The reason for the difference in biological activity between semicarbazones and thiosemicarbazones is open to speculation. The sulfur analogs do appear to interact strongly with aryl groups which does not appear to be the case with semicarbazones. Presumably the difference in electronegativity of sulfur *versus* oxygen can account for the more extensive delocalization in the sulfur case. The resulting lower electron density on the sulfur atom makes certain thiosemicarbazones less susceptible to oxidation. Therefore, the thiosemicarbazone is not as easily reduced by metal ions and forms more stable chelates. In semicarbazones the smaller, more electronegative oxygen atom does not allow as stable a chelate to form as in the sulfur analogs. This hypothesis suggests that the biological activity of thiosemicarbazones is intimately connected with their chelating ability, an accepted fact in many instances [see Palenik, Rendle & Carter (1974) for a more extensive discussion].

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The Crystal and Molecular Structure of Δ^6-6-Azidobetamethasone-21-acetate

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Crystals of the title steroid are monoclinic with $a=8{\cdot}065$ (2), $b=11{\cdot}173$ (2), $c=15{\cdot}337$ (3) Å, $\beta=102{\cdot}67$ (3)°, $Z=2$, space group $P2_1$. The structure was determined by direct methods from diffractometer data and refined by least-squares methods to a final R of 0·042 for 1662 independent reflexions. The B, C and D rings are *trans*-fused. Ring A is planar, and the conformation of rings B, C and D are half-chair, chair and half-chair respectively. The molecules are linked by 'head-to-tail' hydrogen bonds between the C(17) hydroxy group and the C(3) keto group.

Introduction

The Δ^6-6-azido moiety influences the biological activity of the progestagen and corticoid series. Recent articles (Duax, Wolff & Weeks, 1973; Teutsch, Weber, Page, Shapiro, Herzog, Neri & Collins, 1973) emphasize the importance of correlating activity with three-dimensional structure. Data on the synthesis and biological activity of this compound are given by Green *et al.* (1974).

Experimental

The crystals as supplied by Dr M. J. Green of the Schering corporation had been grown by slow evaporation of an acetone solution. This procedure gave crystals containing one molecule of acetone per molecule of steroid (as determined by n.m.r. spectroscopy). Microanalysis yielded the following results.

	%C	%H	%N
Found	61·09	6·42	7·71
Calculated for $C_{24}H_{28}FO_6N_3.CH_3COCH_3$	61·00	6·45	7·91

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* External Staff, Medical Research Council.

The crystals are prismatic needles elongated along **a**. A single crystal (0·3 × 0·3 × 0·6 mm) was used. The lattice constants were obtained from a least-squares analysis of the settings of 25 reflexions measured on a four circle diffractometer with Mo $K\alpha$ radiation.

Crystal data

Molecular formula $C_{24}H_{28}FO_6N_3.CH_3COCH_3$
$M = 531·64$
Space group $P2_1$
$a = 8·065\ (2)$ Å
$b = 11·173\ (2)$
$c = 15·337\ (3)$
$\beta = 102·67\ (3)°$
$V = 1348·35$ Å^3
$D_m = 1·28$ g cm^{-3}
$D_c = 1·309$ g cm^{-3} for $Z = 2$
$\mu = 0·61$ cm^{-1}
$F(000) = 564$.

The intensities were collected on a Philips PW 1100 computer-controlled four-circle diffractometer operating in the ω-scan mode (scan width = 0·9°, scan speed 0·03° s^{-1}). With Zr-filtered Mo $K\alpha$ radiation ($\lambda = 0·7107$ Å), 1759 reflexions up to $2\theta = 42°$ were measured. With the criterion $I_{\rm rel} > 1·65\sigma\ (I_{\rm rel})$ for an observed reflexion, 97 reflexions were omitted as unobserved leaving 1662 unique reflexions which were employed in the structural analysis. Lorentz and polarization corrections were applied and structure amplitudes and $|E|$ values derived. No correction was made for absorption.

Solution and refinement

The structure was solved by weighted multisolution tangent refinement (Germain, Main & Woolfson, 1971). Difficulty was experienced in selecting a suitable starting set. This was eventually resolved by finding the set which gave the most rapid phase expansion in a 'pseudotangent refinement' (Roberts, Pettersen, Sheldrick, Isaacs & Kennard, 1973) and led to the starting set of reflexions given in Table 1. The first three reflexions were used to define the origin and the fourth to fix the enantiomorph.

