Supporting Information

Site-selective deuteration of diaryl alcohols by D₂O via quantum dots photocatalysis

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1. General Information

$^1$H NMR spectra were recorded using a Bruker Avance DPX 400 MHz instrument with tetramethylsilane (TMS) as internal standard. $^{13}$C NMR spectra were obtained at 100 MHz and referenced to the internal solvent signals. $^{19}$F NMR spectra were obtained at 377 MHz. Mass spectra were recorded using a Trio-2000 GC-MS spectrometer and an ApexIII (7.0 tesal) FTICR mass spectrometer (Bruker). The generated photoproducts of $H_2$ gases were characterized by GC analysis (14B Shimadzu) using Argon as the carrier gas with a molecular sieve column (5 Å; 30 m × 0.53 mm) and a thermal conductivity detector. The time-resolved emission was measured on FLS920 and fitted by exponential function with luminescence spectrometer software L900. UV-vis absorption spectra were recorded with a Shimadzu 1601PC spectrophotometer. Photoluminescence (PL) measurements were performed at room temperature using a Hitachi 4500 fluorescence spectrophotometer. EPR spectra were collected on a JES-FA200 at 300K. High-resolution transmission electron microscopy was performed by JEM 2100F (operated at an accelerating voltage of 200 kV). Commercially available reagents were used directly.
2. Synthesis of water-soluble QDs

**Water-soluble CdSe QDs.** Water-soluble CdSe QDs were synthesized by the following procedure.⁴ 40 mg selenium powder was added into 100 mL Na₂SO₃ (189 mg) aqueous solution. Then, the resulting mixture was refluxed until the Se powder completely dissolved to enable transparent Na₂SeSO₃ solution. 20 mL Na₂SeSO₃ was mixed with 380 mL solution containing 3-mercaptopropionic acid (MPA, 52 μL) and CdCl₂·5/2H₂O (92 mg). The solution was adjusted to pH = 11 with 1.0 M NaOH. The mixture was placed in a three-necked flask and deaerated with Argon for 30 min. Finally, the transparent solution was refluxed to promote the growth of CdSe QDs. Aliquots of the reaction solution were taken out at regular intervals for UV-vis absorption and emission measurements.
3. Characterization of CdSe QDs

Figure S1. High-resolution TEM image of MPA-CdSe QDs (left), indicating that the QDs give a size distribution around of ~2.0 nm. Steady-state absorption and emission spectra of the as-synthesized CdSe QDs (right), demonstrating that the first excitonic absorption peak of CdSe QDs is at 430 nm and the emission is around 470 nm (400 nm excitation).
4. Experimental procedures

A 4 mL aliquot of CdSe QDs aqueous solution was treated with 0.1 M nitric acid to remove the surface ligands on QDs. Then, the precipitates were isolated by centrifugation (6000 rpm, 4 min) and washed with MeCN to remove any residual ligands. The precipitates were redispersed in 2 mL MeCN and sonicated for 30 min in a 10 mL Pyrex tube equipped with a rubber septum and magnetic stir bar. Benzophenone (0.2 mmol), TEA (2 equiv., 0.4 mmol) and D$_2$O (25 equiv., 5 mmol) were added to the above 2 mL CdSe QDs MeCN solution. The Pyrex tube was sealed and the mixture was bubbled with an Ar gas for 30 min. The tube was then irradiated under 450 nm LEDs at room temperature. And the generated H$_2$ gas in the headspace of reactor was taken with a gas-tight syringe and measured by using a gas chromatograph (Shimadzu GC2014CAFC/APC) equipped with a thermal conductivity detector and a 5 Å molecular sieves GC packed column. Ar was used as a carrier gas. After the reaction, this solution was extracted with EtOAc (3 × 5 mL). The combined organic phase was washed with brine. Upon removal of solvent under vacuum, the residue was purified by silica gel chromatography to give the final product.

Figure S2. The photoreactor used in our system.
5. Reaction Optimization Tables

Table S1. Optimization of the reductive deuteration reaction of benzophenone.\(^a\)

![Diagram showing the optimization of the reductive deuteration reaction of benzophenone.]

<table>
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<th>Entry</th>
<th>Solvent (mL)</th>
<th>Reductant (eq.)</th>
<th>Deuterated reagent (eq.)</th>
<th>3a (%)</th>
<th>4a (%)</th>
<th>2a (%)</th>
<th>D incorp. (%)</th>
<th>Cum. (%a)</th>
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\(^a\)Conditions: 0.2 mmol 1a, TEA (2 equiv.), CdSe QDs (2.0 × 10\(^{-5}\) M) in 2 mL of CH\(_3\)CN. The system was irradiated by 450 nm LEDs under Ar for 12 h at room temperature. Abbreviations: N.R. = not reaction. Yield was detected by \(^1\)H NMR using 1,3,5-trimethoxybenzene as an internal standard. \(^b\)Isolated yields were provided. \(^c\)The D content was determined by \(^1\)H NMR spectroscopy. \(^d\)The reaction was carried out under air conditions. \(^e\)Bulk CdSe was used as photocatalysts. \(^f\)CdSe QDs was absent from the reaction system. \(^g\)The reaction was carried out in the dark. \(^h\)TEA was absent from the reaction system.
6. $^1$H NMR and $^2$H NMR of 2a
Figure S3. $^1$H NMR and $^2$H NMR of diphenyl methanol in CD$_3$CN (a, b); $^1$H NMR and $^2$H NMR of 2a in CD$_3$CN (c, d). As shown in Figure S3, the deuterium incorporation of 2a is 96%. Compared with $^1$H NMR and $^2$H NMR of diphenyl methanol, the $^1$H NMR and $^2$H NMR of 2a indicates that the hydrogen at the benzyl position of 2a is deuterated and the deuterium incorporation is 96%.
7. The reductive deuteration of benzoylcyclohexane, 1-cyclohexylethan-1-one and benzophenone imine

