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TETRAHEDRON

Manganese(III) acetate promoted acetoxylation of various α,β-unsaturated cyclopentanones

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Abstract—We describe the results of manganese(III) acetate based regioselective oxidation of various α,β-unsaturated cyclopentanones leading to α′-acetoxy α,β-unsaturated cyclopentanones in good yields. Products due to monophenyl and diphenyl substituted dimerization have been identified as byproducts of the reaction. © 2003 Elsevier Science Ltd. All rights reserved.

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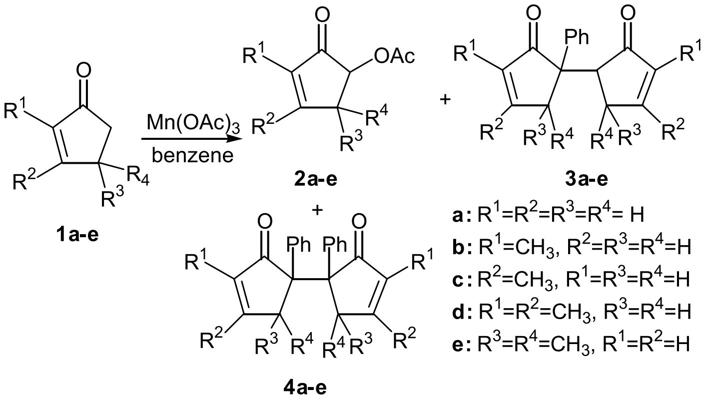
1. Introduction

Synthetic methods for the selective oxidation of common functional groups occupy a central position in organic synthesis. In particular, regioselective oxidation of enones to α′-acetoxy enones using lead(IV) acetate,[1,2] MoOPh,[3–5] triphenylphosphite ozonide (TPPO),[6,7] MCPBA[8,9] and manganese(III) acetate[10,11] has wide applicability. In the literature, there are numerous examples of regioselective oxidation of α,β-unsaturated cyclohexanone derivatives using lead(IV)[12–14] and manganese(III) acetate.[15–17] However, only a few examples of lead(IV) acetate mediated selective oxidation of α,β-unsaturated cyclopentanone derivatives[18] are to be found. Sasaki and Eguchi reported that this procedure was not always effective.[19] So far, no general procedure for the synthesis of α′-acetoxy α,β-unsaturated cyclopentanones has been reported with manganese(III) acetate. The lack of a selective oxidation method for the α′-position of α,β-unsaturated cyclopentanones prompted us to develop the method. We describe herein the results obtained from the $Mn(OAc)_3$ oxidation of various α,β-unsaturated cyclopentanones and our optimization of the reaction conditions.

2. Results and discussion

In the screening reactions, cyclopentenones **1** were examined first with $Mn(OAc)_3$ by varying the molar ratio of substrate/oxidizing agent from 1:1 to 1:3 in benzene as the solvent. A 1:2 molar ratio proved to be suitable for the regioselective acetoxylation of cyclopentenones **1** to afford products **2**. Characterization of the major product revealed that an acetoxy group had been introduced at the α′ position **2**. The dimers **3** and **4** were also observed as minor components (Scheme 1, Table 1).



Scheme 1.

Keywords: enones; manganese and compounds; oxidation.

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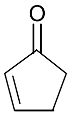
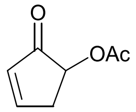
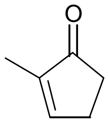
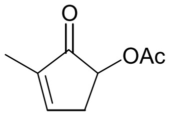
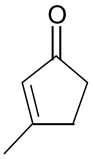
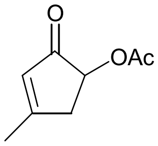
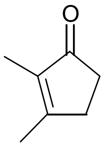
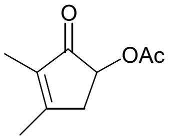
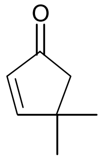
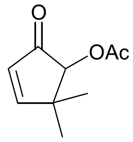
Table 1. Selective oxidation of α,β-unsaturated cyclopentanones with $Mn(OAc)_3$ in benzene

Entry	Reactant	Product	2	Yield (%)	3	Yield (%)	4	Yield (%)	Time (h)
1			**2a**	65	–		**4a**	6	8
2[a]			**2b**	79	–		**4b**	7	10
3			**2c**	81	**3c**	7	**4c**	8	11
4[b]			**2d**	77	–		**4d**	6	12
5[c]			**2e**	63	–		–		12

[a] 2-Methyl-5-phenyl-2-cyclopentenone **8b** was also isolated in 4% yield.

