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Sterically Crowded Ring Systems. Preparation and X-ray Crystal Structure of 9,10,11,11,13,13-Hexamethyl-12-oxo-9,10-propanoanthracene[1]

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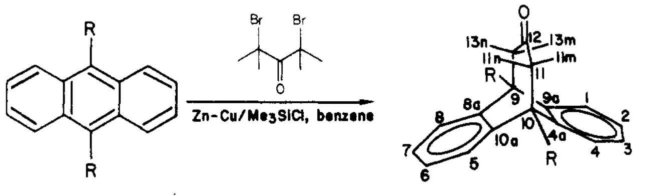
9,10,11,11,13,13-Hexamethyl-12-oxo-9,10-propanoanthracene (**2a**) and 9,10-dimethoxy-11,11,13,13-tetramethyl-12-oxo-9,10-propanoanthracene (**2b**) have been obtained by reductive intermolecular cyclodehalogenation of 2,4-dibromo-2,4-dimethyl-3-pentanone with zinc–copper couple/chlorotrimethylsilane in the presence of the requisite 9,10-disubstituted anthracenes (**1a,b**). The cycloadducts were characterized spectroscopically (^{1}H NMR, ^{13}C NMR, IR, mass spectra), the IR carbonyl band of **2a** appearing at 1677 cm^{-1} in KBr and at 1681 cm^{-1} in CCl$_4$. Compound **2a** was also characterized by single-crystal structure analysis. The crystals are monoclinic, space group $P2_1$, with a = 9.737 (2) Å, b = 9.651 (2) Å, c = 9.741 (2) Å, β = 103.06 (2)°, and Z = 2. The structure was solved by direct methods and refined to a final R of 0.043 (R_w = 0.048) for 1306 out of 1554 measured data satisfying the condition $I > 1.5\sigma(I)$. This X-ray analysis of **2a** shows the clash of the six methyl groups which forces bond angle deformations, nonbonded repulsions, and eclipsing strains. The molecule adopts an approximate C_{2v} structure, with the five carbon atoms C(10), C(11), C(12), C(13), and C(9) defining the bridge, being almost coplanar, and having an internal carbonyl angle of 130°. The preparations of **2a** and **2b** are the first examples for 4 + 3 → 7 cyclizations in which two diquaternary carbon–carbon bonds are made. The principle of constructing crowded carbon–carbon bonds by combining nucleophilic but weakly basic π systems and allyl cations or similar S$_N$1-like alkylating agents seems general.

Scheme I. Preparation of Crowded 9,10-Dihydro-9,10-propanoanthracenes 2a,b



1a, R = Me
b, R = OMe
c, R = H

2a, R = Me (40% yield)
b, R = OMe (30% yield)
c, R = H (71%[4] yield)

Scheme II. Proposed Route to Key Electrophilic Intermediate 9

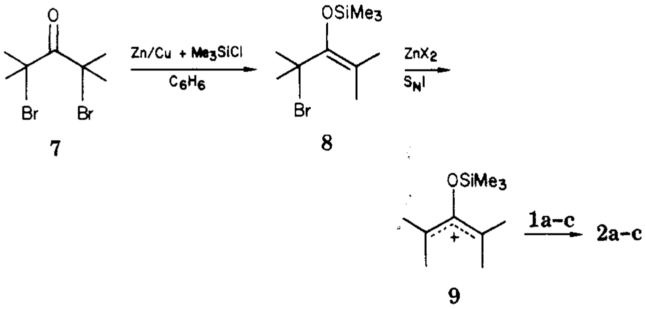
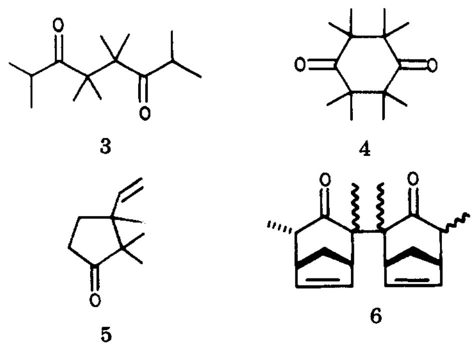


