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N,*N*-Dimethylbiguanidium nitrate

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Key indicators

Single-crystal X-ray study
T = 184 K
Mean σ(C–N) = 0.003 Å
R factor = 0.054
wR factor = 0.129
Data-to-parameter ratio = 10.1

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

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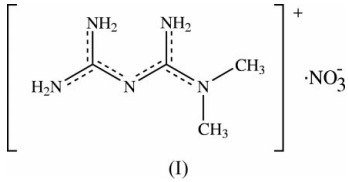
The cation in the title compound, $C_4H_{12}N_5^+\cdot NO_3^-$, is protonated at one imino group, and through this an intramolecular N—H···N hydrogen bond is formed, which stabilizes the conformation of the cation in the structure. The dihedral angle between the two guanidine groups is 51.7 (1)°.

Comment

Metformin is an antihyperglycemic agent which improves glucose tolerance in type-2 diabetic patients, lowering both basal and postprandial plasma glucose levels. There are some benefits for these diabetic patients who use it in order to control their plasma glucose levels. From pharmacological studies, metformin acts by improving peripheral sensitivity to insulin, inhibiting gastrointestinal absorption of glucose, and decreasing hepatic glucose production. Researchers indicate that metformin alone does not produce hypoglycemia in either diabetic or non-diabetic individuals (Davidson & Peters, 1997; Jackson *et al.*, 1987; Klip & Leiter, 1990). In previous reports, we studied that structures of complexes of metformin with Zn^{2+}, Cu^{2+} and Ni^{2+} (Zhu *et al.*, 2002, 2002*a*,*b*). Magnesium, a ubiquitous element that plays a fundamental role in many cellular reactions, is involved in more than 300 enzymatic reactions in which food is catabolized and new chemical products are formed; these include glycogen breakdown, fat oxidation, protein synthesis, ATP synthesis, and the second messenger system (Lukaski, 2000). In order to obtain information regarding the interaction between metformin and magnesium ions, magnesium(II) nitrate was employed in our current research. However, the title compound, (I), was obtained instead of the Mg^{2+} complex. It can be compared with $(C_4H_{12}N_5)[TlBr_4]$ (He *et al.*, 2002) and $C_4H_{12}N_5^+\cdot Cl^-$ (Hariharan *et al.*, 1989).



(I)

Selected geometric parameters are listed in Table 1, and a perspective view of the structure is shown in Fig. 1. In the structure of (I), the planes of the two guanidine groups with a dihedral angle of 51.7 (1)° planar angle. Atoms C1 and C2 deviate by −0.042 (3) and −0.019 (3) Å from the planes N1/N2/N3/C1 and N3/N4/N5/C2, respectively. The C—N bond distances of (I), in the range of 1.324 (3)–1.336 (3) Å, are shorter than a double bond, indicating a delocalization of

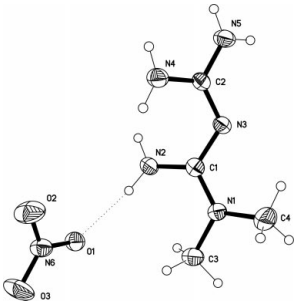


Figure 1
A view of the asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii. Dotted lines represent hydrogen bonds.

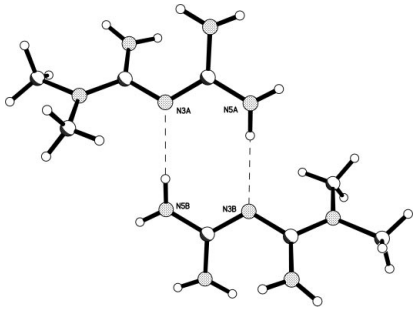


Figure 2
The dimer formed *via* hydrogen bonds, indicated by dashed lines.

π-electron density across the biguanide group. 1,1-Dimethylbiguanide as a form of cation appears in several compounds, such as $(C_4H_{12}N_5)[ZnCl_3]$ (Zhu *et al.*, 2002), and those previously mentioned. Compared with these compounds, a notable difference can be detected from the packing diagrams. As shown in Fig. 2, two molecules are connected by two N5—H···N3 hydrogen bonds, forming an elongated hexagon. The hydrogen bonding information is given in Table 2 and a packing diagram is shown in Fig. 3. In addition, the molecules in the crystal are held together by van der Waals forces and by a number of intermolecular N—H···O interactions. A weak N—H···N intramolecular hydrogen bond stabilizes the cation conformation (Fig. 1).

