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Nickel-catalyzed quantitative deuterated reduction of quinolines and alkenes

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ABSTRACT

Site-selective deuterated reduction of quinoline to deuterium-labelled 1,2,3,4-tetrahydroquinoline scaffold can offer a powerful strategy for drug development, mechanistic studies, and synthesis of isotopically labeled standards. Existing deuterated reduction methods predominantly produce fully deuterated piperidines, lacking control over regioselectivity and deuterium stoichiometry. Herein, we report a Ni-catalyzed sequential deuteration protocol that enables regio- and multiplicity-controlled synthesis of mono-, di-, and tri-deuterated tetrahydroquinolines with simple deuterating reagents. Nucleophilic and electrophilic deuterating sources were combined to achieve site-selective reductive deuteration at 2-, 3- and 4-positions of quinolines. The method features broad functional group compatibility and delivers D-labeled tetrahydroquinolines in high yields with excellent positional deuterium incorporation. The protocol was further extended to the double-deuteration of different classes of olefins, quinoxalines and acridines, thus providing a unified platform for efficient synthesis of selectively D-labeled *N*-containing heterocycles and alkenes derivatives.

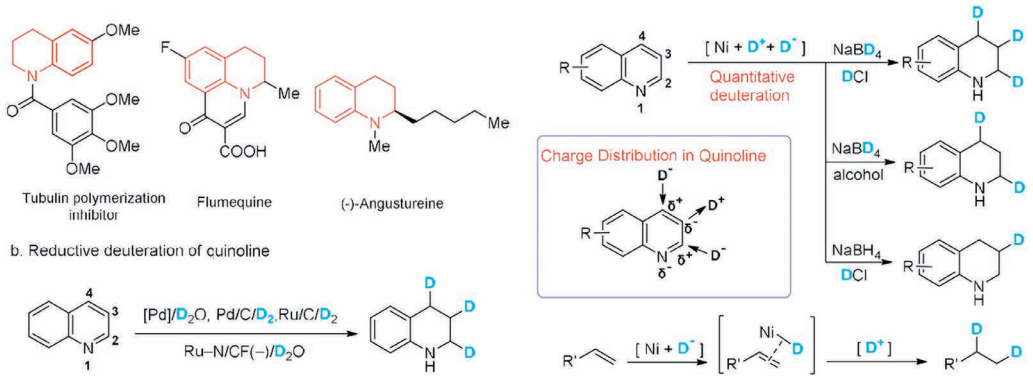
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1,2,3,4-Tetrahydroquinolines can be generated through reduction of quinolines [1–16], converting the planar aromatic ring system into saturated three-dimensional scaffold, enhancing molecular conformational flexibility [17–19]. This scaffold is widely found in various pharmaceutical molecules [20–22] such as the antibacterial agent flumequine, a tubulin polymerization inhibitor with anti-tumor activity, and a deuterated topiramate, which exhibits enhanced metabolic stability than its non-deuterated counterpart (Scheme 1a) [23]. Substituting hydrogen atoms with deuterium atoms in the piperidine ring of tetrahydroquinoline, without drastically altering its biological function, can provide a promising route for developing next-generation drugs [24–28]. These methods typically involve catalytic deuteration using heterogeneous catalytic systems such as Pd or Ru catalysts in the presence of D_2 or D_2O (Scheme 1b), which usually result in full deuteration of heterocycles [29,30]. More recently, electrochemical technique has been used in the field of reductive deuteration of quinolines by Lei and Li, in which *N*-containing heterocycles and even benzene rings can be fully deuterated [31]. These developments also deliver products that are fully deuterated at the 2-, 3-, and 4-positions of quinolines, lacking control over site-selectivity, particularly the deuterium-labelling multiplicity (*i.e.*, whether one, two, or three sites on the pyridine ring are deuterated). Therefore, a selective and number-controllable deuteration manner at specific positions within the tetrahydroquinolines scaffold remains elusive.

Charge-distribution analysis reveals that the quinoline ring possesses partial positive charge character at the C-2 and C-4 positions, while the C-3 position exhibits partial negative charge character [32]. Therefore, in principle, the 2- and 4-positions are susceptible to attack by nucleophilic deuteride (D^-), whereas the 3-position favors attack the electrophilic deuterium cations (D^+). Inspired by the NiH-catalyzed reduction of π system [33–48], herein, we designed a Ni-catalyzed sequential deuteration reaction utilizing a combination of nucleophilic and electrophilic deuterating reagents to selectively introduce 1, 2, or 3 deuterium atoms onto the quinoline ring (Scheme 1c). Nearly quantitative deuteration at each position were achieved in the mono-deuteration, di-deuteration, and tri-deuteration products. We further successfully extend this scope to various alkenes and other *N*-containing heterocycles. Scale-up reaction demonstrates the utility of our method. The difference between this method and reported method are also displayed in the example of a real drug molecule Quinfamide-d_3.

