1:1). **1H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.03 (d, J = 16.7 Hz, 1H), 7.80 – 7.24 (m, 11H), 6.78 (d, J = 38.3 Hz, 1H), 6.12 (s, 0.5H), 6.05 (s, 0.5H), 4.00 (dd, J = 39.1, 17.2 Hz, 1H), 3.63 (d, J = 21.1 Hz, 3H), 3.13 (dd, J = 30.0, 17.2 Hz, 1H), 2.07 (d, J = 20.7 Hz, 9H), 1.59 (s, 6H). **^{13}C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 196.6, 171.1, 170.0, 165.3, 164.9, 160.8, 153.5, 152.8, 145.7, 145.5, 137.3, 137.0, 135.5, 135.3, 135.1, 134.9, 134.8, 134.6, 129.0, 128.7, 128.6, 128.5, 128.3, 127.8, 127.3, 127.0, 126.0, 125.6, 124.7, 124.5, 118.3, 117.7, 113.7, 112.4, 86.7, 86.3, 81.9, 81.9, 81.8, 51.9, 51.9, 40.6, 36.0, 35.4, 35.3, 30.7. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{34}H_{35}N_2O_6$: 567.2490; Found: 567.2466.

Methyl 2-((2-methoxy-2-oxo-1-(p-tolyl)ethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (13)



According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (28.5 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **13** and as a yellow oil in 82% yield (37.7 mg, d.r. = 1:1). **1H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.02 (d, J = 11.9 Hz, 1H), 7.79 – 7.03 (m, 10H), 6.90 – 6.73 (m, 1H), 6.01 (s, 0.5H), 5.94 (s, 0.5H), 4.05 (dd, J = 44.0, 17.1 Hz, 1H), 3.67 (dd, J = 41.0, 31.7 Hz, 6H), 3.19 (dd, J = 51.7, 17.1 Hz, 1H), 2.31 (d, J = 24.5 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 196.5, 171.3, 170.1, 167.6, 160.9, 153.4, 152.7, 145.9, 145.7, 139.0, 138.8, 137.6, 137.4, 135.6, 135.2, 134.5, 134.4, 132.7, 131.9, 129.4, 129.1, 128.1, 127.7, 127.5, 127.2, 126.3, 125.9, 124.9, 124.8, 118.6, 118.1, 113.9, 112.7, 87.2, 86.1, 81.4, 81.2, 53.2, 53.1, 52.0, 51.9, 34.9, 34.7, 21.2, 21.1. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{26}H_{25}N_2O_6$: 461.1707; Found: 461.1680.

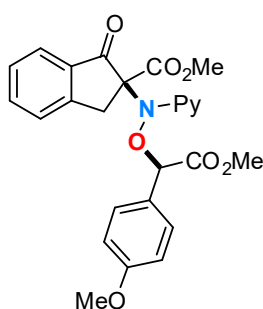
Methyl 2-((2-methoxy-2-oxo-1-(4-(trifluoromethyl)phenyl)ethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (14)



According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (36.6 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **14** and as a yellow oil in 47% yield (24.1 mg, d.r. = 1:1). **1H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.08 (d, J = 8.3 Hz, 1H), 7.78 – 7.21 (m, 10H), 6.93 (s, 0.5H), 6.82 (s,

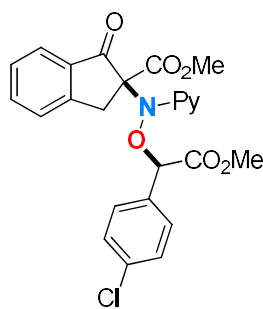
0.5H), 6.07 (d, $J = 11.0$ Hz, 1H), 4.15 – 4.03 (m, 1H), 3.77 – 3.60 (m, 6H), 3.23 (dd, $J = 42.1, 17.2$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 170.6, 169.4, 167.3, 167.1, 160.6, 153.4, 153.1, 146.2, 146.1, 139.5, 139.0, 137.9, 137.6, 135.8, 135.6, 134.4, 134.2, 128.5, 127.8, 127.6, 127.4, 126.9, 126.4, 126.0, 125.7, 125.7, 125.2, 125.0, 124.7, 119.0, 118.5, 113.9, 112.6, 86.6, 85.9, 81.3, 81.2, 53.3, 53.2, 52.4, 52.3, 34.9, 34.7. **HRMS** (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{26}\text{H}_{22}\text{F}_3\text{N}_2\text{O}_6$: 515.1424; Found: 515.1402.

Methyl 2-((2-methoxy-1-(4-methoxyphenyl)-2-oxoethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (15)



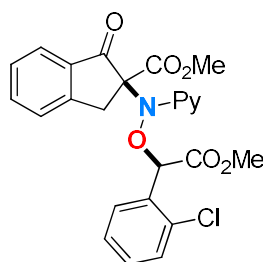
According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (30.9 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **15** and as a yellow oil in 91% yield (43.2 mg, d.r. = 1:1). ^1H NMR (400 MHz, CDCl_3 , 300 K): δ (ppm) = 8.00 (d, $J = 9.6$ Hz, 1H), 7.76 – 7.24 (m, 8H), 6.79 (dd, $J = 46.1, 6.6$ Hz, 3H), 5.97 (s, 0.5H), 5.90 (s, 0.5H), 4.03 (dd, $J = 45.6, 17.1$ Hz, 1H), 3.75 – 3.67 (m, 6H), 3.59 (d, $J = 30.4$ Hz, 3H), 3.15 (dd, $J = 50.5, 17.2$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 196.4, 171.3, 170.0, 167.5, 167.2, 160.9, 160.1, 159.9, 153.4, 152.7, 145.9, 145.6, 137.6, 137.4, 135.6, 135.2, 134.4, 134.3, 129.7, 129.3, 127.8, 127.5, 127.2, 126.3, 125.9, 124.8, 124.7, 118.5, 118.0, 114.0, 113.7, 112.6, 86.7, 85.7, 81.3, 81.1, 55.1, 55.1, 53.1, 53.1, 52.0, 51.8, 34.9, 34.7. **HRMS** (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{26}\text{H}_{25}\text{N}_2\text{O}_7$: 477.1656; Found: 477.1634.

Methyl 2-((1-(4-chlorophenyl)-2-methoxy-2-oxoethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (16)



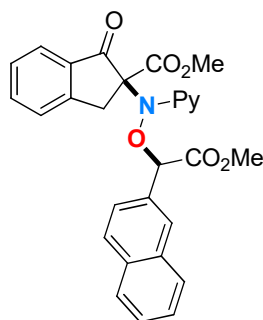
According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (31.5 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **16** and as a yellow oil in 84% yield (40.3 mg, d.r. = 1:1). ^1H NMR (400 MHz, CDCl_3 , 300 K): δ (ppm) = 8.05 (s, 1H), 7.80 – 7.16 (m, 10H), 6.85 (d, $J = 37.9$ Hz, 1H), 6.02 (s, 0.5H), 5.97 (s, 0.5H), 4.06 (dd, $J = 32.4, 17.1$ Hz, 1H), 3.67 (dd, $J = 35.5, 30.7$ Hz, 6H), 3.19 (dd, $J = 44.7, 17.2$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 196.4, 170.8, 169.5, 167.3=4, 167.1, 160.7, 153.3, 152.9, 146.1, 145.8, 137.7, 137.5, 135.7, 135.4, 135.1, 134.7, 134.3, 134.1, 133.5, 129.6, 129.0, 128.9, 128.6, 128.5, 127.9, 127.5, 127.3, 126.3, 125.9, 124.9, 124.7, 118.8, 118.3, 113.8, 112.5, 86.4, 85.6, 81.2, 53.2, 53.1, 52.2, 52.1, 34.9, 34.7. **HRMS** (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{25}\text{H}_{22}\text{ClN}_2\text{O}_6$: 481.1161; Found: 481.1144.

Methyl 2-((1-(2-chlorophenyl)-2-methoxy-2-oxoethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (17)



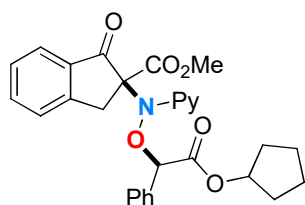
According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (31.5 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **17** and as a yellow oil in 52% yield (24.9 mg, d.r. = 1:1). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.02 (d, J = 18.9 Hz, 1H), 7.80 – 7.06 (m, 10H), 6.87 (s, 0.5H), 6.79 (s, 0.5H), 6.38 (s, 1H), 4.07 (t, J = 15.3 Hz, 1H), 3.76 – 3.59 (m, 6H), 3.23 (t, J = 19.2 Hz, 1H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 170.4, 169.3, 167.7, 167.4, 160.9, 153.2, 152.5, 146.1, 145.8, 137.7, 137.6, 135.6, 135.1, 134.7, 134.5, 134.1, 133.8, 133.6, 133.4, 130.9, 130.3, 129.9, 129.9, 129.6, 129.4, 127.5, 127.3, 127.2, 126.7, 126.3, 125.9, 125.0, 124.8, 118.7, 118.2, 113.6, 112.1, 84.5, 83.3, 81.4, 81.0, 53.2, 53.2, 52.3, 52.2, 34.9, 34.8. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₂₅H₂₂ClN₂O₆: 481.1161; Found: 481.1133.

Methyl 2-((2-methoxy-1-(naphthalen-2-yl)-2-oxoethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (18)



According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (33.9 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **18** and as a yellow oil in 70% yield (34.5 mg, d.r. = 1:1). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.04 – 7.41 (m, 12H), 7.33 – 6.52 (m, 3H), 6.72 (s, 0.5H), 6.59 (s, 0.5H), 6.21 (d, J = 2.5 Hz, 1H), 4.09 (dd, J = 37.2, 17.2 Hz, 1H), 3.78 – 3.58 (m, 6H), 3.25 (dd, J = 49.7, 17.2 Hz, 1H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 197.4, 171.2, 170.0, 167.6, 167.2, 160.8, 153.4, 152.7, 145.9, 145.7, 137.7, 137.5, 135.7, 135.2, 134.4, 133.4, 133.2, 133.1, 133.0, 132.8, 132.4, 128.6, 128.3, 128.2, 128.0, 127.6, 127.5, 127.5, 127.3, 127.2, 126.6, 126.5, 126.4, 126.3, 126.3, 126.1, 125.8, 125.1, 124.9, 124.8, 124.7, 118.7, 118.2, 113.9, 112.8, 87.6, 86.5, 81.3, 81.2, 53.2, 53.1, 52.2, 52.0, 34.9, 34.7. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₂₉H₂₅N₂O₆: 497.1707; Found: 497.1684.

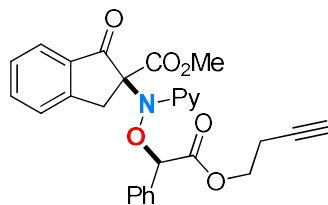
Methyl 2-((2-(cyclopentyloxy)-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (19)



According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (34.5 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **19** and as a yellow oil in 75% yield (37.6 mg, d.r. = 1:1). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.01 (s, 1H), 7.79 – 7.19 (m, 11H), 6.86 (s, 0.5H), 6.75 (s, 0.5H), 5.95 (d, J = 11.4 Hz, 1H), 5.12 (d, J = 40.1 Hz, 1H), 4.07 (dd, J = 28.8, 17.1 Hz, 1H), 3.70 (d, J = 47.8 Hz, 3H), 3.21 (dd, J = 45.7, 17.1 Hz, 1H), 1.83 – 1.42 (m, 8H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 170.3, 169.2, 167.5, 167.2, 160.8, 153.2, 152.7, 145.8, 145.7, 137.6, 137.3, 136.1, 135.5, 135.3, 135.1, 134.4, 134.4, 128.8, 128.5, 128.4, 128.1, 128.0, 127.5, 127.4, 127.2, 126.2, 125.8, 124.9, 124.7, 118.5, 118.0, 113.8, 112.7, 87.4,

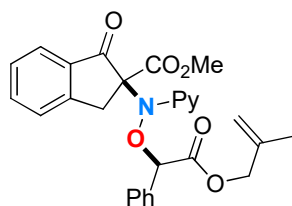
86.6, 81.3, 81.1, 78.1, 78.0, 53.1, 53.1, 34.8, 34.7, 32.4, 32.3, 32.3, 32.2, 32.0, 23.5, 23.4, 23.3.
HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₂₉H₂₉N₂O₆: 501.2020; Found: 501.1996.

Methyl 2-((2-(but-3-yn-1-yloxy)-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (20)



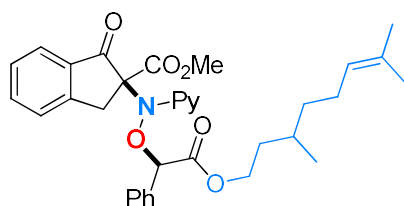
According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (32.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **20** and as a yellow oil in 63% yield (30.6 mg, d.r. = 1:1). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.03 (s, 1H), 7.65 – 7.22 (m, 11H), 6.82 (d, *J* = 38.6 Hz, 1H), 6.05 (s, 0.5H), 5.99 (s, 0.5H), 4.25 – 3.98 (m, 3H), 3.71 (d, *J* = 39.3 Hz, 3H), 3.21 (dd, *J* = 49.6, 17.1 Hz, 1H), 2.41 (dt, *J* = 33.6, 7.3 Hz, 2H), 1.92 – 1.86 (m, 1H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 170.4, 169.2, 167.5, 167.1, 160.8, 153.4, 152.7, 145.9, 145.7, 137.6, 137.4, 135.7, 135.6, 135.2, 134.9, 134.4, 134.3, 129.1, 128.7, 128.7, 128.3, 128.1, 127.6, 127.6, 127.3, 126.5, 126.3, 125.9, 124.9, 124.8, 118.7, 118.2, 113.8, 112.7, 87.4, 86.4, 81.3, 81.3, 79.4, 79.3, 70.0, 69.9, 63.4, 62.6, 62.5, 53.2, 53.1, 34.8, 34.7, 18.6, 18.5. **HRMS** (ESI) *m/z*: [M+H]⁺ Calcd for C₂₈H₂₅N₂O₆: 485.1707; Found: 485.1683.

Methyl 2-((2-((2-methylallyl)oxy)-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (21)



According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (32.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **21** and as a yellow oil in 64% yield (31.0 mg, d.r. = 1:1). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.02 (s, 1H), 7.77 – 7.23 (m, 11H), 6.90 (s, 0.5H), 6.77 (s, 0.5H), 6.05 (d, *J* = 22.3 Hz, 1H), 4.79 (d, *J* = 12.2 Hz, 2H), 4.44 (d, *J* = 55.8 Hz, 2H), 4.07 (dd, *J* = 31.9, 17.1 Hz, 1H), 3.71 (d, *J* = 39.3 Hz, 3H), 3.21 (dd, *J* = 48.6, 17.2 Hz, 1H), 1.58 (s, 3H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 170.4, 169.1, 167.6, 167.3, 160.9, 153.3, 152.8, 145.9, 145.7, 139.3, 139.2, 137.7, 137.4, 135.9, 135.6, 135.2, 134.5, 134.4, 129.0, 128.7, 128.7, 128.5, 128.3, 128.2, 127.7, 127.5, 127.3, 126.5, 126.3, 125.9, 125.0, 124.8, 118.6, 118.2, 113.9, 113.0, 112.9, 112.8, 87.3, 86.5, 81.3, 81.3, 68.1, 67.9, 53.2, 53.1, 34.9, 34.8, 19.1, 19.1. **HRMS** (ESI) *m/z*: [M+H]⁺ Calcd for C₂₈H₂₇N₂O₆: 487.1864; Found: 487.1844.

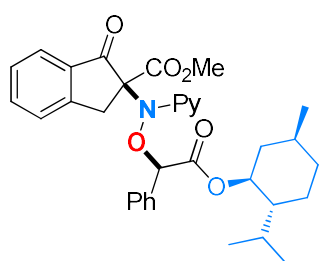
Methyl 2-((2-((3,7-dimethyloct-6-en-1-yl)oxy)-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (22)



According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (45.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **22** and as a yellow oil in 60% yield (34.2 mg, d.r. = 1:1). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.02 (s, 1H), 7.82 – 7.45 (m, 5H), 7.33 (d, *J* = 9.8 Hz, 4H), 7.21 (d, *J* =

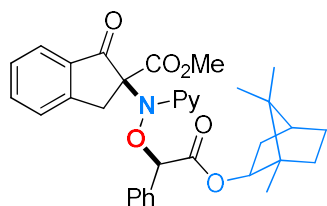
6.6 Hz, 2H), 6.82 (d, J = 34.9 Hz, 1H), 5.99 (d, J = 15.5 Hz, 1H), 5.04 (d, J = 7.7 Hz, 1H), 4.10 (d, J = 15.0 Hz, 2H), 3.77 (d, J = 10.4 Hz, 2H), 3.66 (s, 2H), 3.21 (dd, J = 48.4, 17.2 Hz, 1H), 1.94 – 1.81 (m, 2H), 1.67 (s, 3H), 1.57 (s, 3H), 1.24 (t, J = 56.2 Hz, 5H), 0.80 (t, J = 9.6 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 170.9, 169.6, 167., 167.3, 160.9, 153.3, 152.8, 145.8, 145.7, 137.6, 137.4, 136.0, 135.6, 135.4, 135.2, 134.4, 131.1, 128.9, 128.6, 128.4, 128.2, 128.1, 127.8, 127.6, 127.5, 127.3, 126.5, 126.4, 126.3, 125.9, 124.9, 124.8, 124.6, 124.5, 124.5, 118.6, 118.1, 113.9, 112.8, 87.4, 86.6, 81.3, 81.2, 63.6, 63.5, 53.2, 53.1, 53.1, 52.7, 36.8, 36.8, 35.1, 35.0, 35.0, 34.8, 34.7, 30.2, 29.2, 25.6, 25.3, 19.1, 19.1, 19.0, 17.6. **HRMS** (ESI) m/z : [M+H]⁺ Calcd for C₃₄H₃₉N₂O₆: 571.2803; Found: 571.2777.

Methyl 2-((2-(((1S,2R,5S)-2-isopropyl-5-methylcyclohexyl)oxy)-2-oxo-1-phenylethoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (23)



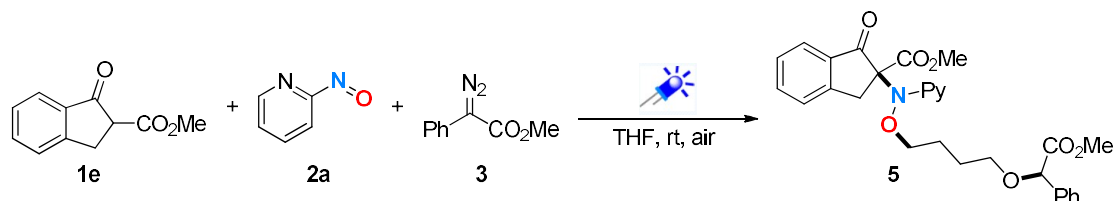
According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (45.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **23** and as a yellow oil in 62% yield (35.3 mg, d.r. = 1:1). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.03 (s, 1H), 7.78 – 7.17 (m, 11H), 6.80 (d, J = 49.7 Hz, 1H), 6.07 – 5.91 (m, 1H), 4.74 – 4.47 (m, 1H), 4.15 – 3.98 (m, 1H), 3.78 – 3.60 (m, 3H), 3.21 (dd, J = 45.7, 17.2 Hz, 1H), 2.22 – 1.89 (m, 1H), 1.66 – 1.34 (m, 3H), 1.27 – 0.70 (m, 10H), 0.62 – 0.29 (m, 4H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 169.1, 167.6, 167.3, 160.9, 153.4, 145.8, 145.8, 137.6, 137.5, 137.4, 137.3, 136.2, 135.6, 135.5, 135.1, 134.4, 128.9, 128.8, 128.6, 128.5, 128.2, 128.1, 128.0, 127.7, 127.5, 127.4, 127.3, 127.2, 126.3, 126.2, 125.9, 125.8, 125.0, 124.9, 124.7, 118.5, 118.0, 114.1, 113.7, 112.7, 112.6, 87.6, 87.1, 86.8, 86.7, 81.4, 81.3, 81.0, 75.3, 75.1, 74.9, 53.1, 53.1, 45.0, 46.8, 46.6, 40.8, 40.6, 40.0, 39.8, 35.0, 34.9, 34.7, 34.1, 34.0, 31.3, 31.3, 31.2, 31.1, 25.6, 25.4, 25.2, 23.1, 23.0, 22.8, 22.1, 21.9, 21.8, 20.8, 20.4, 20.3, 16.1, 15.8, 15.6, 15.4. **HRMS** (ESI) m/z : [M+H]⁺ Calcd for C₃₄H₃₉N₂O₆: 571.2803; Found: 571.2786.

Methyl 1-oxo-2-((2-oxo-1-phenyl-2-(((1R,4S)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)oxy)ethoxy)(pyridin-2-yl)amino)-2,3-dihydro-1H-indene-2-carboxylate (24)



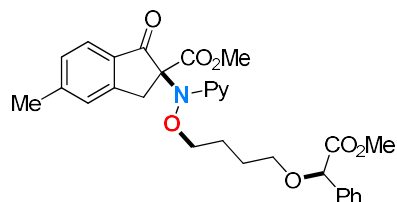
According to *GPI* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (44.5 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL DCM for 12 h. Purification by silica gel chromatography afforded the desired **24** and as a yellow oil in 71% yield (40.1 mg, d.r. = 1:1). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.01 (s, 1H), 7.79 – 7.19 (m, 11H), 6.81 (d, J = 35.0 Hz, 1H), 6.03 (d, J = 8.5 Hz, 1H), 4.98 – 4.69 (m, 1H), 4.08 (t, J = 17.2 Hz, 1H), 3.69 (d, J = 52.9 Hz, 3H), 3.23 (dd, J = 44.8, 17.1 Hz, 1H), 2.39 – 2.09 (m, 1H), 1.92 – 1.04 (m, 6H), 0.81 (d, J = 11.5 Hz, 7H), 0.57 – 0.42 (m, 2H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 171.1, 167.6, 167.3, 160.8, 145.8, 145.6, 137.8, 137.7, 137.5, 136.2, 135.5, 135.1, 134.5, 128.8, 128.6, 128.4, 128.2, 128.1, 127.9, 127.5, 127.4, 127.4, 127.2, 127.2, 126.2, 125.9, 125.9, 125.0, 124.8, 118.6, 118.1, 113.9, 112.9, 112.8, 87.5, 87.2, 86.9, 81.3, 81.0, 80.6, 53.2, 53.2, 53.1, 49.0, 48.8, 48.8, 48.7, 47.8, 47.7, 44.8, 44.8, 44.6, 36.1, 36.1, 36.0, 34.8,

34.8, 34.7, 34.7, 27.8, 27.7, 27.6, 27.6, 26.9, 26.8, 26.7, 19.6, 19.5, 18.8, 18.7, 18.7, 13.3, 13.2, 13.0, 12.9. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{34}H_{37}N_2O_6$: 569.2652; Found: 569.2639.



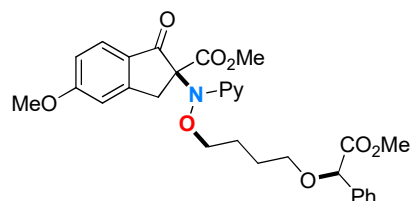
General procedure (GP2): To a 10 mL Schlenk flask equipped with a magnetic stir bar was added **1e** (0.1 mmol), **2a** (0.1 mmol), **3** (0.15 mmol), dry THF (1.0 mL). The resulting mixture was stirred at a distance of ~3 cm from a 24 w blue LED at room temperature for 12 h. The solvent was removed by vacuum and the crude product was purified by flash chromatography on silica gel silica: 200~300; eluant: petroleum ether/ethyl acetate (20:1~5:1) to provide pure product **5** as a yellow oil in 99% yield (51.5 mg). **¹H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.11 (s, 1H), 7.78 (t, J = 9.7 Hz, 1H), 7.59 (d, J = 8.3 Hz, 2H), 7.44 – 7.31 (m, 7H), 7.02 (d, J = 8.3 Hz, 1H), 6.87 (s, 1H), 4.79 (s, 1H), 4.33 – 4.07 (m, 3H), 3.73 (d, J = 33.7 Hz, 6H), 3.39 (d, J = 35.4 Hz, 2H), 3.07 (dd, J = 17.3, 9.0 Hz, 1H), 1.58 (s, 4H). **¹³C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 195.7, 171.3, 167.5, 161.4, 153.3, 146.6, 137.8, 136.5, 135.6, 134.3, 128.6, 128.5, 127.6, 127.1, 126.3, 124.9, 117.8, 111.0, 111.0, 81.0, 79.8, 79.8, 75.5, 69.4, 69.3, 53.2, 52.1, 34.9, 26.0, 24.8, 24.7. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{29}H_{31}N_2O_7$: 519.2126; Found: 519.2090.

Methyl 2-((4-(2-methoxy-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-5-methyl-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (25**)**



According to GP2 with β -keto ester (20.4 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **25** and as a colorless oil in 88% yield (46.9 mg). **¹H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.11 (s, 1H), 7.63 (dt, J = 27.4, 8.5 Hz, 2H), 7.37 (dd, J = 25.1, 6.7 Hz, 5H), 7.17 (dd, J = 19.1, 11.6 Hz, 2H), 7.02 (d, J = 8.4 Hz, 1H), 6.86 (s, 1H), 4.79 (s, 1H), 4.24 (d, J = 55.1 Hz, 2H), 4.05 (d, J = 14.1 Hz, 1H), 3.75 (s, 3H), 3.69 (s, 3H), 3.39 (d, J = 34.8 Hz, 2H), 3.02 (dd, J = 17.3, 8.7 Hz, 1H), 2.42 (s, 3H), 1.57 (s, 4H). **¹³C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 195.1, 171.3, 167.6, 161.5, 153.8, 147.0, 146.6, 137.8, 136.5, 132.0, 128.9, 128.6, 128.5, 127.0, 126.6, 124.8, 117.7, 111.0, 111.0, 80.9, 80.0, 80.0, 75.5, 69.4, 69.3, 53.2, 52.1, 34.7, 26.0, 24.7, 24.7, 22.1. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{30}H_{33}N_2O_7$: 533.2287; Found: 533.2260.

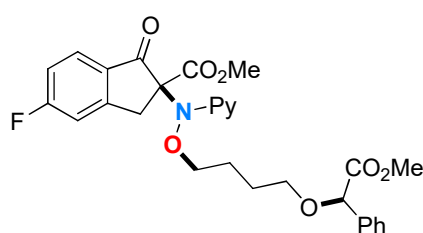
Methyl 5-methoxy-2-((4-(2-methoxy-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (26**)**



According to GP2 with β -keto ester (22.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **26** and as a colorless

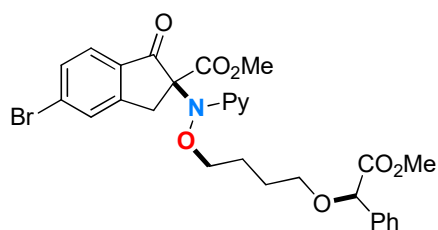
oil in 94% yield (51.6 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.12 (s, 1H), 7.75 – 7.67 (m, 1H), 7.60 (t, *J* = 8.1 Hz, 1H), 7.37 (dd, *J* = 29.4, 6.7 Hz, 5H), 7.03 (d, *J* = 8.1 Hz, 1H), 6.87 (d, *J* = 8.5 Hz, 3H), 4.79 (s, 1H), 4.25 (d, *J* = 62.4 Hz, 2H), 4.06 (d, *J* = 17.3 Hz, 1H), 3.86 (s, 3H), 3.75 (s, 3H), 3.69 (s, 3H), 3.39 (d, *J* = 35.8 Hz, 2H), 3.02 (dd, *J* = 17.4, 8.7 Hz, 1H), 1.58 (s, 4H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 193.8, 171.3, 167.8, 166.0, 161.6, 156.4, 146.6, 137.7, 136.5, 136.5, 128.5, 128.5, 127.5, 127.0, 126.7, 117.7, 115.9, 111.1, 111.0, 109.3, 80.9, 80.0, 80.0, 75.5, 69.4, 69.3, 55.6, 53.1, 52.1, 34.9, 26.0, 24.7, 24.7. **HRMS** (ESI) *m/z*: [M+H]⁺ Calcd for C₃₀H₃₃N₂O₈: 549.2231; Found: 549.2213.

methyl 5-fluoro-2-((4-(2-methoxy-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (27)



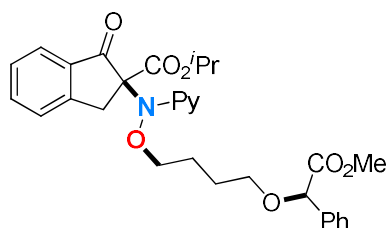
According to *GP2* with β-keto ester (20.8 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **27** and as a colorless oil in 88% yield (47.2 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.10 (s, 1H), 7.79 (dt, *J* = 14.3, 6.8 Hz, 1H), 7.63 – 7.57 (m, 1H), 7.42 – 7.31 (m, 5H), 7.05 (dd, *J* = 18.1, 8.5 Hz, 3H), 6.88 (s, 1H), 4.79 (s, 1H), 4.24 (d, *J* = 38.5 Hz, 2H), 4.09 (d, *J* = 17.3 Hz, 1H), 3.77 (s, 3H), 3.69 (s, 3H), 3.40 (d, *J* = 36.8 Hz, 2H), 3.05 (dd, *J* = 17.6, 8.3 Hz, 1H), 1.58 (s, 4H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 194.5, 171.3, 167.1, 161.2, 154.7, 146.6, 137.9, 136.5, 133.2, 131.3, 131.1, 129.6, 128.6, 128.6, 127.1, 126.1, 126.1, 118.0, 111.1, 111.0, 81.0, 79.9, 79.8, 75.6, 69.4, 69.3, 53.3, 52.2, 34.5, 26.0, 24.8, 24.7. **HRMS** (ESI) *m/z*: [M+H]⁺ Calcd for C₂₉H₃₀FN₂O₇: 537.2032; Found: 537.2018.

Methyl 5-bromo-2-((4-(2-methoxy-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (28)



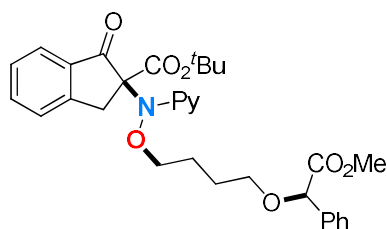
According to *GP2* with β-keto ester (26.8 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **28** and as a colorless oil in 89% yield (53.0 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.08 (s, 1H), 7.67 – 7.58 (m, 3H), 7.50 (d, *J* = 8.3 Hz, 1H), 7.42 – 7.32 (m, 5H), 7.02 (d, *J* = 8.3 Hz, 1H), 6.87 (s, 1H), 4.80 (s, 1H), 4.23 (d, *J* = 27.4 Hz, 2H), 4.07 (d, *J* = 17.3 Hz, 1H), 3.76 (s, 3H), 3.69 (s, 3H), 3.41 (d, *J* = 37.0 Hz, 2H), 3.04 (dd, *J* = 17.6, 9.4 Hz, 1H), 1.58 (s, 4H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 194.5, 171.3, 167.1, 161.2, 154.7, 146.6, 137.9, 136.5, 133.2, 131.3, 131.1, 129.6, 128.6, 128.6, 127.1, 126.1, 126.1, 118.0, 111.1, 111.0, 81.0, 79.9, 79.8, 75.6, 69.4, 69.3, 53.3, 52.2, 34.5, 26.0, 24.8, 24.7. **HRMS** (ESI) *m/z*: [M+H]⁺ Calcd for C₂₉H₃₀BrN₂O₇: 597.1231; Found: 597.1204.

Isopropyl 2-((4-(2-methoxy-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (29)



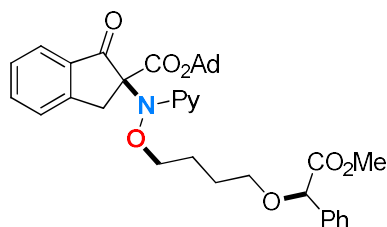
According to *GP2* with β -keto ester (21.8 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **29** and as a colorless oil in 94% yield (51.3 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.06 (s, 1H), 7.78 (t, J = 9.0 Hz, 1H), 7.57 (d, J = 8.3 Hz, 2H), 7.43 – 7.31 (m, 7H), 7.00 (d, J = 8.4 Hz, 1H), 6.83 (s, 1H), 5.12 – 5.00 (m, 1H), 4.80 (s, 1H), 4.25 (d, J = 32.2 Hz, 2H), 4.09 (d, J = 17.3 Hz, 1H), 3.69 (s, 3H), 3.40 (d, J = 37.5 Hz, 2H), 3.05 (dd, J = 17.3, 7.9 Hz, 1H), 1.58 (s, 4H), 1.26 (d, J = 5.4 Hz, 3H), 1.13 (d, J = 5.6 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.4, 171.3, 166.3, 161.3, 153.3, 146.3, 137.6, 136.5, 135.2, 134.6, 128.5, 128.5, 127.4, 127.1, 126.1, 124.8, 117.6, 110.9, 80.9, 79.9, 75.4, 69.6, 69.4, 69.4, 52.1, 35.2, 26.0, 24.8, 24.7, 21.5, 21.1. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₃₁H₃₅N₂O₇: 547.2439; Found: 547.2417.

Tert-butyl 2-((4-(2-methoxy-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (30)



According to *GP2* with β -keto ester (23.2 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **30** and as a colorless oil in 91% yield (51.0 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.05 (s, 1H), 7.76 (t, J = 8.7 Hz, 1H), 7.59 – 7.52 (m, 2H), 7.41 – 7.29 (m, 7H), 6.97 (d, J = 8.4 Hz, 1H), 6.82 (d, J = 6.7 Hz, 1H), 4.79 (s, 1H), 4.22 (d, J = 34.2 Hz, 2H), 4.03 (d, J = 17.3 Hz, 1H), 3.67 (s, 3H), 3.40 (d, J = 34.6 Hz, 2H), 3.00 (dd, J = 17.3, 7.3 Hz, 1H), 1.58 (s, 4H), 1.38 (s, 9H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.5, 171.3, 165.5, 161.3, 153.2, 146.4, 137.6, 136.5, 135.1, 134.8, 128.5, 127.3, 127.1, 126.0, 124.7, 117.4, 110.7, 81.9, 80.9, 80.4, 75.4, 69.4, 69.4, 52.1, 35.4, 27.6, 26.1, 24.8, 24.7. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₃₂H₃₇N₂O₇: 561.2595; Found: 561.2582.

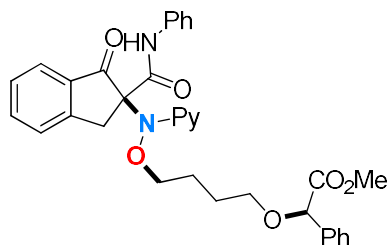
Adamantan-1-yl 2-((4-(2-methoxy-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (31)



According to *GP2* with β -keto ester (31.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **31** and as a colorless oil in 88% yield (56.2 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.06 (s, 1H), 7.78 (t, J = 8.7 Hz, 1H), 7.56 (q, J = 6.8 Hz, 2H), 7.43 – 7.31 (m, 7H), 6.99 (d, J = 8.2 Hz, 1H), 6.82 (s, 1H), 4.81 (s, 1H), 4.25 (d, J = 21.9 Hz, 2H), 4.03 (d, J = 17.2 Hz, 1H), 3.69 (s, 3H), 3.42 (d, J = 32.5 Hz, 2H), 3.01 (dd, J = 17.4, 6.8 Hz, 1H), 2.08 (d, J = 13.8 Hz, 9H), 1.60 (s, 10H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.5, 171.4, 165.2, 161.2, 153.2, 146.4,

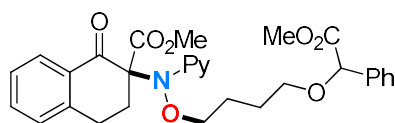
137.6, 136.5, 135.0, 134.8, 128.5, 127.3, 127.1, 126.0, 124.7, 117.4, 110.7, 82.0, 81.0, 80.6, 75.4, 69.5, 69.4, 52.1, 40.8, 36.1, 35.4, 30.8, 26.1, 24.8, 24.7. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{38}H_{43}N_2O_7$: 639.3065; Found: 639.3023.

Methyl 2-(4-(((1-oxo-2-(phenylcarbamoyl)-2,3-dihydro-1H-inden-2-yl)(pyridin-2-yl)amino)oxy)butoxy)-2-phenylacetate (32)



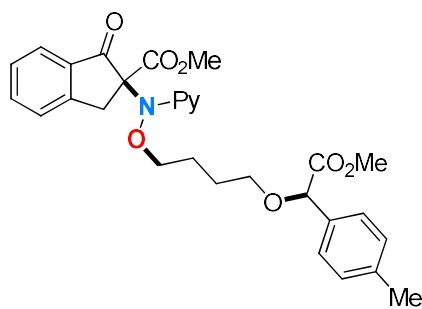
According to *GP2* with β -keto ester (25.1 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **32** and as a brown oil in 97% yield (56.2 mg). **1H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.70 (s, 1H), 8.11 (s, 1H), 7.76 (t, J = 8.2 Hz, 1H), 7.58 (dd, J = 43.4, 8.0 Hz, 4H), 7.43 – 7.26 (m, 9H), 7.10 – 7.03 (m, 2H), 6.88 (s, 1H), 4.79 (s, 1H), 4.40 – 4.12 (m, 3H), 3.69 (d, J = 4.1 Hz, 3H), 3.41 (d, J = 38.7 Hz, 2H), 2.91 (d, J = 22.7 Hz, 1H), 1.64 (s, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 197.3, 171.3, 163.9, 161.1, 153.8, 147.3, 138.1, 138.0, 136.5, 135.5, 134.3, 128.8, 128.6, 128.5, 127.5, 127.1, 126.3, 124.8, 124.0, 119.8, 118.4, 111.0, 81.3, 80.9, 75.6, 69.3, 69.3, 52.2, 32.8, 26.0, 24.8, 24.8. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{34}H_{34}N_3O_7$: 580.2442; Found: 580.2425.

Methyl 2-((4-(2-methoxy-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-1,2,3,4-tetrahydronaphthalene-2-carboxylate (33)



According to *GP2* with β -keto ester (20.4 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (26.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **33** and as a colorless oil in 92% yield (49.0 mg). **1H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.07 (s, 1H), 7.99 (d, J = 8.0 Hz, 1H), 7.62 – 7.56 (m, 1H), 7.48 – 7.40 (m, 3H), 7.35 – 7.21 (m, 5H), 7.02 (d, J = 8.3 Hz, 1H), 6.79 (t, J = 5.8 Hz, 1H), 4.82 (s, 1H), 3.96 (d, J = 8.1 Hz, 1H), 3.71 (d, J = 10.6 Hz, 7H), 3.42 (dd, J = 38.0, 6.2 Hz, 2H), 3.20 – 2.98 (m, 3H), 2.78 (d, J = 15.3 Hz, 1H), 1.64 (s, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$, 300 K): δ (ppm) = 190.8, 171.3, 169.4, 146.6, 142.9, 137.9, 136.5, 133.2, 133.0, 128.6, 128.6, 128.5, 128.1, 127.1, 126.6, 116.6, 109.0, 81.0, 77.6, 75.1, 69.4, 52.5, 52.2, 26.2, 26.0, 24.7. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for $C_{30}H_{33}N_2O_7$: 533.2282; Found: 533.2253.

