

Leveraging Radical Ligand Transfer Catalysis for the Construction of Carbon-Heteroatom Bonds

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Abstract: Radical ligand transfer (RLT) has emerged as a powerful strategy for the selective construction of carbon–heteroatom bonds through the interception of carbon-centred radicals by redox-active metal complexes. This review highlights recent contributions from our group to RLT-mediated catalysis synergized with electrochemical, photochemical, and mechanochemical activation modes. These RLT-based strategies enabled the development of electrochemical halonitration reactions, photoredox nitrative difunctionalization, and carbochlorination of alkenes, and more recently, mechanochemical nitronitroxylation, and vicinal dihalogenation reactions. Together, these studies establish RLT as a versatile and sustainable platform for radical-mediated carbon–heteroatom bond formation.

Keywords: Alkene difunctionalization · Cobalt catalysis · Iron catalysis · Radical ligand transfer · Synergistic catalysis



Anthony J. Fernandes earned his PhD in 2018 from the Université de Bordeaux, France, in the Y. Landais group. After a postdoc in collaboration with the groups of F. Leroux (Université Strasbourg) and S. Thibaudau (University Poitiers), he joined the N. Maulide group in 2020 (University of Vienna). He was the recipient of a Global MSCA fellowship and an SNSF fellowship in 2022, he received a Holcim Stiftung in

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Dmitry Katayev studied at the D. I. Mendeleev University of Technology (HCC RAS, Moscow), where he obtained his MSc. He then received his PhD in the group of Prof. E. P. Kündig at the University of Geneva. After two postdoctoral stays funded by the SNSF in the groups of Prof. L. J. Goossen at TU Kaiserslautern and Prof. A. Togni at ETH Zurich, he was awarded an Ambizione grant and launched his independent research

at ETH Zurich in 2018. As a recipient of an Eccellenza Professorial Grant, he then joined the University of Fribourg. In 2022, he was appointed as an assistant professor with tenure track at the University of Bern. His research focuses on developing innovative catalytic radical strategies for the selective construction of C–C, C–halogen, and C–heteroatom bonds, driven by sustainable activation modes such as visible-light, electrochemistry, and mechanochemistry.

1. Introduction

The selective construction of carbon–heteroatom bonds is a central objective in synthetic organic chemistry, given the ubiquitous presence of heteroatom-containing motifs in pharmaceuticals, agrochemicals, natural products, and functional materials.^[1–3] Among the numerous approaches developed for this purpose, radical-mediated transformations have emerged as particularly powerful strategies because they operate under mild conditions, exhibit broad functional-group tolerance, and display unique reactivity profiles.^[4] In recent years, substantial progress in photoredox catalysis, electrochemistry, and transition-metal catalysis has significantly expanded the synthetic utility of carbon-centred radicals for functionalization reactions.^[5–8]

Despite these advances, the selective conversion of transient alkyl radicals into C–heteroatom bonds remains a major challenge. Traditional radical processes often rely on atom-transfer pathways^[9] or on oxidation events, leading to radical-polar crossover mechanisms.^[10–13] In this context, radical ligand transfer (RLT) has recently emerged as a conceptually distinct and versatile strategy for radical functionalization.^[14–19] Inspired by mechanistic paradigms operating in metalloenzymes such as cytochrome P450,^[20] where radical rebound processes govern selective C–H functionalization *via* transient carbon-centred radical intermediates (Fig. 1), RLT involves the outer-sphere transfer of a ligand from a redox-active metal complex to a carbon-centred radical, enabling direct formation of C–heteroatom bonds while simultaneously reducing the metal centre through asynchronous single-electron transfer (SET).^[20–25] This mechanistic platform offers several important advantages. First, RLT enables catalytic, selective radical functionalization using inexpensive, readily available nucleophilic heteroatom sources.^[24,26,27] Second, because the ligand-transfer step proceeds rapidly through radical intermediates, these transformations often outcompete alternative pathways and display excellent compatibility with sensitive functional groups (FGs) and electronically diverse substrates.

Over the past several years, our group has focused on the development of various catalytic radical reactions.^[13,28–31] In particular, we have focused on the merger of RLT catalysis with electrochem-

