Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Iron and Base Catalyzed C(α)-Alkylation and One-pot Sequential Alkylation-Hydroxylation of Oxindoles with Secondary Alcohols

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Table of Contents

General experimental	S2
General experimental procedure for the C(α)-alkylation of oxindole	S3
General condition for gram scale synthesis of **P_1**	S5
General condition for one-pot alkylation-hydroxylation of oxindole	S6
General condition for gram scale synthesis of **P_{30}**	S6
Preparative scale for the determination of Green Metrics (CHEM21 green metrics toolkit, *E*-factor and Eco scale)	S7
NMR data of products and ^{1}H and ^{13}C NMR spectrum	S14
Mechanistic studies for C(α)-alkylation of oxindole	S73
Mechanistic studies for C(α)-alkylation and C-H hydroxylation of oxindole	S77
X-ray structure determination	S80
Zero Pass for method A-D using CHEM21 green metrics toolkit	S82
First Pass for method A-D using CHEM21 green metrics toolkit	S86
References	S94

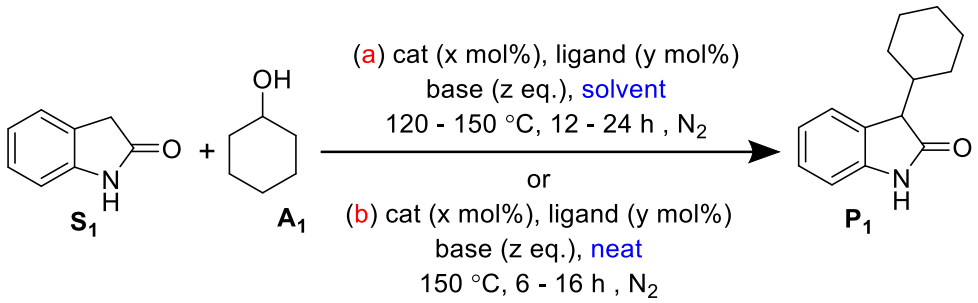
General experimental

All catalytic C(α)-alkylation of oxindole were performed under dry nitrogen atmosphere using standard Schlenk or glovebox (MBraun) techniques. Catalytic α-alkylation of oxindole were performed in Ace pressure tubes purchased from Sigma-Aldrich. Catalytic C-H hydroxylation of α-alkylated oxindole were performed in air. Analysis and purification of the products of alkylations and hydroxylation were carried out in air. Solvents were purchased from Merck and Spectrochem. For the air-sensitive experiments, solvents (1,4 dioxane, toluene, *p*-xylene, *t*AmOH and benzene) were distilled, degassed and stored over 3 Å molecular sieves. Deuterated solvents ($CDCl_3$ and DMSO-d_6) were purchased from Sigma-Aldrich. TLC was performed on Merck Kiesel gel 60, F254 plates with the layer thickness of 0.25 mm. Column chromatography was performed using silica gel (100-200 mesh) as stationary phase. $FeCl_2$, 1,10- phenanthroline, NaO*t*Bu, butylated hydroxytoluene (BHT), TEMPO, anisole(dry), all oxindole substrate and all secondary alcohols were purchased from Sigma Aldrich, Alfa Aesar and TCI Chemicals and used without further purification.

^1H and $^{13}C\{^1H\}$ NMR spectra were recorded at Bruker AV-400 and JEOL-400 (^1H at 400 MHz and ^{13}C at 101 MHz). ^1H and $^{13}C\{^1H\}$ NMR chemical shifts are referenced in parts per million (ppm) with respect to residual solvent peaks ($CDCl_3$: δ 7.26 and 77.16 ppm; DMSO-d6: 2.50 and 39.52 ppm). The coupling constants (*J*) are reported in hertz (Hz). The following abbreviations are used to describe multiplicity: s = singlet, brs = broad siglet, d = doublet, t = triplet, q = quadtrate, m = multiplate. GC was recorded using Shimadzu GC-2010 instrument. High resolution mass spectra (HRMS) were recorded on a Bruker micrOTOF-Q II Spectrometer. Gas chromatography (Thermo Scientific Trace 1110 GC) with a 60/80 Carboxen-1000 column, which was equipped with a thermal conductivity detector (TCD) was used to quantify gaseous products and high-purity Ar (99.999%) was used as carrier gas.

General experimental procedure for the C(α)-alkylation of oxindole:

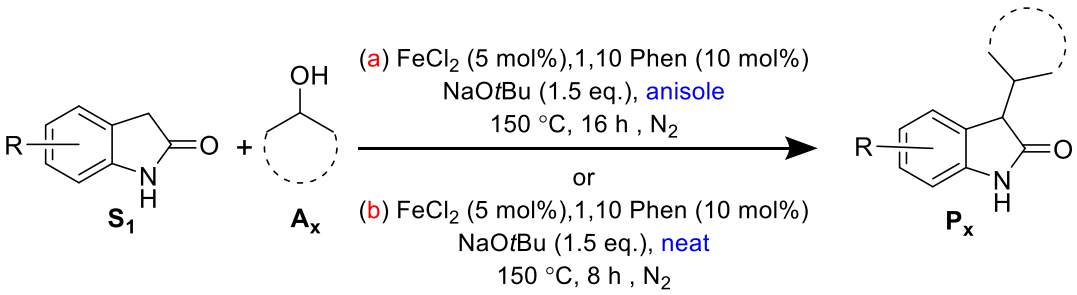
General condition for reaction optimization:



Procedure A: In a 15 mL dried pressure tube fitted with a magnetic stir bar, an appropriate amount of 2-oxindole (**S1**) (0.04 g, 0.30 mmol), cyclohexanol (**A1**) (0.06 g, 0.6 mmol), $FeCl_2$ (3 to 5 mol%), ligand (6 to 10 mol%), base (1 to 2 equivalent) and solvent (1 mL) were added successively under a nitrogen atmosphere. The reaction mixture was heated at an appropriate temperature in a preheated oil bath for the appropriate time. Thereafter the reaction mixture was cooled down to r.t. *n*-dodecane (0.051 g, 0.3 mmol) was added to the resultant mixture, and the product mixture was analyzed by GC. Occasionally the crude product was purified by column chromatography using silica as stationary phase and hexane as eluent.

Procedure B: In a 15 mL dried pressure tube fitted with a magnetic stir bar, an appropriate amount of 2-oxindole (**S1**) (0.04 g, 0.30 mmol), cyclohexanol (**A1**) (0.09 g, 0.9 mmol), $FeCl_2$ (5 mol%), ligand (10 mol%) and NaO*t*Bu (1.5 equivalent) were added successively under a nitrogen atmosphere in absence of any added solvent. The reaction mixture was heated at 150 ^{o}C temperature in a preheated oil bath for the appropriate time. Thereafter the reaction mixture was cooled down to r.t. *n*-dodecane (0.051 g, 0.3 mmol) was added to the resultant mixture, and the product mixture was analyzed by GC. Occasionally the crude product was purified by column chromatography using silica as stationary phase and hexane as eluent.

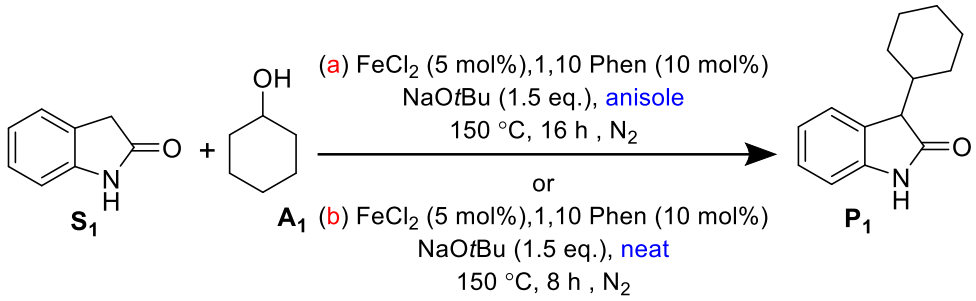
General condition for substrate screening:



Procedure A: In a 15 mL dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (**S1**) (0.3 mmol), secondary alcohols (**Ax**) (0.6 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10-phenanthroline (0.054 g, 10 mol%), NaO*t*Bu (0.45 mmol, 1.5 eq.), and anisole (1 mL) were successively added under a nitrogen atmosphere. Then the reaction mixture was heated at 150 °C in a preheated oil bath for 16 h. Thereafter, the reaction mixture was cooled down to r.t, and the crude product was purified by column chromatography (silica as stationary phase and a mixture of hexanes and ethyl acetate as eluent) to give a pure product.

Procedure B: In a 15 mL dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (**S1**) (0.3 mmol), secondary alcohols (**Ax**) (0.9 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10-phenanthroline (0.054 g, 10 mol%) and NaO*t*Bu (0.45 mmol, 1.5 eq.) were successively added under a nitrogen atmosphere in absence of any added solvent. Then the reaction mixture was heated at 150 °C in a preheated oil bath for 8 h. Thereafter, the reaction mixture was cooled down to r.t, and the crude product was purified by column chromatography (silica as stationary phase and a mixture of hexanes and ethyl acetate as eluent) to give a pure product.

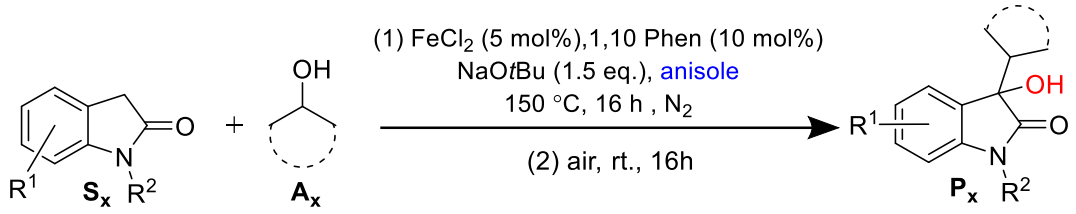
General condition for gram scale synthesis of 3-cyclohexylindolin-2-one (P_1):



Procedure A: In a 50 mL dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (1.065 g, 10.0 mmol), cyclohexanol (2.003 g, 20 mmol), $FeCl_2$ (0.063 g, 5 mol%), 1,10- phenanthroline (0.180 g, 10 mol%), NaO*t*Bu (1.440 g, 15 mmol) and anisole (10 mL) were added successively under a nitrogen atmosphere. The reaction mixture was heated at 150 °C in a preheated oil bath for 16 h. Thereafter, the reaction mixture was cooled down to r.t. and the crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give a white solid as pure product (2.002 g, 93%).

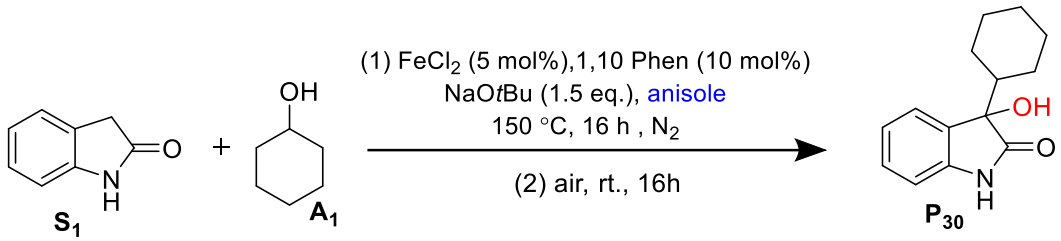
Procedure B: In a 50 mL dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (1.065 g, 10.0 mmol), cyclohexanol (3.004 g, 30 mmol), $FeCl_2$ (0.063 g, 5 mol%), 1,10- phenanthroline (0.180 g, 10 mol%) and NaO*t*Bu (1.440 g, 15 mmol) were added successively under a nitrogen atmosphere in absence of any added solvent. The reaction mixture was heated at 150 °C in a preheated oil bath for 8 h. Thereafter, the reaction mixture was cooled down to r.t. and the crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give a white solid as pure product (1.937 g, 90%).

General condition for one-pot alkylation-hydroxylation of oxindole:



In a 15 mL dried pressure tube fitted with a magnetic stir bar, a mixture of oxindole (0.3 mmol), secondary alcohols (0.6 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10- phenanthroline (0.054 g, 10 mol%), NaO*t*Bu (1.5 eq.) and anisole (1 mL) were successively added under a nitrogen atmosphere. Then the reaction mixture was heated at 150 °C in a preheated oil bath for 16 h. After cooling down to room temperature the reaction mixture was stirred under air for 16 h. Finally, concentrated by removing the solvent under vacuum, and the residue was purified by column chromatography with hexane/ethyl acetate (hexane/ethyl acetate = 7:3) as eluent to give the desired product.

General condition for gram scale synthesis of 3-cyclohexyl-3-hydroxyindolin-2-one (P_{30}):

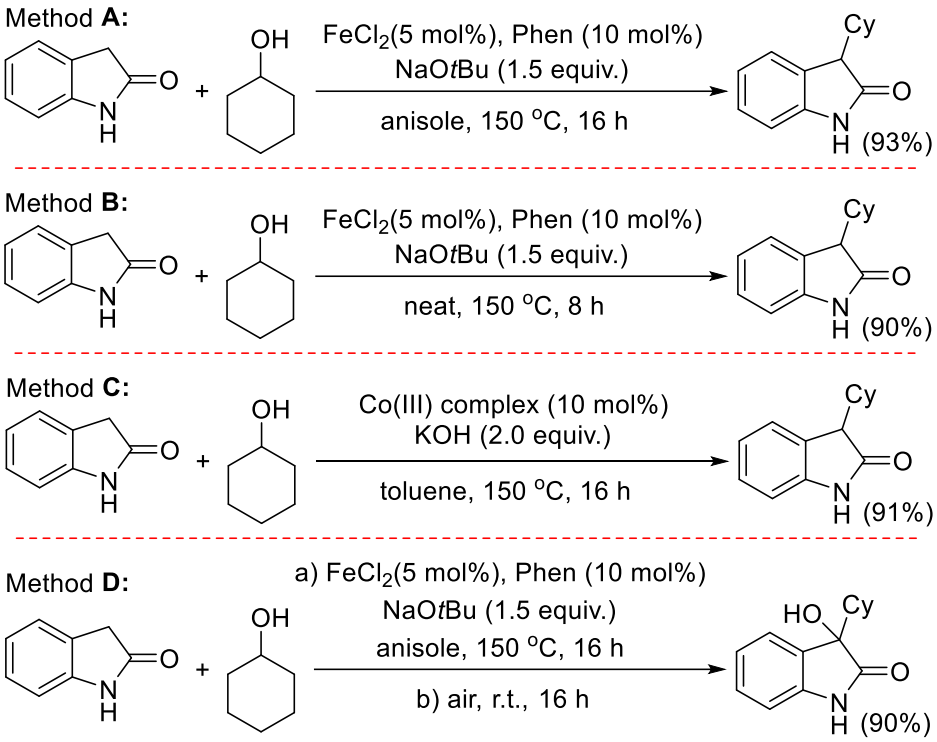


In a 100 mL dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (1.065 g, 10.0 mmol), cyclohexanol (2.003 g, 20 mmol), $FeCl_2$ (0.063 g, 5 mol%), 1,10-phenanthroline (0.180 g, 10 mol%), NaO*t*Bu (1.440 g, 15 mmol) and anisole (10 mL) were added successively under a nitrogen atmosphere. Then the reaction mixture was heated at 150 °C in a preheated oil bath for 16 h. After cooling down to room temperature the reaction mixture was stirred under air for 16 h. Finally, concentrated by removing the solvent under vacuum, and the residue was purified by column chromatography with hexane/ethyl acetate (hexane/ethyl acetate = 7:3) as eluent to give the desired product.

Preparative scale for the determination of Green Metrics:

Analysis of optimized reactions with CHEM21 green metrics toolkit: In order to evaluate the advantages and shortcomings of our two optimized catalytic protocols for the C(α)-alkylation of 2-oxindole with cyclohexanol (Method A and B), we analyzed them with the CHEM21 green metrics toolkit (Table S1). The results were also compared with the first report of base metal catalyzed 2-oxindole alkylation with secondary alcohol published by Sundararaju group (Method C).[S1] We also analyzed the optimized synthetic protocol for the one-pot alkylation-hydroxylation of 2-oxindole with cyclohexanol (Method D). In this context, we have employed green, amber, and red flags, representing acceptable, acceptable with concerns and unacceptable processes, respectively. All four methods A, B, C and D gave full conversion, excellent yield and complete selectivity and hence all methods received green flags for those metrics. Good atom economy and acceptable reaction mass efficiency were found in our optimized methods (Method A, B and D). In contrast, poor reaction mass efficiency was noted in Method C. We have used catalyst and green solvents (anisole or solvent-free) and thus, Method A, B and D earn green flags in solvent and catalyst metrics. However, Method C got amber flag for the use of hazardous toluene. Catalyst recovery is an important feature; however, catalysts could not be recovered in all the methods and they all received amber flags. We used most sustainable transition metal iron in these alkylations (Method A and B) and one-pot alkylation/hydroxylation (Method D) and they received green flags. In contrast, the alkylation method published by Sundararaju group (Method C) received amber flag for element metric due to use of cobalt. The reactions were performed in batches instead of continuous flow reactions. Thus, all methods earned amber flag. Our reaction conditions in terms of health & safety are in line with green metrics and received green flags. However, Method C got amber flag due to the use of toluene. So, we concluded that our optimized methods of alkylation have more green and sustainable features as compared to the past report.

Table S1: Comparison of optimized methods for the alkylation and one-pot alkylation-hydroxylation of 2-oxindole.



Metric	Meth. A	Meth. B	Meth. C	Meth. D
Yield	93	90	91	90
Conversion	100	100	100	100
Selectivity	100	100	100	100
Atom economy	65.4	65.4	77.1	64.0
Mass efficiency	40.8	28.7	44.0	42.4
Solvents	anisole	neat	toluene	anisole
Catalyst	Yes	Yes	Yes	Yes
Cat. recovery	No	No	No	No
Element	Fe	Fe	Co	Fe
Reactor	Batch	Batch	Batch	Batch
Health & safety				

***E*-factor for the synthesis of 3-cyclohexylindolin-2-one in presence of anisole as solvent (Method A, in anisole):**

Reagent 1:	Oxindole (10 mmol)	= 1.33 g
Reagent 2:	Cyclohexanol (20 mmol)	= 2.002 g
Base:	NaOtBu (15 mmol)	= 1.411 g
Catalyst:	$FeCl_2$ (5 mol %)	= 0.063 g
Ligand:	1,10-Phenantholine (10 mol %)	= 0.180 g
Reaction solvent:	anisole (10 mL, d= 0.995) [Solvent anisole (10 mL of reaction and 5 mL of washing) was placed in a round bottom flask and roughly 80 %; 11.94 g of anisole was recovered via solvent distillation process; waste = (14.925 – 11.94) = 3.01 g]	= 9.95 g
Product:	3-cyclohexylindolin-2-one (93% conversion; 2.002 g)	

$$E\text{-factor} = \frac{mass\ (waste)}{mass\ (product)}$$

$$= \frac{1.33\ g\ (Oxindole) + 2.002\ g\ (cyclohexanol) + 1.411\ g\ (base) + 0.243\ g\ (FeCl_2 + Ligand) + 3.01\ g\ (solvent\ anisole)}{2.002\ g\ (product)}$$

$$= 7.996\ g / 2.002\ g$$

$$= \mathbf{3.99\ kg\ waste / 1\ kg\ of\ product}$$

***E*-factor for the synthesis of 3-cyclohexylindolin-2-one under neat reaction condition (Method B, neat):**

Reagent 1:	Oxindole (10 mmol)	= 1.33 g
Reagent 2:	Cyclohexanol (40 mmol)	= 4.006 g
Base:	NaOtBu (15 mmol)	= 1.411 g
Catalyst:	$FeCl_2$ (5 mol %)	= 0.063 g
Ligand:	1,10-Phenantholine (10 mol %)	= 0.180 g
Reaction solvent:	Neat	= 00.00 g
Workup solvent:	ethyl acetate (5 x 5 mL) x 0.902 [Combined EtOAc was placed in a round bottom flask and roughly 95 %; 21.42 g of ethyl acetate was recovered via solvent distillation process; waste = (22.55 – 21.42) = 1.13 g]	= 22.55 g
Product:	3-cyclohexylindolin-2-one (90% conversion; 1.937 g)	

$$E\text{-factor} = \frac{mass\ (waste)}{mass\ (product)}$$

$$= \frac{1.33\ g\ (Oxindole) + 4.006\ g\ (cyclohexanol) + 1.411\ g\ (base) + 0.243\ g\ (FeCl_2 + Ligand) + 1.13\ g\ (ethyl\ acetate\ during\ isolation\ of\ the\ product)}{1.937\ g\ (product)}$$

$$= 8.12\ g / 1.937\ g$$

$$= \mathbf{4.19\ kg\ waste / 1\ kg\ of\ product}$$

***E*-factor for the synthesis of 3-cyclohexyl-3-hydroxyindolin-2-one (Method D, in anisole):**

Reagent 1:	Oxindole (10 mmol)	= 1.33 g
Reagent 2:	Cyclohexanol (20 mmol)	= 2.002 g
Base:	NaOtBu (15 mmol)	= 1.411 g
Catalyst:	$FeCl_2$ (5 mol %)	= 0.063 g
Ligand:	1,10-Phenantholine (10 mol %)	= 0.180 g
Reaction solvent:	anisole (10 mL, d= 0.995) [Solvent anisole (10 mL of reaction and 5 mL of washing) was placed in a round bottom flask and roughly 83 %; 12.39 g of anisole was recovered via solvent distillation process; waste = (14.925 – 12.39) = 2.54 g]	= 9.95 g
Product:	3-cyclohexyl-3-hydroxyindolin-2-one (90% conversion; 2.081 g)	

$$E\text{-factor} = \frac{mass\ (waste)}{mass\ (product)}$$

$$= \frac{1.33\ g\ (Oxindole) + 2.002\ g\ (cyclohexanol) + 1.411\ g\ (base) + 0.243\ g\ (FeCl_2 + Ligand) + 2.54\ g\ (solvent\ anisole)}{2.081\ g\ (product)}$$

$$= 7.526\ g / 2.081\ g$$

$$= \mathbf{3.61\ kg\ waste / 1\ kg\ of\ product}$$

Table S2. Calculation of Eco Scale for the synthesis of 3-cyclohexylindolin-2-one (Method A in anisole)

Eco Scale = 100 – Sum of individuals penalties

Score on EcoScale: >75, Excellent; >50, Acceptable; <50, Inadequate

A) Calculations of Penalty Points

Parameters		**Penalty Points**
1. Yield (100 – % yield)/2 = (100 – 93)/2 = 3.5	**=**	**3.5**
2. Price of reaction components		
(To obtain 10 mmol of 3-cyclohexylindolin-2-one as end product)		
(a) Oxindole = 12.18 mmol = 1.691 g = USD 4.90		
(b) Cyclohexanol (24.36 mmol) = 2.536 mL = USD 0.08		
(c) NaOtBu (18.27 mmol) = 1.754 g = 0.45 USD		
(d) $FeCl_2$ = 5 mol % = 0.077 g = USD 0.085		
(e) 1,10-Phenantholine (10 mol %) = 0.219 g = 0.64 USD		
(f) solvent anisole (10 mL) = 1 USD		
Total cost for the synthesis of 3-cyclohexylindolin-2-one = USD 7.155		
Thus, inexpensive since it is <USD 10	=	0
3. Safety		
1,10-Phenantholine (T)	=	5
4. Technical Setup		
Glove Box	=	3
5. Temperature and time		
Heating, >1 h	=	3
6. Workup and purification		
Removal of solvent with bp <150°C	=	0
Classical chromatography	=	10
Total Penalty Points	**=**	**24.5**

B) EcoScale calculation:

EcoScale score = 100 – 24.5 = 75.5 (>75; it is an excellent synthetic process)

Table S3. Calculation of EcoScale for the synthesis of 3-cyclohexylindolin-2-one in presence of anisole as solvent (Method B, under neat)

Eco Scale = 100 – Sum of individuals penalties

Score on EcoScale: >75, Excellent; >50, Acceptable; <50, Inadequate

A) Calculations of Penalty Points

Parameters		**Penalty Points**
1. Yield (100 – % yield)/2 = (100 – 90)/2 = 5	=	**5**
2. Price of reaction components		
(To obtain 10 mmol of 3-cyclohexylindolin-2-one as end product)		
(a) Oxindole = 12.18 mmol = 1.691 g = USD 4.90		
(b) Cyclohexanol (48.72 mmol) = 5.072 mL = USD 0.16		
(c) NaOtBu (18.27 mmol) = 1.754 g = 0.45 USD		
(d) $FeCl_2$ = 5 mol % = 0.077 g = USD 0.085		
(e) 1,10-Phenantholine (10 mol %) = 0.219 g = 0.64 USD		
Total cost for the synthesis of 3-cyclohexylindolin-2-one = USD 6.235		
Thus, inexpensive since it is <USD 10	=	0
3. Safety		
1,10-Phenantholine (T)	=	5
4. Technical Setup		
Glove Box	=	3
5. Temperature and time		
Heating, >1 h	=	3
6. Workup and purification		
Removal of solvent with bp <150°C	=	0
Classical chromatography	=	10
Total Penalty Points	**=**	**26**

B) EcoScale calculation:

EcoScale score = 100 – 26 = 74 (>50; it is an acceptable synthetic process)

Table S4. Calculation of Eco Scale for the synthesis of 3-cyclohexyl-3-hydroxyindolin-2-one (Method D, in anisole)

Eco Scale = 100 – Sum of individuals penalties

Score on EcoScale: >75, Excellent; >50, Acceptable; <50, Inadequate

A) Calculations of Penalty Points

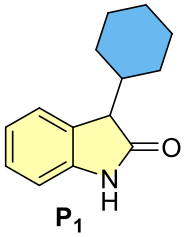
Parameters		**Penalty Points**
1. Yield (100 – % yield)/2 = (100 – 90)/2 = 5	**=**	**5**
2. Price of reaction components		
(To obtain 10 mmol of 3-cyclohexylindolin-2-one as end product)		
(a) Oxindole = 12.18 mmol = 1.691 g = USD 4.90		
(b) Cyclohexanol (36.54 mmol) = 3.804 mL = USD 0.12		
(c) NaOtBu (18.27 mmol) = 1.754 g = 0.45 USD		
(d) $FeCl_2$ = 5 mol % = 0.077 g = USD 0.085		
(e) 1,10-Phenantholine (10 mol %) = 0.219 g = 0.64 USD		
(f) solvent anisole (10 mL) = 1 USD		
Total cost for the synthesis of 3-cyclohexylindolin-2-one = USD 7.195		
Thus, inexpensive since it is <USD 10	=	0
3. Safety		
1,10-Phenantholine (T)	=	5
4. Technical Setup		
Glove Box	=	3
5. Temperature and time		
Heating, >1 h	=	3
6. Workup and purification		
Removal of solvent with bp <150°C	=	0
Classical chromatography	=	10
Total Penalty Points	**=**	**26**

B) EcoScale calculation:

EcoScale score = 100 – 26 = 74 (>50; it is an acceptable synthetic process)

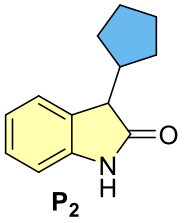
NMR data of pure alkylated products:

The following products are obtained by α-alkylation of fluorene with primary and secondary alcohols using the standard catalytic protocol. Known compounds are characterized by ^{1}H and $^{13}C\{^{1}H\}$ NMR spectroscopies and new compounds are characterized by ^{1}H and $^{13}C\{^{1}H\}$ NMR spectroscopies and HRMS:



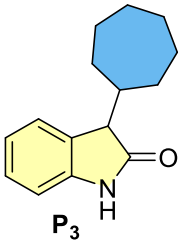
P_1

3-cyclohexylindolin-2-one (P_1):[1] The crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give pure product **P_1** as a colourless solid (0.055 g, 93%).^{1}H NMR (400 MHz, $CDCl_3$) δ 9.04 (brs, 1H), 7.30 – 7.26 (m, 1H), 7.22 (t, J = 7.7 Hz, 1H), 7.03 (t, J = 7.5 Hz, 1H), 6.93 (d, J = 7.6 Hz, 1H), 3.40 (d, J = 3.2 Hz, 1H), 2.21 – 2.15 (m, 1H), 1.83 – 1.43 (m, 6H), 1.35 – 1.10 (m, 4H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 180.4, 142.1, 128.8, 127.8, 124.8, 122.1, 109.7, 52.2, 41.0, 30.5, 28.5, 26.8, 26.4, 26.2.



P_2

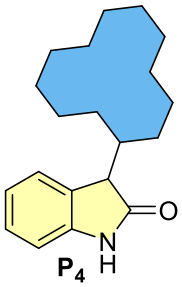
3-cyclopentylindolin-2-one (P_2):[1] The crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give pure product **P_2** as a colourless solid (0.054 g, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.30 (s, 1H), 7.19 (d, J = 7.5 Hz, 1H), 7.12 (t, J = 7.7 Hz, 1H), 6.91 (t, J = 7.5 Hz, 1H), 6.84 (d, J = 7.7 Hz, 1H), 3.45 (d, J = 5.2 Hz, 1H), 2.46 – 2.33 (m, 1H), 1.90 – 1.70 (m, 1H), 1.73 – 1.63 (m, 1H), 1.62 – 1.38 (m, 5H), 1.30 – 1.16 (m, 1H). $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 180.9, 142.1, 129.2, 127.9, 124.8, 122.1, 109.9, 49.3, 42.0, 30.0, 28.4, 25.2, 25.1.



P_3

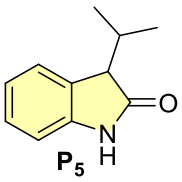
3-cycloheptylindolin-2-one (P_3):[1] The crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give pure product **P_3** as a colourless solid (0.06 g, 87%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (s, 1H), 7.20 (d, J = 9.7 Hz, 1H), 7.13 (t, J = 7.7 Hz, 1H),

6.93 (t, J = 7.5 Hz, 1H), 6.83 (d, J = 7.7 Hz, 1H), 3.38 (d, J = 2.8 Hz, 1H), 2.29 – 2.21 (m, 1H), 1.75 – 1.66 (m, 2H), 1.61 – 1.48 (m, 4H), 1.43 – 1.32 (m, 4H), 1.30 – 1.12 (m, 2H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 180.4, 142.1, 128.9, 127.9, 124.6, 122.2, 109.7, 53.3, 42.4, 32.7, 30.5, 27.9, 27.6, 27.4, 27.1.



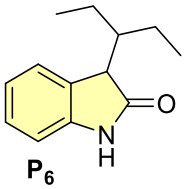
P4

3-cyclododecylindolin-2-one (P4):[3] The crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give pure product **P4** as pale yellow oil (0.073 g, 82%). ^1H NMR (400 MHz, $CDCl_3$) δ 8.94 (s, 1H), 7.30 – 7.18 (m, 2H), 7.02 (t, J = 7.2 Hz, 1H), 6.92 (d, J = 7.7 Hz, 1H), 3.52 (d, J = 2.7 Hz, 1H), 1.71 – 1.63 (m, 1H), 1.52 – 1.24 (m, 21H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 180.7, 142.1, 129.3, 127.7, 124.5, 122.1, 109.8, 49.3, 35.3, 27.7, 26.5, 24.3, 24.2, 24.1, 23.4, 23.2, 22.9, 22.7.



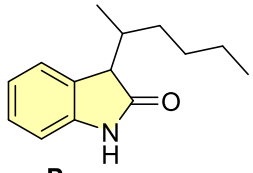
P5

3-isopropylindolin-2-one (P5):[1] The crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give pure product **P5** as a pale yellow solid (0.039 g, 75%). ^1H NMR (400 MHz, $CDCl_3$) δ 8.98 (s, 1H), 7.28 (d, J = 6.1 Hz, 1H), 7.23 (t, J = 7.8 Hz, 1H), 7.03 (t, J = 7.5 Hz, 1H), 6.93 (d, J = 7.7 Hz, 1H), 3.43 (d, J = 3.4 Hz, 1H), 2.62 – 2.43 (m,1H), 1.15 (d, J = 7.0 Hz, 3H), 0.94 (d, J = 6.8 Hz, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 180.3, 142.2, 128.5, 127.92, 124.8, 122.2, 109.8, 52.3, 30.9, 20.0, 18.1.



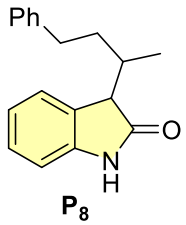
P6

3-(pentan-3-yl)indolin-2-one (P6):[1] The crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give pure product **P6** as a pale yellow oil (0.051 g, 84%). ^1H NMR (400 MHz, $CDCl_3$) δ 8.93 (s, 1H), 7.22 (t, J = 7.2 Hz, 2H), 7.02 (t, J = 7.5 Hz, 1H), 6.93 (d, J = 7.9 Hz, 1H), 3.63 (d, J = 2.6 Hz, 1H), 2.10 – 1.99 (m, 1H), 1.72 – 1.62 (m, 1H), 1.53 – 1.35 (m, 2H), 1.30 – 1.17 (m, 1H), 1.05 (t, J = 7.4 Hz, 3H), 0.89 (t, J = 7.4 Hz, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 181.0, 142.0, 128.8, 127.8, 124.6, 122.2, 109.8, 48.4, 44.3, 24.5, 23.6, 12.5, 12.3.



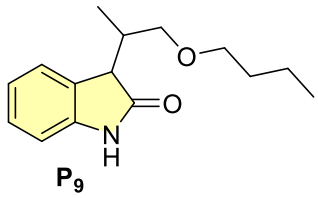
P7

3-(hexan-2-yl)indolin-2-one (P7):[1] The crude product was purified by column chromatography (hexane/EtOAc 8.5:1.5 as eluent) to give pure product **P7** as a pale yellow oil (0.053 g, 81%). Two isomers ratio 1.45:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.20 (brs, 1H, m_j), 9.09 (s, 1H, m_n), 7.27 – 7.19 (m, 3H), 7.03 (t, *J* = 7.5 Hz, 2H), 6.93 (t, *J* = 7.0 Hz, 2H), 3.52 (d, *J* = 3.1 Hz, 1H, m_j), 3.48 (d, *J* = 2.9 Hz, 1H, m_n), 2.43 – 2.34 (m, 1H, m_j), 2.33 – 2.23 (m, 1H, m_n), 1.65 – 1.57 (m, 1H, m_n), 1.55 – 1.48 (m, 1H, m_j), 1.45 – 1.38 (m, 3H, m_n), 1.32 – 1.25 (m, 5H, m_j), 1.04 (d, *J* = 6.9 Hz, 2H, m_n), 0.95 (t, *J* = 6.9 Hz, 3H, m_j), 0.89 (t, *J* = 6.9 Hz, 2H, m_n), 0.79 (d, *J* = 6.8 Hz, 3H, m_j). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 181.0, 180.3, 142.3, 142.1, 129.2, 128.1, 127.9, 127.8, 124.9, 124.4, 122.2, 122.1, 109.9, 109.8, 51.5, 51.0, 35.9, 35.5, 34.3, 32.5, 29.9, 29.8, 22.8, 17.0, 15.4, 14.2.



P8

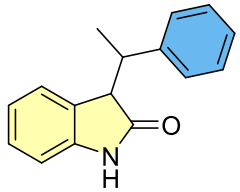
3-(4-phenylbutan-2-yl)indolin-2-one (P8): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P8** as a pale yellow oil (0.065 g, 82%). Two isomers ratio 1.66:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.33 (m_j), 9.21 (m_n) (two s, 1H), 7.24 – 7.04 (m, 7H), 6.95 – 6.77 (m, 2H), 3.44 (m_j), 3.41 (m_n) (two d, *J* = 3 Hz, 1H), 2.77 – 2.45 (m, 2H), 2.39 – 2.19 (m, 1H), 1.89 – 1.47 (m, 2H), 0.98 (m_j), 0.79 (m_n) (two d, *J* = 6.8 Hz, 3H).^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 180.8, 180.2, 142.3, 142.2, 142.1, 128.9, 128.6, 128.5, 128.4, 128.3, 128.0, 127.9, 127.8, 126.0, 125.8, 124.8, 124.3, 122.2, 122.1, 110.0, 109.9, 51.2, 51.1, 36.5, 35.4, 35.3, 34.7, 34.1, 33.9, 16.9, 15.3. HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ Calcd for $[C_{18}H_{20}NO]$ 266.1545; Found 266.1544.



P9

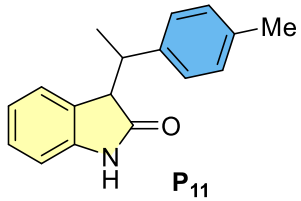
3-(1-butoxypropan-2-yl)indolin-2-one (P9): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P9** as a pale yellow oil (0.058 g, 79%). Two isomers ratio 1:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.13 (m_j), 9.00 (m_n) (two brs, 1H), 7.13 (dt, *J* = 7.8, 5.7 Hz, 2H), 6.93 (t, *J* = 7.5 Hz, 1H), 6.83 (t, *J* = 8.5 Hz, 1H), 3.70 (m_j), 3.56 (m_n) (two d, *J* = 2.8 Hz, 1H), 3.53 – 3.20 (m, 4H), 2.73 – 2.46 (m, 1H), 1.58 – 1.38 (m, 2H), 1.37 – 1.17 (m, 2H), 0.87 (m_j), 0.84 (m_j)

(one d, J = 7.1 Hz, one t, J = 8 Hz, 3H), 0.80 (m_n), 0.62 (m_n) (one d, J = 7.1 Hz, one t, J = 8 Hz 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 181.0, 180.0, 142.4, 142.3, 128.8, 127.9, 127.8, 127.5, 125.3, 124.1, 122.1, 109.9, 109.7, 73.1, 72.7, 71.0, 70.9, 48.2, 47.9, 36.2, 35.2, 32.0, 31.8, 19.5, 19.4, 14.1, 14.0, 13.7, 12.0. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{15}H_{22}NO_2]$ 248.1651; Found 248.1674.



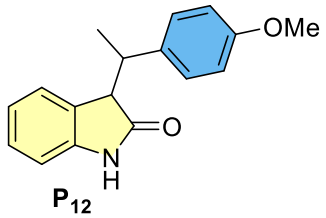
P10

3-(1-phenylethyl)indolin-2-one (P10):[1] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P10** as a colourless oil (0.055 g, 78%). Two isomers ratio 1.79:1. ^1H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer): δ 8.93 (brs, 1H, m_j), 8.56 (brs, 1H, m_n), 7.41 – 7.33 (m, 4H, m_j), 7.32 – 7.26 (m, 1H, m_n), 7.22 – 7.15 (m, 3H, m_j), 7.13 – 7.05 (m, 2H, m_n), 6.97 (t, J = 7.5 Hz, 1H, m_n), 6.92 – 6.85 (m, 2H, m_j), 6.78 (d, J = 7.7 Hz, 1H, m_n), 6.53 (d, J = 7.4 Hz, 1H, m_j), 3.87 – 3.82 (m, 1H, m_j), 3.81 (d, J = 3.7 Hz, 1H, m_j), 3.69 (d, J = 5.5 Hz, 1H, m_n), 3.62 – 3.44 (m, 1H, m_n), 1.66 (d, J = 7.2 Hz, 3H, m_n), 1.21 (d, J = 7.0 Hz, 3H, m_j). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 179.7, 179.3, 142.9, 142.2, 142.1, 141.8, 128.5, 128.2, 128.1, 128.0, 127.9, 127.0, 126.9, 126.8, 125.2, 122.0, 109.7, 53.1, 52.5, 41.9, 39.6, 19.4, 13.6.



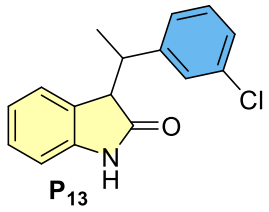
P11

3-(1-(p-tolyl)ethyl)indolin-2-one (P11):[1] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P11** as a pale yellow oil (0.056 g, 75%). Two isomers ratio 2.22:1. ^1H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 8.48 (s, 1H, m_j), 8.12 (s, 1H, m_n), 7.21 – 7.14 (m, 5H), 7.08 (d, J = 7.4 Hz, 1H, m_n), 6.96 (s, 2H), 6.86 (t, J = 7.9 Hz, 2H), 6.74 (d, J = 7.8 Hz, 1H, m_n), 6.54 (d, J = 7.4 Hz, 1H, m_j), 3.79 – 3.70 (m, 2H), 3.64 (d, J = 5.4 Hz, 1H, m_n), 3.55 – 3.42 (m, 1H, m_n), 2.36 (s, 3H, m_j), 2.24 (s, 3H, m_n), 1.60 (d, J = 7.2 Hz, 3H, m_n), 1.16 (d, J = 6.9 Hz, 3H, m_j). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 179.4, 179.0, 142.0, 141.7, 139.8, 139.2, 136.3, 129.2, 128.8, 128.3, 128.1, 128.0, 127.9, 127.8, 127.2, 125.3, 122.0, 109.6, 53.1, 52.5, 41.6, 39.2, 21.2, 21.1, 19.5, 13.7.



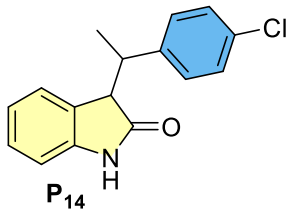
P12

3-(1-(4-methoxyphenyl)ethyl)indolin-2-one (P_{12}):[3] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{12}** as a pale yellow oil (0.064 g, 80%). Two isomers ratio 1.56:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.13 (brs, 1H, m_j), 8.76 (brs, 1H, m_n), 7.24 (d, J = 8.6 Hz, 2H, m_j), 7.19 (t, J = 7.7 Hz, 1H, m_j), 7.13 (d, J = 7.4 Hz, 1H, m_n), 7.02 – 6.95 (m, 2H, m_n), 6.93 – 6.86 (m, 4H, m_j), 6.78 (d, J = 7.7 Hz, 1H, m_n), 6.68 (d, J = 8.6 Hz, 1H, m_j), 6.56 (d, J = 7.4 Hz, 1H, m_n), 3.84 (s, 3H, m_j), 3.81 – 3.77 (m, 1H, m_j), 3.76 (d, J = 3.2 Hz, 1H, m_j), 3.72 (s, 3H, m_n), 3.65 (d, J = 5.3 Hz, 1H, m_n), 3.56 – 3.48 (m, 1H, m_n) 1.63 (d, J = 7.2 Hz, 2H, m_n), 1.19 (d, J = 6.8 Hz, 3H, m_j). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 179.9, 179.6, 158.4, 158.3, 142.2, 141.9, 134.8, 134.2, 129.0, 128.9, 128.2, 128.1, 128.0, 127.2, 125.2, 125.2, 122.0, 113.8, 113.4, 109.8, 109.7, 55.4, 55.2, 53.3, 52.8, 41.1, 38.9, 19.6, 13.9.



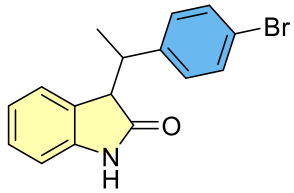
P_{13}

3-(1-(3-chlorophenyl)ethyl)indolin-2-one (P_{13}): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{13}** as a pale yellow oil (0.055 g, 68%). Two isomers ratio 1.75:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer): δ 9.03 (s, 1H, m_j), 8.70 (s, 1H, m_n), 7.36 – 7.25 (m, 3H), 7.23 – 7.18 (m, 2H), 7.16 – 7.06 (m, 2H), 7.04 – 6.97 (m, 1H), 6.91 (t, J = 7.5 Hz, 2H), 6.81 (d, J = 7.7 Hz, 1H), 6.57 (d, J = 7.5 Hz, 1H), 3.84 – 3.72 (m, 2H, m_j), 3.67 (d, J = 5.1 Hz, 1H, m_n), 3.59 – 3.46 (m, 1H, m_n), 1.62 (d, J = 7.2 Hz, 3H, m_n), 1.21 (d, J = 6.7 Hz, 3H, m_j).^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 179.4, 179.0, 145.0, 144.2, 142.1, 141.8, 134.4, 133.8, 129.7, 129.3, 128.4, 128.3, 128.3, 128.2, 127.7, 127.0, 126.7, 126.4, 126.2, 125.1, 125.0, 122.2, 109.9, 52.8, 52.3, 41.6, 39.4, 19.0, 13.7. HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ Calcd for $[C_{16}H_{15}ClNO]$ 272.0842; Found 272.0852.



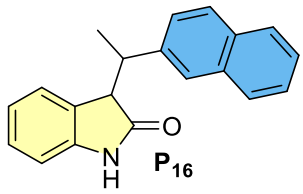
P_{14}

3-(1-(4-chlorophenyl)ethyl)indolin-2-one (P_{14}):[3] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{14}** as a pale yellow oil (0.058 g, 71%). Two isomers ratio 1.47:1 ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.18 (brs, 1H, m_j), 8.85 (brs, 1H, m_n), 7.32 (d, J = 8.5 Hz, 2H, m_j), 7.25 (d, J = 8.5 Hz, 2H, m_n), 7.19 (d, J = 7.9 Hz, 2H, m_j), 7.09 (d, J = 8.4 Hz, 1H, m_n), 7.01 (d, J = 7.7 Hz, 2H, m_j), 6.91 (t, J = 7.7 Hz, 2H, m_j), 6.80 (d, J = 7.7 Hz, 1H, m_n), 6.59 (d, J = 7.4 Hz, 1H, m_j), 3.82 – 3.73 (m, 2H, m_j), 3.67 (d, J = 5.0 Hz, 1H, m_n), 3.61 – 3.52 (m, 1H, m_n), 1.62 (d, J = 7.2 Hz, 3H, m_n), 1.22 (d, J = 6.9 Hz, 3H, m_j). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 179.6, 179.2, 142.1, 141.9, 141.3, 140.4, 132.6, 129.4, 129.3, 128.6, 128.3, 128.2, 128.1, 127.7, 126.9, 125.1, 125.0, 122.1, 110.0, 109.9, 52.9, 52.5, 41.2, 39.2, 19.2, 14.0.



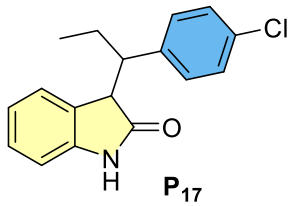
P15

3-(1-(4-bromophenyl)ethyl)indolin-2-one (P15):[1] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P15** as a yellow oil (0.068 g, 72%). Two isomers ratio 1.43:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.04 (s, 1H, m_j), 8.70 (s, 1H, m_n), 7.36 (d, J = 8.4 Hz, 1H,), 7.29 – 7.22 (m, 1H), 7.14 (d, J = 8.4 Hz, 2H), 7.11 – 7.02 (m, 4H), 6.91 (t, J = 7.5 Hz, 1H, m_n), 6.85 – 6.76 (m, 3H), 6.69 (d, J = 7.7 Hz, 1H, m_n), 6.49 (d, J = 7.4 Hz, 1H, m_j), 3.79 – 3.64 (m, 1H, m_j), 3.62 (d, J = 6.1 Hz, 1H, m_j), 3.56 (d, J = 5.1 Hz, 1H, m_n), 3.49 – 3.40 (m, 1H, m_n), 1.51 (d, J = 7.2 Hz, 3H, m_n), 1.11 (d, J = 7.1 Hz, 3H, m_j). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 179.5, 179.2, 142.1, 141.9, 141.8, 140.9, 131.5, 131.1, 129.8, 129.7, 128.4, 128.2, 128.1, 127.7, 126.8, 125.1, 125.0, 122.1, 120.7, 120.6, 110.0, 109.9, 52.8, 52.4, 41.2, 39.3, 19.2, 13.9.



P16

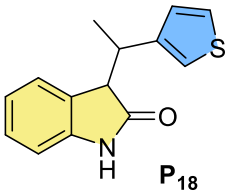
3-(1-(naphthalen-2-yl)ethyl)indolin-2-one (P16):[1] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P16** as a brown viscous oil (0.070 g, 81%). Two isomers ratio 1.5:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.28 (s, 1H, m_j), 8.82 (s, 1H, m_n), 7.92 – 7.83 (m, 3H), 7.78 – 7.72 (m, 1H), 7.71 – 7,64 (m, 1H), 7.58 – 7.50 (m, 3H), 7.45 – 7.39 (m, 1H), 7.25 (d, J = 8.4 Hz, 1H), 7.19 (t, J = 7.7 Hz, 1H), 7.14 (d, J = 7.4 Hz, 1H, m_n), 6.98 (t, J = 7.5 Hz, 1H, m_n), 6.92 (d, J = 7.7 Hz, 1H, m_j), 6.84 (t, J = 7.5 Hz, 1H, m_j), 6.76 (d, J = 7.7 Hz, 1H, m_n), 6.49 (d, J = 7.5 Hz, 1H, m_j), 4.04 – 3.96 (m, 1H, m_j), 3.93 (d, J = 3.6 Hz, 1H, m_j), 3.79 (d, J = 3.6 Hz, 1H, m_n), 3.76 – 3.69 (m, 1H, m_n), 1.75 (d, J = 7.1 Hz, 3H, m_n), 1.31 (d, J = 7.0 Hz, 3H, m_j). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 179.9, 179.4, 142.2, 141.9, 140.4, 139.8, 133.4, 133.3, 132.5, 132.4, 128.3, 128.2, 128.1, 128.0, 127.9, 127.7, 127.6, 127.5, 127.0, 126.9, 126.7, 126.3, 126.2, 126.1, 125.9, 125.8, 125.5, 125.2, 125.1, 122.0, 109.9, 52.9, 52.5, 42.1, 39.6, 19.4, 13.5.



