minimum peaks in final difference Fourier synthesis, 0·18 and −0·22 e Å^{-3}, respectively. Atomic scattering factors and anomalous-dispersion corrections taken from *International Tables for X-ray Crystallography* (1974), and all computer programs from the *SDP* system (Frenz, 1982).* Final atomic coordinates are listed in Table 1. Bond lengths and bond angles are given in Table 2. The molecular structure is depicted in Fig. 1 along with the atomic numbering scheme.

Related literature. The structures of several triazoles substituted with groups containing C or N atoms bonded to N(1) have been reported (Bauer, Boulton, Fedeli, Katritzky, Majid-Hamid, Mazza & Vaciago, 1972; Kokkou & Rentzeperis, 1975*a*,*b*; Murray-Rust, McManus, Lennon, Porter & Rechka, 1984; Sen & Venkatesan, 1984), as well as structures without substitution at N(1) (Kálmán, Simon, Schawartz & Horváth, 1974; Párkányi, Kálmán, Argay & Schawartz, 1977).

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* Lists of structure factors, anistropic temperature factors, H-atom positions, least-squares planes, and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52121 (52 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

BAUER, H., BOULTON, A. J., FEDELI, W., KATRITZKY, A. R., MAJID-HAMID, A., MAZZA, F. & VACIAGO, A. (1972). *J. Chem. Soc. Perkin Trans.* 2, pp. 662–667.
BOYER, J. H., MANIMARAN, T. & RAMAKRISHNAN, V. T. (1987). *J. Chem. Soc. Perkin Trans.* 1, pp. 2163–2169.
FRENZ, B. A. (1982). *The Enraf–Nonius CAD-4 SDP – A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography,* edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI. Delft Univ. Press.
International Tables for X-ray Crystallography (1974). Vol IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
JOHNSON, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
KÁLMÁN, A., SIMON, K., SCHAWARTZ, J. & HORVÁTH, G. (1974). *J. Chem. Soc. Perkin Trans.* 2, pp. 1849–1852.
KOKKOU, S. C. & RENTZEPERIS, P. J. (1975*a*). *Acta Cryst.* B31, 1564–1570.
KOKKOU, S. C. & RENTZEPERIS, P. J. (1975*b*). *Acta Cryst.* B31, 2788–2793.
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
MURRAY-RUST, P., MCMANUS, J., LENNON, S. P., PORTER, A. E. A. & RECHKA, J. A. (1984). *J. Chem. Soc. Perkin Trans.* 2, pp. 713–716.
PÁRKÁNYI, L., KÁLMÁN, A., ARGAY, G. & SCHAWARTZ, J. (1977). *Acta Cryst.* B33, 3102–3106.
SEN, N & VENKATESAN, K. (1984). *Acta Cryst.* C40, 1901–1905.

Structure at 173 K of a Chiral, Tricyclic Aminonitrobenzene

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Abstract. R-(3aα,4aβ,7aβ,7bα)-Octahydro-4-(4-nitrophenyl)-1H,5H-dicyclopenta[b,d]pyrrole, $C_{16}H_{20}N_2O_2$, $M_r = 272\cdot3$, orthorhombic, $P2_12_12_1$, $a = 8\cdot696$ (4), $b = 9\cdot630$ (3), $c = 16\cdot428$ (5) Å, $V = 1375\cdot7$ (8) Å^3, $Z = 4$, $D_x = 1\cdot315$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71073$ Å, $\mu = 0\cdot82$ cm^{-1}, $F(000) = 584$, $T = 173$ K, $R = 0\cdot0409$ for 1220 reflections $[F_o \geq 6\sigma(F_o)]$. Crystal chirality was assigned to correspond to the known chirality of the parent amine. Pairs of neighboring molecules stack in a head-to-tail arrangement with a psuedo-inversion center.

