Related literature. Related structures with planar benzopyran and benzoxazole rings: Cantrell & Stalzer (1982); Dreher, Bracht, Mobayed, Hütter, Winter & Rieker (1982).

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Structure of a 1,4-Oxazepin-5-one Derivative

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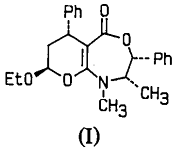
Abstract. (2*S*,3*R*,6*R*,8*R*)-8-Ethoxy-2,3,7,8-tetrahydro-1,2-dimethyl-3,6-diphenyl-1*H*,6*H*-pyrano[2,3-*e*][1,4]oxazepin-5-one–dichloromethane (1/1), $C_{24}H_{27}NO_4.CH_2Cl_2$, $M_r = 478.42$, orthorhombic, $P2_12_12_1$, $a = 11.885(1)$, $b = 12.738(1)$, $c = 15.817(2)$ Å, $V = 2394.43$ Å^3, $Z = 4$, $D_x = 1.327$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0.71069$ Å, $\mu = 0.30$ mm^{-1}, $F(000) = 1008$, $T = 298$ K, $R = 0.049$ for 3497 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. The seven-membered and the six-membered rings both adopt a half-chair conformation.

Experimental. Title compound (I): crystal size 0·4 × 0·4 × 0·6 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 4882 reflections measured, $2\theta_{\max}$ 50°, $\pm h$ $+k$ $+l$, three check reflections with no significant intensity change. 4265 unique reflections ($R_{\rm int} = 0.022$), of which 3497 with $F > 4\sigma(F)$ were used for all calculations (*SHELXS*86, Sheldrick, 1985; *SHELX*76, Sheldrick, 1976). Cell constants refined from $\pm 2\theta$ values of 40 reflections in the range 20–25°. Absorption correction was not applied. Extinction correction was applied yielding a value of 0·0021 (3) for the secondary-extinction coefficient. Structure solution by direct methods. Refinement on F to $R = 0.049$, $wR = 0.058$; all non-H atoms anisotropic, H atoms were included using a riding model [C—H 0·96 Å, U(H) = 0·08 Å^2, except for methyl protons U(H) = 0·1 Å^2]. We refined 290 parameters, $S = 1.853$, weighting scheme $w^{-1} = \sigma^2(F) + 0.0004F^2$ which led to a featureless analysis of variance in terms of sinθ and F_o, max. $\Delta/\sigma = 0.006$, max. and min. heights in final $\Delta\rho$ map 0·37 and −0·35 e Å^{-3} respectively. A Rogers (1981) η-refinement [$\eta = 1.0(2)$] confirmed the known absolute configuration. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).



(I)

Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows a thermal ellipsoid plot with the atom numbering, and Fig. 2 indicates the conformation of the molecule.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51152 (18 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* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

