1020, 1000, 900, 850 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.90 (t, 3 H, CH_3CH_2), 1.25 (m, 6 H, CH_2), 2.10 (s, 3 H, CH_3CO), 2.40 (t, 2 H, CH_2CO), 7.25 (m, 5 H, Ph). The aliphatic region of the ^{13}C NMR spectrum was identical with that of authentic 2-heptanone.[9] Evaporation of chloroform followed by distillation (Kugelrohr) afforded 0.64 g (56%) of 2-heptanone having IR and ^1H NMR spectra which were identical with those of an authentic sample.

Registry No. 1, 100-57-2; $CH_3(CH_2)_4C{\equiv}CH$, 628-71-7; $CH_3(CH_2)_3C{\equiv}CH$, 693-02-7; $CH_3(CH_2)_7C{\equiv}CH$, 764-93-2; $CH_3(CH_2)_3C{\equiv}C(CH_2)_3CH_3$, 1942-46-7; $PhC{\equiv}CH$, 536-74-3; $C_2H_5OC(O)C{\equiv}CH$, 623-47-2; $CH_3(CH_2)_4C(O)CH_3$, 110-43-0; $CH_3(CH_2)_3C(O)CH_3$, 591-78-6; $CH_3(CH_2)_7C(O)CH_3$, 693-54-9; deuterium, 7782-39-0; cyclohexylacetylene, 931-48-6; (1-hydroxycyclohexyl)acetylene, 78-27-3; 1-ethynylcyclopentanol, 17356-19-3; *p*-ethynylanisole, 768-60-5; acetylcyclohexane, 823-76-7; 1-acetylcyclohexanol, 1123-27-9; 1-acetylcyclopentanol, 17160-89-3; 1-heptynylphenylmercury,, 82080-25-9.

(9) Treatment of such chloroform solutions with aqueous hydrochloric acid (1.5 equiv) for 1 h at 50 °C led to a 79% isolated yield of phenylmercuric chloride (presumably from reaction with regenerated 1). Efforts at developing a catalytic system based on 1 failed, apparently due to the inability of 1 to react further with alkynes after being in contact with an aqueous phase. In a control experiment, 1 showed no reactivity toward 1-heptyne present in a water–chloroform mixture.

Efficient Regiocontrolled Synthesis of Sarkomycin and Homosarkomycin

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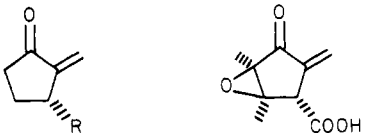
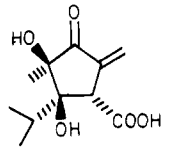
Received January 25, 1982

Sarkomycin (1), parent member of the cyclopentanoid class of antibiotic–antitumor agents which now includes methylenomycin A (3),[2] xanthocidin (4),[3] and pentenomycin I (5),[4] has attracted considerable attention as a synthetic target since its initial isolation in 1953[5] and structural elucidation in 1955.[6,7] By and large, however, published routes to this antitumor agent have been nonregiocontrolled.[8] Indeed, only the very recent synthesis by Marx and Minaskanian (1979),[9] the elegant chiral approach of Boeckman and collaborators (1980),[10] and the palladium-catalyzed cyclization route of Tsuji and Kobayashi (1981),[11] are in fact regiocontrolled. Each of these sequences, however, has the disadvantage of length (ca. 9 or 10 steps).



1, R = COOH
2, R = CH_2COOH

3



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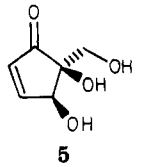

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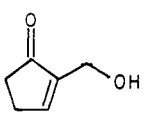
In connection wiith our continuing interest in the cyclopentanoid class of antibiotics, we are pleased to record here the synthesis of both sarkomycin (1) and its congener homosarkomycin (2).[12] Both synthetic sequences are short, efficient, and regiocontrolled; furthermore, both take advantage of the ready availability of α-(hydroxymethyl)cyclopentenone (6) or its immediate precursor ketal



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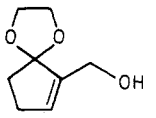

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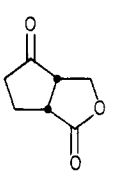

8

7, prepared by application of the α-oxovinyl anion methodology developed recently in our[13] and other laboratories.[14] Finally we note that our approach to sarkomycin takes advantage of the Marx–Minaskanian[9] acid-catalyzed retrolactonization of lactone 8 (i.e., cyclosarkomycin).

