Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

A Challenging Heck Reaction of Maleimides

Li Hui Lim and Jianrong (Steve) Zhou

Division of Chemistry and Biological Chemistry

School of Physical and Mathematical Sciences

Nanyang Technological University, 21 Nanyang Link, Singapore 637371

Fax: (+65) 67911961; E-mail: jrzhou@ntu.edu.sg

Supporting Information: procedures and characterization of compounds

Table of Contents

I. General

^{1}H NMR spectra were acquired on Bruker 400 MHz spectrometers and chemical shifts were recorded relative to tetramethylsilane (δ 0.00) or residual protiated solvent ($CDCl_3$: δ 7.26). Multiplicities were given as: s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). The number of protons (n) for a given resonance was indicated by nH. Coupling constants were reported as a *J* value in Hz. ^{13}C NMR spectra were obtained at 100 MHz on 400 MHz instruments and chemical shifts were recorded relative to solvent resonance ($CDCl_3$: δ 77.16). Proof of purity of new compounds was demonstrated with copies of ^{1}H and ^{13}C NMR spectra.

Glassware was dried in an oven at 120 ºC for at least 2 hours before use. Ethylene carbonate was purchased from Fluka and was used without further purification.

Unless noted otherwise, commercially available chemicals were used without further purification. The GC standard, *n*-tetradecane was degassed with argon bubbling and dried over activated 4 Å molecular sieve beads for a few days in the glove box before use.

Thin-layer chromatography (TLC) was conducted with Merck 60 F254 coated silica gel plate (0.2 mm thickness). Flash chromatography was performed using Merck silica gel 60 (0.040-0.063 mm).

Gas chromatography (GC) analysis was performed on a Shimadzu GC-2010 instrument with Agilent J & W GC column DB-5MS-UI. GC/MS analysis was conducted on a Thermo Scientific DSQ II single quadrupole GC/MS instrument with Agilent J & W GC column DB-5MS-UI. ESI/MS analysis was conducted on a ThermoFinnigan LCQ Fleet MS spectrometer.

II. Synthesis of *N*-substituted maleimide

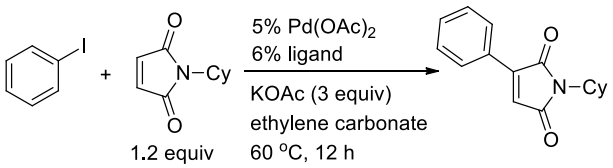
N-Substituted maleimides were synthesized by using a reported procedure with modification (Reddy, P. Y.; Kondo, S.; Toru, T.; Ueno, Y. *J. Org. Chem.* **1997**, *62*, 2652). Under argon, maleic anhydride (2.16 g, 22 mmol) was added to dry toluene (120 mL) and the mixture was stirred for 10 min. The corresponding primary amine

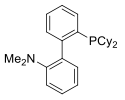
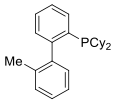
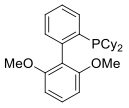
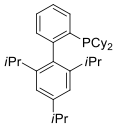
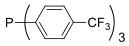
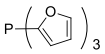
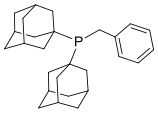
(22 mmol) was added dropwise and the reaction was stirred at room temperature for 1 h. Dry ZnBr (4.50 g, 20 mmol) was added in one portion and then a solution of $HN(SiMe_3)_2$ (6.3 mL, 30 mmol) in toluene (20 mL) was added dropwise over 30 min using a syringe pump while the reaction was heating to 80 ºC. After addition, the reaction was stirred with reflux for 1 h. The reaction was then cooled to rt and 2 M HCl (100 mL) was added. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (100 mL × 3). Then combined organic layer was combined and dried over anhydrous Na_2SO_4 and then concentrated on a rotary evaporator. The product was purified by silica gel flash chromatography using EA/hexane as eluent. The purity of compounds was proved by NMR spectroscopy.