P17

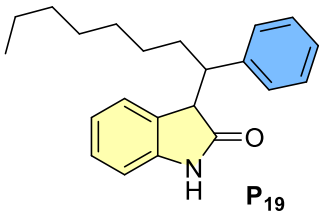
3-(1-(4-chlorophenyl)propyl)indolin-2-one (P17): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P17** as a yellowish solid (0.066 g, 78%). Two isomers ratio 1.5:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.10 (m_n), 8.83(m_j) (two s, 1H), 7.19 – 7.02 (m, 3H), 6.98 – 6.80 (m, 3H), 6.78 – 6.64 (m, 2H), 3.62 (m_j), 3.56 (m_n) (two d, J = 3.5 Hz, 1H), 3.29 – 3.16 (m, 1H), 2.13 – 1.67

(m, 2H), 0.84 (m_j), 0.76 (m_n) (two t, J = 7.3 Hz, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 179.6, 179.5, 142.0, 141.8, 139.1, 138.4, 132.5, 132.4, 130.0, 129.9, 128.4, 128.3, 128.1, 128.0, 127.8, 127.5, 125.2, 124.7, 122.1, 122.0, 110.0, 109.9, 52.2, 51.3, 48.6, 48.1, 26.4, 22.7, 12.5, 12.4. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{17}H_{17}ClNO]$ 286.0999; Found 286.0982.



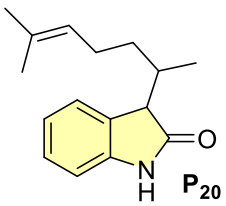
P18

3-(1-(thiophen-3-yl)ethyl)indolin-2-one (P18): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P18** as a yellowish oil (0.054 g, 74%). Two isomers ratio 2.32:1. ^1H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.24 (s, 1H, m_j), 8.90 (s, 1H, m_n), 7.27 – 7.24 (m, 1H, m_j), 7.13 – 7.06 (m, 2H, m_j), 7.02 (d, J = 4.6 Hz, 1H, m_n), 6.97 (d, J = 4.8 Hz, 1H, m_n), 6.91 (d, J = 4.6 Hz, 1H, m_j), 6.79 (dd, J = 16.3, 7.6 Hz, 2H, m_j), 6.69 (dd, J = 15.9, 6.3 Hz, 1H, m_n), 6.37 (d, J = 7.4 Hz, 1H, m_j), 3.79 – 3.70 (m, 2H, m_j), 3.68 – 3.60 (m, 1H, m_n), 3.55 (d, J = 4.6 Hz, 1H, m_n), 1.52 (d, J = 7.2 Hz, 3H, m_n), 1.07 (d, J = 6.8 Hz, 3H, m_j). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 179.8, 179.6, 144.4, 142.8, 142.2, 142.0, 128.2, 128.1, 128.1, 127.5, 127.3, 127.0, 125.9, 125.0, 124.9, 124.8, 122.1, 122.0, 121.3, 121.0, 109.9, 109.8, 52.5, 52.4, 37.3, 35.7, 19.6, 14.2. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{14}H_{14}NOS]$ 244.0796; Found 244.0784.



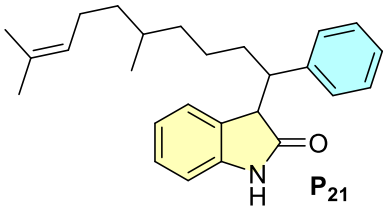
P19

3-(1-phenyloctyl)indolin-2-one (P19): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P19** as a pale yellowish oil (0.068 g, 71%). Two isomers ratio 1.47:1. ^1H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 8.34 (s, 1H, m_n), 7.99 (s, 1H, m_j), 7.24 – 7.12 (m, 3H), 7.08 (t, J = 7.5 Hz, 2H, m_j), 6.99 (t, J = 7.5 Hz, 1H, m_n), 6.92 (d, J = 6.6 Hz, 1H, m_n), 6.88 (d, J = 8.0 Hz, 2H, m_j), 6.81 (d, J = 8.0 Hz, 2H, m_j), 6.78 (d, J = 7.8 Hz, 1H, m_n), 6.70 (d, J = 7.7 Hz, 1H, m_j), 3.68 (d, J = 4.8 Hz, 1H, m_j), 3.65 (d, J = 3.4 Hz, 1H, m_n), 3.49 – 3.43 (m, 1H, m_n), 3.38 – 3.31 (m, 1H, m_j), 2.12 – 1.99 (m, 2H, m_j), 1.85 – 1.72 (m, 1H, m_n), 1.32 – 1.16 (m, 17H), 0.86 (t, J = 6.8 Hz, 3H, m_j), 0.82 (t, J = 7.1 Hz, 3H, m_n). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 179.3, 179.2, 141.9, 141.7, 138.0, 137.2, 136.2, 129.0, 128.7, 128.6, 128.5, 128.2, 128.1, 128.0, 127.8, 125.4, 125.1, 122.0, 109.6, 109.5, 52.8, 51.6, 47.2, 46.3, 33.5, 32.0, 31.9, 29.7, 29.6, 29.3, 29.3, 27.8, 27.8, 22.8, 22.7, 21.2, 21.1, 14.2. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{22}H_{28}NO]$ 322.2171; Found 322.2178.



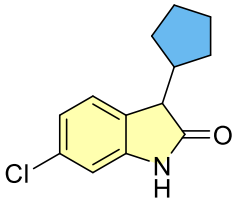
P20

3-(6-methylhept-5-en-2-yl)indolin-2-one (P_{20}): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{20}** as a colorless oil (0.055 g, 77%). Two isomers ratio 1.11:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 8.97 (s, 1H, m_j), 8.87 (s, 1H, m_n), 7.17 – 7.10 (m, 4H), 6.93 (t, *J* = 7.5 Hz, 2H), 6.83 (t, *J* = 7.4 Hz, 2H), 5.07 (t, *J* = 7.0 Hz, 1H, m_j), 5.00 (t, *J* = 7.0 Hz, 1H, m_n), 3.42 (d, *J* = 3.0 Hz, 1H, m_j), 3.39 (d, *J* = 2.8 Hz, 1H, m_n), 2.34 – 2.19 (m, 2H), 2.10 – 1.90 (m, 4H), 1.63 (s, 3H, m_j), 1.59 (s, 3H, m_n), 1.54 (s, 3H, m_j), 1.50 (s, 3H, m_n), 1.33 – 1.03 (m, 4H), 0.95 (d, *J* = 6.9 Hz, 3H, m_n), 0.72 (d, *J* = 6.8 Hz, 3H, m_j). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 180.7, 180.1, 142.3, 142.1, 132.1, 131.7, 129.1, 128.0, 127.9, 127.8, 124.9, 124.4, 124.3, 124.1, 122.2, 122.1, 109.8, 109.7, 51.3, 51.1, 35.4, 35.2, 34.7, 32.9, 26.2, 26.0, 25.9, 25.8, 17.8, 17.7, 16.9, 15.2. HRMS (ESI-TOF) *m*/*z*: $[M + H]^+$ Calcd for $[C_{16}H_{22}NO]$ 244.1701; Found 244.1710.



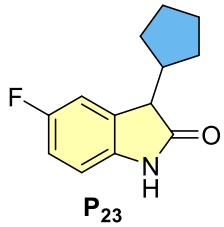
P_{21}

3-(5,9-dimethyl-1-phenyldec-8-en-1-yl)indolin-2-one (P_{21}): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{21}** as a yellowish oil (0.077 g, 68%). Two isomers ratio 1.47:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 8.48 (brs, 1H, m_n), 8.16 (brs, 1H, m_j), 7.25 – 7.13 (m, 6H), 7.10 – 7.04 (m, 3H), 7.03 – 6.96 (m, 1H), 6.95 – 6.90 (m, 3H), 6.78 (d, *J* = 7.7 Hz, 1H, m_n), 6.70 (d, *J* = 7.7 Hz, 1H, m_j), 5.21 – 4.94 (m, 2H), 3.70 (d, *J* = 4.8 Hz, 1H, m_j), 3.67 (d, *J* = 4.8 Hz, 1H, m_n), 3.53 – 3.47 (m, 1H, m_n), 3.42 – 3.35 (m, 1H, m_j), 2.13 – 2.01 (m, 2H, m_n), 1.98 – 1.84 (m, 4H, m_j), 1.67 (s, 3H, m_j), 1.66 (s, 3H, m_n), 1.58 (s, 3H, m_j), 1.56 (s, 3H, m_n), 1.38 – 1.18 (m, 9H, m_j), 1.14 – 1.02 (m, 2H, m_n), 0.81 (t, *J* = 7.7 Hz, 3H, m_j), 0.76 (t, *J* = 7.7 Hz, 3H, m_n). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 179.4, 179.3, 141.9, 141.7, 141.1, 141.0, 140.3, 131.2, 131.1, 128.8,128.7, 128.6, 128.3, 128.2, 128.1, 128.0, 127.9, 126.8, 126.7, 125.4, 125.2, 125.1, 125.0, 122.0, 109.7, 109.6, 52.7, 52.6, 51.6, 51.5, 47.6, 46.7, 37.3, 37.2, 37.0, 36.9, 33.7, 32.4, 25.8, 25.6, 25.6, 25.2, 19.7, 19.6, 19.6, 19.5, 17.8, 17.7. HRMS (ESI-TOF) *m*/*z*: $[M + Na]^+$ Calcd for $[C_{26}H_{33}NNaO]$ 398.2460; Found 398.2462.



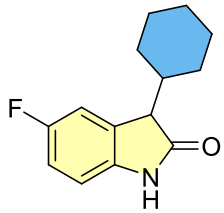
P_{22}

6-chloro-3-cyclopentylindolin-2-one (P_{22}):[2] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{22}** as a colourless solid (0.056 g, 79%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.39 (s, 1H), 7.17 (d, *J* = 7.9 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 1H), 6.93 (s, 1H), 3.49 (d, *J* = 5.3 Hz, 1H), 2.56 – 2.29 (m, 1H), 1.97 – 1.88 (m, 1H), 1.78 – 1.69 (m, 1H), 1.67 – 1.48 (m, 5H), 1.35 – 1.23 (m, 1H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 180.8, 143.2, 133.6, 127.6, 125.7, 122.1, 110.5, 49.0, 41.9, 30.0, 28.4, 25.2, 25.1.



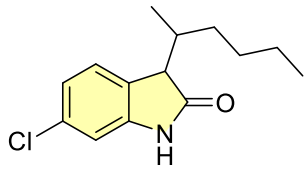
P23

3-cyclopentyl-5-fluoroindolin-2-one (P23):[2] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P23** as a yellowish brown solid (0.047 g, 71%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.54 (brs, 1H), 7.01 (d, *J* = 8.3 Hz, 1H), 6.94 – 6.80 (m, 2H), 3.52 (d, *J* = 5.2 Hz, 1H), 2.53 – 2.36 (m, 1H), 1.98 – 1.86 (m, 1H), 1.79 – 1.71 (m, 1H), 1.69 – 1.47 (m, 5H), 1.37 – 1.24 (m, 1H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 180.8, 160.1, 157.7, 138.1, 130.9, 130.8, 114.3, 114.1, 112.8, 112.6, 110.3, 110.2, 49.8, 41.9, 29.9, 28.4, 25.2, 25.1. ^{19}F{^{1}H} NMR (377 MHz, $CDCl_3$) δ -121.26.



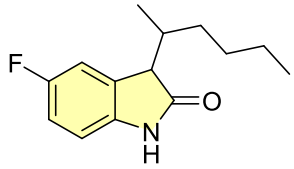
P24

3-cyclohexyl-5-fluoroindolin-2-one (P24):[3] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P24** as a brown solid (0.045 g, 65%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.03 (s, 1H), 7.00 (d, *J* = 7.8 Hz, 1H), 6.94 – 6.79 (m, 2H), 3.36 (d, *J* = 2.2 Hz, 1H), 2.18 – 2.06 (m, 1H), 1.80 – 1.52 (m, 5H), 1.45 – 1.09 (m, 5H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 180.2, 160.1, 157.8, 138.0, 130.4, 130.4, 114.2, 114.0, 112.9, 112.6, 110.2, 110.1, 52.7, 41.0, 30.4, 28.4, 26.7, 26.4, 26.1. ^{19}F{^{1}H} NMR (377 MHz, $CDCl_3$) δ -121.14.



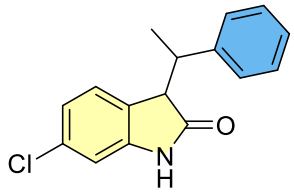
P25

6-chloro-3-(hexan-2-yl)indolin-2-one (P25): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P25** as a pale yellow oil (0.056 g, 74%). Two isomers ratio 1.55:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.50 (m_j), 9.43 (m_n) (two s, 1H), 7.17 – 7.12 (m, 1H), 7.03 – 6.93 (m, 2H), 3.48 (m_j), 3.45(m_n) (two d, *J* = 2.9 Hz, 1H), 2.42 – 2.20 (m, 1H), 1.58 – 1.25 (m, 6H), 1.02 (m_n), 0.94 (m_j) (one d, *J* = 6.9 Hz, one t, *J* = 6.9 Hz, 3H), 0.88 (m_n), 0.78 (m_j) (one t, *J* = 6.8 Hz, one d, *J* = 6.8 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 181.1, 180.4, 143.5, 143.3, 133.6, 133.5, 127.5, 126.4, 125.7, 125.2, 122.2, 122.1, 110.6, 110.5, 51.2, 50.7, 35.8, 35.5, 34.2, 32.5, 31.7, 29.9, 22.8, 22.7, 16.9, 15.4, 14.3, 14.2. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{14}H_{19}ClNO]$ 252.1155; Found 252.1152.



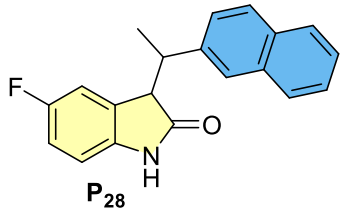
P26

5-fluoro-3-(hexan-2-yl)indolin-2-one (P_{26}): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{26}** as a brown oil (0.045 g, 64%). ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.38 (m_j), 9.28 (m_n) (two s, 1H), 7.00 – 6.87 (m, 2H), 6.87 – 6.81 (m, 1H), 3.49 (m_j), 3.45 (m_n) (two d, J = 2.5 Hz, 1H), 2.39 – 2.20 (m, 1H), 1.59 – 1.21 (m, 6H), 1.01 (m_j), 0.92 (m_n), (one d, J = 7.0 Hz, one t, J = 6.9 Hz, 3H), 0.85 (m_n), 0.77 (m_j), (one t, J = 6.9 Hz, one d, J = 6.8 Hz, 3H). ^{13}C{1H} NMR (101 MHz, $CDCl_3$) δ 180.9, 180.2, 160.2, 160.1, 157.8, 157.7, 138.3, 138.0, 130.8, 130.8, 129.7, 129.6, 114.3, 114.2, 114.1, 114.0, 113.0, 112.7, 112.4, 112.2, 110.3, 110.2, 110.1, 52.0, 51.5, 35.9, 35.5, 34.2, 32.5, 29.9, 29.8, 22.8, 16.9, 15.4, 14.2. ^{19}F{^{1}H} NMR (377 MHz, $CDCl_3$) δ -121.20 (m_j), -121.24 (m_n). HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{14}H_{19}FNO]$ 236.1451; Found 236.1460.



P27

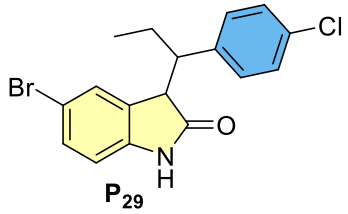
6-chloro-3-(1-phenylethyl)indolin-2-one (P_{27}): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{27}** as a pale yellow solid (0.050 g, 62%). Two isomers ratio 2.32:1. ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 8.86 (brs, 1H, m_j), 8.51 (brs, 1H, m_n), 7.41 – 7.35 (m, 2H, m_j), 7.34 – 7.29 (m, 3H, m_j), 7.22 – 7.16 (m, 1H, m_n), 7.11 – 7.06 (m, 1H, m_n), 6.94 (d, J = 8.1 Hz, 1H, m_n), 6.91 (d, J = 1.7 Hz, 1H, m_j), 6.86 (d, J = 8.0 Hz, 1H, m_j), 6.80 (d, J = 1.3 Hz, 1H, m_n), 6.41 (d, J = 8.0 Hz, 1H, m_j), 3.85 – 3.78 (m, 1H, m_j), 3.76 (d, J = 3.7 Hz, 1H, m_j), 3.64 (d, J = 5.8 Hz, 1H, m_n), 3.53 – 3.44 (m, 1H, m_n), 1.65 (d, J = 7.2 Hz, 3H, m_n), 1.21 (d, J = 7.0 Hz, 3H, m_j). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 179.5, 179.1, 143.2, 142.8, 142.5, 141.9, 133.8, 128.6, 128.3, 128.0, 127.9, 127.1, 127.0, 126.6, 126.2, 126.1, 125.4, 122.0, 121.9, 110.3, 52.7, 52.2, 41.9, 39.6, 19.4, 13.5. HRMS (ESI-TOF) *m/z*: $[M + H]^+$ Calcd for $[C_{16}H_{15}ClNO]$ 272.0842; Found 272.0852.



P28

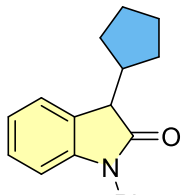
5-fluoro-3-(1-(naphthalen-2-yl)ethyl)indolin-2-one (P_{28}): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{28}** as a pale yellow oil (0.055 g, 60%). ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 9.20 (s, 1H, m_j), 8.76 (s, 1H, m_n), 7.89 – 7.79 (m, 3H), 7.76 – 7.69 (m, 1H), 7.65 (t, J = 8.2 Hz, 1H), 7.54 – 7.46 (m, 3H), 7.44 – 7.36 (m, 1H), 7.20 (d, J = 8.4 Hz, 1H), 6.93 – 6.75 (m, 3H), 6.68 –

6.61 (m,1H), 6.24 (d, J = 8.2 Hz, 1H), 3.99 – 3.90 (m, 1H, m_j), 3.88 (d, J = 3.1 Hz, 1H, m_j), 3.75 (d, J = 5.3 Hz, 1H, m_n), 3.69 (m, 1H, m_n), 1.71 (d, J = 7.0 Hz, 3H, m_n), 1.29 (d, J = 7.0 Hz, 3H, m_j). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.7, 179.2, 159.9, 159.8, 157.5, 157.4, 139.9, 139.2, 138.1, 137.8, 133.4, 133.3, 132.6, 132.5, 129.8, 129.7, 128.7, 128.6, 128.4, 128.0, 127.9, 127.8, 127.7, 127.6, 126.9, 126.5, 126.4, 126.2, 126.1, 126.0, 125.9, 125.7, 114.7, 114.6, 114.4, 114.3, 113.3, 113.2, 113.0, 112.9, 110.3, 110.2, 53.3, 53.0, 41.9, 39.7, 19.3, 13.6. ^{19}F{^{1}H} NMR (377 MHz, $CDCl_3$) δ -120.70 (m_j), -120.98 (m_n). HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $[C_{20}H_{16}FNONa]$ 328.1114; Found 328.1128.



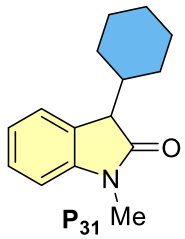
P29

5-bromo-3-(1-(4-chlorophenyl)propyl)indolin-2-one (P29): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P29** as a yellow solid (0.068 g, 62%). ^{1}H NMR (400 MHz, $CDCl_3$) (m_j: major isomer, m_n: minor isomer) δ 8.85 (m_n), 8.61 (m_j) (two s, 1H), 7.43 – 7.28 (m, 2H), 7.23 (d, J = 8.4 Hz, 1H, m_n), 7.12 (d, J = 8.4 Hz, 1H, m_j), 7.07 (d, J = 8.4 Hz, 1H, m_n), 6.86 (d, J = 8.4 Hz, 1H, m_j), 6.68 (m_n), 6.64 (m_j) (two d, J = 8.3 Hz, 1H), 3.72 (m_j), 3.67 (m_n) (two d, J = 3.3 Hz, 1H), 3.36 – 3.24 (m, 1H), 2.13 – 1.87 (m, 2H), 0.96 (m_j), 0.88 (m_n) (two t, J = 7.3 Hz, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 179.3, 178.7, 141.0, 140.7, 138.6, 137.9, 132.8, 132.7, 131.2, 131.0, 130.0, 129.9, 128.6, 128.3, 128.2, 127.8, 114.8, 114.7, 111.4, 111.2, 52.0, 51.4, 48.6, 48.3, 26.3, 23.0, 12.5, 12.4. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $[C_{17}H_{16}BrClNO]$ 364.0104; Found 364.0086.



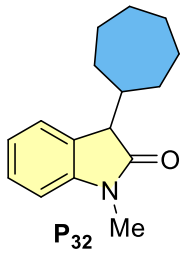
P30

3-cyclopentyl-1-phenylindolin-2-one (P30):[2] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P30** as a pale yellow oil (0.068 g, 82%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (t, J = 7.8 Hz, 2H), 7.43 – 7.34 (m, 4H), 7.20 (t, J = 7.6 Hz, 1H), 7.06 (t, J = 7.5 Hz, 1H), 6.79 (d, J = 7.9 Hz, 1H), 3.69 (d, J = 5.1 Hz, 1H), 2.64 – 2.52 (m, 1H), 2.00 – 1.87 (m, 1H), 1.85 – 1.77 (m, 1H), 1.70 – 1.53 (m, 5H), 1.46 – 1.36 (m, 1H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 177.1, 144.7, 134.7, 129.7, 128.4, 128.1, 127.8, 126.8, 124.9, 122.6, 109.2, 48.8, 42.6, 29.9, 28.2, 25.2, 25.1.



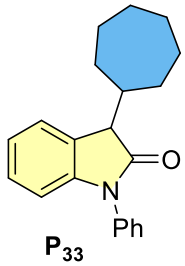
P31

3-cyclohexyl-1-methylindolin-2-one (P_{31}):[1] The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{31}** as a yellowish brown oil (0.040 g, 58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, J = 6.0 Hz, 1H), 7.06 (t, J = 7.5 Hz, 1H), 6.83 (d, J = 8.0 Hz, 1H), 3.36 (d, J = 3.2 Hz, 1H), 3.21 (s, 1H), 2.23 – 2.12 (m, 1H), 1.81 – 1.64 (m, 2H), 1.50 – 1.38 (m, 1H), 1.33 – 1.24 (m, 1H), 1.17 – 1.09 (m, 1H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 177.6, 144.8, 128.2, 127.8, 124.5, 122.2, 107.9, 51.6, 41.1, 30.5, 28.4, 26.8, 26.4, 26.2, 26.1.



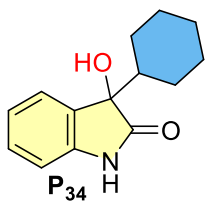
P_{32}

3-cycloheptyl-1-methylindolin-2-one (P_{32}): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{32}** as a pale yellow oil (0.038 g, 52%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, J = 7.3 Hz, 1H), 7.19 (t, J = 3.8 Hz, 1H), 6.96 (t, J = 7.5 Hz, 1H), 6.74 (d, J = 7.7 Hz, 1H), 3.34 (d, J = 3.0 Hz, 1H), 3.12 (s, 3H), 2.31 – 2.20 (m, 1H), 1.71 – 1.63 (m, 2H), 1.59 – 1.47 (m, 5H), 1.41 – 1.32 (m, 5H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 177.6, 144.9, 128.3, 127.9, 124.4, 122.2, 107.9, 52.8, 42.4, 32.8, 30.5, 27.9, 27.6, 27.4, 27.1, 26.2. HRMS (ESI-TOF) *m*/*z*: [M + H]$^+$ Calcd for [$C_{16}H_{22}NO$] 244.1701; Found 244.1693.



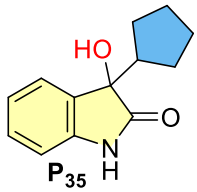
P_{33}

3-cycloheptyl-1-phenylindolin-2-one (P_{33}): The crude product was purified by column chromatography (hexane/EtOAc 8:2 as eluent) to give pure product **P_{33}** as a colourless solid (0.064 g, 70%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (t, J = 7.7 Hz, 2H), 7.43 – 7.33 (m, 4H), 7.19 (t, J = 7.7 Hz, 1H), 7.07 (t, J = 7.4 Hz, 1H), 6.79 (d, J = 7.8 Hz, 1H), 3.59 (d, J = 3.0 Hz, 1H), 2.48 – 2.34 (m, 1H), 1.87 – 1.76 (m, 2H), 1.66 – 1.58 (m, 5H), 1.51 – 1.40 (m, 5H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 177.0, 144.8, 134.8, 129.7, 128.2, 128.1, 127.7, 126.8, 124.6, 122.7, 109.2, 52.7, 43.0, 32.6, 30.6, 28.0, 27.5, 27.4, 27.1. HRMS (ESI-TOF) *m*/*z*: [M]$^+$ Calcd for [$C_{21}H_{23}NO$] 305.1780; Found 305.1769.



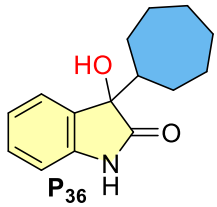
P_{34}

3-cyclohexyl-3-hydroxyindolin-2-one (P_{34}): The crude product was purified by column chromatography (hexane/EtOAc 7:3 as eluent) to give pure product **P_{34}** as a white solid (0.057 g, 82%). ^{1}H NMR (400 MHz, DMSO – d_6) δ 10.18 (s, 1H), 7.23 – 7.12 (m, 2H), 6.93 (t, J = 7.4 Hz, 1H), 6.77 (d, J = 7.7 Hz, 1H), 5.73 (s, 1H), 1.90 – 1.82 (m, 1H), 1.79 – 1.67 (m, 2H), 1.60 – 1.46 (m, 3H), 1.18 – 1.04 (m, 3H), 1.01 – 0.89 (m, 1H), 0.69 – 0.55 (m, 1H). ^{13}C{^{1}H} NMR (101 MHz, DMSO – d_6) δ 179.6, 142.2, 131.2, 128.7, 124.6, 121.3, 109.3, 78.2, 45.1, 26.1, 26.0, 25.8, 25.6, 25.4. HRMS (ESI-TOF) m/z: [M – H_2O]$^+$ Calcd for [$C_{14}H_{16}NO$] 214.1232; Found 214.1242.



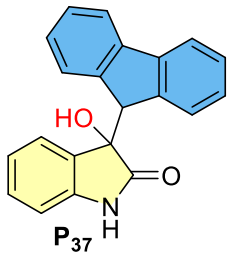
P_{35}

3-cyclopentyl-3-hydroxyindolin-2-one (P_{35}): The crude product was purified by column chromatography (hexane/EtOAc 7:3 as eluent) to give pure product **P_{35}** as a white solid (0.049 g, 75%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.41 (d, J = 7.4 Hz, 1H), 7.29 – 7.24 (m, 1H), 7.07 (t, J = 7.5 Hz, 1H), 6.89 (d, J = 7.7 Hz, 1H), 3.35 (s, 1H), 2.48 (m, 1H), 1.84 – 1.76 (m, 1H), 1.72 – 1.63 (m, 1H), 1.58 – 1.47 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 181.1, 140.8, 130.2, 129.6, 125.0, 123.0, 110.3, 78.7, 47.6, 26.6, 26.5, 25.6, 25.5. HRMS (ESI-TOF) m/z: [M – H_2O]$^+$ Calcd for [$C_{13}H_{14}NO$] 200.1075; Found 200.1078.



P_{36}

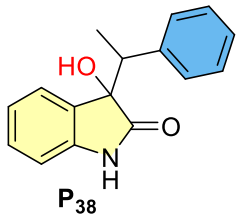
3-cycloheptyl-3-hydroxyindolin-2-one (P_{36}): The crude product was purified by column chromatography (hexane/EtOAc 7:3 as eluent) to give pure product **P_{36}** as a white solid (0.056 g, 77%). ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.22 (s, 1H), 7.25 – 7.16 (m, 2H), 6.93 (t, J = 7.4 Hz, 1H), 6.78 (d, J = 7.6 Hz, 1H), 5.79 (s, 1H), 2.13 – 2.05 (m, 1H), 1.94 – 1.86 (m, 1H), 1.76 – 1.68 (m, 1H), 1.58 – 1.27 (m, 10H). ^{13}C NMR (101 MHz, DMSO-d_6) δ 179.7, 142.3, 131.0, 128.8, 124.6, 121.4, 109.4, 78.1, 46.0, 28.0, 27.9, 27.4, 26.9, 26.6, 26.5. HRMS (ESI-TOF) m/z: [M – H_2O]$^+$ Calcd for [$C_{15}H_{18}NO$] 228.1388; Found 228.1398.



P_{37}

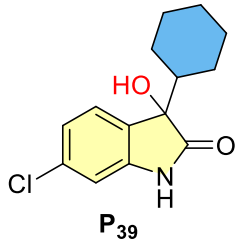
3-(9H-fluoren-9-yl)-3-hydroxyindolin-2-one (P_{37}): The crude product was purified by column chromatography (hexane/EtOAc 7:3 as eluent) to give pure product **P_{37}** as a pale brown solid (0.077 g, 82%). ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.45 (s, 1H), 7.98 (d, J = 6.7 Hz, 1H), 7.82 (d, J = 7.5 Hz, 1H), 7.65 (d, J = 7.4 Hz, 1H), 7.47 (t, J = 7.4 Hz, 1H), 7.38 (t, J = 6.9 Hz,

2H), 7.21 (t, J = 7.3 Hz, 1H), 7.13 (t, J = 7.4 Hz, 1H), 6.90 (t, J = 7.6 Hz, 1H), 6.68 (s, 1H), 6.54 (d, J = 7.7 Hz, 1H), 6.41 (t, J = 7.5 Hz, 1H), 5.79 (d, J = 6.9 Hz, 1H), 4.44 (s, 1H). $^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO-d_6) δ 178.5, 143.1, 141.7, 141.6, 141.3, 140.6, 129.0, 128.7, 127.8, 127.5, 127.4, 126.6, 126.5, 124.4, 123.4, 120.8, 109.2, 77.5, 52.5. HRMS (ESI-TOF) m/z: [M – H_2O]$^+$ Calcd for [$C_{21}H_{14}NO$] 296.1075; Found 296.1060.



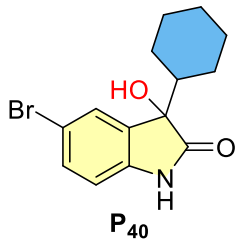
P38

3-hydroxy-3-(1-phenylethyl)indolin-2-one (P38): The crude product was purified by column chromatography (hexane/EtOAc 7:3 as eluent) to give pure product **P38** as a pale brown solid (0.058 g, 76%). ^{1}H NMR (400 MHz, DMSO-d_6) (m_j: major isomer, m_n: minor isomer) δ 10.19 (s, 1H, m_n), 9.83 (s, 1H, m_j), 7.34 (d, J = 7.3 Hz, 1H, m_j), 7.18 (t, J = 6.5 Hz, 2H, m_j), 7.12 (dd, J = 13.0, 6.7 Hz, 2H, m_n), 7.04 (dd, J = 13.0, 6.7 Hz, 3H, m_j), 6.96 (t, J = 7.5 Hz, 1H, m_n), 6.81 (t, J = 7.5 Hz, 1H, m_n), 6.72 (d, J = 6.8 Hz, 2H, m_j), 6.67 (d, J = 7.7 Hz, 1H, m_j), 6.61 (d, J = 7.3 Hz, 1H, m_j), 6.55 (d, J = 7.7 Hz, 1H, m_j), 6.05 (s, 1H, m_j), 5.98 (s, 1H, m_n), 3.29 (q, J = 7.1 Hz, 1H, m_n), 3.22 (q, J = 7.0 Hz, 1H, m_j), 1.47 (d, J = 7.1 Hz, 3H, m_j), 1.14 (d, J = 7.1 Hz, 2H, m_n). $^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO- d_6) δ 179.2, 179.0,142.1, 142.0, 140.6, 140.3, 130.2, 129.9, 129.4, 129.0, 128.8, 128.4, 127.4, 127.2, 126.5, 126.4, 125.2, 125.0, 121.0, 120.9, 109.2, 109.1, 78.5, 77.9, 46.4, 45.1, 14.6, 13.9. HRMS (ESI-TOF) m/z: [M – H_2O]$^+$ Calcd for [$C_{16}H_{14}NO$] 236.1075; Found 236.1083.



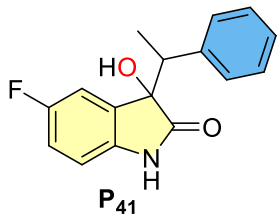
P39

6-chloro-3-cyclohexyl-3-hydroxyindolin-2-one (P39): The crude product was purified by column chromatography (hexane/EtOAc 7:3 as eluent) to give pure product **P39** as a pale brown solid (0.062 g, 78%). ^{1}H NMR (400 MHz, DMSO- d_6) δ 10.36 (s, 1H), 7.22 (d, J = 7.9 Hz, 1H), 6.99 (d, J = 9.2 Hz, 1H), 6.79 (s, 1H), 5.85 (s, 1H), 1.90 – 1.44 (m, 7H), 1.26 – 0.91 (m, 6H). $^{13}C\{^{1}H\}$ NMR (101 MHz, DMSO-d_6) δ 179.5, 143.7, 133.0, 130.1, 126.1, 121.0, 109.4, 77.8, 44.9, 26.0, 25.9, 25.8, 25.6, 25.3. HRMS (ESI-TOF) m/z: [M – H_2O]$^+$ Calcd for [$C_{14}H_{15}ClNO$] 248.0842; Found 248.0856.



P40

5-bromo-3-cyclohexyl-3-hydroxyindolin-2-one (P_{40}): The crude product was purified by column chromatography (hexane/EtOAc 7:3 as eluent) to give pure product **P_{40}** as pale brown solid (0.075 g, 81%). ^{1}H NMR (400 MHz, DMSO- *d*$_6$) δ 10.47 (s, 1H), 7.44 – 7.28 (m, 2H), 6.76 (d, *J* = 8.2 Hz, 1H), 2.03 – 1.94 (m, 1H), 1.75 – 1.38 (m, 6H), 1.24 – 1.13 (m, 4H). ^{13}C{^{1}H} NMR (101 MHz, DMSO- *d*$_6$) δ 177.7, 142.4, 131.3, 130.2, 127.0, 112.9, 110.9, 51.0, 29.1, 28.3, 26.1, 25.9, 25.8. HRMS (ESI-TOF) *m*/*z*: [M – H_2O]$^+$ Calcd for [$C_{14}H_{15}BrNO$] 292.0337; Found 292.0347.



P_{41}

5-fluoro-3-hydroxy-3-(1-phenylethyl)indolin-2-one (P_{41}): The crude product was purified by column chromatography (hexane/EtOAc 7:3 as eluent) to give pure product **P_{41}** as a colourless solid (0.059 g, 78%). ^{1}H NMR (400 MHz, DMSO-*d*$_6$) (m_j: major isomer, m_n: minor isomer) δ 10.23 (s, 1H, m_n), 9.90 (s, 1H, m_j), 7.24 – 7.16 (m, 1H), 7.15 – 7.04 (m, 4H), 7.03 – 6.90 (m, 1H), 6.82 – 6.75 (m, 2H), 6.65 (dd, *J* = 8.4, 4.4 Hz, 1H, m_n), 6.55 (dd, *J* = 8.4, 4.4 Hz, 1H, m_j), 6.46 – 6.41 (m, 1H, m_n), 6.22 (s, 1H, m_j), 6.17 (s, 1H, m_n), 3.31 (q, *J* = 7.1 Hz, 1H, m_n), 3.22 (q, *J* = 7.1 Hz, 1H, m_j), 1.47 (d, *J* = 7.2 Hz, 3H, m_j), 1.18 (d, *J* = 7.2 Hz, 1H, m_n). ^{13}C{^{1}H} NMR (101 MHz, DMSO-*d*$_6$) δ 179.2, 158.9, 158.7, 156.5, 156.3, 140.3, 140.0, 138.3, 138.2, 132.3, 132.2, 131.9, 131.8, 129.3, 128.5, 127.6, 127.4, 126.8, 126.7, 115.4, 115.2, 115.1, 115.0, 113.1, 112.9, 112.8, 112.5, 110.0, 109.97 , 78.9, 78.2, 46.4, 45.3, 14.4, 13.8. ^{19}F{^{1}H} NMR (377 MHz, DMSO-*d*$_6$) δ -122.12 (m_j), -122.18 (m_n). HRMS (ESI-TOF) *m*/*z*: [M – H_2O]$^+$ Calcd for [$C_{16}H_{13}FNO$] 254.0981; Found 254.0975.

^{1}H and ^{13}C NMR spectrum:

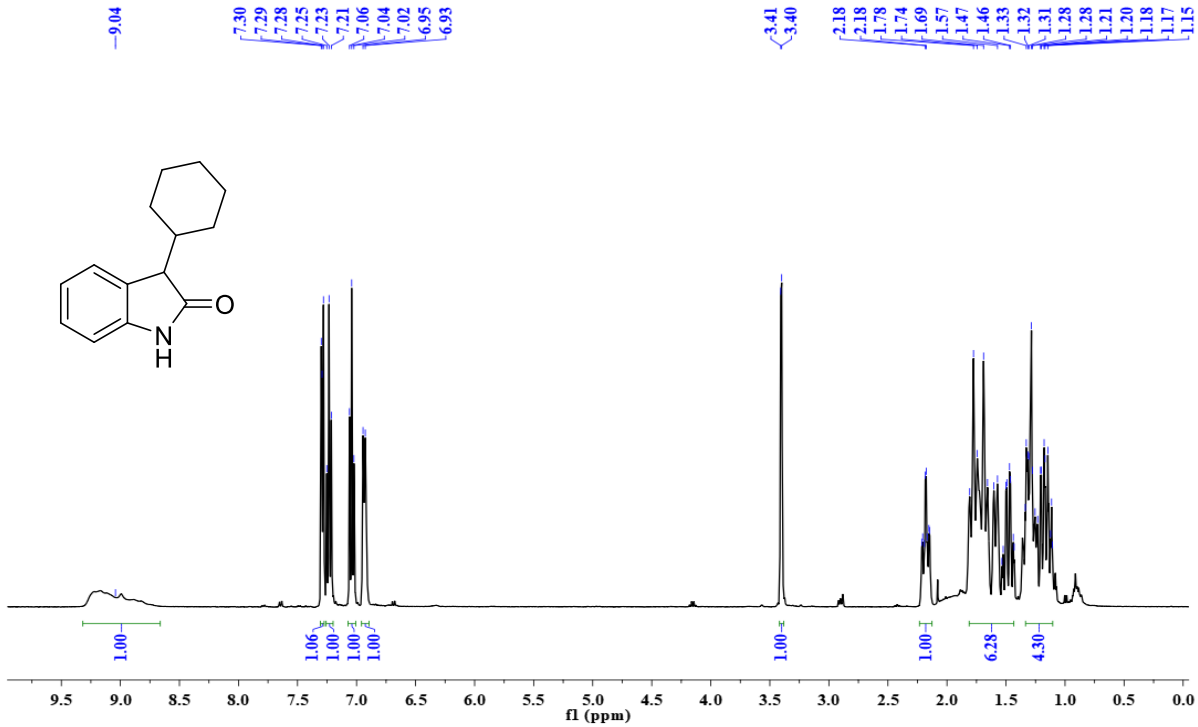


Figure S01. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cyclohexylindolin-2-one (**P1**).

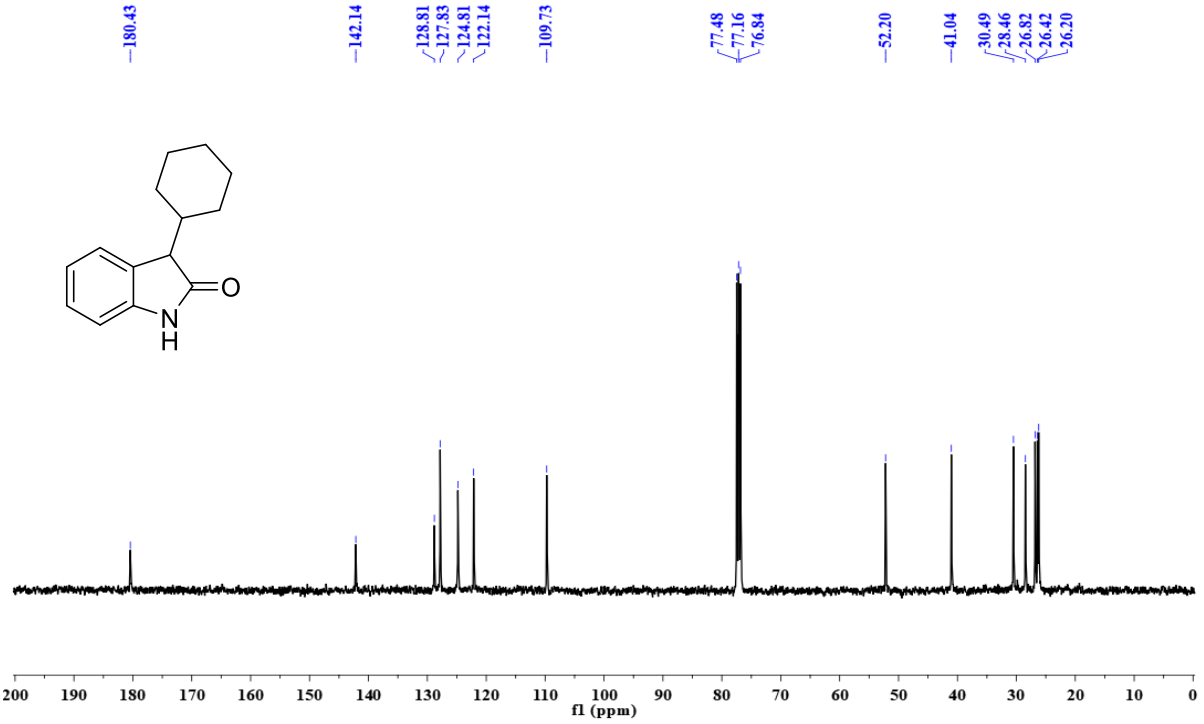


Figure S02. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-cyclohexylindolin-2-one (**P1**).

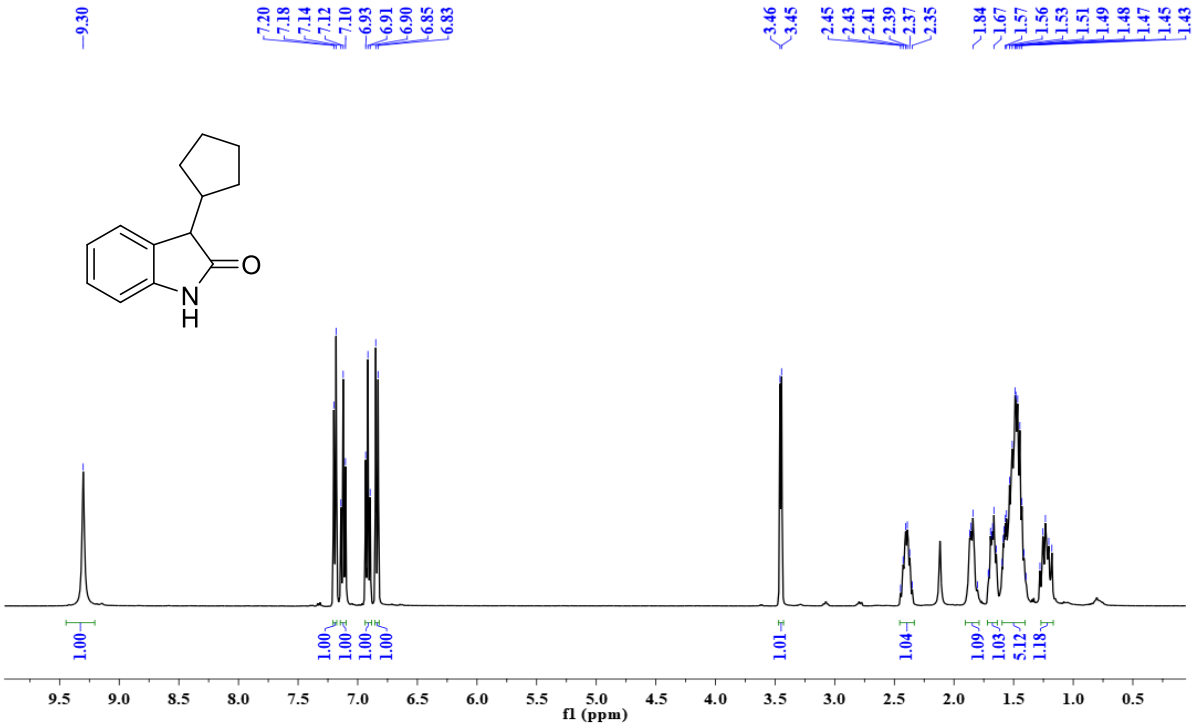


Figure S03. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cyclopentylindolin-2-one (**P₂**).

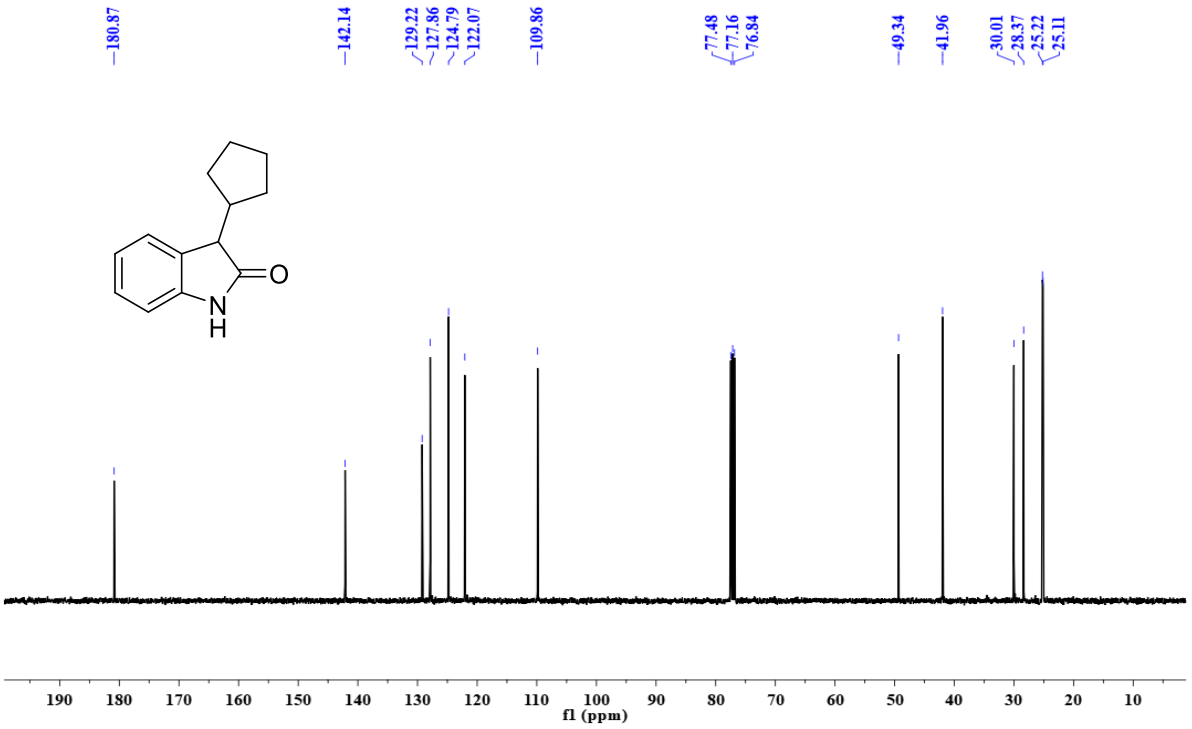


Figure S04. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) of 3-cyclopentylindolin-2-one (**P₂**).

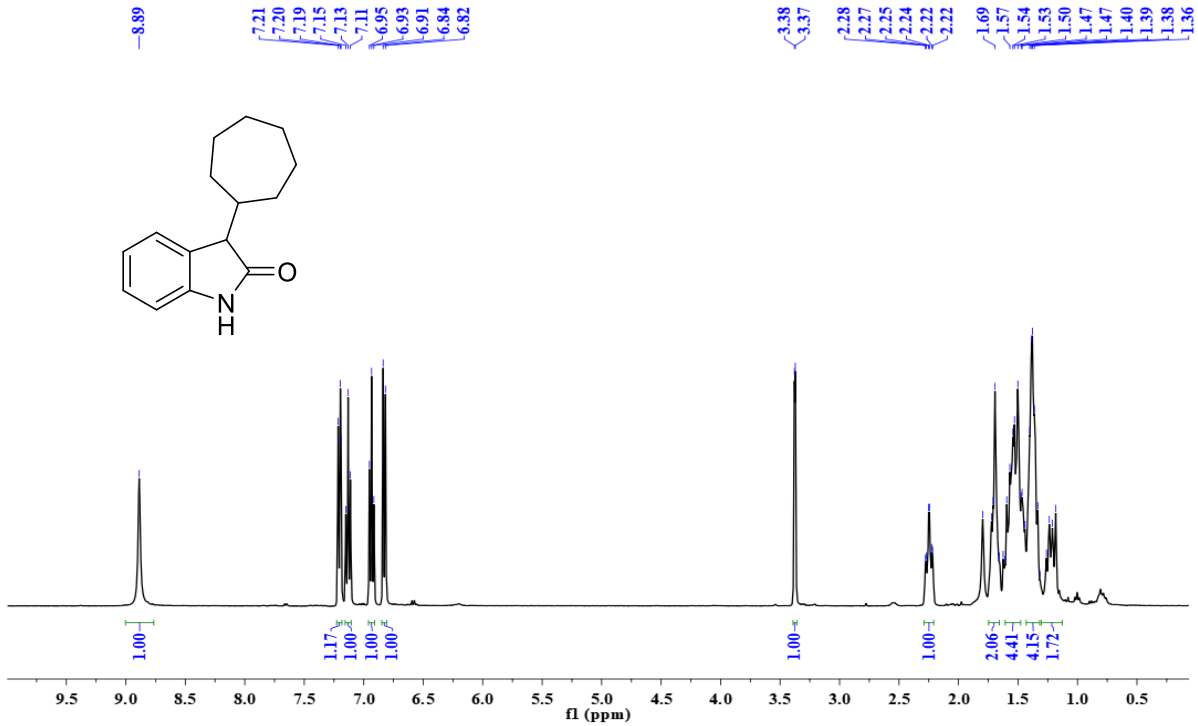


Figure S05. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cycloheptylindolin-2-one (**P3**).

