Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: LI1090). 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(methoxycarbonyl)-3-phenylguanidine

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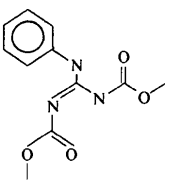
Abstract

The structure of 1,2-bis(methoxycarbonyl)-3-phenylguanidine, $C_{11}H_{13}N_3O_4$, is stabilized into a planar configuration by two intramolecular hydrogen bonds.

Comment

There is a scarcity of structural infomation for guanidine compounds, even though they have been shown to be mutagens and carcinogens (Gichner & Veleminsky, 1982) and are used for the acceleration of the curing of rubber (Brown & Gash, 1984). A search of the chemical literature confirmed the novelty of the title compound (I). The determination of the structure of (I) was undertaken to provide conclusive evidence for the existence of the very stable intramoleculary hydrogen-bonded bis(methoxycarbonyl)guanidine system.



(I)

The guanidine moiety is planar, the sum of the three bond angles around C4 being 360.0°. The bond angles and distances for the guanidine moiety are consistent with the mean values calculated from the Cambridge Structural Database (Krygowski & Wozniak, 1991). The rest of the compound is stabilized into an almost flat conformation by two intramolecular hydrogen bonds; this has also been observed in similar guanidine compounds (Nordenson & Hvoslef, 1981). The r.m.s. deviation of the atoms of the phenyl ring and the atoms involved in hydrogen bonding from the least-squares plane through them is 0.025 Å. Atoms C9 and O3 also lie in this plane. The sums of the three bond angles for the non-H atoms of the six-membered rings in which hydrogen bonding is present (O1, C1, N1, C4, N3, H1 and O2, C5, N2, C4, N1, H) are 370.1 and 372.5°, respectively. The lack of a regular hexagon is probably caused by the presence of the hetero atoms

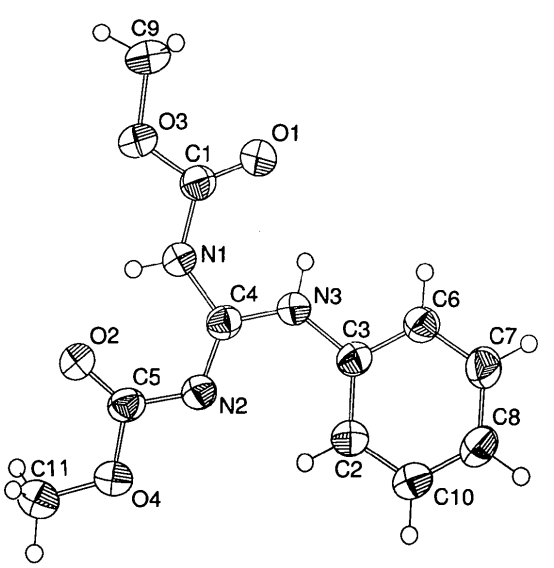


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

and the fact that the H atoms were not located but their positions fixed geometrically. The two hydrogen bonds, between NH and a carbonyl group, are 1.935 (3) and 1.992 (3) Å in length.

Experimental

The compound was prepared by the reaction of *N,N'*-bis-methoxycarbonyl-*S*-methylisothiourea and aniline in methanol containing a little acetic acid as acid catalyst. Recrystallization was from methanol. ^1H and ^{13}C NMR spectra are consistent with the structure shown and the compound also gave a satisfactory microanalysis (0.3% error) for C, H and N.

Crystal data

$C_{11}H_{13}N_3O_4$
$M_r = 251.24$
Monoclinic
$P2_1/a$
$a = 7.889$ (20) Å
$b = 16.824$ (8) Å
$c = 9.5187$ (14) Å
$\beta = 110.168$ (13)°
$V = 1186.0$ (31) Å^3
$Z = 4$
$D_x = 1.407$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 11$–19°
$\mu = 0.109$ mm^{-1}
$T = 293$ (2) K
Transparent block
0.8 × 0.5 × 0.4 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
1543 measured reflections
1292 independent reflections
1276 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.0245$
$\theta_{max} = 24.99°$
$h = -12 \rightarrow 11$
$k = -5 \rightarrow 27$
$l = -3 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0453$
$wR(F^2) = 0.1248$
$S = 0.742$
1292 reflections
164 parameters
$w = 1/[\sigma^2(F_o^2) + (0.1145P)^2 + 1.1510P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ 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)

