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Key indicators

Single-crystal X-ray study
T = 120 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.083
wR factor = 0.222
Data-to-parameter ratio = 16.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Hydrogen-bonded chains in 1-[3-(4-nitrobenzylideneamino)propyl]-2-(4-nitrophenyl)-hexahydropyrimidine methanol solvate

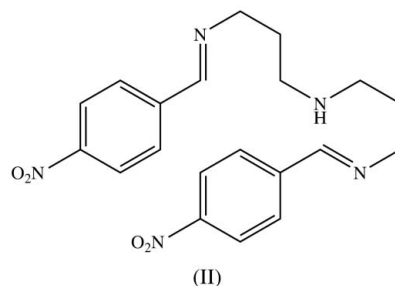
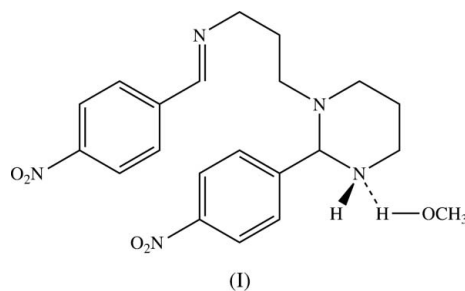
In the title compound, $\text{C}_{20}\text{H}_{23}\text{N}_5\text{O}_4 \cdot \text{CH}_4\text{O}$, the molecular components are linked into $\text{C}_2^2(4)$ chains by a combination of $\text{O}-\text{H} \cdots \text{N}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds.

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Comment

We report here the structure of the title compound, (I) (Fig. 1), a stoichiometric methanol solvate, wherein the pyrimidine component was formed by spontaneous cyclization of the intermediate (II) (see scheme) produced by condensation of bis(3-aminopropyl)amine with two molar equivalents of 4-nitrobenzaldehyde. We have recently reported the supramolecular structures of the 4-nitrobenzylidene derivatives of 1,2-diaminoethane and 1,3-diaminopropane (Bomfim *et al.*, 2005), 1,8-diaminooctane (Glidewell *et al.*, 2005*b*) and tris(2-aminoethyl)amine (Glidewell *et al.*, 2005*a*). The target compound, (II), was intended to complement that series but, in the event, the ring-closed product (I) resulted.



For the hexahydropyrimidine ring (N11/C12/N13/C14–C16), the ring-puckering parameters [$\theta = 176.1(3)^\circ$ and $\varphi = 162(5)^\circ$; Cremer & Pople, 1975] indicate an almost perfect chair conformation. The substituents at N11 and C12 both occupy equatorial sites, as expected, but the N–H bond at N13 occupies an axial site with the lone pair at N13 equatorial. The bond lengths and angles show no unexpected features.

Within the selected asymmetric unit (Fig. 1) the components are linked by an almost linear $\text{O}-\text{H} \cdots \text{N}$ hydrogen bond (Table 1). In addition, atom N13 at (x, y, z) acts as hydrogen-

bond donor to methanol atom O41 at $(1 - x, \frac{1}{2} + y, \frac{3}{2} - z)$, so forming a $C_2^2(4)$ (Bernstein *et al.*, 1995) chain running parallel to the [010] direction and generated by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{3}{4})$ (Fig. 2). Two such chains, related to one another by inversion and hence antiparallel, pass through each unit cell, but there are no significant direction-specific interactions between adjacent chains. Two C—H...O contacts between chains (Table 1) both involve aliphatic C—H bonds of very low acidity, both are long, and hence are probably not structurally significant.

Experimental

A solution of 4-nitrobenzaldehyde (4 mmol) and bis(3-aminopropyl)amine (2 mmol) in methanol (25 ml) was heated under reflux for 2 h. The mixture was cooled to ambient temperature and the solvent was removed under reduced pressure. Recrystallization of the crude solid product from methanol provided crystals of compound (I) suitable for single-crystal X-ray diffraction.

