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17α-Phenyl-5-androstene-3β,17β-diol–Cyclohexane (2:1), $C_{25}H_{34}O_2.\frac{1}{2}C_6H_{12}$

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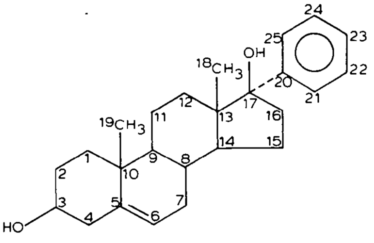
Abstract. $M_r = 408\cdot6$, orthorhombic, $P2_12_12_1$, $a = 18\cdot1460$ (8), $b = 21\cdot933$ (1), $c = 12\cdot4877$ (6) Å, $V = 4970\cdot1$ Å^3, $Z = 8$, $D_m = 1\cdot11$, $D_x = 1\cdot09$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5\cdot16$ cm^{-1}, $F(000) = 1792$, $T = 293$ K. Final $R = 4\cdot8\%$ for 3748 unique observed reflections. The title compound was prepared by reaction of phenyllithium with 3β-hydroxy-5-androsten-17-one and proven to be the α epimer. The two crystallographically independent molecules have normal 5-ene geometry.

Introduction. The crystal structure of the title compound (17-PAD) was determined in order to confirm the configuration at C(17). The presence of a rigid, axial phenyl moiety at this position provides a useful derivative for probing the steroid-binding sites of certain proteins.



Experimental. Bromobenzene (Aldrich) in six volumes of diethyl ether was treated with one equivalent of *n*-butyllithium (Aldrich) under nitrogen. After stirring for 5 min at room temperature, 3β-hydroxy-5-androsten-17-one (as the 3-acetate) (Steraloids), 0·33 equivalents, was added in ether–benzene. Mixing was continued for 30 min as a white solid precipitated from solution. Ice was added to decompose excess reactant, and the solid was collected by filtration and crystallized from ethanol–water to yield colorless needles with a sharp m.p. 452–453 K (uncorrected).

D_m measured by flotation. Crystal size 0·4 × 0·6 × 0·6 mm. Syntex $P3$ diffractometer. Lattice parameters determined using 25 reflections in range $60 < 2\theta < 65°$. Systematic absences $h00$ for $h = 2n + 1$, $0k0$ for $k = 2n + 1$, and $00l$ for $l = 2n + 1$. No absorption correction. Intensity measurements in range $4 < 2\theta < 116°$ ($0 \leq h \leq 19$, $0 \leq k \leq 23$, $0 \leq l \leq 13$). Four intensity standards [080 1108 (13); 0,12,0 9664 (102); 0,0,10 1583 (16); 7,10,4 989 (12)]. 3806 unique reflections measured, 58 unobserved reflections ($F < 2\sigma_F$). Solution by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Least-squares refinement using F magnitudes. Steroid H atoms located on difference maps. Positional parameters for all atoms except cyclohexane H atoms and anisotropic thermal parameters for the two steroid molecules refined. Weights ($1/\sigma_F^2$) with σ_F defined by Stout & Jensen (1968) and instability correction 0·06. Using observed data, final $R = 4\cdot8$, $wR = 6\cdot4\%$, $S = 1\cdot97$. $(\Delta/\sigma)_{\max} = 0\cdot43$, $(\Delta/\sigma)_{\text{av}} = 0\cdot05$. No secondary extinction correction used. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Computer programs used included Enraf–Nonius (1979) *SDP*, *ORTEP* (Johnson, 1965), and various in-house programs for data reduction and geometrical analysis.

Discussion. The crystal-structure determination of 17-PAD proves that C(17) in the product has the α configuration. The detection of only the α-phenyl substituted epimer is consistent with the expected aryllithium attack of the 17-carbonyl from the less hindered α face of the molecule. Preliminary biochemical studies with 17-PAD have shown binding to adrenocortical cholesterol side-chain cleavage cytochrome P-450 indicating that the steroid-binding site of this enzyme will accept the axial phenyl substituent.

