Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1224). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Enraf–Nonius (1977). *CAD-4 Operations Manual.* Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.
Hagen, K., Hedberg, L. & Hedberg, K. (1982). *J. Phys. Chem.* **86**, 117–121.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kaftory, M. (1983). *The Chemistry of Functional Groups*, Supplement D, edited by S. Patai & Z. Rappoport, p. 1241. Chichester: Wiley.
Kaftory, M., Apeloig, Y. & Rappoport, Z. (1985). *J. Chem. Soc. Perkin Trans.* 2, pp. 29–37.
Luo, Y., Barton, R. J., Robertson, B. E., Lee, C. C. & Wanigasekera, D. (1987). *Acta Cryst.* C**43**, 73–76.
MacKenzie, R. K., MacNicol, D. D., Mills, H. H., Raphael, R. A., Wilson, F. B. & Zabkiewicz, J. A. (1972). *J. Chem. Soc. Perkin Trans.* 2, pp. 1632–1638.
Mugnoli, A. & Simonetta, M. (1976). *J. Chem. Soc. Perkin Trans.* 2, pp. 822–825.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Wijsman, G. W., de Wolf, W. H., Bickelhaupt, F., Kooijman, H. & Spek, A. L. (1992). *J. Am. Chem. Soc.* **114**, 9191–9192.
Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

---

*Acta Cryst.* (1997). C**53**, 86–88

# 1,2-Diphenyl-3-benzylguanidine Hydrochloride

L. Sudha,[a] K. Subramanian,[a] J. Senthil Selvan,[a] Thomas Steiner,[b] Gertraud Koellner,[b] K. Ramdas[c] and N. Srinivasan[c]

[a]*Department of Physics, College of Engineering, Anna University, Madras 600 025, India,* [b]*Institut für Kristallographie, Freie Universität Berlin, Takustrasse 6, D-14195 Berlin, Germany, and* [c]*Centre for Agrochemical Research, SPIC Science Foundation, Madras 600 032, India. E-mail: steiner@chemie.fu-berlin.de*

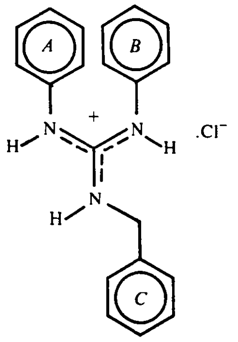
(*Received 18 June 1996; accepted 25 September 1996*)

## Abstract

In the title compound, 3-benzyl-1,2-diphenylguanidinium chloride $C_{20}H_{20}N_3^+.Cl^-$, the guanidine moiety is protonated. Steric strain due to the phenyl substituents leads to distortions of the guanidine skeleton. All three guanidinium N—H groups donate hydrogen bonds to symmetry-related chloride ions.

## Comment

The conformation of the title compound, (1), as observed in the crystal structure is shown in Fig. 1. Since (1) has been crystallized as a hydrochloride, the guanidine moiety is protonated (*i.e.* N7, N9 and N10 all carry one H atom each). The three guanidinium C—N bond lengths are almost identical [1.337 (2), 1.337 (2) and 1.332 (2) Å for C8—N7, C8—N9 and C8—N10, respectively]; this is in agreement with the previously reported structure of a related substituted guanidinium salt (Sudha *et al.*, 1996) and contrasts with related uncharged molecules, where the guanidine C—N bonds are unequal, with a defined C═N double bond (Sudha *et al.*, 1995*a*,*b*).



(1)

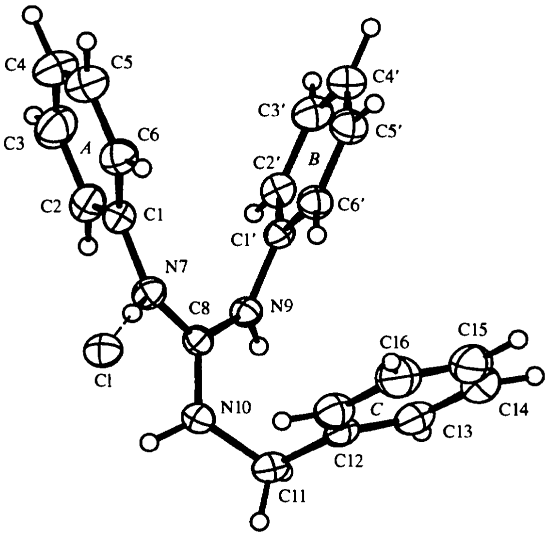


Fig. 1. The molecular structure and atomic labelling of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

