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FULL PAPER

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A novel Pt/C-catalyzed transfer hydrogenation reaction of *p*-benzoquinone to produce *p*-hydroquinone using cyclohexanone as an unexpectedly effective hydrogen source

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A new method for the production of *p*-hydroquinone via a Pt/C-catalyzed reduction of *p*-benzoquinone is developed. Different from the conventional transfer hydrogenation reactions that usually use secondary alcohols such as isopropanol as the hydrogen source, in this work, it is unexpectedly found that cyclohexanone is a more effective hydrogen source than secondary alcohols, even cyclohexanol. This reaction affords acceptable yields of *p*-hydroquinone with very high turnover number (1109) of the Pt/C catalyst. A mechanism of this interesting reaction is proposed on the basis of the results of a series of control experiments, GC–MS analysis as well as dynamic studies.

KEYWORDS
cyclohexanone, hydroquinone, *p*-benzoquinone, platinum, transfer hydrogenation

1 | INTRODUCTION

Hydroquinone (HQ) is a significant industrial intermediate being abundantly and extensively used in the preparation of electrode materials, black-and-white developing agents, anthraquinone and azo dyes, rubber antioxidants, stabilizers, etc.[1] In industry, it is conventionally produced by reduction of *p*-benzoquinone (BQ) with iron powder or gaseous hydrogen as the reductant.[2] However, the former method inevitably leads to the generation of large amounts of iron mud, a solid waste of which it is difficult to dispose; whereas the latter method suffers from potential dangers due to the use of flammable and explosive gaseous hydrogen. Therefore, new methods that can avoid the generation of solid waste or those can be performed under much safer conditions are of great value from the industrial production viewpoint.

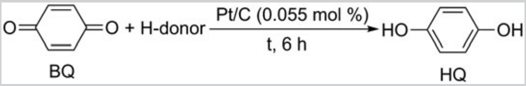
Transfer hydrogenation reactions using certain organic molecules as hydrogen sources have also attracted much attention of chemists, and have been comprehensively employed in both academia and industry, attributed to the high efficiencies of many hydrogen donors and transfer hydrogenation reactions.[3,4] Many transfer hydrogenation reactions such as coal liquefaction with tetralin can be performed under mild and safe conditions, and at industrial-level production.[5] During our continuing works in developing new catalytic methods of potential application in industry,[6] we developed a Pt/C-catalyzed method for transfer hydrogenation of mesityl oxide to methyl isobutyl ketone using isopropanol as the hydrogen source.[7] More recently, Pt/C was also found to be a good catalyst for transfer hydrogenation of BQ to produce HQ. However, interestingly, different from known methods,[7,8] in this novel Pt/C-catalyzed reaction, cyclohexanone was unexpectedly found to be a much more effective hydrogen donor than the commonly used secondary alcohols. Herein, we report the details of this work.

2 | RESULTS AND DISCUSSION

The Pt-catalyzed reduction reaction of BQ with isopropanol as the hydrogen donor was initially tested, but unfortunately only 5.1% of BQ was converted, while HQ, the desired product, was not detected (Table 1, entry 1). Experiments with isooctanol as an alternative hydrogen source were then conducted, but still no HQ was generated, even at elevated reaction temperature (Table 1, entries 2 and 3). We were glad to find that the reaction with cyclohexanol led to almost full conversion of BQ, and HQ was generated in 18.8% yield (Table 1, entry 4). Surprisingly, the reaction with cyclohexanone, which has rarely been employed as hydrogen source, afforded even higher HQ yield (32.8%) owning to the increased reaction selectivity (Table 1, entry 5). The excellent performances of cyclohexanone over other regular hydrogen donors may be attributed to a completely new reaction mechanism (*vide infra*).

