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Propane-1,3-diaminium bis(tetrafluoroborate)–18-crown-6 (1/2)

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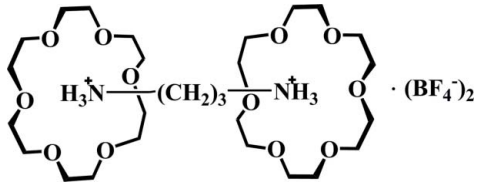
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; disorder in solvent or counterion; R factor = 0.077; wR factor = 0.212; data-to-parameter ratio = 12.7.

In the title compound, $C_3H_{12}N_2^{2+}\cdot 2BF_4^-\cdot 2C_{12}H_{24}O_6$, the central C atom of the propane-1,3-diammonium cation lies on a crystallographic twofold rotation axis. The terminal NH_3^+ groups insert into the crown rings through strong N—H···O hydrogen-bonding interactions, resulting in the formation of a 1:2 supramolecular $[(C_3H_{12}N_2)\cdot(C_{12}H_{24}O_6)_2]^{2+}$ complex. The anions are linked to the supramolecular complexes *via* weak C—H···F hydrogen bonds. The F atoms of the anion are disordered over two orientations with site occupancies of 0.5.

Related literature

For the structures and properties of a related compounds, see: Fu *et al.* (2011); Zhao (2012) and references therein.



Experimental

Crystal data

$C_3H_{12}N_2^{2+}\cdot 2BF_4^-\cdot 2C_{12}H_{24}O_6$
M_r = 778.39
Monoclinic, $C2/c$
a = 22.615 (5) Å
b = 8.8423 (18) Å
c = 21.077 (4) Å
β = 113.41 (3)°
V = 3867.8 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 K
0.10 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.910, T_{max} = 1.000
16100 measured reflections
3413 independent reflections
2018 reflections with $I > 2\sigma(I)$
R_{int} = 0.079

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.212
S = 1.13
3413 reflections
268 parameters
37 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O2	0.89	2.06	2.915 (3)	161
N1—H1A···O4	0.89	2.03	2.911 (4)	169
N1—H1C···O6	0.89	2.08	2.967 (4)	179
C12—H12B···F4′	0.97	2.48	3.316 (19)	144
C13—H13A···F2	0.97	2.47	3.346 (12)	150
C13—H13A···F2′	0.97	2.42	3.361 (16)	162
C5—H5A···F3′^{i}	0.97	2.41	3.350 (18)	162
C10—H10B···F1′^{ii}	0.97	2.41	3.355 (17)	166
C10—H10B···F2′^{ii}	0.97	2.44	3.235 (19)	139
C8—H8A···F3′^{iii}	0.97	2.50	3.412 (14)	156

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2697).

References

Fu, D.-W., Zhang, W., Cai, H.-L., Zhang, Y., Ge, J.-Z., Xiong, R.-G. & Huang, S. P. D. (2011). *J. Am. Chem. Soc.* **133**, 12780–12786.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhao, M.-M. (2012). *Acta Cryst.* E**68**, o327.

supporting information

Acta Cryst. (2012). E**68**, o444 [doi:10.1107/S1600536812001572]

Propane-1,3-diaminium bis(tetrafluoroborate)–18-crown-6 (1/2)

Min-Min Zhao

S1. Comment

As a continuation of the research project devoted to the synthesis and characterization of novel phase transition crystals of amino compounds (Zhao, 2012), the crystal structure of the title compound is reported herein.

The title compound is composed of $[(C_3H_{12}N_2).(C_{12}H_{24}O_6)_2]^{2+}$ cations and BF_4^- anions (Fig. 1). A 1:2 supramolecular rotator-stator structure is generated between one propane-1,3-diammonium dication and two 18-crown-6 molecules through six N—H···O hydrogen bonds (Table 1) occurring between the protons of the NH_3^+ groups and the O atoms of the crown ethers. The supramolecular rotator has crystallographically imposed twofold rotation symmetry, the central C atom of the propane-1,3-diammonium cation lying on a crystallographic twofold rotation axis. The macrocycle adopts a conformation with approximate D_{3d} symmetry, with all O-C-C-O torsion angles being *gauche* and alternating in sign, and all C-O-C-C torsion angles being *trans*. The C–N bonds of the cation are almost perpendicular to the mean planes of the oxygen atoms of the crown ethers. The supramolecular structure is introduced as counter cation to BF_4^- anions. The B atom has a flattened tetrahedral coordination geometry provided by four F atoms [range of *cis*-bond angles = 121.6 (5)-89.1 (9) °; dav(F-B) = 1.226 (9)-1.445 (9)Å]. All F atoms of the BF_4^- anion are disordered over two orientations. In the crystal structure (Fig. 2), cations and anions are linked by weak interionic C—H···F hydrogen bonds (Table 1).

