Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,6-Di-*tert*-butyl-4-[(*N*-methylanilino)-methyl]phenol

Tao Zeng[a]* and Ju-Feng Sun[b]

[a]Chemistry and Biology College, Yantai University, Yantai 264005, People's Republic of China, and [b]Binzhou Medical University, Yantai 264003, People's Republic of China
Correspondence e-mail: zengtaotj@126.com

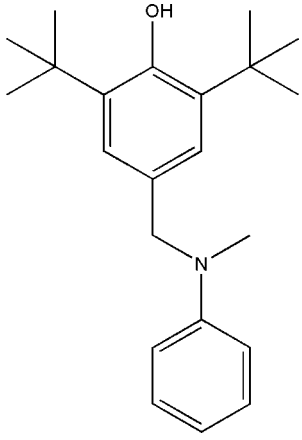
Received 21 November 2007; accepted 24 December 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.055; wR factor = 0.165; data-to-parameter ratio = 13.5.

The title compound, $C_{22}H_{31}NO$, has been synthesized from 4-bromomethyl-2,6-di-*tert*-butylphenol and *N*-methylaniline. There are two independent molecules in the asymmetric unit. The aniline ring in each of the independent molecules was found to be disordered, with site occupation factors 0.621 (10)/0.379 (10) and 0.605 (10)/0.395 (10).

Related literature

For related literature, see: Yamazaki & Seguchi (1997); Rieker *et al.* (1968).



Experimental

Crystal data

$C_{22}H_{31}NO$
M_r = 325.48
Triclinic, $P\overline{1}$
a = 9.535 (7) Å
b = 12.106 (9) Å
c = 17.959 (14) Å
α = 109.561 (13)°
β = 92.691 (13)°
γ = 90.394 (14)°
V = 1951 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 294 (2) K
0.24 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.984, T_{max} = 0.987
10188 measured reflections
6861 independent reflections
3684 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.165
S = 1.02
6861 reflections
510 parameters
472 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge financial support from the Start Foundation for Doctors of Yantai University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2054).

References

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Yamazaki, T. & Seguchi, T. (1997). *J. Polym. Sci. A: Polym. Chem.* **35**, 2431–2439.

  doi:10.1107/S1600536807068365

supplementary materials

Acta Cryst. (2008). E**64**, o410 [**doi:10.1107/S1600536807068365**]

2,6-Di-*tert*-butyl-4-[(*N*-methylanilino)methyl]phenol

T. Zeng and J.-F. Sun

Comment

Hindered phenol antioxidants are widely used in polymers and lubricants. They protect polymers by increasing both their process stability and long-term stability against oxidative degradation (Yamazaki and Seguchi, 1997).

There are two independent molecules in the crystal structure. Both of the two aniline rings in the two independent molecules were found disordered.

The bond lengths and angles of the crystal are within normal ranges and selected values are given in Table 1.

Experimental

The 4-bromomethyl-2,6-di-*tert*-butyl-phenol was synthesized according to the method described by Rieker *et al.* (1968). *N*-methylaniline (3.21 g, 0.03 mol) and 4-bromomethyl-2,6-di-*tert*-butyl-phenol (9.0 g, 0.03 mol) were added, with stirring to THF(40 ml) at 278 K. The reaction mixture was stirred at 278–283 K for a further 2 h. then a solution of Na_2CO_3(1.60 g, 0.015 mol) in water (10 ml) was added. The solvent THF was evaporated under reduced pressure and the product was extracted by diethyl ether. The product (7.95 g) was obtained in a yield of 81.4%. Suitable crystals were obtained by slow evaporation of a mixture of ethyl acetate and methanol.

Refinement

The phenyl goups of amines were disordered in tow positions with occupy fators 0.605 (10)/0.395 (10) and 0.621 (10)/ 0.379 (10) respectively. The disordered phenyl groups were constrained to a hexagon with the C—C distances 1.39 A. The H atoms of O—H were restrained on their parent atoms with O—H restrained 0.83 and 0.84Å at positions based on difference map peaks close to the parent O atoms.. In absence of significant anomalous dispersion effects, Friedel-pair reflections were merged prior to refinement. All H other atoms were positioned geometrically and refined using a riding model, in the range of 0.93–0.97 Å, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

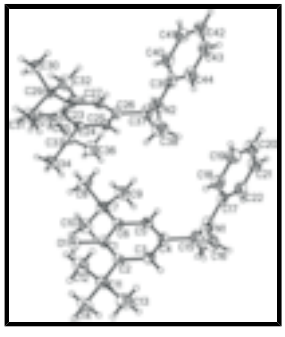

Fig. 1. The two crystallygraphically independent molecules in the structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. To be clarity, the disordered part of phenyl rings (C17' to C22' and C39' to C44') were omitted.

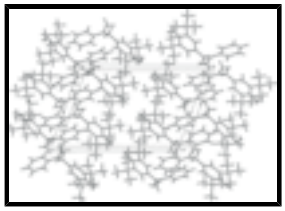

Fig. 2. The packing diagram of (I).

2,6-Di-*tert*-butyl-4-[(*N*-methylanilino)methyl]phenol

Crystal data

$C_{22}H_{31}NO$	$Z = 4$
$M_r = 325.48$	$F_{000} = 712$
Triclinic, $P\overline{1}$	$D_x = 1.108$ Mg m^{-3}
$a = 9.535$ (7) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 12.106$ (9) Å	Cell parameters from 2492 reflections
$c = 17.959$ (14) Å	$\theta = 2.4$–23.5°
$\alpha = 109.561$ (13)°	$\mu = 0.07$ mm^{-1}
$\beta = 92.691$ (13)°	$T = 294$ (2) K
$\gamma = 90.394$ (14)°	Bolck, colourless
$V = 1951$ (3) Å^3	0.24 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer	6861 independent reflections
Radiation source: fine-focus sealed tube	3684 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.026$
$T = 294$(2) K	$\theta_{max} = 25.0^\circ$
φ and ω scans	$\theta_{min} = 1.2^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 11$
$T_{min} = 0.984$, $T_{max} = 0.987$	$k = -14 \rightarrow 9$
10188 measured reflections	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.054$	H-atom parameters constrained
$wR(F^2) = 0.165$	$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.5357P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.004$
6861 reflections	$\Delta\rho_{max} = 0.25$ e Å^{-3}
510 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}