[b] 2,3-Dimethyl-5-phenyl-2-cyclopentenone **8d** was also isolated in 7% yield.

[c] No byproducts were isolated.

The reaction presumably proceeds via the formation of the Mn(III) enolate **5c**, which loses Mn(II) upon one-electron oxidation to give α′-keto radical **6c**[20,21] (Scheme 2). Oxidation of intermediate **6c** by another equivalent of $Mn(OAc)_3$ provides α′-acetoxy cyclopentenones **2c**.

In the screening reaction of entry 3, we found that the product distribution depends mainly on the substrate/oxidizing agent molar ratio. Using a 1:1 molar ratio, compounds **2c**, **3c** and **4c** were formed as a 5:3:2 mixture. When the substrate/oxidizing agent molar ratio was 1:2, the distribution of compounds **2c**, **3c** and **4c** was found to be 8:1:1. A similar product distribution was obtained using a 1:3 molar ratio.

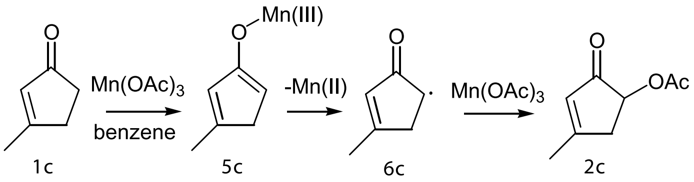
In conclusion, $Mn(OAc)_3$ oxidation of cyclopentenones can be used to introduce an α′-acetoxy group. The applicability of a one-pot oxidation method is attractive for further use in synthesis.

3. Experimental

^1H NMR spectra were recorded in $CDCl_3$ on Bruker Spectrospin Avance DPX 400 spectrometers. Chemical shifts are given in ppm from tetramethylsilane. IR spectra were obtained from a Perkin–Elmer Model 1600 series FT-IR spectrometer and are reported in cm^{-1}. Mass spectra were recorded on a Varian MAT 212. Thin layer chromatography (TLC) was performed on Merck plastic-backed silica gel plates. Compounds were visualized under an UV lamp. Column chromatography was performed using silica gel (Flash Silica 60, 32–63 μm). Cyclopentenones were purchased from Aldrich.

3.1. General procedure for the $Mn(OAc)_3$ oxidation of α,β-unsaturated ketones 2

A mixture of $Mn(OAc)_3$ (3.25 g, 14.0 mmol) in benzene (or corresponding solvent) (150 mL) was refluxed for 45 min



Scheme 2.

under a Dean-Stark trap, then cooled to room temperature. The cyclopentenone (7.0 mmol) was gradually added and the mixture was allowed to reflux until the dark brown color disappeared (TLC monitoring). The reaction mixture was diluted with ethyl acetate (150 mL) and the organic phase was washed with 1 M HCl (150 mL), saturated $NaHCO_3$ (150 mL) and brine (150 mL). The organic phase was dried over $MgSO_4$ and evaporated in vacuo. The crude product mixture was separated by flash column chromatography using ethyl acetate/hexane as eluent to afford the products noted in Table 1.

