Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å²)

$$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.703 (2)	0.7750 (9)	0.589 (2)	0.065 (8)
C2	0.596 (2)	0.8210 (4)	0.600 (2)	0.064 (4)
C3	0.492 (2)	0.7751 (8)	0.623 (2)	0.065 (8)
C4	0.496 (2)	0.674 (1)	0.624 (2)	0.069 (7)
C5	0.602 (3)	0.6281 (5)	0.606 (2)	0.078 (5)
C6	0.700 (2)	0.6788 (9)	0.590 (2)	0.069 (8)
C7	0.812 (2)	0.832 (1)	0.565 (3)	0.10 (1)
C8	0.832 (2)	0.800 (1)	0.450 (2)	0.12 (1)
C9	0.946 (2)	0.820 (1)	0.688 (2)	0.12 (1)
C10	0.375 (2)	0.827 (1)	0.634 (2)	0.071 (8)
C11	0.359 (2)	0.792 (1)	0.757 (2)	0.11 (1)
C12	0.245 (2)	0.817 (1)	0.517 (2)	0.14 (2)
C13	0.586 (4)	0.5195 (6)	0.606 (3)	0.12 (1)
C14	0.512 (2)	0.4799 (9)	0.481 (2)	0.18 (2)
C15	0.586 (2)	0.475 (1)	0.705 (2)	0.17 (2)
N	0.607 (2)	0.9265 (5)	0.607 (3)	0.086 (5)
O1	0.522 (2)	0.9657 (7)	0.501 (2)	0.15 (1)
O2	0.659 (2)	0.966 (1)	0.702 (2)	0.14 (1)

Table 2. *Geometric parameters* (Å, °)

C1—C2	1.38 (3)	C5—C13	1.54 (1)
C1—C6	1.35 (2)	C7—C8	1.47 (4)
C1—C7	1.55 (3)	C7—C9	1.54 (3)
C2—C3	1.41 (3)	C10—C11	1.55 (3)
C2—N	1.488 (9)	C10—C12	1.49 (3)
C3—C4	1.42 (2)	C13—C14	1.42 (3)
C3—C10	1.51 (3)	C13—C15	1.30 (4)
C4—C5	1.41 (4)	N—O1	1.29 (3)
C5—C6	1.35 (3)	N—O2	1.14 (3)
C2—C1—C6	116 (2)	C1—C7—C8	114 (2)
C2—C1—C7	121 (1)	C1—C7—C9	106 (2)
C6—C1—C7	123 (2)	C8—C7—C9	110 (2)
C1—C2—C3	125 (1)	C3—C10—C11	110 (1)
C1—C2—N	115 (2)	C3—C10—C12	113 (2)
C3—C2—N	120 (2)	C3—C10—H10	108 (7)
C2—C3—C4	116 (2)	C11—C10—C12	110 (2)
C2—C3—C10	124 (1)	C5—C13—C14	114 (2)
C4—C3—C10	120 (2)	C5—C13—C15	122 (3)
C3—C4—C5	119 (2)	C14—C13—C15	118 (2)
C4—C5—C6	121 (1)	C2—N—O1	111 (2)
C4—C5—C13	111 (2)	C2—N—O2	122 (2)
C6—C5—C13	128 (3)	O1—N—O2	124 (1)
C1—C6—C5	123 (2)		

The compound was synthesized according to literature procedures (Newton, 1943). Absent reflections *hkl*, $h + k + l = 2n + 1$ and *h*0*l*, $h = 2n + 1$, indicated the non-standard space group *Ia* [No. 9, standard setting *Cc*, *International Tables for X-ray Crystallography* (1965, Vol. I)]. The structure was solved by direct methods (*SIMPEL*; Schenk & Hall, 1990) and refined by full-matrix least-squares calculations with anisotropic temperature factors for the non-H atoms and isotropic temperature factors for the H atoms kept fixed at 0.08 Å². The H atoms were positioned geometrically and included as riding atoms in the structure-factor calculations. Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CELCON* program comparable to *Xtal LATCON* (Hall & Stewart, 1990). Data reduction: *Xtal ADDREF*. Program(s) used to solve structure: *Xtal SIMPEL*. Program(s) used to refine structure: *Xtal CRYLSQ*. Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *Xtal BONDLA*, *CIFIO*.

The authors thank Ing. Jan Fraanje for collecting the X-ray data.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71333 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1053]

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Structure of 6-*tert*-Butyl-3,3-dimethyl-3*H*-indolium-1-oxide

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Abstract

Crystallographic characterization of 6-*tert*-butyl-3,3-dimethyl-3*H*-indolium-1-oxide has shown that the bicyclic ring moiety is almost planar. The N—O bond of 1.29 (1) Å has mainly single-bond character.

