

University of Wollongong

## Research Online

---

Australian Institute for Innovative Materials -  
Papers

Australian Institute for Innovative Materials

---

2000

### Two 1-substituted 4-nitroimidazoles

Maciej Kubicki

*Adam Mickiewicz University*

Teresa Borowiak

*Adam Mickiewicz University*

Jerzy Suwinski

*Silesian University of Technology*

Pawel Wagner

*University of Wollongong, pawel@uow.edu.au*

Follow this and additional works at: <https://ro.uow.edu.au/aiimpapers>



Part of the [Engineering Commons](#), and the [Physical Sciences and Mathematics Commons](#)

---

#### Recommended Citation

Kubicki, Maciej; Borowiak, Teresa; Suwinski, Jerzy; and Wagner, Pawel, "Two 1-substituted 4-nitroimidazoles" (2000). *Australian Institute for Innovative Materials - Papers*. 1127.  
<https://ro.uow.edu.au/aiimpapers/1127>

Research Online is the open access institutional repository for the University of Wollongong. For further information contact the UOW Library: [research-pubs@uow.edu.au](mailto:research-pubs@uow.edu.au)

---

## Two 1-substituted 4-nitroimidazoles

### Abstract

Crystalline 4-nitro-1-phenylimidazole, C<sub>9</sub>H<sub>7</sub>N<sub>3</sub>O<sub>2</sub>, (I), and 4'-nitro-1-phenyl-4,1'-biiimidazole, C<sub>12</sub>H<sub>9</sub>N<sub>5</sub>O<sub>2</sub>, (II), contain C–H⋯O and C–H⋯N hydrogen bonds, connecting the molecules into infinite chains. The aromatic fragments in both compounds are nearly planar. The dihedral angles between the benzene and imidazole rings are 26.78 (5)° in (I) and 29.36 (8)° in (II).

### Keywords

1, substituted, 4, nitroimidazoles, two

### Disciplines

Engineering | Physical Sciences and Mathematics

### Publication Details

Kubicki, M., Borowiak, T., Suwinski, J. & Wagner, P. (2000). Two 1-substituted 4-nitroimidazoles. *Acta Crystallographica, Section C: Crystal Structure*, 57 (1), 106-108.

## Two 1-substituted 4-nitroimidazoles

Maciej Kubicki,<sup>a</sup> Teresa Borowiak,<sup>a</sup> Jerzy Suwiński<sup>b</sup> and Paweł Wagner<sup>b\*</sup>

<sup>a</sup>Faculty of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland, and <sup>b</sup>Institute of Organic Chemistry and Technology, Silesian Technical University, Krzywoustego 4, 44-100 Gliwice, Poland  
Correspondence e-mail: wagner@polsl.gliwice.pl

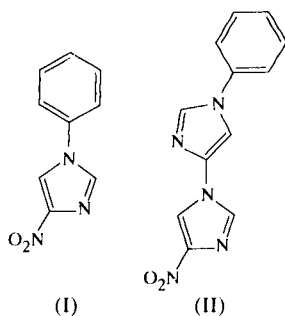
Received 30 May 2000

Accepted 20 October 2000

Crystalline 4-nitro-1-phenylimidazole, C<sub>9</sub>H<sub>7</sub>N<sub>3</sub>O<sub>2</sub>, (I), and 4'-nitro-1-phenyl-4,1'-biiimidazole, C<sub>12</sub>H<sub>9</sub>N<sub>5</sub>O<sub>2</sub>, (II), contain C—H...O and C—H...N hydrogen bonds, connecting the molecules into infinite chains. The aromatic fragments in both compounds are nearly planar. The dihedral angles between the benzene and imidazole rings are 26.78 (5)° in (I) and 29.36 (8)° in (II).

### Comment

The imidazole ring is present in a number of biologically active compounds, as well as in many natural products (Josephy & Mason, 1985). 1-Substituted 4-nitroimidazoles are generated in excellent yields from the reaction of 1,4-dinitroimidazoles with primary amines (Suwiński & Salwińska, 1990; Suwiński & Wagner, 1997). We present here the crystal structures of two compounds of this kind, *i.e.* (I) and (II).



