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Review

Recent advances in the applications of [1.1.1]propellane in organic synthesis

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ABSTRACT

As a highly strained small molecule, [1.1.1]propellane has been widely used in various synthetic transformations owing to the exceptional reactivity of the central bond between the two bridgehead carbons. Utilizing strain-release approaches, the rapid development of strategies for the construction of bicyclo[1.1.1]pentane (BCP) and cyclobutane derivatives using [1.1.1]propellane as the starting material has been witnessed in the past few years. In this review, we highlight the most recent advances in this field. Accordingly, the reactivity of [1.1.1]propellane can be divided into three pathways, including radical, anionic and transition metal-catalyzed pathways under appropriate conditions.

1. Introduction

Installation of three-dimensional carbon framework into the structure of drug candidates has attracted increasing attention in drug discovery, thus altering the pharmacokinetic properties [1–4]. As the smallest member of propellane family, the highly strained [1.1.1]propellane **1** possess unique reactivity across the central bond. To perfect ring opening processes of [1.1.1]propellane **1**, tremendous efforts have been devoted to the development of efficient synthetic methodologies leading to highly functionalized bicyclo[1.1.1]pentane (BCP) and cyclobutane derivatives [5–8]. Among them, 1,3-disubstituted bicyclo[1.1.1]pentane motif has emerged as bioisosteres for phenyl, *tert*-butyl and alkyne functional groups in modern medicinal chemistry. For example, the replacement of a *para*-substituted fluorophenyl ring in the γ-secretase inhibitor BMS708,163 with a bicyclo[1.1.1]pentane moiety results in improved aqueous solubility and passive permeability, as well as improved oral absorption in a mouse model of γ-secretase inhibition [9]. The bicyclo[1.1.1]pentane analogue of the anti-leukemia drug imatinib was found to exhibit higher themodynamic solubility (Scheme 1) [10].

The first synthesis of [1.1.1]propellane **1** was reported by Wiberg and Walker in 1982 *via* reductive intramolecular coupling of 1,3-dibromobicyclo[1.1.1]pentane [11]. Later in 1985, Szeimies and co-workers developed an improved method for the preparation of [1.1.1]propellane **1** by using the commercially available 1,1-dibromo-2,2-bis(chloromethyl)cyclopropane as the starting material (Scheme 2) [12,13].

In recent years, the synthetic and medicinal community have witnessed fast and impressive development of the [1.1.1]propellane chemistry, and a variety of approaches for the construction of bicyclo[1.1.1]pentane and cyclobutane derivatives have been realized. Although several reviews focusing on the synthesis of bicyclo[1.1.1]pentane derivatives have recently been reported [6,7], the applications of [1.1.1]propellane **1** in organic synthesis still lacks a concise review in this fast growing field. This review herein is aimed to summarize recent progress in the field of synthetic transformations starting from [1.1.1]propellane **1**, highlighting three different reaction pathways for the cleavage of the central bond.

2. Radical addition to the central bond of [1.1.1]propellane

As mentioned above, radical addition to [1.1.1]propellane **1** represents the most frequently observed reaction attributed to its high reactivity, with many different radicals and radical sources being reported including halides, chalcogenides to carbon-centered species. In this transformation, the central bond of

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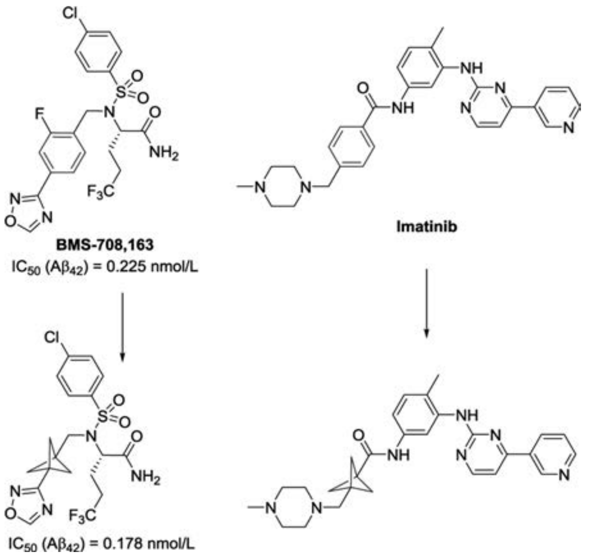
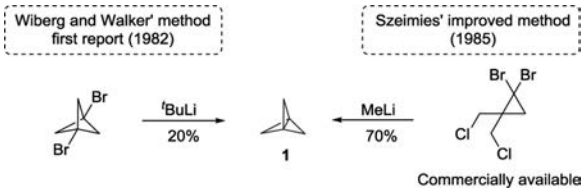
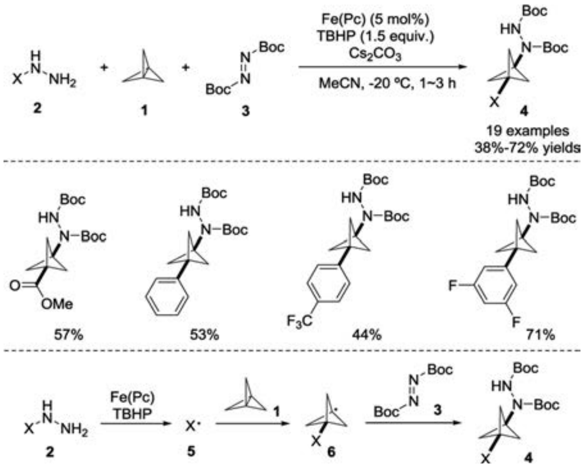


Scheme 1. Bicyclo[1.1.1]pentane as a bioisostere for a phenyl ring in medicinal chemistry.

