Supporting Information

Organocatalytic Hydroalkoxylation/Claisen Rearrangement /Michael Addition Tandem Sequence: Divergent Synthesis of Multi-Substituted 2,3-Dihydrofurans and 2,3-Dihydropyrroles from Cyanohydrins

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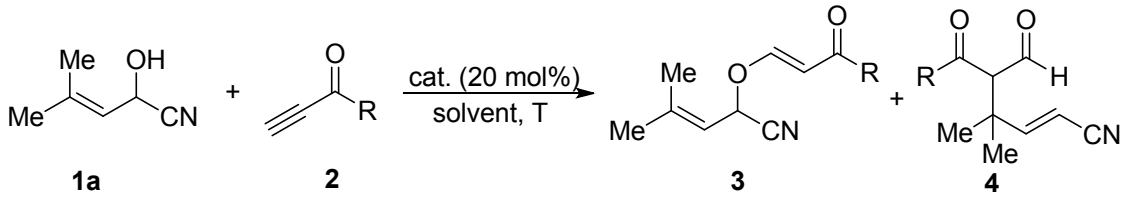
I. General Information

All reactions were carried out under inert atmospheric condition unless otherwise noted, and solvents were dried according to established procedures. Reactions were monitored by thin layer chromatography (TLC) visualizing with ultraviolet light (UV), $KMnO_4$, p-anisaldehyde stain, and phosphomolybdic acid (PMA) stain; column chromatography purifications were carried out using silica gel. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a 300 or 500 MHz spectrometer in $CDCl_3$, and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on 125 MHz spectrometer in $CDCl_3$ unless otherwise noted. Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane (TMS) and are referenced to residual protium in the NMR solvent ($CHCl_3$ = δ 7.26 ppm). Chemical shifts for carbon are reported in parts per million downfield from tetramethylsilane (TMS) and are referenced to the carbon resonances of the solvent residual peak ($CDCl_3$ = δ 77.16 ppm). NMR data are presented as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant in Hertz (Hz), integration. Mass spectra were recorded on the Bruker MicrOTOF Q II.

II. Reaction Condition Screening

Table S1. Reaction Condition Optimization of Hydroalkoxylation Reaction of Allylic Cyanohydrin and Activated Alkyne [a]



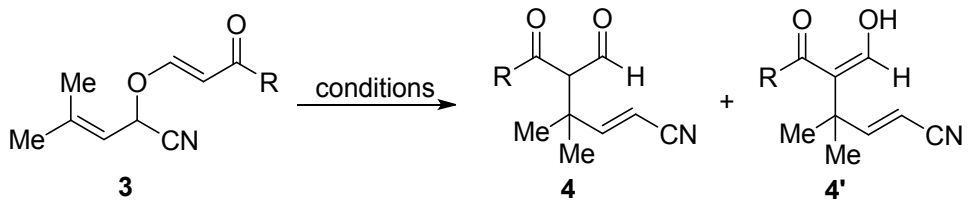
1a **2** **3** **4**

2a: R = $N(CH_2)_4O$
2b: R = OEt

Entry	cat.	solvent	2	*T* (°C)	*t* (h)	Yield (%) [b] 3+4	*E*/*Z* [c] 3	3(*E*)/4 [c]
1	DABCO	CH_3CN	2a	0	12	47 (3a+4a)	3.7/1	8.5/1
2	DABCO	DCM	2a	0	12	30 (3a+4a)	2.5/1	3.9/1
3	DABCO	THF	2a	0	12	71 (3a+4a)	4/1	4.2/1
4	DABCO	MTBE	2a	0	12	50 (3a+4a)	4/1	8.9/1
5	DABCO	Toluene	2a	0	12	73 (3a+4a)	2.8/1	3/1
6	DABCO	DMF	2a	0	12	30 (3a+4a)	6.3/1	3.6/1
7	DABCO	Anisole	2a	0	12	57 (3a+4a)	3/1	10/1
8	DABCO	EtOAc	2a	0	12	60 (3a+4a)	4.4/1	7.3/1
9	DABCO	Acetone	2a	0	12	41 (3a+4a)	16/1	4.6/1
10	DABCO	H_2O	2a	0	12	0 (3a+4a)	--	--
11	DABCO	EtOH	2a	0	12	52 (3a+4a)	3/1	4.9/1
12	DABCO	THF	2a	30	12	62 (3a+4a)	3/1	4.8/1
13	DMAP	THF	2a	30	12	trace	--	--
14	DBU	THF	2a	30	12	trace	--	--
15	PPh_3	THF	2a	30	12	nd	--	--
16	PBu_3	THF	2a	30	12	trace	--	--
17	PMe_3	THF	2a	30	12	trace	-	-
18	TEA	THF	2a	30	12	< 5	--	--
19[d]	DABCO	Toluene	2a	0	12	68 (3a+4a)	6/1	5/1
20	DABCO	Toluene	2b	0	10	75 (3b)	9/1	>19/1

[a] Reaction Conditions: 1 (0.33 mmol), 2 (0.3 mmol), and catalyst (20 mol%) in solvent (c = 0.2 *M*). [b] Calculation based on combining isolated yields of 3(*E*) and 4 with ^{1}HNMR analysis. [c] Determined by ^{1}HNMR analysis. [d] Performed with DABCO (10 mol%).

Table S2. Claisen Rearrangement of Allyl Vinyl Ether 3 [a]



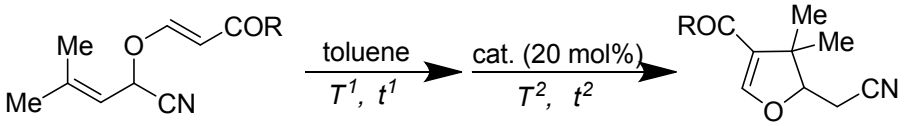
3 **4** **4'**

3b: R = OEt
3a: R = $N(CH_2)_4O$

Entry	conditions	3	Yield (%)[b]	4/4' [c]
1	Toluene, 70 °C, 12 h	3b	85 (4b+4b')	2/1
2	CH_3CN, 70 °C, 12 h	3b	36 (4b+4b')	1.7/1
3	THF, 70 °C, 12 h	3b	29 (4b+4b')	2.4/1
4	Toluene, 90 °C, 12 h	3b	89 (4b+4b')	1.7/1
5	Toluene, 110 °C, 6 h	3b	83 (4b+4b')	1.5/1
6	Toluene, 50 °C, 12 h	3b	70 (4b+4b')	6.7/1
7	Toluene, DABCO (20 mo%), 30 °C, 12 h	3b	trace	-
8[d]	Toluene, 70 °C, 12 h	3a(*E*)	90 (4a)	>19/1
9[b]	Toluene, 70 °C, 12 h	3a(*Z*)	48 (4a)	>19/1

[a] Reactions were performed with 3 (0.2 mmol) (C = 0.2 *M*). [b] Determined by ^{1}HNMR analysis with CH_2Br_2 as internal standard. [c] Determined by ^{1}HNMR analysis. [d] The inseparable mixture of 3a(*E*) and 4a (3a(*E*)/4a = 2.2/1) was employed. The yield was calculated based on 3a(*E*).

Table S3. Claisen Rearrangement/Cyclization of Allyl Vinyl Ether 3 [a]



3
3b: R = OEt
3a: R = $N(CH_2)_4O$

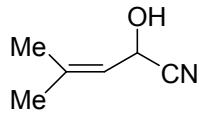
5

Entry	cat.	3	T^1/T^2 (°C)	t^1/t^2 (h)	Yield (%)[b]
1	DBU	3b	70/30	12/5	83
2	DABCO	3b	70/30	12/12	61
3	TEA	3b	70/30	12/5	24
4	DABCO	3b	70/70	12/5	85
5[c]	DABCO	3a(*E*)	70/70	12/6	80

[a] Reactions were performed with 3 (0.2 mmol) in toluene (C = 0.2). [b] Isolated yields. [c] The inseparable mixture of 3a(*E*) and 4a (3a(*E*)/4a = 2.2/1) was employed.

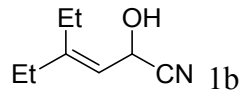
III. Preparation of Substrates and Procedure for Preparation of Compounds 3, 4 and 5 in Reaction Screening

Cyanohydrins 1 [1,2] and activated alkynes 2 [3,4] were prepared according to the reported procedure.



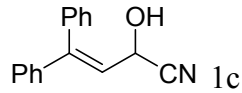
1a

Yellow oi. ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.44-5.38 (m, 1H), 5.12 (d, *J* = 8.6 Hz, 1H), 2.71 (brs, 1H), 1.81 (d, *J* = 1.5 Hz, 3H), 1.77 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.26, 119.53, 119.51, 58.08, 25.68, 18.51.



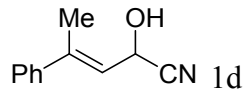
1b

Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.35 (d, *J* = 8.9 Hz, 1H), 5.17 (d, *J* = 8.7 Hz, 1H), 2.67 (brs, 1H), 2.18 – 2.07 (m, 4H), 1.11 – 1.00 (m, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.03, 119.74, 117.50, 57.79, 28.97, 24.32, 13.33, 12.08. HRMS (ESI): calcd. for $C_8H_{13}NNaO$ ($[M+Na]^+$): 162.0889, found 162.0893.



1c

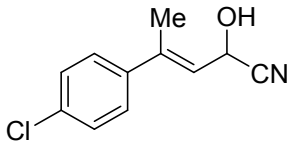
Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 – 7.39 (m, 3H), 7.36 – 7.28 (m, 3H), 7.28 – 7.23 (m, 2H), 7.25 – 7.19 (m, 2H), 6.16 (d, *J* = 9.3 Hz, 1H), 4.96 (dd, *J* = 9.4, 6.0 Hz, 1H), 2.50 – 2.48 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.28, 140.12, 137.54, 129.63, 129.03, 128.89, 128.83, 128.59, 128.04, 121.38, 119.08, 59.39. HRMS (ESI): calcd. for $C_{16}H_{14}NO$ ($[M+H]^+$): 236.1070, found 236.1068.



