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A Mild One-Pot Conversion of Alkenes into Amines through Tandem Ozonolysis and Reductive Amination

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Abstract

The selective reduction of hydroperoxyacetals to aldehydes by sodium triacetoxyborohydride provides the basis for a mild one-pot synthesis of amines from alkenes.

Keywords: ozonolysis, hydroperoxyacetal, reductive amination, amine, sodium triacetoxyborohydride

Reductive amination is a widely used transformation in organic synthesis.² The carbonyl precursors are often prepared from alkenes through ozonolysis followed by reduction of the ozonide or peroxide intermediates.^{3, 4} In the course of investigations into new transformations based upon fragmentation of ozonolysis intermediates,⁵ we became interested in the development of a mild method for a one-pot conversion of alkenes to amines. Reductive amination is frequently achieved through the reaction of carbonyls and amines in the presence of deactivated boron hydrides,^{2b, 6, 7} and NaCNBH₃-promoted reductive amination has been applied in tandem with ozonolysis.⁸ However, NaCNBH₃ reduces ozonides (1,2,4-trioxolanes) very slowly,⁹ and some of the reported transformations may actually involve amine-promoted E₁cb fragmentation of terminal ozonides.^{10, 11} We investigated the reaction of NaCNBH₃ with more reactive hydroperoxyacetals, but observed significant formation of alcohols. Our attention was therefore turned to the less reactive triacetoxyborohydride, which has been investigated little in ozonolysis/amination sequences.^{12, 13} We now report that NaBH(OAc)₃ allows rapid and efficient reduction of ozonolysis-derived hydroperoxyacetals to aldehydes, enabling a mild and convenient one-pot synthesis of amines from alkenes based upon ozonolysis and reductive amination.

The starting hydroperoxyacetals were readily available through ozonolysis of precursor alkenes or enol ethers in 3% MeOH–CH₂Cl₂ (Table 1).^{14, 15}

Reduction conditions were initially screened on hydroperoxyacetal **1b** using ¹H NMR of crude reaction mixtures to monitor the ratio of starting hydroperoxyacetal, aldehyde (nonanal), and the alcohol (nonanol) derived from overreduction. It was found that NaBH(OAc)₃ offered high selectivity for aldehyde formation in several different solvent systems (CH₂Cl₂, DCE, THF) and under several sets of conditions. Performing the reduction with one equivalent of NaBH(OAc)₃ proved optimal; and excess of reducing reagent (1.5–3 equiv) led to slow overreduction of the aldehyde. In contrast, the use of the more reactive NaCNBH₃ in stoichiometric amounts cleanly generated alcohol, while a mixture of alcohol and starting material was observed even with only 0.33 equivalent of reagent.

Table 1. Preparation of Hydroperoxyacetals

alkene	$\xrightarrow[\text{MeOH-CH}_2\text{Cl}_2]{\text{O}_3/\text{O}_2}$	hydroperoxyacetal	
Alkene		Hydroperoxyacetal	Yield
			1b (70%)
1a			
			2b (67%)
2a			
			3b (69%)
3a			
			4b (38%) ^a
4a			
			5b (74%)
5a			
			6b (63%)
6a			

^a Partial decomposition upon chromatography.

Reduction with NaBH(OAc)₃ was next applied to a wider group of hydroperoxyacetals (Table 2). Hydroperoxyacetals reacted faster (2 h) than hydroperoxyketals (5–8 h). In both cases, a high yield of the carbonyl was obtained with little or no overreduction.

Reductive amination was easily conducted through reduction of the hydroperoxyacetal with NaBH(OAc)₃ (1 equiv), followed by addition of amine and additional hydride (Scheme 1). The reactions were found to give good yields of monoalkylated amines. For the amination of aldehyde **2b** with benzylamine, 8% of the tertiary amine derived from overalkylation was isolated, in addition to the expected secondary amine **2f**.

Finally, it was found that ozonolysis and reductive amination could be easily combined into a one-pot transformation

Scheme 2

While there are many procedures for conversion of alkenes to amines, most require separate oxidation and amination steps, each involving isolation and purification. The procedure reported here, which allows a one-pot conversion of alkenes to a variety of amines while avoiding any accumulation of peroxide intermediates, may be of particular use for preparation of an array of amines from a common precursor, or in procedures limited by concerns with isolation or handling of peroxide intermediates. Utilization of $\text{NaBH}(\text{OAc})_3$ in place of NaCNBH_3 provides a more economical, safe, and selective protocol for the one-pot tandem ozonolysis/reductive amination sequence for the conversion of alkenes to amines. The speed and selectivity of the reduction of the hydroperoxyacetals suggests the formation and decomposition of a peroxyborate, $\text{ROOBH}(\text{OAc})_2$.

All reagents were used as received from commercial vendors, with the exception of CH_2Cl_2 , which was distilled from CaH_2 , and THF, which was distilled from Na/benzophenone . TLC was performed on 0.25 mm hard-layer silica G plates; developed plates were visualized with a hand-held UV lamp or by staining: 1% $\text{Ce}(\text{SO}_4)_2$ and 10% $(\text{NH}_4)_2\text{MoO}_4$ in 10% H_2SO_4 (general stain, after charring); 1% N,N' -dimethyl-*p*-phenylenediamine solution in 1:20:100 $\text{AcOH-H}_2\text{O-MeOH}$ (specific for peroxides);¹⁶ 1% aq KMnO_4 (for unsaturated compounds); 3% vanillin in 3% H_2SO_4 in EtOH (general stain after charring). NMR spectra were recorded at 400 MHz (^1H) or 100 MHz (^{13}C) and in CDCl_3 unless otherwise indicated; peaks are reported as: chemical shift (multiplicity, *J* couplings in Hz, number of protons). IR spectra were recorded as neat films (ZnSe , ATR mode) with selected absorbances reported in wavenumbers (cm^{-1}). Melting points were collected using a melting point apparatus and all values are uncorrected unless otherwise noted.