Benzoylcyclohexane, 1-cyclohexylethan-1-one and benzophenone imine were carried out under the optimal conditions. No deuteration products were observed. The possible reason was that the benzoylcyclohexane, 1-cyclohexylethan-1-one and benzophenone imine could not be reduced by photogenerated electrons in the conduction band of QDs.
8. Gram-scale synthesis, reusability experiments and drug molecular synthesis

Gram-scale synthesis

A 120 mL aliquot of CdSe QDs aqueous solution was treated with 0.10 M hydrochloric acid to precipitate the CdSe QDs. The precipitates were isolated by centrifugation (6000 rpm, 4.0 min) and washed with CH$_3$CN to remove any residual ligands. The precipitates were redispersed in 60 mL CH$_3$CN and sonicated for 0.5 h, in a 100 mL Pyrex tube equipped with a rubber septum and magnetic stir bar. phenyl(o-tolyl)methane (5.0 mmol, 0.98 g), D$_2$O (2.5 mL) and TEA (10.0 mmol, 1.39 mL) were added into the above 60 mL CdSe QDs CH$_3$CN solution. The Pyrex tube was sealed and the mixture was bubbled with Ar gas for 30 min. The tube was then irradiated under 450 nm LEDs at room temperature for 24 h. After reaction, this solution was extracted with EtOAc (3 × 30 mL). The combined organic phase was washed with brine. Upon removal of solvent under vacuum, the residue was purified by silica gel chromatography to give the final product.

Reusability experiments

For cycle 1, using the general procedure, reusability reaction was performed under standard conditions. After completion of each-cycle reaction, the QDs catalyst was separated from the reaction mixture by centrifugation (8000 rpm, 6.0 min), and the obtained photocatalyst was washed three times by CH$_3$CN to remove any volatile organic compound from the surface. The QDs photocatalyst was then used again for another cycle of photocatalysis under the standard reaction conditions.
Figure S4. Reusability experiment of CdSe QDs.
Drug molecular synthesis

2b (0.2 mmol), N,N-Dimethylamino chloroethane hydrochloride (2.0 equiv., 0.4 mmol) and KOH (3 equiv., 0.6 mmol) in 4.0 mL DMSO at room temperature for 12 h. This solution was extracted with EtOAc (3 × 10 mL) after reaction. The combined organic phase was washed with brine. Upon removal of solvent under vacuum, the residue was purified by silica gel chromatography to give the final product.

2d (0.2 mmol), N,N-Dimethylamino chloroethane hydrochloride (2.0 equiv., 0.4 mmol) and KOH (3 equiv., 0.6 mmol) in 4.0 mL DMSO at room temperature for 12 h. This solution was extracted with EtOAc (3 × 10 mL) after reaction. The combined organic phase was washed with brine. Upon removal of solvent under vacuum, the residue was purified by silica gel chromatography to give the final product.
9. Mechanism study

Radical trapping experiments

Radical trapping experiments were performed as follows: 0.2 mmol 1a, TEA (2 equiv.), TEMPO (2.0 equiv., 0.20 mmol) and CdSe QDs (2.0 × 10^{-5} M) in 2 mL of CH\textsubscript{3}CN. The system was irradiated by 450 nm LEDs under Ar for 12 h at room temperature. Yield was detected by \textsuperscript{1}H NMR.

Steady-state emission quenching of CdSe QDs

First, 4.0 mL of a solution of water-soluble CdSe QDs was adjusted using 0.1 M hydrochloric acid until the QDs aggregated. After the precipitate was separated by centrifugation (6000 rpm, 4.0 min), the particles were re-dispersed in 2.0 mL CH\textsubscript{3}CN in a quartz cell. The quencher concentration of TEA and 1a was increased incrementally from 0 to 2.5 × 10^{-5} M. The emission from the CdSe QDs monitored during the addition. Quenching of the band edge reflects surface interactions of QDs.

Figure S5. The quenching of steady-state emission spectra of CdSe QDs with different amount of TEA (left) and 1a (right) in the solution (405 nm excitation).
Control Experiment

Figure S6. Control experiments. It indicated that the gas phase product of H₂ was significantly suppressed in the presence of benzophenone (1a).

EPR Experiment

Electron paramagnetic resonance (EPR) measurements were performed at room temperature using a Bruker EMX-10/12 EPR spectrometer operated at X-band frequency. For EPR measurements, a solution of 1a (5.0 × 10⁻² M), DMPO (0.02 M) and CdSe QDs (1.0 × 10⁻⁵ M) in MeCN was irradiated with 450 nm LEDs for 30 s under Ar atmosphere in a sealed glass capillary. After irradiation, the sealed glass tube was placed in the microwave cavity of EPR spectrometer.
10. Characterization data for products
diphenylmethan-d$_1$-ol (2a)

The product is obtained as white solid (isolated yield: 97%). D incorporation by $^1$H NMR: 96%. $^1$H NMR (400 MHz, Chloroform-d$_2$) δ 7.45–7.29 (m, 8H), 7.29–7.21 (m, 2H), 5.81 (s, 0.04H), 2.28 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 143.78, 128.51, 127.59, 126.57, 75.86 (t, $J = 44.33$ Hz). HRMS (EI) Calcd. for C$_{13}$H$_{11}$OD [M$^+$]: 185.0951, Found: 185.0945.

