Orthorhombic
Pbca
$a = 18.4645\,(9)$ Å
$b = 8.0647\,(6)$ Å
$c = 21.9392\,(9)$ Å
$V = 3267.0\,(3)$ Å^3
$Z = 8$
$D_x = 1.538$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 12–13°$
$\mu = 2.525$ mm^{-1}
$T = 300\,(2)$ K
Needle
0.36 × 0.18 × 0.15 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega–2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.435$, $T_{\max} = 0.685$
4569 measured reflections
3199 independent reflections

1645 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 25.97°$
$h = 0 \rightarrow 22$
$k = -9 \rightarrow 7$
$l = -27 \rightarrow 0$
3 standard reflections frequency: 60 min intensity decay: <1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.121$
$S = 0.925$
3199 reflections
266 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.753$ e Å^{-3}
$\Delta\rho_{\min} = -0.823$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0029 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *CAD*-4 *VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX/PC*. Data reduction: *NRCVAX* (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

We thank the Malaysian National Science Council for an R&D grant (09-02-03-0004), the National Nature Science Foundation of China and the Nature Science Foundation of Jiangsu Province, People's Republic of China, for supporting this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1205). Services for accessing these data are described at the back of the journal.

References

Bencini, A., Gatteschi, D., Zanchini, C., Haasnoot, J. G., Prins, R. & Reedijk, J. (1985). *Inorg. Chem.* **24**, 2812–2815.
Bencini, A., Gatteschi, D., Zanchini, C., Haasnoot, J. G., Prins, R. & Reedijk, J. (1987). *J. Am. Chem. Soc.* **109**, 2926–2931.
Enraf–Nonius (1988). *CAD*-4 *VAX/PC Operator's Manual*. Enraf–Nonius, Delft, The Netherlands.
Gabe, E. J., Le Page, Y., Charland, J. P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Grimmel, H. W., Guenther, A. & Morgan, J. F. (1946). *J. Am Chem. Soc.* **68**, 539–542.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Klingsberg, E. (1958). *J. Org. Chem.* **23**, 1086–1087.
Koningsbruggen, P. J. van, Gatteschi, D., de Graaff, R. A. G., Haasnoot, J. G., Reedijk, J. & Zanchini, C. (1995). *Inorg. Chem.* **34**, 5175–5182.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990*a*). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1990*b*). *SHELXTL/PC User Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1998). C**54**, 852–854

4-Benzoyl-4-(2-cyanoethyl)heptanedinitrile at 143 K

Michael Bolte and Maria Scholtyssik

Institut für Organische Chemie der Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany. E-mail: bolte@chemie.uni-frankfurt.de

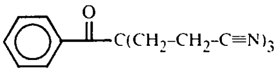
(*Received 22 May 1997; accepted 18 November 1997*)

Abstract

The title compound, $C_{17}H_{17}N_3O$, crystallizes with two molecules in the asymmetric unit which differ significantly only in the conformation of one torsion angle. All bonds between the methylenic C atoms display antiperiplanar conformations, except one, which is synclinal. The carbonyl bond is twisted by 32° out of the plane of the phenyl ring and is synperiplanar to one of the methylenic C atoms.

Comment

The title compound, (I), crystallizes with two molecules (Figs. 1*a* and 1*b*) in the asymmetric unit.



(I)

Bond lengths and angles are in the usual ranges (*International Tables for Crystallography*, Vol. C); the mean values of the C_{sp^3}—C_{sp} and the C_{sp}—N bonds, for example, are 1.462 (4) and 1.141 (4) Å, respectively.

A least-squares fit of all non-H atoms, excluding C23 and N23 (r.m.s. deviation 0.103 Å), shows