The atomic positional and isotropic thermal parameters are given in Table 1,* and Fig. 1 illustrates

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* Lists of structure factors, anisotropic thermal parameters and the unrefined cyclohexane H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38767 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates* ($\times 10^4$; $\times 10^3$ *for* H) *and equivalent isotropic thermal parameters for the non*-H *atoms* ($\times 10$) *with e.s.d.'s in parentheses*

The cyclohexane C atoms were each assigned fixed B_{iso} values of 32 Å^2 during the final refinement cycle. H atoms were assigned B_{iso} values equal to the equivalent B_{iso} values for the non-H atoms to which they were bonded. The cyclohexane H atoms (indicated by *T*) were placed at their geometrically expected positions and their coordinates have been deposited.

$$B_{\text{eq}} = \tfrac{8}{3}\pi^2 (U_{11} + U_{22} + U_{33}).$$

	x	y	z	B_{eq} (Å^2)
C(1)	1161 (2)	5087 (1)	1195 (3)	49 (1)
C(2)	1289 (2)	5721 (1)	728 (3)	52 (1)
C(3)	812 (2)	5821 (1)	−241 (2)	46 (1)
C(4)	3 (2)	5709 (1)	1 (2)	45 (1)
C(5)	−122 (1)	5096 (1)	522 (2)	39 (1)
C(6)	−628 (2)	4714 (1)	159 (2)	47 (1)
C(7)	−817 (2)	4110 (1)	648 (2)	43 (1)
C(8)	−525 (1)	4052 (1)	1786 (2)	35 (1)
C(9)	287 (1)	4272 (1)	1813 (2)	33 (1)
C(10)	350 (1)	4956 (1)	1501 (2)	37 (1)
C(11)	678 (1)	4120 (1)	2873 (2)	37 (1)
C(12)	605 (1)	3453 (1)	3182 (2)	36 (1)
C(13)	−205 (1)	3269 (1)	3248 (2)	34 (1)
C(14)	−563 (1)	3390 (1)	2156 (2)	33 (1)
C(15)	−1321 (1)	3079 (1)	2240 (2)	42 (1)
C(16)	−1193 (1)	2543 (1)	3013 (2)	43 (1)
C(17)	−379 (1)	2577 (1)	3385 (2)	37 (1)
C(18)	−593 (2)	3626 (1)	4147 (2)	42 (1)
C(19)	111 (2)	5372 (1)	2431 (3)	52 (1)
C(20)	129 (1)	2180 (1)	2720 (2)	36 (1)
C(21)	−39 (2)	1981 (1)	1677 (2)	43 (1)
C(22)	454 (2)	1644 (1)	1085 (2)	52 (1)
C(23)	1124 (2)	1494 (1)	1490 (3)	54 (1)
