$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 0.2648P]$ where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—N2	1.215 (3)	C3—C4	1.366 (3)
O2—N2	1.222 (3)	C4—C5	1.365 (3)
O3—C9	1.225 (2)	C5—C6	1.382 (3)
N1—C7	1.358 (2)	C7—C8	1.347 (3)
N1—C1	1.389 (2)	C7—C11	1.503 (2)
N2—C4	1.468 (3)	C8—C9	1.432 (3)
C1—C6	1.384 (3)	C9—C10	1.504 (3)
C1—C2	1.394 (3)	C10—C11	1.518 (3)
C2—C3	1.372 (3)	N1—H1	0.87 (2)
C7—N1—C1	131.6 (2)	C8—C7—N1	132.2 (2)
O1—N2—O2	123.7 (2)	C7—C8—C9	109.6 (2)
C6—C1—N1	125.4 (2)	O3—C9—C8	126.5 (2)
N1—C1—C2	116.3 (2)		

The structure was solved using the structure solution program *SHELXS*86 (Sheldrick, 1985). The remaining atoms were located in succeeding difference Fourier syntheses. All H atoms except H1, for which parameters were refined isotropically, were included in the refinement.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) and/or *PLUTON* (Spek, 1991). Software used to prepare material for publication: *ORTEP*II and/or *PLUTON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1319). Services for accessing these data are described at the back of the journal.

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4,4′-Dinitro-2,2′-bipyridine

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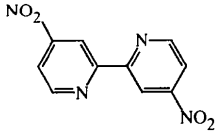
Abstract

The title compound, $C_{10}H_6N_4O_4$, has crystallographic C_i symmetry and is almost planar. Analysis of its crystal packing reveals molecular tapes formed by C—H···O hydrogen bonds.

Comment

The structure determination of the title compound, (I), has been undertaken to check the outcome of the chemical synthesis (Maerker & Case, 1958).



(I)

The title molecule sits on a centre of symmetry. It is almost planar. The NO_2 group is slightly displaced from the best plane through N1—C2—C3—C4—C5—C6 [N2 0.062 (2), O1 0.107 (2) and O2 0.059 (2) Å]. The planes of the two aromatic rings are slightly offset, as indicated by the torsion angle C3—C2—C2′—N1′ of 1.1 (3)°. The small angle C2—N1—C6 of 117.8 (1)° may be explained by the fact that a lone pair at an N atom needs more space than an N—C bonding electron pair (Gillespie & Hargittai, 1991). The C2—C2′ distance is about 0.1 Å longer than the C—C bonds in the ring and has strong single-bond character (Watson, Brammer, Orpen & Taylor, 1992).

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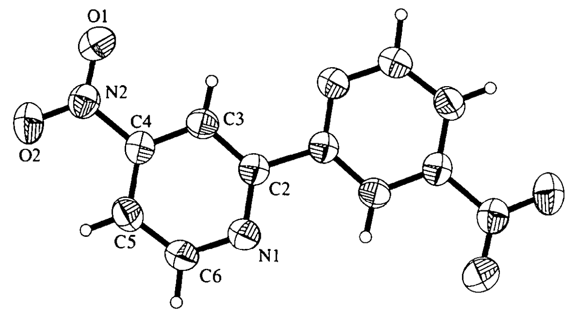

Fig. 1. The molecular structure of the title compound showing the atomic numbering and 50% probability displacement ellipsoids.

The crystal packing may be described in terms of molecular tapes related by the 2_1 axes and stacked along the a axes by translation. Tapes run along (110) in one stack and $(1\bar{1}0)$ in the other. The distance between the best molecular planes of the two closest molecules in adjacent tapes is 3.6 Å, to be compared with the van der Waals radius of a benzene ring which is 1.80 Å (Kitaigorodsky, 1973). Adjacent molecules in a tape are related by a centre of symmetry and are bridged by two N2—O1···H6—C6 hydrogen bonds of 2.57 (2) Å.

