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Pt/C-Catalyzed Deuteration of Polycyclic Aromatic Hydrocarbons with Suppressed Arene Hydrogenation

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ABSTRACT

Deuterated polycyclic aromatic hydrocarbons (PAHs) are indispensable for investigating isotope effects in π-conjugated materials; however, catalytic H–D exchange of readily hydrogenated PAHs is often compromised by competitive ring hydrogenation. This article reports a Pt/C-catalyzed H–D exchange protocol employing inexpensive D_2O as the deuterium source and 2-propanol as the hydrogen donor under an Ar atmosphere. Additive screening identified decalin as a critical reaction modulator, enabling efficient deuterium incorporation while suppressing the formation of partially hydrogenated byproducts. Under the optimized conditions (80°C, 12 h), $\leq$97% deuterium incorporation was achieved for anthracene. Additionally, a range of fused PAHs, including 9-aryl anthracenes, pyrene, triphenylene, and triptycene, were converted to their deuterated analogs in good-to-excellent isolated yields with negligible ring hydrogenation. This operationally simple method provides streamlined access to deuterated PAHs for materials-oriented investigations and isotopic-labeling applications.

1 | Introduction

Polycyclic aromatic hydrocarbons (PAHs) are pivotal π-conjugated scaffolds that are widely employed in organic electronic materials, particularly organic light-emitting diodes (OLEDs) [1–6]. Their rigid aromatic frameworks and extended π-systems promote efficient exciton generation and charge transport. As a representative example, anthracene derivatives have been studied extensively as blue-emitting materials and host components owing to their high fluorescence quantum yields and robust thermal stability [1, 3, 6].

To improve the photophysical and operational properties of organic π-conjugated systems, isotopic substitution is widely recognized as an effective strategy [7–10]. Specifically, replacing C—H bonds with C—D bonds attenuates high-frequency vibrational modes and suppresses nonradiative decay pathways via internal conversion. Both experimental and theoretical studies have demonstrated that deuteration can prolong excited-state lifetimes and enhance photoluminescence efficiencies without substantially perturbing the electronic structure. These effects are particularly beneficial for enhancing the operational stability of OLED materials, especially blue emitters.

Despite these advantages, access to deuterated anthracene derivatives remains limited. Conventional deuteration methods often require harsh reaction conditions or costly deuterium sources, such as D_2 gas or deuterated organic solvents, and can induce partial hydrogenation or degradation of polyaromatic frameworks [11–15]. Accordingly, the development of mild, practical deuteration methods that utilize inexpensive, readily available deuterium sources is essential.

Naoya Sakurada and Sae Tomoto contributed equally to this study.

Heterogeneous platinum catalysts are known to promote hydrogen–deuterium (H–D) exchange in aromatic compounds in the presence of deuterium oxide (D_2O) [16–24]. In such systems, 2-propanol (2-PrOH) can serve as a hydrogen donor, enabling H–D exchange without the requirement for external hydrogen gas [19, 20, 22–24]. This approach enhances safety and operational simplicity, with prior studies underscoring its chemoselectivity, wherein H–D exchange occurs preferentially over the hydrogenation of reducible functionalities such as aromatic carbonyl groups and internal olefins. However, for readily hydrogenated PAHs, achieving high deuterium incorporation while minimizing competing ring hydrogenation remains challenging.

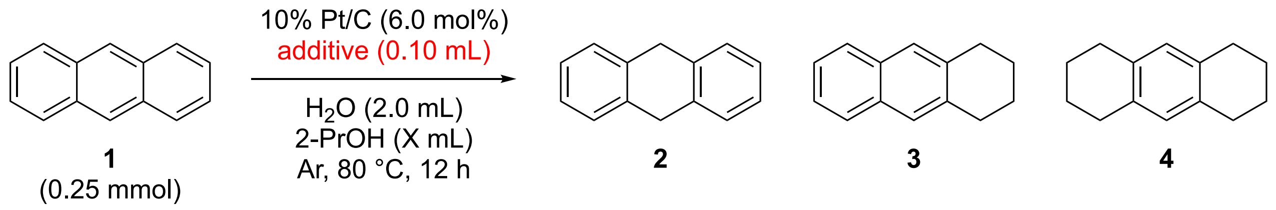
In this study, a practical method is developed for the deuteration of PAHs via Pt/C-catalyzed H–D exchange in a D_2O/2-PrOH system. This protocol employs inexpensive D_2O as the deuterium source under mild conditions to minimize ring hydrogenation of the anthracene core and enable efficient deuterium incorporation across a range of fused PAHs. Decalin is employed as an additive to suppress competing ring hydrogenation without compromising H–D exchange efficiency. Ultimately this work aims to provide streamlined access to a broad range of deuterated PAHs for materials-oriented studies and isotopic-labeling applications.

