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1,3,5-Tris(1*H*-pyrazol-3-yl)benzene

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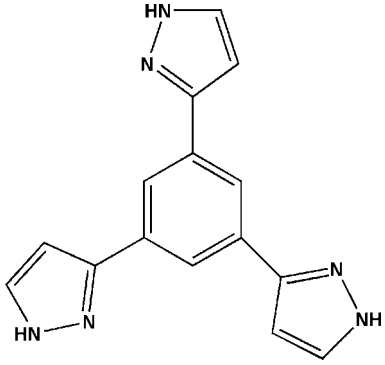
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.112; data-to-parameter ratio = 8.8.

The title compound, $C_{15}H_{12}N_6$, is a tri- or multidentate ligand. The asymmetric unit contains one molecule. The crystal packing is stabilized by intermolecular N—H···N hydrogen-bond interactions, resulting in a three-dimensional supramolecular network.

Related literature

For related literature, see: Peng *et al.* (2006); Pleier *et al.* (2001); Zheng *et al.* (2003).



Experimental

Crystal data

$C_{15}H_{12}N_6$
M_r = 276.31
Orthorhombic, $Pna2_1$
a = 8.183 (5) Å
b = 17.128 (5) Å
c = 9.173 (5) Å
V = 1285.7 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.43 × 0.28 × 0.27 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.962, T_{max} = 0.976
7598 measured reflections
1670 independent reflections
1342 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.112
S = 1.03
1670 reflections
190 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 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
N5—H5···N2^{i}	0.86	2.10	2.960 (3)	173
N3—H3···N6^{ii}	0.86	2.04	2.875 (3)	163
N1—H1···N4^{iii}	0.86	2.01	2.855 (3)	169

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z+1$; (ii) $-x+1, -y+1, z-\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2069).

References

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supplementary materials

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1,3,5-Tris(1*H*-pyrazol-3-yl)benzene

W.-X. Zhao, Y.-X. Gao, S.-F. Dong, Y. Li and W.-P. Zhang

Comment

Recently, there has been much interest in the study of crystal engineering of supramolecular architectures using N-donor ligands (Zheng *et al.*, 2003; Peng *et al.*, 2006). The title compound, $C_{15}H_{12}N_6$, can be an important organic tri- or polydentate ligand to form new metal complexes.

The asymmetric unit (Fig. 1), which contains one molecule. Molecules are linked together *via* intermolecular N—H···N hydrogen bond interactions (Table. 1). The intermolecular N—H···N contacts (2.856 (3)–2.961 (3) Å) satisfy the definition of standard hydrogen bonds and resulted in a three dimensional supramolecular network (Fig. 2). Interestingly, all of the non-H atoms in the title compound are not coplanar (the mean deviation of the atoms from the least-squares plane is 0.153 Å).

Experimental

All reagents were purchased (Aldrich) and used without further purification. The title compound was synthesized according to the literature method (Pleier *et al.*, 2001). The title compound (0.552 g, 0.002 mol) was dissolved into 20 ml DMF. After heating at 70 °C for 20 min, the mixture was allowed to cool and evaporate naturally. Yellow block crystals suitable for single-crystal X-ray diffraction were obtained by evaporating the mixture at room temperature for a period of two weeks. Analysis found: C 65.3, H 4.4, N 30.3%; $C_{15}H_{12}N_6$ requires: C 65.21, H 4.38, N 30.42%.

Refinement

All H atoms were visible in difference Fourier maps but were placed in calculated positions with C—H= 0.93 Å (CH) or N—H= 0.90 Å (NH), U_{iso}(H)= 1.2 times U_{eq}(C) or U_{eq}(N) for CH or NH. All other non-H atoms were refined anisotropically. The maximum positive peak of 0.26 e Å^{-3} in the final difference electron density map was located 0.73 Å from atom C5. As the absolute structure cannot been determined for a light atom structure with Mo K-α radiation under normal circumstances, the Friedel-pair reflections have been merged before final refinement.

Figures

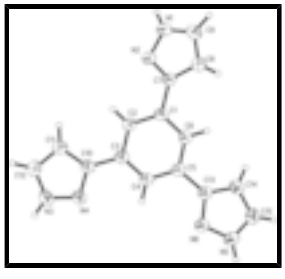

Fig. 1. The asymmetric unit of the title compound with the atomic labeling scheme. Displacement ellipsoids are shown at the 50% probability level.