Table 1. *Reflexions used in the starting set with allowed phases*

h	k	l	φ (rad)
−1	0	7	0
−1	1	8	0
−1	1	1	$\pi/4$, $3\pi/4$
−3	2	3	$\pi/4$, $3\pi/4$
3	5	0	$\pi/4$, $3\pi/4$, $5\pi/4$, $7\pi/4$
1	3	0	$3\pi/4$

An index $R_\alpha = [\sum_h g(\alpha - k\langle\alpha^2\rangle^{1/2})^2/\sum_h g\alpha^2]^{1/2}$ was calculated after 8 cycles of weighted tangent refinement for each of 64 phase permutations. E maps were calculated for the solutions having low values of R_α, and the resulting peak coordinates used to calculate E values and a reliability index $R_A = [\sum(E_{\rm obs} - kE_{\rm calc})^2/\sum(E_{\rm obs})^2]^{1/2}$, where k was chosen to minimize R_A. The best solution had $R_\alpha = 0·248$ and $R_A = 0·294$. The resulting E map yielded the complete steroid molecule but not the acetone of crystallization. Three cycles of full-matrix least-squares refinement and a subsequent difference map showed the positions of the acetone molecule. The final refinement was carried out with anisotropic temperature factors for all the heavy atoms. The hydrogen atoms were constrained to be 1·08 Å from their corresponding carbon atoms, their position being dictated by the geometry of the molecule. The hydrogen atoms in the methyl groups were refined as rigid groups and the hydroxyl hydrogens H(O2) and H(O3), whose positions could not be predicted, were located by difference maps and thereafter constrained to ride on the oxygen atoms. The isotropic temperature factors of all the hydrogen atoms were refined as one common parameter. This technique of constrained least-squares refinement, using rigid groups, bond length constraints and location and refinement of hydrogen atoms is discussed by Sheldrick (1974).

The refinement converged to $R_w = \sum w^{1/2}|F_o - F_c|/\sum w^{1/2}|F_o| = 0·046$ and a corresponding unweighted R of 0·042.*

Table 2. *Analysis of variance*

(*a*) As a function of sin θ

sin θ	0·00–0·17	0·17–0·22	0·22–0·25	0·25–0·28	0·28–0·30	0·30–0·32	0·32–0·33	0·33–0·35	0·35–0·37	0·37–0·38
N	171	187	167	206	153	190	96	209	234	49
V	73	67	55	48	45	49	45	43	43	40

(*b*) As a function of $\sqrt{(F/F_{\rm max})}$

$\sqrt{(F/F_{\rm max})}$	0·00–0·21	0·21–0·23	0·23–0·26	0·26–0·29	0·29–0·31	0·31–0·34	0·34–0·38	0·38–0·42	0·42–0·50	0·50–1·00
N	220	117	172	197	153	146	188	154	163	152
V	51	47	49	54	52	58	58	54	53	46

(*c*) By parity groups

	ggg	*ugg*	*gug*	*uug*	*ggu*	*ugu*	*guu*	*uuu*	All
N	220	220	193	200	213	225	194	197	1662
V	49	56	60	50	49	53	50	52	53

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30517 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2 shows an analysis of variance computed after the final cycle. As a final check of the correctness of the structure a difference map was computed. This had no peaks $>0{\cdot}2$ e Å^{-3}.

Tables 3, 4 and 5 show the final atomic coordinates and temperature factors. The isotropic temperature factor of the H atoms refined to a value $U=0{\cdot}053$ Å^2.

