Data collection

Nicolet *R*3*M* diffractometer
ω scans
Absorption correction:
ψ scans
$T_{\min} = 0.590$, $T_{\max} = 0.907$
1520 measured reflections
1438 independent reflections
1214 observed reflections
$[I > 2\sigma(I)]$

$R_{\rm int} = 0.0664$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 8$
$l = -12 \rightarrow 12$
3 standard reflections
monitored every 97 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0400$
$wR(F^2) = 0.0829$
$S = 1.068$
1438 reflections
184 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.002$
$\Delta\rho_{\max} = 0.615$ e Å^{-3}
$\Delta\rho_{\min} = -0.504$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
Br1	0.89489 (7)	0.15280 (14)	0.55164 (6)	0.0430 (3)
S1	0.5895 (2)	−0.0750 (2)	0.8244 (2)	0.0229 (4)
O1	0.7964 (4)	0.2711 (7)	1.0333 (4)	0.0222 (11)
O2	0.9771 (4)	0.1211 (7)	1.0025 (4)	0.0251 (12)
O3	0.6447 (4)	0.1306 (7)	0.8099 (4)	0.0198 (10)
O4	0.4485 (4)	−0.0553 (8)	0.7762 (5)	0.0307 (13)
O5	0.6600 (5)	−0.2061 (8)	0.7659 (5)	0.0343 (13)
C1	0.7813 (8)	0.5164 (13)	0.5454 (7)	0.040 (2)
C2	0.7624 (7)	0.3239 (12)	0.6000 (6)	0.028 (2)
C3	0.6267 (6)	0.2451 (12)	0.5387 (6)	0.034 (2)
C4	0.7995 (7)	0.3369 (11)	0.7479 (6)	0.024 (2)
C5	0.7873 (5)	0.1597 (13)	0.8217 (5)	0.0212 (13)
C6	0.8659 (6)	0.1800 (11)	0.9635 (5)	0.0189 (14)
C7	0.8637 (7)	0.2963 (12)	1.1696 (6)	0.027 (2)
C8	0.6309 (6)	−0.1103 (9)	0.9921 (6)	0.021 (2)
C9	0.7515 (6)	−0.1997 (10)	1.0527 (7)	0.024 (2)
C10	0.7843 (7)	−0.2118 (12)	1.1874 (7)	0.037 (2)
C11	0.7013 (8)	−0.1408 (11)	1.2567 (7)	0.036 (2)
C12	0.5830 (8)	−0.0564 (12)	1.1960 (7)	0.035 (2)
C13	0.5467 (7)	−0.0415 (10)	1.0631 (6)	0.026 (2)

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

Br1—C2	1.999 (8)	C2—C3	1.497 (9)
S1—O5	1.424 (5)	C2—C4	1.526 (9)
S1—O4	1.424 (5)	C4—C5	1.509 (11)
S1—O3	1.593 (5)	C5—C6	1.527 (7)
S1—C8	1.745 (6)	C8—C13	1.383 (9)
O1—C6	1.329 (8)	C8—C9	1.402 (9)
O1—C7	1.452 (7)	C9—C10	1.390 (9)
O2—C6	1.195 (7)	C10—C11	1.365 (11)
O3—C5	1.464 (6)	C11—C12	1.368 (11)
C1—C2	1.521 (12)	C12—C13	1.372 (10)
O5—S1—O4	120.0 (3)	O3—C5—C4	106.9 (5)
O5—S1—O3	108.8 (3)	O3—C5—C6	111.6 (4)
O4—S1—O3	103.6 (3)	C4—C5—C6	110.0 (6)
O5—S1—C8	109.6 (3)	O2—C6—O1	126.1 (5)
O4—S1—C8	110.0 (3)	O2—C6—C5	122.1 (6)
O3—S1—C8	103.4 (3)	O1—C6—C5	111.8 (5)
C6—O1—C7	115.4 (5)	C13—C8—C9	121.4 (6)
C5—O3—S1	120.0 (5)	C13—C8—S1	119.1 (5)
C3—C2—C1	111.7 (6)	C9—C8—S1	119.5 (5)
C3—C2—C4	115.6 (6)	C10—C9—C8	117.1 (7)
C1—C2—C4	108.5 (6)	C11—C10—C9	121.0 (7)
C3—C2—Br1	106.8 (5)	C10—C11—C12	121.2 (7)
C1—C2—Br1	106.7 (5)	C11—C12—C13	119.7 (8)
C4—C2—Br1	107.2 (5)	C12—C13—C8	119.5 (7)
C5—C4—C2	117.1 (6)		

Data collection: *SHELXTL-Plus* (Sheldrick, 1991). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

We thank Professor W. T. Robinson, University of Canterbury, for data collection. This work was supported by a University of Otago Division of Sciences Research Grant.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AB1251). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Shaw, J. P. & Tan, E. W. (1995). In preparation.

