a 500 W tungsten halogen lamp at a distance of 10 cm from the reaction vessel. After 4 h, the mixture was concentrated and subjected to column chromatography. Elution with hexane afforded the title compound in 25% yield as a white solid; recrystallization from a chloroform solution of the compound gave colourless crystals with m.p. 412–413 K; literature m.p. 414 K (Kern *et al.*, 1959).

*Crystal data*

$C_{20}H_{18}$
$M_r = 258.36$
Monoclinic
$P2_1/n$
$a = 7.983\,(2)$ Å
$b = 10.908\,(1)$ Å
$c = 9.264\,(1)$ Å
$\beta = 114.51\,(1)^\circ$
$V = 734.0\,(2)$ Å$^3$
$Z = 2$
$D_x = 1.169$ Mg m$^{-3}$
$D_m$ not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 53.1\text{–}55.0^\circ$
$\mu = 0.493$ mm$^{-1}$
$T = 293$ K
Needle
$0.44 \times 0.11 \times 0.11$ mm
Colourless

*Data collection*

Rigaku AFC-6*R* diffractometer
$\omega\text{–}2\theta$ scans
Absorption correction: analytical (De Meulenaer & Tompa, 1965)
$T_{\min} = 0.871$, $T_{\max} = 0.957$
1679 measured reflections
1537 independent reflections

1142 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 75.0^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 13$
$l = -11 \rightarrow 9$
3 standard reflections every 400 reflections intensity decay: −0.45%

*Refinement*

Refinement on $F$
$R = 0.053$
$wR = 0.068$
$S = 2.828$
1142 reflections
91 parameters
H atoms fixed in calculated positions, C—H = 0.97 Å

$w = 1/[\sigma^2(F) + (0.0125F)^2]$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.22$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.17$ e Å$^{-3}$
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SIR*92 (Burla *et al.* 1989). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

The Australian Research Committee is thanked for support.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1034). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). **C54**, 149–151

# 2,2′-Bipyridine 1,1′-Dioxide

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*(Received 4 June 1997; accepted 16 October 1997)*

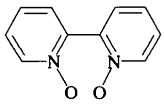
## Abstract

The title molecule, $C_{10}H_8N_2O_2$, lies on a crystallographic twofold axis. The dihedral angle between the planes of the pyridine groups is 67.5° and the non-bonded distance between the O atoms in the *N*-oxide groups is 3.046 (2) Å. Although the skew conformation of the skeleton is almost the same as that of the coordinated form, the dihedral angle is the largest among the corresponding values for the 2,2′-bipyridine 1,1′-dioxide complexes.

## Comment

2,2′-Bipyridine 1,1′-dioxide (bpdo), (I), forms a skew seven-membered chelate ring with a metal ion. Recently, we reported that the tetraammine(bpdo)cobalt(III) complex was resolved into a pair of enantiomers which rapidly racemized in water (Kanno *et al.*, 1997). This result indicates that the skewed bpdo-chelate ring is chiral and changes its conformation easily in solution.

Although a large number of metal complexes with bpdo have been synthesized, the crystal structures of only a few complexes have been reported and there has been no reports on the molecular structure of bpdo. The present study was undertaken in order to determine the molecular structure of an uncoordinated form of bpdo.



(I)

The pyridine ring is planar with a maximum deviation of 0.017 Å, but the O atom deviates by 0.09 Å from the pyridine plane away from the other O atom. The dihedral angle between the two rings is 67.5°, which is the largest value reported in bpdo-chelate complexes; 60.7, 61.3 and 62.3° in $[La(bpdo)_4](ClO_4)_3$ (Al-Karaghouli, Day & Wood, 1978), 53.7° in $[UO_2(NO_3)_2(bpdo)]$ (Alcock & Roberts, 1987), 52.3 (green isomer) and 59.3° (yellow–orange isomer) in $[Cu_2Cl_4(bpdo)_2]$ (Baran *et al.*, 1991), and 57.6 and 63.0° in $[CuCl_2(bpdo)(CH_3OH)]$ (Koman, Baran & Valigura, 1991), where the latter four values are calculated in the present study from the reported coordinates. Chelation of bpdo to a metal ion seems to reduce the dihedral angle.