Description of the structure and discussion

The predicted chemical structure (Schering Corporation) is shown in Fig. 1, and a perspective view of the molecule showing the numbering scheme in Fig .2. The fluorine atom at C(9) and the hydroxyl group at C(17) are in the α-position while the hydroxyl group at C(11) and the methyl group at C(16) are in the β-position. The bond lengths C(1)–C(2) (1·339 Å), C(4)–C(5) (1·377 Å) and C(6)–C(7) (1·326 Å) are all in good agreement with accepted values for a C(sp^2)–C(sp^2) bond length (Kennard, 1962). The structure is thus in complete agreement with the predicted chemical structure shown in Fig. 1. The geometry of the azide group at C(6) is similar to that found in other compounds. Using a text-searching program developed by Dr S. Motherwell we have scanned the bibliographic tapes of the Cambridge Crystallographic Data Centre and found four other compounds containing azide groups bonded to carbon rings.

Table 3. *Fractional atomic coordinates of the heavy atoms* (× 10^4)

	x/a	y/b	z/c
C(1)	−5721 (5)	−4691 (5)	−9153 (3)
C(2)	−7232 (5)	−4649 (5)	−9728 (3)
C(3)	−7406 (6)	−4247 (5)	−10642 (3)
C(4)	−5891 (5)	−3839 (5)	−10915 (3)
C(5)	−4335 (5)	−3823 (4)	−10325 (3)
C(6)	−2860 (5)	−3288 (4)	−10559 (3)
C(7)	−1459 (5)	−3046 (5)	−9951 (3)
C(8)	−1303 (5)	−3252 (4)	−8967 (3)
C(9)	−3043 (5)	−3451 (4)	−8752 (3)
C(10)	−4098 (5)	−4366 (4)	−9417 (3)
C(11)	−2992 (5)	−3619 (4)	−7763 (3)
C(12)	−1897 (5)	−2650 (4)	−7170 (2)
C(13)	−157 (5)	−2456 (4)	−7391 (2)
C(14)	−398 (5)	−2219 (4)	−8399 (2)
C(15)	1364 (6)	−1809 (5)	−8497 (3)
C(16)	1969 (5)	−987 (5)	−7675 (3)
C(17)	764 (5)	−1263 (4)	−7023 (3)
C(18)	1016 (5)	−3530 (5)	−7091 (3)
C(19)	−3122 (6)	−5579 (5)	−9438 (3)
C(20)	1557 (5)	−1342 (4)	−6033 (3)
C(21)	553 (6)	−844 (5)	−5399 (3)
C(22)	1097 (7)	−2334 (5)	−4304 (3)
C(23)	2066 (9)	−2652 (6)	−3367 (3)
C(24)	3901 (5)	−1024 (6)	−7328 (3)
F(1)	−3949 (3)	−2356	−8952 (1)
N(1)	−3098 (5)	−3019 (5)	−11499 (2)
N(2)	−1989 (6)	−2362 (5)	−11697 (3)
N(3)	−1073 (8)	−1774 (7)	−11945 (4)
O(1)	−8808 (4)	−4239 (4)	−11180 (2)
O(2)	−2397 (4)	−4800 (4)	−7504 (2)
O(3)	−462 (3)	−308 (3)	−7075 (2)
O(4)	2925 (4)	−1827 (4)	−5747 (2)
O(5)	1293 (4)	−1178 (4)	−4494 (2)
O(6)	245 (7)	−3002 (5)	−4820 (3)
C(25)	−5487 (9)	−5304 (10)	−5999 (6)
C(26)	−4628 (13)	−4264 (10)	−5445 (7)
C(27)	−6698 (10)	−6045 (13)	−5667 (7)
O(7)	−5181 (6)	−5484 (6)	−6732 (4)

