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Hexane-1,6-diammonium bis(pyridine-2-carboxylate)

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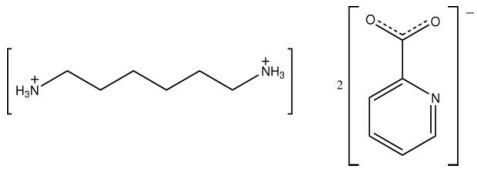
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.057; wR factor = 0.124; data-to-parameter ratio = 14.0.

The title compound, $C_6H_{18}N_2^{2+}\cdot 2C_6H_4NO_2^-$, consists of a doubly protonated hexamethylenediammonium dication and two pyridine-2-carboxylate anions. These ions interact by means of intermolecular N—H···O and N—H···N hydrogen bonds to form a two-dimensional array. The carboxylate groups of the anions appear to be delocalized on the basis of the C—O bond lengths.

Related literature

For the crystal structures of $(C_6H_{18}N_2)X_2$ (X = Cl, Br or I), see: Binnie & Robertson (1949*a*,*b*); Borkakoti *et al.* (1978); van Blerk & Kruger (2008). For details of some other hexane-1,6-diammonium compounds, see: Phan Thanh *et al.* (2000); Mousdis *et al.* (2000); Rakovský *et al.* (2002); Dammak *et al.* (2007); Sun *et al.* (2007); Yang *et al.* (2007); Wilkinson & Harrison (2007); Wang & Wei (2007). For the structure of pyridine-2-carboxylic acid, see: Hamazaki *et al.* (1998).



Experimental

Crystal data

$C_6H_{18}N_2^{2+}\cdot 2C_6H_4NO_2^-$
M_r = 362.43
Monoclinic, $P2_1/c$
a = 9.8182 (7) Å
b = 9.1569 (7) Å
c = 21.6423 (17) Å
β = 99.038 (2)°
V = 1921.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.33 × 0.25 × 0.18 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.685, T_{max} = 0.984
13964 measured reflections
4752 independent reflections
1740 reflections with $I > 2\sigma(I)$
R_{int} = 0.089

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.124
S = 0.93
4752 reflections
339 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O4	1.07 (3)	1.70 (3)	2.747 (3)	165 (3)
N3—H3B···O1^{i}	0.96 (3)	2.29 (3)	3.088 (3)	140 (2)
N3—H3B···N1^{i}	0.96 (3)	2.15 (3)	2.962 (3)	142 (2)
N3—H3C···O1^{ii}	0.92 (3)	1.91 (3)	2.835 (3)	177 (3)
N4—H4A···O3^{iii}	0.97 (3)	2.27 (3)	3.064 (3)	139 (2)
N4—H4A···N2^{iii}	0.97 (3)	2.12 (3)	2.963 (3)	144 (2)
N4—H4B···O3^{iv}	1.07 (3)	1.67 (3)	2.740 (3)	175 (3)
N4—H4C···O2^{v}	1.05 (3)	1.70 (4)	2.754 (3)	179 (3)
C1—H1···O4^{vi}	1.02 (3)	2.45 (3)	3.328 (4)	145 (2)
C16—H16B···O3^{iv}	1.01 (3)	2.58 (3)	3.426 (4)	140.8 (18)

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2459).