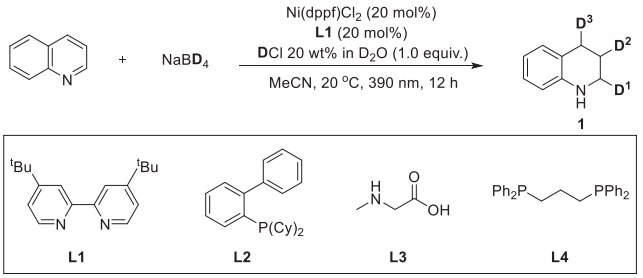
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Scheme 1. Deuteration methods of quinolines and alkenes.

Table 1
Optimization studies.[a]



Entry	Deviations	Yield (%)[b]	Ratio (%)[c]		
			D^1	D^2	D^3
1	None	91	97	94	94
2	NiI_2 instead of $Ni(dppf)Cl_2$	32	94	64	95
3	$NiCl_2$ instead of $Ni(dppf)Cl_2$	20	64	66	68
4	$B(OD)_3$ instead of DCl	35	98	95	92
5	^{t}BuOD instead of DCl	35	97	34	86
6	**L2** instead of **L1**	62	97	94	89
7	**L3** instead of **L1**	65	98	97	81
8	**L4** instead of **L1**	30	83	43	92
9	Without light	71	96	82	92

[a] Conditions: quinoline (0.10 mmol), $NaBD_4$ (2.0 equiv.), DCl 20 wt% in D_2O (1.0 equiv.), $Ni(dppf)Cl_2$ (20 mol%), Ligand (20 mol%), and MeCN (0.50 mL) were irradiated with light at 20 °C for 12 h under argon atmosphere.
[b] Isolated yields.
[c] Deuterium incorporation was determined by ^{1}H NMR spectroscopy.

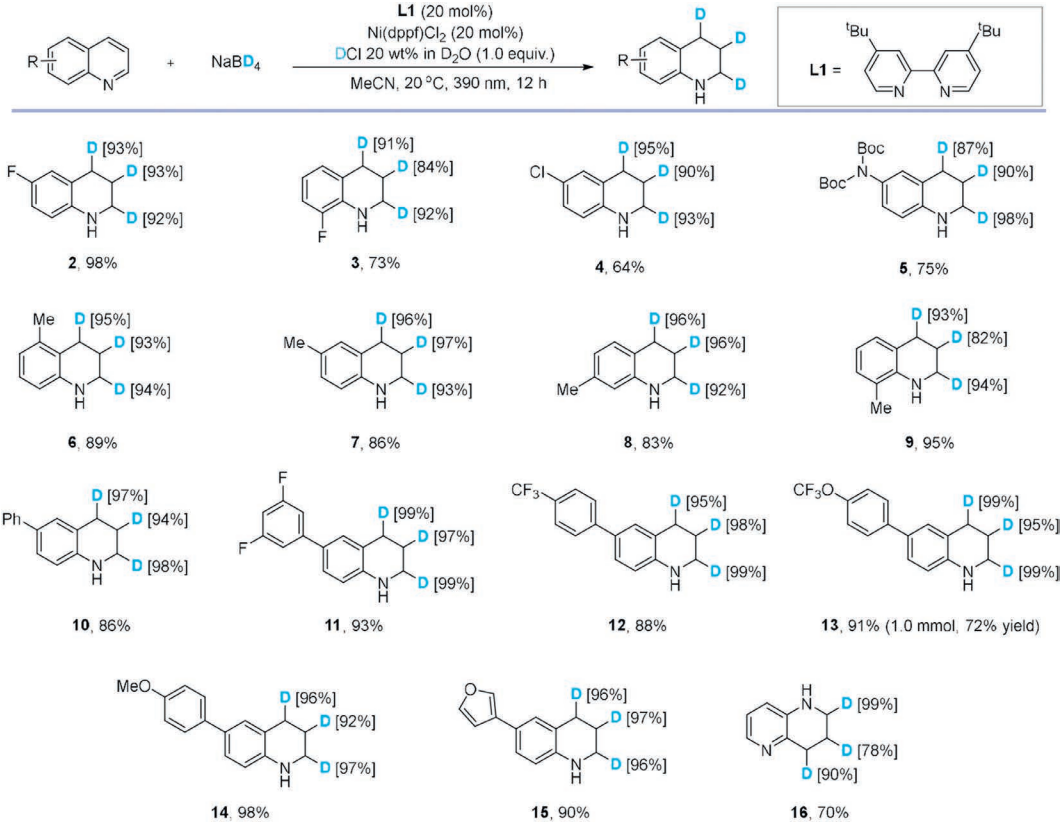
Unsubstituted quinoline was chosen as the model substrate to test our design (Table 1). The reaction was conducted in CH_3CN under purple LED light irradiation over 12 h with $Ni(dppf)Cl_2$ as the catalyst, 4,4'-di-*tert*-butyl-2,2'-bipyridyl **L1** as the ligand, $NaBD_4$ as the reductive deuterating reagent, and DCl as the electrophilic deuterating reagent. Tetrahydroquinoline-d_3 **1** was obtained with 91% yield with approximately 95% deuterium incorporation (D-incorporation) at each position of piperidine ring (entry 1). Replacing the catalyst with NiI_2 or $NiCl_2$ resulted in a dramatic decrease in both yield and D-incorporation selectivity (entries 2 and 3). Furthermore, replacement of DCl with other electrophilic deuterium sources (entries 4 and 5) or substitution of **L1** with other ligands (entries 6–8) also resulted in reduced yield and deuteration ratio. In the absence of light, the reaction did proceed, but the yield declined by 20% and the deuteration ratio of at the 2-position (D^2) decreased by 12% (entry 9). The observation suggests an added beneficial effect of light irradiation (for UV–vis experiment, see Fig. S5 in Supporting information), although we do not have any structure information of potential intermediate under excited state.

