Table 2. *Bond lengths* (Å) *and angles* (°) *with their e.s.d.'s in parentheses*

N(1)–C(2)	1·266 (3)	C(7)–C(8)	1·498 (3)
N(1)–C(8a)	1·472 (3)	C(8)–C(8a)	1·529 (3)
C(2)–O(3)	1·354 (2)	C(9)–C(10)	1·394 (3)
C(2)–C(9)	1·486 (3)	C(9)–C(14)	1·391 (3)
O(3)–C(4)	1·441 (3)	C(10)–C(11)	1·391 (3)
C(4)–C(4a)	1·509 (3)	C(11)–C(12)	1·377 (3)
C(4a)–C(5)	1·523 (3)	C(12)–C(13)	1·379 (3)
C(4a)–C(8a)	1·519 (3)	C(12)–Cl(15)	1·748 (2)
C(5)–C(6)	1·490 (3)	C(13)–C(14)	1·374 (3)
C(6)–C(7)	1·315 (4)		

C(2)–N(1)–C(8a)	117·7 (3)	N(1)–C(8a)–C(4a)	110·7 (3)
N(1)–C(2)–O(3)	128·2 (3)	N(1)–C(8a)–C(8)	109·9 (3)
N(1)–C(2)–C(9)	120·8 (3)	C(4a)–C(8a)–C(8)	112·0 (3)
O(3)–C(2)–C(9)	111·0 (3)	C(2)–C(9)–C(10)	121·5 (3)
C(2)–O(3)–C(4)	117·1 (3)	C(2)–C(9)–C(14)	119·5 (3)
O(3)–C(4)–C(4a)	111·3 (3)	C(10)–C(9)–C(14)	119·0 (3)
C(4)–C(4a)–C(5)	111·7 (3)	C(9)–C(10)–C(11)	119·9 (3)
C(4)–C(4a)–C(8a)	107·1 (3)	C(10)–C(11)–C(12)	119·3 (3)
C(5)–C(4a)–C(8a)	111·9 (3)	C(11)–C(12)–C(13)	121·9 (3)
C(4a)–C(5)–C(6)	112·5 (3)	C(11)–C(12)–Cl(15)	119·3 (3)
C(5)–C(6)–C(7)	123·1 (4)	C(13)–C(12)–Cl(15)	118·7 (3)
C(6)–C(7)–C(8)	124·1 (4)	C(12)–C(13)–C(14)	118·3 (3)
C(7)–C(8)–C(8a)	113·2 (3)	C(9)–C(14)–C(13)	121·6 (3)

Related literature. The synthesis and ^{1}H and ^{13}C NMR studies of this and other related compounds are discussed by Bernáth, Stájer, Szabó, Fülöp & Sohár (1985). The structure determination of a related compound with a *trans* ring junction is reported in Argay, Kálmán, Ribár & Bernáth (1986).

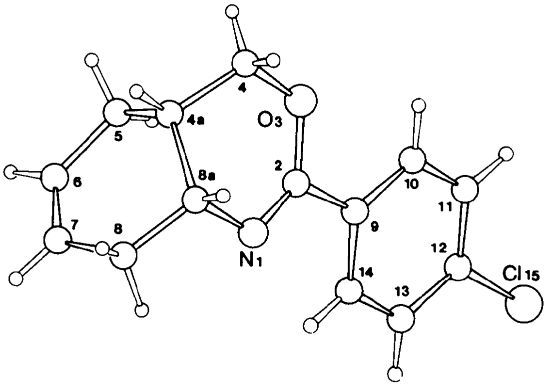

Fig. 1. A perspective view of the molecule with the numbering scheme. Numbers refer to C atoms unless otherwise indicated.

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Stereochemical Studies. 115.* Structure of *trans*-2-(*p*-Chlorophenyl)-1,2,4a,5,8,8a-hexahydro-4*H*-3,1-benzoxazine

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Abstract. $C_{14}H_{16}ClNO$, $M_r = 249\cdot74$, orthorhombic, $P2_12_12_1$, $a = 16\cdot725\ (4)$, $b = 12\cdot833\ (4)$, $c = 5\cdot939\ (1)$ Å, $V = 1274\cdot7\ (9)$ Å^3, $Z = 4$, $D_x = 1\cdot301$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 0\cdot28$ mm^{-1}, $F(000) = 528$, $T = 293\ (2)$ K, $R = 0\cdot061$ for 1063 unique observed reflections. The opposite signs of the endocyclic torsion angles about the C—C bond in the ring junction substantiate the stereospecificity of the reaction in which a *trans*-amino alcohol (obtained by reduction from *trans*-2-amino-

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* Part 114: Kálmán, Argay, Bernáth & Stájer (1986).

