systems from the dione 4 unambiguously establishes the endo,syn,endo stereostructure of the bisadduct 1. We are now looking into the different conditions of photolysis to achieve intramolecular [2 + 2] photocycloaddition of the bisadduct 1 and the analogues 4 and 5 to lead to a bishomo-seco heptaprismane derivative and related polycycles.

Experimental Section

IR spectra were recorded on a Perkin-Elmer 781 spectrophotometer. ^{1}H NMR (270, 90, 60 MHz) and ^{13}C NMR (67.89, 22.5 MHz) were recorded on Brucker WH 270, JEOL FX 90Q, and Varian T 60 spectrometers. Chemical shifts and splittings are reported in standard fashion (δ) with reference to internal tetramethylsilane; "st" indicates additional fine structure. Melting points are uncorrected. Elemental analyses were carried out on a Carlo Erba 1106 instrument. Acme 60-120-mesh silica gel was used for column chromatography. Acme silica gel G (containing 13% calcium sulfate as binder) was used for TLC. Liquid ammonia was obtained from the Mysore Ammonia Co. in cylinders and was dried over sodium and distilled directly into the reaction flask prior to use. THF was distilled over sodium benzophenone ketyl. The bisadduct 1 was prepared according to the literature procedure[2] from freshly prepared 1,4-cyclohexadiene (from benzene) and 1,2,3,4-tetrachloro-5,5-dimethoxycyclopentadiene (from hexachlorocyclopentadiene).

1,4,4a,5,8,8a,9,9a,10,10a-Decahydro-11,11,12,12-tetramethoxy-1,4;5,8-dimethanoanthracene (5). Freshly distilled ammonia (300 mL) was placed in a 500-mL round-bottom flask containing a magnetic stirring bar. A solution of the bisadduct 1[2] (1.8 g, 3 mmol) in dry tetrahydrofuran (20 mL) and dry ethanol (2 mL) was carefully added. Sodium (1.38 g, 0.06 mol) was added to this magnetically stirred solution in small pieces over a period of 30 min, and the resultant mixture was stirred for an additional 1 h and quenched with solid ammonium chloride. Ammonia was evaporated over a period of 4 h, and the residue was taken up in 50 mL of water and extracted with methylene chloride (4 × 30 mL). The extract was washed with brine, dried over anhydrous Na_2SO_4, and concentrated. Purification of the residue by passing through a silica gel (20 g) column using methylene chloride as eluent furnished 800 mg (81%) of the dechlorinated bis(ketal) 5: mp 237-40 °C; IR ($CHCl_3$) ν_{max} 1125, 1100, 1060 cm^{-1}; ^{1}H NMR (60 MHz, CCl_4) δ 5.87 (4 H, t, J = 2.2 Hz), 3.1 (3 H, s), 3.0 (3 H, s), 2.54 (4 H, m), 1.8-2.35 (4 H, m), 1.22 (2 H, m), 0.4 (2 H, m). Anal. Calcd for $C_{20}H_{28}O_4$: C, 72.26; H, 8.49. Found: C, 72.35; H, 8.54.

1,4,4a,5,8,8a,9,9a,10,10a-Decahydro-1,4;5,8-dimethanoanthracene-11,12-dione (4). A solution of the bis(ketal) 5 (300 mg, 0.91 mmol) in methylene chloride (20 mL) was placed in a 100-mL round-bottom flask followed by the addition of 20% aqueous sulfuric acid (20 mL). The two layers were vigorously stirred for 16 h at room temperature. The organic layer was separated and the aqueous layer extracted with methylene chloride (2 × 10 mL). The combined organic layer was washed with saturated aqueous sodium bicarbonate solution followed by brine and dried over anhydrous Na_2SO_4. Evaporation of the solvent furnished 200 mg (92%) of crystalline dione 4, which was recrystallized from a mixture of benzene-ethanol: mp 178-180 °C; IR ($CHCl_3$) ν_{max} 1785, 1765 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 6.43 (4 H, t, J = 3 Hz), 2.85 (4 H, br s), 2.22-2.35 (4 H, m), 1.5 (2 H, td, J = 13, 4 Hz), 0.62 (2 H, q, J = 13 Hz). Anal. Calcd for $C_{16}H_{16}O_2$: C, 79.97; H, 6.71. Found: C, 79.71, H, 6.67.

