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L-Lysinium trifluoroacetate

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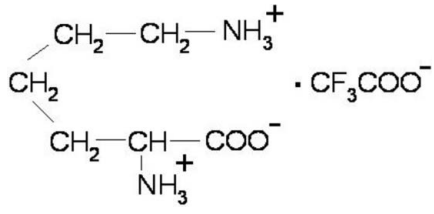
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.045; wR factor = 0.123; data-to-parameter ratio = 7.9.

Ions of the title compound, $C_6H_{15}N_2O_2^+\cdot C_2F_3O_2^-$, a new organic nonlinear optical crystal, are linked by N—H···O hydrogen-bonding interactions. Both the amino groups of the L-lysinium cation are protonated. A three-dimensional network of hydrogen bonds is observed, forming a closed ring. Intermolecular N—H···O hydrogen bonds involving L-lysinium cations and trifluoroacetate anions link the ions into extended chains which run parallel to the [010] direction. The F atoms of the trifluoroacetate anion are disordered over two sites with site occupancies of 0.423 (18) and 0.577 (18). The asymmetric unit consists of two cations and two anions.

Related literature

For related literature, see: Babu, Sethuraman, Gopalakrishnan & Ramasamy (2006); Babu, Sethuraman, Vijayan *et al.* (2006); Chandra *et al.* (1998); Debrus *et al.* (2005); Drozd & Marchewka (2006); Kurtz & Perry (1968); Marchewka *et al.* (2003); Prasad & Vijayan (1993); Pratap *et al.* (2000); Suresh *et al.* (1994); Xu *et al.* (1983); Yokotani *et al.* (1989).



Experimental

Crystal data

$C_6H_{15}N_2O_2^+\cdot C_2F_3O_2^-$
M_r = 260.22
Monoclinic, $P2_1$
a = 5.6985 (2) Å
b = 23.5430 (8) Å
c = 8.5007 (3) Å
β = 91.630 (2)°
V = 1139.99 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 293 (2) K
0.35 × 0.29 × 0.12 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.95, T_{max} = 0.98
8405 measured reflections
2674 independent reflections
2470 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.122
S = 1.06
2674 reflections
340 parameters
43 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 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
N4—H4E···O7^{i}	0.89	1.96	2.838 (4)	168
N4—H4D···O1^{i}	0.89	2.63	3.080 (4)	112
N4—H4D···F2^{i}	0.89	2.54	3.079 (4)	120
N4—H4D···O3^{i}	0.89	2.05	2.842 (4)	147
N3—H3E···O8^{ii}	0.89	1.98	2.866 (4)	172
N3—H3D···O4^{iii}	0.89	1.98	2.864 (4)	171
N3—H3C···O6^{iv}	0.89	2.40	3.148 (4)	142
N3—H3C···O5^{iv}	0.89	2.23	3.064 (4)	157
N2—H2E···O6^{v}	0.89	1.97	2.853 (4)	174
N2—H2D···O3^{vi}	0.89	1.94	2.800 (4)	163
N2—H2C···O4^{vii}	0.89	2.12	2.934 (4)	151
N1—H1E···O5^{iv}	0.89	1.99	2.870 (4)	172
N1—H1C···O7^{i}	0.89	2.52	3.212 (4)	136
N1—H1C···O8^{i}	0.89	2.04	2.901 (4)	163

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2034).

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L-Lysinium trifluoroacetate

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S1. Comment

Over the past two decades, the discovery of promising optical properties in *L*-arginine phosphate monohydrate (LAP) and its deuterated complex has stimulated a strong interest in the nonlinear optcial (NLO) crystals of l-larginine family and other amino acids. (Xu *et al.*, 1983; Yokotani *et al.*, 1989). As one of the three diamino carboxylic acids, l-lysine reacted with other carboxylic acids such as formic, acetic, succinic, glycolic, oxalic and maleic acids has also been studied for its intrinsic polarities (Prasad & Vijayan, 1993; Suresh *et al.*, 1994; Marchewka *et al.*, 2003; Chandra *et al.*, 1998; Pratap *et al.*, 2000). Several NLO crystals composed of l-lysine have been grown and characterized (Babu, Sethuraman, Gopalakrishnan & Ramasamy, 2006; Debrus *et al.*, 2005; Babu, Sethuraman, Vijayan, Bhagavannarayana, Gopalakrishnan & Ramasamy, 2006). Duo to the low UV cutoff (210 nm at 0.1%, *v*/*v*) and effectiveness as an ion-pairing agent, trifluoroacetic acid attracts our attention. Hence, it may be useful to synthesize the amino acid compounds with trifluoroacetic acid and investigate their properties.

