resulting mixture was extracted with ether, and the ether extracts were washed with water and dried over Na_2SO_4. Flash chromatography over silica gel (elution with 80% hexane–20% ethyl acetate) afforded 1,2,3,4-tetraphenylbenzene (**7a**), 0.65 g (85%), mp 188–189 °C (lit.[8] mp 190 °C); ^{1}H NMR δ 6.75 (m, 4 H), 6.9 (m, 6 H), 7.08 (m, 4 H), 7.15 (m, 6 H), 7.3 (s, 2 H).

2,3-Bis(*p*-methoxyphenyl)-1,4-diphenylbenzene (7b). A 0.518-g (2.2-mmol) portion of **13a** and 0.879 g (2.0 mmol) of **4b** were reacted as described for the previous reaction between **13a** and **4a**. 2,3-Bis(*p*-methoxyphenyl)-1,4-diphenylbenzene (**7b**) was isolated by flash chromatography as a white crystalline solid: 0.80 g (90%); mp 186–189 °C (lit.[22] mp 185–186 °C); ^{1}H NMR δ 3.7 (s, 6 H), 6.5 (d, 4 H, J = 8 Hz), 6.7 (d, 4 H, J = 8 Hz), 7.1–7.3 (m, 10 H), 7.5 (s, 2 H).

2,3-Bis(*p*-methylphenyl)-1,4-diphenylbenzene (7c). **13a** (0.583 g, 2.4 mmol) and 0.825 g (2.0 mmol) of **4c** were reacted as described for the previous reaction between **13a** and **4a**. 2,3-Bis(*p*-methylphenyl)-1,4-diphenylbenzene (**7c**) was isolated by flash chromatography as a white crystalline solid: 0.472 g (58%); mp 204–206 °C; ^{1}H NMR δ 2.1 (s, 6 H), 6.65 (d, 4 H), 6.70 (d, 4 H), 7.0–7.1 (s, 10 H), 7.4 (s, 2 H). Anal. Calcd for $C_{32}H_{26}$: C, 93.62; H, 6.38. Found: C, 93.51; H, 6.29.

2,3-Bis(*p*-(dimethylamino)phenyl)-1,4-diphenylbenzene (7d). A 0.540-g (2-mmol) sample of **13a** and 1.1371 g (2.4 mmol) of **4d** were reacted as described for the previous reaction between **13a** and **4a**. 2,3-Bis(*p*-(dimethylamino)phenyl)-1,4-diphenylbenzene (**7d**) was isolated as a light brown solid, 0.46 g (49%), mp 234–236 °C; white solid, mp 236–237 °C after recrystallization from ethanol and flash chromatography; ^{1}H NMR δ 3.0 (s, 12 H), 6.55 (d, 4 H), 6.85 (d, 4 H), 7.3–7.4 (m, 10 H), 7.7 (s, 2 H); exact mass calcd for $(C_{34}H_{32}N_2 + H^+)$ 469.2644, found 469.2646.

Reaction of 13a (without Isolation) with 4a. 2-Ethylfuran (**11a**) (0.807 g, 8.40 mmol) and **12** (0.1.19 g, 8.38 mmol) were heated overnight at 100 °C under nitrogen. At that point, 30 mL of diglyme and 1.54 g (4.01 mmol) of **4a** were added. The mixture was refluxed overnight. TLC analysis of the resulting solution demonstrated the absence of **4a** and the presence of only **7a** and **13a**.

Reaction of a 1:1:1 Mixture of 11b, 12, and 4a. A mixture of 0.919 g (7.4 mmol) of **11b**, 1.05 g (7.4 mmol) of **12**, and 2.850 g (7.4 mmol) of **4a** ws refluxed in diglyme for 42 h under nitrogen. The mixture was then diluted with an equal quantity of water and extracted with benzene. The combined benzene extracts were dried over $MgSO_4$ and then evaporated in vacuum. Filtration afforded 1,2,3,4-tetraphenylbenzene (**7a**) as a white powder, mp 178–180 °C, together with a supernatant liquid. Recrystallization afforded 2.60 g (92%) of white crystalline material, mp 194 °C (uncorr.) (lit.[18] mp 190 °C). Flash chromatography of the supernatant liquid from the initial filtration afforded 3,4-dicarbomethoxy-2-*n*-butylfuran (**14b**): 1.07 g (61%); ^{1}H NMR δ 0.95 (t, 3 H), 1.37 (m, 2 H), 1.67 (m, 2 H), 2.90 (t, 2 H), 3.83 (s, 3 H), 3.87 (s, 3 H), 7.79 (s, 1 H). Anal. Calcd for $C_{12}H_{16}O_5$: C, 59.99; H, 6.71. Found: C, 60.31; H, 6.40.