2 | Results and Discussion

2.1 | Effect of Additives on Suppressing Anthracene Ring Hydrogenation Under Pt/C-Catalyzed Conditions

To establish optimal reaction conditions for suppressing undesired ring hydrogenation during the deuteration of readily hydrogenated PAHs, the influence of different additives on the reaction product distribution was evaluated using anthracene (**1**) as a model substrate (Table 1). Standard conditions included the reaction of **1** (0.25 mmol), 10% Pt/C (6.0 mol%), H_2O (2.0 mL), 2-PrOH (1.0 mL), and the desired additive (0.10 mL) under an Ar atmosphere at 80°C for 12 h. Under these conditions, ring hydrogenation of **1** afforded partially hydrogenated products, namely 9,10-dihydroanthracene (**2**), 1,2,3,4-tetrahydroanthracene (**3**), and 1,2,3,4,5,6,7,8-octahydroanthracene (**4**). In the absence of an additive, partial hydrogenation of the aromatic core was observed: **2** and **3** were formed in 2% and 24% yields, respectively, while a large portion of **1** (74%) remained unreacted, as determined by a proton nuclear magnetic resonance (^{1}H NMR) ratio of 74:2:24:0 (**1**/**2**/**3**/**4**; entry 1). Screening of representative additives revealed additive-dependent suppression of hydrogenation. Such sensitivity of PAH hydrogenation to the reaction medium is consistent with previous reports showing that the product distribution in PAH hydrogenation depends strongly on the catalyst and solvent, and that hydrogenation on Pt surfaces can be significantly influenced by the surrounding liquid environment [25, 26]. Specifically, 1,2-dichloroethane hindered the formation of hydrogenated products, giving a product ratio of 93:1:6:0 (entry 2). In contrast, chlorocyclohexane completely inhibited the formation of hydrogenated byproducts, and only **1** was detected by ^{1}H NMR spectroscopy (100:0:0:0; entry 3). Cyclohexane exhibited the opposite trend, promoting ring hydrogenation to afford a product ratio of 43:6:51:0.4 (entry 4). Decalin also suppressed ring hydrogenation, affording a product ratio of 91:1:8:0 (entry 5). This result is notable in light of previous reports showing that decalin is not always an inert medium under supported noble-metal-catalyzed hydrogenation conditions and that interactions between the liquid medium and the catalyst surface can affect hydrogenation behavior [27, 28]. Further optimization in the presence of decalin demonstrated that the amount of added 2-PrOH strongly influenced the product distribution: upon reducing the added 2-PrOH volume to 0.10 mL, no hydrogenated products were detected, with quantitative recovery of **1** being reported (100:0:0:0; entry 6). Collectively, these results indicate that certain additives effectively minimize undesired ring hydrogenation and that the extent of hydrogenation can be controlled by tuning the amount of 2-PrOH under otherwise identical conditions.

TABLE 1 | Effects of additives in suppressing anthracene ring hydrogenation under Pt/C-catalyzed conditions using 2-PrOH.



Entry	Additive	2-PrOH (X mL)	Product ratio 1/2/3/4[a]
1	none	1.0	74:2:24:0
2	1,2-dichloroethane	1.0	93:1:6:0
3	chlorocyclohexane	1.0	100:0:0:0
4	cyclohexane	1.0	43:6:51:0.4
5	decalin	1.0	91:1:8:0
6[b]	decalin	0.10	100:0:0:0

[a] The ratio of products **1**, **2**, **3**, and **4** was determined by ^{1}H NMR spectroscopy.
[b] Isolated yield of **1** = 97%.