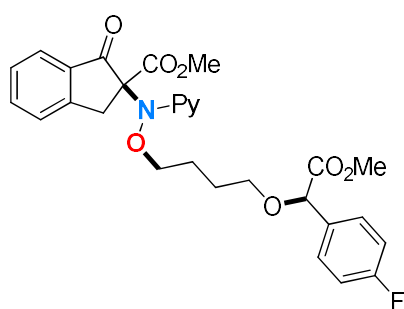
Methyl 2-((4-(2-methoxy-2-oxo-1-(p-tolyl)ethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (34)



According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (28.5 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **34** and as a colorless oil in 87% yield (46.3 mg). **1H NMR** (400 MHz, $CDCl_3$, 300 K): δ (ppm) = 8.10 (s, 1H), 7.78 (t, J = 9.0 Hz, 1H), 7.63 – 7.57 (m, 2H), 7.44 – 7.32 (m, 2H), 7.28 (d, J = 8.3

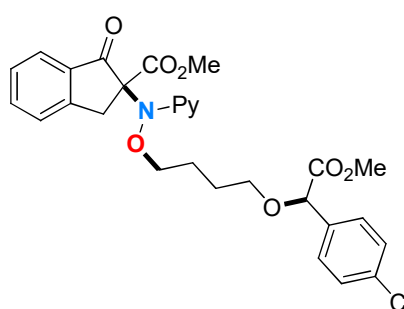
Hz, 2H), 7.15 (d, $J = 6.3$ Hz, 2H), 7.02 (d, $J = 8.3$ Hz, 1H), 6.87 (t, $J = 5.7$ Hz, 1H), 4.75 (s, 1H), 4.34 – 4.06 (m, 3H), 3.77 (s, 3H), 3.68 (s, 3H), 3.45 – 3.27 (m, 2H), 3.12 – 3.01 (m, 1H), 2.33 (s, 3H), 1.56 (s, 4H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 195.7, 171.5, 167.5, 161.4, 153.3, 146.6, 138.4, 137.8, 135.6, 134.3, 133.6, 129.2, 127.6, 127.1, 126.3, 124.9, 117.8, 111.0, 111.0, 80.8, 79.9, 79.8, 75.6, 69.2, 69.2, 53.2, 52.1, 34.9, 26.0, 24.8, 24.7, 21.14. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{30}\text{H}_{33}\text{N}_2\text{O}_7$: 533.2282; Found: 533.2256.

Methyl 2-((4-(1-(4-fluorophenyl)-2-methoxy-2-oxoethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (35)



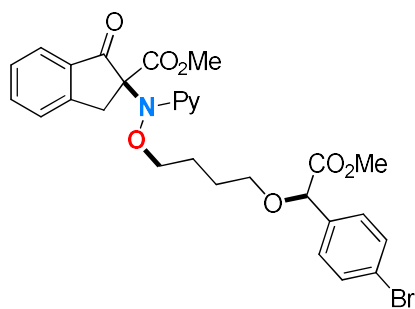
According to GP2 with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (29.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **35** and as a colorless oil in 94% yield (50.4 mg). ^1H NMR (400 MHz, CDCl_3 , 300 K): δ (ppm) = 8.11 (s, 1H), 7.81 – 7.74 (m, 1H), 7.60 (t, $J = 8.0$ Hz, 2H), 7.40 (dd, $J = 21.7, 7.3$ Hz, 4H), 7.02 (d, $J = 7.9$ Hz, 3H), 6.87 (t, $J = 6.5$ Hz, 1H), 4.76 (s, 1H), 4.35 – 4.07 (m, 3H), 3.77 (s, 3H), 3.69 (s, 3H), 3.48 – 3.27 (m, 2H), 3.07 (dd, $J = 17.5, 8.1$ Hz, 1H), 1.58 (s, 4H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 195.7, 171.2, 167.4, 164.0, 161.6, 161.4, 153.4, 146.7, 137.8, 135.6, 134.3, 132.4, 128.9, 128.8, 127.6, 126.3, 124.9, 117.8, 115.6, 115.4, 111.0, 110.9, 80.2, 79.9, 75.5, 69.4, 69.4, 53.2, 52.2, 34.9, 26.0, 24.7, 24.7. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{29}\text{H}_{30}\text{FN}_2\text{O}_7$: 537.2032; Found: 537.2014.

Methyl 2-((4-(1-(4-chlorophenyl)-2-methoxy-2-oxoethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (36)



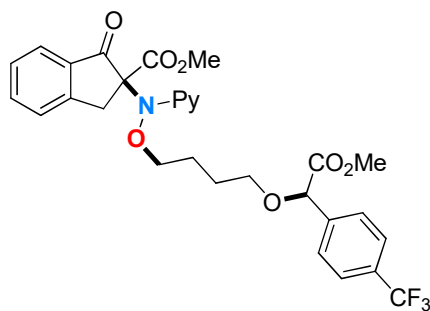
According to GP2 with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (29.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **36** and as a colorless oil in 86% yield (47.5 mg). ^1H NMR (400 MHz, CDCl_3 , 300 K): δ (ppm) = 8.10 (s, 1H), 7.77 (dd, $J = 14.5, 6.5$ Hz, 1H), 7.60 (t, $J = 8.0$ Hz, 2H), 7.44 – 7.29 (m, 6H), 7.02 (d, $J = 11.0$ Hz, 1H), 6.87 (s, 1H), 4.75 (s, 1H), 4.34 – 4.07 (m, 3H), 3.77 (s, 3H), 3.69 (s, 3H), 3.38 (d, $J = 44.2$ Hz, 2H), 3.06 (dd, $J = 17.6, 9.3$ Hz, 1H), 1.57 (s, 4H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 195.7, 171.0, 167.4, 161.4, 153.4, 146.7, 137.8, 135.6, 135.1, 134.5, 134.3, 128.7, 128.4, 127.6, 126.3, 124.9, 117.9, 111.0, 80.2, 79.9, 75.5, 69.5, 69.4, 53.2, 52.3, 34.9, 26.0, 24.7, 24.7. HRMS (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{29}\text{H}_{30}\text{ClN}_2\text{O}_7$: 553.1736; Found: 553.1717.

Methyl 2-((4-(1-(4-bromophenyl)-2-methoxy-2-oxoethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (37)



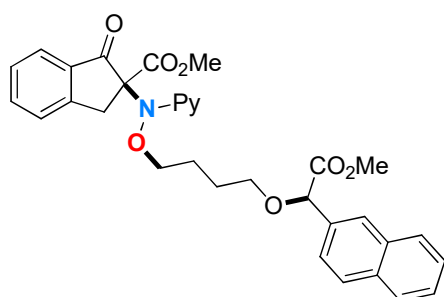
According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (38.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **37** and as a colorless oil in 88% yield (52.5 mg). ¹H NMR (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.12 (s, 1H), 7.77 (dd, J = 15.3, 7.7 Hz, 1H), 7.60 (t, J = 8.0 Hz, 2H), 7.50 – 7.32 (m, 4H), 7.28 (d, J = 7.8 Hz, 2H), 7.02 (d, J = 8.2 Hz, 1H), 6.87 (t, J = 6.4 Hz, 1H), 4.74 (s, 1H), 4.35 – 4.07 (m, 3H), 3.77 (s, 3H), 3.69 (s, 3H), 3.39 (dd, J = 46.5, 6.4 Hz, 2H), 3.06 (dd, J = 17.3, 10.2 Hz, 1H), 1.57 (s, 4H). ¹³C NMR (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.7, 170.9, 167.4, 161.4, 153.4, 146.7, 137.8, 135.6, 134.3, 131.7, 128.7, 127.6, 126.3, 124.9, 124.9, 122.6, 117.9, 111.0, 80.3, 79.9, 79.8, 75.5, 69.5, 69.4, 53.2, 52.3, 26.0, 24.7, 24.6. HRMS (ESI) m/z : [M+H]⁺ Calcd for C₂₉H₃₀BrN₂O₇: 597.1231; Found: 597.1222.

Methyl 2-((4-(2-methoxy-2-oxo-1-(4-(trifluoromethyl)phenyl)ethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (38)



According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (36.6 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **38** and as a colorless oil in 75% yield (44.0 mg). ¹H NMR (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.12 (s, 1H), 7.77 (dd, J = 17.5, 7.8 Hz, 1H), 7.63 – 7.52 (m, 6H), 7.45 – 7.32 (m, 2H), 7.02 (d, J = 8.4 Hz, 1H), 6.87 (t, J = 3.7 Hz, 1H), 4.85 (s, 1H), 4.35 – 4.08 (m, 3H), 3.77 (s, 3H), 3.70 (d, J = 2.2 Hz, 3H), 3.52 – 3.45 (m, 1H), 3.34 (s, 1H), 3.12 – 2.99 (m, 1H), 1.59 (s, 4H). ¹³C NMR (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.7, 170.7, 167.4, 161.4, 153.4, 146.7, 140.5, 137.8, 135.6, 134.3, 127.6, 127.3, 126.3, 125.5, 125.5, 124.9, 117.9, 110.9, 80.4, 79.9, 75.5, 69.8, 69.7, 53.2, 52.4, 34.9, 26.0, 24.7, 24.6. HRMS (ESI) m/z : [M+H]⁺ Calcd for C₃₀H₃₀F₃N₂O₇: 587.2000; Found: 587.1982.

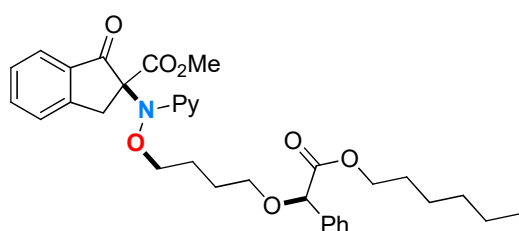
Methyl 2-((4-(2-methoxy-1-(naphthalen-2-yl)-2-oxoethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (39)



According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (33.9 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **39** and as a colorless oil in 88% yield (50.0 mg). ¹H NMR (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.09 (s, 1H), 7.82 (q, J = 9.4, 7.2 Hz, 5H), 7.59 – 7.29 (m, 7H), 7.02 (d, J = 8.4

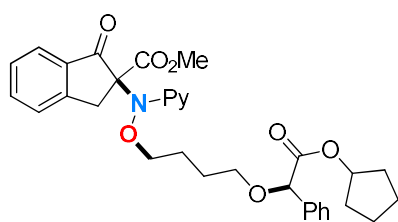
Hz, 1H), 6.84 (d, J = 6.6 Hz, 1H), 4.95 (s, 1H), 4.33 – 4.04 (m, 3H), 3.76 (s, 3H), 3.68 (s, 3H), 3.52 – 3.33 (m, 2H), 3.05 (t, J = 18.0 Hz, 1H), 1.60 (s, 4H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 195.6, 171.2, 167.4, 161.4, 153.3, 146.6, 137.8, 135.5, 134.3, 133.9, 133.3, 133.0, 128.4, 128.0, 127.6, 127.5, 126.6, 126.3, 126.3, 126.2, 124.9, 124.9, 124.4, 117.8, 111.0, 110.9, 81.1, 79.8, 79.8, 75.6, 69.4, 69.3, 53.2, 52.2, 34.9, 26.0, 24.8, 24.7. **HRMS** (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{33}\text{H}_{33}\text{N}_2\text{O}_7$: 569.2282; Found: 569.2263.

Methyl 2-((4-(2-(hexyloxy)-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (40)



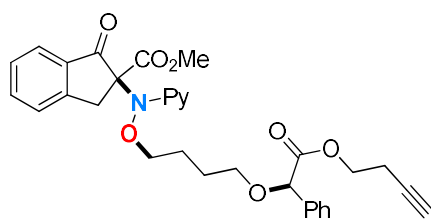
According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (36.9 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **40** and as a colorless oil in 89% yield (52.4 mg). ^1H NMR (400 MHz, CDCl_3 , 300 K): δ (ppm) = 8.11 (s, 1H), 7.78 (t, J = 9.5 Hz, 1H), 7.59 (t, J = 7.6 Hz, 2H), 7.43 – 7.30 (m, 7H), 7.03 (d, J = 8.3 Hz, 1H), 6.87 (t, J = 5.7 Hz, 1H), 4.77 (s, 1H), 4.33 – 4.06 (m, 5H), 3.76 (s, 3H), 3.40 (d, J = 39.3 Hz, 2H), 3.13 – 3.01 (m, 1H), 1.59 (d, J = 19.8 Hz, 6H), 1.20 (s, 6H), 0.83 (d, J = 7.8 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 195.7, 171.0, 167.5, 161.4, 153.3, 146.6, 137.8, 136.7, 135.5, 134.3, 128.4, 127.6, 127.0, 127.0, 126.3, 124.9, 117.8, 111.0, 111.0, 81.0, 79.8, 75.6, 69.3, 69.3, 65.1, 53.2, 34.9, 31.2, 28.4, 26.0, 25.3, 24.8, 24.7, 22.4, 13.9. **HRMS** (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{34}\text{H}_{41}\text{N}_2\text{O}_7$: 589.2908; Found: 589.2878.

Methyl 2-((4-(2-(cyclopentyloxy)-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (41)



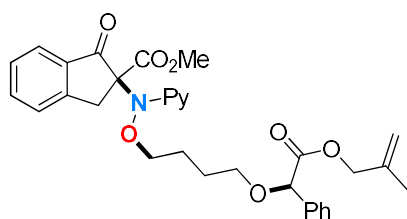
According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (34.5 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **41** and as a colorless oil in 81% yield (46.4 mg). ^1H NMR (400 MHz, CDCl_3 , 300 K): δ (ppm) = 8.11 (s, 1H), 7.78 (t, J = 9.3 Hz, 1H), 7.58 (d, J = 8.1 Hz, 2H), 7.36 (dd, J = 25.1, 9.1 Hz, 7H), 7.03 (d, J = 8.4 Hz, 1H), 6.86 (t, J = 6.4 Hz, 1H), 5.17 (s, 1H), 4.73 (s, 1H), 4.35 – 4.07 (m, 3H), 3.76 (s, 3H), 3.40 (d, J = 35.9 Hz, 2H), 3.07 (dd, J = 17.4, 8.8 Hz, 1H), 1.82 – 1.48 (m, 12H). ^{13}C NMR (100 MHz, CDCl_3 , 300 K): δ (ppm) = 195.7, 170.6, 167.5, 161.4, 153.3, 146.6, 137.8, 136.8, 135.5, 134.3, 128.4, 128.3, 127.5, 127.0, 126.9, 126.2, 124.9, 117.8, 111.0, 111.0, 81.0, 79.8, 77.9, 75.6, 69.3, 69.2, 53.2, 34.9, 32.4, 32.4, 26.0, 24.8, 24.8, 23.5, 23.4. **HRMS** (ESI) m/z : $[\text{M}+\text{H}]^+$ Calcd for $\text{C}_{33}\text{H}_{37}\text{N}_2\text{O}_7$: 573.2595; Found: 573.2566.

Methyl 2-((4-(2-(but-3-yn-1-yloxy)-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (42)



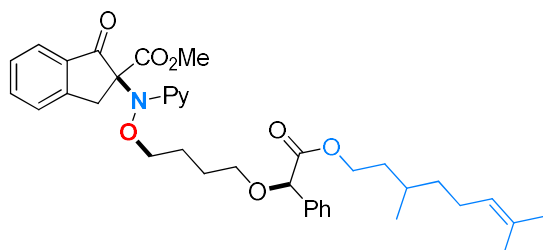
According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (32.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **42** and as a colorless oil in 97% yield (54.0 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.11 (s, 1H), 7.83 – 7.75 (m, 1H), 7.60 (t, J = 7.5 Hz, 2H), 7.37 (dd, J = 32.8, 6.6 Hz, 7H), 7.02 (d, J = 8.2 Hz, 1H), 6.87 (t, J = 6.4 Hz, 1H), 4.80 (s, 1H), 4.33 – 4.07 (m, 5H), 3.76 (s, 3H), 3.41 (d, J = 36.5 Hz, 2H), 3.07 (dd, J = 17.3, 8.9 Hz, 1H), 2.45 (s, 2H), 1.92 (d, J = 5.6 Hz, 1H), 1.59 (s, 4H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.7, 170.7, 167.5, 161.4, 153.4, 146.7, 137.8, 136.4, 135.6, 134.3, 128.6, 128.5, 127.6, 127.1, 126.3, 124.9, 117.8, 111.0, 80.9, 79.8, 79.5, 75.6, 70.0, 69.4, 69.3, 62.6, 53.2, 34.9, 26.0, 24.8, 24.7, 18.8. **HRMS** (ESI) m/z : [M+H]⁺ Calcd for C₃₂H₃₃N₂O₇: 557.2282; Found: 557.2260.

Methyl 2-((4-(2-((2-methylallyl)oxy)-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (43)



According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (32.4 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **43** and as a colorless oil in 94% yield (52.5 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.11 (d, J = 4.8 Hz, 1H), 7.78 (t, J = 9.3 Hz, 1H), 7.59 (t, J = 7.4 Hz, 2H), 7.37 (dd, J = 36.8, 7.4 Hz, 7H), 7.02 (d, J = 8.3 Hz, 1H), 6.86 (t, J = 4.2 Hz, 1H), 4.81 (s, 3H), 4.50 (t, J = 15.9 Hz, 2H), 4.34 – 4.06 (m, 3H), 3.77 (s, 3H), 3.41 (d, J = 38.4 Hz, 2H), 3.07 (dd, J = 17.5, 9.1 Hz, 1H), 1.60 (s, 7H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.7, 170.5, 167.5, 161.4, 153.4, 146.6, 139.4, 137.8, 136.6, 135.6, 134.4, 128.6, 128.5, 127.6, 127.1, 126.3, 124.9, 117.8, 113.0, 111.0, 81.00, 79.8, 75.6, 69.4, 69.3, 68.0, 53.2, 34.9, 26.0, 24.8, 24.7, 19.2. **HRMS** (ESI) m/z : [M+H]⁺ Calcd for C₃₂H₃₅N₂O₇: 559.2439; Found: 559.2412.

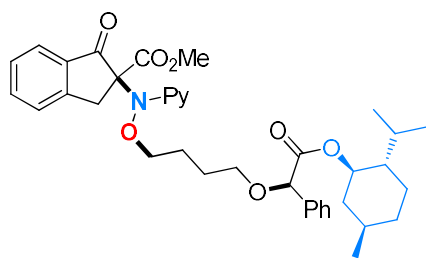
Methyl 2-((4-(2-((3,7-dimethyloct-6-en-1-yl)oxy)-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (44)



According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (45.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **44** and as a colorless oil in 92% yield (59.0 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.10 (s, 1H), 7.78 (t, J = 9.8 Hz, 1H), 7.58 (d, J = 8.0 Hz, 2H), 7.44 – 7.30 (m, 7H), 7.02 (d, J = 8.3 Hz, 1H), 6.87 (t, J = 6.1 Hz, 1H), 5.04 (s, 1H), 4.76 (s, 1H), 4.37 – 4.05 (m, 5H), 3.76 (s, 3H), 3.39 (d, J = 42.0 Hz, 2H), 3.07 (dd, J = 17.3, 9.1 Hz,

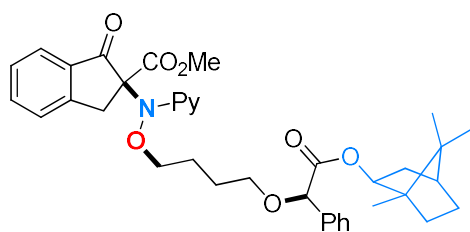
1H), 1.97 – 1.81 (m, 2H), 1.67 (s, 3H), 1.57 (s, 7H), 1.42 – 1.05 (m, 5H), 0.81 (dd, $J = 7.3, 4.2$ Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.7, 170.9, 167.5, 161.4, 153.3, 146.6, 137.8, 136.7, 135.5, 134.3, 131.2, 128.4, 127.6, 127.0, 126.3, 124.9, 124.5, 117.8, 111.0, 81.0, 79.8, 69.3, 69.3, 63.6, 53.2, 36.8, 36.8, 35.3, 34.9, 29.3, 29.3, 26.0, 25.6, 25.3, 24.8, 24.7, 19.2, 19.1, 17.6. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₃₈H₄₇N₂O₇: 643.3378; Found: 643.3354.

Methyl 2-((4-(2-(((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl)oxy)-2-oxo-1-phenylethoxy)butoxy)(pyridin-2-yl)amino)-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (45)



According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (45.1 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **45** and as a colorless oil in 98% yield (63.0 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.10 (s, 1H), 7.78 (t, $J = 9.3$ Hz, 1H), 7.59 (t, $J = 7.1$ Hz, 2H), 7.44 – 7.29 (m, 7H), 7.03 (d, $J = 8.3$ Hz, 1H), 6.87 (d, $J = 6.4$ Hz, 1H), 4.78 – 4.56 (m, 2H), 4.36 – 4.05 (m, 3H), 3.77 (s, 3H), 3.40 (d, $J = 35.3$ Hz, 2H), 3.07 (dd, $J = 17.3, 8.7$ Hz, 1H), 1.80 – 1.54 (m, 8H), 1.48 – 1.13 (m, 3H), 1.05 – 0.80 (m, 7H), 0.64 (dd, $J = 23.1, 4.1$ Hz, 3H), 0.43 – 0.40 (m, 1H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 170.5, 167.5, 161.5, 153.4, 146.6, 137.8, 135.6, 134.4, 128.4, 128.4, 127.6, 127.2, 126.9, 126.9, 126.3, 124.9, 117.8, 111.0, 81.2, 79.9, 75.6, 75.1, 75.0, 69.3, 69.2, 53.2, 47.1, 46.9, 40.8, 40.2, 34.9, 34.1, 31.3, 31.3, 26.1, 26.1, 25.4, 24.9, 23.3, 22.9, 22.0, 21.9, 20.6, 20.5, 16.1, 15.6. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₃₈H₄₇N₂O₇: 643.3378; Found: 643.3354.

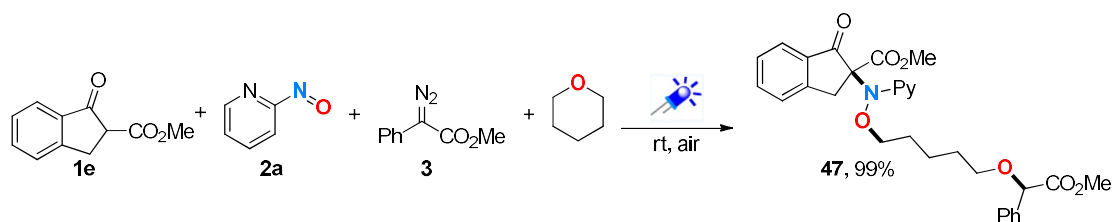
Methyl 1-oxo-2-((4-(2-oxo-1-phenyl-2-(((4R)-1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl)oxy)ethoxy)butoxy)(pyridin-2-yl)amino)-2,3-dihydro-1H-indene-2-carboxylate (46)



According to *GP2* with β -keto ester (19.0 mg, 0.10 mmol, 1.0 equiv.), 2-nitrosopyridine (10.8 mg, 0.10 mmol, 1.0 equiv.), aryldiazoacetates (44.5 mg, 0.15 mmol, 1.5 equiv.) in 1.0 mL THF for 12 h. Purification by silica gel chromatography afforded the desired **46** and as a colorless oil in 94% yield (60.2 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.10 (s, 1H), 7.84 – 7.73 (m, 1H), 7.66 – 7.54 (m, 2H), 7.44 – 7.29 (m, 7H), 7.03 (d, $J = 8.2$ Hz, 1H), 6.86 (s, 1H), 4.94 – 4.82 (m, 1H), 4.79 (s, 1H), 4.35 – 4.06 (m, 3H), 3.77 (s, 3H), 3.42 (d, $J = 33.2$ Hz, 2H), 3.07 (dd, $J = 17.4, 8.9$ Hz, 1H), 2.28 (dd, $J = 26.9, 16.2$ Hz, 1H), 1.86 – 1.57 (m, 7H), 1.29 – 1.11 (m, 2H), 0.96 – 0.79 (m, 8H), 0.56 (s, 2H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.7, 171.2, 171.1, 167.5, 161.4, 153.4, 146.6, 137.8, 137.1, 136.9, 135.5, 134.4, 128.4, 128.3, 127.6, 127.1, 127.0, 126.9, 126.9, 126.3, 124.9, 117.8, 111.0, 81.2, 81.1, 80.7, 80.4, 79.8, 69.3, 53.2, 48.9, 48.7, 47.8, 44.8, 44.6, 36.4, 36.4, 34.9, 27.9, 27.7, 27.0, 26.8, 26.1, 24.8, 19.6, 18.7, 13.4, 13.1. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₃₈H₄₅N₂O₇: 641.3221; Found: 641.3182.

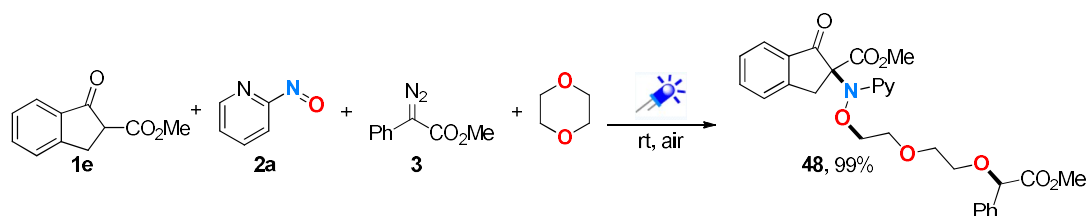
4. Follow-up chemistry

(a) Four-component trisubstituted hydroxylamines formation in tetrahydropyran



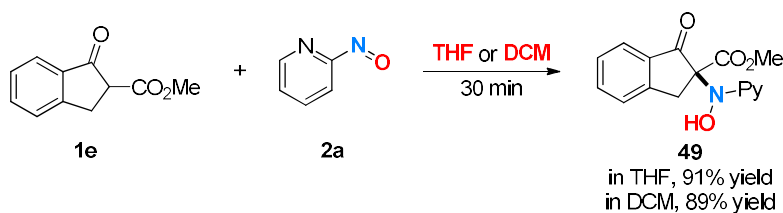
To a 10 mL Schlenk flask equipped with a magnetic stir bar was added **1e** (0.1 mmol), **2a** (0.1 mmol), **3** (0.15 mmol), tetrahydropyran (1.0 mL). The resulting mixture was stirred at a distance of ~3 cm from a 24 w blue LED at room temperature for 12 h. The solvent was removed by vacuum and the crude product was purified by flash chromatography on silica gel silica: 200~300; eluant: petroleum ether/ethyl acetate (20:1~5:1) to provide pure product **47** as a colorless oil in 99% yield (52.7 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.11 (s, 1H), 7.80 – 7.74 (m, 1H), 7.59 (p, J = 7.3, 6.7 Hz, 2H), 7.38 (dd, J = 31.9, 6.3 Hz, 7H), 7.02 (d, J = 8.2 Hz, 1H), 6.86 (t, J = 6.4 Hz, 1H), 4.82 (s, 1H), 4.33 – 4.08 (m, 3H), 3.77 (s, 3H), 3.69 (s, 3H), 3.38 – 3.26 (m, 2H), 3.08 (d, J = 17.2 Hz, 1H), 1.61 – 1.44 (m, 4H), 1.37 – 1.23 (m, 2H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.6, 171.3, 167.5, 161.4, 153.3, 146.6, 137.8, 136.6, 135.5, 134.3, 128.6, 128.5, 127.5, 127.0, 126.2, 124.9, 117.8, 111.0, 81.0, 79.7, 75.8, 69.5, 53.2, 52.1, 34.9, 29.2, 27.8, 22.4. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₃₀H₃₃N₂O₇: 533.2282; Found: 533.2274.

(b) Four-component trisubstituted hydroxylamines formation in 1,4-dioxane

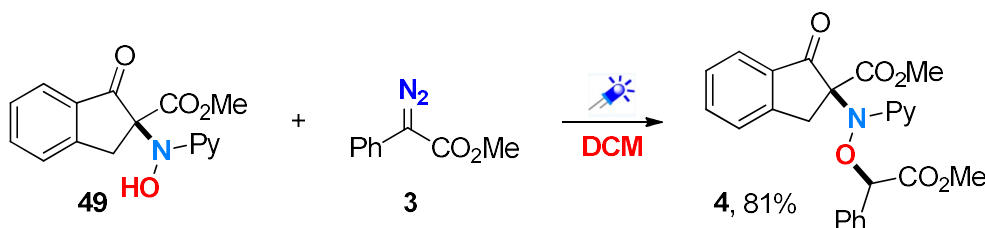


To a 10 mL Schlenk flask equipped with a magnetic stir bar was added **1e** (0.1 mmol), **2a** (0.1 mmol), **3** (0.15 mmol), 1,4-dioxane (1.0 mL). The resulting mixture was stirred at a distance of ~3 cm from a 24 w blue LED at room temperature for 12 h. The solvent was removed by vacuum and the crude product was purified by flash chromatography on silica gel silica: 200~300; eluant: petroleum ether/ethyl acetate (20:1~5:1) to provide pure product **48** as a colorless oil in 99% yield (53.0 mg). **¹H NMR** (400 MHz, CDCl₃, 300 K): δ (ppm) = 8.09 (s, 1H), 7.79 (d, J = 7.6 Hz, 1H), 7.62 – 7.50 (m, 2H), 7.44 – 7.29 (m, 8H), 6.85 (s, 1H), 4.98 (s, 1H), 4.38 (s, 2H), 4.09 (d, J = 17.0 Hz, 1H), 3.77 (s, 3H), 3.68 – 3.46 (m, 9H), 3.10 (d, J = 17.1 Hz, 1H). **¹³C NMR** (100 MHz, CDCl₃, 300 K): δ (ppm) = 195.8, 171.2, 167.4, 161.5, 153.4, 146.4, 138.0, 136.3, 135.6, 134.3, 128.6, 128.5, 127.5, 127.2, 126.3, 124.9, 117.9, 111.4, 81.2, 80.1, 75.6, 70.5, 70.4, 69.1, 69.1, 68.9, 68.8, 53.2, 52.1, 34.8. **HRMS** (ESI) m/z : $[M+H]^+$ Calcd for C₂₉H₃₁N₂O₈: 535.2075; Found: 535.2052.

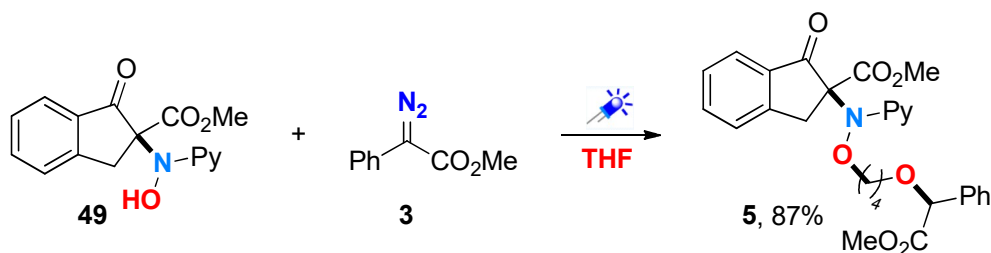
5. Mechanism studies



To a 10 mL Schlenk flask equipped with a magnetic stir bar was added **1e** (0.1 mmol) and **2a** (0.1 mmol) in THF or DCM (1.0 mL). The resulting mixture was stirred at room temperature for 30 min. The solvent was removed by vacuum and the crude product was purified by flash chromatography on silica gel silica: 200~300; eluant: petroleum ether/ethyl acetate (20:1~5:1) to provide pure product **49** as a yellow oil. ¹H NMR (400 MHz, CDCl₃, 300 K): δ (ppm) = 7.94 (s, 1H), 7.85 (d, *J* = 7.7 Hz, 1H), 7.62 (d, *J* = 7.3 Hz, 1H), 7.44 (dd, *J* = 19.6, 10.1 Hz, 3H), 7.17 (d, *J* = 7.2 Hz, 1H), 6.76 (t, *J* = 4.2 Hz, 1H), 4.09 (d, *J* = 16.5 Hz, 1H), 3.75 (s, 3H), 3.19 (d, *J* = 16.9 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃, 300 K): δ (ppm) = 196.6, 167.7, 160.5, 153.3, 145.6, 137.4, 135.6, 134.4, 127.6, 126.3, 125.1, 117.6, 112.5, 80.3, 53.3, 34.1. HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₆H₁₅N₂O₄: 299.1026; Found: 299.1013.



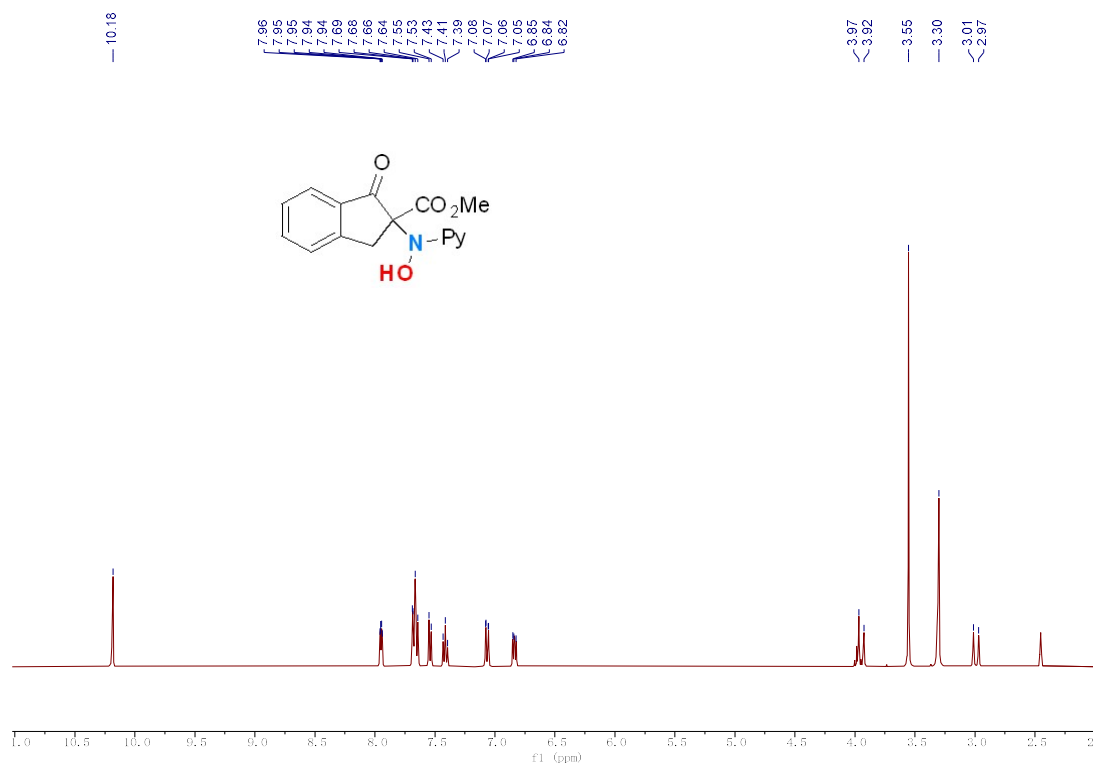
To a 10 mL Schlenk flask equipped with a magnetic stir bar was added **49** (0.1 mmol) and **3** (0.15 mmol) in DCM (1.0 mL). The resulting mixture was stirred at a distance of ~3 cm from a 24 w blue LED at room temperature for 12 h. The solvent was removed by vacuum and the crude product was purified by flash chromatography on silica gel silica: 200~300; eluant: petroleum ether/ethyl acetate (20:1~5:1) to provide pure product **4** as a yellow oil in 81% yield (36.2 mg).



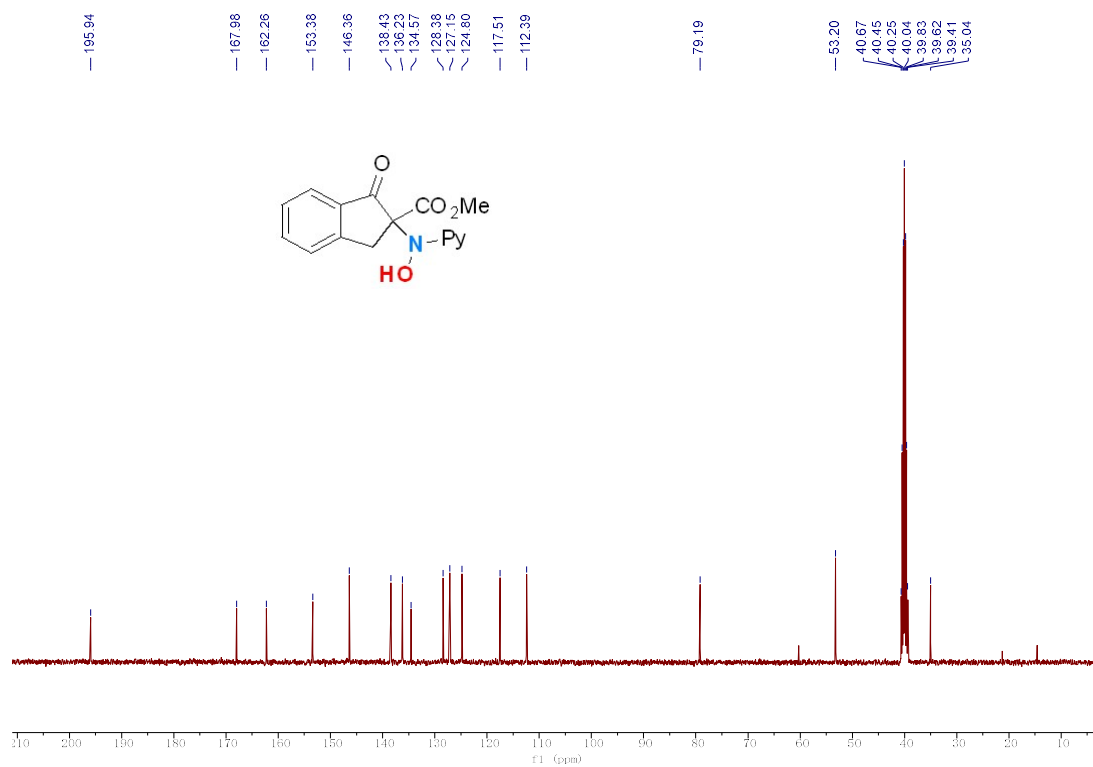
To a 10 mL Schlenk flask equipped with a magnetic stir bar was added **49** (0.1 mmol) and **3** (0.15 mmol) in THF (1.0 mL). The resulting mixture was stirred at a distance of ~3 cm from a 24 w blue LED at room temperature for 12 h. The solvent was removed by vacuum and the crude product was purified by flash chromatography on silica gel silica: 200~300; eluant: petroleum ether/ethyl acetate (20:1~5:1) to provide pure product **5** as a colorless oil in 87% yield (45.1 mg).