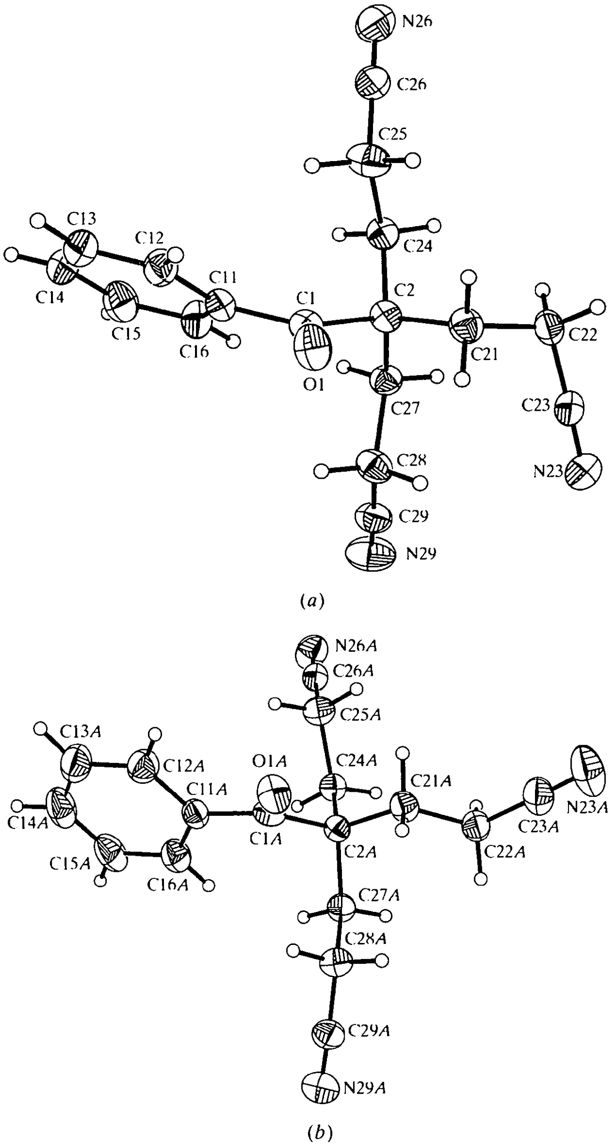


Fig. 1. Perspective views of the two independent molecules of (I) with the atom numbering; displacement ellipsoids are at the 50% probability level.

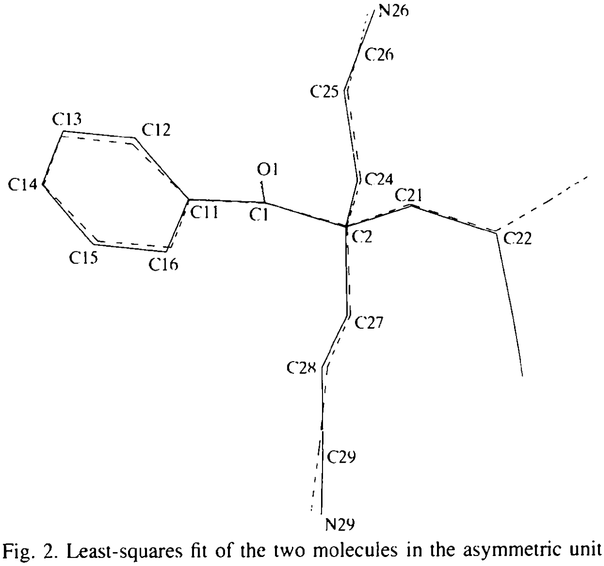


Fig. 2. Least-squares fit of the two molecules in the asymmetric unit; fitted atoms are labelled.

the similarities of the two molecules (Fig. 2). Only the torsion angles C2—C21—C22—C23 and C2*A*—C21*A*—C22*A*—C23*A* are significantly different in the two molecules, having values of −85.1 (3) and 175.7 (2)°, respectively. While five of the six bonds between the methylenic C atoms are in antiperiplanar conformations, the conformation about C21—C22 is synclinal. The carbonyl group in each molecule is twisted out of the plane of the phenyl ring [by 31.9 (3) (O1—C1—C11—C12) and 26.7 (3)° (O1*A*—C1*A*—C11*A*—C12*A*)] and it is synperiplanar to one of the methylenic C atoms [O1—C1—C2—C21 −7.2 (3) and O1*A*—C1*A*—C2*A*—C21*A* −7.8 (3)°].

Experimental

The title compound was obtained by dissolving 0.5 ml of KOH (30%) in a solution of 0.1 mol of acetophenone in 40 ml of *tert*-butanol and then slowly adding 0.3 mol of acrylnitrile in 30 ml of *tert*-butanol. After stirring for 1 h at 273 K and adding 20 ml of petroleum ether to the solution, the reaction product precipitated and was dried *in vacuo*. Single crystals were obtained by slow evaporation of an ethanol–toluol solution.

