Selective oxidation of primary benzylic alcohols to aldehydes using NaNO₃ under ball milling

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Abstract
A facile method for oxidation of primary benzylic alcohols to the corresponding aldehydes is reported using NaNO₃/P₂O₅ under high-speed ball-milling conditions. This approach is clean, efficient, and exhibits broad functional group compatibility.

Keywords
alcohols, aldehydes, ball milling, NaNO₃, oxidation

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Introduction
The selective oxidation of alcohols to the corresponding aldehydes/ketones plays a central role in the synthetic chemist’s toolkit and has received increasing attention in recent years. Many reagents have been developed for oxidation of benzylic alcohols to the corresponding carbonyl compounds, such as chromium reagents, manganese oxidizing reagents, 2,2,6,6-tetramethylpiperidyl-1-oxy (TEMPO)-related reagents, and nitrite- and nitrate-related reagents. However, these reactions usually suffer from disadvantages, including the application of heavy-metal-based reagents, harsh reaction conditions, long reaction times, and toxic solvents. With ever-increasing environmental concerns, the development of a general and efficient synthetic protocol for the oxidation of benzylic alcohols is still highly desirable.

Mechanochemical organic methodologies have recently attracted significant attention as a powerful tool for organic chemists. Mechanochemistry is generally performed under solvent-free conditions, and as the reagent concentrations are high, this often leads to accelerated reactions and alterations in product selectivity, typically in favor of the discovery of new chemical transformations. In addition, the technique also covers multiple aspects of green chemistry, which can avoid bulk solvents and reduce environmental pollution.

In our previous work in the area of mechanochemical synthesis, the combination of Bi(NO₃)₃·5H₂O and MgSO₄ was successfully employed for the nitration of aromatic...
compounds. Recently, we reported that the combination of Fe(NO$_3$)$_3$·9H$_2$O and P$_2$O$_5$ could convert deactivated arenes into the corresponding nitrated arenes in excellent yields under high-speed ball-milling (HSBM) conditions. We proposed that the N$_2$O$_4$/NO$_2$, which was produced in situ from the reaction of nitrates and the auxiliary, should play a major role in these mechanochemical nitrations. Since N$_2$O$_4$/NO$_2$ species are also well-known as oxidants, we set out to explore the oxidative nature of the combination of nitrates and auxiliary. In this paper, a combination of NaNO$_3$ and P$_2$O$_5$ is employed as a simple, stable, and inexpensive oxidant for the oxidation of primary benzylic alcohols to aldehydes under HSBM conditions.

### Results and discussion

Initially, 4-methylbenzyl alcohol (1a) was chosen as the model substrate and the molar ratio of 1a/nitrate salt/P$_2$O$_5$ was fixed at 1:3:1 to optimize the reaction conditions (Table 1, entries 1–5). The reactions were conducted in a standard 2.5-mL screw-capped milling beaker and milled with one stainless steel ball of 6.0-mm diameter in a high-energy vibrational MM400 mixer mill at a frequency of 28 Hz for 2 h at room temperature. A variety of different nitrate salts were tested for oxidation of the alcohol to an aldehyde under HSBM conditions (Table 1, entries 1–5). Several nitrate salts including Cu(NO$_3$)$_2$·9H$_2$O, Al(NO$_3$)$_3$·9H$_2$O, Cr(NO$_3$)$_3$, and Fe(NO$_3$)$_3$·9H$_2$O were ineffective in producing the corresponding aldehyde (Table 1, entries 1–4). Encouragingly, 1a underwent smooth oxidation to afford a 57% yield of the desired 4-methylbenzaldehyde (2a), together with 23% of over-oxidized product 3a and 4% of nitrate ester 5a, when NaNO$_3$ was used (Table 1, entry 5).

The control experiments revealed that both the nitrate salt and P$_2$O$_5$ were indispensable for the current oxidation reaction, since no conversion of 1a was observed in the absence of P$_2$O$_5$ and only a trace amount of 2a was afforded in the absence of NaNO$_3$ (Table 1, entries 6 and 7).

