

within experimental error, its mean plane making an angle of 58.6° with the four-atom plane of the neighbouring ring.

Non-bonded distances correspond to van der Waals contacts with a few exceptions. $H(55) \cdots O(1)$ is 2.42 \AA , suggesting a $C-H \cdots O$ interaction, with an angle of 168° subtended at the H. This is probably responsible for holding the tosyl group in the conformation observed. A similar, but weaker, intermolecular interaction occurs between $O(1')$ and $H(56)[-x, \frac{1}{2} + y, \frac{1}{2} - z]$. $O \cdots H$ is 2.58 \AA and the angle at H is 156° . The packing arrangement in the unit cell is shown in Fig. 2.

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References

- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
 CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
 HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
 MCCONNELL, J. F., ANGYAL, S. J. & STEVENS, J. D. (1972). *J. Chem. Soc. Perkin II*, pp. 2039–2044.
 PHILLIPS, S. E. V. & TROTTER, J. (1976). *Acta Cryst.* **B32**, 3091–3094.
 RICHE, C. & PASCARD-BILLY, C. (1975). *Acta Cryst.* **B31**, 2565–2570.
 ROSENTHAL, A. & RATCLIFFE, M. (1976). *Carbohydr. Res.* Submitted.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
 WILLY, W. E., BINSCH, G. & ELIEL, E. L. (1970). *J. Amer. Chem. Soc.* **92**, 5394–5402.

Acta Cryst. (1977). **B33**, 1007–1012

The Crystal Structure of 1-Phenyl-4,5-dihydro-7,8-dihydroxy-1*H*-benz[*g*]indazole, $C_{17}H_{14}N_2O_2$

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The crystal structure of 1-phenyl-4,5-dihydro-7,8-dihydroxy-1*H*-benz[*g*]indazole, $C_{17}H_{14}N_2O_2$, has been determined and refined by least-squares methods. The crystals are triclinic, space group $P\bar{1}$, $Z = 4$ with $a = 10.9774$ (7), $b = 11.1683$ (8), $c = 14.2764$ (8) \AA , $\alpha = 92.164$ (5), $\beta = 121.613$ (4), $\gamma = 109.132$ (6) $^\circ$. The final R value is 0.056 for the 5166 reflections used in the structure determination. The two independent molecules are linked in parallel chains by two strong hydrogen bonds, $O(14) \cdots N(23)$ (2.719 \AA) and $O(35) \cdots N(2)$ (2.728 \AA), approximately in the $[0\bar{1}1]$ direction.

Introduction

The title compound is a highly substituted pyrazole, synthesized for potential use in cancer chemotherapy (Hashem, Berlin, Chesnut & Durham, 1976). It inhibits the growth of *Bacillus subtilis* at $91 \mu\text{g ml}^{-1}$ and reduces KB cell proliferation by 50% at a concentration less than $25 \mu\text{g ml}^{-1}$. The present compound is from a group of five for which we hope to obtain structure–activity relations.

Experimental

A single crystal of parallelepiped shape with the dimensions $0.14 \times 0.18 \times 0.22$ mm, obtained from a slow evaporation of a 95% ethanol solution at room temperature, was chosen for the data collection. The space group was determined as triclinic. The least-squares cell dimensions were determined with $\text{Cu } K\alpha_1$ radiation ($\lambda = 1.5405 \text{ \AA}$) from the 2θ values of 44 reflections spaced throughout reciprocal space. The crystal data

are given in Table 1. The intensities of all 5166 reflections with $2\theta \leq 150^\circ$ were measured with Cu $K\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$) on a Nonius CAD-4 diffractom-

eter. The data were collected with $\theta-2\theta$ scans in which the θ -scan width was calculated as $(0.8 + 0.08 \tan \theta)^\circ$. The maximum scan time was 60 s with 40 s used for scanning the peak and 20 s used for scanning both high- and low- θ backgrounds. The intensity of a monitor reflection was measured every 20 reflections. The largest monitor intensity variation was 1.3% for the whole data collection. Subsequently all data were placed on a common scale. For 930 reflections, the intensities were less than twice the standard error based on counting statistics and were assigned intensities of $1.4T^{1/2}$ (T is the total count) for least-squares purposes. Experimental weights were assigned to each reflection (Ealick, van der Helm & Weinheimer, 1975).