S2. Experimental

Commercial 18-crown-6 (6 mmol), HBF_4 (6 mmol) and propane-1,3-diamine (3 mmol) were dissolved in a water/EtOH (1:1 *v*/*v*) solution. The solvent was slowly evaporated in air affording colourless block-shaped crystals of the title compound suitable for X-ray analysis.

The dielectric constant of the title compound as a function of temperature indicates that the permittivity is basically temperature-independent, suggesting that this compound should be not a real ferroelectrics or there may be no distinct phase transition occurred within the measured temperature range. Similarly, below the melting point (412 K) of the compound, the dielectric constant as a function of temperature also goes smoothly, and there is no dielectric anomaly observed (dielectric constant ranging from 4.5 to 8.8).

S3. Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C). The positional parameters of the N-bound H atoms were intially refined freely, subsequently they were restrained using a N–H distance of 0.89 (2) Å, and in the final refinements treated as riding with U_{iso}(H) = 1.5U_{eq}(N). All F atoms were disordered over two sites with occupancies of 0.5, and were refined anisotropically using ADP restraints (SIMU and DELU).

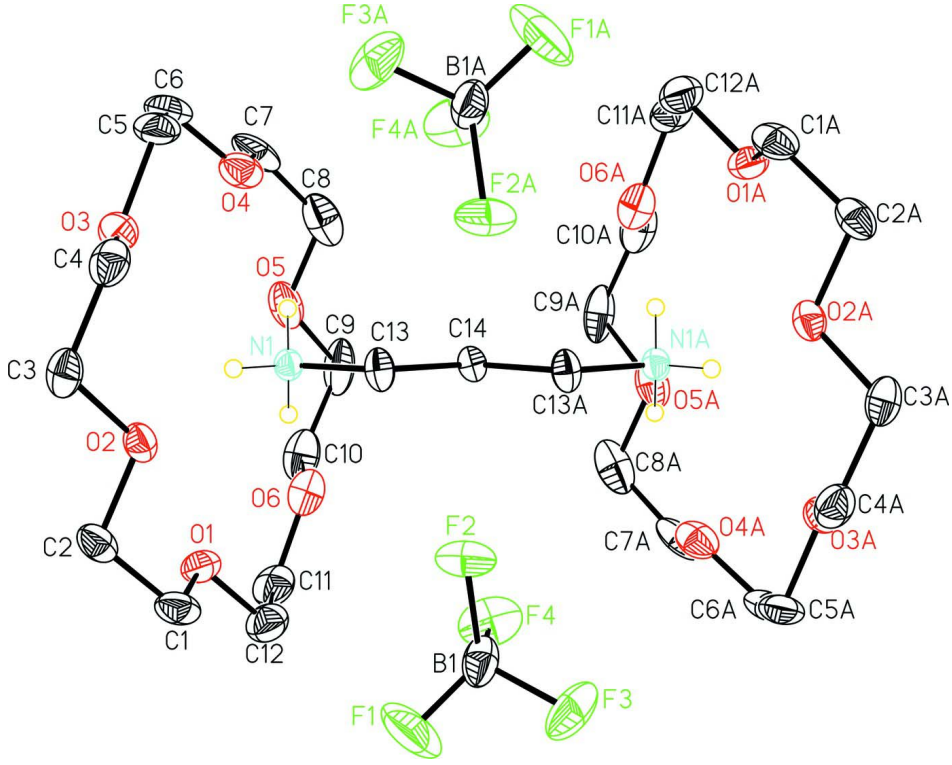

Figure 1

The structure of the title compound with displacement ellipsoids drawn at the 30% probability level. Only H atoms of the NH_3^+ groups are shown. Symmetry code: (A) -x, +y, 1/2-z.