C(24)	1309 (2)	1675 (1)	2514 (3)	53 (1)
C(25)	817 (2)	2011 (1)	3117 (3)	44 (1)
O(3*B*)	929 (1)	6437 (1)	−593 (2)	51 (1)
O(17*B*)	−292 (1)	2422 (1)	4500 (1)	47 (1)
C(1′)	3230 (2)	2607 (1)	10911 (2)	42 (1)
C(2′)	3734 (2)	2681 (1)	11883 (2)	45 (1)
C(3′)	4284 (1)	3173 (1)	11680 (2)	43 (1)
C(4′)	3898 (2)	3766 (2)	11460 (2)	49 (1)
C(5′)	3360 (1)	3722 (1)	10540 (2)	41 (1)
C(6′)	3362 (2)	4127 (1)	9759 (2)	48 (1)
C(7′)	2844 (2)	4132 (1)	8831 (2)	48 (1)
C(8′)	2205 (2)	3688 (1)	8951 (2)	39 (1)
C(9′)	2469 (1)	3096 (1)	9461 (2)	36 (1)
C(10′)	2817 (1)	3193 (1)	10592 (2)	36 (1)
C(11′)	1888 (2)	2588 (1)	9447 (2)	44 (1)
C(12′)	1558 (2)	2487 (1)	8333 (2)	42 (1)
C(13′)	1236 (1)	3077 (1)	7882 (2)	39 (1)
C(14′)	1853 (1)	3555 (1)	7862 (2)	38 (1)
C(15′)	1533 (2)	4077 (1)	7205 (3)	50 (1)
C(16′)	986 (2)	3769 (1)	6434 (3)	51 (1)
C(17′)	1005 (1)	3076 (1)	6674 (2)	39 (1)
C(18′)	581 (2)	3290 (2)	8569 (3)	51 (1)
C(19′)	2224 (2)	3335 (2)	11430 (2)	49 (1)
C(20′)	1534 (1)	2722 (1)	5972 (2)	42 (1)
C(21′)	2082 (2)	2998 (2)	5356 (2)	52 (1)
C(22′)	2555 (2)	2653 (3)	4733 (2)	67 (1)
C(23′)	2506 (2)	2037 (2)	4705 (3)	68 (1)
C(24′)	1973 (2)	1752 (2)	5307 (3)	63 (1)
C(25′)	1490 (2)	2085 (1)	5923 (2)	49 (1)
O(3*B*′)	4782 (1)	3263 (1)	12565 (2)	54 (1)
O(17*B*′)	280 (1)	2816 (1)	6561 (2)	48 (1)
C(26)	2906 (9)	382 (9)	3057 (30)	320
C(27)	3674 (11)	497 (5)	3130 (17)	320
C(28)	4078 (9)	−44 (6)	3597 (22)	320
C(29)	3755 (10)	−536 (6)	3399 (21)	320
C(30)	3045 (9)	−579 (9)	3415 (31)	320
C(31)	2699 (9)	−139 (11)	2661 (22)	320
H(1*A*)	136 (2)	476 (1)	65 (3)	
H(1*B*)	142 (2)	505 (1)	190 (3)	
H(2*A*)	179 (2)	581 (1)	45 (3)	
H(2*B*)	110 (2)	599 (2)	124 (3)	
H(3*A*)	106 (2)	554 (1)	−82 (3)	
H(3O)	67 (2)	649 (2)	−108 (3)	
H(4*A*)	−28 (2)	570 (1)	−76 (3)	
H(4*B*)	−14 (2)	605 (1)	49 (3)	
H(6)	−90 (2)	483 (1)	−47 (3)	
H(7*A*)	−59 (2)	373 (1)	23 (2)	
H(7*B*)	−134 (2)	405 (1)	66 (3)	
H(8*B*)	−87 (1)	431 (1)	224 (2)	