Parallel experiments were then performed to further optimize the reaction conditions. First, considering the full conversion of BQ and the cost of the reaction, using cyclohexanone/BQ = 9.0 and 0.055 mol% of Pt/C, as we initially employed, should be the preferable reaction conditions (Table 2, entry 2 versus entries 1 and 3). Reducing and enhancing the amount of Pt catalyst both led to decreased HQ yield (Table 2, entry 2 versus entries 4 and 5). HQ began to be generated at a temperature higher than 60 °C (Table 2, entries 7 versus 6). Although the reaction at 80 °C afforded higher HQ selectivity than that at 142 °C, the BQ conversion ratio was low (Table 2, entry 7). It was found that 100 °C was the most favorable reaction temperature, and BQ was fully converted to produce HQ in increased yield (Table 2, entries 8 versus 9). It should also be pointed out that, beside the cyclohexanone dehydrogenation product cyclohex-2-en-1-one (**1**), 4-(cyclohexa-1,5-dien-1-yloxy) phenol (**2**) and (1,1′-bi(cyclohexan))-3-ene-2,2′,5-trione (**3**) were also observed in GC–MS analysis as the byproducts of the reaction (Figure 1). Thus, further efforts were made to enhance the selectivity and yield of HQ through inhibiting the generation of the byproducts.

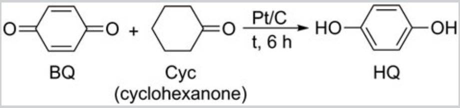
TABLE 1 Transfer hydrogenation of BQ with various hydrogen donors[a]



Entry	Hydrogen donor	t (°C)	Conv. (%)	Sel. (%)	Yield (%)
1	Isopropanol	80	5.1	0	0
2	Isooctanol	80	6.1	0	0
3	Isooctanol	142	16.8	0	0
4	Cyclohexanol	142	99.3	18.9	18.8
5	Cyclohexanone	142	100	32.8	32.8

[a] 50 mmol of BQ, 450 mmol of hydrogen donor and 540 mg of Pt/C (contains 1 wt% of Pt) were employed. Abbreviations: conv. = BQ conversion, sel. = HQ selectivity, yield = HQ yield; these results were obtained using HPLC and calculated through the external curve.

TABLE 2 Condition optimizations[a]



Entry	Cyc/BQ	Pt (%)	t (°C)	Conv. (%)	Sel. (%)	Yield (%)
1	4.5	0.055	142	97.6	32.4	31.6
2	9.0	0.055	142	100	32.8	32.8
3	15.0	0.055	142	100	33.6	33.6
4	9.0	0.025	142	98.7	30.4	30.0
5	9.0	0.11	142	100	30.8	30.8
6	9.0	0.055	60	15.4	0	0
7	9.0	0.055	80	52.7	42.2	22.2
8	9.0	0.055	100	100	36.6	36.6
9	9.0	0.055	120	100	36.4	36.4

[a] 50 mmol of BQ was employed. Abbreviations: Cyc/BQ = molar ratio of cyclohexanone versus BQ, Pt = Pt molar ratio on the basis of BQ amount, conv. = BQ conversion, sel. = HQ selectivity, yield = HQ yield; these results were obtained using HPLC and calculated through the external curve.

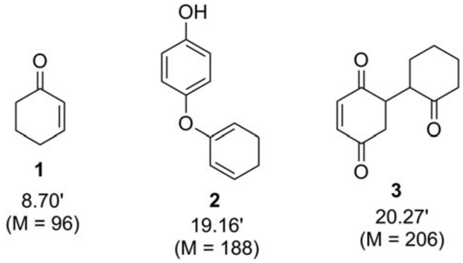


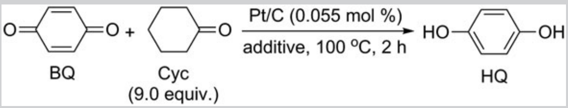
FIGURE 1 Observed byproducts of the reaction (detected using GC–MS)