In the crystal structure of the title compound, (I) (Fig. 1), both the amino groups in the *L*-lysine$^+$ cations are protonated. All the C—N bonds (Table 1) are typical and bond lengths are somewhat shorter than the respective value of C—NH_3^+ (cal. 1.490 A) of *L*-lysine cations (Drozd & Marchewka, 2006). The C—F bond lengths of C16 are shorter than respective values of C13 due to the positional disorder of fluorin atoms F4, F5 and F6 of trifluoroacetate ion.

The packing of the title compound is shown in Fig. 2. A three-dimensional network of hydrogen bonds connects l-lysine cations and trifluoroacetate anions together. The hydrogen bonds of N—H···O are dominative among the negatively charged carboxylate groups, positively charged protonated amino groups. The introduction of trifluoroacetate anions optimizes the orientation of *L*-lysine through interactions among carboxyl groups and amino groups.

The second harmonic geneartion (SHG) of crystals of (I) was studied by the powder SHG method (Kurtz & Perry, 1968). The green light beam was observed, which confirms its non-centrosymmetric structure.

S2. Experimental

High optical-quality crystals used for X-ray analysis were obtained from an aqueous solution of *L*-lysine and trifluoroacetate acid, mixed in 1:1 molar ratio, after several days at 313 K.

S3. Refinement

The Flack parameter was inconclusive due to the lack of significant anomalous scatterer. The F atoms of the CF_3 group are probably disordered.

All atoms of the disordered group were refined with restrained bond distance and displacemens to improve convergence. Occupancy of both positions of disordered group was refined and converged to 0.577 (18) and 0.423 (18) respectively. H atoms attached to C and N atoms were positioned geometrically and treated as riding, with N—H = 0.89%A and C—H = 0.97 or 0.98%A, and their isotropic displacement parameters were set to 1.2 times the equivalent

displacement parameter of their parent atoms.

