O(3)—H···O(8)	2.681 (4)	O(7)—H···O(4^{v})	2.876 (4)
O(4)—H···O(7)	2.803 (4)	O(7)—H···O(6^{vi})	2.788 (4)
O(5)—H···O(1^{ii})	2.576 (4)		

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

Calculations were performed using *SHELXS*86 (Sheldrick, 1985).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71713 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1071]

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1-Methyl-5-nitro-2-phenylimidazole and 2-(*p*-Aminophenyl)-1-methyl-5-nitroimidazole

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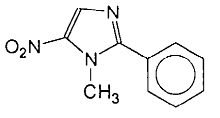
Abstract

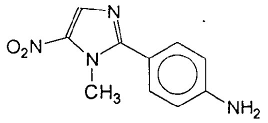
1-Methyl-5-nitro-2-phenylimidazole (I), $C_{10}H_9N_3O_2$, and 2-(*p*-aminophenyl)-1-methyl-5-nitroimidazole (II), $C_{10}H_{10}N_4O_2$, show similar bond distances and angles. Some differences are observed in the bond distances and angles in the phenyl rings due to the presence of the amino substituent and hydrogen bonds in (II).

Comment

Perspective views showing the atomic numbering scheme for (I) and (II) are given in Figs. 1 and 2. In both structures the imidazole and phenyl rings are planar within experimental error. The dihedral angles between the phenyl and imidazole rings in (I) and (II) are 138.58 (6) and 143.1 (1)°, respectively. The angle between the nitro group and the imidazole plane is 8.1 (2)° in (I) and 6.4 (3)° in (II).



(I)



(II)

The amino and nitro groups in (II) are involved in hydrogen bonding [N24—H241···O502^{i} 2.11 (5) Å, 151 (3)°; N24—H242···O501^{ii} 2.33 (4) Å, 167 (4)°; symmetry codes: (i) $x-1, y, z$; (ii) $x-1, -y+\frac{1}{2}, z-\frac{1}{2}$]. Each molecule of (II) is surrounded by four other molecules. This results in folded layers parallel to (010).

The bond lengths and bond angles in both compounds are similar, except for those in the phenyl rings. A comparison of the angles in the phenyl rings shows the influence of the amino substituent in (II). The differences found are

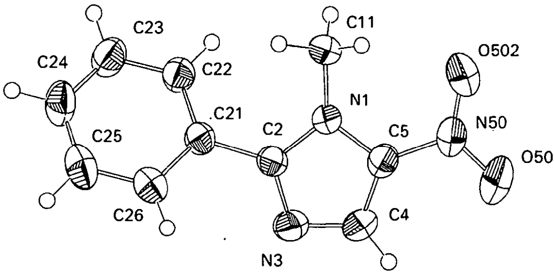


Fig. 1. A perspective view of molecule (I) with atomic numbering scheme. The displacement ellipsoids are plotted at the 50% probability level.

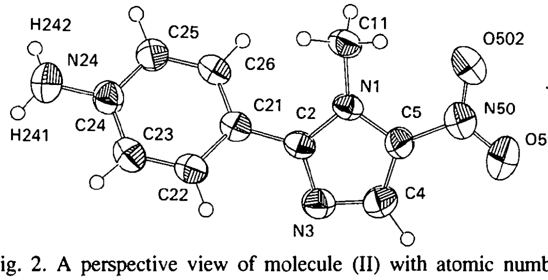


Fig. 2. A perspective view of molecule (II) with atomic numbering scheme. The displacement ellipsoids are plotted at the 50% probability level.

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larger than those given by Domenicano & Murray-Rust (1979) and are caused by the presence of the hydrogen bonding in (II).

Experimental

Compound (I)

Crystal data

$C_{10}H_9N_3O_2$
$M_r = 203.20$
Monoclinic
$P2_1/c$
$a = 11.127$ (1) Å
$b = 7.575$ (1) Å
$c = 12.046$ (1) Å
$\beta = 108.85$ (1)°
$V = 960.9$ (2) Å^3
$Z = 4$
$D_x = 1.4046$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 38 reflections
$\theta = 10.03$-12.48°
$\mu = 0.0951$ mm^{-1}
$T = 293$ K
Needle
0.30 × 0.10 × 0.10 mm
Colourless

Data collection

Stoe Stadi-4 four-circle diffractometer
ω scans
Absorption correction: none
4660 measured reflections
2224 independent reflections
1580 observed reflections [$I > 2.0\sigma(I)$]

$R_{\rm int} = 0.016$
$\theta_{\rm max} = 27.89$°
$h = -14 \rightarrow 14$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 16$
6 standard reflections frequency: 60 min intensity variation: 5%