H(9*A*)	54 (1)	404 (1)	124 (2)	
H(11*A*)	118 (2)	424 (1)	273 (2)	
H(11*B*)	53 (2)	437 (1)	339 (3)	
H(12*A*)	87 (1)	318 (1)	260 (2)	
H(12*B*)	88 (1)	336 (1)	393 (2)	
H(14*A*)	−24 (1)	313 (1)	155 (2)	
H(15*A*)	−153 (2)	295 (1)	157 (3)	
H(15*B*)	−165 (2)	334 (1)	258 (2)	
H(16*A*)	−126 (2)	214 (1)	270 (2)	
H(16*B*)	−149 (2)	261 (1)	363 (3)	
H(17O)	−37 (2)	206 (1)	451 (3)	
H(18*A*)	−52 (2)	407 (1)	401 (2)	
H(18*B*)	−103 (2)	349 (1)	426 (3)	
H(18*C*)	−35 (2)	356 (1)	487 (3)	
H(19*A*)	3 (2)	583 (1)	222 (3)	
H(19*B*)	−37 (2)	526 (1)	257 (3)	
H(19*C*)	51 (2)	534 (1)	305 (3)	
H(21)	−56 (2)	213 (1)	131 (2)	
H(22)	23 (2)	148 (1)	31 (3)	
H(23)	150 (2)	127 (1)	110 (3)	
H(24)	173 (2)	158 (1)	276 (3)	
H(25)	89 (2)	207 (1)	379 (3)	
H(1*A*′)	355 (2)	251 (1)	1029 (2)	
H(1*B*′)	287 (2)	229 (1)	1101 (2)	
H(2*A*′)	403 (2)	229 (1)	1200 (2)	
H(2*B*′)	344 (2)	278 (1)	1243 (3)	
H(3*A*′)	454 (2)	305 (1)	1101 (3)	
H(3O′)	494 (2)	290 (1)	1278 (3)	
H(4*A*′)	419 (2)	409 (1)	1134 (3)	
H(4*B*′)	359 (2)	388 (1)	1212 (3)	
H(6′)	371 (2)	450 (1)	978 (2)	
H(7*A*′)	312 (2)	401 (1)	821 (3)	
H(7*B*′)	258 (2)	456 (2)	884 (3)	
H(8*B*′)	184 (2)	387 (1)	938 (2)	
H(9*A*′)	288 (2)	296 (1)	904 (2)	
H(11*A*′)	216 (2)	223 (1)	966 (3)	
H(11*B*′)	150 (2)	274 (1)	997 (3)	
H(12*A*′)	196 (2)	236 (1)	784 (3)	
H(12*B*′)	118 (2)	218 (1)	841 (2)	
H(14*A*′)	226 (2)	333 (1)	743 (2)	
H(15*A*′)	197 (2)	429 (1)	678 (3)	
H(15*B*′)	128 (2)	434 (1)	764 (3)	
H(16*A*′)	107 (2)	386 (1)	573 (3)	
H(16*B*′)	50 (2)	393 (1)	658 (3)	
H(17O′)	18 (2)	275 (1)	598 (3)	
H(18*A*′)	75 (2)	341 (1)	929 (3)	
H(18*B*′)	36 (2)	365 (1)	834 (3)	
H(18*C*′)	27 (2)	298 (1)	863 (3)	
H(19*A*′)	244 (2)	341 (1)	1223 (3)	
H(19*B*′)	189 (2)	369 (1)	1123 (3)	
H(19*C*′)	190 (2)	292 (1)	1144 (3)	
H(21′)	212 (2)	344 (2)	527 (3)	
H(22′)	292 (2)	289 (2)	432 (3)	
H(23′)	284 (2)	176 (2)	429 (3)	
H(24′)	185 (2)	135 (2)	532 (3)	
H(25′)	116 (2)	189 (1)	631 (3)	

