Electronic Supplementary Material (ESI) for Chemical Communications.

Supporting Information

Regioselective C(sp^3)-H Amidation of 8-methyl Quinolines with N-hydroxyphthalimides

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1. General consideration

Reagent Information

Unless otherwise stated, all reactions were carried out under inert atmosphere in glove box using screw cap reaction vials. All solvents were purchased from Aldrich and TCI and used as such. All chemicals were purchased from Sigma Aldrich, Alfa-aesar and TCI. For column chromatography, silica gel (230-400 mesh) from Merck was used. A gradient elution using *n*-hexane and ethyl acetate was performed based on Merck aluminium TLC sheets (silica gel $60F_{254}$).

Analytical Information

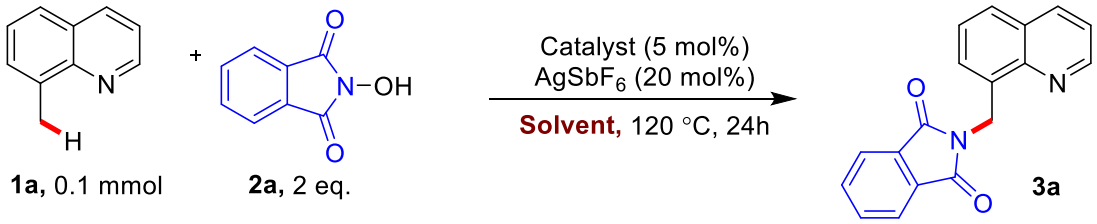
All isolated compounds are characterized by ^{1}H NMR, ^{13}C NMR, LC-MS and IR. In addition, all the compounds are further characterized by HRMS. Mass spectra were recorded on Water Q-ToF-Micromass and maXis Impact mass spectrometer**.** IR was analyzed by Shimadzu IR Prestige-21with ZnSe Single reflection ATR accessory. Nuclear magnetic resonance spectra were recorded either on a Bruker-Avance 600 or 300 MHz instrument. All ^{1}H NMR experiments are reported in units, parts per million (ppm) and were measured relative to the signals for residual chloroform (7.26) deuterated solvent. All $^{13}C\{1H\}$ NMR spectra were reported in ppm relative to deuterated chloroform (77.16), all were obtained with ^{1}H decoupling. Optimization studies were done by NMR and NMR yield were calculated by using TCE as internal standard.

2. General procedure for the preparation of starting materials

Compounds **1b** and **1c** were prepared according to the literature report.[1] Compounds **1d-f, 1l-p, 1t and 1u** are already known and prepared according to literature reports.[2] Compounds **1g-1i** were synthesized from the reported method.[3] Compound **1j** was synthesized using reported method,[4] compounds **1q, 1r, 1s** and **1v** were also prepared using reported method.[5] All other substituted 8-methyl quinolines were used from the commercially available sources. Compounds **4b, 4c, 4d** and **4e** were synthesized from the already known method.[6] Compound **6a** was synthesized according to the known literature report.[7] **1a-d3** was prepared by following the literature procedure.[8]

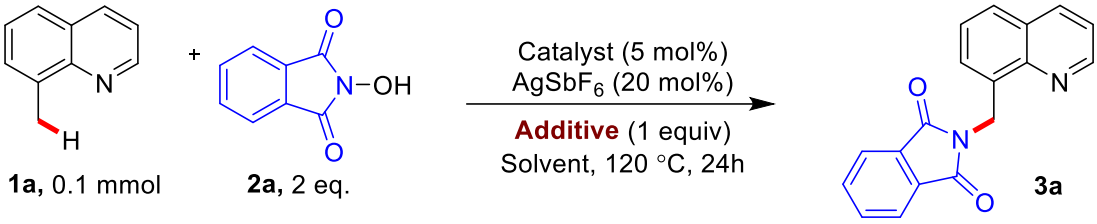
3. Optimization Details

*3.1. Catalyst screening (at 120 °C) (**Table S1**)*



S. No.	Catalyst (mol%)	Solvent (ml)	Temp (°C)	3a [NMR Yield (%)]
1	**$[RhCp^*Cl_2]_2$**	**HFIP**	**120**	**93**
2	$[RhCp^*Cl_2]_2$	DCE	120	61
3	$[CoCOCp^*I_2]$	HFIP	120	nr
4	$[RuCl_2(p\text{-cymene})]_2$	HFIP	120	nr
5	$[IrCp^*Cl_2]_2$	HFIP	120	nr
6	$[RhCp^*Cl_2]_2$	HFIP	rt	nr

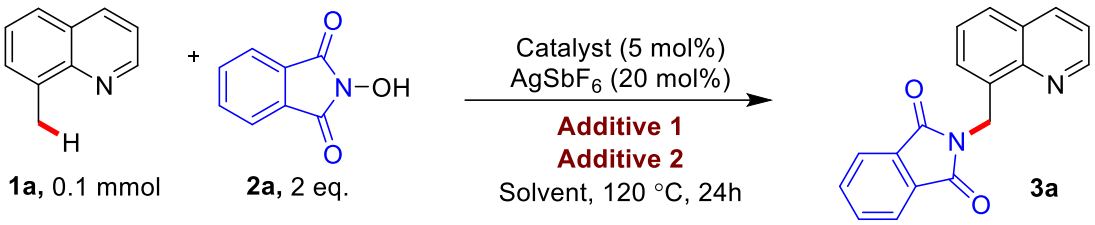
*3.2. Additive screening (at 120 °C) (**Table S2**)*



S. No.	Catalyst (mol%)	Additive (equiv.)	Solvent	Temp (°C)	3a [NMR Yield (%)]
1	$[RhCp^*Cl_2]_2$	PivOH	HFIP	120	66
2	$[RhCp^*Cl_2]_2$	NaOPiv	HFIP	120	55
3	$[RhCp^*Cl_2]_2$	AdCOOH	HFIP	120	42
4	**$[RhCp^*Cl_2]_2$**	**-**	**HFIP**	**120**	**93**
5	$[RhCp^*Cl_2]_2$	PivOH	DCE	120	<5

6	$[RhCp^*Cl_2]_2$	PivOH	TFE	120	30
7	$[CoCOCp^*I_2]$	PivOH	HFIP	120	nr
8	$[RuCl_2(p\text{-cymene})]_2$	PivOH	HFIP	120	nr
9	$[IrCp^*Cl_2]_2$	PivOH	HFIP	120	nr

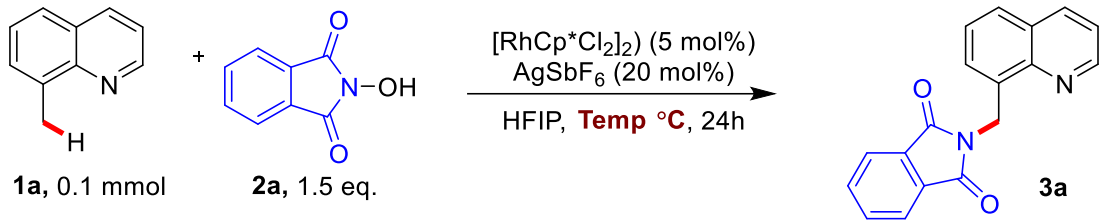
*3.3. Reaction sing cobalt catalyst (at 120 °C) (**Table S3**)*



S. No.	Catalyst (mol%)	Additive 1 (1 equiv.)	Additive 2 (1 equiv.)	Solvent (ml)	Temp (°C)	3a [NMR Yield (%)]
1	$[CoCOCp^*I_2]$	PivOH	-	HFIP	120	-
2	$[CoCOCp^*I_2]$	Sod. Benzoate	-	DCE	120	-
3	$[CoCOCp^*I_2]$	$[(CuOH)_2CO_3]$	-	DMF	120	-
4	$[CoCOCp^*I_2]$	Sod. Benzoate	Ag_2CO_3	HFIP	120	-
5	$[CoCOCp^*I_2]$	Ag_2CO_3	K_2CO_3	HFIP	120	-
6	$[Co(OAc)_2]$	Ag_2CO_3	K_2CO_3	$PhCF_3$	120	-
7	$[CoCOCp^*I_2]$	AdCOOH	-	HFIP	120	-
8	$[CoCOCp^*I_2]$	PivOH	NaOAc	HFIP	120	-

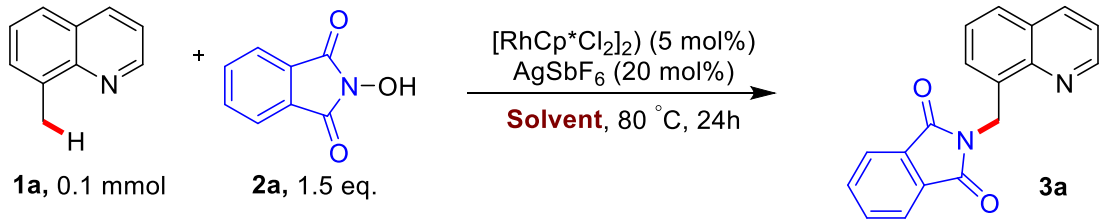
9	[CoCOCp*I_2]	PivOH	KOPiv	HFIP	120	-

*3.4. Temperature variation (**Table S4**)*



S. No.	Catalyst (mol%)	Temp (°C)	3a [NMR Yield (%)]
1	[RhCp*Cl_2]$_2$	120	93
2	[RhCp*Cl_2]$_2$	110	94
3	[RhCp*Cl_2]$_2$	100	94
4	**[RhCp*Cl_2]$_2$**	**80**	**97**
5	[RhCp*Cl_2]$_2$	60	20
6	[RhCp*Cl_2]$_2$)	rt	nr

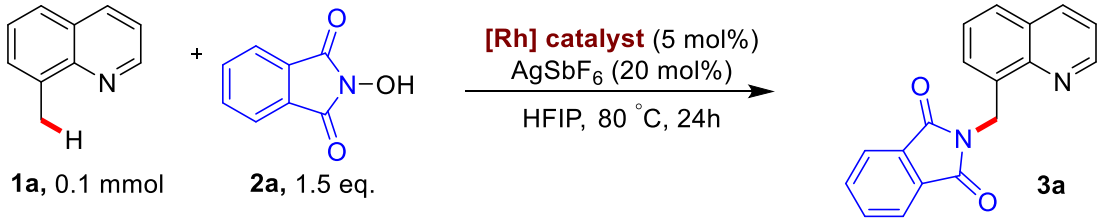
*3.5. Solvent screening(**Table S5**)*



S. No.	Catalyst (mol%)	Solvent	NMR Yield (%)
1	**[RhCp*Cl_2]$_2$**	**HFIP**	**97**

2	[RhCp*Cl$_2$]$_2$	DCE	34
3	[RhCp*Cl$_2$]$_2$	Trifluorotoluene	-
4	[RhCp*Cl$_2$]$_2$	Toluene	-
5	[RhCp*Cl$_2$]$_2$	TFE	68
6	[RhCp*Cl$_2$]$_2$)	Benzene	-

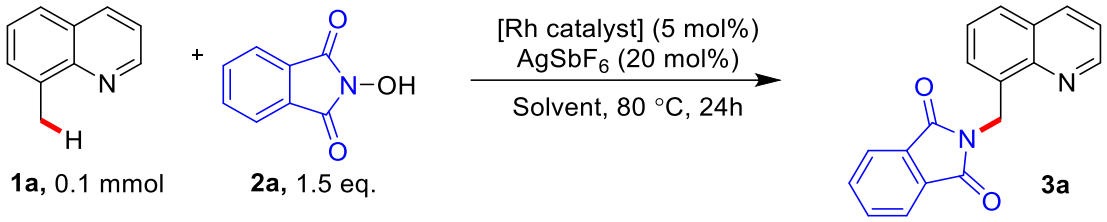
*3.6. Rhodium catalyst screening (**Table S6**)*



S. No.	Rh Catalyst (mol%)	Solvent	NMR Yield (%)
1	**[RhCp*Cl$_2$]$_2$**	**HFIP**	**97**
2	[RhCp*Cl$_2$]$_2$	HFIP	33*
3	Rh$_2$(OAc)$_4$	HFIP	-
4	Rh(Cl)$_3$	HFIP	-
5	RhCl(PPh$_3$)$_3$	HFIP	-

* AgBF$_4$ as silver salt

*Control experiments (**Table S7**)*



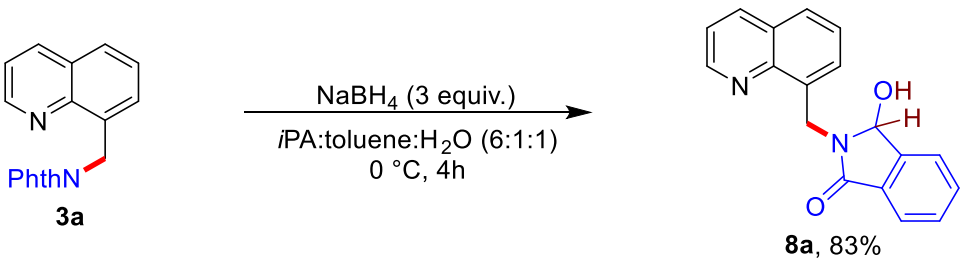
S. No.	Rh Catalyst (mol%)	Silver salt	Solvent	NMR Yield (%)
1	-	$AgSbF_6$	HFIP	-
2	$[RhCp^*Cl_2]_2$)	-	HFIP	94
3	-	-	HFIP	-
4	$[RhCp^*Cl_2]_2$)	$AgSbF_6$	-	-

4. General Procedure for Rh(III)-Catalyzed sp^3 C-H amidation of 8-methyl quinolines with *N*-hydroxyphthalimides.

To an oven-dried 15 mL Schlenk tube was added substituted 8-methyl quinoline, **1** (0.20 mmol), corresponding *N*-hydroxyphthalimide, **2a** (0.3 mmol, 1.5 equiv.), $[RhCp^*Cl_2]_2$ (6.1 mg, 5 mol%), $AgSbF_6$ (13.7 mg, 20 mol%), HFIP (1 mL, 0.2 M). The tube was then stirred for 24 h at 80 °C on a preheated IKA dry block, followed by cooling to room temperature. The resulting mixture was quenched by adding EtOAc (5 mL) and H_2O (5 mL). The organic layer was dried over Na_2SO_4 and solvent was evaporated under the reduced pressure. The remaining residue was purified by coloumn/flash chromatography using *n*-hexane/EtOAc as the eluent to afford the product **3.**

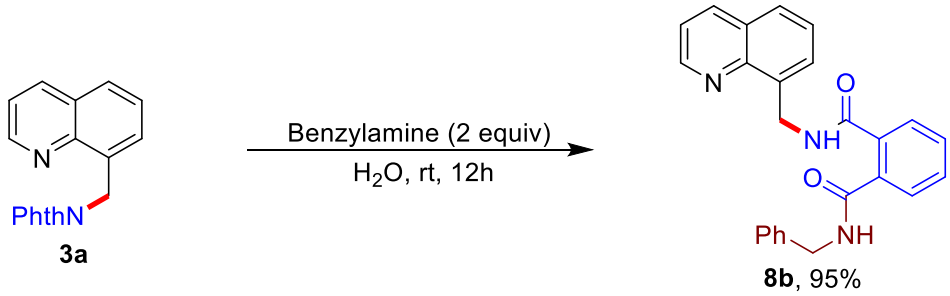
5. Post synthetic transformations

a) Reduction of product using $NaBH_4$[9]



To an oven-dried screw cap reaction vial charged with a spin vane magnetic stir-bar, 2-(quinolin-8-ylmethyl)isoindoline-1,3-dione (3a) (0.1 mmol), $NaBH_4$ (3.0 equiv.) and *i*-propanol:toluene:H2O (6:1:1, 0.5 mL) were added. The subsequent reaction mixture was stirred at 0 °C for 4 hours. After completion, the reaction mixture was concentrated under the reduced pressure and crude product was purified by flash chromatography on silica gel (230−400 mesh size) with *n*-hexane:EtOAc to afford the desired product.

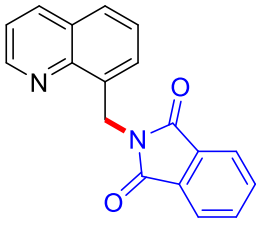
b) Ring opening reaction of 3a using benzylamine[10]



To an oven-dried screw cap reaction vial charged with a spin vane magnetic stir-bar, 2-(quinolin-8-ylmethyl)isoindoline-1,3-dione (3a) (0.1 mmol), benzylamine (2.0 equiv.) and H_2O (0.2 mL) were added. The subsequent reaction mixture was stirred at room temperature for 12 hours. After completion, the reaction mixture was extracted with ethyl acetate, and the organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel (230−400 mesh size) with n-hexane:EtOAc to afford the desired product.

6. Characterization Data

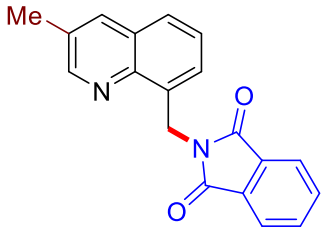
*2-[(quinolin-8-yl)methyl]-isoindoline-1,3-dione (Scheme 2, Entry **3a**)*



Following the general procedure for sp^3 C-H amidation, 8-methylquinoline (**1a**) (31.4 mg, 0.2 mmol), *N*-hydroxyphthalimide (**2a**) (28.6 mg, 0.3 mmol), [$RhCp^*Cl_2$]$_2$ (6.1 mg, 5 mol%), $AgSbF_6$ (13.7 mg, 20 mol%), HFIP (1 mL, 0.2 M) were used and reaction was run at 80 °C for 24 h. Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 97% (55.9 mg). Mp = 219-221 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.97 (dd, J = 4.2, 1.8 Hz, 1H), 8.15 (d, J = 8.4 Hz, 1H), 7.90 (dd, J = 5.4, 3.0 Hz, 2H), 7.75-7.72 (m, 3H), 7.45-7.42 (m, 3H), 5.65 (s, 2H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.5, 149.7, 146.0, 140.2, 136.3, 134.1, 132.4, 128.4, 127.5, 126.4, 126.2, 123.5, 121.4, 38.3 IR (ZnSe) ν_{max} (cm^{-1}): 3309, 3051, 2360, 2341, 1707, 1602, 1496, 1273, 1032, 939, 740. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{18}H_{13}N_2O_2^+$; 289.0972; found, 289.0972.

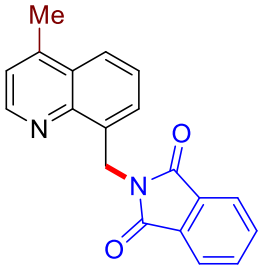
*2-[(3-methylquinolin-8-yl)methyl]-isoindoline-1,3-dione (Scheme 2, Entry **3b**)*

Following the general procedure for sp^3 C-H amidation, 3,8-dimethylquinoline (**1b**) (31.4 mg, 0.2 mmol), *N*-hydroxyphthalimide (**2a**) (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(6.**1 mg, 5 mol**%)**, $AgSbF_6$ **(13.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 h**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 91% (55.2 mg). Mp = 191-193 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.81 (s, 1H), 7.91-7.89 (m, 3H), 7.75-7.73 (m, 2H), 7.65 (d, J = 7.8 Hz, 1H), 7.39 (t, J = 7.8 Hz, 1H), 7.35 (d, J = 7.2 Hz, 1H), 5.63 (s, 2H), 2.52 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.5, 151.7, 144.3, 135.0, 134.1, 133.9, 132.4, 130.8, 128.3, 126.9, 126.3, 125.6, 123.5, 38.3, 18.8. IR (ZnSe) ν_{max} (cm^{-1}): 2245, 2094, 1697, 1489, 1392, 1180, 1103, 952, 709. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{15}N_2O_2^+$; 303.1128; found, 303.1128.

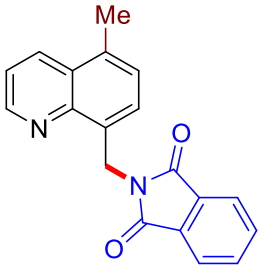


*2-[(4-methylquinolin-8-yl)methyl]-isoindoline-1,3-dione (Scheme 2, Entry **3c**)*

Following the general procedure for sp^3 C-H amidation, 4,8-dimethylquinoline (**1c**) (31.4 mg, 0.2 mmol), *N*-hydroxyphthalimide (**2a**) (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(6.**1 mg, 5 mol**%)**, $AgSbF_6$ **(13.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 h**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 87% (52.7 mg). Mp = 195-197 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.82 (d, J = 4.2 Hz, 1H), 7.93-7.89 (m, 2H), 7.75-7.74 (m, 2H), 7.47-7.44 (m, 1H), 7.41 (d, J = 7.2 Hz, 1H), 7.28 (d, J = 3.6 Hz, 1H), 5.65 (s, 2H), 2.71 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.5, 149.3, 142.9, 134.5, 134.1, 132.5, 128.4, 126.2, 125.9, 123.7, 123.55, 123.50, 122.3, 38.6, 19.1. IR (ZnSe) ν_{max} (cm^{-1}): 2237, 1921, 1701, 1384, 1097, 952, 844, 721, 613. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{15}N_2O_2^+$; 303.1128; found, 303.1129.



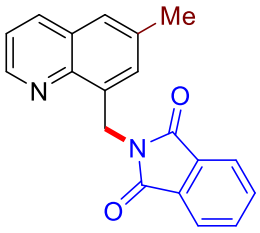
*2-[(5-methylquinolin-8-yl)methyl]-isoindoline-1,3-dione (Scheme 2, Entry **3d**)*



Following the general procedure for sp^3 C-H amidation, 5,8-dimethylquinoline (**1d**) (31.4 mg, 0.2 mmol), *N*-hydroxyphthalimide (**2a**) (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(6.**1 mg, 5 mol**%)**, $AgSbF_6$ **(13.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 h**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 98% (59.3 mg). Mp = 127 - 129 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.98 (dd, J = 4.2, 1.2 Hz, 1H), 8.33 (d, J = 8.4 Hz, 1H), 7.89 (dd, J = 5.4, 3.0 Hz, 2H), 7.74 (dd, J = 5.4, 3.0 Hz, 2H), 7.47 (dd, J = 9.0, 4.2 Hz, 1H), 7.32 (d, J = 7.2 Hz, 1H), 7.26 (d, J = 7.2 Hz, 1H), 5.61 (s, 2H), 2.64 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.5, 149.2, 146.1, 134.3, 134.1, 132.9, 132.4, 132.0, 127.8, 126.7, 126.4, 123.5, 121.0, 38.4, 18.7. IR (ZnSe) ν_{max} (cm^{-1}): 3315, 3039, 2360, 2341, 1766, 1702, 1600, 1103, 746. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{15}N_2O_2^+$; 303.1128; found, 303.1147.

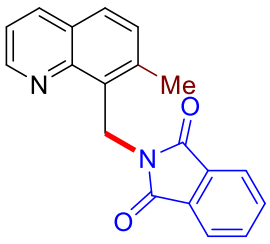
*2-((6-methylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3e**)*

Following the general procedure for sp^3 C-H amidation, 6,8-dimethylquinoline **(1e)** (31.4 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(6.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 95% (57.4 mg). Mp = 195 - 197 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.89-8.88 (m, 1H), 8.05 (d, J = 8.4, 1H), 7.92-7.90 (m, 2H), 7.76-7.74 (m, 2H), 7.48 (s, 1H), 7.39 (dd, J = 8.4, 4.2, 1H), 7.22 (s, 1H), 5.61 (s, 2H), 2.43 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.5, 148.8, 144.7, 136.1, 135.6, 134.1, 133.7, 132.4, 128.6, 128.5, 126.3, 123.6, 121.4, 38.2, 21.9. IR (ZnSe) νmax (cm^{-1}): 2360, 2341, 1770, 1653, 1506, 1309, 1103, 950, 719. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{15}N_2O_2^+$; 303.1128; found, 303.1144.



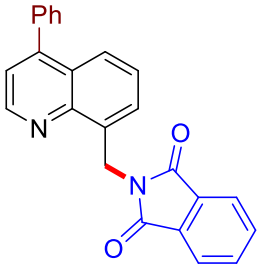
*2-((7-methylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3f**)*

Following the general procedure for sp^3 C-H amidation, 7,8-dimethylquinoline **(1f)** (31.4 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(6.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 91% (55.1 mg). Mp = 146 - 148 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.85 (dd, J = 4.2, 1.8 Hz, 1H), 8.06 (dd, J = 8.4, 1.8 Hz, 1H), 7.76 (dd, J = 5.4, 3.0 Hz, 2H), 7.67 (d, J = 8.4 Hz, 1H), 7.64 (dd, J = 5.4, 3.0 Hz, 2H), 7.37 (d, J = 8.4 Hz, 1H), 7.30 (dd, J = 8.4, 4.2 Hz, 1H), 5.60 (s, 2H), 2.67 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.3, 149.6, 147.3, 139.2, 136.1, 133.8, 132.2, 130.8, 129.9, 127.5, 126.6, 123.1, 120.3, 35.6, 20.6. IR (ZnSe) νmax (cm^{-1}): 2962, 2360, 2341, 1770, 1716,. 1652, 1388, 1047, 710. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{15}N_2O_2^+$; 303.1128; found, 303.1147.



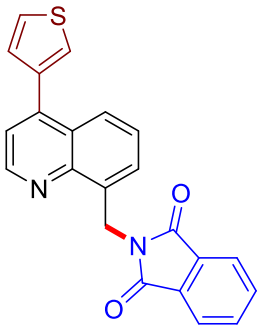
*2-((4-phenylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3g**)*

Following the general procedure for sp^3 C-H amidation, 4-phenyl-8-methylquinoline **(1g)** (43.8 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(6.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 94% (68.6 mg). Mp = 175 - 177 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.99 (d, J = 4.2 Hz, 1H), 7.91 (dd, J = 5.4, 3.0 Hz, 2H), 7.83 (d, J = 7.8 Hz, 1H), 7.75 (dd, J = 5.4, 3.0 Hz, 2H), 7.53-7.51 (m, 5H), 7.44-7.43 (m, 1H), 7.40-7.37 (m, 2H), 5.70 (s, 2H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.5, 149.2, 148.8, 146.4, 138.3, 134.4, 134.1, 132.4, 129.7, 128.7, 128.5, 126.9, 126.3, 126.2, 125.6, 123.5, 121.7, 38.6. IR (ZnSe) νmax (cm^{-1}): 2360, 2341, 1770, 1706, 1652, 1463, 1386, 1114, 696. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{24}H_{17}N_2O_2^+$; 365.1285; found, 365.1304.



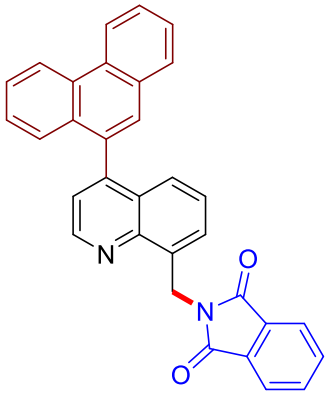
*2-((4-(thiophen-3-yl)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3h**)*

Following the general procedure for sp^3 C-H amidation, 8-methyl-4-(thiophen-3-yl)quinoline **(1h)** (44.8 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**RhCp*Cl_2**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 91% (67.5 mg). Mp = 162 - 164 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.96 (d, J = 4.2 Hz, 1H), 7.98 (d, J = 7.8 Hz, 1H), 7.90 (dd, J = 5.4, 3.0 Hz, 2H), 7.76 - 7.73 (m, 2H), 7.50-7.49 (m, 2H), 7.44-7.40 (m, 3H), 7.31-7.30 (m, 1H), 5.68 (s, 2H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.5, 149.2, 146.4, 143.5, 138.7, 134.4, 134.1, 132.4, 129.0, 126.9, 126.4, 126.35, 126.28, 125.4, 125.1, 123.5, 121.4, 38.6. IR (ZnSe) νmax (cm^{-1}): 2920, 2360, 2341, 1766, 1707, 1506, 1386, 1114, 952, 712. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{22}H_{15}N_2O_2S^+$; 371.0849; found, 371.0861.



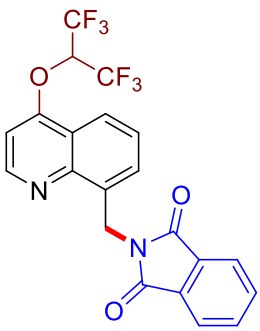
*2-((4-(phenanthren-9-yl)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3i**)*

Following the general procedure for sp^3 C-H amidation, 8-methyl-4-(phenanthren-9-yl)quinoline **(1h)** (63.8 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**RhCp*Cl_2**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 89% (82.8 mg). Mp = 246 - 284 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 9.11 (d, J = 4.2 Hz, 1H), 8.80 (dd, J = 15.0, 8.4 Hz, 2H), 7.94-7.91 (m, 3H), 7.77-7.73 (m, 4H), 7.69-7.65 (m, 2H), 7.54 (d, J = 4.2 Hz, 1H), 7.44-7.41 (m, 3H), 7.36 (d, J = 8.4 Hz, 1H), 7.25-7.23 (m, 1H), 5.77 (s, 2H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.6, 149.3, 134.6, 134.5, 134.3, 134.2, 132.5, 132.3, 131.3, 131.2, 130.6, 130.4, 129.9, 129.0, 128.4, 128.3, 127.4, 127.3, 127.2, 127.0, 126.6, 126.4, 126.2, 123.6, 123.1, 122.9, 122.8, 38.6. IR (ZnSe) νmax (cm^{-1}): 3290, 3059, 2233, 1951, 1712, 1388, 1095, 729, 516. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{32}H_{21}N_2O_2^+$; 465.1598; found, 465.1598.



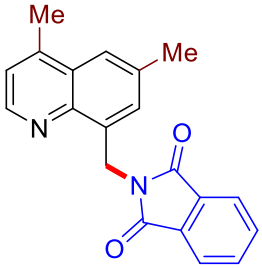
*2-((4-((1,1,1,3,3,3-hexafluoropropan-2-yl)oxy)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3j**)*

Following the general procedure for sp^3 C-H amidation, 4-((1,1,1,3,3,3-hexafluoropropan-2-yl)oxy)-8-methylquinoline **(1j)** (61.8 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**RhCp*Cl_2**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 93% (82.8 mg). Mp = 182 - 184 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.89 (d, J = 5.4 Hz, 1H), 8.15 (d, J = 7.2 Hz, 1H), 7.91-7.89 (m, 2H), 7.76-7.74 (m, 2H), 7.530-7.49 (m, 2H), 6.93 (d, J = 4.8 Hz, 1H), 5.62 (s, 2H), 5.27-5.21 (m, 1H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.4, 159.9, 150.0, 147.6,



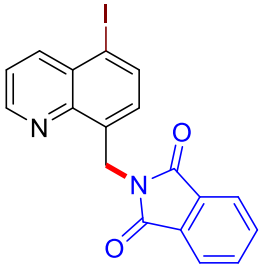
134.21, 134.17, 132.4, 131.6, 128.1, 126.7, 123.6, 121.02, 121.00, 120.9 (q, $J_{C\text{-}F}$ = 282 Hz), 102.4, 74.25 (sept, $J_{C\text{-}F}$ = 33.7 Hz), 38.4. ^{19}F NMR (565 MHz, $CDCl_3$, δ): -73.11. IR (ZnSe) νmax (cm^{-1}):2121, 2063, 1716, 1388, 1222, 1126, 1074, 960, 713. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{21}H_{13}F_6N_2O_2^+$; 455.0825; found, 455.0825.

*2-((4,6-dimethylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3k**)*



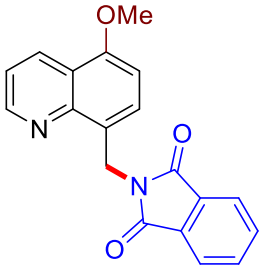
Following the general procedure for sp^3 C-H amidation, 4,6,8-trimethylquinoline **(1k)** (34.2 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 93% (58.8 mg). Mp = 222 - 224 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.74 (d, J = 4.2 Hz, 1H), 7.91 (dd, J = 5.4, 3.0 Hz, 2H), 7.75 (dd, J = 5.4, 3.0 Hz, 2H), 7.66 (s, 1H), 7.22 (d, J = 4.8 Hz, 1H), 7.20 (s, 1H), 5.61 (s, 2H), 2.66 (s, 3H), 2.45 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.5, 148.4, 144.2, 143.8, 135.7, 134.1, 134.09, 132.4, 128.5, 128.1, 123.5, 122.4, 122.2, 38.5, 22.2, 19.1. IR (ZnSe) νmax (cm^{-1}): 3311. 3050, 2360, 2341, 1706, 1602, 1494, 1272, 1153, 740. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{20}H_{17}N_2O_2^+$; 317.1285; found, 317.1285.

*2-((5-iodoquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3l**)*



Following the general procedure for sp^3 C-H amidation, 5-iodo-8-methylquinoline **(1l)** (53.8 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 94% (77.8 mg). Mp = 185 - 187 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.92 (dd, J = 4.2, 1.8 Hz, 1H), 8.38 (dd, J = 8.4, 1.2 Hz, 1H), 8.00 (d, J = 7.8 Hz, 1H), 7.90 (dd, J = 5.4, 3.0 Hz, 2H), 7.75 (dd, J = 5.4, 3.0 Hz, 2H), 7.51 (dd, J = 8.4, 4.2 Hz, 1H), 7.17 (d, J = 7.2 Hz, 1H), 5.59 (s, 2H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.4, 150.5, 146.4, 140.6, 137.4, 135.5, 134.2, 132.3, 130.1, 127.8, 123.6, 123.1, 97.8, 38.1. IR (ZnSe) νmax (cm^{-1}): 3311, 2360, 2341, 1604, 1486, 1270, 1153, 941, 739, 686. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{18}H_{12}IN_2O_2^+$; 414.9938; found, 414.9956.

*2-((5-methoxyquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3m**)*

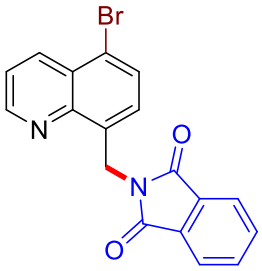


Following the general procedure for sp^3 C-H amidation, 5-methoxy-8-methylquinoline **(1m)** (34.6 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 97% (61.7 mg). Mp = 140 - 142 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.96 (dd, J = 4.2, 1.8 Hz, 1H), 8.57 (dd, J = 8.4, 1.2 Hz, 1H), 7.88 (dd, J = 5.4, 3.0 Hz, 2H), 7.73 (dd, J = 5.4, 3.0 Hz, 2H), 7.42-

7.39 (m, 2H), 6.75 (d, J = 7.8 Hz, 1H), 5.53 (s, 2H), 3.96 (s, 3H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.6, 154.9, 150.0, 134.5, 134.0, 132.4, 131.2, 127.3, 125.8, 123.7, 123.5, 121.0, 120.5, 103.8, 55.8, 38.1. IR (ZnSe) νmax (cm^{-1}): 2925, 2358, 2343, 1707, 1591, 1386, 1186, 954, 777. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{15}N_2O_2^+$; 319.1077; found, 319.1097.

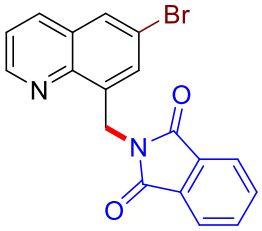
*2-((5-bromoquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3n**)*

Following the general procedure for sp^3 C-H amidation, 5-bromo-8-methylquinoline **(1n)** (44.2 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2]_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 96% (69.8 mg). Mp = 106 - 108 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.97 (dd, J = 4.2, 1.8 Hz, 1H), 8.53 (dd, J = 8.4, 1.2 Hz, 1H), 7.89 (dd, J = 4.8, 3.6 Hz, 2H), 7.75-7.73 (m, 2H), 7.71 (d, J = 7.8 Hz, 1H), 7.53 (dd, J = 9.0, 4.2 Hz, 1H), 7.31 (d, J = 7.8 Hz, 1H), 5.58 (s, 1H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.4, 150.3, 146.6, 135.8, 134.4, 134.2, 132.3, 129.9, 127.7, 127.1, 123.6, 122.5, 121.4, 38.1. IR (ZnSe) νmax (cm^{-1}): 2921, 2360, 2341, 1766, 1706, 1471, 1386, 1336, 1107, 902, 709. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{18}H_{12}BrN_2O_2^+$; 367.0077; found, 367.0093.



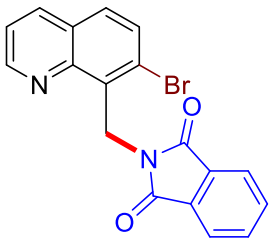
*2-((6-bromoquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3o**)*

Following the general procedure for sp^3 C-H amidation, 6-bromo-8-methylquinoline **(1o)** (44.2 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2]_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 99% (72.7 mg). Mp = 195 - 197 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.96 (dd, J = 4.2, 1.8 Hz, 1H), 8.06 (dd, J = 8.4, 1.8 Hz, 1H), 7.92 (dd, J = 5.4, 3.0 Hz, 2H), 7.90 (d, J = 1.8 Hz, 1H), 7.77 (dd, J = 5.4, 3.0 Hz, 2H), 7.47-7.45 (m, 2H), 5.61 (s, 2H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.3, 150.0, 144.7, 136.6, 135.4, 134.3, 132.3, 129.9, 129.53, 129.51, 123.7, 122.3, 120.3, 37.8. IR (ZnSe) νmax (cm^{-1}): 2360, 2355, 1763, 1703, 1652, 1388, 1105, 949, 870, 717. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{18}H_{12}BrN_2O_2^+$; 367.0077; found, 367.0089.



*2-((7-bromoquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3p**)*

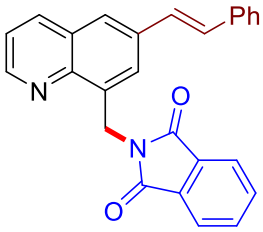
Following the general procedure for sp^3 C-H amidation, 7-bromo-8-methylquinoline **(1p)** (44.2 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2]_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 93% (68.3 mg). Mp = 144 - 146 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.87 (dd, J = 4.2, 1.2 Hz, 1H), 8.10 (dd, J = 7.8, 1.8 Hz, 1H), 7.76 (dd, J = 5.4, 3.6 Hz, 2H), 7.69 (d, J = 9.0 Hz, 1H), 7.65 (dd, J = 5.4, 3.0 Hz, 2H), 7.63 (d, J = 8.4 Hz, 1H), 7.39 (dd, J = 8.4, 4.2 Hz, 1H), 5.71 (s,



2H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.1, 150.4, 147.7, 136.4, 133.8, 133.0, 132.2, 131.2, 128.9, 127.2, 126.4, 123.2, 121.5, 38.8. IR (ZnSe) νmax (cm^{-1}): 3041, 2360, 2341, 1770, 1707, 1653, 1330, 1118, 947, 833, 713. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{18}H_{12}BrN_2O_2^+$; 367.0077; found, 367.0081.

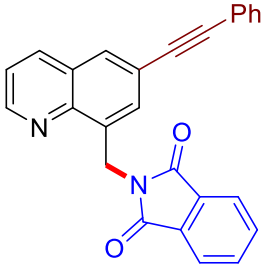
*(E)-2-((6-styrylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3q**)*

Following the general procedure for sp^3 C-H amidation, 4-phenyl-8-methylquinoline **(1q)** (49 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 97% (75.7 mg). Mp = 207 - 209 °C. ^1H NMR (600 MHz, $CDCl_3$, δ): 8.91-8.89 (m, 1H), 8.13-8.11 (m, 1H), 7.93 (dd, J = 5.4, 3.0 Hz, 2H), 7.77-7.75 (m, 3H), 7.65 (s, 1H), 7.49-7.48 (m, 2H), 7.42-7.40 (m, 1H), 7.35-7.32 (m, 2H), 7.27-7.24 (m, 1H), 7.18-7.10 (m, 2H), 5.65 (s, 2H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.5, 149.5, 145.8, 137.0, 136.2, 135.2, 134.5, 134.2, 132.4, 130.3, 128.8, 128.7, 128.1, 128.0, 126.7, 125.34, 125.32, 125.1, 123.6, 121.8, 38.2. IR (ZnSe) νmax (cm^{-1}): 3028, 2360, 2341, 1766, 1703, 1653, 1494, 1388, 1107, 944, 731. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{26}H_{19}N_2O_2^+$; 391.1441; found, 391.1457.



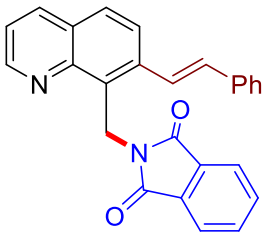
*2-((6-(phenylethynyl)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3r**)*

Following the general procedure for sp^3 C-H amidation, 8-methyl-6-(phenylethynyl)quinoline **(1r)** (48.6 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 97% (75.3 mg). Mp = 128 - 130 °C. ^1H NMR (600 MHz, $CDCl_3$, δ): 8.96 (dd, J = 4.2, 1.8 Hz, 1H), 8.13 (dd, J = 8.4, 1.8 Hz, 1H), 7.93 (dd, J = 5.4, 3.0 Hz, 3H), 7.76 (dd, J = 5.4, 3.0 Hz, 3H), 7.52-7.50 (m, 3H), 7.46 (dd, J = 8.4, 4.2 Hz, 1H), 7.33-7.32 (m, 3H), 5.63 (s, 2H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.5, 150.3, 145.4, 136.1, 134.6, 134.2, 132.4, 131.8, 130.9, 128.9, 128.7, 128.5, 128.2, 123.71, 123.67, 122.9, 122.1, 121.4, 90.7, 89.1, 38.0. IR (ZnSe) νmax (cm^{-1}): 2931, 2360, 2341, 1707, 1388, 1317, 1110, 953, 881, 719. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{26}H_{17}N_2O_2^+$; 389.1285; found, 389.1306.



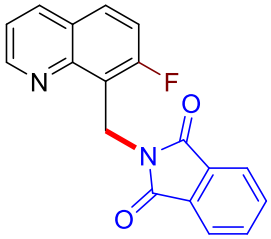
*(E)-2-((7-styrylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3s**)*

Following the general procedure for sp^3 C-H amidation, (E)-8-methyl-7-styrylquinoline **(1s)** (49 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 93% (72.7 mg). Mp = 168 - 170 °C. ^1H NMR (600 MHz, $CDCl_3$, δ): 8.96 (dd, J = 4.2, 1.2 Hz, 1H), 8.11 (dd, J = 8.4, 1.8



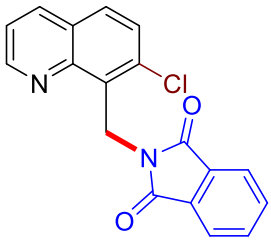
Hz, 1H), 7.81 (d, J = 8.4 Hz, 1H), 7.78-7.7 (m, 2H), 7.68 (dd, J = 5.4, 3.0 Hz, 2H), 7.57 (dd, J = 5.4, 3.0 Hz, 2H), 7.557.54 (m, 2H), 7.39-7.35 (m, 3H), 7.28-7.25 (m, 1H), 7.04 (d, J = 16.2 Hz, 1H), 5.87 (s, 2H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.3, 149.9, 147.6, 137.8, 137.0, 136.1, 134.4, 133.8, 132.8, 132.1, 130.9, 128.8, 128.1, 128.0, 127.5, 127.1, 126.1, 124.9, 123.7, 123.1, 121.0, 34.6. IR (ZnSe) νmax (cm^{-1}): 3311, 3049, 2360, 2341, 1602, 1496, 1273, 939, 739, 688. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{26}H_{18}N_2O_2^+$; 391.1441; found, 391.1461.

