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). C**51**, 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

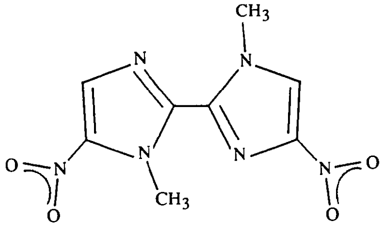
The two imidazole rings in the title compound, $C_8H_8N_6O_4$, have slightly different structure parameters, but both are essentially planar; they are rotated 29.4° about the central C—C bond. The planes of the 4-NO_2 and 5'-NO_2 groups form angles of 2.7 and 6.0°, respectively, with their imidazole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

against hypoxic cells (Edwards, 1990). These pharmacological applications have increased the interest in the structure and coordination chemistry of these compounds (Segalas & Beauchamp, 1992; De Bondt, Blaton, Peeters & De Ranter, 1994). In this paper, we report the crystal structure of 1,1′-dimethyl-4,5′-dinitro-2,2′-biimidazole (DMDNBIm), (I), which we obtained as a secondary product when preparing 1,1′-dimethyl-5-nitro-2,2′-biimidazole (Melloni, Dradi, Logeman, de Carneri & Trane, 1972).



(I)

The molecular structure and atomic numbering scheme of DMDNBIm are shown in Fig. 1. Rings *A* (N11—C12—N13—C14—C15) and *B* (N21—C22—N23—C24—C25) are both planar (χ^2 = 1.8 and 0.7, respectively), and are mutually rotated 29.4° about the central C12—C22 bond. The bond distances and angles in these rings are slightly different, the most significant differences being between the angles N13—C14—C15 and N23—C24—C25, and between N11—C15—C14 and N21—C25—C24; in both cases the angle is about 5° wider when the nitro group is present. The two nitro groups are twisted 6.0 and 2.7° with respect to planes of rings *A* and *B*, respectively.

Comparison of this structure with that of 2,2′-biimidazole (Cromer, Ryan & Storm, 1987) shows that the most significant difference affects the dihedral angle between the two imidazole rings, which changes from 4.6° in the unsubstituted biimidazole to the 29.4° of DMDNBIm, essentially because of the steric influence of the *N*-methyl groups. The angles that the two nitro groups form with the rings are narrower than in 4,4′,5,5′-tetranitro-2,2′-biimidazole (31.6° for 4-NO_2 and 17.1° for 5-NO_2) (Cromer & Storm, 1990).

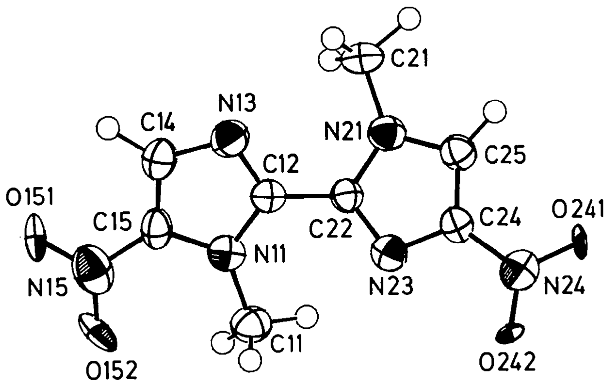


Fig. 1. *ORTEP*II (Johnson, 1976) drawing showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 80% probability level.

Experimental

Single crystals were obtained by slow evaporation of an aqueous ethanol solution.

Crystal data

$C_8H_8N_6O_4$
M_r = 252.19
Monoclinic
$P2_1/c$
a = 10.809 (2) Å
b = 9.581 (2) Å
c = 10.986 (2) Å
β = 109.54 (1)°
V = 1072.1 (6) Å^3
Z = 4
D_x = 1.562 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 5.5–13.2°
μ = 0.12 mm^{-1}
T = 293 K
Prismatic
0.30 × 0.25 × 0.15 mm
Amber

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min}$ = 0.986, $T_{\max}$ = 1.039
3439 measured reflections
3135 independent reflections

876 observed reflections [$I > 3\sigma(I)$]
$R_{\rm int}$ = 0.030
$\theta_{\max}$ = 30°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 13$
$l = -15 \rightarrow 15$
2 standard reflections frequency: 120 min intensity decay: none

Refinement

Refinement on F
R = 0.0515
wR = 0.0524
S = 1.11
876 reflections
164 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max}$ = 0.001

$\Delta\rho_{\max}$ = 0.18 e Å^{-3}
$\Delta\rho_{\min}$ = −0.23 e Å^{-3}
Extinction correction: Zachariasen (1963)
Extinction coefficient: 9.84 × 10^{-8}
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	$U_{\rm eq}$
O151	0.4764 (5)	0.2873 (6)	0.6244 (5)	0.079 (4)
O152	0.5166 (4)	0.0943 (5)	0.7325 (4)	0.060 (3)
O241	1.1180 (4)	−0.4158 (4)	0.5048 (4)	0.028 (2)
O242	1.0915 (4)	−0.3661 (5)	0.6879 (4)	0.034 (3)
N11	0.6824 (4)	0.0128 (5)	0.5930 (4)	0.042 (2)
N13	0.7258 (4)	0.1450 (5)	0.4441 (4)	0.053 (3)
N15	0.5307 (4)	0.1741 (6)	0.6518 (4)	0.070 (4)
N21	0.8548 (4)	−0.0970 (5)	0.3690 (4)	0.045 (3)

