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Structure of a Benzopyranopyranooxazepinone

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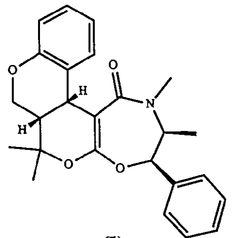
Abstract. *cis*-(+)-(3*S*,4*R*,7a*S*,13b*R*)-2,3,7,7-Tetramethyl-4-phenyl-1,2,3,4,7,7a,8,13b-octahydro-8*H*-[1]-benzopyrano[4′,3′:4,5]pyrano[3,2-*f*][1,4]oxazepin-1-one monohydrate, $C_{25}H_{27}NO_4.H_2O$, $M_r = 423{\cdot}49$, monoclinic, $P2_1$, $a = 6{\cdot}563\,(1)$, $b = 7{\cdot}922\,(2)$, $c = 20{\cdot}877\,(4)$ Å, $\beta = 94{\cdot}32\,(2)°$, $V = 1082{\cdot}46$ Å^3, $Z = 2$, $D_x = 1{\cdot}30$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 448$, $T = 298$ K, $R = 0{\cdot}057$ for 2256 observed reflections. The structure was investigated to determine the relative configuration at C(7a) and C(13b), which could not be established unambiguously by NMR. The absolute configuration at C(3) and C(4) was already known from the educt L-ephedrine. The seven-membered oxazepine ring shows a pseudo-boat conformation. The two dihydropyran rings adopt a half-chair conformation. The title compound cocrystallized with one molecule of water, which forms a weak hydrogen bond [O(6)···O(90) = 2·84 Å].

Experimental. (I): crystal size 0·4 × 0·6 × 0·1 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 2814 reflections measured, $2\theta_{\max}$ 45°, $\pm h + k + l$, three check reflections with no significant intensity change. 2814 unique reflections, of which 2256 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 32 reflections in the range 20–25°. Absorption correction was deemed unnecessary. Extinction correction was applied yielding a secondary-extinction coefficient x of 0·0049 (8), where $F_c^* = F_c(1 + 0{\cdot}002xF_c^2/\sin 2\theta)^{-0{\cdot}25}$. Structure solution by direct methods. Refinement on F to $R = 0{\cdot}057$, $wR = 0{\cdot}062$; all non-H atoms anisotropic, H atoms were refined using a riding model [C—H 0·96 Å, $U(\text{H}) = 0{\cdot}08$ Å^2, except for methyl protons $U(\text{H}) = 0{\cdot}12$ Å^2]. H atoms attached to water O(90) were not located. 280 parameters were refined, $S = 1{\cdot}47$, weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0006F^2$ which led to a featureless analysis of variance in terms of $\sin\theta$ and F_o, max. $\Delta/\sigma = 0{\cdot}55$ for U_{23} of O(90), max. and min. heights in final $\Delta\rho$ map 0·31 and $-0{\cdot}43$ e Å^{-3} respectively. 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.

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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 52146 (13 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* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