3.1.1. 5-Acetoxy-2-cyclopentenone 2a. (0.64 g, 65%) as a colorless oil; R_f (EtOAc/hexane 1:3) 0.38; ν_{max} (neat) 1743, 1635 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.76–7.78 (1H, m, C*H*=CHCO), 6.34–6.36 (1H, m, CH=C*H*CO), 5.20 (1H, dd, *J*=3, 7 Hz, C*H*OAc), 3.21–3.29 (1H, m, C*H*$_a$H$_b$CHOAc), 2.66–2.73 (1H, ddd, *J*=3, 5, 19 Hz, CH$_a$*H*$_b$CHOAc), 2.22 (3H, s, *Me*CO_2); δ_C (100.6 MHz, $CDCl_3$) 202.5, 174.2, 169.1, 129.3, 75.2, 40.4, 21.2; HRMS (EI): M^+, found 140.0468. $C_7H_8O_3$ requires 140.0473.

3.1.2. 5-Acetoxy-2-methyl-2-cyclopentenone 2b. (0.85 g, 79%) as a colorless oil; R_f (EtOAc/hexane 1:2) 0.35; ν_{max} (neat) 3018, 2926, 1730, 1716, 1216 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.05 (1H, d, *J*=7 Hz, C*H*C=CMe), 4.90 (1H, d, *J*=7 Hz, C*H*OAc), 2.80–2.85 (1H, ddd, *J*=7, 7, 18 Hz, C*H*$_a$H$_b$CHOAc), 2.24 (1H, d, *J*=18 Hz, CH$_a$*H*$_b$CHOAc), 1.91 (3H, s, *Me*CO_2), 1.58 (3H, s, *Me*C=); δ_C (100.6 MHz, $CDCl_3$) 204.2, 170.9, 155.2, 140.9, 72.2, 34.1, 21.1, 10.8; *m/z* (EI) 154 (100), 112 (6%); HRMS (EI): M^+, found 154.0625. $C_8H_{10}O_3$ requires 154.0630.

3.1.3. 5-Acetoxy-3-methyl-2-cyclopentenone 2c. (0.87 g, 81%) as a colorless oil; R_f (EtOAc/hexane 1:3) 0.36; ν_{max} (neat) 1740, 1629 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 5.91 (1H, s, C*H*=), 5.05 (1H, dd, *J*=3, 7 Hz, C*H*OAc), 2.96 (1H, dd, *J*=7, 18 Hz, C*H*$_a$H$_b$CHOAc), 2.43 (1H, dd, *J*=3, 18 Hz, CH$_a$*H*$_b$CHOAc), 2.09 (3H, s, *Me*CO_2), 2.05 (3H, s, *Me*C=); δ_C (100.6 MHz, $CDCl_3$) 203.6, 176.2, 170.8, 129.1, 73.1, 40.0, 21.1, 20.1; HRMS (EI): M^+, found 154.0628. $C_8H_{10}O_3$ requires 154.0630.

3.1.4. 5-Acetoxy-2,3-dimethyl-2-cyclopentenone 2d. (0.90 g, 77%) as a colorless oil; R_f (EtOAc/hexane 1:4) 0.36; ν_{max} (neat) 3018, 2923, 1742, 1708, 1219 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 5.05 (1H, d, *J*=6 Hz, C*H*OAc), 2.89–2.95 (1H, dd, *J*=6, 18 Hz, C*H*$_a$H$_b$CHOAc), 2.37 (1H, d, *J*=18 Hz, CH$_a$*H*$_b$CHOAc), 2.06 (3H, s, *Me*CO_2), 2.00 (3H, s, *Me*C=CMeCO), 1.66 (3H, s, *Me*COC=); δ_C (100.6 MHz, $CDCl_3$) 203.6, 170.9, 167.7, 135.4, 72.0, 39.1, 21.1, 17.5, 8.3; *m/z* (EI) 168 (100), 126 (23), 108 (8%); HRMS (EI): M^+, found 168.0779. $C_9H_{12}O_3$ requires 168.0786.

