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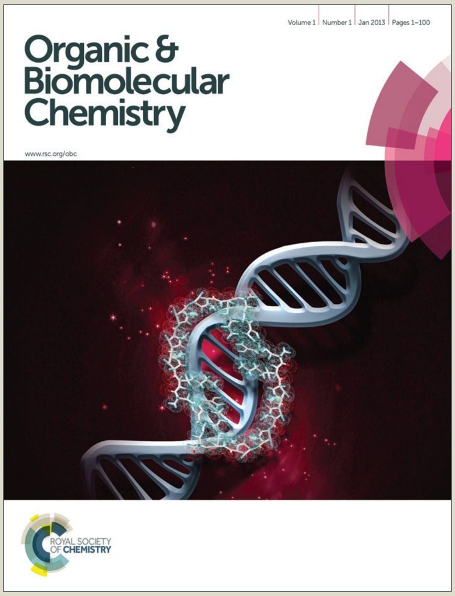

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Iron(III)-catalyzed aerobic dioxygenation of Styrenes Using *N*-hydroxyphthalimide and *N*-hydroxybenzotriazole

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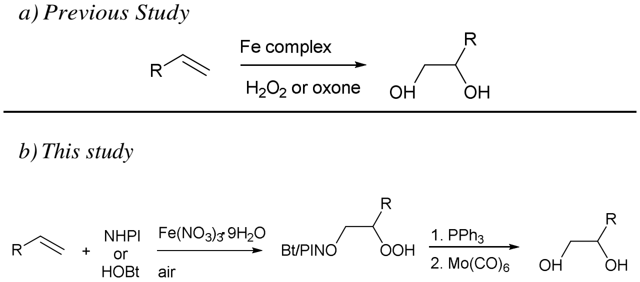
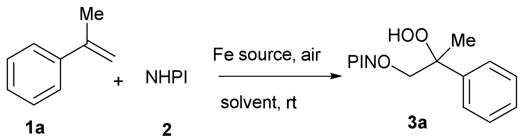
Iron-catalyzed dioxygenation of alkenes with air using *N*-hydroxyphthalimide (NHPI) and *N*-hydroxybenzotriazole (HOBt) is described at room temperature. The products can be readily converted into 1,2-vicinal diols using PPh_3 and $Mo(CO)_6$. The use of $Fe(NO_3)_3 \bullet 9H_2O$ as the catalyst, mild reaction conditions and the selectivity are the significant practical features. The radical scavanger experiment suggests that the reaction proceeds via a radical intermediate. The synthetic and mechanistic aspects have been presented.

Iron is relatively less-toxic and the second most abundant (4.7 wt %) metal in the earth crust.[1] In addition, iron is essential key element in various biological systems, for examples, it takes part in metalloproteins for the transport or metabolism of small molecules (oxygen, methane etc.) and electron transfer reactions.[2] Considerable effort has thus been made on the development of iron-based functional models.[3] Among them, the dioxygenation of alkenes is attractive due to their broad interests in synthetic chemistry.[4] For examples, iron-catalyzed dioxygenation of alkenes has been considerably explored using H_2O_2 and oxone as the oxidants (Scheme 1a).[4] Herein, we wish to report an efficient aerobic iron-catalyzed hydroperoxidation of alkenes using NHPI and HOBt at room temperature (Scheme 1b). The selectivity, mild reaction conditions and the use of air as the oxygen source[5] are the significant practical advantages. The hydroperoxides can be transformed into 1,2-diols using PPh_3 and $Mo(CO)_6$ in high yields.



Scheme 1. Iron-catalyzed dioxygenation of alkenes.

Table 1. Optimization of the reaction conditions.



Entry	Iron source	Solvent	Yield (%)[a,b]
1	$Fe(NO_3)_3·9H_2O$	DCE	81
2	$Fe(NO_3)_3·9H_2O$	toluene	22
3	**$Fe(NO_3)_3·9H_2O$**	**CH_2Cl_2**	**84**
4	$Fe(NO_3)_3·9H_2O$	DMSO	trace
5	$Fe(NO_3)_3·9H_2O$	H_2O	n.d.
6	$Fe(NO_3)_3·9H_2O$	CH_2Cl_2	78[c]
7	$Fe(NO_3)_3·9H_2O$	CH_2Cl_2	trace[d]
8	$Fe(acac)_3$	CH_2Cl_2	72
9	$FeBr_2$	CH_2Cl_2	n.d.
10	$K_3Fe(CN)_6$	CH_2Cl_2	34
11	$Fe(NO_3)_3·9H_2O$	CH_2Cl_2	67[e]
12	$Fe(NO_3)_3·9H_2O$	CH_2Cl_2	71[f]
13	-	CH_2Cl_2	n.d.

[a] Reaction conditions: alkene **1a** (0.5 mmol), NHPI **2** (0.25 mmol), $Fe(NO_3)_3·9H_2O$ (20 mol %), solvent (1.5 mL), rt, 9 h, air. [b] Isolated yield. [c] O_2 balloon used. [d] N_2 balloon used. [e] 15 mol % $Fe(NO_3)_3·9H_2O$ used. [f] 1.5 Equiv **1a** used. n.d.= not detected.

Catalytic vicinal dioxygenation of alkenes is a critical process in both academia and pharmaceutical industries. The OsO_4 catalysed (or Upjohn) dihydroxylation is among the commonly used for this purpose.[6] Recently, Sigman and co-workers have reported Pd-catalyzed dialkoxylation of alkenes with molecular oxygen.[7a] Subsequently, diacetoxylation of alkenes has been accomplished using Pd[7b-c] or Cu[7d] based catalytic systems with $PhI(OAc)_2$ or molecular oxygen, while dilauroyl peroxide is used for the oxidation of alkenes to produce *β*-hydroxy-*N*-alkoxy carbamates under molecular oxygen.[8] Later, Cu[9] and I_2-tBOOH[10] based catalytic systems have been investigated for

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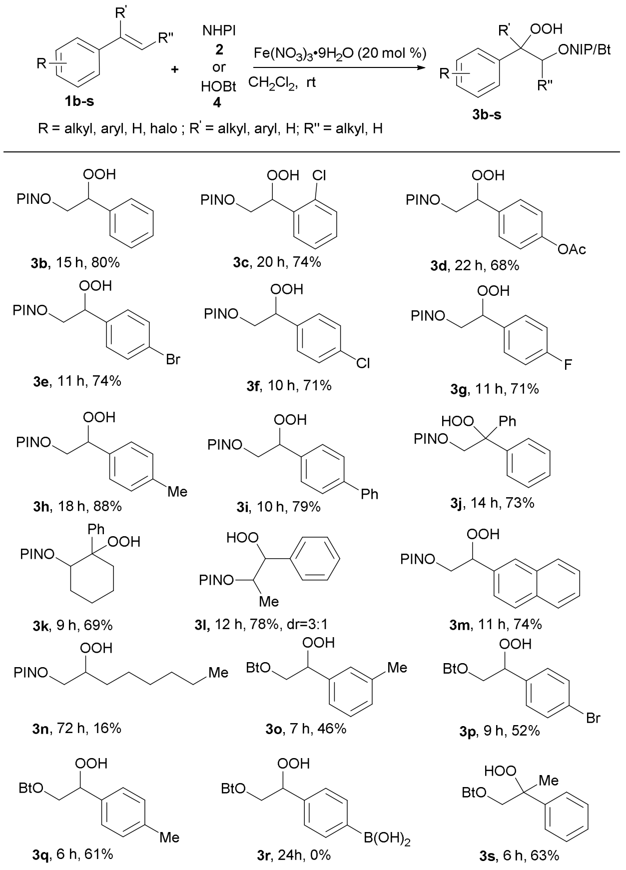
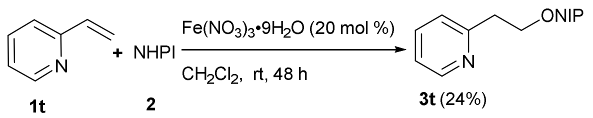
Electronic Supplementary Information (ESI) available: [Crystal structure and data of **5i** and NMR spectra (^1H and ^{13}C) of **3a-t**, **5a-m**, **6a-b**, **7a-h** and **8**]. See DOI: 10.1039/x0xx00000x the dioxygeantion of alkenes with molecular oxygen to give *β*-keto-/*β*-hydroxy-*N*-alkoxyamines.