We noticed that the nitrate/P$_2$O$_5$ molar ratio had a strong influence on the outcome of the reactions during our exploration of these HSBM reactions. To obtain the best yield of the product, the HSBM reaction conditions were further optimized by adjusting the molar ratio of NaNO$_3$ and P$_2$O$_5$ in relation to 1a (Table 1, entries 8–15). First, in order to find a relatively good molar ratio of P$_2$O$_5$, the molar ratio of 1a/NaNO$_3$ was fixed as 1:3 and milled with different quantities of P$_2$O$_5$ (Table 1, entries 8–11). When performing the reaction using 0.75 equiv. of P$_2$O$_5$, a 68% yield of 2a was achieved (Table 1, entry 8). On further decreasing the quantity of P$_2$O$_5$ to 0.5 equiv., the yield of 2a sharply increased to 86% (Table 1, entry 9). However, further decreasing the quantity of P$_2$O$_5$ to 0.25 equiv. and 0.125 equiv. resulted in decreased conversions of 1a to 80% and 27%, leading to significant decreases of the yields of 2a to 60% and 20%, respectively, implying that the best molar ratio of 1a/P$_2$O$_5$

### Table 1. Optimization of the reaction conditions.

| Entry | Nitrate          | Molar ratio$^b$ | Reaction time (h) | Conversion (%) | Yield (%)$^c$ |
|-------|------------------|-----------------|-------------------|---------------|--------------|
| 1     | Cr(NO$_3$)$_3$   | 1:3:1           | 2                 | 96            | 36           |
| 2     | Cu(NO$_3$)$_2$·9H$_2$O | 1:3:1           | 2                 | 95            | 48           |
| 3     | Al(NO$_3$)$_3$·9H$_2$O | 1:3:1           | 2                 | 94            | 43           |
| 4     | Fe(NO$_3$)$_3$·9H$_2$O | 1:3:1           | 2                 | 95            | 13           |
| 5     | NaNO$_3$        | 1:3:1           | 2                 | 97            | 57           |
| 6     | NaNO$_3$        | 1:3:1           | 2                 | 0             | N.D.         |
| 7     | NaNO$_3$        | 1:0:1           | 2                 | 96            | Trace        |
| 8     | NaNO$_3$        | 1:3:0.75        | 2                 | 96            | 68           |
| 9     | NaNO$_3$        | 1:3:0.5         | 2                 | 97            | 86           |
| 10    | NaNO$_3$        | 1:3:0.25        | 2                 | 80            | 60           |
| 11    | NaNO$_3$        | 1:3:0.125       | 2                 | 27            | 20           |
| 12    | NaNO$_3$        | 1:5:0.5         | 2                 | 96            | 86           |
| 13    | NaNO$_3$        | 1:4:0.5         | 2                 | 96            | 86           |
| 14    | NaNO$_3$        | 1:2:0.5         | 2                 | 91            | 80           |
| 15    | NaNO$_3$        | 1:1:0.5         | 2                 | 77            | 71           |
| 16    | NaNO$_3$        | 1:3:0.5         | 1                 | 82            | 76           |
| 17    | NaNO$_3$        | 1:3:0.5         | 0.5              | 47            | 44           |

N.D.: not detected.
The side products are known compounds and were determined by $^1$H NMR (see ESI for details).

$^a$Reaction conditions: 1a (0.05 mmol), reaction frequency (28 Hz), 2 h.

$^b$Molar ratio of 1a/nitrate/P$_2$O$_5$.