phenyl(o-tolyl)methan-d$_1$-ol (2b)

The product is obtained as white solid (isolated yield: 86%). D incorporation by $^1$H NMR: 92%. $^1$H NMR (400 MHz, Chloroform-d$_2$) δ 7.38 (d, $J = 7.4$ Hz, 1H), 7.19 (s, 4H), 7.17–7.05 (m, 3H), 7.02 (d, $J = 7.0$ Hz, 1H), 5.83 (s, 0.08H), 2.30 (s, 1H), 2.12 (s, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 142.90, 141.47, 135.41, 130.57, 128.50, 127.58, 127.16, 126.36, 126.17, 72.96 (t, $J = 44.04$ Hz), 19.41. HRMS (EI) Calcd. for C$_{14}$H$_{13}$OD [M$^+$]: 199.0917, Found: 199.0124.

phenyl(m-tolyl)methan-d$_1$-ol (2c)

The product is obtained as white solid (isolated yield: 79%). D incorporation by $^1$H NMR: 96%. $^1$H NMR (400 MHz, Chloroform-d$_2$) δ 7.39–7.28 (m, 4H), 7.27–7.12 (m, 4H), 7.06 (d, $J = 7.1$ Hz, 1H), 5.75 (s, 0H), 2.31 (s, 4H), 2.20 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 143.87, 143.78, 138.19, 128.49, 128.43, 128.37, 127.52, 127.23, 126.54, 123.67, 75.88 (t, $J = 44.21$ Hz), 21.50. HRMS (EI) Calcd. for C$_{14}$H$_{13}$OD [M$^+$]: 199.0917, Found: 199.1101.

phenyl(p-tolyl)methan-d$_1$-ol (2d)

The product is obtained as white solid (isolated yield: 91%). D incorporation by $^1$H NMR: 88%. $^1$H NMR (400 MHz, Chloroform-d$_2$) δ 7.30–7.19 (m, 4H), 7.15 (d, $J = 7.7$ Hz, 3H), 7.04 (d, $J = 7.7$ Hz, 2H), 5.67 (s, 0.12H), 2.26 (s, 1H), 2.23 (s, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) δ 144.03, 143.97, 141.03, 140.97, 137.28, 129.21, 128.47, 127.47, 126.58, 126.51, 75.68 (t, $J = 44.44$ Hz), 21.15. HRMS (EI) Calcd. for C$_{14}$H$_{13}$OD [M$^+$]: 199.0917, Found: 199.1101.

(2-fluorophenyl)(phenyl)methan-d$_1$-ol (2e)
The product is obtained as white solid (isolated yield: 78%). D incorporation by $^1$H NMR: 87%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.50 (t, $J$ = 7.5 Hz, 1H), 7.40 (d, $J$ = 7.6 Hz, 2H), 7.33 (t, $J$ = 7.2 Hz, 2H), 7.29–7.21 (m, 2H), 7.14 (t, $J$ = 7.5 Hz, 1H), 7.06–6.96 (m, 1H), 6.13 (s, 0.13H), 2.34 (s, 1H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 159.94 (d, $J$ = 246.2 Hz), 142.70, 130.91 (d, $J$ = 13.1 Hz), 129.18, 129.10, 128.53, 127.74, 126.39, 124.33 (d, $J$ = 3.5 Hz), 115.39 (d, $J$ = 21.6 Hz), 70.12 (t, $J$ = 44.24 Hz). $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -118.53. HRMS (EI) Calcd. for C$_{13}$H$_{10}$FOD [M]$^+$: 203.0857, Found: 203.0850.

(3-fluorophenyl)(phenyl)methan-$d_1$-ol (2f)

The product is obtained as white solid (isolated yield: 85%). D incorporation by $^1$H NMR: 91%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.37 (d, $J$ = 4.6 Hz, 4H), 7.34–7.24 (m, 3H), 7.21–7.07 (m, 2H), 7.02–6.92 (m, 1H), 5.81 (s, 0.09H), 2.43 (s, 1H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 161.90 (d, $J$ = 246.1 Hz), 145.27 (d, $J$ = 6.7 Hz), 142.22, 128.90 (d, $J$ = 8.1 Hz), 127.61, 126.86, 125.54, 121.02 (d, $J$ = 2.9 Hz), 113.30 (d, $J$ = 21.2 Hz), 112.36 (d, $J$ = 22.2 Hz), 74.21 (t, $J$ = 44.44 Hz). $^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -111.94. HRMS (EI) Calcd. for C$_{13}$H$_{10}$FOD [M]$^+$: 203.0857, Found: 203.0851.

(4-fluorophenyl)(phenyl)methan-$d_1$-ol (2g)

The product is obtained as white solid (isolated yield: 78%). D incorporation by $^1$H NMR: 91%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.39–7.31 (m, 6H), 7.29 (d, $J$ = 6.2 Hz, 1H), 7.02 (t, $J$ = 8.8 Hz, 2H), 5.84 (s, 0.11H), 2.17 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 162.185 (d, $J$ = 245.43 Hz), 143.60, 142.35, 133.47, 128.77, 128.73, 128.03, 127.98, 126.67, 75.37 (t, $J$ = 44.14 Hz). HRMS (EI) Calcd. for C$_{13}$H$_{10}$ClOD [M]$^+$: 219.0561, Found: 219.0552.