*2-((7-fluoroquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3t**)*



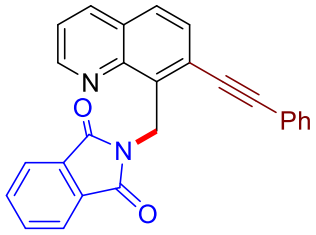
Following the general procedure for sp^3 C-H amidation, 7-fluoro-8-methylquinoline **(1t)** (32.2 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 94% (57.6 mg). Mp = 156 - 158 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.90 (dd, J = 4.2, 1.8 Hz, 1H), 8.10 (dd, J = 8.4, 1.8 Hz, 1H), 7.81-7.79 (m, 2H), 7.75 (dd, J = 9.0, 6.0 Hz, 1H), 7.66 (dd, J = 5.4, 3.0 Hz, 1H), 7.35 (dd, J = 8.4, 4.2 Hz, 1H), 7.31-7.28 (m, 1H), 5.59 (s, 2H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.1, 161.4 (d, J_{C-F} = 250.5 Hz), 150.8, 147.4 (d, J_{C-F} = 9.0 Hz), 136.2, 133.8, 133.3, 129.5 (d, J_{C-F} = 11.2 Hz), 125.2, 123.3, (d, J_{C-F} = 3.0 Hz), 118.3 (d, J_{C-F} = 12.0 Hz), 117.0 (d, J_{C-F} = 27.0 Hz), 32.3 (d, J_{C-F} = 3.0 Hz). ^{19}F NMR (565 MHz, $CDCl_3$, δ): -111.52. IR (ZnSe) νmax (cm^{-1}): 3311, 3028, 2360, 2343, 1770, 1703, 1602, 1496, 1249, 1113, 939, 688. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{18}H_{12}FN_2O_2^+$; 307.0877; found, 307.0888.

*2-((7-chloroquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3u**)*

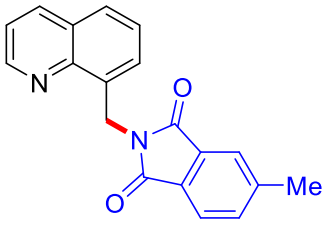


Following the general procedure for sp^3 C-H amidation, 7-chloro-8-methylquinoline **(1u)** (35.6 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 96% (62 mg). Mp = 138 - 140 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.90 (s, 1H), 8.11 (d, J = 5.4 Hz, 1H), 7.76-7.65 (m, 5H), 7.51 (d, J = 8.4 Hz, 1H), 7.39-7.37 (m, 1H), 5.70 (s, 2H). $^{13}C\{^{1}H\}$ NMR (150 MHz, $CDCl_3$, δ): 168.1, 150.5, 147.5, 136.4, 136.0, 133.8, 132.2, 131.0, 128.8, 128.3, 126.8, 123.2, 121.3, 36.0. IR (ZnSe) νmax (cm^{-1}): 3051, 2360, 2341, 1772, 1710, 1489, 1392, 1334, 1105, 952, 711. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{18}H_{12}ClN_2O_2^+$; 323.0582; found, 323.0589.

*2-((7-(phenylethynyl)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3v**)*

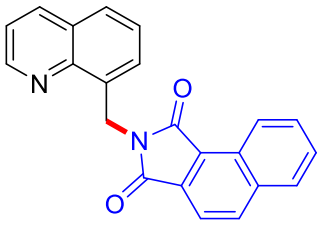
Following the general procedure for sp^3 C-H amidation, 8-methyl-7-(phenylethynyl)quinoline **(1v)**  (48.6 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (25% EtOAc/*n*-hexane) as white solid, yield = 90% (69.8 mg). Mp = 142 - 144 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.87 (dd, J = 4.2, 1.8 Hz, 1H), 8.10 (dd, J = 8.4, 1.8 Hz, 1H), 7.77-7.75 (m, 2H), 7.69 (d, J = 9.0 Hz, 1H), 7.66-7.64 (m, 2H), 7.63 (d, J = 9.0 Hz, 1H), 7.39 (d, J = 7.8, 1.8 Hz, 1H), 5.71 (s, 2H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.1, 150.4, 147.7, 136.4, 133.8, 133.0, 132.2, 131.1, 128.9, 127.2, 126.4, 123.2, 121.5, 38.8. IR (ZnSe) νmax (cm^{-1}): 2358, 2353, 1770, 1709, 1652, 1435, 1390, 1122, 947, 713. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{26}H_{17}N_2O_2^+$; 389.1285; found, 389.1283.

*5-methyl-2-(quinolin-8-ylmethyl)isoindoline-1,3-dione (Scheme 2, Entry **3w**)*



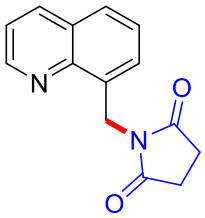
Following the general procedure for sp^3 C-H amidation, 8-methylquinoline **(1a)** (28.6 mg, 0.2 mmol), 2-hydroxy-5-methylisoindoline-1,3-dione **(2b)** (53.2 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 87% (52.7 mg). Mp = 208 - 210 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.95 (dd, J = 4.2, 1.8 Hz, 1H), 8.14 (dd, J = 8.4, 1.8 Hz, 1H), 7.77 (d, J = 7.8 Hz, 1H), 7.71 (d, J = 7.2 Hz, 1H), 7.69 (s, 1H), 7.52 (d, J = 7.8 Hz, 1H), 7.43-7.39 (m, 3H), 5.63 (s, 2H), 2.51 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.7, 168.6, 149.7, 146.0, 145.4, 136.3, 134.6, 134.3, 132.8, 129.8, 128.3, 127.4, 126.3, 126.2, 124.1, 123.4, 121.4, 38.2, 22.1. IR (ZnSe) νmax (cm^{-1}): 3278, 1337, 2148, 1955, 1708, 1411, 1226, 918, 721. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{15}N_2O_2^+$; 303.1128; found, 303.1128.

*2-(quinolin-8-ylmethyl)-1H-benzo[e]isoindole-1,3(2H)-dione (Scheme 2, Entry **3x**)*



Following the general procedure for sp^3 C-H amidation, 8-methylquinoline **(1a)** (28.6 mg, 0.2 mmol), 2-hydroxy-1H-benzo[e]isoindole-1,3(2H)-dione **(2c)** (63.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 84% (57 mg). Mp = 198 - 200 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.99 - 8.98 (m, 1H), 8.97 (d, J = 8.4 Hz, 1H), 8.20 - 8.17 (m, 2H), 7.97 (d, J = 8.4 Hz, 1H), 7.92 (d, J = 7.8 Hz, 1H), 7.75 - 7.71 (m, 2H), 7.66 (t, J = 7.8 Hz, 1H), 7.52 (d, J = 7.2 Hz, 1H), 7.46 - 7.44 (m, 2H), 5.71 (s, 2H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 169.6, 169.0, 149.5, 136.7, 136.4, 135.0, 134.3, 131.5, 129.5, 128.8, 128.7, 128.3, 128.1, 127.6, 127.4, 126.6, 126.3, 125.1, 121.3, 118.6, 38.0. IR (ZnSe) νmax (cm^{-1}):3133, 2198, 1994, 1693, 1388, 1103, 948, 779. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{22}H_{15}N_2O_2^+$; 339.1128; found, 339.1127.

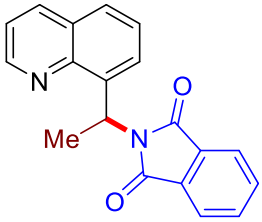
*1-(quinolin-8-ylmethyl)pyrrolidine-2,5-dione (Scheme 3, Entry **3y**)*



Following the general procedure for sp^3 C-H amidation, 8-methylquinoline **(1a)** (28.6 mg, 0.2 mmol), n-hydroxysuccinimide **(2d)** (34.5 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 80 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 58% (28 mg). Mp = 212 - 214 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.95 (d, J = 4.2 Hz, 1H), 8.15 (d, J = 7.8 Hz, 1H), 7.74 (d, J = 8.4 Hz, 1H), 7.46 (t, J = 7.8 Hz, 1H), 7.43 (dd, J = 8.4, 4.2 Hz, 1H), 7.37 (d, J = 7.2 Hz, 1H), 5.46 (s, 2H), 2.84 (s, 4H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 177.3, 149.8, 146.1, 136.3, 133.4, 130.7, 128.4, 127.7, 126.7, 126.2, 121.4, 39.1, 28.5. IR (ZnSe) νmax (cm^{-1}): 3251, 2137, 1705, 1377, 1083, 948, 717. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{14}H_{13}N_2O_2^+$; 241.0972; found, 241.0971.

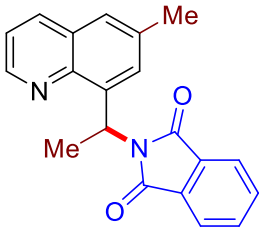
*2-(1-(quinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 3, Entry **5a**)*

Following the general procedure for sp^3 C-H amidation, 8-ethylquinoline **(4a)** (31.4 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 77% (46.6 mg). Mp = 122 - 124 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.88 (dd, J = 4.2, 1.8 Hz, 1H), 8.10 (dd, J = 8.4, 1.8 Hz, 1H), 8.01 (d, J = 7.2 Hz, 1H), 7.78 (dd, J = 5.4, 3.6 Hz, 2H), 7.75-7.74 (m, 1H), 7.65 (dd, J = 6.0, 3.0 Hz, 2H), 7.54 (t, J = 7.8 Hz, 1H), 7.34 (dd, J = 7.8, 4.2 Hz, 1H), 6.82 (dd, J = 14.4, 7.2 Hz, 1H), 2.05 (d, J = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.6, 149.8, 145.8, 138.3, 136.3, 134.4, 133.8, 132.8, 132.2, 128.2, 128.1, 127.8, 126.1, 123.7, 123.1, 121.1, 45.5, 18.4. IR (ZnSe) νmax (cm^{-1}): 3047, 2360, 2353, 1772, 1699, 1599, 1496, 1354, 1053, 791, 713. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{15}N_2O_2^+$; 303.1128; found, 303.1144.



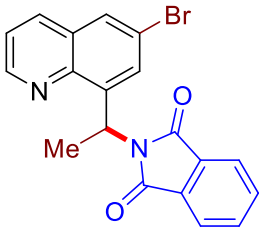
*2-(1-(6-methylquinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 3, Entry **5b**)*

Following the general procedure for sp^3 C-H amidation, 6-methyl-8-ethylquinoline **(4b)** (34.2 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 63% (39.8 mg). Mp = 127 - 129 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.80-8.79 (m, 1H), 8.01 (d, J = 7.8 Hz, 1H), 7.82 (s, 1H), 7.80-7.78 (m, 2H), 7.66-7.65 (m, 2H), 7.50 (s, 1H), 7.30 (dd, J = 8.4, 4.2 Hz, 1H), 6.77 (dd, J = 14.4, 7.2 Hz, 1H), 2.54 (s, 3H), 2.01 (d, J = 7.8 Hz, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.6, 148.9, 144.5, 138.0, 135.9, 135.6, 134.4, 133.8, 132.3, 130.3, 128.4, 126.6, 123.7, 123.2, 121.1, 45.5, 22.0, 18.4. IR (ZnSe) νmax (cm^{-1}): 3314, 2359, 2021, 1705, 1365, 1060, 705, 493. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{20}H_{17}N_2O_2^+$; 317.1285; found, 317.1286.



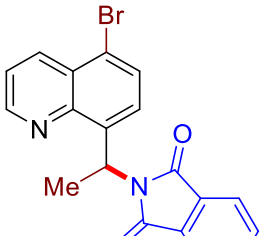
*2-(1-(6-bromoquinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 3, Entry **5c**)*

Following the general procedure for sp^3 C-H amidation, 6-bromo-8-ethylquinoline **(4c)** (46.4 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**RhCp*Cl_2**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 57% (22.9 mg). Mp = 163 - 165 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.86 (dd, J = 4.2, 1.8 Hz, 1H), 8.06 (s, 1H), 8.01 (d, J = 7.8 Hz, 1H), 7.91 (d, J = 2.4 Hz, 1H), 7.87 (dd, J = 5.4, 3.0 Hz, 1H), 7.80 (dd, J = 5.4, 3.0 Hz, 2H), 7.76 (dd, J = 5.4, 3.0 Hz, 1H), 7.67 (dd, J = 5.4, 3.0 Hz, 2H), 7.36 (dd, J = 8.4, 4.2 Hz, 1H), 6.75 (dd, J = 14.4, 7.2 Hz, 1H), 2.00 (d, J = 7.8 Hz, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.4, 150.1, 144.5, 140.6, 135.3, 134.5, 133.9, 132.8, 132.2, 131.8, 129.8, 129.4, 123.7, 123.3, 121.9, 120.3, 45.1, 18.3. IR (ZnSe) νmax (cm^{-1}):3120, 2360, 2199, 1712. 1342, 1029, 844, 694, 478. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{14}BrN_2O_2^+$; 381.0233; found, 381.0234.



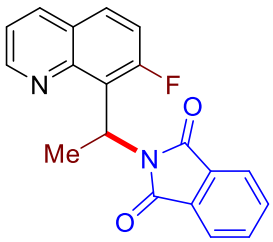
*2-(1-(5-bromoquinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 3, Entry **5d**)*

Following the general procedure for sp^3 C-H amidation, 5-bromo-8-ethylquinoline **(4d)** (46.4 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**RhCp*Cl_2**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 68% (27.3 mg). Mp = 167 - 169 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): δ 8.87 (dd, J = 4.2, 1.8 Hz, 1H), 8.48 (d, J = 8.4 Hz, 1H), 7.89 (d, J = 7.8 Hz, 1H), 7.82 (d, J = 7.8 Hz, 1H), 7.78-7.77 (m, 2H), 7.66-7.64 (m, 2H), 7.44 (dd, J = 8.4, 4.2 Hz, 1H), 6.75 (dd, J = 14.4, 7.2 Hz, 1H), 1.99 (d, J = 7.2 Hz, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.4, 150.3, 146.5, 138.3, 135.7, 133.9, 132.2, 129.9, 128.7, 127.5, 123.2, 122.2, 121.8, 45.2, 18.2. IR (ZnSe) νmax (cm^{-1}): 3614, 1708, 1350, 1041, 844, 725, 678. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{14}BrN_2O_2^+$; 381.0233; found, 381.0230.

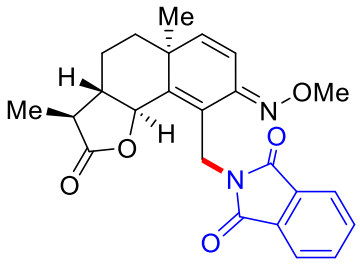


*2-(1-(7-fluoroquinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 3, Entry **5e**)*

Following the general procedure for sp^3 C-H amidation, 8-ethyl-7-fluoroquinoline **(4e)** (35 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**RhCp*Cl_2**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 43% (27.6 mg). Mp = 125 - 127 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.91 (d, J = 4.2 Hz, 1H), 8.10 (d, J = 8.4 Hz, 1H), 7.88 - 7.86 (m, 2H), 7.78 - 7.73 (m, 2H), 7.65 - 7.64 (m, 1H), 7.35 (dd, J = 8.4, 4.2 Hz, 1H), 7.32 (t, J = 10.2 Hz, 1H), 6.84 - 7.81 (m, 1H), 2.09 (dd, J = 7.8, 3.0 Hz, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.2 (d, $J_{C\text{-}F}$ = 51.0 Hz) ,150.5, 136.4, 134.5, 133.8, 132.8, 132.3, 129.40 (d, $J_{C\text{-}F}$ = 10.5 Hz), 125.3, 123.8, 123.1, 122.3 (d, $J_{C\text{-}F}$ = 10.5 Hz), 120.4 (d, $J_{C\text{-}F}$ = 3.0 Hz), 118.03 (d, $J_{C\text{-}F}$ = 27.0 Hz), 44.53, 18.60 (d, $J_{C\text{-}F}$ = 6.0 Hz). ^{19}F NMR (565 MHz, $CDCl_3$, δ): -107.64. IR (ZnSe) νmax (cm^{-1}): 3182, 2923, 2044, 1716, 1342, 1029, 686. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{14}FN_2O_2^+$; 321.1034; found, 321.1034.

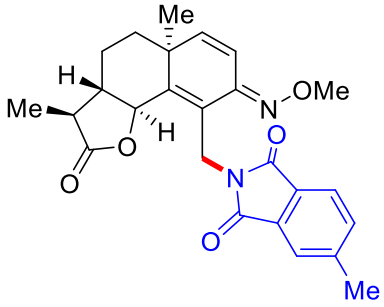


*8-(methoxyimino)-3,5a-dimethyl-2-oxo-2,3,3a,4,5,5a,8,9b-octahydronaphtho[1,2-b]furan-9-yl)methyl)isoindoline-1,3-dione (Scheme 4, Entry **7a**)*

Following the general procedure for sp^3 C-H amidation, santonin oxime **(6a)** (55 mg, 0.2 mmol), 

N-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 120 °C for 72 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as light green solid, yield = 87% (73.3 mg). Mp = 84 - 86 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 7.76 - 7.75 (m, 2H), 7.65 - 7.63 (m, 2H), 6.68 (d, J = 10.2 Hz, 1H), 5.93 (d, J = 9.6 Hz, 1H), 5.13 (d, J = 13.8 Hz, 1H), 5.03 (d, J = 13.8 Hz, 1H), 4.98 (d, J = 10.4 Hz, 1H), 3.73 (s, 3H), 2.44 - 2.37 (m, 2H), 2.04 (d, J = 12.6 Hz, 1H), 1.76 (d, J = 13.2 Hz, 1H), 1.70 (dd, J = 12.0, 3.6 Hz, 1H), 1.64 (d, J = 42 Hz, 1H), 1.32 (d, J = 9.0 Hz, 6H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 177.8, 168.1, 147.4, 144.9, 143.9, 133.6, 132.6, 122.9, 120.6, 112.6, 82.6, 62.2, 51.8, 41.7, 41.2, 38.6, 34.6, 26.1, 24.1, 12.6. IR (ZnSe) νmax (cm^{-1}):2931, 1774, 1705, 1396, 1138, 1037, 802, 713, 516. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{24}H_{25}N_2O_5^+$; 421.1758; found, 421.1759.

*8-(methoxyimino)-3,5a-dimethyl-2-oxo-2,3,3a,4,5,5a,8,9b-octahydronaphtho[1,2-b]furan-9-yl)methyl)-5-methylisoindoline-1,3-dione (Scheme 4, Entry **7b**)*

Following the modified procedure for sp^3 C-H amidation, santonin oxime **(6a)** (55 mg, 0.2 mmol), 

2-hydroxy-5-methylisoindoline-1,3-dione **(2w)** (53.1 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, AgOAc **(**16.7 mg**.** 0.5 equiv**)** HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 76% (61.2 mg). Mp = 124 - 126 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 7.62 (d, J = 7.8 Hz, 1H), 7.55 (s, 1H), 7.42 (d, J = 7.8 Hz, 1H), 6.67 (d, J = 10.2 Hz, 1H), 5.92 (d, J = 10.2 Hz, 1H), 5.10 (d, J = 14.4 Hz, 1H), 5.01 - 4.96 (m, 2H), 3.72 (s, 3H), 2.46 (s, 3H), 2.41 - 2.38 (m, 2H), 2.03 - 2.01 (m, 1H), 1.76 - 1.73 (m, 1H), 1.70 - 1.76 (m, 1H), 1.62 (dd, J = 12.6, 4.2 Hz, 1H), 1.32 - 1.29 (m, 6H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 177.9, 168.3, 168.2, 147.3, 144.9, 144.6, 143.8, 134.1, 132.9, 129.9, 123.4, 122.8, 120.7, 112.5, 82.6, 62.2, 51.8, 41.6, 41.1, 38.5, 34.5, 26.1, 24.1, 22.0, 12.6. IR (ZnSe) νmax (cm^{-1}): 2511, 2407, 2229, 2048, 1963, 1774, 1701, 1381,941. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{25}H_{26}N_2O_5Na^+$; 457.1734; found, 457.1748.

*8-((benzyloxy)imino)-3,5a-dimethyl-2-oxo-2,3,3a,4,5,5a,8,9b-octahydronaphtho[1,2-b]furan-9-yl)methyl)isoindoline-1,3-dione (Scheme 4, Entry **7c**)*

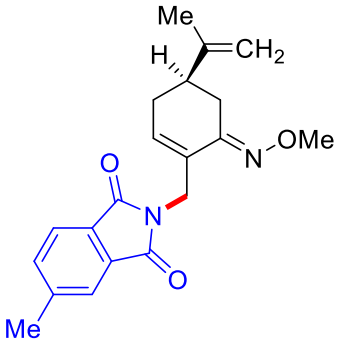
Following the modified procedure for sp^3 C-H amidation, 8-((benzyloxy)imino)-3,5a,9-trimethyl-3a,4,5,5a,8,9b-hexahydronaphtho[1,2-b]furan-2(3H)-one **(6b)** (70.2 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, AgOAc **(**16**.**7 mg**.** 0**.**5 equiv**)** HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as light yellow solid, yield = 71% (71.6 mg). Mp = 96 - 98 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 7.69 (dd, J = 5.4, 3.0 Hz, 2H), 7.61 (dd, J = 5.4, 3.0 Hz, 2H), 7.22 - 7.21 (m, 2H), 7.15 - 7.09 (m, 3H), 6.78 (d, J = 10.2 Hz, 1H), 5.93 (d, J = 10.2 Hz, 1H), 5.11 (d, J = 13.8 Hz, 1H), 5.04 - 4.97 (m, 4H), 2.42 - 2.37 (m, 2H), 2.05 - 2.01 (m, 1H), 1.77 - 1.74 (m, 1H), 1.71 - 1.66 (m, 1H), 1.65 - 1.62(m, 1H), 1.31 (d, J = 7.2 Hz, 6H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 177.8, 168.1, 147.8, 144.9, 144.2, 138.1, 133.5, 132.4, 128.2, 128.0, 127.6, 122.9, 120.6, 112.9, 82.6, 76.4, 51.8, 41.7, 41.1, 38.5, 34.7, 26.0, 24.1, 12.6. IR (ZnSe) νmax (cm^{-1}): 2881, 2353, 2175, 1982, 1770, 1689, 1381, 1029, 941. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{30}H_{29}N_2O_5^+$; 497.2071; found, 497.2080.

*2-((6-(methoxyimino)-4-(prop-1-en-2-yl)cyclohex-1-en-1-yl)methyl)isoindoline-1,3-dione (Scheme 4, Entry **7d**)*

Following the modified procedure for sp^3 C-H amidation, 2-methyl-5-(prop-1-en-2-yl)cyclohex-2-en-1-one *O*-methyl oxime **(6c)** (36 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6**.**1 mg, 5 mol**%)**, $AgSbF_6$ **(**13**.**7 mg, 20 mol**%)**, AgOAc **(**16**.**7 mg**.** 0**.**5 equiv**)** HFIP **(**1 mL, 0**.**2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as light white solid, yield = 62% (40.3 mg). Mp = 103 - 105 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 7.87 (dd, J = 5.4, 3.0 Hz, 2H), 7.73 (dd, J = 5.4, 3.0 Hz, 2H), 5.88 - 5.86 (m, 1H), 4.76 (t, J = 1.8 Hz, 1H), 4.74 - 4.73 (m, 1H), 4.56 - 4.54 (m, 2H), 3.87 (s, 3H), 3.15 - 3.11 (m, 1H), 2.36 - 2.31 (m, 1H), 2.67 - 2.22 (m, 1H), 2.09 - 1.99 (m, 2H), 1.71 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.3, 153.8, 147.7, 134.1, 132.3, 130.8, 128.6, 123.4, 110.2, 62.0, 40.0, 38.1, 30.1, 27.7, 20.7. IR (ZnSe) νmax (cm^{-1}): 2403, 2160, 2048, 1921, 1774, 1701, 1381, 1029, 941. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{19}H_{21}N_2O_3^+$; 325.1547; found, 325.1562.

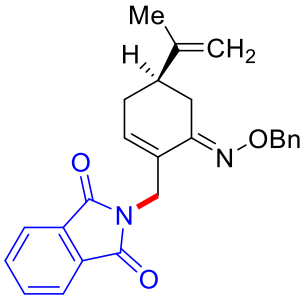
*2-((6-(methoxyimino)-4-(prop-1-en-2-yl)cyclohex-1-en-1-yl)methyl)-5-methylisoindoline-1,3-dione (Scheme 4, Entry **7e**)*

Following the modified procedure for sp^3 C-H amidation, 2-methyl-5-(prop-1-en-2-yl)cyclohex-2-en-1-one O-methyl oxime **(6c)** (36 mg, 0.2 mmol), 2-hydroxy-5-methylisoindoline-1,3-dione **(2w)** (53.1 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, AgOAc **(**16.7 mg**.** 0.5 equiv**)** HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 120 °C for 24 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as white solid, yield = 67% (45.3 mg). Mp = 111 - 113 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 7.74 (d, J = 7.8 Hz, 1H), 7.67 (s, 1H), 7.51 (d, J = 7.8 Hz, 1H), 5.84 (dd, J = 6.0, 1.8 Hz, 1H), 4.75 (d, J = 14.4 Hz, 2H), 4.53 (s, 2H), 3.87 (s, 3H), 3.15 - 3.11 (m, 1H), 2.51 (s, 3H), 2.35 - 2.30 (m, 1H), 2.26 - 2.21 (m, 1H), 2.08 - 1.99 (m, 2H), 1.71 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 177.8, 168.1, 147.4, 144.9, 143.9, 133.6, 132.6, 122.9, 120.6, 112.6, 82.6, 62.2, 51.8, 41.7, 41.2, 38.6, 34.6, 26.1, 24.1, 12.6. IR (ZnSe) νmax (cm^{-1}): 2920, 2314, 2184, 2048, 1978, 1716, 1384, 1107, 729. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{20}H_{23}N_2O_3^+$; 339.1703; found, 339.1712.



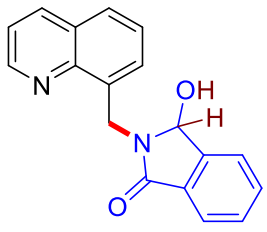
*2-((6-((benzyloxy)imino)-4-(prop-1-en-2-yl)cyclohex-1-en-1-yl)methyl)isoindoline-1,3-dione (Scheme 4, Entry **7f**)*

Following the modified procedure for sp^3 C-H amidation, 2-methyl-5-(prop-1-en-2-yl)cyclohex-2-en-1-one *O*-benzyl oxime **(6d)** (51.6 mg, 0.2 mmol), *N*-hydroxyphthalimide **(2a)** (48.9 mg, 0.3 mmol), **[**$RhCp^*Cl_2$**]**$_2$ **(**6.1 mg, 5 mol**%)**, $AgSbF_6$ **(**13.7 mg, 20 mol**%)**, AgOAc **(**16.7 mg**.** 0.5 equiv**)** HFIP **(**1 mL, 0.2 M**)** were used and reaction was run at 120 °C for 36 hours**.** Title compound was isolated from flash chromatography (20% EtOAc/*n*-hexane) as colorless viscous liquid, yield = 69% (55.2 mg). ^{1}H NMR (600 MHz, $CDCl_3$, δ): 7.85 (dd, J = 5.4, 3.0 Hz, 2H), 7.72 (dd, J = 5.4, 3.0 Hz, 2H), 7.35 - 7.30 (m, 4H), 7.28 - 7.27 (m, 1H), 5.91 - 5.89 (m, 1H), 5.11 (s, 2H), 4.74 (d, J = 11.4 Hz, 2H), 4.55 (s, 2H), 3.19 - 3.16 (m, 1H), 2.38 - 2.32 (m, 1H), 2.27 - 2.21 (m, 1H), 2.09 - 2.04 (m, 2H), 1.71 (s, 3H). ^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$, δ): 168.2, 154.1, 147.6, 137.9, 133.9, 132.2, 131.1, 128.5, 128.3, 128.2, 127.7, 123.3, 110.2, 76.2, 39.9, 38.1, 30.0, 27.8, 20.5. IR (ZnSe) νmax (cm^{-1}): 2924, 2337, 2144, 2040, 2036, 1716, 1427, 1384, 948. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{25}H_{25}N_2O_3^+$; 401.1860; found, 401.1862.



*3-hydroxy-2-(quinolin-8-ylmethyl)isoindolin-1-one (Scheme 5, Entry **8a**)*

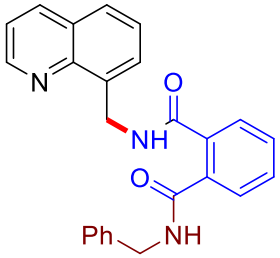
Following the general procedure of post synthetic transformations (a), **3a** (28.8 mg, 0.1 mmol), $NaBH_4$ **(**11.4 mg, 3 equiv**)**, *i*-propanol**:**toluene**:**H_2O **(**6**:**1**:**1, 0.5 mL**)** were used and reaction was run at 0 °C in ice bath for 4 hours**.** Title compound was isolated from flash chromatography (25% EtOAc/*n*-hexane) as brown solid, yield = 83% (48.3 mg). Mp = 190 - 192 °C. ^{1}H NMR (600 MHz, $CDCl_3$, δ): 8.99 - 8.98 (m, 1H), 8.27 (d, J = 8.4 Hz, 1H), 8.00 (d, J = 7.2 Hz, 1H), 7.84 (d, J = 7.8 Hz, 1H), 7.78 (d, J = 7.8 Hz, 1H), 7.58 (t, J = 8.4 Hz, 1H), 7.54 - 7.49 (m, 3H), 7.46 - 7.43 (m, 1H), 5.68 (s, 1H), 5.42 (d, J = 14.4 Hz, 1H), 5.21 (d,



J = 13.8 Hz, 1H). $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, δ): 167.0, 149.9, 146.7, 143.4, 137.9, 134.4, 133.2, 132.5, 131.9, 129.5, 128.7, 128.7, 127.5, 123.4, 123.2, 121.5, 81.1, 39.4. IR (ZnSe) νmax (cm^{-1}):2924, 1685, 1419, 1060, 763, 721, 540. HRMS (ESI-TOF) (m/z): $[M + H]^+$ calcd for $C_{18}H_{15}N_2O_2^+$; 291.1128; found, 291.1128.

*N^1-benzyl-N^2-(quinolin-8-ylmethyl)phthalamide (Scheme 5, Entry **8b**)*

Following the general procedure of post synthetic transformations (b), **3a** (28.8 mg, 0.1 mmol), benzylamine (21.1 mg, 2.0 equiv.) and H_2O (0.2 mL) were used and reaction was run at room temperature 12 hours**.** Title compound was isolated from flash chromatography (30% EtOAc/*n*-hexane) as white solid, yield = 87% (75.2 mg). Mp = 181 - 183 °C. ^1H NMR (600 MHz, $CDCl_3$, δ): 8.90 (d, J = 4.2 Hz, 1H), 8.20 (d, J = 7.8 Hz, 1H), 7.80 - 7.75 (m, 4H), 7.52 - 7.50 (m, 1H), 7.46 - 7.39 (m, 4H), 7.29 (t, J = 7.2 Hz, 3H), 7.25 - 7.23 (m, 1H), 5.11 (d, J = 6.0 Hz, 2H), 4.31 (d, J = 5.4 Hz, 2H). $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$, δ): 169.6, 168.3, 159.1, 156.2, 143.5, 138.1, 135.2, 134.5, 130.5, 130.2, 129.6, 129.4, 128.8, 128.73, 128.72, 128.0, 127.84, 127.79, 127.5, 126.7, 121.5, 44.0, 42.0. IR (ZnSe) νmax (cm^{-1}): 2333, 2017, 1693, 1384, 1366, 1091, 952, 528. HRMS (ESI-TOF) (m/z): $[M + Na]^+$ calcd for $C_{25}H_{21}N_3O_2Na^+$; 418.1526; found, 418.1526.



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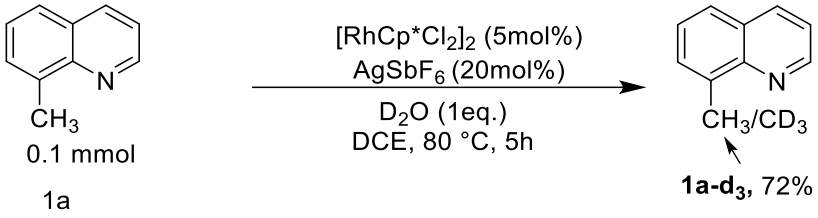
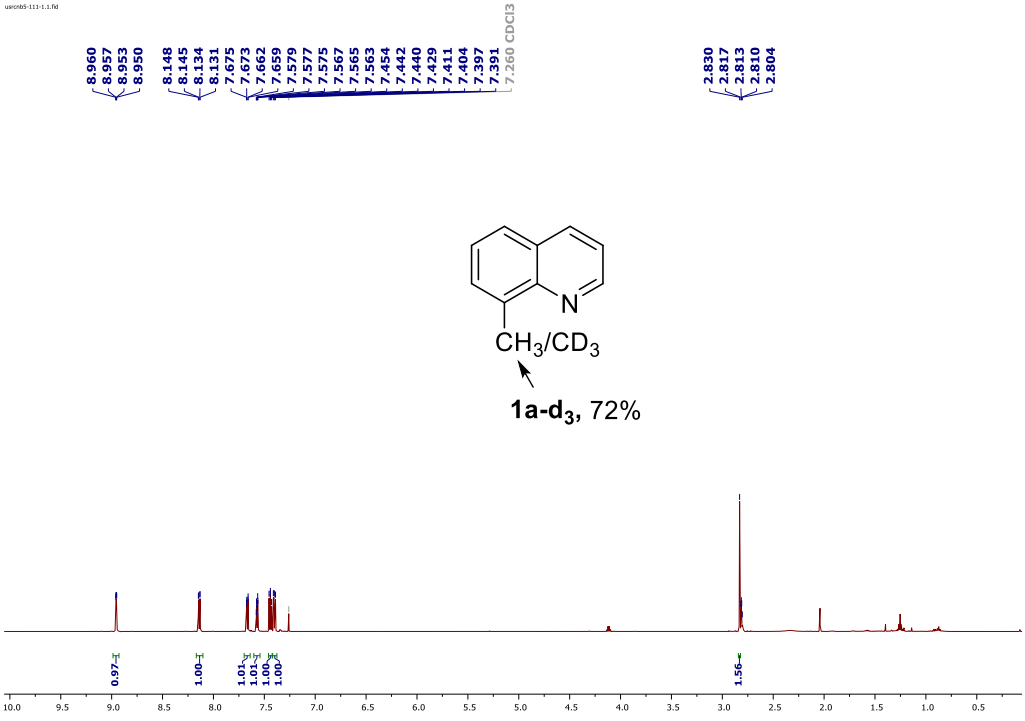
8. Mechanistic studies

*8.1. H/D exchange reaction (**Scheme S2**)*

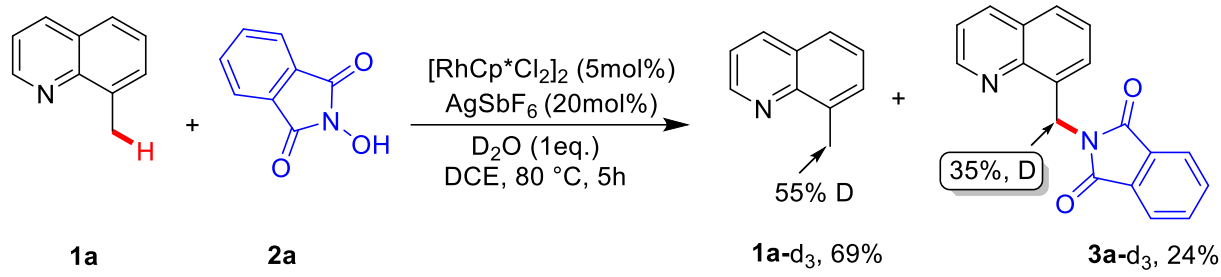


To an oven-dried 15 mL Schlenk tube was added substituted 8-methyl quinoline **1** (0.10 mmol), [RhCp*Cl_2]$_2$ (3 mg, 5 mol%), $AgSbF_6$ (6.8 mg, 20 mol%), DCE (0.5 mL, 0.2 M) and D_2O (20 mg, 1 equiv.). The tube was then stirred for 5 h at 80 °C on a preheated IKA dry block followed by cooling and the contents were dried under reduced pressure. The contents were subjected to flash chromatography (5% EtOAc/*n*-hexane) to give the product as colorless oil with 72% *d* incorporation at C-8 position.

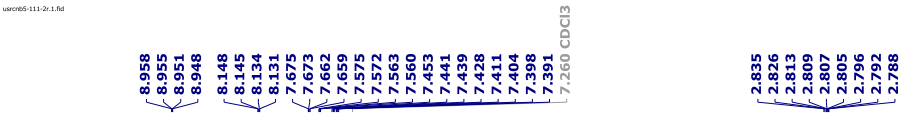


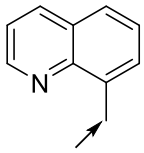
^{1}H NMR of isolated 8-methyl quinoline-*d*$_3$ (1a-d$_3$)

6.2 *H/D exchange reaction with N*-hydroxy phthalimide **2a** (***Scheme S3***)



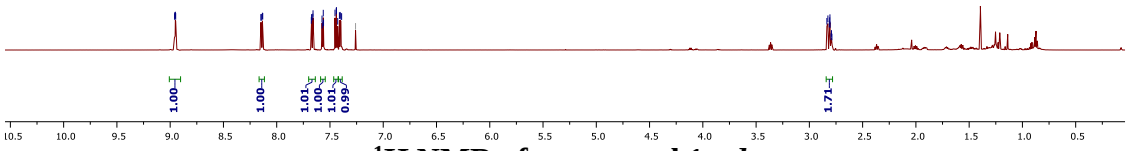
To an oven-dried 15 mL Schlenk tube was added substituted 8-methyl quinoline **1a** (0.20 mmol, 28.6 mg), *N*-hydroxy phthalimide **2a** (48 mg, 1.5 equiv.), [RhCp*Cl_2]$_2$ (3 mg, 5 mol%), $AgSbF_6$ (6.8 mg, 20 mol%), DCE (0.5 mL, 0.2 M) and D_2O (20 mg, 1 equiv.). The tube was then stirred for 5 h at 80 °C on a preheated IKA dry block followed by cooling and the contents were dried under reduced pressure. The contents were subjected to flash chromatography, the reactant ***(1a)*** was recovered in 69*%* yield with 55*% d*-incorporation and product ***(3a)*** was obtained in 24*%* yield with 24*% d*-incorporation at benzylic position.



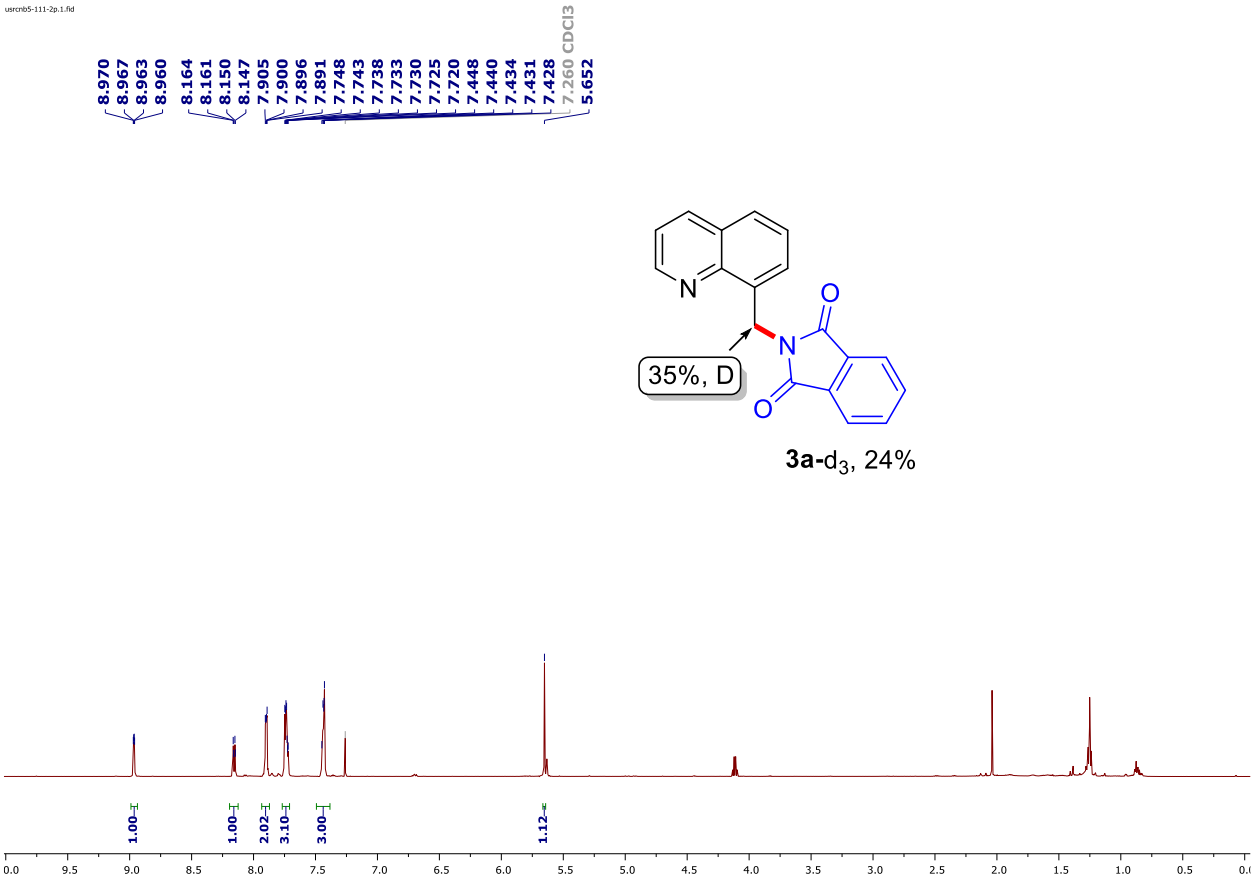


55% D

1a, Reactant
Recovered: 69%

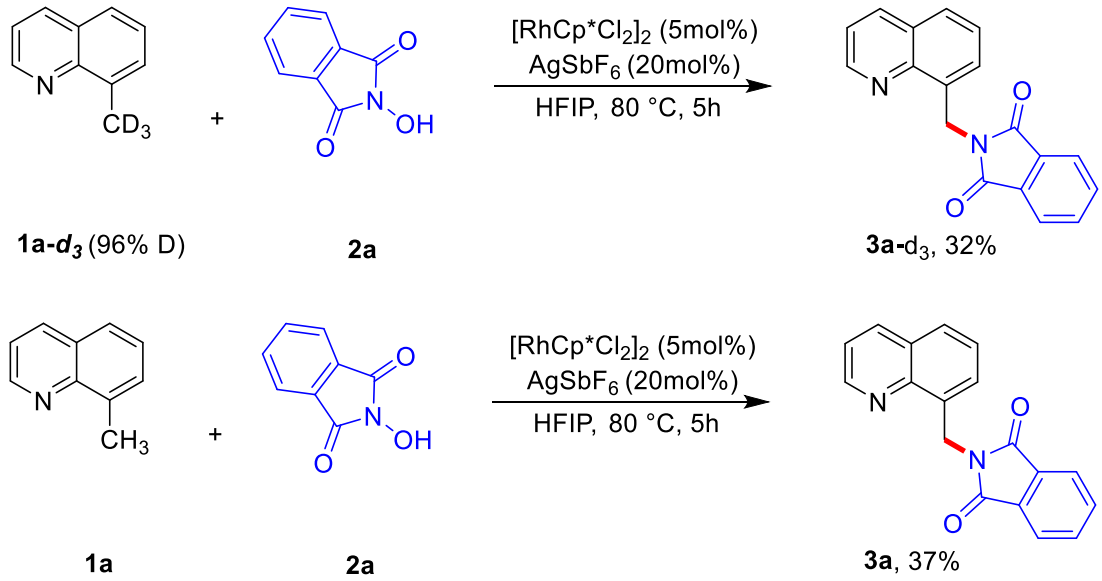


^{1}H NMR of compound 1a-*d*$_3$



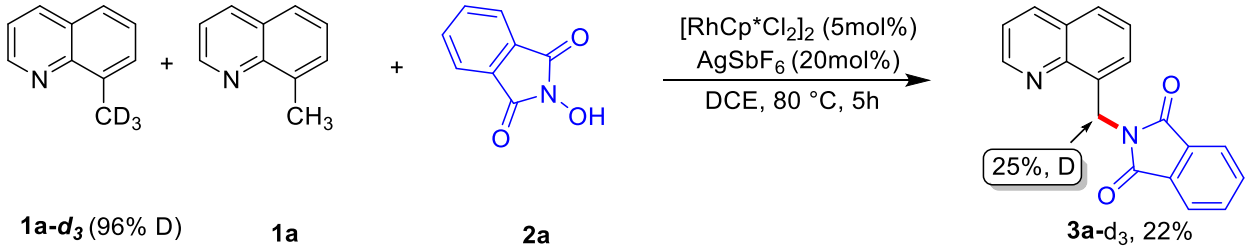
^{1}H NMR of compound 3a

*6.3. Parallel experiments for KIE (Kinetic Isotopic Effect) (**Scheme S4**)*



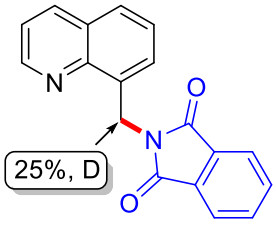
Two independent reactions were carried out using isotopically labelled 8-methylquinoline-d_3 (0.1 mmol) and 8-methyl quinoline (0.1 mmol), *N*-hydroxy phthalimide (**2a**, 1.5 eq.) [$RhCp^*Cl_2]_2$ (5mol%, 3.1 mg), $AgSbF_6$ (20 mol%, 6.8 mg) and in HFIP (0.5 mL) was stirred at 80 °C for 5 hours. After cooling to room temperature, the solvent was removed under reduced pressure. The contents were subjected to flash chromatography, product (3a-d_3) was obtained in 32% yield and product (3a) was obtained in 37% yield indicating that K_H/K_D value is 1.15.

