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Graphical Abstract:

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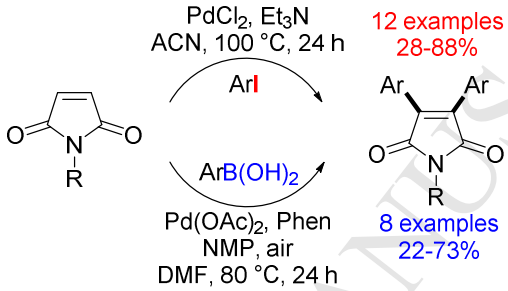

Palladium-Catalyzed Direct Arylation of Maleimides: A Simple Route to Bisaryl-Substituted Maleimides

Farnaz Jafarpour[b,*], Mitra Shamsianpour, Salumeh Issazadeh, Masoumeh Dorrani and Hamideh Hazrati

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Abstract— Palladium-catalyzed direct arylation of maleimides via Heck as well as organoboron-mediated Heck-type reactions are developed. These methods offer an approach to a wide variety of biologically interesting 3,4-diarylmaleimide scaffolds from readily accessible starting materials. These approaches led to the feasible one-pot construction of bisaryl-substituted maleimides which have historically been problematic**.**

Keywords: Boronic acids; Direct arylation; Maleimide; Oxidative Heck reaction; Palladium;

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1. Introduction

Bisaryl-substituted maleimides are important constituents in pharmaceutical drug candidates. The pharmaceutucal compositions comprising compounds of type **I** have been confirmed to be effective in treatment of metabolic bone diseases such as bone metastatic cancer, rheumatoid arthritis, periodontal disease and Paget's disease (Figure 1).[1] Natural product based maleimides of type **II** are proved to show valuable photophysical properties which may have applications as molecular probes to study biological processes like intracellular trafficking, membrane association and autotoxicity.[2] 3-Aryl-4-indolyl-maleimide **III** possesses remarkable potency against Vascular endothelial growth factor receptor 2 (VEGF-R2)/KDR.[3]

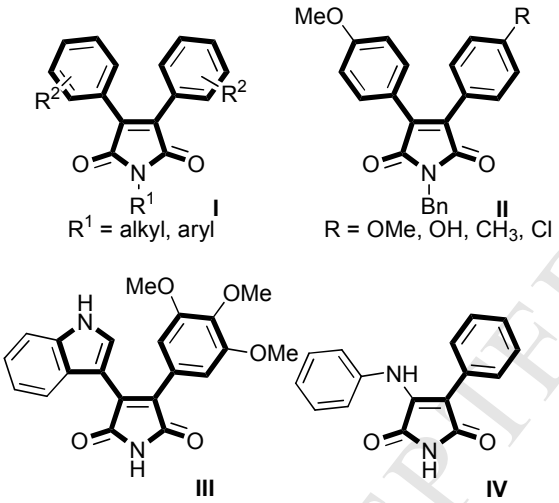


Fig. 1. Biologically active compounds containing the arylmaleimide framework.

Besides, some symmetrical or unsymmetrical bis(hetero)arylmaleimide derivatives have exhibited potent COX(Cyclooxygenase)-2 inhibitory activity and selectivity,[4] have the potent to induce B- to Z-DNA transition[5] and may have potential for clinical developments as antiangiogenic drugs.[3b] Furthermore π-extended diarylmaleimides have found some applications in organic light-emitting diodes and fluorescence devices.[6]

