

Fig. 1. Structure of (1).

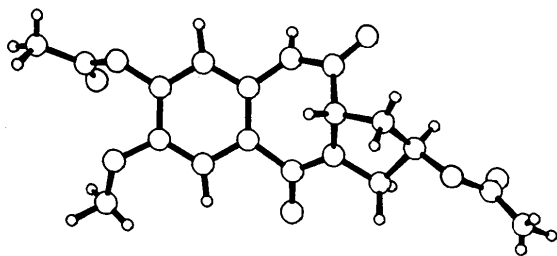


Fig. 2. Structure of (2).

Cheetham, Remers, Hill & Hurley, 1990). The non-covalent binding of (2) to DNA has been studied (Jones *et al.*, 1990).

We are grateful to the Cancer Research Campaign for grants (to SN and DET).

#### References

- ARORA, S. K. (1979). *Acta Cryst.* B35, 2945–2948.  
 BOYD, F. L., CHEETHAM, S. F., REMERS, W., HILL, G. C. & HURLEY, L. H. (1990). *J. Am. Chem. Soc.* 112, 3279–3289.  
 FRENZ, B. A. (1980). *Enraf-Nonius Structure Determination Package*. Version 17. College Station, Texas, USA.  
 JONES, G. B., DAVEY, C. L., JENKINS, T. C., KAMAL, A., KNEALE, G. G., NEIDLE, S., WEBSTER, G. D. & THURSTON, D. E. (1990). *Anti-Cancer Drug Design*, 5, 249–264.  
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.  
 MOSTAD, A., RØMMING, C. & STORM, B. (1978). *Acta Chem. Scand. Ser. B*, 32, 639–645.

*Acta Cryst.* (1991). C47, 2680–2682

## Structure of ( $\pm$ )-*exo*-6-Methyl-*endo*-6-nitrobicyclo[2.2.1]heptan-*exo*-2-ol\*

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(Received 7 May 1991; accepted 2 July 1991)

**Abstract.** C<sub>8</sub>H<sub>13</sub>NO<sub>3</sub>,  $M_r = 171.20$ , monoclinic,  $C2/c$ ,  $a = 27.89$  (2),  $b = 6.327$  (2),  $c = 22.04$  (1) Å,  $\beta = 114.65$  (4)°,  $V = 3533.53$  Å<sup>3</sup>,  $Z = 16$ ,  $D_m = 1.29$  (floatation in aqueous KsCl solution),  $D_x = 1.29$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.106$  mm<sup>-1</sup>,  $F(000) = 1472$ , room temperature, final  $R = 0.072$  for 2458 unique observed reflections. Two molecules constitute the asymmetric unit, and four molecules are associated as cyclic tetramers by hydrogen bonds involving only the OH groups. The conformations of both nitro groups, given by C(5)—C(6)—N(1)—O(3) and C(5')—C(6')—N(1')—O(3') torsion angles of  $-9.5$  (6) and  $-9.2$  (6)° respectively, are consistent with orientations in which the C(5)—C(6) bond of the rigid bicyclic skeleton is nearly eclipsed.

**Experimental.** The title compound and its *exo*-5-methyl-*endo*-5-nitro isomer were obtained by hydroboration and oxidation of *exo*-5-methyl-*endo*-5-nitrobicyclo[2.2.1]hept-2-ene as described previously (Michael, Maqutu & Howard, 1989). Products were separated by chromatography on silica gel. Single crystals of the appropriate isomer were grown by diffusion of hexane vapour into a solution of the compound in benzene. The analysis was performed on a colourless cube of approximate dimensions 0.5 × 0.5 × 0.5 mm. The space group  $C2/c$  (No. 15) and preliminary lattice constants were determined from oscillation and Weissenberg photographs. Diffraction data were collected on an Enraf-Nonius CAD-4 automatic diffractometer with graphite-monochromated Mo  $K\alpha$  radiation [ $\omega/2\theta$  scan technique,  $\Delta\omega = (0.6 + 0.35\tan\theta)^\circ$ , variable scan speed 1.0–5.5° min<sup>-1</sup>,  $3 < \theta < 30^\circ$ ,  $-39 < h < 39$ ,  $0 < k < 8$ ,  $0 < l < 30$ ]. Cell dimensions were obtained by least-squares refinement of 25 accurately measured

\* Nitrobicyclo[2.2.1]heptanes. 9. Part 8: Michael, Blom & Glin-tenkamp (1991).