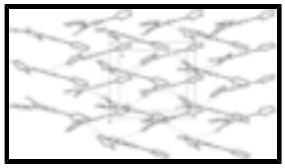

Fig. 2. The packing of the title compound, viewed down the *b* axis, showing three-dimensional supramolecular network connected by N—H···N hydrogen bonds (dashed lines).

1,3,5-Tris(1*H*-pyrazol-3-yl)benzene

Crystal data

$C_{15}H_{12}N_6$	$D_x = 1.427$ Mg m^{-3}
$M_r = 276.31$	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
Orthorhombic, $Pna2_1$	Cell parameters from 864 reflections
$a = 8.183$ (5) Å	$\theta = 3.7$–22.8º
$b = 17.128$ (5) Å	$\mu = 0.09$ mm^{-1}
$c = 9.173$ (5) Å	$T = 293$ (2) K
$V = 1285.7$ (11) Å^3	Block, yellow
$Z = 4$	0.43 × 0.28 × 0.27 mm
$F_{000} = 576$	

Data collection

Bruker SMART APEX II CCD diffractometer	1670 independent reflections
Radiation source: fine-focus sealed tube	1342 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.037$
$T = 293(2)$ K	$\theta_{max} = 28.3$º
φ and ω scans	$\theta_{min} = 2.4$º
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -10 \rightarrow 9$
$T_{min} = 0.962$, $T_{max} = 0.976$	$k = -19 \rightarrow 21$
7598 measured reflections	$l = -12 \rightarrow 11$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2 + 0.0158P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.112$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.03$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
1670 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
190 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), Number of Friedel pairs?

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Flack parameter: 10 (10)

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}
C1	0.3602 (3)	0.18881 (14)	0.5920 (3)	0.0366 (5)
C2	0.3385 (3)	0.26016 (13)	0.5217 (3)	0.0369 (5)
H2	0.3054	0.2612	0.4247	0.044*
C3	0.3662 (3)	0.33028 (14)	0.5959 (3)	0.0374 (6)
C4	0.4121 (3)	0.32782 (14)	0.7426 (3)	0.0383 (6)
H4	0.4291	0.3742	0.7929	0.046*
C5	0.4326 (3)	0.25690 (13)	0.8144 (3)	0.0378 (6)
C6	0.4073 (3)	0.18830 (14)	0.7377 (3)	0.0393 (6)
H6	0.4221	0.1407	0.7848	0.047*
C7	0.3371 (3)	0.11425 (14)	0.5129 (2)	0.0359 (5)
C8	0.4001 (4)	0.04043 (15)	0.5471 (3)	0.0473 (7)
H8	0.4687	0.0274	0.6241	0.057*
C9	0.3388 (4)	−0.00815 (16)	0.4426 (3)	0.0513 (7)
H9	0.3563	−0.0617	0.4369	0.062*
C10	0.3529 (3)	0.40462 (14)	0.5151 (3)	0.0372 (5)
C11	0.3294 (3)	0.41669 (15)	0.3655 (3)	0.0446 (6)
H11	0.3131	0.3787	0.2945	0.053*
C12	0.3355 (4)	0.49591 (16)	0.3455 (3)	0.0486 (7)
H12	0.3255	0.5221	0.2571	0.058*
C13	0.4818 (3)	0.25235 (13)	0.9689 (3)	0.0373 (5)
C14	0.4694 (3)	0.18895 (14)	1.0637 (3)	0.0435 (6)
H14	0.4265	0.1400	1.0427	0.052*
C15	0.5333 (3)	0.21336 (15)	1.1941 (3)	0.0444 (6)
H15	0.5424	0.1837	1.2787	0.053*
N1	0.2498 (3)	0.03384 (13)	0.3505 (3)	0.0482 (6)
H1	0.2004	0.0152	0.2754	0.058*
N2	0.2472 (3)	0.11020 (12)	0.3905 (2)	0.0428 (5)
N3	0.3585 (3)	0.52856 (13)	0.4757 (3)	0.0538 (6)
H3	0.3642	0.5780	0.4908	0.065*
N4	0.3717 (3)	0.47320 (13)	0.5816 (2)	0.0478 (6)

