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Ladders in the supramolecular structure of 1-(4-bromophenyl)-3-[2-(2-hydroxyethyl)amino-5-nitroanilino]-1-propanone dimethylformamide solvate

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The title compound, $C_{17}H_{18}BrN_3O_4\cdot C_3H_7NO$, has a supramolecular structure which is based on two antiparallel *C*(11) chains, formed by O—H···O hydrogen bonds. It is enhanced by a further linkage *via* two N—H···O hydrogen bonds to the DMF solvent molecule, forming an $R^1_2(7)$. C—H···O weak hydrogen bonds link adjacent molecules in the chain. These chains are then linked together by two C—H···O weak hydrogen bonds forming a molecular ladder, which runs parallel to the *b* axis and comprises a series of ring structures.

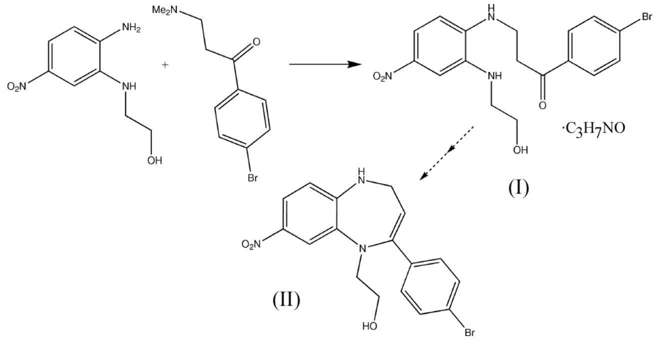
Key indicators

Single-crystal X-ray study
T = 120 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.088
Data-to-parameter ratio = 17.0

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

Comment

In our ongoing research into the synthesis of new biologically interesting diazepine fused derivatives we attempted the reaction of 3-dimethylamino-1-(4-bromophenyl)-1-propanone, a precursor of the corresponding *α*,*β*-unsaturated ketone, with 2-(2-amino-4-nitroanilino)ethanol, a highly functionalized *ortho*-diamino compound, but instead of the expected diazepine derivative we isolated only the intermediate title compound, (I).



The asymmetric unit of (I) was chosen such that the bromo molecule and the solvent DMF molecule form an $R^1_2(7)$ ring (Bernstein *et al.*, 1997), *via* the N1—H1···O21 and N12—H12···O21 hydrogen bonds (Fig. 1).

In the following discussion, symmetry codes are as in Table 1. The main supramolecular structure is defined by the O12—H12*A*···O151^{i} hydrogen bond, which links the molecules into a *C*(11) chain running parallel to the *b* axis. The C21—H21···O152^{i} interaction forms a link in which the solvent molecule acts a bridge to adjacent molecules in the chain, creating an $R^3_3(12)$ ring.

Centres-of-symmetry at points ($\frac{3}{2}$, $n+\frac{1}{2}$, $\frac{1}{2}$), where *n* is any positive or negative integer, produce an antiparallel chain, which is linked to the original by the C13—H13···O12^{ii} and C14—H14···O151^{iii} weak hydrogen bonds. The former, with its symmetry-related bond C13^{ii}—H13^{ii}···O12, form a series of $R^2_2(14)$ rings, centred on centres of symmetry, as described above where *n* is even. The latter, with its symmetry-related

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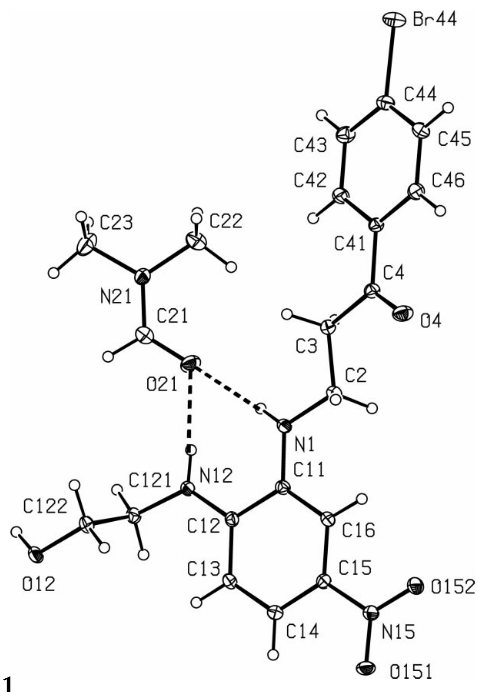

Figure 1
A view of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are indicated by dashed lines.

bond C14^{iii}—H13^{iii}$\cdots$O12, forms a series of $R_2^2(10)$ rings, centred on centres of symmetry, as described above, where *n* is odd. These two bonds, along with the O12—H12*A*$\cdots$O151 hydrogen bond in each antiparallel chain, form a series of staggered $R_3^2(7)$ rings which lie between each of the centrosymmetric rings. This produces molecular ladders, which lie parallel (001) and run along the *b* axis.