The central C—C bond distance, 1.476 (2) Å, is shorter than a normal C—C single-bond distance. This shortening, which is the same trend as those observed in bpdo complexes, probably shows that the central C—C bond has a little π-bond character.

There are no unusual short intermolecular distances in the crystal structure.

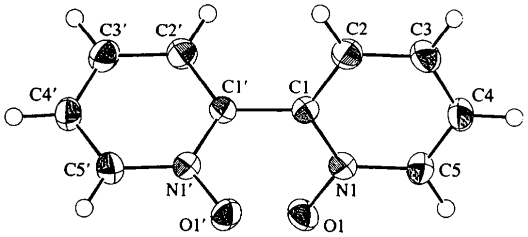


Fig. 1. *ORTEP* drawing (Johnson, 1965) of the whole molecule, half of which is in an asymmetric unit, with the atomic numbering scheme. Displacement ellipsoids of the non-H atoms are shown at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

## Experimental

The title compound was prepared according to the method of Murase (1956). Single crystals of this compound were obtained by sublimation in a vacuum.

*Crystal data*

$C_{10}H_8N_2O_2$
$M_r = 188.19$
Monoclinic
$C2/c$
$a = 12.616$ (4) Å
$b = 5.336$ (2) Å
$c = 12.571$ (4) Å
$\beta = 104.67$ (3)°
$V = 818.7$ (5) Å$^3$
$Z = 4$
$D_x = 1.527$ Mg m$^{-3}$
$D_m = 1.53$ Mg m$^{-3}$
$D_m$ measured by flotation in a mixture of chloroform and 1,2-dibromoethane

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 22 reflections
$\theta = 15.6$–17.4°
$\mu = 0.110$ mm$^{-1}$
$T = 294$ K
Block
0.28 × 0.25 × 0.18 mm
Colourless

*Data collection*

MacScience MXC3 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
1122 measured reflections
870 independent reflections
839 reflections with $I > 1.25\sigma(I)$

$R_{\rm int} = 0.018$
$\theta_{\rm max} = 26.43°$
$h = 0 \rightarrow 16$
$k = -6 \rightarrow 0$
$l = -16 \rightarrow 15$
3 standard reflections every 100 reflections intensity decay: <1%

*Refinement*

Refinement on $F$
$R = 0.056$
$wR = 0.069$
$S = 1.836$
839 reflections
64 parameters
H atoms not refined
$w = 1/[\sigma^2(F) + 0.0005F^2]$

$(\Delta/\sigma)_{\rm max} = 0.0001$
$\Delta\rho_{\rm max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.34$ e Å$^{-3}$
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—N1 | 1.302 (2) | C1—C2 | 1.382 (2) |
| N1—C1 | 1.369 (2) | C2—C3 | 1.383 (2) |
| N1—C5 | 1.369 (2) | C3—C4 | 1.382 (3) |
| C1—C1$^{\rm i}$ | 1.476 (2) | C4—C5 | 1.366 (2) |
| O1—N1—C1 | 120.0 (1) | C1$^{\rm i}$—C1—C2 | 122.2 (2) |
| O1—N1—C5 | 120.5 (2) | C1—C2—C3 | 120.0 (2) |
| C1—N1—C5 | 119.5 (2) | C2—C3—C4 | 118.5 (2) |
| N1—C1—C1$^{\rm i}$ | 117.1 (2) | C3—C4—C5 | 121.1 (2) |
| N1—C1—C2 | 120.5 (2) | N1—C5—C4 | 120.3 (2) |

Symmetry code: (i) $-x, y, \frac{1}{2} - z$.

The space group was finally determined from the Wilson plot. The structure was solved by direct methods and refined by full-matrix least-squares calculations using *CRYSTAN* (Gilmore & Brown, 1988). H atoms were restricted to their parent atoms with C—H distances of 0.96 Å and isotropic displacement parameters of 0.05 Å$^2$.