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high- $\theta$ -angle values ( $10 < \theta < 20^\circ$ ). The maximum time allowed per reflection was 60 s. Orientation was monitored every 100 reflections, and three standard reflections monitored every hour showed a 0.6% variation over the data collection. Data were corrected for Lorentz, polarization and empirical absorption effects (North, Phillips & Mathews, 1968). Transmission coefficients were in the range 0.945–1.000. Scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B). 5680 reflections, 4186 unique reflections, 2458 with  $F_o > 3\sigma(F)$ ,  $R_{\text{int}}$  0.072,  $\Delta/\sigma < 0.01$ .

The structure was solved on a CDC Cyber 750 computer with the aid of the centrosymmetric direct methods option of the program *SHELX84* (Sheldrick, 1984).  $E$  maps, calculated from reflections with  $E > 1.2$ , revealed all non-H atoms, and the positions of these were refined using  $F$  magnitudes by full-matrix least-squares methods. H atoms were located from difference Fourier maps. After refinement with anisotropic temperature factors for non-H atoms and isotropic temperature factors for H, a conventional  $R$  of 0.099 (unit weights) was attained.  $R$  was improved to 0.072 by placing all H (other than OH, which was not located) in calculated positions (C—H 1.08 Å) riding with an overall isotropic temperature factor that refined to a final  $U$  of 0.078 (4) Å<sup>2</sup>. The rather high  $R$  value is ascribed to weakly diffracting crystals. Residual electron density ranged from  $-0.24$  to  $0.27$  e Å<sup>-3</sup>. The final structure is shown in Fig. 1. Atomic parameters are in Table 1, and bond lengths and angles are in Table 2.\*

Two molecules belonging to the same enantiomeric series constitute the asymmetric unit. An intermolecular distance of 2.726 Å was noted between the O(1) and O(1') atoms of the hydroxy groups in these two molecules. This distance is short enough

\* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54418 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

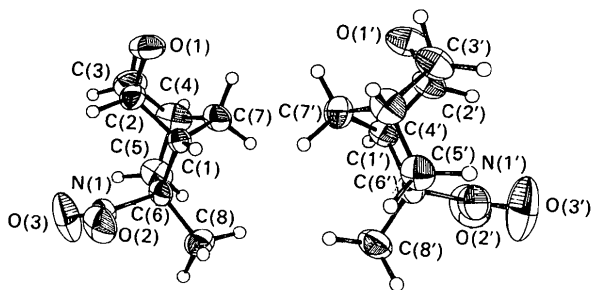


Fig. 1. ORTEP (Johnson, 1965) diagram of the asymmetric unit of the title compound (thermal ellipsoids at 50% probability).

Table 1. Fractional coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors (Å<sup>2</sup>  $\times 10^3$ ) for non-H atoms

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$U_{\text{eq}}$
O(1)	3073 (1)	8609 (6)	-217 (2)	51 (2)
O(2)	4517 (2)	10809 (6)	1353 (2)	57 (2)
O(3)	4925 (2)	10541 (8)	727 (3)	82 (4)
N(1)	4663 (2)	9809 (7)	991 (2)	44 (2)
C(1)	3904 (2)	7244 (7)	628 (2)	32 (2)
C(2)	3634 (2)	8638 (8)	5 (2)	41 (2)
C(3)	3784 (2)	7543 (11)	-525 (3)	58 (3)
C(4)	4102 (2)	5631 (10)	-149 (3)	51 (3)
C(5)	4648 (2)	6356 (9)	360 (3)	52 (3)
C(6)	4509 (2)	7440 (7)	900 (2)	35 (2)
C(7)	3824 (2)	5050 (8)	302 (3)	44 (3)
C(8)	4791 (2)	6520 (9)	1606 (3)	53 (3)
O(1')	2628 (2)	-258 (7)	626 (2)	58 (2)
O(2')	3876 (2)	-1878 (7)	2441 (3)	71 (3)
O(3')	3435 (3)	-1037 (8)	3010 (3)	91 (4)
N(1')	3629 (2)	-633 (7)	2631 (3)	53 (3)
C(1')	3323 (2)	1469 (8)	1557 (2)	38 (2)
C(2')	2813 (2)	137 (8)	1326 (3)	45 (3)
C(3')	2428 (2)	1572 (9)	1489 (3)	51 (3)
C(4')	2761 (2)	3548 (8)	1791 (3)	46 (3)
C(5')	3181 (2)	2996 (8)	2490 (3)	45 (3)
C(6')	3574 (2)	1628 (7)	2328 (2)	37 (2)
C(7')	3092 (2)	3720 (8)	1380 (3)	48 (3)
C(8')	4138 (2)	2483 (9)	2614 (3)	51 (3)