(4-chlorophenyl)(phenyl)methan-$d_1$-ol (2h)

The product is obtained as white solid (isolated yield: 89%). D incorporation by $^1$H NMR: 91%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.33 (d, $J$ = 4.3 Hz, 4H), 7.30 (s, 5H), 5.79 (s, 0.09H), 2.27 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 143.56, 142.35, 133.47, 128.77, 128.73, 128.03, 127.98, 126.67, 75.37 (t, $J$ = 44.14 Hz). HRMS (EI) Calcd. for C$_{13}$H$_{10}$ClOD [M]$^+$: 219.0561, Found: 219.0552.

(3-chlorophenyl)(phenyl)methan-$d_1$-ol (2i)
The product is obtained as white solid (isolated yield: 89%). D incorporation by $^1$H NMR: 80%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.39 (s, 1H), 7.34 (d, $J = 4.1$ Hz, 5H), 7.23 (s, 3H), 5.77 (s, 0.20H), 2.35 (s, 1H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 145.74 (d, $J = 5.8$ Hz), 143.20 (d, $J = 6.1$ Hz), 134.42, 129.74, 128.69, 127.95, 127.65, 126.59, 124.64, 75.29 (t, $J = 44.14$ Hz). HRMS (EI) Calcd. for C$_{13}$H$_{10}$ClOD [M]$^+$: 219.0561, Found: 219.0557.

**phenyl(4-(trifluoromethyl)phenyl)methan-$d_7$-ol (2j)**

![Image](image.png)

The product is obtained as white solid (isolated yield: 56%). D incorporation by $^1$H NMR: 85%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.58 (d, $J = 7.7$ Hz, 2H), 7.50 (d, $J = 7.7$ Hz, 2H), 7.34 (s, 4H), 7.30 (s, 1H), 5.87 (s, 0.15H), 2.33 (s, 1H).

$^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 147.46, 143.10, 129.53 (q, $J = 32.2$ Hz), 128.78, 128.11, 126.67, 126.63, 125.41 (q, $J = 3.8$ Hz), 122.79, 75.36 (t, $J = 44.29$ Hz).

$^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -62.47.


**phenyl(3-(trifluoromethyl)phenyl)methan-$d_7$-ol (2k)**

![Image](image.png)

The product is obtained as white solid (isolated yield: 74%). D incorporation by $^1$H NMR: 75%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.71 (s, 1H), 7.58–7.50 (m, 2H), 7.45 (t, $J = 7.7$ Hz, 1H), 7.37 (d, $J = 4.3$ Hz, 4H), 7.34–7.29 (m, 1H), 5.88 (s, 0.25H), 2.39 (s, 1H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 144.61 (d, $J = 6.0$ Hz), 143.12 (d, $J = 6.1$ Hz), 130.81 (q, $J = 32.2$ Hz), 129.84, 128.88, 128.78, 128.09, 126.63, 124.32 (q, $J = 3.8$ Hz), 123.16 (q, $J = 3.9$ Hz), 75.35 (t, $J = 44.52$ Hz).

$^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -62.53.


**phenyl(2-(trifluoromethyl)phenyl)methan-$d_7$-ol (2l)**

![Image](image.png)

The product is obtained as white solid (isolated yield: 73%). D incorporation by $^1$H NMR: 94%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.67 (dd, $J = 11.0$, 8.0 Hz, 2H), 7.55 (t, $J = 7.7$ Hz, 1H), 7.44–7.31 (m, 6H), 7.31–7.24 (m, 1H), 6.31 (s, 0.06H), 2.48 (s, 1H). $^{13}$C NMR (101 MHz, Chloroform-$d$) $\delta$ 142.72, 142.31, 132.35, 129.54, 128.41, 127.76, 127.58, 126.47, 125.55 (q, $J = 5.8$ Hz), 70.52 (t, $J = 45.32$ Hz).

$^{19}$F NMR (377 MHz, CDCl$_3$) $\delta$ -57.60.


**methyl 4-(hydroxy(phenyl)methyl-$d_7$)benzoate (2m)**

![Image](image.png)
The product is obtained as white solid (isolated yield: 57%). D incorporation by $^1$H NMR: 94%. $^1$H NMR (400 MHz, Chloroform- $d$) $\delta$ 8.00 (d, $J = 8.1$ Hz, 2H), 7.47 (d, $J = 8.0$ Hz, 2H), 7.35 (s, 4H), 7.31–7.27 (m, 1H), 5.88 (s, 0.06H), 3.90 (s, 3H), 2.35 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 166.90, 148.60, 143.20, 129.81, 129.30, 128.72, 127.99, 126.65, 126.33, 75.54 (t, $J = 64.99$ Hz), 52.10. HRMS (EI) Calcd. for C$_{15}$H$_{13}$O$_3$D [M]$^+$: 243.1006, Found: 243.0992.

(4-(allyloxy)phenyl)(phenyl)methan- $d_1$-ol (2n)

The product is obtained as white solid (isolated yield: 40%). D incorporation by $^1$H NMR: 93%. $^1$H NMR (400 MHz, Methanol- $d_4$) $\delta$ 7.41–7.16 (m, 7H), 6.86 (d, $J = 8.6$ Hz, 2H), 6.03 (ddt, $J = 15.9$, 10.5, 5.2 Hz, 1H), 5.72 (s, 0.07H), 5.45–5.30 (m, 1H), 5.22 (d, $J = 10.6$ Hz, 1H), 4.50 (d, $J = 5.1$ Hz, 2H), 3.30 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 161.85, 148.59, 140.78, 137.52, 131.74, 131.57, 130.62, 130.14, 119.92, 118.05, 78.88 (t, $J = 64.64$ Hz), 72.34. HRMS (EI) Calcd. for C$_{16}$H$_{15}$O$_2$D [M]$^+$: 241.1213, Found: 241.1206.

