

dihedral angle of $0.3(2)^\circ$, whereas phenyl ring C11–C16 inclusive is inclined at an angle of $9.1(2)^\circ$ to this skeleton. In the molecule 2 both phenyl rings are inclined at an angle of $5.5(2)^\circ$. The dihedral angles between the phenyl-ring planes are $9.1(1)$ and 0° for molecules 1 and 2 respectively.

In the unit cell there are no intermolecular contacts which are significantly less than the sum of the respective van der Waals radii.

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N-Adamant-1-yl-*N'*-(2-iodophenyl)guanidinium Chloride

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Abstract. *N*-(2-Iodophenyl)-*N'*-(tricyclo[3.3.1.1^{3,7}]-decan-1-yl)guanidinium chloride, $C_{17}H_{23}ClIN_3$, $M_r = 431.75$, monoclinic, $P2_1/c$, $a = 11.696(5)$, $b = 20.120(4)$, $c = 7.754(2)$ Å, $\beta = 95.26(3)^\circ$, $V = 1817(1)$ Å³, $Z = 4$, $D_x = 1.578$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.89$ mm⁻¹, $F(000) = 864$, $T = 295$ K, $R = 0.071$ for 1169 observed [$I \geq 3\sigma(I)$] reflections. Both the phenyl and the adamantyl groups lie *syn* to the unsubstituted nitrogen of the guanidinium ion. The plane of the phenyl ring is nearly perpendicular to that of the guanidinium group.

Introduction. *N,N'*-Di-*ortho*-tolylguanidine and its congeners are highly potent and selective ligands for the haloperidol-sensitive sigma (σ) receptor (Weber, Sonders, Quarum, McLean, Pou & Keana, 1986; Largent, Wikström, Gundlach & Snyder, 1987). As such, they are of considerable current interest as potential atypical antipsychotic agents, devoid of the severe and long-lasting side effects of currently available antipsychotic neuroleptic drugs (Snyder & Largent, 1989; Largent, Wikström, Snowman & Snyder, 1988; Deutsch, Weizman, Goldman & Morihisa, 1988). In addition, certain *N,N'*-

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diarylguanidines are also potent ligands for the *N*-methyl-D-aspartate/PCP (NMDA/PCP) receptor (Keana *et al.*, 1989). Such compounds have been shown to have potent neuroprotective properties against glutamate-induced neuronal cell death (Cavalheiro, Lehmann & Turski, 1988; Olney, Labruyere & Price, 1989). They hold promise as an entirely new therapeutic tool for the treatment of neurodegenerative symptoms of stroke or heart attack (Choi, 1988).

We have examined the structure–affinity relationships of a large series of *N,N'*-disubstituted guanidines (Scherz *et al.*, 1990). Briefly, *N,N'*-diarylguanidines bearing either iodine or small alkyl substituents (*e.g.* CH₃, C₂H₅) in the *ortho* or *meta* position bind tightly to the σ receptor and the NMDA/PCP receptor. Replacement of one of the aryl rings with a small saturated carbocycle such as cyclohexyl, norbornyl, or adamantyl completely abolishes binding to the NMDA/PCP receptor, and increases significantly binding to the σ receptor.

We sought to identify the structural requirements of *N,N'*-disubstituted guanidines for potent, selective binding to the σ receptor. To this end we undertook the X-ray crystallographic analysis of one of the most potent and selective σ receptor ligands available from our studies, *N*-adamant-1-yl-*N'*-2-iodo-

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phenylguanidine hydrochloride. Our results are reported here.

Experimental. The compound was synthesized by reaction of adamant-1-yl cyanamide and 2-iodoaniline hydrochloride in hot chlorobenzene, as previously described (Scherz *et al.*, 1990; Keana *et al.*, 1989). Colorless prisms were obtained by crystallization from EtOH/Et₂O. The crystals were colorless air-stable prisms of moderate diffracting quality. All crystals examined diffracted weakly, and twinning was prevalent. Data were collected from the major twin component of one crystal for which 19 out of 25 centered reflections in the range $11 \leq 2\theta \leq 14^\circ$ could be satisfactorily indexed and a unit cell deduced. Improved cell parameters were later obtained from 25 reflections in the range $32 \leq 2\theta \leq 40^\circ$.

Fiber-mounted crystal, $0.10 \times 0.10 \times 0.25$ mm; Rigaku AFC6R diffractometer, graphite monochromator; ω - 2θ scans; scan speed $16^\circ \text{ min}^{-1}$ on ω ; scan width $(1.47 + 0.30 \tan \theta)^\circ$; three reference reflections every 150, no systematic change; $2\theta_{\text{max}} 50^\circ$; $-13 \leq h \leq 13$, $-23 \leq k \leq 0$, $0 \leq l \leq 8$; 2956 reflections measured, 2762 independent ($R_{\text{int}} 0.053$), 1169 with $I \geq 3\sigma(I)$ used in refinement.