$^c$Yields were determined by $^1$H NMR analysis of the crude reaction mixture using dibromomethane as an internal standard.
was 1:0.5 for this HSBM reaction (Table 1, entries 10 and 11). Next, by keeping the molar ratio of 1a/NaNO3 as 1:0.5, the amount of NaNO3 was studied. On increasing of the quantity of NaNO3 to 4 equiv. and 5 equiv., the product yield plateaued at 86% (Table 1, entries 12 and 13), while decreasing the quantity of NaNO3, to 2 equiv. and 1 equiv. led to lower yields of 80% and 71%, respectively (Table 1, entries 14 and 15), implying that the best molar ratio of 1a/NaNO3 was 1:3. In light of these results, a molar ratio of alcohol/NaNO3/P2O5 of 1:3:0.5 was selected for the HSBM reactions in further studies. When the ratio of alcohol/NaNO3/P2O5 was set as 1:3:0.5, shortening the reaction times to 1 and 0.5 h resulted in sharp decreases in the conversion of benzyl alcohol, leading to 76% and 44% yields of aldehyde 2a, respectively (Table 1, entries 16 and 17).

The conversion of 1a into the corresponding aldehyde in the presence of NaNO3/P2O5 was also carried out under conventional refluxing conditions in different organic solvents (Table 2). On refluxing in tetrahydrofuran, the reaction was not complete, even after 8 h and consequently gave a poor yield (Table 2, entry 1). Other solvents like acetonitrile, ethyl acetate, and dichloromethane gave even lower yields of product 2a (Table 2, entries 2–4). These results indicate that ball milling gives better results in comparison to conventional heating for this application.

With optimized reaction conditions in hand, we next explored the scope of our oxidation system on a series of benzylic primary alcohols. Benzyl alcohol (1b) and 4-phenylbenzyl alcohol (1c) afforded the desired aldehydes 2b and 2c in high yields (Table 3, entries 1 and 2). The primary benzylic alcohols 1d–k with electron-withdrawing groups on the phenyl ring were smoothly oxidized to give the corresponding aldehydes 2d–k in good to excellent yields (78%–100%) (Table 3, entries 3–10). It should be noted that all of these substrates afforded the corresponding aldehydes, accompanied with traces of over-oxidized products. These results are similar to those of Tokunaga using tert-butyl nitrite as the oxidizing agent in solution chemistry; however, the reaction yields obtained in our work are much higher. For primary benzylic alcohols 1l–o, bearing electron-donating groups gave somewhat lower yields than those with electron-withdrawing groups (55%–74% vs 78%–100%) (Table 3, entries 11–14). These results imply that the presence of electron-donating groups leads to reduced reactivity during the oxidation.

Many hydrolyzable and oxidatively sensitive functional groups such as amido, ester, and formyl are tolerated under these ball-milling conditions, indicating that our NaNO3/P2O5 system is an effective combination for obtaining aldehydes, and can be used widely for the oxidation of various substituted benzylic primary alcohols. In addition, 2-naphthalenemethanol (1p) was converted into 2-naphthaldehyde (2p) in 74% yield (Table 3, entry 15). Extending the reaction time to 4 h increased the yield to 82% and shows that this protocol is also applicable to the oxidation of fused-ring aromatic primary alcohols.
We also examined the properties of NaNO3/P2O5 as an oxidant reagent for secondary benzylic alcohols. In contrast to the behavior of primary benzylic alcohols, 6a–c mainly gave the corresponding nitrate esters 7 and ethers 8 (Table 4, entries 1–3). A similar pattern was observed in the oxidation of benzylic alcohols and ethers using nitric acid in dichloromethane reported by Strazzolini and Runcio.15 It can be assumed that compared with primary benzylic alcohols, 1, the presence of an α-substituted group in the secondary benzylic alcohols 6 facilitates the formation of the intermediate cations during the reaction, depressing the yields of carbonyl products owing to the competitive esterification and etherification to afford esters 7 and ethers 8.

Mechanistically, a nitrate ester could be a candidate intermediate toward the formation of aromatic aldehydes.33,34 Since small amount of nitrate ester 5 was detected in our experiment, we performed the same ball-milling reaction in Table 3 by replacing primary aromatic alcohol 1 with 5b to explore if the aromatic aldehyde is formed from nitrate ester in the present work. The results revealed that 87% of 5b were remained unchanged, while 10% of 4-nitrobenzyl nitrate and 3% of 2-nitrobenzyl nitrate ester were obtained, respectively. No corresponding benzyl aldehyde 2b was formed (Supplemental Figure S2, ESI), indicating that the nitrate ester could not be an intermediate in our benzylic oxidation as proposed in some instances.