472 restraints

Primary atom site location: structure-invariant direct methods

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0156 (16)

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}	Occ. (<1)
O1	1.02388 (18)	0.41746 (16)	0.41523 (10)	0.0651 (5)	
H1	0.9788	0.3812	0.4393	0.078*	
O2	0.55834 (18)	0.91928 (16)	0.41754 (10)	0.0643 (5)	
H2	0.6027	0.8831	0.4428	0.077*	
N1	0.8263 (3)	0.0870 (2)	0.06541 (13)	0.0699 (7)	
N2	0.6869 (3)	0.5888 (2)	0.06764 (13)	0.0707 (7)	
C1	1.0031 (2)	0.3369 (2)	0.34025 (14)	0.0432 (6)	
C2	1.1039 (2)	0.2513 (2)	0.31399 (14)	0.0414 (6)	
C3	1.0841 (2)	0.1748 (2)	0.23807 (15)	0.0469 (6)	
H3	1.1494	0.1166	0.2191	0.056*	
C4	0.9719 (3)	0.1805 (2)	0.18891 (14)	0.0485 (6)	
C5	0.8740 (2)	0.2641 (2)	0.21751 (14)	0.0464 (6)	
H5	0.7974	0.2677	0.1844	0.056*	
C6	0.8845 (2)	0.3435 (2)	0.29364 (14)	0.0417 (6)	
C7	0.7697 (3)	0.4322 (2)	0.32466 (15)	0.0514 (7)	
C8	0.8241 (3)	0.5576 (3)	0.3400 (2)	0.0804 (10)	
H8A	0.8574	0.5641	0.2920	0.121*	
H8B	0.8996	0.5760	0.3798	0.121*	
H8C	0.7494	0.6113	0.3579	0.121*	
C9	0.6445 (3)	0.4117 (3)	0.26502 (17)	0.0695 (8)	
H9A	0.5733	0.4672	0.2869	0.104*	
H9B	0.6072	0.3336	0.2534	0.104*	
H9C	0.6744	0.4215	0.2173	0.104*	
C10	0.7112 (3)	0.4194 (3)	0.39917 (17)	0.0714 (9)	
H10A	0.6362	0.4731	0.4163	0.107*	
H10B	0.7844	0.4364	0.4403	0.107*	
H10C	0.6763	0.3406	0.3878	0.107*	
C11	1.2294 (2)	0.2416 (2)	0.36724 (15)	0.0525 (7)	
C12	1.3197 (3)	0.3536 (3)	0.39181 (18)	0.0712 (9)	

H12A	1.3991	0.3460	0.4245	0.107*	
H12B	1.2651	0.4188	0.4209	0.107*	
H12C	1.3519	0.3666	0.3455	0.107*	
C13	1.3231 (3)	0.1410 (3)	0.32503 (19)	0.0792 (9)	
H13A	1.3605	0.1549	0.2802	0.119*	
H13B	1.2690	0.0689	0.3075	0.119*	
H13C	1.3988	0.1358	0.3608	0.119*	
C14	1.1797 (3)	0.2174 (3)	0.43998 (17)	0.0742 (9)	
H14A	1.1290	0.1438	0.4237	0.111*	
H14B	1.1194	0.2789	0.4682	0.111*	
H14C	1.2595	0.2145	0.4738	0.111*	
C15	0.9607 (3)	0.0940 (3)	0.10617 (16)	0.0725 (9)	
H15A	1.0311	0.1148	0.0756	0.087*	
H15B	0.9824	0.0170	0.1083	0.087*	
C16	0.7267 (4)	0.0108 (4)	0.0818 (2)	0.1105 (13)	
H16A	0.6340	0.0406	0.0798	0.166*	
H16B	0.7482	0.0067	0.1336	0.166*	
H16C	0.7308	−0.0661	0.0432	0.166*	
C17	0.8148 (8)	0.1256 (7)	0.0005 (3)	0.0610 (17)	0.605 (10)
C18	0.9201 (7)	0.1988 (6)	−0.0091 (3)	0.0751 (17)	0.605 (10)
H18A	0.9957	0.2221	0.0278	0.090*	0.605 (10)
C19	0.9126 (7)	0.2373 (5)	−0.0739 (3)	0.0833 (18)	0.605 (10)
H19A	0.9831	0.2863	−0.0804	0.100*	0.605 (10)
C20	0.7997 (8)	0.2025 (6)	−0.1291 (3)	0.084 (2)	0.605 (10)
H20A	0.7947	0.2282	−0.1725	0.101*	0.605 (10)
C21	0.6944 (6)	0.1292 (7)	−0.1195 (3)	0.083 (2)	0.605 (10)
H21A	0.6189	0.1059	−0.1564	0.100*	0.605 (10)
C22	0.7019 (7)	0.0908 (6)	−0.0547 (4)	0.0725 (16)	0.605 (10)
H22A	0.6314	0.0418	−0.0482	0.087*	0.605 (10)
C17'	0.8006 (13)	0.1132 (11)	−0.0028 (5)	0.061 (2)	0.395 (10)
C18'	0.8780 (11)	0.1974 (10)	−0.0213 (5)	0.071 (2)	0.395 (10)
H18B	0.9514	0.2384	0.0129	0.086*	0.395 (10)
C19'	0.8455 (12)	0.2203 (8)	−0.0909 (5)	0.085 (2)	0.395 (10)
H19B	0.8972	0.2766	−0.1033	0.102*	0.395 (10)
C20'	0.7357 (12)	0.1589 (9)	−0.1420 (4)	0.079 (3)	0.395 (10)
H20B	0.7140	0.1742	−0.1886	0.095*	0.395 (10)
C21'	0.6584 (9)	0.0748 (10)	−0.1235 (5)	0.084 (2)	0.395 (10)
H21B	0.5849	0.0337	−0.1577	0.100*	0.395 (10)
C22'	0.6909 (11)	0.0519 (10)	−0.0539 (5)	0.073 (2)	0.395 (10)
H22B	0.6391	−0.0044	−0.0415	0.087*	0.395 (10)
C23	0.5635 (2)	0.8380 (2)	0.34270 (14)	0.0435 (6)	
C24	0.4570 (2)	0.7526 (2)	0.31693 (14)	0.0417 (6)	
C25	0.4620 (2)	0.6758 (2)	0.24087 (14)	0.0459 (6)	
H25	0.3923	0.6177	0.2219	0.055*	
C26	0.5657 (3)	0.6814 (2)	0.19177 (14)	0.0483 (6)	
C27	0.6696 (3)	0.7653 (2)	0.22026 (15)	0.0489 (6)	
H27	0.7406	0.7688	0.1873	0.059*	
C28	0.6733 (2)	0.8452 (2)	0.29632 (14)	0.0430 (6)	
C29	0.7944 (3)	0.9346 (2)	0.32725 (15)	0.0512 (7)	

