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Stereoselective Nitration of Olefins via Visible-Light-Induced Iron-Complex β-Homolysis

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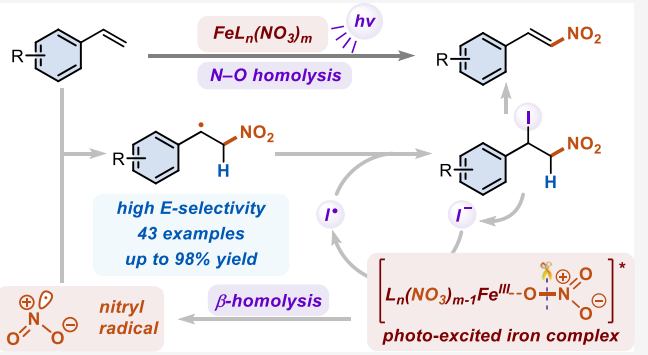
ABSTRACT: Herein, we develop a green and efficient method for the stereoselective synthesis of nitroolefins with ferric nitrate as the nitration reagent. With a visible-light-induced iron-complex β-homolysis pathway and I^- as a crucial media for electron transfer and dehydrogenation, a broad scope of nitroolefins including some valuable biorelevant derivatives are achieved in high *E*-selectivity. The reaction is conveniently controlled by nitrate equivalences, and scale-up experiments in batch and flow indicate excellent practicability for this protocol.



INTRODUCTION

The nitro group is one of the most popular functional groups in organic synthesis since it can be efficiently transformed into other nitrogen-containing compounds.[1] Nitro moiety is also prevalent in pharmaceuticals,[2] agriculture,[2b,c] dyes,[3] materials,[4] and explosives.[1] Therefore, the construction of nitro-containing compounds has always attracted much attention.[5] Nitroolefins, one of the most important nitro-containing compounds, play a significant role in various amounts of organic reactions[1,6] such as the Micheal addition[6a] and Baylis−Hillman reaction.[6b] They are also a prominent class of synthetic intermediates, which have been utilized to produce a wide variety of biorelevant compounds and pharmaceuticals.[7] Generally, nitroolefins are produced by the Henry reaction[8] referring to base-mediated dehydration condensation of nitroalkanes with aldehydes or ketones. However, it always suffers from high temperature and poor functional group tolerance. To address these issues, the *ipso*-nitration of cinnamic acids via a decarboxylation process was carried out in the past few decades (Scheme 1a).[9] Nonetheless, the substrate scope remains limited due to the structural constraint. Correspondingly, direct olefin nitration appears to be an ideal solution, and many pioneering works were developed for achieving this target in recent years[5a,10] (Scheme 1b) with the nitryl radical generated from superstoichiometric $AgNO_3$/*tert*-butyl nitrite/ferric nitrate under heating[10abc−d] or $NaNO_2$ with strong oxidant and acid.[10e] Moreover, novel organic nitrating reagents and photocatalysts were also introduced,[10fg−h] leading to room temperature conditions as well as additional reagent-preparing steps for direct alkene nitration. Therefore, mild, convenient, and environmentally friendly protocols for accessing nitrated olefins remain to be developed.

The past decade has witnessed a rapid development of photocatalysis as an environmentally friendly, sustainable, and innovative approach for organic transformations.[11] Recently, an intriguing method for photoactivation called ligand-to-metal charge transfer (LMCT) has attracted great attention, relying on an inner-sphere excited state that boosts the homolysis of the metal−ligand bond and results in the generation of reduced metal complexes and ligand-center radicals.[12] Many notable groups[13] including us[14] revealed a variety of simple earth-abundant transition metal complexes to be capable of undergoing this unique photoinduced α-homolysis process. Lately, our group reported an enzyme-mimicking protocol on visible-light-driven arene C−H nitration with ferric nitrates as the nitrating reagent, in which a nitryl radical generated from unusual β-homolysis of a ferric-nitrate complex was proved to be the key nitration species.[15] Based on these observations, we propose that this pathway can be utilized in the nitration of olefins and thus offer operational convenience and ecological benefits. Accordingly, we herein established a stereoselective method for visible light-induced alkene nitration with ferric nitrates via the β-homolysis pathway and I^- as a crucial media for electron transfer and dehydrogenation (Scheme 1c).

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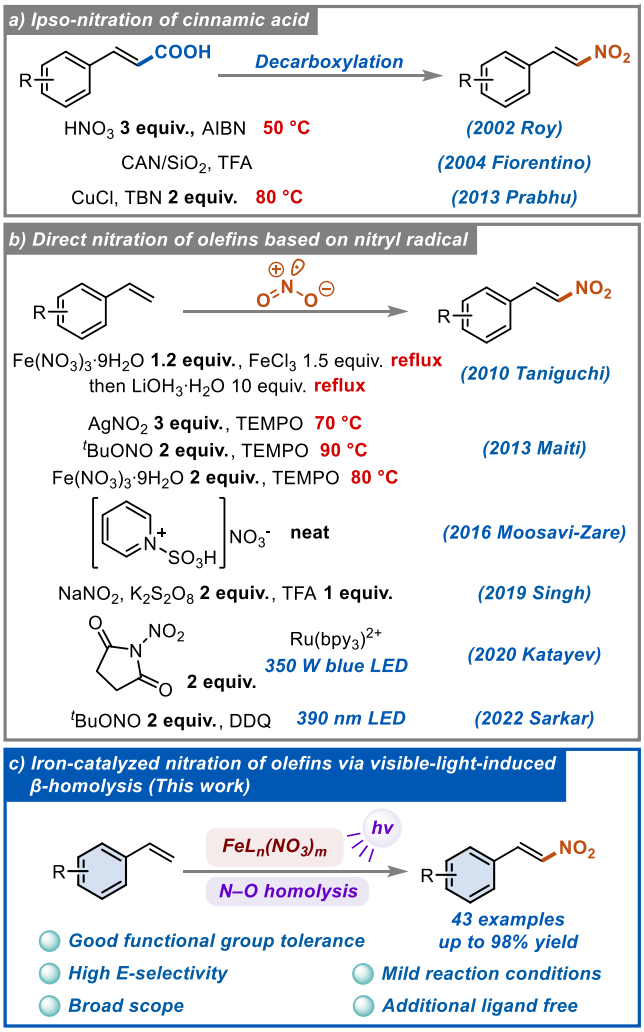
Scheme 1. Approaches for the Synthesis of Nitroolefins

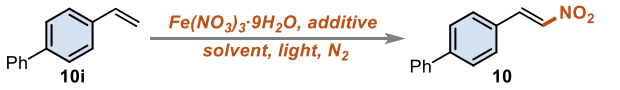


Various olefins including biorelevant compound-derived ones were efficiently and *E*-selectively nitrated under mild conditions.

