

$-0.2 e \text{ \AA}^{-3}$. Final parameters are given in Table 1,* and Table 2 shows anion bond distances and angles. ORTEPII (Johnson, 1976) diagrams of cation, anion and cell are shown in Figs. 1, 2 and 3, respectively.

Related literature. The absolute configuration of strychnine has been reported by Peerdeman (1956). Gould & Walkinshaw (1984) have reported on the bilayer packing pattern of strychnine salts. In a subsequent work, Gould, Taylor & Walkinshaw (1987) reported the structures of strychnine salts for both enantiomers of tartaric acid. The possibility of thermal and photochemically induced isomerization of the phosphorothionate entity in malathion, to the more toxic phosphorodithiolate present in isomalathion, is presented in several papers (Metcalf & March, 1953; Rengasammy & Parmar, 1988;

* Lists of anisotropic thermal parameters, H-atom coordinates, bond distances and angles for the *N*-methylstrychninium cation and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55585 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1007]

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Structure of (3 α ,4 α ,7 α ,7 α)-2,2-Dimethyl-3 α ,4,7,7 α -tetrahydro-1,3-benzodioxolane-4,7-diamine

BY M. F. MACKAY

Department of Chemistry, La Trobe University, Bundoora, Victoria 3083, Australia

AND M. G. BANWELL AND S. L. RICHARDS

School of Chemistry, The University of Melbourne, Parkville, Victoria 3052, Australia

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Abstract. $C_9H_{16}N_2O_2$, $M_r = 184.2$, rhombohedral, $R3$, $a = 12.594(2) \text{ \AA}$, $\alpha = 118.03(1)^\circ$, $V = 720(2) \text{ \AA}^3$, $Z = 3$, $D_x = 1.274 \text{ Mg m}^{-3}$, $\lambda(\text{Cu } K\alpha) = 1.5418 \text{ \AA}$, $\mu = 0.70 \text{ mm}^{-1}$, $F(000) = 300$, $T = 290(1) \text{ K}$, final $R = 0.047$ for 1191 reflections with $I \geq 2\sigma(I)$. Although the title molecule (I) is a *meso* compound, it does not have exact mirror symmetry in the solid state. The observed distortions, which no doubt arise from the fusion of the two rings, results in two enantiomeric forms with any given crystal containing only the one enantiomer (e.g. with 3 α S,-4*R*,7*S*,7 α *R* configurations). The cyclohexene ring

is in a fairly regular boat form with asymmetry parameter [Duax & Norton (1975). *Atlas of Steroid Structure*, Vol. I, pp. 18–19. New York: Plenum] $\Delta C_s = 5.8^\circ$ and the amino substituents in equatorial positions. The conformation of the *cis*-fused oxolane ring is between a half chair and an envelope as is reflected by the pseudorotational parameters [Altona, Geise & Romers (1968). *Tetrahedron*, **24**, 13–32] $\Delta = 12.5^\circ$ and $\varphi_m = 35.6^\circ$. Intermolecular hydrogen bonds involving the exocyclic N atoms link each molecule to four adjacent molecules to form a three-dimensional network: the N(4)⋯N(4) [$(z, x, -1 + y)$,

Table 1. Final atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (\AA^2) for the non-H atoms with e.s.d.'s in parentheses

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

| | x | y | z | U_{eq} |
|-------|-----------|-----------|------------|-----------------|
| O(1) | 5387† | 4856 (2) | 2494 (2) | 0.058 (1) |
| C(2) | 4106 (8) | 3306 (8) | 243 (6) | 0.057 (1) |
| O(3) | 2403 (6) | 2755 (6) | -1104 (6) | 0.051 (1) |
| C(3a) | 3726 (6) | 5026 (6) | 838 (6) | 0.043 (1) |
| C(4) | 1726 (6) | 4216 (6) | -176 (6) | 0.051 (1) |
| N(4) | 751 (6) | 3978 (6) | -1654 (6) | 0.045 (1) |
| C(5) | 2924 (8) | 6165 (8) | 2039 (8) | 0.055 (1) |
| C(6) | 4423 (9) | 7139 (9) | 3982 (9) | 0.059 (1) |
| C(7) | 4944 (8) | 6294 (8) | 3969 (8) | 0.054 (1) |
| N(7) | 7081 (6) | 7990 (6) | 6485 (6) | 0.053 (1) |
| C(7a) | 5611 (6) | 6333 (6) | 3235 (6) | 0.046 (1) |
| C(1') | 6032 (10) | 4771 (11) | 970 (10) | 0.083 (1) |
| C(2') | 2451 (11) | 770 (10) | -1421 (10) | 0.099 (1) |

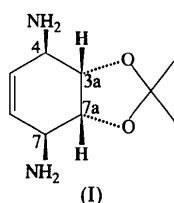
† Coordinate fixed for origin definition for space group R3.

