

2-[(Cyclopenta-1,3-dien-2-yl)diphenylmethyl]-1-methyl-1*H*-imidazoleQi Sun,[‡] Wanli Nie and Maxim V. Borzov*

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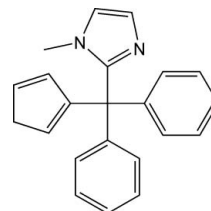
Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.040; wR factor = 0.120; data-to-parameter ratio = 8.8.

The title compound, $\text{C}_{22}\text{H}_{20}\text{N}_2$, (**Ib**), forms along with 2-[(cyclopenta-1,3-dien-1-yl)diphenylmethyl]-1-methyl-1*H*-imidazole, (**Ia**), which differs with respect to the position of the double-bonds in the C_5H_5 ring, in an approximately 3:7 ratio (**Ia**:**Ib**; NMR spectroscopy data). However, in a single crystal, only compound (**Ib**) is present. H atoms of the CH_2 group (C_5H_5 ring) were found from the difference Fourier synthesis and refined isotropically using the riding model. Hypothesis on possible presence of the (**Ia**) isomer in crystal lattice (model with a C_5H_5 ring disordered between two positions) was especially checked and rejected due to its inconsistency. In the crystal structure, no significant hydrogen-bonding interactions between the CH_2 groups of the C_5H_5 rings and nonsubstituted N-atoms of the imidazole rings were observed. Despite the fact that the chemically achiral compound (**I**) crystallizes in a chiral space group $P2_12_12_1$, neither the absolute structure determination nor assignment of the inversion twinning was possible in the absence of a heavy atom.

Related literature

For the structural parameters of mono-alkyl substituted cyclopentadienes (organic structures only), see: Tacke *et al.* (2001); Liebling & Marsh (1965); Haumann *et al.* (1996); Deck *et al.* (2004); Malpass *et al.* (2004); Cheung *et al.* (2005); Bauer *et al.* (2001); Huerlander *et al.* (2002); Millelr & Bercaw (2004); Li *et al.* (2005); Brunner *et al.* (2004); Otero *et al.* (2007); Hutton *et al.* (2008). For the structural parameters of 1,2-dialkyl-1*H*-imidazoles (organic structures only, not bi- or oligocyclic, non-ionic), see: Bruijninx *et al.* (2005); Aakeroy *et al.* (2006); Zhang *et al.* (2007); Upadhyaya *et al.* (1997); Braussaud *et al.* (2001); Peters *et al.* (2005); Laus *et al.* (2008).

For the structural parameters of Li, Ti, and Zr complexes derived from 1*H*-imidazol(in)-2-yl side-chain-functionalized cyclopentadienes, see: Krut'ko *et al.* (2006); Nie *et al.* (2008); Wang *et al.* (2009). For a description of the Cambridge Structural database, see: Allen (2002).



Experimental

Crystal data

$\text{C}_{22}\text{H}_{20}\text{N}_2$	$V = 1700.6$ (14) Å ³
$M_r = 312.40$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 10.563$ (5) Å	$\mu = 0.07$ mm ⁻¹
$b = 10.603$ (5) Å	$T = 295$ K
$c = 15.185$ (7) Å	$0.20 \times 0.05 \times 0.05$ mm

Data collection

Bruker SMART APEXII diffractometer	8785 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1922 independent reflections
$T_{\min} = 0.986$, $T_{\max} = 0.996$	1543 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	5 restraints
$wR(F^2) = 0.120$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.27$ e Å ⁻³
1922 reflections	$\Delta\rho_{\text{min}} = -0.11$ e Å ⁻³
218 parameters	

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008) and OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: SHELXTL and OLEX2.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2523).

