KRIEGER, C., KOCAK, A. & BEKAROGLU, O. (1985). *Helv. Chim. Acta*, **68**, 581–583.
LIPKOWSKI, J., HERBICH, J. & ANDREETTI, G. D. (1985). 9th Eur. Crystallogr. Meet., Turin, Italy. Abstracts, pp. 663–664.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
VISSER, G. J. & VOS, A. (1971). *Acta Cryst.* B**27**, 1793–1801.
VISSER, G. J., VOS, A., DE GROOT, A. & WYNBERG, H. (1968). *J. Am. Chem. Soc.* **90**, 3253–3254.
WOŹNIAK, K., KRYGOWSKI, T., KARIUKI, B. & JONES, W. (1990). *Acta Cryst.* C**46**, 1946–1947.

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Structure of a Pyrimidoazepine Derivative

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Abstract. Ethyl 1-oxoperhydropyrrolo[1′,2′:3,2;2′,3′-*b*]pyrimido[1,2-*a*]azepin-2-ylideneacetate, $C_{15}H_{20}N_2O_3$, $M_r = 276{\cdot}3$, monoclinic, $P2_1/c$, $a = 20{\cdot}472\ (3)$, $b = 8{\cdot}869\ (1)$, $c = 7{\cdot}806\ (2)$ Å, $\beta = 99{\cdot}27\ (1)°$, $V = 1398{\cdot}8\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}312$ Mg m^{-3}, λ(Cu $K\alpha_1$) $= 1{\cdot}5405$ Å, $\mu = 0{\cdot}759$ mm^{-1}, $F(000) = 592$, $T = 293$ K, final $R = 0{\cdot}056$ for 1533 observed reflections. The seven- and six-membered ring has half-boat puckering with a folded *cis* conformation. The puckering parameter, the angle between the base plane C(2)—C(1)—C(7)—N(6) and C(2)—C(3)—C(4)—C(5), is 56·9 (5)°, and between the base plane C(8)—N(6)—C(7)—N(11)—C(10) and C(10)—C(9)—C(8) is 51·3 (5)°.

Experimental. A reddish needle, 0·15 × 0·05 × 0·40 mm, by recrystallization from CH_2Cl_2. Rigaku AFC-5 four-circle diffractometer used with the ω–2θ scan method, ω-scan width $(1{\cdot}3 + 0{\cdot}41\tan\theta)°$ and scan speed 16° min^{-1}. Lattice parameters obtained from least-squares analysis of 20 reflections with 2θ values ranging from 56 to 61°. Of 2467 reflections scanned [within the index range h −22→22, k 0→9, l 0→8 up to $\sin\theta/\lambda \le 0{\cdot}56$ Å^{-1} including 182 equivalent reflections ($R_{int} = 0{\cdot}03$)], 2076 unique reflections [$F > \sigma(F)$] classified as observed. Three standard reflections measured every 150 reflections, intensity variation < 3%. 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,

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)
C(1)	0·1507 (1)	0·2031 (3)	0·7572 (4)	3·46 (10)
C(2)	0·0802 (1)	0·1746 (5)	0·6770 (6)	4·45 (12)
C(3)	0·0484 (2)	0·0499 (5)	0·7708 (8)	5·88 (17)
C(4)	0·0725 (2)	−0·1096 (5)	0·7399 (8)	6·17 (17)
C(5)	0·1389 (2)	−0·1229 (5)	0·6829 (6)	5·19 (14)
N(6)	0·1939 (1)	−0·0540 (2)	0·8006 (3)	3·72 (9)
C(7)	0·1982 (1)	0·0955 (3)	0·8157 (4)	3·24 (9)
C(8)	0·2484 (2)	−0·1500 (4)	0·8792 (5)	4·74 (12)
C(9)	0·2839 (2)	−0·0772 (4)	1·0434 (5)	4·63 (12)
C(10)	0·3099 (1)	0·0754 (4)	1·0036 (5)	3·81 (11)
N(11)	0·2570 (1)	0·1605 (2)	0·8992 (3)	3·24 (7)
C(12)	0·2480 (1)	0·3141 (3)	0·9000 (4)	3·12 (9)
C(13)	0·1781 (1)	0·3439 (3)	0·8085 (4)	3·49 (10)
O(14)	0·1546 (1)	0·4738 (2)	0·7871 (3)	4·62 (8)
C(15)	0·2891 (1)	0·4259 (4)	0·9631 (4)	3·53 (10)
C(16)	0·3591 (1)	0·4231 (4)	1·0239 (4)	4·01 (11)
O(17)	0·3989 (1)	0·3336 (3)	0·9870 (4)	6·04 (10)
O(18)	0·3776 (1)	0·5435 (3)	1·1267 (3)	5·32 (8)
C(19)	0·4474 (2)	0·5544 (8)	1·1990 (8)	7·30 (20)
C(20)	0·4547 (4)	0·6724 (13)	1·3294 (13)	8·30 (25)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·500 (5)	C(1)—C(7)	1·386 (5)
C(1)—C(13)	1·401 (5)	C(2)—C(3)	1·529 (7)
C(3)—C(4)	1·530 (7)	C(4)—C(5)	1·502 (8)
C(5)—N(6)	1·466 (5)	N(6)—C(7)	1·333 (4)
N(6)—C(8)	1·459 (5)	C(8)—C(9)	1·512 (6)
C(9)—C(10)	1·505 (6)	C(10)—N(11)	1·458 (5)
N(11)—C(7)	1·397 (4)	N(11)—C(12)	1·375 (4)
C(12)—C(13)	1·516 (5)	C(12)—C(15)	1·341 (5)
C(13)—O(14)	1·250 (4)	C(15)—C(16)	1·436 (5)
C(16)—O(17)	1·204 (5)	C(16)—O(18)	1·352 (5)
O(18)—C(19)	1·453 (6)	C(19)—C(20)	1·450 (13)

