

There is only a slight overlap of bases, a situation similar to that found in the three other active cytokinins. Conformational features observed in this structure are in agreement with those observed for active cytokinins and are in disagreement with those in *N*<sup>6</sup>-benzoyladenine which is reported to show mild cytokinin activity.

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## Structure of 5-(4,5,6,7-Tetrahydrobenzimidazol-2-yl)valeronitrile, C<sub>12</sub>H<sub>17</sub>N<sub>3</sub>

BY YUKISHIGE KITANO

*Toray Research Center, Inc., Sonoyama, Ohtsu 520, Japan*

HARUYO SATO AND SHINZO IMAMURA

*Chemicals Research Laboratory, Basic Research Laboratories, Toray Industries, Inc., 9-1, Ooe-cho, Minato-ku, Nagoya 455-91, Japan*

AND TAMAICHI ASHIDA

*Faculty of Engineering, Nagoya University, Chikusa-ku, Nagoya 464, Japan*

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**Abstract.**  $M_r = 203.29$ , monoclinic,  $P2_1/n$ ,  $a = 9.794$  (3),  $b = 9.044$  (4),  $c = 26.605$  (3) Å,  $\beta = 97.67$  (3)°,  $V = 2335.5$  Å<sup>3</sup>,  $Z = 8$ ,  $D_m = 1.16$ ,  $D_x = 1.157$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu K}\alpha) = 1.5405$  Å,  $\mu = 0.56$  mm<sup>-1</sup>,  $F(000) = 880$ ,  $T = 298$  K. Final  $R = 0.098$  for 2402 independent reflections. The aliphatic side chain of one molecule is statistically disordered over two sites and therefore three kinds of conformation of the side chains are observed: *trans-trans-gauche* with respect to the ring carbon for one molecule, and *gauche-trans-trans* and *gauche-trans-gauche* for the others. Both molecules are linked together by NH...N hydrogen bonds in strings of an infinite length along *a*.

**Introduction.** In the reaction of 2-aminocyclohexanone oxime with ethyl acetimidate hydrochloride a new compound has been synthesized (Sato, Imamura & Kitano, 1983). The structure of the product was characterized by chemical and spectroscopic methods but could not be established exclusively. The compound, obtained in the form of single crystals, was then subjected to X-ray analysis and the structure was determined as 5-(4,5,6,7-tetrahydrobenzimidazol-2-yl)valeronitrile.

**Experimental.** Crystals grown from an ethyl acetate solution by slow evaporation at room temperature as

colorless monoclinic laths elongated along **a**. Density measured by flotation in carbon tetrachloride-hexane mixture. Rigaku automated four-circle diffractometer, graphite-monochromated Cu  $K\alpha$  radiation; single crystal,  $0.35 \times 0.17 \times 0.07$  mm, mounted with **a** coincident with the goniostat axis. Unit-cell dimensions determined by least-squares refinement of angular settings of 15 reflections.  $2\theta$ - $\omega$  scan, scan range  $\Delta\omega = (0.8 + 0.142 \tan\theta)^\circ$ , scan rate and background counting time  $10^\circ \text{ min}^{-1}$  and 1 s at each terminus of the scans for  $0 < 2\theta \leq 100^\circ$ ,  $5^\circ \text{ min}^{-1}$  and 2 s for  $100 < 2\theta \leq 110^\circ$ ,  $3^\circ \text{ min}^{-1}$  and 4 s for  $110 < 2\theta \leq 128^\circ$ . Range of  $hkl$ :  $0 \leq h \leq 10$ ,  $0 \leq k \leq 10$ ,  $-31 \leq l \leq 30$ . Three standard reflections ( $0\ 0\ 10$ ,  $0\ 4\ 5$ ,  $4\ 0\ \bar{2}$ ) measured periodically, no significant change in intensities. 3275 [ $2402 F_o \geq 2\sigma(F_o)$ ] unique reflections.  $R_{\text{int}} = 0.083$ . Corrections for Lorentz-polarization, not for absorption. Direct methods (*MULTAN78*, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); block-diagonal least-squares procedure, anisotropic temperature factors for non-H atoms, isotropic for H. First  $E$  map calculated using  $220 |E| > 1.92$  revealed all non-H atoms except for terminal N(3) atoms of the two independent molecules, located by successive Fourier calculations. In course of refinements temperature factors of C(9) and C(10) in second molecule remained anomalously high; difference syntheses examined revealed C(9) and C(10) disordered at the two sites, their occupancies assumed to be 0.5 and not varied in refinements. H atoms of ordered portion of molecule easily located on a difference map and refined isotropically, but those of disordered portion fixed at calculated positions with  $B = B_{\text{eq}}$  of bonded C. Function minimized  $\sum w(|F_o| - |F_c|)^2$  with  $w = [\sigma^2(F_o) + a|F_o| + b|F_o|^2]^{-1}$ , where  $\sigma(F_o)$  is standard deviation based on counting statistics. Final refinement ( $a = 0.1690$ ,  $b = 0.0011$ ) gave  $R = 0.098$ ,  $wR = 0.104$ ,  $S = 1.13$ .  $\Delta/\sigma = 0.05$ .  $\Delta\rho = 0.10\text{--}0.24 \text{ e } \text{\AA}^{-3}$ . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computer programs from *UNICS-NAGOYA* (Ashida, 1981).