RESULTS AND DISCUSSION

We started our exploration with the model reaction using 4-vinylbiphenyl (**10i**) in the presence of $Fe(NO_3)_3·9H_2O$ (0.5 equiv.) in MeCN under irradiation of 395 nm LEDs at room temperature for 8 h (Table 1, entry 1). Unexpectedly, only a trace amount of (*E*)-4-(2-nitrovinyl)-1,1′-biphenyl (**10**) was detected by GC−MS with no starting materials remaining. To lower the photon energy input and inhibit substrate decomposition, longer illumination wavelengths were tried (entries 2−4), and we were pleased to find that 455 nm appeared to be the best choice with **10** obtained in 22% yield with a majority of the starting materials reserving. Later, different additives were investigated to promote the reaction efficiency (entries 5−9). Among them, acid (entry 6) could facilitate the reaction a little while base (entry 5) and reductant (entry 7) showed no positive effect. Fortunately, a satisfying yield (96%) with high *E*-isomer selectivity was acquired when TBAI was employed (entry 9). The influence of solvents was explored as well, and other solvents including acetone, DCE, and DMSO produced lower yields of **10** than MeCN (entries 10−12). Variation in the atmosphere exhibited a negative

Table 1. Optimization of the Reaction Conditions[a]



Entry	Additive	Solvent	Light	Yield [%][b]
1	-	MeCN	395 nm	trace
2	-	MeCN	427 nm	10
3	-	MeCN	455 nm	22
4	-	MeCN	520 nm	trace
5	K_2CO_3	MeCN	455 nm	18
6	TFA	MeCN	455 nm	47
7	HE	MeCN	455 nm	N.D.
8	KI	MeCN	455 nm	20
9	TBAI	MeCN	455 nm	96
10	TBAI	Acetone	455 nm	49
11	TBAI	DCE	455 nm	32
12	TBAI	DMSO	455 nm	N.R.
13[c]	TBAI	MeCN	455 nm	45
14	TBAI	MeCN	-	N.R.
15	TBAI	MeCN	CFL	43
16[d]	TBAI	MeCN	455 nm	N.D.

[a]Reaction conditions: **10i** 0.2 mmol, $Fe(NO_3)_3·9H_2O$ 0.5 equiv., additive 0.5 equiv., solvent 2 mL, 30 W LED, N_2, rt, 8 h. [b]Isolated yield. [c]Under air. [d]$Cu(NO_3)_2·3H_2O$ (0.75 equiv.) instead of $Fe(NO_3)_3·9H_2O$. TFA = trifluoroacetic acid. HE = Hantzsch ester. TBAI = tetrabutylammonium iodide. N.D.: not detected. N.R.: no reaction.

impact (entry 13), and the control experiment indicated that the reaction could not occur without visible light as an energy source (entry 14). Switching the visible light source from 455 nm LEDs to CFL (Compact Fluorescent Lamp) resulted in a lower yield (entry 15). $Cu(NO_3)_2·3H_2O$, a metal nitrate proposed to own similar photoexcited properties to ferric nitrate, was employed as the nitro source, but no desired product was obtained (entry 16).

With the optimal reaction conditions in hand, various olefins were employed to examine the generality of our protocol. As shown in Scheme 2, a broad range of olefins proceeded efficiently, and gave corresponding nitroolefins in good to excellent yields with high *E*-isomer selectivity (**1**−**43**). Different electron-donating groups substrated olefins such as methyl (**2**, **18**, **19**, **21**, **22**), methoxy (**3**), phenoxy (**4**), isopropyl (**5**), and *tert*-butyl (**8**) were all suitable substrates and corresponding nitroolefins were obtained in pretty good yields. As vital synthons, halogen-substituted olefins performed well and gave corresponding nitration products in excellent yields (**7**−**9**, **23**, **24**), providing good chances for further cross-couplings. Trimethylsilyl as an active group also remained untouched, offering a good opportunity for further derivatization (**11**). As an important building block, phthaloyl-substituted olefin was likewise compatible (**12**). Additionally, we were pleased to find that mono- (**13**) or di-nitration (**14**) could be conveniently modulated for 1,3-divinylbenzene by the usage of different equivalences of ferric nitrates and TBAI, offering open space for the following modifications. Olefins with naphthalene and benzocyclobutene were tested next, and

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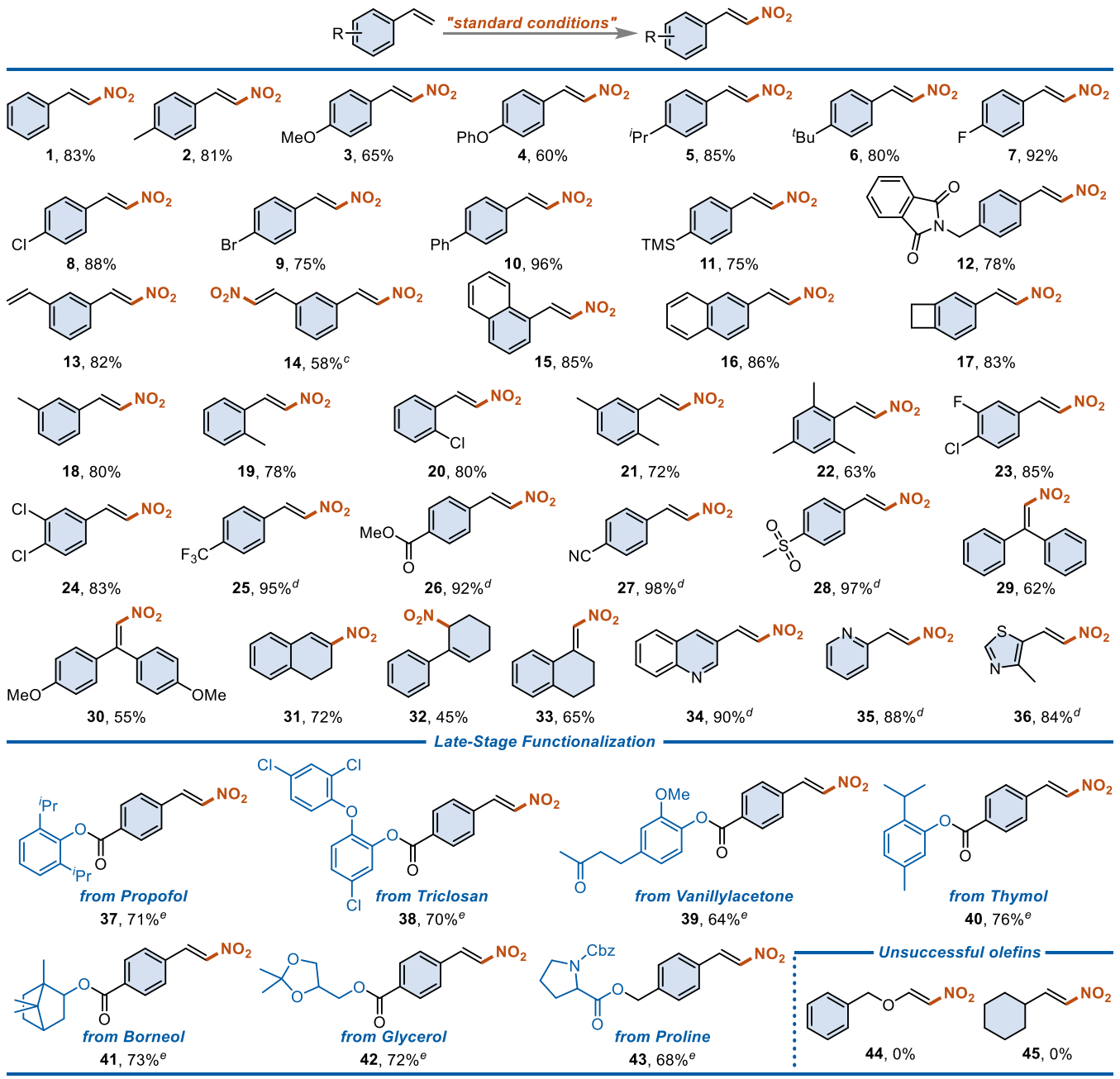
J. Org. Chem. 2025, 90, 7458−7467

Scheme 2. Substrate Scope for the Visible-Light-Induced Stereoselective Nitration of Olefins[a,b]



[a]Reaction conditions: olefin **i** 0.2 mmol, $Fe(NO_3)_3{\cdot}9H_2O$ 0.5 equiv., TBAI 0.5 equiv., MeCN 2 mL, 30 W 455 nm LED, N_2, room temperature, 8 h. [b]Isolated yield. [c]One equiv. of $Fe(NO_3)_3{\cdot}9H_2O$ and 1 equiv. of TBAI was added. [d]12 h. [e]16 h.

