SHELXTL, Sheldrick (1984). Table 1* lists final atomic coordinates and Table 2 bond lengths and angles. Fig. 1 shows the molecular structure and Fig. 2 shows a packing diagram.

Related literature. Bertinsson (1983) has reported the structures of the chloronickel and iodonickel analogues of the cation as tetraphenylborate salts; these analogues and the title complex are structurally very similar.

This investigation was supported by a Royal Society University Research Fellowship (CEH) and the Hariri Foundation (BAMS).

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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 52867 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1990). **C46**, 1551–1553

Structure at 173 K of a Chiral, Tricyclic Aminopyranone

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(*Received* 27 *October* 1989; *accepted* 12 *February* 1990)

Abstract. *Chemical Abstracts* name {3a*S*-[3aα,4(5*R**,6*R**),4aβ,7aβ,7bα]}-4-(decahydro-1*H*-dicyclopenta[*b*,*d*]pyrrol-4-yl)-5,6-dihydro-5-methyl-6-(1-methylethyl)-2*H*-pyran-2-one, $C_{19}H_{29}NO_2$, $M_r = 303{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 13{\cdot}187\,(4)$, $b = 13{\cdot}144\,(4)$, $c = 9{\cdot}668\,(3)$ Å, $V = 1675{\cdot}8\,(9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}203$ g cm^{-3}, $\mu = 0{\cdot}72$ cm^{-1}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $F(000) = 664$, $T = 173$ K, $R = 0{\cdot}0418$ for 1617 reflections [$F_o \geq 6\sigma(F_o)$]. Crystal chirality was assigned to correspond to the known chirality of the parent amine. All five-membered rings in the title compound exhibit envelope conformations with atom C(4) occupying the flap position for ring 1 (E^4), and rings 2 (E^6) and 3 (E^6) sharing the same flap position [C(6)]. Ring 3 adopts a conformation different from the E^7 conformation of similar tricyclic amine compounds studied in this laboratory. The short N—C(11) and C(12)—C(13) bonds [1·350 (3) Å and 1·436 (3) Å, respectively] and the long C(11)—C(12) double bond [1·372 (3) Å] indicate an extended conjugation along all bonds between N and O(2). Such conjugation is also supported by the small torsion angles of C(1)—N—C(11)—C(12) [−1·1 (3)°] and C(10)—N—C(11)—C(15) [3·2 (3)°]. The N atom is slightly outside the plane of C(1), C(10) and C(11) [0·036 (5) Å]. The molecules pack in a head-to-tail arrangement along a 2_1 screw axis.

Experimental. Crystals of the title compound were obtained from a chloroform/ethyl acetate solution, and had m.p. 483–484 K and $[\alpha]_D^{25^\circ\mathrm{C}} + 134^\circ$ (ethanol, $c = 0{\cdot}69$). The data crystal had dimensions $0{\cdot}12 \times 0{\cdot}17 \times 0{\cdot}22$ mm. A Nicolet *R3m/V* diffractometer equipped with a graphite monochromator and a Nicolet LT-2 low-temperature delivery system (173 K) was used to collect the data. The lattice parameters were obtained from the least-squares refinement of 50 reflections with $22{\cdot}01 < 2\theta < 28{\cdot}65^\circ$. The data were collected using the ω-scan technique with a 2θ range 3·0–55·0° and a 1·0° ω scan at 3–6° min^{-1} ($h = 0 \rightarrow 16$, $k = -16 \rightarrow 16$, $l = -11 \rightarrow 0$). Of the 3728 reflections measured, 1888 were unique, with $R_{\mathrm{int}} = 0{\cdot}012$ from averaging symmetry-equivalent reflections. Four reflections (302, $\bar{1}2\bar{2}$, 032, $\bar{6}\bar{4}2$) were remeasured every 96 reflections to monitor instrument and crystal stability