N23	0.9058 (4)	−0.1712 (5)	0.5722 (4)	0.044 (2)
N24	1.0670 (4)	−0.3490 (5)	0.5716 (4)	0.051 (3)
C11	0.6861 (5)	−0.1047 (7)	0.6797 (5)	0.056 (4)
C12	0.7472 (4)	0.0238 (6)	0.5073 (4)	0.037 (3)
C14	0.6435 (5)	0.2172 (7)	0.4909 (6)	0.053 (4)
C15	0.6167 (5)	0.1378 (6)	0.5819 (5)	0.044 (3)
C21	0.7958 (6)	−0.0199 (7)	0.2468 (5)	0.058 (4)
C22	0.8340 (5)	−0.0816 (6)	0.4853 (5)	0.036 (3)
C24	0.9724 (4)	−0.2426 (6)	0.5079 (5)	0.037 (3)
C25	0.9446 (5)	−0.2011 (6)	0.3841 (5)	0.043 (3)

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

O151—N15	1.223 (8)	N21—C21	1.477 (7)
O152—N15	1.218 (7)	N21—C22	1.377 (7)
O241—N24	1.235 (7)	N21—C25	1.363 (8)
O242—N24	1.226 (6)	N23—C22	1.324 (7)
N11—C11	1.467 (7)	N23—C24	1.351 (7)
N11—C12	1.352 (7)	N24—C24	1.448 (6)
N11—C15	1.377 (7)	C12—C22	1.453 (8)
N13—C12	1.333 (7)	C14—C15	1.363 (9)
N13—C14	1.356 (8)	C24—C25	1.351 (7)
N15—C15	1.433 (8)		
C11—N11—C12	127.2 (5)	N11—C12—N13	112.9 (5)
C11—N11—C15	128.7 (5)	N11—C12—C22	125.2 (5)
C12—N11—C15	104.1 (4)	N13—C12—C22	121.9 (6)
C12—N13—C14	105.7 (5)	N13—C14—C15	108.7 (5)
O151—N15—O152	124.8 (6)	N11—C15—N15	124.9 (5)
O151—N15—C15	115.3 (5)	N11—C15—C14	108.7 (5)
O152—N15—C15	119.9 (5)	N15—C15—C14	126.4 (5)
C21—N21—C22	129.5 (5)	N21—C22—N23	111.2 (5)
C21—N21—C25	123.4 (5)	N21—C22—C12	122.9 (4)
C22—N21—C25	107.2 (5)	N23—C22—C12	125.8 (5)
C22—N23—C24	103.7 (4)	N23—C24—N24	121.2 (4)
O241—N24—O242	124.6 (4)	N23—C24—C25	113.6 (5)
O241—N24—C24	117.5 (4)	N24—C24—C25	125.3 (5)
O242—N24—C24	118.0 (5)	N21—C25—C24	104.4 (5)

The structure was solved by direct methods and refined by a full-matrix least-squares procedure. Non-H atoms were refined with anisotropic displacement parameters. H atoms were obtained from a difference map and included in the structure-factor calculations with $B_{\rm iso}$ = 4.0 Å^2 fixed.

Data collection: *Enraf–Nonius CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *SDP*-VAX, Version 3.0 (1986) (Frenz, 1978). Program used to solve structure: *SHELXS*86, VMS version (Sheldrick, 1985). Program used to refine structure: *SDP*-VAX. Molecular graphics: *ORTEP*II (Johnson, 1976).

We thank the Xunta de Galicia, Spain, for financial support under project XUGA 20316B94.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1036). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cromer, D. T., Ryan, R. R. & Storm, C. B. (1987). *Acta Cryst.* **C43**, 1435–1437.
Cromer, D. T. & Storm, C. B. (1990). *Acta Cryst.* **C46**, 1957–1958.
De Bondt, H. L., Blaton, N. M., Peeters, O. M. & De Ranter, C. J. (1994). *Acta Cryst.* **C50**, 180–181.
Edwards, D. I. (1990). *Comprehensive Medicinal Chemistry*, edited by C. Hansh, P. G. Sammes & J. B. Taylor, Vol. 2, pp. 725–751. Oxford: Pergamon Press.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Melloni, P., Dradi, E., Logemann, W., de Carneri, I. & Trane, F. (1972). *J. Med. Chem.* **15**, 926–930.
Segalas, I. & Beauchamp, A. L. (1992). *Can. J. Chem.* **70**, 943–951.
Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.
Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1995). **C51**, 1416–1418

6-(2-Pyridyl)-3,5-hexadiyn-1-ol

M. C. Suh, S. N. Kim, H. J. Lee and S. C. Shim*

Department of Chemistry, Korea Advanced Institute of Science and Technology, 373-1 Kusung-Dong, Yusung-Gu, Taejon 305-701, Korea

I.-H. Suh, J.-H. Lee and J.-R. Park

Department of Physics, Chungnam National University, 220 Gung-Dong, Yusung-Gu, Taejon 305-764, Korea

(*Received* 12 *July* 1994; *accepted* 24 *January* 1995)

Abstract

The crystal structure of 6-(2-pyridyl)-3,5-hexadiyn-1-ol (PyHxD), $C_{11}H_9NO$, has been determined by a single-crystal X-ray diffraction study. The backbone diyne chain linking the pyridyl ring and the C(10)—C(11)—O group is linear. A dimer with a centre of symmetry is formed through two O—H···N hydrogen bonds and the crystal structure indicates that solid-state polymerization is inhibited because of these hydrogen bonds.

Comment

There is a growing interest in polydiacetylenes (PDAs) from both basic and applicational viewpoints. These polymers are of particular interest because of their conducting and nonlinear optical properties. Some diacetylenes (R—C≡C—C≡C—R') undergo solid-state polymerization upon heating, irradiation or mechanical stimulation. The polymerization reactivity of diacetylenes in the crystalline state is governed by the molecular packing of the monomer (Barrow, Milburn & Zeng, 1994). The title compound, (I), was synthesized in