[1,1'-biphenyl]-4-yl(phenyl)methan- $d_1$-ol (2o)

The product is obtained as white solid (isolated yield: 80%). D incorporation by $^1$H NMR: 87%. $^1$H NMR (400 MHz, Chloroform- $d$) $\delta$ 7.60–7.58 (m, 4H), 7.51–7.42 (m, 6H), 7.38 (t, $J = 7.5$ Hz, 3H), 7.31 (t, $J = 7.2$ Hz, 1H), 5.89 (s, 0.13H), 2.40 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 142.67, 141.75, 139.74, 139.43, 127.71, 127.50, 126.59, 126.25, 126.19, 126.03, 125.93, 125.51, 74.57 (t, $J = 44.44$ Hz). HRMS (EI) Calcd. for C$_{19}$H$_{15}$OD [M]$^+$: 261.1264, Found: 261.1260.

(4-phenoxyphenyl)(phenyl)methan- $d_1$-ol (2p)

The product is obtained as white solid (isolated yield: 36%). D incorporation by $^1$H NMR: 80%. $^1$H NMR (400 MHz, Chloroform- $d$) $\delta$ 7.47–7.24 (m, 9H), 7.11 (t, $J = 7.4$ Hz, 1H), 7.07–6.93 (m, 4H), 5.84 (s, 0.1 H), 2.26 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 157.12, 156.73, 143.84, 143.78, 138.71, 138.65, 129.76, 128.55, 128.09, 127.63, 126.49, 123.35, 118.98, 118.76, 75.41 (t, $J = 44.44$ Hz). HRMS (EI) Calcd. for C$_{19}$H$_{15}$O$_2$D [M]$^+$: 277.1213, Found: 277.1209.

di([1,1'-biphenyl]-4-yl)methan- $d_1$-ol (2q)

The product is obtained as white solid (isolated yield: 83%). D incorporation by $^1$H NMR: 90%. $^1$H NMR (400 MHz, Chloroform- $d$) $\delta$ 7.58 (dd, $J = 7.6$, 3.0 Hz, 8H), 7.49 (d, $J = 8.1$ Hz, 4H), 7.42 (t, $J = 7.5$ Hz, 4H), 7.33 (t, $J = 7.3$ Hz, 2H), 5.93 (s, 0.10H), 2.30 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$
142.73, 140.79, 140.62, 128.78, 127.34, 127.11, 126.99, 75.46 (t, $J = 43.43$ Hz). HRMS (EI) Calcd. for C$_{25}$H$_{19}$OD [M]$^+$: 337.1577, Found: 337.1133.

**bis(4-chlorophenyl)methan-1-ol (2r)**

![Structure of bis(4-chlorophenyl)methan-1-ol (2r)](image)

The product is obtained as white solid (isolated yield: 90%). D incorporation by $^1$H NMR: 85%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.35–7.23 (m, 8H), 5.77 (s, 0.15H), 2.29 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 141.95, 133.79, 128.89, 127.99, 74.93 (t, $J = 43.43$ Hz). HRMS (EI) Calcd. for C$_{13}$H$_9$Cl$_2$OD [M]$^+$: 253.0171, Found: 253.0166.

**bis(4-bromophenyl)methan-1-ol (2s)**

![Structure of bis(4-bromophenyl)methan-1-ol (2s)](image)

The product is obtained as white solid (isolated yield: 52%). D incorporation by $^1$H NMR: 91%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.45 (d, $J = 8.3$ Hz, 4H), 7.20 (d, $J = 8.3$ Hz, 4H), 5.72 (s, 0.09H), 2.34 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 142.39, 131.86, 128.33, 121.92, 74.79 (t, $J = 64.64$ Hz). HRMS (EI) Calcd. for C$_{13}$H$_9$Br$_2$OD [M]$^+$: 340.9161, Found: 340.9134.

**4,4'-(hydroxymethylene-d$_1$)dibenzonitrile (2t)**

![Structure of 4,4'-(hydroxymethylene-d$_1$)dibenzonitrile (2t)](image)

The product is obtained as white solid (isolated yield: 77%). D incorporation by $^1$H NMR: 70%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.63 (d, $J = 8.2$ Hz, 4H), 7.49 (d, $J = 8.2$ Hz, 4H), 5.91 (s, 0.30H), 2.79 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 147.73, 132.59, 127.16, 118.45, 111.91, 74.46 (t, $J = 63.63$ Hz). HRMS (EI) Calcd. for C$_{15}$H$_{11}$N$_2$OD [M]$^+$: 235.0856, Found: 235.0855.

**phenyl(thiophen-2-yl)methan-d$_1$-ol (2u)**

![Structure of phenyl(thiophen-2-yl)methan-d$_1$-ol (2u)](image)

The product is obtained as yellow solid (isolated yield: 50%). D incorporation by $^1$H NMR: 84%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.57 (d, $J = 7.3$ Hz, 2H), 7.49 (t, $J = 7.4$ Hz, 2H), 7.44 (d, $J = 7.2$ Hz, 1H), 7.38 (d, $J = 5.0$ Hz, 1H), 7.09–7.04 (m, 1H), 7.01 (s, 1H), 6.18 (s, 0.16H), 2.57 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 148.08, 143.08, 128.55, 128.02, 126.66, 126.31, 125.41, 124.89, 72.06 (t, $J = 45.45$ Hz). HRMS (EI) Calcd. for C$_{11}$H$_9$SOD [M]$^+$: 191.0515, Found: 191.0509.