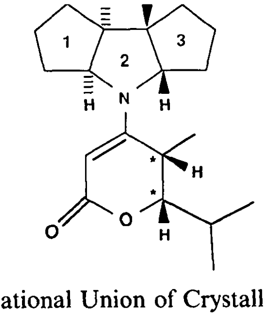

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Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}(Å^2)
O(1)	0·6171 (1)	0·3726 (1)	0·2153 (2)	0·029 (1)
O(2)	0·5866 (1)	0·2409 (1)	0·0799 (2)	0·041 (1)
N	0·3071 (1)	0·4367 (1)	0·2334 (2)	0·021 (1)
C(1)	0·2304 (2)	0·3642 (2)	0·1820 (2)	0·022 (1)
C(2)	0·2239 (2)	0·2634 (2)	0·2654 (3)	0·032 (1)
C(3)	0·1227 (2)	0·2670 (2)	0·3431 (3)	0·030 (1)
C(4)	0·0553 (2)	0·3352 (2)	0·2544 (3)	0·030 (1)
C(5)	0·1279 (2)	0·4177 (2)	0·2027 (2)	0·023 (1)
C(6)	0·1489 (2)	0·5016 (2)	0·3100 (2)	0·023 (1)
C(7)	0·0872 (2)	0·5985 (2)	0·2888 (3)	0·030 (1)
C(8)	0·1476 (3)	0·6615 (3)	0·1955 (7)	0·137 (2)
C(9)	0·2518 (2)	0·6202 (2)	0·1782 (3)	0·032 (1)
C(10)	0·2599 (2)	0·5315 (1)	0·2840 (2)	0·023 (1)
C(11)	0·4073 (2)	0·4171 (2)	0·2225 (2)	0·021 (1)
C(12)	0·4441 (2)	0·3289 (2)	0·1656 (3)	0·026 (1)
C(13)	0·5506 (2)	0·3095 (2)	0·1492 (3)	0·029 (1)
C(14)	0·5769 (2)	0·4366 (2)	0·3247 (2)	0·024 (1)
C(15)	0·4840 (2)	0·4950 (2)	0·2718 (2)	0·021 (1)
C(16)	0·6644 (2)	0·5026 (2)	0·3747 (2)	0·027 (1)
C(17)	0·7580 (2)	0·4391 (2)	0·4092 (3)	0·043 (1)
C(18)	0·6330 (2)	0·5656 (2)	0·5000 (3)	0·036 (1)
C(19)	0·5105 (2)	0·5695 (2)	0·1549 (3)	0·028 (1)

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

O(1)—C(13)	1·366 (3)	O(1)—C(14)	1·451 (3)
O(2)—C(13)	1·219 (3)	N—C(1)	1·476 (3)
N—C(10)	1·476 (3)	N—C(11)	1·350 (3)
C(1)—C(2)	1·554 (3)	C(1)—C(5)	1·537 (3)
C(2)—C(3)	1·532 (4)	C(3)—C(4)	1·526 (3)
C(4)—C(5)	1·529 (3)	C(5)—C(6)	1·539 (3)
C(6)—C(7)	1·525 (3)	C(6)—C(10)	1·536 (3)
C(7)—C(8)	1·461 (6)	C(8)—C(9)	1·487 (5)
C(9)—C(10)	1·556 (3)	C(11)—C(12)	1·372 (3)
C(11)—C(15)	1·516 (3)	C(12)—C(13)	1·436 (3)
C(14)—C(15)	1·533 (3)	C(14)—C(16)	1·522 (3)
C(15)—C(19)	1·536 (3)	C(16)—C(17)	1·527 (4)
C(16)—C(18)	1·525 (4)		
C(13)—O(1)—C(14)	117·3 (2)	C(1)—N—C(10)	111·6 (2)
C(1)—N—C(11)	121·4 (2)	C(10)—N—C(11)	126·8 (2)
N—C(1)—C(2)	114·4 (2)	N—C(1)—C(5)	105·3 (2)
C(2)—C(1)—C(5)	105·9 (2)	C(1)—C(2)—C(3)	106·0 (2)
C(2)—C(3)—C(4)	104·5 (2)	C(3)—C(4)—C(5)	103·6 (2)
C(1)—C(5)—C(4)	105·6 (2)	C(1)—C(5)—C(6)	104·9 (2)
C(4)—C(5)—C(6)	113·6 (2)	C(5)—C(6)—C(7)	114·3 (2)
C(5)—C(6)—C(10)	104·2 (2)	C(7)—C(6)—C(10)	105·8 (2)
C(6)—C(7)—C(8)	105·4 (2)	C(7)—C(8)—C(9)	111·5 (3)
C(8)—C(9)—C(10)	105·3 (3)	N—C(10)—C(6)	103·9 (2)
N—C(10)—C(9)	116·3 (2)	C(6)—C(10)—C(9)	103·5 (2)
N—C(11)—C(12)	122·6 (2)	N—C(11)—C(15)	120·0 (2)
C(12)—C(11)—C(15)	117·4 (2)	C(11)—C(12)—C(13)	122·7 (2)
O(1)—C(13)—O(2)	117·1 (2)	O(1)—C(13)—C(12)	117·9 (2)
O(2)—C(13)—C(12)	125·0 (2)	O(1)—C(14)—C(15)	109·8 (2)
O(1)—C(14)—C(16)	106·6 (2)	C(15)—C(14)—C(16)	115·2 (2)
C(11)—C(15)—C(14)	107·5 (2)	C(11)—C(15)—C(19)	110·6 (2)
C(14)—C(15)—C(19)	112·5 (2)	C(14)—C(16)—C(17)	111·8 (2)
C(14)—C(16)—C(18)	110·9 (2)	C(17)—C(16)—C(18)	110·1 (2)

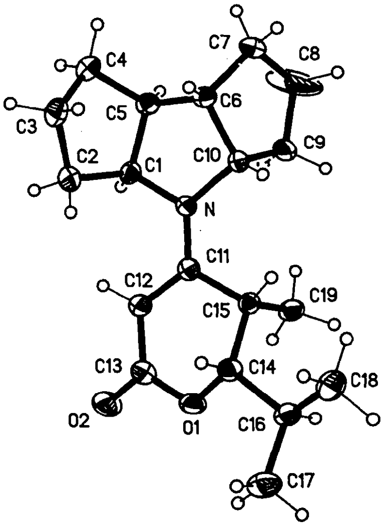

Fig. 1. Molecular structure with 50% probability ellipsoids, showing the atomic numbering scheme.

