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1,2,3,4-Tetrahydroquinolin-7-amine

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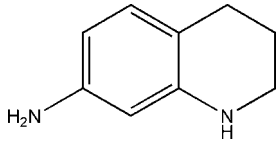
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.093; data-to-parameter ratio = 7.9.

The title compound, $C_9H_{12}N_2$, crystallizes with two almost identical molecules in the asymmetric unit. The ring containing the N atom in the tetrahydroquinoline system adopts a half-chair conformation. The crystal structure is stabilized by intermolecular N—H···N and N—H···π hydrogen bonds.

Related literature

For related literature, see: Field & Hammond (1994).



Experimental

Crystal data

$C_9H_{12}N_2$
M_r = 148.21
Monoclinic, $P2_1$
a = 8.7642 (15) Å
b = 8.7401 (14) Å
c = 11.0393 (18) Å
β = 106.459 (7)°
V = 811.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 113 (2) K
0.10 × 0.04 × 0.04 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.993, T_{max} = 0.997
8269 measured reflections
1762 independent reflections
1586 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.093
S = 1.09
1762 reflections
224 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N2^{i}	0.90 (3)	2.39 (3)	3.271 (3)	164 (2)
N4—H4B···$Cg2^{i}$	0.90 (3)	3.195	4.015	149
N2—H2C···N1^{ii}	0.90 (3)	2.40 (3)	3.194 (3)	147 (2)
N2—H2D···$Cg1^{iii}$	0.90 (3)	2.53	3.446	168
N3—H3···N4^{iv}	0.92 (3)	2.22 (3)	3.078 (3)	156 (2)
N1—H1···N3^{iv}	0.90 (3)	2.44 (3)	3.323 (3)	167 (2)

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z$; (ii) $-x+2, y+\frac{1}{2}, -z$; (iii) $x+1, y+1, z$; (iv) $-x+1, y+\frac{1}{2}, -z$. $Cg1$ is the centroid of the ring C13–C18 and $Cg2$ is the centroid of the ring C4–C9.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2542).

References

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Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. University of Göttingen, Germany.

supplementary materials

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1,2,3,4-Tetrahydroquinolin-7-amine

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Comment

1,2,3,4-Tetrahydroquinolin-7-amine is an important intermediate for the preparation of 7-hydroxy-1,2,3,4-tetrahydroquinoline which is an intermediate useful for the economic manufacture of laser dyes for wavelengths between 540 and 610 nm (Field & Hammond, 1994). The present X-ray crystal structure analysis was undertaken in order to study the stereochemistry and crystal packing of 1,2,3,4-tetrahydroquinolin-7-amine The molecular structure of the title compound is illustrated in Fig. 1. There are two almost identical molecules in the asymmetric unit. The ring containing the nitrigen atom in the tetrahydroquinolin adopts a partical chair conformation. The crystal structure is stabilized by intermolecular N—H···N and N—H···π hydrogen bonds.

Experimental

7-nitro-1, 2, 3, 4-tetrahydroquinoline (0.05 mol, 8.91 g), 80 ml of methanol and 1.5 g of Raney nickel slurry were rinsed with methanol under nitrogen. Addition of a solution of 5.5 ml (0.1 mol) hydrazine hydrate in 10 ml of methanol to the stirred mixture started the reaction. The reaction mixture was heated under reflux to complete the reduction, the catalyst was filtered off through celite and washed with methanol. The filtrate was concentrated *in vacuo* and reconcentrated twice with toluene to remove water. The residue was crystallized from PE to give 6.95 g of 7-amine-1,2,3,4-tetrahydroquinolin as a white needle solid, suitable for X-ray analysis. 1,2,3,4-tetrahydroquinolin-7-amine was quite sensitive to air, it quickly changed black solid.

Refinement

H atoms were positioned geometrically with C—H = 0.93–0.98 Å and refined using riding model with U_{iso} (H) = 1.2 U_{eq} (carrier). H atoms bonded to N were located from difference map and freely refined. Friedel pairs were merged and the absolute structure was arbitrarily assigned.

Figures

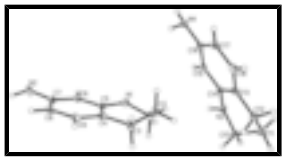

Fig. 1. The molecular structure of the title compound, drawn with 30% probability ellipsoids. H atoms are drawn as spheres of arbitrary radius.

