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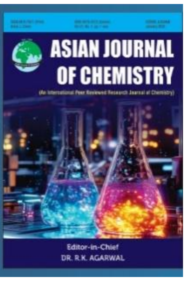

Iron(II) Chloride Catalyzed Ligand-Free Double Carbonylation of *o*-Diiodoarenes with Anilines: A Novel Synthetic Approach to Phthalimide Derivatives

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Herein, an iron-catalyzed, ligand-free method for the double carbonylation of ortho-diiodoarenes with anilines in the presence of carbon monoxide is reported. This protocol proceeds efficiently, affording *N*-substituted phthalimides with excellent selectivity and high yields. The results highlight a novel and sustainable synthetic approach to phthalimide derivatives, underscoring the potential of iron catalysis in heterocycle construction.

Keywords: Amino carbonylation, Carbon monoxide, Iron(II) chloride, *o*-Diiodoarenes, Phthalimides.

INTRODUCTION

Phthalimide compounds are extensively utilized in synthetic and medicinal chemistry owing to their diverse biological activities. They are known to modulate multiple physiological pathways, including the inhibition of peptidase, cyclooxygenase (COX) and glucosidase enzymes, as well as the blockade of the androgen receptor [1-10]. Beyond these activities, phthalimide derivatives have also been explored in the immunological and inflammatory contexts, including the management of chronic graft-versus-host disease (GVHD) and the regulation of tumor necrosis factor (TNF) signaling pathways [11-13]. Their potential in anticancer therapy has likewise been highlighted, with several derivatives exhibiting significant activity against various cancer cell lines [14,15]. In addition, the phthalimide frameworks have been reported to display antibacterial efficacy, including activity against resistant microbial strains [16-18] and continue to serve as versatile scaffolds in the medicinal chemistry research [19].

Traditional syntheses of phthalimides typically rely on the palladium-catalyzed carbonylative cyclizations of *o*-dihaloarenes or *o*-diiodobenzenes with amines under CO atmosphere [20-24]. While effective, these methods often require costly noble metals, specially designed ligands or hazardous reagents. CO-free or CO-surrogate systems have also been developed [25,26]; however, challenges remain, such as limited substrate scope, high catalyst loading and operational complexity.

Recently, iron catalysts have gained growing attention in organic synthesis due to their natural abundance, low cost and environmentally benign nature compared to noble metals such as palladium, rhodium or iridium [27-29]. Iron-based catalytic systems have been successfully applied in diverse transformations including cross-coupling, C–H activation and carbonylation reactions [30,31]. Their ability to mediate carbonylative cyclizations under relatively mild conditions makes them particularly attractive for the sustainable synthesis of heterocycles.

Inspired by these advantages and in continuation of our efforts toward developing sustainable methodologies, herein an $FeCl_2$-catalyzed ligand-free reductive carbonylation of *o*-diiodobenzenes with primary amines under a CO atmosphere is conducted. This protocol employs $FeCl_2$ as catalyst, triethylamine as base and 1,4-dioxane as solvent, providing a variety of N-substituted phthalimides in good to excellent yields under mild reaction conditions.

EXPERIMENTAL

Most of the chemical reagents were purchased from Aldrich and unless stated, they were used without further purification. Thin-layer chromatography (TLC) was performed using a