**Discussion.** Final atomic parameters are in Table 1.\* The bond distances, angles and torsion angles are listed in Table 2. The conformations of the two molecules are shown in Fig. 1.

The aliphatic side chains turn approximately normal to the benzimidazole ring plane. The torsion angles flanking the C(9)–C(10) bond differ between the three conformers. In molecule **A** the side chain has a *trans-trans-gauche* conformation with respect to the

\* Lists of structure factors, anisotropic thermal parameters for non-H atoms and atomic parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38701 (16 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 ( $\times 10^4$ ) with *e.s.d.*'s in parentheses and equivalent isotropic thermal parameters ( $\times 10$ ) as defined by Hamilton (1959)

Molecule A	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
N(1)	1413 (4)	1600 (5)	1454 (2)	37
N(2)	3623 (4)	1426 (5)	1382 (2)	39
N(3)	4614 (8)	3276 (10)	-649 (3)	103
C(1)	1878 (5)	308 (6)	1679 (2)	35
C(2)	3236 (5)	190 (6)	1635 (2)	36
C(3)	4127 (6)	-1068 (8)	1821 (2)	50
C(4)	3257 (8)	-2243 (9)	2035 (4)	83
C(5)	2071 (8)	-1763 (11)	2265 (4)	93
C(6)	1060 (6)	-783 (7)	1937 (2)	48
C(7)	2504 (5)	2252 (6)	1277 (2)	37
C(8)	2413 (7)	3678 (7)	991 (3)	54
C(9)	2403 (8)	3385 (8)	408 (3)	63
C(10)	2280 (8)	4757 (8)	105 (3)	69
C(11)	2357 (9)	4497 (10)	-447 (3)	76
C(12)	3643 (8)	3834 (9)	-556 (3)	70

Molecule B	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
N(1)	1412 (4)	2500 (5)	6446 (2)	39
N(2)	3639 (4)	2380 (5)	6407 (2)	41
N(3)	4565 (9)	2639 (11)	4260 (4)	118
C(1)	1846 (5)	1167 (6)	6665 (2)	36
C(2)	3218 (5)	1123 (6)	6640 (2)	37
C(3)	4107 (6)	-143 (8)	6828 (2)	50
C(4)	3237 (9)	-1334 (9)	7036 (4)	90
C(5)	2000 (9)	-898 (11)	7229 (4)	90
C(6)	1038 (6)	87 (7)	6911 (2)	48
C(7)	2511 (5)	3187 (6)	6300 (2)	36
C(8)	2417 (7)	4662 (7)	6056 (2)	51
C(9)	1925 (10)	4582 (12)	5458 (4)	35
C(9')	2817 (20)	4648 (18)	5527 (7)	90
C(10)	3119 (10)	3918 (11)	5197 (4)	31
C(10')	1899 (20)	3947 (21)	5131 (6)	89
C(11)	2540 (11)	3929 (9)	4622 (3)	85
C(12)	3684 (10)	3265 (9)	4429 (3)	81

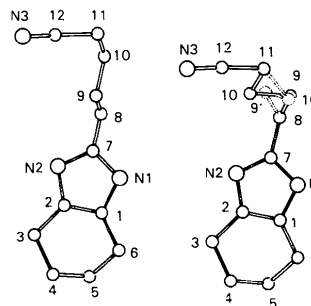


Fig. 1. Perspective views of molecule **A** (left) and molecule **B** (right) viewed along the imidazole plane normal, with the numbering scheme. The disordered portions are shown by dotted lines.

