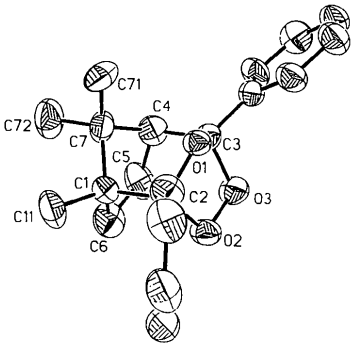

Fig. 1. *ORTEP* (Johnson, 1965) drawing at 50% probability of *endo,endo*-2,3-diphenylbornane-2,3-ozonide showing the adopted labeling. The labels for the phenyl carbons have been omitted for clarity and are assigned C21–C26 on C2 and C32–C36 on C3.

NBEPXP10) in which the ozonide ring is part of a more complex fused-ring system and their range of structural parameters includes those of the title structure [*cf.* average C—O(—C) = 1·420 (9), C—O(—O) = 1·448 (22) and O—O = 1·475 Å in the CSD files and 1·419, 1·454 and 1·467 Å for those parameters in the title structure; bond angles are more variable in the literature but the ranges include our values]. Far less common are those structural determinations in which the ozonide ring is 'strain-free', *i.e.* not part of a larger fused system (Groth, 1970; Schaap, Siddiqui, Prasad, Rahman & Oliver, 1984). Hitchcock & Beheshti (1979) have discussed O—O bond lengths according to ring size. Asymmetry parameters (ΔC_s and ΔC_2) are described by Ladd & Palmer (1985).

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8-(*N*-Methyl-*N*-*p*-tolylsulfonylamino)bicyclo[4.2.0]octan-7-one

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Abstract. $C_{16}H_{21}NO_3S$, $M_r = 307{\cdot}41$, triclinic, $P\bar{1}$, $a = 6{\cdot}473$ (6), $b = 16{\cdot}012$ (6), $c = 15{\cdot}791$ (6) Å, $\alpha = 106{\cdot}22$ (3), $\beta = 92{\cdot}83$ (6), $\gamma = 91{\cdot}22$ (6)°, $V = 1569$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}30$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 2{\cdot}17$ cm^{-1}, $F(000) = 656$, $T = 291$ K, $R = 0{\cdot}053$ for 2956 observed reflections. The two independent molecules in the asymmetric unit have similar geometry. The configuration of the substituent at C8 is *exo*. The puckering of the cyclobutanone ring as indicated by the dihedral angle about the C1—C7 diagonal is 31 (1)° in molecule *A* and 29 (1)° in molecule *B*.

Experimental. The title compound results from a (2 + 2) cycloaddition of a keteniminium salt to cyclohexene. Because stereochemical assignments in four-membered ring systems based on ^{1}H NMR coupling constants are ambiguous, an X-ray analysis was undertaken in order to assess the configuration at C8. Crystals obtained by evaporation from cyclo-

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2$)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Molecule *A*				
C1	−796 (6)	9291 (2)	1088 (3)	3·47 (8)
C2	−368 (6)	10273 (3)	1419 (3)	4·52 (9)
C3	−2118 (6)	10754 (3)	1919 (3)	4·71 (9)
C4	−4173 (6)	10499 (3)	1405 (3)	4·94 (10)
C5	−4700 (6)	9534 (3)	1268 (3)	5·11 (10)
C6	−3039 (6)	8947 (2)	771 (3)	4·27 (9)
C7	−2531 (6)	8201 (3)	1148 (3)	4·25 (9)
C8	−822 (6)	8745 (2)	1758 (3)	3·49 (8)
N9	1082 (5)	8368 (2)	1984 (2)	4·09 (7)
C10	1855 (6)	7626 (3)	1326 (3)	4·68 (9)
S11	1931 (2)	8568 (1)	3009 (1)	4·44 (2)
O12	4109 (4)	8471 (2)	3002 (2)	6·05 (7)
O13	1097 (5)	9376 (2)	3467 (2)	5·57 (7)
C14	874 (6)	7750 (3)	3424 (3)	3·89 (9)
C15	−1062 (7)	7837 (3)	3764 (3)	4·69 (10)
C16	−1870 (7)	7180 (3)	4071 (3)	5·00 (10)
C17	−798 (8)	6429 (3)	4032 (3)	5·01 (10)
C18	1124 (8)	6364 (3)	3699 (3)	5·22 (11)
C19	1980 (7)	7010 (3)	3391 (3)	4·65 (9)
C20	−1724 (9)	5713 (3)	4351 (3)	7·27 (13)
O21	−3010 (5)	7440 (2)	934 (2)	6·09 (7)
Molecule *B*				
C1	4627 (6)	4252 (2)	1104 (3)	3·71 (8)
C2	4454 (7)	5239 (3)	1447 (3)	4·98 (10)
C3	6435 (8)	5697 (3)	1859 (3)	5·77 (11)
C4	8215 (7)	5410 (3)	1268 (4)	6·08 (12)
C5	8586 (7)	4438 (3)	1119 (4)	6·36 (12)
C6	6650 (6)	3874 (3)	720 (3)	4·38 (9)
C7	6254 (6)	3148 (3)	1140 (3)	4·24 (9)
C8	4827 (6)	3714 (3)	1775 (3)	3·82 (8)
N9	2962 (5)	3359 (2)	2046 (2)	4·33 (7)
C10	1914 (6)	2596 (3)	1420 (3)	4·81 (9)
S11	2482 (2)	3575 (1)	3078 (1)	4·69 (3)
O12	3496 (5)	4397 (2)	3502 (2)	5·85 (7)
O13	297 (5)	3471 (2)	3113 (2)	6·12 (8)
C14	3661 (7)	2776 (3)	3487 (3)	4·18 (9)
C15	2536 (7)	2016 (3)	3464 (3)	4·79 (10)
C16	3507 (8)	1379 (3)	3755 (3)	5·43 (11)
C17	5538 (8)	1485 (3)	4086 (3)	5·20 (11)
C18	6605 (7)	2246 (3)	4112 (3)	5·40 (11)
C19	5698 (7)	2895 (3)	3815 (3)	4·80 (10)
C20	6558 (9)	774 (4)	4393 (4)	7·72 (14)
O21	6664 (4)	2395 (2)	953 (2)	5·91 (7)

