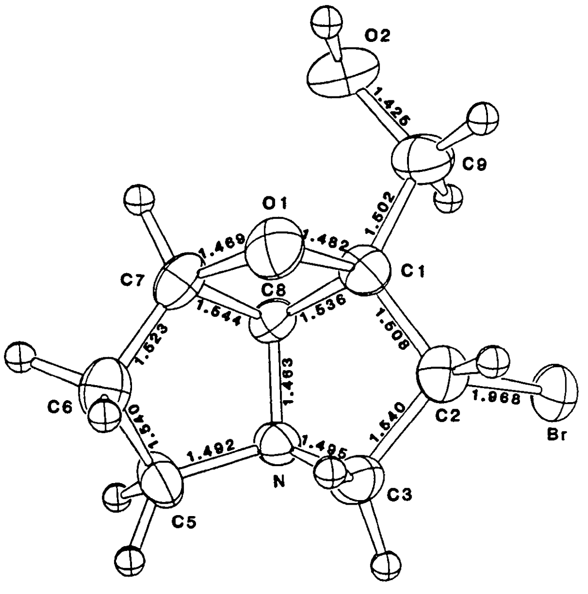

Fig. 1. 2α-Bromo-1β,7β-epoxytrachelanthamidine (the bond-length errors are within 0·007 Å).

C(7) and O(1), has all angles close to 90° (±2·9°) with O(1) 0·233 Å out of the plane of C(1), C(8) and C(7).

The intermolecular distance O(2)···N′ (N′ at $-1+x, y, z$) of 2·826 Å is below the combined van der Waals radii and the distance H(O2)···N′ of 1·82 Å is considerably less than the combined van der Waals radii (Hamilton & Ibers, 1968) suggesting the existence of a hydrogen bond.

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References

Bates, R. B. & Morehead, S. R. (1972). *Tetrahedron Lett.* pp. 1629–1630.
Gelbaum, L. T., Glinski, J. A., VanDerveer, D. & Zalkow, L. H. (1985). *Acta Cryst.* C**41**, 1342–1345.
Gelbaum, L. T., Gordon, M. M., Miles, M. & Zalkow, L. H. (1982). *J. Org. Chem.* **47**, 2501–2504.
Glinski, J. A., VanDerveer, D. & Zalkow, L. H. (1985). *Acta Cryst.* C**41**, 1345–1348.
Hamilton, W. C. & Ibers, J. A. (1968). *Hydrogen Bonding in Solids*, p. 16. New York: Benjamin.
Huxtable, R. J. (1979). *Gen. Pharmacol.* **10**, 159–167.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
Roitman, J. N. (1983). *Am. Chem. Soc. Symp. Ser.* **234**, 345–378.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Warren, F. L. (1970). *The Alkaloids*, Vol. XII, edited by R. H. F. Manske, ch. 4, pp. 246–262. London: Academic Press.
Warren, F. L. & Von Klemperer, M. E. (1958). *J. Chem. Soc.* pp. 4574–4575.
Yunusov, S. Yu. & Akramov, S. T. (1955). *Zh. Obshch. Khim.* **25**, 1813–1820.
Zalkow, L. H., Glinski, J. A., Gelbaum, L. T., Fleischmann, T. J., McGowan, L. S. & Gordon, M. M. (1985). *J. Med. Chem.* In the press.

Acta Cryst. (1986). C**42**, 1239–1242

Structure of 5,5,6-Trimethyl-1,4-diphenyl-2,3,7-trioxa[2.2.1]bicycloheptane

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Abstract. $C_{19}H_{20}O_3$, $M_r = 296{\cdot}37$, monoclinic, $P2_1/n$, $a = 19{\cdot}078\,(7)$, $b = 7{\cdot}149\,(1)$, $c = 24{\cdot}221\,(4)$ Å, $\beta = 104{\cdot}37\,(4)°$, $V = 3199{\cdot}9$ Å^3, $Z = 8$, $D_x = 1{\cdot}230$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}80$ cm^{-1}, $F(000) = 1264$, $T = 298$ K, final $R = 0{\cdot}038$ for 1656 unique observed reflections based on $I > 3\sigma(I)$. The two molecules of the asymmetric unit are very similar, including the torsion angles of the phenyl groups. Relatively long O—O bond lengths of 1·484 (4) and 1·485 (3) Å and small C—O—C angles of 95·7 (3) and 96·7 (3)° are observed.

