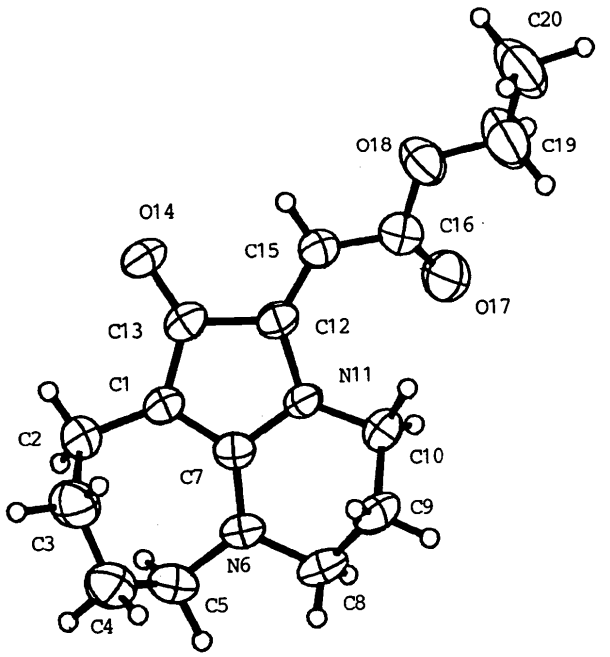

Fig. 1. Thermal ellipsoid plot. Ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

Germain, Declercq & Woolfson, 1980). The refinement was carried out by the full-matrix least-squares method with anisotropic temperature factors for non-H atoms. The function minimized was $\sum w[|(|F_o|)^2 - (|F_c|)^2|]^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}02 \times (F_o)^2]$; $\sigma(F_o)$ determined from counting statistics. All H atoms were located from a difference map and refined isotropically. Final discrepancy indices $R = 0{\cdot}056$, $wR = 0{\cdot}061$, $S = 1{\cdot}664$ for 1533 reflections with $F > 3\sigma(F)$. Maximum $\Delta/\sigma = 0{\cdot}14$ in final least-squares cycle. Final difference Fourier excursions 0·22 and $-0{\cdot}20$ e Å^{-3}. All major computations performed on a PANAFACOM computer with *RCRYSTAN* (Rigaku Corporation, 1985), an X-ray analysis program. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Final atomic parameters are listed in Table 1.* Bond lengths and angles are listed in Table 2. Fig. 1 shows a thermal ellipsoid plot of the molecule.

Related literature. The title compound was obtained from the reaction of 1,8-diazabicyclo[5.4.0]undec-7-ene with diethyl acetylenedicarboxylate in CH_2Cl_2 at room temperature. See also Hermecz (1987) for the preparation of related compounds.

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54001 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,3,9-Triphenylindeno[3,2-*b*]pyridine

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Abstract. $C_{30}H_{21}N$, $M_r = 395{\cdot}5$, triclinic, $P\bar{1}$, $a = 11{\cdot}826$ (2), $b = 10{\cdot}853$ (1), $c = 10{\cdot}074$ (1) Å, $\alpha = 116{\cdot}30$ (2), $\beta = 111{\cdot}94$ (1), $\gamma = 83{\cdot}02$ (1)°, $V = 1073{\cdot}7$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}223$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1·5405 Å, $\mu = 0{\cdot}548$ mm^{-1}, $F(000) = 416$, $T = 293$ K, final $R = 0{\cdot}048$ for 3563 observed reflections. The dihedral angles between azafluorene and the three phenyl rings (substituted on positions 1, 3 and 9) are 8·4 (2), 55·4 (3) and 44·0 (2)°, respectively.

