

Fig. 1. ORTEP plot (Johnson, 1976) of the molecule showing the atomic numbering scheme. Thermal ellipsoids are plotted at the 50% probability level. H atoms are plotted with arbitrary radii.

Vol. IV), and all computer programs from the CAD4-SDP system (Frenz, 1982).^{*} Positional parameters and thermal parameters are given in Table 1, distances and angles in Table 2. Fig. 1 shows the atom numbering and Fig. 2 the unit-cell contents.

Related literature. The structure of the title compound is very similar to that of 2,5-dibromo-

^{*} Lists of structure factors, anisotropic thermal parameters, bond distances and angles involving H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53474 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

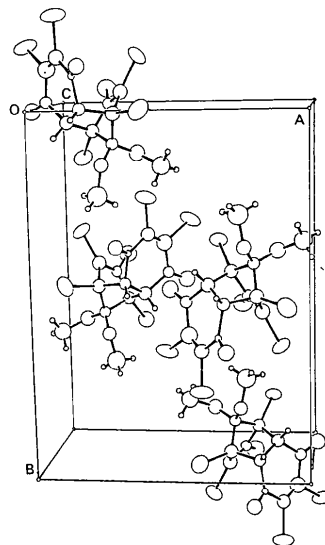


Fig. 2. Contents of the unit cell.

tricyclo[6.2.1.0^{2,7}]undeca-4,9-diene-3,6-dione (Syed, Umrigar, Griffin & Stevens, 1984). Both compounds will undergo photo-induced [2 + 2] intramolecular cyclization to the corresponding trishomocubanes.

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Structure of 7-Methoxy-5H-dibenz[c,e]azepine

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Abstract. C₁₅H₁₃NO, *M_r* = 223.27, monoclinic, *P*2₁/*c*, *a* = 8.172 (6), *b* = 11.105 (8), *c* = 12.943 (7) Å, β = 99.83 (5)°, *V* = 1157 (1) Å³, *Z* = 4, *D_x* = 1.281 Mg m⁻³, *F*(000) = 472, λ(Mo Kα) =

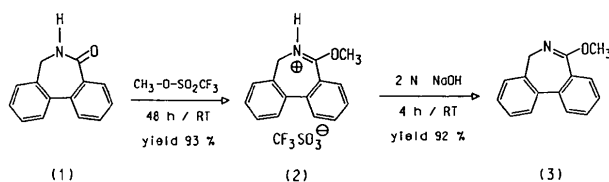
0.71073 Å, μ = 0.08 mm⁻¹, *T* = 291 (1) K, final *R* = 0.048 for 1311 unique observed [*F* ≥ 3.0σ(*F*)] diffractometer data. The central seven-membered ring has a boat conformation. The angle [41.47 (9)°] between

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the two benzenoid rings fused to the heterocycle and the torsion angle N(6)—C(7)—C(7a)—C(11a) [40.0 (4)°] indicate that conjugation of the cyclic CN double bond [1.266 (3) Å] with the biphenyl system is not efficient. The angle N(6)—C(7)—C(7a) [129.3 (2)°] has an unusually large value and the angle O—C(7)—C(7a) is reduced to 110.3 (2)°. The remaining bond distances and angles are in the normal range.

Experimental. The title compound was obtained from the reactions shown below (Röhrkasten, 1990) and 1.0 g was dissolved in 6 ml hot and dry methanol, the solution was slowly cooled to 298 K and after one day large crystals precipitated. The mixture was then cooled over a longer period (7 d/278 K) in order to obtain small single crystals.



A crystal of size $\sim 0.08 \times 0.34 \times 0.24$ mm was used. D_m was not determined. Intensity data were collected with $\omega/2\theta$ scans, variable scan speed $1.00\text{--}10.19^\circ \text{ min}^{-1}$ in θ , scan width 1.2° + dispersion. A Nicolet R3m/V diffractometer with graphite-monochromated Mo $K\alpha$ radiation was used. The lattice parameters were determined from least-squares fit of 25 reflections with $2\theta_{\text{max}} = 28.82^\circ$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. The crystal diffracted weakly. Six standard reflections (400, 020, 006, $\bar{4}00$, $0\bar{2}0$, $00\bar{6}$) were recorded every 300 reflections, only random deviations were detected during 148.85 h of X-ray exposure; 3945 reflections with $3.0 \leq 2\theta \leq 45.0^\circ$, $-9 \leq h \leq 9$, $0 \leq k \leq 12$, $-14 \leq l \leq 14$ were measured. The data were corrected for Lorentz-

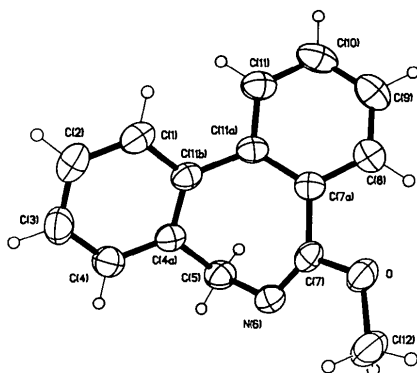


Fig. 1. General view (SHELXTL-Plus graphic) of the molecule, showing the atom-numbering scheme.