III. Condition optimization of Heck reaction of maleimides

A typical procedure: In an argon-filled glove box, a dry 10-mL Schlenk tube containing a magnetic stirbar was charged with $Pd(OAc)_2$ (5 mol%, 1.1 mg, 0.005 mmol), dppf (6 mol%, 3 mg, 0.006 mmol), *N*-cyclohexylmaleimide (22 mg, 0.12 mmol), KOAc (29 mg, 0.3 mmol) and dry ethylene carbonate (0.4 ml). Iodobenzene (11 μL, 0.1 mmol) and GC standard *n*-tetradecane (10 μL) were added via a syringe. The tube was capped tightly and the mixture was vigorously stirred in an oil bath maintained at 60 ºC for 12 h. An aliquot was then taken from the reaction mixture and passed through a short plug of silica gel with EA washings. The filtrate was subjected to GC analysis to determine the conversion of iodobenzene and maleimide and the calibrated yield of the Heck product.

Table S1 Effect of Phosphine Ligands



Ligand	Conversion of PhI (%)	Conversion of maleimide (%)	Yield (%)
 Cy-JohnPhos	100	115	61
 DavePhos	50	117	33
 **MePhos**	**100**	**112**	**93**
 **SPhos**	**100**	**111**	**92**
 XPhos	99	116	61
	100	113	63
PPh_3	100	117	60
	95	107	73
PCy_3	**100**	**120**	**90**
tBu$_3$PHBF$_4$	17	79	11
tBu$_3$PHBF$_4$ + 6% KOtBu	69	114	45
	97	112	78

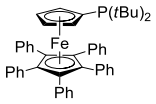
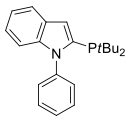
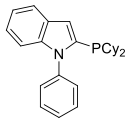
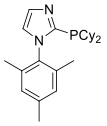
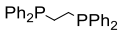
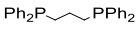
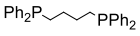
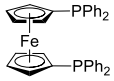
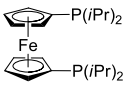
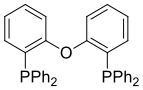
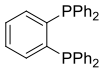
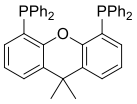
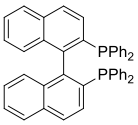
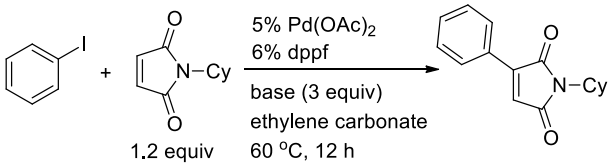
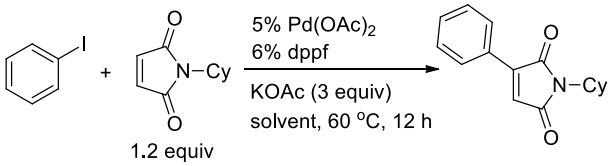
	**98**	**106**	**97**
CTC-Q-Phos			
	83	116	70
	2	118	0
	0	118	0
 dppe	100	116	89
 dppp	57	119	39
dppb	87	116	63
 dpppe	88	116	65
 **dppf**	**100**	**106**	**90**
 dippf	97	115	87
 **DPEPhos**	**100**	**105**	**96**
 dppbz	8	117	1
 Xantphos	96	108	78
 rac-BINAP	5	80	1
no ligand	85	100	64

Table S2 Effect of Bases



Base	Conversion of PhI (%)	Conversion of maleimide (%)	Yield (%)
LiOAc	25	57	17
NaOAc	40	89	31
KOAc	**100**	**116**	**87**
$NaHCO_3$	91	116	71
$KHCO_3$	100	120	80
Li_2CO_3	9	98	5
Na_2CO_3	49	74	41
K_2CO_3	21	120	4
Cs_2CO_3	35	120	0
K_3PO_4	88	119	49
KF	80	120	52
Et_3N	72	119	30
iPr_2NEt	97	119	36

Table S3 Effect of Solvents

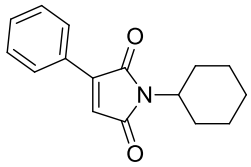


Solvent	Conversion of PhI (%)	Conversion of maleimide (%)	Yield (%)
Ethylene carbonate	**100**	**112**	**88**
Dimethyl carbonate	84	85	67
Diethyl carbonate	61	65	50
Ethyl acetate	87	86	74
Diglyme	97	102	64
Triglyme	99	107	63
1,4-Dioxane	82	85	69
THF	85	92	62
Dichloromethane	22	27	15
1,2-Dichloroethane	55	56	46
Toluene	26	35	15
$PhCF_3$	69	73	55