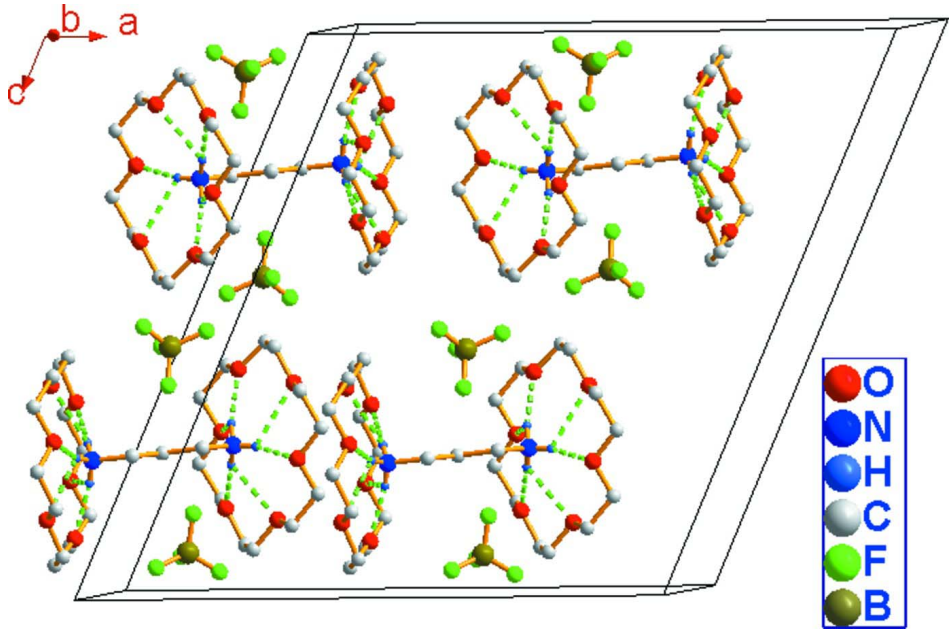

Figure 2

Crystal packing of the title compound approximately viewed along the *b* axis. Only hydrogen atoms involved in N—H···O hydrogen bonds (dashed line) are shown.

Propane-1,3-diaminium bis(tetrafluoroborate)–18-crown-6 (1/2)

Crystal data

$C_3H_{12}N_2^{2+}\cdot 2BF_4^-\cdot 2C_{12}H_{24}O_6$
$M_r = 778.39$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 22.615\ (5)$ Å
$b = 8.8423\ (18)$ Å
$c = 21.077\ (4)$ Å
$\beta = 113.41\ (3)°$
$V = 3867.8\ (16)$ Å^3
$Z = 4$
$F(000) = 1656$
$D_x = 1.337$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3410 reflections
$\theta = 2.9–27.5°$
$\mu = 0.12$ mm^{-1}
$T = 298$ K
Block, colourless
0.10 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD profile fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.910$, $T_{max} = 1.000$
16100 measured reflections
3413 independent reflections
2018 reflections with $I > 2\sigma(I)$
$R_{int} = 0.079$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -26\rightarrow 26$
$k = -10\rightarrow 10$
$l = -25\rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.077$
$wR(F^2) = 0.212$
$S = 1.13$
3413 reflections
268 parameters
37 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.080P)^2 + 3.2068P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-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.

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
O2	0.19727 (10)	0.6715 (3)	0.26770 (12)	0.0558 (6)	
O3	0.15021 (11)	0.7740 (3)	0.12991 (13)	0.0650 (7)	
N1	0.10547 (11)	0.9201 (3)	0.23703 (13)	0.0433 (7)	
H1A	0.0918	0.9641	0.1957	0.065*	