1d

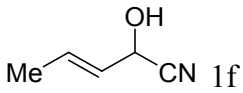
Yellow solid, mp: 52.9-53.1 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.29 (m, 5H), 5.89 (dq, *J* = 8.3, 1.4 Hz, 1H), 5.30 (dd, *J* = 8.3, 6.1 Hz, 1H), 2.90 (d, *J* = 6.2 Hz, 1H), 2.16 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR

(125 MHz, $CDCl_3$) δ 143.63, 141.25, 128.65, 128.58, 126.12, 121.28, 119.17, 58.45, 16.84. HRMS (ESI): calcd. for $C_{11}H_{12}NO$ ($[M+H]^+$): 174.0913, found 174.0914.



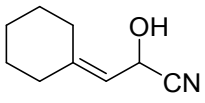
1e

Yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 4H), δ 5.88 (dt, *J* = 8.3, 1.4 Hz, 1H), 5.32-5.29 (m, 1H), 2.64 (d, *J* = 5.5 Hz, 1H), 2.15 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.53, 139.62, 134.58, 128.85, 127.44, 121.75, 118.93, 58.42, 16.84. HRMS (ESI): calcd. for $C_{11}H_{11}ClNO$ ($[M+H]^+$): 208.0524, found 208.0523.



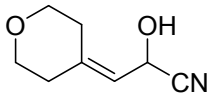
1f

Yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 6.10 (dq, *J* = 13.5, 6.6 Hz, 1H), 5.67-5.62 (m, 1H), 4.93 (t, *J* = 6.7 Hz, 1H), 2.61 – 2.48 (m, 1H), 1.80 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ133.17, 125.21, 118.53, 61.94, 17.65.



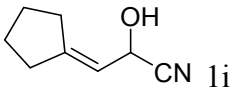
1g

Yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 5.35 (d, *J* = 8.6 Hz, 1H), 5.17 (dd, *J* = 8.6, 5.8 Hz, 1H), 3.07 (d, *J* = 5.9 Hz, 1H), 2.22 – 2.14 (m, 4H), 1.63 – 1.55 (m, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 149.68, 119.77, 116.15, 57.21, 36.74, 29.49, 28.12, 27.54, 26.31. HRMS (ESI): calcd. for $C_9H_{14}NO$ ($[M+H]^+$): 152.1070, found 152.1068.



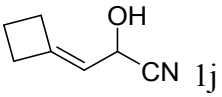
1h

Green oil. ^1H NMR (500 MHz, $CDCl_3$) δ 5.48 (d, *J* = 8.4 Hz, 1H), 5.19-5.14 (m, 1H), 3.76 – 3.72 (m, 4H), 3.06 – 2.82 (m, 1H), 2.38 (t, *J* = 5.7 Hz, 2H), 2.32 – 2.29 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 119.39, 118.16, 118.13, 68.98, 68.17, 56.96, 36.47, 30.35. HRMS (ESI): calcd. for $C_8H_{12}NO_2$ ($[M+H]^+$): 154.0863, found 154.0867.



1i

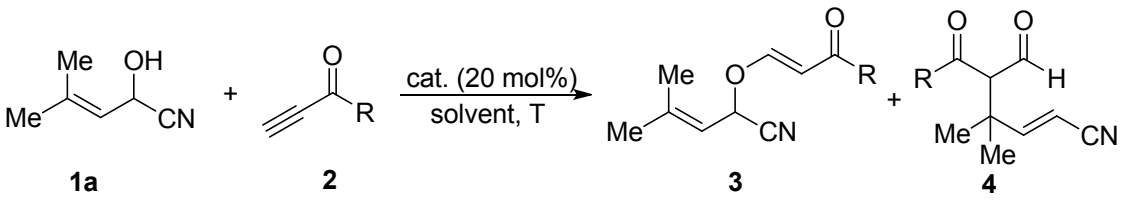
Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.51-5.48 (m, 1H), 5.03 (d, *J* = 8.5 Hz, 1H), 3.06 (brs, 1H), 2.39 – 2.27 (m, 4H), 1.79 – 1.69 (m, 2H), 1.71 – 1.62 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 153.77, 119.46, 114.90, 59.42, 34.09, 29.27, 26.26, 25.99. HRMS (ESI): calcd. for $C_8H_{12}NO$ ($[M+H]^+$): 138.0913, found 138.0912.



1j

Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.34-5.30 (m, 1H), 4.88 (dd, *J* = 8.3, 5.5 Hz, 1H), 3.73 – 3.69 (m, 1H), 2.84 – 2.80 (m, 2H), 2.77 – 2.73 (m, 2H), 2.07 – 2.00 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.23, 119.27, 114.76, 57.97, 31.17, 29.58, 16.88. HRMS (ESI): calcd. for $C_7H_{10}NO$ ($[M+H]^+$): 124.0757, found 124.0758.

Procedure for Preparation of Compounds 3 and 4 (Table S1)

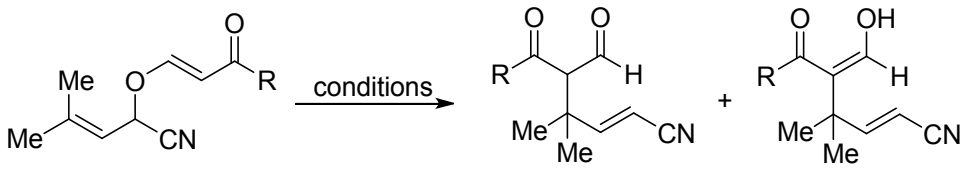


1a **2** **3** **4**

2a: R = $N(CH_2)_4O$
2b: R = OEt

To a solution of compound 1a (0.33 mmol) in solvent (1.5 mL) was added DABCO (20 mol%) at 0 ^{o}C under a N_2 atmosphere. Then 2 (0.3 mmol) was added, and the mixture was stirred at indicated temperature and monitoring by TLC. Upon completion, the reaction was quenched with saturated ammonium chloride solution, and extracted with EtOAc. The combined organic phases were washed with brine dried over Na_2SO_4, and concentrated. The residue was purified by flash column chromatography on a silica gel using petroleum ether (60-90 ^{o}C)/EtOAc as the eluent to give the desired products 3 or 4.

Procedure for Preparation of Compounds 4 (Table S2)



3

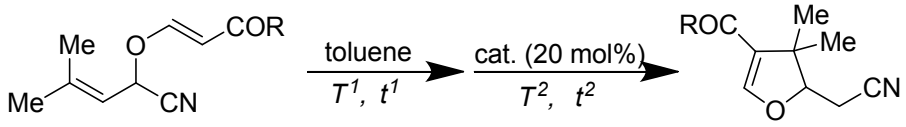
4

4'

3b: R = OEt
3a: R = $N(CH_2)_4O$

A solution of 3 (0.2 mmol) in toluene (1 mL) was stirred at indicated temperature under a N_2 atmosphere and monitoring by TLC. Upon completion, the reaction mixture was concentrated *in vacuo* and the crude mixture was analyzed by ^{1}HNMR to determine the yields of 4.

General Procedure for Preparation of Compounds 5 (Table S3)

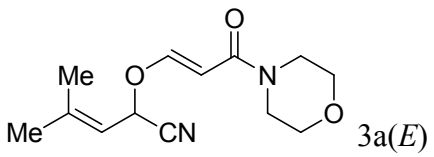


3

5

3b: R = OEt
3a: R = $N(CH_2)_4O$

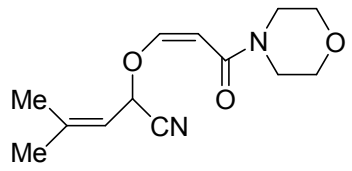
A solution of 3 (0.2 mmol) in toluene (1 mL) was stirred at indicated temperature under a N_2 atmosphere and monitoring by TLC. Upon completion, catalyst (20 mol%) was added. The mixture was stirred at indicated temperature. Upon completion, HCl (1 M) (10 mL) was added, and the resultant mixture was extracted with ethyl acetate (10 mL×3). The combined organic layer was washed with brine and dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by column chromatography (silica gel, EtOAc/Petroleum ether (60-90 ^{o}C) to afford the desired product 5.



3a(*E*)

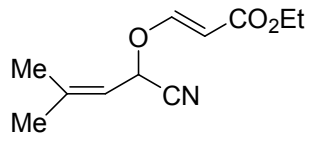
Yellow oil. Inseparable mixture of 3a(*E*) and 4a (3a(*E*)/4a = 2.2/1). 3a(*E*): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 11.7 Hz, 1H), 5.86 (d, *J* = 11.7 Hz, 1H), 5.43 – 5.40 (m, 1H), 5.23 (d, *J* = 8.7 Hz, 1H), 3.74 – 3.60 (m, 8H), 1.86 (s, 3H), 1.80 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.06, 157.67, 145.20, 116.17, 115.95, 99.71, 66.92, 66.83, 65.55, 25.80, 18.84. HRMS (ESI): calcd. for $C_{13}H_{19}N_2O_3$

([M+H]$^+$): 251.1390, found 251.1392.



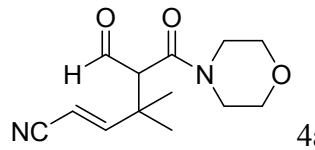
3a(*Z*)

Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.37 (d, *J* = 7.1 Hz, 1H), 5.42-5.39 (m, 1H), 5.27 (d, *J* = 8.7 Hz, 1H), 5.19 (d, *J* = 7.1 Hz, 1H), 3.77 – 3.56 (m, 8H), 1.85 (d, *J* = 1.5 Hz, 3H), 1.79 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.04, 145.96, 145.11, 116.53, 116.04, 102.77, 67.07, 66.88, 66.34, 47.22, 41.96, 25.80, 18.86. HRMS (ESI): calcd. for $C_{13}H_{19}N_2O_3$ ([M+H]$^+$): 251.1390, found 251.1387.