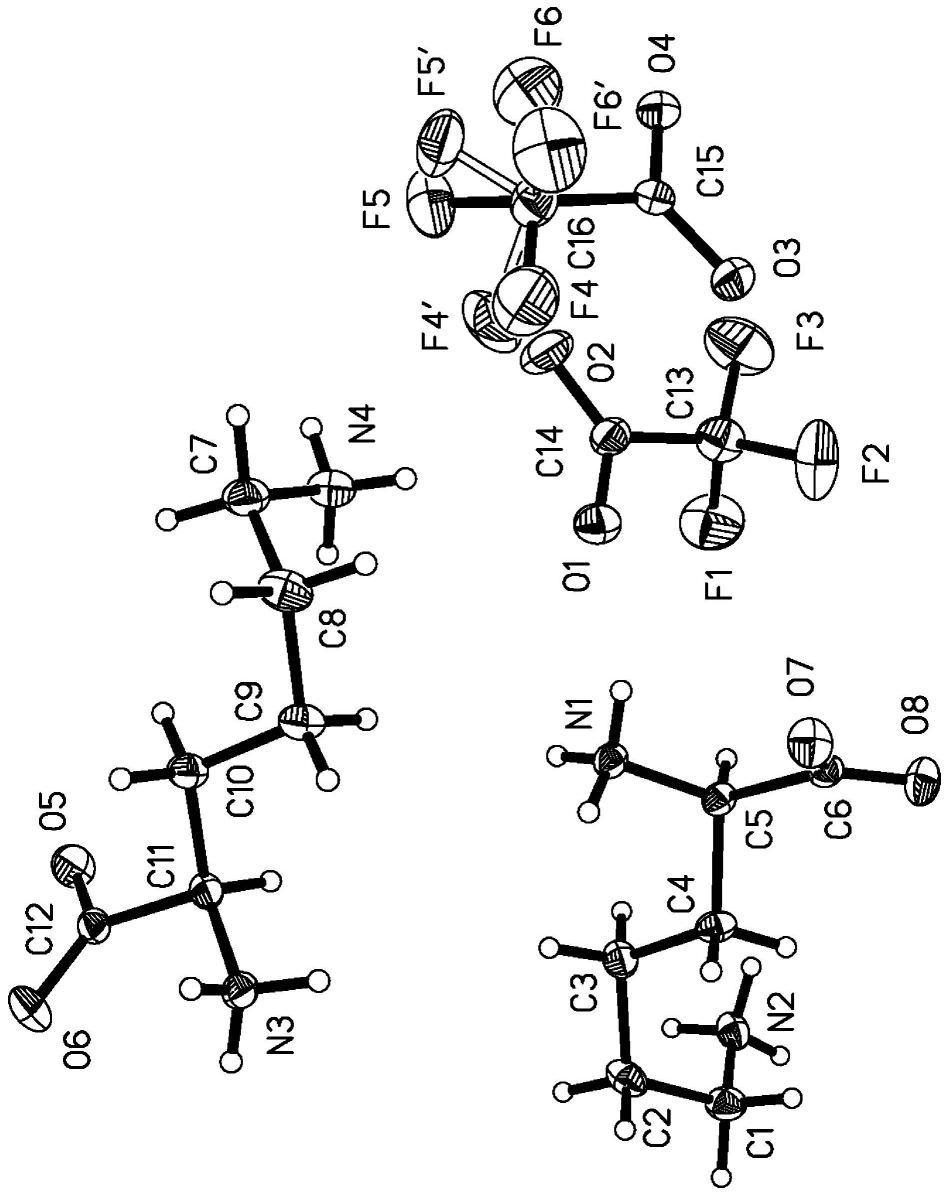

Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids.

