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Graphical Abstract

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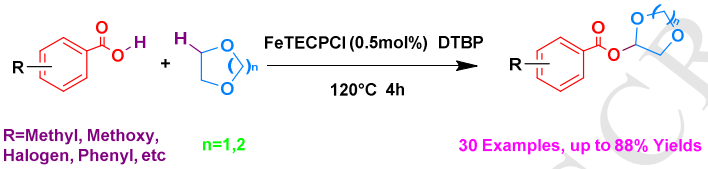

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Iron porphyrin-catalyzed C(SP^3) - H activation for the formation of C- O bond via cross-dehydrogenative coupling of cycloether and aromatic acid

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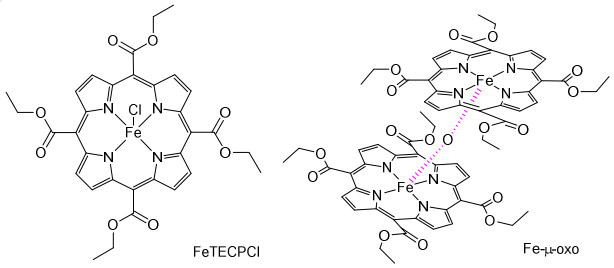
ABSTRACT

An efficient cyclic ether benzoxylation was achieved by using iron porphyrin as the catalyst and di-*tert*-butyl peroxide oxidant. The benzoic acid substrates bearing electron donating or withdrawing groups could react with cyclic ether smoothly to afford the desired products. It was found iron porphyrin catalyzed oxidative C(sp^3)- H activating esterification had the advantage of short reaction time and low catalyst loading. The reaction had been proved to proceed via a radical process.

1. Introduction

The development of efficient, convenient, straightforward and environmentally benign methods for C- X (C, O, N, etc.) bonds formation has attracted continuous interest in recent years[1]. Cross-dehydrogenative coupling (CDC) reaction plays an important role for the construction of C- X bond, in which transition metals such as palladium[2], ruthenium[3], nickel[4], indium[5] are the most commonly used catalysts. An alternative catalyst is iron. Since the first report of $FeCl_2$ catalyzed CDC reactions in 2007[6]. Lots of effort has been devoted to the iron catalyzed CDC reactions[7]. It demonstrated that iron catalyst was available for C-O bond formation by the coupling of C(sp3)-H with O(sp3)-H[8] or O(sp2) - H[9] and C(sp2) - H with O(sp2) - H[10] via CDC reactions. The direct esterification of a- C(sp3) - H bonds is one of the most important application of CDC reaction, which may prepare some biologically active ester of practical usage in medicine like artemisinin[11]. Compared to conventional esterification reactions, the CDC reaction has the advantage of using alkane instead of alcohol substrates. In 2014, Han et al. had reported the iron-catalyzed oxidative CDC esterification of cyclic ether C- H bonds with carboxylic acids, the reaction was carried out at 20 mol% catalyst loading for 24 hours at temperature 120 ^{o}C[12]. The reaction time could be further reduced to 12 hours by using copper catalyst[13].



Scheme 1. Molecular Structures of FeTECPCl and Fe-μ-oxo

Metalloporphyrins are a class of versatile catalysts[14]. It shows good catalytic activity in many reactions such as olefin oxidation[15], cyclopropanation[16], olefination[17], asymmetric synthesis[18], three- component reactions[19] and C- H bond insertion reactions[20]. Although there are many reports on metalloporphyrin catalyzed reactions, the use of metalloporphyrin catalysts in CDC reactions is scarce. In 2014, metal porphyrin was firstly used in catalytic CDC reaction which enables direct activation of two different sp3 C-H bonds of isoquinoline and nitromethane under solvent-free conditions[21]. Two years later, Guo and his co-workers had also reported the application of metal porphyrin in CDC reaction[22], in which they had successfully achieved aerobic oxidative coupling of terminal alkynes. More recently, our group has also reported the esterification of C(sp^3) - H via copper-porphyrin catalyzed CDC reaction[23]. To continue our efforts in the metalloporphyrin catalyzed CDC reactions, we here wish to report the CDC esterification of cyclic ether by using iron chloro-5,10,15,20-tetra-(ethoxylcarbonyl) porphyrin catalyst (Scheme 1, FeTECPCl) and TBHP oxidant. The reaction completed in 4 hours with catalyst loading only 0.5%, showing iron porphyrin was efficient in this kind of reaction.

2. Result and discussion

X-ray diffraction analysis showed that the structure of FeTECPCl had a formulation unit in the cell in the orthorhombic P-1 space group, and no solvent molecule was crystallized. All crystallographically independent atoms are located in the usual position, except for the iron atom, which is located in the center of the inversion of the crystal. The 24-membered macrocyclic core of porphyrin is coplanar, and the five-coordinated iron atom is located at the center of this almost completely flat porphyrin macrocycle and forms a tetrahedron with four nitrogen atoms and one chlorine atom (Scheme 2).

The Fe–N bond lengths range from 2.058(4) to 2.066(4) Å with an average value of 2.063(4) Å, the Fe-Cl bond length is 2.2188(15) Å, which is weaker interaction than the bond of metal iron and nitrogen atom (Table 1). The bond between the four nitrogen atoms (N1, N2, N3 and N4) and the iron atom and the chlorine atom is 99.60(12), 104.11(12), 101.46(12), 104.16(12).