C30	0.7421 (3)	1.0594 (3)	0.3435 (2)	0.0813 (10)	
H30A	0.8202	1.1140	0.3602	0.122*	
H30B	0.6761	1.0769	0.3844	0.122*	
H30C	0.6972	1.0655	0.2961	0.122*	
C31	0.8662 (3)	0.9218 (3)	0.40190 (16)	0.0665 (8)	
H31A	0.8965	0.8427	0.3907	0.100*	
H31B	0.8011	0.9401	0.4433	0.100*	
H31C	0.9460	0.9747	0.4185	0.100*	
C32	0.9081 (3)	0.9143 (3)	0.26745 (18)	0.0716 (9)	
H32A	0.8691	0.9226	0.2194	0.107*	
H32B	0.9435	0.8368	0.2565	0.107*	
H32C	0.9833	0.9709	0.2888	0.107*	
C33	0.3407 (2)	0.7418 (2)	0.36998 (15)	0.0516 (7)	
C34	0.4034 (3)	0.7176 (3)	0.44301 (17)	0.0764 (9)	
H34A	0.3291	0.7093	0.4752	0.115*	
H34B	0.4649	0.7818	0.4731	0.115*	
H34C	0.4554	0.6467	0.4265	0.115*	
C35	0.2552 (3)	0.8536 (3)	0.39465 (19)	0.0754 (9)	
H35A	0.2143	0.8661	0.3483	0.113*	
H35B	0.3157	0.9192	0.4235	0.113*	
H35C	0.1821	0.8457	0.4276	0.113*	
C36	0.2386 (3)	0.6414 (3)	0.32762 (19)	0.0809 (10)	
H36A	0.1943	0.6551	0.2825	0.121*	
H36B	0.1684	0.6363	0.3630	0.121*	
H36C	0.2884	0.5693	0.3105	0.121*	
C37	0.5604 (3)	0.5946 (3)	0.10895 (16)	0.0724 (9)	
H37A	0.5391	0.5174	0.1113	0.087*	
H37B	0.4839	0.6147	0.0786	0.087*	
C38	0.7901 (4)	0.5130 (4)	0.0833 (2)	0.1084 (13)	
H38A	0.7766	0.4356	0.0453	0.163*	
H38B	0.7807	0.5101	0.1356	0.163*	
H38C	0.8823	0.5425	0.0795	0.163*	
C39	0.6836 (7)	0.6260 (6)	0.0019 (3)	0.0625 (17)	0.621 (10)
C40	0.5768 (7)	0.6981 (6)	−0.0081 (3)	0.0727 (16)	0.621 (10)
H40A	0.5086	0.7217	0.0290	0.087*	0.621 (10)
C41	0.5720 (7)	0.7351 (5)	−0.0736 (3)	0.0802 (17)	0.621 (10)
H41A	0.5005	0.7834	−0.0803	0.096*	0.621 (10)
C42	0.6739 (7)	0.6999 (6)	−0.1291 (3)	0.0838 (19)	0.621 (10)
H42A	0.6707	0.7246	−0.1729	0.101*	0.621 (10)
C43	0.7807 (6)	0.6278 (7)	−0.1191 (3)	0.0820 (19)	0.621 (10)
H43A	0.8490	0.6042	−0.1562	0.098*	0.621 (10)
C44	0.7856 (6)	0.5908 (6)	−0.0536 (3)	0.0715 (15)	0.621 (10)
H44A	0.8570	0.5425	−0.0470	0.086*	0.621 (10)
C39'	0.7001 (13)	0.6128 (11)	−0.0017 (5)	0.063 (2)	0.379 (10)
C40'	0.6191 (12)	0.6958 (10)	−0.0204 (5)	0.073 (2)	0.379 (10)
H40B	0.5530	0.7371	0.0142	0.088*	0.379 (10)
C41'	0.6366 (12)	0.7171 (8)	−0.0909 (5)	0.083 (3)	0.379 (10)
H41B	0.5824	0.7726	−0.1034	0.100*	0.379 (10)
C42'	0.7353 (12)	0.6554 (9)	−0.1426 (4)	0.077 (3)	0.379 (10)

