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1-Ethyl-2-nitroguanidine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.106
Data-to-parameter ratio = 11.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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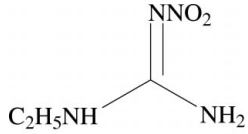
The structure of the title compound, $C_3H_8N_4O_2$, is similar to other nitrimines. The nitroguanyl group is planar and is stabilized by an intramolecular hydrogen bond. Intermolecular hydrogen bonds hold the molecules together in the crystal.

Comment

As a continuation of work on the structures of nitrimines (Choi, 1981; Nordenson, 1981*a*,*b*; Nordenson & Hvoslef, 1981; Rice *et al.*, 1984; Oyumi *et al.*, 1987; Gao *et al.*, 1991; Astachov *et al.*, 2001; Vasiliev *et al.*, 2001), we report here the crystal structure of 1-ethyl-2-nitroguanidine, (I). The molecular structure of (I) corresponds to that of previously investigated nitrimines. Like other nitrimines, the delocalization of π-electron density gives values of C—N, N—N and N—O bond lengths intermediate between those characteristic of single and double bonds (Table 1). The formal double bond C1═N2 is, in fact, 0.052–0.057 Å longer than the C1—N3 and C1—N4 bonds. The planar geometry of the nitroguanyl group (r.m.s. deviation 0.065 Å and maximum deviation 0.104 Å) is stabilized by an O1···H1 intramolecular hydrogen bond (Table 2). In other nitrimines, the N—H···O angles are in the range 105–126°, and the O···H distance is in the range 1.72–2.24 Å (Allen, 2002). The ethyl group does not participate in conjugation of the nitroguanyl fragment and, as a consequence, the N4—C2 bond length is close to those observed in compounds with a single C—N bond (Allen, 2002).



(I)

In the crystal structure, each molecule of (I) is connected by three intermolecular hydrogen bonds with two neighbouring molecules (Fig. 1 and Table 2). Atom O1 is involved in intramolecular hydrogen bonding and forms a hydrogen bond with H2 of another molecule. In addition, the molecules are connected, in pairs, by two N2···H3 hydrogen bonds. The second O atom, O2, does not participate in hydrogen bonding and, for this reason, the N1—O2 bond is shorter than the N1—O1 bond by 0.027 Å. This hydrogen-bond network is typical for nitrimines (Allen, 2002).

The structure of (I) agrees well with the methyl derivative of nitroguanidine 1-methyl-2-nitroguanidine, (II) (Nordenson, 1981*a*). The low-temperature study of (II) exhibits almost identical geometry and, with the exception of N1—N2, all

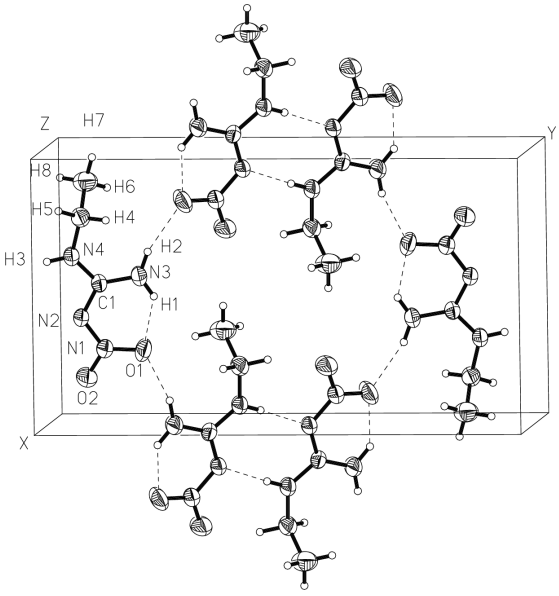


Figure 1
The packing arrangement (I) in the crystal, with the atomic numbering scheme. Dashed lines indicate intra- and intermolecular hydrogen bonds.

other differences in bond lengths between (I) and (II) do not exceed 2σ. The molecular packing in (I) and (II) is also very similar: they have the same space-group symmetry and number of molecules in the unit cell, and the same hydrogen bonds with nearly equal geometric parameters.

Experimental

Compound (I) was synthesized as described previously by Fishbein & Gallaghan (1954). Single crystals were obtained by evaporation in air of an aqueous solution of (I).

Crystal data

$C_3H_8N_4O_2$
M_r = 132.13
Monoclinic, $P2_1/n$
a = 8.9336 (9) Å
b = 16.087 (2) Å
c = 4.3173 (4) Å
β = 96.119 (8)°
V = 616.92 (11) Å^3
Z = 4
D_x = 1.423 Mg m^{-3}
Cu Kα radiation
Cell parameters from 25 reflections
θ = 20–27°
μ = 1.02 mm^{-1}
T = 293 (2) K
Lump, colourless
0.32 × 0.27 × 0.26 mm

Data collection

Kuma KM-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1325 measured reflections
1168 independent reflections
1049 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 69.9°
$h = -10 \rightarrow 10$
$k = -19 \rightarrow 16$
$l = 0 \rightarrow 5$
2 standard reflections every 50 reflections
intensity variation: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.106
S = 1.04
1168 reflections
102 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.1271P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.044 (3)

Table 1
Selected geometric parameters (Å, °).

N2—N1	1.3276 (15)	C1—N3	1.3164 (17)
N2—C1	1.3718 (16)	C1—N4	1.3188 (17)
O2—N1	1.2303 (15)	N4—C2	1.4528 (17)
O1—N1	1.2555 (15)	C2—C3	1.502 (2)
N1—N2—C1	119.72 (10)	O2—N1—O1	120.36 (11)
N3—C1—N4	121.28 (12)	O2—N1—N2	115.99 (11)
N3—C1—N2	126.41 (12)	O1—N1—N2	123.64 (11)
N4—C1—N2	112.31 (11)	N4—C2—C3	113.56 (12)
C1—N4—C2	125.65 (11)		
N1—N2—C1—N3	4.4 (2)	C1—N2—N1—O2	−176.20 (12)
N1—N2—C1—N4	−176.27 (11)	C1—N2—N1—O1	4.9 (2)
N3—C1—N4—C2	−1.5 (2)	C1—N4—C2—C3	89.75 (17)
N2—C1—N4—C2	179.13 (12)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H3···N2^{i}	0.82 (2)	2.23 (2)	3.035 (2)	170 (1)
N3—H2···O1^{ii}	0.87 (2)	2.04 (2)	2.896 (2)	165 (1)
N3—H1···O1	0.85 (2)	1.89 (2)	2.565 (2)	136 (1)

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

H atoms were located in a difference Fourier map and were refined as riding atoms for CH_2 and CH_3 groups and as free atoms for NH and NH_2 groups. Furthermore, the C—H distance was refined as one parameter for CH_2 and CH_3 groups.

Data collection: *KM*-4 *Software* (Kuma, 1991); cell refinement: *KM*-4 *Software*; data reduction: *DATARED* in *KM*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL*97.

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