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4-cyclohexene-1-carboxylic acid) is cyclized by an aldehyde (p-ClC$_6$H$_4$CHO) to bicyclic hexahydro-4*H*-3,1-benzoxazine. Accordingly, the only diastereomer formed is that in which the H atoms bound to C atoms adjacent to N are equally in axial positions, with the *p*-chlorophenyl ring fixed equatorially. The cyclohexene ring assumes an almost perfect half-chair shape with a local pseudo-twofold symmetry axis bisecting the C(4a)—C(8a) bond at the *trans* ring junction. It has, however, no effect on the hetereo ring which exhibits a chair conformation.

Experimental. Colourless crystal (0·15 × 0·2 × 0·4 mm) of the title compound mounted on a glass fiber by its long axis. Syntex $P\bar{1}$ diffractometer, graphite-monochromated Mo $K\bar{\alpha}$ radiation. $0·059 \leq \sin\theta/\lambda \leq 0·595$ Å^{-1}. ω–2θ scan, h 0 to 19, k 0 to 15 and l 0 to 7. Cell parameters by least-squares fit for 15 centred reflections. Systematic absences: $h = 2n + 1$ in $h00$, $k = 2n + 1$ in $0k0$ and $l = 2n + 1$ in $00l$. Of 1309 unique reflections 246 with $I < \sigma(I)$ were taken as unobserved. No absorption correction. Three standard reflections, intensity variation ±3%. The phases for 306 reflections having $E \geq 1·20$ were obtained with *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The *E* map computed with the best figures of merit (ABS FOM 1·59, PSIZERO 2·40, RESID 42·6) revealed the positions of all non-H atoms. The H atom bound to N was located in a $\Delta\rho$ map. The other H-atom positions were generated from assumed geometries and were only included with a mean isotropic temperature factor (4·0 Å^2) in structure-factor calculations. The full-matrix least-squares procedure minimized $\sum w(\Delta F)^2$ using the weighting scheme $w = [\sigma^2(F_o) + 0·25(pF_o)^2]^{-1}$ with $p = 0·01$ for 155 parameters. Final $R = 0·061$, $wR = 0·054$, $R_{\text{tot}} = 0·078$ and $S = 0·97$. Extinction coefficient (Zachariasen, 1963): $1·2742 \times 10^{-6}$. Max. peak height in final $\Delta\rho$ map 0·3 e Å^{-3}. Max. Δ/σ 0·36. Scattering factors from *International Tables for X-ray Crystallography* (1962). Program system applied: Enraf–Nonius (1982) *SDP* with local modification adapted to a PDP11/34 minicomputer (64 K). The structure is defined in Tables 1 and 2 and Fig. 1.*

Related literature. The synthesis and ^{1}H and ^{13}C NMR studies of this and other related compounds are discussed by Bernáth, Stájer, Szabó, Fülöp & Sohár (1985). The structure determination of a related compound with a *cis* ring junction is reported in Kálmán, Argay, Bernáth & Stájer (1986).

Table 1. *Atomic coordinates and equivalent isotropic temperature factors for the non-*H *atoms with e.s.d.'s in parentheses*

$B_{\text{eq}} = \frac{4}{3}\text{trace}(B\mathbf{G})$ where **G** is the direct metric tensor.

