
Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares-planes data, along with diagrams showing the molecular stacking for both compounds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71171 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1015]

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Structure of Hydralazine Hydrochloride

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Abstract

The hydrazine side chain of 1(2*H*)-phthalazinone hydrazone hydrochloride is nearly coplanar, with the phthalazine ring having a torsion angle N(2)—C(1)—N(4)—N(5) of −0.4 (5)°. The N(2) atom of the phthalazine ring is protonated and forms an intermolecular hydrogen bond with the amino N atom of the side chain. Another hydrogen bond is formed between the imino N atom of the side chain and the Cl ion.

Comment

1(2*H*)-Phthalazinone hydrazone, also known as hydralazine, is a potent therapeutic drug for hypertension (Perry, 1973). This compound causes site-specific DNA damage (Yamamoto & Kawanishi, 1991) and has mutagenic and/or carcinogenic potency (Toth, 1980; Parodi *et al.*, 1981). A complete structural analysis of this compound therefore seemed very important for understanding its biological function. In this study, the structure of the hydrochloride form of hydralazine has been determined.

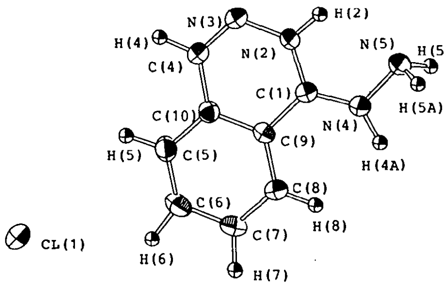

Fig. 1. Perspective view of 1(2*H*)-phthalazinone hydrazone hydrochloride with atomic numbering, viewed down the *x* axis.

Experimental

Crystal data

$C_8H_9N_4^+.Cl^-$
$M_r = 196.64$
Monoclinic
$P2_1/a$
$a = 6.644\ (2)$ Å
$b = 14.531\ (3)$ Å
$c = 9.413\ (1)$ Å
$\beta = 103.57\ (2)^\circ$
$V = 883.4\ (3)$ Å^3
$Z = 4$
$D_x = 1.478$ Mg m^{-3}
$D_m = 1.478\ (1)$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 17 reflections
$\theta = 10.35\text{–}13.50^\circ$
$\mu = 0.385$ mm^{-1}
$T = 296$ K
Needle
0.30 × 0.20 × 0.20 mm
Colorless

Data collection

Rigaku AFC-5*R* diffractometer
ω-2θ scans
Absorption correction: *DIFABS* (Walker & Stuart, 1983)
$T_{\min} = 0.89$, $T_{\max} = 1.03$
2299 measured reflections
2128 independent reflections
1184 observed reflections [$I > 3.00\sigma(I)$]

$R_{\rm int} = 0.024$
$\theta_{\max} = 27.5^\circ$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 18$
$l = -12 \rightarrow 11$
3 standard reflections monitored every 150 reflections intensity variation: 5.08% decay

Refinement

Refinement on F^2
Final $R = 0.047$
$wR = 0.056$
$S = 1.61$
1184 reflections
118 parameters
H-atom parameters not refined
$w = 4F_o^2/\sigma^2(F_o)^2$
$(\Delta/\sigma)_{\max} = 0.01$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2A)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl(1)	0.9171 (2)	0.13564 (7)	0.8161 (1)	3.88 (4)
N(2)	0.0287 (4)	0.4194 (2)	0.1857 (3)	2.5 (1)
N(3)	0.0725 (5)	0.4496 (2)	0.3277 (3)	2.7 (1)
N(4)	−0.0241 (5)	0.3150 (2)	−0.0009 (3)	2.9 (1)
N(5)	−0.0545 (5)	0.3889 (2)	−0.1023 (3)	3.3 (1)
C(1)	0.0160 (5)	0.3322 (2)	0.1402 (4)	2.2 (1)
C(4)	0.1020 (5)	0.3856 (2)	0.4267 (4)	2.4 (1)
C(5)	0.1244 (6)	0.2233 (3)	0.5072 (4)	2.7 (1)
C(6)	0.1141 (6)	0.1313 (3)	0.4725 (4)	2.9 (1)
C(7)	0.0735 (6)	0.1026 (2)	0.3258 (4)	2.7 (1)
C(8)	0.0402 (5)	0.1668 (2)	0.2143 (4)	2.5 (1)
C(9)	0.0487 (5)	0.2603 (2)	0.2492 (4)	2.0 (1)
C(10)	0.0925 (5)	0.2889 (2)	0.3951 (3)	2.1 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *between non*-H *atoms and hydrogen-bond geometry* (Å, °)

