



ISSN 2414-3146

N,N'-Bis(pyridin-2-yl)octanediamide

Błażej Dziuk,^a Borys Ośmiałowski,^b Krzysztof Ejsmont^a and Bartosz Zarychta^{a*}

^aFaculty of Chemistry, University of Opole, Oleska 48, 45-052 Opole, Poland, and ^bFaculty of Chemical Technology and Engineering, University of Technology and Life Sciences, Seminaryjna 3, 85-326 Bydgoszcz, Poland. *Correspondence e-mail: bartosz.zarychta@uni.opole.pl

Received 2 August 2016

Accepted 13 August 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

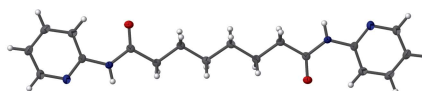
Keywords: crystal structure; adamantane; anti-viral effects; MOF self-assembly; hydrogen bonding.

CCDC reference: 1499041

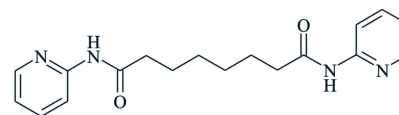
Structural data: full structural data are available from iucrdata.iucr.org

The complete molecule of the title compound, C₁₈H₂₂N₄O₂, is generated by crystallographic inversion symmetry. In the crystal, N—H···N hydrogen bonds connect the molecules into [010] chains, which feature *R*₂²(8) loops. The packing is consolidated by C—H···O interactions.

3D view



Chemical scheme



Structure description

In the last decade, bidentate, flexible ligands have gained considerable interest from the metal–organic framework (MOF) and crystal engineering communities owing to their use as building blocks for coordination polymers (Hennigar *et al.*, 1997; Awaleh *et al.*, 2005; Chen *et al.*, 2007; Cheng *et al.* 2009). For those ligands, the longer the backbone chain is, the less predictable the resulting network, resulting in a number of structural types. Herein we report the structure of *N,N'*-bis(pyridin-2-yl)octanediamide, as a candidate to expand studies on self-assembly of MOFs (Ośmiałowski *et al.*, 2010, 2013).

There is one independent half-molecule in the asymmetric unit (Fig. 1), with an inversion centre at the mid-point of the C—C bond of the backbone chain. The molecule is almost planar with C(N)—C(N)—C—C torsion angles in the range 174.3 (1) to 180.0 (1)°. The atoms in the backbone chain are arranged in an antiperiplanar conformation. The oxygen atoms deviate most from the planarity of the molecule. Nevertheless the distance between the plane defined by C1/N2/C6/C7/C8/C9 and the O1 atom is less than 0.10 Å. The pyridine ring is co-planar with the amide bond, and the C1—N2 bond length of 1.4000 (16) Å is notably shorter than its average literature value [1.465 (7) Å; Allen, *et al.* 2006]. This suggests partial conjugation between those two π -electron systems. An intramolecular C2—H2···O1 hydrogen bond is observed.

The crystal structure (Fig. 2) features two symmetrically independent hydrogen bonds (Table 1). The N2—H2A···N1(−*x*, −*y* + 1, −*z* + 2) hydrogen bond generates [010] chains incorporating inversion dimers. This is reinforced by the C4—H4···O1(−*x* + $\frac{1}{2}$, *y* + $\frac{1}{2}$,

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N2–H2A···N1 ⁱ	0.86	2.45	3.3065 (15)	171
C2–H2···O1	0.93	2.28	2.8716 (15)	121
C4–H4···O1 ⁱⁱ	0.93	2.42	3.1585 (15)	136

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

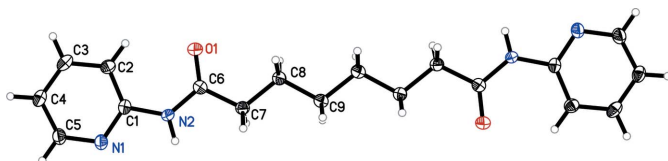


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. Only the asymmetric unit is labeled.