*6.4. Competetion experiments for KIE (Kinetic Isotopic Effect) (**Scheme S5**)*

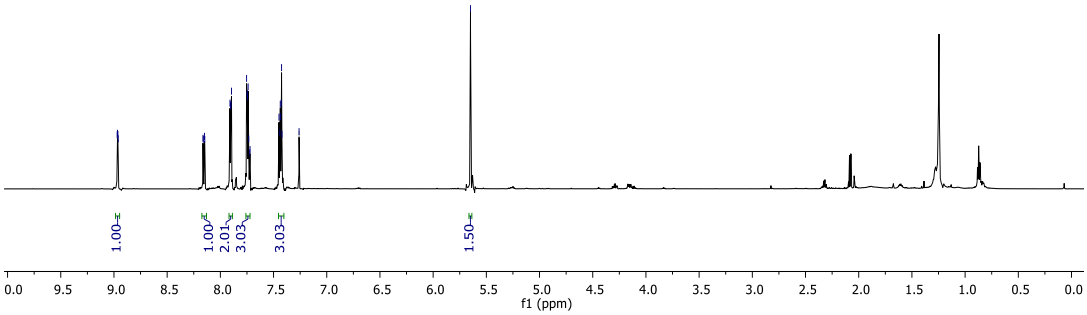


1a-d_3 (96% D) **1a** **2a** **3a-d_3**, 22%

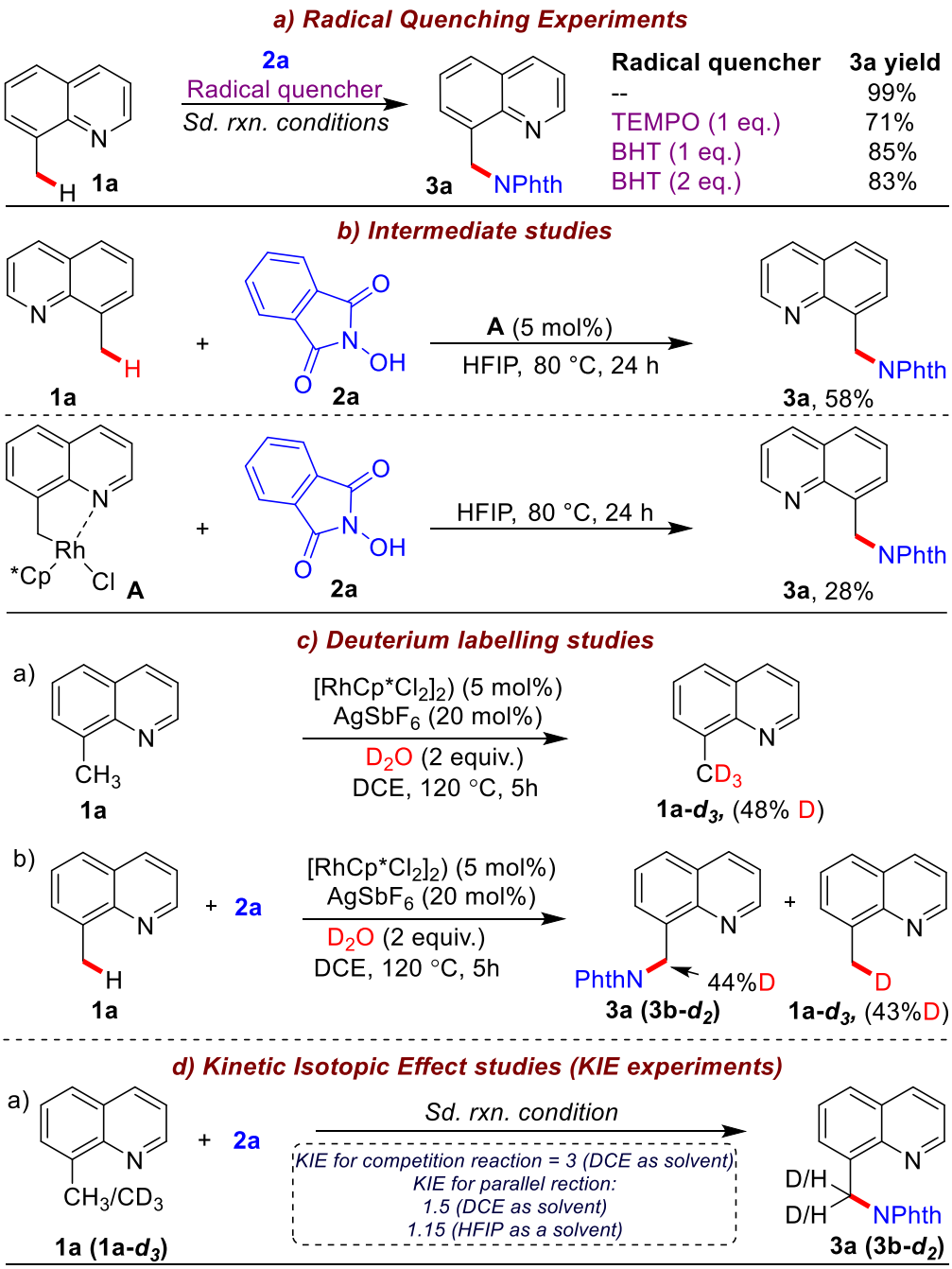
A reaction was carried out using isotopically labelled 8-methylquinoline-d3 (0.1 mmol) and 8-methyl quinoline (0.1 mmol), N-hydroxy phthalimide (**2a**, 1.5 eq.) [$RhCp^*Cl_2]_2$ (5mol%, 6.12 mg), $AgSbF_6$ (20 mol%, 6.8 mg) and in DCE (1 mL) was stirred at 80 °C for 5 hours. After cooling to room temperature, the solvent was removed under reduced pressure. The contents were subjected to flash chromatography, product (**3a-d_3**) was obtained in 22% yield with 25% d-incorporation at benzylic position indicating that KH/KD value is 3.



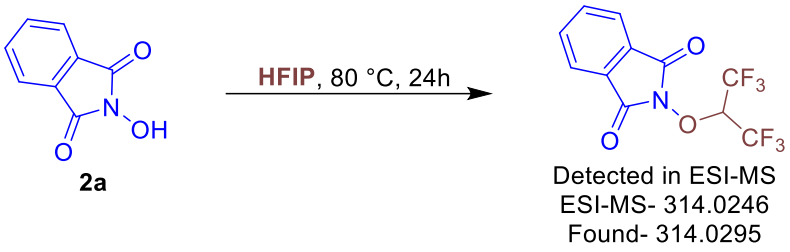
3a-d_3, 22%



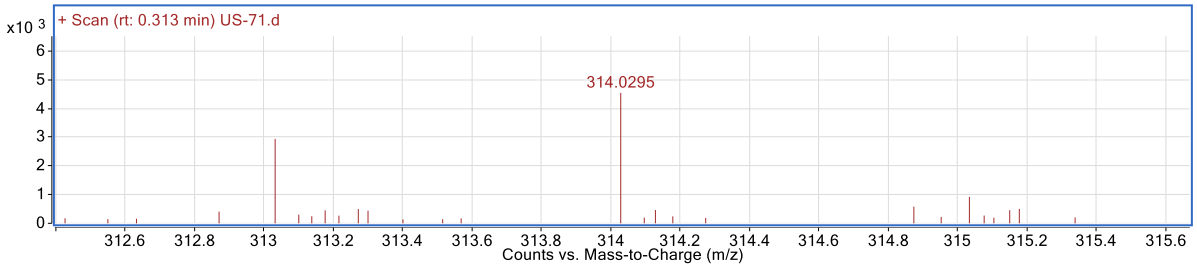
*6.5. Overall mechanistic studies (**Scheme S6**)*



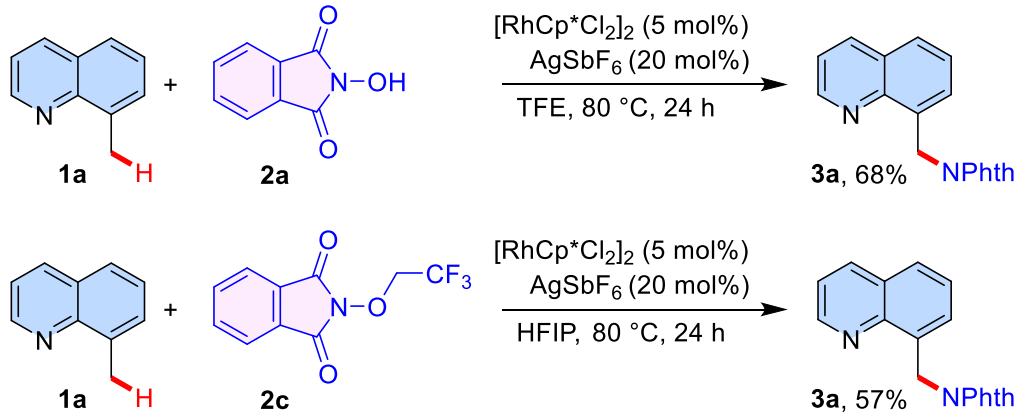
*6.5. Experiment for in-situ exchange of HFIP with N-hydroxyphthalimide (**2a**) (**Scheme S7**)*



A reaction was carried out using *N*-hydroxy phthalimide (**2a**, 0.1 mmol) in HFIP (0.5 mL) at 80 °C for 24 hours. After cooling to room temperature, the contents of the reactions were subjected for ESI-MS analysis. The predicted intermediate was detected in ESI-MS analysis suggesting HFIP may have a role in the in-situ exchange with -OH.

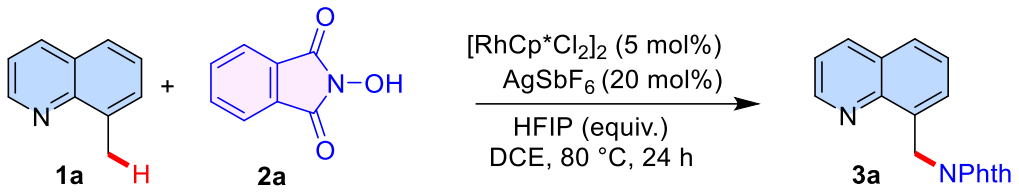


6.6 Experiment with hypothesized intermediate



In order to prove the hypothesis, we synthesized product which was formed by the exchange of TFE with the –OH of *N*-hydroxyphthalimide (**2a**). When we subjected the synthesized product in our standard reaction conditions similar results were obtained when the reaction was carried out in TFE as a solvent.

*6.7 Studies for exchange of **2a** with HFIP*

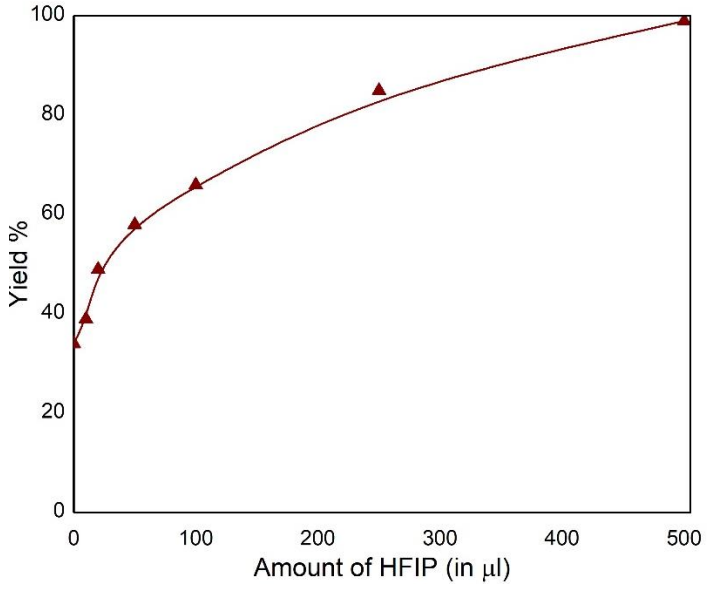


In order to confirm that an exchange between 2a and HFIP takes place during the reaction, series of experiments were carried out by varying the amount of HFIP in DCE as a solvent. The obtained results indicate that HFIP is accelerating the leaving tendency of hydroxyl group which has a profound effect on the yield of product.

Results:

S. No	Quantity of HFIP in DCE	NMR Yield of **3a**
1	-	34%
2	10.5 µL (1 equiv)	39%
3	21.0 µL (2 equiv)	49%
4	52.6 µL (5 equiv)	58%
5	105.0 µL (10 equiv)	66%
6	250 µL (1:1)	85%
7	500 (HFIP only)	99%

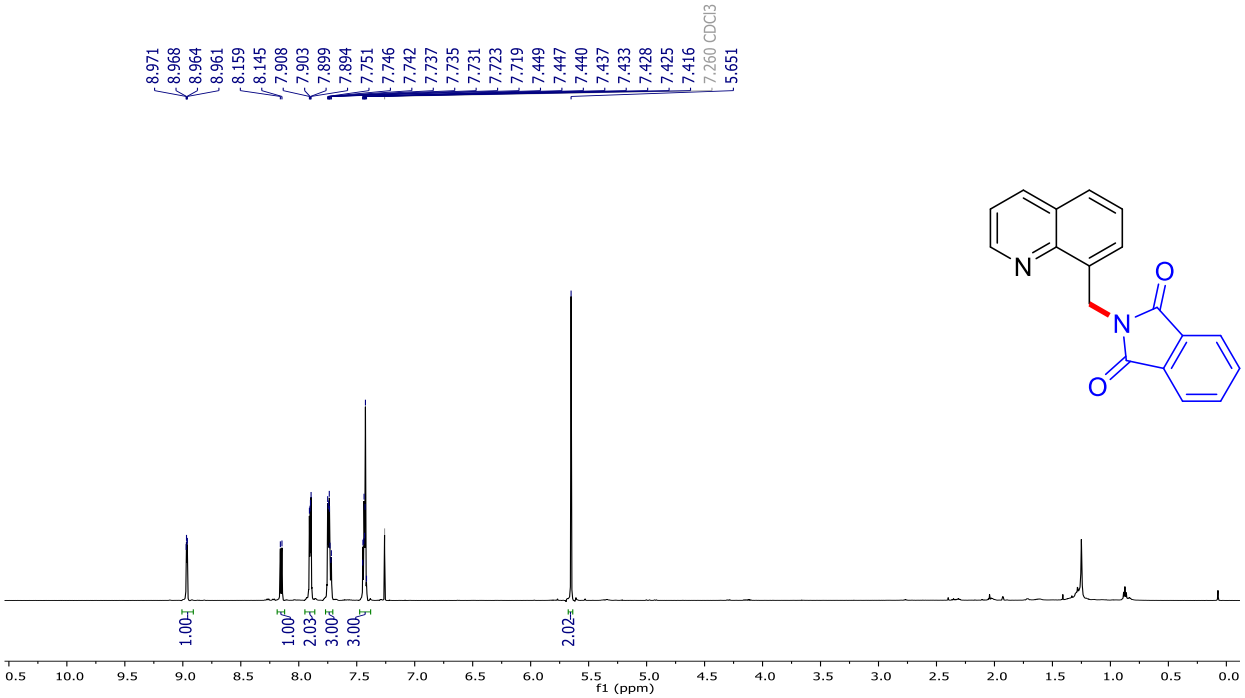
Graph:



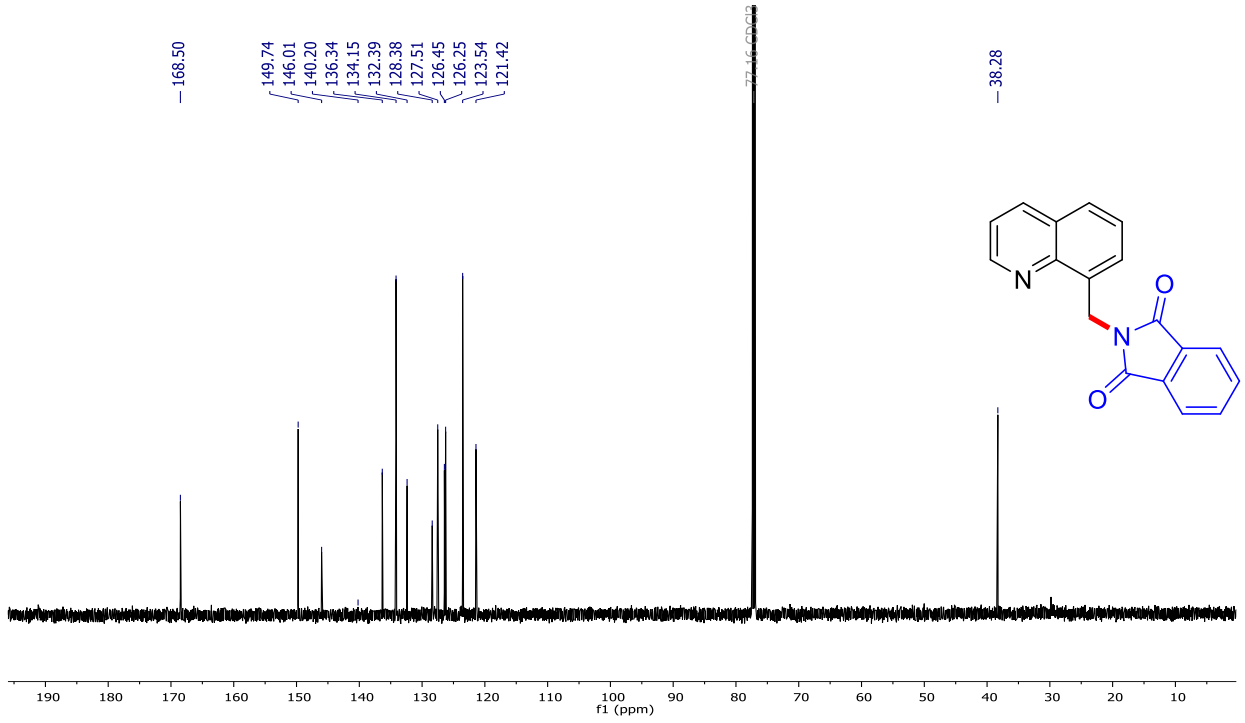
9. ^{1}H and ^{13}C Spectral data

*2-(quinolin-8-ylmethyl)isoindoline-1,3-dione (Scheme 2, Entry **3a**)*

^{1}H NMR (600 MHz)

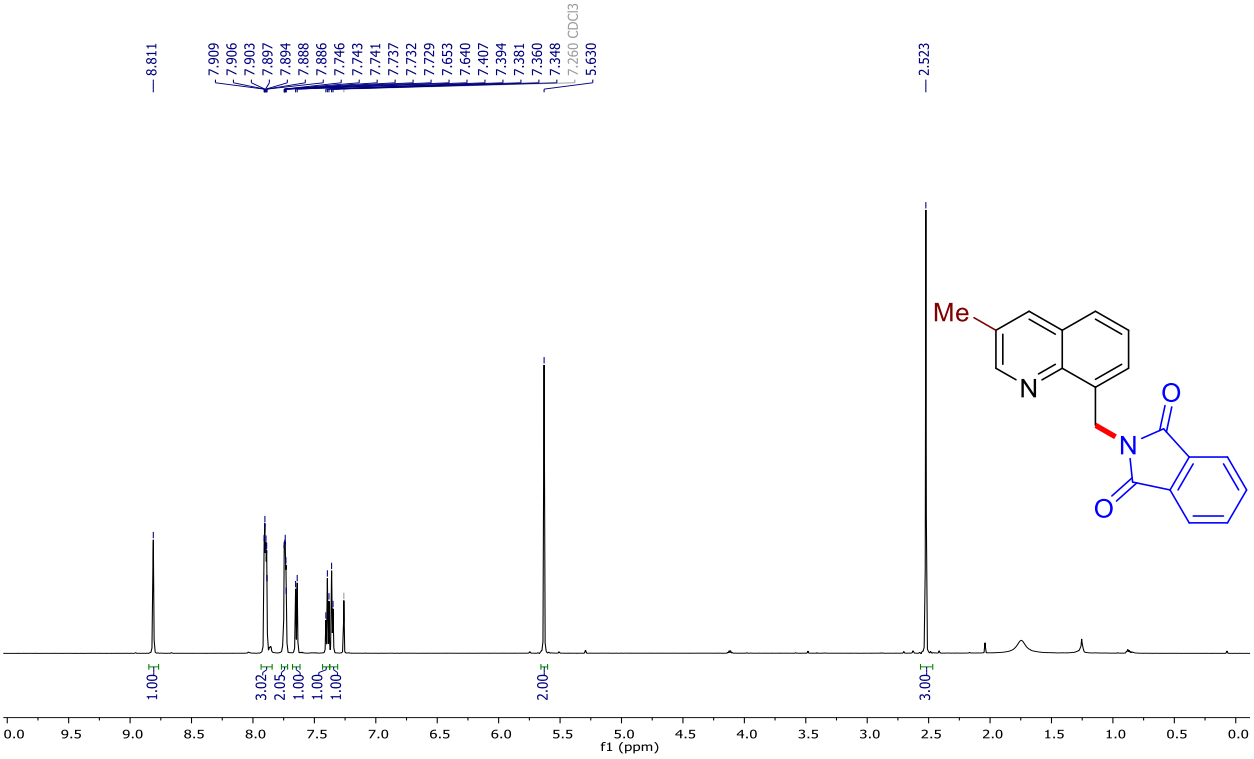


^{13}C{^{1}H} NMR (150 MHz)

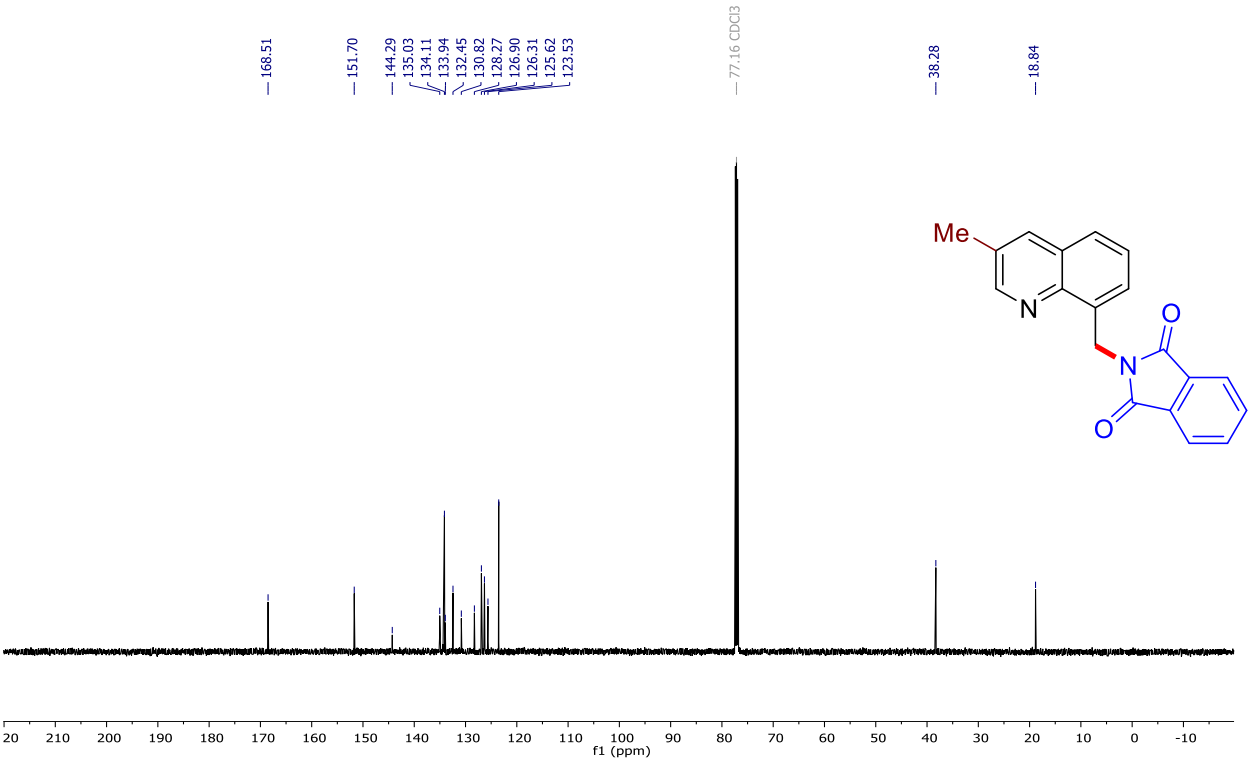


*2-((3-methylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3b**)*

^{1}H NMR *(*600 MHz*)*

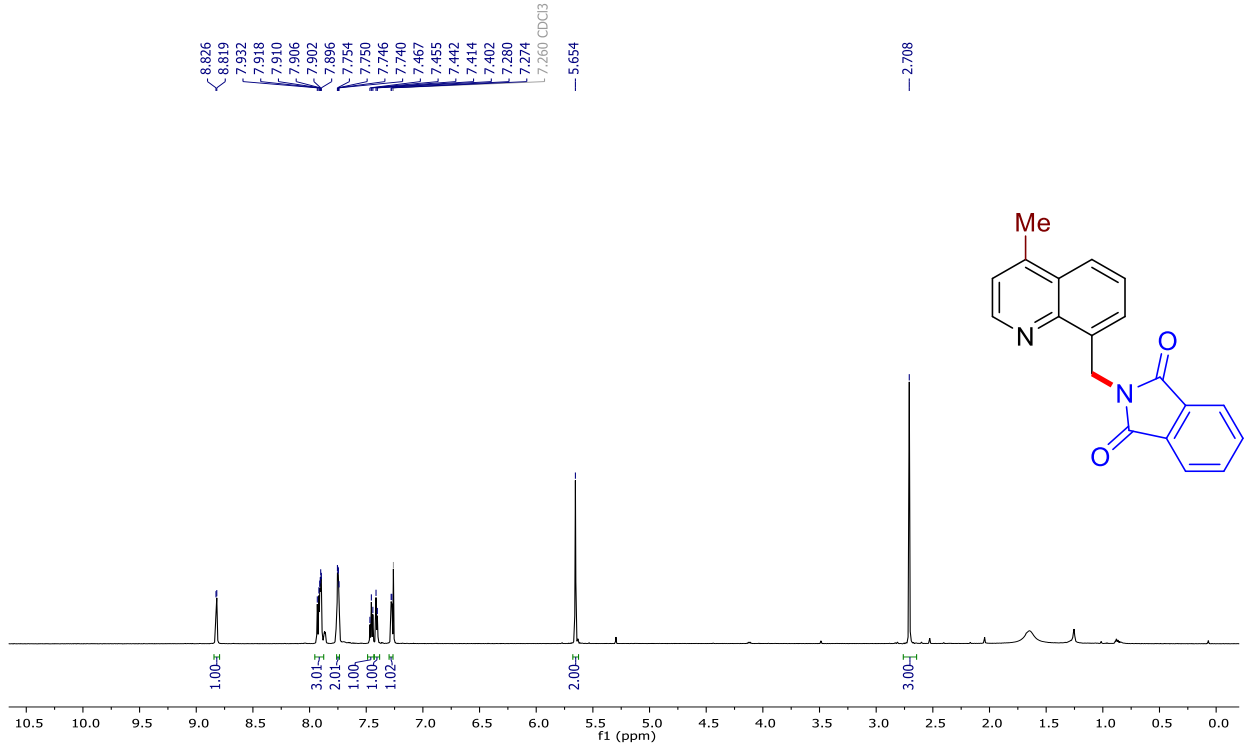


^{13}C{^{1}H} NMR *(*150 MHz*)*

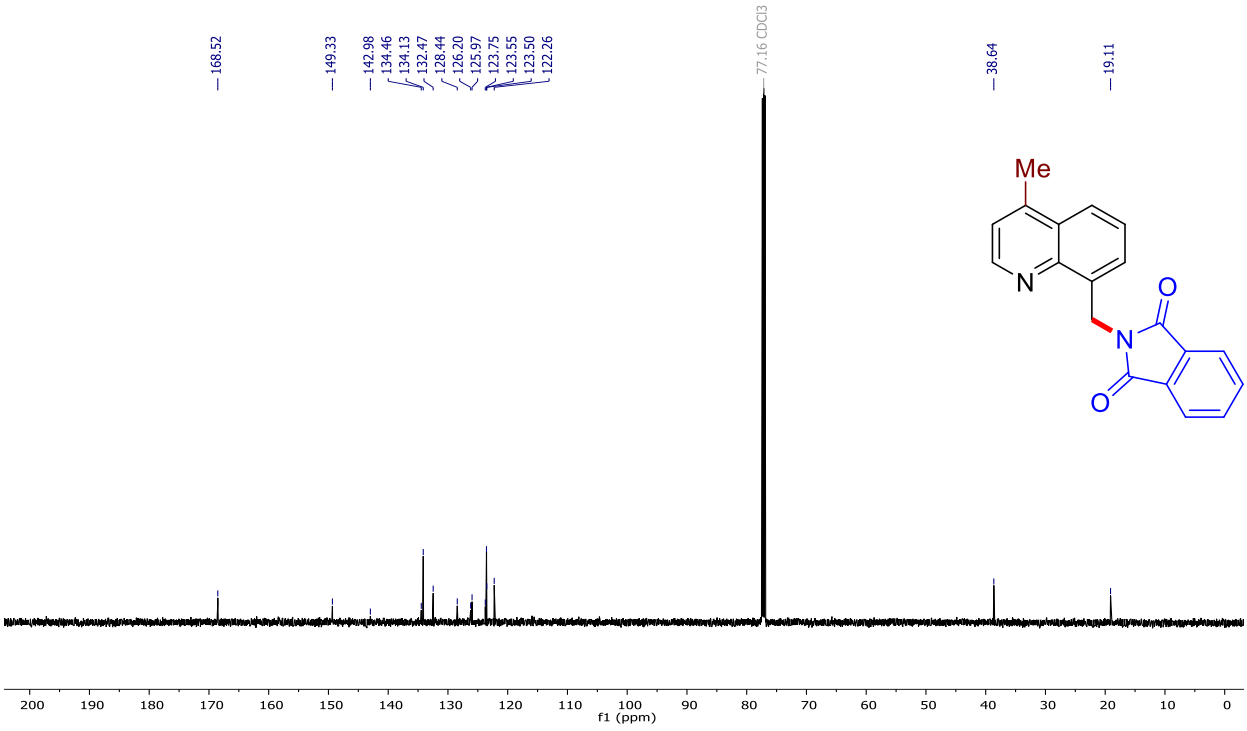


*2-((4-methylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3c**)*

^{1}H NMR *(*600 MHz*)*

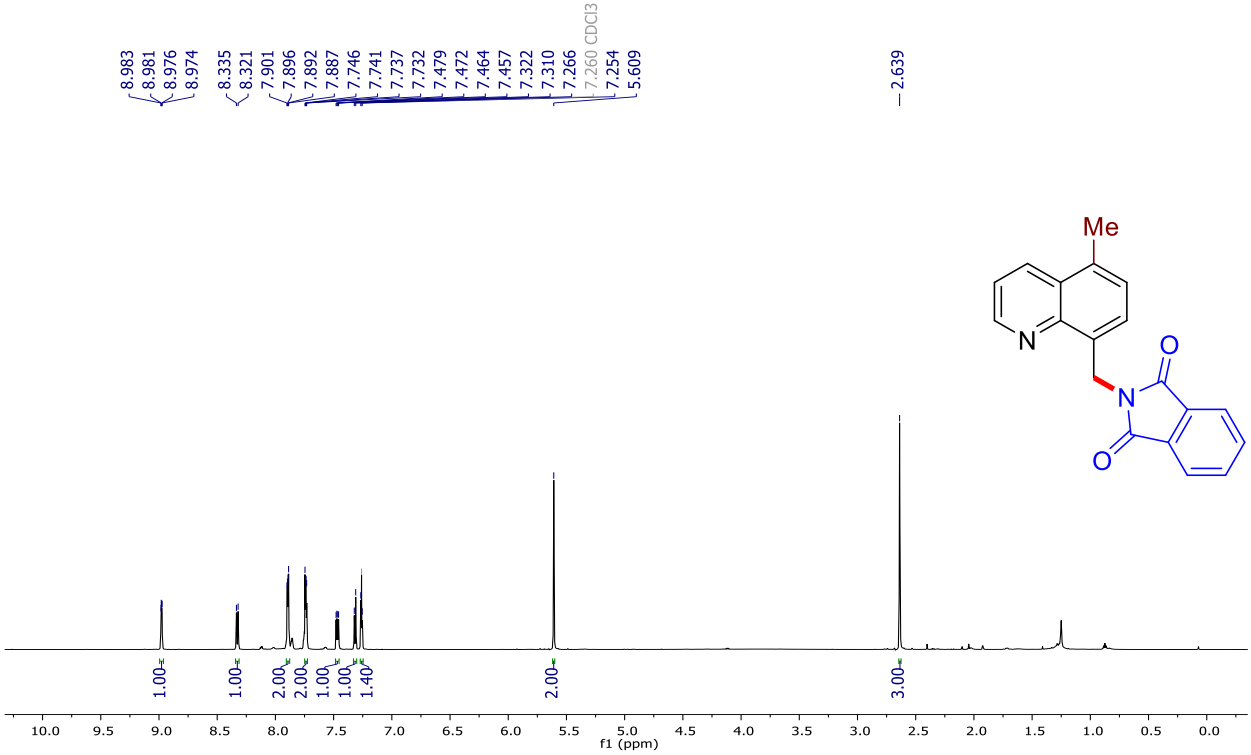


^{13}C{^{1}H} NMR *(*150 MHz*)*

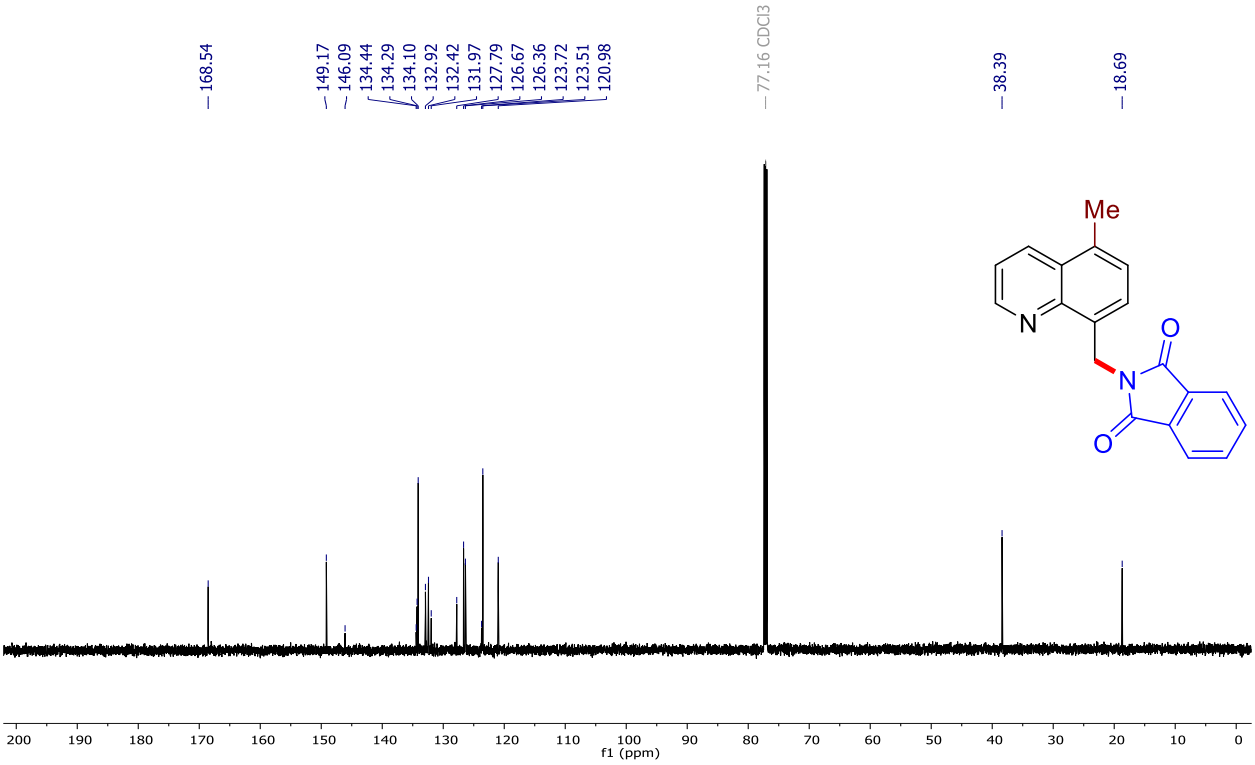


*2-((5-methylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3d**)*

^{1}H NMR *(*600 MHz*)*

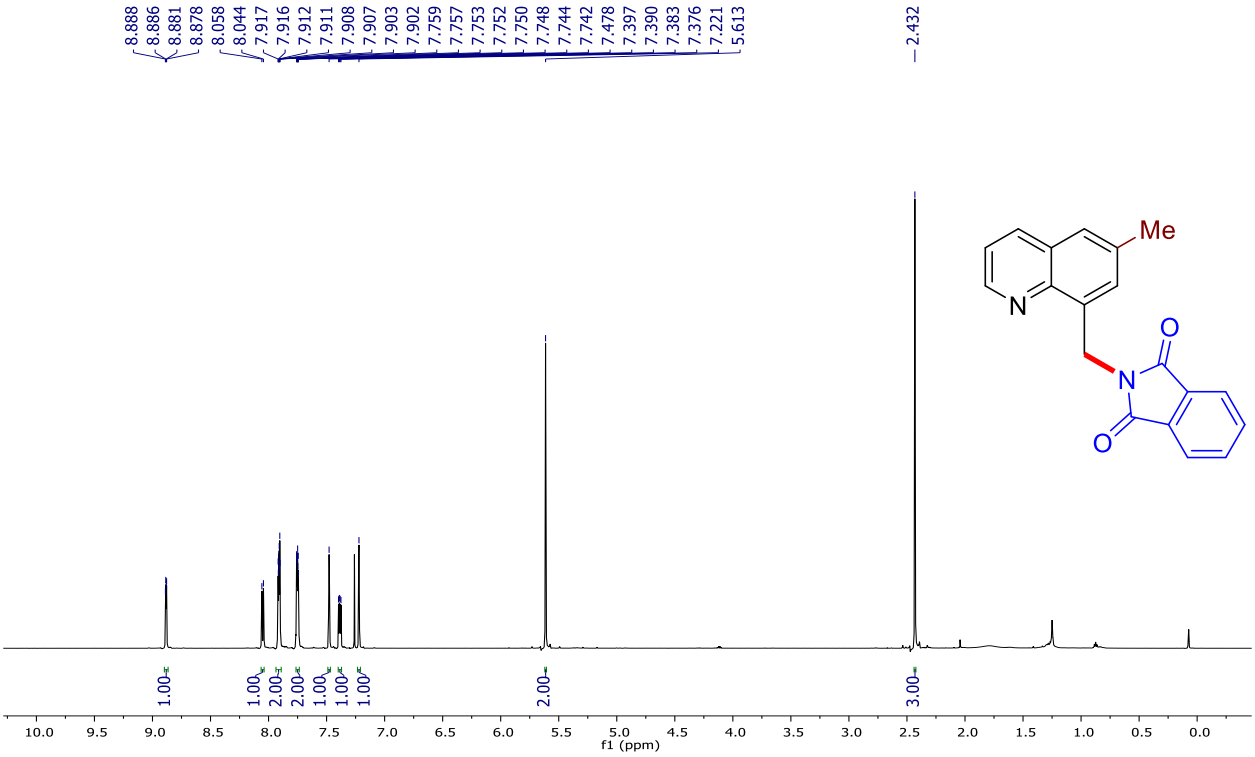


^{13}C{^{1}H} NMR *(*150 MHz*)*

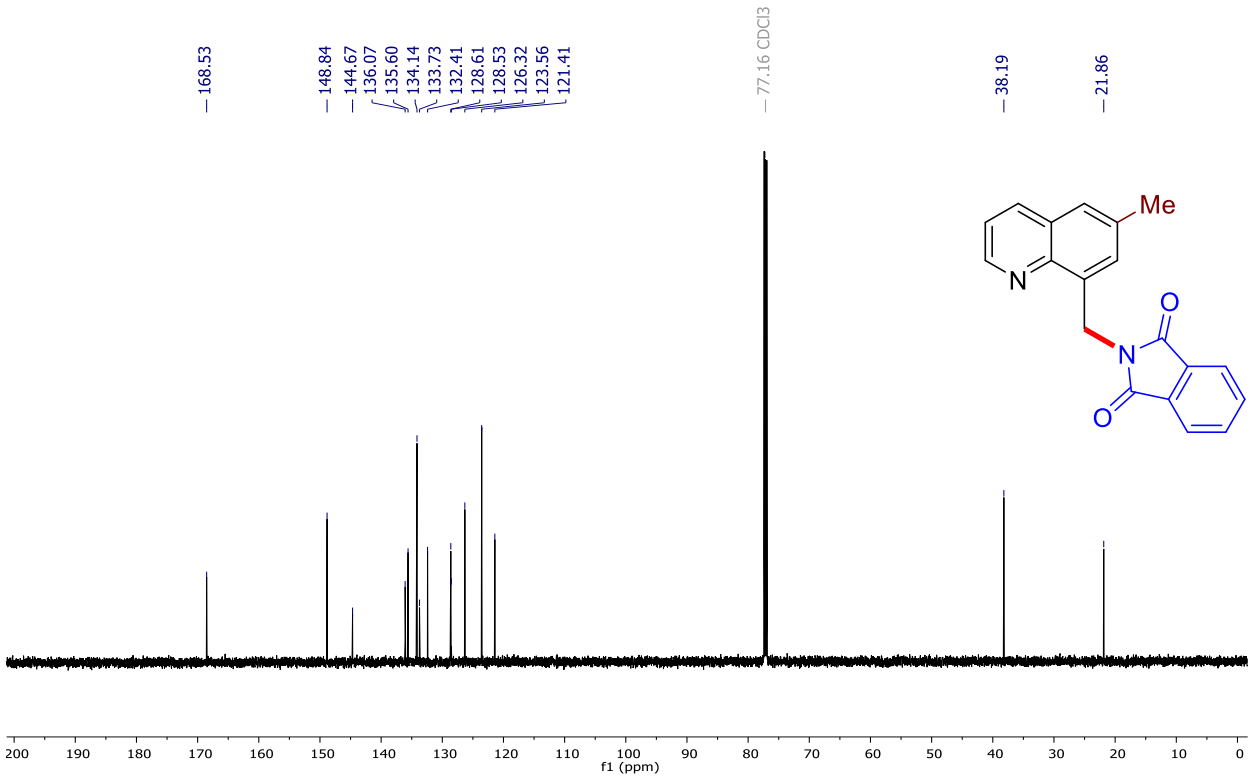


*2-((6-methylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3e**)*

^{1}H NMR (600 MHz)