Considering that NaNO3/P2O5 system works effectively for the oxidation of primary benzylic alcohols, we therefore turned our attention to primary non-benzylic alcohols. Interestingly, while the oxidation of primary benzylic alcohols proceeded well, 2-(4-methylphenyl)ethanol (9) was found to be inert toward the present reaction conditions (Scheme 1). This result agrees with Nishiguchi’s work using metallic nitrates supported on silica gel as oxidants.12 In addition, we also observed a brown gas during the experiments, just as Nishiguchi reported, when we opened the mill beaker straight after the reaction. This result suggests nitrogen dioxide, which is a radical, is formed under the HSBM condition.

Since excellent oxidation of the benzylic primary hydroxy group was observed, while an aliphatic primary alcohol was inert under this NaNO3/P2O5 system, we decided to evaluate the selectivity of the reagent. The selective oxidation of diols containing both a primary benzylic and an aliphatic hydroxy group was studied as shown in Scheme 2. The diol 10 gave 70% conversion and underwent chemoselective oxidation of the primary benzylic hydroxy group to give 11, without oxidation of the aliphatic hydroxy group, in 55% yield, together with a 15% yield of unknown side products. This result showed the practicality and efficiency of the NaNO3/P2O5 system as a chemoselective oxidant for primary benzylic alcohols.

Currently, we do not understand the precise mechanism of this reaction. Nishiguchi and Strazzolini have presumed a radical mechanism for the oxidation of alcohols.12,15 Thus, we propose a radical pathway for our oxidation of benzylic alcohols since our results are comparable with those of Strazzolini and Nishiguchi (Scheme 3). First, NaNO3 would react with P2O5 to generate nitric acid, which could be decomposed to generate NO2/N2O4 under the mechanical force, as described in our previous work.27,28 Next, the in situ-produced NO2/N2O4 should be responsible for the oxidation, most probably, following the radical-type mechanism reported by Nishiguchi and Strazzolini.12,15

**Conclusion**

In conclusion, we have reported a methodology for the selective oxidation of primary benzylic alcohols to the corresponding aldehydes using a combination of NaNO3 and P2O5 under HSBM conditions. Chemoselective oxidation of a diol containing both a primary benzylic and a primary aliphatic hydroxy group was also performed. This procedure offers several advantages, including good to excellent yields of aldehydes, compatibility with a broad range of functional groups, the use of simple and inexpensive oxidants, and solvent-free reaction conditions. These features make the present method a useful alternative to current methods for the oxidation of benzylic alcohols.

**Experimental**

**General**

All chemicals and reagents were purchased from Meryer (Shanghai, China), Alfa Aesar (Ward Hill, MA, USA), or Energy Chemical (Shanghai, China), and were used as received. The reactions were conducted in a stainless steel milling beaker (2.5 mL) using a stainless steel milling ball.
in an MM400 mixer ball mill (Retsch GmbH, Germany). Column chromatography was performed using 100–200 mesh silica gel purchased from Qingdao Haiyang Chemical Co. Ltd. (China). Appropriate solvent systems for chromatography were selected according to thin-layer chromatography (TLC) analysis using UV light (254 and 365 nm) to visualize the reaction components. Melting points (m.p.) were determined on a digital melting point apparatus and are uncorrected. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AV 400 spectrometer (Billerica, MA, USA) at 400 MHz (1H NMR) and at 101 MHz (13C NMR). The peak patterns are designated as follows: s, singlet; d, doublet; t, triplet; q, quartet; dd, doublet of doublets; dt, doublet of triplets; m, multiplet. Coupling constants, J, are reported in Hz. The product was dissolved in the appropriate deuterium reagent (CDCl3 or dimethyl sulfoxide (DMSO)) and tetramethylsilane (TMS) or the solvent peak was used as a standard. An APEX II (Bruker Inc.) mass spectrometer was used for high-resolution mass spectrometry (HRMS) analysis.