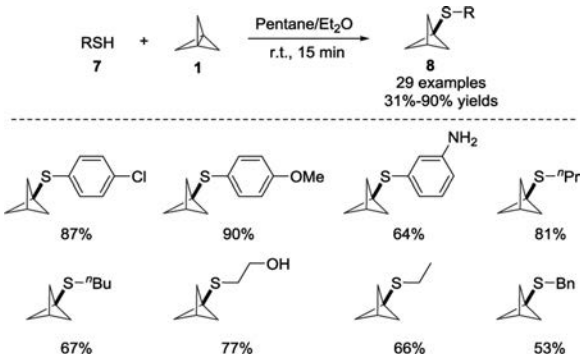


Scheme 2. The reported routes for the synthesis of [1.1.1]propellane.

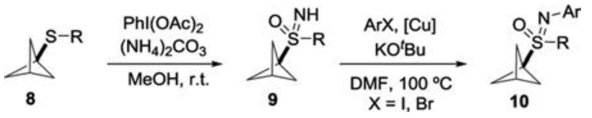
[1.1.1]propellane **1** is broken to yield kinetically stable bicyclo [1.1.1]-pentyl radical, giving rise to various functionalized bicyclo [1.1.1]-pentane derivatives. It is noteworthy that the oligomerization side products could be observed when the low chain-transfer constant of free-radical addition pathway occurred. In 2017, Uchiyama and co-workers developed a radical multicomponent carboamination of [1.1.1]propellane **1** by employing iron(II) phthalocyanine as the catalyst and TBHP as the oxidant (Scheme 3) [14]. Under the mild conditions, hydrazyl reagent **2** was used as a radical precursor with di-*tert*-butyl azodicarboxylate **3** as a radical acceptor for the construction of multi-functionalized bicyclo[1.1.1] pentane derivatives **4**, which could be further transformed into synthetically useful 3-substituted BCP-amines. On the basis of the experimental observations and DFT calculations, hydrazines were shown to efficiently undergo oxidative denitrogenation under iron (II) phthalocyanine and TBHP to generate the free radical **5** *in situ*. Radical addition of **5** with [1.1.1]propellane **1** gave rise to the BCP-radical intermediate **6**, which could be trapped by di-*tert*-butyl azodicarboxylate **3** to yield the desired product **4**.



Scheme 3. Iron(II)-catalyzed multicomponent carboamination of [1.1.1]propellane **1**.



Scheme 4. Addition of thiols with [1.1.1]propellane **1** to yield BCP sulfides.



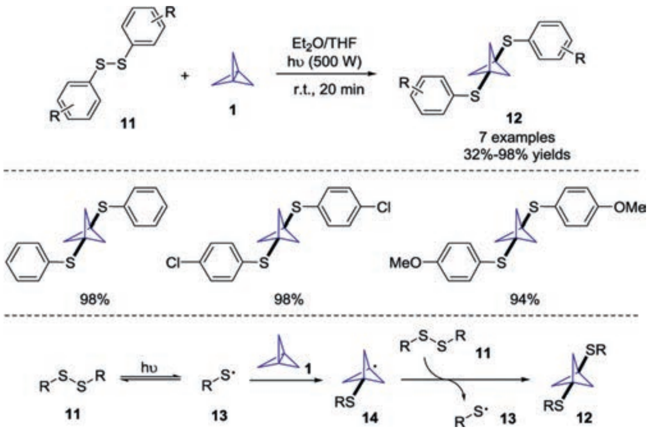
Scheme 5. Formation of BCP sulfoximines applied in copper(I)-catalyzed *N*-arylation.

The reaction of [1.1.1]propellane **1** with thiophenol **7** was first achieved by Szeimies and co-workers in 1985, providing an effective tool for the quantification of propellane solutions [12]. In 2018, the group of Bräse further investigated the reaction in details and enlarged the scope of thiols **7**, which showed that halo-, hydroxy-, methoxy-, carboxy-, amino- and nitro-substituted thiols were well tolerated (Scheme 4) [15]. The reaction proceeded at room temperature without any catalyst, and no by-products were detected. The deuterium labelling experiments revealed that the reaction underwent a radical chain process.

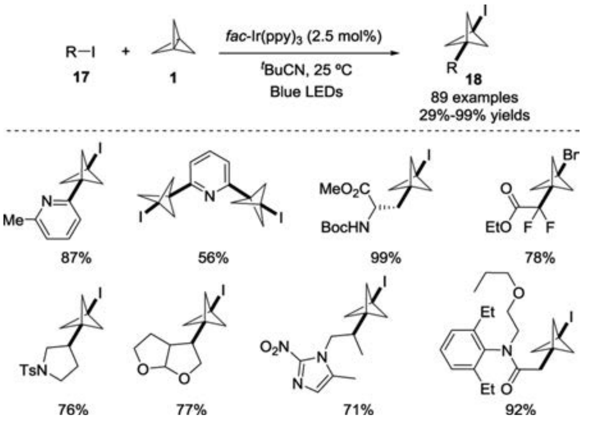
Recently, the synthetic utility of this method was further demonstrated through the conversion of BCP sulfides **8** into the corresponding sulfoximines **9** (Scheme 5) [16]. Moreover, copper(I)-catalyzed *N*-arylations of BCP sulfoximines **9** with aryl halides were carried out under optimized conditions, showing a wide substrate scope.