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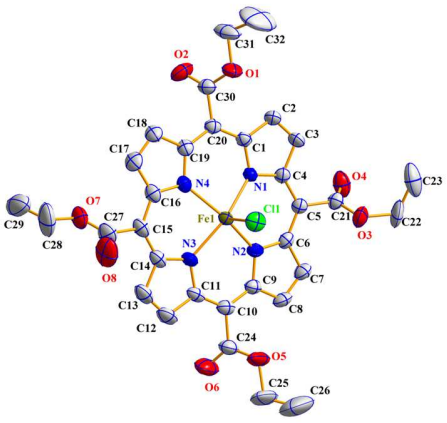


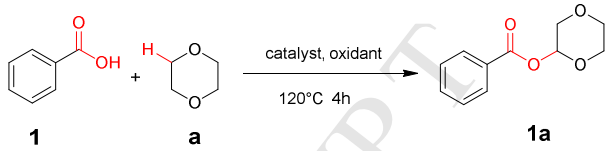
Bor

N1

N2-Fe1-Cl1	104.11(12)	N3-Fe1-N2	87.56(16)
N3-Fe1-Cl1	101.46(12)	N4-Fe1-N1	104.16(12)
N4-Fe1-Cl1	104.16(12)	N4-Fe1-N2	86.76(16)
N2-Fe1-N1	156.29(16)	N4-Fe1-N3	154.38(16)

Initially, the intermolecular oxidative reaction of benzoic acid (**1**) with 1,4-dioxane (**a**) was chosen as a model reaction to determine the optimum conditions. We commenced our investigation in the presence of FeTECPCl as the catalyst and DTBP as the oxidant under a solvent-free condition. At first, we discussed the quantity of catalyst from 0.05 to 1.0 mol %, the most efficiency of this transformation was 0.5 mol % (Table 2, entries 1-4). No significant improvement in the product yield was observed with 3 equiv of the DTBP (Table 2, entry 5). The effects of other oxidants and catalysts on the reaction was also investigated to pursue the higher yield (Table 2, entries 6-14). Unfortunately, we did not get satisfactory results. The yield decreased when the reaction was performed at 100 ºC (Table 2, entries 15). No significant improvement in the yield was observed when the reaction was conducted at 140 ºC (Table 2, entries 16). Note that the product **1a** was not observed in the absence of catalyst or oxidant (Table 2, entries 17-18).

Table 2
Optimization of Reaction Conditions[a]



1 **a** **1a**

Entry	Catalyst (amt, %)	Oxidant	Yield %[b]
1	FeTECPCl (0.05)	DTBP	32
2	FeTECPCl (0.25)	DTBP	61
3	**FeTECPCl (0.5)**	**DTBP**	**74**
4	FeTECPCl (1.0)	DTBP	59
5	FeTECPCl (0.5)	DTBP[c]	73
6	FeTECPCl (0.5)	$PhI(AcO)_2$	NR
7	FeTECPCl (0.5)	PhIO	NR
8	FeTECPCl (0.5)	*m*-CPBA	NR
9	FeTECPCl (0.5)	H_2O_2[d]	NR
10	FeTECPCl (0.5)	TBHP	Trace
11	FeTPP (0.5)	DTBP	15
12	MnTECPCl (0.5)	DTBP	Trace
13	CoTECP (0.5)	DTBP	Trace
14	$FeCl_3$ (0.5)	DTBP	NR
15	FeTECPCl (0.5)	DTBP	25[e]
16	FeTECPCl (0.5)	DTBP	73[f]
17	FeTECPCl (0.5)		NR
18		DTBP	NR

DTBP=di-*tert*-butyl peroxide, TBHP=*tert*-butyl hydroperoxide, 70 wt% in H_2O, m-CPBA=3-Chloroperbenzoic acid.

[a] Reaction condition: benzoic acid (0.5mmol), 1,4-dioxane (1mL), catalyst (0.5 mol %), oxidant (2 equiv), 120 ºC, 4 h.

[b] Isolated yield based on carboxylic acid.

[c] DTBP (3 equiv).

[d] 30 wt% in H_2O.