Reaction of 13b (without Isolation) with 15. A 0.479-g (3.86-mmol) portion of 2-*n*-butylfuran (**11b**) and 0.550 g (3.87 mmol) of **12** were heated overnight at 100 °C under nitrogen. At that point, 15 mL of diglyme and 1.0 g (1.92 mmol) of **15** were added. The reaction was allowed to reflux for 20 h. The reaction mixture was worked up as described for **7a** to afford 2.15 g of crude material, from which deposited ca 0.85 g (86%) of 1,4-dimethyl-2,3-diphenylbenzene (**16a**) as off-white crystals, mp 102 °C. Flash chromatography afforded white crystals, mp 110–112 °C (lit. 109 °C[13] and 113 °C[12]); ^{1}H NMR δ 2.2 (s, 6 H), 7.07 (dd, 4 H), 7.18 (m, 2 H), 7.24 (m, 4 H), 7.32 (s, 2 H).

Reaction of a 1:1:1 Mixture of 11b, 12, and 15. A mixture consisting of 0.481 g (3.88 mmol) of **11b**, 0.549 g (3.87 mmol) of **12**, and 1.02 g (1.96 mmol) of **15** in 15 mL of diglyme was refluxed for 3 days. The reaction was worked up as usual to afford a light brown viscous liquid, which slowly deposited 1,4-dimethyl-2,3-diphenyl-5,6-dicarbomethoxybenzene (**16b**) as an off-white crystalline material: mp 120–142 °C; 0.321 g (0.86 mmol, 22%); ^{1}H NMR δ 2.06 (s, 6 H), 3.90 (s, 6 H), 6.95 (dd, 4 H), 7.1–7.25 (m, 6 H). Flash chromatography over silica gel afforded material of mp 210–212 °C (lit.[13] mp 212 °C). TLC analysis demonstrated the absence of **16a** in this material but indicated the presence of a small amount of **16a** in the supernatant liquid, in addition to more **16b**.

Acknowledgment. Financial support by the National Science Foundation in the form of Grant no. 85-02078 to A.J.F. and a grant for partial support of the 400-MHz NMR spectrometer used in this work is gratefully acknowledged. Compound **4d** was synthesized in this laboratory by Peter C. Fox according to directions kindly provided by Dr. M. A. Ogliaruso of Virginia Polytechnic Institute and State University.

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Crystal Structure Analysis of an Osmium(VI) Bisglycolate Produced by Reaction of a Sterically Hindered Chiral Nonracemic Alkene with Osmium Tetroxide

Matthew R. Sivik, Judith C. Gallucci,[1] and Leo A. Paquette*

Evans Chemical Laboratories, The Ohio State University, Columbus, Ohio 43210

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Osmium tetroxide is widely recognized to be the premier reagent for accomplishing clean syn-dihydroxylation of carbon–carbon double bonds.[2] Since introduction of the process by Criegee in 1936,[3] several mechanistic investigations of stoichiometric level reactions have appeared.[4] More recently, Sharpless and his co-workers have attempted to gain detailed insight into the course of chiral-catalyzed asymmetric variants to this serviceable reaction.[5] A detailed kinetic study by Erdik and Matteson of OsO_4-catalyzed trimethylamine *N*-oxide oxidations has also just appeared.[6] *N*-Methylmorpholine *N*-oxide (NMO) constitutes an excellent alternative oxidizing agent.[7]

When amine oxides are present, the kinetics have been construed to require operation of the mechanistic sequence depicted in Scheme I, especially when hindered alkenes (e.g., α-pinene) are involved.[5b,6] Particularly noteworthy is the need to claim intervention of the osmium(VI) bisglycolate **3**. A direct consequence of the relatively substitution-inert character of such little known esters is that the step involving the conversion of **3** to **4** is rate-determining overall.

In those circumstances where the diol components to **3** are quite bulky, oxidation at osmium should be sterically retarded. Therefore, the concentration of **3** might be ex-

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[1] Author to whom queries concerning the X-ray crystallographic analysis should be directed.

