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5-[3-(Dimethylamino)propyl]-5*H*-dibenzo[*a*,*d*]cycloheptan-5-ol

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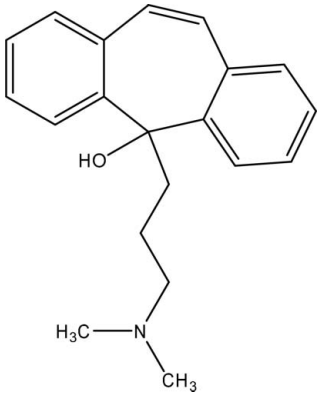
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.010 Å; R factor = 0.080; wR factor = 0.201; data-to-parameter ratio = 10.6.

The title compound, $C_{20}H_{23}NO$, was synthesized by the reaction of dibenzo[*a*,*d*]cyclohepta-1,4,6-trien-5-one and dimethylaminopropylmagnesium. Intramolecular O—H···N hydrogen bonding causes the formation of a non-planar seven-membered ring. The benzene rings are oriented at a dihedral angle of 51.37 (3)°.

Related literature

For related literature, see: Nakagawa *et al.* (1996); Wang *et al.* (1999). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{20}H_{23}NO$
M_r = 293.39
Orthorhombic, $P2_12_12_1$
a = 11.127 (2) Å
b = 11.325 (2) Å
c = 13.155 (3) Å
V = 1657.7 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 298 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.952, T_{max} = 0.963
1858 measured reflections
1858 independent reflections
1113 reflections with $I > 2\sigma(I)$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.080
$wR(F^2)$ = 0.201
S = 1.01
1858 reflections
175 parameters
53 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O—H0A···N	0.85	2.10	2.703 (8)	127

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2346).

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supplementary materials

Acta Cryst. (2007). E**63**, o4460 [**doi:10.1107/S1600536807052026**]

5-[3-(Dimethylamino)propyl]-5*H*-dibenzo[*a*,*d*]cycloheptan-5-ol

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Comment

Triene derivatives represent an interesting class of compounds possessing broad spectrum biological activities (Nakagawa *et al.*, 1996; Wang *et al.*, 1999). These compounds are known to exhibit diverse biological effects, such as insecticidal and fungicidal activities (Wang *et al.*, 1999). We report herein the crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The intramolecular O—H···N hydrogen bond (Table 1) causes to the formation of a non-planar seven-membered ring A (N/C3–C6/O/H0A). Rings B (C7–C12) and D (C15–C20) are nearly planar and they are oriented at a dihedral angle of 51.37 (3)°. The seven-membered ring C (C6/C7/C12–C15/C20) is not-planar.

In the crystal packing (Fig. 2), the molecules are stacked along the *a* axis.

Experimental

For the preparation of the title compound, (I), dibenzo[a,d]cyclohepta-1,4,6-triene-5-one (620 mg, 2 mmol) and dimethylaminopropyl magnesium (690 mg, 5 mmol) were added in a flask (25 ml) and reacted in an oil bath (363 K) for 6 h. After cooling and filtering, crude compound (I) was obtained. Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetone solution (m.p. 480 K).

Refinement

H atoms were positioned geometrically, with O—H = 0.85 Å (for OH) and C—H = 0.93, 0.97 and 0.96 Å, for aromatic, methylene and methyl H atoms and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*O*), where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

Figures

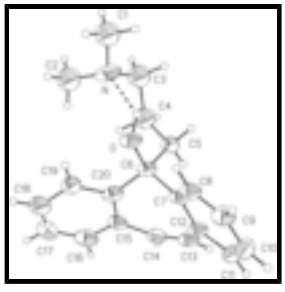

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

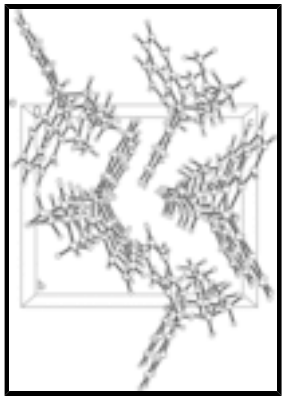

Fig. 2. A packing diagram of (I).