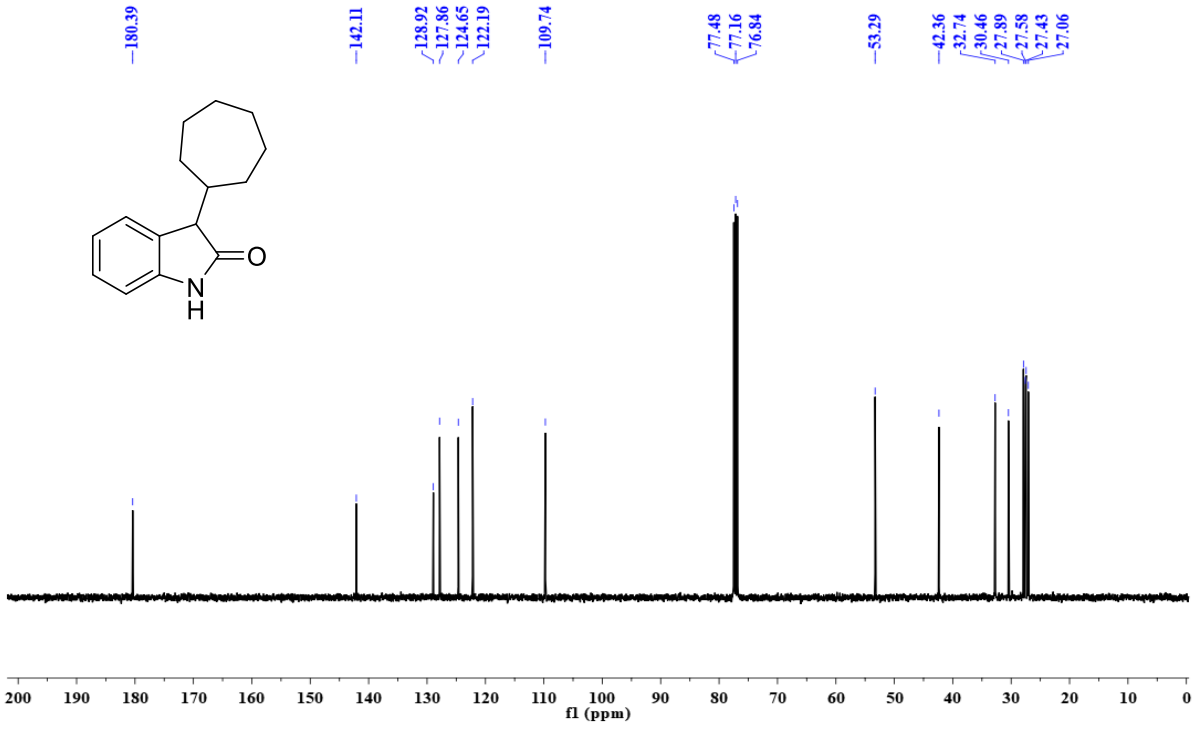


Figure S06. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-cycloheptylindolin-2-one (**P3**).

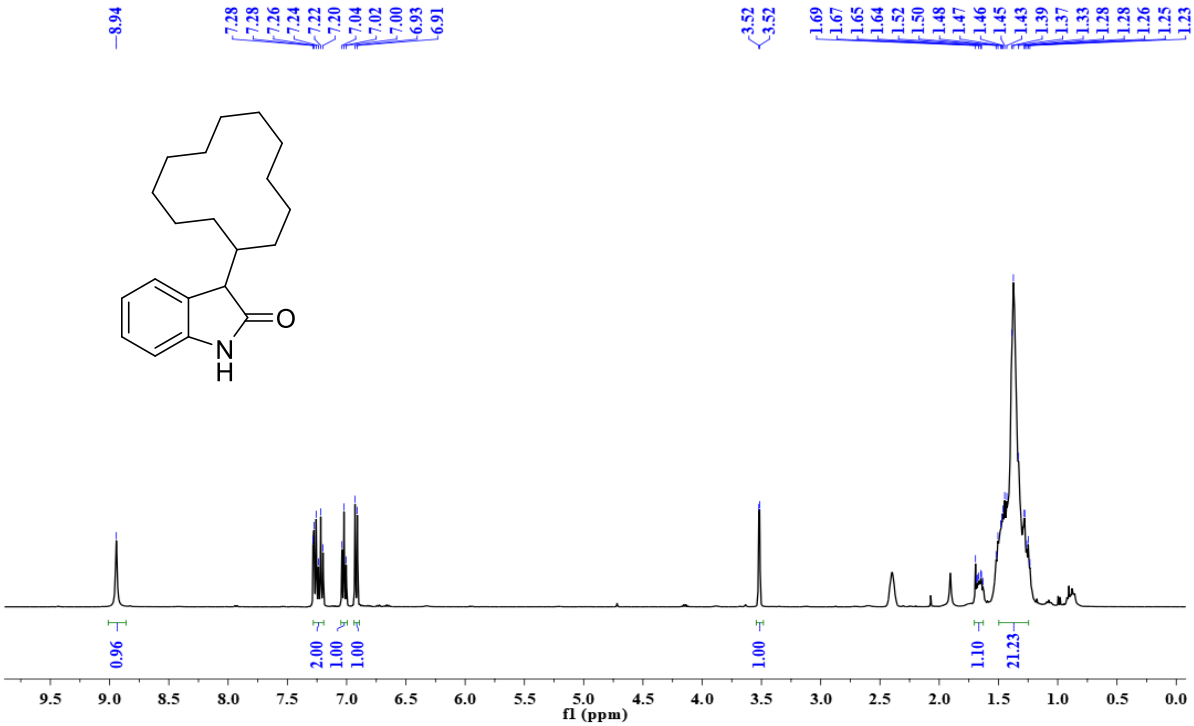


Figure S07. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cyclododecylindolin-2-one (**P4**).

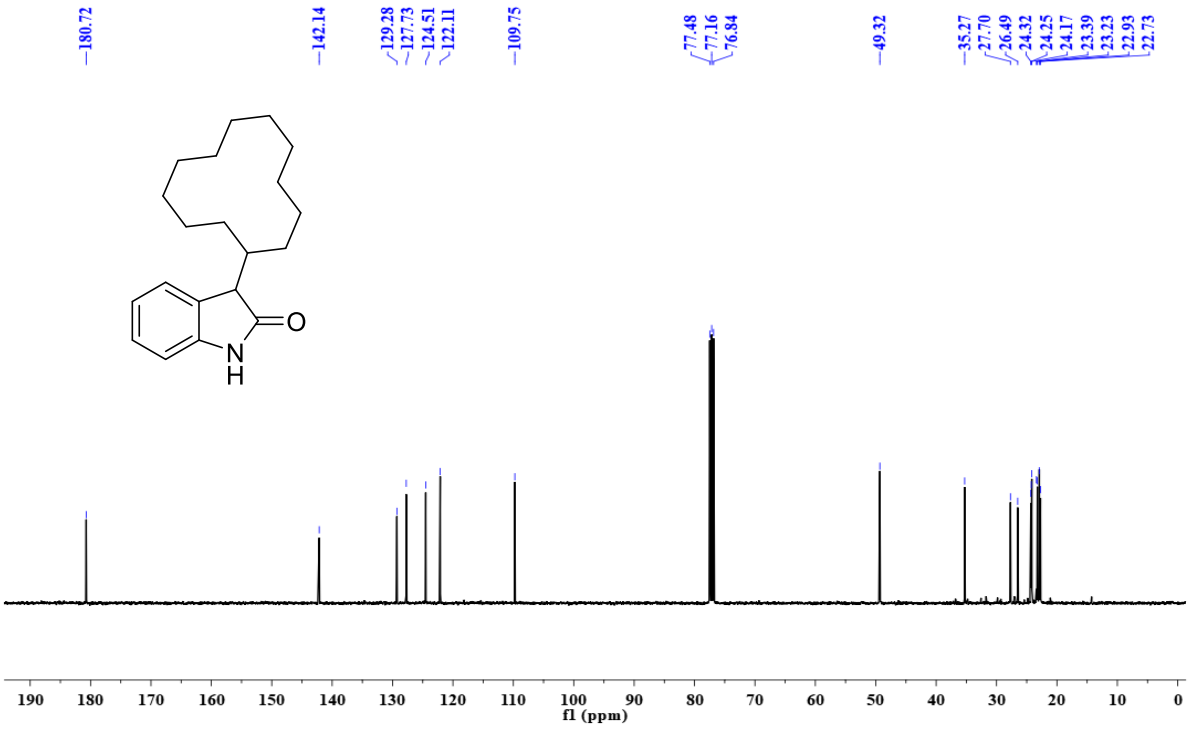


Figure S08. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-cyclododecylindolin-2-one (**P4**).

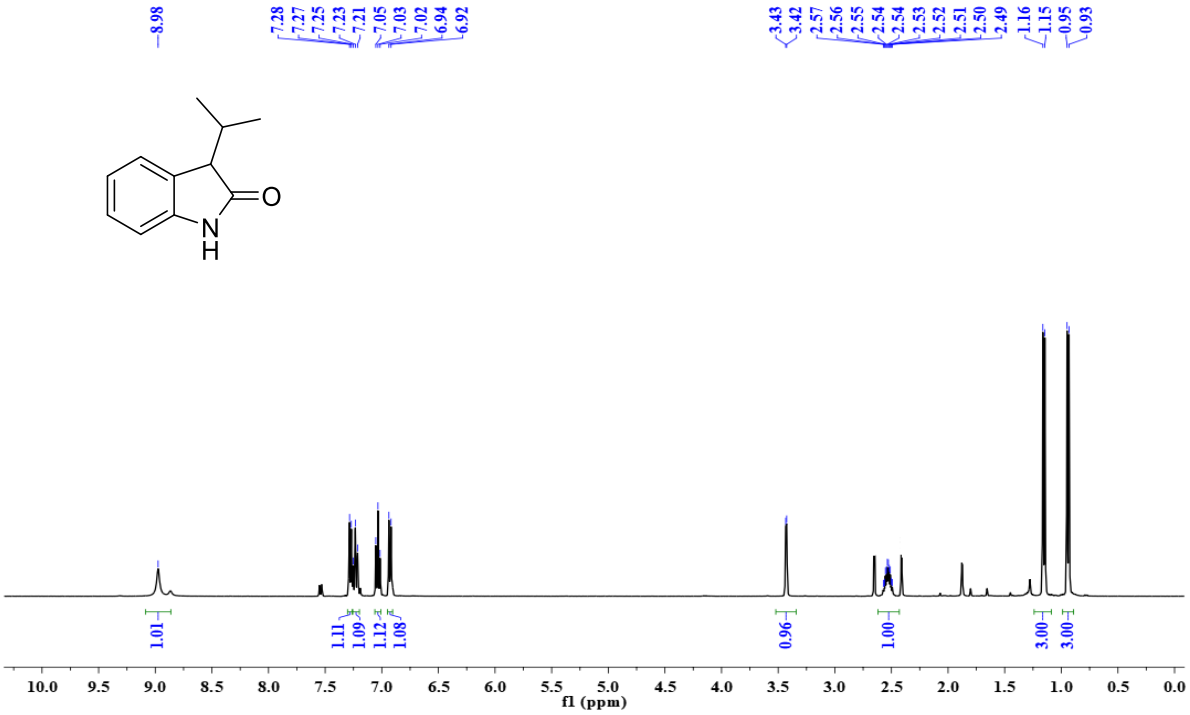


Figure S09. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-isopropylindolin-2-one (**P5**).

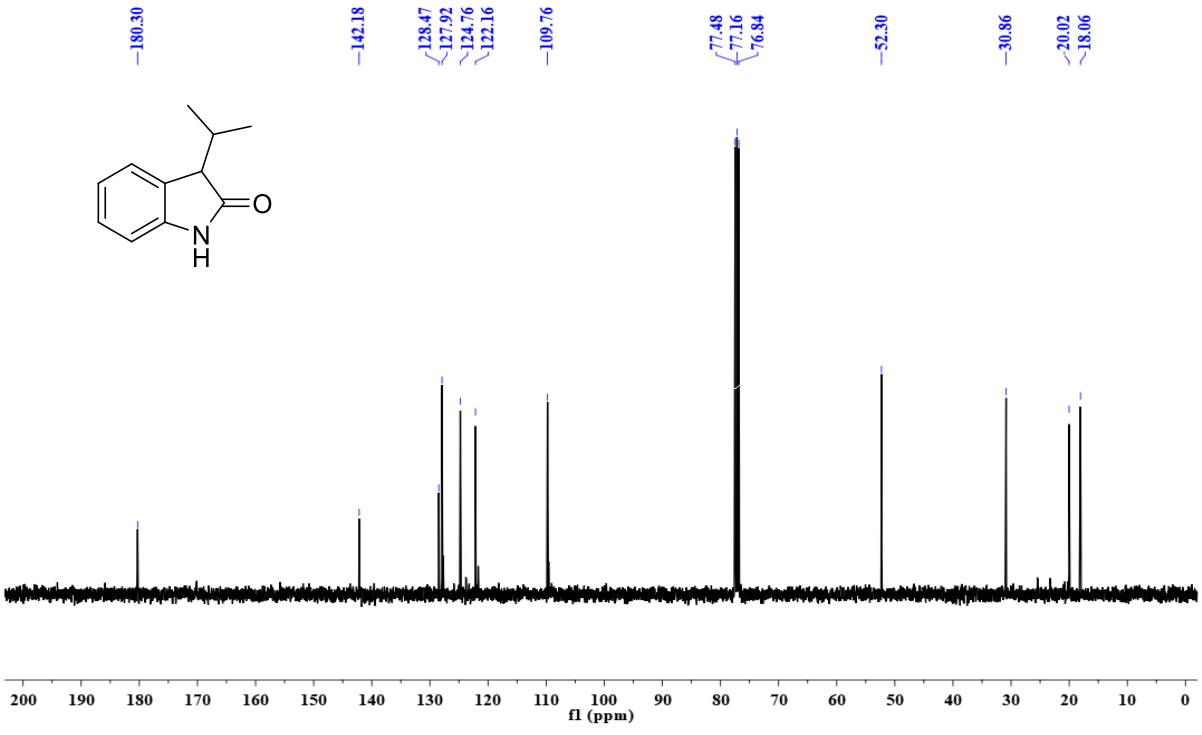


Figure S10. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-isopropylindolin-2-one (**P5**).

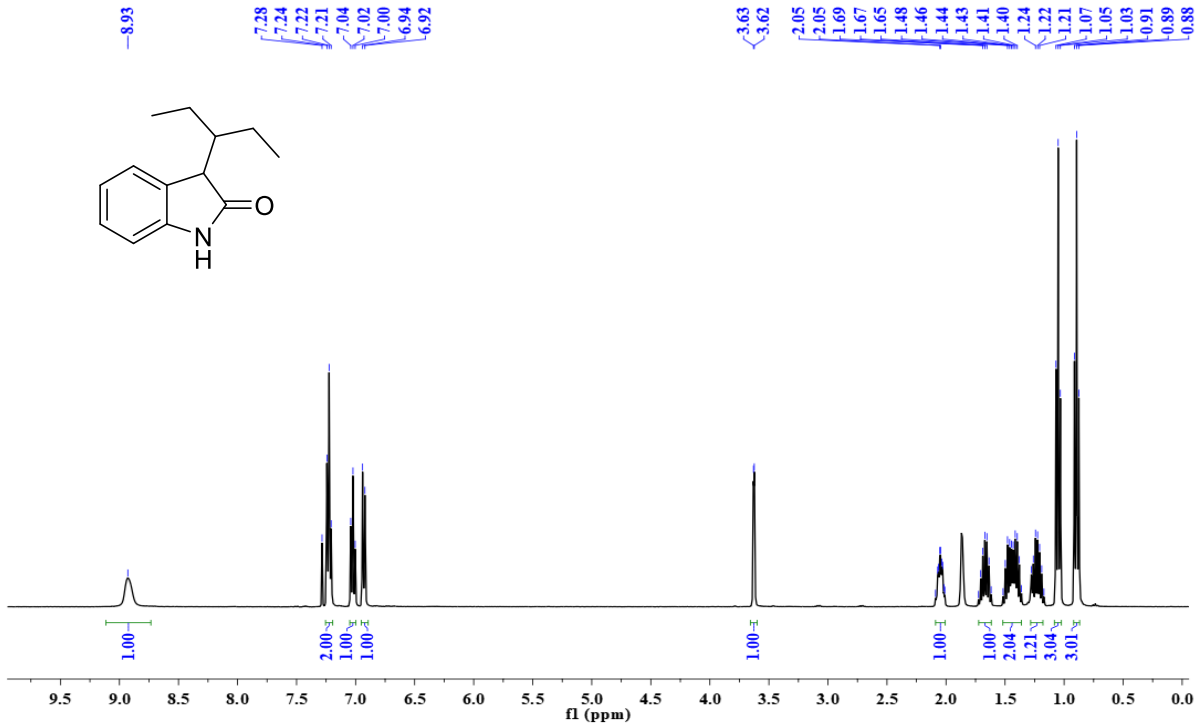


Figure S11. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(pentan-3-yl)indolin-2-one (**P6**).

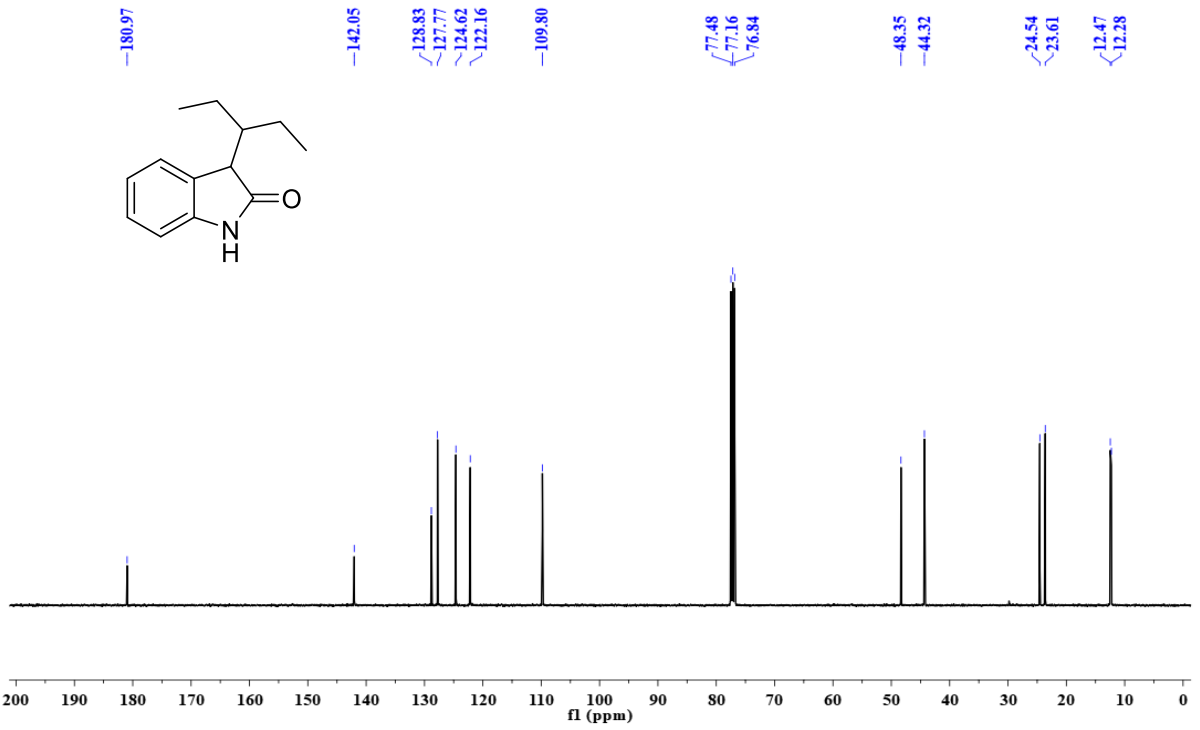


Figure S12. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(pentan-3-yl)indolin-2-one (**P6**).

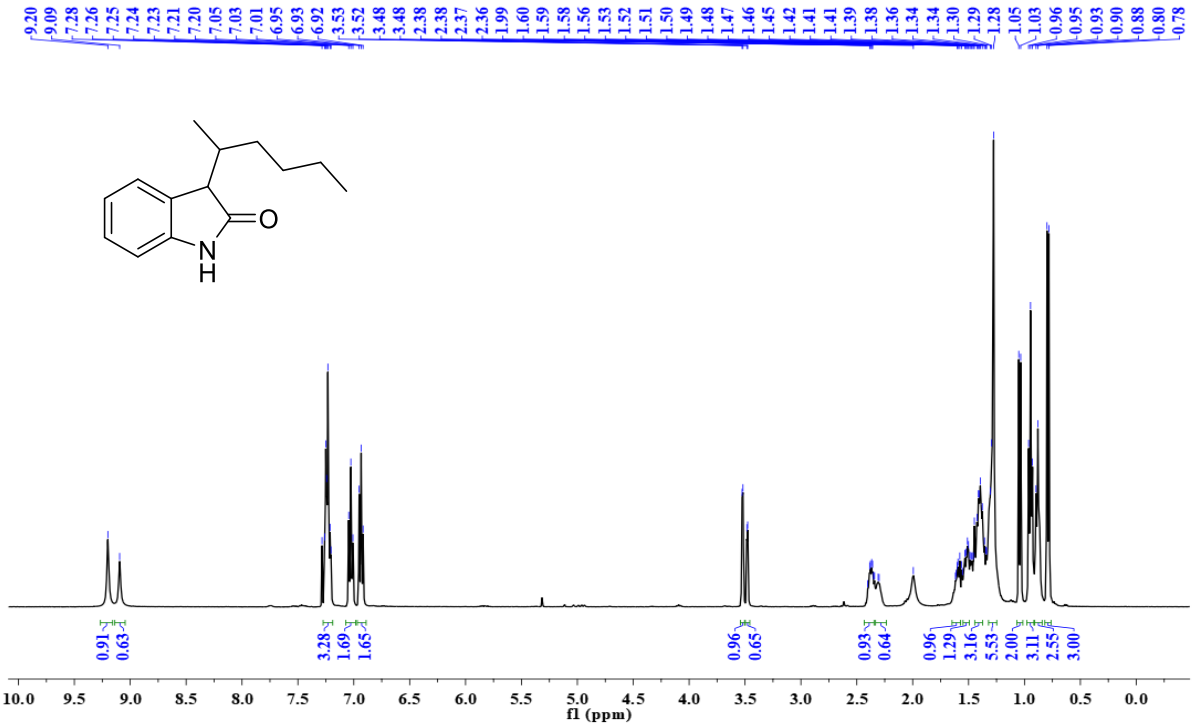


Figure S13. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(hexan-2-yl)indolin-2-one (**P7**).

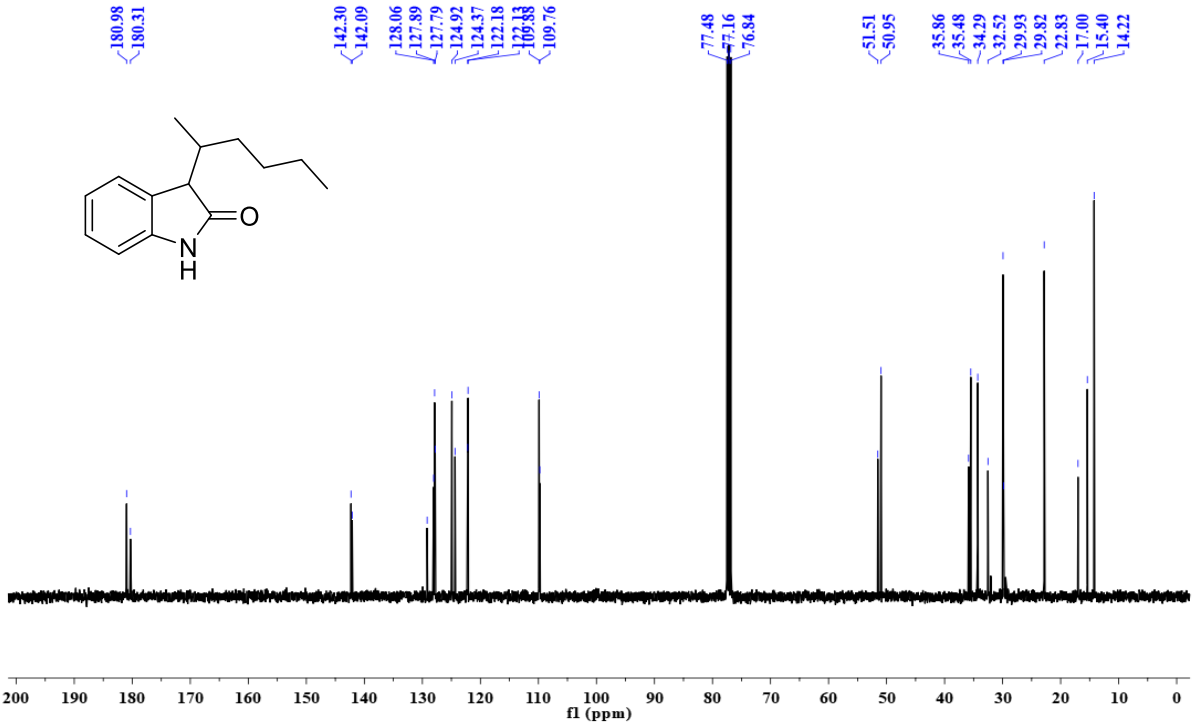


Figure S14. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(hexan-2-yl)indolin-2-one (**P7**).

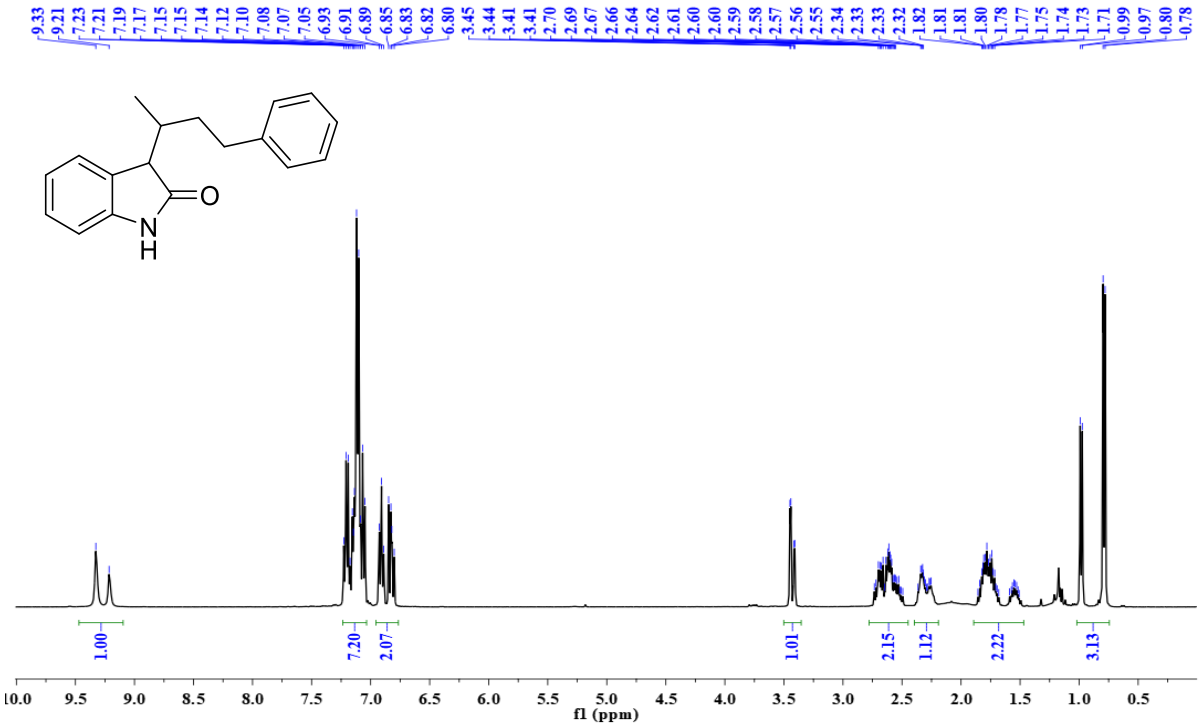


Figure S15. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(4-phenylbutan-2-yl)indolin-2-one (**P8**).

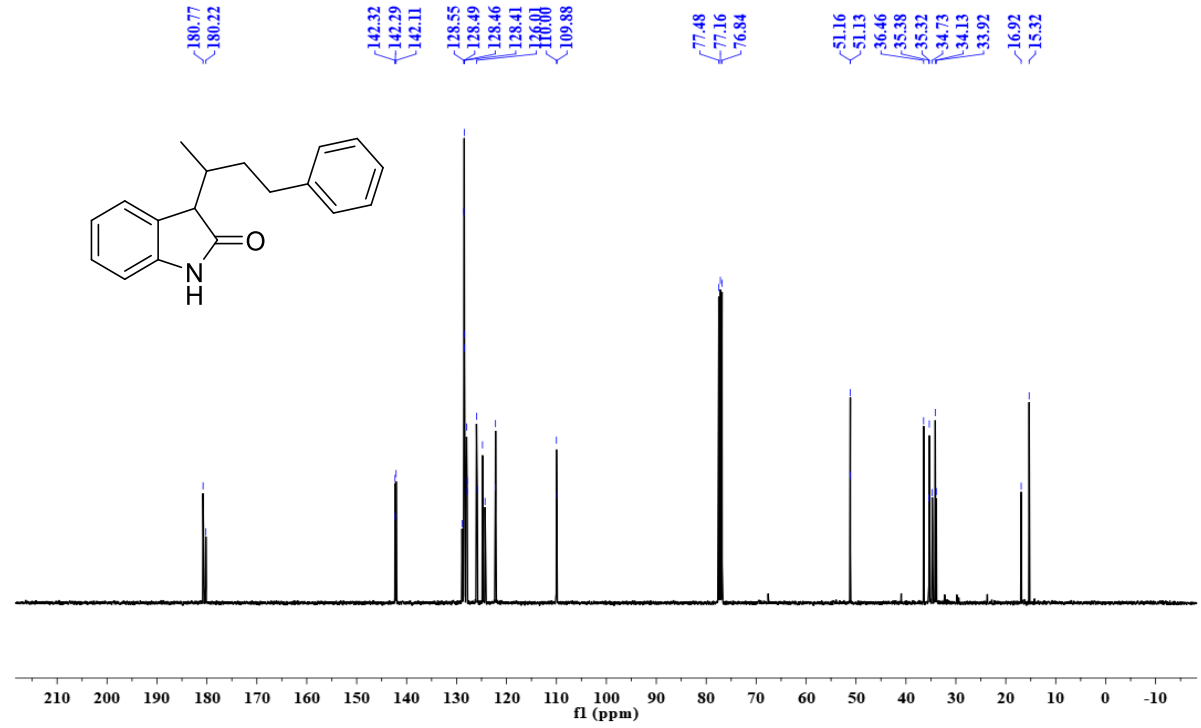


Figure S16. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(4-phenylbutan-2-yl)indolin-2-one (**P8**).

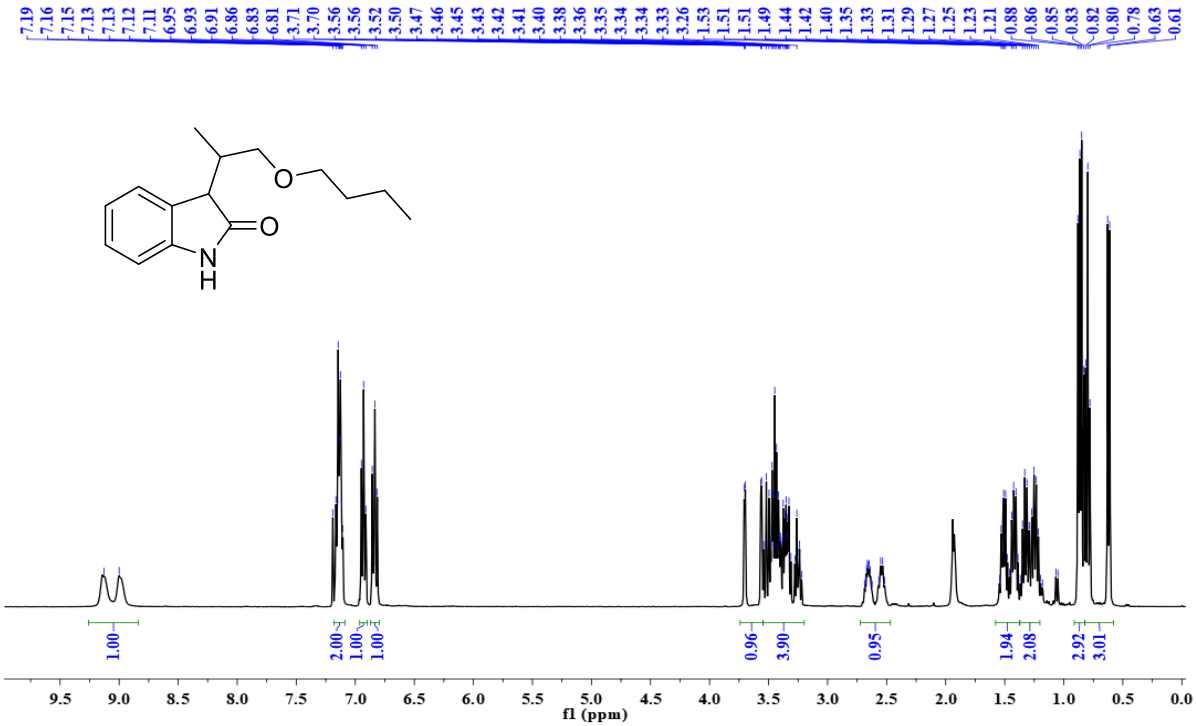


Figure S17. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-butoxypropan-2-yl)indolin-2-one (**P9**).

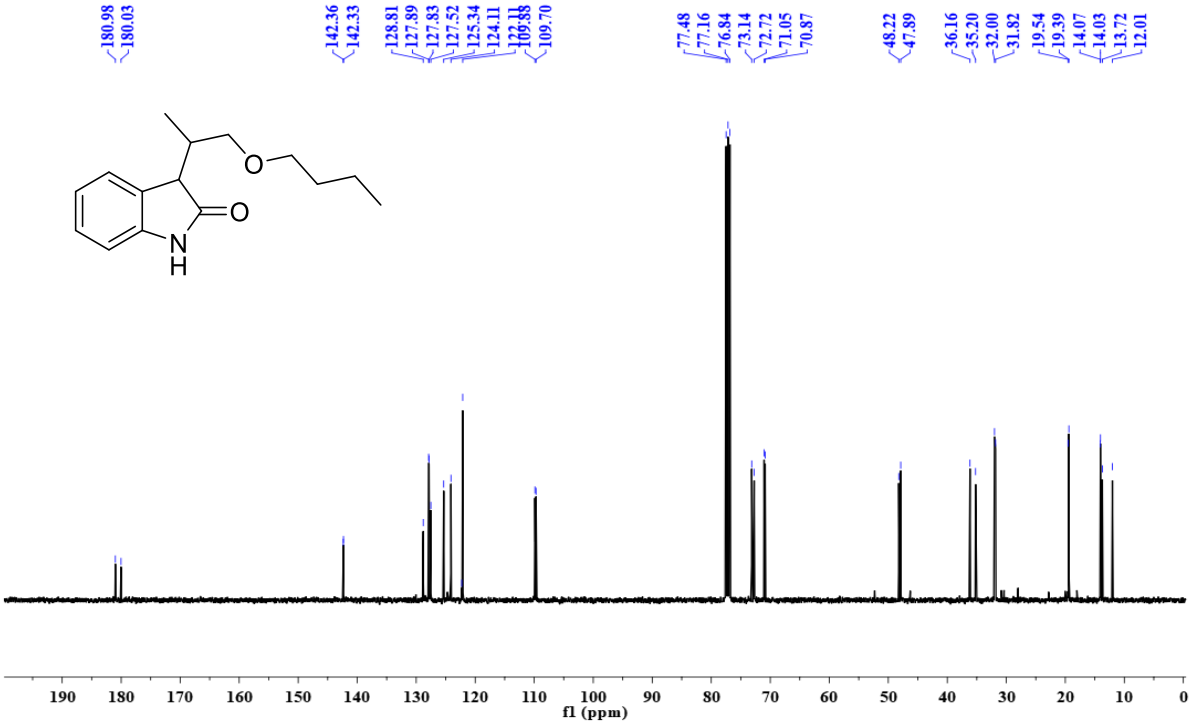


Figure S18. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-butoxypropan-2-yl)indolin-2-one (**P9**).

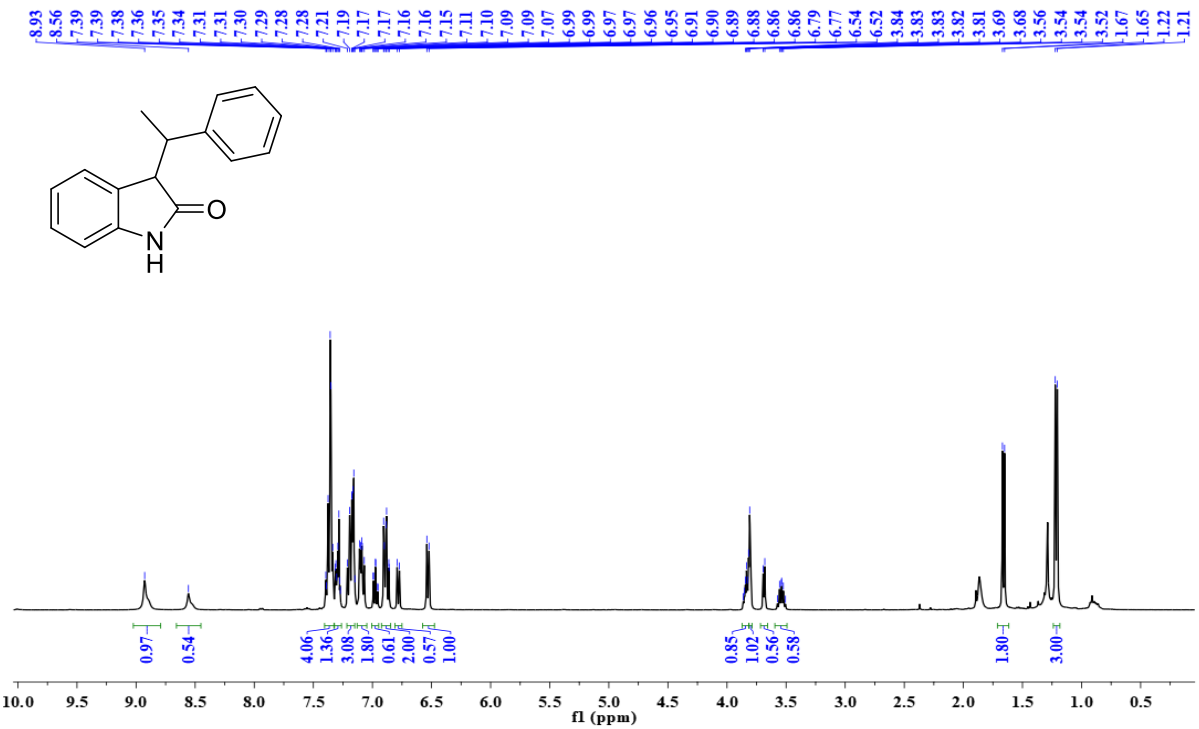


Figure S19. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-phenylethyl)indolin-2-one (**P_{10}**).

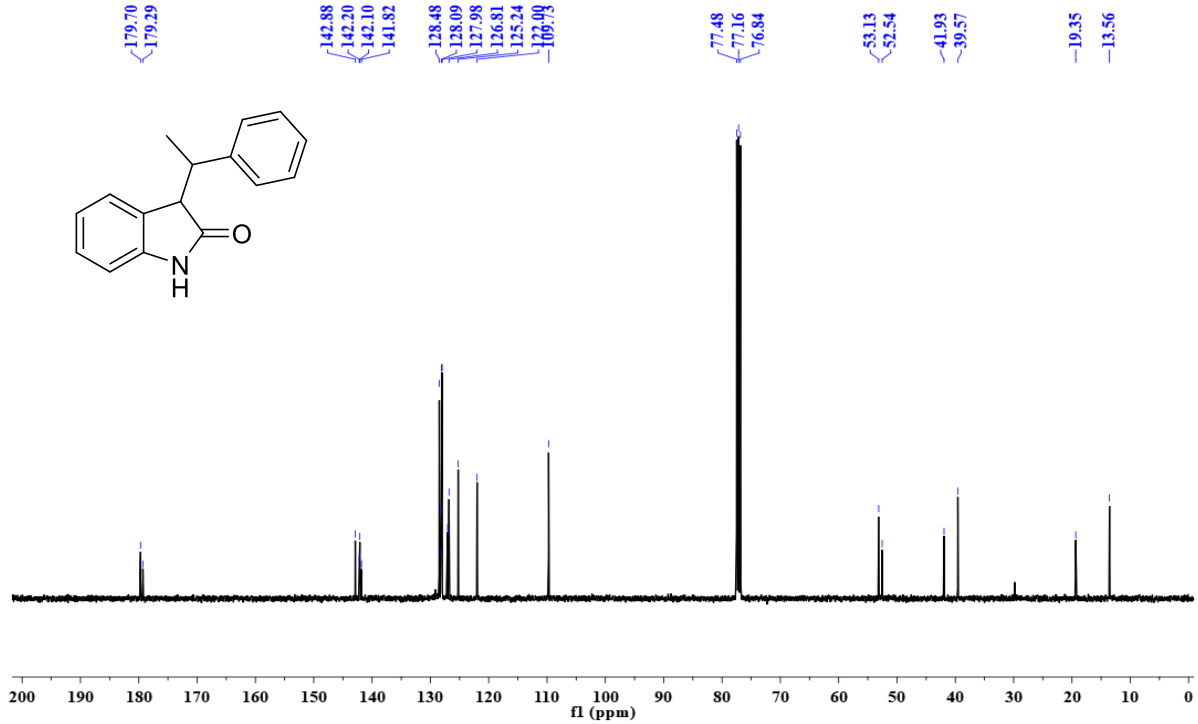


Figure S20. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-phenylethyl)indolin-2-one (**P_{10}**).

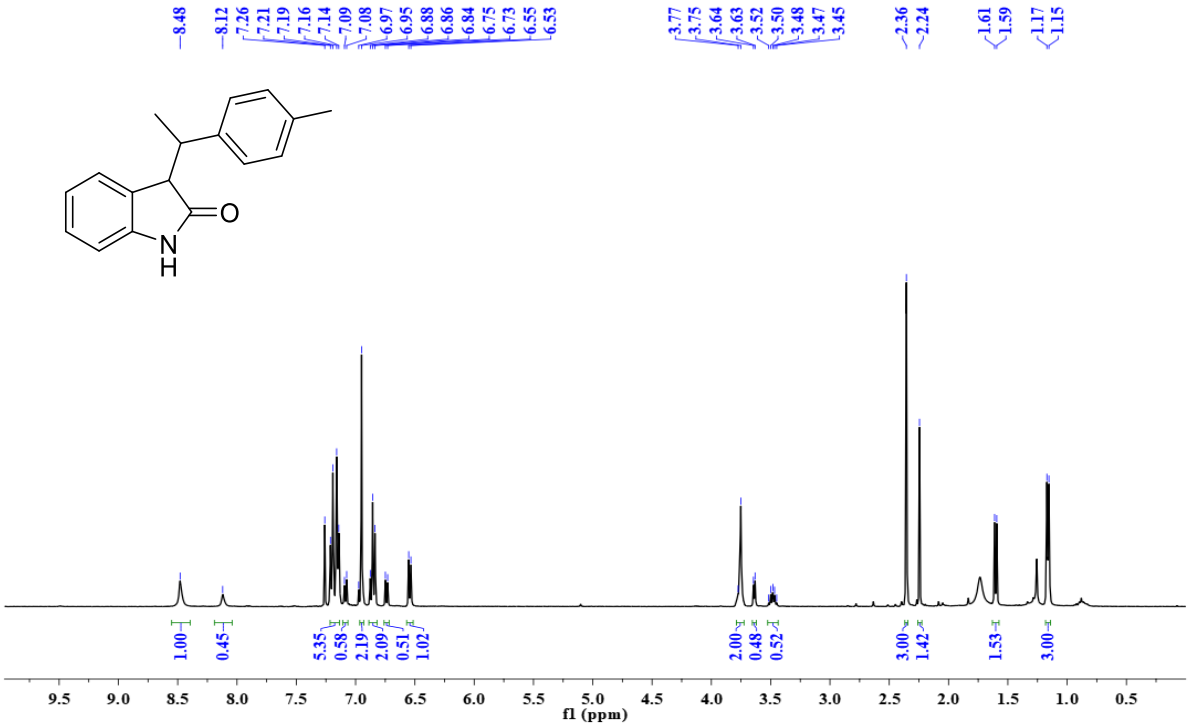


Figure S21. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-(p-tolyl)ethyl)indolin-2-one (**P11**).

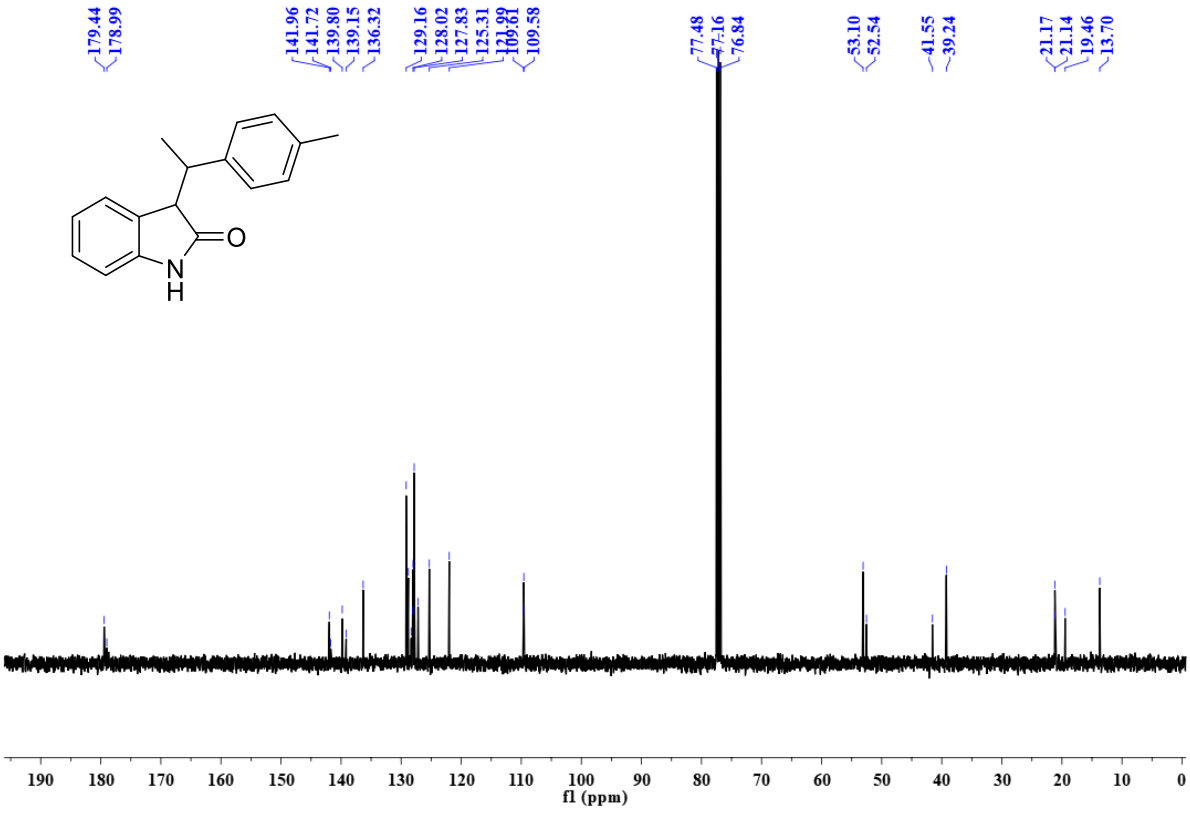


Figure S22. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-(p-tolyl)ethyl)indolin-2-one (**P11**).