Hydroperoxyacetals from Alkenes; General Procedure 1 (GP 1)

A solution of alkene (~1 mmol) and MeOH (0.3 mL) in CH_2Cl_2 (10 mL) in a round-bottomed flask was cooled to -78°C and a gaseous stream of 2% O_3/O_2 (approximately 1 mmol O_3/min) was bubbled through the solution. Once the pale blue color of ozone appeared, the ozonizer voltage was set to zero and the reaction was sparged with O_2 until the blue color dissipated. The reaction was quenched with sat. aq NaHCO_3 (15 mL) and the resulting mixture was allowed to warm to r.t. The separated aqueous layer was extracted with CH_2Cl_2 (2×25 mL) and the combined organic layers were dried (Na_2SO_4). The residue obtained upon concentration in vacuo was purified by flash chromatography using EtOAc-hexanes .

Carbonyl Compounds from Hydroperoxyacetals; General Procedure 2 (GP 2)

To a r.t. solution of hydroperoxyacetal (~0.5 mmol) in DCE (10 mL) was added $\text{NaBH}(\text{OAc})_3$ (0.5 mmol). After product formation was complete (TLC, eluent: EtOAc-hexanes), the reaction was diluted with CH_2Cl_2 (~40 mL) and washed sequentially with sat. aq NaHCO_3 (~30 mL) and brine (~30 mL). The organic layer was dried (Na_2SO_4) and the residue obtained upon concentration in vacuo was purified by flash chromatography using EtOAc-hexanes .

Amines from Hydroperoxyacetals; General Procedure 3 (GP 3)

To a solution of hydroperoxyacetal (~0.5 mmol) in DCE (10 mL) was added $\text{NaBH}(\text{OAc})_3$ (0.5 mmol). The reaction mixture was stirred for an hour at r.t., after which additional $\text{NaBH}(\text{OAc})_3$ (1 mmol) was added followed by amine (0.5 mmol). After the reaction appeared complete (TLC, eluent: EtOAc-hexanes), the solution was diluted with CH_2Cl_2 (40 mL), and washed sequentially with sat. aq NaHCO_3 (30 mL) and brine (30 mL). The organic layer was dried (Na_2SO_4) and the residue obtained upon concentration was purified by flash chromatography with $\text{CH}_2\text{Cl}_2\text{-MeOH}$ (1-2%) containing a few drops of Et_3N .

Direct Synthesis of Amines from Alkenes; General Procedure 4 (GP 4)

Stepwise (Method A): A solution of alkene (~1 mmol) and MeOH (0.3 mL) in CH_2Cl_2 (10 mL) in a round-bottomed flask was ozonized as in GP 1. Following removal of free ozone, the cooling bath was removed and $\text{NaBH}(\text{OAc})_3$ (1 mmol) was added. The reaction mixture was stirred at r.t. for 45 min, after which amine (~1 mmol) and $\text{NaBH}(\text{OAc})_3$ (2 mmol) were sequentially added. The mixture was stirred at r.t. for 1-2 h and then filtered through Celite and the pad washed with CH_2Cl_2 (20 mL). The filtrate was concentrated on a rotary evaporator, and the residue was purified as in GP 3.

One Step (Method B): A solution of alkene (~1 mmol) and MeOH (0.3 mL) in CH_2Cl_2 (10 mL) in a round-bottomed flask was ozonized as in GP 1. Following removal of free ozone, the cooling bath was removed and $\text{NaBH}(\text{OAc})_3$ (3 mmol) and the amine (1-2 mmol) were sequentially added. The reaction mixture was stirred at r.t. for 1-2 h and then filtered through Celite and the pad washed with CH_2Cl_2 (20 mL). The filtrate was concentrated on a rotary evaporator, and the residue was purified as in GP 3.

Hydroperoxyacetals from Alkenes 1-Methoxynonyl Hydroperoxide (1b) [20525-41-1]

Prepared from **1a** (1.40 g, 10 mmol) by GP 1; yield: 1.33 g (70%); R_f = 0.22 (10% EtOAc-hexanes).

^1H NMR: δ = 8.19 (s, 1 H), 4.76 (t, *J* = 5.6 Hz, 1 H), 3.52 (s, 3 H), 1.60-1.80 (m, 2 H), 1.20-1.50 (m, 12 H), 0.90 (t, *J* = 6.8 Hz, 3 H).

^{13}C NMR: δ = 108.8, 55.8, 31.9, 31.3, 29.43, 29.38, 29.3, 24.6, 22.7, 14.1.

9-Hydroperoxy-9-methoxynonanol Acetate Ester (2b) [1255126-65-8]

Prepared from **2a** (2.0 g, 10 mmol) by GP 1; yield: 1.66 g (67%); R_f = 0.33 (25% EtOAc-hexanes).