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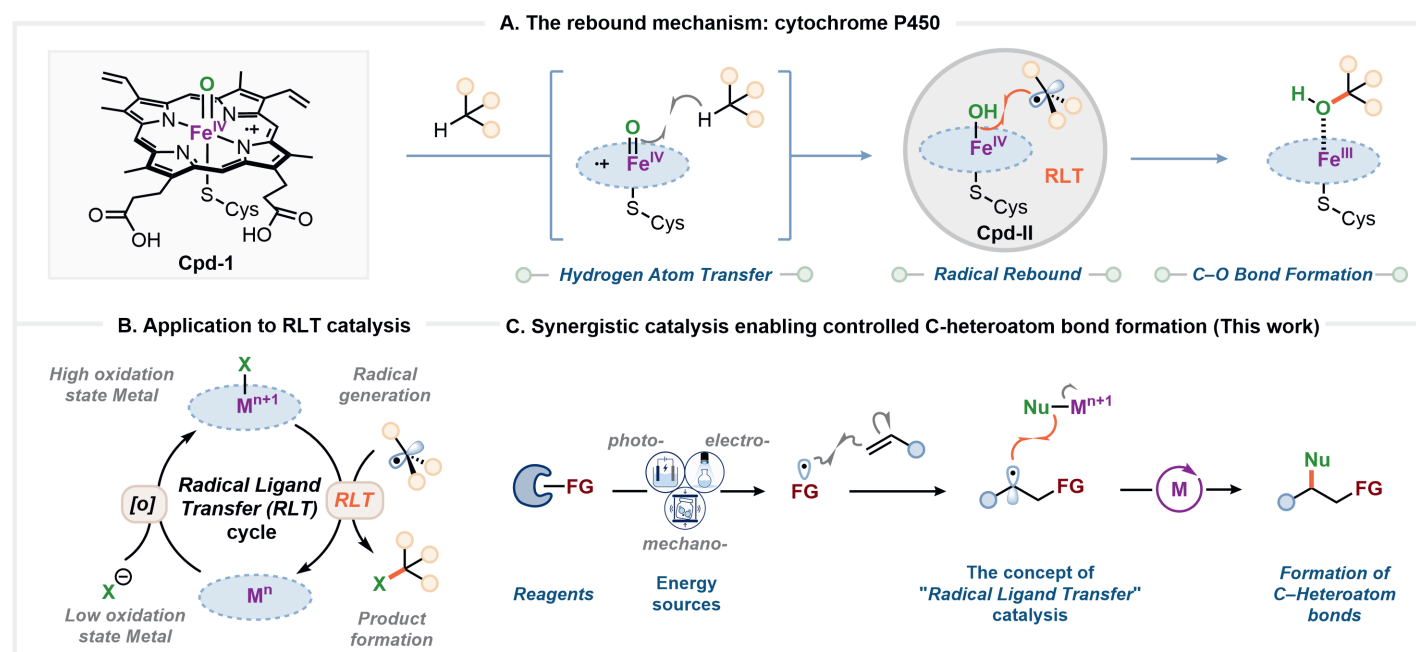


Fig. 1. (A–B) The radical rebound mechanism and application of its principles to RLT catalysis and C–X bond construction. (C) Our group's focus: merging RLT catalysis with diverse energy sources to enable controlled, modular difunctionalization of alkenes.

ical, photochemical, and mechanochemical activation strategies (Fig. 1C). These concepts allowed us to successfully develop selective and modular alkene difunctionalization reactions, including nitrate difunctionalization,^[32,33] carbahlogenation,^[34] nitronitroxylation,^[35] and vicinal dihalogenation^[36] reactions. These studies demonstrated that transient alkyl radicals generated *via* various activation modes can be efficiently intercepted by transition-metal complexes through RLT processes, enabling the selective installation of halide and nitrate functionalities under mild conditions.

In this review, we summarize our recent contributions in this area, with particular emphasis on mechanistic design principles and the role of radical ligand transfer catalysis in enabling modular radical-mediated difunctionalization reactions.

2. Merging RLT and Electrochemistry

In our first contribution to RLT-mediated catalysis, we developed a unified protocol for nitrative functionalization of unsaturated hydrocarbons.^[33]

Electrochemically assisted bromonitration and chloronitration of alkenes, in the presence of stoichiometric iron nitrate and lithium halide, proceeded with excellent chemo- and regioselectivity, providing access to a wide range of β -halogenated nitroalkanes with excellent scalability up to decagrams (Fig. 2). The methodology exhibited broad functional-group tolerance, accommodating alcohols, ethers, azides, aldehydes, and esters, and was compatible with Michael acceptors and complex biomolecule-derived substrates. In addition, the protocol tolerated various simple, unactivated alkenes, as shown in example **2d** (Fig. 2). Extensive mechanistic studies on the halonitration reaction were conducted. These studies (including pulse radiolysis, cyclic voltammetry, radical-clock experiments, Hammett analysis, and on/off electrolysis experiments) supported the formation of nitril radicals *via* single-electron reduction of $\text{Fe}(\text{NO}_3)_3$ at the cathode (Fig. 3). Following the addition of this nitril radical onto an alkene substrate, the resulting β -nitroalkyl radical intermediate subsequently undergoes RLT with the iron(III) halide species, ultimately producing the desired halonitration product and regeneration of an iron(II) species.