Additionally, the same group reported the UV-initiated radical reaction for the synthesis of symmetrical and unsymmetrical 1,3-bissulfanylbicyclo[1.1.1]pentanes from disulfides **11** and [1.1.1]propellane **1** (Scheme 6) [17]. By changing the ratio of [1.1.1]propellane to disulfide, the reaction could provide only BCP product or a mixture of BCPs and [2]staffanes. However, the separation of these products was challenging due to the similar polarity. The authors also proposed a possible reaction mechanism to explain the formation of different products.

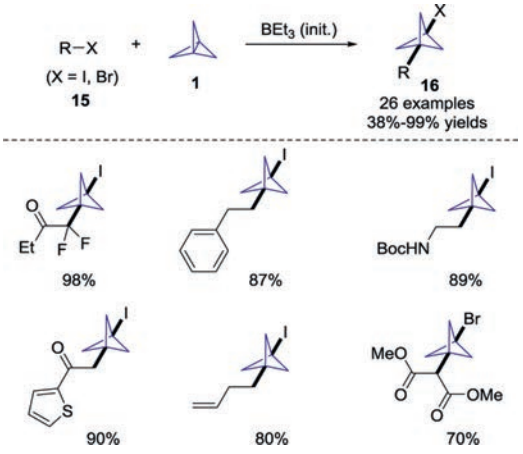
In 2018, Anderson and co-workers described an efficient triethylborane-initiated atom-transfer radical addition of [1.1.1]propellane **1** with alkyl halides **15** to access 1-halo-3-substituted bicyclo[1.1.1]pentanes **16** (Scheme 7) [18]. This protocol exhibited a broad substrate scope and good functional



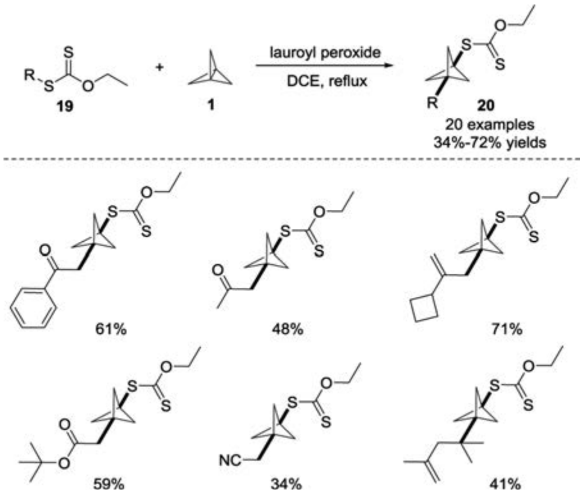
Scheme 6. UV-promoted radical reaction of disulfides and [1.1.1]propellane **1**.



Scheme 8. Photoredox-catalyzed radical reaction of organic halides with [1.1.1]propellane **1**.



Scheme 7. Synthesis of 1-halo-3-substituted bicyclo[1.1.1]pentanes through triethylborane-initiated radical addition pathway.



Scheme 9. Metal-free synthesis of BCP xanthate derivatives in the presence of dilauroyl peroxide.

group tolerance. Furthermore, the synthetic applicability of this process was also demonstrated through late-functionalization of complex molecules such as nucleosides, peptides and drug-like molecules.

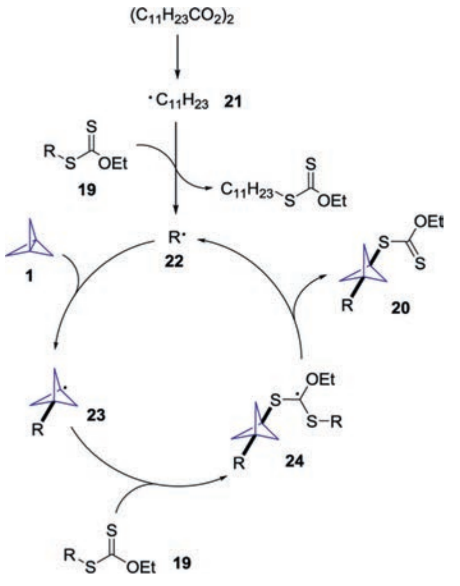
Subsequently, the same group further disclosed a visible light promoted radical addition of organic halides **17** with [1.1.1]propellane **1** for the functionalization of carbon-carbon σ-bonds (Scheme 8) [19]. Under mild conditions, both (hetero)aryl radicals and nonstabilized alkyl radicals were generated *in situ* by using *fac*-Ir(ppy)$_3$ as the photocatalyst, affording a variety of (hetero) arylated and polycyclic BCP products **18** with a broad substrate scope. Additionally, the importance of the photocatalyst in this process was demonstrated according to the mechanistic investigations.

In 2019, Riant and co-workers reported a radical exchange strategy by incorporating xanthate derivatives **19** into [1.1.1]propellane **1** under metal-free conditions (Scheme 9) [20]. By employing substoichiometric amounts of dilauroyl peroxide (DLP) as a radical initiator, this method enabled the synthesis of functionalized BCP derivatives with xanthate moieties. The reaction featured a broad substrate scope and functional group tolerance respect to the xanthate moiety.