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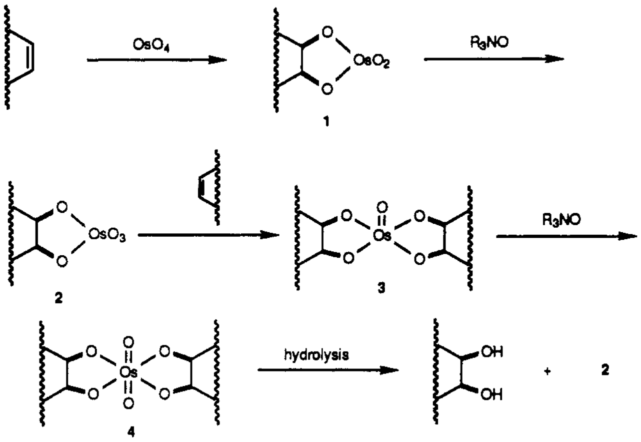
0022-3263/90/1955-0391$02.50/0

Scheme I



pected to increase to the point where isolation should prove feasible, as long as other modes of degradation are not operative. Of the several esters of type 3 that have been examined crystallographically,[8] all have been prepared by condensation of OsO_4 or $K_2OsO_4 \cdot 2H_2O$ with a 1,2-glycol.[3,9,10] A structural feature common to oxobis(1,2-diolato)osmates is the location of the metal in a square-pyramidal environment with four oxygens constituting the base such that S_2 crystallographic symmetry is attained. Herein we describe the isolation and full characterization of an intermediate akin to 3 obtained directly from an alkene osmylation, thereby providing strong supportive evidence for the mechanistic pathway advanced above.

During studies aimed at a total synthesis of austalide B,[11] we subjected 5 having an optical purity of 50% ee[12] to cis-hydroxylation according to the procedure of Van Rheenen et al.[7] Due to the slow rate of reaction noted under the usual catalytic conditions, the amount of OsO_4 was increased to 15 mol %. With this molar ratio of oxidant, reaction was complete in approximately 1 week as judged by TLC analysis. Within 2 h of mixing of the reagents, an intense green color developed and persisted until sodium dithionite was introduced during the workup. Diol 7 was subsequently isolated in 70% yield (Scheme II).

The colored intermediate was successfully isolated when a ratio of OsO_4:5:NMO of 1:2:1 was utilized. After 10 days, direct solvent evaporation provided a green oil, chromatography of which on silica gel furnished in 70% yield a mixture of osmium(VI) bisglycolates rich in 6.[13] This ester, which exhibits a characteristic downfield triplet at δ 5.44 (J = 2.7 Hz) in $CDCl_3$, was easily obtained in a pure state by recrystallization from ethyl acetate–petroleum ether. As a likely consequence of its high structural symmetry (both halves have the same sense of chirality), 6 crystallizes much more readily than the minor isomer. The olive-green hexagonal rods of 6 have well-formed faces.

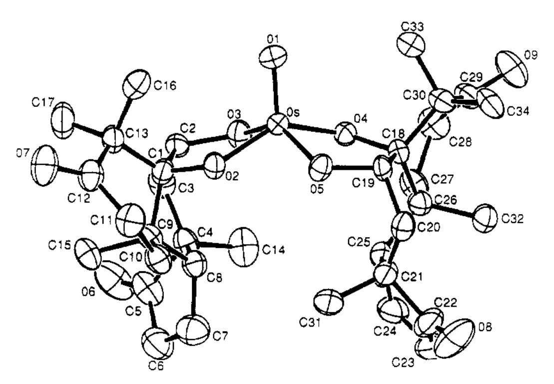


Figure 1. ORTEP drawing for 6 showing the arbitrary numbering scheme. The hydrogen atoms have been omitted for clarity.

The ORTEP diagram in Figure 1, which shows that C–O bond formation has occurred on the more open face of 5, does not display the ethyl acetate molecule of solvation that is also present (as 6:0.5 EtOAc). The osmium(VI) atom is square-pyramidal, and the molecule displays a noncrystallographic 2-fold axis along the Os–O(1) bond. An interesting feature is the relative position of the metal, which resides 0.66 Å out of the least-squares plane defined by atoms O(2), O(3), O(4), and O(5). The terminal Os=O bond that forms the apex of the pyramid is somewhat shorter in length (1.660–1.730 Å).[8] Of the four basal Os–O distances, two bond lengths (mean 1.893 Å) are significantly longer than the other two (mean 1.882 Å). Also, the individual O(apical)–Os–O(basal) angles range from 109.8 to 111.5°. A slight deviation away from square-pyramidal toward trigonal-bipyramidal coordination is thus indicated.