	x	y	z	U_{eq}*
O(90)	13097 (11)	−2506 (12)	8077 (3)	235 (4)
C(5)	5401 (6)	830	7543 (2)	47 (1)
N(4)	6245 (5)	1901 (5)	7995 (1)	45 (1)
C(3)	8440 (6)	1923 (5)	8165 (2)	45 (1)
C(2)	9118 (7)	418 (6)	8577 (2)	49 (1)
O(1)	8409 (5)	−1164 (4)	8286 (1)	62 (1)
C(5b)	8051 (7)	−1312 (6)	7636 (2)	51 (2)
O(6)	9086 (5)	−2698 (4)	7463 (1)	57 (1)
C(7)	8345 (7)	−3433 (6)	6839 (2)	60 (2)
C(7a)	8094 (7)	−2005 (6)	6346 (2)	52 (2)
C(8)	10084 (7)	−1053 (7)	6273 (2)	62 (2)
O(9)	9870 (5)	156 (5)	5773 (1)	73 (1)
C(9a)	8162 (8)	1115 (7)	5742 (2)	61 (2)
C(10)	8097 (9)	2432 (7)	5300 (2)	81 (2)
C(11)	6361 (11)	3386 (8)	5209 (3)	100 (3)
C(12)	4696 (10)	3097 (8)	5560 (3)	85 (2)
C(13)	4743 (8)	1791 (7)	5996 (2)	63 (2)
C(13a)	6494 (7)	781 (6)	6105 (2)	48 (2)
C(13b)	6454 (7)	−759 (6)	6539 (2)	48 (2)
C(5A)	6793 (7)	−370 (6)	7256 (2)	43 (1)
O(14)	3571 (5)	812 (6)	7381 (2)	75 (1)
C(15)	4856 (8)	2991 (8)	8330 (2)	67 (2)
C(16)	9184 (8)	3593 (6)	8468 (2)	59 (2)
C(17)	8335 (7)	455 (6)	9240 (2)	49 (2)
C(18)	6471 (8)	−202 (7)	9359 (2)	62 (2)
C(19)	5732 (9)	−36 (7)	9965 (3)	77 (2)
C(20)	6888 (9)	803 (8)	10444 (2)	79 (2)
C(21)	8730 (11)	1422 (8)	10335 (2)	76 (2)
C(22)	9494 (8)	1235 (7)	9741 (2)	62 (2)
C(23)	10035 (10)	−4697 (10)	6696 (3)	85 (2)
C(24)	6379 (9)	−4337 (10)	6921 (3)	79 (2)

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

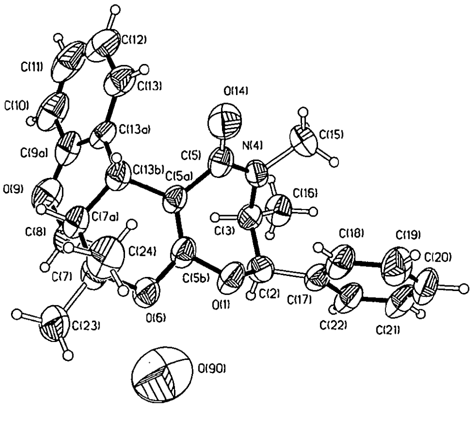

Fig. 1. Thermal ellipsoid plot of the molecule showing the atom numbering.

Related literature. For the preparation of the compound see Pfeiffer (1985). For the preparation of some related compounds see Tietze, Brand, Pfeiffer, Antel, Harms & Sheldrick (1987) and Pfeiffer (1988). For related structures see Antel, Sheldrick, Pfeiffer & Tietze (1988*a*,*b*).

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

C(5)—N(4)	1·356 (4)	C(5)—C(5a)	1·477 (5)
C(5)—O(14)	1·223 (5)	N(4)—C(3)	1·457 (5)
N(4)—C(15)	1·471 (6)	C(3)—C(2)	1·517 (6)
C(3)—C(16)	1·530 (6)	C(2)—O(1)	1·454 (5)
C(2)—C(17)	1·513 (6)	O(1)—C(5b)	1·365 (5)
C(5b)—O(6)	1·354 (5)	C(5b)—C(5a)	1·330 (6)
O(6)—C(7)	1·475 (5)	C(7)—C(7a)	1·531 (6)
C(7)—C(23)	1·540 (8)	C(7)—C(24)	1·496 (8)
C(7a)—C(8)	1·525 (7)	C(7a)—C(13b)	1·537 (7)
C(8)—O(9)	1·416 (6)	O(9)—C(9a)	1·352 (6)
C(9a)—C(10)	1·392 (7)	C(9a)—C(13a)	1·403 (7)
C(10)—C(11)	1·368 (9)	C(11)—C(12)	1·379 (10)
C(12)—C(13)	1·378 (8)	C(13)—C(13a)	1·405 (7)
C(13a)—C(13b)	1·521 (6)	C(13b)—C(5a)	1·528 (6)
C(17)—C(18)	1·369 (7)	C(17)—C(22)	1·390 (6)
C(18)—C(19)	1·395 (7)	C(19)—C(20)	1·380 (8)
C(20)—C(21)	1·341 (9)	C(21)—C(22)	1·379 (7)