	x	*y*	*z*	U_{eq}*
O(1)	195 (2)	8945 (2)	8964 (1)	62 (1)
C(1)	1209 (2)	8805 (2)	8917 (2)	45 (1)
O(2)	1522 (2)	7995 (2)	8397 (1)	51 (1)
C(3)	2664 (2)	7902 (2)	8127 (2)	43 (1)
C(4)	3413 (2)	7546 (2)	8857 (2)	43 (1)
N(5)	3787 (2)	8438 (2)	9366 (1)	45 (1)
C(5*A*)	3114 (2)	9262 (2)	9562 (2)	39 (1)
O(6)	3717 (2)	9939 (1)	10054 (1)	55 (1)
C(7)	3246 (2)	10917 (2)	10375 (2)	46 (1)
C(8)	2365 (2)	11312 (2)	9787 (2)	47 (1)
C(9)	1468 (2)	10472 (2)	9667 (2)	42 (1)
C(9*A*)	2007 (2)	9453 (2)	9360 (2)	40 (1)
C(1′)	2688 (2)	7133 (2)	7401 (2)	42 (1)
C(2′)	1853 (3)	6396 (2)	7275 (2)	53 (1)
C(3′)	1901 (3)	5692 (2)	6606 (2)	59 (1)
C(4′)	2813 (3)	5716 (3)	6069 (2)	62 (1)
C(5′)	3667 (3)	6438 (2)	6190 (2)	59 (1)
C(6′)	3602 (3)	7144 (2)	6855 (2)	50 (1)
C(4*M*)	2900 (3)	6697 (3)	9402 (2)	61 (1)
C(5*M*)	4967 (2)	8410 (3)	9630 (2)	60 (1)
O(1*E*)	4145 (2)	11610 (2)	10440 (1)	58 (1)
C(1*E*)	4771 (3)	11582 (3)	11213 (2)	72 (1)
C(2*E*)	5713 (4)	10848 (4)	11208 (3)	97 (2)
C(1″)	723 (2)	10368 (2)	10460 (2)	40 (1)
C(2″)	990 (3)	9697 (3)	11113 (2)	54 (1)
C(3″)	346 (3)	9673 (3)	11843 (2)	63 (1)
C(4″)	−592 (3)	10298 (3)	11910 (2)	60 (1)
C(5″)	−871 (3)	10955 (3)	11265 (2)	60 (1)
C(6″)	−214 (3)	10995 (3)	10541 (2)	50 (1)
Cl(1)	1344 (1)	1036 (1)	7408 (1)	101 (1)
Cl(2)	2658 (1)	2838 (1)	6930 (1)	108 (1)
C(1*C*)	1450 (4)	2109 (3)	6740 (3)	81 (1)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

O(1)—C(1)	1·221 (3)	C(1)—O(2)	1·372 (3)
C(1)—C(9*A*)	1·439 (4)	O(2)—C(3)	1·428 (3)
C(3)—C(4)	1·527 (4)	C(3)—C(1′)	1·509 (4)
C(4)—N(5)	1·461 (3)	C(4)—C(4*M*)	1·511 (4)
N(5)—C(5*A*)	1·355 (3)	N(5)—C(5*M*)	1·464 (4)
C(5*A*)—O(6)	1·365 (3)	C(5*A*)—C(9*A*)	1·376 (4)
O(6)—C(7)	1·457 (3)	C(7)—C(8)	1·489 (4)
C(7)—O(1*E*)	1·390 (3)	C(8)—C(9)	1·522 (4)
C(9)—C(9*A*)	1·527 (4)	C(9)—C(1″)	1·541 (4)
C(1′)—C(2′)	1·381 (4)	C(1′)—C(6′)	1·388 (4)
C(2′)—C(3′)	1·387 (4)	C(3′)—C(4′)	1·378 (5)
C(4′)—C(5′)	1·383 (5)	C(5′)—C(6′)	1·386 (4)
O(1*E*)—C(1*E*)	1·431 (4)	C(1*E*)—C(2*E*)	1·459 (6)
C(1″)—C(2″)	1·377 (4)	C(1″)—C(6″)	1·376 (4)
C(2″)—C(3″)	1·385 (5)	C(3″)—C(4″)	1·374 (5)
C(4″)—C(5″)	1·361 (5)	C(5″)—C(6″)	1·387 (5)
Cl(1)—C(1*C*)	1·733 (4)	Cl(2)—C(1*C*)	1·737 (5)