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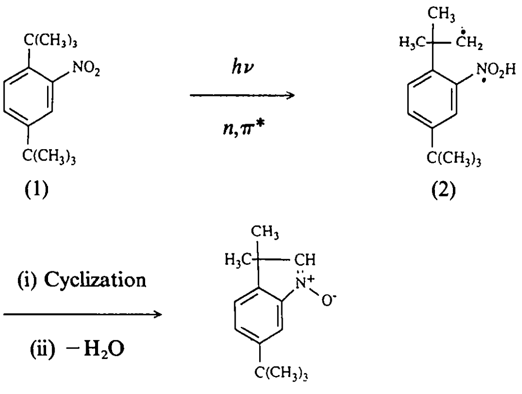
Comment

1,4-Di-*tert*-butyl-2-nitrobenzene (1) has been used frequently in studies of photochemical properties of *o-tert*-butylnitrobenzene compounds (Döpp, 1971*a*,*b*; Döpp & Brugger, 1973, 1979*a*). Exposure to light of powdered crystals of (1) gives a biradical intermediate (2), which consecutively yields the title compound (3) by an intramolecular ring closure (Döpp & Brugger, 1979*b*). A crystal study of 6-*tert*-butyl-3,3-dimethyl-3*H*-indolium-1-oxide was undertaken to examine the planarity of the —C=N$^+$—O$^-$ moiety.



The crystals were of poor quality which is reflected in the low accuracy of the bond distances and angles. The indole ring is essentially planar, the maximum deviation of an atom from the least-squares plane through its nine atoms being 0.06 (2) Å; the distance of the O atom from this plane is 0.09 (1) Å. The distances and angles around the N atom are of particular interest. The sum of the bond angles at nitrogen is 361 (2)°. The N1—C2 bond of 1.28 (2) Å is mainly double in character. The N1—O distance corresponds to the essentially σ bond found in (*E*)-4,4′-azopyridine-1,1′-dioxide (1.283 Å; Eichhorn, 1956) or in 2-(2-pyridyl)isatogen (1.280 Å; Adams, Hooper, Swain, Raper & Stoddart, 1986). These results corroborate the structure given in the reaction scheme.

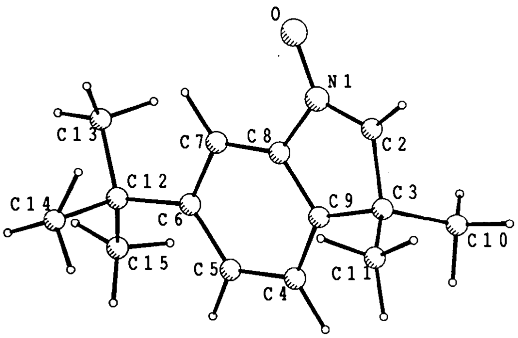


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) drawing showing the numbering system of the title compound. The H atoms are shown but not labelled.

Experimental

Crystal data

$C_{14}H_{19}NO$
M_r = 217.31
Orthorhombic
Pcab
a = 10.974 (2) Å
b = 11.892 (1) Å
c = 19.952 (3) Å
V = 2603.6 (6) Å^3
Z = 8
D_x = 1.109 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 23 reflections
θ = 25–34.2°
μ = 5.07 cm^{-1}
T = 293 K
Plate
0.30 × 0.25 × 0.05 mm
Colourless

Data collection

Enraf-Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{\min}$ = 0.48, $T_{\max}$ = 1.97
3126 measured reflections
3040 independent reflections

688 observed reflections [$I > 2.5\sigma(I)$]
$\theta_{\max}$ = 74.49°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 25$
2 standard reflections
frequency: 60 min
intensity variation: <2%

Refinement

Refinement on F
Final R = 0.082
wR = 0.135
S = 0.525
685 reflections
222 parameters
H atoms refined with distances and angles restrained
$w = 1/(2.66 + F_o + 0.032F_o^2)$
$(\Delta/\sigma)_{\max}$ = 0.163