Table 1. *Crystal data*

$C_{17}H_{14}N_2O_2$	FW = 278
Triclinic	Space group $P\bar{1}$
$a = 10.9774 (7) \text{ \AA}$	$Z = 4; F(000) = 584$
$b = 11.1683 (8)$	$V = 1359.59 \text{ \AA}^3$
$c = 14.2764 (8)$	$D_c = 1.358 \text{ g cm}^{-3}$
$\alpha = 92.164 (8)^\circ$	$D_m = 1.357$ (floatation method
$\beta = 121.613 (4)$	in hexane and CCl_4)
$\gamma = 109.132 (6)$	$\mu(\lambda = 1.5418 \text{ \AA}) = 7.439 \text{ cm}^{-1}$

Table 2. *Positional ($\times 10^4$) and thermal ($\text{\AA}^2 \times 10^4$) parameters*

Calculated standard deviations for the last digit are in parentheses. Thermal parameters are of the form: $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

Molecule <i>A</i>	<i>x</i>	<i>y</i>	<i>z</i>	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2421 (2)	118 (1)	-3535 (1)	580 (9)	353 (7)	431 (7)	257 (7)	346 (7)	203 (6)
N(2)	2549 (2)	-1056 (1)	-3584 (1)	801 (11)	390 (7)	587 (9)	361 (8)	479 (9)	279 (7)
C(3)	2863 (3)	-1160 (2)	-4358 (2)	834 (14)	460 (10)	602 (11)	406 (10)	501 (11)	259 (8)
C(4)	3291 (3)	343 (2)	-5650 (2)	838 (14)	625 (12)	595 (11)	493 (11)	551 (11)	304 (9)
C(5)	3984 (3)	1778 (2)	-5409 (2)	783 (14)	596 (12)	660 (12)	388 (11)	586 (12)	320 (9)
C(6)	3117 (2)	3615 (2)	-5454 (1)	642 (12)	426 (9)	420 (8)	234 (9)	380 (9)	205 (7)
C(7)	2393 (2)	4241 (2)	-5203 (1)	719 (12)	373 (8)	392 (8)	286 (9)	348 (9)	211 (7)
C(8)	1750 (2)	3724 (2)	-4608 (1)	606 (11)	408 (8)	339 (8)	299 (8)	300 (8)	160 (7)
C(9)	1824 (2)	2598 (2)	-4274 (1)	538 (10)	404 (8)	366 (8)	250 (8)	316 (8)	192 (7)
C(10)	2575 (2)	1966 (1)	-4518 (1)	453 (9)	340 (7)	311 (7)	196 (7)	235 (7)	141 (6)
C(11)	2670 (2)	738 (2)	-4261 (1)	486 (10)	386 (8)	365 (8)	227 (7)	280 (8)	178 (7)
C(12)	2970 (2)	-65 (2)	-4799 (2)	632 (12)	460 (9)	446 (9)	330 (9)	355 (9)	202 (8)
C(13)	3216 (2)	2471 (2)	-5129 (1)	473 (10)	395 (8)	363 (8)	202 (8)	269 (8)	154 (7)
O(14)	2269 (2)	5374 (1)	-5466 (1)	1306 (13)	528 (7)	678 (8)	586 (9)	755 (10)	425 (7)
O(15)	1029 (2)	4353 (1)	-4357 (1)	945 (10)	562 (7)	542 (7)	537 (8)	534 (8)	320 (6)
C(16)	2281 (2)	624 (2)	-2678 (1)	472 (10)	421 (8)	367 (8)	250 (8)	279 (8)	217 (7)
C(17)	3479 (2)	1749 (2)	-1843 (2)	605 (12)	498 (10)	487 (10)	203 (9)	306 (10)	140 (8)
C(18)	3340 (3)	2292 (2)	-1046 (2)	1054 (20)	720 (15)	514 (12)	424 (14)	411 (13)	111 (11)