Data collection: *MXC* (MacScience Co. Ltd, 1989). Cell refinement: *MXC*. Data reduction: *CRYSTAN*. Program(s) used to solve structure: *CRYSTAN*. Program(s) used to refine structure: *CRYSTAN*. Molecular graphics: *ORTEP* (Johnson, 1965) in *CRYSTAN*. Software used to prepare material for publication: *CRYSTAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1357). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). C**54**, 151–152

# *endo*-2,4-Dibromo-3a,4,7,7a-tetrahydro-4,7-methanoindene-1,8-dione 8-Ethylene Acetal

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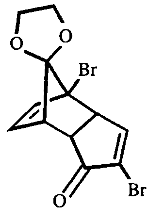
(*Received 28 July 1997; accepted 29 September 1997*)

## Abstract

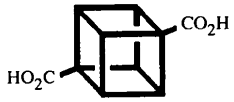
In the title compound, $C_{12}H_{10}Br_2O_3$, the $C_{sp^2}$—Br bond is found to be shorter than the $C_{sp^3}$—Br bond, while the bonds involving the apical C atom (the methano bridge) have distances between those observed for the corresponding bonds in the chloro-substituted compound 1,2,4,5,6,7,8,9-octachloro-10,10-dimethoxytricyclo[5.2.1.0$^{2,6}$]deca-4,8-diene-3-one [Galešić *et al.* (1985). *Acta Cryst.* **C41**, 1662–1664] and those observed for the corresponding bonds in similar compounds without halogen substituents.

## Comment

The title compound, (1), is a key precursor in the synthesis of cubane-1,4-dicarboxylic acid, (2). The preparation of (1) was first described by Eaton & Cole (1964) and later modified by Chapman *et al.* (1970). We report herein the X-ray structure determination of (1).



(1)



(2)

The C4—Br2 distance is significantly shorter than the distance found for the other C—Br bond; a similar situation is observed in 1,2,4,5,6,7,8,9-octachloro-10,10-dimethoxytricyclo[5.2.1.0$^{2,6}$]deca-4,8-diene-3-one (Galešić *et al.*, 1985), where the $C_{sp^2}$—Cl bonds were found to be shorter than the $C_{sp^3}$—Cl bonds. The atoms of the cyclopentene ring are coplanar, with the maximum deviation from the plane being 0.024 (2) Å (C6), while the cyclopentane and oxolane rings have envelope conformations (Altona *et al.*, 1968). The dihedral angle between the cyclopentene ring and the plane defined by atoms C1, C9, C8 and C7 is 59.07 (14)°. The bonds involving the apical C atom, C7—C10 and C1—C10, have lengths between those observed for the corresponding bonds in the above-mentioned Cl-substituted compound (1.573–1.576 Å; Galešić *et al.*, 1985) and those observed for the corresponding bonds in similar compounds without halogen substitutents (1.519–1.547 Å), such as diethyl 4,7-dimethyl-5,10-dioxo-*endo*-tricyclo[5.2.1.0$^{2,6}$]deca-3,8-diene-3,8-dicarboxylate (Smits *et al.*, 1988) and 1-methyl-3-oxotricyclo[5.2.1.0$^{2,6}$]deca-4,8-diene-6-carboxylic acid (Watson *et al.*, 1990). In contrast, the C1—C2 and C6—C7 bond lengths are at the lower end of the range observed for such compounds (1.562–1.622 Å).

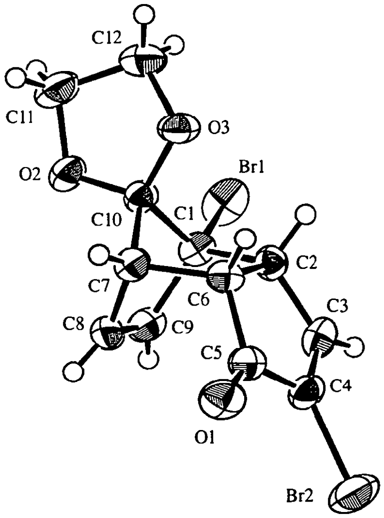


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (1). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been given arbitrary radii (0.1 Å).

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