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## Structure of *cis*-2,3-Tetralindiol, *cis*-1,2,3,4-Tetrahydro-2,3-naphthalenediol, C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>

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**Abstract.**  $M_r = 164.0$ , monoclinic,  $P2_1/c$ ,  $a = 9.401(4)$ ,  $b = 11.664(1)$ ,  $c = 17.632(9)$  Å,  $\beta = 117.97(4)^\circ$ ,  $V = 1707.5$  Å<sup>3</sup>,  $Z = 8$ ,  $D_m = 1.29$ ,  $D_x = 1.28$  g cm<sup>-3</sup>,  $\text{Cu } K\alpha_1$   $\lambda = 1.54051$  Å,  $\mu = 7.27$  cm<sup>-1</sup>,  $F(000) = 704$ ,  $T = 300$  K,  $R = 0.053$  for 1088 reflections. The title compound is a naphthalene derivative with adjacent molecules linked by intermolecular hydrogen bonds.

**Introduction.** Many polycyclic aromatic hydrocarbons are common environmental pollutants and potent chemical carcinogens. The active forms are believed to be metabolic derivatives of the parent hydrocarbons (Harvey, 1981, 1982). A series of naphthalene derivatives which model various metabolites of other carcinogenic hydrocarbons and are, in some cases, themselves natural metabolites of naphthalene (Tsang, Griffin, Horning & Stillwell, 1982) have been chosen for study. The structure of *cis*-2,3-tetralindiol confirms a stereochemical assignment made on the basis of its NMR spectra.

**Experimental.** Colorless, needle-like crystal, approximately  $0.6 \times 0.1 \times 0.1$  mm,  $D_m$  by flotation in dioxane/CHCl<sub>3</sub>; unit-cell dimensions and systematic absences  $h0l$ ,  $l = 2n$ , and  $0k0$ ,  $k = 2n$ , indicated monoclinic,  $P2_1/c$  confirmed by successful determination of structure; lattice constants from least-squares fit of 37 carefully measured  $2\theta$  values ( $2^\circ$  take-off angle,  $0.05^\circ$  slit) for  $2\theta > 61^\circ$ ; General Electric XRD-490 automated diffractometer, station-

ary-crystal-stationary-counter, balanced Ni and Co filters, 2648 reflections measured,  $2\theta_{\text{max}} = 120^\circ$ , 1088 (41%) considered statistically significant by  $[I_{\text{Ni}} - 2\sigma(I_{\text{Ni}})] - [I_{\text{Co}} - 2\sigma(I_{\text{Co}})] > 500$  with  $\sigma$ 's based entirely on counting statistics and  $F > 68$ , data corrected for polarization and background as a function of  $2\theta$ ,  $\varphi$  absorption minimal and not corrected for, three standard reflections after every 100 reflections, no significant change in intensity; direct methods, normalized structure factors, scaled by a  $k$  curve (Karle, Hauptman & Christ, 1958), phases determined with *MULTAN* (Germain, Main & Woolfson, 1971); initial  $E$  map contained twenty-four non-hydrogen peaks corresponding to two asymmetric molecular units; these 24 atoms refined isotropically on  $F$ , block-diagonal least squares,  $R = 0.154$ ; ring hydrogen atoms calculated on the basis of  $sp^2$  or  $sp^3$  geometry and C–H bond distance of 1.0 Å; refinement of hydrogen-atom thermal parameters resulted in one set of temperature factors approximately twice as large as another set so average value in each set was calculated and hydrogen atoms assigned values for  $U$  of 0.0569 or 0.1139 Å<sup>2</sup>; continued block-diagonal least squares of nonhydrogen atom positions (anisotropic) and hydrogen-atom positions (fixed isotropic) resulted in C–H bond distances between 0.93 (7) and 1.15 (6) Å; all four hydroxyl hydrogen atoms participate in an intermolecular hydrogen-bond network; coordinates for two of the hydroxyl hydrogen atoms [H(B1) and H(B1')] found on a difference map, coordinates for remaining two hydroxyl hydrogen atoms [H(B2) and H(B2')]

