Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: AB1419). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4,4′-Dimethyl-2,2′-bipyridine 1,1′-Dioxide

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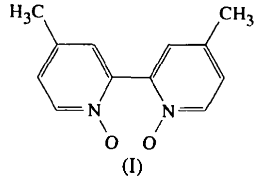
Abstract

There are four molecules in a unit cell of the title compound, $C_{12}H_{12}N_2O_2$, and the structure of only half a molecule has been determined because of its twofold rotational symmetry. The dihedral angle between the two pyridine rings is 61.3° and the non-bonded distance between O atoms in the *N*-oxide groups is 2.96 Å. The skew conformation of the skeleton is almost the same as that of the tetrakis(2,2′-bipyridine 1,1′-dioxide)-lanthanum(III) complex.

Comment

The bidentate ligand 2,2′-bipyridine 1,1′-dioxide (bpdo) forms a skewed seven-membered ring with a metal ion. Since the skew conformation produces a pair of enantiomers, a tris(bpdo) complex has four possible racemic pairs of conformational isomers. The tris-(bpdo)–Cr^{III} complex, however, crystallizes in one of the four isomers and then isomerizes and racemizes on dissolution in water (Kanno & Fujita, 1987). This indicates that the skewed bpdo-chelate ring is flexible and changes its conformation rather easily in solution. The dihedral angle between the pyridine rings, which seems to represent a distortion of the seven-membered ring, is 61.4° in the La^{III} complex (Al-Karaghouli, Day & Wood, 1978) and 53.7° in the U^{IV} complex (Alcock & Roberts, 1987). The present study was undertaken in order to estimate the dihedral angle between the pyridine rings of the bpdo molecule with methyl groups at the 4,4′-positions, (I).



(I)

The title molecule has a twofold rotational axis and the parameters of half of the molecule have been determined (Fig. 1). The pyridine ring is planar within 0.03 Å, but the O atom of the *N*-oxide group is slightly out of the plane (0.13 Å), away from the other O atom

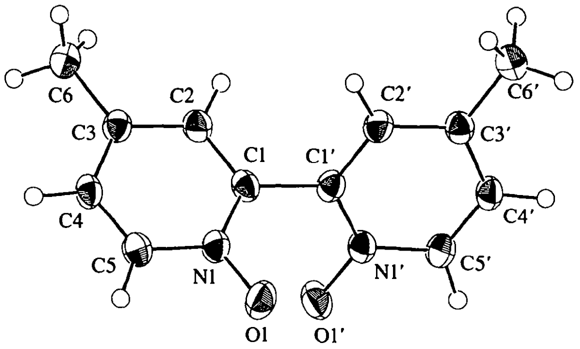


Fig. 1. *ORTEP* drawing (Johnson, 1965) of the whole title molecule, half of which is an asymmetric unit, showing the atomic numbering scheme. Displacement ellipsoids of the non-H atoms are shown at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

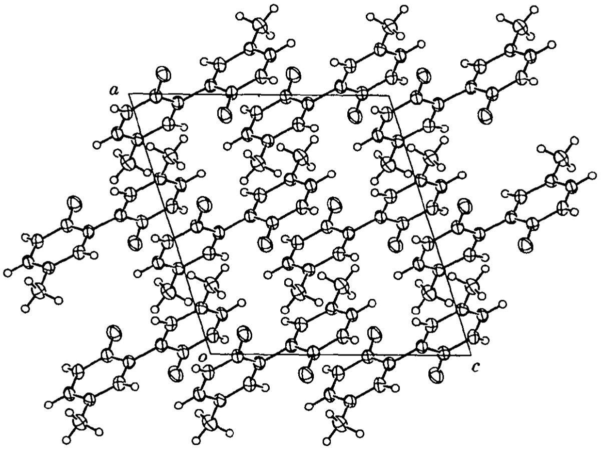


Fig. 2. The crystal structure of (I) viewed along the *b* axis.

(Fig. 2). The dihedral angle between the pyridine rings is 61.3°, in good agreement with the value for the La^{III} complex (Al-Karaghouli, Day & Wood, 1978). The central C—C distance is also similar to that in the La^{III} complex, but the N—O distance is shorter by 0.03 Å. There are no unusual short intermolecular distances.

Experimental

The title compound was prepared according to the method of Kanno, Yamamoto, Murahashi, Utsuno & Fujita (1991) and recrystallized from methanol.

Crystal data

$C_{12}H_{12}N_2O_2$
M_r = 216.24
Monoclinic
$C2/c$
a = 11.232 (4) Å
b = 9.175 (3) Å
c = 10.444 (4) Å
β = 107.09 (3)°
V = 1028.8 (7) Å^3
Z = 4
D_x = 1.396 Mg m^{-3}
D_m = 1.38 Mg m^{-3}
D_m measured by flotation in a mixture of benzene and carbon tetrachloride

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 23 reflections
θ = 14.3–17.5°
μ = 0.097 mm^{-1}
T = 298 K
Needle
0.40 × 0.30 × 0.25 mm
Colourless

Data collection

MAC Science MXC3 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
1328 measured reflections
1087 independent reflections
948 reflections with $I > 1.25\sigma(I)$

R_{int} = 0.027
θ_{max} = 26.43°
$h = -14 \rightarrow 13$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 13$
3 standard reflections every 100 reflections intensity decay: < 2%

Refinement

Refinement on F
R = 0.0572
wR = 0.0585
S = 1.590
948 reflections
73 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.0005F_o^2]$

$(\Delta/\sigma)_{max}$ = 0.0025
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 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	1.301 (2)	C2—C3	1.391 (2)
N1—C1	1.370 (2)	C3—C4	1.387 (2)
N1—C5	1.362 (2)	C3—C6	1.494 (3)
C1—C1^{i}	1.482 (2)	C4—C5	1.361 (2)
C1—C2	1.375 (2)		
O1—N1—C1	120.8 (2)	C1—C2—C3	122.1 (2)
O1—N1—C5	120.4 (2)	C2—C3—C4	115.7 (2)
C1—N1—C5	118.8 (2)	C2- C3—C6	121.4 (2)
N1—C1—C1^{i}	117.3 (2)	C4—C3—C6	122.8 (2)
N1—C1—C2	120.0 (2)	C3—C4—C5	122.1 (2)
C1^{i}—C1—C2	122.6 (2)	N1—C5—C4	121.1 (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 found from the difference Fourier map at the final stage and were restricted to their parent atoms with C—H distances of 0.96 Å and the corresponding equivalent isotropic displacement parameters.

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

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: OH1103). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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meso-2,2,2′,2′-Tetrachloro-1,1′-diphenyl-1,1′-bicyclopropyl and *meso*-2,2,2′,2′-Tetrabromo-1,1′-diphenyl-1,1′-bicyclopropyl

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Abstract

Molecules of *meso*-2,2,2′,2′-tetrachloro-1,1′-diphenyl-1,1′-bicyclopropyl, $C_{18}H_{14}Cl_4$, (1), and *meso*-2,2,2′,2′-tetrabromo-1,1′-diphenyl-1,1′-bicyclopropyl, $C_{18}H_{14}Br_4$, (2), adopt *gauche* conformations. Compound (1) produced two types of crystals upon recrystallization. The phenyl rings of the two forms of compound (1), as well as those of compound (2), are in a nearly perpendicular orientation. All three structures have unsymmetrical