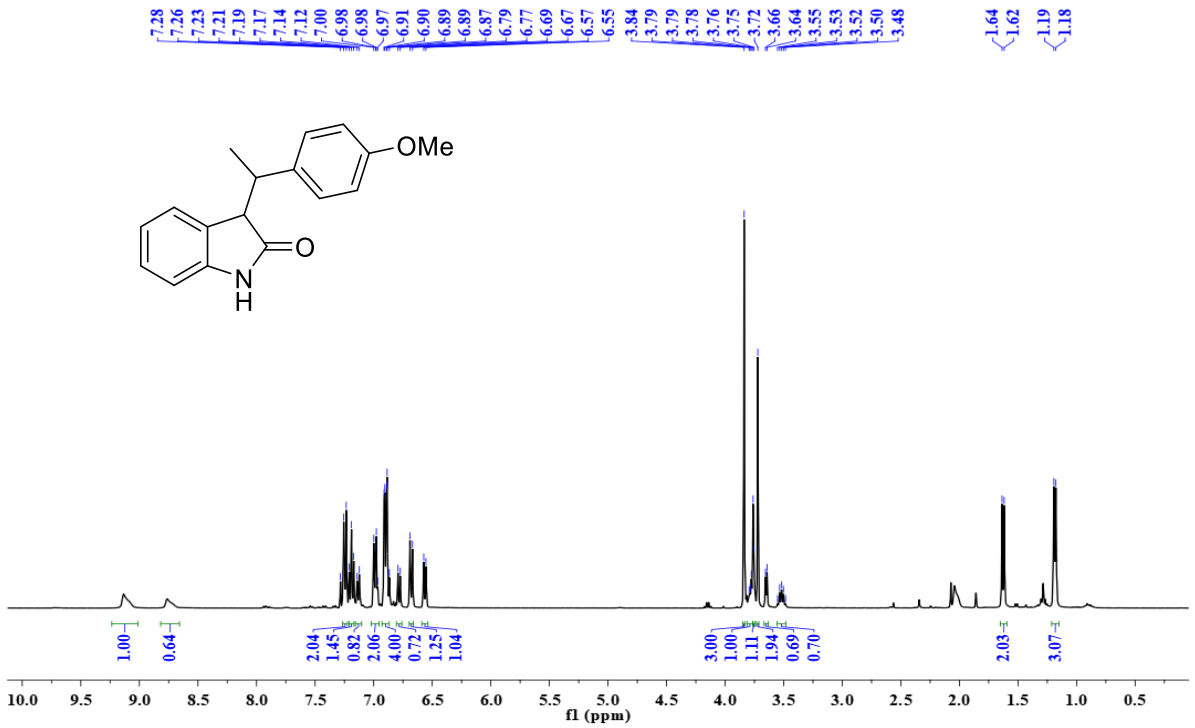


Figure S23. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-(4-methoxyphenyl)ethyl)indolin-2-one (**P12**).

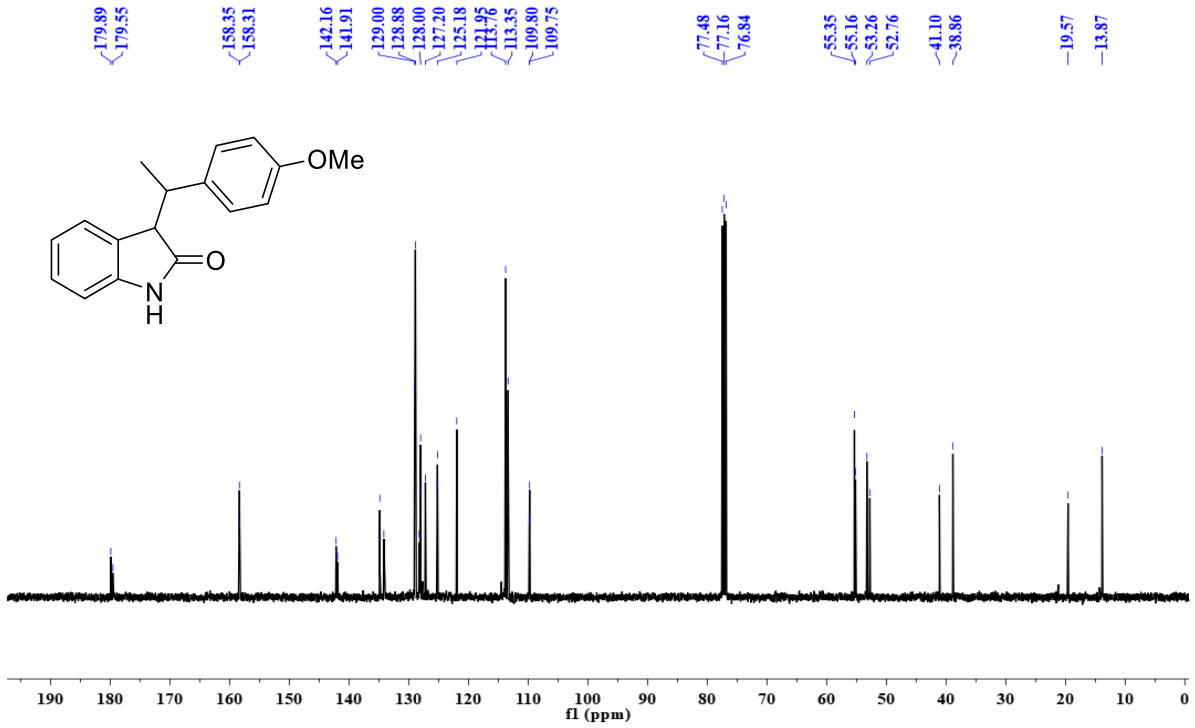


Figure S24. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) of 3-(1-(4-methoxyphenyl)ethyl)indolin-2-one (**P12**).

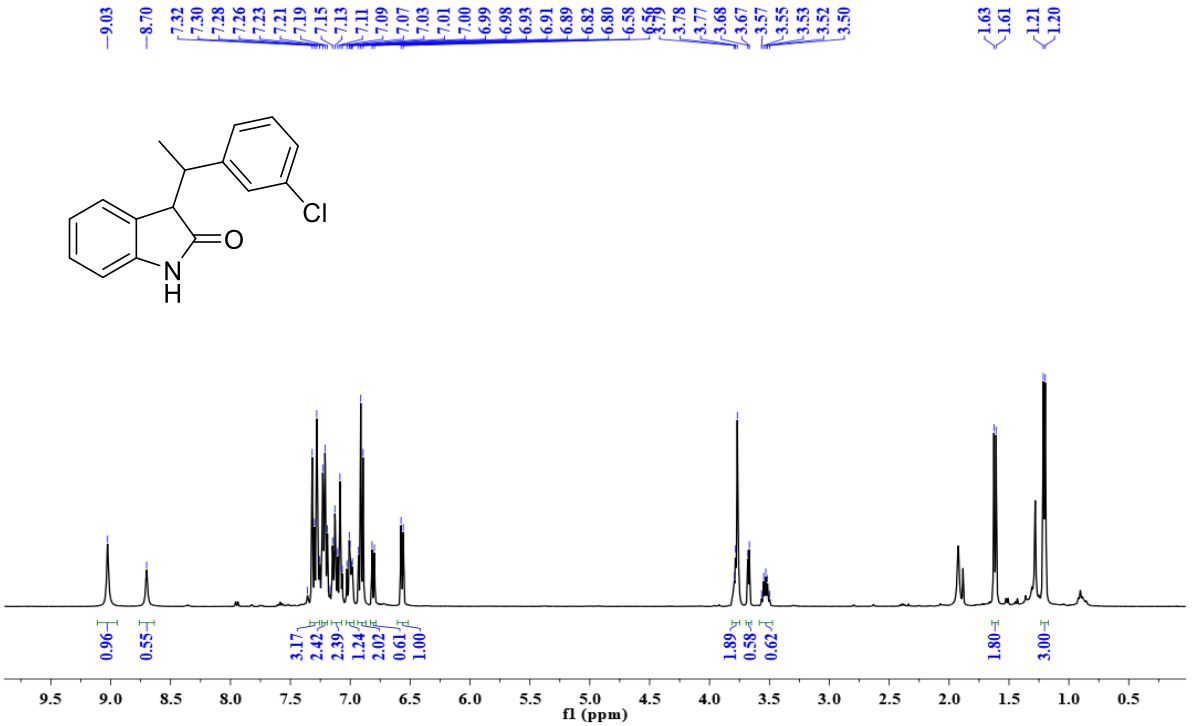


Figure S25. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-(3-chlorophenyl)ethyl)indolin-2-one (**P13**).

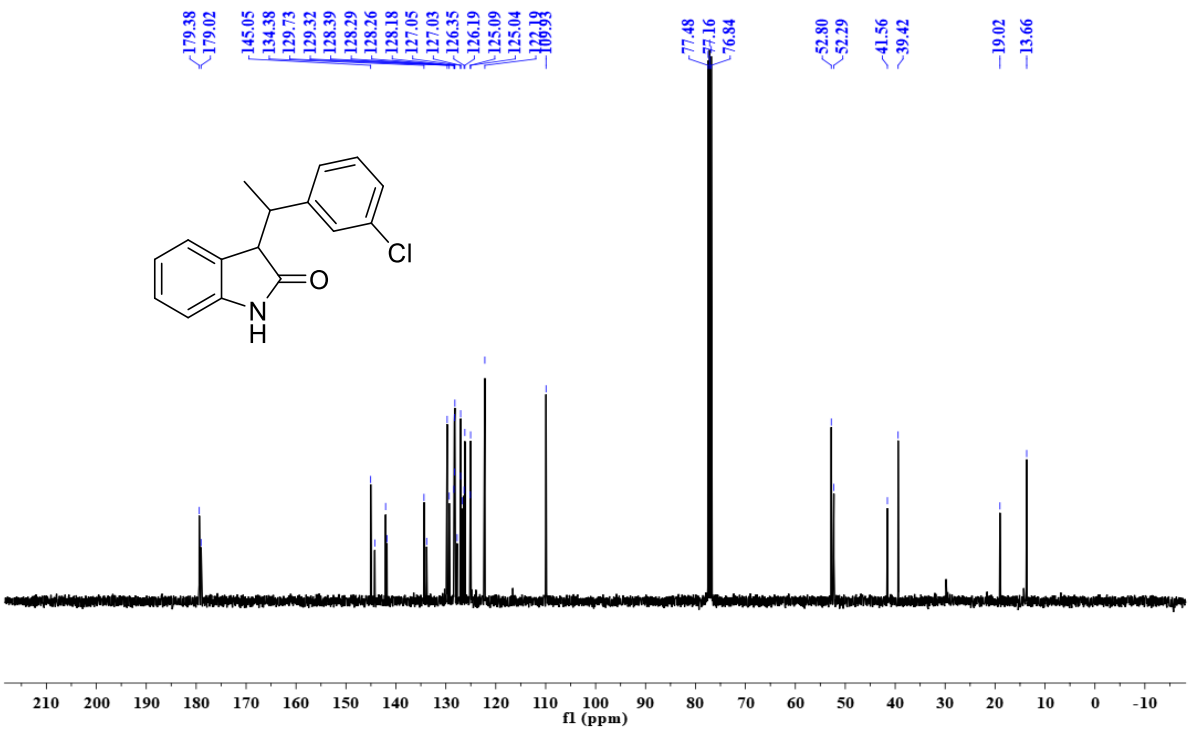


Figure S26. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-(3-chlorophenyl)ethyl)indolin-2-one (**P13**).

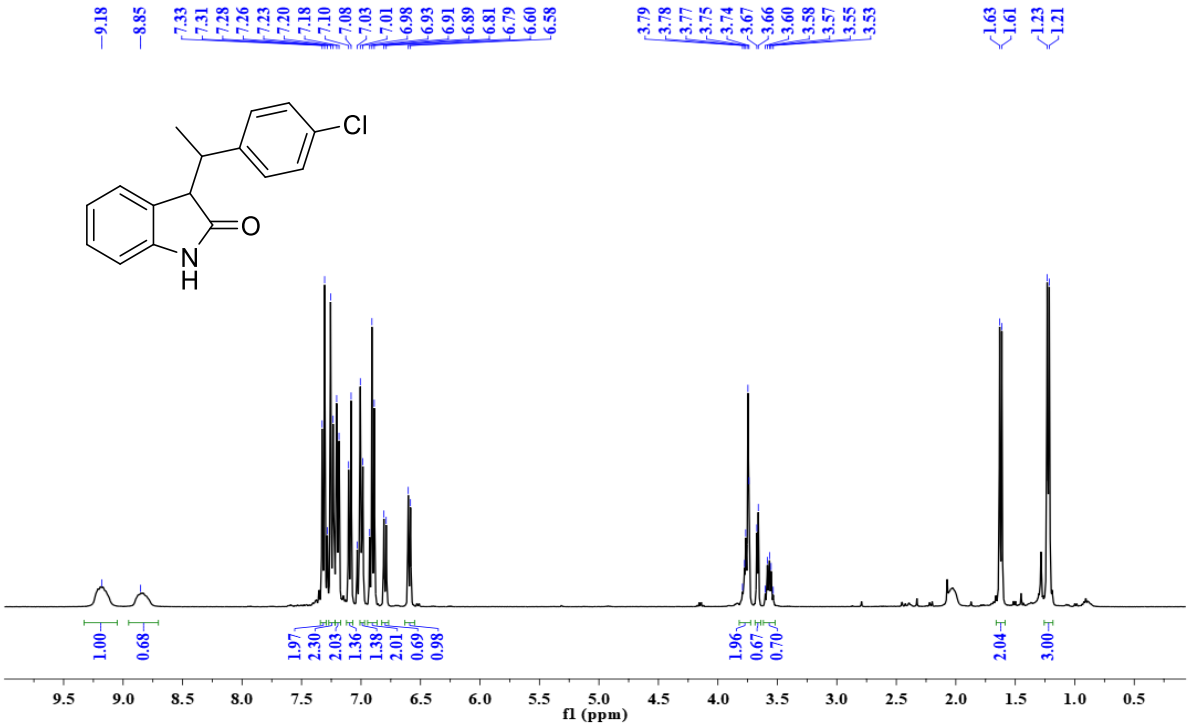


Figure S27. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-(4-chlorophenyl)ethyl)indolin-2-one (**P14**).

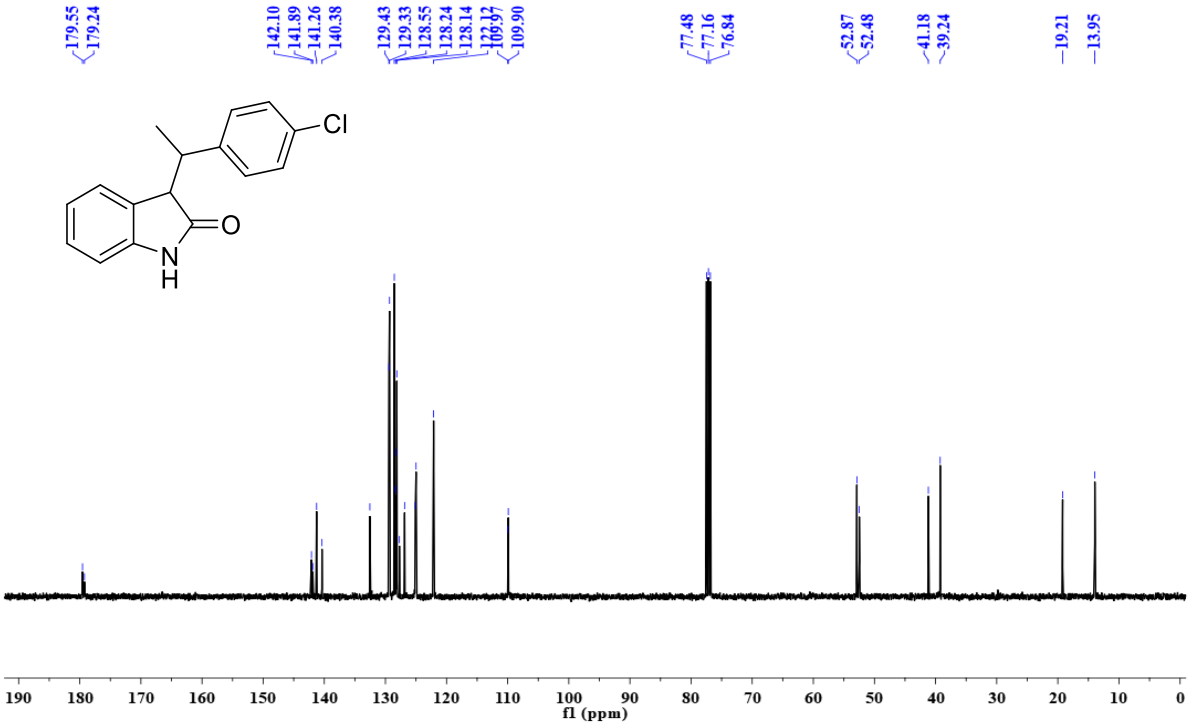


Figure S28. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-(4-chlorophenyl)ethyl)indolin-2-one (**P14**).

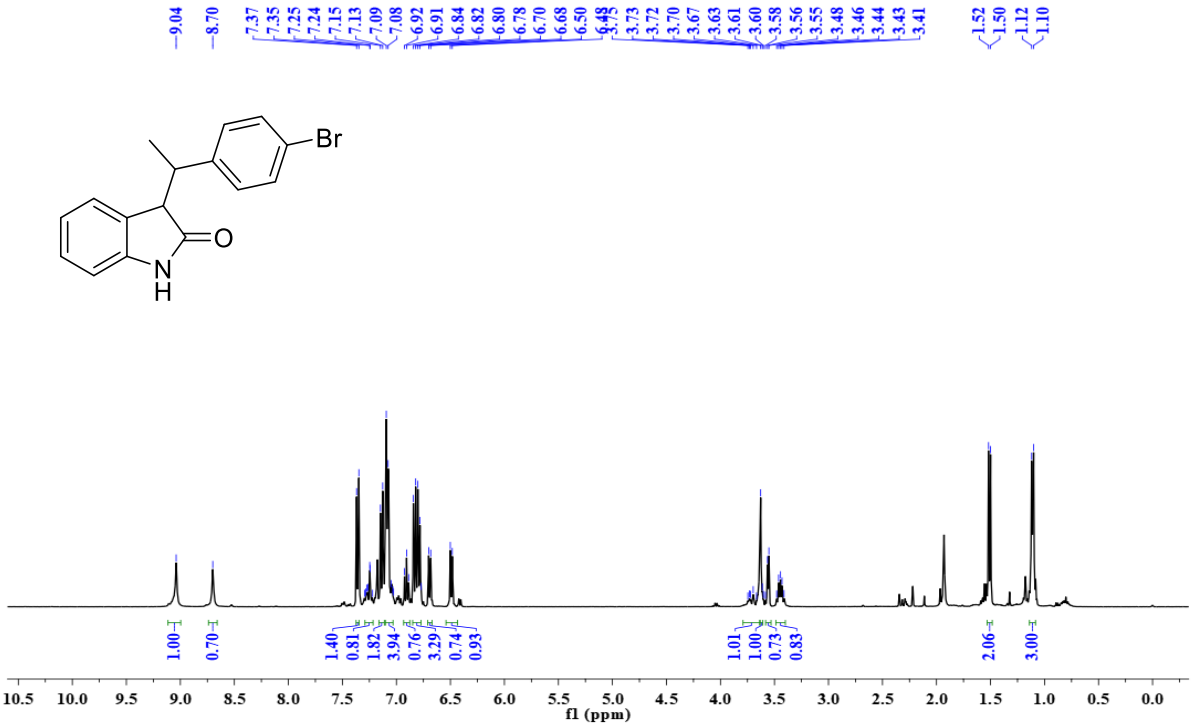


Figure S29. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-(4-bromophenyl)ethyl)indolin-2-one (**P15**).

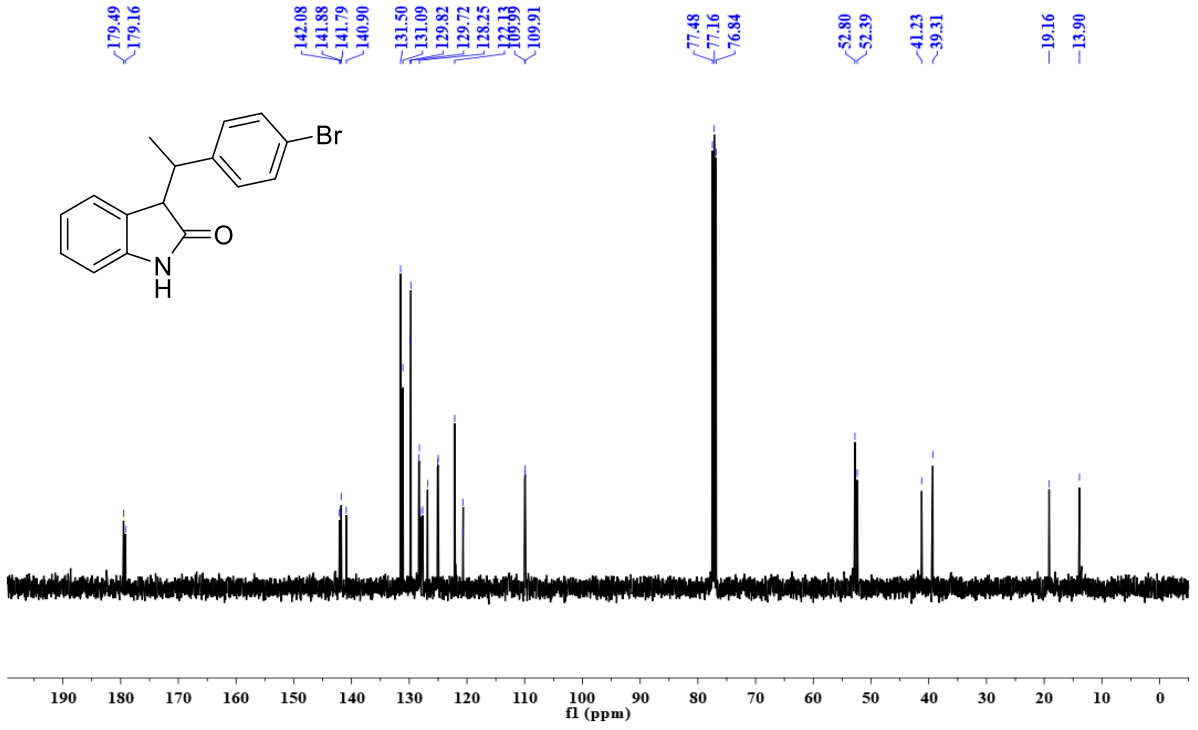


Figure S30. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-(4-bromophenyl)ethyl)indolin-2-one (**P15**).

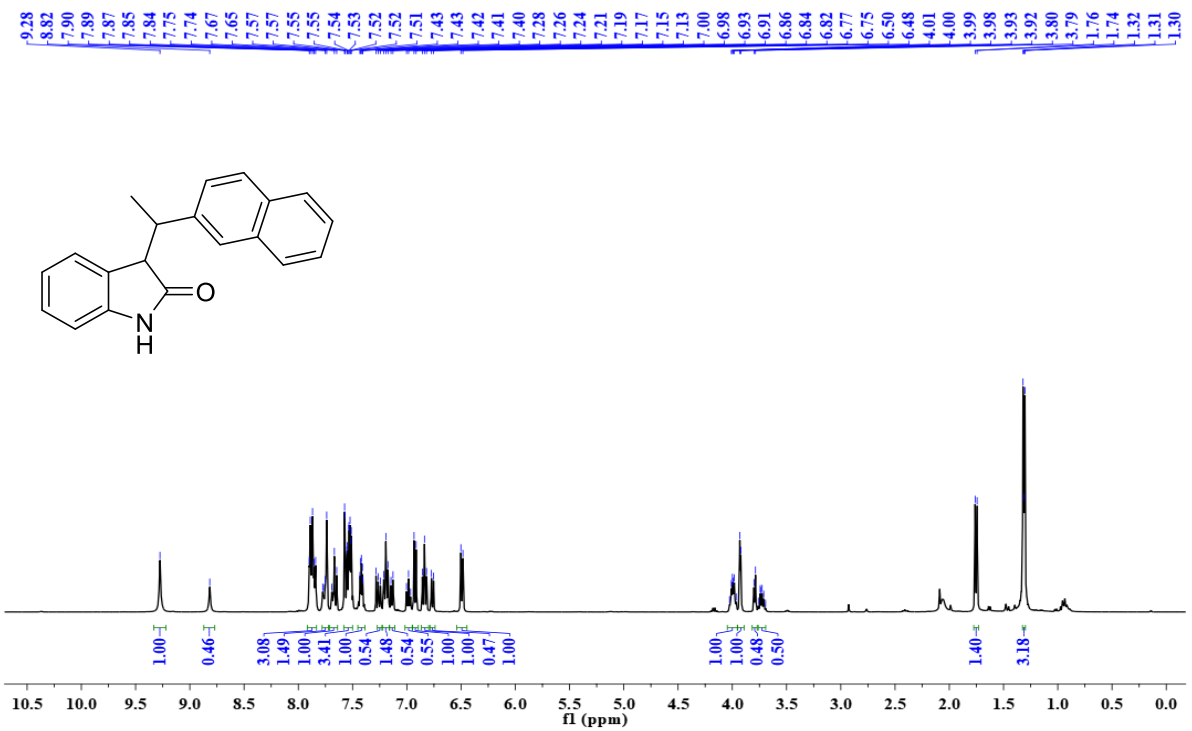


Figure S31. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-(naphthalen-2-yl)ethyl)indolin-2-one (**P16**).

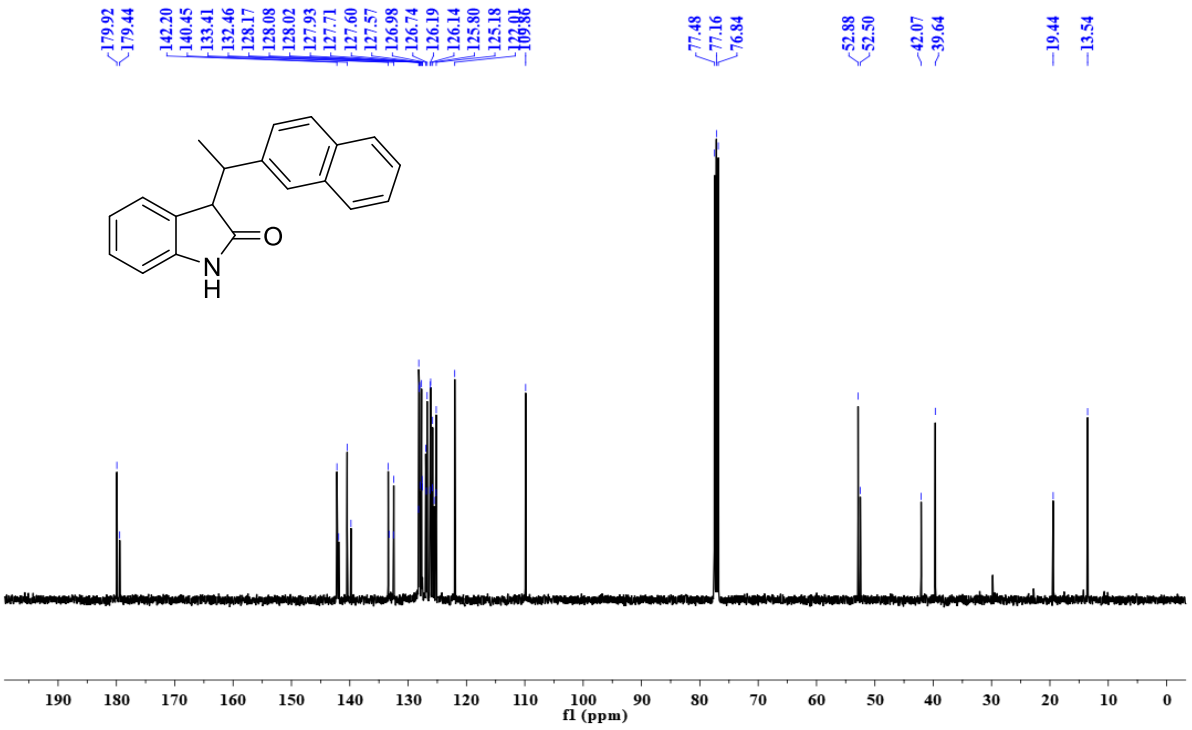


Figure S32. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-(naphthalen-2-yl)ethyl)indolin-2-one (**P16**).

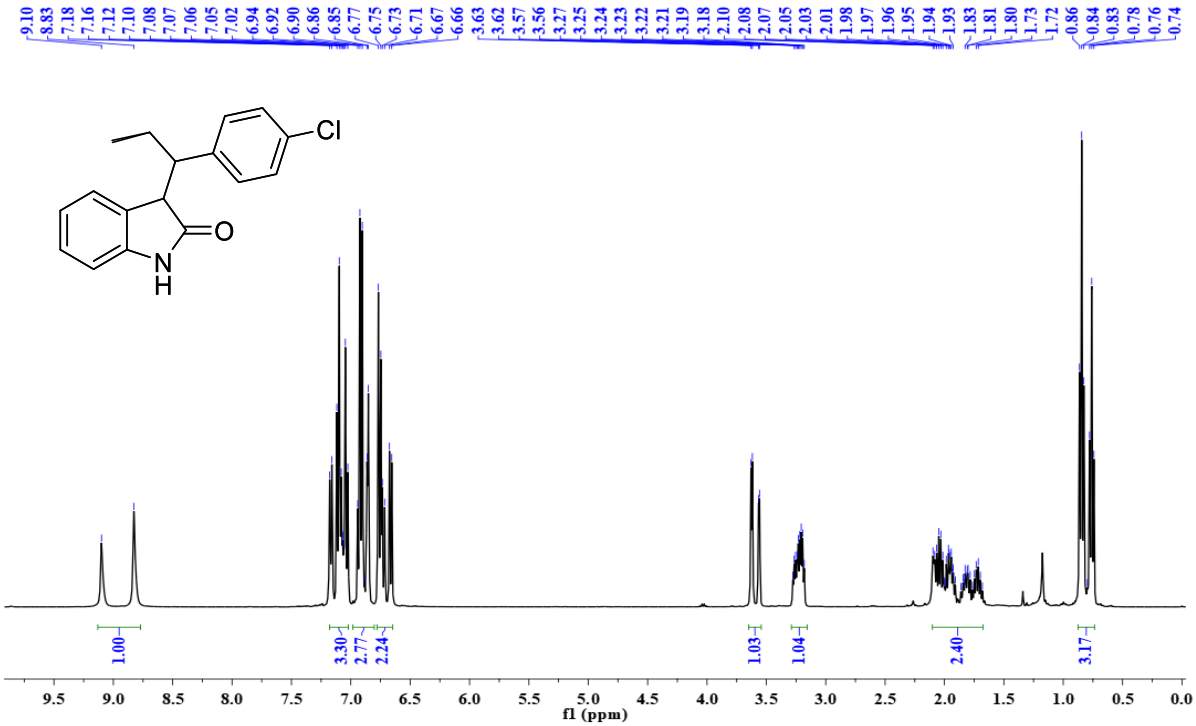


Figure S33. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-(4-chlorophenyl)propyl)indolin-2-one (**P17**).

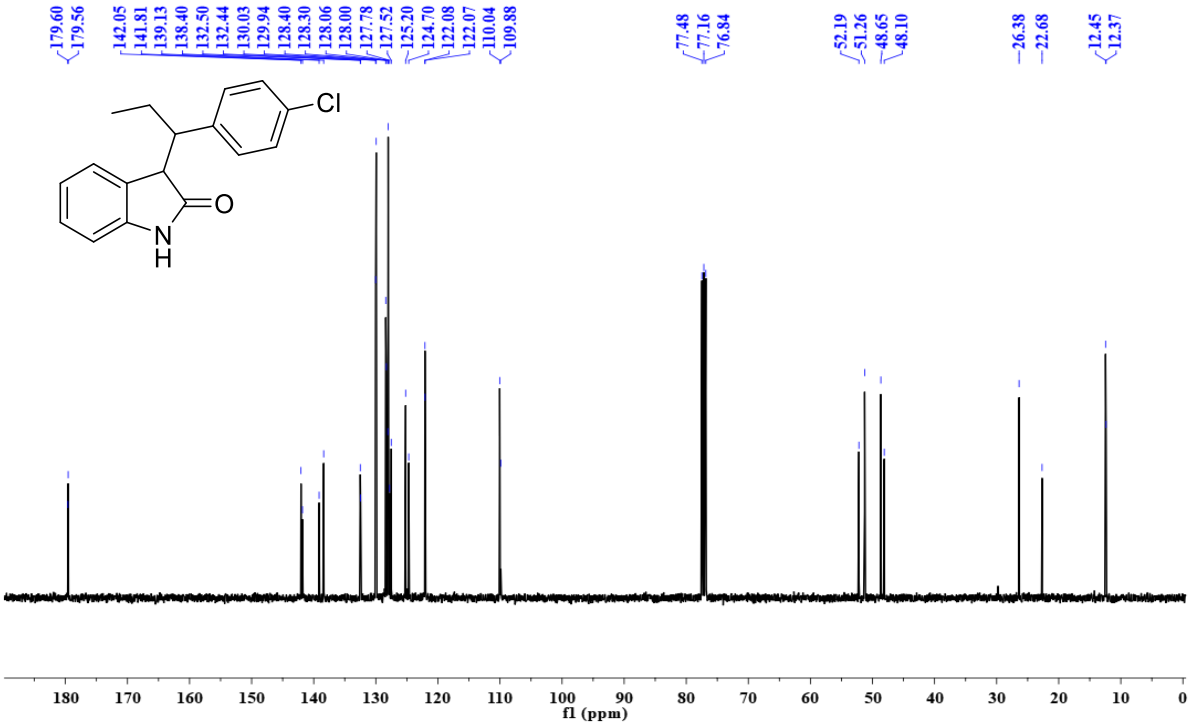


Figure S34. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-(4-chlorophenyl)propyl)indolin-2-one (**P17**).

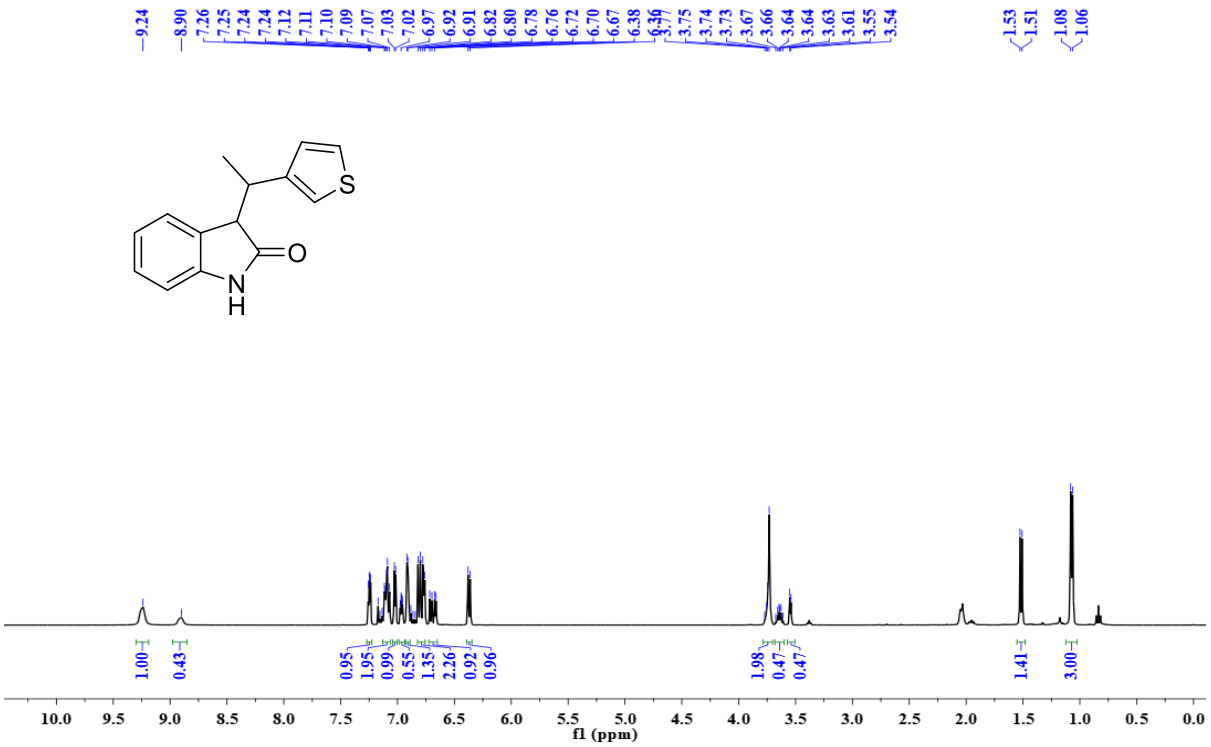


Figure S35. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-(thiophen-3-yl)ethyl)indolin-2-one (**P18**).

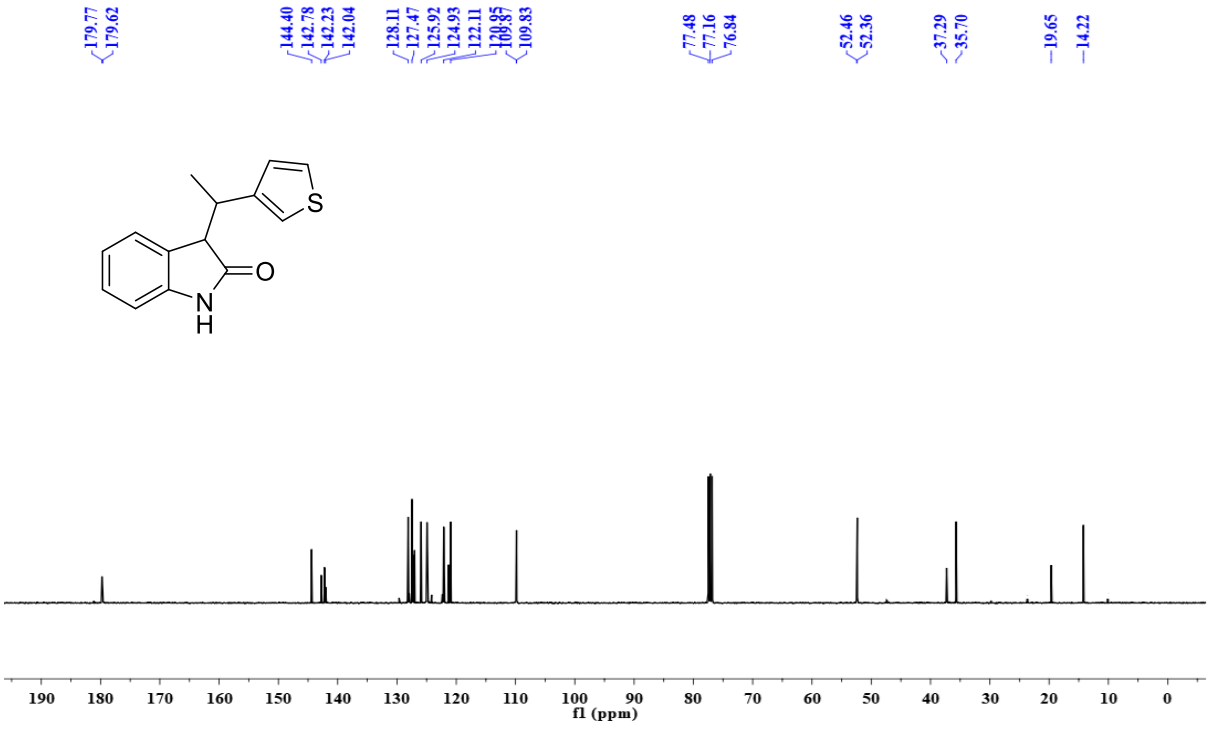


Figure S36. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-(thiophen-3-yl)ethyl)indolin-2-one (**P18**).

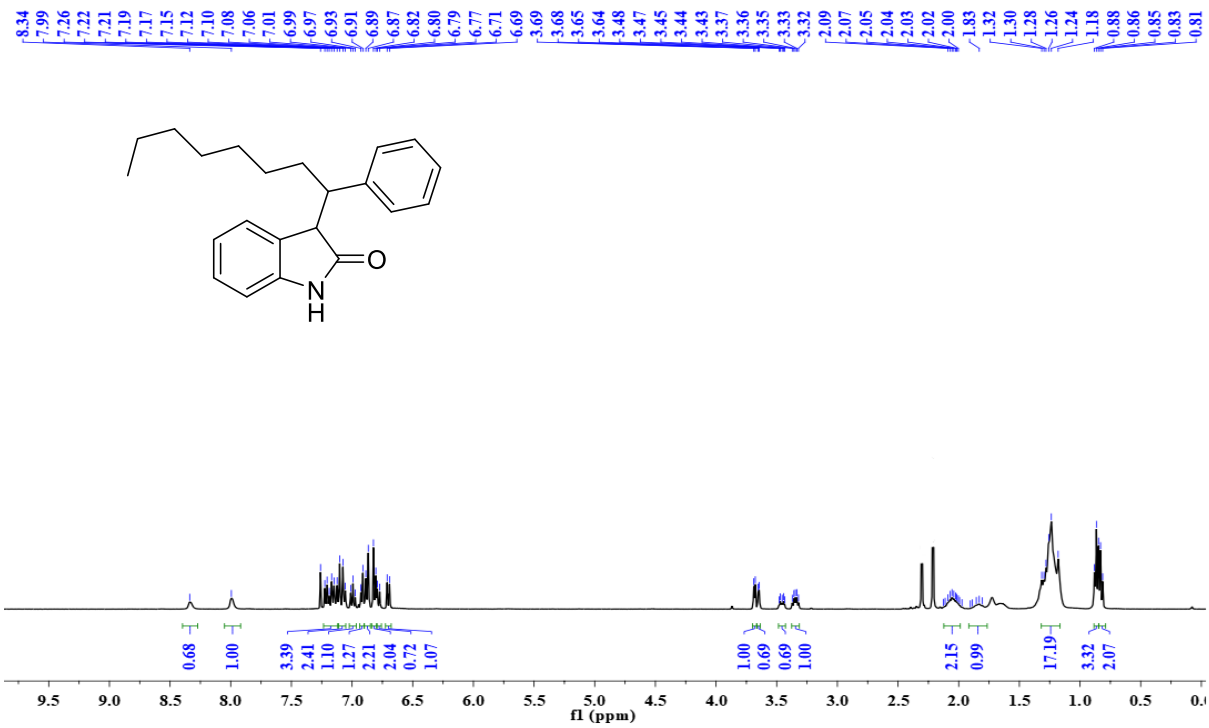


Figure S37. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-phenyloctyl)indolin-2-one (**P19**).

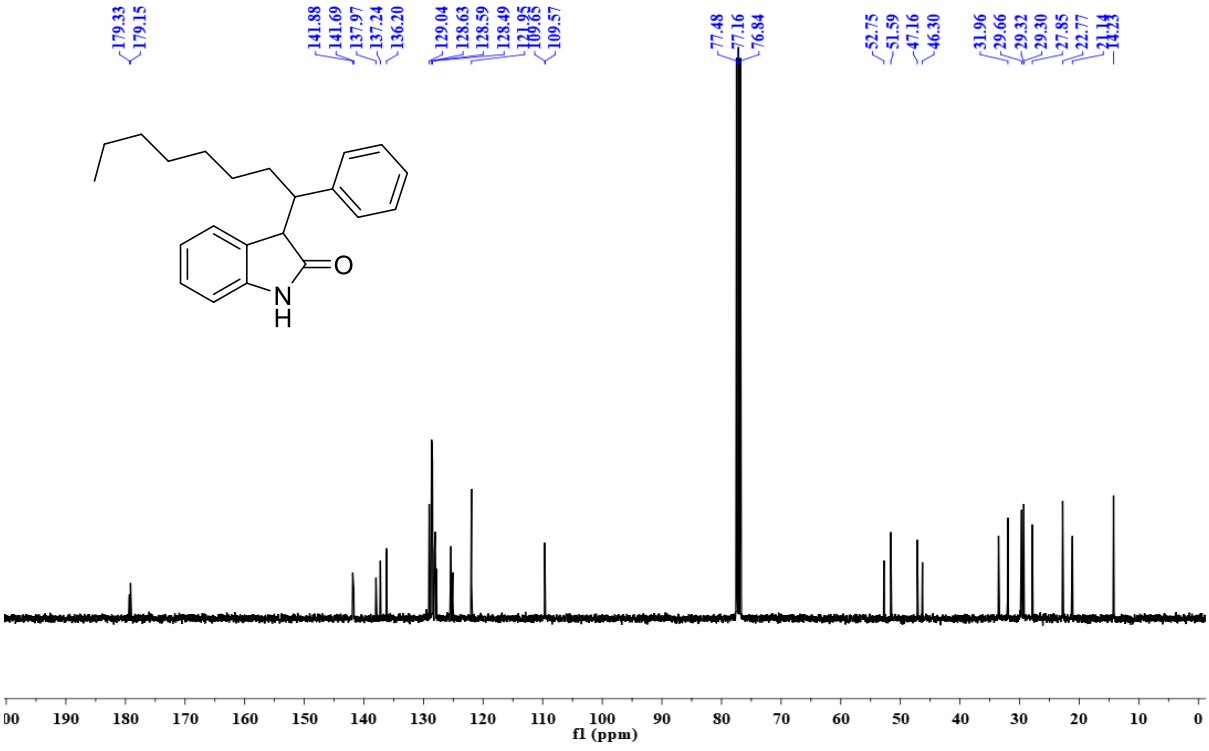


Figure S38. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) of 3-(1-phenyloctyl)indolin-2-one (**P19**).

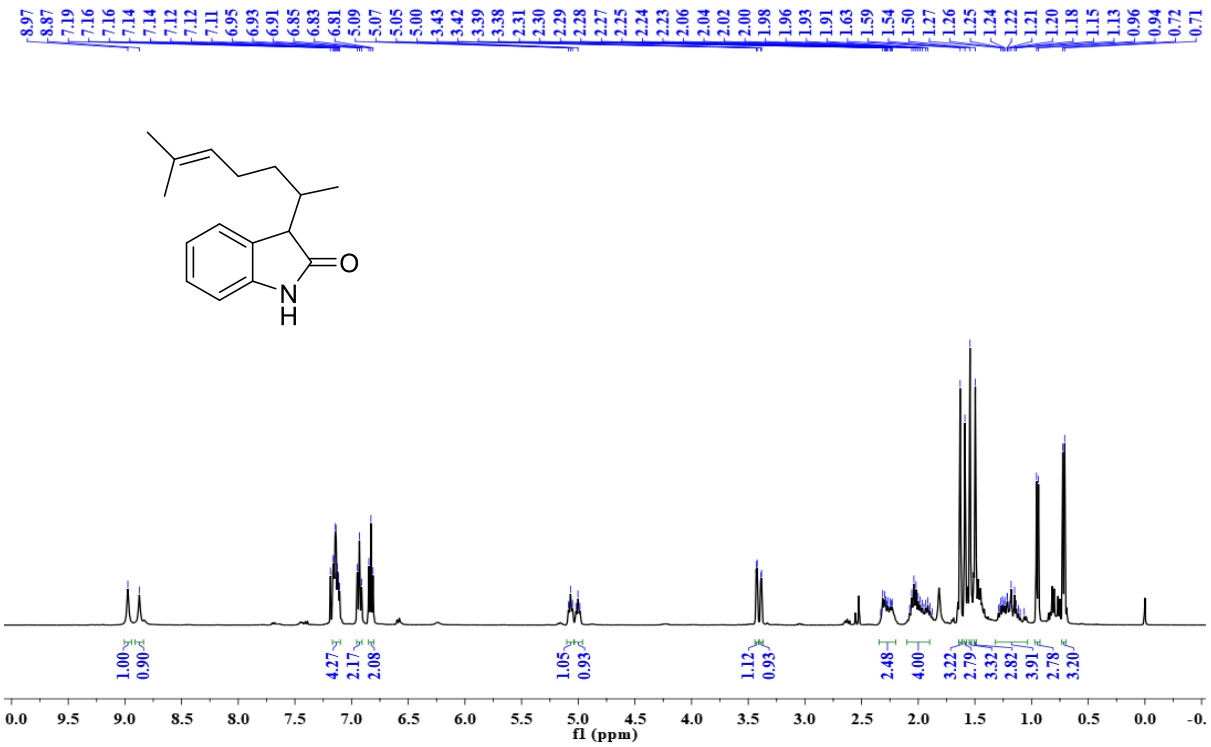


Figure S39. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(6-methylhept-5-en-2-yl)indolin-2-one (**P20**).

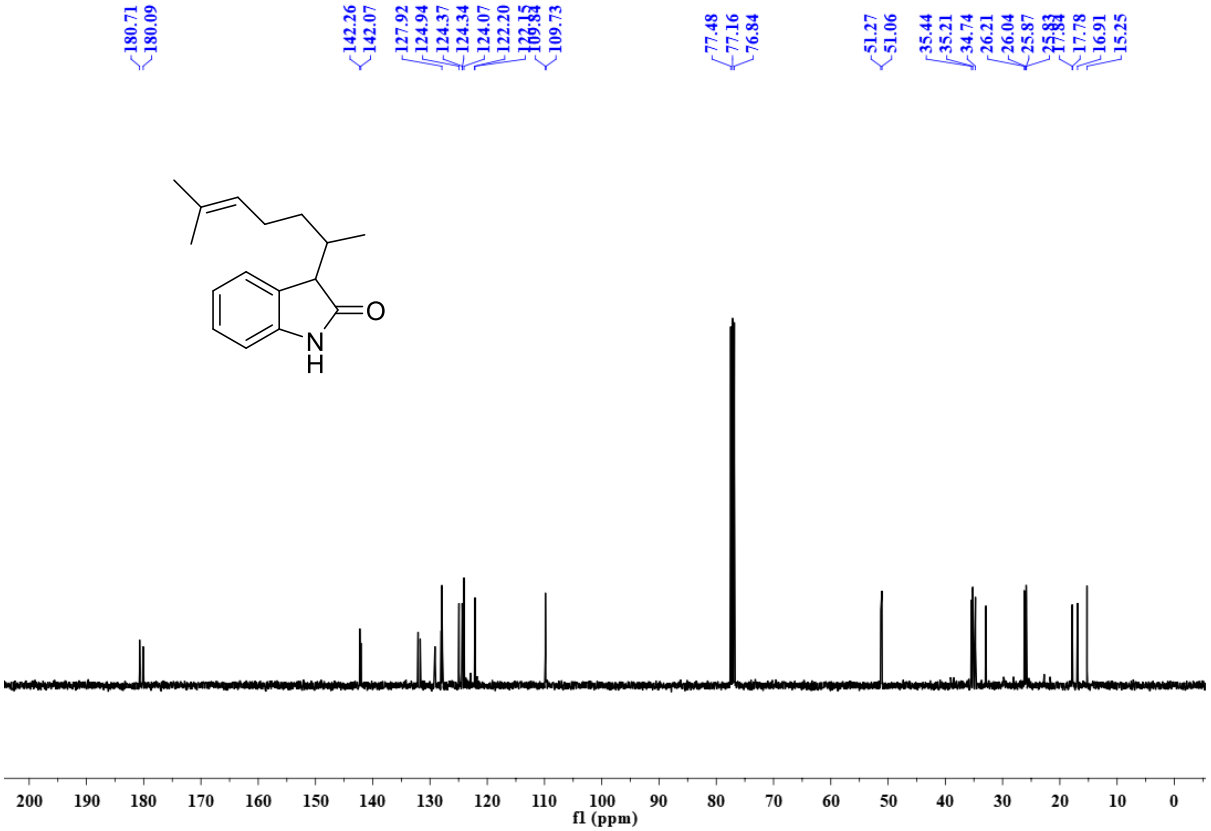


Figure S40. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(6-methylhept-5-en-2-yl)indolin-2-one (**P20**).