Experimental. A colorless prism, 0·30 × 0·25 × 0·40 mm, by recrystallization from benzene. Rigaku AFC-5 four-circle diffractometer used with ω–2θ scan method, ω-scan width $(1{\cdot}3 + 0{\cdot}41\tan\theta)$° and

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

$B_{eq} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq} (Å^2)
N(1)	0·5674 (1)	0·1198 (1)	0·3920 (1)	4·20 (3)
C(2)	0·4998 (1)	0·0171 (1)	0·2546 (1)	4·13 (4)
C(3)	0·5146 (1)	−0·1213 (1)	0·2257 (1)	4·37 (4)
C(4)	0·5977 (1)	−0·1592 (1)	0·3411 (1)	4·24 (4)
C(5)	0·6656 (1)	−0·0531 (1)	0·4832 (1)	4·08 (4)
C(6)	0·6473 (1)	0·0822 (1)	0·5007 (1)	4·01 (4)
C(7)	0·7666 (1)	0·0553 (1)	0·6292 (1)	4·31 (4)
C(8)	0·8031 (1)	0·0973 (1)	0·7317 (1)	4·38 (4)
C(9)	0·8937 (1)	0·1603 (1)	0·8805 (2)	5·35 (5)
C(10)	0·9110 (1)	0·3020 (1)	0·9518 (2)	5·72 (6)
C(11)	0·8383 (1)	0·3813 (1)	0·8774 (2)	5·65 (6)
C(12)	0·7479 (1)	0·3191 (1)	0·7289 (2)	4·90 (5)
C(13)	0·7318 (1)	0·1763 (1)	0·6566 (1)	4·20 (4)
C(14)	0·4061 (1)	0·0618 (1)	0·1389 (1)	4·34 (4)
C(15)	0·4019 (1)	0·2002 (1)	0·1707 (2)	5·54 (6)
C(16)	0·3120 (1)	0·2469 (2)	0·0720 (2)	6·80 (7)
C(17)	0·2246 (1)	0·1563 (2)	−0·0626 (2)	7·01 (7)
C(18)	0·2273 (1)	0·0193 (2)	−0·0967 (2)	7·10 (7)
C(19)	0·3167 (1)	−0·0278 (1)	0·0038 (2)	5·90 (6)
C(20)	0·6136 (1)	−0·3068 (1)	0·3112 (1)	4·37 (4)
C(21)	0·6421 (1)	−0·4005 (1)	0·1818 (2)	6·24 (6)
C(22)	0·6625 (2)	−0·5360 (1)	0·1593 (2)	7·10 (8)
C(23)	0·6524 (1)	−0·5789 (1)	0·2638 (2)	6·54 (7)
C(24)	0·6224 (1)	−0·4878 (1)	0·3911 (2)	6·69 (7)
C(25)	0·6032 (1)	−0·3518 (1)	0·4150 (2)	5·79 (6)
C(26)	0·9176 (1)	−0·1366 (1)	0·4886 (2)	5·60 (6)
C(27)	1·0106 (1)	−0·2192 (2)	0·4517 (2)	6·57 (7)
C(28)	1·0592 (1)	−0·3070 (2)	0·5200 (2)	6·88 (7)
C(29)	1·0155 (1)	−0·3139 (2)	0·6255 (2)	7·49 (8)
C(30)	0·9211 (1)	−0·2327 (1)	0·6622 (2)	6·01 (6)
C(31)	0·8708 (1)	−0·1435 (1)	0·5932 (1)	4·29 (4)

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

N(1)—C(2)	1·349 (1)	N(1)—C(6)	1·337 (2)
C(2)—C(3)	1·399 (2)	C(2)—C(14)	1·493 (2)
C(3)—C(4)	1·399 (2)	C(4)—C(5)	1·388 (2)
C(4)—C(20)	1·494 (2)	C(5)—C(6)	1·399 (2)
C(5)—C(7)	1·517 (2)	C(7)—C(8)	1·524 (2)
C(7)—C(31)	1·518 (2)	C(8)—C(9)	1·390 (2)
C(8)—C(13)	1·394 (2)	C(9)—C(10	1·382 (3)
C(10)—C(11)	1·397 (3)	C(11)—C(12)	1·386 (2)
C(12)—C(13)	1·391 (2)	C(13)—C(6)	1·470 (2)