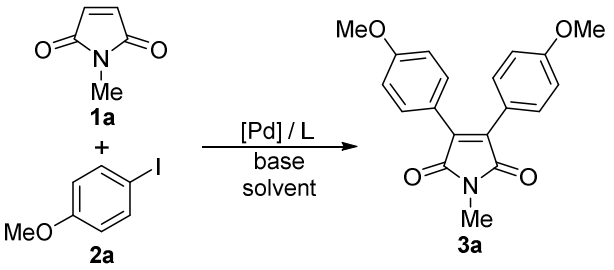
Given the broad spectrum of interesting properties, several research groups have aimed at preparation of these privileged structures. 3,4-Disubstituted maleimides are typically synthesized by two main approaches: a) formation of the maleimide ring in a linear synthetic sequence[7] and b) functionalization of prehalogenated maleimides via metal-catalyzed coupling reactions.[8] However, more synthetically viable preparation of these scaffolds via direct arylation of maleimides continues to be problematic. Very recently Zhou et al.[9] disclosed a Heck arylation of maleimides employing a combination of KOAc and a carbonate solvent. In spite of the importance of this contribution, monoarylated products were produced predominantly and exploratory experiments revealed the necessity for N-protection and the use of bulky ligands such as DPEphos. Bearing in mind the difficulty of generating tetrasubstituted alkenes using Heck coupling, direct arylation of maleimides leading to concise synthesis of 3,4-diaryl maleimides is not precedented. Driven by the need for an efficient synthetic route to these privileged motifs, we hypothesized the development of an efficient direct arylation of unfunctionalized maleimides and their free NH derivatives via Heck as well as organoboron-mediated Heck-type reactions employing iodoarene and boronic acid coupling partners, respectively. These protocols offer ligand-free efficient conditions for construction of crowded disubstituted maleimides taking advantage of precluding installment of disposable functionalities. Furthermore, double Heck process takes advantage of site selective C-functionalization of free-NH maleimides.

2. Results and Discussion

To test the hypothesis, model Heck coupling of maleimide **1a** with iodoanisole **2a** was conducted. Different reaction parameters including solvent and base were varied, and a combination of $Pd(OAc)_2$ and an alkylamine in acetonitrile one pot direct diarylation of maleimide was pleasingly achieved in 63% yield (Table 1, entries 1-9). The reaction efficiency was comparable or slightly lower in the presence of alkyl/aryl phosphine or pyridine ligands (entries 10-13). We also screened solvents and

Table 1

Optimization of reaction condition[a]



Entry	Catalyst	Ligand	Base	Solvent	Yield (%)
1	$Pd(OAc)_2$		AgOAc	DMF	12
2	$Pd(OAc)_2$		AgOAc	AcOH	28
3	$Pd(OAc)_2$		K_2CO_3	NMP	0
4	$Pd(OAc)_2$		K_2CO_3	ACN	22
5	$Pd(OAc)_2$		KOAc	ACN	5
6	$Pd(OAc)_2$		py	ACN	0
7	$Pd(OAc)_2$		TEA	ACN	18
8	$Pd(OAc)_2$		benzylamine	ACN	12
9	$Pd(OAc)_2$		Et_3N	ACN	63
10	$Pd(OAc)_2$	bpy	Et_3N	ACN	62
11	$Pd(OAc)_2$	$P(o\text{-}Tol)_3$	Et_3N	ACN	60
12	$Pd(OAc)_2$	dppe	Et_3N	ACN	58
13	$Pd(OAc)_2$	PPh_3	Et_3N	ACN	57
14	$Pd(OAc)_2$		Et_3N	dioxane	40
15	$Pd(OAc)_2$		Et_3N	DMF	22
16	$Pd(OAc)_2$		Et_3N	toluene	5
17	$Pd_2(dba)_3$		Et_3N	ACN	20
18	$Pd(acac)_2$		Et_3N	ACN	10
19	**$PdCl_2$**		**Et_3N**	**ACN**	**73**

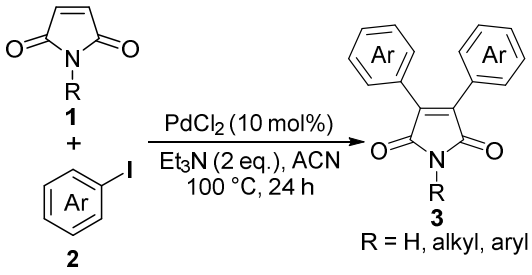
[a] Reaction conditions: maleimide **1a** (0.05 mmol, 1 eq.), iodoanisole **2a** (2 eq.), Pd catalyst (10 mol %), ligand (20 mol%), base (2 eq.), solvent (0.1 mL) at 100 °C for 24 h.