[e] 100 ºC

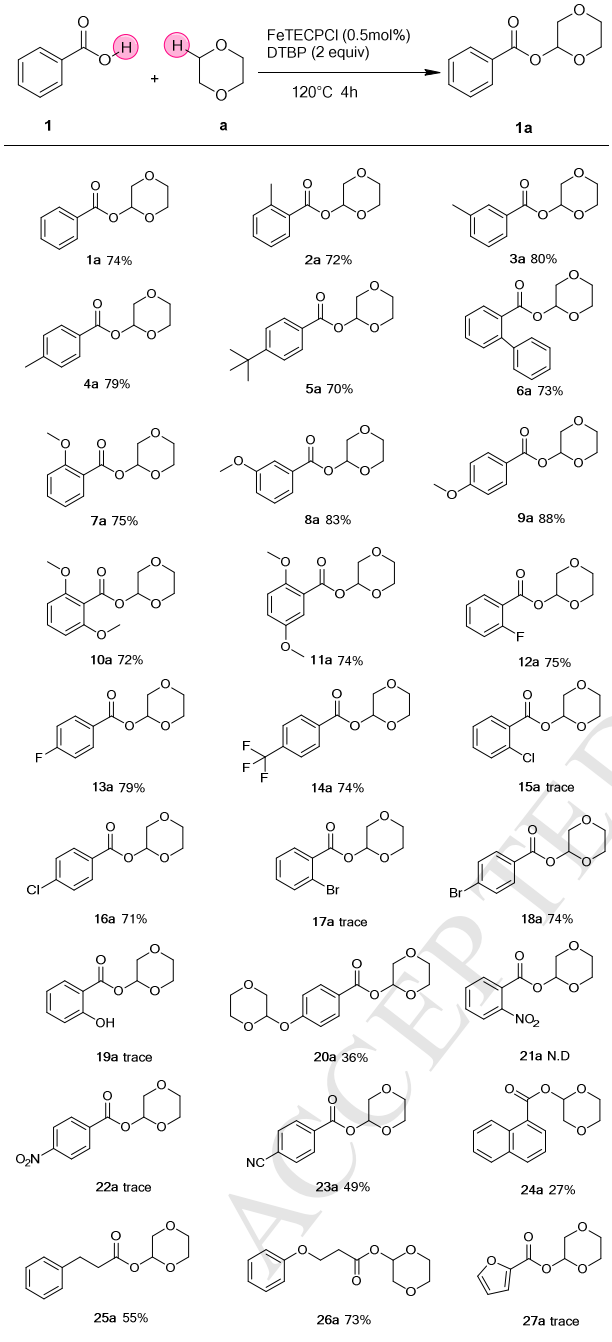
[f] 140 ºC

With the above optimized conditions in hand, we explored the 1,4-dioxane with other benzoic acid derivatives and the results were listed in Table 3. The CDC reaction between methyl and methoxy substituent tolerated in moderate yields of 72-84% (**2a-4a, 7a-9a**). Gratifyingly, 4-*tert*-butylbenzoic acid also examined and the desired product was obtained in 70% isolated yield (**5a**). Not only alkyl substituents were tested, but also 2-phenylbenzoic acid was investigated, affording **6a** with an isolated yield of 74% . There is no doubt that *ortho*, *meta*, *para*-benzoic acid has a good yield. The reaction yield of *ortho*-substituents is minimally reduced relative to *meta*- and *para*-positions. *ortho*-substituted acids were lower yields as compared to their *meta* and *para*-substituted analogues was due to steric hindrance imparted by *ortho*-substituents (**2a,7a,12a,15a**). At the same time, we used *ortho* and *para*-halogen substituents and hydroxyl substituents to illustrate the substitution effect of the substrate on the reaction (**12a-18a**). Strikingly, Electron deficient aromatic acids such as 4-nitrobenzoic acid, 4-halogenbenzoic acid, and 4-cyanobenzoic acid have lower yields in comparison to electron donating groups (**19a-23a**). Unfortunately, 2-naphthoic acid is not suitable for this

reaction (**24a**). Hydrocinnamic acid was used in this system to react with 1,4-dioxane under the typical reaction conditions, we were pleased to find that the reaction proceeded well and gave the corresponding product **25a** in 55% yield. 3-phenoxypropanoic acid were reacted with 1,4-dioxane and gave the corresponding products **26a** (73%). Even more, the reaction did not work when furan-3-carboxylic acid was used as substrate in this system (**27a**).

Table 3

Iron-catalyzed CDC reaction of aromatic acid with 1,4-dioxane[a]

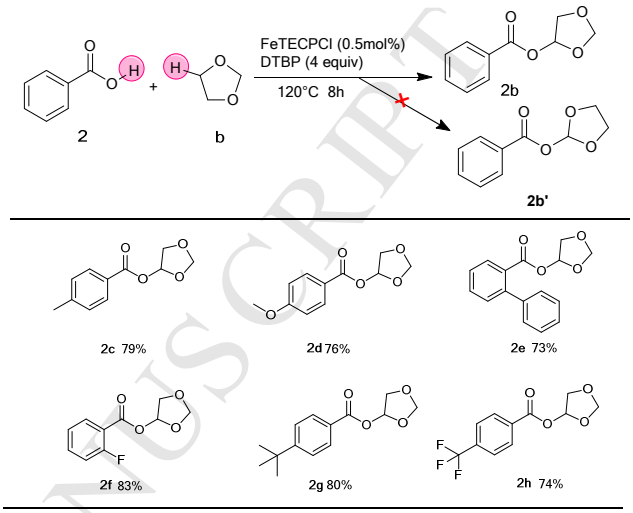


[a] Catalytic condition: benzoic acid (0.5mmol), 1,4-dioxane (1mL), catalyst (0.5 mol %), oxidant (2 equiv), 120 ºC, 4h.

After finishing the investigation of the 1,4-dioxane as a substrate, we attempted to extend the substrate range by using 1,3-dioxolane to explore the selectivity and activity of the reaction (Table 4). However, no reasonable yield of the product could be obtained with the same reaction conditions. When double the oxidant amount and prolong the reaction time to 8 hours, the yield of 2b may reach 40%, the further increasing of the reaction time did not affect the yield significantly. Interestingly, no 2b' product could be isolated. The reaction of other benzoic acid derivatives bearing electron-donating or withdrawing groups such as 4-methylbenzoic acid (**2c**), 4-methoxybenzoic acid (**2d**), 2-phenylbenzoic acid (**2e**), 2-fluorobenzoic acid (**2f**), 4-*tert*-butylbenzoic acid (**2g**) and α,α,α-trifluoro-*p*-toluic acid (**2h**) with 1,4-dioxane provided moderate yield of 73-83%.

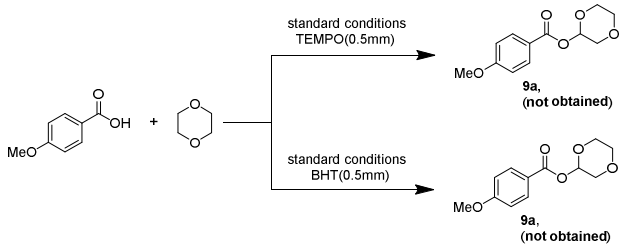
Table 4

Iron-catalyzed CDC reaction of aromatic acid with 1,3-dioxolane[a]

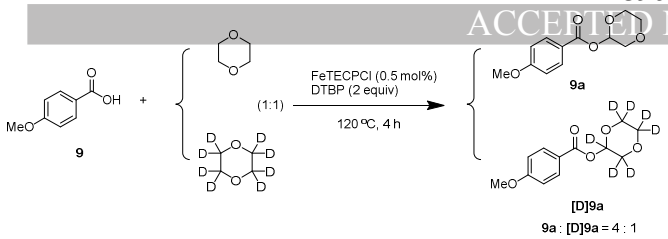


[a] Catalytic condition: benzoic acid (0.5mmol), 1,4-dioxane (1mL), catalyst (0.5 mol %), oxidant (4 equiv), 120 ºC, 8h.

