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Iron-catalyzed decarboxylative radical addition to chiral azomethine imines upon visible light

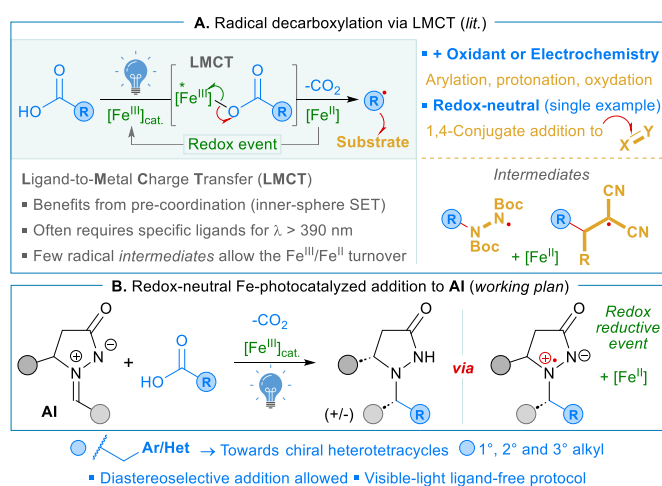
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Herein, we disclose an eco-efficient redox-neutral iron-catalyzed decarboxylative radical addition to chiral azomethine imines upon visible light (427 nm) giving cyclic hydrazine derivatives with dr ranging from 82:18 to >96:4. This earth-abundant metal promoted sequence proceeds efficiently under ligand-free conditions based on a LMCT process and open a route to new chiral heterocycles.

In pursuance of more sustainable chemistry, the use of photocatalysts derived from earth-abundant 3d transition-metals has witnessed a renewed interest, a field in which iron complexes stand out due to their low cost and environmentally benign impact.¹ However, iron-based photo-active complexes suffer from very short charge-transfer-state lifetimes.^{1b} Their use in synthetically useful outer-sphere single-electron transfer (SET) processes^{1b} has thus been limited by this rapid excited-state deactivation, explaining why expensive but potent Ru-, Ir-complexes and some redox organophotocatalysts have been widely exploited in this domain.² Recently, the catalytic ligand-to-metal charge-transfer (LMCT) excited states approach, using non-precious Cu-, Ce-, Ni- and, especially, Fe-complexes, afforded a promising alternative tactic,³ as nicely illustrated by the versatile and synthetically useful radical decarboxylation methodologies exploiting highly distributed carboxylic acid precursors.^{3c,4} This approach benefits from a pre-coordination of the carboxylate function to an Fe(III) complex at the ground state, allowing the population of the LMCT excited states upon absorption of light followed by the homolysis and extrusion of CO₂, generating thereby the reduced Fe(II) complex (Scheme 1A).^{3b-d} This inner-sphere SET mechanism overcomes the short-lived excited state of Fe-complexes, and avoids the required thermodynamically-driven redox potentials match of the "regular" photoredox catalysis based on outer-sphere SET.^{3b-d} Then, an orthogonal functional group differentiation can occur and oxidative sensitive substrates can be better tolerated.



Scheme 1. Radical decarboxylation by LCMT approaches.

Since 2019, following the pioneering contribution of Sugimori and Yamada in 1986 with a stoichiometric amount of Fe complexes,⁵ the groups of Jin, Wang and Juli a-Hern andez achieved Fe-photocatalyzed decarboxylative heteroaromatic C-H alkylations (Minisci type C-C bond forming reactions).⁶ Recently, West, Guo and Xia reported an elegant hydrofluoroalkylation of alkenes from fluorocarboxylic acids, while Li and Zeng developed an Fe-catalyzed Giese or imination reaction.⁷ However, these contributions required the use of an oxidant, an electrochemical approach, or the use of additives to close the Fe(II)/Fe(III) catalytic cycle. Jin *et al.* published a single example of the radical-conjugate addition to azodicarboxylates and malononitriles, in which these specific substrates proved adapted for a redox-neutral sequence (Scheme 1A).⁸ The presence of heterocyclic bidentate pyridine-ligands in these pioneering investigations allowed the use of blue LED as visible light (wavelength > 390 nm). Recently,⁹ the LMCT Fe-catalyzed decarboxylative sequence was extended to the construction of C-H,^{9d} C-O,^{9a, 9g, 9i} C-N,^{9b, 9f, 9h} C-Cl^{9e, 9j}, C-S^{7d,9k} and C-F bonds.^{9c}