In contrast to conventional electrocatalysis, this strategy demonstrated that electrons are carried out throughout the catalytic cycle, enabling redox-neutral radical transformations with only a catalytic amount of electricity (so-called 'electron catalysis').^[37]

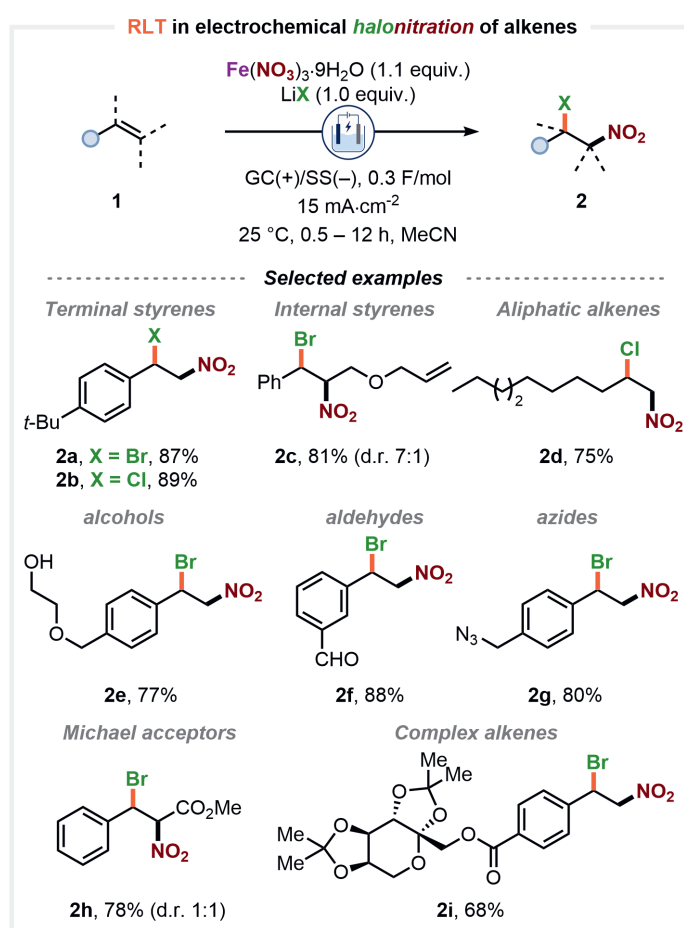


Fig. 2. RLT-mediated electrochemical halonitration of unsaturated hydrocarbons.

3. Merging RLT and Photochemistry

Building on these initial electrochemical studies, we next sought to merge RLT catalysis with visible-light photoredox activation. In this context, our group developed a dual photoredox/cobalt catalytic platform for the nitrative difunctionalization of

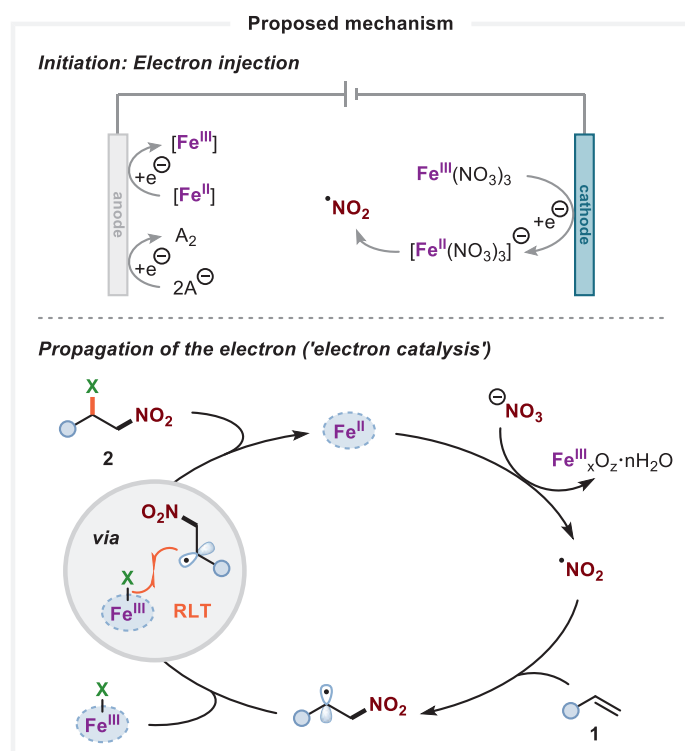


Fig. 3. Proposed mechanism.

unsaturated hydrocarbons, enabling the synthesis of synthetically valuable 1,2-halonitroalkanes under mild and redox-neutral conditions. The strategy combines visible-light photoredox catalysis with cobalt-mediated RLT, using bench-stable *N*-nitrosuccinimide as a controllable nitryl radical precursor and inexpensive ammonium halides as the halogen sources.^[32]

Under the optimized conditions, highly regioselective chloronitration and bromonitration of a broad range of activated and unactivated alkenes were achieved with excellent functional-group tolerance (Fig. 4). Electron-rich, electron-poor, α -substituted, internal, and complex olefins all underwent efficient difunctionalization, while sensitive groups such as esters, aldehydes, and nitro substituents remained intact. The methodology was also successfully applied to the late-stage functionalization of structurally complex molecules.