To investigate the reaction mechanism, a series of experiments was conducted. When the reaction was carried out in the presence of radical scavenger such as 2,2,6,6-tetramethylpyridine N-oxide (TEMPO) or butylated hydroxytoluene (BHT), no desired product could be obtained (Scheme 3), indicating the reaction via a radical mediated pathway. The intermolecular competing kinetic isotope effect (KIE) experiment showed a KIE effect of $k_H/k_D = 4$ (the KIE was determined by 1H NMR spectroscopy by analyzing the ratio of 9a and [D]9a (Scheme 4). This suggested that C(sp3) - H bond cleavage of 1,4-dioxane was the kinetic rate-determining step[24]. Interestingly, the UV-Vis spectra of the catalyst changed remarkably after adding oxidant (Scheme 5), such a spectra changes revealed the transformation of catalyst FeTECP to its Fe- μ -oxo species[25]. That is, the real catalyst is a porphyrin Fe- μ -oxo complex. (Scheme 5).

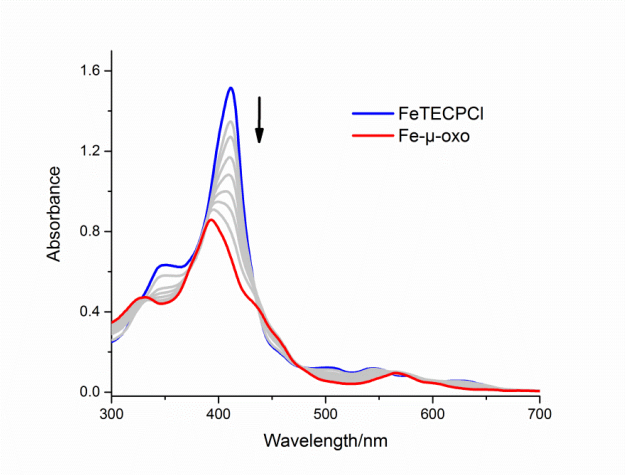


Scheme 3. Reaction carried out in the presence of **TEMPO** and **BHT**

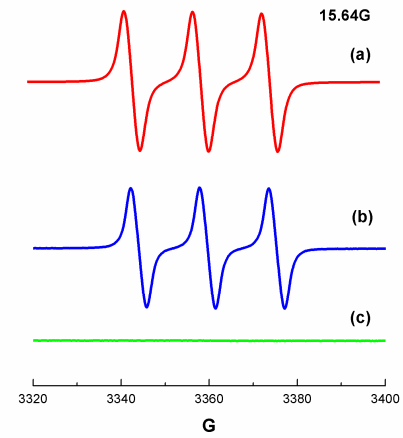


Scheme 4. Kinetic Isotope Effect

The TEMPO was detected by electron paramagnetic resonance (EPR) in the reaction mixture of benzoic acid, 1,4-dioxane and FeTECPCl as shown in Scheme 6, line a, which is TEMPO as a concentration standard[26]. At 25 °C and then temperature increased up to 120 °C the free radicals were slightly weakened as shown in Scheme 6, line b, and after 25 min no change was observed. Followed by the addition of DTBP in the mixture at 120 °C it very quickly reacts with TEMPO, no EPR signal was observe. The radicals disappear shown as Scheme 3, line c, indicating that the reactants produced free radicals at high temperatures, confirming the free radical reaction.

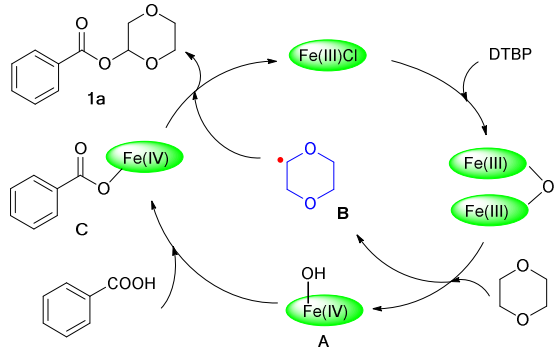


Scheme 5. UV spectrum of 1,4-dioxane (3mL), FeTECPCl (0.5 mol %), DTBP (570ul) at room temperature, measure every ten minutes.

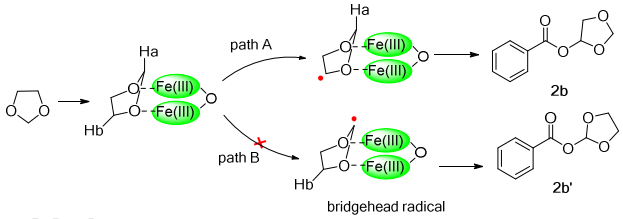


Scheme 6. EPR spectrum of (a) Benzoic acid (0.5mmol), 1,4-dioxane (1mL), FeTECPCl (0.5 mol %) and TEMPO (50mM) in room temperature; (b) Benzoic acid (0.5mmol), 1,4-dioxane (1mL), FeTECPCl (0.5 mol %) and TEMPO (50mM) in 120 °C after 25 min; (c) Followed by the addition of DTBP (2 equiv) in the mixture at 120 °C after 5 min.

Based on the above results and the previous reports[12], we proposed that the mechanism is likely to proceeds by a radical way (Scheme 7). Firstly, FeTECPCl is oxidized by by di-*tert*-*butyl* peroxide to form the active catalyst $(FeTECP)_2O$ (μ-oxo-bis[5,10,15,20-tetraarylporphyrinatoiron(III)]), as evidenced by UV-Vis experiment (Scheme 5). Secondly, $(FeTECP)_2O$ captures the hydrogen of 1,4-dioxane to form a tetravalent iron-hydroxyl porphyrin **A** and the active free radical **B** via a single electron transfer (SET) mechanism. The axial ligand exchange of A with benzoic acid to generate intermediate **C**. Finally, the intermediate **C** and the intermediate **B** undergoes a cross-coupling process**,** giving the final product **1a** and regenerate the catalyst FeTECPCl.