*Acta Crystallographica Section C*
ISSN 0108-2701 © 1997

Both phenyl rings are inclined with respect to the guanidinium plane, but not exactly perpendicular to it [the dihedral angles with respect to the guanidinium group are 60.3 (1)° for ring *A* and 65.7 (1)° for ring *B*]; the dihedral angle between the phenyl rings is 40.8 (1)°. Steric conflict of the phenyl substituents leads to opening of the angles at the N7, C8 and N9 atoms, and a significant distortion of the guanidinium moiety [C8—N7—C1 126.5 (1), N7—C8—N9 122.3 (1) and C8—N9—C1′ 124.8 (1)°]. This means that the pivot atoms of the phenyl groups, C1 and C1′, are 2.968 (2) Å apart. The benzyl group is twisted out of the guanidinium plane.

The crystal packing scheme is shown in Fig. 2. The three guanidinium N—H groups donate N—H···Cl⁻ hydrogen bonds to symmetry-related chloride ions: N7—H···Cl⁻, with N7···Cl⁻ 3.182 (1) Å, N9—H···Cl⁻(1 + *x*, *y*, *z*), with N9···Cl⁻ 3.194 (2) Å, and N10—H···Cl⁻(−*x*, 1 − *y*, 1 − *z*), with N10···Cl⁻ 3.230 (2) Å.

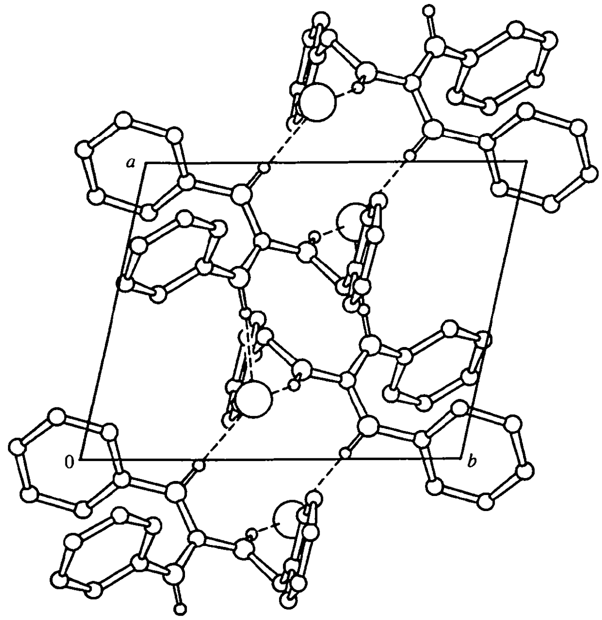


Fig. 2. The crystal packing in a projection along the *z* axis. H atoms bonded to C atoms have been omitted for clarity. N—H bonds are drawn filled, while N—H···Cl⁻ hydrogen bonds are shown as dashed lines.

## Experimental

The title compound, (1), was synthesized at and supplied by the SPIC Science Foundation, Madras, India. The synthesis involves the desulfurization of 1,3-diaryl thiourea in the presence of an appropriate amine (Ramadas & Srinivasan, 1995). Crystals were grown as the hydrochloride from MeOH.

### *Crystal data*

$C_{20}H_{20}N_3^+.Cl^-$
$M_r = 337.84$
Triclinic
$P\overline{1}$
$a = 7.9656\,(11)$ Å
$b = 9.7996\,(10)$ Å
$c = 12.036\,(2)$ Å
$\alpha = 86.45\,(2)°$
$\beta = 80.28\,(2)°$
$\gamma = 77.45\,(2)°$
$V = 903.7\,(2)$ Å$^3$
$Z = 2$
$D_x = 1.242$ Mg m$^{-3}$
$D_m$ not measured

Cu *Kα* radiation
$\lambda = 1.54176$ Å
Cell parameters from 25 reflections
$\theta = 8.1–24.9°$
$\mu = 1.897$ mm$^{-1}$
$T = 293\,(2)$ K
Prism
0.55 × 0.25 × 0.15 mm
Colourless

