Instrumental Achievements

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

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The crystal and molecular structure of the title compound were investigated in order to determine unambiguously its tautomeric form as exhibited by these 1-aryl-3-methyltriazenes compounds: ArN=NNHR⟷ ArNHN=NR and its conformation in the solid state. The molecular structure of the title compound is shown in Fig. 2. This shows that the molecules in the crystal adopt the *N*-oxide tautomeric form, ArNHN=NR in agreement with the spectroscopic evidence and the previously reported crystal structures of similar compounds: 3-(4-carbamoylphenyl)-1-methyltriazene-1-oxide;[1] several 3-aryl-1-aryltriazene-1-oxides[2], 3-(4-ethoxy-

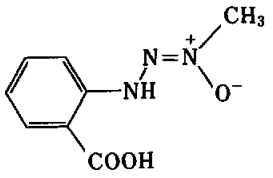

Fig. 1 Chemical structure.

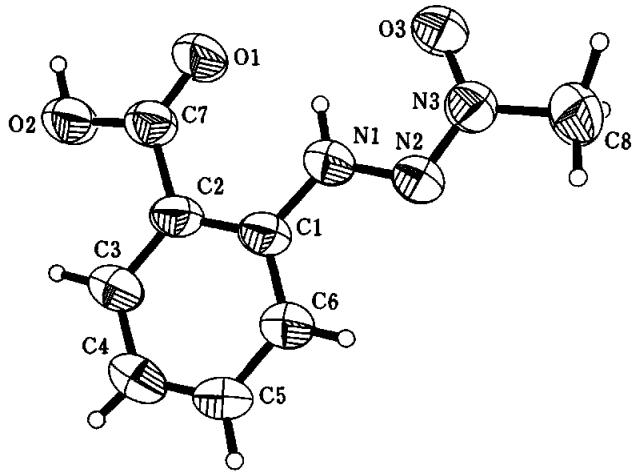

Fig. 2 The molecular structure of the title compound with the atom labelling. Thermal ellipsoids are drawn at the 50% probability level.

Table 1 Crystal and experimental data

Formula:	$C_8H_9N_3O_3$
Formula weight=	195.18
Crystal system:	monoclinic
Space group:	$P2_1/c$ $Z=4$
a=	6.700(3) Å
b=	8.388(2) Å
c=	16.748(6) Å
β=	97.00(3)°
V=	934.2(6) Å^3
D_x=	1.388 g/cm^3
μ(Cu K$_\alpha$)=	0.923 mm^{-1}
T=	293 K
colorless	
F(0 0 0)=	408
0.20×0.23×0.25 mm	
Radiation=	Cu K$_\alpha$
R=	0.054 (on F)
Rw=	0.095 (on I)
No. of reflections used=	1169
No. of parameters=	163
Goodness-of-fit=	1.37
Measurement:	Nicolet P3/F
Program system:	SHELXL-Plus
Structure determination:	direct methods (SHELXS-86)
Refinement:	full matrix (SHELXL-93)

Table 2 Final atomic coodinates and equivalent isotropic temperature factors (Å^2)[a]

Atom	x	y	z	U_{eq}
C1	-0.0566(5)	0.2548(4)	0.9505(2)	0.059(1)
C2	0.1206(5)	0.1687(4)	0.9448(2)	0.058(1)
C3	0.2550(6)	0.1462(5)	1.0142(2)	0.070(1)
C4	0.2124(7)	0.2032(5)	1.0879(2)	0.079(1)
C5	0.0387(7)	0.2827(5)	1.0926(2)	0.082(1)
C6	-0.0956(6)	0.3090(5)	1.0256(2)	0.077(1)
C7	0.1657(5)	0.1030(4)	0.8663(2)	0.059(1)
C8	-0.6491(7)	0.4789(6)	0.8172(3)	0.078(1)
N1	-0.1899(4)	0.2832(4)	0.8823(2)	0.065(1)
N2	-0.3594(4)	0.3655(3)	0.8883(1)	0.062(1)
N3	-0.4578(4)	0.3909(3)	0.8195(2)	0.059(1)
O1	0.0610(4)	0.1211(3)	0.8026(1)	0.075(1)
O2	0.3362(4)	0.0219(3)	0.8722(1)	0.077(1)
O3	-0.3991(3)	0.3431(3)	0.7537(1)	0.065(1)