Introduction. We recently reported (Kirschenheuter & Griffin, 1983) that the photoinduced 9,10-dicyanoanthracene (DCA) sensitized photooxidation of 3,3,4-trimethyl-1,2-diphenylcyclobutene (1) gives a pair of epimeric ozonides: 5,5,6-trimethyl-1,4-diphenyl-2,3,7-trioxa[2.2.1]bicycloheptanes (2) and (3), respectively. These epimeric ozonides were tentatively assigned the

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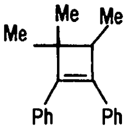
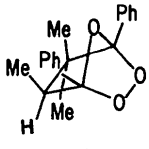
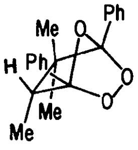
structures designated below on the basis of the high- and low-field methyl doublet signals present in their respective ^{1}H NMR spectra. In view of recent reports (Miura, Ikegami, Nojima, Kusabayashi, McCullough & Nagase, 1983) of X-ray crystallographic structural studies on several ozonides we wish to describe here our X-ray structural data obtained on (2) which confirm the tentative gross structural as well as stereochemical assignments made on the basis of ^{1}H and ^{13}C NMR spectroscopy (Kirschenheuter & Griffin, 1983).



(1)



(2)



(3)

Experimental. Suitable crystal of approximate dimensions 0·20 × 0·30 × 0·40 mm recrystallized from methanol; Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares fit of 25 carefully measured reflections on the diffractometer with $18 < 2\theta \leq 34°$; systematic absences $0k0$, $k = 2n + 1$ and $h0l$, $h + l = 2n + 1$ confirmed the space group $P2_1/n$; $2\theta_{\max} = 40°$ $(0 \leq h \leq 18,\ 0 \leq k \leq 6,\ -23 \leq l \leq 23)$; intensities of 2966 unique reflections measured, of which 1656 treated as observed in the refinement based on $I > 3\sigma(I)$; $\sigma(I)$ from counting statistics; $\omega{:}2\theta$ scan mode; three standards monitored every 2 h show no significant variation in the intensity; orientation control every 200 reflections; empirical absorption correction *via* ψ scans and Lorentz–polarization correction applied. Initial attempts to solve the structure by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982; Frenz, 1982) based on 359 largest E values, $1{\cdot}57 \leq E \leq 3{\cdot}57$, were unsuccessful. However, after a careful survey of several E maps, a 22-atom fragment from one of the E maps was taken as a correctly orientated fragment and input to *MULTAN* which ultimately gave positions of all the non-hydrogen atoms of the two molecules present in the asymmetric unit. The methylene H atoms were calculated by assuming tetrahedral geometry and a C—H distance of 0·95 Å; other H atoms located in subsequent difference Fourier syntheses. Full-matrix least-squares refinement of the complete structure converged to $R = 0{\cdot}038$, $wR = 0{\cdot}044$, $S = 1{\cdot}68$, $(\Delta/\sigma)_{\max} = 0{\cdot}16$; minimizing the function $\sum w(|\,|F_o| - |F_c|\,|)^2$, $w = 1/\sigma^2(F)$ where $\sigma^2(F^2) = \sigma^2_{\rm c.s.} + (0{\cdot}03F^2)^2$. No significant peaks in the final difference Fourier map (highest peak 0·18 e Å^{-3}). Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). No correction for extinction. All calculations performed on a PDP 11/34 computer using the *SDP* program system (Frenz, 1982).

Table 1. *Positional parameters and equivalent isotropic thermal parameters, with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)].$$