Table 2. Selected intramolecular dimensions (\AA , °) with e.s.d.'s in parentheses

| | | | |
|-----------------------|-----------|-----------------------|-----------|
| O(1)—C(2) | 1.41 (1) | C(3a)—C(7a) | 1.54 (1) |
| O(1)—C(7a) | 1.43 (1) | C(4)—N(4) | 1.43 (1) |
| C(2)—O(3) | 1.43 (1) | C(4)—C(5) | 1.49 (1) |
| C(2)—C(1') | 1.50 (2) | C(5)—C(6) | 1.31 (2) |
| C(2)—C(2') | 1.51 (1) | C(6)—C(7) | 1.49 (2) |
| O(3)—C(3a) | 1.43 (1) | C(7)—N(7) | 1.44 (1) |
| C(3a)—C(4) | 1.53 (1) | C(7)—C(7a) | 1.52 (2) |
| C(2)—O(1)—C(7a) | 108.7 (3) | C(4)—C(3a)—C(7a) | 114.8 (6) |
| O(1)—C(2)—O(3) | 104.6 (3) | C(3a)—C(4)—C(5) | 110.0 (7) |
| C(2)—O(3)—C(3a) | 106.8 (6) | C(4)—C(5)—C(6) | 120.8 (8) |
| O(3)—C(3a)—C(7a) | 103.7 (6) | C(5)—C(6)—C(7) | 119.2 (8) |
| O(1)—C(7a)—C(3a) | 104.4 (3) | C(6)—C(7)—C(7a) | 109.4 (7) |
| C(3a)—C(7a)—C(7) | 113.6 (7) | | |
| C(7a)—O(1)—C(2)—O(3) | -31 (1) | C(3a)—C(4)—C(5)—C(6) | -43 (1) |
| O(1)—C(2)—O(3)—C(3a) | 35 (1) | C(4)—C(5)—C(6)—C(7) | 1 (2) |
| C(2)—O(3)—C(3a)—C(7a) | -26 (1) | C(5)—C(6)—C(7)—C(7a) | 46 (1) |
| O(3)—C(3a)—C(7a)—O(1) | 7 (1) | C(6)—C(7)—C(7a)—C(3a) | -47 (1) |
| C(3a)—C(7a)—O(1)—C(2) | 14 (1) | C(7)—C(7a)—C(3a)—C(4) | 7 (1) |
| C(2)—O(1)—C(7a)—C(7) | 136 (1) | C(7a)—C(3a)—C(4)—C(5) | 36 (1) |
| C(2)—O(3)—C(3a)—C(4) | -149 (1) | | |

(y, 1 + z, x)] distance is 2.78 (1) \AA and the N(7)⋯N(7) [(z, x, y), (y, z, x)] distance is 2.84 (2) \AA .

Experimental. A colourless prismatic crystal of (I) with dimensions ca 0.49 \times 0.49 \times 0.62 mm was aligned on a Rikaku AFC diffractometer. Cell



parameters were determined by least-squares treatment of the 2θ values for 25 strong reflections ($43 < 2\theta < 74^\circ$). Intensities were measured using Cu $K\alpha$ radiation (graphite-crystal monochromator) and ω - 2θ scans, with 2θ rate 2° min^{-1} and scan range $(1.20 + 0.5 \tan \theta)^\circ$, to $2\theta_{\text{max}} = 130^\circ$. 10 s stationary background counts were made. Three standard reflections, monitored every 50 reflections, showed

no significant intensity variation. 2117 data were measured in a triclinic setting, $a = 12.594$ (2), $b = 5.354$ (1), $c = 12.969$ (2) \AA , $\alpha = 74.03$ (1), $\beta = 59.02$ (1), $\gamma = 81.85$ (1)° ($h - 12$ to 12, $k - 6$ to 6, $l 0$ to 15), which converted to rhombohedral axes with the conversion matrix

$$\begin{vmatrix} 0 & -1 & -1 \\ -1 & 0 & 0 \\ 1 & 0 & -1 \end{vmatrix}$$

1191 ($R_{\text{int}} = 0.041$) observed data [$I \geq 2\sigma(I)$] yielded. Intensities were corrected for Lorentz and polarization effects, as well as for absorption (transmission factors 0.679–0.779). The structure was solved by direct methods with *SHELXS86* (Sheldrick, 1985) and full-matrix refinement using *SHELX76* (Sheldrick, 1976), with anisotropic temperature factors given to C, N and O atoms and isotropic for H atoms, which were included at idealized positions (those of the amino groups omitted as attempts to locate their sites were unsuccessful), converged at $R = 0.047$, $wR = 0.069$, $S = 3.89$ (128 parameters varied); function minimized $\sum w(|F_o| - |F_c|)^2$ with $w = (\sigma^2 |F_o|^2 + 0.00027 |F_o|^2)^{-1}$; $(\Delta/\sigma)_{\text{max}} = 0.017$; $(\Delta\rho)_{\text{max}}$, $(\Delta\rho)_{\text{min}} = 0.44$, -0.22 e \AA^{-3} . An isotropic extinction correction of the form $F = F_c [1 - (1.80 \times 10^{-5} F^2 / \sin \theta)]$ was applied to the calculated structure amplitudes. Atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). Fig. 1 was prepared from the output of *ORTEPII* (Johnson, 1976). Calculations were performed on a VAX 8800 computer. Atomic parameters for non-H atoms are given in Table 1; * Table 2 contains selected molecular dimensions while Fig. 1 shows a perspective view of the molecule with the atom numbering.