Table 4. *Anisotropic temperature factors of the heavy atoms* (Å^2 × 10^3)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	46 (3)	47 (3)	48 (3)	−10 (2)	8 (2)	−12 (3)
C(2)	42 (3)	52 (3)	47 (3)	−16 (2)	10 (2)	−7 (2)
C(3)	45 (3)	48 (3)	57 (3)	−22 (3)	1 (3)	−6 (3)
C(4)	45 (3)	46 (3)	43 (3)	−12 (2)	2 (2)	0 (2)
C(5)	39 (3)	35 (3)	37 (3)	−6 (2)	3 (2)	3 (2)
C(6)	56 (3)	43 (3)	27 (2)	−6 (2)	12 (2)	1 (3)
C(7)	37 (3)	47 (3)	42 (3)	−6 (2)	12 (2)	0 (2)
C(8)	33 (2)	39 (3)	30 (2)	−3 (2)	10 (2)	1 (2)
C(9)	32 (2)	26 (2)	37 (2)	−1 (2)	4 (2)	4 (2)
C(10)	41 (3)	36 (3)	34 (3)	−4 (2)	8 (2)	0 (2)
C(11)	43 (3)	36 (3)	34 (2)	0 (2)	12 (2)	−2 (2)
C(12)	36 (2)	36 (3)	32 (2)	−5 (2)	8 (2)	−3 (2)
C(13)	32 (2)	33 (2)	29 (2)	0 (2)	6 (2)	3 (2)
C(14)	30 (2)	40 (3)	32 (2)	2 (2)	6 (2)	−1 (2)
C(15)	48 (3)	59 (3)	48 (3)	−4 (3)	19 (2)	−11 (3)
C(16)	34 (2)	42 (3)	42 (3)	3 (2)	5 (2)	−3 (2)
C(17)	29 (2)	32 (2)	41 (2)	0 (2)	6 (2)	5 (2)
C(18)	45 (3)	36 (3)	46 (3)	−2 (2)	−1 (2)	11 (2)
C(19)	65 (3)	33 (3)	52 (3)	−6 (2)	14 (2)	4 (2)
C(20)	38 (2)	30 (2)	40 (2)	2 (2)	0 (2)	0 (2)
C(21)	59 (3)	43 (3)	37 (3)	2 (2)	5 (2)	3 (2)
C(22)	90 (4)	49 (4)	45 (3)	3 (3)	12 (3)	−25 (3)
C(23)	138 (5)	64 (4)	39 (3)	12 (3)	−5 (3)	−27 (4)
C(24)	33 (2)	83 (4)	58 (3)	−4 (3)	10 (2)	−7 (3)
F(1)	33 (1)	35 (1)	45 (1)	−2 (1)	3 (1)	7 (1)
N(1)	69 (3)	75 (3)	33 (2)	3 (2)	7 (2)	−15 (3)
N(2)	71 (3)	79 (3)	43 (3)	15 (3)	12 (2)	−2 (3)
N(3)	93 (4)	133 (5)	89 (4)	41 (4)	28 (3)	−26 (4)
O(1)	48 (2)	84 (3)	64 (2)	−25 (2)	−11 (2)	−2 (2)
O(2)	64 (2)	37 (2)	48 (2)	6 (2)	10 (2)	−4 (2)
O(3)	42 (2)	30 (2)	47 (2)	2 (1)	5 (1)	12 (1)
O(4)	47 (2)	66 (2)	50 (2)	3 (2)	−7 (2)	12 (2)
O(5)	83 (2)	40 (2)	36 (2)	−3 (2)	10 (2)	−12 (2)
O(6)	151 (4)	63 (3)	65 (3)	4 (2)	−11 (3)	−56 (3)
C(25)	75 (4)	155 (9)	105 (6)	73 (6)	37 (4)	58 (5)
C(26)	133 (7)	114 (8)	146 (8)	14 (7)	11 (6)	58 (7)
C(27)	92 (5)	236 (13)	161 (8)	122 (9)	45 (5)	30 (7)
O(7)	88 (3)	149 (5)	115 (4)	39 (4)	43 (3)	15 (3)

The relevant bond lengths and angles of the azide groups in these compounds are listed in Table 6.

The principal bond lengths and angles are shown in Tables 7 and 8. The conformation of the molecule can be seen on examination of the torsion angles shown in Table 9. Rings *B*, *C* and *D* are *trans*-fused. Ring *A* is nearly planar as shown by the torsion angles and the calculations of least-squares planes which are listed in Table 10. Ring *B* has a half-chair conformation while ring *C* is a chair. Ring *D* is a half-chair with a phase angle Δ of $-3{\cdot}1°$ and φ_m of $48{\cdot}7°$ (Altona, Geise & Romers, 1968). There are two hydrogen bonds: O(2)–H(O2)$\cdots$O(7) links the steroid to the acetone molecule of crystallization, while O(3)–H(O3)$\cdots$O(1) links one steroid molecule to another related by the 2_1 symmetry axis. The lengths and angles of the hydrogen bonds are given in Table 11. Fig. 3 shows a packing diagram of the structure, the hydrogen bonds being indicated by dotted lines.

