A Stabilized Formulation of IBX (SIBX) for Safe Oxidation Reactions Including a New Oxidative Demethylation of Phenolic Alkyl Aryl Ethers

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Experimental Section

General. All oxidations were run under nitrogen atmosphere using stabilized *o*-iodoxybenzoic acid (SIBX, Simafex, France) as the oxidizing agent.[1] Tetrahydrofuran (THF) was purified by distillation from sodium/benzophenone under N_2 prior to use. Dichloromethane (CH_2Cl_2), ethyl acetate (EtOAc), toluene ($PhCH_3$), acetone, acetonitrile (CH_3CN), *N*-methylpyrrolidone (NMP) and dimethyl sulfoxide (DMSO) were used as received. Evaporations were conducted under reduced pressure at room temperature. Column chromatography was carried out under positive pressure using 40-63 μm silica gel (Merck) and the indicated solvents. Further drying of the residues was accomplished under high vacuum except for volatile products. Melting points are uncorrected. NMR spectra of samples in the indicated solvent were run at 200, 250 or 300 MHz. Carbon multiplicities were determined by DEPT135 experiments. Electron impact mass spectra (EIMS) were obtained at 50-70 eV. Electron impact mass spectrometry high resolution (HRMS) were obtained from the mass spectrometry laboratory at the CESAMO, Université Bordeaux 1.

Procedure for the Preparation of SIBX. *o*-Iodobenzoic acid (200 g) and isophtalic acid (133 g) are introduced into a solution of oxone (625 g) in water (2 L). The mixture is maintained under stirring at 70°C for 3 hours and then a solution of sodium benzoate (128 g) in water (500 mL) is added at 40°C. After cooling to 20°C, the precipitate is filtered off, washed with water (700 mL) and dried in a ventilated oven at 60°C to obtain 420 g (90%) of stabilized IBX composition (49% w/w of IBX).

Procedure A. To a stirred solution of alcohol (10 mmol) in NMP or DMSO (50 mL, *ca.* 0.2 M) was added SIBX (1.2 equiv per alcohol to oxidize) in one portion. The resulting

suspension was stirred vigorously at room temperature. After 2 h, the reaction mixture was poured into water (100 mL) and EtOAc (60 mL), neutralized with 10 M NaOH, and filtered. The filtrate was decanted, separated and the organic layer was washed with water (60 mL), dried over Na_2SO_4, filtered and evaporated to dryness.

Procedure B. To a stirred solution of alcohol (1 mmol) in the indicated solvent (7 mL, *ca.* 0.14 M) was added SIBX (1.2 equiv per alcohol to oxidize) in one portion. The resulting suspension was stirred vigorously at the selected temperature (see Table 2). After TLC monitoring indicated completion of the reaction, the white suspension was cooled to room temperature and filtered. The filter cake was washed with EtOAc (or CH_2Cl_2 for volatile compounds) (3 × 7 mL). When the solvent was water-miscible (THF, acetone and CH_3CN), the combined filtrate and washings were concentrated, EtOAc (or CH_2Cl_2 for volatile compounds) was added (7 mL), and the resulting solution was washed with saturated aqueous $NaHCO_3$ (4 × 14 mL) and water (14 mL), dried over Na_2SO_4, filtered and evaporated to dryness.

Procedure C. To a stirred solution of alcohol (1 mmol) in dry THF (20 mL, *ca.* 0.05 M) was added SIBX (1.2 equiv per alcohol to oxidize) in one portion. The resulting suspension was stirred vigorously at room temperature. After TLC monitoring indicated completion of the reaction, the white suspension was filtered. The filter cake was washed with CH_2Cl_2 (3 x 7 mL). The combined filtrate and washings were concentrated in vacuum, and EtOAc was added (7 mL). This organic solution was washed with 1% aqueous $NaHCO_3$ (4 × 14 mL) and water (14 mL), dried over Na_2SO_4, filtered and evaporated to dryness.