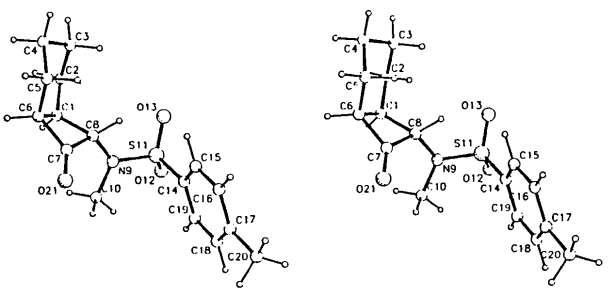

Fig. 1. Stereoscopic view of molecule *A*.

hexane. D_m not measured. Parallelepiped crystal with dimensions 0·40 × 0·16 × 0·06 mm. Lattice parameters refined using 15 reflections in the range $5 \le 2\theta \le 25°$. Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation. 4643 $h \pm k \pm l$ independent reflections with $\sin\theta/\lambda \le 0{\cdot}56$ Å^{-1}; $0 \le h \le 7$, $-17 \le k \le 17$, $-17 \le l \le 17$, 2956 with $I \ge 2{\cdot}5\sigma(I)$. Standard reflection ($0\bar{6}4$) checked every 50 reflections; no significant deviation. Structure solved by *SHELXS*86 (Sheldrick, 1985). All H atoms in idealized positions (C—H = 1·08 Å, H—C—H = 109·5°). Anisotropic block-diagonal least-squares refinement (*SHELX*76, Sheldrick, 1976) using F; H isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0{\cdot}00021F^2)$. $R = 0{\cdot}053$, $wR = 0{\cdot}053$ for 2956 observed reflections. Final maximum shift to e.s.d. = 0·02. $S = 1{\cdot}84$. Maximum and minimum heights in final difference Fourier synthesis = 0·30 and −0·27 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Table 2. *Bond lengths* (Å) *and angles* (°)

	A	*B*
C2—C1	1·528 (5)	1·530 (5)
C6—C1	1·550 (5)	1·534 (5)
C8—C1	1·548 (6)	1·544 (6)
C3—C2	1·511 (6)	1·488 (6)
C4—C3	1·510 (6)	1·516 (7)
C5—C4	1·528 (6)	1·533 (7)
C6—C5	1·539 (6)	1·529 (6)
C7—C6	1·512 (7)	1·514 (7)
C8—C7	1·515 (5)	1·516 (5)
O21—C7	1·199 (5)	1·197 (5)
N9—C8	1·457 (5)	1·457 (5)
C10—N9	1·459 (5)	1·468 (5)
S11—N9	1·625 (3)	1·616 (4)
O12—S11	1·421 (3)	1·428 (3)
O13—S11	1·427 (3)	1·426 (3)
C14—S11	1·759 (5)	1·758 (5)
C15—C14	1·382 (6)	1·395 (6)
C19—C14	1·388 (6)	1·381 (6)
C16—C15	1·379 (7)	1·380 (7)
C17—C16	1·389 (7)	1·379 (7)
C18—C17	1·369 (7)	1·378 (7)
C20—C17	1·501 (8)	1·508 (8)
C19—C18	1·377 (7)	1·385 (7)
C6—C1—C2	118·6 (3)	119·5 (3)
C8—C1—C2	119·7 (3)	119·0 (3)
C8—C1—C6	88·5 (3)	88·9 (3)
C3—C2—C1	113·0 (3)	113·2 (4)
C4—C3—C2	111·5 (3)	111·4 (4)
C5—C4—C3	110·5 (4)	111·2 (4)
C6—C5—C4	112·1 (4)	111·9 (4)
C5—C6—C1	113·4 (3)	114·2 (3)
C7—C6—C1	85·7 (3)	86·6 (3)
C7—C6—C5	113·2 (4)	113·2 (4)
C8—C7—C6	91·1 (3)	90·7 (3)
O21—C7—C6	133·9 (4)	133·8 (4)
O21—C7—C8	133·6 (4)	134·7 (4)
C7—C8—C1	85·6 (3)	86·1 (3)
N9—C8—C1	119·5 (3)	119·3 (3)
N9—C8—C7	122·0 (3)	122·0 (3)
C10—N9—C8	118·0 (3)	117·7 (3)
S11—N9—C8	120·3 (2)	120·7 (2)
S11—N9—C10	119·2 (3)	118·6 (3)
O12—S11—N9	106·6 (2)	106·1 (2)
O13—S11—N9	106·2 (2)	106·8 (2)
O13—S11—O12	120·4 (2)	119·8 (2)
C14—S11—N9	107·4 (2)	106·9 (2)
C14—S11—O12	107·6 (2)	108·5 (2)
C14—S11—O13	108·1 (2)	108·0 (2)
C15—C14—S11	120·2 (3)	119·5 (3)
C19—C14—S11	119·5 (3)	120·1 (3)
C19—C14—C15	120·2 (4)	120·4 (4)
C16—C15—C14	119·1 (4)	118·8 (4)
C17—C16—C15	121·5 (4)	121·7 (5)
C18—C17—C16	118·1 (5)	118·2 (5)
C20—C17—C16	120·7 (5)	120·4 (5)
C20—C17—C18	121·2 (5)	121·4 (5)
C19—C18—C17	121·9 (4)	121·9 (4)
C18—C19—C14	119·2 (4)	118·9 (4)