Information on chemical shift studies

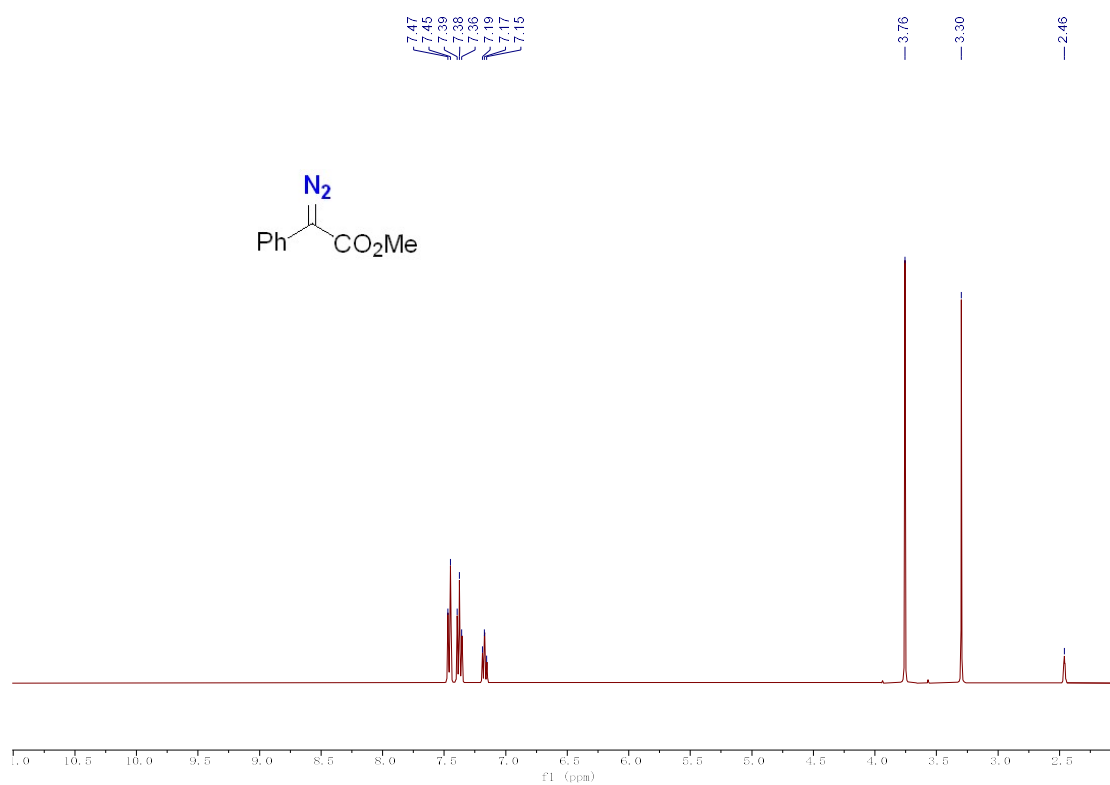
^1H NMR (400 MHz) Spectrum of 49 in $(\text{CD}_3)_2\text{SO}$



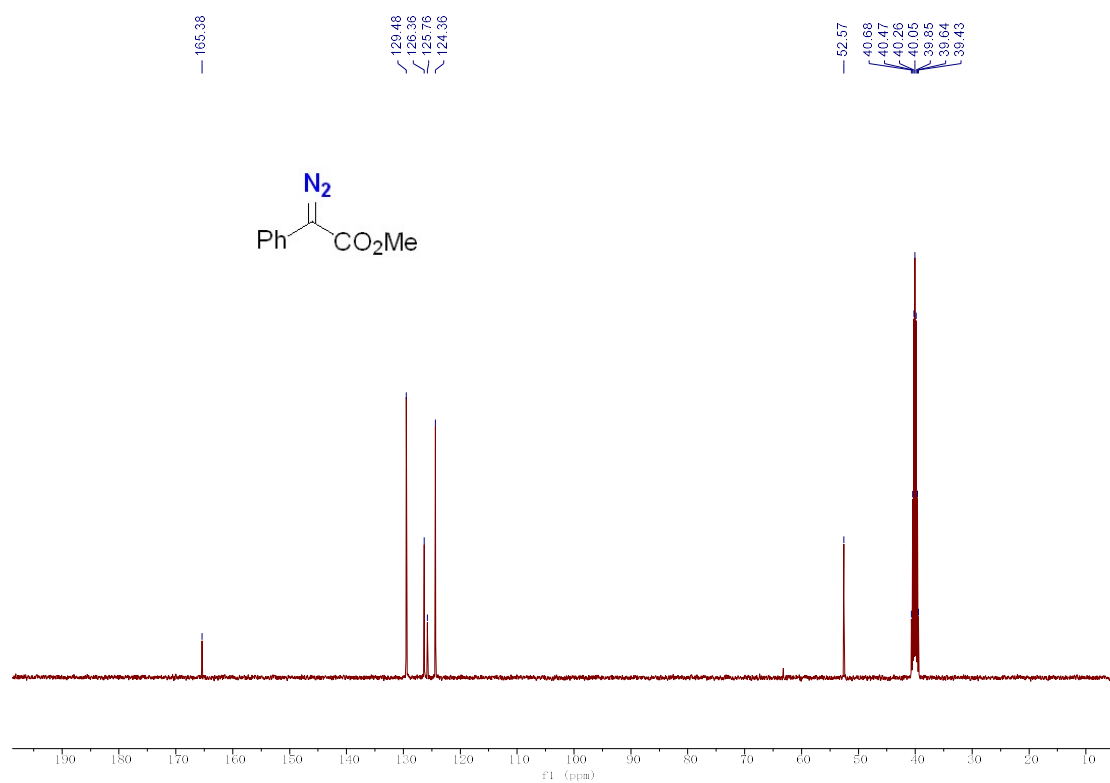
^{13}C NMR (100 MHz) Spectrum of 49 in $(\text{CD}_3)_2\text{SO}$



¹H NMR (400 MHz) Spectrum of 3 in (CD₃)₂SO



^{13}C NMR (100 MHz) Spectrum of 3 in $(\text{CD}_3)_2\text{SO}$



Chemical reaction scheme showing the synthesis of compound 10.19 from 1-phenyl-1-diazo-2-methoxyethene and methyl 2-hydroxy-2-(pyridin-2-yl)-3-oxoindane-1-carboxylate.

COC(=O)C(=N=[N])c1ccccc1 + COC(=O)C1(O)C(=O)c2ccccc2N1c3ccncc3 → COC(=O)C1(O)C(=O)c2ccccc2N1C(=O)C(=N=[N])c3ccccc3 (10.19)

¹H NMR spectrum (CDCl₃) of compound 10.19. The spectrum shows peaks in the aromatic region (6.82–7.96 ppm), a methoxy singlet (3.98 ppm), a hydroxyl singlet (3.74 ppm), a pyridine ring singlet (3.55 ppm), and a methyl ester singlet (3.30 ppm). Integration values are provided for each peak.

Chemical Shift (ppm)	Integration
7.96	0.02
7.95	0.02
7.95	0.02
7.94	0.02
7.94	0.02
7.94	0.02
7.69	0.02
7.67	0.02
7.66	0.02
7.63	0.02
7.54	0.02
7.52	0.02
7.46	0.02
7.45	0.02
7.43	0.02
7.43	0.02
7.40	0.02
7.39	0.02
7.38	0.02
7.37	0.02
7.36	0.02
7.34	0.02
7.17	0.02
7.15	0.02
7.13	0.02
7.08	0.02
7.08	0.02
7.06	0.02
7.06	0.02
6.85	0.02
6.83	0.02
6.83	0.02
6.82	0.02
3.98	0.02
3.93	0.02
3.74	0.02
3.55	0.02
3.30	0.02
3.02	0.02
2.97	0.02

Chemical structures of the reactants are shown above the spectrum:

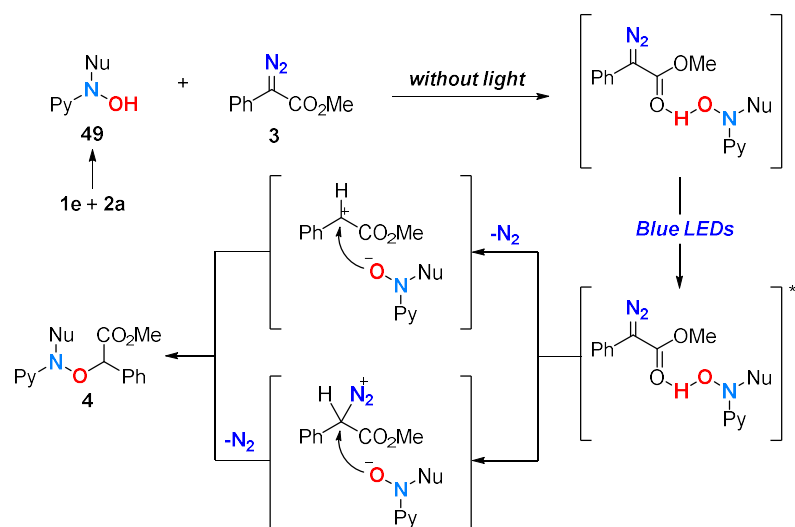
1. CC(=O)C(=[N+]=[N-])c1ccccc1 (Methyl 2-diazo-2-phenylacetate)

2. CCOC(=O)C1(O)N(C2CCCCC2)C3=CC=CC=C3C1=O (Methyl 1-(pyridin-2-yl)-2-hydroxy-2-oxo-1,2,3,4-tetrahydronaphthalene-1-carboxylate)

The spectrum displays the following chemical shifts (ppm):

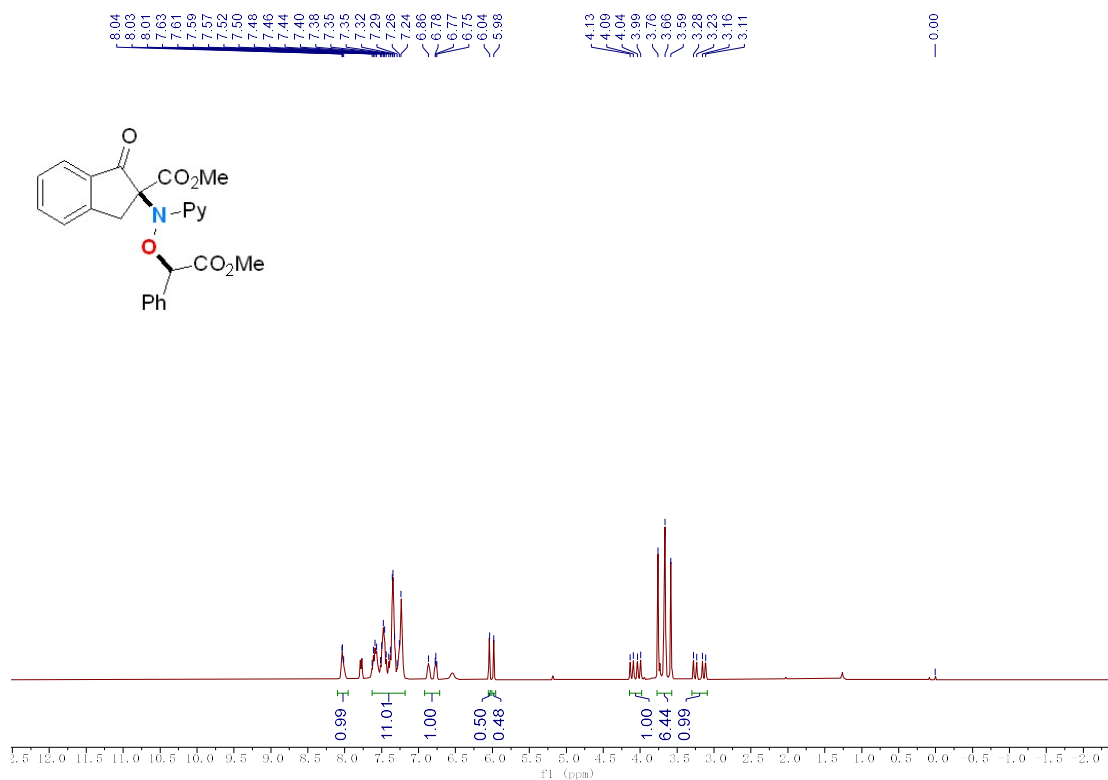
195.94, 167.98, 165.37, 162.28, 153.38, 146.35, 138.40, 136.20, 134.58, 129.47, 128.36, 127.13, 126.35, 125.76, 124.79, 124.35, 117.49, 112.39, 79.21, 53.18, 52.55, 49.67, 40.45, 40.25, 40.04, 39.83, 39.62, 39.41, 35.04.

Photo-induced proton transfer mechanism

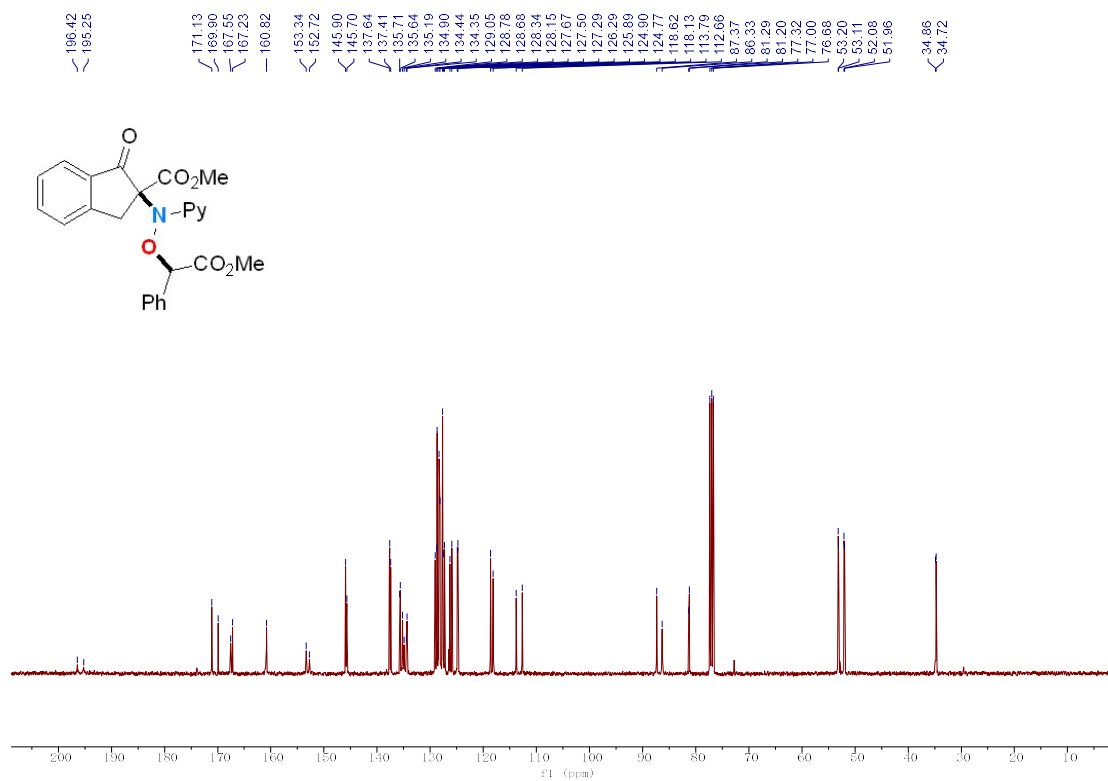


6. Copies of ^1H NMR and ^{13}C NMR Spectra

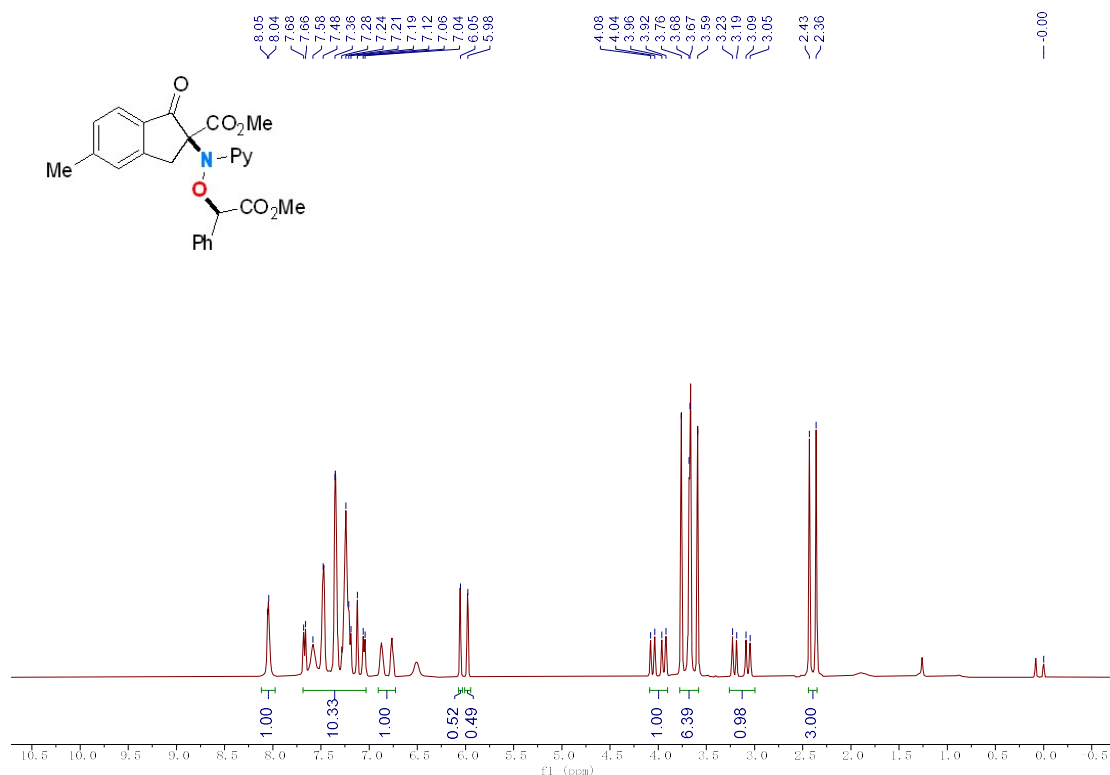
^1H NMR (400 MHz) Spectrum of 4 in CDCl_3



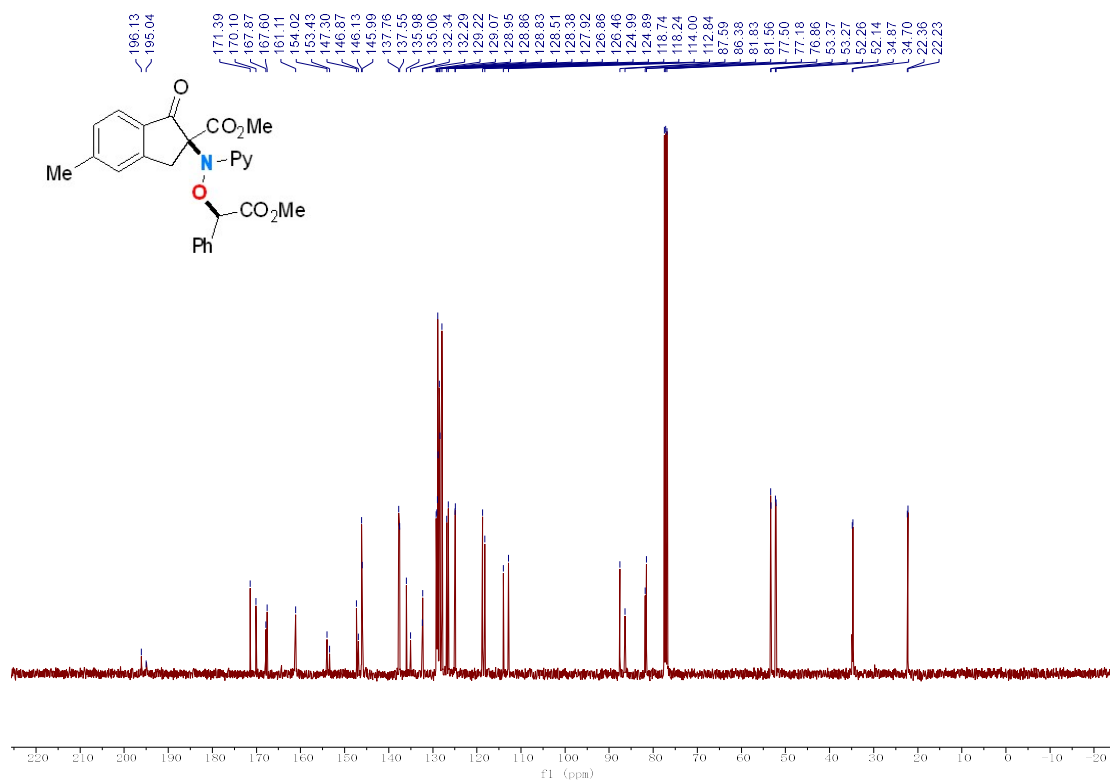
^{13}C NMR (100 MHz) Spectrum of 4 in CDCl_3



