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# Mechanochemical base-catalyzed isomerization and deuteration of allylbenzenes

Ruoxuan Liu,<sup>†</sup> Xiaochun He,<sup>†</sup> Ruiling Qu, Chengcheng Li, Xiaohong Wang,  
Xuemei Zhang\* and Zhong Lian<sup>†</sup>\*

Trideuteromethylation has gained significant importance in modern drug development for its ability to fine-tune pharmacokinetic properties and enhance metabolic stability through the deuterium kinetic isotope effect. Currently, most methods for alkene double-bond migration (e.g., transition-metal catalysis) suffer from drawbacks such as substantial waste generation, high reaction temperatures, and extended reaction durations. Herein, we report a universal mechanochemical strategy for the rapid and metal-free isomerization and deuteration of allylbenzenes. Utilizing only catalytic KOtBu and stoichiometric DMSO-*d*<sub>6</sub>, this method enables precise CD<sub>3</sub> incorporation within 15 minutes under ambient conditions. The protocol exhibits broad functional group tolerance, is scalable, and applicable to late-stage deuteration of natural products, offering a practical and sustainable approach to deuterated pharmaceuticals. In addition, mechanistic studies indicate that the H/D exchange catalyzed by KOtBu is a radical process.

engagement, and materials science underscores the demand for cost-effective, operationally simple, and highly selective methods to incorporate the CD<sub>3</sub> group.<sup>6</sup>

In recent years, isomerization of alkene has emerged as a powerful strategy to access valuable internal olefins, which serve as key intermediates in pharmaceuticals, fragrances, food, and synthetic precursors.<sup>7</sup> Numerous catalytic systems have been developed to achieve selective C=C bond migration, including noble metal complexes (e.g., Ru, Rh, Pd, Ir)<sup>8–11</sup> and earth-abundant metal catalysts (e.g., Co, Ni, Mn, Fe, Mo, Cr)<sup>12–17</sup> (Fig. 1B(i)). Despite their utility, these methods typically generate metal/ligand waste, require stoichiometric reductants or additives, and increase process costs. Lewis acidic boranes such as B(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub> or HB(C<sub>6</sub>F<sub>5</sub>)<sub>2</sub> have also been explored, but suffer from harsh conditions including high temperatures, long reaction times, and poor functional group tolerance<sup>18,19</sup> (Fig. 1B(ii)). Alkali metal alkoxides and ammonium bases can facilitate isomerization; however, a stoichiometric amount of these substances is required for the process.<sup>7,20</sup> Although NaTMP (TMP = 2,2,6,6-tetramethylpiperidide) exhibits catalytic activity, it requires the tridentate Lewis donor PMDETA for effectiveness, restricting its applicability.<sup>21</sup> The base-catalyzed remote hydrogermylation reported by Schoenebeck and coworkers represents a notable advance, yet depends on germylation to drive isomerization and operates under elevated temperatures with extended reaction times<sup>22</sup> (Fig. 1B(iii)). Additionally, photoredox/transition-metal dual catalysis, as well as electrocatalytic strategies, have facilitated alkene migration under mild conditions, but remain hampered by prolonged reaction durations (16–20 h)<sup>23,24</sup> (Fig. 1B(iv)). Despite these advances, persistent challenges include reliance on transition metals, high temperatures, long reaction times, and the use of homemade reagents.

Mechanochemistry has recently evolved into an efficient and sustainable strategy for organic synthesis, characterized by solvent-free nature, short reaction times, operational simplicity, and compatibility with ambient conditions.<sup>25–55</sup> Inspired by these advantages, we developed a mechanochemical strategy for base-catalyzed isomerization and deuteration of allylbenzenes

## Introduction

The introduction of a methyl group is known to enhance the binding affinity, bioavailability, and metabolic stability of drug molecules, a phenomenon commonly referred to as the “magical methyl effect”.<sup>1</sup> Analogously, the C–D bond exhibits greater stability than the C–H bond due to its shorter length (0.005 Å) and higher dissociation energy (by 1.2–1.5 kcal mol<sup>−1</sup>),<sup>2</sup> making deuterium incorporation a valuable tool for tuning the absorption, distribution, metabolism, and excretion (ADME) properties of drug candidates.<sup>3</sup> Since the approval of the first deuterated drug, deutetrabenazine (Austedo), by FDA in 2017,<sup>4</sup> the trideuteromethyl (CD<sub>3</sub>) group has garnered increasing attention in drug development (Fig. 1A).<sup>5</sup> The broad utility of trideuteromethylation in therapeutics, metabolic tracing, pharmacokinetics, target

Department of Dermatology, State Key Laboratory of Biotherapy and Cancer Center, West China Hospital, Sichuan University, Chengdu 610041, China. E-mail: xuemeizhang@scu.edu.cn; lianzhong@scu.edu.cn

<sup>†</sup> These authors contributed equally.