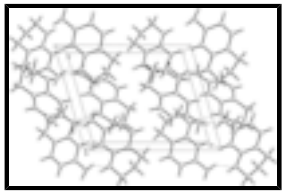

Fig. 2. The crystal structure of the title compound, viewed along the *b* axis.

1,2,3,4-Tetrahydroquinolin-7-amine

Crystal data

$C_9H_{12}N_2$	$D_x = 1.214$ Mg m^{-3}
$M_r = 148.21$	Melting point: 91-93 K
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$a = 8.7642$ (15) Å	Cell parameters from 2296 reflections
$b = 8.7401$ (14) Å	$\theta = 1.9$–27.5º
$c = 11.0393$ (18) Å	$\mu = 0.07$ mm^{-1}
$\beta = 106.459$ (7)º	$T = 113$ (2) K
$V = 811.0$ (2) Å^3	Prism, colorless
$Z = 4$	0.10 × 0.04 × 0.04 mm
$F_{000} = 320$	

Data collection

Rigaku Saturn diffractometer	1586 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	$R_{int} = 0.043$
Monochromator: confocal	$\theta_{max} = 26.4$º
$T = 113$(2) K	$\theta_{min} = 3.0$º
ω scans	$h = -10 \rightarrow 10$
Absorption correction: multi-scan (Jacobson, 1998)	$k = -10 \rightarrow 10$
$T_{min} = 0.993$, $T_{max} = 0.997$	$l = -13 \rightarrow 13$
8269 measured reflections	Standard reflections: ?
1762 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.0684P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
1762 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
224 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.7907 (2)	0.5321 (2)	0.1189 (2)	0.0251 (5)
N2	1.2746 (3)	0.7827 (2)	0.1012 (2)	0.0268 (5)
N3	0.3190 (2)	0.2353 (3)	0.1461 (2)	0.0244 (5)
N4	0.7647 (3)	−0.0285 (3)	0.0695 (2)	0.0267 (5)
C1	0.6786 (3)	0.4944 (3)	0.1903 (2)	0.0278 (6)
H1A	0.5921	0.4292	0.1385	0.033*
H1B	0.6306	0.5892	0.2123	0.033*
C2	0.7669 (3)	0.4098 (3)	0.3100 (2)	0.0313 (6)
H2A	0.8169	0.3164	0.2879	0.038*
H2B	0.6912	0.3787	0.3571	0.038*
C3	0.8946 (3)	0.5144 (3)	0.3923 (2)	0.0308 (6)
H3A	0.8432	0.5951	0.4300	0.037*
H3B	0.9647	0.4540	0.4620	0.037*
C4	0.9940 (3)	0.5883 (3)	0.3161 (2)	0.0227 (5)
C5	0.9367 (3)	0.5982 (3)	0.1844 (2)	0.0208 (5)
C6	1.0290 (3)	0.6669 (3)	0.1147 (2)	0.0216 (5)
H6	0.9884	0.6736	0.0254	0.026*
C7	1.1788 (3)	0.7255 (3)	0.1737 (2)	0.0229 (5)
C8	1.2358 (3)	0.7169 (3)	0.3057 (2)	0.0259 (6)
H8	1.3378	0.7567	0.3481	0.031*
C9	1.1429 (3)	0.6501 (3)	0.3738 (2)	0.0266 (6)
H9	1.1824	0.6465	0.4633	0.032*
C10	0.2362 (3)	0.2950 (3)	0.2333 (3)	0.0330 (7)
H10A	0.1309	0.3353	0.1853	0.040*
H10B	0.2983	0.3800	0.2831	0.040*
C11	0.2152 (3)	0.1690 (4)	0.3212 (3)	0.0356 (7)
H11A	0.1514	0.0849	0.2714	0.043*
H11B	0.1574	0.2092	0.3796	0.043*
C12	0.3775 (3)	0.1080 (3)	0.3969 (2)	0.0281 (6)
H12A	0.4321	0.1861	0.4591	0.034*
H12B	0.3626	0.0154	0.4439	0.034*
C13	0.4798 (3)	0.0691 (3)	0.3116 (2)	0.0219 (5)
C14	0.4495 (3)	0.1397 (3)	0.1925 (2)	0.0209 (5)