Mechanistically, the reaction operates through photoinduced single-electron reduction of *N*-nitrosuccinimide to generate the nitryl radical, which undergoes regioselective addition onto the olefin to form a transient alkyl radical intermediate (Fig. 5). This species is subsequently intercepted by high-valent cobalt halide species *via* an RLT process to form C–Cl or C–Br bonds, thereby regenerating the cobalt catalyst and completing the dual catalytic cycle. Mechanistic investigations, including Stern–Volmer quenching studies, Hammett analysis, radical-clock experiments, quantum-yield measurements, and stereochemical probes, supported the proposed mechanism and excluded alternative carbocationic or radical-chain pathways.

Interestingly, in the absence of the cobalt catalyst, the β -nitroalkyl radical intermediate was found to be oxidized by the iridium photocatalyst, thereby shifting the mechanism from an RLT manifold to a radical-polar crossover process. This divergent reactivity was subsequently leveraged to develop intermolecular nitrate difunctionalization reactions involving various *O*-, *S*-, and *N*-centred nucleophiles.

Beyond the direct radical nitration of alkenes, our group later reported a complementary carbochlorination strategy involving electrophilic alkyl radicals^[28,38,39] bearing an electron-withdrawing group in the α -position, such as α -nitroalkyl and diverse

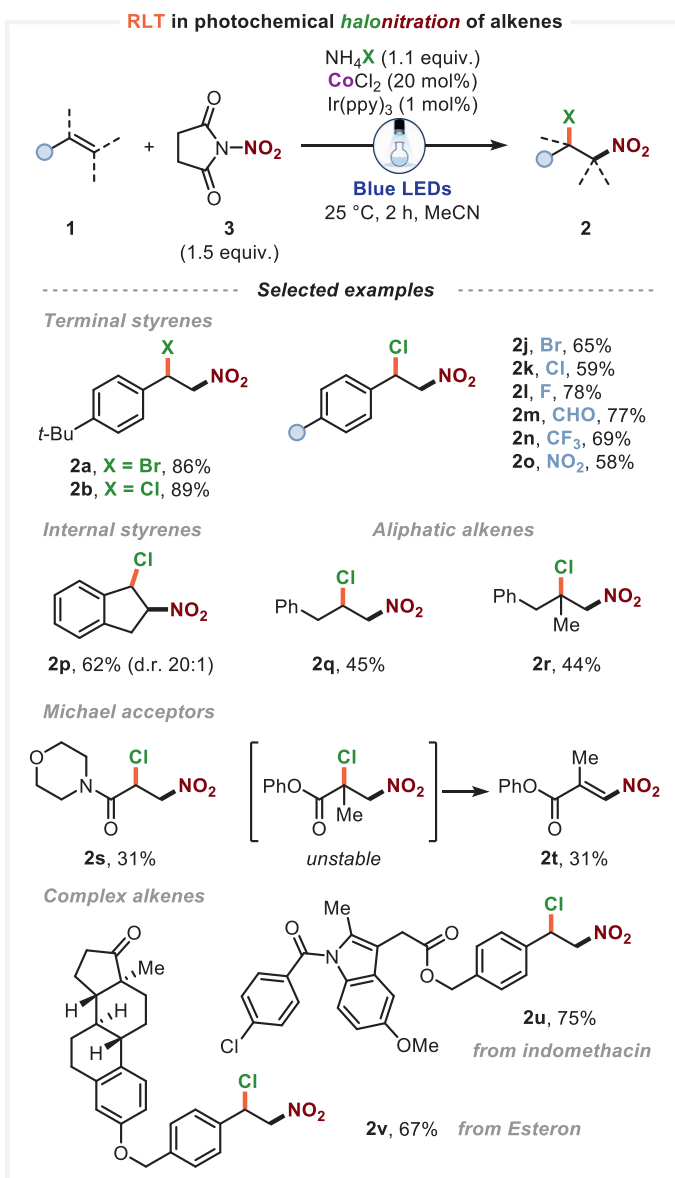


Fig. 4. RLT-mediated photochemical halonitration of alkenes.