5-[3-(Dimethylamino)propyl]-5*H*-dibenzo[a,d]cycloheptan-5-ol

Crystal data

$C_{20}H_{23}NO$	$D_x = 1.176$ Mg m^{-3}
$M_r = 293.39$	Melting point: 480 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 25 reflections
$a = 11.127$ (2) Å	$\theta = 9$–12º
$b = 11.325$ (2) Å	$\mu = 0.07$ mm^{-1}
$c = 13.155$ (3) Å	$T = 298$ (2) K
$V = 1657.7$ (6) Å^3	Plate, colourless
$Z = 4$	0.40 × 0.10 × 0.10 mm
$F_{000} = 632$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.0000$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0$º
Monochromator: graphite	$\theta_{min} = 2.4$º
$T = 298(2)$ K	$h = 0\rightarrow13$
$\omega/2\theta$ scans	$k = 0\rightarrow13$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0\rightarrow16$
$T_{min} = 0.952$, $T_{max} = 0.963$	3 standard reflections
1858 measured reflections	every 120 min
1858 independent reflections	intensity decay: none
1113 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained

$R[F^2 > 2\sigma(F^2)] = 0.080$

$wR(F^2) = 0.201$

$S = 1.01$

1858 reflections

175 parameters

53 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 2P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.28$ e Å^{-3}

$\Delta\rho_{min} = -0.25$ e Å^{-3}

Extinction correction: none

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 $F^2 > 2$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}
O	0.1483 (3)	0.9481 (4)	0.1297 (3)	0.0623 (12)
H0A	0.0960	0.9318	0.1746	0.075*
N	−0.0027 (5)	1.0514 (7)	0.2639 (5)	0.092 (2)
C1	−0.1191 (7)	0.9969 (8)	0.2686 (7)	0.106
H1A	−0.1597	1.0074	0.2048	0.159*
H1B	−0.1654	1.0328	0.3219	0.159*
H1C	−0.1101	0.9141	0.2823	0.159*
C2	−0.0201 (8)	1.1710 (7)	0.2385 (7)	0.108
H2A	0.0563	1.2076	0.2262	0.162*
H2B	−0.0599	1.2106	0.2935	0.162*
H2C	−0.0687	1.1762	0.1783	0.162*
C3	0.0684 (8)	1.0329 (7)	0.3559 (6)	0.102
H3A	0.0616	0.9511	0.3770	0.122*
H3B	0.0371	1.0821	0.4101	0.122*
C4	0.2023 (7)	1.0633 (7)	0.3378 (7)	0.094
H4A	0.2072	1.1355	0.2984	0.113*
H4B	0.2405	1.0778	0.4029	0.113*
C5	0.2718 (5)	0.9653 (5)	0.2820 (4)	0.0548 (15)
H5A	0.2398	0.8896	0.3032	0.066*
H5B	0.3552	0.9684	0.3034	0.066*
C6	0.2681 (5)	0.9710 (6)	0.1624 (4)	0.0464 (14)
C7	0.3438 (5)	0.8681 (6)	0.1212 (4)	0.0520 (14)