References

Binnie, W. P. & Robertson, J. M. (1949*a*). *Acta Cryst.* **2**, 116–120.
Binnie, W. P. & Robertson, J. M. (1949*b*). *Acta Cryst.* **2**, 180–188.
Blerk, C. van & Kruger, G. J. (2008). *Acta Cryst.* C**64**, o537–o542.
Borkakoti, N., Lindley, P. F., Moss, D. S. & Palmer, R. A. (1978). *Acta Cryst.* B**34**, 3431–3433.
Bruker (2000). *SADABS*, *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dammak, T., Fourati, N., Boughzala, H., Mlayah, A. & Abid, Y. (2007). *J. Lumin.* **127**, 404–408.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hamazaki, H., Hosomi, H., Takeda, S., Kataoka, H. & Ohba, S. (1998). *Acta Cryst.* C**54** IUC9800049.
Mousdis, G. A., Papavassiliou, G. C., Raptopoulou, C. P. & Terzis, A. (2000). *J. Mater. Chem.* **10**, 515–518.
Phan Thanh, S., Renaudin, J. & Maisonneuve, V. (2000). *Solid State Sci.* **2**, 143–148.
Rakovský, E., Žúrková, L. & Marek, J. (2002). *Monatsh. Chem.* **133**, 277–283.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Sun, D., Zhang, H., Zhang, J., Zheng, G., Yu, J. & Gao, S. (2007). *J. Solid State Chem.* **180**, 393–399.
Wang, Z.-L. & Wei, L.-H. (2007). *Acta Cryst.* E**63**, o995–o996.
Wilkinson, H. S. & Harrison, W. T. A. (2007). *Acta Cryst.* E**63**, m902–m904.
Yang, S., Li, G., Tian, S., Liao, F., Xiong, M. & Lin, J. (2007). *J. Solid State Chem.* **180**, 2225–2232.

supplementary materials

Acta Cryst. (2009). E**65**, o1415 [**doi:10.1107/S1600536809019424**]

Hexane-1,6-diammonium bis(pyridine-2-carboxylate)

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Comment

The title compound, $C_6H_{18}N_2^{2+}.2C_6H_4NO_2^-$, consists of a doubly protonated hexamethylenediammonium dication and two pyridinecarboxylate anions (Fig. 1). The carboxylate groups of the anions appear to be delocalized on the basis of the C—O bond lengths (C—O: 1.241 (3)–1.247 (3) Å). The N3—C13—C14—C15 and C16—C17—C18—N4 torsion angles [64.9 (4)° and -66.6 (4)°, respectively] display the *gauche* conformation for the two groups within the dication, whereas C13—C14—C15—C16, C14—C15—C16—C17 and C15—C16—C17—C18 atoms show the anti conformation [their torsion angles lie in the range of 174.6 (3)°–177.3 (3)°]. In the crystal, the component ions interact by means of many intermolecular N—H···O and N—H···N hydrogen bonds and C—H···O contacts to form a 2-D array (Table 1 and Fig. 2).

Experimental

A solution of 1,6-diaminohexane (0.200 g, 1.721 mmol) and pyridine-2-carboxylic acid (1.180 g, 8.606 mmol) in H_2O (20 ml) was stirred for 2 h at 333 K. The solvent was removed under vacuum and the residue was washed with acetone to give a white powder (0.5972 g). Crystals were obtained by slow evaporation from an ethanol solution.

Refinement

All H atoms were located from Fourier difference maps and refined isotropically; C—H = 0.96 (3)–1.13 (3) Å and N—H = 0.92 (3)–1.07 (3) Å.

Figures

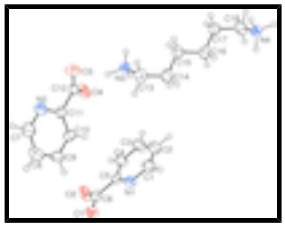

Fig. 1. The molecular structures of the components (I), with displacement ellipsoids drawn at the 30% probability level for non-H atoms.

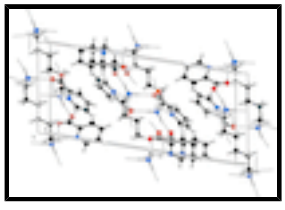

Fig. 2. View of the unit-cell contents for (I). Hydrogen-bond interactions are drawn with dashed lines.

