ment converged at $R = 0{\cdot}047$ and $wR = 0{\cdot}055$, where $w = 1/\sigma^2(F)$ and $S = 0{\cdot}3$. The overall thermal parameter used for H atoms refined to $U = 0{\cdot}086\,(3)$ Å^2. $\Delta/\sigma = 0{\cdot}02$ (av.) and 0·10 (max.) for non-H-atom parameters and $\Delta/\sigma = 0{\cdot}06$ (av.) and 0·13 (max.) for H-atom parameters; $-0{\cdot}24 < \Delta\rho <$ 17 e Å^{-3}. Scattering factors were taken from *SHELX*. Final atomic parameters and isotropic thermal parameters are given in Table 1.* Bond lengths and bond angles are given in Table 2. The conformation of the steroid molecule and atom-numbering scheme are shown in Fig. 1. The molecular packing and hydrogen bonding are illustrated in Fig. 2, which shows a stereoview down **c**.

Related literature. Structural data of 3-oxo-4-ene steroids have been reviewed by Griffin, Duax & Weeks (1984). Related steroid structures are gestodene (13-ethyl-17β-hydroxy-18,19-dinor-17α-pregna-4,15-dien-20-yn-3-one: van Geerestein, Duisenberg, Duitz, Kanters & Kroon, 1987) and 3-ketodesogestrel (13-ethyl-17β-hydroxy-11-methylene-18,19-dinor-17α-pregn-4-en-20-yn-3-one: van Geerestein, Kanters & Kroon, 1987).

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References

Geerestein, V. J. van, Duisenberg, A. J. M., Duitz, M. H. G., Kanters, J. A. & Kroon, J. (1987). *Acta Cryst.* C**43**, 2402–2405.

Geerestein, V. J. van, Kanters, J. A. & Kroon, J. (1987). *Acta Cryst.* C**43**, 2398–2401.

Griffin, J. F., Duax, W. L. & Weeks, C. M. (1984). *Atlas of Steroid Structure*, Vol. II. New York: Ronald Press.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS86*. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.

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* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44466 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Cycloaddition Product from the Reaction of Benzoquinone and 9-Bromoanthracene

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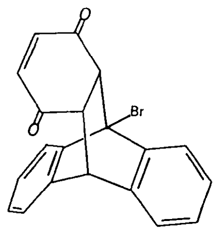
Abstract. 9-Bromo-4a,9,9a,10-tetrahydro-9,10-*o*-benzenoanthracene-1,4-dione, $C_{20}H_{13}BrO_2$, $M_r = 365{\cdot}23$, triclinic, $P\bar{1}$, $a = 6{\cdot}988\,(2)$, $b = 8{\cdot}273\,(1)$, $c = 13{\cdot}109\,(4)$ Å, $\alpha = 93{\cdot}55\,(2)$, $\beta = 98{\cdot}17\,(2)$, $\gamma = 101{\cdot}49\,(2)°$, $V = 731{\cdot}9\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}657$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 27{\cdot}85$ cm^{-1}, $F(000) = 368$, $T = 293$ K, $R = 0{\cdot}0548$ for 2241 independent reflections. The six-membered diketone ring is in a boat conformation with the diketone moieties bent away from the adjacent phenyl ring. The ketone O(1) adjacent to the Br is bent away from the anthracene further [C(18)C(17)C(16)O(1) = −154·9 (6)°] than is O(2) [C(17)C(18)C(19)O(2) = 166·8 (6)°]. The interplanar angles around the C(7)—C(14) direction are 123·7 (7)° between C(1)C(6)-C(7)C(14) and C(7)C(8)C(13)C(14), 118·5 (7)° between C(1)C(6)C(7)C(14) and C(7)C(14)C(15)C(20), and 117·8 (7)° for the remaining angle.