the corresponding products were achieved in satisfying yields (**15**–**17**). Olefins with ortho- and meta-substituents were also viable substrates, affording exclusively desired products in satisfactory efficiency (**18**–**20**). Di- and trisubstituted olefins also supplied the nitration product in good yields (**21**–**24**). Notably, we were gratified to find that a variety of electron-deficient substituents also participated well in the reaction after prolonging the reaction time. Substrates respectively with trifluoromethyl (**25**), ester (**26**), cyano (**27**), and sulfonyl (**28**) were all readily converted to the corresponding nitroolefins in excellent yields. In addition, substrates with a substituent flanking the α-position of olefins (**29**, **30**, **33**) and a typical inner-alkene dihydronaphthalene (**31**) were all suitable candidates, and desired β-nitroolefins were obtained in medium to good yields. An exception was found that allylic nitro product **32** was afforded rather than the β-nitrostyrene, which should be attributed to the stabilized intermediate and the easy β-hydrogen elimination from the other side. Furthermore, diverse heterocyclic nitro-alkenes involving quinoline (**34**), pyridine (**35**), and thiazole (**36**) were also acquired in 84%–90% yields. When an electron-rich olefin (**44**) was used as a substrate, no desired product was obtained due to its instability under the oxidizing conditions. Attempts to apply the method to an alkyl olefin (**45**) also proved unsuccessful, achieving a result attributed to its inherently low reactivity.

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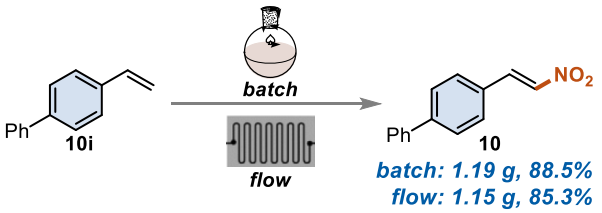
J. Org. Chem. 2025, 90, 7458−7467

Broadening the applicability to late-stage photonitration of olefins containing drugs and biorelevant motifs will significantly increase the application value of our strategy in functional molecule modification. According to this thought, substrates with drug molecules were tried first, and the corresponding products from *Propofol* (**37**) and *Triclosan* (**38**) exhibited good results, indicating an application potential in pharmaceutical and agrochemical discovery. Sequentially, the conversion of alkenes derived from diverse biogenic complex molecules was tested, and nitration products from *Vanillylacetone* (**39**), *Thymol* (**40**), *Borneol* (**41**), *Glycerol* (**42**), and *Proline* (**43**) were all achieved efficiently, further demonstrating the wonderful biocompatibility and potential in the post-modification synthesis.

Next, gram-scale experiments of **10i** were performed to illustrate the practicability of this method (Scheme 3). In a 6 mmol-scale batch reaction, 1.19 g of **10** was smoothly produced in 89% yield without obvious loss of efficiency. To further improve the light utilization efficiency, a well-designed continuous-flow photocatalytic microreactor was attempted and demonstrated to be capable of generating **10** in a similar result with a reduced time and attenuated light irradiation than the batch reaction.

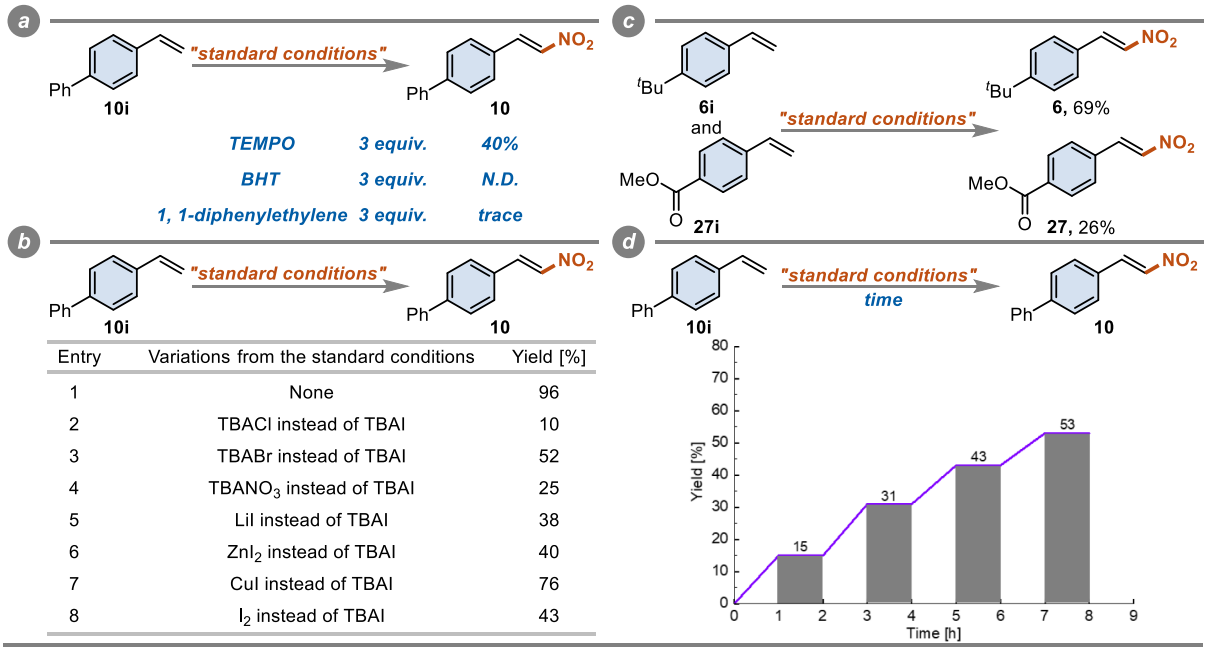
Scheme 3. Scale-Up Experiments



To detailedly probe the reaction mechanism, several trapping experiments were conducted first (Scheme 4a). When stoichiometric 2,6-di-*tert*-butyl-4-methylphenol (BHT) and 1,1-diphenyl-ethylene were added into the reaction system respectively, the yields of **10** slashed dramatically, which suggested that a radical process should be involved. Interestingly, when another general radical trap 2, 2, 6, 6-tetramethylpiperidinooxy (TEMPO) was added, **10** could still give a 40% yield, consistent with the reported ability of TEMPO for abstracting H atom from the intermediate.[10a,b] Control experiments of anions and cations were next carried out to investigate the role of the additive TBAI (Scheme 4b). When different tetrabutylammonium (TBA) salts such as TBACl, TBABr, and $TBANO_3$ were added instead, only TBABr could produce **10** in a moderate yield and the other two nearly failed, suggesting that TBAI did not act as a phase transfer catalyst in the system (entries 2−4). When different iodine salts were added to replace TBAI, the yield of **10** just slashed to some extent and CuI could render a competitive yield, revealing that the soluble iodide ion should play a key role in the reaction (entries 5−7). Meanwhile, iodine can only produce **10** in a moderate yield (entry 8). A following competition reaction of **6i** and **27i** showed the reaction reactivity was markedly affected by electronic effects (Scheme 4c). Similar outcomes were also acquired when analyzing the substrate scope. A light-on−off experiment was carried out then, reflecting that continuous light irradiation was crucial for this transformation (Scheme 4d). Moreover, the quantum yield of the reaction with **10i** as the substrate was measured to be 0.013 (see details in the Supporting Information, part IV, iv), indicating that the radical chain process was excluded from the reaction process.

