2837 reflections
300 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.2002P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.319$ e Å^{-3}
$\Delta\rho_{min} = -0.224$ e Å^{-3}
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.004 (17)

Table 1. *Selected geometric parameters (Å, °)*

O1—C5	1.394 (4)	C4—C5	1.525 (3)
O1—C2	1.396 (3)	S3—O32	1.4264 (19)
C2—N3	1.497 (3)	S3—O31	1.427 (2)
N3—C4	1.491 (3)	S3—C31	1.762 (3)
N3—S3	1.6441 (19)		
C5—O1—C2	110.7 (2)	O32—S3—O31	120.33 (12)
O1—C2—N3	106.0 (2)	O32—S3—N3	106.30 (10)
C4—N3—C2	107.67 (18)	O31—S3—N3	106.04 (12)
C4—N3—S3	116.96 (16)	O32—S3—C31	107.92 (12)
C2—N3—S3	116.36 (16)	O31—S3—C31	107.91 (12)
N3—C4—C5	100.2 (2)	N3—S3—C31	107.77 (10)
O1—C5—C4	107.1 (2)		
C5—O1—C2—N3	8.2 (3)	C25—C21—C22—C23	−23.5 (3)
O1—C2—N3—C4	11.2 (3)	C21—C22—C23—C24	−1.1 (3)
C2—N3—C4—C5	−23.7 (3)	C2—C21—C25—C24	165.9 (2)
C2—O1—C5—C4	−24.1 (4)	C22—C21—C25—C24	38.3 (2)
N3—C4—C5—O1	28.9 (3)	C23—C24—C25—C21	−39.9 (3)

The structure was solved by extracting the position of the S atom from a sharpened Patterson list and extending the structure with a tangent expansion using *SHELXS*96 (Sheldrick, 1990), and then refined with *SHELXL*96 (Sheldrick, 1996) by full-matrix least-squares methods. All H atoms were located by difference Fourier syntheses and refined with fixed individual displacement parameters [U(H) = 1.5U_{eq}(C_{methyl}) or U(H) = 1.2U_{eq}(C)], using a riding model with C—H(tertiary) = 0.98, C—H(secondary) = 0.97, C—H(methyl) = 0.96 or C—H(aromatic) = 0.93 Å.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SDP*. Data reduction: *SDP*. Molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1271). Services for accessing these data are described at the back of the journal.

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2,3,4,5-Tetrahydrocyclohepta[*b*]pyrido-[2,1-*a*]benzimidazole-7-carbonitrile

GALAL E. H. ELGEMEIE,[a] NAHED M. FATHY,[b] HENNING HOPF,[c] INA DIX[c] AND PETER G. JONES[d]*

[a]*Chemistry Department, Faculty of Science, Helwan University, Helwan, Cairo, Egypt,* [b]*Photochemistry Laboratory, National Research Centre, Cairo, Egypt,* [c]*Institut für Organische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and* [d]*Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany. E-mail: jones@xray36.anchem.nat.tu-bs.de*

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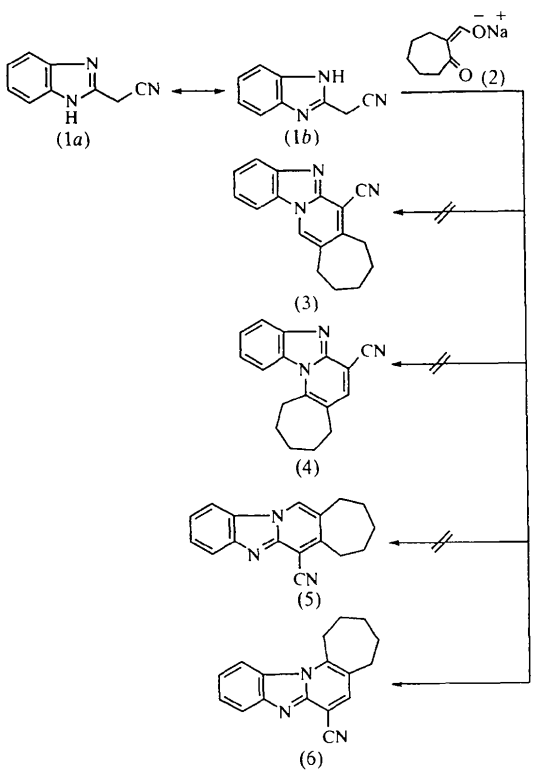
Abstract

The title compound, $C_{17}H_{15}N_3$, is a tetracyclic system in which the atoms of the three unsaturated rings are coplanar to within 0.026 Å. The molecules are stacked in pairs about inversion centres, with the shortest C···C separation being 3.386 (4) Å.