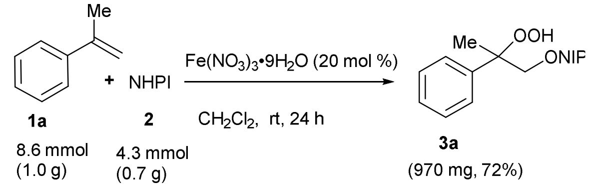


Scheme 2. Reaction conditions: alkene **1b-s** (0.5 mmol), NHPI **2** or HOBT **4** (0.25 mmol), $Fe(NO_3)_3{\cdot}9H_2O$ (20 mol %), CH_2Cl_2 (1.5 mL), rt, air. Yields are isolated. dr Determined by ^1H NMR.

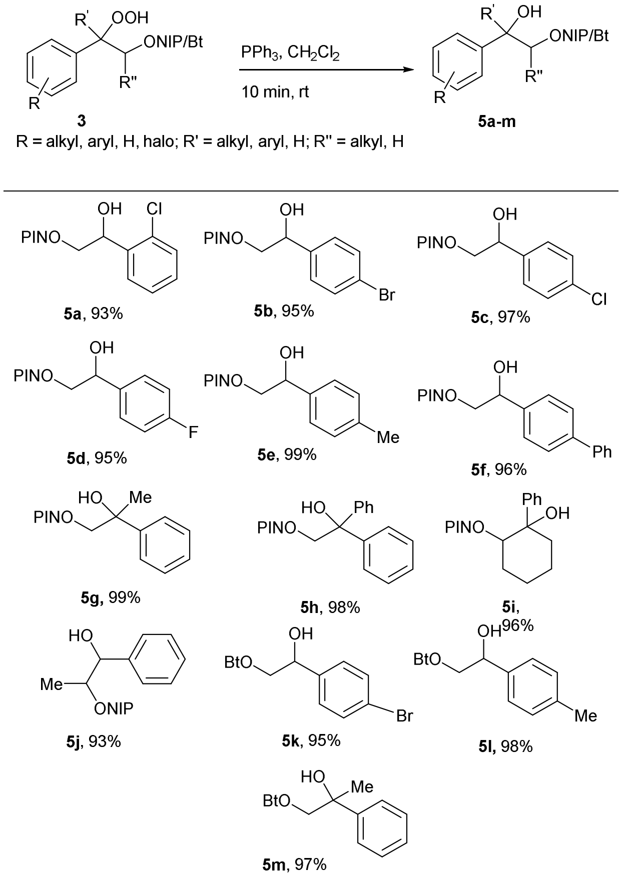
First, the reaction was optimized using *α*-methylstyrene **1a** and *N*-hydroxyphthalimide (NHPI) **2** as the model substrates (Table 1). To our delight, the oxidation selectively occurred to give *N*-(*β*-hydroperoxyalkoxy)phthalimide **3a** in 81% yield when the substrates were stirred with 20 mol % $Fe(NO_3)_3{\bullet}9H_2O$ for 9 h in 1,2-dichloroethane (DCE) under air (entry 1). The use of dichloromethane instead of DCE as a solvent led to an increase the yield to 84%, while DMSO, toluene and H_2O afforded inferior results (entries 2-5). Similar result was observed with molecular oxygen (entry 6). In contrast, the protocol with N_2 furnished **3a** in trace amount (entry 7). In a set of iron sources screened, $Fe(NO_3)_3{\bullet}9H_2O$, $FeBr_2$, $Fe(acac)_3$ and $K_3Fe(CN)_6$, the former gave the best results (entries 8-10). Decreasing the quantity of the iron source (15 mol %) or alkene (1.5 equiv) led to drop the yield to <71% (entries 11-12). Control experiment confirmed that without the iron source the formation of **3a** was not observed (entry 13).



Scheme 3. Reaction of 2-vinylpyridine with NHPI.



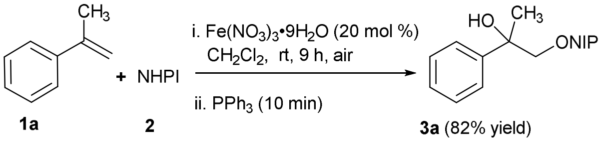
Scheme 4. Gram scale synthesis.



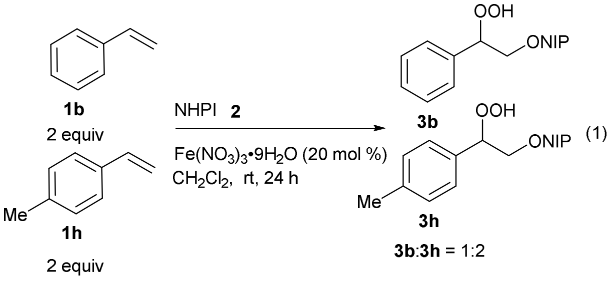
Scheme 5. Reaction conditions: peroxide (0.1 mmol), PPh_3 (0.1 mmol), CH_2Cl_2 (1.5 mL), rt, 10 min. Yields are isolated.

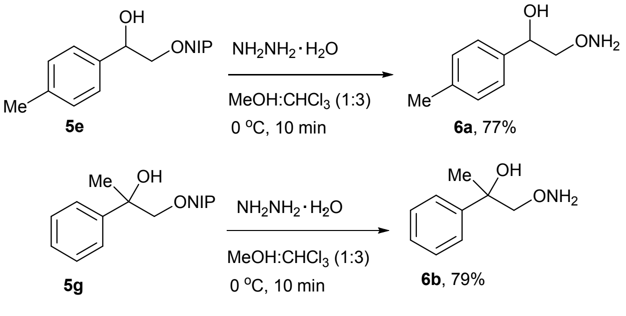
Having these results in hand, the scope of the procedure was explored (Scheme 2). Styrene **1b** underwent oxidation to give **3b** in 80% yield. The reaction of the alkenes **1c-i** bearing 2-chloro, 4-acetoxy, 4-bromo, 4-chloro, 4-fluoro, 4-methyl and 4-phenyl substituents produced the corresponding peroxides **3c-i** in 68-88% yields. In addition, di- and trisubstituted alkenes such as 1-phenylstyrene **1j** and 1-phenylcyclohexene **1k** oxidized to **3j** and **3k** in 73 and 69% yields, respectively, while the reaction of β-methylstyrene **1l** produced **3l** in 78% yield as a 3:1 diastereoisomers. Further, 2-vinylnaphthalene **1m** oxidized to **3m** in 74% yield. Under these conditions, 1-octene **1n** was less reactive and gave **3n** in 16% yield, while 2-vinylpyridine **1t** underwent reaction to produce alkoxyamine **3t** in 24% yield (Scheme 3). The substrates bearing electron donating group exhibited greater reactivity compared to that containing electron withdrawing groups (eq. 1).

Next, the utility of the protocol was extended to the reaction of *N*-hydroxybenzotriazole (HOBt) (Scheme 2). These reactions were less selective compared to NHPI based processes. For examples, the oxidation of the alkenes **1o**, **1e**, **1h** and **1a** having substituents on the aryl ring as well as alkene occurred to afford the peroxides **3o-s** in moderate to good yields along with ketones (~5%) as by-product.



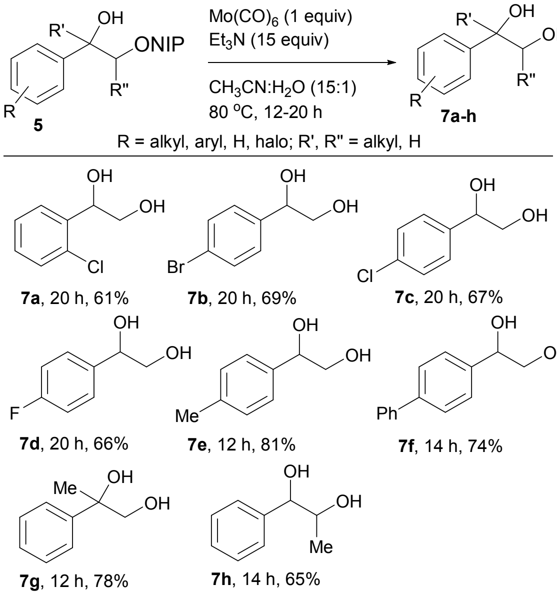
Scheme 6. One-pot synthesis of alcohol.



