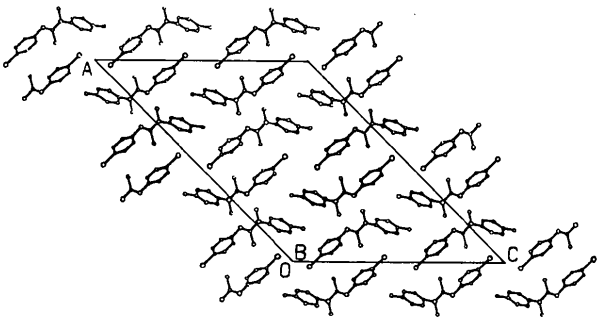

Fig. 2. Projection of the structure down **b**.

molecular overcrowding the phenyl rings are twisted relative to the plane of the amidine group by 90·1 (2) (*p*-bromophenyl) and 67·0 (2)° (*p*-tolyl), showing lack of conjugation between the amidine and the aromatic fragments.

Molecular packing is shown in Fig. 2. No intermolecular contacts significantly shorter than the sum of the van der Waals radii are observed.

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References

BELLUCCI, F., BERTOLASI, V., FERRETTI, V. & GILLI, G. (1985). *Acta Cryst.* C**41**, 544–546.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JASKÓLSKI, M. (1982). *Fourth Symposium on Organic Crystal Chemistry*, Poznań, September 1982, edited by Z. KAŁUSKI, pp. 70–71. A Mickiewicz Univ.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A**30**, 580–584.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NORRESTAM, R., MERTZ, S. & CROSSLAND, I. (1983). *Acta Cryst.* C**39**, 1554–1556.

OSZCZAPOWICZ, J., TYKARSKA, E., JASKÓLSKI, M. & KOSTURKIEWICZ, Z. (1986). *Acta Cryst.* C**42**, 1816–1818.

RACZYŃSKA, E., OSZCZAPOWICZ, J. & WALCZAK, M. (1985). *J. Chem. Soc. Perkin Trans.* 2, pp. 1087–1090.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

WINKLER, F. K. & DUNITZ, J. D. (1971). *J. Mol. Biol.* **59**, 169–182.

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Structures of Two Bicyclic Octanes

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Abstract. (3*S*,8*S*)-6-Benzyl-3-methoxy-6-aza-2-oxabicyclo[3.2.1]octane-8-carbonitrile (4), $C_{15}H_{18}N_2O_2$, $M_r = 258{\cdot}31$, m.p. 370–372 K, $[\alpha]_D^{19°C}$ −15·9° (c = 3·5 g dm^{-3} in chloroform), orthorhombic, $P2_12_12_1$, $a = 8{\cdot}116$ (1), $b = 9{\cdot}659$ (1), $c = 17{\cdot}526$ (2) Å, V = 1373·9 Å^3, $Z = 4$, $D_x = 1{\cdot}25$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 5·93 cm^{-1}, $F(000) = 552$, T = 295 K, final $R = 0{\cdot}078$, $wR = 0{\cdot}053$ for 1356 unique observed [$I > 2\sigma(I)$] reflections. (3*S*,8*S*)-6-Benzyl-8-chloro-3-methoxy-6-aza-2-oxabicyclo[3.2.1]octane (3), $C_{14}H_{18}ClNO_2$, $M_r = 267{\cdot}75$, m.p. 360–361 K, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}056$ (4), $b = 9{\cdot}622$ (2), c = 17·512 (3) Å, $V = 1357{\cdot}4$ Å^3, $Z = 4$, D_x = 1·31 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 2·31 cm^{-1}, $F(000) = 568$, $T = 295$ K, final $R = 0{\cdot}053$, $wR = 0{\cdot}037$ for 1745 unique ($I > 0$) reflections. The conformations of the two bicyclic octanes are nearly identical and are determined by geometric restrictions imposed by the two-atom bridge between C(1) and C(5). The pyranose rings adopt highly puckered distorted chair conformations, and the five-membered rings are halfway between envelope and twist forms with C(5) and C(8) out of the plane.