We next explored the substrate scope of this protocol (Scheme 2). A range of quinolines bearing electron withdrawing groups such as F (**2**, **3**), Cl (**4**) furnished corresponding deuterated tetrahydroquinolines-d_3 (1,2,3,4-tetrahydroquinoline-2,3,4-d_3) in approximately 64%–98% yields with deuteration rates about 90% at each position. The electron-donating groups such as $N(Boc)_2$ (**5**), methyl (**6**–**9**) at various sites on the quinoline ring were converted to the labelled products in 75%–95% yields with about 90% deuteration rates. Additionally, phenyl substituted quinolines bearing either electron withdrawing groups (F, CF_3, OCF_3) or electron-donating groups (OMe) reacted smoothly to afford desired products **10**–**15** with 86%–98% isolated yields, and deuteration rates range from 92% to 99%. Furthermore, 1,5-naphthyridine was also applicable to this system with 70% yield (**16**).

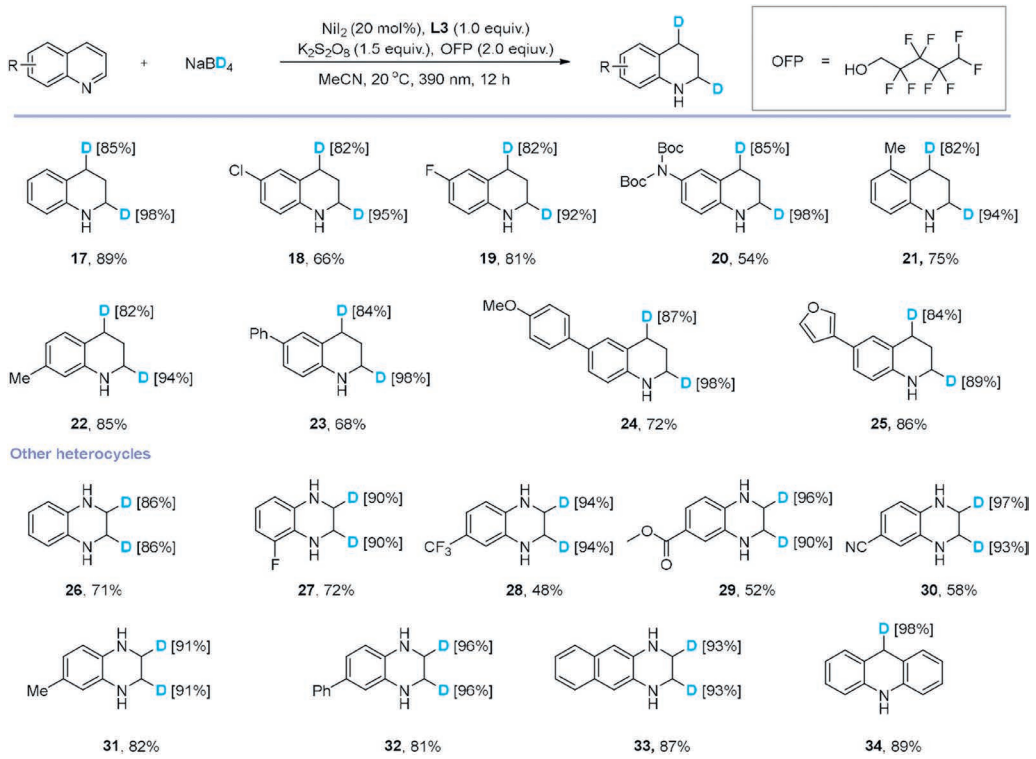
To exploit the advantage of the sequential reduction strategy, tetrahydroquinoline-d_2 was further explored (Scheme 3). After slight adjustment of the optimized reaction conditions, the reaction performed best in CH_3CN under purple LED irradiation for 12 h using NiI_2 as the catalysts, sarcosine **L3** as the ligand, $NaBD_4$ as the reductive deuterium reagent, and 2,2,3,3,4,4,5,5-octafluoro-1-pentanol (OFP) as the electrophilic proton source. $K_2S_2O_8$ was added to enhance the reaction yield (For more details of the reaction condition optimization, see Table S4 in Supporting information).