C(19)	2049 (4)	1736 (3)	-1063 (2)	1209 (23)	1153 (21)	573 (13)	736 (19)	601 (15)	312 (14)
C(20)	873 (3)	606 (3)	-1863 (2)	820 (18)	1472 (25)	862 (17)	565 (18)	695 (16)	506 (18)
C(21)	959 (3)	24 (2)	-2703 (2)	523 (12)	813 (15)	532 (11)	186 (11)	330 (10)	196 (10)
Molecule <i>B</i>									
N(22)	2313 (2)	-4287 (1)	1849 (1)	578 (9)	372 (7)	375 (7)	245 (7)	321 (7)	184 (5)
N(23)	2092 (2)	-3971 (1)	2664 (1)	733 (11)	468 (8)	422 (8)	300 (8)	405 (8)	203 (6)
C(24)	1930 (3)	-2844 (2)	2565 (2)	767 (14)	481 (10)	448 (10)	308 (10)	415 (10)	177 (8)
C(25)	2043 (3)	-1215 (2)	1267 (2)	929 (15)	416 (9)	533 (10)	388 (10)	503 (11)	221 (8)
C(26)	2997 (3)	-939 (2)	769 (2)	933 (15)	365 (9)	580 (11)	336 (10)	521 (12)	225 (8)
C(27)	2578 (2)	-2072 (2)	-1012 (2)	705 (13)	348 (8)	525 (10)	279 (9)	432 (10)	254 (8)
C(28)	2219 (2)	-3175 (2)	-1738 (1)	621 (11)	432 (9)	436 (8)	297 (8)	384 (9)	236 (7)
C(29)	1919 (2)	-4383 (2)	-1475 (1)	585 (11)	364 (8)	430 (8)	264 (8)	345 (8)	173 (7)
C(30)	1977 (2)	-4469 (2)	-490 (1)	572 (11)	333 (8)	408 (8)	235 (8)	323 (8)	187 (7)
C(31)	2334 (2)	-3359 (2)	244 (1)	476 (10)	344 (8)	364 (8)	210 (7)	267 (8)	166 (6)
C(32)	2296 (2)	-3359 (2)	1254 (1)	509 (10)	345 (8)	355 (8)	205 (7)	267 (8)	165 (6)
C(33)	2078 (2)	-2405 (2)	1712 (1)	640 (11)	396 (8)	403 (8)	266 (8)	333 (9)	164 (7)
C(34)	2627 (2)	-2139 (2)	-21 (1)	559 (11)	334 (8)	422 (8)	220 (8)	322 (8)	163 (7)
O(35)	2124 (2)	-3185 (1)	-2732 (1)	1089 (11)	453 (7)	579 (8)	434 (8)	643 (9)	313 (6)
O(36)	1551 (2)	-5492 (1)	-2177 (1)	1005 (11)	405 (6)	528 (7)	350 (7)	561 (8)	207 (5)
C(37)	2755 (2)	-5358 (2)	1845 (1)	507 (10)	337 (8)	326 (8)	198 (7)	239 (7)	152 (6)
C(38)	1732 (2)	-6614 (2)	1616 (2)	566 (12)	412 (9)	646 (12)	155 (9)	364 (10)	194 (9)
C(39)	2208 (3)	-7623 (2)	1644 (2)	874 (16)	319 (9)	759 (14)	190 (10)	455 (13)	185 (9)
C(40)	3657 (3)	-7393 (2)	1890 (2)	898 (16)	502 (11)	552 (11)	429 (11)	402 (11)	203 (9)
C(41)	4662 (3)	-6139 (2)	2114 (2)	647 (14)	636 (13)	722 (13)	367 (11)	380 (12)	214 (11)
C(42)	4217 (3)	-5114 (2)	2097 (2)	511 (11)	421 (9)	691 (13)	180 (9)	331 (10)	190 (9)

Lorentz-polarization corrections were applied. Absorption corrections were not calculated ($\mu = 7.4 \text{ cm}^{-1}$).