Table 1. Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^4$)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
O	0.8722 (2)	0.1856 (2)	0.7703 (1)	461
C(1)	0.2827 (3)	0.3428 (2)	0.6030 (2)	471
C(2)	0.1916 (3)	0.3001 (3)	0.5107 (2)	559
C(3)	0.2681 (3)	0.2764 (3)	0.4268 (2)	549
C(4)	0.4369 (3)	0.2957 (2)	0.4344 (2)	463
C(4a)	0.5308 (3)	0.3399 (2)	0.5262 (2)	371
C(5)	0.7151 (3)	0.3576 (2)	0.5375 (2)	438
N(6)	0.8045 (2)	0.2646 (2)	0.6054 (2)	416
C(7)	0.7932 (3)	0.2693 (2)	0.7017 (2)	379
C(7a)	0.7064 (3)	0.3588 (2)	0.7581 (2)	360
C(8)	0.7810 (3)	0.3950 (2)	0.8582 (2)	449
C(9)	0.7076 (4)	0.4798 (2)	0.9130 (2)	556
C(10)	0.5570 (4)	0.5290 (2)	0.8691 (2)	563
C(11)	0.4794 (3)	0.4921 (2)	0.7717 (2)	476
C(11a)	0.5498 (3)	0.4061 (2)	0.7138 (2)	370
C(11b)	0.4541 (3)	0.3627 (2)	0.6127 (2)	372
C(12)	0.9664 (3)	0.0962 (2)	0.7256 (2)	576

Table 2. Bond distances (\AA), bond angles, torsion angles ($^\circ$), least-squares planes and dihedral angle ($^\circ$)

O—C(7)	1.369 (3)	N(6)—C(7)	1.266 (3)
O—C(12)	1.437 (3)	C(7)—C(7a)	1.484 (3)
C(1)—C(2)	1.380 (4)	C(7a)—C(8)	1.393 (3)
C(1)—C(11b)	1.403 (3)	C(7a)—C(11a)	1.411 (3)
C(2)—C(3)	1.367 (4)	C(8)—C(9)	1.378 (4)
C(3)—C(4)	1.383 (4)	C(9)—C(10)	1.377 (4)
C(4)—C(4a)	1.390 (3)	C(10)—C(11)	1.374 (4)
C(4a)—C(5)	1.501 (3)	C(11)—C(11a)	1.397 (4)
C(4a)—C(11b)	1.396 (4)	C(11a)—C(11b)	1.486 (3)
C(5)—N(6)	1.468 (3)		
C(7)—O—C(12)	115.7 (2)	C(7)—C(7a)—C(11a)	121.7 (2)
C(2)—C(1)—C(11b)	121.0 (3)	C(7)—C(7a)—C(8)	118.7 (3)
C(1)—C(2)—C(3)	120.1 (3)	C(8)—C(7a)—C(11a)	119.6 (2)
C(2)—C(3)—C(4)	120.0 (3)	C(7a)—C(8)—C(9)	121.1 (3)
C(3)—C(4)—C(4a)	120.9 (3)	C(8)—C(9)—C(10)	119.7 (3)
C(4)—C(4a)—C(11b)	119.5 (3)	C(9)—C(10)—C(11)	120.0 (3)
C(4)—C(4a)—C(5)	121.9 (2)	C(10)—C(11)—C(11a)	121.9 (3)
C(5)—C(4a)—C(11b)	118.6 (2)	C(7a)—C(11a)—C(11)	117.7 (2)
C(4a)—C(5)—N(6)	110.8 (2)	C(11)—C(11a)—C(11b)	119.4 (3)
C(5)—N(6)—C(7)	116.6 (2)	C(7a)—C(11a)—C(11b)	122.8 (2)
O—C(7)—N(6)	120.3 (2)	C(4a)—C(11b)—C(11a)	121.6 (3)
N(6)—C(7)—C(7a)	129.3 (2)	C(1)—C(11b)—C(11a)	119.8 (3)
O—C(7)—C(7a)	110.3 (2)	C(1)—C(11b)—C(4a)	118.6 (3)
C(12)—O—C(7)—N(6)	0.6 (4)	C(5)—N(6)—C(7)—C(7a)	-2.4 (4)
C(12)—O—C(7)—C(7a)	-177.7 (2)	N(6)—C(7)—C(7a)—C(11a)	40.0 (4)
C(5)—C(4a)—C(11b)—C(11a)	1.4 (4)	O—C(7)—C(7a)—C(11a)	-141.9 (3)
C(11b)—C(4a)—C(5)—N(6)	70.3 (3)	C(7)—C(7a)—C(11a)—C(11b)	5.5 (4)
C(4a)—C(5)—N(6)—C(7)	-69.2 (3)	C(7a)—C(11a)—C(11b)—C(4a)	-42.8 (4)
C(5)—N(6)—C(7)—O	179.7 (2)		