C(2)—N(1)—C(6)	116·2 (1)	N(1)—C(2)—C(3)	121·9 (1)
N(1)—C(2)—C(14)	115·3 (1)	C(3)—C(2)—C(14)	122·6 (1)
C(2)—C(3)—C(4)	121·2 (1)	C(3)—C(4)—C(5)	116·6 (1)
C(3)—C(4)—C(20)	121·4 (1)	C(5)—C(4)—C(20)	121·8 (1)
C(4)—C(5)—C(6)	118·4 (1)	C(4)—C(5)—C(7)	131·2 (1)
C(6)—C(5)—C(7)	110·2 (1)	C(5)—C(6)—N(1)	125·4 (1)
C(5)—C(6)—C(13)	109·0 (1)	N(1)—C(6)—(13)	125·4 (1)
C(5)—C(7)—C(8)	101·7 (1)	C(5)—C(7)—C(31)	113·6 (1)
C(8)—C(7)—C(31)	114·0 (1)	C(7)—C(8)—C(9)	128·8 (1)
C(7)—C(8)—C(13)	110·9 (1)	C(9)—C(8)—C(13)	120·2 (1)
C(8)—C(9)—C(10)	118·7 (1)	C(9)—C(10)—C(11)	120·9 (1)
C(10)—C(11)—C(12)	120·5 (1)	C(11)—C(12)—C(13)	118·3 (1)
C(12)—C(13)—C(6)	130·8 (1)	C(12)—C(13)—C(8)	121·1 (1)
C(6)—C(13)—C(8)	108·0 (1)		

scan speed 8° min^{-1}. Lattice parameters obtained from least-squares analysis of 25 reflections with 2θ values ranging from 25 to 55°. Of 3641 reflections scanned (within index range $h-14\rightarrow14$, $k-13\rightarrow13$, l $0\rightarrow12$ up to $\sin\theta/\lambda \leq 0{\cdot}59$ Å^{-1}), 3563 unique reflections [$F > 3\sigma(F)$] classified as observed. Three standard reflections measured every 200 reflections, no significant intensity variation. Intensities corrected for Lorentz and polarization factors, but absorption correction not applied. Structure solved using *SAPI*85 (Yao, Zheng, Qian, Han, Gu & Fan, 1985), a version of *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). The refinement was carried out by the full-matrix least-squares method with anisotropic temperature factors for non-H atoms. The function minimized was $\sum w[(|F_o|)^2 - (|F_c|)^2|]^2$ with $w = 1/[\sigma^2(F_o) + 0{\cdot}02(F_o)^2]$; $\sigma(F_o)$ determined from counting statistics. All H atoms were located from a difference map and refined isotropically. Final discrepancy indices $R = 0{\cdot}048$, $wR = 0{\cdot}054$, $S = 1{\cdot}760$. Maximum $\Delta/\sigma = 0{\cdot}22$ in final least-squares cycle. Final difference Fourier excursions 0·16 and −0·25 e Å^{-3}. All major computations performed on PANAFACOM computer with *RCRYSTAN* (Rigaku Corporation, 1985), an X-ray analysis program. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Final atomic parameters are listed in Table 1.* Selected bond lengths and angles are listed in Table 2. Fig. 1 shows a thermal ellipsoid plot of the molecule.

Related literature. The title compound was obtained from the neat thermolysis of 4,6-diphenyl-1,2,3-triazine at 523 K (Ohsawa, Arai, Ohnishi, Itoh, Kaihoh, Okada & Igeta, 1985).