Introduction. In our program of heteroprostaglandin research, a route for the stereospecific synthesis of a chiral synthon (5) for 9-azaprostaglandins has been developed (Holzapfel, Koekemoer & Verdoorn, 1987). A crucial step in the synthesis involved the introduction of a nitrile group at C(8) of the bicyclic compound (1) as a one-carbon moiety required for completion of the prostaglandin ω side chain. Two compounds were isolated after substitution of the sulfonate group of (2) with cyanide. The major product was spectroscopically characterized as the nitrile (4) [stereochemistry at C(8) unknown], while the minor

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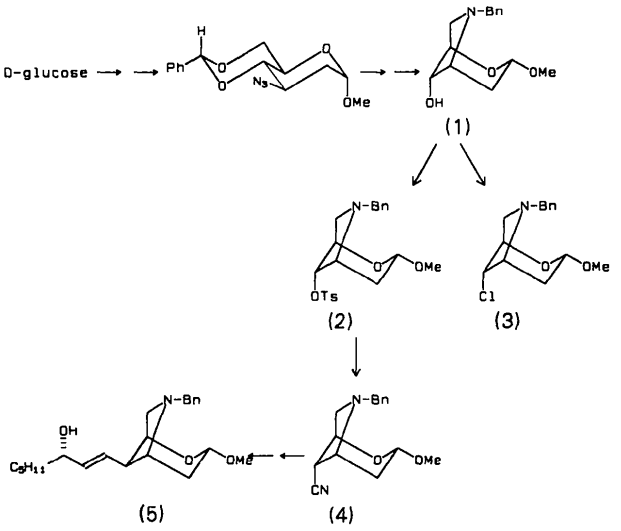
product, at first assumed to be the other C(8) epimer of (4), was characterized as the chloride (3) [stereochemistry at C(8) unknown]. Compound (3) was probably formed during the preparation of the tosylate (2) from (1) and carried along undetected since its mobility on thin-layer chromatography is the same as that of (2). Since the absolute configuration of C(8) of both compounds (3) and (4) could not be determined from the ^{1}H NMR data, X-ray crystallographic structure determinations were undertaken.



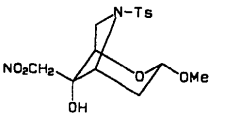
Experimental. The nitrile (4) was synthesized in sixteen steps from D-glucose (Holzapfel, Koekemoer & Verdoorn, 1987). The two final steps involved treatment of (1) with *p*-toluenesulfonyl chloride in pyridine to furnish (2) which was subsequently treated with potassium cyanide (5 equivalents) and hexamethylphosphoric triamide (5 equivalents) in dimethylformamide for 90 h at 383 K. Column chromatography of the crude reaction product (eluent: ethyl acetate–hexane, 1:1) furnished the nitrile (4) as the major component and the chloride (3) as a minor constituent. Compound (4) was recrystallized from ethyl acetate–hexane to give clear, colourless needles, m.p. 370–372 K. Analysis: calculated for $C_{15}H_{18}N_2O_2$: C 69·8, H 7·0%; found: C 70·0, H 7·1%. $[\alpha]_D^{19°C}$ −15·9° (c = 3·5 g dm^{-3} in chloroform), ν_{max} 2221 cm^{-1}, *m/e* 258 (M^+), ^{1}H NMR (500 MHz): δ = 1·92 (*ddd*, J = 13·6, 8·9, 1·8 Hz, 1H), 1·96 (*m*, J = 13·6, 6·8, 4·3, 1·6 Hz, 1H), 2·70 (*ddd*, J = 4·6, 2·4, 1·6 Hz, 1H), 2·85 (*dd*, J = 12·2, 4·1 Hz, 1H), 3·22 (*d*, J = 12·2 Hz, 1H), 3·49 (*s*, 3H), 3·56 (*m*, J = 4·6, 4·3, 1·8 Hz, 1H), 3·78 (*m*, 2H), 4·65 (*ddd*, J = 4·1, 2·4, 1·8 Hz, 1H), 4·90 (*dd*, J = 8·9, 6·8 Hz, 1H), 7·27–7·33 (*m*, 5H). Compound (3) was also crystallized from ethyl acetate–hexane to give clear, colourless prisms, m.p. 360–361 K. Analysis: calculated for $C_{14}H_{18}ClNO_2$: C 62·8, H 6·8, Cl 13·2%; found: C 62·5, H 6·9, Cl 12·9%. *m/e* 268 (M^+), ^{1}H NMR (500 MHz): δ = 1·82 (*m*, J = 13·3, 4·2 Hz, 1H), 2·09 (*ddd*, J = 13·3, 9·1, 1·5 Hz, 1H), 2·92 (*dd*, J = 11·9, 4·1 Hz, 1H), 3·21 (*d*, J = 11·9 Hz, 1H), 3·30 (*ddd*, J = 4·9, 4·2, 1·5 Hz, 1H), 3·51 (*s*, 3H), 3·84 (*m*, 2H), 3·99 (*dd*, J = 4·9, 2·8 Hz, 1H), 4·32 (*dd*, J = 4·1, 2·8 Hz, 1H), 4·92 (*dd*, J = 9·1, 4·2 Hz, 1H), 7·23–7·33 (*m*, 5H).