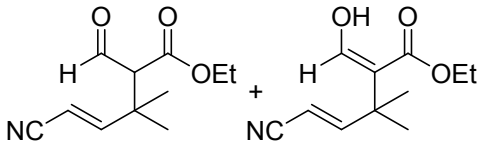
3b

Yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (d, *J* = 12.6 Hz, 1H), 5.42 (d, *J* = 12.7 Hz, 1H), 5.44 – 5.38 (m, 1H), 5.24 (d, *J* = 8.6 Hz, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 1.86 (d, *J* = 1.4 Hz, 3H), 1.80 (d, *J* = 1.4 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.68, 158.12, 145.20, 115.85, 115.80, 101.02, 64.99, 60.39, 25.79, 18.87, 14.40. HRMS (ESI): calcd. for $C_{11}H_{16}NO_3$ ([M+H]$^+$): 210.1125, found 210.1126.



4a

Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.67 (d, *J* = 3.8 Hz, 1H), 7.11 (d, *J* = 16.7 Hz, 1H), 5.33 (d, *J* = 16.7 Hz, 1H), 3.82 – 3.41 (m, 8H), 3.35 (d, *J* = 3.8 Hz, 1H), 1.28 (s, 3H), 1.25 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 198.80, 165.60, 159.87, 117.21, 98.86, 66.88, 66.59, 61.86, 46.95, 42.38, 41.12, 25.44, 24.53. HRMS (ESI): calcd. for $C_{13}H_{19}N_2O_3$ ([M+H]$^+$): 251.1390, found 251.1389.

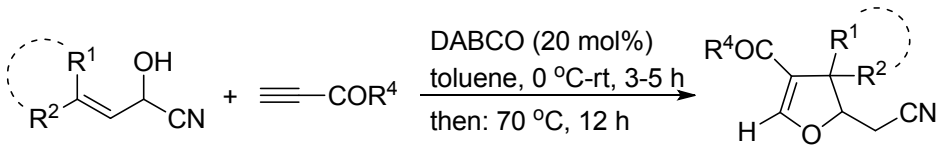


4b 4b’

Yellow oil. Inseparable mixture of 4b/4b’ (4b/4b’ = 2/1). 4b: ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.69 (d, *J* = 3.7 Hz, 1H), 6.90 (d, *J* = 16.7 Hz, 1H), 5.34 (d, *J* = 16.6 Hz, 1H), 4.32 – 4.15 (m, 2H), 3.14 (d, *J* = 3.7 Hz, 1H), 1.39 – 1.20 (m, 9H). 4b’: ^{1}H NMR (500 MHz, $CDCl_3$) δ 12.18 (d, *J* = 12.5 Hz, 1H), 7.24 (d, *J* = 12.4 Hz, 1H), 6.78 (d, *J* = 16.6 Hz, 1H), 5.24 (d, *J* = 16.6 Hz, 1H), 4.32 – 4.15 (m, 2H), 1.39 – 1.20 (m, 9H). HRMS (ESI): calcd. for $C_{11}H_{16}NO_3$ ($[M+H]^+$): 210.1125, found 210.1123.

IV. General Procedure and Experimental Details

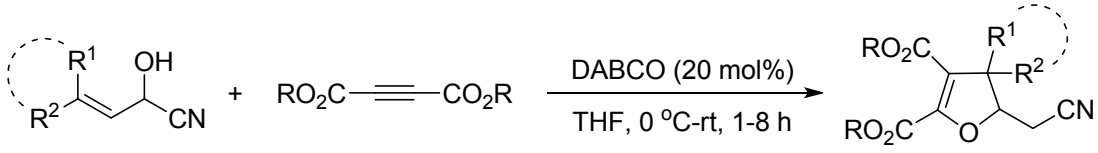
General Procedure for preparation of compounds 5a-5p



1 **2** **5**

To a solution of compound 1 (0.33 mmol) in toluene (1.5 mL) was added DABCO (20 mol%) at 0 °C under a N_2 atmosphere. Then 2 (0.3 mmol) was added and the resultant mixture was stirred at 30 °C and monitoring by TLC. Upon completion of 2, the mixture was warmed to 70 °C. After completion, HCl (1 M) (10 mL) was added, and the resultant mixture was extracted with ethyl acetate (10 mL×3). The combined organic layer was washed with brine and dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by column chromatography (silica gel, EtOAc/Petroleum ether (60-90 °C) to afford the desired product 5.

General Procedure for preparation of compounds 5q-5y

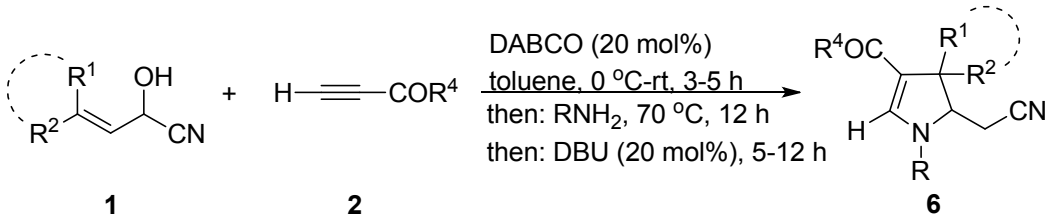


1 **2** **5**

To a solution of compound 1 (0.33 mmol) in THF (1.5 mL) was added DABCO (20 mol%) at 0 °C

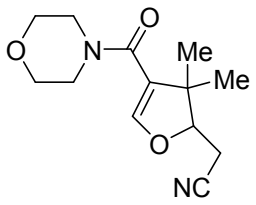
under a N_2 atmosphere. Then 2 (0.3 mmol) was added and the mixture was warmed to 30 °C and monitoring by TLC. Upon completion, HCl (1 M) (10 mL) was added, and the resultant mixture was extracted with ethyl acetate (10 mL×3). The combined organic layer was washed with brine and dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by column chromatography (silica gel, EtOAc/Petroleum ether (60-90 °C) to afford the desired product 5.

General Procedure for preparation of compounds 6a-6l



1 **2** **6**

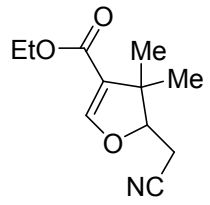
To a solution of compound 1 (0.55 mmol) in toluene (2.5 mL) was added DABCO (20 mol%) at 0 °C under a N_2 atmosphere. Then 2 (0.5 mmol) was added, and keeping the reaction at 30 °C and monitoring by TLC. Upon completion, RNH_2 (0.55 mmol) was added, and the mixture was stirred at 70 °C. After 12h, DBU (20 mol%) was added. Upon completion, HCl (1 M) (10 mL) was added to the cooled mixture, and the resultant mixture was extracted with ethyl acetate (10 mL×3). The combined organic layer was washed with brine and dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by column chromatography (silica gel, EtOAc/Petroleum ether (60-90 °C) to afford the desired product 6.



5a

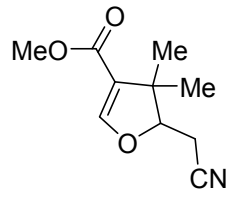
White solid (25 mg, 33% yield). mp: 79.8-80.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.49 (s, 1H), 4.36 (t, J = 6.2 Hz, 1H), 3.66 (s, 8H), 2.69-2.67 (m, 2H), 1.37 (s, 3H), 1.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.60, 146.47, 117.68, 116.93, 86.45, 66.94, 47.06, 45.30, 26.89, 20.27, 18.76. HRMS (ESI): calcd. for $C_{13}H_{19}N_2O_3$ ($[M+H]^+$): 251.1390, found 251.1389. HRMS (ESI): calcd. for

$C_{13}H_{19}N_2O_3$ ([M+H]$^+$): 251.1390, found 251.1389.



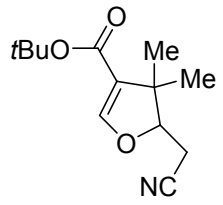
5b

Yellow oil (49 mg, 78% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17 (s, 1H), 4.48 (dd, *J* = 7.5, 6.0 Hz, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 2.85 – 2.58 (m, 2H), 1.39 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.25 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.20, 155.21, 118.16, 116.61, 88.51, 59.85, 44.37, 26.84, 20.50, 18.90, 14.40. HRMS (ESI): calcd. for $C_{11}H_{16}NO_3$ ([M+H]$^+$): 210.1125, found 210.1123.



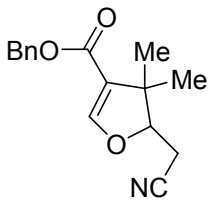
5c

Yellow oil (51 mg, 87% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.17 (s, 1H), 4.48 (dd, *J* = 7.6, 5.9 Hz, 1H), 3.71 (s, 3H), 2.73 (dd, *J* = 16.9, 7.7 Hz, 1H), 2.67 (dd, *J* = 16.8, 5.8 Hz, 1H), 1.39 (s, 3H), 1.25 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.55, 155.36, 117.87, 116.57, 88.50, 51.04, 44.34, 26.80, 20.46, 18.89. HRMS (ESI): calcd. for $C_{10}H_{13}NNaO_3$ ([M+Na]$^+$): 218.0788, found 218.0790.



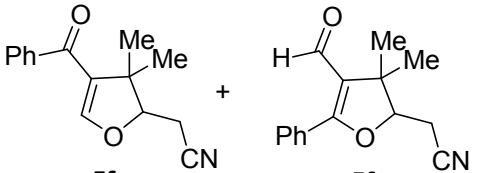
5d

Yellow oil (60 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.08 (s, 1H), 4.45 (dd, *J* = 7.5, 6.1 Hz, 1H), 2.72 (dd, *J* = 16.8, 7.8 Hz, 1H), 2.65 (dd, *J* = 16.8, 5.9 Hz, 1H), 1.48 (s, 9H), 1.37 (s, 3H), 1.22 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.67, 154.73, 119.54, 116.69, 88.43, 80.43, 44.34, 28.44, 26.82, 20.51, 18.88. HRMS (ESI): calcd. for $C_{13}H_{19}NNaO_3$ ([M+Na]$^+$): 260.1257, found 260.1259.



