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

O1—C5	1.361 (2)	C4—C5	1.384 (3)
O1—C8	1.442 (2)	C5—C6	1.389 (3)
O2—C1	1.207 (2)	C6—C7	1.375 (3)
C1—C2	1.459 (3)	C8—C9	1.503 (3)
C2—C3	1.385 (3)	C9—C10	1.518 (3)
C2—C7	1.389 (3)	C10—C10^{i}	1.514 (4)
C3—C4	1.380 (3)		
C5—O1—C8	118.4 (1)	O1—C5—C6	116.3 (2)
O2—C1—C2	125.5 (2)	C4—C5—C6	119.2 (2)
C1—C2—C3	121.8 (2)	C5—C6—C7	120.4 (2)
C1—C2—C7	120.4 (2)	C2—C7—C6	121.0 (2)
C3—C2—C7	117.8 (2)	O1—C8—C9	108.6 (2)
C2—C3—C4	121.9 (2)	C8—C9—C10	112.0 (2)
C3—C4—C5	119.6 (2)	C9—C10—C10^{i}	113.6 (2)
O1—C5—C4	124.6 (2)		

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

The unit weights applied in the refinement gave reasonably constant average values of $<w\Delta^2>$ between different F_o and $\sin^2\theta$ intervals.

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET*4 and *CELDIM* (*CAD-4/PC Software*). Data reduction: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *CRYSTALS*. Molecular graphics: *CRYSTALS*. Software used to prepare material for publication: *CRYSTALS*.

This work was supported by the Centre National de la Recherche Scientifique.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1393). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-(2-Aminophenyl)-1,3,4-oxadiazol-2(3*H*)-one

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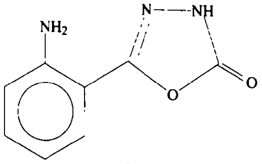
Abstract

The title molecule, $C_8H_7N_3O_2$, is nearly planar [angle of 2.0 (1)° between the two ring planes], and contains an intramolecular N—H···N hydrogen bond and two intermolecular N—H···O hydrogen bonds

Comment

The structure of the title compound, (I), was determined to confirm the molecular structure assigned on the basis of the methods of preparation (Davidson, 1984, 1988)



(I)

and spectroscopic evidence (Tihanyi, Gál & Dvortsák, 1983). The molecule is nearly planar [with a maximum deviation of 0.037 (2) Å for O1]. The two rings are each almost exactly planar [maximum deviation of 0.012 (3) Å for C1 in the five-membered ring, and 0.004 (3) Å for three atoms in the six-membered ring], with an angle of 2.0° between the mean planes of the rings. The planarity is probably assisted by an

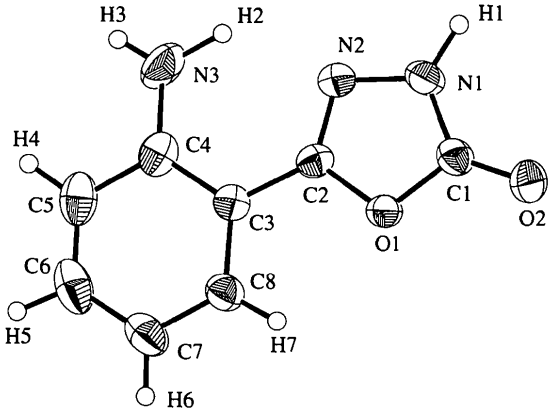


Fig. 1. View of the title molecule with displacement ellipsoids plotted at the 33% probability level. H atoms are drawn as circles of arbitrary radius.

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intramolecular N3—H···N2 hydrogen bond [N···N = 2.757 (4) Å, N—H···N = 130 (3)°]. Bond lengths and angles are normal, and close to those in related molecules (*e.g.* King, Preston, Suffolk & Turnbull, 1979; Allen *et al.*, 1987). Molecules are linked by an N1—H···O2($\frac{1}{2} + x$, $\frac{1}{2} - y$, $-z$) hydrogen bond [N···O = 2.771 (3) Å, N—H···O = 173 (2)°] and a weaker N3—H···O2($x + 1$, y, $z + 1$) bond [N···O = 2.977 (4) Å, N—H···O = 133 (3)°].

Experimental

The title material was synthesized by reaction of anthranilic acid hydrazide with 1,1-carbonyldiimidazole (Davidson, 1984).