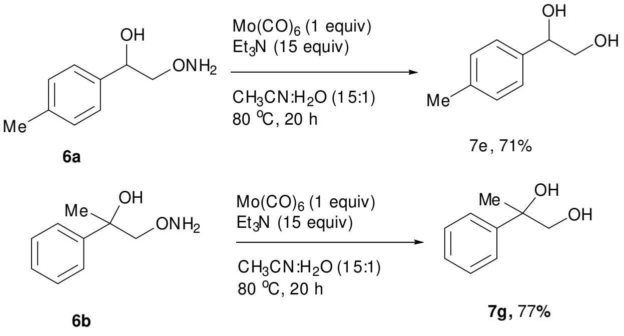


Scheme 7. Synthesis of β-hydroxy-*N*-alkoxyamines.

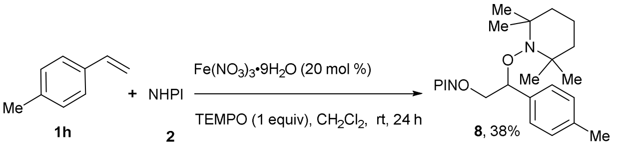
To reveal the scale up of the protocol, the reaction of **1a** was carried out in gram scale (Scheme 4). The oxidation readily occurred to afford the peroxide **3a** in 72% yield. Next, the utility of the protocol was studied for the transformation into alcohols employing the compounds **3a**, **3c**, **3e-l**, **3p-q** and **3s** as the representative examples (Scheme 5).[11] The reactions readily occurred to produce the hydroxy compounds **5a-m** in 93-99% yields. Compound **5i** gave single crystal whose structure was determined by X-ray analysis (see SI). In addition, the reaction conditions can be utilized for one-pot conversion of alkene to hydroxy compound in high yield (Scheme 6).



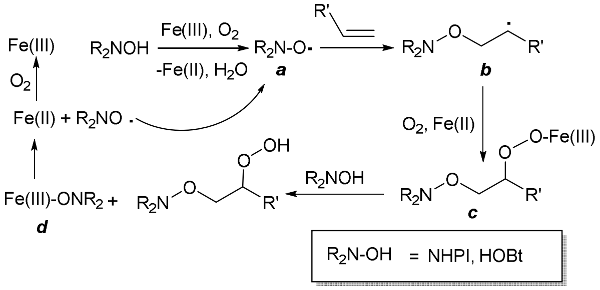
Scheme 8. Reaction conditions: **5a-g** or **5j** (0.2 mmol), Et_3N (3.0 mmol), $Mo(CO)_6$ (0.2 mmol), CH_3CN:H_2O (2 mL), 80 °C. Yields are isolated.



Scheme 9. Reaction conditions: β-hydroxy-*N*-alkoxyamine **6a-b** (0.2 mmol), $Mo(CO)_6$ (0.2 mmol), Et_3N (3.0 mmol), CH_3CN/H_2O (15:1, 2 mL), 80 °C. Yields are isolated.



Scheme 10. Trapping of styrenyl radical by TEMPO.



Scheme 11. Proposed catalytic cycle.

Finally, the hydrolysis of **5e** and **5g** was studied with hydrazine hydrate as the representative examples (Scheme 7).[12] The reactions were readily occurred to give the medicinally important β-hydroxyalkoxyamines **6a** and **6b** in 77 and 79% yields, respectively. In addition, the products can be readily transformed into vicinal 1,2-diols (Scheme 8).[13,14] For examples, the transformation of **5a-g** and **5j** to vicinal 1,2-diols was investigated using $Mo(CO)_6$.[15b] The reaction underwent readily to afford the corresponding 1,2-diols **7a-h** in 61-81% yields. Furthermore, β-hydroxyalkoxyamines **6a** and **6b** can be transformed into 1,2-diols **7e** and **7g** in 71 and 75% yields, respectively (Scheme 9).

To get insight into the reaction pathway, the reaction of **1h** was studied with TEMPO, and the compound **8** was isolated in 38% yield, which suggests that the reaction involves a radical intermediate (Scheme 10).[16] Thus, NHPI/HOBt[15,17] with Fe(III) and air can produce the radical ***a***, which may undergo addition with alkene to yield the secondary radical ***b*** (Scheme 11). The radical ***b*** may react with dioxygen and Fe(II) to furnish ***c***, which may react with *N*-hydroxylamine to yield the product and R_2N-O-Fe(III) species ***d***. The latter may undergo homolytic cleavage to $R_2NO\cdot$ radical ***a*** and Fe(II) species that may be oxidized to Fe(III) by air to complete the catalytic cycle.[18]

In summary, iron-catalyzed direct dioxygenation of alkenes has been accomplished at ambient conditions. The reaction is selective, scalable and utilizes the readily available inexpensive NHPI and HOBt as the radical precursor, iron salt as the catalyst and dioxygen from air as the oxygen source.

Acknowledgements

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EXPERIMENTAL SECTION

General Information. Styrene, *N*-hydroxyphthalimide (97%) and 2,2,6,6-tetramethylpiperdine-1-oxyl (99%) were purchased from Aldrich. *N*-Hydroxybenzotriazole (98%) was purchased from Spectrochem. $Fe(NO_3)_3{\cdot}9H_2O$ (98%) and hydrazine hydrate (90-100%) were obtained from Merck. Merck silica gel G/GF 254 plates were used for analytical TLC and Rankem silica gel (60-120 mesh) was utilized for column chromatography. DRX-400 Varian and Bruker Avance III 600 spectrometers were used for recording NMR (^{1}H and ^{13}C) using $CDCl_3$ and DMSO-d_6 as the solvents and TMS as an internal standard. Chemical shifts (δ) and spin-spin coupling constant (J) are reported in ppm and in Hz, respectively, and other data are reported as follows: s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, and br s = broad singlet. Melting points were determined with a Büchi B-540 apparatus and are uncorrected. FT-IR spectra were collected on PerkinElmer IR spectrometer. Q-Tof ESI-MS instrument (model HAB 273) was used for recording mass spectra. Single crystal X-ray data were determined using Bruker SMART APEX-II CCD diffractometer, which is equipped with 1.75 kW sealed-tube Mo-Kα irradiation (λ = 0.71073 Å) at 298(2) K. The crystal structure was solved by direct method using SHELXL-97 (Göttingen, Germany) and refined with full-matrix least squares on F^2 using SHELXL-97.

General Procedure for the Dioxygenation of Alkenes. Alkene **1** (0.5 mmol), *N*-hydroxyphthalimide **2** or *N*-hydroxybenzotriazole **4** (0.25 mmol) and $Fe(NO_3)_3{\cdot}9H_2O$ (20 mol %, 0.05 mmol, 20.2 mg) were stirred in CH_2Cl_2 (1.5 mL) at room temperature under air. The progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluent. The resultant mixture was extracted with CH_2Cl_2 (3 x 10 mL) and successively washed with brine (1 x 10 mL). The organic solution was dried over Na_2SO_4 and evaporated on a rotary evaporator to produce a residue that was purified on silica gel column chromatography using hexane and ethyl acetate as eluent.

General Procedure for the Synthesis of α-Oxygenated-β-Hydroxy Compounds. To a solution of peroxide **3** (0.1 mmol) in CH_2Cl_2 (1.5 mL) was added PPh_3 (0.1 mmol, 26.2 mg). The reaction mixture was stirred for 10 minutes at room temperature. The progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluent. The resultant mixture was extracted with CH_2Cl_2 (3 x 10 mL) and successively washed with brine (1 x 10 mL). The organic solution was dried over Na_2SO_4 and evaporated on a rotary evaporator to give a residue that was purified on silica gel column chromatography using hexane and ethyl acetate as eluent.

