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3,4-Dichloro-1-nitrobenzene–1,4-dioxane (4/1)

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.081
Data-to-parameter ratio = 15.0

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

The solvate structure of 3,4-dichloro-1-nitrobenzene with 1,4-dioxane, $C_6H_3Cl_2NO_2\cdot0.25C_4H_8N_2$, is reported. The asymmetric unit comprises two independent 3,4-dichloro-1-nitrobenzene molecules and half of a 1,4-dioxane molecule, the solvent molecule being disposed about a centre of inversion. Double chains of 3,4-dichloro-1-nitrobenzene are linked by Cl···Cl interactions and 1,4-dioxane molecules *via* C—H···O hydrogen bonds into a two-dimensional sheet.

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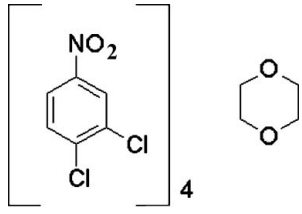
Comment

The title compound, (I), was produced during an automated parallel crystallization polymorph screen on 3,4-dichloro-nitrobenzene (3,4-DCNB). The sample was identified as a novel form using multi-sample X-ray powder diffraction analysis of all recrystallized samples (Florence *et al.*, 2003). Subsequent manual recrystallization from a saturated 1,4-dioxane solution by slow evaporation at 298 K yielded samples suitable for single-crystal X-ray analysis. Compound (I) crystallizes in the space group $P\overline{1}$ with two molecules of 3,4-DCNB and one half-molecule of 1,4-dioxane (disposed about a centre of inversion) in the asymmetric unit (Fig. 1).



(I)

The crystal structure of (I) is characterized by double chains of 3,4-DCNB, linked by Cl···Cl interactions and 1,4-dioxane

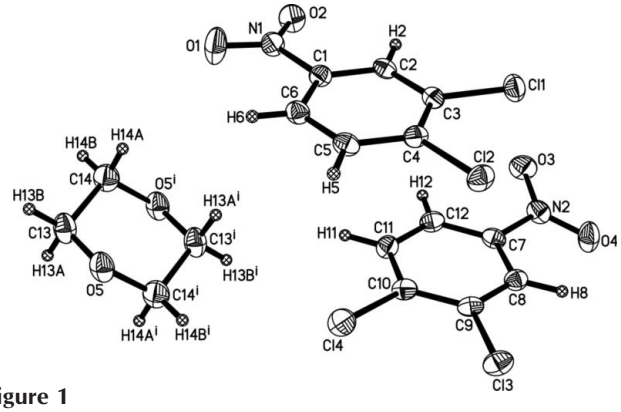


Figure 1
The molecular structure of (I), showing the numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

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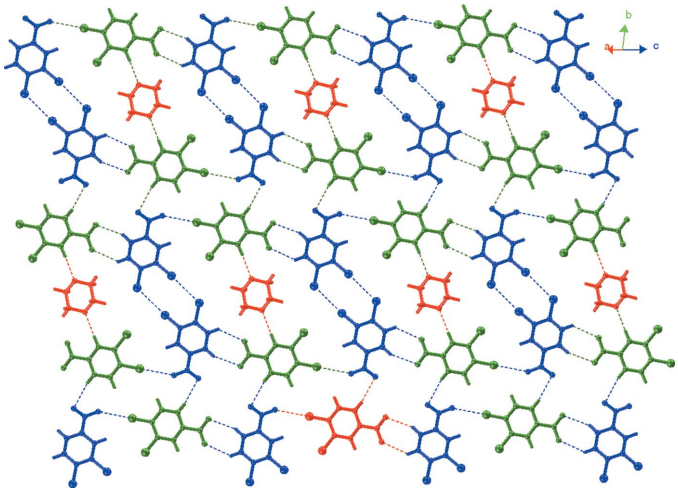

Figure 2
The two-dimensional network formed by (I), showing the intermolecular interactions involved (dashed lines). Colour key: 3,4-DCNB molecule 1 - green; 3,4-DCNB molecule 2 - blue; 1,4-dioxane - red.

molecules to give a two-dimensional sheet parallel to the (212) plane (Fig. 2). Details of the hydrogen-bonding interactions are given in Table 1. 3,4-DCNB molecules of type 2 (C7–C12) are connected to molecules of type 1 (C1–C6) *via* C—H···O hydrogen bonds and N—O···Cl interactions [O4···Cl2^{iv} = 3.013 (1) Å and N2—O4···Cl2^{iv} = 146.9 (1)°; symmetry code: (iv) $2-x, -y, -z$]. This chain is linked to another identical, but antiparallel, chain by a second set of C—H···O hydrogen bonds. These double chains are joined by Cl···Cl interactions through the type 2 molecules [Cl3···Cl4^{v} = Cl4···Cl3^{v} = 3.480 (1) Å and C9—Cl3···Cl4^{v} = C10—Cl4···Cl3^{v} = 160.3 (1)°; symmetry code: (v): $2-x, 1-y, -z$], while the type 1 molecules are linked *via* the 1,4-dioxane solvent molecules by C—H···O hydrogen bonds, thereby forming a two-dimensional sheet. These sheets stack parallel to the (212) plane in an *ABAB* fashion (Fig. 3).

Experimental

A single crystal of the title compound was obtained by recrystallization from a 1,4-dioxane solution by slow evaporation at 298 K.

Crystal data

$C_6H_3Cl_2NO_2\cdot0.25C_4H_8N_2$
M_r = 214.02
Triclinic, $P\overline{1}$
a = 7.3850 (3) Å
b = 9.7359 (3) Å
c = 13.7218 (5) Å
α = 69.347 (2)°
β = 87.209 (2)°
γ = 67.945 (2)°
V = 851.63 (5) Å^3
Z = 4
D_x = 1.669 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3895 reflections
θ = 1.0–27.9°
μ = 0.72 mm^{-1}
T = 123 (2) K
Rod, colourless
0.60 × 0.20 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: none
15060 measured reflections
4003 independent reflections
3176 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 27.8°
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 17$

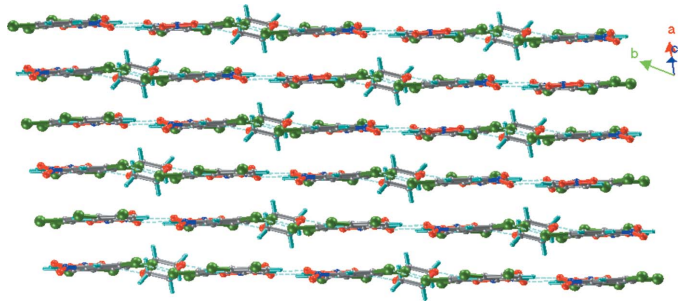

Figure 3
Packing diagram showing the stacking of the sheets.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.081
S = 1.04
4003 reflections
266 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 0.3147P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O1^{i}	0.93 (2)	2.51 (2)	3.396 (2)	158 (2)
C6—H6···O3^{ii}	0.91 (2)	2.55 (2)	3.452 (2)	169 (2)
C2—H2···O5^{iii}	0.95 (2)	2.39 (2)	3.325 (2)	168 (2)

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

The H atoms were refined without constraint. The range of C—H bond distances is 0.91 (2)–1.01 (2) Å.

Data collection: *COLLECT* (Hooft, 1988) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000) and *OLEX* (Dolomanov *et al.*, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek 2003).

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