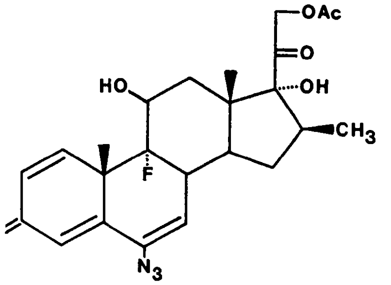

Fig. 1. Structural formula of the compound.

Table 5. *Fractional atomic coordinates of the hydrogen atoms* (× 10^3)

	x/a	*y/b*	*z/c*
H(1)	−567	−497	−847
H(2)	−835	−492	−950
H(4)	−599	−354	−1159
H(7)	−38	−268	−1017
H(8)	−54	−405	−880
H(11)	−427	−351	−766
H(121)	−171	−292	−648
H(122)	−258	−181	−726
H(14)	−129	−152	−867
H(151)	221	−256	−848
H(152)	128	−132	−911
H(16)	184	−4	−782
H(181)	225	−331	−721
H(182)	59	−437	−741
H(183)	110	−361	−638
H(191)	−201	−536	−969
H(192)	−390	−620	−989
H(193)	−273	−598	−878
H(211)	−73	−119	−558
H(212)	53	12	−545
H(231)	207	−361	−329
H(232)	148	−224	−287
H(233)	336	−234	−328
H(241)	427	−37	−681
H(242)	446	−80	−789
H(243)	434	−190	−708
H(O2)	−314	−484	−701
H(O3)	−73	11	−769
H(261)	−462	−433	−474
H(262)	−538	−350	−573
H(263)	−334	−416	−553
H(271)	−664	−559	−504
H(272)	−659	−700	−555
H(273)	−790	−585	−612

Table 7. *Bond lengths* (Å)

C(1)—C(2)	1·339 (6)	C(11)–O(2)	1·431 (5)
C(1)—C(10)	1·496 (6)	C(12)–C(13)	1·529 (5)
C(2)—C(3)	1·449 (7)	C(13)–C(14)	1·538 (5)
C(3)—C(4)	1·449 (6)	C(13)–C(17)	1·569 (6)
C(3)—O(1)	1·245 (5)	C(13)–C(18)	1·535 (6)
C(4)—C(5)	1·377 (6)	C(14)–C(15)	1·532 (6)
C(5)—C(6)	1·445 (5)	C(15)–C(16)	1·550 (6)
C(5)—C(10)	1·493 (6)	C(16)–C(17)	1·572 (6)
C(6)—N(1)	1·444 (6)	C(16)–C(24)	1·532 (5)
N(1)—N(2)	1·245 (7)	C(17)–C(20)	1·515 (6)
N(2)—N(3)	1·115 (7)	C(17)–O(3)	1·445 (5)
C(6)—C(7)	1·326 (6)	C(20)–C(21)	1·502 (7)
C(7)—C(8)	1·505 (5)	C(20)–O(4)	1·221 (5)
C(8)—C(9)	1·527 (5)	C(21)–O(5)	1·435 (5)
C(8)—C(14)	1·530 (6)	C(22)–O(5)	1·341 (6)
C(9)—C(10)	1·558 (6)	C(22)–C(23)	1·520 (7)
C(9)—C(11)	1·521 (5)	C(22)–O(6)	1·191 (6)
C(9)—F(1)	1·424 (4)	O(7)—C(25)	1·219 (8)
C(10)–C(19)	1·571 (6)	C(25)–C(26)	1·514 (13)
C(11)–C(12)	1·558 (6)	C(25)–C(27)	1·455 (12)

Table 8. *Bond angles* (°)