Structure determination and refinement

The structure was solved by direct methods with *MULTAN* (Germain, Main & Woolfson, 1971) and the 477 E values larger than 1.68. From the distribution of the E 's, *i.e.* 0.77 for $\langle |E| \rangle$, 1.01 for $\langle |E|^2 \rangle$ and 1.04 for $\langle |E|^2 - 1 \rangle$, the space group is concluded to be centrosymmetric, $P\bar{1}$ (Hanic, 1966). The structure was refined anisotropically by block-diagonal least-squares methods (Ahmed, 1966). The quantity minimized was $\sum w_F (|kF_o| - |F_c|)^2$. The scattering factors of C, N, O were from *International Tables for X-ray Crystallography* (1974) and those of H were taken from Stewart, Davidson & Simpson (1965). The H atom locations were found from a difference Fourier synthesis and refined with isotropic temperature factors. 20 strong reflections showed evidence of extinction. A secondary isotropic extinction correction was applied in the final cycle of refinement [$2g = 2.93 \times 10^{-6}$, where $F_c^2/F_o^2 = \exp(-2gI_o)$]. In the final cycle the maximum shift/e.s.d. for a non-hydrogen coordinate was 0.31 and the agreement index for all the reflections (observed and unobserved) was $R = \sum ||kF_o| - |F_c|| / \sum |kF_o| = 0.056$.

Table 3. Positional and isotropic temperature factors for hydrogen atoms

	x	y	z	$B (\text{\AA}^2)$
H(3A)	0.299 (2)	-0.194 (2)	-0.456 (2)	5.3 (4)
H(4A)	0.384 (2)	-0.010 (2)	-0.579 (1)	4.0 (4)
H(4B)	0.213 (3)	0.006 (2)	-0.654 (2)	8.1 (6)
H(5A)	0.413 (2)	0.207 (2)	-0.598 (2)	4.4 (4)
H(5B)	0.520 (3)	0.213 (2)	-0.461 (2)	7.5 (6)
H(6A)	0.360 (2)	0.401 (2)	-0.583 (2)	5.3 (5)
H(9A)	0.134 (2)	0.225 (1)	-0.388 (1)	3.3 (3)
H(14A)	0.225 (3)	0.548 (2)	-0.614 (2)	7.9 (6)
H(15A)	0.121 (3)	0.515 (2)	-0.461 (2)	8.3 (6)
H(17A)	0.443 (3)	0.212 (2)	-0.180 (2)	7.3 (6)
H(18A)	0.420 (3)	0.315 (2)	-0.045 (2)	8.2 (6)
H(19A)	0.192 (3)	0.215 (2)	-0.051 (2)	9.0 (7)
H(20A)	0.004 (4)	0.019 (3)	-0.191 (3)	12.3 (9)
H(21A)	0.025 (3)	-0.077 (2)	-0.324 (2)	6.7 (5)
H(24A)	0.170 (2)	-0.246 (2)	0.307 (2)	5.1 (4)
H(25A)	0.240 (2)	-0.045 (2)	0.186 (1)	4.2 (4)
H(25B)	0.090 (3)	-0.135 (2)	0.064 (2)	7.2 (6)
H(26A)	0.283 (2)	-0.023 (2)	0.036 (2)	5.3 (4)
H(26B)	0.425 (3)	-0.053 (2)	0.150 (2)	7.7 (6)
H(27A)	0.274 (2)	-0.127 (2)	-0.125 (2)	4.5 (4)
H(30A)	0.177 (2)	-0.533 (2)	-0.032 (1)	3.8 (4)
H(35A)	0.225 (2)	-0.240 (2)	-0.289 (2)	5.4 (5)
H(36A)	0.155 (3)	-0.527 (2)	-0.279 (2)	8.7 (7)
H(38A)	0.080 (2)	-0.674 (2)	0.154 (2)	6.5 (5)
H(39A)	0.154 (3)	-0.847 (2)	0.153 (2)	7.6 (6)
H(40A)	0.397 (2)	-0.813 (2)	0.189 (2)	5.8 (5)
H(41A)	0.571 (3)	-0.597 (2)	0.230 (2)	7.1 (6)
H(42A)	0.489 (2)	-0.425 (2)	0.224 (2)	5.9 (5)

Standard deviations are in parentheses.