The reaction was then optimized by tuning the pH values of the solution with HOAc or $(n\text{-Bu})_3N$. It was found that both acid and base conditions facilitated the process to enhance the BQ conversion (Table 3, entries 1 and 2 versus 3; 4 versus 3). Among these conditions, the weak alkaline condition with pH = 7.5 was preferable, affording almost full conversion of BQ and enhanced HQ selectivity and yield at 55.8 and 55.7% respectively (Table 3, entries 4 versus 1–3). Other bases, including both inorganic and organic bases, were also tested to adjust the reaction pH, and $(n\text{-Bu})_3N$, as we initially employed, was found to be the favorable one (Table 3, entries 4 versus 5–9). The ligand effect of organonitrogen bases may affect the catalytic activity of Pt and lead to different results even at the same pH value (Table 3, entries 4–9).

Reactions with various amounts of $(n\text{-Bu})_3N$ were conducted (Table 4, entries 1–7). With 1.7 mol% of $(n\text{-Bu})_3N$, HQ was afforded in 34.1% yield (41.4% selectivity), while 82.3% of BQ was converted within 2 h (Table 4, entry 1). Doubling the amount of base led to almost full conversion (99.8%) of BQ and the selectivity of HQ was enhanced to 61.1% (Table 4, entry 2). The reaction was hardly improved by increasing the amount of base, and 3.3 mol% of $(n\text{-Bu})_3N$ was finally screened to be the most favorable amount of base (Table 4, entry 2 versus entries 3–7).

Furthermore, the reactions with Pt, Cu, Ni and Pd catalysts were simultaneously performed for comparison (Table 5, entries 1–4). With the same weight (540 mg) of Pt/C, $CuCl_2$, Raney nickel and Pd/C as catalyst, the transfer hydrogenation reactions led to BQ conversions of 99.8, 86.9, 99.7and 99.6% and HQ selectivities of 61.1, 36.1, 53.6 and 59.2, respectively (Table 5, entries 1–4). Among the four catalysts, Pt/C was undoubtedly the most active one, affording an overwhelmingly high reaction turnover number (TON = 1109) over the other three catalysts (Table 5, entry 1 versus entries 2–4). Scanning electron microscopy images of the Pt/C catalyst (given in supporting information) showed that the materials possessed nanoscale structures, which provided large specific surface area and anchored Pt in the materials as the catalytically active reaction sites for the hydrogenation reactions.[9]

TABLE 3 Screenings of additives for the reaction[a]



Entry	pH	Additive	Conv. (%)	Sel. (%)	Yield (%)
1	2.5	HOAc	80.6	49.6	40.0
2	4.5	HOAc	69.2	46.2	32.0
3	6.0	HOAc	55.4	41.0	22.7
4	7.5	**$(n\text{-Bu})_3N$**	99.9	55.8	55.7
5	7.5	$NaHCO_3$	65.0	41.3	26.8
6	7.5	$NH_3 \cdot H_2O$	58.3	36.1	21.0
7	7.5	$(i\text{-}C_8H_{17})_3N$	63.9	46.1	29.5
8	7.5	Pyridine	99.8	53.1	53.0
9	7.5	3-Picoline	99.9	54.9	54.8

[a] 50 mmol of BQ, 450 mmol of cyclohexanone and 540 mg of Pt/C (contains 1 wt% of Pt) were employed. Abbreviations: conv. = BQ conversion, sel. = HQ selectivity, yield = HQ yield; these results were obtained using HPLC and calculated through the external curve.

TABLE 4 Screening of amount of $(n\text{-Bu})_3N$[a]

Entry	$(n\text{-Bu})_3N$/BQ (%)	Conv. (%)	Sel. (%)	Yield (%)
1	1.7	82.3	41.4	34.1
2	3.3	99.8	61.1	61
3	8.3	100	60.5	60.5
4	16.6	99.8	60.3	60.2
5	33.3	99.6	58.2	58
6	66.6	99.5	56.1	55.8
7	100	99.4	52.5	52.2

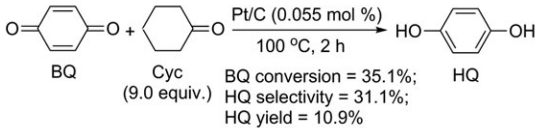
[a] Reaction was performed under the optimized conditions described in Table 3, entry 4 (except for amount of base). Abbreviations: conv. = BQ conversion, sel. = HQ selectivity, yield = HQ yield; these results were obtained using HPLC and calculated through the external curve.