N(4)—C(5)—C(5a)	116·9 (3)	N(4)—C(5)—O(14)	122·8 (3)
C(5a)—C(5)—O(14)	120·3 (3)	C(5)—N(4)—C(3)	121·5 (3)
C(5)—N(4)—C(15)	117·6 (3)	C(3)—N(4)—C(15)	120·9 (3)
N(4)—C(3)—C(2)	111·8 (3)	N(4)—C(3)—C(16)	112·9 (4)
C(2)—C(3)—C(16)	112·0 (3)	C(3)—C(2)—O(1)	111·7 (3)
C(3)—C(2)—C(17)	113·5 (4)	O(1)—C(2)—C(17)	106·2 (3)
C(2)—O(1)—C(5b)	121·0 (3)	O(1)—C(5b)—O(6)	106·4 (3)
O(1)—C(5b)—C(5a)	126·7 (4)	O(6)—C(5b)—C(5a)	126·7 (4)
C(5b)—O(6)—C(7)	114·6 (3)	O(6)—C(7)—C(7a)	108·3 (3)
O(6)—C(7)—C(23)	103·5 (4)	C(7a)—C(7)—C(23)	113·1 (4)
O(6)—C(7)—C(24)	108·6 (4)	C(7a)—C(7)—C(24)	112·3 (4)
C(23)—C(7)—C(24)	110·5 (5)	C(7)—C(7a)—C(8)	112·5 (4)
C(7)—C(7a)—C(13b)	109·9 (4)	C(8)—C(7a)—C(13b)	109·3 (4)
C(7a)—C(8)—O(9)	111·7 (4)	C(8)—O(9)—C(9a)	116·8 (4)
O(9)—C(9a)—C(10)	115·9 (5)	O(9)—C(9a)—C(13a)	123·2 (4)
C(10)—C(9a)—C(13a)	120·9 (5)	C(9a)—C(10)—C(11)	119·3 (5)
C(10)—C(11)—C(12)	121·4 (5)	C(11)—C(12)—C(13)	119·7 (6)
C(12)—C(13)—C(13a)	120·8 (5)	C(9a)—C(13a)—C(13)	117·9 (4)
C(9a)—C(13a)—C(13b)	121·5 (4)	C(13)—C(13a)—C(13b)	120·2 (4)
C(7a)—C(13b)—C(13a)	108·4 (4)	C(7a)—C(13b)—C(5a)	109·5 (3)
C(13a)—C(13b)—C(5a)	114·5 (4)	C(5)—C(5a)—C(5b)	119·6 (4)
C(5) C(5a) C(13b)	118·6 (4)	C(5b)—C(5a)—C(13b)	120·6 (4)
C(2)—C(17)—C(18)	121·8 (4)	C(2)—C(17)—C(22)	119·5 (4)
C(18)—C(17)—C(22)	118·6 (4)	C(17)—C(18)—C(19)	120·3 (5)
C(18)—C(19)—C(20)	119·6 (5)	C(19)—C(20)—C(21)	120·4 (5)
C(20)—C(21)—C(22)	120·5 (5)	C(17)—C(22)—C(21)	120·5 (5)

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Structure of a 2-Azaspiro[4.5]decadiene

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Abstract. Tetramethyl 3-amino-4-cyano-1-methylene-2-phenyl-2-azaspiro[4.5]deca-3,6-diene-7,8,9,10-tetracarboxylate, $C_{25}H_{25}N_3O_8$, $M_r = 495{\cdot}49$, monoclinic, $C2/c$, $a = 11{\cdot}148$ (5), $b = 17{\cdot}156$ (5), $c = 26{\cdot}939$ (5) Å, $\beta = 93{\cdot}99$ (4)°, $V = 5139{\cdot}7$ Å^3, $Z = 8$, $D_x = 1{\cdot}281$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 7{\cdot}724$ cm^{-1}, $F(000) = 2080$, $T = 293$ K. The final R value is 0·071 for 3376 significant [$I > 3\sigma(I)$] reflections. The title compound is one of the products obtained by thermally or photochemically induced cyclization of the 1:2 adduct of 2-amino-4,5-dimethyl-1-phenylpyrrole-3-carbonitrile and dimethyl acetylenedicarboxylate.