N5	0.5799 (3)	0.28760 (13)	1.1773 (2)	0.0469 (6)
H5	0.6233	0.3156	1.2448	0.056*
N6	0.5498 (3)	0.31309 (11)	1.0397 (2)	0.0444 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0490 (13)	0.0301 (12)	0.0306 (12)	−0.0023 (10)	−0.0015 (10)	−0.0039 (10)
C2	0.0520 (14)	0.0333 (13)	0.0253 (11)	−0.0002 (10)	−0.0035 (10)	−0.0033 (10)
C3	0.0473 (13)	0.0303 (12)	0.0347 (13)	0.0015 (10)	−0.0005 (11)	−0.0027 (10)
C4	0.0538 (14)	0.0292 (13)	0.0320 (12)	−0.0017 (10)	−0.0050 (11)	−0.0058 (10)
C5	0.0476 (13)	0.0352 (14)	0.0306 (13)	−0.0021 (10)	−0.0036 (11)	−0.0037 (9)
C6	0.0581 (15)	0.0308 (13)	0.0292 (12)	−0.0004 (10)	−0.0058 (11)	−0.0008 (10)
C7	0.0517 (13)	0.0298 (12)	0.0261 (11)	−0.0020 (10)	−0.0010 (10)	−0.0018 (10)
C8	0.0674 (17)	0.0356 (13)	0.0389 (14)	0.0061 (12)	−0.0106 (13)	−0.0025 (11)
C9	0.0731 (18)	0.0324 (14)	0.0485 (17)	0.0068 (13)	−0.0084 (14)	−0.0068 (13)
C10	0.0472 (13)	0.0301 (12)	0.0344 (12)	0.0014 (10)	−0.0004 (11)	−0.0037 (10)
C11	0.0618 (16)	0.0363 (14)	0.0356 (13)	−0.0012 (11)	−0.0063 (13)	−0.0054 (11)
C12	0.0675 (17)	0.0423 (15)	0.0361 (13)	−0.0018 (13)	−0.0035 (12)	0.0056 (12)
C13	0.0489 (13)	0.0331 (13)	0.0299 (12)	−0.0017 (10)	−0.0030 (11)	−0.0043 (9)
C14	0.0592 (16)	0.0308 (12)	0.0405 (15)	−0.0041 (11)	−0.0026 (12)	−0.0006 (11)
C15	0.0564 (15)	0.0408 (14)	0.0361 (14)	0.0013 (12)	0.0006 (12)	0.0026 (12)
N1	0.0695 (15)	0.0361 (13)	0.0389 (11)	−0.0025 (10)	−0.0096 (11)	−0.0085 (9)
N2	0.0653 (14)	0.0327 (11)	0.0303 (11)	−0.0027 (10)	−0.0084 (10)	−0.0034 (9)
N3	0.0862 (17)	0.0317 (12)	0.0436 (13)	−0.0032 (11)	0.0032 (13)	0.0023 (10)
N4	0.0768 (16)	0.0301 (11)	0.0364 (12)	−0.0014 (10)	0.0042 (12)	−0.0023 (9)
N5	0.0678 (15)	0.0413 (12)	0.0316 (11)	−0.0026 (10)	−0.0083 (11)	−0.0051 (10)
N6	0.0699 (15)	0.0327 (10)	0.0307 (11)	−0.0049 (10)	−0.0084 (11)	−0.0028 (9)

Geometric parameters (Å, °)

C1—C6	1.392 (3)	C10—N4	1.332 (3)
C1—C2	1.393 (3)	C10—C11	1.401 (4)
C1—C7	1.481 (3)	C11—C12	1.370 (3)
C2—C3	1.399 (3)	C11—H11	0.9300
C2—H2	0.9300	C12—N3	1.332 (4)
C3—C4	1.397 (3)	C12—H12	0.9300
C3—C10	1.477 (3)	C13—N6	1.347 (3)
C4—C5	1.392 (3)	C13—C14	1.395 (3)
C4—H4	0.9300	C14—C15	1.371 (4)
C5—C6	1.385 (3)	C14—H14	0.9300
C5—C13	1.475 (4)	C15—N5	1.337 (4)
C6—H6	0.9300	C15—H15	0.9300
C7—N2	1.344 (3)	N1—N2	1.358 (3)
C7—C8	1.401 (3)	N1—H1	0.8600
C8—C9	1.365 (4)	N3—N4	1.362 (3)
C8—H8	0.9300	N3—H3	0.8600
C9—N1	1.327 (3)	N5—N6	1.358 (3)
C9—H9	0.9300	N5—H5	0.8600

