Enantioselective α-Arylation of *N*-Acyloxazolidinones with Copper(II)-bisoxazoline Catalysts and Diaryliodonium Salts

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Supporting Information

Experimental procedures and data

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

I. General Experimental

Solvents: All anhydrous solvents were dried by standard techniques and freshly distilled before use. Diethyl ether and tetrahydrofuran were distilled from lithium aluminium hydride; acetonitrile, dichloromethane and toluene from calcium hydride; and triethylamine from potassium hydroxide.

Reagents: All reagents were purified by standard procedures or used as obtained from commercial sources.[1] Copper (II) triflate was purchased from Alfa Aesar, dried under vacuum at 80 °C and stored under nitrogen before use.

Chromatography: All flash chromatography was carried out using dry packed Merck 9385 Kieselgel 60 silica gel and thin layer chromatography was carried out on Merck Kieselgel 60 PF254 0.2 mm plates. Visualisation was accomplished using ultra violet light (254 nm) and chemical staining with *para*-anisaldehyde or acidic potassium permanganate solutions as appropriate.

Reactions: All reactions were carried out using oven dried glassware and under an atmosphere of nitrogen unless otherwise stated. All yields and *ee* reported in the manuscript are averages of at least two experiments, yields and *ee* found in the supporting information represent the data for single reactions.

Data Collection: ^{1}H NMR spectra were recorded on a Bruker DPX 400 or 500 spectrometer in deuterochloroform ($CDCl_3$), unless stated otherwise. ^{13}C NMR spectra were recorded at 100 or 125 MHz on the same machines. ^{19}F NMR spectra were recorded at 376 MHz on a Brucker DPX 400 spectrometer. Chemical shifts (δ) are quoted in parts per million (ppm) relative to residual solvent ($CHCl_3$: δ = 7.26 ppm for ^{1}H and δ = 77.0 for ^{13}C; C_6D_6: δ = 7.15 ppm for ^{1}H and δ = 128.0 for ^{13}C; Acetone-D_6: δ = 2.09 ppm for ^{1}H and δ = 205.87 for ^{13}C=O or relative to external $C^{19}FCl_3$). Coupling constants (*J*) are corrected and quoted to the nearest 0.1 Hz. The following abbreviation are used to indicate the multiplicity of the signals: s = singlet; d = doublet; t = triplet; q = quartet; qn = quintet; st = sextet, sp = septet; m = multiplet; br = broad; app = apparent; and associated combinations, e.g. dd = doublet of doublets. The temperature of the acquisition of the NMR spectra was 298 ± 3K. DEPT135, nOe experiments and 2-dimensional experiments (COSY, HMBC and HMQC) were used to support assignments where appropriate but are not included. NMR yields were determined with 1,3,5-trimethozybenzene as NMR standard.

High resolution mass spectra (HRMS) were measured on a Micromass Q-TOF spectrometer using EI (electron impact) or ES (electrospray ionisation) techniques at the Department of Chemistry, University of Cambridge or at the EPSRC Mass Spectrometry Service at the University of Swansea.

Infared (IR) spectra were recorded on a Perkin Elmer 1FT-IR Spectrometer fitted with an ATR

[1] Armarego, W. L. F.; Perrin, D. D., *Purification of Laboratory Chemicals*, 5th Ed., Butterworth-Heinemann, **1996**.

sampling accessory as either solids or neat films, either through direct application or deposited in $CHCl_3$, with absorptions reported in wavenumbers (cm^{-1}). Optical rotations were measured in $CHCl_3$ on a Perkin Elmer 343 Polarimeter using a sodium lamp (λ 589 nm, D-line). $[\alpha]_D^{20}$ values are reported in 10^{-1} degrees cm^2 g^{-1} and concentration in mg mL^{-1}.

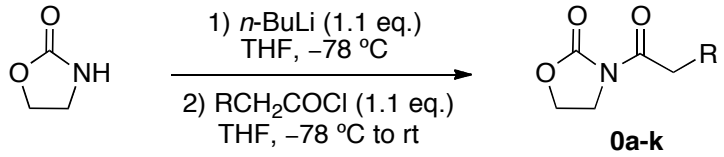
Melting points (m.p.) were recorded using a Reichert hot stage apparatus and are reported uncorrected.

Chiral HPLC analysis was performed on HP Agilent 1100 apparatus using chiralpak columns (IA, IC, AD-H and OD).

X-ray crystallography was performed on a Nonius Kappa CCD at the Cambridge University Chemistry X-Ray Laboratory.

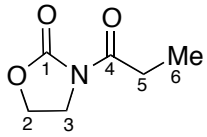
II. Preparation of *N*-acyl-oxazolidin-2-ones

General Procedure A: Preparation of *N*-acyl-oxazolidin-2-ones 0a-0k[2]



A solution of *n*-butyllithium (1.6 M in hexanes, 1.0–1.1 eq.) was added dropwise *via* syringe to a stirred solution of oxazolidin-2-one (1.0 eq.) in THF at −78 ˚C under a nitrogen atmosphere and the mixture was allowed to stir for 15 minutes. The appropriate *acid chloride*[3] (1.0–1.1 eq.) was then added dropwise at −78 ˚C. The reaction mixture was stirred at −78 ˚C for 30 minutes and then allowed to warm to room temperature over 1 hour. A saturated aqueous solution of NH_4Cl was added and the reaction extracted with dichloromethane. The combined organic extracts were washed with a saturated solution of aqueous $NaHCO_3$, dried over Na_2SO_4, and concentrated *in vacuo*. The crude product was then purified by the stated method to afford the desired N-*acyl oxazolidin-2-one* **0**.

3-Propionyloxazolidin-2-one **0a**



Prepared according to General Procedure A using oxazolidin-2-one (10.0 g, 115 mmol), *n*-butyllithium (79.2 mL, 127 mmol) and propionyl chloride (11.0 mL, 127 mmol) in THF (120 mL); flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 7:3) provided the title compound (13.7 g, 95.7 mmol, 83% yield) as a white solid; IR ν_{max} (solid) / cm^{-1} 2991, 2923, 1764, 1696; ^1H NMR (400 MHz, $CDCl_3$) δ: 4.40 (2H, app t, J = 7.6 Hz, H^2), 4.02 (2H, app t, J = 8.3 Hz, H^3), 2.93 (2H, q, J = 7.3 Hz, H^5), 1.16 (3H, t, J = 7.4 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.2, 153.6, 62.0, 42.7, 28.7, 8.3.

Analytical data in accordance with literature precedent.[2]

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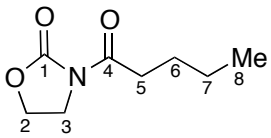
[2] Feuillet, F. J. P.; Cheeseman, M.; Mahon, M. F.; Bull, S. D. *Org. Biomol. Chem*. **2005**, *3*, 2976.

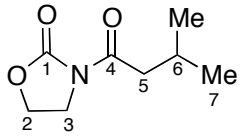
[3] Acid chlorides were prepared as follows: an oven dried round bottomed flask was charged with the appropriate acid (1.0 eq.) and then dichloromethane was added (0.1 M solution). The reaction mixture was cooled to 0 °C and 2 drops of DMF were added followed by freshly distilled oxalyl chloride (2.0 eq.). The reaction mixture was stirred at 0 °C for 30 minutes and then allowed to reach room temperature whilst stirring for 1 hour. The volatiles were then removed *in vacuo* to furnish the desired *acid chloride*.

3-Pentanoyloxazolidin-2-one **0b**



Prepared according to General Procedure A using oxazolidin-2-one (5.00 g, 57.4 mmol), *n*-butyllithium (39.5 mL, 63.1 mmol) and pentanoyl chloride (7.65 mL, 63.1 mmol) in THF (60 mL); flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 7:3) provided the title compound (6.73 g, 39.3 mmol, 68% yield) as a colourless semi-solid; IR ν_{max} (film) / cm^{-1} 2960, 2935, 2875, 1771, 1695, 1480, 1385, 1362, 1272, 1221, 1197, 1128, 1091, 1038; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.42–4.38 (2H, m, H^2), 4.03–3.99 (2H, m, H^3), 2.91 (2H, t, J = 7.6 Hz, H^5), 1.68–1.60 (2H, m, H^6), 1.43–1.34 (2H, m, H^7), 0.93 (3H, t, J = 7.4 Hz, H^8); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.5, 153.5, 61.9, 42.5, 34.8, 26.3, 22.2, 13.8; HRMS (CI) calculated for $C_8H_{14}NO_3$ [M+H^+] m/z 172.0968, found 172.0970.

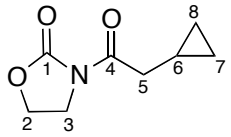
3-(3-Methylbutanoyl)oxazolidin-2-one **0c**



Prepared according to General Procedure A using oxazolidin-2-one (3.28 g, 37.6 mmol), *n*-butyllithium (25.8 mL, 41.4 mmol) and valeroyl chloride (5.05 mL, 41.4 mmol) in THF (40 mL); flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 7:3) provided the title compound (6.40 g, 37.3 mmol, 99% yield) as a colourless solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.41–4.38 (2H, m, H^2), 4.03–4.00 (2H, m, H^3), 2.82 (2H, d, J = 7.0 Hz, H^5), 2.95 (1H, app sp, J = 7.2 Hz, H^6), 0.99 (6H, d, J = 7.4 Hz, H^7); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 172.9, 153.5, 61.9, 43.5, 42.5, 25.0, 22.5.

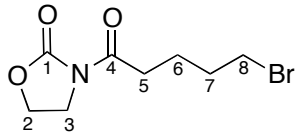
Analytical data in accordance with literature precedent.[2]

3-(2-Cyclopropylacetyl)oxazolidin-2-one **0d**



To a stirred solution of oxazolidin-2-one (478 mg, 5.49 mmol) and 2-cyclopropylacetic acid (500 mg, 4.99 mmol) in THF (100 mL) was added lithium chloride (423 mg, 9.98 mmol) and the resulting slurry was stirred for 30 minutes at room temperature. The resulting mixture was cooled to 0 ˚C and pivaloyl chloride (1.53 mL, 12.4 mmol) was added in one-portion followed by dropwise addition of triethylamine (1.8 mL, 12.9 mmol) over the course of 20 minutes. The reaction mixture was stirred at 0 ˚C for 1 hour, and then allowed to reach room temperature whilst stirring for 1 hour. The reaction mixture was concentrated, ethyl acetate (100 mL) was added followed by a saturated aqueous solution of K_2CO_3 (100 mL). The aqueous phase was separated, extracted with ethyl acetate (3 × 50 mL), combined organic phases washed with brine (100 mL), dried over Na_2SO_4 and evaporated. The crude residue was purified by column chromatography on silica gel (40–60 petroleum ether:ethyl acetate 7:3 to 1:1) to provide the title compound (737 mg, 4.35 mmol, 87% yield) as a white solid; IR ν_{max} (solid) / cm^{-1} 3000, 2932, 2909, 1758, 1686; m.p. 58–60 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.43–4.39 (2H, m, H^2), 4.05–4.01 (2H, m, H^3), 2.84 (2H, d, J = 7.0 Hz, H^5), 1.13–1.07 (1H, m, H^6), 0.56–0.53 (2H, m, H^7 and H^8), 0.21–0.17 (2H, m, $H^{7'}$ and $H^{8'}$); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.1, 153.6, 62.0, 42.5, 40.2, 6.2, 4.2; HRMS (ESI) calculated for $C_8H_{12}NO_3$ [M+H^+] m/z 170.0812, found 170.0811.

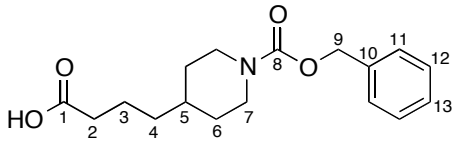
3-(5-Bromopentanoyl)oxazolidin-2-one **0e**



Prepared according to General Procedure A using oxazolidin-2-one (2.18 g, 25.0 mmol), *n*-butyllithium (15.6 mL, 25.0 mmol) and 5-bromopentanoyl chloride (5.0 g, 25.0 mmol) in THF (40 mL); flash column chromatography on silica gel (40–60 petroleum ether:ethyl acetate 7:3) provided the title compound (6.22 g, 24.9 mmol, 99% yield) as a white solid; IR ν_{max} (film) / cm^{-1} 2963, 2918, 2862, 1761, 1697; m.p. 38–40 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.44–4.40 (2H, m, H^2), 4.02 (2H, app t, J = 8.1 Hz, H^3), 3.43 (2H, t, J = 6.5 Hz, H^8), 2.95 (2H, t, J = 7.2 Hz, H^5), 1.97–

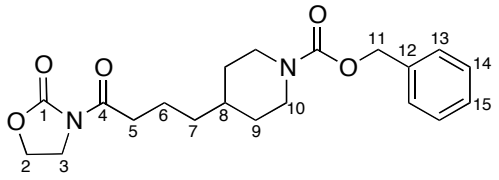
1.90 (2H, m, H^6 or H^7), 1.86–1.79 (2H, m, J = 7.4 Hz, H^6 or H^7); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 172.8, 153.5, 62.1, 42.5, 34.2, 33.1, 32.0, 22.8; HRMS (ESI) calculated for $C_8H_{13}BrNO_3$ [M+H^+] m/z 250.0073, found 250.0078.

4-(1-((Benzyloxy)carbonyl)piperidin-4-yl)butanoic acid



To a solution of 4-piperidine butyric acid hydrochloride (5.0 g, 24.0 mmol) in water (18 mL) and aqueous NaOH (50 mL, 1M) at 0 ˚C was added dropwise a solution of Cbz-Cl (4.92 g, 4.12 mL, 28.8 mmol) in dioxane (5 mL) whilst maintaining a pH between 9 and 10. The reaction solution was stirred for one additional hour at 0 ˚C and then overnight at room temperature. The reaction mixture was subsequently concentrated *in vacuo* and the resulting basic solution was washed with ethyl acetate (2 × 25 mL). The aqueous phase was acidified through the addition of conc. HCl to pH = 0 and extracted with ethyl acetate (3 × 50 mL). Combined organic phases were washed with brine, dried over Na_2SO_4 and evaporated to afford the title compound (5.95 g, 19.4 mmol, 81%) as a white solid; IR ν_{max} (film) / cm^{-1} 2922, 2860, 2326, 2110, 1091, 1731, 1698, 1673, 1473, 1433; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.36–7.29 (5H, m, $H^{11\text{-}13}$), 5.12 (2H, s, H^9), 4.16 (2H, bs, H^7), 2.75 (2H, bs, $H^{7'}$), 2.37 (2H, m, H^2), 1.66 (4H, bs, H^3 and H^6), 1.42 (1H, m, H^5), 1.33–1.23 (2H, m, H^4), 1.18–1.03 (2H, m, $H^{6'}$); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.3, 136.9, 128.4, 127.9, 127.8, 67.0, 44.2, 35.8, 35.7, 31.9, 21.8; HRMS (ESI) calculated for $C_{17}H_{22}NO_4$ [M-H^+] m/z 304.1554, found 304.1553.

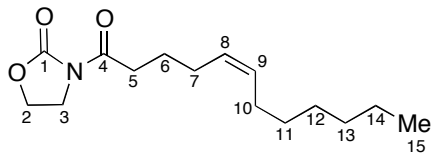
Benzyl 4-(4-oxo-4-(2-oxooxazolidin-3-yl)butyl)piperidine-1-carboxylate **0f**



Prepared according to General Procedure A using oxazolidin-2-one (1.5 g, 17.2 mmol), *n*-butyllithium (11.8 mL, 18.9 mmol) and freshly prepared benzyl 4-(4-chloro-4-oxobutyl)piperidine-1-carboxylate[3] (6.14 g, 18.9 mmol) in THF (100 mL); flash column chromatography on silica gel (40–60 petroleum ether:ethyl acetate 9:1 to 1:1) provided the title

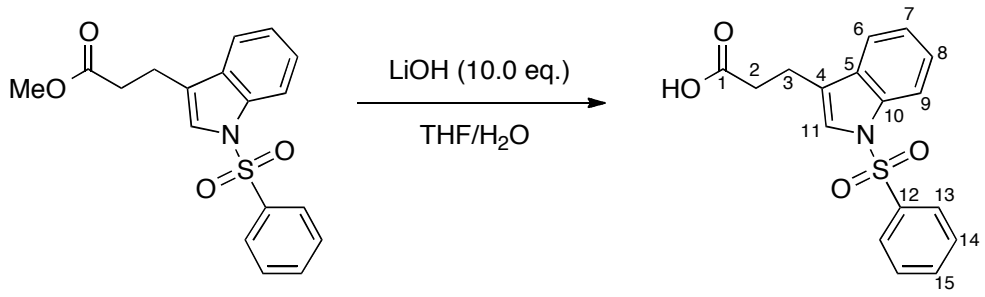
compound (6.17 g, 16.4 mmol, 95% yield) as a colourless oil; IR ν_{max} (solid) / cm^{-1} 2959, 2923, 1761, 1683, 1469, 1454, 1433, 1391, 1272, 1218; m.p. 62 ºC; ^{1}H NMR (400 MHz $CDCl_3$) δ: 7.36–7.29 (5H, m, H^{13-15}), 5.12 (2H, s, H^{11}), 4.43–4.38 (2H, m, H^2), 4.23-4.08 (2H, bs, H^{10}), 4.03–3.99 (2H, m, H^3), 2.90 (2H, t, J = 7.7 Hz, H^5), 2.81–2.70 (2H, bs, $H^{10'}$), 1.72–1.62 (4H, m, H^6 and H^9), 1.49–1.36 (1H, m, H^8), 1.34–1.24 (2H, m, H^7), 1.17–1.04 (2H, m, $H^{9'}$); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.3, 155.3, 153.5, 137.0, 128.4, 127.9, 127.8, 66.9, 62.0, 44.2, 42.5, 35.8, 35.1, 31.9, 31.8, 21.3; HRMS (ESI) calculated for $C_{20}H_{27}N_2O_5$ [M+H^+] m/z 375.1914, found 375.1913.

(Z)-3-(Dodec-5-enoyl)oxazolidin-2-one **0g**



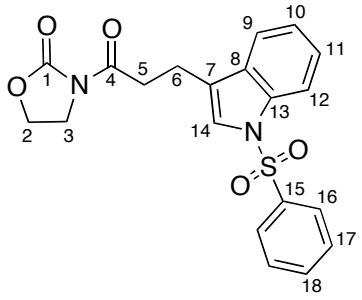
Prepared according to General Procedure A using oxazolidin-2-one (878 mg, 10.0 mmol), *n*-butyllithium (6.25 mL, 10.0 mmol) and freshly prepared (*Z*)-dodec-5-enoyl chloride[3] (2.16 g, 10.0 mmol) in THF (30 mL); flash column chromatography on silica gel (40–60 petroleum ether:ethyl acetate 9:1 to 7:3) provided the title compound (2.58 g, 9.6 mmol, 96% yield) as a colourless oil; IR ν_{max} (film) / cm^{-1} 2924, 2856, 1776, 1698, 1480, 1458, 1384, 1362; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 5.44–5.30 (2H, m, H^8 and H^9), 4.42–4.38 (2H, m, H^2), 4.03–3.99 (2H, m, H^3), 2.94–2.90 (2H, m, H^5), 2.14–2.08 (2H, m, H^7), 2.04–1.98 (2H, m, H^{10}), 1.76–1.68 (2H, m, H^6), 1.34–1.24 (8H, m, H^{11-14}), 0.89–0.86 (2H, m, H^{15}); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.4, 153.5, 131.1, 128.4, 62.0, 42.5, 34.6, 31.8, 29.6, 29.0, 27.2, 26.5, 24.2, 22.6, 14.1; HRMS (ESI) calculated for $C_{15}H_{26}NO_3$ [M+H^+] m/z 268.1907, found 268.1913

*3-(1-(Phenylsulfonyl)-1*H*-indol-3-yl)propanoic acid*



To a solution of methyl 3-(1-(phenylsulfonyl)-1*H*-indol-3-yl)propanoate[4] (4.14 g, 11.5 mmol) in THF (150 mL) was added a solution of LiOH (4.86 g, 115 mmol) in H_2O (20 mL) and the reaction mixture was stirred for 2 hours at room temperature. The solution was subsequently acidified to pH = 0 by dropwise addition of conc. HCl (12 N) at 0 ˚C, the aqueous phase was then extracted with dichloromethane (3 × 50 mL), the combined organic phases were dried over Na_2SO_4 and concentrated in *vacuo* to afford the title compound (3.46 g, 10.5 mmol, 91%) as a pale yellow solid which was used without further purification; IR ν_{max} (solid) / cm^{-1} 2918, 2920, 1765, 1697; m.p. 172–175 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.00 (1H, dt, *J* = 8.3 and 0.8 Hz, H^6), 7.87–7.84 (2H, m, H^{13}), 7.52–7.48 (2H, m, H^{11} and H^{15}), 7.44–7.39 (3H, m, H^9 and H^{14}), 7.36–7.32 (1H, m, H^7), 7.28–7.24 (1H, m, H^8) 3.05–3.01 (2H, m, H^2 or H^3), 2.79–2.75 (2H, m, H^2 or H^3); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 177.2, 138.2, 135.3, 133.7, 130.5, 129.2, 126.7, 124.9, 123.3, 123.0, 121.4, 119.2, 113.8, 33.1, 20.0; HRMS (ESI) calculated for $C_{17}H_{14}NO_4S$ [M-H^+] m/z 328.0649, found 328.0643.

*3-(3-(1-(Phenylsulfonyl)-1*H*-indol-3-yl)propanoyl)oxazolidin-2-one* **0h**

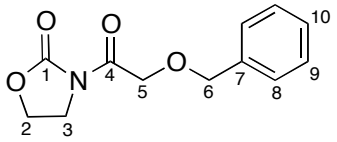


Prepared according to General Procedure A using oxazolidin-2-one (507 mg, 5.82 mmol), *n*-butyllithium (3.63 mL, 5.82 mmol) and freshly prepared 3-(1-(phenylsulfonyl)-1*H*-indol-3-yl)propanoyl chloride[3] (2.02 g, 5.82 mmol) in THF (30 mL); flash chromatography on silica gel

[4] Prepared according to: Jui, N. T.; Lee, E. C. Y.; MacMillan D. W. C., *J. Am. Chem. Soc.* **2010**, *132*, 10015.

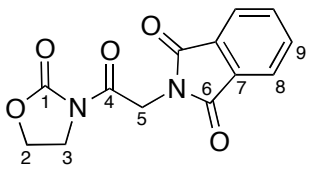
(40–60 petroleum ether:dichloromethane:ethyl acetate 5:5:1 to 1:1:1) provided the title compound (2.08 g, 5.22 mmol, 90% yield) as a white solid; m.p. 122–126 ºC; IR ν_{max} (solid) / cm^{-1} 2919, 1774, 1698, 1479, 1447, 1388, 1363; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98 (1H, dt, *J* = 8.2 and 0.9 Hz, H^9), 7.88–7.85 (2H, m, H^{16}), 7.55–7.50 (2H, m, H^{14} and H^{18}), 7.45–7.40 (3H, m, H^{12} and H^{17}), 7.34–7.29 (1H, m, H^{10}), 7.26–7.22 (1H, m, H^{11}), 4.42–4.38 (2H, m, H^2), 4.03–3.99 (2H, m, H^3), 3.35–3.28 (2H, m, H^5 or H^6), 3.07–3.03 (2H, m, H^5 or H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 172.2, 153.5, 138.3, 135.2, 133.7, 130.6, 129.2, 126.7, 124.8, 123.2 (x 2), 121.6, 119.4, 113.7, 62.1, 42.5, 34.7, 19.5; HRMS (ESI) calculated for $C_{20}H_{22}N_3O_5S$ [M+NH_4^+] m/z 416.1275, found 416.1277.

3-(2-(Benzyloxy)acetyl)oxazolidin-2-one **0i**



Prepared according to General Procedure A using oxazolidin-2-one (1.23 g, 14.1 mmol), *n*-butyllithium (9.7 mL, 15.5 mmol) and 2-(benzyloxy)acetyl chloride (2.86 g, 15.5 mmol) in THF (30 mL); flash column chromatography on silica gel (40–60 petroleum ether:ethyl acetate 1:1) provided the title compound (2.78 g, 11.8 mmol, 83% yield) as a colourless semi-solid; IR ν_{max} (solid) / cm^{-1} 3003, 2924, 1759, 1715; m.p. 120–122 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.41–7.28 (5H, m, $H^{8\text{-}10}$), 4.70 (2H, s, H^5 or H^6), 4.67 (2H, s, H^5 or H^6), 4.47–4.45 (2H, m, H^2), 4.06–4.02 (2H, m, H^3); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.7, 153.4, 137.1, 128.5, 128.1, 128.0, 73.5, 69.3, 63.1, 42.0; HRMS (ESI) calculated for $C_{12}H_{14}NO_4$ [M+H^+] m/z 236.0917, found 236.0919.

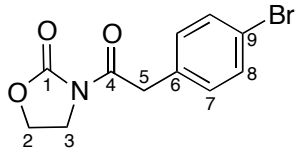
2-(2-Oxo-2-(2-oxooxazolidin-3-yl)ethyl)isoindoline-1,3-dione **0j**



Prepared according to General Procedure A using oxazolidin-2-one (2.0 g, 22.9 mmol), *n*-butyllithium (14.3 mL, 22.9 mmol) and freshly prepared 2-(1,3-dioxoisoindolin-2-yl)acetyl chloride[3] (5.64 g, 25.2 mmol) in THF (60 mL); flash column chromatography on silica gel (dichloromethane:ethyl acetate 10:1) provided the title compound (2.08 g, 7.58 mmol, 33% yield) as a white solid; IR ν_{max} (solid) / cm^{-1} 2995, 2956, 1765, 1701, 1616; m.p. 204–207 ºC; ^{1}H NMR

(400 MHz, $CDCl_3$) δ: 7.92–7.87 (2H, m, H^8 or H^9), 7.77–7.73 (2H, m, H^8 or H^9), 5.04 (2H, s, H^5), 4.54–4.50 (2H, m, H^2), 4.08–4.04 (2H, m, H^3); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.7, 166.4, 153.6, 134.2, 132.1, 123.6, 63.1, 42.2, 41.4; HRMS (ESI) calculated for $C_{13}H_{11}N_2O_5$ [M+H^+] m/z 275.0662, found 275.0667.

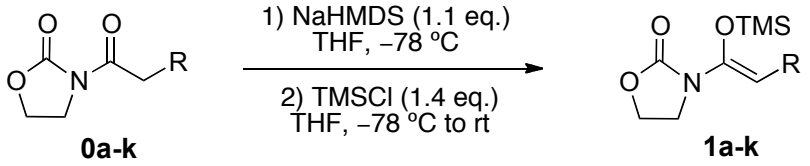
3-(2-(4-Bromophenyl)acetyl)oxazolidin-2-one **0k**



Prepared according to General Procedure A using oxazolidin-2-one (5.00 g, 57.4 mmol), *n*-butyllithium (39.5 mL, 63.1 mmol) and freshly prepared 4-bromophenylacetyl chloride[3] (57.4 mmol) in THF (60 mL); trituration from diethyl ether and 40–60 petroleum ether provided the title compound (8.32 g, 29.3 mmol, 51% yield) as a white solid; m.p. 117 °C; IR ν_{max} (solid) / cm^{-1} 2981, 1778, 1698, 1493, 1472, 1387, 1370, 1310, 1266, 1220; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.46–7.44 (2H, m, H^8), 7.20–7.18 (2H, m, H^7), 4.43–4.40 (2H, m, H^2), 4.23 (2H, s, H^5), 4.02 (2H, app t, J = 8.1 Hz, H^3); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.7, 153.4, 132.4, 131.6, 131.5, 121.3, 62.0, 42.6, 40.5; HRMS (ESI) calculated for $C_{11}H_{11}BrNO_3$ [M+H^+] m/z 283.9917, found 283.9919.

III. Preparation of Silyl Ketenimides

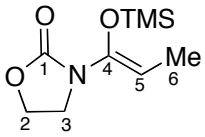
General Procedure B: Preparation of silyl ketenimides 1a-k[5]



Sodium bis(trimethylsilyl)amide (2.0 M in THF, 1.1 eq.) was added to a stirred solution of the appropriate N-*acyloxazolidin-2-one* (1.0 eq.) in THF (0.3 M) at –78 ˚C and stirred for 30 minutes. Trimethylsilyl chloride (1.4 eq.) was added slowly at –78 ˚C and then the reaction mixture was allowed to warm to room temperature over 1 hour. Volatiles were then removed *in vacuo* under a nitrogen atmosphere and the resulting residue dried under a reduced pressure (2 mbar) for 30 minutes. Dry diethyl ether was then added to the residue and the resulting suspension was filtered quickly through Celite™. The filtrate was concentrated *in vacuo* under a nitrogen atmosphere to provide the desired *silyl ketenimide* **1** in quantitative yield (judged to be 90–95% pure by NMR).

Compounds **1a-k** were synthesised according to General Procedure B and used without further purification.

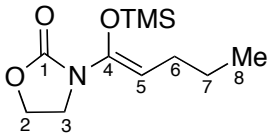
(Z)-3-(1-((Trimethylsilyl)oxy)prop-1-en-1-yl)oxazolidin-2-one **1a**



^{1}H NMR (400 MHz, C_6D_6) δ: 4.78 (1H, q, J = 6.9 Hz, H^5), 3.31–3.29 (2H, m, H^2), 2.87–2.83 (2H, m, H^3), 1.56 (3H, d, J = 6.9 Hz, H^6), 0.17 (9H, s, –Si(CH_3)$_3$); ^{13}C NMR (100 MHz, C_6D_6) δ: 155.7, 141.6, 98.7, 61.6, 45.5, 11.5, 0.8.

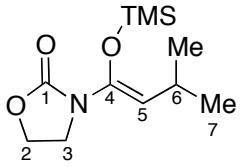
[5] Procedure for preparation adapted from: Hou, D-R.; Reibenspies, J. H.; Burgess, K., *J. Org. Chem*. **2001**, *66*, 206

(Z)-3-(1-((Trimethylsilyl)oxy)pent-1-en-1-yl)oxazolidin-2-one **1b**



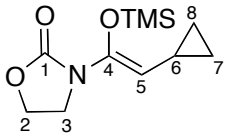
^{1}H NMR (400 MHz, C_6D_6) δ: 4.77 (1H, t, J = 7.3 Hz, H^5), 3.33–3.29 (2H, m, H^2), 2.87–2.83 (2H, m, H^3), 2.09 (2H, app q, J = 7.3 Hz, H^6), 1.38 (2H, app st, J = 7.4 Hz, H^7), 0.92 (3H, t, J = 7.3 Hz, H^8), 0.19 (9H, s, –Si(CH_3)$_3$).

(Z)-3-(3-Methyl-1-((trimethylsilyl)oxy)but-1-en-1-yl)oxazolidin-2-one **1c**



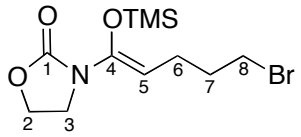
^{1}H NMR (400 MHz, C_6D_6) δ: 4.60 (1H, d, J = 9.6 Hz, H^5), 3.34–3.20 (2H, m, H^2), 2.83–2.81 (2H, m, H^3), 2.74–2.65 (1H, m, H^6), 1.04 (6H, d, J = 6.8 Hz, H^7), 0.18 (9H, s, –Si(CH_3)$_3$); ^{13}C NMR (100 MHz, C_6D_6) δ: 155.5, 140.0, 111.7, 61.5, 45.7, 26.1, 24.0, 0.72.

(Z)-3-(2-Cyclopropyl-1-((trimethylsilyl)oxy)vinyl)oxazolidin-2-one **1d**



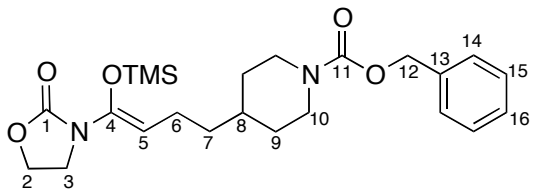
^{1}H NMR (400 MHz, C_6D_6) δ: 4.60 (1H, d, J = 9.2 Hz, H^5), 3.33–3.29 (2H, m, H^2), 2.88–2.84 (2H, m, H^3), 1.56–1.49 (1H, m, H^6), 0.57–0.53 (2H, m, H^7 and H^8), 0.30–0.22 (11H, m, $H^{7'}$, $H^{8'}$ and -Si(CH_3)$_3$); ^{13}C NMR (100 MHz, C_6D_6) δ: 155.6, 140.8, 108.8, 61.6, 45.5, 9.3, 7.7, 0.80.

(Z)-3-(5-Bromo-1-((trimethylsilyl)oxy)pent-1-en-1-yl)oxazolidin-2-one **1e**



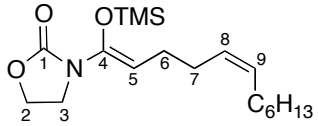
^{1}H NMR (400 MHz, C_6D_6) δ: 4.60 (1H, t, J = 7.3 Hz, H^5), 3.32–3.28 (2H, m, H^2), 3.05 (2H, t, J = 6.6 Hz, H^8), 2.82–2.78 (2H, m, H^3), 2.09 (2H, app q, J = 7.3 Hz, H^6), 1.65–1.58 (2H, m, H^7), 0.16 (9H, s, –Si(CH_3)$_3$); ^{13}C NMR (100 MHz, C_6D_6) δ: 155.5, 141.7, 101.8, 61.5, 45.5, 34.0, 33.4, 24.9, 0.78.

(Z)-Benzyl 4-(4-(2-oxooxazolidin-3-yl)-4-((trimethylsilyl)oxy)but-3-en-1-yl)piperidine-1-carboxylate **1f**



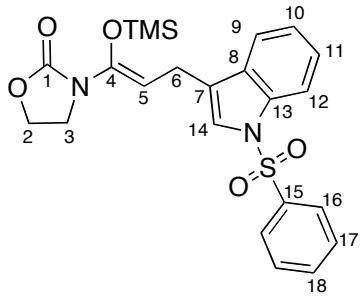
^{1}H NMR (400 MHz, C_6D_6) δ: 7.29–7.24 (2H, bd, J = 7.7 Hz), 7.14–7.08 (2H, bt, J = 7.1 Hz), 7.08–7.02 (1H, m), 5.15 (2H, s), 4.72 (1H, t, J = 7.2 Hz), 4.34 (1H, bs), 4.02 (1H, bs), 3.44–3.40 (2H, m), 2.96–2.92 (2H, m), 2.46 (2H, dt, J = 12.9 and 2.7 Hz), 2.00 (2H, q, J = 7.3 Hz), 1.39–1.25 (2H, m), 1.15–1.05 (3H, m), 0.95–0.81 (2H, m), 0.17 (9H, s, –Si(CH_3)$_3$).

3-((1Z,5Z)-1-((Trimethylsilyl)oxy)dodeca-1,5-dien-1-yl)oxazolidin-2-one **1g**



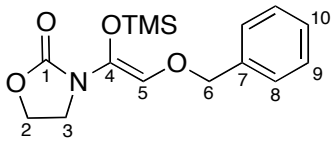
^{1}H NMR (400 MHz, C_6D_6) δ: 5.55–5.46 (2H, m, H^8 and H^9), 4.88–4.78 (1H, m, H^2), 3.34–3.27 (2H, m, H^3), 2.91–2.78 (2H, m), 2.26–2.16 (4H, m), 2.10–2.03 (2H, m), 1.40–1.16 (8H, m), 0.94–0.82 (3H, m), 0.23 (9H, s, –Si(CH_3)$_3$).

*(Z)-3-(3-(1-(Phenylsulfonyl)-1*H*-indol-3-yl)-1-((trimethylsilyl)oxy)prop-1-en-1-yl)oxazolidin-2-one* **1h**



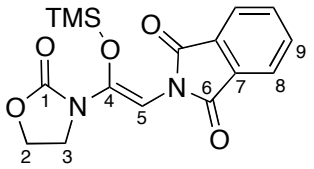
^{1}H NMR (400 MHz, C_6D_6) δ: 8.28 (1H, app d, J = 8.2 Hz, ArH), 7.79–7.76 (2H, m, ArH), 7.61 (1H, s, ArH), 7.44 (1H, m, ArH), 7.17–7.13 (1H, m, ArH), 7.08–7.04 (1H, m, ArH), 6.70–6.65 (3H, m, ArH), 4.84 (1H, t, J = 7.4 Hz, H^5), 3.35–3.27 (4H, m, H^2 and H^6), 2.83–2.74 (2H, m, H^3), 0.12 (9H, s, –Si(CH_3)$_3$).

(Z)-3-(2-(Benzyloxy)-1-((trimethylsilyl)oxy)vinyl)oxazolidin-2-one **1i**



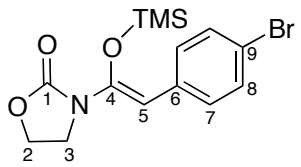
^{1}H NMR (400 MHz, C_6D_6) δ: 7.20–7.05 (5H, m, H^{8-10}), 5.96 (1H, s, H^5), 4.39 (2H, s, H^6), 3.32–3.28 (2H, m, H^2), 2.86–2.82 (2H, m, H^3), 0.29 (9H, s, –Si(CH_3)$_3$.

(Z)-2-(2-(2-Oxooxazolidin-3-yl)-2-((trimethylsilyl)oxy)vinyl)isoindoline-1,3-dione **1j**



^{1}H NMR (400 MHz, C_6D_6) δ: 7.53–7.49 (2H, m, H^7 or H^8),), 6.94–6.89 (2H, m, H^7 or H^8), 5.76 (1H, s, H^5), 3.19–3.15 (2H, m, H^2), 2.74–2.69 (2H, m, H^3), 0.22 (9H, s, –Si(CH_3)$_3$).

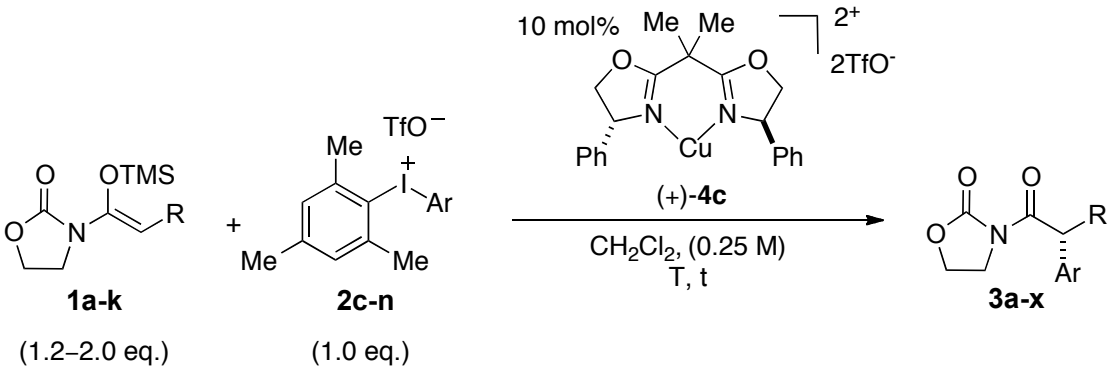
(Z)-3-(2-(4-Bromophenyl)-1-((trimethylsilyl)oxy)vinyl)oxazolidin-2-one **1k**



^{1}H NMR (400 MHz, C_6D_6) δ: 7.39 (2H, d, J = 8.6 Hz, H^7 or H^8), 7.34 (2H, d, J = 8.6 Hz, H^7 or H^8), 5.82 (1H, H^5), 3.43–3.36 (2H, m, H^2), 2.95–2.90 (2H, m, H^3), 0.33 (9H, s, –Si(CH_3)$_3$).