Chart I. Diquaternary Carbon–Carbon Bonds via S$_N$1-like Alkylations of Covalent Enolates



The synthesis, structure, and properties of crowded molecules continue to attract general interest.[3] We have prepared the title compound **2a** and 9,10-dimethoxy-11,11,13,13-tetramethyl-12-oxo-9,10-propanoanthracene (**2b**) in good yields by the simple, one-pot reductive intermolecular cyclodehalogenation of 2,4-dibromo-2,4-dimethyl-3-pentanone with zinc–copper couple/chlorotrimethylsilane in the presence of 9,10-dimethylanthracene or 9,10-dimethoxyanthracene (Scheme I).[4] Compounds **2a** and **2b** have two diquaternary carbon–carbon bonds, the formation of which is not trivial. Previous examples from our own work for making diquaternary carbon–carbon bonds include the preparation of 2,4,4,5,5,7-hexamethyloctane-3,6-dione (**3**),[5] octamethyl-1,4-cyclohexanedione (**4**),[6] 2,2,3-trimethyl-3-vinylcyclopentanone (**5**),[7] and the tetracyclic diketone **6**[8] (Chart I). The underlying principle of these preparations has been to combine a relatively nonbasic, covalent enolate and a carbon cation or quasication in an S$_N$1-like reaction. In the present reaction the silicon enolate **8**, rather than a zinc enolate which was used previously,[5-8] is visualized as an intermediate which ionizes in a zinc halide assisted heterolysis to form the key allyl cation **9**. In turn, **9**, which is both electrophilic and nucleophilic and has two fully alkylated carbon termini, adds to the disubstituted anthracene to give the products **2a** and **2b**, respectively. By use of *preformed* silicon enolates [rather than silicon enolates formed in situ (Scheme II)] and S$_N$1-like alkylating agents, crowded carbon-carbon bonds have also been

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[8] Hoffmann, H. M. R.; Rawson, D. I., unpublished experiments. Rawson, D. I. Ph.D. Thesis, University of London, 1976. Compound **6** was prepared in analogy to the method for **3**,[5] the solvent being 1,2-dimethoxyethane–methanol (4:1).

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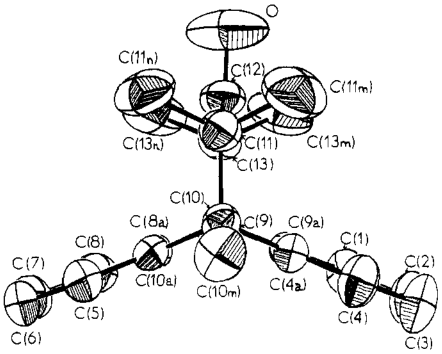
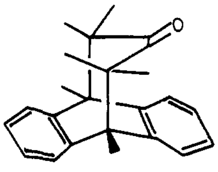


Figure 1. Single molecule of **2a** showing the atom-numbering scheme used.

made recently by Chan,[9] Reetz,[10] and Paterson and Fleming,[11] as well as by Sakurai et al.[12]