H42B	0.7470	0.6696	−0.1897	0.093*	0.379 (10)
C43'	0.8164 (9)	0.5724 (10)	−0.1239 (5)	0.085 (2)	0.379 (10)
H43B	0.8824	0.5311	−0.1584	0.101*	0.379 (10)
C44'	0.7988 (11)	0.5512 (10)	−0.0534 (6)	0.076 (2)	0.379 (10)
H44B	0.8530	0.4956	−0.0409	0.091*	0.379 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0643 (12)	0.0726 (13)	0.0444 (11)	−0.0033 (10)	−0.0048 (9)	0.0022 (10)
O2	0.0628 (12)	0.0703 (13)	0.0475 (11)	0.0003 (9)	0.0070 (9)	0.0029 (10)
N1	0.0725 (17)	0.0811 (18)	0.0475 (15)	−0.0112 (13)	−0.0130 (12)	0.0124 (13)
N2	0.0778 (17)	0.0805 (18)	0.0485 (15)	0.0116 (14)	0.0158 (12)	0.0127 (13)
C1	0.0411 (14)	0.0476 (15)	0.0379 (14)	−0.0039 (11)	−0.0004 (11)	0.0109 (12)
C2	0.0350 (13)	0.0479 (15)	0.0429 (15)	−0.0026 (11)	−0.0004 (11)	0.0177 (12)
C3	0.0432 (14)	0.0455 (15)	0.0516 (16)	0.0038 (11)	0.0043 (12)	0.0155 (13)
C4	0.0471 (15)	0.0515 (16)	0.0428 (15)	0.0014 (12)	0.0004 (12)	0.0106 (12)
C5	0.0421 (14)	0.0568 (16)	0.0414 (15)	0.0018 (12)	−0.0043 (11)	0.0187 (13)
C6	0.0383 (13)	0.0447 (14)	0.0428 (15)	0.0004 (10)	0.0010 (11)	0.0160 (12)
C7	0.0469 (15)	0.0499 (16)	0.0571 (17)	0.0059 (12)	0.0027 (12)	0.0175 (13)
C8	0.082 (2)	0.055 (2)	0.102 (3)	0.0074 (16)	0.0060 (19)	0.0233 (18)
C9	0.0525 (17)	0.082 (2)	0.075 (2)	0.0202 (15)	−0.0028 (15)	0.0274 (17)
C10	0.0568 (17)	0.090 (2)	0.067 (2)	0.0140 (16)	0.0169 (15)	0.0239 (17)
C11	0.0417 (14)	0.0630 (18)	0.0549 (17)	−0.0010 (13)	−0.0057 (12)	0.0238 (14)
C12	0.0488 (16)	0.086 (2)	0.080 (2)	−0.0135 (15)	−0.0104 (15)	0.0326 (18)
C13	0.0593 (18)	0.092 (2)	0.085 (2)	0.0227 (17)	−0.0095 (16)	0.0297 (19)
C14	0.0699 (19)	0.097 (2)	0.068 (2)	−0.0063 (17)	−0.0130 (16)	0.0463 (19)
C15	0.074 (2)	0.079 (2)	0.0503 (18)	0.0122 (16)	−0.0069 (15)	0.0034 (16)
C16	0.118 (3)	0.121 (3)	0.095 (3)	−0.034 (3)	−0.012 (2)	0.042 (2)
C17	0.065 (3)	0.058 (3)	0.044 (3)	0.014 (3)	−0.002 (2)	−0.004 (3)
C18	0.082 (4)	0.065 (3)	0.069 (3)	0.013 (3)	0.001 (3)	0.010 (3)
C19	0.087 (4)	0.080 (3)	0.082 (3)	0.015 (3)	0.004 (3)	0.026 (3)
C20	0.095 (4)	0.087 (4)	0.068 (3)	0.014 (3)	−0.005 (3)	0.024 (3)
C21	0.089 (4)	0.088 (4)	0.069 (3)	0.018 (3)	−0.004 (3)	0.023 (3)
C22	0.073 (3)	0.079 (4)	0.057 (3)	0.012 (3)	−0.003 (2)	0.012 (3)
C17'	0.071 (4)	0.055 (4)	0.050 (4)	0.013 (3)	0.005 (3)	0.007 (3)
C18'	0.080 (4)	0.063 (4)	0.066 (4)	0.013 (4)	0.002 (3)	0.015 (3)
C19'	0.091 (5)	0.077 (4)	0.082 (4)	0.011 (4)	0.001 (4)	0.022 (4)
C20'	0.091 (5)	0.076 (4)	0.075 (4)	0.016 (4)	0.001 (4)	0.033 (4)
C21'	0.094 (4)	0.086 (5)	0.066 (4)	0.023 (4)	−0.001 (3)	0.018 (4)
C22'	0.078 (4)	0.077 (5)	0.052 (3)	0.023 (4)	−0.007 (3)	0.008 (3)
C23	0.0429 (14)	0.0455 (15)	0.0373 (14)	0.0023 (11)	0.0005 (11)	0.0078 (12)
C24	0.0373 (13)	0.0456 (14)	0.0444 (15)	0.0035 (11)	0.0013 (11)	0.0181 (12)
C25	0.0416 (14)	0.0464 (15)	0.0471 (16)	−0.0054 (11)	−0.0049 (11)	0.0135 (13)
C26	0.0478 (15)	0.0522 (16)	0.0403 (15)	−0.0016 (12)	0.0005 (12)	0.0100 (12)
C27	0.0469 (15)	0.0572 (17)	0.0449 (16)	−0.0014 (12)	0.0102 (12)	0.0190 (13)
C28	0.0409 (14)	0.0441 (14)	0.0459 (15)	0.0013 (11)	0.0005 (11)	0.0179 (12)
C29	0.0488 (15)	0.0475 (16)	0.0569 (17)	−0.0064 (12)	0.0000 (12)	0.0177 (13)

