electrostatically favorable anti-parallel fashion with C(5)···O(51) non-bonded contacts of 3·172 (7) Å.

In other directions, the only contacts less than van der Waals contacts are between O and H atoms. The amino H(N6) atom, a potential H-bond donor, approaches the nitro O(12) atom $(1-x, 1-y, 1-z)$, at a distance of 2·33 Å; this is significantly shorter than the O···H van der Waals distance (2·6 Å), but it would be considered a very weak hydrogen bond. Other close approaches are: carbonyl O(21) to H(C8) $(x-1, y, z)$, 2·25 Å; nitro O(13) to H(C7) $(x-1, y, z)$ 2·55 Å; nitro O(43) to H(C7) $(x, y-1, z)$ 2·41 Å; and nitro O(42) to H(N6) $(x, y-1, z)$ 2·37 Å.

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Structure of (5S)-9(10→5)abeo-1-Estrene-3,10,17-trione

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Abstract. $C_{18}H_{22}O_3$, $M_r = 286·37$, monoclinic, $P2_1$, $a = 11·337$ (1), $b = 6·436$ (1), $c = 11·751$ (2) Å, $\beta = 112·98$ (1)°, $V = 789·4$ (2) Å^3, $Z = 2$, $D_x = 1·205$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·753$ cm^{-1}, $F(000) = 308$, $T = 291$ K, $R = 0·0421$ for 972 observed reflections. The title compound is a synthetic steroid and exhibits a 9(10→5)abeo skeleton; the carbon at position 5 becomes a spiro center with absolute configuration S.

Introduction. Treatment of the quinol (1) (Bieri, Prewo, Briansó & Piniella, 1985) under acidic conditions (p-toluenesulfonic acid) affords the title compound (2) (72% yield) as described by Planas, Tomas & Bonet (1987). Its structure could not be solved by means of spectroscopic and analytical information. The present X-ray structure determination was undertaken to determine unambiguously the connectivity of the molecule and to confirm the absolute configuration at C5 which was assumed to be S according to the proposed reaction mechanism.
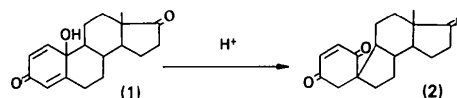
Experimental. Pale-yellow crystals obtained by slow evaporation from an acetone/petroleum ether solution. Parallelepiped crystal with dimensions 0·29 × 0·23 × 0·13 mm. Lattice parameters refined from 25 reflections in the range $5 < 2\theta < 20°$. Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation. 1516 independent reflections with $(\sin\theta)/\lambda \le 0·592$ Å^{-1}, $-12 \le h \le 12$, $0 \le k \le 7$, $0 \le l \le 13$, 972 with $I \ge 2·5\sigma(I)$. One standard reflection checked every 50 measurements; no significant deviation.

$C_{18}H_{22}O_3$

The application of the program systems *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and *YZARC*80 (Declercq, Germain, Wright & Woolfson, 1980; 100 sets with 100 random phases for each one) failed to show a recognizable fragment of the structure, which was eventually solved using the Patterson search program *ROTSEARCH*87 (Rius & Miravitlles, 1987) by introducing the *C* and *D* rings of the related steroid 5α-hydroxy-6β-methoxyandrost-3,17-dione (Piniella, 1985). The development of this solution with the highest figure of merit by Karle-recycling (*MULTAN*80) revealed the positions of 19 atoms. H atoms from difference Fourier synthesis. Full-matrix least-squares refinement on *F* using *SHELX*76 (Sheldrick, 1976); H

experimentally determined and refined with a common temperature factor $(B = 5.9\ \text{Å}^2)$, $w = 1/(\sigma^2 + 0.00778F^2)$, $R = 0.036$, $wR = 0.040$, $S = 0.66$ for 972 observed reflections. Final $(\Delta/\sigma)_{max} = 0.13$. Max. and min. heights in final Fourier synthesis 0.13 and $-0.17\ \text{e Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 1. *Final fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (Å^2) with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