Shaw, J. P., Tan, E. W. & Blackman, A. G. (1995). *Acta Cryst.* C**51**, 134–135.

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. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Acta Cryst. (1995). C**51**, 1412–1414

Phenyl Carbamidonitrile

Qing-chuan Yang,* Dan-mei Huang, Hui-ying Chen and You-qi Tang

Department of Chemistry, Peking University, Beijing 100871, People's Republic of China

(*Received* 15 *August* 1994; *accepted* 7 *December* 1994)

Abstract

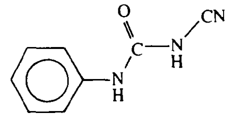
The molecule of 3-phenyl-1-ureidonitrile, $C_8H_7N_3O$, has an extended conformation with a nearly planar carbamidonitrile group (to within 0.04 Å). The angle between the phenyl ring plane and the plane of the carbamidonitrile group is 27.6 (4)°. The carbamido N(7) and N(10) atoms are sp^2 hybridized. Except for the nitrile C(11)≡N(12) triple bond, the remaining C—N

Acta Crystallographica Section C
ISSN 0108-2701 ©1995

bonds in the molecule are partial double bonds. There is a strongly delocalized π-electronic system throughout the whole molecule.

Comment

The potential applications of the non-linear optical and electroactive properties of organic and organic polymeric substances having strongly delocalized π-electronic systems means that the study of such compounds is becoming the subject of intense interest (Williams, 1984). The synthesis of a novel conjugated polynitrile with noticeable electric bistability has been reported (Chen, He, Geng, Xue & Gao, 1994). The crystal structure analysis of the title compound, (I), provides the related monomer structural information for the study of the solid-state behaviour of a polyphenylcarbamidonitrile on the molecular level.



(I)

The carbamidonitrile group is planar to within 0.04 Å. The angle between the planes of the phenyl and carbamidonitrile moieties is 27.6 (4)°. The whole molecule has an extended conformation with an H(4)···N(12) distance of 9.42 (4) Å, almost along the *a* axis. Atoms N(7) and N(10) of the carbamido group are approximately sp^2 hybridized. Except for the nitrile triple bond [C(11)≡N(12)] of 1.139 (3) Å, the remaining C—N bond lengths range from 1.335 (3) to 1.425 (3) Å, which are in the range between a pure single C_{sp^2}—N_{sp^3} bond length of 1.44 Å and a double C=N bond length of 1.27 Å (Gilli, Bertolasi, Bellucci, Fabrizio & Ferretti, 1986). A view of the molecular structure is shown in Fig. 1.

The geometry described above indicates that there is a strongly delocalized π-electronic system throughout the whole molecule. Intermolecular hydrogen bonds connect the carbonyl O atoms with the carbamido N(7) and N(10) atoms of the nearest neighbouring molecules, forming infinite molecular chains almost parallel to the *b* axis. The angles between the O(9)···O(9^{i}) direction and the *a*, *b* and *c* axes are 88.8, 178.8 and 90.0°, respectively [O(9)···N(7^{i}) 2.895 (2), N(7^{i})—H(7^{i}) 0.86 (3) Å, O(9)···H(7^{i})—N(7^{i}) 149 (2)°; O(9)···N(10^{i}) 2.819 (2), N(10^{i})—H(10^{i}) 0.85 (2) Å, O(9)···H(10^{i})—N(10^{i}) 149 (2)°; symmetry code: (i) $\frac{3}{2}-x$, $y-\frac{1}{2}$, z]. The molecular chains pack in a parallel fashion with van der Waals interactions between them.