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The structure of **2a** was investigated next. What is the shape of the **2a** ring system and how does the molecule accommodate the conflicting energetic demands of nonbonded repulsions, eclipsing strains, and bond angle deformations? Dreiding models would suggest a strongly bent bridge as in **10**. However, the molecule adopts an approximate C_{2v} structure with the five carbon atoms C(10), C(11), C(12), C(13), and C(9), defining the bridge, almost coplanar, as can be seen from Figure 1, an edgewise view along the line of intersection of the planes of the two benzenoid rings. Results of some least-squares-plane calculations are given in Table I and relevant dihedral angles in Table II. The deviations from exact C_{2v} symmetry can be detected in various ways. First of all, the bridge system C(9)–C(13)–C(12)–O–C(11)–C(10) shows small but significant deviations from planarity with individual atom displacements of up to 0.05 Å (Table I). Similarly, the staggering of the carbonyl and bridgehead methyl bonds with the *gem*-dimethyls is not perfect, as can be seen from the dihedral angles (Table II) C(9m)–C(9)–C(13)–C(13m), C(13n) and O–C(12)–C(13)–C(13m), C(13n), etc., which range from 52.4 to 62.5°. The bridgehead methyl···*gem*-dimethyl and oxygen···*gem*-dimethyl nonbonded contacts, which range from 2.99 to 3.06 Å and from 2.72 to 2.81 Å, respectively (Table III), are very short, and the molecule suffers considerable strain. Examination of bond angles and bond lengths involving atoms other than hydrogen (Table IV) reveals interesting details of the effect of steric crowding on the geometry, especially on the bridge system. As a consequence of the flattening of the bridge, the internal angles C(9)–C(13)–C(12) and C(10)–C(11)–C(12) have been widened from normal tetrahedral values to 116°; more significantly, the internal angle at the carbonyl carbon, C(11)–C(12)–C(13), has expanded from a normal 120° to 130°. The effect of nonbonded repulsions, i.e., Me···Me and O···Me compressions, on bond lengths seems to be small, but the C(sp^3)–C(sp^2) bonds [C(13)–C(12) and C(11)–C(12)] and the C(sp^3)–C(sp^3) bonds [C(9)–C(13) and C(10)–C(11)] are 0.02 Å longer than might perhaps be expected. The difference between the sizes of the oxygen atom and methyl groups is reflected in the angles at C(11) and C(13) involving the *gem*-dimethyl groups. Thus, the four angles on the side of the oxygen atom [C(12)–C(11)–C(11m), C(12)–C(11)–C(11n), C(12)–C(13)–C(13m), and C(12)–C(13)–C(13n)], averaging 104.9 (5)°, are smaller than the four angles on the side of the bridgehead methyl groups [C(9)–C(13)–C(13m), C(9)–C(13)–C(13n), C(10)–C(11)–C(11m), and C(10)–C(11)–C(11n)], with an average of 110.8 (4)°. In view of the close contacts on both sides of the three-carbon bridge one might have expected the two geminal Me–C–Me angles to open up to relieve compressions, especially so since the transannular distances C(11m)···C(13m) and C(11n)···C(13n) are >4.0 Å. That this does not happen and that, in fact, the angles are actually contracted must be the result of "hybridization compensation" brought about by the wide internal angles at C(11) and C(13).

Table I. Least-Squares Planes Data[a,b]

plane 1	C(11), C(12), C(13) $9.442x - 1.201y - 4.130z = -1.606$ {C(9) [−0.172], C(10) [−0.153], O [0.032] }
plane 2	C(9) [−0.005], C(10) [0.005], C(11) [−0.005], C(13) [0.005] $9.156x - 1.912y - 4.694z = -2.357$ {O [−0.159], C(9m) [0.008], C(10m) [0.006], C(11m) [1.261], C(11n) [−1.247], C(12) [−0.067], C(13m) [1.275], C(13n) [−1.209] }
plane 3	C(8a) [0.004], C(9) [−0.002], C(10) [0.002], C(10a) [−0.004] $-0.536x + 6.818y + 6.816z = 6.156$ {C(5) [0.008], C(6) [0.046], C(7) [0.074], C(8) [0.053] }
plane 4	C(4a) [−0.005], C(9) [−0.002], C(9a) [0.005], C(10) [0.002] $6.499x + 5.355y + 3.243z = 4.371$ {C(1) [0.055], C(2) [0.112], C(3) [0.062], C(4) [0.006] }
plane 5	C(9) [−0.028], C(10) [−0.018], C(11) [0.037], C(12) [0.002], C(13) [0.047], O [−0.040] $9.284x - 1.637y - 4.464z = -2.094$

[a] The planes are defined by the atoms in the first line(s) with, where relevant, atomic deviations, in angstroms, in square brackets. The equation of the plane is given in the following line in the form $Ax + By + Cz = D$, where x, y, z correspond to fractional coordinates. Atomic deviations of other atoms from the plane, in angstroms, are given in the subsequent lines. [b] Angles between planes: plane 1-plane 2, 5.92°; plane 3-plane 4, 45.11°.

The geometry at the carbonyl group is of special interest in view of the low-frequency IR carbonyl bands at 1677 (KBr) and 1681 cm^{-1} (CCl_4). Interestingly, the anisotropy of the thermal motion of the carbonyl oxygen atom is greater than that of the attached carbon atom; the orientation of its ellipsoid of vibration (Figure 1) suggests that a small amount of pyramidalization of the carbonyl carbon may be occurring as part of the thermal motion.