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

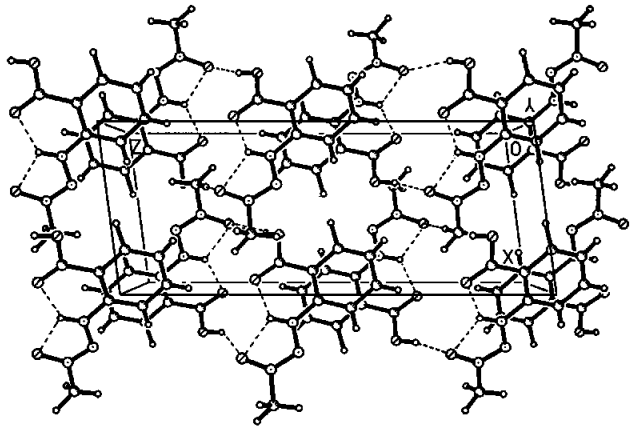

Fig. 3 A unit cell drawing of the packing arrangement and dashed lines indicate N–H···O and O–H···O hydrogen bonds..

Table 3 Bond distances (Å) and angles (°)

C1 - N1	1.383(4)	C1 - C6	1.392(4)
C1 - C2	1.403(4)	C2 - C3	1.393(4)
C2 - C7	1.490(4)	C3 - C4	1.386(5)
C4 - C5	1.352(5)	C5 - C6	1.368(5)
C7 - O1	1.213(4)	C7 - O2	1.323(4)
C8 - N3	1.476(5)	N1 - N2	1.343(4)
N2 - N3	1.275(3)	N3 - O3	1.278(3)
N1 - C1 - C6	121.1(3)	N1 - C1 - C2	120.0(3)
C6 - C1 - C2	118.9(3)	C3 - C2 - C1	118.7(3)
C3 - C2 - C7	120.4(3)	C1 - C2 - C7	121.0(3)
C4 - C3 - C2	120.9(4)	C5 - C4 - C3	119.7(4)
C4 - C5 - C6	121.1(4)	C5 - C6 - C1	120.8(4)
O1 - C7 - O2	122.1(3)	O1 - C7 - C2	124.9(3)
O2 - C7 - C2	113.0(3)	N2 - N1 - C1	119.6(3)
N3 - N2 - N1	111.5(2)	N2 - N3 - O3	123.2(3)
N2 - N3 - C8	117.3(3)	O3 - N3 - C8	119.5(3)

carbonylphenyl)-1-methyltriazene[3] and 3-(2-ethoxycarbonylphenyl)-1-methyl triazene-1-oxide.[3] Bond lengths and angles are in agreement with related structures.[1-3] The average bond length of C–C bonds is 1.382(5) Å for the phenyl ring and this is planar. The N2–N3 bond is shorter than the N1–N2 bond by 0.068(4) Å. The carboxyl group at C2 is oriented to optimize the interaction between O1 with the imine N1–H group of the triazene-*N*-oxide side at C1; it forms an intramolecular hydrogen bond. The dihedral angle between the carboxyl group and the phenyl ring is 2.5(2)° showing the possibility of conjugation. The four atoms of the triazene *N*-oxide group are also planar within the range of 0.003(2) Å and the plane makes a dihedral angle with the phenyl ring of 5.6(2)°. The molecules in the crystal are internally stabilized by two intramolecular N–H···O hydrogen bonds. The hydrogen atom at N1 position participates in a bifurcated hydrogen bond with O1 (N1···O1=2.647(4); H···O1=2.02(3) Å; N1–H···O1= 124(2)°) and O3 (N1···O3=2.474(3); H···O3=2.02(3) Å; N1–H···O3=109(2)°) atoms. The molecular packing is shown in Fig. 3. The arrangement of molecules in the crystal appears to be determined by a hydrogen bond as the dominant intermolecular interaction and by van der Waals interactions. The hydrogen bond is between O2–H group of the carboxyl group and the oxide O(O3) atom at ($-x$, $y-0.5$, $-z+1.5$). The O2···O3 and H···O3 distances are 2.662(3) and 1.81(4) Å and the O2–H···O3 angle is 164(3)°). All H-atoms were located *via* inspection of the difference-Fourier maps and refined with isotropic temperature factors.

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