2.2 | Effects of Additives on the Deuteration of Anthracene

The influence of different additives on the deuteration of **1** was subsequently investigated (Table 2). For this purpose, standard conditions included the reaction of **1** (0.25 mmol), 10% Pt/C (6.0 mol%), D_2O (2.0 mL, ca. 110 mmol; 440 equiv relative to **1**, corresponding to 44 equiv per potentially exchangeable C—H), 2-PrOH (0.10 mL), and an additive (0.90 mL) under an Ar atmosphere at 80°C for 12 h. Deuterated anthracene (**1-*d*$_n$**) was obtained under these conditions, and both the deuterated sites and degree of incorporation were determined by ^{1}H NMR analysis. In the absence of an additive, deuterium incorporation was modest [23% at H_a (C9), 34% at H_b (C1), and 36% at H_c (C2)], and **1-*d*$_{10}$** was isolated in 82% yield; concomitant ring hydrogenation was also observed (entry 1). Using a reduced amount of decalin (0.10 mL), deuterium incorporation remained limited (21% at H_a, 54% at H_b, and 69% at H_c; entry 2). In contrast, upon the addition of 0.90 mL decalin, enhanced deuterium incorporation was achieved at H_b and H_c (94% and 97%, respectively), whereas H_a showed lower incorporation (39%; entry 3). In comparison, cyclohexane led to decreased incorporation at all positions (9% at H_a, 46% at H_b, and 77% at H_c; entry 4), and the use of 1,2-dichloroethane further reduced deuterium incorporation (3% at H_a, 12% at H_b, and 50% at H_c; entry 5). Chlorocyclohexane, which effectively suppressed ring hydrogenation under H_2O conditions, afforded only minimal deuteration (6%, 7%, and 8% at H_a, H_b, and H_c, respectively; entry 6). Taken together, these results indicate that the additive plays a critical role not only in suppressing undesired hydrogenation but also in facilitating efficient H–D exchange. Consequently, decalin was identified as the most suitable additive for achieving high deuterium incorporation in anthracene.

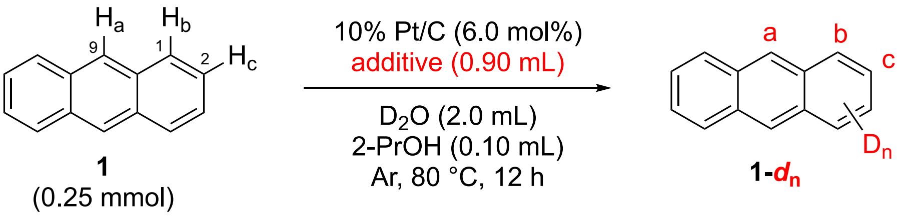
To enhance deuterium incorporation at the less reactive H_a site under decalin conditions, the reaction parameters were further optimized. For instance, increasing the catalyst loading to 10 mol% raised deuterium incorporation at H_a to 75% while maintaining high incorporation at both H_b and H_c (75% at H_a and 97% at H_b and H_c; entry 7). Under these optimized conditions, **1-*d*$_n$** was isolated in 90% yield. In contrast, the exclusion of 2-PrOH completely suppressed deuteration (0% at H_a, 0% at H_b, and 0% at H_c; entry 8). However, variations in temperature or reaction time did not lead to any meaningful improvement. Detailed data for these additional studies are provided in the Supporting Information.

Taken together, these results suggest that the additives influence not only suppression of undesired hydrogenation but also the accessibility of exchange-active catalytic species. In particular, 2-PrOH appears to be essential for H–D exchange, whereas decalin provides a reaction environment that favors deuteration over ring hydrogenation. In contrast, 1,2-dichloroethane and chlorocyclohexane suppress both hydrogenation and deuteration, suggesting broader inhibition of catalyst-surface reactivity rather than selective promotion of H–D exchange.

2.3 | Substrate Scope of the Deuteration of PAHs Under Pt/C-Catalyzed Conditions in the Presence of Decalin

With the optimized conditions in hand, the substrate scope of the Pt/C-catalyzed H–D exchange of PAHs was evaluated in a D_2O/2–PrOH system (Table 3). Anthracene derivatives bearing aryl substituents, including 9-(1-naphthyl)anthracene (**2**) and