H1B	0.1379	0.8578	0.2419	0.065*
H1C	0.1189	0.9907	0.2698	0.065*
O1	0.22029 (12)	0.8928 (3)	0.37179 (12)	0.0646 (7)
O4	0.07376 (13)	1.0406 (3)	0.09880 (15)	0.0786 (9)
O5	0.10078 (13)	1.2567 (3)	0.20532 (18)	0.0850 (9)
O6	0.14972 (14)	1.1589 (3)	0.34468 (17)	0.0771 (8)
C2	0.25128 (17)	0.6683 (4)	0.3313 (2)	0.0652 (11)
H2A	0.2862	0.7248	0.3269	0.078*
H2B	0.2654	0.5647	0.3431	0.078*
C3	0.21010 (19)	0.5983 (4)	0.2141 (2)	0.0652 (11)
H3A	0.2180	0.4915	0.2244	0.078*
H3B	0.2483	0.6418	0.2112	0.078*
C6	0.0933 (2)	0.9686 (6)	0.0513 (2)	0.0876 (15)
H6A	0.1345	1.0089	0.0555	0.105*
H6B	0.0621	0.9881	0.0047	0.105*
C1	0.2343 (2)	0.7359 (5)	0.3861 (2)	0.0714 (12)
H1D	0.1971	0.6845	0.3879	0.086*
H1E	0.2700	0.7243	0.4306	0.086*
C7	0.0639 (2)	1.2022 (6)	0.0872 (3)	0.0953 (18)
H7A	0.1024	1.2499	0.0868	0.114*
H7B	0.0289	1.2203	0.0427	0.114*
C5	0.0984 (2)	0.8034 (5)	0.06412 (19)	0.0801 (13)
H5A	0.0580	0.7645	0.0633	0.096*
H5B	0.1075	0.7525	0.0282	0.096*
C10	0.1474 (3)	1.3118 (5)	0.3255 (3)	0.0974 (16)
H10A	0.1872	1.3399	0.3215	0.117*
H10B	0.1429	1.3738	0.3613	0.117*
C4	0.1550 (2)	0.6196 (5)	0.1473 (2)	0.0736 (12)
H4A	0.1616	0.5609	0.1118	0.088*
H4B	0.1157	0.5848	0.1509	0.088*
C11	0.2008 (3)	1.1288 (6)	0.4107 (2)	0.0910 (15)
H11A	0.1929	1.1804	0.4472	0.109*
H11B	0.2412	1.1654	0.4106	0.109*
C9	0.0925 (3)	1.3385 (5)	0.2604 (3)	0.0963 (17)
H9A	0.0528	1.3078	0.2641	0.116*
H9B	0.0895	1.4458	0.2499	0.116*
C8	0.0481 (2)	1.2698 (6)	0.1433 (4)	0.1030 (18)
H8A	0.0369	1.3756	0.1333	0.124*
H8B	0.0114	1.2179	0.1462	0.124*
C12	0.2061 (2)	0.9645 (6)	0.4233 (2)	0.0851 (13)
H12A	0.2395	0.9425	0.4684	0.102*
H12B	0.1654	0.9272	0.4224	0.102*
B1	0.0793 (3)	0.6074 (7)	0.4303 (3)	0.0773 (15)
C14	0.0000	0.9285 (5)	0.2500	0.0449 (11)
H14A	0.0184	0.9930	0.2904	0.054*
C13	0.05211 (15)	0.8336 (4)	0.2431 (2)	0.0552 (9)
H13A	0.0699	0.7687	0.2834	0.066*
H13B	0.0330	0.7690	0.2028	0.066*