Detrahydroquinoline-d_2 (1,2,3,4-tetrahydroquinoline-2,4-d_2, **17**) was afforded in 89% yield with deuteration rates about 90% at 2- and 4-positions. The 3-position was selectively bound with a protium atom. Quinolines bearing electron withdrawing groups such as halogens (**18** and **19**) or delivered the di-deuteration products in 66%–81% yields. Moreover, electron-rich substrates containing $N(Boc)_2$ **20**, methyl (**21** and **22**), phenyl (**23** and **24**), or furanyl (**25**) reacted smoothly, providing the products in up to 86% yield with deuteration rates greater than 85%. Additionally, other heterocycles such as quinoxaline (**26**) and its derivatives bearing F (**27**), CF_3 (**28**), CO_2Me (**29**), and CN (**30**), Me (**31**), or phenyl (**32**) afforded the desired products in 50%–80% yields. Di-deuteration can also occur for polycyclic fused aromatic rings such as naphthalene derivatives (**33**) and acridine (**34**) in 87%–89% yield with deuteration rates of 93%–98%.

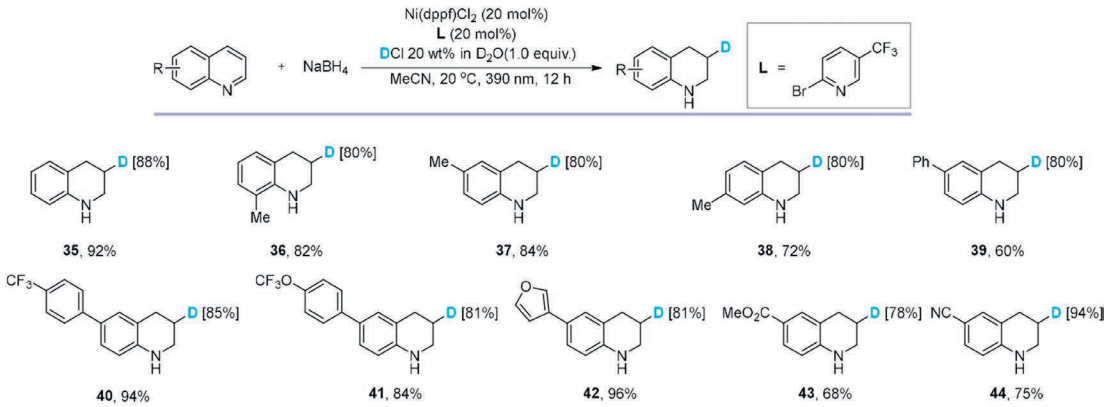
We further inverted the protium and deuterium sources displayed in Scheme 3, by replacing the nucleophilic $NaBD_4$ and electrophilic OFP to nucleophilic $NaBH_4$ and electrophilic DCl (Scheme 4), to evaluate the versatility of our sequential-reduction protocol. Under the optimized conditions, $Ni(dppf)Cl_2$ (20 mol%), ligand (20 mol%), $NaBH_4$ (2.0 equiv.), DCl (2.0 equiv.), light irradiation, MeCN, 20 °C, 12 h, quinoline was converted to the mono-deuterated 1,2,3,4-tetrahydroquinoline-3-d_1 (**35**) in 92% yield, with selective deuteration at the 3-position (88% D rates, see Table S5



