organic papers

1-Chloro-3,4-dinitrobenzene–1,4-dioxane (1/1)

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Key indicators

Single-crystal X-ray study
$T = 150$ K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.084
Data-to-parameter ratio = 12.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The solvate structure of 1-chloro-3,4-dinitrobenzene with 1,4-dioxane, $C_6H_3ClN_2O_4 \cdot C_4H_8N_2$, is reported. Alternating molecules of 3,4-dinitro-1-chlorobenzene and 1,4-dioxane are linked by C—H···O hydrogen bonds into a continuous two-dimensional sheet.

Comment

The title compound, (I), was produced during an experimental crystallization polymorph screen on 1-chloro-3,4-dinitro-benzene (3,4-DNCB). Compound (I) crystallizes in the space group $P\bar{1}$ with one molecule of 3,4-DNCB and one molecule of 1,4-dioxane in the asymmetric unit (Fig. 1).

(I)
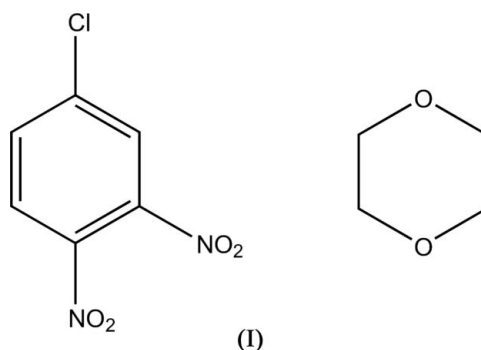
The crystal structure of (I) is characterized by alternating molecules of 3,4-DNCB and 1,4-dioxane, linked by a series of C—H···O hydrogen bonds (Table 1) into a continuous two-dimensional sheet which lies parallel to the $(11\bar{1})$ plane (Fig. 2). Alternating 3,4-DNCB molecules and 1,4-dioxane are linked by pairwise C—H···O hydrogen bonds, forming a chain
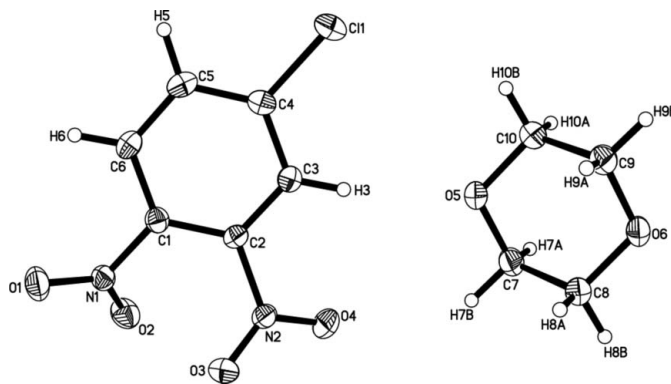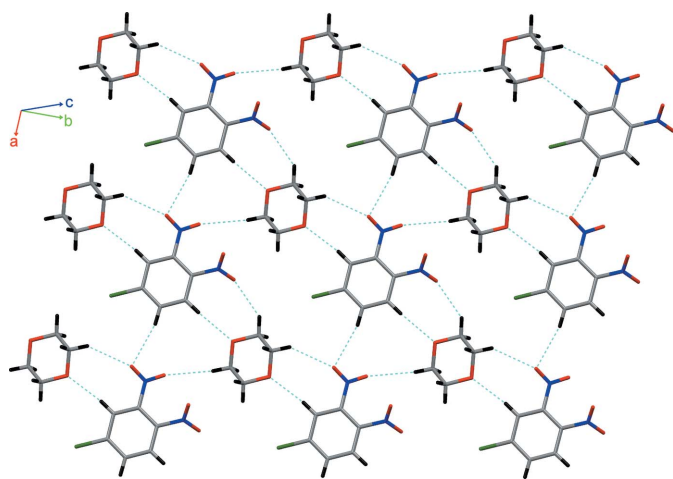

Figure 1
The structure of the asymmetric unit of (I), showing the numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level.

Figure 2
The two-dimensional network formed by compound (I), showing the intermolecular interactions as thin pale-blue lines. Key: C grey, N blue, O red, Cl green and H black.

Figure 3
Packing diagram, showing the stacking of the sheets. C—H···O interactions are shown as thin pale-blue lines. Key: C grey, N blue, O red, Cl green and H black.

which runs parallel to the body diagonal (111). These chains are then hydrogen-bonded together, forming a sheet *via* two C—H···O interactions between two 3,4-DNCB molecules and one C—H···O interaction between the 3,4-DNCB molecule and a 1,4-dioxane molecule of the adjacent chain. Viewing the crystal structure down the *a* axis reveals that there are alternating layers of 3,4-DNCB and 1,4-dioxane (Fig. 3).

Experimental

The title compound was recrystallized from 1,4-dioxane solution by slow evaporation at 298 K.

Crystal data

$C_6H_3ClN_2O_4 \cdot C_4H_8O_2$	$Z = 2$
$M_r = 290.66$	$D_x = 1.616$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 8.2976$ (12) Å	Cell parameters from 3669
$b = 8.7112$ (13) Å	reflections
$c = 8.8015$ (13) Å	$\theta = 2.5–28.2°$
$\alpha = 103.661$ (2)°	$\mu = 0.35$ mm^{-1}
$\beta = 103.909$ (2)°	$T = 150$ (2) K
$\gamma = 91.718$ (2)°	Block, yellow
$V = 597.52$ (15) Å^3	$0.86 \times 0.67 \times 0.45$ mm

Data collection

Bruker SMART APEX diffractometer	2650 independent reflections
	2529 reflections with $I > 2\sigma(I)$
Narrow–frame ω scans	$R_{int} = 0.014$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	$\theta_{max} = 28.2°$
	$h = -10 \rightarrow 10$
$T_{min} = 0.738$, $T_{max} = 0.856$	$k = -11 \rightarrow 11$
4424 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$+ 0.1587P]$
$wR(F^2) = 0.084$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
2647 reflections	$\Delta\rho_{max} = 0.36$ e Å^{-3}
216 parameters	$\Delta\rho_{min} = -0.32$ e Å^{-3}
All H-atom parameters refined	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O5	0.938 (15)	2.331 (15)	3.2553 (14)	168.3 (13)
C5—H5···O4^i	0.969 (17)	2.697 (18)	3.6166 (16)	158.7 (13)
C6—H6···O6^{ii}	0.949 (15)	2.468 (15)	3.4017 (14)	168.1 (12)
C7—H7B···O4	0.990 (17)	2.624 (17)	3.4618 (16)	142.5 (13)
C8—H8B···O1^{iii}	0.996 (19)	2.446 (18)	3.2604 (16)	138.6 (13)
C9—H9B···O3^{iv}	0.951 (18)	2.649 (18)	3.5930 (16)	172.3 (14)

Symmetry codes: (i) $x - 1, y, z$; (ii) $x - 1, y - 1, z - 1$; (iii) $x + 1, y + 1, z + 1$; (iv) $x, y + 1, z + 1$.

H atoms were located in a difference Fourier map and refined freely [C—H = 0.938 (15)–0.996 (19) Å]. The three reflections with the greatest discrepancies were omitted from the refinement.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

References

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