	x	y	z	$B_{\rm eq}$(Å^2)
O(1)	0·2438 (2)	0·2137 (4)	0·5867 (1)	3·62 (7)
O(1′)	−0·0897 (2)	0·1954 (4)	0·8435 (1)	4·23 (8)
O(2′)	−0·0002 (2)	0·3497 (5)	0·9028 (1)	5·40 (9)
O(2)	0·1685 (2)	0·1714 (5)	0·6434 (1)	5·09 (9)
O(3′)	0·0113 (2)	0·3516 (5)	0·8444 (1)	5·51 (9)
O(3)	0·1261 (2)	0·2143 (5)	0·5846 (1)	5·22 (9)
C(1′)	−0·0781 (2)	0·3115 (7)	0·8921 (2)	4·1 (1)
C(1)	0·2374 (2)	0·1040 (7)	0·6337 (2)	3·6 (1)
C(2)	0·2244 (3)	−0·0937 (7)	0·6103 (2)	4·3 (1)
C(2′)	−0·1169 (3)	0·4940 (7)	0·8703 (2)	4·9 (1)
C(3′)	−0·1061 (3)	0·4984 (7)	0·8088 (2)	4·8 (1)
C(3)	0·1801 (3)	−0·0527 (7)	0·5480 (2)	4·1 (1)
C(4)	0·1757 (2)	0·1618 (7)	0·5508 (2)	3·8 (1)
C(4′)	−0·0606 (2)	0·3194 (7)	0·8093 (2)	4·1 (1)
C(5′)	−0·0534 (2)	0·2356 (7)	0·7548 (2)	4·0 (1)
C(5)	0·1563 (2)	0·2746 (7)	0·4971 (2)	3·9 (1)
C(6)	0·2091 (3)	0·3344 (7)	0·4714 (2)	4·7 (1)
C(6′)	−0·1006 (3)	0·1071 (7)	0·7250 (2)	5·0 (1)
C(7′)	−0·0945 (3)	0·0289 (8)	0·6750 (2)	6·0 (2)
C(7)	0·1915 (3)	0·4298 (8)	0·4214 (2)	5·8 (2)
C(8)	0·1223 (3)	0·4724 (8)	0·3965 (2)	6·3 (2)
C(8′)	−0·0368 (3)	0·0821 (8)	0·6543 (2)	6·7 (2)
C(9′)	0·0129 (3)	0·2112 (9)	0·6826 (2)	6·5 (2)
C(9)	0·0665 (3)	0·4196 (8)	0·4220 (2)	6·4 (2)
C(10)	0·0845 (3)	0·3194 (8)	0·4719 (2)	5·5 (1)
C(10′)	0·0043 (3)	0·2868 (8)	0·7333 (2)	5·1 (1)
C(11′)	−0·0878 (2)	0·2181 (6)	0·9446 (2)	3·9 (1)
C(11)	0·2930 (2)	0·1447 (7)	0·6871 (2)	3·6 (1)
C(12)	0·2927 (3)	0·0511 (7)	0·7368 (2)	4·9 (1)
C(12′)	−0·0789 (3)	0·3222 (7)	0·9944 (2)	4·6 (1)
C(13′)	−0·0824 (3)	0·2328 (8)	1·0445 (2)	5·6 (1)
C(13)	0·3433 (3)	0·0907 (7)	0·7870 (2)	5·3 (1)
C(14′)	−0·0933 (3)	0·0439 (8)	1·0455 (2)	5·7 (2)
C(14)	0·3944 (3)	0·2235 (7)	0·7878 (2)	5·2 (1)
C(15′)	−0·1025 (3)	−0·0558 (8)	0·9964 (2)	5·9 (2)
C(15)	0·3957 (3)	0·3160 (8)	0·7383 (2)	5·4 (1)
C(16′)	−0·0991 (3)	0·0288 (7)	0·9462 (2)	4·8 (1)
C(16)	0·3458 (3)	0·2775 (7)	0·6888 (2)	4·3 (1)
C(17′)	−0·1926 (3)	0·500 (1)	0·8767 (3)	8·6 (2)
C(17)	0·2929 (3)	−0·2064 (8)	0·6173 (2)	6·8 (2)
C(18′)	−0·1751 (3)	0·4864 (9)	0·7621 (2)	7·5 (2)
C(18)	0·2184 (3)	−0·1097 (8)	0·5036 (2)	6·1 (2)
C(19)	0·1053 (3)	−0·1451 (9)	0·5346 (2)	6·9 (2)
C(19′)	−0·0643 (4)	0·6736 (8)	0·7989 (2)	7·7 (2)

Discussion. The atomic coordinates are listed in Table 1. Bond distances and angles are given in Table 2, along with a few torsion angles of interest.*

An *ORTEP* plot (Johnson, 1976) of the conformation of one of the two molecules in the asymmetric unit is shown in Fig. 1. The bond distances and angles show in general good agreement between chemically equivalent distances as well as a very close similarity of the molecular geometries of the two molecules. Even the phenyl groups adopt identical conformations with respect to the ozonide ring system in both molecules.

