

Fig. 1. The molecule of the title compound in the crystal, showing the atom-labelling scheme. Radii are arbitrary.

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Structure of 17 β -Acetoxy-2 β -bromo-5-methyl-5 β -estran-3-one

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Abstract. $C_{21}H_{31}BrO_3$, $M_r = 411.4$, orthorhombic, $P2_12_12_1$, $a = 7.264$ (2), $b = 8.400$ (1), $c = 33.217$ (5) Å, $U = 2027$ (1) Å³, $Z = 4$, $D_x = 1.35$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 26.88$ cm⁻¹, $F(000) = 864$, $T = 298$ K, final $R = 0.064$ for 2448 observed reflections and 238 variable parameters. The α -bromo-ketone moiety is planar with a dihedral angle of 0.3 (9°).

Experimental. The title compound was formed by the bromination of 17 β -acetoxy-5-methyl-5 β -estran-3-one with pyridinium hydrobromide perbromide (Boeyens, Bull & van Rooyen, 1980) and yielded colourless needles (methylene chloride/methanol, m.p. 491–494 K). Crystal $0.44 \times 0.42 \times 0.51$ mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromatized radiation, unit cell from 25 reflections ($17 \leq \theta \leq 51^\circ$), 2516 reflections for $5 \leq \theta \leq 78^\circ$ in the range $0 \leq h \leq 9$, $0 \leq k \leq 10$, $0 \leq l \leq 42$ using $\omega/2\theta$ scans where ω changed as $0.74 + 0.14 \tan \theta^\circ$ with a variable but maximum speed that corresponds to $5.49^\circ \text{ min}^{-1}$. Three standard reflections were measured every hour, decay 5.6%, decay correction: minimum 1.000, maximum 1.029, average 1.014, Lorentz-polarization correction. 2516 reflections measured, 147 had $F_o \leq 4\sigma(F_o)$, 2448 unique reflections with $F_o > 0$ used.

in CH_3CN (Roesky, Lucas, Weber, Djarrah, Egert, Noltemeyer & Sheldrick, 1985).

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References

- CLEGG, W. (1981). *Acta Cryst.* **A37**, 22–28.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 ROESKY, H. W., LUCAS, J., WEBER, K.-L., DJARRAH, H., EGERT, E., NOLTEMEYER, M. & SHELDRIK, G. M. (1985). *Chem. Ber.* **118**, 2396–2406.
 SHELDRIK, G. M. (1986). *SHELX*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

Structure solved using *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), blocked least-squares refinement using *SHELX76* (Sheldrick, 1976), F magnitudes, unit weights, all non-hydrogen atoms anisotropic, all H atoms isotropic in calculated positions with a common thermal parameter that was also refined [$U_{\text{iso}}(\text{H}) = 0.079$ (5) Å²], final $R = 0.064$ for 238 variables refined, maximum positional shift/e.s.d. = 0.22, residual electron density = $0.74 \text{ e } \text{Å}^{-3}$. Scattering factors from *International Tables for X-ray Crystallography* (1974). Table 1 gives the atom parameters, and Fig. 1 shows the molecular structure and atomic numbering scheme drawn by *ORTEP* (Johnson, 1965).*

Puckering parameters (Cremer & Pople, 1975) for ring A: $\theta = 2.0^\circ$, $\varphi = 29.9^\circ$, $Q = 0.58$ Å, 1C_4 chair conformation; ring B: $\theta = 173.4^\circ$, $\varphi = 350.0^\circ$, $Q = 0.57$ Å, 8C_5 chair conformation; ring C: $\theta = 6.9^\circ$, $\varphi = 273.6^\circ$, $Q = 0.58$ Å, $^8C_{12}$ chair conformation; ring D: $\varphi = 349.8^\circ$, $Q = 0.47$ Å, ^{13}T twist conformation.