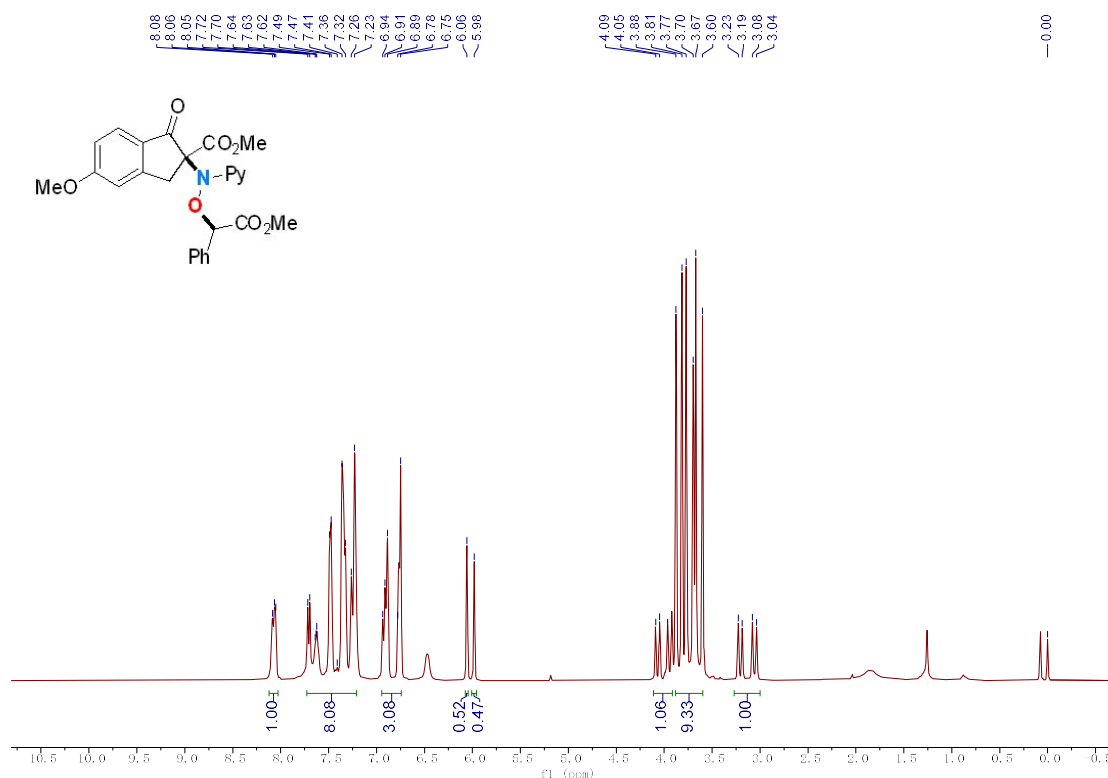
¹H NMR (400 MHz) Spectrum of 6 in CDCl₃



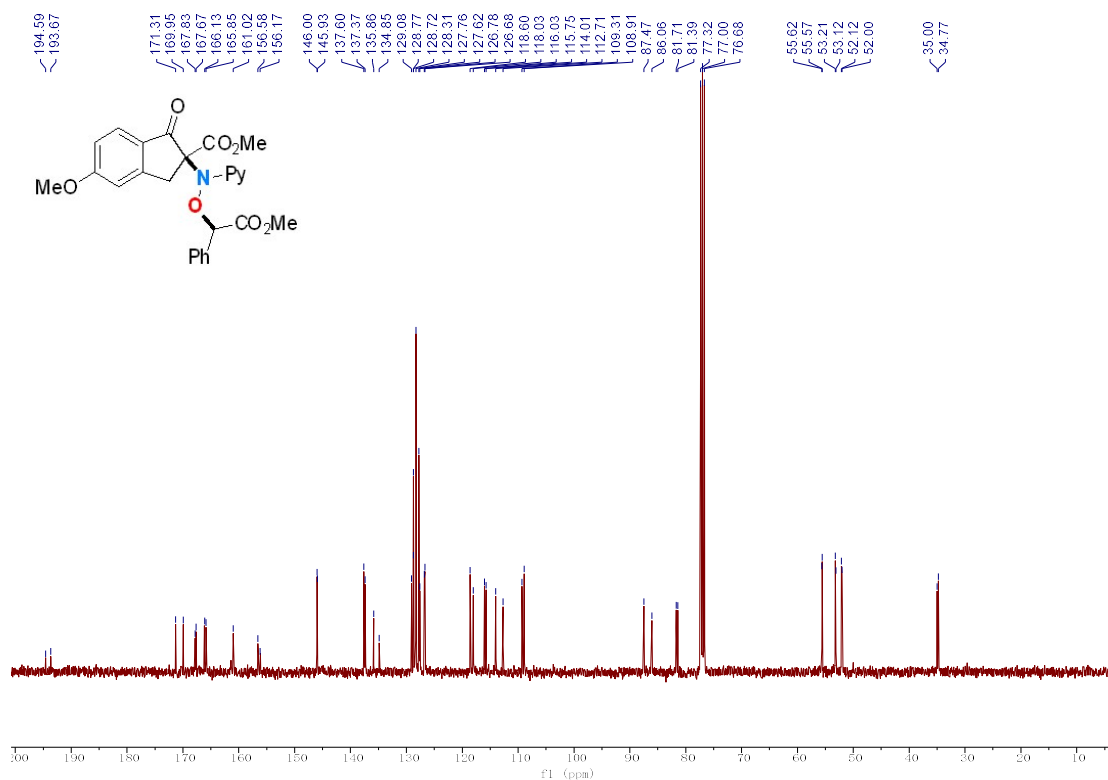
¹³CNMR (100 MHz) Spectrum of 6 in CDCl₃



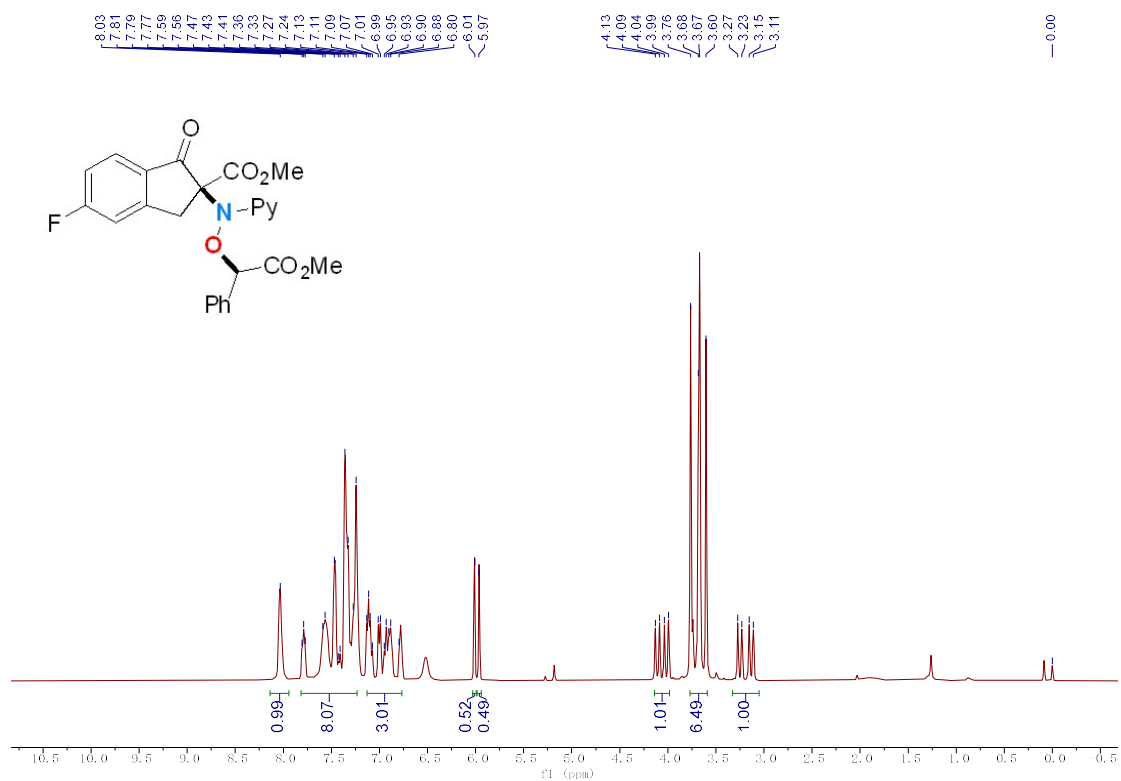
¹H NMR (400 MHz) Spectrum of 7 in CDCl₃



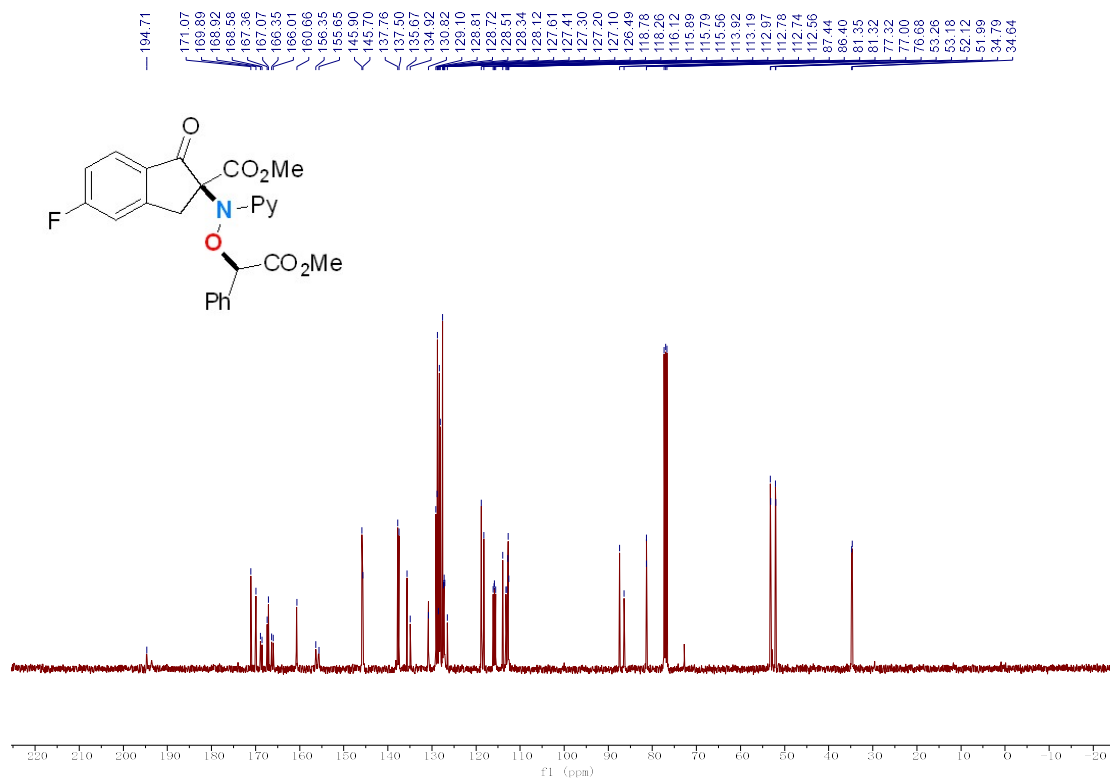
¹³CNMR (100 MHz) Spectrum of 7 in CDCl₃



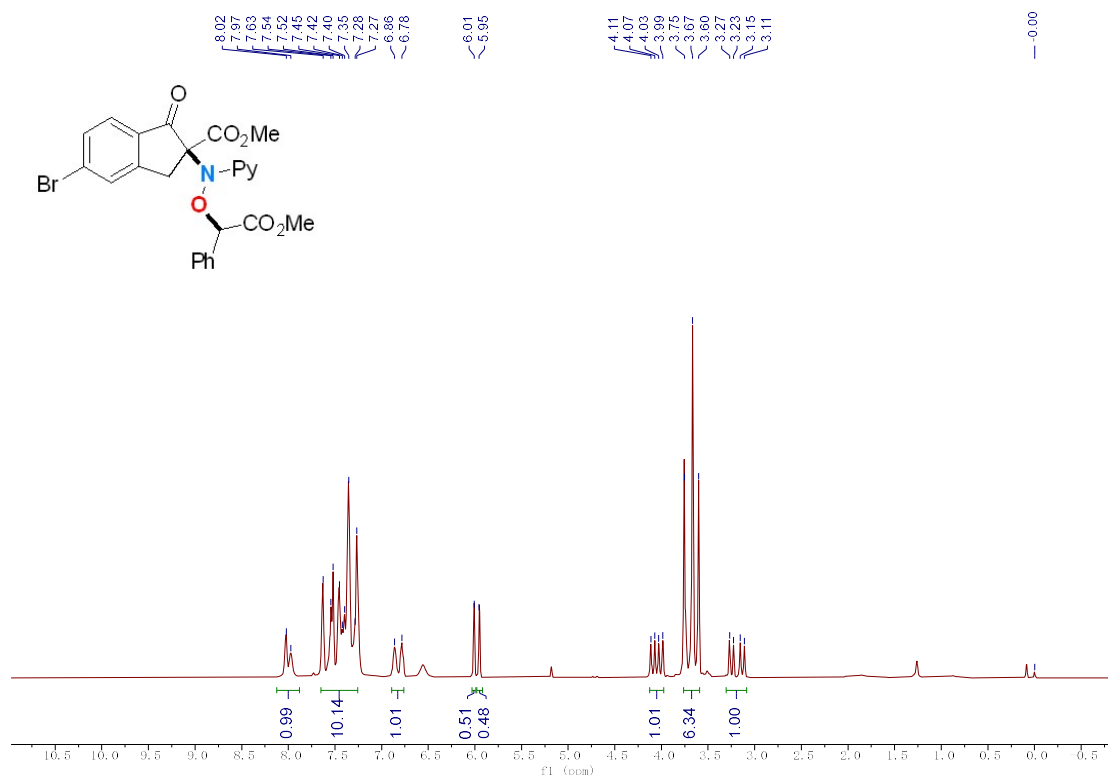
¹H NMR (400 MHz) Spectrum of 8 in CDCl₃



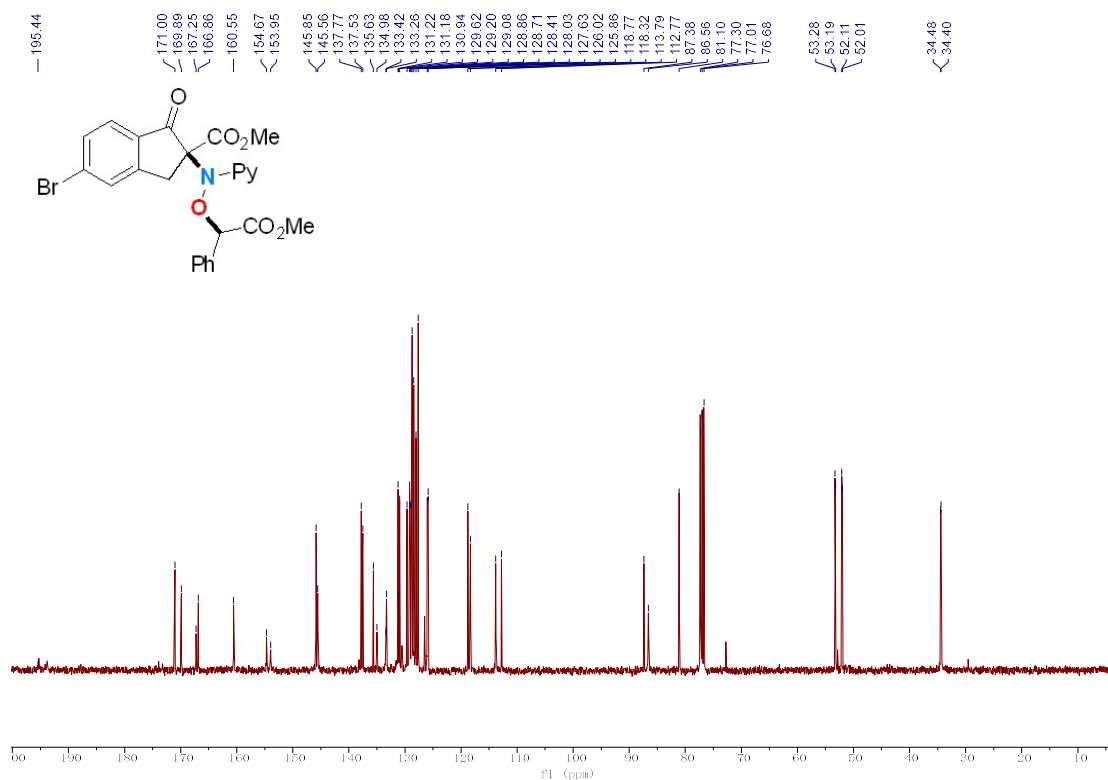
¹³CNMR (100 MHz) Spectrum of 8 in CDCl₃



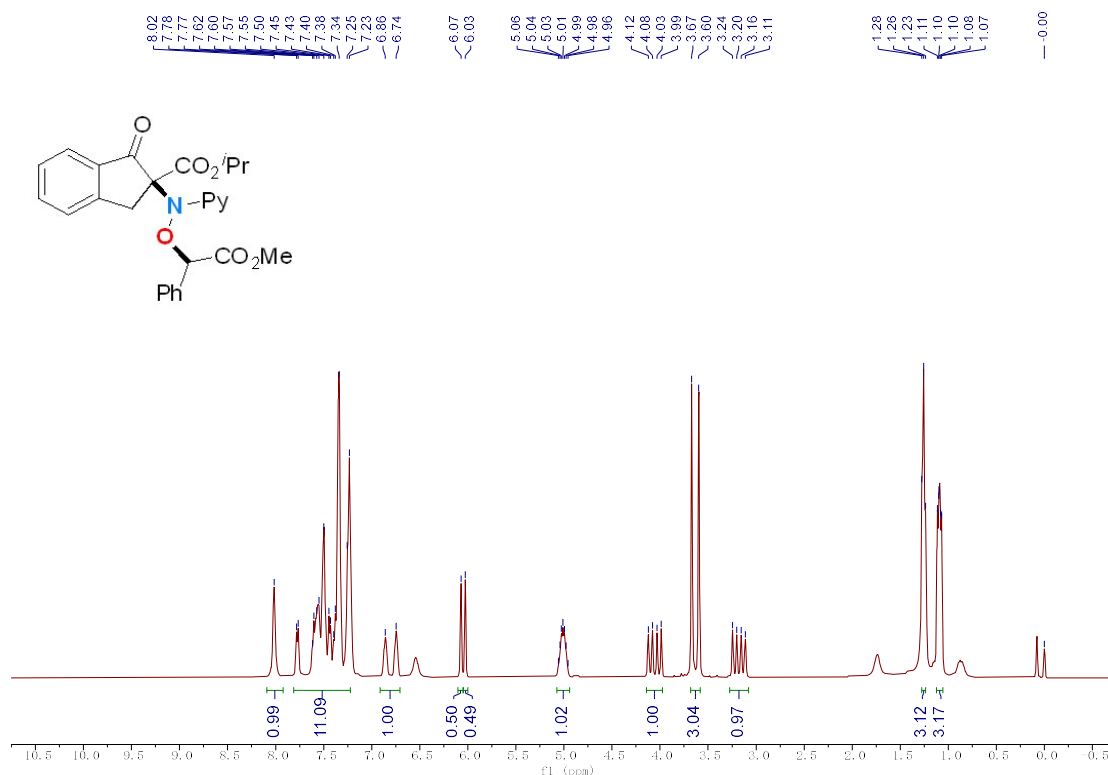
¹H NMR (400 MHz) Spectrum of 9 in CDCl₃



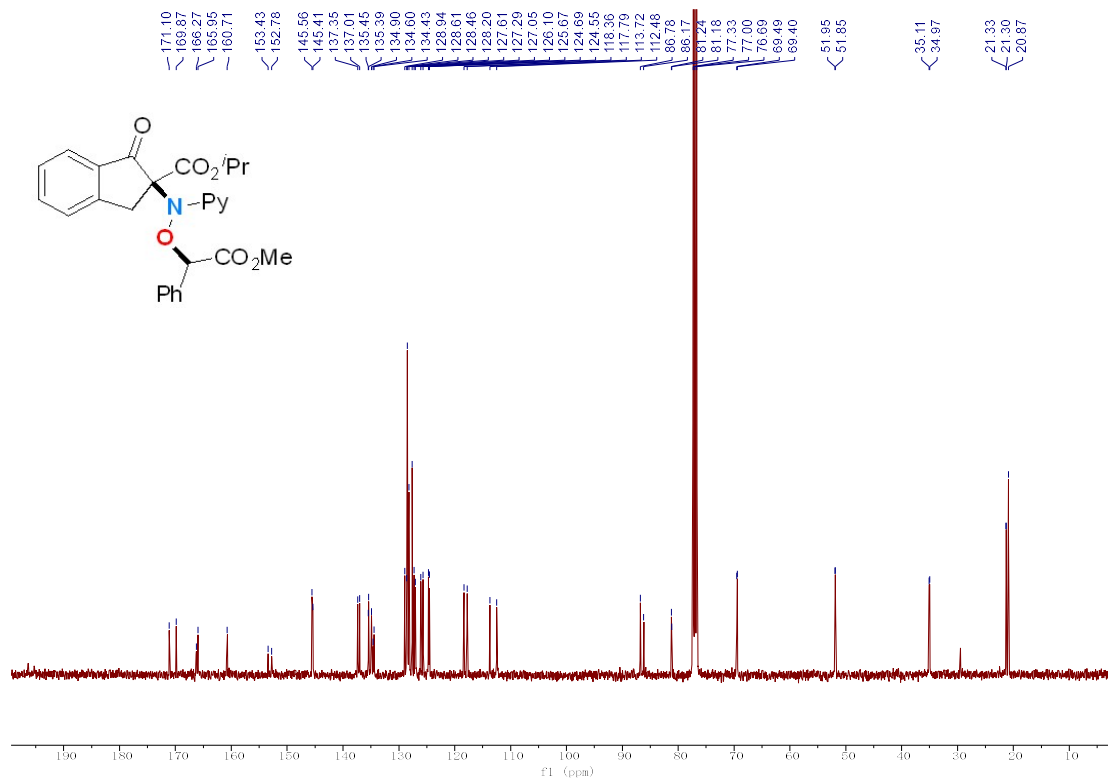
¹³CNMR (100 MHz) Spectrum of 9 in CDCl₃



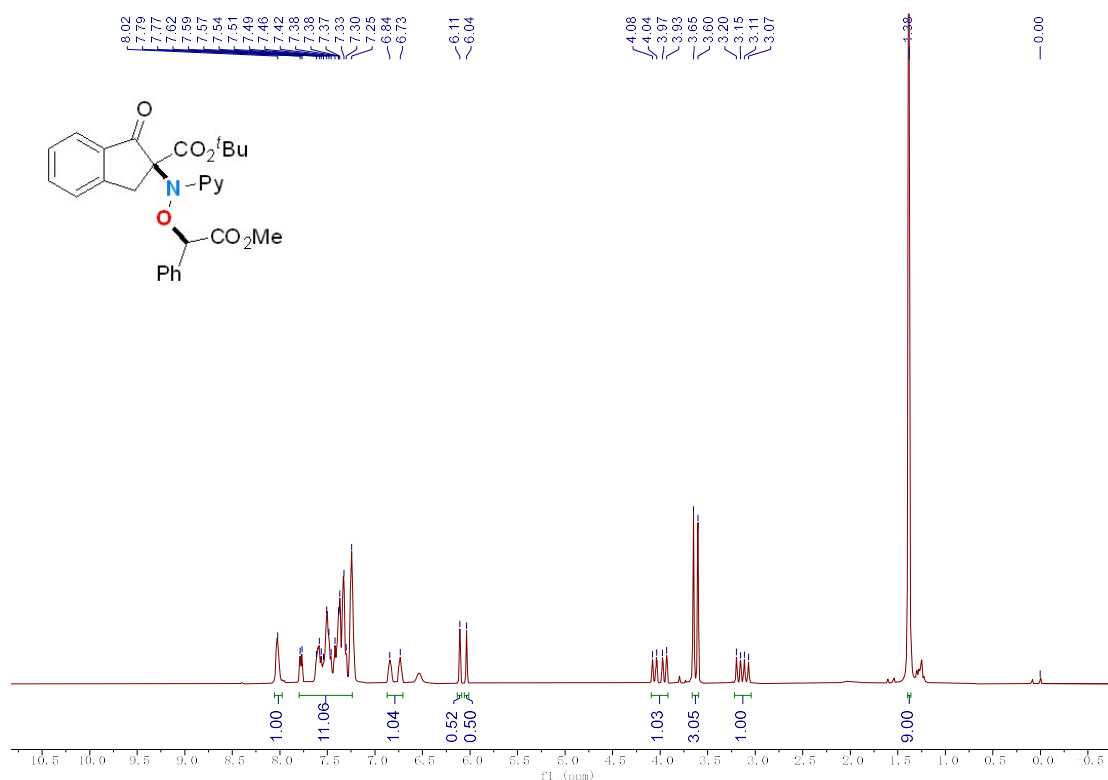
¹H NMR (400 MHz) Spectrum of 10 in CDCl₃



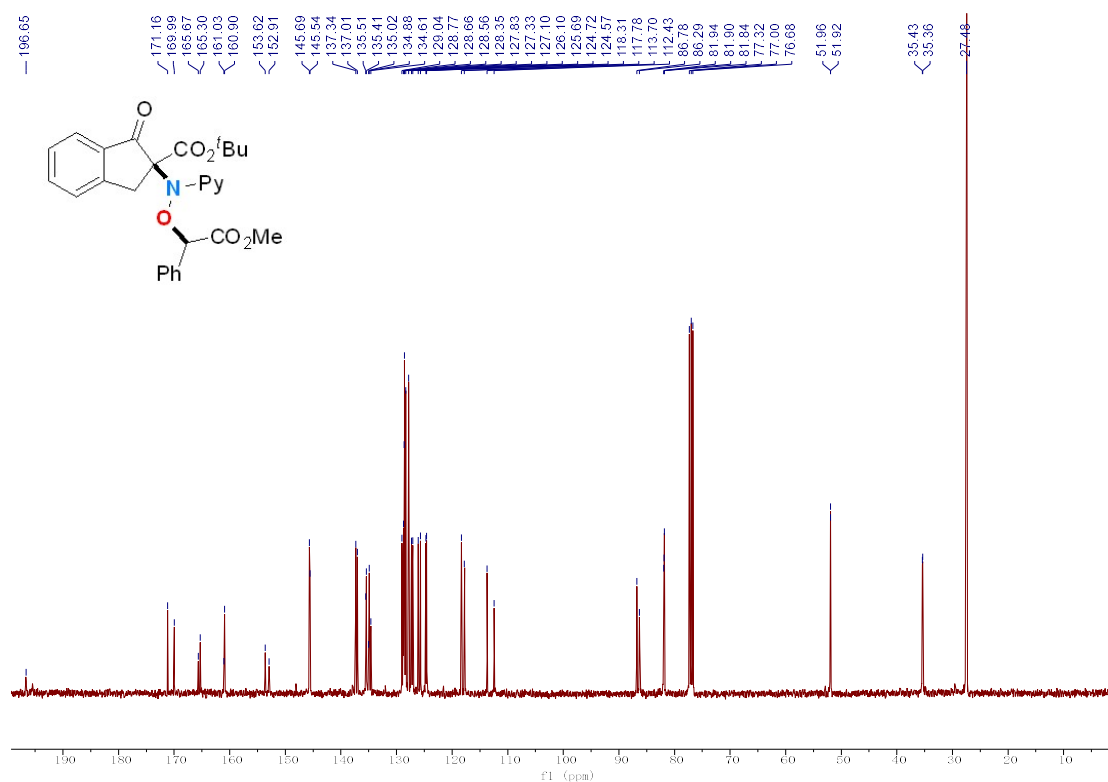
¹³C NMR (100 MHz) Spectrum of 10 in CDCl₃



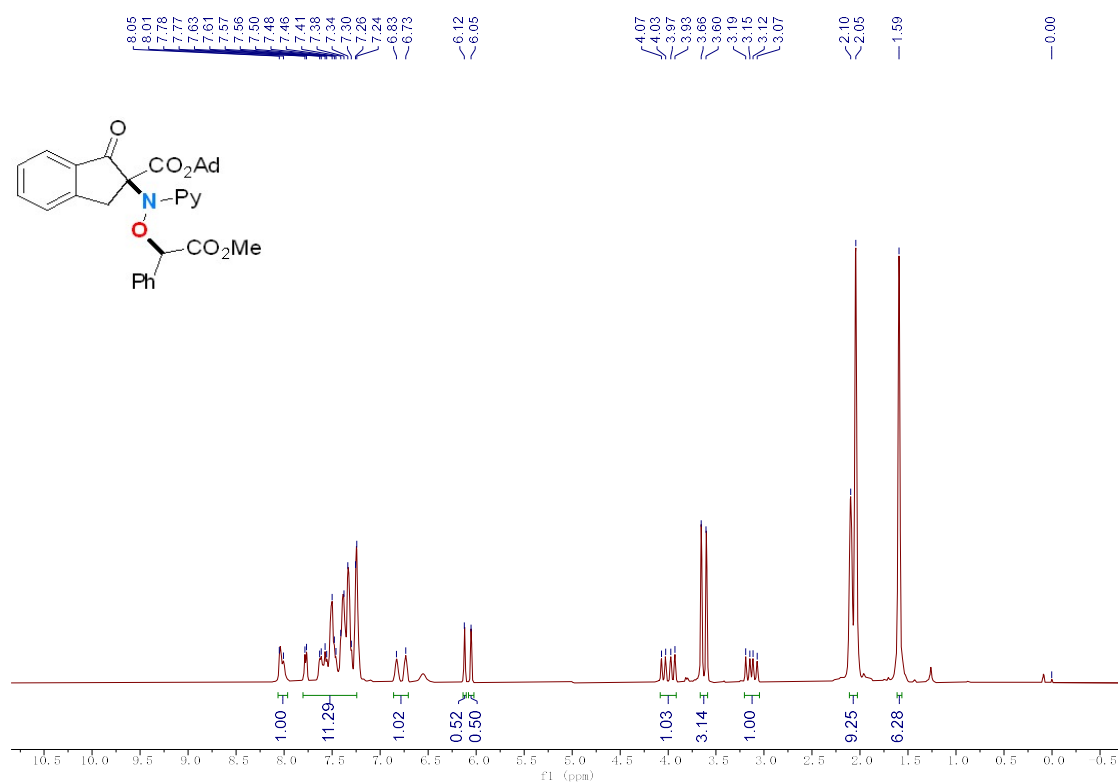
¹H NMR (400 MHz) Spectrum of 11 in CDCl₃



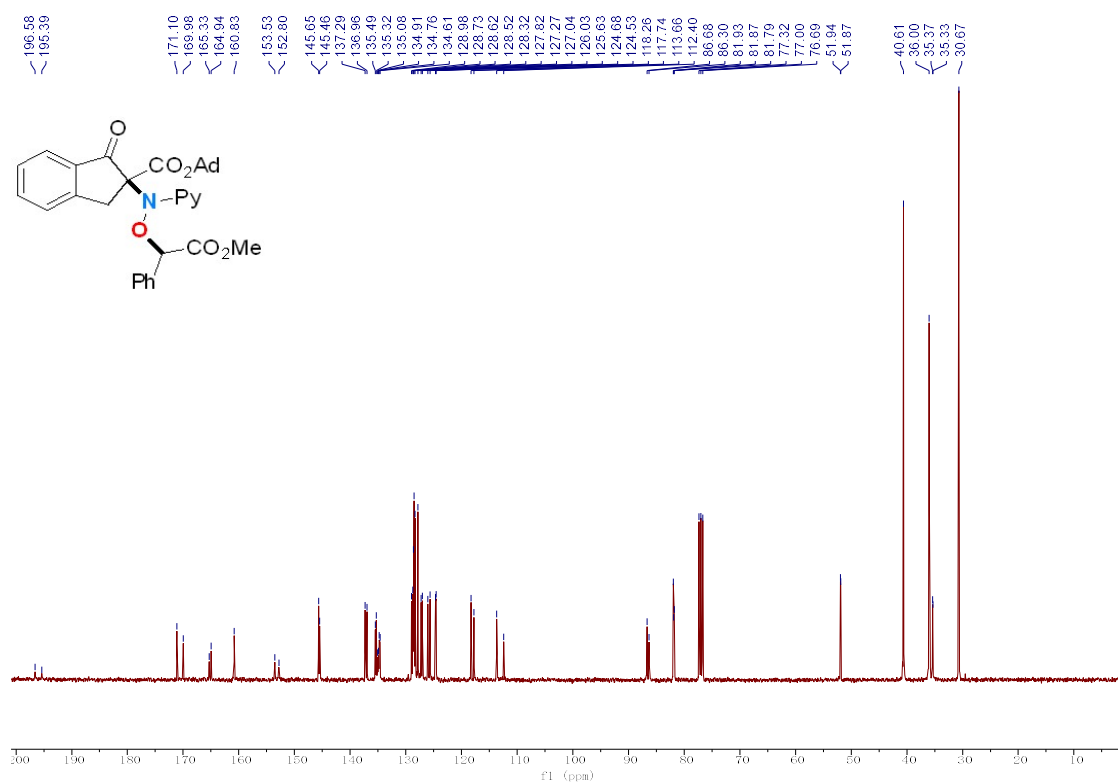
¹³CNMR (100 MHz) Spectrum of 11 in CDCl₃



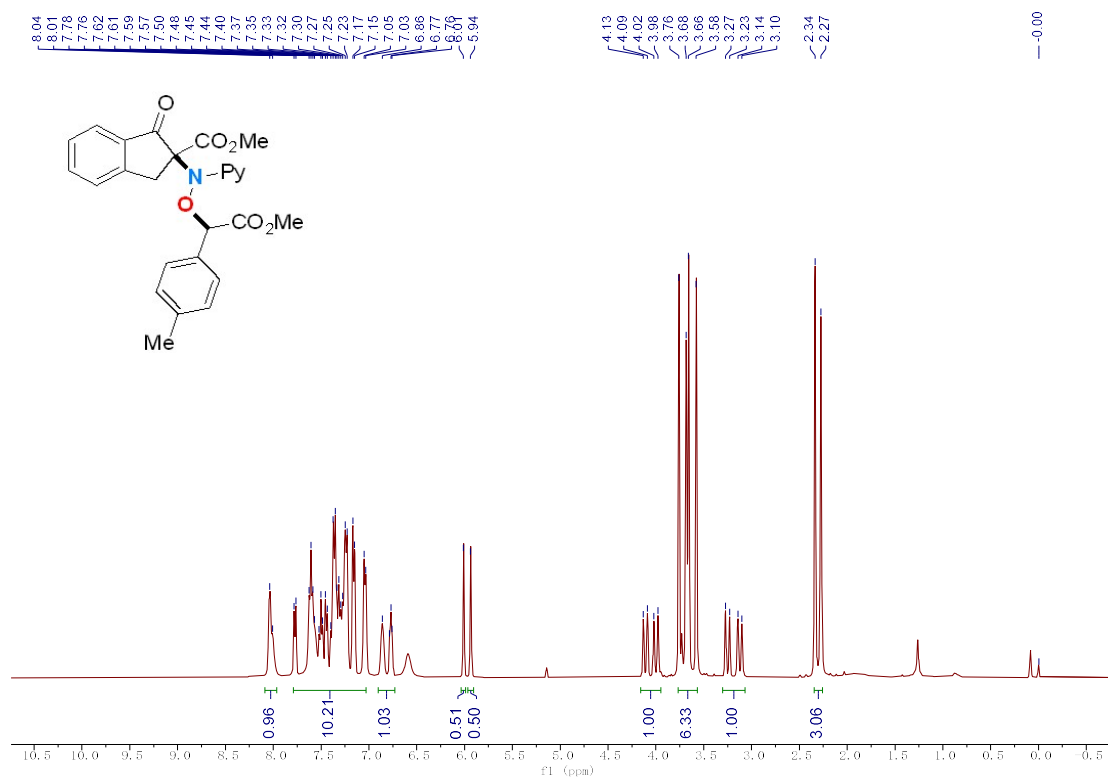
¹H NMR (400 MHz) Spectrum of 12 in CDCl₃



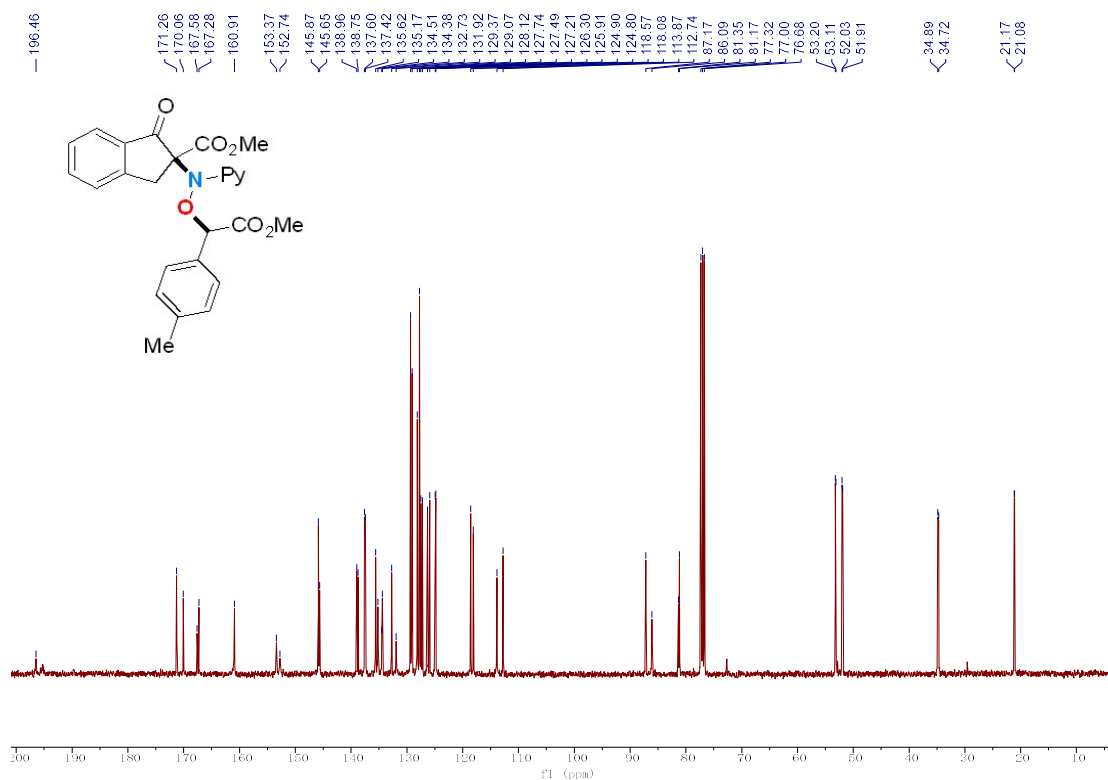
¹³C NMR (100 MHz) Spectrum of 12 in CDCl₃



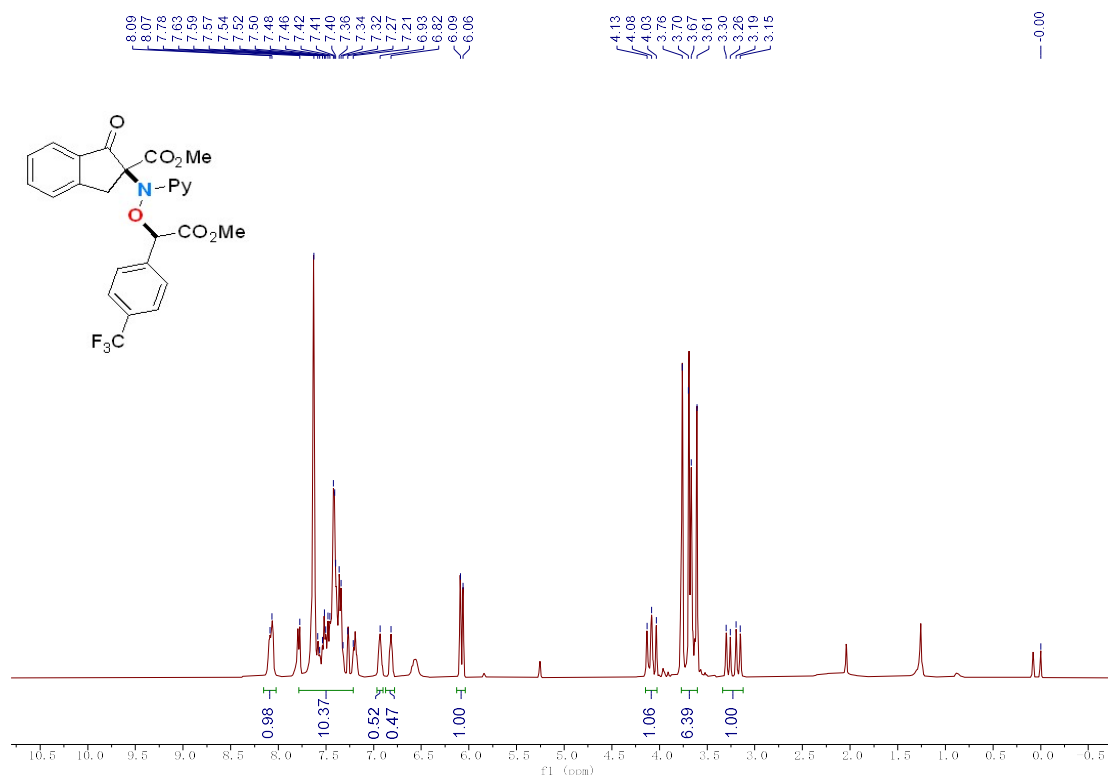
¹H NMR (400 MHz) Spectrum of 13 in CDCl₃



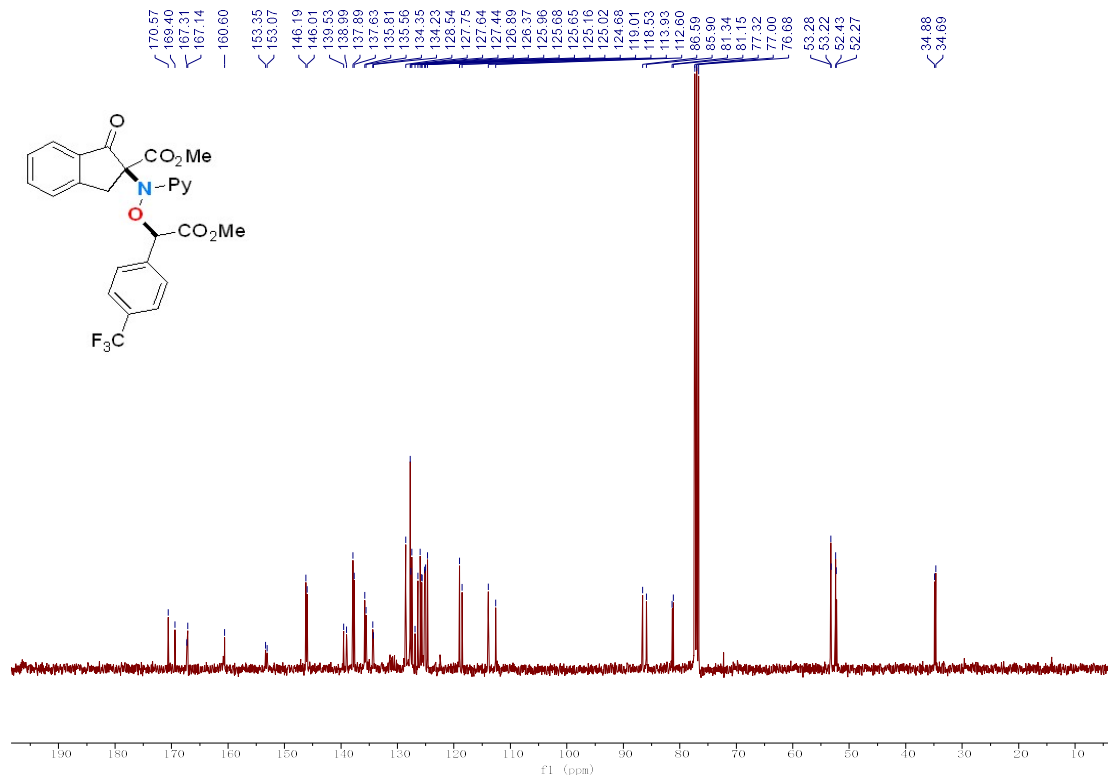
¹³CNMR (100 MHz) Spectrum of 13 in CDCl₃



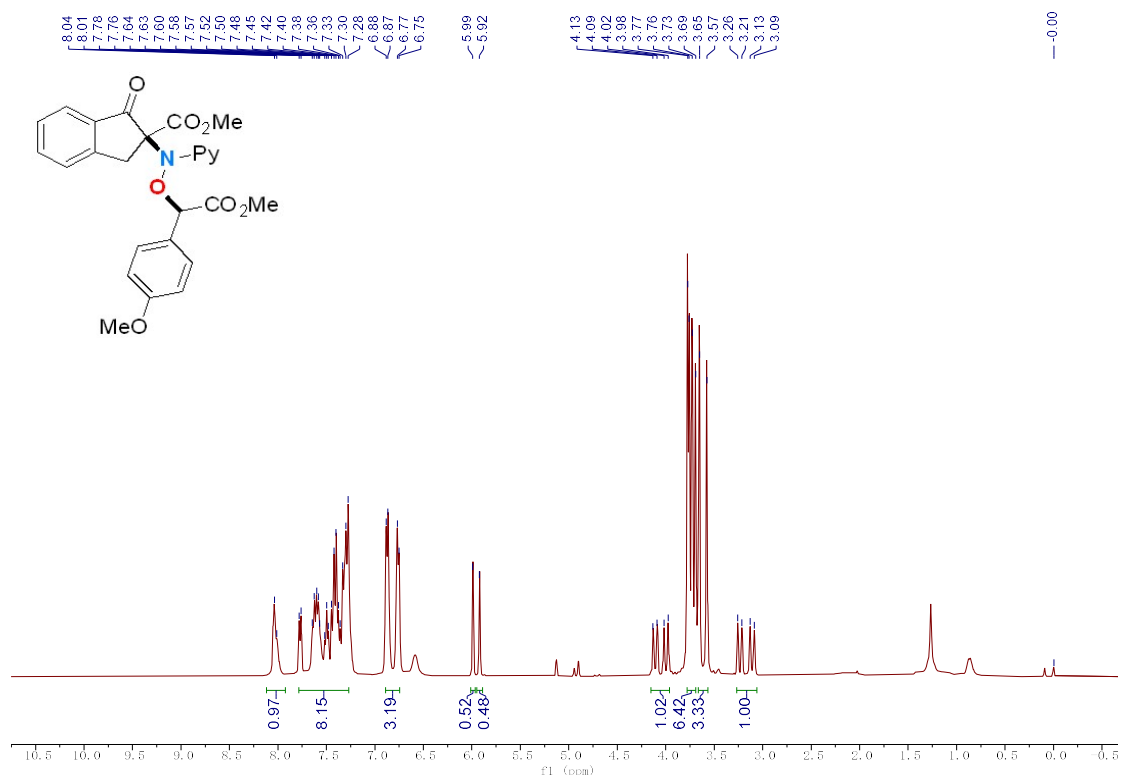
¹H NMR (400 MHz) Spectrum of 14 in CDCl₃



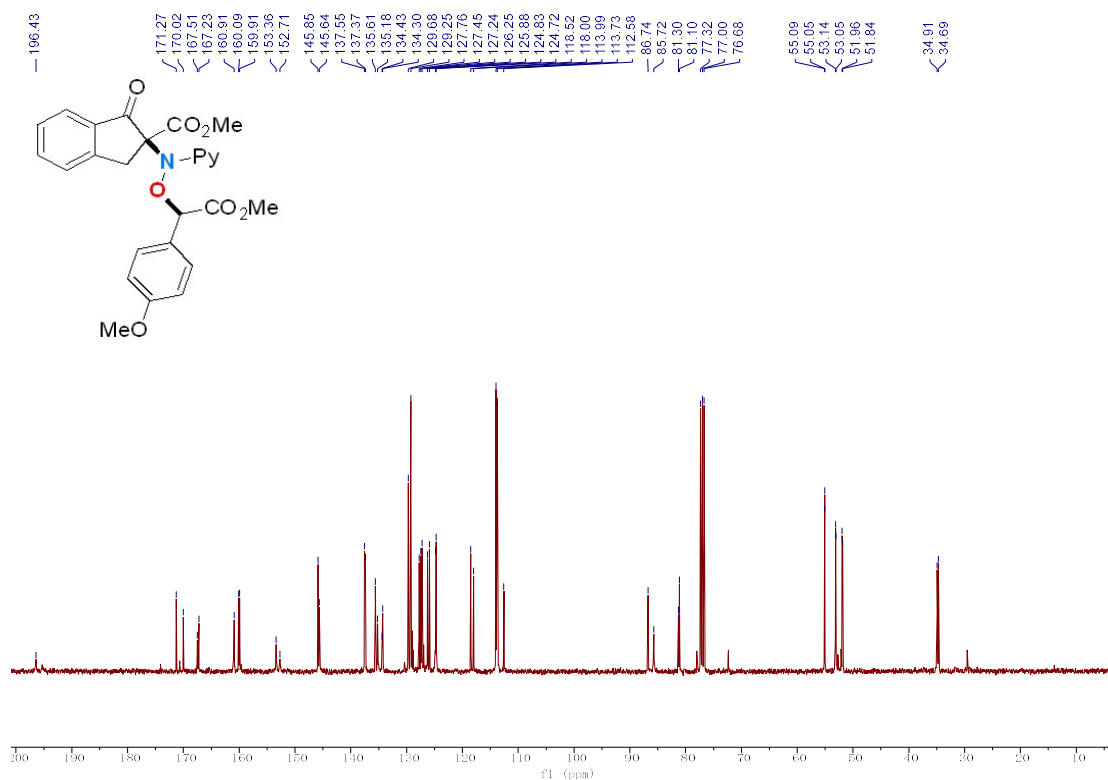
¹³CNMR (100 MHz) Spectrum of 14 in CDCl₃



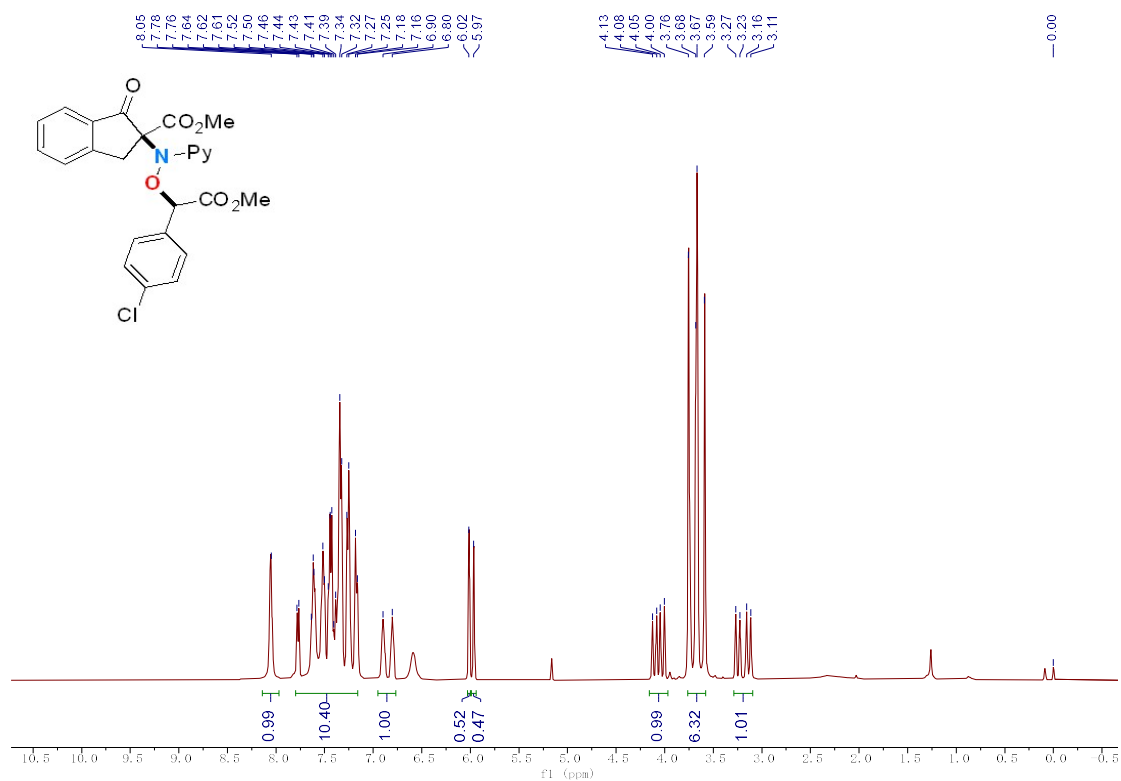
¹H NMR (400 MHz) Spectrum of 15 in CDCl₃



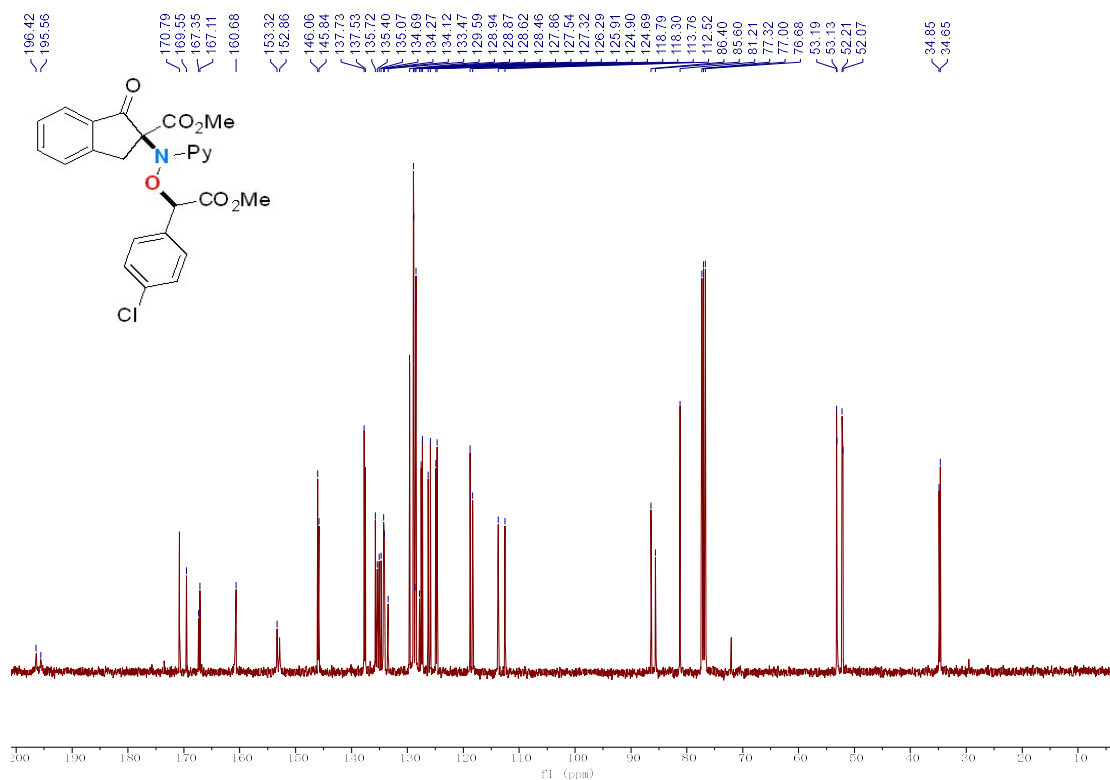
¹³CNMR (100 MHz) Spectrum of 15 in CDCl₃



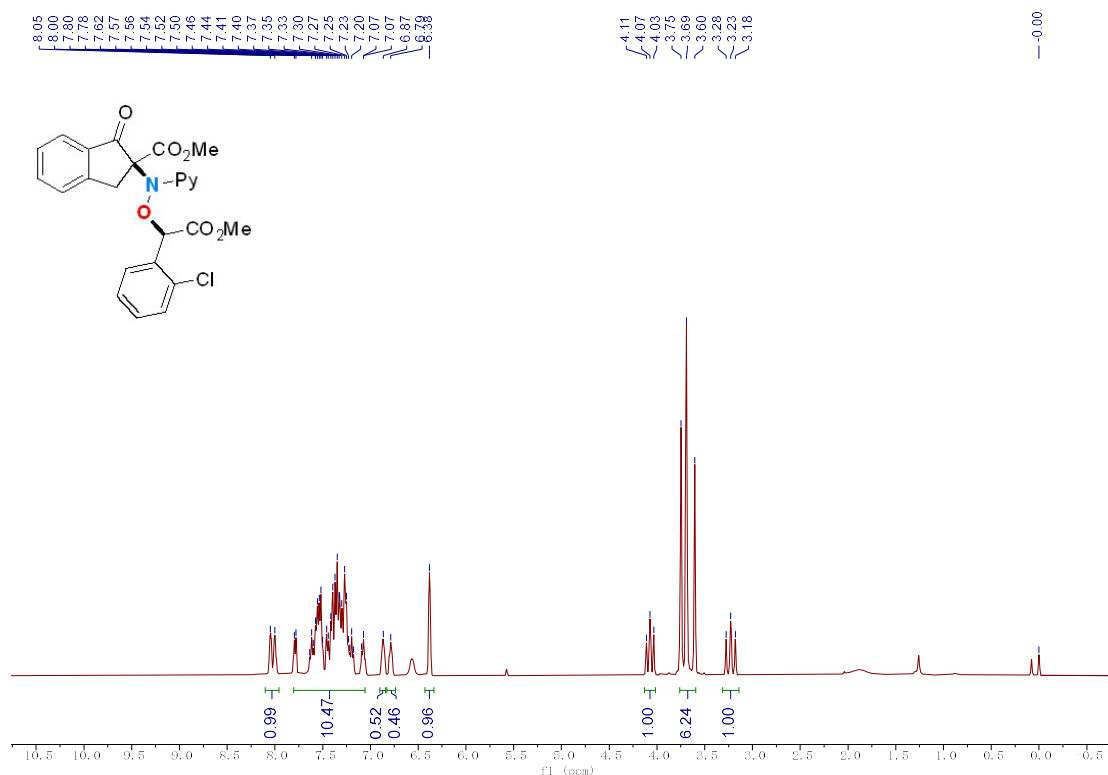
¹H NMR (400 MHz) Spectrum of 16 in CDCl₃