**phenyl(pyridin-2-yl)methan-d$_1$-ol (2v)**

![Structure of phenyl(pyridin-2-yl)methan-d$_1$-ol (2v)](image)
The product is obtained as yellow solid (isolated yield: 40%). D incorporation by $^1$H NMR: 89%.

$^1$H NMR (400 MHz, Chloroform-d) $\delta$ 8.51 (d, $J = 4.9$ Hz, 1H), 7.70–7.60 (m, 3H), 7.38–7.32 (m, 3H), 7.28–7.24 (m, 1H), 7.20 – 7.16 (m, 1H), 5.77 (s, 0.11H), 4.70 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 162.13, 147.23, 143.57, 137.12, 128.57, 128.24, 127.25, 126.80, 122.32, 121.43, 72.43 (t, $J = 9.09$ Hz). HRMS (EI) Calcd. for C$_{12}$H$_{10}$NOD [M]$^+$: 186.0903, Found: 186.0912.

**phenyl(pyridin-3-yl)methan-d$_1$-ol (2w)**

The product is obtained as yellow solid (isolated yield: 74%). D incorporation by $^1$H NMR: 86%.

$^1$H NMR (400 MHz, Chloroform-d) $\delta$ 8.47 (s, 1H), 8.32 (d, $J = 4.3$ Hz, 1H), 7.69 (d, $J = 7.7$ Hz, 1H), 7.38–7.29 (m, 4H), 7.27 (d, $J = 5.1$ Hz, 1H), 7.23–7.16 (m, 1H), 5.80 (s, 0.14H), 4.02 (s, 1H).

$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 148.13, 147.90, 143.25, 139.89, 134.58, 128.66, 127.84, 126.56, 123.52, 73.36 (t, $J = 9.09$ Hz).


**phenyl(pyridin-4-yl)methan-d$_1$-ol (2x)**

The product is obtained as yellow solid (isolated yield: 62%). D incorporation by $^1$H NMR: 86%.

$^1$H NMR (400 MHz, Chloroform-d) $\delta$ 7.60 (d, $J = 7.9$ Hz, 2H), 7.50 (d, $J = 7.9$ Hz, 2H), 7.32 (d, $J = 7.3$ Hz, 5H), 5.85 (s, 0.14H), 2.52 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 148.86, 142.83, 132.26, 128.88, 128.30, 127.02, 126.69, 118.80, 111.17, 75.23 (t, $J = 9.09$ Hz). HRMS (EI) Calcd. for C$_{12}$H$_{10}$NOD [M]$^+$: 186.0903, Found: 186.0897.

**naphthalen-2-yl(phenyl)methan-d$_1$-ol (2y)**

The product is obtained as white solid (isolated yield: 50%). D incorporation by $^1$H NMR: 85%. $^1$H NMR (400 MHz, Chloroform-d) $\delta$ 7.87 (s, 1H), 7.84–7.73 (m, 3H), 7.50–7.43 (m, 2H), 7.40 (d, $J = 8.6$ Hz, 3H), 7.32 (t, $J = 7.5$ Hz, 2H), 7.26 (d, $J = 7.1$ Hz, 1H), 5.96 (s, 0.85H), 2.39 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 143.80, 141.29, 133.48, 135.11, 128.68, 128.46, 128.23, 127.82, 126.88, 126.32, 126.01, 125.23, 124.93, 76.35 (s, $J = 9.09$ Hz). HRMS (EI) Calcd. for C$_{17}$H$_{13}$OD [M]$^+$: 235.1107, Found: 235.1100.

**9H-fluoren-9-d$_1$-9-ol (2z)**

The product is obtained as white solid (isolated yield: 72%). D incorporation by $^1$H NMR: 99%. $^1$H NMR (400 MHz, Chloroform-d) $\delta$ 7.74–7.67 (m, 1H), 7.66–7.57 (m, 2H), 7.49–7.43 (m, 1H), 7.42–
7.35 (m, 2H), 7.34–7.19 (m, 2H), 4.40 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 145.63, 140.05, 129.08, 127.81, 125.15, 119.97. HRMS (EI) Calcd. for C$_{13}$H$_9$OD [M]$^+$: 183.0793, Found: 183.0791.

10,11-dihydro-5H-dibenzo[a,d][7]annulen-5-d$_1$-5-ol (2aa)

![Structure of 10,11-dihydro-5H-dibenzo[a,d][7]annulen-5-d$_1$-5-ol (2aa)](structure.png)

The product is obtained as white solid (isolated yield: 68%). D incorporation by $^1$H NMR: 90%. $^1$H NMR (400 MHz, Chloroform-d) $\delta$ 7.34 (d, $J$ = 6.4 Hz, 2H), 7.07 (dd, $J$ = 16.2, 6.1 Hz, 6H), 5.83 (s, 0.10H), 3.43–3.24 (m, 2H), 3.06–2.90 (m, 2H), 2.25 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 140.46, 138.97, 130.20, 127.98, 127.14, 127.05, 126.19, 76.19 (t, $J$ = 9.09 Hz), 32.42. HRMS (EI) Calcd. for C$_{15}$H$_{13}$OD [M]$^+$: 211.1107, Found: 211.1096.