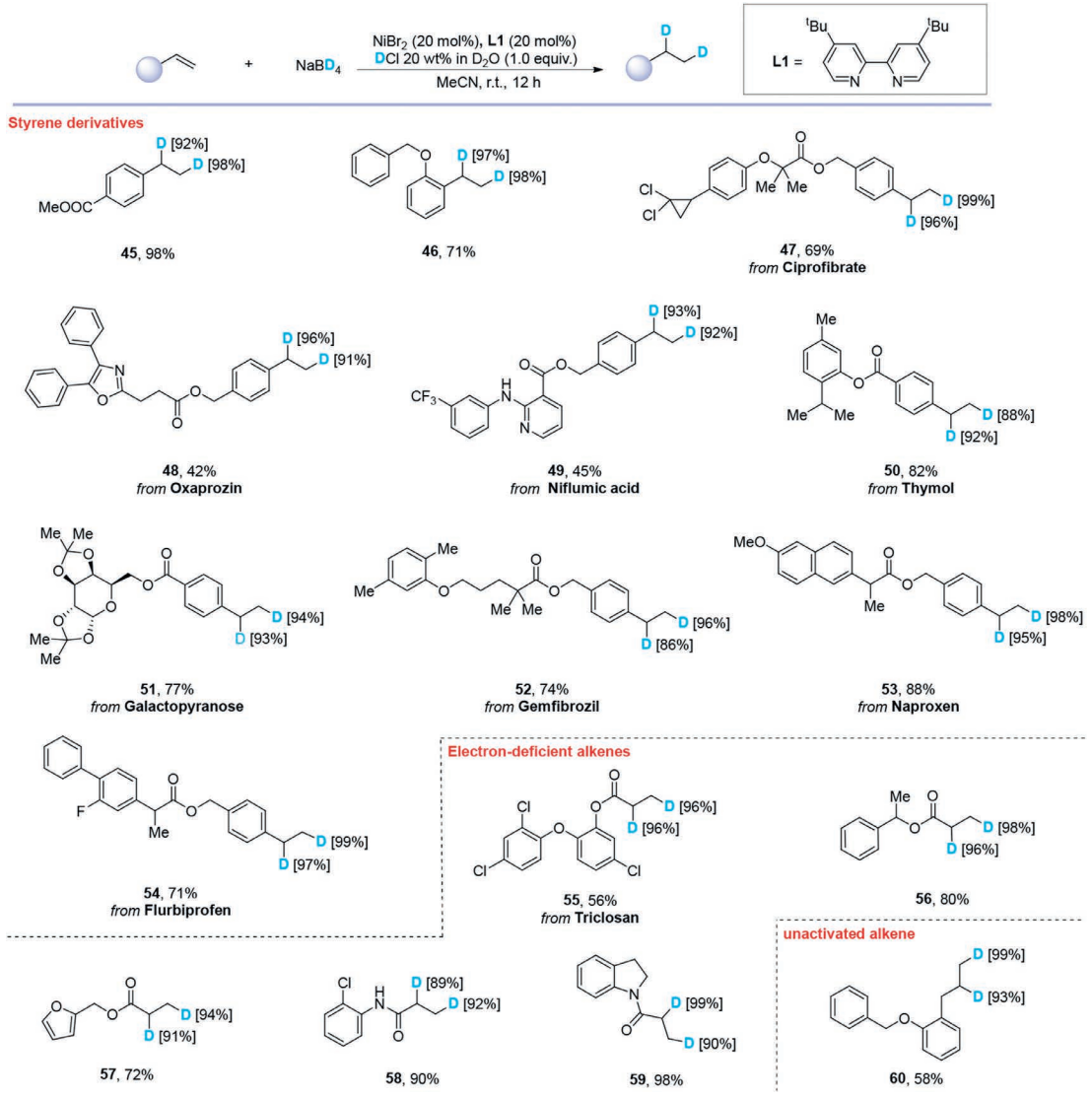
Scheme 2. Substrate scope of substituted 1,2,3,4-tetrahydroquinoline-2,3,4-d_3. Standard conditions: Ni(dppf)Cl_2 (20 mol%), ligand (20 mol%), $NaBD_4$ (0.20 mmol, 2.0 equiv.), MeCN (0.50 mL), quinoline (0.10 mmol, 1.0 equiv.) and DCl 20 wt% in D_2O (0.10 mmol, 1.0 equiv.) were irradiated with light at room temperature for 12 h under argon atmosphere. Isolated yield was given. Deuterium incorporation was determined by ^1H NMR spectroscopy.