Experimental. Pale yellow, transparent platelet of the title compound (1) from benzene solution of a sample prepared in 1945, 0·40 × 0·57 × 0·10 mm; Nicolet *R3M/μ* update of $P2_1$ diffractometer; data collected in Wyckoff mode (2θ fixed, ω varied; $3{\cdot}0 \le 2\theta \le 50{\cdot}0°$), variable scan rate (4–29·3° min^{-1}), graphite-mono-



(1)

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Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Br	1922 (1)	7584 (1)	5170 (1)	39 (1)
C(1)	1727 (6)	6575 (6)	7227 (4)	24 (1)
C(2)	1729 (7)	4977 (6)	6843 (4)	32 (2)
C(3)	1730 (8)	3756 (6)	7528 (5)	40 (2)
C(4)	1686 (8)	4100 (6)	8552 (5)	39 (2)
C(5)	1629 (7)	5716 (6)	8942 (4)	34 (2)
C(6)	1687 (7)	6947 (5)	8278 (4)	24 (1)
C(7)	1591 (7)	8728 (6)	8557 (4)	27 (2)
C(8)	3291 (7)	9825 (5)	8179 (4)	24 (1)
C(9)	4668 (7)	11082 (6)	8764 (4)	28 (2)
C(10)	6151 (8)	12007 (7)	8308 (4)	36 (2)
C(11)	6221 (8)	11692 (7)	7277 (4)	36 (2)
C(12)	4829 (7)	10421 (6)	6676 (4)	31 (2)
C(13)	3367 (7)	9490 (5)	7125 (4)	24 (1)
C(14)	1703 (7)	8073 (5)	6614 (4)	23 (1)
C(15)	−279 (7)	8675 (6)	6722 (4)	29 (2)
C(16)	−2070 (8)	7487 (7)	6111 (4)	39 (2)
O(1)	−2574 (6)	7546 (6)	5191 (3)	55 (2)
C(17)	−3242 (8)	6240 (7)	6661 (5)	48 (2)
C(18)	−3251 (8)	6470 (7)	7675 (6)	50 (2)
C(19)	−2039 (7)	7933 (7)	8320 (4)	38 (2)
O(2)	−2331 (7)	8255 (6)	9186 (4)	60 (2)
C(20)	−359 (7)	8996 (6)	7891 (4)	29 (2)

Table 2. *Bond distances* (Å) *and valence angles* (°)

Br—C(14)	1·945 (5)	C(1)—C(2)	1·385 (7)
C(1)—C(6)	1·399 (7)	C(1)—C(14)	1·520 (7)
C(2)—C(3)	1·392 (8)	C(3)—C(4)	1·361 (9)
C(4)—C(5)	1·411 (8)	C(5)—C(6)	1·379 (7)
C(6)—C(7)	1·512 (7)	C(7)—C(8)	1·509 (7)
C(7)—C(20)	1·573 (7)	C(8)—C(9)	1·374 (6)
C(8)—C(13)	1·402 (7)	C(9)—C(10)	1·391 (7)
C(10)—C(11)	1·371 (8)	C(11)—C(12)	1·395 (6)
C(12)—C(13)	1·378 (7)	C(13)—C(14)	1·521 (6)
C(14)—C(15)	1·584 (7)	C(15)—C(16)	1·518 (6)
C(15)—C(20)	1·550 (7)	C(16)—O(1)	1·215 (7)
C(16)—C(17)	1·475 (9)	C(17)—C(18)	1·332 (10)
C(18)—C(19)	1·470 (7)	C(19)—O(2)	1·204 (8)
C(19)—C(20)	1·518 (7)		
C(2)—C(1)—C(6)	120·6 (5)	C(2)—C(1)—C(14)	127·0 (4)
C(6)—C(1)—C(14)	112·3 (4)	C(1)—C(2)—C(3)	118·7 (5)
C(2)—C(3)—C(4)	121·3 (5)	C(3)—C(4)—C(5)	120·3 (5)
C(4)—C(5)—C(6)	119·0 (5)	C(1)—C(6)—C(5)	120·0 (4)
C(1)—C(6)—C(7)	113·8 (4)	C(5)—C(6)—C(7)	126·1 (5)
C(6)—C(7)—C(8)	107·8 (4)	C(6)—C(7)—C(20)	105·6 (3)
C(8)—C(7)—C(20)	106·7 (4)	C(7)—C(8)—C(9)	125·8 (4)
C(7)—C(8)—C(13)	114·0 (4)	C(9)—C(8)—C(13)	120·2 (4)
C(8)—C(9)—C(10)	119·6 (5)	C(9)—C(10)—C(11)	120·4 (4)
C(10)—C(11)—C(12)	120·5 (5)	C(11)—C(12)—C(13)	119·5 (5)
C(8)—C(13)—C(12)	119·9 (4)	C(8)—C(13)—C(14)	112·1 (4)
C(12)—C(13)—C(14)	128·0 (4)	Br—C(14)—C(1)	112·2 (3)
Br—C(14)—C(13)	110·9 (3)	C(1)—C(14)—C(13)	109·0 (4)
Br—C(14)—C(15)	111·4 (3)	C(1)—C(14)—C(15)	107·6 (4)
C(13)—C(14)—C(15)	105·5 (4)	C(14)—C(15)—C(16)	111·6 (4)
C(14)—C(15)—C(20)	108·0 (4)	C(16)—C(15)—C(20)	114·9 (4)
C(15)—C(16)—O(1)	122·0 (5)	C(15)—C(16)—C(17)	118·6 (5)
O(1)—C(16)—C(17)	119·4 (5)	C(16)—C(17)—C(18)	121·6 (5)
C(17)—C(18)—C(19)	122·4 (6)	C(18)—C(19)—O(2)	120·7 (5)
C(18)—C(19)—C(20)	118·8 (5)	O(2)—C(19)—C(20)	120·5 (4)
C(7)—C(20)—C(15)	110·3 (4)	C(7)—C(20)—C(19)	105·4 (4)
C(15)—C(20)—C(19)	117·4 (4)		