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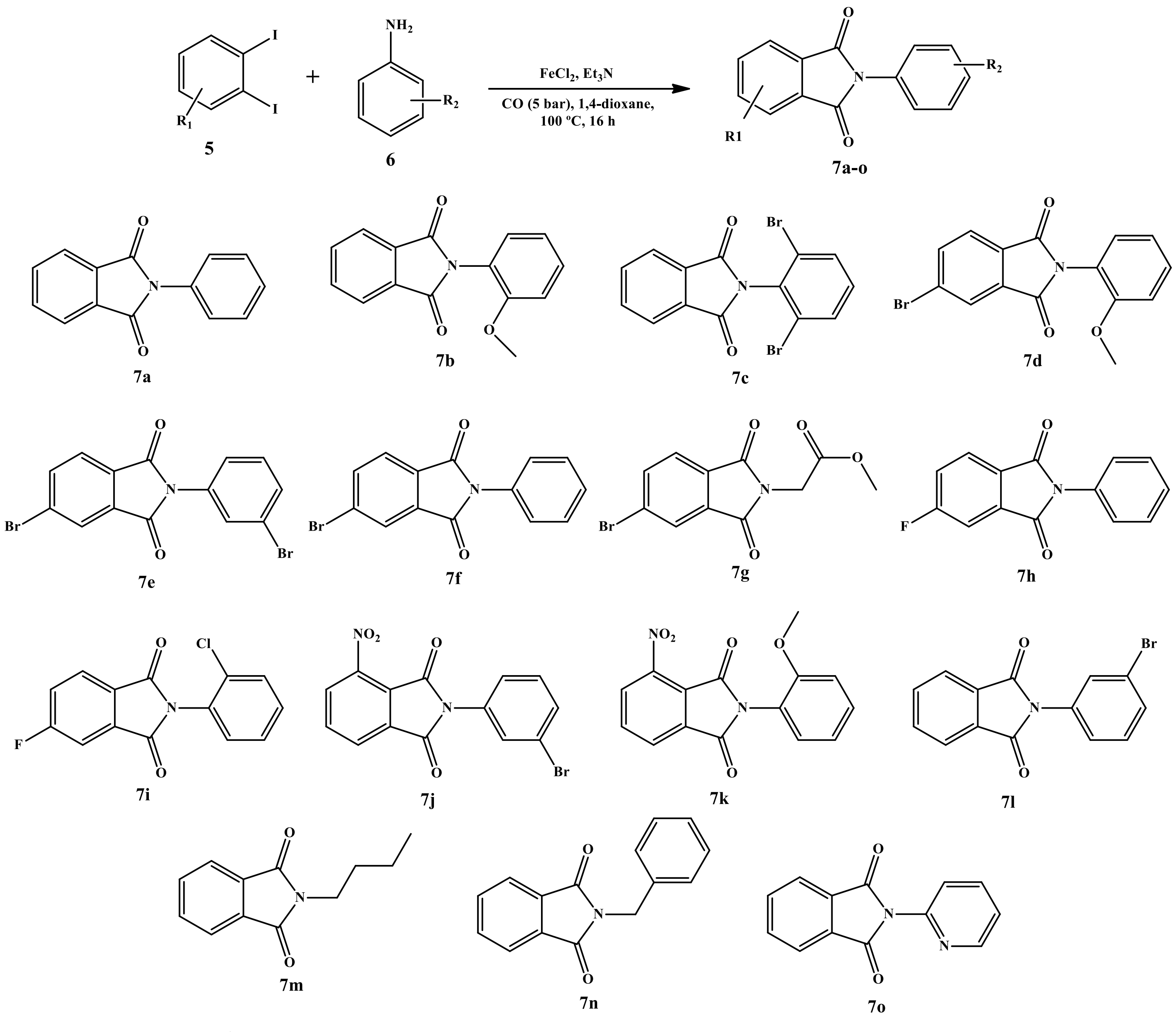
Merck 60 F_{254} precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualised using UV radiation (254 nm). Further visualisation was carried out by staining with an ethanolic solution of ninhydrin. Flash-column chromatography was performed using silica gel (100-200 mesh) with commercially available solvents. ^{1}H NMR spectra were recorded on Bruker Advance III HD, 400 and 500 MHz spectrophotometers using TMS as an internal standard. Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (0.0 ppm) and relative to the signal of chloroform-*d* (7.26 ppm). DMSO-d_6 (2.50 ppm) multiplicities were given as s (singlet); d (doublet); t (triplet); q (quartet) or m (multiplet). Coupling constants are reported as a *J* value in hertz. Melting points were recorded on a Büchi Melting Point B-545 and are uncorrected.

General procedure for synthesis of phthalimides (7a-o): To a stirred solution of aryl iodide 5 (1.0 mmol), amine 6 (1.5 mmol) and 1,4-dioxane (5 mL) within a dry steel bomb, $FeCl_2$ (10 mol%) and Et_3N (3.0 mmol) were added. The resulting mixture was then sealed and stirred at 100 ºC for 16 h under 5 bar CO pressure. The completion of the reaction was monitored using TLC and LC-MS and the reaction mixture was then cooled to room temperature. The excess solvent was concentrated under reduced pressure to give the crude product, which was further purified through silica gel (100-200 mesh size) column chromatography. Ethyl acetate or petroleum ether was used as eluent to ensure a pure compound (**Scheme-I**).

