$Z = 8$
$D_x = 1.388$ Mg m^{-3}

0.30 × 0.20 × 0.11 mm
Colourless
Crystal source: from ethyl acetate/n-hexane

Data collection

Rigaku AFC-5R diffractometer
ω scans
Absorption correction: none
1686 measured reflections
1686 independent reflections
1176 observed reflections [$I > 3\sigma(I)$]

$\theta_{\max} = 28°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 23$
3 standard reflections
frequency: 15 min
intensity variation: ±0.3%

Refinement

Refinement on F
$R = 0.038$
$wR = 0.039$
$S = 1.42$
1176 reflections
140 parameters
$w = 1/[\sigma^2(|F_o|) + 0.0001(|F_o|)^2]$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELX76* (Sheldrick, 1976)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
O(1)	0.1565 (2)	0.9448 (1)	0.0971 (1)	1.62 (4)
O(2)	−0.0057 (2)	1.1083 (1)	0.1007 (1)	2.28 (5)
O(3)	−0.0144 (2)	0.5313 (1)	0.1682 (1)	1.90 (4)
C(1)	0.1845 (2)	0.8167 (2)	0.1214 (1)	1.36 (6)
C(3)	0.0066 (3)	0.9954 (2)	0.1052 (1)	1.60 (5)
C(4)	−0.1330 (3)	0.9112 (2)	0.1152 (1)	1.56 (6)
C(5)	−0.1171 (2)	0.7882 (2)	0.1130 (1)	1.37 (6)
C(6)	0.0456 (2)	0.7253 (2)	0.1043 (1)	1.25 (5)
C(7)	0.0615 (2)	0.6281 (2)	0.1658 (1)	1.39 (6)
C(8)	0.1815 (3)	0.6740 (2)	0.2221 (1)	1.68 (6)
C(9)	0.2108 (3)	0.8117 (2)	0.2036 (1)	1.68 (6)
C(10)	0.0556 (3)	0.6626 (2)	0.0294 (1)	1.82 (6)

Table 2. *Selected geometric parameters* (Å, °)

O(1)—C(1)	1.453 (2)	O(1)—C(3)	1.344 (3)
O(2)—C(3)	1.209 (2)	O(3)—C(7)	1.203 (2)
C(1)—C(6)	1.526 (3)	C(1)—C(9)	1.523 (3)
C(3)—C(4)	1.460 (3)	C(4)—C(5)	1.317 (3)
C(5)—C(6)	1.494 (3)	C(6)—C(7)	1.536 (3)
C(6)—C(10)	1.529 (3)	C(7)—C(8)	1.504 (3)
C(8)—C(9)	1.524 (3)		
C(1)—O(1)—C(3)	119.0 (1)	C(1)—C(6)—C(7)	102.5 (1)
O(1)—C(1)—C(6)	114.8 (1)	C(1)—C(6)—C(10)	115.1 (2)
O(1)—C(1)—C(9)	111.0 (1)	C(5)—C(6)—C(7)	107.4 (1)
C(6)—C(1)—C(9)	106.6 (2)	C(5)—C(6)—C(10)	109.8 (2)
O(1)—C(3)—O(2)	117.8 (2)	C(7)—C(6)—C(10)	111.1 (2)
O(1)—C(3)—C(4)	118.4 (2)	O(3)—C(7)—C(6)	124.1 (2)
O(2)—C(3)—C(4)	123.7 (2)	O(3)—C(7)—C(8)	126.0 (2)
C(3)—C(4)—C(5)	122.0 (2)	C(6)—C(7)—C(8)	109.9 (2)
C(4)—C(5)—C(6)	122.4 (2)	C(7)—C(8)—C(9)	105.2 (2)
C(1)—C(6)—C(5)	110.5 (2)	C(1)—C(9)—C(8)	103.4 (2)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	A	D—H	H···A	D···A	D—H···A
C(8)	H′(C8)	O(2$^{\rm i}$)	1.01 (2)	2.76 (2)	3.525 (3)	133 (1)
C(4)	H(C4)	O(3$^{\rm ii}$)	0.98 (2)	2.34 (2)	3.292 (2)	163 (1)
C(9)	H(C9)	O(3$^{\rm iii}$)	1.01 (2)	2.74 (2)	3.681 (2)	154 (1)

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, z$; (ii) $-\frac{1}{2} - x, y + \frac{1}{2}, z$; (iii) $-x, y + \frac{1}{2}, \frac{1}{2} - z$.