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2-[(Cyclopenta-1,3-dien-2-yl)diphenylmethyl]-1-methyl-1*H*-imidazole

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Comment

1*H*-Imidazol(in)-2-yl side-chain functionalized cyclopentadienes were introduced as ligands into the organometallic chemistry, and, particularly into that of the Group 4 transition metals, not long ago (Krut'ko *et al.*, 2006; Nie *et al.*, 2008; Wang *et al.*, 2009). This contribution reports the first structural characterization of a ligand of question in its CH-acid form.

The title compound, C₂₂H₂₀N₂, (**I**), presents a mixture of two isomers on the substituent position in respect to the double bond system in the C₅H₅-ring, 2-[(cyclopenta-1,3-dien-1-yl)diphenylmethyl]-1-methyl-1*H*-imidazole, (*Ia*), and 2-[(cyclopenta-1,3-dien-2-yl)diphenylmethyl]-1-methyl-1*H*-imidazole, (*Ib*), what are formed in an approximately 3: 7 ratio. However, in a single-crystal, only compound (*Ib*) is present. H-atoms of the CH₂-group (C₅H₅-ring) were found from the difference Fourier synthesis and refined isotropically using the riding model. Hypothesis on possible presence of the (*Ia*) isomer in crystal lattice (model with a C₅H₅-ring disordered between two positions) was especially checked and rejected due to its inconsistency. The Cp-ring is planar within 0.004 Å, with the bridging carbon C5 deviating from the r.m.s. C11 through C15 plane by 0.084 (4) Å. As for the rest of the molecule, all the bond lengths and angles are within normal ranges (see Related literature section). Both phenyl rings C21 through C26 and C31 through C36 are planar within 0.02 Å. The imidazole moiety C1/N1/C2/C3/N2/ is planar within 0.005 Å, with atoms C4 (1-methyl group) and C5 (bridging carbon atom) deviating from the imidazole ring plane by 0.091 (5) and 0.021 (4) Å, respectively. No special intermolecular contacts in the crystal lattice were observed. Despite the fact that a chemically achiral compound (**I**) crystallizes in a chiral space group *P*2₁2₁2₁, neither the absolute structure determination nor approval of the inversion twinning was possible due to evident reasons (Mo-*K*α radiation with no atoms heavier than nitrogen).

Experimental

All operations were performed on an Ar-vacuum line using the conventional Schlenk technique. — NMR spectra were recorded on Varian INOVA-400 instrument in CDCl₃ at 298 K. For ¹H and ¹³C{¹H} spectra, the TMS resonances (δ_H = 0.0 and δ_C = 0.0) were used as internal reference standards. — Chromato-mass spectrum was measured on Agilent 6890 Series GC system equipped with HP 5973 mass-selective detector. — The elemental analysis was performed on the Vario ELIII CHNOS automated analyzer.

To a solution of 1-methyl-1*H*-imidazole (1.23 g, 15.0 mmol) in tetrahydrofuran (THF; 60 ml), a solution of *n*-BuLi in hexane (8.3 ml, 1.82 *M*, 15.1 mmol) was added under stirring at 195 K (acetone bath) during 30 min. The reaction mixture was stirred at the same temperature for additional 45 min, that gave a bright-yellow solution. To this slurry, a solution of 6,6-diphenylfulvene (3.45 g, 15.0 mmol) in THF (45 ml) was added dropwise during 30 min. The color of the reaction mixture turned brown. The reaction mixture was allowed to warm up gradually up to ambient temperature and the stirring was continued for the next 12 h. The mixture was quenched with water (50 ml; ice-bath cooling), the organic phase was separated, the water phase was extracted with CH₂Cl₂ (3 × 30 ml), and the combined extracts were dried with Na₂SO₄. Concentrating of the organic extracts on a rotary evaporator gave crude (**I**) as a brown solid. Crude (**I**) was refluxed with