Procedure D. To a stirred suspension of SIBX (857 mg, 1.5 mmol) in dry THF (14 mL, *ca.* 0.05 M) was added 2-methoxyphenol (0.7 mmol). After stirring in the dark at room temperature for 16 h, the white suspension was filtered out from the resulting red solution. The filter cake was washed with CH_2Cl_2 (20 mL) and the combined filtrate and washings were poured into water (30 mL). After separation, the aqueous layer was further extracted with CH_2Cl_2 (30 mL). The combined organic layers were washed with saturated aqueous $NaHCO_3$ (4 × 15 mL) and treated with an aqueous solution (2 mL) of $Na_2S_2O_4$ (730 mg, 4.2 mmol) for 10 min with vigorous stirring under nitrogen in the dark. The resulting yellow solution was washed with water (10 mL), brine (10 mL), dried over Na_2SO_4, filtered and evaporated to dryness.

Pyridine-3-carbaldehyde (2). Oxidation of alcohol **1** was performed in DMSO according to the procedure A to furnish **2** as a colorless oil (73%): IR (NaCl) 1704 cm^{-1}; ^1H NMR ($CDCl_3$,

250 MHz) δ 7.48 (dd, *J* = 5.0, 7.9 Hz, 1H), 8.15-8.20 (m, 1H), 8.85 (dd, *J* = 1.6, 5.0 Hz, 1H), 9.08 (d, *J* = 1.6 Hz, 1H), 10.11 (s, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 190.7, 154.6, 151.9, 135.8, 131.4, 124.1; EIMS *m/z* (rel intensity) 107 (M^+, 100), 106 (45), 78 (74).

3,4,5-Trimethoxybenzaldehyde (4). Oxidation of alcohol **3** was performed in NMP according to the procedure A to give **4** as a white solid (86%): mp 65-68 °C (lit.[2] mp 73-74 °C); IR (NaCl) 1686 cm^{-1}; ^{1}H NMR ($CDCl_3$, 250 MHz) δ 3.90 (s, 3H), 3.91 (s, 6H), 7.10 (s, 2H), 9.84 (s, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 191.0, 153.5, 143.4, 131.6, 106.6, 60.9, 56.2; EIMS *m/z* (rel intensity) 196 (M^+, 100), 181 (43), 125 (27).

Cyclooctanone (6). Oxidation of alcohol **5** was run in NMP according to the procedure A to give **6** as a colorless oil (70%): IR (NaCl) 1697 cm^{-1}; ^{1}H NMR ($CDCl_3$, 250 MHz) δ 1.30-1.50 (m, 2H), 1.53-1.57 (m, 4H), 1.81-1.90 (m, 4H), 2.36-2.41 (m, 4H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 218.4, 41.9, 27.1, 25.6, 24.6; EIMS *m/z* (rel intensity) 126 (M^+, 100), 98 (81), 84 (38), 56 (31), 55 (100).

2-Isopropyl-5-methylcyclohexanone [*p*-menthan-3-one] (8). Oxidation of alcohol **7** was carried out either in NMP according to the procedure A, or in refluxing EtOAc for 6 h according to the procedure B, to furnish **8** as a colorless oil (87% and 93%, respectively): IR (NaCl) 1708 cm^{-1}; ^{1}H NMR ($CDCl_3$, 250 MHz) δ 0.76 (d, *J* = 6.7 Hz, 3H), 0.82 (d, *J* = 6.7 Hz, 3H), 0.92 (d, *J* = 6.1 Hz, 3H), 1.22-1.33 (m, 2H), 1.74-2.09 (m, 6H), 2.22-2.29 (m, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 212.4, 55.8, 50.8, 35.4, 33.8, 27.8, 25.8, 22.2, 21.1, 18.6; EIMS *m/z* (rel intensity) 154 (M^+, 31), 139 (43), 112 (100), 69 (64), 41 (65).

Benzo[1,3]dioxole-5-carbaldehyde [piperonal] (10). Oxidation of alcohol **9** was performed in the indicated solvent at the selected temperature (see Table 2) with 3 equivalents of SIBX according to the procedure B to furnish **10** as a yellow solid (66-100%): mp 34-37 °C (lit.[3] mp 37 °C); IR (NaCl) 1685 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.02 (s, 2H), 6.87 (d, *J* = 8.1 Hz, 1H), 7.25 (d, *J* = 1.5 Hz, 1H), 7.35 (dd, *J* = 1.5, 8.1 Hz, 1H), 9.74 (s, 1H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 190.2, 153.0, 148.7, 131.8, 128.6, 108.3, 106.8, 102.1; EIMS *m/z* (rel intensity) 150 (M^+, 85), 149 (100), 121 (25).