C30	0.078 (2)	0.0521 (19)	0.111 (3)	−0.0090 (16)	−0.0032 (19)	0.0261 (18)
C31	0.0524 (16)	0.080 (2)	0.0643 (19)	−0.0107 (14)	−0.0093 (14)	0.0219 (16)
C32	0.0572 (17)	0.083 (2)	0.077 (2)	−0.0221 (15)	0.0062 (15)	0.0302 (18)
C33	0.0410 (14)	0.0634 (18)	0.0519 (16)	0.0011 (13)	0.0057 (12)	0.0208 (14)
C34	0.075 (2)	0.107 (3)	0.063 (2)	0.0053 (18)	0.0124 (16)	0.0478 (19)
C35	0.0522 (17)	0.089 (2)	0.084 (2)	0.0163 (16)	0.0162 (15)	0.0257 (19)
C36	0.0620 (19)	0.095 (2)	0.085 (2)	−0.0220 (17)	0.0149 (16)	0.0278 (19)
C37	0.075 (2)	0.080 (2)	0.0504 (18)	−0.0099 (16)	0.0108 (15)	0.0059 (16)
C38	0.119 (3)	0.119 (3)	0.096 (3)	0.041 (3)	0.019 (2)	0.046 (2)
C39	0.068 (3)	0.060 (3)	0.044 (3)	−0.012 (3)	0.004 (2)	−0.002 (2)
C40	0.081 (4)	0.064 (3)	0.065 (3)	−0.010 (3)	0.006 (3)	0.010 (2)
C41	0.088 (4)	0.072 (3)	0.080 (3)	−0.011 (3)	−0.002 (3)	0.025 (3)
C42	0.097 (4)	0.086 (4)	0.066 (3)	−0.016 (3)	0.013 (3)	0.022 (3)
C43	0.088 (4)	0.086 (4)	0.069 (3)	−0.018 (3)	0.008 (3)	0.023 (3)
C44	0.075 (3)	0.079 (4)	0.054 (3)	−0.010 (3)	0.010 (2)	0.013 (3)
C39'	0.074 (4)	0.057 (4)	0.052 (4)	−0.011 (3)	−0.001 (3)	0.010 (3)
C40'	0.083 (4)	0.063 (4)	0.067 (4)	−0.013 (4)	0.002 (3)	0.015 (3)
C41'	0.091 (5)	0.077 (4)	0.080 (4)	−0.008 (4)	0.004 (4)	0.025 (4)
C42'	0.088 (5)	0.079 (4)	0.072 (4)	−0.013 (4)	0.004 (4)	0.034 (4)
C43'	0.095 (4)	0.087 (5)	0.067 (4)	−0.022 (4)	0.008 (4)	0.019 (4)
C44'	0.083 (4)	0.081 (5)	0.055 (4)	−0.021 (4)	0.008 (3)	0.009 (4)

Geometric parameters (Å, °)

O1—C1	1.377 (3)	C19'—H19B	0.9300
O1—H1	0.8404	C20'—C21'	1.3900
O2—C23	1.378 (3)	C20'—H20B	0.9300
O2—H2	0.8327	C21'—C22'	1.3900
N1—C17'	1.376 (6)	C21'—H21B	0.9300
N1—C17	1.393 (4)	C22'—H22B	0.9300
N1—C16	1.427 (4)	C23—C28	1.388 (3)
N1—C15	1.433 (4)	C23—C24	1.391 (3)
N2—C39'	1.381 (6)	C24—C25	1.374 (3)
N2—C39	1.397 (4)	C24—C33	1.528 (3)
N2—C38	1.431 (4)	C25—C26	1.372 (3)
N2—C37	1.436 (4)	C25—H25	0.9300
C1—C6	1.394 (3)	C26—C27	1.366 (3)
C1—C2	1.397 (3)	C26—C37	1.505 (4)
C2—C3	1.371 (3)	C27—C28	1.383 (3)
C2—C11	1.526 (3)	C27—H27	0.9300
C3—C4	1.372 (3)	C28—C29	1.528 (3)
C3—H3	0.9300	C29—C32	1.525 (4)
C4—C5	1.367 (3)	C29—C31	1.531 (4)
C4—C15	1.503 (4)	C29—C30	1.531 (4)
C5—C6	1.381 (3)	C30—H30A	0.9600
C5—H5	0.9300	C30—H30B	0.9600
C6—C7	1.528 (3)	C30—H30C	0.9600
C7—C9	1.527 (4)	C31—H31A	0.9600
C7—C10	1.528 (4)	C31—H31B	0.9600

C7—C8	1.531 (4)	C31—H31C	0.9600
C8—H8A	0.9600	C32—H32A	0.9600
C8—H8B	0.9600	C32—H32B	0.9600
C8—H8C	0.9600	C32—H32C	0.9600
C9—H9A	0.9600	C33—C36	1.516 (4)
C9—H9B	0.9600	C33—C35	1.530 (4)
C9—H9C	0.9600	C33—C34	1.532 (4)
C10—H10A	0.9600	C34—H34A	0.9600
C10—H10B	0.9600	C34—H34B	0.9600
C10—H10C	0.9600	C34—H34C	0.9600
C11—C13	1.522 (4)	C35—H35A	0.9600
C11—C12	1.524 (4)	C35—H35B	0.9600
C11—C14	1.526 (4)	C35—H35C	0.9600
C12—H12A	0.9600	C36—H36A	0.9600
C12—H12B	0.9600	C36—H36B	0.9600
C12—H12C	0.9600	C36—H36C	0.9600
C13—H13A	0.9600	C37—H37A	0.9700
C13—H13B	0.9600	C37—H37B	0.9700
C13—H13C	0.9600	C38—H38A	0.9600
C14—H14A	0.9600	C38—H38B	0.9600
C14—H14B	0.9600	C38—H38C	0.9600
C14—H14C	0.9600	C39—C40	1.3900
C15—H15A	0.9700	C39—C44	1.3900
C15—H15B	0.9700	C40—C41	1.3900
C16—H16A	0.9600	C40—H40A	0.9300
C16—H16B	0.9600	C41—C42	1.3900
C16—H16C	0.9600	C41—H41A	0.9300
C17—C18	1.3900	C42—C43	1.3900
C17—C22	1.3900	C42—H42A	0.9300
C18—C19	1.3900	C43—C44	1.3900
C18—H18A	0.9300	C43—H43A	0.9300
C19—C20	1.3900	C44—H44A	0.9300
C19—H19A	0.9300	C39'—C40'	1.3900
C20—C21	1.3900	C39'—C44'	1.3900
C20—H20A	0.9300	C40'—C41'	1.3900
C21—C22	1.3900	C40'—H40B	0.9300
C21—H21A	0.9300	C41'—C42'	1.3900
C22—H22A	0.9300	C41'—H41B	0.9300
C17'—C18'	1.3900	C42'—C43'	1.3900
C17'—C22'	1.3900	C42'—H42B	0.9300
C18'—C19'	1.3900	C43'—C44'	1.3900
C18'—H18B	0.9300	C43'—H43B	0.9300
C19'—C20'	1.3900	C44'—H44B	0.9300
C1—O1—H1	97.6	C19'—C20'—H20B	120.0
C23—O2—H2	98.8	C22'—C21'—C20'	120.0
C17'—N1—C17	7.9 (7)	C22'—C21'—H21B	120.0
C17'—N1—C16	116.7 (5)	C20'—C21'—H21B	120.0
C17—N1—C16	124.4 (4)	C21'—C22'—C17'	120.0
C17'—N1—C15	125.5 (5)	C21'—C22'—H22B	120.0