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Et₂O (10 ml) cooled down to room temperature and the filtered-off solid was recrystallized from CH₂Cl₂ – Et₂O mixture (1.8: 10) that gave pure (**I**) as yellowish crystals. Yield 1.6 g, (34%). — ¹H NMR: δ = 2.92 (s, 3 H, NCH₃, *a*), 2.96 (s, 3 H, NCH₃, *b*), 3.05 (m, 2 H, CH₂ in Cp, *b*), 3.10 (m, 2 H, CH₂ in Cp, *a*), 5.91, 6.05, 6.27, 6.28, 6.37, 6.38 (all m, all 1 H in respective ratios, CH in Cp, *a,b*), 6.84, 7.07 (both d, 1 H + 1 H, ³J_{HH} = 1.22 Hz, CH=CH in imidazole, *b*), 6.85, 7.06 (both d, 1 H + 1 H, ³J_{HH} = 1.22 Hz, CH=CH in imidazole, *a*), 7.21–7.32 (m, 10 H in respect to the sum of the relative integral intensities for *a* and *b*, CH in Ph, *a,b*). Molar ratio *a*: *b* equals to 3: 7. — ¹³C{¹H} NMR: δ = 34.75 (NCH₃, *b*), 34.90 (NCH₃, *a*), 40.32 (CH₂ in Cp, *b*), 43.85 (CH₂ in Cp, *a*), 57.14 (CPh₂, *b*), 58.02 (CPh₂, *a*), 122.31, 126.63 (CH=CH in imidazole, *a,b*), 126.43 (*p*-CH in Ph, *a*), 126.51 (*p*-CH in Ph, *b*), 127.73, 129.38 (*o*- and *m*-CH in Ph, *b*), 127.79, 129.16 (*o*- and *m*-CH in Ph, *a*), 128.76, 131.98, 135.94 (CH in Cp, *b*), 130.58, 130.91, 134.00 (CH in Cp, *a*), 142.67 (*ipso*-C in Ph, *b*), 143.32 (*ipso*-C in Ph, *a*), 150.14 (N=C=N, *b*), 150.67 (N=C=N, *a*), 150.60 (C in Cp, *b*), 153.13 (C in Cp, *a*). — EI MS (70 eV) *m/z* (%): 312 (100) [*M*], 311 (66) [*M* – H], 297 (3) [*M* – CH₃], 285 (34) [*M* – HCN], 247 (9) [*M* – C₅H₅], 235 (72) [*M* – Ph]. — Anal. Found: C, 83.81; H, 6.35; N, 8.76%. C₂₂H₂₀N₂ Calc.: C, 84.58; H, 6.45; N, 8.97%. Single crystal of (**I**) suitable for X-ray diffraction analysis was obtained by crystallization of (**I**) from CH₂Cl₂ – Et₂O mixture (1: 6 vol.)

Refinement

Non-H atoms were refined anisotropically. H atoms were treated as riding atoms with distances C—H = 0.96 (CH₃), 0.97 (CH₂), 0.93 Å (C_{Ar}H), and *U*_{iso}(H) = 1.5 *U*_{eq}(C), 1.2 *U*_{eq}(C), and 1.2 *U*_{eq}(C), respectively. The components of the anisotropic displacement parameters (ADP-s) for C12 through C15-atoms of the C₅H₄-group along 1,2- and 1,3-directions were restrained to be the same with *su* of 0.002 Å² (DELU instruction). Despite the fact that an achiral compound (**I**) crystallizes in a chiral space group *P*2₁2₁2₁, neither the absolute structure determination nor approval of the inversion twinning was possible due to evident reasons (Mo-*K*α radiation with no atoms heavier than nitrogen). Thus, the Friedel opposites were merged and treated as equivalents.

Figures

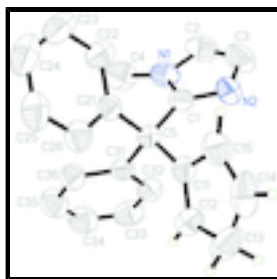


Fig. 1. A view of the molecule of compound (**I**). Displacement ellipsoids are shown at the 50% probability level. All H-atoms except of those in the C₅H₅-ring are omitted for clarity.