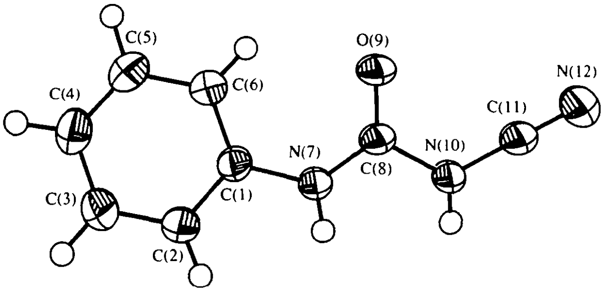


Fig. 1. The molecular structure and atom-numbering system of the title compound. Displacement ellipsoids are plotted at the 50% probability level.

The arrangement of nitrile groups in the unit cell warrants attention since it is responsible for possible polymerization. As can be seen in Fig. 2, the C(11) atom, which is potentially involved in the polymerization, and the nearest N atom, N(12^{ii}) of a neighbouring molecular chain, are 3.288 (3) Å apart, while atom N(12) is 5.449 (3) Å from the second nearest nitrile C(11^{iii}) atom [symmetry codes: (ii) $1-x$, $1-y$, $1-z$; (iii) $x-\frac{1}{2}$, $\frac{1}{2}-y$, $1-z$]. Investigation of this possible polymerization is underway.

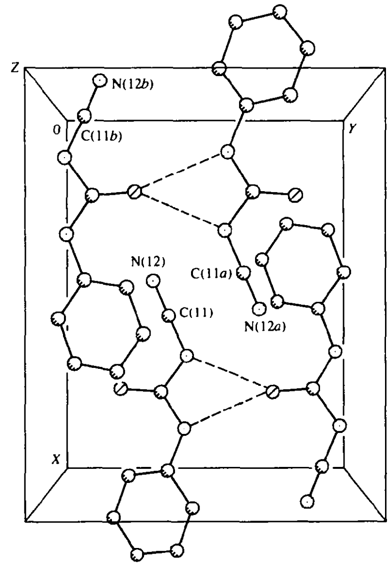


Fig. 2. The arrangement of molecules in the title compound showing the shortest intermolecular contact distances between the nitrile groups and also hydrogen bonds.

Experimental

The title compound was prepared from phenyl isocyanate and cyanamide according to the method of Rabjohn (1963). Colourless needle crystals were obtained by vapour diffusion with tetrahydrofuran as solvent and pentane as precipitant.

Crystal data

$C_8H_7N_3O$
$M_r = 161.2$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Orthorhombic
Pbca
$a = 11.528\,(3)$ Å
$b = 9.166\,(1)$ Å
$c = 14.655\,(2)$ Å
$V = 1548.6\,(5)$ Å^3
$Z = 8$
$D_x = 1.383$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 7.06$–$10.88°$
$\mu = 0.091$ mm^{-1}
$T = 296$ K
Needle-like prism
0.75 × 0.25 × 0.15 mm
Colourless

Data collection

Siemens *R3m/V* diffractometer
ω scans
Absorption correction: none
3006 measured reflections
1529 independent reflections
1038 observed reflections [$F > 4\sigma(F)$]

$R_{\rm int} = 0.024$
$\theta_{\rm max} = 26.0°$
$h = -14 \rightarrow 0$
$k = -11 \rightarrow 0$
$l = -18 \rightarrow 18$
2 standard reflections monitored every 100 reflections
intensity decay: 2.5%

Refinement

Refinement on F
$R = 0.0436$
$wR = 0.0455$
$S = 1.51$
1038 reflections
130 parameters
Only coordinates of H atoms refined

$w = 1/[\sigma^2(F) + 0.0002F^2]$
$(\Delta/\sigma)_{\rm max} = 0.108$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	$U_{\rm eq}$
C(1)	0.9644 (2)	0.3728 (2)	0.3632 (2)	0.040 (1)
C(2)	1.0584 (2)	0.4566 (2)	0.3903 (2)	0.050 (1)
C(3)	1.1701 (2)	0.4051 (3)	0.3778 (2)	0.056 (1)
C(4)	1.1881 (2)	0.2710 (3)	0.3384 (2)	0.059 (1)
C(5)	1.0947 (2)	0.1897 (3)	0.3092 (2)	0.058 (1)
C(6)	0.9824 (2)	0.2408 (3)	0.3201 (2)	0.047 (1)
N(7)	0.8513 (1)	0.4298 (2)	0.3801 (1)	0.047 (1)
C(8)	0.7548 (2)	0.3526 (2)	0.3965 (1)	0.040 (1)
O(9)	0.7458 (1)	0.2201 (1)	0.3949 (1)	0.049 (1)
N(10)	0.6589 (1)	0.4391 (2)	0.4165 (2)	0.051 (1)
C(11)	0.5546 (2)	0.3789 (2)	0.4293 (2)	0.048 (1)
N(12)	0.4648 (2)	0.3302 (2)	0.4403 (2)	0.065 (1)