Based on the above results and reported literature,[15] a plausible reaction mechanism for the present visible-light-induced stereoselective nitration of olefins was proposed in Scheme 5. Initially, a photoactive complex **I** composed of a ferric ion center and several nitrate/acetonitrile ligands was stimulated to reach an excited state **I*** under visible-light irradiation. A spontaneous β-N−O homolysis released a desired nitryl radical **III** and a Fe^{IV}=O complex **II**.

Scheme 4. Mechanistic Studies



Entry	Variations from the standard conditions	Yield [%]
1	None	96
2	TBACl instead of TBAI	10
3	TBABr instead of TBAI	52
4	$TBANO_3$ instead of TBAI	25
5	LiI instead of TBAI	38
6	ZnI_2 instead of TBAI	40
7	CuI instead of TBAI	76
8	I_2 instead of TBAI	43

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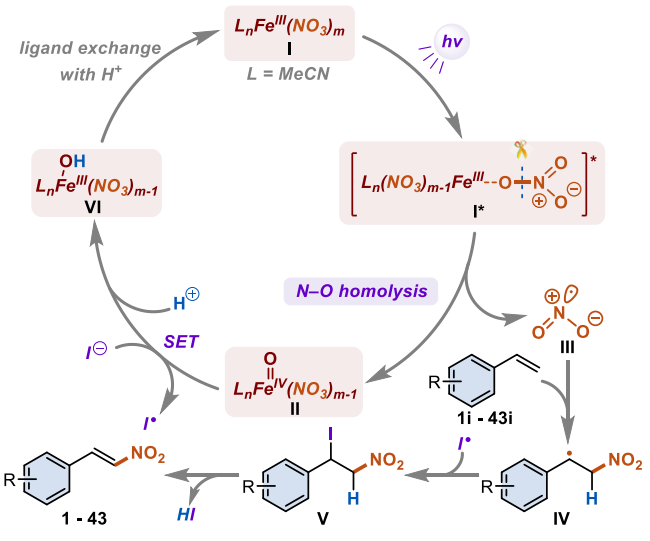
J. Org. Chem. 2025, 90, 7458−7467

Scheme 5. Proposed Mechanism



Subsequently, electrophilic addition between the electron-deficient radical **III** and olefin **i** resulted in a benzyl radical **IV**. The radical **IV** was then trapped by an iodine radical that was generated through a single electron transfer (SET) process between the Fe^{IV}=O complex **II** and the iodine ion. The *β*-iodonitro alkane **V** eliminated HI spontaneously,[16] affording the desired *E*-nitroolefins. Finally, an in situ neutralization and ligand exchange of complex **VI** regenerated the photoactive complex **I**.

CONCLUSION

Overall, we successfully developed a practical protocol for visible-light-driven stereoselective olefin nitration under simple and mild conditions. A nitryl radical generated by the unusual *β*-homolysis of a ferric-nitrate complex is the key nitrification reagent. This approach took many advantages including ligand-free conditions, high efficiency, broad scope, good functional group tolerance, and potential in late-stage functionalization. The reaction can be easily controlled by different equivalences of nitrates, and scale-up experiments in batch and flow suggest excellent practicability. We anticipate this work will inspire chemists to uncover other novel applications in nitration chemistry via nitryl radicals provided by photoinduced *β*-homolysis.

EXPERIMENTAL SECTION

General Information. Magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded on Bruker Avance NEO 400 or Bruker Avance NEO 500 with $CDCl_3$ or DMSO-d_6 or CD_3CN as the solvent, and chemical shifts were internally referenced to TMS and residual portion solvent signals (note: TMS referenced at 0.00 ppm; $CDCl_3$ referenced at 7.26 and 77.16 ppm respectively; DMSO-d_6 referenced at 2.50 and 39.52 ppm respectively; CD_3CN referenced at 1.94 ppm). Data are shown as follows: chemical shifts (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, h = heptet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, br = broad), coupling constant (J Hz). Commercially available reagents and solvents were purchased from Energy Chemical, Bide Pharmatech, and Shanghai Macklin Biochemical, and used as received unless otherwise specified. Chromatographic purification of products was accomplished by column chromatography on silica gel (Qingdao Haiyang, 100−200 mesh). Thin layer chromatography (TLC) was performed on Qingdao Haiyang 0.2 mm silica gel plates. Analytical balances Mettler Toledo ME204*E*/02 and MS205DU/A were used to weigh samples. The HRMS analysis was performed on a Q Exactive 120 plus mass spectrometer (Thermo Fisher Scientific, Germany) equipped with an electrospray ionization (ESI) source. For light-promoted reactions, 455 nm LEDs were purchased from Epistar Corp. (Taiwan, China) and assembled by Shenzhen Xinxingyuan Optoelectronics Co., Ltd. (China).

General Procedure for the Synthesis of the Starting Materials.[17] To a 100 mL three-neck flask, methyltriphenylphosphonium bromide (15 mmol, 3 equiv., 5.36 g) and dry THF (20 mL) were added with a magnetic stir bar under N_2. The mixture was allowed to stir at 0 °C for 5 min before KO^{t}Bu (15 mmol, 3 equiv., 1.68 g) was added, and a bright yellow solution was obtained. After stirring at 0 °C for 30 min, benzaldehyde (5 mmol) in dry THF was added dropwise under N_2. The mixture was allowed to stir at room temperature for 16 h. Upon completion, the reaction was quenched by 50 mL of H_2O and extracted with EtOAc (2 × 30 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (PE/EtOAc = 50:1−5:1) to give the desired olefin.

General Procedure for the Synthesis of Compounds 1−43. Olefin **1i**−**43i** (0.2 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (0.1 mmol, 0.5 equiv., 40.4 mg), TBAI (0.1 mmol, 0.5 equiv., 36.9 mg) and MeCN (2 mL) were added into a 10 mL Schlenk tube with a magnetic stir bar in sequence. The mixture was allowed to stir under N_2 with irradiation of a blue LED (30 W, 455 nm) at room temperature for 8 h. Upon completion, the reaction solution was diluted with EtOAc and washed with saturated EDTA solution and brine. The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by silica gel flash column chromatography (PE/EtOAc = 30:1−5:1) to give the desired product **1**−**43**.

(*E*)-(2-Nitrovinyl)benzene (1).[10b] According to the general procedure, **1** was obtained in 83% yield (24.7 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, J = 13.7 Hz, 1H), 7.59 (d, J = 13.6 Hz, 1H), 7.57−7.53 (m, 2H), 7.50 (d, J = 7.1 Hz, 1H), 7.48−7.43 (m, 2H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 139.2, 137.2, 132.3, 130.2, 129.5, 129.2. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_8H_8NO_2^+$, 150.0550; found, 150.0547.

(*E*)-1-Methyl-4-(2-nitrovinyl)benzene (2).[10b] According to the general procedure, **2** was obtained in 81% yield (26.4 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99 (d, J = 13.7 Hz, 1H), 7.57 (d, J = 13.6 Hz, 1H), 7.45 (d, J = 8.1 Hz, 2H), 7.26 (d, J = 8.1 Hz, 2H), 2.41 (s, 3H). ^{13}C {^{1}H} NMR (125 MHz, $CDCl_3$): δ 143.3, 139.3, 136.4, 130.3, 129.3, 127.4, 21.8. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_9H_{10}NO_2^+$, 164.0706; found, 164.0715.

(*E*)-1-Methoxy-4-(2-nitrovinyl)benzene (3).[10b] According to the general procedure, **3** was obtained in 65% yield (23.2 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 (d, J = 13.6 Hz, 1H), 7.53−7.46 (m, 3H), 6.94 (d, J = 8.3 Hz, 2H), 3.85 (s, 3H). ^{13}C {^{1}H} NMR (125 MHz, $CDCl_3$): δ 163.0, 139.1, 135.0, 131.3, 122.5, 115.0, 55.6. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_9H_{10}NO_3^+$, 180.0655; found, 180.0664.