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. H atoms were found in difference syntheses and refined with an overall isotropic temperature factor [$U_{iso} = 0.027 (2)$ Å^2]. Refinement was by full-matrix least-squares methods. Programs used were *SHELXS86* (Sheldrick, 1985), *SHELX76* (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). Most of the calculations were performed at the Weizmann Institute of Science, Israel.

I would like to thank the late Dr R. C. Boscaini of the Departamento de Química, FFCLRP, Universidade de São Paulo, Brazil, for the gift of the sample used in this work, the Associação de Amigos do Instituto Weizmann em São Paulo for a scholarship, the Weizmann Institute of Science for providing X-ray facilities, and Dr F. Frolow for collecting the data.

Lists of structure factors and anisotropic displacement parameters have been deposited with the IUCr (Reference: LI1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A 5,6-Dihydroimidazo[2,1-*b*]thiazole Derivative

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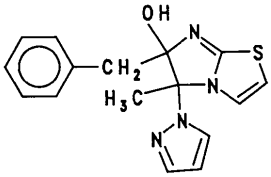
Abstract

In 6-benzyl-5-methyl-5-(1-pyrazolyl)-5,6-dihydroimidazo[2,1-*b*]thiazol-6-ol, $C_{16}H_{16}N_4OS$, the phenyl,

pyrazole and thiazole rings are planar, and the imidazole moiety has an envelope conformation. The molecular packing involves a hydrogen bond and a short N···H—C contact [2.365 (4) Å].

Comment

The interest in imidazo[2,1-*b*]thiazole chemistry arises from the commercial success of tetramisole hydrochloride as a broad-spectrum anthelminthic agent (Thienpont & Janssen, 1966). During a search for a new route to synthesize 5,6-dihydroimidazo-[2,1-*b*]thiazoles (Kascheres, Corrêa Fho & Cunha, 1993), the title compound (I) was obtained. In order to establish unambiguously its stereochemistry, a crystal structure determination was undertaken.



(I)

The phenyl and pyrazole rings, and the thiazole moeity are each planar to within experimental accuracy, $\sigma_{\rm av}$ [defined as $(\sum_i d_i^2/N-3)^{1/2}$] being 0.004, 0.006 and 0.0134 Å, respectively; the imidazole moiety has an envelope conformation with C(3) 0.168 (2) Å out of the plane defined by the other four atoms. The dihedral angles between the phenyl ring plane, (1), the pyrazole ring plane, (2), and the best least-squares plane, (3), through the fused rings are: $(1)\wedge(2) = 106.4$ (1), $(1)\wedge(3) = 29.81$ (9) and $(2)\wedge(3) = 98.5$ (1)°. The internal and external angles of the pyrazole ring follow the set of empirical rules given by Bonati & Bovio (1990). C(7)—C(8) is shorter than C(8)—C(9), as expected in neutral pyrazole rings (Zukerman-Schpector, Castellano, Oliva, Massabni & Pinto, 1984, and references therein).

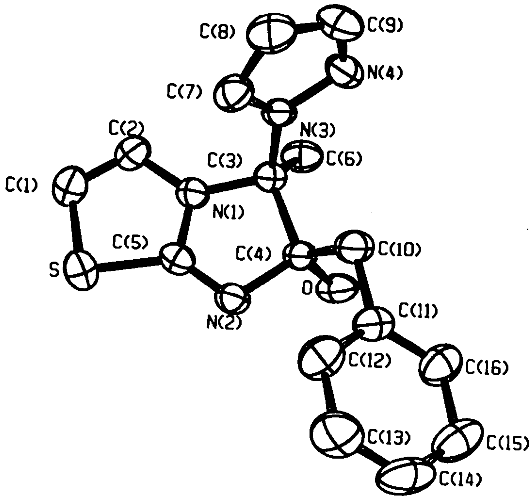


Fig. 1. The molecular structure of $C_{16}H_{16}N_4OS$ showing the atom labelling; 50% probability displacement ellipsoids are shown.