O(1)—C(1)—O(2)	114·5 (2)	O(1)—C(1)—C(9*A*)	122·5 (2)
O(2)—C(1)—C(9*A*)	123·0 (2)	C(1)—O(2)—C(3)	120·0 (2)
O(2)—C(3)—C(4)	110·6 (2)	O(2)—C(3)—C(1′)	107·4 (2)
C(4)—C(3)—C(1′)	111·8 (2)	C(3)—C(4)—N(5)	111·3 (2)
C(3)—C(4)—C(4*M*)	114·1 (2)	N(5)—C(4)—C(4*M*)	111·4 (2)
C(4)—N(5)—C(5*A*)	123·3 (2)	C(4)—N(5)—C(5*M*)	115·4 (2)
C(5*A*)—N(5)—C(5*M*)	121·3 (2)	N(5)—C(5*A*)—O(6)	108·1 (2)
N(5)—C(5*A*)—C(9*A*)	130·4 (2)	O(6)—C(5*A*)—C(9*A*)	121·5 (2)
C(5*A*)—O(6)—C(7)	122·5 (2)	O(6)—C(7)—C(8)	109·9 (2)
O(6)—C(7)—O(1*E*)	105·9 (2)	C(8)—C(7)—O(1*E*)	111·8 (2)
C(7)—C(8)—C(9)	109·4 (2)	C(8)—C(9)—C(9*A*)	110·1 (2)
C(8)—C(9)—C(1″)	111·2 (2)	C(9*A*)—C(9)—C(1″)	115·3 (2)
C(1)—C(9*A*)—C(5*A*)	129·9 (2)	C(1)—C(9*A*)—C(9)	111·5 (2)
C(5*A*)—C(9*A*)—C(9)	118·5 (2)	C(3)—C(1′)—C(2′)	122·6 (2)
C(3)—C(1′)—C(6′)	118·8 (2)	C(2′)—C(1′)—C(6′)	118·6 (3)
C(1′)—C(2′)—C(3′)	121·4 (3)	C(2′)—C(3′)—C(4′)	119·3 (3)
C(3′)—C(4′)—C(5′)	120·4 (3)	C(4′)—C(5′)—C(6′)	119·7 (3)
C(1′)—C(6′)—C(5′)	120·6 (3)	C(7)—O(1*E*)—C(1*E*)	116·6 (2)
O(1*E*)—C(1*E*)—C(2*E*)	114·1 (3)	C(9)—C(1″)—C(2″)	122·1 (2)
C(9)—C(1″)—C(6″)	119·4 (2)	C(2″)—C(1″)—C(6″)	118·5 (3)
C(1″)—C(2″)—C(3″)	120·8 (3)	C(2″)—C(3″)—C(4″)	120·0 (3)
C(3″)—C(4″)—C(5″)	119·7 (3)	C(4″)—C(5″)—C(6″)	120·3 (3)
C(1″)—C(6″)—C(5″)	120·7 (3)	Cl(1)—C(1*C*)—Cl(2)	112·1 (2)

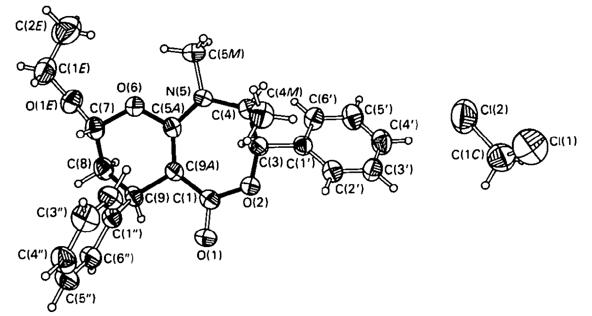

Fig. 1. The asymmetric unit showing 50% thermal displacement ellipsoids.

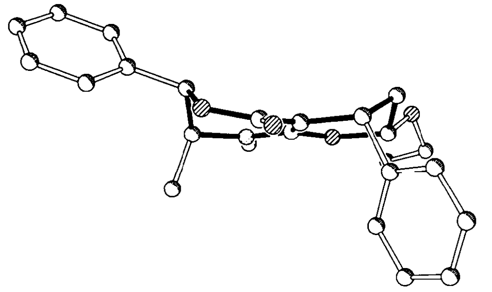

Fig. 2. The conformation of the tetrahydropyranooxazepine ring system (H atoms omitted).

Related literature. For the preparation of the compound see Pfeiffer (1988). For the preparation of some related compounds see Tietze, Brand, Pfeiffer, Antel, Harms & Sheldrick (1987). For a related structure see Antel, Sheldrick, Pfeiffer & Tietze (1988).

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