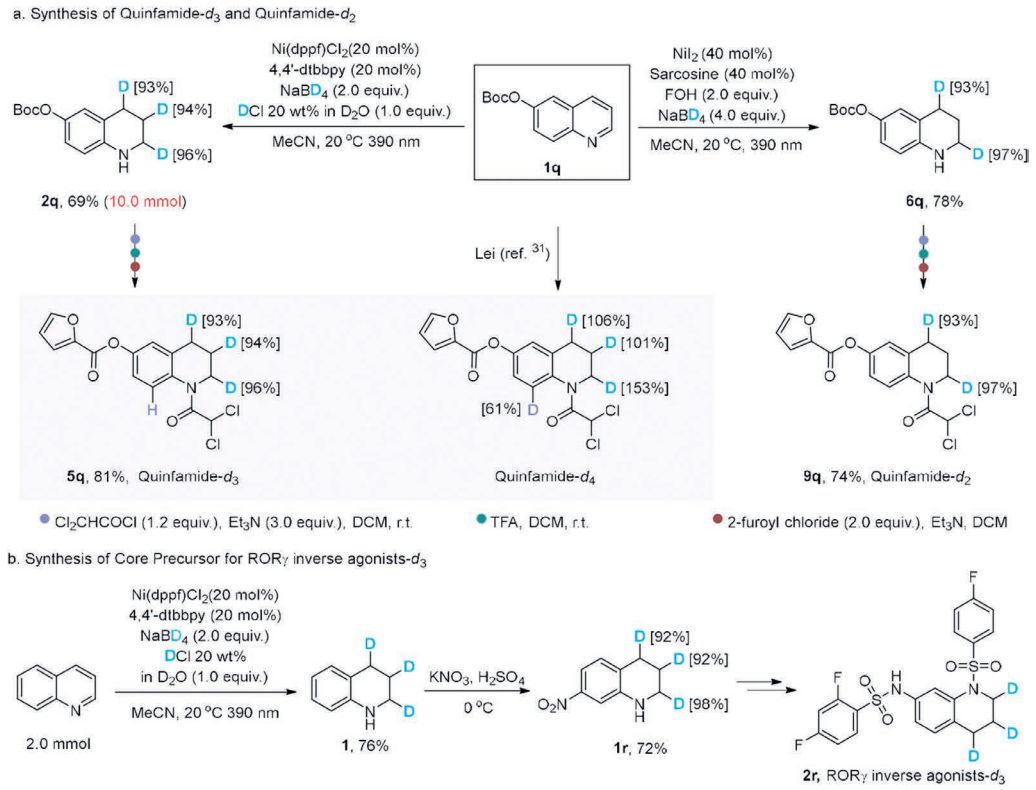
Scheme 3. Substrate scope of substituted 1,2,3,4-tetrahydroquinoline-2,4-d_2. Standard conditions: NiI_2 (20 mol%), Sarcosine (0.10 mmol, 1.0 equiv.), $K_2S_2O_8$ (0.15 mmol, 1.5 equiv.), $NaBD_4$ (0.20 mmol, 2.0 equiv.), MeCN (0.50 mL), quinoline (0.10 mmol, 1.0 equiv.) and 2,2,3,3,4,4,5,5-octafluoro-1-pentanol (0.20 mmol, 2.0 equiv.) were irradiated with light at room temperature for 12 h under argon atmosphere. Isolated yield was given. Deuterium incorporation was determined by ^1H NMR spectroscopy.



Scheme 4. Substrate scope of substituted 1,2,3,4-tetrahydroquinoline-3-d_1. Standard conditions: Ni(dppf)Cl_2 (20 mol%), ligand (20 mol%), $NaBH_4$ (0.20 mmol, 2.0 equiv.), MeCN (0.50 mL), quinoline (0.10 mmol, 1.0 equiv.) and DCl 20 wt% in D_2O (0.10 mmol, 1.0 equiv.) were irradiated with light at room temperature for 12 h under argon atmosphere. Isolated yield was given. Deuterium incorporation was determined by ^{1}H NMR spectroscopy.



Scheme 5. Substrate scope of reductive deuteration of alkenes. Standard conditions: alkene (0.10 mmol, 1.0 equiv.), $NiBr_2$ (0.020 mmol, 20 mol%), 4,4'-dtbbpy (0.020 mmol, 20 mol%), $NaBD_4$ (0.20 mmol, 2.0 equiv.), MeCN (0.50 mL) and DCl 20 wt% in D_2O (0.10 mmol, 1.0 equiv.) were irradiated at room temperature for 12 h under argon atmosphere. Isolated yield was given. Deuterium incorporation was determined by ^{1}H NMR spectroscopy.



