Instrumental Achievements

Crystal Structure of 3-(*o*-Hydroxyphenyl)-1-methyltriazene-1-oxide

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The crystal and molecular structure of the title compound was investigated in order to determine the conformation and crystal packing and also to confirm its stereochemistry. Bond lengths and angles are in agreement with some related structures.[1-3] There are two independent molecules in the asymmetric unit, denoted by A and B, respectively. Perspective drawings of the two molecules are shown in Fig. 2. Both molecules are closely similar as regards bond lengths and angles. The phenyl rings are planar to within the limits of experimental error and show normal geometries, 1.382(4) Å and 120.0(3)° for the mean C(sp^2)–C(sp^2) bond distance and the mean internal angle. The double bond is located on the N2–N3 rather than N1–N2. Both bond lengths N3–O2 [1.295(3) and 1.287(3)] are between a single and double bond, N–O. The four atoms of the triazene *N*-oxide group are also planar within 0.004(2) Å and 0.008(2) Å for A and B, respectively. The dihedral angles between the triazene *N*-oxide group and phenyl rings for A and B molecules are 9.8(2)° and 6.5(1)°. Both molecules are stabilized internally by two intramolecular

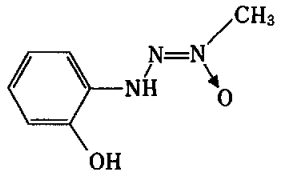

Fig. 1 Chemical structure.

Table 1 Crystal and experimental data

Formula: $C_7H_9N_3O_2$	
Formula weight=167.17	
Crystal system: triclinic	
Space group: $P\bar{1}$	$Z=4$
a=10.501(2) Å	
b=11.329(3) Å	
c=7.314(2) Å	
α=105.14(2)°	
β=104.56(2)°	
γ=91.92(2)°	
V=808.3(3) Å^3	
D_x=1.374 g/cm^3	
μ(Cu K$_\alpha$)=0.872 mm^{-1}	
T=293 K	
colorless	
F(0 0 0)=352	
0.25×0.20×0.15 mm	
Radiation=Cu K$_\alpha$	
R=0.055 (on F)	
Rw=0.200 (on I)	
No. of reflections used=1910	
No. of parameters=298	
Goodness-of-fit=1.13	
Measurement: Nicolet P3/F	
Program system: SHELXL-Plus	
Structure determination: direct methods (SHELXS-86)	
Refinement: full matrix (SHELXL-93)	

Table 2 Final coordinates and equivalent temperature factors (Å^2)

Atom	x	y	z	U_{eq}
C1A	0.5221(3)	0.5395(2)	0.2732(4)	0.0552(7)
C2A	0.6027(3)	0.6500(2)	0.3402(4)	0.0582(7)
C3A	0.7343(3)	0.6505(3)	0.3369(5)	0.0676(8)
C4A	0.7856(3)	0.5423(3)	0.2707(5)	0.0705(8)
C5A	0.7051(3)	0.4324(3)	0.2072(5)	0.0719(9)
C6A	0.5736(3)	0.4304(3)	0.2049(4)	0.0651(8)
C7A	0.0865(4)	0.3677(3)	0.1624(6)	0.0797(10)
N1A	0.3897(2)	0.5467(2)	0.2741(3)	0.0598(7)
N2A	0.3062(2)	0.4457(2)	0.2223(3)	0.0606(7)
N3A	0.1888(2)	0.4703(2)	0.2164(3)	0.0599(7)
O1A	0.5455(2)	0.7537(2)	0.4056(4)	0.0783(7)
O2A	0.1554(2)	0.5813(2)	0.2536(3)	0.0700(6)
C1B	0.8807(3)	0.0480(2)	0.2797(4)	0.0564(7)
C2B	0.8340(3)	0.1545(2)	0.3705(4)	0.0565(7)
C3B	0.7004(3)	0.1554(3)	0.3588(4)	0.0638(8)
C4B	0.6149(3)	0.0507(3)	0.2618(4)	0.0703(8)
C5B	0.6613(3)	-0.0553(3)	0.1738(5)	0.0765(9)
C6B	0.7938(3)	-0.0573(3)	0.1825(5)	0.0684(8)
C7B	1.2662(4)	-0.1243(3)	0.1717(5)	0.0682(8)
N1B	1.0161(3)	0.0537(2)	0.2917(4)	0.0632(7)
N2B	1.0704(2)	-0.0463(2)	0.2204(3)	0.0600(7)
N3B	1.1937(2)	-0.0226(2)	0.2435(3)	0.0587(7)
O1B	0.9253(2)	0.2554(2)	0.4652(3)	0.0712(7)
O2B	1.2539(2)	0.0868(2)	0.3193(3)	0.0784(7)

$U_{eq}=(U_{11}\times U_{22}\times U_{33})^{1/3}$.