C(10)–C(1)—C(2)	122·5 (4)	C(14)–C(13)–C(12)	109·1 (3)
C(3)—C(2)—C(1)	121·9 (4)	C(17)–C(13)–C(12)	115·2 (3)
C(4)—C(3)—C(2)	117·9 (4)	C(17)–C(13)–C(14)	99·3 (3)
O(1)—C(3)—C(2)	121·7 (4)	C(18)–C(13)–C(12)	111·0 (3)
O(1)—C(3)—C(4)	120·4 (5)	C(18)–C(13)–C(14)	111·7 (3)
C(5)—C(4)—C(3)	121·3 (4)	C(18)–C(13)–C(17)	110·1 (3)
C(6)—C(5)—C(4)	121·6 (4)	C(15)–C(14)–C(8)	120·4 (3)
C(10)–C(5)—C(4)	121·3 (4)	C(15)–C(14)–C(13)	103·6 (3)
C(10)–C(5)—C(6)	117·1 (3)	C(13)–C(14)–C(8)	112·4 (3)
C(7)—C(6)—C(5)	122·1 (4)	C(16)–C(15)–C(14)	103·3 (3)
N(1)—C(6)—C(5)	113·4 (4)	C(17)–C(16)–C(15)	105·8 (3)
N(1)—C(6)—C(7)	124·5 (4)	C(24)–C(16)–C(15)	112·4 (4)
C(8)—C(7)—C(6)	123·0 (4)	C(24)–C(16)–C(17)	119·8 (3)
C(9)—C(8)—C(7)	111·1 (3)	O(3)—C(17)–C(20)	103·4 (3)
C(14)–C(8)—C(7)	112·3 (3)	O(3)—C(17)–C(16)	109·3 (3)
C(14)–C(8)—C(9)	109·5 (3)	O(3)—C(17)–C(13)	110·1 (3)
C(10)–C(9)—C(8)	110·5 (3)	C(20)–C(17)–C(16)	117·7 (3)
C(11)–C(9)—C(8)	114·1 (3)	C(20)–C(17)–C(13)	112·0 (3)
C(11)–C(9)—C(10)	117·6 (3)	C(16)–C(17)–C(13)	104·4 (3)
		C(21)–C(20)–C(17)	117·3 (4)
F(1)—C(9)—C(8)	106·4 (3)	O(4)—C(20)–C(17)	122·3 (4)
F(1)—C(9)—C(10)	104·1 (3)	O(4)—C(20)–C(21)	120·4 (4)
F(1)—C(9)—C(11)	102·7 (3)	O(5)—C(21)–C(20)	111·2 (4)
C(5)—C(10)–C(1)	114·2 (3)	C(22)–O(5)—C(21)	114·6 (4)
C(9)—C(10)–C(1)	111·5 (3)	O(6)—C(22)–O(5)	122·5 (5)
C(9)—C(10)–C(5)	106·4 (3)	O(6)—C(22)–C(23)	125·8 (5)
C(19)–C(10)–C(1)	105·6 (4)	O(5)—C(22)–C(23)	111·7 (5)
C(19)–C(10)–C(5)	107·1 (3)	N(2)—N(1)—C(6)	114·9 (4)
C(19)–C(10)–C(9)	112·0 (3)	N(3)—N(2)—N(1)	174·2 (5)
C(12)–C(11)–C(9)	112·6 (3)		
O(2)—C(11)–C(9)	109·0 (3)	C(26)–C(25)–O(7)	118·9 (8)
O(2)—C(11)–C(12)	111·5 (3)	C(27)–C(25)–O(7)	121·0 (11)
C(13)–C(12)–C(11)	113·5 (3)	C(27)–C(25)–C(26)	120·0 (10)

Table 6. *Geometry of azide groups attached to carbon rings* C–N(1)–N(2)–N(3)

Compound	*a*	*b*	*c*	*d*	*e*
C——N(1) (Å)	1·444	1·417	1·46	1·426	1·50
N(1)–N(2) (Å)	1·245	1·270	1·24	1·248	1·14
N(2)–N(3) (Å)	1·115	1·127	1·12	1·122	1·15
C——N(1)–N(2) (°)	114·9	115·0	119·2	116	120·9
N(1)–N(2)–N(3) (°)	174·2	173·4	168·3	172	174·0

(*a*) Present work.
(*b*) *p*-Nitrophenylazide (Mugnoli, Mariani & Simonetta, 1965).
(*c*) Picryl azide–bis-(8-hydroxyquinolinato)copper(II) (Bailey & Prout, 1965).
(*d*) 3-Azidotropolone (Cruickshank, Filippini & Mills, 1972).
(*e*) 14*β*-Azido-5*α*-androstane (Chiaroni, Riche & Pascard-Billy, 1974).

