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7-Phenyl-5*H*-dibenz[*c*,*e*]azepine *N*-oxide

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Key indicators

Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.108
Data-to-parameter ratio = 10.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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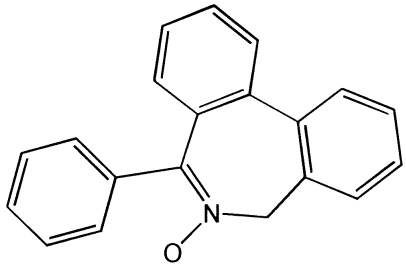
The structure of the title compound, $C_{20}H_{15}NO$, has been determined as part of a study of the preparation of 5-substituted dibenz[*c*,*e*]azepines. The geometry of this important precursor shows a conformation favouring attack by incoming reagents.

Comment

In the course of studies on the synthesis of nitrogen heterocycles with potential physiological activity, the title compound, (I), has proved to be a powerful precursor for 5-substituted derivatives of dibenz[*c*,*e*]azepines [(II), R = H; Cullen & Sharp, 1993]. Thus, it can be converted to the 5-acetoxy derivative [(II), R = $OCOCH_3$] in high yield by a Polonovski-type reaction (Bell & Childress, 1962) and thence to the 5-hydroxy derivative [(III), R = OH] by base-induced hydrolysis. The latter has the potential for conversion into a range of 5-substituted compounds *via* the Mitsunobu substitution reaction of the hydroxy group (Hughes, 1992).



(I)

The crystal structure of (I) was undertaken to obtain accurate geometry for the reactive centre of the *N*-oxide. The molecular structure is shown with the numbering system used in Fig. 1. The seven-membered ring is essentially in a boat conformation, with the plane defined by N1, C2, C5 and C6 as the base (r.m.s.d. = 0.009 Å), and a pseudo-mirror through C7 and the midpoint of the C3—C4 bond. The 'stern' is defined by C2, C3, C4 and C5, which are essentially coplanar with C31, C32, C33 and C34 (r.m.s.d. for 8 atoms = 0.025 Å). The stern and the bowsprit, defined by N1, C7 and C6, make angles of 35.94 (6) and 57.96 (12)°, respectively, with the base. The puckering parameters (Cremer & Pople, 1975) for the seven-membered ring are: Q = 0.899, $q2$ = 0.875, $q3$ = 0.203 Å, $\varphi2$ = 105.4, $\varphi3$ = 151.0, θ = 13.1° (Gould *et al.*, 1995). The third ring of the condensed system (C6, C5, C51, C52, C53 and C54; r.m.s.d. = 0.003 Å) bends away from the basal plane in the opposite direction, making an angle of 33.58 (7)°. The effect of this conformation is to place O1 and the H atoms on C7 in an

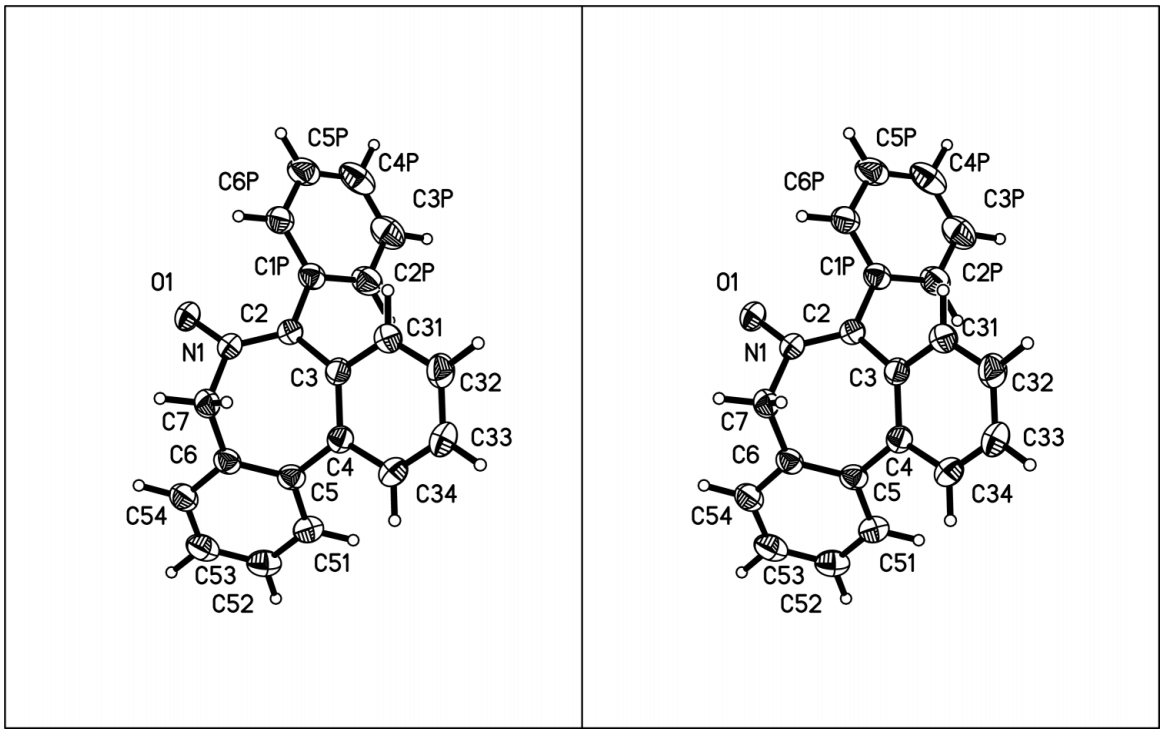


Figure 1
Stereoview of (II), showing the atom-numbering scheme and 50% probability displacement ellipsoids.

uncrowded position to interact with incoming atoms. The two fused six-membered rings make up a constrained diphenyl moiety with a twist of 40.01 (5)°.