(*E*)-1-(2-Nitrovinyl)-4-phenoxybenzene (4).[18] According to the general procedure, **4** was obtained in 60% yield (28.9 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, J = 13.6 Hz, 1H), 7.55−7.50 (m, 3H), 7.43−7.39 (m, 2H), 7.24−7.20 (m, 1H), 7.10−7.06 (m, 2H), 7.02 (d, J = 8.7 Hz, 2H). ^{13}C {^{1}H} NMR (125 MHz, $CDCl_3$): δ 161.5, 155.4, 138.7, 135.9, 131.3, 130.2, 124.9, 124.4, 120.3, 118.5. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{14}H_{12}NO_3^+$, 242.0812; found, 242.0818.

(*E*)-1-Isopropyl-4-(2-nitrovinyl)benzene (5). According to the general procedure, **5** was obtained in 85% yield (32.4 mg). Eluent: PE/EtOAc (30:1). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, J = 13.7 Hz, 1H), 7.57 (d, J = 13.6 Hz, 1H), 7.48 (d, J = 8.1 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H), 2.96 (hept, J = 7.0 Hz, 1H), 1.27 (d, J = 6.9 Hz, 6H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 154.1, 139.3,

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J. Org. Chem. 2025, 90, 7458−7467

136.5, 129.5, 127.8, 127.7, 34.4, 23.8. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{11}H_{14}NO_2^+$, 192.1019; found, 192.1017.

(*E*)-1-(*tert*-Butyl)-4-(2-nitrovinyl)benzene (6).[10h] According to the general procedure, **6** was obtained in 80% yield (32.8 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, J = 13.6 Hz, 1H), 7.61 (d, J = 13.7 Hz, 1H), 7.54−7.48 (m, 4H), 1.37 (s, 9H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 156.3, 139.2, 136.5, 129.2, 127.4, 126.6, 35.3, 31.2. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{12}H_{16}NO_2^+$, 206.1176; found, 206.1172.

(*E*)-1-Fluoro-4-(2-nitrovinyl)benzene (7).[10b] According to the general procedure, **7** was obtained in 92% yield (30.7 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 (d, J = 13.6 Hz, 1H), 7.58−7.51 (m, 3H), 7.15 (t, J = 8.6 Hz, 2H). ^{13}C {^{1}H} NMR (125 MHz, $CDCl_3$): δ 165.1 (d, J = 254.7 Hz), 138.0, 137.0, 131.4 (d, J = 8.9 Hz), 126.5 (d, J = 3.4 Hz), 116.9 (d, J = 22.4 Hz). ^{19}F NMR (376 MHz, $CDCl_3$): δ −105.7. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_8H_6FNO_2Na^+$, 190.0275; found, 190.0273.

(*E*)-1-Chloro-4-(2-nitrovinyl)benzene (8).[10b] According to the general procedure, **8** was obtained in 88% yield (32.2 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, J = 13.7 Hz, 1H), 7.56 (d, J = 13.7 Hz, 1H), 7.51−7.47 (m, 2H), 7.46−7.41 (m, 2H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 138.5, 137.8, 137.5, 130.4, 129.9, 128.7. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_8H_7ClNO_2^+$, 184.0160; found, 184.0168.

(*E*)-1-Bromo-4-(2-nitrovinyl)benzene (9).[10b] According to the general procedure, **9** was obtained in 75% yield (34.1 mg). Eluent: PE/EtOAc (30:1). Pale-yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, J = 13.7 Hz, 1H), 7.63−7.55 (m, 3H), 7.42 (d, J = 8.2 Hz, 2H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 137.9, 137.6, 132.9, 130.5, 129.1, 126.9. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_8H_7BrNO_2^+$, 227.9655; found, 227.9658.

(*E*)-4-(2-Nitrovinyl)-1,1′-biphenyl (10).[10h] According to the general procedure, **10** was obtained in 96% yield (43.2 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.05 (d, J = 13.7 Hz, 1H), 7.69 (d, J = 8.4 Hz, 2H), 7.66−7.61 (m, 5H), 7.53−7.46 (m, 2H), 7.45−7.39 (m, 1H). ^{13}C {^{1}H} NMR (125 MHz, $CDCl_3$): δ 145.1, 139.7, 138.9, 137.0, 129.8, 129.2, 129.0, 128.5, 128.1, 127.2. HR-MS (ESI) m/z: $[M + K]^+$ calcd for $C_{14}H_{11}NO_2K^+$, 264.0421; found, 264.0415.

(*E*)-Trimethyl(4-(2-nitrovinyl)phenyl)silane (11). According to the general procedure, **11** was obtained in 75% yield (33.1 mg). Eluent: PE/EtOAc (30:1). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, J = 13.6 Hz, 1H), 7.62 (d, J = 4.4 Hz, 1H), 7.60 (d, J = 6.5 Hz, 2H), 7.51 (d, J = 8.1 Hz, 2H), 0.30 (s, 9H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 146.6, 139.3, 137.3, 134.4, 130.3, 128.3, −1.2. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{11}H_{15}NO_2SiNa^+$, 244.0764; found, 244.0761.

(*E*)-2-(4-(2-Nitrovinyl)benzyl)isoindoline-1,3-dione (12). According to the general procedure, **12** was obtained in 78% yield (33.1 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^{1}H NMR (500 MHz, CD_3CN): δ 8.02 (d, J = 13.7 Hz, 1H), 7.88−7.83 (m, 2H), 7.83−7.79 (m, 2H), 7.76 (d, J = 13.7 Hz, 1H), 7.64 (d, J = 8.3 Hz, 2H), 7.44 (d, J = 8.2 Hz, 2H), 4.84 (s, 2H). ^{13}C {^{1}H} NMR (125 MHz, DMSO-d_6): δ 167.7, 140.8, 138.8, 138.0, 134.6, 131.6, 130.1, 129.5, 128.0, 123.3, 40.7. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{17}H_{12}N_2O_4Na^+$, 331.0689; found, 331.0685.

(*E*)-1-(2-Nitrovinyl)-3-vinylbenzene (13). According to the general procedure, **13** was obtained in 82% yield (28.7 mg). Eluent: PE/EtOAc (10:1). Pale-yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, J = 13.6 Hz, 1H), 7.60 (d, J = 13.7 Hz, 1H), 7.57−7.51 (m, 2H), 7.46−7.39 (m, 2H), 6.73 (dd, J = 17.6, 10.9 Hz, 1H), 5.82 (d, J = 17.6 Hz, 1H), 5.37 (d, J = 10.9 Hz, 1H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 139.1, 139.0, 137.5, 135.7, 130.5, 129.9, 129.7, 128.3, 127.1, 115.9. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{10}H_9NO_2Na^+$, 198.0525; found, 198.0522.

1,3-Bis((*E*)-2-nitrovinyl)benzene (14). According to the general procedure, **14** was obtained in 58% yield (25.5 mg). Eluent: PE/EtOAc (10:1). Yellow solid. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.39 (s, 1H), 8.31 (d, J = 13.5 Hz, 2H), 8.10 (d, J = 13.6 Hz, 2H), 7.94 (d, J = 7.8 Hz, 2H), 7.59 (t, J = 7.8 Hz, 1H). ^{13}C {^{1}H} NMR (100 MHz, DMSO-d_6): δ 139.0, 138.1, 133.5, 131.3, 130.1, 129.3. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{10}H_8N_2O_4Na^+$, 243.0376; found, 243.0372.

