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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.

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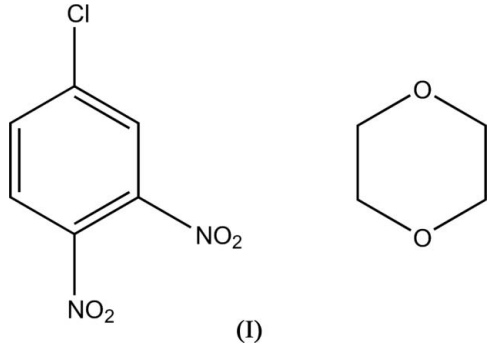
The solvate structure of 1-chloro-3,4-dinitrobenzene with 1,4-dioxane, $C_6H_3ClN_2O_4\cdot C_4H_8N_2$, is reported. Alternating molecules of 3,4-dinitro-1-chlorobenzene and 1,4-dioxane are linked by C—H···O hydrogen bonds into a continuous two-dimensional sheet.

Comment

The title compound, (I), was produced during an experimental crystallization polymorph screen on 1-chloro-3,4-dinitrobenzene (3,4-DNCB). Compound (I) crystallizes in the space group $P\overline{1}$ 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 $(11\overline{1})$ plane (Fig. 2). Alternating 3,4-DNCB molecules and 1,4-dioxane are linked by pairwise C—H···O hydrogen bonds, forming a chain

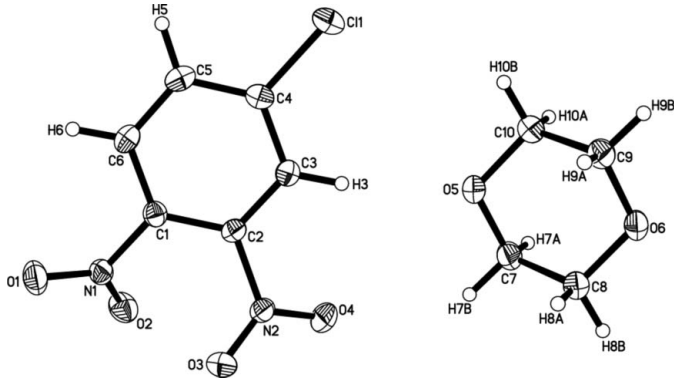


Figure 1
The structure of the asymmetric unit of (I), showing the numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level.

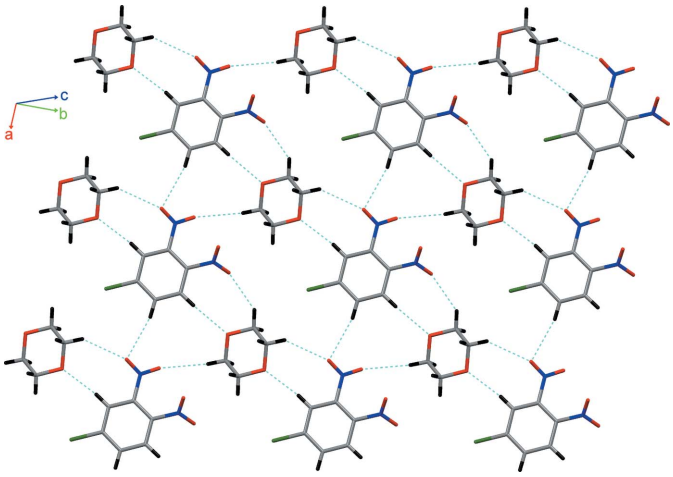


Figure 2
The two-dimensional network formed by compound (I), showing the intermolecular interactions as thin pale-blue lines. Key: C grey, N blue, O red, Cl green and H black.

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

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

Data collection

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

Refinement

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

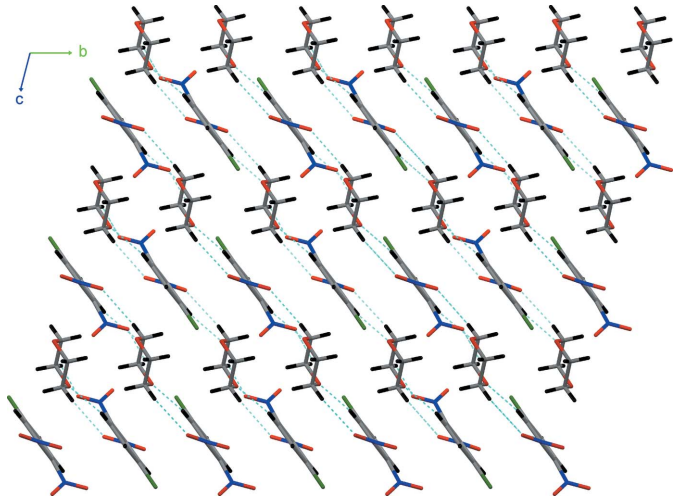


Figure 3
Packing diagram, showing the stacking of the sheets. C—H···O interactions are shown as thin pale-blue lines. Key: C grey, N blue, O red, Cl green and H black.

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—H7*B*···O4	0.990 (17)	2.624 (17)	3.4618 (16)	142.5 (13)
C8—H8*B*···O1^{iii}	0.996 (19)	2.446 (18)	3.2604 (16)	138.6 (13)
C9—H9*B*···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).

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