Comment

The imidazole and imidazo[1,2-*a*]pyridine ring systems are components of heterocycles that have shown anti-tumor activity (Elgemeie *et al.*, 1996). Our interest in the development of novel non-classical antimetabolites directed us towards the synthesis of new tetracyclic antimetabolites involving the imidazo[1,2-*a*]pyridine system (Elgemeie & Hussain, 1994; Elgemeie *et al.*, 1994). A single-step synthesis of the title derivative (6) was anticipated *via* the cyclocondensation of 2-cyanobenzimidazole and the sodium salt of 2-(hydroxymethylene)-1-cycloheptanone. However, this reaction could give three other possible regioisomers [structures (3)–(5)]. The direction of ring closure in such cyclocondensations is difficult to predict, and spectroscopic data cannot differentiate between these structures. In order to establish unambiguously the structure of the product, the crystal structure was determined.



The X-ray analysis confirms the exclusive presence of form (6) in the solid state (Fig. 1). This implies that 2-cyanobenzimidazole is present in solution in form (1*b*) and its active methylene carbon will initially attack the unhindered formyl group of (2) to give (6), rather than attacking the hindered and electronically disfavoured ketonic group leading to (5). The aromatic ring system (N1, N2, C6–C16) is planar (r.m.s. deviation 0.026 Å). The saturated section of the seven-membered ring is displaced to one side of the plane of the aromatic ring system, with deviations of 1.060 (4), 1.173 (4) and 1.228 (4) Å for atoms C2, C3 and C4, respectively. A search of the Cambridge Structural Database (Allen & Kennard, 1993) revealed no other structures with the same tetracyclic ring system. Bond lengths and angles may be regarded as normal and are similar to those in analogous regions of 1,2,3,4-tetrahydrobenzo[*d*]imidazo-[2,1-*b*]quinazoline (Elgemeie *et al.*, 1998). As expected, the most marked deviations from ideal bond angles are associated with the five-membered ring [*e.g.* C11—C14—N1 134.1 (3) and C15—N2—C16 104.3 (2)°]. The molecules are arranged in pairs with parallel ring systems about inversion centres. The shortest non-bonded C···C distance is one of 3.386 (4) Å involving C13···C8($1-x$, $1-y$, $1-z$).

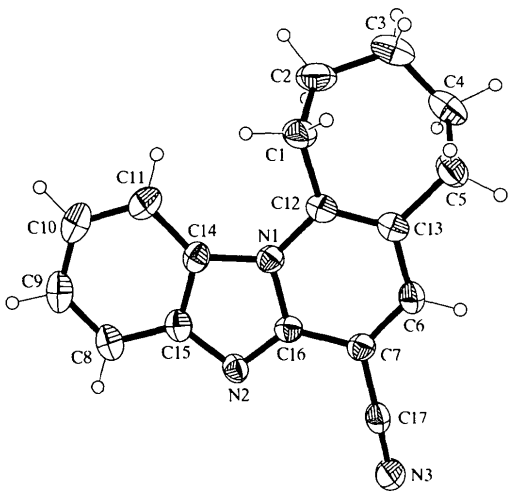


Fig. 1. The molecule of the title compound in the crystal. Ellipsoids represent 50% probability levels and H-atom radii are arbitrary.

Experimental

A solution of 2-cyanobenzimidazole [(1); 1.57 g, 0.01 mol], the sodium salt of 2-(hydroxymethylene)-1-cycloheptanone [(2); 1.62 g, 0.01 mol], and piperidine acetate (1 ml) in water (50 ml) and ethanol (50 ml) was refluxed for 15 min. Acetic acid (1.5 ml) was added to the hot solution. The precipitate was collected by filtration and crystallized from benzene in 65% yield (m.p. 481 K).

Crystal data

$C_{17}H_{15}N_3$
M_r = 261.32
Triclinic
$P\overline{1}$
a = 8.105 (4) Å
b = 9.292 (4) Å
c = 10.283 (4) Å
α = 109.67 (3)°
β = 102.76 (3)°
γ = 107.14 (2)°
V = 651.3 (5) Å^3
Z = 2
D_x = 1.333 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 40 reflections
θ = 10.0–11.5°
μ = 0.081 mm^{-1}
T = 143 (2) K
Pyramidal
0.4 × 0.3 × 0.3 mm
Yellow

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
2422 measured reflections
2284 independent reflections
1530 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

θ_{max} = 24.99°
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 10$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.121
S = 1.094
2284 reflections
182 parameters
H atoms: see below

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL93*
Extinction coefficient: 0.021 (2)