C17—N1—C15	118.9 (4)	C17'—C22'—H22B	120.0
C16—N1—C15	114.2 (3)	O2—C23—C28	118.9 (2)
C39'—N2—C39	9.1 (7)	O2—C23—C24	118.1 (2)
C39'—N2—C38	115.4 (5)	C28—C23—C24	123.0 (2)
C39—N2—C38	124.4 (3)	C25—C24—C23	116.6 (2)
C39'—N2—C37	126.2 (6)	C25—C24—C33	120.8 (2)
C39—N2—C37	118.3 (4)	C23—C24—C33	122.5 (2)
C38—N2—C37	114.4 (3)	C26—C25—C24	122.7 (2)
O1—C1—C6	119.7 (2)	C26—C25—H25	118.7
O1—C1—C2	117.7 (2)	C24—C25—H25	118.7
C6—C1—C2	122.6 (2)	C27—C26—C25	118.6 (2)
C3—C2—C1	116.8 (2)	C27—C26—C37	122.8 (2)
C3—C2—C11	121.3 (2)	C25—C26—C37	118.6 (2)
C1—C2—C11	121.9 (2)	C26—C27—C28	122.5 (2)
C2—C3—C4	122.7 (2)	C26—C27—H27	118.8
C2—C3—H3	118.6	C28—C27—H27	118.8
C4—C3—H3	118.6	C27—C28—C23	116.6 (2)
C5—C4—C3	118.6 (2)	C27—C28—C29	121.0 (2)
C5—C4—C15	122.6 (2)	C23—C28—C29	122.4 (2)
C3—C4—C15	118.8 (2)	C32—C29—C28	111.4 (2)
C4—C5—C6	122.5 (2)	C32—C29—C31	105.8 (2)
C4—C5—H5	118.7	C28—C29—C31	110.4 (2)
C6—C5—H5	118.7	C32—C29—C30	107.7 (2)
C5—C6—C1	116.7 (2)	C28—C29—C30	110.6 (2)
C5—C6—C7	121.0 (2)	C31—C29—C30	110.8 (2)
C1—C6—C7	122.3 (2)	C29—C30—H30A	109.5
C9—C7—C10	105.6 (2)	C29—C30—H30B	109.5
C9—C7—C6	111.6 (2)	H30A—C30—H30B	109.5
C10—C7—C6	110.6 (2)	C29—C30—H30C	109.5
C9—C7—C8	106.6 (2)	H30A—C30—H30C	109.5
C10—C7—C8	111.1 (2)	H30B—C30—H30C	109.5
C6—C7—C8	111.1 (2)	C29—C31—H31A	109.5
C7—C8—H8A	109.5	C29—C31—H31B	109.5
C7—C8—H8B	109.5	H31A—C31—H31B	109.5
H8A—C8—H8B	109.5	C29—C31—H31C	109.5
C7—C8—H8C	109.5	H31A—C31—H31C	109.5
H8A—C8—H8C	109.5	H31B—C31—H31C	109.5
H8B—C8—H8C	109.5	C29—C32—H32A	109.5
C7—C9—H9A	109.5	C29—C32—H32B	109.5
C7—C9—H9B	109.5	H32A—C32—H32B	109.5
H9A—C9—H9B	109.5	C29—C32—H32C	109.5
C7—C9—H9C	109.5	H32A—C32—H32C	109.5
H9A—C9—H9C	109.5	H32B—C32—H32C	109.5
H9B—C9—H9C	109.5	C36—C33—C24	111.9 (2)
C7—C10—H10A	109.5	C36—C33—C35	106.8 (2)
C7—C10—H10B	109.5	C24—C33—C35	110.1 (2)
H10A—C10—H10B	109.5	C36—C33—C34	107.0 (2)
C7—C10—H10C	109.5	C24—C33—C34	110.4 (2)
H10A—C10—H10C	109.5	C35—C33—C34	110.5 (2)

H10B—C10—H10C	109.5	C33—C34—H34A	109.5
C13—C11—C12	107.1 (2)	C33—C34—H34B	109.5
C13—C11—C2	111.7 (2)	H34A—C34—H34B	109.5
C12—C11—C2	110.2 (2)	C33—C34—H34C	109.5
C13—C11—C14	106.8 (2)	H34A—C34—H34C	109.5
C12—C11—C14	110.5 (2)	H34B—C34—H34C	109.5
C2—C11—C14	110.4 (2)	C33—C35—H35A	109.5
C11—C12—H12A	109.5	C33—C35—H35B	109.5
C11—C12—H12B	109.5	H35A—C35—H35B	109.5
H12A—C12—H12B	109.5	C33—C35—H35C	109.5
C11—C12—H12C	109.5	H35A—C35—H35C	109.5
H12A—C12—H12C	109.5	H35B—C35—H35C	109.5
H12B—C12—H12C	109.5	C33—C36—H36A	109.5
C11—C13—H13A	109.5	C33—C36—H36B	109.5
C11—C13—H13B	109.5	H36A—C36—H36B	109.5
H13A—C13—H13B	109.5	C33—C36—H36C	109.5
C11—C13—H13C	109.5	H36A—C36—H36C	109.5
H13A—C13—H13C	109.5	H36B—C36—H36C	109.5
H13B—C13—H13C	109.5	N2—C37—C26	114.9 (2)
C11—C14—H14A	109.5	N2—C37—H37A	108.5
C11—C14—H14B	109.5	C26—C37—H37A	108.5
H14A—C14—H14B	109.5	N2—C37—H37B	108.5
C11—C14—H14C	109.5	C26—C37—H37B	108.5
H14A—C14—H14C	109.5	H37A—C37—H37B	107.5
H14B—C14—H14C	109.5	N2—C38—H38A	109.5
N1—C15—C4	115.1 (2)	N2—C38—H38B	109.5
N1—C15—H15A	108.5	H38A—C38—H38B	109.5
C4—C15—H15A	108.5	N2—C38—H38C	109.5
N1—C15—H15B	108.5	H38A—C38—H38C	109.5
C4—C15—H15B	108.5	H38B—C38—H38C	109.5
H15A—C15—H15B	107.5	C40—C39—C44	120.0
N1—C16—H16A	109.5	C40—C39—N2	119.5 (4)
N1—C16—H16B	109.5	C44—C39—N2	120.5 (4)
H16A—C16—H16B	109.5	C41—C40—C39	120.0
N1—C16—H16C	109.5	C41—C40—H40A	120.0
H16A—C16—H16C	109.5	C39—C40—H40A	120.0
H16B—C16—H16C	109.5	C42—C41—C40	120.0
C18—C17—C22	120.0	C42—C41—H41A	120.0
C18—C17—N1	118.7 (4)	C40—C41—H41A	120.0
C22—C17—N1	121.3 (4)	C41—C42—C43	120.0
C17—C18—C19	120.0	C41—C42—H42A	120.0
C17—C18—H18A	120.0	C43—C42—H42A	120.0
C19—C18—H18A	120.0	C44—C43—C42	120.0
C20—C19—C18	120.0	C44—C43—H43A	120.0
C20—C19—H19A	120.0	C42—C43—H43A	120.0
C18—C19—H19A	120.0	C43—C44—C39	120.0
C21—C20—C19	120.0	C43—C44—H44A	120.0
C21—C20—H20A	120.0	C39—C44—H44A	120.0
C19—C20—H20A	120.0	N2—C39'—C40'	122.2 (7)