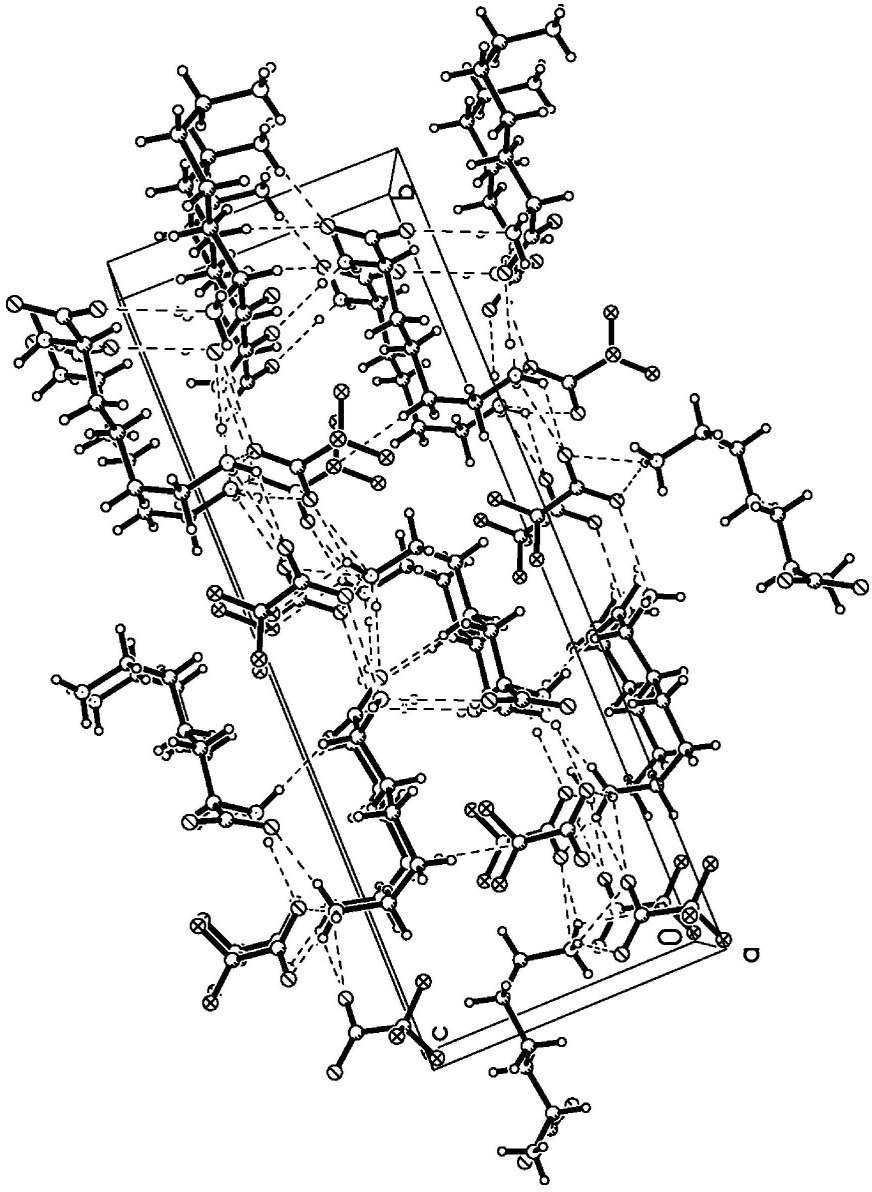

Figure 2

The crystal packing of (I), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

L-Lysinium trifluoroacetate

Crystal data

$C_6H_{15}N_2O_2^+\cdot C_2F_3O_2^-$
$M_r = 260.22$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 5.6985\ (2)$ Å
$b = 23.5430\ (8)$ Å
$c = 8.5007\ (3)$ Å
$\beta = 91.630\ (2)°$
$V = 1139.99\ (7)$ Å^3
$Z = 4$
$F(000) = 544$
$D_x = 1.516$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3919 reflections
$\theta = 3.0–27.3°$
$\mu = 0.15$ mm^{-1}
$T = 293$ K
Prism, colourless
0.35 × 0.29 × 0.12 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.95, T_{max} = 0.98
8405 measured reflections
2674 independent reflections
2470 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.5°, θ_{min} = 1.7°
$h = -5 \rightarrow 7$
$k = -30 \rightarrow 25$
$l = -11 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.122
S = 1.07
2674 reflections
340 parameters
43 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
$w = 1/[\sigma^2(F_o^2) + (0.0705P)^2 + 0.3205P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.017 (4)

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}	Occ. (<1)
C1	0.2035 (7)	0.19654 (15)	0.9377 (4)	0.0386 (7)	
H1A	0.0417	0.2088	0.9236	0.046*	
H1B	0.2078	0.1557	0.9233	0.046*	
C2	0.3517 (7)	0.22473 (15)	0.8138 (4)	0.0394 (8)	
H2A	0.2868	0.2149	0.7106	0.047*	
H2B	0.5091	0.2091	0.8218	0.047*	
C3	0.3684 (6)	0.28929 (15)	0.8256 (4)	0.0340 (7)	
H3A	0.4339	0.2994	0.9284	0.041*	
H3B	0.4753	0.3029	0.7473	0.041*	
C4	0.1328 (6)	0.31898 (14)	0.8015 (4)	0.0320 (7)	
H4A	0.0210	0.3020	0.8715	0.038*	
H4B	0.0763	0.3124	0.6944	0.038*	
C5	0.1405 (5)	0.38324 (12)	0.8314 (3)	0.0260 (6)	
H5	0.1926	0.3898	0.9408	0.031*	
C6	−0.1062 (5)	0.40846 (13)	0.8071 (4)	0.0274 (6)	