C6—C1—C2	119.0 (2)	C11—C10—C3	129.0 (2)
C6—C1—C7	120.1 (2)	C12—C11—C10	105.8 (2)
C2—C1—C7	120.9 (2)	C12—C11—H11	127.1
C1—C2—C3	120.5 (2)	C10—C11—H11	127.1
C1—C2—H2	119.8	N3—C12—C11	107.5 (3)
C3—C2—H2	119.8	N3—C12—H12	126.3
C4—C3—C2	119.1 (2)	C11—C12—H12	126.3
C4—C3—C10	121.9 (2)	N6—C13—C14	109.3 (2)
C2—C3—C10	118.9 (2)	N6—C13—C5	122.4 (2)
C5—C4—C3	121.0 (2)	C14—C13—C5	128.4 (2)
C5—C4—H4	119.5	C15—C14—C13	106.2 (2)
C3—C4—H4	119.5	C15—C14—H14	126.9
C6—C5—C4	118.8 (2)	C13—C14—H14	126.9
C6—C5—C13	118.9 (2)	N5—C15—C14	107.4 (2)
C4—C5—C13	122.3 (2)	N5—C15—H15	126.3
C5—C6—C1	121.6 (2)	C14—C15—H15	126.3
C5—C6—H6	119.2	C9—N1—N2	111.0 (2)
C1—C6—H6	119.2	C9—N1—H1	124.5
N2—C7—C8	110.0 (2)	N2—N1—H1	124.5
N2—C7—C1	121.6 (2)	C7—N2—N1	105.46 (19)
C8—C7—C1	128.4 (2)	C12—N3—N4	111.0 (2)
C9—C8—C7	104.9 (2)	C12—N3—H3	124.5
C9—C8—H8	127.5	N4—N3—H3	124.5
C7—C8—H8	127.5	C10—N4—N3	106.1 (2)
N1—C9—C8	108.6 (2)	C15—N5—N6	111.2 (2)
N1—C9—H9	125.7	C15—N5—H5	124.4
C8—C9—H9	125.7	N6—N5—H5	124.4
N4—C10—C11	109.5 (2)	C13—N6—N5	106.0 (2)
N4—C10—C3	121.4 (2)		

C6—C1—C2—C3	1.0 (4)	N4—C10—C11—C12	0.1 (3)
C7—C1—C2—C3	−177.7 (2)	C3—C10—C11—C12	−176.5 (2)
C1—C2—C3—C4	−1.6 (4)	C10—C11—C12—N3	−0.9 (3)
C1—C2—C3—C10	175.9 (2)	C6—C5—C13—N6	162.0 (3)
C2—C3—C4—C5	1.0 (4)	C4—C5—C13—N6	−17.4 (4)
C10—C3—C4—C5	−176.4 (2)	C6—C5—C13—C14	−17.5 (4)
C3—C4—C5—C6	0.2 (4)	C4—C5—C13—C14	163.0 (3)
C3—C4—C5—C13	179.7 (3)	N6—C13—C14—C15	−0.4 (3)
C4—C5—C6—C1	−0.8 (4)	C5—C13—C14—C15	179.2 (3)
C13—C5—C6—C1	179.7 (3)	C13—C14—C15—N5	0.4 (3)
C2—C1—C6—C5	0.2 (4)	C8—C9—N1—N2	−0.7 (3)
C7—C1—C6—C5	178.9 (2)	C8—C7—N2—N1	1.7 (3)
C6—C1—C7—N2	159.7 (2)	C1—C7—N2—N1	−178.7 (2)
C2—C1—C7—N2	−21.6 (4)	C9—N1—N2—C7	−0.6 (3)
C6—C1—C7—C8	−20.7 (4)	C11—C12—N3—N4	1.4 (3)
C2—C1—C7—C8	158.0 (3)	C11—C10—N4—N3	0.7 (3)
N2—C7—C8—C9	−2.1 (3)	C3—C10—N4—N3	177.6 (2)
C1—C7—C8—C9	178.3 (2)	C12—N3—N4—C10	−1.3 (3)
C7—C8—C9—N1	1.7 (3)	C14—C15—N5—N6	−0.3 (3)