Experimental

Crystals of (I) were grown from an aqueous solution of magnesium(II) nitrate hexahydrate (100.0 mmol) and *N,N*-dimethylbiguanide hydrochloride (100.0 mmol). The solution was left at room temperature and crystals formed after a few days. The elemental analysis result was in agreement with the structural composition of (I).

Crystal data

$C_4H_{12}N_5^+\cdot NO_3^-$
M_r = 192.20
Triclinic, $P\overline{1}$
a = 7.204 (3) Å
b = 7.534 (3) Å
c = 8.821 (4) Å
α = 78.109 (5)°
β = 73.979 (6)°
γ = 85.353 (6)°
V = 450.1 (3) Å^3
Z = 2
D_x = 1.418 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2484 reflections
θ = 2.8–24.7°
μ = 0.12 mm^{-1}
T = 184 (2) K
Block, colorless
0.30 × 0.20 × 0.10 mm

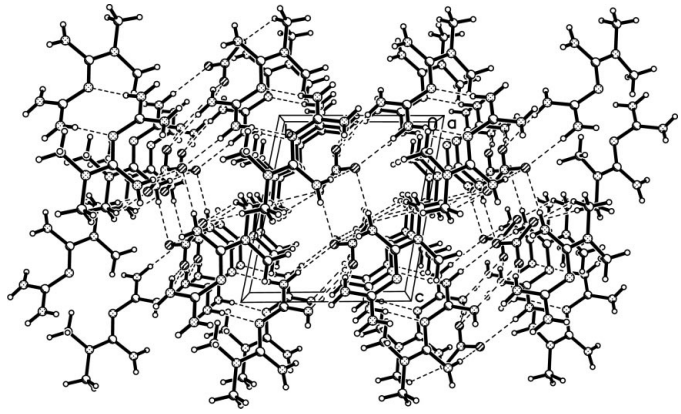


Figure 3
A packing diagram of the structure of the title compound.

Data collection

Bruker SMART 1K CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.965, T_{max} = 0.988
1875 measured reflections
1559 independent reflections
1181 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°
$h = -5 \rightarrow 8$
$k = -7 \rightarrow 8$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.129
S = 1.01
1559 reflections
155 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.327 (3)	C2—N5	1.324 (3)
C1—N2	1.335 (3)	C2—N3	1.331 (3)
C1—N3	1.343 (3)	C2—N4	1.336 (3)
N1—C1—N2	118.7 (2)	N3—C2—N4	124.1 (2)
N1—C1—N3	119.4 (2)	C1—N1—C4	121.3 (2)
N2—C1—N3	121.6 (2)	C1—N1—C3	121.2 (2)
N5—C2—N3	118.1 (2)	C2—N3—C1	121.5 (2)
N5—C2—N4	117.8 (2)		
N2—C1—N1—C4	−164.5 (3)	N5—C2—N3—C1	−159.1 (2)
N3—C1—N1—C4	21.6 (4)	N4—C2—N3—C1	23.8 (4)
N2—C1—N1—C3	6.3 (4)	N1—C1—N3—C2	−147.5 (2)
N3—C1—N1—C3	−167.6 (2)	N2—C1—N3—C2	38.8 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H52···O1^{i}	0.85 (3)	2.11 (3)	2.962 (3)	175 (3)
N5—H51···N3^{ii}	0.91 (3)	2.10 (3)	3.003 (3)	171 (3)
N4—H41···O3^{i}	0.84 (3)	2.12 (3)	2.932 (3)	162 (3)
N2—H21···O1	0.87 (3)	2.13 (3)	2.980 (3)	166 (3)
N2—H22···O1^{iii}	0.89 (3)	2.14 (3)	3.009 (3)	168 (3)
N4—H42···O3^{iv}	0.90 (3)	2.10 (3)	2.936 (3)	153 (3)
N4—H42···N2	0.90 (3)	2.58 (3)	2.913 (3)	103 (2)

Symmetry codes: (i) $x, y, 1+z$; (ii) $1-x, -y, 2-z$; (iii) $1-x, 1-y, 1-z$; (iv) $-x, 1-y, 1-z$.

H atoms attached to C and N atoms were located in a difference Fourier map and refined with a global U_{iso} value. The C—H and N—H distances are in the ranges 0.93 (3)–0.98 (3) and 0.84 (3)–0.91 (3) Å, respectively.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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