

C2	-0.1568 (1)	0.9195 (4)	0.5209 (2)	0.0436 (8)
C3	-0.1975 (1)	1.0409 (4)	0.4673 (2)	0.0432 (8)
C4	-0.1845 (1)	1.2508 (5)	0.4538 (2)	0.0577 (9)
C5	-0.1281 (1)	1.3149 (5)	0.5222 (2)	0.070 (1)
C6	-0.0842 (1)	1.1604 (5)	0.5466 (2)	0.068 (1)
C7	-0.0597 (1)	0.8361 (5)	0.6219 (2)	0.0513 (9)
C8	-0.0199 (1)	0.7488 (5)	0.5959 (2)	0.0500 (9)
C9	0.0240 (1)	0.6110 (5)	0.6587 (2)	0.0519 (9)
C10	0.0593 (1)	0.5060 (5)	0.6287 (2)	0.0524 (9)
C11	0.1080 (1)	0.3807 (4)	0.6904 (2)	0.0465 (8)
C12	0.2022 (1)	0.1500 (5)	0.7251 (2)	0.059 (1)
C13	0.1809 (1)	-0.0169 (5)	0.7557 (2)	0.059 (1)
C14	0.1466 (1)	0.0576 (5)	0.7931 (2)	0.0585 (10)
C15	-0.2542 (1)	0.9695 (5)	0.4237 (2)	0.0494 (9)

Miyake, M., Haramoto, Y. & Kamogawa, H. (1994). *Acta Cryst.* **C50**, 805–807.  
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Table 2. Selected geometric parameters (Å, °)

S1—C11	1.813 (3)	N1—C2	1.344 (4)
S1—C14	1.802 (3)	N1—C6	1.459 (4)
S2—C11	1.815 (3)	N1—C7	1.452 (4)
S2—C12	1.804 (3)	N2—C15	1.150 (4)
C11—S1—C14	99.9 (1)	N1—C7—C8	113.7 (2)
C11—S2—C12	100.3 (1)	S1—C11—S2	112.3 (2)
C2—N1—C6	118.3 (3)	S1—C11—C10	110.1 (2)
C2—N1—C7	122.2 (3)	S2—C11—C10	107.2 (2)
C6—N1—C7	119.4 (2)	S2—C12—C13	114.1 (2)
N1—C2—C3	124.1 (3)	S1—C14—C13	115.1 (2)
N1—C6—C5	112.4 (3)	N2—C15—C3	176.9 (3)

H atoms were placed in calculated positions. In the weighting scheme,  $\sigma^2(F^2) = S^2(C + R^2B) + (0.030F^2)/Lp^2$ , where  $S$  = scan rate,  $C$  = total integrated peak count,  $R$  = ratio of scan to background counting times,  $B$  = total background count, and  $Lp$  = Lorentz-polarization factor.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SAP191* (Fan, 1991). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: VJ1034). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 1-Cyanomethyl-6,7,8-trioxabicyclo[3.2.1]-octane

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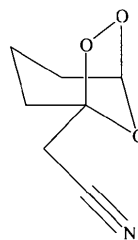
(Received 10 July 1995; accepted 3 October 1995)

## Abstract

The title nitrile (6,7,8-trioxabicyclo[3.2.1]octane-1-acetonitrile, C<sub>7</sub>H<sub>9</sub>NO<sub>3</sub>) was prepared by ozonolysis of cyclopentenylacetonitrile. Crystals of the compound display typical metric parameters.

## Comment

Ozonolysis of substituted cyclopentenes is known to generate primary ozonides that regioselectively fragment to the corresponding bicyclic ozonides (Bunnelle & Lee, 1992). Most of these bicyclic trioxanes have been analyzed by NMR and only a few have afforded crystals suitable for X-ray crystallography (Bunnelle, Isbell, Barnes & Qualls, 1991). During our studies on the reactivity of unsaturated nitriles (Fleming & Huang, unpublished results), we prepared the title compound, (I), by ozonolysis of cyclopentenylacetonitrile.



(I)

The six-membered pyran ring exists in a chair conformation, in accord with theoretical calculations (Bunnelle & Isbell, 1992). Recently, average parameters were published for bicyclic ozonides [O—O 1.473, C—(O—O) 1.443 and C—O 1.418 Å (Buckleton, Clark & Rickard, 1995), derived from the Cambridge Structural Database], and the metric parameters of (I) are quite similar. Buckleton, Clark & Rickard (1995) note that many of these ozonides display marked asymmetry in the C—O bonds involving the peroxy group, and similar asymmetry is found in the title compound. The underlying causes of the asymmetry in the different ozonides must be compound specific. For example, in two cases which have chemically equivalent substituents on the two ozonide ring C atoms, one exhibits asymmetry (Cummins, Drew, Mann & Walsh, 1983) while the other does not (Jerzykiewicz, Dzięwońska-Baran, Baran & Lis, 1993). In the title compound, we suggest that the asymmetry is the net result of three possible effects: an interplay between the ether and peroxide components of the ozonide; an inductive effect of the nitrile; a steric packing effect. The first of these seems the most likely since the asymmetry in the C—O bonds involving the peroxy group typically accompanies asymmetry in the C—O bonds of the ether linkage, and the C atom that participates in the shorter C—O ether bond usually participates in the longer C—O peroxy bond (Cummins *et al.*, 1983; Bunnelle *et al.*, 1991; Karban, McAtee Jr, Belew, Mullica, Milligan & Korp, 1978; Miura, Ikegami, Nojima, Kusabayashi, McCullough & Nagase, 1983).