4-Nitrobenzaldehyde (12). Oxidation of alcohol **11** was run in refluxing THF during 30 min according to the procedure B to give **12** as a yellow solid (87%): mp 100-102 °C (lit.[4] mp 104-105 °C); IR (NaCl) 1708 cm^{-1}; ^{1}H NMR ($CDCl_3$, 250 MHz) δ 8.05 (d, *J* = 8.5 Hz, 2H), 8.36 (d, *J* = 8.8 Hz, 2H), 10.14 (s, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 190.3, 151.0, 139.9, 130.4, 124.2; EIMS *m/z* (rel intensity) 151 (M^+, 100), 150 (84), 105 (28), 77 (81).

Benzaldehyde (14). Oxidation of alcohol **13** was performed in refluxing THF during 30 min according to the procedure B to furnish **14** as a colorless oil (85%): IR (NaCl) 1701 cm^{-1}; ^1H NMR ($CDCl_3$, 200 MHz) δ 7.42-7.62 (m, 3H), 7.81-7.87 (m, 2H), 9.97 (d, J = 0.3 Hz, 1H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 192.3, 136.2, 134.3, 129.6, 128.8; EIMS *m/z* (rel intensity) 106 (M^+, 100), 105 (96), 77 (96).

1,2-Diphenylethane-1,2-dione (16). Oxidation of alcohol **15** was performed in refluxing THF for 1 h according to the procedure B to furnish **16** as a yellow solid (92%): mp 85-88 °C (lit.[2] mp 95 °C); IR (NaCl) 1664 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 7.47-7.53 (m, 4H), 7.61-7.68 (m, 2H), 7.95-7.98 (m, 4H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 194.5, 134.8, 132.9, 129.8, 128.9; EIMS *m/z* (rel intensity) 210 (M^+, 4), 105 (100), 77 (40).

(*E*)-3-Phenylpropenal [(*E*)-cinnamaldehyde] (18). Oxidation of alcohol **17** was carried out in refluxing THF for 30 min according to the procedure B to give **18** as a colorless oil (100%): IR (NaCl) 1675 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 6.70 (dd, J = 7.9, 16.2 Hz, 1H), 7.41-7.57 (m, 6H), 9.70 (d, J = 7.6 Hz, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 193.6, 152.7, 133.8, 131.2, 129.0, 128.5, 128.4; EIMS *m/z* (rel intensity) 132 (M^+, 78), 131 (100), 103 (66), 77 (64).

(*E*)-3,7-Dimethylocta-2,6-dienal [(*E*)-geranial] (20). Oxidation of alcohol **19** was performed in THF/DMSO (9:1) at room temperature for 1 h according to the procedure B to give **20** as a colorless oil (82%): IR (NaCl) 1674 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 1.55 (s, 3H), 1.62 (s, 3H), 2.11 (d, J = 1.2 Hz, 3H), 2.17 (m, 4H), 4.98-5.04 (m, 1H), 5.81 (dd, J = 1.2, 8.2 Hz, 1H), 9.93 (d, J = 8.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 191.1, 163.6, 132.7, 127.2, 122.4, 40.4, 25.6, 25.5, 17.5, 17.4; EIMS *m/z* (rel intensity) 152 (M^+, 6), 123 (5), 109 (5), 94 (12).

Cycloheptanone (22). Oxidation of alcohol **21** was performed in refluxing THF during 30 min according to the procedure B to furnish **22** as a colorless oil (77%): IR (NaCl) 1700 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 1.62-1.66 (m, 8H), 2.41-2.46 (m, 4H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 215.4, 43.7, 30.3, 24.2; EIMS *m/z* (rel intensity) 112 (M^+, 29), 97 (5), 84 (82), 69 (38), 55 (100).

3-Phenylpropanal (24). Oxidation of alcohol **23** was performed in refluxing EtOAc during 2 h according to the procedure B to give **24** as a colorless oil (93%): IR (NaCl) 1723 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 2.75-2.81 (m, 2H), 2.97 (t, J = 7.6 Hz, 2H), 7.19-7.35 (m, 5H), 9.81-9.82 (m, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 201.4, 140.2, 128.5, 128.2, 126.2, 45.1, 27.9; EIMS *m/z* (rel intensity) 134 (M^+, 47), 105 (30), 91 (100), 77 (20).