	x	y	z	B_{eq}(Å^2)
N(1)	0·3827 (3)	−0·1671 (3)	−0·1369 (8)	3·5 (2)
C(2)	0·3247 (3)	−0·1314 (5)	0·0260 (11)	3·5 (2)
O(3)	0·3164 (2)	−0·1975 (3)	0·2166 (7)	4·1 (2)
C(4)	0·2939 (3)	−0·3002 (5)	0·1506 (11)	4·3 (3)
C(4a)	0·3567 (3)	−0·3482 (5)	−0·0050 (10)	3·6 (2)
C(5)	0·3351 (4)	−0·4585 (5)	−0·0792 (12)	5·2 (3)
C(6)	0·3858 (5)	−0·4918 (5)	−0·2687 (13)	5·7 (3)
C(7)	0·4306 (4)	−0·4306 (5)	−0·3948 (11)	5·3 (3)
C(8)	0·4338 (4)	−0·3146 (5)	−0·3598 (10)	4·3 (3)
C(8a)	0·3684 (3)	−0·2754 (4)	−0·2065 (11)	3·8 (2)
C(9)	0·3478 (3)	−0·0232 (4)	0·1079 (10)	3·1 (2)
C(10)	0·3844 (3)	−0·0075 (4)	0·3138 (10)	3·6 (2)
C(11)	0·4080 (4)	0·0914 (4)	0·3795 (10)	4·0 (3)
C(12)	0·3921 (3)	0·1730 (4)	0·2378 (11)	3·9 (3)
C(13)	0·3547 (4)	0·1607 (5)	0·0322 (12)	4·3 (3)
C(14)	0·3318 (3)	0·0596 (5)	−0·0302 (11)	3·8 (3)
Cl(15)	0·4204 (1)	0·2995 (1)	0·3171 (4)	5·9 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *with their e.s.d.'s in parentheses*

N(1)—C(2)	1·445 (8)	C(7)—C(8)	1·504 (9)
N(1)—C(8a)	1·470 (7)	C(8)—C(8a)	1·509 (9)
C(2)—O(3)	1·421 (8)	C(9)—C(10)	1·382 (8)
C(2)—C(9)	1·521 (8)	C(9)—C(14)	1·369 (8)
O(3)—C(4)	1·426 (7)	C(10)—C(11)	1·385 (7)
C(4)—C(4a)	1·529 (8)	C(11)—C(12)	1·369 (8)
C(4a)—C(5)	1·526 (9)	C(12)—C(13)	1·381 (9)
C(4a)—C(8a)	1·531 (9)	C(12)—Cl(15)	1·755 (5)
C(5)—C(6)	1·473 (10)	C(13)—C(14)	1·403 (9)
C(6)—C(7)	1·319 (10)		

C(2)—N(1)—C(8a)	112·3 (8)	N(1)—C(8a)—C(4a)	112·2 (8)
N(1)—C(2)—O(3)	114·2 (8)	N(1)—C(8a)—C(8)	111·5 (8)
N(1)—C(2)—C(9)	109·5 (8)	C(4a)—C(8a)—C(8)	111·1 (8)
O(3)—C(2)—C(9)	108·4 (8)	C(2)—C(9)—C(10)	121·9 (8)
C(2)—O(3)—C(4)	111·0 (7)	C(2)—C(9)—C(14)	117·9 (9)
O(3)—C(4)—C(4a)	110·9 (8)	C(10)—C(9)—C(14)	120·2 (9)
C(4)—C(4a)—C(5)	112·7 (9)	C(9)—C(10)—C(11)	120·6 (9)
C(4)—C(4a)—C(8a)	108·3 (8)	C(10)—C(11)—C(12)	118·2 (9)
C(5)—C(4a)—C(8a)	111·8 (8)	C(11)—C(12)—C(13)	122·9 (10)
C(4a)—C(5)—C(6)	110·7 (10)	C(11)—C(12)—Cl(15)	119·3 (8)
C(5)—C(6)—C(7)	126·0 (12)	C(13)—C(12)—Cl(15)	117·7 (8)
C(6)—C(7)—C(8)	122·1 (11)	C(12)—C(13)—C(14)	117·6 (10)
C(7)—C(8)—C(8a)	112·8 (9)	C(9)—C(14)—C(13)	120·4 (10)

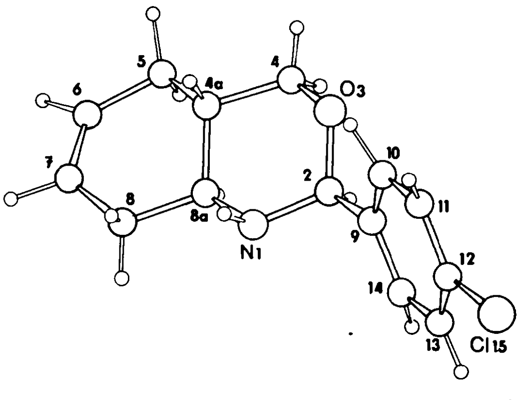

Fig. 1. A perspective view of the molecule with the numbering scheme. Numbers refer to C atoms unless otherwise indicated.