C15	0.5473 (3)	0.1068 (3)	0.1147 (2)	0.0201 (5)
H15	0.5266	0.1549	0.0346	0.024*
C16	0.6734 (3)	0.0052 (3)	0.1529 (2)	0.0223 (5)
C17	0.7039 (3)	−0.0655 (3)	0.2708 (2)	0.0263 (6)
H17	0.7899	−0.1352	0.2984	0.032*
C18	0.6071 (3)	−0.0325 (3)	0.3470 (2)	0.0243 (5)
H18	0.6284	−0.0814	0.4269	0.029*
H1	0.749 (3)	0.574 (3)	0.042 (2)	0.023 (7)*
H3	0.323 (3)	0.297 (3)	0.080 (3)	0.032 (8)*
H2C	1.218 (3)	0.832 (4)	0.031 (3)	0.037 (8)*
H2D	1.360 (3)	0.841 (4)	0.147 (3)	0.039 (8)*
H4A	0.769 (3)	0.048 (4)	0.015 (3)	0.041 (9)*
H4B	0.866 (4)	−0.066 (4)	0.107 (3)	0.048 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0245 (10)	0.0299 (13)	0.0195 (11)	−0.0025 (9)	0.0039 (8)	−0.0003 (10)
N2	0.0271 (11)	0.0245 (13)	0.0289 (13)	−0.0037 (9)	0.0084 (9)	0.0005 (10)
N3	0.0270 (11)	0.0248 (12)	0.0234 (11)	0.0066 (9)	0.0104 (9)	0.0049 (10)
N4	0.0248 (11)	0.0268 (13)	0.0303 (12)	−0.0002 (9)	0.0109 (9)	−0.0055 (11)
C1	0.0248 (12)	0.0271 (15)	0.0326 (14)	−0.0044 (11)	0.0097 (10)	−0.0008 (12)
C2	0.0334 (13)	0.0317 (15)	0.0310 (15)	−0.0051 (12)	0.0125 (11)	0.0009 (13)
C3	0.0364 (14)	0.0321 (15)	0.0251 (14)	−0.0082 (12)	0.0103 (11)	0.0006 (12)
C4	0.0270 (12)	0.0217 (13)	0.0199 (12)	0.0029 (10)	0.0073 (9)	0.0004 (10)
C5	0.0221 (11)	0.0159 (12)	0.0238 (12)	0.0037 (10)	0.0055 (9)	−0.0023 (10)
C6	0.0241 (11)	0.0209 (13)	0.0186 (11)	0.0038 (10)	0.0043 (9)	−0.0023 (10)
C7	0.0255 (12)	0.0181 (13)	0.0260 (13)	0.0012 (10)	0.0086 (10)	0.0005 (11)
C8	0.0256 (12)	0.0234 (14)	0.0254 (13)	−0.0028 (11)	0.0020 (10)	−0.0032 (11)
C9	0.0325 (14)	0.0256 (15)	0.0194 (12)	0.0025 (11)	0.0038 (10)	−0.0005 (11)
C10	0.0405 (15)	0.0298 (16)	0.0337 (15)	0.0131 (12)	0.0184 (12)	0.0063 (13)
C11	0.0396 (15)	0.0400 (17)	0.0327 (14)	0.0091 (13)	0.0191 (12)	0.0043 (14)
C12	0.0382 (14)	0.0267 (15)	0.0218 (12)	0.0026 (11)	0.0124 (11)	0.0017 (11)
C13	0.0271 (12)	0.0179 (13)	0.0196 (11)	−0.0035 (10)	0.0049 (9)	−0.0021 (10)
C14	0.0208 (11)	0.0166 (13)	0.0240 (12)	−0.0017 (10)	0.0043 (9)	−0.0036 (10)
C15	0.0237 (12)	0.0180 (13)	0.0183 (11)	−0.0030 (10)	0.0054 (9)	0.0003 (10)
C16	0.0226 (11)	0.0173 (12)	0.0264 (13)	−0.0024 (10)	0.0060 (10)	−0.0053 (11)
C17	0.0220 (12)	0.0217 (14)	0.0312 (14)	0.0024 (10)	0.0013 (10)	−0.0019 (12)
C18	0.0284 (12)	0.0220 (13)	0.0191 (11)	−0.0020 (10)	0.0014 (9)	0.0011 (11)

Geometric parameters (Å, °)

N1—C5	1.403 (3)	C6—C7	1.387 (3)
N1—C1	1.461 (3)	C6—H6	0.9500
N1—H1	0.90 (3)	C7—C8	1.402 (3)
N2—C7	1.405 (3)	C8—C9	1.385 (3)
N2—H2C	0.90 (3)	C8—H8	0.9500
N2—H2D	0.93 (3)	C9—H9	0.9500
N3—C14	1.392 (3)	C10—C11	1.513 (4)