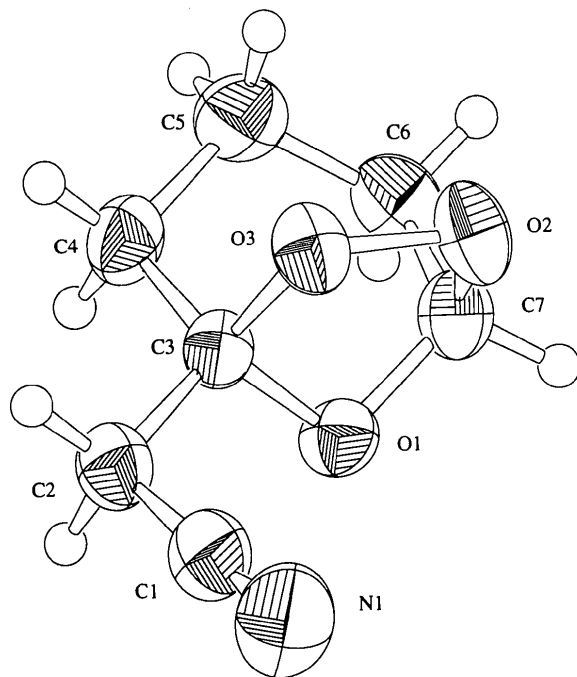


Fig. 1. A perspective drawing of the title compound with displacement ellipsoids drawn at the 50% probability level.

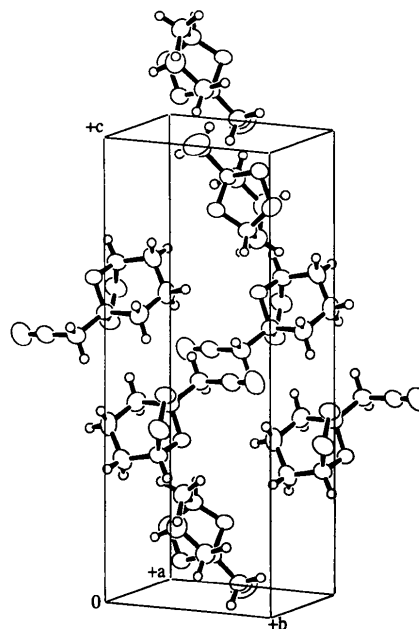


Fig. 2. A cell packing diagram with displacement ellipsoids drawn at the 50% probability level.

## Experimental

A stream of ozone was passed through a cold (195 K) dry  $\text{CH}_2\text{Cl}_2$  solution (5 ml) of cyclopentenylacetonitrile (112.3 mg, 1.05 mmol) until the solution became blue. Nitrogen was then passed through the solution until the excess ozone was displaced. The solution was allowed to warm to room temperature, and the solvent was removed under reduced pressure (rotary evaporator, vacuum pump) to afford 115.4 mg of the title compound (71%) as colorless crystals.

### Crystal data

$\text{C}_7\text{H}_9\text{NO}_3$   
 $M_r = 155.15$   
 Monoclinic  
 $P2_1/c$   
 $a = 5.8867(8) \text{ \AA}$   
 $b = 7.023(1) \text{ \AA}$   
 $c = 17.9952(7) \text{ \AA}$   
 $\beta = 92.473(8)^\circ$   
 $V = 743.2(1) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.386 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
 $\lambda = 0.7107 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 20.06\text{--}23.05^\circ$   
 $\mu = 0.109 \text{ mm}^{-1}$   
 $T = 294.2 \text{ K}$   
 Rhomboid  
 $0.55 \times 0.40 \times 0.20 \text{ mm}$   
 Colorless

### Data collection

AFC-7R diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  
 $\psi$  scans (TEXSAN;  
 Molecular Structure  
 Corporation, 1992b)  
 $T_{\min} = 0.962$ ,  $T_{\max} = 0.999$   
 2028 measured reflections  
 1853 independent reflections