calculated assuming an O—H bond distance of 1.05 Å and an O—H...O angle of 180°; coordinates and isotropic thermal parameters ( $U = 0.0569 \text{ \AA}^2$ ) of hydroxyl hydrogen atoms not refined;  $R = 0.053$ ,  $R_w = 0.059$ ,  $w = 1/\sigma^2$ ,  $\sigma = \frac{1}{2}F(1+B)/(1-B)^{1/2}$ ,  $B = I_{Co}/I_{Ni}$ ,  $S = 3.38$ ;  $\Delta_{\max}/\sigma = 0.02$ , maximum and minimum  $\Delta\rho$  excursions +0.29 and -0.21 e Å<sup>-3</sup>; scattering factors from *International Tables for X-ray Crystallography* (1968) for hydrogen and from Cromer & Waber (1965) for carbon and oxygen.\*

**Discussion.** Final fractional coordinates are given in Table 1. The numbering system, bond lengths and bond angles may be found in Fig. 1.

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38668 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and isotropic thermal parameters (Å<sup>2</sup>)

	x	y	z	$U_{eq}^*/U_{iso}$
O(1)	0.4701 (5)	0.1664 (4)	0.0352 (3)	0.06028
O(2)	0.4790 (5)	-0.0685 (4)	0.0923 (3)	0.05608
O(1')	0.8324 (5)	0.4140 (4)	0.4484 (3)	0.05621
O(2')	1.1392 (5)	0.3890 (4)	0.4583 (3)	0.06141
C(1)	0.4602 (7)	0.1536 (6)	0.1684 (4)	0.05807
C(2)	0.5590 (7)	0.1317 (6)	0.1239 (4)	0.05603
C(3)	0.6166 (7)	0.0085 (6)	0.1295 (4)	0.05646
C(4)	0.7210 (8)	-0.0201 (6)	0.2248 (4)	0.05774
C(5)	0.6533 (7)	0.0176 (5)	0.2819 (4)	0.05306
C(6)	0.7138 (9)	-0.0314 (6)	0.3636 (5)	0.07382
C(7)	0.6636 (10)	0.0064 (7)	0.4194 (4)	0.09295
C(8)	0.5516 (8)	0.0932 (7)	0.3979 (4)	0.07600
C(9)	0.4913 (7)	0.1434 (6)	0.3176 (5)	0.06112
C(10)	0.5385 (7)	0.1033 (5)	0.2593 (4)	0.05242
C(1')	0.8277 (7)	0.4013 (6)	0.3100 (4)	0.05724
C(2')	0.8562 (7)	0.3397 (6)	0.3923 (4)	0.04593
C(3')	1.0298 (7)	0.2963 (5)	0.4389 (4)	0.04466
C(4')	1.0597 (7)	0.2098 (5)	0.3841 (4)	0.04914
C(5')	0.9983 (7)	0.2477 (5)	0.2917 (4)	0.03904
C(6')	1.0493 (7)	0.1891 (6)	0.2398 (4)	0.05288
C(7')	0.9938 (8)	0.2182 (6)	0.1555 (4)	0.06501
C(8')	0.8849 (8)	0.3074 (6)	0.1212 (4)	0.07207
C(9')	0.8346 (7)	0.3674 (5)	0.1710 (4)	0.05177
C(10')	0.8883 (7)	0.3376 (5)	0.2567 (4)	0.03850
H(1A)	0.354 (6)	0.129 (4)	0.149 (3)	0.0569
H(1B)	0.446 (6)	0.251 (5)	0.172 (3)	0.0569
H(2)	0.662 (6)	0.178 (5)	0.146 (3)	0.0569
H(3)	0.699 (6)	-0.001 (4)	0.103 (3)	0.0569
H(4A)	0.732 (6)	-0.115 (4)	0.234 (3)	0.0569
H(4B)	0.848 (6)	0.011 (5)	0.240 (3)	0.0569
H(6)	0.795 (6)	-0.098 (5)	0.372 (3)	0.0569
H(7)	0.689 (6)	-0.033 (4)	0.475 (3)	0.0569
H(8)	0.501 (6)	0.120 (4)	0.432 (3)	0.0569
H(9)	0.403 (6)	0.203 (4)	0.303 (3)	0.0569
H(1A')	0.713 (6)	0.416 (5)	0.282 (3)	0.0569
H(1B')	0.881 (6)	0.474 (5)	0.321 (3)	0.0569
H(2')	0.776 (6)	0.265 (5)	0.375 (3)	0.0569
H(3')	1.051 (6)	0.254 (5)	0.404 (3)	0.0569
H(4A')	1.183 (6)	0.188 (5)	0.407 (3)	0.0569
H(4B')	1.005 (6)	0.132 (4)	0.387 (3)	0.0569
H(6')	1.117 (8)	0.118 (6)	0.269 (4)	0.1139
H(7')	1.002 (7)	0.152 (6)	0.117 (4)	0.1139
H(8')	0.847 (7)	0.328 (6)	0.064 (4)	0.1139
H(9')	0.774 (6)	0.437 (5)	0.145 (3)	0.0569
H(81)	0.376	0.154	0.012	0.0569
H(82)	0.500	-0.106	0.043	0.0569
H(81')	0.709	0.420	0.424	0.0569
H(82')	1.150	0.465	0.494	0.0569