5H-dibenzo[a,d][7]annulen-5-d$_1$-5-ol (2ab)

![Structure of 5H-dibenzo[a,d][7]annulen-5-d$_1$-5-ol (2ab)](structure.png)

The product is obtained as white solid (isolated yield: 99%). D incorporation by $^1$H NMR: 90%. $^1$H NMR (400 MHz, Chloroform-d) $\delta$ 7.61 (d, $J$ = 7.0 Hz, 2H), 7.41–7.29 (m, 4H), 7.25 (d, $J$ = 7.1 Hz, 2H), 7.05 (s, 2H), 5.33 (s, 0.10H), 2.55 (s, 1H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 140.30, 132.61, 131.15, 130.94, 130.91, 128.68, 128.58, 126.82. HRMS (EI) Calcd. for C$_{15}$H$_{11}$OD [M]$^+$: 209.0951, Found: 209.0941.

phenyl(3,4,5-trifluorophenyl)methan-d$_1$-ol (2ac)

![Structure of phenyl(3,4,5-trifluorophenyl)methan-d$_1$-ol (2ac)](structure.png)

The product is obtained as white solid (isolated yield: 78%). D incorporation by $^1$H NMR: 88%. $^1$H NMR (400 MHz, Chloroform-d) $\delta$ 7.30–7.21 (m, 5H), 6.99–6.85 (m, 2H), 5.62 (s, 0.12H), 2.44 (s, 1H). $^{13}$C NMR (101 MHz, Chloroform-d) $\delta$ 152.37 (dd, $J$ = 10.0, 3.8 Hz), 149.89 (dd, $J$ = 10.0, 3.9 Hz), 142.54, 139.91 (d, $J$ = 5.6 Hz), 137.61 (t, $J$ = 15.5 Hz), 128.91, 128.40, 126.56, 110.47 (d, $J$ = 6.0 Hz), 110.31 (d, $J$ = 5.9 Hz), 74.52 (t, $J$ = 41.93 Hz). $^{19}$F NMR (377 MHz, Chloroform-d) $\delta$ -133.92 (d, $J$ = 20.4 Hz, 2F), -162.16 (t, $J$ = 20.4 Hz, 1F). HRMS (EI) Calcd. for C$_{13}$H$_4$F$_3$OD [M]$^+$: 239.0668, Found: 239.0659.

(perfluorophenyl)(phenyl)methan-d$_1$-ol (2ad)

![Structure of (perfluorophenyl)(phenyl)methan-d$_1$-ol (2ad)](structure.png)

The product is obtained as white solid (isolated yield: 87%). D incorporation by $^1$H NMR: 91%. $^1$H NMR (400 MHz, Chloroform-d) $\delta$ 7.29 (d, $J$ = 5.5 Hz, 4H), 7.25–7.18 (m, 1H), 6.14 (s, 0.09H), 2.77 (s, 1H). $^{13}$C NMR (101 MHz, Chloroform-d) $\delta$ 145.86 (td, $J$ = 3.8 Hz, 3.9 Hz), 143.42 (td, $J$ = 3.8 Hz, 3.8 Hz)
Hz), 140.53, 138.83 (td, $J = 17.7$ Hz, 4.9 Hz), 136.45 (td, $J = 17.6$ Hz, 4.9 Hz), 128.76, 128.28, 125.37, 116.90 (t, $J = 15.0$ Hz), 67.43 (t, $J = 41.9$ Hz). \[ ^{13}C \text{NMR (101 MHz, CDCl}_3) \delta 143.54, 142.05, 137.73, 136.46, 132.41, 131.09, 130.11, 129.73, 128.57, 127.71, 126.52, 75.46 \]

HRMS (EI) Calcd. for C\textsubscript{13}H\textsubscript{16}F\textsubscript{5}OD [M]$: 275.0480, Found: 275.0472.

**phenyl(4-(p-tolylthio)phenyl)methan-d\textsubscript{1}-ol (2ae)**

The product is obtained as white solid (isolated yield: 51%). D incorporation by $^1H$ NMR: 93%. $^1H$ NMR (400 MHz, Chloroform-d) $\delta$ 7.31–7.09 (m, 11H), 7.03 (d, $J = 7.7$ Hz, 2H), 5.67 (s, 0.07H), 2.24 (s, 3H), 2.22 (s, 1H). $^{13}C$ NMR (101 MHz, CDCl\textsubscript{3}) $\delta$ 143.54, 142.05, 137.73, 136.46, 132.41, 131.09, 130.11, 129.73, 128.57, 127.71, 126.52, 75.46 (t, $J = 43.4$ Hz), 21.16. HRMS (EI) Calcd. for C\textsubscript{20}H\textsubscript{17}SOD [M]$: 307.1141, Found: 307.1133.

**isopropyl 2-(4-((4-chlorophenyl)(hydroxy)methyl-d\textsubscript{1})phenoxy)-2-methylpropanoate (2af)**

The product is obtained as white solid (isolated yield: 40%). D incorporation by $^1H$ NMR: 92%. $^1H$ NMR (400 MHz, Chloroform-d) $\delta$ 7.33 (s, 4H), 7.22 (d, $J = 7.6$ Hz, 2H), 6.84 (d, $J = 7.6$ Hz, 2H), 5.79 (s, 0H), 5.10 (p, $J = 6.6$ Hz, 1H), 2.18 (s, 1H), 1.60 (s, 6H), 1.24 (d, $J = 6.3$ Hz, 6H). $^{13}C$ NMR (101 MHz, CDCl\textsubscript{3}) $\delta$ 173.59, 155.26, 142.26, 136.83, 128.52, 127.82, 127.47, 118.95, 79.15, 68.96, 25.42, 25.38, 21.55. HRMS (EI) Calcd. for C\textsubscript{20}H\textsubscript{22}O\textsubscript{4}ClD [M]$: 363.1348, Found: 363.1340.

