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.

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

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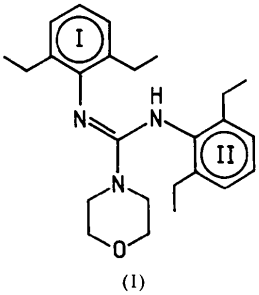
Abstract

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,

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Jankie & Kresge, 1982); in the title compound, the sum of the angles at the N24 atom is 340.2 (4)°.

Both phenyl rings are oriented roughly perpendicular to the guanidine plane [the dihedral angles with the guanidine plane are 78.2 (1)° for ring I and 68.9 (1)° for ring II]; the dihedral angle between the phenyl rings is 54.4 (1)°. The ethyl groups attached to the phenyl rings exhibit very large displacement parameters, indicating strong thermal vibrations or maybe even possible disorder (Fig. 1).

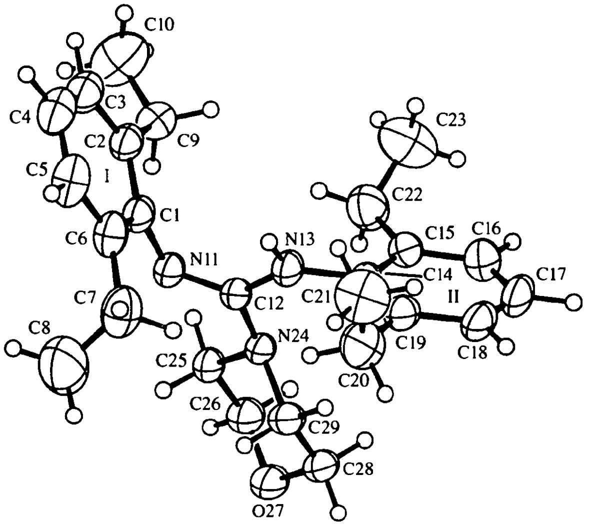

Fig. 1. The molecular structure and atom labelling of the title compound, with displacement ellipsoids drawn at the 30% probability level. The structure cannot be drawn without some overlap.

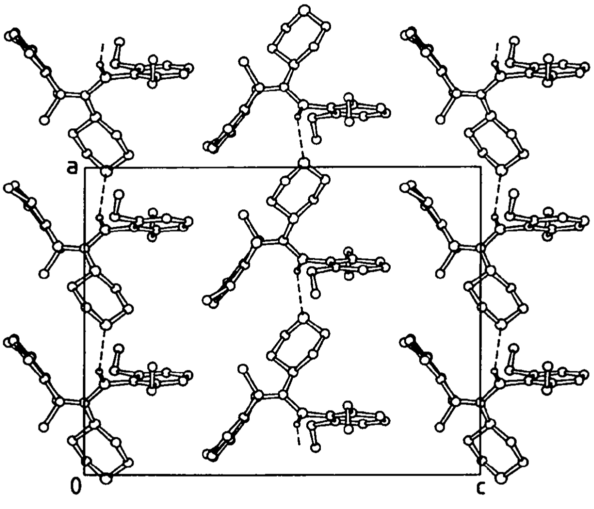

Fig. 2. The crystal-packing arrangement shown in the section y/b = 0–$\frac{1}{2}$. H atoms have been omitted for clarity, except for the H atom of the N—H group, which is involved in hydrogen bonding.

The guanidine N—H group donates a hydrogen bond to the oxydiethyl O atom of a neighbouring molecule [N13···O27($\frac{1}{2}+x$, $\frac{1}{2}-y$, z) 3.000 (4) Å]. The crystal packing is shown in Fig. 2.

Experimental

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

Crystal data

$C_{25}H_{35}N_3O$
M_r = 393.56
Orthorhombic
$Aba2$
a = 14.978 (3) Å
b = 16.703 (3) Å
c = 18.847 (3) Å
V = 4715.1 (15) Å^3
Z = 8
D_x = 1.109 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.54176 Å
Cell parameters from 25 reflections
θ = 10.2–24.3°
μ = 0.525 mm^{-1}
T = 293 (2) K
Prism
0.55 × 0.15 × 0.10 mm
Colourless

Data collection

Enraf–Nonius Turbo-CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
1808 measured reflections
1808 independent reflections
1523 observed reflections [$I > 2\sigma(I)$]

$\theta_{\max}$ = 59.90°
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 21$
3 standard reflections frequency: 60 min intensity decay: 0.6%

Refinement

Refinement on F^2
$R(F)$ = 0.0450
$wR(F^2)$ = 0.1331
S = 1.056
1804 reflections
262 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2 + 0.7239P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max}$ = 0.135 e Å^{-3}
$\Delta\rho_{\min}$ = −0.123 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −0.4 (6)

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}
C1	0.3146 (2)	0.3157 (2)	−0.0978 (2)	0.0685 (10)
C2	0.3808 (3)	0.2744 (2)	−0.1356 (2)	0.0717 (10)
C3	0.4408 (3)	0.3192 (3)	−0.1763 (2)	0.0916 (14)
C4	0.4368 (4)	0.4012 (4)	−0.1792 (3)	0.104 (2)
C5	0.3735 (4)	0.4410 (3)	−0.1405 (3)	0.107 (2)
C6	0.3105 (3)	0.4000 (2)	−0.0996 (2)	0.0848 (12)
C7	0.2382 (4)	0.4451 (3)	−0.0611 (3)	0.110 (2)
C8	0.1540 (6)	0.4519 (6)	−0.1017 (5)	0.202 (5)
C9	0.3848 (3)	0.1860 (3)	−0.1338 (3)	0.0873 (12)

