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### Reversible Hydrogenation–Oxidative Dehydrogenation of Quinolines over a Highly Active Pt Nanowire Catalyst under Mild Conditions

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Catalytic hydrogenation (reduction) and dehydrogenation (oxidation) reactions are of fundamental importance in the synthesis of organic molecules.<sup>[1]</sup> The hydrogenation of aldehydes/ketones and imines yields the corresponding alcohols and amines. In the reverse direction, dehydrogenation of alcohols and amines forms the corresponding aldehydes/ketones and imines. The catalytic hydrogenations and dehydrogenations of N-heterocycles are particularly important transformations, as both saturated and unsaturated heterocycles are important structural units in natural products and pharmaceuticals.<sup>[2]</sup>

Previous methods for the hydrogenation of N-heterocycles have been catalyzed with homogeneous precious-metal catalysts, such as Ir,<sup>[3]</sup> Ru,<sup>[4]</sup> Rh,<sup>[5]</sup> and Mo.<sup>[6]</sup> Those catalytic systems all exhibit high activity and selectivity, but separation of the catalyst from the reaction mixture is guite difficult, and amines in particular suffer from contamination by the heavy-metal ions. Heterogeneous catalysis has also been used for the hydrogenation of N-heterocycles. Liao and Shi used Pd nanoparticles supported on tannin-grafted collagen fibers as a recyclable catalyst for the hydrogenation of guinoline.<sup>[7]</sup> This catalyst showed excellent catalytic activity at an initial hydrogen pressure of 2 MPa. Bianchini found that silica-supported Pd nanoparticles could catalyze the hydrogenation of 1,10-phenanthroline at 30 bar (1 bar = 100 kPa) of hydrogen.<sup>[8]</sup> Raney-Ni can also be used as a catalyst for the hydrogenation of phenanthrolines with good to excellent yields under 27 bar hydrogen pressure.<sup>[9]</sup> As encouraging as these results are, the process is not suitable for industrial production because of the high working pressure. It is also energy intensive and suffers from some safety issues. Therefore, the development of a heterogeneous metal catalyst with superior catalytic activity and reusability is a very attractive research target. Kobayashi reported that sub-nanometer-sized Pd clusters stabilized by random copolymers act as catalysts for the hydrogenation of quinoline to 1.2.3.4-terahydroguinoline under mild reaction conditions.<sup>[10]</sup> Recently, Au/TiO<sub>2</sub>,<sup>[11]</sup> Rh/AIO(OH),<sup>[12]</sup> and heterogeneous multi-

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metallic nanoparticle catalysts (72 catalysts based on Rh, Pt, Ir, and Ru) supported on metal oxides<sup>[13]</sup> were also screened. They also need high hydrogen pressure (more than 8 bar). Pd@OmpG-C<sub>3</sub>N<sub>4</sub><sup>[14]</sup> (OmpG=outer membrane protein G) is the best catalyst that has been reported for the hydrogenation of quinolones, and it shows high activity and selectivity under mild reaction conditions (1 bar hydrogen and 40-100 °C). Platinum oxide (PtO<sub>2</sub>) can also be used in the hydrogenation of N-heterocycles in good yields.<sup>[15]</sup> However, the utilization of Pt-based catalysts has been less investigated, even though Pt is regarded as one of the best metal catalysts for hydrogen activation. Kobayashi successfully applied polymer-incarcerated Pt nanoparticles (<3 nm) to the catalytic hydrogenation of unsaturated compounds including heterocycles in good yields under mild reaction conditions [5 atm of hydrogen (1 atm = 101.3 kPa)].<sup>[16]</sup> Somorjai and Yang demonstrated that the structure of the nanomaterials can affect the selectivity of the reactions: Pt nanocubes were used as a catalyst for the hydrogenation of pyrrole, and they showed selectivity towards n-butylamine that was higher than that shown by nanopolyhedra.<sup>[17]</sup> These reported Pt-based catalysts are highly active and selective, which encouraged us to explore Pt-based heterogeneous catalysts in the hydrogenation of N-heterocycles.