C7	0.8434 (7)	0.56316 (15)	0.4189 (4)	0.0417 (8)	
H7A	1.0015	0.5512	0.3955	0.050*	
H7B	0.8324	0.6036	0.3988	0.050*	
C8	0.6728 (7)	0.53266 (16)	0.3112 (4)	0.0415 (8)	
H8A	0.7097	0.5418	0.2034	0.050*	
H8B	0.5164	0.5470	0.3296	0.050*	
C9	0.6694 (6)	0.46783 (15)	0.3289 (4)	0.0348 (7)	
H9A	0.6187	0.4584	0.4337	0.042*	
H9B	0.5552	0.4522	0.2540	0.042*	
C10	0.9055 (6)	0.44005 (14)	0.3031 (4)	0.0337 (7)	
H10A	1.0215	0.4572	0.3741	0.040*	
H10B	0.9523	0.4478	0.1964	0.040*	
C11	0.9066 (5)	0.37617 (13)	0.3288 (3)	0.0266 (6)	
H11	0.8574	0.3684	0.4362	0.032*	
C12	1.1556 (5)	0.35209 (13)	0.3097 (4)	0.0278 (6)	
C13	0.2120 (7)	0.5547 (2)	0.9656 (5)	0.0490 (9)	
C14	0.3551 (6)	0.56626 (15)	0.8184 (5)	0.0399 (8)	
C15	0.1624 (6)	0.69327 (14)	0.6885 (4)	0.0332 (7)	
C16	0.3002 (6)	0.69936 (14)	0.5366 (4)	0.0463 (9)	
F1	0.2737 (7)	0.50496 (14)	1.0285 (4)	0.0803 (10)	
F2	−0.0167 (5)	0.5514 (2)	0.9324 (3)	0.0847 (11)	
F3	0.2391 (8)	0.59289 (17)	1.0789 (4)	0.0907 (12)	
N1	0.3086 (4)	0.41146 (12)	0.7262 (3)	0.0286 (5)	
H1C	0.4547	0.4054	0.7622	0.043*	
H1D	0.2802	0.4486	0.7238	0.043*	
H1E	0.2917	0.3973	0.6295	0.043*	
N2	0.2873 (5)	0.21051 (13)	1.0996 (3)	0.0344 (6)	
H2C	0.4418	0.2052	1.1077	0.052*	
H2D	0.2162	0.1881	1.1678	0.052*	
H2E	0.2544	0.2466	1.1204	0.052*	
N3	0.7381 (4)	0.34760 (12)	0.2158 (3)	0.0299 (5)	
H3C	0.5918	0.3535	0.2461	0.045*	
H3D	0.7672	0.3105	0.2144	0.045*	
H3E	0.7550	0.3619	0.1199	0.045*	
N4	0.8017 (6)	0.55253 (14)	0.5879 (4)	0.0403 (7)	
H4C	0.6511	0.5587	0.6072	0.060*	
H4D	0.8906	0.5759	0.6465	0.060*	
H4E	0.8383	0.5167	0.6113	0.060*	
O1	0.3110 (5)	0.53384 (12)	0.7075 (4)	0.0461 (6)	
O2	0.4978 (6)	0.60529 (15)	0.8282 (5)	0.0703 (10)	
O3	0.0036 (5)	0.65772 (12)	0.6844 (4)	0.0475 (7)	
O4	0.2218 (5)	0.72643 (11)	0.7963 (3)	0.0406 (6)	
O5	1.3033 (4)	0.36431 (13)	0.4147 (3)	0.0441 (6)	
O6	1.1940 (4)	0.32512 (12)	0.1873 (3)	0.0418 (6)	
O7	−0.1433 (4)	0.43812 (12)	0.6875 (3)	0.0411 (6)	
O8	−0.2560 (4)	0.39525 (12)	0.9057 (3)	0.0401 (6)	
F4	0.240 (3)	0.6636 (6)	0.4230 (12)	0.090 (4)	0.423 (18)
F5	0.5288 (10)	0.6952 (6)	0.5549 (13)	0.069 (3)	0.423 (18)