Scheme 7. Proposed Mechanism for Esterification



Scheme 8. Regioselectivity of the Reaction on 1,3-Dioxolane

According to the previous report on the stereoelectronic effects in hydrogen atom abstraction from cyclic ethers, we proposed the mechanism is illustrated in Scheme 8. The reaction of benzoic acid with 1,4-dioxane go through the pathway A rather than the pathway B, this is probably mainly due to the fact that bridgehead radicals are notoriously more difficult to form. Therefore, we constructed the abnormal regiosectivity of the reaction to give the final products **2b**.

3. Conclusion

In summary, we have demonstrated iron-porphyrin catalyzed $C(SP^3)$ - H activation for the formation of C-O bond via cross-dehydrogenative coupling of cycloether and aromatic acid. This examples the first application of Fe porphyrin in cross-dehydrogenative coupling reactions. When using phenol and cycloether as substrate, the CDC reaction could go smoothly also. Systematic investigation of iron porphyrin catalyzed CDC reactions is still going on in our laboratory.

4. Experimental section

4.1. General experimental

All the reagents were commercial grade and purified according to the established procedures. Organic extracts were dried over anhydrous sodium sulphate. Solvents were removed in a rotary evaporator under reduced pressure. Silica gel (100–200 mesh size) was used for the column chromatography. UV-Visible absorption spectra were measured on Hitachi 3900H in 1 cm optical path length quartz cell at room temperature. NMR spectra were recorded in $CDCl_3$ with tetramethylsilane as the internal standard for ^1H NMR (400 MHz) $CDCl_3$ solvent as the internal standard for ^{13}C NMR (100 MHz). HR–MS spectra were recorded on Bruker maxis impact mass spectrometer with an ESI source.

4.2. Preparation of Catalyst

The synthesis of 5,10,15,20-tetrakis(ethoxycarbonyl)-porphyrin ligand (TECP) and 5,10,15,20-tetrakis-(ethoxycarbonyl)porphyrin manganese (III) (MnTECPCl) were performed by our recently reported procedure[27]. The metalloporphyrin 5,10,15,20-tetrakis(ethoxycarbonyl)porphyrin Cobalt(II) (CoTECP), 5,10,15,20-tetrakis(ethoxycarbonyl)-porphyrin iron (III) (FeTECPCl) and μ-oxo-bis[5,10,15,20-tetraarylporphyrinatoiron (III) ($(FeTECP)_2O$) were prepared using literature procedures[28].

FeTECPCl was obtained by refluxing TECP with $FeCl_3$•$4H_2O$ in DMF solution for 2 h. After that it was diluted with CH_2Cl_2 and washed with saturated aqueous solution of NaCl several times. The organic layer was collected and dried over anhydrous Na_2SO_4. The filtrate was concentrated and the crude product was purified on silica gel (100–200 mesh) using CH_2Cl_2 as eluent. Purple crystal was obtained after slow evaporation from CH_2Cl_2/hexane (1:4 v/v). Yield: 90%. HR–MS calcd for $[M\text{-}Cl]^+$: $C_{32}H_{28}FeN_4O_8$ 652.1252, found 652.1248.

MnTECPCl was synthesized similar to the procedure of FeTECPCl, except $FeCl_3$•$4H_2O$ was used instead of $Mn(OAc)_2$•$4H_2O$. Yield: 80%[17].

CoTECP was synthesized similar to the procedure of FeTECPCl, except $FeCl_3$•$4H_2O$ was used instead of $Co(OAc)_2$•$4H_2O$. Yield: 82%. HR–MS calcd for $[M]^+$: $C_{32}H_{28}CoN_4O_8$ 655.1234, found 655.1236.

Fe-μ-oxo was synthesized by FeTECPCl with NaOH solution for 1 h, Yield: 95%. MALDI-TOF calcd for $[M+Na]^-$: $C_{64}H_{56}Fe_2N_8O_{17}Na$ 1343.2340, found 1343.0590.

4.3. General procedure

In a Schlenk tube equipped with a magnetic stir bar was added FeTECPCl (0.33 mg, 0.5 μmol) and benzoic acid (61 mg, 0.5 mmol). 1,4-Dioxane (1.0 mL, 7.5 – 12.5 mmol) and DTBP (di-tert-butyl peroxide, 1 mmol, 190 μL) were added. The resulting reaction mixture was stirred at 120 °C for 4 h. After the required reaction time, the mixture was cooled to room temperature. The reaction mixture was extracted with dichloromethane. The organic phases were evaporated under reduced pressure. The crude product was purified by column chromatography on silica gel (hexane/ethyl acetate 95/5) to accord the corresponding product.

For ESR experiments, 1 mL glass micropipettes equipped with a stopper at the bottom were used as ESR tubes (inner diameter 1.2mm). Spectra were recorded on an X-band ESR instrument (Bruker-A300) using the following adjustments: HF (100 kHz) field modulation amplitude 100 kHz; microwave power 20 mW; scan range 100 G; sweep time 122 s; conversion time 120 ms, time constant 655 ms, temperature 120 -121° C and 23-24 ° C.

4.4. Mechanistic investigation: Trapping of radical intermediates with radical scavenger.

TEMPO (or BHT): An oven-dried reaction vessel was charged with p-Anisic acid (2) (76 mg, 0.5 mmol), FeTECPCl (1.7 mg, 0.5 mol%), DTBP (190 μL, 1 mmol), radical scavenger (0.5 mmol) in 1,4-dioxane (a) (1 mL). The flask was fitted to a condenser and the resultant reaction mixture was stirred in a preheated oil bath at 120 ℃ for 4 h. The reaction after 4 h afforded traces (<5%) of the desired product (9a).