|	x	y	z	B_{eq}
C1	2082 (4)	−3184 (11)	5245 (5)	5·03
C2	2398 (4)	−1671 (10)	6076 (5)	5·12
C3	1932 (4)	464 (0)	5739 (4)	4·79
O3	2372 (5)	1886 (9)	6462 (3)	8·01
C4	895 (4)	730 (10)	4495 (4)	4·50
C5	993 (3)	−627 (9)	3456 (4)	3·88
C6	−245 (4)	−462 (12)	2256 (4)	5·00
C7	164 (4)	−153 (12)	1174 (4)	5·25
C8	1587 (4)	−714 (10)	1693 (3)	3·87
C9	2047 (4)	97 (10)	3011 (3)	3·88
C10	1274 (4)	−2862 (9)	3916 (4)	4·30
O10	909 (4)	−4345 (9)	3230 (3)	6·54
C11	3465 (4)	−354 (12)	3784 (4)	5·06
C12	4304 (4)	489 (13)	3120 (4)	5·63
C13	3845 (4)	−364 (10)	1804 (4)	4·57
C14	2422 (4)	186 (10)	1082 (3)	4·17
C15	2219 (4)	−295 (12)	−259 (4)	5·19
C16	3434 (5)	604 (12)	−343 (4)	5·79
C17	4437 (4)	575 (11)	971 (4)	5·37
O17	5527 (3)	1195 (11)	1293 (3)	7·57
C18	4134 (5)	−2704 (12)	1816 (6)	6·12
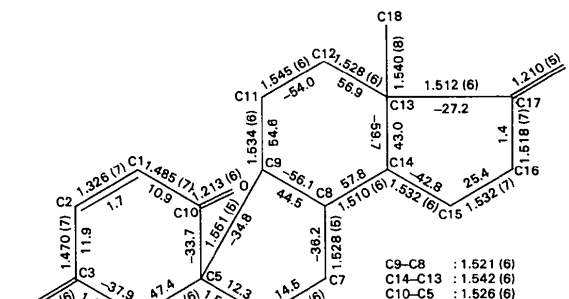
Fig. 1. Numbering scheme according to IUPAC rules; bond lengths (Å) (e.s.d.'s in parentheses) and intra-annular torsion angles (e.s.d.'s 0·2–0·4°).
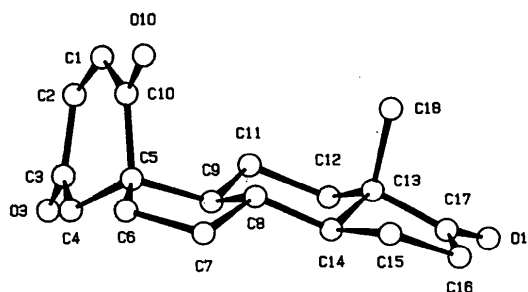
Fig. 2. A perspective view of the molecule (*ESTER*; Gaete, 1985).

