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(*E*)-1-[(2-Methoxyphenyl)methyleneamino]-guanidinium chloride

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Key indicators

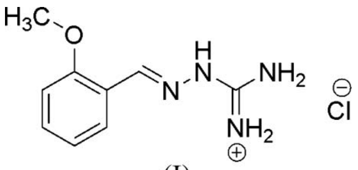
Single-crystal X-ray study
T = 90 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.119
Data-to-parameter ratio = 18.4

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

In the structure of the title compound, $C_9H_{13}ClN_4O$, the guanidinium group is nearly coplanar with the 2-methoxyphenyl ring, C═N having *E* geometry. The chloride ions are involved in intermolecular hydrogen bonds with the H atoms of the aminoguanidinium ion.

Comment

The basic guanidine unit is part of many biologically active compounds. Guanylhydrazones derived from arylaldehydes and aminoguanidine also contain the guanidine unit and they exhibit a wide range of biological activities. An important representative of this class of compounds is the centrally acting antihypertensive agent guanabenz (2,6-dichlorobenzylideneaminoguanidine) in the treatment of high blood pressure (Baum, *et al.*, 1969). In view of their biological activities, we have synthesized a series of guanylhydrazones. The title compound, (I), was synthesized by the condensation reaction of 2-methoxybenzaldehyde with aminoguanidine hydrochloride under reflux in methanol, to afford a single geometrical isomer. The present X-ray crystallographic determination was carried out in order to confirm the double-bond geometry, and to obtain more detailed information on the conformation of the cation.



(I)

The molecular structure and atom-numbering scheme of (I) are shown in Fig. 1, and selected bond lengths and angles are listed in Table 1. In the title compound, the C7═N1 double bond connecting the guanidinium group with the 2-methoxyphenyl ring system has *E* geometry. The guanidinium group is nearly coplanar with the 2-methoxyphenyl ring, facilitating partial conjugation between them as indicated by shortening of the C6—C7 bond in comparison with the standard value for a $C_{ar}—Csp^2$ single bond [1.470 (15) Å; Allen *et al*, 1987].

The observed bond lengths C1—O1 and C9—O1 are comparable to the values of aromatic methoxy bonds (Domiano *et al.*, 1979), and there is an asymmetry of the angles around atom C1, as is typical of anisoles.

In the crystal structure, each chloride ion interacts *via* hydrogen bonding with three N—H bonds of the guanidinium ion (Table 2).

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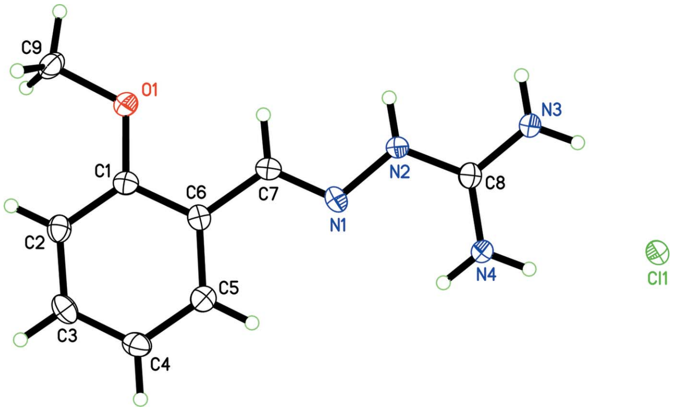

Figure 1
A view of (I) with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius.

Experimental

A mixture of 2-methoxybenzaldehyde (204 mg, 1.5 mmol) and aminoguanidine hydrochloride (110 mg, 1 mmol) was taken up in methanol (15 ml) and the mixture was refluxed for 8 h. After evaporation of the solvent, the residue was stirred in chloroform and filtered to remove unreacted 2-methoxybenzaldehyde in the filtrate. The solid product was dried and recrystallized from methanol.

Crystal data

$C_9H_{13}N_4O^+ \cdot Cl^-$
$M_r = 228.68$
Monoclinic, $P2_1/c$
$a = 6.9550$ (1) Å
$b = 10.9089$ (2) Å
$c = 14.5481$ (3) Å
$\beta = 92.034$ (1)°
$V = 1103.09$ (3) Å^3
$Z = 4$
$D_x = 1.377$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.33$ mm^{-1}
$T = 90.0$ (2) K
Plate, colourless
0.30 × 0.20 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.908$, $T_{\max} = 0.978$
4915 measured reflections
2526 independent reflections
1837 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.119$
$S = 1.05$
2526 reflections
137 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.3498P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.367 (2)	C8—N3	1.330 (2)
C6—C7	1.461 (3)	C8—N2	1.335 (2)
C7—N1	1.275 (2)	C9—O1	1.434 (2)
C8—N4	1.324 (2)	N1—N2	1.381 (2)
O1—C1—C2	123.84 (18)	O1—C1—C6	115.80 (17)
O1—C1—C2—C3	179.52 (17)	C1—C6—C7—N1	−176.25 (18)
C5—C6—C7—N1	5.8 (3)	C7—N1—N2—C8	178.96 (17)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···Cl1$^{\rm i}$	0.88	2.39	3.1861 (17)	150
N3—H3*A*···Cl1	0.88	2.55	3.3035 (17)	144
N3—H3*B*···Cl1$^{\rm i}$	0.88	2.46	3.2435 (17)	149
N4—H4*A*···Cl1	0.88	2.41	3.1964 (18)	149
N4—H4*B*···Cl1$^{\rm ii}$	0.88	2.62	3.3035 (17)	135

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

H atoms were positioned geometrically and treated as riding, with C—H = 0.95 or 0.98 Å for aromatic and methyl atoms, respectively, N—H = 0.88 Å, and $U_{\rm iso}({\rm H}) = xU_{\rm eq}$(C,N), where $x = 1.5$ for methyl H atoms and $x = 1.2$ for all other H atoms.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*–SMN (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1995); software used to prepare material for publication: *SHELX97* and local procedures.

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