$w = 1/[\sigma^2(F_o^2) + (0{,}0426P)^2 + 0{,}7916P]$ mit $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

Atomformfaktoren aus *International Tables for Crystallography* (1992, Vol. C, Tabelle 4.2.6.8 und 6.1.1.4)

Tabelle 1. *Atomkoordinaten und isotrope äquivalente Verschiebungsparameter* (Å^2)

$U_{äq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{äq}$
I1	0,31235 (6)	0,72114 (8)	0,12654 (6)	0,0510 (2)
I2	0,30187 (7)	0,97944 (6)	−0,02583 (7)	0,0652 (2)
N1	0,2500 (8)	1/4	1/4	0,063 (3)
N3	0,3217 (7)	0,4728 (6)	0,2610 (7)	0,048 (1)
C2	0,2464 (9)	0,3927 (9)	0,1862 (8)	0,054 (2)
C4	0,4678 (8)	0,4074 (9)	0,2572 (8)	0,051 (2)

Tabelle 2. *Geometrische Parameter* (Å, °)

I2—I1	2,7457 (11)	N3—C4	1,482 (9)
N1—C2	1,447 (10)	N3—C2	1,493 (11)
N3—C4^i	1,481 (10)	N3···I1	2,593 (6)
N3···I1—I2	177,6 (2)	C4^i—N3—C4	108,8 (7)
C4^i—N3···I1	113,9 (4)	C4^i—N3—C2	109,1 (6)
C4—N3···I1	109,6 (5)	C4—N3—C2	108,7 (6)
C2—N3···I1	106,6 (4)	N1—C2—N3	111,1 (8)
C2—N1—C2^i	109,2 (7)	N3^{ii}—C4—N3	109,8 (7)

Symmetriebezeichnungen: (i) z, x, y; (ii) y, z, x.

Die Identität des Präparats läßt sich durch einen Vergleich des beobachteten mit dem berechneten (Yvon, Jeitschko & Parthé, 1977) Pulverdiagramm sichern. Die endgültige Zuordnung der Raumgruppe $R3c$ gelingt über die Lauesymmetrie, die Auslöschung und letztlich die Strukturanalyse. Die Zellbesetzung $Z = 2$ läßt sich über Volumeninkremente (Biltz, 1934; Farida, 1994) abschätzen.

Die Lagen der Iodatome sind in der Patterson-Synthese auffindbar. Mit Fourier-Methoden läßt sich das Strukturmodell schrittweise vervollständigen und anisotrop bis $R1 = 0{,}0262$ verfeinern. Dabei wurden die H-Lagen für ideale Methylengruppen bei tetraedrischer Geometrie vor jeden Verfeingsschritt berechnet und mit einem gemeinsamen isotropen Auslenkungsparameter versehen.

Datensammlung: *CAD*-4 *Software* (Enraf-Nonius, 1989). Gitterverfeinerung: *CAD*-4 *Software*. Datenreduktion: *MolEN* (Fair, 1990). Lösung der Struktur: *SHELXS*86 (Sheldrick, 1990). Verfeinerung der Struktur: *SHELXL*93 (Sheldrick, 1993). Zeichenprogramme: *SCHAKAL*92 (Keller, 1993), *ORTEP* (Davenport, Hall & Dreissig, 1990). Programme zur Berechnung der geometrischen Daten: *ORFFE*4 (Busing *et al.*, 1976), *PARST* (Nardelli, 1983).

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Die Listen der Strukturfaktoren, anisotropen Verschiebungsparameter, H-Atom-Koordinaten und vollständigen geometrischen Daten sind bei der IUCr (Aktenzeichen: JZ1025) hinterlegt. Kopien sind erhältlich durch: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Anhydrous Form of Carnidazole, $C_8H_{12}N_4O_3S$

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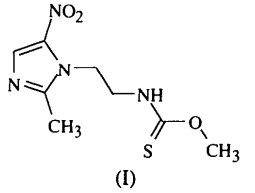
Abstract

The molecular conformations of the anhydrous and hydrated forms of carnidazole {*O*-methyl [2-(2-methyl-5-nitro-1*H*-imidazol-1-yl)ethyl]carbamothioate} differ essentially in the relative orientations of the nitroimidazole and thiocarbamate moieties. In the anhydrous form, the

molecules are connected by a network of hydrogen bonds and show stacking interactions between the nitroimidazole moieties.

Comment

Nitroimidazole and its derivatives are well known and are used as radiosensitizers and antibiotic drugs. Investigation of the possible mechanism of their radiosensitizing properties led us to study the radiation damage in carnidazole {*O*-methyl [2-(2-methyl-5-nitro-1*H*-imidazol-1-yl)ethyl]thiocarbamate, (I)} by an electron paramagnetic resonance (EPR) single-crystal study which has been reported elsewhere (Berclaz, Bernardinelli, Geoffroy & Rajalakshmi, 1992). Both the hydrated and anhydrous forms of carnidazole can be obtained simultaneously from H_2O/EtOH solution.