Scheme 6. Synthetic applications.

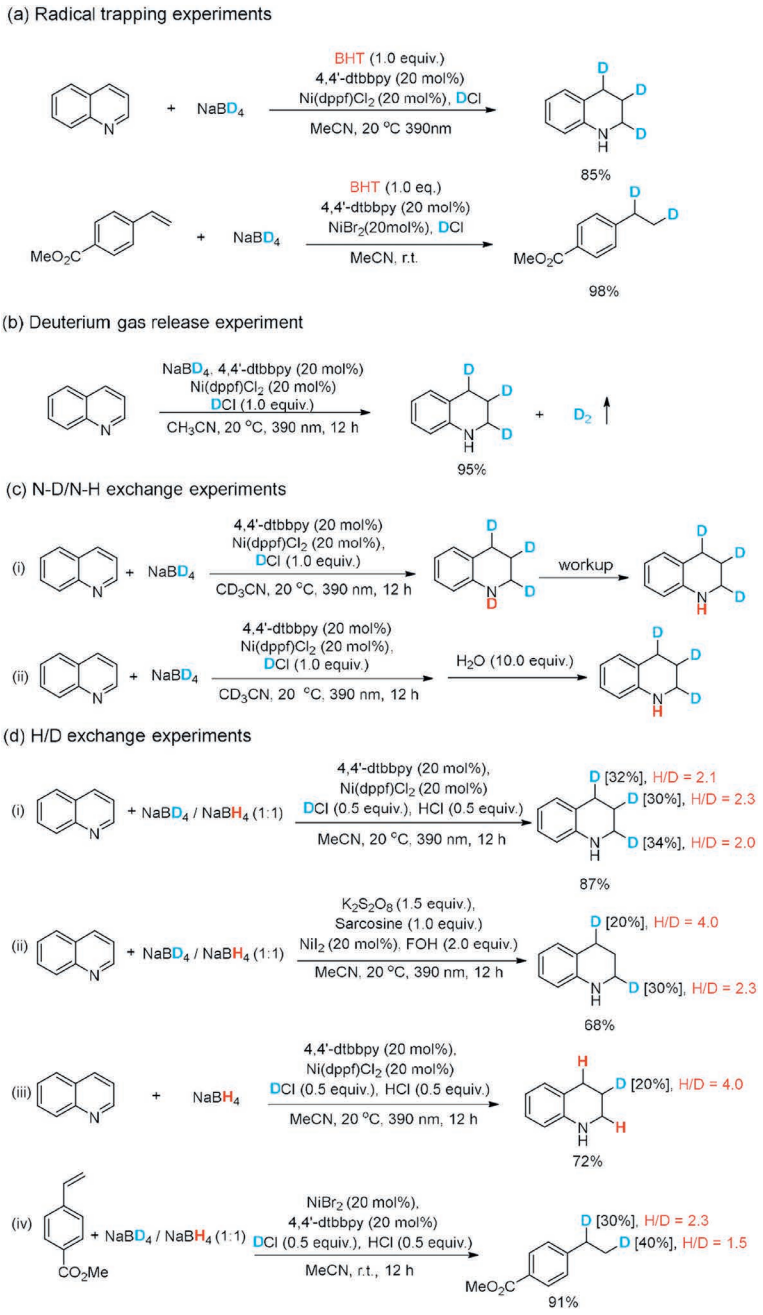
in Supporting information for details). This protocol proved broadly tolerant of diverse functionality. Electron-rich substrates bearing methyl (**36**–**38**), phenyl (**39**–**41**), or furyl (**42**) groups underwent mono-deuteration in 60 %–90 % yields with approximately 80 % D-incorporation rates. Electron-withdrawing substituents such as ester and cyano on the quinoline ring were likewise tolerated in the reaction, furnishing products **43** and **44** in around 70 % yields and high site-selectivity.

Building on the versatile and selective deuteration of quinolines, the methodology was extended to reductive deuteration of alkenes (Scheme 5) [49–52]. Under similar conditions as in Scheme 1, with $NiBr_2$ replacing the original Ni catalyst (see Table S6 in Supporting information for details), various drug derivatives such as ciprofibrate (**47**), oxaprozin (**48**), niflumic acid (**49**), thymol (**50**), galactopyranose (**51**), gemfibrozil (**52**), naproxen (**53**) and flurbiprofen (**54**) successfully delivered the desired deuterated reduction products in 42 %–88 % yields with generally greater than 90 % deuteration rates. Moreover, electron-deficient alkenes bearing β-unsaturated ester or amide groups produced 1,2-di-deuterated products **55**–**59** in 56 %–98 % yields with deuterium incorporation rates greater than 90 % at both the α- and β-positions. Notably, unactivated alkenes also underwent effective double-deuteration, as seen in the conversion of compound **60**, with the deuteration rate reach 97 %.