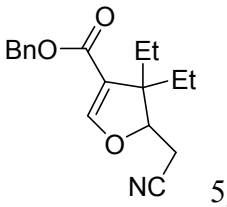
5e

Yellow oil (60 mg, 74% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.28 (m, 5H), 7.20 (s, 1H), 5.15 (s, 2H), 4.46 (dd, *J* = 7.8, 5.8 Hz, 1H), 2.69 (dd, *J* = 16.8, 7.7 Hz, 1H), 2.63 (dd, *J* = 16.9, 5.8 Hz, 1H), 1.38 (s, 3H), 1.23 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.80, 155.64, 136.15, 128.54, 128.11, 128.00, 117.69, 116.55, 88.47, 65.51, 44.21, 26.69, 20.38, 18.72. HRMS (ESI): calcd. for $C_{16}H_{18}NO_3$ ($[M+H]^+$): 272.1281, found 272.1279.



5fα **5fβ**

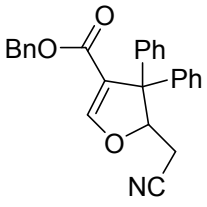
Yellow oil (65 mg, 90% yield), inseparable mixture, 5fα/5fβ = 1/4. Compound 5fα: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 – 7.50 (m, 5H), 6.99 (s, 1H), 4.55 (dd, *J* = 7.4, 5.9 Hz, 1H), 2.76 – 2.70 (m, 2H), 1.50 (s, 3H), 1.39 (s, 3H). Compound 5fβ: ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.74 (s, 1H), 7.65 – 7.59 (m, 3H), 7.50 – 7.46 (m, 2H), 4.58 (dd, *J* = 7.7, 5.6 Hz, 1H), 2.81 (dd, *J* = 16.9, 7.7 Hz, 1H), 2.75 (dd, *J* = 16.8, 5.9 Hz, 1H), 1.50 (s, 3H), 1.36 (s, 3H). The mixture of 5fα/5fβ: ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.17, 187.07, 170.97, 158.83, 140.25, 131.88, 131.80, 129.45, 128.82, 128.48, 128.33, 128.08, 126.38, 122.95, 116.57, 116.55, 88.84, 87.05, 46.08, 45.74, 26.45, 26.43, 20.26, 19.98, 18.90, 18.83. HRMS (ESI): calcd. for: $C_{15}H_{16}NO_2$ ($[M+H]^+$): 242.1176, found 242.1171.



5g

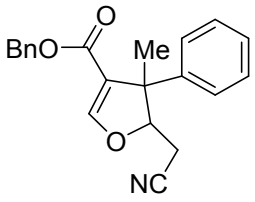
Yellow oil (79 mg, 88% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.27 (m, 6H), 5.16 (s, 2H), 4.64 (dd, *J* = 9.1, 4.9 Hz, 1H), 2.80 (dd, *J* = 16.9, 9.1 Hz, 1H), 2.68 (dd, *J* = 16.9, 5.0 Hz, 1H), 1.93-1.89 (m, 1H), 1.69-1.64 (m, 2H), 1.61-1.55 (m, 1H), 0.87-0.83 (m, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ

164.28, 156.89, 136.33, 128.64, 128.21, 128.01, 116.84, 113.84, 85.70, 65.63, 51.79, 29.38, 26.13, 18.94, 10.00, 9.11. HRMS (ESI): calcd. for $C_{18}H_{21}NNaO_3$ ([M+Na]$^+$): 322.1414, found 322.1417.



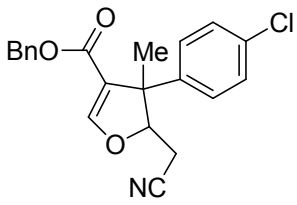
5h

Yellow oil (42 mg, 35% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (s, 1H), 7.32 – 7.30 (m, 2H), 7.26-7.20 (m, 9H), 7.11-7.09 (m, 2H), 7.06-7.04 (m, 2H), 5.52 (dd, *J* = 9.8, 3.4 Hz, 1H), 5.01 (d, *J* = 12.5 Hz, 1H), 4.94 (d, *J* = 12.4 Hz, 1H), 2.16 (dd, *J* = 17.0, 9.8 Hz, 1H), 1.85 (dd, *J* = 17.0, 3.3 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.46, 156.75, 144.62, 137.94, 135.95, 129.83, 128.55, 128.49, 128.34, 128.21, 128.19, 127.96, 127.86, 127.53, 116.82, 116.67, 89.83, 65.97, 62.13, 22.59. HRMS (ESI): calcd. for $C_{26}H_{22}NO_3$ ([M+H]$^+$): 396.1594, found 396.1592.



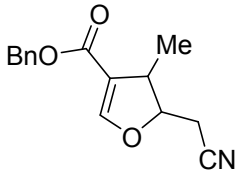
5i

Yellow oil (80 mg, 80% yield, dr = 2/1). The major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (s, 1H), 7.39 – 7.21 (m, 7H), 7.12-7.08 (m, 2H), 5.12 (d, *J* = 12.6 Hz, 1H), 5.02 (d, *J* = 12.6 Hz, 1H), 4.72 (dd, *J* = 8.6, 5.4 Hz, 1H), 2.18 (dd, *J* = 17.0, 8.6 Hz, 1H), 2.00 (dd, *J* = 16.9, 5.4 Hz, 1H), 1.88 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.64, 156.99, 138.83, 136.13, 129.00, 128.56, 128.10, 127.87, 127.81, 127.14, 118.26, 116.33, 89.84, 65.70, 51.81, 25.36, 20.54. HRMS (ESI): calcd. for $C_{21}H_{20}NO_3$ ([M+H]$^+$): 334.1438, found 334.1439. The minor isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (s, 1H), 7.40 – 7.21 (m, 7H), 7.10-7.07 (m, 2H), 5.10 (d, *J* = 12.6 Hz, 1H), 5.98 (d, *J* = 12.6 Hz, 1H), 4.79 (dd, *J* = 8.2, 5.0 Hz, 1H), 2.75 (dd, *J* = 16.9, 8.2 Hz, 1H), 2.67 (dd, *J* = 16.9, 5.0 Hz, 1H), 1.67 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.52, 156.87, 144.20, 136.04, 128.93, 128.53, 128.08, 127.84, 127.35, 126.24, 118.38, 116.34, 90.91, 65.71, 51.84, 18.79, 18.23. HRMS (ESI): calcd. for $C_{21}H_{20}NO_3$ ([M+H]$^+$): 334.1438, found 334.1430.



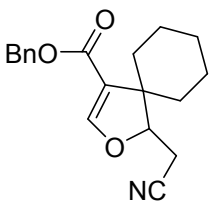
5j

Yellow oil (90 mg, 82% yield, dr = 2.4/1) The major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (s, 1H), 7.36 – 7.26 (m, 5H), 7.19 – 7.05 (m, 4H), 5.13 (d, *J* = 12.6 Hz, 1H), 5.00 (d, *J* = 12.5 Hz, 1H), 4.70 (dd, *J* = 8.2, 5.8 Hz, 1H), 2.22 (dd, *J* = 16.9, 8.2 Hz, 1H), 2.01 (dd, *J* = 16.9, 5.9 Hz, 1H), 1.86 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.37, 157.05, 137.43, 135.94, 133.89, 129.10, 128.64, 128.55, 128.19, 127.87, 118.10, 115.96, 89.44, 65.76, 51.43, 25.32, 20.40. HRMS (ESI): calcd. for $C_{21}H_{19}ClNO_3$ ($[M+H]^+$): 368.1048, found 368.1049. The minor isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (s, 1H), 7.35 – 7.23 (m, 7H), 7.11-7.07 (m, 2H), 5.12 (d, *J* = 12.4 Hz, 1H), 4.97 (d, *J* = 12.5 Hz, 1H), 4.76 (dd, *J* = 7.9, 5.5 Hz, 1H), 2.76 (dd, *J* = 16.9, 7.9 Hz, 1H), 2.68 (dd, *J* = 16.9, 5.5 Hz, 1H), 1.67 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.32, 156.97, 142.81, 135.89, 133.40, 129.07, 128.59, 128.25, 127.99, 127.78, 118.26, 116.06, 90.68, 65.86, 51.54, 18.79, 18.23. HRMS (ESI): calcd. for $C_{21}H_{19}ClNO_3$ ($[M+H]^+$): 368.1048, found 368.1050.



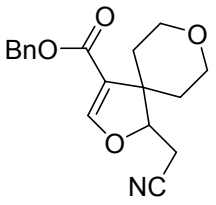
5k

Yellow oil (52 mg, 68% yield), dr = 11/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 – 7.29 (m, 5H), 7.21 (d, *J* = 1.5 Hz, 1H), 5.20 (d, *J* = 12.4 Hz, 1H), 5.15 (d, *J* = 12.4 Hz, 1H), 4.50 (dd, *J* = 11.5 Hz, *J* = 6.0 Hz, 1H), 3.08 – 3.00 (m, 1H), 2.71-2.62 (m, 2H), 1.31 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.22, 155.63, 136.18, 128.67, 128.30, 128.23, 115.85, 113.89, 86.45, 65.90, 41.18, 23.50, 19.58. HRMS (ESI): calcd. for: $C_{15}H_{15}NNaO_3$ ($[M+Na]^+$): 280.0944, found 280.0945.



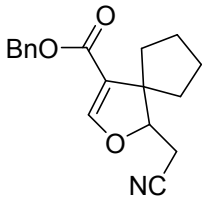
5l

Yellow oil (69 mg, 74% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.29 (m, 5H), 7.22 (s, 1H), 5.15 (s, 2H), 4.81 (t, *J* = 6.2 Hz, 1H), 2.66 (d, *J* = 6.4 Hz, 2H), 1.87 (td, *J* = 13.3, 3.9 Hz, 1H), 1.90 – 1.84 (m, 1H), 1.77 – 1.52 (m, 5H), 1.35 – 1.24 (m, 2H), 1.04-0.96 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.01, 155.75, 136.29, 128.68, 128.25, 128.16, 117.26, 116.62, 85.46, 65.65, 48.34, 34.84, 27.26, 24.92, 24.40, 22.51, 19.82. HRMS (ESI): calcd. for: $C_{19}H_{21}NNaO_3$ ($[M+Na]^+$): 334.1414, found 341.1409.