The isolation of 6 provides important confirmatory insight into the mechanism of OsO_4 oxidations. Whereas variations in the relative amounts of NMO (from 1.5 to 6.0 equiv) had no effect on the ultimate yield of diol 7 (70 ± 2% at 15 mol % of OsO_4), the capacity for isolating 6 dropped off progressively as the NMO levels were increased. More importantly, the pure osmium(VI) bisglycolate 6 has been observed to turn over very well under the normal *catalytic* osmylation reaction conditions in the presence of 5 and oxidizing agent (NMO), but without any other source of osmium. The relevance of 6 (and presumably other structurally comparable osmate esters) to the pathway proposed in Scheme I has now been directly documented. Although the osmium atom in 6 is sterically

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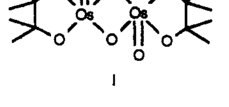
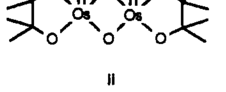


i



ii

(10) Treatment of cyclohexene with $OsO_4 \cdot C_7H_{13}N$, the monoquinuclidine complex, in diethyl ether at a molar ratio of 1:1 yields a species structurally related to i: Cartwright, B. A.; Griffith, W. P.; Schroder, M.; Skapski, A. C. *J. Chem. Soc., Chem. Commun.* **1978**, 853.

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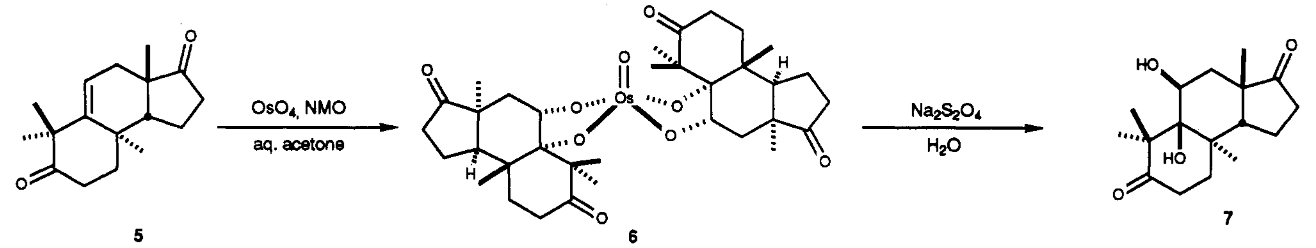
Scheme II



shielded, this ester serves an important role as a key intermediate in the catalytic osmylation process.

Experimental Section

(3a*S*,5*S*,5a*S*,9a*R*,9b*R*)-Octahydro-5,5a-dihydroxy-3a,6,6,9a-tetramethyl-1*H*-benz[*e*]indene-3,7(2*H*,3a*H*)-dione (7). A magnetically stirred solution of 5[12] (2.50 g, 9.6 mmol) and 4-methylmorpholine *N*-oxide (1.69 g, 14.4 mmol) in 10% aqueous acetone (35 mL) was treated with 2.8 mL of 0.5 M osmium tetroxide in acetone and stirred at room temperature for 7 days. The dark green reaction mixture was treated with excess aqueous sodium dithionite solution, stirred for an additional 12 h, and diluted with ethyl acetate (20 mL). The separated aqueous phase was extracted with ethyl acetate (2 × 10 mL), and the combined organic solutions were washed with brine (20 mL), dried, and concentrated. The residue was purified by flash chromatography on silica gel (elution with 30% ethyl acetate in petroleum ether) to give 7 (1.98 g, 70%) as a colorless crystalline solid: mp 178–179 °C (from ethyl acetate/petroleum ether); IR ($CHCl_3$, cm^{-1}) 3610, 1740, 1695, 1210; ^1H NMR (300 MHz, $CDCl_3$) δ 3.94 (dd, J = 21.1, 6.1 Hz, 1 H), 2.90–2.79 (m, 1 H), 2.47–1.65 (series of m, 11 H), 1.56 (dd, J = 11.0, 13.0 Hz, 1 H), 1.33 (s, 3 H), 1.28 (s, 3 H), 1.09 (s, 3 H), 0.63 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 220.81, 217.84, 79.45, 67.96, 52.45, 50.40, 48.95, 41.50, 35.25, 34.09, 33.60, 33.08, 27.38, 25.36, 23.01, 18.59, 18.08; MS *m/z* (M^+) calcd 294.1831, obsd 294.1831; $[\alpha]^{25}_D$ +110° (*c* 1.0, $CHCl_3$).