To demonstrate the synthetic utility of the methodology, we attempted to prepare compound **2q** *via* our deuterated reduction method on a 10.0 mmol scale, which afforded 1.74 g of compound **2q** in 69 % yield (Scheme 6a). Following up a 3-step transformation, **2q** can produce Quinfamide-d_3 **5q** in 81 % total yield with deuteration rate greater than 90 % at each position. Very recently, Lei and Li reported an electrocatalytic pathway can also yield Quinfamide-d_4, while the deuteration rate and site-selectivity are different from our approach (Scheme 6a) [31]. Moreover, we can also transfer **6q** into Quinfamide-d_2 **9q**, to provide a potential alternative for drug development (Scheme 6a). Furthermore, compound **1**, synthesized by our methods, can be nitrated to afford compound **1r** with 72 % yield, which is a key precursor for synthesizing drug molecule **2r** (Scheme 6b) [53].

Mechanistic experiments were conducted to gain deeper insight into the reaction (Scheme 7). First, the addition of the radical-trapping agent butylated hydroxytoluene (BHT) did not inhibit the reaction, suggesting that a radical-mediated mechanism is unlikely (Scheme 7a). Second, we detected the generation of deuterium gas (D_2) under standard conditions with online mass spectrometer (PM QMS) (for D_2−MS, see Fig. S3 in Supporting information). With slow releasing of *in-situ* generated D_2 gas, the target reaction proceeded successfully, which demonstrate the reaction is unlikely to involve D_2-mediated reduction of quinoline (Scheme 7b). Furthermore, N–D bond formed in the reaction system, which was subsequently converted to N–H during workup (Scheme 7c). Under the reaction conditions, a trace amount of 1,2-dihydroquinoline-2-d_1 (~5 %) was also isolated and confirmed by ^{1}H NMR spectroscopy (see Supporting information, Page S371), suggesting an initial 1,2-addition to the C=N bond at 1,2-positions (Fig. S6 in Supporting information). What's more, kinetic isotope effect (KIE) experiments, including intermolecular competition experiment of tri-deuteration, di-deuteration, and mono-deuteration, revealed that C-D bond formation is possibly the rate-limiting step (Scheme 7d). Overall, the reaction is likely mediated by a Ni(II)–D species [54] and proceeds in two stages: Addition of Ni–D to C=N and C=C bonds, and subsequent protonolysis of the Ni intermediates by selected electrophilic D^+ or H^+ reagents (see Fig. S6 for details).

In summary, we have developed a Ni-catalyzed, site- and stoichiometry-selective deuteration strategy that enables precise incorporation of deuterium atoms at specific positions on *N*-containing heterocycles and various alkenes, overcoming limitation of the precedent methods that reply on a single deuterium source and often furnish fully deuterated tetrahydroquinolines. By leveraging a sequential protocol that combines nucleophilic and electrophilic deuterium and protium sources, controlled mono-, di-,



Scheme 7. Mechanistic studies.

and tri-deuteration of 1,2,3,4-tetrahydroquinolines was achieved under operationally simple conditions. The method demonstrates broad functional group tolerance with the consumption of inexpensive, commercially available deuterating reagents, to realize high deuterium incorporation with excellent site specificity, and can potentially shed light on drug discovery as well as mechanistic study applications.

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.

CRediT authorship contribution statement

Wenyan Zhao: Writing – review & editing, Writing – original draft, Project administration, Methodology, Investigation, Formal analysis, Data curation. **Tiantian Liang:** Writing – review & editing, Writing – original draft, Resources, Formal analysis, Data curation. **Dongcheng Zheng:** Investigation, Formal analysis, Data curation. **Jiali Qiu:** Formal analysis, Data curation. **Juan Zheng:** Formal analysis, Data curation. **Yandong Wu:** Resources, Formal analysis, Data curation. **Wenhan Xu:** Formal analysis, Data curation. **Liang Liu:** Writing – review & editing, Writing – original draft, Investigation, Formal analysis, Data curation. **Fei Ye:** Writing – review & editing, Writing – original draft, Methodology, Funding acquisition.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2025.112035.

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