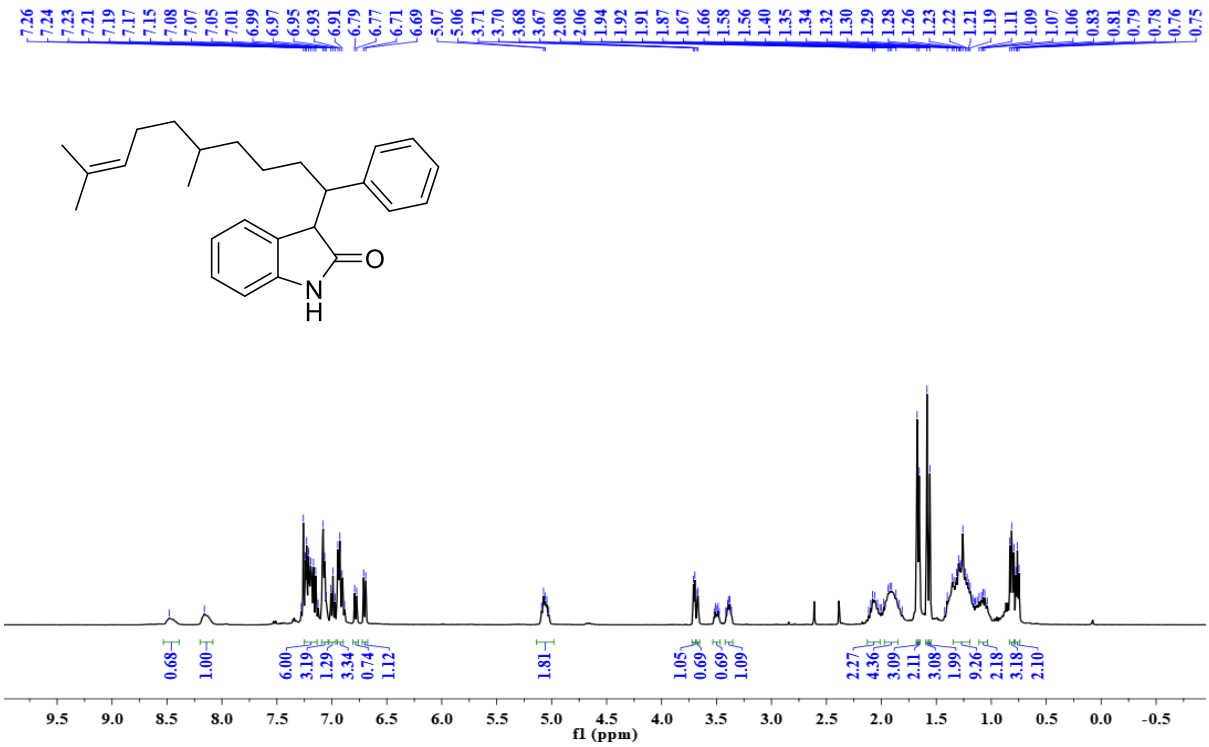


Figure S41. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(5,9-dimethyl-1-phenyldec-8-en-1-yl)indolin-2-one (**P21**).

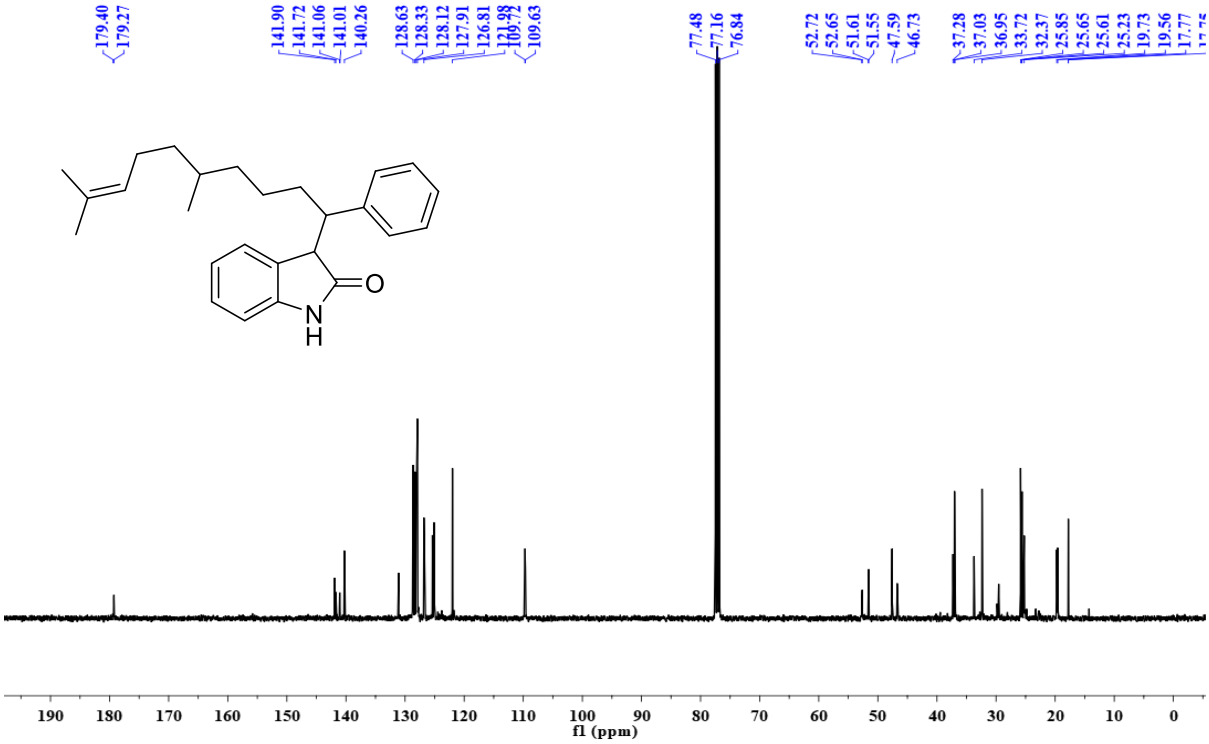


Figure S42. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(5,9-dimethyl-1-phenyldec-8-en-1-yl)indolin-2-one (**P21**).

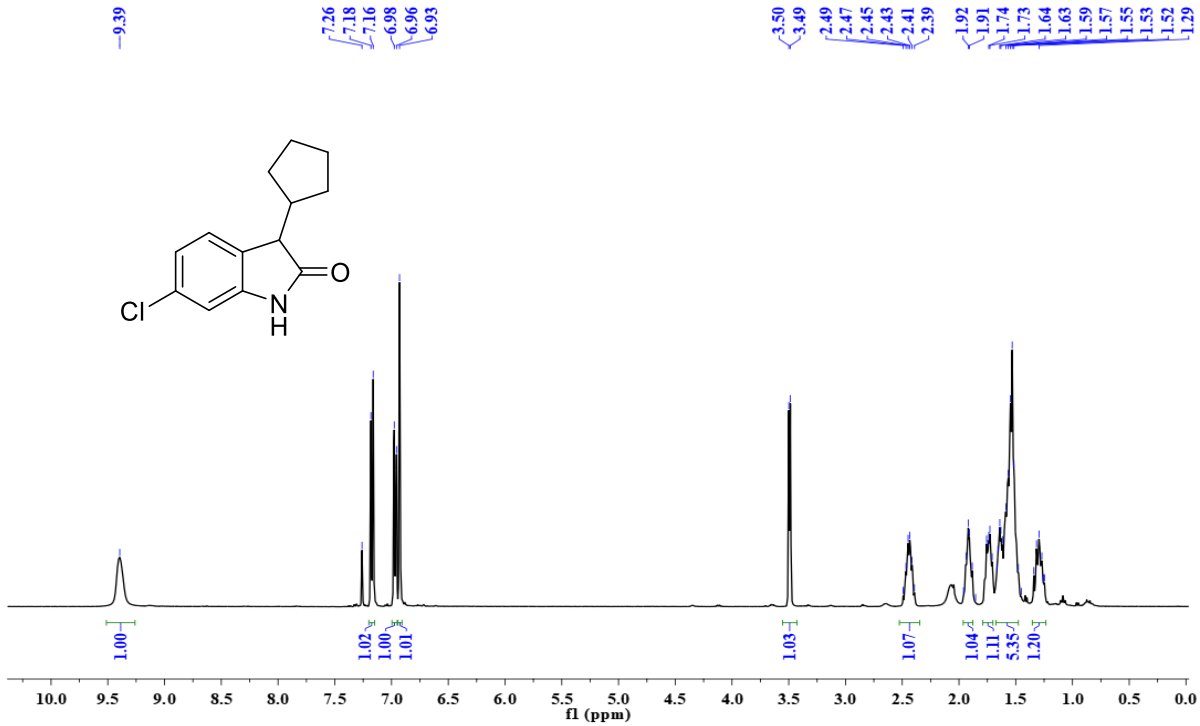


Figure S43. ^{1}H NMR (400 MHz, $CDCl_3$) of 6-chloro-3-cyclopentylindolin-2-one (**P22**).

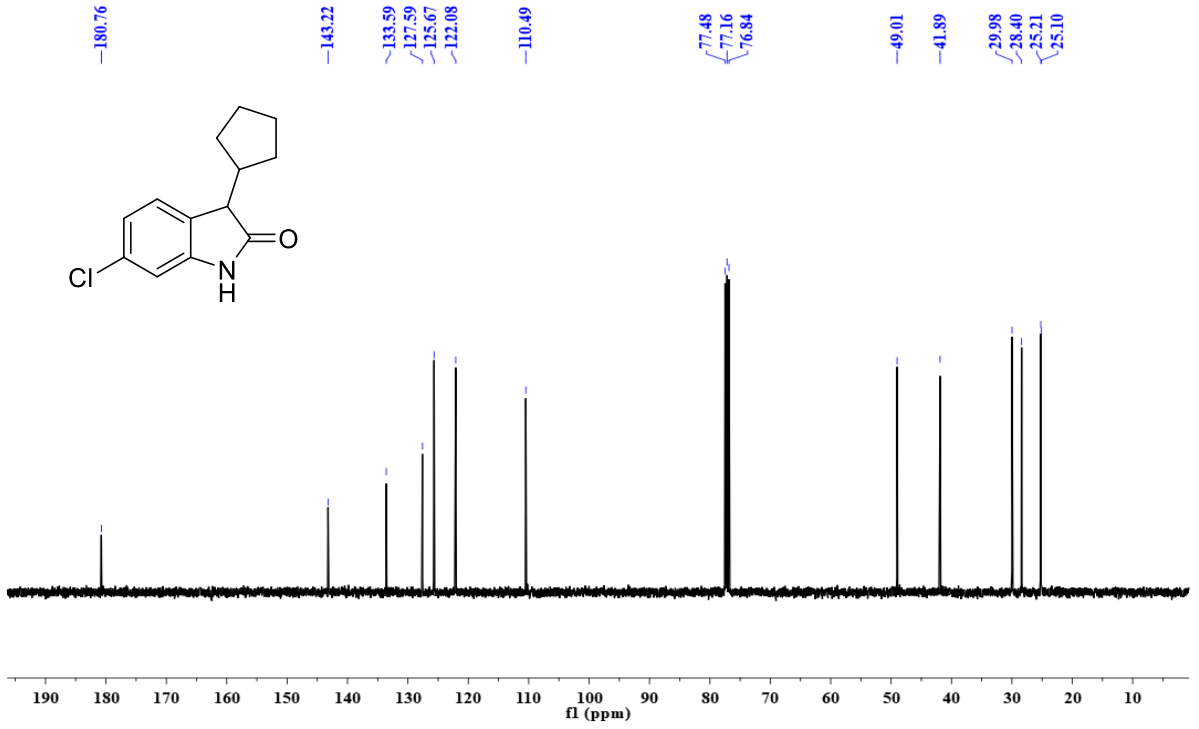


Figure S44. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 6-chloro-3-cyclopentylindolin-2-one (**P22**).

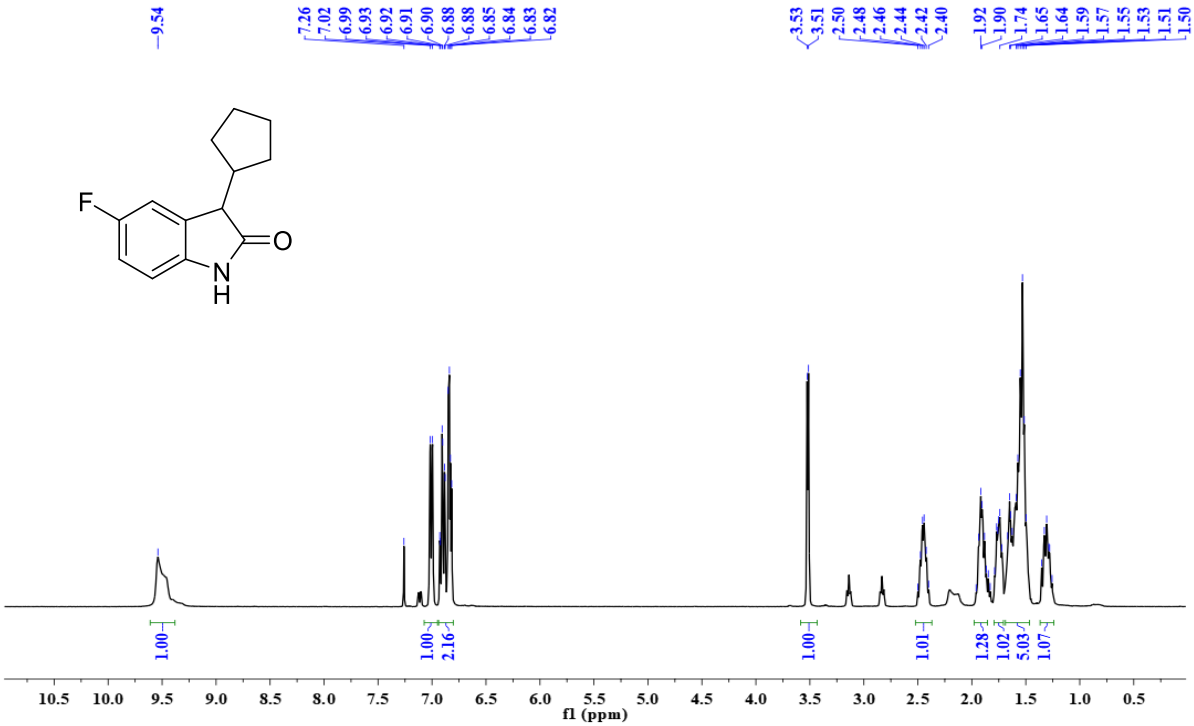


Figure S45. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cyclopentyl-5-fluoroindolin-2-one (**P23**).

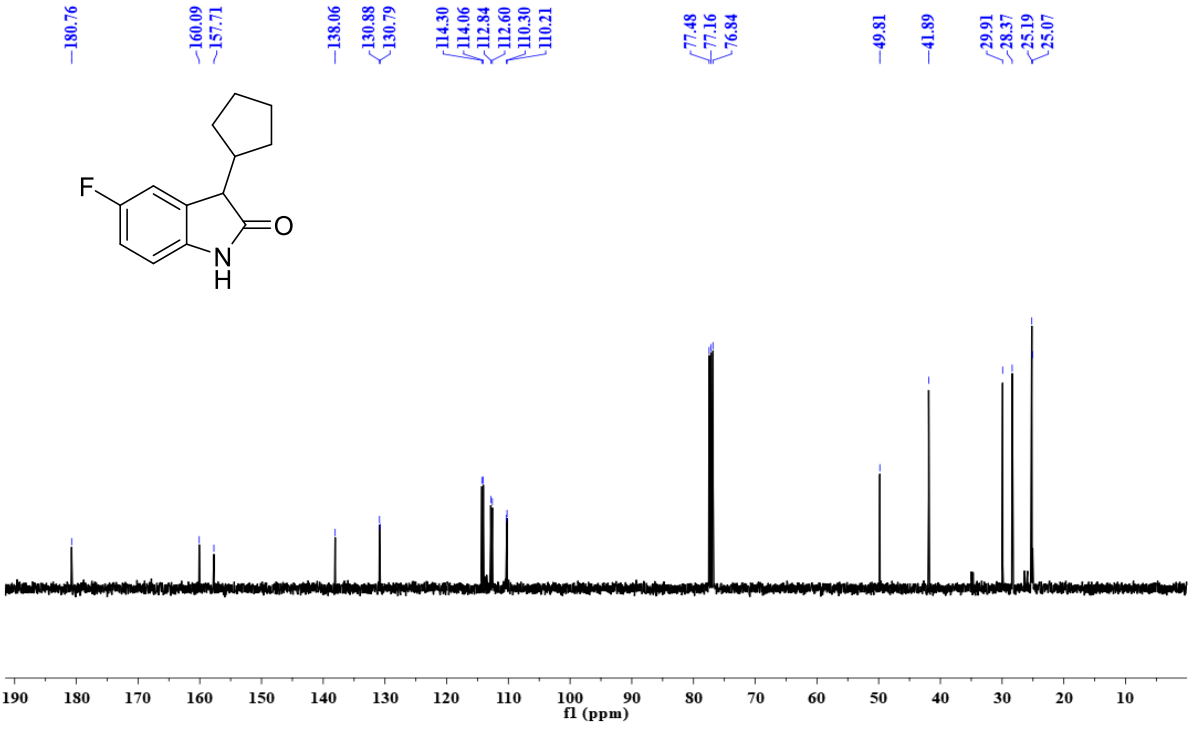


Figure S46. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-cyclopentyl-5-fluoroindolin-2-one (**P23**).

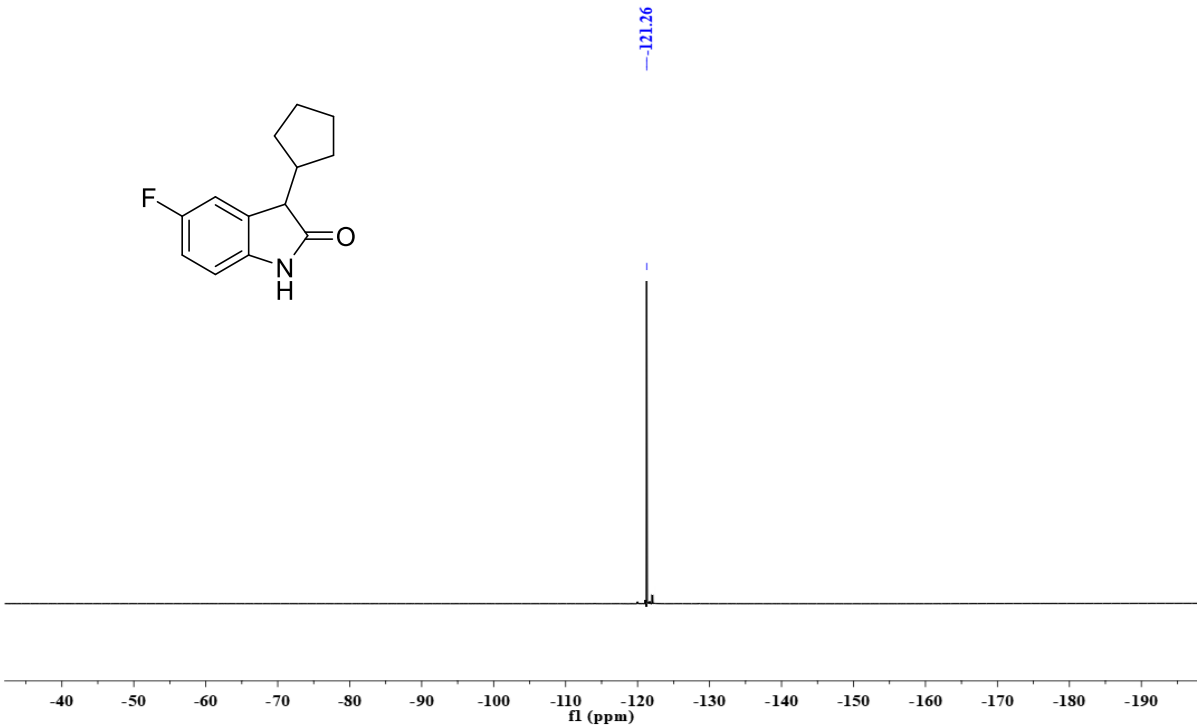


Figure S47. $^{19}F\{^{1}H\}$ NMR (377 MHz, $CDCl_3$) of 3-cyclopentyl-5-fluoroindolin-2-one (**P23**).

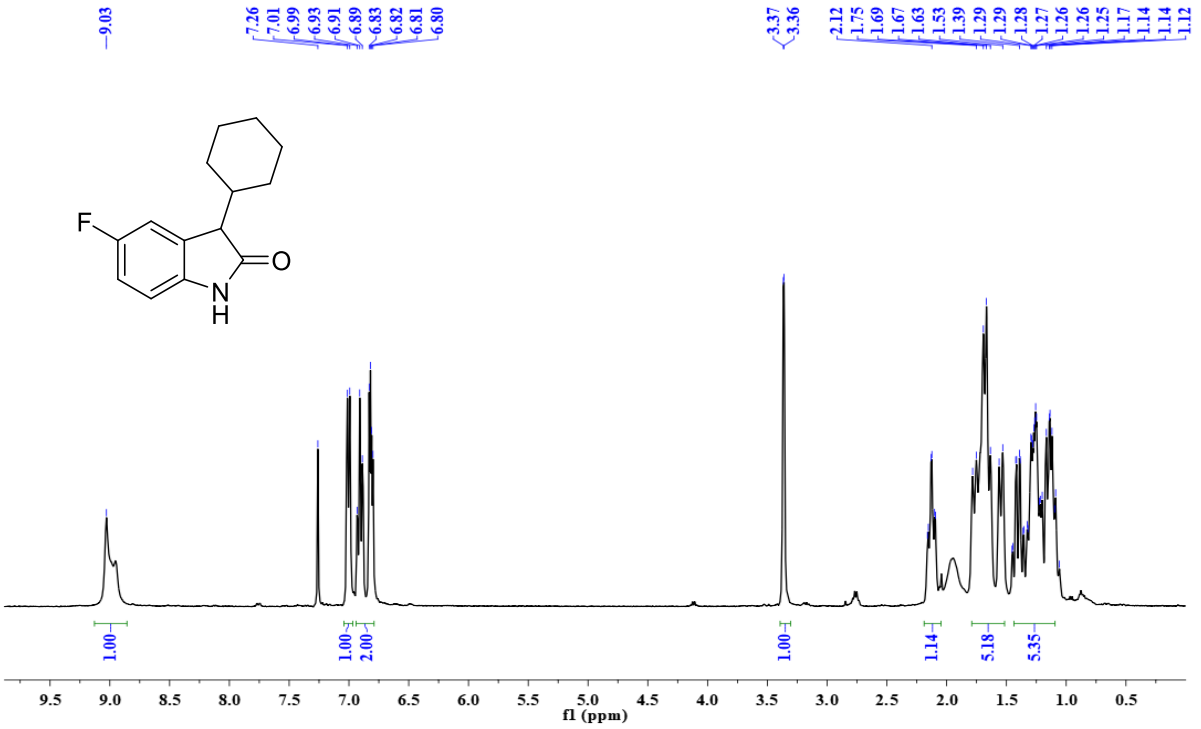


Figure S48. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cyclohexyl-5-fluoroindolin-2-one (**P24**).

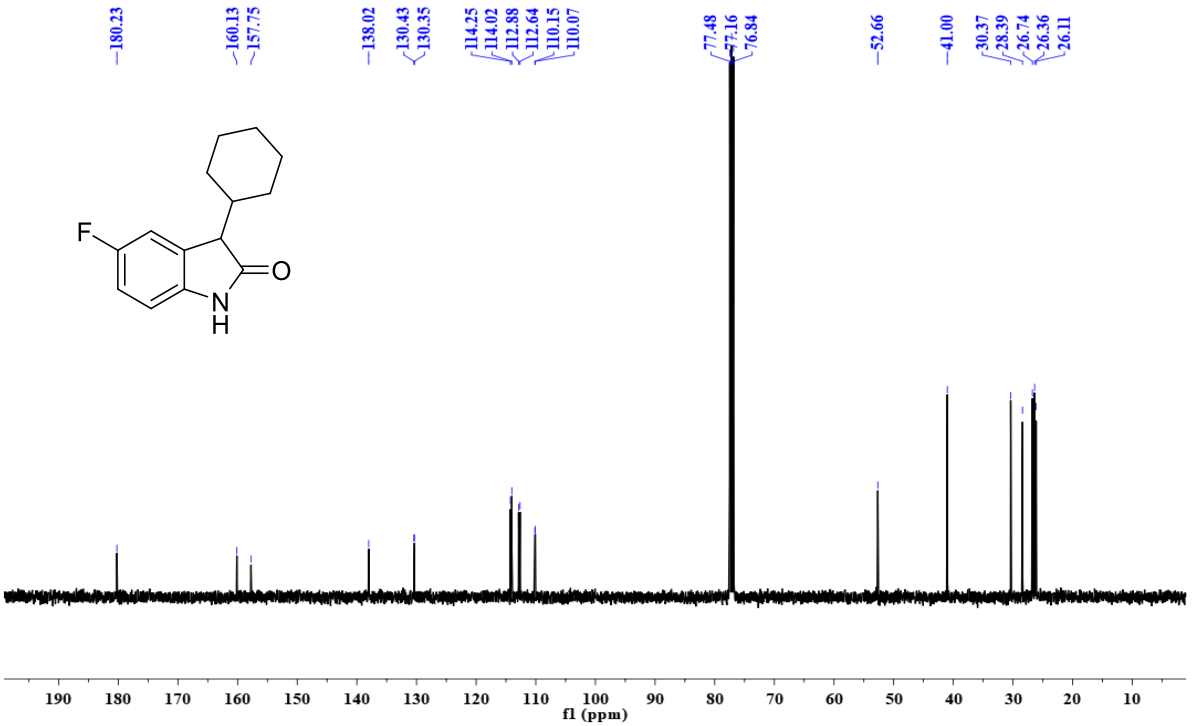


Figure S49. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-cyclohexyl-5-fluoroindolin-2-one (**P24**).

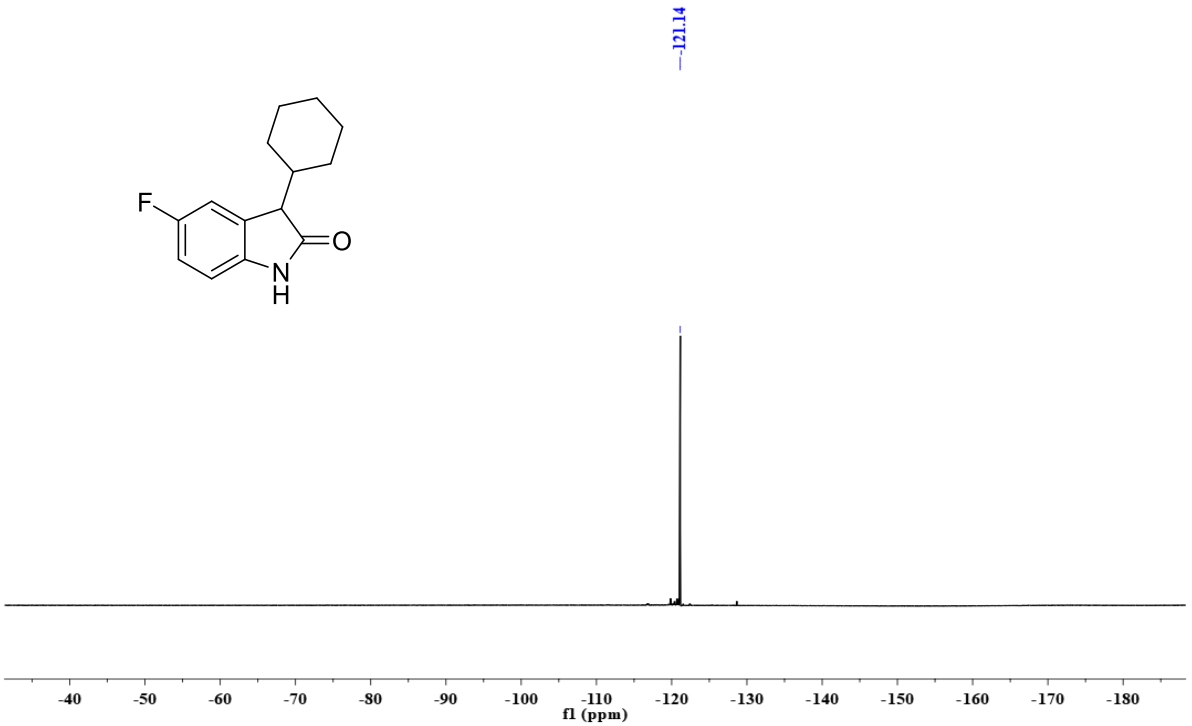


Figure S50. $^{19}F\{^{1}H\}$ NMR (377 MHz, $CDCl_3$) of 3-cyclohexyl-5-fluoroindolin-2-one (**P24**).

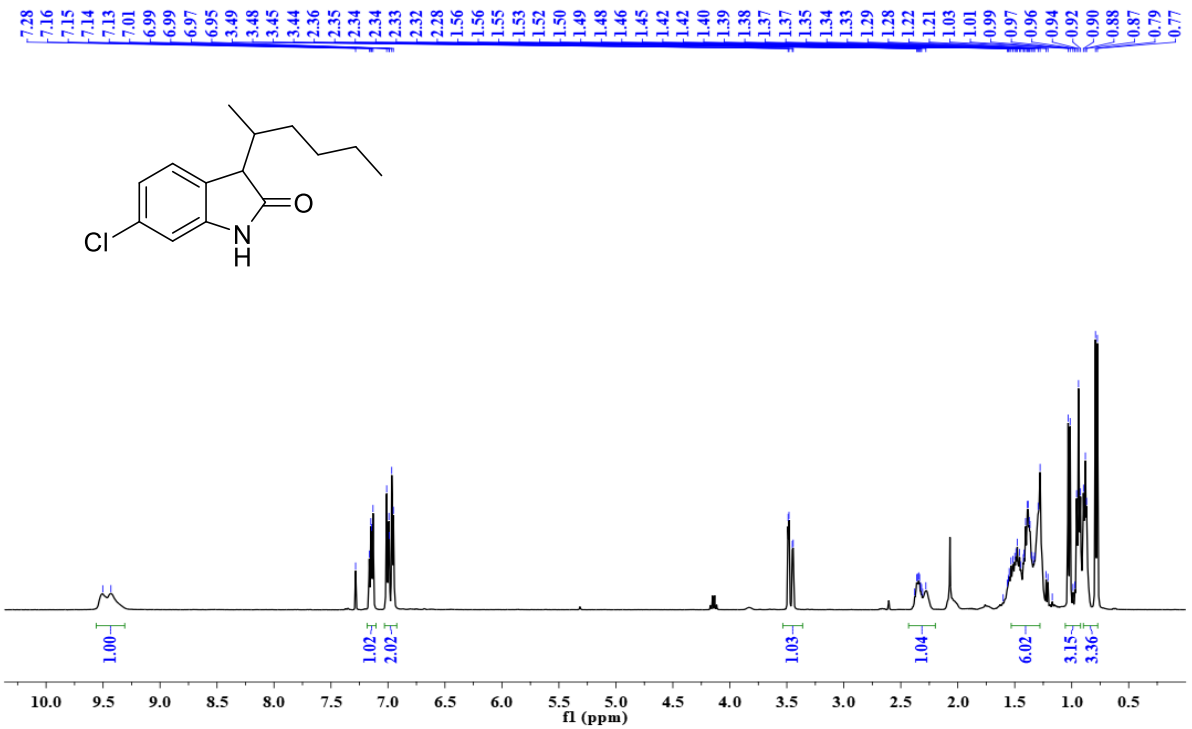


Figure S51. ^{1}H NMR (400 MHz, $CDCl_3$) of 6-chloro-3-(hexan-2-yl)indolin-2-one (**P25**).

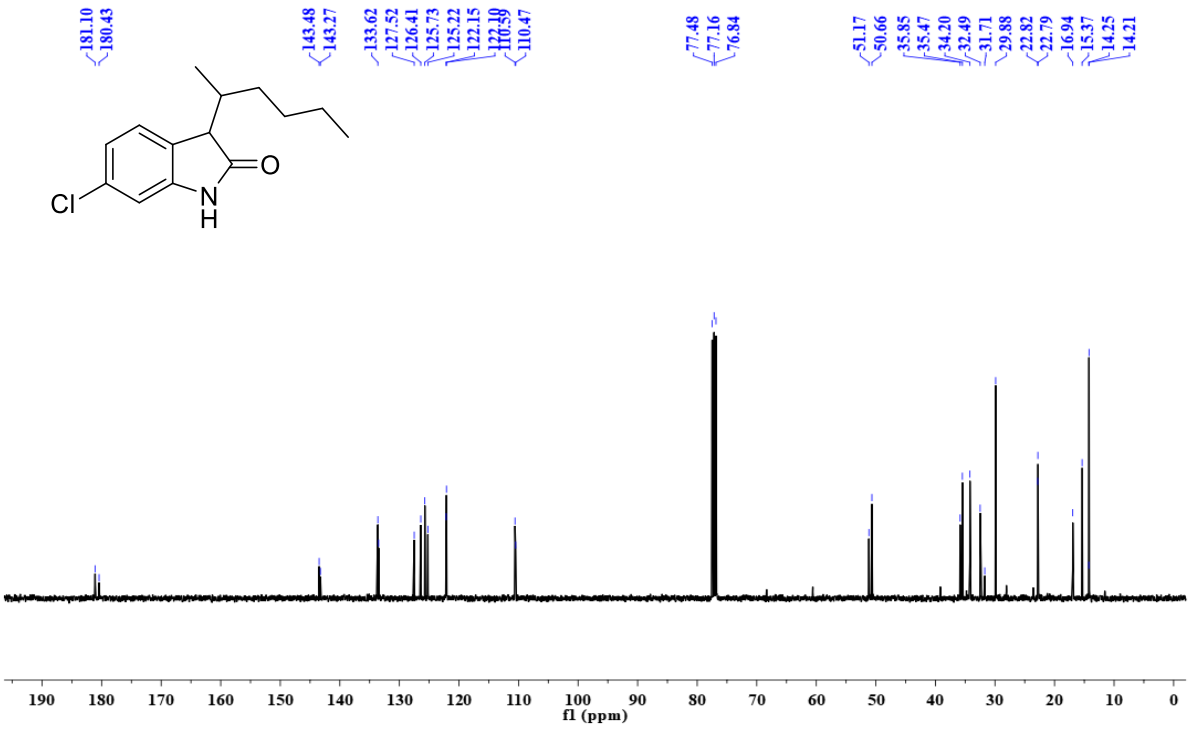


Figure S52. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) of 6-chloro-3-(hexan-2-yl)indolin-2-one (**P25**).

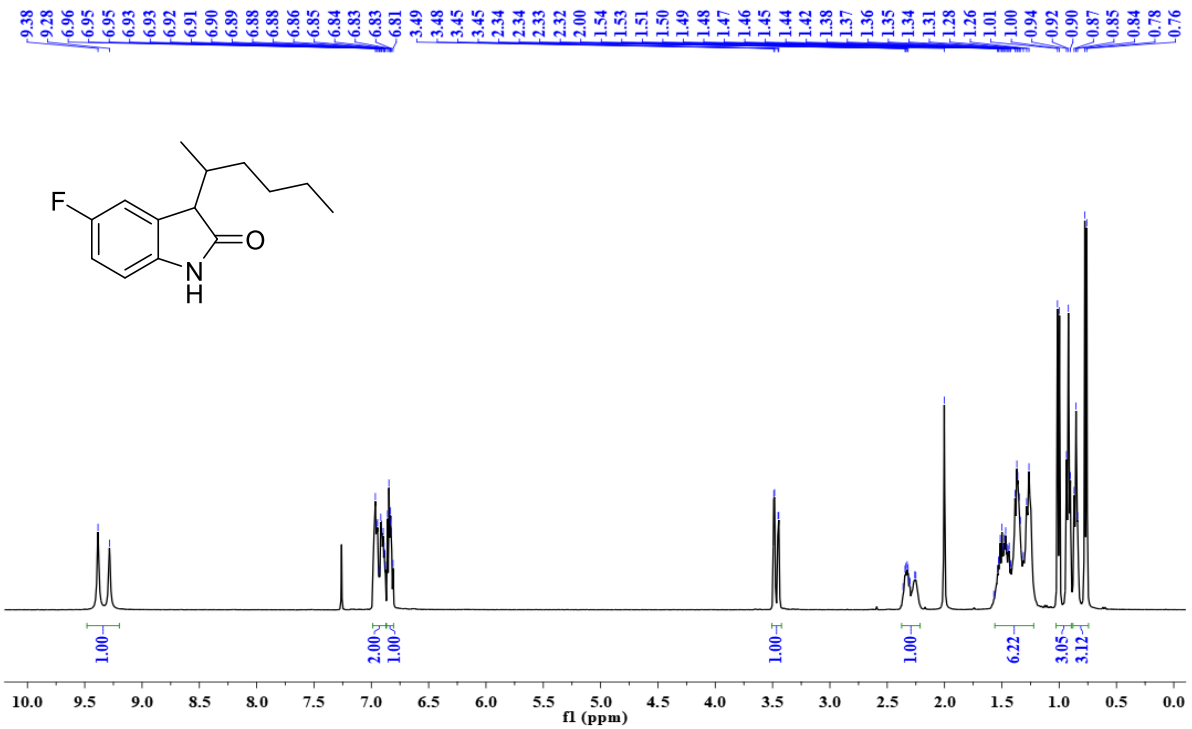


Figure S53. ^{1}H NMR (400 MHz, $CDCl_3$) of 5-fluoro-3-(hexan-2-yl)indolin-2-one (**P26**).

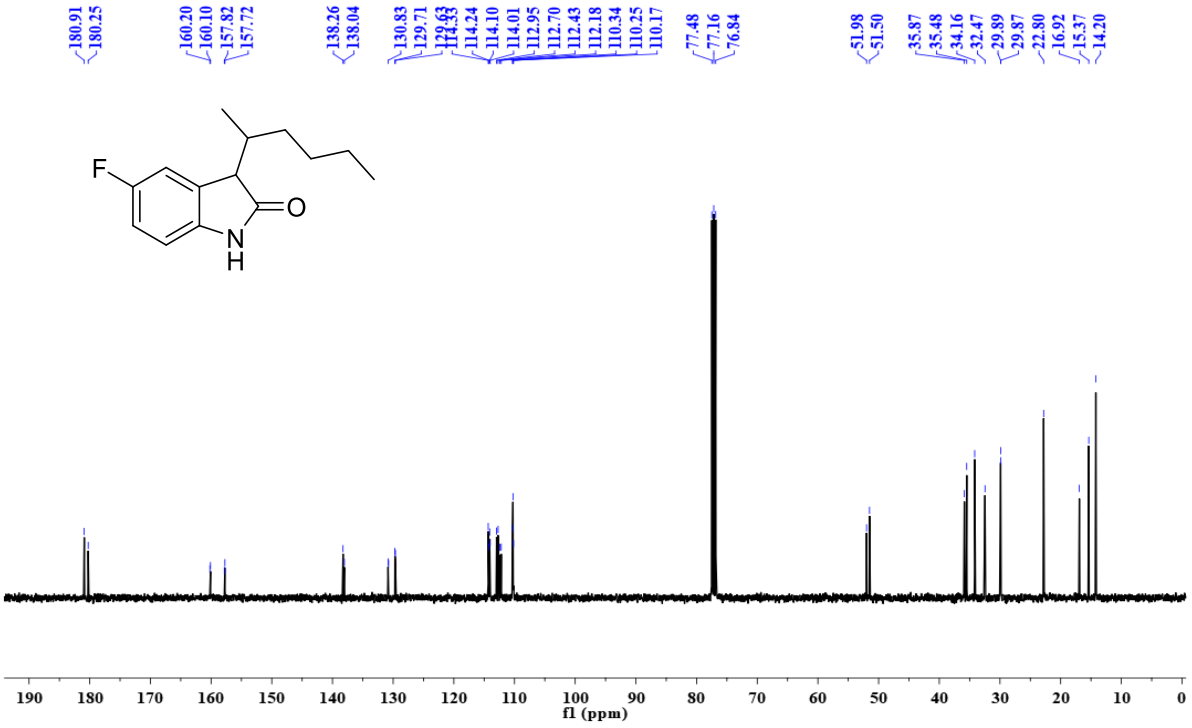


Figure S54. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) of 5-fluoro-3-(hexan-2-yl)indolin-2-one (**P26**).

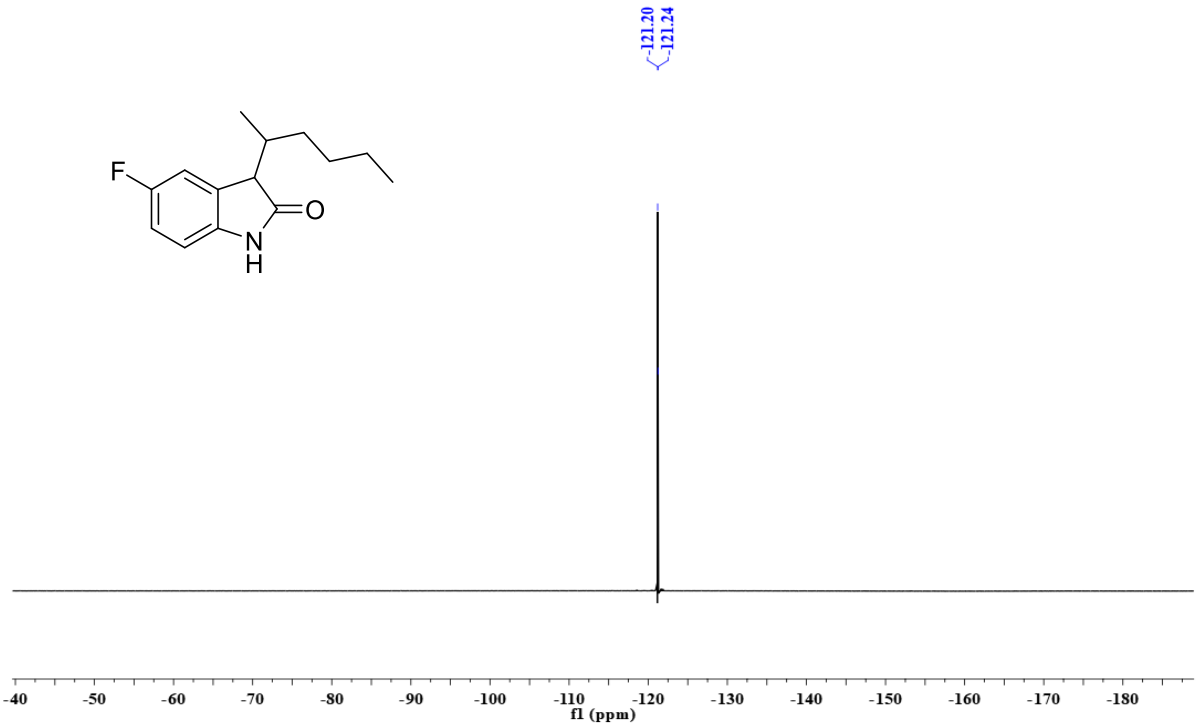


Figure S55. $^{19}F\{^{1}H\}$ NMR (377 MHz, $CDCl_3$) of 5-fluoro-3-(hexan-2-yl)indolin-2-one (**P$_{26}$**).

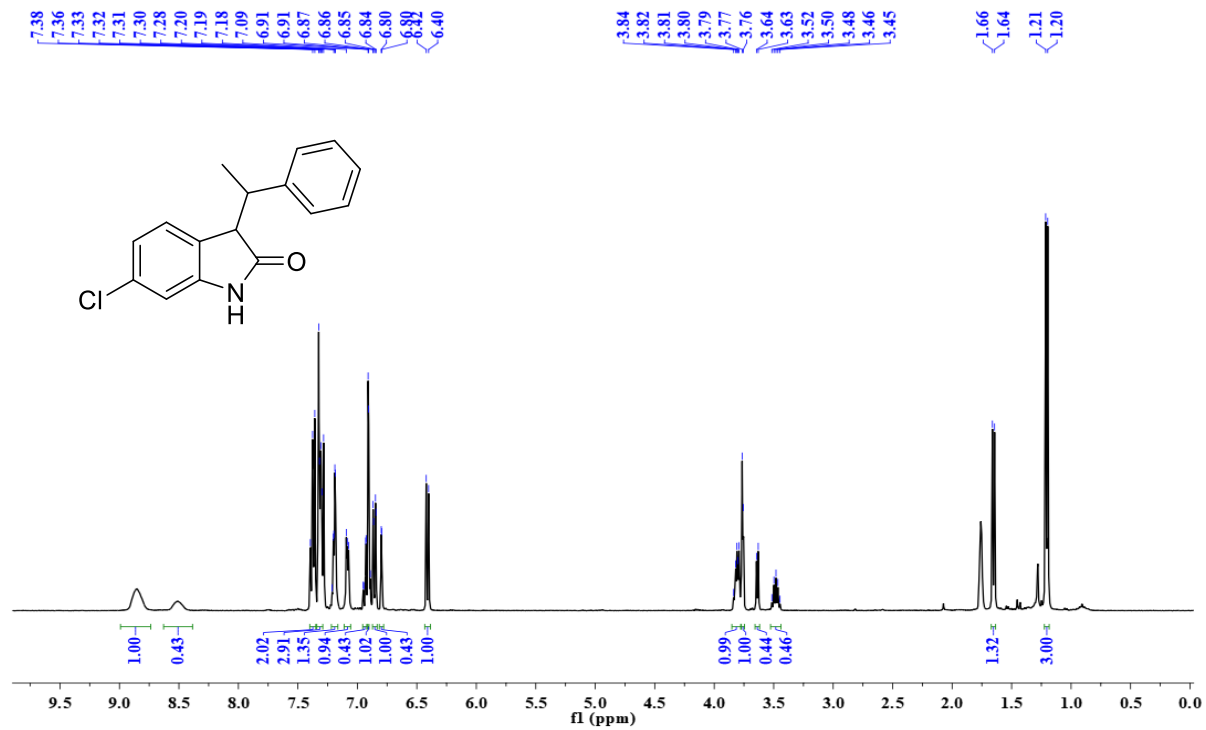


Figure S56. ^{1}H NMR (400 MHz, $CDCl_3$) of 6-chloro-3-(1-phenylethyl)indolin-2-one (**P$_{27}$**).

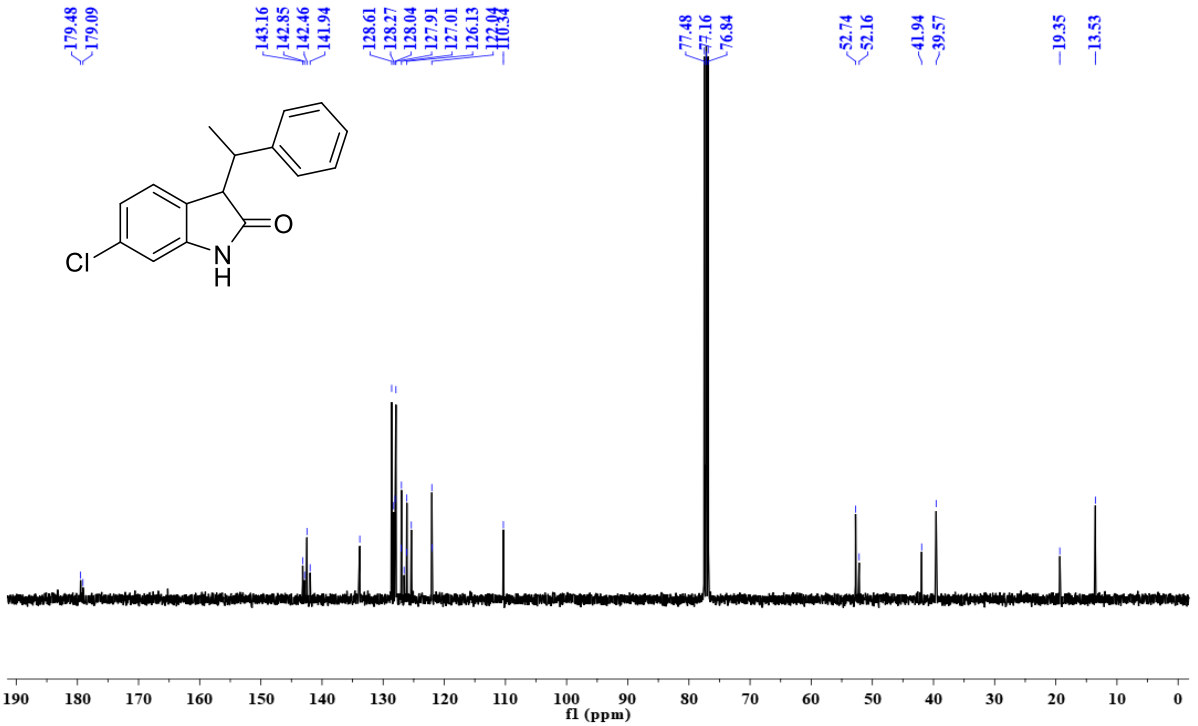


Figure S57. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 6-chloro-3-(1-phenylethyl)indolin-2-one (**P27**).