In (I), both the benzene and imidazole rings are almost perfectly planar [maximum deviations from the least-squares planes are 0.0070 (9) and 0.0048 (7) Å for the benzene and imidazole rings, respectively] and the dihedral angle between these planes is 26.78 (5)°. The nitro group is twisted slightly but significantly [by 6.91 (14)°] with respect to the plane of the five-membered ring. The C2—N3 bond length is shorter than N3—C4 [1.311 (2) and 1.363 (2) Å, respectively].

In (II), all rings are also planar, with maximum deviations from the least-squares planes of 0.0039 (13), 0.0046 (12) and 0.0049 (17) Å for the nitroimidazole, imidazole and benzene

rings, respectively. The dihedral angle between the benzene and imidazole rings is 29.36 (8)° [a similar value was found for (I)], while the angle between the two imidazole rings is significantly smaller, 11.28 (13)°. The reason for this may be geometrical; the five-membered rings afford more space around the junction bond than the six-membered ring. The twist angle of the nitro group is smaller than in (I) [3.3 (3)°].

In compound (I), there is a statistically significant deformation of the endocyclic bond angles pattern in the benzene ring due to the influence of the electron-accepting nitroimidazole substituent. These deformations are generally consistent with those described by Domenicano (1988) and are comparable with those found for the cyano group. In (II), these effects are negligible because of the weaker electron-

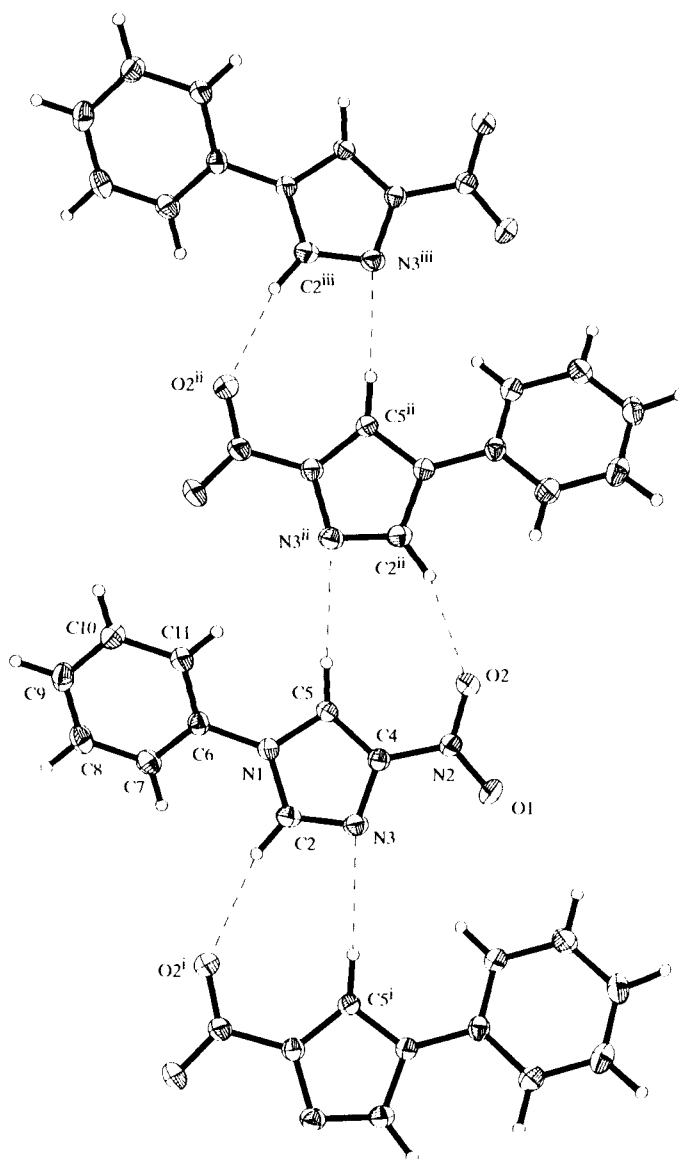


Figure 1

View of the hydrogen-bonded motif of compound (I) (Siemens, 1989) with the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and the H atoms are depicted as spheres of arbitrary radii. [Symmetry codes: (i)  $x - \frac{1}{2}, \frac{1}{2} - y, 1 - z$ ; (ii)  $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$ ; (iii)  $1 + x, y, z$ .]