3.1.5. 5-Acetoxy-4,4-dimethyl-2-cyclopentenone 2e. (0.74 g, 63%) as a colorless oil; R_f (EtOAc/hexane 1:3) 0.34; ν_{max} (neat) 2968, 2929, 2871, 1738, 1721, 1236 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.30 (1H, d, *J*=6 Hz, C*H*=CHCO), 5.88 (1H, d, *J*=6 Hz, CH=C*H*CO), 4.85 (1H, s, C*H*OAc), 1.98 (3H, s, *Me*CO_2), 1.13 (3H, s, *Me*MeC), 0.86 (3H, s, Me*Me*C); δ_C (100.6 MHz, $CDCl_3$) 203.3, 171.7, 170.5, 129.3, 80.0, 44.4, 26.2, 23.5, 20.7; HRMS (EI): M^+, found 168.0782. $C_9H_{12}O_3$ requires 168.0786.

3.2. Byproducts of the reaction

3.2.1. 4,4′-Dimethyl-1-phenyl-bicyclopentyl-3,3′-diene-2,2′-dione 3c. (0.13 g, 7%) as a colorless oil; R_f (EtOAc/hexane 1:3) 0.33; ν_{max} (neat) 1717, 1623, 755 cm^{-1}; δ_H (400 MHz, $CDCl_3$) (7:3 diastereomeric mixture) (major isomer) 7.03–7.27 (5H, m, *Ph*), 5.97 (1H, s, C*H*=), 5.94 (1H, s, C*H*=), 3.49–3.53 (1H, m, C*H*CO), 3.20–3.25 (1H, m, C*H*$_a$H$_b$CPh), 3.00 (2H, m, C*H*$_2$CHCO), 2.61 (1H, m, CH$_a$*H*$_b$CPh), 2.17 (3H, s, *Me*C=), 2.16 (3H, s, *Me*C=); (minor isomer) 7.03–7.27 (5H, m, *Ph*), 5.93 (1H, s, C*H*=), 5.90 (1H, s, C*H*=), 3.49–3.53 (1H, m, C*H*CO), 3.20–3.25 (1H, m, C*H*$_a$H$_b$CPh), 3.00 (2H, m, C*H*$_2$CHCO), 2.61 (1H, m, CH$_a$*H*$_b$CPh), 2.15 (3H, s, *Me*C=), 2.13 (3H, s, *Me*C=); δ_C (100.6 MHz, $CDCl_3$) (major isomer) 209.0, 207.2, 177.8, 169.8, 156.0, 137.8, 129.5, 128.9, 128.7, 128.5, 127.6, 124.5, 84.6, 52.7, 42.8, 35.5, 21.7, 19.6; (minor isomer) 202.0, 200.6, 173.9, 159.5, 139.8, 130.7, 129.8, 128.8, 128.6, 128.4, 125.6, 126.9, 124.6, 85.4, 46.9, 38.8, 21.1, 19.4; *m/z* (EI) 266 (6), 232 (9), 172 (100), 171 (74), 141 (10), 128 (39), 105 (42), 77 (26%); HRMS (EI): ($M-C_6H_8O$), found 172.0885. $C_{12}H_{10}O$ requires 172.0888.

3.2.2. 1,1′-Diphenyl-bicyclopentyl-3,3′-diene-2,2′-dione 4a. (0.13 g, 6%) as a colorless oil; R_f (EtOAc/hexane 1:3) 0.36; ν_{max} (neat) 1673, 1632, 785 cm^{-1}; δ_H (400 MHz, $CDCl_3$) (8:2 diastereomeric mixture) (major isomer) 7.44–7.52 (2×1H, m, C*H*=CHCO), 7.07–7.35 (2×5H, m, Ph), 6.06–6.12 (2×1H, m, CH=C*H*CO), 2.65–3.25 (2×2H, m, C*H*$_2$); (minor isomer) 7.38–7.41 (2×1H, m, C*H*=CHCO), 7.07–7.35 (2×5H, m, Ph), 6.01–6.04 (2×1H, m, CH=C*H*CO), 2.65–3.25 (2×2H, m, C*H*$_2$); δ_C (100.6 MHz, $CDCl_3$) (major isomer) 204.3, 200.3, 177.3, 176.5, 144.7, 135.3, 130.2, 128.6, 128.4, 128.1, 127.8, 127.6, 127.3, 71.5, 71.3, 45.4, 43.2; (minor isomer) 200.1, 175.8, 142.3, 129.5, 128.5, 128.2, 127.5, 71.0, 44.1; *m/z* (EI) 314 (100), 252 (7), 158 (23%). HRMS (EI): M^+, found 314.1313. $C_{22}H_{18}O_2$ requires 314.1306.