the thermal motion of the steroid molecules. Atoms in the second, crystallographically independent, steroid molecule are denoted by primes. The cyclohexane molecule has very high thermal motion, and its C atoms were assigned the fixed average isotropic thermal parameter of 32 Å^2 during the final least-squares refinement cycle. The intramolecular bond distances and angles involving non-H atoms (Table 2) do not deviate significantly from average values observed in similar 5-ene steroids (Duax & Norton, 1975). The

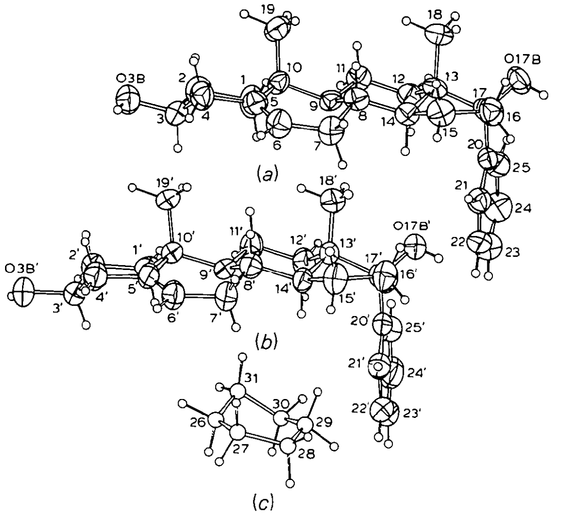

Fig. 1. Perspective views of the molecules in the 17-PAD–cyclohexane complex. The atomic numbering and thermal vibration ellipsoids (scaled to 50% probability) of the non-H atoms in the steroid molecules are illustrated. (*a*) 17-PAD molecule (1), (*b*) 17-PAD molecule (2), and (*c*) the cyclohexane molecule.

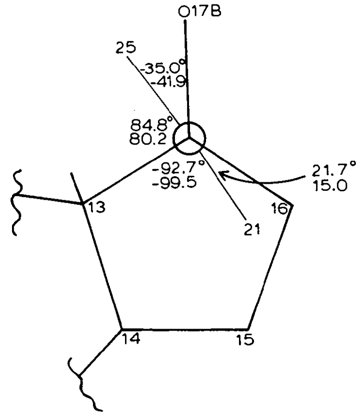

Fig. 2. [C(17)–C(20)] Newman projection. Torsion-angle values for the unprimed molecule are shown above those for the primed molecule. E.s.d.'s are 0·2°.

Table 2. *Intramolecular bond lengths* (Å) *and angles* (°)

17-PAD

	Molecule (1)	Molecule (2)		Molecule (1)	Molecule (2)		Molecule (1)	Molecule (2)		Molecule (1)	Molecule (2)
C(1)–C(2)	1·525 (4)	1·529 (4)	C(6)–C(7)	1·497 (4)	1·492 (4)	C(12)–C(13)	1·526 (3)	1·526 (4)	C(17)–O(17*B*)	1·441 (3)	1·440 (3)
C(1)–C(10)	1·547 (4)	1·542 (4)	C(7)–C(8)	1·522 (4)	1·523 (4)	C(13)–C(14)	1·534 (3)	1·535 (4)	C(20)–C(21)	1·406 (4)	1·396 (4)
C(2)–C(3)	1·504 (5)	1·491 (4)	C(8)–C(9)	1·551 (3)	1·523 (4)	C(13)–C(17)	1·561 (3)	1·566 (4)	C(20)–C(25)	1·393 (4)	1·401 (4)
C(3)–C(4)	1·519 (4)	1·503 (4)	C(8)–C(14)	1·525 (3)	1·530 (4)	C(13)–C(18)	1·539 (4)	1·539 (4)	C(21)–C(22)	1·377 (4)	1·383 (5)
C(3)–O(3*B*)	1·438 (3)	1·441 (3)	C(9)–C(10)	1·554 (3)	1·561 (3)	C(14)–C(15)	1·539 (4)	1·523 (4)	C(22)–C(23)	1·357 (5)	1·355 (8)
C(4)–C(5)	1·511 (4)	1·511 (4)	C(9)–C(11)	1·538 (4)	1·534 (4)	C(15)–C(16)	1·541 (4)	1·540 (5)	C(23)–C(24)	1·381 (5)	1·376 (6)
C(5)–C(6)	1·323 (4)	1·319 (4)	C(10)–C(19)	1·539 (4)	1·532 (4)	C(16)–C(17)	1·550 (4)	1·551 (4)	C(24)–C(25)	1·381 (4)	1·376 (5)
C(5)–C(10)	1·525 (4)	1·523 (4)	C(11)–C(12)	1·518 (3)	1·531 (4)	C(17)–C(20)	1·516 (3)	1·515 (4)			