C8	0.2919 (6)	0.7768 (6)	0.0715 (4)	0.0604 (16)
H8A	0.2094	0.7793	0.0604	0.072*
C9	0.3542 (7)	0.6811 (7)	0.0370 (5)	0.0734 (19)
H9A	0.3132	0.6208	0.0038	0.088*
C10	0.4754 (7)	0.6721 (6)	0.0501 (6)	0.080 (2)
H10A	0.5179	0.6074	0.0254	0.096*
C11	0.5326 (6)	0.7614 (6)	0.1010 (5)	0.0702 (18)
H11A	0.6145	0.7544	0.1139	0.084*
C12	0.4697 (5)	0.8675 (6)	0.1358 (4)	0.0589 (15)
C13	0.5424 (6)	0.9602 (8)	0.1862 (5)	0.0655 (19)
H13A	0.6102	0.9355	0.2216	0.079*
C14	0.5207 (6)	1.0687 (8)	0.1853 (5)	0.073 (2)
H14A	0.5766	1.1134	0.2212	0.088*
C15	0.4259 (6)	1.1385 (6)	0.1395 (4)	0.0618 (16)
C16	0.4494 (7)	1.2484 (7)	0.1066 (5)	0.083 (2)
H16A	0.5244	1.2805	0.1211	0.099*
C17	0.3680 (8)	1.3173 (7)	0.0518 (6)	0.084 (2)
H17A	0.3858	1.3946	0.0333	0.101*
C18	0.2607 (7)	1.2657 (8)	0.0267 (5)	0.090 (3)
H18A	0.2081	1.3061	−0.0158	0.108*
C19	0.2278 (6)	1.1554 (6)	0.0623 (4)	0.0655 (18)
H19A	0.1505	1.1277	0.0498	0.079*
C20	0.3088 (5)	1.0841 (6)	0.1173 (4)	0.0597 (17)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O	0.0347 (19)	0.103 (3)	0.049 (2)	−0.008 (2)	−0.0014 (18)	−0.008 (3)
N	0.052 (3)	0.151 (6)	0.073 (4)	0.005 (4)	0.018 (3)	−0.024 (5)
C1	0.106	0.106	0.106	0.000	0.000	0.000
C2	0.108	0.108	0.108	0.000	0.000	0.000
C3	0.102	0.102	0.102	0.000	0.000	0.000
C4	0.094	0.094	0.094	0.000	0.000	0.000
C5	0.053 (3)	0.073 (4)	0.038 (3)	0.003 (3)	−0.006 (3)	−0.003 (3)
C6	0.033 (3)	0.082 (4)	0.023 (2)	−0.006 (3)	−0.003 (2)	−0.009 (3)
C7	0.044 (3)	0.089 (4)	0.023 (2)	−0.001 (3)	0.006 (2)	0.003 (3)
C8	0.053 (3)	0.094 (4)	0.034 (3)	−0.001 (3)	0.002 (3)	0.007 (3)
C9	0.074 (4)	0.086 (4)	0.060 (4)	−0.004 (4)	0.008 (4)	−0.002 (4)
C10	0.078 (4)	0.087 (4)	0.073 (4)	0.027 (4)	0.006 (4)	0.008 (4)
C11	0.056 (4)	0.094 (4)	0.061 (4)	0.017 (3)	0.007 (3)	0.010 (3)
C12	0.050 (3)	0.095 (4)	0.032 (3)	0.007 (3)	0.006 (3)	0.002 (3)
C13	0.042 (3)	0.117 (6)	0.037 (3)	0.003 (4)	0.002 (3)	0.006 (4)
C14	0.049 (4)	0.122 (6)	0.049 (4)	−0.006 (4)	0.008 (3)	0.018 (4)
C15	0.055 (4)	0.103 (5)	0.028 (3)	−0.003 (4)	0.003 (3)	0.005 (3)
C16	0.080 (5)	0.109 (6)	0.058 (5)	−0.016 (5)	0.010 (4)	−0.012 (5)
C17	0.094 (6)	0.089 (5)	0.069 (5)	−0.010 (5)	0.011 (5)	0.012 (4)
C18	0.079 (5)	0.145 (8)	0.045 (4)	0.009 (6)	0.000 (4)	0.012 (5)
C19	0.060 (4)	0.102 (5)	0.035 (3)	0.011 (4)	−0.004 (3)	−0.005 (4)

C20	0.046 (3)	0.104 (5)	0.030 (3)	0.000 (3)	−0.001 (3)	−0.017 (3)

Geometric parameters (Å, °)