Anisole	23	33	14
DMF	66	120	38
DMA	96	108	64
DMSO	8	120	0

IV. Isolation of Heck products

In an argon-filled glove box, a dry 10-mL Schlenk tube containing a magnetic stirbar was charged with $Pd(OAc)_2$ (5.6 mg, 0.025 mmol), dppf (17 mg, 0.03 mmol), *N*-substituted maleimide (0.6 mmol), KOAc (147.0 mg, 1.5 mmol), ethylene carbonate (1.98 g) and aryl iodide (0.5 mmol). The Schlenk tube was capped tightly and the mixture was heated with vigorous stirring in a 60 ºC oil bath. After the aryl iodide was fully consumed (monitored by GC), the reaction mixture was passed through a pad of silica gel with EA washings to remove the catalyst and inorganic salts. The filtrate was then concentrated on a rotary evaporator and the residue was directly subjected to silica gel flash chromatography.

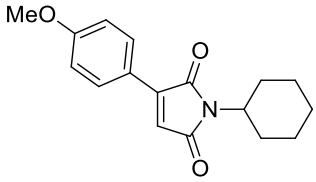


***N*-Cyclohexyl-3-phenylmaleimide [16213-23-3].** In an argon-filled glove box, a dry 10-mL Schlenk tube containing a magnetic stir bar was charged with $Pd(OAc)_2$ (5.6 mg, 0.025 mmol), dppf (16.6 mg, 0.03 mmol), *N*-cyclohexylmaleimide (107.4 mg, 0.6 mmol), KOAc (147 mg, 1.5 mmol), ethylene carbonate (1.98 g) and iodobenzene (102 mg, 0.5 mmol). The Schlenk tube was capped tightly and the mixture was heated with vigorous stirring in a pre-warmed 60 ºC oil bath for 8 h. The product was directly purified by flash chromatography (EA/hexanes 1: 20) as pale yellow solid (110 mg, 86%). NMR data of this compound were reported previously (K. Onimura, M. Matsushima, M. Nakamura, T. Tominaga, K. Yamabuki and T. Oishi, *J. Polym. Sci. A: Polym. Chem.* 2011, **49**, 3550).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91-7.89 (m, 2H), 7.46-7.44 (m, 3H), 6.66 (s, 1H), 3.98 (tt, *J* = 12.1, 3.8 Hz, 1H), 2.12 (ψqd, *J* = 12.4, 2.7 Hz, 2H), 1.87-1.84 (m, 2H),

1.73-1.67 (m, 3H), 1.40-1.22 (m, 3H).

GCMS (EI): Calcd for $C_{16}H_{17}NO_2$: 255.3. Found: 255.1.

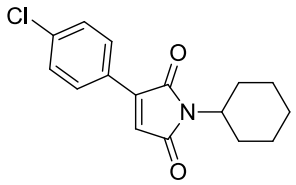


***N*-Cyclohexyl-3-(4-methoxyphenyl)maleimide.** The reaction was stirred at 60 ºC for 6 h. The product was purified by flash chromatography (EA/hexanes 1: 20) as bright yellow solid (115 mg, 79%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92-7.90 (m, 2H), 6.97-6.95 (m, 2H), 6.53(s, 1H), 3.96 (tt, *J* = 12.3, 3.8 Hz, 1H), 3.86 (s, 3H), 2.11 (ψqd, *J*= 12.5, 3.2 Hz, 2H), 1.86-1.83 (m, 2H), 1.71-1.66 (m, 3H), 1.40-1.18 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 171.2, 171.0, 162.0, 142.9, 130.5, 121.8, 121.3, 114.6, 55.6, 50.9, 30.2, 26.2, 25.3.