Experimental. The title compound crystallized as yellow rods and elongated prisms by slow evaporation from ethanol. It was synthesized by aromatic substitution of the tricyclic amine (Whitesell, Minton & Chen, 1988) on p-fluoronitrobenzene. The data crystal had dimensions $0\cdot09 \times 0\cdot17 \times 0\cdot29$ 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 47 reflections with $10\cdot0 < 2\theta < 21\cdot0°$. The data were collected using the ω scan technique with a 2θ range from $3\cdot0$–$55\cdot0°$ and a $1\cdot2°$ ω scan at 2–$4°$ min^{-1} (three symmetry-related octants: $h = 0 \rightarrow 11$, $k = 0 \rightarrow 12$, $l = 0 \rightarrow 21$; $h = 0 \rightarrow 11$, $k = -12 \rightarrow 0$, $l = -21 \rightarrow 0$; $h = -11 \rightarrow 0$, $k = -12 \rightarrow 0$, $l = 0 \rightarrow 21$). Of

the 5463 reflections measured, 1821 were unique, with $R_{int} = 0.0303$ from averaging symmetry-equivalent reflections. Four reflections ($\overline{2}10$, 210, $\overline{2}1\overline{1}$, $\overline{2}02$) were remeasured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was $< 1.2\%$). The data were also corrected for Lp effects and for abosorption (numerical corrections based on measurement of indexed crystal faces, $\mu = 0.82$ cm^{-1}). Minimum and maximum transmission were 0.9985 and 0.9993, respectively. Reflections having $F_o < 6\sigma(F_o)$ were considered unobserved (601 reflections). 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, 262 parameters were refined. The non-H atoms were refined with anisotropic thermal parameters. Most of the H-atom positions were obtained from a ΔF map. Phenyl H atoms were initially calculated in idealized positions but were refined unconstrained during the final refinement cycles. The H atoms were refined with isotropic thermal parameters. The correct enantiomorph was chosen based on the synthesis from an amine of known chirality. The function $\sum w(|F_o| - |F_c|)^2$, was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0.5kI^{-1/2}\{[\sigma(I)]^2 + (0.02I)^2\}^{1/2}$. The intensity, I, is given by $(I_{peak} - I_{background}) \times$ (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 $\sigma(I) = [(I_{peak} + I_{background})^{1/2} \times$ (scan rate)]. An extinction correction $\chi = 0.0017$ (4) [where $F^* = F(1 + 0.002\chi F^2/\sin2\theta)^{-1/4}$] was also applied (Sheldrick, 1987). The final $R = 0.0409$ for 1821 reflections, with $wR = 0.0437$ ($R_{all} = 0.0772$, $wR_{all} = 0.0562$) and goodness-of-fit = 1.40. The minimum and maximum peaks in the final ΔF map were -0.20 and 0.17 e Å^{-3}, respectively, and the maximum $|\Delta/\sigma|$ was 0.005. 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).† Figures were generated using *SHELXTL-Plus* (Sheldrick, 1987). The positional

† Tables of anisotropic thermal parameters, H-atom positional parameters, bond lengths and angles involving the H atoms, torsion angles, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52092 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* (Å$^2 \times 10^3$).

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

	x	y	z	U_{eq}
O(1)	1117 (4)	4940 (3)	7813 (2)	50 (1)
O(2)	1626 (4)	6531 (3)	6932 (2)	59 (1)
N(1)	1761 (3)	1307 (3)	4674 (2)	29 (1)
N(2)	1417 (4)	5305 (3)	7113 (2)	39 (1)
C(1)	2088 (4)	1627 (3)	3820 (2)	28 (1)
C(2)	768 (5)	2314 (5)	3351 (2)	40 (1)
C(3)	371 (6)	1346 (5)	2647 (3)	48 (2)
C(4)	1738 (5)	373 (4)	2559 (2)	37 (1)
C(5)	2313 (5)	200 (4)	3431 (2)	30 (1)
C(6)	1389 (4)	−819 (3)	3957 (2)	27 (1)
C(7)	2183 (5)	−2209 (4)	4070 (2)	34 (1)
C(8)	3390 (5)	−1906 (4)	4723 (2)	37 (1)
C(9)	2556 (5)	−969 (4)	5320 (2)	38 (1)
C(10)	1370 (4)	−155 (3)	4812 (2)	26 (1)
C(11)	1667 (4)	2279 (3)	5265 (2)	26 (1)
C(12)	2011 (4)	3690 (4)	5105 (2)	31 (1)
C(13)	1934 (4)	4663 (3)	5711 (2)	34 (1)
C(14)	1509 (4)	4270 (3)	6481 (2)	30 (1)
C(15)	1153 (4)	2907 (4)	6659 (2)	31 (1)
C(16)	1223 (4)	1929 (4)	6064 (2)	29 (1)