Data collection

Kuma KM4-CCD diffractometer  
 $\omega/2\theta$  scans  
 6713 measured reflections  
 2882 independent reflections  
 1046 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.056$   
 $\theta_{max} = 29.35^\circ$   
 $h = -19 \rightarrow 15$   
 $k = -5 \rightarrow 5$   
 $l = -27 \rightarrow 28$

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.046$   
 $wR(F^2) = 0.105$   
 $S = 0.781$   
 2882 reflections  
 209 parameters  
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.16 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{min} = -0.17 \text{ e } \text{Å}^{-3}$   
 Extinction correction: *SHELXL97*  
 (Sheldrick, 1997)  
 Extinction coefficient: 0.0034 (10)

Table 4

Hydrogen-bonding geometry (Å, °) for (II).

| $D-H \cdots A$                     | $D-H$      | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|------------------------------------|------------|--------------|--------------|----------------|
| C7-H71 $\cdots$ O2 <sup>i</sup>    | 0.959 (19) | 2.34 (2)     | 3.249 (3)    | 158.9 (15)     |
| C5-H51 $\cdots$ N8 <sup>ii</sup>   | 0.955 (19) | 2.56 (2)     | 3.505 (3)    | 168.7 (15)     |
| C2-H21 $\cdots$ N3 <sup>ii</sup>   | 0.959 (19) | 2.56 (2)     | 3.309 (3)    | 135.2 (16)     |
| C10-H101 $\cdots$ N3 <sup>ii</sup> | 0.919 (19) | 2.65 (2)     | 3.460 (3)    | 148.0 (16)     |

Symmetry codes: (i)  $-x, -y, 1-z$ ; (ii)  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ .

(Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation* (Siemens, 1989); software used to prepare material for publication: *Stereochemical Workstation*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1151). Services for accessing these data are described at the back of the journal.

References

- Desiraju, G. R. & Steiner, T. (1999). In *The Weak Hydrogen Bond in Structural Chemistry and Biology*. Oxford University Press.  
 Domenicano, A. (1988). In *Stereochemical Applications of Gas-Phase Electron Diffraction, Part B*. Weinheim: VCH Publishers.  
 Josephy, P. D. & Mason, R. P. (1985). In *Bioactivation of Foreign Compounds*. New York: Academic Press.  
 Kuma Diffraction (1999). *CrysAlis CCD and CrysAlis Red*. Kuma Diffraction, Wrocław, Poland.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467-473.  
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.  
 Siemens (1989). *Stereochemical Workstation Operation Manual*. Release 3.4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Suwiński, J. & Salwińska, E. (1990). *Pol. J. Chem.* **64**, 813-817.  
 Suwiński, J. & Wagner, P. (1997). *Zesz. Nauk. Politech. Slask.* **135**, 73-80.

Table 3

Selected geometric parameters (Å, °) for (II).

|             |           |        |           |
|-------------|-----------|--------|-----------|
| N1-C2       | 1.357 (2) | N6-C11 | 1.423 (2) |
| N1-C5       | 1.360 (2) | N8-C7  | 1.317 (3) |
| N1-C9       | 1.415 (2) | C4-N3  | 1.362 (2) |
| N3-C2       | 1.308 (3) | C4-C5  | 1.353 (3) |
| N6-C7       | 1.353 (2) | C9-N8  | 1.354 (2) |
| N6-C10      | 1.374 (2) | C9-C10 | 1.345 (3) |
|             |           |        |           |
| C16-C11-C12 | 120.4 (2) |        |           |

For both compounds, data collection: *CrysAlis CCD* (Kuma Diffraction, 1999); cell refinement: *CrysAlis Red* (Kuma Diffraction, 1999) for compound (I), *CrysAlis CCD* for compound (II); data reduction: *CrysAlis Red* for compound (I), *CrysAlis CCD* for compound (II); program(s) used to solve structure: *SHELXS97*