TABLE 5 Comparison of catalytic metals[a]

Entry	Catalyst	Conv. (%)	Sel. (%)	Yield (%)	TON
1	1% Pt/C (0.055 mol%)	99.8	61.1	61	1109
2	$CuCl_2$ (8 mol%)	86.9	36.1	31.4	3.9
3	Raney nickel (18.4 mol%)	99.7	53.6	53.4	2.9
4	5% Pd/C (0.5 mol%)	99.6	59.2	59	118

[a] Reaction was performed under the optimized conditions described in Table 3, entry 4 (except for the catalyst). Abbreviations: conv. = BQ conversion; sel. = HQ selectivity; yield = HQ yield; TON = turnover number of the metal catalyst; these results were obtained using HPLC and calculated through the external curve.

The mechanisms of this interesting transfer hydrogenation reaction were our next concern. In order to get more information for a mechanism study, a control experiment under the optimized reaction conditions described in Table 3 but without base was performed (Scheme 1). It was found that the reaction was obviously retarded (Scheme 1 versus Table 3, entry 4) and the BQ conversion fell to 35.1% at 2 h, while both HQ selectivity and yield decreased as well (Scheme 1).



SCHEME 1 Control experiment

Dynamic studies were performed to further disclose the role of the base in the process. In the presence of $(n\text{-Bu})_3N$, BQ was completely converted within 2 h (Figure 2; pH = 7.5); whereas, in the absence of $(n\text{-Bu})_3N$, it took 6 h for the same reaction to complete the full conversion of BQ (Figure 2; pH = 6.2). Comparison of the experimental results in Scheme 1 with those in Table 3 (entry 4) as well as the dynamic study results illustrated in Figure 2 showed that base was crucial for the reaction to convert BQ quickly.

Moreover, besides BQ conversion, addition of base could also enhance the HQ selectivity of the reaction. As shown in Figure 3, the HQ selectivity of the reaction with base quickly reached its peak within 2 h (Figure 3; pH = 7.5), while it took a longer time for the reaction without base to afford the highest HQ selectivity (Figure 3; pH = 6.2). Therefore, considering the fact that the reaction without base required prolonged reaction time and led to decreased HQ selectivity, it was supposed that base might not only accelerate but also direct the reaction steps during the processes.

A series of parallel experiments were then conducted at 30–110 °C under the optimized conditions (pH = 7.5; Table 3, entry 4) to investigate the effect of the reaction temperature. As shown in Figure 4, although BQ could be completely converted at low temperatures such as 30 °C, the selectivity of the product could be enhanced by elevating the reaction temperature. This phenomenon suggested that this reaction might involve multiple steps or directions and the higher reaction temperature facilitated the reduction of BQ to HQ.