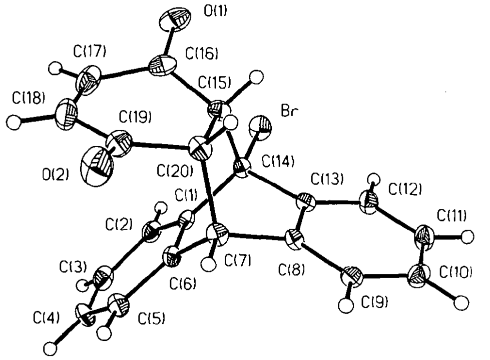

Fig. 1. Drawing of the title compound with thermal ellipsoids at the 35% probability level and H atoms represented by spheres of arbitrary size.

chromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 15 reflections ($20{\cdot}28 \leq 2\theta \leq 27{\cdot}50°$), angles measured by a centering routine; no systematic absences, chemistry and statistics consistent with space group $P\bar{1}$, Laue symmetry $\bar{1}$; monitored reflections ($0\bar{4}3$ and $\bar{2}\bar{3}4$) show no statistically significant changes in intensities; 2572 independent reflections measured ($0 \leq h \leq 8$, $-9 \leq k \leq 9$, $-15 \leq l \leq 15$), $2241 \geq 3\sigma(I)$; Lorentz–polarization corrections, ψ-scan empirical absorption correction (transmission factors 1·000 to 0·431, $R_{\text{merge}} = 0{\cdot}041$); structure solved by direct-methods techniques, block-cascade least-squares refinement, H atoms allowed to ride at a fixed distance from C atoms with a single fixed isotropic temperature parameter; $R = 0{\cdot}0548$, $wR = 0{\cdot}0555$ for 209 parameters and 2241 reflections ($R = 0{\cdot}0679$, $wR = 0{\cdot}0894$ for all 2572 reflections), $S = 1{\cdot}791$, $(\Delta/\sigma)_{\text{max}} = 0{\cdot}005$, $(\Delta/\sigma)_{\text{avg}} = 0{\cdot}001$; largest peaks in the final difference map of 0·63 and −0·50 e Å^{-3} located 1·09 Å from the Br atom; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}000621F_o^2]^{-1}$; extinction correction $F^* = [1{\cdot}0 + 0{\cdot}002gF_c^2/\sin(2\theta)]^{0{\cdot}25}$ applied, g refined to 0·03 (1). All computer programs supplied by Nicolet (Nicolet Instrument Corporation, 1986) for Desktop 30 Microeclipse and Nova 4/C configurations; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Fig. 1 is a drawing of compound (1). Table 1 lists atomic positional parameters, and Table 2 gives interatomic distances and valence angles.†

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† Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44425 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The 4a,9,9a,10-tetrahydro-9,10-*o*-benzenoanthracene-1,4-diones are intermediates in the synthesis of triptycenes (Bartlett, Cohen, Cotman, Kornblum, Landry & Lewis, 1950). The above compounds and the corresponding triptycenes have been the subjects of EPR investigations (Quast & Fuchsbauer, 1986; Bertsch & Reinhardt, 1986).