Refinement

Refinement on F
$R = 0.041$
$wR = 0.063$
$S = 1.2695$
1580 reflections
172 parameters
All H-atom parameters refined
$w = 1.0179/[\sigma^2(F) + 0.002064F^2]$

$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.22$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1989)

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

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

	x	y	z	$U_{\rm eq}$
N1	0.6610 (1)	0.0941 (2)	0.7529 (1)	0.0364 (4)
C11	0.6372 (2)	0.0142 (2)	0.6370 (1)	0.0435 (5)
C2	0.5730 (1)	0.1261 (2)	0.8064 (1)	0.0375 (4)
C21	0.4347 (1)	0.1169 (2)	0.7485 (1)	0.0389 (4)
C22	0.3772 (1)	0.1849 (2)	0.6365 (1)	0.0452 (5)
C23	0.2462 (2)	0.1863 (3)	0.5876 (2)	0.0568 (6)
C24	0.1718 (2)	0.1214 (3)	0.6504 (2)	0.0629 (7)
C25	0.2276 (2)	0.0540 (3)	0.7615 (2)	0.0602 (8)
C26	0.3586 (2)	0.0512 (2)	0.8111 (2)	0.0487 (6)
N3	0.6259 (1)	0.1744 (2)	0.9181 (1)	0.0507 (5)
C4	0.7524 (2)	0.1753 (2)	0.9367 (1)	0.0537 (6)
C5	0.7758 (1)	0.1261 (2)	0.8372 (1)	0.0443 (6)
N50	0.8940 (1)	0.1195 (2)	0.8171 (2)	0.0623 (6)
O501	0.9887 (1)	0.1454 (3)	0.9012 (2)	0.1055 (9)
O502	0.8965 (1)	0.0952 (3)	0.7175 (2)	0.0971 (9)

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

N1—C11	1.465 (1)	C23—C24	1.380 (3)
N1—C2	1.357 (1)	C24—C25	1.378 (3)
N1—C5	1.372 (1)	C25—C26	1.385 (2)
C2—C21	1.472 (1)	N3—C4	1.352 (2)
C2—N3	1.334 (1)	C4—C5	1.358 (2)
C21—C22	1.392 (1)	C5—N50	1.413 (2)
C21—C26	1.395 (2)	N50—O501	1.219 (2)
C22—C23	1.385 (2)	N50—O502	1.223 (3)
C2—N1—C5	105.0 (1)	C23—C24—C25	120.2 (2)
C11—N1—C5	128.1 (1)	C24—C25—C26	120.4 (2)
C11—N1—C2	126.2 (1)	C21—C26—C25	119.9 (2)
N1—C2—N3	112.2 (1)	C2—N3—C4	105.4 (1)
N1—C2—C21	124.8 (1)	N3—C4—C5	109.8 (1)
C21—C2—N3	123.0 (1)	N1—C5—C4	107.7 (1)
C2—C21—C26	119.0 (1)	C4—C5—N50	128.0 (1)
C2—C21—C22	121.7 (1)	N1—C5—N50	124.2 (1)
C22—C21—C26	119.1 (1)	C5—N50—O502	119.4 (1)
C21—C22—C23	120.4 (1)	C5—N50—O501	117.0 (2)
C22—C23—C24	120.0 (2)	O501—N50—O502	123.5 (2)

Compound (II)

Crystal data

$C_{10}H_{10}N_4O_2$
$M_r = 218.21$
Monoclinic
$P2_1/c$
$a = 11.864$ (1) Å
$b = 7.434$ (1) Å
$c = 12.216$ (2) Å
$\beta = 110.05$ (1)°
$V = 1012.1$ (2) Å^3
$Z = 4$
$D_x = 1.4321$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 47 reflections
$\theta = 10.07$-12.94°
$\mu = 0.0978$ mm^{-1}
$T = 293$ K
Needle
0.30 × 0.10 × 0.10 mm
Orange

Data collection

Stoe Stadi-4 four-circle diffractometer
ω scans
Absorption correction: none
2686 measured reflections
2334 independent reflections
790 observed reflections [$I > 2.0\sigma(I)$]

$R_{\rm int} = 0.018$
$\theta_{\rm max} = 27.49$°
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 16$
3 standard reflections frequency: 60 min intensity variation: 7%

Refinement

Refinement on F
$R = 0.037$
$wR = 0.040$
$S = 0.9902$
790 reflections
185 parameters
All H-atom parameters refined
$w = 0.6504/[\sigma^2(F) + 0.000440F^2]$

$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1989)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

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

	x	y	z	$U_{\rm eq}$
N1	0.6626 (2)	0.0962 (4)	0.2523 (2)	0.043 (1)
C11	0.6331 (4)	0.0214 (6)	0.1343 (3)	0.050 (2)