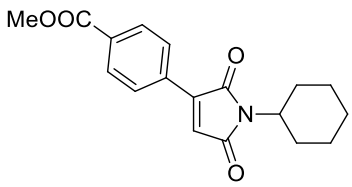
GCMS (EI): Calcd for $C_{17}H_{19}NO_3$: 285.3. Found: 285.1.



***N*-Cyclohexyl-3-(4-chlorophenyl)maleimide [311328-77-5].** The reaction was stirred at 60 ºC for 7 h. The product was purified by flash chromatography (EA/hexanes 1: 20) as white solid (115 mg, 79%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87-7.85 (m, 2H), 7.43-7.42 (m, 2H), 6.66 (s, 1H), 3.97 (tt, *J* = 12.4, 3.9 Hz, 1H), 2.20 (ψqd, *J*= 12.6, 3.1 Hz, 2H), 1.87-184 (m, 2H), 1.72-1.68 (m, 3H), 1.40-1.19 (m, 3H).

GCMS (EI): Calcd for $C_{16}H_{16}ClNO_2$: 289.8. Found: 289.1.

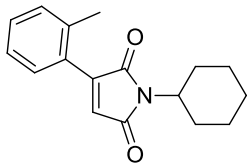


Methyl 4-(1-cyclohexyl-2,5-dioxo-2,5-dihydro-1H-pyrrol-3-yl)benzoate. The reaction was stirred at 60 ºC for 20 h. The product was purified by flash chromatography (EA/hexanes 1: 20) as white solid (106 mg, 68%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.12-8.09 (m, 2H), 7.99-7.97 (m, 2H), 6.77 (s, 1H), 3.99 (tt, *J*= 12.4, 3.8 Hz, 1H), 3.95 (s, 3H), 2.11 (ψqd, *J*= 12.5, 3.3 Hz, 2H), 1.88-1.84 (m, 2H), 1.73-1.67 (m, 3H), 1.41-1.19 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.4, 170.1, 166.5, 142.3, 133.1, 132.1, 130.1, 128.7, 126.0, 52.5, 51.2, 30.1, 26.1, 25.2.

GCMS (EI): Calcd for $C_{18}H_{19}NO_4$: 313.4. Found: 313.1.

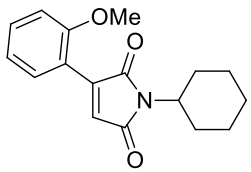


***N*-Cyclohexyl-3-(*o*-tolyl)maleimide.** The reaction was stirred at 60 ºC for 21 h. The product was purified by flash chromatography (EA/hexanes 1: 30) as colourless oil (117 mg, 87%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48-7.46 (m, 1H), 7.36-7.26 (m, 3H), 6.52 (s, 1H), 3.99 (tt, *J* = 12.3, 3.8 Hz, 1H), 2.38 (s, 3H), 2.12 (ψqd, *J* = 12.5, 3.2 Hz, 2H), 1.87-1.84 (m, 2H), 1.75-1.67 (m, 3H), 1.39-1.22 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.8, 144.9, 137.4, 131.1, 130.4, 130.2, 128.4, 128.2, 126.0, 51.2, 30.1, 26.1, 25.2, 21.1.

GCMS (EI): Calcd for $C_{17}H_{19}NO_2$: 269.3. Found: 269.1.



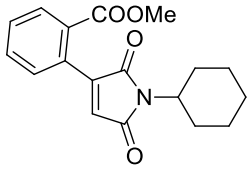
***N*-Cyclohexyl-3-(2-methoxyphenyl)maleimide.** The reaction was stirred in a 60 ºC oil bath for 21 h. The product was purified by flash chromatography (EA/hexanes 1: 20) as yellow oil (137 mg, 96%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.21 (dd, *J* = 17.8, 1.6 Hz, 1H), 7.40 (ψtd, *J*= 7.8, 1.6

Hz, 1H), 7.06 (d, J= 8 Hz, 1H) 7.02 (s, 1H), 6.97 (d, *J*= 8.4 Hz, 1H), 3.98 (tt, *J* = 12.3, 3.9 Hz, 1H), 3.91 (s, 3H), 2.12 (ψqd, *J*= 12.5, 3.2 Hz, 2H), 1.86-1.83 (m, 2H), 1.71-1.66 (m, 3H), 1.39-1.22 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 171.9, 171.7, 159.5, 138.5, 131.8, 131.5, 127.8, 120.8, 118.1, 111.1, 55.6, 50.9, 30.1, 26.2, 25.3.