C4—C3—C10—N4	−5.3 (4)	C14—C13—N6—N5	0.2 (3)
C2—C3—C10—N4	177.3 (2)	C5—C13—N6—N5	−179.4 (2)
C4—C3—C10—C11	171.0 (3)	C15—N5—N6—C13	0.1 (3)
C2—C3—C10—C11	−6.4 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5···N2^{i}	0.86	2.10	2.960 (3)	173
N3—H3···N6^{ii}	0.86	2.04	2.875 (3)	163
N1—H1···N4^{iii}	0.86	2.01	2.855 (3)	169

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

Fig. 1

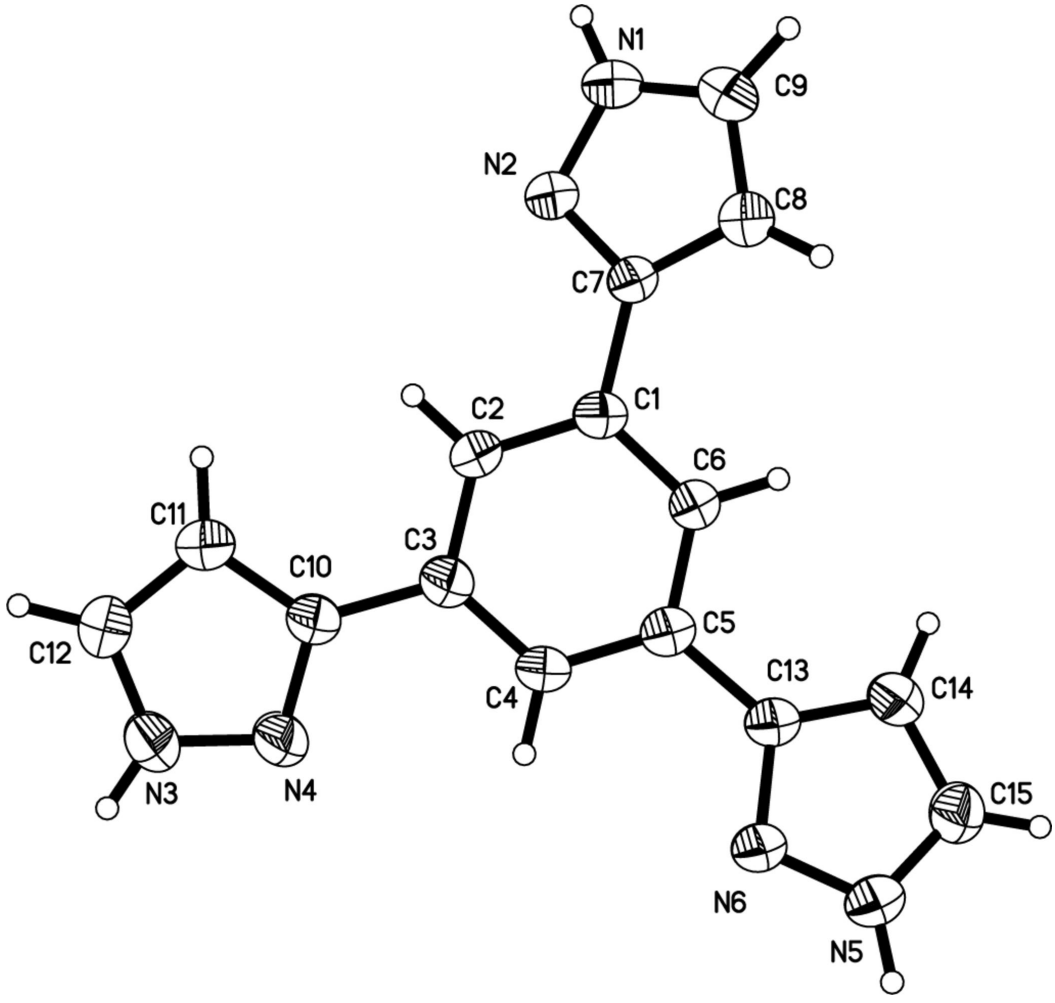

Fig. 2