Experimental

Treatment of the parent azepine (III) with *m*-chloroperbenzoic acid in dry dichloromethane followed by extraction with sodium bicarbonate solution gave crude (I), which was recrystallized from ethyl acetate in more than 90% yield, giving colourless prisms suitable for X-ray diffraction. The crystals melt at 449–450 K and analyse to give C 84.00, H 5.19 and N 5.09%; $C_{20}H_{15}NO$ requires C 84.2, H 5.30, N 4.90%. Parent ion in FAB mass spectrum: m/*z* = 285.1152; $C_{20}H_{15}NO$ requires m/*z* = 285.1154. Mass spectra were obtained using a Kratos MS50TS spectrometer in the +FAB mode with a glycerol matrix.

Crystal data

$C_{20}H_{15}NO$
M_r = 285.33
Monoclinic, $P2_1/n$
a = 11.2060 (17) Å
b = 9.3052 (10) Å
c = 14.4925 (18) Å
β = 101.979 (8)°
V = 1478.3 (3) Å^3
Z = 4
D_x = 1.282 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 28 reflections
θ = 39–42°
μ = 0.62 mm^{-1}
T = 220 (1) K
Prism, colourless
0.70 × 0.42 × 0.23 mm

Data collection

Stoe Stadi-4 diffractometer
θ–ω scans
Absorption correction: analytical
T_{min} = 0.659, T_{max} = 0.865
3787 measured reflections
2666 independent reflections
2349 reflections with $I > 2\sigma(I)$
R_{int} = 0.009
θ_{max} = 69.9°
$h = -13 \rightarrow 12$
$k = -5 \rightarrow 11$
$l = -16 \rightarrow 17$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
S = 1.05
2666 reflections
245 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.4324P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.017
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0040 (5)

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.3130 (17)	O1—N1	1.2960 (14)
C2—C3	1.4828 (19)	C2—C1*P*	1.4796 (19)
C3—C4	1.4132 (19)	C3—C31	1.400 (2)
C4—C5	1.4781 (19)	C4—C34	1.4038 (19)
C5—C6	1.3992 (19)	C5—C51	1.403 (2)
C6—C7	1.495 (2)	C6—C54	1.395 (2)
N1—C7	1.4911 (17)		
C7—N1—C2	119.69 (11)	C1*P*—C2—C3	118.71 (11)
N1—C2—C3	120.65 (12)	C31—C3—C2	116.51 (13)
C2—C3—C4	124.89 (12)	C31—C3—C4	118.47 (13)
C3—C4—C5	123.28 (12)	C34—C4—C3	118.15 (13)
C4—C5—C6	120.56 (13)	C34—C4—C5	118.57 (13)
C5—C6—C7	119.05 (13)	C51—C5—C4	120.82 (13)
C6—C7—N1	109.20 (11)	C51—C5—C6	118.59 (13)
O1—N1—C7	115.70 (11)	C54—C6—C5	120.07 (14)
O1—N1—C2	124.57 (11)	C54—C6—C7	120.88 (13)
C1*P*—C2—N1	120.62 (12)	C32—C31—C3	122.14 (15)
C7—N1—C2—C3	0.08 (19)	O1—N1—C2—C1*P*	4.2 (2)
N1—C2—C3—C4	46.2 (2)	C7—N1—C2—C1*P*	−178.31 (12)
C2—C3—C4—C5	−5.3 (2)	O1—N1—C2—C3	−177.45 (12)
C3—C4—C5—C6	−39.58 (19)	N1—C2—C3—C31	−137.92 (14)
C4—C5—C6—C7	2.18 (19)	C1*P*—C2—C3—C31	40.50 (18)
C5—C6—C7—N1	69.55 (16)	C1*P*—C2—C3—C4	−135.36 (14)
C6—C7—N1—C2	−72.58 (16)	C31—C3—C4—C34	−0.68 (19)

C2—C3—C4—C34	175.11 (13)	C51—C5—C6—C54	−0.5 (2)
C31—C3—C4—C5	178.96 (13)	C4—C5—C6—C54	−178.40 (12)
C34—C4—C5—C6	140.06 (14)	C51—C5—C6—C7	−179.86 (12)
C34—C4—C5—C51	−37.85 (19)	O1—N1—C7—C6	105.17 (13)
C3—C4—C5—C51	142.51 (14)	C54—C6—C7—N1	−109.86 (14)

Data collection: *DIF*4 (Stoe & Cie, 1990); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1990); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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