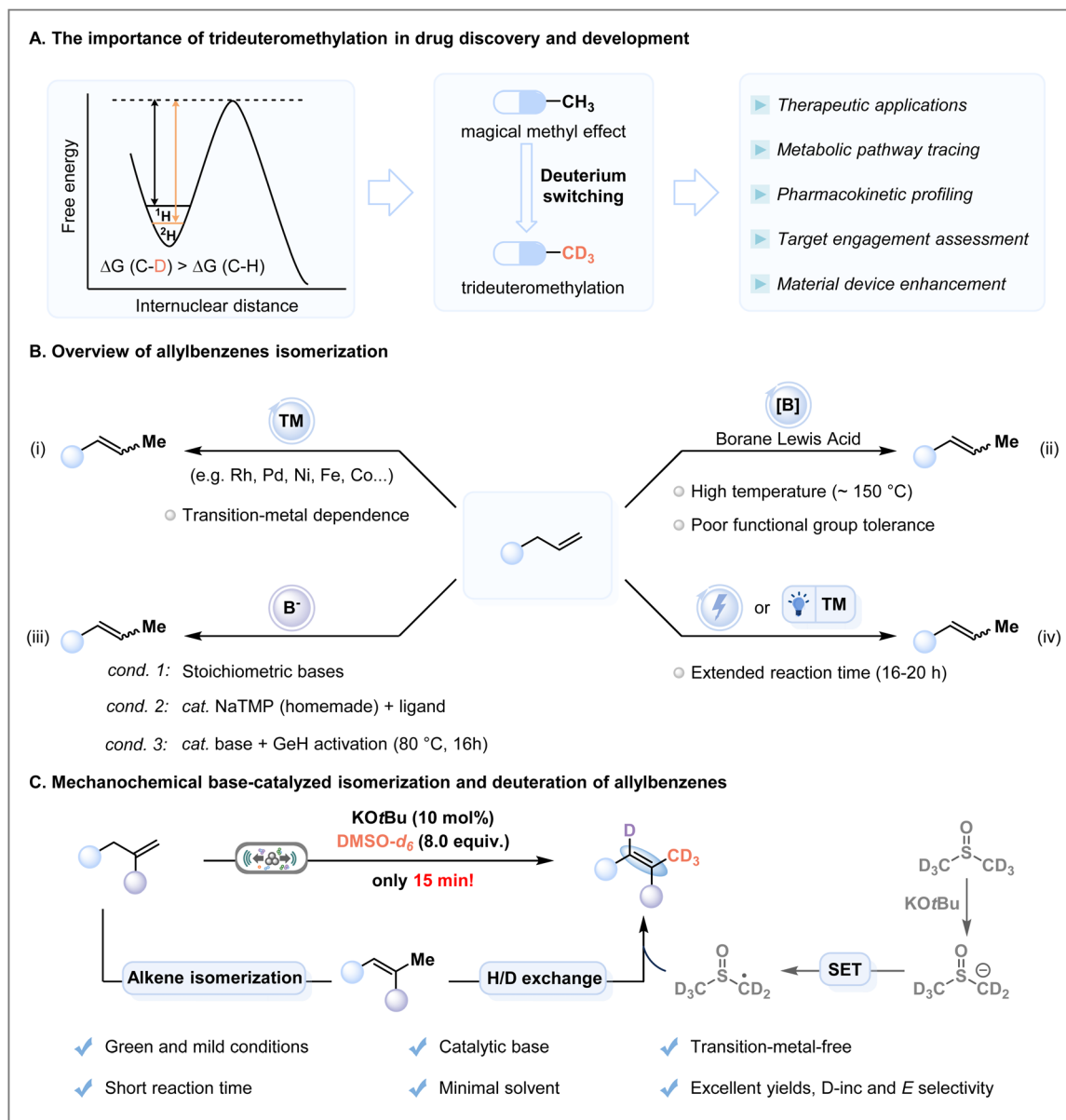


Fig. 1 (A) The importance of trideuteromethylation in drug discovery and development. (B) Overview of allylbenzenes isomerization. (C) Mechanochemical base-catalyzed isomerization and deuteration of allylbenzenes.