Unit-cell and intensity measurements were conducted according to standard procedures, with data taken on an Enraf–Nonius CAD-4 diffractometer. Experimental parameters are listed in Table 1. Both structures were solved by direct methods using *SHELXS*84 (Sheldrick, 1983). The absolute configuration was assigned from the known configuration at C(3). Scattering factors from *International Tables for X-ray Crystallography* (1974). The structures were refined by full-matrix least-squares procedures minimizing the function $\sum w(|F_o| - |F_c|)^2$ with $w = [\sigma(F)]^{-2}$. The full-matrix least-squares program *SHELX*76 (Sheldrick, 1976) was used. Since difference Fourier syntheses did not reveal the positions of all the H atoms, all H atoms were in both cases refined in calculated positions (C—H 1·08 Å), with common isotropic temperature factors. The methyl groups were refined as rigid groups free to rotate. Refinement parameters are given in Table 1.

Discussion. Final atomic coordinates and equivalent isotropic temperature factors for the non-H atoms are listed in Table 2.* Table 3 contains comparative data for selected bond distances and angles and Table 4 the relevant information describing the conformation of the bicyclic system. Fig. 1 shows perspective drawings of compounds (3) and (4), as well as the atomic labelling.

The crystal structures show that the substituents at C(8) are in both cases in the α position. Owing to angular strain, steric restrictions and neighbouring-group participation, the substitution reactions proceeded with retention of configuration *via* an S_N1 mechanism (Holzapfel, Koekemoer & Verdoorn, 1987).

The X-ray structure of a related bicyclic compound, (3*S*,8*R*)-8-hydroxy-3-methoxy-8-nitromethyl-6-*p*-toluenesulfonyl-6-aza-2-oxabicyclo[3.2.1]octane (6), was reported by Arndt, Boessenkool, Lourens & Boeyens (1981). In all three cases, (3), (4) and (6), the con-



(6)

* Lists of structure factors, H-atom parameters and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43817 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Experimental data and structure-refinement parameters*

	(4)	(3)
Crystal data		
Formula	$C_{15}H_{18}N_2O_2$	$C_{14}H_{18}ClNO_2$
Crystal habit	Colourless needles	Colourless prisms
Crystal size (mm)	0·40×0·25×0·25	0·40×0·40×0·40
Symmetry	Orthorhombic, $P2_12_12_1$	Orthorhombic, $P2_12_12_1$
Systematic absences	$h00, h \neq 2n$	$h00, h \neq 2n$
	$0k0, k \neq 2n$	$0k0, k \neq 2n$
	$00l, l \neq 2n$	$00l, l \neq 2n$
Unit-cell determination:		
least-squares fit to	25 reflections	25 reflections
θ range (°)	$16 < \theta < 48$	$7 < \theta < 20$
Experimental data		
Radiation	Cu $K\alpha$	Mo $K\alpha$
Monochromator	Graphite	Graphite
Collection mode	ω–2θ ($2\theta < 78°$)	ω–2θ ($2\theta < 28°$)
hkl range	$0 < h < 10$	$0 < h < 10$
	$0 < k < 12$	$0 < k < 12$
	$0 < l < 21$	$0 < l < 23$
Reflections measured	1716	1910
Reflections used in refinement	1356 with $I > 2\sigma$	1745 with $I > 0$
Stability	3 reflections, no variation	3 reflections, no variation
μ (cm^{-1})	5·9	2·3
Absorption correction	None	None
Solution and refinement		
No. of variables	176	167
Residual electron density (e $Å^{-3}$)	0·3	0·3
Max. Δ/σ	0·05	0·08
Common U_{iso} for H atoms ($Å^2$)	0·104 (5)	0·093 (3)
Final *R*, *wR*	0·078, 0·053	0·053, 0·037