fluorinated radicals (Fig. 6). This methodology combines visible-light photoredox catalysis with cobalt-mediated RLT catalysis to selectively intercept transient alkyl radicals from bromoalkanes and promote chlorine atom transfer, thereby circumventing con-

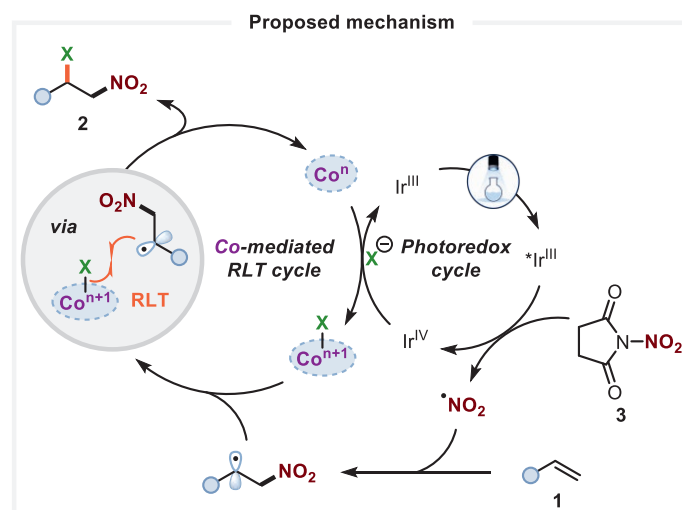


Fig. 5. Proposed mechanism.

ventional atom-transfer radical addition (ATRA) pathways that commonly dominate when brominated radical precursors are employed. A major challenge in this transformation stems from the simultaneous release of bromide ions during radical generation, which can interfere with the desired chlorine transfer. In this context, silver carbonate proves crucial for suppressing bromine atom transfer and promoting the selective chlorination event.

The reaction was demonstrated on a broad range of aliphatic alkenes and displayed excellent functional-group tolerance (Fig. 6). While α -bromo nitroalkanes served as convenient radical precursors, other redox-active precursors, such as iodo- and pyridinium N-oxide derivatives, also proved effective under the reaction conditions.

Mechanistically, this transformation follows a catalytic manifold (Fig. 7) analogous to that described for the previous halonitration reaction. Computational studies, performed by DFT at the (U)M06L-D3/Def2TZVP,SMD(MeCN) // (U)M06L-D3/Def2SVP,SMD(MeCN) level of theory, revealed that cobalt-mediated RLT is both kinetically (barrierless)^[40,41] and thermodynamically favoured over the competing ATRA pathways and further clarified the critical role of Ag_2CO_3 . In particular, silver carbonate acts both as a bromide-ion scavenger, suppressing undesired bromine atom transfer, and as a promoter of cobalt oxidation, facilitating the formation of the active Co^{III} chlorinating species.

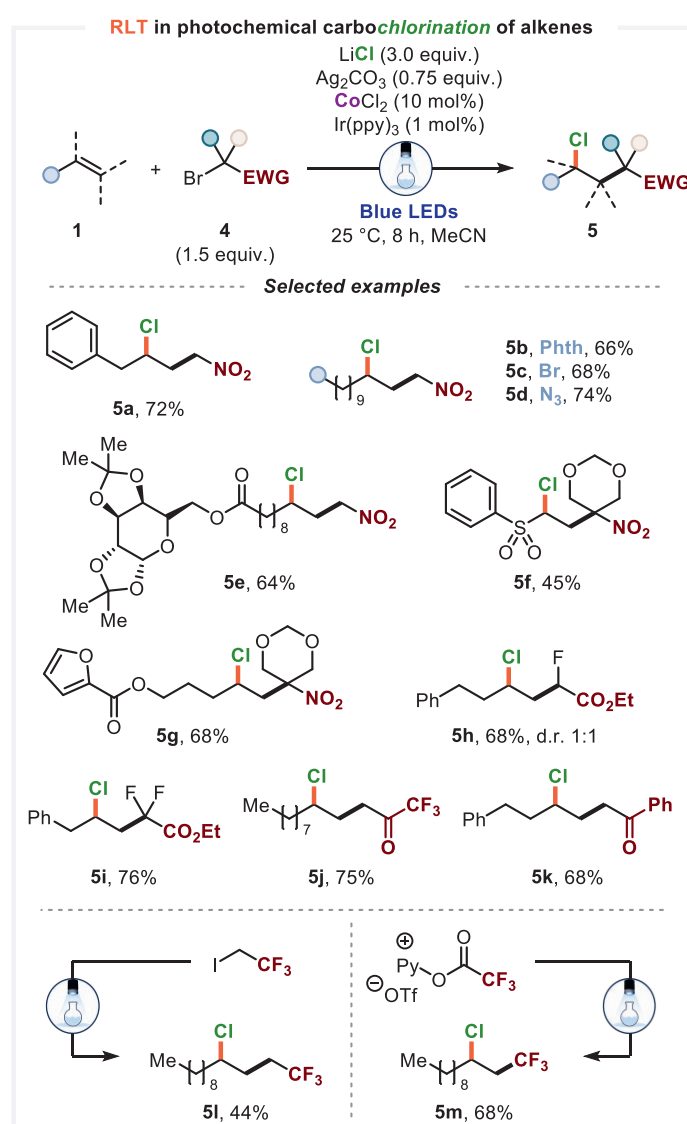


Fig. 6. RLT-mediated photochemical carbohalogenation of alkenes.