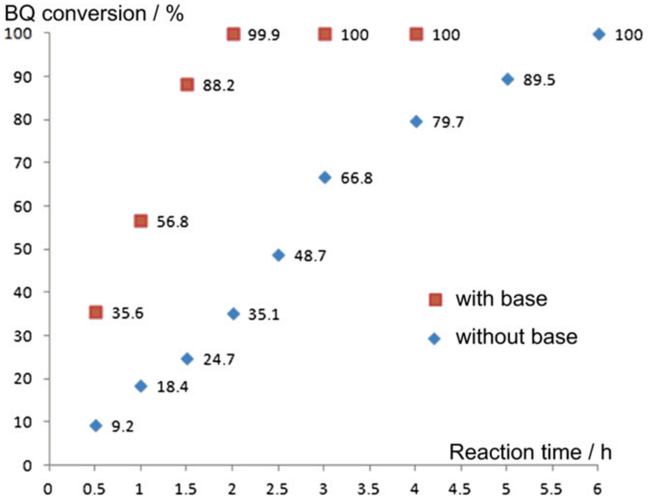


FIGURE 2 Comparison of BQ conversion in parallel reactions with/without base

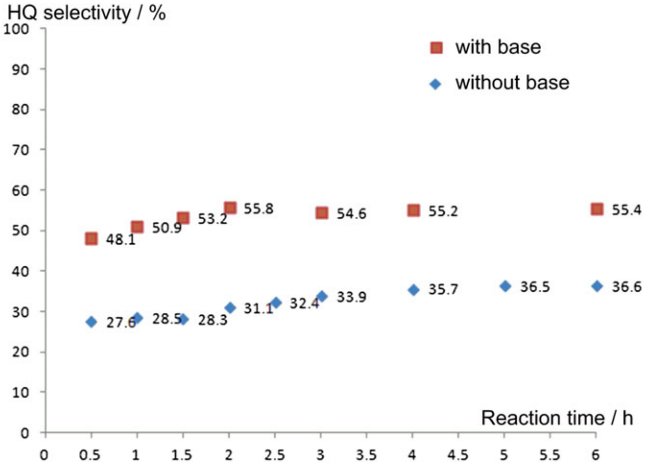


FIGURE 3 Comparison of HQ selectivity in parallel reactions with/without base

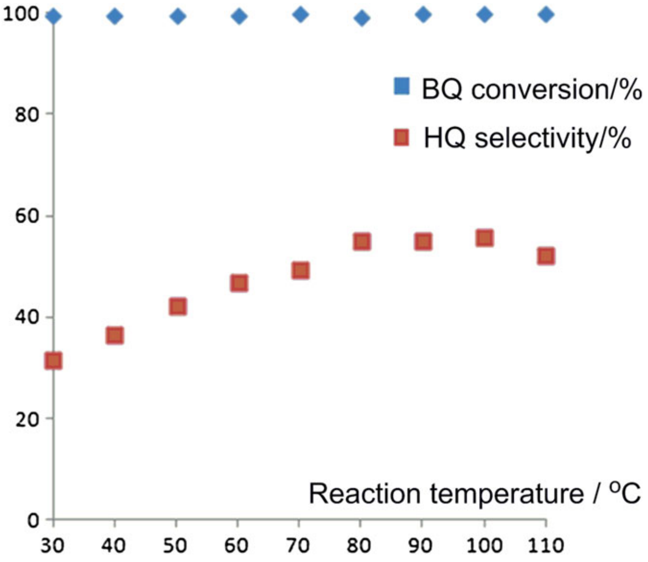
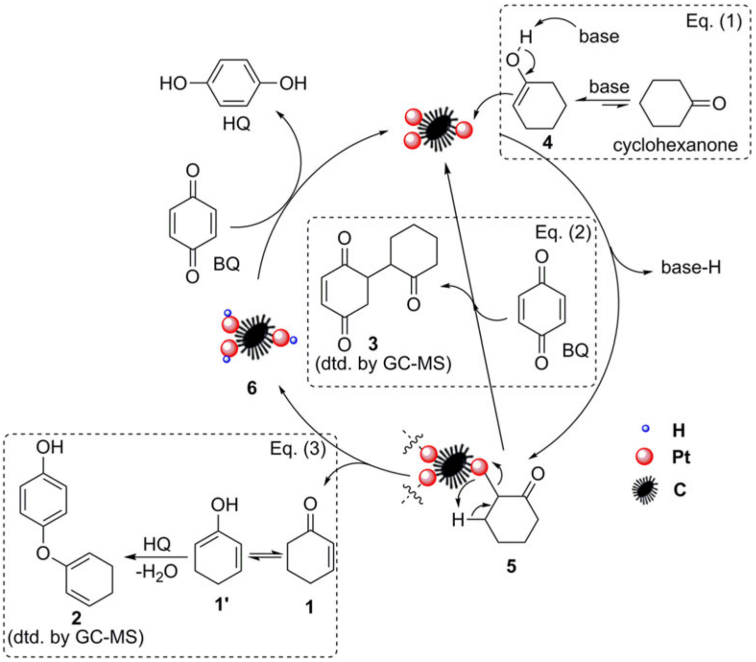