$-z + \frac{3}{2}$) interaction, which generates (101) layers, connected to each other by weak π – π (pyridine ring) interactions and short H···H (backbone) contacts. The perpendicular separation of the mean planes through the rings is 3.287 Å while the H8B···H8B($-x, y, -z + \frac{3}{2}$) distance is 2.290 Å (sum of van der Waals radii = 2.4 Å).

Synthesis and crystallization

Suberoyl chloride (1 equivalent) was added as a solution in dichloromethane (20 ml) to a magnetically stirred mixture of 2-aminopyridine (2 equivalents) and triethylamine in dichloromethane (50 ml). The reaction was stirred for 24 h at room temperature and the solvent evaporated under vacuum. The residual organic phase was treated with saturated Na₂CO₃ solution and extracted with chloroform. The obtained extracts were dried with MgSO₄ and evaporated to dryness and recrystallized from ethanol solution. Crystals suitable for XRD analysis were obtained by dissolving a small portion of the title compound in chloroform and allowing the solvent to evaporate slowly.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

References

- Allen, F. H., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (2006). *International Tables for Crystallography*, Vol. C, ch. 9.5, pp. 790–811.
- Awaleh, M. O., Badia, A. & Brisse, F. (2005). *Cryst. Growth Des.* **5**, 1897–1906.

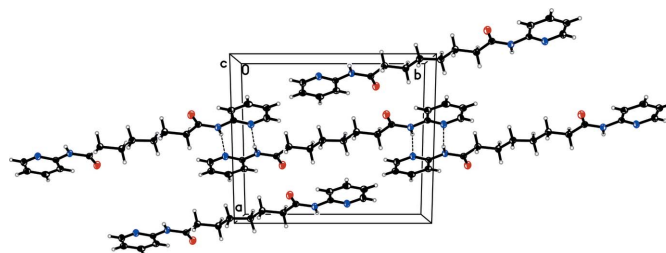


Figure 2

The crystal packing of the title compound, viewed along the *c* axis showing the N2–H2A···N1 ($-x, -y + 1, -z + 2$) hydrogen bonds as dashed lines.

Table 2

Experimental details.

Crystal data	
Chemical formula	C ₁₈ H ₂₂ N ₄ O ₂
<i>M_r</i>	326.39
Crystal system, space group	Monoclinic, <i>C2/c</i>
Temperature (K)	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.9289 (7), 13.2908 (6), 11.5000 (6)
β (°)	111.497 (7)
<i>V</i> (Å ³)	1696.43 (17)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.09
Crystal size (mm)	0.25 × 0.23 × 0.18
Data collection	
Diffractometer	Oxford Diffraction Xcalibur
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	5224, 1494, 1125
<i>R</i> _{int}	0.024
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.594
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.032, 0.073, 0.95
No. of reflections	1494
No. of parameters	110
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	0.24, -0.15

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2008), *SHELXS2013* (Sheldrick, 2008) and *SHELXL2013* (Sheldrick, 2015).

- Chen, H.-C., Hu, H.-L., Chan, Z.-K., Yeh, C.-W., Jia, H.-W., Wu, C.-P., Chen, J.-D. & Wang, J.-C. (2007). *Cryst. Growth Des.* **7**, 698–704.
- Cheng, P.-C., Wu, C.-J., Chen, H.-C., Chen, J.-D. & Wang, J.-C. (2009). *Acta Cryst.* **E65**, o1825.
- Hennigar, T., MacQuarrie, D. C., Losier, P., Rogers, R. D. & Zaworotko, M. J. (1997). *Angew. Chem. Int. Ed. Engl.* **36**, 972–973.
- Osmiałowski, B., Kolehmainen, E., Dobosz, R., Gawinecki, R., Kauppinen, R., Valkonen, A., Koivukorpi, J. & Rissanen, K. (2010). *J. Phys. Chem. A*, **114**, 10421–10426.
- Osmiałowski, B., Kolehmainen, E., Ejsmont, K., Ikonen, S., Valkonen, A., Rissanen, K. & Nonappa (2013). *J. Mol. Struct.* **1054–1055**, 157–163.
- Oxford Diffraction (2008). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.