to access vinyl-trideuteromethylated derivatives (Fig. 1C). This approach employs only a catalytic amount of base (KOtBu) and stoichiometric DMSO-*d*<sub>6</sub>, enabling precise and rapid incorporation of a CD<sub>3</sub> group within only 15 minutes. Notably, the reaction proceeds under mild conditions with significantly reduced solvent usage and shorter reaction times compared to conventional methods. Mechanistic studies indicate that the H/D exchange catalyzed by KOtBu is a radical process.

## Results and discussion

To showcase the practicality of this strategy, 4-allylanisole (**1a**), DMSO-*d*<sub>6</sub> (8.0 equivalents), and KOtBu (10 mol%) were employed as model substrates to optimize the

mechanochemical alkene double-bond migration deuteration under ball-milling conditions at a frequency of 30 Hz (Fig. 2). A screening of various bases revealed that KOtBu exhibited superior efficiency compared to the alternatives and could be used in a catalytic amount (Fig. 2A). Deuterium source evaluation showed that neither D<sub>2</sub>O nor CD<sub>3</sub>OD afforded the desired product, whereas CD<sub>3</sub>CN resulted in moderate yield with low deuterium incorporation (Fig. 2B). Notably, the reaction reached completion within merely 15 minutes, affording the target product **2a** in 99% isolated yield and excellent *E*-selectivity (>99:1). Under identical conditions, a control experiment was conducted using DMSO in place of DMSO-*d*<sub>6</sub>, affording the non-deuterated product **2a**. The *E*-configuration of **2a** was confirmed by the coupling constant ( $J = 16.0$  Hz) of the olefinic