	Molecule (1)	Molecule (2)		Molecule (1)	Molecule (2)		Molecule (1)	Molecule (2)
C(2)–C(1)–C(10)	114·1 (2)	114·0 (2)	C(1)–C(10)–C(5)	107·4 (2)	109·4 (2)	C(14)–C(15)–C(16)	104·2 (2)	104·6 (2)
C(1)–C(2)–C(3)	110·7 (2)	110·0 (2)	C(1)–C(10)–C(9)	108·1 (2)	108·5 (2)	C(15)–C(16)–C(17)	107·1 (2)	107·1 (2)
C(2)–C(3)–C(4)	111·9 (2)	110·2 (2)	C(1)–C(10)–C(19)	110·2 (2)	109·5 (2)	C(13)–C(17)–C(16)	102·0 (2)	101·1 (2)
C(2)–C(3)–O(3*B*)	107·3 (2)	112·8 (2)	C(5)–C(10)–C(9)	110·7 (2)	109·1 (2)	C(13)–C(17)–C(20)	112·0 (2)	112·9 (2)
C(4)–C(3)–O(3*B*)	110·8 (2)	108·3 (2)	C(5)–C(10)–C(19)	109·0 (2)	109·2 (2)	C(13)–C(17)–O(17*B*)	108·2 (2)	109·9 (2)
C(3)–C(4)–C(5)	112·0 (2)	112·6 (2)	C(9)–C(10)–C(19)	111·2 (2)	111·2 (2)	C(16)–C(17)–C(20)	112·8 (2)	113·9 (2)
C(4)–C(5)–C(6)	121·4 (2)	121·2 (2)	C(9)–C(11)–C(12)	112·8 (2)	112·6 (2)	C(16)–C(17)–O(17*B*)	112·5 (2)	110·4 (2)
C(4)–C(5)–C(10)	116·1 (2)	115·8 (2)	C(11)–C(12)–C(13)	110·6 (2)	111·2 (2)	C(20)–C(17)–O(17*B*)	109·1 (2)	108·6 (2)
C(6)–C(5)–C(10)	122·5 (2)	123·1 (2)	C(12)–C(13)–C(14)	108·4 (2)	107·9 (2)	C(17)–C(20)–C(21)	123·6 (2)	123·2 (2)
C(5)–C(6)–C(7)	125·4 (2)	125·3 (2)	C(12)–C(13)–C(17)	117·3 (2)	117·1 (2)	C(17)–C(20)–C(25)	120·2 (2)	120·0 (2)
C(6)–C(7)–C(8)	112·1 (2)	113·5 (2)	C(12)–C(13)–C(18)	110·2 (2)	110·4 (2)	C(21)–C(20)–C(25)	116·2 (2)	116·7 (2)
C(7)–C(8)–C(9)	109·0 (2)	110·3 (2)	C(14)–C(13)–C(17)	100·3 (2)	100·4 (2)	C(20)–C(21)–C(22)	121·5 (2)	120·9 (3)
C(7)–C(8)–C(14)	110·4 (2)	110·6 (2)	C(14)–C(13)–C(18)	111·5 (2)	111·4 (2)	C(21)–C(22)–C(23)	120·8 (2)	121·3 (3)
C(9)–C(8)–C(14)	109·5 (2)	110·0 (2)	C(17)–C(13)–C(18)	108·8 (2)	109·3 (2)	C(22)–C(23)–C(24)	119·6 (2)	119·0 (3)
C(8)–C(9)–C(10)	111·4 (2)	112·9 (2)	C(8)–C(14)–C(13)	114·5 (2)	114·8 (2)	C(23)–C(24)–C(25)	120·1 (2)	120·8 (3)
C(8)–C(9)–C(11)	112·9 (2)	113·4 (2)	C(8)–C(14)–C(15)	118·8 (2)	119·7 (2)	C(20)–C(25)–C(24)	121·8 (2)	121·2 (2)
C(10)–C(9)–C(11)	113·0 (2)	112·8 (2)	C(13)–C(14)–C(15)	104·0 (2)	104·2 (2)			