Crystal data

$C_8H_7N_3O_2$
$M_r = 177.16$
Orthorhombic
$P2_12_12_1$
$a = 6.4957$ (6) Å
$b = 24.723$ (2) Å
$c = 5.1393$ (4) Å
$V = 825.3$ (1) Å^3
$Z = 4$
$D_x = 1.426$ Mg m^{-3}
D_m not measured

Cu *Kα* radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 27.7$–$54.1°$
$\mu = 0.854$ mm^{-1}
$T = 294$ K
Needle
0.45 × 0.15 × 0.15 mm
Colorless

Data collection

Rigaku AFC-6*S* diffractometer
ω–2θ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
  $T_{\min} = 0.892$, $T_{\max} = 1.000$
1073 measured reflections
1073 independent reflections

819 observed reflections [$I > 3\sigma(I)$]
$\theta_{\max} = 77.39°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 31$
$l = 0 \rightarrow 6$
3 standard reflections monitored every 200 reflections
  intensity decay: 1.0%

Refinement

Refinement on F
$R = 0.031$
$wR = 0.041$
$S = 1.51$
819 reflections
147 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o) + 0.00016|F_o|^2]$
$(\Delta/\sigma)_{\max} = 0.0000$

$\Delta\rho_{\max} = 0.13$ e Å^{-3}
$\Delta\rho_{\min} = -0.10$ e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 0.000014158
Atomic scattering factors from International Tables for Crystallography (1992, Vol. C, Tables 4.2.6.8, 6.1.1.1 and 6.1.1.2)

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	$U_{\rm eq}$
O1	−0.1333 (2)	0.34297 (6)	0.4049 (4)	0.0511 (5)
O2	−0.2357 (3)	0.29247 (8)	0.0600 (4)	0.0709 (6)
N1	0.0822 (3)	0.28408 (8)	0.2596 (5)	0.0573 (6)
N2	0.1809 (3)	0.31069 (9)	0.4608 (5)	0.0537 (6)
N3	0.4107 (4)	0.3562 (1)	0.8553 (7)	0.0835 (10)
C1	−0.1064 (4)	0.30373 (9)	0.2208 (6)	0.0524 (7)
C2	0.0477 (3)	0.34553 (9)	0.5426 (5)	0.0464 (6)
C3	0.0651 (4)	0.38532 (9)	0.7470 (5)	0.0476 (6)
C4	0.2434 (4)	0.3889 (1)	0.8991 (6)	0.0589 (8)
C5	0.2484 (6)	0.4278 (1)	1.0979 (7)	0.072 (1)
C6	0.0856 (8)	0.4616 (1)	1.1391 (7)	0.080 (1)
C7	−0.0892 (6)	0.4584 (1)	0.9885 (7)	0.0722 (10)
C8	−0.0995 (5)	0.4204 (1)	0.7945 (6)	0.0559 (8)

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

O1—C1	1.367 (3)	C2—C3	1.444 (3)
O1—C2	1.374 (3)	C3—C4	1.400 (4)
O2—C1	1.211 (3)	C3—C8	1.398 (3)
N1—N2	1.383 (3)	C4—C5	1.405 (4)
N1—C1	1.333 (3)	C5—C6	1.364 (5)
N2—C2	1.291 (3)	C6—C7	1.376 (5)
N3—C4	1.373 (4)	C7—C8	1.373 (4)
C1—O1—C2	106.2 (2)	C2—C3—C8	119.3 (2)
N2—N1—C1	111.4 (2)	C4—C3—C8	119.8 (2)
N1—N2—C2	104.5 (2)	N3—C4—C3	121.8 (3)
O1—C1—O2	123.2 (2)	N3—C4—C5	120.3 (3)
O1—C1—N1	105.8 (2)	C3—C4—C5	117.9 (3)
O2—C1—N1	131.0 (2)	C4—C5—C6	121.0 (3)
O1—C2—N2	112.1 (2)	C5—C6—C7	121.1 (3)
O1—C2—C3	118.2 (2)	C6—C7—C8	119.2 (3)
N2—C2—C3	129.7 (2)	C3—C8—C7	121.0 (3)
C2—C3—C4	120.9 (2)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2$^{\rm i}$	0.98 (3)	1.79 (3)	2.771 (3)	173 (2)
N3—H2···N2	1.07 (5)	1.94 (5)	2.757 (4)	130 (3)
N3—H3···O2$^{\rm ii}$	0.80 (4)	2.37 (4)	2.977 (4)	133 (3)