GCMS (EI): Calcd for $C_{17}H_{19}NO_3$: 285.3. Found: 285.1.

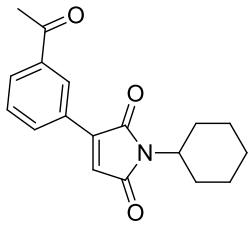


Methyl 2-(1-cyclohexyl-2,5-dioxo-2,5-dihydro-1H-pyrrol-3-yl)benzoate. The reaction was stirred at 60 ºC in an oil bath for 22 h. The product was purified by flash chromatography (EA/hexanes 1: 10) as colourless oil (116 mg, 74%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (dd, *J*= 7.6, 1.2 Hz, 1H), 7.60 (ψtd, *J*= 7.4, 1.6 Hz, 1H), 7.54 (ψtd, *J*= 7.6, 1.6 Hz, 1H), 7.36 (dd, *J*= 7.2, 1.2 Hz, 1H), 6.50 (s, 1H), 3.96 (tt, *J*= 12.3, 3.9 Hz, 1H), 3.82 (s, 3H), 2.10 (ψqd, *J*= 12.5, 3.0 Hz, 2H), 1.86-1.83 (m, 2H), 1.74-1.65 (m, 3H), 1.38-1.21 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.7, 170.1, 167.2,148.2, 132.4, 131.0, 130.6, 130.5, 130.3, 130.1, 125.2, 52.5, 51.2, 30.1, 26.1, 25.2.

GCMS (EI): Calcd for $C_{18}H_{19}NO_4$: 313.4. Found: 313.1.



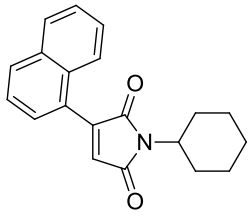
***N*-Cyclohexyl-3-(3-acetylphenyl)maleimide.** The reaction was stirred at 60 ºC in an oil bath for 8 h. The product was purified by flash chromatography (EA/hexanes 1: 8) as white solid (114 mg, 77%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (t, *J* = 1.6 Hz, 1H), 8.08 (td, *J*= 8.0, 1.6 Hz, 1H), 8.03 (td, *J*= 8.0, 1.3 Hz, 1H), 7.56 (t, *J* = 7.8 Hz, 1H), 6.76 (s, 1H), 3.98 (tt, *J* = 12.3,

3.9 Hz, 1H), 2.64 (s, 3H), 2.11 (ψqd, *J* = 12.4, 3.0 Hz, 2H), 1.87-1.83 (m, 2H), 1.73-1.66 (m, 3H), 1.39-1.18 (m, 3 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 197.4, 170.6, 170.2, 142.4, 137.8, 132.9, 130.5, 129.5, 129.4, 128.6, 125.2, 51.2, 30.1, 26.8, 26.1, 25.2.

GCMS (EI): Calcd for $C_{18}H_{19}NO_3$: 297.4. Found: 297.1.

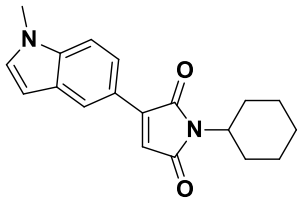


***N*-Cyclohexyl-3-(1-naphthyl)maleimide.** DPEPhos (16 mg, 6 mol%) was used as the ligand instead of DPPF. The reaction was stirred at 60 ºC in an oil bath for 15 h. The product was purified by flash chromatography (EA/hexanes 1: 10) as yellow oil (151 mg, 99%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01-7.90 (m, 3H), 7.68 (dd, *J*= 7.2, 1.2 Hz, 1H), 7.57-7.53 (m, 3H), 6.77 (s, 1H), 4.06 (tt, *J*= 12.3, 3.9 Hz, 1H), 2.17 (ψqd, *J*= 12.5, 3.3 Hz, 2H), 1.90-1.68 (m, 5H), 1.43-1.24 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.9, 143.8, 134.0, 131.3, 130.9, 129.1, 129.0, 128.9, 127.3, 126.5, 126.3, 125.2, 124.7, 51.4, 30.3, 26.2, 25.3.