(*E*)-1-(2-Nitrovinyl)naphthalene (15).[10h] According to the general procedure, **15** was obtained in 85% yield (33.8 mg). Eluent: PE/EtOAc (20:1). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.84 (d, J = 13.5 Hz, 1H), 8.14 (d, J = 8.4 Hz, 1H), 8.01 (d, J = 8.2 Hz, 1H), 7.92 (d, J = 8.1 Hz, 1H), 7.76 (d, J = 7.2 Hz, 1H), 7.66 (d, J = 12.9 Hz, 2H), 7.60 (t, J = 7.3 Hz, 1H), 7.53 (t, J = 7.8 Hz, 1H). ^{13}C {^{1}H} NMR (125 MHz, $CDCl_3$): δ 138.7, 136.3, 133.9, 132.7, 131.7, 129.2, 127.9, 127.1, 127.0, 126.5, 125.5, 123.1. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{12}H_{10}NO_2^+$, 200.0706; found, 200.0713.

(*E*)-2-(2-Nitrovinyl)naphthalene (16).[10b] According to the general procedure, **16** was obtained in 86% yield (34.2 mg). Eluent: PE/EtOAc (20:1). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.13 (d, J = 13.6 Hz, 1H), 7.97 (s, 1H), 7.92−7.81 (m, 3H), 7.67 (d, J = 13.7 Hz, 1H), 7.63−7.50 (m, 3H). ^{13}C {^{1}H} NMR (125 MHz, $CDCl_3$): δ 139.3, 137.2, 135.0, 133.2, 132.4, 129.4, 128.9, 128.5, 128.0, 127.6, 127.4, 123.4. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{12}H_{10}NO_2^+$, 200.0706; found, 200.0702.

(*E*)-3-(2-Nitrovinyl)bicyclo[4.2.0]octa-1(6),2,4-triene (17).[19] According to the general procedure, **17** was obtained in 83% yield (29.1 mg). Eluent: PE/EtOAc (20:1). Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, J = 13.6 Hz, 1H), 7.55 (d, J = 13.6 Hz, 1H), 7.38 (dd, J = 7.6, 1.5 Hz, 1H), 7.24 (s, 1H), 7.13 (d, J = 7.6 Hz, 1H), 3.23 (s, 4H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 151.5, 147.2, 140.6, 136.1, 129.4, 128.9, 123.7, 122.4, 30.1, 29.5. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{10}H_{10}NO_2^+$, 176.0706; found, 176.0702.

(*E*)-1-Methyl-3-(2-nitrovinyl)benzene (18).[19] According to the general procedure, **18** was obtained in 80% yield (26.1 mg). Eluent: PE/EtOAc (20:1). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, J = 13.9 Hz, 1H), 7.55 (d, J = 13.8 Hz, 1H), 7.35−7.25 (m, 4H), 2.38 (s, 3H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 139.2, 136.8, 133.0, 129.9, 129.7, 129.2, 126.4, 21.2. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_9H_{10}NO_2^+$, 164.0706; found, 164.0714.

(*E*)-1-Methyl-2-(2-nitrovinyl)benzene (19).[10f] According to the general procedure, **19** was obtained in 78% yield (25.4 mg). Eluent: PE/EtOAc (20:1). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.30 (d, J = 13.6 Hz, 1H), 7.55−7.50 (m, 2H), 7.41 (t, J = 7.5 Hz, 1H), 7.32−7.26 (m, 2H), 2.49 (s, 3H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 139.2, 137.6, 136.7, 131.9, 131.4, 128.9, 127.4, 126.8, 19.8. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_9H_{10}NO_2^+$, 164.0706; found, 164.0705.

(*E*)-1-Chloro-2-(2-nitrovinyl)benzene (20).[10b] According to the general procedure, **20** was obtained in 80% yield (29.2 mg). Eluent: PE/EtOAc (20:1). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.40 (d, J = 13.7 Hz, 1H), 7.61−7.57 (m, 2H), 7.50 (d, J = 8.1 Hz, 1H), 7.45−7.40 (m, 1H), 7.34 (t, J = 7.6 Hz, 1H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 139.0, 136.2, 135.2, 133.0, 130.9, 128.7, 128.7, 127.6. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_8H_7ClNO_2^+$, 184.0160; found, 184.0159.

(*E*)-1,4-Dimethyl-2-(2-nitrovinyl)benzene (21).[10f] According to the general procedure, **21** was obtained in 72% yield (25.4 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, J = 13.6 Hz, 1H), 7.49 (d, J = 13.5 Hz, 1H), 7.31 (s, 1H), 7.22−7.13 (m, 2H), 2.42 (s, 3H), 2.34 (s, 3H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 137.3, 136.8, 136.3, 136.3, 132.9, 131.3, 128.7, 127.8, 20.8, 19.4. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{10}H_{12}NO_2^+$, 178.08636; found, 178.0869.

(*E*)-1,3,5-Trimethyl-2-(2-nitrovinyl)benzene (22).[10b] According to the general procedure, **22** was obtained in 63% yield (24.1 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25 (d, J = 13.9 Hz, 1H), 7.29 (d, J = 13.9 Hz, 1H), 6.96 (s, 2H), 2.39 (s, 6H), 2.32 (s, 3H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 140.9, 139.6, 138.5, 136.5, 129.9, 125.7, 21.5, 21.2. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{11}H_{14}NO_2^+$, 192.1019; found, 192.1017.

(*E*)-1-Chloro-2-fluoro-4-(2-nitrovinyl)benzene (23). According to the general procedure, **23** was obtained in 85% yield (34.1 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^{1}H NMR (400 MHz,

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J. Org. Chem. 2025, 90, 7458−7467

$CDCl_3$): δ 7.92 (d, J = 13.7 Hz, 1H), 7.54 (d, J = 13.8 Hz, 1H), 7.50 (t, J = 7.8 Hz, 1H), 7.36−7.27 (m, 2H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 158.5 (d, J = 251.5 Hz), 138.4, 136.8 (d, J = 2.5 Hz), 131.9, 130.6 (d, J = 7.2 Hz), 125.8 (d, J = 3.7 Hz), 125.2 (d, J = 17.9 Hz), 116.6 (d, J = 22.0 Hz). ^{19}F NMR (376 MHz, $CDCl_3$): δ −112.9. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_8H_6FClNO_2^+$, 202.0066; found, 202.0064.

(*E*)-1,2-Dichloro-4-(2-nitrovinyl)benzene (24).[20] According to the general procedure, **24** was obtained in 83% yield (35.9 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^1H NMR (500 MHz, $CDCl_3$): δ 7.90 (d, J = 13.8 Hz, 1H), 7.64 (s, 1H), 7.59−7.51 (m, 2H), 7.39 (d, J = 8.3 Hz, 1H). ^{13}C {^1H} NMR (125 MHz, $CDCl_3$): δ 138.4, 136.5, 136.5, 134.1, 131.6, 130.7, 130.1, 128.0. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_8H_6Cl_2NO_2^+$, 217.9770; found, 217.9772.

(*E*)-1-(2-Nitrovinyl)-4-(trifluoromethyl)benzene (25).[10f] According to the general procedure, **25** was obtained in 95% yield (41.2 mg). Eluent: PE/EtOAc (10:1). Pale-yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, J = 13.8 Hz, 1H), 7.74−7.65 (m, 4H), 7.63 (d, J = 13.8 Hz, 1H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 139.0, 137.3, 133.6, 133.1 (q, J = 33.0 Hz), 129.4, 126.4 (q, J = 3.9 Hz), 123.6 (q, J = 272.5 Hz). ^{19}F NMR (376 MHz, $CDCl_3$): δ −63.2. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_9H_7F_3NO_2^+$, 218.0423; found, 218.0428.

