

Ya-Ming Wu, Guang-Liang Song,
Ke-Le Wang, Jing Heng and
Hong-Jun Zhu*Department of Applied Chemistry, College of
Science, Nanjing University of Technology,
Nanjing 210009, People's Republic of China

Correspondence e-mail: zhuhj@njut.edu.cn

Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.049

wR factor = 0.139

Data-to-parameter ratio = 19.8

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

Hexaethylguanidinium chloride

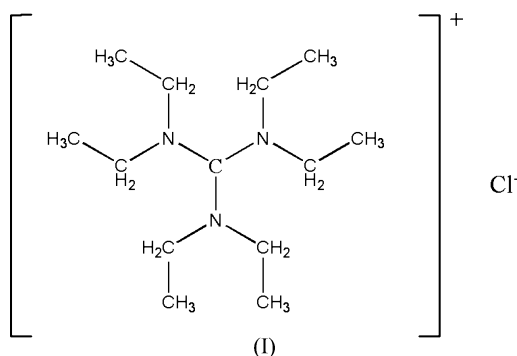
The title compound, $\text{C}_{13}\text{H}_{30}\text{N}_3^+\cdot\text{Cl}^-$, is a phase-transfer catalyst. The geometry at the central C atom of the guanidinium cation, which lies on a twofold rotation axis, is almost ideal trigonal planar, with N—C—N angles of 119.8 (2) and 120.08 (11)°. The chloride anion also lies on a twofold rotation axis.

Received 12 June 2006

Accepted 21 July 2006

Comment

Hexaalkylguanidinium salts are widely used as phase-transfer catalysts due to their exceptional stability at high temperature (Caringi & Faler, 1999). In this paper, we report the crystal structure of the title chloride salt, (I).



The structure of (I) is shown in Fig. 1, with selected geometric parameters listed in Table 1. In the structure of (I), the guanidinium cation is located on a twofold rotation axis, as is also the chloride anion. The geometry at the central C atom is almost ideal trigonal planar (Table 1). Similar geometric parameters are observed for tricyclohexylguanidinium chloride (Cai & Hu, 2006).

Experimental

Compound (I) was prepared by the reported procedure of Caringi & Faler (1999). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of solvent from a solution of (I) (2.0 g) in a mixture of equal volumes of heptane and ethyl acetate (250 ml) under anhydrous conditions.

Crystal data

$\text{C}_{13}\text{H}_{30}\text{N}_3^+\cdot\text{Cl}^-$
 $M_r = 262.86$
 Monoclinic, $C2/c$
 $a = 7.3110 (15) \text{ \AA}$
 $b = 16.274 (3) \text{ \AA}$
 $c = 13.802 (3) \text{ \AA}$
 $\beta = 97.77 (3)^\circ$
 $V = 1627.1 (6) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.077 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.22 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Block, white
 $0.40 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Enraf–Nonius CAD-4
diffractometer
 $\omega/2\theta$ scans
Absorption correction: ψ scan
(North *et al.*, 1968)
 $T_{\min} = 0.916$, $T_{\max} = 0.957$
1710 measured reflections

1582 independent reflections
1191 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.013$
 $\theta_{\max} = 26.0^\circ$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.139$
 $S = 1.03$
1582 reflections
80 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.96P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e } \text{Å}^{-3}$
Extinction correction: *SHELXL97*
(Sheldrick, 1997)
Extinction coefficient: 0.018 (2)

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-----------------------|-------------|------------------------|-------------|
| N1–C1 | 1.336 (3) | N2–C5 | 1.467 (2) |
| N1–C6 | 1.470 (2) | N2–C7 | 1.469 (2) |
| N2–C1 | 1.3393 (19) | | |
| C1–N1–C6 | 120.76 (11) | N1–C1–N2 | 120.08 (11) |
| C6–N1–C6 ⁱ | 118.5 (2) | N2 ⁱ –C1–N2 | 119.8 (2) |
| C1–N2–C5 | 120.81 (15) | N2–C5–C2 | 112.45 (18) |
| C1–N2–C7 | 120.37 (15) | N2–C7–C4 | 112.72 (17) |
| C5–N2–C7 | 118.82 (15) | | |

Symmetry code: (i) $-x + 1, y, -z + \frac{3}{2}$.

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------|-------|-------------|-------------|---------------|
| C5–H5B \cdots N1 | 0.97 | 2.52 | 2.872 (2) | 101 |
| C6–H6B \cdots N2 | 0.97 | 2.52 | 2.863 (3) | 101 |
| C7–H7B \cdots N2 ⁱ | 0.97 | 2.52 | 2.858 (3) | 101 |

Symmetry code: (i) $-x + 1, y, -z + \frac{3}{2}$.

All H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C–H distances of 0.96–0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97*

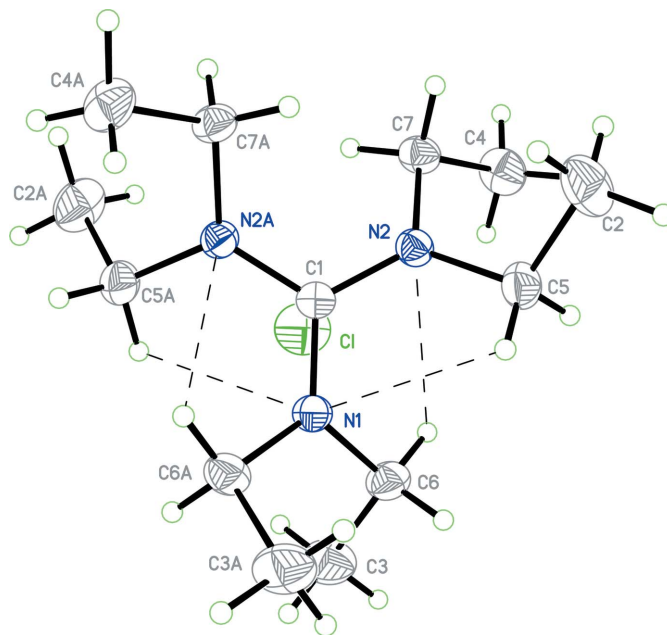


Figure 1

The structure of (I). Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate weak intramolecular C–H \cdots N interactions. [Symmetry code: (A) $1 - x, y, \frac{3}{2} - z$.]

(Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL97*.

The authors thank the Centre for Testing and Analysis, Nanjing University, for support.

References

- Bruker (2000). *XSCANS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cai, X.-Q. & Hu, M.-L. (2006). *Acta Cryst.* **E62**, o1260–o1261.
- Caringi, J. J. & Faler, G. R. (1999). General Electric, USA. US Patent Appl. US 5872294 A1.
- Enraf–Nonius (1985). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.