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Table 9. *Torsion angles* (°)

Ring *A*		Ring *C*	
C(2)—C(1)-C(10)-C(5)	8·7	C(14)-C(8)—C(9)—C(11)	−53·2
C(10)-C(1)-C(2)—C(3)	−2·1	C(8)—C(9)—C(11)-C(12)	48·1
C(1)—C(2)-C(3)—C(4)	−2·4	C(9)—C(11)-C(12)-C(13)	−48·0
C(2)—C(3)-C(4)—C(5)	0·0	C(11)-C(12)-C(13)-C(14)	52·4
C(3)—C(4)-C(5)—C(10)	7·9	C(12)-C(13)-C(14)-C(8)	−58·9
C(4)—C(5)-C(10)-C(1)	−11·5	C(9)—C(8)—C(14)-C(13)	59·0
Ring *B*		Ring *D*	
C(6)—C(5)-C(10)-C(9)	45·4	C(17)-C(13)-C(14)-C(15)	48·7
C(10)-C(5)-C(6)—C(7)	−14·5	C(13)-C(14)-C(15)-C(16)	−40·6
C(5)—C(6)-C(7)—C(8)	−2·8	C(14)-C(15)-C(16)-C(17)	15·6
C(6)—C(7)-C(8)—C(9)	−14·7	C(15)-C(16)-C(17)-C(13)	14·2
C(7)—C(8)-C(9)—C(10)	47·1	C(14)-C(13)-C(17)-C(16)	−37·9
C(8)—C(9)-C(10)-C(5)	−61·9		

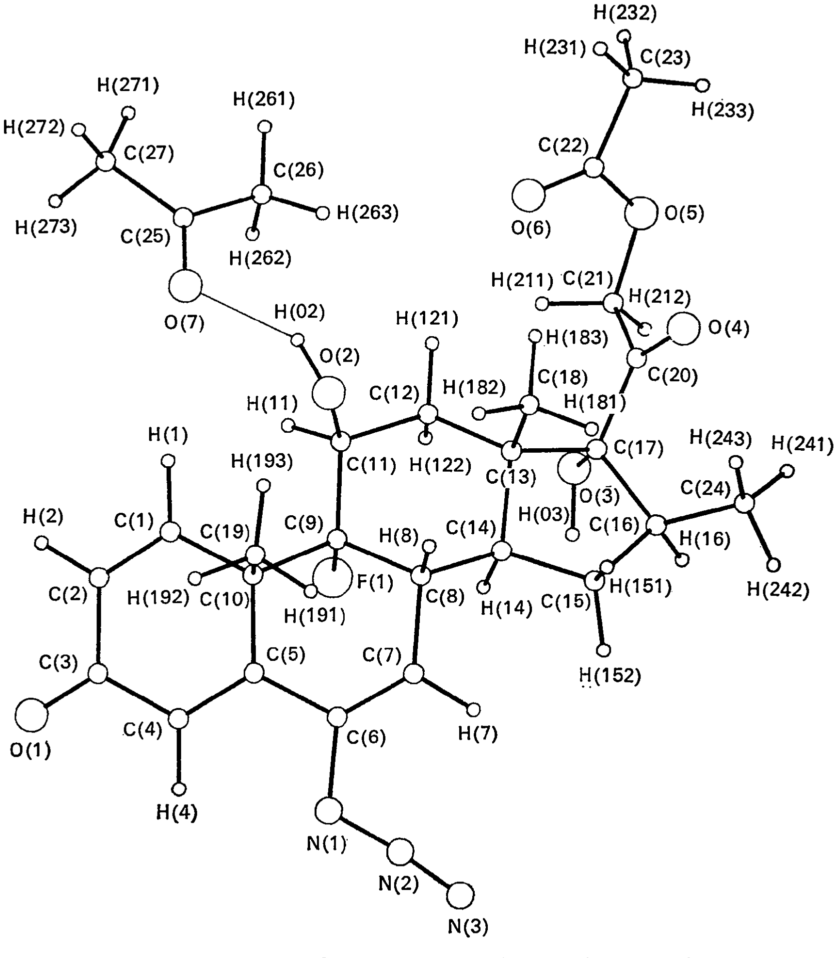

Fig. 2. Molecular structure of the compound with atomic nomenclature.