2-Phenylisoindoline-1,3-dione (7a): Off white solid; 60 mg, yield: 90%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.96 (dd, *J* = 5.5, 3.3 Hz, 2H), 7.79 (dd, *J* = 5.4, 1.5 Hz, 2H), 7.53-7.49 (m, 2H), 7.48-7.38 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 167.3, 134.41, 131.79, 131.70, 129.14, 128.13, 126.60, 123.77; LCMS (ESI-TOF) calcd. for $C_{14}H_9NO_2$ $(M+H)^+$: 224.06; found: 224.34.

2-(2-Methoxyphenyl)isoindoline-1,3-dione (7b): Pale-brown solid; 65 mg, yield: 85%; ^{1}H NMR ($CDCl_3$, 400 MHz)



Scheme-I: Substrate scope for $FeCl_2$-catalyzed phthalimide synthesis; **Reaction conditions: 5** (1.0 mmol), **6** (3.0 mmol), catalyst (10 mol %), base (3.0 mmol), solvent (10 mL), 100 ºC, 16 h δ ppm: 7.96 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.79 (dd, *J* = 5.6, 3.2 Hz, 2H), 7.45-7.41 (m, 1H), 7.27-7.24 (m, 1H), 7.09-7.04 (m, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 167.34, 155.39, 134.07, 132.23, 130.64, 129.95, 123.62, 120.83, 120.23, 112.10, 55.78; LCMS (ESI-TOF) calcd. for $C_{15}H_{11}NO_3$ $(M+H)^+$: 254.07; found: 254.05.

2-(4-Bromophenyl)isoindoline-1,3-dione (7c): Off white solid; 75 mg, yield: 85%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 8.01-7.97 (m, 2H), 7.85-7.81 (m, 2H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.25 (t, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 165.62, 134.65, 132.46, 132.0, 131.80, 131.26, 125.21, 124.17; LCMS (ESI-TOF) calcd. for $C_{14}H_7Br_2NO_2$: 379.88; found: 380.22.

5-Bromo-2-(2-methoxyphenyl) isoindoline-1,3-dione (7d): Pale-brown solid; 66 mg, yield: 82%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 8.07 (d, *J* = 1.6 Hz, 1H), 7.91 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.46-7.41 (m, 1H), 7.25-7.23 (m, 1H), 7.09-7.03 (m, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 166.56, 166.0, 155.32, 137.14, 133.90, 130.88, 130.79, 129.88, 129.05, 127.03, 125.07, 120.91, 119.95, 112.16, 55.84; LCMS (ESI-TOF) calcd. for $C_{15}H_{10}BrNO_3$: 330.98; found: 332.34.

5-Bromo-2-(3-bromophenyl)isoindoline-1,3-dione (7e): Off white solid; 70 mg, yield: 75%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.98-7.95 (m, 2H), 7.69-7.64 (m, 1H), 7.63-7.62 (m, 1H), 7.51-7.53 (m, 1H), 7.41-7.35 (m, 2H), 3.40-3.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 165.97, 165.44, 137.65, 133.10, 132.58, 131.31, 130.32, 130.0, 129.59, 129.36, 127.21, 125.23, 124.94, 122.45; LCMS (ESI-TOF) calcd. for $C_{14}H_7Br_2NO_2$: 379.88; found = 380.22.

5-Bromo-2-phenylisoindoline-1,3-dione (7f): Off white solid; 59 mg, yield: 80%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 8.09 (d, *J* = 1.6 Hz, 1H), 7.93 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.53-7.49 (m, 2H), 7.43-7.39 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 167.44, 166.58, 166.06, 137.26, 133.58, 130.48, 129.23, 127.0, 124.97, 52.74, 38.87; LCMS (ESI-TOF) calcd. for $C_{15}H_{11}NO_3$ $(M+H)^+$: 301.97, found: 302.41

Methyl 2-(5-bromo-1,3-dioxoisoindolin-2-yl)acetate (7g): Pale-brown solid; 54 mg, yield: 75%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 8.02 (d, *J* = 1.6 Hz, 1H), 7.90 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.76 (d, *J* = 8.0 Hz, 1H), 4.44 (s, 2H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 166.43, 166.90, 166.06, 137.42, 133.35, 131.35, 130.25, 129.32, 129.16, 128.29, 127.07, 126.44, 125.10; LCMS (ESI-TOF) calcd. for $C_{11}H_8BrNO_4$ $(M+H)^+$: 297.96; found: 298.31.