F2	0.0550 (6)	0.5710 (10)	0.3586 (6)	0.090 (3)	0.50
F3	0.0439 (7)	0.5494 (15)	0.4626 (7)	0.110 (5)	0.50
F4	0.0679 (5)	0.7681 (8)	0.4203 (4)	0.118 (3)	0.50
F1	0.1357 (3)	0.5838 (17)	0.4687 (6)	0.163 (5)	0.50
F1′	0.1185 (8)	0.4723 (15)	0.4546 (6)	0.220 (6)	0.50
F2′	0.0789 (8)	0.5857 (18)	0.3725 (7)	0.172 (7)	0.50
F3′	0.0256 (7)	0.5942 (19)	0.4373 (9)	0.166 (7)	0.50
F4′	0.1152 (8)	0.7196 (18)	0.4632 (7)	0.186 (6)	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0451 (13)	0.0560 (14)	0.0655 (16)	0.0094 (11)	0.0213 (12)	0.0071 (12)
O3	0.0583 (15)	0.0699 (17)	0.0626 (17)	−0.0108 (13)	0.0197 (13)	−0.0076 (13)
N1	0.0392 (14)	0.0452 (15)	0.0476 (15)	0.0051 (12)	0.0196 (12)	0.0064 (12)
O1	0.0721 (17)	0.0758 (17)	0.0456 (14)	−0.0101 (14)	0.0230 (13)	0.0011 (12)
O4	0.0700 (18)	0.088 (2)	0.0635 (18)	−0.0096 (15)	0.0110 (14)	0.0276 (16)
O5	0.0607 (18)	0.0645 (18)	0.128 (3)	0.0193 (14)	0.0355 (18)	0.0222 (17)
O6	0.093 (2)	0.0527 (16)	0.104 (2)	−0.0128 (15)	0.0591 (19)	−0.0153 (15)
C2	0.048 (2)	0.066 (2)	0.071 (3)	0.0021 (18)	0.011 (2)	0.024 (2)
C3	0.074 (3)	0.045 (2)	0.092 (3)	0.0139 (19)	0.049 (2)	0.0021 (19)
C6	0.069 (3)	0.125 (4)	0.045 (2)	−0.035 (3)	−0.002 (2)	0.018 (3)
C1	0.068 (3)	0.074 (3)	0.057 (2)	−0.004 (2)	0.009 (2)	0.027 (2)
C7	0.059 (3)	0.097 (4)	0.088 (3)	−0.019 (2)	−0.015 (2)	0.061 (3)
C5	0.069 (3)	0.112 (4)	0.044 (2)	−0.026 (2)	0.006 (2)	−0.014 (2)
C10	0.113 (4)	0.065 (3)	0.144 (5)	−0.001 (3)	0.083 (4)	−0.027 (3)
C4	0.078 (3)	0.072 (3)	0.081 (3)	−0.009 (2)	0.043 (3)	−0.023 (2)
C11	0.099 (3)	0.111 (4)	0.069 (3)	−0.025 (3)	0.040 (3)	−0.037 (3)
C9	0.100 (4)	0.046 (2)	0.183 (6)	0.021 (3)	0.100 (4)	0.011 (3)
C8	0.066 (3)	0.071 (3)	0.160 (6)	0.007 (2)	0.032 (4)	0.047 (3)
C12	0.087 (3)	0.108 (4)	0.062 (3)	−0.019 (3)	0.032 (2)	−0.012 (3)
B1	0.073 (4)	0.090 (4)	0.087 (4)	−0.020 (3)	0.051 (3)	−0.033 (3)
C14	0.038 (2)	0.038 (2)	0.059 (3)	0.000	0.020 (2)	0.000
C13	0.0443 (18)	0.0430 (18)	0.084 (3)	−0.0031 (16)	0.0320 (18)	−0.0038 (18)
F2	0.118 (7)	0.076 (4)	0.053 (4)	0.018 (4)	0.010 (4)	0.005 (3)
F3	0.155 (12)	0.100 (6)	0.111 (6)	−0.061 (7)	0.091 (7)	−0.012 (4)
F4	0.178 (8)	0.075 (4)	0.099 (5)	−0.020 (4)	0.052 (5)	−0.016 (3)
F1	0.057 (4)	0.207 (10)	0.160 (8)	−0.022 (5)	−0.025 (4)	0.059 (9)
F1′	0.347 (17)	0.195 (9)	0.148 (9)	0.138 (11)	0.131 (10)	0.066 (8)
F2′	0.168 (14)	0.252 (14)	0.091 (8)	0.078 (9)	0.048 (9)	−0.038 (7)
F3′	0.094 (7)	0.203 (15)	0.246 (18)	−0.048 (8)	0.115 (11)	−0.116 (11)
F4′	0.227 (13)	0.193 (9)	0.198 (12)	−0.162 (10)	0.146 (10)	−0.128 (10)

Geometric parameters (Å, º)