full crystallographic data

IUCrData (2016). **1**, x161309 [doi:10.1107/S2414314616013092]

N,N'-Bis(pyridin-2-yl)octanediamide

Błażej Dziuk, Borys Ośmiałowski, Krzysztof Ejsmont and Bartosz Zarychta

N,N'-Bis(pyridin-2-yl)octanediamide*Crystal data*

$C_{18}H_{22}N_4O_2$	$F(000) = 696$
$M_r = 326.39$	$D_x = 1.278 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 11.9289 (7) \text{ \AA}$	Cell parameters from 5224 reflections
$b = 13.2908 (6) \text{ \AA}$	$\theta = 3.5\text{--}25.2^\circ$
$c = 11.5000 (6) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 111.497 (7)^\circ$	$T = 100 \text{ K}$
$V = 1696.43 (17) \text{ \AA}^3$	Irregular, colourless
$Z = 4$	$0.25 \times 0.23 \times 0.18 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur diffractometer	1494 independent reflections
Radiation source: fine-focus sealed tube	1125 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.024$
Detector resolution: 1024×1024 with blocks 2 x 2 pixels mm^{-1}	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 3.5^\circ$
ω -scan	$h = -14 \rightarrow 13$
5224 measured reflections	$k = -15 \rightarrow 15$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.032$	$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2]$
$wR(F^2) = 0.073$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.95$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1494 reflections	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
110 parameters	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$
0 restraints	Extinction correction: SHELXL2013 (Sheldrick, 2015),
Primary atom site location: structure-invariant direct methods	$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0012 (3)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.15817 (8)	0.27316 (7)	0.84143 (8)	0.0259 (3)
N1	0.10057 (9)	0.57410 (8)	0.92312 (10)	0.0228 (3)
N2	0.08189 (9)	0.40263 (7)	0.92055 (10)	0.0189 (3)
H2A	0.0393	0.4149	0.9652	0.023*
C1	0.12421 (10)	0.48726 (9)	0.87675 (11)	0.0177 (3)
C2	0.18323 (11)	0.48219 (10)	0.79260 (12)	0.0213 (3)
H2	0.1975	0.4206	0.7621	0.026*
C3	0.21992 (11)	0.57095 (10)	0.75567 (13)	0.0238 (3)
H3	0.2594	0.5699	0.6994	0.029*
C4	0.19788 (11)	0.66159 (10)	0.80261 (12)	0.0235 (3)
H4	0.2225	0.7223	0.7795	0.028*
C5	0.13834 (11)	0.65855 (9)	0.88437 (13)	0.0253 (3)
H5	0.1229	0.7195	0.9154	0.030*
C6	0.09926 (11)	0.30281 (9)	0.90189 (11)	0.0184 (3)
C7	0.03884 (11)	0.23330 (9)	0.96366 (11)	0.0196 (3)
H7A	-0.0464	0.2492	0.9333	0.023*
H7B	0.0713	0.2462	1.0529	0.023*
C8	0.05278 (11)	0.12258 (9)	0.94244 (12)	0.0203 (3)
H8A	0.1378	0.1071	0.9668	0.024*
H8B	0.0141	0.1080	0.8540	0.024*
C9	-0.00115 (12)	0.05536 (9)	1.01516 (12)	0.0196 (3)
H9A	0.0430	0.0652	1.1038	0.023*
H9B	-0.0839	0.0756	0.9973	0.023*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0303 (5)	0.0225 (5)	0.0322 (6)	0.0020 (4)	0.0199 (5)	-0.0011 (4)
N1	0.0243 (6)	0.0180 (6)	0.0279 (7)	-0.0012 (5)	0.0117 (5)	0.0013 (5)
N2	0.0216 (6)	0.0172 (6)	0.0227 (6)	0.0009 (5)	0.0138 (5)	0.0007 (5)
C1	0.0144 (6)	0.0184 (7)	0.0180 (7)	-0.0003 (5)	0.0032 (5)	0.0028 (5)
C2	0.0187 (7)	0.0242 (7)	0.0215 (7)	0.0010 (6)	0.0080 (6)	0.0004 (6)
C3	0.0176 (7)	0.0335 (8)	0.0211 (7)	-0.0009 (6)	0.0081 (6)	0.0057 (6)
C4	0.0193 (7)	0.0247 (8)	0.0271 (8)	-0.0011 (6)	0.0091 (6)	0.0064 (6)
C5	0.0265 (8)	0.0185 (8)	0.0330 (8)	-0.0014 (6)	0.0133 (6)	0.0005 (6)
C6	0.0168 (6)	0.0192 (7)	0.0173 (7)	0.0011 (5)	0.0040 (6)	-0.0003 (5)
C7	0.0199 (7)	0.0200 (7)	0.0198 (7)	0.0010 (5)	0.0084 (6)	-0.0001 (5)
C8	0.0231 (7)	0.0190 (7)	0.0200 (7)	0.0009 (5)	0.0093 (6)	0.0002 (5)
C9	0.0199 (7)	0.0203 (7)	0.0174 (7)	0.0021 (5)	0.0055 (6)	-0.0002 (5)