### *Data collection*

Enraf–Nonius Turbo-CAD-4 diffractometer
$\omega$–$2\theta$ scans
Absorption correction: $\psi$ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.682$, $T_{max} = 0.752$
3326 measured reflections
2661 independent reflections

2498 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.0138$
$\theta_{max} = 59.86°$
$h = -1 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$
25 standard reflections
frequency: 30 min
intensity decay: 5.7%

### *Refinement*

Refinement on $F^2$
$R(F) = 0.0353$
$wR(F^2) = 0.0929$
$S = 1.078$
2661 reflections
238 parameters
Only H-atom *U*'s refined
$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2 + 0.2788P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.001$

$\Delta\rho_{max} = 0.229$ e Å$^{-3}$
$\Delta\rho_{min} = -0.180$ e Å$^{-3}$
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.064 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å$^2$)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|
| Cl | −0.20341 (5) | 0.58493 (4) | 0.62494 (4) | 0.0530 (2) |
| C1 | 0.0647 (2) | 0.8833 (2) | 0.64390 (14) | 0.0466 (4) |
| C2 | 0.1693 (3) | 0.9663 (2) | 0.5841 (2) | 0.0583 (5) |
| C3 | 0.1135 (4) | 1.1122 (2) | 0.5910 (2) | 0.0797 (8) |
| C4 | −0.0442 (4) | 1.1694 (3) | 0.6551 (2) | 0.0870 (8) |
| C5 | −0.1457 (4) | 1.0849 (3) | 0.7130 (2) | 0.0855 (7) |
| C6 | −0.0929 (3) | 0.9425 (2) | 0.7086 (2) | 0.0657 (6) |
| C1′ | 0.3470 (2) | 0.7888 (2) | 0.78119 (14) | 0.0407 (4) |
| C2′ | 0.4398 (3) | 0.8931 (2) | 0.7772 (2) | 0.0531 (5) |
| C3′ | 0.4025 (3) | 0.9845 (2) | 0.8670 (2) | 0.0672 (6) |
| C4′ | 0.2769 (3) | 0.9709 (2) | 0.9568 (2) | 0.0666 (6) |
| C5′ | 0.1868 (3) | 0.8661 (2) | 0.9604 (2) | 0.0634 (5) |
| C6′ | 0.2221 (2) | 0.7740 (2) | 0.87266 (15) | 0.0513 (5) |
| N7 | 0.1093 (2) | 0.73502 (14) | 0.63669 (12) | 0.0452 (4) |
| C8 | 0.2658 (2) | 0.6532 (2) | 0.64188 (13) | 0.0393 (4) |
| N9 | 0.3853 (2) | 0.69343 (14) | 0.69067 (11) | 0.0413 (3) |
| N10 | 0.3022 (2) | 0.52695 (15) | 0.59742 (12) | 0.0476 (4) |
| C11 | 0.4173 (2) | 0.4070 (2) | 0.6437 (2) | 0.0512 (5) |
| C12 | 0.3605 (2) | 0.3858 (2) | 0.7681 (2) | 0.0484 (4) |
| C13 | 0.4750 (3) | 0.3764 (2) | 0.8436 (2) | 0.0666 (6) |
| C14 | 0.4158 (5) | 0.3583 (2) | 0.9596 (2) | 0.0893 (9) |

| | | | | |
|---|---|---|---|---|
| C15 | 0.2455 (5) | 0.3537 (3) | 0.9975 (2) | 0.0960 (9) |
| C16 | 0.1325 (4) | 0.3643 (3) | 0.9231 (2) | 0.0873 (8) |
| C17 | 0.1883 (3) | 0.3798 (2) | 0.8095 (2) | 0.0642 (5) |

Table 2. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N7 | 1.423 (2) | C8—N9 | 1.337 (2) |
| C1′—N9 | 1.429 (2) | N10—C11 | 1.468 (2) |
| N7—C8 | 1.337 (2) | C11—C12 | 1.505 (3) |
| C8—N10 | 1.332 (2) | | |
| C8—N7—C1 | 126.5 (1) | C8—N9—C1′ | 124.8 (1) |
| N10—C8—N7 | 118.4 (1) | C8—N10—C11 | 122.2 (1) |
| N10—C8—N9 | 119.3 (2) | N10—C11—C12 | 111.6 (1) |
| N7—C8—N9 | 122.3 (1) | | |
| C8—N7—C1—C2 | 45.7 (2) | N7—C8—N10—C11 | −148.1 (2) |
| C8—N9—C1′—C2′ | −135.4 (2) | N9—C8—N7—C1 | 22.6 (3) |
| C8—N10—C11—C12 | 52.8 (2) | N9—C8—N10—C11 | 31.5 (3) |
| N10—C11—C12—C13 | −127.1 (2) | N10—C8—N7—C1 | −157.9 (2) |
| N7—C8—N9—C1′ | 28.9 (2) | N10—C8—N9—C1′ | −150.6 (2) |