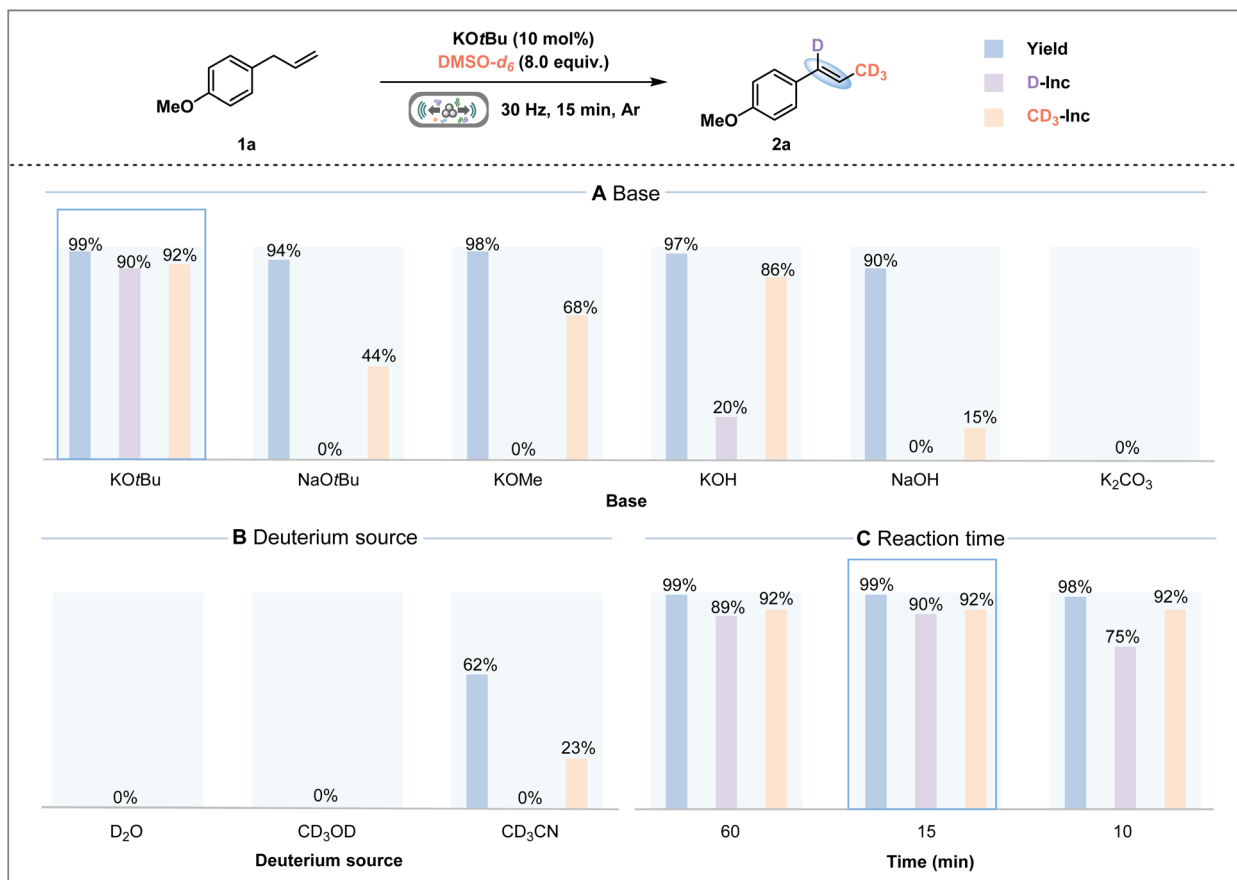


Fig. 2 Screening of reaction conditions. 4-allylanisole (**1a**, 0.4 mmol), KOtBu (10 mol%), DMSO-*d*<sub>6</sub> (8.0 equiv.) in stainless-steel milling jar (5 mL) using two stainless-steel balls (diameter: 7 mm), ball milling under Ar for 15 min at 30 Hz. The yield was determined by GC using decane as an internal standard. Deuterium incorporation was determined by <sup>1</sup>H NMR spectrum. (A) Base. (B) Deuterium source. (C) Reaction time.

proton in its <sup>1</sup>H NMR spectrum. This result further demonstrates that the deuterated product obtained in the original reaction is likewise the *E*-isomer. Impressively, the deuterium incorporation reached 90% for the C(sp<sup>2</sup>)-D moiety and 92% for the C(sp<sup>3</sup>)-D moiety (Fig. 2C).

After establishing the optimized conditions for mechanochemical alkene double-bond migration deuteration, we proceeded to investigate the substrate scope of the reaction, with the results presented in Fig. 3. We first examined a range of functionalized terminal (monosubstituted and 1,1-disubstituted) alkenes under mechanochemical conditions. Allylbenzenes substrates containing various functional groups (*e.g.*, -Me, -*t*Bu, -OMe, -OPh, -OCH<sub>2</sub>Ph, -NPh<sub>2</sub>, -Ph) at different positions on the benzene ring were found to be compatible with the reaction, furnishing 1,2-disubstituted olefins **2b–l** in 61–99% yields with excellent deuterium incorporation (>90% D) at the terminal CD<sub>3</sub> group and outstanding *E*-selectivity. Notably, deuterium labeling was also observed at the benzylic position. Furthermore, substrates containing methyl (**2b–c**) or benzyloxy (**2i**) groups underwent additional deuteration on the methyl and benzyl sites, respectively. Allylic substrates bearing halogens (-F, -I) also underwent efficient migratory deuteration to afford the desired products **2m** and **2n**. These groups are of high value for further modification and build-up of molecular

complexity.<sup>56,57</sup> Substrates incorporating polycyclic aromatic rings, including naphthalene, anthracene were smoothly transformed into the corresponding products (**2o–p**) in excellent deuterium incorporation. The yield of product **2o** was relatively low, primarily due to the formation of a significant amount of dimer byproduct.<sup>43</sup> Oxygen- and nitrogen-containing heteroaromatic compounds were also tolerated in this reaction, giving the corresponding double bond migration deuterated products (**2q–s**). Terminal CD<sub>3</sub>-containing vinyl sulfides were obtained with excellent deuterium incorporation (**2t–u**). Notably, allyltriphenylsilane and allyldiphenylphosphine were also isomerized to yield the corresponding 2-alkenes (**2v–w**). In addition, 1,1-disubstituted allylic analogues were well tolerated, delivering the corresponding products (**2x–3a**) in high yields with excellent deuterium incorporation. The protocol was also applicable to a trisubstituted allylic substrate (**3b**), further underscoring its broad scope and robust reliability. Under mechanochemical conditions, a range of naturally occurring compounds, such as anethole, eugenol, a polysphorine precursor, nothosmyrnol, and safrole,<sup>13</sup> were efficiently functionalized with a CD<sub>3</sub> group, yielding products **2a** and **3c–f** with excellent conversion, high deuterium incorporation at the allylic position, and good stereoselectivity.