General Procedure for Hydrolysis to β-Hydroxyalkoxyamines. To a solution of **5** (0.34 mmol) in MeOH/$CHCl_3$ (1:3) was added hydrazine monohydrate (1.01 mmol, 51 mg). The reaction mixture was stirred at 0 ℃ for 10 minutes under air. The progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluent. After completion, the reaction mixture was evaporated. The colorless precipitate was filtered and washed with Et_2O (10 mL). The combined solution was evaporated under reduced pressure and the resultant oil was dissolved in Et_2O (4 mL). When the solution was subjected to dry HCl gas a colorless precipitate was formed that was filtered and washed with Et_2O (3 x 10 mL) to give the pure HCL salt of **6**.[12]

General Procedure for the Synthesis of 1,2-diols. To a solution of **5** or **6** (0.2 mmol) in CH_3CN/H_2O (15:1, 2 mL) was added $Mo(CO)_6$ (0.2 mmol, 53 mg) and Et_3N (3.0 mmol, 0.42 mL). The reaction mixture was stirred at 80 °C for 12-20 h. The progress of the reaction was monitored by TLC using ethyl acetate and hexane as eluent. The resultant mixture was neutralized by using aqueous ammonium chloride and extracted with ethyl acetate (3 x 10 mL). Drying (Na_2SO_4) and evaporation of the solvent gave a residue that was purified on silica gel column chromatography using hexane and ethyl acetate as eluent.

2-(2-Hydroperoxy-2-phenylpropoxy)isoindoline-1,3-dione 3a. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.36; white solid; mp 118-119 °C; yield 84% (66 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.86 (br s, 1H), 7.87-7.85 (m, 2H), 7.79-7.77 (m, 2H), 7.51 (d, J = 8.0 Hz, 2H), 7.38 (t, J = 7.2 Hz, 2H), 7.30 (t, J = 7.2 Hz, 1H), 4.72-4.63 (m, 2H), 1.66 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 164.0, 140.9, 135.0, 128.8, 128.7, 128.1, 125.5, 124.0, 84.6, 79.9, 23.0; FT-IR (KBr) 3371, 2993, 2924, 1782, 1720, 1494, 1446, 1400, 1379, 1186, 1141, 1082, 1019, 1003, 965, 878, 763, 700 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{17}$H$_{15}$NO$_5$: 336.0848, found: 336.0849.

2-(2-Hydroperoxy-2-phenylethoxy)isoindoline-1,3-dione 3b. Analytical TLC on silica gel, 1:4 ethyl acetate/hexane R_f = 0.38; liquid; yield 80% (60 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.66 (br s, 1H), 7.87-7.85 (m, 2H), 7.78-7.76 (m, 2H), 7.41-7.33 (m, 5H), 5.43-5.40 (m, 1H), 4.51-4.49 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 164.0, 135.8, 135.0, 129.0, 128.9, 128.8, 127.2, 124.0, 85.6, 79.1; FT-IR (neat) 3439, 1788, 1731, 1494, 1467, 1454, 1375, 1187, 1134, 1082, 1018, 997, 877, 699 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{16}$H$_{13}$NO$_5$: 322.0691, found: 322.1780.

2-(2-(2-Chlorophenyl)-2-hydroperoxyethoxy)isoindoline-1,3-dione 3c. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.39; liquid; yield 74% (62 mg); ^{1}H NMR (600 MHz, CDCl$_3$) δ 9.97 (br s, 1H), 7.86-7.84 (m, 2H), 7.78-7.76 (m, 2H), 7.58 (d, J = 6.4 Hz, 1H), 7.34-7.32 (m, 1H), 7.31-7.24 (m, 2H), 5.84 (d, J = 6.0 Hz, 1H), 4.51-4.47 (m, 1H), 4.29-4.26 (m, 1H); ^{13}C NMR (150 MHz, CDCl$_3$) δ 163.9, 134.9, 133.6, 132.6, 129.89, 129.8, 128.7 128.1, 127.3, 123.9, 83.1, 77.8; FT-IR (neat) 3441, 1789, 1729, 1467, 1443, 1374, 1187, 1134, 1081, 1034, 1018, 997, 877, 759, 701 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{16}$H$_{12}$ClNO$_5$: 356.0302, found: 356.0297.

4-(2-(1,3-Dioxoisoindolin-2-yloxy)-1-hydroperoxyethyl)phenyl acetate 3d. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.32; liquid; yield 68% (61 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.71 (br s, 1H), 7.88-7.85 (m, 2H), 7.79-7.77 (m, 2H), 7.44 (d, J = 8.4 Hz, 2H), 7.11 (d, J = 8.8 Hz, 2H), 5.42-5.39 (m, 1H), 4.50-4.48 (m, 2H) 2.29 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 169.5, 163.9, 151.1, 135.0, 133.5, 128.8, 128.5, 124.0, 122.1, 85.0, 78.8, 21.3; FT-IR (neat) 3409, 3066, 2945, 1784, 1751, 1736, 1722, 1606, 1509, 1465, 1373, 1224, 1202, 1169, 1137, 1082, 1015, 990, 911, 878, 701 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{18}$H$_{15}$NO$_5$: 380.0746, found: 380.0740.

2-(2-(4-Bromophenyl)-2-hydroperoxyethoxy)isoindoline-1,3-dione 3e. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.37; white solid; mp 114-115 °C; yield 74% (70 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.87 (br s, 1H), 7.83-7.81 (m, 2H), 7.77-7.75 (m, 2H), 7.48 (d, J = 8.0 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 5.38-5.34 (m, 1H), 4.47 (d, J = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 163.9, 134.9, 131.9, 131.7, 129.0, 128.6, 123.9, 122.9, 84.7, 78.4; FT-IR (KBr) 3452, 2948, 2918, 1783, 1717, 1486, 1465, 1378, 1187, 1132, 1080, 1016, 995, 877, 818, 697 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{16}$H$_{12}$BrNO$_5$: 399.9797, found: 399.9796.

2-(2-(4-Chlorophenyl)-2-hydroperoxyethoxy)isoindoline-1,3-dione 3f. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.36; liquid; yield 71% (60 mg); ^{1}H NMR (600 MHz, CDCl$_3$) δ 9.72 (br s, 1H), 7.86-7.84 (m, 2H), 7.78-7.77 (m, 2H), 7.36-7.33 (m, 4H), 5.38-5.36 (m, 1H), 4.50-4.44 (m, 2H); ^{13}C NMR (150 MHz, CDCl$_3$) δ 163.9, 135.0, 134.9, 134.4, 129.1, 128.8, 128.7, 124.0, 84.8, 78.6; FT-IR (neat) 3370, 3036, 2938, 1784, 1716, 1492, 1464, 1359, 1190, 1121, 1082, 1015, 987, 909, 876, 699 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{16}$H$_{12}$ClNO$_5$: 356.0302, found: 356.0302.

2-(2-(4-Fluorophenyl)-2-hydroperoxyethoxy)isoindoline-1,3-dione 3g. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.37; liquid; yield 71% (57 mg); ^{1}H NMR (600 MHz, CDCl$_3$) δ 9.67 (br s, 1H), 7.87-7.85 (m, 2H), 7.79-7.77 (m, 2H), 7.41-7.38 (m, 2H), 7.07-7.04 (m, 2H), 5.39-5.37 (m, 1H), 4.52-4.46(m, 2H); ^{13}C NMR (150 MHz, CDCl$_3$) δ 163.99, 163.9 (d, $J_{C\text{-}F}$ = 246.4 Hz), 135.0, 131.7, 129.28 (d, $J_{C\text{-}F}$ = 7.8 Hz), 128.8, 124.0, 115.9 (d, $J_{C\text{-}F}$ = 21.9 Hz), 84.8, 78.8; FT-IR (neat) 3434, 2923, 1789, 1732, 1605, 1511, 1467, 1375, 1225, 1187, 1160, 1132, 1082, 1018, 905, 878, 837, 702 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{16}$H$_{12}$FNO$_5$: 340.0597, found: 340.0596.

2-(2-Hydroperoxy-2-*p*-tolylethoxy)isoindoline-1,3-dione 3h. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.36; liquid; yield 88% (69 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.57 (br s, 1H), 7.87-7.85 (m, 2H), 7.78-7.76 (m, 2H), 7.29 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 5.37 (t, J = 6.4 Hz, 1H), 4.50-4.49 (m, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 163.9, 138.8, 134.8, 132.8, 129.4, 128.7, 127.2, 123.9, 85.3, 78.9, 21.3; FT-IR (neat) 3439, 2922, 1788, 1729, 1631, 1515, 1467, 1375, 1186, 1132, 1082, 1018, 996, 877, 814, 701 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{17}$H$_{15}$NO$_5$: 336.0848, found: 336.0852.