F6	0.263 (2)	0.7512 (4)	0.4766 (16)	0.084 (3)	0.423 (18)
F4′	0.3461 (18)	0.6491 (2)	0.4707 (10)	0.078 (2)	0.577 (18)
F5′	0.5098 (11)	0.7221 (5)	0.5632 (9)	0.074 (2)	0.577 (18)
F6′	0.1885 (16)	0.7293 (6)	0.4287 (9)	0.092 (3)	0.577 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.049 (2)	0.0265 (16)	0.0408 (18)	−0.0012 (14)	0.0043 (15)	0.0011 (14)
C2	0.054 (2)	0.0284 (17)	0.0366 (17)	0.0102 (15)	0.0107 (15)	0.0013 (13)
C3	0.0359 (17)	0.0298 (16)	0.0368 (16)	0.0041 (13)	0.0077 (13)	0.0052 (13)
C4	0.0374 (17)	0.0214 (14)	0.0371 (16)	−0.0017 (12)	−0.0037 (12)	0.0021 (12)
C5	0.0241 (13)	0.0260 (15)	0.0277 (13)	−0.0013 (10)	−0.0030 (10)	0.0025 (11)
C6	0.0241 (13)	0.0240 (14)	0.0337 (15)	−0.0008 (10)	−0.0033 (11)	−0.0007 (12)
C7	0.054 (2)	0.0270 (17)	0.0443 (19)	−0.0018 (15)	0.0063 (15)	0.0009 (14)
C8	0.056 (2)	0.0327 (18)	0.0352 (17)	0.0103 (16)	−0.0063 (15)	0.0015 (13)
C9	0.0388 (17)	0.0270 (15)	0.0382 (16)	0.0024 (13)	−0.0057 (13)	−0.0033 (13)
C10	0.0402 (17)	0.0253 (16)	0.0358 (16)	−0.0003 (13)	0.0046 (13)	−0.0020 (13)
C11	0.0270 (13)	0.0271 (15)	0.0260 (13)	0.0023 (11)	0.0037 (10)	0.0009 (11)
C12	0.0258 (13)	0.0253 (14)	0.0325 (15)	0.0023 (11)	0.0054 (11)	0.0018 (11)
C13	0.052 (2)	0.050 (2)	0.044 (2)	0.0021 (18)	−0.0073 (16)	−0.0040 (18)
C14	0.0298 (16)	0.0286 (18)	0.061 (2)	0.0016 (13)	−0.0010 (14)	0.0052 (16)
C15	0.0340 (16)	0.0242 (15)	0.0417 (18)	0.0024 (13)	0.0043 (13)	−0.0025 (13)
C16	0.050 (2)	0.050 (2)	0.0390 (19)	−0.0059 (17)	0.0059 (16)	0.0046 (17)
F1	0.111 (3)	0.065 (2)	0.0658 (19)	0.0023 (18)	0.0032 (17)	0.0254 (16)
F2	0.0436 (13)	0.150 (3)	0.0610 (16)	−0.0034 (19)	0.0068 (12)	0.009 (2)
F3	0.116 (3)	0.095 (3)	0.0607 (18)	0.012 (2)	−0.0090 (19)	−0.0309 (19)
N1	0.0241 (11)	0.0268 (13)	0.0349 (13)	0.0002 (10)	−0.0007 (10)	0.0027 (10)
N2	0.0363 (15)	0.0318 (15)	0.0356 (14)	0.0077 (11)	0.0082 (11)	0.0073 (11)
N3	0.0240 (11)	0.0253 (12)	0.0404 (14)	0.0009 (10)	0.0021 (10)	0.0007 (10)
N4	0.0505 (17)	0.0314 (15)	0.0386 (15)	0.0006 (13)	−0.0038 (12)	−0.0031 (12)
O1	0.0524 (15)	0.0326 (14)	0.0534 (16)	0.0008 (11)	0.0057 (12)	0.0023 (12)
O2	0.0528 (18)	0.0406 (17)	0.117 (3)	−0.0165 (14)	0.0025 (19)	0.0017 (19)
O3	0.0430 (14)	0.0419 (15)	0.0583 (16)	−0.0118 (11)	0.0129 (12)	−0.0149 (13)
O4	0.0426 (13)	0.0324 (13)	0.0471 (14)	−0.0041 (10)	0.0063 (11)	−0.0083 (11)
O5	0.0300 (12)	0.0520 (16)	0.0497 (15)	0.0023 (11)	−0.0063 (10)	−0.0119 (12)
O6	0.0397 (13)	0.0486 (15)	0.0372 (12)	0.0141 (11)	0.0048 (10)	−0.0094 (11)
O7	0.0385 (13)	0.0448 (15)	0.0396 (13)	0.0084 (11)	−0.0048 (10)	0.0129 (11)
O8	0.0269 (11)	0.0480 (15)	0.0454 (13)	0.0013 (10)	0.0009 (9)	0.0113 (12)
F4	0.096 (5)	0.103 (6)	0.070 (5)	−0.027 (4)	0.016 (4)	−0.021 (4)
F5	0.055 (4)	0.088 (5)	0.066 (4)	0.017 (3)	0.016 (3)	0.017 (4)
F6	0.102 (5)	0.078 (5)	0.071 (5)	0.002 (4)	0.016 (4)	0.030 (4)
F4′	0.100 (4)	0.075 (3)	0.063 (3)	0.020 (3)	0.037 (3)	−0.010 (3)
F5′	0.061 (3)	0.094 (5)	0.068 (3)	−0.027 (3)	0.023 (2)	−0.007 (3)
F6′	0.095 (4)	0.117 (5)	0.064 (3)	0.023 (4)	−0.004 (3)	0.036 (3)