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54002 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

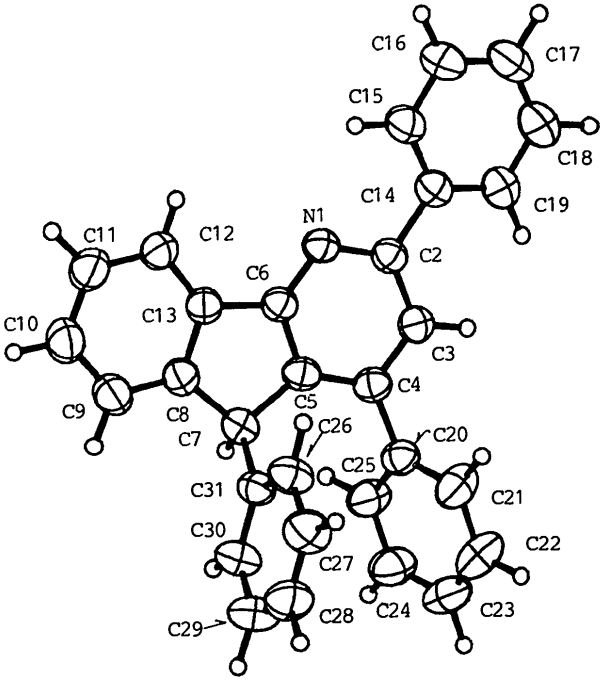

Fig. 1. Thermal ellipsoid plot. Ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

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Structure of a 1,4-Dien-3-one-6α-hydroxy Steroid

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Abstract. 9α,21-Dichloro-6α,11β,17α-trihydroxy-16α-methyl-3,20-dioxopregna-1,4-dien-17-yl 2-furoate, $C_{27}H_{30}Cl_2O_7$, $M_r = 537{\cdot}43$, monoclinic, $P2_1$, $a = 11{\cdot}524$ (2), $b = 15{\cdot}751$ (4), $c = 7{\cdot}975$ (2) Å, $\beta = 111{\cdot}42$ (1)°, $V = 1347{\cdot}7$ (6) Å^3, $Z = 2$, $D_x = 1{\cdot}324$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 2{\cdot}53$ mm^{-1}, $F(000) = 564$, $T = 295$ K, $R = 0{\cdot}047$ for 2298 observed reflections [$F_o > 3\sigma(F_o)$]. The molecules are linked by intermolecular hydrogen bonds; O(26)—HO(27)($1-x$, $y-\frac{1}{2}$, $1-z$) = 2·697 (5) [2·00 (6) for O···H] and O(31)—HO(26) ($2-x$, $\frac{1}{2}+y$, $2-z$) = 2·895 (6) Å [2·04 (8) Å].

Experimental. Colorless plate crystals obtained from methanol. Crystal of dimensions 0·3 × 0·3 × 0·1 mm. Rigaku AFC-5R diffractometer, graphite-monochromatized Cu $K\alpha$. Cell dimensions determined from 2θ angles for 25 reflections in the range $26 < 2\theta < 46°$. Intensities measured up to $\theta = 70°$ in h −13/14, k 0/19 and l −9/0, ω–2θ scans, ω-scan width $(2 + 0{\cdot}2\tan\theta)°$, three standard reflections monitored every 100 measurements showed no significant change. 2571 unique reflections measured, 2298 intensities observed [$F_o \le 3\sigma(F_o)$ and one very strong reflection rejected], no absorption correction. Structures solved by direct methods with *MULTAN*84 (Main, Germain & Woolfson, 1984). H atoms located on a difference density map. Positional parameters of all atoms and anisotropic thermal parameters of non-H atoms refined by block-diagonal least squares. Temperature factor of each H atom equal to B_{eq} of the bonded atom. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0{\cdot}00146|F_o|^2]$, $w = 0$ for 53 reflections with $w^{1/2}|\Delta F| \ge 3$. Final $R = 0{\cdot}047$, $wR = 0{\cdot}055$, $S = 1{\cdot}1150$. Max. Δ/σ in the final cycle 0·03. The highest and lowest peaks in the final difference map are 0·6 and −0·5 e Å^{-3}. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1,...,4$) (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Calculations performed on a VAX station 3100 computer. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Bond lengths and