O2—C2	1.410 (4)	C10—C9	1.459 (7)
O2—C3	1.429 (4)	C10—H10A	0.9700
O3—C4	1.407 (5)	C10—H10B	0.9700

O3—C5	1.439 (4)	C4—H4A	0.9698
N1—C13	1.477 (4)	C4—H4B	0.9701
N1—H1A	0.8900	C11—C12	1.473 (7)
N1—H1B	0.8900	C11—H11A	0.9699
N1—H1C	0.8900	C11—H11B	0.9699
O1—C12	1.400 (5)	C9—H9A	0.9699
O1—C1	1.428 (5)	C9—H9B	0.9700
O4—C6	1.397 (6)	C8—H8A	0.9700
O4—C7	1.452 (5)	C8—H8B	0.9700
O5—C8	1.380 (6)	C12—H12A	0.9699
O5—C9	1.442 (6)	C12—H12B	0.9700
O6—C10	1.405 (5)	B1—F1	1.226 (9)
O6—C11	1.436 (6)	B1—F2′	1.228 (15)
C2—C1	1.481 (6)	B1—F3′	1.286 (14)
C2—H2A	0.9701	B1—F4′	1.296 (9)
C2—H2B	0.9700	B1—F3	1.342 (11)
C3—C4	1.475 (6)	B1—F2	1.423 (13)
C3—H3A	0.9699	B1—F4	1.445 (9)
C3—H3B	0.9699	B1—F1′	1.454 (11)
C6—C5	1.482 (6)	C14—C13	1.501 (4)
C6—H6A	0.9701	C14—C13^{i}	1.501 (4)
C6—H6B	0.9700	C14—H14A	0.9700
C1—H1D	0.9701	C13—H13A	0.9701
C1—H1E	0.9699	C13—H13B	0.9700
C7—C8	1.490 (7)	F2—F2′	0.52 (3)
C7—H7A	0.9700	F3—F3′	0.66 (2)
C7—H7B	0.9699	F4—F4′	1.174 (18)
C5—H5A	0.9700	F1—F1′	1.058 (15)
C5—H5B	0.9700	F1—F4′	1.275 (16)

C2—O2—C3	111.8 (3)	O5—C9—C10	110.0 (4)
C4—O3—C5	112.0 (3)	O5—C9—H9A	110.2
C13—N1—H1A	109.5	C10—C9—H9A	110.3
C13—N1—H1B	109.5	O5—C9—H9B	109.1
H1A—N1—H1B	109.5	C10—C9—H9B	109.2
C13—N1—H1C	109.5	H9A—C9—H9B	108.0
H1A—N1—H1C	109.5	O5—C8—C7	109.2 (4)
H1B—N1—H1C	109.5	O5—C8—H8A	109.8
C12—O1—C1	112.1 (3)	C7—C8—H8A	109.7
C6—O4—C7	113.5 (4)	O5—C8—H8B	109.8
C8—O5—C9	112.4 (4)	C7—C8—H8B	109.9
C10—O6—C11	112.4 (4)	H8A—C8—H8B	108.4
O2—C2—C1	109.8 (3)	O1—C12—C11	109.5 (4)
O2—C2—H2A	109.3	O1—C12—H12A	109.9
C1—C2—H2A	109.9	C11—C12—H12A	110.5
O2—C2—H2B	109.6	O1—C12—H12B	109.8
C1—C2—H2B	109.9	C11—C12—H12B	108.8
H2A—C2—H2B	108.3	H12A—C12—H12B	108.4