GCMS (EI): Calcd for $C_{20}H_{19}NO_2$: 305.4. Found: 305.1.



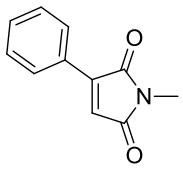
***N*-Cyclohexyl-3-(*N*-methyl-5-indolyl)maleimide.** DPPF was used as the ligand. THF (1.5 mL) was used as the solvent instead of ethylene carbonate. The reaction was stirred at 90 ºC in an oil bath for 3 h. The product was purified by flash chromatography (EA/hexanes 1: 10) as orange solid (118 mg, 76%). The product can be easily oxidized in air or in slightly acidic $CDCl_3$. The NMR data of the compound was reported (Liu, Y.; Zhang, W. *Angew. Chem. Int. Ed.* **2013**, *52*, 2203). If ethylene

carbonate was used as a solvent, aonly 10% yield was obtained. If *N*H 5-iodoindole was used, the reaction proceeded smoothly in good yield, but the product is very susceptible towards air oxidation during flash chromatography.

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.39 (d, *J*=1.2 Hz, 1H), 7.71 (dd, *J*= 8.8, 1.6 Hz, 1H), 7.35 (d, *J*= 8.8 Hz, 1H), 7.09 (d, *J*= 2.4 Hz, 1H), 6.59-6.57 (m, 2H), 3.99 (tt, *J*= 12.3, 3.6 Hz, 1H), 3.81 (s, 3H), 2.15 (ψqd, *J*= 12.3, 3.1 Hz, 2H), 1.87-1.84 (m, 2H), 1.74-1.67 (m, 3H), 1.41-1.23 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 171.6, 144.8, 138.3, 130.5, 129.1, 123.1, 122.3, 121.1, 120.8, 110.2, 103.1, 52.1, 33.4, 30.4, 26.5, 25.6.

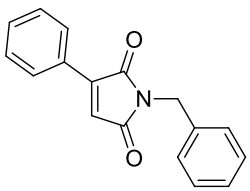
GCMS (EI): Calcd for $C_{20}H_{19}NO_2$: 308.4. Found: 308.1.



***N*-Methyl-3-phenylmaleimide [54433-49-7].** DPEPhos (16 mg, 6 mol%) was used as the ligand instead of DPPF. The reaction was stirred at 60 ºC in an oil bath for 2 h. The product was purified by flash chromatography (EA/hexanes 1: 8) as pale yellow solid (81.2 mg, 87%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93-7.90 (m, 2H), 7.48-7.44 (m, 3H), 6.73 (s, 1H), 3.08 (s, 3H).

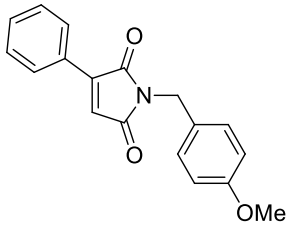
GCMS (EI): Calcd for $C_{17}H_{13}NO_2$: 187.2. Found: 187.1.



***N*-Benzyl-3-phenylmaleimide [15093-83-1].** The reaction was stirred at 60 ºC in an oil bath for 4 h. The product was purified by flash chromatography (EA/hexanes 1: 20) as white solid (121.2 mg, 92%). NMR data of this compound were reported previously (K. Komeyama, T. Kashihara and K. Takaki, *Tetrahedron Lett.,* 2013, **54**, 1084).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93-7.90 (m, 2H), 7.47-7.39 (m, 5H), 7.35-7.27 (m, 3H), 6.74 (s, 1H), 4.74 (s, 2H).

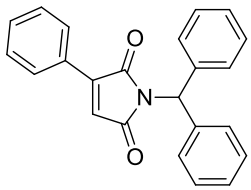
GCMS (EI): Calcd for $C_{17}H_{13}NO_2$: 263.3. Found: 263.1.