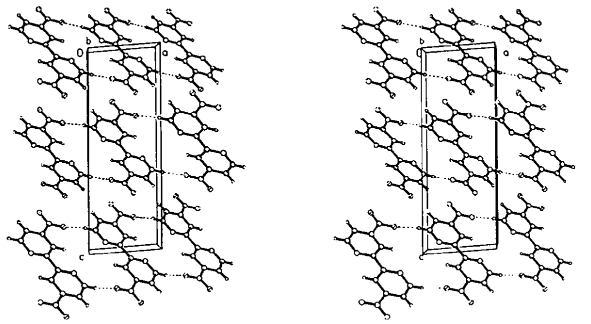

Fig. 2. Stereoview of the packing of the title compound.

Experimental

Pale yellow–orange crystals of the title compound, prepared by reduction of 4,4′-dinitro-2,2′-bipyridyl N,N'-dioxide with PCl_3, were grown from hot dichloromethane solution; m.p. 468 K, literature m.p. 468–470 K (Maerker & Case, 1958).

Crystal data

$C_{10}H_6N_4O_4$
$M_r = 246.19$
Monoclinic
$P2_1/c$
$a = 5.8295\ (7)$ Å
$b = 5.5407\ (7)$ Å
$c = 16.345\ (2)$ Å
$\beta = 94.607\ (2)°$
$V = 526.24\ (11)$ Å^3
$Z = 2$
$D_x = 1.554$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 1497 reflections
$\theta = 2.50–23.24°$
$\mu = 0.124$ mm^{-1}
$T = 295\ (2)$ K
Plate
$0.50 \times 0.30 \times 0.15$ mm
Pale yellow–orange

Data collection

Siemens SMART CCD system
ω scans, 1271 frames, 0.30°, 10 s, detector distance 5.5 cm, detector angle 23.0°
Absorption correction: none

2446 measured reflections
975 independent reflections
821 reflections with $F > 4\sigma(F)$
$R_{\rm int} = 0.045$
$\theta_{\rm max} = 26.55°$
$h = -6 \rightarrow 6$
$k = -6 \rightarrow 6$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.089$
$S = 2.688$
973 reflections
95 parameters
All H atoms refined
$w = 1/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\rm max} = 0.025$

$\Delta\rho_{\rm max} = 0.206$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.130$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.04 (1)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

N1—C6	1.333 (2)	C4—C5	1.368 (2)
N1—C2	1.337 (2)	C4—N2	1.479 (2)
C2—C3	1.391 (2)	C5—C6	1.383 (2)
C2—C2$^{\rm i}$	1.492 (2)	N2—O1	1.216 (2)
C3—C4	1.372 (2)	N2—O2	1.221 (2)
C6—N1—C2	117.8 (1)	C3—C4—N2	118.7 (1)
N1—C2—C3	122.8 (1)	C4—C5—C6	116.7 (1)
N1—C2—C2$^{\rm i}$	116.6 (2)	N1—C6—C5	123.8 (1)
C3—C2—C2$^{\rm i}$	120.6 (1)	O1—N2—O2	124.4 (1)
C4—C3—C2	117.1 (1)	O1—N2—C4	117.7 (1)
C5—C4—C3	121.7 (1)	O2—N2—C4	117.9 (1)
C5—C4—N2	119.5 (1)		
C3—C2—C2$^{\rm i}$—N1$^{\rm i}$	1.1 (3)		

Symmetry code: (i) $1 - x, -y, -z$.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *SHELXTL* (Siemens, 1996). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1243). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1721–1723

(3a*R**,4*S**,5*S**,7a*R**)-4-Nitrobenzoic Acid 5-Methyl-1,3,3a,4,5,7a-hexahydroisobenzofuran-4-yl Ester