The molecular packing involves (*a*) a hydrogen bond [O···N($2^{\rm i}$) = 2.876 (3), N($2^{\rm i}$)···H(O) = 1.931 (3), O—H(O) = 0.979 (2) Å and O—H(O)···N($2^{\rm i}$) = 161.3 (3)°; symmetry code: (i) $\frac{1}{2}-x, \frac{1}{2}-y, -z$] and (*b*) a relatively strong C—H···N interaction [C(2)···N($4^{\rm ii}$) = 3.433 (4), N($4^{\rm ii}$)···H(C2) = 2.365 (4) Å, C(2)—H(C2)···N(4) = 161.8 (4)°; symmetry code: (ii) $-x, 1-y, -z$]. This type of contact has been found before in compounds containing a thiazole moiety (Bolognesi, Catellani, Destri & Porzio, 1987).

Experimental

Crystal data

$C_{16}H_{16}N_4OS$
$M_r = 312.40$
Monoclinic
$C2/c$
$a = 21.217$ (2) Å
$b = 11.555$ (1) Å
$c = 13.079$ (2) Å
$\beta = 108.61$ (1)°
$V = 3039$ (1) Å^3
$Z = 8$
$D_x = 1.366$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8$–21°
$\mu = 0.210$ mm^{-1}
$T = 292$ K
Irregular
0.30 × 0.25 × 0.15 mm
Colourless
Crystal source: from acetate anhydride

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
$T_{\rm min} = 0.95$, $T_{\rm max} = 1.06$
2566 measured reflections
2428 independent reflections
1872 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.015$
$\theta_{\rm max} = 25°$
$h = -25 \rightarrow 23$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 15$
2 standard reflections
frequency: 30 min
intensity variation: ±0.5%

Refinement

Refinement on F
$R = 0.037$
$wR = 0.041$
$S = 1.58$
1872 reflections
200 parameters
$w = 1/[\sigma^2(|F_o|) + 0.0003|F_o|^2]$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELX76* (Sheldrick, 1976)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
S	0.1850 (1)	0.2400 (1)	0.2680 (1)	3.86 (2)
O	0.1813 (1)	0.3420 (2)	−0.0884 (1)	3.35 (5)
N(1)	0.1180 (1)	0.3541 (2)	0.1025 (2)	2.93 (6)

N(2)	0.2244 (1)	0.3313 (2)	0.1019 (2)	2.81 (6)
N(3)	0.1127 (1)	0.5500 (2)	0.0538 (2)	2.52 (5)
N(4)	0.0931 (1)	0.6367 (2)	−0.0190 (2)	3.35 (6)
C(1)	0.1004 (1)	0.2700 (2)	0.2458 (2)	4.08 (9)
C(2)	0.0732 (1)	0.3313 (2)	0.1573 (2)	3.58 (8)
C(3)	0.1178 (1)	0.4311 (2)	0.0152 (2)	2.53 (6)
C(4)	0.1907 (1)	0.4036 (2)	0.0075 (2)	2.47 (6)
C(5)	0.1808 (1)	0.3112 (2)	0.1487 (2)	2.74 (7)
C(6)	0.0610 (1)	0.4078 (2)	−0.0875 (2)	3.32 (8)
C(7)	0.1349 (1)	0.5899 (2)	0.1567 (2)	3.31 (8)
C(8)	0.1283 (1)	0.7074 (2)	0.1503 (2)	4.23 (9)
C(9)	0.1029 (1)	0.7314 (2)	0.0420 (2)	4.13 (9)
C(10)	0.2315 (1)	0.5136 (2)	0.0082 (2)	3.22 (7)
C(11)	0.3031 (1)	0.4876 (2)	0.0202 (2)	2.99 (7)
C(12)	0.3481 (1)	0.4701 (2)	0.1221 (2)	4.02 (9)
C(13)	0.4138 (1)	0.4436 (3)	0.1354 (3)	5.1 (1)
C(14)	0.4351 (1)	0.4339 (3)	0.0468 (3)	5.0 (1)
C(15)	0.3914 (1)	0.4526 (2)	−0.0546 (3)	4.6 (1)
C(16)	0.3252 (1)	0.4793 (2)	−0.0679 (2)	3.77 (8)

Table 2. *Selected geometric parameters* (Å, °)