Hexane-1,6-diammonium bis(pyridine-2-carboxylate)

Crystal data

$C_6H_{18}N_2^{2+}\cdot 2C_6H_4NO_2^-$	F_{000} = 776
M_r = 362.43	D_x = 1.253 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1472 reflections
a = 9.8182 (7) Å	θ = 2.6–24.0º
b = 9.1569 (7) Å	μ = 0.09 mm^{-1}
c = 21.6423 (17) Å	T = 296 K
β = 99.038 (2)º	Block, colourless
V = 1921.6 (3) Å^3	0.33 × 0.25 × 0.18 mm
Z = 4	

Data collection

Bruker SMART 1000 CCD diffractometer	4752 independent reflections
Radiation source: fine-focus sealed tube	1740 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.089
T = 296 K	θ_{max} = 28.3º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −13→13
T_{min} = 0.685, T_{max} = 0.984	k = −12→12
13964 measured reflections	l = −21→28

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	All H-atom parameters refined
$wR(F^2)$ = 0.124	$w = 1/[\sigma^2(F_o^2) + (0.0318P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.93	$(\Delta/\sigma)_{max} < 0.001$
4752 reflections	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
339 parameters	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.39197 (19)	0.3983 (2)	0.43294 (9)	0.0607 (6)
O2	0.3440 (2)	0.1705 (2)	0.40142 (11)	0.0809 (7)
N1	0.5671 (2)	0.4287 (2)	0.34905 (10)	0.0514 (6)
C1	0.6534 (3)	0.4405 (4)	0.30699 (15)	0.0632 (9)
H1	0.704 (3)	0.538 (3)	0.3085 (12)	0.078 (10)*
C2	0.6711 (3)	0.3335 (4)	0.26461 (16)	0.0684 (9)
H2	0.739 (3)	0.351 (3)	0.2336 (14)	0.100 (11)*
C3	0.5975 (4)	0.2072 (4)	0.26545 (16)	0.0702 (10)
H3	0.605 (3)	0.128 (3)	0.2356 (13)	0.080 (10)*
C4	0.5110 (3)	0.1905 (3)	0.30952 (14)	0.0586 (8)
H4	0.458 (2)	0.099 (3)	0.3141 (12)	0.061 (8)*
C5	0.4980 (3)	0.3030 (3)	0.35030 (12)	0.0428 (6)
C6	0.4034 (3)	0.2901 (3)	0.39927 (14)	0.0508 (7)
O3	0.1328 (2)	0.4425 (2)	0.07130 (9)	0.0654 (6)
O4	0.2162 (2)	0.2478 (2)	0.12601 (10)	0.0760 (7)
N2	0.0026 (2)	0.5187 (3)	0.16852 (11)	0.0617 (7)
C7	−0.0394 (4)	0.5760 (4)	0.21915 (18)	0.0749 (10)
H7	−0.127 (3)	0.630 (3)	0.2088 (13)	0.084 (10)*
C8	0.0265 (4)	0.5565 (4)	0.27899 (17)	0.0718 (10)
H8	−0.010 (3)	0.603 (3)	0.3131 (14)	0.080 (10)*
C9	0.1416 (4)	0.4708 (4)	0.28807 (15)	0.0653 (9)
H9	0.195 (3)	0.456 (3)	0.3287 (12)	0.066 (9)*
C10	0.1851 (3)	0.4064 (3)	0.23713 (14)	0.0545 (8)
H10	0.268 (3)	0.344 (3)	0.2409 (13)	0.083 (10)*
C11	0.1141 (3)	0.4333 (3)	0.17813 (13)	0.0443 (7)
C12	0.1582 (3)	0.3693 (3)	0.12002 (14)	0.0479 (7)
N3	0.4265 (3)	0.1748 (3)	0.06174 (13)	0.0505 (7)
H3A	0.334 (3)	0.200 (3)	0.0800 (14)	0.114 (12)*
H3B	0.467 (3)	0.090 (3)	0.0830 (15)	0.095 (12)*
H3C	0.415 (3)	0.154 (3)	0.0194 (16)	0.092 (12)*
N4	1.1032 (3)	0.3355 (3)	−0.04832 (13)	0.0487 (6)
H4A	1.046 (3)	0.403 (3)	−0.0754 (12)	0.068 (10)*
H4B	1.117 (3)	0.372 (3)	−0.0007 (16)	0.099 (11)*