1,9-Nonanedial (26). Oxidation of diol **25** was performed in refluxing EtOAc during 2 h according to the procedure B to give **26** as a colorless oil (77%): IR (NaCl) 1723 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 1.24 (s, 6H), 1.49-1.57 (m, 4H), 2.30-2.37 (m, 4H), 9.65 (dd, J = 1.8, 3.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 202.5, 43.5, 28.8, 28.6, 21.7; EIMS *m/z* (rel intensity) 156 (M^+, 0.2), 112 (8), 95 (100), 82 (26), 57 (54), 44 (40).

3,5-Di-*tert*-butyl-4-hydroxybenzaldehyde (28). Oxidation of the phenolic alcohol **27** was run in refluxing EtOAc for 1.5 h according to the procedure B to furnish **28** as a yellow solid (94%): mp 180-181 °C (lit.[5] mp 190 °C); IR (NaCl) 1667 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 1.47 (s, 18H), 5.88 (s, 1H), 7.73 (s, 2H), 9.85 (s, 1H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 191.8, 159.7, 136.5, 128.7, 127.6, 34.3, 30.0; EIMS *m/z* (rel intensity) 234 (M^+, 35), 219 (100), 191 (15).

4-Hydroxy-3-methoxy-benzaldehyde [vanillin] (30). Oxidation of the vanillyl alcohol **29** was performed according to the procedure C. The reaction was run for 6 h, after which time it was processed as indicated. The residue was then submitted to column chromatography, eluting with hexanes/ether (1:2), to furnish vanillin (**30**) as a white solid (32%): mp 79-80 °C (lit.[6] mp 82-83 °C); IR (NaCl) 3190, 1664 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.94 (s, 3H), 6.42 (s, 1H), 7.03 (d, J = 8.6 Hz, 1H), 7.40-7.42 (m, 2H), 9.80 (s, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 190.9, 151.7, 147.1, 129.7, 127.5, 114.4, 108.7, 56.0; EIMS *m/z* (rel intensity) 152 (M^+, 100), 123 (18), 109 (28).

4-Hydroxy-3-methoxy-propenal [(*E*)-coniferaldehyde] (32). Oxidation of coniferyl alcohol **31** was performed according to the procedure C. The reaction was run for 7 h, after which time it was processed as indicated. The residue was then submitted to column chromatography, eluting with pentanes/acetone (1:1), to furnish **32** as an orange oil (18%): IR (NaCl) 3300, 1663, 1286 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.92 (s, 3H), 6.28 (bs, 1H), 6.60 (dd, J = 7.7, 15.7 Hz, 1H), 6.95 (d, J = 8.2 Hz, 1H), 7.05-7.12 (m, 2H), 7.39 (d, J = 15.9 Hz, 1H), 9.62 (d, J = 7.9 Hz, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 193.8, 153.3, 148.9, 146.9, 126.5, 126.2, 124.0, 114.9, 109.5, 55.9; EIMS *m/z* (rel intensity) 178 (M^+, 100), 150 (10), 135 (49), 124 (24).

4-Propenyl-benzene-1,2-diol [4-allylcatechol] (34). Oxidation of 4-allyl-2-methoxy-phenol [eugenol] (**33)** was performed according to the procedure D to furnish **34** as a viscous orange solid (77%): IR (NaCl) 3362, 1610 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 3.27 (d, J = 6.7 Hz, 2H), 5.02-5.09 (m, 2H), 5.50 (bs, 2H), 5.84-6.00 (m, 1H), 6.61-6.81 (m, 3H); ^{13}C NMR

($CDCl_3$, 62.9 MHz) δ 143.3, 141.5, 137.6, 133.3, 121.0, 115.8, 115.6, 115.4, 39.4; EIMS *m/z* (rel intensity) 150 (M^+, 100), 133 (20), 123 (37), 105 (11).

4-Propyl-benzene-1,2-diol (36). Oxidation of 2-methoxy-4-propylphenol (**35**) was performed according to the procedure D. The reaction was run for 12 h, after which time it was processed as indicated to furnish **36** as a viscous orange solid (89%): IR (NaCl) 3364 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 0.91 (t, *J* = 7.3 Hz, 3H), 1.50-1.65 (m, 2H), 2.46 (t, *J* = 7.3 Hz, 2H), 5.43 (s, 2H), 6.70 (m, 3H); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 143.3, 141.2, 136.0, 120.8, 115.6, 115.2, 37.3, 24.6, 13.7; EIMS *m/z* (rel intensity) 152 (M^+, 26), 123 (100), 77 (12).