The direct oxidation of saturated N-heterocycles is an equally important organic transformation, as the structure unit can be found in many biologically active natural products and pharmacologically active molecules.<sup>[2]</sup> Pd/C as a commercial catalyst proved to be effective in the oxidation of 1,2,3,4-tetrahydroquinolines to quinolines under mild reaction conditions.<sup>[18]</sup> Ru-based catalysts can also be used in the oxidation of N-heterocycles to provide the products in good to excellent yields.<sup>[19]</sup> Pt has proved to be one of the best catalysts for oxidation<sup>[20]</sup> and has also been used as a heterogeneous catalyst in the oxidation of saturated N-heterocycles. Kaneda et al.<sup>[21]</sup> found that supported Cu nanoparticles can be used in the dehydrogenation of 1,2,3,4-tetrahydroquinoline at 150  $^\circ\text{C}$  with the evolution of hydrogen gas. This is good for hydrogen transformations with organic molecules. This catalyst also shows excellent catalytic activity and selectivity for the hydrogenation of quinoline, and it is the first catalytic system that can be used in the hydrogenation of quinoline and in the dehydrogenation of hydrogenated quinoline. However, oxidative dehydrogenation is also an important transformation of organic molecules, and to the best of our knowledge, no catalytic system has been reported that can be used as an effective cat-

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alyst for the reversible hydrogenation and oxidative dehydrogenation of N-heterocycles.

In our previous work, we demonstrated that Pt nanowire (NW) shows high catalytic activity and selectivity towards the hydrogenation of unsaturated compounds under mild reaction conditions.<sup>[22]</sup> The NW can be used multiple times with no detected Pt ion leakage. In this paper, we report a heterogeneous catalytic system that is equally effective in the hydrogenation and oxidative dehydrogenation of guinolines by using an ultrathin Pt NW catalyst. In addition, the remarkable stability of the Pt NW catalyst without a supporter and the exceptionally high catalytic activity fulfill all the requirements for catalysis under mild reaction conditions, which is in sharp contrast to most and supported nanoparticle catalysts homogeneous metal catalysts.

To test the feasibility of our concept, the ultrathin Pt NW catalyst was synthesized by acidic etching of FePt NW in air followed by multiple washings in methanol.<sup>[23]</sup> The NWs are several hundreds of nanometers in length and have an average diameter of about 1.5 nm with a very narrow size distribution (Figure S1, Supporting Information). The predominant plane is (111) from the XRD pattern (Figure S2, Supporting Information), and all Fe was removed, as confirmed by inductively coupled plasma mass spectrometry (ICPMS).

To investigate the catalytic activity of this Pt NW catalyst for the partial hydrogenation of quinolines, quinoline was selected as the model reaction substrate to optimize the reaction conditions. The partial hydrogenation of quinoline (1) generated two main products: 1,2,3,4-tetrahydroquinoline (2) and 5,6,7,8tetrahydroquinoline (3, Scheme 1). Table 1 shows the partial hydrogenation results of quinoline under different reaction conditions. Water was first used as the solvent under an initial hydrogen pressure of 1 bar. Compound 2 was obtained in 92.1% yield after 24 h with a selectivity of 94.1% (Table 1,



Scheme 1. Partial hydrogenation of quinoline (1) to 1,2,3,4-tetrahydroquinoline (2) or 5,6,7,8-tetrahydroquinoline (3).

Entry	Catalyst	Solvent	Conversion <sup>[b]</sup> [%]	Selectivity for <b>2</b> <sup>[b]</sup> [%]			
1	Pt NW	H₂O	97.9	94.1			
2	Pt NW	CH₃OH	91.3	97.1			
3	Pt NW	$C_2H_5OH$	95.5	93.1			
4 <sup>[c]</sup>	Pt NW	CHCl <sub>3</sub>	60.4	82.2			
5 <sup>[c]</sup>	Pt NW	THF	75.1	98.0			
6	Pt NP	H <sub>2</sub> O	58.8	51.8			
7	Pt NR	H <sub>2</sub> O	97.7	75.4			
8	Pd NW	H₂O	18.8	94.1			
[a] Quinoline (1 mmol), solvent (3 mL), and $H_2$ (1 bar) with the catalyst (0.005 mmol) at 80 °C for 24 h. [b] Determined by GC and GC–MS. [c] Reaction performed at 40 °C.							

entry 1). Alcohols (methanol or ethanol) could also be used as the solvent in this reaction (Table 1, entries 2 and 3). Of all the solvents we used, water was the most suitable for this reaction, and it is green and effectively free. Without a catalyst or hydrogen, no hydrogenated products were observed. We also used Pt nanoparticle (NP), Pt nanorod (NR), and Pd NW catalysts (Figure S3, Supporting Information) to compare their catalytic activities with that of our Pt NW. The Pt NW showed the highest catalytic activity and selectivity of these catalysts (Table 1, entries 6–8).