IV. α-Arylation of silyl ketenimides

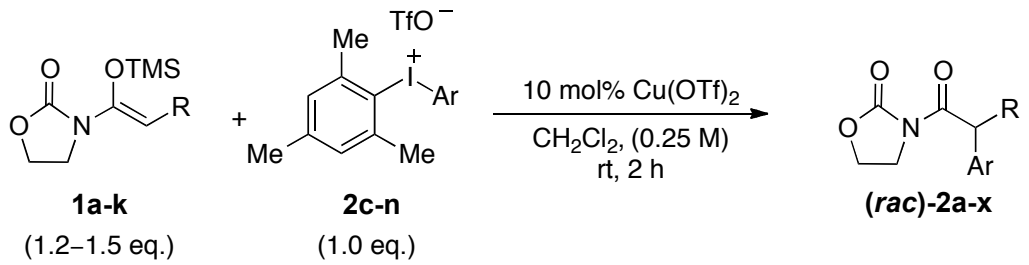
General Procedure C: Enantioselective α-Arylation of silyl ketenimides 1a-1k



An oven dried microwave tube was charged with the appropriate *mesitylaryliodonium triflate* **2** (0.50 mmol, 1.0 eq.). The tube was sealed and back filled with nitrogen and then dichloromethane was added *via* syringe. A preformed stock solution of **(+)-4c**[6] in dichloromethane (0.1 M, 5-10 mol%) was added *via* syringe followed by the appropriate *silyl ketenimide* **1** and the reaction mixture stirred for the stated time at the stated temperature. On completion, the reaction was quenched by addition of a saturated aqueous solution of $NaHCO_3$. The aqueous phase was extracted into dichloromethane (3 x 10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. The crude product was then purified by column chromatography on silica gel using the noted solvent mixture to afford the desired *α-arylated* N-*acyl-oxazolidin-2-one* **3.**

[6] The stock solution **4c** was prepared as followed: an oven-dried sealable tube was charged with $Cu(OTf)_2$ (245 mg, 0.679 mmol, 1.0 eq.) and MS 4Å (~200 mg), then the tube was sealed and heated to 200 °C for 5 minutes under *vacuum*. After cooling down to room temperature, the tube was back-filled with N_2 and the process was repeated twice. Subsequently, a solution of (+)-2,2′-Isopropylidenebis[(4*R*)-4-phenyl-2-oxazoline] ((+)PhBOX, **lc)** (250 mg, 0.747 mmol, 1.1 eq.) in dichloromethane (6.79 mL) was added and the resulting green solution was stirred overnight at room temperature. The resulting green solution was stored under N_2 in the sealed tube and used as required.

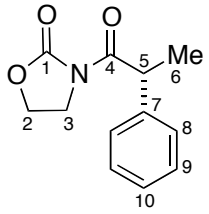
General Procedure D: Racemic α-Arylation of silyl ketenimides



1a-k (1.2–1.5 eq.) **2c-n** (1.0 eq.) **(*rac*)-2a-x**

An oven dried microwave tube was charged with the appropriate *mesitylaryliodonium triflate* **2** (0.50 mmol, 1.0 eq.) and $Cu(OTf)_2$ (10 mol%). The tube was sealed and back filled with nitrogen and then dichloromethane was added *via* syringe followed by the appropriate *silyl ketenimide* **1** and the reaction mixture stirred for 2 hours at room temperature. On completion, the reaction was quenched by addition of a saturated aqueous solution of $NaHCO_3$. The aqueous phase was extracted into dichloromethane (3 x 10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. The crude product was then purified by column chromatography on silica gel using the noted solvent mixture to afford the desired (±)-*α-arylated* N-*acyl-oxazolidin-2-one* **(*rac*)-2**.

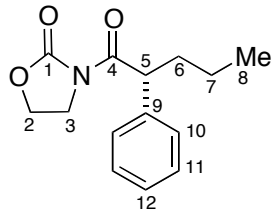
*(*R*)-3-(2-Phenylpropanoyl)oxazolidin-2-one* **3a**



Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1a** (129 mg, 0.60 mmol), **(+)-4c** (0.5 mL, 5 mol%) in dichloromethane (1.75 mL) at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 6:4) provided the title compound (106 mg, 0.48 mmol, 97% yield, 92% *ee*) as a pale yellow semi-solid; IR ν_{max} (film) / cm^{-1} 2980, 2927, 1773, 1694, 1601; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.38–7.36 (2H, m, H^9), 7.33–7.28 (2H, m, H^8), 7.26–7.22 (1H, m, H^{10}), 5.11 (1H, q, J = 7.0 Hz, H^5), 4.40–4.34 (1H, m, H^2), 4.32–4.25 (1H, m, $H^{2'}$), 4.09–4.02 (1H, m, H^3), 3.96–3.89 (1H, m, $H^{3'}$), 1.50 (3H, d, J = 7.0 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.6, 153.0, 140.2, 128.6, 128.1, 127.2, 61.7, 42.9, 42.8, 19.3; HRMS (ESI) calculated for $C_{12}H_{14}NO_3$ [M+H^+] m/z 220.0968, found 220.0969; : $[\alpha]_D^{20}$ = –69.2° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 40% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated

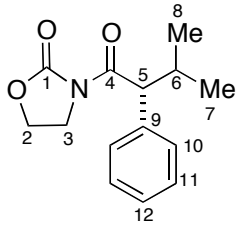
92% *ee*: t_R (minor) = 5.95 minutes, t_R (major) = 6.99 minutes.[7]

(R)-3-(2-Phenylpentanoyl)oxazolidin-2-one **3b**



Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1b** (183 mg, 0.75 mmol), **(+)-4c** (0.5 mL, 10 mol%) in dichloromethane (1.50 mL) at 10 °C for 20 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 6:4) provided the title compound (120 mg, 0.48 mmol, 97% yield, 90% *ee*) as a pale yellow oil; IR ν_{max} (film) / cm^{-1} 2959, 1774, 1694, 1455, 1384, 1285, 1264, 1214; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.36–7.33 (2H, m, H^{11}), 7.28–7.24 (2H, m, H^{10}), 7.22–7.18 (1H, m, H^{12}), 4.99 (1H, t, J = 7.6 Hz, H^5), 4.34–4.29 (1H, m, H^2), 4.26–4.19 (1H, m, $H^{2'}$), 4.04–3.97 (1H, m, H^3), 3.90–3.83 (1H, m, $H^{3'}$), 2.08–1.99 (1H, m, H^6), 1.79–1.70 (1H, m, $H^{6'}$), 1.30–1.15 (2H, m, H^5), 0.86 (3H, t, J = 7.3 Hz, H^8); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.2, 153.0, 138.8, 128.6, 128.4, 127.2, 61.6, 48.1, 42.8, 36.1, 20.6, 13.8; HRMS (CI) calculated for $C_{14}H_{18}NO_3$ [M+H$^+$] m/z 248.1281, found 248.1281; $[\alpha]_D^{20}$ = –50.4° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 40% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 90% *ee*: t_R (minor) = 4.83 minutes, t_R (major) = 5.51 minutes.

*(*R*)-3-(3-Methyl-2-phenylbutanoyl)oxazolidin-2-one* **3c**

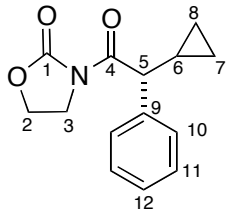


Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1c** (183 mg, 0.75 mmol), **(+)-4c** (0.5 mL, 10 mol%) in dichloromethane (1.5 mL) at 10 °C for 20 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 1:0 to 8:2) provided the title compound (50 mg, 0.20 mmol, 40% yield, 92% *ee*) as a pale yellow oil; IR ν_{max} (solid) / cm^{-1} 2961,

[7] Following hydrolysis of the oxazolidinone according to the method used to prepare **5** (S-31), optical rotation of the corresponding (*R*)-phenylpropionic acid was recorded; $[\alpha]_D^{20}$ = –45.8° (c = 1, $CHCl_3$, commercial ref. = –69.3°).

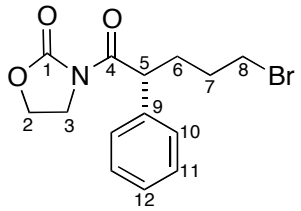
2928, 2872, 1773, 1692; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42–7.39 (2H, m, H^{11}), 7.32–7.30 (2H, m, H^{10}), 7.29–7.22 (1H, m, H^{12}), 4.74 (1H, d, *J* = 10.6 Hz, H^5) 4.39–4.33 (1H, m, H^2), 4.31–4.25 (1H, m, $H^{2'}$), 4.09–4.02 (1H, m, H^3), 3.94–3.88 (1H, m, $H^{3'}$), 2.51–2.42 (1H, m, H^6), 1.03 (1H, d, *J* = 6.6 Hz, H^7 or H^8), 0.70 (1H, d, *J* = 6.6 Hz, H^7 or H^8); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.3, 153.1, 138.0, 129.2, 128.4, 127.3, 61.5, 55.9, 42.8, 32.1, 21.6, 20.1; HRMS (ESI) calculated for $C_{14}H_{18}NO_3$ [M+H$^+$] m/z 248.1281, found 248.1275; $[\alpha]_D^{20}$ = −63.8° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 5% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 92% *ee*: t_R (minor) = 10.41 minutes, t_R (major) = 11.71 minutes.

*(*R*)-3-(2-Cyclopropyl-2-phenylacetyl)oxazolidin-2-one* **3d**



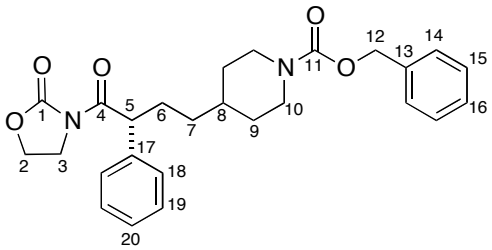
Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1d** (181 mg, 0.75 mmol), **(+)-4c** (0.5 mL, 10 mol%) in dichloromethane (1.50 mL) at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 7:5) provided the title compound (116 mg, 0.47 mmol, 95% yield, 94% *ee*) as off white needles; IR ν_{max} (solid) / cm^{-1} 2923, 2932, 1780, 1683; m.p. 118–120 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.41–7.40 (2H, m, H^{11}), 7.35–7.31 (2H, m, H^{10}), 7.29–7.25 (1H, m, H^{12}), 4.41–4.33 (1H, m, H^2), 4.31–4.27 (2H, m, $H^{2'}$ and H^5), 4.10–4.06 (1H, m, H^3), 3.98–3.92 (1H, m, $H^{3'}$), 1.61–1.54 (1H, m, H^6), 0.66–0.61 (1H, m, H^7 and H^8), 0.52–0.47 (1H, m, H^7 and H^8), 0.35–0.31 (1H, m, H^7 and H^8), 0.29–0.22 (1H, m, H^7 and H^8); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.2, 153.1, 138.7, 128.5, 128.5, 127.3, 61.7, 53.4, 42.8, 15.0, 5.0, 3.9; HRMS (ESI) calculated for $C_{14}H_{16}NO_3$ [M+H$^+$] m/z 246.1125, found 246.1127; $[\alpha]_D^{20}$ = −113.0° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 40% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 94% *ee*: t_R (minor) = 5.15 minutes, t_R (major) = 5.91 minutes.

*(*R*)-3-(5-Bromo-2-phenylpentanoyl)oxazolidin-2-one* **3e**



Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1e** (242 mg, 0.75 mmol), **(+)-4c** (0.5 mL, 10 mol%) in dichloromethane at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 6:4) provided the title compound (155mg, 0.48 mmol, 96% yield, 87% *ee*) as a pale yellow oil; IR ν_{max} (film) / cm^{-1} 2923, 2100, 1772, 1692; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.39–7.36 (2H, m, H^{11}), 7.34–7.30 (2H, m, H^{12}), 7.28–7.24 (1H, m, H^{12}), 5.01 (1H, t, *J* = 7.6 Hz, H^5), 4.41–4.35 (1H, m, H^2), 4.32–4.26 (1H, m, $H^{2'}$), 4.10–4.00 (1H, m, H^3), 3.95–3.85 (1H, m, $H^{3'}$), 3.37 (2H, t, *J* = 6.7 Hz, H^8), 2.26–2.17 (1H, m, H^6), 2.02–1.91 (1H, m, $H^{6'}$), 1.89–1.70 (2H, m, H^7); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.6, 153.0, 138.0, 128.7, 128.6, 127.5, 61.7, 47.7, 42.9, 33.1, 32.4, 30.6; HRMS (ESI) calculated for $C_{14}H_{17}BrNO_3$ [M+H^+] m/z 326.0386, found 326.0386; $[\alpha]_D^{20}$ = –43.3° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 40% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 87% *ee*: t_R (minor) = 5.62 minutes, t_R (major) = 7.67 minutes.

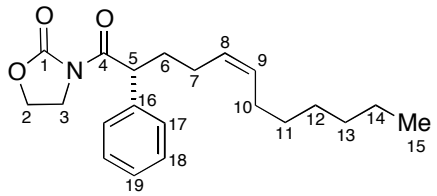
*(*R*)-Benzyl 4-(4-oxo-4-(2-oxooxazolidin-3-yl)-3-phenylbutyl)piperidine-1-carboxylate* **3f**



Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1f** (446 mg, 1.00 mmol), **(+)-4c** (0.30 mL, 10 mol%) in dichloromethane at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 7:4 to 1:1) provided the title compound (214 mg, 0.47 mmol, 95% yield, 94% *ee*) as a colourless oil; IR ν_{max} (film) / cm^{-1} 2922, 2855, 2113, 1776, 1694; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.30–7.17 (10H, m, ArH), 5.03 (2H, bs, H^{12}) 4.89 (1H, t, *J* = 7.6 Hz, H^5), 4.30–4.15 (2H, m, H^2), 4.11–3.91 (4H, m, H^{10} and H^3), 3.85–3.78

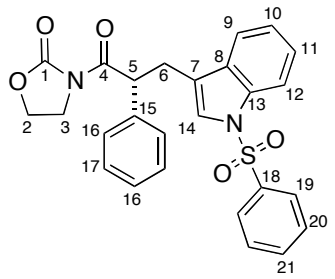
(1H, m, $H^{3'}$), 2.65 (2H, bs, $H^{10'}$), 2.08–1.99 (1H, m, H^{6}), 1.78–1.68 (1H, m, $H^{6'}$), 1.56 (2H, bs, H^{9}), 1.37–1.24 (1H, m, H^{8}), 1.19–0.89 (4H, m, H^{7}, $H^{7'}$ and $H^{9'}$); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.9, 155.1, 153.0, 138.5, 136.9, 129.0, 128.5, 127.8, 127.3, 124.0, 118.1, 66.8, 61.6, 48.5, 44.1, 42.8, 35.8, 34.0, 31.8, 31.1; HRMS (EI) calculated for $C_{26}H_{31}N_2O_5$ [M+H^+] m/z 451.2227, found 451.2225; $[\alpha]_D^{20}$ = –30.2° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 50% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 94% *ee*: t_R (minor) = 15.23 minutes, t_R (major) = 23.83 minutes.

(S*, Z)-3-(2-Phenyldodec-5-enoyl)oxazolidin-2-one* **3g**



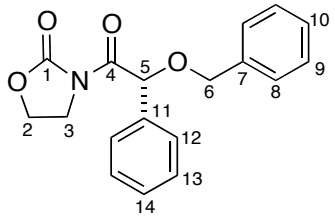
Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1g** (254 mg, 0.75 mmol), **(+)-4c** (0.30 mL, 10 mol%) in dichloromethane at room temperature for 20 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 9:1 to 7:3) provided the title compound (142 mg, 0.41 mmol, 83% yield, 88% *ee*) as a colourless oil; IR ν_{max} (thin film) / cm^{-1} 2923, 2856, 1776, 1695; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.40–7.37 (2H, m, H^{18}), 7.32–7.28 (2H, m, H^{17}), 7.26–7.22 (1H, m, H^{19}), 5.41–5.30 (2H, m, H^{8} and H^{9}), 5.02 (1H, t, *J* = 7.4 Hz, H^{5}), 4.39–4.33 (1H, m, H^{2}), 4.31–4.25 (2H, m, $H^{2'}$), 4.08–4.01 (1H, m, H^{3}), 3.95–3.89 (1H, m, $H^{3'}$), 2.20–2.12 (1H, m, H^{6}), 2.04–1.80 (5H, m, $H^{6'}$, H^{7} and H^{10}), 1.31–1.24 (8H, m, $H^{11\text{-}14}$), 0.88 (3H, t, *J* = 7.0 Hz, H^{15}); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 174.1, 153.0, 138.6, 131.0, 128.7, 128.5, 128.3, 127.3, 61.6, 47.9, 42.9, 33.9, 31.7, 29.6, 28.9, 27.2, 25.1, 22.6, 14.1; HRMS (ESI) calculated for $C_{21}H_{30}NO_3$ [M+NH_4^+] m/z 344.2220, found 344.2227; $[\alpha]_D^{20}$ = –59.7° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 0.5% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 88% *ee*: t_R (minor) = 8.60 minutes, t_R (major) = 11.53 minutes.

*(*R*)-3-(2-phenyl-3-(1-(phenylsulfonyl)-1*H*-indol-3-yl)propanoyl)oxazolidin-2-one* **3h**



Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1h** (363 mg, 0.75 mmol), **(+)-4c** (0.30 mL, 10 mol%) in dichloromethane at 10 °C for 18 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 7:3 to 1:1) provided the title compound (190 mg, 0.40 mmol, 80% yield, 94% *ee*) as a white solid; IR ν_{max} (thin film) / cm^{-1} 2921, 2113, 1775, 1694; m.p. 84 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95–7.92 (1H, m, ArH), 7.75–7.72 (2H, m, ArH), 7.53–7.49 (2H, m, ArH), 7.42–7.34 (4H, m, ArH), 7.32–7.21 (6H, m, ArH), 5.41 (1H, t, *J* = 7.6 Hz, H^5), 4.31–4.21 (1H, m, H^2), 3.99–3.86 (2H, m, H^3), 3.51 (1H, ddd, *J* = 14.6, 8.2 and 0.9 Hz, H^6), 3.11 (1H, ddd, *J* = 14.7, 7.3 and 0.9 Hz, $H^{6'}$); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 173.2, 152.9, 138.2, 137.8, 135.0, 133.5, 130.7, 129.2, 128.7, 127.6, 126.6, 124.7, 124.1, 123.3, 119.9, 119.5, 113.6, 61.7, 48.3, 42.8, 29.3; HRMS (ESI) calculated for $C_{26}H_{26}N_3O_5S$ [M+NH_4^+] m/z 492.1588, found 492.1583; $[\alpha]_D^{20}$ = –41.0° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 30% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 93% *ee*: t_R (minor) = 18.98 minutes, t_R (major) = 22.78 minutes.

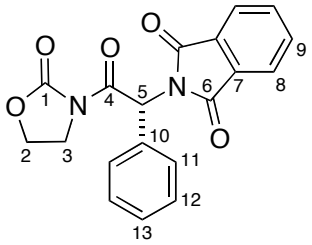
*(*S*)-3-(2-(Benzyloxy)-2-phenylacetyl)oxazolidin-2-one* **3i**



Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1i** (230 mg, 0.75 mmol), **(+)-4c** (0.25 mL, 5 mol%) in dichloromethane at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 5:1 to 2:1) provided the title compound (127 mg, 0.41 mmol, 82% yield, 92% *ee*) as a colourless oil; IR ν_{max} (solid) / cm^{-1} 3031, 2916, 1772, 1703; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57–7.54 (2H, m, Ar*H*), 7.37–7.26 (8H, m, Ar*H*), 6.24 (1H, s, H^5), 4.55 (1H, s, H^6), 4.41–4.35 (1H, m, H^2), 4.34–4.28 (1H, m, $H^{2'}$), 4.03 (1H, ddd,

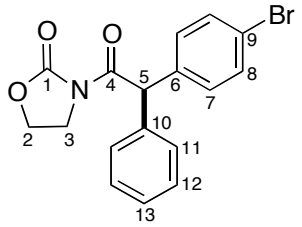
J = 11.0, 7.3 and 2.1 Hz, H^3), 3.92–3.86 (1H, m, $H^{3'}$); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.7, 152.9, 137.2, 135.6, 129.0, 128.7, 128.6, 128.4, 128.3, 128.0, 78.0, 71.5, 62.3, 42.6; HRMS (ESI) calculated for $C_{18}H_{21}N_2O_4$ [M+NH_4^+] m/z 329.1496, found 329.1499; $[\alpha]_D^{20}$ = –53.0° (c = 1.0, $CHCl_3$); HPLC analysis (OD, 20% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 92% *ee*: t_R (minor) = 23.23 minutes, t_R (major) = 33.88 minutes.

*(*R*)-2-(2-Oxo-2-(2-oxooxazolidin-3-yl)-1-phenylethyl)isoindoline-1,3-dione* **3j**



Prepared according to General Procedure C using MesPhIOTf (204 mg, 0.43 mmol), **1j** (300 mg, 0.86 mmol), **(+)-4c** (0.43 mL, 10 mol%) in dichloromethane at 10 °C for 6 hours; flash chromatography on silica gel (dichloromethane:ethyl acetate 15:1) provided the title compound (91 mg, 0.26 mmol, 60% yield, 53% *ee*) as a white solid; IR ν_{max} (film) / cm^{-1} 1776, 1717, 1383; m.p. 88–90 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.83 (2H, dd, J = 5.6 and 3.0 Hz, H^8 or H^9), 7.71 (2H, dd, J = 5.6 and 3.0 Hz, H^8 or H^9), 7.47–7.44 (2H, m, H^{12}), 7.38–7.33 (2H, m, H^{11} and H^{13}), 6.87 (1H, m, H^5), 4.44–4.40 (2H, m, H^2), 4.13 (2H, t, J = 8.1 Hz, H^3); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.7, 167.2, 152.4, 134.2, 133.5, 131.7, 129.5, 128.7, 128.4, 123.6, 62.5, 57.1, 42.8; HRMS (ESI) calculated for $C_{19}H_{15}N_2O_5$ [M+H^+] m/z 351.0975, found 351.980; $[\alpha]_D^{20}$ = +28.8° (c = 1.0, $CHCl_3$); HPLC analysis (IA, 50% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 53% *ee*: t_R (major) = 17.39 minutes, t_R (minor) = 23.12 minutes.

*(*R*)-3-(2-(4-bromophenyl)-2-phenylacetyl)oxazolidin-2-one* **3k**



Prepared according to General Procedure C using MesPhIOTf (236 mg, 0.50 mmol), **1k** (214 mg,

0.60 mmol), **(+)-4c** (0.5 mL, 10 mol%) in dichloromethane at 0 °C for 20 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 6:4) provided the title compound (139 mg, 0.39 mmol, 77% yield, 57% *ee*) as a colourless semi-solid; R_f 0.25 (40–60 petroleum ether:ethyl acetate 8:2); IR ν_{max} (film) / cm^{-1} 2919, 1775, 1697, 1488, 1454, 1387, 1385, 1364, 1278; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.45–7.43 (2H, m, H^8), 7.34–7.30 (4H, m, Ar*H*), 7.29–7.27 (1H, m, Ar*H*), 7.24–7.21 (2H, m, H^7), 6.48 (1H, s, H^5), 4.40–4.37 (2H, m, H^2), 4.09–4.05 (2H, m, H^3); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 171.9, 152.9, 137.7, 137.3, 131.7, 130.9, 129.0, 128.7, 127.6, 121.5, 61.8, 53.4, 43.0; HRMS (ESI) calculated for $C_{17}H_{15}BrNO_3$ [M+H^+] m/z 360.0234, found 283.9919; HPLC analysis (IC, 5% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 58% *ee*: t_R (minor) = 21.74 minutes, t_R (major) = 26.02 minutes.

The product (139 mg, 0.39 mmol) was dissolved in hot methanol (2.5 mL) and dichloromethane (0.2 mL) and the mixture allowed to cool overnight. Filtration of the resulting crystals followed by washing with cold MeOH provided the title compound (90 mg, 0.25 mmol, 50% yield, 92% *ee*) as colourless crystals; m.p. 136–138 °C; $[\alpha]_D^{20}$ = −14.6° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 5% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 92% *ee*: t_R (minor) = 21.79 minutes, t_R (major) = 25.90 minutes.[8]

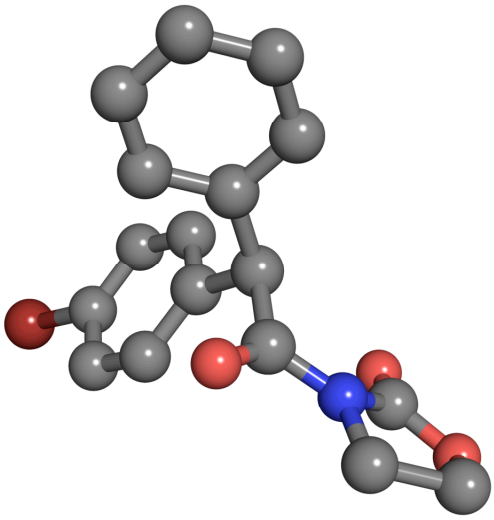
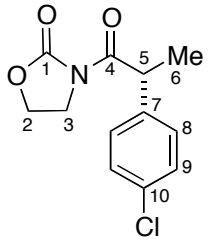


Figure 1: X-Ray Crystal structure of 3k

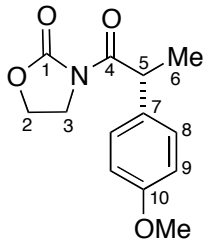
[8] The absolute configuration was determined by X-ray diffraction: **CCDC 831502**.

*(*R*)-3-(2-(4-chlorophenyl)propanoyl)oxazolidin-2-one* **3l**



Prepared according to General Procedure C using 4-chlorophenyl(mesityl)iodonium trifluoromethanesulfonate (253 mg, 0.50 mmol), **1a** (162 mg, 0.75 mmol), **(+)-4c** (0.25 mL, 5 mol%) in dichloromethane at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 1:1) provided the title compound (107 mg, 0.42 mmol, 84% yield, 94% *ee*) as a colourless oil; IR ν_{max} (film) / cm^{-1} 2984, 2928, 1773, 1694; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.32–7.26 (4H, m, H^8 and H^9), 5.08 (1H, q, J = 6.9 Hz, H^5), 4.47–4.28 (2H, m, H^2), 4.09–4.02 (1H, m, H^3), 3.96–3.90 (1H, m, $H^{3'}$), 1.48 (3H, d, J = 6.9 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.3, 153.0, 133.1, 129.6, 128.7, 61.8, 42.9, 42.3, 29.7, 19.3; HRMS (CI) calculated for $C_{14}H_{13}NO_3Cl$ [M+H^+] m/z 254.0578, found 254.0582; $[\alpha]_D^{20}$ = –64.7° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 10% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 94% *ee*: t_R (minor) = 10.26 minutes, t_R (major) = 11.22 minutes.

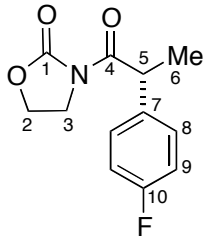
*(*R*)-3-(2-(4-methoxyphenyl)propanoyl)oxazolidin-2-one* **3m**



Prepared according to General Procedure C using 4-methoxyphenyl(mesityl)iodonium trifluoromethanesulfonate (251 mg, 0.50 mmol), **1a** (162 mg, 0.75 mmol), **(+)-4c** (0.25 mL, 5 mol%) in dichloromethane (1.75 mL) at room temperature for 2 hours; flash chromatography on silica gel (toluene/ethyl acetate 19:1 to 8:2) provided the title compound (117 mg, 0.47 mmol, 94% yield, 92% *ee*) as a pale brown oil; IR ν_{max} (film) / cm^{-1} 2972, 2929, 2829, 1772, 1693, 1609; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.31–7.29 (2H, m, H^8), 6.86–6.82 (2H, m, H^9), 5.05 (1H, q, J = 6.9 Hz, H^5), 4.40–4.26 (2H, m, H^2), 4.11–4.06 (1H, m, H^3), 4.07–3.96 (1H, m, $H^{3'}$), 3.78 (3H, m, –OMe), 1.47 (3H, d, J = 6.9 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 175.0, 158.8, 153.0, 132.3, 129.2, 114.0, 61.7, 55.2, 42.9, 41.9, 19.3; HRMS (ESI) calculated for $C_{13}H_{16}NO_4$ [M+H^+] m/z 250.1074,

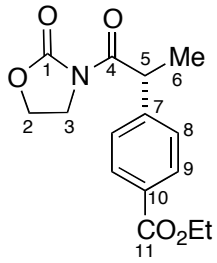
found 250.1076; $[\alpha]_D^{20}$ = –49.9° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 15% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 92% *ee*: t_R (minor) = 15.75 minutes, t_R (major) = 17.91 minutes.

*(*R*)-3-(2-(4-fluorophenyl)propanoyl)oxazolidin-2-one* **3n**



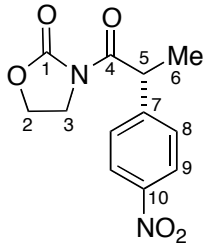
Prepared according to General Procedure C using 4-fluorophenyl(mesityl)iodonium trifluoromethanesulfonate (245 mg, 0.50 mmol), **1a** (162 mg, 0.75 mmol), **(+)-4c** (0.25 mL, 5 mol%) in dichloromethane (1.75 mL) at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether/ethyl acetate 19:1 to 8:2) provided the title compound (104 mg, 0.44 mmol, 88% yield, 93% *ee*) as a pale yellow oil; IR ν_{max} (film) / cm^{-1} 1772, 1694, 1602, 1508; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.36–7.32 (2H, m, H^9), 7.01–6.97 (2H, m, H^8), 5.09 (1H, q, *J* = 7.1 Hz, H^5), 4.41–4.28 (2H, m, H^2), 4.09–4.02 (1H, m, H^3), 3.96–3.90 (1H, m, $H^{3'}$), 1.48 (3H, d, *J* = 7.1 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.5, 161.8 (1C, d, $J_{C\text{-}F}$ = 246 Hz, C^{10}), 153.0, 135.9 (1C, d, $J_{C\text{-}F}$ = 3.2 Hz, C^7), 129.8 (2C, d, $J_{C\text{-}F}$ = 8.0 Hz, C^8), 115.4 (2C, d, $J_{C\text{-}F}$ = 21.4 Hz, C^9), 61.8, 42.8, 42.0, 19.4; ^{19}F NMR (376 MHz, $CDCl_3$) δ: –115.7 (1F, s); HRMS (ESI) calculated for $C_{15}H_{18}NO_5$ [M+H^+] m/z 238.0874, found 238.0879; $[\alpha]_D^{20}$ = –59.8° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 15% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 93 % *ee*: t_R (minor) = 8.03 minutes, t_R (major) = 8.56 minutes.

*(*R*)-ethyl 4-(1-oxo-1-(2-oxooxazolidin-3-yl)propan-2-yl)benzoate* **3o**



Prepared according to General Procedure C using (4-(ethoxycarbonyl)phenyl) (mesityl)iodonium trifluoromethanesulfonate (272 mg, 0.50 mmol), **1a** (162 mg, 0.75 mmol), **(+)-4c** (0.25 mL, 5 mol%) in dichloromethane (1.75 mL) at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether/ethyl acetate 19:1 to 8:2) provided the title compound (138 mg, 0.47 mmol, 95% yield, 92% *ee*) as a colourless oil; IR ν_{max} (film) / cm^{-1} 2985, 2937, 2333, 1774, 1698; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.99–7.96 (2H, m, H^9), 7.44–7.41 (2H, m, H^8), 5.14 (1H, q, J = 6.8 Hz, H^5), 4.37–4.27 (4H, m, H^2 and -$CO_2CH_2CH_3$), 4.09–4.02 (1H, m, H^3), 3.97–3.90 (1H, m, $H^{3'}$), 1.48 (3H, d, J = 6.7 Hz, H^6), (2H, t, J = 7.2 Hz, -$CO_2CH_2CH_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.9, 166.3, 152.9, 145.2, 129.8, 129.5, 128.1, 61.8, 60.9, 43.0, 42.9, 19.2, 14.3; HRMS (ESI) calculated for $C_{15}H_{18}NO_5$ [M+H^+] m/z 292.1179, found 292.1185; $[\alpha]_D^{20}$ = –64.0° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 15% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 92 % *ee*: t_R (minor) = 42.49 minutes, t_R (major) = 46.15 minutes.

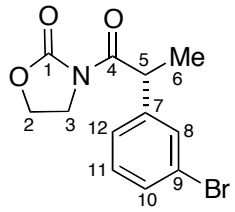
*(*R*)-3-(2-(4-nitrophenyl)propanoyl)oxazolidin-2-one* **3p**



Prepared according to General Procedure C using 4-nitrophenyl(mesityl)iodonium trifluoromethanesulfonate (259 mg, 0.50 mmol), **1c** (162 mg, 0.75 mmol), **(+)-4c** (0.25 mL, 5 mol%) in dichloromethane (3.75 mL, 0.5 M) at room temperature for 4 hours; flash chromatography on silica gel (toluene/ethyl acetate 19:1 to 8:2) provided the title compound (107 mg, 0.42 mmol, 68% yield, 92% *ee*) as a yellow oil; IR ν_{max} (film) / cm^{-1} 1771, 1693, 1605, 1518; ^{1}H

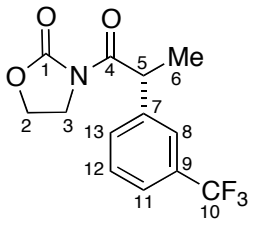
NMR (400 MHz, $CDCl_3$) δ: 8.18–8.14 (2H, m, H^9), 7.56–7.52 (2H, m, H^8), 5.20 (1H, q, *J* = 7.0 Hz, H^5), 4.45–4.32 (2H, m, H^2), 4.17–4.05 (1H, m, H^3), 4.00–3.93 (1H, m, $H^{3'}$), 1.53 (3H, d, *J* = 7.0 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.3, 153.0, 147.5, 147.1, 129.1, 123.8, 61.9, 42.9, 42.9, 19.2; HRMS (CI) calculated for $C_{12}H_{13}N_2O_5$ [M+H$^+$] m/z 265.0819, found 265.0817; $[\alpha]_D^{20}$ = –62.2° (c = 1, $CHCl_3$); HPLC analysis (IC, 15% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 92% *ee*: t_R (major) = 30.37 minutes, t_R (minor) = 33.47 minutes.

*(*R*)-3-(2-(3-bromophenyl)propanoyl)oxazolidin-2-one* **3q**



Prepared according to General Procedure C using 3-bromophenyl(mesityl)iodonium trifluoromethanesulfonate (278 mg, 0.50 mmol), **1a** (162 mg, 0.75 mmol), **(+)-4c** (0.25 mL, 5 mol%) in dichloromethane (1.75 mL) at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether/ethyl acetate 19:1 to 8:2) provided the title compound (104 mg, 0.44 mmol, 87% yield, 87% *ee*) as a white oil; IR ν_{max} (film) / cm^{-1} 2984, 2928, 1772, 1694; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.51 (1H, t, *J* = 1.9 Hz, H^8), 7.51 (1H, ddd, *J* = 7.9, 1.9 and 1.1 Hz, H^{10}), 7.31 (1H, app dt, *J* = 7.8 and 1.3 Hz, H^{12}), 7.31 (1H, t, *J* = 7.9 Hz, H^{11}), 5.08 (1H, q, *J* = 7.1 Hz, H^5), 4.41–4.30 (2H, m, H^2), 4.10–4.01 (1H, m, H^3), 3.99–3.93 (1H, m, $H^{3'}$), 1.49 (3H, d, *J* = 7.0 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.0, 153.0, 142.4, 131.0, 130.4, 130.1, 127.1, 122.6, 61.8, 42.9, 42.5, 19.3; HRMS (CI) calculated for $C_{12}H_{13}NO_3Br$ [M+H$^+$] m/z 298.0073, found 298.0075; $[\alpha]_D^{20}$ = –44.3° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 20% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 87% *ee*: t_R (minor) = 7.30 minutes, t_R (major) = 8.70 minutes.

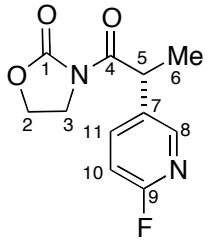
*(*R*)-3-(2-(3-(trifluoromethyl)phenyl)propanoyl)oxazolidin-2-one* **3r**



Prepared according to General Procedure C using 3-trifluoromethylphenyl(mesityl)iodonium trifluoromethanesulfonate (270 mg, 0.50 mmol), **1a** (161 mg, 0.75 mmol), **(+)-4c** (0.25 mL,

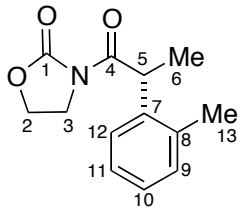
5 mol%) in dichloromethane at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 19:1 to 3:1) provided the title compound (121 mg, 0.42 mmol, 84% yield, 91% *ee*) as a pale yellow oil; IR ν_{max} (film) / cm^{-1} 2986, 2937, 1775, 1697; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.61 (1H, s, H^8), 7.56 (1H, d, J = 7.7 Hz, H^{11}), 7.50 (1H, d, J =7.7 Hz, H^{13}), 7.42 (1H, t, J = 7.5 Hz, H^{12}), 5.16 (1H, q, J = 6.8 Hz, H^5), 4.40–4.24 (2H, m, H^2), 4.08–4.01 (1H, m, H^3), 3.97–3.91 (1H, m, $H^{3'}$), 1.51 (3H, d, J = 7.0 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.9, 153.0, 141.1, 131.8, 129.0, 124.8 (m, $J_{C\text{-}F}$ = 3.5 Hz), 124.1 (m, $J_{C\text{-}F}$ = 3.5 Hz), 61.8, 42.8, 42.7, 19.3, (C^{10} not observed); ^{19}F NMR (376 MHz, $CDCl_3$) δ: –62.74; HRMS (CI) calculated for $C_{13}H_{13}F_3NO_3$ [M+H^+] m/z 288.0842, found 288.0837; $[\alpha]_D^{20}$ = –47.7° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 15% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 91% *ee*: t_R (minor) = 6.21 minutes, t_R (major) = 6.98 minutes.