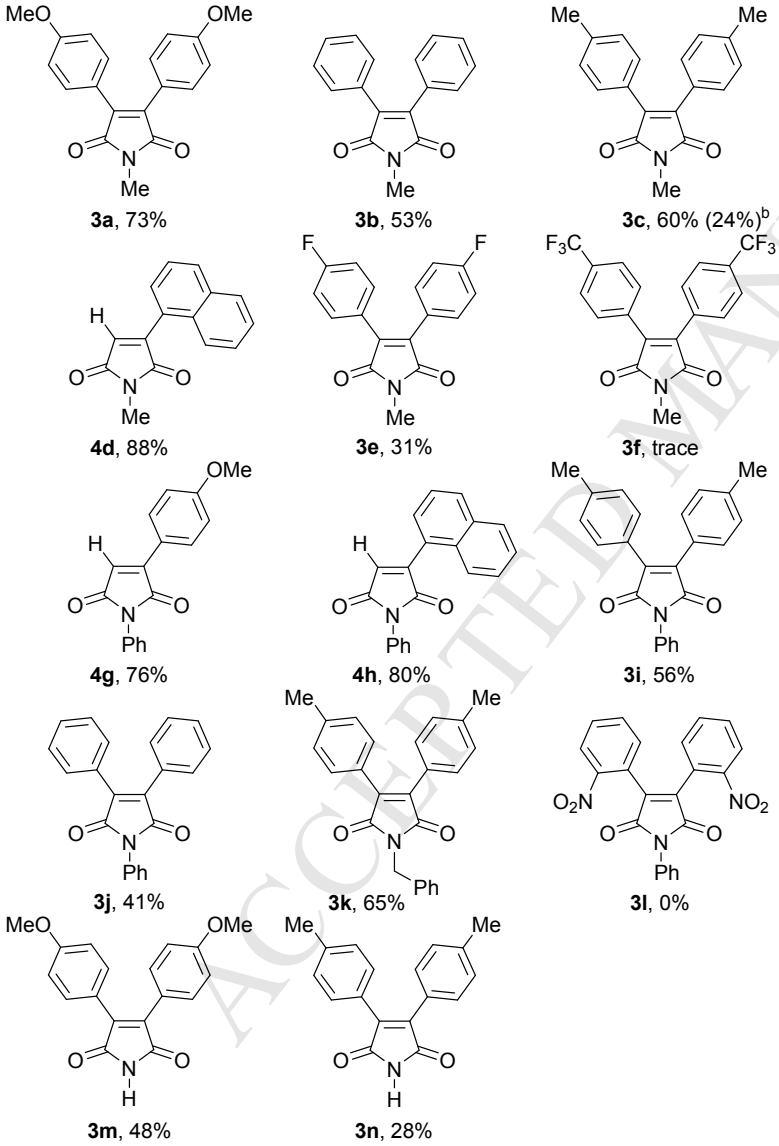
acetonitrile proved to be superior to other solvents such as DMF, DMSO, toluene, 1,4-dioxane, THF and NMP (entries 14–16). Finally the screening reactions were performed with respect to palladium sources and it turned out that replacement of $Pd(OAc)_2$ with $PdCl_2$ ensures the satisfactory result (entries 17-19). We were delighted to see that under the optimized reaction conditions, iodoarene (2.0 eq.), $PdCl_2$ (10 mol %), and NEt_3 (2.0 eq.) in acetonitrile at 100 °C for 24 h, 3,4-bis(4-methoxyphenyl)maleimide **3a** was obtained in 73% isolated yield (entry 19). It is noteworthy that 3,4-diarylmaleimides with introduced methoxy substituents on the phenyl rings have exhibited promising P-glycoprotein- modulating activity in Pgp-over expressing breast cancer cell lines without causing any cytotoxicity toward normal cells.[10]