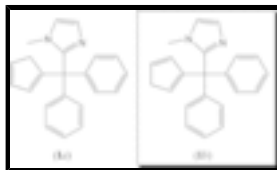


Fig. 2. Isomers (**Ia**) and (**Ib**).

2-[(Cyclopenta-1,3-dien-2-yl)diphenylmethyl]-1-methyl-1*H*-imidazole*Crystal data*

$C_{22}H_{20}N_2$	$F(000) = 664$
$M_r = 312.40$	$D_x = 1.220 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: P 2ac 2ab	Cell parameters from 5639 reflections
$a = 10.563 (5) \text{ \AA}$	$\theta = 2.3\text{--}28.0^\circ$
$b = 10.603 (5) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$c = 15.185 (7) \text{ \AA}$	$T = 295 \text{ K}$
$V = 1700.6 (14) \text{ \AA}^3$	Prism, colorless
$Z = 4$	$0.20 \times 0.05 \times 0.05 \text{ mm}$

Data collection

Bruker SMART APEXII diffractometer	1922 independent reflections
Radiation source: fine-focus sealed tube graphite	1543 reflections with $I > 2\sigma(I)$
Detector resolution: $8.333 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.033$
ϕ and ω scans	$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 1996)	$h = -12 \rightarrow 13$
$T_{\text{min}} = 0.986$, $T_{\text{max}} = 0.996$	$k = -13 \rightarrow 13$
8785 measured reflections	$l = -10 \rightarrow 18$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.120$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0736P)^2 + 0.110P]$
1922 reflections	where $P = (F_o^2 + 2F_c^2)/3$
218 parameters	$(\Delta/\sigma)_{\text{max}} < 0.001$
5 restraints	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.11 \text{ e \AA}^{-3}$

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

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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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.1464 (2)	0.6352 (2)	0.49551 (13)	0.0578 (6)
N2	0.2989 (2)	0.73895 (18)	0.56444 (14)	0.0588 (6)
C1	0.2381 (2)	0.6304 (2)	0.55926 (15)	0.0460 (5)
C2	0.1512 (3)	0.7552 (3)	0.4606 (2)	0.0725 (8)
H2	0.0996	0.7876	0.4164	0.087*
C3	0.2442 (3)	0.8160 (3)	0.5025 (2)	0.0756 (9)
H3	0.2685	0.8987	0.4914	0.091*
C4	0.0623 (3)	0.5371 (3)	0.4627 (2)	0.0785 (9)
H4C	0.0274	0.4912	0.5115	0.118*
H4A	0.1091	0.4805	0.4256	0.118*
H4B	-0.0050	0.5749	0.4294	0.118*
C5	0.2651 (2)	0.5151 (2)	0.61653 (14)	0.0412 (5)
C11	0.3890 (2)	0.5365 (2)	0.66661 (15)	0.0457 (5)
C12	0.4947 (2)	0.4661 (3)	0.66347 (17)	0.0561 (6)
H12	0.5063	0.3969	0.6267	0.067*
C13	0.5894 (3)	0.5134 (3)	0.7259 (2)	0.0778 (8)
H13A	0.6656	0.5395	0.6954	0.093*
H13B	0.6113	0.4489	0.7686	0.093*
C14	0.5283 (3)	0.6217 (3)	0.76939 (19)	0.0790 (8)
H14	0.5641	0.6719	0.8130	0.095*
C15	0.4055 (3)	0.6363 (2)	0.73309 (18)	0.0616 (6)
H15	0.3464	0.6974	0.7484	0.074*
C21	0.1592 (2)	0.4951 (2)	0.68599 (14)	0.0443 (5)
C22	0.0450 (2)	0.5593 (3)	0.68353 (18)	0.0580 (7)
H22	0.0312	0.6208	0.6409	0.070*
C23	-0.0488 (3)	0.5321 (3)	0.7443 (2)	0.0754 (9)
H23	-0.1253	0.5754	0.7420	0.090*
C24	-0.0307 (3)	0.4438 (3)	0.8067 (2)	0.0807 (10)
H24	-0.0959	0.4237	0.8453	0.097*
C25	0.0844 (3)	0.3828 (3)	0.81354 (18)	0.0688 (8)
H25	0.0980	0.3246	0.8584	0.083*
C26	0.1786 (3)	0.4083 (2)	0.75399 (16)	0.0562 (6)
H26	0.2562	0.3675	0.7589	0.067*
C31	0.2772 (2)	0.3977 (2)	0.55630 (14)	0.0430 (5)
C32	0.3568 (2)	0.4048 (2)	0.48296 (16)	0.0537 (6)
H32	0.4036	0.4777	0.4728	0.064*
C33	0.3665 (3)	0.3046 (3)	0.42553 (18)	0.0650 (7)
H33	0.4188	0.3112	0.3765	0.078*