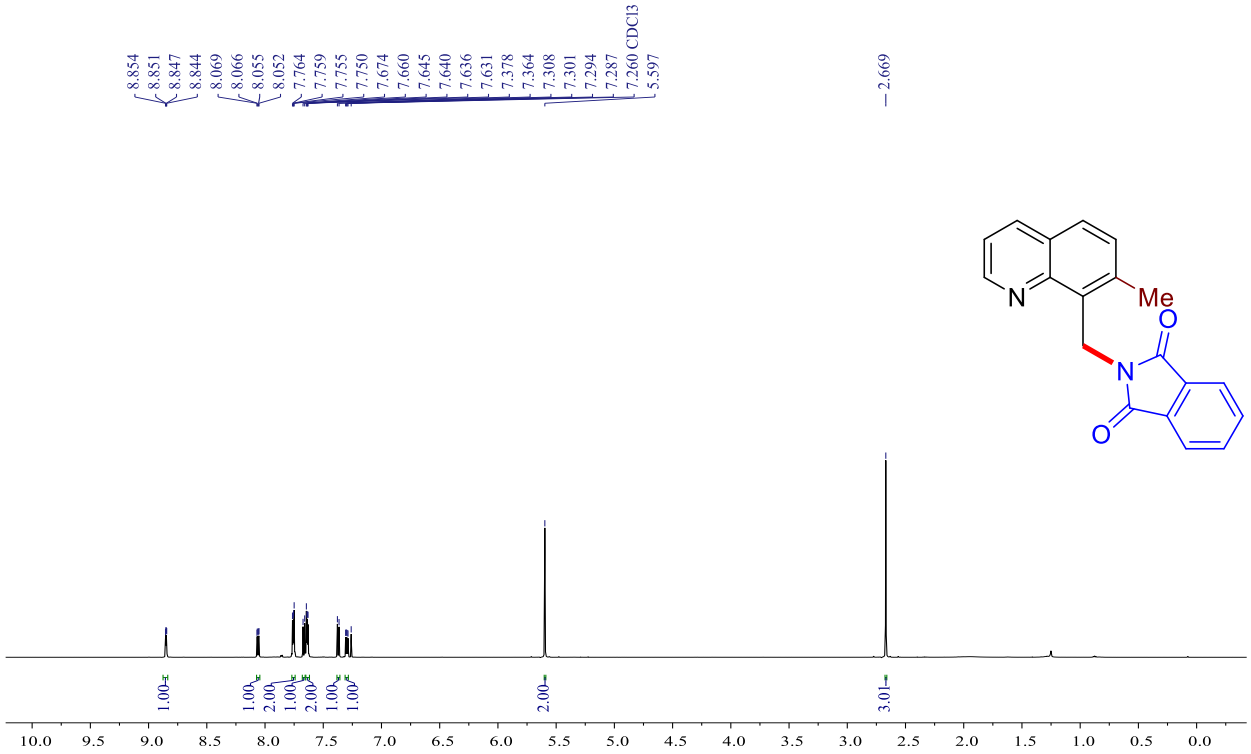


^{13}C{^{1}H} NMR *(*150 MHz*)*

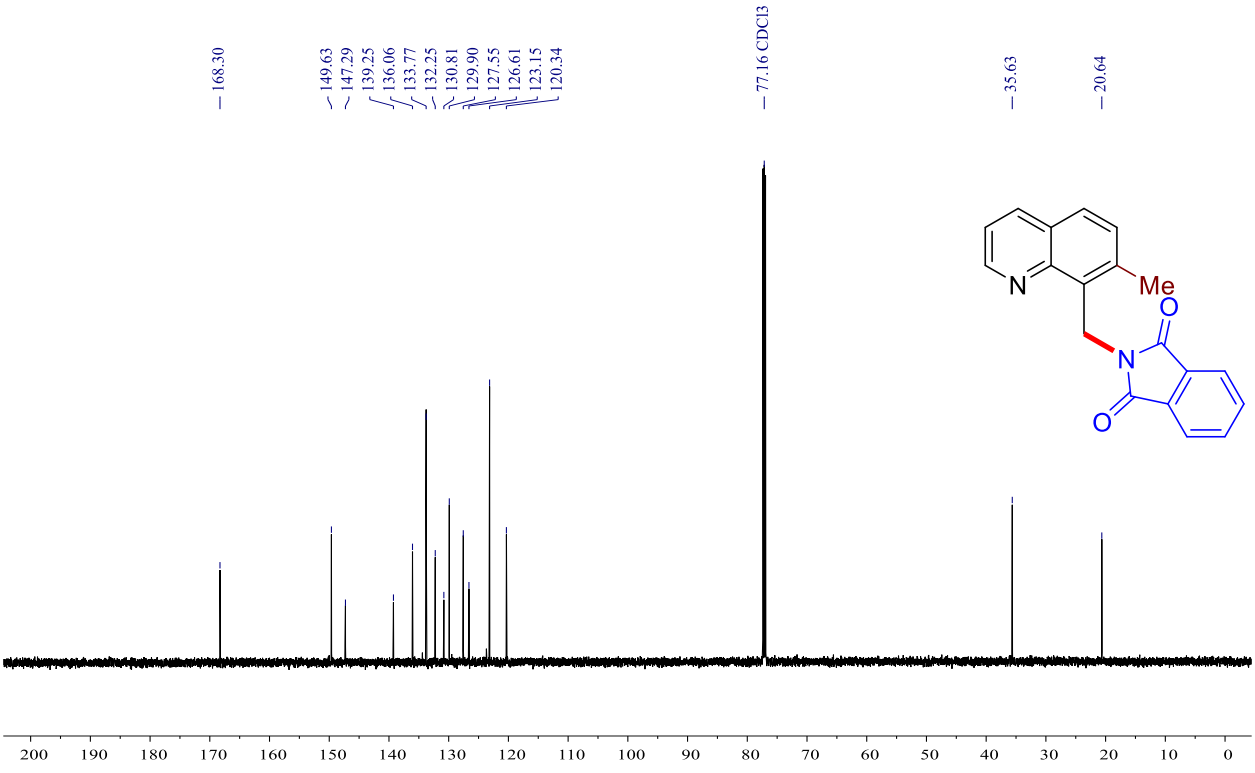


*2-((7-methylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3f**)*

^{1}H NMR (600 MHz)

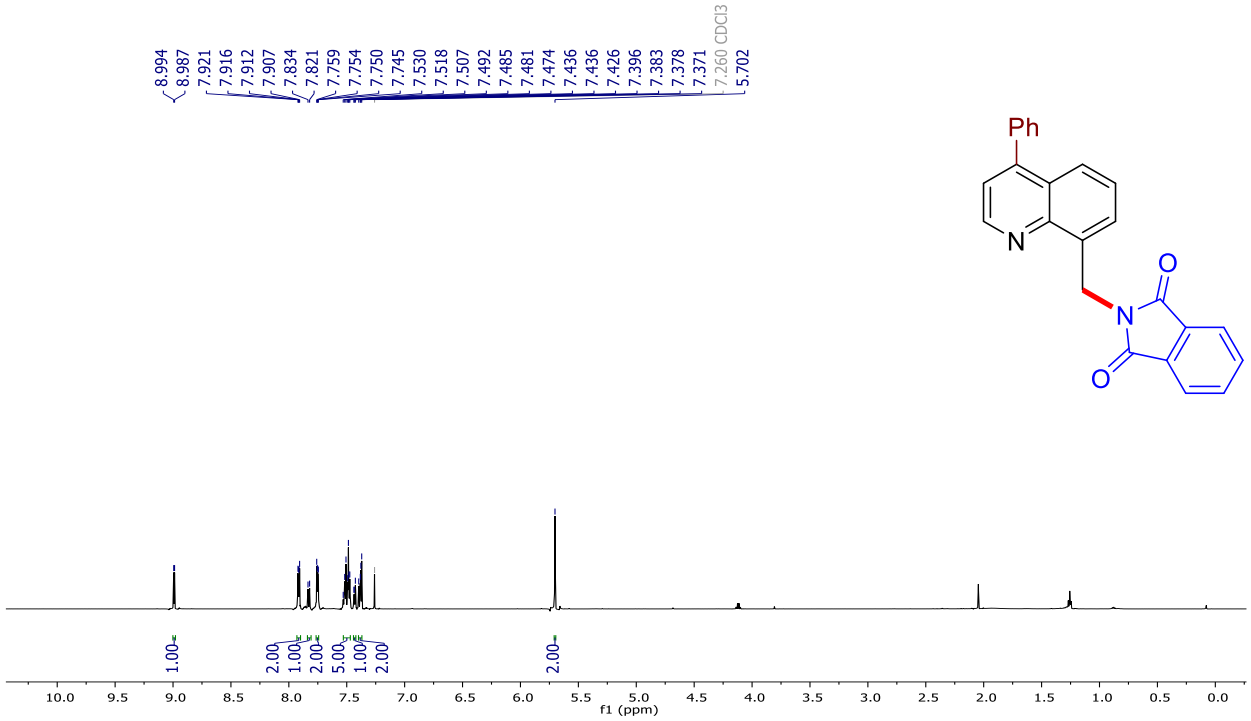


^{13}C{^{1}H} NMR *(*150 MHz*)*

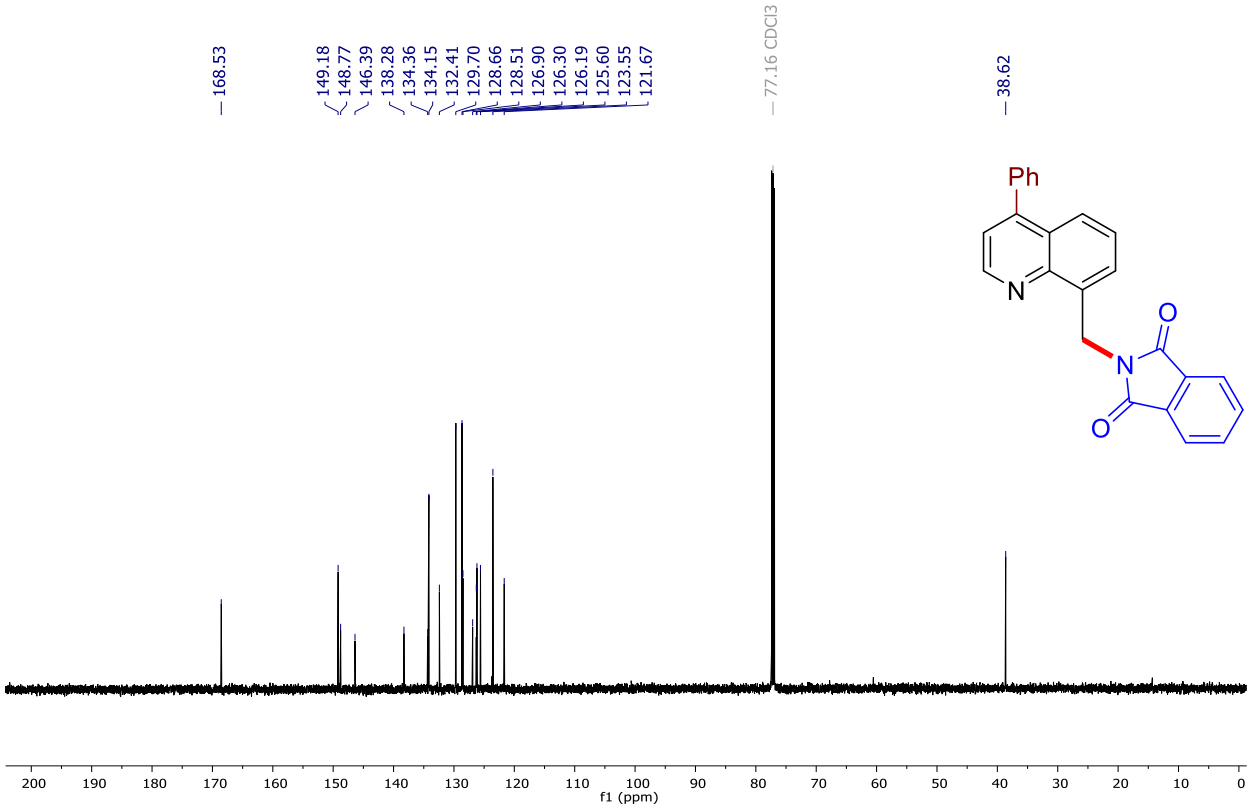


*2-((4-phenylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3g**)*

^{1}H NMR (600 MHz)

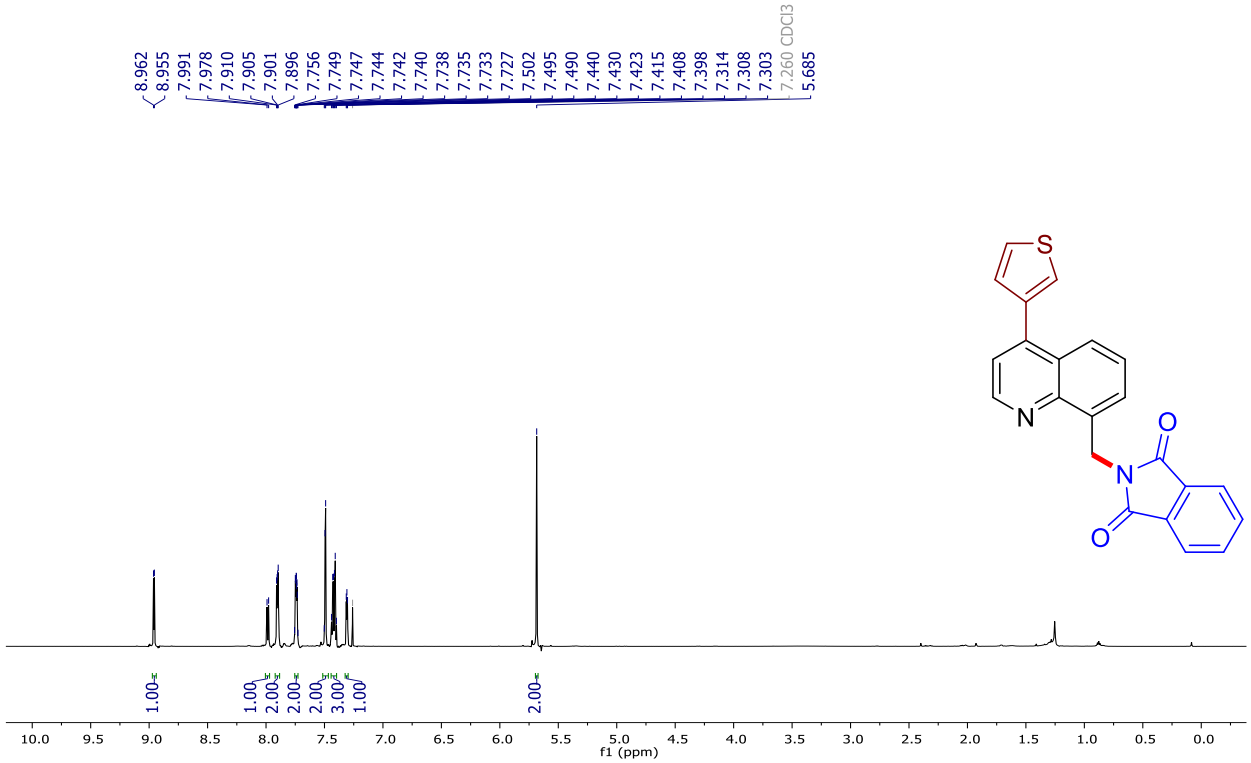


^{13}C{^{1}H} NMR *(*150 MHz*)*

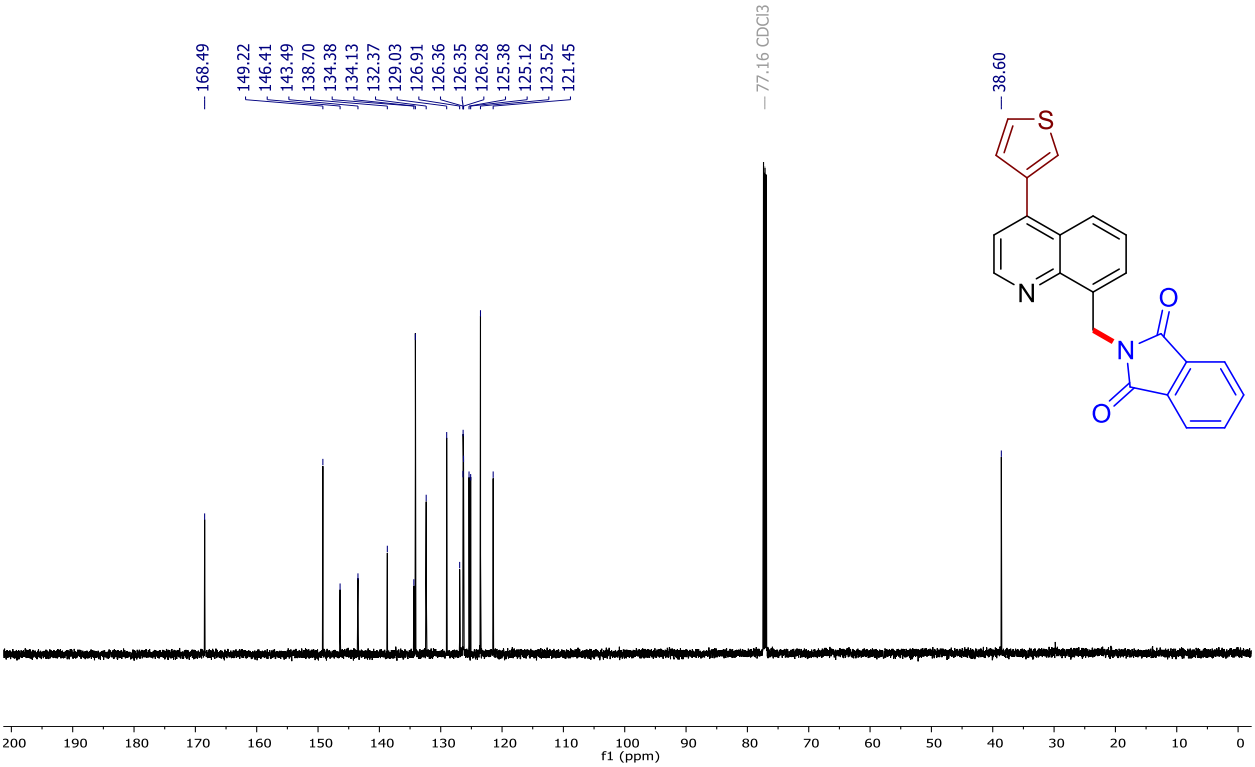


*2-((4-(thiophen-3-yl)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3h**)*

^{1}H NMR (600 MHz)

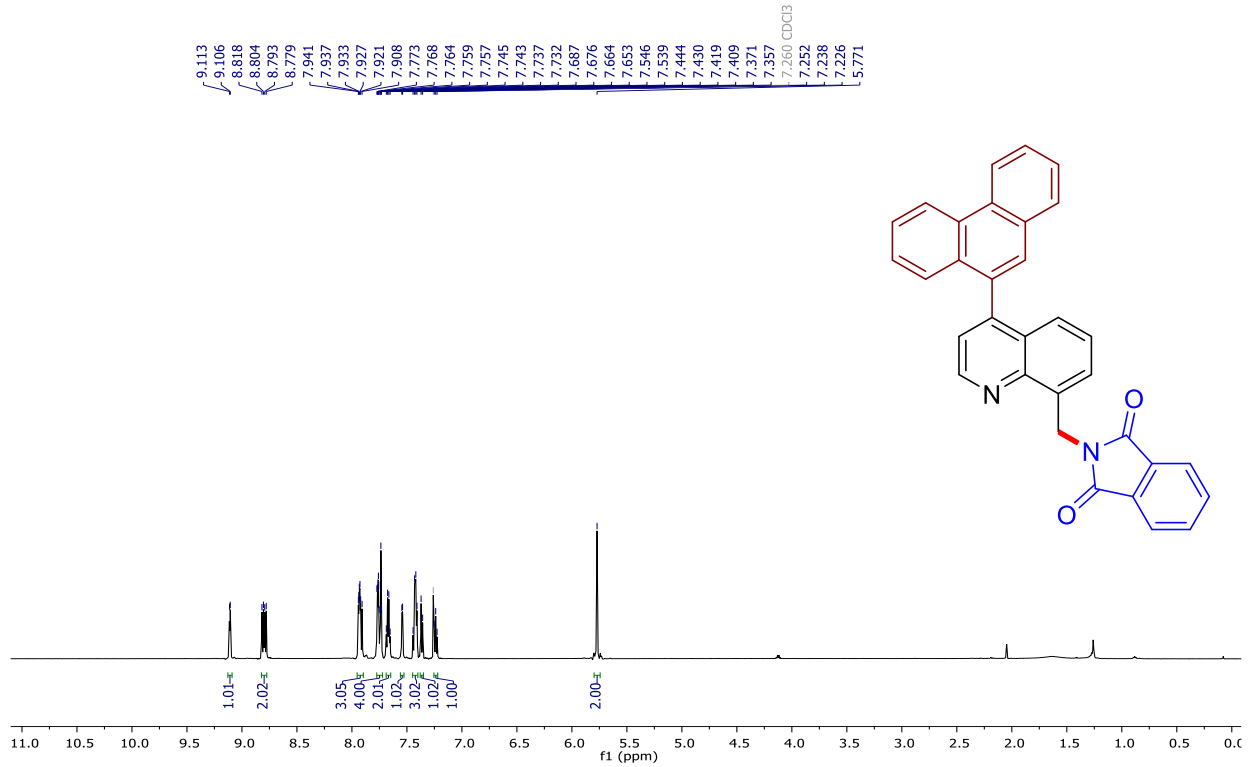


^{13}C{^{1}H} NMR *(*150 MHz*)*

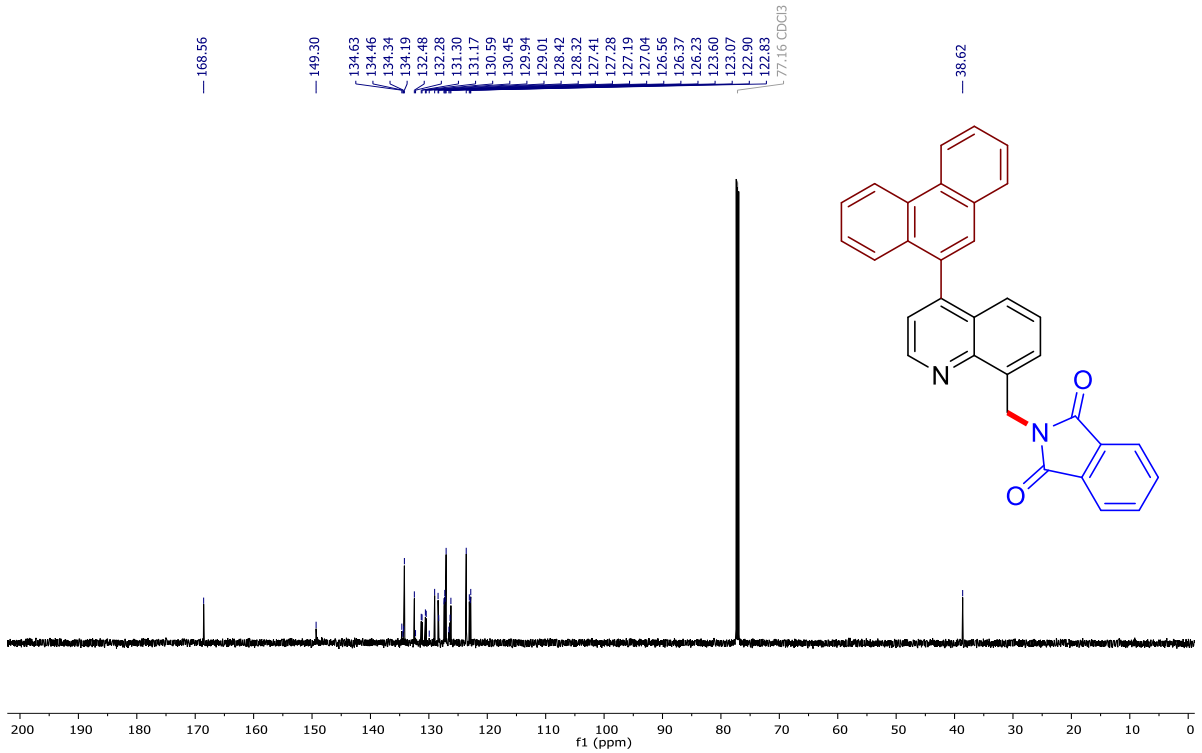


*2-((4-(phenanthren-9-yl)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3i**)*

^{1}H NMR (600 MHz)

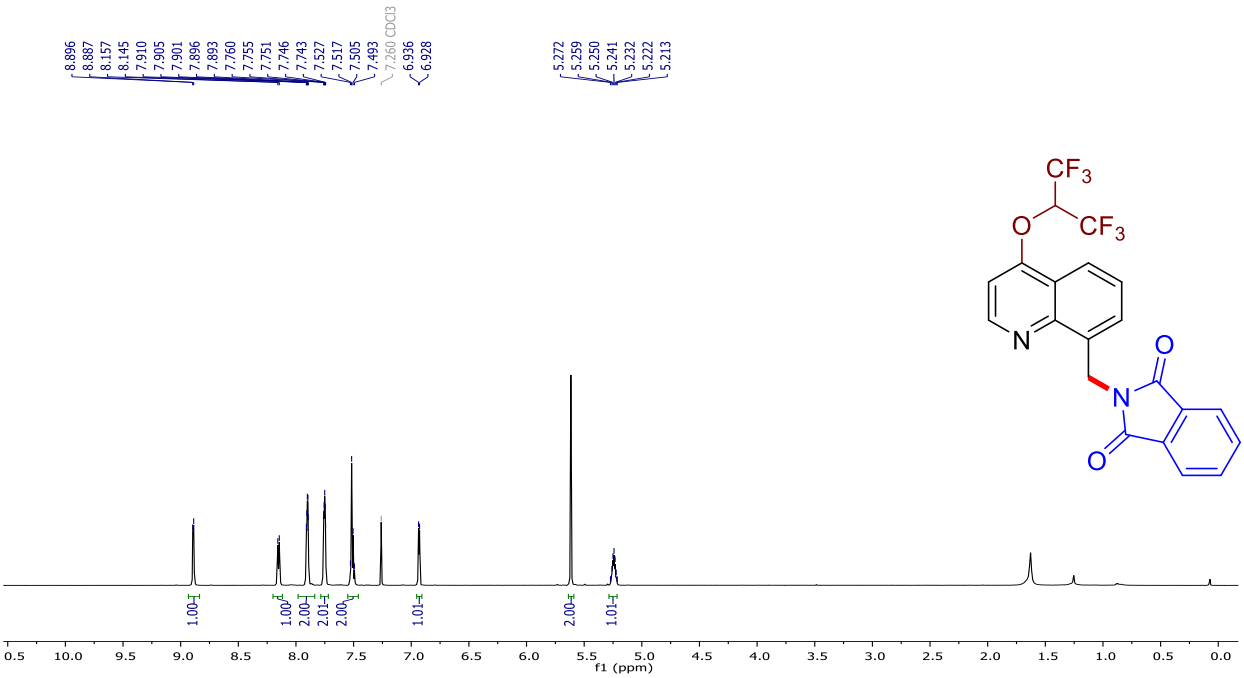


^{13}C{^{1}H} NMR *(*150 MHz*)*

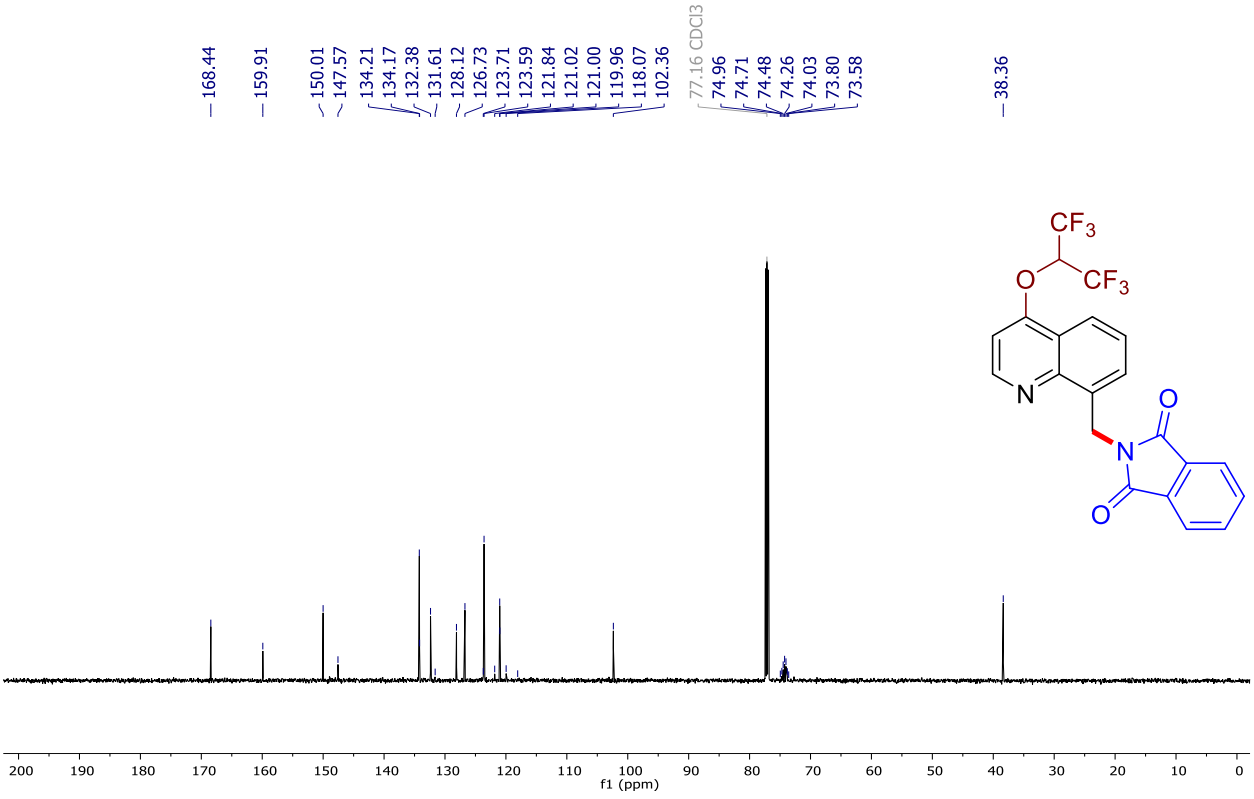


*2-((4-((1,1,1,3,3,3-hexafluoropropan-2-yl)oxy)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3j**)*

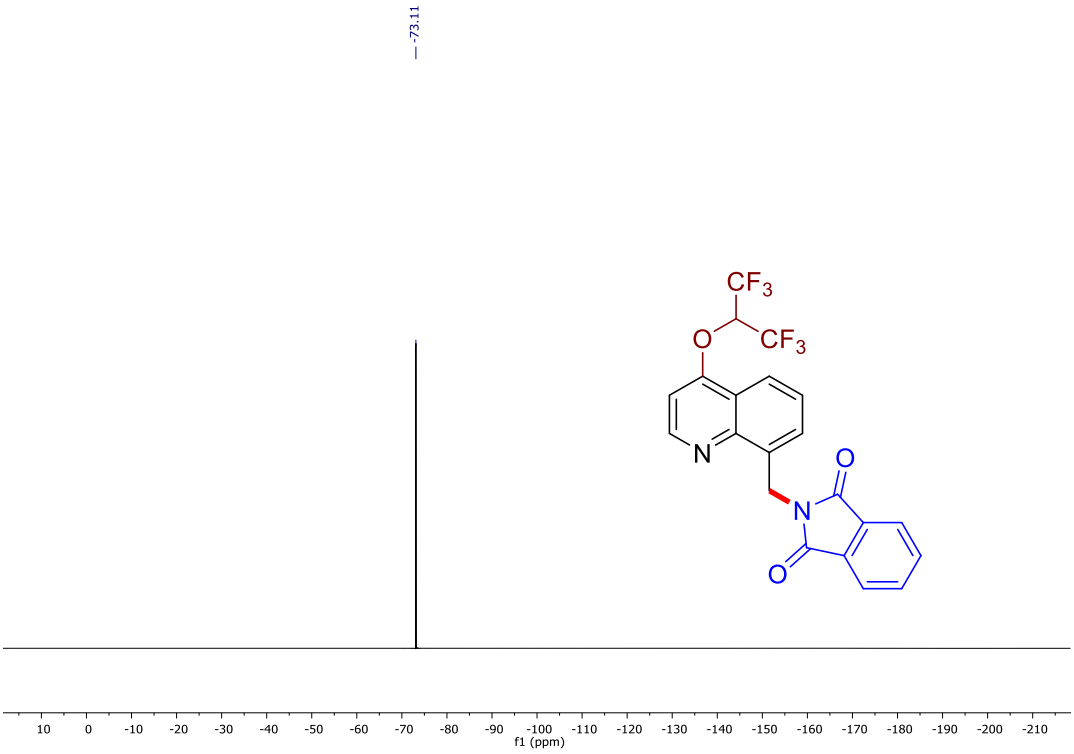
^{1}H NMR (600 MHz)



^{13}C{^{1}H} NMR *(*150 MHz*)*

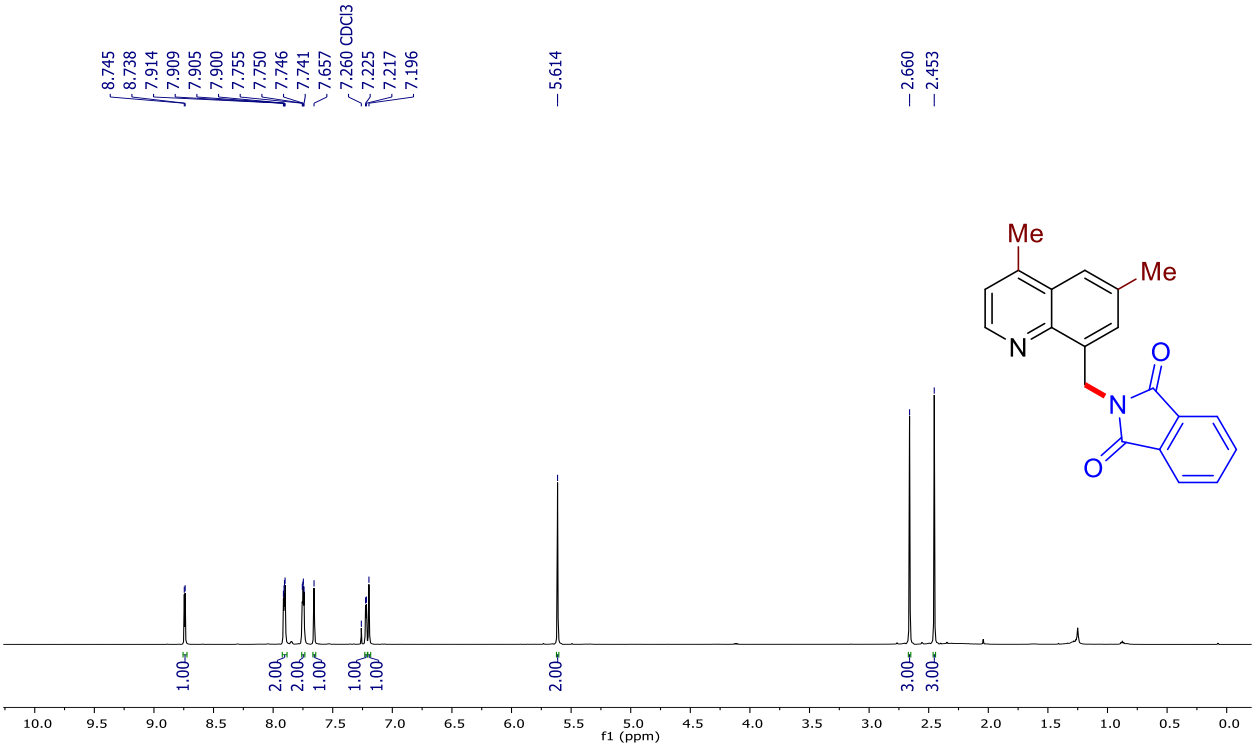


^{19}F NMR (565 MHz)

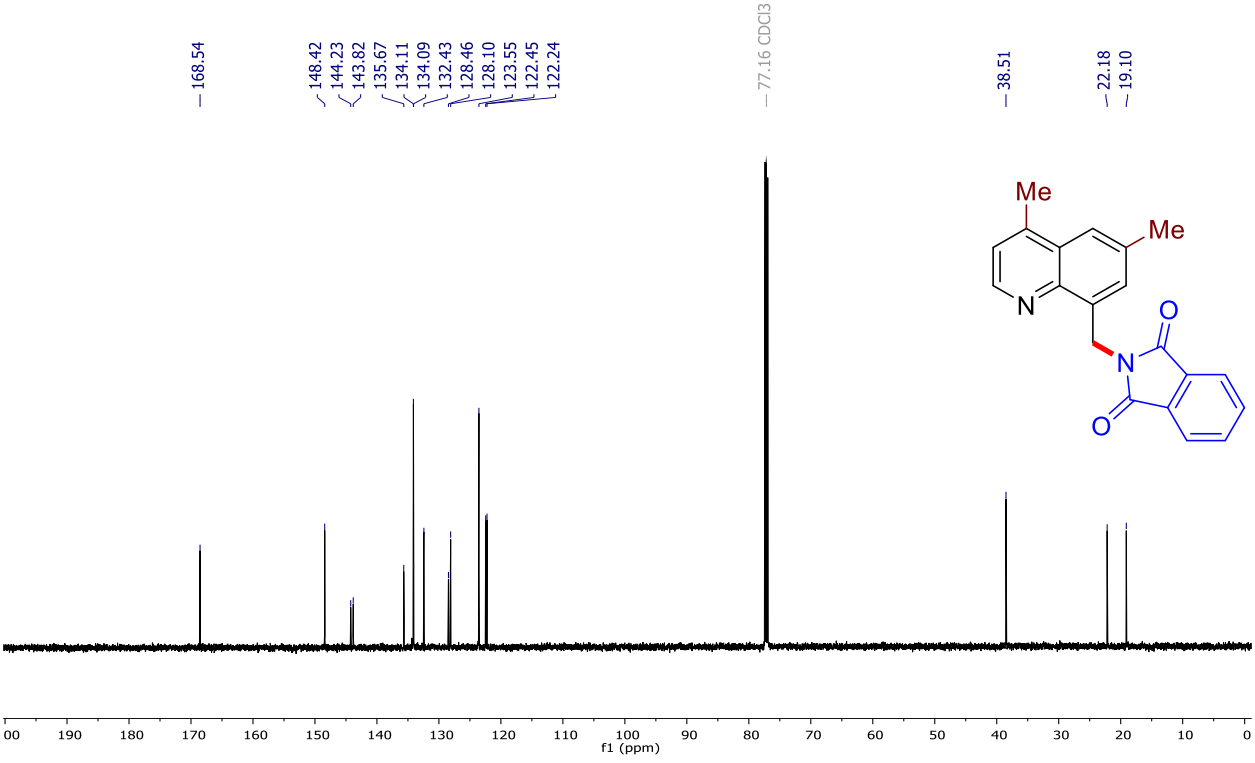


*2-((4,6-dimethylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3k**)*

^{1}H NMR (600 MHz)