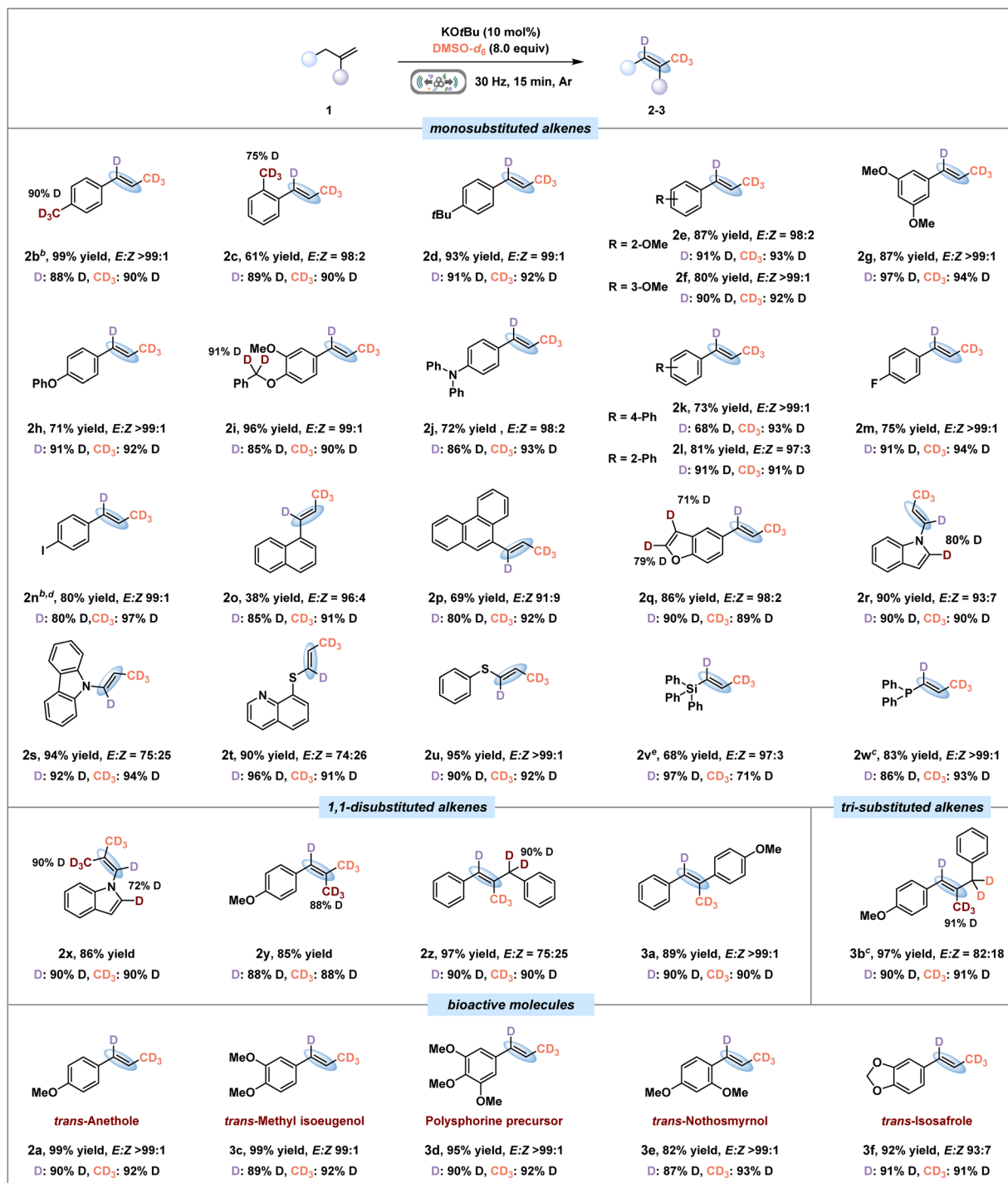


Fig. 3 Substrates scope. <sup>a</sup>Conditions: **1a** (0.4 mmol), KOtBu (10 mol%), DMSO-*d*<sub>6</sub> (8.0 equiv), in stainless-steel milling jar (5 mL) using two stainless-steel balls (diameter: 7 mm), ball milling under Ar for 15 min at 30 Hz. Deuterium incorporation and *E/Z* ratios were determined by <sup>1</sup>H NMR spectrum, isolated yields. <sup>b</sup>DMSO-*d*<sub>6</sub> (10.0 equiv). <sup>c</sup>30 min. <sup>d</sup>1 h. <sup>e</sup>3 h.