Does the solid-state structure, especially the shape of the C(10), C(11), C(12), C(13), C(9) unit result from packing forces? Examination of the crystal packing (Figure 2, supplementary material) shows that while the carbonyl oxygen atom is effectively clamped between two carbon atoms, one in each of two neighboring molecules, with O···C contacts of 3.27 and 3.36 Å, these are close to the expected van der Walls radii sums for oxygen and carbon (1.4 + 1.8 = 3.2 Å) and do not in our opinion rep-

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Table II. Selected Dihedral Angles[a]

C(9m)-C(9)-C(13)-C(13m)	62.4 (5)	C(10m)-C(10)-C(11)-C(11m)	−62.5 (5)
C(9m)-C(9)-C(13)-C(13n)	−58.4 (5)	C(10m)-C(10)-C(11)-C(11n)	59.3 (5)
C(9m)-C(9)-C(13)-C(12)	−177.5 (4)	C(10m)-C(10)-C(11)-C(12)	177.5 (4)
C(9)-C(13)-C(12)-O	174.7 (4)	O-C(12)-C(11)-C(10)	−175.5 (4)
C(13m)-C(13)-C(12)-O	−62.0 (6)	O-C(12)-C(11)-C(11m)	61.5 (5)
C(13n)-C(13)-C(12)-O	52.4 (5)	O-C(12)-C(11)-C(11n)	−53.8 (5)
C(9)-C(8a)-C(10a)-C(10)	−0.8 (5)	C(10)-C(4a)-C(9a)-C(9)	1.1 (6)
C(8a)-C(10a)-C(10)-C(4a)	41.8 (5)	C(8a)-C(9)-C(9a)-C(4a)	39.8 (5)
C(10a)-C(10)-C(4a)-C(9a)	−41.7 (5)	C(10a)-C(8a)-C(9)-C(9a)	−39.8 (5)
C(8a)-C(9)-C(13)-C(12)	−55.5 (5)	C(10a)-C(10)-C(11)-C(12)	56.5 (9)
C(9a)-C(9)-C(13)-C(12)	62.1 (5)	C(4a)-C(10)-C(11)-C(12)	−61.5 (4)
C(9)-C(13)-C(12)-C(11)	−7.0 (6)	C(10)-C(11)-C(12)-C(13)	6.2 (6)

[a] Errors in dihedral angles have been calculated by using the formula of R. H. Stanford and J. Waser (*Acta Crystallogr., Sect. A* **1972**, *A28*, 213), assuming isotropic positional standard deviations.

Table III. Important Nonbonded Contacts[a]

(a) Intramolecular			
O···C(13m)	2.81	O···C(11m)	2.81
O···C(13n)	2.72	O···C(11n)	2.72
C(9m)···C(13m)	3.06	C(10m)···C(11m)	3.03
C(9m)···C(13n)	3.02	C(10m)···C(11n)	2.99
(b) Intermolecular			
O···C(7)^1	3.27	O···C(13m)^2	3.36

$C(7)^1 = C(7) \times [-x, {}^1/_2 + y, 2 - z]$
$C(13m)^2 = C(13m) \times [1 - x, {}^1/_2 + y, 2 - z]$

[a] In angstroms. Esd's have been calculated as described in the footnote to Table IV and are all close to 0.01 Å.

resent unusually strong packing forces.

While the foregoing discussion has centered on the effects of steric crowding in the bridge, a brief word about the "basal cyclohexadiene" moiety is in order. By virtue of the three-carbon bridge, the six-membered ring has been locked into a shallow boat, the sum of the absolute values of its six dihedral angles (Table II) being 1.1 + 41.7 + 41.8 + 0.0 + 39.8 = 164.2°. This value compares with a value of 0° for planar benzene (6 sp^2 carbons) and 336° for cyclohexane[13] (no sp^2 carbons).