All 5166 reflections were used for standard deviation calculations. The final parameters are given in Tables 2 and 3.*

Discussion

There are two independent molecules in the asymmetric unit. For structures containing the pyrazole ring one finds a number of examples in which the asymmetric unit contains more than one molecule (for example, Bechtel, Gaultier & Hauw, 1973*a,b*; Ehrlich, 1960). An *ORTEP* drawing (Johnson, 1965) for both molecules, showing the numbering scheme, is given in Fig. 1. The bond lengths and angles are shown in Figs. 2 and 3 respectively. The standard deviations are 0.002 to 0.005 \AA for bond lengths and 0.2 to 0.3 $^\circ$ for bond angles. For C-H bonds the lengths are in the range 0.85 to 0.98 \AA , and for O-H bonds 0.96 to 0.98 \AA . Agreement between corresponding bond lengths in these two molecules is fairly good. Even so, the differences are larger than would be expected from the standard deviations. The largest bond-length discrepancy is between C(4)-C(5), 1.467 (3) \AA , in molecule *A* and the corresponding bond C(25)-C(26), 1.510 \AA , in molecule *B*. A final difference Fourier synthesis shows no indication of disorder for either C(4) or C(5).

The corresponding bond lengths in the five-membered rings agree very well in the two molecules. The largest difference is 0.004 \AA . However, they do not agree with the bond lengths in pyrazole obtained from neutron diffraction data (Larsen, Lehmann, S\o tofte & Rasmussen, 1970), microwave data (Nygaard *et al.*, 1974) and low-temperature X-ray diffraction data (LaCour & Rasmussen, 1973), which are not consistent among themselves. On average, the

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32146 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

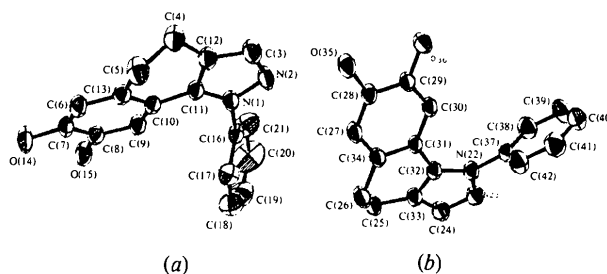


Fig. 1. *ORTEP* drawing of the two molecules in the asymmetric unit with the numbering scheme.

bonds N(1)—N(2) and C(11)—N(1) are elongated by 0.013 Å in the present structure while there are insignificant differences in the other three bonds. The elongation of these bonds is probably caused by substitution on C(11), C(12), and N(1). Other comparisons made with 5*H*-pyrazolo[3,4-*d*]pyrimidine-4-thione (Gadret, Goursole & Leger, 1974) and allopurinol (Prusiner & Sundaralingam, 1972) also show an elongation of the C(11)—N(1) bond, and in addition a shortening of the C(3)—C(12) bond in the present structure.

The five-membered rings in both molecules are planar and the bond lengths indicate that they are aromatic. Table 4 gives the least-squares planes of the five-membered rings and the deviations of the atoms from the planes. It is interesting to note that despite the aromaticity of the five-membered rings, neither phenyl C atom C(16) nor C(37) is in the plane of the respective five-membered ring. The distances are 0.177 Å for

C(16) and 0.212 Å for C(37). Also the fused benzene rings and phenyl rings are planar, with an average distance from the respective least-squares planes of 0.003 Å and a maximum distance of 0.011 Å for the 36 atoms involved. The exocyclic atoms are out of the planes, however. For the fused benzene rings the largest distances are observed for C(11) (0.060 Å) and C(32) (0.109 Å) with smaller distances for C(5) (0.021 Å) and C(26) (0.018 Å) while the average distance of the O atoms is 0.025 Å. The N atoms are 0.076 Å [N(1)] and 0.027 Å [N(22)] out of the plane of the respective phenyl rings. The interplanar angles between the least-squares planes of the five-membered rings and the attached phenyl group are 63.0° for molecule *A* and 115.7° for molecule *B*. In general, the conformation of both molecules is the same. From Table 4 it can be seen that the phenyl group and hydroxy groups are on opposite sides of the five-membered ring for both molecules. However, the conformations in detail are