H4C	1.196 (4)	0.333 (3)	−0.0672 (15)	0.126 (13)*
C13	0.5150 (3)	0.3063 (4)	0.07920 (18)	0.0655 (9)
H13A	0.500 (3)	0.334 (3)	0.1259 (15)	0.101 (11)*
H13B	0.475 (3)	0.388 (3)	0.0459 (14)	0.099 (11)*
C14	0.6652 (4)	0.2789 (4)	0.07806 (17)	0.0676 (10)
H14A	0.718 (3)	0.372 (3)	0.0970 (13)	0.086 (10)*
H14B	0.697 (3)	0.194 (3)	0.1038 (14)	0.090 (12)*
C15	0.6995 (3)	0.2449 (4)	0.01361 (16)	0.0600 (9)
H15A	0.654 (3)	0.150 (3)	−0.0029 (13)	0.073 (9)*
H15B	0.654 (3)	0.336 (3)	−0.0184 (14)	0.100 (11)*
C16	0.8544 (3)	0.2304 (4)	0.01326 (16)	0.0578 (8)
H16A	0.895 (3)	0.153 (3)	0.0455 (12)	0.072 (9)*
H16B	0.898 (2)	0.328 (3)	0.0250 (12)	0.070 (9)*
C17	0.8877 (3)	0.1893 (4)	−0.05053 (16)	0.0623 (9)
H17A	0.841 (3)	0.261 (3)	−0.0839 (13)	0.075 (10)*
H17B	0.851 (3)	0.083 (3)	−0.0641 (14)	0.103 (11)*
C18	1.0393 (4)	0.1871 (4)	−0.05523 (19)	0.0660 (9)
H18A	1.054 (3)	0.150 (3)	−0.1001 (15)	0.110 (12)*
H18B	1.088 (3)	0.123 (3)	−0.0212 (15)	0.102 (13)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0655 (13)	0.0674 (13)	0.0506 (12)	0.0062 (11)	0.0135 (11)	−0.0096 (11)
O2	0.0771 (15)	0.0718 (15)	0.1046 (19)	−0.0218 (12)	0.0482 (14)	−0.0166 (14)
N1	0.0577 (15)	0.0545 (15)	0.0435 (14)	−0.0030 (12)	0.0132 (12)	−0.0014 (12)
C1	0.077 (2)	0.062 (2)	0.053 (2)	−0.0093 (19)	0.0165 (19)	−0.0003 (19)
C2	0.069 (2)	0.079 (3)	0.062 (2)	−0.006 (2)	0.0240 (19)	−0.007 (2)
C3	0.083 (2)	0.071 (2)	0.061 (2)	0.008 (2)	0.026 (2)	−0.019 (2)
C4	0.065 (2)	0.0547 (19)	0.058 (2)	−0.0009 (17)	0.0143 (17)	−0.0086 (18)
C5	0.0387 (15)	0.0484 (16)	0.0397 (16)	0.0027 (14)	0.0009 (13)	0.0009 (15)
C6	0.0406 (17)	0.0568 (19)	0.0536 (19)	0.0032 (16)	0.0037 (15)	−0.0004 (17)
O3	0.0893 (16)	0.0646 (13)	0.0413 (12)	0.0046 (11)	0.0075 (11)	0.0000 (12)
O4	0.0864 (16)	0.0717 (14)	0.0775 (16)	0.0331 (13)	0.0361 (13)	0.0103 (13)
N2	0.0638 (17)	0.0723 (17)	0.0496 (16)	0.0235 (14)	0.0108 (14)	0.0054 (14)
C7	0.076 (3)	0.083 (3)	0.066 (2)	0.031 (2)	0.014 (2)	0.005 (2)
C8	0.088 (3)	0.073 (2)	0.060 (2)	0.010 (2)	0.029 (2)	−0.002 (2)
C9	0.077 (3)	0.077 (2)	0.041 (2)	−0.001 (2)	0.0073 (19)	0.007 (2)
C10	0.0522 (19)	0.062 (2)	0.0489 (19)	0.0088 (16)	0.0048 (17)	0.0112 (18)
C11	0.0437 (17)	0.0441 (16)	0.0447 (17)	0.0016 (14)	0.0055 (14)	0.0061 (15)
C12	0.0424 (17)	0.0504 (18)	0.0510 (19)	−0.0023 (14)	0.0073 (15)	0.0037 (16)
N3	0.0516 (16)	0.0521 (16)	0.0487 (17)	0.0030 (14)	0.0111 (14)	0.0051 (15)
N4	0.0480 (15)	0.0531 (16)	0.0445 (15)	0.0048 (13)	0.0060 (14)	0.0050 (14)
C13	0.066 (2)	0.055 (2)	0.079 (3)	−0.0051 (18)	0.021 (2)	−0.008 (2)
C14	0.063 (2)	0.070 (2)	0.072 (3)	−0.010 (2)	0.016 (2)	−0.013 (2)
C15	0.052 (2)	0.066 (2)	0.063 (2)	−0.0077 (18)	0.0128 (18)	0.000 (2)
C16	0.052 (2)	0.058 (2)	0.064 (2)	−0.0051 (17)	0.0086 (17)	0.0004 (19)
C17	0.059 (2)	0.069 (2)	0.061 (2)	−0.0086 (19)	0.0140 (18)	−0.008 (2)