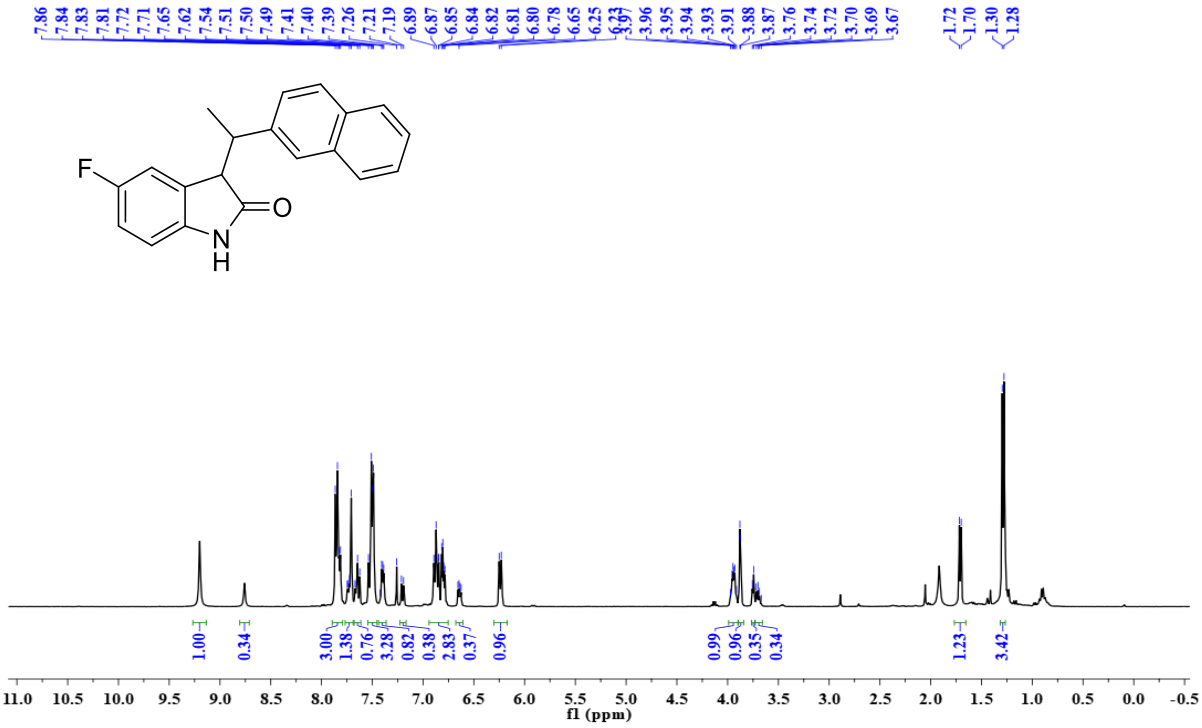


Figure S58. ^{1}H NMR (400 MHz, $CDCl_3$) of 5-fluoro-3-(1-(naphthalen-2-yl)ethyl)indolin-2-one (**P28**).

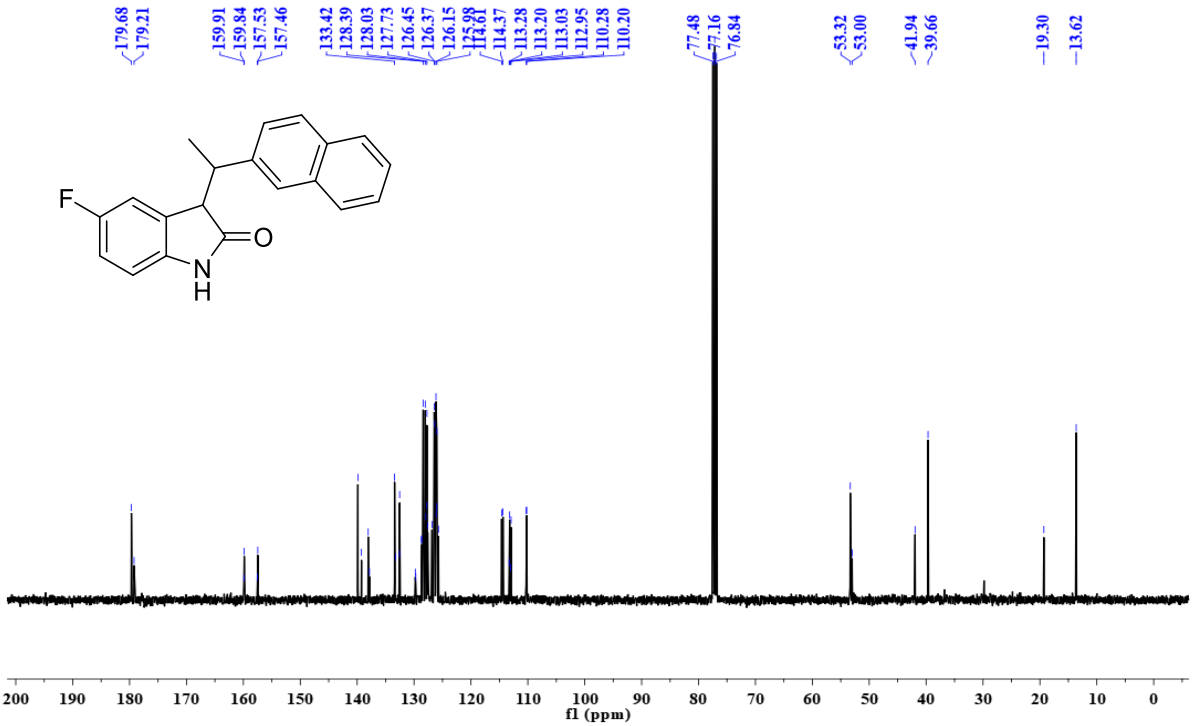


Figure S59. ^{1}H NMR (400 MHz, $CDCl_3$) of 5-fluoro-3-(1-(naphthalen-2-yl)ethyl)indolin-2-one (**P28**).

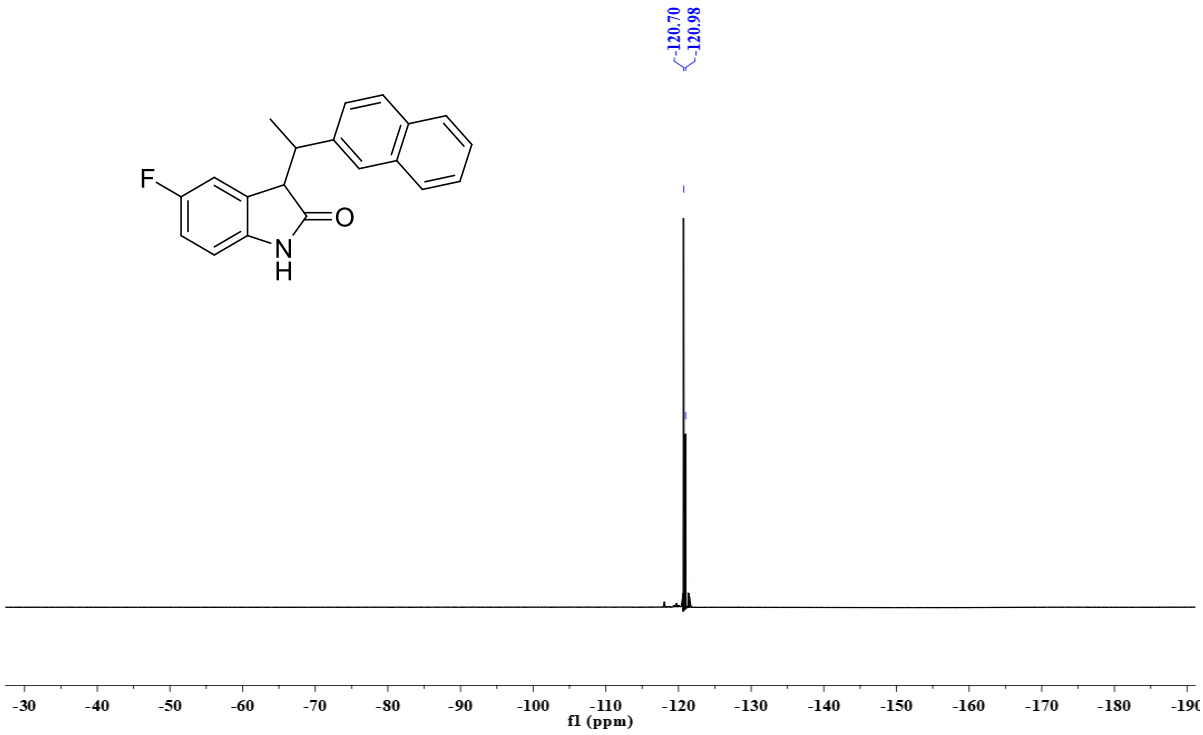


Figure S60. $^{19}F\{^{1}H\}$ NMR (377 MHz, $CDCl_3$) of 5-fluoro-3-(1-(naphthalen-2-yl)ethyl)indolin-2-one (**P28**).

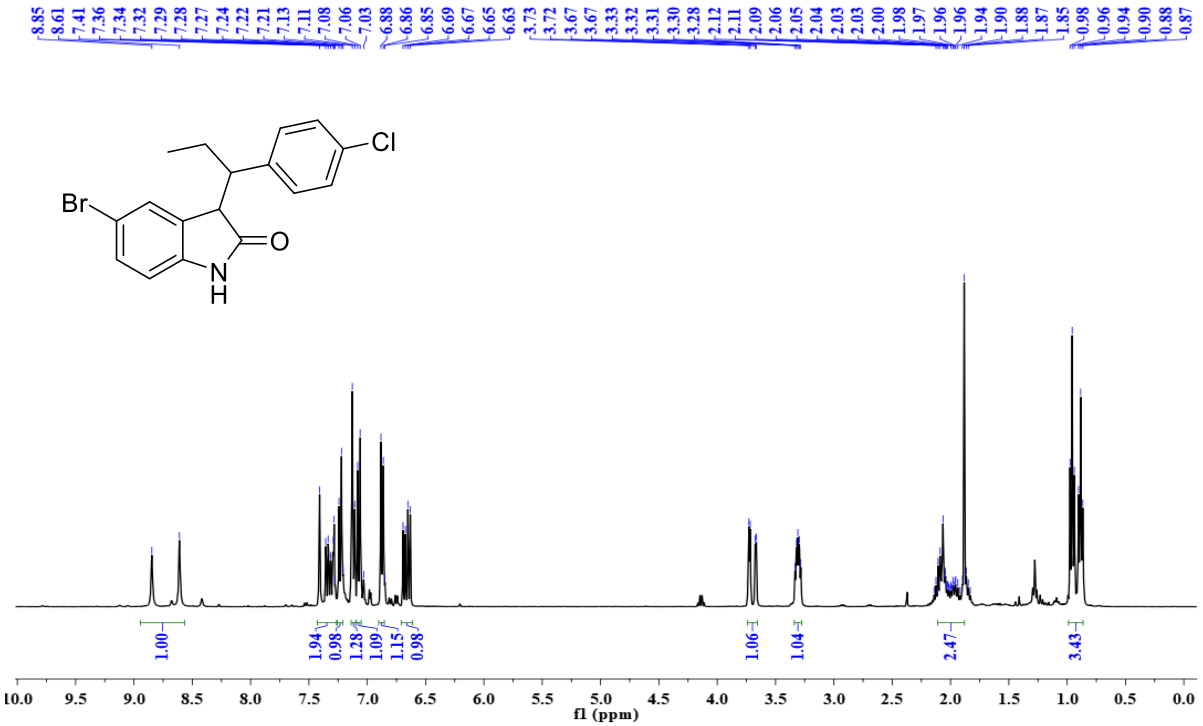


Figure S61. ^{1}H NMR (400 MHz, $CDCl_3$) of 5-bromo-3-(1-(4-chlorophenyl)propyl)indolin-2-one (**P29**).

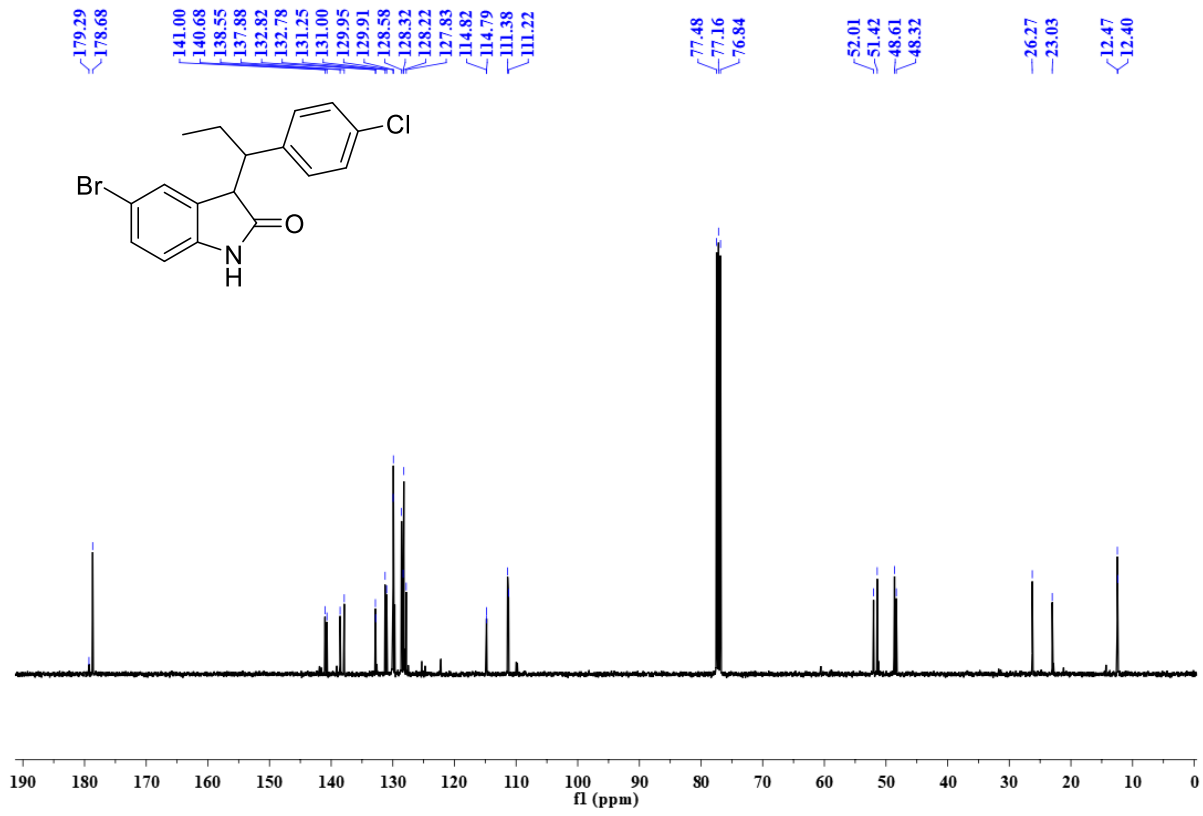


Figure S62. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 5-bromo-3-(1-(4-chlorophenyl)propyl)indolin-2-one (**P29**).

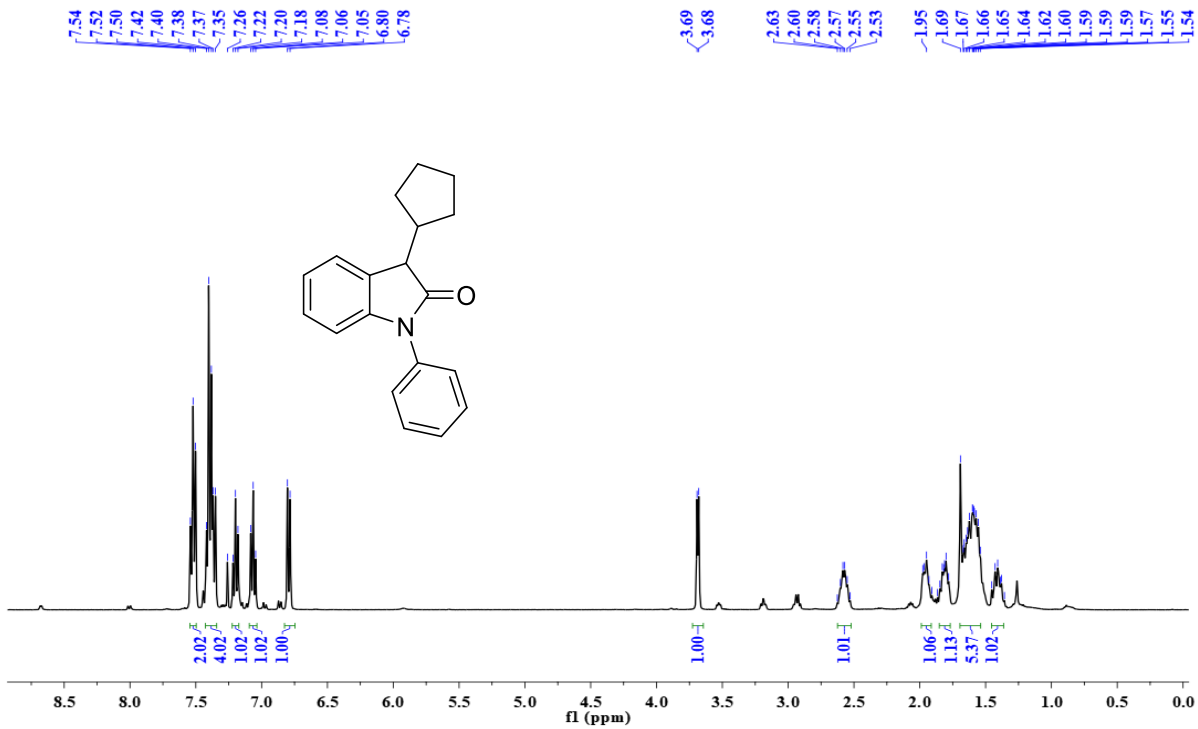


Figure S63. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cyclopentyl-1-phenylindolin-2-one (**P30**).

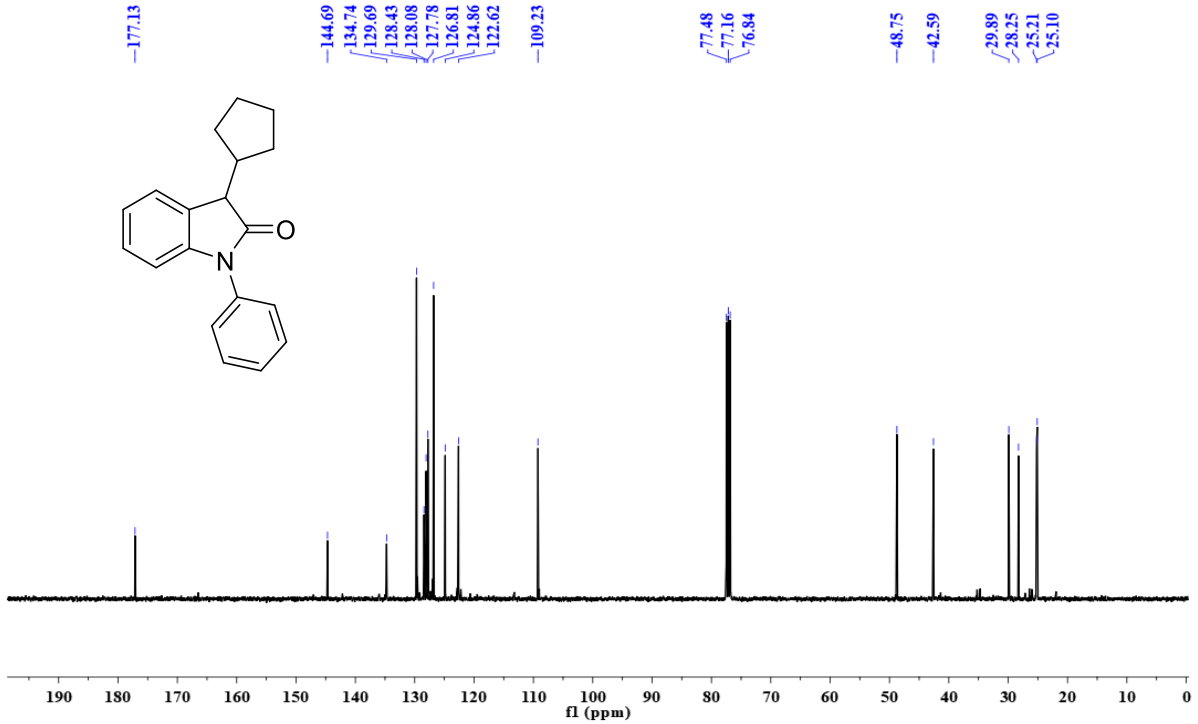


Figure S64. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) of 3-cyclopentyl-1-phenylindolin-2-one (**P30**).

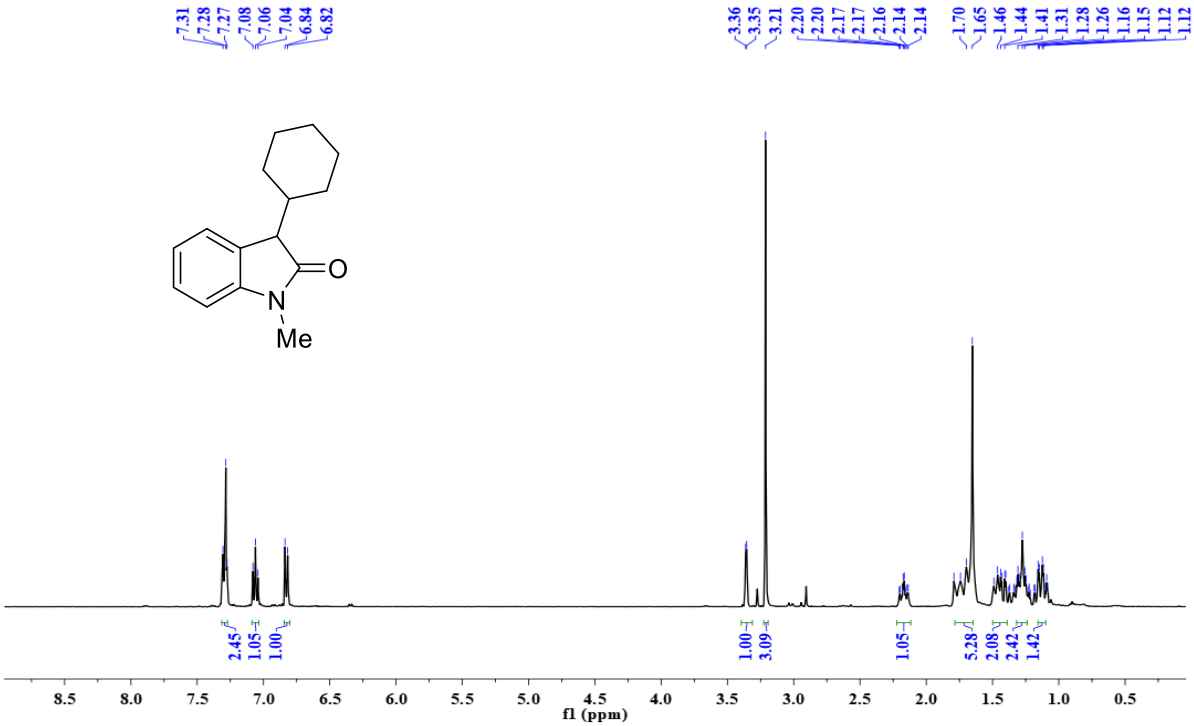


Figure S65. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cyclohexyl-1-methylindolin-2-one (**P31**).

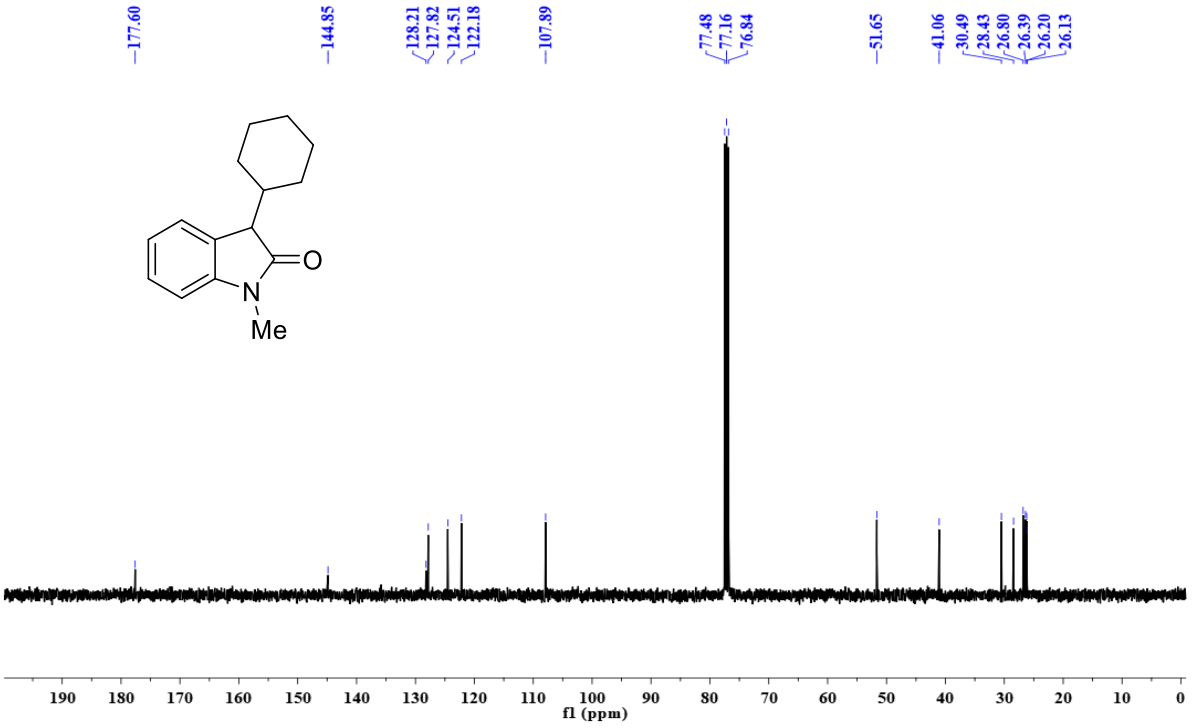


Figure S66. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-cyclohexyl-1-methylindolin-2-one (**P31**).

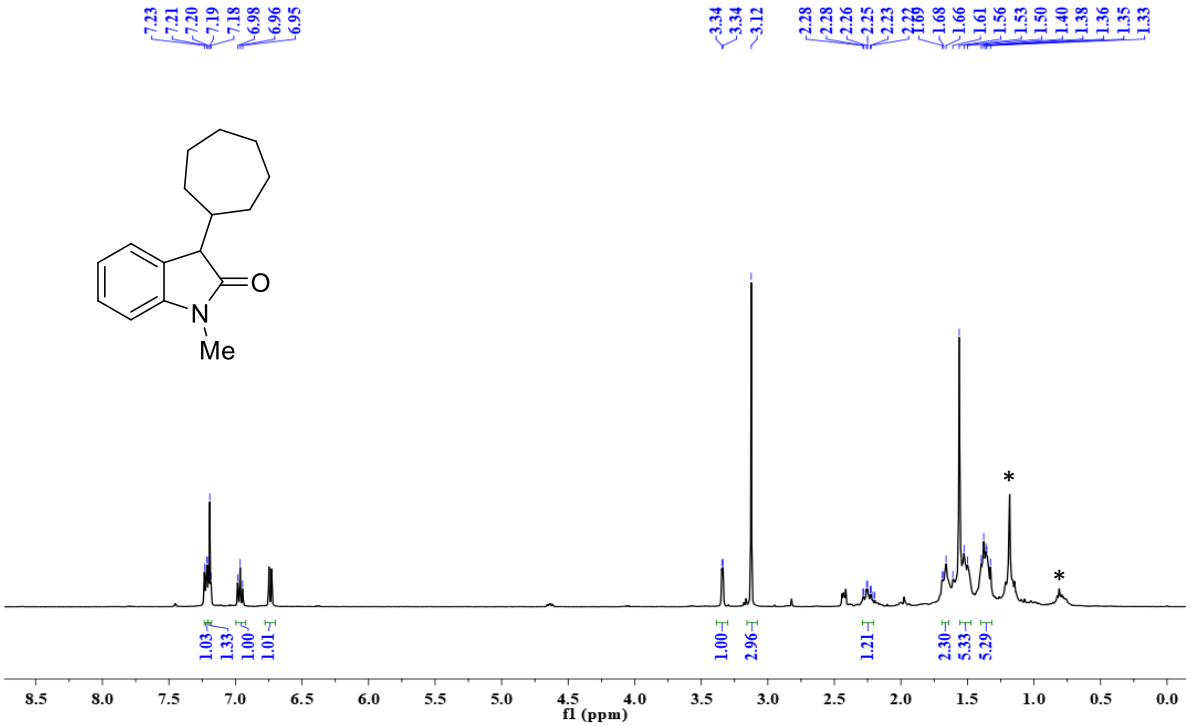


Figure S67. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cycloheptyl-1-methylindolin-2-one (**P32**) ('*' is H-grease).

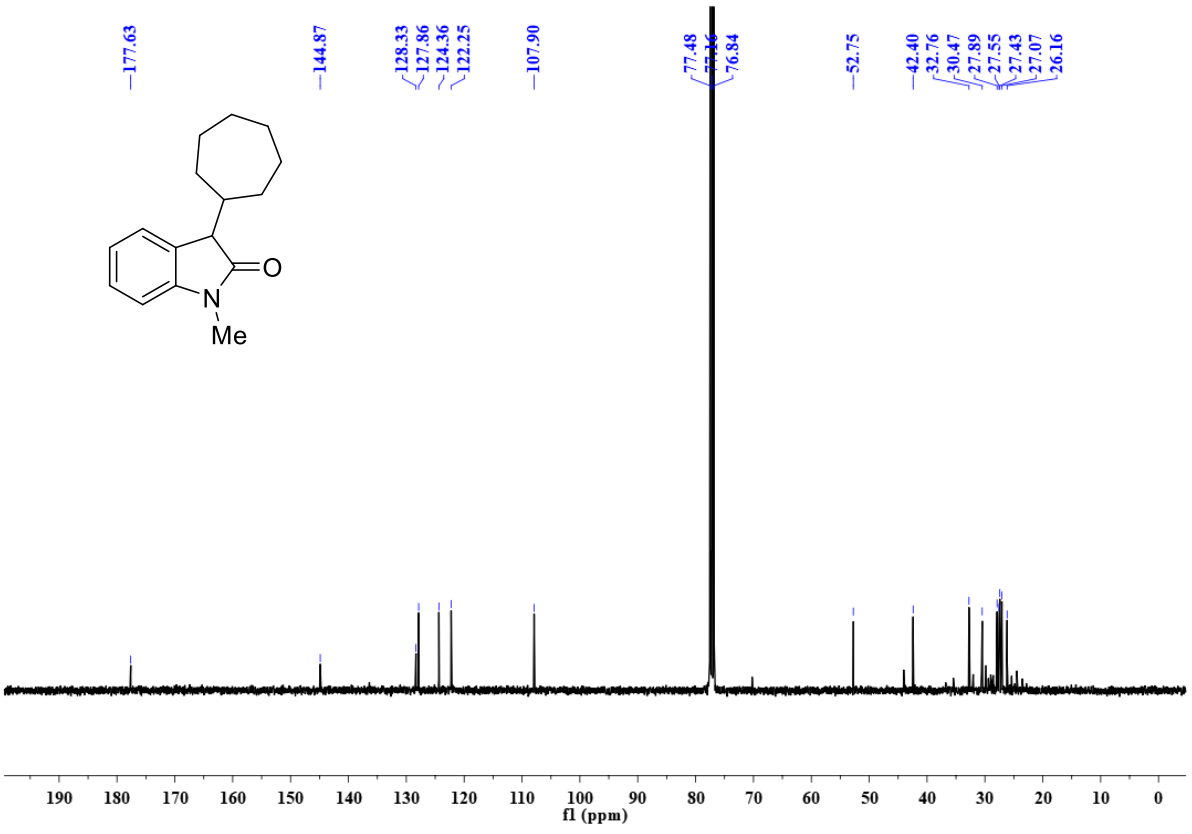


Figure S68. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-cycloheptyl-1-methylindolin-2-one (**P32**).

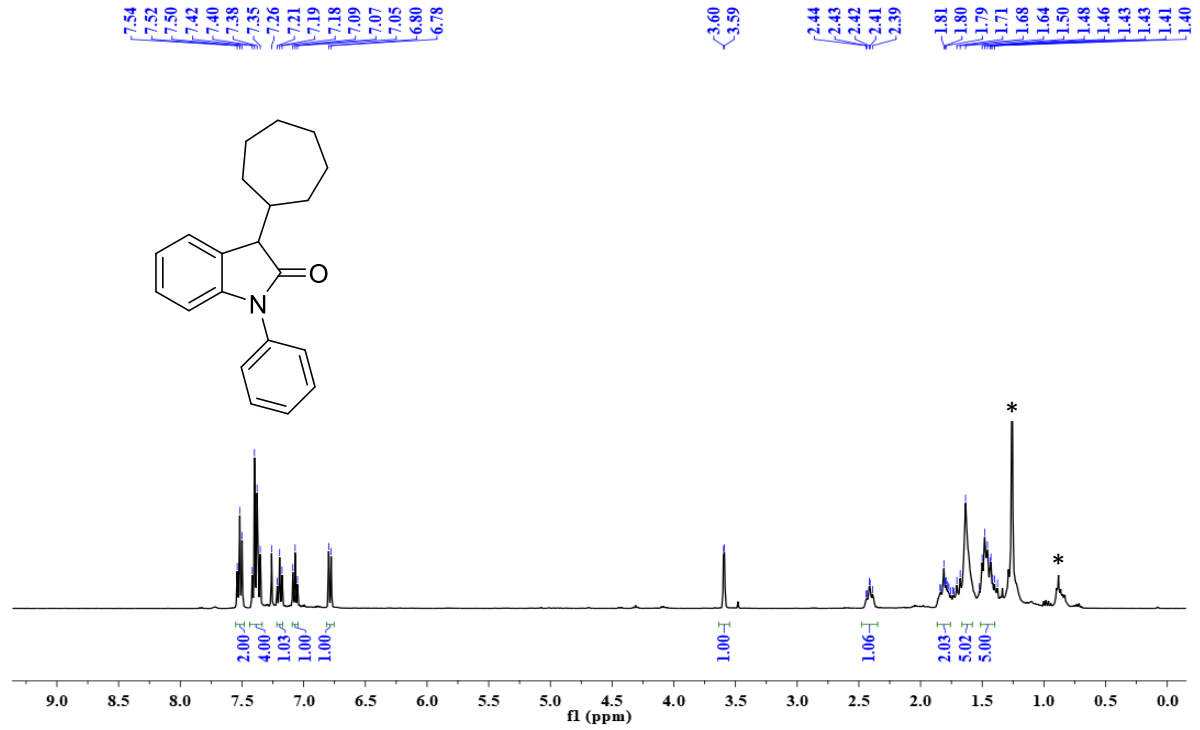


Figure S69. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cycloheptyl-1-phenylindolin-2-one (**P33**) ('*' is H-grease).

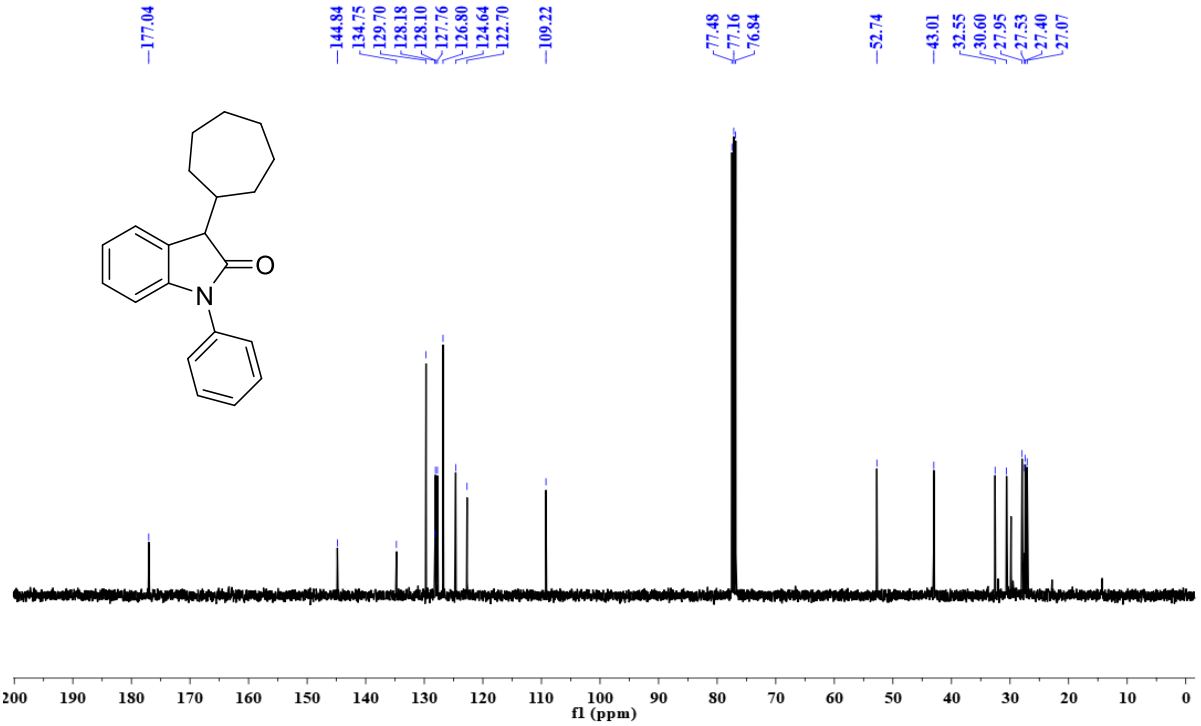


Figure S70. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-cycloheptyl-1-phenylindolin-2-one (**P33**).

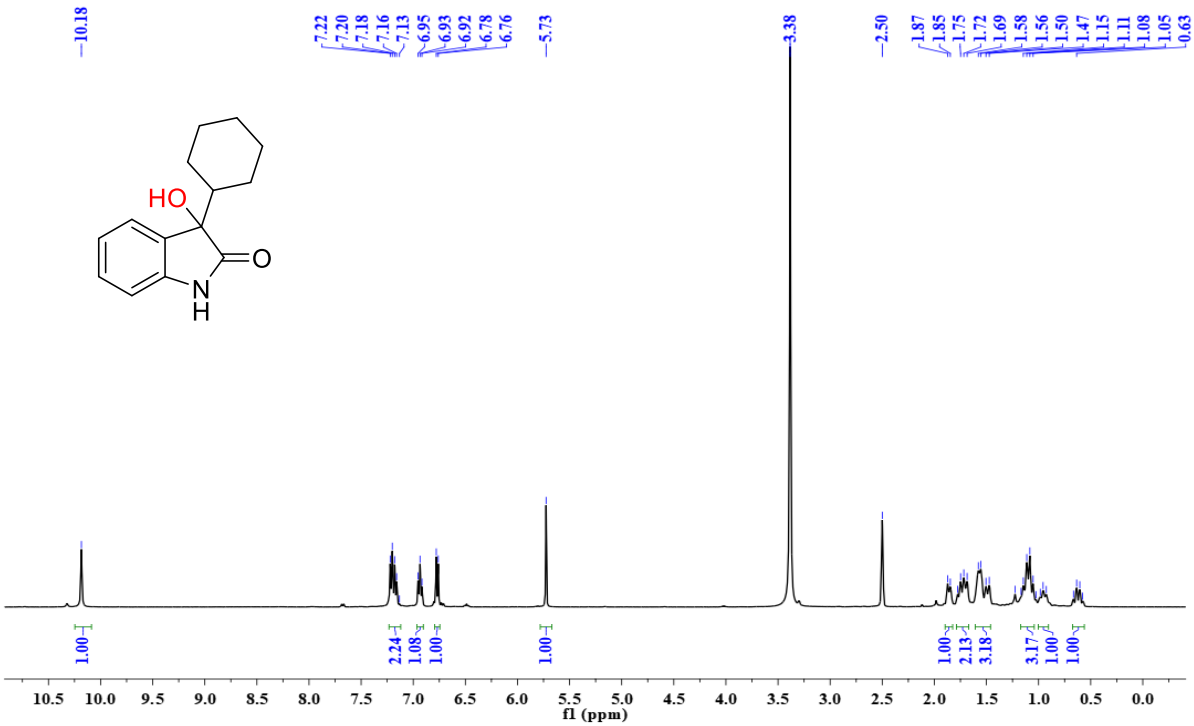


Figure S71. ^{1}H NMR (400 MHz, DMSO-d_6) of 3-cyclohexyl-3-hydroxyindolin-2-one (**P34**).

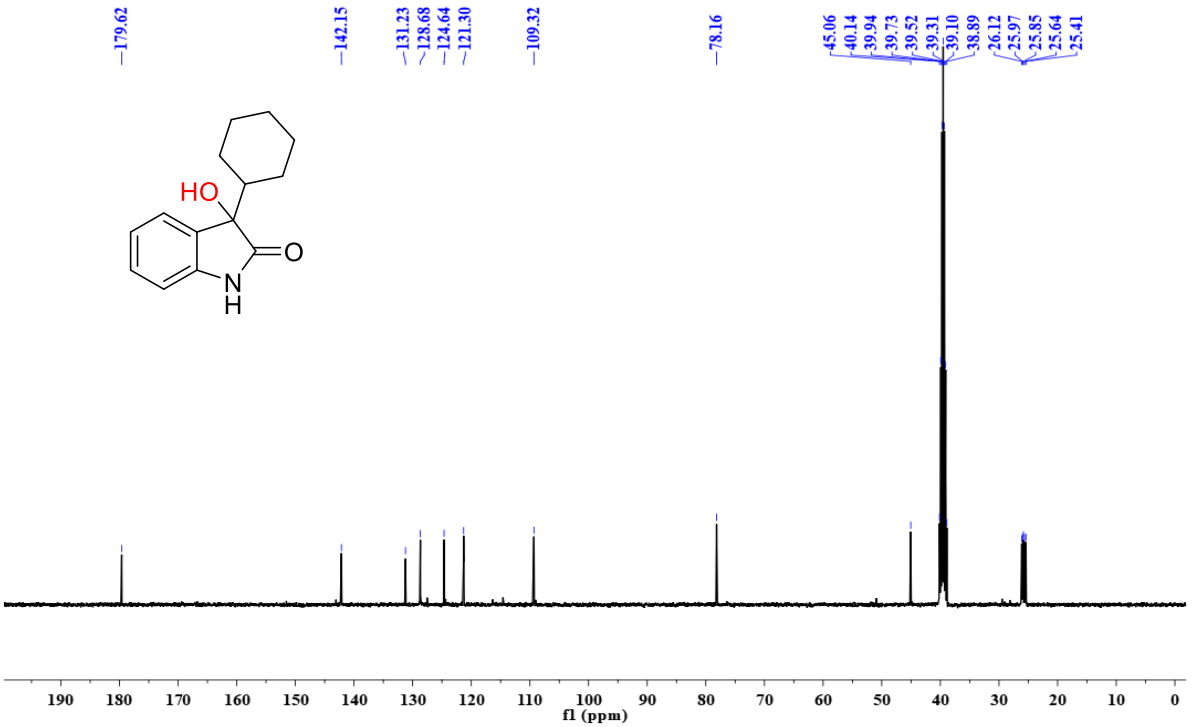


Figure S72. ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) of 3-cyclohexyl-3-hydroxyindolin-2-one (**P34**).

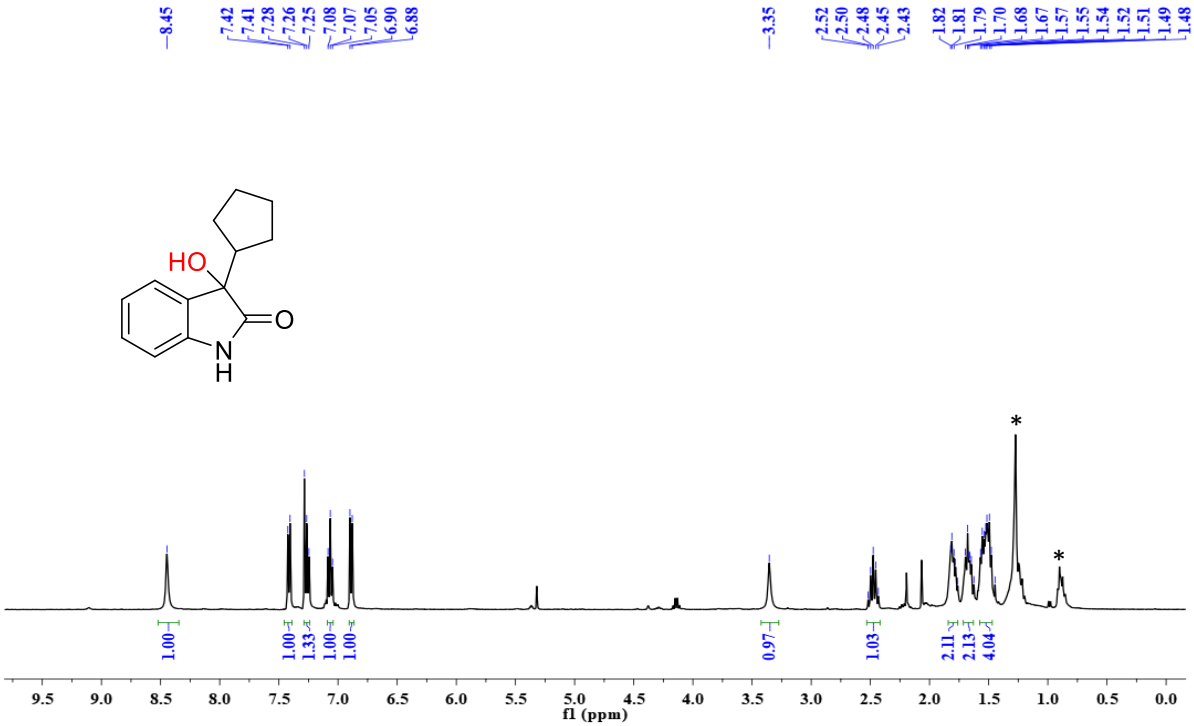


Figure S73. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-cyclopentyl-3-hydroxyindolin-2-one (**P35**) ('*' is H-grease).

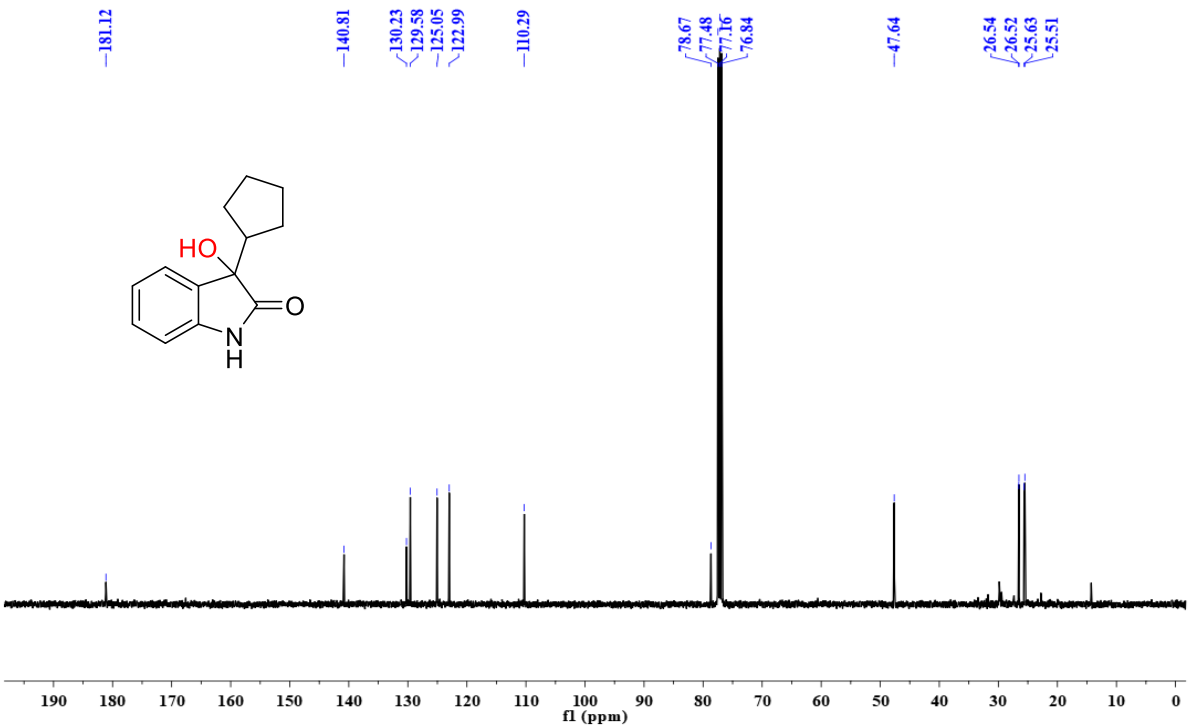


Figure S74. ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) of 3-cyclopentyl-3-hydroxyindolin-2-one (**P35**).