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

The absolute configuration of the crystal studied could not be determined from the small anomalous dispersion terms.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR*92 (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and two packing diagrams have been deposited with the IUCr (Reference: FG1227). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,3-(Oxydiethyl)-1,2-di(*o*-methylphenyl)guanidinium Chloride Monohydrate

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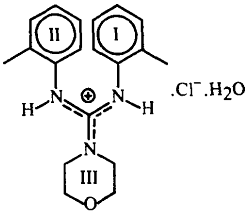
Abstract

In the title compound, bis(2-methylphenylamino)-(morpholino)methylium chloride hydrate, $C_{19}H_{24}N_3O^+.Cl^-.H_2O$, the guanidinium moiety is protonated; the three C—N bond lengths are therefore almost identical. Steric strain as a result of the bulky substituents leads to distortions of the guanidinium skeleton. The co-crystallized water molecule accepts no conventional hydrogen bonds but only weak C—H···O interactions.

Comment

The agrochemical and pharmaceutical activities of guanidines are well established in the literature (Molina, Alajarin & Saez, 1983). The conformation of the title compound, (1), as observed in the crystal structure is shown in Fig. 1. Since (1) has been crystallized as a chloride, the guanidine moiety is protonated (*i.e.* N8 and N10 both carry an H atom). The three guanidyl C—N bond lengths are identical within their e.s.d.s [1.337 (2), 1.335 (2) and 1.335 (2) Å for C9—N8, C9—N10 and C9—N11, respectively], indicating delocalization of the positive charge. The conformation around N11 is planar with a sum of valence angles of 360.0 (2)°. This contrasts with the geometry of related uncharged molecules such as 3,3-(oxydiethyl)-1,2-diphenylguanidine where the guanidine C—N bonds are unequal with a defined C=N double bond [1.291 (9) Å], and a significantly pyramidal geometry of the N atom to which the oxydiethyl moiety is bonded (Sudha *et al.*, 1995*b*).



(1)

The N11-oxydiethyl moiety represents a morpholino group which is in a chair conformation. Generally, the pyramidality of morpholino N atoms may cover wide ranges between 337 and 360° (Wong-Ng, Nyburg, Awwal, Jankie & Kresge, 1982). If N is part of an uncharged guanidine group, we have found sums of angles of 351.8 (9) (Sudha *et al.*, 1995*b*) and 358.2 (6)° (Sudha *et al.*, 1995*a*). Both phenyl rings are oriented roughly perpendicular to the guanidine plane [dihedral angle 79.1 (1)° for ring I and 71.7 (1)° for ring II]; the dihedral angle between the phenyl rings is 32.5 (1)°. Steric conflict of the bulky methylphenyl substituents leads to significant distortion of the guanidine moiety, in particular to opening of the angles at N8, C9 and N10 [C9—N8—C1 = 125.2 (2), N8—C9—N10 = 121.4 (2),

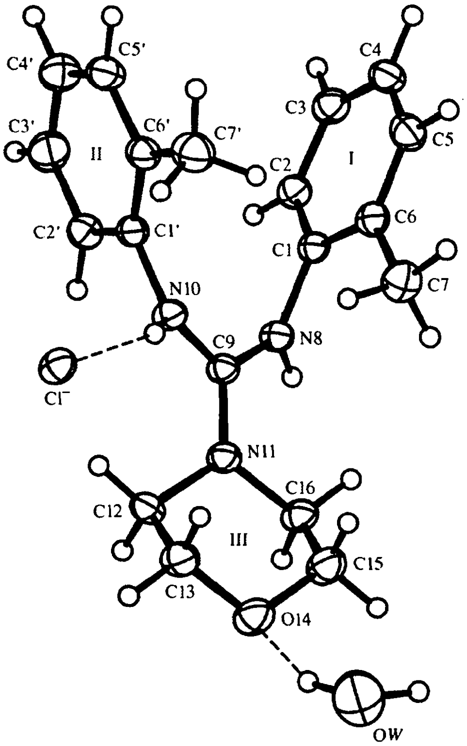


Fig. 1. Molecular structure and atom labelling of the title compound shown in a minimal overlap projection. Displacement ellipsoids are drawn at the 30% probability level.