2-(2-(Biphenyl-4-yl)-2-hydroperoxyethoxy)isoindoline-1,3-dione 3i. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.35; liquid; yield 79% (74 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.72 (br s, 1H), 7.85-7.83 (m, 2H), 7.76-7.73 (m, 2H), 7.59-7.54 (m, 4H), 7.48 (d, J = 8.0 Hz, 2H), 7.42 (t, J = 7.6 Hz, 2H), 7.34 (t, J = 7.2 Hz, 1H), 5.47-5.46 (m, 1H), 4.55-4.53 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 163.9, 141.9, 140.6, 134.9, 134.8, 128.9, 128.8, 127.7, 127.68, 127.63, 127.2, 124.0, 85.3, 78.9; FT-IR (neat) 3439, 1787, 1728, 1632, 1487, 1374, 1187, 1133, 1081, 1018, 996, 877, 731, 699 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{22}$H$_{17}$NO$_5$: 398.1004, found: 398.1004.

2-(2-Hydroperoxy-2,2-diphenylethoxy)isoindoline-1,3-dione 3j. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.37; white solid; mp 173-174 °C; yield 73% (69 mg); ^{1}H NMR (600 MHz, CDCl$_3$) δ 10.34 (br s, 1H), 7.87-7.85 (m, 2H), 7.78-7.77 (m, 2H), 7.50 (d, J = 7.2 Hz, 4H), 7.35 (t, J = 7.2 Hz, 4H), 7.30-7.27 (m, 2H), 5.12 (s, 2H); ^{13}C NMR (150 MHz, CDCl$_3$) δ 164.0, 140.1, 135.1, 128.8, 128.6, 128.3, 127.0, 124.1, 88.4, 79.4; FT-IR (KBr) 3381, 3058, 2914, 1792, 1716, 1467, 1450, 1384, 1137, 1073, 1028, 985, 879, 744, 700 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{22}$H$_{17}$NO$_5$: 398.1004, found: 398.1007.

2-(2-Hydroperoxy-2-phenylcyclohexyloxy)isoindoline-1,3-dione 3k. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.38; liquid; yield 69% (61 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.15 (br s, 1H), 7.82-7.79 (m, 3H), 7.77-7.73 (m, 3H), 7.40 (t, J = 7.6 Hz, 2H), 7.30 (t, J = 7.6 Hz, 1H), 4.73-4.70 (m, 1H), 2.41-2.35 (m, 1H), 2.23-2.12 (m, 2H), 1.95-1.71 (m, 3H), 1.47-1.37 (m, 2H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 164.6, 138.3, 134.9, 128.8, 128.6, 128.4, 127.1, 123.8, 88.2, 86.8, 31.8, 28.0, 22.4, 21.8; FT-IR (neat) 3439, 2937, 2866, 1788, 1725, 1630, 1467, 1376, 1188, 1127, 1081, 1016, 984, 878, 762, 699 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{20}$H$_{19}$NO$_5$: 376.1161, found: 376.1165.

2-(1-Hydroperoxy-1-phenylpropan-2-yloxy)isoindoline-1,3-dione 3l. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.36; dr = 3:1, liquid; yield 78% (61 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 10.09 (br s, 1H), 9.79 (br s, 0.26H), 7.88-7.86 (m, 2.3H), 7.80-7.78 (m, 2.4H), 7.45-7.42 (m, 2H), 7.38-7.33 (m, 4H), 5.16-5.15 (m, 1H), 5.12-5.10 (m, 0.34H), 4.82-4.79 (m, 1H), 4.66-4.59 (m, 0.27H), 1.29 (d, J = 6.8 Hz, 3H), 1.20 (d, J = 6.4 Hz, 0.73H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 164.8, 164.7, 135.05, 135.01, 134.9, 128.9, 128.89, 128.84, 128.6, 128.3, 128.0, 124.03, 124.0, 88.2, 84.5, 17.1, 14.2; FT-IR (neat) 3415, 2926, 1788, 1730, 1632, 1467, 1454, 1381, 1188,1124, 1081, 1016, 979, 878, 701 cm^{-1}; HRMS (ESI) m/z [M+Na]$^+$ calcd for C$_{17}$H$_{15}$NO$_5$: 336.0848, found: 336.0848.

2-(2-Hydroperoxy-2-(naphthalen-2-yl)ethoxy)isoindoline-1,3-dione 3m. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.38; liquid; yield 74% (65 mg); ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.70 (br s, 1H), 7.90-7.88 (m, 1H), 7.83-7.78 (m, 5H), 7.75-7.72 (m, 2H), 7.49-7.45 (m, 3H), 5.58 (t, J = 6.0 Hz, 1H), 4.59 (d, J = 5.2 Hz, 2H); ^{13}C NMR (100

MHz, $CDCl_3$) δ 164.0, 134.9, 133.5, 133.3, 133.2, 128.8, 128.7, 128.3, 127.8, 126.7, 126.6, 126.5, 124.6, 123.9, 85.7, 79.0; FT-IR (neat) 3415, 3052, 2922, 1786, 1719, 1465, 1372, 1188, 1142, 1126, 1081, 1021, 1003, 952, 878, 822, 752, 702 cm^{-1}; HRMS (ESI) *m/z* [M+Na]$^+$ calcd for $C_{20}H_{15}NO_5$: 372.0848, found: 372.0850.

2-(2-Hydroperoxyoctyloxy)isoindoline-1,3-dione 3n. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.43; liquid; yield 16% (12 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.71 (br s, 1H), 7.88-7.85 (m, 2H), 7.79-7.77 (m, 2H), 4.50 (q, *J* = 6.4 Hz, 1H), 4.27-4.21 (m, 2H), 1.70-1.64 (m, 2H), 1.33-1.25 (m, 8H), 0.90-0.87 (m, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.1, 135.0, 128.9, 124.0, 82.6, 79.0, 31.8, 29.3, 29.0, 25.7, 22.7, 14.2; FT-IR (neat) 3438, 2926, 2854, 1789, 1731, 1637, 1466, 1376, 1187, 1124, 1075, 1018, 877, 701 cm^{-1}; HRMS (ESI) *m/z* [M+Na]$^+$ calcd for $C_{16}H_{21}NO_5$: 330.1317, found: 330.1318

1-(2-Hydroperoxy-2-*m*-tolylethoxy)-1*H*-benzo[*d*][1,2,3]triazole 3o. Analytical TLC on silica gel, 1:2 ethyl acetate/hexane R_f = 0.36; liquid; yield 46% (33 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.60 (br s, 1H), 7.98 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 1H), 7.50 (t, *J* = 6.8 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.18-7.14 (m, 3H), 5.38 (t, *J* = 6.0 Hz, 1H), 4.80 (d, *J* = 6.4 Hz, 2H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.4, 138.8, 135.4, 130.1, 128.9, 128.4, 128.0, 127.6, 125.1, 124.4, 120.2, 109.2, 84.6, 80.5, 21.5; FT-IR (neat) 3439, 2920, 2850, 1610, 1489, 1445, 1361, 1265, 1241, 1159, 1098, 971, 782, 744, 702 cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{15}H_{15}N_3O_3$: 286.1192, found: 286.1193.

1-(2-(4-Bromophenyl)-2-hydroperoxyethoxy)-1*H*-benzo[*d*][1,2,3]triazole 3p. Analytical TLC on silica gel, 1:2 ethyl acetate/hexane R_f = 0.38; liquid; yield 52% (45 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.25 (br s, 1H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.62 (d, *J* = 8.4 Hz, 1H), 7.55-7.51 (m, 3H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.39 (t, *J* = 6.0 Hz, 1H), 4.79 (d, *J* = 5.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.4, 134.6, 132.2, 129.1, 128.6, 127.5, 125.2, 123.4, 120.3, 109.0, 84.0, 79.9; FT-IR (neat) 3471, 2923, 2853, 1640, 1486, 1445, 1362, 1265, 1241, 1099, 1071, 1011, 972, 821, 782, 743 cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{14}H_{12}BrN_3O_3$: 350.0140, found: 350.0141.