N3—C10	1.456 (3)	C10—H10A	0.9900
N3—H3	0.92 (3)	C10—H10B	0.9900
N4—C16	1.411 (3)	C11—C12	1.527 (4)
N4—H4A	0.90 (3)	C11—H11A	0.9900
N4—H4B	0.92 (3)	C11—H11B	0.9900
C1—C2	1.520 (4)	C12—C13	1.512 (3)
C1—H1A	0.9900	C12—H12A	0.9900
C1—H1B	0.9900	C12—H12B	0.9900
C2—C3	1.529 (4)	C13—C18	1.393 (3)
C2—H2A	0.9900	C13—C14	1.407 (3)
C2—H2B	0.9900	C14—C15	1.404 (3)
C3—C4	1.516 (3)	C15—C16	1.387 (3)
C3—H3A	0.9900	C15—H15	0.9500
C3—H3B	0.9900	C16—C17	1.397 (3)
C4—C9	1.389 (3)	C17—C18	1.383 (3)
C4—C5	1.400 (3)	C17—H17	0.9500
C5—C6	1.401 (3)	C18—H18	0.9500
C5—N1—C1	118.0 (2)	C9—C8—C7	119.7 (2)
C5—N1—H1	112.7 (16)	C9—C8—H8	120.1
C1—N1—H1	116.1 (16)	C7—C8—H8	120.1
C7—N2—H2C	112.6 (17)	C8—C9—C4	122.4 (2)
C7—N2—H2D	113.5 (16)	C8—C9—H9	118.8
H2C—N2—H2D	112 (3)	C4—C9—H9	118.8
C14—N3—C10	118.9 (2)	N3—C10—C11	109.7 (2)
C14—N3—H3	115.7 (16)	N3—C10—H10A	109.7
C10—N3—H3	117.0 (17)	C11—C10—H10A	109.7
C16—N4—H4A	114.3 (19)	N3—C10—H10B	109.7
C16—N4—H4B	115.7 (17)	C11—C10—H10B	109.7
H4A—N4—H4B	110 (3)	H10A—C10—H10B	108.2
N1—C1—C2	108.92 (19)	C10—C11—C12	109.9 (2)
N1—C1—H1A	109.9	C10—C11—H11A	109.7
C2—C1—H1A	109.9	C12—C11—H11A	109.7
N1—C1—H1B	109.9	C10—C11—H11B	109.7
C2—C1—H1B	109.9	C12—C11—H11B	109.7
H1A—C1—H1B	108.3	H11A—C11—H11B	108.2
C1—C2—C3	109.4 (2)	C13—C12—C11	111.3 (2)
C1—C2—H2A	109.8	C13—C12—H12A	109.4
C3—C2—H2A	109.8	C11—C12—H12A	109.4
C1—C2—H2B	109.8	C13—C12—H12B	109.4
C3—C2—H2B	109.8	C11—C12—H12B	109.4
H2A—C2—H2B	108.2	H12A—C12—H12B	108.0
C4—C3—C2	111.3 (2)	C18—C13—C14	117.7 (2)
C4—C3—H3A	109.4	C18—C13—C12	122.5 (2)
C2—C3—H3A	109.4	C14—C13—C12	119.7 (2)
C4—C3—H3B	109.4	N3—C14—C15	118.6 (2)
C2—C3—H3B	109.4	N3—C14—C13	121.6 (2)
H3A—C3—H3B	108.0	C15—C14—C13	119.7 (2)
C9—C4—C5	117.8 (2)	C16—C15—C14	121.2 (2)
C9—C4—C3	121.6 (2)	C16—C15—H15	119.4