C18	0.069 (2)	0.060 (2)	0.074 (3)	−0.0004 (19)	0.025 (2)	−0.010 (2)

Geometric parameters (Å, °)

O1—C6	1.245 (3)	N3—C13	1.498 (4)
O2—C6	1.245 (3)	N3—H3A	1.07 (3)
N1—C5	1.339 (3)	N3—H3B	0.96 (3)
N1—C1	1.341 (3)	N3—H3C	0.92 (3)
C1—C2	1.371 (4)	N4—C18	1.495 (4)
C1—H1	1.02 (3)	N4—H4A	0.97 (3)
C2—C3	1.365 (4)	N4—H4B	1.07 (3)
C2—H2	1.03 (3)	N4—H4C	1.05 (3)
C3—C4	1.381 (4)	C13—C14	1.500 (4)
C3—H3	0.98 (3)	C13—H13A	1.08 (3)
C4—C5	1.375 (3)	C13—H13B	1.07 (3)
C4—H4	1.00 (2)	C14—C15	1.518 (4)
C5—C6	1.520 (3)	C14—H14A	1.05 (3)
O3—C12	1.241 (3)	C14—H14B	0.98 (3)
O4—C12	1.247 (3)	C15—C16	1.528 (4)
N2—C11	1.335 (3)	C15—H15A	1.01 (3)
N2—C7	1.337 (4)	C15—H15B	1.13 (3)
C7—C8	1.365 (4)	C16—C17	1.515 (4)
C7—H7	0.99 (3)	C16—H16A	1.03 (3)
C8—C9	1.365 (4)	C16—H16B	1.01 (3)
C8—H8	0.97 (3)	C17—C18	1.508 (4)
C9—C10	1.376 (4)	C17—H17A	1.03 (3)
C9—H9	0.96 (3)	C17—H17B	1.06 (3)
C10—C11	1.378 (4)	C18—H18A	1.06 (3)
C10—H10	0.99 (3)	C18—H18B	1.01 (3)
C11—C12	1.512 (4)		
C5—N1—C1	117.3 (3)	H3B—N3—H3C	107 (3)
N1—C1—C2	123.8 (3)	C18—N4—H4A	108.7 (15)
N1—C1—H1	114.0 (15)	C18—N4—H4B	111.5 (16)
C2—C1—H1	122.2 (15)	H4A—N4—H4B	111 (2)
C3—C2—C1	118.2 (3)	C18—N4—H4C	108.3 (18)
C3—C2—H2	122.6 (17)	H4A—N4—H4C	104 (2)
C1—C2—H2	119.1 (17)	H4B—N4—H4C	113 (2)
C2—C3—C4	119.1 (3)	N3—C13—C14	113.3 (3)
C2—C3—H3	121.3 (16)	N3—C13—H13A	105.3 (16)
C4—C3—H3	119.6 (16)	C14—C13—H13A	109.7 (16)
C5—C4—C3	119.3 (3)	N3—C13—H13B	104.9 (16)
C5—C4—H4	117.4 (15)	C14—C13—H13B	111.3 (16)
C3—C4—H4	123.3 (15)	H13A—C13—H13B	112 (2)
N1—C5—C4	122.2 (3)	C13—C14—C15	114.2 (3)
N1—C5—C6	116.6 (2)	C13—C14—H14A	106.5 (15)
C4—C5—C6	121.2 (3)	C15—C14—H14A	111.1 (15)
O2—C6—O1	126.3 (3)	C13—C14—H14B	110.6 (18)
O2—C6—C5	115.8 (3)	C15—C14—H14B	105.3 (18)
O1—C6—C5	117.9 (3)	H14A—C14—H14B	109 (2)