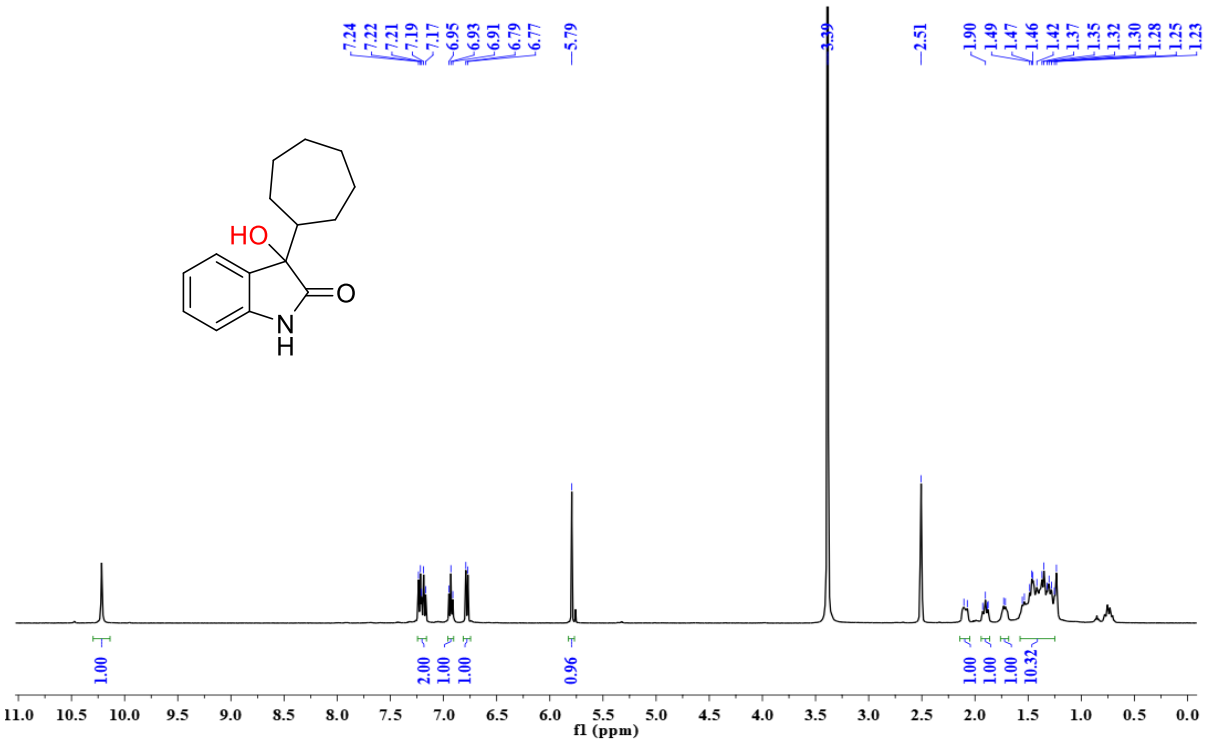


Figure S75. ^{1}H NMR (400 MHz, DMSO-d_6) of 3-cycloheptyl-3-hydroxyindolin-2-one (**P36**).

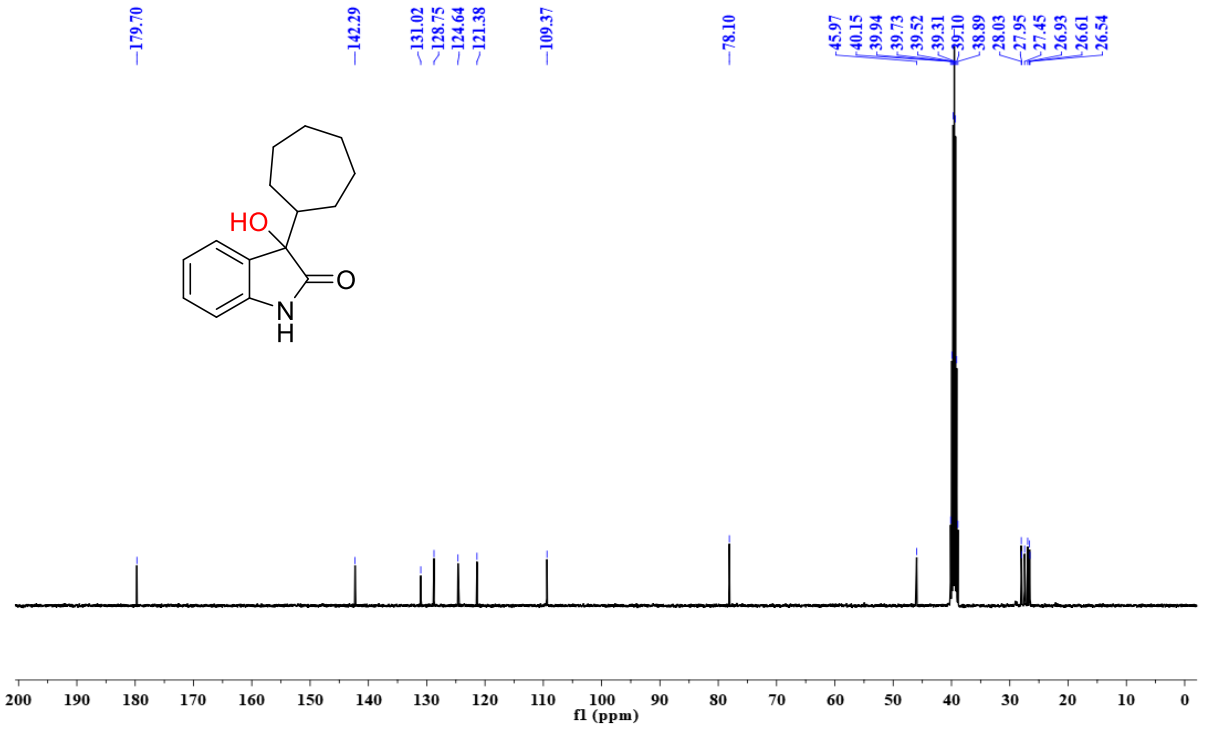


Figure S76. ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) of 3-cycloheptyl-3-hydroxyindolin-2-one (**P36**).

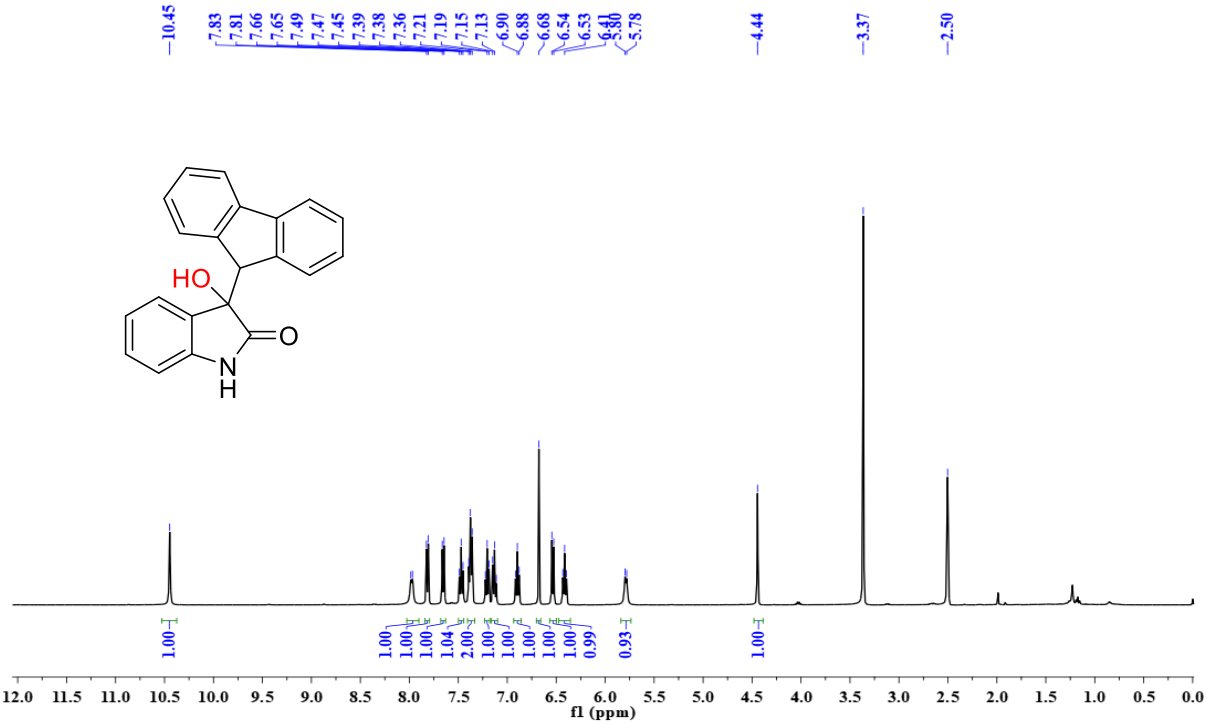


Figure S77. ^{1}H NMR (400 MHz, DMSO-d_6) of 3-(9H-fluoren-9-yl)-3-hydroxyindolin-2-one (**P37**).

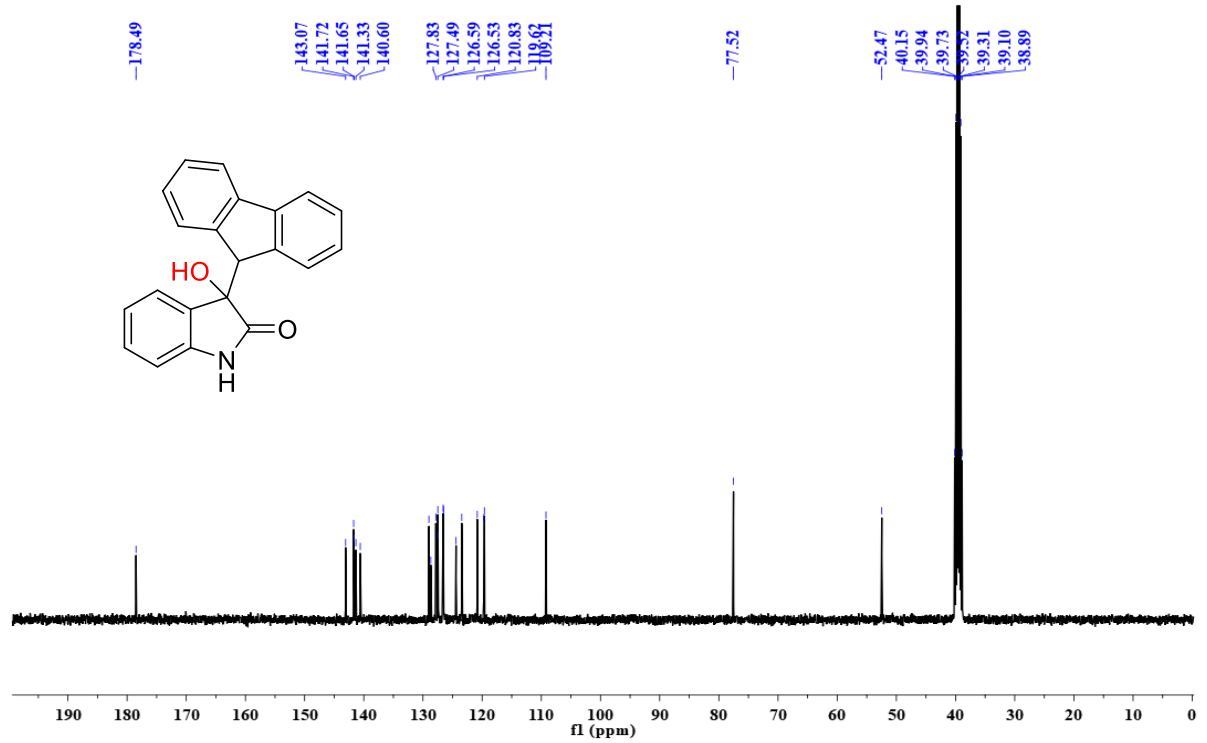


Figure S78. ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) of 3-(9H-fluoren-9-yl)-3-hydroxyindolin-2-one (**P37**).

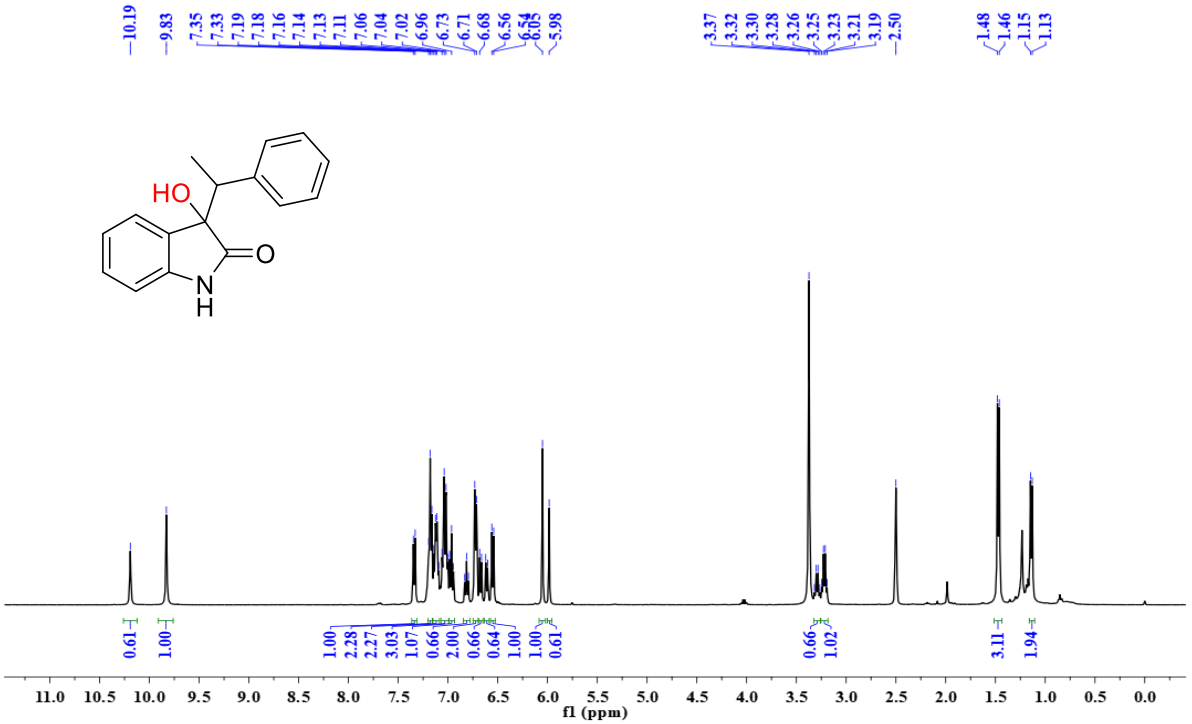


Figure S79. ^{1}H NMR (400 MHz, DMSO-d_6) of 3-hydroxy-3-(1-phenylethyl)indolin-2-one (**P38**).

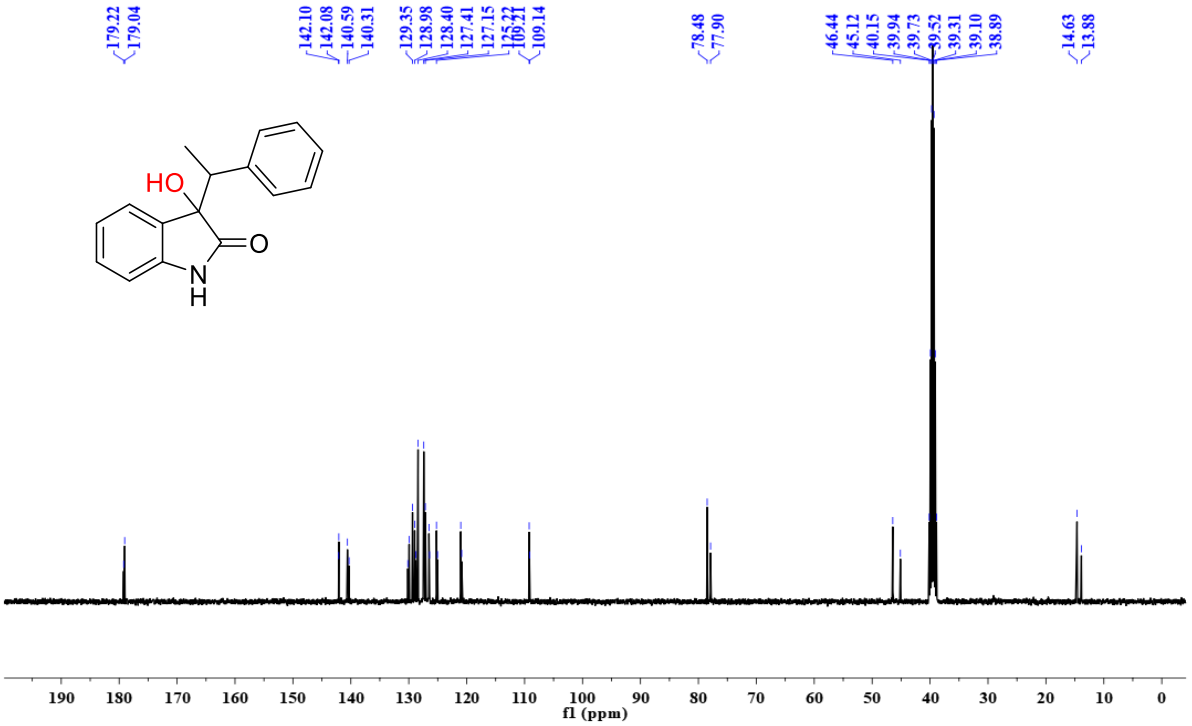


Figure S80. ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) of 3-hydroxy-3-(1-phenylethyl)indolin-2-one (**P38**).

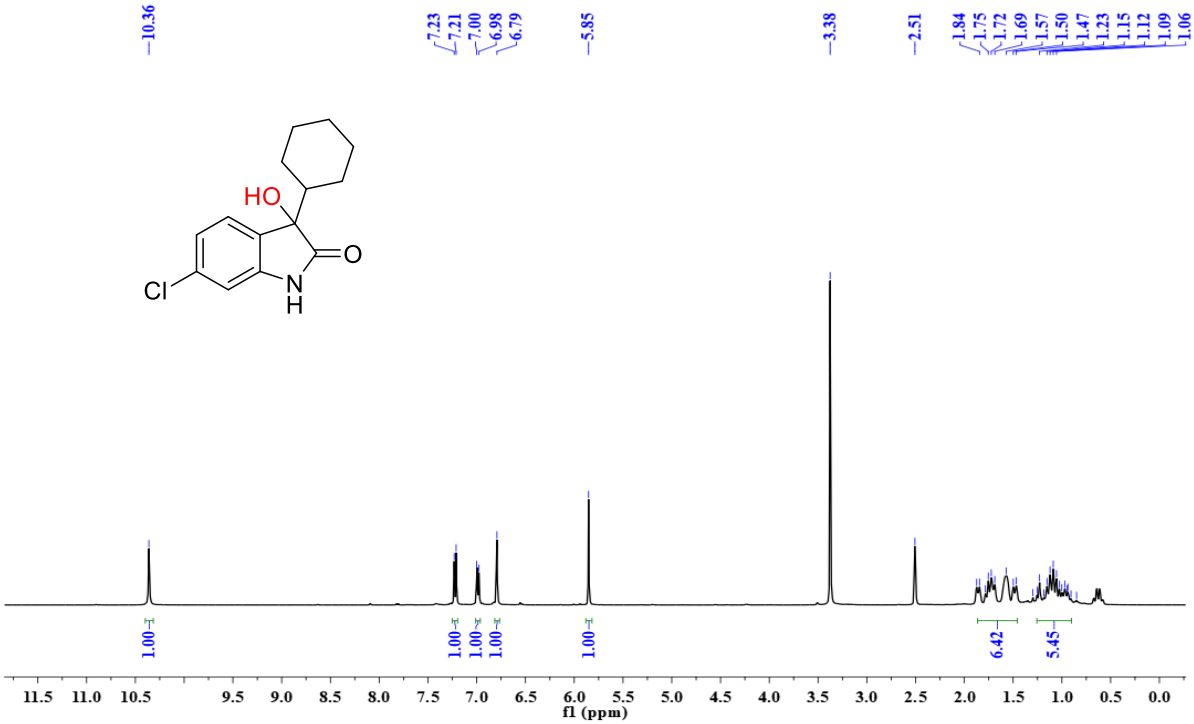


Figure S81. ^{1}H NMR (400 MHz, DMSO-d_6) of 6-chloro-3-cyclohexyl-3-hydroxyindolin-2-one (**P39**).

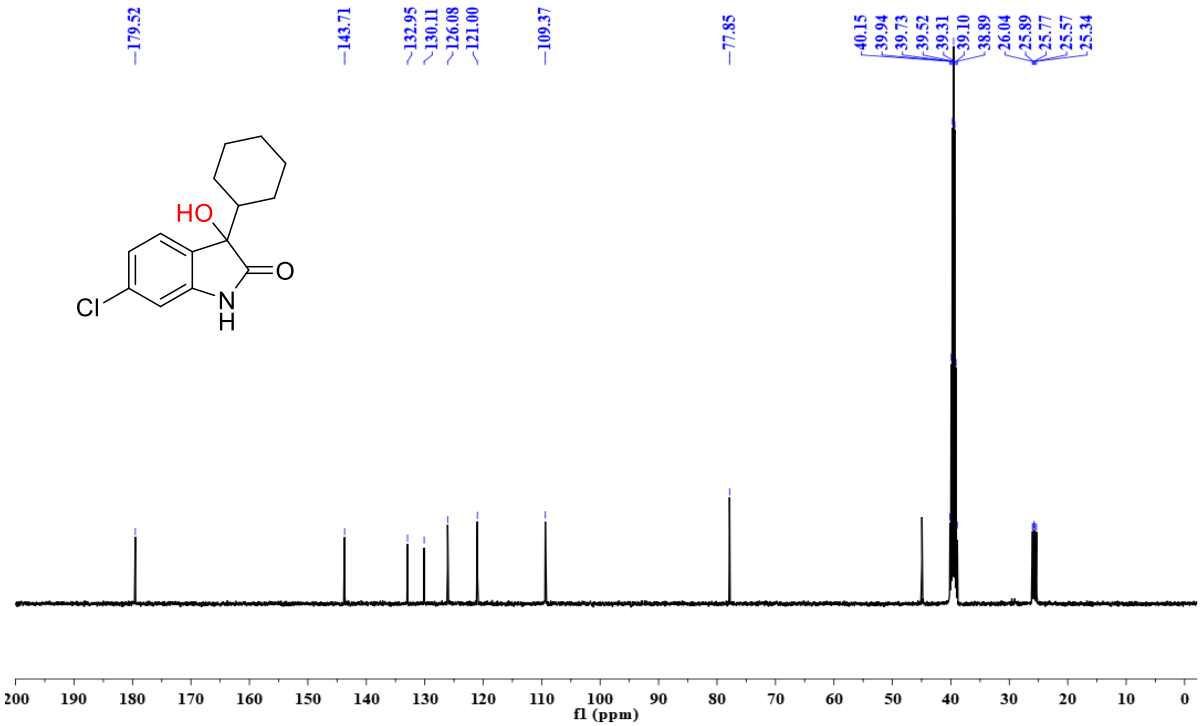


Figure S82. ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) of 6-chloro-3-cyclohexyl-3-hydroxyindolin-2-one (**P39**).

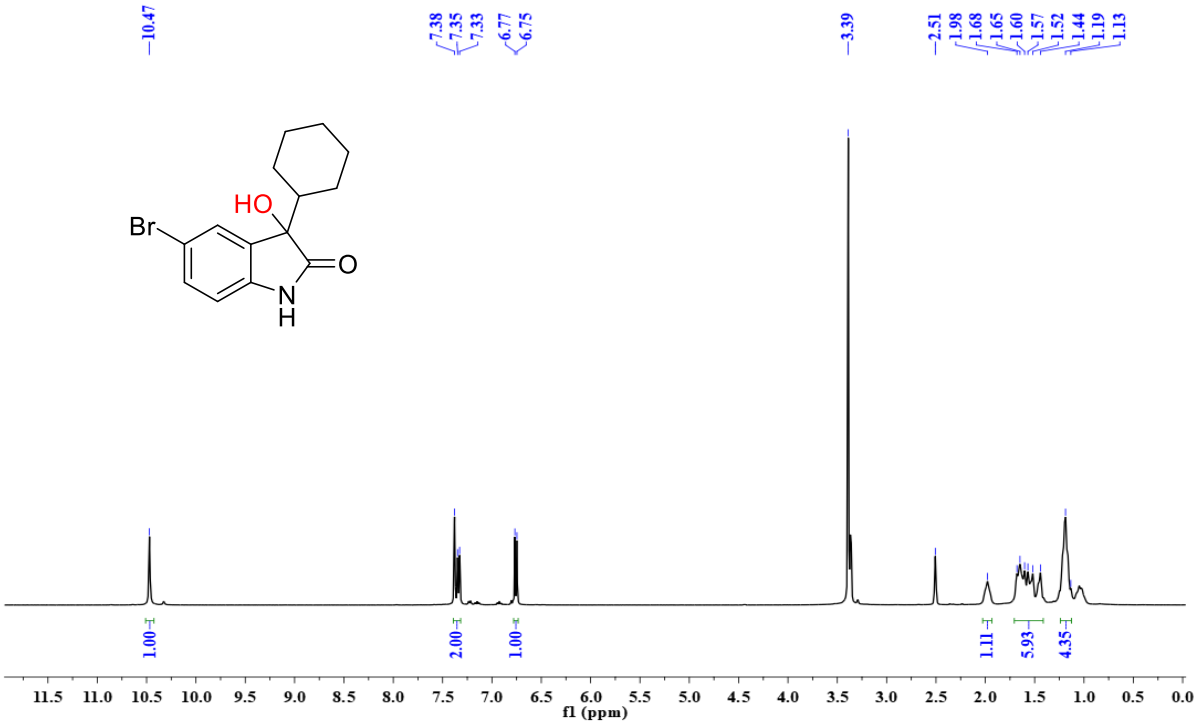


Figure S83. ^{1}H NMR (400 MHz, DMSO-d_6) of 5-bromo-3-cyclohexyl-3-hydroxyindolin-2-one (**P$_{40}$**).

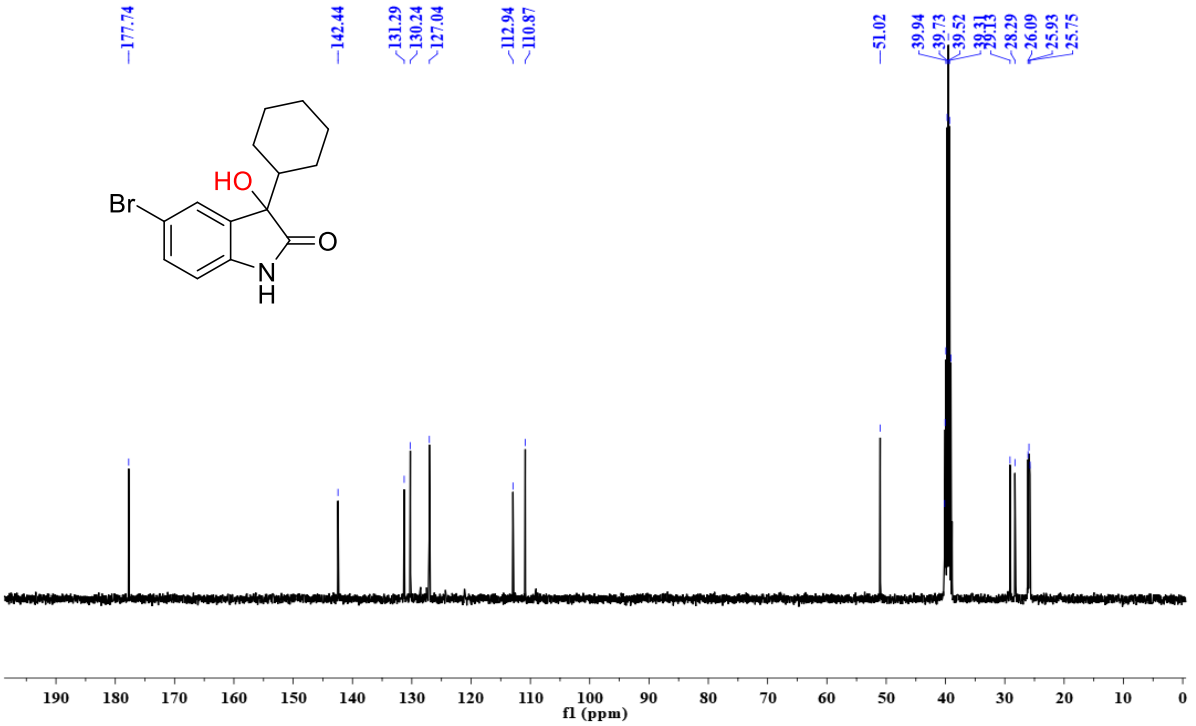


Figure S84. ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) of 5-bromo-3-cyclohexyl-3-hydroxyindolin-2-one (**P$_{40}$**).

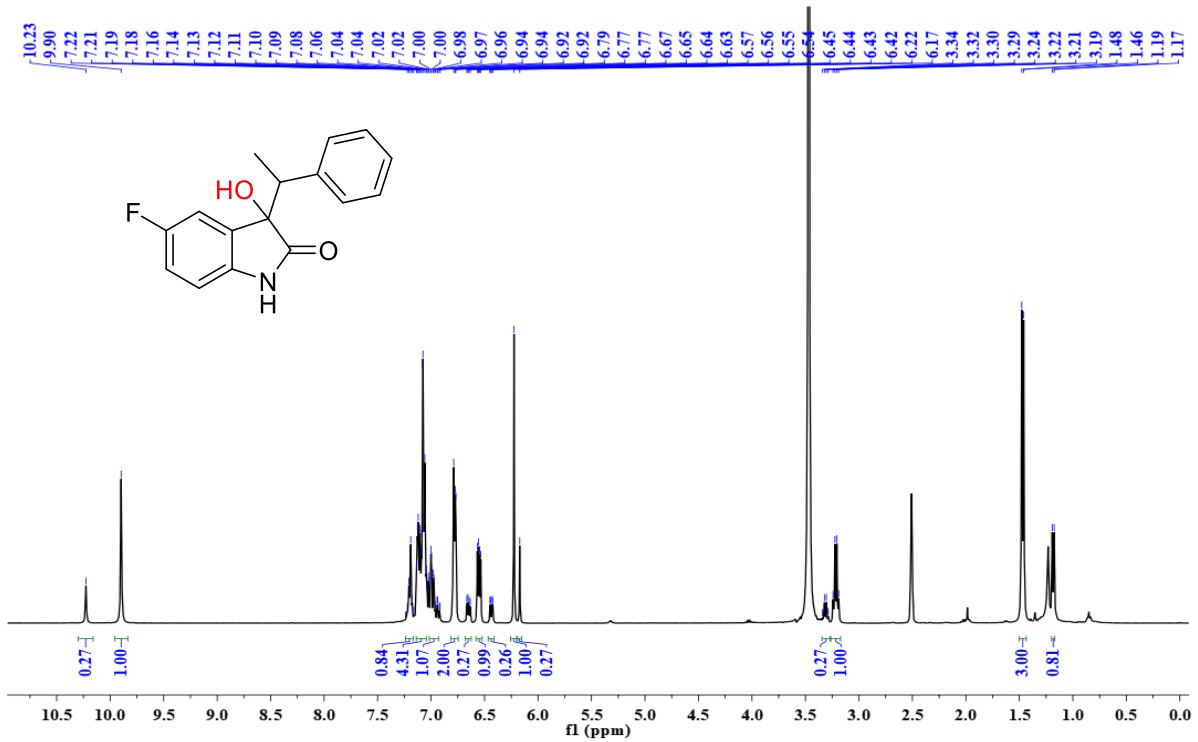


Figure S85. ^{1}H NMR (400 MHz, DMSO-d_6) of 5-fluoro-3-hydroxy-3-(1-phenylethyl)indolin-2-one (**P$_{41}$**).

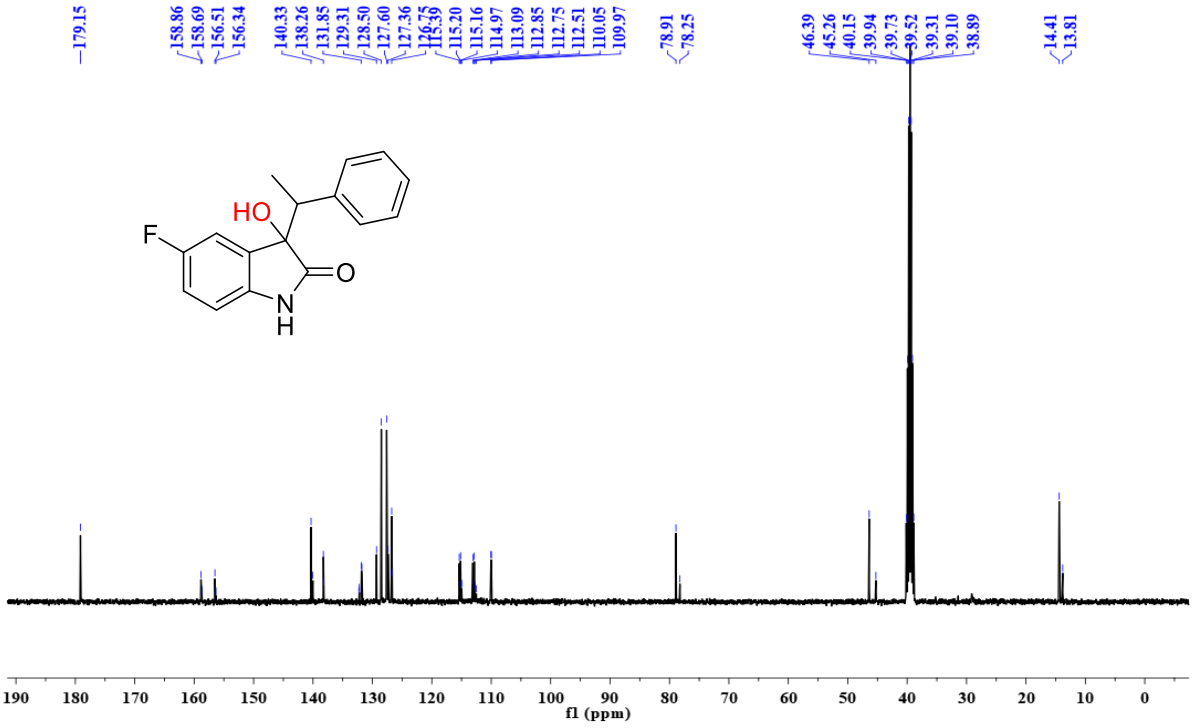


Figure S86. ^{13}C{^{1}H} NMR (101 MHz, DMSO-d_6) of 5-fluoro-3-hydroxy-3-(1-phenylethyl)indolin-2-one (**P$_{41}$**).

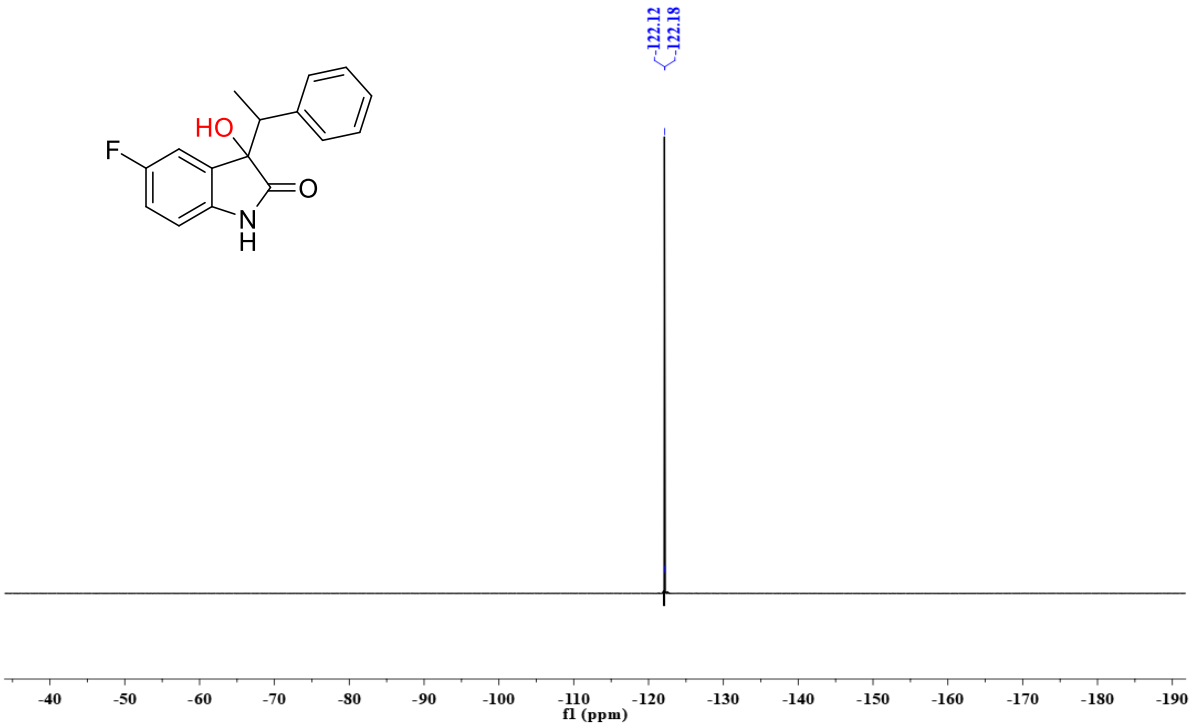
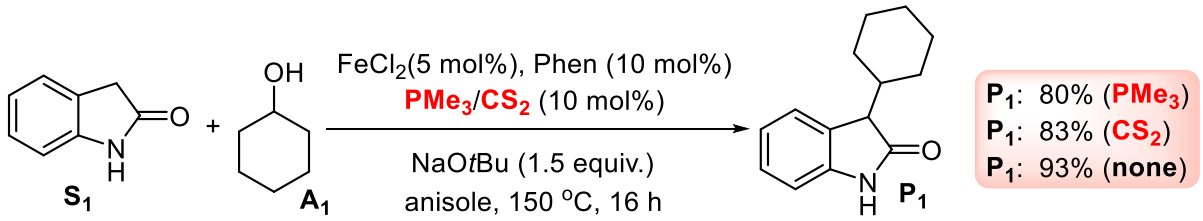


Figure S87. $^{19}F\{^{1}H\}$ NMR (377 MHz, DMSO-d_6) of 5-fluoro-3-hydroxy-3-(1-phenylethyl)indolin-2-one (**P$_{41}$**).

Mechanistic studies for C(α)-alkylation of oxindole:

(a) Catalyst Poisoning test:

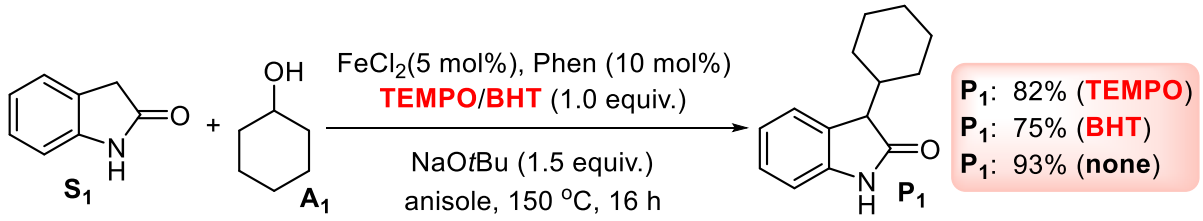


In a dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (0.040 g, 0.30 mmol), cyclohexanol (0.06 g, 0.6 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10- phenanthroline (0.054 g, 10 mol%), NaO*t*Bu (0.044 g, 0.45 mmol) and anisole (1 mL) was added under a nitrogen atmosphere. To this reaction mixture, PMe_3/CS_2 (5 mol%) was added. Then the reaction mixture was stirred at 150 °C for 16 h. The reaction mixture was cooled down to room temperature upon completion and concentrated in vacuum. The purification of product was done by column chromatography using hexane/EtOAc as eluent. The isolation of the expected product **P1** in good yield suggested the homogeneous behaviour of the catalyst.

Table S5: Product %yield upon varying of catalyst poisoners:

Entry	Catalyst Poisoners (3 mol%)	Yields
1	PMe_3	(0.051 g, 80%)
2	CS_2	(0.053 g, 83%)

(b) Radical scavenging test:

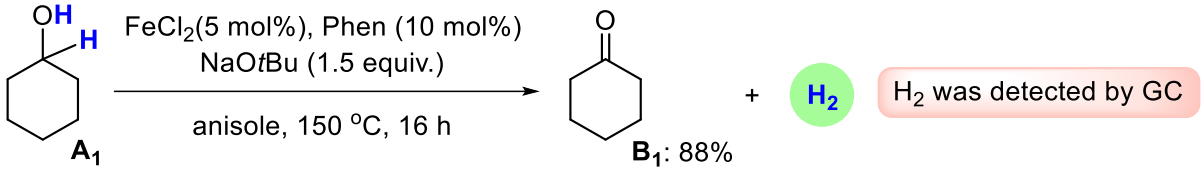


In a dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (0.040 g, 0.30 mmol), cyclohexanol (0.06 g, 0.6 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10- phenanthroline (0.054 g, 10 mol%), NaO*t*Bu (0.044 g, 0.45 mmol), radical scavenger (1 equiv.) and anisole (1 mL) was added under a nitrogen atmosphere. Then the reaction mixture was stirred at 150 °C for 16 h. The reaction mixture was cooled down to room temperature upon completion and concentrated in vacuum. The purification of product was done by column chromatography using hexane/EtOAc as eluent. The isolation of the expected product in **P1** high yields (listed below) suggested a mechanistic path which does not follow radical pathway.

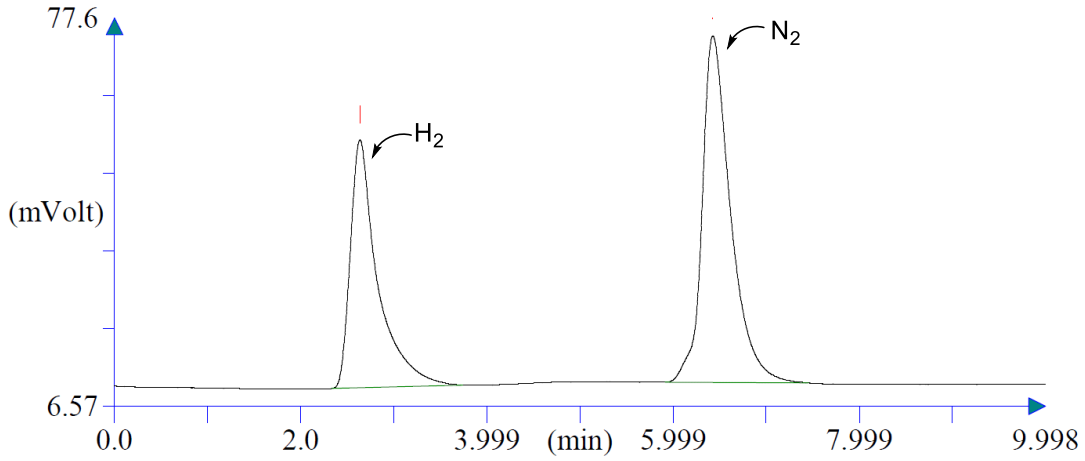
Table S6: Product %yield upon varying of radical quencher:

Entry	Radical Scavenger (1 equiv)	Yields
1	TEMPO	(0.052 g, 82%)
2	BHT	(0.048 g, 75%)

(c) Dehydrogenation of benzyl alcohol: Dihydrogen detection



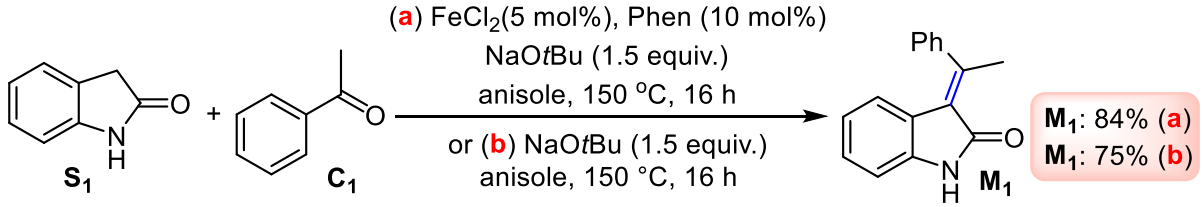
To an oven-dried 10 mL resealable vial, $FeCl_2$ (0.1 mmol, 1 equiv.), 1,10-phenanthroline (0.4 mmol, 2 equiv.), cyclohexanol (2.0 mmol, 20 equiv.), NaO*t*Bu (4.0 mmol, 4 equivalent), anisole (3 mL) were added inside the glove box. The vial was sealed and the reaction mixture was heated at 150 °C for 16 h. Then, the gaseous phase inside the vial was analysed on GC (TCD detector) which showed the formation of dihydrogen.



Peak Number (#)	Retention Time (min)	Area (.1*uV*sec)	Area % (%)
1	2.637	9245668	40.598
2	6.427	13528190	59.402
		22773860	

Figure S88. GC chromatogram of hydrogen gas evolved in the dehydrogenation of cyclohexanol.

(d) C=C bond formation by Knoevenagel condensation: Involvement of aldehyde intermediate



Procedure A: In a dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (0.040 g, 0.30 mmol), acetophenone (0.06 g, 0.6 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10-phenanthroline (0.054 g, 10 mol%), NaO*t*Bu (0.044 g, 0.45 mmol) and anisole (1 mL) was added under a nitrogen atmosphere. Then the reaction mixture was stirred at 150 °C for 16 h. The reaction mixture was cooled down to room temperature upon completion and concentrated in vacuum. The residue was purified by column chromatography using hexane/EtOAc as an eluent to afford pure product 3-(1-phenylethylidene)indolin-2-one (**M1**) as a yellow oil (0.059 g, 84%). The desired product **M1** was characterized by ^1H and ^{13}C NMR spectroscopies.

Procedure B: In a dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (0.040 g, 0.30 mmol), acetophenone (0.06 g, 0.6 mmol), NaO*t*Bu (0.044 g, 0.45 mmol) and anisole (1 mL) was added under a nitrogen atmosphere. Then the reaction mixture was stirred at 150 °C for 16 h. The reaction mixture was cooled down to room temperature upon completion and concentrated in vacuum. The residue was purified by column chromatography using hexane/EtOAc as an eluent to afford pure product 3-(1-phenylethylidene)indolin-2-one (**M1**) as a yellow oil (0.053 g, 75%). The desired product **M1** was characterized by ^1H and ^{13}C NMR spectroscopies.[4] ^1H NMR (400 MHz, $CDCl_3$) δ 8.99 (s, 1H), 7.55 – 7.46 (m, 3H), 7.33 (d, *J* = 6.5 Hz, 2H), 7.10 (t, *J* = 7.6 Hz, 1H), 6.87 (d, *J* = 7.7 Hz, 1H), 6.64 (t, *J* = 7.7 Hz, 1H), 6.17 (d, *J* = 7.8 Hz, 1H), 2.84 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.5, 155.6, 143.1, 139.9, 129.3, 128.5, 128.2, 126.6, 124.0, 123.5, 123.2, 121.4, 109.5, 23.0.

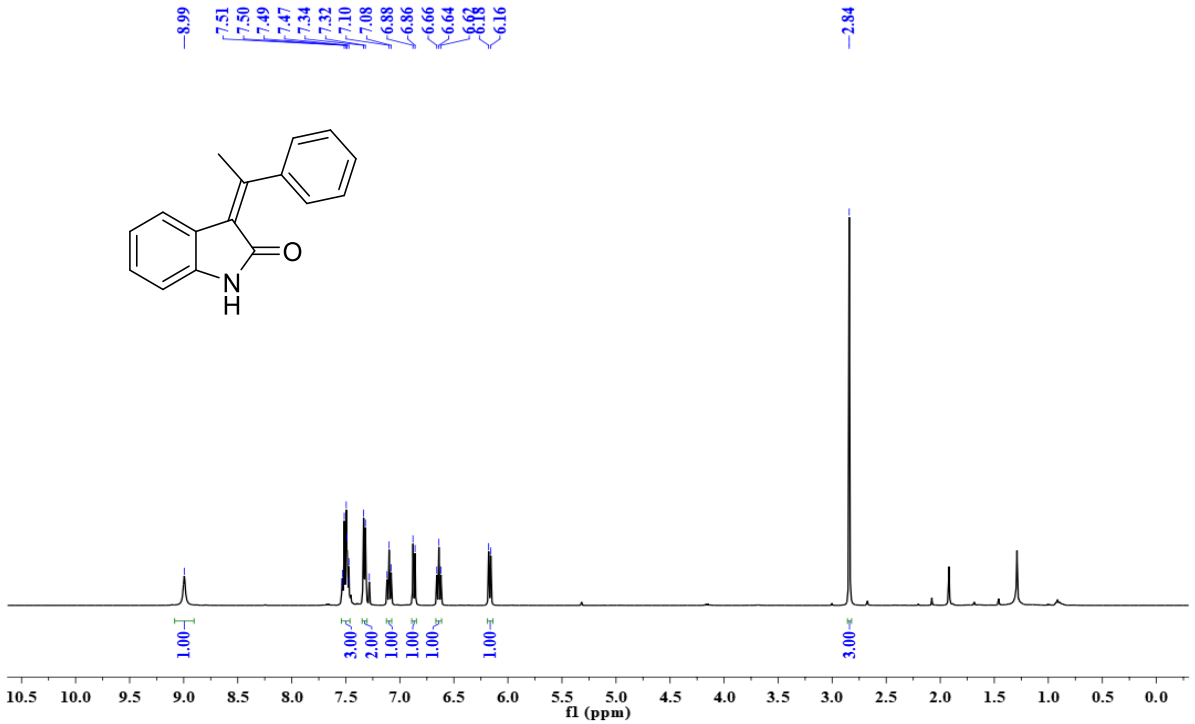


Figure S89. ^{1}H NMR (400 MHz, $CDCl_3$) of 3-(1-phenylethylidene)indolin-2-one (**M1**).