^1H NMR (300 MHz): δ = 9.46 (s, 1 H), 4.66 (t, *J* = 5.8 Hz, 1 H), 3.97 (t, *J* = 6.7 Hz, 2 H), 3.43 (s, 3 H), 1.98 (s, 3 H), 1.66-1.48 (m, 4 H), 1.33-1.23 (m, 10 H).

^{13}C NMR (75 MHz): δ = 171.6, 108.5, 64.7, 55.7, 31.3, 29.2, 29.1, 28.9, 28.4, 25.7, 24.5, 20.9.

4-Benzyloxy-1-methoxybutyl Hydroperoxide (3b) [1255126-67-0]

Prepared from **3a** (1.76 g, 10 mmol) by GP 1; yield: 1.56 g (69%); R_f = 0.37 (25% EtOAc-hexanes).

^1H NMR: δ = 8.93 (s, 1 H), 7.39-7.29 (m, 5 H), 4.77-4.74 (m, 1 H), 4.54 (s, 2 H), 3.58-3.50 (s, 5 H), 1.93-1.71 (m, 4 H).

^{13}C NMR: δ = 138.1, 128.4, 127.8, 127.7, 108.4, 72.9, 69.8, 55.9, 28.0, 24.6.

(3-Hydroperoxy-3-methoxybutyl)benzene (4b)

Prepared from 4a (1.0 g, 5.6 mmol) by GP 1; yield: 0.427 g (38%); R_f = 0.36 (25% EtOAc-hexanes).

IR (neat): 3345, 2943, 1102, 1069, 697 cm^{-1} .

^1H NMR: δ = 7.49 (s, 1 H), 7.31-7.22 (m, 5 H), 3.37 (s, 3 H), 2.76-2.69 (m, 2 H), 2.08-2.02 (m, 2 H), 1.45 (s, 3 H).

^{13}C NMR: δ = 141.7, 128.5, 128.3, 126.0, 106.9, 49.1, 36.9, 30.5, 19.1.

HRMS-ESI: m/z calcd for $\text{C}_{11}\text{H}_{16}\text{O}_3 + \text{Na}$ ($\text{M} + \text{Na}$) $^+$: 219.0997; found: 219.1007.

4-(*t*-Butyl)-1-hydroperoxy-1-methoxycyclohexane (5b) [169294-55-7]

Prepared as a mixture of diastereomers from 5a (1.52 g, 10 mmol) by GP 1; yield: 1.49 g (74%); R_f = 0.41 (20% EtOAc-hexanes).

^1H NMR: δ = 7.50 (s, 0.25 H), 7.48 (s, 0.68 H), 3.34 (s, 0.79 H), 3.31 (s, 2.18 H), 2.20-2.28 (m, 0.54 H), 2.09-2.19 (m, 1.52 H), 1.66-1.77 (m, 2 H), 1.33-1.46 (m, 2 H), 1.12-1.32 (m, 2 H), 1.00-1.11 (m, 1 H), 0.89 (s, 9 H).

^{13}C NMR: δ = 105.7, 105.4, 48.5, 48.3, 47.6, 47.5, 32.3, 31.5, 30.9, 27.6, 23.6, 23.4.

2-Hydroperoxy-2-methoxyadamantane (6b) [55975-21-8]

Prepared from 6a (1.0 g, 5.61 mmol) by GP 1; yield: 0.700 g (63%); R_f = 0.50 (25% EtOAc-hexanes).

^1H NMR: δ = 7.34 (s, 1 H), 3.32 (s, 3 H), 2.23 (m, 2 H), 2.01-1.86 (m, 6 H), 1.70-1.66 (m, 6 H).

^{13}C NMR: δ = 107.5, 47.4, 37.1, 33.71, 33.68, 31.9, 27.0, 26.9.

Carbonyls from HydroperoxyacetalsNonanal (1c) [124-19-6]

Prepared from 1b (0.095 g, 0.5 mmol) by GP 2; reaction time: 2 h; yield: 0.045 g (63%); R_f = 0.66 (10% EtOAc-hexanes).

^1H NMR: δ = 9.78 (t, J = 1.8 Hz, 1 H), 2.43 (dt, J = 1.8, 7.3 Hz, 2 H), 1.64 (m, 2 H), 1.31-1.28 (m, 10 H), 0.89 (t, J = 6.9 Hz, 3 H).

^{13}C NMR: δ = 203.0, 43.9, 31.8, 29.3, 29.2, 29.1, 22.6, 22.0, 14.0.

9-Hydroxynonanal Acetate Ester (2c) [29541-97-7]

Prepared from 2b (0.496 g, 2 mmol) by GP 2; reaction time: 2 h; yield: 0.341 g (85%); R_f = 0.44 (25% EtOAc-hexanes).

^1H NMR: δ = 9.78 (t, J = 1.8 Hz, 1 H), 4.07 (t, J = 6.8 Hz, 2 H), 2.45 (dt, J = 1.8, 7.3 Hz, 2 H), 2.07 (s, 3 H), 1.64 (m, 4 H), 1.34 (m, 8 H).

^{13}C NMR: δ = 202.7, 171.1, 64.5, 43.8, 29.1, 28.97, 28.95, 28.5, 25.8, 21.9, 20.9.

4-(Benzyloxy)butanal (3c) [5470-84-8]

Prepared from 3b (0.107 g, 0.47 mmol) by GP 2; reaction time: 2 h; yield: 0.076 g (91%); R_f = 0.33 (25% EtOAc-hexanes).