With the optimized reaction conditions in hand, the scope of double-Heck process was explored and the results are summarized in Table 2. First Heck cross-coupling of *N*-methylmaleimide with iodoarenes possessing varying steric and electronic properties was investigated. Good to moderate yields of bisarylmaleimides were attained employing iodobenzene and toluene (**3b** and **3c**). 4-Bromotoluene was also tolerated under the reaction conditions where the desired product **3c** was obtained albeit in 24% yield. Surprisingly, 1-iodonaphthalene with a sterically demanding substitution pattern led to a monoarylated maleimide **4d** in 88% yield, where the diarylated isomer was not observed at all. Double Heck coupling is likely disfavored due to steric repulsion between two bulky naphthalene groups on the double bond. As expected, compared to 4-iodoanisole, fluorinated arenes such as 4-fluoro- and 4-trifluoromethylbenzene exhibited lower reactivity (**3e** and **3f**). More electron deficient arenes with nitro substitutions remained unreactive under the optimized reaction conditions. Notably, N-aryl and N-benzylmaleimides were tolerated and arylated maleimides were obtained in 41–80% yields. Gratifyingly, free (NH) maleimides also were found to be amenable to this cross-coupling reaction and regioselective direct C-arylations proceeded smoothly. To the best of our knowledge it is the first report on direct arylation of maleimides with no requisite for N-protection which is often necessary.

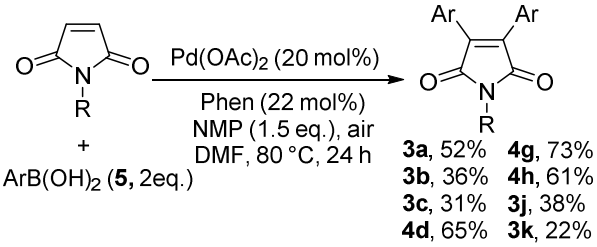
Motivated by these results, next we set out to explore direct diarylation of maleimides via organoboron-mediated Heck-type reaction[11] considering the commercial availability and low toxicity of boron reagents. In this regard, *N*-methylmaleimide **1a** was reacted with (4-methoxyphenyl)boronic acid **5a** (Scheme 1). To our delight, direct diarylation of maleimide employing boronic acid was simply achieved using some altered reaction conditions and adduct **3a** was obtained in 52% isolated yield (Table S2, Supporting Information). A similar trend was observed using various boronic acids as coupling partners where arylated N-alkyl(aryl) maleimides were obtained in 22–73% yields (Scheme 1).

Table 2 Scope of palladium-catalyzed direct arylation of maleimides[a]





[a]All reactions were run under the optimized reaction conditions. [b]Bromotoluene was employed.



Scheme 1. Palladium catalyzed oxidative Heck arylation of maleimides.

3. Summary and conclusions

In conclusion, we have discovered a simple route to diarylmaleimides which are of potent interest in medicinal chemistry via a one pot sequential double Heck arylation reaction. Construction of highly functionalized maleimides which is not feasible via direct arylation of maleimides is also achieved via an oxidative Heck coupling employing readily accessible arylboronic acids. The substrate scope is broad and further investigations to extend the substrate scope to more synthetic applications are underway.

4. Experimental Section

4.1. General

Anhydrous solvents were systematically used. Other reagents, palladium catalysts and ligands were commercially available and used as received. These reactions were carried out in an oil bath using Microwave Vials (2–5 mL). ^{1}H and ^{13}C NMR spectra were recorded at room temperature on 500 MHz spectrometers using $CDCl_3$ as the NMR solvent. ^{1}H NMR spectra are referenced to tetramethylsilane and ^{13}C NMR spectra are referenced from the solvent central peak. Chemical shifts are given in ppm. IR is reported as characteristic bands (cm^{-1}) in their maximal intensity.