Hexacyclo[6.6.1.0^{2,7}.0^{3,12}.0^{6,11}.0^{9,14}]pentadec-4-en-15-one (6). The dione 4 (200 mg, 0.85 mmol) was placed in a 25-mL round-bottom flask filled with nitrogen. The flask was immersed in an oil bath, preheated and equilibrated to 200 °C, for 5 min. The pyrolysate was cooled, taken in benzene, and purified by passing through a silica gel (10 g) column. Alternatively, the pyrolysis can also be carried out in toluene solution (5 mL) in a sealed tube at the same temperature for 10 min. The hexacyclic ketone 6 (140 mg, 79.5%) was further purified by a bulb-to-bulb distillation: mp 140-143 °C; IR (melt) ν_{max} 3060, 1770 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 6.38 (2 H, dd, J = 4.5, 3 Hz), 2.34 (2 H, br s), 2.28 (2 H, br s), 2.04 (2 H, d, J = 13.5 Hz), 1.89 (2 H, s), 1.74 (2 H, s), 1.63 (2 H, br s), 1.27 (2 H, d, J = 13.5 Hz); ^{13}C NMR (22.5 MHz, $CDCl_3$) δ 212.2 (s), 135.2 (d), 44.8 (d), 34.9 (d), 30.5 (d), 29.1 (d), 26.5 (d), 24.9 (t); mass spectrum, m/e 212, 184, 142, 130, 93, 77. Anal. Calcd for $C_{15}H_{16}O$: C, 84.87; H, 7.59. Found: C, 83.62; H, 7.65.

Pentacyclo[8.4.0.0^{3,8}.0^{4,14}.0^{7,11}]tetradeca-5,12-diene (7). A benzene solution (500 mL) of the dione 4 (500 mg, 2.12 mmol) was carefully purged with a slow stream of nitrogen for 15 min. The solution was then irradiated with a Hanovia 450-W medium-pressure mercury vapor lamp in a quartz immersion vessel. After being irradiated for 30 min, solvent was evaporated and placed on a silica gel (20 g) column. Elution with hexane furnished 180 mg (50%) of the crystalline diene 7: mp 240 °C (hexane); IR ($CHCl_3$) ν_{max} 1600 cm^{-1}; ^{1}H NMR (60 MHz, $CDCl_3$) δ 5.75 (4 H, dd, J = 5, 2.5 Hz), 2.7-2.4 (4 H, br s), 2.2 (4 H, br s), 1.8 (2 H, d, J = 11 Hz), 0.85 (2 H, d with st, J = 11 Hz); ^{13}C NMR (22.5 MHz, $CDCl_3$) δ 131.2 (d), 45.7 (d), 38.3 (d), 28.3 (t). Further elution of the column with benzene furnished 60 mg (15%) of the hexacyclic ketone 6, which was characterized by spectral comparison with the sample obtained in the previous experiment. Final elution of the column with chloroform furnished 140 mg of the starting dione 4.

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Registry No. 1, 110507-79-4; 4, 110419-02-8; 5, 110419-03-9; 6, 110419-04-0; 7, 110433-13-1.

Micelle-Based Nucleophile Selectivity: Alkoxymercuration in Aqueous Sodium Dodecyl Sulfate

Mordechai Livneh, James K. Sutter, and Chaim N. Sukenik*

Department of Chemistry, Case Western Reserve University, Cleveland, Ohio 44106

Received March 25, 1987

Olefin hydroxymercuration and alkoxymercuration have become important parts of standard synthetic methodology in the last 2 decades. Their success depends on the electrophilicity of mercuric ions toward olefins, on the efficiency of mercurinium ion trapping by water or alcohol, and on the clean manipulation of the resulting alkyl mercurial. While mercury-mediated hydration of olefins in mixed THF/water solvent works well for a wide range of simple olefins,[1] we have previously described[2] the benefits of conducting the mercuration of dienes in aqueous sodium dodecyl sulfate (SDS) solution. We now report the ability of SDS micelles to enhance ether production via olefin alkoxymercuration. In many respects, this process compares favorably with both alkoxymercuration in alcohol solvent[3] and with olefin mercuration in the presence of stoichiometric amounts of alcohol in inert solvent. Our SDS-mediated process also provides new insight into the potential use of aqueous micelles to control the selectivity of organic reactions. The chemistry that is the basis of this work is summarized in eq 1.