C11—N2—C7	116.9 (3)	C14—C15—C16	112.8 (3)
N2—C7—C8	124.4 (3)	C14—C15—H15A	110.5 (15)
N2—C7—H7	112.3 (17)	C16—C15—H15A	108.0 (15)
C8—C7—H7	123.2 (17)	C14—C15—H15B	106.8 (15)
C9—C8—C7	118.0 (3)	C16—C15—H15B	111.0 (14)
C9—C8—H8	122.6 (17)	H15A—C15—H15B	108 (2)
C7—C8—H8	119.4 (17)	C17—C16—C15	112.5 (3)
C8—C9—C10	119.0 (3)	C17—C16—H16A	109.2 (14)
C8—C9—H9	122.0 (16)	C15—C16—H16A	109.9 (14)
C10—C9—H9	118.9 (16)	C17—C16—H16B	107.8 (15)
C9—C10—C11	119.4 (3)	C15—C16—H16B	107.9 (14)
C9—C10—H10	122.4 (17)	H16A—C16—H16B	110 (2)
C11—C10—H10	118.2 (17)	C18—C17—C16	114.9 (3)
N2—C11—C10	122.2 (3)	C18—C17—H17A	107.2 (15)
N2—C11—C12	115.7 (3)	C16—C17—H17A	110.3 (15)
C10—C11—C12	122.2 (3)	C18—C17—H17B	105.3 (16)
O3—C12—O4	126.7 (3)	C16—C17—H17B	111.6 (16)
O3—C12—C11	116.8 (3)	H17A—C17—H17B	107 (2)
O4—C12—C11	116.5 (3)	N4—C18—C17	112.6 (3)
C13—N3—H3A	103.1 (16)	N4—C18—H18A	105.7 (17)
C13—N3—H3B	110.6 (18)	C17—C18—H18A	110.1 (17)
H3A—N3—H3B	108 (2)	N4—C18—H18B	108.5 (18)
C13—N3—H3C	113.2 (19)	C17—C18—H18B	108.7 (18)
H3A—N3—H3C	115 (3)	H18A—C18—H18B	111 (2)
C5—N1—C1—C2	2.0 (4)	C8—C9—C10—C11	1.9 (5)
N1—C1—C2—C3	−0.5 (5)	C7—N2—C11—C10	−1.1 (4)
C1—C2—C3—C4	−1.4 (5)	C7—N2—C11—C12	179.5 (3)
C2—C3—C4—C5	1.8 (5)	C9—C10—C11—N2	−0.9 (4)
C1—N1—C5—C4	−1.5 (4)	C9—C10—C11—C12	178.5 (3)
C1—N1—C5—C6	178.6 (2)	N2—C11—C12—O3	30.2 (3)
C3—C4—C5—N1	−0.3 (4)	C10—C11—C12—O3	−149.2 (3)
C3—C4—C5—C6	179.6 (3)	N2—C11—C12—O4	−149.6 (2)
N1—C5—C6—O2	−177.3 (3)	C10—C11—C12—O4	31.0 (4)
C4—C5—C6—O2	2.8 (4)	N3—C13—C14—C15	64.9 (4)
N1—C5—C6—O1	2.9 (4)	C13—C14—C15—C16	175.1 (3)
C4—C5—C6—O1	−177.0 (3)	C14—C15—C16—C17	177.3 (3)
C11—N2—C7—C8	2.2 (5)	C15—C16—C17—C18	174.6 (3)
N2—C7—C8—C9	−1.2 (6)	C16—C17—C18—N4	−66.6 (4)
C7—C8—C9—C10	−0.9 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···O4	1.07 (3)	1.70 (3)	2.747 (3)	165 (3)
N3—H3B···O1^{i}	0.96 (3)	2.29 (3)	3.088 (3)	140 (2)
N3—H3B···N1^{i}	0.96 (3)	2.15 (3)	2.962 (3)	142 (2)
N3—H3C···O1^{ii}	0.92 (3)	1.91 (3)	2.835 (3)	177 (3)
N4—H4A···O3^{iii}	0.97 (3)	2.27 (3)	3.064 (3)	139 (2)