C34	0.3004 (3)	0.1959 (3)	0.4395 (2)	0.0713 (8)
H34	0.3082	0.1288	0.4004	0.086*
C35	0.2234 (3)	0.1862 (3)	0.5106 (2)	0.0707 (8)
H35	0.1785	0.1120	0.5204	0.085*
C36	0.2110 (2)	0.2860 (2)	0.56874 (17)	0.0536 (6)
H36	0.1575	0.2780	0.6170	0.064*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0615 (14)	0.0586 (12)	0.0533 (11)	0.0116 (11)	-0.0076 (11)	0.0052 (11)
N2	0.0645 (14)	0.0426 (10)	0.0692 (13)	-0.0012 (10)	0.0033 (12)	0.0092 (10)
C1	0.0457 (13)	0.0445 (12)	0.0479 (11)	0.0063 (11)	0.0039 (10)	0.0027 (10)
C2	0.085 (2)	0.0667 (17)	0.0661 (16)	0.0255 (17)	-0.0024 (17)	0.0166 (16)
C3	0.090 (2)	0.0497 (14)	0.087 (2)	0.0101 (16)	0.0023 (19)	0.0205 (15)
C4	0.073 (2)	0.082 (2)	0.0800 (19)	0.0081 (17)	-0.0301 (17)	-0.0082 (18)
C5	0.0389 (11)	0.0402 (11)	0.0445 (11)	0.0008 (10)	0.0002 (9)	0.0015 (9)
C11	0.0440 (12)	0.0439 (12)	0.0491 (12)	-0.0060 (10)	0.0033 (10)	0.0054 (11)
C12	0.0452 (13)	0.0594 (14)	0.0638 (14)	0.0000 (12)	-0.0020 (11)	-0.0009 (12)
C13	0.0457 (13)	0.103 (2)	0.0843 (19)	-0.0100 (13)	-0.0138 (13)	0.0156 (17)
C14	0.0928 (19)	0.0814 (18)	0.0630 (16)	-0.0413 (15)	-0.0177 (15)	0.0016 (14)
C15	0.0736 (15)	0.0530 (13)	0.0584 (14)	-0.0067 (13)	0.0016 (13)	-0.0091 (14)
C21	0.0438 (12)	0.0425 (11)	0.0467 (11)	-0.0046 (10)	0.0029 (10)	-0.0078 (11)
C22	0.0454 (13)	0.0666 (16)	0.0621 (15)	0.0010 (12)	0.0014 (12)	-0.0066 (15)
C23	0.0467 (14)	0.095 (2)	0.085 (2)	-0.0048 (15)	0.0135 (15)	-0.017 (2)
C24	0.073 (2)	0.092 (2)	0.077 (2)	-0.0333 (19)	0.0245 (18)	-0.0198 (19)
C25	0.083 (2)	0.0648 (17)	0.0583 (15)	-0.0207 (16)	0.0146 (15)	0.0004 (14)
C26	0.0612 (15)	0.0536 (13)	0.0539 (13)	-0.0056 (12)	0.0048 (12)	-0.0029 (12)
C31	0.0394 (11)	0.0423 (11)	0.0474 (11)	0.0039 (10)	-0.0033 (10)	-0.0012 (9)
C32	0.0508 (14)	0.0553 (13)	0.0552 (13)	0.0013 (12)	0.0043 (12)	-0.0002 (12)
C33	0.0619 (16)	0.0782 (18)	0.0550 (14)	0.0102 (15)	0.0035 (13)	-0.0121 (15)
C34	0.0768 (19)	0.0639 (17)	0.0734 (18)	0.0084 (16)	-0.0021 (16)	-0.0177 (16)
C35	0.081 (2)	0.0531 (14)	0.0779 (18)	-0.0053 (15)	-0.0032 (18)	-0.0113 (14)
C36	0.0538 (14)	0.0488 (13)	0.0583 (14)	-0.0027 (11)	0.0019 (13)	-0.0059 (11)