$$C_6H_{13}CH{=}CH_2 + Hg^{2+} \xrightarrow[2.\ NaBH_4]{1.\ H_2O/ROH} C_6H_{13}CH(OH)CH_3 + C_6H_{13}CH(OR)CH_3 \quad (1)$$

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Table I. Alkoxymercuration of 1-Octene

entry	ROH [concn, M]/medium	concn, M C_8H_{16}	concn, M $Hg(OAc)_2$	time, h	2-octyl-OH/2-octyl-OR
1	C_2H_5OH [0.03]/0.03 M SDS	0.003	0.003	0.1–0.3	98/2 (±1)
2	C_4H_9OH [0.03]/0.03 M SDS	0.003	0.003	0.1–0.3	96/4 (±1)
3	$C_6H_{13}OH$ [0.03]/0.03 M SDS	0.003	0.003	0.1–0.3	83/17 (±2)
4	$C_6H_{13}OH$ [0.03]/0.03 M SDS	0.003	0.003	24	81/19 (±2)
5	$C_8H_{17}OH$ [0.03]/0.03 M SDS	0.003	0.003	0.1–0.3	72/28 (±2)
6	$C_8H_{17}OH$ [0.03]/0.03 M SDS	0.008	0.008	0.1–0.3	69/31 (±2)
7	$C_8H_{17}OH$ [0.03]/0.03 M SDS	0.003	0.003	24	48/52 (±2)
8	$C_8H_{17}OH$ [0.03]/0.03 M SDS	0.008	0.008	21	53/47 (±2)
9	$C_8H_{17}OH$ [0.03]/0.03 M SDS	0.03	0.03	22	26/74 (±2)
10	$C_8H_{17}OH$ [0.015]/0.03 M SDS	0.003	0.003	24	81/29 (±2)
11	$C_8H_{17}OH$ [0.09]/0.03 M SDS	0.003	0.003	24	41/59 (±2)
12	$C_{10}H_{21}OH$ [0.03]/0.03 M SDS	0.003	0.003	0.1–0.3	56/44 (±2)
13	$C_{10}H_{21}OH$ [0.03]/0.03 M SDS	0.003	0.003	24	33/67 (±2)
14	$C_{10}H_{21}OH$ [0.03]/0.03 M SDS	0.03	0.03	24	40/60 (±2)
15	$C_8H_{17}OH$ [0.03]/50% aq THF	0.003	0.003	24	98/2 (±1)
16	$C_8H_{17}OH$ [0.3] + 0.3 M H_2O/THF	0.03	0.03	40	52/48 (±3)[95% C_8H_{16}]

The conventional approach[3] to ether production as per the above equation requires that the mercuration be conducted in alcohol solvent. This limits the scope of this reaction to cases where ROH is an inexpensive liquid alcohol that can be used as a reaction solvent. Extensive studies of such alkoxymercurations and their dependence on mercury counterion as well as olefin and alcohol structure have recently been reported.[4] We reasoned that the demonstrated micellar enhancement of olefin mercuration,[2c] along with the micelle's capacity to exclude water and promote intramolecular cyclic ether formation,[2b] might be combined with the micelle's ability to concentrate hydrophobic reagents. We could thus promote intermolecular alkoxymercuration despite the presence of a large excess of water in the reaction medium. To this end, we allowed the mercuration of 1-octene to proceed in SDS micelles with various added alcohols. The results obtained for added primary alcohols, along with two nonmicellar control reactions, are recorded in Table I. Since ether formation was not significant (<5%) when secondary or tertiary alcohols were employed, those results are not considered.