1-(2-Hydroperoxy-2-*p*-tolylethoxy)-1*H*-benzo[*d*][1,2,3]triazole 3q. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.36; liquid; yield 61% (43 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.80 (br s, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 1H), 7.27 (d, *J* = 7.6 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 5.38-5.35 (m, 1H), 4.85-4.76 (m, 2H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.3, 139.2, 132.5, 129.6, 128.4, 127.6, 127.3, 125.0, 120.1, 109.2, 84.3, 80.4, 21.3; FT-IR (neat) 3439, 2921, 1615, 1515, 1445, 1362, 1265, 1240, 1157, 1098, 969, 815, 766, 744 cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{15}H_{15}N_3O_3$: 286.1192, found: 286.1190.

1-(2-Hydroperoxy-2-phenylpropoxy)-1*H*-benzo[*d*][1,2,3]triazole 3s Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.37; liquid; yield 63% (45 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.36 (br s, 1H), 7.97 (d, *J* = 8.4 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 3H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.39 (t, *J* = 8.0 Hz, 2H), 7.33 (t, *J* = 8.0 Hz, 2H), 4.97-4.91 (m, 2H), 1.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.4, 140.3, 128.9, 128.4, 128.3, 127.2, 125.7, 125.0, 120.2, 109.2, 84.6, 82.7, 22.1; FT-IR (neat) 3439, 2987, 2850, 1640, 1496, 1446, 1373, 1264, 1159, 1099, 983, 844, 764, 744, 699 cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{15}H_{15}N_3O_3$: 286.1192, found: 286.1191.

2-(2-(Pyridin-2-yl)ethoxy)isoindoline-1,3-dione 3t. Analytical TLC on silica gel, 1:2 ethyl acetate/hexane R_f = 0.22; liquid; yield 24% (16 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (d, *J* = 6.8 Hz, 1H), 7.83-7.81 (m, 2H), 7.75-7.73 (m, 2H), 7.66-7.62 (m, 1H), 7.39 (d, *J* = 7.6 Hz, 1H), 7.14 (t, *J* = 6.4 Hz, 1H), 4.64 (t, *J* = 6.8 Hz, 2H), 3.32 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 157.5, 149.5, 136.7, 134.6, 129.0, 123,9, 123.7, 121.9, 77.4, 37.1; FT-IR (neat) 2924, 1789, 1732, 1592, 1570, 1467, 1437, 1373, 1187, 1129, 1082, 1017, 991, 877, 700 cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{15}H_{12}N_2O_3$: 269.0926, found: 269.0934.

2-(2-(2-Chlorophenyl)-2-hydroxyethoxy)isoindoline-1,3-dione 5a. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.44; liquid; yield 93% (29.5 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.89 (m, 2H), 7.82-7.80 (m, 2H), 7.71 (d, *J* = 7.6 Hz, 1H), 7.34-7.29 (m, 2H), 7.26-7.21 (m, 1H), 5.38 (d, *J* = 8.8 Hz, 1H), 4.53 (d, *J* = 11.2 Hz, 1H), 3.96 (t, *J* = 10.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.6, 135.7, 135.1, 131.7, 129.4, 129.3, 128.8, 128.0, 127.5, 124.2, 82.1, 67.9; FT-IR (neat) 3525, 2945, 2922, 1781, 1726, 1465, 1436, 1381, 1361, 1305, 1283, 1187, 1136, 1082, 1062, 1031, 994, 957, 879, 763, 704, 694 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{16}H_{12}ClNO_4$: 300.0427, found: 300.0428.

2-(2-(4-Bromophenyl)-2-hydroxyethoxy)isoindoline-1,3-dione 5b. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.44; white solid; mp 113-114 ℃; yield 95% (34.4 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.86 (m, 2H), 7.82-7.79 (m, 2H), 7.48 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.4 Hz, 2H), 4.98 (d, *J* = 10.0 Hz, 1H), 4.35 (d, *J* = 11.6 Hz, 1H), 4.06 (t, *J* = 11.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5, 137.2, 135.1, 131.8, 128.8, 128.0, 124.1, 122.2, 83.5, 70.3; FT-IR (KBr) 3509, 3089, 2941, 1782, 1732, 1716, 1488, 1466, 1399, 1374, 1313, 1186, 1129, 1076, 1009, 991, 877, 837, 698 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{16}H_{12}BrNO_4$: 343.9922, found: 343.9919.

2-(2-(4-Chlorophenyl)-2-hydroxyethoxy)isoindoline-1,3-dione 5c. Analytical TLC on silica gel, 1:4 ethyl acetate/hexane R_f = 0.45; white solid; mp 119-120 ℃; yield 97% (30.8 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.87 (m, 2H), 7.81-7.79 (m, 2H), 7.34-7.29 (m, 4H), 4.99 (d, *J* = 7.6 Hz, 1H), 4.36 (d, *J* = 11.6 Hz, 1H), 4.07 (t, *J* = 10.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5, 136.7, 135.1, 134.0, 128.9, 128.8, 127.7, 124.1, 83.6, 70.3; FT-IR (KBr) 3504, 3093, 2984, 2941, 1787, 1733, 1716, 1491, 1466, 1403, 1373, 1365, 1319, 1236, 1184, 1128, 1087, 1076, 1017, 993, 889, 877, 837, 700, 692 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{16}H_{12}ClNO_4$: 300.0427, found: 300.0429.

2-(2-(4-Fluorophenyl)-2-hydroxyethoxy)isoindoline-1,3-dione 5d. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.44; white solid; mp 101-102 ℃; yield 95% (28.6 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.87 (m, 2H), 7.81-7.79 (m, 2H), 7.38-7.34 (m, 2H), 7.03 (t, *J* = 8.4 Hz, 2H), 4.99 (d, *J* = 7.6 Hz, 1H), 4.35 (d, *J* = 11.6 Hz, 1H), 4.08 (t, *J* = 10.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5, 163.9 (d, $J_{C\text{-}F}$ = 245.0 Hz), 135.1, 134.0, 128.8, 128.1 (d, $J_{C\text{-}F}$ = 8.0 Hz), 124.1, 115.7 (d, $J_{C\text{-}F}$ = 22.0 Hz), 83.7, 70.3; FT-IR (KBr) 3504, 2942, 2898, 1787, 1726, 1606, 1512, 1466, 1410, 1366, 1317, 1225, 1186, 1177, 1160, 1124, 1110, 1077, 1015, 995, 876, 831, 787, 704 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{16}H_{12}FNO_4$: 284.0723, found: 284.0721.

2-(2-Hydroxy-2-*p*-tolylethoxy)isoindoline-1,3-dione 5e. Analytical TLC on silica gel, 1:4 ethyl acetate/hexane R_f = 0.41; white solid; mp 104-105 ℃; yield 99% (29.4 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.86 (m, 2H), 7.80-7.78 (m, 2H), 7.27 (d, *J* = 7.6 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 4.98 (d, *J* = 9.6 Hz, 1H), 4.36 (d, *J* = 11.6 Hz, 1H), 4.18 (br s, 1H), 4.11 (t, *J* = 11.6 Hz, 1H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.5, 138.0, 135.2, 135.0, 129.4, 128.9, 126.3, 124.0, 83.8, 70.8, 21.3; FT-IR (KBr) 3525, 2920, 1784, 1719, 1466, 1379, 1314, 1187, 1137, 1018, 994, 877, 815, 698 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{17}H_{15}NO_4$: 280.0974, found: 280.0967.

2-(2-(Biphenyl-4-yl)-2-hydroxyethoxy)isoindoline-1,3-dione 5f. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.40; white solid; mp 129-130 ℃; yield 96% (34.5 mg); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82-7.80 (m, 2H), 7.72-7.70 (m, 2H), 7.52 (t, *J* = 7.2 Hz, 4H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.39 (t, *J* = 7.8 Hz, 2H), 7.30 (t, *J* = 7.2 Hz, 1H), 5.06 (d, *J* = 9.6 Hz, 1H), 4.40 (d, *J* = 12.0 Hz, 1H), 4.16 (t, *J* =

9.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4, 141.0, 140.6, 137.2, 134.9, 128.8, 128.7, 127.4, 127.3, 127.1, 126.7, 123.9, 83.6, 70.6; FT-IR (KBr) 3508, 3027, 2919, 1784, 1722, 1466, 1485, 1378, 1314, 1231, 1187, 1133, 1082, 1066, 1018, 992, 876, 767, 735, 699 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{22}H_{17}NO_4$: 342.1130, found: 342.1130.