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

O(1)—C(2)	1.430 (6)	O(1')—C(2')	1.429 (6)
O(2)—N(1)	1.213 (5)	O(2')—N(1')	1.230 (6)
O(3)—N(1)	1.202 (6)	O(3')—N(1')	1.197 (6)
N(1)—C(6)	1.549 (6)	N(1')—C(6')	1.559 (6)
C(1)—C(2)	1.538 (6)	C(1')—C(2')	1.543 (6)
C(1)—C(6)	1.542 (6)	C(1')—C(6')	1.547 (6)
C(1)—C(7)	1.536 (6)	C(1')—C(7')	1.545 (7)
C(2)—C(3)	1.558 (7)	C(2')—C(3')	1.560 (7)
C(3)—C(4)	1.524 (8)	C(3')—C(4')	1.533 (7)
C(4)—C(5)	1.537 (7)	C(4')—C(5')	1.537 (7)
C(4)—C(7)	1.540 (8)	C(4')—C(7')	1.543 (7)
C(5)—C(6)	1.556 (7)	C(5')—C(6')	1.551 (7)
C(6)—C(8)	1.535 (7)	C(6')—C(8')	1.528 (6)
O(2)—N(1)—O(3)	123.8 (5)	O(2')—N(1')—O(3')	125.6 (5)
O(2)—N(1)—C(6)	116.0 (4)	O(2')—N(1')—C(6')	114.2 (5)
O(3)—N(1)—C(6)	120.1 (5)	O(3')—N(1')—C(6')	120.2 (5)
C(2)—C(1)—C(6)	110.4 (4)	C(2')—C(1')—C(6')	109.4 (4)
C(2)—C(1)—C(7)	100.3 (4)	C(2')—C(1')—C(7')	101.0 (4)
C(6)—C(1)—C(7)	100.2 (4)	C(6')—C(1')—C(7')	99.7 (4)
O(1)—C(2)—C(1)	110.8 (4)	O(1')—C(2')—C(1')	108.7 (4)
O(1)—C(2)—C(3)	109.6 (4)	O(1')—C(2')—C(3')	112.1 (4)
C(1)—C(2)—C(3)	103.3 (4)	C(1')—C(2')—C(3')	103.5 (4)
C(2)—C(3)—C(4)	102.9 (4)	C(2')—C(3')—C(4')	102.9 (4)
C(3)—C(4)—C(5)	109.6 (5)	C(3')—C(4')—C(5')	108.6 (4)
C(3)—C(4)—C(7)	101.8 (4)	C(3')—C(4')—C(7')	101.9 (4)
C(5)—C(4)—C(7)	102.2 (4)	C(5')—C(4')—C(7')	102.2 (4)
C(4)—C(5)—C(6)	101.7 (4)	C(4')—C(5')—C(6')	102.3 (4)
N(1)—C(6)—C(1)	109.2 (3)	N(1')—C(6')—C(1')	109.2 (4)
N(1)—C(6)—C(5)	112.4 (4)	N(1')—C(6')—C(5')	111.4 (4)
C(1)—C(6)—C(5)	104.4 (4)	C(1')—C(6')—C(5')	104.4 (4)
N(1)—C(6)—C(8)	103.8 (4)	N(1')—C(6')—C(8')	104.4 (4)
C(1)—C(6)—C(8)	112.3 (4)	C(1')—C(6')—C(8')	113.1 (4)
C(5)—C(6)—C(8)	114.9 (4)	C(5')—C(6')—C(8')	114.4 (4)
C(1)—C(7)—C(4)	94.1 (4)	C(1')—C(7')—C(4')	94.3 (4)

(< 3.4 Å) to imply the existence of a hydrogen bond between these sites (Olovsson & Jonsson, 1976). The pair of molecules at  $x, y, z$  associates with another pair at symmetry position  $0.5 - x, 1.5 - y, 1 - z$  such that O(1) and O(1') of the former pair are 2.767 Å away from O(1') and O(1), respectively, of the latter pair. The four O atoms form the corners of a parallelogram with angles of 87.8 (8) and 92.1 (2)°. Four

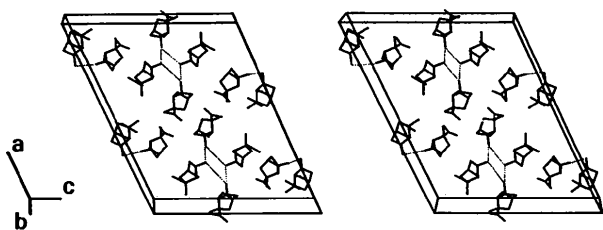


Fig. 2. Stereoview of the contents of the unit cell showing O...O close contacts.

molecules of the nitronorbornanol thus associate as hydrogen-bonded cyclic tetramers in which the hydroxy, but not the nitro, groups are involved. Fig. 2 shows unit-cell contents and the putative hydrogen-bonded cyclic tetramers.