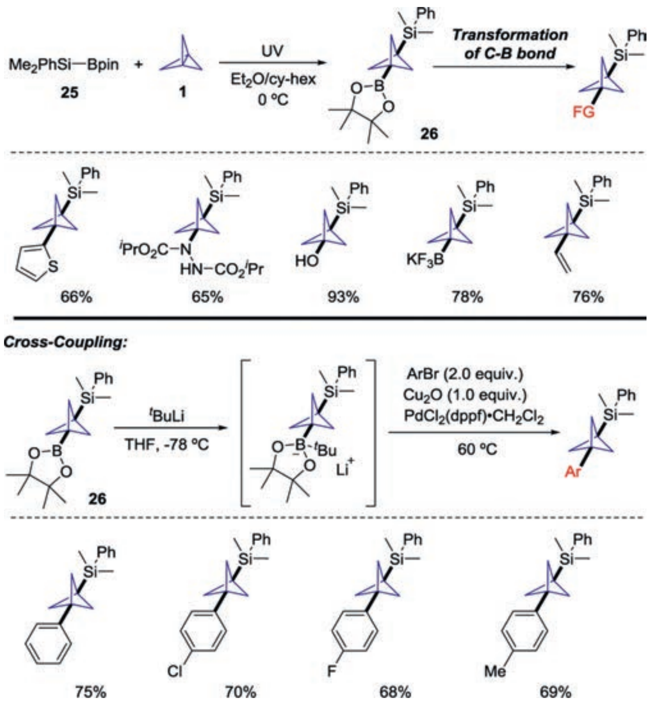
On the basis of mechanistic experiments, the authors proposed a plausible mechanism for this transformation (Scheme 10). Initially, DLP would undergo fragmentation/decarboxylation to provide the decyl radical **21**, which would react with xanthate **19** to generate radical **22** *in situ*. The addition of radical **22** with [1.1.1]propellane **1** would give rise to the radical intermediate **23**. Subsequently, the reaction of radical **23** with xanthate **19** would produce the stabilized intermediate radical **24**, followed by radical cleavage to produce the BCP product **20**.

The UV-initiated silaboration of [1.1.1]propellane **1** to access BCP derivatives bearing B and Si functional groups was achieved by Uchiyama and co-workers (Scheme 11) [21]. Under mild and additive-free conditions, the unsymmetrically silaborated products **26** could be obtained on a gram-scale without the need for column-chromatographic purification. On the basis of experimental studies and DFT calculations, it was suggested that a radical chain mechanism was involved in this transformation. Notably, the synthetic applicability of the process was also demonstrated through the conversion of the C–B/C–Si bonds on the BCP scaffold into various functional groups. In particular, by combination of highly activated BCP boronic esters, Cu$_2$O, and PdCl$_2$(dppf) catalyst, the authors developed a facile approach for Suzuki–Miyaura cross-coupling reaction at the highly sterically hindered BCP bridgehead sp^3 carbon center.

Bicyclo[1.1.1]pentylamine has been considered as a high-value bioisostere for aniline to improve antimetabolic clearance activity

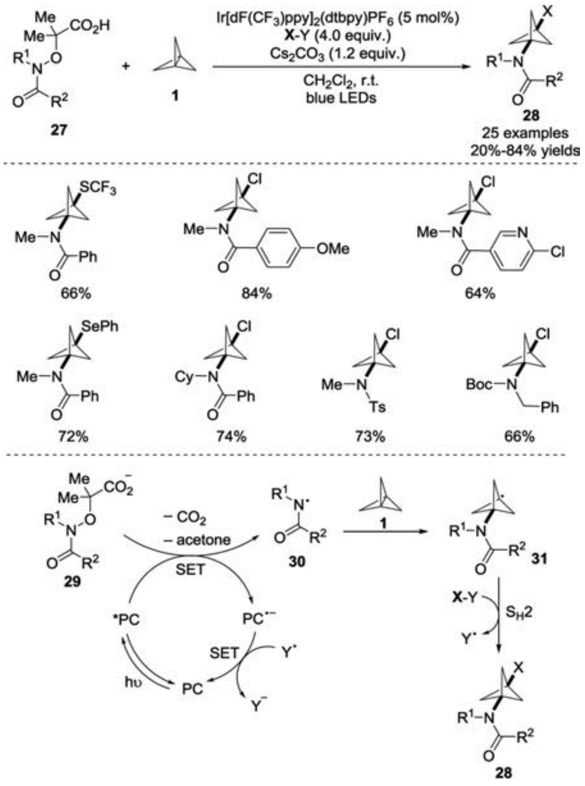


Scheme 10. Proposed mechanism for the formation of BCP xanthate derivatives.



Scheme 11. UV-initiated silaboration of [1.1.1]propellane **1** and further applications.