3.2.3. 3,3′-Dimethyl-1,1′-diphenyl-bicyclopentyl-3,3′-diene-2,2′-dione 4b. (0.17 g, 7%) as a colorless oil; R_f (EtOAc/hexane 1:2) 0.33; ν_{max} (neat) 1680, 1644, 787 cm^{-1}; δ_H (400 MHz, $CDCl_3$) (7:3 diastereomeric mixture) (major isomer) 7.05–7.53 (2×5H, m, Ph), 6.91–7.01 (2×1H, m, C*H*=), 2.91–3.94 (2×2H, m, C*H*$_2$), 1.47 (3H, s, *Me*C=), 1.42 (3H, s, *Me*C=); (minor isomer) 7.05–7.53 (2×5H, m, Ph), 6.81 (2×1H, d, *J*=7 Hz, C*H*=), 2.91–3.94 (2×2H, m, C*H*$_2$), 1.47 (2×3H, s, *Me*C=); δ_C (100.6 MHz, $CDCl_3$) (major isomer) 204.0, 202.5, 177.6, 177.2, 143.4, 140.5, 131.5, 129.2, 129.4, 128.6, 128.5, 127.9, 127.8, 127.5, 127.4, 127.1, 74.5, 70.6, 46.2, 45.3, 20.8, 19.3; (minor isomer) 200.6, 176.2, 143.1, 129.0, 128.9, 128.3, 127.6, 74.1, 45.0, 20.5; *m/z* (EI) 342 (100), 278 (26), 172 (33%). HRMS (EI): M^+, found 342.1636. $C_{24}H_{22}O_2$ requires 342.1620.

3.2.4. 4,4′-Dimethyl-1,1′-diphenyl-bicyclopentyl-3,3′-diene-2,2′-dione 4c. (0.19 g, 8%) as a colorless oil; R_f (EtOAc/hexane 1:3) 0.32; ν_{max} (neat) 1687, 1631, 780 cm^{-1}; δ_H (400 MHz, $CDCl_3$) (7:3 diastereomeric mixture) (major isomer) 6.99–7.20 (2×5H, m, Ph), 5.64–5.68 (2×1H, m, C*H*=), 3.85–3.90 (2×1H, m, C*H*$_a$H$_b$), 2.48–2.54 (2×1H, m, CH$_a$*H*$_b$), 1.99 (3H, s, *Me*C=), 1.97

(3H, s, *Me*C=); (minor isomer) 6.99–7.20 (2×5H, m, Ph), 5.74 (2×1H, s, C*H*=), 3.85–3.90 (2×1H, m, CH_aH$_b$), 2.48–2.54 (2×1H, m, CH$_a$$H_b$), 1.99 (2×3H, s, *Me*C=); δ_C (100.6 MHz, $CDCl_3$) (major isomer) 208.9, 206.5, 177.8, 176.7, 140.4, 139.7, 130.4, 129.9, 129.7, 128.7, 128.6, 128.5, 128.3, 128.2, 128.1, 127.6, 127.5, 127.4, 127.0, 74.1, 60.1, 53.2, 45.9, 20.1, 19.7; (minor isomer) 206.1, 177.1, 140.2, 129.5, 127.7, 128.9, 127.3, 74.4, 53.1, 20.4; *m/z* (EI) 342 (13), 172 (100), 141 (3), 128 (12), 115 (4), 77 (3%). HRMS (EI): ($M-C_{12}H_{10}O$), found 172.0888. $C_{12}H_{12}O$ requires 172.0888.