In conclusion, the shape and conformational mobility of the three-carbon bridge in substituted bicyclo[3.2.2]-nona-6,8-dienes[14] can vary considerably and may depend on the nature of the substituents. The **2a** molecule is an extreme example in that it shows very little puckering, with the five contiguous atoms C(9)–C(13)–C(12)–C(11)–C(10) being almost in one plane. The series of compounds **2a–c** also adds a facet to the use of 9,10-bridged anthracenes as models for studying steric effects and steric congestion.[15] Despite the bulky bridge, these compounds can be prepared readily, presumably by a stepwise reaction.

Experimental Section

9,10,11,11,13,13-Hexamethyl-12-oxo-9,10-propanoanthracene (2a). 9,10-Dimethylanthracene[16] (1.15 g, 5.6 mmol) is dissolved in the minimum amount of dry benzene (10 mL) or, alternatively dioxane, with stirring at 80 °C in a 50-mL, three-necked flask fitted with a reflux condenser, drying tube, and pressure-equalizing dropping funnel. Commercial zinc dust (2.0 g, 32 mmol) and copper(I) chloride (0.32 g, 3.2 mmol) are added through a powder funnel, and the hot mixture is stirred for several minutes. A solution of 2,4-dimethyl-2,4-dibromo-3-pentanone (2.72 g, 10 mmol) and chlorotrimethylsilane (1.3 g, 12 mmol) is then added dropwise over a 0.5-h period. After being stirred continuously at 80 °C for 20 h, the hot slurry is suction filtered through a dichloromethane-moistened Celite 545 pad (3.0 g) contained in a no. 2 sintered-glass funnel and washed with 50 mL of dichloromethane. The combined filtrate is concentrated on a rotary evaporator and the crude product chromatographed on silica gel 60 (Macherey and Nagel, 0.05–0.2 mm; 80:1); elution with dichloromethane gives, after 9,10-dimethylanthracene, **2a** as colorless needles: 0.72 g (40%); mp 252–254 °C (see Table V for additional data).

9,10-Dimethoxy-11,11,13,13-tetramethyl-12-oxo-9,10-propanoanthracene (2b). This compound was prepared in the same manner, substituting 9,10-dimethoxyanthracene[17] (**1b**; 1.30 g, 5.6 mmol) for 9,10-dimethylanthracene. After workup and chromatography, **2b** was obtained as colorless needles: 0.60 g (30%); mp 180–181 °C.

A sample of **2a** for X-ray analysis was prepared by recrystallization from petroleum ether (bp 70 °C), subsequent sublimation (140 °C, 0.2 mm Hg), and final evaporative recrystallization from petroleum ether (bp 40–50 °C), giving long and thin transparent plates. The specimen used for X-ray work was a fragment from a larger crystal with dimensions of 0.34 × 0.26 × 0.05 mm and was mounted in a 0.3-mm Lindemann capillary. The unit cell parameters and crystal orientation were determined by using SEARCH and INDEX routines on a Nonius CAD4 diffractometer. Accurate parameters were obtained by least-squares analysis of the setting angles for 15 reflections with $25 < \theta < 35°$ automatically centred on the diffractometer. The values obtained are $a = 9.737$ (2) Å, $b = 9.651$ (2) Å, $c = 9.741$ (2) Å, $\beta = 103.06$ (2)°, $V = 891.7$ Å^3. Monoclinic symmetry was confirmed by a fast, low-angle data collection. The density of the crystals was measured by flotation (aqueous KBr) as 1.18 g cm^{-3}: for $z = 2$, the calculated density is 1.19 g cm^{-3}. Intensity data were recorded in a manner described previously[18] by using Ni-filtered Cu Kα radiation and a maximum measuring time of 90 s. Of the 1554 intensities measured in the range $3.0 < \theta < 60°$, 1304 satisfied the condition $I > 1.5\sigma(I)$ and were used for the structure refinement. No absorption corrections were applied (μ(Cu Kα) = 4.6 cm^{-1}). The systematic absences $0k0$ ($k = 2n + 1$) indicated two possible space groups, $P2_1$ and $P2_1/m$. E statistics ($\langle |E^2 - 1| \rangle = 0.79$) suggested the noncentrosymmetric space group $P2_1$.