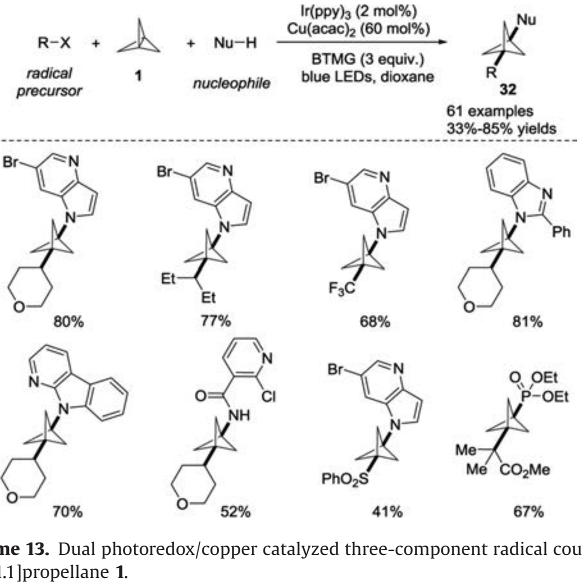
in modern medicinal chemistry. Recently, Leonori and co-workers reported the first example of visible-light-promoted radical strain-release-functionalization strategyfor the divergent construction of 1,3-disubstituted bicyclo[1.1.1]pentylamines **28** (Scheme 12) [22]. The reaction tolerated a variety of *N*-substituted amides **27**, which served as precursors for generating amidyl radicals, representing an umpolung approach to ionic strain-release amination.



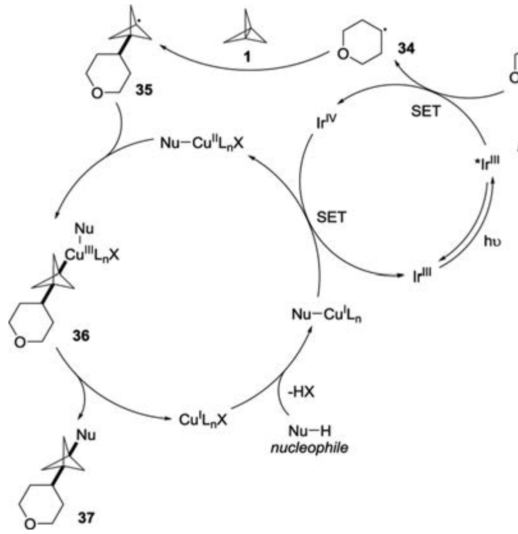
Scheme 12. Visible-light-mediated strain-release multicomponent reaction of [1.1.1]propellane **1** with electrophilic nitrogen-radicals.

Accordingly, the putative mechanism revealed that the visible-light excited photocatalyst was quenched by radical precursor **29** *via* a single electron transfer (SET) to provide the amidyl radical **30**. The addition of amidyl radical **30** to [1.1.1]propellane **1** would produce stabilized radical intermediate **31**, followed by atom/group-transfer reaction (S_H2) with SOMOphiles (X–Y) to deliver the desired product **28**. Finally, the electron poor radical Y· would undergo reduction by the reduced state of photocatalyst to complete the catalytic cycle.

Most recently, the MacMillan group developed a three-component radical coupling of [1.1.1]propellane **1** in the presence of dual photoredox/copper catalysis (Scheme 13) [23]. This elegant



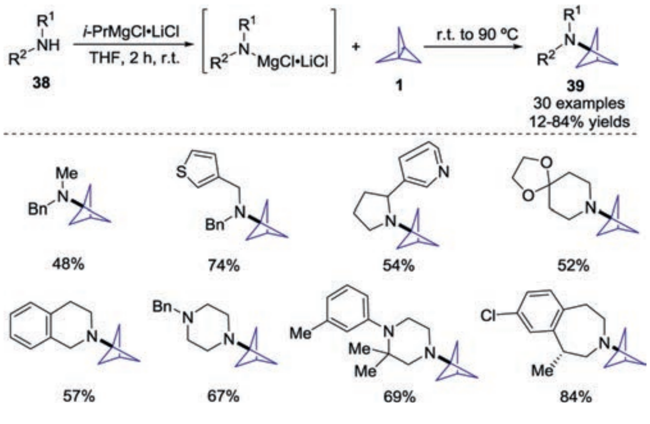
Scheme 13. Dual photoredox/copper catalyzed three-component radical coupling of [1.1.1]propellane **1**.



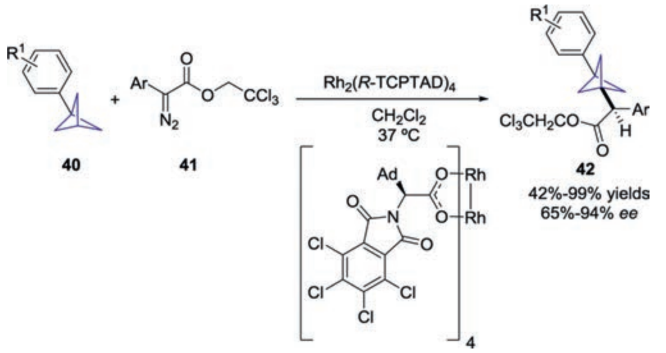
Scheme 14. Proposed mechanism for the synthesis of polysubstituted BCP products.

protocol enabled the one-step synthesis of diverse functionalized BCP derivatives **32** within short timescales (5 min to 1 h). Remarkably, the reaction featured a broad substrate scope and good functional group tolerance, and a variety of radical precursors including those which generated alkyl, α-acyl, trifloromethyl, and sulfonyl radicals as well as multiple classes of *N*-, *P*-, and *S*-nucleophiles were proven to amenable for this transformation. Moreover, this metallaphotoredox catalysis strategy was successfully applied in the late-stage functionalization of drugs and natural products and preparation of pharmaceutical analogues.