***N*-Benzyl-3-(3,4-dihydroxyphenyl)-propionamide (38).** Oxidation of *N*-benzyl-3-(4-hydroxy-3-methoxyphenyl)-propionamide (**37**) was performed according to the procedure D. The residue was then submitted to column chromatography, eluting with pentanes/EtOAc/AcOH (1:1:0.01), to furnish **38** as a colorless oil (41%): IR (NaCl) 3318 cm^{-1}; ^1H NMR (acetone-d_6, 300 MHz) δ 2.48 (t, *J* = 7.2 Hz, 2H), 2.80 (t, *J* = 7.2 Hz, 2H), 3.04 (s, 1H), 4.36 (d, *J* = 6.0 Hz, 2H), 6.54 (dd, *J* = 1.9, 7.9 Hz, 1H), 6.69 (s, 1H); 6.72-6.73 (m, 1H), 7.17-7.29 (m, 5H), 7.52 (s, 1H), 7.88 (s, 1H); ^{13}C NMR (acetone-d_6, 75.5 MHz) δ 172.7, 145.8, 144.1, 140.5, 133.9, 129.1, 128.2, 127.5, 120.4, 116.4, 115.9, 43.4, 38.9, 31.9; EIMS *m/z* (rel intensity) 271 (M^+, 98), 180 (4), 137 (17), 123 (29), 91 (100); HRMS (EI) calcd for $C_{16}H_{17}NO_3$ 271.1208, found 271.1206.

4-*tert*-Butyldiphenylsilyloxymethylcatechol (40). Oxidation of 5-(*tert*-butyldiphenylsilyloxymethyl)-2-methoxyphenol (**39**) was performed according to the procedure D to furnish **40** as an orange oil (97%): IR (NaCl) 3376 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 1.10 (s, 9H), 4.66 (s, 2H), 6.72-6.88 (m, 2H), 6.89 (s, 1H), 7.35-7.44 (m, 6H), 7.69-7.33 (m, 5H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 143.4, 142.4, 135.5, 133.9, 133.4, 129.7, 127.7, 118.8, 115.2, 113.7, 65.2, 26.8, 19.2; EIMS *m/z* (rel intensity) 378 (M^+, 0.2), 321 (31), 199 (100), 123 (8); HRMS (EI) calcd for $C_{23}H_{26}O_3Si$ 378.1651, found 378.1646.

1,2-Naphthoquinone (42). To a stirred suspension of SIBX (857 mg, 1.5 mmol) in dry THF (14 mL, *ca.* 0.05 M) was added 2-methoxynaphthol (**41**, 122 mg, 0.7 mmol) in two portions in 30 minutes. After stirring in the dark at room temperature for 60 h, the white suspension was filtered out from the resulting red solution. The filter cake was washed with CH_2Cl_2 (20 mL) and the combined filtrates and washings were poured into water (30 mL). After separation, the aqueous layer was further extracted with CH_2Cl_2 (30 mL). The combined organic layers were washed with saturated aqueous $NaHCO_3$ (4 × 15 mL) and water (20 mL), dried over Na_2SO_4, filtered and evaporated. The residue was further dried under vacuum to furnish pure

42 as a red solid (91 mg, 83%): mp 126-129 °C (lit.[7] mp 119 °C); IR (NaCl) 1658 cm^{-1}; ^1H NMR ($CDCl_3$, 250 MHz) δ 6.45 (d, J = 10.4 Hz, 1H), 7.37 (dd, J = 1.2, 7.6 Hz, 1H), 7.41 (d, J = 10.4 Hz, 1H), 7.51 (dt, J = 1.5, 7.6 Hz, 1H), 7.66 (dt, J = 1.5, 7.6 Hz, 1H), 8.12 (dt, J = 1.5, 7.6 Hz, 1H); ^{13}C NMR ($CDCl_3$, 62.9 MHz) δ 180.8, 178.7, 145.4, 135.8, 134.6, 131.4, 130.7, 130.0, 129.8, 127.7; EIMS *m/z* (rel intensity) 158 (M^+, 7), 130 (100), 102 (82), 76 (24), 50 (22).

Additional References

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