*(*R*)-3-(2-(6-fluoropyridin-3-yl)propanoyl)oxazolidin-2-one* **3s**



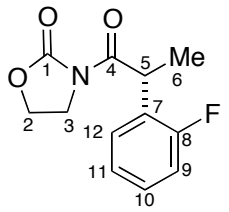
Prepared according to General Procedure C using 6-fluoropyridin-3-yl)(mesityl)iodonium trifluoromethanesulfonate (245 mg, 0.5 mmol), **1a** (215 mg, 1.0 mmol), **(+)-4c** (1.0 mL, 20 mol%) in dichloromethane at room temperature for 20 hours; flash chromatography on silica gel (toluene:ethyl acetate 7:3) provided the title compound (45 mg, 0.19 mmol, 38% yield, 47% *ee*) as a brown oil; IR ν_{max} (film) / cm^{-1} 1769, 1693, 1596, 1479, 1386, 1363; ^1H NMR (400 MHz, $CDCl_3$) δ: 8.20 (1H, d, J = 2.5 Hz, H^8), 7.83 (1H, app dt, J = 8.3 and 2.5 Hz, H^{11}), 6.89 (1H, dd, J = 8.3 and 3.0 Hz, H^{10}), 4.45–4.33 (2H, m, H^2), 4.11–4.03 (1H, m, H^3), 4.00–3.93 (1H, m, $H^{3'}$), 1.52 (3H, d, J = 7.1 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.8, 164.0 (app s), 153.0, 147.3 (d, $J_{C\text{-}F}$ = 14.9 Hz), 141.1 (d, $J_{C\text{-}F}$ = 8.2 Hz), 133.5 (d, $J_{C\text{-}F}$ = 4.7 Hz), 109.5 (d, $J_{C\text{-}F}$ = 37.7 Hz), 62.9, 42.9, 39.8, 19.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ: –70.1 (1F, s); HRMS (CI) calculated for $C_{13}H_{13}F_3NO_3$ [M+H^+] m/z 288.0842, found 288.0837; $[\alpha]_D^{20}$ = –17.0° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 30% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 47% *ee*: t_R (minor) = 21.35 minutes, t_R (major) = 23.50 minutes.

*(*R*)-3-(2-(o-tolyl)propanoyl)oxazolidin-2-one* **3t**



Prepared according to General Procedure C using 2-methylphenyl(mesityl)iodonium trifluoromethanesulfonate (243 mg, 0.50 mmol), **1a** (162 mg, 0.75 mmol), **(+)-4c** (0.25 mL, 5 mol%) in dichloromethane (1.75 mL) at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether/ethyl acetate 19:1 to 8:2) provided the title compound (68 mg, 0.29 mmol, 58% yield, 17% *ee*) as a pale brown oil; IR ν_{max} (film) / cm^{-1} 2976, 2929, 1774, 1693; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.19–7.08 (4H, m, ArH), 5.14 (1H, q, J = 7.0 Hz, H^5), 4.40–4.26 (2H, m, H^2), 4.13–4.06 (1H, m, H^3), 4.02–3.06 (1H, m, $H^{3'}$), 2.44 (3H, s, 1.49, H^{13}), 1.42 (3H, d, J = 7.0 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.9, 152.9, 139.0, 136.5, 130.7, 127.0, 126.2, 125.6, 61.8, 43.0, 40.4, 19.2, 18.2; HRMS (EI) calculated for $C_{13}H_{16}NO_3$ [M+H^+] m/z 234.1125, found 234.1127; $[\alpha]_D^{20}$ = –42.0° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 30% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 17% *ee*: t_R (minor) = 6.36 minutes, t_R (major) = 8.06 minutes.

*(*R*)-3-(2-(2-fluorophenyl)propanoyl)oxazolidin-2-one* **3u**

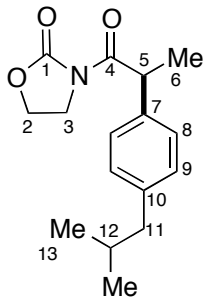


Prepared according to General Procedure C using 2-fluorophenyl(1,3,5-triisopropylbenzene)iodonium trifluoromethanesulfonate[9] (287 mg, 0.5 mmol), **1a** (215 mg, 1.0 mmol), **(+)-4c** (0.5 mL, 10 mol%) in dichloromethane (2.63 mL, 0.19 M) at 10 °C for 20 hours; flash chromatography on silica gel (ethyl acetate in toluene, 0–5%) provided the title compound (105 mg, 0.44 mmol, 89% yield, 91% *ee*) as a colourless oil; IR ν_{max} (film) / cm^{-1} 1773, 1695, 1585, 1492, 1452; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28–7.21 (2H, m, ArH), 7.11 (1H, dt, J = 7.5 and

[9] 2-fluorophenyl(1,3,5-**triisopropylbenzene**)iodonium trifluoromethanesulfonate was used as we observed some mesityl transfer when 2-fluorophenyl(**mesityl**)iodonium trifluoromethanesulfonate was employed.

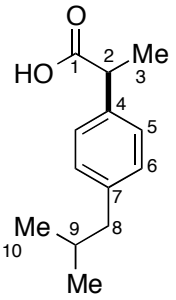
1.2 Hz, ArH) 7.06–7.02 (1H, m, H^9), 5.25 (1H, q, J = 7.1 Hz, H^5), 4.43–4.31 (2H, m, H^2), 4.11–3.97 (2H, m, H^3), 1.51 (3H, d, J = 7.1 Hz, H^6); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.0, 160.4 (1C, d, $J_{C\text{-}F}$ = 248 Hz, C^8), 152.8, 128.7 (1C, d, $J_{C\text{-}F}$ = 8.4 Hz), 128.5 (1C, d, $J_{C\text{-}F}$ = 4.0 Hz), 127.5 (1C, d, $J_{C\text{-}F}$ = 15.0 Hz, C^7), 124.1 (1C, d, $J_{C\text{-}F}$ = 3.5 Hz) 115.4 (1C, d, $J_{C\text{-}F}$ = 22.0 Hz, C^9), 61.9, 42.8, 37.3 (1C, d, $J_{C\text{-}F}$ = 2.4 Hz, C^5), 17.5; ^{19}F NMR (376 MHz, $CDCl_3$) δ: –117.5 (1F, s); HRMS (EI) calculated for $C_{12}H_{13}NO_3F$ [M+H$^+$] m/z 238.0874, found 238.0878; $[\alpha]_D^{20}$ = –82.0° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 15% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 91% *ee*: t_R (minor) = 13.32 minutes, t_R (major) = 17.32 minutes.

*(*S*)-3-(2-(4-isobutylphenyl)propanoyl)oxazolidin-2-one* **3v**



Prepared according to General Procedure C using 4-isobutylphenyl(mesityl)iodonium trifluoromethanesulfonate (3.77 g, 7.15 mmol), **1a** (1.84 g, 8.54 mmol), **(–)-4c** (1.43 mL, 2.0 mol%) in dichloromethane at room temperature for 6 hours; flash chromatography on silica gel (40–60 petroleum ether/ethyl acetate 9:1 to 7:3) provided the title compound (1.95 g, 7.08 mmol, 99% yield, 93% *ee*) as a white solid; IR ν_{max} (film) / cm^{-1} 2956, 2935, 2871, 1766, 1698; m.p. 66 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28–7.25 (2H, m, H^8), 7.10–7.06 (2H, m, H^9), 5.09 (1H, q, 7.0 Hz, H^5), 4.40–4.26 (2H, m, H^2), 4.09–4.02 (1H, m, H^3), 3.97–3.90 (1H, m, $H^{3'}$), 2.43 (2H, d, J = 7.1 Hz, H^{11}), 1.84 (1H, sp, J = 6.8 Hz, H^{11}), 1.48 (3H, d, J = 7.0 Hz, H^6), 0.89 (6H, d, J = 6.6 Hz, H^{13}); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 174.9, 153.0, 140.7, 137.4, 129.3, 127.8, 61.7, 45.1, 42.9, 42.4, 30.1, 22.1, 19.3; HRMS (ESI) calculated for $C_{16}H_{22}NO_3$ [M+H$^+$] m/z 276.1594, found 276.1591; $[\alpha]_D^{20}$ = +53.6° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 20% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 93 % *ee*: t_R (major) = 7.01 minutes, t_R (minor) = 7.81 minutes.

(S)-2-(4-isobutylphenyl)propanoic acid **5**

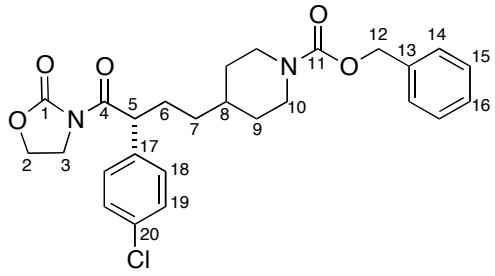


To a solution of **3x** (140 mg, 0.50 mmol) in THF (30 mL) and H_2O (5.5 mL) at 0 °C was added dropwise H_2O_2 (0.57 mL, 5.0 mmol, 30% in H_2O) followed by a solution of LiOH (127 mg, 3.0 mmol) in H_2O (2.0 mL) and the reaction mixture was stirred at this temperature for 2 hours. The reaction was quenched with a sat. aqueous solution of $Na_2S_2O_3$ (20 mL), further stirred for 10 minutes at 0 °C, then concentrated *in vacuo* to remove most of the THF. The resulting basic aqueous solution was washed with ethyl acetate (20 mL), then acidified at 0 °C with conc. HCl to pH = 1 and extracted with diethyl ether (3 x 30 mL). The combined organic extracts were dried over Na_2SO_4 and evaporated to provide the title compound (96 mg, 0.46 mmol, 92%) as a white solid; IR ν_{max} (film) / cm^{-1} 2953, 2928, 2867, 1703; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.25–7.21 (2H, m, H^5), 7.14–7.08 (2H, m, H^6), 3.72 (1H, q, J = 7.2 Hz, H^2), 2.46 (2H, d, J = 7.1 Hz, H^8), 1.86 (1H, sp, J = 6.8 Hz, H^9), 1.52 (3H, d, J = 7.2 Hz, H^3), 0.91 (6H, d, J = 6.6 Hz, H^{10}); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 181.0, 140.8, 137.0, 129.4, 127.3, 45.0, 44.0, 30.1, 22.4, 18.1; $[\alpha]_D^{20}$ = +46.7° (c = 1.0, $CHCl_3$, Lit. = +45.4°).[10]
Data in agreement with commercial reference.

To determine enantiomeric excess the corresponding methyl ester was prepared by treating a solution of the acid (1.0 eq.) in ethyl acetate (0.20 M) with $TMSCH_2N_2$ (10.0 eq.) at 0 °C. After 5 minutes the reaction was quenched by addition of HCl (1 M) until the solution was colourless. The organic layer was separated, dried with Na_2SO_4 and concentrated *in vacuo* to provide (*S*)-ibuprofen methyl ester. HPLC analysis of the ester (AD-H, 0.5% *i*-PrOH in *n*-hexane, 0.5 mL/min, 254 nm) indicated 93% ee: t_R (major) = 10.76 minutes, t_R (minor) = 12.17 minutes.

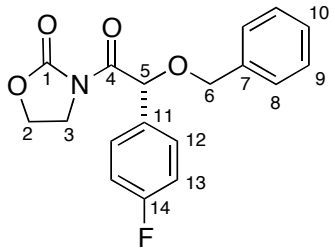
[10] Piccolo, O.; Spreafico, F.; Visentin, G.; Valoti. E. *J. Org. Chem.,* **1987***, 52*, 10.

*(*R*)-benzyl 4-(3-(4-chlorophenyl)-4-oxo-4-(2-oxooxazolidin-3-yl)butyl)piperidine-1-carboxylate* **3w**



Prepared according to General Procedure C using 4-chlorophenyl(mesityl)iodonium trifluoromethanesulfonate (152 mg, 0.30 mmol), **1a** (268 mg, 0.60 mmol), **(+)-4c** (0.30 mL, 10 mol%) in dichloromethane at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether:ethyl acetate 7:4 to 1:1) provided the title compound (140 mg, 0.29 mmol, 96% yield, 95% *ee*) as colourless crystals); IR ν_{max} (film) / cm^{-1} 2922, 2857, 1777, 1694; m.p. 40–42 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28–7.18 (9H, m, ArH), 5.02 (2H, s, H^{12}) 4.85 (1H, t, J = 7.4 Hz, H^5), 4.33–4.19 (2H, m, H^2), 4.05 (2H, bs, H^{10}), 4.00–3.93 (1H, m, H^3), 3.87–3.80 (1H, m, $H^{3'}$), 2.64 (2H, bs, $H^{10'}$), 2.05–1.95 (1H, m, H^6), 1.74–1.64 (1H, m, $H^{6'}$), 1.54 (2H, bs, H^9), 1.35–1.25 (1H, m, H^8), 1.18–1.07 (1H, m, H^7), 1.06–0.95 (3H, m, $H^{7'}$ and $H^{9'}$); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 173.7, 155.2, 153.0, 136.9, 136.9, 133.3, 130.0, 128.7, 128.4, 127.9, 127.8, 66.9, 61.7, 47.9, 44.1, 42.8, 35.8, 34.0, 31.9, 31.1; HRMS (EI) calculated for $C_{26}H_{33}ClN_3O_5$ [M+NH^+] m/z 502.2095, found 502.2099; $[\alpha]_D^{20}$ = −39.7° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 50% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 95% *ee*: t_R (minor) = 12.71 minutes, t_R (major) = 15.29 minutes.

*(*R*)-3-(2-(benzyloxy)-2-(4-fluorophenyl)acetyl)oxazolidin-2-one* ***3x***



Prepared according to General Procedure C using 4-fluorophenyl(mesityl)iodonium trifluoromethanesulfonate (245 mg, 0.50 mmol), **1i** (231 mg, 0.75 mmol), **(+)-4c** (0.50 mL, 10 mol%) in dichloromethane at room temperature for 2 hours; flash chromatography on silica gel (40–60 petroleum ether/ethyl acetate 9:1 to 6:4) provided the title compound (122 mg, 0.39 mmol,

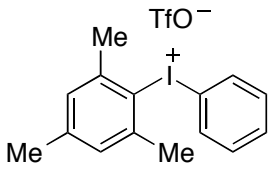
74% yield, 91% *ee*) as a colourless oil; IR ν_{max} (film) / cm^{-1} 1774, 1703, 1603, 1508; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.55–7.52 (2H, m, H^{12}), 7.36–7.27 (5H, m, H^{8-10}), 7.08–7.02 (2H, m, H^{13}), 6.20 (1H, s, H^{5}), 4.54 (2H, d, J = 0.81 Hz, H^{6}), 4.43–4.30 (2H, m, H^{2}), 4.07–4.00 (1H, m, H^{3}), 3.93–3.87 (1H, m, $H^{3'}$); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.6, 163.1 (1C, d, $J_{C\text{-}F}$ = 247 Hz, C^{10}), 153.0, 137.1, 131.5 (1C, d, $J_{C\text{-}F}$ = 2.9 Hz, C^{7}), 130.6 (2C, d, $J_{C\text{-}F}$ = 8.3 Hz, C^{8}), 128.4 128.3, 128.0, 115.6 (2C, d, $J_{c\text{-}F}$ = 21.2 Hz, C^{9}), 77.2, 71.5, 62.4, 42.6; ^{19}F NMR (376 MHz, $CDCl_3$) δ: -112.7 (1F, s); HRMS (ESI) calculated for $C_{18}H_{20}FN_2O_4$ $[M+NH_4^+]$ m/z 347.1402, found 347.1407; $[\alpha]_D^{20}$ = –45.0° (c = 1.0, $CHCl_3$); HPLC analysis (IC, 15% *i*-PrOH in *n*-hexane, 1.0 mL/min, 210 nm) indicated 91 % *ee*: t_R (major) = 10.71 minutes, t_R (minor) = 14.67 minutes.

V. Preparation of diaryliodonium salts[11]

General Procedure E: Preparation of Aryl(mesityl)iodonium trifluoromethane sulfonates

To a solution of the appropriate *iodoarene* (45.0 mmol) and *m*CPBA (dried under vacuum at room temperature for 1 hour, assume 65%) 13.2g) in dichloromethane (120 ml) at 0 ˚C was added trifluoromethanesulfonic acid (6.6 mL) dropwise over 2 mins. The ice bath was then removed and the reaction stirred for 2 hours at room temperature. It was then cooled to 0 ˚C and mesitylene (7.0 ml) was added dropwise over 2 minutes. The reaction was allowed to warm to room temperature and stirred overnight. The solvent was removed *in vacuo* and diethyl ether added. The resulting solid was filtered and washed on the filter with diethyl ether to give the iodonium triflate as a solid that was dried at 100 ˚C under vacuum for 1 hour.

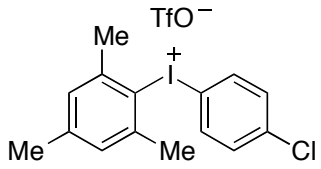
Phenyl(mesityl)iodonium trifluoromethanesulfonate **2c**



Diphenyliodonium acetate (29.0 g, 90.0 mmol) and mesitylene (25.0 mL, 180 mmol) were suspended in dichloromethane (450 mL) and cooled to 0 ˚C. TfOH (16 mL, 180 mmol) was added slowly at 0 ˚C and the reaction mixture was allowed to warm to room temperature whilst stirring for 1 hour. The reaction mixture was then concentrated *in vacuo* to give a red oil. The residue was triturated in diethyl ether, placed at the sonicator for 15 minutes and then stored in the freezer for 2.5 hours. The cold suspension was then filtered and washed with cold diethyl ether (× 3) before being left to dry under vacuum (0.2 mbar) overnight to furnish the title compound as a white solid (41.6 g, 88.0 mmol, 98% yield) ; IR ν_{max} (solid) / cm^{-1} 1474, 1444, 1382, 1249, 1222, 1158; m.p 137–138 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.71–7.67 (2H, m), 7.54–7.50 (1H, m), 7.42–7.37 (2H, m), 7.09 (2H, s), 2.61 (6H, s), 2.34 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.4, 142.5, 133.0, 132.2, 131.7, 130.3, 120.3, 111.7, 27.0, 21.0; HRMS (ESI) calculated for $C_{15}H_{16}I$ [M-TfO^-] m/z 323.0297, found 323.0309.

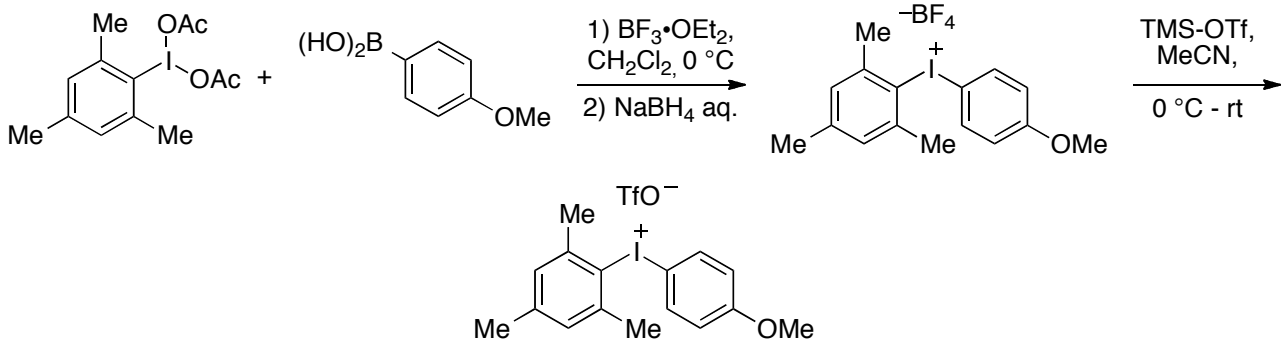
[11] For references on preparation see: a) Bielawski, M.; Olofsson, B.,*Chem. Commun.* **2007**, 2521; b) Bielawski, M.; Zhu, M.; Olofsson, B., *Adv. Synth. Cat.* **2007**, *349*, 2610; c) Phipps, R. J.; Gaunt, M. J., *Science* **2009**, *323*, 1593.

4-Chlorophenyl(mesityl)iodonium trifluoromethanesulfonate **2d**



Prepared according to General Procedure E from 4-chloroiodobenzene. Product obtained as an off-white solid (19.9 g, 39 mmol 87%); IR ν_{max} (solid) / cm^{-1} 1474, 1393, 1244, 1222, 1164; m.p 132–133 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.66–7.62 (2H, m), 7.38–7.34 (2H, m,), 7.10 (2H, s), 2.62 (6H, s), 2.35 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.5, 142.4, 134.3, 132.2, 130.2, 27.0, 21.1; HRMS (ESI) calculated for $C_{15}H_{15}ClI$ [M-TfO^-] m/z 356.9907, found 356.9920.

4-Methoxyphenyl(mesityl)iodonium trifluoromethanesulfonate **2e**

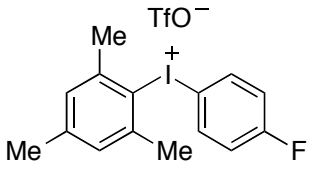


2e

To a stirred solution of 4-methoxy boronic acid (2.35 g, 15.5 mmol) in dichloromethane (150 mL) at 0 ˚C was added BF_3•OEt_2 (2.09 mL, 17.0 mmol). To this was added a solution of iodomesitylene diacetate (6.19 g, 17.0 mmol) in dichloromethane (60 mL) and the solution stirred at 0 ˚C for 2 hours before the addition of a saturated aqueous solution of sodium tetrafluoroborate (300 mL) with fast stirring. After 30 minutes, the phases were separated and the aqueous phase extracted with dichloromethane. The combined organic phases were dried over $MgSO_4$ and the solvent evaporated *in vacuo*. The crude residue was dissolved in hot dichloromethane and addition of diethyl ether led to the precipitation of a white solid. The solid was filtered, washed with diethyl ether and dried under vacuum to give 4-methoxyphenyl(mesityl)iodonium tetrafluoroborate as a white solid (5.4 g, 12.3 mmol, 80% yield); m.p. 150-152 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.84 (2H, d, J = 9.2 Hz), 7.20 (2H, s), 7.03 (2H, d, J = 9.2 Hz,), 3.82 (3H, s), 2.66 (6H, s), 2.33 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.3, 145.7, 143.3, 137.5, 131.3, 119.0, 102.6, 56.4, 27.0, 21.0; HRMS (ESI) calculated for $C_{16}H_{18}OI$ [M-BF_4]$^+$ m/z 353.0402, found 353.0395.

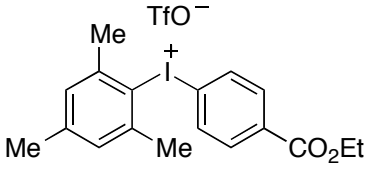
TMS-OTf (3.34 mL, 18.4 mmol) was added to a stirred solution of 4-methoxyphenyl(mesityl)iodonium tetrafluoroborate as a white solid (5.4 g, 12.3 mmol) in acetonitrile (50 mL) at 0 ˚C and the solution allowed to warm to room temperature. After stirring for 15 hours, the solvent was removed *in vacuo* and the resulting residue taken up in diethyl ether and stirred at –20 ˚C for 24 hours. The resulting crystals were filtered and washed with diethyl ether to give the title compound as an off-white solid (5.95 g, 11.8 mmol, 96% yield); IR ν_{max} (solid) / cm^{-1} 1572, 1486, 1461, 1379, 1273, 1240, 1225, 1176, 1160; m.p. 160-162 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67–7.64 (2H, m,), 7.06 (2H, s), 6.92–6.90 (2H, m), 3.79 (3H, s), 2.63 (6H, s), 2.32 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.4, 144.1, 142.1, 135.4, 130.2, 121.0, 117.9, 99.9, 55.6, 26.9, 20.9; HRMS (ESI) calculated for $C_{16}H_{18}OI$ $[M\text{-}OTf]^+$ m/z 353.0397, found 353.0394.

4-Fluorophenyl(mesityl)iodonium trifluoromethanesulfonate **2f**



Prepared according to General Procedure E from 4-fluoroiodobenzene. Product obtained as an off-white solid (14.6g, 29.7 mmol, 66%); IR ν_{max} (solid) / cm^{-1} 1575, 1482, 1224, 1167; m.p. 177-178 ˚C; ^{1}H NMR (400 MHz, MeOD) δ: 7.98 (2H, dd, J = 4.8 and 9.1 Hz), 7.29 (2H, t, J = 8.8 Hz), 7.25 (2H, s), 2.68 (6H, s), 2.36 (3H, s); ^{13}C NMR (100 MHz, MeOD) δ: 166.2 (d, J = 253 Hz), 145.9, 143.4, 138.1 (d, J = 9.0 Hz), 131.3, 122.7, 120.6 (d, J = 23.4 Hz), 107.8 (d, J = 3.3 Hz), 27.0, 21.0; HRMS (ESI) calculated for $C_{15}H_{15}FI$ $[M\text{-}OTf]^+$ m/z 341.0197, found 341.0193.

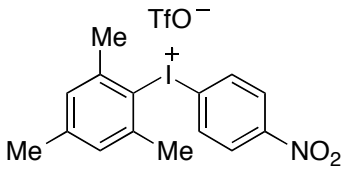
(4-(Ethoxycarbonyl)phenyl)(mesityl)iodonium trifluoromethanesulfonate **2g**



Prepared according to General Procedure E from ethyl 4-iodobenzoate. Product obtained as a white solid (15.7 g, 28.8 mmol 87%); IR ν_{max} (solid) / cm^{-1} 2987, 1722, 1272, 1224; m.p. 178-179 ˚C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.01 (2H, d, J = 8.7 Hz), 7.76 (2H, d, J = 8.7 Hz), 7.10 (2H, s), 4.36 (2H, q, J = 7.1 Hz), 2.61 (6H, s), 2.35 (3H, s), 1.36 (3H, t, J = 7.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.7, 144.5, 142.5, 133.4, 132.7, 132.6, 130.3, 120.6, 116.3, 61.7, 27.0, 21.1, 14.1; HRMS (ESI)

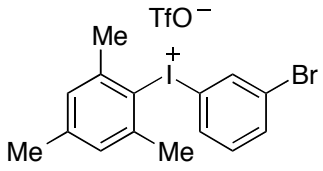
calculated for $C_{18}H_{20}IO_2$ [M-OTf]$^+$ m/z 395.0503, found 395.0498.

4-Nitrophenyl(mesityl)iodonium trifluoromethanesulfonate **2h**



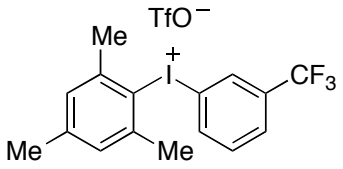
Prepared according to General Procedure E from 4-iodonitrobenzene with the exception that all of the reagents apart from the trifluoromethanesulfonic acid were stirred together at room temperature for 20 hours before the addition of the latter reagent. Product obtained as a light yellow solid (14.0 g, 27.1 mmol, 60%); IR ν_{max} (solid) / cm^{-1} 1534, 1355, 1236, 1224; m.p. 208 °C; ^{1}H NMR (400 MHz, MeOD) δ: 8.30 (2H, d, *J* = 9.1 Hz), 8.12 (2H, d, *J* = 9.1 Hz), 7.28 (2H, s), 2.67 (6H, s), 2.38 (3H, s); ^{13}C NMR (100 MHz, MeOD) δ: 151.3, 146.4, 143.7, 136.2, 131.6, 127.5, 122.4, 119.8, 27.1, 21.1; HRMS (ESI) calculated for $C_{15}H_{15}INO_2$ [M-OTf]$^+$ m/z 368.0142, found 368.0138.

3-Bromophenyl(mesityl)iodonium trifluoromethanesulfonate **2i**



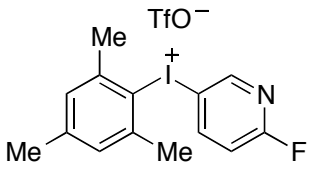
Prepared according to General Procedure E from 3-bromoiodobenzene. Product obtained as a white solid (9.92 g, 18.0 mmol, 40%); IR ν_{max} (solid) / cm^{-1} 1454, 1223, 1165; m.p. 174-175 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.77 (1H, t, *J* = 1.6 Hz), 7.69 (1H, d, *J* = 8.2 Hz), 7.63 (1H, d, *J* = 8.1 Hz), 7.27 (1H, t *J* = 8.1 Hz), 7.11 (2H, s), 2.62 (6H, s), 2.35 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.5, 142.4, 134.9, 134.8, 132.9, 131.6, 130.3, 124.9, 120.7, 111.8, 27.0, 21.1; HRMS (ESI) calculated for $C_{15}H_{15}BrI$ [M-OTf]$^+$ m/z 400.9396, found 400.9393.

3-Trifluoromethylphenyl(mesityl)iodonium trifluoromethanesulfonate **2j**



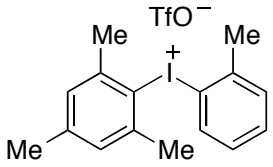
Prepared according to General Procedure E from 3-iodobenzotrifluoride. Product obtained as an off-white solid (19.2 g 35.5 mmol, 79%); IR ν_{max} (solid) / cm^{-1} 1242, 1133; m.p. 181-183 °C; ^{1}H NMR (400 MHz, MeOD) δ: 8.30 (1H, s), 8.09 (1H, d, *J* = 8.1 Hz), 7.98 (1H, d, *J* = 7.8 Hz), 7.73 (1H, t, *J* = 8.0 Hz), 7.30 (2H, s), 2.70 (6H, s), 2.40 (3H, s); ^{13}C NMR (100 MHz, MeOD) δ: 146.3, 143.6, 138.4, 134.6 (q, *J* = 33.6Hz) 134.1, 131.7 (q, *J* = 3.7 Hz), 131.5, 130.0 (q, *J* = 3.2 Hz), 124.2 (q, *J* = 273 Hz), 122.4, 114.0, 27.0, 21.0; HRMS (ESI) calculated for $C_{16}H_{15}F_3I$ $[M\text{-}OTf]^+$ m/z 391.0165, found 391.0159.

6-Fluoropyridin-3-yl(mesityl)iodonium trifluoromethanesulfonate **2k**



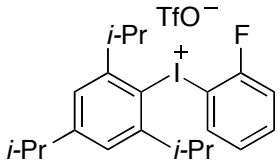
A round bottomed flask was charged with *m*-CPBA (dried under vacuum at room temperature for 1 hour, assume 65%, 2.42 g, 8.96 mmol) and 2-fluoro-5-iodopyridine (2.0 g, 8.96 mmol) in dichloromethane (40 mL). The reaction mixture was heated at reflux for 2 hours before mesitylene (1.37 mL, 9.86 mmol) and trifluoromethanesulfonic acid (2.65 mL, 9.86 mmol) were added at 0 °C and the mixture was slowly warmed to room temperature over 4 hours. The reaction mixture was then concentrated *in vacuo* and the residue was triturated in diethyl ether and then stored in the freezer for 2.5 hours. The cold suspension was then filtered and washed with cold diethyl ether (× 3) before being left to dry under vacuum (0.2 mbar) overnight to furnish the title compound as an off white solid (1.76 g, 3.58 mmol, 40% yield); IR ν_{max} (solid) / cm^{-1} 1582, 1558, 1471, 1375, 1242, 1222, 1171; m.p. 178-180 °C; ^{1}H NMR (400 MHz, Acetone-D_6) δ: 8.60–8.58 (1H, app d, *J* = 2.3 Hz), 8.40 (1H, ddd, *J* = 9.0, 6.8 and 2.5 Hz), 7.08 (1H, dd, *J* = 8.8, and 2.6 Hz), 7.01 (2H, bs), 2.48 (6H, s), 2.09 (3H, s); ^{13}C NMR (100 MHz, Acetone-D_6) δ: 165.2, (d, *J* = 243.7), 153.2 (d, *J* = 16.3 Hz), 148.3 (d, *J* = 9.1 Hz), 145.1, 143.1, 130.8, 122.0, 114.5 (d, *J* = 39.1 Hz), 107.5 (d, *J* = 4.0 Hz), 26.7, 20.6; HRMS (ESI) calculated for $C_{14}H_{14}NIF$ $[M\text{-}TfO^-]$ m/z 342.0155, found 342.0163.

2-Methylphenyl(mesityl)iodonium trifluoromethanesulfonate **2l**



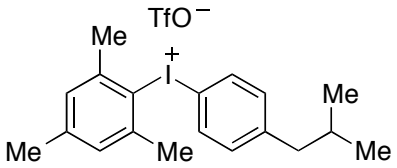
Prepared according to General Procedure E from 2-iodotoluene. Product obtained as a white solid (18.6 g, 38.3 mmol, 85%); IR ν_{max} (solid) / cm^{-1} 1468, 1244, 1154; m.p. 167–168 °C; ^1H NMR (400 MHz, $CDCl_3$ δ: 7.41-7.48 (3H, m), 7.16 (1H, dt, *J* = 1.8 and 7.7 Hz), 7.09 (2H, s), 2.57 (9H, s), 2.33 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.4, 142.3, 140.1, 133.7, 132.4, 132.4, 130.6, 129.7, 119.4, 115.6, 26.8, 24.7, 20.9; HRMS (ESI) calculated for $C_{16}H_{18}I$ $[M\text{-}OTf]^+$ m/z 337.0447, found 337.0444.

(2-Fluorophenyl)(2,4,6-triisopropylphenyl)iodonium trifluoromethanesulfonate **2m**



Trifluoromethanesulfonic acid (1.31 mL) was added dropwise to a solution of 2-fluoroiodobenzene (1.98 g, 8.92 mmol) and *m*CPBA (dried under vacuum at room temperature for 1 hour, assume 65%, 2.64g, 9.94 mmol) in dichloromethane (20 ml) at 0 °C. The reaction was allowed to slowly warm to room temperature over the course of 2 hours and then re-cooled prior to the addition of 1,3,5-triisopropyl benzene (2.39 mL, 9.89 mmol) at 0 °C. The reaction mixture was then allowed to warm to room temperature and stirred for 20 hours. The solvent was removed *in vacuo* and diethyl ether added and the resulting crystals were filtered, washed on the filter with cold diethyl ether and then dried at 100 °C under vacuum for 1 hour to give the title compound (3.68 g, 6.41 mmol, 71%) as a white solid; IR ν_{max} (solid) / cm^{-1} 1472, 1275, 1243, 1223, 1158; m.p. 156–157 °C; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.72–7.68 (1H, m), 7.62–7.56 (1H, m), 7.29 (1H, dt, *J* = 1.3 and 8.4 Hz), 7.26–7.22 (1H, m), 7.15 (2H, s), 3.35 (2H, sp, *J* = 6.7 Hz), 2.94 (1H, sp, *J* = 6.9 Hz), 1.26 (18H, d, *J* = 6.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.4 (d, *J* = 254 Hz), 155.4, 152.3, 135.2, 135.0 (d, *J* = 7.7 Hz), 127.6 (d, *J* = 2.9 Hz), 125.5, 121.8, 117.4 (d, *J* = 21.7 Hz), 98.8 (d, *J* = 22.5 Hz), 39.9, 34.1, 24.3, 23.6; HRMS (ESI) calculated for $C_{21}H_{27}FI$ $[M\text{-}TfO^-]$ m/z 425.1136, found 425.1132.

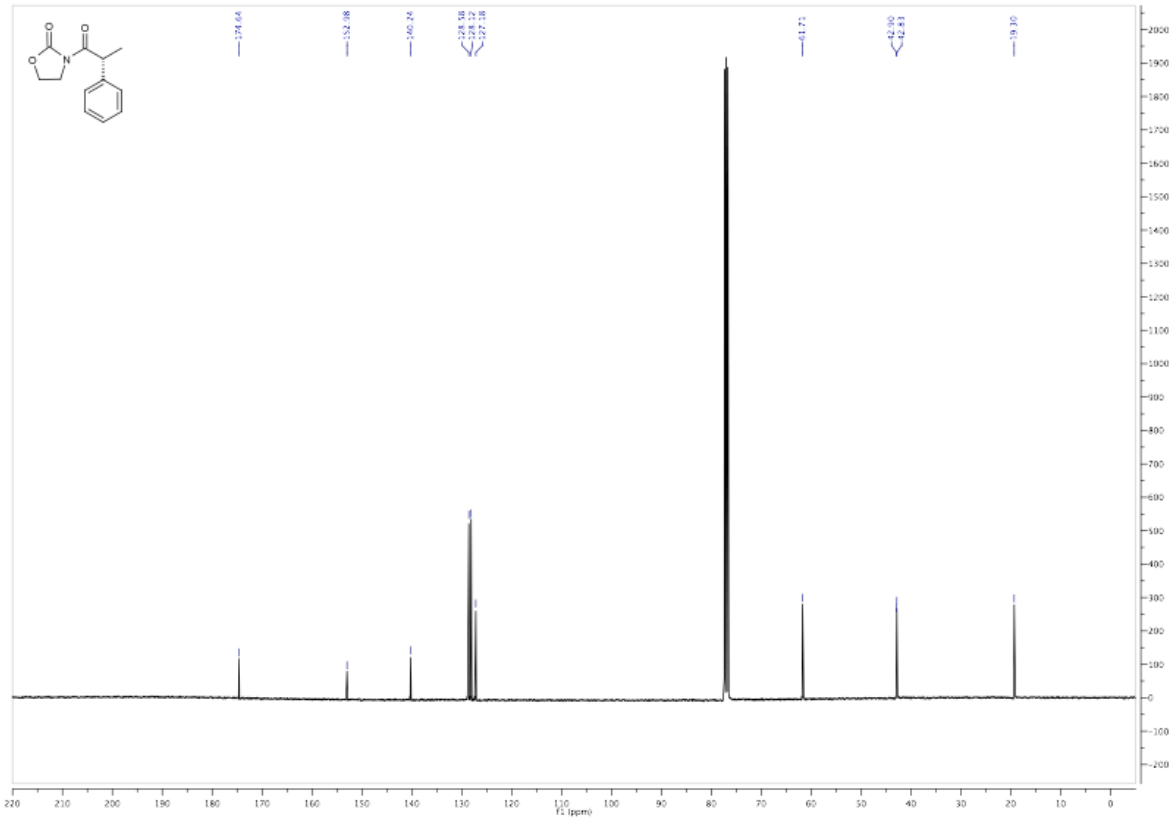
(4-Isobutylphenyl)(mesityl)iodonium trifluoromethanesulfonate **2n**

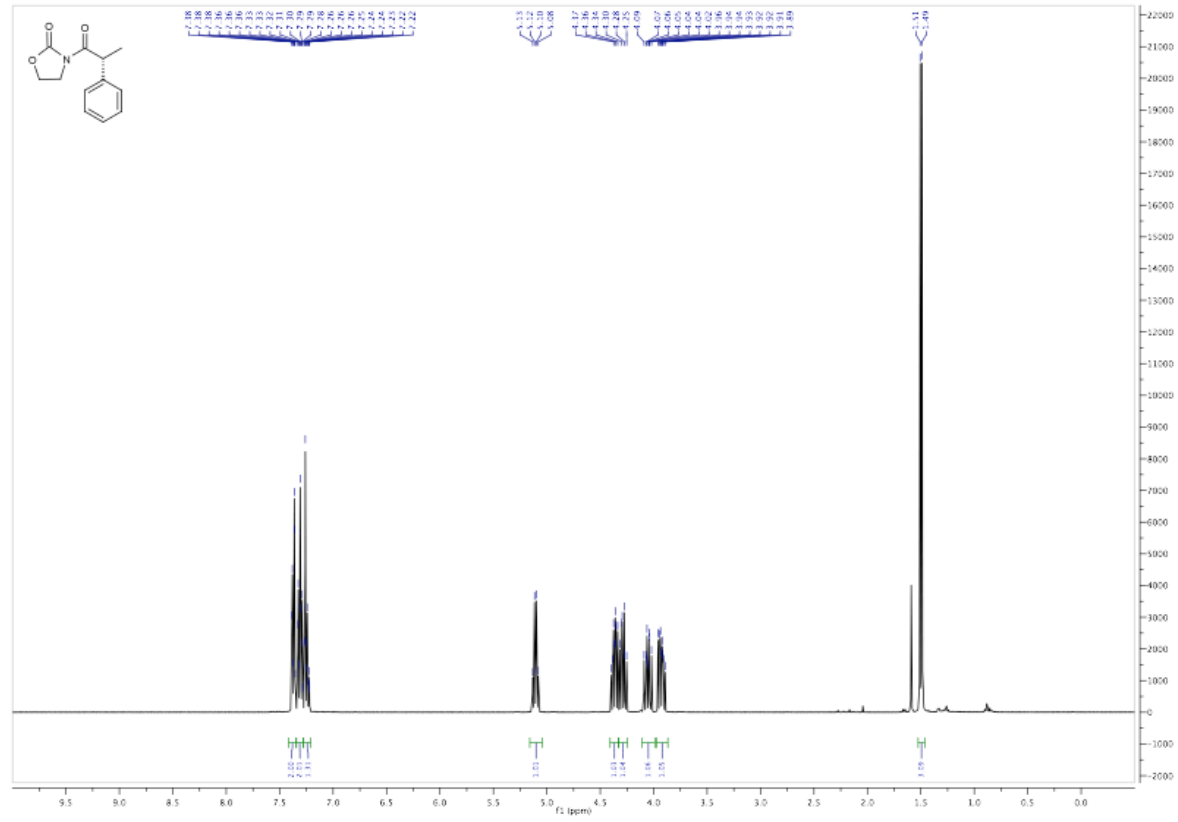


Trifluoromethanesulfonic acid (2.67 mL, 25.2 mmol) was added dropwise to a solution of 4-isobutylbenzene (4.75 mL, 30.2 mmol) and iodomesitylene diacetate (10.0 g, 27.5 mmol) in dichloromethane (200 ml) at 0 °C. The reaction was allowed to slowly warm to room temperature and stirred for 20 hours. The solvent was removed *in vacuo* and diethyl ether added and the resulting crystals were filtered, washed on the filter with cold diethyl ether and then dried at 100 °C under vacuum for 1 hour to give the title compound (11.0 g, 20.1 mmol, 78%) as a white solid; IR ν_{max} (solid) / cm^{-1} 1273, 1244, 1222, 1160; m.p. 195 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.59–7.56 (2H, m), 7.21–7.17 (2H, m), 7.11 (2H, s), 2.63 (6H, s), 2.48 (2H, d, 7.2 Hz), 2.36 (3H, s), 1.83 (1H, sp, *J* = 6.9 Hz), 0.87 (6H, d, 6.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.8, 144.6, 142.5, 133.2, 132.8, 130.5, 120.1, 44.8, 30.1, 27.1, 22.2, 21.1; HRMS (ESI) calculated for $C_{19}H_{24}I$ [M-TfO^-] m/z 379.0917, found 379.0924.