3.2.5. 3,3′,4,4′-Tetramethyl-1,1′-diphenyl-bicyclopentyl-3,3′-diene-2,2′-dione 4d. (0.16 g, 6%) as a colorless oil; R_f (EtOAc/hexane 1:4) 0.33; ν_{max} (neat) 3835, 3742, 3615, 1691, 1651 cm^{-1}; δ_H (400 MHz, $CDCl_3$) (6:4 diastereomeric mixture) (major isomer) 7.06–7.18 (2×5H, m, Ph), 3.79–3.84 (2×1H, m, CH_aH$_b$), 2.39–2.48 (2×1H, m, CH$_a$$H_b$), 1.88 (3H, s, *Me*C=CMe), 1.86 (3H, s, *Me*C=CMe), 1.53 (3H, s, MeC=C*Me*), 1.52 (3H, s, MeC=C*Me*); (minor isomer) 7.06–7.18 (2×5H, m, Ph), 3.79–3.84 (2×1H, m, CH_aH$_b$), 2.39–2.48 (2×1H, m, CH$_a$$H_b$), 1.88 (2×3H, s, *Me*C=CMe), 1.53 (2×3H, s, MeC=C*Me*); δ_C (100.6 MHz, $CDCl_3$) (major isomer) 206.4, 202.4, 177.1, 175.2, 145.4, 142.1, 130.2, 129.7, 129.3, 128.8, 128.4, 128.2, 127.8, 127.5, 127.2, 127.1, 76.1, 75.3, 44.6, 44.2, 20.2, 19.8, 19.2, 9.8; (minor isomer) 204.5, 175.8, 143.6, 129.9, 128.7, 128.6, 127.9, 127.4, 75.8, 46.7, 17.3, 10.9; *m/z* (EI) 370 (2), 186 (100), 142 (11%); HRMS (EI): M^+, found 370.1932. $C_{26}H_{26}O_2$ requires 370.1934.

3.2.6. 2-Methyl-5-phenyl-2-cyclopentenone 8b. (0.05 g, 4%) as a colorless oil; R_f (EtOAc/hexane 1:2) 0.38; ν_{max} (neat) 3010, 2913, 1692, 1643, 782 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.16–7.37 (5H, m, Ph), 7.07 (1H, d, *J*=7 Hz, C*H*=), 3.49 (1H, dd, *J*=2, 7 Hz, C*H*Ph), 3.01–3.07 (1H, m, CH_aH$_b$), 2.60 (1H, dd, *J*=2, 19 Hz, CH$_a$$H_b$), 1.77 (3H, s, *Me*C=); δ_C (100.6 MHz, $CDCl_3$) 206.3, 177.2, 140.5, 130.5, 129.2, 128.7, 127.9, 127.1, 126.9, 61.2, 44.1, 17.3; HRMS (EI): M^+, found 172.0886. $C_{12}H_{12}O$ requires 172.0888.

3.2.7. 2,3-Dimethyl-5-phenyl-2-cyclopentenone 8d. (0.09 g, 7%) as a colorless oil; R_f (EtOAc/hexane 1:4) 0.39; ν_{max} (neat) 3025, 2933, 1668, 1632 cm^{-1}; δ_H (400 MHz, $CDCl_3$) 7.02–7.52 (5H, m, Ph), 3.47 (d, 1H, *J*=7 Hz),1.67 (s, 3H), 2.03 (s, 3H), 2.50 (d, 1H, *J*=18 Hz), 2.91–2.98 (dd, 1H, *J*=7, 18 Hz). ^{13}C NMR: δ 10.1, 17.8, 45.2, 75.3, 127.2, 127.4, 127.8, 128.4, 129.1, 130.2, 143.4, 176.1, 204.5; *m/z* (EI) 186 (100), 171 (46), 143 (46), 128 (23), 104 (15%); HRMS (EI): M^+, found 186.1040. $C_{13}H_{14}O$ requires 186.1045.

Acknowledgements

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