4.3.1. 1,4-Dioxan-2-yl benzoate **(1a)**. Yellow oil, yield 74%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 7.7 Hz, 2H), 7.46 (d, J = 7.4 Hz, 1H), 7.35 (t, J = 7.6 Hz, 2H), 5.99 (s, 1H), 4.16 – 4.06 (m, 1H), 3.78 (s, 2H), 3.71 (d, J = 5.1 Hz, 2H), 3.56 (d, J = 11.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.19, 133.40, 129.89, 129.74, 128.46, 89. 83, 67.81, 66.10, 61.75.

4.3.2. 1,4-dioxan-2-yl 2-methylbenzoate **(2a)**. Yellow oil, yield 72%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 8.03 (m, 1H), 7.47 – 7.39 (m, 1H), 7.28 (t, J = 7.0 Hz, 2H), 6.10 (t, J = 1.8 Hz, 1H), 4.31 – 4.16 (m, 1H), 3.90 (d, J = 1.8 Hz, 2H), 3.83 (dd, J = 6.7, 2.6 Hz, 2H), 3.70 (dt, J = 11.8, 2.6 Hz, 1H), 2.67 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.97, 140.86, 132.49, 131.84, 131.04, 128.87, 125.80, 89.69, 67.93, 66.13, 61.89, 21.93.

4.3.3. 1,4-dioxan-2-yl 3-methylbenzoate **(3a)**. Yellow oil, yield 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 5.8 Hz, 2H), 7.34 (dt, J = 15.6, 7.6 Hz, 2H), 6.07 (t,1H), 4.29 – 4.12 (m, 1H), 3.86 (t, J = 6.1 Hz, 2H), 3.80 (dd, J = 6.6, 2.1 Hz, 2H), 3.65 (dt, J = 11.8, 2.6 Hz, 1H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.37, 138.27, 134.18, 130.35, 129.69, 128.35, 127.09, 89.78, 67.87, 66.12, 61.82, 21.23.

4.3.4. 1,4-dioxan-2-yl 4-methylbenzoate **(4a)**. Yellow oil, yield 79%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (t, J = 8.3 Hz, 2H), 7.24 (d, J = 8.0 Hz, 2H), 6.08 (s, 1H), 4.30 – 4.10 (m, 1H), 3.88 (d, J = 1.8 Hz, 2H), 3.81 (d, J = 5.6 Hz, 2H), 3.66 (dt, J = 11.6, 2.2 Hz, 1H), 2.40 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.23, 144.15, 129.94, 129.15, 127.00, 89.67, 67.86, 66.10, 61.79, 21.64.

4.3.5. 1,4-dioxan-2-yl 4-(tert-butyl)benzoate **(5a)**. Yellow oil, yield 70%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.5 Hz, 2H), 7.46 (d, J = 8.5 Hz, 2H), 6.08 (s, 1H), 4.30 – 4.11 (m, 1H), 3.91 – 3.76 (m, 4H), 3.65 (dt, J = 11.7, 2.5 Hz, 1H), 1.33 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.19, 157.13, 129.83, 126.97, 125.43, 89.63, 67.89, 66.13, 61.78, 35.12, 31.10.

4.3.6. 1,4-dioxan-2-yl [1,1'-biphenyl]-2-carboxylate **(6a)**. Yellow oil, yield 73%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, J = 6.9 Hz, 1H), 7.58 (t, J = 7.5 Hz, 1H), 7.48 – 7.33 (m, 7H), 5.85 (s, 1H), 3.70 – 3.57 (m, 4H), 3.42 (dd, J = 28.5, 10.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.51, 142.74, 141.42, 131.55, 130.87, 130.44, 130.14, 128.53, 128.17, 127.23, 90.12, 67.39, 65.84, 61.57.

4.3.7. 1,4-Dioxan-2-yl 2-methoxybenzoate **(7a)**. Yellow oil, yield 75%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 7.5 Hz, 1H), 7.48 (t, J = 7.7 Hz, 1H), 7.06 – 6.87 (m, 2H), 6.08 (s, 1H), 4.34 – 4.12 (m, 1H), 3.90 (s, 3H), 3.86 (s, 2H), 3.79 (d, J = 4.9 Hz, 2H), 3.66 (d, J = 11.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.56, 159.60, 134.02, 131.87, 120.09, 119.40, 112.13, 89.60, 67.80, 66.10, 61.73, 55.98.

4.3.8. 1,4-Dioxan-2-yl 3-methoxybenzoate **(8a)**. Yellow oil, yield 83%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 8.8 Hz, 2H), 6.87 (d, J = 8.7 Hz, 2H), 6.01 (s, 1H), 4.26 – 4.06 (m, 1H), 3.82 (d, J = 0.5 Hz, 2H), 3.79 (s, 3H), 3.75 (dd, J = 6.4, 2.2 Hz, 2H), 3.61 (d, J = 11.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.09, 159.61, 131.02, 129.48, 122.27, 119.75, 114.43, 89.92, 67.77, 66.08, 61.79, 55.43.

4.3.9. 1,4-Dioxan-2-yl 4-methoxybenzoate **(9a)**. Yellow oil, yield 88%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 8.8 Hz, 2H), 6.87 (d, J = 8.7 Hz, 2H), 6.01 (s, 1H), 4.26 – 4.06 (m, 1H), 3.82 (d, J = 0.5 Hz, 2H), 3.79 (s, 3H), 3.75 (dd, J = 6.4, 2.2 Hz, 2H), 3.61 (d, J = 11.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.86, 163.73, 131.96, 122.00, 113.69, 89.49, 67.87, 66.08, 61.77, 55.40.