^1H NMR: δ = 9.79 (t, J = 1.5 Hz, 1 H), 7.39-7.27 (m, 5 H), 4.51 (s, 2 H), 3.53 (t, J = 6.0 Hz, 2 H), 2.58 (dt, J = 1.32, 7.1 Hz, 2 H), 1.98 (quint, J = 6.5 Hz, 2 H).

^{13}C NMR: δ = 202.3, 138.3, 128.4, 127.6, 72.9, 69.1, 40.9, 22.6.

4-Phenylbutan-2-one (4c) [2550-26-7]

Prepared from 4b (0.098 g, 0.5 mmol) by GP 2; reaction time: 5 h; yield: 0.052 g (70%); R_f = 0.37 (25% EtOAc-hexanes).

^1H NMR: δ = 7.33-7.29 (m, 2 H), 7.24-7.20 (m, 3 H), 2.92 (t, J = 7.6 Hz,

2 H), 2.79 (t, J = 7.6 Hz, 2 H), 2.16 (s, 3 H).

^{13}C NMR: δ = 207.9, 141.0, 128.5, 128.3, 126.1, 45.2, 30.1, 29.7.

4-(*tert*-Butyl)cyclohexanone (5c) [98-53-3]

Prepared from 5b (0.101 g, 0.5 mmol) by GP 2; reaction time: 5 h; yield: 0.053 g (68%); R_f = 0.50 (25% EtOAc-hexanes).

^1H NMR: δ = 2.40-2.26 (m, 4 H), 2.09-2.06 (m, 2 H), 1.52-1.38 (m, 3 H), 0.91 (s, 9 H).

^{13}C NMR: δ = 212.5, 46.7, 41.3, 32.4, 27.6.

Adamantan-2-one (6c) [700-58-3]

Prepared from 6b (0.099 g, 0.5 mmol) by GP 2; reaction time: 8 h; yield: 0.044 g [68% based on 0.015 g (15%) starting material recovery]; R_f = 0.47 (25% EtOAc-hexanes).

^1H NMR: δ = 2.54 (m, 2 H), 2.09-1.92 (m, 12 H).

^{13}C NMR: δ = 218.4, 46.9, 39.2, 36.3, 27.4.

Amines from Hydroperoxyacetals9-Morpholinononanol Acetate Ester (2d)

Prepared from 2b (0.124 g, 0.5 mmol) with morpholine (0.5 mmol) by GP 3; reaction time: 5 h; yield: 0.115 g (85%); R_f = 0.32 (10% MeOH- CH_2Cl_2).

IR (neat): 2927, 2853, 1738, 1118 cm^{-1} .

^1H NMR: δ = 4.05 (t, J = 6.8 Hz, 2 H), 3.72 (t, J = 4.4 Hz, 4 H), 2.44 (m, 4 H), 2.32 (t, J = 7.4 Hz, 2 H), 2.04 (s, 3 H), 1.65-1.57 (m, 2 H), 1.48 (m, 2 H), 1.35-1.29 (m, 10 H).

^{13}C NMR: δ = 171.2, 66.9, 64.6, 59.2, 53.8, 29.42, 29.38, 29.1, 28.5, 27.4, 26.5, 25.8, 20.9.

HRMS-FAB: m/z calcd for $\text{C}_{15}\text{H}_{30}\text{NO}_3$ ($\text{M} + \text{H}$) $^+$: 272.2226; found: 272.2213.

N-Phenyl-9-aminononanol Acetate Ester (2e)

Prepared from 2b (0.124 g, 0.5 mmol) with aniline (0.5 mmol) by GP 3; reaction time: 5 h; yield: 0.104 g (75%); R_f = 0.50 (10% EtOAc-hexanes).

IR (neat): 3401, 2926, 2854, 1734, 1236 cm^{-1} .

^1H NMR: δ = 7.22-7.18 (m, 2 H), 6.74-6.62 (m, 3 H), 4.09 (t, J = 6.76 Hz, 2 H), 3.66 (s, 1 H), 3.13 (t, J = 7.16 Hz, 2 H), 2.08 (s, 3 H), 1.68-1.63 (m, 4 H), 1.43-1.36 (m, 10 H).

^{13}C NMR: δ = 171.3, 148.6, 129.2, 117.0, 112.7, 64.6, 43.9, 29.6, 29.5, 29.4, 29.2, 28.6, 27.2, 25.9, 21.1.

HRMS-FAB: m/z calcd for $\text{C}_{17}\text{H}_{28}\text{NO}_2$ ($\text{M} + \text{H}$) $^+$: 278.2115; found: 278.2123.

N-Benzyl-9-aminononanol Acetate Ester (2f)

Prepared from 2b (0.124 g, 0.5 mmol) with benzylamine (2.5 mmol) by GP 3; reaction time: 5 h; yield: 0.098 g (67%); R_f = 0.42 (10% MeOH- CH_2Cl_2).

IR (neat): 2923, 2856, 1737, 1236 cm^{-1} .

^1H NMR: δ = 7.39-7.25 (m, 5 H), 4.21 (s, 1 H), 4.04 (t, J = 6.8 Hz, 2 H), 3.84 (s, 2 H), 2.65 (t, J = 7.5 Hz, 2 H), 2.04 (s, 3 H), 1.62-1.59 (m, 4 H), 1.28 (m, 10 H).

^{13}C NMR: δ = 171.2, 138.2, 128.6, 128.5, 127.4, 64.6, 53.3, 48.7, 29.4, 29.3, 29.1, 28.6, 27.2, 25.9, 21.0.