The mechanism for this multicomponent transformation was proposed as follows: firstly, the alkyl radical **34** was generated through the SET reduction of iodoniumdicarboxylate **33** (E_{pc} [**33**/**33**$^{\bullet-}$] = -0.82 V *vs.* SCE in acetonitrile) by excited *Ir$^{\mathrm{III}}$ ($E_{1/2}{}^{\mathrm{red}}$ [Ir$^{\mathrm{IV}}$/*Ir$^{\mathrm{III}}$] = -1.81 V *vs.* SCE in acetonitrile). Subsequently, the addition of alkyl radical **34** to [1.1.1]propellane **1** would afford stabilized radical intermediate **35**. The combination of radical intermediate **35** with nucleophile-ligated copper complex would lead to Cu$^{\mathrm{III}}$ species **36**, which would go through reductive elimination to afford the desired product **37**. Finally, the nucleophile-ligated Cu$^{\mathrm{I}}$ complex was oxidized by Ir$^{\mathrm{IV}}$ ($E_{1/2}{}^{\mathrm{red}}$ [Ir$^{\mathrm{IV}}$/Ir$^{\mathrm{III}}$] = +0.77 V *vs.* SCE in acetonitrile) to provide Cu$^{\mathrm{II}}$ complex with the release of Ir$^{\mathrm{III}}$ species to facilitate both catalytic cycles (Scheme 14).



Scheme 15. Turbo-amide enabled ring opening of [1.1.1]propellane **1** to access mono-substituted bicyclo[1.1.1]pentylamines.



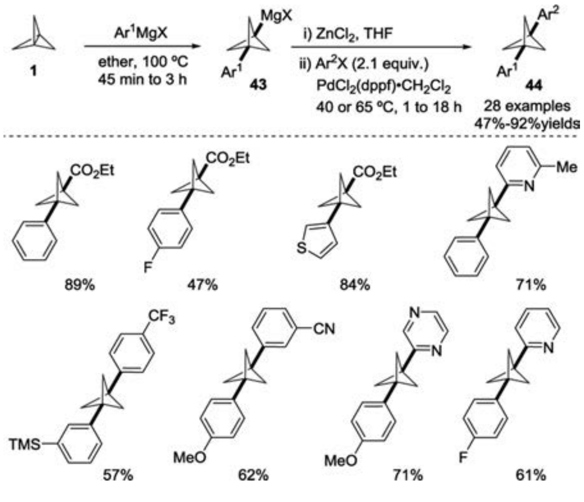
Scheme 16. Chiral dirhodium complex-catalyzed asymmetric C–H functionalization of BCPs towards chiral substituted BCPs.

3. Nucleophilic addition to the central bond of [1.1.1]propellane

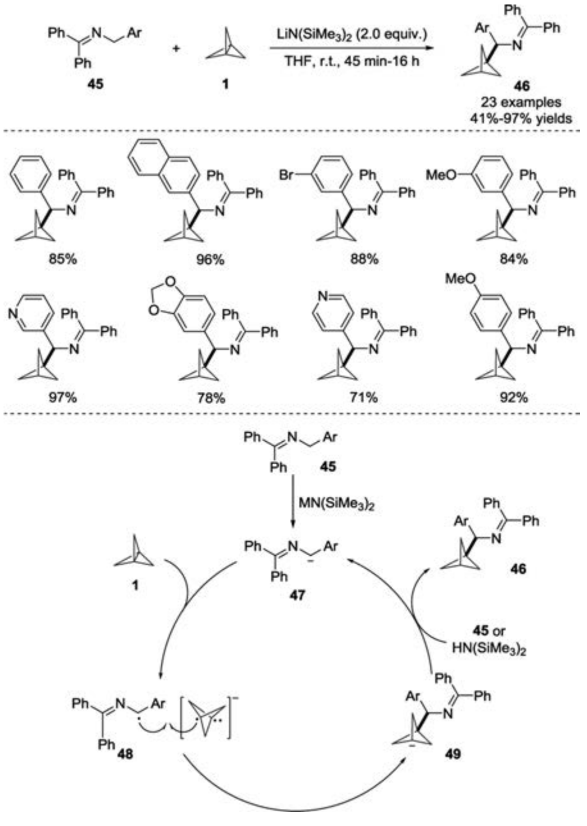
Considering the importance of BCP functionality, nucleophilic addition to the central bond of [1.1.1]propellane **1** has become an alternative and complementary method for that of radical additions. The breakthrough was made by Baran and co-workers in 2016, and they found that [1.1.1]propellane **1** would undergo ionic strain-release amination by deprotonated dialkyl amines, solving a long standing issue for the synthesis of monosubstituted bicyclo[1.1.1]pentylamines **39** (Scheme 15) [24]. It was found that the use of turbo-amides was the key to complete the transformation.

The aryl-substituted BCPs **40** could be prepared according to Baran's method using aryl Grignard reagent instead of turbo-amides. Recently, the synthetic utility of aryl-substituted BCPs **40** was demonstrated by Davies and co-workers, through enantioselective intermolecular C(sp^3)–H insertion reactions of donor/acceptor diazo compounds **41** (Scheme 16) [25]. By employing the chiral dirhodium complex Rh$_2$(TCPTAD)$_4$ as the catalyst, a variety of 1,3-disubstituted BCPs **42** bearing a proximal stereocenter were obtained with up to 99% yield and 94% *ee*.