Sarkomycin. With cyclosarkomycin (8) as our initial target, conjugate addition of lithium divinylcuprate[15] to α-(hydroxymethyl)cyclopentenone (6) at −78 °C afforded, after the usual workup (saturated aqueous NH_4Cl), unsaturated keto alcohol 9 in 73% yield as an epimeric mixture. Ozonolysis of the latter (−78 °C, CH_2Cl_2), fol-

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(12) While the trivial name, homosarkomycin, has been employed previously by Jambowski (see *Tetrahedron Lett.* **1971**, 1733) to describe the six-membered ring congener of sarkomycin, we believe that this name more suitably describes 2.

(13) Guaciaro, M. A.; Wovkulich, P. M.; Smith, A. B., III *Tetrahedron Lett.* **1978**, 4661. Also see ref 4a.

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(15) House, H. O.; Chu, C.; Wilkins, J. M.; Umen, M. J. *J. Org. Chem.* **1975**, *40*, 1460.

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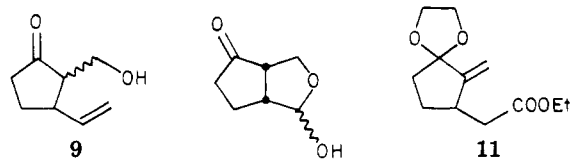

9 10 11

lowed by reduction of the derived ozonide with dimethyl sulfide and subsequent in situ treatment with DBU afforded the highly unstable lactol **10** again as a mixture of epimers. No evidence for the corresponding open hydroxy aldehyde was found. This mixture of lactols was then effected with pyridinium chlorochromate[16] to give cyclosarkomycin (**8**) as a crystalline solid (mp 45–46 °C). It is important to note that although the above mixture of lactols **10** could be isolated and characterized spectroscopically, it was found most convenient to effect the conversion of **10** directly to cyclosarkomycin (**8**) in "one pot", the overall yield being 76% for the three steps. Cyclosarkomycin (**8**) prepared in this manner was identical in all respects with that of Marx and Minaskanian.[9] Final conversion of cyclosarkomycin (**8**) to sarkomycin (**1**) was then achieved as reported.[9]

Homosarkomycin. Preparation of homosarkomycin (**2**), on the other hand, took direct advantage of the ortho-Claisen rearrangement developed by Johnson and colleagues[17] as applied to the ethylene ketal of α-(hydroxymethyl)cyclopentenone (**7**).[11] To this end, reaction of **7** with excess ethyl orthoacetate containing a catalytic amount of propanoic acid at 140 °C for 2 h afforded ethyl ester **11** as an oil in 71% yield. Consistent with the assigned structure, the high-field NMR spectrum displayed, in addition to the ethyl group resonances, doublets at δ 5.00 and 5.27 (J = 2 Hz, 1 H each) characteristic of the exomethylene protons. Subsequent basic hydrolysis of the ester functionality (5% NaOH, EtOH) followed by an acidic work-up (10% aq HCl) afforded homosarkomycin (**2**) in 89% yield. That indeed homosarkomycin was in hand was apparent from the IR, high field NMR and elemental composition data (see experimental section).

Experimental Section

Materials and Equipment. Melting points were obtained on a Thomas-Hoover apparatus and are corrected; boiling points are uncorrected. Infrared spectra (IR) were recorded on a Perkin-Elmer Model 337 spectrophotometer while proton NMR spectra were obtained on a Varian Model A60 or T60A (60 MHz) or a Bruker WH 360 (360 MHz) or WP 250 (250 MHz) spectrometer. Chemical shifts are reported as δ values in parts per million relative to Me_4Si (δ 0.00). High-resolution mass spectra were obtained at the University of Pennsylvania Mass Spectrometry Service Center on a Hitachi Perkin-Elmer RMH-2 or VG 70/20 Micromass high-resolution spectrometer interfaced to a Kratos DS-20-s data system.

Commercial silica gel plates (250 μm) with a fluorescent indicator (E. M. Merck) were used for analytical thin-layer chromatography (TLC). Preparative separations were performed on precoated 1000-μm silica gel GF (Analtech) plates. Silica gel with a particle size of 0.04–0.063 mm (supplied by E. Merck) was used for flash chromatography.[18]

Preparation of Vinyllithium. According to the procedure of Seyferth and Weiner,[19] a benzene solution of phenyllithium (583 mg, 6.94 mmol) was added dropwise to a stirring solution of triphenylvinyltin (2.6 g, 6.94 mmol) in 13.5 mL of anhydrous Et_2O at room temperature. After the mixture was stirred for 30 min, the solid tetraphenyltin was removed by filtering the slurry through a Shlenk tube to afford a clear colorless ethereal benzene solution of vinyllithium.