Geometric parameters (\AA , $^\circ$)

N1—C1	1.370 (3)	C15—H15	0.9300
N1—C2	1.380 (3)	C21—C22	1.386 (3)
N1—C4	1.456 (4)	C21—C26	1.398 (3)
N2—C1	1.321 (3)	C22—C23	1.384 (4)
N2—C3	1.373 (3)	C22—H22	0.9300
C1—C5	1.527 (3)	C23—C24	1.346 (5)
C2—C3	1.336 (4)	C23—H23	0.9300
C2—H2	0.9300	C24—C25	1.381 (5)
C3—H3	0.9300	C24—H24	0.9300
C4—H4C	0.9600	C25—C26	1.372 (4)
C4—H4A	0.9600	C25—H25	0.9300
C4—H4B	0.9600	C26—H26	0.9300

supplementary materials

C5—C11	1.530 (3)	C31—C36	1.388 (3)
C5—C31	1.550 (3)	C31—C32	1.398 (3)
C5—C21	1.552 (3)	C32—C33	1.378 (3)
C11—C12	1.344 (3)	C32—H32	0.9300
C11—C15	1.473 (3)	C33—C34	1.364 (4)
C12—C13	1.466 (4)	C33—H33	0.9300
C12—H12	0.9300	C34—C35	1.357 (4)
C13—C14	1.474 (5)	C34—H34	0.9300
C13—H13A	0.9700	C35—C36	1.384 (4)
C13—H13B	0.9700	C35—H35	0.9300
C14—C15	1.417 (4)	C36—H36	0.9300
C14—H14	0.9300		
C1—N1—C2	106.3 (2)	C14—C15—C11	107.3 (3)
C1—N1—C4	130.2 (2)	C14—C15—H15	126.4
C2—N1—C4	123.4 (2)	C11—C15—H15	126.4
C1—N2—C3	105.8 (2)	C22—C21—C26	118.1 (2)
N2—C1—N1	110.7 (2)	C22—C21—C5	122.9 (2)
N2—C1—C5	124.9 (2)	C26—C21—C5	119.1 (2)
N1—C1—C5	124.4 (2)	C23—C22—C21	120.2 (3)
C3—C2—N1	106.8 (3)	C23—C22—H22	119.9
C3—C2—H2	126.6	C21—C22—H22	119.9
N1—C2—H2	126.6	C24—C23—C22	120.8 (3)
C2—C3—N2	110.5 (3)	C24—C23—H23	119.6
C2—C3—H3	124.8	C22—C23—H23	119.6
N2—C3—H3	124.8	C23—C24—C25	120.3 (3)
N1—C4—H4C	109.5	C23—C24—H24	119.8
N1—C4—H4A	109.5	C25—C24—H24	119.8
H4C—C4—H4A	109.5	C26—C25—C24	119.8 (3)
N1—C4—H4B	109.5	C26—C25—H25	120.1
H4C—C4—H4B	109.5	C24—C25—H25	120.1
H4A—C4—H4B	109.5	C25—C26—C21	120.7 (3)
C1—C5—C11	108.92 (18)	C25—C26—H26	119.7
C1—C5—C31	108.80 (16)	C21—C26—H26	119.7
C11—C5—C31	110.03 (17)	C36—C31—C32	117.2 (2)
C1—C5—C21	111.18 (18)	C36—C31—C5	124.3 (2)
C11—C5—C21	107.37 (17)	C32—C31—C5	118.42 (19)
C31—C5—C21	110.53 (17)	C33—C32—C31	120.4 (2)
C12—C11—C15	108.9 (2)	C33—C32—H32	119.8
C12—C11—C5	127.6 (2)	C31—C32—H32	119.8
C15—C11—C5	123.3 (2)	C34—C33—C32	121.0 (3)
C11—C12—C13	110.7 (3)	C34—C33—H33	119.5
C11—C12—H12	124.6	C32—C33—H33	119.5
C13—C12—H12	124.6	C35—C34—C33	119.7 (3)
C12—C13—C14	104.9 (2)	C35—C34—H34	120.2
C12—C13—H13A	110.8	C33—C34—H34	120.2
C14—C13—H13A	110.8	C34—C35—C36	120.4 (3)
C12—C13—H13B	110.8	C34—C35—H35	119.8
C14—C13—H13B	110.8	C36—C35—H35	119.8
H13A—C13—H13B	108.8	C35—C36—C31	121.2 (2)