Table 2. *Bond angles (°) with e.s.d.'s in parentheses*

|				
C10–C1–C2	123·4 (4)	C5–C10–C1	117·5 (4)
C3–C2–C1	121·5 (4)	O10–C10–C1	120·0 (4)
O3–C3–C2	120·6 (4)	O10–C10–C5	122·5 (4)
C4–C3–C2	115·9 (4)	C12–C11–C9	110·1 (4)
C4–C3–O3	123·5 (4)	C13–C12–C11	110·6 (4)
C5–C4–C3	116·1 (3)	C14–C13–C12	110·2 (4)
C6–C5–C4	111·1 (3)	C17–C13–C12	116·7 (4)
C9–C5–C4	113·6 (3)	C17–C13–C14	100·7 (3)
C9–C5–C6	102·4 (3)	C18–C13–C12	110·8 (4)
C10–C5–C4	108·9 (3)	C18–C13–C14	113·7 (4)
C10–C5–C6	112·0 (4)	C18–C13–C17	104·5 (4)
C10–C5–C9	108·7 (3)	C13–C14–C8	111·5 (3)
C7–C6–C5	107·7 (3)	C15–C14–C8	122·8 (4)
C8–C7–C6	104·7 (3)	C15–C14–C13	103·0 (3)
C9–C8–C7	102·4 (3)	C16–C15–C14	102·2 (3)
C14–C8–C7	118·7 (3)	C17–C16–C15	105·4 (4)
C14–C8–C9	108·9 (3)	C16–C17–C13	108·5 (4)
C8–C9–C5	103·7 (3)	O17–C17–C13	125·9 (4)
C11–C9–C5	120·7 (3)	O17–C17–C16	125·6 (4)
C11–C9–C8	113·7 (3)		
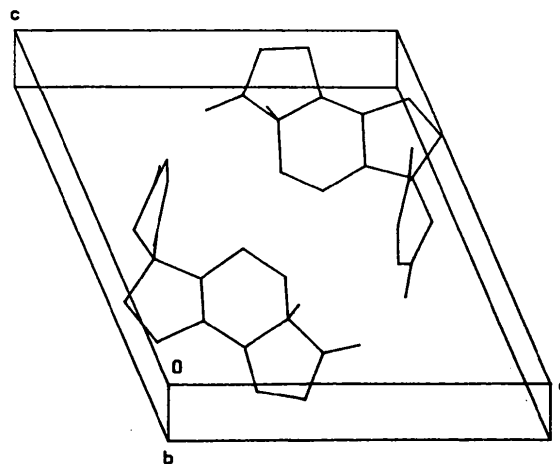
Fig. 3. Monoscopic view of the crystal packing (*ESTER*; Gaete, 1985).

Discussion. Final atomic parameters and equivalent isotropic temperature factors are given in Table 1.* Numbering scheme, bond distances and intra-annular torsion angles are depicted in Fig. 1; bond angles are shown in Table 2.

Bond distances and angles are normal for this kind of molecule. In the A ring there is electron delocalization along the Δ^1-3,10-dioxo system; bond distances around the spiro center C5 are slightly longer than normal (Birnbaum & Ferguson, 1969). The A ring has a flattened half-chair conformation due to the Δ^1-3,10-dioxo system. Rotational symmetry is dominant ($\Delta C_2^{1-2} = 3\cdot0$). The average of the intra-annular torsion angles is $23\cdot9°$. The five-membered B ring exhibits an 8β-envelope conformation ($\Delta C_2^6 = 1\cdot9$). The C ring has a highly symmetrical chair conformation; all asymmetry parameters are below $4\cdot6$. The D-ring conformation is a 14α envelope ($\Delta C_s^{14} = 1\cdot3$). All ring junctions are *trans* as indicated by the relevant torsion angles. The configuration at C5 is S, deduced by comparison with other known chiral centers in the molecule; this is in agreement with chemical considerations of the reaction mechanism (Planas, Tomas & Bonet, 1987). Fig. 2 is a perspective view of the molecule and Fig. 3 is a monoscopic drawing of the molecular packing.

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* Lists of structure amplitudes, anisotropic thermal parameters, and complete molecular geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44608 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Triphenylphosphine–Cyanoborane

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Abstract. $[P(C_6H_5)_3].BH_2CN$, $M_r = 301\cdot0$, monoclinic, $P2_1/n$ (from Weissenberg photograph and single-crystal diffractometry), $a = 18\cdot445$ (3), $b = 9\cdot554$ (2), $c = 18\cdot782$ (2) Å, $\beta = 93\cdot35$ (1)°, $V = 3304\cdot2$ (9) Å^3, $Z = 8$ (two formula units), $D_m = 1\cdot206$ (aq. KI), $D_x = 1\cdot210$ Mg m^{-3}, $\bar{\lambda}$(Mo $K\alpha$) = $0\cdot71073$ Å, $\mu = 16\cdot5$ mm^{-1}, $F(000) = 1264$, room temperature (293 K), final $R = 0\cdot044$ and $wR = 0\cdot057$ for 2623 observed reflections. The molecules have a tetrahedral overall structure and are held in the crystal by van der Waals forces only. The P–B bond distances in the two independent molecules differ slightly. The B–C≡N moiety is slightly non-linear.

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Introduction. Various derivatives of amine–boranes, in particular the cyano derivatives, $(CH_3)_3N.BH_2CN$,