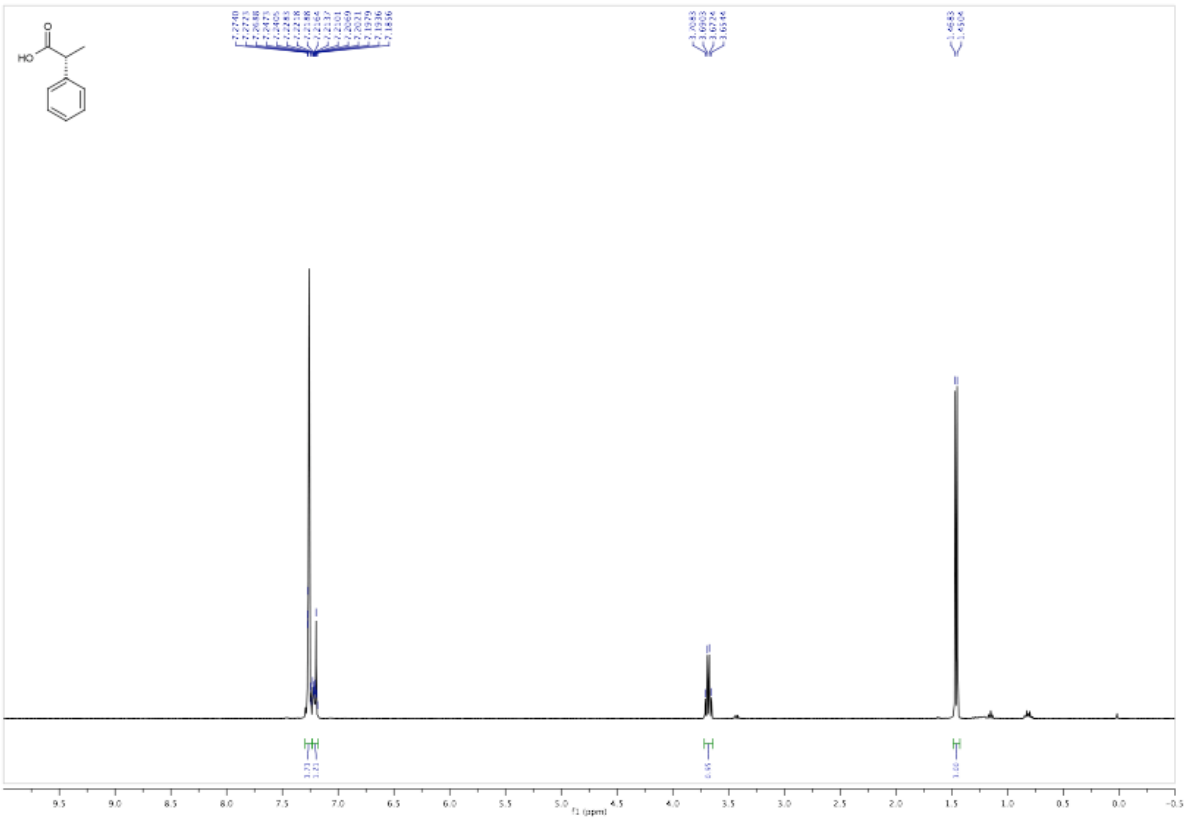
VI. ^{1}H and ^{13}C NMR Spectra

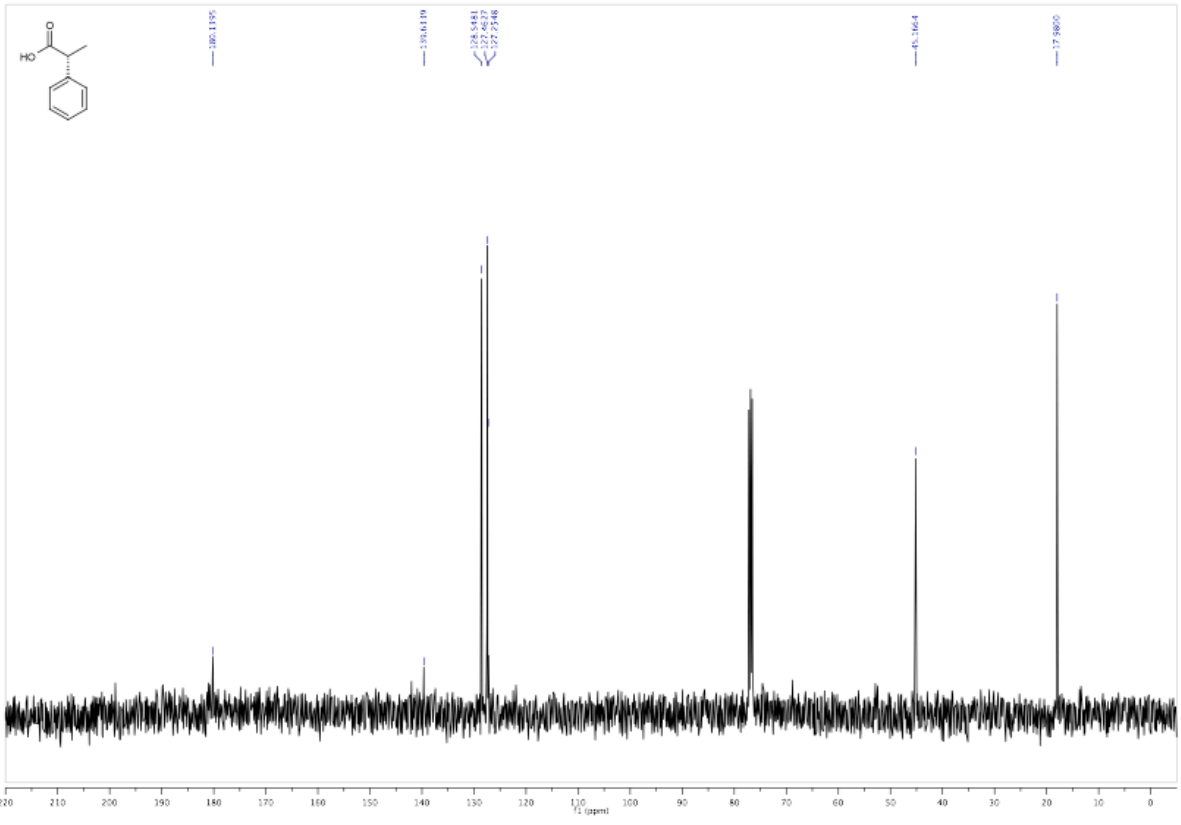
3a





(*R*)-Phenylpropionic acid





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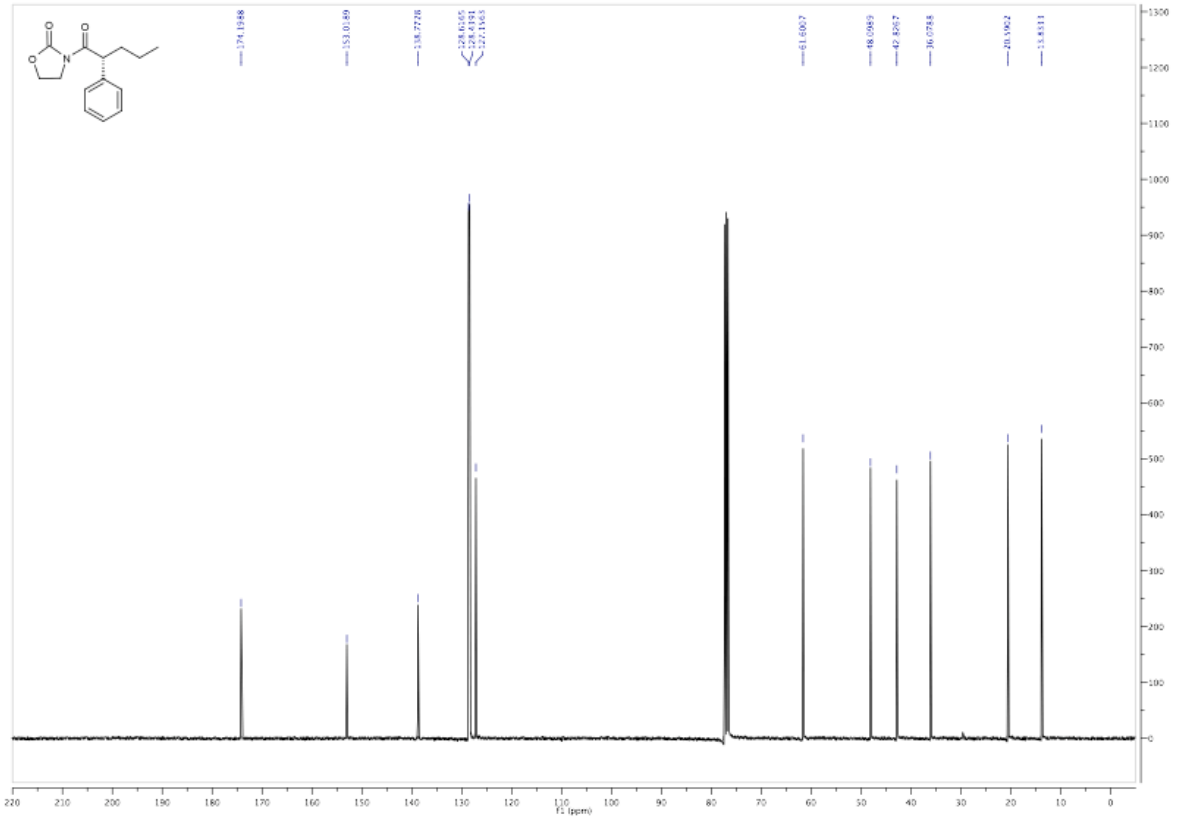

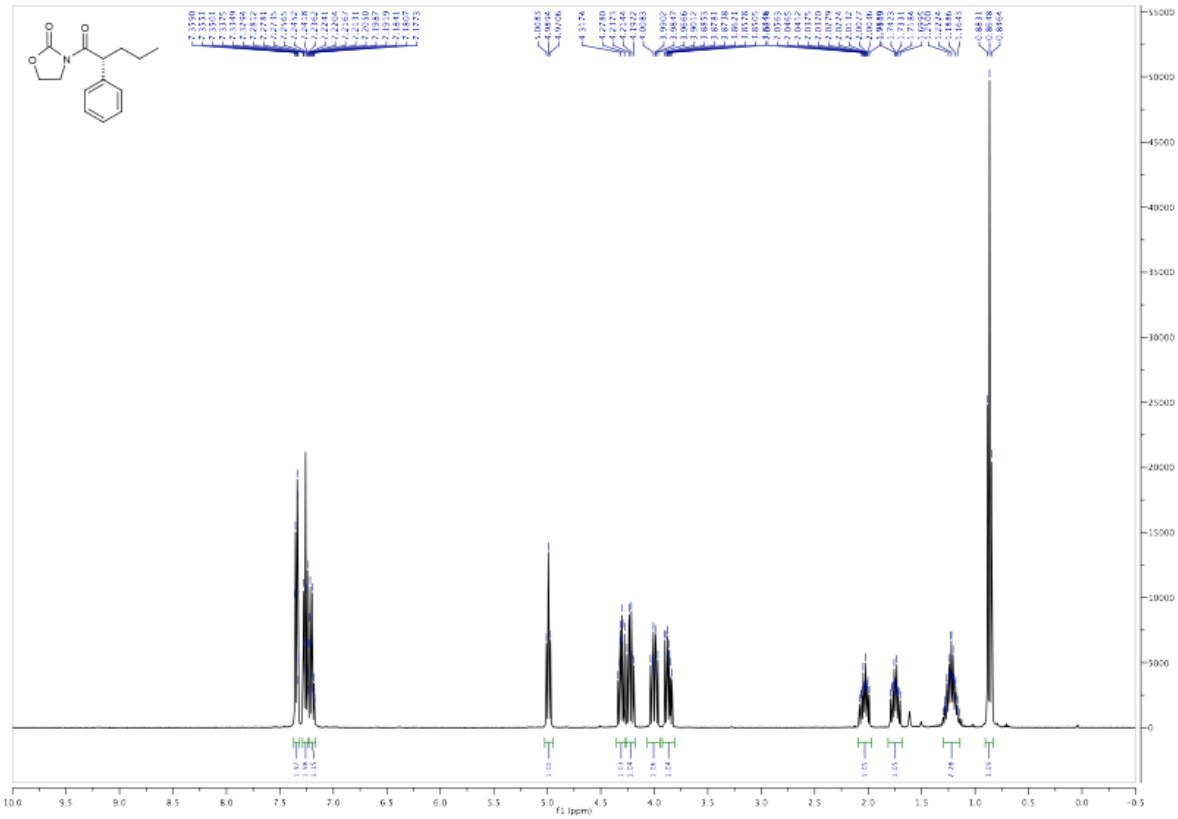

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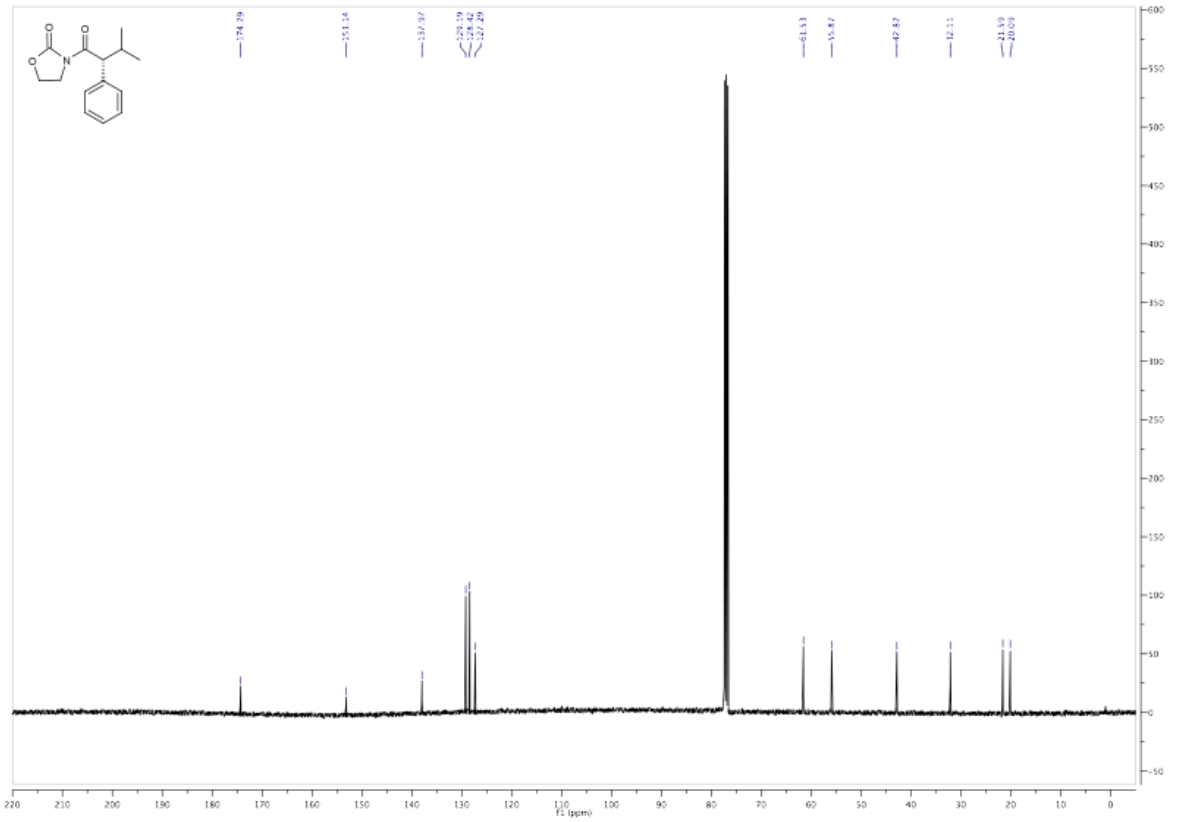

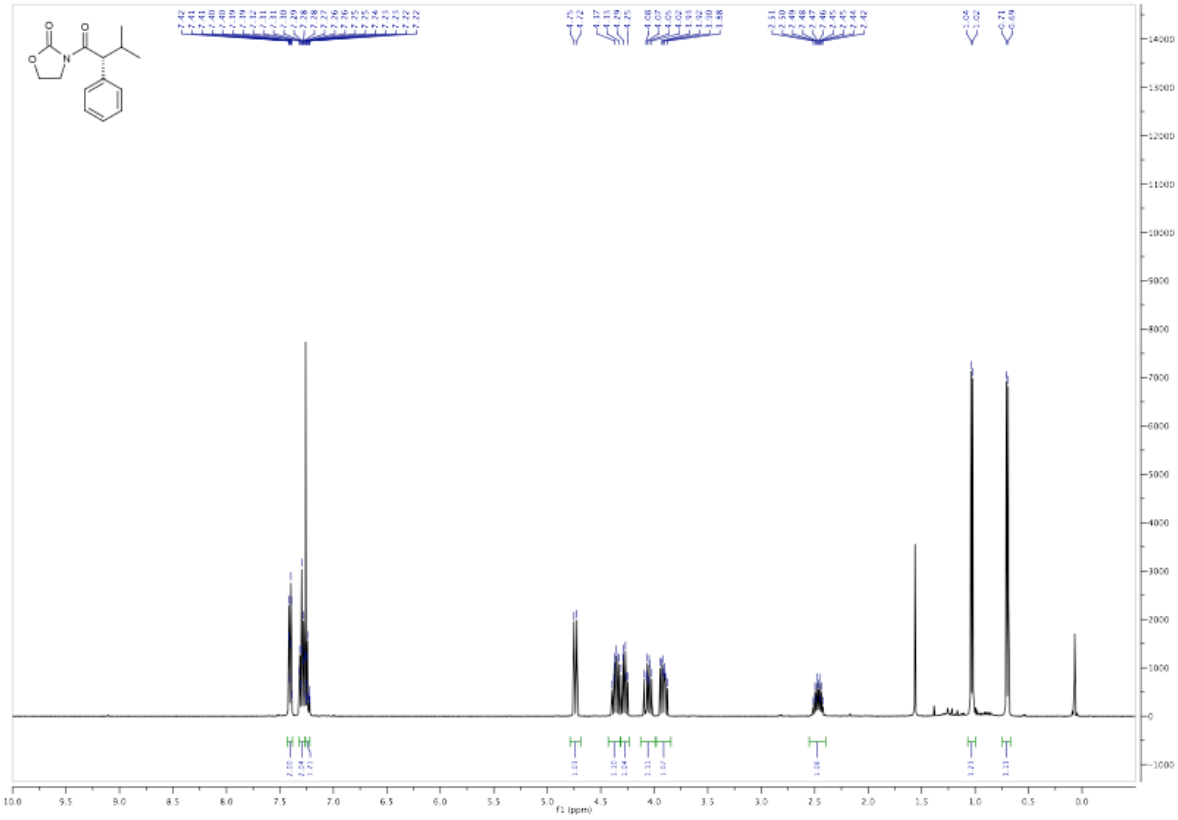

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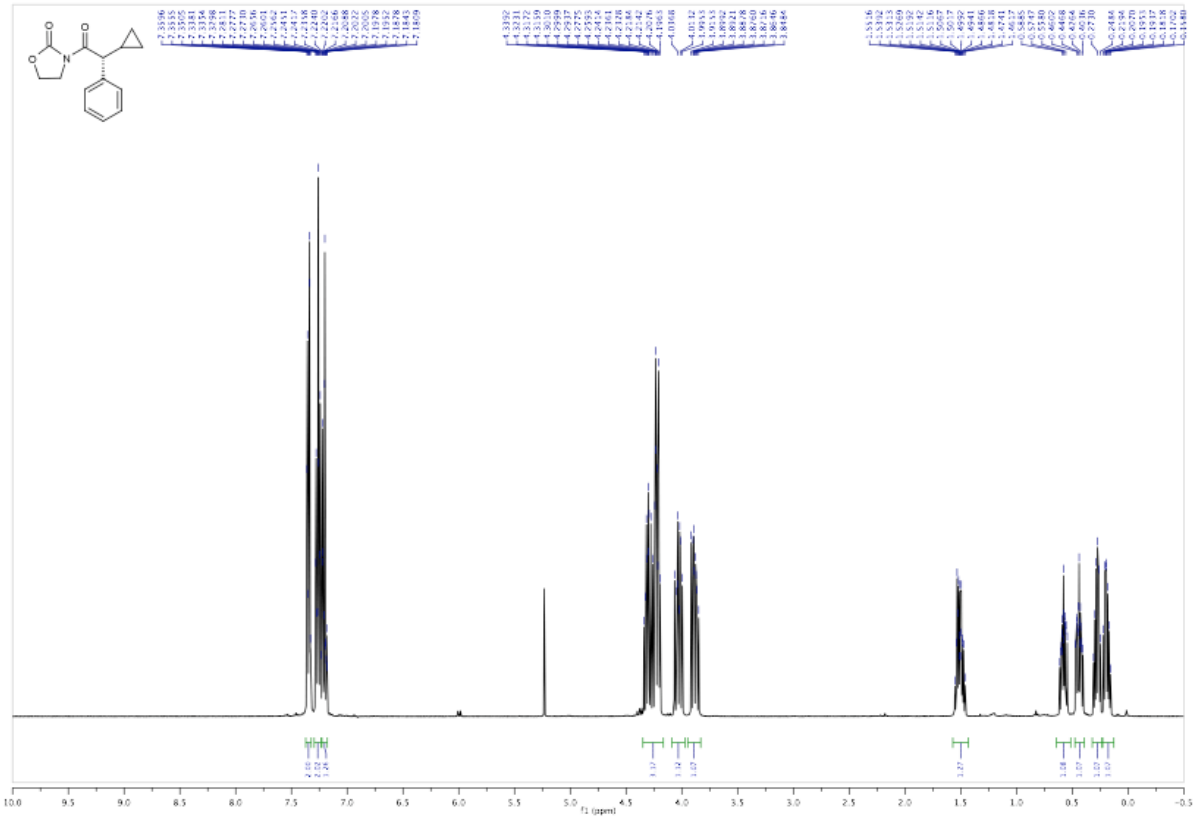

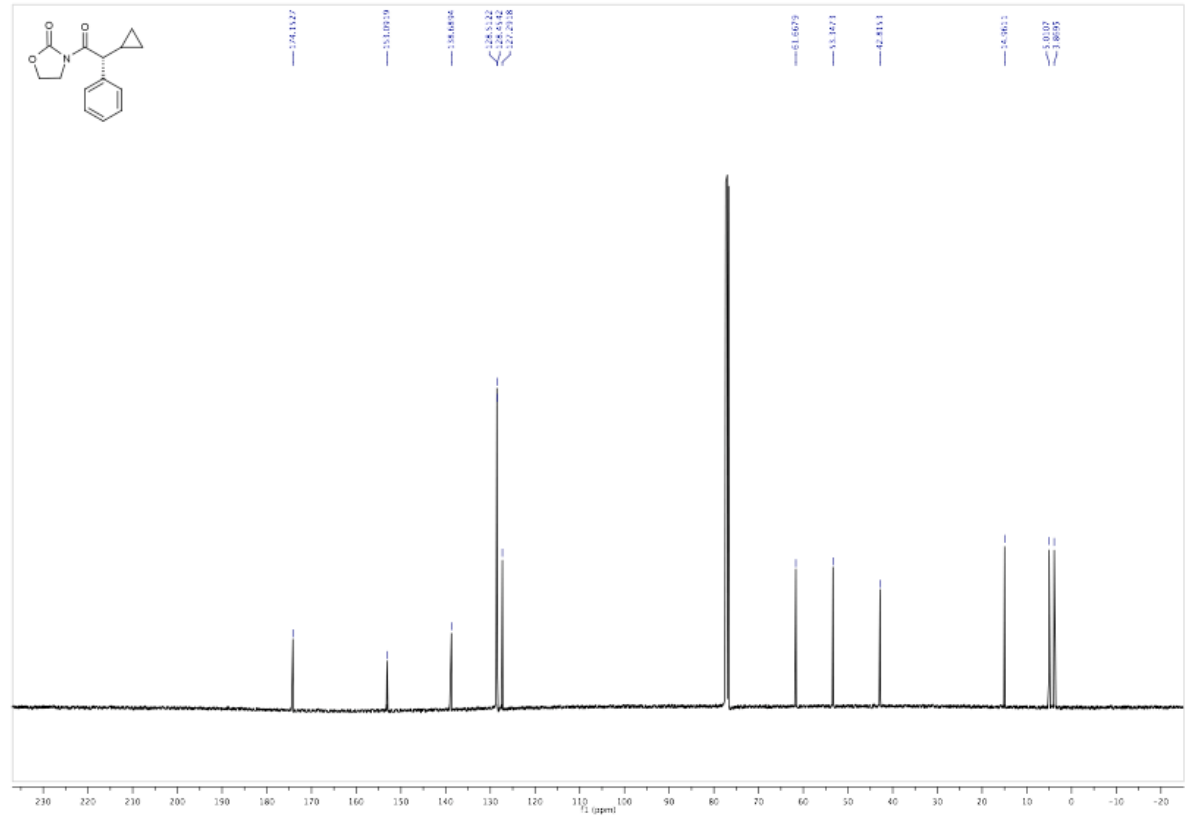

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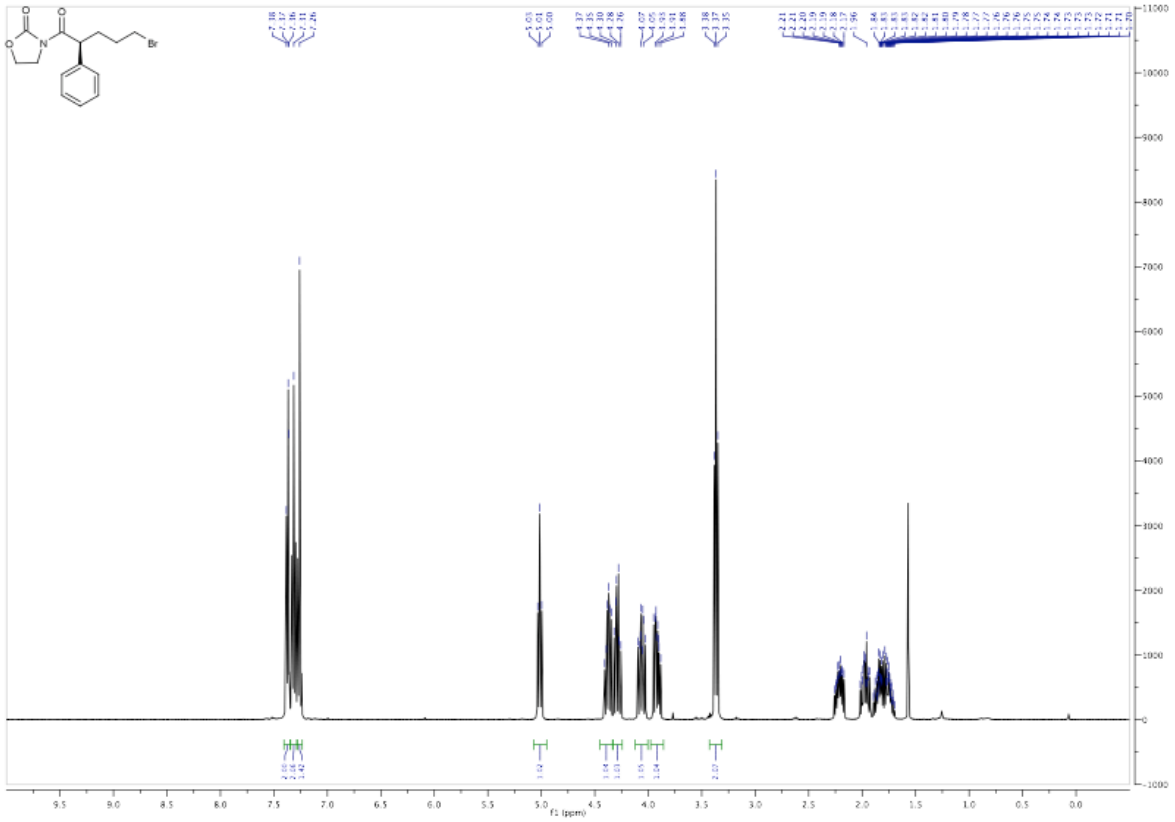

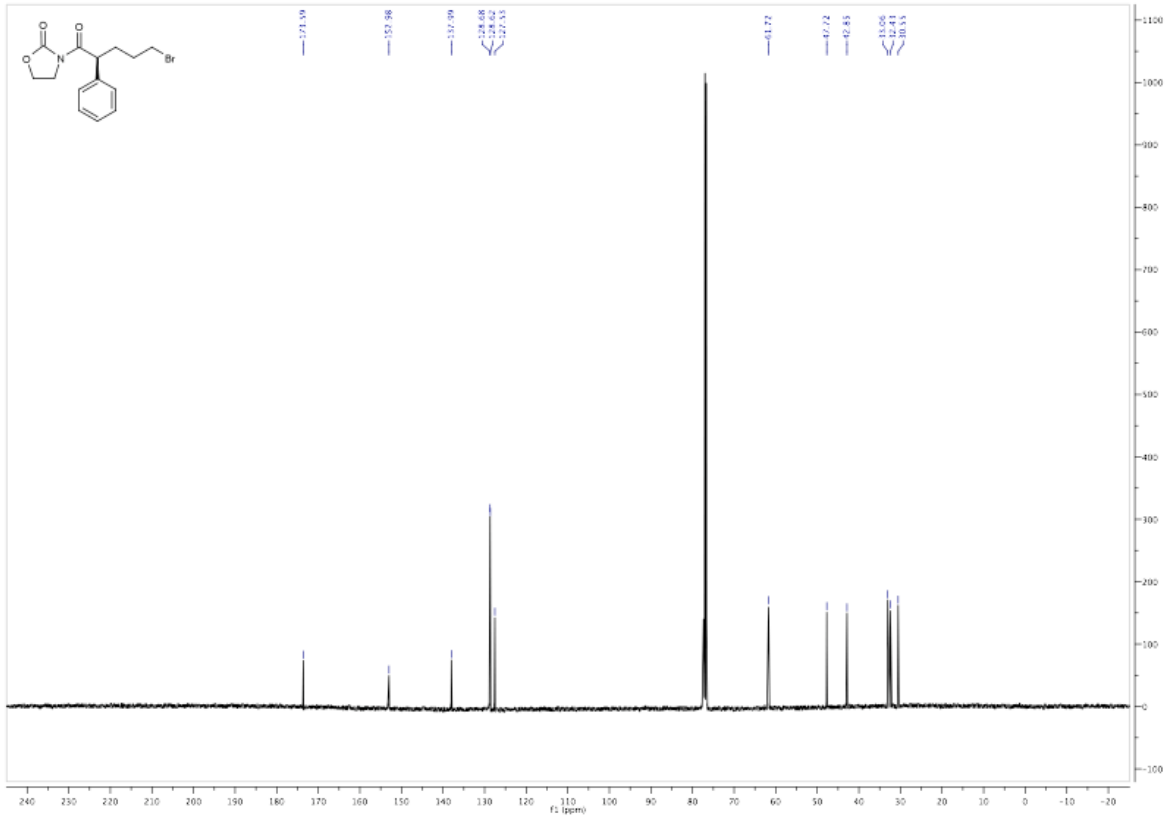

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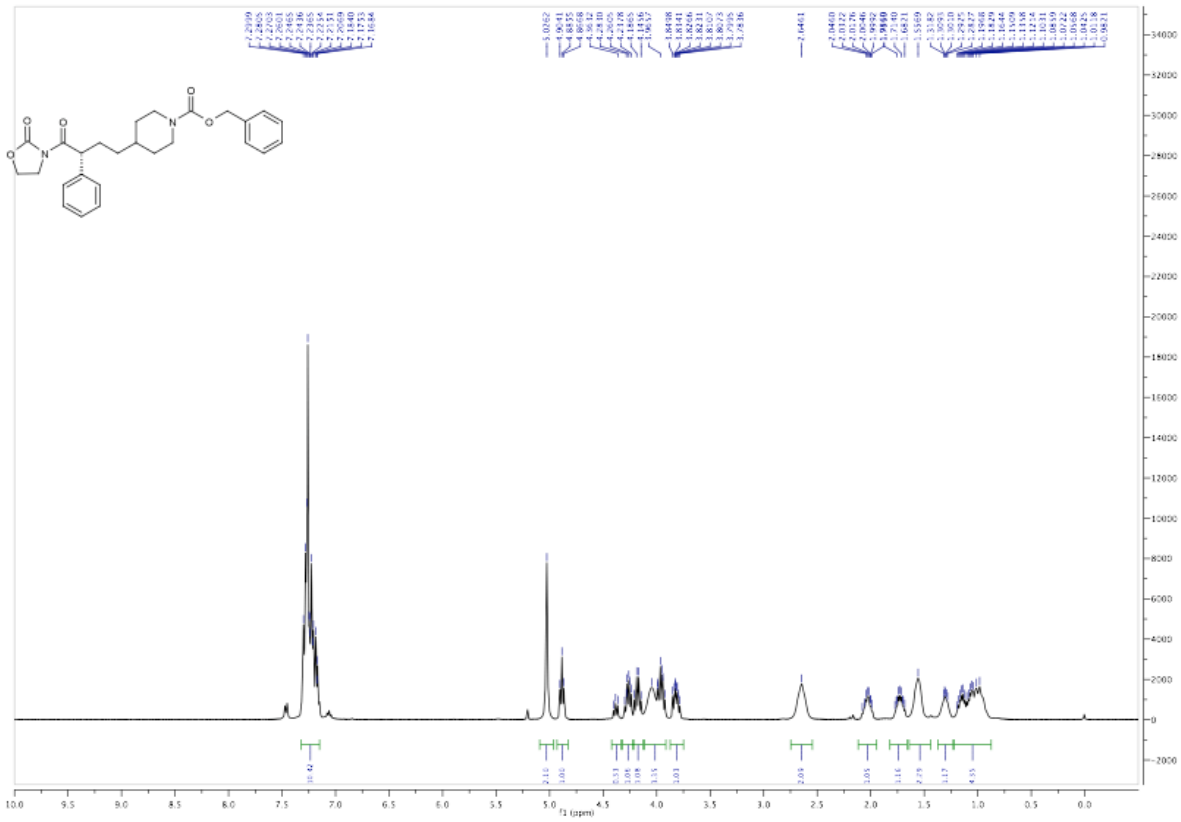