The atomic parameters are given in Table 1.* The bond lengths and angles are listed in Table 2. Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*, Motherwell & Clegg, 1978).

Related literature. There are only eight structures which contain the bicyclo[4.2.0]octan-7-one skeleton (Cambridge Structural Database, version 4.10: Allen *et al.*, 1979). From these, 8-[(2,2-dimethyl-3-oxocyclohexyl)hydroxymethyl]-1-methylbicyclo[4.2.0]-octan-7-one (Fair, Clark & Nikaido, 1985) is the only structure with monosubstitution at C8; in this molecule the puckering of the cyclobutanone ring is 26·1 (2)°.

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52873 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 2,3-Dimethylquinoxaline

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Abstrast. $C_{10}H_{10}N_2$, $M_r = 158{\cdot}20$, monoclinic, $C2/c$, $a = 7{\cdot}641\ (2)$, $b = 9{\cdot}896\ (1)$, $c = 11{\cdot}480\ (1)$ Å, $\beta = 99{\cdot}10\ (1)°$, $V = 857{\cdot}12\ (25)$ Å^3, $Z = 4$, $D_x = 1{\cdot}226$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}699$ cm^{-1}, $F(000) = 336$, $T = 291$ K, $R = 0{\cdot}0396$ for 623 unique observed reflections. The structure consists of 2,3-dimethylquinoxaline molecules oriented about the twofold axes. The molecule is planar.

Experimental. Crystals of 2,3-dimethylquinoxaline (hereafter abbreviated DMQ) were crystallized from acetonitrile. An Enraf–Nonius CAD-4 diffractometer was used with graphite-monochromatized Mo $K\alpha$ radiation. The crystal size was 0·20 × 0·30 × 0·35 mm. Unit-cell parameters were obtained by least-squares fit of the setting angles of 25 reflections in the θ range $3 < 2\theta < 13°$. The intensities of 4105 reflections were measured ($\sin\theta \le 30°$, $-9 \le h \le 9$, $0 \le k \le 11$, $0 \le l < 13$, ω–2θ scan mode). No significant variation (< 3%) was found in the intensities of the intensity control reflections $\bar{3}12$, $\bar{2}22$ and 110. The data were corrected for Lorentz and polarization effects but no absorption correction was applied. 1654 reflections with $|F| \ge 3\sigma(F)$ were used in the calculations, $R_{int} = 0{\cdot}040$. The structure was solved with multisolution direct methods (Sheldrick, 1986), and refined using full-matrix least-squares methods (Sheldrick, 1976), minimizing $\sum w(|F_o| - |F_c|)^2$, $w = 6{\cdot}8151/[\sigma^2(F) + 0{\cdot}00007F^2]$. Heavy atoms were refined with anisotropic and H atoms with isotropic temperature factors; 76 parameters were varied. The refinement converged to $R = 0{\cdot}0396$, $wR = 0{\cdot}0402$. $(\Delta/\sigma)_{max} = 0{\cdot}002$, $(\Delta/\sigma)_{mean} = 0{\cdot}001$ and $(\Delta\rho)_{max}/(\Delta\rho)_{min} = +0{\cdot}12/-0{\cdot}12$ e Å^{-3}. Fractional coordinates and equivalent isotropic temperature coefficients for non-H atoms are given in Table 1.* The

* Lists of atomic parameters for H atoms, anisotropic thermal parameters for non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52920 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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