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

O(1)—N(2)	1.231 (4)	O(2)—N(2)	1.230 (4)
N(1)—C(1)	1.464 (4)	N(1)—C(10)	1.466 (4)
N(1)—C(11)	1.351 (4)	N(2)—C(14)	1.441 (5)
C(1)—C(2)	1.533 (6)	C(1)—C(5)	1.528 (5)
C(2)—C(3)	1.525 (6)	C(3)—C(4)	1.521 (6)
C(4)—C(5)	1.525 (5)	C(5)—C(6)	1.535 (5)
C(6)—C(7)	1.518 (5)	C(6)—C(10)	1.544 (4)
C(7)—C(8)	1.529 (6)	C(8)—C(9)	1.516 (6)
C(9)—C(10)	1.540 (5)	C(11)—C(12)	1.416 (5)
C(11)—C(16)	1.409 (4)	C(12)—C(13)	1.368 (5)
C(13)—C(14)	1.372 (5)	C(14)—C(15)	1.380 (5)
C(15)—C(16)	1.358 (5)		
C(1)—N(1)—C(10)	113.3 (2)	C(1)—N(1)—C(11)	123.7 (3)
C(10)—N(1)—C(11)	122.7 (3)	O(1)—N(2)—O(2)	122.1 (3)
O(1)—N(2)—C(14)	119.2 (3)	O(2)—N(2)—C(14)	118.7 (3)
N(1)—C(1)—C(2)	115.3 (3)	N(1)—C(1)—C(5)	103.7 (3)
C(2)—C(1)—C(5)	105.9 (3)	C(1)—C(2)—C(3)	106.7 (3)
C(2)—C(3)—C(4)	105.8 (4)	C(3)—C(4)—C(5)	103.6 (3)
C(1)—C(5)—C(4)	104.7 (3)	C(1)—C(5)—C(6)	105.8 (3)
C(4)—C(5)—C(6)	115.2 (3)	C(5)—C(6)—C(7)	113.2 (3)
C(5)—C(6)—C(10)	104.7 (3)	C(7)—C(6)—C(10)	105.0 (3)
C(6)—C(7)—C(8)	103.3 (3)	C(7)—C(8)—C(9)	103.8 (3)
C(8)—C(9)—C(10)	105.9 (3)	N(1)—C(10)—C(6)	104.7 (2)
N(1)—C(10)—C(9)	114.7 (3)	C(6)—C(10)—C(9)	105.9 (3)
N(1)—C(11)—C(12)	121.2 (3)	N(1)—C(11)—C(16)	121.3 (3)
C(12)—C(11)—C(16)	117.4 (3)	C(11)—C(12)—C(13)	120.8 (3)
C(12)—C(13)—C(14)	119.7 (3)	N(2)—C(14)—C(13)	119.3 (3)
N(2)—C(14)—C(15)	119.6 (3)	C(13)—C(14)—C(15)	121.1 (3)
C(14)—C(15)—C(16)	119.8 (3)	C(11)—C(16)—C(15)	121.1 (3)