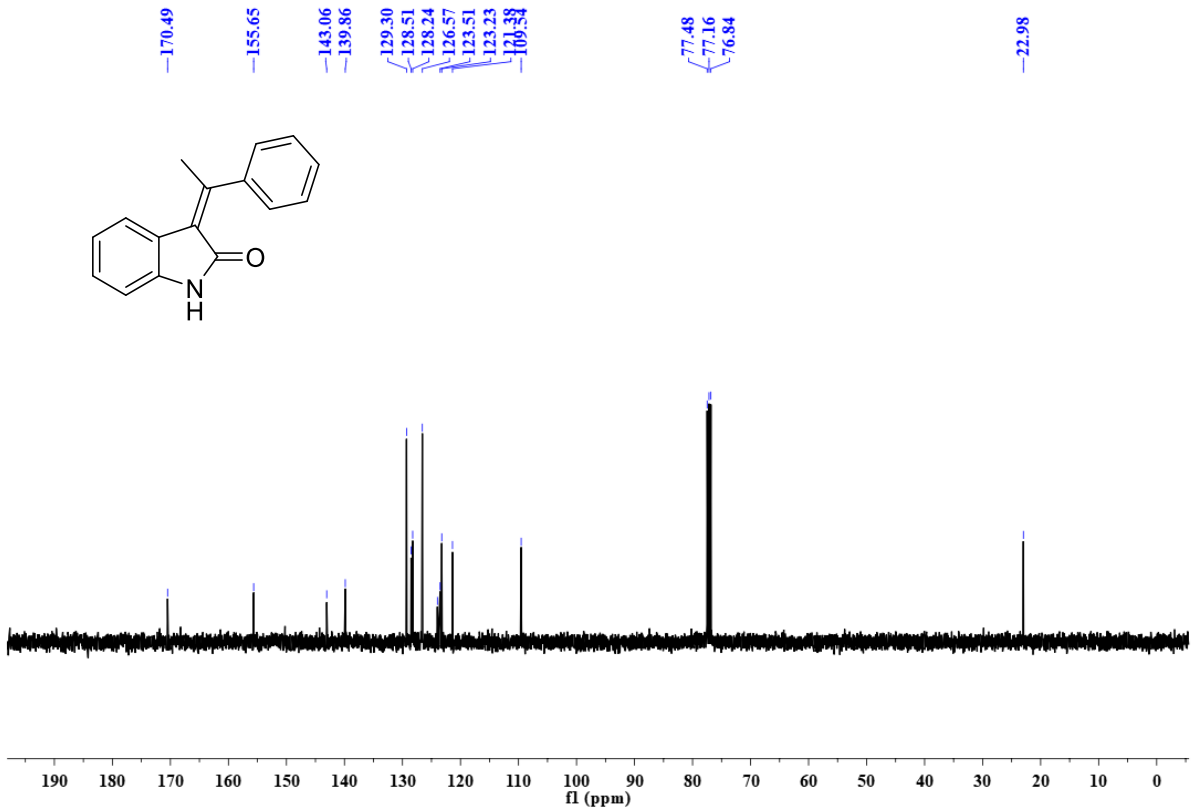
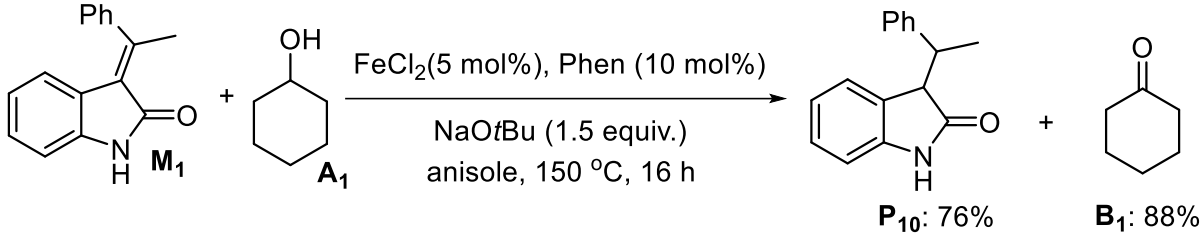


Figure S90. $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) of 3-(1-phenylethylidene)indolin-2-one (**M1**).

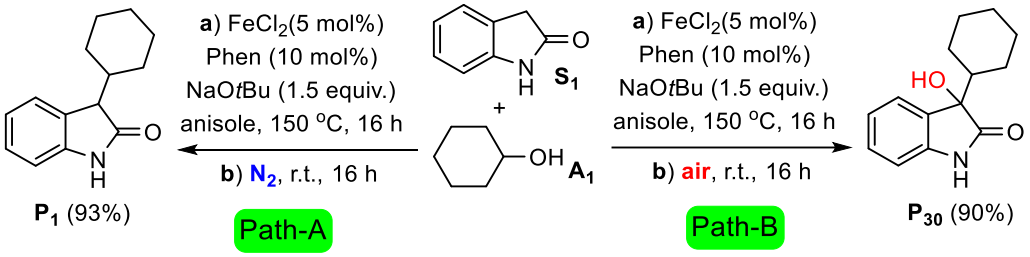
(e) Hydrogenation of α,β-unsaturated compound: Involvement of α,β-unsaturated intermediate



In a dried pressure tube fitted with a magnetic stir bar, a mixture of **M1** (0.07 g, 0.30 mmol), cyclohexanol (0.06 g, 0.6 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10-phenanthroline (0.054 g, 10 mol%), NaO*t*Bu (0.044 g, 0.45 mmol) and anisole (1 mL) was added under a nitrogen atmosphere. Then the reaction mixture was stirred at 150 °C for 16 h. The reaction mixture was cooled down to room temperature upon completion and concentrated in vacuum. The residue was purified by column chromatography using hexane/EtOAc as an eluent to afford pure product **P10** as a colourless oil (0.54 g, 76%).

Mechanistic studies for C(α)-alkylation and C-H hydroxylation of oxindole:

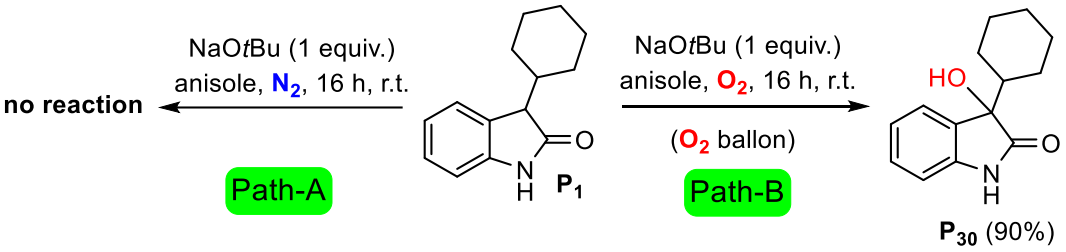
(a) One-pot alkylation-hydroxylation of 2-oxindole under inert and aerobic condition:



Path-A: In a dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (0.040 g, 0.30 mmol), cyclohexanol (0.06 g, 0.6 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10- phenanthroline (0.054 g, 10 mol%), NaO*t*Bu (0.044 g, 0.45 mmol) and anisole (1 mL) was added under a nitrogen atmosphere. Then the reaction mixture was heated at 150 °C in a preheated oil bath for 16 h. After cooling down to room temperature and the reaction mixture was stirred under nitrogen for 16 h. Finally, concentrated by removing the solvent under vacuum, and the residue was purified by column chromatography with hexane/ethyl acetate (hexane/ethyl acetate = 8.5:1.5) as eluent to give the desired product. The successful isolation of product **P1** in a high yield implies that under the nitrogen atmosphere, there is an absence of any hydroxylated product.

Path-B: In a dried pressure tube fitted with a magnetic stir bar, a mixture of 2-oxindole (0.040 g, 0.30 mmol), cyclohexanol (0.06 g, 0.6 mmol), $FeCl_2$ (0.020 g, 5 mol%), 1,10- phenanthroline (0.054 g, 10 mol%), NaO*t*Bu (0.044 g, 0.45 mmol) and anisole (1 mL) was added under a nitrogen atmosphere. Then the reaction mixture was heated at 150 °C in a preheated oil bath for 16 h. After cooling down to room temperature and the reaction mixture was stirred under nitrogen for 16 h. Finally, concentrated by removing the solvent under vacuum, and the residue was purified by column chromatography with hexane/ethyl acetate (hexane/ethyl acetate = 7:3) as eluent to give the desired product. The successful isolation of the anticipated product **P30** in a favorable yield indicates that in the presence of air, hydroxylated products are likely to form.

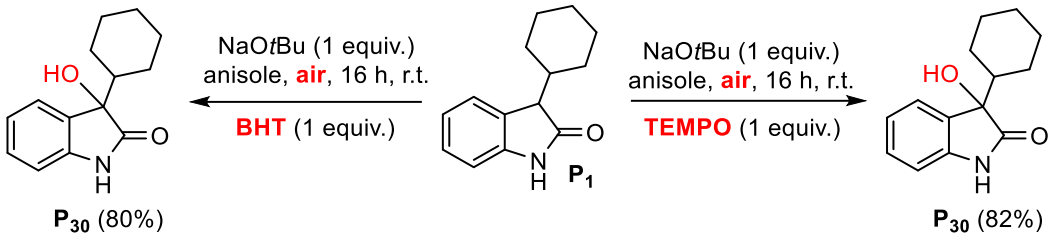
(b) **Hydroxylation of 3-alkyl-2-oxindole under inert and aerobic condition:**



Path-A: In a dried pressure tube fitted with a magnetic stir bar, a mixture of 3-cyclohexylindolin-2-one (**P1**) (0.043 g, 0.20 mmol), NaO*t*Bu (0.020 g, 0.2 mmol) and anisole (1 mL) was added. Then the reaction mixture was stirred under nitrogen at room temperature for 16 h. Finally, concentrated by removing the solvent under vacuum, and the residue was purified by column chromatography with hexane/ethyl acetate (hexane/ethyl acetate = 8.5:1.5) as eluent to give the desired product. The successful isolation of product **P1** in a high yield implies that under the nitrogen atmosphere, there is an absence of any hydroxylated product.

Path-B: In a dried pressure tube fitted with a magnetic stir bar, a mixture of 3-cyclohexylindolin-2-one (**P1**) (0.043 g, 0.20 mmol), NaO*t*Bu (0.020 g, 0.2 mmol) and anisole (1 mL) was added. Then the reaction mixture was stirred under oxygen (using oxygen ballon) at room temperature for 16 h. Finally, concentrated by removing the solvent under vacuum, and the residue was purified by column chromatography with hexane/ethyl acetate (hexane/ethyl acetate = 7:3) as eluent to give the desired product. The successful isolation of the desired product **P30** in a favorable yield indicates that in the presence of oxygen, this is a base catalyzed hydroxylation process.

(c) Radical scavenging test:



In a dried pressure tube fitted with a magnetic stir bar, a mixture of 3-cyclohexylindolin-2-one (**P1**) (0.043 g, 0.20 mmol), NaO*t*Bu (0.020 g, 0.2 mmol), radical scavenger (1 equiv.) and anisole (1 mL) was added under aerobic condition. Then the reaction mixture was stirred at room temperature for 16 h. Upon completion of the reaction, the reaction mixture was concentrated in vacuum. The purification of product was done by column chromatography using hexane/EtOAc as eluent. The isolation of the expected product in **P30** high yields (listed below) suggested a mechanistic path which does not follow radical pathway.

Table S7: Product %yield upon varying of radical quencher:

Entry	Radical Scavenger (1 equiv)	Yields
1	TEMPO	(0.052 g, 82%)
2	BHT	(0.048 g, 80%)

X-ray structure determination

Crystallographic data and structure determinations details are compiled in Table **S7**. The crystals were obtained by slow evaporation of **P5**, **P18** and **P34** in DCM at r.t. The crystals were coated with silicon oil on a glass slide and a suitable single crystal was mounted on a glass fibre. Crystal data were collected with a Rigaku Oxford diffractometer and with an INCOATEC micro source (Cu-Kα radiation, λ = 1.54184 Å, multilayer optics) at 100 K and 293 K respectively. The structure was determined using direct methods employed in ShelXT,[5] OleX,[6] and refinement was carried out using least-square minimization implemented in ShelXL.[7] All nonhydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom positions were fixed geometrically in idealized positions and were refined using a riding model. CCDC 2354252 (for **P5**), CCDC 2354253 (for **P18**) and CCDC 2354254 (for **P34**) contains the supplementary crystallographic data for this paper.

Table S8. Crystallographic Data and Refinement Parameters for **P5** and **P63.**

	P5	**P18**	**P34**
Empirical formula	$C_{11}H_{13}NO$	$C_{13}H_{14}ClNO$	$C_{32}H_{30}N_2O_4$
CCDC	2354252	2354253	2354254
Formula weight (g mol^{-1})	175.22	235.70	506.58
Temperature	297.15	100.01(10)	100.04(10)
Wavelength	1.54184	1.54184	1.54184
Crystal system	monoclinic	monoclinic	monoclinic
Space group	*I2/a*	*P2$_1$/n*	*Ia*
a (Å)	21.3851(7)	10.9223(2)	7.2634(2)
b (Å)	5.29270(10)	5.97560(10)	24.2647(7)
c (Å)	18.3278(6)	18.2274(4)	14.6681(4)
α (deg)	90	90	90
β (deg)	111.340(4)	103.405(2)	95.182(2)
γ (deg)	90	90	90
volume (Å^3)	1932.20(11)	1157.24(4)	2574.60(12)
Z	8	4	4
ρ_{calc} (g/cm^3)	1.205	1.353	1.307
μ (mm^{-1})	0.610	2.729	0.693
F(000)	752.0	496.0	1072.0
Crystal Size	0.2 × 0.2 × 0.1 mm^3	0.2 × 0.2 × 0.1 mm^3	0.2 × 0.2 × 0.1 mm^3
2θ Range (deg)	8.878 - 150.942	8.654 – 150.748	7.064 - 156.516
Index Ranges	-26 ≤ h ≤ 26, -6 ≤ k ≤ 6, -23 ≤ l ≤ 22	-13 ≤ h ≤ 13, -7 ≤ k ≤ 6, -22 ≤ l ≤ 22	-9 ≤ h ≤ 4, -25 ≤ k ≤ 30, -18 ≤ l ≤ 18
Reflections collected	10008	9232	10691
Independent reflections (R_{int})	1973(0.0364)	2344 (0.0465)	3351 (0.0592)
Completeness to theta = 25.07^0	99.7	99.9	99.96
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2

Data/Restraints/parameters	1973/0/120	2344/0/145	3351/2/347
Goodness-of-fit on *F*2	1.055	1.068	1.074
Final *R* indices [I>2σ(I)]	R_1 = 0.0403, wR_2 = 0.1097	R_1 = 0.0364, wR_2 = 0.0998	R_1 = 0.0436, wR_2 = 0.1102
R indices (all data)	R_1 = 0.0473, wR_2 = 0.1157	R_1 = 0.0386, wR_2 = 0.1017	R_1 = 0.0485, wR_2 = 0.1155
Largest diff. peak/hole (e Å^{-3})	0.12/-0.21	0.46/-0.37	0.22/-0.21

Figure S91. Molecular Structure of complex **P5** showing 50% Ellipsoids

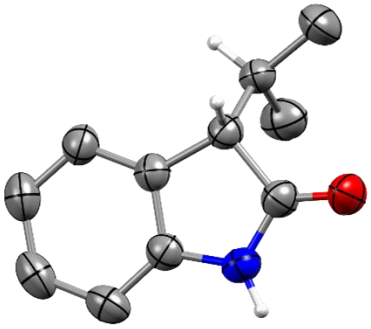


Figure S92. Molecular Structure of complex **P18** showing 50% Ellipsoids

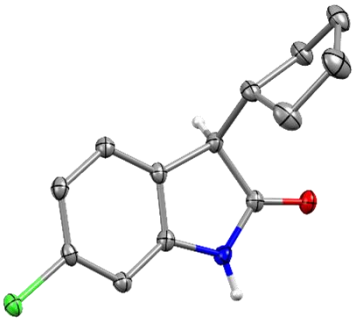


Figure S93. Molecular Structure of complex **P34** showing 50% Ellipsoids

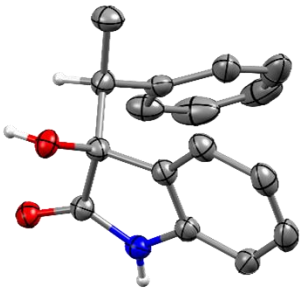


Table S9. Method A (anisole, [Fe] 5 mol%, 1,10-Phen 10 mol%, NaO*t*Bu 1.5 eq, 150 °C, 16 h): Zero Pass CHEM21 green metrics toolkit

Supplementary Information: Appendix 2 — **Summary of Zero Pass Metrics Toolkit**

Yield, conversion, selectivity, AE, RME

Reactant (Limiting Reactant First)	Mass (g)	MW	Mol	Catalyst	Mass (g)	Reagent	Mass (g)	Reaction solvent	Volume (cm^3)	Density ($g\ ml^{-1}$)	Mass (g)	Work up chemical	Mass (g)	Workup solvent	Volume (cm3)	Density ($g\ ml^{-1}$)	Mass (g)
2-oxindole	1.33	133.15	0.01	FeCl2	0.06			anisole	10.00	1.00	9.95			Ethyl acetate	25.00	0.90	22.55
Cyclohexanol	2.00	100.15	0.02	1,10-Phen	0.18												
NaOtBu	1.41	96.10	0.01								0.00						0.00
Total	4.74	329.40			0.24		0.00				9.95		0.00				22.55

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecualr\ weight\ of\ reactants} \times 100$$

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

		Flag
Yield	90.0	90.0
Conversion	100.0	100.0
Selectivity	90.0	90.0
AE	65.4	
RME	40.8	

	mass	mw	mol
Product	1.937	215.296	0.0089969

	mass
Unreacted limiting reactant	0.000

Solvents (Zero Pass)

Highly hazardous solvents (Red flag for any of the following)	List Highly Hazardous Solvents Below
Et_2O, Benzene, CCl_4, chloroform, DCE, nitromethane, CS_2, HMPA	None

Health and Safety (Zero Pass)

Health & safety (Red flag for any of the following)		List substances plus the red flagged H-codes below
Highly explosive	H200, H201, H202, H203	None
Explosive thermal runaway	H240	None
Fatally toxic	H300, H310, H330	None
Mutagenic	H350	None
Repro-toxic	H360	None
Serious environmental implications	H420	None

Table S10. Method B (neat, [Fe] 5 mol%, 1,10-Phen 10 mol%, NaO*t*Bu 1.5 eq, 150 °C, 8 h): Zero Pass CHEM21 green metrics toolkit

Supplementary Information: Appendix 2 — **Summary of Zero Pass Metrics Toolkit**

Yield, conversion, selectivity, AE, RME

Reactant (Limiting Reactant First)	Mass (g)	MW	Mol	Catalyst	Mass (g)	Reagent	Mass (g)	Reaction solvent	Volume (cm^3)	Density ($g\ ml^{-1}$)	Mass (g)	Work up chemical	Mass (g)	Workup solvent	Volume (cm3)	Density ($g\ ml^{-1}$)	Mass (g)
2-oxindole	1.33	133.15	0.01	FeCl2	0.06						0.00			Ethyl acetate	25.00	0.90	22.55
Cyclohexanol	4.01	100.15	0.04	1,10-Phen	0.18												
NaOtBu	1.41	96.10	0.01								0.00						0.00
Total	6.75	329.40			0.24		0.00				0.00		0.00				22.55

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecualr\ weight\ of\ reactants} \times 100$$

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

		Flag
Yield	90.0	90.0
Conversion	100.0	100.0
Selectivity	90.0	90.0
AE	65.4	
RME	28.7	

	mass	mw	mol
Product	1.937	215.296	0.0089969
	mass		
Unreacted limiting reactant	0.000		

Solvents (Zero Pass)

Highly hazardous solvents (Red flag for any of the following)	List Highly Hazardous Solvents Below
Et_2O, Benzene, CCl_4, chloroform, DCE, nitromethane, CS_2, HMPA	None

Health and Safety (Zero Pass)

Health & safety (Red flag for any of the following)		List substances plus the red flagged H-codes below
Highly explosive	H200, H201, H202, H203	None
Explosive thermal runaway	H240	None
Fatally toxic	H300, H310, H330	None
Mutagenic	H350	None
Repro-toxic	H360	None
Serious environmental implications	H420	None

Table S11. Method C (toluene, [Co] 10 mol%, KOH 2 eq, 150 °C, 16 h): Zero Pass CHEM21 green metrics toolkit

Supplementary Information: Appendix 2 — **Summary of Zero Pass Metrics Toolkit**

Yield, conversion, selectivity, AE, RME

Reactant (Limiting Reactant First)	Mass (g)	MW	Mol	Catalyst	Mass (g)	Reagent	Mass (g)	Reaction solvent	Volume (cm^3)	Density ($g\ ml^{-1}$)	Mass (g)	Work up chemical	Mass (g)	Workup solvent	Volume (cm3)	Density ($g\ ml^{-1}$)	Mass (g)
2-oxindole	1.33	133.15	0.01	FeCl2	0.06			Toluene	10.00	1.00	9.95			Ethyl acetate	20.00	0.90	18.04
Cyclohexanol	2.00	100.15	0.02	1,10-Phen	0.18												
NaOtBu	1.41	96.10	0.01								0.00						0.00
Total	4.74	329.40			0.24		0.00				9.95		0.00				18.04

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecualr\ weight\ of\ reactants} \times 100$$

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

		Flag	
Yield	91.0		91.0
Conversion	100.0		100.0
Selectivity	91.0		91.0
AE	65.4		
RME	41.3		

	mass	mw	mol
Product	1.959	215.296	0.0090991
	mass		
Unreacted limiting reactant	0.000		

Solvents (Zero Pass)

Highly hazardous solvents (Red flag for any of the following)	List Highly Hazardous Solvents Below
Et_2O, Benzene, CCl_4, chloroform, DCE, nitromethane, CS_2, HMPA	None

Health and Safety (Zero Pass)

Health & safety (Red flag for any of the following)		List substances plus the red flagged H-codes below
Highly explosive	H200, H201, H202, H203	None
Explosive thermal runaway	H240	None
Fatally toxic	H300, H310, H330	None
Mutagenic	H350	None
Repro-toxic	H360	None
Serious environmental implications	H420	None

Table S12. Method D (anisole,[Fe] 5 mol%, 1,10-Phen 10 mol%, NaO*t*Bu 1.5 eq, 150 °C, 32 h): Zero Pass CHEM21 green metrics toolkit

Supplementary Information: Appendix 2 — **Summary of Zero Pass Metrics Toolkit**

Yield, conversion, selectivity, AE, RME

Reactant (Limiting Reactant First)	Mass (g)	MW	Mol	Catalyst	Mass (g)	Reagent	Mass (g)	Reaction solvent	Volume (cm^3)	Density ($g\ ml^{-1}$)	Mass (g)	Work up chemical	Mass (g)	Workup solvent	Volume (cm3)	Density ($g\ ml^{-1}$)	Mass (g)
2-oxindole	1.33	133.15	0.01	FeCl2	0.06			anisole	10.00	1.00	9.95			Ethyl acetate	25.00	0.90	22.55
Cyclohexanol	2.00	100.15	0.02	1,10-Phen	0.18												
NaOtBu	1.41	96.10	0.01								0.00						0.00
Oxygen	0.16	32.00	0.01								0.00						0.00
Total	4.90	361.40			0.24		0.00				9.95		0.00				22.55

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecualr\ weight\ of\ reactants} \times 100$$

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

		Flag
Yield	90.0	90.0
Conversion	100.0	100.0
Selectivity	90.0	90.0
AE	64.0	
RME	42.4	

	mass	mw	mol
Product	2.081	231.295	0.0089972

	mass
Unreacted limiting reactant	0.000

Solvents (Zero Pass)

Highly hazardous solvents (Red flag for any of the following)	List Highly Hazardous Solvents Below
Et_2O, Benzene, CCl_4, chloroform, DCE, nitromethane, CS_2, HMPA	None

Health and Safety (Zero Pass)

Health & safety (Red flag for any of the following)		List substances plus the red flagged H-codes below
Highly explosive	H200, H201, H202, H203	None
Explosive thermal runaway	H240	None
Fatally toxic	H300, H310, H330	None
Mutagenic	H350	None
Repro-toxic	H360	None
Serious environmental implications	H420	None

Table S13. Method A (anisole, [Fe] 5 mol%, 1,10-Phen 10 mol%, NaO*t*Bu 1.5 eq, 150 °C, 16 h): First Pass CHEM21 green metrics toolkit

Supplementary Information: Appendix 2 — **Summary of First Pass Metrics Toolkit**

Yield, AE, RME, MI/PMI and OE

Reactant (Limiting Reactant First)	Mass (g)	MW	Mol	Catalyst	Mass (g)	Reagent	Mass (g)	Reaction solvent	Volume (cm^3)	Density ($g\ ml^{-1}$)	Mass (g)	Work up chemical	Mass (g)	Workup solvent	Volume (cm3)	Density ($g\ ml^{-1}$)	Mass (g)
2-oxindole	1.33	133.15	0.01	FeCl2	0.06			anisole	10.00	1.00	9.95			Ethyl acetate	25.00	0.90	22.55
Cyclohexanol	2.00	100.15	0.02	1,10-Phen	0.18												
NaOtBu	1.41	96.10	0.01								0.00						0.00
Total	4.74	329.40			0.24		0.00				9.95		0.00				22.55

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecualr\ weight\ of\ reactants} \times 100$$

$$mass\ intensity = \frac{total\ mass\ in\ a\ process\ or\ process\ step}{mass\ of\ product}$$

$$OE = \frac{RME}{AE} \times 100$$

		Flag
Yield	90.0	90.0
Conversion	100.0	100.0
Selectivity	90.0	90.0
AE	65.4	
RME	40.8	OE 62.5
PMI total	19.4	
PMI Reaction	7.7	
PMI reactants, reagents, catlyst	2.6	
PMI reaction solvents	5.1	
PMI Workup	11.6	
PMI Workup chemical	0.0	
PMI workup solvents	11.6	

	Mass	MW	Mol
Product	1.937	215.296	0.01
	mass		
Unreacted limiting reactant	0.00		

Solvents (First Pass)

		List solvents below
Preferred solvents	**water,** EtOH, nBuOH, AcOipr, AcOnBu, PhOMe, MeOH, tBuOH, BnOH, ethylene glycol, acetone, MEK, MIBK, **AcOEt,** sulfolane	PhOMe
Problematic solvents: (acceptable only if substitution does not offer advantages)	DMSO, cyclohexanone, DMPU, AcOH, Ac2O, Acetonitrile, AcOMe, THF, heptane, Me-cyclohexane, toluene, xylene, MTBE, **cyclohexane**, chlorobenzene, formic acid, pyridine, Me-THF	none
Hazardous solvents: These solvents have significant health and/or safety concerns.	dioxane, pentane, TEA, diisopropyl ether, DME, DCM, DMF, DMA, NMP, methoxyethanol, hexane	none
Highly hazardous solvents: The solvents which are agreed not to be used, even in screening	Et_2O, Benzene, CCl_4, chloroform, DCE, nitromethane, CS_2, HMPA	none

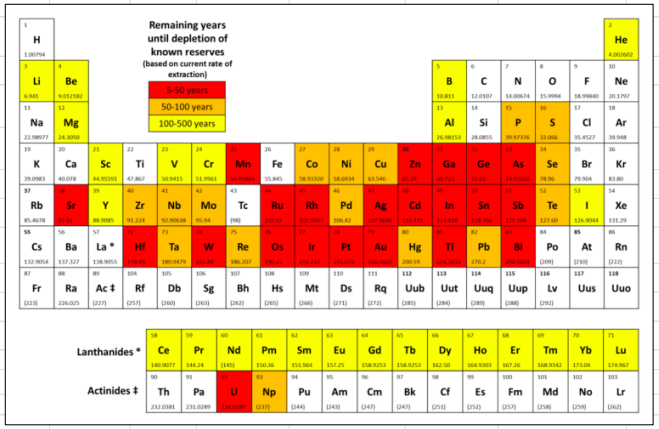
Catalyst/enzyme (First Pass)

		Tick
Catalyst or enzyme used, **or** reaction takes place	Green Flag	X
Use of stoichiometric quantities of reagents	Amber Flag	
Use of reagents in excess	Red Flag	

		Tick
Facile recovery of catalyst/enzyme	Green Flag	
catalyst/enzyme not recovered	Amber Flag	X

Critical elements

Supply remaining	Flag colour	Note element
5-50 years	Red Flag	
50-500 years	Amber Flag	
+500 years	Green Flag	Fe



Energy (First Pass)		Tick
Reaction run between 0 to 70°C	Green Flag	
Reaction run between -20 to 0 or 70 to 140°C	Amber Flag	X
Reaction run below -20 or above 140°C	Red Flag	

		Tick
Reaction run at reflux	Red Flag	
Reaction run 5°C or more below the solvent boiling point	Green Flag	X

Batch/flow		Tick
Flow	Green Flag	
Batch	Amber Flag	X

Work Up		List
quenching filtration centrifugation crystallisation Low temperature distillation/evaporation/ sublimation (< 140 °C at atmospheric	Green Flag	
solvent exchange, quenching into aqueous solvent	Amber Flag	
chromatography/ion exchange high temperature multiple recrystallisation	Red Flag	Chromatography

Health & safety

	Red Flag	Amber Flag	Green Flag	List substances and H-codes	List substances and H-codes	List substances and H-codes
Highly explosive	H200, H201, H202, H203	H205, H220, H224	If no red or amber flagged H codes present then green flag	**1,10-Phenanthroline**: H410	**Cyclohexanol**: H412; **NaOtBu:** H351	**2-oxindole:** H302, H312, H332,; **Cyclohexanol**: H302, H312, H315; H332, H319, H335 **NaOtBu:** H225, H251, H314, H335, H336, **Ethylacetate:** H225, H319, H336. **Anisole**: H226, H336; **$FeCl_2$**: H290, H302, H315, H318; **1,10-Phenanthroline**: H301
Explosive thermal runaway	H230, H240, H250	H241				
Toxic	H300, H310, H330	H301, H311, H331,				
Long Term toxicity	H340, H350, H360, H370, H372	H341, H351, H361, H371, H373				
Environmental implications	H400, H410, H411, H420	H401, H412				

Use of chemicals of environmental concern		List substances of very high concern
Chemical identified as Substances of Very High Concern by ChemSec which are utilised	Red Flag	None

Table S14. Method B (neat, [Fe] 5 mol%, 1,10-Phen 10 mol%, NaO*t*Bu 1.5 eq, 150 °C, 8 h): First Pass CHEM21 green metrics toolkit

Supplementary Information: Appendix 2 — **Summary of First Pass Metrics Toolkit**

Yield, AE, RME, MI/PMI and OE

Reactant (Limiting Reactant First)	Mass (g)	MW	Mol	Catalyst	Mass (g)	Reagent	Mass (g)	Reaction solvent	Volume (cm^3)	Density ($g\ ml^{-1}$)	Mass (g)	Work up chemical	Mass (g)	Workup solvent	Volume (cm3)	Density ($g\ ml^{-1}$)	Mass (g)
2-oxindole	1.33	133.15	0.01	FeCl2	0.06						0.00			Ethyl acetate	25.00	0.90	22.55
Cyclohexanol	4.01	100.15	0.04	1,10-Phen	0.18												
NaOtBu	1.41	96.10	0.01								0.00						0.00
Total	6.75	329.40			0.24		0.00				0.00		0.00				22.55

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecualr\ weight\ of\ reactants} \times 100$$

$$mass\ intensity = \frac{total\ mass\ in\ a\ process\ or\ process\ step}{mass\ of\ product}$$

$$OE = \frac{RME}{AE} \times 100$$

		Flag	
Yield	90.0		90.0
Conversion	100.0		100.0
Selectivity	90.0		90.0
AE	65.4		
RME	28.7	OE	43.9
PMI total	15.3		
PMI Reaction	3.6		
PMI reactants, reagents, catlyst	3.6		
PMI reaction solvents	0.0		
PMI Workup	11.6		
PMI Workup chemical	0.0		
PMI workup solvents	11.6		

	Mass	MW	Mol
Product	1.937	215.296	0.01
	mass		
Unreacted limiting reactant	0.00		

Solvents (First Pass)

		List solvents below
Preferred solvents	**water,** EtOH, nBuOH, AcOipr, AcOnBu, PhOMe, MeOH, tBuOH, BnOH, ethylene glycol, acetone, MEK, MIBK, **AcOEt,** sulfolane	neat
Problematic solvents: (acceptable only if substitution does not offer advantages)	DMSO, cyclohexanone, DMPU, AcOH, Ac2O, Acetonitrile, AcOMe, THF, heptane, Me-cyclohexane, toluene, xylene, MTBE, **cyclohexane**, chlorobenzene, formic acid, pyridine, Me-THF	none
Hazardous solvents: These solvents have significant health and/or safety concerns.	dioxane, pentane, TEA, diisopropyl ether, DME, DCM, DMF, DMA, NMP, methoxyethanol, hexane	none
Highly hazardous solvents: The solvents which are agreed not to be used, even in screening	Et_2O, Benzene, CCl_4, chloroform, DCE, nitromethane, CS_2, HMPA	none

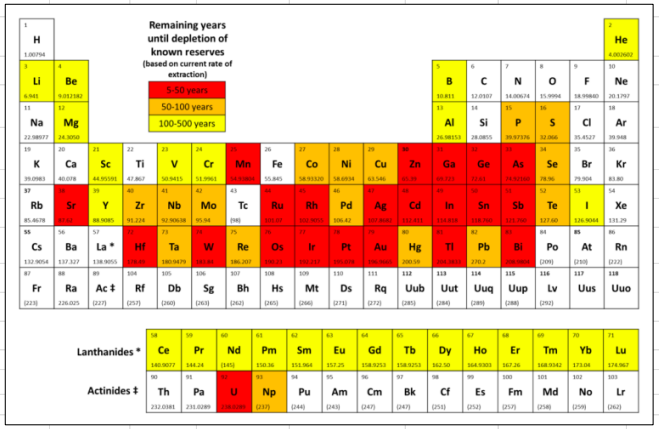
Catalyst/enzyme (First Pass)

		Tick
Catalyst or enzyme used, **or** reaction takes place	Green Flag	X
Use of stoichiometric quantities of reagents	Amber Flag	
Use of reagents in excess	Red Flag	

		Tick
Facile recovery of catalyst/enzyme	Green Flag	
catalyst/enzyme not recovered	Amber Flag	X

Critical elements

Supply remaining	Flag colour	Note element
5-50 years	Red Flag	
50-500 years	Amber Flag	
+500 years	Green Flag	Fe



Energy (First Pass)		Tick
Reaction run between 0 to 70°C	Green Flag	
Reaction run between -20 to 0 or 70 to 140°C	Amber Flag	X
Reaction run below -20 or above 140°C	Red Flag	

		Tick
Reaction run at reflux	Red Flag	
Reaction run 5°C or more below the solvent boiling point	Green Flag	X

Batch/flow		Tick
Flow	Green Flag	
Batch	Amber Flag	X

Work Up		List
quenching filtration centrifugation crystallisation Low temperature distillation/evaporation/ sublimation (< 140 °C at atmospheric	Green Flag	
solvent exchange, quenching into aqueous solvent	Amber Flag	
chromatography/ion exchange high temperature multiple recrystallisation	Red Flag	Chromatography

Health & safety

	Red Flag	Amber Flag	Green Flag	List substances and H-codes	List substances and H-codes	List substances and H-codes
Highly explosive	H200, H201, H202, H203	H205, H220, H224	If no red or amber flagged H codes present then green flag	**1,10-Phenanthroline**: H410	**Cyclohexanol**: H412; **NaOtBu:** H351	**2-oxindole:** H302, H312, H332,; **Cyclohexanol**: H302, H312, H315; H332, H319, H335 **NaOtBu:** H225, H251, H314, H335, H336, **Ethylacetate:** H225, H319, H336. **Anisole**: H226, H336; **$FeCl_2$**: H290, H302, H315, H318; **1,10-Phenanthroline**: H301
Explosive thermal runaway	H230, H240, H250	H241				
Toxic	H300, H310, H330	H301, H311, H331,				
Long Term toxicity	H340, H350, H360, H370, H372	H341, H351, H361, H371, H373				
Environmental implications	H400, H410, H411, H420	H401, H412				

Use of chemicals of environmental concern		List substances of very high concern
Chemical identified as Substances of Very High Concern by ChemSec which are utilised	Red Flag	None

Table S15. Method C (toluene, [Co] 10 mol%, KOH 2 eq, 150 °C, 16 h): First Pass CHEM21 green metrics toolkit

Supplementary Information: Appendix 2 — **Summary of First Pass Metrics Toolkit**

Yield, AE, RME, MI/PMI and OE

Reactant (Limiting Reactant First)	Mass (g)	MW	Mol	Catalyst	Mass (g)	Reagent	Mass (g)	Reaction solvent	Volume (cm^3)	Density ($g\ ml^{-1}$)	Mass (g)	Work up chemical	Mass (g)	Workup solvent	Volume (cm3)	Density ($g\ ml^{-1}$)	Mass (g)
2-oxindole	1.33	133.15	0.01	[Co]	0.48			Toluene	10.00	0.87	8.67			Ethyl acetate	20.00	0.90	18.04
Cyclohexanol	2.00	100.15	0.02														
KOH	1.12	46.10	0.02								0.00						0.00
Total	4.46	279.40			0.48		0.00				8.67		0.00				18.04

		Flag	
Yield	91.0		91.0
Conversion	100.0		100.0
Selectivity	91.0		91.0
AE	77.1		
RME	44.0	OE	57.1
PMI total	16.2		
PMI Reaction	6.9		
PMI reactants, reagents, catlyst	2.5		
PMI reaction solvents	4.4		
PMI Workup	9.2		
PMI Workup chemical	0.0		
PMI workup solvents	9.2		

	Mass	MW	Mol
Product	1.959	215.296	0.01
	mass		
Unreacted limiting reactant	0.00		

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecualr\ weight\ of\ reactants} \times 100$$

$$mass\ intensity = \frac{total\ mass\ in\ a\ process\ or\ process\ step}{mass\ of\ product}$$

$$OE = \frac{RME}{AE} \times 100$$

Solvents (First Pass)

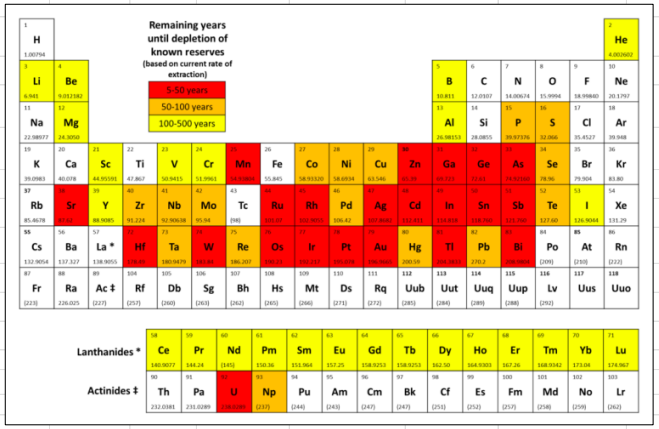
		List solvents below
Preferred solvents	**water,** EtOH, nBuOH, AcOipr, AcOnBu, PhOMe, MeOH, tBuOH, BnOH, ethylene glycol, acetone, MEK, MIBK, **AcOEt,** sulfolane	none
Problematic solvents: (acceptable only if substitution does not offer advantages)	DMSO, cyclohexanone, DMPU, AcOH, Ac2O, Acetonitrile, AcOMe, THF, heptane, Me-cyclohexane, toluene, xylene, MTBE, **cyclohexane**, chlorobenzene, formic acid, pyridine, Me-THF	Toluene
Hazardous solvents: These solvents have significant health and/or safety concerns.	dioxane, pentane, TEA, diisopropyl ether, DME, DCM, DMF, DMA, NMP, methoxyethanol, hexane	none
Highly hazardous solvents: The solvents which are agreed not to be used, even in screening	Et_2O, Benzene, CCl_4, chloroform, DCE, nitromethane, CS_2, HMPA	none

Catalyst/enzyme (First Pass)		**Tick**
Catalyst or enzyme used, **or** reaction takes place	Green Flag	X
Use of stoichiometric quantities of reagents	Amber Flag	
Use of reagents in excess	Red Flag	

		Tick
Facile recovery of catalyst/enzyme	Green Flag	
catalyst/enzyme not recovered	Amber Flag	X

Critical elements

Supply remaining	Flag colour	Note element
5-50 years	Red Flag	
50-500 years	Amber Flag	Co
+500 years	Green Flag	



Energy (First Pass)		Tick
Reaction run between 0 to 70°C	Green Flag	
Reaction run between -20 to 0 or 70 to 140°C	Amber Flag	X
Reaction run below -20 or above 140°C	Red Flag	

		Tick
Reaction run at reflux	Red Flag	X
Reaction run 5°C or more below the solvent boiling point	Green Flag	

Batch/flow		Tick
Flow	Green Flag	
Batch	Amber Flag	X

Work Up		List
quenching filtration centrifugation crystallisation Low temperature distillation/evaporation/ sublimation (< 140 °C at atmospheric	Green Flag	
solvent exchange, quenching into aqueous solvent	Amber Flag	
chromatography/ion exchange high temperature multiple recrystallisation	Red Flag	Chromatography

Health & safety

	Red Flag	Amber Flag	Green Flag	List substances and H-codes	List substances and H-codes	List substances and H-codes
Highly explosive	H200, H201, H202, H203	H205, H220, H224	If no red or amber flagged H codes present then green flag	None	**Cyclohexanol**: H412	**2-oxindole:** H302, H312, H332,; **Cyclohexanol**: H302, H312, H315; H332, H319, H335 **KOH:** H290, H302, H314, **Ethylacetate:** H225, H319, H336. **Anisole**: H226, H336
Explosive thermal runaway	H230, H240, H250	H241				
Toxic	H300, H310, H330	H301, H311, H331,				
Long Term toxicity	H340, H350, H360, H370, H372	H341, H351, H361, H371, H373				
Environmental implications	H400, H410, H411, H420	H401, H412				

Use of chemicals of environmental concern		List substances of very high concern
Chemical identified as Substances of Very High Concern by ChemSec which are utilised	Red Flag	None

Table S16. Method D (anisole,[Fe] 5 mol%, 1,10-Phen 10 mol%, NaO*t*Bu 1.5 eq, 150 °C, 32 h): First Pass CHEM21 green metrics toolkit

Supplementary Information: Appendix 2 — **Summary of First Pass Metrics Toolkit**

Yield, AE, RME, MI/PMI and OE

Reactant (Limiting Reactant First)	Mass (g)	MW	Mol	Catalyst	Mass (g)	Reagent	Mass (g)	Reaction solvent	Volume (cm^3)	Density ($g\ ml^{-1}$)	Mass (g)	Work up chemical	Mass (g)	Workup solvent	Volume (cm3)	Density ($g\ ml^{-1}$)	Mass (g)
2-oxindole	1.33	133.15	0.01	FeCl2	0.06			anisole	10.00	1.00	9.95			Ethyl acetate	25.00	0.90	22.55
Cyclohexanol	2.00	100.15	0.02	1,10-Phen	0.18												
NaOtBu	1.41	96.10	0.01								0.00						0.00
Oxygen	0.16	32.00	0.01								0.00						0.00
Total	4.90	361.40			0.24		0.00				9.95		0.00				22.55

$$RME = \frac{mass\ of\ isolated\ product}{total\ mass\ of\ reactants} \times 100$$

$$AE = \frac{molecular\ weight\ of\ product}{total\ molecualr\ weight\ of\ reactants} \times 100$$

$$mass\ intensity = \frac{total\ mass\ in\ a\ process\ or\ process\ step}{mass\ of\ product}$$

$$OE = \frac{RME}{AE} \times 100$$

		Flag	
Yield	90.0		90.0
Conversion	100.0		100.0
Selectivity	90.0		90.0
AE	64.0		
RME	42.4	OE	66.3
PMI total	18.1		
PMI Reaction	7.3		
PMI reactants, reagents, catlyst	2.5		
PMI reaction solvents	4.8		
PMI Workup	10.8		
PMI Workup chemical	0.0		
PMI workup solvents	10.8		

Product	Mass	MW	Mol
	2.081	231.295	0.01
	mass		
Unreacted limiting reactant	0.00		

Solvents (First Pass)

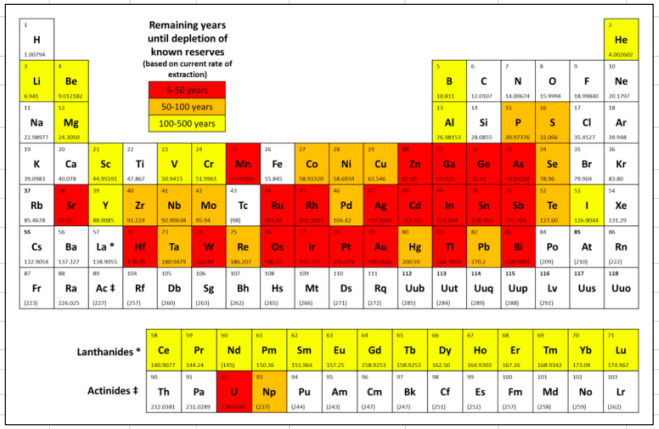
		List solvents below
Preferred solvents	**water,** EtOH, nBuOH, AcOipr, AcOnBu, PhOMe, MeOH, tBuOH, BnOH, ethylene glycol, acetone, MEK, MIBK, **AcOEt,** sulfolane	PhOMe
Problematic solvents: (acceptable only if substitution does not offer advantages)	DMSO, cyclohexanone, DMPU, AcOH, Ac2O, Acetonitrile, AcOMe, THF, heptane, Me-cyclohexane, toluene, xylene, MTBE, **cyclohexane**, chlorobenzene, formic acid, pyridine, Me-THF	none
Hazardous solvents: These solvents have significant health and/or safety concerns.	dioxane, pentane, TEA, diisopropyl ether, DME, DCM, DMF, DMA, NMP, methoxyethanol, hexane	none
Highly hazardous solvents: The solvents which are agreed not to be used, even in screening	Et_2O, Benzene, CCl_4, chloroform, DCE, nitromethane, CS_2, HMPA	none

Catalyst/enzyme (First Pass)		Tick
Catalyst or enzyme used, **or** reaction takes place	Green Flag	X
Use of stoichiometric quantities of reagents	Amber Flag	
Use of reagents in excess	Red Flag	

		Tick
Facile recovery of catalyst/enzyme	Green Flag	
catalyst/enzyme not recovered	Amber Flag	X

Critical elements

Supply remaining	Flag colour	Note element
5-50 years	Red Flag	
50-500 years	Amber Flag	
+500 years	Green Flag	Fe



Energy (First Pass)		Tick
Reaction run between 0 to 70°C	Green Flag	
Reaction run between -20 to 0 or 70 to 140°C	Amber Flag	X
Reaction run below -20 or above 140°C	Red Flag	

		Tick
Reaction run at reflux	Red Flag	
Reaction run 5°C or more below the solvent boiling point	Green Flag	X

Batch/flow		Tick
Flow	Green Flag	
Batch	Amber Flag	X

Work Up		List
quenching filtration centrifugation crystallisation Low temperature distillation/evaporation/ sublimation (< 140 °C at atmospheric	Green Flag	
solvent exchange, quenching into aqueous solvent	Amber Flag	
chromatography/ion exchange high temperature multiple recrystallisation	Red Flag	Chromatography

Health & safety

	Red Flag	Amber Flag	Green Flag	List substances and H-codes	List substances and H-codes	List substances and H-codes
Highly explosive	H200, H201, H202, H203	H205, H220, H224	If no red or amber flagged H codes present then green flag	**1,10-Phenanthroline**: H410	**Cyclohexanol**: H412; **NaOtBu:** H351	**2-oxindole:** H302, H312, H332,; **Cyclohexanol**: H302, H312, H315; H332, H319, H335 **NaOtBu:** H225, H251, H314, H335, H336, **Ethylacetate:** H225, H319, H336. **Anisole**: H226, H336; **$FeCl_2$**: H290, H302, H315, H318; **1,10-Phenanthroline**: H301
Explosive thermal runaway	H230, H240, H250	H241				
Toxic	H300, H310, H330	H301, H311, H331,				
Long Term toxicity	H340, H350, H360, H370, H372	H341, H351, H361, H371, H373				
Environmental implications	H400, H410, H411, H420	H401, H412				

Use of chemicals of environmental concern		List substances of very high concern
Chemical identified as Substances of Very High Concern by ChemSec which are utilised	Red Flag	None

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