Table 2. *Fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2 \times 10^3$) *for the non-*H *atoms*

	x	*y*	*z*	U_{eq}*
Compound (3)				
C(1)	140 (5)	6316 (4)	7098 (2)	57 (2)
O(2)	639 (3)	7696 (2)	6898 (1)	61 (1)
C(3)	1156 (5)	7824 (4)	6109 (2)	60 (2)
C(4)	−125 (5)	7210 (4)	5566 (2)	60 (2)
C(5)	−610 (4)	5750 (4)	5847 (2)	53 (2)
N(6)	937 (4)	4965 (3)	6010 (2)	52 (2)
C(7)	1367 (5)	5199 (4)	6838 (2)	56 (2)
C(8)	−1364 (4)	5863 (4)	6641 (2)	58 (2)
Cl	−3124 (1)	7021 (1)	6727 (1)	74 (1)
O(11)	1309 (4)	9244 (3)	5964 (2)	74 (2)
C(12)	2789 (6)	9826 (4)	6287 (3)	77 (3)
C(13)	850 (5)	3477 (4)	5812 (2)	61 (2)
C(14)	1002 (4)	3245 (4)	4959 (2)	52 (2)
C(15)	1728 (5)	4221 (4)	4477 (2)	59 (2)
C(16)	1879 (5)	3961 (4)	3703 (2)	73 (3)
C(17)	1304 (5)	2720 (5)	3401 (2)	78 (3)
C(18)	605 (5)	1736 (5)	3877 (3)	73 (3)
C(19)	445 (4)	1974 (4)	4662 (2)	65 (3)
Compound (4)				
C(1)	254 (7)	6237 (6)	7127 (3)	61 (4)
O(2)	666 (5)	7645 (4)	6934 (2)	65 (2)
C(3)	1202 (9)	7797 (6)	6146 (3)	65 (4)
C(4)	−36 (7)	7148 (5)	5595 (3)	62 (4)
C(5)	−443 (7)	5671 (6)	5868 (3)	56 (3)
N(6)	1116 (6)	4929 (4)	6032 (2)	53 (3)
C(7)	1530 (7)	5169 (6)	6854 (3)	58 (3)
C(8)	−1249 (7)	5753 (6)	6670 (3)	58 (3)
C(9)	−2690 (9)	6684 (8)	6715 (4)	76 (5)
N(10)	−3830 (8)	7395 (6)	6730 (4)	100 (5)
O(11)	1297 (6)	9212 (4)	6008 (2)	76 (3)
C(12)	2798 (8)	9817 (7)	6297 (4)	79 (4)
C(13)	1055 (8)	3446 (6)	5853 (3)	62 (4)
C(14)	1098 (7)	3178 (6)	5001 (3)	54 (3)
C(15)	1713 (7)	4140 (6)	4491 (3)	67 (4)
C(16)	1764 (9)	3862 (8)	3711 (3)	84 (5)
C(17)	1187 (10)	2602 (8)	3446 (4)	85 (5)
C(18)	609 (8)	1622 (8)	3951 (4)	80 (5)
C(19)	550 (7)	1908 (7)	4731 (3)	69 (4)

* U_{eq} is defined as the geometric mean of the diagonal elements of the diagonalized matrix of U_{ij}.