(maximum correction on I was $< 1{\cdot}1\%$). The data were also corrected for Lp effects. Reflections having $F_o < 6\sigma(F_o)$ were considered unobserved (271 reflections). Absorption corrections were not applied due to the small size of the crystal and the small value of the absorption coefficient, $\mu = 0{\cdot}72\ \text{cm}^{-1}$. Data reduction and decay correction were performed using the Nicolet XRD *SHELXTL-Plus* software package (Sheldrick, 1987). The structure was solved by direct methods (Sheldrick, 1987) and refined by full-matrix least squares (Sheldrick, 1976). In all, 308 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. The H-atom positions were obtained from a ΔF map. The H atoms were refined with isotropic thermal parameters except for those bonded to C(8) which were calculated in idealized positions and their isotropic thermal parameters fixed. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{\text{peak}} - I_{\text{background}}) \times$ (scan rate); the factor 0·02 serves to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects and decay. $\sigma(I)$ was estimated from counting statistics as $\sigma(I) = [(I_{\text{peak}} + I_{\text{background}})^{1/2} \times \text{(scan rate)}]$. An extinction correction $\chi = 0{\cdot}0005\ (2)$ $\{$where $F^* = F[1 + 0{\cdot}002\chi F^2/\sin(2\theta)]^{-1/4}\}$ was also applied (Sheldrick, 1987). The final $R = 0{\cdot}0418$ for 1617 reflections, with $wR = 0{\cdot}0480$ ($R_{\text{all}} = 0{\cdot}0528$, $wR_{\text{all}} = 0{\cdot}0508$) and goodness-of-fit = 1·59. The minimum and maximum peaks in the final ΔF map were $-0{\cdot}52$ and $0{\cdot}54\ \text{e Å}^{-3}$, respectively, and the maximum $|\Delta/\sigma|$ was 0·002. The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974).

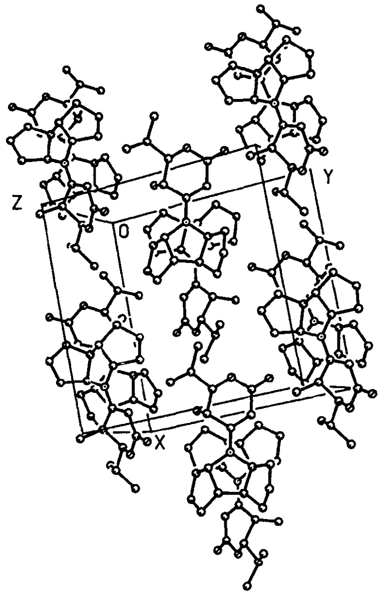

Fig. 2. Molecular packing.

Figures were generated using *SHELXTL-Plus* (Sheldrick, 1987). The positional and equivalent isotropic thermal parameters for the non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are in Table 2.* The drawing of the molecule with the atomic labeling scheme is shown in Fig. 1, and the packing diagram is in Fig. 2.

Related literature. The tricyclic amine was used for stereochemical control in the formation of the tricyclic aminopyranone where the absolute configuration at the two starred C atoms is controlled by asymmetric induction (Whitesell, Minton & Chen, 1988). Interpretation of these results required knowledge of the absolute stereochemistry at both new chiral centers, determined here by internal correlation with the known configuration of the amine subunit (Lynch, Minton, Whitesell & Davis, 1990). Conformations of similar tricyclic amine compounds are discussed in Chen, Whitesell, Price, Abboud & Davis (1990) and Abboud, Minton, Whitesell & Davis (1990).

Acknowledgement is made to the Donors of the Petroleum Research Fund, administered by the American Chemical Society, for partial support of this research (Grant ACS-PRF AC-20714 to JKW) and to the Robert A. Welch Foundation (Grant F-626 to JKW and F-233 to RED).

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* Lists of structure factors, anisotropic displacement coefficients, H-atom coordinates, bond lengths and angles involving H atoms, torsion angles and a structure determination summary have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52700 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1990). C**46**, 1553–1556

Structure at 198 K of a Chiral, Tricyclic Aminobiphenyl

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(*Received* 27 *October* 1989; *accepted* 12 *February* 1990)

Abstract. (*S*)-(3aα,4aβ,7aβ,7bα)-4-(4-Biphenylyl)-octahydro-1*H*,5*H*-dicyclopenta[*b*,*d*]pyrrole, $C_{22}H_{25}N$, $M_r = 303{\cdot}4$, triclinic, $P1$, $a = 8{\cdot}521$ (2), $b = 8{\cdot}656$ (2), $c = 12{\cdot}002$ (3) Å, $\alpha = 104{\cdot}51$ (2), $\beta = 90{\cdot}18$ (2), $\gamma = 92{\cdot}58$ (2)°, $V = 856{\cdot}1$ (3) Å^3, $Z = 2$ [two independent molecules, (*A*) and (*B*), in the

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