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Acta Cryst. (1982). **B38**, 2286–2288

Structure of 2,2',6,6'-Tetranitro-4,4'-isopropylidenediphenol

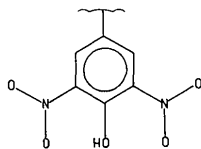
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(Received 24 August 1981; accepted 16 March 1982)

Abstract. $C_{15}H_{12}N_4O_{10}$, $M_r = 408.28$, monoclinic, $C2/c$, $a = 17.202$ (1), $b = 9.477$ (1), $c = 11.263$ (1) Å, $\beta = 107.0$ (1)°, $Z = 4$, $D_c = 1.544$, $D_m = 1.535$ g cm⁻³. The structure was solved by direct methods and refined by full-matrix anisotropic least squares to a final R of 0.068 for 1590 reflections. The molecule has C_2 symmetry. The dihedral angle between the two phenyl rings is 63.9 (5)°. The -OH group forms an ordered intramolecular hydrogen bond with one of the two neighboring nitro groups. The other nitro group is rotated through 46.5 (3)° from coplanarity with the phenyl ring.

Introduction. In the title compound each hydroxy group lies between two nitro groups.



The present structure analysis was undertaken to study the intramolecular hydrogen bonds in this symmetrical molecule.

The compound was synthesized according to du Pont de Nemours & Co. (1937). Yellow crystals were crystallized from benzene solution. Intensity data were collected at room temperature using a θ - 2θ scan

technique on a CAD-4F four-circle automatic diffractometer with graphite-monochromatized Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å). No time-decay correction was made, since three monitoring reflections were measured periodically which showed only slight random fluctuations. 1622 independent reflections ($2\theta \leq 140^\circ$) were corrected for Lorentz and polarization effects, but not for absorption ($\mu = 11.69$ cm⁻¹). 1590 reflections had $|F_o| > 2\sigma|F_o|$.

The structure was solved by *MULTAN* (Germain, Main & Woolfson, 1971). The E map revealed the molecular structure and confirmed the space group $C2/c$. The non-hydrogen-atom coordinates with isotropic thermal parameters were refined by full-matrix least-squares techniques. The phenyl H atoms were then inserted into chemically reasonable positions and the methyl and hydroxyl H atoms were located on difference maps. The final cycle of the refinement with anisotropic thermal parameters for all non-hydrogen atoms, except for the central C(7), yielded R and R_w values of 0.068 and 0.03, respectively, where $R = \sum |F_o| - |F_c| / \sum |F_o|$ and $R_w = [\sum w(|F_o| - |F_c|)^2 / \sum wF_o^2]^{1/2}$. * The weighting scheme of Stout &

* Lists of structure factors, anisotropic thermal parameters and unrefined H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36800 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and thermal parameters ($\times 10^4$, $\times 10^3$ for H), with their estimated standard deviations

The equivalent thermal parameters (\AA^2) are of the form $U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$. All hydrogen atoms were kept fixed during the refinement and assigned the same isotropic temperature factors as their attached heavy atoms.

	x	y	z	U_{eq}/U_{iso}		x	y	z	U_{eq}/U_{iso}
C(1)	9239 (1)	2411 (2)	2030 (2)	372 (11)	C(2)	9260 (1)	1202 (2)	1362 (2)	457 (12)
C(3)	8574 (1)	376 (3)	894 (2)	484 (12)	C(4)	7821 (1)	708 (3)	1050 (2)	447 (12)
C(5)	7816 (1)	1950 (2)	1721 (2)	429 (11)	C(6)	8494 (1)	2778 (2)	2195 (2)	399 (11)
C(7)	10000	3335 (3)	2500	351 (7)	C(8)	9962 (1)	4263 (3)	3600 (3)	533 (14)
N(1)	8671 (1)	-912 (2)	239 (2)	685 (13)	N(2)	7063 (1)	2396 (3)	1975 (2)	543 (12)
O(1)	7131 (1)	-23 (2)	604 (2)	595 (11)	O(2)	9332 (2)	-1215 (3)	158 (3)	1209 (19)
O(3)	8070 (1)	-1644 (2)	-219 (2)	858 (14)	O(4)	6881 (1)	3641 (2)	1831 (2)	763 (13)
O(5)	6678 (1)	1520 (3)	2355 (3)	1046 (17)	H(C2)	975	92	123	32
H(C6)	851	349	268	30	H(C81)	988	369	429	39
H(C82)	961	480	341	39	H(C83)	1041	471	391	39
H(O1)	720	-70	10	42					