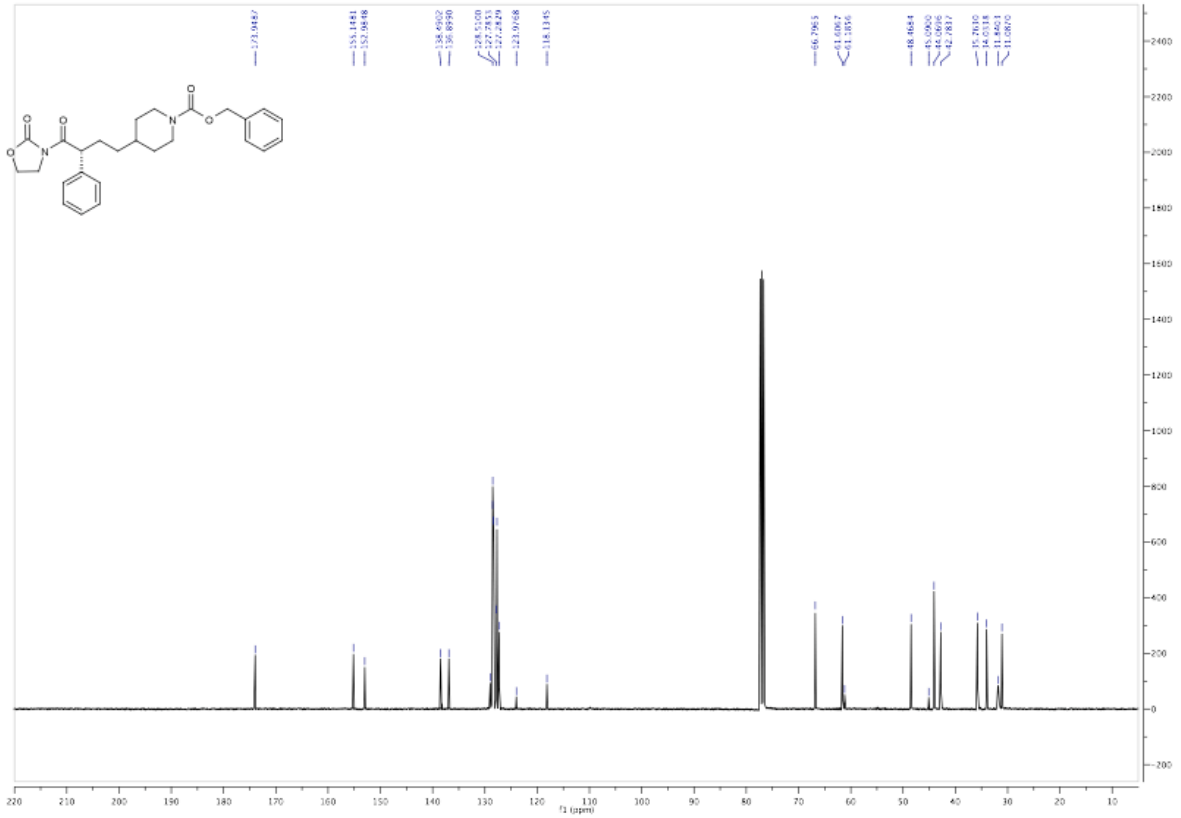

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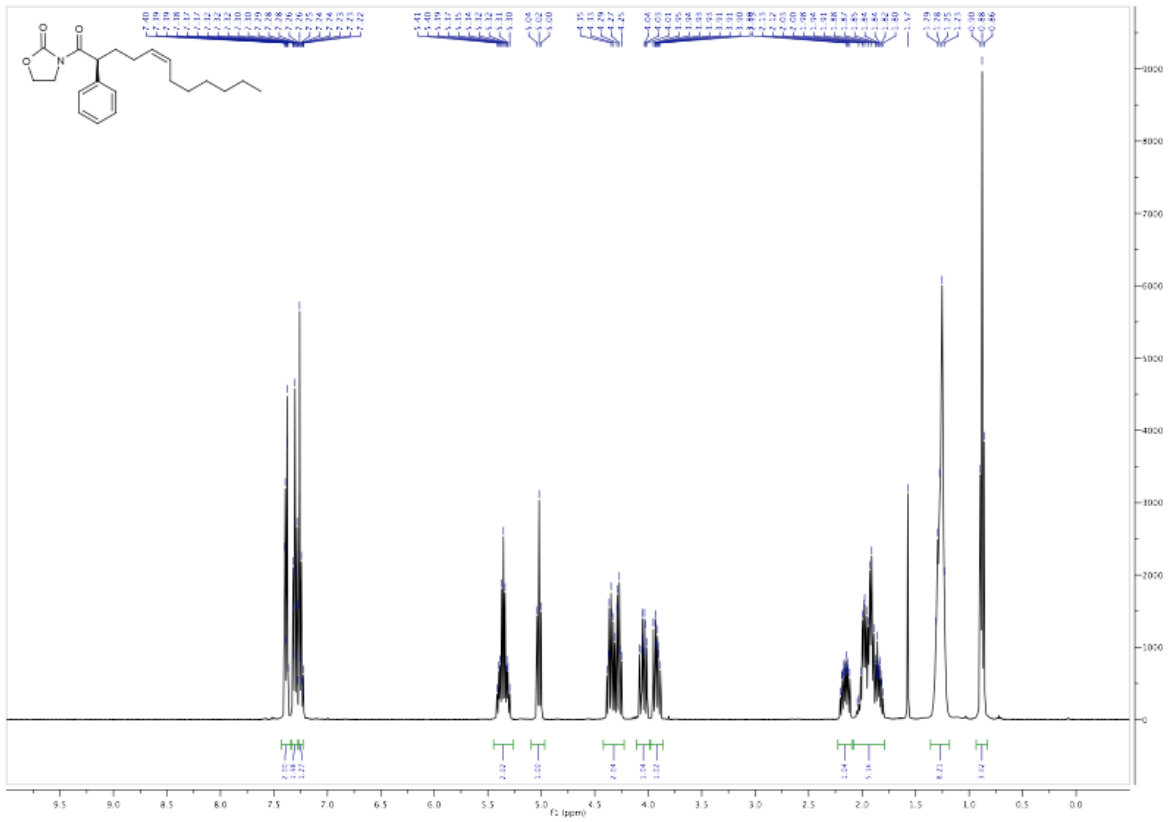

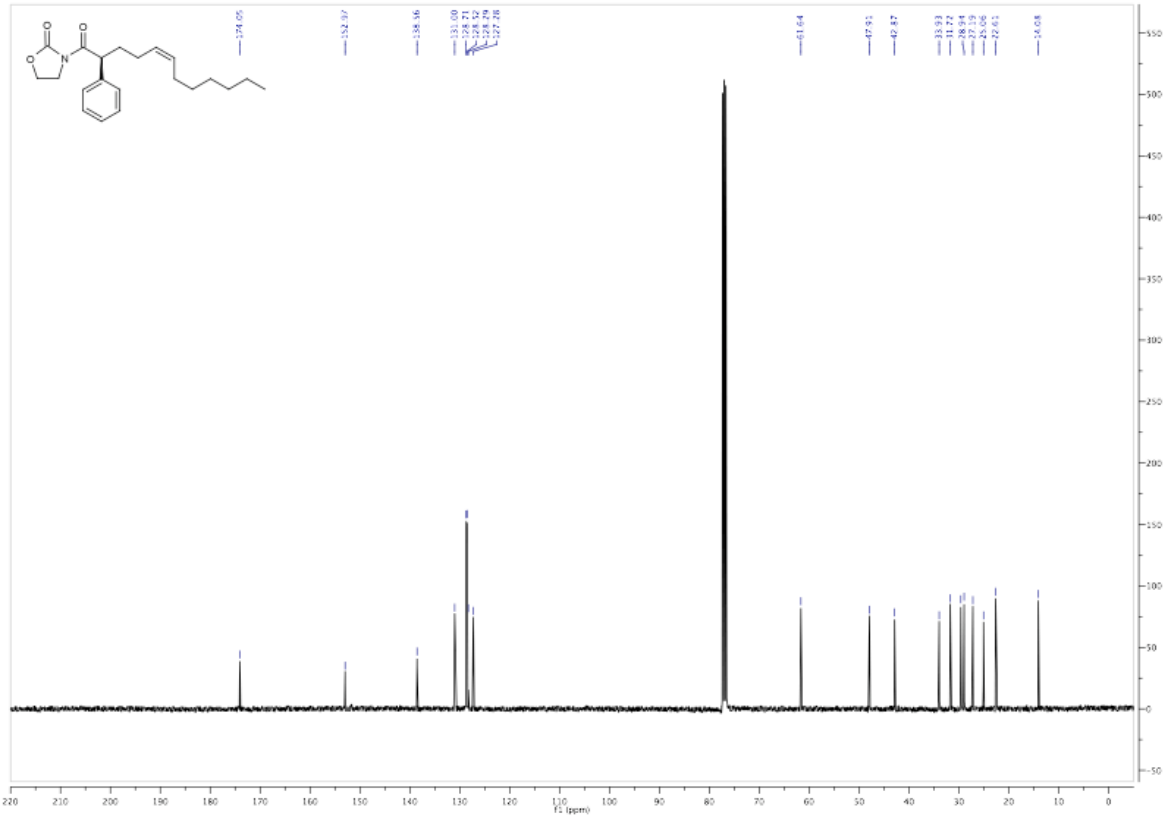

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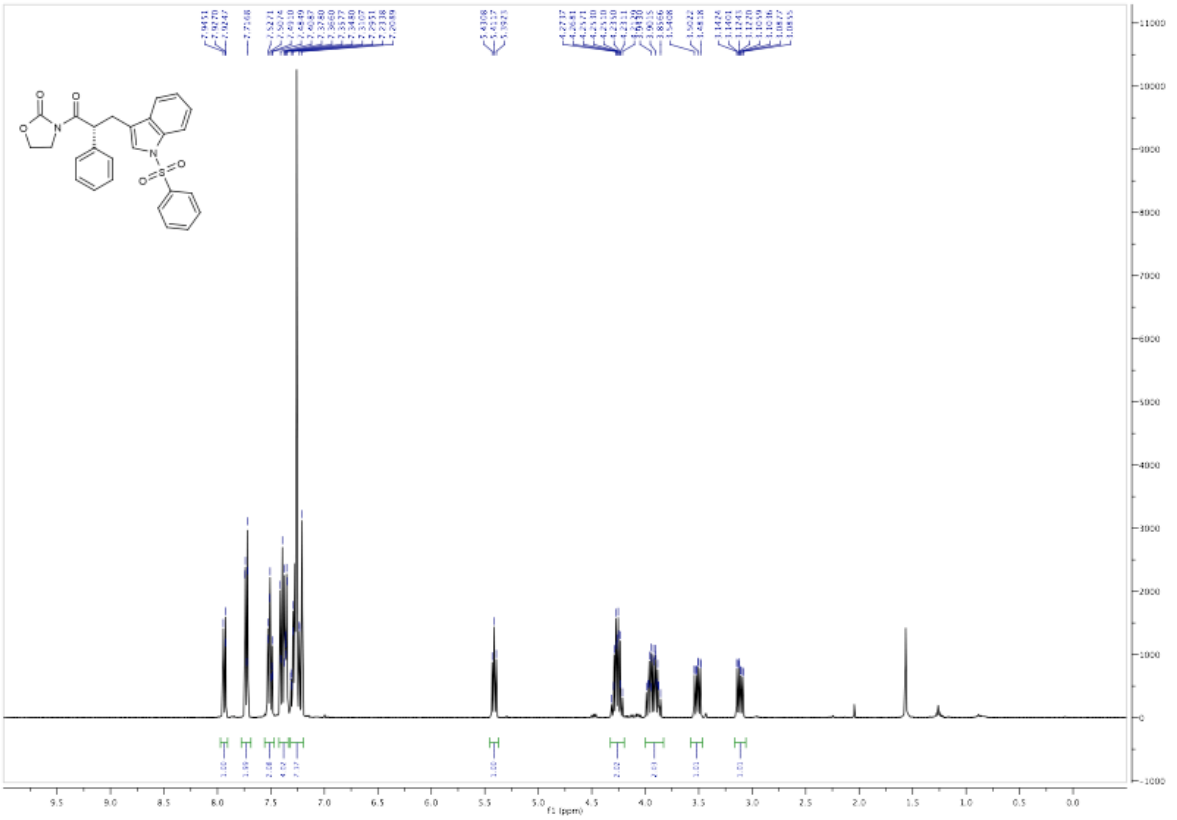

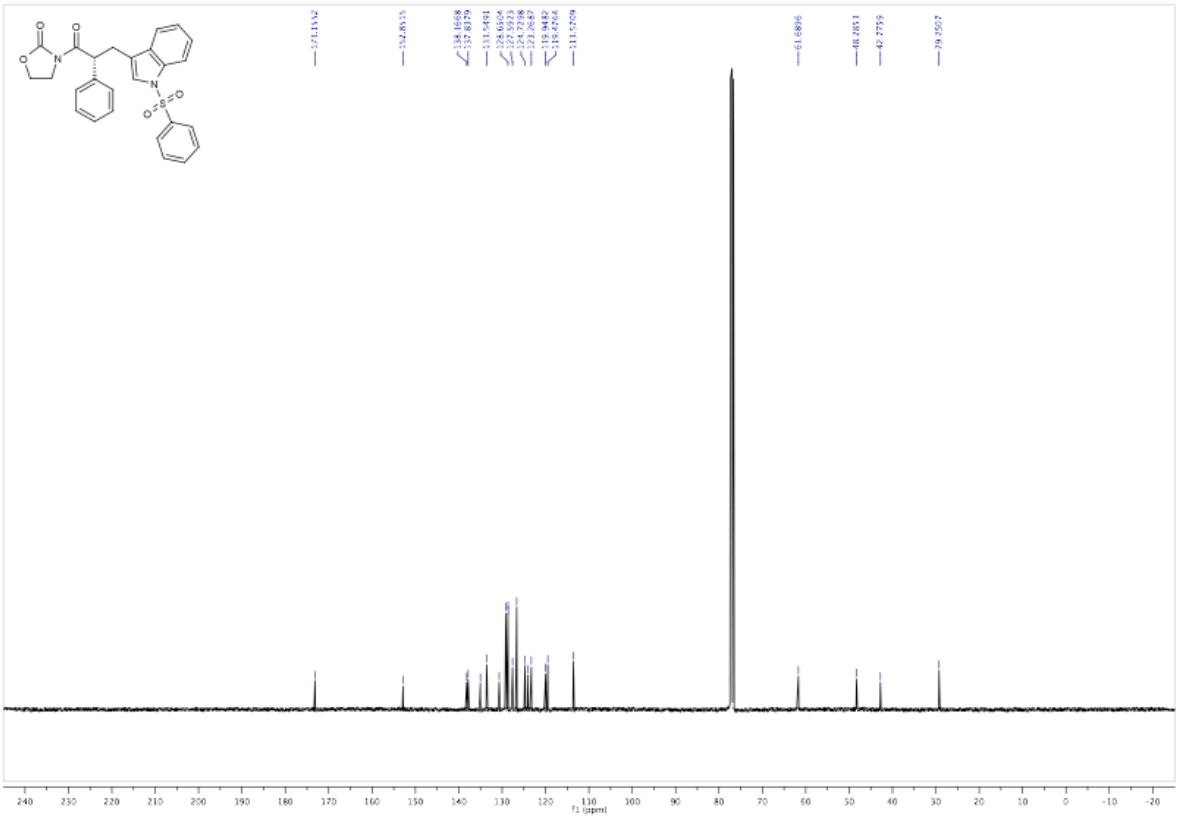

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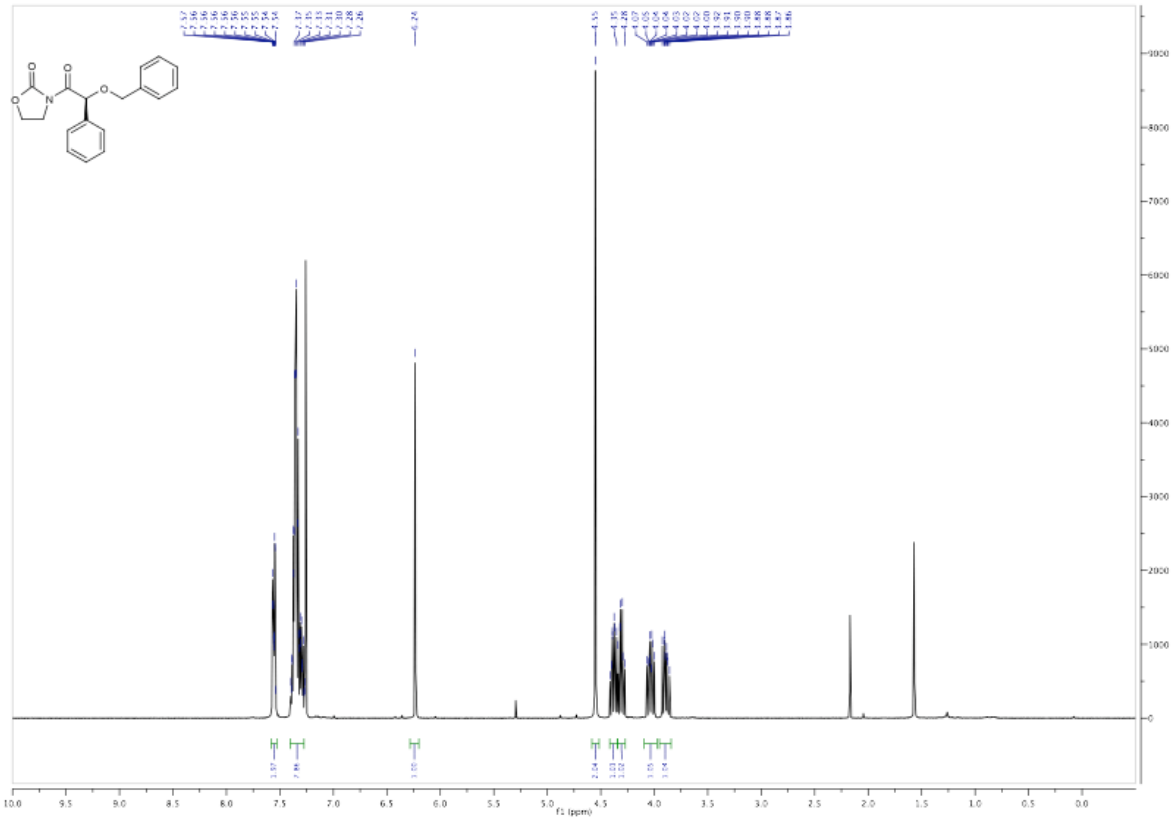

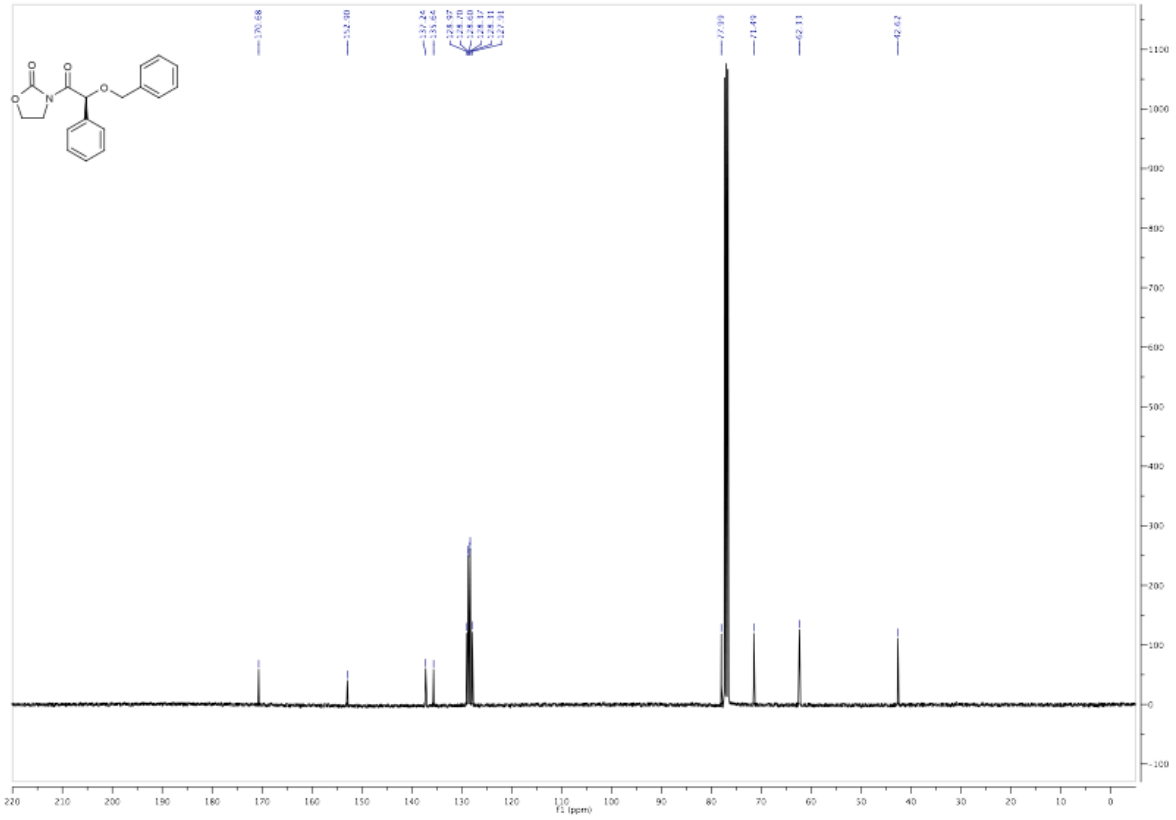

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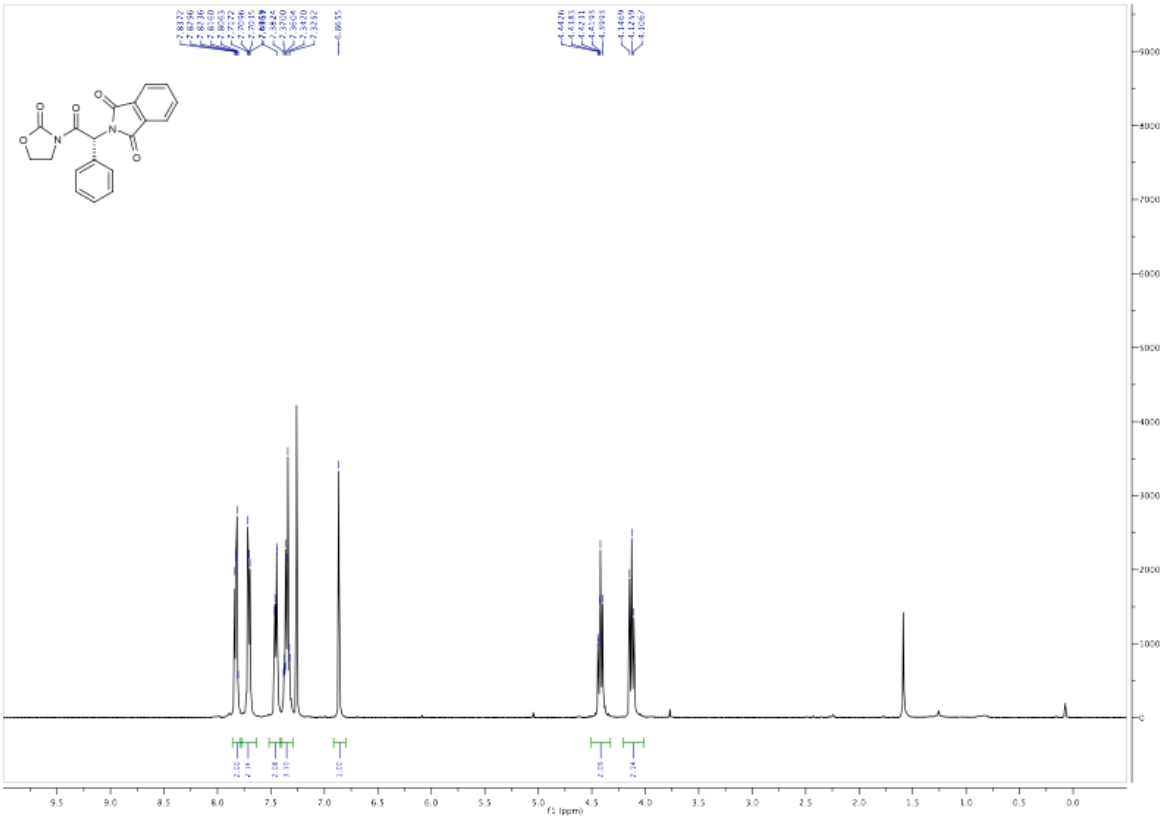

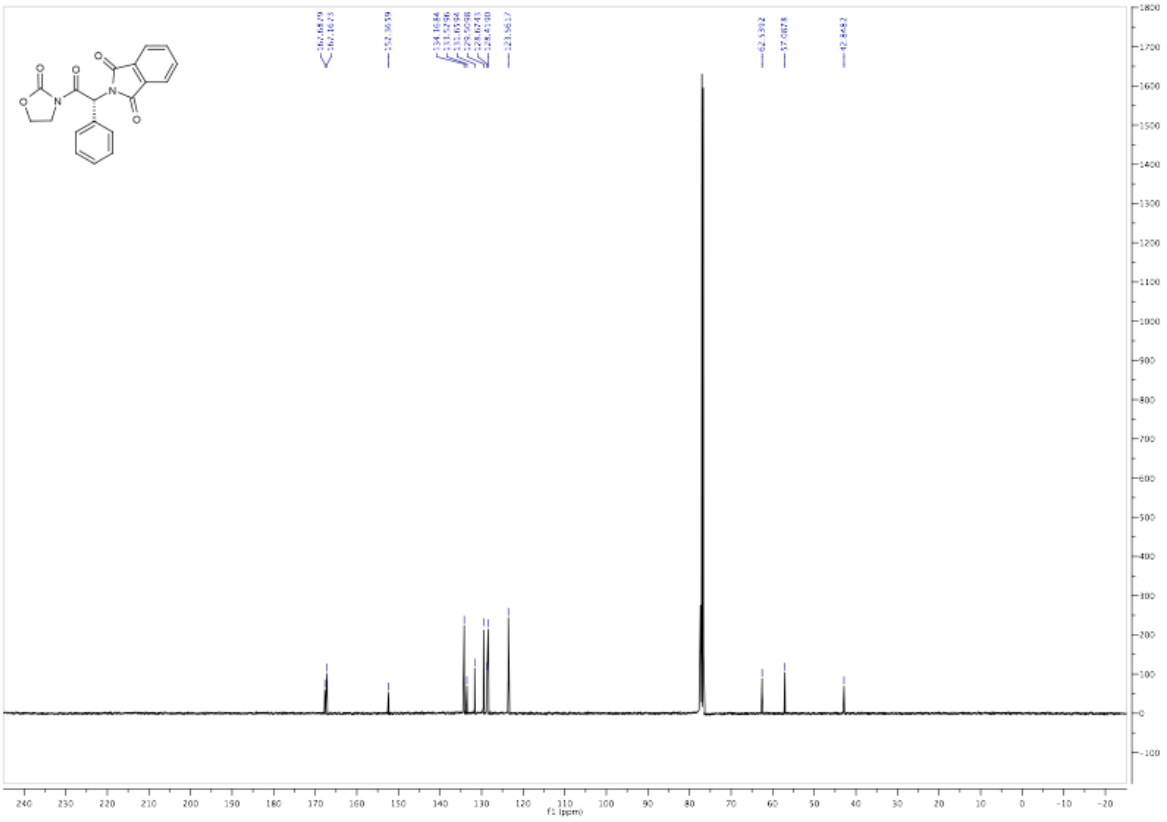

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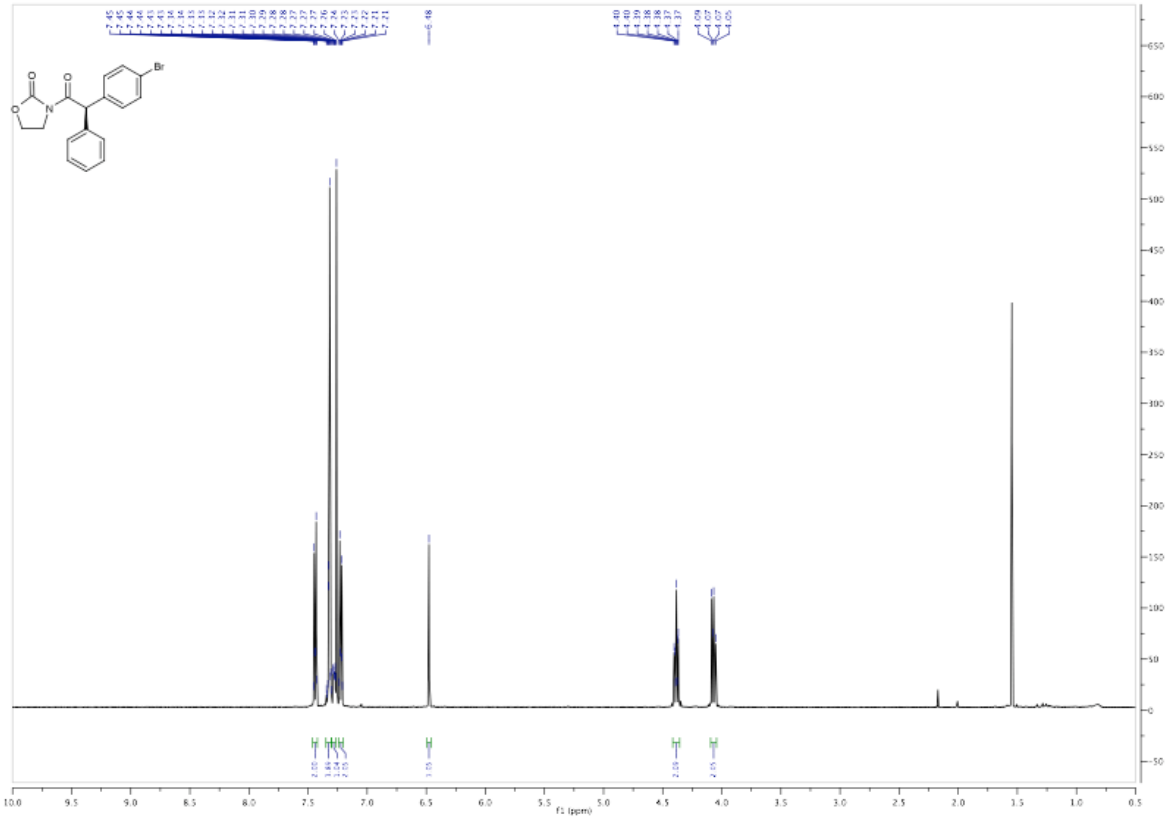

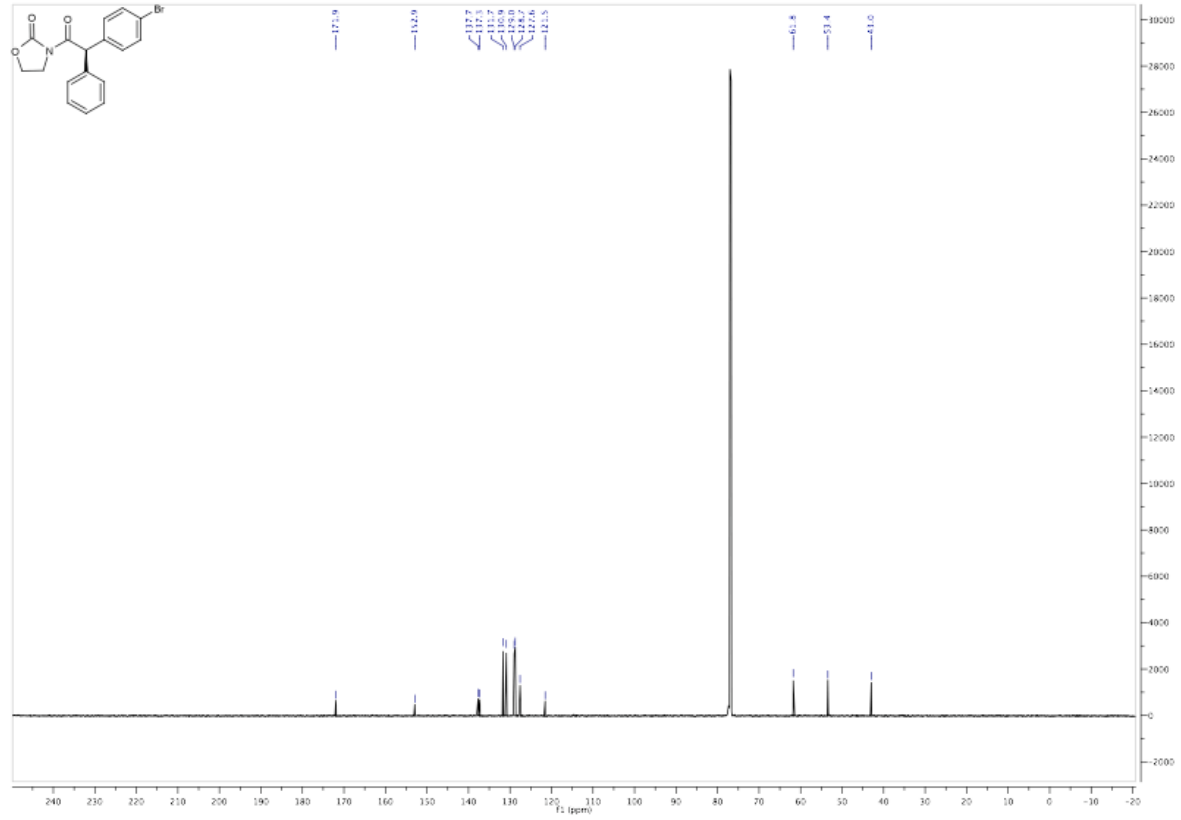

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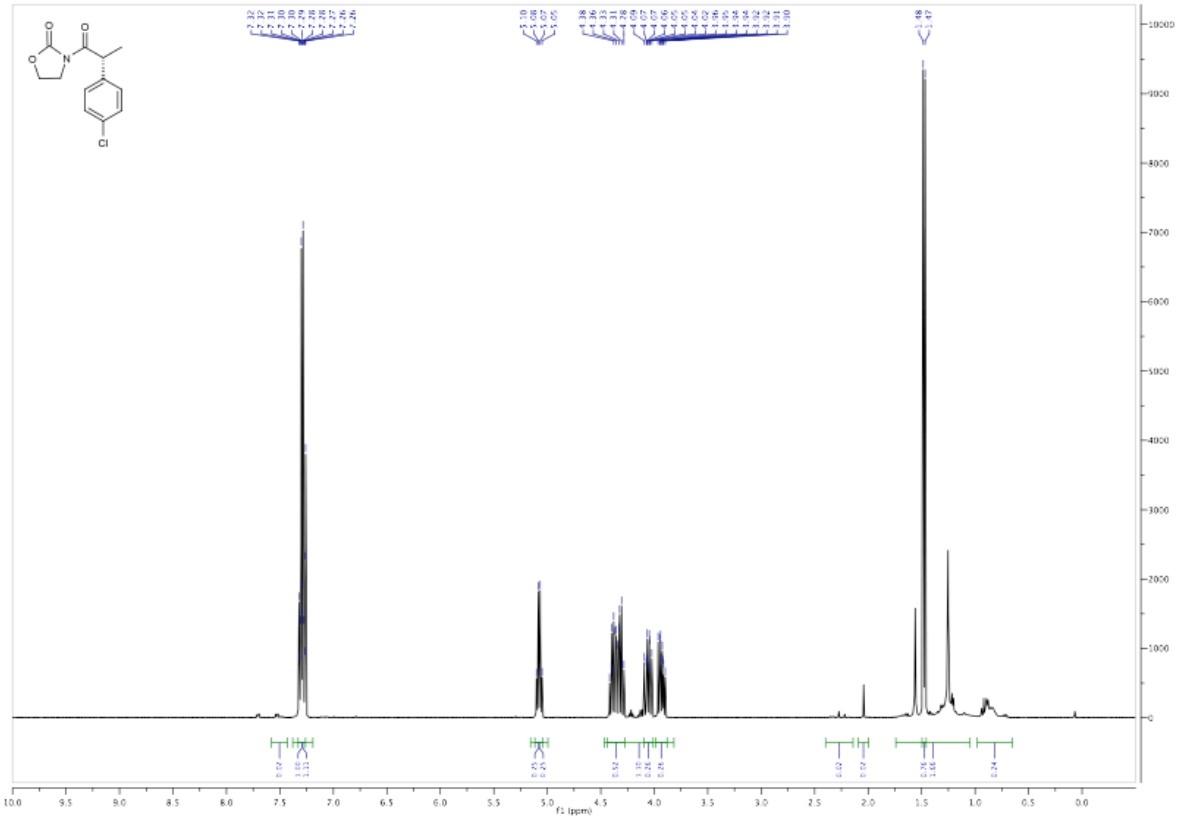

