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Instrumental Achievements

Crystal Structure of 2,3-Diaza-1,3-diphenylpropene

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The title compound crystallizes in monoclinic space group $P2_1/c$. There are two molecules in the asymmetric unit. While one of these molecules is in a general position, the other is in disordered position. The angle between the directions of the molecules in the asymmetric unit is 87.5(1)˚.

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The hydrazines and related compounds have been the subject of extensive study because of their chemical properties and biological activities.[1] As a part of a series of structural investigations of a compound class containing the phenylhydrazine moiety, the title compound was synthesized by the treatment of benzaldehyde with phenylhydrazine. The resultant orange needle-shaped crystals were recrystallized in an ethanol solution. The crystal data were collected on an Enraf-Nonius CAD-4 diffractometer. Data collection and cell refinement were performed using Enraf-Nonius CAD-4 software.[2] MolEN[3] was used for data reduction. The structure was solved by direct methods using SHELXS97.[4] A refinement was carried out by full-matrix least-squares methods using SHELXL97.[5] The disordered atoms (N27, N28 and C29) were assigned to have a site occupation factor of 0.5. Hydrogen atoms, except for H27 and H29, which are bonded to disordered atoms, were located from difference Fourier maps and refined isotropically. The H27 and H29 atoms were placed geometrically and refined by the riding method. Molecular graphics were prepared using ORTEPII.[6] Figure 1 shows a chemical diagram of the title compound. The obtained crystal data and experimental details are given in Table 1. The final atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms are listed in Table 2. The interatomic distances and angles are presented in Table 3.

The crystal structure of the title compound contains two molecules in an asymmetric unit, as shown in Fig. 2. One is an ordered molecule (A) in a general position; the other (B) is a disordered molecule placed with regard to a crystallographic center of symmetry. The angle between the directions of the ordered and disordered molecule in the asymmetric unit is 87.5(1)˚. In A the angle between the two benzene rings is 8.8(2)˚; these rings are parallel in the disordered molecule B due to crystallographic symmetry. In the crystal, the following intermolecular contacts were observed: C11···N28^{ii}; 3.361(8)Å and C26···N28^{iii}; 2.72(1), for these contacts the C–H···N angles are 134.6(9)˚ and 104.1(9)˚, respectively [symmetry codes: (i) $-1+x$, $1/2-y$, $-1/2+z$, (ii) $1-x$, $-y$, $1-z$]. The above interactions can not be regarded as hydrogen bonding, since the bond length is very long in the first interaction and the concerned angle is

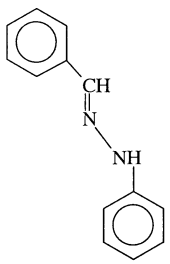

Fig. 1 Chemical structure of the title compound.

Table 1 Crystal and experimental data

Formula: $C_{13}H_{12}N_2$	
Formula weight = 196.25	
Crystal system: monoclinic	
Space group: $P2_1/c$	$Z = 6$
a = 6.053(1)Å	
b = 17.679(1)Å	β = 92.206(8)˚
c = 15.207(1)Å	
V = 1626.1(3)Å^3	
D_x = 1.202 g/cm^3	
R = 0.0440	wR = 0.1070
Goodness-of-fit = 1.307	
$(\Delta/\sigma)_{max}$ = 0.020	
$(\Delta\rho)_{max}$ = 0.163 eÅ^{-3}	
$(\Delta\rho)_{min}$ = –0.114 eÅ^{-3}	
Scan type: $\omega/2\theta$	
Radiation: Mo K_α	
F(0 0 0) = 624	
Crystal dimensions (mm): 0.40 × 0.32 × 0.20	
No. of reflections (measured, unique, used): 2504, 2503, 1955	
No. of parameters refined: 286	
Standard reflections (no., max. intensity variation): 3, 2.3%	
Measurements: Enraf-Nonius CAD4	
Program system: Enraf-Nonius CAD4 software	
Structure determination: SHELXS-97	
Refinement: SHELXL-97	

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Table 2 Final coordinates and equivalent isotropic displacement parameters of the non-hydrogen atoms