1095 observed reflections  
 $[I > 3\sigma(I)]$   
 $R_{\text{int}} = 0.0642$   
 $\theta_{\max} = 27.50^\circ$   
 $h = 0 \rightarrow 7$   
 $k = 0 \rightarrow 9$   
 $l = -23 \rightarrow 23$   
 3 standard reflections  
 monitored every 150 reflections  
 intensity decay: 9.41%

## Refinement

Refinement on $F$	$(\Delta/\sigma)_{\max} = 0.0072$
$R = 0.0370$	$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$
$wR = 0.0424$	$\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$
$S = 1.913$	Extinction correction: none
1095 reflections	Atomic scattering factors
100 parameters	from <i>International Tables</i>
H-atom parameters not refined	for <i>X-ray Crystallography</i>
$w = 4F^2/\sigma^2(F^2)$	(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

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

	$x$	$y$	$z$	$U_{\text{eq}}$
O(1)	0.1941 (2)	-0.1448 (2)	0.68535 (6)	0.0435 (3)
O(2)	-0.0589 (2)	0.0916 (2)	0.68345 (7)	0.0554 (4)
O(3)	-0.0157 (2)	0.0357 (2)	0.60615 (6)	0.0474 (4)
N(1)	-0.2140 (4)	-0.4225 (3)	0.5738 (1)	0.0811 (7)
C(1)	-0.0464 (4)	-0.3452 (3)	0.5674 (1)	0.0540 (6)
C(2)	0.1650 (3)	-0.2412 (3)	0.5580 (1)	0.0483 (5)
C(3)	0.1907 (3)	-0.0758 (2)	0.61182 (9)	0.0382 (4)
C(4)	0.3927 (3)	0.0512 (3)	0.6000 (1)	0.0509 (5)
C(5)	0.3898 (3)	0.2202 (3)	0.6529 (1)	0.0580 (6)
C(6)	0.3273 (3)	0.1604 (3)	0.7298 (1)	0.0537 (6)
C(7)	0.1349 (3)	0.0195 (3)	0.72563 (10)	0.0467 (5)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O(1)—C(3)	1.408 (2)	C(1)—C(2)	1.459 (3)
O(1)—C(7)	1.414 (2)	C(2)—C(3)	1.515 (2)
O(2)—O(3)	1.478 (2)	C(3)—C(4)	1.509 (2)
O(2)—C(7)	1.435 (2)	C(4)—C(5)	1.522 (3)
O(3)—C(3)	1.445 (2)	C(5)—C(6)	1.508 (3)
N(1)—C(1)	1.136 (2)	C(6)—C(7)	1.503 (3)
C(3)—O(1)—C(7)	102.0 (1)	O(3)—C(3)—C(4)	109.6 (1)
O(3)—O(2)—C(7)	103.8 (1)	C(2)—C(3)—C(4)	114.9 (1)
O(2)—O(3)—C(3)	104.8 (1)	C(3)—C(4)—C(5)	110.0 (1)
N(1)—C(1)—C(2)	178.3 (2)	C(4)—C(5)—C(6)	111.5 (2)
C(1)—C(2)—C(3)	111.9 (1)	C(5)—C(6)—C(7)	110.5 (2)
O(1)—C(3)—O(3)	103.3 (1)	O(1)—C(7)—O(2)	103.0 (1)
O(1)—C(3)—C(2)	109.5 (1)	O(1)—C(7)—C(6)	111.1 (1)
O(1)—C(3)—C(4)	110.9 (1)	O(2)—C(7)—C(6)	112.0 (2)
O(3)—C(3)—C(2)	108.0 (1)		

In the weighting scheme  $\sigma^2(F^2) = S^2(C + R^2B) + (0.04F^2)^2/Lp^2$ , where  $S$  = scan rate,  $C$  = total integrated peak count,  $R$  = ratio of scan to background counting times,  $B$  = total background count and  $Lp$  = Lorentz-polarization factor.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1992a). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992b). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Financial support from the Jacob A. and Frieda M. Hunkele Charitable Trust and the Kresge Foundation is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1178). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Two Schiff Base Ligands Derived from 1,2-Diaminocyclohexane

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## Abstract

The crystal structures of the free ligands 2,2'-(1,2-cyclohexanediyl)bis(nitrilomethylidene)bisphenol, C<sub>20</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>, (I), and 2,2'-(1,2-cyclohexanediyl)bis(nitriloethylidene)bisphenol, C<sub>22</sub>H<sub>26</sub>N<sub>2</sub>O<sub>2</sub>, (II), have been determined. In both molecules the N—O distances are indicative of intramolecular hydrogen bonding. In compound (I), the two aromatic rings are inclined at an angle of 56.5 (1)° and the O···O separation is 6.082 (3) Å; in compound (II) the corresponding values are 83.15 (8)° and 5.544 (5) Å. Thus, it is evident that the methyl groups in (II) have a very significant effect upon the overall conformation.

## Comment

Relatively few crystal structures have been reported for quadridentate Schiff base ligands (Corden, Errington,