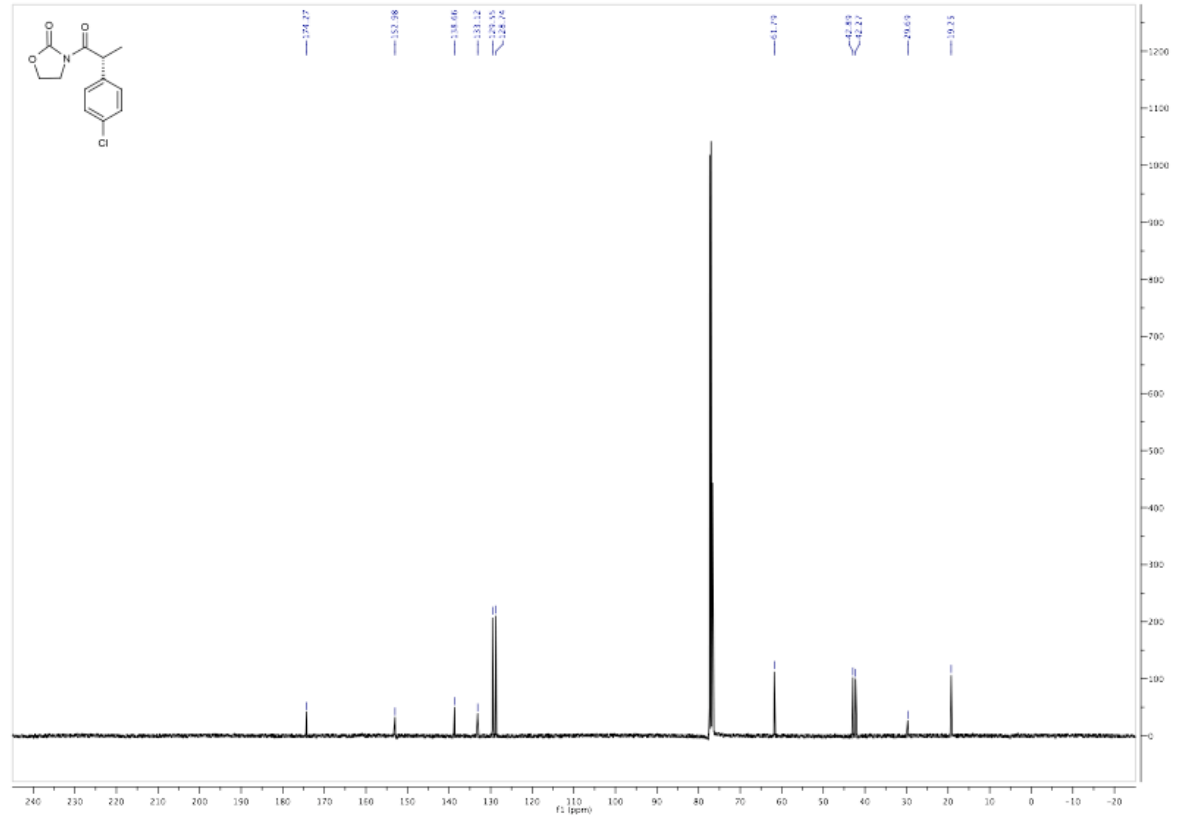

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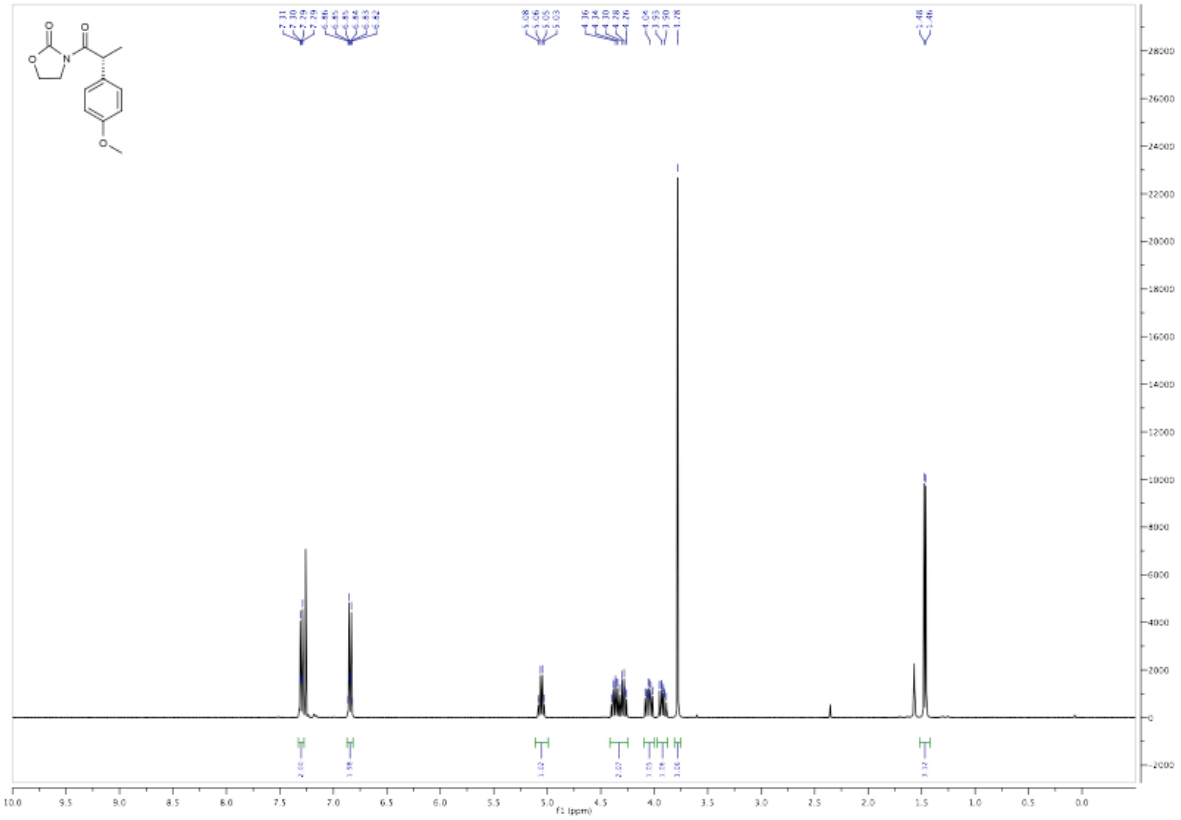

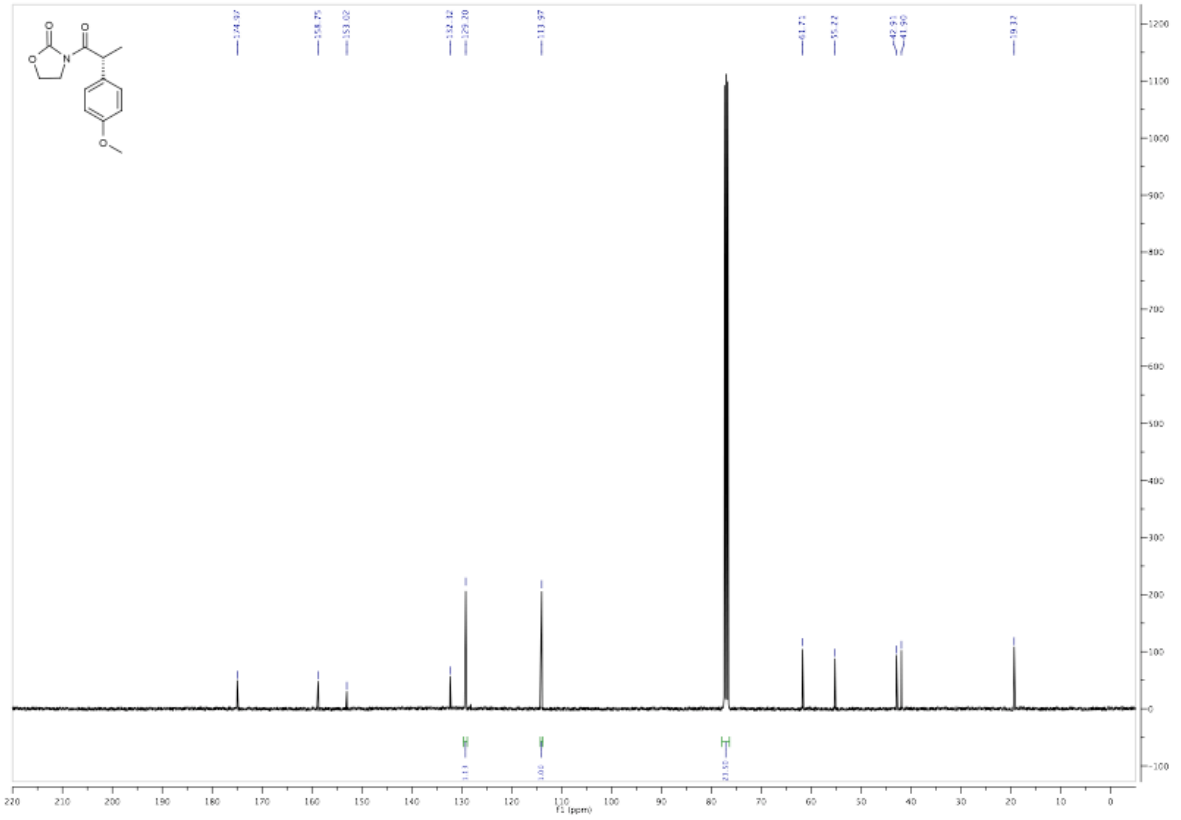

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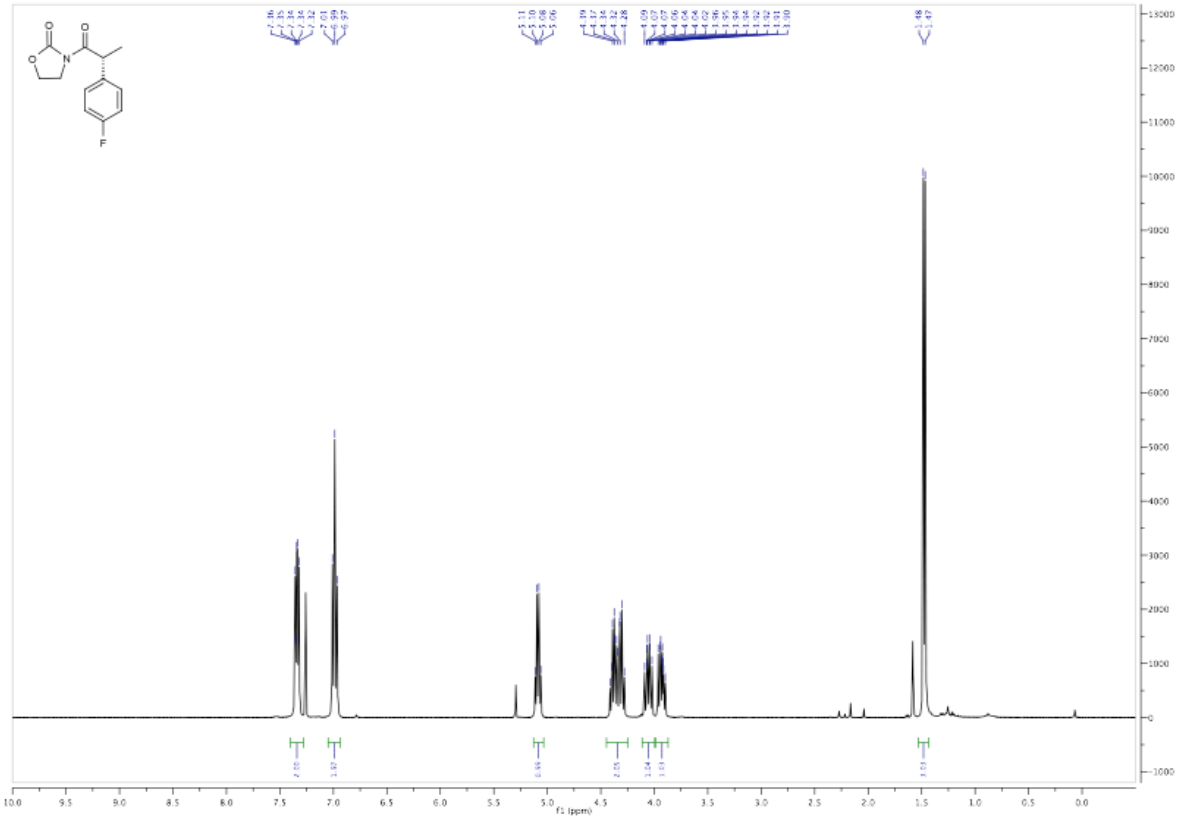

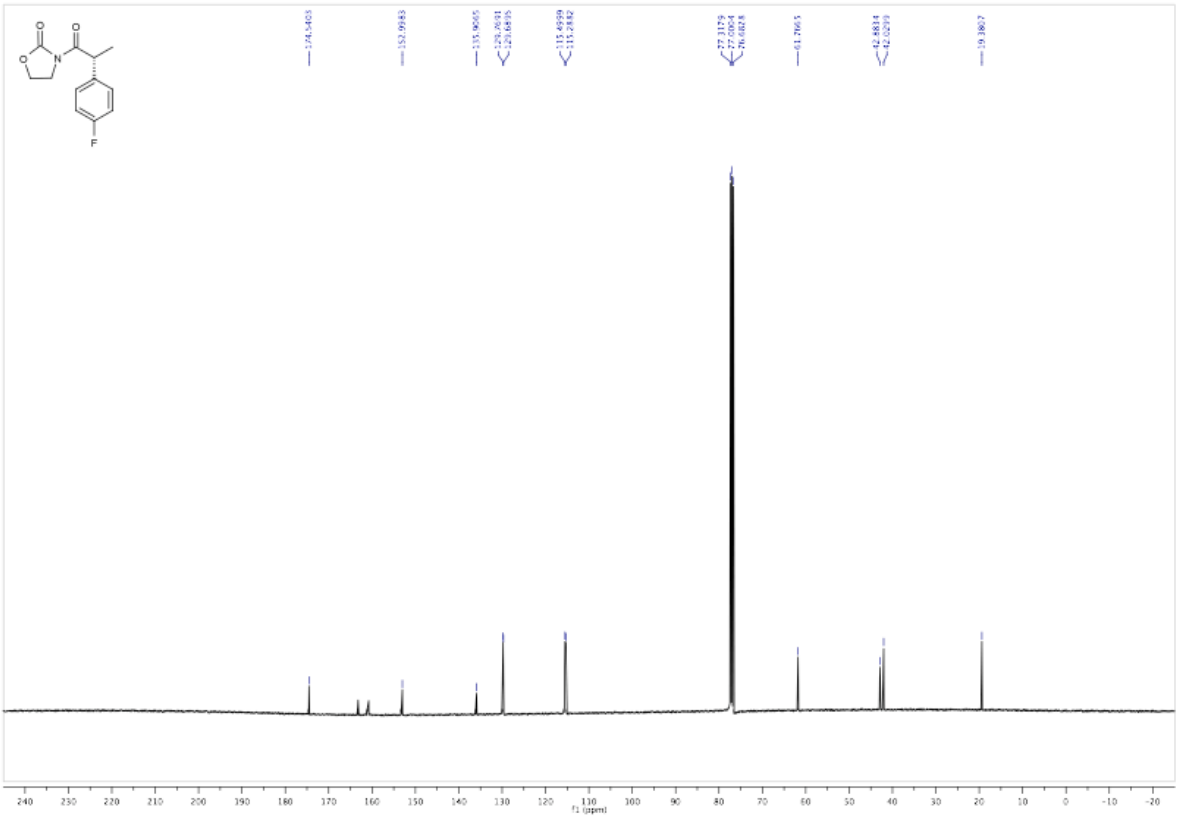

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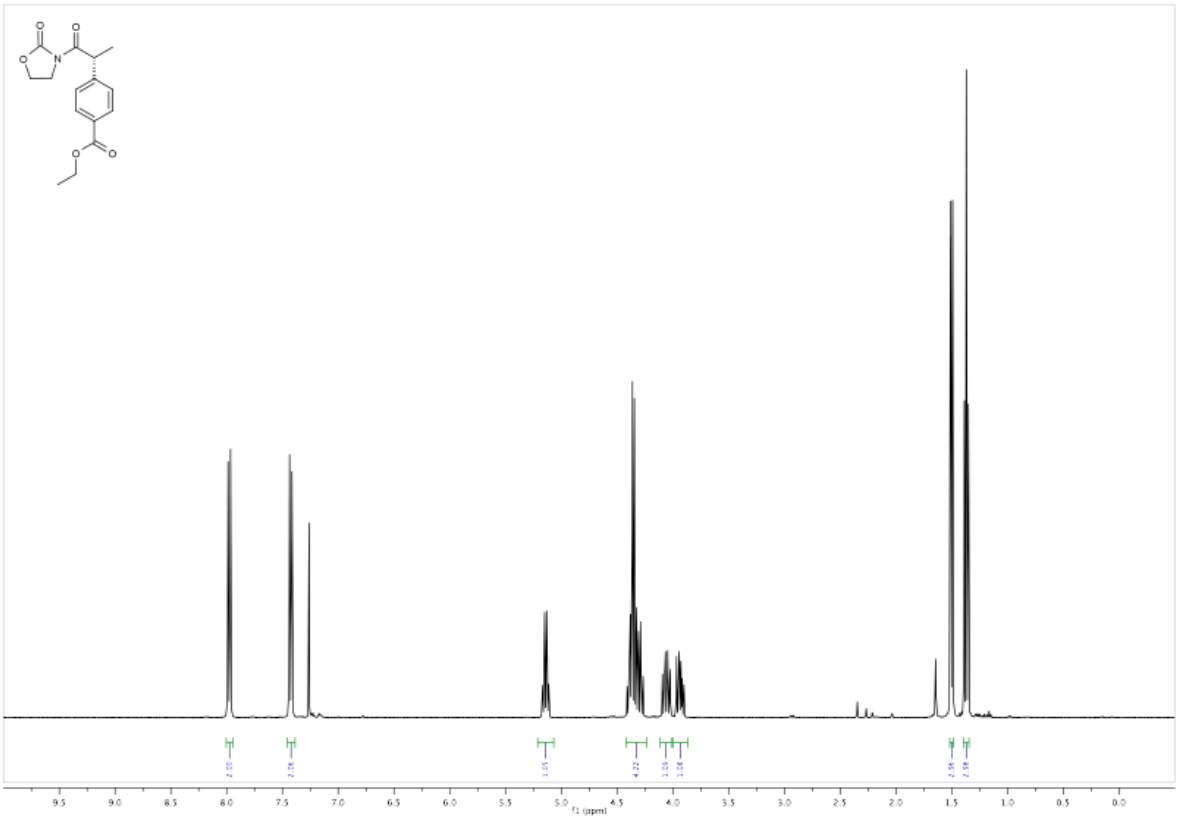

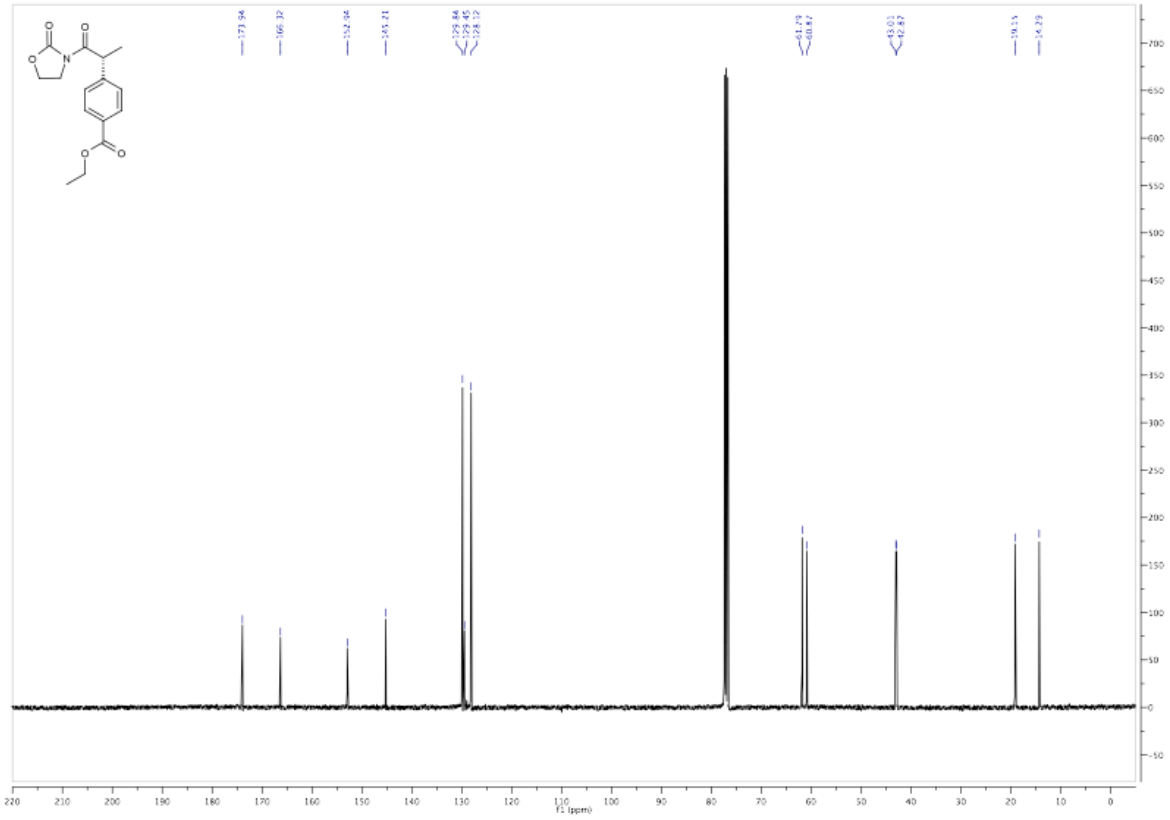

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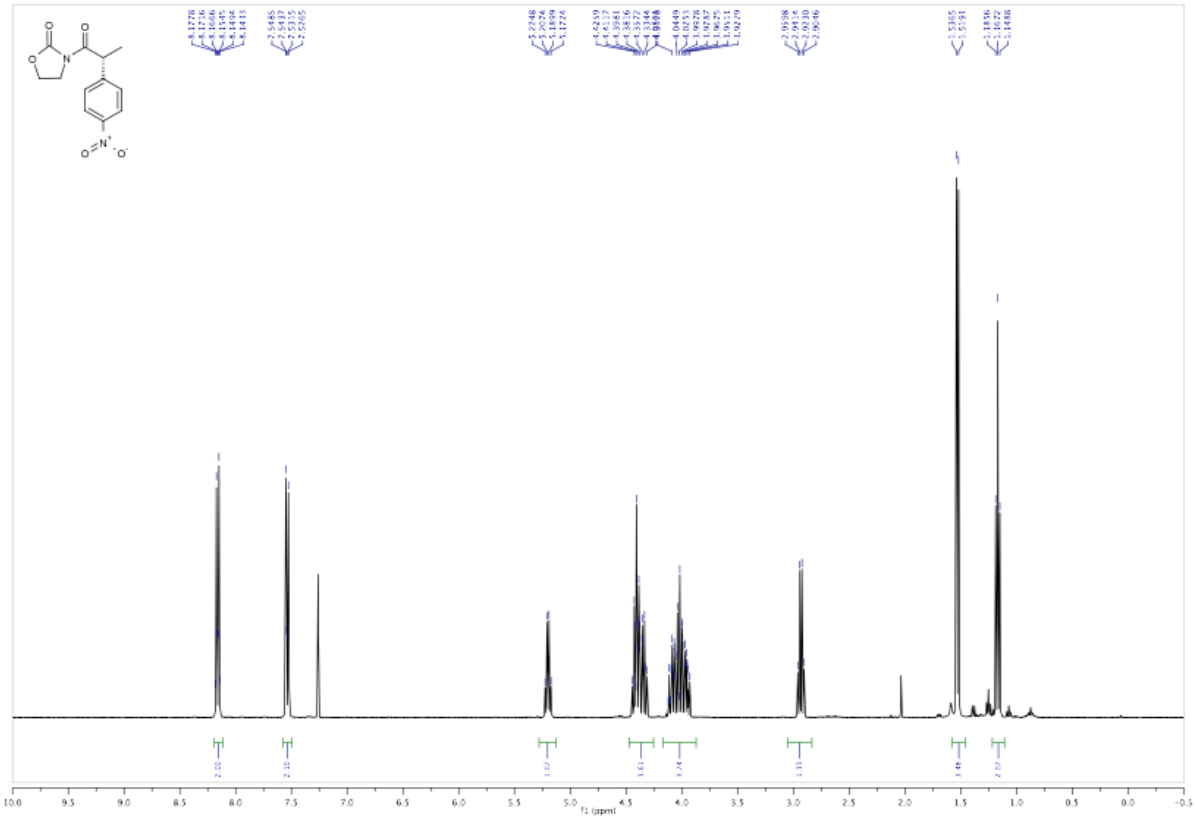

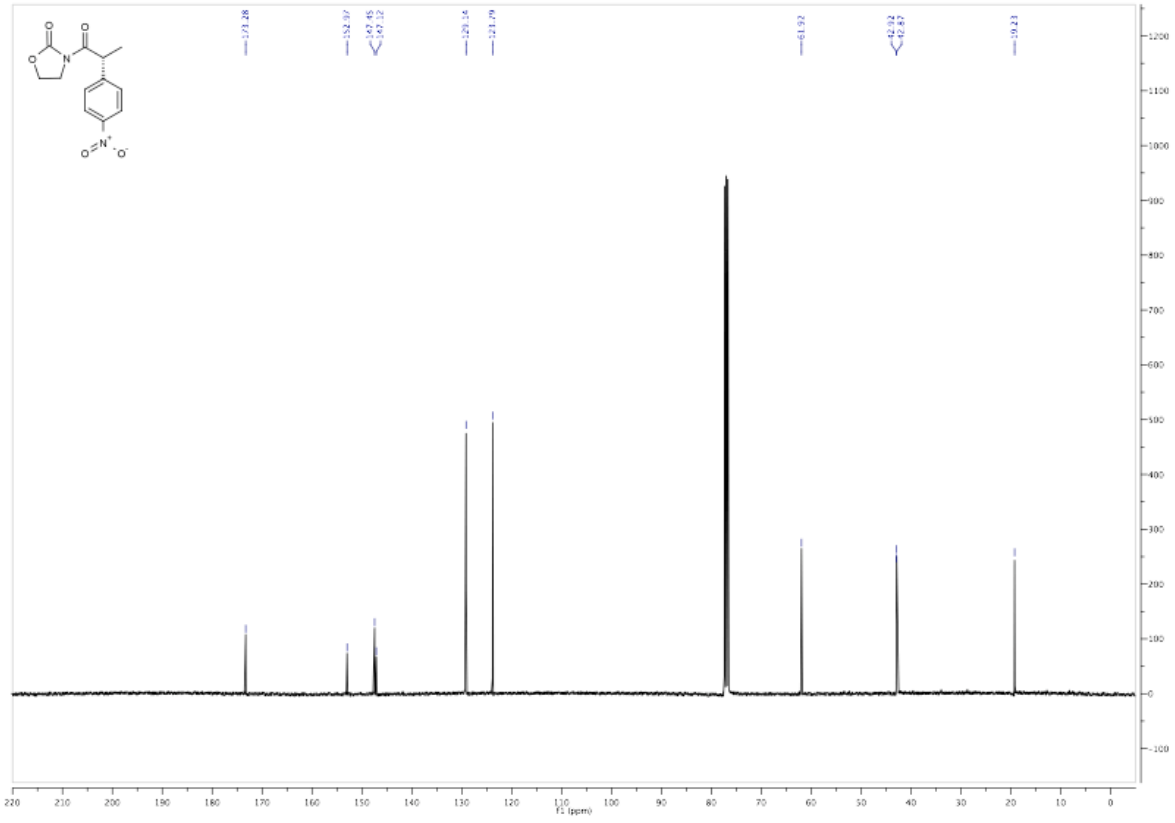

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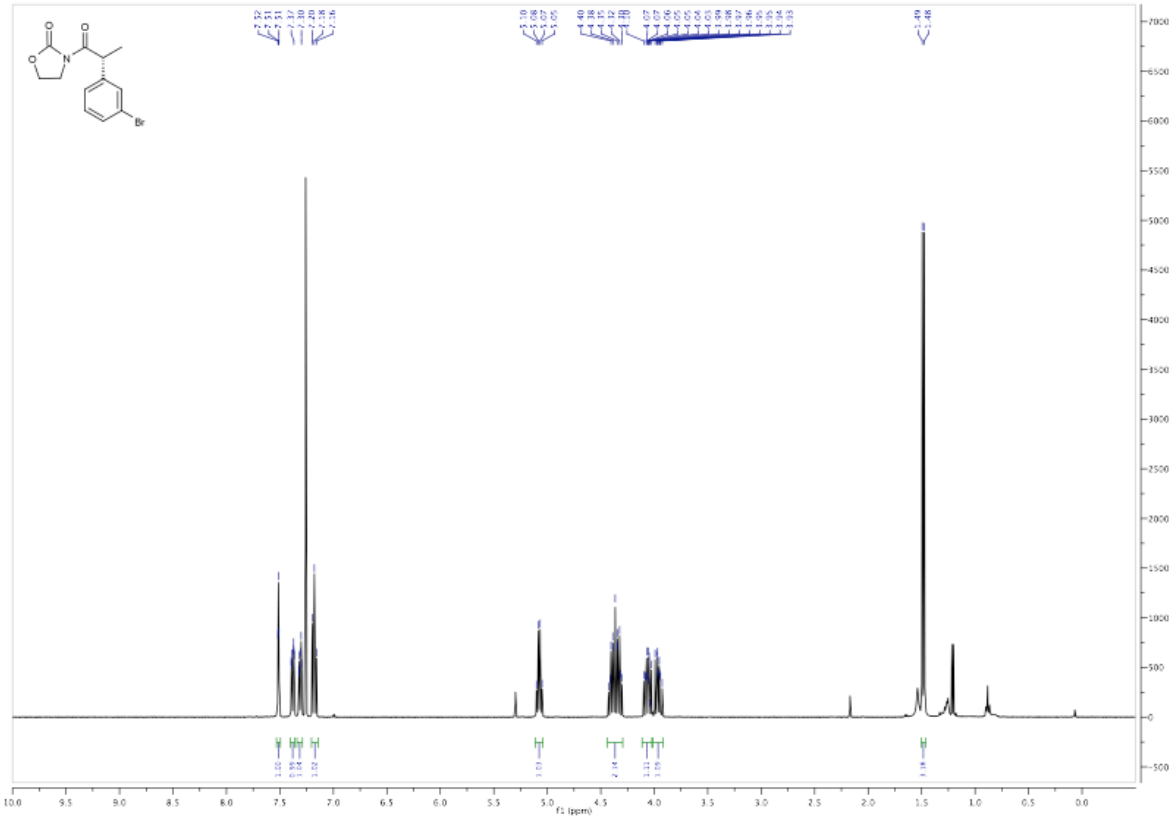

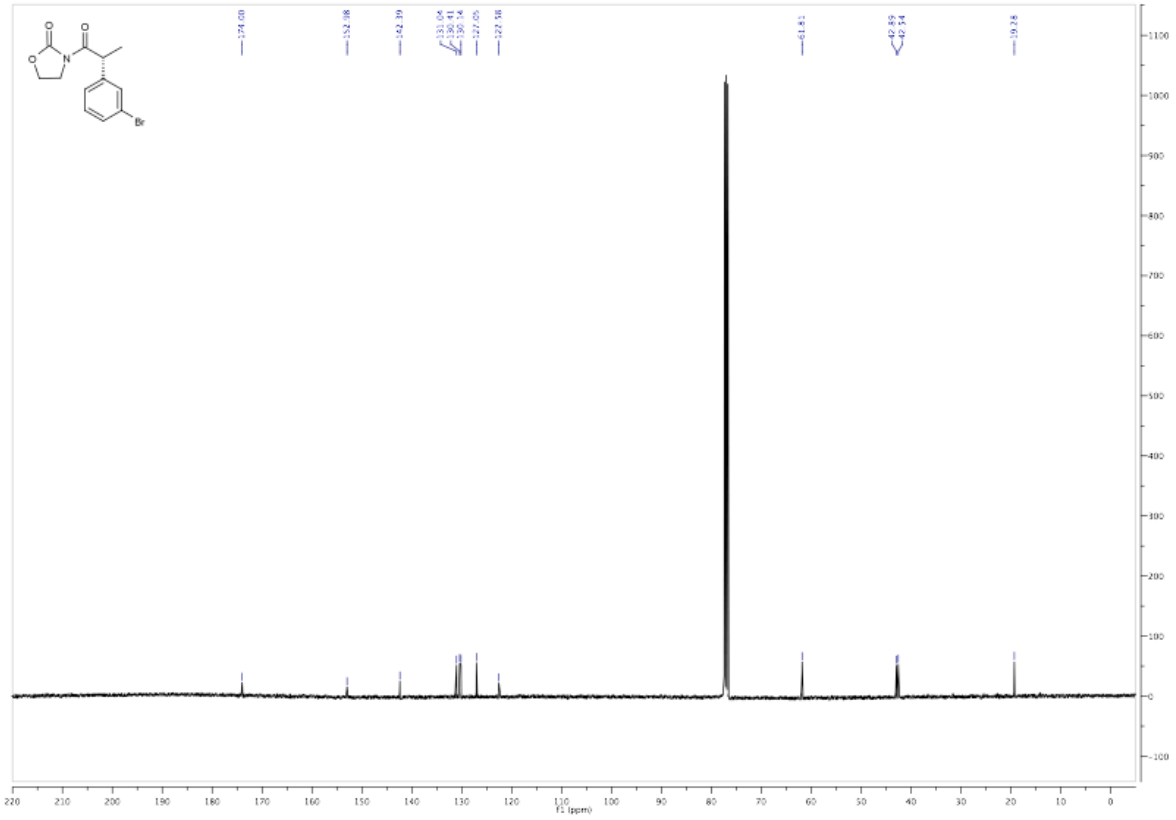

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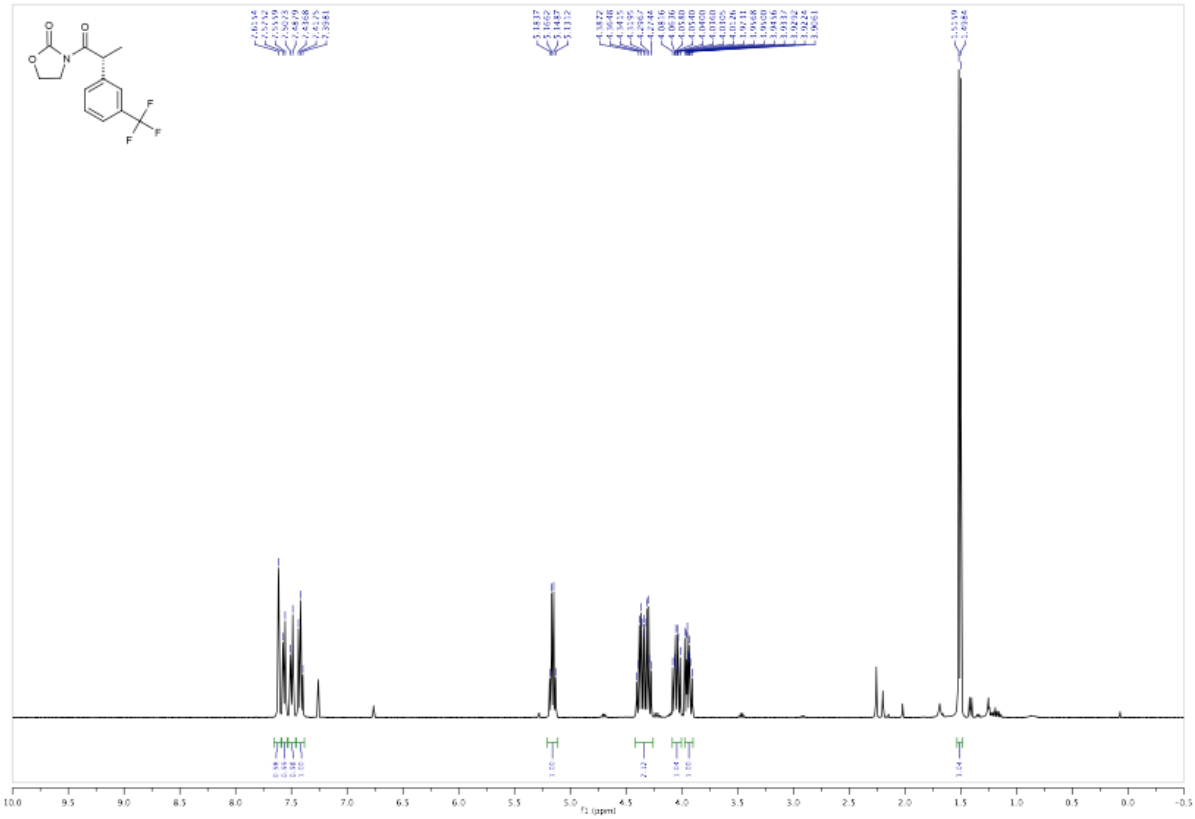

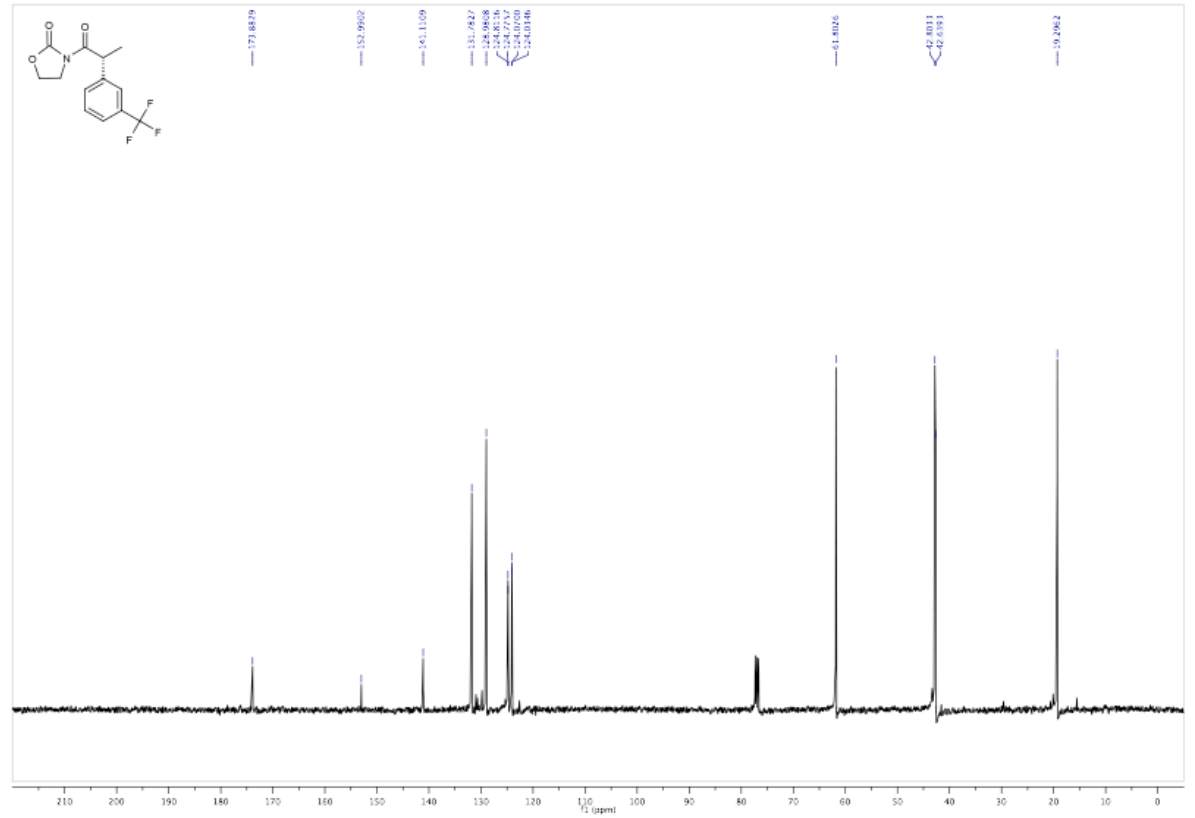

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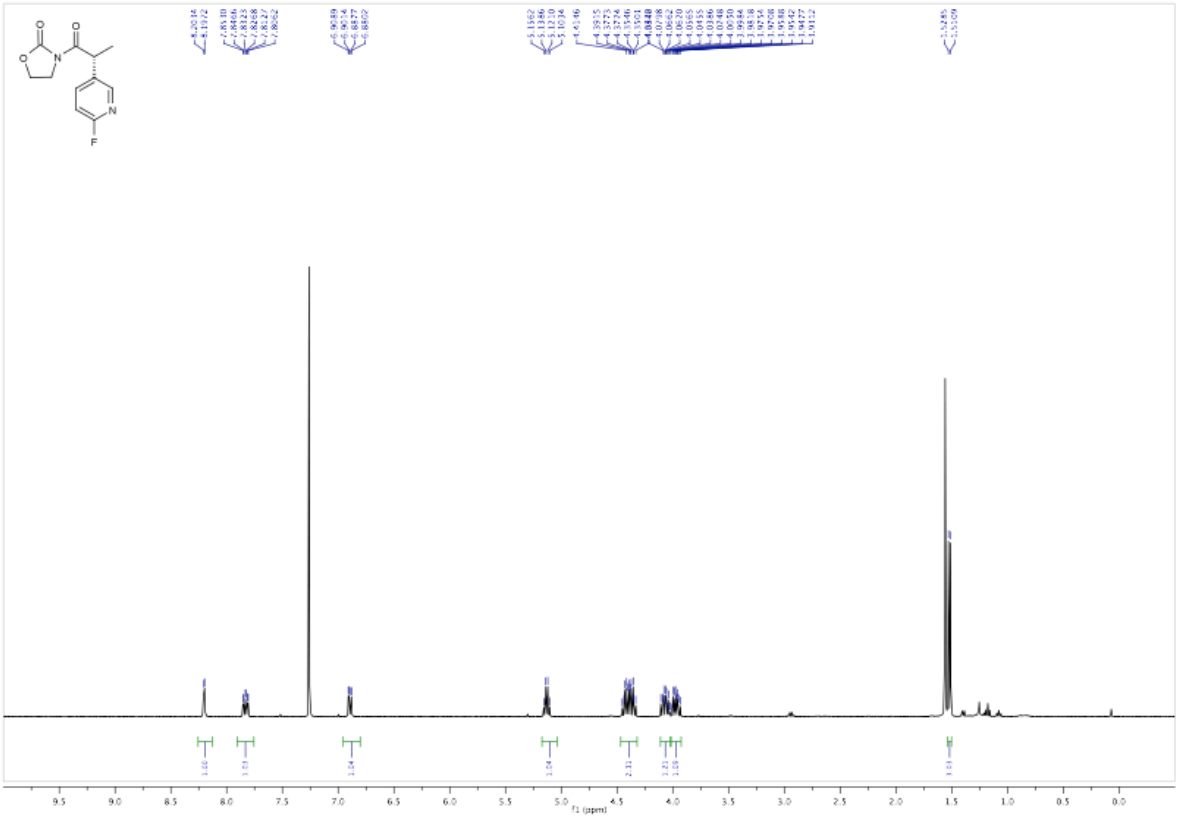