* Lists of structure-factor amplitudes, anisotropic thermal parameters, H-atom coordinates with their isotropic temperature factors and short intermolecular approaches have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55590 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HL1011]

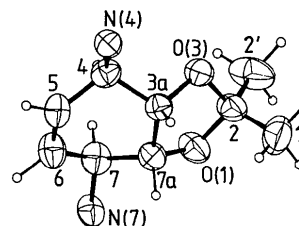


Fig. 1. Perspective view of the title compound with thermal ellipsoids scaled to 50% probability. H atoms are represented by spheres of arbitrary radii. For C atoms the C symbol is omitted.

Related literature. The title molecule, formed by an unusual reaction pathway from *cis*-1,2-dihydrocatechol (Banwell, Mackay, Reum, Richards & Stasi, 1992), is a member of the synthetically and biologically important aminoconduritol class of compounds (Balci, Suteyaz & Secen, 1990).

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Structure of *N*-(2-Furylmethyl)- α -cyano-2-furanacrylamide

BY O. DURRUTHY

Department of Physics, ISPJAM, CP 90900, Santiago de Cuba, Cuba

AND R. POMÉS,* F. FAJARDO AND A. DAGO

CNIC, CP 11600, Apartado 6880, Ciudad de La Habana, Cuba

(Received 19 December 1991; accepted 29 June 1992)

Abstract. C₁₃H₁₀N₂O₃, $M_r = 242.2$, triclinic, $P\bar{1}$, $a = 5.305$ (1), $b = 9.486$ (2), $c = 11.437$ (3) Å, $\alpha = 94.1$ (2), $\beta = 93.8$ (2), $\gamma = 93.6$ (2)°, $V = 571$ (1) Å³, $Z = 2$, $D_x = 1.41$ g cm⁻³, graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å, $\mu = 0.96$ cm⁻¹, $F(000) = 252$, room temperature, $R = 0.048$ for 2586 observed reflections with $I \geq 3\sigma(I)$. There is an intramolecular hydrogen bond N(1)—H(6)···N(2) with N(1)···N(2) = 3.108 (5) Å. The structure is built up by discrete molecules linked by van der Waals forces.

Experimental. Intensity data were obtained from a prismatic crystal, 0.2 × 0.2 × 0.3 mm, on an Enraf–Nonius CAD-4 diffractometer using the ω -2 θ scan mode. Unit-cell parameters were determined from least-squares refinement of 25 automatically centred reflections in the range $11 \leq \theta \leq 15^\circ$. Three standard reflections showed intensity variation < 5%. 2897 reflections were measured ($1 \leq \theta \leq 26^\circ$; $0 \leq h \leq 6$, $-12 \leq k \leq 12$, $-14 \leq l \leq 14$) of which 2586 with $I \geq 3\sigma(I)$ were used for refinement. Lorentz and polarization corrections were applied, but absorption was ignored. The structure was solved by direct methods (Sheldrick, 1976) and refined using a full-matrix least-squares routine based on F . Non-H atoms were

refined with anisotropic displacement parameters; H atoms were located from a difference Fourier map and refined isotropically. $R = 0.048$, $wR = 0.065$ $\{w = [\sigma^2(F) + 0.014F^2]^{-1}\}$; $\Delta\rho_{\max} = 0.21$, $\Delta\rho_{\min} = -0.42$ e Å⁻³; $(\Delta/\sigma)_{\max} = 0.32$. 203 parameters were refined. All refinement calculations were carried out using *SHELX76* (Sheldrick, 1976). Atomic scattering factors were those stored in *SHELX76*. Positional parameters and equivalent isotropic displacement parameters are given in Table 1.† Bond lengths, bond angles and selected torsion angles are listed in Table 2. The numbering system for the molecule can be found in Fig. 1.

Related literature. The title compound was synthesized and spectroscopically investigated by Bartroli, Lamí & Díaz (1984). Research on bioactivity by Bermello (1990) using Golender & Rezenblit (1983) algorithms showed fungicide and bactericide proper-

† Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, and bond distances, bond angles and torsion angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55537 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0276]

* To whom correspondence should be addressed.