3-Vinyl-2-(hydroxymethyl)cyclopentanone (9). A solution of Me_2S·CuBr (714 mg, 3.47 mmol), 3.5 mL of Et_2O and 2.5 mL of Me_2S was cooled to −78 °C. As the solution cooled, a white crystalline complex precipitated. Vinyllithium (6.94 mmol), generated as described above, was then added via a double-tipped needle over a 10-min period. Stirring was continued for an additional 10 min. To the resulting dark solution was added α-(hydroxymethyl)cyclopentenone (185 mg, 1.65 mmol), dropwise in 2 mL of THF. The reaction mixture was stirred for 30 min at −78 °C and then quenched with saturated NH_4Cl. The organic phase was separated and dried over $MgSO_4$. Concentration in vacuo afforded a yellow oil which upon purification via flash chromatography (50% Et_2O/50% hexane) yielded 170 mg (73%) of **9**: IR (CCl_4) 3450 (br), 3000–2875 (s), 1700 (s) cm^{-1}; NMR ($CDCl_3$, 360 MHz) δ 1.60–1.78 (m, 1 H), 2.0–2.20 (m, 3 H), 2.28–2.50 (m, 2 H), 2.58–2.74 (m, 1 H) 3.70 (dd, J = 6, 11.6 Hz, 1 H), 3.88 (dd, J = 4, 11.6 Hz, 1 H), 5.08 (d, J = 8.4 Hz, 1 H), 5.15 (d, J = 13.9 Hz, 1 H), 5.82 (ddd, J = 6, 8.4, 13.9 Hz, 1 H); mass spectrum, m/e 140.0842 (m^+; calcd for $C_8H_{12}O_2$, 140.0837).

Anal. Calcd for $C_8H_{12}O_2$: C, 68.54; H, 8.63. Found: C, 68.73; H, 8.49.

Cyclosarkomycin (8). To a preformed solution of alcohol **9** (100 mg, 0.78 mmol) in 5 mL of CH_2Cl_2 held at −78 °C was bubbled ozone until a light blue-gray color persisted. The ozonolysis was discontinued, and the reaction mixture flushed with O_2 until the blue-gray color discharged. The resultant ozonide was then reduced with Me_2S at −78 °C and the reaction mixture allowed to warm to room temperature, whereupon 100 μL of DBU was added and the resultant mixture stirred at room temperature for 2 h.

In a separate run, isolation consisting of a $NaHSO_3$ wash, drying with $MgSO_4$, and concentration in vacuo afforded a mixture of lactols **10**: IR ($CHCl_3$) 3600 (w) 3500–3200 (br), 3050–2825 (m), 1750 (s) cm^{-1}; NMR ($CDCl_3$, 250 MHz) δ 1.70–1.84 (m, 1 H), 2.14–2.40 (m, 3 H), 2.84–3.06 (m, 3 H), 4.10 (dd, J = 4, 10 Hz, 1 H), 4.22 (dd, J = 8, 10 Hz, 1 H), 5.40–5.54 (m, 1 H).

The above CH_2Cl_2 solution was then evaporated via a stream of argon, 10 mL of CH_2Cl_2 added, and the resultant mixture treated at room temperature with one portion of pyridinium chlorochromate (335 mg, 1.6 mmol). After a 2-h period the reaction mixture was diluted with ether, and the resultant chromium salts were removed via filtration with the aid of Celite. Removal of the solvent in vacuo and preparative TLC (ether) afforded 83 mg of cyclosarkomycin (**8**) as a crystalline solid (mp 45–47 °C), identical in all respects with that prepared by Mark and Minaskanian:[9] IR (CCl_4) 3000–2850 (m), 1775 (s), 1745 (s) cm^{-1}; NMR ($CDCl_3$, 250 MHz) δ 2.15–2.58 (m, 4 H), 3.02 (dd, J = 7.5, 9 Hz, 1 H) 3.40 (dd, J = 7.5, 9 Hz, 1 H), 4.42 (m, 2 H).