Table 10. *Least-squares planes calculation*

The equations of the planes are expressed in orthogonalized space as $lX+mY+nZ=P$. '*d*' is the perpendicular distance (Å) from the plane. Atoms marked (*) were not included in the least-squares plane calculation.

Plane 1 (Ring *A*)

$-0{\cdot}1668X+0{\cdot}9267Y+0{\cdot}3367Z=-8{\cdot}4754$

	d		*d*
C(1)	−0·016	C(5)	0·013
C(2)	0·018	C(10)*	−0·126
C(3)	−0·004	O(1)*	−0·006
C(4)	−0·012		

Plane 2 (Ring *A*)

$-0{\cdot}1396X+0{\cdot}9261Y+0{\cdot}3506Z=8{\cdot}8170$

	d		*d*
C(1)	0·025	C(5)	0·055
C(2)	0·019	C(10)	−0·060
C(3)	−0·027	O(1)*	−0·066
C(4)	−0·012		

Plane 3 (Ring *B*)

$-0{\cdot}4303X+0{\cdot}8933Y+0{\cdot}1296Z=-4{\cdot}4212$

	d		*d*
C(5)	0·121	C(9)	0·338
C(6)	0·074	C(10)	−0·327
C(7)	−0·070	N(1)*	0·243
C(8)	−0·136		

Plane 4 (Ring *C*)

$-0{\cdot}5189X+0{\cdot}8450Y-0{\cdot}1293Z=-0{\cdot}5473$

	d		*d*
C(8)	−0·243	C(12)	0·198
C(9)	0·198	C(13)	−0·246
C(11)	−0·177	C(14)	0·270

Plane 5 (Ring *D*)

$0{\cdot}6448X-0{\cdot}7434Y+0{\cdot}1776Z=-0{\cdot}3355$

	d		*d*
C(13)	0·288	C(16)	0·002
C(14)	−0·298	C(17)	−0·064
C(15)	0·173		

Table 11. *Hydrogen bonds*

O(2)—H(O2)	1·069 Å	O(3)-H(O3)	1·026 Å
H(O2)-O(7)	1·928	O(1)-H(O3)	1·847
O(2)—O(7)	2·862	O(1)-O(3)	2·872
O(2)—H(O2)-O(7)	144·0°	O(3)-H(O3)-O(1)	176·3°

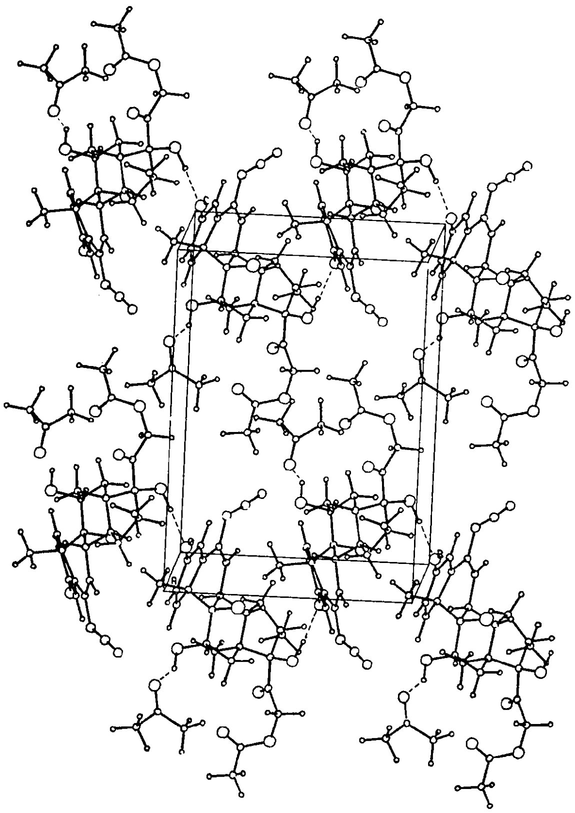

Fig. 3. Molecular packing showing the hydrogen bonding.

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