N(2)—N(3)	1.371 (4)	C(5)—C(6)	1.374 (5)
N(2)—C(1)	1.334 (4)	C(5)—C(10)	1.400 (5)
N(3)—C(4)	1.299 (4)	C(6)—C(7)	1.406 (5)
N(4)—N(5)	1.420 (4)	C(7)—C(8)	1.383 (5)
N(4)—C(1)	1.316 (4)	C(8)—C(9)	1.396 (5)
C(1)—C(9)	1.444 (4)	C(9)—C(10)	1.398 (4)
C(4)—C(10)	1.435 (4)		
N(3)—N(2)—C(1)	126.9 (3)	C(6)—C(7)—C(8)	120.3 (3)
N(2)—N(3)—C(4)	115.5 (3)	C(7)—C(8)—C(9)	119.2 (3)
N(5)—N(4)—C(1)	119.8 (3)	C(1)—C(9)—C(8)	123.1 (3)
N(2)—C(1)—N(4)	119.2 (3)	C(1)—C(9)—C(10)	116.4 (3)
N(2)—C(1)—C(9)	118.1 (3)	C(8)—C(9)—C(10)	120.5 (3)
N(4)—C(1)—C(9)	122.7 (3)	C(4)—C(10)—C(5)	121.2 (3)
N(3)—C(4)—C(10)	124.1 (3)	C(4)—C(10)—C(9)	118.9 (3)
C(6)—C(5)—C(10)	119.5 (3)	C(5)—C(10)—C(9)	119.9 (3)
C(5)—C(6)—C(7)	120.6 (3)		

X—H···Y	$X\cdots Y$	H···Y	X—H···Y
N(4)—H(4A)···Cl(1^{i})	3.098 (3)	2.122 (1)	173.1 (2)
N(2)—H(2)···N(5^{ii})	2.910 (4)	2.143 (3)	134.6 (2)

Symmetry codes: (i) $x-1, y, z-1$; (ii) $-x, 1-y, -z$.

Data collection and cell refinement: *Rigaku MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). Programs used to solve structure: *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). All calculations including data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Data were collected using a scan rate of 32° min^{-1} in ω and a scan width of $(1.47 + 0.30\tan\theta)°$. The ratio of peak counting time to background counting time was 2:1. Refinement was by full-matrix least-squares methods with anisotropic thermal parameters for all non-H atoms. The coordinates of the H atoms were determined from difference Fourier synthesis but the H-atom parameters were not included in the refinement.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71254 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1021]

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Structure of Cyclo-1,1′,4,4′-bis(1,1,3,3-tetramethyl-1,3-disiloxanediyl)dibenzene

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Abstract

The structure of 2,2,4,4,9,9,11,11-octamethyl-3,10-dioxa-2,4,9,11-tetrasilatricyclo[10.2.2.2^{5,8}]octadeca-1(14),5,7,12,15,17-hexaene (I) consists of molecules located on crystallographic centers of inversion (half a molecule in the asymmetric unit). The two phenyl rings are parallel to each other with a separation of 3.63 Å. The title compound (I) has been considered as a monomer for catalytic ring-opening polymerization. It was synthesized, using allyl(chloro)dimethylsilane, by the condensation of bis(hydroxydimethylsilyl)benzene.

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