O—C6	1.424 (6)	C8—C9	1.364 (9)
O—H0A	0.8500	C8—H8A	0.9300
N—C2	1.409 (10)	C9—C10	1.363 (9)
N—C1	1.436 (9)	C9—H9A	0.9300
N—C3	1.460 (10)	C10—C11	1.369 (7)
C1—H1A	0.9600	C10—H10A	0.9300
C1—H1B	0.9600	C11—C12	1.465 (9)
C1—H1C	0.9600	C11—H11A	0.9300
C2—H2A	0.9600	C12—C13	1.481 (9)
C2—H2B	0.9600	C13—C14	1.253 (10)
C2—H2C	0.9600	C13—H13A	0.9300
C3—C4	1.547 (11)	C14—C15	1.450 (9)
C3—H3A	0.9700	C14—H14A	0.9300
C3—H3B	0.9700	C15—C16	1.344 (9)
C4—C5	1.540 (9)	C15—C20	1.470 (8)
C4—H4A	0.9700	C16—C17	1.396 (10)
C4—H4B	0.9700	C16—H16A	0.9300
C5—C6	1.575 (7)	C17—C18	1.370 (10)
C5—H5A	0.9700	C17—H17A	0.9300
C5—H5B	0.9700	C18—C19	1.383 (8)
C6—C20	1.483 (9)	C18—H18A	0.9300
C6—C7	1.537 (8)	C19—C20	1.410 (8)
C7—C8	1.353 (8)	C19—H19A	0.9300
C7—C12	1.414 (8)		
C6—O—H0A	118.1	C8—C7—C6	121.1 (5)
C2—N—C1	107.4 (7)	C12—C7—C6	119.9 (6)
C2—N—C3	114.2 (7)	C7—C8—C9	123.5 (7)
C1—N—C3	113.1 (7)	C7—C8—H8A	118.3
N—C1—H1A	109.5	C9—C8—H8A	118.3
N—C1—H1B	109.5	C8—C9—C10	121.3 (8)
H1A—C1—H1B	109.5	C8—C9—H9A	119.3
N—C1—H1C	109.5	C10—C9—H9A	119.3
H1A—C1—H1C	109.5	C9—C10—C11	117.8 (7)
H1B—C1—H1C	109.5	C9—C10—H10A	121.1
N—C2—H2A	109.5	C11—C10—H10A	121.1
N—C2—H2B	109.5	C10—C11—C12	122.5 (6)
H2A—C2—H2B	109.5	C10—C11—H11A	118.8
N—C2—H2C	109.5	C12—C11—H11A	118.8
H2A—C2—H2C	109.5	C7—C12—C11	115.8 (6)
H2B—C2—H2C	109.5	C7—C12—C13	126.8 (6)
N—C3—C4	111.3 (7)	C11—C12—C13	117.4 (6)
N—C3—H3A	109.4	C14—C13—C12	125.9 (7)
C4—C3—H3A	109.4	C14—C13—H13A	117.1
N—C3—H3B	109.4	C12—C13—H13A	117.1
C4—C3—H3B	109.4	C13—C14—C15	132.7 (8)

H3A—C3—H3B	108.0	C13—C14—H14A	113.6
C5—C4—C3	113.4 (7)	C15—C14—H14A	113.6
C5—C4—H4A	108.9	C16—C15—C14	119.8 (7)
C3—C4—H4A	108.9	C16—C15—C20	119.8 (7)
C5—C4—H4B	108.9	C14—C15—C20	120.0 (6)
C3—C4—H4B	108.9	C15—C16—C17	123.9 (8)
H4A—C4—H4B	107.7	C15—C16—H16A	118.1
C4—C5—C6	115.7 (6)	C17—C16—H16A	118.1
C4—C5—H5A	108.4	C18—C17—C16	116.9 (8)
C6—C5—H5A	108.4	C18—C17—H17A	121.6
C4—C5—H5B	108.4	C16—C17—H17A	121.6
C6—C5—H5B	108.4	C17—C18—C19	122.3 (8)
H5A—C5—H5B	107.4	C17—C18—H18A	118.9
O—C6—C20	108.8 (5)	C19—C18—H18A	118.9
O—C6—C7	105.6 (5)	C18—C19—C20	121.4 (7)
C20—C6—C7	110.3 (4)	C18—C19—H19A	119.3
O—C6—C5	108.6 (4)	C20—C19—H19A	119.3
C20—C6—C5	115.3 (5)	C19—C20—C15	115.4 (6)
C7—C6—C5	107.8 (5)	C19—C20—C6	120.3 (5)
C8—C7—C12	119.0 (6)	C15—C20—C6	123.6 (5)
C2—N—C3—C4	−71.8 (9)	C10—C11—C12—C13	−177.5 (6)
C1—N—C3—C4	165.1 (7)	C7—C12—C13—C14	−32.6 (11)
N—C3—C4—C5	−79.0 (8)	C11—C12—C13—C14	150.3 (7)
C3—C4—C5—C6	87.5 (8)	C12—C13—C14—C15	0.0 (13)
C4—C5—C6—O	−67.0 (7)	C13—C14—C15—C16	−147.4 (8)
C4—C5—C6—C20	55.4 (7)	C13—C14—C15—C20	24.8 (11)
C4—C5—C6—C7	179.0 (5)	C14—C15—C16—C17	172.8 (6)
O—C6—C7—C8	−4.1 (7)	C20—C15—C16—C17	0.5 (9)
C20—C6—C7—C8	−121.5 (6)	C15—C16—C17—C18	−3.4 (10)
C5—C6—C7—C8	111.9 (6)	C16—C17—C18—C19	6.7 (11)
O—C6—C7—C12	176.5 (5)	C17—C18—C19—C20	−7.4 (11)
C20—C6—C7—C12	59.1 (6)	C18—C19—C20—C15	4.1 (9)
C5—C6—C7—C12	−67.5 (7)	C18—C19—C20—C6	174.6 (6)
C12—C7—C8—C9	2.1 (10)	C16—C15—C20—C19	−0.8 (8)
C6—C7—C8—C9	−177.3 (6)	C14—C15—C20—C19	−173.0 (5)
C7—C8—C9—C10	−0.5 (11)	C16—C15—C20—C6	−171.0 (5)
C8—C9—C10—C11	1.2 (12)	C14—C15—C20—C6	16.8 (8)
C9—C10—C11—C12	−3.6 (11)	O—C6—C20—C19	6.6 (7)
C8—C7—C12—C11	−4.1 (9)	C7—C6—C20—C19	122.0 (6)
C6—C7—C12—C11	175.3 (5)	C5—C6—C20—C19	−115.6 (6)
C8—C7—C12—C13	178.7 (6)	O—C6—C20—C15	176.4 (5)
C6—C7—C12—C13	−1.9 (10)	C7—C6—C20—C15	−68.2 (6)
C10—C11—C12—C7	5.0 (9)	C5—C6—C20—C15	54.2 (7)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O—H0A···N	0.85	2.10	2.703 (8)	127