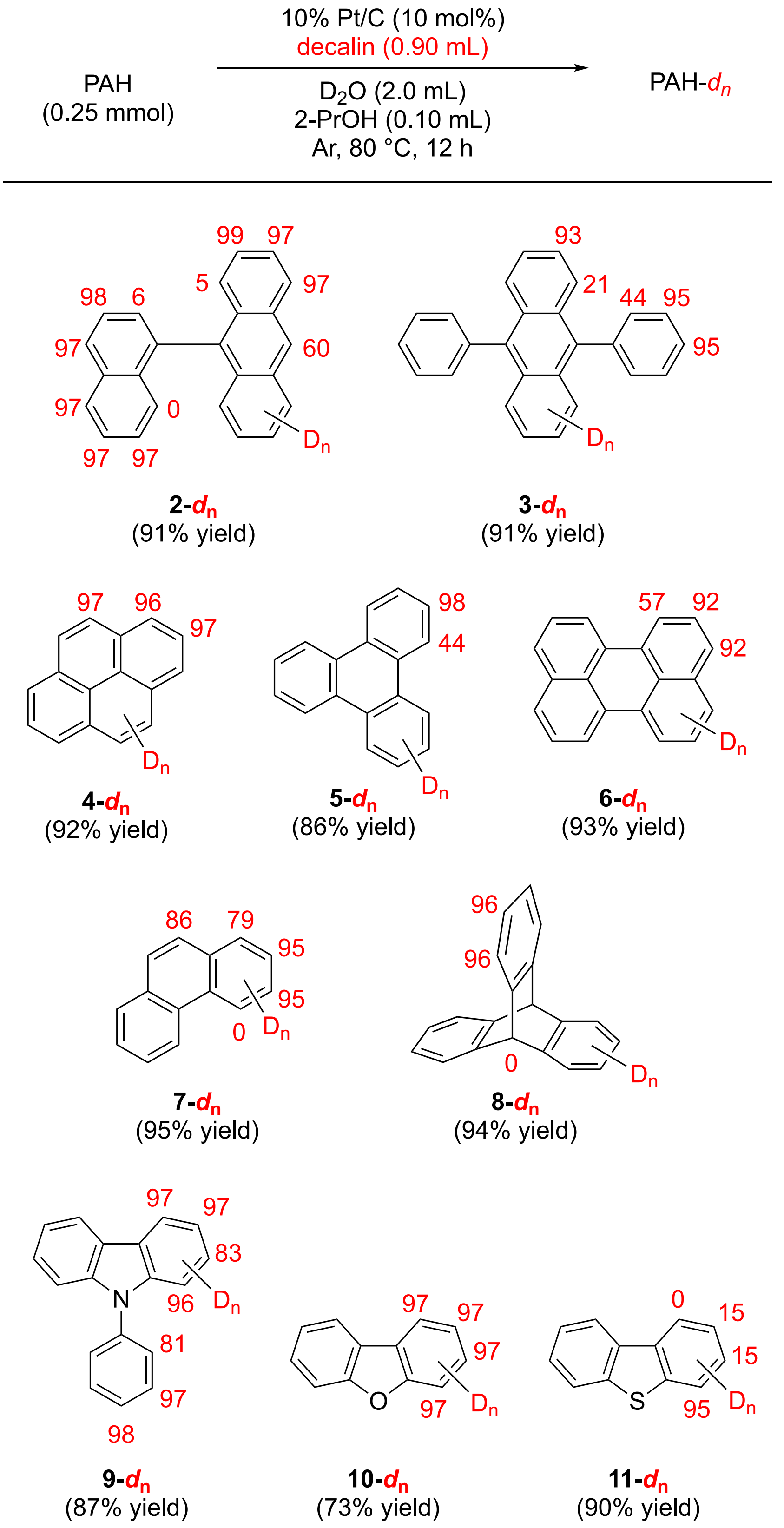
TABLE 2 | Effects of additives on anthracene deuteration under Pt/C-catalyzed conditions.



Entry	Additive (variation from standard conditions)	D content, %[a]		
		H_a	H_b	H_c
1[b]	None	23	34	36
2[c]	Decalin (0.10 mL)	16	51	67
3	Decalin	30	94	97
4	Cyclohexane	9	46	77
5	1,2-dichloroethane	3	12	50
6	Chlorocyclohexane	6	7	8
7[d,e]	Decalin (10 mol% Pt)	74	97	97
8[f]	Decalin (without 2-PrOH)	0	0	0

[a] Deuterium content (%) was determined by ^{1}H NMR spectroscopy using 0.25 mmol 1,1,2,2-tetrachloroethane as the internal standard.
[b] Isolated yield of **1-*d*$_n$** = 82%.
[c] 0.10 mL decalin was used.
[d] 10 mol% 10% Pt/C was used.
[e] Isolated yield of **1-*d*$_n$** = 90%.
[f] Excluding 2-PrOH.

TABLE 3 | Substrate scope for the deuteration of PAHs under Pd/C-catalyzed conditions.[a]



[a] Deuterium content (%) was determined by ^{1}H NMR spectroscopy using 0.25 mmol 1,1,2,2-tetrachloroethane as the internal standard.