C20—C21—C22	120.0	N2—C39'—C44'	117.8 (7)
C20—C21—H21A	120.0	C40'—C39'—C44'	120.0
C22—C21—H21A	120.0	C41'—C40'—C39'	120.0
C21—C22—C17	120.0	C41'—C40'—H40B	120.0
C21—C22—H22A	120.0	C39'—C40'—H40B	120.0
C17—C22—H22A	120.0	C42'—C41'—C40'	120.0
N1—C17'—C18'	123.0 (7)	C42'—C41'—H41B	120.0
N1—C17'—C22'	117.0 (7)	C40'—C41'—H41B	120.0
C18'—C17'—C22'	120.0	C41'—C42'—C43'	120.0
C19'—C18'—C17'	120.0	C41'—C42'—H42B	120.0
C19'—C18'—H18B	120.0	C43'—C42'—H42B	120.0
C17'—C18'—H18B	120.0	C44'—C43'—C42'	120.0
C18'—C19'—C20'	120.0	C44'—C43'—H43B	120.0
C18'—C19'—H19B	120.0	C42'—C43'—H43B	120.0
C20'—C19'—H19B	120.0	C43'—C44'—C39'	120.0
C21'—C20'—C19'	120.0	C43'—C44'—H44B	120.0
C21'—C20'—H20B	120.0	C39'—C44'—H44B	120.0
O1—C1—C2—C3	−177.5 (2)	O2—C23—C24—C25	177.4 (2)
C6—C1—C2—C3	2.3 (3)	C28—C23—C24—C25	−2.1 (3)
O1—C1—C2—C11	3.3 (3)	O2—C23—C24—C33	−3.6 (3)
C6—C1—C2—C11	−176.9 (2)	C28—C23—C24—C33	176.8 (2)
C1—C2—C3—C4	0.3 (3)	C23—C24—C25—C26	0.0 (4)
C11—C2—C3—C4	179.5 (2)	C33—C24—C25—C26	−179.0 (2)
C2—C3—C4—C5	−1.7 (4)	C24—C25—C26—C27	1.3 (4)
C2—C3—C4—C15	178.9 (2)	C24—C25—C26—C37	−178.9 (2)
C3—C4—C5—C6	0.6 (4)	C25—C26—C27—C28	−0.6 (4)
C15—C4—C5—C6	179.9 (2)	C37—C26—C27—C28	179.6 (3)
C4—C5—C6—C1	1.8 (4)	C26—C27—C28—C23	−1.3 (4)
C4—C5—C6—C7	−177.3 (2)	C26—C27—C28—C29	177.5 (2)
O1—C1—C6—C5	176.5 (2)	O2—C23—C28—C27	−176.8 (2)
C2—C1—C6—C5	−3.3 (3)	C24—C23—C28—C27	2.8 (4)
O1—C1—C6—C7	−4.4 (3)	O2—C23—C28—C29	4.4 (3)
C2—C1—C6—C7	175.8 (2)	C24—C23—C28—C29	−176.0 (2)
C5—C6—C7—C9	5.4 (3)	C27—C28—C29—C32	−5.1 (3)
C1—C6—C7—C9	−173.6 (2)	C23—C28—C29—C32	173.6 (2)
C5—C6—C7—C10	122.7 (3)	C27—C28—C29—C31	−122.4 (3)
C1—C6—C7—C10	−56.3 (3)	C23—C28—C29—C31	56.4 (3)
C5—C6—C7—C8	−113.4 (3)	C27—C28—C29—C30	114.6 (3)
C1—C6—C7—C8	67.5 (3)	C23—C28—C29—C30	−66.6 (3)
C3—C2—C11—C13	0.5 (3)	C25—C24—C33—C36	−0.9 (3)
C1—C2—C11—C13	179.6 (2)	C23—C24—C33—C36	−179.8 (2)
C3—C2—C11—C12	119.4 (3)	C25—C24—C33—C35	−119.5 (3)
C1—C2—C11—C12	−61.4 (3)	C23—C24—C33—C35	61.6 (3)
C3—C2—C11—C14	−118.2 (3)	C25—C24—C33—C34	118.2 (3)
C1—C2—C11—C14	60.9 (3)	C23—C24—C33—C34	−60.7 (3)
C17'—N1—C15—C4	117.7 (7)	C39'—N2—C37—C26	−119.0 (7)
C17—N1—C15—C4	112.5 (5)	C39—N2—C37—C26	−113.5 (5)
C16—N1—C15—C4	−84.5 (4)	C38—N2—C37—C26	84.8 (4)
C5—C4—C15—N1	−13.7 (4)	C27—C26—C37—N2	13.1 (4)