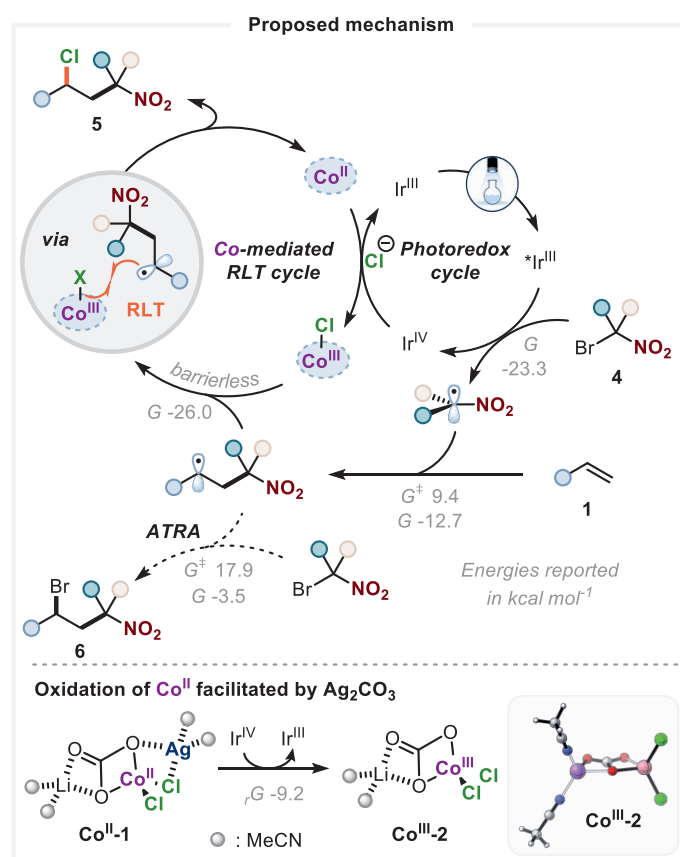


Fig. 7. Proposed mechanism and key Co^{III} species.

4. Merging RLT and Mechanochemistry

Following our efforts to develop various RLT-based transformations in solution, we became interested in extending these catalytic manifolds to mechanochemistry, an emerging activation mode that offers significant advantages for sustainability and solvent minimization.

Capitalizing on our previous electrochemical halonitration strategy, we first explored the nitronitroxylation of alkenes under solvent-free ball milling or liquid-assisted grinding (LAG) conditions by merging RLT and electron-driven nitration chemistry.^[35] We envisioned that ferric nitrate could simultaneously serve as a nitril radical precursor upon SET reduction and as a source of nitrate. In the presence of catalytic amounts of (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO) and mechanical force, ferric nitrate undergoes single-electron reduction to generate nitril radicals (Fig. 8). Following radical addition onto the alkene, the resulting β -nitroalkyl radical intermediate is intercepted through iron-mediated RLT to install a nitrate functionality, ultimately enabling highly selective 1,2-nitronitroxylation reactions.

Optimization studies revealed that ball milling dramatically enhances the reaction efficiency relative to analogous liquid-phase conditions. Under optimized solvent-free conditions using a stainless-steel milling jar, a broad range of activated and unactivated alkenes underwent chemo- and regioselective nitronitroxylation in high yields (Fig. 9).

The methodology tolerated numerous sensitive functionalities, including halides, aldehydes, esters, nitrate esters, amides, free acids, and sulfamides (Fig. 9). Furthermore, complex substrates could be efficiently functionalized under the mechanochemical conditions.