***N*-(4-Methoxybenzyl)-3-phenylmaleimide [935689-24-0].** The reaction was stirred at 60 ºC in an oil bath for 7 h. The product was purified by flash chromatography (EA/hexanes 1: 20) as white solid (135 mg, 92%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92-7.89 (m, 2H), 7.46-7.44 (m, 3H), 7.36-7.34 (m, 2H), 6.86-6.84 (m, 2H), 6.72 (s, 1H), 4.68 (s, 2H), 3.78 (s, 3H).

GCMS (EI): Calcd for $C_{18}H_{15}NO_3$: 293.3. Found: 293.1.

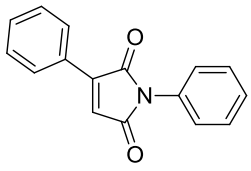


***N*-(1-Benzhydryl)-3-phenylmaleimide.** The reaction was stirred at 60 ºC in an oil bath for 7 h. The product was purified by flash chromatography (EA/hexanes 1: 20) as white solid (149 mg, 83%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93-7.90 (m, 2H), 7.45-7.44 (m, 3H), 7.35-7.30 (m, 10H), 6.76 (s, 1H), 6.59 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 170.4, 170.1, 143.8, 138.4, 131.4, 129.1, 128.9, 128.82, 128.79, 128.6, 127.9, 124.2, 57.9.

GCMS (EI): Calcd for $C_{17}H_{19}NO_3$: 339.4. Found: 339.1.

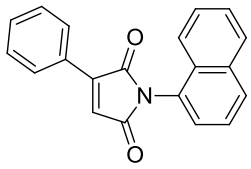


***N*-Phenyl-3-phenylmaleimide [75066-70-5].** DPEPhos (16.1 mg, 6 mol%) was used as the ligand instead of DPPF. The reaction was stirred at 60 ºC in an oil bath for 6 h.

The product was purified by flash chromatography (EA/hexanes 1: 20) as bright yellow solid (99 mg, 80%). NMR data of this compound were reported previously (N. Matuszak, G. G. Muccioli, G. Labar and D. M. Lambert, *J. Med. Chem.,* 2009, **52**, 7410).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00-7.97 (m, 2H), 7.51-7.48 (m, 5H), 7.42-7.37 (m, 3H), 6.89 (m, 1H).

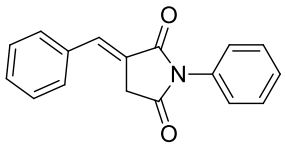
GCMS (EI): Calcd for $C_{16}H_{11}NO_2$: 249.3. Found: 249.1.



***N*-(1-Naphthyl)-3-phenylmaleimide [1082665-73-3].** DPEPhos (16.1 mg, 6 mol%) was used as the ligand instead of DPPF. The reaction was stirred at 60 ºC in an oil bath for 7 h. The product was purified by flash chromatography (EA/hexanes 1: 8) as white solid (139 mg, 93%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06-8.04 (m, 2H), 7.99-7.93 (m, 2H), 7.65-7.50 (m, 7H), 7.44 (dd, *J*= 7.2, 0.8 Hz, 1H), 7.01 (s, 1H).

GCMS (EI): Calcd for $C_{20}H_{13}NO_2$: 299.3. Found: 299.1.



3-Methylene-1,4-diphenylpyrrolidine-2,5-dione [838846-38-1]. DPEPhos (16.1 mg, 6 mol%) was used as the ligand instead of DPPF. The reaction was stirred at 60 ºC in an oil bath for 6 h. The product was purified by flash chromatography (EA/hexanes 1: 8) as white solid (92 mg, 70%). The structure was assigned by comparing with reported NMR data (Liu, Y.; Zhang, W. *Angew. Chem. Int. Ed.* **2013**, 52, 2203).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (t, *J*= 2.2 Hz, 1H), 7.56-7.38 (m, 10H), 3.78 (d, *J*= 2.4 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$): δ 173.2, 170.2, 135.6, 134.3, 132.2, 130.5, 130.4, 129.4, 129.3, 128.8, 126.6, 123.2, 34.5.

GCMS (EI): Calcd for $C_{17}H_{13}NO_2$: 263.3. Found: 263.0.