Table 3. *Bonding parameters*

	(3)	(4)
(*a*) Bond lengths (Å)		
C(1)–O(2)	1·431 (4)	1·441 (6)
C(1)–C(7)	1·530 (5)	1·538 (7)
C(1)–C(8)	1·516 (4)	1·533 (7)
O(2)–C(3)	1·448 (4)	1·455 (6)
C(3)–C(4)	1·522 (5)	1·528 (7)
C(3)–O(11)	1·395 (4)	1·390 (6)
C(4)–C(5)	1·540 (4)	1·540 (6)
C(5)–N(6)	1·485 (4)	1·482 (6)
C(5)–C(8)	1·521 (4)	1·552 (6)
N(6)–C(7)	1·506 (4)	1·498 (5)
N(6)–C(13)	1·475 (4)	1·467 (6)
C(8)–Cl/C(9)	1·809 (3)	1·478 (8)
C(9)–N(10)	—	1·152 (8)
O(11)–C(12)	1·434 (5)	1·443 (6)
C(13)–C(14)	1·517 (4)	1·516 (6)
C(14)–C(15)	1·391 (4)	1·383 (6)
C(14)–C(19)	1·402 (4)	1·388 (6)
C(15)–C(16)	1·384 (5)	1·394 (7)
C(16)–C(17)	1·386 (5)	1·384 (8)
C(17)–C(18)	1·382 (5)	1·378 (9)
C(18)–C(19)	1·400 (5)	1·395 (7)
(*b*) Bond angles (°)		
O(2)–C(1)–C(7)	113·4 (3)	113·8 (5)
O(2)–C(1)–C(8)	111·2 (3)	110·5 (5)
C(7)–C(1)–C(8)	99·0 (3)	99·6 (4)
C(1)–O(2)–C(3)	113·2 (3)	112·8 (4)
O(2)–C(3)–C(4)	111·6 (3)	111·2 (5)
O(2)–C(3)–O(11)	106·4 (3)	106·4 (5)
C(4)–C(3)–O(11)	109·0 (3)	109·3 (5)
C(3)–C(4)–C(5)	109·1 (3)	109·0 (4)
C(4)–C(5)–N(6)	108·2 (3)	109·0 (4)
C(4)–C(5)–C(8)	109·2 (3)	108·9 (4)
N(6)–C(5)–C(8)	101·3 (3)	102·1 (4)
C(5)–N(6)–C(7)	107·6 (3)	107·6 (4)
C(5)–N(6)–C(13)	114·1 (3)	113·7 (5)
C(7)–N(6)–C(13)	112·5 (3)	111·3 (4)
C(1)–C(7)–N(6)	104·1 (3)	104·7 (4)
C(1)–C(8)–C(5)	100·6 (3)	98·9 (4)
C(1)–C(8)–Cl/C(9)	113·9 (2)	114·5 (5)
C(5)–C(8)–Cl/C(9)	115·7 (2)	114·4 (5)
C(3)–O(11)–C(12)	112·6 (3)	112·6 (5)
N(6)–C(13)–C(14)	111·8 (3)	112·1 (5)
C(13)–C(14)–C(15)	122·1 (3)	122·1 (5)
C(13)–C(14)–C(19)	117·9 (4)	118·7 (5)
C(15)–C(14)–C(19)	119·9 (3)	119·2 (5)
C(14)–C(15)–C(16)	120·6 (4)	121·1 (6)
C(15)–C(16)–C(17)	120·0 (4)	119·2 (7)
C(16)–C(17)–C(18)	119·7 (4)	120·2 (7)
C(17)–C(18)–C(19)	121·2 (4)	120·3 (7)
C(14)–C(19)–C(18)	118·5 (4)	119·9 (6)
C(8)–C(9)–N(10)	—	177·9 (8)

Table 4. *Parameters describing the conformation of the five- and six-membered rings*

	(3)	(4)
(*a*) Endocyclic torsion angles (°)		
C(8)–C(1)–O(2)–C(3)	−62·8 (4)	−65·0 (6)
C(1)–O(2)–C(3)–C(4)	50·6 (4)	51·7 (6)
O(2)–C(3)–C(4)–C(5)	−49·0 (4)	−49·7 (6)
C(3)–C(4)–C(5)–C(8)	60·8 (4)	61·9 (6)
C(4)–C(5)–C(8)–C(1)	−67·4 (3)	68·6 (5)
C(5)–C(8)–C(1)–O(2)	68·5 (3)	70·0 (5)
C(8)–C(1)–C(7)–N(6)	36·2 (4)	36·0 (6)
C(1)–C(7)–N(6)–C(5)	−8·0 (4)	−7·0 (6)
C(7)–N(6)–C(5)–C(8)	−23·5 (4)	−24·5 (6)
N(6)–C(5)–C(8)–C(1)	46·6 (3)	46·5 (5)
C(5)–C(8)–C(1)–C(7)	−51·1 (3)	−50·0 (5)
(*b*) Puckering parameters (Cremer & Pople, 1975)		
Ring [O(2)–C(3)–C(4)–C(5)–C(8)–C(1)]		
φ (°)	52 (1)	54 (2)
θ (°)	165·5 (3)	164 (2)
Q (Å)	0·632 (3)	0·656 (6)
Conformation	$^4C_1$	$^4C_1$
Ring [N(6)–C(7)–C(5)–C(8)–C(1)]		
$\varphi(2)$ (°)	98·7 (4)	99 (1)
$q(2)$ (Å)	0·509 (4)	0·514 (6)
Conformation	$^3T_4$–E_4	$^3T_4$–E_4