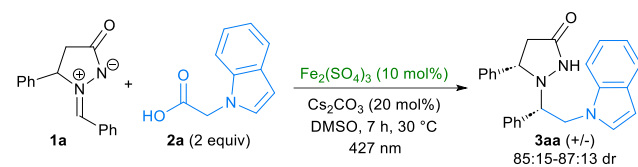
The addition of radical species to dipoles is an active field of research,¹⁰ among which the transformation of azomethine imines emerged recently with the seminal contributions of Xiang, Chen, Yang, Paix o, Cao, Chan and Li, who developed Ru-

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, Ir- or organophotoredox catalytic methodologies.¹¹ Our group proposed a complementary sequence based on redox-active *N*-(acyloxy)phthalimide esters upon a cathodic reductive decarboxylation which allowed an unprecedented highly stereoselective addition of radicals to chiral azomethine imines (**1a**, Scheme 1B).¹² Unfortunately, the use of an oxidative decarboxylation of native carboxylic acids, and hence a more atom-economy approach, failed in our hand. We supposed that the closely related redox potentials of the azomethine imine ($\approx E_{\text{ox}} = +1.5$ V vs SCE in DMF), the hydrazine addition product ($\approx E_{\text{ox}} = +1.1$ V)¹² and the carboxylate ($\approx E_{\text{ox}} = +1.3$ V)¹³ led to unselective oxidative decomposition events. We report herein a redox-neutral iron-catalyzed decarboxylative process upon visible light which overcomes this limitation. While extending the scope for the diastereoselective addition of radicals to chiral azomethine imines, this approach also affords new opportunities for the synthesis of azaarenes (Scheme 1B).

Table 1. Proof of principle and optimization.^a



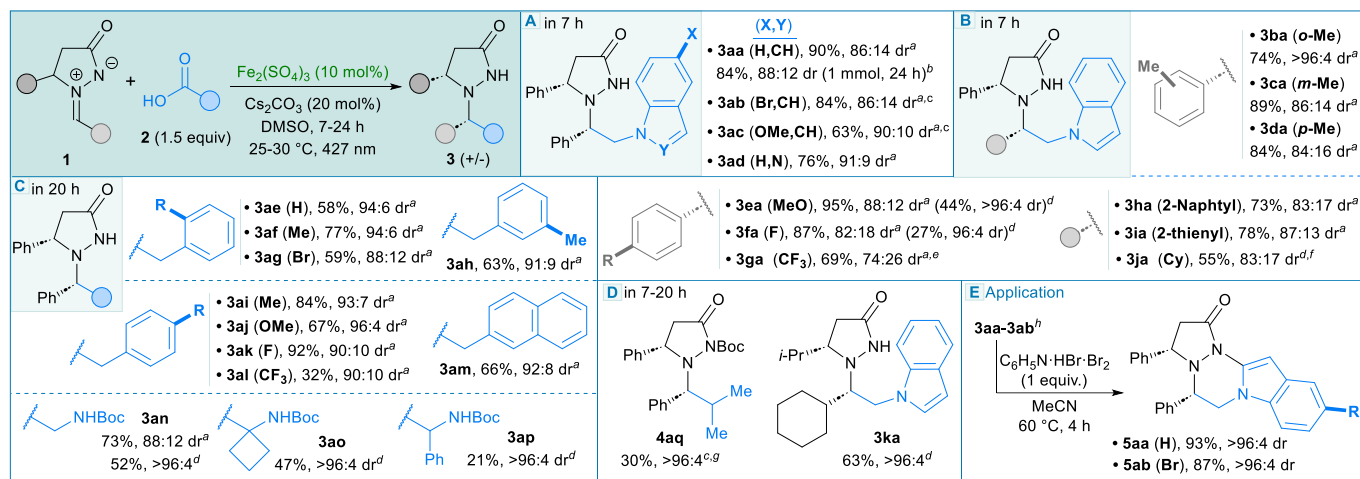
| Entry | Deviation from the standard conditions | Yield [%], dr 3aa |
|-------|--|---|
| 1 | None | 91 (82) ^b , 86:14 ^b |
| 2 | No irradiation or no Fe ₂ (SO ₄) ₃ | <5, - |
| 3 | no Cs ₂ CO ₃ base | 84 (75) ^b , 87:13 |
| 4 | MeCN as solvent instead of DMSO | 67, - |
| 5 | (CH ₂ Cl) ₂ as solvent instead of DMSO | 26, - |
| 6 | Carboxylic acid 2a (1.5 equiv.) | 95, 86:14 |
| 7 | Carboxylic acid 2a (1.0 equiv.) | 45, - |
| 8 | Fe ₂ (SO ₄) ₃ ·H ₂ O (10 mol%), 2a (1.5 equiv.) | 90, - |
| 9 | Fe(acac) ₃ (10 mol%), 2a (1.5 equiv.) | 89, - |
| 10 | Fe(NO ₃) ₃ ·H ₂ O (10 mol%), 2a (1.5 equiv.) | 81, - |
| 11 | Fe ₂ (SO ₄) ₃ (2 mol%), 2a (1.5 equiv.) | 84 (76) ^c , 85:15 |
| 12 | 450–455 nm instead of 427 nm | 89 (86) ^c , 87:13 |
| 13 | 525–530 nm instead of 427 nm | 62, - |
| 14 | 405 nm | 81 (76) ^c , 85:15 |
| 15 | 405 nm, no Fe ₂ (SO ₄) ₃ | 52, - |
| 16 | 405 nm, no Fe ₂ (SO ₄) ₃ , no Cs ₂ CO ₃ base | 6, - |