O2—C3—C4	109.6 (3)	F1—B1—F2′	102.9 (11)
O2—C3—H3A	109.9	F1—B1—F3′	133.6 (11)
C4—C3—H3A	110.5	F2′—B1—F3′	117.7 (12)
O2—C3—H3B	109.6	F1—B1—F4′	60.7 (9)
C4—C3—H3B	109.0	F2′—B1—F4′	114.0 (11)
H3A—C3—H3B	108.1	F3′—B1—F4′	115.3 (10)
O4—C6—C5	110.0 (4)	F1—B1—F3	105.8 (9)
O4—C6—H6A	109.5	F2′—B1—F3	132.3 (11)
C5—C6—H6A	110.0	F4′—B1—F3	113.2 (9)
O4—C6—H6B	109.6	F1—B1—F2	121.6 (10)
C5—C6—H6B	109.4	F3′—B1—F2	96.7 (10)
H6A—C6—H6B	108.2	F4′—B1—F2	129.6 (9)
O1—C1—C2	109.4 (3)	F3—B1—F2	113.3 (9)
O1—C1—H1D	109.9	F1—B1—F4	110.0 (8)
C2—C1—H1D	109.9	F2′—B1—F4	94.5 (9)
O1—C1—H1E	109.6	F3′—B1—F4	89.1 (9)
C2—C1—H1E	109.8	F4′—B1—F4	50.4 (8)
H1D—C1—H1E	108.3	F3—B1—F4	110.0 (8)
O4—C7—C8	109.3 (4)	F2—B1—F4	95.5 (6)
O4—C7—H7A	110.5	F1—B1—F1′	45.5 (7)
C8—C7—H7A	109.4	F2′—B1—F1′	89.1 (9)
O4—C7—H7B	109.7	F3′—B1—F1′	110.9 (11)
C8—C7—H7B	109.8	F4′—B1—F1′	106.0 (12)
H7A—C7—H7B	108.1	F3—B1—F1′	84.9 (9)
O3—C5—C6	109.2 (3)	F2—B1—F1′	96.5 (7)
O3—C5—H5A	110.7	F4—B1—F1′	155.2 (9)
C6—C5—H5A	109.9	C13—C14—C13^{i}	112.0 (4)
O3—C5—H5B	108.9	C13—C14—H14A	109.6
C6—C5—H5B	110.0	C13^{i}—C14—H14A	108.8
H5A—C5—H5B	108.2	N1—C13—C14	114.8 (3)
O6—C10—C9	110.3 (4)	N1—C13—H13A	108.3
O6—C10—H10A	110.0	C14—C13—H13A	108.3
C9—C10—H10A	110.4	N1—C13—H13B	108.9
O6—C10—H10B	108.9	C14—C13—H13B	108.7
C9—C10—H10B	109.0	H13A—C13—H13B	107.6
H10A—C10—H10B	108.1	F2′—F2—B1	58 (3)
O3—C4—C3	108.7 (3)	F3′—F3—B1	70.8 (17)
O3—C4—H4A	109.7	F4′—F4—B1	58.2 (6)
C3—C4—H4A	109.7	F1′—F1—B1	78.7 (9)
O3—C4—H4B	110.6	F1′—F1—F4′	140.6 (13)
C3—C4—H4B	109.7	B1—F1—F4′	62.4 (7)
H4A—C4—H4B	108.5	F1—F1′—B1	55.8 (6)
O6—C11—C12	109.5 (4)	F2—F2′—B1	101 (3)
O6—C11—H11A	110.6	F3—F3′—B1	80 (2)
C12—C11—H11A	110.4	F4—F4′—F1	126.9 (10)
O6—C11—H11B	109.4	F4—F4′—B1	71.4 (8)
C12—C11—H11B	108.6	F1—F4′—B1	57.0 (6)
H11A—C11—H11B	108.2		