$\Delta\rho_{\max}$ = 0.244 e Å^{-3}
$\Delta\rho_{\min}$ = −0.242 e Å^{-3}
Extinction correction: Zachariasen (1967)
Extinction coefficient: 9 (10) × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O	0.3992 (8)	1.0046 (8)	0.4232 (5)	0.074 (6)
N1	0.3557 (8)	0.9105 (9)	0.4453 (6)	0.049 (6)
C2	0.385 (1)	0.856 (1)	0.4983 (7)	0.071 (9)
C3	0.314 (1)	0.749 (1)	0.5082 (6)	0.052 (7)
C4	0.146 (1)	0.680 (1)	0.4199 (6)	0.046 (7)
C5	0.0832 (9)	0.714 (1)	0.3620 (6)	0.044 (7)
C6	0.106 (1)	0.816 (1)	0.3293 (6)	0.048 (7)
C7	0.199 (1)	0.889 (1)	0.3552 (7)	0.049 (8)
C8	0.2581 (9)	0.8536 (9)	0.4115 (6)	0.049 (7)
C9	0.235 (1)	0.756 (1)	0.4465 (5)	0.052 (7)
C10	0.396 (1)	0.642 (1)	0.5098 (8)	0.08 (1)
C11	0.236 (1)	0.754 (2)	0.5714 (6)	0.073 (9)
C12	0.031 (1)	0.852 (1)	0.2678 (7)	0.068 (9)
C13	0.103 (2)	0.930 (2)	0.2212 (9)	0.10 (1)
C14	−0.082 (2)	0.921 (3)	0.293 (1)	0.18 (3)
C15	−0.010 (3)	0.749 (2)	0.227 (1)	0.15 (2)

Table 2. *Geometric parameters* (Å, °)

O—N1	1.29 (1)	C5—C6	1.40 (2)
N1—C2	1.28 (2)	C6—C7	1.43 (2)
N1—C8	1.44 (1)	C6—C12	1.54 (2)

C2—C3	1.50 (2)	C7—C8	1.36 (2)
C3—C9	1.50 (2)	C8—C9	1.37 (2)
C3—C11	1.53 (2)	C12—C13	1.53 (2)
C3—C10	1.56 (2)	C12—C15	1.55 (3)
C4—C5	1.41 (2)	C12—C14	1.56 (3)
C4—C9	1.43 (2)		
C2—N1—O	129 (1)	C6—C5—C4	123 (1)
C2—N1—C8	110 (1)	C5—C6—C7	119 (1)
O—N1—C8	122 (1)	C5—C6—C12	121 (1)
N1—C2—C3	114 (1)	C7—C6—C12	120 (1)
C9—C3—C2	98 (1)	C8—C7—C6	117 (1)
C9—C3—C11	111 (1)	C7—C8—C9	126 (1)
C9—C3—C10	113 (1)	C7—C8—N1	127 (1)
C2—C3—C11	111 (1)	C9—C8—N1	107 (1)
C2—C3—C10	113 (1)	C8—C9—C4	118 (1)
C11—C3—C10	110 (1)	C8—C9—C3	111 (1)
C5—C4—C9	117 (1)	C4—C9—C3	131 (1)

The structure was solved by direct methods (*SIMPEL*; Schenk & Hall, 1990) and refined by full-matrix least-squares calculations; anisotropic for non-H atoms and isotropic for H atoms. The H atoms were positioned geometrically and included as riding atoms in the structure-factor calculations. After isotropic refinement an empirical absorption correction (*DIFABS*; Walker & Stuart, 1983) was applied. Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1989). Cell refinement: *CELCON* program comparable to *Xtal LATCON* (Hall & Stewart, 1990). Data reduction: *Xtal ADDREF, SORTRF.* Program(s) used to solve structure: *Xtal SIMPEL.* Program(s) used to refine structure: *Xtal CRYLSQ.* Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *Xtal BONDLA, CIFIO.*

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71334 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1054]

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Acta Cryst. (1993). C**49**, 1973-1975

Structure of *rac*-(3a*S*,9b*R*)-3a,4,5,9b-Tetrahydronaphtho[1,2-*b*]furan-2(3*H*)-one

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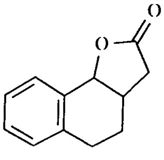
Abstract

The molecule of the title compound presents a *cis B/C* ring junction. The *B* ring adopts an almost perfect sofa conformation, the *C* ring an almost perfect envelope.

Comment

The preparation of the title compound (I) will be reported elsewhere (Ghosh, 1993). Crystals suitable for X-ray diffraction were found in the original sample. The crystal-structure determination has been undertaken to establish the relative stereochemical configuration of the title compound.



(I)

There are no unusual geometric features present in the molecule; the C—O bond distances in the five-membered ring fall within the range given by Allen *et al.* (1987). The γ-lactone ring fusion is *cis* and the H3a—C3a—C9b—H9b torsion angle is 36 (2)°. The phenyl ring is essentially planar; the maximum deviation of an atom from the six-atom plane is 0.011 (3) Å. The cyclohexene ring adopts a sofa conformation with atoms C5, C5a, C9a, C9b and C3a virtually coplanar and atom C4 displaced 0.678 (4) Å out of the plane; the ring displacement asymmetry parameter (Duax, Weeks & Rohrer, 1976) ΔC_s(C4) is 1.2 °. The γ-lactone ring is in an envelope conformation, in which C3a

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