HRMS-ESI: m/z calcd for $\text{C}_{15}\text{H}_{29}\text{NO}_2 + \text{Na}$ ($\text{M} + \text{Na}$) $^+$: 314.2091; found: 314.2110.

***N,N'*-(Bis-9-acetoxynonyl)benzylamine**

The tertiary amine, *N,N'*-(bis-9-acetoxynonyl)benzylamine, resulting from the reductive amination of **2f** by **2b** was also isolated in 8% yield; $R_f = 0.65$ (10% MeOH-CH₂Cl₂).

IR (neat): 2925, 2855, 1738, 1232, 1043 cm⁻¹.

¹H NMR: $\delta = 7.35$ – 7.22 (m, 5 H), 4.06 (t, $J = 6.8$ Hz, 4 H), 3.56 (s, 2 H), 2.40 (t, $J = 7.2$ Hz, 4 H), 2.06 (s, 6 H), 1.66–1.59 (m, 4 H), 1.47–1.45 (m, 4 H), 1.34–1.27 (20 H).

¹³C NMR: $\delta = 171.2$, 140.5, 128.8, 128.0, 126.6, 64.6, 58.6, 53.8, 29.5, 29.4, 29.2, 28.6, 27.4, 27.0, 25.9, 21.0.

HRMS-FAB Calcd for C₂₉H₅₀NO₄ (M + H)⁺: 476.3740; found: 476.3725.

***N*-Phenethyl-9-aminononanol Acetate Ester (2g)**

Prepared from **2b** (0.124 g, 0.5 mmol) with phenethylamine (2 mmol) by GP 3; reaction time: 5 h; yield: 0.096 g (63%); $R_f = 0.60$ (10% MeOH-CH₂Cl₂).

IR (neat): 3676, 2988, 2901, 1736 cm⁻¹.

¹H NMR: $\delta = 7.32$ – 7.19 (m, 5 H), 4.06 (t, $J = 6.7$ Hz, 2 H), 2.91–2.87 (m, 2 H), 2.84–2.80 (m, 2 H), 2.62 (t, $J = 7.3$ Hz, 2 H), 2.06 (s, 3 H), 1.62 (q, $J = 7.2$ Hz, 2 H), 1.47 (m, 2 H), 1.29 (m, 10 H).

¹³C NMR: $\delta = 171.3$, 140.2, 128.7, 128.4, 126.1, 64.6, 51.3, 49.9, 36.5, 30.1, 29.5, 29.4, 29.2, 28.6, 27.3, 25.9, 21.0.

HRMS-FAB: m/z calcd for C₁₉H₃₂NO₂ (M + H)⁺: 306.2428; found: 306.2439.

***4*-[4-(Benzyloxy)butyl]morpholine (3d)**

Prepared from **3b** (0.107 g, 0.47 mmol) with morpholine (0.5 mmol) by GP 3; reaction time: 5 h; yield: 0.104 g (89%); $R_f = 0.33$ (5% MeOH-CH₂Cl₂).

IR (neat): 2942, 2853, 2806, 1116 cm⁻¹.

¹H NMR: $\delta = 7.34$ – 7.27 (m, 5 H), 4.50 (s, 2 H), 3.72 (t, $J = 4.7$ Hz, 4 H), 3.49 (t, $J = 6.1$ Hz, 2 H), 2.44 (m, 4 H), 2.35 (t, $J = 7.2$ Hz, 2 H), 1.66–1.56 (m, 4 H).

¹³C NMR: $\delta = 138.6$, 128.4, 127.6, 127.5, 72.9, 70.1, 66.9, 58.8, 53.7, 27.7, 23.2.

HRMS-FAB: m/z calcd for C₁₅H₂₄NO₂ (M + H)⁺: 250.1802; found: 250.1812.

***N*-[4-(Benzyloxy)butyl]aniline (3e)**

Prepared from **3b** (0.107 g, 0.47 mmol) with aniline (0.5 mmol) by GP 3; reaction time: 4 h; yield: 0.083 g (69%); $R_f = 0.51$ (20% EtOAc-hexanes).

IR (neat): 3403, 3026, 2935, 2858, 1601, 1505 cm⁻¹.

¹H NMR: $\delta = 7.38$ – 7.30 (m, 5 H), 7.21–7.17 (m, 2 H), 6.71 (t, $J = 7.3$ Hz, 1 H), 6.61–6.59 (m, 2 H), 4.54 (s, 2 H), 3.73 (s, 1 H), 3.55 (t, $J = 5.9$ Hz, 2 H), 3.18–3.15 (m, 2 H), 1.79–1.57 (m, 4 H).

¹³C NMR: $\delta = 148.6$, 138.6, 129.3, 128.5, 127.8, 127.7, 117.2, 112.8, 73.1, 70.2, 43.8, 27.5, 26.5.

HRMS-FAB: m/z calcd for C₁₇H₂₂NO (M + H)⁺: 256.1696; found: 256.1692.

***N*-[4-(Benzyloxy)butyl]benzylamine (3f) [60058-23-3]**

Prepared from **3b** (0.107 g, 0.47 mmol) with benzylamine (2.5 mmol) by GP 3; reaction time: 5 h; yield: 0.079 g (63%); $R_f = 0.51$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.39$ – 7.24 (m, 10 H), 4.52 (s, 2 H), 3.80 (s, 2 H), 3.50 (t, $J = 6.2$ Hz, 2 H), 2.67 (t, $J = 6.9$ Hz, 2 H), 1.73–1.59 (m, 4 H), 1.55 (s, 1 H).