C3—O2—C2—C1	−176.1 (3)	F4—B1—F1—F1′	−175.3 (10)
C2—O2—C3—C4	−173.6 (3)	F2′—B1—F1—F4′	110.5 (12)
C7—O4—C6—C5	−176.6 (3)	F3′—B1—F1—F4′	−98.1 (14)
C12—O1—C1—C2	−177.8 (3)	F3—B1—F1—F4′	−108.0 (10)
O2—C2—C1—O1	−65.5 (4)	F2—B1—F1—F4′	120.9 (11)
C6—O4—C7—C8	−175.9 (3)	F4—B1—F1—F4′	10.8 (9)
C4—O3—C5—C6	176.1 (4)	F1′—B1—F1—F4′	−173.9 (14)
O4—C6—C5—O3	−66.6 (4)	F4′—F1—F1′—B1	9 (2)
C11—O6—C10—C9	−176.4 (4)	F2′—B1—F1′—F1	109.2 (14)
C5—O3—C4—C3	178.3 (3)	F3′—B1—F1′—F1	−131.3 (15)
O2—C3—C4—O3	66.2 (4)	F4′—B1—F1′—F1	−5.5 (13)
C10—O6—C11—C12	−172.4 (4)	F3—B1—F1′—F1	−118.2 (13)
C8—O5—C9—C10	175.9 (4)	F2—B1—F1′—F1	128.9 (12)
O6—C10—C9—O5	−65.0 (5)	F4—B1—F1′—F1	10 (2)
C9—O5—C8—C7	171.7 (3)	F1—B1—F2′—F2	155 (3)
O4—C7—C8—O5	65.7 (4)	F3′—B1—F2′—F2	−2 (3)
C1—O1—C12—C11	172.4 (3)	F4′—B1—F2′—F2	−142 (3)
O6—C11—C12—O1	60.6 (5)	F3—B1—F2′—F2	29 (3)
C13^{i}—C14—C13—N1	−179.2 (4)	F4—B1—F2′—F2	−94 (3)
F1—B1—F2—F2′	−29 (3)	F1′—B1—F2′—F2	111 (3)
F3′—B1—F2—F2′	178 (3)	F1—B1—F3′—F3	−20 (4)
F4′—B1—F2—F2′	47 (3)	F2′—B1—F3′—F3	128 (3)
F3—B1—F2—F2′	−157 (3)	F4′—B1—F3′—F3	−93 (3)
F4—B1—F2—F2′	88 (3)	F2—B1—F3′—F3	127 (3)
F1′—B1—F2—F2′	−70 (3)	F4—B1—F3′—F3	−137 (3)
F1—B1—F3—F3′	165 (3)	F1′—B1—F3′—F3	28 (3)
F2′—B1—F3—F3′	−70 (3)	B1—F4—F4′—F1	13.8 (11)
F4′—B1—F3—F3′	101 (3)	F1′—F1—F4′—F4	−25 (3)
F2—B1—F3—F3′	−59 (3)	B1—F1—F4′—F4	−15.7 (12)
F4—B1—F3—F3′	46 (3)	F1′—F1—F4′—B1	−9 (2)
F1′—B1—F3—F3′	−154 (3)	F1—B1—F4′—F4	166.8 (11)
F1—B1—F4—F4′	−12.2 (10)	F2′—B1—F4′—F4	75.1 (12)
F2′—B1—F4—F4′	−117.7 (11)	F3′—B1—F4′—F4	−65.6 (13)
F3′—B1—F4—F4′	124.6 (11)	F3—B1—F4′—F4	−97.4 (11)
F3—B1—F4—F4′	104.0 (11)	F2—B1—F4′—F4	58.4 (14)
F2—B1—F4—F4′	−138.8 (9)	F1′—B1—F4′—F4	171.4 (9)
F1′—B1—F4—F4′	−20.1 (19)	F2′—B1—F4′—F1	−91.7 (13)
F2′—B1—F1—F1′	−75.6 (13)	F3′—B1—F4′—F1	127.6 (13)
F3′—B1—F1—F1′	76 (2)	F3—B1—F4′—F1	95.7 (11)
F4′—B1—F1—F1′	173.9 (14)	F2—B1—F4′—F1	−108.4 (14)
F3—B1—F1—F1′	65.9 (14)	F4—B1—F4′—F1	−166.8 (11)
F2—B1—F1—F1′	−65.2 (12)	F1′—B1—F4′—F1	4.5 (11)

Symmetry code: (i) $-x, y, -z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O2	0.89	2.06	2.915 (3)	161
N1—H1*A*···O4	0.89	2.03	2.911 (4)	169
N1—H1*C*···O6	0.89	2.08	2.967 (4)	179
C12—H12*B*···F4′	0.97	2.48	3.316 (19)	144
C13—H13*A*···F2	0.97	2.47	3.346 (12)	150
C13—H13*A*···F2′	0.97	2.42	3.361 (16)	162
C5—H5*A*···F3′^{i}	0.97	2.41	3.350 (18)	162
C10—H10*B*···F1′^{ii}	0.97	2.41	3.355 (17)	166
C10—H10*B*···F2′^{ii}	0.97	2.44	3.235 (19)	139
C8—H8*A*···F3′^{iii}	0.97	2.50	3.412 (14)	156

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