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* Lists of structure factors, anisotropic thermal parameters, bond lengths involving H atoms, torsion angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43245 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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endo-3,8-Dioxatricyclo[3.2.1.$0^{2,4}$]octane-6-carbonitrile

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Abstract. $C_7H_7NO_2$, $M_r = 137{\cdot}14$, $P2_1/c$, monoclinic, $a = 8{\cdot}572\ (4)$, $b = 8{\cdot}693\ (4)$, $c = 11{\cdot}706\ (5)$ Å, $\beta = 131{\cdot}93\ (6)°$, $V = 649\ (2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}41\ (3)$, $D_x = 1{\cdot}40$ Mg m^{-3}, Cu $K\bar{\alpha}$, $\lambda = 1{\cdot}5418$ Å, $\mu = 0{\cdot}88$ mm^{-1}, $F(000) = 288$, $T = 293\ (2)$ K, final $R = 0{\cdot}082$ for 979 observed reflections. The boat conformation of the six-membered ring is not distorted by the cyano group, the dihedral angles C(3)—C(4)—C(5)—C(6) and C(3)—C(2)—C(1)—C(6) being equal to 0·0 (5)°. Short intermolecular distances are observed between the H atoms of the molecule and the nucleophilic O and N atoms of the surrounding molecules. All the H atoms except H(51) and H(52) are electrophilic. These interactions are responsible for packing, the density of this compound being significantly higher than the density ($\simeq 1{\cdot}1$ Mg m^{-3}) of other cyano compounds [Viani, Cossu & Lapasset (1981). *Acta Cryst.* B**37**, 484–486].

Experimental. The two stereoisomers (*endo* and *exo*) were prepared by epoxidation of 5-cyano-7-oxabicyclo[2.2.1]hept-2-ene isomers (Kienzle, 1975) and separated by thin-layer chromatography. Crystal size 0·21 × 0·35 × 0·36 mm. D_m measured by flotation. Enraf–Nonius CAD-3 diffractometer used in the θ–2θ scan mode. No absorption correction. $P2_1/c$ symmetry and cell parameters determined by preliminary Weissenberg pictures. Standard reflection: 132 stable throughout experiment (intensity variation < 8%), monitored after every 60 reflections. 1106 measured reflections and 979 observed with $I > 2{\cdot}5\sigma(I)$. h 0–15, k 0–11, l −15–15. $\theta_{max} = 65°$. Structure solved by direct methods (*MULTAN78*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). The E map based on phases corresponding to the best figure of merit yielded all the non-H atoms. Structure refined by full-matrix least squares with *SHELX76* (Sheldrick, 1976), $\sum w(\Delta F)^2$ minimized. All the H atoms revealed by difference Fourier synthesis were introduced in the

Table 1. *Final fractional coordinates* (× 10^4) *and isotropic thermal parameters for the non-*H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}(Å^2)
C(1)	4794 (4)	3160 (3)	−2381 (3)	4·7 (2)
C(2)	4988 (4)	3026 (3)	−1066 (3)	4·4 (2)
C(3)	7148 (4)	3719 (3)	230 (3)	4·4 (2)
C(4)	8750 (4)	2494 (3)	592 (3)	4·5 (2)
C(5)	8500 (4)	2634 (3)	−845 (3)	4·9 (2)
C(6)	6830 (5)	3912 (3)	−1735 (3)	4·8 (2)
O(7)	7335 (3)	4855 (2)	−537 (2)	5·1 (2)
O(8)	3538 (3)	4111 (2)	−2249 (2)	5·6 (2)
C(9)	8376 (4)	941 (3)	848 (3)	4·6 (2)
N(10)	8064 (4)	−270 (3)	1031 (3)	5·7 (2)

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