¹³C NMR: $\delta = 140.5$, 138.6, 128.4, 128.3, 128.1, 127.6, 127.5, 126.9, 72.9, 70.3, 54.0, 49.2, 27.6, 26.8.

***N*-[4-(Benzyloxy)butyl]-2-phenethylamine (3g)**

Prepared from **3b** (0.107 g, 0.47 mmol) with phenethylamine (2.5 mmol) by GP 3; reaction time: 4 h; yield: 0.087 g (65%); $R_f = 0.22$ (5% MeOH-CH₂Cl₂).

IR (neat): 3026, 2932, 2854, 1097, 696 cm⁻¹.

¹H NMR: $\delta = 7.37$ – 7.22 (m, 10 H), 4.52 (s, 2 H), 3.50 (t, $J = 6.2$ Hz, 2 H), 2.92–2.88 (m, 2 H), 2.84–2.80 (m, 2 H), 2.68–2.65 (t, $J = 7.1$ Hz, 2 H), 1.69–1.55 (m, 4 H), 1.37 (s, 1 H).

¹³C NMR: $\delta = 140.1$, 138.6, 128.7, 128.5, 128.4, 127.6, 127.5, 126.1, 72.9, 70.3, 51.2, 49.7, 36.5, 27.6, 26.9.

HRMS-FAB: m/z calcd for C₁₉H₂₆NO (M + H)⁺: 284.2009; found: 284.2018.

***4*-(4-Phenylbutan-2-yl)morpholine (4d) [2832-95-3]**

Prepared from **4b** (0.098 g, 0.5 mmol) with morpholine (0.5 mmol) by GP 3; reaction time: 24 h; yield: 0.049 g (45%); $R_f = 0.37$ (5% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.32$ – 7.18 (m, 5 H), 3.75–3.72 (m, 4 H), 2.75–2.62 (m, 2 H), 2.60–2.52–2.44 (m, 5 H), 1.90–1.83 (m, 1 H), 1.66 (m, 1 H), 1.64–1.54 (m, 1 H), 1.03 (d, $J = 6.7$ Hz, 2 H).

¹³C NMR: $\delta = 142.6$, 128.4, 128.3, 125.7, 67.5, 58.4, 48.7, 35.3, 32.8, 13.9.

***N*-(4-Phenylbutan-2-yl)aniline (4e) [72641-00-0]**

Prepared from **4b** (0.098 g, 0.5 mmol) with aniline (0.5 mmol) by GP 3; reaction time: 36 h; yield: 0.068 g (60%); $R_f = 0.51$ (10% EtOAc-hexanes).

¹H NMR: $\delta = 7.33$ – 7.28 (m, 2 H), 7.23–7.15 (m, 5 H), 6.69 (t, $J = 7.3$ Hz, 1 H), 6.56 (d, $J = 8.2$ Hz, 2 H), 3.52 (m, 1 H), 3.45 (s, 1 H), 2.75 (t, $J = 7.8$ Hz, 2 H), 1.95–1.86 (m, 1 H), 1.84–1.75 (m, 1 H), 1.24 (d, $J = 6.3$ Hz, 3 H).

¹³C NMR: $\delta = 147.6$, 142.0, 129.3, 128.5, 128.4, 125.9, 116.9, 113.2, 47.9, 38.8, 32.5, 20.9.

***N*-Benzyl-4-phenylbutan-2-amine (4f) [68164-04-5]**

Prepared from **4b** (0.098 g, 0.5 mmol) with benzylamine (0.5 mmol) by GP 3; reaction time: 36 h; yield: 0.089 g (75%); $R_f = 0.25$ (5% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.36$ – 7.20 (m, 10 H), 3.87 (d, $J = 13.0$ Hz, 1 H), 3.77 (d, $J = 13.0$ Hz, 1 H), 2.82–2.64 (m, 3 H), 1.90–1.81 (m, 1 H), 1.76–1.66 (m, 1 H), 1.41 (s, 1 H), 1.19 (d, $J = 6.4$ Hz, 3 H).

¹³C NMR: $\delta = 142.5$, 140.7, 128.44, 128.39, 128.2, 127.9, 126.9, 125.7, 52.0, 51.3, 38.7, 32.3, 20.4.

***N*-Phenethyl-4-phenylbutan-2-amine (4g) [161012-69-7]**

Prepared from **4b** (0.138 g, 0.7 mmol) with phenethylamine by GP 3; reaction time: 30 h; yield: 0.092 g (52%); $R_f = 0.30$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.36$ – 7.15 (m, 10 H), 2.99–2.91 (m, 1 H), 2.88–2.79 (m, 3 H), 2.72–2.66 (m, 1 H), 2.64–2.57 (m, 2 H), 1.84–1.75 (m, 1 H), 1.68–1.59 (m, 1 H), 1.30 (s, 1 H), 1.13 (d, $J = 6.3$ Hz, 3 H).

¹³C NMR: $\delta = 142.3$, 140.1, 128.7, 128.5, 128.4, 128.3, 126.2, 125.7, 55.4, 48.5, 38.7, 36.6, 32.3, 20.3.

Amines from Alkenes 1-Benzylazapine (7) [20422-13-3]

Prepared by reacting cyclohexene (0.082 g, 1 mmol) with benzylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.107 g (57%); $R_f = 0.57$ (10% MeOH-CH₂Cl₂).