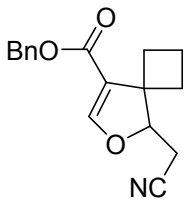
5m

Yellow oil (75 mg, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.30 (m, 5H), 7.27 (s, 1H), 5.17 (s, 2H), 4.93 (t, *J* = 6.1 Hz, 1H), 4.01 – 3.90 (m, 2H), 3.51 (td, *J* = 12.4, 2.3 Hz, 1H), 3.24 (td, *J* = 12.5, 2.1 Hz, 1H), 2.83-2.77 (m, 5.0 Hz, 1H), 2.66 (d, *J* = 6.1 Hz, 2H), 2.28 – 2.19 (m, 1H), 1.53-1.49 (m, 1H), 1.44-1.41 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.61, 156.29, 136.09, 128.67, 128.30, 128.20, 116.64, 115.44, 84.93, 65.79, 65.71, 64.05, 46.18, 34.65, 27.72, 19.98. HRMS (ESI): calcd. for $C_{18}H_{19}NNaO_4$ ($[M+H]^+$): 336.1206, found 336.1207.



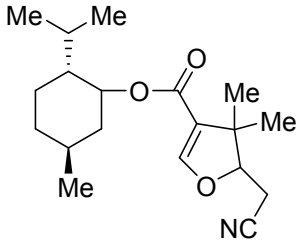
5n

Yellow oil (63 mg, 71% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 5H), 7.24 (s, 1H), 5.16 (s, 2H), 4.54 (dd, *J* = 7.6, 5.1 Hz, 1H), 2.72 – 2.57 (m, 2H), 2.24 – 2.04 (m, 2H), 1.85-1.80 (m, 2H), 1.72-1.67 (m, 1H), 1.62 – 1.54 (m, 2H), 1.54 – 1.42 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.93, 155.89, 136.27, 128.68, 128.28, 128.21, 116.73, 115.58, 89.04, 65.69, 54.81, 39.22, 30.18, 25.76, 24.65, 20.04. HRMS (ESI): calcd. for $C_{18}H_{20}NO_3$ ($[M+H]^+$): 298.1438, found 298.1439.



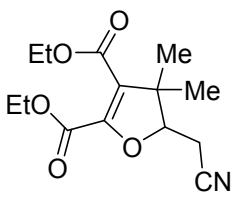
5o

Yellow oil (55 mg, 69% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.30 (m, 5H), 7.19 (s, 1H), 5.23 (d, *J* = 12.5 Hz, 1H), 5.20 (d, *J* = 12.5 Hz, 1H), 4.77 (dd, *J* = 6.9, 5.3 Hz, 1H), 2.83 (dd, *J* = 16.8, 5.4 Hz, 1H), 2.79 – 2.71 (m, 2H), 2.70 – 2.61 (m, 1H), 2.26 – 2.10 (m, 2H), 2.02-2.00 (m, 1H), 1.87 – 1.74 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.23, 155.77, 136.29, 128.72, 128.33, 128.26, 116.34, 115.66, 89.01, 65.80, 49.73, 34.45, 27.05, 20.78, 15.71. HRMS(ESI): calcd. for $C_{17}H_{17}NNaO_3$ ([M+Na]$^+$): 306.1101, found 306.1102.



5p

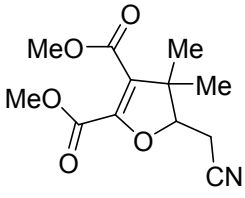
Yellow oil (71 mg, 74% yield), inseparable mixture of diastereoisomers, dr =1/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.15 (s, 1H), 7.14 (s, 1H), 4.78 – 4.72 (m, 2H), 4.50 – 4.45 (m, 2H), 2.73 (ddd, *J* = 16.8, 7.7, 1.9 Hz, 2H), 2.66 (ddd, *J* = 16.8, 5.9, 1.1 Hz, 2H), 2.07 – 1.99 (m, 2H), 1.89 – 1.83 (m, 4H), 1.72 – 1.66 (m, 3H), 1.53 – 1.46 (m, 2H), 1.39 (s, 3H), 1.38 (s, 3H), 1.24 (s, 3H), 1.23 (s, 3H), 1.12 – 0.95 (m, 3H), 0.93 – 0.88 (m, 16H), 0.78 (d, *J* = 7.0 Hz, 3H), 0.77 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.92, 155.11, 155.06, 118.60, 118.58, 116.63, 73.75, 73.71, 47.39, 47.37, 44.42, 41.26, 34.41, 31.55, 26.92, 26.89, 26.60, 23.67, 22.16, 20.93, 20.64, 20.63, 18.93, 18.89, 16.55. HRMS (ESI): calcd. for: $C_{19}H_{30}NO_3$ ([M+H]$^+$): 320.2221, found 320.2200.



5q

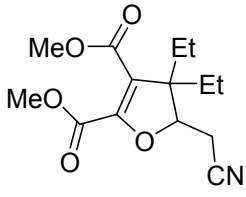
Yellow oil (73 mg, 86% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.53 (t, *J* = 6.9 Hz, 1H), 4.32 (q, *J* = 7.2 Hz, 2H), 4.21 (q, *J* = 7.2 Hz, 2H), 2.82 (dd, *J* = 16.8, 7.1 Hz, 1H), 2.72 (dd, *J* = 16.9, 6.7 Hz, 1H),

1.42 (s, 3H), 1.35 (t, J = 7.1 Hz, 3H), 1.30 (s, 3H), 1.28 (t, J = 7.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.17, 160.34, 151.63, 118.49, 116.09, 87.53, 62.44, 60.80, 46.88, 26.07, 20.33, 18.45, 14.15, 14.04. HRMS(ESI): calcd. for $C_{14}H_{19}NNaO_5$ ([M+Na]$^+$): 304.1155, found 304.1152.



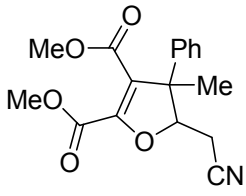
5r

Colourless oil (70 mg, 92% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.54 (t, J = 6.9 Hz, 1H), 3.87 (s, 3H), 3.76 (s, 3H), 2.81 (dd, J = 16.8, 7.0 Hz, 1H), 2.71 (dd, J = 16.9, 6.8 Hz, 1H), 1.41 (s, 3H), 1.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.55, 160.56, 151.42, 118.87, 116.03, 87.55, 53.00, 51.86, 46.88, 26.03, 20.25, 18.42. HRMS (ESI): calcd. for: $C_{12}H_{15}NNaO_5$ ([M+Na]$^+$): 276.0842 , found 276.0841.



5s

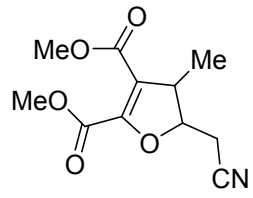
Yellow oil (70 mg, 83% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.72 (dd, J = 8.1, 5.9 Hz, 1H), 3.87 (s, 3H), 3.75 (s, 3H), 2.88 (dd, J = 16.9, 8.1 Hz, 1H), 2.74 (dd, J = 16.9, 5.9 Hz, 1H), 1.91-1.83 (m, 1H), 1.78 – 1.60 (m, 3H), 0.92 (t, J = 7.5, 3H), 0.91 (t, J = 7.5, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.12, 160.53, 152.20, 116.33, 115.87, 84.62, 54.59, 53.04, 52.02, 29.49, 26.58, 18.63, 9.69, 9.12. HRMS(ESI): calcd. for $C_{14}H_{19}NNaO_5$ ([M+Na]$^+$): 304.1155, found 304.1152.



5t

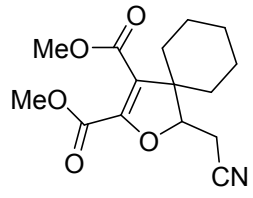
Yellow oil (74 mg, 78% yield, dr = 1.7/1), the inseparable mixture. The major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (t, J = 7.6 Hz, 2H), 7.34-7.31 (m, 1H), 7.26 (d, J = 7.1 Hz, 2H), 4.76 (dd, J = 7.8, 6.2 Hz, 1H), 3.94 (s, 3H), 3.62 (s, 3H), 2.28 (dd, J = 16.9, 7.8, 1H), 2.03 (dd, J = 16.9, 6.2 Hz, 1H), 1.90 (s, 3H). The minor isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 5H), 4.83 (dd, J = 7.8,

5.4 Hz, 1H), 3.91 (s, 3H), 3.59 (s, 3H), 2.81 (dd, J = 16.9, 7.8 Hz, 1H), 2.71 (dd, J = 16.9, 5.4 Hz, 1H), 1.74 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.19, 163.11, 160.74, 160.40, 153.95, 152.83, 142.60, 137.77, 129.13, 129.04, 128.22, 127.77, 126.97, 126.32, 119.07, 117.79, 115.84, 115.81, 89.76, 88.82, 54.46, 53.68, 53.24, 53.16, 51.91, 24.52, 19.90, 18.30, 17.80. HRMS(ESI): calcd. for $C_{17}H_{17}NNaO_5$ ($[M+Na]^+$): 338.0999, found 338.0998.



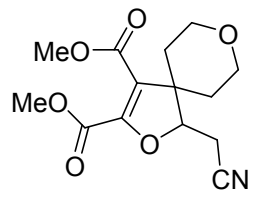
5u

Yellow oil (52 mg, 73% yield), dr = 10/1. ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.55 (q, J = 6.0 Hz, 1H), 3.89 (s, 3H), 3.76 (s, 3H), 3.24-3.19 (m, 1H), 2.80-2.70 (m, 2H), 1.35 (d, J = 6.8 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.39, 160.40, 152.35, 115.43, 113.84, 85.24, 53.08, 51.92, 43.28, 23.22, 19.03. HRMS (ESI): calcd. for $C_{11}H_{14}NO_5$ ($[M+H]^+$): 240.0866, found 240.0863.