5-Fluoro-2-phenylisoindoline-1,3-dione (7h): Off white solid; 59 mg, yield: 85%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.79-7.75 (m, 2H), 7.52-7.48 (m, 2H), 7.46-7.39 (m, 4 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 166.07, 166.04, 163.83, 159.16, 156.52, 136.95, 136.87, 133.87, 131.22, 129.11, 128.27, 126.53, 122.78, 122.58, 119.92, 119.98, 117.57, 117.45; LCMS (ESI-TOF) calcd. for $C_{14}H_8FNO_2$ $(M+H)^+$: 242.05; found: 242.38.

2-(2-Chlorophenyl)-5-fluoroisoindoline-1,3-dione (7i): Pale-brown solid; 64 mg, yield: 82%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.82-7.78 (m, 2H), 7.59-7.56 (m, 1H), 7.49-7.40 (m, 3H), 7.36-7.33 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ ppm: 165.41, 163.14, 159.26, 156.61, 137.07, 136.99, 133.99, 133.18, 130.83, 130.61, 130.45, 129.18, 127.76, 122.92, 122.73, 120.17, 120.14, 117.84, 117.71; LCMS (ESI-TOF) calcd. for $C_{14}H_7ClFNO_2$ $(M+H)^+$: 276.01; found: 276.33.

2-(3-Bromophenyl)-4-nitroisoindoline-1,3-dione (7j): Pale-brown solid; 70 mg, yield: 76%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 8.24 (dd, *J* = 7.6, 0.8 Hz, 1H), 8.18 (dd, *J* = 8.0, 0.8 Hz, 1H), 8.02 (t, *J* = 7.6 Hz, 1H), 7.63 (d, *J* = 1.6 Hz, 1H), 7.58−7.56 (m, 1H), 7.42-7.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 164.38, 161.48, 145.48, 135.94, 133.50, 132.07, 131.76, 130.43, 129.49, 129.08, 127.55, 125.06, 123.14, 122.52; LCMS (ESI-TOF) calcd. for $C_{14}H_7N_2BrO_4$ $(M+H)^+$: 346.96; found: 346.99.

2-(2-Methoxyphenyl)-4-nitroisoindoline-1,3-dione (7k): Off white solid; 64 mg, yield: 81%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 8.20 (d, *J* = 7.6 Hz, 1H), 8.15 (d, *J* = 8.0 Hz, 1H), 7.97 (t, *J* = 7.6 Hz, 1H), 7.47-7.43 (m, 1H), 7.26-7.24 (m, 1H), 7.09-7.04 (m, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 164.86, 161.83, 155.24, 145.36, 135.48, 134.24, 131.18, 129.81, 128.68, 127.40, 123.90, 120.93, 119.5, 112.18, 55.88; LCMS (ESI-TOF) calcd. for $C_{15}H_{10}N_2O$ $(M+H)^+$: 299.06; found: 299.10.

2-(3-Bromophenyl)isoindoline-1,3-dione (7l): Off white solid; 73 mg, yield: 80%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.98-7.94 (m, 2H), 7.83-7.78 (m, 2H), 7.65–7.64 (m, 1H), 7.55-7.52 (m, 1H), 7.44-7.35 (m, 2H), 3.40-3.38 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 166.79, 134.6, 132.92, 131.53, 131.10, 130.27, 129.48, 125.06, 123.89, 122.40; LCMS (ESI-TOF) calcd. for $C_{14}H_8BrNO_2$ $(M+H)^+$: 301.97, found: 301.94.

2-Butylisoindoline-1,3-dione (7m): Off white solid; 48 mg, yield: 79%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.85-7.81 (m, 2H), 7.72-7.69 (m, 2H), 3.70 (t, *J* = 7.2 Hz, 2H), 1.70-1.62 (m, 2H), 1.41-1.32 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 2H); ^{13}C NMR (400 MHz, $CDCl_3$) δ ppm: 168.45, 133.79, 132.16, 123.10, 37.77, 30.61, 20.05, 13.61; calcd. for $C_{12}H_{13}NO_2$ $(M+H)^+$: 204.09; found = 204.03.

