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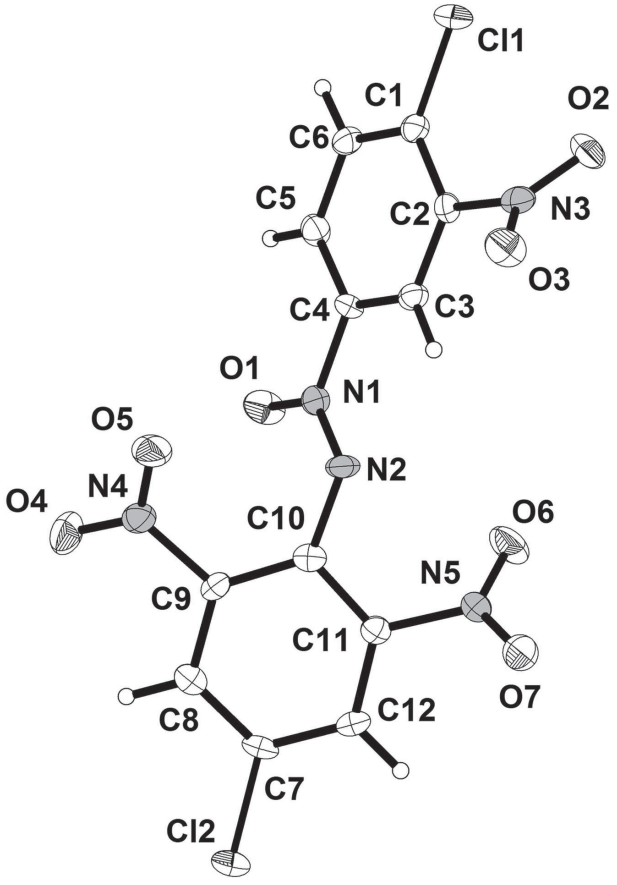
Crystal structure of 2-(4-chloro-2,6-dinitrophenyl)-1-(4-chloro-3-nitrophenyl)diazene 1-oxide, $C_{12}H_5Cl_2N_5O_7$



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Abstract

$C_{12}H_5Cl_2N_5O_7$, triclinic, $P\bar{1}$ (no. 2), $a = 8.2425(8)$ Å, $b = 8.2814(7)$ Å, $c = 12.4713(12)$ Å, $\alpha = 93.370(4)°$, $\beta = 101.590(4)°$, $\gamma = 118.222(3)°$, $V = 723.03(12)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0516$, $wR_{ref}(F^2) = 0.0858$, $T = 173(2)$ K.

CCDC no.: 1850275

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless sheet
Size:	0.19 × 0.17 × 0.05 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.50 mm^{-1}
Diffractometer, scan mode:	Photon 100, ω
θ_{max}, completeness:	25.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7671, 2626, 0.089
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1683
$N(param)_{refined}$:	235
Programs:	Bruker [6], SHELX [7], Olex2 [8]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.7600(5)	0.7866(4)	0.4462(3)	0.0180(8)
C2	0.8634(5)	0.7049(4)	0.4969(3)	0.0160(8)
C3	0.8398(5)	0.6405(4)	0.5953(3)	0.0164(8)
H3	0.9139	0.5895	0.6306	0.020*
C4	0.7070(5)	0.6520(4)	0.6407(3)	0.0158(8)
C5	0.6029(5)	0.7315(4)	0.5938(3)	0.0184(8)
H5	0.5118	0.7381	0.6273	0.022*
C6	0.6325(5)	0.8017(4)	0.4974(3)	0.0201(8)
H6	0.5646	0.8609	0.4660	0.024*
C7	0.7299(4)	0.2928(4)	1.0799(3)	0.0165(8)
C8	0.7384(4)	0.4634(4)	1.0794(3)	0.0167(8)
H8	0.7338	0.5270	1.1437	0.020*
C9	0.7536(4)	0.5400(4)	0.9837(3)	0.0155(8)
C10	0.7560(4)	0.4492(4)	0.8861(3)	0.0159(8)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C11	0.7494(4)	0.2788(4)	0.8933(3)	0.0152(8)
C12	0.7385(4)	0.2004(4)	0.9877(3)	0.0162(8)
H12	0.7370	0.0851	0.9892	0.019*
Cl1	0.79370(13)	0.87469(11)	0.32644(7)	0.0256(3)
Cl2	0.70640(13)	0.19242(12)	1.19747(7)	0.0248(2)
N1	0.6725(4)	0.5764(3)	0.7422(2)	0.0168(7)
N2	0.7855(4)	0.5200(4)	0.7871(2)	0.0186(7)
N3	1.0025(4)	0.6857(4)	0.4484(2)	0.0212(7)
N4	0.7751(4)	0.7269(4)	0.9895(2)	0.0219(7)
N5	0.7539(4)	0.1743(4)	0.7954(2)	0.0202(7)
O1	0.5385(3)	0.5729(3)	0.77512(19)	0.0250(6)
O2	0.9566(4)	0.6263(3)	0.34839(19)	0.0299(6)
O3	1.1540(3)	0.7212(3)	0.51273(19)	0.0287(6)
O4	0.6984(4)	0.7694(3)	1.0515(2)	0.0314(7)
O5	0.8703(4)	0.8288(3)	0.93295(19)	0.0327(7)
O6	0.6406(3)	0.1458(3)	0.70681(19)	0.0301(7)
O7	0.8698(4)	0.1203(3)	0.8099(2)	0.0307(7)

Source of material

The title compound was prepared by nitrifying 1,2-bis(4-chlorophenyl)-diazenze. The raw product was recrystallized from ether at room temperature to give colourless crystals.

Experimental details

All hydrogen atoms were positioned geometrically and treated as riding, with C—H bond lengths constrained to 0.95 Å. The U_{iso} values of the hydrogen atoms were set to 1.2 U_{eq}(C).

Comment

Polynitroazobenzene systems have been investigated extensively owing to their favorable thermally stable performance. [1–3]. Recently, these so called "high energy density materials" have attracted renewed attention in conjunction with their favorable detonation performance and thermally stable performance [4, 5]. In order to study the synthesis mechanism of these compounds, as a by product, 2-(4-chloro-2,6-dinitrophenyl)-1-(4-chloro-3-nitrophenyl)diazene 1-oxide was synthesized and separated.

As shown in the figure two substituated phenyl moieties exist in the molecular structure. The dihedral angle between these rings is 44.7° due a rotation about the the C—N bonds. Owing to steric effects, dihedral angle of the nitro groups and their parent rings are 44.6 (N3/O2,O3), 31.1° (N4/O4,O5) and 52.4° (N5/O6,O7), respectively.

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