4.3.10. 1,4-Dioxan-2-yl 2,6-dimethoxybenzoate **(10a)**. Yellow oil, yield 72%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, J = 8.4 Hz, 1H), 6.56 (d, J = 8.4 Hz, 2H), 6.10 (s, 1H), 4.32 – 4.20 (m, 1H), 3.86 – 3.77 (m, 10H), 3.65 (d, J = 11.8 Hz, 1H). ^{13}C NMR (101 MHz,

$CDCl_3$) δ 165.34, 157.55, 131.29, 112.63, 103.96, 89.84, 67.74, 66.11, 61.36, 56.00.

4.3.11. 1,4-dioxan-2-yl 2,4-dimethoxybenzoate **(11a)**. Yellow oil, yield 74%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.4 Hz, 1H), 6.44 (d, J = 10.2 Hz, 2H), 6.00 (s, 1H), 4.22 – 4.08 (m, 1H), 3.83 (s, 3H), 3.79 (s, 5H), 3.74 (d, J = 6.1 Hz, 2H), 3.60 (d, J = 11.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.71, 163.79, 161.94, 134.11, 111.52, 104.69, 98.92, 89.25, 67.91, 66.11, 61.81, 55.96, 55.47.

4.3.12. 1,4-dioxan-2-yl 2-fluorobenzoate **(12a)**. Yellow oil, yield 75%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (t, J = 7.5 Hz, 1H), 7.48 (dd, J = 13.5, 6.9 Hz, 1H), 7.17 (dd, J = 14.8, 7.2 Hz, 1H), 7.12 – 7.05 (m, 1H), 6.05 (s, 1H), 4.28 – 4.05 (m, 1H), 3.82 (s, 2H), 3.61 (d, J = 11.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.51, 160.92, 134.92, 132.30), 124.01, 118.27, 117.22, 90.22, 67.72, 66.14, 61.80. ^{19}F NMR (376 MHz, $CDCl_3$) δ -108.66 (s).

4.3.13. 1,4-dioxan-2-yl 4-fluorobenzoate **(13a)**. Yellow oil, yield 79%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (dd, J = 8.9, 5.4 Hz, 2H), 7.11 (t, J = 8.7 Hz, 2H), 6.07 (t, J = 1.7 Hz, 1H), 4.26 – 4.13 (m, 1H), 3.87 (d, J = 1.9 Hz, 2H), 3.81 (dd, J = 6.8, 2.6 Hz, 2H), 3.66 (dt, J = 11.8, 2.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.30, 164.77, 164.23, 132.55, 125.99, 115.75, 115.53, 89.97, 67.81, 66.11, 61.80. ^{19}F NMR (376 MHz, $CDCl_3$) δ -104.83 (s).

4.3.14. 1,4-dioxan-2-yl 4-(trifluoromethyl) benzoate **(14a)**. Yellow oil, yield 74%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, J = 8.1 Hz, 2H), 7.63 (d, J = 8.3 Hz, 2H), 6.03 (s, 1H), 4.26 – 4.07 (m, 1H), 3.82 (s, 2H), 3.75 (d, J = 4.8 Hz, 2H), 3.60 (d, J = 11.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.04, 134.96, 134.64, 133.04, 130.27, 125.47, 90.40, 67.68, 66.07, 61.76. ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.23 (s).

4.3.15. 1,4-Dioxan-2-yl 4-chlorobenzoate **(16a)**. White solid, yield 71%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.6 Hz, 2H), 7.43 (d, J = 8.6 Hz, 2H), 6.08 (s, 1H), 4.28 – 4.10 (m, 1H), 3.88 (d, J = 1.8 Hz, 2H), 3.82 (dd, J = 6.7, 2.5 Hz, 2H), 3.68 (dt, J = 11.8, 2.5 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.41, 139.93, 131.30, 128.85, 128.20, 90.07, 67.79, 66.13, 61.81.

4.3.16. 1,4-Dioxan-2-yl 4-bromobenzoate **(18a)**. Yellow solid, yield 74%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 8.4 Hz, 2H), 7.55 (d, J = 8.4 Hz, 2H), 6.04 (s, 1H), 4.22 – 4.04 (m, 1H), 3.84 (s, 2H), 3.78 (d, J = 5.3 Hz, 2H), 3.63 (d, J = 11.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.46, 131.80, 131.38, 128.65, 128.56, 90.06, 67.73, 66.08, 61.75.

4.3.17. 1,4-dioxan-2-yl 4-((1,4-dioxan-2-yl)oxy)benzoate **(20a)**. Yellow soild, yield 36%; HR–MS calcd for [M+Na]-: $C_{15}H_{18}NaO_7$ 333.2918, found 333.0945. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 8.8 Hz, 2H), 7.13 (d, J = 8.8 Hz, 2H), 6.07 (s, 1H), 5.36 (d, J = 13.8 Hz, 1H), 4.27 – 4.05 (m, 2H), 4.00 – 3.73 (m, 8H), 3.64 (dd, J = 26.7, 11.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.13, 161.14, 132.00, 123.23, 121.52, 116.01, 115.38, 93.19, 89.43, 67.90, 66.11, 61.71, 61.07.

4.3.18. 1,4-Dioxan-2-yl 4-cyanobenzoate **(23a)**. Yellow solid, yield 49%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, J = 8.3 Hz, 2H), 7.76 (d, J = 8.3 Hz, 2H), 6.10 (s, 1H), 4.36 – 4.07 (m, 1H), 3.90 (d, J = 1.7 Hz, 2H), 3.84 (dd, J = 6.9, 2.3 Hz, 2H), 3.69 (d, J = 11.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.70, 133.61, 132.32, 130.39, 117.88, 116.85, 90.65, 67.66, 66.10, 61.79.