There are a number of trends that are clear in the above data. Entries 1, 2, 3, 5, 6, and 12 each represent two samples taken from mercuration mixtures and quenched with $NaBH_4$ after a 5–15 min reaction time. They each showed <5% unreacted olefin. This rapid mercuration, despite the very dilute reaction conditions relative to standard THF/H_2O mercurations,[3,4] was consistent with our previous experience.[2] These results also demonstrate the ability of an alcohol to compete with water in mercurinium ion trapping to an extent that is directly related to alcohol chain length: C_2, 2%; C_4, 4%; C_6, 17%; C_8, 28%; C_{10}, 44%. They presumably reflect enhanced local alcohol concentration in the micelle. Since olefin mercuration is a reversible process, we can further induce ether formation by equilibration. Again, the efficacy of this enhancement parallels alcohol chain length: C_6, 17% → 19%; C_8, 28% → 52%; C_{10}, 44% → 67%. This reflects the greater thermodynamic stability in the micelle of the more hydrophobic alkoxymercurinium versus the more hydrophilic hydroxymercurinium ion.

Entries 7–9 confirm the ability to achieve significant levels of ether product over a range of olefin and mercury concentrations. However, whereas a comparison of these entries might have suggested a trend toward higher levels of ether formation at higher olefin concentrations, we could not confirm such a trend with entries 13 and 14. While trends based on alcohol hydrophobicity are clear up to C_8, the interactions of the C_8 and C_{10} alcohols with the micelle are more complex.

The extent to which the alcohol-swollen micelle is able to enhance alcohol trapping is dramatically demonstrated by comparing the SDS results to those in THF/H_2O (entry 15) and to reaction in THF with only a stoichiometric amount of water present (entry 16). In the standard mercuration medium of 50/50 THF/water the concentration of water is 25–30 M; thus the low level of ether production under these conditions (2%) is to be expected. On this basis alone, any alkoxymercuration in a bulk aqueous medium should exhibit similarly low levels of ether formation. While the extent of enhanced ether production does vary with the concentration of alcohol present in the swollen micelle (compare entries 10, 7, and 11), the contrast to the very low level of ether production seen at comparable alcohol concentration in nonmicellar medium (entry 15) is striking. Though the inherent nucleophilicity of the water and the alcohol in such reactions is comparable (entry 16), it is the enhanced local concentration in the micelle that allows the alcohol to overcome the bulk concentration advantage enjoyed by the water in all the SDS experiments. Enhancing the ratio of alcohol to water by reducing the amount of water and increasing the amount of alcohol (entry 16) leads to an unacceptably slow rate of reaction.

The implications of these results are twofold. They are complimentary to the elegant demonstration of enhanced concentration of anionic nucleophiles at the surface of cationic micelles seen in aryl diazonium decomposition.[5] While the benefits of such enhanced concentration of a neutral reagent might have been anticipated, we are unaware of any other clear demonstration of such an effect. Secondly, where the alcohol in an olefin alkoxymercuration cannot be used as the reaction solvent, using a micelle-mediated aqueous system may be a viable preparative option.

Experimental Section

^1H NMR were obtained on a Varian XL-200 spectrometer and are reported as ppm (δ) downfield from internal TMS (J in hertz). Mass spectra were recorded on an AEI-MS 30 mass spectrometer. Analytical gas chromatography was done on a Perkin-Elmer 3920B with FID, and preparative GC was done on a Varian 90P chro-

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matograph. 1-Octene, $Hg(OAc)_2$, butanol, hexanol, octanol, and decanol were all used as received from Aldrich. Absolute ethanol was purchased from Aaper Alcohol and Chemical Co. THF was freshly distilled under nitrogen from sodium metal. Sodium dodecyl sulfate was purchased from Bio-Rad Laboratories and was recrystallized once from 95% ethanol. A plot of surface tension versus concentration for its solutions in water showed no hysteresis. Water was doubly distilled.