Geometric parameters (\AA , $^\circ$)

O1—C6	1.2209 (14)	C4—H4	0.9300
N1—C1	1.3440 (16)	C5—H5	0.9300
N1—C5	1.3448 (16)	C6—C7	1.5010 (17)

N2—C6	1.3717 (16)	C7—C8	1.5108 (17)
N2—C1	1.4000 (16)	C7—H7A	0.9700
N2—H2A	0.8600	C7—H7B	0.9700
C1—C2	1.3914 (18)	C8—C9	1.5185 (18)
C2—C3	1.3783 (17)	C8—H8A	0.9700
C2—H2	0.9300	C8—H8B	0.9700
C3—C4	1.3840 (18)	C9—C9 ⁱ	1.515 (2)
C3—H3	0.9300	C9—H9A	0.9700
C4—C5	1.3711 (19)	C9—H9B	0.9700
C1—N1—C5	116.16 (11)	O1—C6—C7	123.17 (11)
C6—N2—C1	128.73 (11)	N2—C6—C7	113.27 (10)
C6—N2—H2A	115.6	C6—C7—C8	115.01 (10)
C1—N2—H2A	115.6	C6—C7—H7A	108.5
N1—C1—C2	123.42 (11)	C8—C7—H7A	108.5
N1—C1—N2	113.04 (11)	C6—C7—H7B	108.5
C2—C1—N2	123.53 (12)	C8—C7—H7B	108.5
C3—C2—C1	118.14 (12)	H7A—C7—H7B	107.5
C3—C2—H2	120.9	C7—C8—C9	112.96 (10)
C1—C2—H2	120.9	C7—C8—H8A	109.0
C2—C3—C4	119.85 (13)	C9—C8—H8A	109.0
C2—C3—H3	120.1	C7—C8—H8B	109.0
C4—C3—H3	120.1	C9—C8—H8B	109.0
C5—C4—C3	117.52 (12)	H8A—C8—H8B	107.8
C5—C4—H4	121.2	C9 ⁱ —C9—C8	113.43 (13)
C3—C4—H4	121.2	C9 ⁱ —C9—H9A	108.9
N1—C5—C4	124.92 (12)	C8—C9—H9A	108.9
N1—C5—H5	117.5	C9 ⁱ —C9—H9B	108.9
C4—C5—H5	117.5	C8—C9—H9B	108.9
O1—C6—N2	123.56 (12)	H9A—C9—H9B	107.7

Symmetry code: (i) $-x, -y, -z+2$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A \cdots N1 ⁱⁱ	0.86	2.45	3.3065 (15)	171
C2—H2 \cdots O1	0.93	2.28	2.8716 (15)	121
C4—H4 \cdots O1 ⁱⁱⁱ	0.93	2.42	3.1585 (15)	136

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