The iodine atom was located from the Patterson function; structure expanded by use of *DIRDIF* (Beurskens, 1984); empirical absorption correction *DIFABS* (Walker & Stuart, 1983) was applied after isotropic refinement of non-H atoms (min., max. correction factors 0.630, 1.485). Full-matrix least-squares refinement converged at $R 0.071$, $wR 0.080$, $S 2.01$; anisotropic thermal parameters for all non-H atoms, H atoms included at calculated positions, $U_{\text{H}} = 1.2U_{\text{C}}$; 199 parameters; function minimized

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters (\AA^2) for the non-H atoms

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} [*]
I(1)	5483 (1)	5971.4 (7)	1917 (2)	4.38 (6)
Cl(1)	2642 (4)	5276 (2)	7187 (6)	3.9 (2)
N(1)	2775 (10)	6110 (6)	602 (18)	2.9 (6)
N(2)	2042 (11)	5092 (6)	1099 (17)	3.4 (7)
N(3)	2485 (13)	5829 (6)	3418 (20)	3.8 (7)
C(1)	4404 (12)	6805 (8)	1711 (21)	2.7 (8)
C(2)	3260 (13)	6750 (8)	1111 (21)	2.9 (8)
C(3)	2564 (14)	7302 (8)	1024 (26)	4.0 (11)
C(4)	3010 (16)	7924 (9)	1464 (25)	3.8 (10)
C(5)	4118 (17)	7987 (9)	1956 (29)	4.9 (12)
C(6)	4846 (14)	7435 (10)	2059 (25)	4.1 (11)
C(7)	2457 (12)	5662 (8)	1773 (26)	3.0 (8)
C(8)	1708 (12)	4492 (7)	2022 (21)	2.5 (7)
C(9)	2746 (12)	4169 (8)	3021 (22)	3.2 (8)
C(10)	739 (13)	4598 (8)	3121 (24)	3.0 (8)
C(11)	1300 (12)	4003 (9)	482 (20)	2.9 (7)
C(12)	391 (14)	3925 (8)	3850 (23)	3.4 (8)
C(13)	2382 (15)	3508 (8)	3767 (26)	3.9 (10)
C(14)	922 (14)	3343 (7)	1285 (26)	3.6 (9)
C(15)	1970 (15)	3033 (8)	2311 (31)	5.2 (13)
C(16)	-44 (14)	3460 (9)	2444 (29)	3.6 (8)
C(17)	1430 (16)	3634 (8)	4935 (25)	4.0 (10)

$$*B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

$\sum w(|F_o| - |F_c|)^2$; $w = 1/\sigma^2(F)$; $\sigma(F)$ from counting statistics, $p = 0.03$; max. shift/e.s.d. 0.08 in last cycle; max., min. $\Delta\rho$ 1.9 (near iodine), -0.9 e \AA^{-3} in final difference map. No evidence for solvent of crystallization was found. The *TEXSAN* program suite (Molecular Structure Corporation, 1987), with atomic scattering factors taken from Stewart, Davidson & Simpson (1965) and *International Tables for X-ray Crystallography* (1974 Vol. IV, pp. 71, 148), was used in all calculations.

Discussion. The numbering of the atoms is shown in Fig. 1. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1; bond lengths and inter-bond angles are given in Table 2.* The Cl...N distances (Table 2) are consistent with the presence of hydrogen bonds which link cations and anions into chains extending parallel to the *c* axis. The three C—N bonds in the N₃C group (Table 2) do not differ significantly in length from one another. The dihedral angle between the mean planes of the N₃C and phenyl groups is 85°. Both the phenyl and adamantyl groups lie *syn* to the unsubstituted N atom [N(3)].

A low resolution ($R = 0.15$) structure determination from a twinned crystal of *N*-adamant-1-yl-*N'*-2-iodophenylguanidine free base (Weakley, unpub-

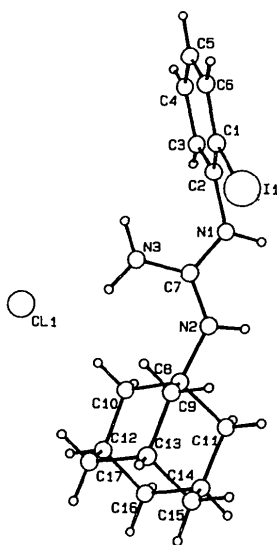


Fig. 1. A view of the asymmetric unit approximately normal to the guanidinium mean plane.