**Related literature.** Other nitronorbornanols investigated in these laboratories show hydrogen bonding between hydroxy and nitro groups (Boeyens, Denner & Michael, 1984*a,b*).

*Acta Cryst.* (1991). C47, 2682–2683

## 2,4,4,6-Tetraphenyl-3(4*H*)-pyridinone

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(Received 7 May 1991; accepted 25 June 1991)

**Abstract.** C<sub>29</sub>H<sub>21</sub>NO, *M<sub>r</sub>* = 399.49, monoclinic, *P*2<sub>1</sub>/*a*, *a* = 21.713 (5), *b* = 13.524 (4), *c* = 7.259 (2) Å,  $\beta$  = 95.20 (2)°, *V* = 2123.0 (11) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.250 g cm<sup>-3</sup>,  $\lambda$ (Mo *K*α) = 0.71073 Å,  $\mu$  = 0.81 cm<sup>-1</sup>, *F*(000) = 840, *T* = 294 K, *R* = 0.055 for 2248 observed reflections. In the pyridine ring the C(5)=C(6)—N(1)=C(2) moiety is almost planar and forms a dihedral angle of 35.0 (2)° with the carbonyl plane of C(2), C(3), C(4) and O(7). The 2- and 6-phenyl groups are slightly rotated from the C(5)=C(6)—N(1)=C(2) plane: the dihedral angles are 10.3 (1) and 23.2 (2)° for the 2- and 6-positions, respectively.

**Experimental.** The title compound was synthesized by the method given by Maeda, Nakamura & Sakai (1983). Recrystallization from acetone–ethanol gave orange plate-like crystals of dimensions 0.5 × 0.2 × 0.1 mm; Rigaku AFC-4 diffractometer; cell parameters were determined from 20 independent 2θ values (25 < 2θ < 30°); intensity measurements were per-

We thank the Foundation for Research Development and the University of the Witwatersrand for funding for this project, and Dr D. C. Levendis for technical assistance.

### References

- BOEYENS, J. C. A., DENNER, L. & MICHAEL, J. P. (1984*a*). *J. Chem. Soc. Perkin Trans. 2*, pp. 767–770.  
 BOEYENS, J. C. A., DENNER, L. & MICHAEL, J. P. (1984*b*). *J. Chem. Soc. Perkin Trans. 2*, pp. 1569–1573.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.  
 MICHAEL, J. P., BLOM, N. F. & GLINTENKAMP, L.-A. (1991). *J. Chem. Soc. Perkin Trans. 1*, pp. 1855–1862.  
 MICHAEL, J. P., MAQUTU, T. L. & HOWARD, A. S. (1989). *J. Chem. Soc. Perkin Trans. 1*, pp. 2389–2395.  
 NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A24, 351–359.  
 OLOVSSON, I. & JONSSON, P. G. (1976). *The Hydrogen Bond*, Vol. 2, edited by P. SCHUSTER, G. ZINDEL & C. J. SANDORFY, pp. 393–456. Amsterdam: North-Holland.  
 SHELDRICK, G. M. (1984). *SHELXS84*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

formed with graphite-monochromated Mo *K*α radiation up to 2θ = 50.0° (*h* = 29 → 29, *k* 0 → 18, *l* 0 → 10), ω–2θ scan technique, scan speed 4° min<sup>-1</sup> in θ, scan width (1.0 + 0.35tanθ)°; background 5 s before and after each scan; three standard reflections (961, 372, 234) were monitored every 50 reflections and showed no significant variation in intensities. 3743 unique reflections were measured, 2252 with  $|F_o| > 3\sigma(|F_o|)$  were used for structure determination; corrections for Lorentz and polarization effects, absorption ignored; direct methods (*MULTAN78*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and full-matrix least-squares refinement (*SHELX76*; Sheldrick, 1976) with anisotropic thermal parameters for non-H atoms and isotropic ones for H atoms, minimizing the function  $\sum w(|F_o| - |F_c|)^2$ ,  $w = [\sigma^2(|F_o|) + 0.0040|F_o|^2]^{-1}$ ; some H atoms located on difference Fourier map, other H-atom positions determined geometrically and included in subsequent refinements; four intense reflections (110, 200, 011, 617) seemed to suffer from extinction and were not used in refinements; final *R* = 0.055 and *wR* = 0.065 for 2248 observed reflec-

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