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# 3,4-Dichloro-1-nitrobenzene–aniline (2/1)

**Sarah A. Barnett,[a]* Andrea Johnston,[b] Alastair J. Florence[b] and Alan R. Kennedy[c]**

[a]Department of Theoretical and Computational Chemistry, University College London, 20 Gordon Street, London WC1H 0AJ, England, [b]Department of Pharmaceutical Sciences, University of Strathclyde, 27 Taylor Street, Glasgow G4 0NR, Scotland, and [c]Department of Pure and Applied Chemistry, University of Strathclyde, 295 Cathedral Street, Glasgow G1 1XL, Scotland

Correspondence e-mail: sarah.barnett@ucl.ac.uk

**Key indicators**

Single-crystal X-ray study
$T$ = 123 K
Mean $\sigma$(C–C) = 0.003 Å
$R$ factor = 0.038
$wR$ factor = 0.080
Data-to-parameter ratio = 14.7

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

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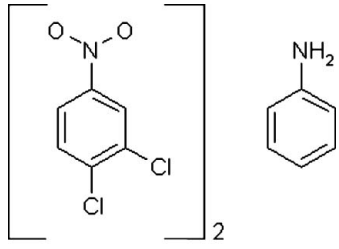
The solvate structure of 3,4-dichloro-1-nitrobenzene with aniline, $2C_6H_3Cl_2NO_2\cdot C_6H_7N$, is reported. Ribbons of 3,4-dichloronitrobenzene, formed by Cl···Cl and N—O···Cl interactions, are linked together *via* N—H···O hydrogen bonds with aniline into an undulating two-dimensional sheet.

## Comment

The title compound, (I), was produced during an automated parallel crystallization polymorph screen on 3,4-dichloronitrobenzene (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 aniline solution by slow evaporation at 298 K yielded samples suitable for single-crystal X-ray analysis. The title solvate, (I), crystallizes in the space group $P2_1/n$ with two molecules of 3,4-DCNB and one molecule of aniline in the asymmetric unit (Fig. 1).




(I)

The crystal structure of (I) is characterized by ribbons of 3,4-DCNB, which are linked by aniline molecules to form a

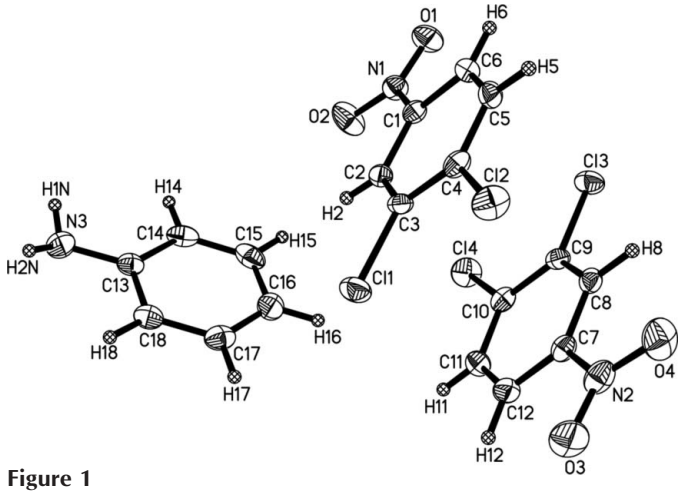



**Figure 1**
The asymmetric unit of (I), showing the numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level.

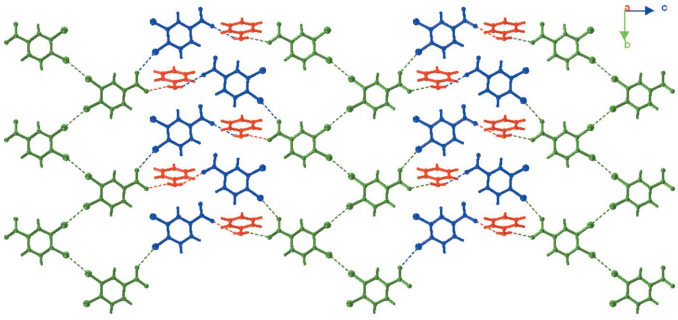


**Figure 2**
The two-dimensional network formed by (I), showing the intermolecular interactions involved as dashed lines (3,4-DCNB molecule 1: green; 3,4-DCNB molecule 2: blue; aniline: red).