In 2017, Knochel and co-workers developed an elegant and practical method for the synthesis of diarylated BCP derivatives **44** through Grignard reagent-mediated ring-opening of [1.1.1]propellane **1** and Negishi coupling after transmetallation with ZnCl$_2$ (Scheme 17) [26]. The bis-arylated BCPs could be regarded as potential bioisosteres of internal alkynes. For instance, BCP analogues of tazarotene and the metabotropic glutamate receptor



Scheme 17. Synthesis of diarylated BCP derivatives.



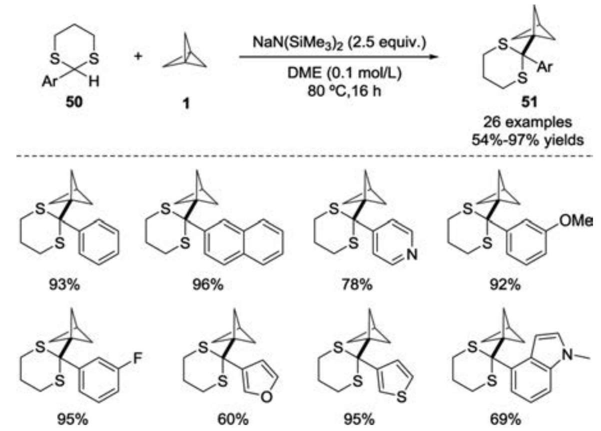
Scheme 18. Additions of 2-azaallyl anions with [1.1.1]propellane **1** to synthesize BCP benzylamines.

5 (mGluR5) antagonist were prepared and their physicochemical properties were evaluated.

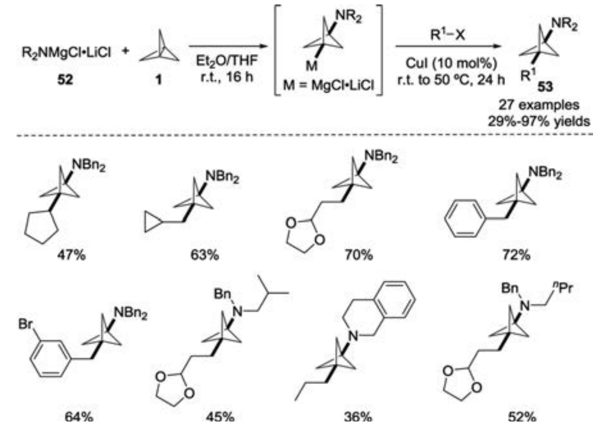
In 2018, the Walsh group developed a new method for the synthesis of BCP benzylamine derivatives **46** through a reaction of [1.1.1]propellane **1** with 2-azaallyl anions generated *in situ* from *N*-benzyl ketimines **45** (Scheme 18) [27]. The reaction proceeded rapidly with high yields and tolerated a broad substrate scope at room temperature. On the basis of their previous work, the authorsproposed several possible mechanisms for this transformation. In one possible pathway, 2-azaallyl anion **47** generated from deprotonation of *N*-benzyl ketimine **45** would undergo a single electron transfer (SET) with [1.1.1]propellane to form azaallyl radical **48** and [1.1.1]propellane radical anion. Subsequently, radical-radical coupling of both species would produce the anion intermediate **49**, followed by protonation at the bridgehead carbon to furnish the desired product **46** and regenerate 2-azaallyl anion **47**.

Later, the same group extended this strategy to the preparation of BCP-containing dithianes **51** (Scheme 19) [28]. The reaction of 2-aryl-1,3-dithianes **50** and [1.1.1]propellane **1** featured a broad substrate scope and was amenable to scale up. The utility of the products was further demonstrated by the conversion of the dithiane moieties into a variety of functional groups. Additionally, on the basis of computational results, it was proposed that a two-electron pathway might be involved in this transformation.

In 2019, Gleason and co-workers disclosed a one-pot aminoalkylation of [1.1.1]propellane **1** toward the synthesis of 3-alkylbicyclo[1.1.1]pentan-1-amines **53** under mild reaction conditions (Scheme 20) [29]. This protocol exhibited a broad substrate scope, providing a facile approach for accessing 1,3-difunctionalized BCPs containing high-value functional groups.



Scheme 19. Reactions of 2-aryl-1,3-dithianes and [1.1.1]propellane **1** to synthesize BCP dithianes.

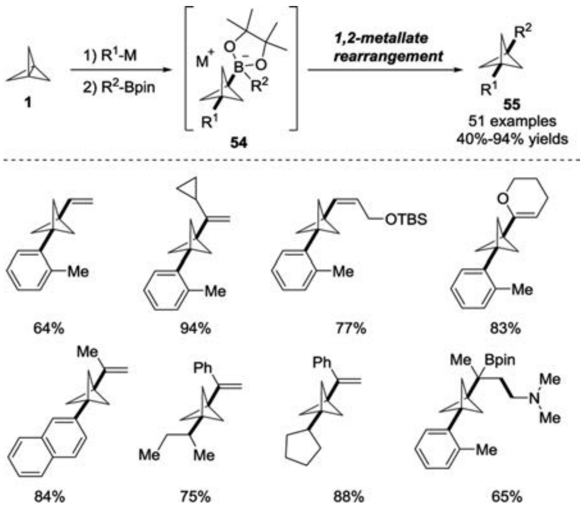


Scheme 20. Aminoalkylation of [1.1.1]propellane **1** for the synthesis of 3-alkylbicyclo[1.1.1]pentan-1-amines.