4.2. General experimental procedures

4.2.1. Pd-catalyzed Heck-type arylation of maleimides with iodoarenes. A vial equipped with a stir bar was charged with maleimide (0.1 mmol, 1.0 eq.), iodoarene (0.2 mmol, 2.0 eq.), triethylamine (2.0 eq.) and $PdCl_2$ (10 mol %). Acetonitrile (0.2 mL) was then added, and the vial was capped. The resulting mixture was heated in an oil bath at 100 ˚C for 24 h, cooled, then filtered through a short plug of silica. Removal of the solvent under reduced pressure gave a crude mixture which was purified by column chromatography (hexanes/EtOAc gradient) to provide the title compound.

4.2.2. Pd-catalyzed oxidative Heck-type arylation of maleimides with boronic acids. A vial equipped with a stir bar was charged with maleimide (0.1 mmol, 1.0 eq.), boronic acid (0.2 mmol, 2.0 eq.), phenanthroline (22 mol%), NMP (0.15 mmol, 1.5 eq.) and $Pd(OAc)_2$ (20 mol %). DMF (0.24 mL) was then added, and the vial was capped. The resulting mixture was heated in an oil bath at 80 °C for 24 h, cooled, and then filtered through a short plug of silica. Removal of the solvent under reduced pressure gave a crude mixture which was purified by column chromatography (hexanes/EtOAc gradient) to provide the title compound.

4.3. Compounds Characterization Data

4.3.1. 3,4-Bis(4-methoxyphenyl)-1-methyl-1H-pyrrole-2,5-dione (3a).

Following procedure 4.2.1 with *N*-methylmaleimide (11 mg, 0.1 mmol) and 1-iodo-4-methoxybenzene (46 mg, 0.2 mmol), compound **3a** was obtained (24 mg, 73%) as yellow solid, m.p. 130–132 °C; [Found: C, 70.23; H, 5.14; N, 4.14. $C_{19}H_{17}NO_4$ requires C, 70.58; H, 5.30; N, 4.33%]; R_f (10% EtOAc/hexane) 0.16; ν_{max} (KBr) 2919, 1694, 1434, 1250 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 3.15 (3H, s, NMe), 3.85 (6, s, OMe), 6.89 (4H, d, *J* 8.5 Hz, Ph), 7.49 (4H, d, *J* 8.5 Hz, Ph); δ_C (100 MHz, $CDCl_3$) 170.4, 159.6, 133.2, 130.3, 120.3, 113.1, 54.3, 23.1; m/z 323 (78 $M^{\bullet+}$), 267 (100), 223 (39), 167 (23), 149 (44%).

4.3.2. 1-Methyl-3,4-diphenyl-1H-pyrrole-2,5-dione (3b).[12]

Following procedure 4.2.1 with *N*-methylmaleimide (11 mg, 0.1 mmol) and iodobenzene (40 mg, 0.2 mmol), compound **3b** was obtained (14 mg, 53%) as yellow solid, m.p. 118–120 °C; [Found: C, 77.81; H, 5.10; N, 5.47. $C_{17}H_{13}NO_2$ requires C, 77.55; H, 4.98; N, 5.32%]; R_f (10% EtOAc/hexane) 0.41; δ_H (250 MHz, $CDCl_3$) 3.19 (3H, s, NMe), 7.29–7.43 (6H, m, Ph), 7.48–7.51 (4H, m, Ph); δ_C (100 MHz, $CDCl_3$) 169.9, 135.3, 128.8, 128.7, 127.6, 127.5, 23.3; m/z 263 (100 $M^{\bullet+}$), 234 (7), 205 (41), 178 (62%).

4.3.3. 1-Methyl-3,4-di-p-tolyl-1H-pyrrole-2,5-dione (3c).