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* Lists of structure factors, anisotropic thermal parameters, and H-atom bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42977 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

E.s.d.'s are given in parentheses; e.s.d.'s for torsion angles are 0·3–0·4°.

O(1)	C(1)	1·412 (4)	C(4′)	C(5′)	1·487 (6)
O(1)	C(4)	1·422 (4)	C(5′)	C(6′)	1·361 (6)
O(1′)	C(1′)	1·412 (5)	C(5′)	C(10′)	1·379 (6)
O(1′)	C(4′)	1·417 (5)	C(5)	C(6)	1·377 (6)
O(2′)	O(3′)	1·484 (4)	C(5)	C(10)	1·392 (6)
O(2′)	C(1′)	1·469 (5)	C(6)	C(7)	1·357 (7)
O(2)	O(3)	1·485 (3)	C(6′)	C(7′)	1·365 (7)
O(2)	C(1)	1·473 (4)	C(7′)	C(8′)	1·372 (8)
O(3′)	C(4′)	1·442 (5)	C(7)	C(8)	1·343 (7)
O(3)	C(4)	1·446 (4)	C(8)	C(9)	1·408 (8)
C(1′)	C(2′)	1·528 (6)	C(8′)	C(9′)	1·378 (8)
C(1′)	C(11′)	1·488 (5)	C(9′)	C(10′)	1·388 (7)
C(1)	C(2)	1·520 (6)	C(9)	C(10)	1·373 (7)
C(1)	C(11)	1·484 (6)	C(11′)	C(12′)	1·391 (6)
C(2)	C(3)	1·563 (6)	C(11′)	C(16′)	1·372 (6)
C(2)	C(17)	1·509 (7)	C(11)	C(12)	1·378 (6)
C(2′)	C(3′)	1·554 (6)	C(11)	C(16)	1·377 (6)
C(2′)	C(17′)	1·491 (8)	C(12)	C(13)	1·381 (7)
C(3′)	C(4′)	1·545 (6)	C(12′)	C(13′)	1·388 (7)
C(3′)	C(18′)	1·510 (8)	C(13′)	C(14′)	1·367 (7)
C(3′)	C(19′)	1·535 (8)	C(13)	C(14)	1·357 (7)
C(3)	C(4)	1·538 (6)	C(14′)	C(15′)	1·360 (7)
C(3)	C(18)	1·499 (7)	C(14)	C(15)	1·375 (7)
C(3)	C(19)	1·532 (7)	C(15′)	C(16′)	1·374 (7)
C(4)	C(5)	1·497 (6)	C(15)	C(16)	1·361 (7)