N4—H4A···N2^{iii}	0.97 (3)	2.12 (3)	2.963 (3)	144 (2)
N4—H4B···O3^{iv}	1.07 (3)	1.67 (3)	2.740 (3)	175 (3)
N4—H4C···O2^{v}	1.05 (3)	1.70 (4)	2.754 (3)	179 (3)
C1—H1···O4^{vi}	1.02 (3)	2.45 (3)	3.328 (4)	145 (2)
C16—H16B···O3^{iv}	1.01 (3)	2.58 (3)	3.426 (4)	140.8 (18)

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1/2; (ii) *x*, −*y*+1/2, *z*−1/2; (iii) −*x*+1, −*y*+1, −*z*; (iv) *x*+1, *y*, *z*; (v) *x*+1, −*y*+1/2, *z*−1/2; (vi) −*x*+1, *y*+1/2, −*z*+1/2.

Fig. 1

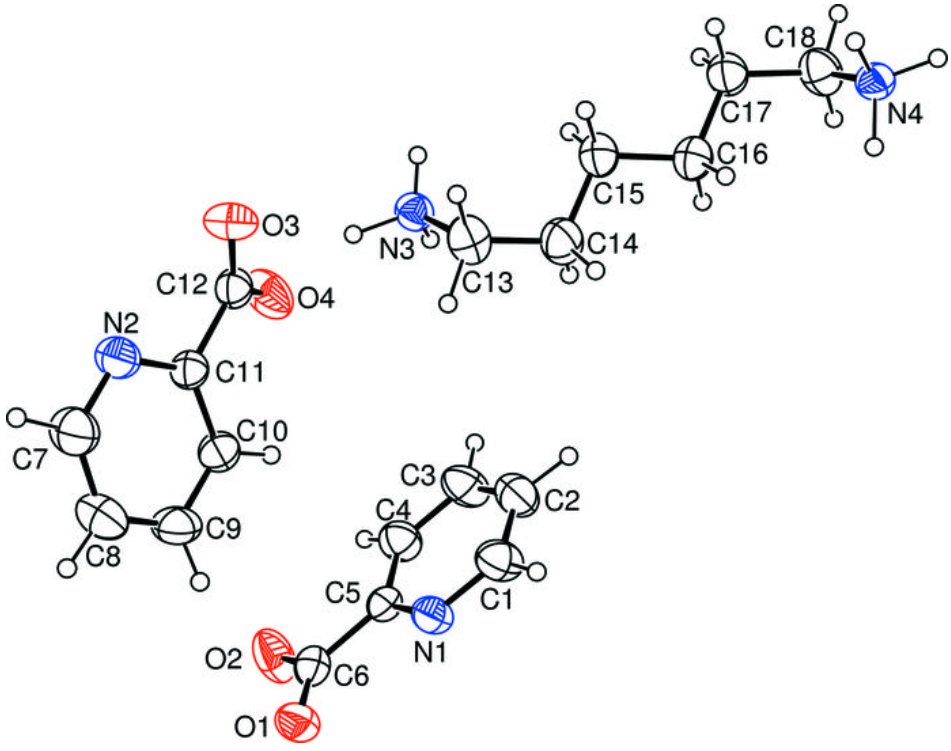
C18
N4
C17
C16
C15
C14
N3
C13
O3
C12
O4
N2
C11
C10
C7
C9
C8
C3
C2
C4
C5
C1
N1
O2
C6
O1

Fig. 2