Methyl (*E*)-4-(2-nitrovinyl)benzoate (26).[19] According to the general procedure, **26** was obtained in 92% yield (38.1 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^1H NMR (500 MHz, $CDCl_3$): δ 8.11 (d, J = 8.0 Hz, 2H), 8.01 (d, J = 13.7 Hz, 1H), 7.65−7.59 (m, 3H), 3.95 (s, 3H). ^{13}C {^1H} NMR (125 MHz, $CDCl_3$): δ 166.1, 138.8, 137.7, 134.3, 133.2, 130.6, 129.1, 52.6. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{10}H_9NO_4Na^+$, 230.0424; found, 230.0420.

(*E*)-4-(2-Nitrovinyl)benzonitrile (27).[10b] According to the general procedure, **27** was obtained in 98% yield (34.1 mg). Eluent: PE/EtOAc (10:1). Pale-yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ 7.99 (d, J = 13.7 Hz, 1H), 7.75 (d, J = 7.9 Hz, 2H), 7.71−7.48 (m, 3H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 139.6, 136.7, 134.5, 133.1, 129.5, 117.9, 115.3. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_9H_6N_2O_2Na^+$, 197.0321; found, 197.0321.

(*E*)-1-(Methylsulfonyl)-4-(2-nitrovinyl)benzene (28). According to the general procedure, **28** was obtained in 97% yield (44.1 mg). Eluent: PE/EtOAc (4:1). Pale-yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.06−7.97 (m, 3H), 7.75 (d, J = 8.0 Hz, 2H), 7.64 (d, J = 13.8 Hz, 1H), 3.09 (s, 3H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 143.2, 139.8, 136.6, 135.4, 129.9, 128.5, 44.5. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_9H_9NO_4SNa^+$, 250.0144; found, 250.0141.

(2-Nitroethene-1,1-diyl)dibenzene (29).[10h] According to the general procedure, **29** was obtained in 62% yield (27.9 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ 7.52−7.44 (m, 5H), 7.42 (t, J = 7.6 Hz, 2H), 7.34−7.30 (m, 2H), 7.28−7.23 (m, 2H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 150.6, 137.2, 135.6, 134.5, 131.0, 129.4, 129.0, 129.0, 128.9, 128.6. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{14}H_{12}NO_2^+$, 226.0863; found, 226.0860.

4,4′-(2-Nitroethene-1,1-diyl)bis(methoxybenzene) (30).[21] According to the general procedure, **30** was obtained in 55% yield (31.3 mg). Eluent: PE/EtOAc (20:1). Yellow solid. ^1H NMR (500 MHz, $CDCl_3$): δ 7.37 (s, 1H), 7.23 (d, J = 8.9 Hz, 2H), 7.16 (d, J = 8.8 Hz, 2H), 6.94 (d, J = 8.7 Hz, 2H), 6.89 (d, J = 8.9 Hz, 2H), 3.86 (s, 3H), 3.84 (s, 3H). ^{13}C {^1H} NMR (125 MHz, $CDCl_3$): δ 162.2, 160.8, 151.0, 132.4, 131.1, 131.1, 130.0, 127.9, 114.4, 113.9, 55.6, 55.5. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{16}H_{16}NO_4^+$, 286.1074; found, 286.1071.

3-Nitro-1,2-dihydronaphthalene (31).[10b] According to the general procedure, **31** was obtained in 72% yield (25.2 mg). Eluent: PE/EtOAc (30:1). Yellow solid. ^1H NMR (500 MHz, $CDCl_3$): δ 7.84 (s, 1H), 7.39−7.31 (m, 2H), 7.30−7.26 (m, 1H), 7.26−7.21 (m, 1H), 3.23−2.92 (m, 4H). ^{13}C {^1H} NMR (125 MHz, $CDCl_3$): δ 148.0, 136.5, 131.8, 131.4, 130.3, 130.2, 128.1, 127.5, 28.1, 22.5. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{10}H_{10}NO_2^+$, 176.0706; found, 176.0703.

2-Nitro-2,3,4,5-tetrahydro-1,1′-biphenyl (32).[10f] According to the general procedure, **32** was obtained in 45% yield (18.2 mg). Eluent: PE/EtOAc (30:1). Yellow oil. ^1H NMR (400 MHz, $CDCl_3$): δ 7.33−7.24 (m, 4H), 6.61−6.30 (m, 1H), 5.84−5.43 (m, 1H), 2.56−2.35 (m, 3H), 2.35−2.08 (m, 2H), 1.95−1.70 (m, 2H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 138.9, 133.9, 131.4, 128.8, 127.9, 125.6, 83.1, 29.1, 25.5, 17.4. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{12}H_{14}NO_2^+$, 204.1019; found, 204.1029.

(*E*)-1-(Nitromethylene)-1,2,3,4-tetrahydronaphthalene (33).[10h] According to the general procedure, **33** was obtained in 65% yield (24.5 mg). Eluent: PE/EtOAc (30:1). Yellow oil. ^1H NMR (400 MHz, $CDCl_3$): δ 7.24−7.17 (m, 4H), 6.33 (t, J = 4.7 Hz, 1H), 5.29 (s, 2H), 2.85 (t, J = 8.2 Hz, 2H), 2.47−2.40 (m, 2H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 136.1, 136.0, 132.0, 128.2, 128.1, 127.8, 126.9, 122.2, 78.1, 27.4, 23.4. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{11}H_{11}NO_2Na^+$, 212.0682; found, 212.0682.

(*E*)-3-(2-Nitrovinyl)quinoline (34).[22] According to the general procedure, **34** was obtained in 90% yield (35.9 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^1H NMR (500 MHz, $CDCl_3$): δ 9.06 (s, 1H), 8.33 (s, 1H), 8.21−8.10 (m, 2H), 7.89 (d, J = 7.9 Hz, 1H), 7.86−7.74 (m, 2H), 7.64 (t, J = 7.4 Hz, 1H). ^{13}C {^1H} NMR (125 MHz, $CDCl_3$): δ 149.3, 148.9, 138.2, 137.6, 136.0, 132.0, 129.8, 128.7, 128.2, 127.5, 123.4. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{11}H_9N_2O_2^+$, 201.0659; found, 201.0656.

(*E*)-2-(2-Nitrovinyl)pyridine (35).[10b] According to the general procedure, **35** was obtained in 88% yield (26.4 mg). Eluent: PE/EtOAc (5:1). Yellow oil. ^1H NMR (400 MHz, $CDCl_3$): δ 8.65 (d, J = 4.8 Hz, 1H), 7.98 (d, J = 13.1 Hz, 1H), 7.89 (d, J = 13.0 Hz, 1H), 7.77 (t, J = 7.7 Hz, 1H), 7.46 (d, J = 7.7 Hz, 1H), 7.36 (m, 1H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 150.7, 149.4, 140.7, 137.3, 137.3, 126.4, 125.8. HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_7H_7N_2O_2^+$, 151.0502; found, 151.0512.

(*E*)-4-Methyl-5-(2-nitrovinyl)thiazole (36).[23] According to the general procedure, **36** was obtained in 84% yield (28.5 mg). Eluent: PE/EtOAc (20:1). Yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.82 (s, 1H), 8.17 (d, J = 13.3 Hz, 1H), 7.37 (d, J = 13.3 Hz, 1H), 2.61 (s, 3H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 160.3, 155.1, 137.0, 129.0, 123.0, 16.0. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_6H_6N_2O_2SNa^+$, 193.0042; found, 193.0040.