(I)

The structure of the hydrated form reported previously (Blaton, Peeters & De Ranter, 1979) differs essentially from the anhydrous form in the relative orientations of the nitroimidazole and thiocarbamate moieties [dihedral angles around the C4—C5 and C5—N4 bonds of the hydrated form are 60.6 (4) and 87.7 (4)°, respectively]. In the anhydrous form, the molecule adopts a step-like conformation where all the non-H atoms are distributed in two parallel (3.3°) mean planes, linked by the C4—C5 bond, which are perpendicular to the *c* axis (Fig. 2). As observed for both the hydrated form (Blaton, Peeters & De Ranter, 1979) and another nitroimidazole derivative (Olszak, Peeters, Blaton & De Ranter, 1994), the nitro group makes a slight dihedral angle [4.4 (3)°] with the imidazole ring (which is planar with a maximum deviation of 0.08 Å). The molecules are connected by weak hydrogen bonds leading to the formation of a seven-membered ring between the imidazole and the thiocarbamate moieties linked through a glide plane symmetry relation [N4···N2^{i} 2.961 (7) Å, N4—H04···N2^{i} 161°; O3···C2^{i} 3.249 (8) Å, O3···H2—C2^{i} 127 (4)°; symmetry code: (i) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$]. Moreover, the molecules are associated into pairs through stacking interactions between the nitroimidazole moieties located around a centre of inversion [the mean interplane distance is 3.37 (2) Å].

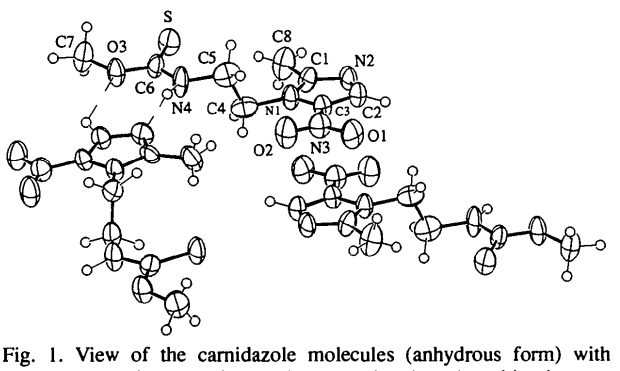


Fig. 1. View of the carnidazole molecules (anhydrous form) with atomic labelling, showing the hydrogen bonds and stacking interactions between the nitroimidazole moieties. Displacement ellipsoids are drawn at the 50% probability level.

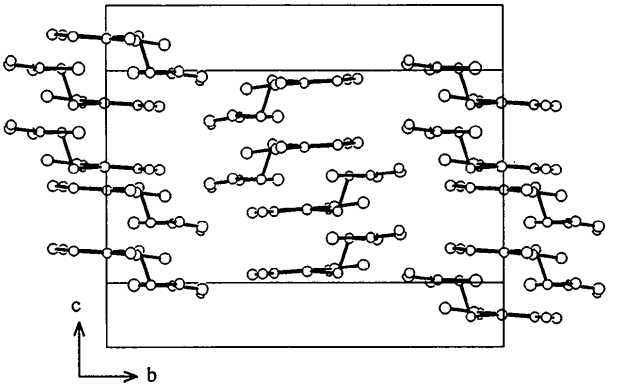


Fig. 2. Projection of the structure onto the *bc* plane showing the step-like conformation of the molecules and the perpendicular orientations of both mean planes with the *c* axis.

Experimental

Crystal data

$C_8H_{12}N_4O_3S$
M_r = 244.3
Monoclinic
$P2_1/n$
a = 7.304 (1) Å
b = 15.437 (3) Å
c = 10.650 (4) Å
β = 109.47 (1)°
V = 1132.1 (5) Å^3
Z = 4
D_x = 1.433 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 8.5–14.5°
μ = 0.285 mm^{-1}
T = 293 K
Prism
0.20 × 0.17 × 0.07 mm
Yellow

Data collection

Philips PW1100 diffractometer
$\omega/2\theta$ scans
Absorption correction: analytical integration
T_{min} = 0.954, T_{max} = 0.979
1592 measured reflections
1395 independent reflections
838 observed reflections [$F > 4.0\sigma(F)$]
R_{int} = 0.041
θ_{max} = 21.99°
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 11$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
R = 0.067
wR = 0.032
$(\Delta/\sigma)_{max}$ = 0.084
$\Delta\rho_{max}$ = 0.512 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