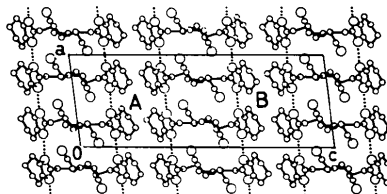


Fig. 2. The molecular arrangements in the crystal viewed along **b**. Hydrogen bonds are shown by dotted lines. **A** and **B** indicate the molecules in Table 1. Disordered C(9') and C(10') atoms of molecule **B** are omitted for clarity.

Table 2. Bond distances (Å), angles and torsion angles (°) for molecules *A* and *B* with e.s.d.'s in parentheses

	<i>A</i>		<i>B</i>		<i>A</i>		<i>B</i>		<i>A</i>		<i>B</i>	
N(1)—C(1)	1.364 (7)	1.381 (7)	C(7)—C(8)	1.493 (9)	1.482 (9)	C(1)—N(1)—C(7)	107.0 (5)	108.1 (5)	N(1)—C(7)—N(2)	110.4 (5)	111.2 (5)	
N(2)—C(2)	1.383 (7)	1.384 (8)	C(8)—C(9)	1.57 (1)	1.60 (1)	C(2)—N(2)—C(7)	106.6 (5)	105.0 (5)	N(1)—C(7)—C(8)	123.7 (5)	122.5 (5)	
N(1)—C(7)	1.359 (7)	1.345 (7)	C(8)—C(9')	—	1.51 (2)	N(1)—C(1)—C(2)	107.7 (5)	104.9 (5)	N(2)—C(7)—C(8)	125.9 (5)	126.3 (5)	
N(2)—C(7)	1.324 (7)	1.323 (7)	C(9)—C(10)	1.48 (1)	1.56 (2)	N(2)—C(2)—C(1)	108.3 (5)	110.8 (5)	C(7)—C(8)—C(9)	110.4 (6)	112.8 (6)	
C(1)—C(2)	1.354 (8)	1.355 (8)	C(9')—C(10')	—	1.44 (3)	C(6)—C(1)—C(2)	125.5 (5)	126.9 (5)	C(7)—C(8)—C(9')	—	113.1 (9)	
C(2)—C(3)	1.479 (9)	1.485 (9)	C(10)—C(11)	1.50 (1)	1.56 (2)	C(1)—C(2)—C(3)	125.0 (5)	123.2 (5)	C(8)—C(9)—C(10)	112.8 (7)	108.4 (8)	
C(3)—C(4)	1.52 (1)	1.52 (1)	C(10')—C(11)	—	1.57 (2)	C(2)—C(3)—C(4)	109.2 (6)	109.7 (6)	C(8)—C(9')—C(10')	—	118.1 (16)	
C(4)—C(5)	1.45 (1)	1.43 (1)	C(11)—C(12)	1.46 (1)	1.43 (2)	C(3)—C(4)—C(5)	118.0 (8)	118.5 (8)	C(9)—C(10)—C(11)	113.1 (7)	103.8 (8)	
C(5)—C(6)	1.52 (1)	1.48 (1)	C(12)—N(3)	1.13 (1)	1.17 (1)	C(4)—C(5)—C(6)	115.5 (8)	117.7 (8)	C(9')—C(10')—C(11)	—	110.7 (15)	
C(6)—C(1)	1.496 (8)	1.465 (9)				C(5)—C(6)—C(1)	107.5 (6)	108.5 (6)	C(10)—C(11)—C(12)	114.6 (7)	98.7 (8)	
						N(1)—C(1)—C(6)	126.9 (5)	128.1 (5)	C(10')—C(11)—C(12)	—	137.8 (11)	
						N(2)—C(2)—C(3)	126.7 (5)	126.0 (5)	C(11)—C(12)—N(3)	177.4 (10)	175.4 (11)	
	<i>A</i>	<i>B</i>	<i>A</i>	<i>B</i>	<i>A</i>	<i>B</i>	<i>A</i>	<i>B</i>	<i>A</i>	<i>B</i>	<i>A</i>	<i>B</i>
N(1)—C(7)—N(2)—C(2)	0.3 (6)	-0.8 (6)	C(3)—C(4)—C(5)—C(6)	53.8 (12)	48.9 (13)	C(8)—C(9)—C(10)—C(11)	176.5 (5)	-178.7 (6)				
C(7)—N(2)—C(2)—C(1)	-0.5 (6)	0.9 (7)	C(4)—C(5)—C(6)—C(1)	-42.5 (10)	-38.5 (11)	C(8)—C(9')—C(10')—C(11)	—	-175.4 (10)				
N(2)—C(2)—C(1)—N(1)	0.5 (6)	-0.6 (7)	C(5)—C(6)—C(1)—C(2)	16.6 (9)	12.5 (10)	C(9)—C(10)—C(11)—C(12)	-60.7 (10)	-178.2 (7)				
C(2)—C(1)—N(1)—C(7)	-0.3 (6)	0.1 (6)	C(6)—C(1)—C(2)—C(3)	2.0 (10)	5.6 (10)	C(9')—C(10')—C(11)—C(12)	—	65.8 (23)				
C(1)—N(1)—C(7)—N(2)	0.0 (7)	0.4 (7)	C(7)—C(8)—C(9)—C(10)	178.6 (5)	-71.3 (10)	C(10)—C(11)—C(12)—N(3)	123.1 (21)	113.1 (14)				
C(1)—C(2)—C(3)—C(4)	4.0 (9)	1.0 (9)	C(7)—C(8)—C(9')—C(10')	—	71.0 (18)	C(10')—C(11)—C(12)—N(3)	—	87.9 (14)				
C(2)—C(3)—C(4)—C(5)	-31.3 (11)	-27.0 (12)										