* Lists of structure factors, bond lengths and angles, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44818 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* (\mathbf{a}_i \cdot \mathbf{a}_j).$$

	x	y	z	U_{eq}
C(1)	7982 (9)	2250 (9)	3060 (2)	51 (2)
C(2)	7325 (12)	1498 (10)	2677 (2)	60 (2)
C(3)	5248 (12)	1997 (9)	2602 (2)	54 (2)
C(4)	4084 (10)	1578 (9)	2945 (2)	50 (2)
C(5)	4721 (9)	2310 (8)	3344 (2)	45 (1)
C(6)	3530 (10)	1791 (9)	3697 (2)	52 (2)
C(7)	3709 (9)	58 (9)	3816 (2)	48 (1)
C(8)	5747 (8)	-329 (7)	3910 (2)	38 (1)
C(9)	6925 (8)	23 (8)	3540 (2)	37 (1)
C(10)	6782 (9)	1787 (8)	3421 (2)	41 (1)
C(11)	8950 (9)	-486 (8)	3609 (2)	43 (1)
C(12)	9174 (9)	-2186 (8)	3762 (2)	45 (1)
C(13)	8018 (9)	-2454 (7)	4138 (2)	38 (1)
C(14)	5974 (8)	-2060 (8)	4037 (2)	39 (1)
C(15)	4898 (10)	-2734 (10)	4395 (2)	59 (2)
C(16)	5987 (11)	-4251 (9)	4513 (2)	58 (2)
C(17)	7747 (10)	-4197 (8)	4259 (2)	44 (1)
C(18)	8738 (12)	-1498 (8)	4497 (2)	48 (2)
C(19)	4580 (12)	4134 (8)	3314 (3)	63 (2)
O(1)	9374 (7)	-4738 (5)	4486 (2)	50 (1)
C(20)	9603 (12)	-6306 (8)	4523 (2)	51 (2)
C(21)	11241 (13)	-6731 (9)	4768 (3)	71 (2)
O(2)	8539 (10)	-7254 (6)	4385 (2)	83 (2)
O(3)	4784 (8)	2669 (8)	2300 (2)	77 (2)
Br	8865 (2)	2132 (2)	2229 (1)	92 (1)

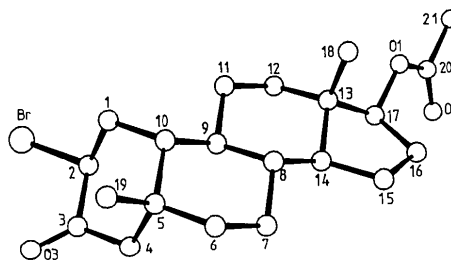


Fig. 1. Perspective view (ORTEP) of the title compound with the numbering scheme.

References

- BOEYENS, J. C. A., BULL, J. R. & VAN ROOYEN, P. H. (1980). *S. Afr. J. Chem.* **33**, 45–50.
- CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- KIRK, D. N. & HARTSHORN, M. P. (1968). *Steroid Reaction Mechanisms*, pp. 257–262. Amsterdam: Elsevier.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- SEDEE, A. G. J., BEIJERSBERGEN VAN HENEGOUWEN, G. M. J., GULT, W. & HAASNOOT, C. A. G. (1985). *J. Org. Chem.* **50**, 4182–4187.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

The bromo-ketone moiety is planar with the Br–C(2)–C(3)–O(3) dihedral angle $0.3(9)^\circ$ and a Br...O(3) distance of 3.0 Å. The packing is unexceptional.

Related literature. 5β -R steroids: Boeyens, Bull & van Rooyen (1980); Sedee, Beijersbergen van Henegouwen, Guijt & Haasnoot (1985); Kirk & Hartshorn (1968).

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A 1,1,3,3-Tetraphenyl-1,3-disiloxanediol Pyridinium Chloride Lattice Complex

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Abstract. C₂₄H₂₂O₃Si₂·C₅H₆N⁺·Cl⁻, $M_r = 530.16$, monoclinic, $P2_1/a$, $a = 20.508(7)$, $b = 9.785(1)$, $c = 14.042(3)$ Å, $\beta = 90.36(2)^\circ$, $V = 2818.8(8)$ Å³, $Z = 4$, $D_x = 1.247$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å,

$\mu = 1.57$ cm⁻¹, $F(000) = 1108$, $T = 298$ K, $R = 0.043$, $wR = 0.053$ for 3083 observed data. The Cl⁻ ion is involved in a fairly strong interaction with the proton on the pyridine N atom [Cl...H(10) 1.95(2) Å] and