Vierhapper reported that in neutral or weakly acidic media, the reaction is selective towards **2**, and in strong acid **3** is preferred.<sup>[24]</sup> Five additives were examined in this catalytic system to detect the reaction activity of the Pt NW catalyst (Table 2). All reactions continued to give high selectivity for **2**. If HCl or

Table 2. Hydrogenation of quinoline with different additives. <sup>[a]</sup>								
Entry	Additive	Conversion <sup>[b]</sup> [%]	Selectivity for $2^{[b]}$ [%]					
1	CH₃COOH	97.9	92.8					
2	HCI	59.9	98.0					
3	$H_2SO_4$	74.4	97.9					
4	H₃PO₄	97.8	94.2					
5	$NH_3 \cdot H_2O$	80.3	96.8					

[a] Quinoline (1 mmol), additive (1 mmol), water (3 mL), and H<sub>2</sub> (1 bar) with the catalyst (0.005 mmol) at 80  $^\circ$ C for 24 h. [b] Determined by GC and GC–MS.

 $H_2SO_4$  (1 equiv.) was added, the conversion decreased to less than 75%, which shows that strong acids decrease the hydrogenation of quinoline under these conditions. If acetic acid or  $H_3PO_4$  was added, the conversion remained around 97%. If ammonia was added, the reaction activity also decreased. None of these additives increased the catalytic activity of this reaction, but the selectivity remained constant. These results confirm that Pt NW is an excellent catalyst for the catalytic partial hydrogenation of 1 to 2.

The Pt NW catalyst can be conveniently recycled and reused. Upon completion of the reaction, the Pt NW can be separated by centrifugation after stirring is stopped, and the catalyst can be reused after washing with methanol and hydrochloric acid (0.1 m). The conversion and selectivity were constant over 10 cycles (Figure 1) and no leached Pt was detected by atomic absorption spectroscopy (AAS) and ICPMS.

This catalyst can also be used in the selective oxidation of amines, which is an equally important organic transformation. When the hydrogenation was finished, **2** could be dehydrogenated back to quinoline with the Pt NW catalyst if the atmosphere was changed to oxygen or air (Scheme 2). The yield was above 99% for the dehydrogenation reaction under mild reaction conditions (40 °C, 1 bar). These results demonstrate that Pt NW can be used as an effective catalyst for the hydrogenation of quinoline and for the oxidative dehydrogenation of 1,2,3,4-tetrahydroquinoline.

The general applicability of this catalytic hydrogenation and oxidative dehydrogenation was tested with a series of

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Figure 1. Catalytic stability of the Pt NW catalyst in the hydrogenation of quinoline.

N-heterocyclic compounds. For the hydrogenations, the reaction was remarkably selective towards the pyridine ring without affecting the fused benzene rings. Table 3 shows the results of the hydrogenation and oxidative dehydrogenation of N-heterocycles. The Pt NW showed high catalytic activity and selectivity in all the hydrogenations of substituted quinolines and the corresponding oxidative dehydrogenations of the products (Table 3, entries 1–9).

Direct hydrogenation of acridine was also achieved. Although the yield of the hydrogenation process was low, 9,10-dihydroacridine could be oxidized to acridine easily under the oxidative conditions (Table 3, entry 10). Under the standard reaction conditions, 1,10-phenanthroline was hydrogenated to 1,2,3,4-tetrahydrophenanthroline in 80.4% yield, and the yield of the oxidative dehydrogenation was 100% (Table 3, entry 11).