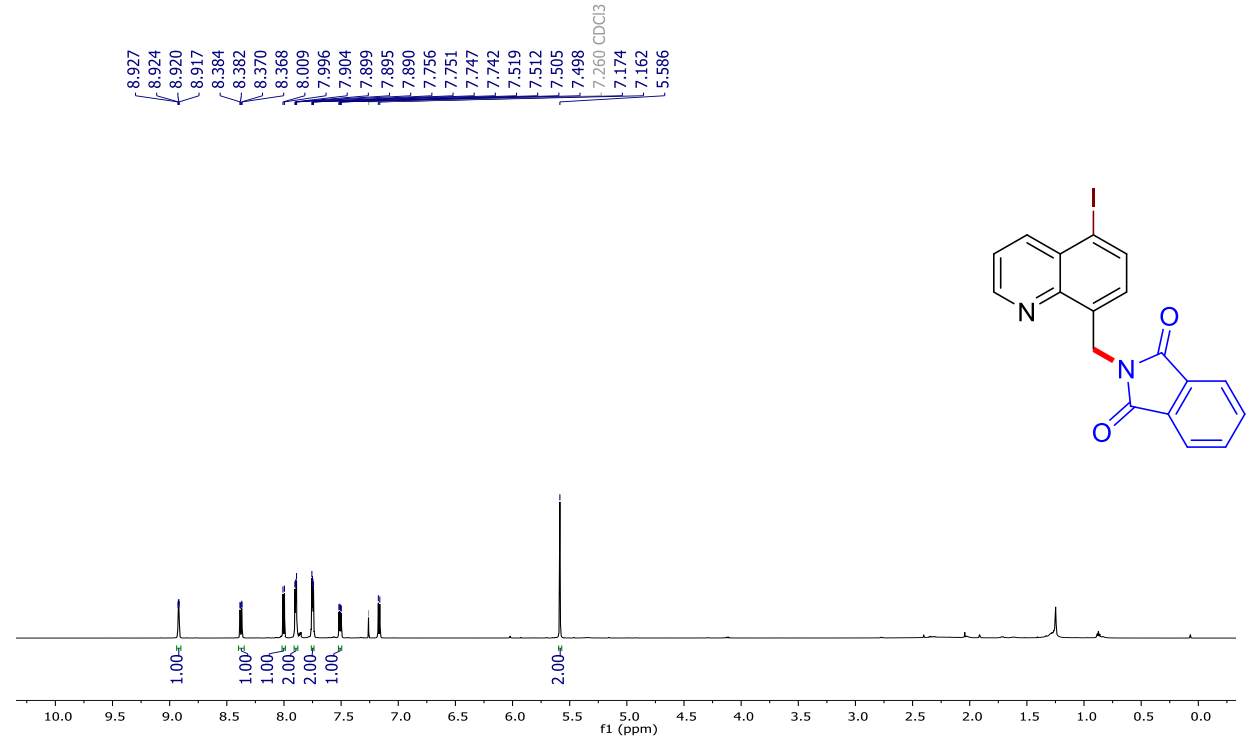


^{13}C{^{1}H} NMR *(*150 MHz*)*

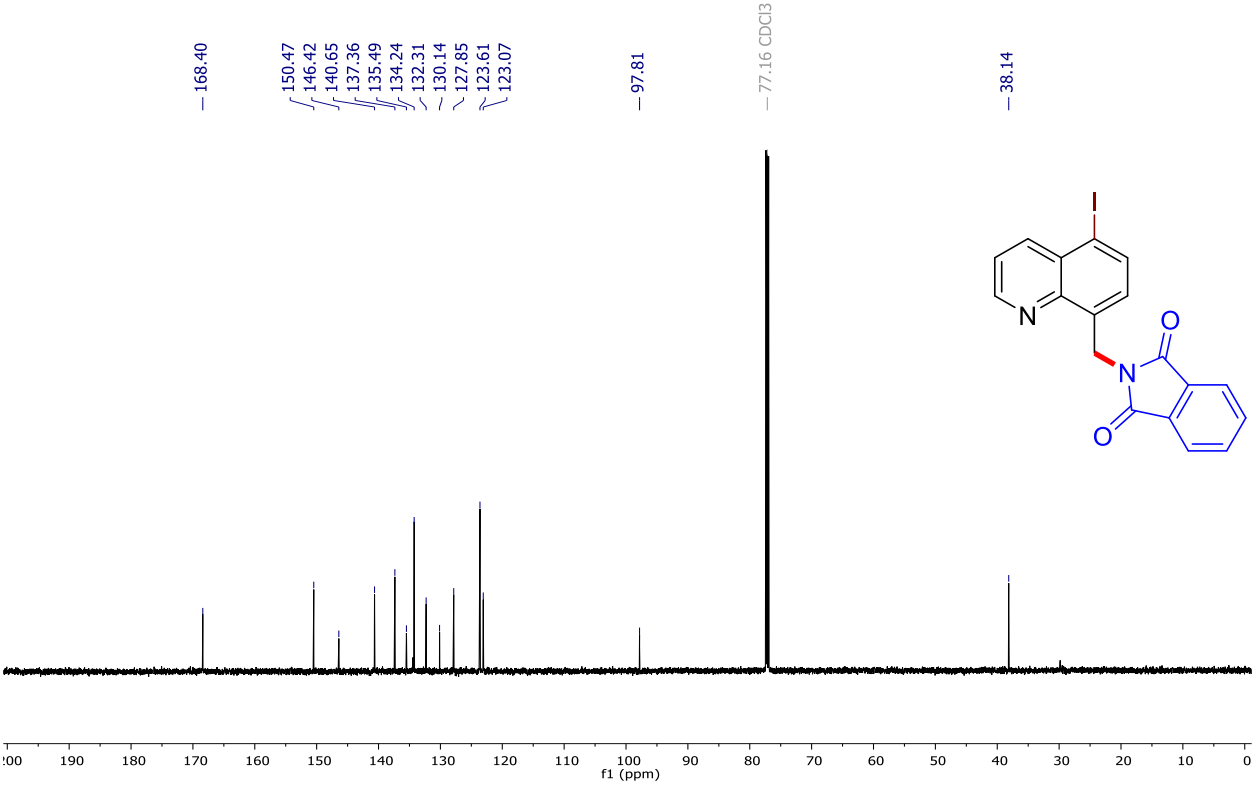


*2-((5-iodooquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3l**)*

^{1}H NMR (600 MHz)

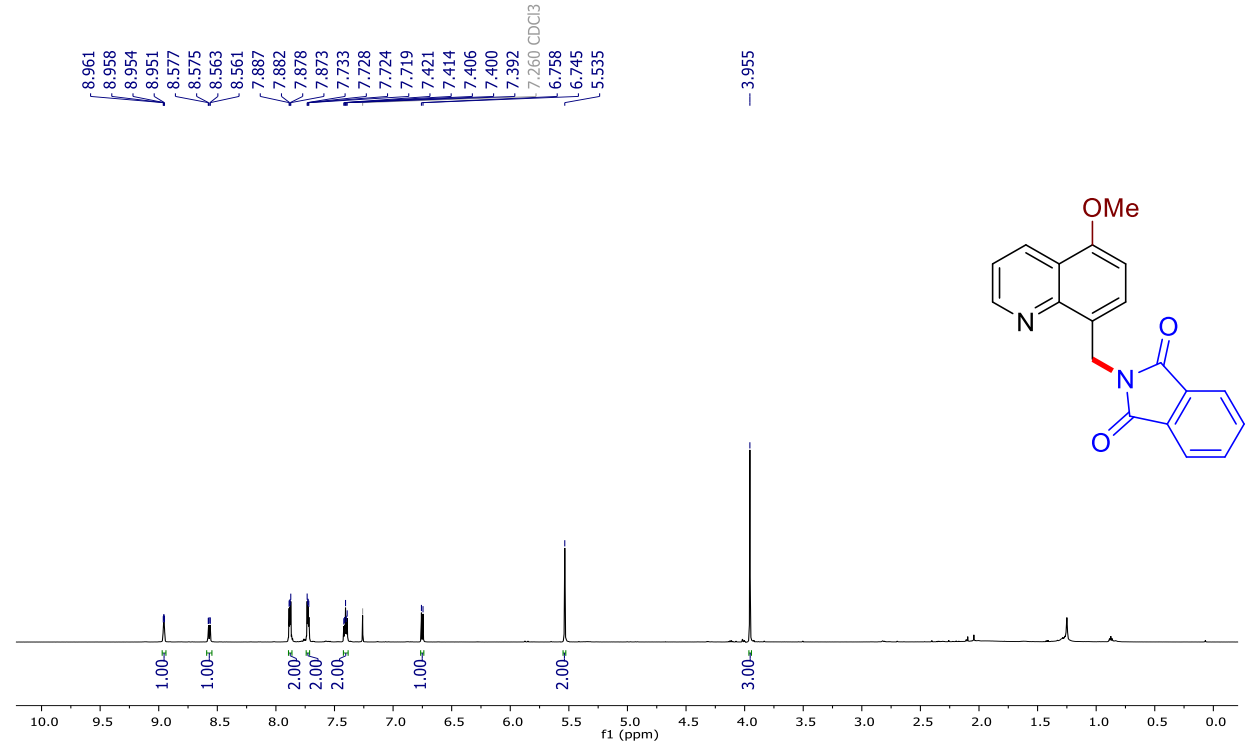


^{13}C{^{1}H} NMR *(*150 MHz*)*

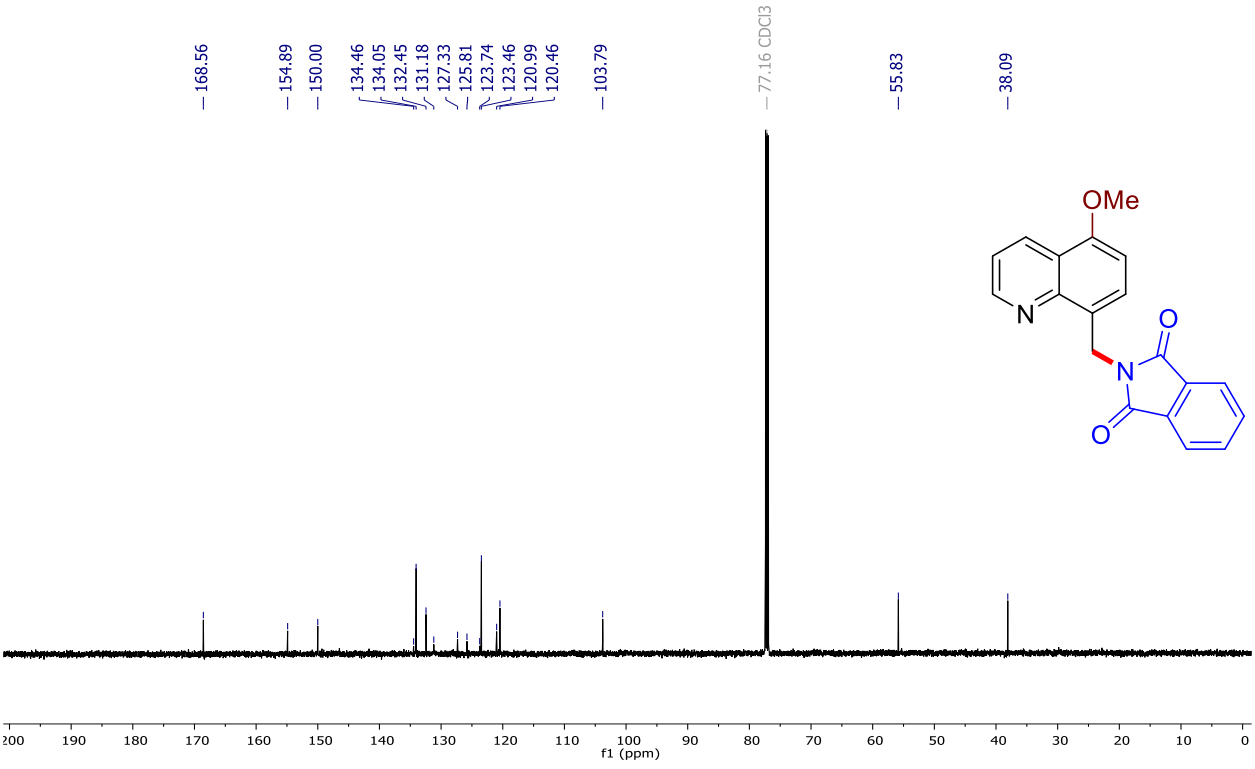


*2-((5-methoxyquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3m**)*

^{1}H NMR (600 MHz)

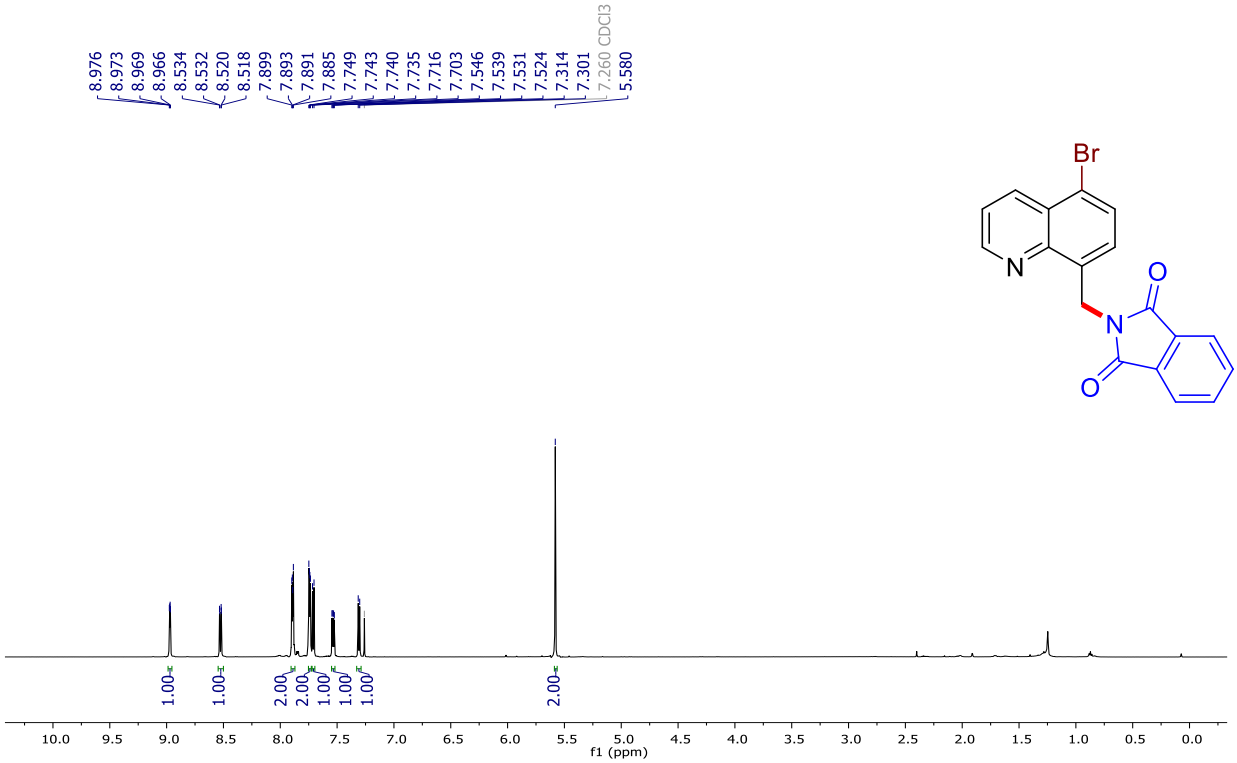


^{13}C{^{1}H} NMR *(*150 MHz*)*

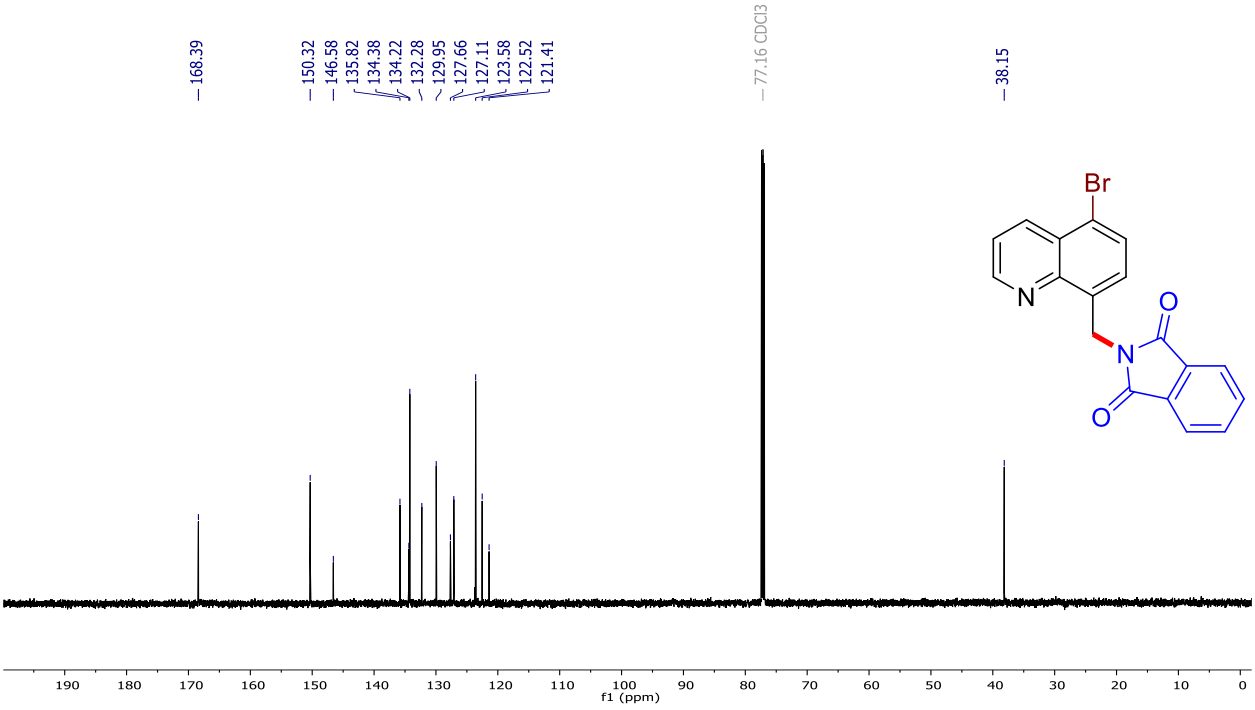


*2-((7-bromoquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3n**)*

^{1}H NMR (600 MHz)

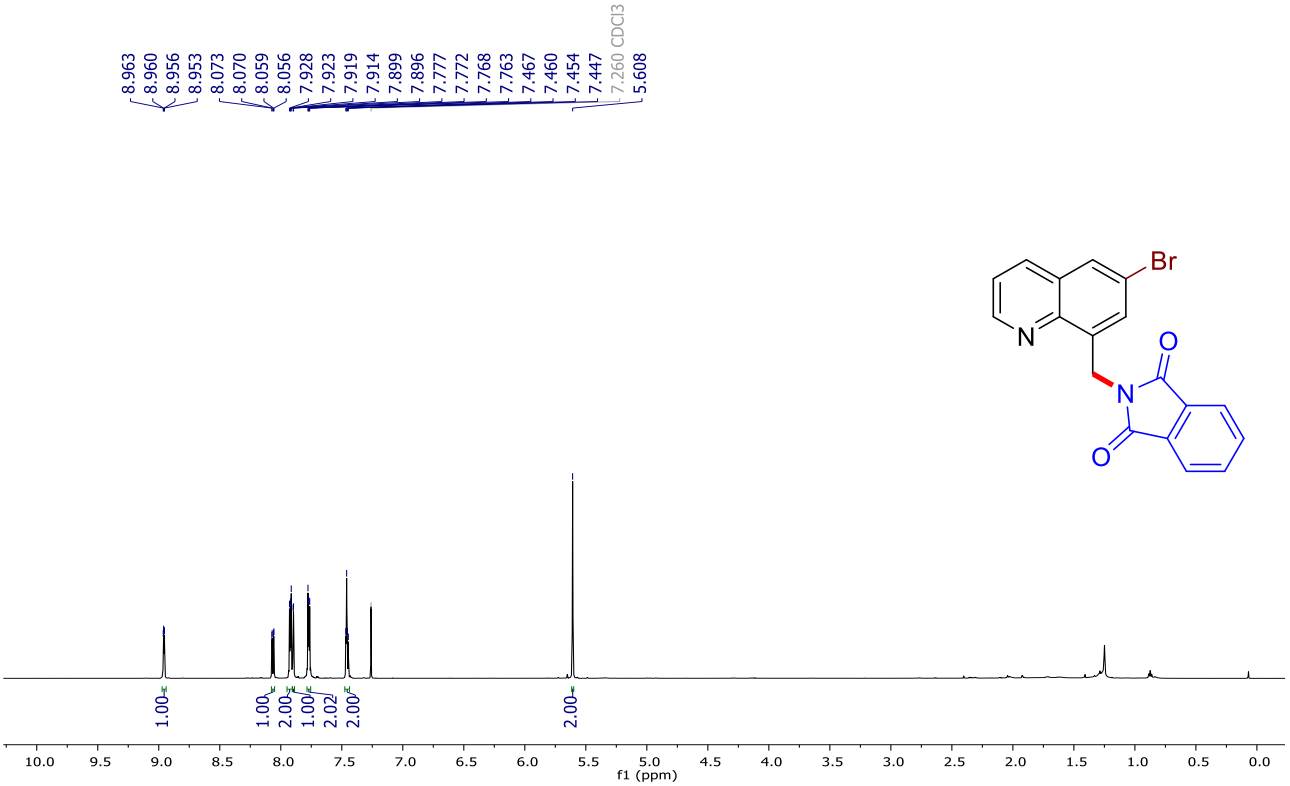


^{13}C{^{1}H} NMR *(*150 MHz*)*

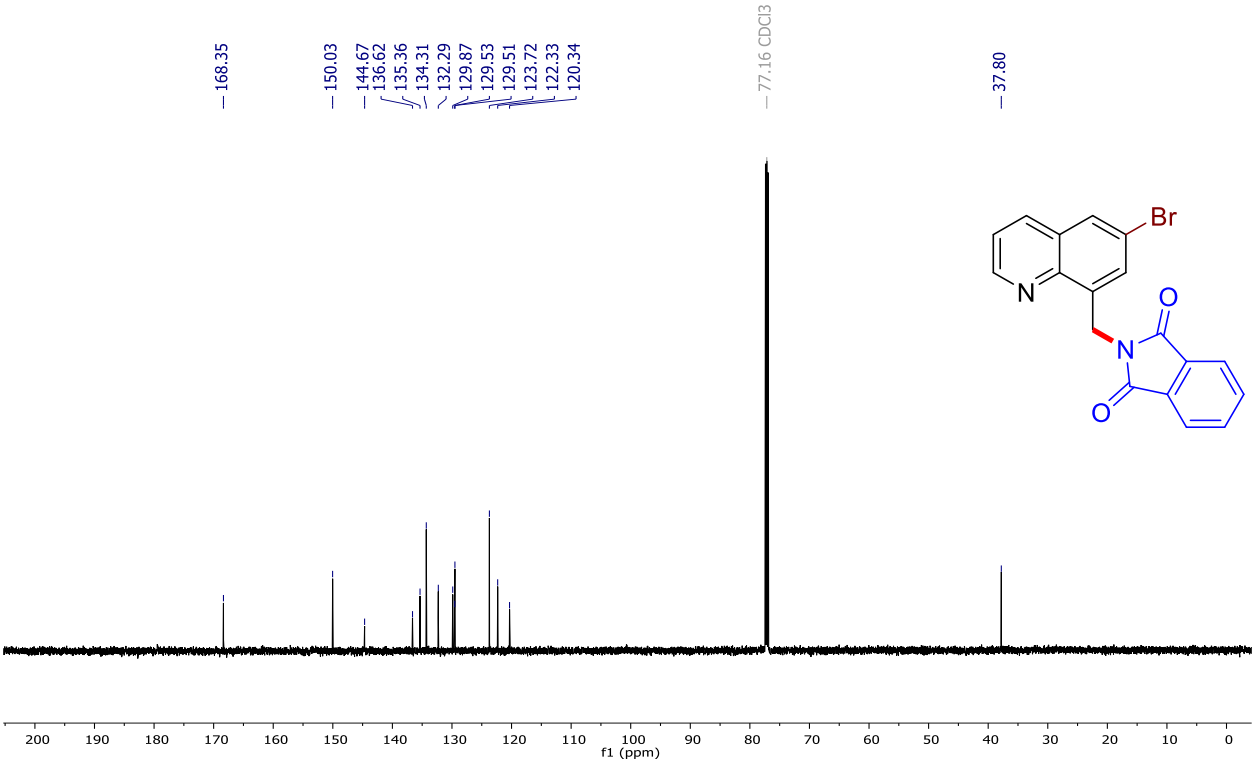


*2-((6-bromoquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3o**)*

^{1}H NMR (600 MHz)

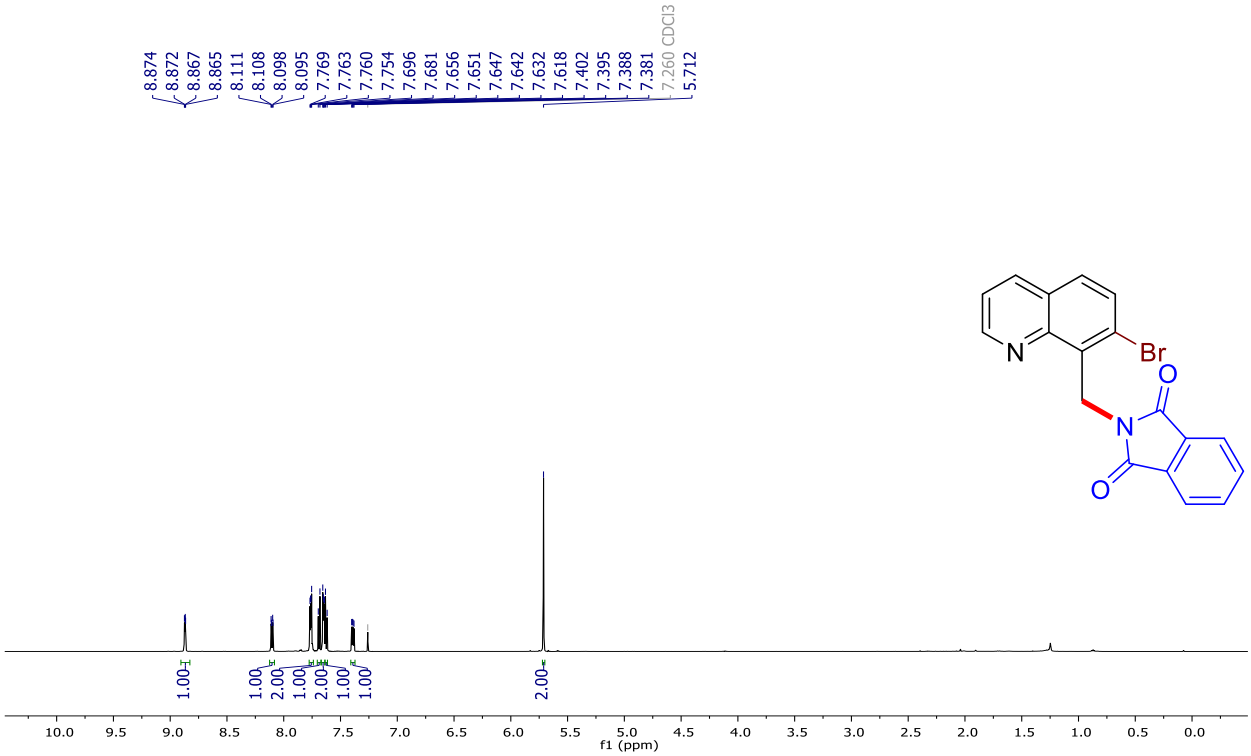


^{13}C{^{1}H} NMR *(*150 MHz*)*

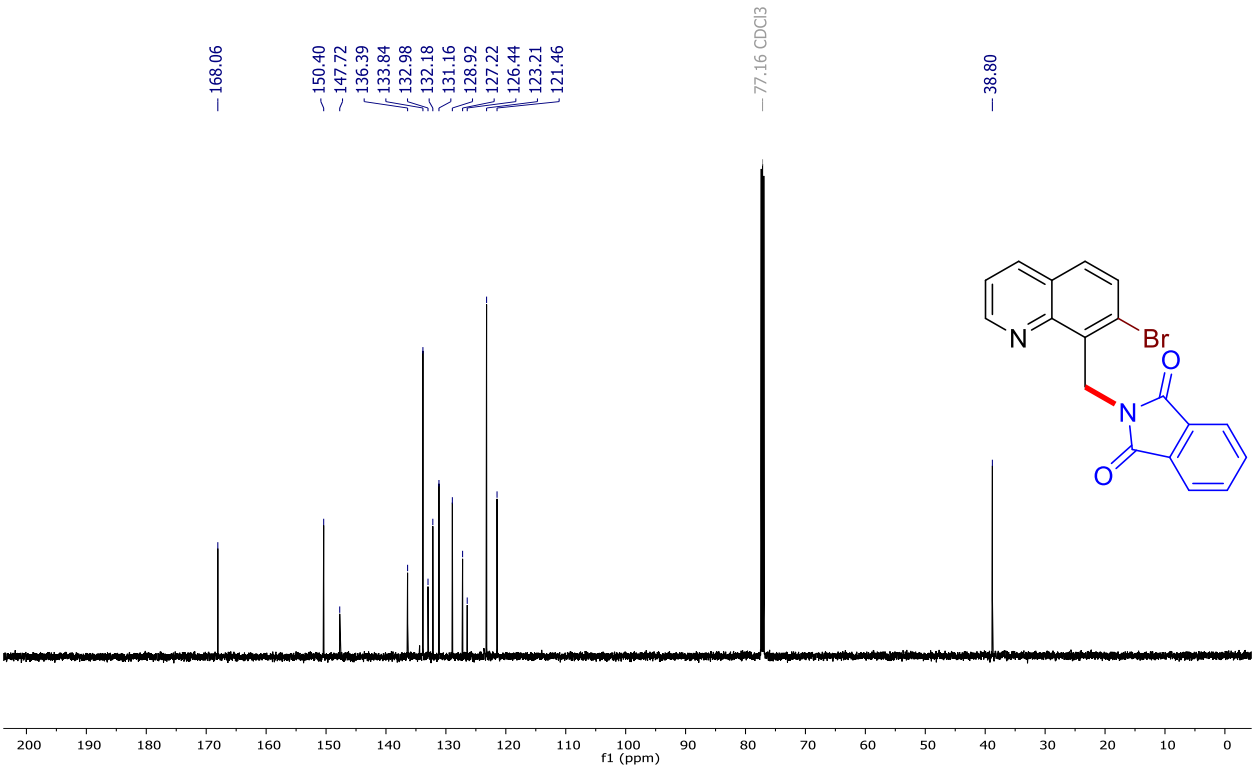


*2-((7-bromoquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3p**)*

^{1}H NMR (600 MHz)

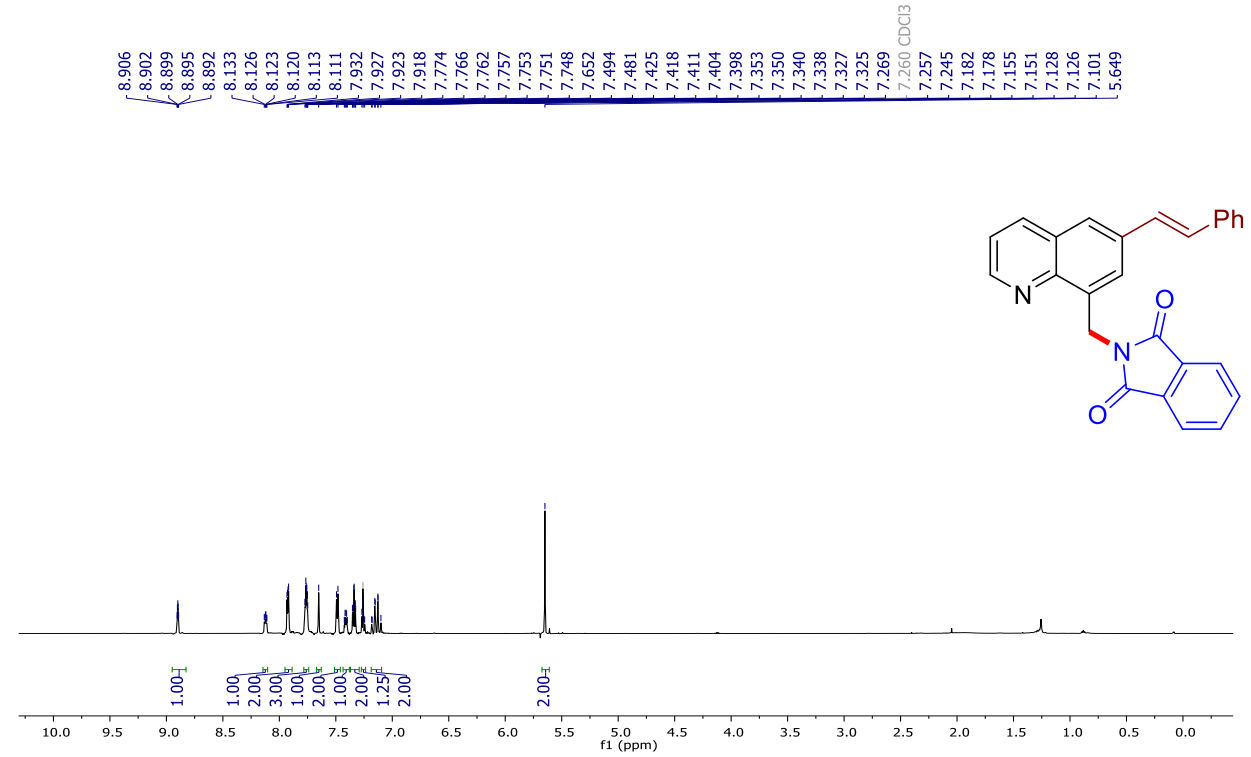


^{13}C{^{1}H} NMR *(*150 MHz*)*

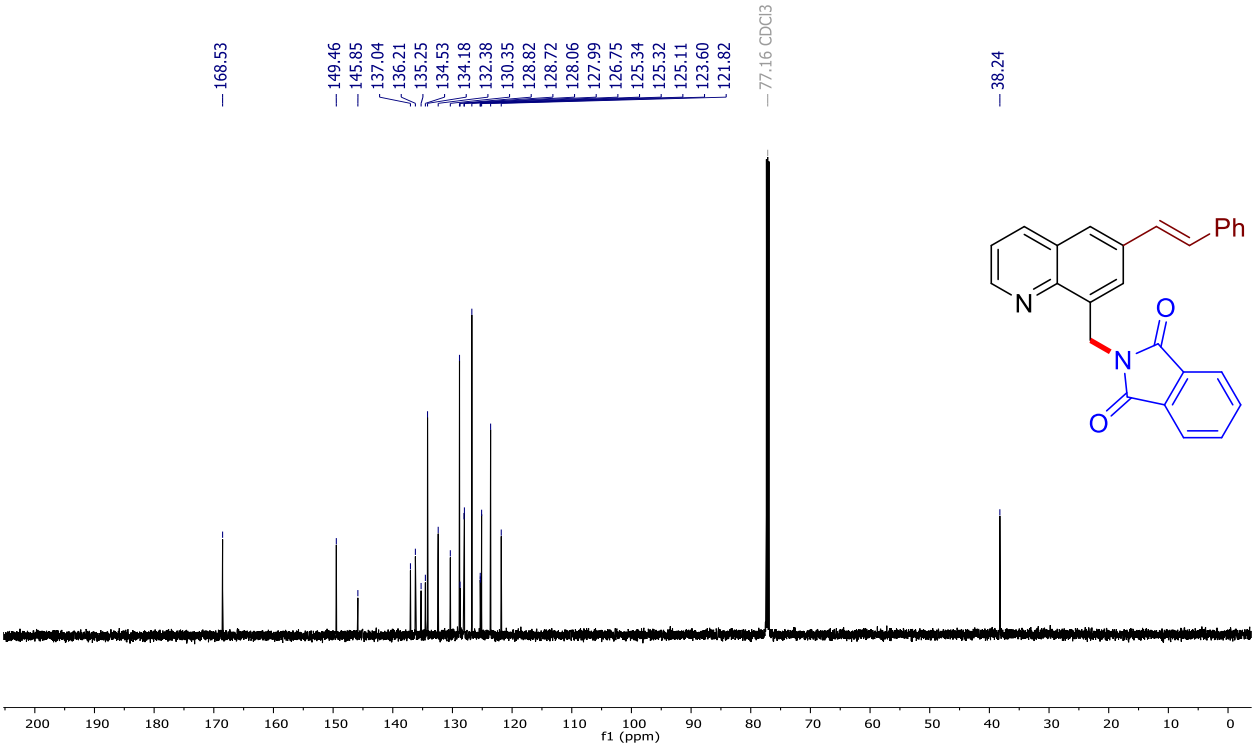


*(E)-2-((6-styrylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3q**)*

^{1}H NMR (600 MHz)

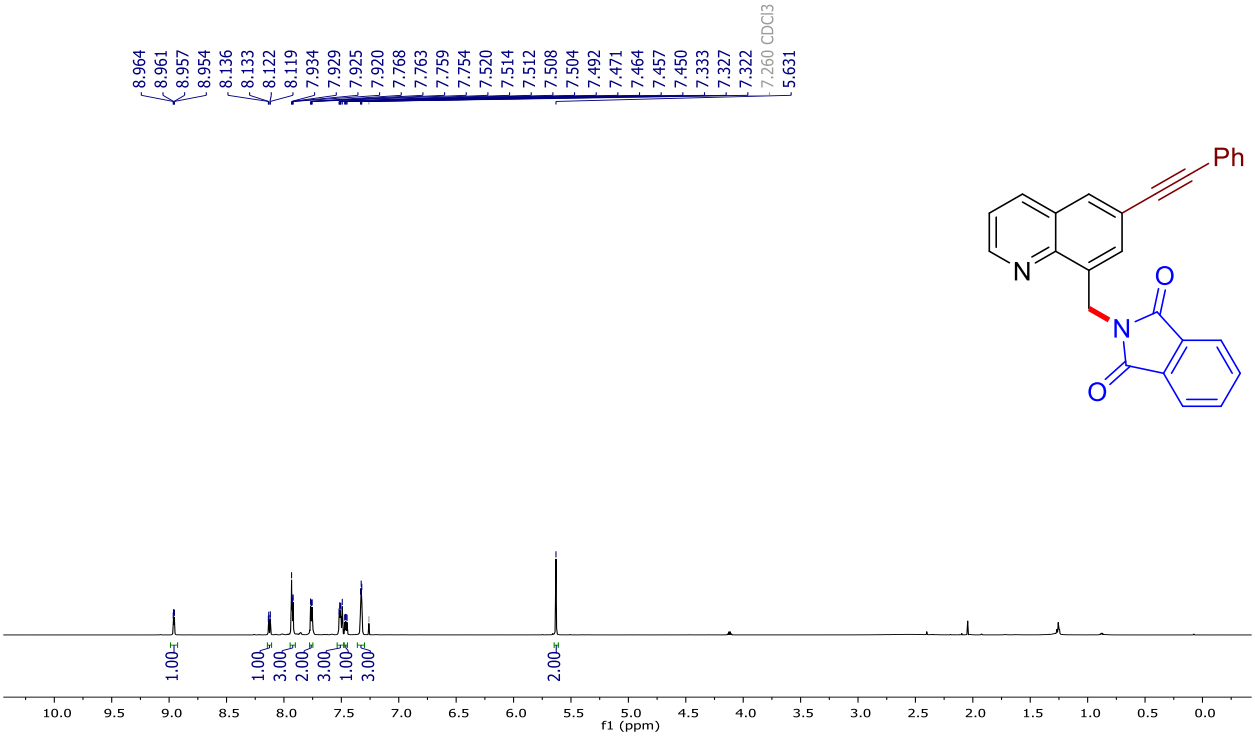


^{13}C{^{1}H} NMR *(*150 MHz*)*

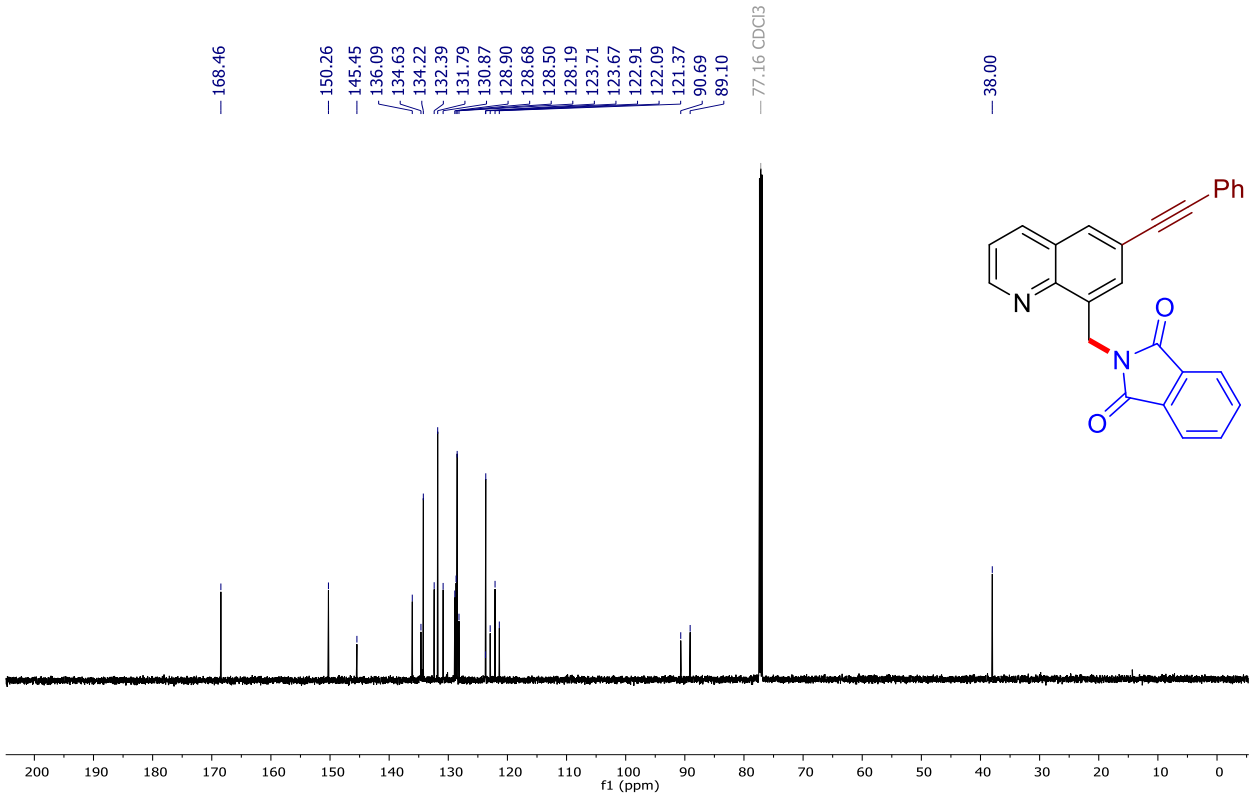


*2-((6-(phenylethynyl)quinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3r**)*

^{1}H NMR (600 MHz)