Reaction conditions: carried out with **1a** (0.2 mmol), **2a** (1.5-2.0 equiv) in DMSO (0.1 M) under nitrogen at 25-30 °C (stabilized by a fan) with a LED lamp. Yield [%] of the mixture of diastereoisomers determined on the crude mixture by ¹H NMR with Bn₂O as internal standard. Diastereoisomeric ratio (dr) determined on the crude when possible or after a quick purification on silica gel. ^bUnder bracket, isolated yield after purification by column chromatography.

Inspired by the elegant recent development of iridium-mediated decarboxylative radical additions of *N*-indole acetic acid precursors to C(sp²),¹⁴ while anticipating the high potential for further transformations of this bio-valuable azaarene (*vide infra*), we investigated the reaction between azomethine imine **1a** and the acid **2a** in DMSO as solvent (Table 1). To our delight, in the presence of Fe₂(SO₄)₃ (10 mol%) and cesium carbonate (20 mol%) upon blue LED irradiation (427 nm), a smooth

decarboxylative addition took place to afford the product **3aa** in 91% NMR yield with a diastereoisomeric ratio (dr) of 86:14 (entry 1) in only 7 hours. It turned out that the dr was not easily measured on the crude NMR spectra due to overlapping of signals (although dr was estimated by HPLC on both the crude and purified mixture), but a column purification addressed this issue giving the diastereoisomeric mixture of **3aa** in an 82% isolated yield. Control experiments demonstrated that no reaction occurred without irradiation or iron-photocatalyst (entry 2), and the base (20 mol% of Cs₂CO₃) allowed a slight improvement in yield from 84% to 91% (entry 3). DMSO proved to be the best solvent (*versus* MeCN and ClCH₂CH₂Cl, entries 4-5) and the amount of indole-carboxylic acid **2a** could be decreased from 2 to 1.5 equivalents (95% of **3aa**), while one equivalent led to a slower process (entries 6-7). Other iron(III) complexes could be used (90-81% yields, entries 8-10), but Fe₂(SO₄)₃ turned out to be the best photocatalyst, and a charge of only 2 mol% furnished the product **3aa** with a slight decrease in yield of 84% (entry 11). At that stage, two observations are worthy of note. First of all, the photo-mediated transformation occurs at irradiation of 450-455 nm (89%, entry 12), and even at 525-530 nm (62%, entry 13) albeit in a slower rate, even without external ligands.^{5,6a, 6b} Importantly, the formation of **3aa** took place in 52% NMR yield at 405 nm, even without Fe-photocatalyst but in the presence of Cs₂CO₃ as base in DMSO (entries 15-16, see SI). While being less efficient than the Fe-catalyzed process (entry 14), these interesting metal-free conditions would deserve a mechanistic insight to be fully understood. As a preliminary hypothesis, the 405 nm LED with more light-energy may trigger DMSO decomposition towards radical species allowing a HAT-mediated decarboxylative process.¹⁵