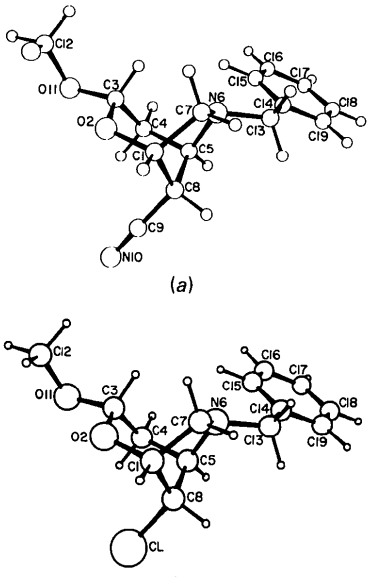


Fig. 1. Perspective drawings of (*a*) compound (4) and (*b*) compound (3), also showing the atomic labelling.

formations of the bicyclic octanes are similar and determined by geometric restrictions imposed by the two-atom bridge between C(1) and C(5). The pyranose rings assume highly puckered distorted chair conformations with rather large endocyclic torsion angles about the C(8)–C(1) and C(8)–C(5) bonds (the effect of which on the related dihedral angles precluded the unambiguous interpretation of the coupling-constant data). The five-membered rings adopt conformations halfway between the envelope and twist forms with C(5) and C(8) out of the plane. Since both the five- and six-membered rings are highly puckered, some of the endocyclic valence angles are rather small (*cf.* Table 3). Except for the C(3)–O(11) bonds which in both compounds (3) and (4) are significantly shorter than the C(3)–O(2), C(1)–O(2) and C(12)–O(11) bonds (*cf.* Table 3), there are no unusual bond distances. The shortening of the C(3)–O(11) bonds is in accordance with the operation of an *exo* anomeric effect (Fuchs, Schleifer & Tartakovsky, 1984).

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References

ARNDT, R. R., BOESSENKOOL, I. K., LOURENS, G. J. & BOEYENS, J. C. A. (1981). *S. Afr. J. Chem.* **34**, 1–7.

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

FUCHS, B., SCHLEIFER, L. & TARTAKOVSKY, E. (1984). *Nouv. J. Chim.* **8**, 275–278.

HOLZAPFEL, C. W., KOEKEMOER, J. M. & VERDOORN, G. H. (1987). *S. Afr. J. Chem.* Submitted.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1983). *SHELXS*84. Direct-methods program (preliminary version). Univ. of Göttingen.

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Structure of a Tetrahydrobenzothieno[3,2-*e*][1,2]oxazine

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Abstract. 2*H*-2-Cyclohexyl-4β-methyl-3,4,4aα,9aα-tetrahydrobenzothieno[3,2-*e*][1,2]oxazine-3α-carbonitrile, $C_{18}H_{22}N_2OS$, $M_r = 314{\cdot}45$, m.p. 425–427 K, monoclinic, $C2/c$, $a = 26{\cdot}97\ (1)$, $b = 6{\cdot}378\ (3)$, $c = 19{\cdot}307\ (7)$ Å, $\beta = 91{\cdot}30\ (3)°$, $V = 3321{\cdot}2$ Å^3, $Z = 8$, $D_x = 1{\cdot}25$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ(Mo $K\alpha$) = 1·58 cm^{-1}, $F(000) = 1344$, $T = 298$ K, final $R = 0{\cdot}069$ for 2118 unique observed [$I > 4\sigma(I)$] reflections. This compound is the first reported example of a crystal structure of a 1,2-oxazine *cis* fused to a dihydrothiophene and of a 1,2-oxazine with this specific stereochemistry of the cyano and methyl substituents. The 1,2-oxazine ring has a highly puckered twist-boat conformation with the *N*-cyclohexyl and methyl groups equatorial and the cyano group in a bisectional position. The endocyclic torsion angle about O(1)–N(2) is exceptionally large [84·5 (3)°].

Introduction. This paper forms part of a series on the structure of 3-cyano-2-(cyclohexyl)tetrahydro-1,2-oxazine ring systems. The structures reported previously (some studied by NMR spectroscopy and some by

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