C(2)—C(1)—C(7)	126·7 (3)	C(2)—C(1)—C(13)	125·6 (3)
C(7)—C(1)—C(13)	107·0 (3)	C(1)—C(2)—C(3)	112·1 (3)
C(2)—C(3)—C(4)	114·8 (5)	C(3)—C(4)—C(5)	116·7 (4)
C(4)—C(5)—C(6)	114·9 (4)	C(5)—N(6)—C(7)	120·1 (3)
C(5)—N(6)—C(8)	118·6 (3)	C(7)—N(6)—C(8)	120·5 (2)
C(1)—C(7)—N(6)	128·5 (3)	C(1)—C(7)—N(11)	112·0 (2)
N(6)—C(7)—N(11)	119·4 (2)	N(6)—C(8)—C(9)	109·7 (3)
C(8)—C(9)—C(10)	110·2 (3)	C(9)—C(10)—N(11)	108·9 (3)
C(10)—N(11)—C(7)	123·6 (2)	C(10)—N(11)—C(12)	126·7 (2)
C(7)—N(11)—C(12)	107·7 (2)	N(11)—C(12)—C(13)	106·6 (2)
N(11)—C(12)—C(15)	131·1 (3)	C(13)—C(12)—C(15)	122·1 (3)
C(12)—C(13)—C(1)	106·4 (2)	C(12)—C(13)—O(14)	122·5 (3)
C(1)—C(13)—O(14)	130·9 (3)	C(12)—C(15)—C(16)	129·9 (3)
C(15)—C(16)—O(17)	127·5 (3)	C(15)—C(16)—O(18)	110·5 (3)
O(17)—C(16)—O(18)	121·8 (3)	C(16)—O(18)—C(19)	116·4 (4)
O(18)—C(19)—C(20)	107·7 (5)		

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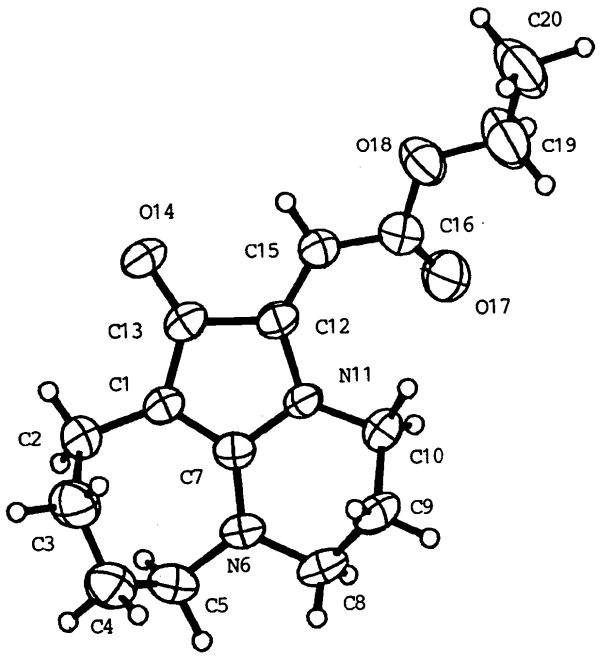

Fig. 1. Thermal ellipsoid plot. Ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

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.

References

HERMECZ, I. (1987). *Chemistry of Diazabicycloundecene and Other Pyrimidoazepines.* In *Advances in Heterocylic Chemistry*, Vol. 42, edited by A. R. KATRITZKY, pp. 84–202. New York: Academic Press, and references cited therein.

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.

Rigaku Corporation (1985). *RCRYSTAN. X-ray Analysis Program System.* Rigaku Corporation, Tokyo, Japan.

YAO, J.-X., ZHENG, C.-D., QIAN, J.-Z., HAN, F.-S., GU, Y.-X. & FAN, H.-F. (1985). *SAPI85.* Institute of Physics, Chinese Academy of Sciences, Beijing, People's Republic of China.

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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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