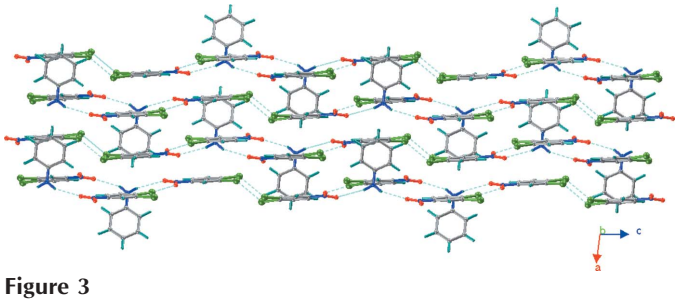


**Figure 3**
Packing diagram viewed perpendicular to the sheets, illustrating the out-of-plane aniline molecules and the stacking arrangement of the sheets. Intermolecular interactions are shown as dashed lines.

continuous sheet (Fig. 2). Molecules of type 1 (C1–C6) form a zigzag chain *via* Cl···Cl interactions [Cl1···Cl2$^{i}$ = 3.399 (1) Å and C3—Cl1···Cl2$^{i}$ = 149.4 (1)°; symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$]. These molecules are then involved in a second contact with molecules of type 2 (C7–C12) *via* N—O···Cl interactions [O2···Cl4$^{ii}$ = 3.056 (2) Å and N1—O2···Cl4$^{ii}$ = 140.1 (1)°; symmetry code: (ii) $1 - x, 1 - y, 1 - z$], thus forming 3,4-DCNB ribbons running parallel to the *b* axis. The aniline solvent molecules, which lie in a perpendicular plane, link these ribbons into an undulating sheet through two N—H···O interactions [N3···O1$^{iii}$ = 2.52 (2) Å and N3—H1N···O1$^{iii}$ = 157 (2)°, and N3···O3$^{i}$ = 2.64 (2) Å and N3—H2N···O3$^{i}$ = 147 (2)°; symmetry code: (iii) $2 - x, 1 - y, 1 - z$]. These sheets form an interdigitated *ABAB* stack parallel to the *a* axis (Fig. 3).

## Experimental

A single-crystal sample of the title compound was recrystallized from aniline solution by slow evaporation at *ca* 293 K.

*Crystal data*

| | |
|---|---|
| $2C_6H_3Cl_2NO_2\cdot C_6H_7N$ | $D_x$ = 1.625 Mg m$^{-3}$ |
| $M_r$ = 477.11 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/n$ | Cell parameters from 4734 reflections |
| $a$ = 6.9774 (2) Å | $\theta$ = 1.0–27.9° |
| $b$ = 10.1668 (3) Å | $\mu$ = 0.64 mm$^{-1}$ |
| $c$ = 27.6762 (7) Å | $T$ = 123 (2) K |
| $\beta$ = 96.495 (2)° | Triangle, orange |
| $V$ = 1950.69 (9) Å$^3$ | 0.45 × 0.30 × 0.15 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | $R_{int}$ = 0.058 |
| $\omega$ and $\varphi$ scans | $\theta_{max}$ = 27.9° |
| Absorption correction: none | $h = -9 \rightarrow 9$ |
| 22354 measured reflections | $k = -13 \rightarrow 13$ |
| 4629 independent reflections | $l = -36 \rightarrow 36$ |
| 3314 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0275P)^2 + 0.7246P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.038 | $(\Delta/\sigma)_{max}$ = 0.002 |
| $wR(F^2)$ = 0.081 | $\Delta\rho_{max}$ = 0.28 e Å$^{-3}$ |
| $S$ = 1.04 | $\Delta\rho_{min}$ = −0.32 e Å$^{-3}$ |
| 4629 reflections | |
| 314 parameters | |
| All H-atom parameters refined | |

C—H distances are in the range 0.09 (2)–1.00 (2) Å, and N—H distances are 0.86 (2) and 0.87 (2) Å.

Data collection: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; cell refinement: *DENZO*; 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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