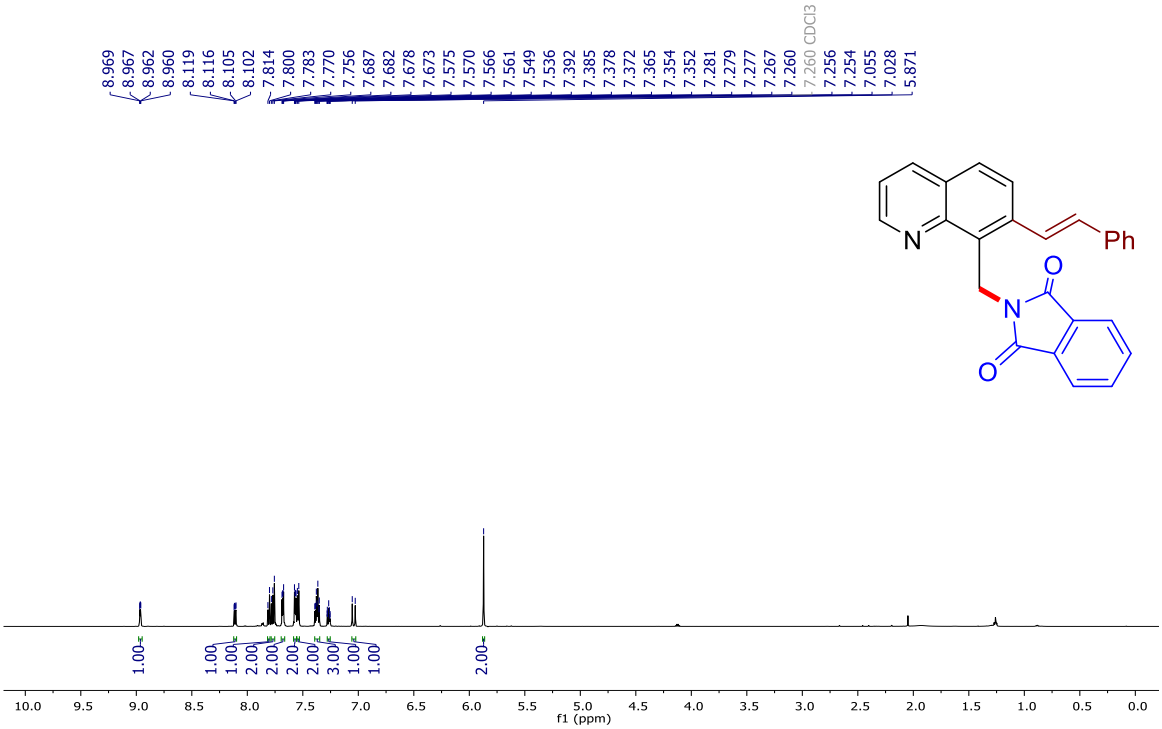


^{13}C{^{1}H} NMR *(*150 MHz*)*

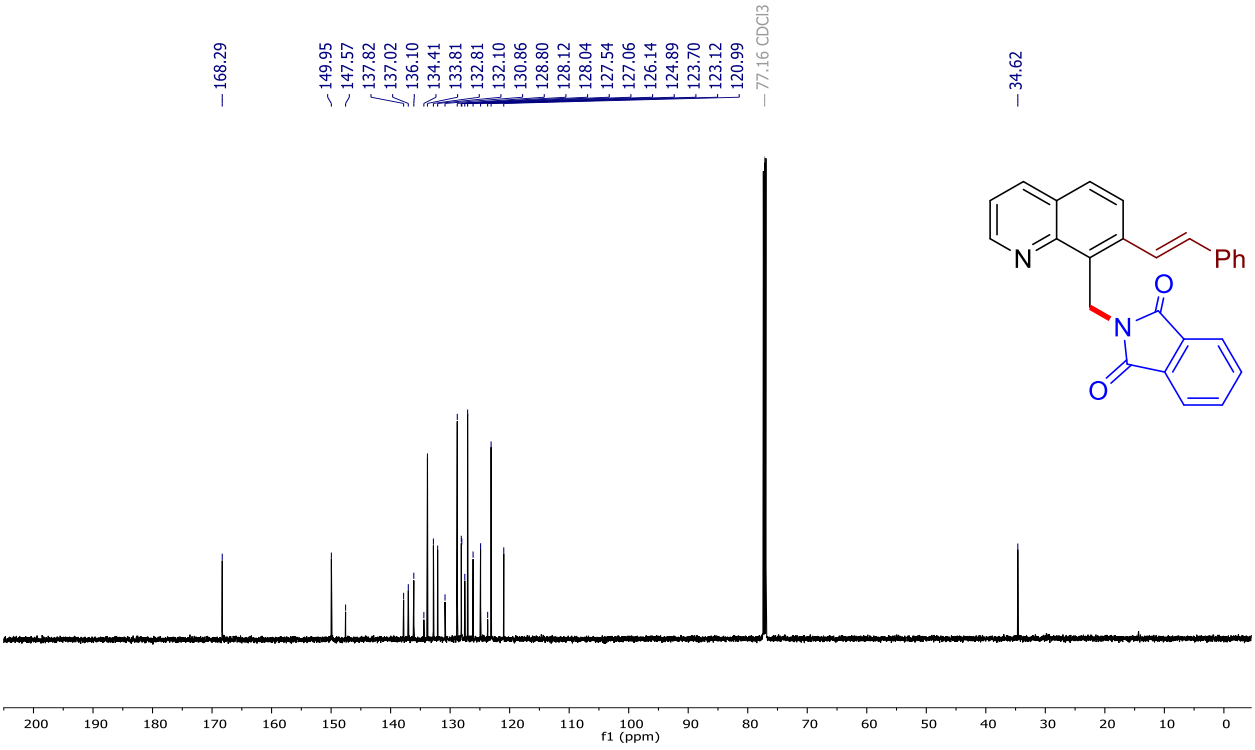


*(E)-2-((7-styrylquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3s**)*

^{1}H NMR (600 MHz)

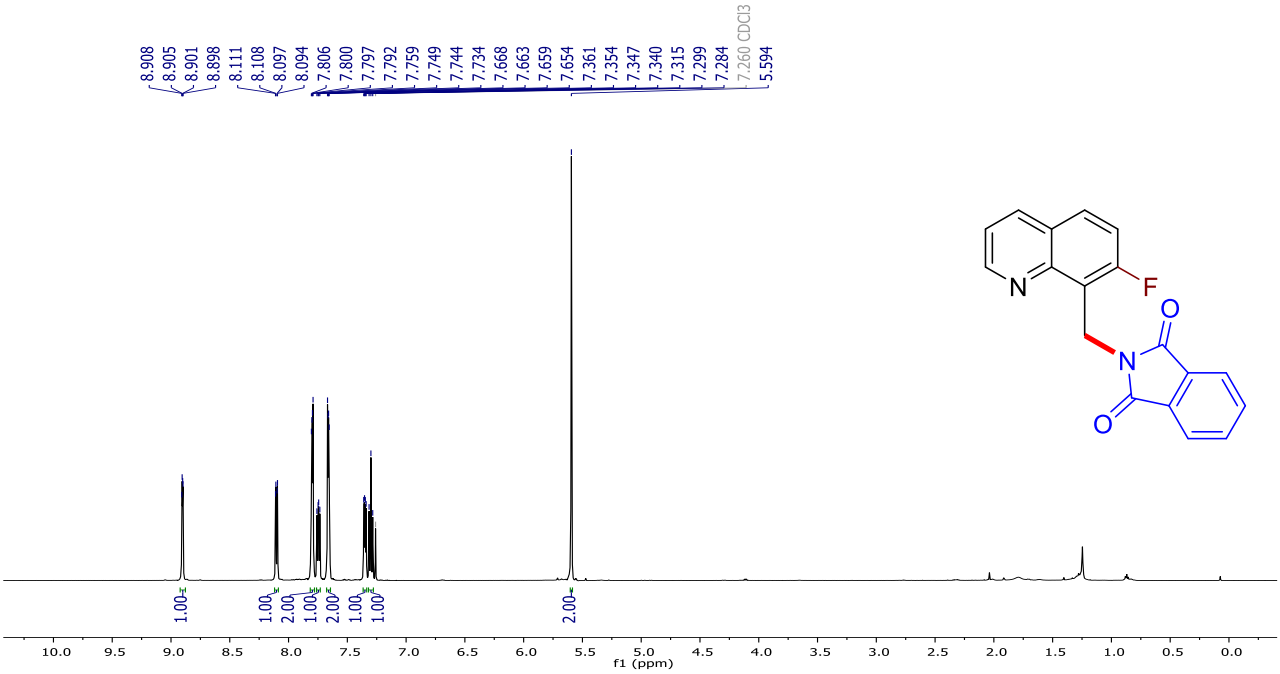


^{13}C{^{1}H} NMR *(*150 MHz*)*

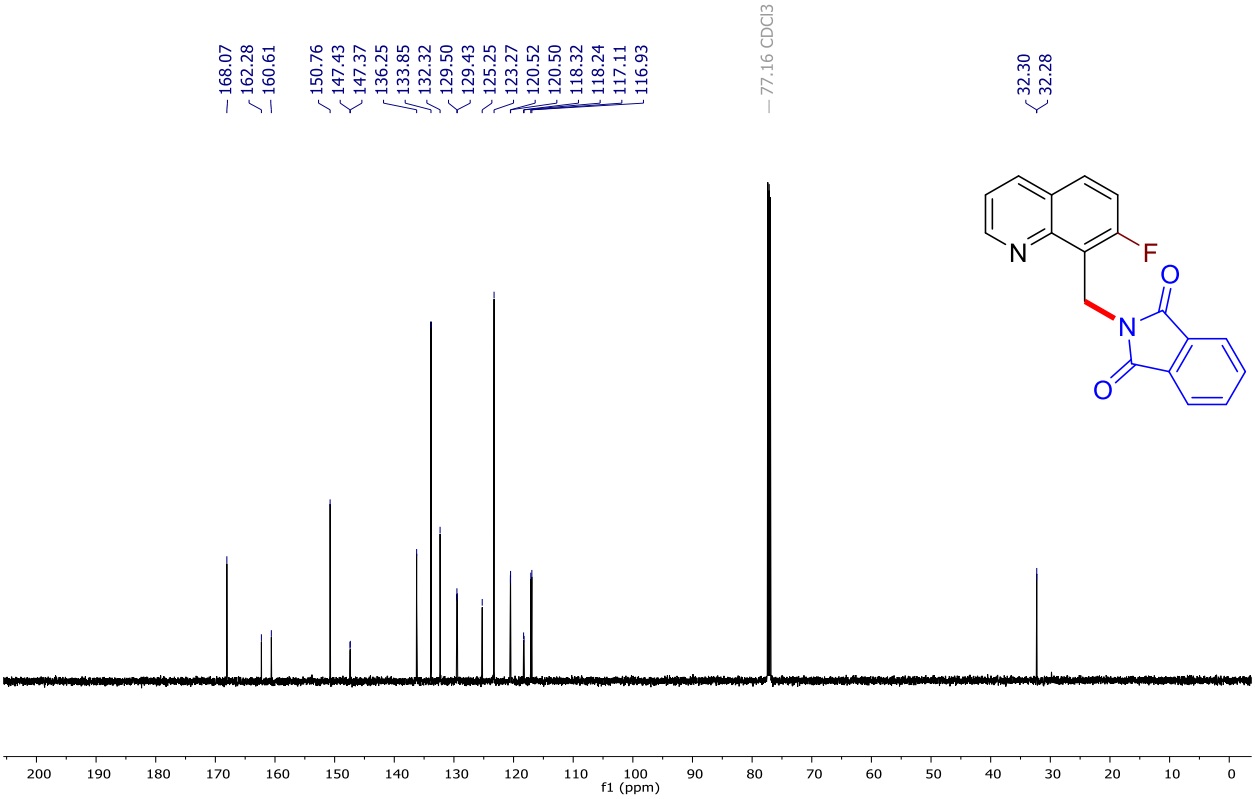


*2-((7-fluoroquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3t**)*

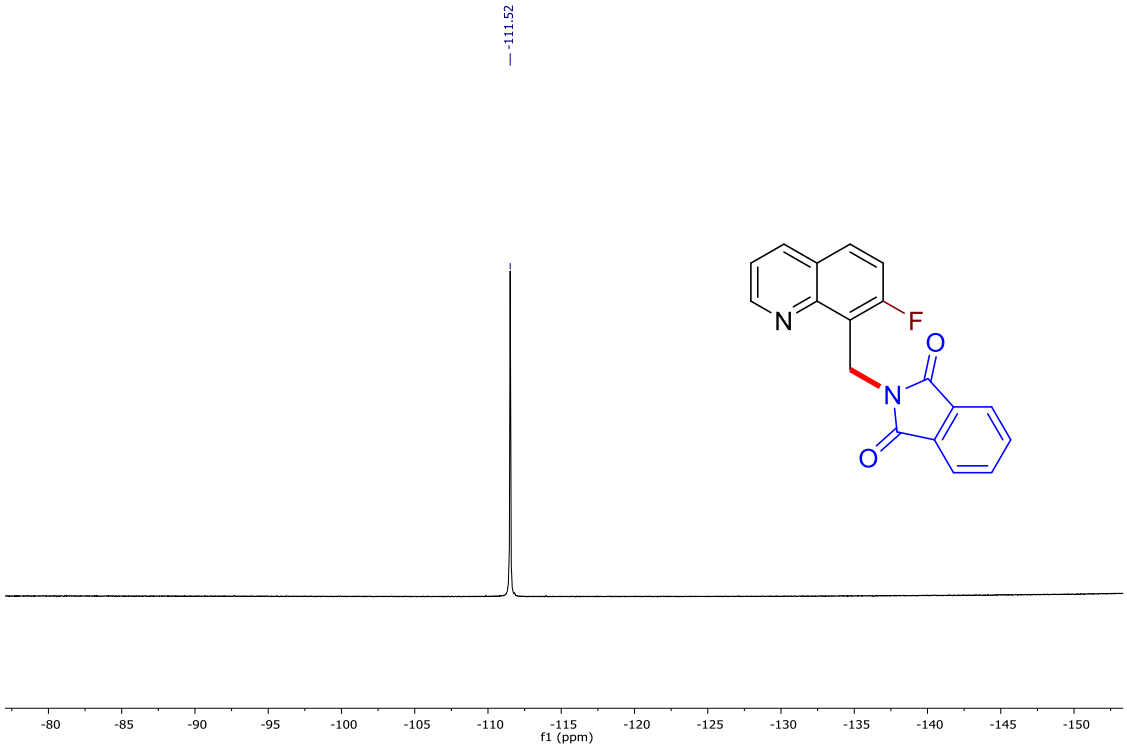
^{1}H NMR (600 MHz)



^{13}C{^{1}H} NMR *(*150 MHz*)*

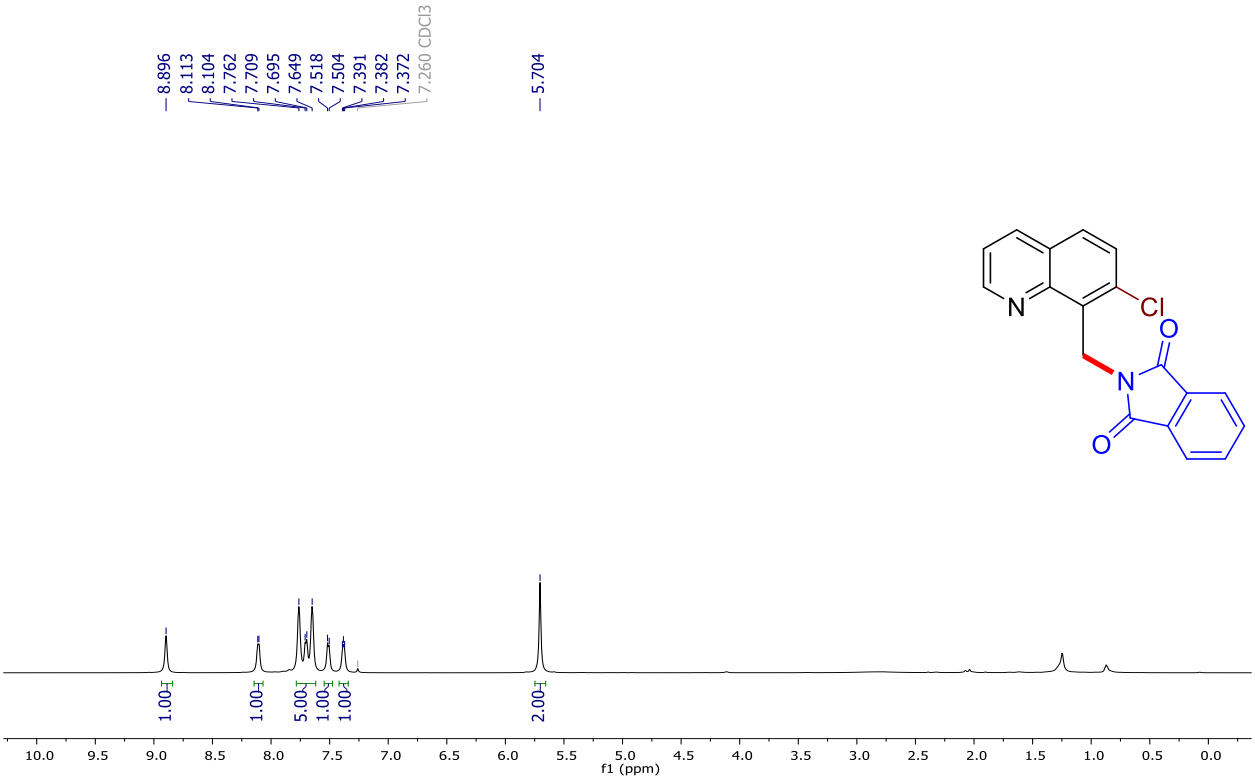


^{19}F NMR (565 MHz)

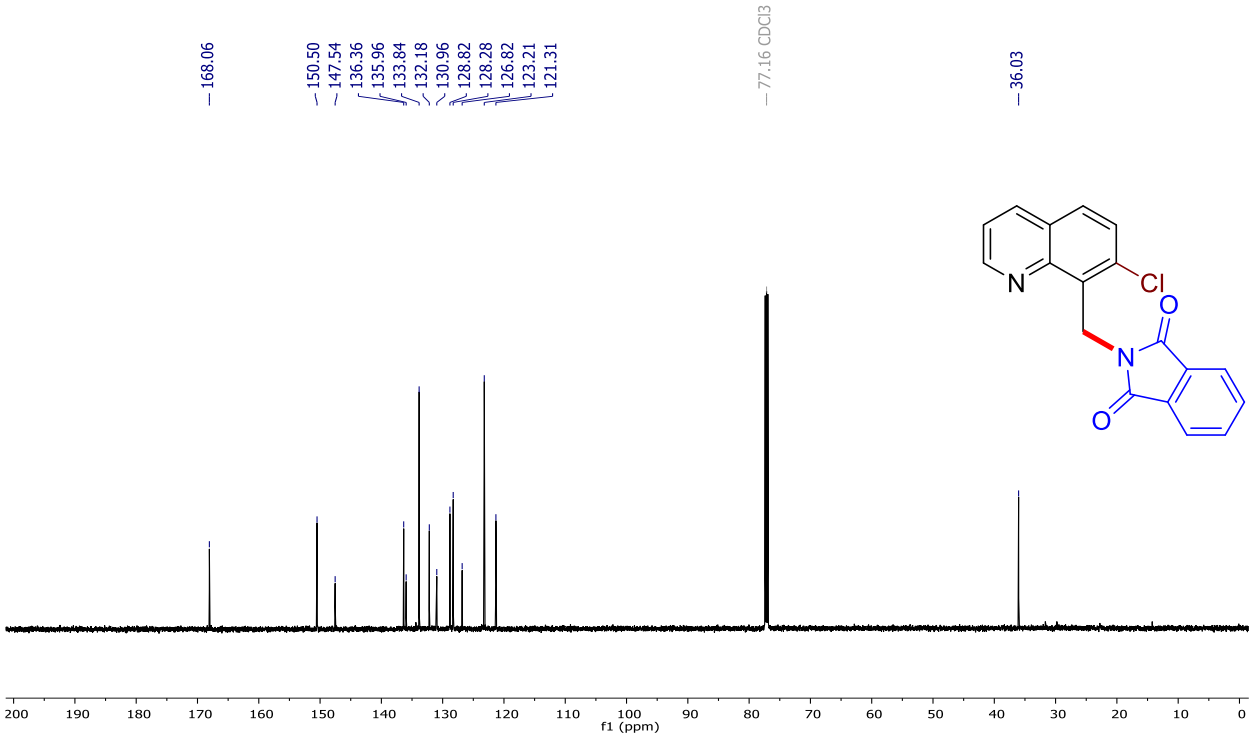


*2-((7-chloroquinolin-8-yl)methyl)isoindoline-1,3-dione (Scheme 2, Entry **3u**)*

^{1}H NMR (600 MHz)

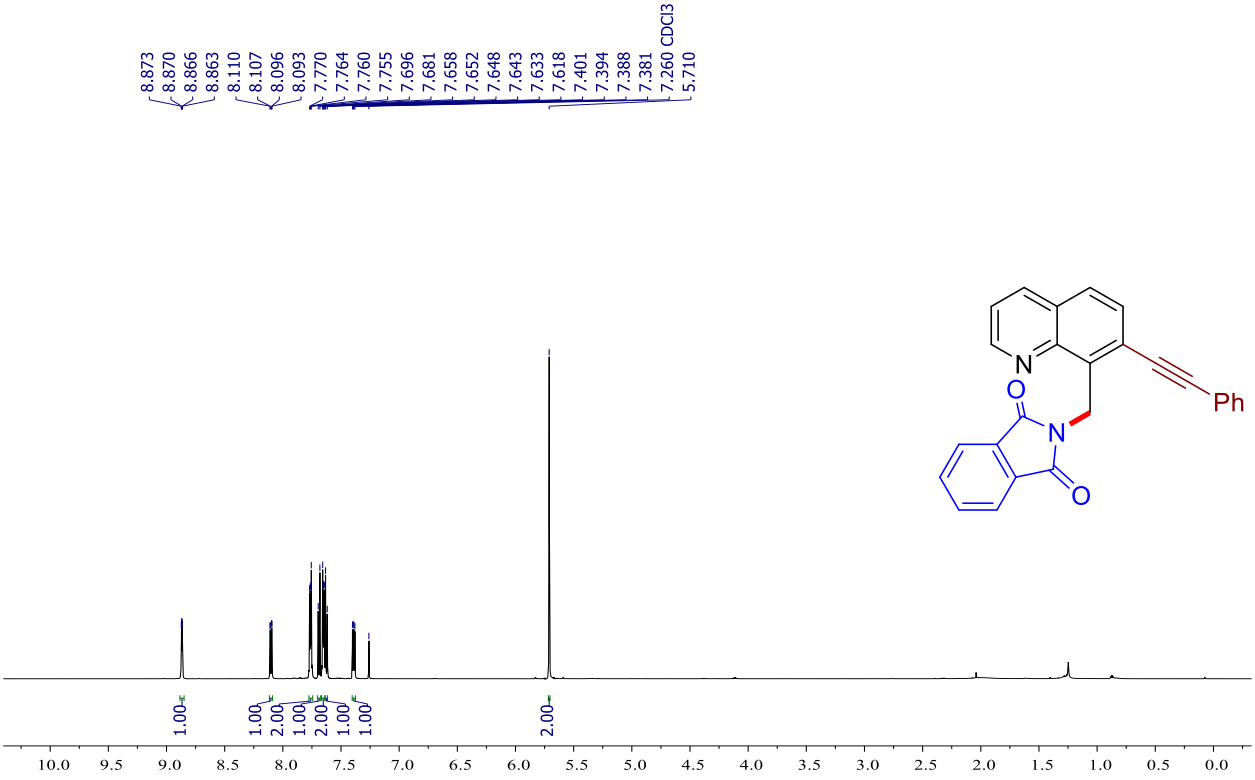


^{13}C{^{1}H} NMR *(*150 MHz*)*

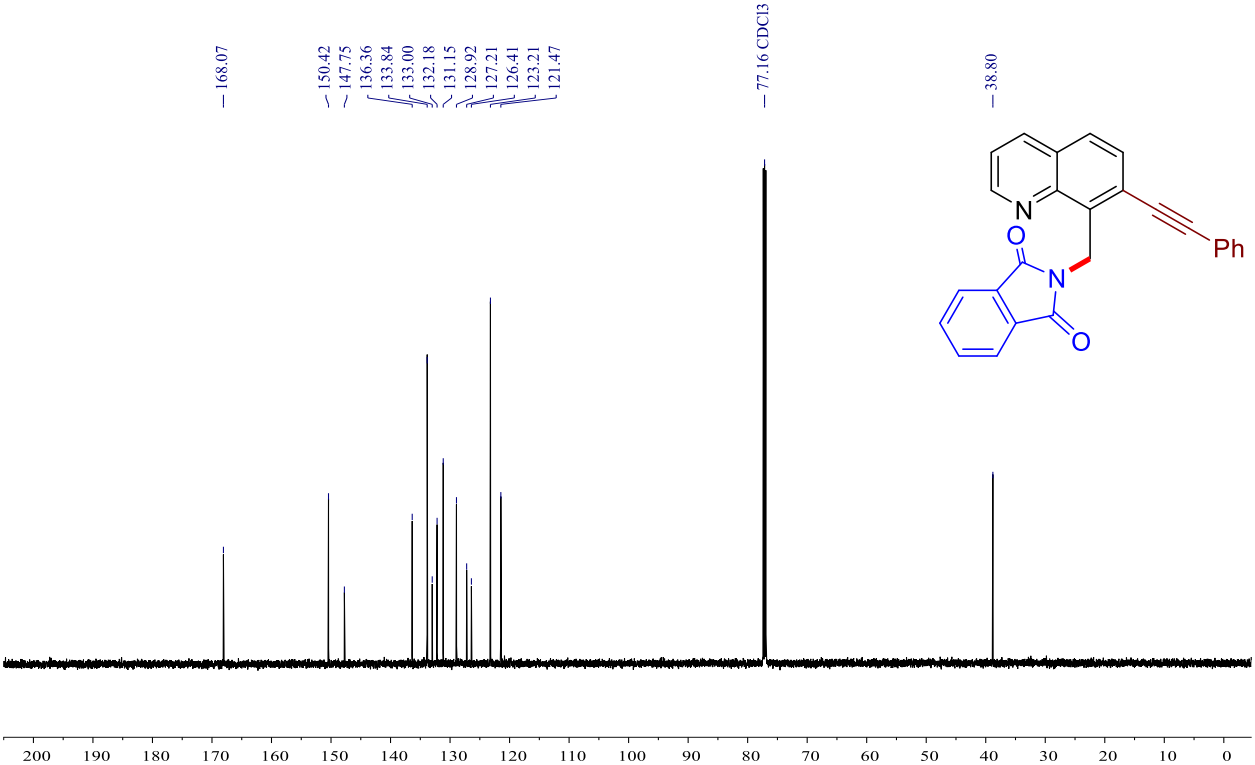


*2-((7-(phenylethynyl)quinolin-8-yl)methyl)isoindoline-1,3-dione (Table 6, Entry **3v**)*

^{1}H NMR (600 MHz)

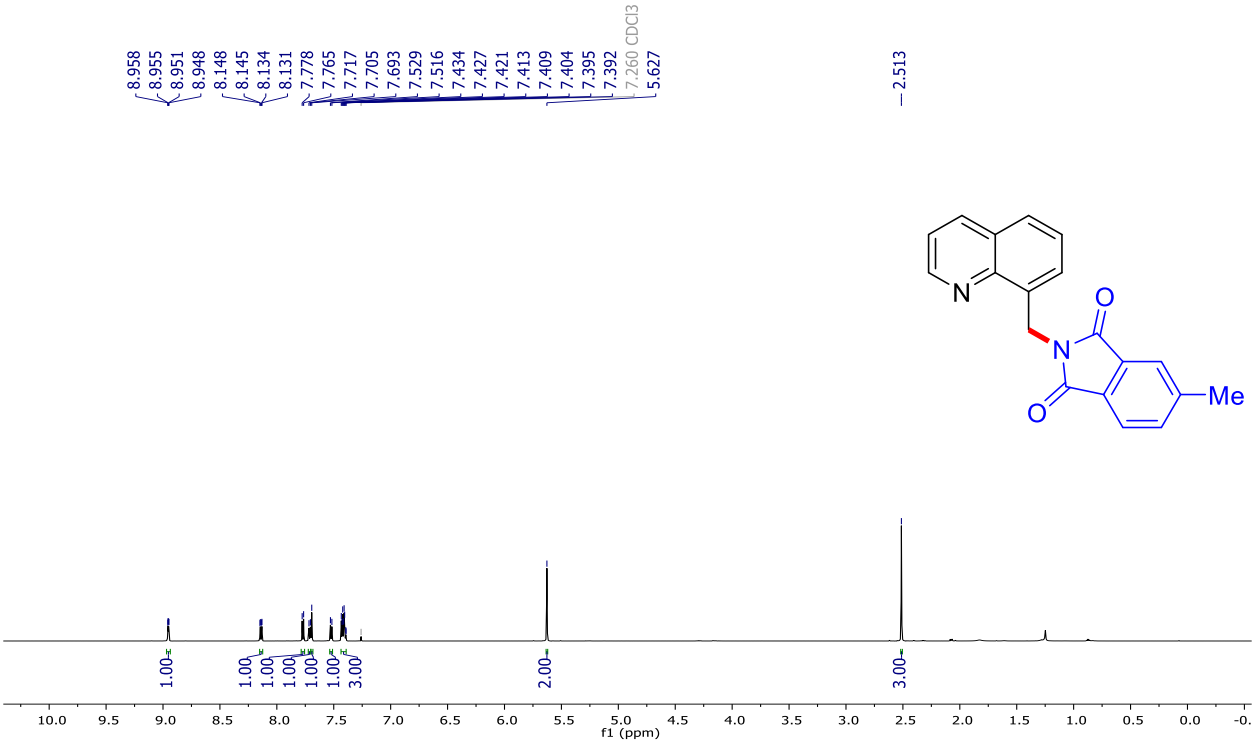


$^{13}C\{^{1}H\}$ NMR *(*150 MHz*)*

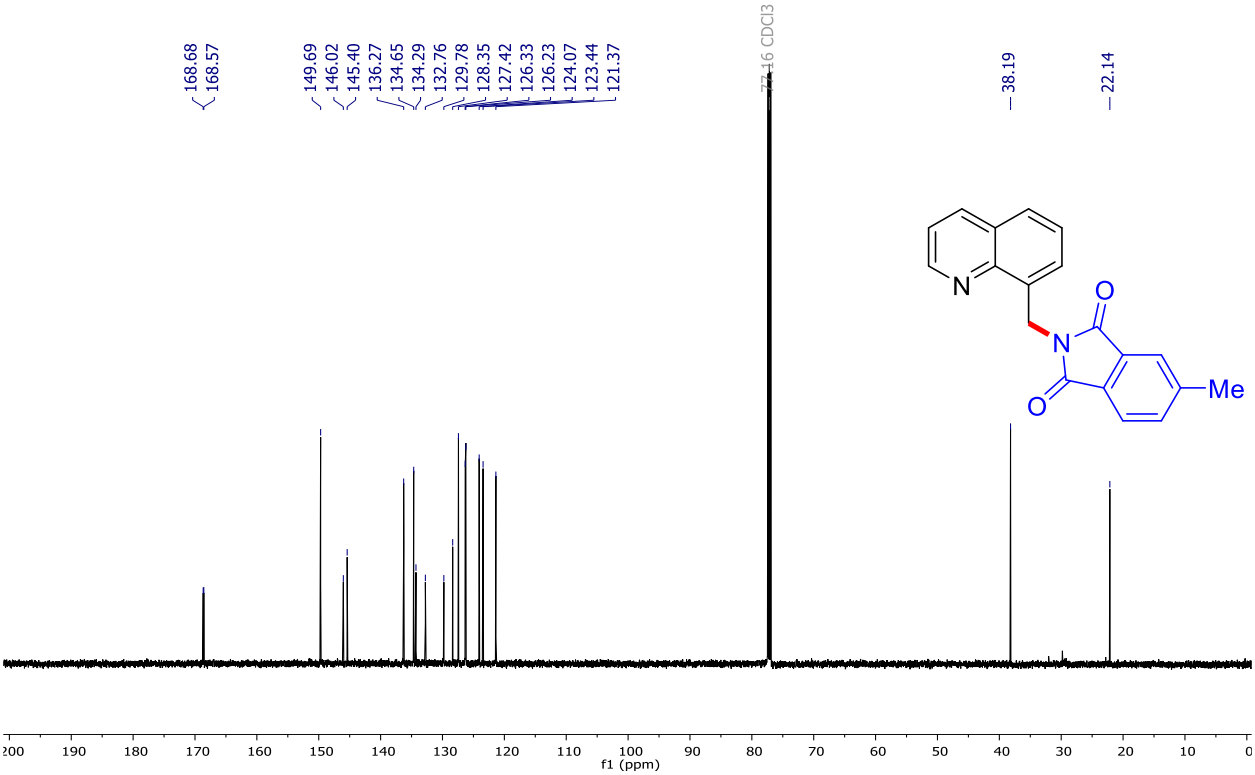


*5-methyl-2-(quinolin-8-ylmethyl)isoindoline-1,3-dione (Scheme 2, Entry **3w**)*

^{1}H NMR (600 MHz)

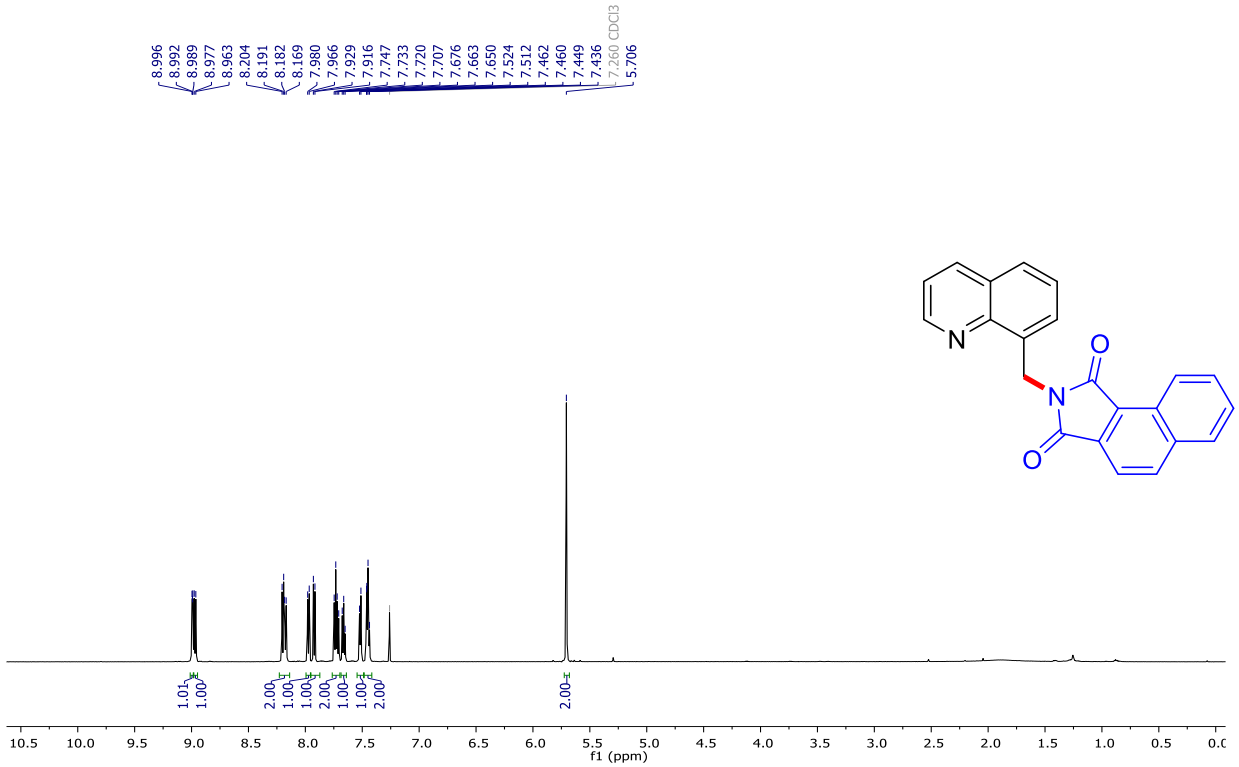


^{13}C{^{1}H} NMR *(*150 MHz*)*

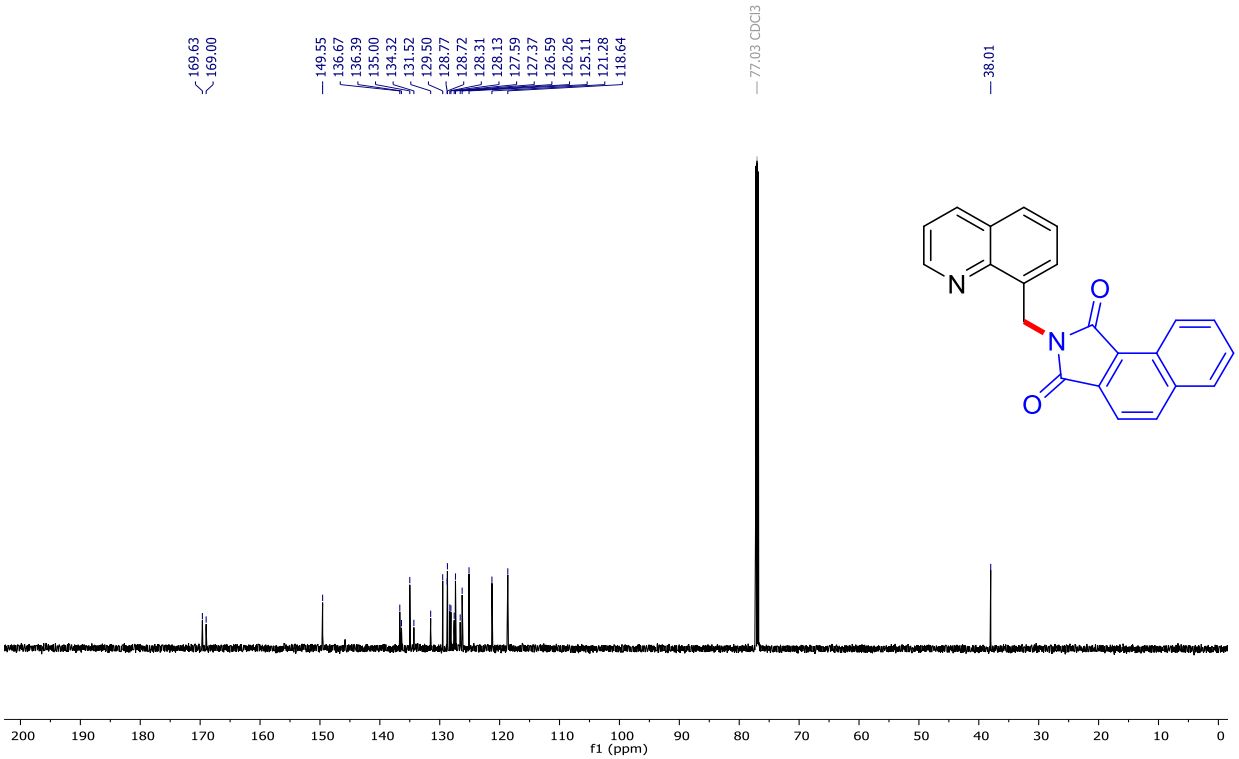


*2-(quinolin-8-ylmethyl)-1H-benzo[e]isoindole-1,3(2H)-dione (Scheme 2, Entry **3x**)*

^{1}H NMR (600 MHz)

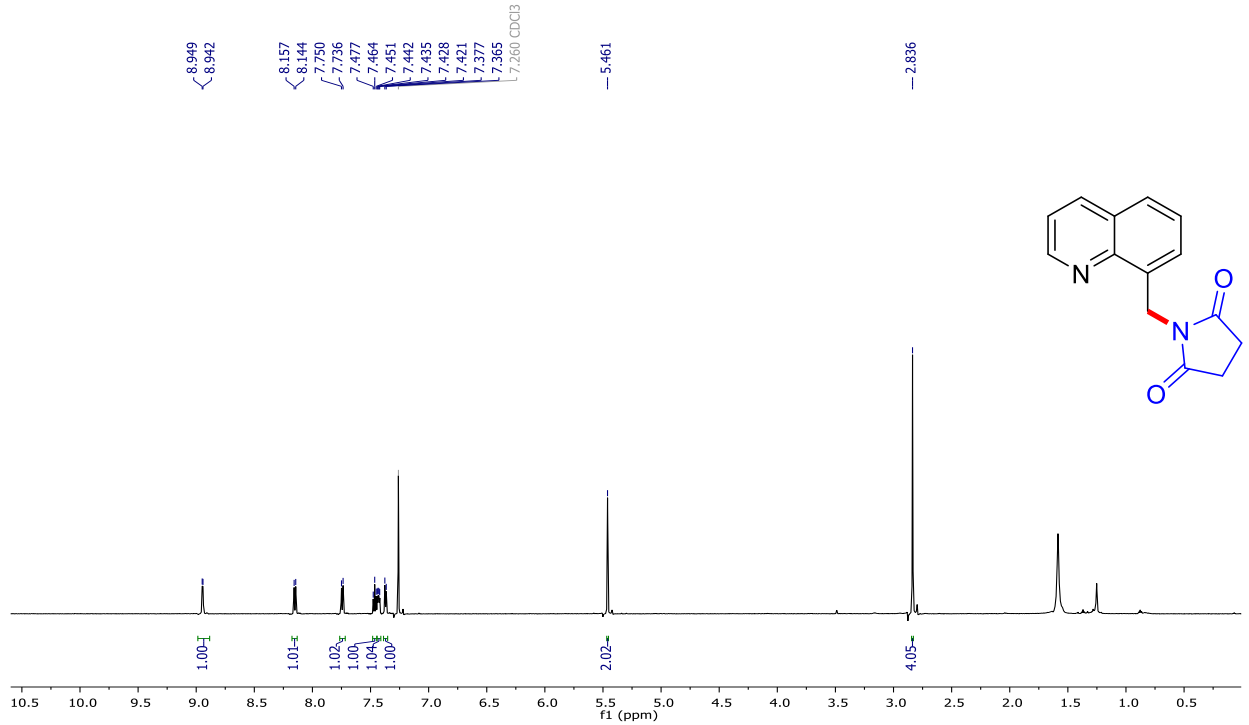


^{13}C{^{1}H} NMR *(*150 MHz*)*

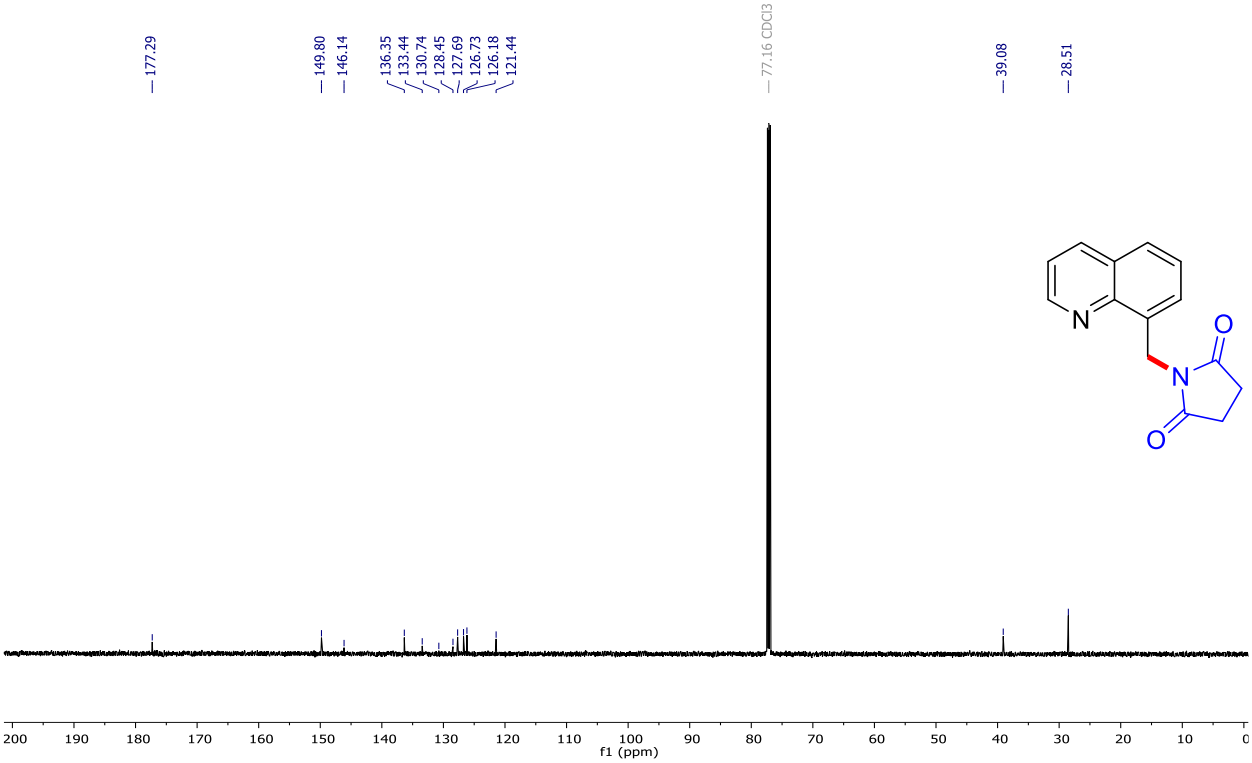


*1-(quinolin-8-ylmethyl)pyrrolidine-2,5-dione (Scheme 4, Entry **3y**)*

^{1}H NMR (600 MHz)