Ethyl Ester 11. To a solution of ketal **7** (156 mg, 1 mmol) dissolved in triethyl orthoacetate (1.134 g, 7 equiv) were added 4 drops of propionic acid. The flask was then fitted with a short-path distillation apparatus and heated at 140 °C for 2 h, distilling off the EtOH as formed. The reaction mixture, upon cooling, was then washed with saturated aqueous $NaHCO_3$ and extracted with three 25-mL portions of Et_2O. The combined organic material was washed with brine and dried over anhydrous $MgSO_4$. Removal of the solvent in vacuo followed by fractional distillation [90 °C (1 mmHg)] afforded 160.2 mg (71% yield) of ester **11**: IR 3080 (w), 2975 (s), 2950 (s), 1725 (s), 905 (s) cm^{-1}; NMR (360 MHz) δ 1.07–1.51, 1.25 (m, t, J = 7 Hz, 5 H), 1.67–2.11 (m, 2 H), 2.24–2.40 (m, 1 H), 2.52–2.65 (dd, J = 15 Hz, J' = 6 Hz, 1 H), 2.90–3.07 (m, 1 H), 3.79–4.19, 4.14 (m, q, J = 7 Hz, 6 H), 5.00 (d, J = 2 Hz, 1 H), 5.27 (d, J = 2 Hz, 1 H).

Anal. Calcd for $C_{12}H_{18}O_4$: C, 63.70; H, 8.02. Found: C, 63.60; H, 7.95.

Homosarkomycin (2). Ester **11** (155 mg, 0.69 mmol) was dissolved in 2 mL each of 5% aqueous NaOH and 95% EtOH and stirred overnight at room temperature under N_2. The reaction mixture was then poured into 20 mL of H_2O and extracted with Et_2O. After separation, the aqueous layer was acidified with 10% aqueous HCl and then extracted with Et_2O, and the latter then was washed with brine and dried. Removal of the solvent in vacuo afforded 103.1 mg of an oil which after TLC purification (1:1 ether/methylene chloride, R_f 0.1) gave 87.2 mg (89% yield) of

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homosarkomycin (2): IR (CCl_4) 3540–2400 (br s), 1710 (br s), 1640 (s), 910 (s) cm^{-1}; NMR (60 mHz) δ 1.27–1.97 (m, 1 H), 2.02–2.68 (m, 5 H), 2.82–3.48 (m, 1 H), 5.29 (d, J = 2.5 Hz, 1 H), 6.07 (d, J = 2.5 Hz, 1 H), 10.37 (br s, 1 H: mass spectrum, m/e 154.0628 (M^+; calcd for $C_8H_{10}O_3$, 154.063).

Acknowledgment. It is a pleasure to acknowledge the support of this investigation by the National Institutes of Health (National Cancer Institute) through Grant No. CA-19033. In addition, we thank Mr. S. T. Bella of the Rockefeller University for the microanalysis and the Middle Atlantic Regional NMR Facility (NIH Grant No. RR542) at the University of Pennsylvania where the 220- and 360-MHz NMR spectra were recorded.

Registry No. 2, 82134-78-9; 6, 68882-71-3; 7, 80963-19-5; 8, 82093-34-3; *cis*-9, 82134-76-7; *trans*-9, 82134-79-0; 10 (isomer 1), 82134-77-8; 10 (isomer 2), 82188-46-3; 11, 82149-57-3; phenyllithium, 591-51-5; triphenylvinyltin, 2117-48-8; vinyllithium, 917-57-7; triethyl orthoacetate, 78-39-7.

Synthesis of Benzo[*a*]fluoranthene and Naphtho[2,1-*a*]fluoranthene

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Received January 29, 1982

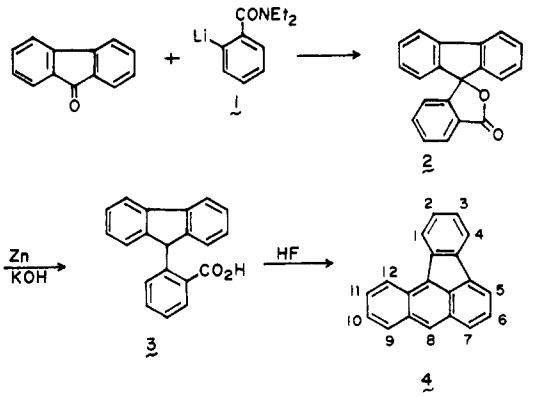
A novel synthetic approach to polycyclic hydrocarbons involving in the key step condensation of *o*-lithioaryl amides with aryl ketones has recently been described.[1-3] The carcinogenic hydrocarbons 3-methylcholanthrene, benz[*a*]anthracene, dibenz[*a*,*h*]anthracene, dibenz[*a*,*j*]anthracene, benzo[*a*]pyrene, and their methyl derivatives have been synthesized via this method in good overall yields.