2,6-Diisopropylphenyl (*E*)-4-(2-nitrovinyl)benzoate (37). According to the general procedure, **37** was obtained in 71% yield (50.1 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^1H NMR (500 MHz, $CDCl_3$): δ 8.32 (d, J = 7.9 Hz, 2H), 8.08 (d, J = 13.8 Hz, 1H), 7.76−7.65 (m, 3H), 7.31−7.21 (m, 3H), 2.95 (hept, J = 6.7 Hz, 2H), 1.21 (d, J = 3.4 Hz, 12H). ^{13}C {^1H} NMR (125 MHz, $CDCl_3$): δ 164.3, 145.7, 140.4, 139.0, 137.5, 135.0, 132.2, 131.1, 129.4, 126.9, 124.2, 27.8, 24.0, 22.7. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{21}H_{23}NO_4Na^+$, 376.1519; found, 376.1516.

5-Chloro-2-(2,4-dichlorophenoxy)phenyl (*E*)-4-(2-nitrovinyl)benzoate (38). According to the general procedure, **38** was obtained in 70% yield (64.8 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, J = 8.5 Hz, 2H), 8.03 (d, J = 13.7 Hz, 1H), 7.64 (dd, J = 11.0, 2.7 Hz, 3H), 7.34 (dd, J = 5.6, 2.5 Hz, 2H), 7.24 (dd, J = 8.8, 2.5 Hz, 1H), 7.16 (dd, J = 8.8, 2.5 Hz, 1H), 6.92 (dd, J = 8.8, 2.9 Hz, 2H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 163.1, 151.0, 146.8, 141.6, 139.2, 137.4, 135.3, 131.3, 131.2, 130.5, 129.7, 129.5, 129.2, 128.3, 127.5, 126.1, 124.6, 120.6, 120.5. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{21}H_{12}Cl_3NO_5Na^+$, 485.9673; found, 485.9667.

2-Methoxy-4-(3-oxobutyl)phenyl (*E*)-4-(2-nitrovinyl)-benzoate (39). According to the general procedure, **39** was obtained in 64% yield (47.2 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.22 (d, J = 8.0 Hz, 2H), 7.99 (d, J = 13.7 Hz, 1H), 7.71−7.59 (m, 3H), 7.02 (d, J = 8.0 Hz, 1H), 6.83 (s, 1H), 6.78 (d, J = 8.1 Hz, 1H), 3.76 (s, 3H), 2.88 (t, J = 7.4 Hz, 2H), 2.76 (t, J = 7.5 Hz, 2H), 2.13 (s, 3H). ^{13}C {^1H} NMR (100 MHz, $CDCl_3$): δ 207.7, 163.8, 150.8, 140.5, 138.8, 137.8, 137.5, 134.7, 132.1, 131.0, 129.1, 122.5, 120.4, 112.7, 55.8, 44.9, 30.0, 29.5. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{20}H_{19}NO_6Na^+$, 392.1105; found, 392.1098.

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J. Org. Chem. 2025, 90, 7458−7467

2-Isopropyl-5-methylphenyl (*E*)-4-(2-nitrovinyl)benzoate (40). According to the general procedure, **40** was obtained in 76% yield (49.4 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.29 (d, J = 8.4 Hz, 2H), 8.07 (d, J = 13.7 Hz, 1H), 7.75−7.64 (m, 3H), 7.27 (d, J = 7.8 Hz, 1H), 7.10 (d, J = 7.8 Hz, 1H), 6.98−6.92 (m, 1H), 3.04 (hept, J = 6.9 Hz, 1H), 2.36 (s, 3H), 1.23 (d, J = 6.9 Hz, 6H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 164.4, 148.0, 139.0, 137.5, 137.1, 136.9, 134.9, 132.6, 131.1, 129.3, 127.6, 126.7, 122.8, 27.4, 23.2, 21.0. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{19}NO_4Na^+$, 348.1206; found, 348.1201.

1,7,7-Trimethylbicyclo[2.2.1]heptan-2-yl (*E*)-4-(2-nitrovinyl)benzoate (41). According to the general procedure, **41** was obtained in 73% yield (47.9 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, J = 8.0 Hz, 2H), 8.02 (d, J = 13.7 Hz, 1H), 7.66−7.60 (m, 3H), 5.12 (d, J = 9.8 Hz, 1H), 2.51−2.42 (m, 1H), 2.13−2.04 (m, 1H), 1.85−1.73 (m, 2H), 1.46−1.37 (m, 1H), 1.34−1.26 (m, 1H), 1.14−1.08 (m, 1H), 0.95 (s, 3H), 0.91 (s, 6H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 165.8, 138.7, 137.8, 134.1, 133.8, 130.5, 129.1, 81.3, 49.2, 48.0, 45.0, 36.9, 28.2, 27.5, 19.8, 19.0, 13.7. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{19}H_{23}NO_4Na^+$, 352.1519; found, 352.1515.

(2,2-Dimethyl-1,3-dioxolan-4-yl)methyl (*E*)-4-(2-nitrovinyl)-benzoate (42). According to the general procedure, **42** was obtained in 72% yield (44.2 mg). Eluent: PE/EtOAc (5:1). Pale-yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.11 (d, J = 8.0 Hz, 2H), 8.00 (d, J = 13.7 Hz, 1H), 7.65−7.59 (m, 3H), 4.47−4.39 (m, 2H), 4.38−4.32 (m, 1H), 4.14 (dd, J = 8.5, 6.3 Hz, 1H), 3.86 (dd, J = 8.5, 5.6 Hz, 1H), 1.44 (s, 3H), 1.37 (s, 3H). ^{13}C {^{1}H} NMR (125 MHz, $CDCl_3$): δ 165.4, 138.8, 137.6, 134.5, 132.7, 130.7, 129.1, 110.1, 73.6, 66.3, 65.6, 26.8, 25.4. HR-MS (ESI) m/z: $[M + Na]^+$ calcd for $C_{15}H_{17}NO_6Na^+$, 330.0948; found, 330.0946.

(*E*)-1-Benzyl 2-(4-(2-nitrovinyl)benzyl) Pyrrolidine-1,2-dicarboxylate (43). According to the general procedure, **43** was obtained in 68% yield (55.8 mg). Eluent: PE/EtOAc (5:1). Yellow oil. The product gives two sets of NMR signals, owing to the presence of rotamers around the tertiary amide. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (t, J = 13.3 Hz, 1H), 7.60−7.51 (m, 2H), 7.43 (t, J = 7.6 Hz, 2H), 7.37−7.34 (m, 2H), 7.30−7.24 (m, 4H), 5.22 (s, 1H), 5.16 (d, J = 6.2 Hz, 1H), 5.10−5.02 (m, 2H), 4.51−4.38 (m, 1H), 3.65−3.49 (m, 2H), 2.31−2.21 (m, 1H), 2.04−1.91 (m, 3H). ^{13}C {^{1}H} NMR (100 MHz, $CDCl_3$): δ 172.6 (172.5), 155.0 (154.3), 140.2 (139.9), 138.6 (138.4), 137.5 (137.4), 136.7 (136.6), 130.0 (129.9), 129.4 (129.4), 128.7 (128.6), 128.6 (128.5), 128.1 (128.0), 127.9 (127.8), 67.1 (67.1), 65.9 (65.8), 59.4 (59.0), 47.1 (46.6), 31.0 (30.0), 24.5 (23.6). HR-MS (ESI) m/z: $[M + H]^+$ calcd for $C_{22}H_{23}N_2O_6^+$, 411.1551; found, 411.1555.

■ ASSOCIATED CONTENT

Data Availability Statement

The data underlying this study are available in the published article and its Supporting Information.

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.joc.5c00908.

Mechanism investigations, synthetic procedures, characterization data, and NMR spectra of these synthesized compounds (PDF)

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Notes

The authors declare no competing financial interest.

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