¹H NMR: $\delta = 7.40$ – 7.27 (m, 5 H), 3.69 (s, 2 H), 2.67–2.66 (m, 4 H), 1.67 (m, 8 H).

¹³C NMR: $\delta = 140.1$, 128.8, 128.1, 126.7, 62.7, 55.6, 28.2, 27.0.

1-Phenethylazapine (8) [65530-43-0]

Prepared by reacting cyclohexene (0.082 g, 1 mmol) with phenethylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.127 g (63%); R_f = 0.57 (10% MeOH-CH₂Cl₂).

¹H NMR: δ = 7.32-7.21 (m, 5 H), 2.83-2.80 (m, 8 H), 1.72 (m, 4 H), 1.64 (m, 4 H).

¹³C NMR: δ = 140.2, 128.8, 128.4, 126.0, 59.9, 55.1, 33.6, 27.2, 27.1.

4-[4-(Benzyloxy)butyl]morpholine (3d)

Prepared from 3a (0.176 g, 1.0 mmol) with morpholine (1 mmol) by GP 4A; reaction time: 2 h; yield: 0.159 g (64%). Spectral data were identical with those of compound 3d described above.

1-Benzylpiperidine (9) [2905-56-8]

Prepared by reacting cyclopentene (0.068 g, 1 mmol) with benzylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.113 g (65%); R_f = 0.50 (10% MeOH-CH₂Cl₂).

¹H NMR: δ = 7.35-7.24 (m, 5 H), 3.50 (s, 2 H), 2.40-2.39 (m, 4 H), 1.64-1.57 (m, 4 H), 1.49-1.46 (m, 2 H).

¹³C NMR: δ = 138.7, 129.2, 128.0, 126.8, 63.9, 54.5, 26.0, 24.4.

1-Phenethylpiperidine (10) [332-14-9]

Prepared by reacting cyclopentene (0.068 g, 1 mmol) with phenethylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.136 g (72%); R_f = 0.55 (10% MeOH-CH₂Cl₂).

¹H NMR: δ = 7.32-7.20 (m, 5 H), 2.86-2.82 (m, 2 H), 2.60-2.50 (m, 2 H), 2.40 (m, 4 H), 1.68-1.62 (m, 4 H), 1.51-1.48 (m, 2 H).

¹³C NMR: δ = 140.7, 128.7, 128.3, 125.9, 61.5, 54.6, 33.7, 26.1, 24.5.

Compound 10 was also prepared by reacting cyclopentene (0.068 g, 1 mmol) with phenethylamine (1 mmol) according to GP 4B; yield: 0.117 g (62%).

9-(N-Morpholino)nonyl Acetate Ester (2d)

Prepared from 2a (0.099 g, 0.5 mmol) with morpholine (1 mmol) by GP 4B; reaction time: 6 h. The product was identical to 2d obtained as above; yield: 0.089 g (66%).

N-Benzylcyclohexylmethanamine (11a) [4352-47-0] and N-benzyl-N-methylcyclohexylmethanamine (11b) [79952-95-7]

Prepared from the reaction of vinylcyclohexane (0.11 g, 1 mmol) with benzylamine (2 mmol) by GP 4B; reaction time: 6 h; yield: 0.102 g (50%); R_f = 0.50 (10% MeOH-CH₂Cl₂).

¹H NMR: δ = 7.36-7.26 (m, 5 H), 3.80 (s, 2 H), 2.49 (d, J = 6.7 Hz, 2 H), 1.84-1.68 (m, 5 H), 1.57-1.46 (m, 1 H), 1.32-1.13 (m, 4 H), 0.99-0.90 (m, 2 H).

¹³C NMR: δ = 140.6, 128.4, 128.0, 126.8, 56.2, 54.1, 37.9, 31.5, 26.7, 26.1.

The reaction also yielded 10% of 11b.

11b

R_f = 0.63 (10% MeOH-CH₂Cl₂).

¹H NMR (600 MHz): δ = 7.35-7.24 (m, 5 H), 3.47 (s, 2 H), 2.18-2.16 (m, 5 H), 1.84 (d, J = 12.4 Hz, 2 H), 1.74-1.67 (m, 3 H), 1.57-1.51 (m, 1 H), 1.28-1.13 (m, 3 H), 0.89-0.82 (m, 2 H).

¹³C NMR (150 MHz): δ = 139.8, 128.9, 128.0, 126.7, 64.7, 62.7, 42.9, 35.8, 31.8, 26.9, 26.2.

3-Phenethyl-3-azabicyclo[3.2.1]octane (12)

Prepared by reacting norbornene (0.094 g, 1 mmol) with phenethylamine (1 mmol) according to GP 4A; reaction time: 2 h; yield: 0.139 g (65%); R_f = 0.63 (10% MeOH-CH₂Cl₂).

IR (neat): 2931, 2757, 1496, 1406 cm⁻¹.

¹H NMR: δ = 7.37-7.24 (m, 5 H), 2.84-2.80 (m, 4 H), 2.64-2.60 (m, 2 H), 2.20-2.14 (m, 4 H), 1.76-1.63 (m, 4 H), 1.56-1.53 (m, 1 H), 1.44-1.42 (m, 1 H).

¹³C NMR: δ = 140.9, 128.8, 128.2, 125.8, 60.2, 60.1, 37.7, 35.2, 33.3, 28.6.