C2	0.5842 (3)	0.1271 (4)	0.3089 (2)	0.042 (1)
C21	0.4545 (3)	0.1193 (5)	0.2560 (3)	0.041 (1)
C22	0.3854 (3)	0.0582 (5)	0.3210 (3)	0.048 (1)
C23	0.2628 (3)	0.0585 (5)	0.2770 (3)	0.055 (2)
C24	0.2017 (3)	0.1215 (5)	0.1645 (3)	0.054 (2)
N24	0.0795 (3)	0.1208 (7)	0.1189 (4)	0.090 (2)
C25	0.2690 (3)	0.1835 (5)	0.0981 (3)	0.052 (2)
C26	0.3927 (3)	0.1819 (5)	0.1440 (3)	0.046 (2)
N3	0.6414 (3)	0.1681 (4)	0.4214 (2)	0.056 (1)
C4	0.7593 (3)	0.1646 (5)	0.4363 (3)	0.058 (2)
C5	0.7748 (3)	0.1216 (5)	0.3348 (3)	0.048 (1)
N50	0.8830 (3)	0.1151 (5)	0.3106 (3)	0.071 (2)
O501	0.9777 (2)	0.1343 (5)	0.3921 (3)	0.093 (1)
O502	0.8786 (2)	0.0923 (6)	0.2105 (3)	0.116 (2)

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

N1—C11	1.471 (4)	C24—N24	1.363 (4)
N1—C2	1.355 (4)	C24—C25	1.397 (5)
N1—C5	1.380 (3)	C25—C26	1.380 (4)
C2—C21	1.452 (4)	N3—C4	1.347 (5)
C2—N3	1.343 (3)	C4—C5	1.353 (5)
C21—C22	1.398 (5)	C5—N50	1.413 (5)
C21—C26	1.392 (4)	N50—O501	1.228 (3)
C22—C23	1.367 (4)	N50—O502	1.218 (5)
C23—C24	1.397 (4)		
C2—N1—C5	105.3 (2)	C23—C24—N24	121.4 (3)
C11—N1—C5	127.8 (3)	N24—C24—C25	120.2 (3)
C11—N1—C2	126.1 (3)	C24—C25—C26	120.1 (3)
N1—C2—N3	111.5 (3)	C21—C26—C25	122.1 (3)
N1—C2—C21	125.1 (3)	C2—N3—C4	105.7 (3)
C21—C2—N3	123.5 (3)	N3—C4—C5	110.0 (3)
C2—C21—C26	123.0 (3)	N1—C5—C4	107.6 (3)
C2—C21—C22	119.9 (3)	C4—C5—N50	128.2 (3)
C22—C21—C26	116.9 (3)	N1—C5—N50	124.1 (3)
C21—C22—C23	121.9 (3)	C5—N50—O502	119.0 (3)
C22—C23—C24	120.7 (3)	C5—N50—O501	118.1 (3)
C23—C24—C25	118.3 (3)	O501—N50—O502	122.9 (4)

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

The authors thank Merck Sharp & Dohme (Rahway, New Jersey) for providing the crystals. One of us (TO) is indebted to the Research Council of K. U. Leuven (Belgium) for a Senior Fellowship No. F/92/32.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71819 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1071]

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Methyl DL-3-Benzyl-2,2-dioxo-1,2,3-oxathiazolidine-4-carboxylate – an Intermediate for Amino Acid Synthesis

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Abstract

The first structural measurements on a cyclic sulfamidate, DL-3-benzyl-2,2-dioxo-1,2,3-oxathiazolidine-4-carboxylate, $C_{11}H_{13}NO_5S$, show that the ring N atom has almost planar bonding geometry (sum of angles at N = 357.8°), in contrast to that found in simple dialkyl sulfonamides and acyclic sulfamate esters. The structure contains a close 1,4 N···O contact [2.764 (3) Å].

Comment

The methyl ester of DL-serine can be converted to the 1,2,3-oxathiazolidine dioxide (1) by three steps: benzylation, cyclization with thionyl chloride, and oxidation with sodium periodate catalysed by ruthenium trichloride. This cyclic sulfamidate reacts with nucleophiles by ring opening at C-5 to produce precursors of new amino acids (Gritsonie, Pilkington & Wallis, 1994; Baldwin, Spivey & Schofield, 1990). We were interested in the bonding geometry at the N atom in (1), and how this might be affected by the sizes of the N-atom substituent and the adjacent carboxylic ester group. By analogy with *N,N*-dimethyl sulfonamides in which the N atom is typically *circa* 0.27 Å out of the plane defined by the atoms to which it is bonded (Häkkinen, Ruostesuo & Kivekäs, 1988; Cromer, Ryan, Karthikeyan & Paine, 1990), pyramidal geometry around the N atom was expected. The only *N,N*-dialkyl sulfamate esters recorded in the Cam-