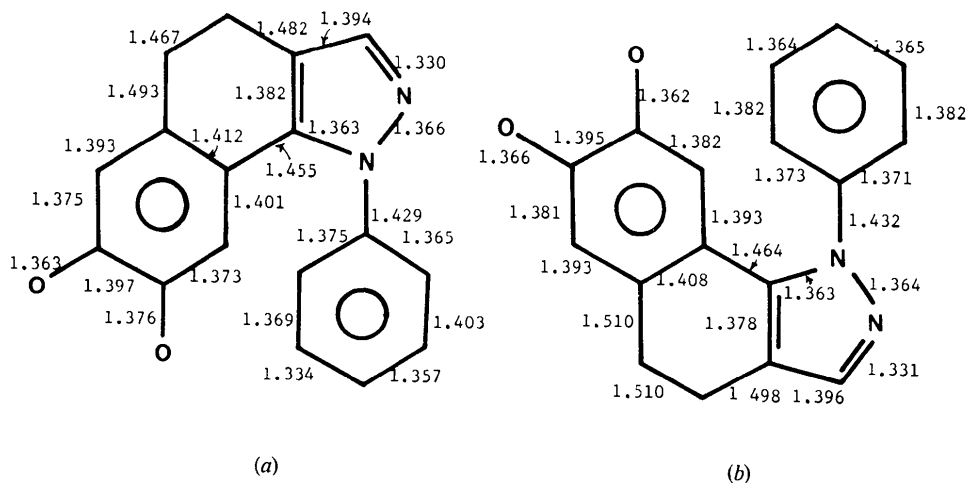


Fig. 2. Bond distances (Å). Standard deviations are between 0.002 and 0.005 Å.

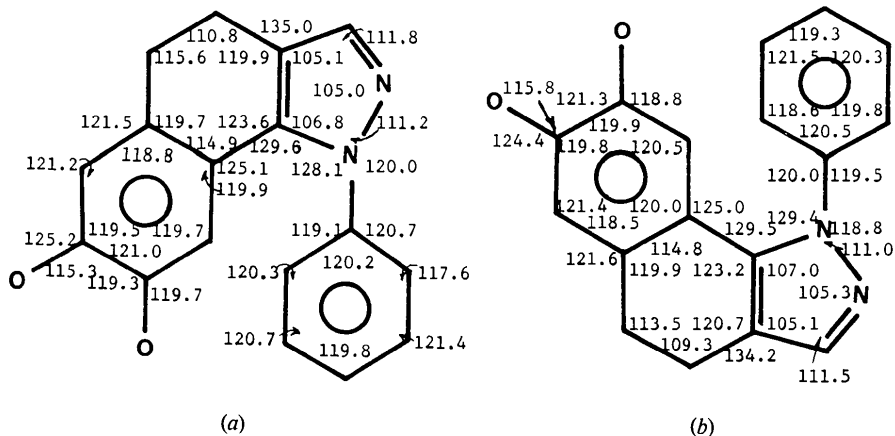


Fig. 3. Bond angles (°). Standard deviations are between 0.2 and 0.3°.

different, as can be seen from the conformational angles in the cyclohexadiene rings (Table 5). Molecule *A* is the enantiomer of *B* (from the coordinates of Table 2). Molecules related to *A* and *B* by a center of symmetry have opposite signs for the conformational angles from those given in Table 5.

An ORTEP packing drawing (Johnson, 1965) showing the intermolecular hydrogen bonds is given in Fig. 4. The detailed description of the hydrogen bonding is presented in Table 6. The two independent molecules

are linked in parallel chains by two strong hydrogen bonds, O(14)···N(23) (2.719 Å) and O(35)···N(2) (2.728 Å) in the [0 $\bar{1}$ 1] direction. The parallel chains are cross-linked in every other pair by a hydrogen bond, O(15)···O(36) (2.841 Å) in the [011] direction. The whole network is parallel to the *bc* plane and located at $x = 0.25$. The networks stack along the [100] direction through van der Waals contacts alone. The strong hydrogen bonding with N(2) and N(23) as acceptors probably indicates localized electron density on these atoms, in line with the observation of Bechtel, Gaultier & Hauw (1973*b*) for a 1-phenyl-5-methyl-3-pyrazolone. One may speculate that the strong nucleophilic character at N(2) and N(23) is partially responsible for the antineoplastic activity of the compound.