**General procedure for the synthesis of compounds 2 and 11**

For all the experiments, a mixture of alcohol (1) (0.05 mmol), NaNO3 (0.15 mmol), and P2O5 (0.025 mmol) was added to a stainless steel milling beaker (2.5 mL), along with a stainless steel milling ball (ø=6 mm). The beaker was sealed and placed in the mixer mill. The mechanochemical reaction was performed at 28 Hz for 2 h at room temperature. Subsequently, the reaction mixture was extracted with dichloromethane (8 × 2 mL). The organic extracts from 12 such reactions were combined, and the mixture was filtered. The filtrate was concentrated in vacuo, and the residue was purified by column chromatography on silica gel to afford aldehyde 2.

**Scheme 2.** Chemoselective oxidation of diol 10.

**Scheme 3.** A proposed mechanism for the oxidation of primary benzylic alcohols.

4-Methylbenzaldehyde (2a): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 30:1) in 86% yield (62.01 mg); colorless liquid (lit.37); 1H NMR (400 MHz, CDCl3): δ 9.97 (s, 1H), 7.78 (d, J = 8.0 Hz, 2H), 7.34 (d, J = 7.9 Hz, 2H), 2.44 (s, 3H); 13C NMR (101 MHz, CDCl3): δ 192.1, 145.6, 134.2, 129.9, 128.9, 21.9; HRMS (ESI): m/z [M + H]+ calculated for C8H9O: 121.0648; found: 121.0641.

Benzaldehyde (2b): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 30:1) in 83% yield (52.85 mg); colorless liquid (lit.36); 1H NMR (400 MHz, CDCl3): δ 10.03 (s, 1H), 7.89 (d, J = 7.4 Hz, 2H), 7.64 (t, J = 7.4 Hz, 1H), 7.54 (t, J = 7.6 Hz, 2H); 13C NMR (101 MHz, CDCl3): δ 192.5, 136.4, 134.5, 128.9, 129.0; HRMS (ESI): m/z [M + Na]+ calculated for C6H5O: 107.0491; found: 107.0495.

4-Biphenylcarboxaldehyde (2c): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 30:1) in 92% yield (68.51 mg); light yellow solid; m.p. 57–59°C (lit.39 44–46°C); 1H NMR (400 MHz, CDCl3): δ 9.97 (s, 1H), 7.96 (d, J = 8.2 Hz, 2H), 7.76 (d, J = 8.2 Hz, 2H), 7.64 (d, J = 7.3 Hz, 2H), 7.49 (t, J = 7.6 Hz, 2H), 7.42 (t, J = 7.4 Hz, 1H); 13C NMR (101 MHz, CDCl3): δ 192.0, 147.3, 139.8, 135.2, 130.3, 129.1, 128.5, 127.7, 127.4; HRMS (ESI): m/z [M + Na]+ calculated for C13H10O: 206.0624; found: 205.0629.

4-Fluorobenzaldehyde (2d): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 30:1) in 92% yield (68.51 mg); light yellow solid; m.p. 57–59°C (lit.39 44–46°C); 1H NMR (400 MHz, CDCl3): δ 9.97 (s, 1H), 7.96 (d, J = 8.5 Hz, 2H), 7.72 (t, J = 8.5 Hz, 2H), 7.64 (d, J = 7.4 Hz, 2H), 7.42 (t, J = 7.4 Hz, 1H); 13C NMR (101 MHz, CDCl3): δ 190.6 (s), 166.6 (d, J = 22.3 Hz), 132.3 (d, J = 9.7 Hz), 116.4 (d, J = 22.3 Hz); 19F NMR (376 MHz, CDCl3): δ −102.34; HRMS (ESI): m/z [M + Na]+ calculated for C7H5FO: 125.0394; found: 125.0397.