$S = 2.00$
836 reflections
181 parameters
Only coordinates of H atoms refined
Weighting scheme based on measured e.s.d.'s
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

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

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

	x	y	z	U_{eq}
S	0.1966 (3)	0.0689 (1)	0.0378 (2)	0.061 (1)
O1	1.3034 (7)	0.0599 (3)	0.4097 (5)	0.070 (3)
O2	1.0605 (7)	0.1469 (3)	0.3333 (5)	0.074 (3)
O3	0.2025 (7)	0.2401 (2)	0.0240 (5)	0.057 (3)
N1	0.8059 (8)	0.0044 (3)	0.2996 (6)	0.046 (4)
N2	0.9002 (9)	−0.1333 (3)	0.3333 (6)	0.051 (4)
N3	1.1283 (9)	0.0741 (4)	0.3630 (6)	0.056 (4)
N4	0.4889 (8)	0.1828 (3)	0.1085 (7)	0.062 (4)
C1	0.750 (1)	−0.0794 (5)	0.2944 (7)	0.047 (4)
C2	1.060 (1)	−0.0827 (4)	0.3622 (7)	0.047 (5)
C3	1.004 (1)	0.0018 (5)	0.3417 (7)	0.041 (4)
C4	0.667 (1)	0.0822 (5)	0.2745 (8)	0.059 (6)
C5	0.640 (1)	0.1112 (5)	0.1395 (9)	0.064 (5)
C6	0.300 (1)	0.1652 (4)	0.0588 (7)	0.049 (4)
C7	−0.004 (1)	0.2372 (6)	−0.0363 (9)	0.069 (7)
C8	0.545 (1)	−0.1082 (6)	0.250 (1)	0.070 (6)

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

S—C6	1.649 (7)	N2—C1	1.329 (9)
O1—N3	1.228 (7)	N2—C2	1.35 (1)
O2—N3	1.226 (8)	N3—C3	1.41 (1)
O3—C6	1.343 (7)	N4—C5	1.520 (9)
O3—C7	1.43 (1)	N4—C6	1.332 (9)
N1—C1	1.352 (9)	C1—C8	1.48 (1)
N1—C3	1.364 (9)	C2—C3	1.36 (1)
N1—C4	1.54 (1)	C4—C5	1.46 (1)
C6—O3—C7	118.6 (6)	N2—C2—C3	109.1 (6)
C1—N1—C3	105.0 (6)	N1—C3—C2	108.0 (6)
C1—N1—C4	124.4 (6)	N1—C4—C5	105.9 (7)
C3—N1—C4	130.3 (6)	N4—C5—C4	106.6 (8)
C1—N2—C2	105.7 (5)	S—C6—O3	124.3 (5)
C5—N4—C6	121.4 (5)	S—C6—N4	127.2 (5)
N1—C1—N2	112.3 (6)	O3—C6—N4	108.5 (5)
C7—O3—C6—S	0 (1)	O2—N3—C3—N1	−6 (1)
C7—O3—C6—N4	177.9 (7)	C5—N4—C6—S	7 (1)
C1—N1—C4—C5	−101.8 (8)	C5—N4—C6—O3	−171.5 (7)
C3—N1—C4—C5	84.7 (9)	N1—C4—C5—N4	175.0 (5)
O1—N3—C3—C2	−3 (1)		

Data collection: Philips PW1100 software. Cell refinement: *Xtal LATCON* (Hall, Flack & Stewart, 1992). Data reduction: *Xtal REFCAL STARTX SORTRF*; *LSABS* (Blanc, Schwarzenbach & Flack, 1991). Program(s) used to solve structure: *MULTAN*87 (Main *et al.*, 1987). Program(s) used to refine structure: *Xtal CRYLSQ*. Molecular graphics: *Xtal ORTEP*. Software used to prepare material for publication: *Xtal CIFIO*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and torsion angles have been deposited with the IUCr (Reference: PA1156). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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6-Methylamino-4-methylthio-5-nitro-2-phenylpyrimidine

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Abstract

The title compound, $C_{12}H_{12}N_4O_2S$, is essentially planar, the dihedral angle between the pyrimidine and phenyl rings being 11.4 (3)°. There are no unusual intra- or intermolecular distances or angles. The molecules are packed with normal van der Waals distances.

Comment

Pyrimidine derivatives are constituents of naturally occurring molecules or, in synthetic form, useful as drugs or agricultural chemicals (Brown, 1984). Nucleosides and nucleotides and some alkaloids bear a pyrimidine ring as a structural element and many functionalized derivatives obtained by synthetic procedures show antibiotic, antimicrobial (*e.g.* pyrimidine sulfonamides),