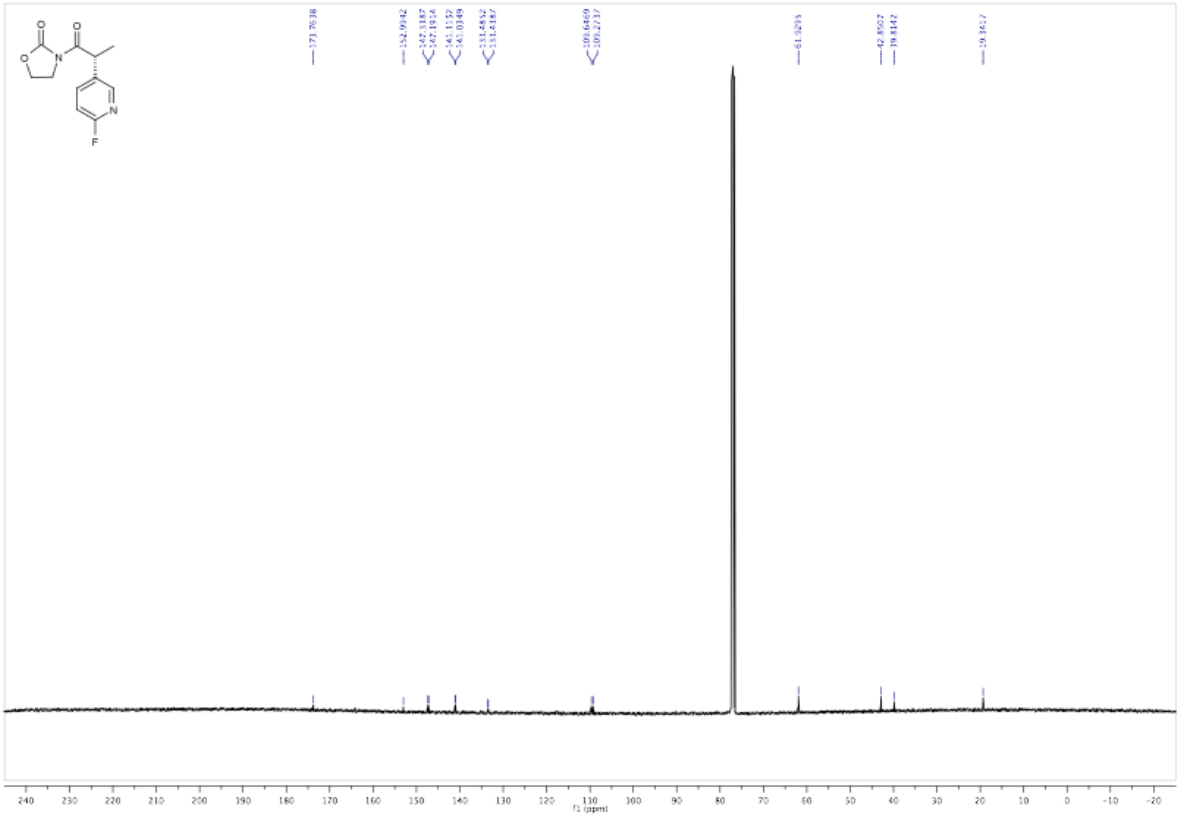

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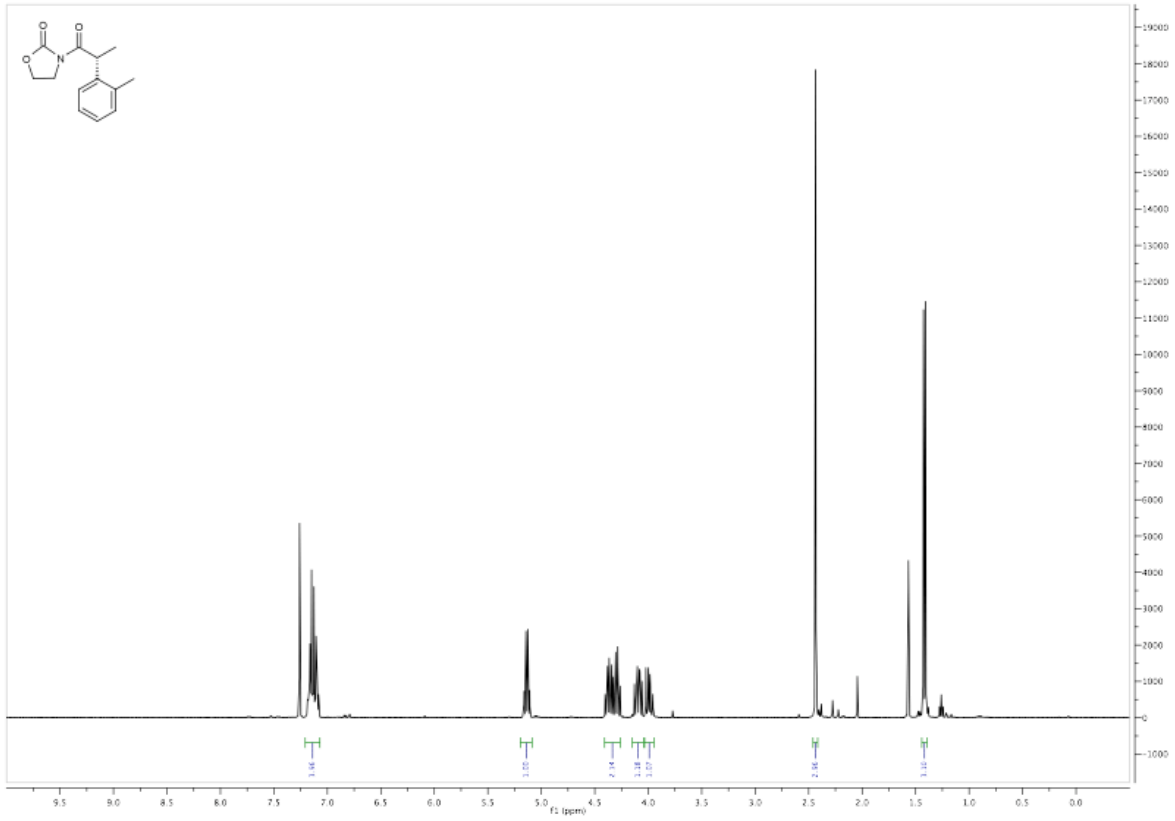
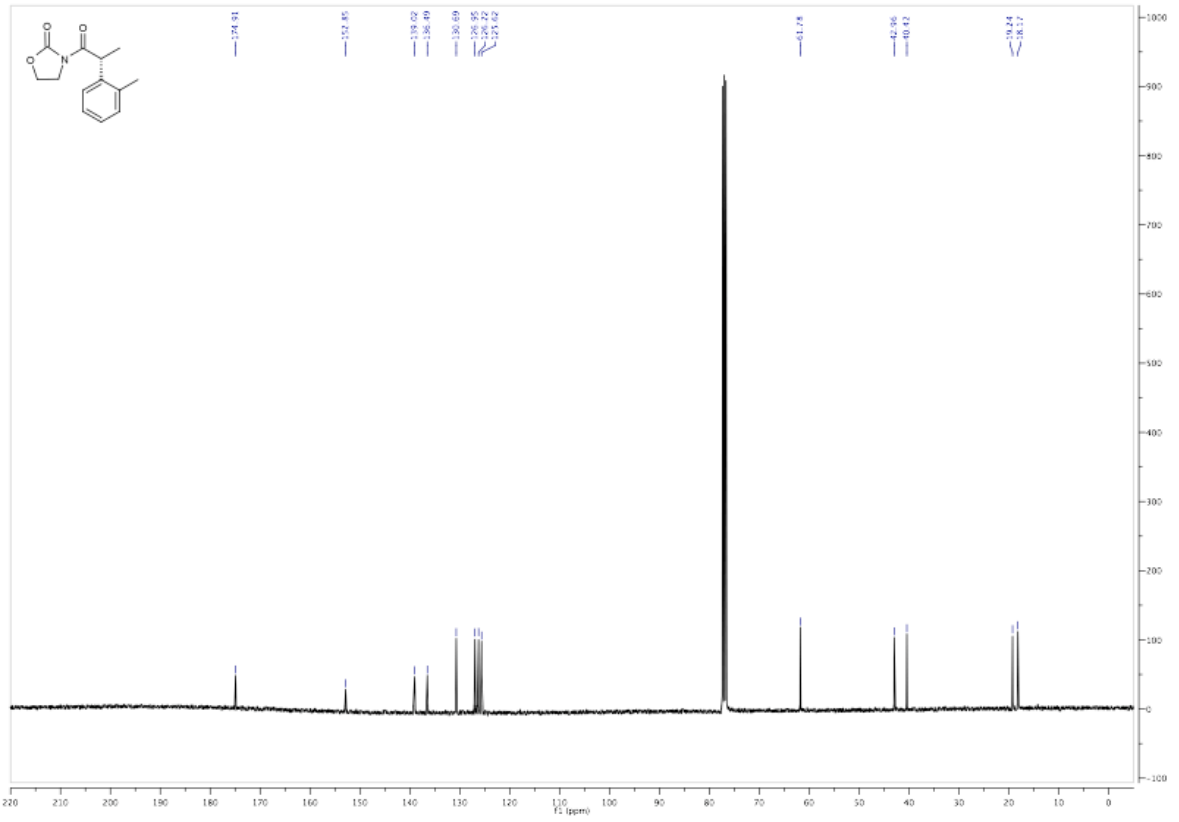
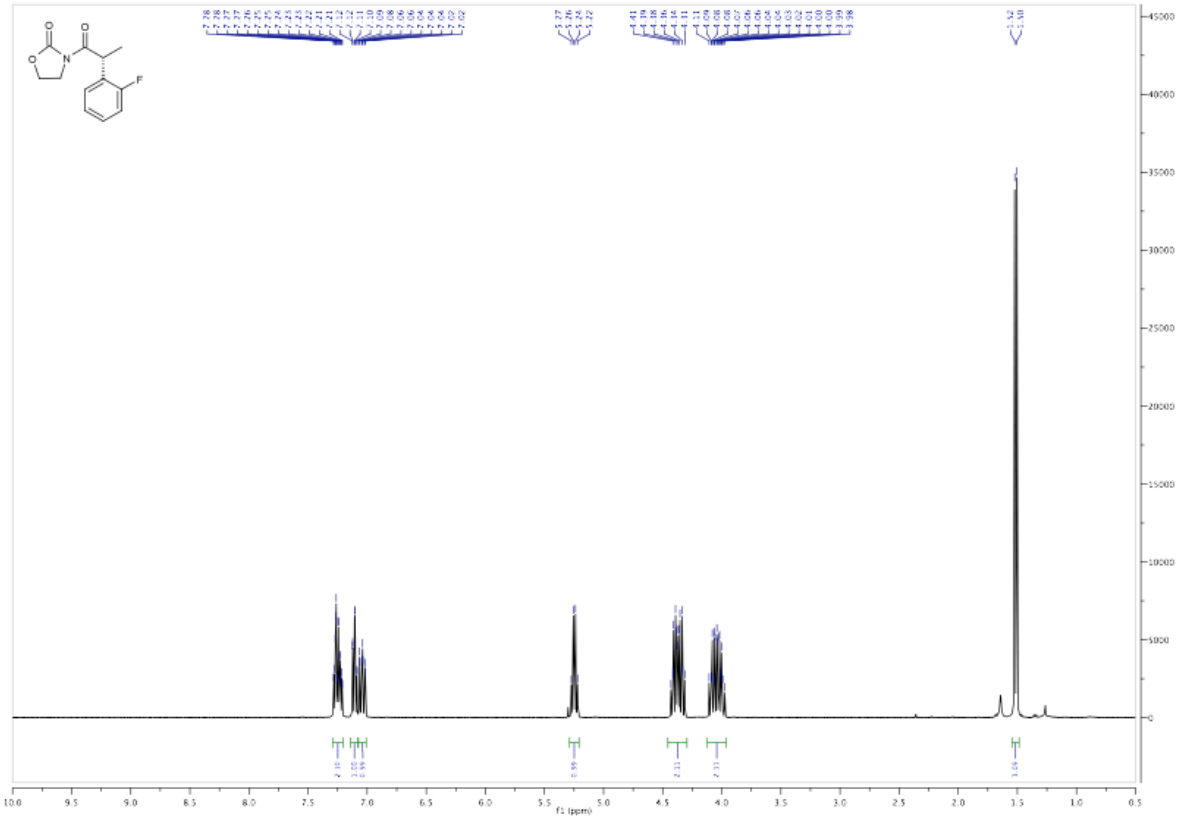
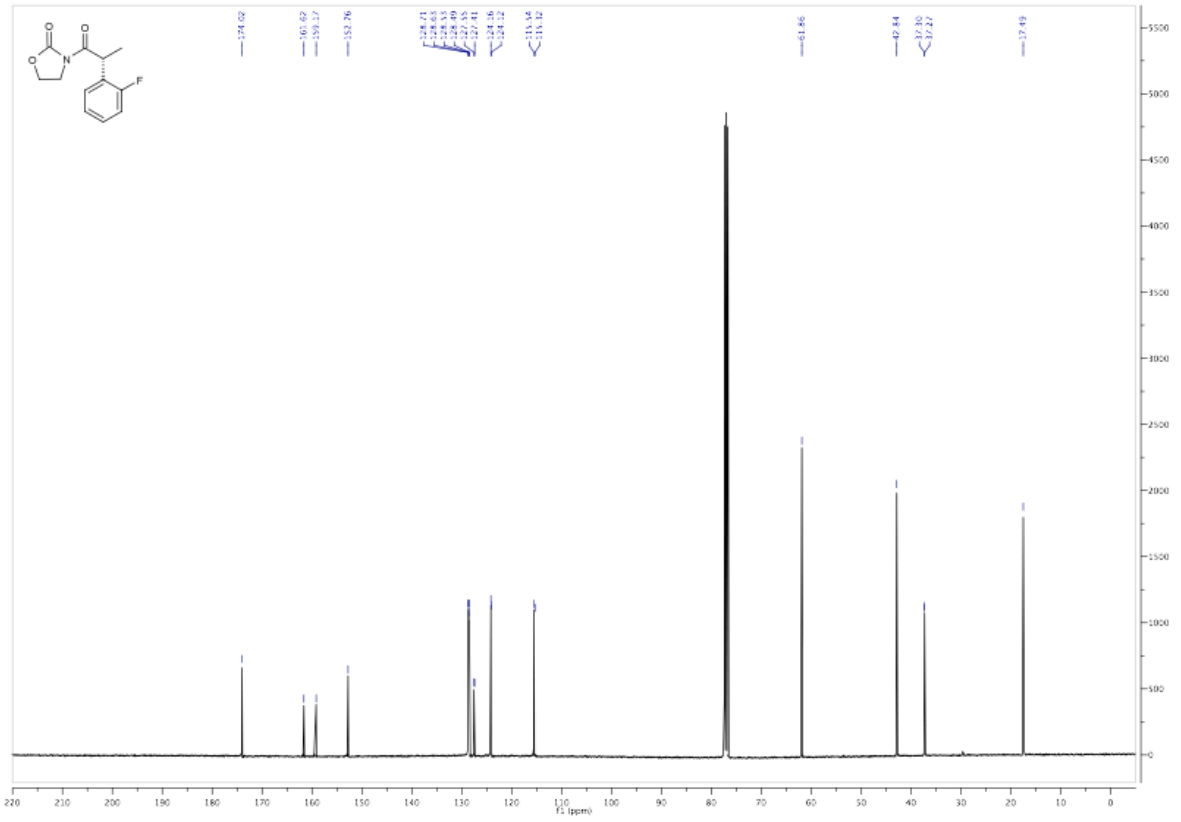


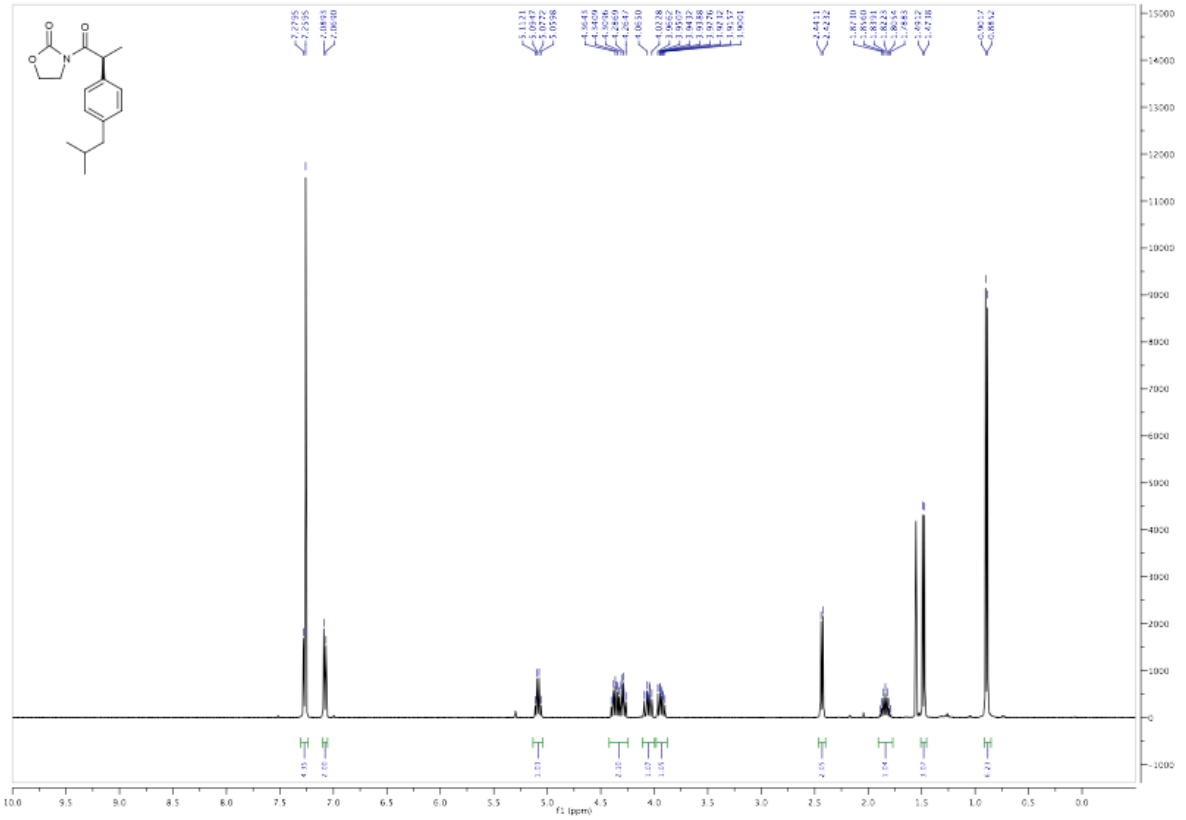


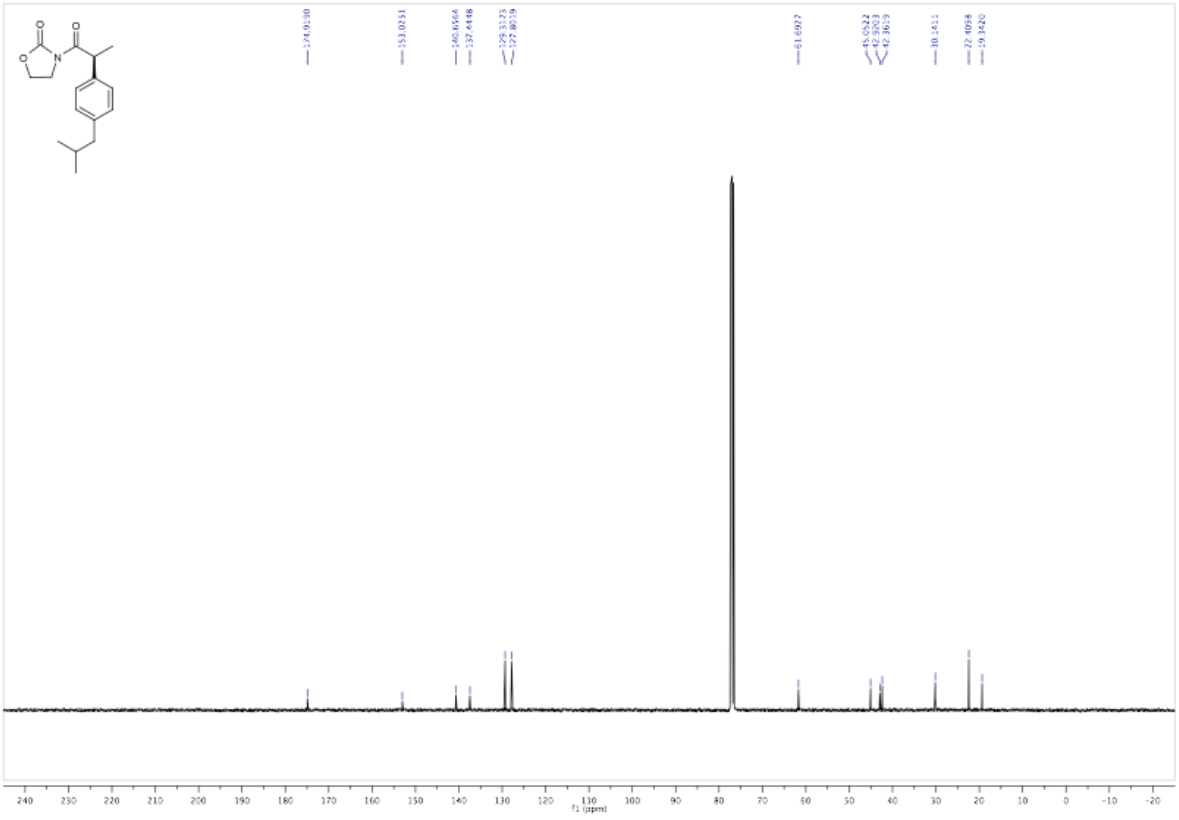
3u



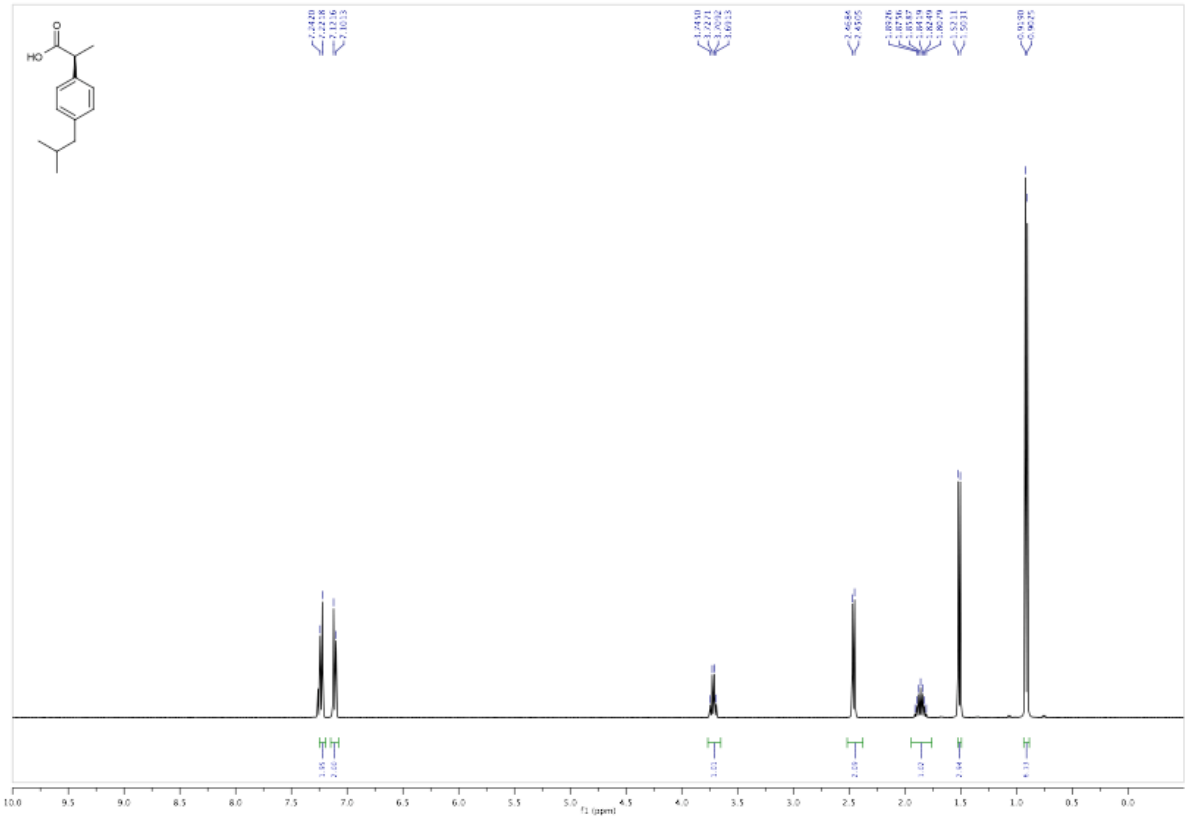


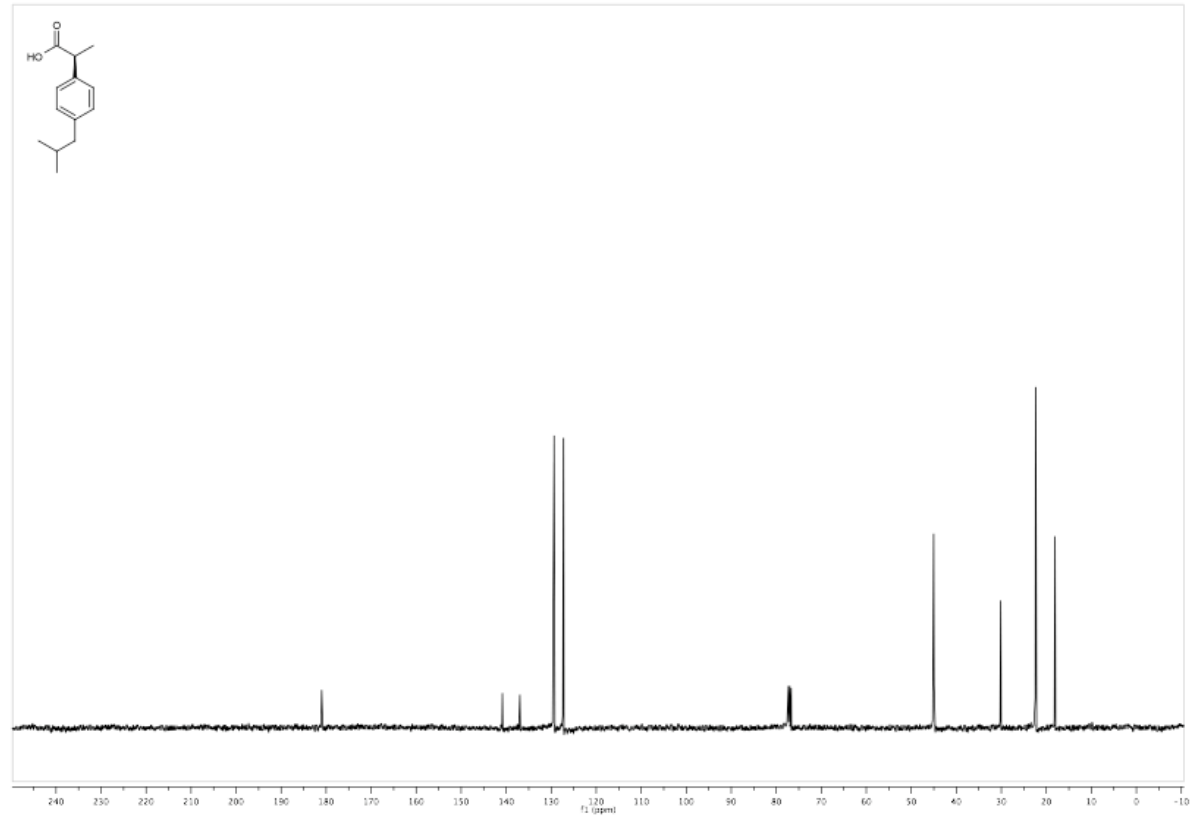
3v



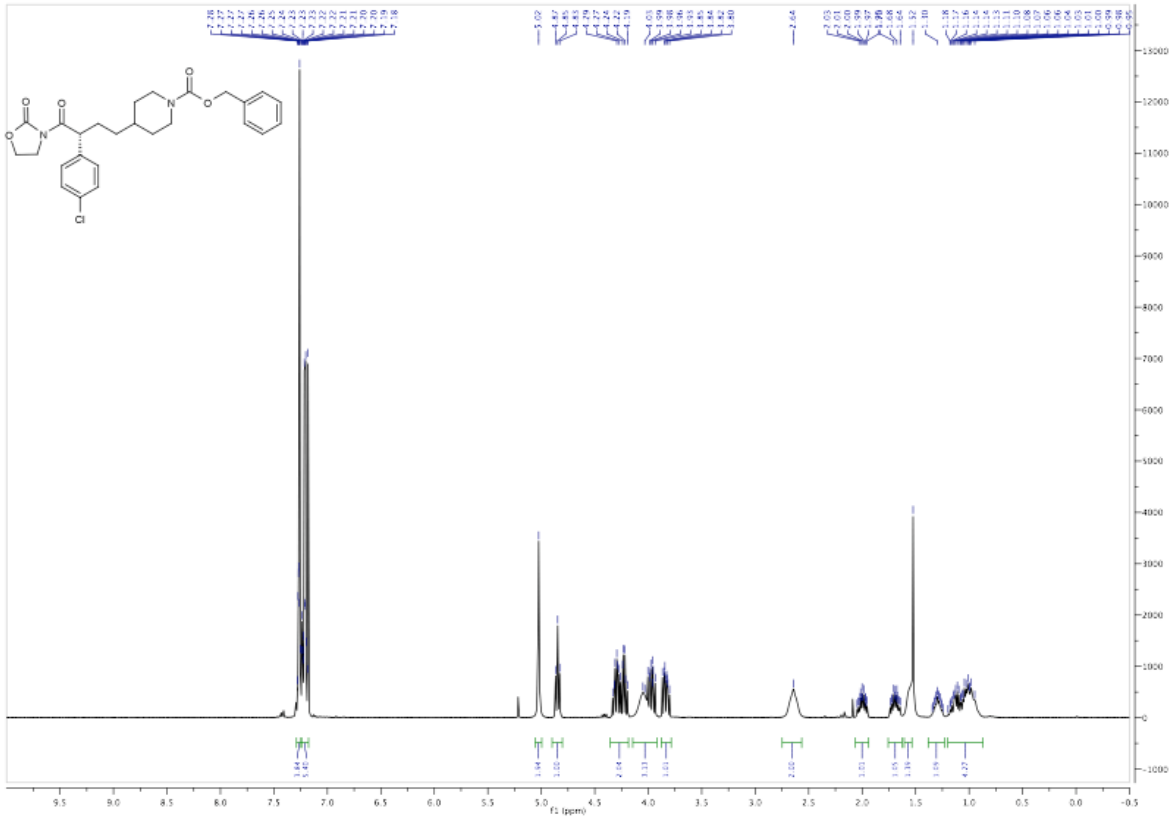


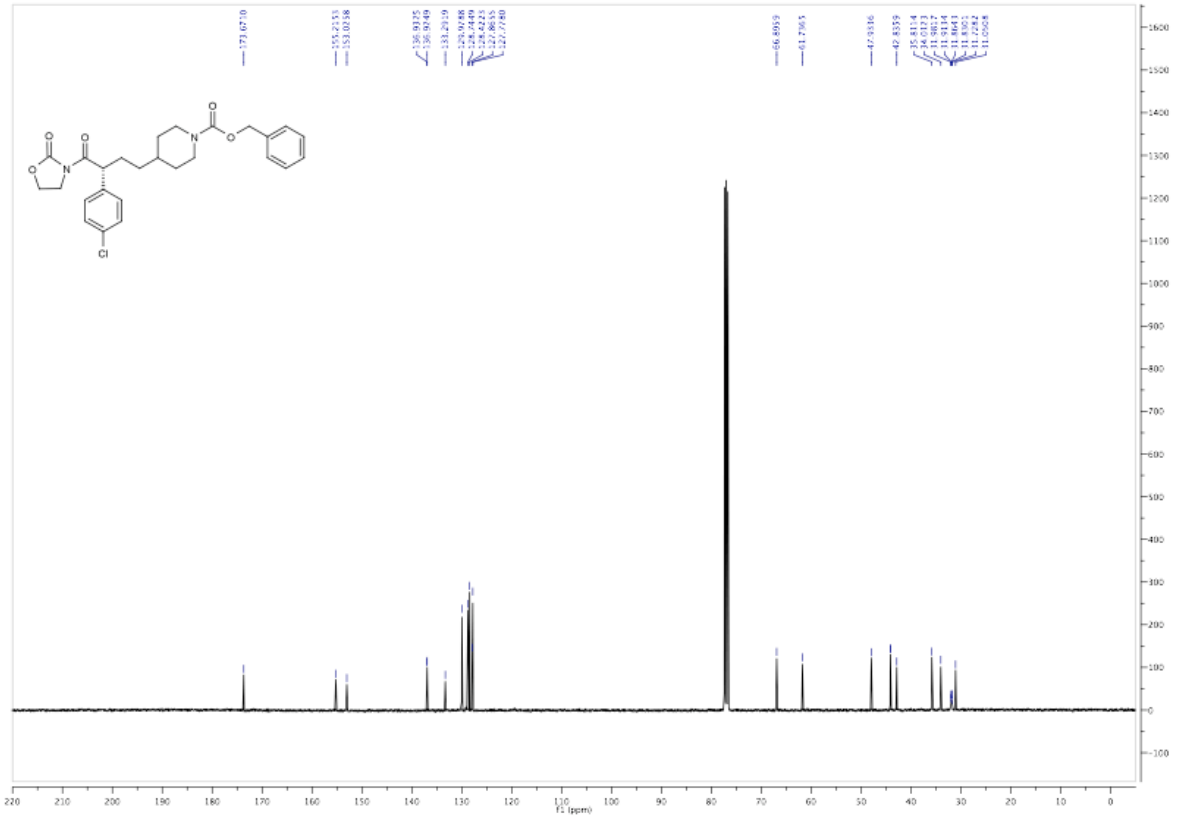
(*S*)-Ibuprofen 5



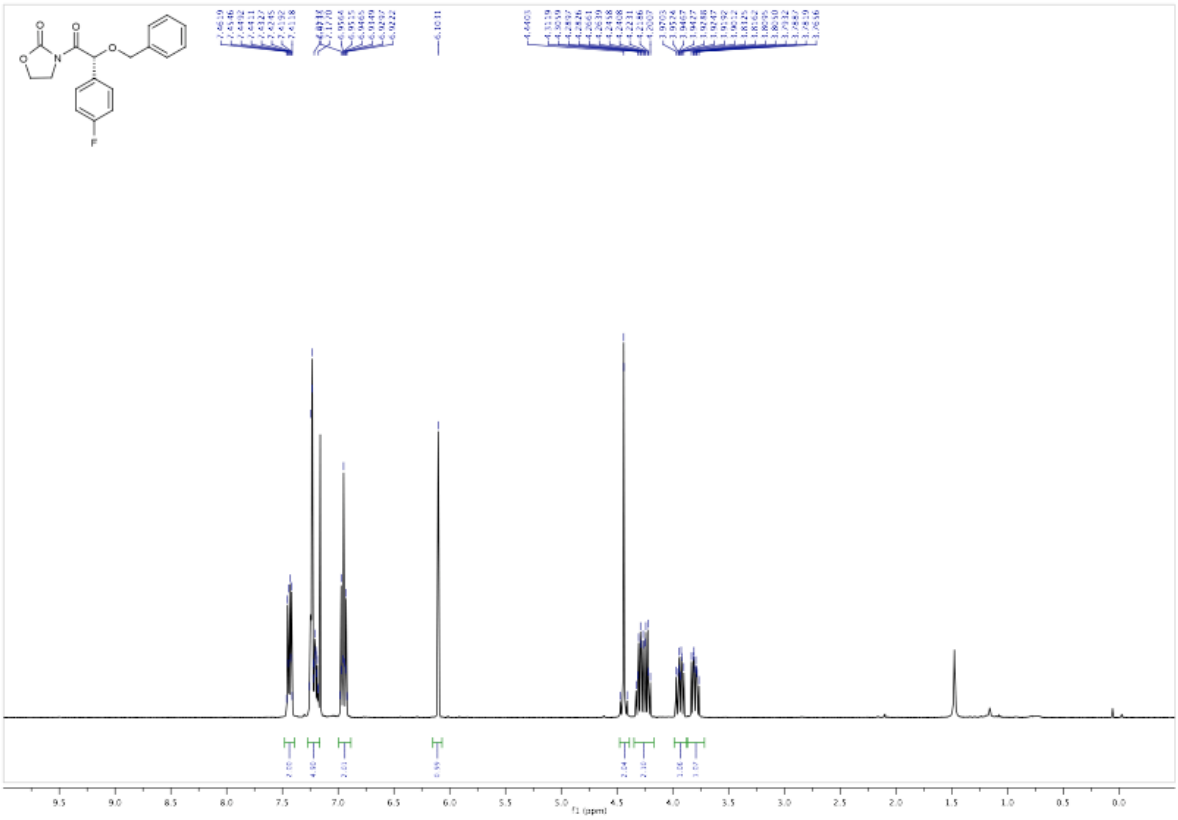


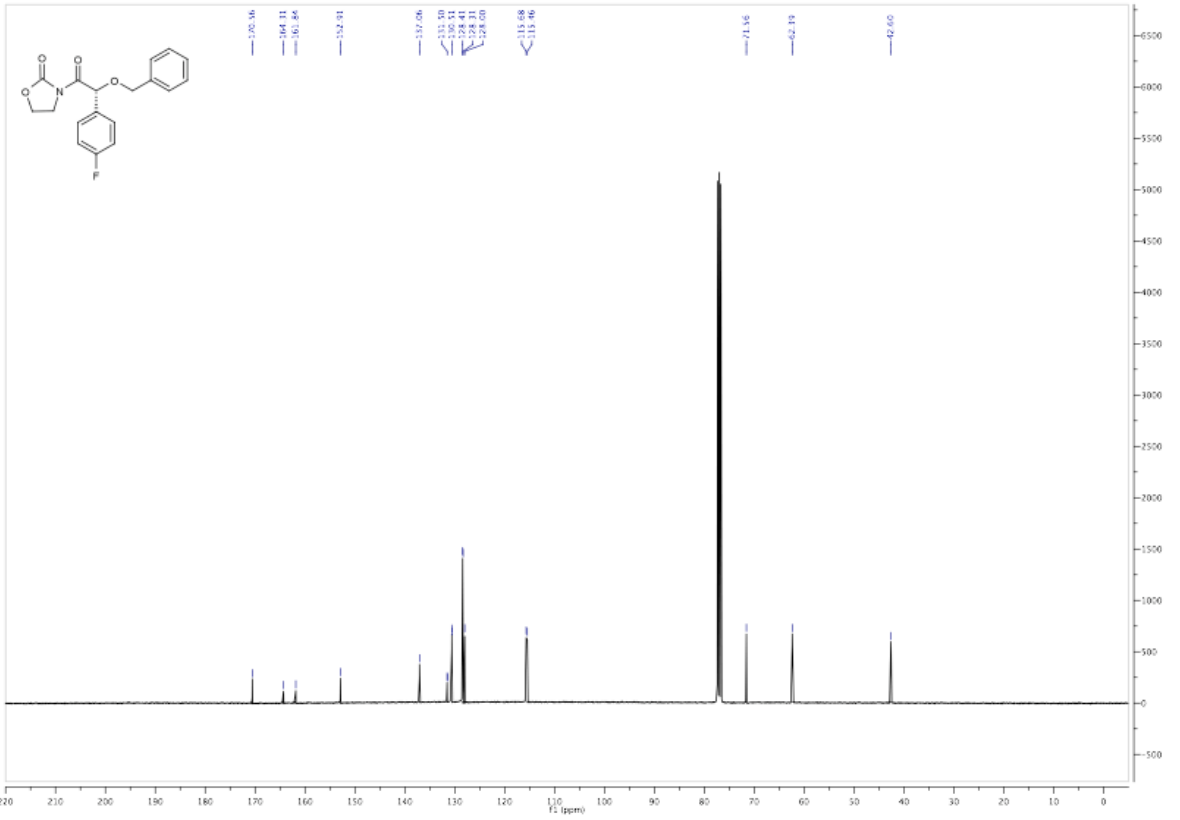
3w



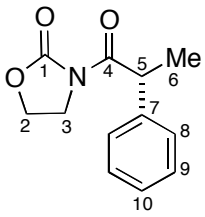


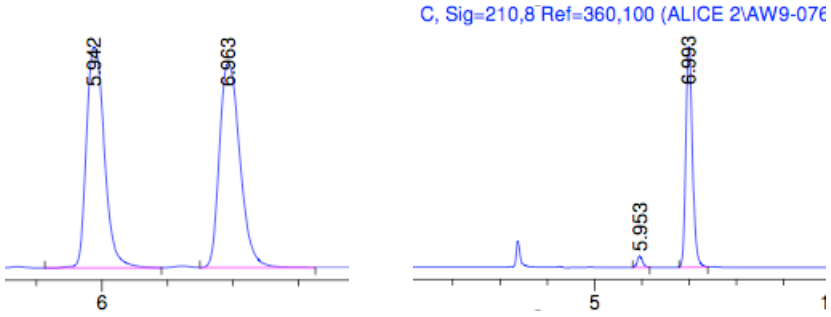
3x





3a





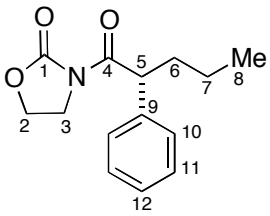
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