\*  $U_{eq} = \frac{1}{3} \text{tr } U$ .

*cis*-2,3-Tetralindiol crystallizes with two unique molecules in the asymmetric unit. In both molecules, one hydroxyl group is axial and the other is equatorial. Additionally, the two molecules are very similar with respect to corresponding bond distances and angles. However, the ring distortions are slightly different for the two molecules, as can be seen from the distances of the saturated carbon atoms from the least-squares planes calculated for the aromatic portion of each molecule [C(5) C(6) C(7) C(8) C(9) C(10) and C(5') C(6') C(7') C(8') C(9') C(10')]. C(2) is 0.32 (1) Å above and C(3) is 0.30 (1) Å below the plane, showing an equivalent distortion in the saturated portion of the molecule. In the other unit, C(2') is -0.46 (1) Å below and C(3') is 0.31 (1) Å above the plane showing an

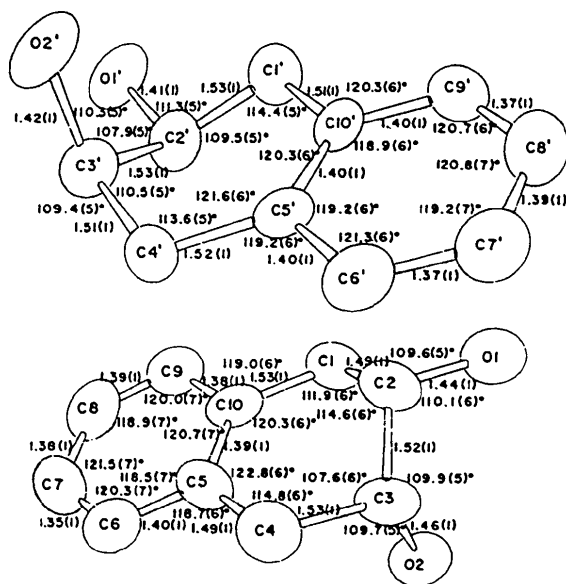


Fig. 1. Bond distances (Å), angles and labeling scheme in *cis*-2,3-tetralindiol. H atoms have been omitted.

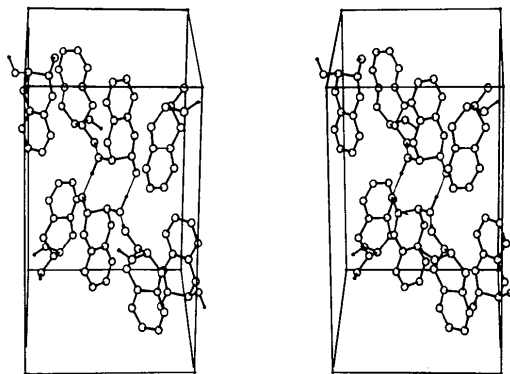


Fig. 2. Stereoview of the molecular packing diagram.

unequal distortion in the saturated portion of the molecule, possibly a result of the hydrogen-bonding requirements.