Alkoxymercuration in SDS. For experiments 1–5, 7, 12, and 13 (Table I), the following procedure was used. In a 50-mL round-bottom flask equipped with a magnetic stirrer were placed SDS (212 mg) and 25 mL of H_2O. 1-Octene (8.4 mg, 0.075 mmol) was added, and the solution was stirred to achieve homogeneity. Primary alcohol (0.75 mmol) was added, and this solution was again stirred to dissolve the alcohol. Some cloudiness persisted with the longer chain alcohols, but no phase separation was observed. $Hg(OAc)_2$ (23.9 mg, 0.075 mmol) in 1 mL of H_2O was added. Aliquots (5–10 mL) were removed from these reactions at the indicated times and were quenched with 2 mL of a 3.0 N NaOH solution followed by 2 mL of 0.5 N $NaBH_4$ in 3.0 N NaOH. The black suspension so obtained was stirred for an additional 30 min to allow the Hg^0 to coagulate. This solution was poured into a 50-mL screw-capped centrifuge tube that contained approximately 2 g of NaCl and 0.25 g of $BaCl_2$. The tube was capped and shaken; a light gray flocculent formed. Diethyl ether was added, and three extractions were done. Each extraction required that the tube be centrifuged for 3 min to cleanly separate the three phases: ether extract on top, compressed gray solid, and H_2O layer on bottom. The ether fraction was removed, and the procedure was repeated. The ether extracts were dried with $MgSO_4$ and concentrated. A similar procedure was used for experiments 6, 8, 9, and 14 with the indicated increases in olefin and Hg^{2+} concentrations. Experiments 10 and 11 were also similar, but the alcohol concentration was changed. GLC analysis (10% OV-17, 15 ft × $^1/_8$ in. on 100/120 Chrom W) at 60–230 °C gave cleanly resolved peaks for unreacted 1-octene, 2-octanol, and the alkyl ether. Relative FID response factors for 2-octanol and the alkyl ether products were determined for authentic mixtures. Assigning an arbitrary value of 1 to the 2-octanol, the relative response was 1.2 for the C_2 and C_4 ethers and 1.8 for the C_6, C_8, and C_{10} ethers. Each aliquot was analyzed three times and integrated by cutting and weighing three copies of each chromatogram.

Alkoxymercuration in THF. Experiment 16: A dry round-bottom flask equipped with a magnetic stirring bar, was charged with 25 mL of dry THF, 1-octene (84 mg), water (135 mg), and 1-octanol (1.185 g). To this solution was added solid $Hg(OAc)_2$ (239 mg). The flask was stoppered, and the resulting clear yellow solution was allowed to stir at room temperature for 40 h. The yellow color persisted throughout. Workup with 3 N NaOH and $NaBH_4$ solutions was followed by ether extraction. The combined ether extracts were dried with $MgSO_4$ and concentrated. Analyses by GC (OV-17 as above) showed >95% unreacted 1-octene. The ratio of 2-octanol to *n*-octyl 2-octyl ether for the small amount that did react was determined. Experiment 15 was identical with the SDS-mediated mercuration (experiment 7), except that the aqueous SDS solution was replaced with a 50/50 mixture (by volume) of water and THF.

***n*-Alkyl 2-Octyl Ethers.** Authentic samples of the ethers were prepared by the method of Brown,[3] where the alcohol is used as the reaction solvent. They were purified by preparative gas chromatography on an OV-17 column (9 ft × $^1/_8$ in., 60/80 mesh Chrom W) at temperatures between 150 and 230 °C with a flow rate of 25 mL/min. While the C_2 ether was a known compound,[6] the other four ethers are new materials. Their ^1H NMR ($CDCl_3$) and mass spectra (exact mass at 15–20 eV) are reported below.