2-(2-Hydroxy-2-phenylpropoxy)isoindoline-1,3-dione 5g. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.41; liquid; yield 99% (29.4 mg); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75-7.74 (m, 2H), 7.71-7.69(m, 2H), 7.50 (d, *J* = 7.2 Hz, 2H), 7.27 (t, *J* = 7.8 Hz, 2H), 7.13 (t, *J* = 7.8 Hz, 1H), 4.79 (br s, 1H), 4.58 (d, *J* = 10.8 Hz, 1H), 4.31 (d, *J* = 10.8 Hz, 1H), 1.60 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 163.8, 143.9, 134.7, 128.6, 128.3, 127.1, 125.0, 123.7, 86.3, 73.4, 26.9; FT-IR (neat) 3316, 2982, 1790, 1717, 1627, 1496, 1464, 1446, 1370, 1255, 1178, 1134, 1082, 1022, 1004, 976, 961, 891, 874, 786, 765, 698 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{17}H_{15}NO_4$: 280.0974, found: 280.0974.

2-(2-Hydroxy-2,2-diphenylethoxy)isoindoline-1,3-dione 5h. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.42; liquid; yield 98% (35.2 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75-7.73 (m, 2H), 7.69-7.67 (m, 2H), 7.52 (d, *J* = 7.6 Hz, 4H), 7.28 (t, *J* = 7.2 Hz, 4H), 7.18 (t, *J* = 7.2 Hz, 2H), 4.80 (s, 2H), 4.77 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7, 143.0, 134.8, 128.5, 128.4, 127.5, 126.5, 123.8, 84.6, 77.4; FT-IR (neat) 3536, 3027, 2948, 2888, 1790, 1722, 1492, 1464, 1449, 1383, 1335, 1257, 1187, 1170, 1124, 1060, 1028, 1011, 977, 908, 875, 772, 697 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{22}H_{17}NO_4$: 342.1130, found: 342.1132.

2-(2-Hydroxy-2-phenylcyclohexyloxy)isoindoline-1,3-dione 5i. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.39; white solid; mp 156-157 ℃; yield 96% (32.4 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76-7.69 (m, 6H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.27-7.25 (m, 1H), 4.47-4.44 (m, 1H), 3.01 (br s, 1H), 2.55-2.49 (m, 1H), 2.32-2.24 (m, 1H), 2.11-1.96 (m, 1H), 1.83-1.69 (m, 3H), 1.54-1.45 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.1, 144.2, 134.6, 128.9, 128.1, 127.6, 127.2, 123.6, 92.1, 74.1, 35.2, 27.5, 21.6, 21.5; FT-IR (KBr) 3434, 3030, 2935, 2867, 1781, 1720, 1493, 1465, 1446, 1377, 1276, 1189, 1163, 1119, 1081, 1043, 1020, 996, 979, 880, 762, 699 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{20}H_{19}NO_4$: 320.1287, found: 320.1291.

2-(1-Hydroxy-1-phenylpropan-2-yloxy)isoindoline-1,3-dione 5j. Analytical TLC on silica gel, 1:3 ethyl acetate/hexane R_f = 0.40; liquid; yield 96% (27.6 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.89 (m, 2H), 7.82-7.79 (m, 2H), 7.34-7.32 (m, 4H), 7.27-7.24 (m, 1H), 5.027-5.021 (m, 1H), 4.50-4.45 (m, 1H), 4.06 (br s, 1H), 1.22 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.1, 138.7, 135.1, 129.0, 128.4, 127.5, 126.0, 124.1, 88.3, 71.7, 11.3; FT-IR (neat) 3504, 2989, 2938, 1787, 1728, 1612, 1494, 1467, 1450, 1375, 1327, 1187, 1125, 1082, 1057, 1015, 977, 878, 747, 700 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{17}H_{15}NO_4$: 280.0974, found: 280.0975.

2-(1*H*-Benzo[*d*][1,2,3]triazol-1-yloxy)-1-(4-bromophenyl)ethanol 5k. Analytical TLC on silica gel, 1:4 ethyl acetate/hexane R_f = 0.41; liquid; yield 95% (31.7 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 8.4 Hz, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.41 (t, *J* = 8.0 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.21-5.18 (m, 1H), 4.63-4.49 (m, 2H), 3.56 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.6, 137.7, 132.0, 128.5, 128.1, 127.3, 125.1, 122.6, 120.4, 108.9, 84.5, 70.8; FT-IR (neat) 3439, 2925, 2854, 1632, 1488, 1445, 1401, 1360, 1264, 1240, 1157, 1088, 1010, 973, 821, 782, 743, cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{14}H_{12}BrN_3O_2$: 334.0191, found: 334.0192.

2-(1*H*-Benzo[*d*][1,2,3]triazol-1-yloxy)-1-*p*-tolylethanol 5l. Analytical TLC on silica gel, 1:4 ethyl acetate/hexane R_f = 0.41; liquid; yield 98% (26.4 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 1H), 7.62 (d, *J* = 8.4 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 1H), 7.37 (t, *J* = 8.0 Hz, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.15-7.13 (m, 2H), 5.18-5.15 (m, 1H), 4.64-4.53 (m, 2H), 4.37 (br s, 1H), 2.31 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.4, 138.4, 135.7, 129.5, 128.4, 127.4, 126.3, 125.1, 120.2, 109.1, 84.8, 71.2, 21.3; FT-IR (neat) 3439, 2921, 1613, 1514, 1446, 1359, 1264, 1240, 1179, 1158, 1090, 1020, 971, 900, 816, 781, 744 cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{15}H_{15}N_3O_2$: 270.1243, found: 270.1243.

1-(1*H*-Benzo[*d*][1,2,3]triazol-1-yloxy)-2-phenylpropan-2-ol 5m. Analytical TLC on silica gel, 3:7 ethyl acetate/hexane R_f = 0.42; liquid; yield 97% (26 mg); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 1H), 7.58 (d, *J* = 7.2 Hz, 2H), 7.46 (t, *J* = 7.8 Hz, 1H), 7.39 (t, *J* = 7.8 Hz, 3H), 7.36 (t, *J* = 8.4 Hz, 1H), 7.31 (t, *J* = 7.8 Hz, 1H), 4.70-4.63 (m, 2H), 3.28 (br s, 1H), 1.80 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 143.58, 143.54, 128.7, 128.2, 127.9, 127.1, 125.3, 124.9, 120.3, 108.8, 87.7, 73.9, 26.6; FT-IR (neat) 3413, 2977, 2926, 2850, 1617, 1494, 1446, 1375, 1263, 1240, 1158, 1097, 974, 743, 700 cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{15}H_{15}N_3O_2$: 270.1243, found: 270.1241.

2-(Aminooxy)-1-*p*-tolylethanol 6a. Analytical TLC on silica gel, 1:2 ethyl acetate/hexane R_f = 0.46; liquid; yield 77% (44 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.99 (br s, 2H), 7.26 (d, *J* = 7.6 Hz, 2H), 7.16 (d, *J* = 7.6 Hz, 2H), 4.87-4.84 (m, 1H), 4.07-3.96 (m, 2H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 138.1, 136.7, 128.7, 126.1, 78.6, 69.9, 20.7; FT-IR (neat) 3439, 3258, 2920, 2690, 1592, 1554, 1342, 1076, 1060, 1026, 946, 890, 814 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_9H_{13}NO_2$: 150.0919, found: 150.0919.

1-(Aminooxy)-2-phenylpropan-2-ol 6b. Analytical TLC on silica gel, 1:2 ethyl acetate/hexane R_f = 0.47; liquid; yield 79% (45 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 11.05 (br s, 2H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.33 (t, *J* = 7.2 Hz, 2H), 7.24 (t, *J* = 7.2 Hz, 1H), 4.09 (s, 2H), 1.47 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 145.2, 127.9, 126.8, 125.3, 80.9, 73.0, 26.2; FT-IR (neat) 3471, 1632, 1554, 1538, 1505, 1382, 1226, 1091, 1015, 917, 700 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_9H_{13}NO_2$: 150.0919, found: 150.0919.