5v

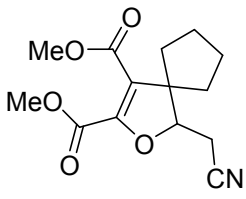
Yellow oil (70 mg, 79% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.82 (dd, J = 8.0, 4.2 Hz, 1H), 3.86 (s, 3H), 3.76 (s, 3H), 2.73 (dd, J = 16.8, 7.9 Hz, 1H), 2.69 (dd, J = 16.9, 4.3 Hz, 1H), 2.07 – 1.98 (m, 1H), 1.92-1.86 (m, 1H), 1.79 – 1.71 (m, 2H), 1.70 – 1.60 (m, 3H), 1.45 – 1.22 (m, 2H), 1.14 – 1.02 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.09, 160.44, 149.89, 119.14, 116.75, 84.60, 52.99, 52.07, 51.16, 34.36, 27.65, 24.81, 23.87, 22.32, 19.40. HRMS(ESI): calcd. for $C_{15}H_{19}NNaO_5$ ($[M+Na]^+$): 316.1155, found 316.1162.



5w

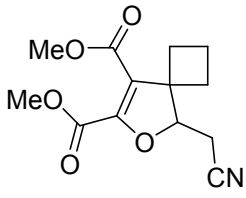
Yellow oil (67 mg, 76% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.91 (dd, J = 6.8, 5.3 Hz, 1H), 3.95-3.90 (m, 2H), 3.87 (s, 3H), 3.78 (s, 3H), 3.62 – 3.54 (m, 1H), 3.35 (td, J = 11.9, 2.4 Hz, 1H), 2.74-2.72

(m, 2H), 2.41-2.40 (m, 1H), 2.27-2.21 (m, 1H), 1.69 – 1.58 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.75, 160.16, 150.61, 117.66, 116.21, 84.33, 65.17, 63.94, 53.08, 52.21, 48.84, 34.19, 28.08, 19.52. HRMS(ESI): calcd. for $C_{14}H_{17}NNaO_6$ ([M+Na]$^+$): 318.0948, found 318.0949.



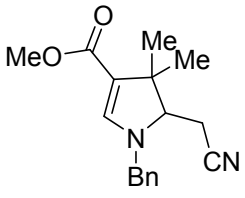
5x

Yellow oil (61 mg, 73% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.60 (dd, *J* = 7.1, 5.6 Hz, 1H), 3.86 (s, 3H), 3.76 (s, 3H), 2.76 (dd, *J* = 16.9, 7.1 Hz, 1H), 2.70 (dd, *J* = 16.9, 5.6 Hz, 1H), 2.25 – 2.17 (m, 1H), 2.04-1.97 (m, 1H), 1.90 – 1.74 (m, 3H), 1.73 – 1.61 (m, 2H), 1.61 – 1.49 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.97, 160.51, 150.41, 118.14, 116.24, 87.71, 57.53, 53.04, 52.07, 38.93, 30.60, 25.83, 24.67, 19.61. HRMS(ESI): HRMS(ESI): calcd. for $C_{14}H_{17}NNaO_5$ ([M+Na]$^+$): 302.0999, found 302.1001.



5y

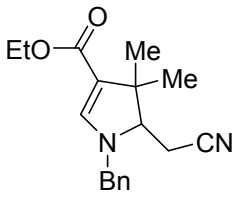
Yellow oil (67 mg, 84% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.79 (t, *J* = 6.1 Hz, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 2.86-2.83 (m, 2H), 2.79 – 2.66 (m, 1H), 2.65 – 2.52 (m, 1H), 2.47 – 2.18 (m, 1H), 2.19 – 1.99 (m, 2H), 1.96 – 1.80 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.07, 160.50, 150.39, 117.17, 115.91, 87.41, 53.02, 52.34, 52.12, 33.93, 27.17, 20.35, 15.68. HRMS(ESI): calcd. for $C_{13}H_{15}NNaO_5$ ([M+Na]$^+$): 288.0842, found 288.0840.



6a

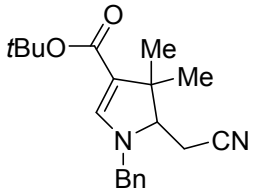
Yellow oil (87 mg, 61% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.31 (m, 3H), 7.29 – 7.25 (m, 2H), 7.05 (s, 1H), 4.40 (d, *J* = 15.0 Hz, 1H), 4.22 (d, *J* = 15.0 Hz, 1H), 3.65 (s, 3H), 3.33 (t, *J* = 6.2 Hz, 1H), 2.54 (d, *J* = 6.7 Hz, 2H), 1.28 (s, 3H), 1.26 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.74,

149.54, 135.70, 129.12, 128.36, 128.20, 117.81, 110.86, 68.48, 52.79, 50.43, 45.43, 27.93, 20.45, 16.78. HRMS (ESI): calcd. for $C_{17}H_{21}N_2O_2$ ([M+H]$^+$): 285.1598, found 285.1599.



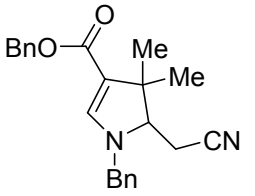
6b

Yellow oil (72 mg, 48% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.22 (m, 5H), 7.06 (s, 1H), 4.39 (d, *J* = 15.1 Hz, 1H), 4.23 (d, *J* = 1.7 Hz, 1H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.32 (t, *J* = 6.1 Hz, 1H), 2.53 (d, *J* = 6.3 Hz, 2H), 1.28 (s, 3H), 1.26 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.34, 149.37, 135.71, 128.99, 128.20, 128.08, 117.76, 111.05, 68.42, 58.88, 52.64, 45.32, 27.86, 20.35, 16.64, 14.53. HRMS (ESI): calcd. for $C_{18}H_{23}N_2O_2$ ([M+H]$^+$): 299.1754, found 299.1755.



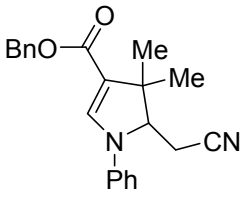
6c

Yellow oil (70 mg, 43% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.34 (m, 2H), 7.36 – 7.30 (m, 1H), 7.28-7.24 (m, 2H), 6.96 (s, 1H), 4.35 (d, *J* = 15.0 Hz, 1H), 4.20 (d, *J* = 15.0 Hz, 1H), 3.30 (t, *J* = 6.2 Hz, 1H), 2.52 (d, *J* = 6.2 Hz, 2H), 1.47 (s, 9H), 1.27 (s, 3H), 1.23 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.25, 149.21, 135.98, 129.08, 128.27, 128.23, 117.93, 113.21, 79.08, 68.89, 52.99, 45.45, 28.68, 27.92, 20.49, 16.77. HRMS (ESI): calcd. for $C_{20}H_{27}N_2O_2$ ([M+H]$^+$): 327.2067, found 327.2063.



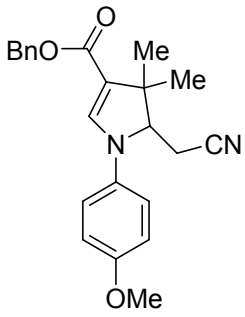
6d

Yellow oil (88 mg, 49% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.28 (m, 10H), 7.12 (s, 1H), 5.16-5.10 (m, 2H), 4.40 (d, *J* = 15.1 Hz, 1H), 4.24 (d, *J* = 15.1 Hz, 1H), 3.36-3.34 (m, 1H), 2.53-2.48 (m, 2H), 1.29 (s, 3H), 1.28 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.08, 149.97, 137.13, 135.60, 129.04, 128.50, 128.27, 128.04, 127.99, 127.85, 117.72, 110.40, 68.35, 64.85, 52.60, 45.33, 27.96, 20.45, 16.66. HRMS (ESI): calcd. for $C_{23}H_{25}N_2O_2$ ([M+H]$^+$): 361.1911, found 361.1913.



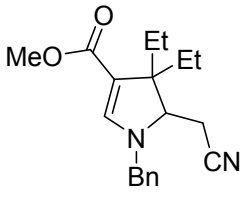
6e

White solid (69 mg, 40% yield). mp: 116.7-117.2 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (s, 1H), 7.41 – 7.26 (m, 7H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 2H), 5.22 – 5.05 (m, 2H), 4.19 (dd, *J* = 8.1, 3.0 Hz, 1H), 2.67 (dd, *J* = 17.2, 8.1 Hz, 1H), 2.59 (dd, *J* = 17.3, 3.1 Hz, 1H), 1.56 (s, 3H), 1.41 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.03, 142.40, 139.83, 136.89, 130.09, 128.62, 128.19, 128.06, 123.21, 117.33, 116.98, 113.92, 68.05, 65.31, 45.15, 29.24, 20.25, 16.52. HRMS (ESI): calcd. for: $C_{22}H_{23}N_2O_2$ ([M+H]$^+$): 347.1754, found 347.1752.



6f

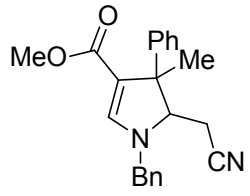
Yellow oil (79 mg, 42% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 – 7.27 (m, 6H), 6.97 (d, *J* = 9.0 Hz, 2H), 6.90 (d, *J* = 9.0 Hz, 2H), 5.19 (d, *J* = 12.5 Hz, 1H), 5.14 (d, *J* = 12.5 Hz, 1H), 4.08 (dd, *J* = 7.7, 3.7 Hz, 1H), 3.79 (s, 3H), 2.61 (dd, *J* = 17.2, 7.7 Hz, 1H), 2.56 (dd, *J* = 17.2, 3.7 Hz, 1H), 1.51 (s, 3H), 1.43 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.20, 156.60, 144.60, 137.06, 133.46, 128.62, 128.17, 128.02, 120.45, 117.38, 115.31, 112.76, 69.28, 65.18, 55.72, 45.10, 29.01, 20.43, 16.62. HRMS (ESI): calcd. for $C_{23}H_{25}N_2O_3$ ([M+H]$^+$): 377.1860, found 377.1861.