Table 4. Deviations (Å) from the planes of the five-membered rings

Least-squares planes were calculated from the five atoms in the five-membered rings.

$$\text{Molecule } A: 6.952x + 0.579y + 2.884z = 0.672$$

$$\text{Molecule } B: 7.394x + 1.084y + 1.621z = 1.544$$

x , y , and z are fractional coordinates.

Molecule <i>A</i>		Molecule <i>B</i>	
N(1)	-0.002	N(22)	0.002
N(2)	0.005	N(23)	0.005
C(3)	-0.006	C(24)	-0.009
C(12)	0.005	C(33)	0.010
C(11)	-0.002	C(32)	-0.007
C(16)	0.177	C(37)	0.212
C(4)	0.006	C(25)	0.041
C(5)	0.641	C(26)	0.695
C(10)	-0.071	C(31)	-0.142
C(13)	0.227	C(34)	0.164
O(14)	-0.360	O(35)	-0.761
O(15)	-0.962	O(36)	-1.345

Table 6. Hydrogen bonding (Å)

<i>D</i> -H··· <i>A</i>	<i>D</i> -H	<i>D</i> ··· <i>A</i>	H··· <i>A</i>
O(14)-H(14 <i>A</i>)···N(23 ⁱ)	0.96	2.719	1.78
*O(15)-H(15 <i>A</i>)···O(15 ⁱⁱ)	0.98	2.925	2.38
O(36)-H(36 <i>A</i>)···O(15 ⁱⁱⁱ)	0.92	2.841	1.98
O(35)-H(35 <i>A</i>)···N(2)	0.89	2.728	1.87

Symmetry code

(i) $x, y + 1, z - 1$

(iv) $-x, -y, -z - 1$

(ii) $-x, -y + 1, -z - 1$

(v) $-x + 1, -y, -z - 1$

(iii) $x, y - 1, z$

* More likely a van der Waals contact than a hydrogen bond, H(15*A*)···H(15*A*ⁱⁱ): 2.16 Å. Other short van der Waals contacts between H atoms: H(4*B*)···H(20*A*^{iv}) = 2.16 and H(4*A*)···H(4*A*^v) = 2.28 Å.

Table 5. Conformational angles (°) around the cyclohexadiene rings

Estimated standard deviations are 0.6°.

Molecule <i>A</i>		Molecule <i>B</i>	
C(10)-C(11)-C(12)-C(4)	3.0	C(31)-C(32)-C(33)-C(25)	-6.9
C(11)-C(12)-C(4)-C(5)	27.8	C(32)-C(33)-C(25)-C(26)	-26.9
C(12)-C(4)-C(5)-C(13)	-45.2	C(33)-C(25)-C(26)-C(34)	47.6
C(4)-C(5)-C(13)-C(10)	34.5	C(25)-C(26)-C(34)-C(31)	-39.0
C(5)-C(13)-C(10)-C(11)	-2.8	C(26)-C(34)-C(31)-C(32)	4.8
C(13)-C(10)-C(11)-C(12)	-16.3	C(34)-C(31)-C(32)-C(33)	19.1

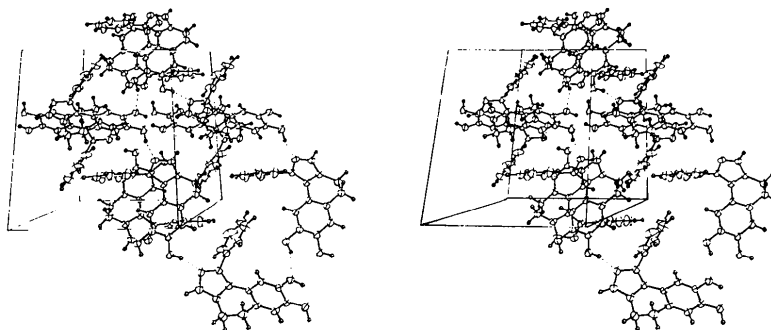


Fig. 4. ORTEP packing drawing and intermolecular hydrogen bonding (shown in dotted lines). View is down the *a* axis.