Details of the hydrogen bonds are given in Table 1 and a stereoview of this supramolecular structure is shown in Fig. 2.

A similar ladder structure, with molecules in the 'uprights' connected by N—H$\cdots$O hydrogen bonds and the 'rungs' consisting of weak C—H$\cdots$O hydrogen bonds, is found in the structure of 2-trifluoromethyl-4-nitroaniline (Glidewell *et al.*, 2002).

In addition, there is a short contact between methyl atom H22*B* and O4^{iv}.

Examination of the structure with *PLATON* (Spek, 2002) showed that there are no solvent-accessible voids in the crystal lattice.

Experimental

A solution of equimolar amounts of 2-(2-amino-4-nitroanilino)-ethanol (700 mg, 3.55 mmol) and 3-dimethylamino-1-(4-bromophenyl)-1-propanone in 15 ml of absolute ethanol was heated to reflux for 30 min. The resulting precipitate was filtered, washed with ethanol and recrystallized from ethanol, yielding red crystals (m.p. 434 K, yield 55%). Analysis calculated for $C_{17}H_{18}BrN_3O_4$: C,50.02, H 4.44, N 10.29%; found: C 50.11, H 4.52, N 10.24%.

Crystal data

$C_{17}H_{18}BrN_3O_4\cdot C_3H_7NO$
$M_r = 481.35$
Triclinic, $P\overline{1}$
$a = 10.5267$ (2) Å
$b = 10.7688$ (3) Å
$c = 10.9397$ (3) Å
$\alpha = 77.646$ (2)°
$\beta = 72.743$ (2)°
$\gamma = 62.7565$ (12)°
$V = 1048.45$ (5) Å^3
$Z = 2$
$D_x = 1.525$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4648 reflections
$\theta = 3.0$–27.5°
$\mu = 2.00$ mm^{-1}
$T = 120$ (1) K
Block, red
0.30 × 0.25 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.599$, $T_{max} = 0.670$
13 949 measured reflections
4648 independent reflections
3927 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 27.5°$
$h = -13 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.088$
$S = 1.05$
4648 reflections
274 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0328P)^2 + 0.7369P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.57$ e Å^{-3}

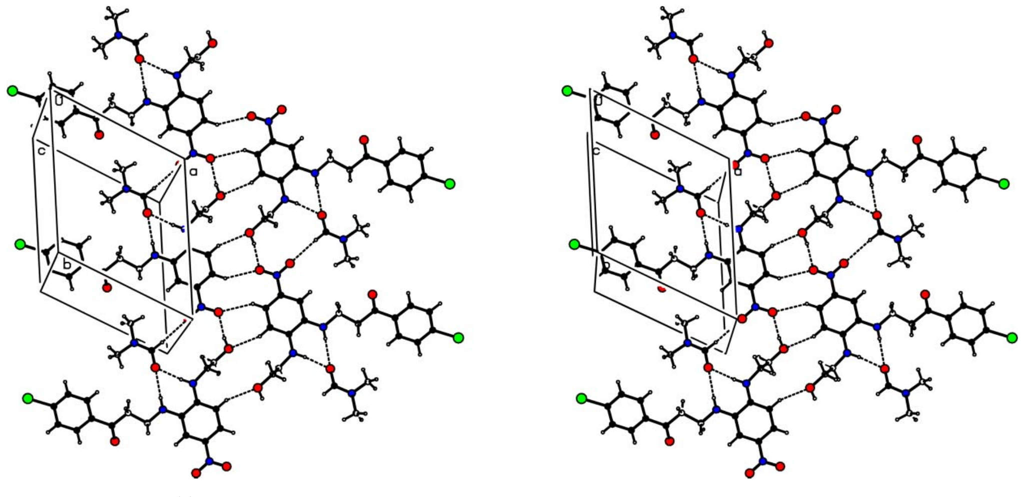

Figure 2
A stereoview of the crystal structure of (I). Hydrogen bonds are indicated by dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O21	0.88	2.17	3.035 (3)	169
O12—H12*E*···O151^{i}	0.84	1.96	2.793 (3)	171
N12—H12···O21	0.88	1.99	2.868 (3)	173
C13—H13···O12^{ii}	0.95	2.44	3.332 (3)	155
C14—H14···O151^{iii}	0.95	2.47	3.315 (3)	148
C21—H21···O152^{i}	0.95	2.47	3.407 (3)	171
C22—H22*B*···O4^{iv}	0.98	2.50	3.439 (4)	160

Symmetry codes: (i) $x, y-1, z$; (ii) $3-x, -y, 1-z$; (iii) $3-x, 1-y, 1-z$; (iv) $1-x, 1-y, 2-z$.

H atoms were treated as riding atoms, with C—H = 0.95–0.99 Å, N—H = 0.88 Å and O—H = 0.84 Å. The position of the hydroxy H atom was determined from a difference map and then idealized.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2002); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

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