Following procedure 4.2.1 with *N*-methylmaleimide (11 mg, 0.1 mmol) and 1-iodo-4-methylbenzene (44 mg, 0.2 mmol), compound **3c** was obtained (17 mg, 60%) as yellow solid, m.p. 178–180 °C; [Found: C, 78.01; H, 5.75; N, 4.62. $C_{19}H_{17}NO_2$ requires C, 78.33; H, 5.88; N, 4.81%]; R_f (10% EtOAc/hexane) 0.45; ν_{max} (KBr) 2922, 1695, 1435, 1380 cm^{-1}; δ_H (250 MHz, $CDCl_3$) 2.37 (6H, s, Me), 3.15 (3H, s, NMe), 7.17 (4H, d, *J* 8.0 Hz, Ph), 7.39 (4H, d, *J* 8.0 Hz, Ph); δ_C (62.5 MHz, $CDCl_3$) 140.0, 129.8, 129.3, 126.0, 24.2, 21.5; m/z 291 (100 $M^{\bullet+}$), 276 (25), 233 (21), 219 (50), 206 (35), 191 (25%).

4.3.4. 1-Methyl-3-(naphthalen-1-yl)-1H-pyrrole-2,5-dione (4d).

Following procedure 4.2.1 with *N*-methylmaleimide (11 mg, 0.1 mmol) and 1-iodonaphthalene (51 mg, 0.2 mmol), compound **4d** was obtained (21 mg, 88%) as yellow solid, m.p. 132–134 °C; [Found: C, 76.25; H, 4.82; N, 6.08. $C_{15}H_{11}NO_2$ requires C, 75.94; H, 4.67; N, 5.90 %]; R_f (10% EtOAc/hexane) 0.28; ν_{max} (KBr) 2922, 1693, 1435, 1381, 1255 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 3.17 (3H, s, NMe), 6.84 (1H, s, =CH), 7.53–7.58 (3H, m, Ph), 7.68–7.70 (1H, m, Ph), 7.91–8.02 (3H, m, Ph); δ_C (100 MHz, $CDCl_3$) 169.8, 169.5, 143.3, 132.7, 129.8, 129.1, 127.8, 127.7, 127.6, 126.1, 125.3, 124.0, 123.3, 122.1, 23.1; m/z 237 (78 $M^{\bullet+}$), 221 (50), 152 (25), 97 (39), 71 (59), 57 (100%).

4.3.5. 3,4-Bis(4-fluorophenyl)-1-methyl-1H-pyrrole-2,5-dione (3e).

Following procedure 4.2.1 with *N*-methylmaleimide (11 mg, 0.1 mmol) and 1-fluoro-4-iodobenzene (44 mg, 0.2 mmol), compound **3e** was obtained (9 mg, 31%) as yellow solid, m.p. 176–178 °C; [Found: C, 68.50; H, 3.81; N, 4.84. $C_{17}H_{11}F_2NO_2$ requires C, 68.23; H, 3.70; N, 4.68 %]; R_f (10% EtOAc/hexane) 0.33; ν_{max} (KBr) 2918, 1704, 1441, 1016 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 3.17 (3H, s, NMe), 7.05–7.11 (4H, m, Ph), 7.46–7.51 (4H, m, Ph); δ_C (100 MHz, $CDCl_3$) 169.6, 162.5 (d, $^1J_{CF}$ 250.5 Hz), 134.1, 130.9 (d, $^3J_{CF}$ 8.5 Hz), 126.5, 114.9 (d, $^2J_{CF}$ 22.0 Hz), 23.3; m/z 299 (100 $M^{\bullet+}$), 241 (35), 214 (88), 149 (43%).

4.3.6. 3-(4-Methoxyphenyl)-1-phenyl-1H-pyrrole-2,5-dione (4g).