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References

- AHMED, F. R. (1966). Program NRC-10, National Research Council, Ottawa, Canada.
- BECHTEL, F., GAULTIER, J. & HAUW, C. (1973*a*). *Cryst. Struct. Commun.* **3**, 469–472.
- BECHTEL, F., GAULTIER, J. & HAUW, C. (1973*b*). *Cryst. Struct. Commun.* **3**, 473–476.
- EALICK, S. E., VAN DER HELM, D. & WEINHEIMER, A. J. (1975). *Acta Cryst.* **B31**, 1618–1626.
- EHRlich, H. W. W. (1960). *Acta Cryst.* **13**, 946–952.
- GADRET, M., GOURSOLLE, M. & LEGER, J. M. (1974). *Acta Cryst.* **B30**, 1598–1602.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
- HANIC, F. (1966). *Acta Cryst.* **21**, 332–340.
- HASHEM, M. M., BERLIN, K. D., CHESNUT, R. W. & DURHAM, N. N. (1976). *J. Med. Chem.* **19**, 229–239.
- International Tables for X-ray Crystallography* (1974). Vol. IV, p. 72. Birmingham: Kynoch Press.
- JOHNSON, C. K. (1965). *ORTEP*. Oak Ridge National Laboratory Report ORNL-3794.
- LACOUR, T. & RASMUSSEN, V. E. (1973). *Acta Chem. Scand.* **27**, 1845–1854.
- LARSEN, F. K., LEHMANN, M. S., SØTOFTE, I. & RASMUSSEN, S. E. (1970). *Acta Chem. Scand.* **24**, 3248–3258.
- NYGAARD, L., CHRISTEN, D., NIELSEN, J. T., PEDERSEN, E. J., SNERLING, O., VESTERGAARD, E. & SORENSEN, G. O. (1974). *J. Mol. Struct.* **22**, 401–413.
- PRUSINER, P. & SUNDARALINGAM, M. (1972). *Acta Cryst.* **B28**, 2148–2152.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1977). **B33**, 1012–1016

The Crystal Structure of 2-Amino-4,5-dihydro-7,8-dimethoxynaphtho[1,2-*d*]thiazole

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The crystal structure of 2-amino-4,5-dihydro-7,8-dimethoxynaphtho[1,2-*d*]thiazole has been determined with three-dimensional X-ray diffraction techniques. The crystals are orthorhombic, space group *Pbca*, with eight molecules in the unit cell; $a = 6.778$ (1), $b = 21.041$ (2), $c = 17.410$ (1) Å. The structure was refined by block-diagonal least-squares techniques to a final *R* value of 0.055 for all 2565 reflections. The structure contains hydrogen-bonded dimers across a center of symmetry. The thiazole ring is perfectly planar but the molecule, as a whole, is slightly bent. Two possible resonance forms do not completely explain the bond distances observed in the thiazole ring.

Introduction

The title compound is a highly substituted thiazole derivative synthesized for potential use in cancer chemotherapy by Hashem, Berlin, Chesnut & Durham (1976). Tests conducted by Berlin show complete growth inhibition of *Bacillus subtilis* at a compound concentration of 25 µg ml⁻¹ and 50% growth inhibition of human tumor cells (KB) at a compound concentration of 50 µg ml⁻¹ (Hashem *et al.*, 1967). The compound is from a group of pyrazolo, isoxazolo and thiazolo systems containing the *A*, *C*, and *D* rings of a heterosteroid. The structure determination of a pyrazolo derivative in this group of compounds has been reported previously (Wu, van der Helm & Berlin, 1977).

Experimental

A yellow block-like crystal showing ten identifiable faces and with approximate dimensions of 0.3 × 0.3 × 0.2 mm was selected from a sample recrystallized from ethanol. The space group was determined as *Pbca* from systematic absences with a GE manual diffractometer. Unit-cell data (Table 1) were measured with Cu $K\alpha_1$ ($\lambda = 1.54051$ Å) radiation. The 2565 intensity data, comprising all unique reflections with 2θ less than 150°, were collected with Ni-filtered Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å) on a Nonius CAD-4 automatic diffractometer using θ - 2θ scan techniques. A variable scan width, calculated as $(1.0 + 0.1 \tan \theta)^\circ$, was used. The aperture, located 173 mm from the