Cyclohexane

C(26)–C(27)	1·420 (25)	C(28)–C(29)	1·254 (21)	C(27)–C(26)–C(31)	117·7 (18)	C(28)–C(29)–C(30)	121·7 (16)
C(26)–C(31)	1·300 (34)	C(29)–C(30)	1·292 (25)	C(26)–C(27)–C(28)	111·2 (15)	C(29)–C(30)–C(31)	111·4 (18)
C(27)–C(28)	1·511 (23)	C(30)–C(31)	1·488 (38)	C(27)–C(28)–C(29)	111·9 (14)	C(26)–C(31)–C(30)	102·0 (17)

conformations of the two crystallographically independent steroid molecules are very similar. The *A* and *C* rings of both molecules have normal chair conformations, and both *D* rings have 13β-envelope conformations. The *B*-ring conformations differ slightly with molecule (1) having an 8β,9α half-chair and molecule (2) a conformation intermediate between a half chair and sofa. The conformation about the C(17)—C(20) bond, which defines the orientation of the phenyl ring with respect to the steroid nucleus, is illustrated in Fig. 2.

There are four crystallographically independent hydrogen bonds, and all available hydroxyls serve as hydrogen donors. The hydrogen bonds (donor → acceptor) and their distances are: O(3*B*) → O(3*B*′) 2·72, O(17*B*) → O(3*B*) 2·81, O(3*B*′) → O(17*B*′) 2·76, O(17*B*′) → O(17*B*) 2·91 Å.

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Dihydrophotoaldrin Acetate,* $C_{14}H_{12}Cl_6O_2$

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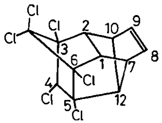
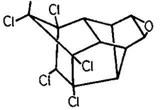
Abstract. $M_r = 425\cdot0$, monoclinic, $P2_1/c$, $a = 9\cdot399\,(4)$, $b = 11\cdot090\,(5)$, $c = 15\cdot920\,(8)$ Å, $\beta = 91\cdot48\,(4)^\circ$, $V = 1659$ Å^3, $Z = 4$, $D_m = 1\cdot70$, $D_x = 1\cdot701$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot7107$ Å, $\mu = 1\cdot03$ mm^{-1}, $F(000) = 856$, $T = 298$ K. Final $R = 0\cdot049$ for 2067 observed reflections. The title compound is one of three isomeric acetates formed by the addition of acetic acid to photoaldrin. This structure determination has confirmed the configuration suggested from chemical evidence for the acetate and analogous photoaldrin derivatives.

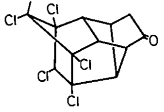
Introduction. Photoaldrin (I) and photodieldrin (II) are more active biologically as insecticides than their respective precursors, aldrin and dieldrin, from which they are formed to some extent *in vivo*. Bieniek & Korte (1969) suggested that the structure of one of the dieldrin metabolites is the ketone (III) but gave no evidence that would exclude the isomeric ketone (IV). The title compound, dihydrophotoaldrin acetate (V), is one of three isomeric acetates formed either by the regioselective reaction in the photocyclization of *exo*-dihydroaldrinol acetate or by the addition of acetic acid to photoaldrin (Bird & Khan, 1980). The structure of the acetate (V) was determined to resolve this ambiguity existing between (III) and (IV) with which (V) is related by chemical interconversion.



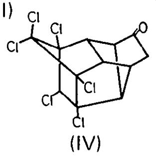
(I)



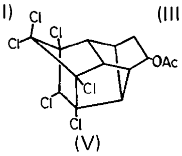
(II)



(III)



(IV)



(V)

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* IUPAC name: 2,3,3,4-*exo*,5,6-hexachloropentacyclo[6.4.0.0^{2,6}.0^{4,12}.0^{7,11}]dodec-9-*exo*-yl acetate.

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