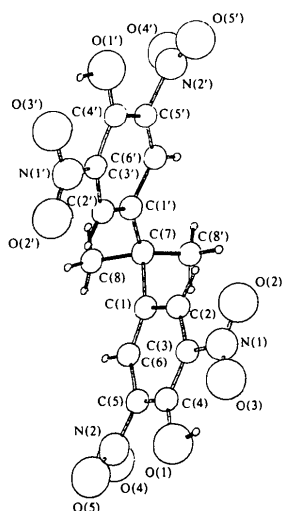
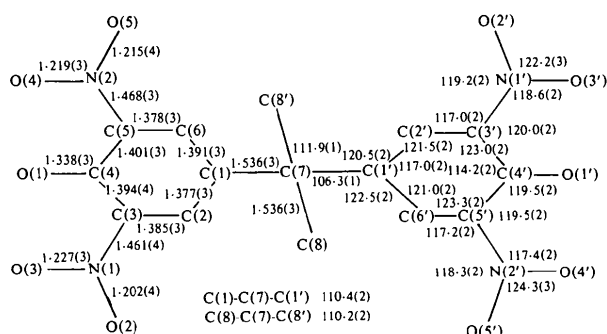


Fig. 1. A perspective view of a molecule of the title compound.

Fig. 2. Bond lengths (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses.

Jensen (1968) was used. All parameters for H atoms were included in the calculation but not refined. The scattering factors used were all taken from *International Tables for X-ray Crystallography* (1962).

The final atomic coordinates are listed in Table 1. A perspective view of the molecule is given in Fig. 1. Selected bond lengths and angles are given in Fig. 2.

Discussion. The molecule has crystallographic C_2 symmetry with its central C atom C(7) on the twofold axis. The angle between the two phenyl-ring planes is $63.9(5)^\circ$. The phenyl ring and its attached functional groups, except for one of the nitro groups, are essentially planar. The displacements of various atoms from the best plane of the phenyl ring defined by the equation $0.0310x + 0.4777y - 0.8780z + 0.0339 = 0$ are: C(1) 0.005, C(2) 0.004, C(3) -0.001, C(4) -0.002, C(5) 0.001, C(6) 0.003, C(7) 0.050, N(1) -0.039, N(2) -0.033, O(1) 0.019, O(2) -0.096, O(3) -0.005, O(4) 0.717, O(5) -0.834 \AA , where x, y, z in the equation are fractional atomic coordinates and refer to C(1), C(2), C(3), C(4), C(5) and C(6). (E.s.d.'s for displacements are $\sim 0.001 \text{\AA}$.)

The N(2) nitro group is rotated through $46.5(3)^\circ$ from coplanarity with the ring. This rotation takes O(4)

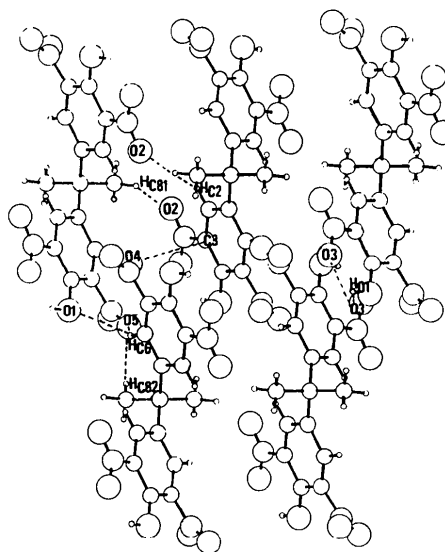
Fig. 3. The molecular packing arrangement viewed along b . The short contacts ($< 3.0 \text{\AA}$) are indicated by broken lines, and the atoms involved are labelled.