Fig. 1

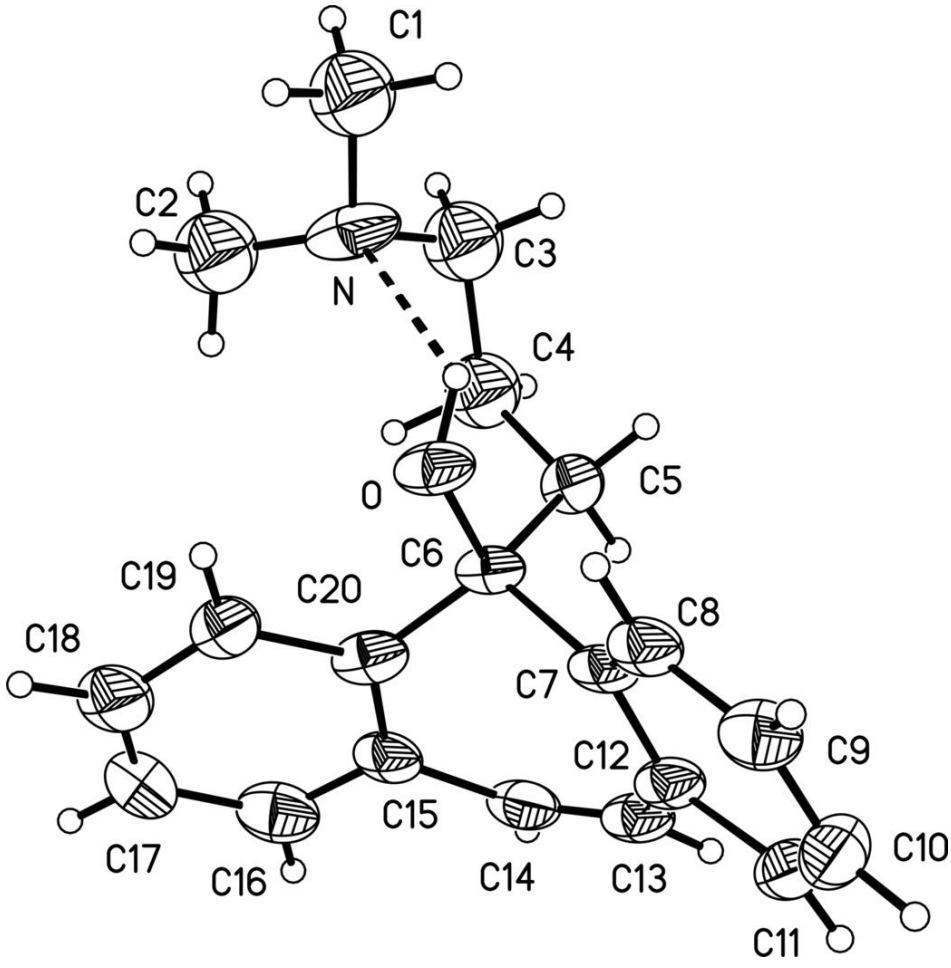
C1
C2
N
C3
C4
O
C5
C6
C19
C20
C8
C18
C7
C12
C9
C15
C10
C17
C16
C14
C13
C11

Fig. 2