ring carbon C(7), while in molecule *B* the two conformers are *gauche-trans-trans* and *gauche-trans-gauche*.

The two independent benzimidazole rings are almost identical. The bond distances and angles within the six-membered rings are all close to those found in 4,5,6,7-tetrahydrobenzimidazole hydrochloride (Carlström, Hacksell & Jönsson, 1981). The C(1)—C(6)—C(5) and C(2)—C(3)—C(4) angles are slightly smaller and the C(3)—C(4)—C(5) and C(4)—C(5)—C(6) angles are slightly larger than the normal  $sp^3$ -hybridized C—C—C angle ( $\sim 111^\circ$ ) of cyclohexane. The imidazole rings are planar, whereas both cyclohexene rings have distorted half-chair conformations with C(4) and C(5) atoms displaced from the imidazole ring planes by 0.15 and -0.45 Å in molecule *A* and 0.07 and -0.49 Å in molecule *B* respectively. The dimensions of the molecules appear to be similar to those in other imidazole rings in compounds such as 6-purinethiol (Sletten, Sletten & Jensen, 1969), 2-(4-thiazolyl)benzimidazole (Trus & Marsh, 1973) and imidazole itself (Martínez-Carrera, 1966).

The packing of the molecules viewed along *b* is shown in Fig. 2. The molecules are linked into chains along *a* by two kinds of NH...N hydrogen bonds. This hydrogen bonding is characterized by the following

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### *trans*-[1,1'-Bibenzo[*c*]thienylidene]-3,3'-dione, C<sub>16</sub>H<sub>8</sub>O<sub>2</sub>S<sub>2</sub>

BY RONALD L. AMEY AND RICHARD L. HARLOW

Central Research and Development Department, E. I. du Pont de Nemours & Company, Experimental Station, Wilmington, DE 19898, USA

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**Abstract.**  $M_r = 296.37$ , monoclinic,  $P2_1/c$ ,  $T = 173$  K,  $a = 3.844$  (1),  $b = 14.188$  (4),  $c = 11.343$  (3) Å,  $\beta = 94.65$  (1)°,  $V = 616.6$  Å<sup>3</sup>,  $D_x = 1.596$  g cm<sup>-3</sup>,  $Z = 2$ , 0108-2701/83/111549-03\$01.50

parameters: N(1)(molecule *A*)...N(2)(molecule *B*) at  $-\frac{1}{2}+x$ ,  $\frac{1}{2}-y$ ,  $-\frac{1}{2}+z$  2.856 (7), H(N1)...N(2) 1.87 (6) Å, N(1)—H(N1)...N(2) 173 (6)° and N(1)(molecule *B*)...N(2)(molecule *A*) at  $-\frac{1}{2}+x$ ,  $\frac{1}{2}-y$ ,  $\frac{1}{2}+z$  2.833 (7), H(N1)...N(2) 1.89 (6) Å, N(1)—H(N1)...N(2) 176 (5)°. Apart from the hydrogen-bond interactions there are no close intermolecular contacts.

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Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å,  $\mu = 4.19$  cm<sup>-1</sup>,  $F(000) = 304$ ,  $R = 0.059$  for 776 reflections with  $I > 2\sigma(I)$ . A comparison of the dithia analog presented

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