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 listed in Table 2. The atom-labeling scheme is shown in Fig. 1 and the packing diagram in Fig. 2. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. The chiral, substituted aminonitrobenzene (1) was prepared (Whitesell *et al.*, 1988)
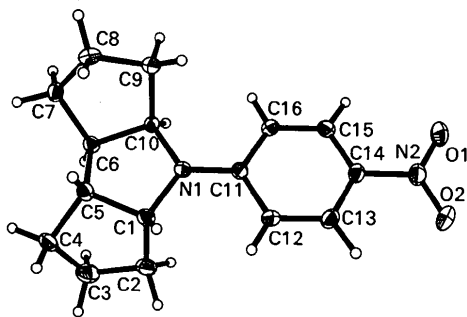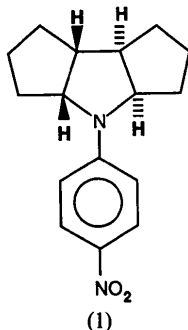
Fig. 1. Molecular structure of (1), with 30% probability ellipsoids, showing the atom-numbering scheme.

as a chiral analog of nitroaniline as a possible candidate for non-linear optical, second-harmonic generation (Chemla & Zyss, 1987).
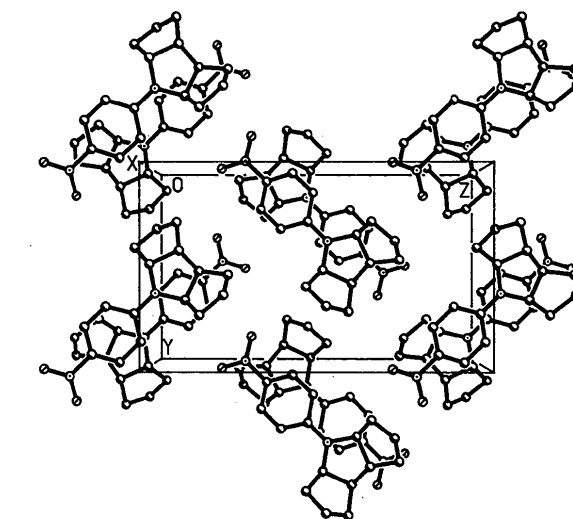

(1)

Fig. 2. Packing diagram of (1).

References

CHEMLA, D. S. & ZYSS, J. (1987). Editors. Nonlinear Optical Properties of Organic Molecules and Crystals, Vols. 1 and 2. New York: Academic Press.

CROMER, D. T. & LIBERMAN, D. (1970). J. Chem. Phys. 53, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). Acta Cryst. A24, 321–324.

GADOL, S. M. & DAVIS, R. E. (1982). Organometallics, 1, 1607–1613.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 55. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

SHELDRICK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1987). SHELXTL-Plus. Nicolet XRD Corporation, Madison, Wisconsin, USA.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). J. Phys. Chem. 42, 3175–3187.

WHITESELL, J. K., MINTON, M. & CHEN, K.-M. (1988). J. Org. Chem. 53, 5383–5384.

Structure of 6-Hydroxymethyl-6,9-dimethylbicyclo[6.3.0]undeca-2,4-dien-1-ol

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Abstract. $C_{14}H_{22}O_2$, $M_r = 222 \cdot 33$, orthorhombic, Pbca, $a = 10 \cdot 207$ (3), $b = 9 \cdot 746$ (6), $c = 25 \cdot 533$ (5) Å, $V = 2540$ (3) Å^3, $Z = 8$, $D_x = 1 \cdot 163$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0 \cdot 71073$ Å, $\mu = 0 \cdot 071$ mm^{-1}, $F(000) = 976$, $T = 293$ (1) K, $R = 0 \cdot 050$ for 1637 unique reflections with $I > 3\sigma(I)$. The molecular dimensions involving the eight-membered ring are normal while C(10) in the five-membered ring is disordered over two sites. The structure is stabilized by a short intermolecular distance $O(2) \cdots H(O1)$ of $1 \cdot 94$ Å. There is a short intramolecular contact $O(1) \cdots H(O2)$ of $2 \cdot 00$ Å.