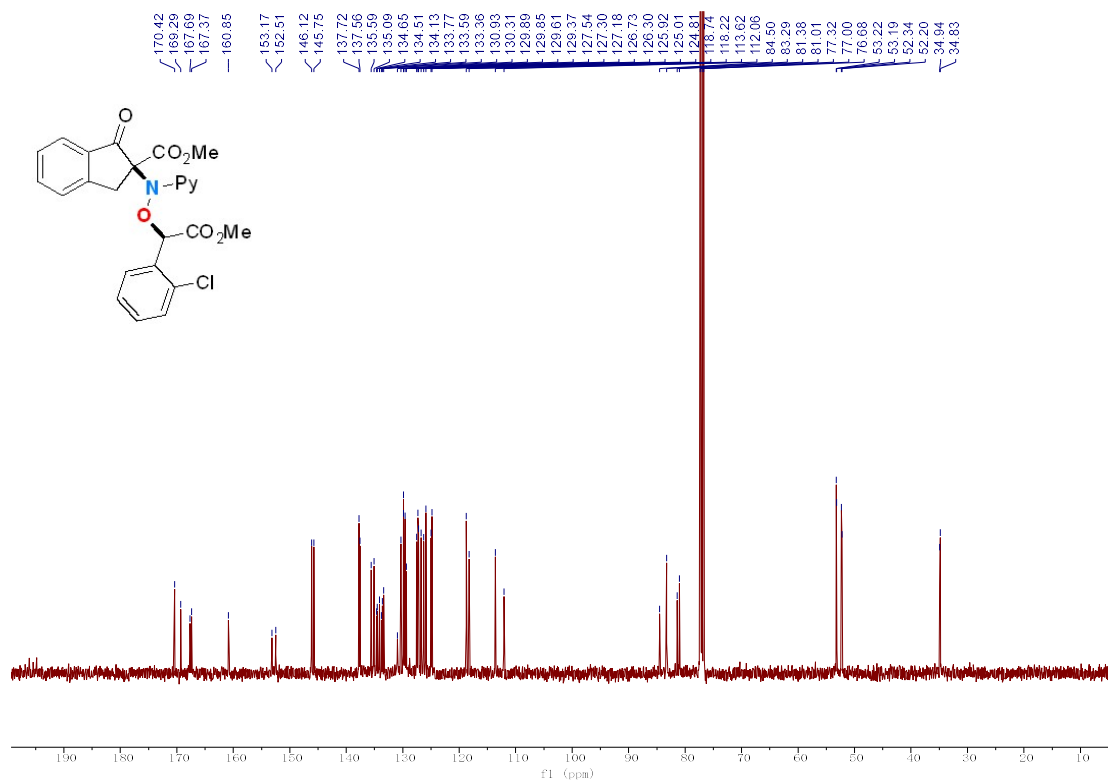
¹³CNMR (100 MHz) Spectrum of 16 in CDCl₃



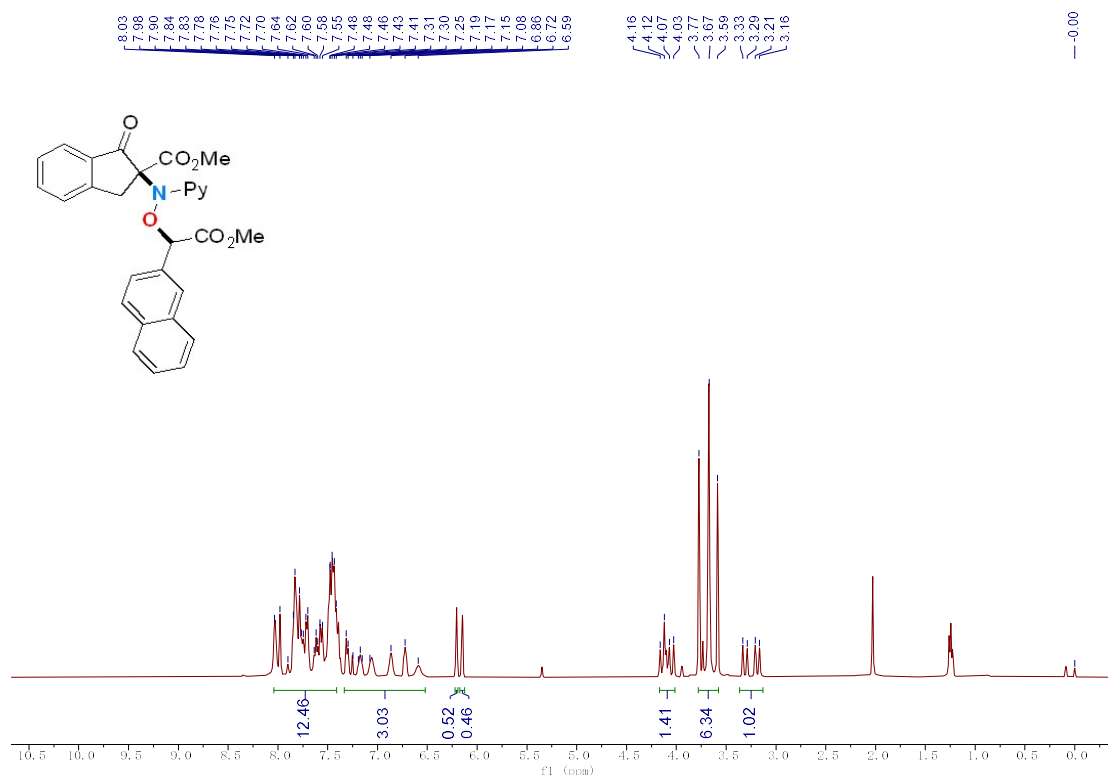
¹H NMR (400 MHz) Spectrum of 17 in CDCl₃



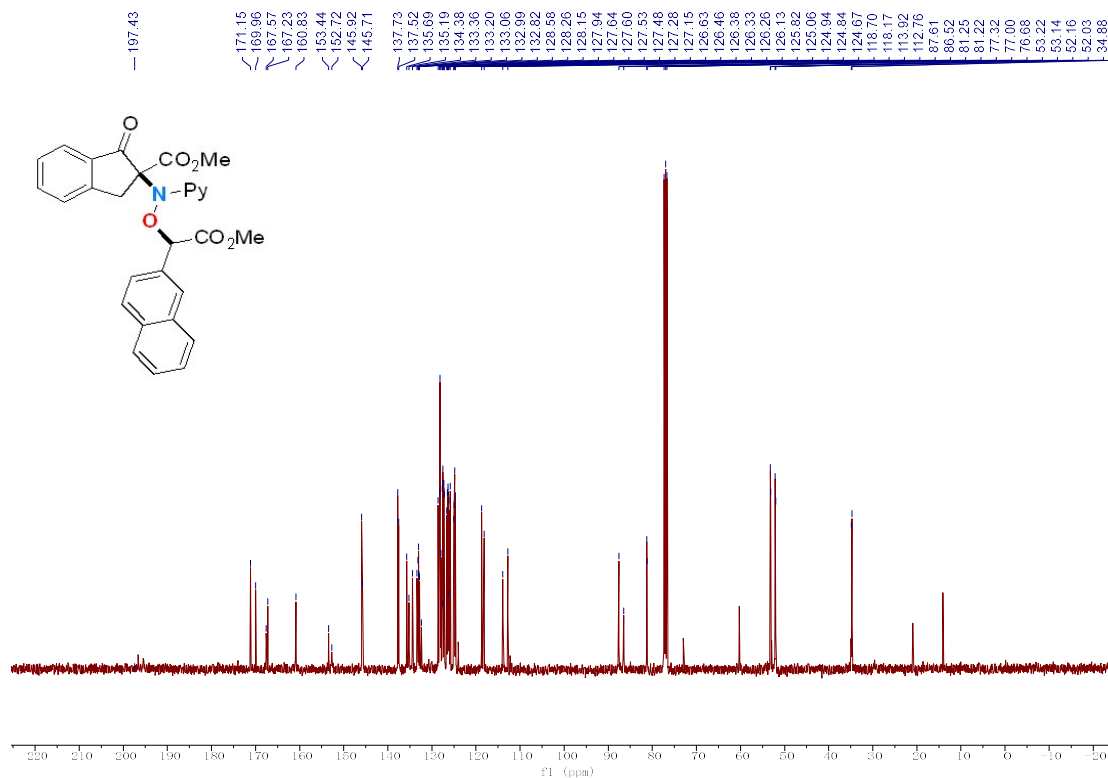
¹³CNMR (100 MHz) Spectrum of 17 in CDCl₃



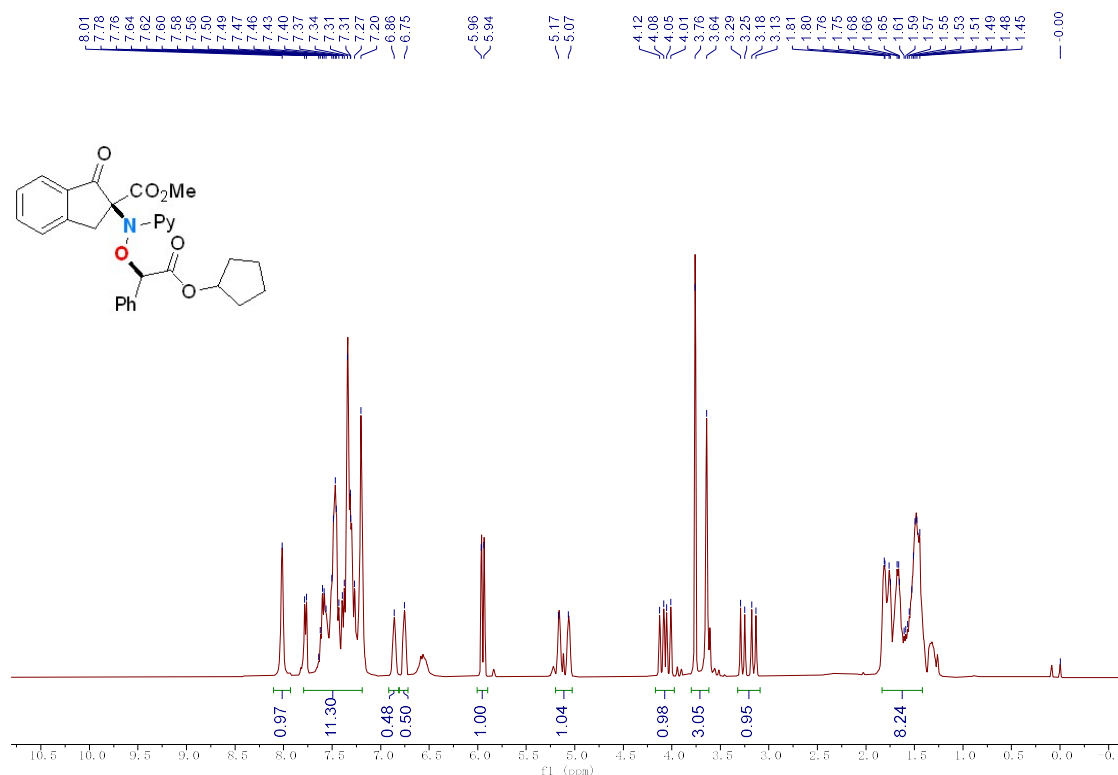
¹H NMR (400 MHz) Spectrum of 18 in CDCl₃



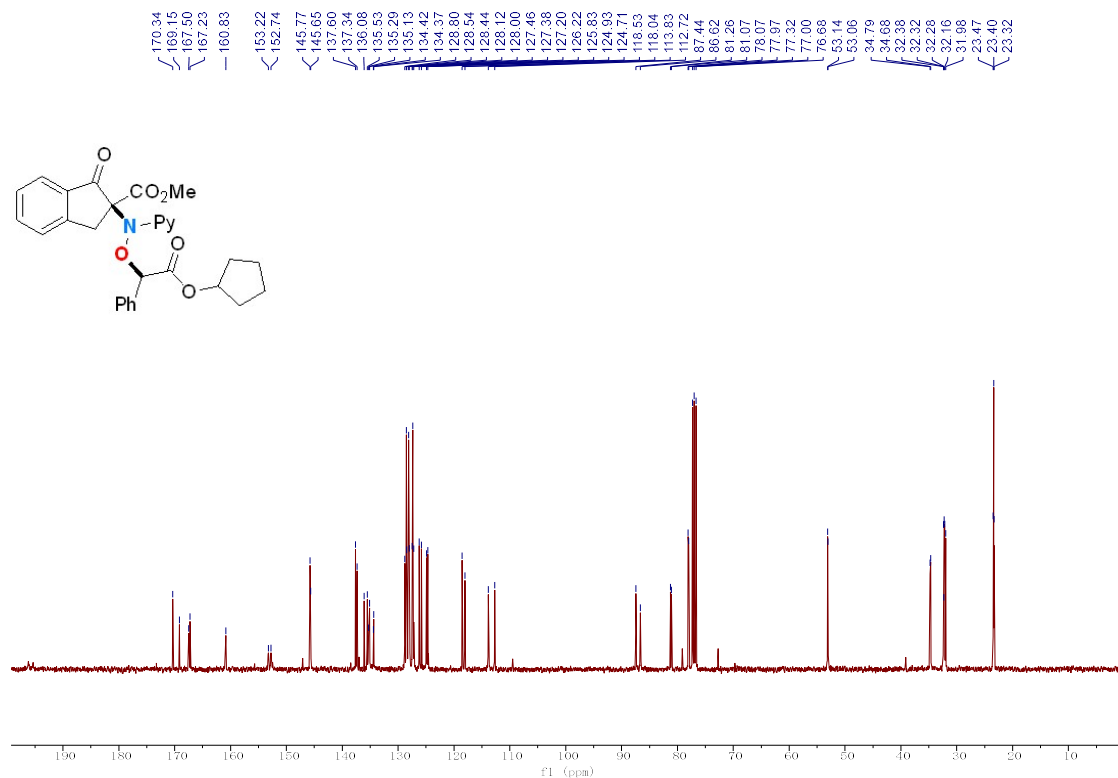
¹³C NMR (100 MHz) Spectrum of 18 in CDCl₃



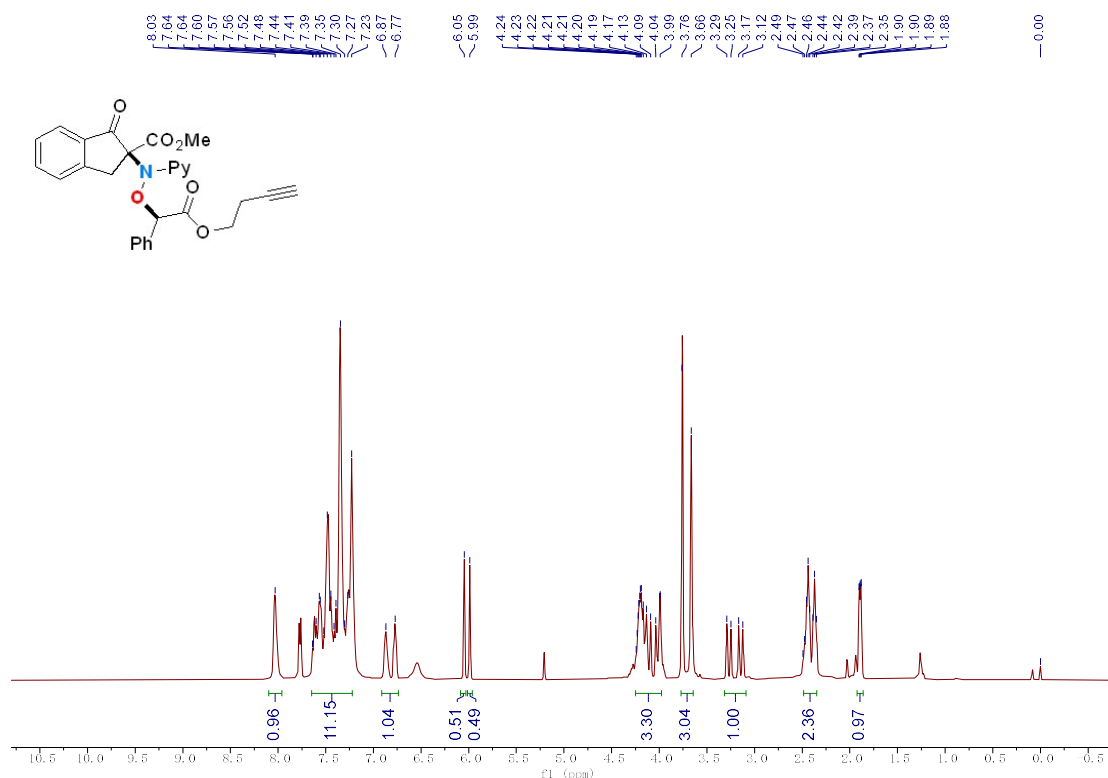
¹H NMR (400 MHz) Spectrum of 19 in CDCl₃



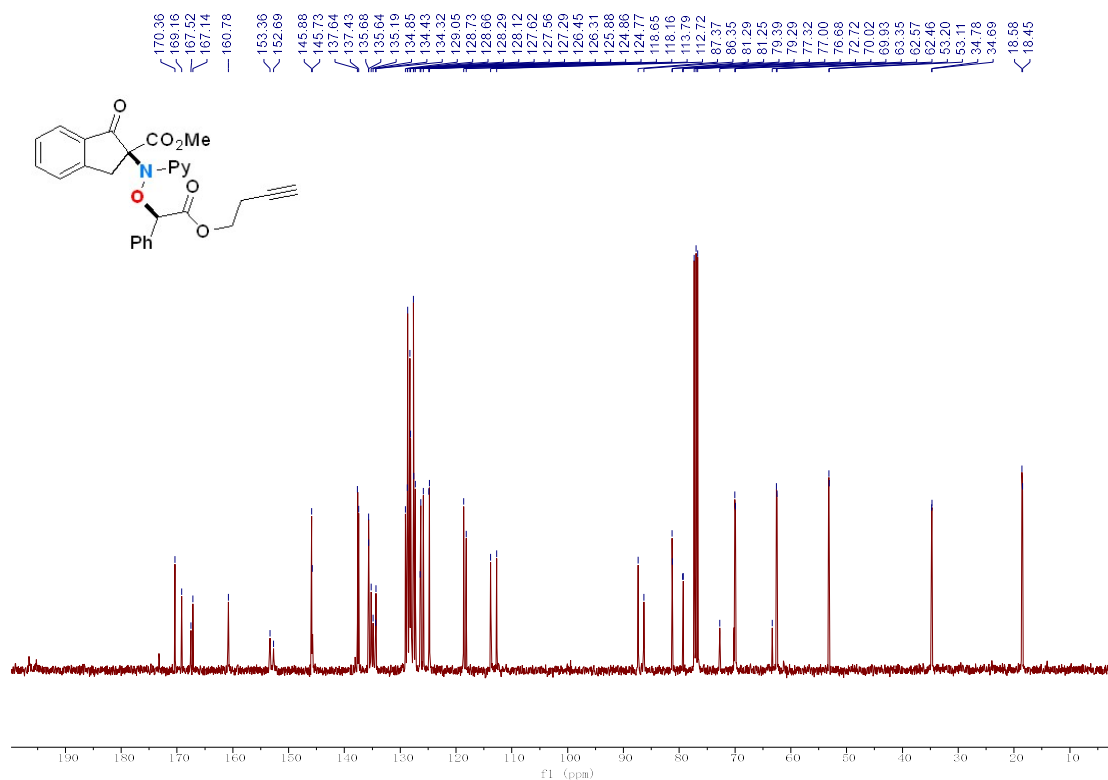
¹³C NMR (100 MHz) Spectrum of 19 in CDCl₃



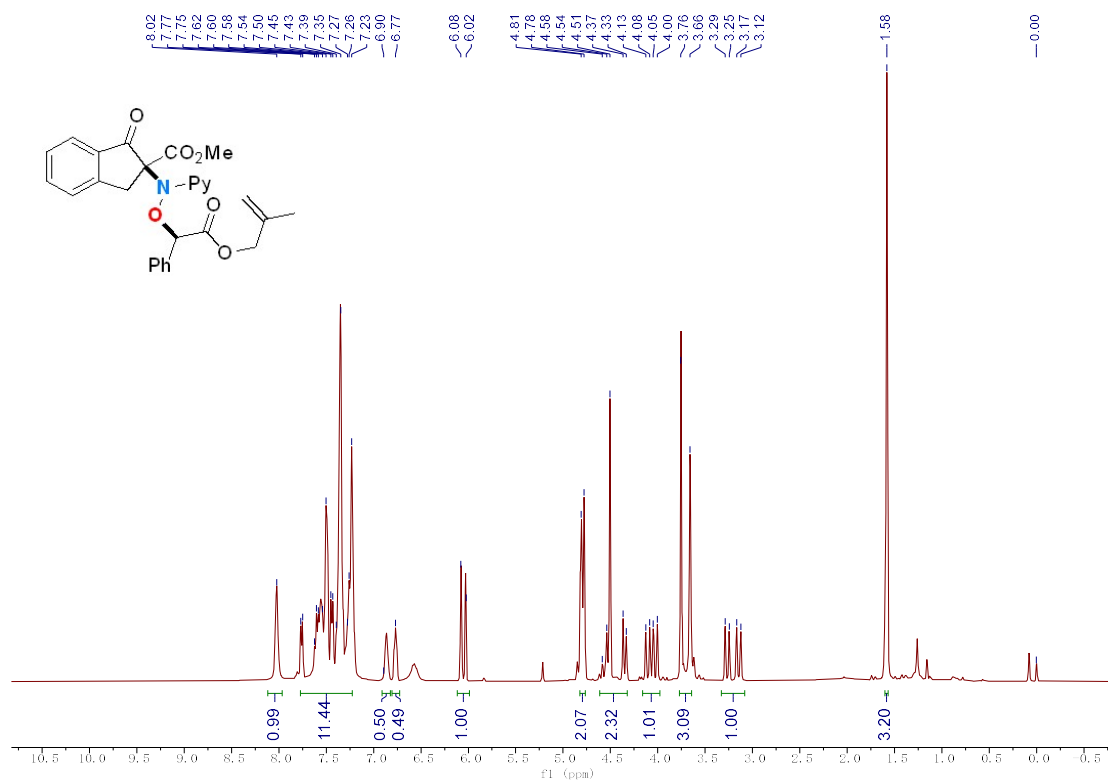
¹H NMR (400 MHz) Spectrum of 20 in CDCl₃



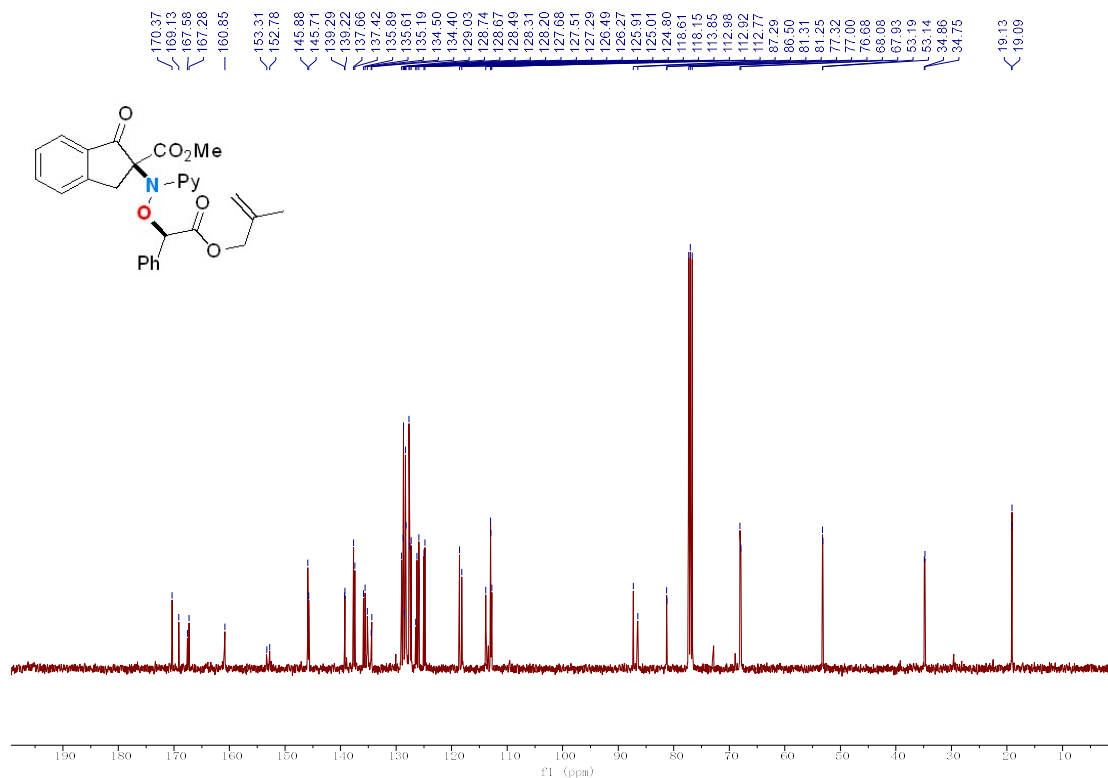
¹³CNMR (100 MHz) Spectrum of 20 in CDCl₃



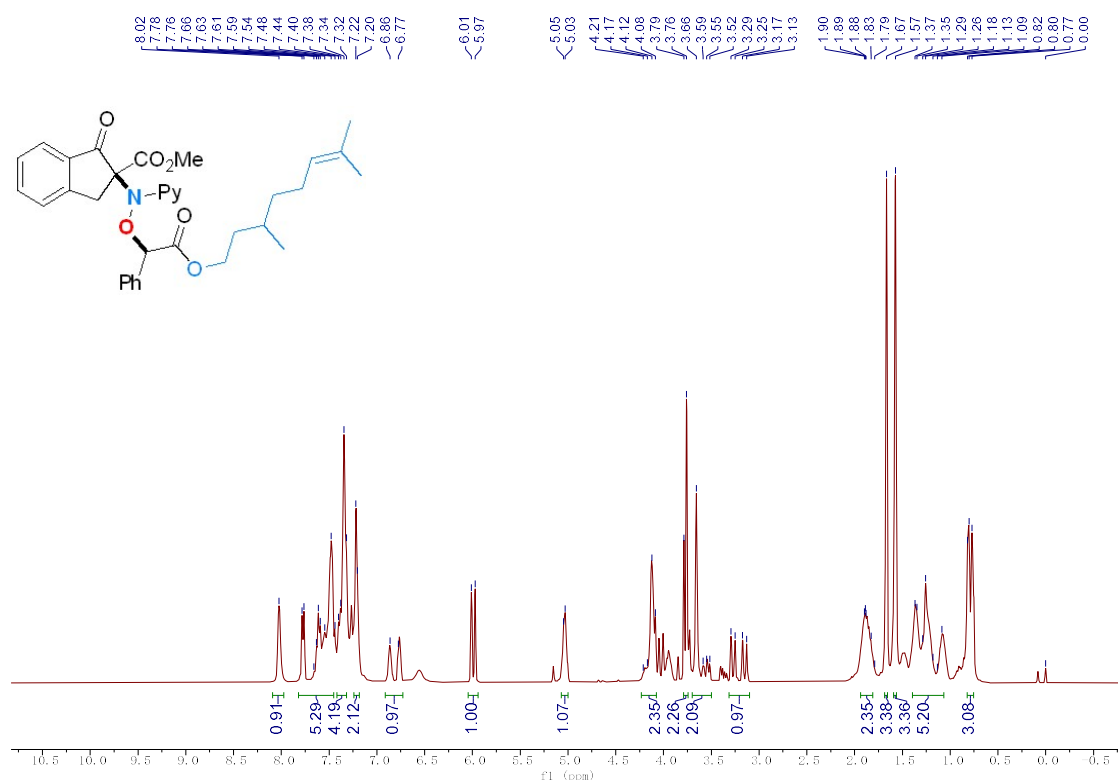
¹H NMR (400 MHz) Spectrum of 21 in CDCl₃



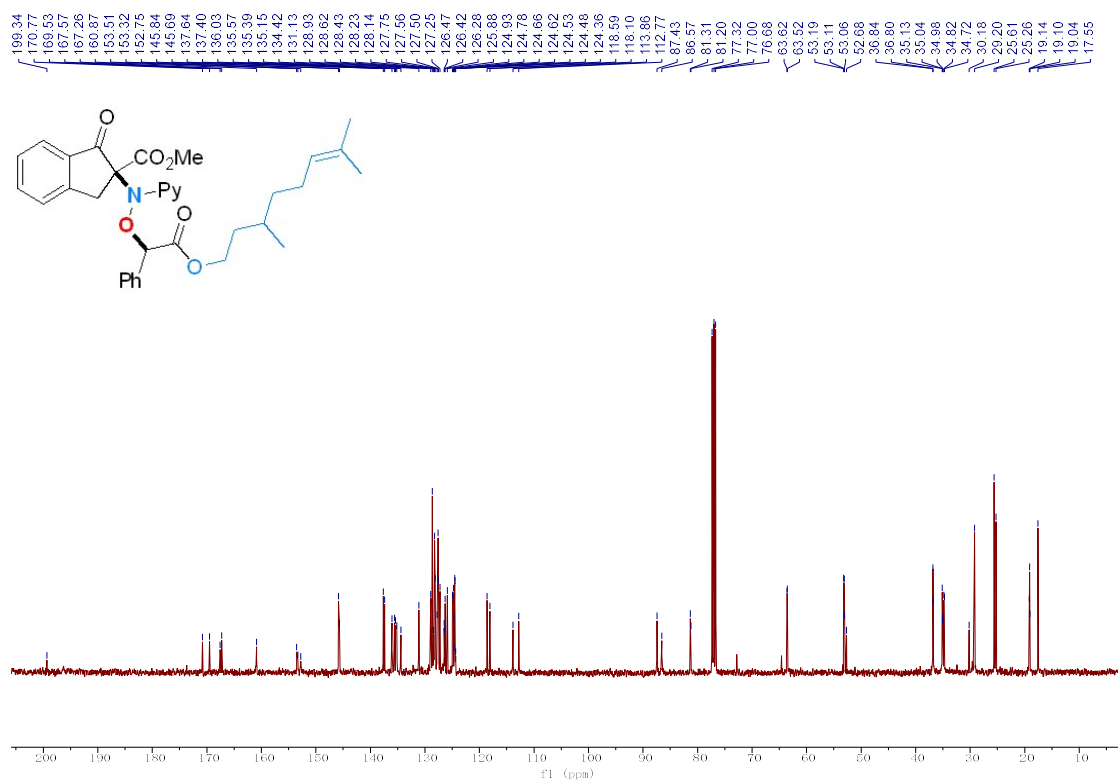
¹³C NMR (100 MHz) Spectrum of 21 in CDCl₃



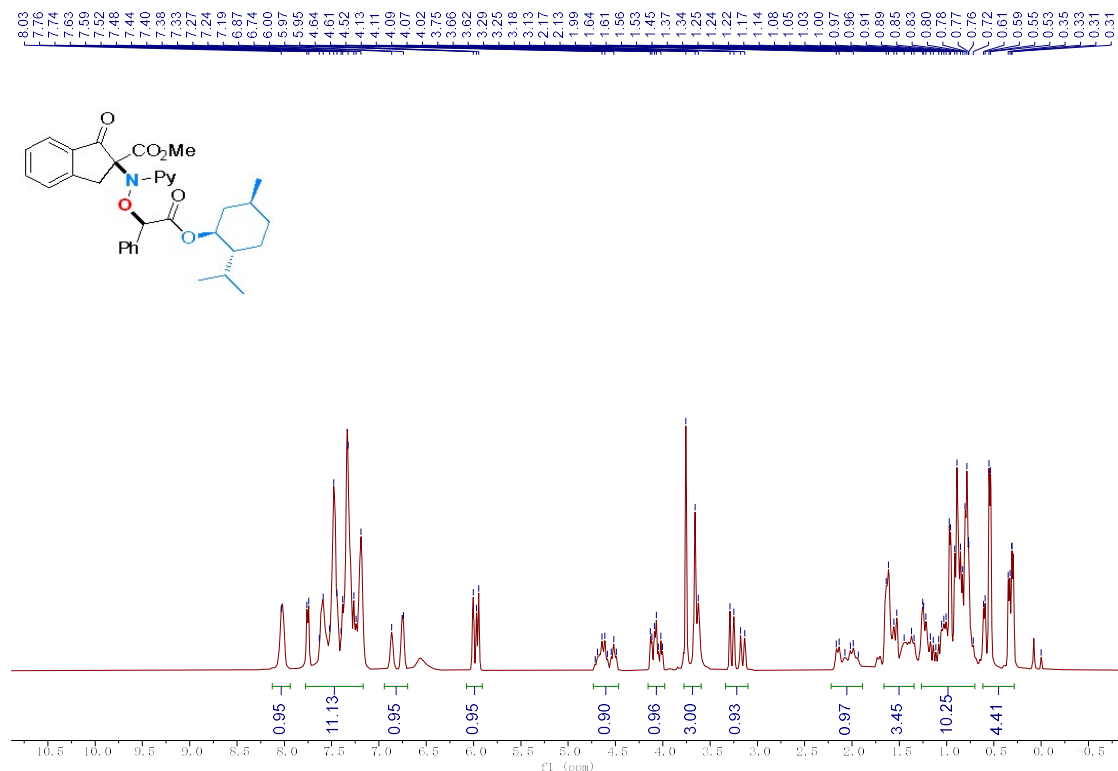
¹H NMR (400 MHz) Spectrum of 22 in CDCl₃



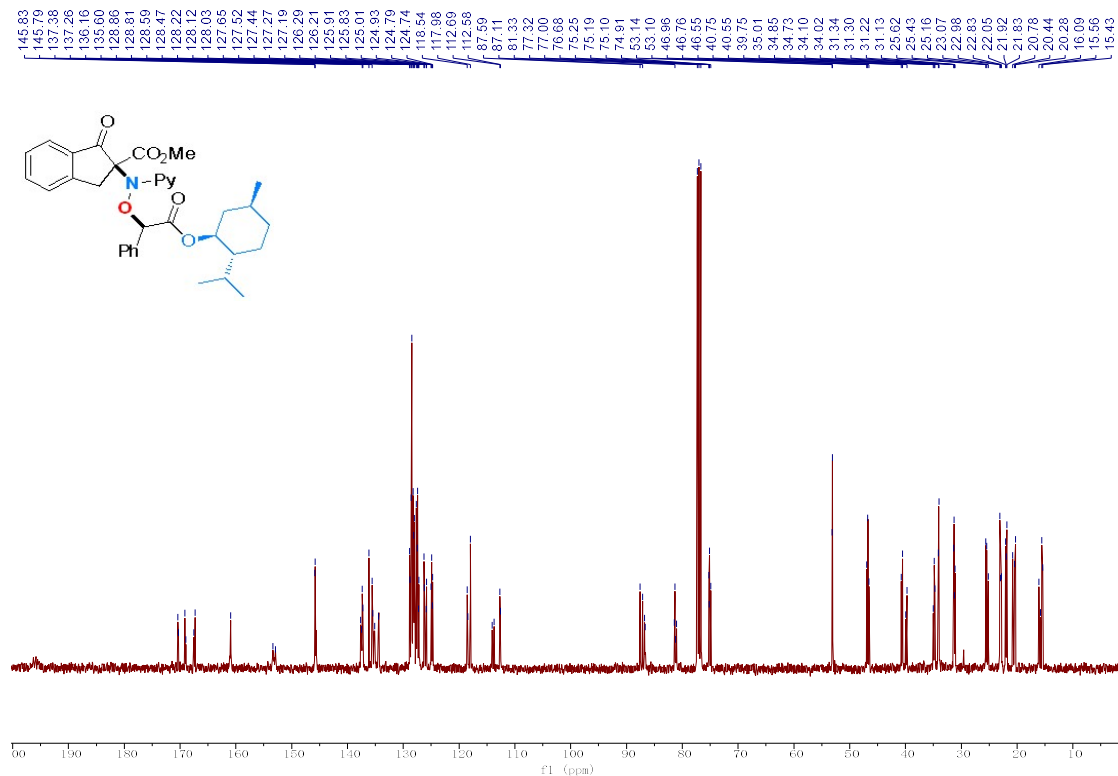
¹³CNMR (100 MHz) Spectrum of 22 in CDCl₃



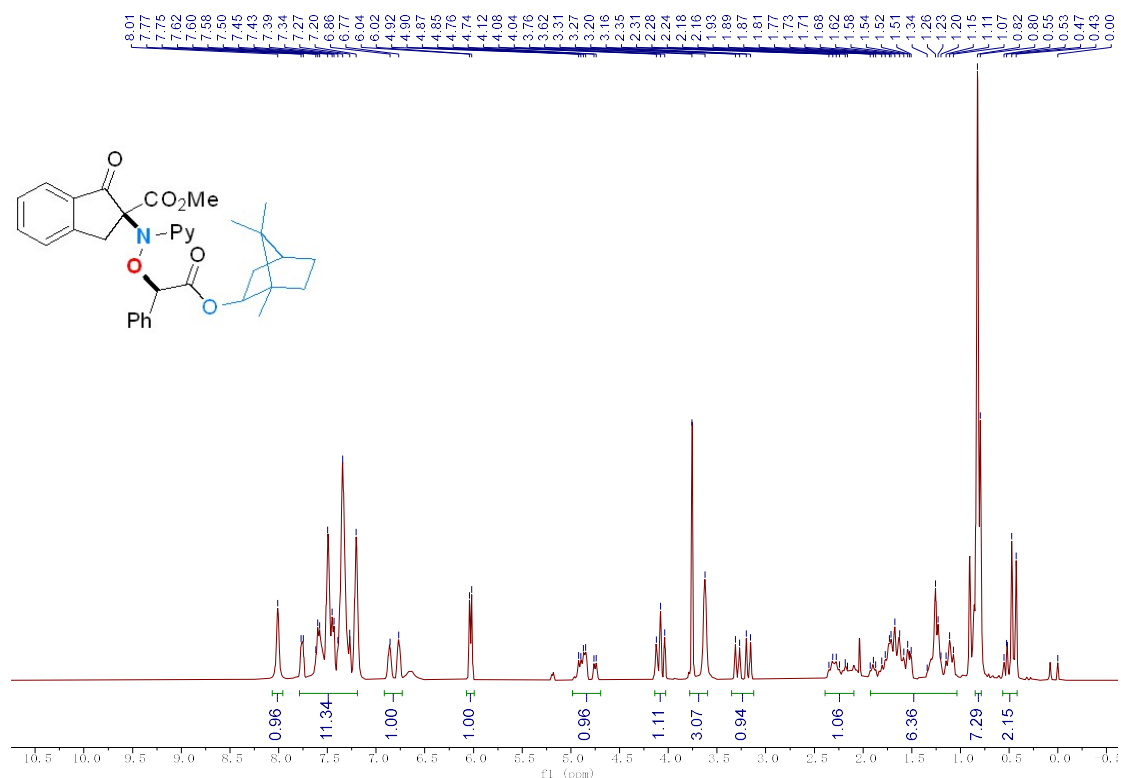
¹H NMR (400 MHz) Spectrum of 23 in CDCl₃