Crystal data

$C_{17}H_{17}N_3O$
$M_r = 279.34$
Triclinic
$P\bar{1}$
$a = 8.5287\ (1)$ Å
$b = 9.8554\ (1)$ Å
$c = 18.6719\ (1)$ Å
$\alpha = 104.195\ (1)^\circ$
$\beta = 95.384\ (1)^\circ$
$\gamma = 91.638\ (1)^\circ$
$V = 1512.64\ (2)$ Å^3
$Z = 4$
$D_x = 1.227$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6961 reflections
$\theta = 1$–25°
$\mu = 0.079$ mm^{-1}
$T = 143$ K
Plate
0.35 × 0.20 × 0.05 mm
Colourless

Data collection

Siemens CCD three-circle diffractometer
ω scans
Absorption correction: none
21 154 measured reflections
6096 independent reflections
3170 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.122$
$\theta_{\rm max} = 26.49^\circ$
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -23 \rightarrow 23$
149 standard reflections
frequency: 960 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.149$
$S = 0.919$
6096 reflections
380 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.317$ e Å^{-3}
$\Delta\rho_{\min} = -0.295$ e Å^{-3}
Extinction correction: *SHELXL*96 (Sheldrick, 1996)
Extinction coefficient: 0.018 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C1—O1	1.222 (3)	C1*A*—O1*A*	1.225 (2)
C23—N23	1.140 (3)	C23*A*—N23*A*	1.136 (3)
C26—N26	1.143 (3)	C26*A*—N26*A*	1.145 (3)
C29—N29	1.146 (3)	C29*A*—N29*A*	1.138 (3)
C2—C1—C11	122.9 (2)	C2*A*—C1*A*—C11*A*	123.6 (2)
C2—C1—O1	119.1 (2)	C2*A*—C1*A*—O1*A*	119.6 (2)

The data collection nominally covered over a sphere of reciprocal space, by a combination of seven sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω. The crystal-to-detector distance was 5.95 cm. Coverage of the unique set is over 99% complete to at least 26° in θ. Crystal decay was monitored by repeating the initial frames at the end of the data collection and analysing the duplicate reflections. All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [$U(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C})$] using a riding model with C—H(aromatic) = 0.95 and C—H(secondary) = 0.99 Å.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART*. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *SHELXS*96 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996). Molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1313). Services for accessing these data are described at the back of the journal.

References

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SHELXL96. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1995). *SMART* and *SAINT. Area Detector Control and Integration Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1998). C**54**, 854–856

1-Benzyl-2-(2,6-dichloroanilinomethyl)-1*H*-benzimidazole

Engin Kendi,[a] Süheyla Özbey,[a] Meral Tunçbilek[b] and Hakan Göker[b]

[a]*Department of Engineering Physics, Hacettepe University, Beytepe 06532, Ankara, Turkey, and* [b]*Department of Pharmaceutical Chemistry, Ankara University, Tandoğan 06100, Ankara, Turkey. E-mail: kendi@eti.cc.hun.edu.tr*

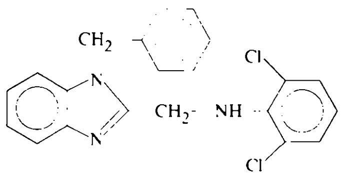
(*Received 15 July 1997; accepted 15 December 1997*)

Abstract

The benzimidazole ring of the title compound, $C_{21}H_{17}Cl_2N_3$, is planar and makes an angle of 98.38 (6)° with the phenyl ring of the benzyl substituent at N1. The molecules are joined together by C—H···N intermolecular hydrogen bonds.

Comment

Benzimidazole is a heterocyclic ring system present in naturally occurring cyanocobalamine. Substituted benzimidazoles, such as omeprazole (Brandstrom *et al.*, 1985), astemizole (Awouters *et al.*, 1983) and emedastine difumarate (Sakai *et al.*, 1989; Saito *et al.*, 1993), have attracted considerable attention as therapeutics for the treatment of some diseases, such as peptic ulcers and severe hypersecretion in Zollinger–Ellison syndrome. To examine their chemical properties and antimicrobial activity, a series of 1-(*p*-substituted-benzyl)benzimidazole compounds were synthesized, one of which is the title compound, (I). Its structure was assigned on the basis of NMR, mass spectrometric and elemental analysis. The X-ray structure was investigated in order to determine the conformation and packing of (I).



(I)

The bond lengths and angles are as expected. Bond lengths N1—C2 [1.364 (3) Å] and C2—N3 [1.308 (3) Å] are in good agreement with the values reported for benzimidazole [1.361 (7) and 1.315 (8) Å; Escande & Galigne, 1974] and 2-(*o*-methoxyphenoxy)-1-methylbenzimidazole [1.360 (2) and 1.295 (2) Å; Vasudevan *et al.*, 1994]. The N1—C10 [1.458 (3) Å] and N2—