**d-Orphenadrine**

The product is obtained as yellow liquid (isolated yield: 68%). D incorporation by $^1H$ NMR: 90%. $^1H$ NMR (400 MHz, Chloroform-d) $\delta$ 7.43 (d, $J = 7.0$ Hz, 1H), 7.30 (s, 4H), 7.18 (dt, $J = 14.8$, 6.9 Hz, 3H), 7.10 (d, $J = 6.4$ Hz, 1H), 5.53 (s, 0.1H), 3.56 (s, 2H), 2.68–2.51 (m, 2H), 2.25 (s, 9H). $^{13}C$ NMR (101 MHz, CDCl\textsubscript{3}) $\delta$ 141.18, 139.84, 135.91, 130.53, 128.28, 127.62, 127.43, 127.39, 127.16, 126.01, 80.95 (t, $J = 43.4$ Hz), 67.63, 59.16, 46.11, 19.50. HRMS (ESI) Calcd. for C\textsubscript{18}H\textsubscript{23}NO\textsubscript{2}D [M+H]$^+$: 271.1915, Found: 271.1904.

**d-Neobenodine**
The product is obtained as yellow liquid (isolated yield: 72%). D incorporation by $^1$H NMR: 91%. $^1$H NMR (400 MHz, Chloroform-$d$) $\delta$ 7.31 (dt, $J = 13.1$, 6.6 Hz, 4H), 7.22 (s, 3H), 7.11 (d, $J = 7.2$ Hz, 2H), 5.33 (s, 0.09H), 3.55 (s, 2H), 2.59 (d, $J = 5.5$ Hz, 2H), 2.30 (s, 3H), 2.26 (s, 6H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 142.53, 139.34, 137.01, 129.05, 128.32, 127.28, 127.01, 126.94, 83.43 (t, $J = 43.43$ Hz), 67.54, 59.08, 46.10, 21.14. HRMS (ESI) Calcd. for C$_{18}$H$_{23}$NOD [M+H]$^+$: 271.1915, Found: 271.1904.
11. NMR spectra

2a [{^1}H NMR and ^{13}C NMR in CDCl\textsubscript{3}]}
2b [^1H NMR and ^13C NMR in CDCl₃]
2c [\textsuperscript{1}H NMR and \textsuperscript{13}C NMR in CDCl\textsubscript{3}]
2d [\(^1\)H NMR and \(^{13}\)C NMR in CDCl\(_3\)]
2e [{$^1$H NMR, $^{13}$C NMR and $^{19}$F NMR in CDCl$_3$}]

![NMR Spectra](image_url)

The NMR spectra show the chemical shifts for $^1$H, $^{13}$C, and $^{19}$F in CDCl$_3$ for compound 2e. The spectra provide detailed information about the chemical structure and composition of the compound.
$^2f [^1H \text{NMR}, ^{13}C \text{NMR and } ^{19}F \text{NMR in CDCl}_3]$
$^{1}H$ NMR, $^{13}C$ NMR and $^{19}F$ NMR in CDCl$_3$
2h $^1$H NMR and $^{13}$C NMR in CDCl$_3$
2i \(^{1}H\) NMR and \(^{13}C\) NMR in CDCl\(_3\)
2j [\(^1\)H NMR, \(^{13}\)C NMR and \(^{19}\)F NMR in CDCl\(_3\)]
2k [\textsuperscript{1}H NMR, \textsuperscript{13}C NMR and \textsuperscript{19}F NMR in CDCl\textsubscript{3}]
$^1$H NMR, $^{13}$C NMR and $^{19}$F NMR in CDCl$_3$
2m [^1^H NMR and ^1^C NMR in CDCl₃]
2n [\(^1\)H NMR and \(^{13}\)C NMR in CDCl\(_3\)]
$2o \ [^{1}H \text{ NMR and } ^{13}C \text{ NMR in CDCl}_3]$
$2\text{p} \left[ ^1\text{H NMR and } ^{13}\text{C NMR in CDCl}_3 \right]$
2q $^1$H NMR and $^{13}$C NMR in CDCl$_3$
2r $[^{1}H\text{ NMR and }^{13}C\text{ NMR in CDCl}_3]$
$^{2s}[^1\text{H NMR and } ^{13}\text{C NMR in CDCl}_3]$
$^1$H NMR and $^{13}$C NMR in CDCl$_3$
$^1$H NMR and $^{13}$C NMR in CDCl$_3$
$2v\ [{^1}H\ NMR\ and\ {^{13}}C\ NMR\ in\ CDCl_3]$
2w [¹H NMR and ¹³C NMR in CDCl₃]
2x $[^1\text{H} \text{NMR and } ^{13}\text{C NMR in CDCl}_3]$
2y [¹H NMR and ¹³C NMR in CDCl₃]
2z $[^1]H$ NMR and $^{13}C$ NMR in CDCl$_3$
2aa \[^1\text{H} \text{NMR and } ^{13}\text{C} \text{ NMR in } \text{CDCl}_3\]
2ab \([^1\text{H} \text{NMR and } ^{13}\text{C} \text{NMR in CDCl}_3]\)
2ac [1H NMR, 13C NMR and 19F NMR in CDCl₃]
2ad [^1H NMR, ^13C NMR and ^19F NMR in CDCl₃]
2ae [^1H NMR and ^13C NMR in CDCl₃]
2af [¹H NMR and ¹³C NMR in CDCl₃]
d-Orphenadrine [1H NMR and 13C NMR in CDCl₃]
d-Neobendine [¹H NMR and ¹³C NMR in CDCl₃]
12. Reference