Crystal data

$C_{20}H_{23}N_5O_4 \cdot CH_4O$	$Z = 4$
$M_r = 429.48$	$D_x = 1.330 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 16.9257 (16) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$b = 7.5144 (8) \text{ \AA}$	$T = 120 (2) \text{ K}$
$c = 16.8911 (15) \text{ \AA}$	Plate, colourless
$\beta = 93.562 (5)^\circ$	$0.24 \times 0.22 \times 0.06 \text{ mm}$
$V = 2144.2 (4) \text{ \AA}^3$	

Data collection

Bruker–Nonius KappaCCD diffractometer	23793 measured reflections
φ and ω scans	4712 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	3010 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.965$, $T_{\max} = 0.994$	$R_{\text{int}} = 0.067$
	$\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0907P)^2 + 2.3771P]$
$R[F^2 > 2\sigma(F^2)] = 0.083$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.222$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
4712 reflections	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
282 parameters	
H-atom parameters constrained	

Table 1

Hydrogen bonds and short intermolecular contacts (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O41—H41...N13	0.84	1.94	2.782 (4)	178
N13—H13...O41 ⁱ	0.88	1.97	2.841 (3)	171
C17—H17B...O32 ⁱⁱ	0.99	2.55	3.371 (4)	140
C18—H18A...O31 ⁱⁱⁱ	0.99	2.56	3.490 (4)	156

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (iii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$.

All H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 (aromatic and =CH—), 0.98 (CH₃), 0.99 (CH₂) or 1.00 \AA (aliphatic CH), N—H = 0.88 \AA and O—H = 0.84 \AA , and with $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C,N,O})$, where $k = 1.5$ for the methanol H atoms and 1.2 for all other H atoms.

Data collection: COLLECT (Hooft, 1999); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduc-

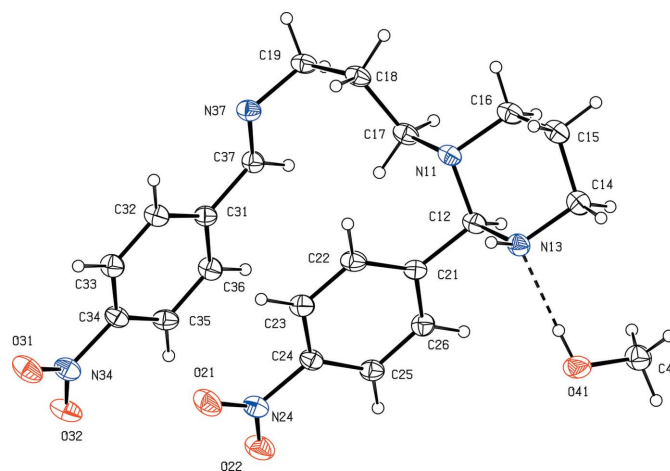


Figure 1

The molecular components of compound (I), showing the atom-labelling scheme and the O—H...N hydrogen bond (dashed line) within the selected asymmetric unit. Displacement ellipsoids are drawn at the 30% probability level.

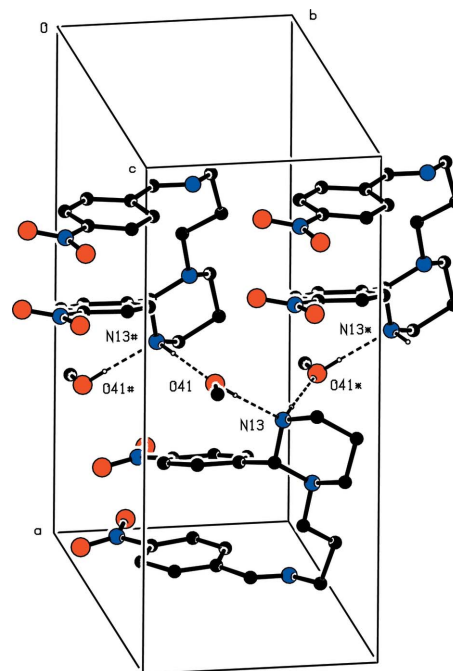


Figure 2

Part of the crystal structure of compound (I), showing the formation of a $C_2^2(4)$ chain along [010]. For the sake of clarity, H atoms not involved in the motif shown have been omitted. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(1 - x, \frac{1}{2} + y, \frac{3}{2} - z)$ and $(1 - x, -\frac{1}{2} + y, \frac{3}{2} - z)$, respectively. Dashed lines indicate hydrogen bonds.

tion: DENZO and COLLECT; program(s) used to solve structure: OSCAIL (McArdle, 2003) and SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: OSCAIL and SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PRPKAPPA (Ferguson, 1999).

X-ray data were collected at the EPSRC National X-ray Crystallography Service, University of Southampton, England, using a Nonius KappaCCD diffractometer. The

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