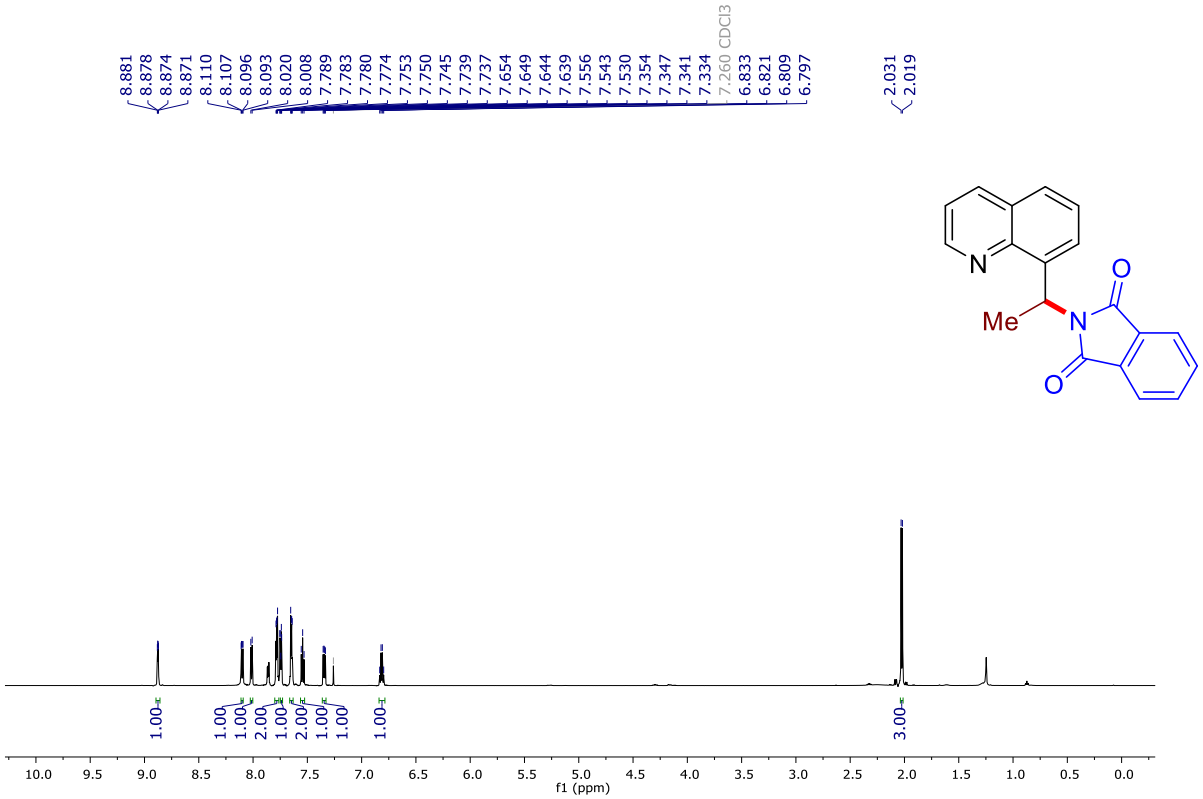


^{13}C{^{1}H} NMR *(*150 MHz*)*

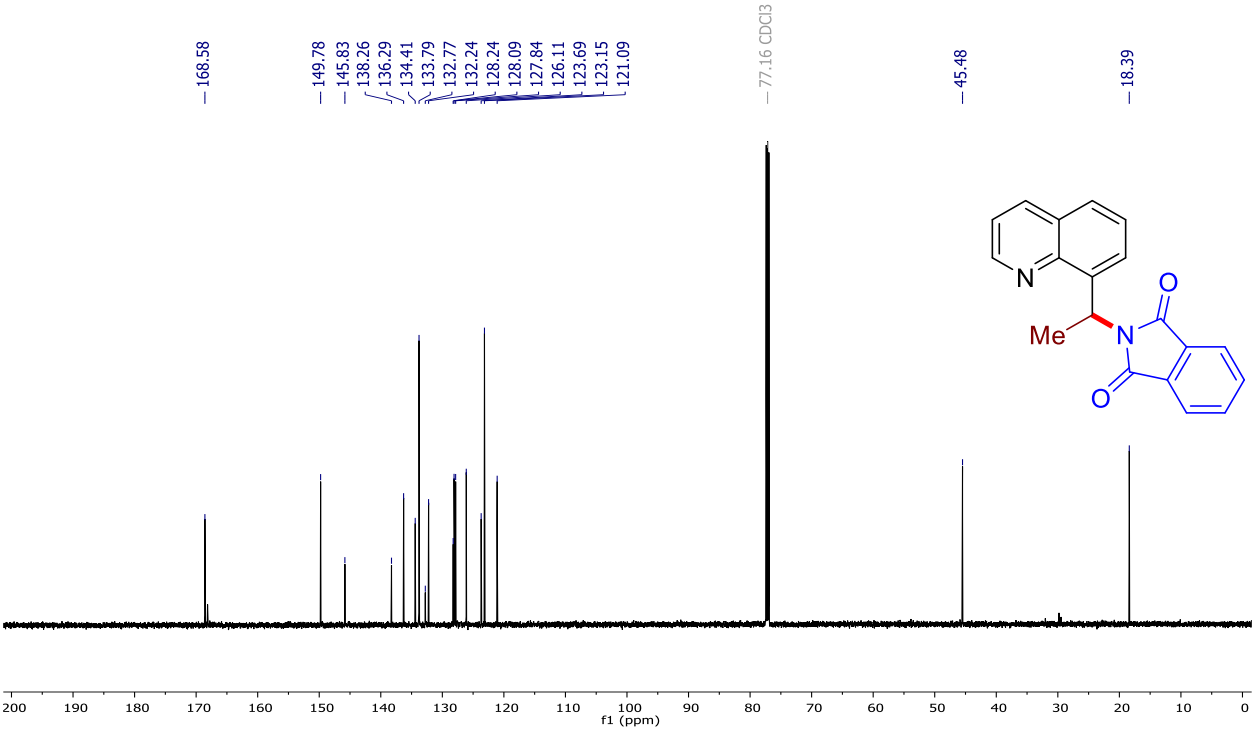


*2-(1-(quinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 4, Entry **5a**)*

^{1}H NMR (600 MHz)

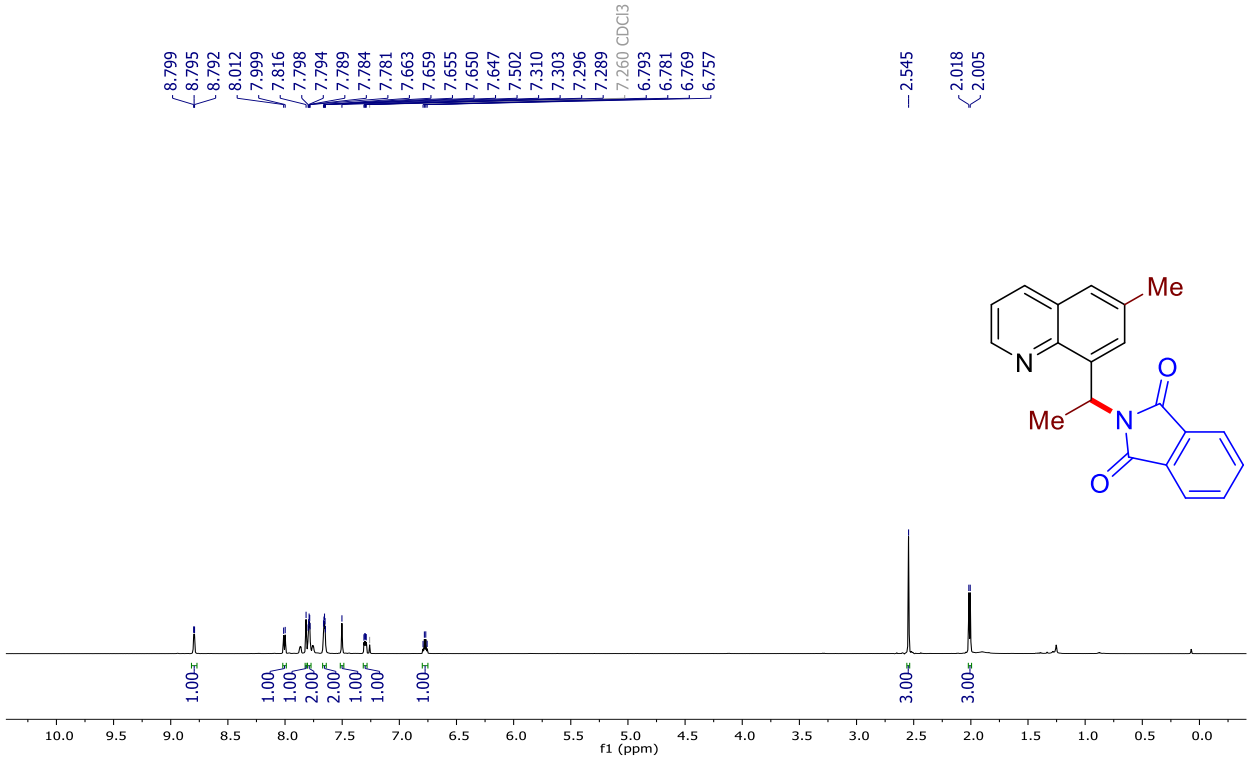


^{13}C{^{1}H} NMR *(*150 MHz*)*

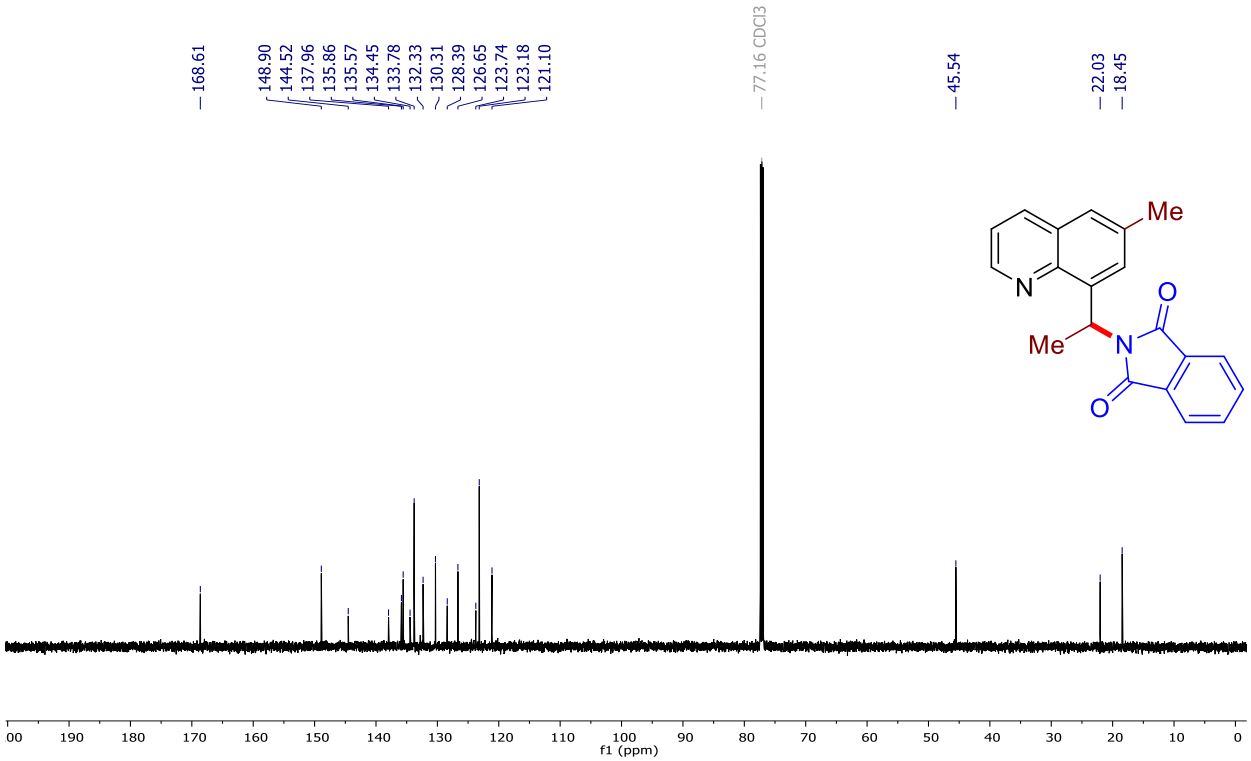


*2-(1-(6-methylquinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 2, Entry **5b**)*

^{1}H NMR (600 MHz)

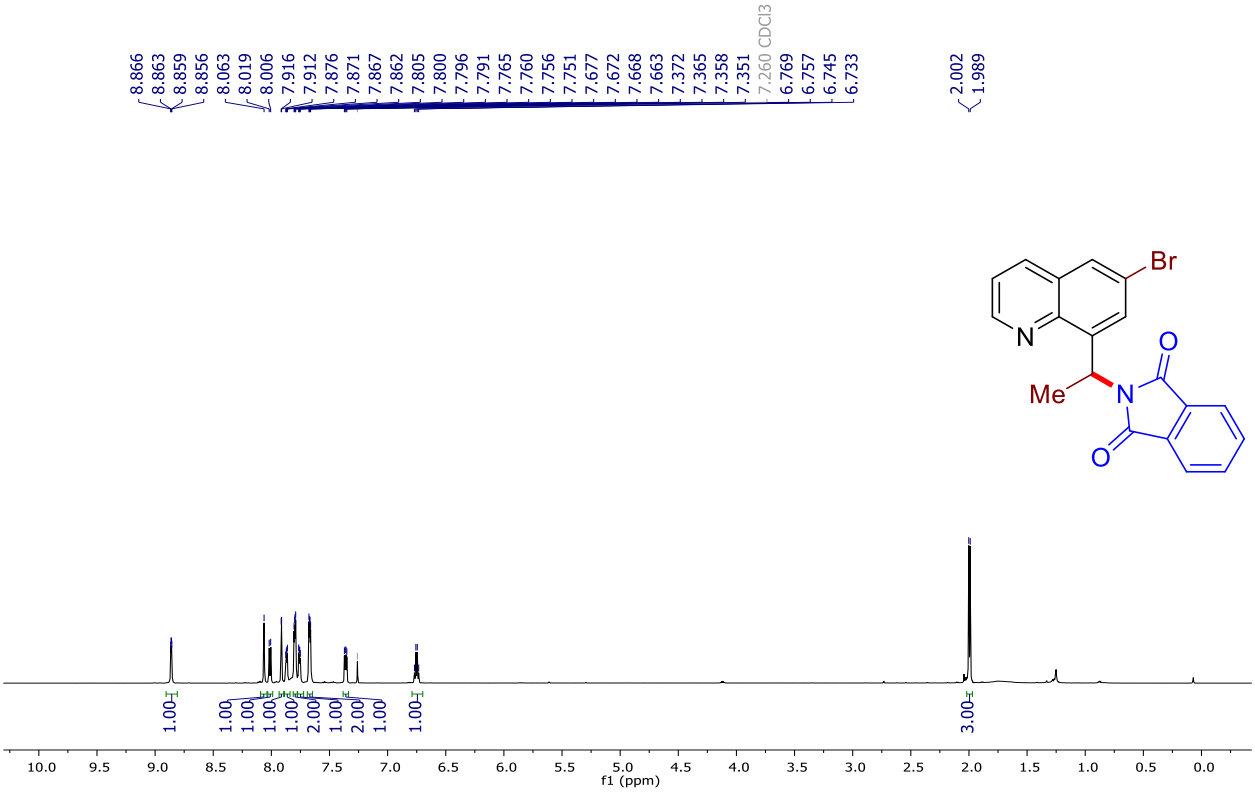


^{13}C{^{1}H} NMR *(*150 MHz*)*

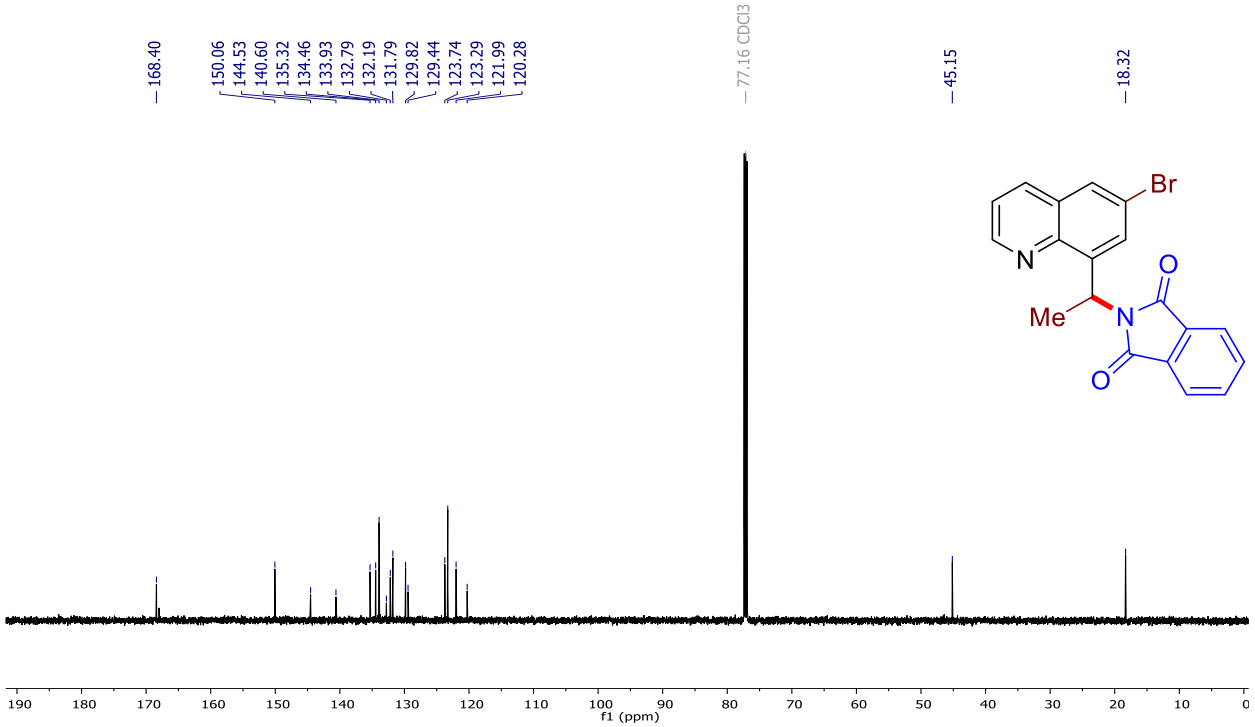


*2-(1-(5-bromoquinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 2, Entry **5c**)*

^{1}H NMR (600 MHz)

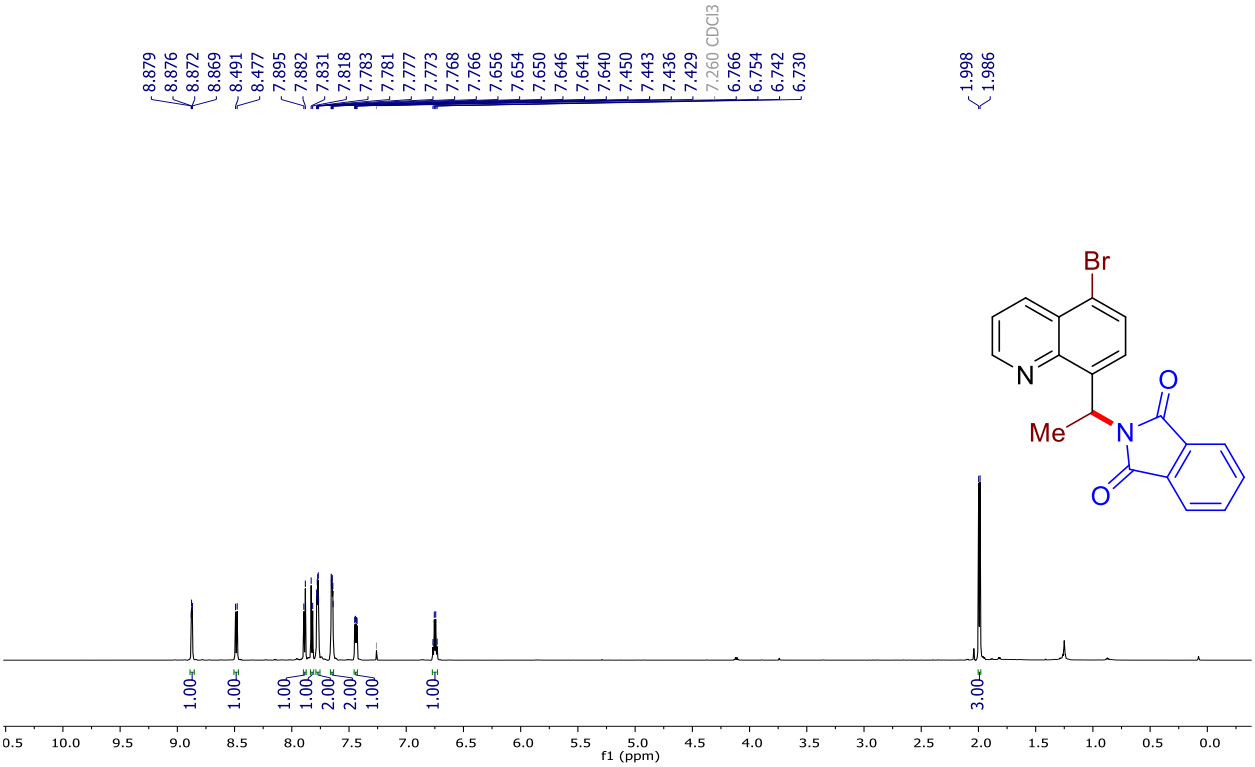


^{13}C{^{1}H} NMR *(*150 MHz*)*

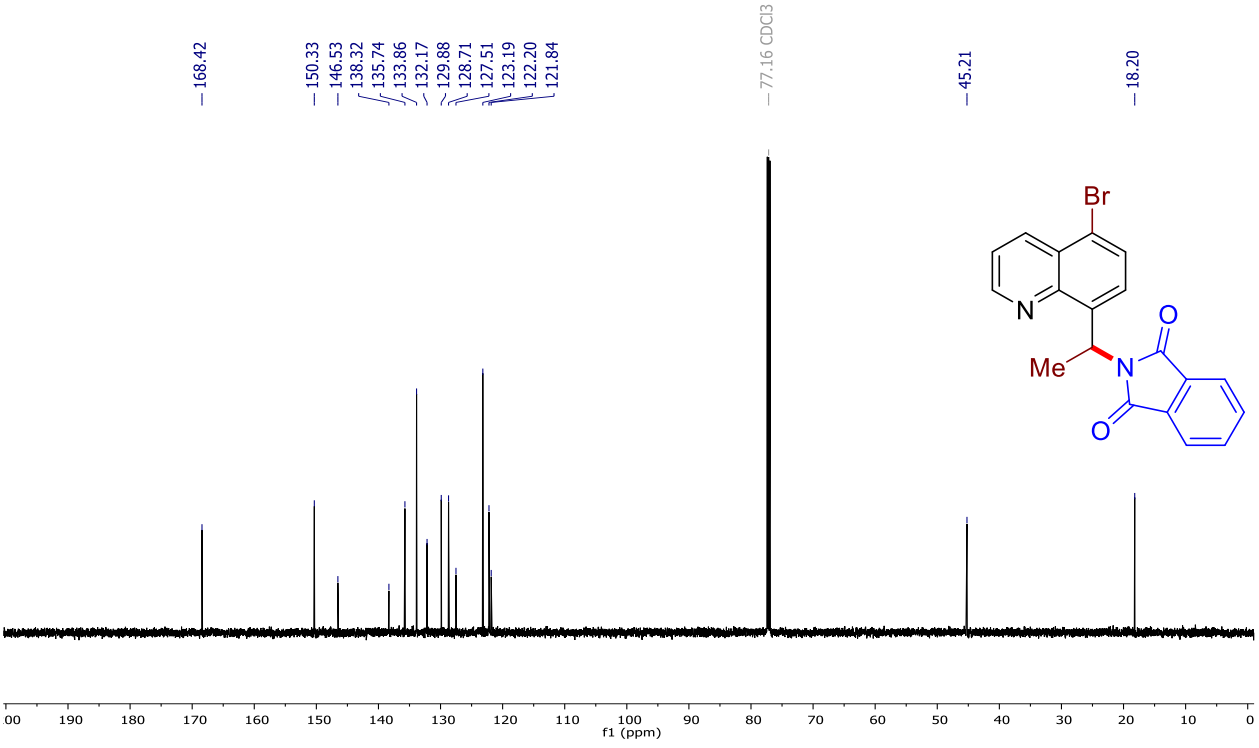


*2-(1-(5-bromoquinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 4, Entry **5d**)*

^{1}H NMR (600 MHz)

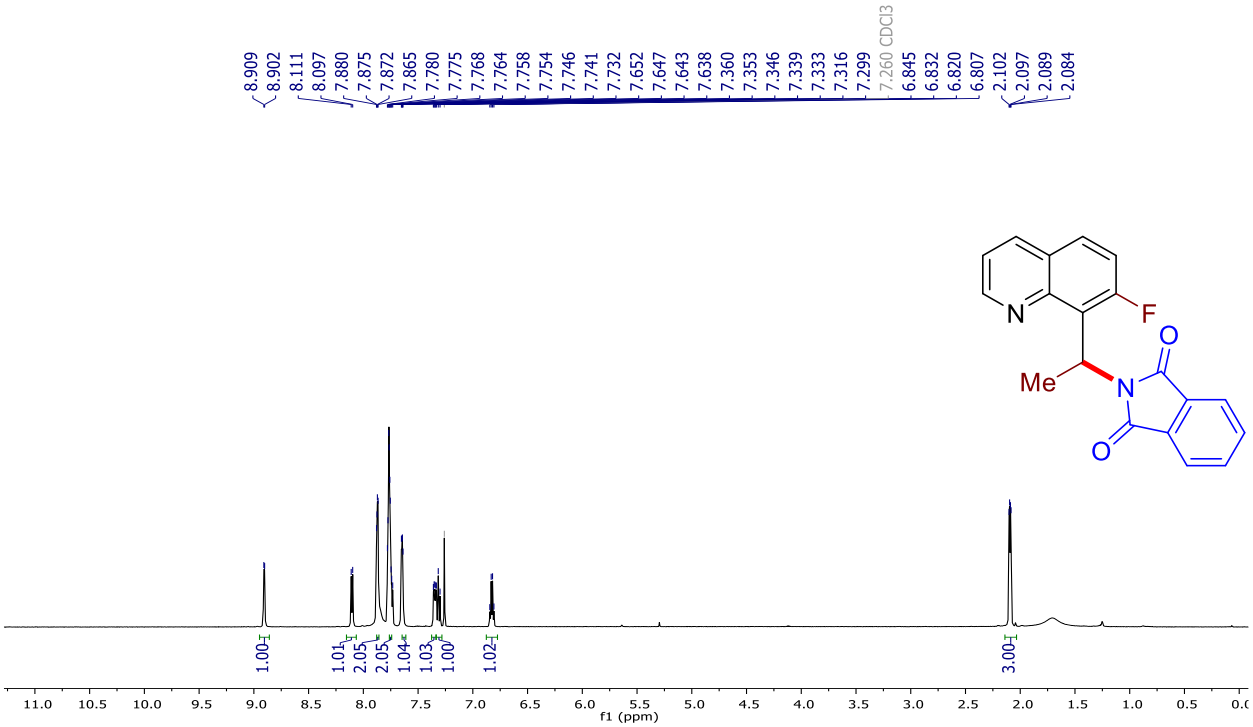


^{13}C{^{1}H} NMR *(*150 MHz*)*

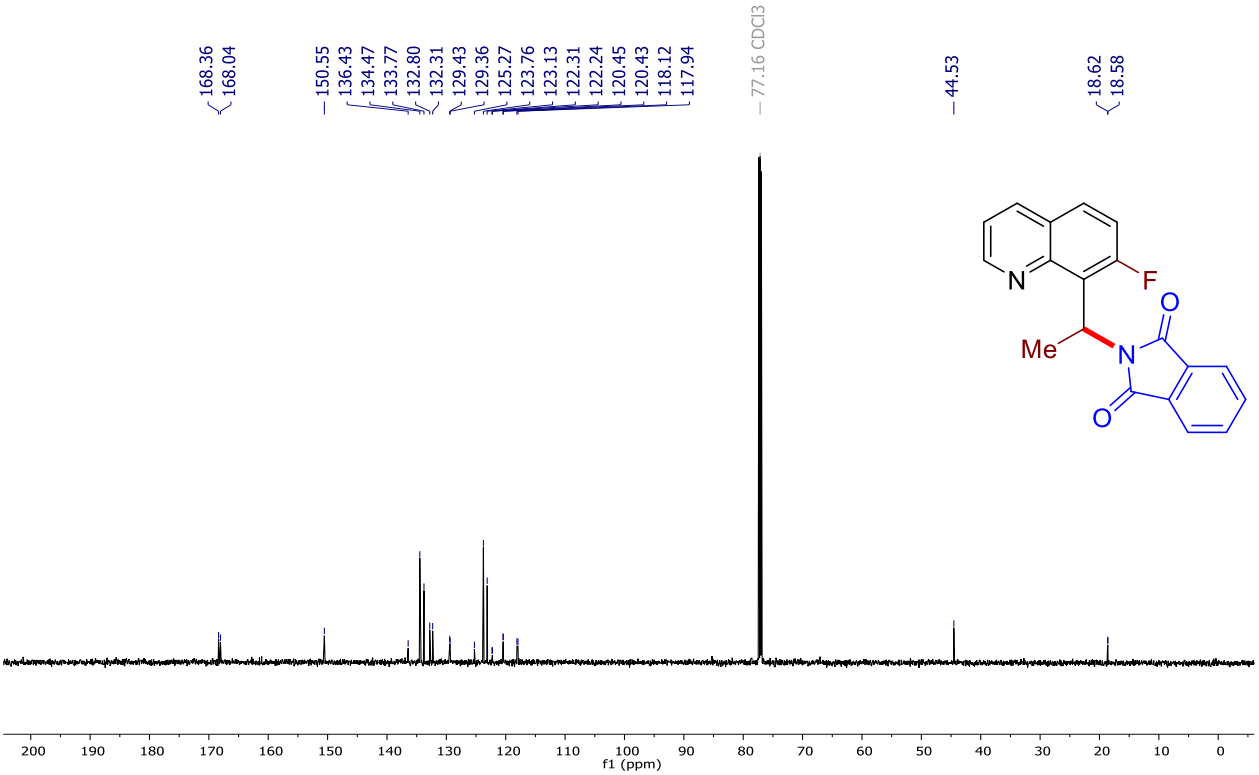


*2-(1-(7-fluoroquinolin-8-yl)ethyl)isoindoline-1,3-dione (Scheme 4, Entry **5e**)*

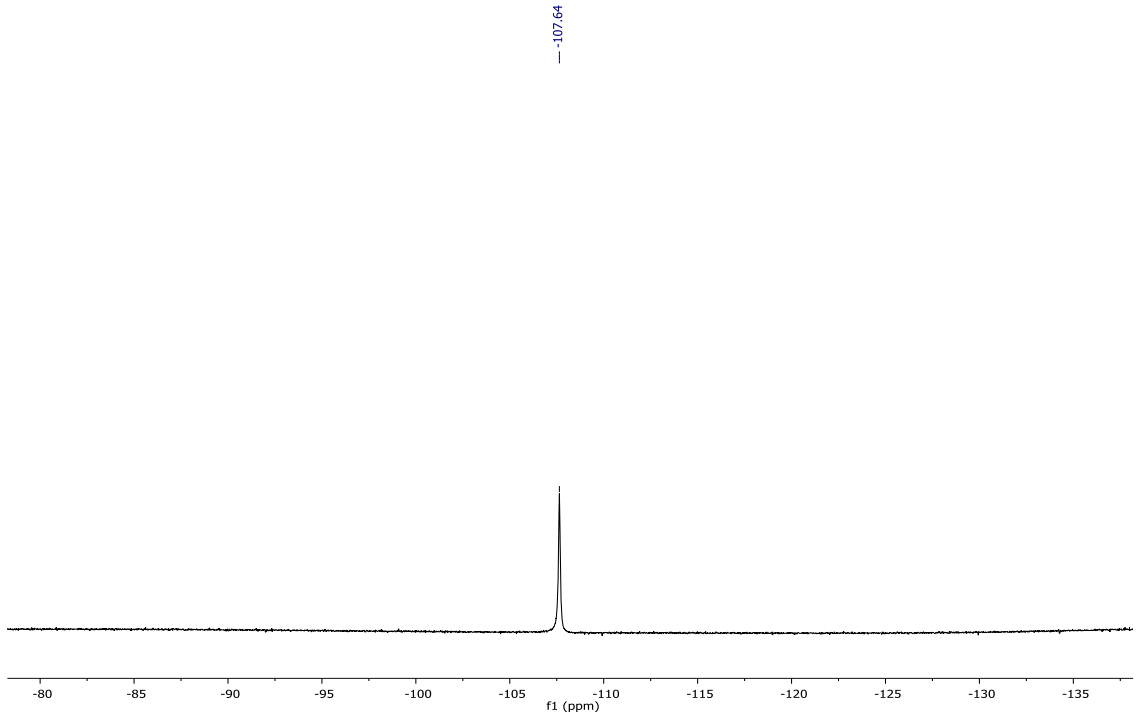
^{1}H NMR (600 MHz)



^{13}C{^{1}H} NMR *(*150 MHz*)*

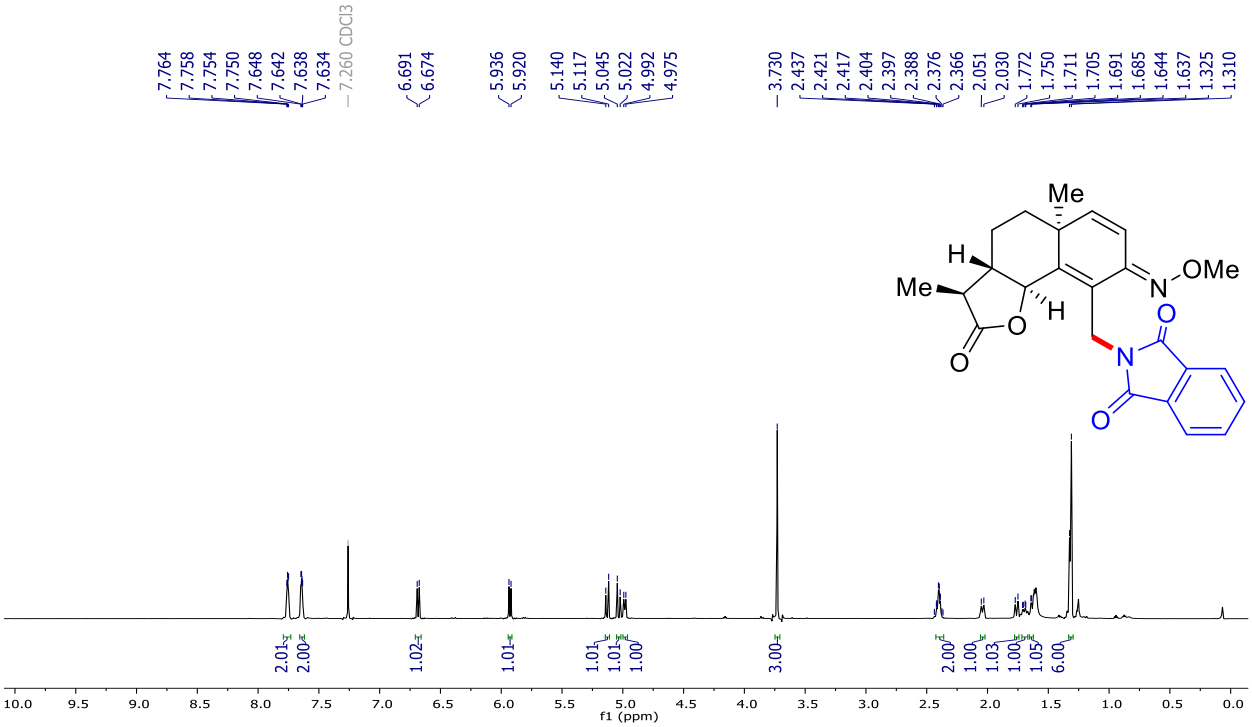


^{19}F NMR *(*565 MHz*)*

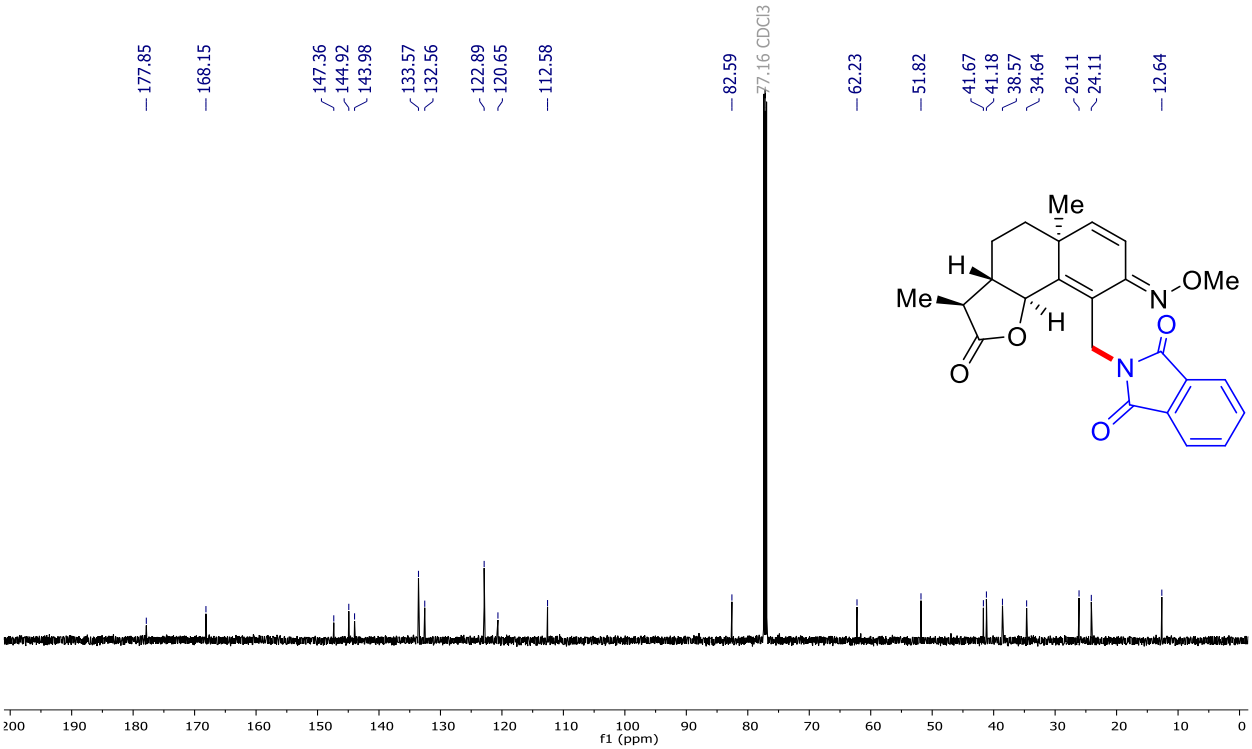


*8-(methoxyimino)-3,5a-dimethyl-2-oxo-2,3,3a,4,5,5a,8,9b-octahydronaphtho[1,2-b]furan-9-yl)methyl)isoindoline-1,3-dione (Scheme 4, Entry **7a**)*

^{1}H NMR (600 MHz)

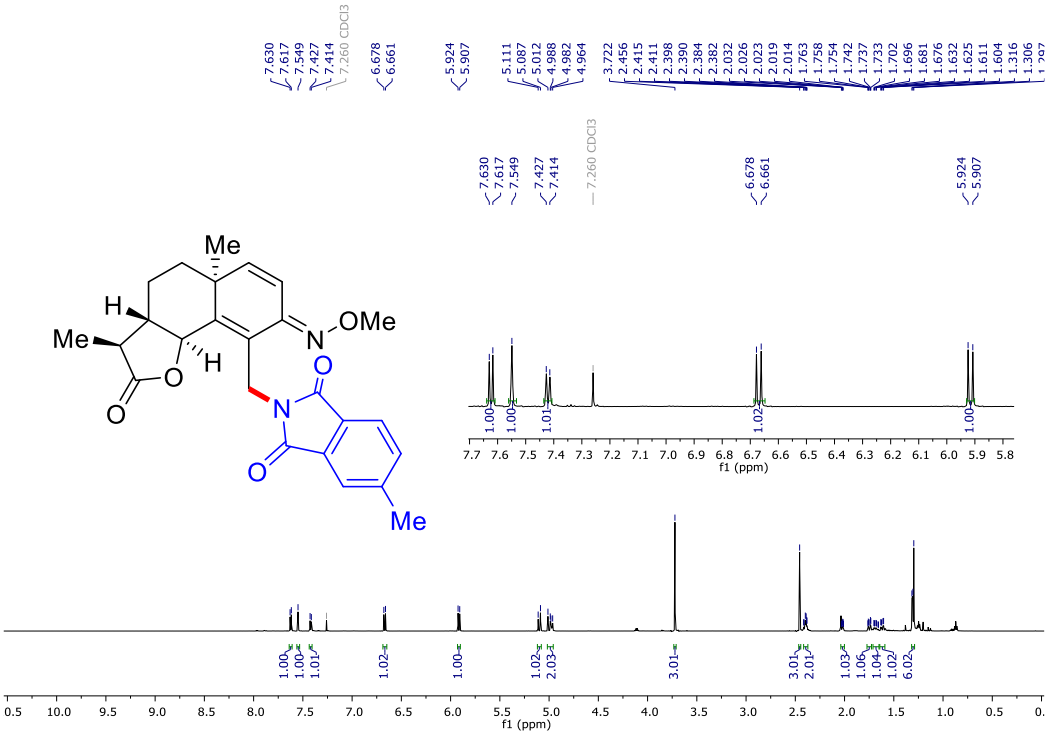


^{13}C{^{1}H} NMR *(*150 MHz*)*

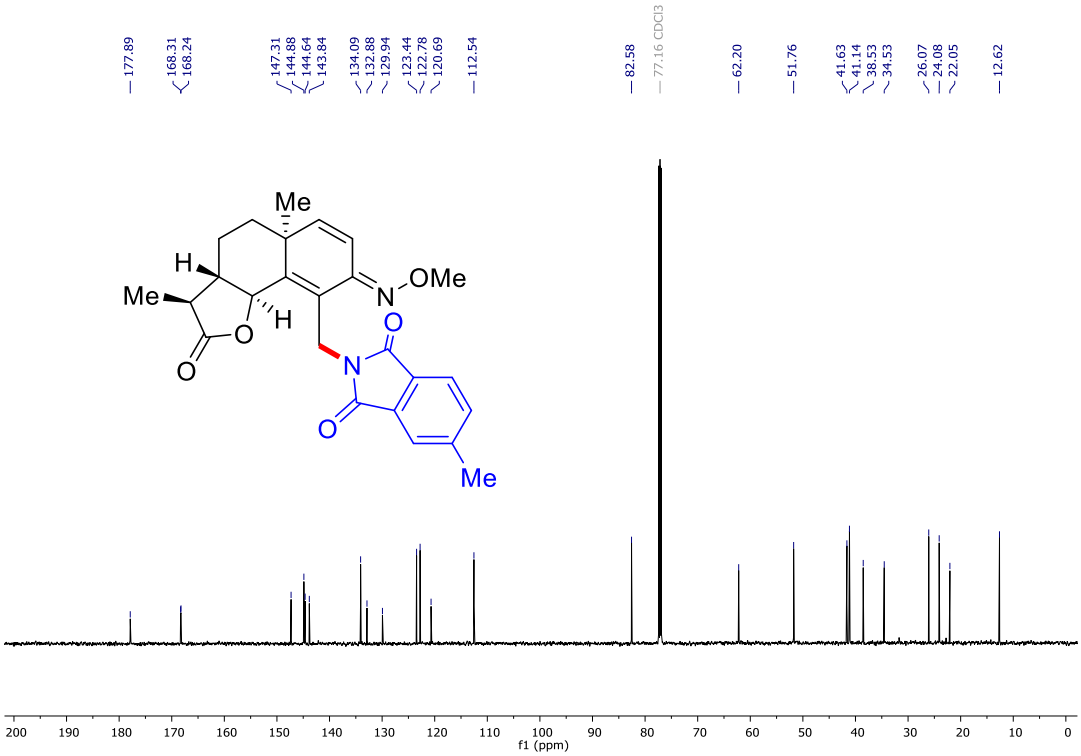


*8-(methoxyimino)-3,5a-dimethyl-2-oxo-2,3,3a,4,5,5a,8,9b-octahydronaphtho[1,2-b]furan-9-yl)methyl)-5-methylisoindoline-1,3-dione (Scheme 4, Entry **7b**)*