Encouraged by these findings, our group subsequently extended mechanochemical RLT catalysis to the vicinal dihalogenation of unsaturated hydrocarbons using inexpensive and bench-stable halogen

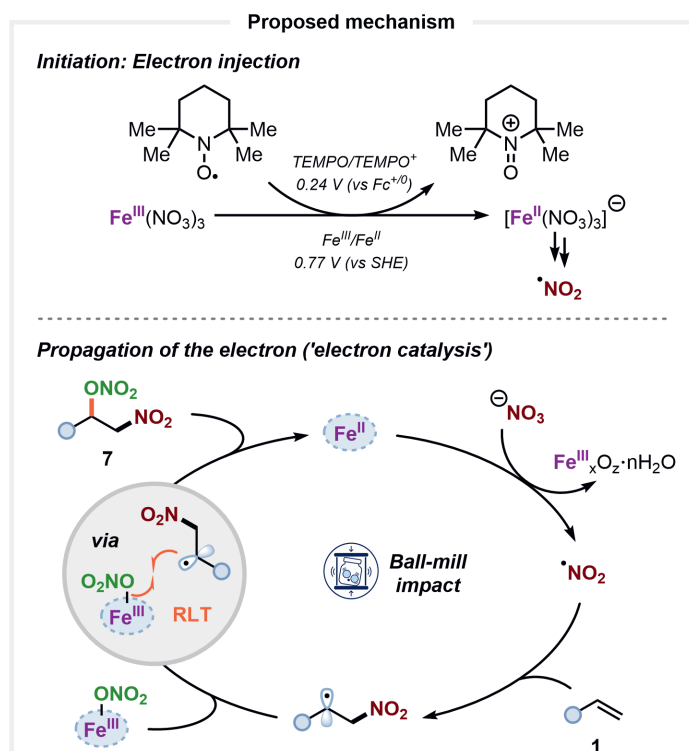


Fig. 8. Proposed mechanism.

sources.^[36] This strategy enables the selective synthesis of dichloro-, dibromo-, and bromochloro-alkanes without the use of hazardous elemental halogens or large quantities of organic solvents.

The mechanistic design relies on the activation of imide-type halogenating reagents such as dibromo-5,5-dimethylhydantoin (DBDMH), dichloro-5,5-dimethylhydantoin (DCDMH), and *N*-bromosuccinimide (NBS) under ball-milling conditions to generate halogen radicals *via* homolytic N–X¹ bond cleavage. Subsequent addition to the alkene produces a transient carbon-centred radical intermediate, which is intercepted by an iron(III) halide species through an RLT event to forge the second C–X² bond. Mechanistic studies involving TEMPO inhibition, radical clock experiments, ring-opening reactions, and comparison with liquid-phase conditions reported in the literature strongly supported the radical nature of the transformation and the involvement of iron-mediated RLT catalysis (Fig. 10).

A broad range of activated and unactivated alkenes, internal alkenes, alkynes, and allenes were efficiently difunctionalized under the optimized conditions (Fig. 11). The methodology displayed excellent functional-group tolerance, accommodating esters, aldehydes, amides, halides, sulfoxides, alcohols, and silanes. The operational simplicity and robustness of the protocols were further demonstrated through late-stage dihalogenation of complex natural products and pharmaceutical derivatives.

Overall, these studies establish mechanochemistry as an effective strategy for radical alkene difunctionalization and demonstrate that RLT can be successfully adapted to solid-state conditions to access structurally diverse molecules in a sustainable manner.

5. Conclusions

RLT has emerged as a powerful and versatile platform for the selective construction of C–heteroatom bonds through the interception of transient carbon-centred radicals by redox-active metal complexes. The presented studies demonstrate that RLT can be successfully merged with electrochemical, photochemical, and mechanochemical activation modes to enable a broad range of alkene difunctionalization and radical functionalization reactions under mild and sustainable conditions.