No.	Plane through atoms	Distances from plane, \AA
1	C(1), C(2), C(3), C(4), C(4a), C(11b)	0.001 (3), 0.004 (3), -0.001 (3), -0.005 (3), 0.008 (2), -0.007 (3)
2	C(7a), C(8), C(9), C(10), C(11), C(11a)	0.015 (3), -0.010 (3), -0.004 (3), 0.011 (3), -0.001 (3), -0.010 (3)

Dihedral angle: 1,2 41.47 (9)

polarization but not for absorption effects and averaged ($R_{\text{int}} = 0.070$) to 1515 unique reflections, 1311 of which had $F \geq 3.0\sigma(F)$. The systematic absences ($h0l$) $l = 2n + 1$, ($0k0$) $k = 2n + 1$ conform to space group $P2_1/c$. The structure was solved *via* direct methods and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and a common isotropic one for the H atoms, which were placed in geometrically calculated positions (C—H 0.96 Å). 155 parameters were refined. Weights $w = 1.0/[\sigma^2(F) + (0.000194F^2)]$ led to a featureless analy-

sis of variance in terms of $\sin\theta$ and F_o . The refinement converged to $S = 1.96$, $R = 0.048$, $wR = 0.045$, $(\Delta/\sigma)_{\max} = 0.001$ (no extinction correction). The largest peaks in final $\Delta\rho$ map were ± 0.2 (1) $e \text{ \AA}^{-3}$. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1. Positional parameters and the equivalent isotropic values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes and dihedral angles are given in Table 2.

Related literature. 5H-Dibenz[*c,e*]azepines substituted with acceptor or donor groups at the α -posi-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53435 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

tion of the heteroatom have been attracting increasing interest (Kreher & Morgenstern, 1983). Investigation of the conformational changes in the seven-membered heterocyclic system with a cyclic CN double bond (Röhrkasten, 1990) on the one hand and with an exocyclic CO double bond (Wahl, Wildonger & Bordner, 1973; 6-methyl-6,7-dihydro-5H-dibenz[*c,e*]azepin-5-one) on the other hand was the main purpose of the present investigation.

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Synthesis of a 2-Oxabrendane Derivative via Reaction of *endo*-5-Acetyl-7,7-dimethoxynorborn-2-ene with *N*-Bromosuccinimide

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Abstract. 9-Bromo-3-dibromomethylene-2-oxatricyclo[4.2.1.0^{4,8}]nonan-7-one dimethyl acetal (3), $C_{11}H_{13}Br_3O_3$, $M_r = 432.95$, triclinic, $P\bar{1}$, $a = 6.532$ (2), $b = 9.193$ (3), $c = 12.230$ (4) \AA , $\alpha = 78.02$ (3), $\beta = 79.08$ (2), $\gamma = 71.27$ (2) $^\circ$, $V = 674.3$ (4) \AA^3 , $Z = 2$, $D_x = 2.132$ g cm^{-3} , $\lambda(\text{Mo } K\alpha) = 0.71073$ \AA , $\mu = 88.80$ cm^{-1} , $F(000) = 416$, $T = 295$ K, $R = 0.0571$ for 2221 reflections. Compound (3) is composed of three fused five-membered rings in envelope conformations: a norbornane moiety with a five-membered ring fused along one side. The

fused five-membered ring results in a C(3)—C(7)—C(6) norbornane angle of 97.7 (4) $^\circ$ which is significantly smaller than the C(2)—C(1)—C(9) angle of 105.2 (5) $^\circ$. This leads to a decrease in the C(7)—C(8) distance of 1.501 (10) \AA relative to the C(1)—C(8) distance of 1.531 (7) \AA .

Experimental. The synthesis of compound (3) is indicated in the Scheme. Compound (1) was prepared via the lithium–ammonia promoted dechlorination of 1,2,3,4-tetrachloro-*endo*-5-acetyl-7,7-dimethoxynorborn-2-ene (2) (McBee, Dively & Burch, 1955). A refluxing carbon tetrachloride solu-

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