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}
O1	0.4393 (3)	0.03291 (13)	0.7490 (2)	0.0552 (6)
N3	0.1857 (3)	0.04556 (13)	0.4731 (3)	0.0457 (6)
O2	0.3373 (3)	−0.17606 (13)	0.3718 (2)	0.0630 (7)
N1	0.3752 (3)	−0.06117 (14)	0.5635 (2)	0.0445 (6)
N2	0.1491 (3)	−0.06532 (14)	0.3232 (2)	0.0446 (6)
O3	0.5908 (3)	−0.08279 (13)	0.7779 (2)	0.0543 (6)
C1	0.4661 (4)	−0.0309 (2)	0.7030 (3)	0.0433 (7)
O4	0.0793 (3)	−0.17178 (14)	0.1797 (2)	0.0623 (7)
C4	0.2328 (4)	−0.0278 (2)	0.4486 (3)	0.0405 (7)
C5	0.2026 (4)	−0.1395 (2)	0.3012 (3)	0.0450 (7)
C2	−0.0603 (4)	0.0803 (2)	0.2355 (3)	0.0528 (8)
C3	0.0477 (4)	0.0961 (2)	0.3822 (3)	0.0409 (7)
C7	−0.1059 (4)	0.2205 (2)	0.3710 (4)	0.0545 (8)
C10	−0.1888 (4)	0.1350 (2)	0.1596 (4)	0.0585 (9)
C8	−0.2132 (4)	0.2046 (2)	0.2255 (4)	0.0598 (9)
C6	0.0239 (4)	0.1666 (2)	0.4484 (3)	0.0466 (7)
C9	0.7021 (5)	−0.0594 (2)	0.9280 (3)	0.0663 (10)
C11	0.1113 (5)	−0.2528 (2)	0.1489 (4)	0.0693 (11)

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

O1—C1	1.205 (4)	N2—C5	1.357 (4)
N3—C4	1.333 (4)	O3—C1	1.325 (4)
N3—C3	1.417 (4)	O3—C9	1.450 (4)
O2—C5	1.211 (4)	O4—C5	1.342 (4)
N1—C1	1.371 (4)	O4—C11	1.435 (4)
N1—C4	1.388 (4)	C2—C10	1.375 (5)
N2—C4	1.309 (3)	C2—C3	1.387 (4)
C4—N3—C3	130.6 (2)	C5—O4—C11	115.7 (2)
C1—N1—C4	128.6 (3)	N2—C4—N3	121.5 (3)
C4—N2—C5	119.9 (2)	N2—C4—N1	122.7 (3)
C1—O3—C9	115.9 (2)	N3—C4—N1	115.8 (2)
O1—C1—O3	125.6 (3)	O2—C5—O4	120.6 (3)
O1—C1—N1	125.7 (3)	O2—C5—N2	129.9 (3)
O3—C1—N1	108.6 (3)	O4—C5—N2	109.5 (3)
C9—O3—C1—O1	0.3 (5)	C1—N1—C4—N3	4.9 (4)
C9—O3—C1—N1	179.1 (3)	C11—O4—C5—O2	5.0 (4)
C4—N1—C1—O1	−2.2 (5)	C11—O4—C5—N2	−175.5 (3)
C4—N1—C1—O3	179.1 (3)	C4—N2—C5—O2	−11.9 (5)
C5—N2—C4—N3	179.0 (3)	C4—N2—C5—O4	168.7 (3)
C5—N2—C4—N1	−0.8 (4)	C10—C2—C3—N3	−179.6 (3)
C3—N3—C4—N2	0.9 (5)	C4—N3—C3—C6	173.9 (3)
C3—N3—C4—N1	−179.3 (3)	C4—N3—C3—C2	−6.7 (5)
C1—N1—C4—N2	−175.3 (3)	N3—C3—C6—C7	179.9 (3)

Data collection and cell refinement: *CAD-4 Software* (Enraf–Nonius, 1989). Data reduction: *PROFIT* (Strel'tsov & Zavodnik, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *ORTEP* (Johnson, 1965).

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