Experimental. The 2-azaspiro[4.5]decadiene crystallizes from chloroform as colorless plates as one of the products of the reaction reported by Eger, Folkers, Frey, Zimmermann & Koop-Kirfel (1988). A single crystal of approximate dimensions 0·10 × 0·40 × 0·40 mm was mounted on a glass fiber. The observed systematic absences determined the space group to be Cc or $C2/c$, the latter was confirmed by the subsequent refinement. In the final full-matrix least-squares refinement all non-H atoms were assigned anisotropic thermal parameters; the H atomic positions were calculated and included in the structure-factor calculation. However, due to crystal packing effects or disorder, there was no unique way of placing the methyl H atoms of the methoxy groups. The procedure applied to find those positions was to locate the methyl H atoms from a difference Fourier map (using low-angle reflections), which revealed at least one or more H of each group.

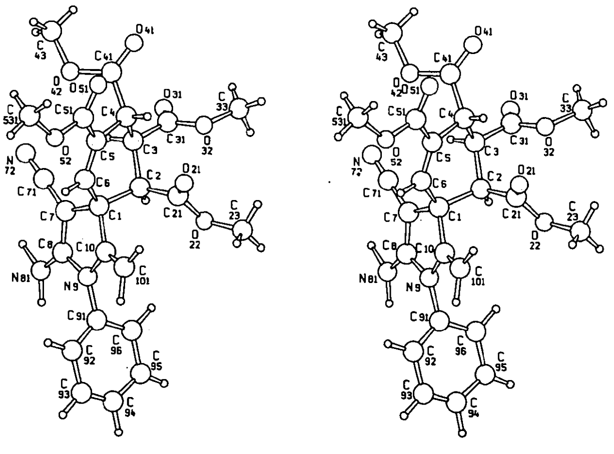

Fig. 1. Stereoview of the molecule with atom labeling using *SCHAKAL* (Keller, 1988).

Table 1. *Data-collection and structure-refinement parameters*

Crystal shape	Plate
Diffractometer used	CAD-4, Enraf–Nonius
Method of intensity measurement	$\theta/2\theta$
No. and θ range of reflections for lattice parameters	25; 9–26°
Method used for absorption correction	*DIFABS* (Walker & Stuart, 1983)
Minimum absorption correction	0·799
Maximum absorption correction	1·375
Average absorption correction	0·933
Maximum value of $(\sin\theta)/\lambda$ reached in intensity measurement (Å^{-1})	0·5877
Range of h, k and l	0→13, −20→20, −31→31
Standard reflections	$0,0,\overline{12}$, 4,0,10, $0,6,\overline{12}$
Interval, standard reflections measured	2h, no intensity variation
Total No. of reflections measured; θ range (°)	7853; 65 (1347 unobserved reflections)
No. of observed unique reflections	3376 with $I > 3\sigma(I)$
Methods used to solve structure	Direct methods (*SHELXS*; Sheldrick, 1985)
Use of F or F^2 in LS refinement	F
Method of locating H atoms	H positions calculated in idealized positions
Weighting scheme	$1/\sigma^2$
Parameters refined	335
Value of R	0·071
Value of wR	0·066
Ratio of max. LS shift to e.s.d. (Δ/σ)	0·001
Max. height in final ΔF map (e Å^{-3})	0·322
Error in an observation of unit weight	2·776
Secondary extinction coefficient	$9{\cdot}417(1) \times 10^{-7}$ (Zachariasen, 1963)
Sources of atomic scattering factors	*International Tables for X-ray Crystallography* (1974)
Computer used	MicroVAX 3500
Programs used	*VAXSDP* version 3.0 (1986) (Frenz, 1978)

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