C5—C4—C3	120.6 (2)	C14—C15—H15	119.4
C4—C5—C6	120.3 (2)	C15—C16—C17	119.4 (2)
C4—C5—N1	121.1 (2)	C15—C16—N4	119.1 (2)
C6—C5—N1	118.6 (2)	C17—C16—N4	121.4 (2)
C7—C6—C5	121.2 (2)	C18—C17—C16	119.1 (2)
C7—C6—H6	119.4	C18—C17—H17	120.5
C5—C6—H6	119.4	C16—C17—H17	120.5
C6—C7—C8	118.7 (2)	C17—C18—C13	122.9 (2)
C6—C7—N2	120.2 (2)	C17—C18—H18	118.6
C8—C7—N2	121.1 (2)	C13—C18—H18	118.6
C5—N1—C1—C2	−47.4 (3)	C14—N3—C10—C11	−43.4 (3)
N1—C1—C2—C3	62.4 (3)	N3—C10—C11—C12	60.2 (3)
C1—C2—C3—C4	−48.7 (3)	C10—C11—C12—C13	−50.2 (3)
C2—C3—C4—C9	−160.7 (2)	C11—C12—C13—C18	−158.1 (2)
C2—C3—C4—C5	20.2 (3)	C11—C12—C13—C14	23.4 (3)
C9—C4—C5—C6	0.8 (3)	C10—N3—C14—C15	−167.7 (2)
C3—C4—C5—C6	179.9 (2)	C10—N3—C14—C13	16.0 (3)
C9—C4—C5—N1	177.0 (2)	C18—C13—C14—N3	175.9 (2)
C3—C4—C5—N1	−3.9 (4)	C12—C13—C14—N3	−5.5 (3)
C1—N1—C5—C4	18.2 (3)	C18—C13—C14—C15	−0.3 (3)
C1—N1—C5—C6	−165.5 (2)	C12—C13—C14—C15	178.3 (2)
C4—C5—C6—C7	0.4 (3)	N3—C14—C15—C16	−176.1 (2)
N1—C5—C6—C7	−175.9 (2)	C13—C14—C15—C16	0.2 (3)
C5—C6—C7—C8	−1.0 (4)	C14—C15—C16—C17	−0.1 (3)
C5—C6—C7—N2	174.8 (2)	C14—C15—C16—N4	177.7 (2)
C6—C7—C8—C9	0.3 (4)	C15—C16—C17—C18	0.1 (3)
N2—C7—C8—C9	−175.4 (2)	N4—C16—C17—C18	−177.6 (2)
C7—C8—C9—C4	0.9 (4)	C16—C17—C18—C13	−0.2 (4)
C5—C4—C9—C8	−1.5 (4)	C14—C13—C18—C17	0.4 (3)
C3—C4—C9—C8	179.5 (2)	C12—C13—C18—C17	−178.2 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4A···N2^{i}	0.90 (3)	2.39 (3)	3.271 (3)	164 (2)
N4—H4B···Cg2^{i}	0.90 (3)	3.195	4.015	149
N2—H2C···N1^{ii}	0.90 (3)	2.40 (3)	3.194 (3)	147 (2)
N2—H2D···Cg1^{iii}	0.90 (3)	2.53	3.446	168
N3—H3···N4^{iv}	0.92 (3)	2.22 (3)	3.078 (3)	156 (2)
N1—H1···N3^{iv}	0.90 (3)	2.44 (3)	3.323 (3)	167 (2)

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*; (ii) −*x*+2, *y*+1/2, −*z*; (iii) *x*+1, *y*+1, *z*; (iv) −*x*+1, *y*+1/2, −*z*.

Fig. 1

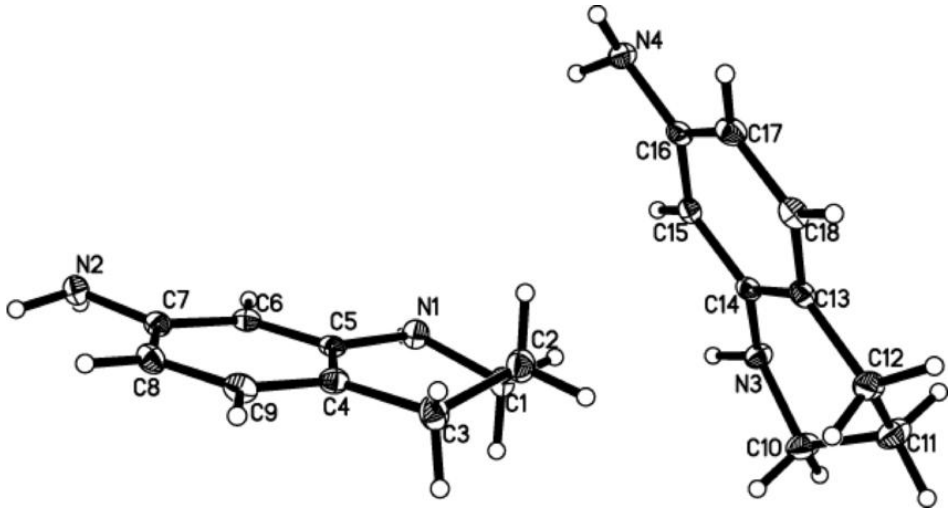
N4
C16
C17
C15
C18
C14
C13
N3
C12
C10
C11
N2
C7
C6
C5
N1
C2
C8
C9
C4
C3
C1

Fig. 2