C(1)	O(1)	C(4)	95·7 (3)	O(3)	C(4)	C(5)	107·3 (4)
C(1′)	O(1′)	C(4′)	96·7 (3)	C(3)	C(4)	C(5)	120·2 (4)
O(3′)	O(2′)	C(1′)	102·5 (3)	O(1′)	C(4′)	O(3′)	102·0 (3)
O(3)	O(2)	C(1)	102·1 (3)	O(1′)	C(4′)	C(3′)	102·8 (3)
O(2′)	O(3′)	C(4′)	102·5 (3)	O(1′)	C(4′)	C(5′)	113·8 (4)
O(2)	O(3)	C(4)	102·2 (3)	O(3′)	C(4′)	C(3′)	108·8 (4)
O(1′)	C(1′)	O(2′)	101·5 (3)	O(3′)	C(4′)	C(5′)	107·7 (4)
O(1)	C(1′)	C(2′)	104·6 (4)	C(3′)	C(4′)	C(5′)	120·2 (4)
O(1′)	C(1′)	C(11′)	114·9 (4)	C(4′)	C(5′)	C(6′)	123·2 (5)
O(2′)	C(1′)	C(2′)	106·5 (4)	C(4′)	C(5′)	C(10′)	119·1 (5)
O(2′)	C(1′)	C(11′)	105·5 (3)	C(6′)	C(5′)	C(10′)	117·6 (5)
C(2′)	C(1′)	C(11′)	121·7 (4)	C(4)	C(5)	C(6)	120·6 (5)
O(1)	C(1)	O(2)	101·9 (3)	C(4)	C(5)	C(10)	120·6 (5)
O(1)	C(1)	C(2)	104·7 (4)	C(6)	C(5)	C(10)	118·8 (5)
O(1)	C(1)	C(11)	114·1 (4)	C(5)	C(6)	C(7)	120·8 (6)
O(2)	C(1)	C(2)	107·1 (4)	C(5′)	C(6′)	C(7′)	123·8 (3)
O(2)	C(1)	C(11)	105·7 (3)	C(6′)	C(7′)	C(8′)	117·6 (6)
C(2)	C(1)	C(11)	121·5 (4)	C(6)	C(7)	C(8)	120·8 (7)
C(1)	C(2)	C(3)	100·6 (4)	C(7)	C(8)	C(9)	120·5 (6)
C(1)	C(2)	C(17)	113·5 (5)	C(7′)	C(8′)	C(9′)	121·3 (6)
C(3)	C(2)	C(17)	117·0 (5)	C(8′)	C(9′)	C(10′)	118·9 (6)
C(1′)	C(2′)	C(3′)	100·4 (4)	C(8)	C(9)	C(10)	118·3 (6)
C(1′)	C(2′)	C(17′)	112·8 (5)	C(5)	C(10)	C(9)	120·7 (6)
C(3′)	C(2′)	C(17′)	117·5 (5)	C(5′)	C(10′)	C(9′)	120·8 (6)
C(2′)	C(3′)	C(4′)	100·6 (4)	C(1′)	C(11′)	C(12′)	119·1 (5)
C(2′)	C(3′)	C(18′)	114·7 (5)	C(1′)	C(11′)	C(16′)	121·4 (5)
C(2′)	C(3′)	C(19′)	111·3 (5)	C(12′)	C(11′)	C(16′)	119·3 (5)
C(4′)	C(3′)	C(18′)	110·0 (5)	C(1)	C(11)	C(12)	120·6 (5)
C(4′)	C(3′)	C(19′)	111·4 (5)	C(1)	C(11)	C(16)	121·4 (4)
C(18′)	C(3′)	C(19′)	108·6 (6)	C(12)	C(11)	C(16)	118·0 (5)
C(2)	C(3)	C(4)	99·6 (4)	C(11)	C(12)	C(13)	121·0 (6)
C(2)	C(3)	C(18)	113·5 (5)	C(11′)	C(12′)	C(13′)	119·3 (6)
C(2)	C(3)	C(19)	112·1 (5)	C(12′)	C(13′)	C(14′)	120·7 (6)
C(4)	C(3)	C(18)	110·1 (4)	C(12)	C(13)	C(14)	119·9 (6)
C(4)	C(3)	C(19)	112·4 (5)	C(13′)	C(14′)	C(15′)	119·3 (6)
C(18)	C(3)	C(19)	109·0 (5)	C(13)	C(14)	C(15)	119·5 (6)
O(1)	C(4)	O(3)	102·3 (3)	C(14′)	C(15′)	C(16′)	121·3 (6)
O(1)	C(4)	C(3)	103·6 (4)	C(14)	C(15)	C(16)	120·7 (6)
O(1)	C(4)	C(5)	112·5 (4)	C(11′)	C(16′)	C(15′)	120·1 (6)
O(3)	C(4)	C(3)	109·5 (4)	C(11)	C(16)	C(15)	120·8 (5)

Molecule 1		Molecule 2	
C(1)C(2)C(3)C(4)	−1·9	C(1′)C(2′)C(3′)C(4′)	−2·0
C(1)O(2)O(3)C(4)	1·2	C(1′)O(2′)O(3′)C(4′)	1·3
C(1)O(1)C(4)C(3)	−57·1	C(1′)O(1′)C(4′)C(3′)	−56·4
C(4)O(1)C(1)C(2)	56·1	C(4′)O(1′)C(1′)C(2′)	55·8
C(4)O(1)C(1)O(2)	−55·3	C(4′)O(1′)C(1′)O(2′)	−54·8
C(1)O(1)C(4)O(3)	56·8	C(1′)O(1′)C(4′)O(3′)	56·4
O(2)C(1)C(2)C(17)	−159·7	O(2′)C(1′)C(2′)C(17′)	−159·8
O(3)C(4)C(3)C(18)	168·1	O(3′)C(4′)C(3′)C(18′)	166·6
O(3)C(4)C(3)C(19)	46·4	O(3′)C(4′)C(3′)C(19′)	46·1
O(1)C(1)C(11)C(12)	178·7	O(1′)C(1′)C(11′)C(12′)	−178·8
O(1)C(4)C(5)C(10)	−146·6	O(1′)C(4′)C(5′)C(10′)	−145·1