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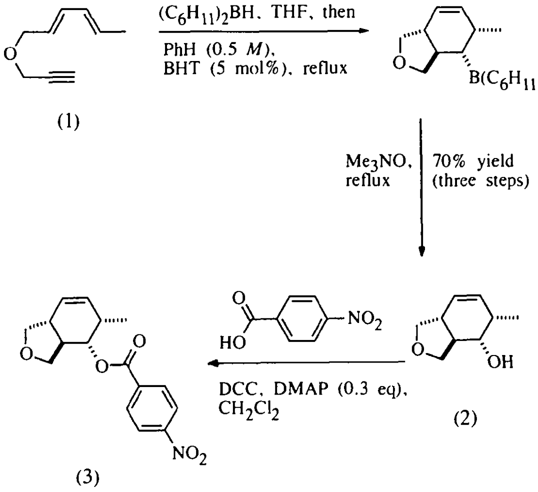
Abstract

The crystal structure determination of the title compound, $C_{16}H_{17}NO_5$, establishes the relative stereochemistry. The molecule contains a furanyl ring with a twist conformation fused to an unsaturated six-membered ring with a twist-chair conformation. There are weak intermolecular C—H···O interactions, with distances C···O$(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$ 3.254 (2) and H···O$(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$ 2.41 Å.

Comment

Recent studies have identified alkenylboranes as reactive dienophiles in Diels–Alder reactions (Matteson, 1995). The potential to control the relative stereochemistry of at least three new stereocentres in the intramolecular variant, and the synthetic utility of the C—B bond in the cycloadducts have prompted us to examine this reaction. During the course of our studies, the first example of this strategy to form bicyclo[4.4.0]decenes was reported (Singleton & Lee, 1995). In contrast, our research has focused on the formation of hydrindene-type structures, using a 'one-pot' procedure (Batey, Lin, Hayhoe & Wong, 1997). Thus, an alkenylborane, generated *in situ* by selective hydroboration of a dienyne, undergoes an intramolecular Diels–Alder reaction, after which the C—B bond in the cycloadduct is transformed. In an intramolecular reaction, two modes of cycloaddition (*endo*/*exo*) are possible. In the case of the dienyne (1) (see reaction scheme below), a single diastereomer, (2), formed as a viscous oil, was derived as the crystalline *p*-nitrobenzoate ester, (3), in order to determine the relative stereochemistry about the six-membered ring.



DMAP = dimethylaminopyridine
DCC = dicylohexylcarbodiimide
BHT = butylated hydroxytoluene

The crystal structure determination establishes the diastereomer as the product of *endo* addition in the intramolecular Diels–Alder reaction. A search of the Cambridge Structural Database (Allen *et al.*, 1979) revealed that there are only three other structures (refcodes: DAHDUL, FUMZIW and JEWRUY) containing similar *trans*-fused six- and five-membered rings as in (3), but (3) is the first compound reported that contains an unsubstituted furanyl group fused to a cyclohexene ring system.

The five-membered furanyl ring is in a twist conformation. C6 is 0.412 (3) Å above and C7 is 0.301 (3) Å below the plane formed by the three atoms C8, C9 and O3. The twist conformation of the furanyl group in (3) is consistent with the conformation of the molecule of tetrahydrofuran that was determined at 103 and 148 K by Luger & Buschmann (1983). The furanyl group in (3) has similar bond lengths and angles to those in tetrahydrofuran; the only exceptions are the magnitudes of the angles C7—C8—O3 and C6—C9—O3 in (3), which are 104.63 (9) and 104.32 (9)°, respectively, compared with 107.4 (4)° at 103 K and 106.7 (4)° at 148 K for tetrahydrofuran (the tetrahydrofuran molecule has crystallographic twofold symmetry).

The six-membered ring of the fused ring system in (3) has a twist-chair conformation. Atoms C3, C4, C5 and C6 form a least-squares plane [with maximum deviation of 0.008 (1) Å for C4] and C7 is 0.644 (2) Å below the plane, while C2 is 0.139 (2) Å above the plane. The six- and five-membered rings are *trans*-fused along the C6—C7 bond (see Fig. 1).