9,10-diphenylanthracene (**3**), were converted to the corresponding deuterated analogs **2-*d*$_n$** and **3-*d*$_n$** in isolated yields of 91% and 91%, respectively, with negligible ring hydrogenation under the optimized conditions. However, deuterium incorporation at sterically hindered positions was less efficient. Notably, the naphthylanthracene motif is widely incorporated in high-triplet-energy blue-emitting and host-type molecular designs for OLED related materials chemistry [29]. Additionally, 9,10-diphenylanthracene is frequently employed as a fluorescence quantum-yield reference standard across different solvents, highlighting the utility of accessing its deuterated isotopologues for photophysical studies [30].

Beyond anthracene derivatives, efficient deuteration was also achieved for a range of representative PAHs. For instance, pyrene (**4**) furnished **4-*d*$_n$** in 92% yield, underscoring compatibility with pyrene-based π-cores, which are widely employed in organic electronic materials [4]. Additionally, triphenylene (**5**), a canonical discotic framework associated with columnar π-stacking and anisotropic charge transport [31], afforded **5-*d*$_n$** in 86% yield. Furthermore, perylene (**6**), a strongly absorbing and emissive chromophore platform [32], provided **6-*d*$_n$** in 93% yield, while phenanthrene (**7**) gave the corresponding deuterated product **7-*d*$_n$** in 95% yield. Finally, triptycene (**8**) was smoothly converted to **8-*d*$_n$** in 94% yield, with no detectable benzylic deuteration, thereby providing access to a rigid three-dimensional aromatic scaffold that is often used to modulate molecular packing in functional materials [33]. In addition, the present method was applicable to heteroaromatic fused systems. *N*-Phenylcarbazole (**9**) afforded **9-*d*$_n$** in 87% yield with high deuterium incorporation across the molecular framework. Dibenzofuran (**10**) likewise underwent efficient deuteration to furnish **10-*d*$_n$** in 73% yield. In contrast, dibenzothiophene (**11**) gave **11-*d*$_n$** in 90% yield, but high deuterium incorporation was observed mainly at the positions adjacent to sulfur (95%), whereas the other aromatic positions showed only low incorporation (0%–15%). These results further expand the scope of the present method to heteroaromatic PAH analogs and suggest that the deuteration pattern can be influenced by the embedded heteroatom. Heteroaromatic fused motifs such as carbazole, dibenzofuran, and dibenzothiophene are widely found in OLED emitter and host design, which further highlights the potential utility of the present deuteration protocol for isotopologue access in functional materials chemistry [34–36].

3 | Conclusion

In summary, a practical Pt/C-catalyzed H–D exchange protocol was developed for the deuteration of PAHs. High levels of deuterium incorporation were achieved while minimizing undesired partial hydrogenation of the aromatic core under the optimized D_2O/2-PrOH/decalin conditions. The role of decalin in balancing H–D exchange and hydrogenation pathways was supported by optimization studies and control experiments. Notably, this method enabled efficient deuteration across a range of PAHs under mild conditions, providing the corresponding deuterated products in good-to-excellent isolated yields with no detectable ring hydrogenation in most cases. These results should facilitate access to deuterated PAHs for materials-oriented investigations and isotopic-labeling applications, including systematic evaluation of isotope effects on photophysical properties and device stability.

4 | Experimental Section

4.1 | Materials and Methods

4.1.1 | Reagents

Deuterium oxide (D_2O, >99.9 atom% D) was purchased from Silantes Inc. (Germany). The 10% Pt/C catalyst was obtained from N.E. CHEMCAT CORPORATION and used as received as a dry-type material. The stated Pt loading (10 wt%) is based on the dry catalyst, according to the supplier specification. 2-Propanol (2-PrOH, super dehydrated grade, H_2O $\leq$10 ppm, packed under inert gas) and decalin (decahydronaphthalene, mixture of *cis*- and *trans*-isomers, Wako 1st Grade) were purchased from FUJIFILM Wako Pure Chemical Corporation. Unless otherwise stated, all substrates and solvents were purchased from commercial suppliers and used without further purification. Flash column chromatography was performed on Silica Gel 60 N (Kanto Chemical Corporation., Inc., 63–210 µm spherical, neutral). Gel permeation chromatography (GPC) was performed using a LaboACE LC-5060 Plus II system (Japan Analytical Industry Co., Ltd.) when additional purification was required. All reactions were monitored by thin-layer chromatography (TLC) on glass-backed silica gel 60 F^{254} (0.2 mm plates, Merck), and compounds were visualized using *p*-anisaldehyde solution as a TLC stain.