Apparently the sterically least-hindered configuration for the phenyl rings [torsion angles O(1)C(1)C(11)-C(12) = 178·7 (4) and O(1)C(4)C(5)C(10) = −146·6 (4)° in one molecule, and 178·8 (4) and −145·1 (3)°, respectively, in the other molecule] is sufficiently favorable to overcome differences in crystal packing forces.

The average length of the peroxide O—O bond, 1·484 (3) Å, is larger than the values reported for less strained ozonides (Karban, McAtee, Belew, Mullica, Milligan & Korp, 1978; Mullica, Korp, Milligan, Belew, McAtee & Karban, 1979; Oliver, Mullica, Milligan, Karban, McAtee & Belew, 1979; Miura *et al.*, 1983), which fall in the range from 1·459 (7) to 1·469 (3) Å. The lengthening of the peroxide bond may be attributed to the high degree of planarity in the C—O—O—C region [C—O—O—C torsion angle 1·3 (4)°] which maximizes the repulsions between lone pairs on the peroxide oxygens. A long O—O distance of 1·501 (2) Å has also been reported for a similar molecule in which the bridging oxygen is replaced by a CH_2 group (Langs, Erman, DeTitta, Coughlin & Salomon, 1978). On the other hand, long O—O distances are also observed in small strained ring systems. For example, a peroxide bond length of 1·48 (1) Å [C—O—O—C torsion angle 21·3 (4)°], has been observed in a nonplanar dioxetene four-membered ring (Numan, Wieringa, Wynberg, Hess & Voss, 1977) and 1·483 (7) Å in a strained five-membered ring (Hitchcock & Beheshti, 1979).

The least-squares planes passing through C(1)C(2)C(3)C(4) [planar within 0·001 (2) Å] and C(1)O(2)O(3)C(4) [planar within 0·006 (2) Å] intersect at 111·4 (4)°. The bridging oxygen O(1) is 0·782 (3) Å away from the first plane and 0·789 (3) Å from the second plane forming two rings in half-chair conformations. The angle at the bridging oxygen, 96·2 (3)°, is significantly less than the corresponding

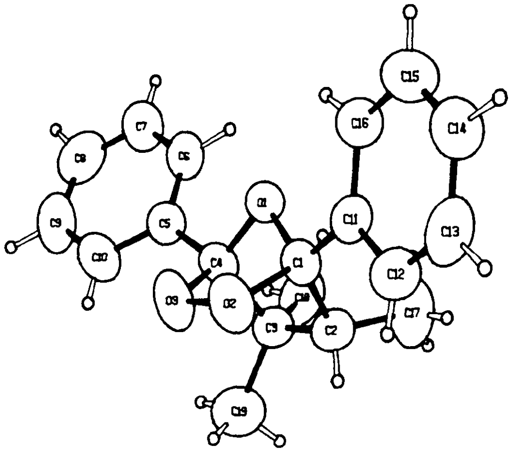
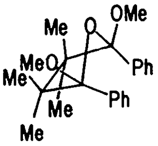


Fig. 1. Perspective view of one of the two molecules in the asymmetric unit showing the labeling scheme. Non-H atoms are represented by thermal ellipsoids drawn at 50% probability level.

C—O—C angles observed in other less stable ozonides which are in the range 101·8 (2)–118·0 (5)° (Miura *et al.*, 1983; McCullough, Walkinshaw & Nojima, 1981). The average C—O(1) distance, 1·416 (2) Å, is within the range of values reported for similar systems, (Groth, 1969; McConnel & Stevens, 1973; Karban *et al.*, 1978; Grand & Cadet, 1978; Mullica *et al.*, 1979; Oliver *et al.*, 1979; Foss, Gardner, Kirschenheuter, Griffin & Stevens, 1986).