HRMS-FAB: m/z calcd for C₁₅H₂₂N (M + H)⁺: 216.1747; found: 216.1748.

This product was also prepared by reacting norbornene (0.041 g, 0.43 mmol) with phenethylamine (0.43 mmol) according to GP 4B; yield: 0.062 g (65%).

1-Phenyl-2-(4-phenylbutan-2-yl)diazene (13) [343222-12-8]

A solution of alkene 4a (0.176 g, 1 mmol) and MeOH (0.3 mL) in CH₂Cl₂ (10 mL) in a round-bottomed flask was cooled to -40 °C and a stream of 2% O₃/O₂ (approximately 1 mmol O₃/min) was bubbled through the reaction solution. Once the pale blue color of ozone was observed, the ozonizer voltage was set to zero and the reaction was sparged for 1 min with N₂. The resulting solution was removed from dry ice/acetone bath, whereupon NaBH(OAc)₃ (2 mmol) and phenylhydrazine (1 mmol) were sequentially added. The reaction was stirred for 2 h at r.t. and then treated with NaCNBH₃ (1 mmol). After stirring for another 30 min, the reaction was filtered through neutral alumina with CH₂Cl₂, and the residue obtained upon concentration was purified by chromatography (alumina) using 10% EtOAc-hexanes to furnish a light yellow oil, which darkened very rapidly. Air was passed over the oil for 2 h to furnish diazene 13 as a yellow oil; yield: 0.135 g (57%); R_f = 0.50 (10% EtOAc-hexanes).

¹H NMR: δ = 7.73-7.70 (m, 2 H), 7.53-7.46 (m, 3 H), 7.33-7.28 (m, 2 H), 7.24-7.20 (m, 3 H), 3.90-3.81 (m, 1 H), 2.66 (t, J = 8.1 Hz, 2 H), 2.41-2.32 (m, 1 H), 2.13-2.04 (m, 1 H), 1.42 (d, J = 6.6 Hz, 3 H).

¹³C NMR: δ = 152.1, 141.9, 130.3, 129.0, 128.5, 128.4, 125.8, 122.2, 72.7, 36.9, 32.6, 19.0.

Supporting Information for this article follows the **References**.

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Supporting Information: A Mild One-pot Conversion of Alkenes to Amines through Tandem Ozonolysis and Reductive Amination.

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1b

1D Proton NMR
TFish0460
CDCl₃, 100MHz

¹³C
TFish0460
CDCl₃, 100MHz

1D Proton
ksk01-08

2b

13C
ksk01-08

3b

1D Proton NMR
ksk01-94
400 MHz, CDCl₃

¹³C
ksk01-94
CDCl₃, 400MHz

1D Proton NMR
ksk01-79

¹³C
ksk01-79

1D Proton NMR
TFish0580
CDCl₃, 400MHz

¹³C
TFish 0580
CDCl₃, 400MHz

1D Proton NMR
KSK01-77

¹³C
KSK01-77

1D Proton NMR
ksk01-66
CDCl₃, 400 MHz

¹³C
ksk01-66
CDCl₃, 400 MHz

1D Proton NMR
ksk01-20
CDCl₃, 400 MHz

¹³C
ksk01-20

CDC13, 400 MHz
ID Proton
ksk01-68

¹³C
ksk01-68

1D Proton NMR
ksk01-80
CDCl₃, 400 MHz

¹³C
ksk01-80

1D Proton NMR
ksk01-67
CDCl₃, 400 MHz

¹³C
ksk01-67

6c

1D Proton NMR
ksk01-78

¹³C
ksk01-78

1D Proton NMR
ksk01-49

¹³C
ksk01-49

1D Proton NMR
ksk01-82

¹³C
ksk01-82

1D Proton NMR
ksk01-32 f2

¹³C
ksk01-32 f2

1D Proton NMR
ksk01-32 f1

¹³C
ksk01-32 f1

1D Proton NMR
ksk01-90

¹³C
ksk01-90

1D Proton NMR
ksk01-71
400MHz, CDCl₃

¹³C
ksk01-71

1D Proton NMR
ksk01-81
CDCl₃, 400 MHz

13C ksk01-81
CDCl₃

1D Proton NMR
ksk01-83

¹³C
ksk01-83

1D Proton NMR
ksk01-95

¹³C
ksk01-95

1D Proton NMR
ksk01-86

13C
ksk01-86

1D Proton NMR
ksk01-87

¹³C
ksk01-87

1D Proton NMR
ksk01-91

¹³C
ksk01-91

1D Proton NMR
ksk01-96

¹³C
ksk01-96

1D Proton NMR
ksk02-63
400 MHz, CDCl₃

¹³C
ksk02-63
400 MHz, CDCl₃

1D Proton NMR
ksk02-64
400 MHz, CDCl₃

¹³C
ksk02-64
400MHz, CDCl₃

9

1D Proton
ksk02-60
CDC13, 400 MHz

13C
ksk02-60
400 MHz, CDC13

1D Proton NMR
ksk01-92

¹³C
ksk01-92

1D Proton NMR
ksk02-95 f2

¹³C
ksk02-95 f2

11b

1D Proton NMR
ksk02-95 f1

¹³C NMR
ksk02-95 f1

12

1D Proton NMR
ksk02-89

¹³C
ksk02-89

1D Proton NMR
ksk01-40
CDCl₃, 400 MHz

¹³C
ksk02-40
CDCl₃, 400 MHz