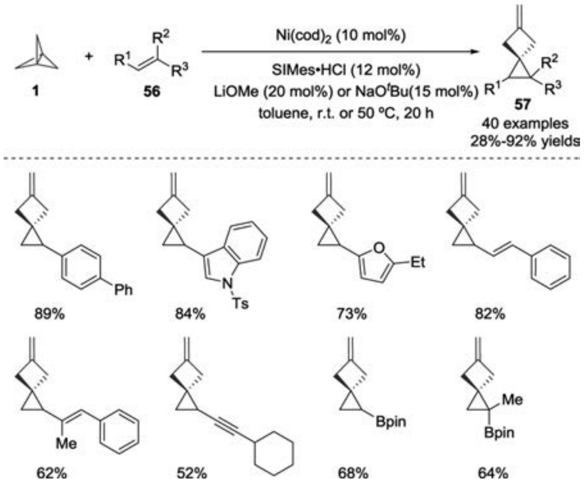
Earlier this year, Aggarwal and co-workers reported a transition metal-free three-component of [1.1.1]propellane **1** with organometallic reagents and organoboronic esters for the rapid synthesis of 1,3-disubstituted BCP derivatives **55** (Scheme 21) [30]. During the reaction process, boronate complexes **54** were generated *in situ* from the BCP-metal species and boronic esters, which would undergo electrophile-induced 1,2-metallate rearrangement for the construction of C—C bonds. Furthermore, this strategy could be extended to access tertiary boronic ester-substituted BCPs containing contiguous quaternary centers *via* a four-component coupling of alkenyl BCP-boronate complexes with a variety of electrophiles.

4. Transition metal-catalyzed transformations of [1.1.1]propellane

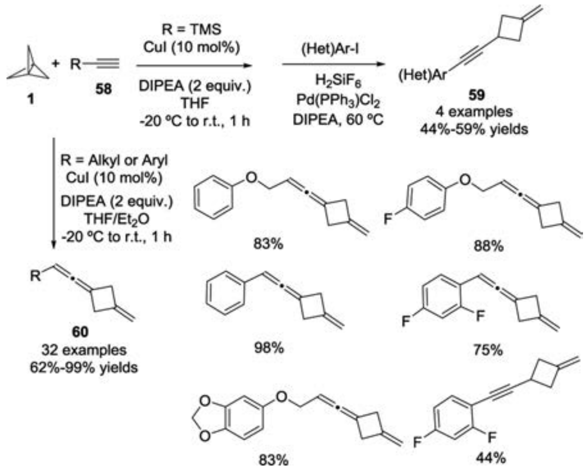
Contrary to the well-developed methods for the preparation of functionalized BCP derivatives through radical or carbanion pathways, transition metal-catalyzed transformation of [1.1.1]propellane are less established. In 2019, Aggarwal, Bedford and co-workers described a nickel-catalyzed cyclopropanation of



Scheme 21. Synthesis of 1,3-disubstituted BCPs through 1,2-metallate rearrangements of boronate complexes.



Scheme 22. Nickel-catalyzed cyclopropanation of alkenes with [1.1.1]propellane **1**.



Scheme 23. Copper-catalyzed divergent synthesis of cyclobutane-containing alkynes and allenes.

alkenes **56** with [1.1.1]propellane **1** as a carbene precursor (Scheme 22) [31]. The reaction exhibited a broad functional group tolerance under mild conditions, leading to the formation of methylenespiro[2.3]hexane derivatives **57**. Both experimental and computational results supported the formation of a nickel carbene through a concerted double C—C bond cleavage of a nickel-[1.1.1] propellane complex.

Almost the same time, a copper-catalyzed formation of cyclobutane-containing alkynes **59** and allenes **60** from the ring opening reactions of [1.1.1]propellane **1** with alkynes **58** was disclosed by Tolnai and co-workers (Scheme 23) [32].

5. Conclusion

In summary, this review highlights the most recent advances of the synthetic applications of [1.1.1]propellane. Overall, a number of different approaches for the synthesis of BCP and cyclobutane derivatives from [1.1.1]propellane have been conceived and developed. Generally, the recent progress of this chemistry shows that the weak central bond of [1.1.1]propellane can be broken either through a radical, anionic or transition metal-catalyzed pathway. For example, radical addition to [1.1.1]propellane produces stabilized BCP-radical intermediate, which can be subsequently trapped by a radical acceptor or transition metal catalyst. Because of its inherent structural features and unique reactivity profile, [1.1.1]propellane possesses a very important role in both synthetic and medicinal community for incorporating a three-dimensional cyclic scaffold into small molecules as bioisosteres.

Despite the recent achievements in this field, there are still many issues existed, which are anticipated to be solved for the chemistry of [1.1.1]propellane. For instance, the synthesis of optically pure compounds bearing a BCP moiety through one-pot multicomponent catalytic asymmetric transformation of [1.1.1]propellane has yet not to be achieved. Furthermore, the development of sustainable processes based on flow chemistry or electrochemistry as well as the discovery of new reactivities with [1.1.1]propellane can be expected in the near future.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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