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

C(1)—C(2)	1.386 (3)	C(1)—C(6)	1.381 (3)
C(1)—N(7)	1.425 (3)	C(2)—C(3)	1.383 (3)
N(7)—C(8)	1.339 (3)	C(3)—C(4)	1.373 (4)
C(8)—O(9)	1.219 (2)	C(4)—C(5)	1.378 (3)
N(10)—C(11)	1.335 (3)	C(5)—C(6)	1.386 (3)
C(11)—N(12)	1.139 (3)	C(8)—N(10)	1.392 (3)
C(2)—C(1)—C(6)	119.9 (2)	C(2)—C(1)—N(7)	117.6 (2)
C(6)—C(1)—N(7)	122.5 (2)	C(1)—C(2)—C(3)	120.1 (2)
C(2)—C(3)—C(4)	120.1 (2)	C(3)—C(4)—C(5)	119.8 (2)
C(4)—C(5)—C(6)	120.7 (2)	C(1)—C(6)—C(5)	119.3 (2)
C(1)—N(7)—C(8)	126.6 (2)	N(7)—C(8)—O(9)	126.4 (2)
N(7)—C(8)—N(10)	113.4 (2)	O(9)—C(8)—N(10)	120.2 (2)
C(8)—N(10)—C(11)	120.7 (2)	N(10)—C(11)—N(12)	178.7 (2)

All H atoms were located from difference Fourier syntheses and refined with fixed isotropic temperature factors of 0.08 Å^2.

Data collection and cell refinement: *P3/V Data Collection Program* (Siemens, 1989). Data reduction, structure solution and refinement, molecular graphics and preparation of material for publication: *SHELXTL-Plus* (Sheldrick, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: AS1148). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chen, H.-Y., He, Y.-K., Geng, F., Xue, Z.-Q. & Gao, H.-J. (1994). *Chin. Chem. Lett.* **5**, 197–198.

Gilli, G., Bertolasi, V., Bellucci, F., Fabrizio, B. & Ferretti, V. (1986). *J. Am. Chem. Soc.* **108**, 2420–2424.

Rabjohn, N. (1963). *Org. Synth. Collect.* **4**, 213–216.

Sheldrick, G. M. (1990). *SHELXTL-Plus. Structure Determination Software Programs.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1989). *P3/V Data Collection Program.* Version 4.11. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Williams, D. J. (1984). *Angew. Chem. Int. Ed. Engl.* **23**, 690–703.

Acta Cryst. (1995). **C51**, 1414–1416

1,1′-Dimethyl-4,5′-dinitro-2,2′-biimidazole, $C_8H_8N_6O_4$

José S. Casas, Alfonso Castiñeiras, Guadalupe Martínez, José Sordo* and José M. Varela

Departamento de Quimica Inorgánica, Universidade de Santiago de Compostela, 15706 Santiago de Compostela, Galicia, Spain

(*Received* 31 *May* 1994; *accepted* 20 *December* 1994)

Abstract

The two imidazole rings in the title compound, $C_8H_8N_6O_4$, have slightly different structure parameters, but both are essentially planar; they are rotated 29.4° about the central C—C bond. The planes of the 4-NO_2 and 5′-NO_2 groups form angles of 2.7 and 6.0°, respectively, with their imidazole rings.

Comment

Nitroimidazoles are valuable for chemotherapy of infections resulting from anaerobic bacteria and protozoa, and for sensitizing hypoxic cells to radiation; attempts are also being made to design nitroimidazoles that behave as cytotoxic agents *per se*

Acta Crystallographica Section C
ISSN 0108-2701 ©1995