If pyridine or a substituted pyridine was used as the substrate, only trace amounts of the products were obtained under the mild reaction conditions. Increasing the initial hydrogen pressure to 10 bar still resulted in low yields (< 50%; Table S1, Supporting Information). This structure-sensitive activity can be explained by the molecular adsorption of the catalyst onto the surface. Quinoline, one of the more basic N-heterocycles, can adsorb onto the surface of a heterogeneous catalyst though two different modes: end-on through the nitrogen atom and side-on through the aromatic ring.<sup>[25]</sup> Lambert has shown that quinoline preferentially adsorbs in the side-on configuration through the aromatic  $\pi$  system onto the Pt (111) surface plane.<sup>[25b]</sup> This adsorption configuration is good for the hydrogenation of quinoline and its derivatives by using active hydrogen on the surface of the Pt NW. However, the adsorption of pyridine is both coverage and temperature dependent. At the reaction temperature (80 °C), pyridine adopts an upright



hydrogenation yield: 92.1% dehydrogenation yield: >99%



[a] Hydrogenation (1 to 2): compound 1 (1 mmol), CH<sub>3</sub>OH (3 mL), and H<sub>2</sub> (1 bar) with the Pt NW catalyst (0.005 mmol) at 40 °C; oxidative dehydrogenation (2 to 1): compound 2 (1 mmol), CH<sub>3</sub>OH (3 mL), and O<sub>2</sub> (1 bar) with the Pt NW catalyst (0.005 mmol) at 40 °C. [b] Determined by GC and GC–MS. The value in parentheses is the yield of the isolated product. [c] Initial pressure of hydrogen = 10 bar.

configuration and covers the Pt (111) surface plane,<sup>[26]</sup> which is not suitable for hydrogenation.

In summary, we have developed a simple and highly efficient method for the reversible hydrogenation–oxidative dehydrogenation of N-heterocycles by using Pt NW as the catalyst under mild reaction conditions. Pt NW shows high selectivity towards the hydrogenation of N-heterocycles, and the hydro-

> genation products can be easily oxidized under the same conditions with oxygen or air. This method avoids high temperatures and pressures, and the catalyst can be recycled easily. We are convinced that this procedure is an appealing option in organic synthesis.

**Scheme 2.** Reversible hydrogenation and oxidative dehydrogenation of quinoline and 1,2,3,4-tetrahydroquinoline by using Pt NW as the catalyst.

### **Experimental Section**

#### General

All reactions were heated under an atmosphere of oxygen or hydrogen at a certain temperature with magnetic stirring. Unless otherwise noted, all materials were obtained from commercial suppliers and were used without further purification. All solvents were reagent grade. Upon completion of the reaction, the hydrogen atmosphere was removed, and the resultant product mixtures were analyzed by GC (Varian CP-3800 GC, HP-5 capillary column, FID detector) and GC–MS (Varian 450-GC & Varian 240-GC) equipped with a CP8944 capillary column (30 m×0.25 mm) and an FID detector. The NMR spectra were measured on a spectrometer at 400 MHz for <sup>1</sup>H nuclei and at 100 MHz for <sup>13</sup>C nuclei in CDCl<sub>3</sub> solution.

## General procedure for the catalytic hydrogenation of N-heterocycles

Pt NWs in methanol (0.005 mmol), quinoline or its derivatives (1 mmol), and solvent (2 mL) were added to a Schlenk tube, and the tube was then sealed. The reaction tube was evacuated (3 times) and flushed with hydrogen, which took place at a certain temperature under a hydrogen atmosphere. Upon completion of the reaction, the hydrogen atmosphere was removed, and the resultant product mixtures were analyzed by GC and GC–MS. 1,2,3,4-Tetrahydroquinoline and its derivatives were characterized by <sup>1</sup>H NMR and <sup>13</sup>C NMR spectroscopy.

# General procedure for the oxidative dehydrogenation of saturated N-heterocycles

The reaction mixture or saturated N-heterocycle (1 mmol) was heated under an atmosphere of oxygen or in air at a certain reaction temperature. Upon completion of the reaction, the oxygen atmosphere was removed, and the resultant product mixtures were analyzed by GC and GC–MS.

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**Keywords:** hydrogenation · nanocatalyst · oxidative dehydrogenation · platinum · reversible reactions

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## COMMUNICATIONS

**Over the wire and back again:** The reversible hydrogenation–oxidative dehydrogenation of quinolines is reported by using Pt nanowire (NW) as the catalyst under mild reaction conditions. Pt NW shows high activity and selectivity in the hydrogenation of quinolines under H<sub>2</sub> pressure (1 bar = 100 kPa), and these hydrogenation products can be easily oxidized under the same conditions in an atmosphere of oxygen (1 bar) or in air.



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Reversible Hydrogenation–Oxidative Dehydrogenation of Quinolines over a Highly Active Pt Nanowire Catalyst under Mild Conditions