4-Chlorobenzaldehyde (2e): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 30:1) in 89% yield (97.31 mg); light yellow solid; m.p. 55–57°C (lit.39 57–59°C); 1H NMR (400 MHz, CDCl3): δ 9.97 (s, 1H), 7.96 (d, J = 8.5 Hz, 2H), 7.76 (d, J = 8.2 Hz, 2H), 7.64 (d, J = 7.3 Hz, 2H), 7.54 (t, J = 7.6 Hz, 2H), 7.42 (t, J = 7.4 Hz, 1H); 13C NMR (101 MHz, CDCl3): δ 190.6 (s), 166.4 (d, J = 22.3 Hz), 132.3 (d, J = 9.7 Hz), 116.4 (d, J = 22.3 Hz); 19F NMR (376 MHz, CDCl3): δ −102.34; HRMS (ESI): m/z [M + Na]+ calculated for C8H4ClO: 121.0641; found: 121.0644.

4-Bromobenzaldehyde (2f): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 30:1) in 98% yield (108.79 mg); light yellow solid; m.p. 56–58°C (lit.39 57–59°C); 1H NMR (400 MHz, CDCl3): δ 9.98 (s, 1H), 7.72 (dd, J = 25.5, 8.5 Hz, 4H); 13C NMR (101 MHz, CDCl3): δ 191.1, 135.1, 132.5, 131.0, 129.8; HRMS (ESI): m/z [M + Na]+ calculated for C8H5BrO: 206.9416; found: 206.9419.

2,4-Dibromobenzaldehyde (2g): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 30:1) in 100% yield (158.35 mg); white crystalline solid; m.p.
81–84 °C (lit. 40 84–85 °C); 1H NMR (400 MHz, CDCl3): δ 10.30 (s, 1H), 7.85 (s, 1H), 7.78 (d, J = 8.3 Hz, 1H), 7.59 (d, J = 8.3 Hz, 1H), 7.28 (d, J = 8.5 Hz, 2H), 2.34 (s, 3H); 13C NMR (101 MHz, CDCl3): δ 191.0, 168.8, 154.5, 134.0, 131.3, 122.4, 21.2; HRMS (ESI): m/z [M + Na]+ calecd for C11H8O4: 187.0367; found: 187.0369.

4-Formylbenzoic acid (2h): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 10:1) in 78% yield (70.26 mg): yellow crystalline solid; m.p. 145–147 °C (lit. 41 145–147 °C); 1H NMR (400 MHz, DMSO-d6): δ 10.10 (s, 1H), 8.12 (d, J = 7.6 Hz, 2H), 7.01 (d, J = 7.7 Hz, 2H); 13C NMR (101 MHz, DMSO-d6): δ 192.8, 151.2, 140.1, 130.5, 129.4, 129.3, 129.2; HRMS (ESI): m/z [M − H]+ calecd for C9H8O3: 187.0367; found: 187.0369.

4-Benzaldehydeisolated by silica gel column chromatography (petroleum ether/ethyl acetate = 10:1) in 100% yield (80.48 mg): white solid; m.p. 114–115 °C (lit. 42 114–115 °C); 1H NMR (400 MHz, CDCl3): δ 10.14 (s, 1H), 8.05 (s, 1H), 7.43 (d, J = 8.6 Hz, 2H), 3.89 (s, 3H); 13C NMR (101 MHz, CDCl3): δ 188.1, 149.6, 134.1, 133.7, 131.4, 129.7, 129.3; HRMS (ESI): m/z [M + Na]+ calecd for C7H5NO3: 152.0347; found: 152.0347.

4-Nitrobenzaldehyde (2l): Isolated by silica gel column chromatography (petroleum ether/ethyl acetate = 25:1) in 55% yield (53.85 mg): yellow crystalline solid; m.p. 144–145 °C (lit. 44 144–145 °C); 1H NMR (400 MHz, CDCl3): δ 9.99 (s, 1H), 7.84 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 7.9 Hz, 2H), 3.93 (t, J = 6.5 Hz, 2H), 2.96 (t, J = 6.4 Hz, 2H); 13C NMR (101 MHz, CDCl3): δ 192.0, 146.2, 135.0, 130.1, 129.7, 63.1, 39.3; HRMS (ESI): m/z [M + Na]+ calecd for C7H5NO3: 150.0373; found: 150.0373.

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