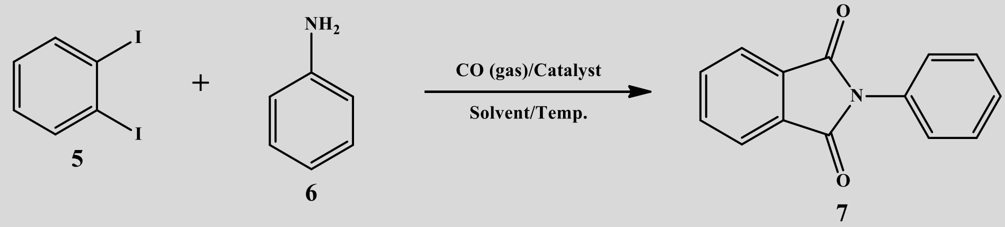
2-Benzylisoindoline-1,3-dione (7n): Off white solid; 61 mg, yield: 85%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 7.86-7.81 (m, 2H), 7.71-7.67 (m, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.33-7.23 (m, 3H), 4.84 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 168.01, 136.34, 133.94, 132.10, 128.64, 127.79, 123.31, 41.58; LCMS (ESI-TOF) calcd. for $C_{15}H_{11}NO_2$ $(M+H)^+$: 238.08; found = 238.06.

2-(Pyridin-2-yl)isoindoline-1,3-dione (7o): Off white solid; 48 mg, yield: 70%; ^{1}H NMR ($CDCl_3$, 400 MHz) δ ppm: 8.7 (d, *J* = 5.2 Hz, 1H), 8.0-7.87 (m, 2H), 7.83-7.80 (m, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.39-7.36 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ ppm: 166.63, 136.34, 149.64, 146.16, 138.25, 134.58, 131.74, 123.94, 123.42, 122.04; LCMS (ESI-TOF) calcd. for $C_{13}H_8N_2O_2$ $(M+H)^+$: 225.06; found: 225.25.

RESULTS AND DISCUSSION

While investigating the effect of solvents, it is found that solvent composition plays a pivotal role in determining the reaction outcome. Several solvents such as acetonitrile, THF, DMF, DMSO, toluene, PEG-400, H_2O and 1,4-dioxane were tested. Among them, 1,4-dioxane proved to be the most suitable solvent when $FeCl_2$ was used as catalyst (Table-1, entries 1-16).

TABLE-1
OPTIMIZATION FOR THE SYNTHESIS OF PHTHALIMIDES



S. No.	Catalyst (10 mol%)	Base/additive	Solvent	Temp. (°C)	Time (h)	Yield[a] (%)
1	$FeCl_2$	Et_3N	1,4-Dioxane	RT	16	Traces
2	$FeCl_2$	Et_3N	1,4-Dioxane	50	16	30
3	$FeCl_2$	Et_3N	1,4-Dioxane	100	16	89
4	$FeCl_2$	Et_3N/Na_2CO_3	1,4-Dioxane	50	16	35
5	$FeCl_2$	Na_2CO_3	1,4-Dioxane	100	16	10
6	$FeCl_2$	DABCO	1,4-Dioxane	100	16	Traces
7	$FeCl_2$	DBU	1,4-Dioxane	100	16	40
8	$FeCl_2$	Et_3N	Acetonitrile	100	16	Traces
9	$FeCl_2$	Et_3N	Toluene	100	16	50
10	$FeCl_2$	Et_3N	THF	100	16	–
11	$FeCl_2$	Et_3N	DMF	100	16	10
12	$FeCl_2$	Et_3N	PEG-400	100	16	–
13	$FeCl_2$	Et_3N	H_2O	100	16	–
14	$Fe(CO)_5$	Et_3N	1,4-Dioxane	100	16	Traces
15	Fe powder	Et_3N	1,4-Dioxane	100	16	6
16	$FeCl_3$	Et_3N	1,4-Dioxane	100	16	8

[a]Isolated yield.

A range of organic and inorganic bases were investigated to improve the product yield. While inorganic bases such as Na_2CO_3 provided low conversions, hindered amines such as DBU, DABCO and Et_3N/Na_2CO_3 offered the moderate results. Among them, Et_3N merged as the most effective, affording the highest yield (89%) under optimized conditions (Table-1, entry-3). The reaction temperature was also investigated and 100 ºC was identified as the optimum. At temperatures above 100 ºC, the yield slightly decreased, likely due to dehalogenation of 1,2-diiodobenzene. Similarly, CO pressure played a critical role in determining the reaction efficiency; a pressure of 5 bar afforded the best results, yielding 89% of the target product after 16 h.