To demonstrate the scalability of the developed protocol, the alkene double-bond migration deuteration of compound **1a** was performed on 8 mmol and 6 mmol scales, affording the target product **2a** in 95% yield with 91% D-inc and 91% CD<sub>3</sub>-inc, and 96% yield with 90% D-inc and 91% CD<sub>3</sub>-inc, respectively (Fig. 4A). To examine whether the metals present in the ball mill contribute to the reaction, substrate **1a** was subjected to the

alkene double-bond migration deuteration using a ZrO<sub>2</sub>/PTFE ball-milling jar and ZrO<sub>2</sub>/PTFE balls instead of the conventional stainless-steel equipment (Fig. 4B). The corresponding product **2a** was obtained in comparable yields, indicating that the metals in the stainless-steel milling assembly do not participate significantly in the catalytic process.<sup>58–61</sup>



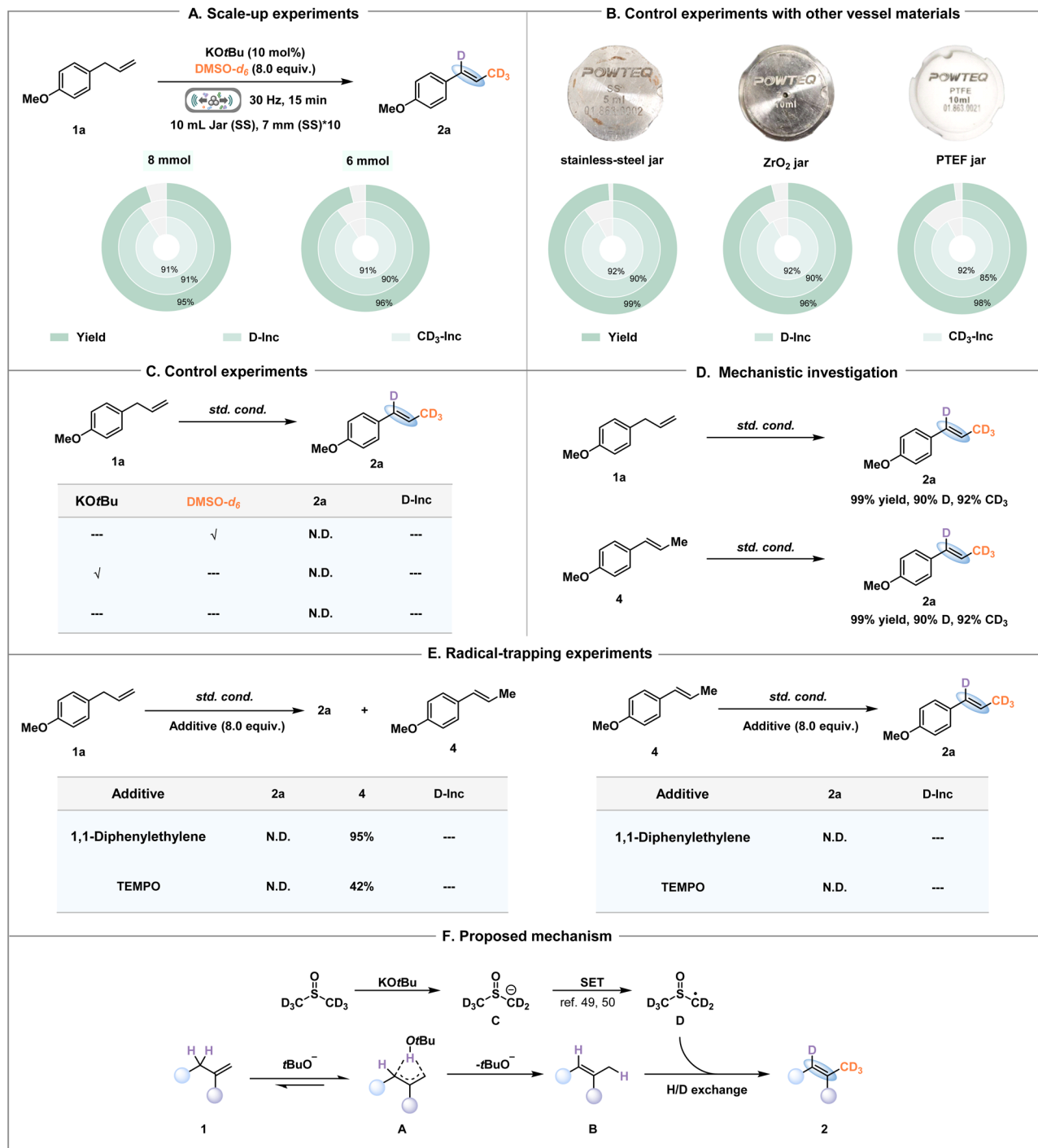


Fig. 4 (A) Scale-up reactions. (B) Control experiments with other vessel materials. (C) Control experiments. (D) Mechanistic investigation. (E) Radical-trapping experiments. (F) Proposed mechanism.

To further elucidate the reaction mechanism, control experiments mechanistic investigation and radical trapping studies were carried out. The control tests revealed that both DMSO-*d*<sub>6</sub> and KOtBu are indispensable for the deuteration involving C=C bond migration (Fig. 4C). When the isomerization product **4** of alkene **1a** was subjected to the standard reaction conditions, the outcome was consistent with that from

alkene **1a**, indicating that the reaction likely proceeds first through alkene migration, followed by a deuteration process (Fig. 4D). Under standard reaction conditions, when radical scavengers 1,1-diphenylethylene and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) were added to the reaction systems of **1a** or **4**, product **2a** could still be obtained, but the deuterium incorporation rate was completely inhibited (Fig. 4E). This



result indicates that the isomerization process does not occur *via* the radical mechanism, while the deuterium incorporation process may involve the radical pathway.