C3—C4—C15—N1	165.6 (2)	C25—C26—C37—N2	−166.7 (2)
C17'—N1—C17—C18	−166 (7)	C39'—N2—C39—C40	170 (6)
C16—N1—C17—C18	−179.3 (3)	C38—N2—C39—C40	178.9 (3)
C15—N1—C17—C18	−18.1 (6)	C37—N2—C39—C40	19.1 (6)
C17'—N1—C17—C22	13 (6)	C39'—N2—C39—C44	−10 (5)
C16—N1—C17—C22	−0.3 (7)	C38—N2—C39—C44	−0.8 (7)
C15—N1—C17—C22	160.9 (3)	C37—N2—C39—C44	−160.5 (3)
C22—C17—C18—C19	0.0	C44—C39—C40—C41	0.0
N1—C17—C18—C19	179.0 (7)	N2—C39—C40—C41	−179.6 (6)
C17—C18—C19—C20	0.0	C39—C40—C41—C42	0.0
C18—C19—C20—C21	0.0	C40—C41—C42—C43	0.0
C19—C20—C21—C22	0.0	C41—C42—C43—C44	0.0
C20—C21—C22—C17	0.0	C42—C43—C44—C39	0.0
C18—C17—C22—C21	0.0	C40—C39—C44—C43	0.0
N1—C17—C22—C21	−179.0 (7)	N2—C39—C44—C43	179.6 (6)
C17—N1—C17'—C18'	4(6)	C39—N2—C39'—C40'	0(5)
C16—N1—C17'—C18'	171.5 (5)	C38—N2—C39'—C40'	−172.0 (5)
C15—N1—C17'—C18'	−31.1 (10)	C37—N2—C39'—C40'	32.0 (10)
C17—N1—C17'—C22'	−176 (7)	C39—N2—C39'—C44'	180 (6)
C16—N1—C17'—C22'	−7.9 (9)	C38—N2—C39'—C44'	7.8 (9)
C15—N1—C17'—C22'	149.5 (4)	C37—N2—C39'—C44'	−148.2 (5)
N1—C17'—C18'—C19'	−179.4 (11)	N2—C39'—C40'—C41'	179.8 (11)
C22'—C17'—C18'—C19'	0.0	C44'—C39'—C40'—C41'	0.0
C17'—C18'—C19'—C20'	0.0	C39'—C40'—C41'—C42'	0.0
C18'—C19'—C20'—C21'	0.0	C40'—C41'—C42'—C43'	0.0
C19'—C20'—C21'—C22'	0.0	C41'—C42'—C43'—C44'	0.0
C20'—C21'—C22'—C17'	0.0	C42'—C43'—C44'—C39'	0.0
N1—C17'—C22'—C21'	179.4 (10)	N2—C39'—C44'—C43'	−179.8 (10)
C18'—C17'—C22'—C21'	0.0	C40'—C39'—C44'—C43'	0.0

Fig. 1

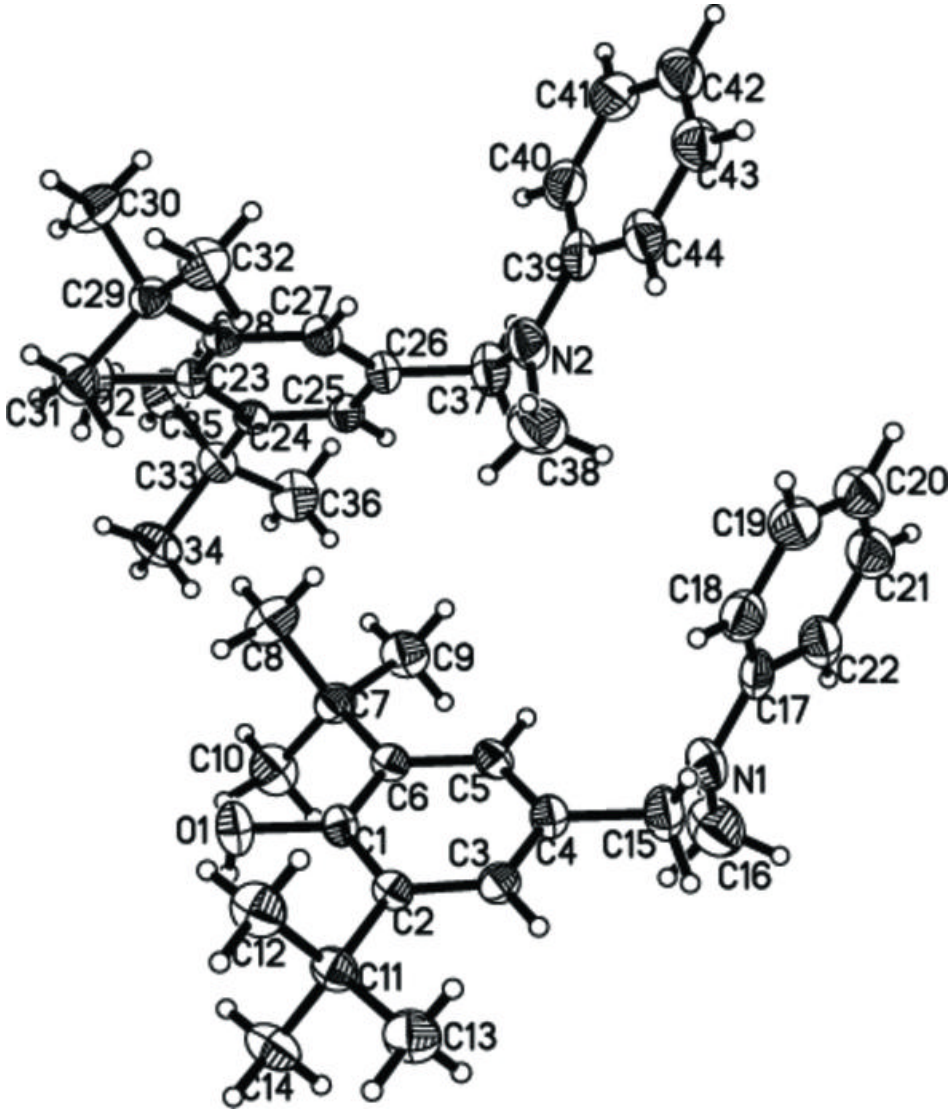
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Fig. 2