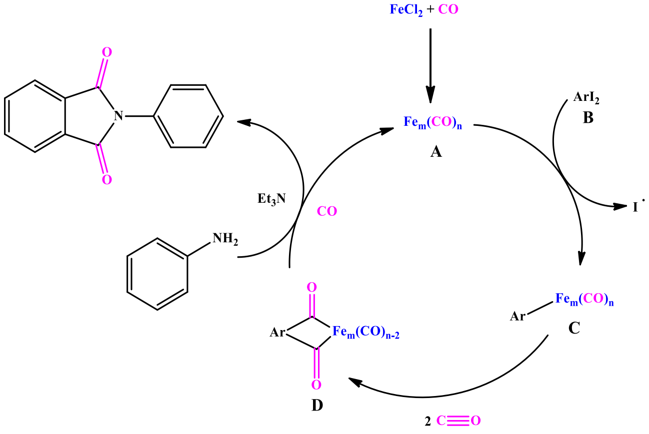
Catalyst screening demonstrated that $FeCl_2$ significantly outperformed other iron sources such as $Fe(CO)_5$, Fe powder, and $FeCl_3$, which produced only traces to low yields (Table-1, entries 14–16). These results confirmed $FeCl_2$ as the most suitable iron catalyst for this transformation. Therefore, the optimized reaction conditions were established as follows: $FeCl_2$ (10 mol%), Et_3N (3.0 mmol), 1,4-dioxane (10 mL), CO pressure 5 bar, temperature 100 °C, and 16 h reaction time.

Using these optimized conditions, the substrate scope was explored. A wide range of diiodoarenes and arylamines underwent smooth carbonylative cyclization to give diverse 2-phenylisoindoline-1,3-diones in good to excellent yields. The model reaction between 1,2-diiodobenzene and aniline afforded the target phthalimide in 89% yield.

Substituted 1,2-diiodobenzenes coupled efficiently with arylamines bearing electron-donating and electron-withdrawing groups, generally affording good to excellent yields. Transformations of *ortho*- and *meta*-substituted aniline derivatives, such as *m*-bromoaniline, *o*-methoxyaniline and *m*-chloroaniline, furnished high yields of the corresponding N-substituted isoindole-1,3-dione derivatives (**7e**, **7b** and **7i**). The reaction with *o*-dibromoaniline gave a moderately good yield, indicating that increased steric hindrance from *ortho*-substitution slightly affects the efficiency (**7c**). Aliphatic amines also proved efficient, delivering the desired products in excellent yields (**7m**). Overall, the methodology tolerates aromatic, aliphatic and heteroaromatic amines, providing moderate to excellent yields across a wide range of substrates (**7a-o**). The scope of this methodology was explored with *o*-diiodobenzenes; other dihalogenated arenes such as *o*-dibromo- and *o*-dichloroarenes were not attempted under the present iron-catalyzed conditions, as these substrates are known to be significantly less reactive in reductive carbonylation reactions.

Although the present study focused on small-scale reactions, the mild reaction conditions, operational simplicity, and use of inexpensive $FeCl_2$ suggest that the methodology could potentially be amenable to larger-scale synthesis and industrial application.

Mechanism: Based on the experimental findings and existing literature [32-35], a plausible reaction mechanism is proposed, as illustrated in **Scheme-II**. Under standard conditions, the interaction between $FeCl_2$ and CO gas leads to the *in situ* generation of $Fe_m(CO)_n$ (A). This is followed by the rapid oxidative addition of aryl iodide (B) to A, resulting in the formation of organoiron complex C. Subsequently, intramolecular CO migratory insertion occurs, yielding organoiron complex D. A nucleophilic attack by the amine on complex D facilitates the formation of the desired phthalimide product, while concurrently regenerating the catalytically active species A in the presence of carbon monoxide.



Scheme-II: A plausible reaction mechanism for phthalimides

Conclusion

In summary, a straightforward and cost-effective iron-catalyzed double carbonylation method is established for the synthesis of *N*-phthalimide, achieving moderate to good yields. To the best of our knowledge, this represents the first ligand free and palladium-free metal-catalyzed approach for the double carbonylation of o-diiodoarenes with anilines to generate *N*-phthalimide derivatives.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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