All H atoms were refined in the riding model, with bond lengths of C—H = 1.00 and N—H = 0.95 Å and *U* allowed to vary (*SHELXL*93; Sheldrick, 1993). The *U* values of the H atoms bonded to N atoms refined to values between 0.06 and 0.08 Å$^2$, indicating that these atoms were treated realistically.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

KS thanks the Deutscher Akademischer Austauschdienst for a grant to stay in Berlin. LS and NS wish to thank CSIR, New Delhi, India, for financial support. The authors thank Professor W. Saenger (Berlin) for making available the X-ray equipment.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Enraf–Nonius (1989). *CAD*-4 *Software*. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Ramadas, K. & Srinivasan, N. (1995). *Tetrahedron Lett.* **36**, 2841–2844.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Sudha, L., Senthil Selvan, J., Subramanian, K., Steiner, Th., Koellner, G., Srinivasan, N. & Ramdas, K. (1995*a*). *Acta Cryst.* C**51**, 2323–2325.

Sudha, L., Senthil Selvan, J., Subramanian, K., Steiner, Th., Koellner, G., Srinivasan, N. & Ramdas, K. (1995*b*). *Acta Cryst.* C**51**, 2329–2331.

Sudha, L., Subramanian, K., Senthil Selvan, J., Steiner, Th., Koellner, G., Ramdas, K. & Srinivasan, N. (1996). *Acta Cryst.* C**52**, 3238–3240.

---

*Acta Cryst.* (1997). C**53**, 88–90

# 1,2-Bis(2,6-diethylphenyl)-3,3-(oxydiethyl)guanidine

L. Sudha,[a] K. Subramanian,[a] J. Senthil Selvan,[a] Thomas Steiner,[b] Gertraud Koellner,[b] K. Ramdas[c] and N. Srinivasan[c]

[a]*Department of Physics, College of Engineering, Anna University, Madras 600 025, India,* [b]*Institut für Kristallographie, Freie Universität Berlin, Takustrasse 6, D-14195 Berlin, Germany, and* [c]*Centre for Agrochemical Research, SPIC Science Foundation, Madras 600 032, India. E-mail: steiner@chemie.fu-berlin.de*

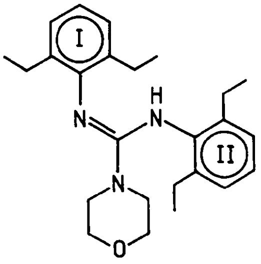
(*Received 1 July 1996; accepted 9 September 1996*)

## Abstract

The phenyl groups of the title compound, $N^1,N^2$-bis(2,6-diethylphenyl)morpholine-4-carboxamidine, $C_{25}H_{35}N_3O$, are oriented roughly perpendicular to the guanidine plane. The peripheral ethyl residues exhibit very high displacement parameters. The guanidine N—H group forms an intermolecular hydrogen bond to the oxydiethyl O atom.

## Comment

The title compound, (1), belongs to a class of compounds well known for its pharmacological activity (Molina, Alajarin & Saez, 1983). Crystal structures of a number of related molecules have already been published (Brown & Sengier, 1984) and the present study is part of a series aimed at correlating the chemical structure and pharmacological activity of substituted guanidines (Sudha *et al.*, 1996, and references therein).



(1)

The guanidine moiety is uncharged and therefore exhibits unequal C—N bond lengths; C12—N13 1.376 (5), C12—N24 1.395 (5) and C12=N11 1.280 (4) Å. The N24 oxydiethyl moiety represents a morpholino group which is in a chair conformation. The pyramidality of the morpholino N atom is known to vary within this class of compounds, with the sums of the angles at nitrogen in the range 337–360° (Wong-Ng, Nyburg, Awwal,

*Acta Crystallographica Section C*
ISSN 0108-2701 © 1997