The C(1)—C(11) [1·486 (5) Å] and C(4)—C(5) [1·492 (6) Å] bonds are significantly shorter than the corresponding distances (Foss *et al.*, 1986) [1·518 (3) and 1·519 (3) Å, respectively] in (4). This probably results from the distortion of C(1) and C(4) away from ideal tetrahedral geometry owing to the bicyclic ring system, resulting in reduced steric interaction with the phenyl rings. The additional freedom in (4) owing to the absence of the peroxide bond allows a twist about the C(2)—C(3) bond to minimize nonbonded repulsion between methyl groups (Foss *et al.*, 1986).



(4)

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References

FOSS, L. I., GARDNER, S. D., KIRSCHENHEUTER, G. P., GRIFFIN, G. W. & STEVENS, E. D. (1986). *Acta Cryst.* Submitted.

FRENZ, B. A. (1982). *The Enraf–Nonius CAD-4 Structure Determination Package*. In *Computing in Crystallography* edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI. Delft Univ. Press.

GRAND, A. & CADET, J. (1978). *Acta Cryst.* B**34**, 1524–1528.

GROTH, P. (1969). *Acta Chem. Scand.* **23**, 1311–1329.

HITCHCOCK, P. B. & BEHESHTI, I. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 126–129.

International Tables for X-ray Crystallography (1974). Vol IV, pp. 72, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.

KARBAN, J., MCATEE, J. L., BELEW, J. S., MULLICA, D. F., MILLIGAN, W. & KORP, J. (1978). *J. Chem. Soc. Chem. Commun.* pp. 729–730.

KIRSCHENHEUTER, G. P. & GRIFFIN, G. W. (1983). *J. Chem. Soc. Chem. Commun.* pp. 596–597.

LANGS, D. A., ERMAN, M. G., DETITTA, G. T., COUGHLIN, D. J. & SALOMON, R. (1978). *J. Cryst. Mol. Struct.* **5**, 239–246.

MCCONNEL, J. F. & STEVENS, J. D. (1973). *Cryst. Struct. Commun.* **2**, 619.

MCCULLOUGH, K. J., WALKINSHAW, M. D. & NOJIMA, M. J. (1981). *J. Chem. Res.* (*M*), pp. 4357–4370; (*S*), p. 369.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MIURA, M., IKEGAMI, A., NOJIMA, M., KUSABAYASHI, S., MCCULLOUGH, K. J. & NAGASE, S. (1983). *J. Am. Chem. Soc.* **105**, 2414–2426.

MULLICA, D. F., KORP, J. D., MILLIGAN, W. O., BELEW, J. S., MCATEE, J. L. & KARBAN, J. (1979). *J. Chem. Soc. Perkins Trans.* 2, pp. 1703–1707.

NUMAN, H., WIERINGA, J. H., WYNBERG, H., HESS, J. & VOSS, A. (1977). *J. Chem. Soc. Chem. Commun.* pp. 591–592.

OLIVER, J. D., MULLICA, D. F., MILLIGAN, W. O., KARBAN, J., MCATEE, J. L. & BELEW, J. S. (1979). *Acta Cryst.* B**35**, 2273–2276.

Acta Cryst. (1986). C**42**, 1242–1246

Structure of 2,4,4,8,8-Pentakis(dimethylamino)-9-methyl-6-methylamino-2,6-imino-$2\lambda^5,4\lambda^5,6\lambda^5,8\lambda^5$-cyclotetra(phosphazene) Monohydrate

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Abstract. $C_{12}H_{37}N_{11}P_4.H_2O$, $M_r = 477\cdot4$, triclinic, $P\bar{1}$, $a = 15\cdot181\ (4)$, $b = 9\cdot388\ (2)$, $c = 18\cdot103\ (5)$ Å, $\alpha = 86\cdot97\ (3)$, $\beta = 101\cdot84\ (3)$, $\gamma = 99\cdot75\ (3)°$, $Z = 4$, $D_x = 1\cdot274\ (1)$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha_1) = 1\cdot54056$ Å, $\mu = 29\cdot91$ cm^{-1}, $F(000) = 1024$, $T = 298$ K. $R = 0\cdot037$ based on 3558 observed reflections. There are two non-equivalent molecules of a bicyclic phosphazene each with a methylimino group bridging two P atoms. The two P—N bond lengths in the bridge are non-equivalent and are longer than any other P—N bonds in the compound. There are two water molecules in the asymmetric unit which are hydrogen-bonded together and these in turn bind the two non-equivalent phosphazene molecules together.

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