Following procedure 4.2.1 with *N*-phenylmaleimide (17 mg, 0.1 mmol) and 1-iodo-4-methoxybenzene (46 mg, 0.2 mmol), compound **4g** was obtained (21 mg, 76%) as yellow solid, m.p. 148–149 °C (Lit.[13] 148–151 °C); [Found: C, 73.44; H, 4.83; N, 5.21. $C_{17}H_{13}NO_3$ requires C, 73.11; H, 4.69; N, 5.02 %]; R_f (10% EtOAc/hexane) 0.15; δ_H (400 MHz, $CDCl_3$) 3.91 (3H, s, OMe), 6.78 (1H, s, =CH), 7.02 (2H, d, *J* 9.0 Hz, Ph), 7.41–7.43 (3H, m, Ph), 7.50 (2H, d, *J* 7.5 Hz, Ph), 8.03 (2H, d, *J* 9.0 Hz, Ph); m/z 279 (37 $M^{\bullet+}$), 132 (46), 71 (63), 57(100%).

4.3.7. 3-(Naphthalen-1-yl)-1-phenyl-1H-pyrrole-2,5-dione (4h).

Following procedure 4.2.1 with *N*-phenylmaleimide (17 mg, 0.1 mmol) and 1-iodonaphthalene (51 mg, 0.2 mmol), compound **4h** was obtained (1824 mg, 80%) as yellow solid, m.p. 152–154 °C; [Found: C, 80.58; H, 4.52; N, 4.88. $C_{20}H_{13}NO_2$ requires C, 80.25; H, 4.38; N, 4.68 %]; R_f (10% EtOAc/hexane) 0.29; ν_{max} (KBr) 2921, 1711, 1499, 1378 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 7.02 (1H, s, =CH), 7.40–7.64 (8H, m, Ph), 7.78–8.13 (4H, m, Ph); δ_C (100 MHz, $CDCl_3$) 173.4, 168.6, 168.3, 143.1, 130.2, 129.4, 128.3,

128.2, 128.1, 128.0, 127.7, 126.9, 126.4, 125.6, 125.5, 125.3, 125.1, 124.4, 124.1, 123.4; m/z 299 (70 $M^{\bullet+}$), 270 (15), 242 (18), 179 (11), 152 (100%).

4.3.8. 1-Phenyl-3,4-di-p-tolyl-1H-pyrrole-2,5-dione (3i).

Following procedure 4.2.1 with *N*-phenylmaleimide (17 mg, 0.1 mmol) and 1-iodo-4-methylbenzene (44 mg, 0.2 mmol), compound **3i** was obtained (20 mg, 56%) as orange solid, m.p. 158–159°C; [Found: C, 81.85; H, 5.56; N, 4.15. $C_{24}H_{19}NO_2$ requires C, 81.56; H, 5.42; N, 3.96%]; R_f (10% EtOAc/hexane) 0.50; ν_{max} (KBr) 2920, 1705, 1603, 1499, 1380 cm^{-1}; δ_H (300 MHz, $CDCl_3$) 2.39 (6H, s, Me), 7.20 (4H, d, *J* 8.0 Hz, Ph), 7.45–7.56 (9H, m, Ph); δ_C (100 MHz, $CDCl_3$) 168.8, 139.2, 134.5, 130.9, 128.9, 128.7, 128.3, 128.0, 126.6, 125.1, 124.8, 20.5; m/z 353 (40 $M^{\bullet+}$), 338 (15), 149 (100), 111 (26), 85 (47), 57 (94%).

4.3.9. 1,3,4-Triphenyl-1H-pyrrole-2,5-dione (3j).[14]

Following procedure 4.2.1 with *N*-phenylmaleimide (17 mg, 0.1 mmol) and iodobenzene (40 mg, 0.2 mmol), compound **3i** was obtained (13 mg, 41%) as yellow solid, m.p. 152–154 °C (Lit.[12] 171 °C); [Found: C, 81.53; H, 4.79; N, 4.47. $C_{22}H_{15}NO_2$ requires C, 81.21; H, 4.65; N, 4.30%]; R_f (10% EtOAc/hexane) 0.48; δ_H (300 MHz, $CDCl_3$) 7.28–7.41 (7H, m, Ph), 7.49–7.55 (8H, m, Ph); δ_C (100 MHz, $CDCl_3$) 125.2, 126.8, 127.5, 127.6, 128.1, 128.9, 129.0, 130.8, 135.3, 168.5; m/z 325 (100 $M^{\bullet+}$), 296 (22), 205 (21), 178 (65%).