FIGURE 4 Effect of reaction temperature

Based on above experimental results as well as literature reports,[10–12] a plausible mechanism is proposed (Scheme 2). The first step of the reaction may occur with the conversion of cyclohexanone into its enol form **4** (equation (1) in Scheme 2). This step can be facilitated by both a base (Figures 2 and 3) and an acid (Table 3, entries 12 versus 3), but clearly the base is more preferable (Table 3, entries 4 versus 1 and 2). Attack of the base to the enol proton destroyed the O— H bond and its electron pair shifted to the C— O to form a carbonyl, while the π-electrons in the enol C═ C transferred to Pt to form the C— Pt bond and generated the intermediate **5**.[10] The generation of intermediate **5** could be confirmed by the



SCHEME 2 Possible mechanism of the transfer hydrogenation reaction

observed byproduct **3** (Figure 1), which was produced via the Michael addition of **5** with BQ (equation (2) in Scheme 2).[11] The dehydrogenation of **5** via β-hydrogen elimination[12] provided the key species **6** with Pt— H bonds as well as the cyclohexenone byproduct **1**. This step is confirmed by the observation of **1** in GC–MS spectra of the reaction mixtures (Figure 1). The byproduct **1** might further react with the product HQ to give the byproduct **2** (equation (3) in Scheme 2), which was also observed in the GC–MS spectra (Figure 1). Formation of the byproducts **2** and **3** (equations (2) and (3) in Scheme 2) may account for the high conversion ratio of BQ but moderate selectivity for HQ in the reaction; consequently, a moderate reaction temperature (such as 100 °C) is required to inhibit the side reactions and ensure the highest yield of HQ (Figure 4). Finally, reduction of BQ by **6** might afford the target product HQ and regenerate the active Pt/C catalyst.

3 | CONCLUSIONS

We report an efficient Pt/C-catalyzed transfer hydrogenation reaction of BQ to produce HQ, which is a very important industrial intermediate. Different from the previously reported transfer hydrogenation reactions, this method employs cyclohexanone as the hydrogen source instead of the classical secondary alcohols such as isopropanol. The starting material BQ was almost fully converted to produce the desired HQ in acceptable yield. Investigations of the mechanisms and applications of this novel reaction are ongoing in our laboratory.

4 | EXPERIMENTAL PROCEDURES

4.1 | General Methods

Reagents were purchased from suppliers with purities of more than 98% and were directly used as received. Solvents were analytically pure (AR) and directly used without any special treatment. HPLC analysis was performed using an Agilent HPLC 1260 instrument with an Agilent Eclipse XDB-C18 chromatographic column (4.6 × 250 mm × 5 μm) using chromatographically pure MeCN as the mobile phase solvent. GC–MS analysis was performed with a Thermo Fisher Trance 1300-ISQ LT instrument.

4.2 | Experimental Details for Transfer Hydrogenation Reaction of BQ to Produce HQ (Typical Example for the Reaction in Table 3, Entry 4)

Amounts of 50 mmol of BQ, 450 mmol of cyclohexanone, 1.65 mmol of (n-Bu)$_3$N (3.3%) and 540 mg of Pt/C (1 wt% Pt) were initially added into a 100 ml round-bottom flask. The mixture was heated at 100 °C for 2 h and sent for HPLC analysis. The detailed data tables for the figures in the text are given in the supporting information.

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SUPPORTING INFORMATION

Additional supporting information may be found online in the Supporting Information section at the end of the article.

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