* Lists of observed and calculated structure factors, anisotropic thermal parameters, calculated H-atom coordinates and isotropic thermal parameters, torsion angles and mean-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53087 (20 pp.). Copies may be obtained through The Technical Editor, *International Union of Crystallography*, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å), inter-bond angles (°) and probable hydrogen bonds (Å)

I(1)—C(1)	2.10 (2)	C(8)—C(9)	1.53 (2)
N(1)—C(2)	1.45 (2)	C(8)—C(10)	1.49 (2)
N(1)—C(7)	1.36 (2)	C(8)—C(11)	1.59 (2)
N(2)—C(7)	1.33 (2)	C(9)—C(13)	1.53 (2)
N(2)—C(8)	1.47 (2)	C(10)—C(12)	1.54 (2)
N(3)—C(7)	1.32 (2)	C(11)—C(14)	1.55 (2)
C(1)—C(2)	1.38 (2)	C(12)—C(16)	1.49 (2)
C(1)—C(6)	1.39 (2)	C(12)—C(17)	1.53 (2)
C(2)—C(3)	1.38 (2)	C(13)—C(15)	1.52 (3)
C(3)—C(4)	1.39 (2)	C(13)—C(17)	1.52 (3)
C(4)—C(5)	1.32 (2)	C(14)—C(15)	1.53 (2)
C(5)—C(6)	1.40 (3)	C(14)—C(16)	1.53 (2)
C(2)—N(1)—C(7)	122 (1)	C(9)—C(8)—C(10)	112 (1)
C(7)—N(2)—C(8)	128 (1)	C(9)—C(8)—C(11)	107 (1)
I(1)—C(1)—C(2)	121 (1)	C(10)—C(8)—C(11)	109 (1)
I(1)—C(1)—C(6)	120 (1)	C(8)—C(9)—C(13)	109 (1)
C(2)—C(1)—C(6)	118 (1)	C(8)—C(10)—C(12)	109 (1)
N(1)—C(2)—C(1)	120 (1)	C(8)—C(11)—C(14)	108 (1)
N(1)—C(2)—C(3)	119 (1)	C(10)—C(12)—C(16)	112 (2)
C(1)—C(2)—C(3)	120 (1)	C(10)—C(12)—C(17)	108 (1)
C(2)—C(3)—C(4)	121 (2)	C(16)—C(12)—C(17)	111 (1)
C(3)—C(4)—C(5)	120 (2)	C(9)—C(13)—C(15)	110 (2)
C(4)—C(5)—C(6)	121 (2)	C(9)—C(13)—C(17)	109 (1)
C(1)—C(6)—C(5)	120 (2)	C(15)—C(13)—C(17)	110 (2)
N(1)—C(7)—N(2)	115 (2)	C(11)—C(14)—C(15)	108 (1)
N(1)—C(7)—N(3)	120 (2)	C(11)—C(14)—C(16)	111 (1)
N(2)—C(7)—N(3)	125 (2)	C(15)—C(14)—C(16)	111 (2)
N(2)—C(8)—C(9)	111 (1)	C(13)—C(15)—C(14)	108 (1)
N(2)—C(8)—C(10)	114 (1)	C(12)—C(16)—C(14)	108 (1)
N(2)—C(8)—C(11)	103 (1)	C(12)—C(17)—C(13)	109 (2)
Cl(1)⋯N(1 ⁱ)	3.13 (2)	Cl(1)⋯N(3)	3.12 (2)
Cl(1)⋯N(2 ⁱ)	3.20 (2)		

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

lished observation) has shown that (i) the N₃C group and attached C(phenyl) and C(adamantyl) atoms are nearly coplanar; (ii) the phenyl ring is at nearly 90° to the N₃C plane and (iii) the phenyl group is *syn*, and the adamantyl group is *anti*, to the terminal N atom.

A molecular geometry similar to that of the free base has been reported in the crystal structure of *N,N'*-bis(2-methylphenyl)guanidine (DTG) free base (Brown & Gash, 1984). The dihedral angle between

the N₃C and the phenyl groups was found to be 106°. The two phenyl rings of this compound were found to lie *syn* and *anti*, respectively, to the unsubstituted nitrogen.

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Crystal Chemistry of *cyclo*-Hexaphosphates. VII. Structure of Tris(ethylene-diammonium) *cyclo*-Hexaphosphate Tellurate Dihydrate

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Abstract. (C₂H₁₀N₂)₃[P₆O₁₈][Te(OH)₆]₂·2H₂O, *M_r* = 1151.458, triclinic, *PI*, *a* = 10.945 (3), *b* = 11.252 (3), *c* = 8.042 (7) Å, α = 90.90 (5), β = 92.97 (5), γ =

116.82 (5)°, *V* = 881.8 Å³, *Z* = 1, *D_x* = 2.168 Mg m⁻³, λ (Ag *K*α) = 0.5608 Å, μ = 1.090 mm⁻¹, *F*(000) = 574, room temperature, final *R* = 0.022 for