C10	0.4312 (6)	0.1462 (4)	−0.1920 (4)	0.157 (3)
N11	0.2438 (2)	0.2722 (2)	−0.06674 (14)	0.0661 (8)
C12	0.2369 (2)	0.2595 (2)	0.0000 (2)	0.0615 (9)
N13	0.2968 (2)	0.2861 (2)	0.0500 (2)	0.0717 (8)
C14	0.3059 (2)	0.2571 (3)	0.1210 (2)	0.0650 (9)
C15	0.3252 (2)	0.1761 (3)	0.1338 (2)	0.0778 (11)
C16	0.3339 (3)	0.1522 (4)	0.2038 (3)	0.1033 (15)
C17	0.3248 (4)	0.2061 (5)	0.2586 (2)	0.110 (2)
C18	0.3072 (3)	0.2835 (5)	0.2457 (2)	0.102 (2)
C19	0.2974 (3)	0.3114 (3)	0.1768 (2)	0.0811 (12)
C20	0.2746 (4)	0.3979 (4)	0.1661 (3)	0.119 (2)
C21	0.3524 (6)	0.4537 (4)	0.1680 (5)	0.155 (3)
C22	0.3391 (4)	0.1175 (4)	0.0737 (3)	0.112 (2)
C23	0.4111 (5)	0.0598 (4)	0.0835 (5)	0.159 (3)
N24	0.1649 (2)	0.2150 (2)	0.02549 (14)	0.0636 (7)
C25	0.1112 (2)	0.1770 (3)	−0.0295 (2)	0.0710 (10)
C26	0.0447 (3)	0.1201 (3)	0.0045 (2)	0.0849 (11)
O27	−0.0116 (2)	0.1614 (2)	0.0527 (2)	0.0979 (9)
C28	0.0404 (3)	0.1972 (4)	0.1067 (2)	0.0971 (14)
C29	0.1060 (3)	0.2563 (3)	0.0769 (2)	0.0839 (12)

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

C1—N11	1.414 (5)	N13—C14	1.430 (5)
N11—C12	1.280 (4)	N24—C25	1.458 (4)
C12—N13	1.376 (5)	N24—C29	1.481 (5)
C12—N24	1.395 (5)		
C12—N11—C1	123.6 (3)	C12—N13—C14	126.4 (3)
N11—C12—N13	124.5 (3)	C12—N24—C25	114.4 (3)
N11—C12—N24	119.3 (3)	C12—N24—C29	115.9 (3)
N13—C12—N24	116.2 (3)	C25—N24—C29	109.9 (3)
C12—N11—C1—C2	106.4 (4)	N13—C12—N24—C25	−170.5 (3)
C12—N13—C14—C15	58.8 (5)	N13—C12—N24—C29	60.1 (4)
C12—N24—C25—C26	171.0 (3)	N24—C12—N11—C1	−179.3 (3)
C12—N24—C29—C28	−172.0 (3)	N24—C12—N13—C14	16.8 (5)
N11—C12—N13—C14	−162.4 (4)	C1—C2—C9—C10	158.0 (6)
N11—C12—N24—C25	8.8 (5)	C1—C6—C7—C8	−83.9 (7)
N11—C12—N24—C29	−120.7 (4)	C14—C15—C22—C23	139.5 (6)
N13—C12—N11—C1	−0.1 (6)	C14—C19—C20—C21	−99.4 (6)

H atoms bonded to C atoms were refined using a riding model, with the torsion angles of the methyl groups defined by the expected staggered conformation (*SHELXL*93; Sheldrick, 1993). H-atom displacement parameters were restricted to be $1.2U$ of the parent atom, except for the methyl groups, in which $U_{\rm H} = 1.5U_{\rm C}$. The H(N) atom was located from a difference Fourier calculation and was then treated using the riding model. The positioning of H(N) at N13 and not at N11 is supported by the C12—N bond lengths [C12—N13 1.376 (5) and C12=N11 1.280 (4) Å] and by the modes of intermolecular interactions; N13—H is involved in a hydrogen bond, with N13···O27($\frac{1}{2}+x$, $\frac{1}{2}-y$, z) 3.000 (4) Å, whereas N11 has no potential hydrogen-bond acceptor within a radius of 4 Å. This treatment of H atoms was used because displacement parameters of the structure are generally very high. Since the structure contains only light atoms, the orientation of the polar axis (z) cannot be unambiguously determined from the Flack parameter (Flack, 1983).

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.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1214). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hexakis(mercaptomethyl)benzene: a Structure Possessing Well Ordered Homodromic [SH···S]$_6$ Interactions

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Abstract

In the molecular crystal of the title compound, 1,2,3,4,5,6-phenylhexakis(methanethiol), $C_{12}H_{18}S_6$, an intermolecular [SH]$_6$ grouping around a point of $\overline{3}$ symmetry is found between molecules which are stacked infinitely along the c-axial direction.

Comment

The title compound, (1), was selected as an attractive candidate for *ab initio* and experimental electron-density studies owing to the potentially high site symmetry in its molecular crystal. The resulting small 'covalently anchored' asymmetric unit was expected to exhibit atten-

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