All unique data were used for structure solution by direct methods. A convergence map was computed by using 272 reflections with $E \geq 1.2$; origin, enantiomorph, and multisolution phases were selected by hand and phase sets obtained by multisolution tangent refinement. After several unsuccessful choices of a starting set, an E map was obtained which gave the positions of 23 out of 24 of the heavy atoms. The remaining atom was located on a difference map. Full-matrix, least-squares refinement with all atoms assigned anisotropic temperature factors gave $R = 0.084$. All hydrogen atoms were located on a difference map and included in the refinement in idealized positions (C–H, 1.08 Å; H–C–H, 109.5°) riding on the parent carbon atoms, with one overall isotropic temperature factor for phenyl hydrogens and one for methyl hydrogens. The refinement converged at $R = 0.043$ and $R_w = 0.048$, where $R = \sum\Delta/\sum|F_o|$ and $R_w = \sum w^{1/2}\Delta/$

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Table IV. Bond Lengths and Angles between Nonhydrogen Atoms[a]

(a) Bond Lengths, Å

C(2)-C(1)	1.402 (7)	C(6)-C(5)	1.381 (6)
C(3)-C(2)	1.345 (8)	C(7)-C(6)	1.358 (7)
C(4)-C(3)	1.378 (7)	C(8)-C(7)	1.377 (6)
C(4a)-C(4)	1.394 (5)	C(8a)-C(8)	1.402 (5)
C(10)-C(4a)	1.519 (6)	C(9)-C(8a)	1.521 (6)
C(10a)-C(10)	1.530 (6)	C(9a)-C(9)	1.539 (6)
C(10a)-C(5)	1.393 (5)	C(9a)-C(1)	1.404 (6)
C(10a)-C(8a)	1.390 (5)	C(9a)-C(4a)	1.393 (6)
C(9m)-C(9)	1.557 (7)	C(10m)-C(10)	1.538 (7)
C(13)-C(9)	1.571 (7)	C(11)-C(10)	1.574 (6)
C(11m)-C(11)	1.533 (7)	C(13m)-C(13)	1.525 (7)
C(11n)-C(11)	1.536 (8)	C(13n)-C(13)	1.540 (7)
C(12)-C(11)	1.552 (7)	C(13)-C(12)	1.534 (8)
O-C(12)	1.213 (6)		

(b) Bond Angles (deg)

C(9a)-C(1)-C(2)	119.5 (5)	C(10a)-C(5)-C(6)	121.3 (4)
C(3)-C(2)-C(1)	121.2 (5)	C(7)-C(6)-C(5)	119.8 (4)
C(4)-C(3)-C(2)	119.6 (4)	C(8)-C(7)-C(6)	119.9 (4)
C(4a)-C(4)-C(3)	121.5 (5)	C(8a)-C(8)-C(7)	121.7 (4)
C(9a)-C(4a)-C(4)	119.2 (4)	C(10a)-C(8a)-C(8)	118.0 (4)
C(4a)-C(9a)-C(1)	119.0 (4)	C(8a)-C(10a)-C(5)	119.3 (4)
C(10)-C(4a)-C(4)	122.3 (4)	C(9)-C(8a)-C(8)	122.8 (4)
C(10)-C(4a)-C(9a)	118.5 (3)	C(10a)-C(8a)-C(9)	119.2 (3)
C(10a)-C(10)-C(4a)	108.1 (3)	C(9a)-C(9)-C(8a)	108.0 (3)
C(10)-C(10a)-C(8a)	118.5 (3)	C(9)-C(9a)-C(4a)	118.9 (4)
C(10)-C(10a)-C(5)	122.2 (4)	C(9)-C(9a)-C(1)	122.1 (4)
C(9m)-C(9)-C(8a)	110.5 (4)	C(10m)-C(10)-C(4a)	110.3 (3)
C(13)-C(9)-C(8a)	109.5 (3)	C(11)-C(10)-C(4a)	109.3 (3)
C(13)-C(9)-C(9a)	108.4 (4)	C(11)-C(10)-C(10a)	108.9 (3)
C(13)-C(9)-C(9m)	110.5 (4)	C(11)-C(10)-C(10m)	109.7 (4)
C(9m)-C(9)-C(9a)	109.8 (4)	C(10m)-C(10)-C(10a)	110.5 (3)
C(12)-C(13)-C(9)	115.8 (4)	C(12)-C(11)-C(10)	116.0 (4)
C(13m)-C(13)-C(9)	111.2 (4)	C(11m)-C(11)-C(10)	110.8 (4)
C(13n)-C(13)-C(9)	110.8 (4)	C(11n)-C(11)-C(10)	110.5 (4)
C(13n)-C(13)-C(13m)	108.5 (6)	C(11n)-C(11)-C(11m)	109.6 (5)
C(13m)-C(13)-C(12)	105.3 (6)	C(12)-C(11)-C(11m)	105.3 (4)
C(13n)-C(13)-C(12)	104.7 (4)	C(12)-C(11)-C(11n)	104.1 (4)
C(13)-C(12)-C(11)	130.2 (3)	C(11)-C(12)-O	114.3 (4)
C(13)-C(12)-O	115.4 (3)		