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References

BARTLETT, P. D., COHEN, S. G., COTMAN, J. D., KORNBLUM, N., LANDRY, J. R. & LEWIS, E. S. (1950). *J. Am. Chem. Soc.* **72**, 1003–1004.

BERTSCH, A. & REINHARDT, G. (1986). *Angew. Chem.* **98**, 361–362.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Nicolet Instrument Corporation (1986). *SHELXTL for Desktop* 30 (*Microeclipse*), PN-269-1040340. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

QUAST, H. & FUCHSBAUER, H. L. (1986). *Chem. Ber.* **119**, 1016–1038.

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Structure of 4-*O*-Ethylascofuranone

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Abstract. [*S*-(*E*,*E*)]-5-Chloro-4-ethoxy-2-hydroxy-6-methyl-3-[3-methyl-7-(tetrahydro-5,5-dimethyl-4-oxo-2-furanyl)-2,6-octadienyl]benzaldehyde, $C_{25}H_{33}ClO_5$, $M_r = 448{\cdot}99$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}724$ (1), $b = 34{\cdot}626$ (2), $c = 6{\cdot}122$ (1) Å, $V = 2485{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}200$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 16{\cdot}13$ cm^{-1}, $F(000) = 960$, $T = 298$ K, final $R = 0{\cdot}050$ for 2423 unique reflections $[F_o^2 > 2\sigma(F_o^2)]$. The title compound has a 'round' molecular conformation, turning at the middle of the sesquiterpenoid moiety. The intramolecular van der Waals contacts, observed at the center of the molecule, stabilize the conformation.

Experimental. Colorless prisms of 4-*O*-ethylascofuranone from *n*-hexane–acetone [1:1 (*v*/*v*)]. Crystal size 0·40 × 0·35 × 0·20 mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu $K\alpha$ radiation, graphite monochromator, θ–2θ scan with scan speed 0·87–4·12° min^{-1} in θ, scan width $(0{\cdot}40 + 0{\cdot}14\tan\theta)°$. Range of indices, $0 \le h \le 14$, $0 \le k \le 43$, $0 \le l \le 7$ ($2\theta < 150°$). Lattice constants determined based on 25 2θ values ($26 < 2\theta < 55°$). Variation of standard <0·6%; 2958 unique reflections measured; 2423 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. Systematic absences $h00$, h odd; $0k0$, k odd; $00l$, l odd. No corrections for absorption. Structure solved by direct methods with *MULTAN* (Main, Woolfson & Germain, 1971). Refined by full-matrix least squares. The locations of all H atoms were found from a difference Fourier map. Non-H atoms refined with anisotropic thermal parameters, and H atoms with isotropic thermal parameters ($B = 5{\cdot}0$ Å^2: fixed). $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 1{\cdot}0$ for $F_o < 1011{\cdot}59$, $w = (1011{\cdot}59/F_o)^2$ for $F_o \ge 1011{\cdot}59$. Final $R = 0{\cdot}050$, $wR = 0{\cdot}049$, $S = 7{\cdot}23$ for 413 variables, secondary-extinction factor $g = 2{\cdot}50\ (4) \times 10^{-6}$ $[|F_o| = |F_c|/(1 + gIc)]$; $\Delta/\sigma < 0{\cdot}47$, largest peak in final ΔF map $+0{\cdot}21$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1984), *ORTEP*II (Johnson, 1976). The structure of the title

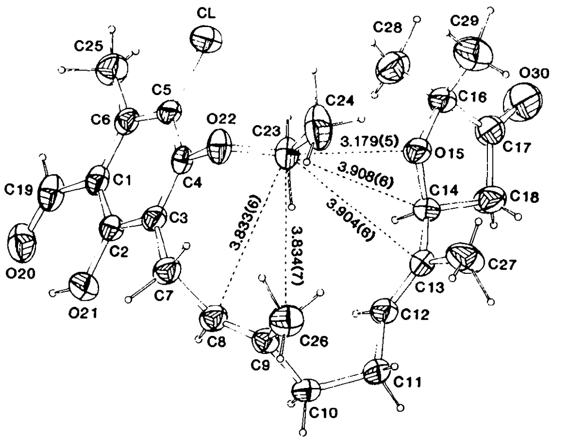

Fig. 1. A perspective view of the molecule with the numbering scheme.

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