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
C(1)	0·4496 (4)	0·2944 (3)	0·3006 (5)	4·1 (1)
C(2)	0·4305 (4)	0·2601 (3)	0·1399 (5)	4·5 (1)
C(3)	0·4636 (5)	0·1717 (3)	0·1247 (5)	4·8 (1)
C(4)	0·5123 (4)	0·1232 (3)	0·2894 (5)	4·2 (1)
C(5)	0·5293 (3)	0·1560 (3)	0·4511 (5)	3·4 (1)
C(6)	0·5800 (4)	0·1035 (3)	0·6214 (5)	3·7 (1)
C(7)	0·6837 (4)	0·1481 (3)	0·7692 (5)	3·7 (1)
C(8)	0·6607 (3)	0·2419 (3)	0·7940 (5)	3·2 (1)
C(9)	0·6162 (3)	0·2900 (3)	0·6137 (4)	3·0 (1)
C(10)	0·4958 (4)	0·2467 (3)	0·4755 (5)	3·5 (1)
C(11)	0·6087 (3)	0·3875 (2)	0·6367 (5)	3·3 (1)
C(12)	0·7241 (4)	0·4244 (2)	0·7837 (5)	3·4 (1)
C(13)	0·7605 (4)	0·3775 (3)	0·9640 (5)	3·4 (1)
C(14)	0·7766 (3)	0·2827 (3)	0·9289 (4)	3·3 (1)
C(15)	0·8322 (4)	0·2440 (3)	1·1144 (5)	4·1 (1)
C(16)	0·9221 (4)	0·3136 (3)	1·2277 (5)	4·2 (1)
C(17)	0·8932 (4)	0·3952 (3)	1·1073 (5)	3·6 (1)
C(18)	0·3848 (4)	0·2431 (3)	0·5421 (6)	4·3 (1)
C(19)	0·6645 (4)	0·3924 (3)	1·0517 (5)	4·1 (1)
C(20)	0·8997 (4)	0·4774 (3)	1·2155 (6)	4·6 (1)
C(21)	0·8873 (6)	0·5591 (4)	1·1128 (8)	6·2 (2)
C(22)	1·0559 (5)	0·2841 (5)	1·3026 (8)	6·9 (2)
Cl(23)	0·7371 (1)	0·2798	0·5149 (1)	3·79 (2)
Cl(24)	0·9575 (3)	0·6477 (1)	1·2487 (3)	10·9 (1)
O(25)	0·4511 (4)	0·1391 (3)	−0·0219 (4)	7·2 (2)
O(26)	0·6146 (3)	0·0219 (2)	0·5876 (4)	5·4 (1)
O(27)	0·4992 (2)	0·4065 (2)	0·6700 (4)	3·9 (1)
O(28)	0·9020 (3)	0·4765 (3)	1·3662 (4)	6·2 (1)
O(29)	0·9740 (2)	0·4019 (2)	1·0035 (4)	3·9 (1)
C(30)	1·0879 (3)	0·4338 (3)	1·0767 (5)	3·9 (1)
O(31)	1·1334 (3)	0·4612 (3)	1·2299 (4)	5·3 (1)
C(32)	1·1485 (4)	0·4369 (3)	0·9464 (6)	4·3 (1)
C(33)	1·1102 (5)	0·4264 (6)	0·7686 (7)	7·5 (2)
C(34)	1·2104 (6)	0·4453 (6)	0·7177 (8)	8·3 (3)
C(35)	1·3070 (5)	0·4622 (4)	0·8680 (7)	5·7 (2)
O(36)	1·2707 (3)	0·4586 (2)	1·0116 (4)	4·5 (1)

* Lists of H-atom coordinates, anisotropic temperature factors of the non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53985 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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