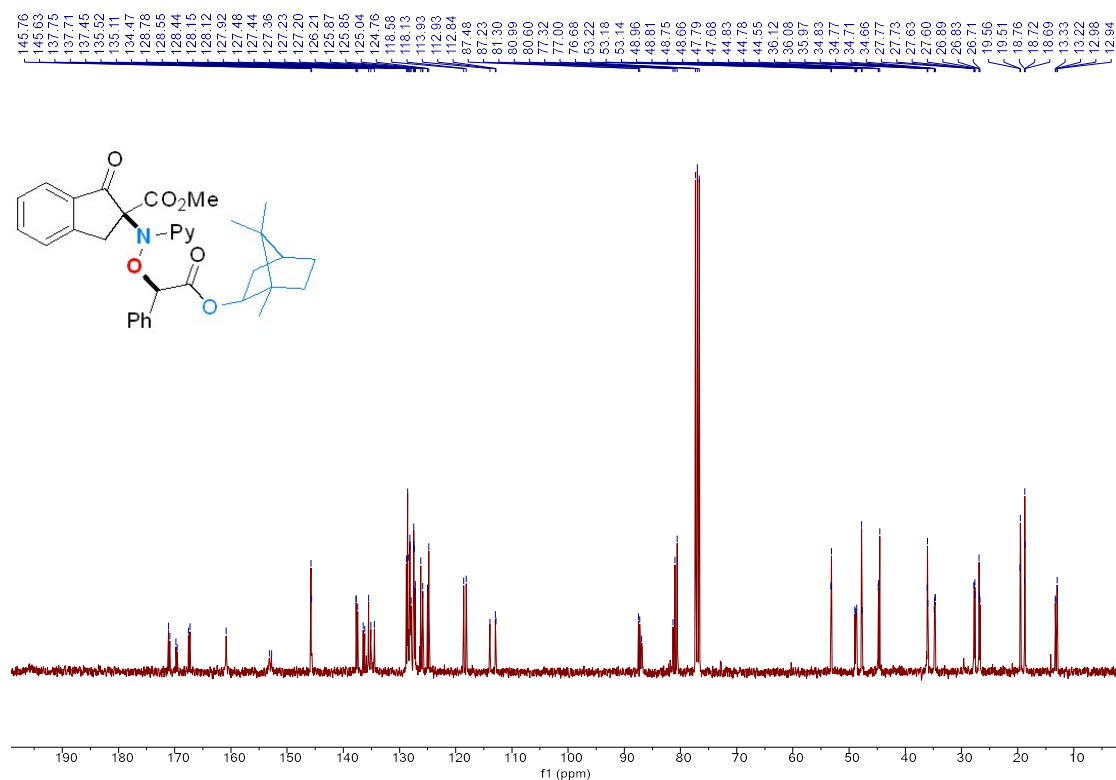
¹³CNMR (100 MHz) Spectrum of 23 in CDCl₃



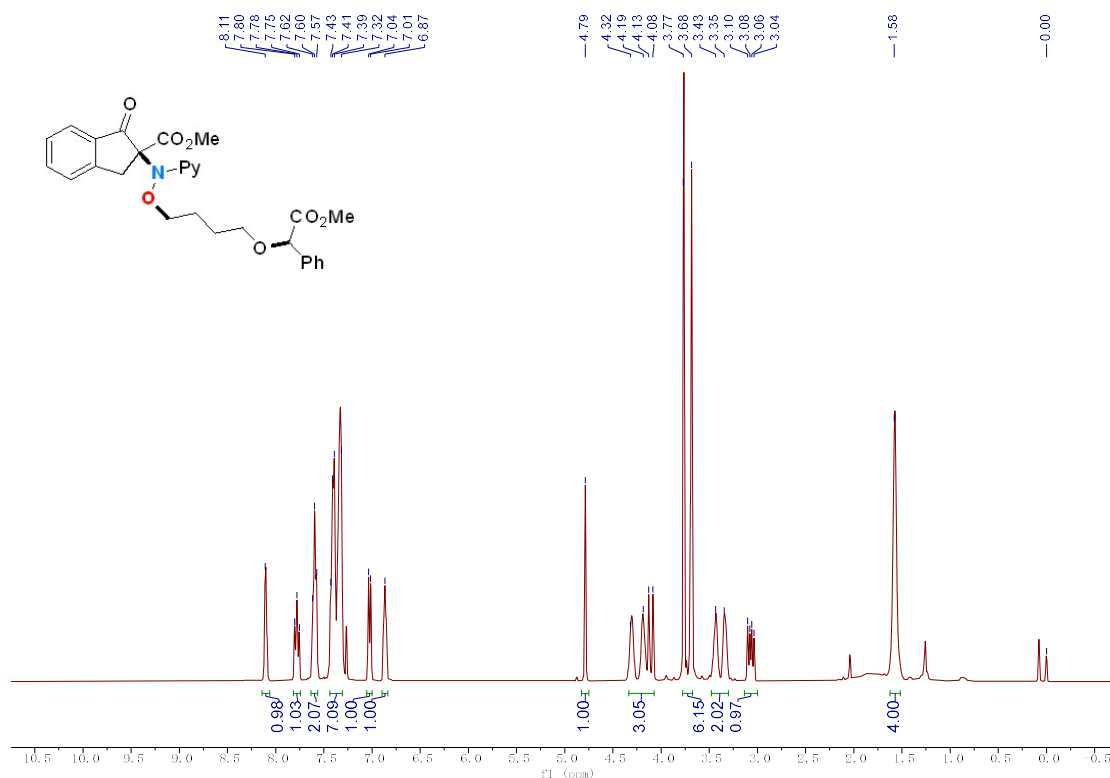
¹H NMR (400 MHz) Spectrum of 24 in CDCl₃



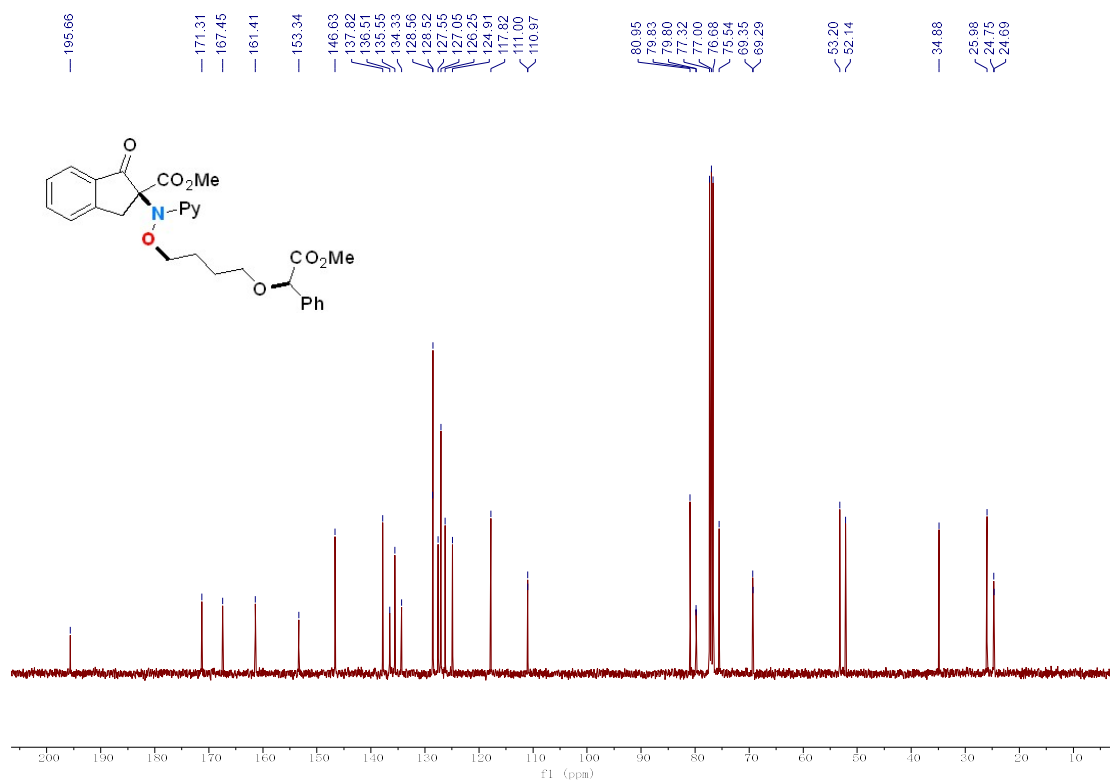
¹³CNMR (100 MHz) Spectrum of 24 in CDCl₃



¹H NMR (400 MHz) Spectrum of 5 in CDCl₃



¹³CNMR (100 MHz) Spectrum of 5 in CDCl₃



[illegible]

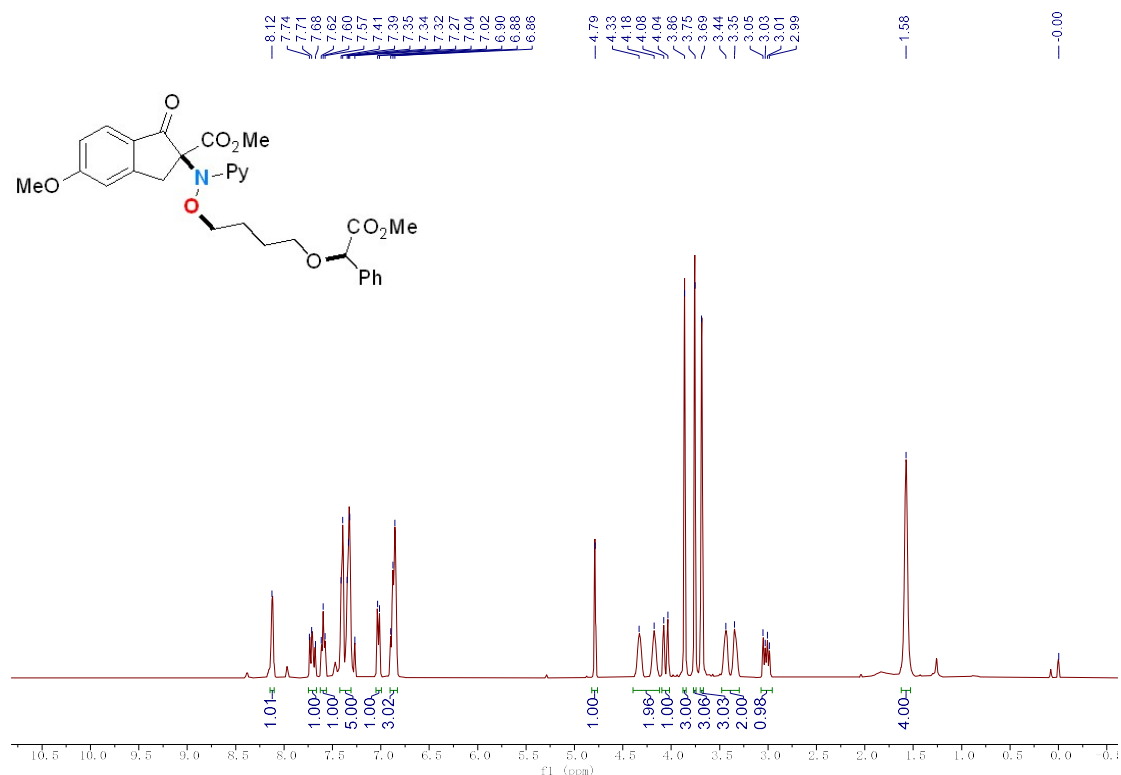
Chemical structure of the compound is shown above the spectrum. The compound is a derivative of a phthalimide, featuring a methyl ester group (CO_2Me), a phenyl group (Ph), and a pyridine ring (Py) attached to the nitrogen atom. The structure also includes a long alkyl chain and an ether linkage.

The ^1H NMR spectrum (400 MHz, CDCl_3) shows the following peaks (ppm):

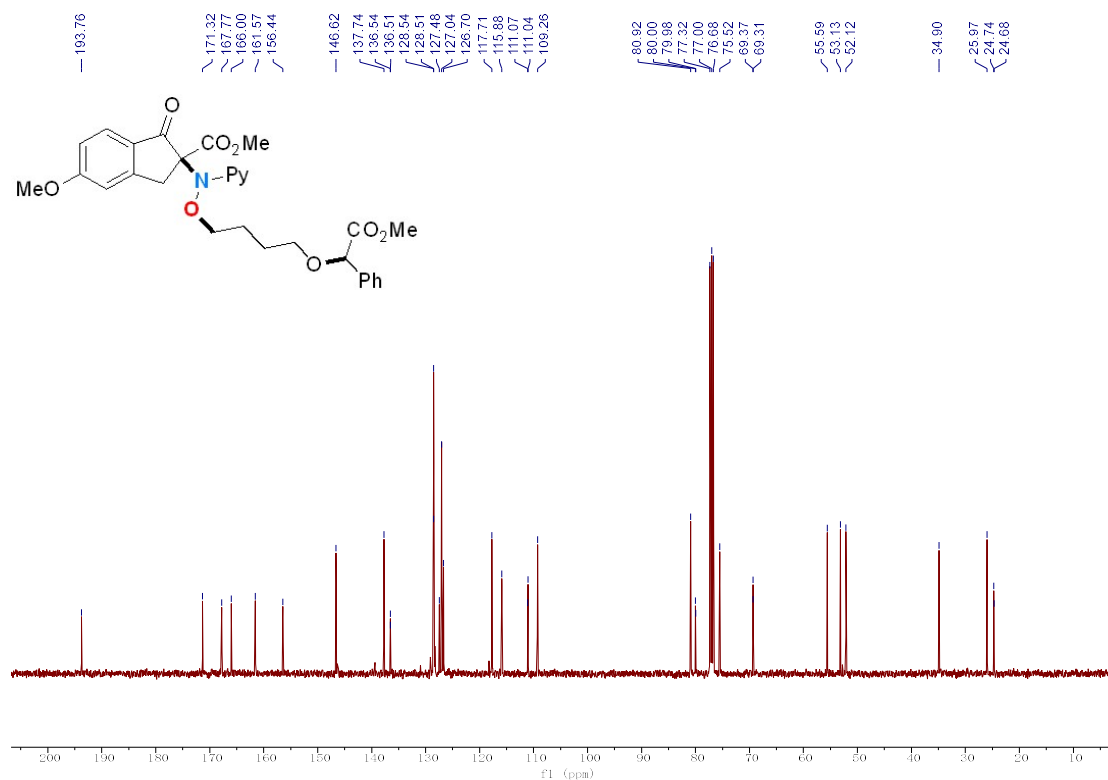
- 105.11
- 171.32
- 167.60
- 161.49
- 153.79
- 147.02
- 146.04
- 137.77
- 135.32
- 133.25
- 128.85
- 128.55
- 128.52
- 127.04
- 126.61
- 124.79
- 117.74
- 111.00
- 110.97
- 80.84
- 80.03
- 80.00
- 77.32
- 77.00
- 76.68
- 73.49
- 69.37
- 69.30
- 53.16
- 52.13
- 34.67
- 25.97
- 24.74
- 24.68
- 22.14

The spectrum displays a complex pattern of peaks, including aromatic signals, carbonyl signals, and aliphatic signals, consistent with the structure of the compound.

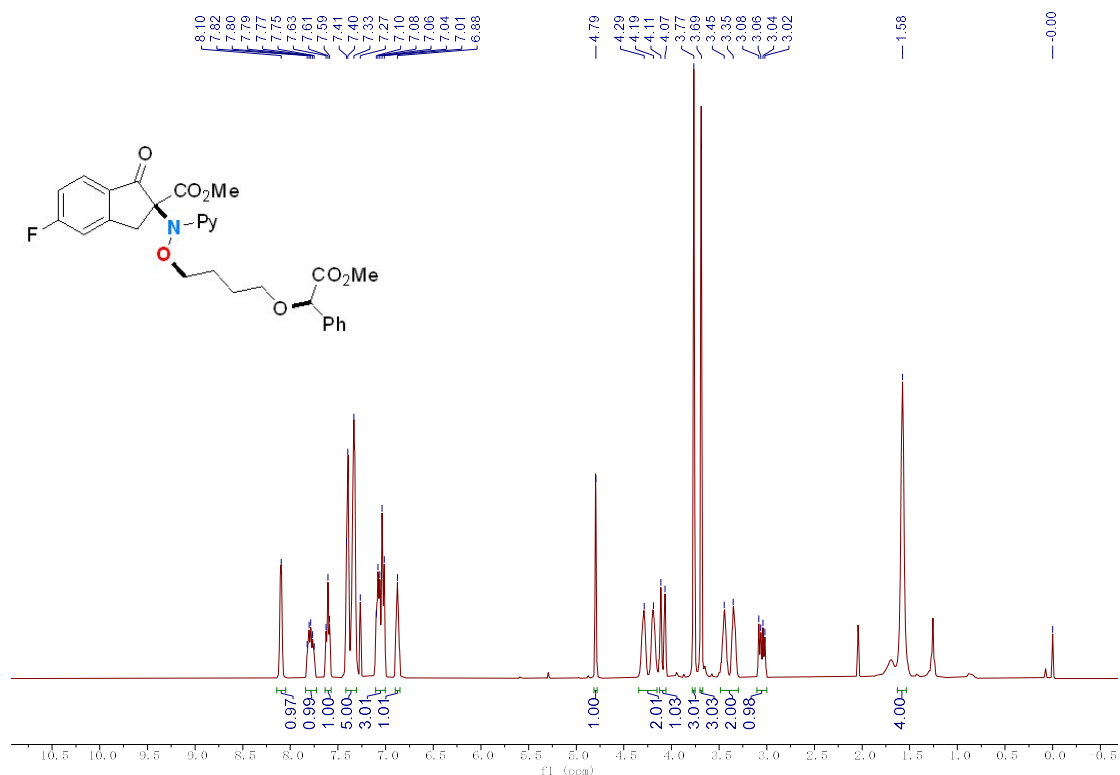
¹H NMR (400 MHz) Spectrum of 26 in CDCl₃



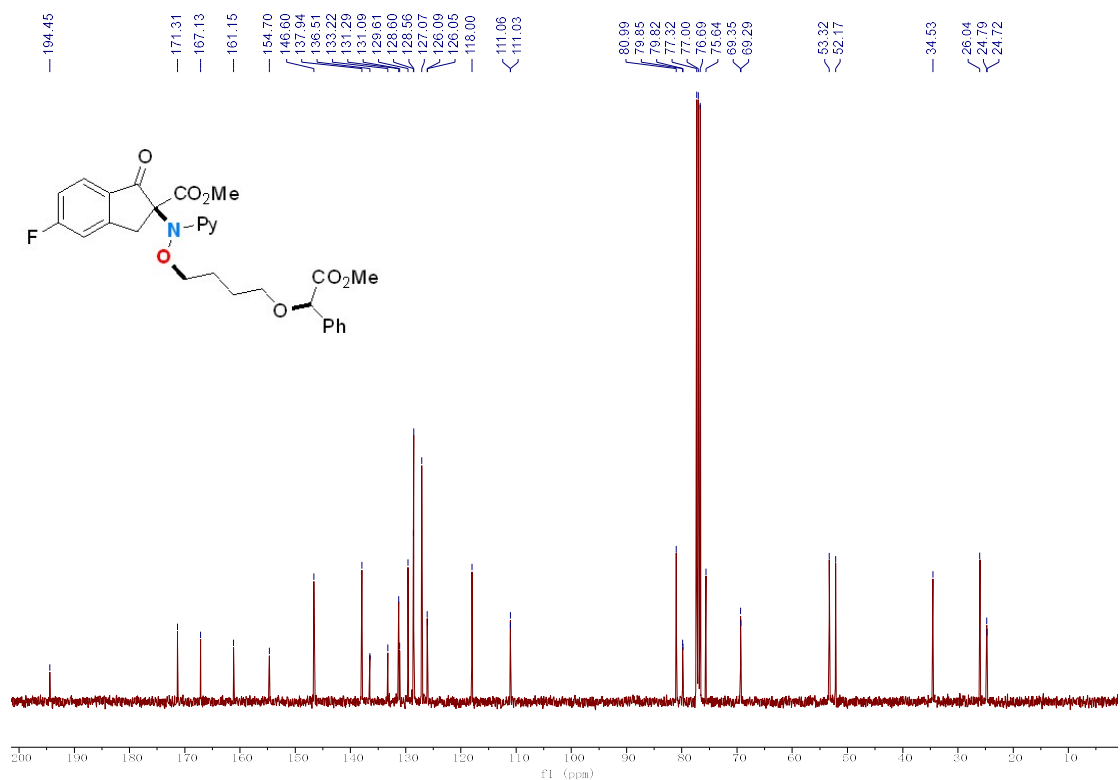
¹³CNMR (100 MHz) Spectrum of 26 in CDCl₃



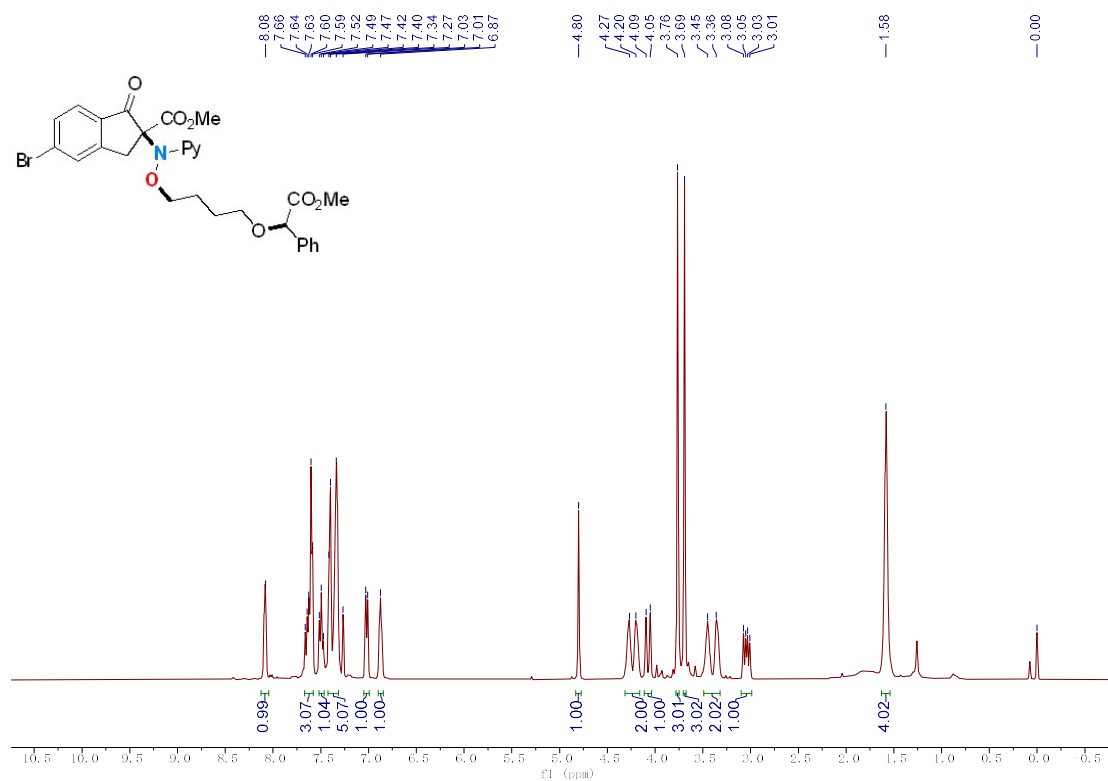
¹H NMR (400 MHz) Spectrum of 27 in CDCl₃



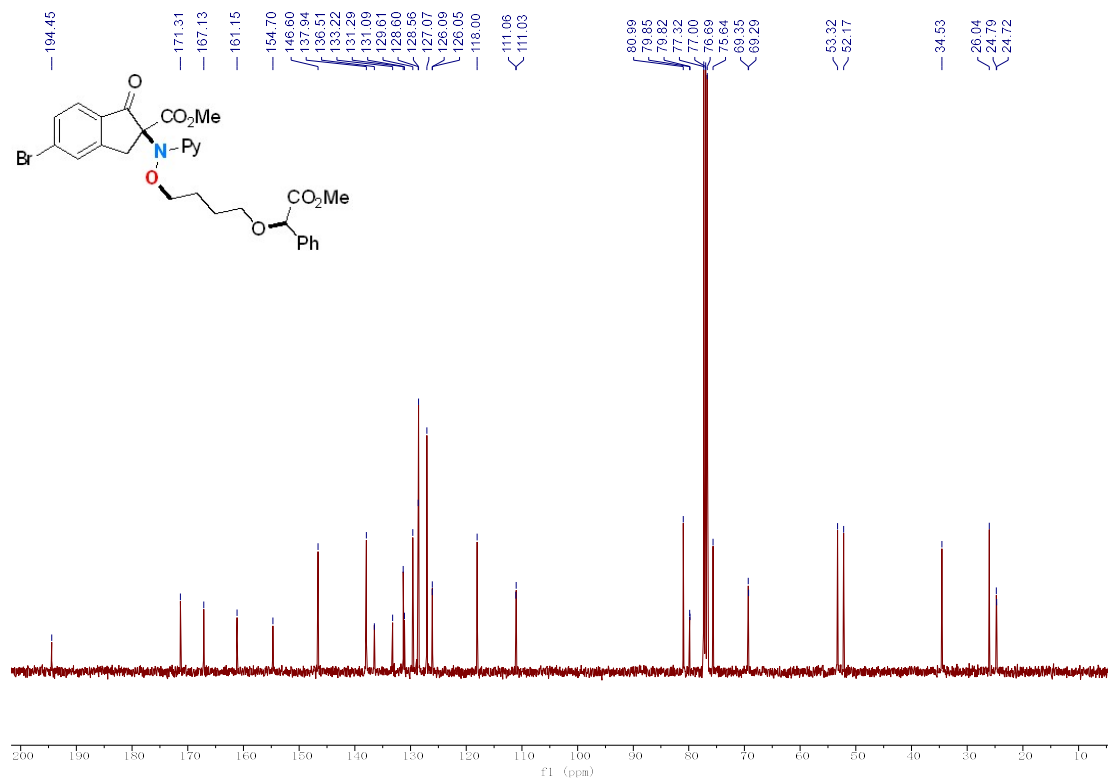
¹³CNMR (100 MHz) Spectrum of 27 in CDCl₃



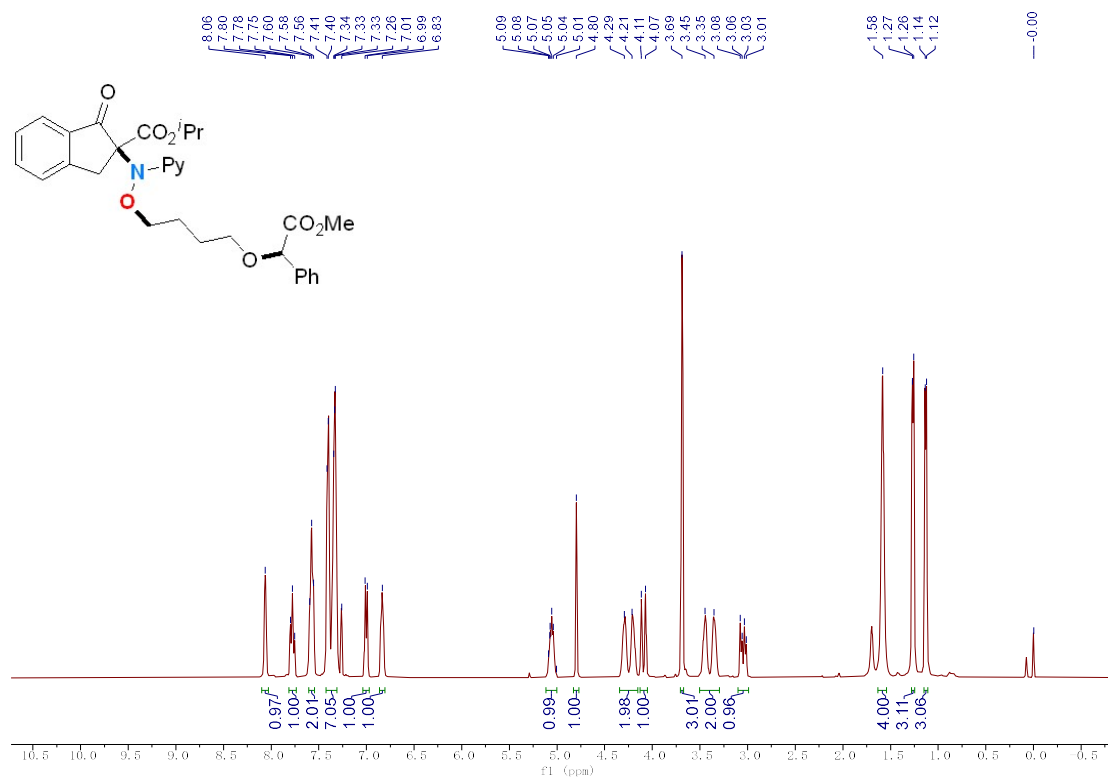
¹H NMR (400 MHz) Spectrum of 28 in CDCl₃



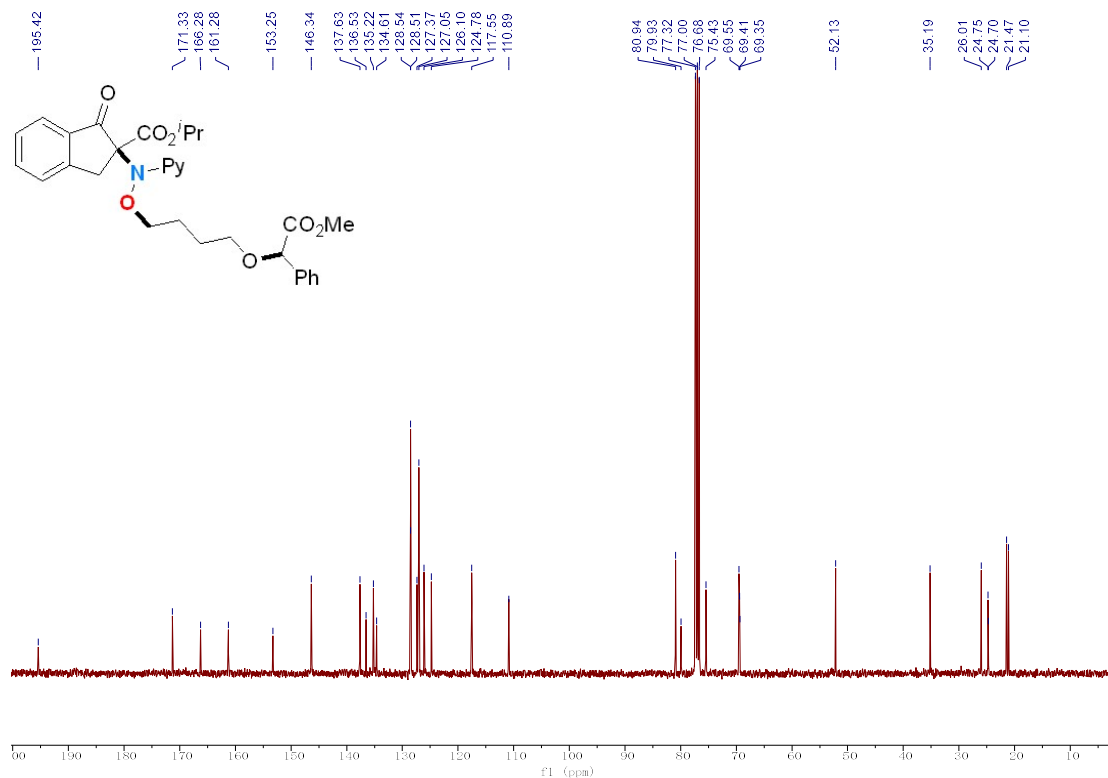
¹³C NMR (100 MHz) Spectrum of 28 in CDCl₃



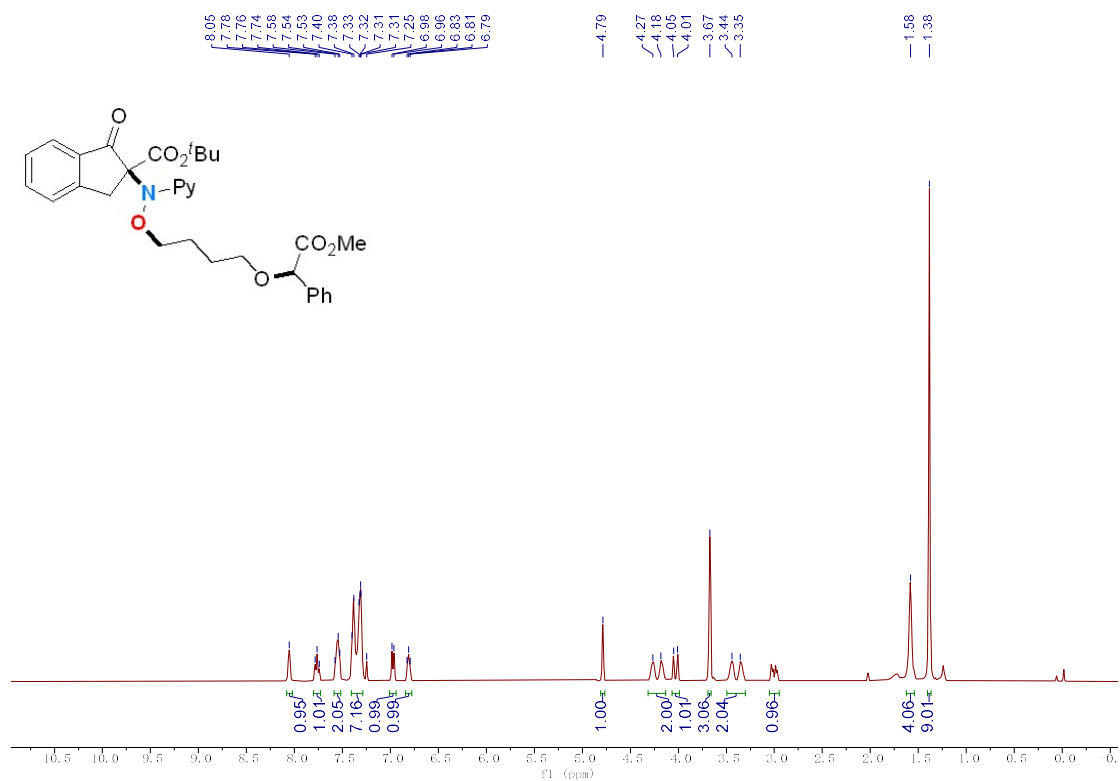
¹H NMR (400 MHz) Spectrum of 29 in CDCl₃



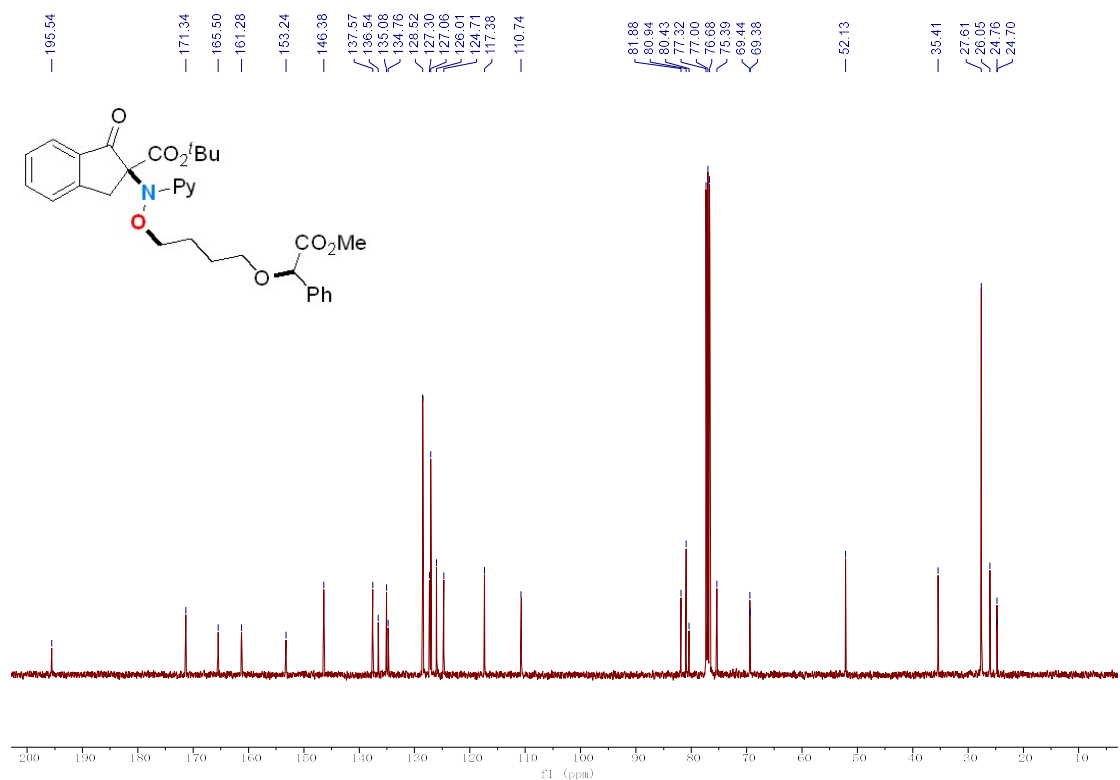
¹³CNMR (100 MHz) Spectrum of 29 in CDCl₃