Table 2. Short intermolecular contacts (Å) (H...H contacts are excluded)

Second atom at $\frac{1}{2} - x, \frac{1}{2} - y, -z$		Second atom at $x, -y, \frac{1}{2} + z$	
C(3)...O(4)	2.08 (1)	C(5)...O(3)	3.36 (1)
C(3)...N(2)	3.75 (1)	C(6)...O(2)	3.55 (1)
C(4)...C(5)	3.72 (1)	C(6)...O(3)	3.38 (1)
N(1)...O(4)	3.11 (1)	C(6)...N(1)	3.79 (1)
C(2)...O(4)	3.49 (1)	N(2)...O(3)	3.21 (1)
C(4)...C(6)	3.96 (1)		
C(5)...C(5)	3.85 (1)	Second atom at $2 - x, -y, -z$	
		C(2)...O(2)	3.35 (1)
Second atom at $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$		O(2)...H(C2)	2.53 (2)
C(4)...O(4)	3.01 (1)	Second atom at $2 - x, 1 - y, 1 - z$	
O(1)...H(C6)	2.86 (2)	C(8)...C(8)	3.42 (1)
O(5)...H(C6)	2.89 (2)		
O(5)...H(C82)	2.68 (2)	Second atom at $\frac{1}{2} - x, -\frac{1}{2} - y, -z$	
C(3)...O(4)	3.33 (1)	O(1)...O(3)	3.19 (1)
C(5)...O(4)	3.50 (1)	O(3)...O(3)	2.70 (1)
O(1)...O(4)	3.16 (1)	O(3)...H(O1)	2.57 (2)
Second atom at $x, -y, -\frac{1}{2} + z$		Second atom at $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$	
O(3)...O(5)	3.06 (1)	C(8)...O(5)	3.45 (1)
O(2)...H(C81)	2.81 (1)		

and O(5) away from close contact with the —OH group, resulting in an ordered intramolecular hydrogen bond between the —OH group and O(3) of the nitro group of N(1). The O(1)...O(3) and H(O1)...O(3) distances are 2.59 (1) and 1.87 (1) Å, respectively,

and the O(1)—H(O1)...O(3) angle is 137.2 (1)°. A similar feature has been observed in the structure of the picric acid—2-amino-1-bromonaphthalene complex (Carstensen-Oeser, Götlicher & Habermehl, 1968). Picric acid also has one hydroxy group between two nitro groups. The dihedral angles of the two nitro groups adjacent to the hydroxy group are 16.5 (4) and 3.4 (4)° with respect to the phenyl-ring plane. The greater rotation of the N(2) nitro group in the present molecule is probably due to different intermolecular interactions in the two compounds.

The crystal packing, shown in Fig. 3, is through short C...O, C—H...O, O...O and hydrophobic contacts. The C(3)...O(4)($\frac{1}{2} - x, \frac{1}{2} - y, -z$) distance is only 2.08 (1) Å. The intermolecular contacts are listed in Table 2.

The authors thank Mr D. J. Hung for supplying the crystals, and Professor T. H. Hseu for helpful discussions.

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Acta Cryst. (1982). **B38**, 2288–2290

trans-9-Ethyl-2,4-dimethylthioxanthene 10-Oxide

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(Received 12 January 1982; accepted 16 March 1982)

Abstract. C₁₇H₁₈OS, triclinic, $P\bar{1}$, $Z = 2$, $M_r = 270.40$, $a = 8.019$ (1), $b = 8.615$ (2), $c = 11.144$ (3) Å, $\alpha = 104.09$ (2), $\beta = 77.30$ (2), $\gamma = 103.75$ (2)°, $V = 714.5$ (3) Å³, $D_x = 1.257$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu(\text{Cu } K\alpha) = 1.844$ mm⁻¹; final $R = 0.049$ for 2121 observed reflections. The 9-ethyl group is in the 'boat-axial' and the 10-oxide group in the 'boat-equatorial' conformation with respect to the central ring of the thioxanthene ring system.

Introduction. 9-Ethyl-2,4-dimethylthioxanthene 10-oxide can exist in the form of *cis* and *trans* isomers.

The crystal structure of the *cis* isomer has been determined (Chu, Napoleone, Massah & Ternay, 1981), and the preparation and NMR data of the title compound have been reported. In the *cis* isomer, the 9-ethyl and 10-oxide groups are both in the 'boat-axial' conformation. The crystal structure of the *trans* isomer is reported in this paper, and its conformation is compared with that of the *cis* isomer.

Single crystals of the title compound (I) were obtained through the courtesy of Dr Andrew L. Ternay Jr of the Chemistry Department of the University of Texas at Arlington. The crystals are transparent