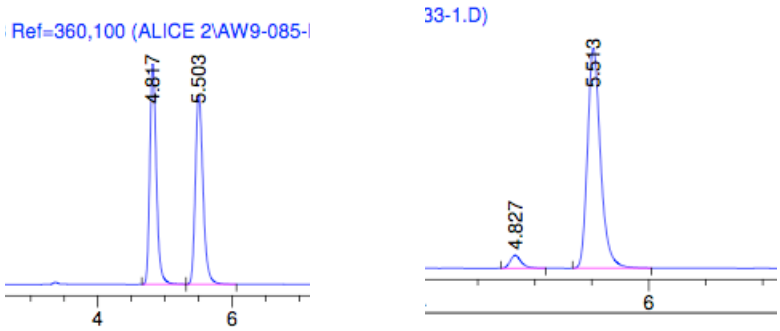
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.942	VV	0.1512	1.99832e4	2096.61670	48.6840
2	6.963	VB	0.1726	2.10636e4	1941.45776	51.3160

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.953	BB	0.1225	43.83521	5.52799	4.0163
2	6.993	BB	0.1492	1047.60388	108.01255	95.9837

3b





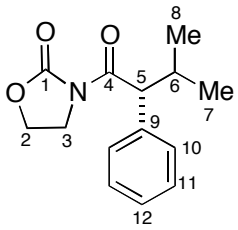
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

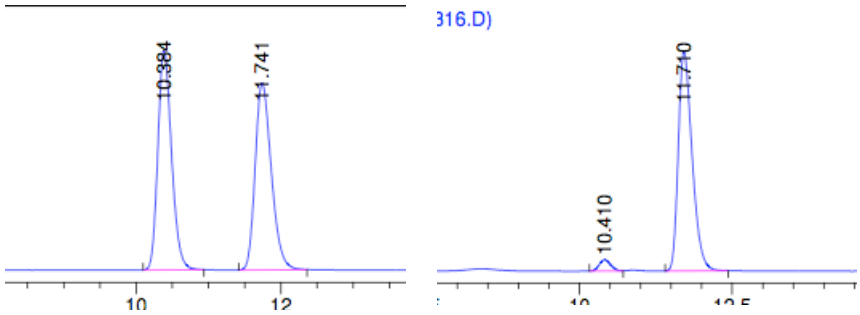
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.817	BV	0.1051	8305.17285	1224.49756	49.7352
2	5.503	VB	0.1231	8393.59863	1051.91235	50.2648

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.827	BB	0.1039	174.06941	25.42773	4.8355
2	5.513	BB	0.1206	3425.78003	431.66425	95.1645

3c





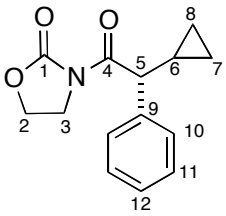
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

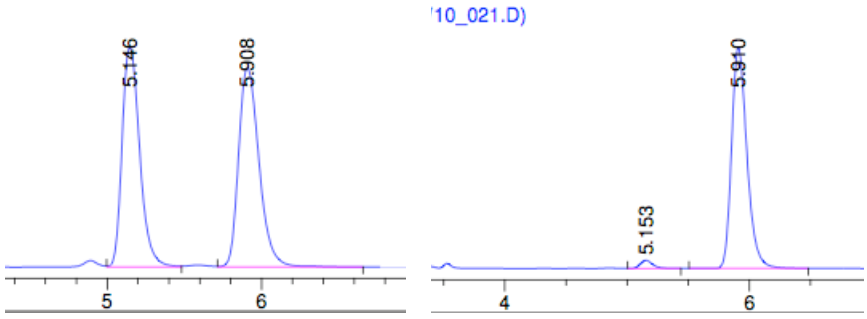
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.384	BB	0.2036	3356.34326	254.90265	49.9276
2	11.741	BB	0.2390	3366.07227	216.98625	50.0724

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.410	BV	0.2045	309.40750	23.36762	4.1913
2	11.710	BB	0.2438	7072.69385	448.99268	95.8087

3d





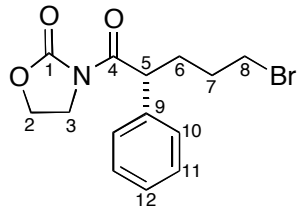
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

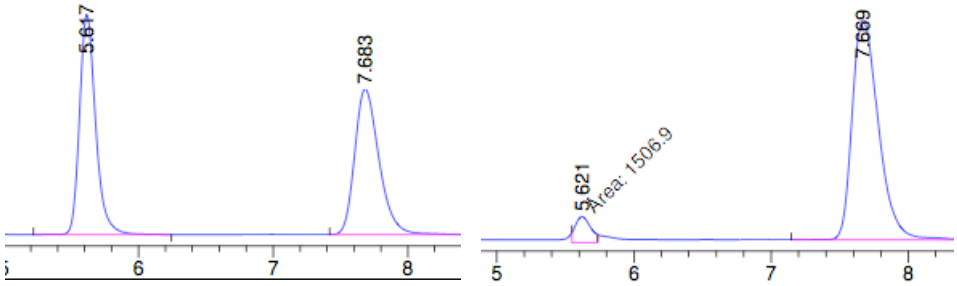
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.146	VV	0.1248	1.55147e4	1951.15491	48.6395
2	5.908	VB	0.1460	1.63826e4	1769.34119	51.3605

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.105	BB	0.1706	13.32258	1.28918	0.1114
2	5.153	VB	0.1102	345.94025	47.92605	2.8929
3	5.910	BB	0.1342	1.15991e4	1324.64563	96.9957

3e





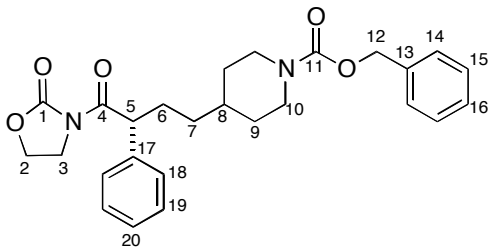
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

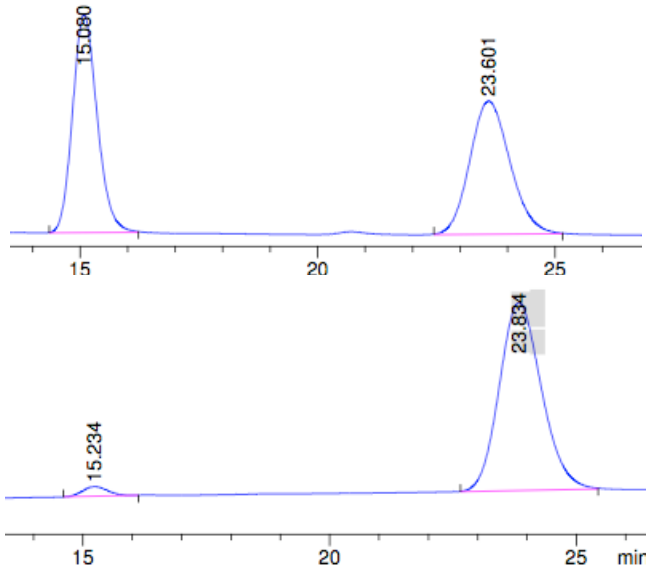
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.617	BB	0.1288	9861.97461	1189.09424	49.8551
2	7.683	BV	0.1929	9919.30957	787.62122	50.1449

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.621	MM	0.1290	1506.89722	194.63623	6.4260
2	7.669	VB	0.2083	2.19432e4	1637.74585	93.5740

3f





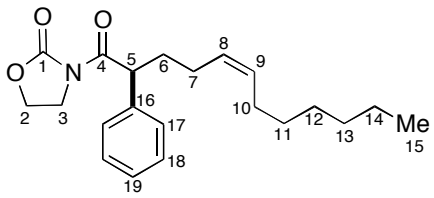
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

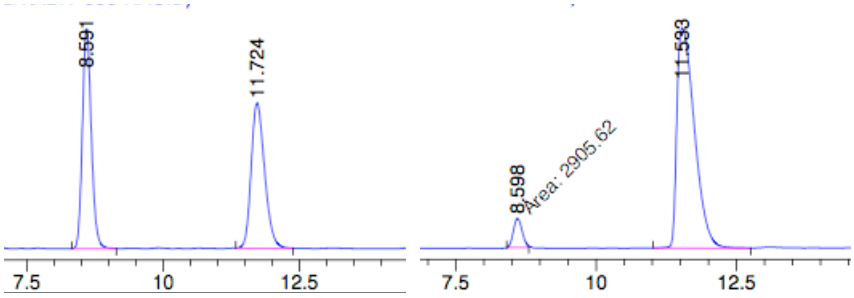
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.080	BB	0.5589	5632.76611	156.11893	50.0096
2	23.601	BB	0.9200	5630.60645	95.48384	49.9904

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.234	BB	0.5218	268.80035	7.65213	2.9557
2	23.834	BB	0.9191	8825.37109	148.97430	97.0443

3g





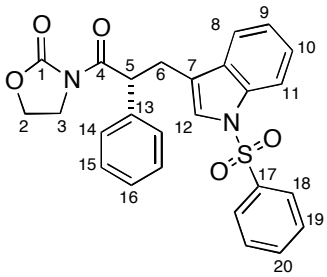
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

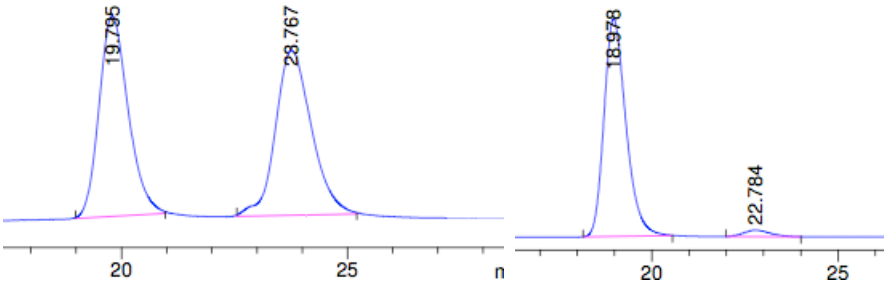
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.591	BB	0.1769	3750.74243	329.37015	49.9197
2	11.724	BB	0.2674	3762.80225	217.97017	50.0803

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.598	MM	0.1792	2905.61792	270.30432	6.0941
2	11.533	VB	0.3433	4.47734e4	2046.36902	93.9059

3h





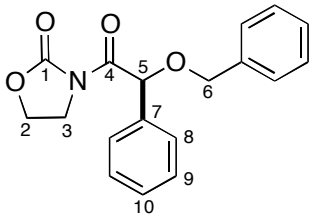
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

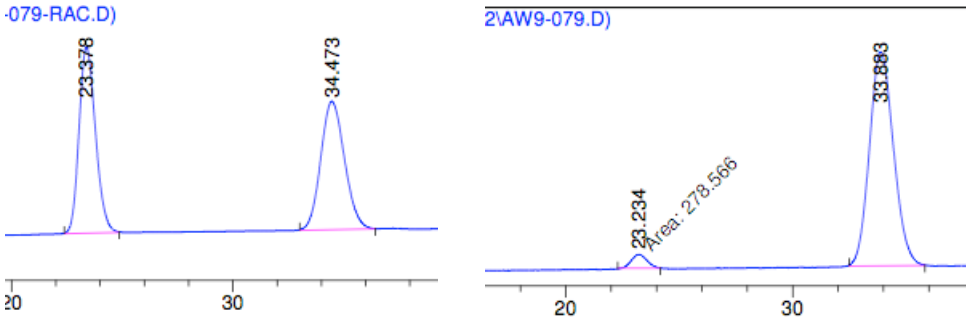
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.795	BB	0.6802	2047.13379	45.56203	49.5990
2	23.767	BB	0.8273	2080.23169	37.49216	50.4010

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.978	BB	0.6279	1.32336e4	323.06815	96.5175
2	22.784	BB	0.7126	477.49567	9.62763	3.4825

3i





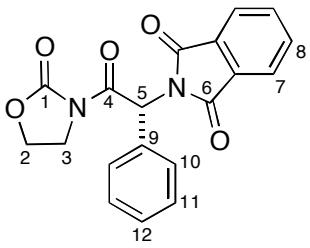
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

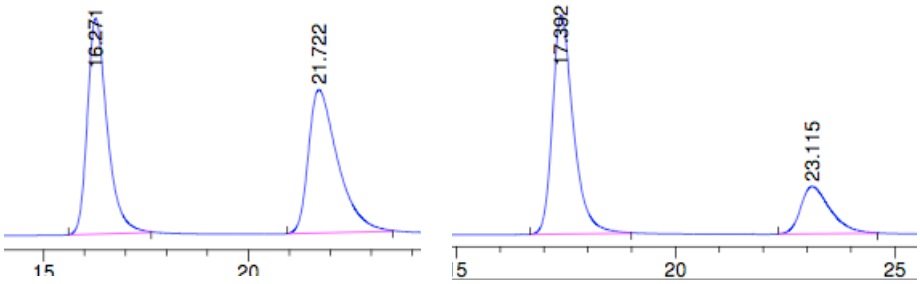
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.378	BB	0.8023	8902.21875	172.43269	50.1144
2	34.473	BB	1.1512	8861.56152	118.94173	49.8856

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.234	MM	0.7990	278.56564	5.81037	3.9770
2	33.883	BB	1.1404	6725.87695	91.19959	96.0230

3j





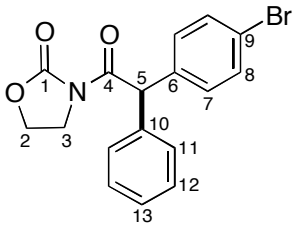
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

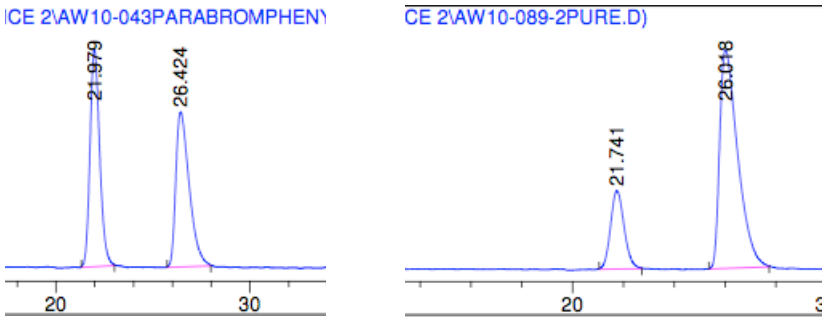
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.271	BB	0.5103	6073.30420	180.59450	50.3245
2	21.722	BB	0.7466	5994.97754	120.05306	49.6755

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.392	BB	0.5011	1.23897e4	373.38782	76.4729
2	23.115	BB	0.7079	3811.73828	80.86451	23.5271

3k





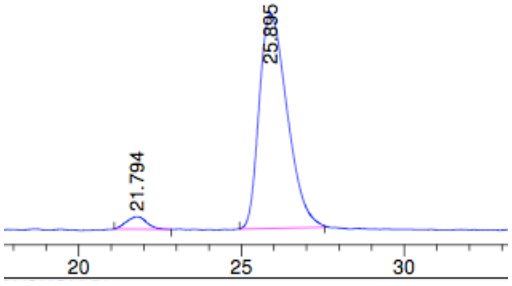
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.979	BB	0.5311	3632.99927	105.15147	50.0972
2	26.424	BB	0.7293	3618.89941	75.22407	49.9028

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.741	BB	0.5721	1804.61719	48.72461	21.2167
2	26.018	BB	0.7386	6701.00977	135.58902	78.7833

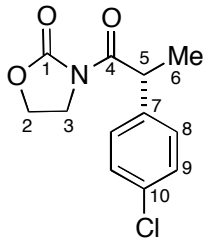
following recrystallisation from MeOH:

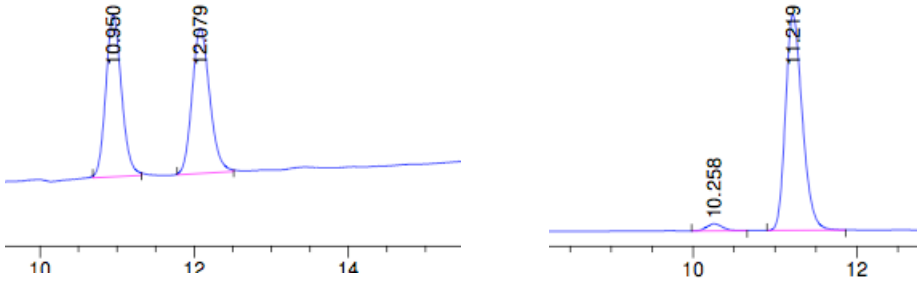


Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.794	BB	0.6160	194.78024	4.43847	4.0892
2	25.895	BB	0.9353	4568.45117	76.22095	95.9108

3l





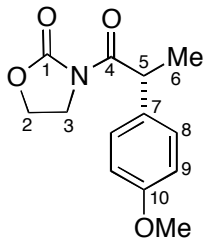
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

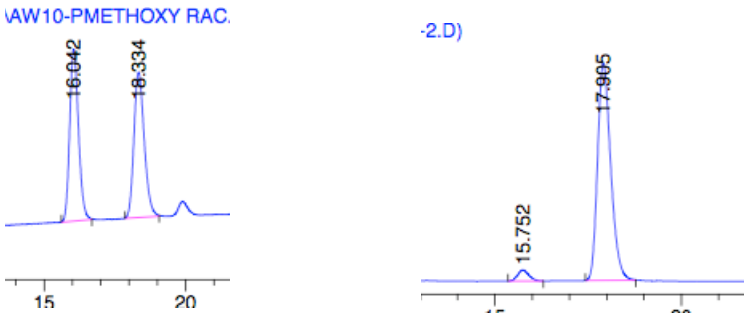
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.950	BB	0.2182	333.91916	23.72912	49.8147
2	12.079	BB	0.2449	336.40277	21.22920	50.1853

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.258	BB	0.2059	231.14499	17.29241	2.9535
2	11.219	BB	0.2208	7594.86670	531.29828	97.0465

3m





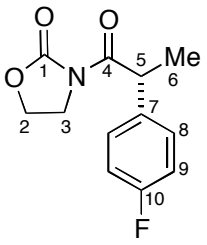
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

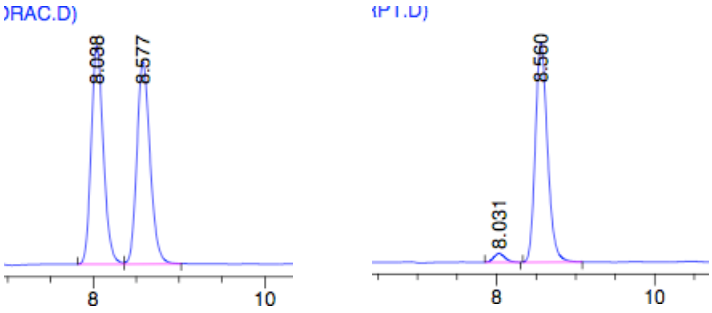
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.823	BB	0.3319	3656.57349	170.73663	50.1581
2	18.109	BB	0.3867	3633.52637	144.81297	49.8419

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.752	BB	0.3233	202.99629	9.73317	4.1508
2	17.905	BB	0.3780	4687.48633	191.24690	95.8492

3n





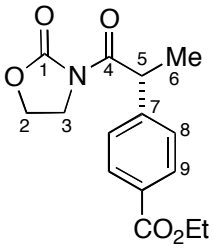
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

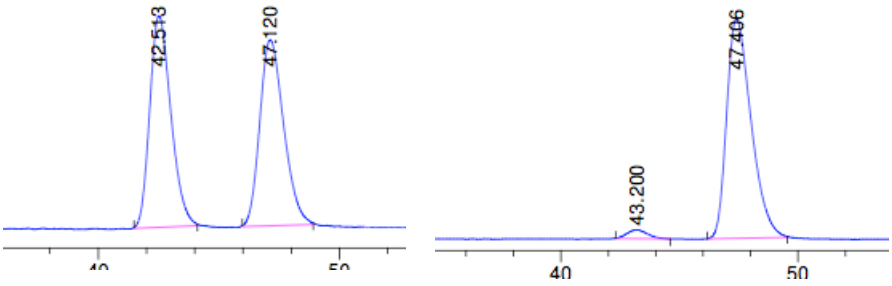
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.038	BV	0.1530	2403.73389	243.86719	49.9236
2	8.577	VB	0.1645	2411.08862	225.87750	50.0764

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.031	BB	0.1519	103.38044	10.59226	3.5617
2	8.560	BB	0.1646	2799.19580	262.01013	96.4383

3o





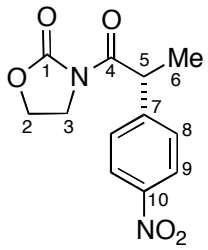
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

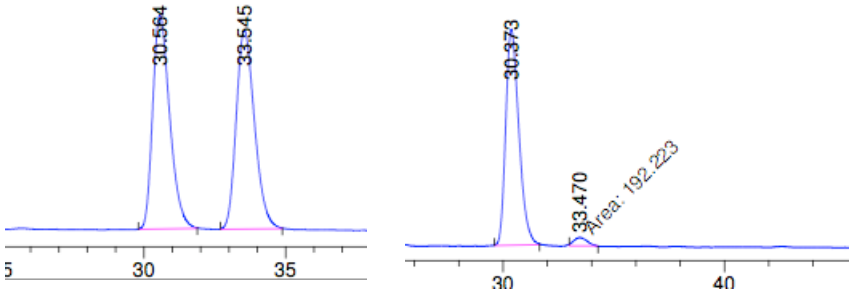
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	42.513	BB	0.9130	3057.27783	50.16308	50.1330
2	47.120	BB	1.0377	3041.06201	44.14628	49.8670

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	42.488	BB	0.9228	2509.01001	41.41380	3.8265
2	46.151	BB	1.1609	6.30602e4	804.12787	96.1735

3p





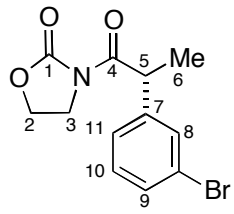
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

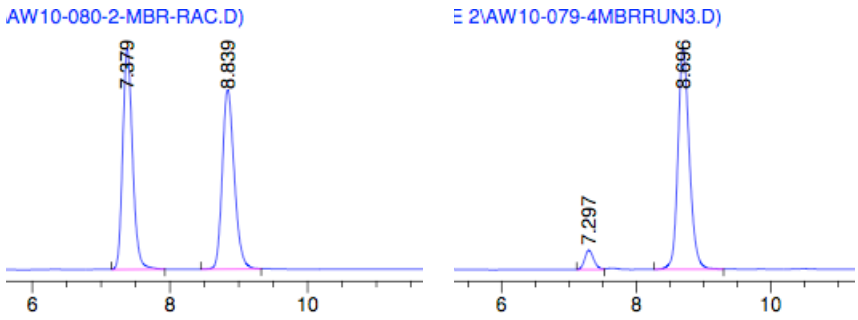
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.564	BB	0.6425	5983.66553	143.49626	50.0244
2	33.545	BB	0.6941	5977.81836	133.07689	49.9756

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.373	BB	0.6327	4961.60254	120.41743	96.2703
2	33.470	MM	0.6915	192.22284	4.63331	3.7297

3q





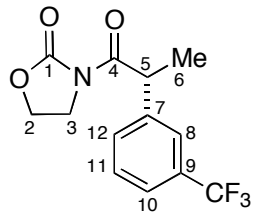
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

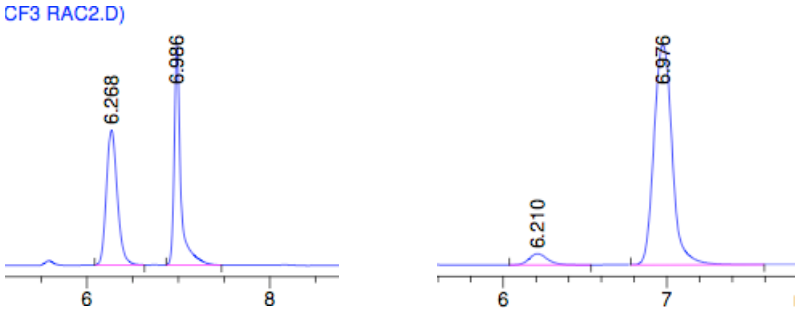
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.379	BB	0.1539	4057.13354	408.26282	50.1498
2	8.839	BB	0.1872	4032.90186	333.10431	49.8502

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.297	BV	0.1426	471.41653	50.65470	6.4745
2	8.696	BB	0.1796	6809.71045	577.51611	93.5255

3r





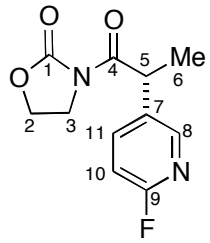
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

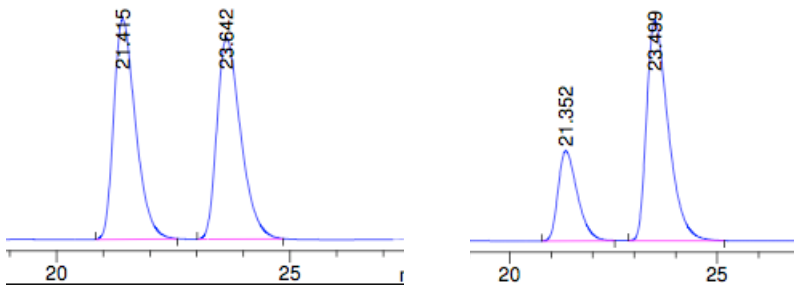
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.268	BB	0.1284	1198.70374	145.07213	50.4506
2	6.986	BB	0.0723	1177.29053	235.48329	49.5494

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.210	BB	0.1216	693.02399	88.26672	4.8930
2	6.976	VB	0.1225	1.34705e4	1736.08533	95.1070

3s





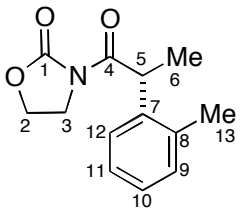
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

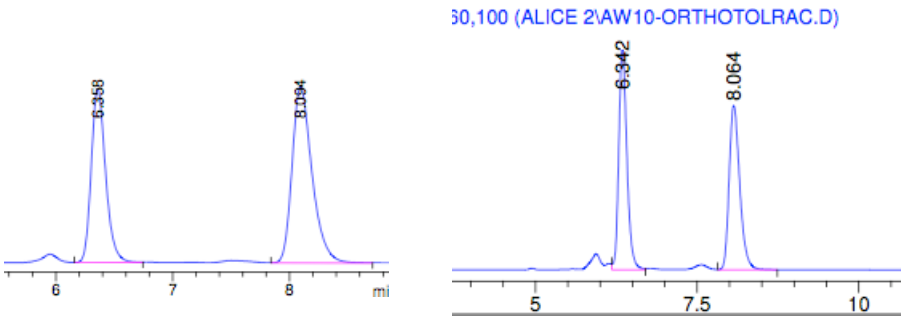
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.415	BB	0.4963	6439.51416	198.59731	50.0360
2	23.642	BB	0.5430	6430.25000	181.66278	49.9640

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.352	BB	0.4987	7894.69775	243.18896	26.7216
2	23.499	BB	0.5574	2.16496e4	593.78351	73.2784

3t





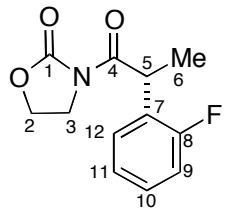
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

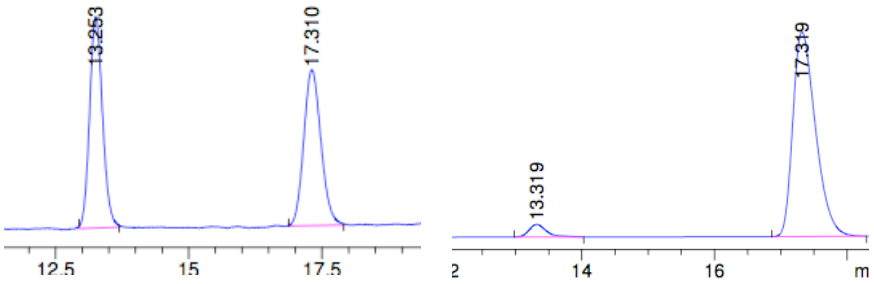
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.342	VV	0.1346	1.07779e4	1225.87476	50.7900
2	8.064	VB	0.1751	1.04426e4	915.53778	49.2100

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.358	VB	0.1298	4727.06494	552.62421	41.6102
2	8.094	VB	0.1805	6633.27588	558.92822	58.3898

3u





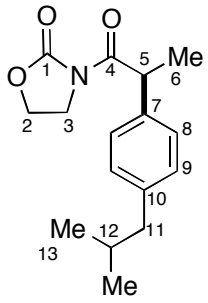
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

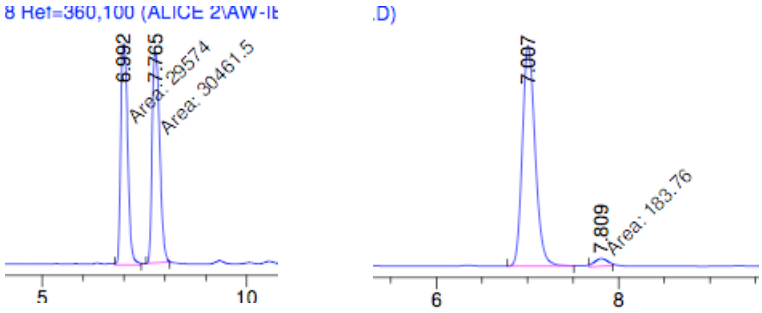
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.253	BB	0.2603	323.23883	19.21063	50.4416
2	17.310	BB	0.3478	317.57938	14.14985	49.5584

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.319	BB	0.2801	838.86084	45.72120	4.5553
2	17.319	BB	0.3665	1.75764e4	741.55920	95.4447

3v





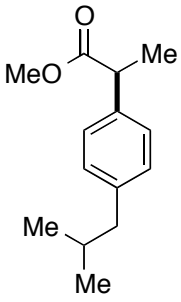
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

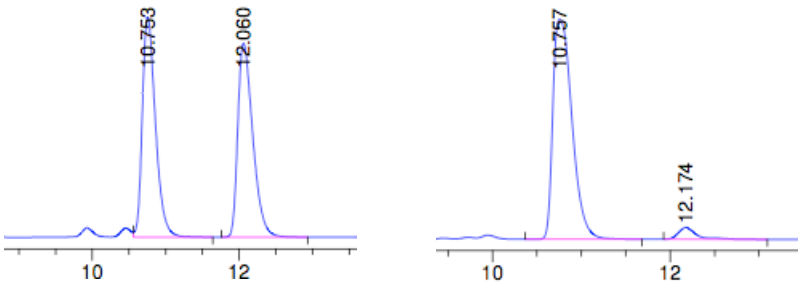
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.992	MM	0.1976	2.95740e4	2495.00049	49.2608
2	7.765	MM	0.2136	3.04615e4	2376.31860	50.7392

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.007	BB	0.1514	4982.27002	512.49017	96.4429
2	7.809	MM	0.1718	183.75990	17.82744	3.5571

Methyl 2-(4-isobutylphenyl)propanoate





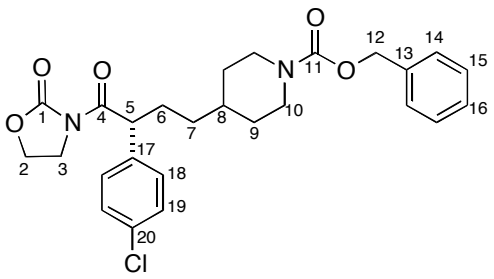
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

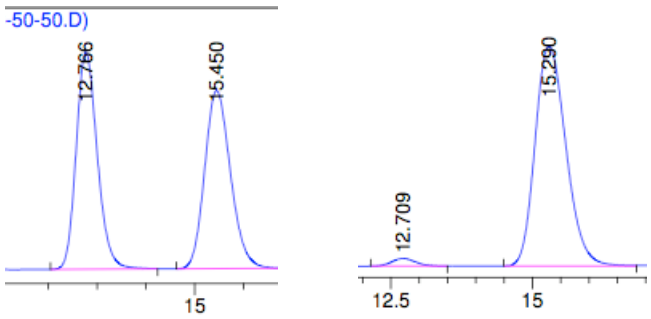
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.753	VB	0.1913	2.22729e4	1788.92078	50.0226
2	12.060	BB	0.2190	2.22528e4	1573.80701	49.9774

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.757	BB	0.2348	3.75048e4	2502.46997	95.1896
2	12.174	BB	0.2126	1895.28345	132.70978	4.8104

3w





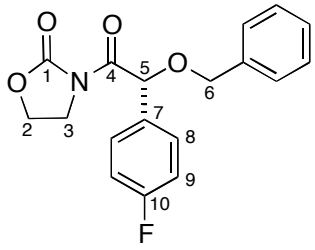
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

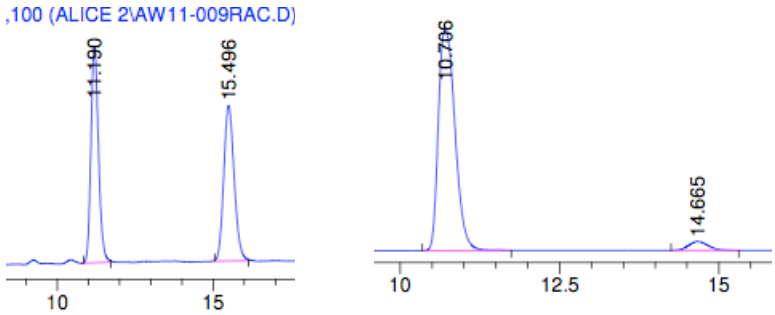
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.766	BB	0.4656	2.41283e4	800.68005	49.7114
2	15.450	BB	0.5727	2.42162e4	656.00055	49.8925
3	17.452	BB	0.4928	192.26001	5.28023	0.3961

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.709	BB	0.4537	1680.17859	57.36927	2.6456
2	15.290	BB	0.5945	6.18276e4	1630.22302	97.3544

3x





Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.190	BB	0.2604	1235.18347	73.36395	50.1033
2	15.496	BB	0.3591	1230.09119	52.93157	49.8967

Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.706	BB	0.2883	3.70591e4	2036.82849	95.4500
2	14.665	BB	0.3347	1766.58630	82.19959	4.5500