We now report convenient syntheses of the nonalternant polycyclic hydrocarbons benzo[*a*]fluoranthene and naptho[2,1-*a*]fluoranthene[4] based on the same general method and involving a novel reductive cyclization.

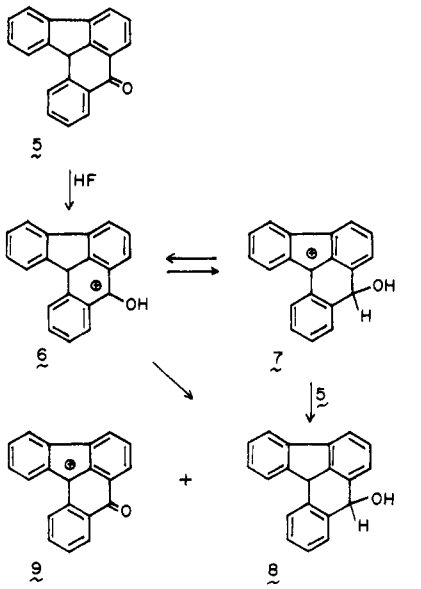
Synthesis of benzo[*a*]fluoranthene (4) is outlined in Scheme I. 2-Lithio-*N*,*N*-diethylbenzamide (1) was generated in situ by directed metalation of *N*,*N*-diethylbenzamide with *sec*-butyllithium by the method of Beak.[5] Reaction of 1 with 9-fluorenone afforded smoothly the lactone 2. Reduction of this intermediate with zinc and alkali yielded the free acid 3. Attempted cyclization of 3 with $ZnCl_2$ in acetic acid–acetic anhydride, the reagent previously employed in analogous cyclizations,[1-3] gave no reaction. However, 3 underwent reductive cyclization in liquid HF to yield benzo[*a*]fluoranthene directly in a single step! The keto intermediate 5, anticipated as the conventional product of this reaction, could not be detected among the residual products.

The mechanism of this reductive cyclization is unknown. The simplest hypothesis (Scheme II) is that the carbonium ion intermediate 6, formed by protonation of 5 in the acidic

Scheme I

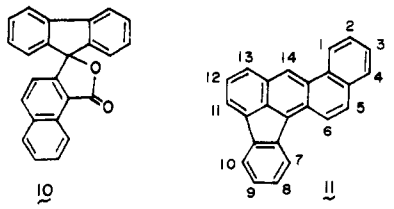


Scheme II



medium, is in equilibrium with the highly stabilized ionic intermediate 7. Hydride transfer from 5 to 6 or 7 furnishes the alcohol 8 which dehydrates to 4 along with simultaneous formation of a new ionic intermediate 9. The latter apparently yields only tarry products which could not be characterized. Since this mechanism implies that addition of an appropriate hydride donor to the medium might enhance the yield,[6] a similar experiment was conducted in the presence of triphenylmethane. Under these conditions the yield of benzo[*a*]fluoranthene virtually doubled from 36% to 71%!

Synthesis of naphtho[2,1-*a*]fluoranthene (11) was accomplished via an analogous sequence. Reaction of 2-lithio-*N*,*N*-diethyl-1-naphthamide with 9-fluorenone furnished the lactone 10 which underwent reduction with zinc



and alkali followed by reductive cyclization in liquid HF to afford 11. As in the preceding example, reductive cyclization in the presence of triphenylmethane doubled the

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(3) Harvey, R. G.; Cortez, C.; Jacobs, S. A. *J. Org. Chem.*, **1982**, *47*, 2120.
(4) According to current IUPAC and CA nomenclature rules, these compounds are designated benz[*a*]aceanthrylene and dibenz[*a*,*j*]aceanthrylene, respectively. The fluoranthene-based names are acceptable alternatives which are employed herein to emphasize the structural relationship to other fluoranthene derivatives of interest in carcinogenesis research.
(5) Beak, P.; Brown, R. A. *J. Org. Chem.* **1977**, *42*, 1823.
(6) We are grateful to a referee for suggesting the use of a hydride donor to increase the yield.

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