2-Oxyethyl octane: ^1H NMR δ 3.45 (3 H, m), 1.25 (10 H, m), 1.16 (3 H, t, J = 7.0 Hz), 1.10 (3 H, d, J = 6.2 Hz), 0.86 (3 H, t, J = 6.1 Hz). 2-Oxybutyl octane: exact mass calcd 186.1985, found 186.1984; ^1H NMR δ 3.42 (3 H, m), 1.41 (14 H, m), 1.15 (3 H, d, J = 6.2 Hz), 0.92 (6 H, t, J = 6.1 Hz). 2-Oxyhexyl octane: exact mass calcd 214.2298, found 214.2301; ^1H NMR δ 3.33 (3 H, m), 1.40 (18 H, m), 1.08 (3 H, d, J = 6.2 Hz), 0.85 (6 H, t, J = 6.1 Hz). 2-Oxyoctyl octane: exact mass calcd 242.2611, found 242.2634; ^1H NMR δ 3.44 (3 H, m), 1.51 (22 H, m), 1.08 (3 H, d, J = 6.0 Hz), 0.85 (6 H, t, J = 6.4 Hz). 2-Oxydecyl octane: exact mass calcd 270.2923, found 270.2944; ^1H NMR δ 3.41 (3 H, m), 1.56 (26 H, m), 1.09 (3 H, d, J = 6.0 Hz), 0.86 (6 H, t, J = 5.9 Hz).

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Registry No. SDS, 151-21-3; $Hg(OAc)_2$, 1600-27-7; $C_6H_{13}CH(OC_2H_5)CH_3$, 63028-01-3; $C_6H_{13}CH(OC_4H_9)CH_3$, 110458-41-8; $C_6H_{13}CH(OC_6H_{13})CH_3$, 51182-98-0; $C_6H_{13}CH(OC_8H_{17})CH_3$, 20012-47-9; $C_6H_{13}CH(OC_{10}H_{21})CH_3$, 51183-01-8; 1-octene, 111-66-0; 2-octyl alcohol, 123-96-6.

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An Azetidinone Route to 2,3-Dideoxy-3-aminopentoses and 2,3-Dideoxy-3-*C*-methyl-3-aminopentoses

Frank M. Hauser,*[1] Suzanne R. Ellenberger, and Richard P. Rhee

Department of Chemical, Biological and Environmental Sciences, Oregon Graduate Center, Beaverton, Oregon 97006

Received April 1, 1987

We have shown previously that the azetidinone **1**, formed through regiospecific cycloaddition of chlorosulfonyl isocyanate and (*E*)-1,3-pentadiene, can be used as an intermediate for efficient, diastereoselective total syntheses of both racemic and optically active daunosamine (**2**)[2] (Figure 1). It was recognized, as indicated in Scheme I, that a parallel reaction sequence from the azetidinone adducts **3a** and **3b** would provide a brief route for preparation of the aminopentoses **7b** and **8b** and the *C*-methylaminopentoses **7d** and **8d**, respectively. However, the value of this approach as a preparative method was somewhat mitigated by the modest yields of azetidinones **3a** and **3b** that were attainable from the cycloaddition of butadiene or isoprene with chlorosulfonyl isocyanate.[3]

Recently, we discovered a procedure for high-yield (>90%) preparation of the needed azetidinones **3a** and **3b**[4] and now have established the generality of the preparative sequence to daunosamine (**2**) as a route to the aminopentoses **7** and **8**. An ancillary finding of this investigation is that the diastereoselectivity of the osmium tetraoxide hydroxylation of amides of allylamines is enhanced by an increase in alkyl substitution α to the amide nitrogen.

The azetidinone starting materials **3a** and **3b** were prepared in 95% yield through respective cycloaddition of butadiene and isoprene with chlorosulfonyl isocyanate.[4] Methanolysis (MeOH/HCl) of **3a** and **3b** followed by benzoylation furnished the corresponding methyl *N*-benzoyl-4-pentenoates **4a** and **4b** in 95% overall yield.

Osmium tetraoxide catalyzed cis hydroxylation of the pentenoate **4a**, with trimethylamino *N*-oxide (TMNO) to regenerate the tetraoxide, gave a 56:44 ratio of trans and cis lactones **5a** and **6a** in 96% yield. The isomers were separated initially by chromatography and then further purified by recrystallization. Subsequently, it was found that the more polar isomer **5a** could be isolated directly

(1) Present address: Department of Chemistry, State University of New York at Albany, Albany, NY 12222.
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