^{1}H NMR (600 MHz)

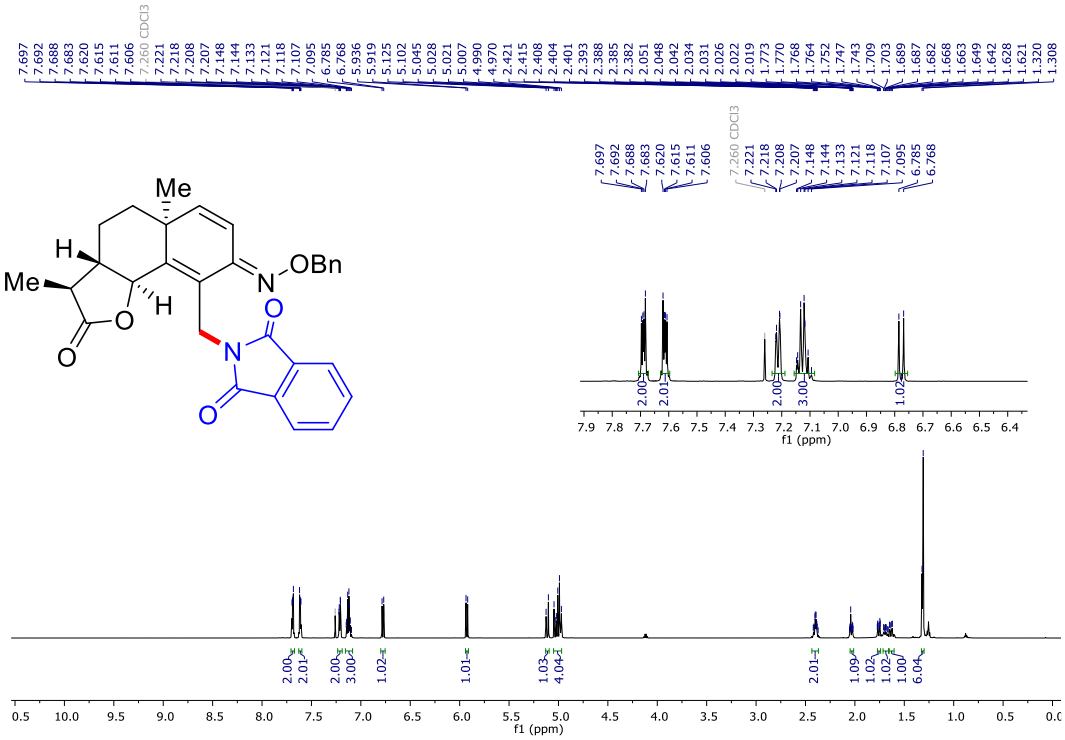


^{13}C{^{1}H} NMR *(*150 MHz*)*

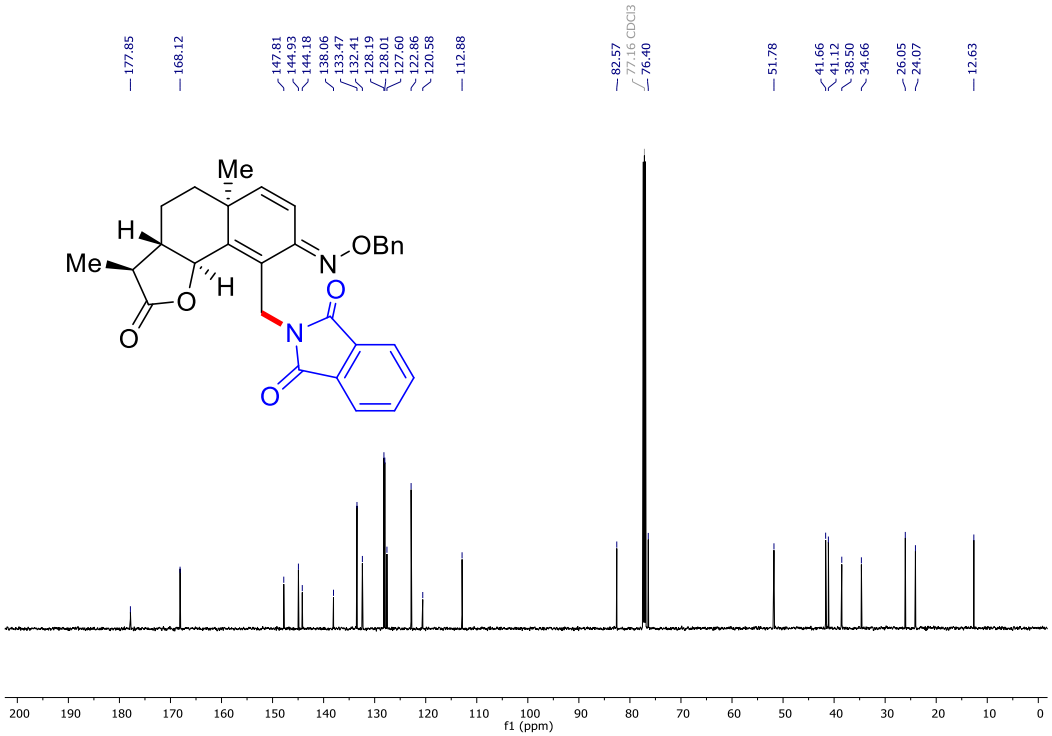


*8-((benzyloxy)imino)-3,5a-dimethyl-2-oxo-2,3,3a,4,5,5a,8,9b-octahydronaphtho[1,2-b]furan-9-yl)methyl)isoindoline-1,3-dione (Scheme 4, Entry **7c**)*

^{1}H NMR (600 MHz)

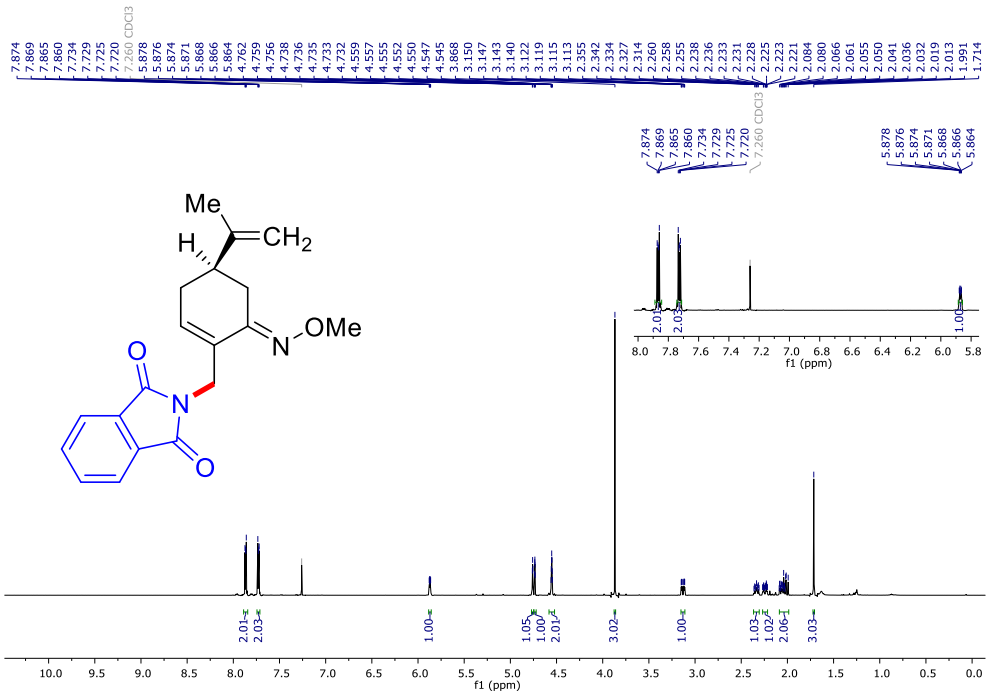


^{13}C{^{1}H} NMR *(*150 MHz*)*

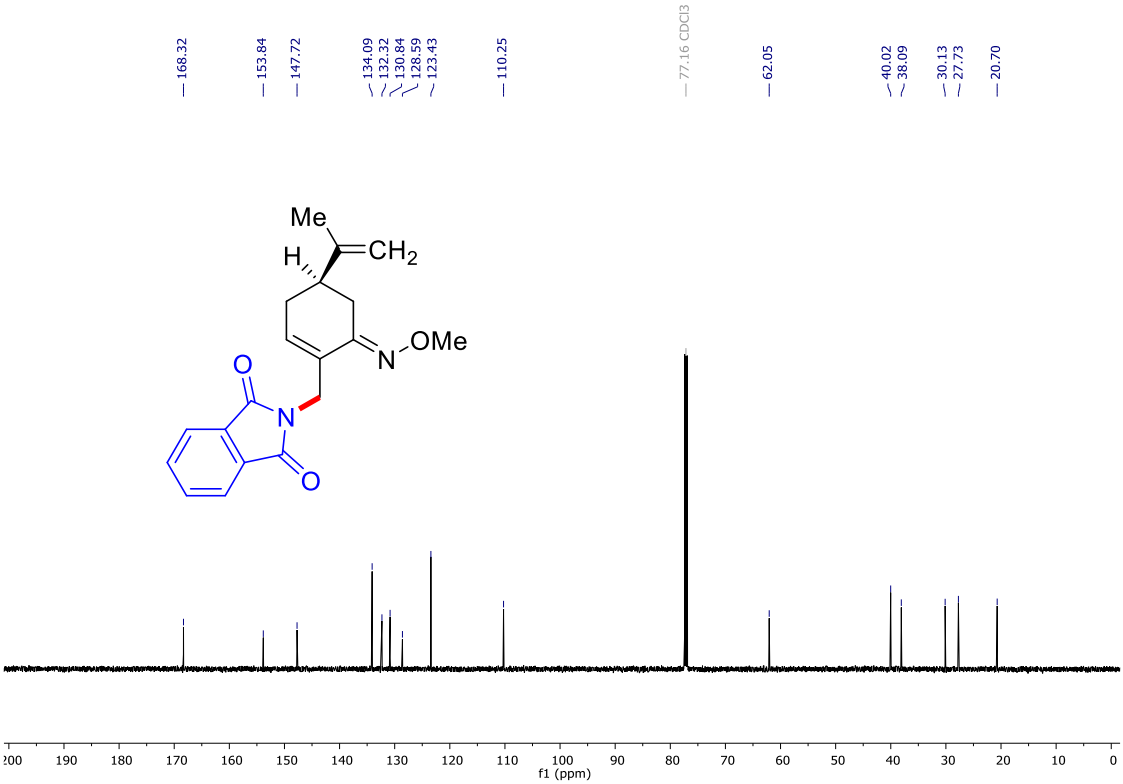


*2-((6-(methoxyimino)-4-(prop-1-en-2-yl)cyclohex-1-en-1-yl)methyl)isoindoline-1,3-dione (Scheme 4, Entry **7d**)*

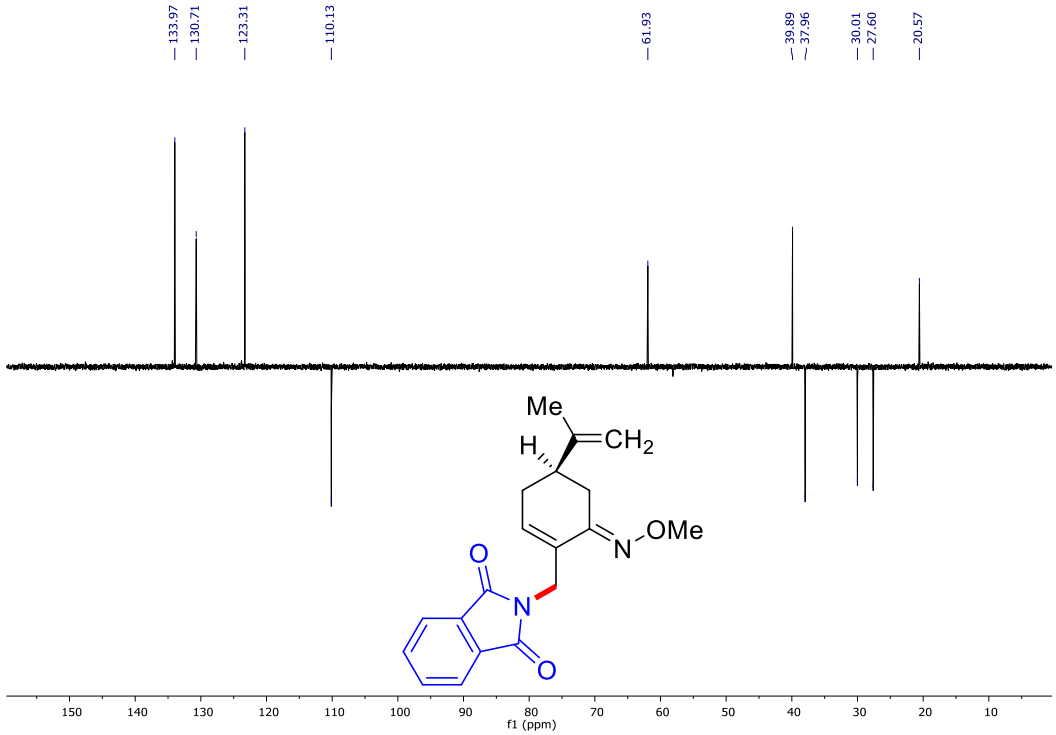
^{1}H NMR (600 MHz)



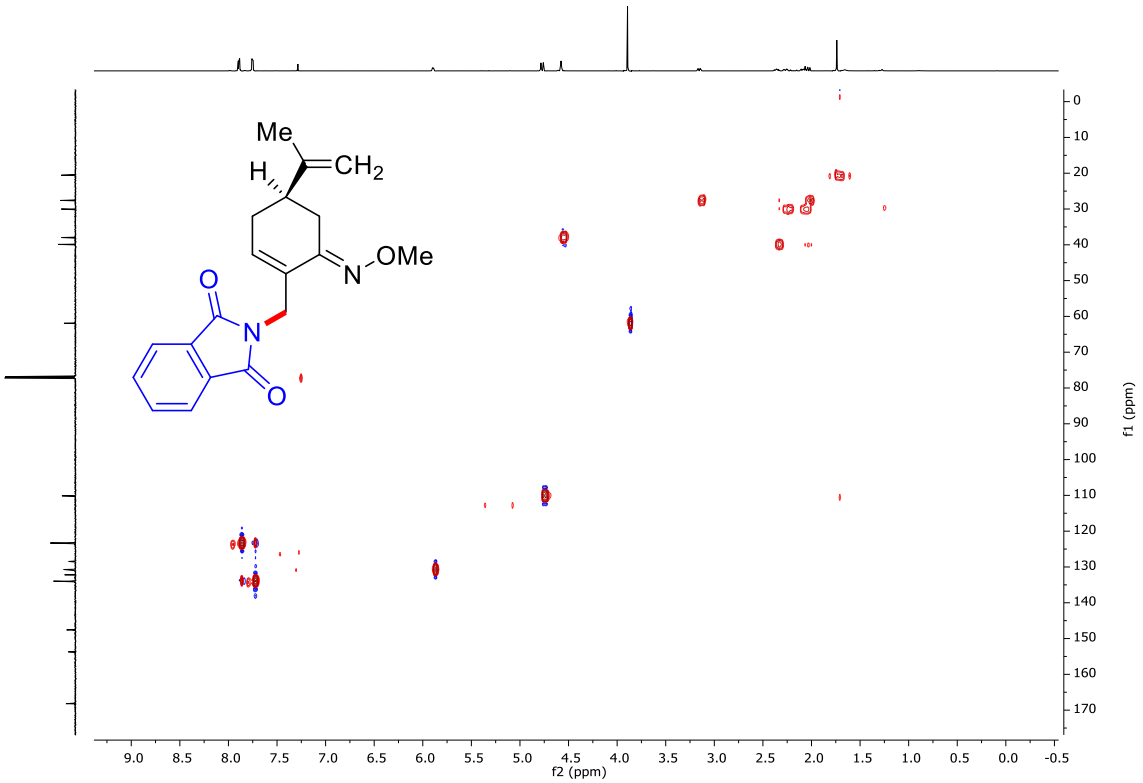
^{13}C{^{1}H} NMR *(*150 MHz*)*



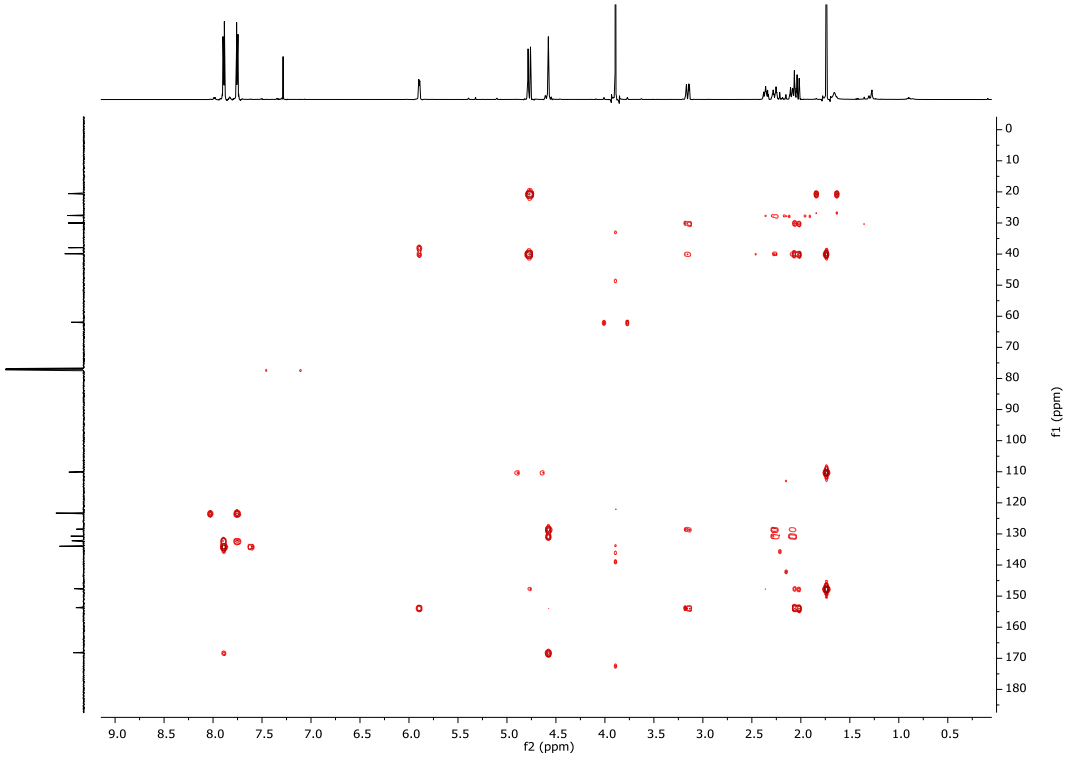
$^{13}C\{^{1}H\}$ DEPT-135 *(*150 MHz*)*



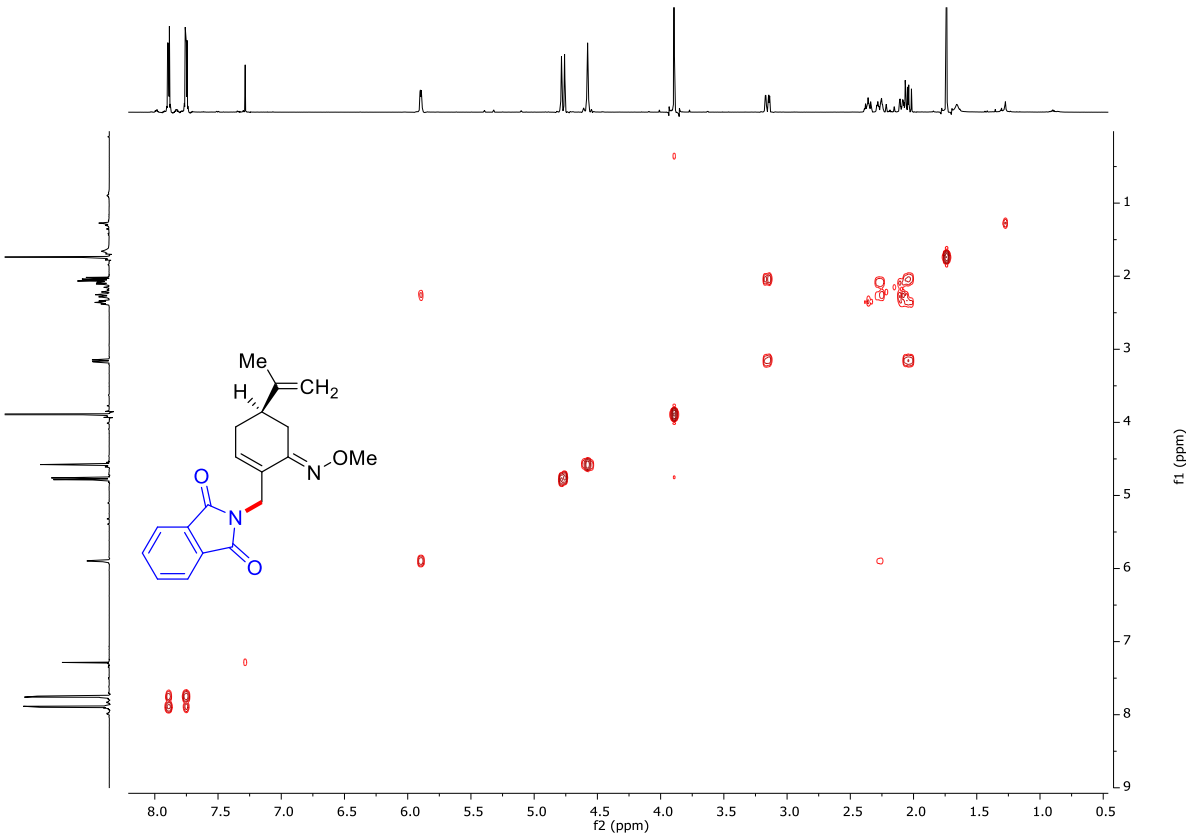
HSQC experiment



HMBC experiment

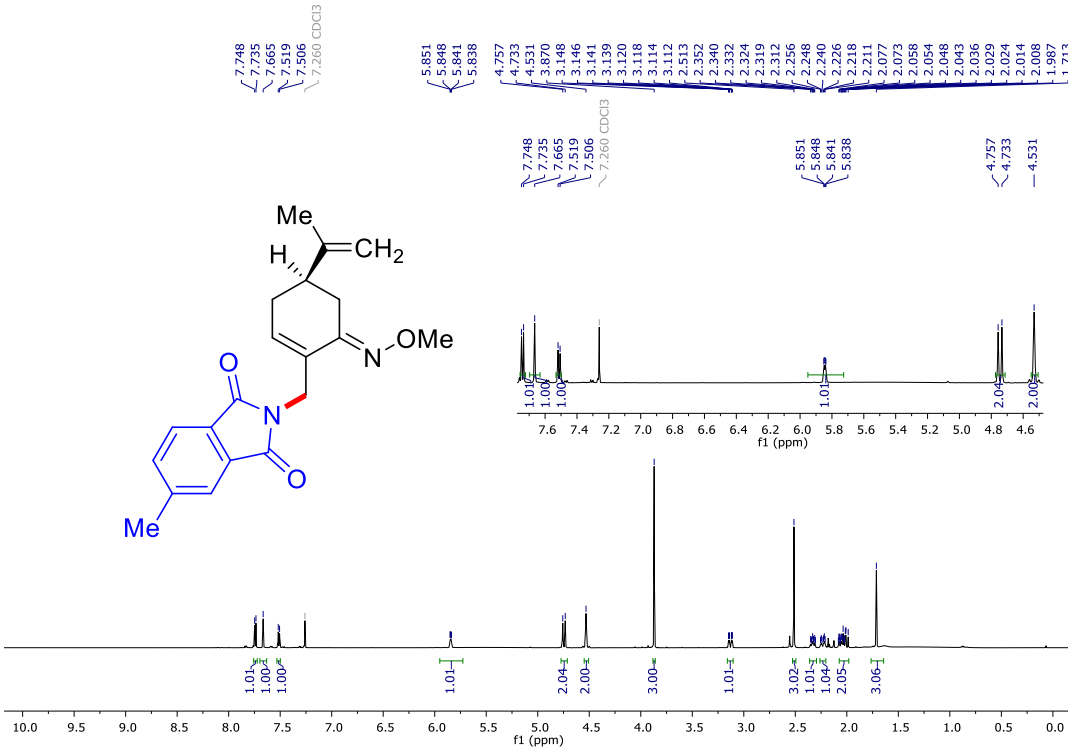


COSY experiment

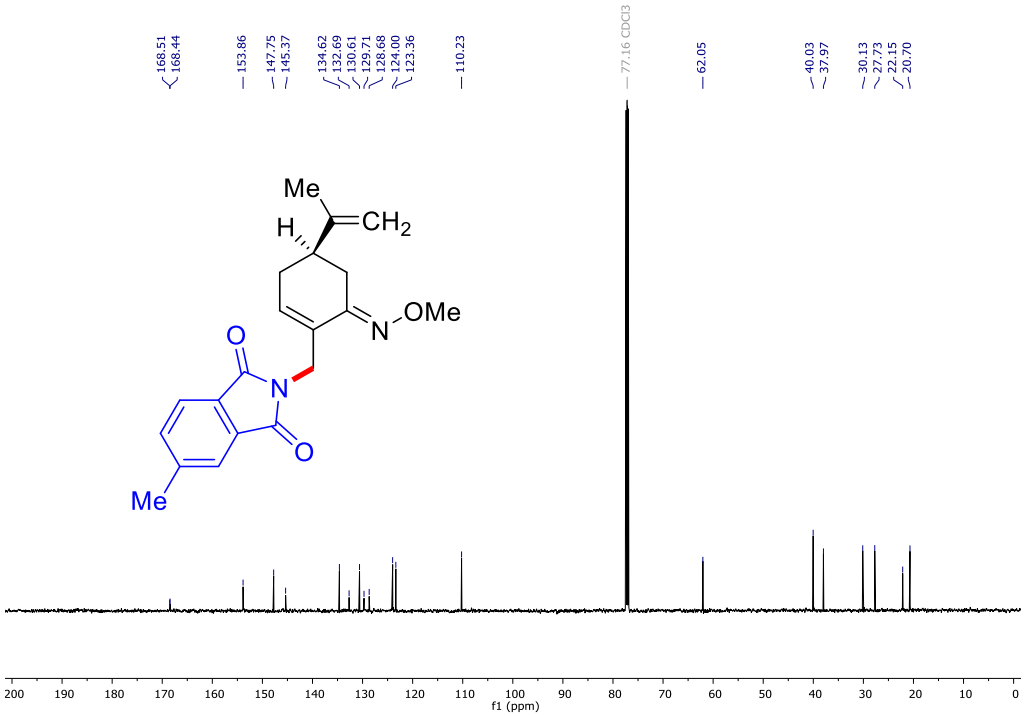


*2-((6-(methoxyimino)-4-(prop-1-en-2-yl)cyclohex-1-en-1-yl)methyl)-5-methylisoindoline-1,3-dione (Scheme 4, Entry **7e**)*

^{1}H NMR (600 MHz)

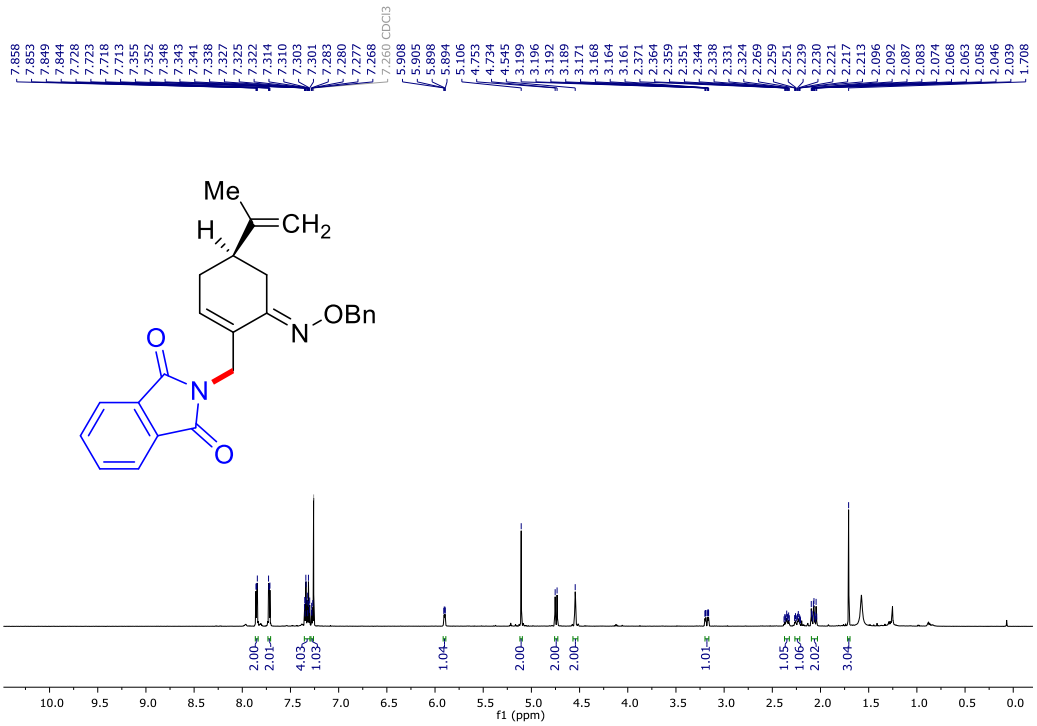


^{13}C{^{1}H} NMR *(*150 MHz*)*

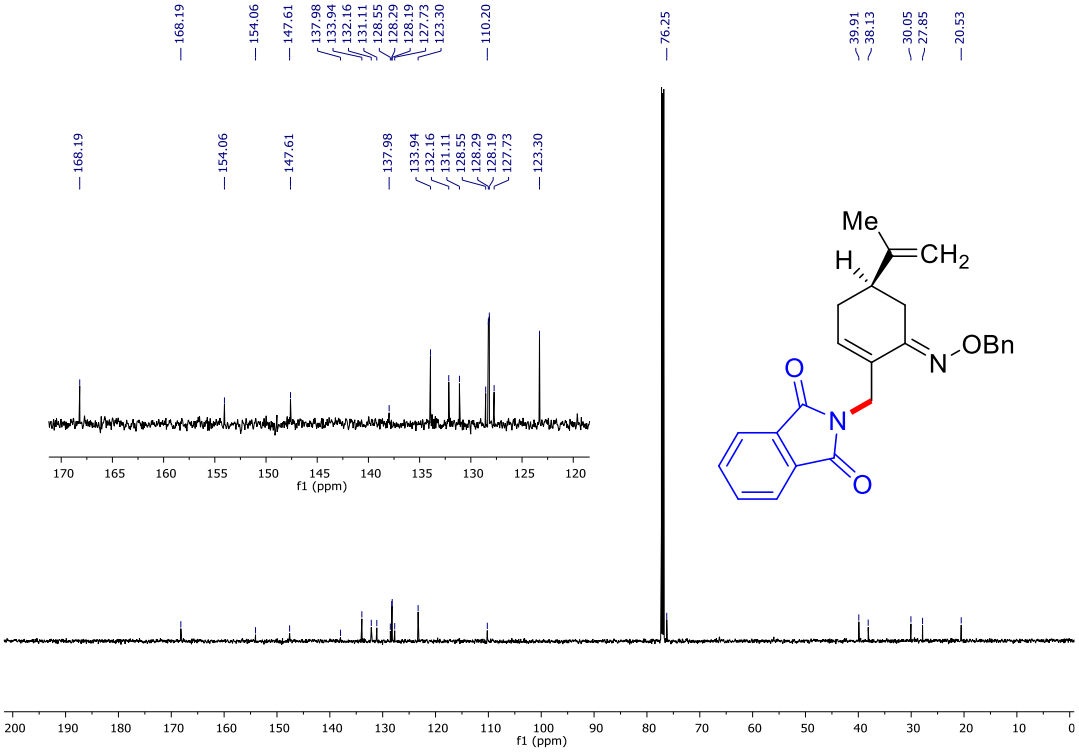


*2-((6-((benzyloxy)imino)-4-(prop-1-en-2-yl)cyclohex-1-en-1-yl)methyl)isoindoline-1,3-dione (Scheme 4, Entry **7f**)*

^{1}H NMR (600 MHz)

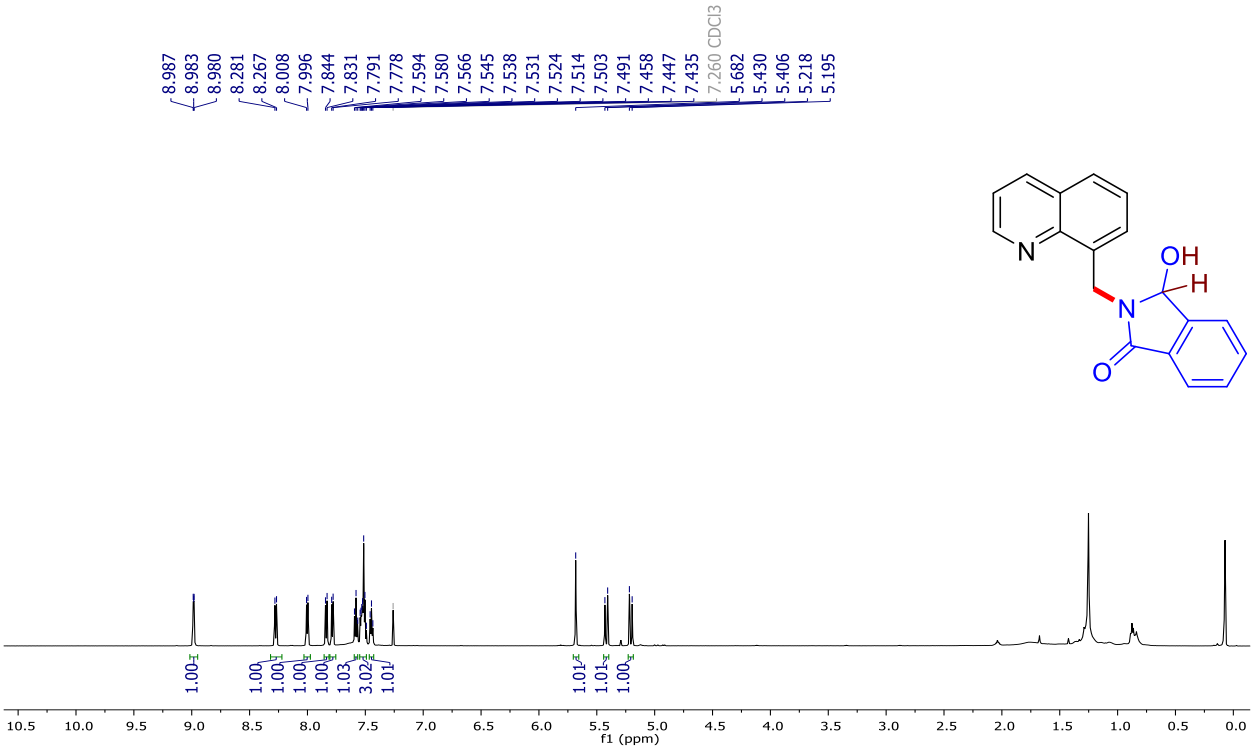


^{13}C{^{1}H} NMR *(*150 MHz*)*

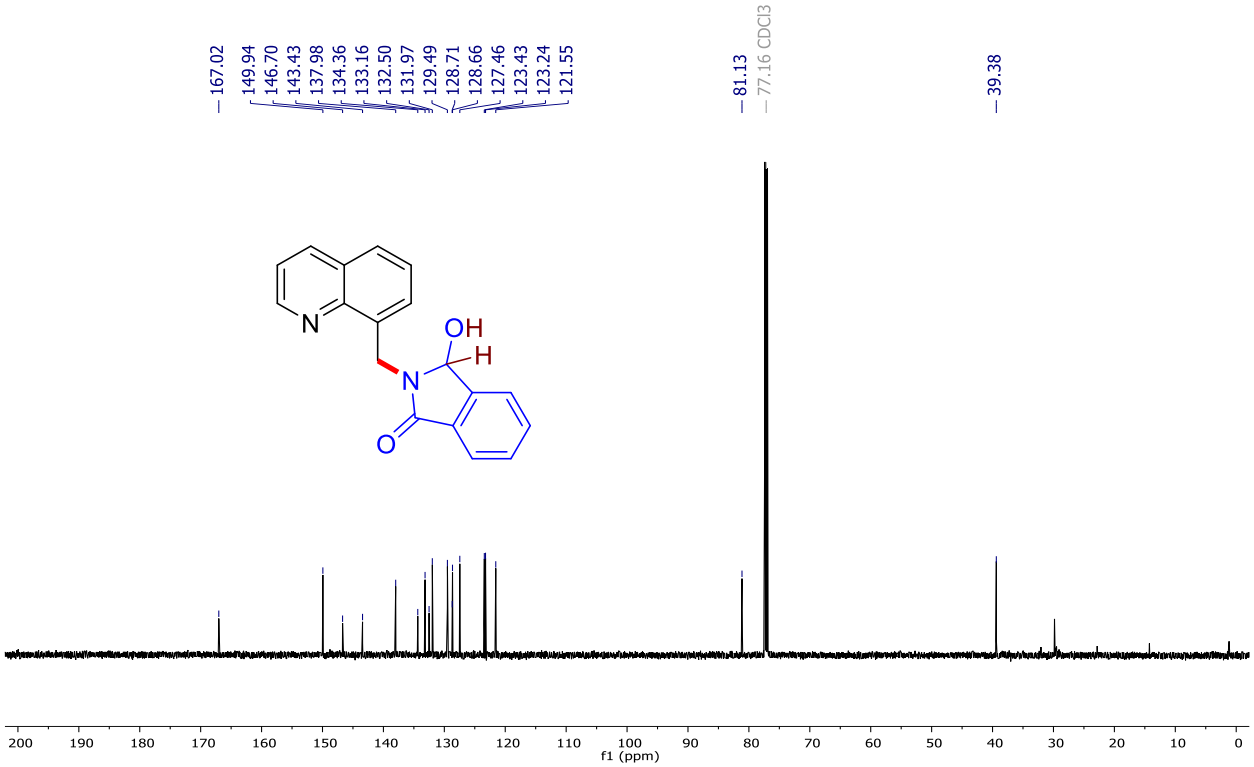


*3-hydroxy-2-(quinolin-8-ylmethyl)isoindolin-1-one (Scheme 5, Entry **8a**)*

^{1}H NMR (600 MHz)

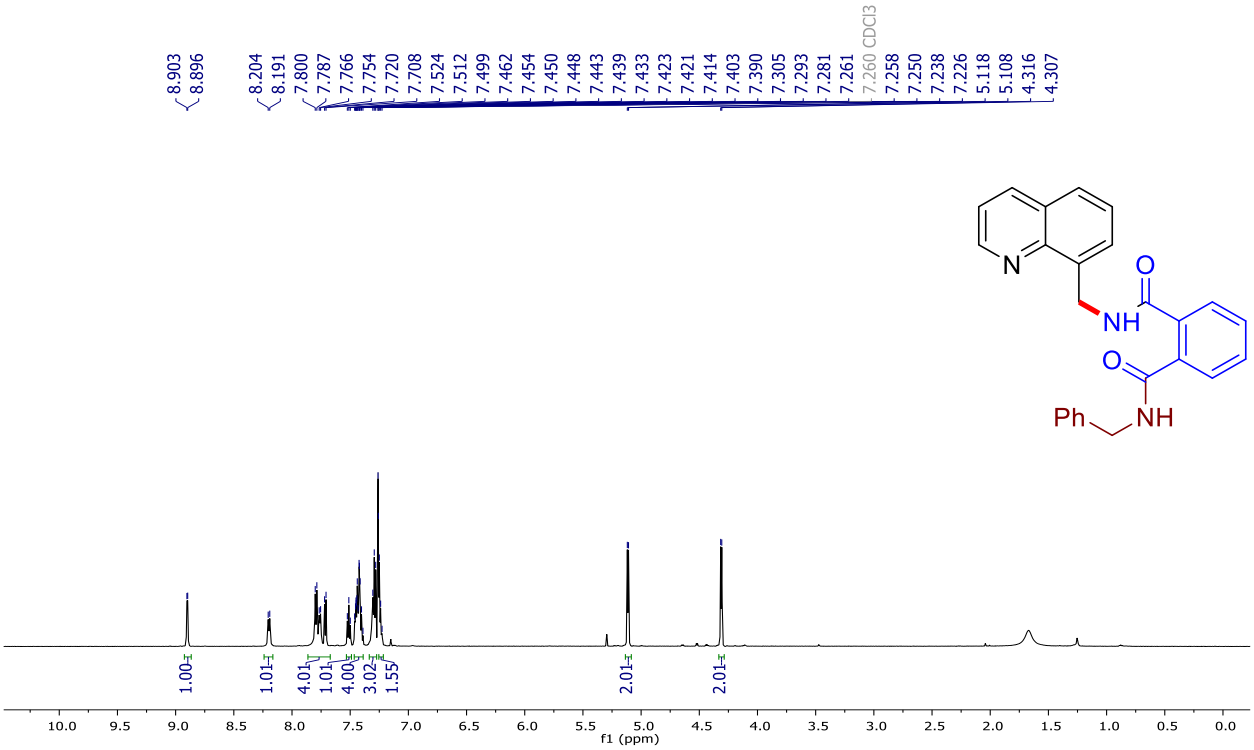


^{13}C{^{1}H} NMR *(*150 MHz*)*



*N^1-benzyl-N^2-(quinolin-8-ylmethyl)phthalamide (Scheme 7, Entry **8b**)*

^{1}H NMR (600 MHz)



^{13}C{^{1}H} NMR *(*150 MHz*)*