6g

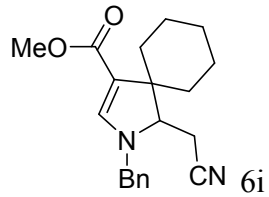
Yellow oil (87 mg, 56% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.25 (m, 5H), 7.16 (s, 1H), 4.37 (d, *J* = 15.0 Hz, 1H), 4.21 (d, *J* = 15.0 Hz, 1H), 3.65 (s, 3H), 3.59 (t, *J* = 6.6 Hz, 1H), 2.60 (d, *J* = 6.6 Hz, 2H), 1.94-1.88 (m, 1H), 1.76 -1.66 (m, 2H), 1.42-1.34 (m, 1H), 0.88 (t, *J* = 7.4 Hz, 3H), 0.69 (t, *J*

= 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.99, 151.54, 135.49, 129.05, 128.35, 118.20, 106.47, 64.41, 53.26, 53.07, 50.52, 30.35, 27.10, 16.45, 10.00, 9.71. HRMS (ESI): calcd. for $C_{19}H_{25}N_2O_2$ ($[M+H]^+$): 313.1911, found 313.1912.



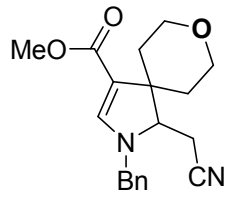
6h

Yellow oil (99 mg, 57% yield, dr = 2/1). The major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 3H), 7.30 – 7.26 (m, 7H), 7.21 – 7.16 (m, 1H), 4.46 (d, J = 14.9 Hz, 1H), 4.22 (d, J = 14.9 Hz, 1H), 3.66 (t, *J* = 5.8 Hz, 1H), 3.53 (s, 3H), 2.61 – 2.51 (m, 2H), 1.69 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.24, 150.50, 146.54, 135.33, 129.07, 128.47, 128.39, 128.30, 126.70, 126.37, 117.63, 110.67, 70.80, 52.89, 52.66, 50.52, 18.05, 16.50. HRMS (ESI): calcd. for $C_{22}H_{23}N_2O_2$ ($[M+H]^+$): 347.1754, found 347.1755. The minor isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 2H), 7.37 – 7.33 (m, 3H), 7.33 – 7.27 (m, 5H), 7.26 – 7.23 (m, 1H), 4.45 (d, *J* = 15.1 Hz, 1H), 4.26 (d, *J* = 15.0 Hz, 1H), 3.60 (t, *J* = 6.4 Hz, 1H), 3.55 (s, 3H), 2.07 (dd, *J* = 17.1, 6.2 Hz, 1H), 1.89 (dd, *J* = 17.0, 6.6 Hz, 1H), 1.75 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.47, 150.98, 140.14, 135.54, 129.18, 128.63, 128.44, 128.26, 127.48, 127.36, 117.59, 111.78, 69.65, 53.02, 52.99, 50.60, 26.68, 18.76. HRMS (ESI): calcd. for $C_{22}H_{23}N_2O_2$ ($[M+H]^+$): 347.1754, found 347.1751.



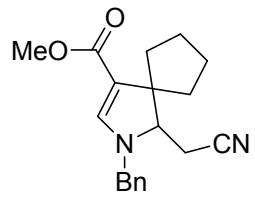
6i

Yellow oil (84 mg, 52% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 5H), 7.05 (s, 1H), 4.50 (s, 2H), 3.68 (t, *J* = 5.4 Hz, 1H), 3.61 (s, 3H), 2.58 – 2.53 (m, 2H), 2.51 – 2.39 (m, 1H), 1.75 – 1.67 (m, 1H), 1.68 – 1.53 (m, 3H), 1.50 – 1.42 (m, 1H), 1.29 – 1.10 (m, 3H), 0.99 – 0.86 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.80, 148.47, 136.10, 129.02, 128.33, 128.29, 118.74, 108.12, 63.15, 52.83, 50.19, 49.41, 34.24, 27.61, 25.17, 24.64, 21.84, 17.01. HRMS (ESI): calcd. for $C_{20}H_{25}N_2O_2$ ($[M+H]^+$): 325.1911, found 325.1913.



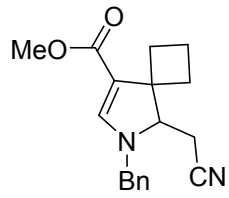
6j

Yellow oil (86 mg, 53% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 5H), 7.13 (s, 1H), 4.53 (d, *J* = 14.8 Hz, 1H), 4.48 (d, *J* = 14.8 Hz, 1H), 3.96 – 3.92 (m, 1H), 3.78 (t, *J* = 5.4 Hz, 1H), 3.75 – 3.70 (m, 1H), 3.65 (s, 3H), 3.31 – 3.26 (m, 1H), 3.21 – 3.16 (m, 1H), 2.92 – 2.86 (m, 1H), 2.55 (d, *J* = 5.4 Hz, 2H), 2.02 – 1.96 (m, 1H), 1.59 – 1.54 (m, 1H), 1.03 – 0.98 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.65, 149.13, 135.82, 129.22, 128.59, 128.34, 118.07, 107.14, 66.17, 63.56, 62.91, 52.94, 50.48, 47.20, 34.40, 28.22, 17.23. HRMS (ESI): calcd. for $C_{19}H_{23}N_2O_3$ ($[M+H]^+$): 327.1703, found 327.1702.



6k

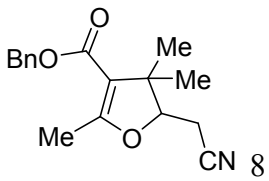
Yellow oil (88 mg, 60% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.27 (m, 5H), 7.12 (s, 1H), 4.49 (d, *J* = 15.0 Hz, 1H), 4.35 (d, *J* = 15.1 Hz, 1H), 3.66 (s, 3H), 3.40 (t, *J* = 5.5 Hz, 1H), 2.57 (dd, *J* = 17.1, 5.3 Hz, 1H), 2.52 (dd, *J* = 17.1, 5.7 Hz, 1H), 2.23 (dt, *J* = 14.0, 9.1 Hz, 1H), 2.09 – 1.98 (m, 1H), 1.95 – 1.84 (m, 1H), 1.83 – 1.72 (m, 2H), 1.56 – 1.43 (m, 2H), 1.39 – 1.29 (m, 1H). ^{13}C NMR (125MHz, $CDCl_3$) δ 165.68, 149.60, 135.94, 129.11, 128.36, 128.19, 118.09, 107.64, 67.78, 56.49, 52.76, 50.38, 39.84, 29.84, 25.77, 23.88, 17.61. HRMS (ESI): calcd. for $C_{19}H_{23}N_2O_2$ ($[M+H]^+$): 311.1754, found 311.1751.



6l

Yellow oil (80 mg, 54% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 – 7.30 (m, 3H), 7.25 – 7.20 (m, 2H), 7.04 (s, 1H), 4.42 (d, *J* = 15.2 Hz, 1H), 4.28 (d, *J* = 15.1 Hz, 1H), 3.70 (s, 3H), 3.62 (t, *J* = 5.5 Hz, 1H), 2.74 – 2.67 (m, 1H), 2.65 (dd, *J* = 7.0, 5.5 Hz, 2H), 2.63 – 2.59 (m, 1H), 2.28 – 2.20 (m, 1H),

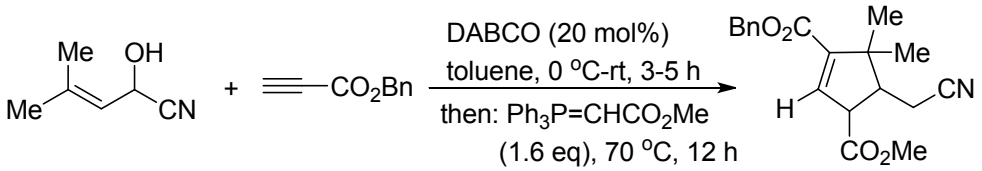
2.16 – 2.07 (m, 1H), 1.81 – 1.71 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.09, 149.50, 135.88, 129.14, 128.36, 128.03, 117.64, 108.53, 68.17, 52.68, 51.46, 50.49, 36.06, 26.84, 18.20, 15.67. HRMS (ESI): calcd. for $C_{18}H_{21}N_2O_2$ ([M+H]$^+$): 297.1598, found 297.1597.



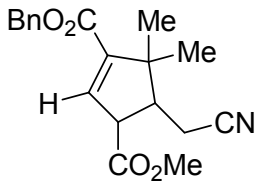
8

Yellow oil (68 mg, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.27 (m, 5H), 5.18 (s, 2H), 4.32 (dd, *J* = 7.7, 5.7 Hz, 1H), 2.71 – 2.58 (m, 2H), 2.18 (s, 3H), 1.33 (s, 3H), 1.19 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.33, 165.05, 136.33, 128.58, 128.08, 128.06, 116.86, 111.55, 85.99, 65.51, 45.52, 26.79, 20.63, 18.65, 14.85. HRMS (ESI): calcd. for: $C_{17}H_{19}NNaO_3$ ([M+Na]$^+$): 308.1257, found 308.1256.

Procedure for preparation of compound 9



To a solution of compound 1a (0.55 mmol) in toluene (2.5 mL) was added DABCO (20 mol%) at 0 °C under a N_2 atmosphere. Then 2a (0.5 mmol) was added, and keeping the reaction at 30 °C and monitoring by TLC. Upon completion, phosphonium ylide (0.8 mmol) was added, and the mixture was stirred at 70 °C. After 12h, DBU (20 mol%) was added. Upon completion, HCl (1 M) (10 mL) was added to the cooled mixture, and the resultant mixture was extracted with ethyl ether (10 mL×3). The combined organic layer was washed with brine and dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by column chromatography (silica gel, EtOAc/Petroleum ether (60-90 °C) to afford the desired product 9.