Then, we probed the scope of this iron-catalyzed decarboxylative process (Scheme 2A). The model reaction was carried out in 7 hours to give **3aa** in 90% isolated yield, and was easily scaled-up to 1 mmol scale in 24 hours with 5 mol% of the iron-complex in 84% isolated yield (**3aa**). Substituted *N*-indole and *N*-indazole acetic acid precursors **2b-d** led to the addition products **3ab-3ad** in 84%, 63% and 76% yields respectively with dr ranging from 84:16 to 90:10, whose structures **3ab-ac** (main diastereoisomer) were ascertained by X-Ray analyses. Azomethine imines **1b-i**, having various aromatic pendant on the iminium moiety, were easily transformed regardless to their substitution pattern into the corresponding *ortho*- **3ba**, *meta*- **3ca** and *para*-phenyl products **3da-fa**, as well as naphthyl **3ha** and 2-thienyl **3ia** with good to excellent yields (73-95%) and 82:18 to >96:4 dr (Scheme 2A), in contrast to the *p*-CF₃ derivatives **1g** affording product **3ga** in lower 74:26 dr and 69% yield. Even the azomethine imine **1j** flanked by an aliphatic cyclohexyl moiety was transformed into product **3ja** in 55% yield (main diastereoisomer) and 83:17 dr. Interestingly, the radical-based additions were also achieved with various benzyl-derived carboxylic acids.

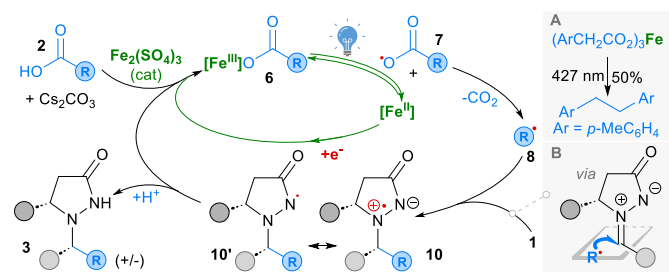


Scheme 2. Scope and limitation. Reaction conditions: carried out with **1** (0.2-1 mmol), **2a** (1.5 equiv) in DMSO (0.1 M) under nitrogen at 25-30 °C (temperature stabilized by a fan) with a blue LED during 7-20 hours. Isolated yield (%) of the main product, diastereoisomer or the mixture of stereoisomers when separation by column chromatography was not feasible. Diastereoisomeric ratio (dr) determined on the crude when possible or after a purification on silica gel. ^aIsolated yield of the mixture of non-separated diastereoisomers after column chromatography. ^bWith Fe₂(SO₄)₃ (5 mol%). ^cStructures **3ab** (CCDC2340999), and **3ac** (CCDC2341000) were ascertained by X-ray analyses. ^dIsolated yield of the main diastereoisomer after purification by column chromatography. ^e75:25 dr ratio was measured by ¹⁹F NMR on the crude reaction mixture. ^f83:17 dr for **3ja** and 93:7 dr for **3ka** measured on the crude by ¹H NMR. ^g30% overall yield after NBoc protection due to instability of the NH product **3aq** (obtained with an estimated NMR yield of 59%, and >90:10 dr by ¹H NMR). ^hReaction carried out from a mixture of diastereoisomers **3aa** (95:5 dr) and **3ab** (86:14 dr).