A stereoscopic diagram of the molecular packing in the unit cell is shown in Fig. 2. Both unique molecules are involved in an extensive intermolecular hydrogen-bond network. Each oxygen donates a hydrogen atom and accepts a hydrogen atom from adjacent molecular units, thus participating in two hydrogen bonds. O—H bond lengths fall between 0.80 (1) and 1.07 (1) Å while H...O distances fall between 1.66 (1) and 1.75 (1) Å. O—H...O angles are between 159.0 (3) and 179.1 (3)°. There is no intramolecular hydrogen bonding.

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## Structure du Méthyl-1 Thioxo-2 Dihydro-1,2 Pyridinecarboxylate-3 de Méthyle,\* C<sub>8</sub>H<sub>9</sub>NO<sub>2</sub>S

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**Abstract.**  $M_r = 183.23$ , orthorhombic,  $Pbca$ ,  $a = 14.123$  (3),  $b = 21.575$  (4),  $c = 11.678$  (3) Å,  $V = 3558.3$  (3) Å<sup>3</sup>,  $Z = 16$ ,  $D_x = 1.368$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 28.19$  cm<sup>-1</sup>,  $F(000) = 1536$ , 290 K,  $R = 0.077$  for 2593 observed reflections. In the crystal, the molecules are linked into pairs across a pseudo centre of symmetry by van der Waals interactions. The vector defined by the centroids of the pyridines in the pairs is nearly parallel to the  $a$  axis [3.5 (1)°] and forms angles of about 9.5° with the directions of the normals to pyridine planes. The lack of aromaticity in the pyridine ring is similar to that found in other non-hypoglycæmic mercaptopyridinecarboxylic acid derivatives.

**Introduction.** La série des acides mercaptopyridinecarboxyliques, dont les différents isomères ont été préparés avant 1970 (Delarge, 1967*a,b*, 1969; Katz, Schroeder & Cohen, 1954; Portyagina & Karpp, 1966) a fait l'objet d'une étude pharmacologique aux Etats-Unis.

Les résultats de ce travail ont été publiés par Blank, Di Tullio, Miao, Owings, Gleason, Ross, Berkoff, Saunders, Delarge & Lapière (1974) et Di Tullio,

\* Méthyl-1 thioxo-2 dihydro-1,2 nicotinate de méthyle.

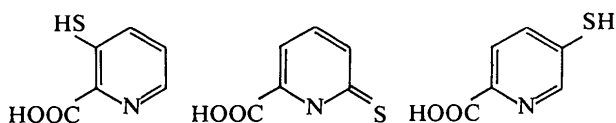
Research sponsored by the Cancer Association of Greater New Orleans.

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Berkoff, Blank, Kostos, Stack & Saunders (1974). Ils révélaient, pour certains isomères de la série, une activité hypoglycémiant de la série, une activité hypoglycémiant de la série, une activité hypoglycémiant de la série, ou ils ne manifestaient aucune activité dans ce domaine, ou ils présentaient une inversion de leurs propriétés.

Les dérivés ayant manifesté une activité intéressante sont l'acide mercapto-3 pyridinecarboxylique-2 et les acides mercapto-6 et mercapto-5 pyridinecarboxylique-2 dans une moindre mesure:



Les différents travaux effectués (Blanck *et al.*, 1974) en vue de modifier la structure des produits et d'améliorer leurs propriétés, ont conduit aux conclusions suivantes.

A l'exception de l'estérification des fonctions acide et soufrée, ou de la méthylation en position 4 sur le noyau, toute modification apportée aux composés actifs, entraînait une suppression, voire une inversion des propriétés.