Based on the above results and previous literature reports,<sup>7,24</sup> a plausible mechanism for mechanochemical base-catalyzed isomerization and deuteration of allylbenzenes is proposed (Fig. 4F). Initially, base-mediated abstraction of the hydrogen from the allylic position leads to the formation of a four-membered ring transition state **A**. Collapse of this transition state affords the alkene isomerization product **B**. Concurrently, KO<sup>t</sup>Bu deprotonates DMSO-*d*<sub>6</sub>, resulting in the formation of the DMSO-*d*<sub>6</sub> carbanion **C**, which undergoes single-electron transfer (SET) to generate the radical species **D**.<sup>62,63</sup> This radical then abstracts hydrogen atoms from the  $\alpha$ -H and methyl in the allylic moiety of intermediate **B** through sequential H/D exchanges, ultimately resulting in the product **2**.

## Conclusions

In summary, we have established a general strategy for the mechanochemical base-catalyzed isomerization and deuteration of allylbenzenes. This system operates with only a catalytic amount of base, stoichiometric DMSO-*d*<sub>6</sub> as the deuterium source, achieving high reactivity and efficient deuterium incorporation within a remarkably short duration. The present double-bond migration deuteration protocol also extends its utility to a selection of pharmacologically interesting natural products featuring the 1-propenylbenzene scaffold. This approach offers a novel pathway for developing efficient, green, and economical deuterated drugs and holds significant potential as a platform for the remote functionalization of alkenes.

## Author contributions

Zhong Lian and Ruoxuan Liu conceived the idea and led the project. Ruoxuan Liu and Chengcheng Li performed the experiments and collected the data. Ruoxuan Liu, Xiaochun He, and Ruiling Qu prepared the SI. Writing – original draft: Ruoxuan Liu. Writing – review & editing: Ruoxuan Liu, Xiaochun He, Zhong Lian, and Xuemei Zhang. All authors participated in result discussions and provided feedback on the manuscript.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

The authors confirm that the data supporting the findings of this study are available within the article and its supplementary information (SI). Supplementary information: experimental procedures, compound characterization data, and NMR spectra. See DOI: <https://doi.org/10.1039/d5mr00126a>.

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