4.3.19. 1,4-Dioxan-2-naphthalenecarboxylic **(24a)**. Yellow solid, yield 27%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.02 (d, J = 8.5 Hz, 1H), 8.36 (d, J = 7.0 Hz, 1H), 8.06 (d, J = 7.9 Hz, 1H), 7.90 (d, J = 7.9 Hz, 1H), 7.64 (t, J = 7.4 Hz, 1H), 7.54 (dd, J = 16.4, 8.1 Hz, 2H), 6.22 (s, 1H), 4.44 – 4.19 (m, 1H), 3.96 (s, 2H), 3.86 (d, J = 5.3 Hz, 2H), 3.73 (d, J = 11.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.95, 133.98, 131.59, 130.97, 128.64, 128.06, 126.26, 125.79, 124.49, 89.90, 67.99, 66.21, 61.94.

4.3.20. 1,4-dioxan-2-yl 3-phenylpropanoate (**25a**). Yellow solid, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.13 (m, 5H), 5.83 (s, 1H), 4.12 – 3.88 (m, 1H), 3.83 – 3.62 (m, 4H), 3.56 (m, 1H), 2.98 (t, J = 7.7 Hz, 2H), 2.71 (t, J = 7.7 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.65, 140.24, 128.54, 128.33, 126.35, 89.31, 67.70, 66.02, 61.65, 35.86, 30.77.

4.3.21. 1,4-dioxan-2-yl 3-phenoxypropanoate (**26a**). Yellow oil, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.24 (m, 2H), 7.00 – 6.87 (m, 3H), 5.91 (t, J = 1.8 Hz, 1H), 4.34 – 4.23 (m, 2H), 4.17 – 4.06 (m, 1H), 3.80 – 3.71 (m, 4H), 3.61 (dt, J = 11.7, 2.6 Hz, 1H), 2.89 (t, J = 6.3 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.92, 158.47, 129.48, 121.13, 114.71, 89.57, 67.67, 66.05, 63.18, 61.65, 34.74. HRMS (ESI$^+$): m/z calcd for [$C_{13}H_{16}O_5$ $^+$ Na] 275.0890, found 275.0886.

4.3.22. 1,4-Dioxolan-4-yl 4-methylbenzoate **(2c)**. Yellow oil, yield 79%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 8.2 Hz, 2H), 7.13 (d, J = 8.0 Hz, 2H), 6.47 (dd, J = 4.0, 1.8 Hz, 1H), 5.06 (d, J = 17.5 Hz, 2H), 4.03 (ddd, J = 11.3, 9.5, 3.0 Hz, 2H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.83, 144.30, 129.83, 129.17, 126.72, 95.85, 94.56, 70.68, 21.66.

4.3.23. 1,4-Dioxolan-4-yl 4-methoxybenzoate **(2d)**. Yellow oil, yield 76%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 8.9 Hz, 2H), 6.88 (d, J = 8.9 Hz, 2H), 6.53 (dd, J = 4.1, 1.9 Hz, 1H), 5.13 (d, J = 17.1 Hz, 2H), 4.10 (ddd, J = 11.4, 9.5, 3.0 Hz, 2H), 3.82 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.48, 163.82, 131.89, 121.76, 113.71, 95.81, 94.43, 70.67, 55.43.

4.3.24. 1,4-Dioxolan-4-yl 2-phenylbenzoate **(2e)**. Yellow oil, yield 73%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 7.9 Hz, 1H), 7.56 (t, J = 7.0 Hz, 1H), 7.49 – 7.27 (m, 8H), 6.32 (dd, J = 4.3, 1.9 Hz, 1H), 4.95 (s, 1H), 4.61 (s, 1H), 3.87 (dd, J = 9.4, 4.4 Hz, 1H), 3.37 (dd, J = 9.4, 1.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.95, 142.83, 141.47, 131.76, 130.82, 130.27, 130.05, 128.45, 128.10, 127.30, 95.49, 94.34, 69.98.

4.3.25. 1,4-Dioxolan-4-yl 2-fluorobenzoate **(2f)**. Yellow oil, yield 83%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (t, J = 7.5 Hz, 1H), 7.51 (dd, J = 12.8, 6.0 Hz, 1H), 7.14 (dt, J = 19.2, 7.8 Hz, 2H), 6.56 (dd, J = 4.1, 1.8 Hz, 1H), 5.14 (d, J = 9.6 Hz, 2H), 4.11 (dd, J = 24.3, 12.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.42, 160.83, 135.12, 132.18, 124.01, 118.00, 117.17, 95.90, 94.83, 70.64. ^{19}F NMR (376 MHz, $CDCl_3$) δ -108.79 (s).

4.3.26. 1,4-Dioxolan-4-yl 4-tert-butylbenzoate **(2g)**. Yellow oil, yield 80%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 8.5 Hz, 2H), 7.35 (d, J = 8.5 Hz, 2H), 6.48 (dd, J = 4.1, 1.9 Hz, 1H), 5.06 (d, J = 14.4 Hz, 2H), 4.03 (ddd, J = 11.3, 9.5, 3.0 Hz, 2H), 1.23 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.77, 157.24, 129.71, 126.70, 125.43, 95.83, 94.55, 70.70, 35.12, 31.07, 28.20.

4.3.27. 1,4-Dioxolan-4-yl 4-(trifluoromethyl)benzoate **(2i)**. Yellow oil, yield 74%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 8.2 Hz, 2H), 7.13 (d, J = 8.0 Hz, 2H), 6.47 (dd, J = 4.0, 1.8 Hz, 1H), 5.06 (d, J = 17.5 Hz, 2H), 4.03 (ddd, J = 11.3, 9.5, 3.0 Hz, 2H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.63, 134.77, 132.73, 130.19, 125.51, 122.17, 96.12, 95.13, 70.71. ^{19}F NMR (376 MHz, $CDCl_3$) δ -63.25 (s).

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Supplementary Material

The Supporting Information is available free of charge at http://dx.doi.org/ 10.1016/j.tetlet.xxxxxxx.

Experimental details; single crystal results of FeTECPCl; spectral and analytical data. (PDF)
Crystallographic data for FeTECPCl (CIF)

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