9

Yellow oil (88 mg, 54% yield, dr = 4.5/1, inseparable mixture). The major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.34 (m, 5H), 6.71 (d, *J* = 2.1 Hz, 1H), 5.22 (d, *J* = 12.5 Hz, 1H), 5.18 (d, *J* = 12.5 Hz, 1H), 3.78 (s, 3H), 3.46-3.43 (m, 1H), 2.66-2.54 (m, 3H), 1.44 (s, 3H), 1.22 (s, 3H). The minor isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.43 – 7.34 (m, 5H), 6.97 (d, *J* = 1.5 Hz, 1H), 5.22-5.15 (m, 2H), 3.80 (s, 3H), 2.83-2.81 (m, 1H), 2.78-2.73 (m, 1H), 2.70-2.68 (m, 1H), 2.66-2.54 (m, 1H), 1.45 (s, 3H), 0.99 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 171.84, 170.68, 164.48, 163.40, 144.69, 142.86, 137.92, 136.91, 135.83, 135.52, 128.80, 128.72, 128.63, 128.58, 128.42, 128.33, 118.70, 118.44, 66.85, 66.36, 61.31, 59.30, 52.72, 52.64, 51.96, 51.52, 48.42, 47.39, 45.32, 26.24, 23.26, 20.91, 17.98, 16.49.

HRMS (ESI): calcd. for: $C_{19}H_{22}NO_4$ ($[M+H]^+$): 328.1543, found 328.1545.

V. References

1. A. Baeza, C. Nájera, M. G. Retamosa, J. M. Sansano, *Synthesis*, 2005, *16*, 2787.

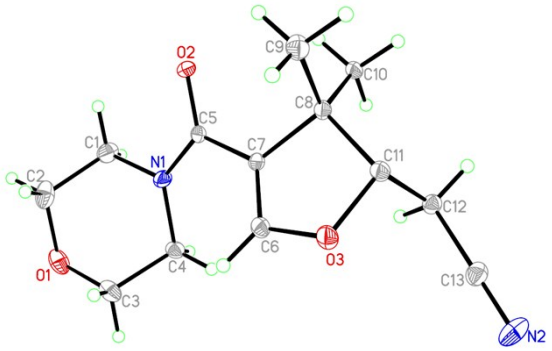
2. K. Y. Park, J. H. Ryu, Park, Lee, S. G. *Green Chem*, 2009, *11*, 946 -948.

3. Y. C. Fan, O. Kwon, *Org. Lett*. 2012, *14*, 3264.

4. Oaklade, J. S.; Sit, R. K.; Fokin, V. V. *Chem. Eur. J*. 2014, *20*, 11101.

VI. Crystal Data and Structure Refinement

1) Compound 3g



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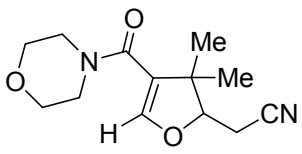

5a

Table 1. Crystal data and structure refinement for 5a.

Identification code	5a	
Empirical formula	C13 H17 N2 O3	
Formula weight	249.29	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 9.9995(3) Å	α= 90°.
	b = 10.3691(3) Å	β= 101.1920(10)°.
	c = 12.6092(3) Å	γ = 90°.
Volume	1282.53(6) $Å^3$	
Z	4	
Density (calculated)	1.291 Mg/m^3	
Absorption coefficient	0.093 mm^{-1}	
F(000)	532	
Crystal size	0.21 x 0.20 x 0.18 mm^3	
Theta range for data collection	2.86 to 25.85°.	
Index ranges	-12<=h<=12, -12<=k<=12, -15<=l<=15	
Reflections collected	18243	
Independent reflections	2467 [R(int) = 0.0227]	
Completeness to theta = 25.85°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9835 and 0.9808	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2467 / 0 / 165	
Goodness-of-fit on F^2	1.090	

Final R indices [I>2sigma(I)]	R1 = 0.0589, wR2 = 0.1366
R indices (all data)	R1 = 0.0602, wR2 = 0.1374
Largest diff. peak and hole	0.798 and -0.280 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for Y. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(2)	8966(1)	3830(1)	1909(1)	21(1)
O(3)	5686(1)	1941(2)	-622(1)	26(1)
C(10)	9207(2)	1173(2)	782(2)	20(1)
O(1)	7747(2)	7832(2)	7(2)	34(1)
C(7)	7486(2)	3019(2)	380(2)	17(1)
N(1)	8418(2)	5229(2)	516(1)	25(1)
C(5)	8335(2)	4051(2)	979(2)	18(1)
C(8)	7725(2)	1590(2)	678(2)	18(1)
C(12)	7381(2)	633(2)	-1242(2)	21(1)
C(4)	7936(2)	5623(2)	-605(2)	24(1)
C(9)	7216(2)	1280(2)	1728(2)	28(1)
C(6)	6293(2)	3102(2)	-302(2)	22(1)
C(11)	6730(2)	956(2)	-272(2)	21(1)
C(3)	7043(2)	6809(2)	-632(2)	31(1)
N(2)	5546(2)	-9(2)	-2933(2)	39(1)
C(13)	6343(2)	268(2)	-2187(2)	27(1)
C(1)	9122(3)	6278(2)	1177(2)	34(1)
C(2)	8177(3)	7432(2)	1098(2)	37(1)

Table 3. Bond lengths [Å] and angles [°] for Y.

O(2)-C(5)	1.240(2)
O(3)-C(6)	1.372(3)
O(3)-C(11)	1.466(2)
C(10)-C(8)	1.525(3)
O(1)-C(2)	1.422(3)
O(1)-C(3)	1.431(3)
C(7)-C(6)	1.331(3)

C(7)-C(5)	1.480(3)
C(7)-C(8)	1.535(3)
N(1)-C(5)	1.363(3)
N(1)-C(4)	1.461(3)
N(1)-C(1)	1.465(3)
C(8)-C(9)	1.542(3)
C(8)-C(11)	1.547(3)
C(12)-C(13)	1.470(3)
C(12)-C(11)	1.530(3)
C(4)-C(3)	1.517(3)
N(2)-C(13)	1.145(3)
C(1)-C(2)	1.516(4)

C(6)-O(3)-C(11)	105.93(15)
C(2)-O(1)-C(3)	110.62(17)
C(6)-C(7)-C(5)	129.57(19)
C(6)-C(7)-C(8)	107.32(17)
C(5)-C(7)-C(8)	121.76(16)
C(5)-N(1)-C(4)	129.00(18)
C(5)-N(1)-C(1)	118.94(17)
C(4)-N(1)-C(1)	112.02(17)
O(2)-C(5)-N(1)	120.58(18)
O(2)-C(5)-C(7)	119.04(18)
N(1)-C(5)-C(7)	120.38(17)
C(10)-C(8)-C(7)	113.43(16)
C(10)-C(8)-C(9)	110.09(17)
C(7)-C(8)-C(9)	110.48(16)
C(10)-C(8)-C(11)	114.47(16)
C(7)-C(8)-C(11)	100.08(15)
C(9)-C(8)-C(11)	107.83(16)
C(13)-C(12)-C(11)	111.20(17)
N(1)-C(4)-C(3)	109.33(18)
C(7)-C(6)-O(3)	115.00(18)
O(3)-C(11)-C(12)	107.51(16)
O(3)-C(11)-C(8)	104.46(15)
C(12)-C(11)-C(8)	113.72(16)
O(1)-C(3)-C(4)	111.43(18)
N(2)-C(13)-C(12)	179.0(2)

N(1)-C(1)-C(2)	108.9(2)
O(1)-C(2)-C(1)	111.18(19)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for Y. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(2)	27(1)	21(1)	15(1)	0(1)	1(1)	0(1)
O(3)	16(1)	25(1)	35(1)	-5(1)	-1(1)	4(1)
C(10)	19(1)	18(1)	22(1)	0(1)	0(1)	4(1)
O(1)	37(1)	21(1)	49(1)	7(1)	16(1)	3(1)
C(7)	18(1)	19(1)	15(1)	-2(1)	5(1)	4(1)
N(1)	37(1)	18(1)	18(1)	1(1)	2(1)	-2(1)
C(5)	20(1)	18(1)	16(1)	-1(1)	5(1)	4(1)
C(8)	19(1)	17(1)	19(1)	-2(1)	3(1)	0(1)
C(12)	21(1)	21(1)	21(1)	-1(1)	3(1)	-2(1)
C(4)	27(1)	26(1)	19(1)	6(1)	7(1)	5(1)
C(9)	34(1)	23(1)	28(1)	2(1)	13(1)	-1(1)
C(6)	19(1)	23(1)	25(1)	-3(1)	3(1)	5(1)
C(11)	18(1)	20(1)	22(1)	-1(1)	2(1)	2(1)
C(3)	30(1)	28(1)	37(1)	8(1)	10(1)	6(1)
N(2)	36(1)	51(1)	28(1)	-9(1)	1(1)	-16(1)
C(13)	32(1)	27(1)	24(1)	-3(1)	6(1)	-2(1)
C(1)	52(2)	20(1)	27(1)	0(1)	1(1)	-9(1)
C(2)	54(2)	20(1)	41(1)	-5(1)	22(1)	-9(1)

Table 5. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^{\ 3}$) for Y.

	x	y	z	U(eq)
H(10A)	9515	1373	126	30
H(10B)	9278	261	911	30

H(10C)	9760	1622	1375	30
H(12A)	7884	1376	-1420	25
H(12B)	8020	-73	-1053	25
H(4A)	7418	4928	-1006	28
H(4B)	8707	5814	-941	28
H(9A)	7272	367	1857	42
H(9B)	6286	1556	1658	42
H(9C)	7772	1723	2323	42
H(6)	5900	3888	-545	27
H(3A)	6764	7099	-1373	37
H(3B)	6229	6589	-360	37
H(1A)	9945	6510	922	41
H(1B)	9378	6002	1924	41
H(2A)	7386	7209	1399	44
H(2B)	8645	8139	1519	44