1-(2-Chlorophenyl)ethane-1,2-diol 7a.[13] Analytical TLC on silica gel, 1:1 ethyl acetate/hexane R_f = 0.46; white solid; mp 106-107 ℃; yield 61% (21 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61-7.58 (m, 1H), 7.35-7.29 (m, 2H), 7.26-7.21 (m, 1H), 5.26-5.23 (m, 1H), 3.92-3.88 (m, 1H), 3.59-3.55 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.7, 131.7, 129.0, 128.4, 127.8, 126.8, 71.2, 66.4; FT-IR (KBr) 3280, 2924, 2855, 1637, 1470, 1437, 1364, 1265, 1129, 1096, 1070, 1031, 899, 758 cm^{-1}; HRMS (ESI) *m/z* [M+Na]$^+$ calcd for $C_8H_9ClO_2$: 195.0189, found: 195.0188.

1-(4-Bromophenyl)ethane-1,2-diol 7b.[13] Analytical TLC on silica gel, 1:1 ethyl acetate/hexane R_f = 0.43; white solid; mp 99-100 ℃; yield 69% (30 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51-7.47 (m, 2H), 7.27-7.24 (m, 2H), 4.81-4.78 (m, 1H), 3.76-3.73 (m, 1H), 3.63-3.59 (m, 1H), 2.69 (br s, 1H), 2.17 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$ + DMSO-d_6) δ 140.7, 130.5, 127.5, 120.3, 73.3, 67.5; FT-IR (KBr) 3406, 2931, 2895, 1651, 1590, 1483, .1458, 1394, 1347, 1228, 1196, 1093, 1068, 1035, 1011, 899, 823 cm^{-1}; HRMS (ESI) *m/z* [M+Na]$^+$ calcd for $C_8H_9BrO_2$: 238.9684, found: 238.9683.

1-(4-Chlorophenyl)ethane-1,2-diol 7c.[13] Analytical TLC on silica gel, 1:1 ethyl acetate/hexane R_f = 0.42; liquid; yield 67% (23 mg); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.37-7.32 (m, 4H), 5.36 (d, *J* = 4.4 Hz, 1H), 4.77 (t, *J* = 6.0 Hz, 1H), 4.52 (q, *J* = 5.6 Hz, 1H), 3.38 (br s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ; 142.5, 131.2, 128.2, 127.7, 73.0, 67.2; FT-IR (neat) 3643, 3437, 2926, 2848, 1634, 1490, 1193, 1083, 1016, 818 cm^{-1}; HRMS (ESI) *m/z* [M+Na]$^+$ calcd for $C_8H_9ClO_2$: 195.0189, found: 195.0187.

1-(4-Fluorophenyl)ethane-1,2-diol 7d.[14a] Analytical TLC on silica gel, 1:1 ethyl acetate/hexane R_f = 0.43; liquid; yield 66% (21 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.29 (m, 2H), 7.06-7.01 (m, 2H), 4.79-4.77 (m, 1H), 3.71-3.68 (m, 1H), 3.62-3.59 (m, 1H), 3.02 (br s, 1H), 2.00 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8 (d, $J_{C\text{-}F}$ = 244.4 Hz), 136.3, 127.9 (d, $J_{C\text{-}F}$ = 8.3 Hz), 115.7 (d, $J_{C\text{-}F}$ = 21.7 Hz), 74.2, 68.2; FT-IR (neat) 3416, 2956, 2922, 1637, 1606, 1510, 1379, 1225, 1078, 1025, 891, 834 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_8H_9FO_2$: 139.0559, found: 139.0564.

1-(*p*-Tolyl)ethane-1,2-diol 7e.[13] Analytical TLC on silica gel, 1:1 ethyl acetate/hexane R_f = 0.41; white solid; mp 69-70 °C; yield 81% (25 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 4.76- 4.73 (m, 1H), 3.69-3.60 (m, 2H), 3.05 (br s, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.8, 137.6, 129.3, 126.2, 74.7, 68.2, 21.3; FT-IR (KBr) 3259, 3158, 2919, 2858, 1514, 1461, 1348, 1234, 1198, 1096, 1070, 1032, 899, 819, 774 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_9H_{12}O_2$: 135.0810, found: 135.0811.

1-([1,1'-Biphenyl]-4-yl)ethane-1,2-diol 7f. Analytical TLC on silica gel, 1:1 ethyl acetate/hexane R_f = 0.44; white solid; mp 147-148 °C; yield 74% (32 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (t, *J* = 7.6 Hz, 4H), 7.44 (t, *J* = 7.6 Hz, 4H), 7.35 (t, *J* = 7.6 Hz, 1H), 4.90-4.88 (m, 1H), 3.84-3.69 (m, 2H), 1.80 (br s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 142.7, 140.2, 138.7, 128.9, 127.2, 126.9, 126.6, 126.2, 73.5, 67.4; FT-IR (KBr) 3349, 3025, 2925, 1634, 1565, 1485, 1404, 1363, 1247, 1191, 1097, 1074, 1043, 899, 845, 759, 725, 689 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_{14}H_{14}O_2$: 197.0966, found: 197.0967.

2-Phenylpropane-1,2-diol 7g.[14b] Analytical TLC on silica gel, 1:1 ethyl acetate/hexane R_f = 0.44; liquid; yield 78% (24 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 7.6 Hz, 2H), 7.36 (t, *J* = 7.2 Hz, 2H), 7.29 (d, *J* = 7.2 Hz, 1H), 3.79 (d, *J* = 11.2 Hz, 1H), 3.63 (d, *J* = 11.2 Hz, 1H), 2.85 (br s, 1H), 2.20 (br s, 1H), 1.52 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.1, 128.6, 127.3, 125.2, 75.0, 71.2, 26.2; FT-IR (neat) 3442, 2954, 2923, 2870, 1766, 1716, 1638, 1494, 1448, 1377, 1240, 1156, 1125, 1045, 955, 864, 764, 701 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_9H_{12}O_2$: 135.0810, found: 135.0810.

1-Phenylpropane-1,2-diol 7h.[13] Analytical TLC on silica gel, 1:1 ethyl acetate/hexane R_f = 0.45; liquid; yield 65% (20 mg); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.28 (m, 5H), 4.68 (d, *J* = 4.0 Hz, 1H), 4.02-3.99 (m, 1H), 2.67 (br s, 1H), 2.04 (br s, 1H), 1.08-1.06 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4, 128.5, 128.0, 126.8, 77.6, 71.4, 17.3; FT-IR (neat) 3414, 2959, 2921, 1639, 1452, 1379, 1259, 1199, 1126, 1080, 1043, 991, 930, 852, 746, 700 cm^{-1}; HRMS (ESI) *m/z* [M-OH]$^+$ calcd for $C_9H_{12}O_2$: 135.0810, found: 135.0814.

2-(2-(2,2,6,6-Tetramethylpiperidin-1-yloxy)-2-*p*-tolylethoxy)isoindoline-1,3-dione 8. Analytical TLC on silica gel, 1:4 ethyl acetate/hexane R_f = 0.56; liquid; yield 38% (42 mg); ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.77-7.76 (m, 2H), 7.70-7.69 (m, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 7.8 Hz, 2H), 5.09 (t, *J* = 7.2 Hz, 1H), 4.74-4.71 (m, 1H), 4.48 (t, *J* = 9.6 Hz, 1H), 2.29 (s, 3H), 1.46-1.25 (m, 9H), 1.17 (s, 3H), 1.03 (s, 3H), 0.73 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 163.1, 137.3, 137.0, 134.2, 128.7, 128.6, 127.8, 123.2, 82.9, 79.9, 60.0, 40.3, 34.0, 21.1, 20.2, 17.0; FT-IR (neat) 2930, 1791, 1735, 1514, 1466, 1375, 1361, 1257, 1186, 1132, 1081, 1017, 997, 877, 817, 700 cm^{-1}; HRMS (ESI) *m/z* [M+H]$^+$ calcd for $C_{26}H_{32}N_2O_4$: 437.2440, found: 437.2441.

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TOC

Iron(III)-catalyzed aerobic dioxygenation of styrenes using *N*-hydroxyphthalimide and *N*-hydroxybenzotriazole

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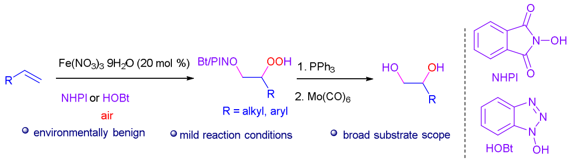

The dioxygenation of alkenes with air and NHPI/HOBt is described using commercial iron salt as the catalyst at ambient conditions with broad substrate scope and functional group tolerance. The products can be readily converted into the corresponding 1,2-diols in high yields.