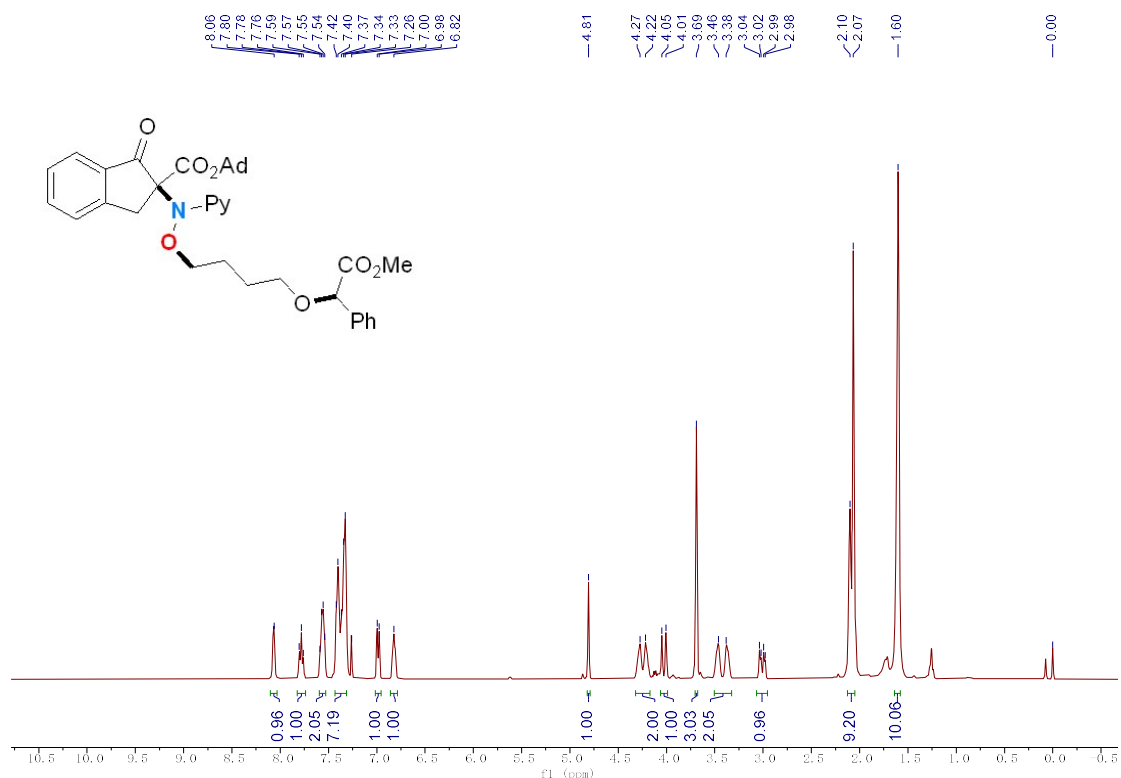
¹H NMR (400 MHz) Spectrum of 30 in CDCl₃



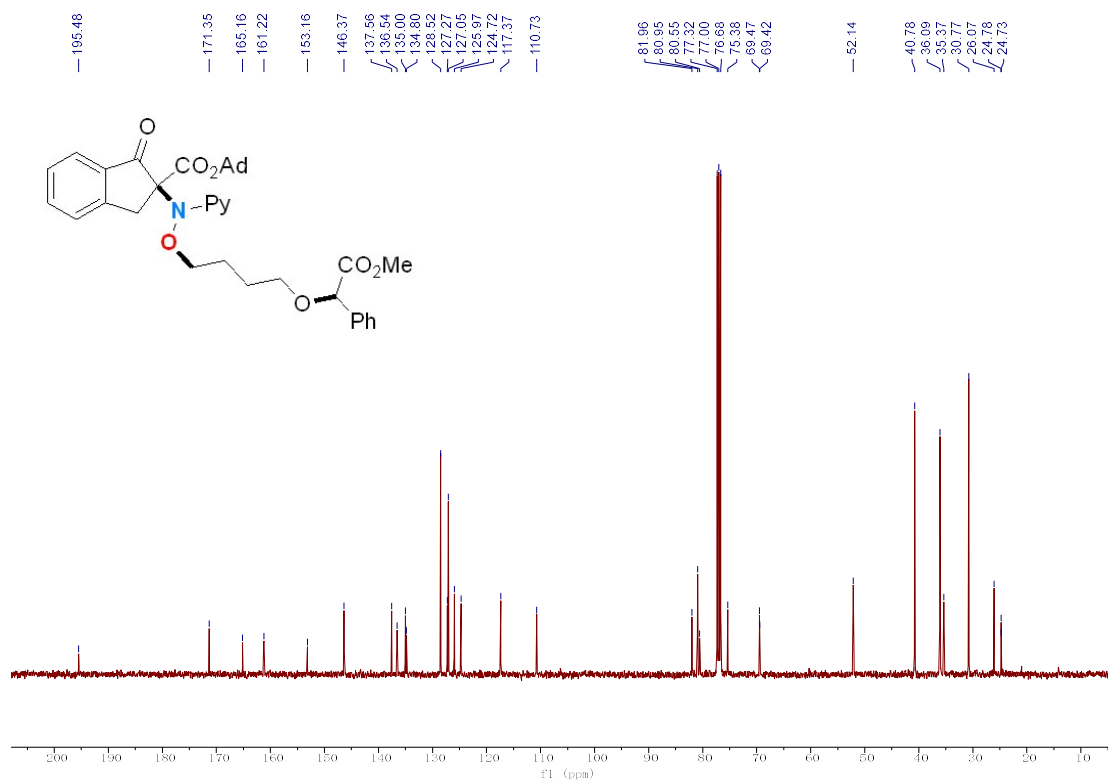
¹³CNMR (100 MHz) Spectrum of 30 in CDCl₃



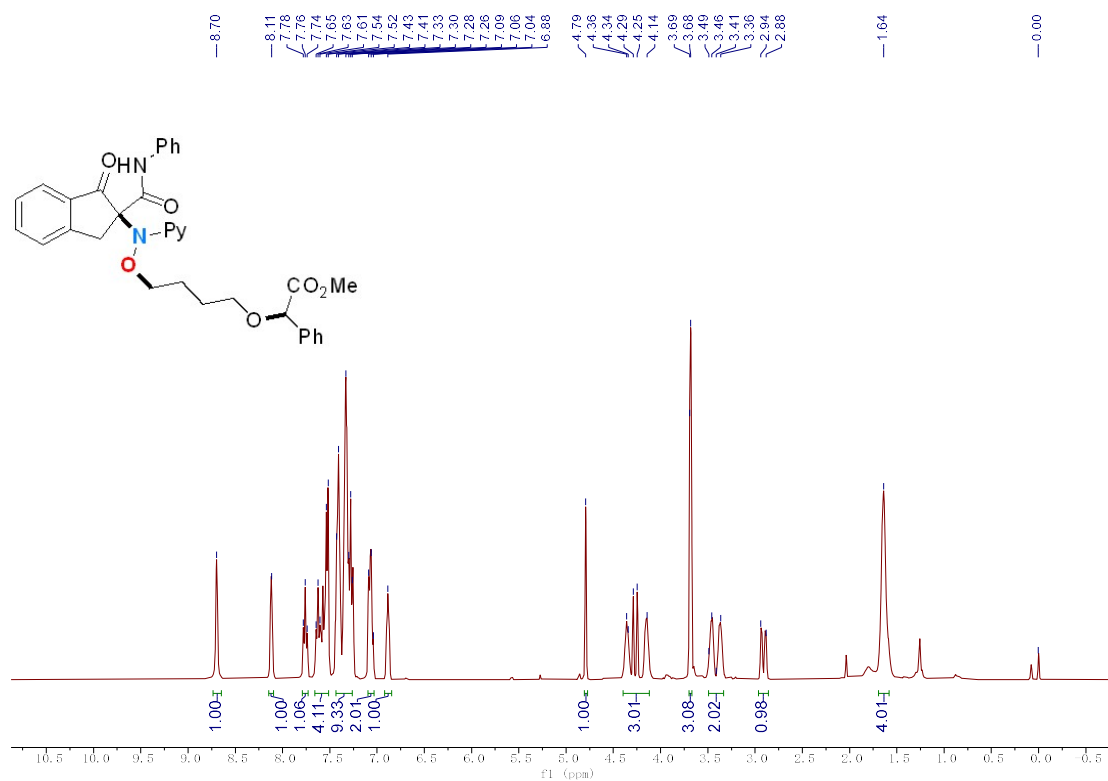
¹H NMR (400 MHz) Spectrum of 31 in CDCl₃



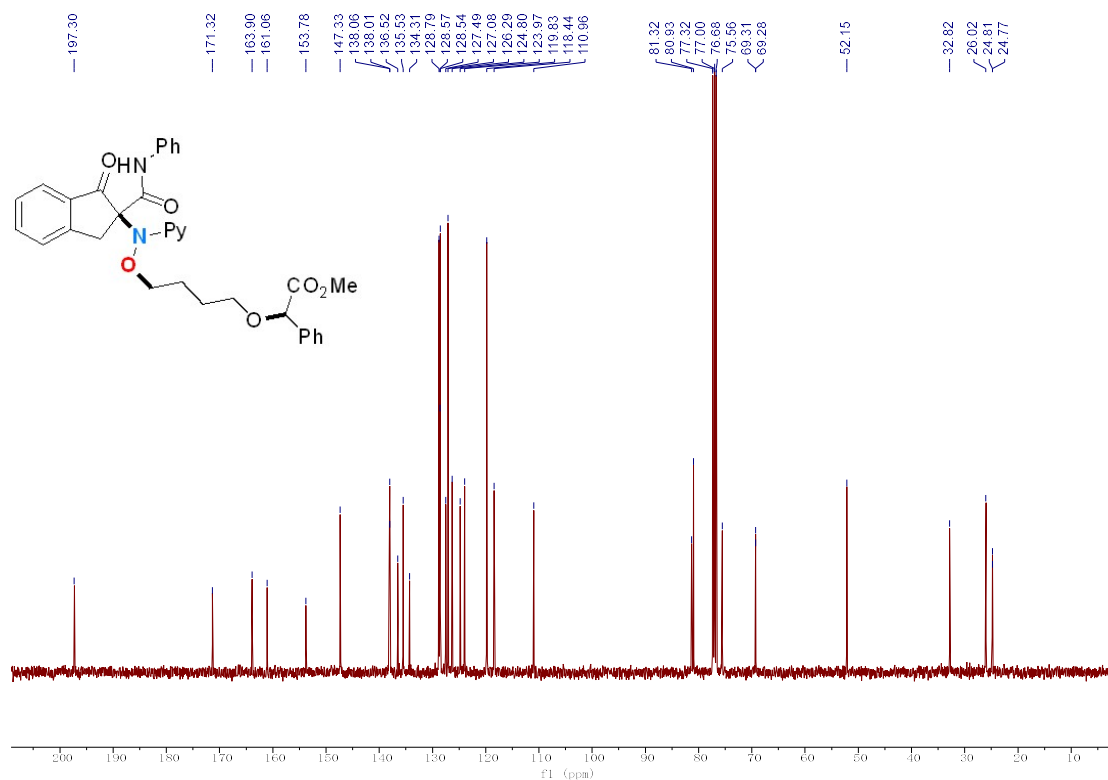
¹³CNMR (100 MHz) Spectrum of 31 in CDCl₃



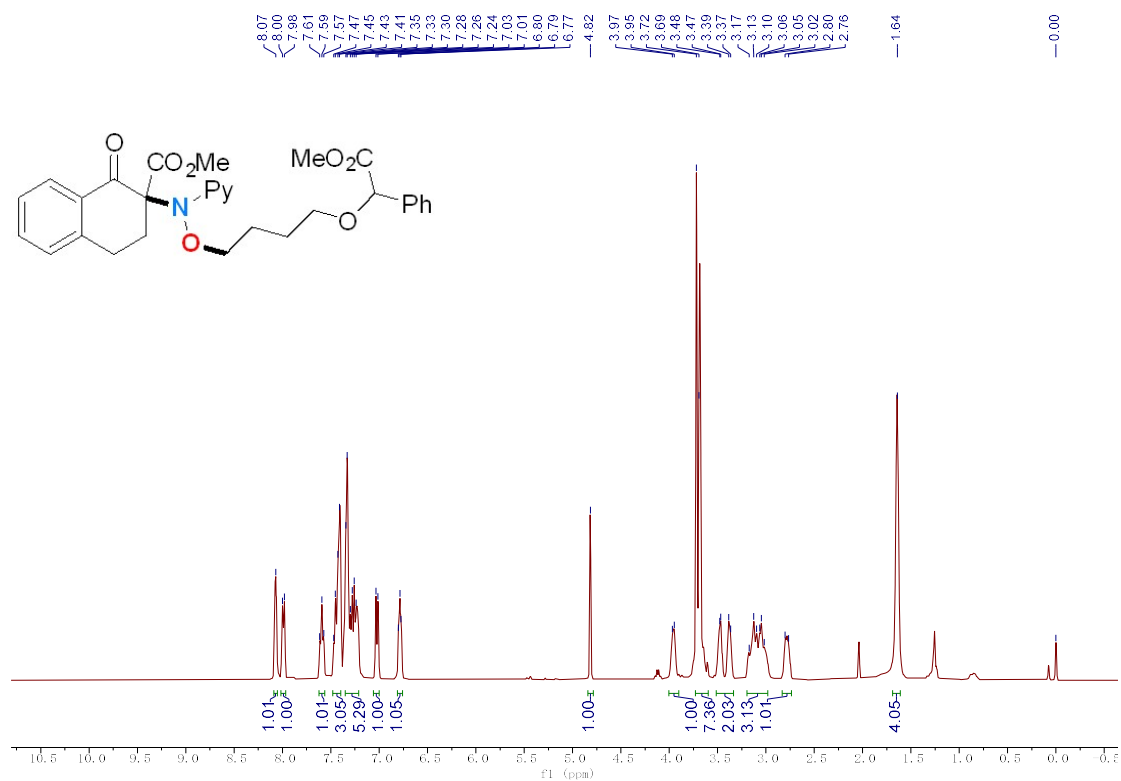
¹H NMR (400 MHz) Spectrum of 32 in CDCl₃



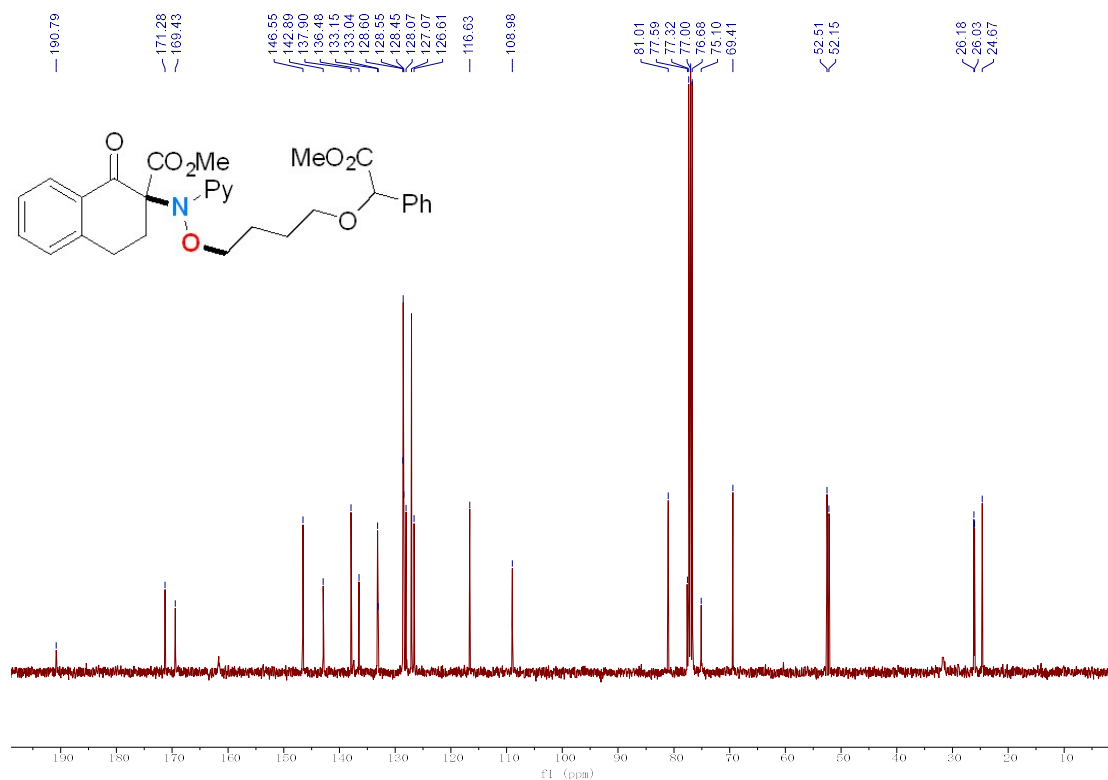
¹³CNMR (100 MHz) Spectrum of 32 in CDCl₃



¹H NMR (400 MHz) Spectrum of 33 in CDCl₃



¹³CNMR (100 MHz) Spectrum of 33 in CDCl₃



Chemical structure of compound 10: COC(=O)C1=CC=C(C=C1)OC(=O)C2=CC=C(C=C2)OC(=O)C3=CC=C(C=C3)OC(=O)C4=CC=C(C=C4)OC(=O)C5=CC=C(C=C5)OC(=O)C6=CC=C(C=C6)OC(=O)C7=CC=C(C=C7)OC(=O)C8=CC=C(C=C8)OC(=O)C9=CC=C(C=C9)OC(=O)C10=CC=C(C=C10)OC(=O)C11=CC=C(C=C11)OC(=O)C12=CC=C(C=C12)OC(=O)C13=CC=C(C=C13)OC(=O)C14=CC=C(C=C14)OC(=O)C15=CC=C(C=C15)OC(=O)C16=CC=C(C=C16)OC(=O)C17=CC=C(C=C17)OC(=O)C18=CC=C(C=C18)OC(=O)C19=CC=C(C=C19)OC(=O)C20=CC=C(C=C20)OC(=O)C21=CC=C(C=C21)OC(=O)C22=CC=C(C=C22)OC(=O)C23=CC=C(C=C23)OC(=O)C24=CC=C(C=C24)OC(=O)C25=CC=C(C=C25)OC(=O)C26=CC=C(C=C26)OC(=O)C27=CC=C(C=C27)OC(=O)C28=CC=C(C=C28)OC(=O)C29=CC=C(C=C29)OC(=O)C30=CC=C(C=C30)OC(=O)C31=CC=C(C=C31)OC(=O)C32=CC=C(C=C32)OC(=O)C33=CC=C(C=C33)OC(=O)C34=CC=C(C=C34)OC(=O)C35=CC=C(C=C35)OC(=O)C36=CC=C(C=C36)OC(=O)C37=CC=C(C=C37)OC(=O)C38=CC=C(C=C38)OC(=O)C39=CC=C(C=C39)OC(=O)C40=CC=C(C=C40)OC(=O)C41=CC=C(C=C41)OC(=O)C42=CC=C(C=C42)OC(=O)C43=CC=C(C=C43)OC(=O)C44=CC=C(C=C44)OC(=O)C45=CC=C(C=C45)OC(=O)C46=CC=C(C=C46)OC(=O)C47=CC=C(C=C47)OC(=O)C48=CC=C(C=C48)OC(=O)C49=CC=C(C=C49)OC(=O)C50=CC=C(C=C50)OC(=O)C51=CC=C(C=C51)OC(=O)C52=CC=C(C=C52)OC(=O)C53=CC=C(C=C53)OC(=O)C54=CC=C(C=C54)OC(=O)C55=CC=C(C=C55)OC(=O)C56=CC=C(C=C56)OC(=O)C57=CC=C(C=C57)OC(=O)C58=CC=C(C=C58)OC(=O)C59=CC=C(C=C59)OC(=O)C60=CC=C(C=C60)OC(=O)C61=CC=C(C=C61)OC(=O)C62=CC=C(C=C62)OC(=O)C63=CC=C(C=C63)OC(=O)C64=CC=C(C=C64)OC(=O)C65=CC=C(C=C65)OC(=O)C66=CC=C(C=C66)OC(=O)C67=CC=C(C=C67)OC(=O)C68=CC=C(C=C68)OC(=O)C69=CC=C(C=C69)OC(=O)C70=CC=C(C=C70)OC(=O)C71=CC=C(C=C71)OC(=O)C72=CC=C(C=C72)OC(=O)C73=CC=C(C=C73)OC(=O)C74=CC=C(C=C74)OC(=O)C75=CC=C(C=C75)OC(=O)C76=CC=C(C=C76)OC(=O)C77=CC=C(C=C77)OC(=O)C78=CC=C(C=C78)OC(=O)C79=CC=C(C=C79)OC(=O)C80=CC=C(C=C80)OC(=O)C81=CC=C(C=C81)OC(=O)C82=CC=C(C=C82)OC(=O)C83=CC=C(C=C83)OC(=O)C84=CC=C(C=C84)OC(=O)C85=CC=C(C=C85)OC(=O)C86=CC=C(C=C86)OC(=O)C87=CC=C(C=C87)OC(=O)C88=CC=C(C=C88)OC(=O)C89=CC=C(C=C89)OC(=O)C90=CC=C(C=C90)OC(=O)C91=CC=C(C=C91)OC(=O)C92=CC=C(C=C92)OC(=O)C93=CC=C(C=C93)OC(=O)C94=CC=C(C=C94)OC(=O)C95=CC=C(C=C95)OC(=O)C96=CC=C(C=C96)OC(=O)C97=CC=C(C=C97)OC(=O)C98=CC=C(C=C98)OC(=O)C99=CC=C(C=C99)OC(=O)C100=CC=C(C=C100)OC(=O)C101=CC=C(C=C101)OC(=O)C102=CC=C(C=C102)OC(=O)C103=CC=C(C=C103)OC(=O)C104=CC=C(C=C104)OC(=O)C105=CC=C(C=C105)OC(=O)C106=CC=C(C=C106)OC(=O)C107=CC=C(C=C107)OC(=O)C108=CC=C(C=C108)OC(=O)C109=CC=C(C=C109)OC(=O)C110=CC=C(C=C110)OC(=O)C111=CC=C(C=C111)OC(=O)C112=CC=C(C=C112)OC(=O)C113=CC=C(C=C113)OC(=O)C114=CC=C(C=C114)OC(=O)C115=CC=C(C=C115)OC(=O)C116=CC=C(C=C116)OC(=O)C117=CC=C(C=C117)OC(=O)C118=CC=C(C=C118)OC(=O)C119=CC=C(C=C119)OC(=O)C120=CC=C(C=C120)OC(=O)C121=CC=C(C=C121)OC(=O)C122=CC=C(C=C122)OC(=O)C123=CC=C(C=C123)OC(=O)C124=CC=C(C=C124)OC(=O)C125=CC=C(C=C125)OC(=O)C126=CC=C(C=C126)OC(=O)C127=CC=C(C=C127)OC(=O)C128=CC=C(C=C128)OC(=O)C129=CC=C(C=C129)OC(=O)C130=CC=C(C=C130)OC(=O)C131=CC=C(C=C131)OC(=O)C132=CC=C(C=C132)OC(=O)C133=CC=C(C=C133)OC(=O)C134=CC=C(C=C134)OC(=O)C135=CC=C(C=C135)OC(=O)C136=CC=C(C=C136)OC(=O)C137=CC=C(C=C137)OC(=O)C138=CC=C(C=C138)OC(=O)C139=CC=C(C=C139)OC(=O)C140=CC=C(C=C140)OC(=O)C141=CC=C(C=C141)OC(=O)C142=CC=C(C=C142)OC(=O)C143=CC=C(C=C143)OC(=O)C144=CC=C(C=C144)OC(=O)C145=CC=C(C=C145)OC(=O)C146=CC=C(C=C146)OC(=O)C147=CC=C(C=C147)OC(=O)C148=CC=C(C=C148)OC(=O)C149=CC=C(C=C149)OC(=O)C150=CC=C(C=C150)OC(=O)C151=CC=C(C=C151)OC(=O)C152=CC=C(C=C152)OC(=O)C153=CC=C(C=C153)OC(=O)C154=CC=C(C=C154)OC(=O)C155=CC=C(C=C155)OC(=O)C156=CC=C(C=C156)OC(=O)C157=CC=C(C=C157)OC(=O)C158=CC=C(C=C158)OC(=O)C159=CC=C(C=C159)OC(=O)C160=CC=C(C=C160)OC(=O)C161=CC=C(C=C161)OC(=O)C162=CC=C(C=C162)OC(=O)C163=CC=C(C=C163)OC(=O)C164=CC=C(C=C164)OC(=O)C165=CC=C(C=C165)OC(=O)C166=CC=C(C=C166)OC(=O)C167=CC=C(C=C167)OC(=O)C168=CC=C(C=C168)OC(=O)C169=CC=C(C=C169)OC(=O)C170=CC=C(C=C170)OC(=O)C171=CC=C(C=C171)OC(=O)C172=CC=C(C=C172)OC(=O)C173=CC=C(C=C173)OC(=O)C174=CC=C(C=C174)OC(=O)C175=CC=C(C=C175)OC(=O)C176=CC=C(C=C176)OC(=O)C177=CC=C(C=C177)OC(=O)C178=CC=C(C=C178)OC(=O)C179=CC=C(C=C179)OC(=O)C180=CC=C(C=C180)OC(=O)C181=CC=C(C=C181)OC(=O)C182=CC=C(C=C182)OC(=O)C183=CC=C(C=C183)OC(=O)C184=CC=C(C=C184)OC(=O)C185=CC=C(C=C185)OC(=O)C186=CC=C(C=C186)OC(=O)C187=CC=C(C=C187)OC(=O)C188=CC=C(C=C188)OC(=O)C189=CC=C(C=C189)OC(=O)C190=CC=C(C=C190)OC(=O)C191=CC=C(C=C191)OC(=O)C192=CC=C(C=C192)OC(=O)C193=CC=C(C=C193)OC(=O)C194=CC=C(C=C194)OC(=O)C195=CC=C(C=C195)OC(=O)C196=CC=C(C=C196)OC(=O)C197=CC=C(C=C197)OC(=O)C198=CC=C(C=C198)OC(=O)C199=CC=C(C=C199)OC(=O)C200=CC=C(C=C200)OC(=O)C201=CC=C(C=C201)OC(=O)C202=CC=C(C=C202)OC(=O)C203=CC=C(C=C203)OC(=O)C204=CC=C(C=C204)OC(=O)C205=CC=C(C=C205)OC(=O)C206=CC=C(C=C206)OC(=O)C207=CC=C(C=C207)OC(=O)C208=CC=C(C=C208)OC(=O)C209=CC=C(C=C209)OC(=O)C210=CC=C(C=C210)OC(=O)C211=CC=C(C=C211)OC(=O)C212=CC=C(C=C212)OC(=O)C213=CC=C(C=C213)OC(=O)C214=CC=C(C=C214)OC(=O)C215=CC=C(C=C215)OC(=O)C216=CC=C(C=C216)OC(=O)C217=CC=C(C=C217)OC(=O)C218=CC=C(C=C218)OC(=O)C219=CC=C(C=C219)OC(=O)C220=CC=C(C=C220)OC(=O)C221=CC=C(C=C221)OC(=O)C222=CC=C(C=C222)OC(=O)C223=CC=C(C=C223)OC(=O)C224=CC=C(C=C224)OC(=O)C225=CC=C(C=C225)OC(=O)C226=CC=C(C=C226)OC(=O)C227=CC=C(C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Chemical structure of compound 10 is shown above the spectrum. The structure is a 1-(4-methylbenzoyloxy)-4-(2-(pyridin-2-yl)-2-oxo-1,2,3,4-tetrahydro-1H-inden-1-yl)butan-1-ol derivative.

¹H NMR spectrum (CDCl₃) of compound 10. The x-axis represents the chemical shift in ppm, ranging from 0 to 10. The spectrum shows several peaks corresponding to the structure, with the following chemical shifts (ppm) labeled above the peaks:

- 195.66
- 171.46
- 167.46
- 161.42
- 153.34
- 146.62
- 136.63
- 135.63
- 135.52
- 134.34
- 133.55
- 129.23
- 127.55
- 127.06
- 126.26
- 124.92
- 117.82
- 111.03
- 110.99
- 80.80
- 79.85
- 79.81
- 77.32
- 77.00
- 76.68
- 75.57
- 69.22
- 69.16
- 53.21
- 52.10
- 34.88
- 26.00
- 24.77
- 24.70
- 21.14

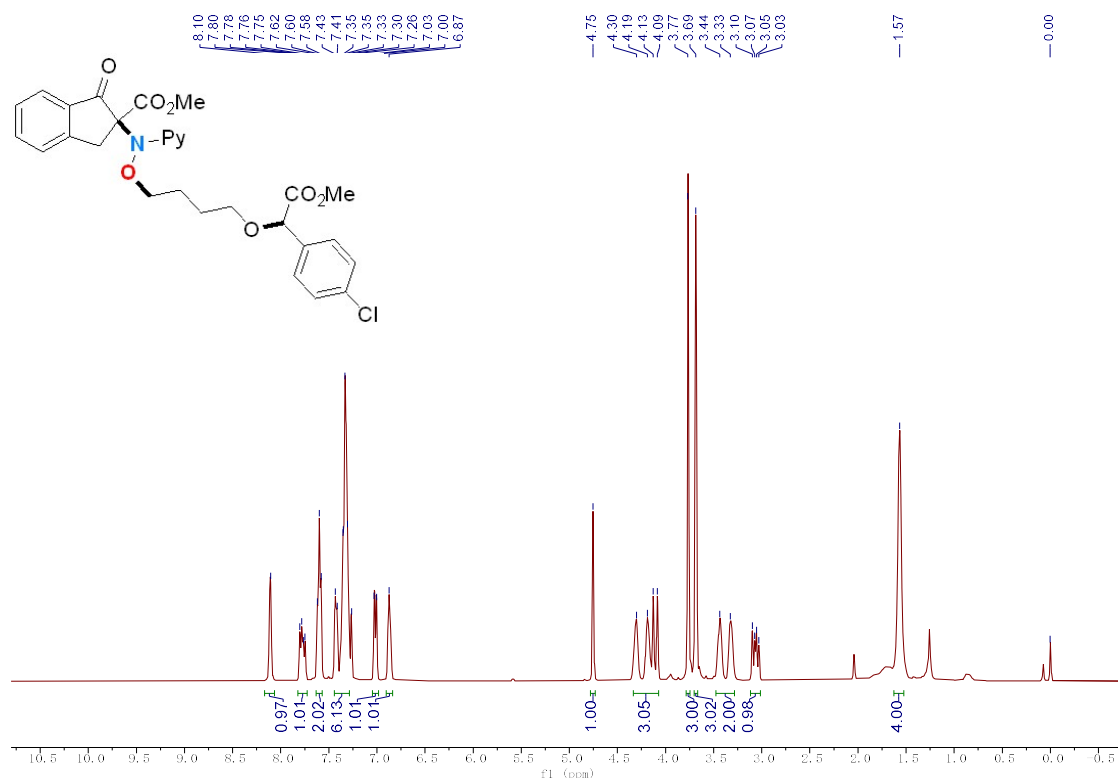
[illegible]

Chemical structure of compound 10 is shown above the spectrum. The structure is a 1-(4-fluorophenyl)-4-methoxycarbonyl-1H-pyrazole-5-carboxamide derivative.

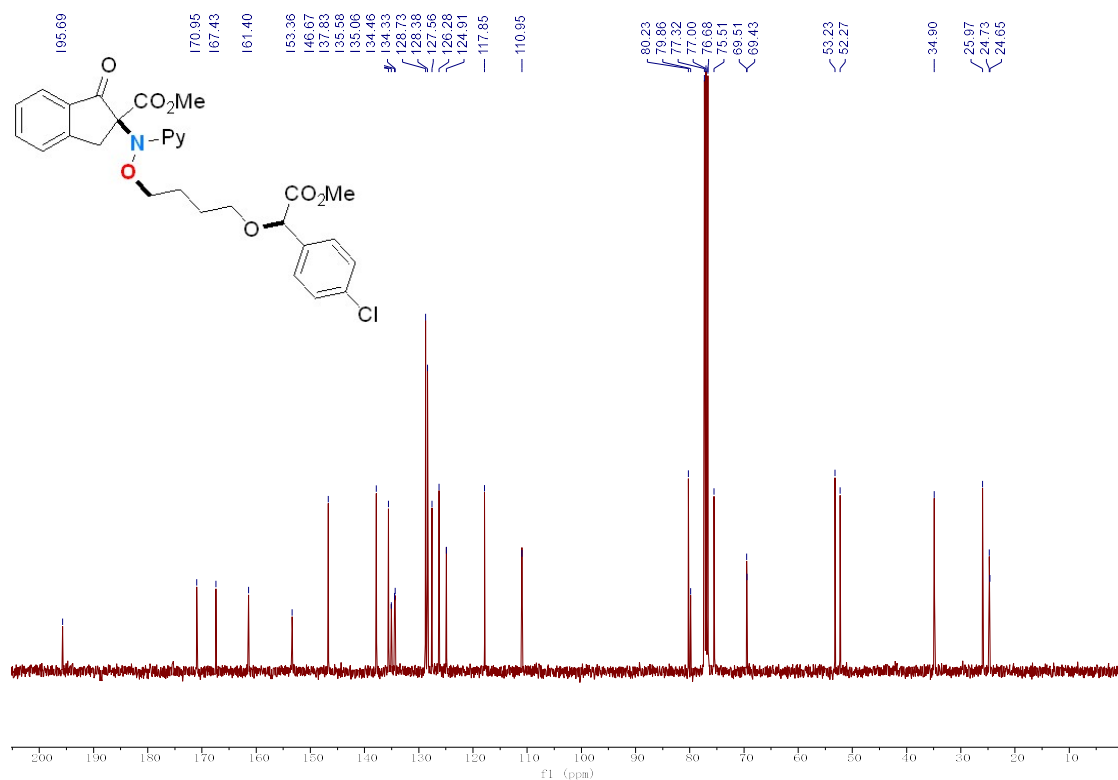
¹H NMR spectrum (CDCl₃) of compound 10. The x-axis represents the chemical shift in ppm (f1), ranging from 200 to 10. The spectrum shows several peaks, with the following chemical shifts (ppm) listed on the right:

- 195.68
- 171.17
- 167.43
- 164.03
- 161.57
- 161.40
- 153.33
- 146.66
- 137.82
- 135.57
- 134.33
- 132.38
- 128.85
- 128.76
- 127.56
- 126.27
- 124.91
- 117.84
- 115.58
- 115.36
- 110.97
- 110.94
- 80.22
- 79.85
- 77.32
- 77.00
- 76.68
- 75.51
- 69.41
- 69.35
- 53.22
- 52.21
- 34.89
- 25.96
- 24.74
- 24.67

¹H NMR (400 MHz) Spectrum of 36 in CDCl₃



¹³CNMR (100 MHz) Spectrum of 36 in CDCl₃



Chemical structure of compound 10 is shown above the spectrum. The spectrum displays peaks from 0.00 to 8.12 ppm. Key peaks include a singlet at 0.00 ppm (3H, Me), a doublet at 1.57 ppm (3H, Me), a multiplet at 3.00-3.45 ppm (4H, OCH₂), a multiplet at 4.00-4.18 ppm (2H, CH₂), a multiplet at 7.00-7.80 ppm (10H, aromatic), and a multiplet at 7.20-7.40 ppm (4H, aromatic). Integration values are provided below the baseline.

Chemical structure of compound 10 is shown above the spectrum. The spectrum displays peaks corresponding to the chemical shifts of the compound, with the following labeled values (ppm):

- 195.68
- 170.87
- 167.43
- 161.40
- 153.37
- 146.67
- 137.83
- 135.58
- 134.33
- 131.06
- 129.59
- 127.78
- 126.20
- 124.91
- 124.83
- 122.64
- 117.85
- 110.95
- 80.27
- 79.85
- 79.83
- 77.32
- 77.00
- 76.68
- 75.90
- 73.90
- 69.32
- 69.45
- 53.22
- 52.28
- 25.96
- 24.73
- 24.64

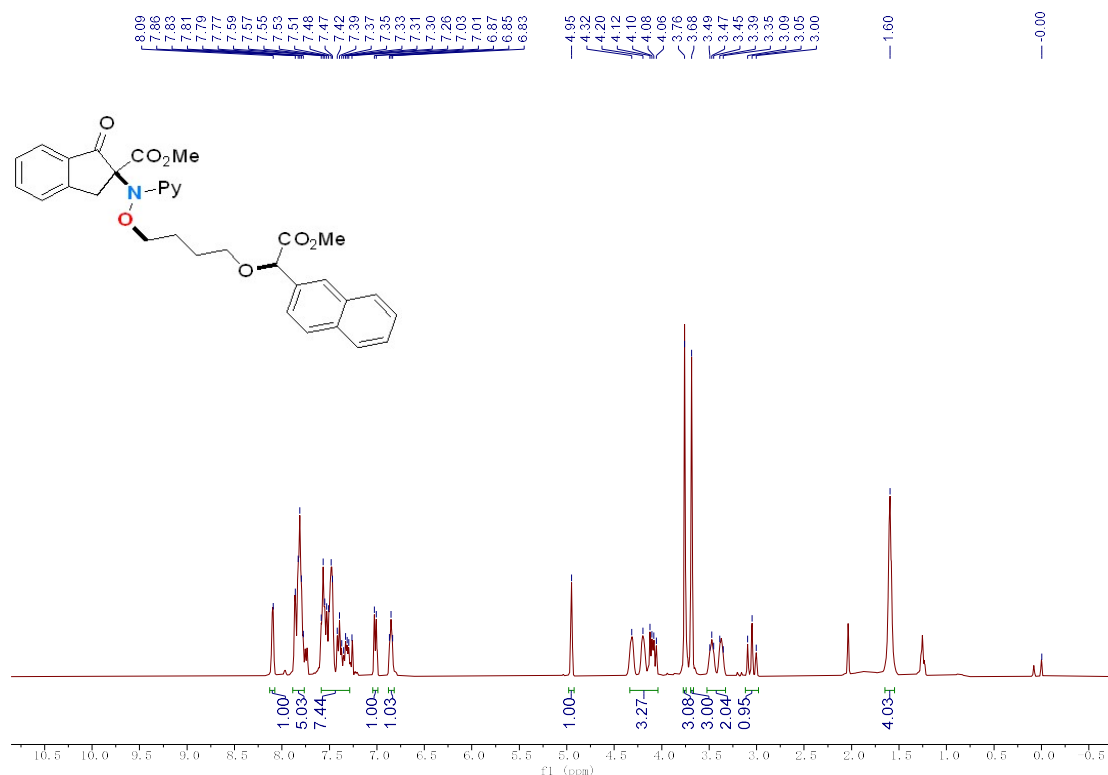
[illegible]

Chemical structure of the compound is shown above the spectrum. The structure is a complex molecule featuring a benzene ring, a carbonyl group, a pyridine ring, and a trifluoromethyl group.