4.1.2 | Analytical Methods

Optical rotation measurements were performed using an ATAGO AP300 automatic polarimeter. The ^1H and ^2H NMR spectra were recorded on a JEOL ECZ 400 (^1H: 400 MHz, ^2H: 61 MHz) or ECA 500 spectrometer (^1H: 500 MHz, ^2H: 77 MHz) at room temperature in $CDCl_3$ or D_2O as the solvent and internal standard (^1H NMR: $\delta = 7.26$ ppm for $CHCl_3$; ^2H NMR: $\delta = 7.26$ ppm for $CDCl_3$, 4.79 ppm for D_2O). The ^1H NMR spectra recorded for the known compounds were identical to those reported in the literature. The degree of deuterium incorporation was determined by integration of the corresponding ^1H NMR signals, using 1,1,2,2-tetrachloroethane as the internal standard. For each substrate, the residual proton signals at the target positions were integrated relative to the internal standard, and the degree of deuterium incorporation was calculated by comparison with the corresponding nondeuterated material. The extent of deuterium incorporation and the labeling positions were confirmed by ^2H NMR spectroscopy.

4.2 | General Procedure

4.2.1 | Condition A (Table 1)

An oven-dried Schlenk tube equipped with a magnetic stir bar was charged with anthracene **1** (0.25 mmol, 1.0 equiv) and 10% Pt/C (6.0 mol% Pt). The tube was then evacuated and back-filled with argon. Using a syringe, H_2O (2.0 mL), 2-PrOH

(1.0 mL), and the desired additive (0.10 mL) were added to the tube under an argon atmosphere. The Schlenk tube was sealed, and the mixture was stirred at 80°C (oil bath) under argon for the indicated time (e.g., until the product distribution became constant). After subsequent cooling to room temperature, the reaction mixture was filtered through a membrane filter (pore size: 0.45 μm). The catalyst on the filter was washed with chloroform (3×5 mL). The solvent was then removed under reduced pressure, and the crude residue was analyzed by ^{1}H NMR spectroscopy to determine the product ratio (**1**/**2**/**3**/**4**; anthracene/9,10-dihydroanthracene/1,2,3,4-tetrahydroanthracene/1,2,3,4,5,6,7,8-octahydroanthracene).

4.2.2 | Condition B (Table 2)

An oven-dried Schlenk tube equipped with a magnetic stir bar was charged with anthracene **1** (0.25 mmol, 1.0 equiv) and 10% Pt/C (6.0 mol% Pt, or the indicated loading). The tube was then evacuated and back-filled with argon. Using a syringe, D_2O (2.0 mL), 2-PrOH (0.10 mL), and the desired additive (0.90 mL) were added to the tube under an argon atmosphere. The Schlenk tube was sealed, and the mixture was stirred at 80°C for 12 h (oil bath) under argon. After subsequent cooling to room temperature, the reaction mixture was filtered through a membrane filter (pore size: 0.45 μm). The catalyst on the filter was washed with chloroform (3×5 mL). The solvent was then removed under reduced pressure, and the crude residue was analyzed by ^{1}H NMR spectroscopy to determine the deuterated sites and degree of deuterium incorporation at positions a–c using 1,1,2,2-tetrachloroethane as the internal standard. To afford deuterated anthracene (**1-d_n**), the crude product was purified by column chromatography on silica gel (ethyl acetate/hexane = 1:30) and gel permeation chromatography (GPC) using a JAI LaboACE LC-5060 Plus II system.

4.2.3 | Condition C (Table 3)

An oven-dried Schlenk tube equipped with a magnetic stir bar was charged with the PAH substrate (0.25 mmol, 1.0 equiv) and 10% Pt/C (10 mol% Pt). The tube was then evacuated and back-filled with argon. Using a syringe, D_2O (2.0 mL), 2-PrOH (0.10 mL), and decalin (0.90 mL) were added to the tube under an argon atmosphere. The Schlenk tube was sealed, and the mixture was stirred at 80°C for 12 h (oil bath) under an argon atmosphere. After subsequent cooling to room temperature, the reaction mixture was filtered through a membrane filter (pore size: 0.45 μm). The catalyst on the filter was washed with chloroform (3×5 mL). The solvent was then removed under reduced pressure, and the crude residue was purified by column chromatography on silica gel (ethyl acetate/hexane = 1:30) to afford the corresponding deuterated PAH. For **2-d_n** and **3-d_n**, the products obtained after silica gel column chromatography were further purified by gel permeation chromatography (GPC) using a JAI LaboACE LC-5060 Plus II system.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material for this article.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.