[a] Estimated standard deviations in parentheses. The full least-squares covariance matrix has been used in estimating the standard deviations in bond lengths and angles; these esd's have been increased to allow for uncertainties in unit cell dimensions.

Table V. Physical Properties and Mass and ^{1}H NMR Spectral Data of Compounds 2a-c

compd	R	mp, °C	molec formula[a] (mol wt)	temp, °C: mass spectrum[b] (70 eV), *m/e* (relative intensity)	^{1}H NMR (90 MHz, $CDCl_3/Me_4Si$) δ
2a	CH_3	252-254	$C_{23}H_{26}O$ (318.2)	at 100: 318 (3, M$^+$) 207 (20), 206 (100), 192 (4), 191 (18)	1.00 (s, 12 H), 1.87 (s, 6 H), 7.23 (m, 4 H), 7.50 (m, 4 H)
2b	OCH_3	180-181	$C_{23}H_{26}O_3$ (350.2)	at 150: 350 (7, M$^+$), 249 (5), 238 (100), 223 (67), 208 (10), 202 (8), 189 (8), 180 (10), 165 (8), 152 (23)	0.97 (s, 12 H), 3.45 (s, 6 H), 7.45 (m, 8 H)
2c	H	146-147	$C_{21}H_{22}O$ (290.4)	at 120: 290 (20, M$^+$), 275 (6), 206 (17), 178 (100)	1.05 (s, 12 H), 3.62 (s, 2 H), 7.19 (m, 8 H)

[a] The microanalyses were in satisfactory agreement with the calculated values: C, ±0.08; H, ±0.11; O, ±0.12. [b] Measured on a Varian CH-5 spectrometer.

$\sum w^{1/2}|F_o|$; the corresponding unweighted and weighted R factors for the full set of data are 0.045 and 0.049, respectively. (The function minimized in the least-squares refinement is $\sum w\Delta^2$.) The average and maximum shifts in the last refinement cycle were, respectively, 0.070 and 1.280. Weights were calculated by using the formula $w = 1/(\sigma^2(F_o) + g|F_o|^2)$, with σ calculated as previously described:[18] a value for g of 0.001 gave satisfactory agreement analyses. A final difference map was featureless. The final atomic parameters are given in Table VI (supplementary material). Lists of observed and calculated structure factors are also available as supplementary material. Crystallographic calculations were carried out on the ICL 1900 and 2980 computers by using the program and scattering factor data sources listed in ref 18.

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Registry No. 1a, 781-43-1; **1b**, 2395-97-3; **2a**, 77256-13-4; **2b**, 77256-14-5; **2c**, 69693-06-7; 2,4-dimethyl-2,4-dibromo-3-pentanone, 17346-16-6.

Supplementary Material Available: Table of atomic coordinates and anisotropic temperature factors and Figure 2 (crystal packing) (3 pages). Ordering information is given on any current masthead page. (Lists of observed and calculated structure factors for 9,10,11,11,13,13-hexamethyl-12-oxo-9,10-propanoanthracene are available from the author.)

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[18] Hursthouse, M. B.; Jones, R. A.; Malik, K. M. A.; Wilkinson, G. *J. Am. Chem. Soc.* **1979**, *101*, 4128.