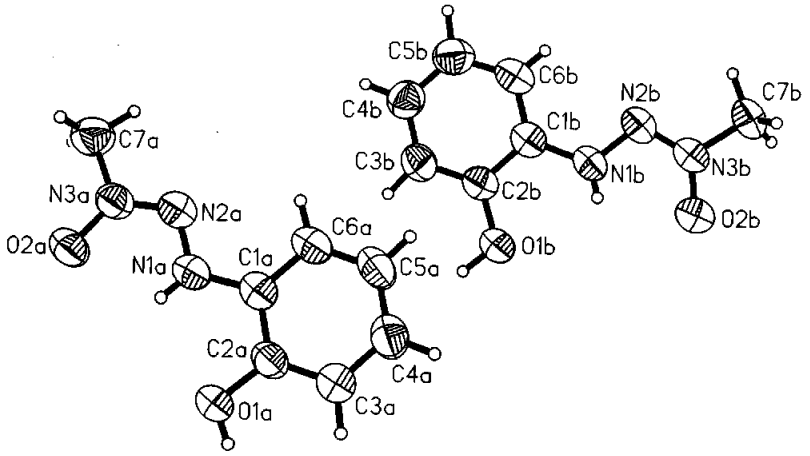

Fig. 2 The molecular structure of the title compound with the atom labeling. Thermal ellipsoids are drawn at the 50% probability level.

Table 3 Bond distances (Å), angles (°) and geometry of hydrogen bonds

	Molecules - A	Molecule - B
C1 - C2	1.387(4)	1.387(4)
C1 - C6	1.389(4)	1.385(4)
C1 - N1	1.397(4)	1.401(4)
C2 - O1	1.366(3)	1.374(3)
C2 - C3	1.389(4)	1.384(4)
C3 - C4	1.375(4)	1.373(4)
C4 - C5	1.382(5)	1.374(5)
C5 - C6	1.376(5)	1.378(5)
C7 - N3	1.458(4)	1.459(4)
N1 - N2	1.327(3)	1.329(3)
N2 - N3	1.266(3)	1.272(3)
N3 - O2	1.295(3)	1.287(3)
C2 - C1 - C6	119.9(3)	119.6(3)
C2 - C1 - N1	116.3(2)	117.1(2)
C6 - C1 - N1	123.8(3)	123.3(3)
C1 - C2 - C3	119.5(3)	119.7(3)
C1 - C2 - O1	116.8(2)	116.7(2)
C3 - C2 - O1	123.7(3)	123.6(2)
C2 - C3 - C4	120.5(3)	120.3(3)
C3 - C4 - C5	119.6(3)	120.1(3)
C4 - C5 - C6	120.7(3)	120.3(3)
C1 - C6 - C5	119.7(3)	120.0(3)
C1 - N1 - N2	120.9(2)	120.8(3)
N1 - N2 - N3	111.9(2)	111.6(2)
N2 - N3 - O2	123.4(2)	122.7(2)
N2 - N3 - C7	117.8(3)	117.5(2)
O2 - N3 - C7	118.9(3)	119.7(2)

D-H...A	D-H	H...A	D...A	D-H...A	symm
N1A-H1NA...O1A	0.79(4)	2.32(4)	2.622(3)	104(3)	x,y,z
N1A-H1NA...O2A	0.79(4)	2.10(4)	2.478(3)	110(4)	x,y,z
O1A-H1OA...O2B	0.83(4)	1.95(4)	2.732(3)	158(4)	-x+2,-y+1,-z+1
N1B-H1NB...O1B	0.75(4)	2.36(5)	2.642(3)	104(4)	x,y,z
N1B-H1NB...O2B	0.75(4)	2.09(5)	2.462(3)	111(4)	x,y,z
O1B-H1OB...O2A	0.93(4)	1.81(4)	2.714(3)	162(3)	-x+1,-y+1,-z+1

N–H···O interactions. The molecular packing is shown in Fig. 3. The arrangement of molecules in the crystal appears to be governed by one intermolecular hydrogen bond, O–H···O. Details of hydrogen bonding scheme is given in Table 3. The H-atoms were located from the difference-Fourier map and refined isotropic temperature factors.

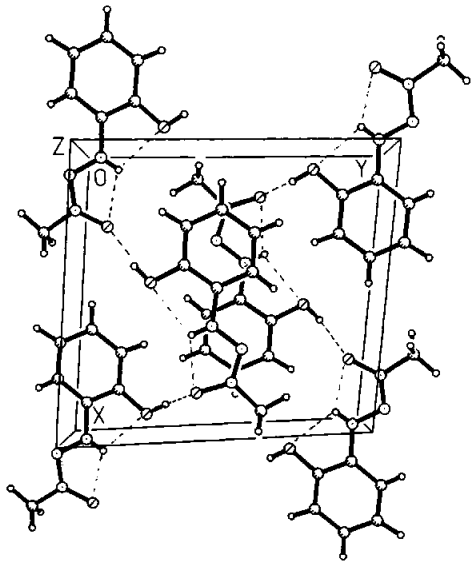

Fig. 3 A unit cell drawing of the packing arrangement and dashed lines indicate O–H···O hydrogen bonds and N–H···O interactions.

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