4.3.10. 1-Benzyl-3,4-di-p-tolyl-1H-pyrrole-2,5-dione (3k).

Following procedure 4.2.1 with *N*-benzylmaleimide (19 mg, 0.1 mmol) and 1-iodo-4-methylbenzene (44 mg, 0.2 mmol), compound **3k** was obtained (24 mg, 65%) as yellow solid, m.p. 131–132 °C (Lit.[15] 131 °C); [Found: C, 81.99; H, 5.90; N, 4.00. $C_{25}H_{21}NO_2$ requires C, 81.72; H, 5.76; N, 3.81%]; R_f

(10% EtOAc/hexane) 0.55; δ_H (300 MHz, $CDCl_3$) 2.36 (6H, s, Me), 4.79 (2H, s, CH_2), 7.14 (4H, d, *J* 7.5 Hz, Ph), 7.26–7.34 (3H, m, Ph), 7.38 (4H, d, *J* 7.5 Hz, Ph), 7.46 (2H, m, Ph); δ_C (100 MHz, $CDCl_3$) 21.5, 41.9, 125.9, 127.8, 128.7, 128.8, 129.2, 129.8, 135.5, 136.6, 140.0; 170.7; m/z 367 (100 $M^{\bullet+}$), 206(22), 91(17%).

4.3.11. 3,4-Bis(4-methoxyphenyl)-1H-pyrrole-2,5-dione (3m).

Following procedure 4.2.1 with maleimide (10 mg, 0.1 mmol) and 1-iodo-4-methoxybenzene (46 mg, 0.2 mmol), compound **3m** was obtained (15 mg, 48%) as yellow solid, m.p. 200–201 °C (Lit.[16b] 240 °C); [Found: C, 70.19; H, 5.05; N, 4.71. $C_{18}H_{15}NO_4$ requires C, 69.89; H, 4.89; N, 4.53%]; R_f (10% EtOAc/hexane) 0.17; δ_H (300 MHz, $CDCl_3$) 3.85 (6, s, OMe), 6.89 (4H, d, *J* 8.5 Hz, Ph), 7.49 (4H, d, *J* 8.5 Hz, Ph); δ_C (125 MHz, $CDCl_3$) 169.8, 159.8, 133.9, 130.4, 120.0, 113.1, 54.3; m/z 309 (7 $M^{\bullet+}$), 296 (55), 175 (100), 97 (28%).

4.3.12. 3,4-Di-p-tolyl-1H-pyrrole-2,5-dione (3n).[16a]

Following procedure 4.2.1 with maleimide (10 mg, 0.1 mmol) and 1-iodo-4-methylbenzene (44 mg, 0.2 mmol), compound **3n** was obtained (8 mg, 28%) as yellow solid, m.p. 172–174 °C; [Found: C, 78.27; H, 5.58; N, 5.20. $C_{18}H_{15}NO_2$ requires C, 77.96; H, 5.45; N, 5.05%]; R_f (10% EtOAc/hexane) 0.47; δ_H (300 MHz, $CDCl_3$) 2.38 (6H, s, Me), 7.18 (4H, d, *J* 8.0 Hz, Ph), 7.39 (4H, d, *J* 8.0 Hz, Ph), 7.55 (1H, bs, NH); δ_C (125 MHz, $CDCl_3$) 169.6, 139.2, 135.3, 128.7, 128.3, 124.6, 20.5; m/z 277 (100 $M^{\bullet+}$), 262 (31), 206 (43), 57 (7%).

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Supplementary data

Supplementary data associated with this article can be found in the online version.

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