S—C(1)	1.759 (3)	S—C(5)	1.741 (2)
O—C(4)	1.400 (3)	N(1)—C(2)	1.386 (4)
N(1)—C(3)	1.447 (3)	N(1)—C(5)	1.368 (3)
N(2)—C(4)	1.473 (3)	N(2)—C(5)	1.283 (3)
N(3)—N(4)	1.353 (3)	N(3)—C(3)	1.480 (3)
N(3)—C(7)	1.357 (3)	N(4)—C(9)	1.331 (3)
C(1)—C(2)	1.322 (4)	C(3)—C(4)	1.613 (4)
C(3)—C(6)	1.515 (3)	C(4)—C(10)	1.536 (3)
C(7)—C(8)	1.365 (4)	C(8)—C(9)	1.373 (4)
C(10)—C(11)	1.507 (4)	C(11)—C(12)	1.384 (4)
C(11)—C(16)	1.379 (4)	C(12)—C(13)	1.383 (4)
C(13)—C(14)	1.376 (5)	C(14)—C(15)	1.371 (5)
C(15)—C(16)	1.394 (4)		
C(1)—S—C(5)	90.1 (1)	C(2)—N(1)—C(3)	134.1 (2)
C(2)—N(1)—C(5)	115.8 (2)	C(3)—N(1)—C(5)	109.2 (2)
C(4)—N(2)—C(5)	105.8 (2)	N(4)—N(3)—C(3)	119.1 (2)
N(4)—N(3)—C(7)	112.2 (2)	C(3)—N(3)—C(7)	127.9 (2)
N(3)—N(4)—C(9)	103.5 (2)	S—C(1)—C(2)	112.8 (2)
N(1)—C(2)—C(1)	112.0 (2)	N(1)—C(3)—N(3)	106.5 (2)
N(1)—C(3)—C(4)	99.6 (2)	N(1)—C(3)—C(6)	113.1 (2)
N(3)—C(3)—C(4)	112.3 (2)	N(3)—C(3)—C(6)	110.1 (2)
C(4)—C(3)—C(6)	114.6 (2)	O—C(4)—N(2)	110.8 (2)
O—C(4)—C(3)	106.9 (2)	O—C(4)—C(10)	110.2 (2)
N(2)—C(4)—C(3)	106.1 (2)	N(2)—C(4)—C(10)	110.0 (2)
C(3)—C(4)—C(10)	112.7 (2)	S—C(5)—N(1)	109.3 (2)
S—C(5)—N(2)	132.4 (2)	N(1)—C(5)—N(2)	118.2 (2)
N(3)—C(7)—C(8)	106.3 (2)	C(7)—C(8)—C(9)	105.2 (3)
N(4)—C(9)—C(8)	112.8 (3)	C(4)—C(10)—C(11)	112.5 (2)
C(10)—C(11)—C(12)	119.5 (2)	C(10)—C(11)—C(16)	121.8 (2)
C(12)—C(11)—C(16)	118.7 (2)	C(11)—C(12)—C(13)	120.7 (3)
C(12)—C(13)—C(14)	120.1 (3)	C(13)—C(14)—C(15)	119.9 (3)
C(14)—C(15)—C(16)	119.9 (3)	C(11)—C(16)—C(15)	120.6 (3)

Data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. H atoms were included as fixed contributors at positions found in difference syntheses and refined with an overall isotropic temperature factor which converged to 0.064 (2) Å^2. Refinement was by full-matrix least-squares techniques. Most of the calculations were performed at the Weizmann Institute of Science, Israel. Programs used were: *SHELXS*86 (Sheldrick, 1985), *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965).

I would like to thank Professor A. J. Kascheres and Dr J. Corrêa Fho. of the Instituto de Química, Universidade Estadual de Campinas, Brazil, for the gift of the sample used in this work, and the Associação de Amigos do Instituto Weizmann em São Paulo, for a scholarship. This work has also received partial support from FAPESP and CNPq.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: LI1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Molecular Co-Crystals of Carboxylic Acids. 18.† 1:1 Adduct of 1,3,5-Trinitrobenzene with 4-Aminobenzoic Acid

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Abstract

The structure of the title compound, $C_6H_3N_3O_6.C_7H_7NO_2$, one of three co-crystalline products formed in the reaction of 2,4,6-trinitrobenzoic acid with 4-aminobenzoic acid, comprises a 1:1 adduct of 1,3,5-trinitrobenzene (TNB) and 4-aminobenzoic acid (PABA), in which both molecules are involved in homomolecular hydrogen bonding. The PABA molecule forms an eight-membered cyclic dimer with another symmetry-generated acid molecule [O···O, 2.65 (1) Å]. The TNB molecule is also involved in possible C—H···O interactions. The layered structure and red col-

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† Part 17: Lynch, Smith, Byriel, Kennard, Whittaker & Hanna (1994).

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