Upon a longer reaction time (Scheme 2C, 20 h), benzylated products **3ae-ak** and **3am** were obtained in higher diastereoselectivities (88:12 to 96:4 dr) and descent to good yields (58-92%). A limitation was met with the electron-poor *para*-CF₃ benzyl derivative **2l**, likely generating a less nucleophilic radical species, which afforded the addition product **3al** in only 32% yield and 90:10 dr. Worthy of note, this iron-photocatalyzed decarboxylation nicely complements our previous approach with redox active esters (RAE), as long as RAE derived from *N*-indole acetic acid **2a** or the benzylic analogue **2e** led to an extensive competitive dimerization event upon electrochemical conditions.¹² NBoc glycine **2n** or cyclobutyl amino acid **2o** -via a tertiary radical- afforded amino-derivatives **3an** (73%) and **3ao** (47%) in good to excellent dr ranging from 88:12 to >96:4. Although, the phenylalanine NBoc precursor allowed a radical decarboxylation sequence to occur, a mixture of 4 diastereoisomers was estimated on the crude NMR mixture, among which only the major one **3ap** could be isolated pure in 21% after column chromatography. On the other hand, the isopropyl derivative **4aq** could be synthesized in 30% yield (>96:4 dr), but after a NBoc protection due to instability issues during purification of the NH precursor **3aq**. The azomethine imine **1k** with only alkyl substituents was also transformed into product **3ka** in 63% yield and excellent 93:7 dr.

To highlight the potential of these indole derived precursors (Scheme 2E), we developed an efficient cyclization sequence of **3aa-ab**, by means of pyridinium tribromide, to open a route to

original tetracyclic products **5aa-ab**, obtained in 87-93% as a single stereoisomer after column chromatography.



(A) Irradiation at 427 nm, 7h, rt, DMCO (0.1M). (B) Front-face approach (less hindered) of the radical **8** was proposed. (C) In the presence TEMPO (3 equiv) a complete inhibition occurred and TEMPO adduct with the putative IndoleN-CH₂* radical was observed by HRMS ([M+H]⁺: 297.2119).

Scheme 3. Speculated catalytic cycle.

Preliminary mechanistic investigations were undertaken (Scheme 3 and see SI). The introduction of TEMPO, as radical trap, completely inhibited the formation of product **3aa**, and the (CH₂)₃(CMe₂)₂NO-CH₂MIndole adduct was found by HRMS analysis. In order to probe the possible inner-sphere SET mechanism, subsequent to the LMCT event, the iron-complex (*p*-MeC₆H₄CH₂CO₂)₃Fe was synthesized, according to literature procedures, and characterized by mass spectrometry analysis before being submitted to 427 nm irradiation.^{9h,9g} The dimer (*p*-MeC₆H₄CH₂)₂ was thus obtained in an estimated 50% NMR yield, likely via the formation of a benzylic radical species (Scheme 3A). Indeed, it was measured that complex (*p*-

MeC₆H₄CH₂CO₂)₃Fe displays UV-vis absorption spectra in the area of 427 nm. Based on literature precedents,⁹ and the recent insightful contribution of Guérinot *at al.*,^{9g} we assume that a ligand-free decarboxylation occurs through a LMCT process upon irradiation of the *in situ* formed iron(III) carboxylate intermediate **6** (likely facilitated by a small amount of Cs₂CO₃, although the base is not mandatory, see Table 1), allowing the addition of the obtained nucleophilic radical **8** to the less shielded face of the chiral azomethine imine **1** (*via* model **B**). The generated Fe(II) would be re-oxidized into Fe(III) during the expected facile reduction of the electron deficient radical cation intermediate **10**, or the amidyl **10'** derived thereof. The formation of product **3** would involve a final protonation step by the carboxylic acid **1**, closing thereby the catalytic cycle and regenerating the carboxylate species **6**. However, despite an intermittent on/off irradiation experiment, proving the necessity to use light to promote the reaction, we cannot rule out a rapid chain radical mechanism whereby intermediate **10** would promote a HAT process (see SI).^{9c} At that stage, the possible formation of a pre-complex between a ferric(III) entity like **6** and the azomethine imine **1**, favoring a radical-based process, cannot be excluded neither. These mechanistic aspects are currently under investigation in our laboratory.

In conclusion, we highlighted an efficient redox-neutral iron-photocatalyzed decarboxylative and stereoselective radical addition to chiral azomethine imines, proceeding under convenient ligand-free Fe-catalyzed LMCT process. The potential of this radical based transformation making use of an earth-abundant metal as photocatalyst is expected to be applicable to other dipole derivatives.

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Conflicts of interest

There are no conflicts to declare.

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