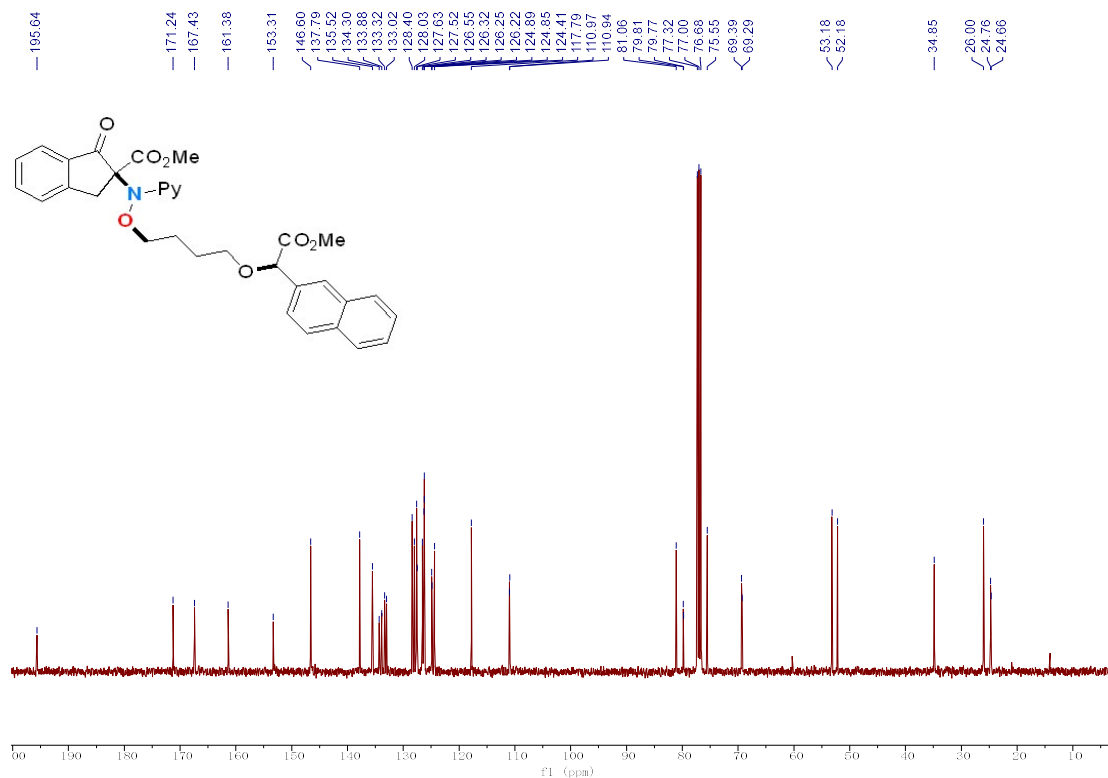
The spectrum displays chemical shifts (ppm) on the x-axis, ranging from 0 to 200. Key peaks are labeled with their corresponding chemical shifts (ppm):

- 195.71
- 170.67
- 167.43
- 161.41
- 153.39
- 146.70
- 140.47
- 137.83
- 135.60
- 134.34
- 127.57
- 127.30
- 126.29
- 125.50
- 125.46
- 124.91
- 117.87
- 110.93
- 80.35
- 79.88
- 77.32
- 77.00
- 76.68
- 75.50
- 69.75
- 69.65
- 53.23
- 52.38
- 34.91
- 25.98
- 24.73
- 24.63

¹H NMR (400 MHz) Spectrum of 39 in CDCl₃



¹³C NMR (100 MHz) Spectrum of 39 in CDCl₃



CCOC(=O)[C@H](N1CCCCC1)C(=O)c2ccccc2

¹H NMR spectrum (CDCl₃) of methyl 1-(1-((S)-4-oxo-4-phenylbutyrate)pyrrolidin-2-yl)indan-1-one-3-carboxylate. The spectrum displays peaks from 0 to 8 ppm, with integrations and a chemical structure of the compound.

Chemical Shift (ppm)	Integration
~7.80	0.98H
~7.75	1.01H
~7.61	2.05H
~7.57	7.02H
~7.43	1.00H
~7.39	1.00H
~7.35	1.00H
~4.77	1.00H
~4.30	5.05H
~4.13	3.00H
~4.07	2.00H
~3.76	0.95H
~3.45	6.05H
~3.35	6.23H
~3.08	3.08H
~1.61	-
~1.57	-
~1.55	-
~1.20	-
~0.84	-
~0.00	-

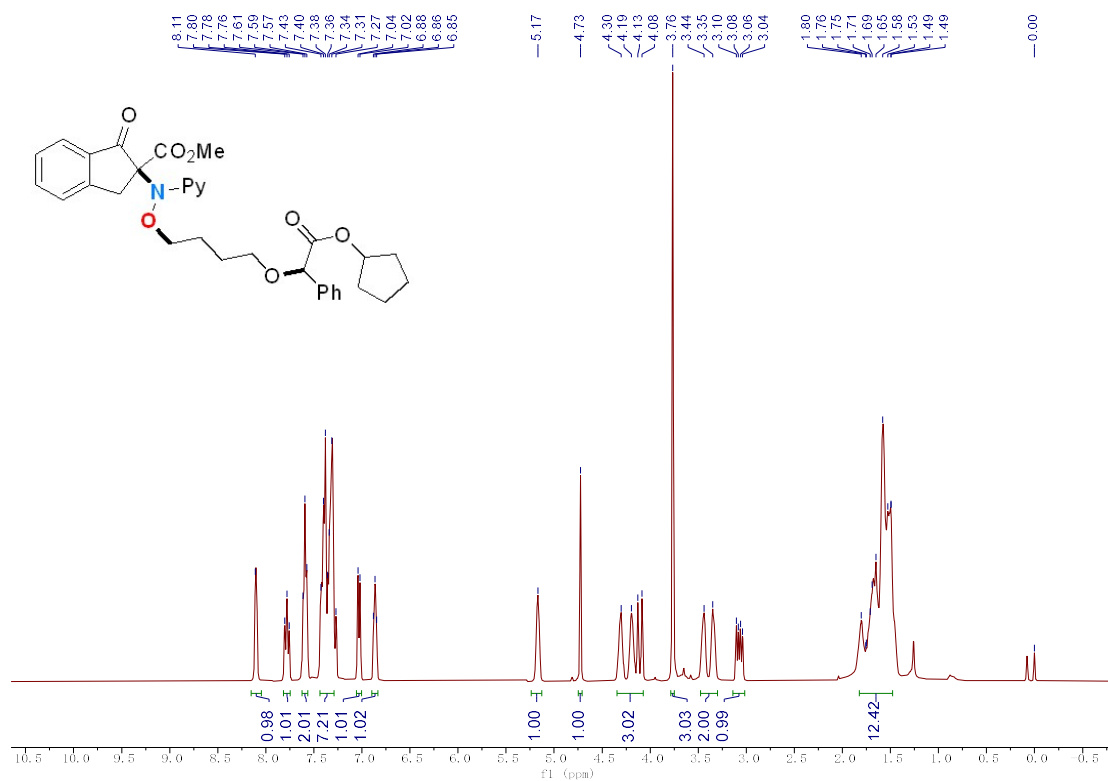
Chemical structure of the compound is shown above the spectrum. The structure is a derivative of a phthalimide, featuring a phthalimide core with a methyl ester group (CO_2Me) and a pyridine ring (Py) attached to the nitrogen. The phthalimide is linked via an ether bridge to a 4-phenylbutyrate ester group.

The ^1H NMR spectrum (400 MHz, CDCl_3) shows the following peaks (ppm):

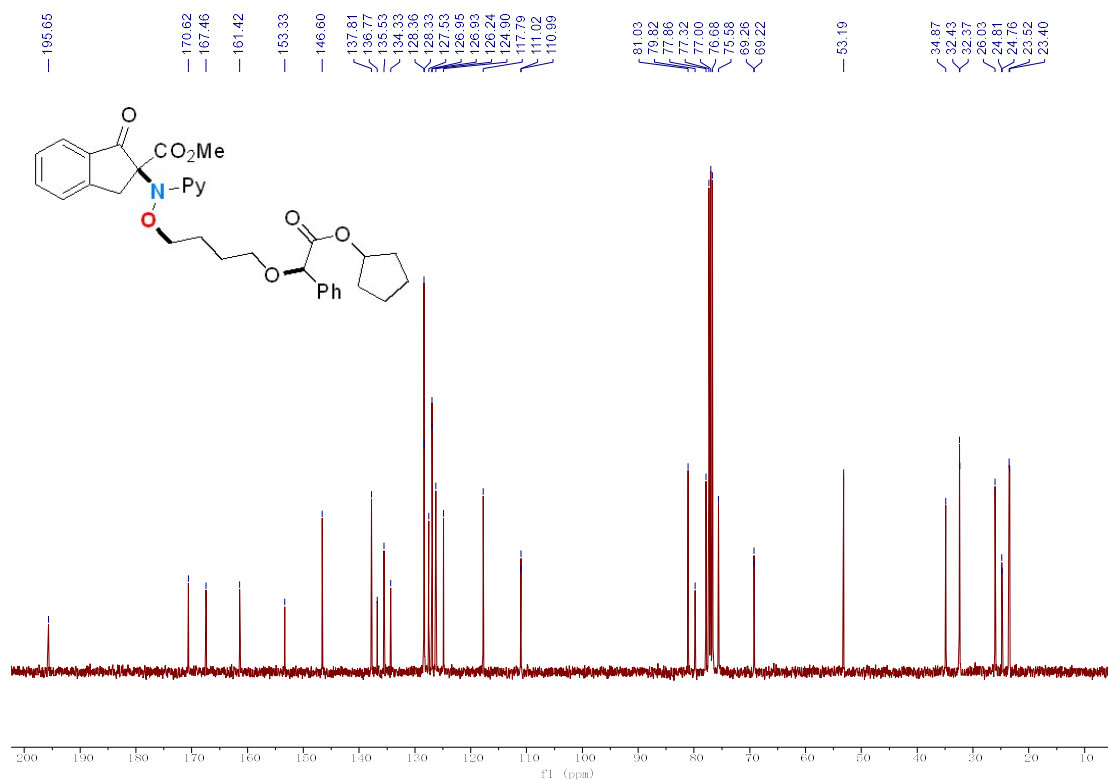
- 195.66
- 170.95
- 167.47
- 161.43
- 153.34
- 146.62
- 137.92
- 136.14
- 135.54
- 134.34
- 128.44
- 127.55
- 127.04
- 127.02
- 126.25
- 124.92
- 117.81
- 111.02
- 110.99
- 81.02
- 79.93
- 77.32
- 77.00
- 76.66
- 73.96
- 69.51
- 69.27
- 65.12
- 53.21
- 34.88
- 31.19
- 28.35
- 26.03
- 25.26
- 24.79
- 24.74
- 22.39
- 13.88

The spectrum displays a complex pattern of peaks, including aromatic signals between 110-140 ppm, a carbonyl region around 170 ppm, and aliphatic signals between 10-40 ppm. A prominent peak is observed at 77.00 ppm, characteristic of the solvent CDCl_3 .

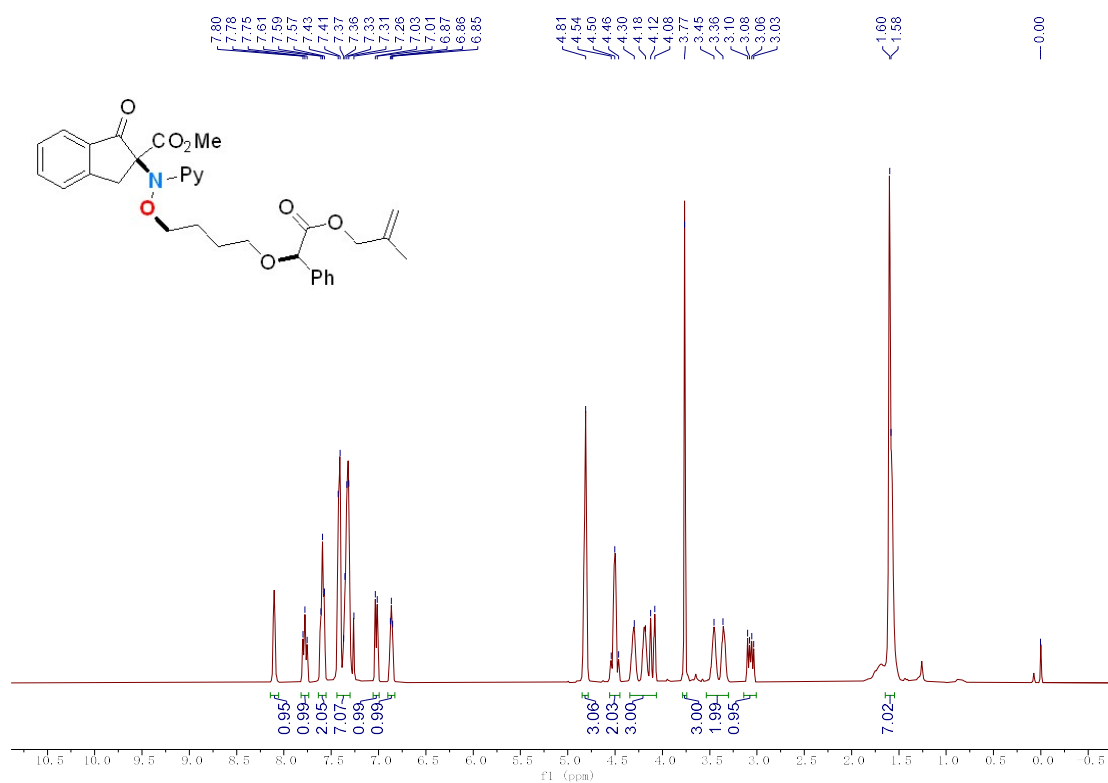
¹H NMR (400 MHz) Spectrum of 41 in CDCl₃



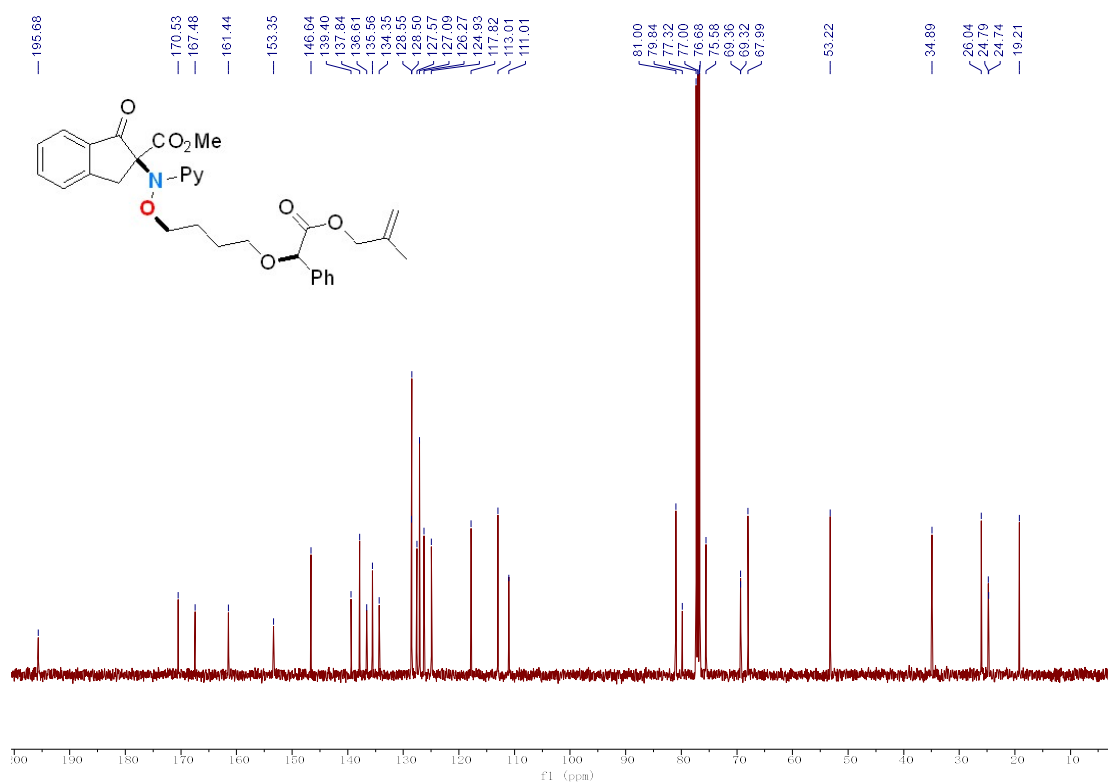
¹³C NMR (100 MHz) Spectrum of 41 in CDCl₃



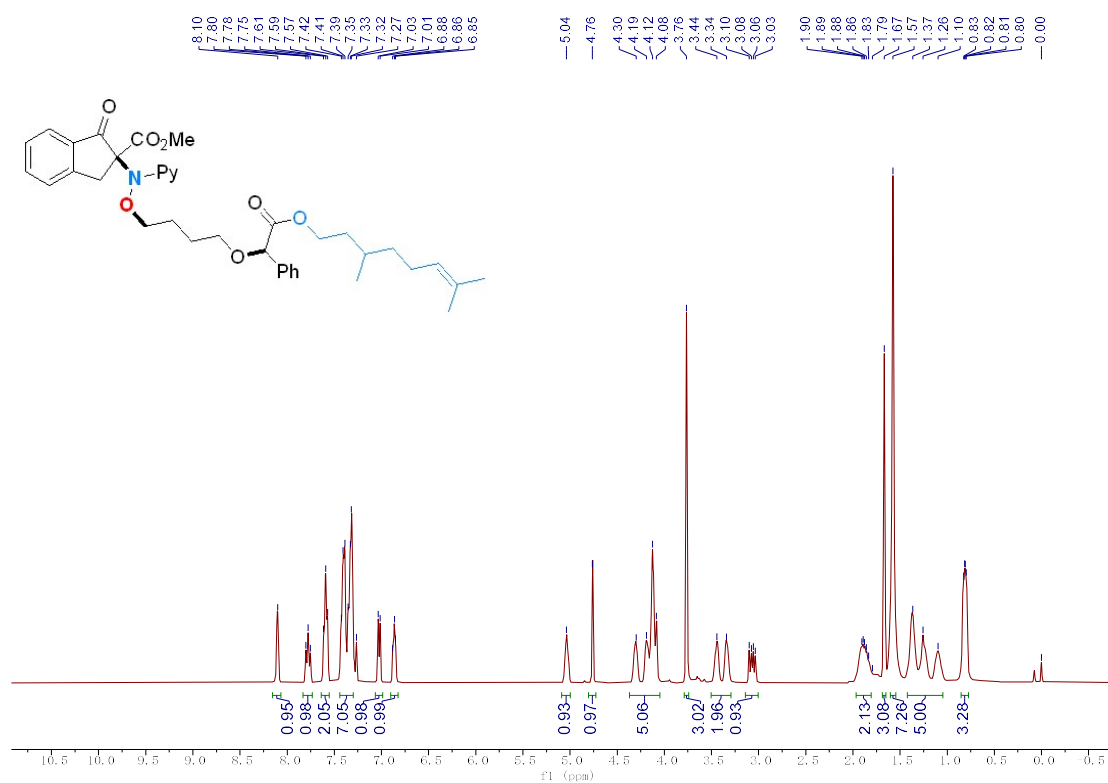
¹H NMR (400 MHz) Spectrum of 43 in CDCl₃



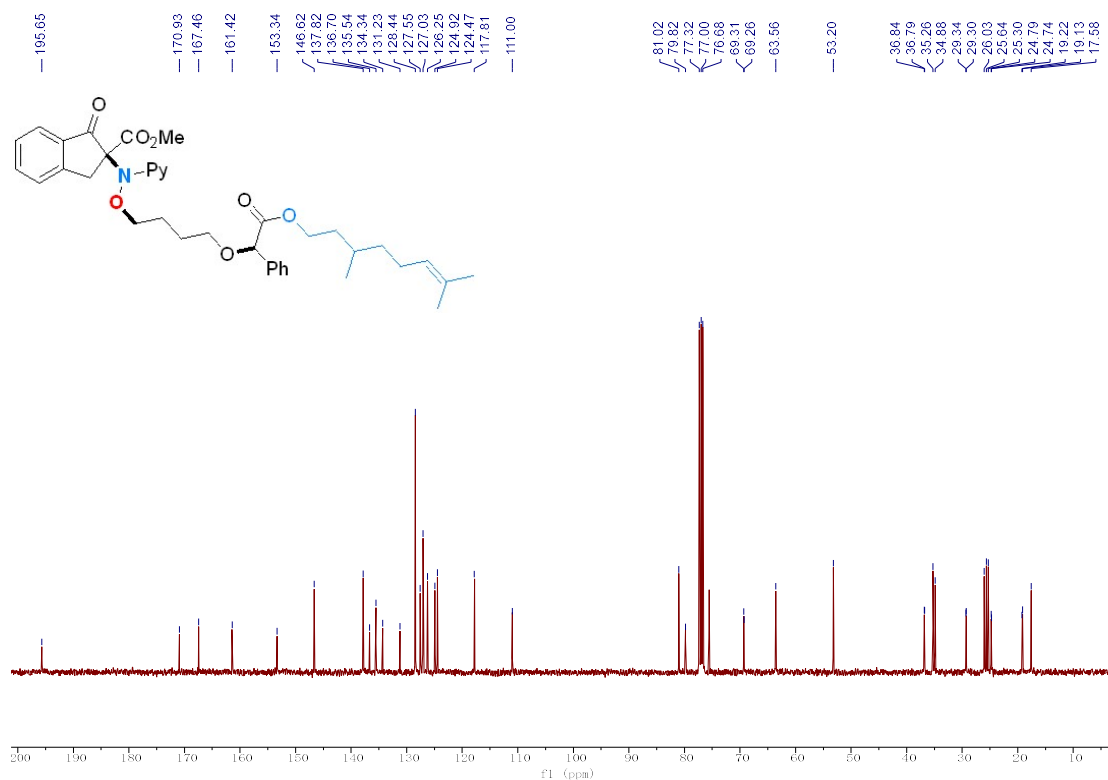
¹³CNMR (100 MHz) Spectrum of 43 in CDCl₃



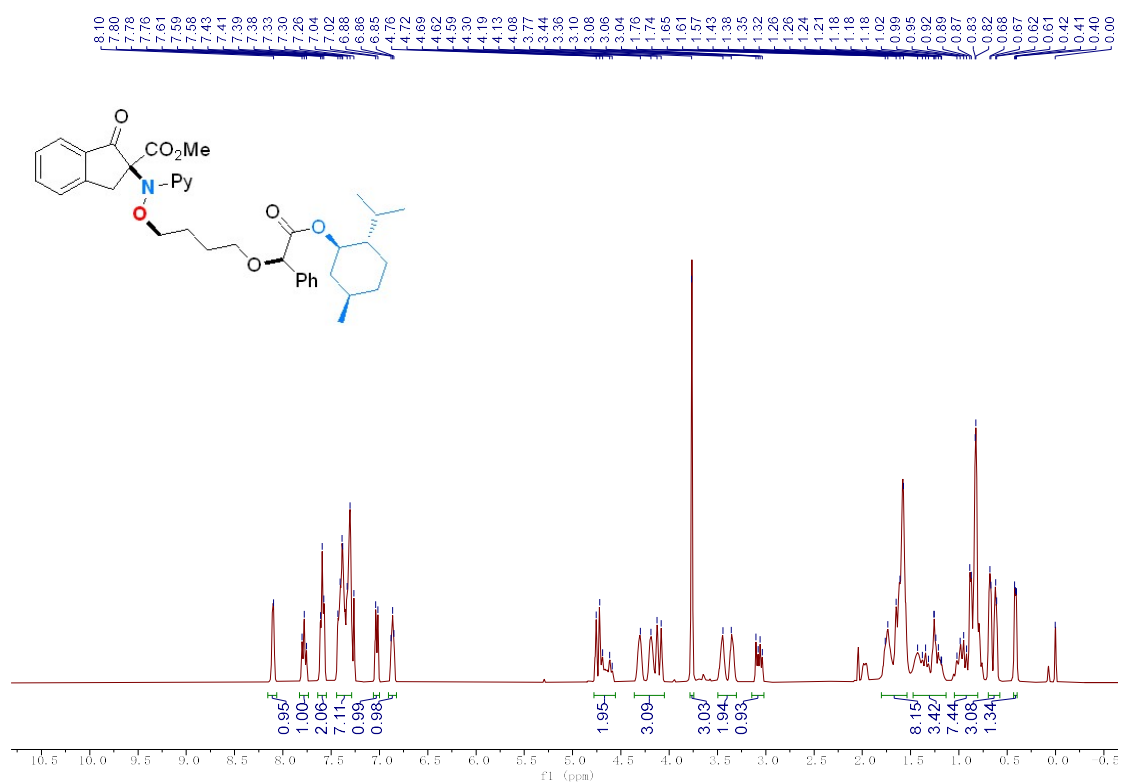
¹H NMR (400 MHz) Spectrum of 44 in CDCl₃



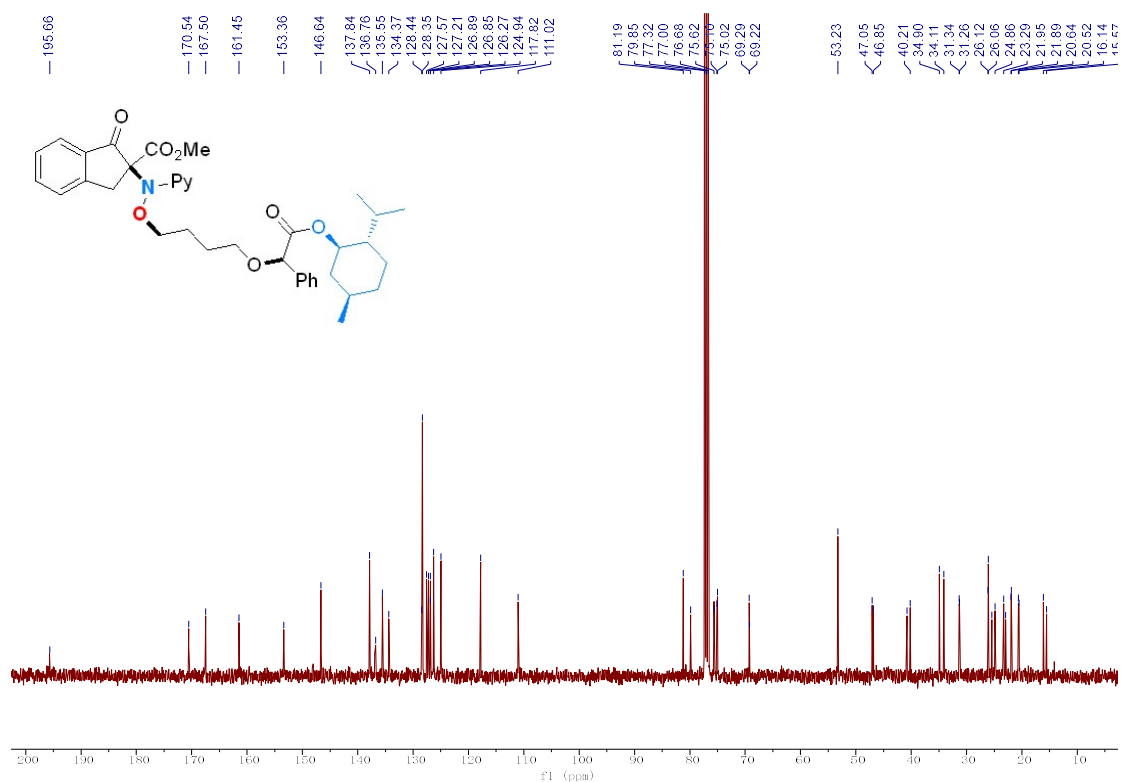
¹³CNMR (100 MHz) Spectrum of 44 in CDCl₃



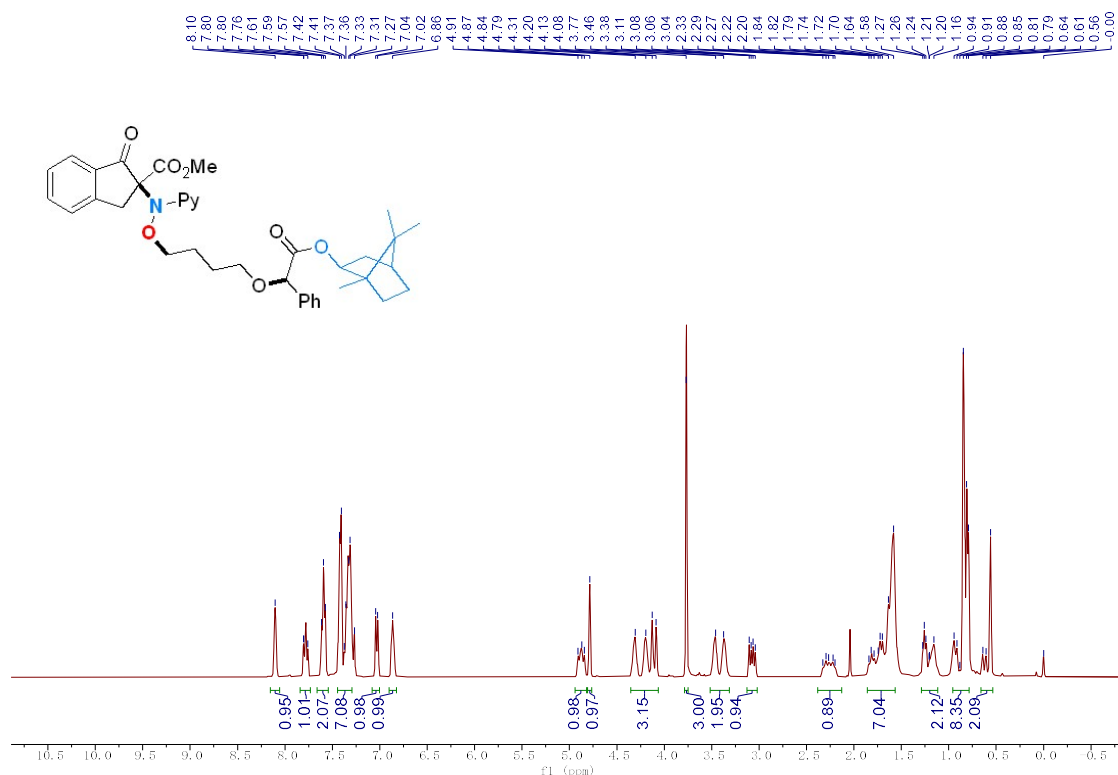
¹H NMR (400 MHz) Spectrum of 45 in CDCl₃



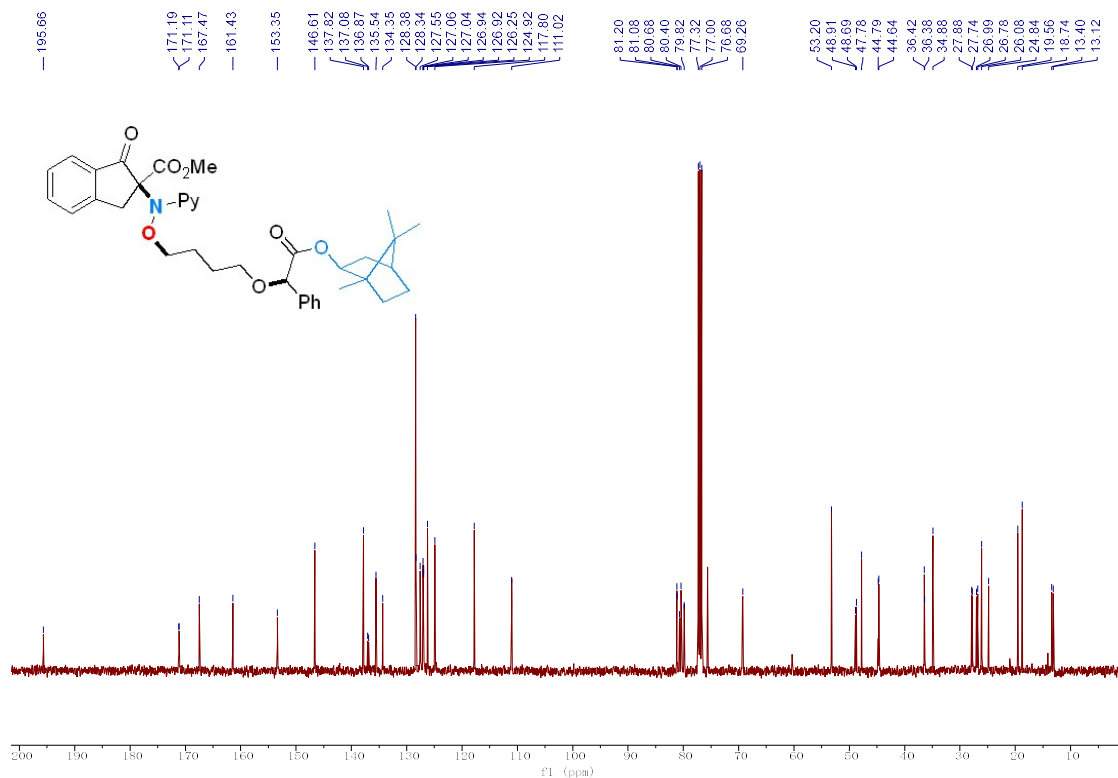
¹³CNMR (100 MHz) Spectrum of 45 in CDCl₃



¹H NMR (400 MHz) Spectrum of 46 in CDCl₃



¹³CNMR (100 MHz) Spectrum of 46 in CDCl₃



Chemical structure of compound 10: CC(=O)O[C@@H](C(=O)OC)CCO[C@H](C(=O)OC)C1=CC=CC=C1

¹H NMR spectrum (CDCl₃) of compound 10. The x-axis represents the chemical shift (δ) in ppm, ranging from 0.00 to 8.11. The spectrum shows several peaks, with integration values provided below the baseline. The chemical shifts (δ) are listed on the right side of the spectrum.

Chemical shifts (δ) in ppm: 8.11, 7.79, 7.77, 7.75, 7.62, 7.60, 7.58, 7.55, 7.43, 7.41, 7.35, 7.33, 7.27, 7.03, 6.88, 6.86, 6.85, 4.82, 4.31, 4.29, 4.27, 4.25, 4.18, 4.17, 4.13, 4.09, 3.77, 3.69, 3.46, 3.44, 3.42, 3.40, 3.38, 3.35, 3.33, 3.32, 3.30, 3.28, 3.10, 3.06, 1.60, 1.58, 1.53, 1.51, 1.49, 1.48, 1.46, 1.37, 1.35, 1.33, 1.30, 1.29, 1.28, 1.27, 0.00.

Integration values: 0.95, 1.02, 2.07, 7.13, 0.99, 1.00, 1.00, 1.00, 3.07, 3.04, 3.05, 2.05, 0.96, 4.10, 2.16.

Chemical structure of the compound is shown above the spectrum. The structure is a derivative of a bicyclic compound, featuring a benzene ring fused to a five-membered ring containing a carbonyl group (C=O) and a nitrogen atom (N) bonded to a pyridine ring (Py). The nitrogen atom is also bonded to a methylene group (CH₂) which is part of a longer chain containing an ether linkage (O) and a chiral center (C*) bonded to a phenyl group (Ph) and a methoxycarbonyl group (CO₂Me).

The ¹³C NMR spectrum (f1 (ppm)) shows the following chemical shifts (ppm):

- 195.64
- 171.33
- 167.45
- 161.42
- 153.32
- 146.90
- 137.68
- 136.66
- 135.50
- 134.30
- 128.55
- 128.52
- 127.49
- 127.04
- 126.22
- 124.87
- 117.77
- 110.95
- 80.95
- 79.74
- 77.32
- 77.00
- 76.68
- 75.97
- 73.77
- 69.51
- 53.17
- 52.12
- 34.88
- 29.23
- 27.79
- 22.35

Chemical structure of compound 10 is shown above the spectrum. The structure is a 1-(4-methoxycarbonyl-2-phenylethoxy)-2-(pyridin-2-yl)-1-phenyl-2-oxo-1,2,3,4-tetrahydroindole-3-carboxylic acid methyl ester.

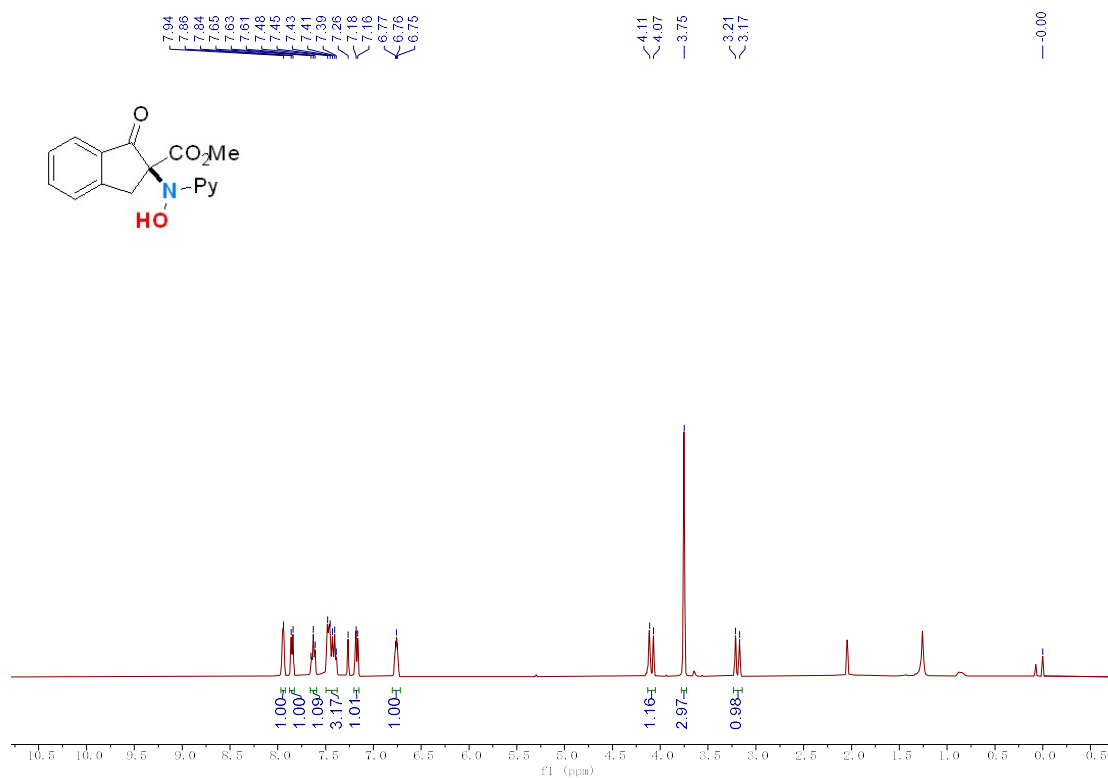
¹H NMR spectrum (CDCl₃) of compound 10. The x-axis is labeled f1 (ppm). The spectrum shows peaks from 0.00 to 8.09 ppm. Integration values are provided below the peaks: 0.94, 0.98, 2.05, 8.14, 0.97, 1.00, 2.00, 1.16, 3.02, 9.32, and 0.95.

Chemical structure of compound 10 is shown. The structure is a phthalimide derivative with a methyl ester group (CO₂Me) and a pyridine ring (Py) attached to the imide ring. The structure also features a 2-methoxy-2-phenylacetate group (CH(Ph)CO₂Me) attached to the imide ring.

¹³C NMR spectrum (f1 (ppm)) of compound 10. The spectrum shows peaks corresponding to the chemical structure, with the following chemical shifts (ppm) labeled above the peaks:

- 195.76
- 171.16
- 167.35
- 161.49
- 153.39
- 146.41
- 138.01
- 136.34
- 135.56
- 134.26
- 128.60
- 128.52
- 127.51
- 127.19
- 126.29
- 124.02
- 117.89
- 111.35
- 81.22
- 80.14
- 77.32
- 77.00
- 76.66
- 73.98
- 70.43
- 69.09
- 69.06
- 68.88
- 68.75
- 53.20
- 52.12
- 34.81

¹H NMR (400 MHz) Spectrum of 49 in CDCl₃



¹³C NMR (100 MHz) Spectrum of 49 in CDCl₃