Atom	*x*	*y*	*z*	U_{eq} [Å^2]	Occ.
N7	0.0785(6)	0.2637(2)	0.0646(3)	0.104(2)	
N8	0.0647(5)	0.3099(2)	0.1328(2)	0.0885(9)	
C1	0.2614(6)	0.2145(2)	0.0554(2)	0.084(1)	
C2	0.2663(8)	0.1681(2)	-0.0172(3)	0.092(1)	
C3	0.4450(8)	0.1223(3)	-0.0304(3)	0.102(1)	
C4	0.6197(8)	0.1200(3)	0.0301(3)	0.102(1)	
C5	0.6145(8)	0.1639(3)	0.1036(3)	0.101(1)	
C6	0.4388(7)	0.2111(3)	0.1172(3)	0.094(1)	
C9	-0.1149(8)	0.3497(2)	0.1384(3)	0.085(1)	
C10	-0.1461(6)	0.4031(2)	0.2057(2)	0.081(1)	
C11	-0.3461(7)	0.4417(3)	0.2091(3)	0.097(1)	
C12	-0.3779(8)	0.4962(3)	0.2717(3)	0.103(1)	
C13	-0.2123(8)	0.5129(3)	0.3333(3)	0.102(1)	
C14	-0.0149(8)	0.4738(3)	0.3313(3)	0.100(1)	
C15	0.0185(7)	0.4199(2)	0.2692(3)	0.091(1)	
N27	0.743(1)	0.0026(4)	0.4485(5)	0.100(2)	0.5
N28	0.4358(9)	0.0192(4)	0.5077(4)	0.086(2)	0.5
C21	0.7795(8)	0.0723(3)	0.4118(3)	0.096(1)	
C22	0.9600(8)	0.0947(3)	0.3661(3)	0.106(1)	
C23	0.967(1)	0.1648(4)	0.3291(3)	0.114(2)	
C24	0.795(1)	0.2149(4)	0.3392(3)	0.116(2)	
C25	0.6158(9)	0.1919(3)	0.3862(4)	0.113(1)	
C26	0.6086(8)	0.1214(3)	0.4221(3)	0.098(1)	
C29	0.441(2)	0.0891(7)	0.4806(8)	0.087(3)	0.5

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

Table 3 Selected bond distances (Å) and angles (˚)

N8	C9	1.300(5)		C21	N27	1.373(9)	
N8	N7	1.325(5)		C26	C29	1.489(9)	
N7	C1	1.419(5)		N27	N28	1.348(9)	
C9	C10	1.411(6)		N28	C29	1.302(9)	
C21	C26	1.364(6)					
				C26	C21	N27	112.8(5)
C9	N8	N7	117.6(4)	C26	C21	C22	119.7(5)
N8	N7	C1	121.9(4)	N27	C21	C22	127.4(5)
C2	C1	N7	119.1(4)	C25	C26	C29	128.6(6)
C6	C1	N7	122.7(4)	C21	C26	C29	111.2(6)
N8	C9	C10	123.0(4)	N28	N27	C21	127.1(6)
C11	C10	C9	119.8(4)	C29	N28	N27	117.3(7)
C15	C10	C9	122.2(4)	N28	C29	C26	125.3(8)

very small in the second interaction for hydrogen bonds.[6] Because the absence of intermolecular hydrogen bonding between these types of molecules causes an unstable structure, the disordered structure is probably caused by the absence of hydrogen bonding between the molecules.

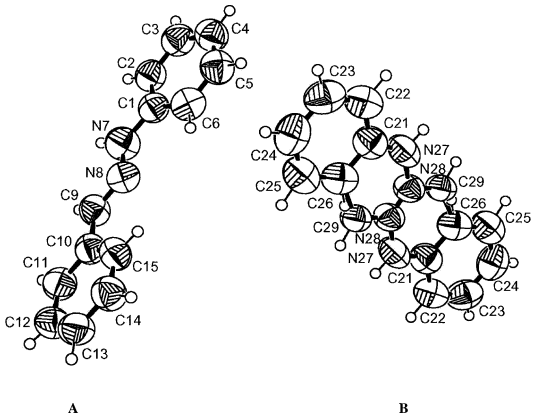

Fig. 2 ORTEP drawing of the asymmetric unit, showing the numbering schemes for atoms at the 50% probability level. Molecule B is disordered.

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