Anal. Calcd for $C_{17}H_{26}O_4$: C, 69.36; H, 8.90. Found: C, 69.24; H, 8.84.

Bis[(3a*S*,5*S*,5a*S*,9a*R*,9b*R*)-octahydro-5,5a-dihydroxy-3a,6,6,9a-tetramethyl-1*H*-benz[*e*]indene-3,7(2*H*,3a*H*)-dionato(2–)-O^5,O^{5a}]oxoosmium(VI) (6). A magnetically stirred solution of 5 (200 mg, 0.77 mmol) and 4-methylmorpholine *N*-oxide (45 mg, 0.38 mmol) in 30% aqueous acetone (5 mL) was treated with 3.25 mL of a 0.12 M solution of osmium tetroxide in *tert*-butyl alcohol and stirred under a nitrogen atmosphere for 10 days. The reaction mixture was concentrated in vacuo, and the residue was purified by silica gel chromatography (elution with 70% ethyl acetate in petroleum ether) to give a dark green oil (210 mg, 70%). Crystallization of this material from ethyl acetate–petroleum ether afforded 6 as olive-green hexagonal rods: mp > 150 °C dec; IR ($CHCl_3$, cm^{-1}) 2960, 1725, 1700, 1225, 1000, 980; ^1H NMR (300 MHz, $CDCl_3$) δ 5.44 (t, J = 2.7 Hz, 2 H), 3.18–3.07 (m, 2 H), 2.72 (dd, J = 2.9, 16.1 Hz, 2 H), 2.49 (td, J = 2.7, 16.1 Hz, 2 H), 2.58–2.42 (m, 4 H), 2.24–1.80 (series of m, 10 H), 1.65–1.53 (m, 2 H), 1.30 (s, 6 H), 1.16 (s, 6 H), 1.09 (s, 6 H), 0.82 (s, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm) 222.52, 215.86, 101.10, 91.93, 56.90, 47.92, 46.82, 42.71, 34.91, 34.04, 33.58, 31.13, 27.23, 22.57, 20.96, 17.89 (one signal not observed). The peaks for ethyl acetate of solvation are not reported in either spectrum; $[\alpha]^{25}_D$ +190° (*c* 0.1, $CHCl_3$).

Demonstration of the Catalytic Action of 6. To a solution of 6 (10 mg, 13 μmol) and 5 (6.6 mg, 25 μmol) in 1 mL of acetone was added 2 drops of water and 4.5 mg (38 μmol) of methylmorpholine *N*-oxide. After 2 days of stirring at room temperature, the majority of 5 had been consumed (TLC analysis). Sodium dithionite (16 mg, 92 μmol) was added followed by 4 drops of water. The mixture was stirred for 30 min, diluted with ethyl acetate (5 mL), and washed successively with water (2 mL), saturated copper(II) sulfate solution (4 mL), and brine (4 mL). Each aqueous washing was extracted with the same portion of ethyl acetate (3 × 5 mL). The combined organic phases were dried and concentrated. The residue was purified by flash chromatography on silica gel (elution with 10% ethyl acetate in petroleum ether) to give 9.7 mg of colorless, crystalline 7 (65% based on total available diol).

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Registry No. 5, 123700-06-1; 6, 123700-08-3; 6 diastereomer, 123805-45-8; 7, 123700-07-2; NMO, 7529-22-8; OsO_4, 20816-12-0; austalide B, 81543-02-4.

Supplementary Material Available: Crystallographic procedure, tables of crystallographic data, bond angles, bond distances, positional parameters, and anisotropic thermal parameters for 6 (15 pages). Ordering information is given on any current masthead page.

Addition of Grignard and Organolithium Reagents to *N*-Sulfonyl Aldimines Generated in Situ from Aldehydes and *N*-Sulfinyl Sulfonamides

Joseph Sisko and Steven M. Weinreb*

Department of Chemistry, The Pennsylvania State University, University Park, Pennsylvania 16802

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The addition of organometallic reagents to imines is often a poor synthetic reaction due to competing processes such as α-deprotonation and reductive dimerization. Alkyl and aryl Grignard and organolithium species have proven particularly troublesome, although some useful solutions to the problem have recently been presented.[1-4]

We have described the reaction of aldehydes with *N*-sulfinyl sulfonamides in the presence of Lewis acids to generate *N*-sulfonyl imines (Kresze reaction[5]) and subsequent trapping of these intermediates in ene-like reactions

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