C15—C14—C13	108.1 (2)	C35—C36—H36	119.4
C15—C14—H14	125.9	C31—C36—H36	119.4
C13—C14—H14	125.9		
C3—N2—C1—N1	0.3 (3)	C1—C5—C21—C22	11.7 (3)
C3—N2—C1—C5	-179.4 (2)	C11—C5—C21—C22	130.7 (2)
C2—N1—C1—N2	-0.8 (3)	C31—C5—C21—C22	-109.2 (2)
C4—N1—C1—N2	175.5 (3)	C1—C5—C21—C26	-168.7 (2)
C2—N1—C1—C5	178.9 (2)	C11—C5—C21—C26	-49.7 (2)
C4—N1—C1—C5	-4.8 (4)	C31—C5—C21—C26	70.3 (2)
C1—N1—C2—C3	0.9 (3)	C26—C21—C22—C23	-3.4 (4)
C4—N1—C2—C3	-175.7 (3)	C5—C21—C22—C23	176.2 (2)
N1—C2—C3—N2	-0.7 (3)	C21—C22—C23—C24	0.2 (4)
C1—N2—C3—C2	0.2 (3)	C22—C23—C24—C25	3.0 (5)
N2—C1—C5—C11	-11.2 (3)	C23—C24—C25—C26	-3.0 (4)
N1—C1—C5—C11	169.1 (2)	C24—C25—C26—C21	-0.3 (4)
N2—C1—C5—C31	-131.2 (2)	C22—C21—C26—C25	3.5 (3)
N1—C1—C5—C31	49.2 (3)	C5—C21—C26—C25	-176.1 (2)
N2—C1—C5—C21	106.9 (3)	C1—C5—C31—C36	-128.3 (2)
N1—C1—C5—C21	-72.8 (2)	C11—C5—C31—C36	112.4 (2)
C1—C5—C11—C12	-120.6 (3)	C21—C5—C31—C36	-6.0 (3)
C31—C5—C11—C12	-1.5 (3)	C1—C5—C31—C32	49.8 (3)
C21—C5—C11—C12	118.9 (3)	C11—C5—C31—C32	-69.5 (2)
C1—C5—C11—C15	64.5 (3)	C21—C5—C31—C32	172.10 (19)
C31—C5—C11—C15	-176.32 (19)	C36—C31—C32—C33	0.9 (3)
C21—C5—C11—C15	-56.0 (3)	C5—C31—C32—C33	-177.3 (2)
C15—C11—C12—C13	-0.6 (3)	C31—C32—C33—C34	-1.0 (4)
C5—C11—C12—C13	-176.1 (2)	C32—C33—C34—C35	0.4 (5)
C11—C12—C13—C14	0.7 (3)	C33—C34—C35—C36	0.3 (5)
C12—C13—C14—C15	-0.5 (3)	C34—C35—C36—C31	-0.4 (4)
C13—C14—C15—C11	0.1 (3)	C32—C31—C36—C35	-0.2 (4)
C12—C11—C15—C14	0.3 (3)	C5—C31—C36—C35	177.9 (2)
C5—C11—C15—C14	176.0 (2)		