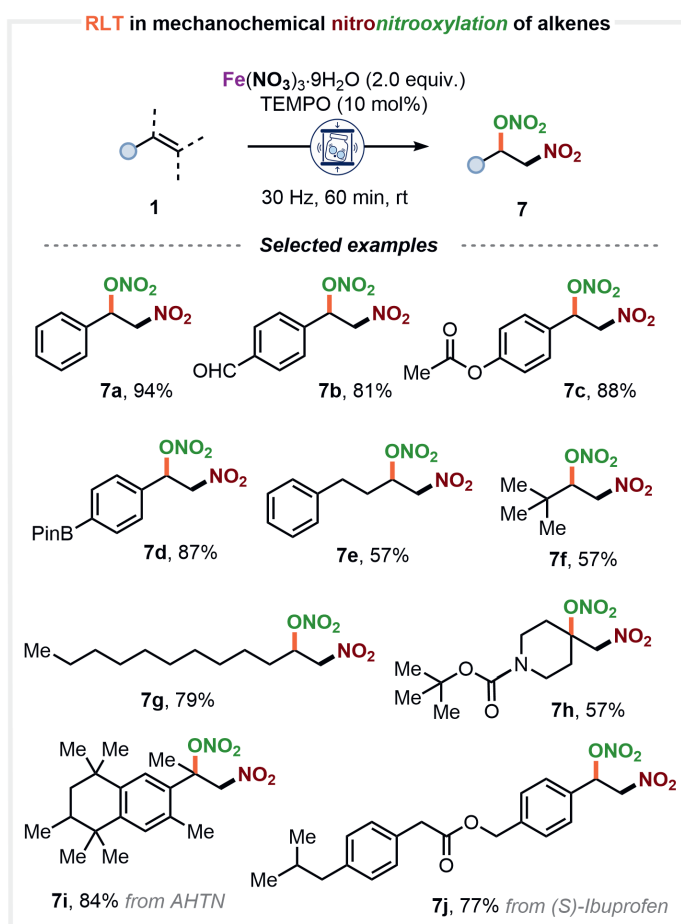


Fig. 9. RLT-mediated mechanochemical nitronitro-xylation of alkenes.

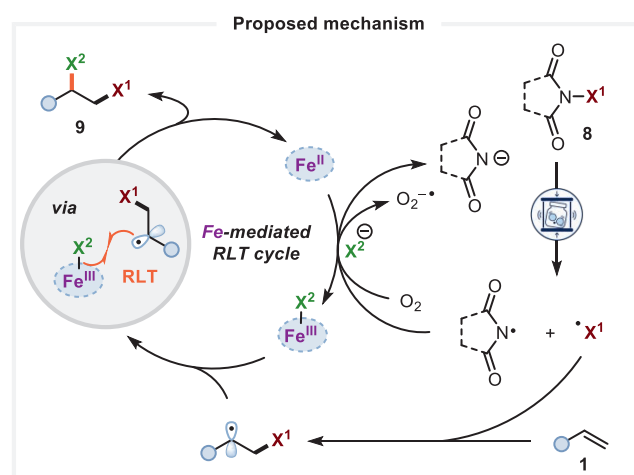


Fig. 10. Proposed mechanism.

Collectively, these studies highlight several key features of RLT catalysis, including broad functional-group tolerance, modularity, compatibility with diverse radical precursors, and the ability to selectively forge C–heteroatom bonds under conditions that differ fundamentally from conventional ionic or atom-transfer pathways. Importantly, the merger of RLT with emerging activation technologies opens new opportunities for the development of sustainable radical transformations and expands the synthetic utility of transition-metal-mediated radical chemistry.

We anticipate that continued advances in mechanistic understanding and activation strategies will further broaden the scope of RLT-mediated transformations and enable the discovery of new catalytic manifolds for selective radical functionalization reactions.

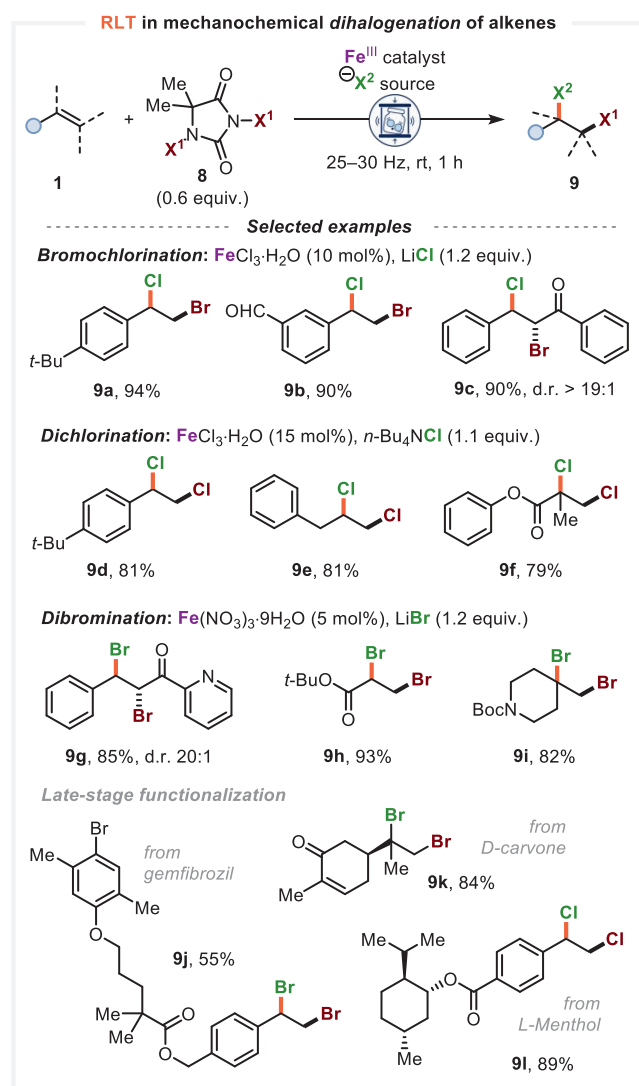


Fig. 11. RLT-mediated mechanochemical dihalogenation of alkenes.

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