Fig. 1

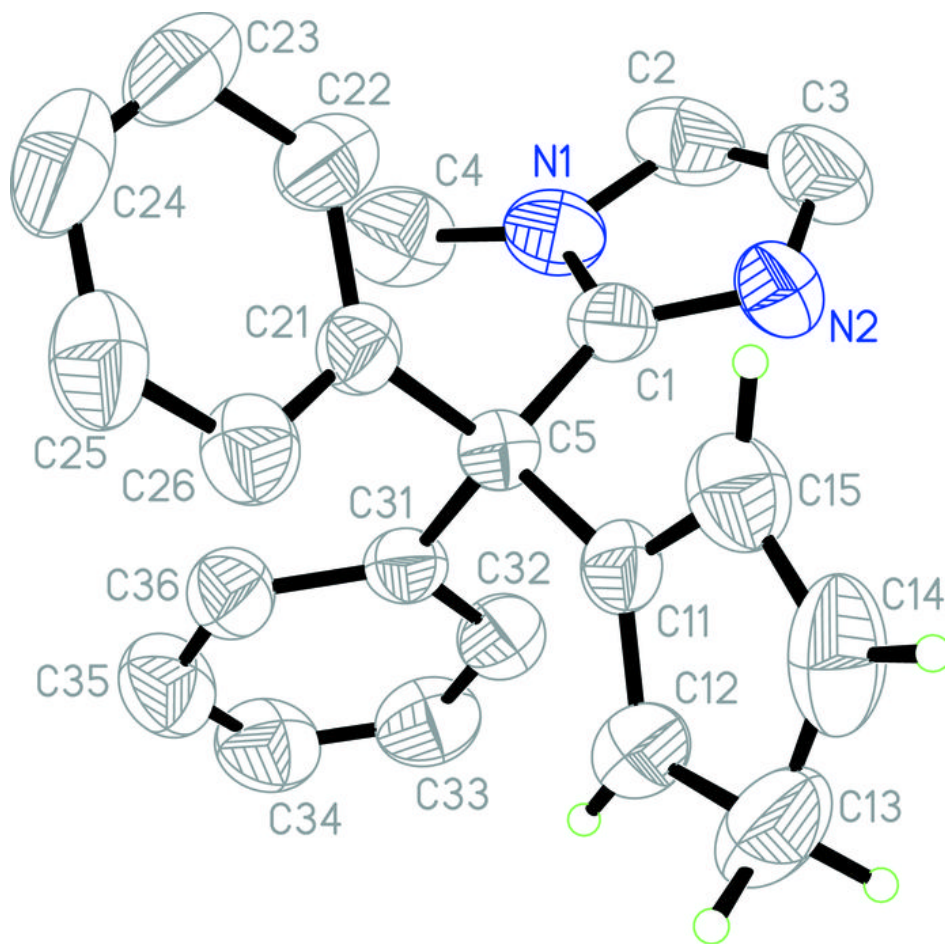


Fig. 2