Geometric parameters (Å, º)

C1—N2	1.481 (5)	C11—C12	1.541 (4)
C1—C2	1.521 (5)	C11—H11	0.9800
C1—H1A	0.9700	C12—O5	1.243 (4)
C1—H1B	0.9700	C12—O6	1.244 (4)
C2—C3	1.526 (5)	C13—F3	1.324 (5)
C2—H2A	0.9700	C13—F2	1.328 (5)
C2—H2B	0.9700	C13—F1	1.330 (5)
C3—C4	1.522 (5)	C13—C14	1.537 (6)
C3—H3A	0.9700	C14—O2	1.228 (5)
C3—H3B	0.9700	C14—O1	1.234 (5)
C4—C5	1.534 (4)	C15—O3	1.232 (4)
C4—H4A	0.9700	C15—O4	1.243 (4)
C4—H4B	0.9700	C15—C16	1.537 (5)
C5—N1	1.486 (4)	C16—F6′	1.308 (5)
C5—C6	1.535 (4)	C16—F5	1.312 (5)
C5—H5	0.9800	C16—F4	1.319 (5)
C6—O7	1.247 (4)	C16—F5′	1.323 (5)
C6—O8	1.253 (4)	C16—F6	1.337 (5)
C7—N4	1.484 (5)	C16—F4′	1.338 (5)
C7—C8	1.500 (5)	N1—H1C	0.8900
C7—H7A	0.9700	N1—H1D	0.8900
C7—H7B	0.9700	N1—H1E	0.8900
C8—C9	1.534 (5)	N2—H2C	0.8900
C8—H8A	0.9700	N2—H2D	0.8900
C8—H8B	0.9700	N2—H2E	0.8900
C9—C10	1.518 (5)	N3—H3C	0.8900
C9—H9A	0.9700	N3—H3D	0.8900
C9—H9B	0.9700	N3—H3E	0.8900
C10—C11	1.520 (5)	N4—H4C	0.8900
C10—H10A	0.9700	N4—H4D	0.8900
C10—H10B	0.9700	N4—H4E	0.8900
C11—N3	1.498 (4)		
N2—C1—C2	112.0 (3)	C12—C11—H11	108.6
N2—C1—H1A	109.2	O5—C12—O6	126.0 (3)
C2—C1—H1A	109.2	O5—C12—C11	116.5 (3)
N2—C1—H1B	109.2	O6—C12—C11	117.4 (3)
C2—C1—H1B	109.2	F3—C13—F2	106.7 (4)
H1A—C1—H1B	107.9	F3—C13—F1	106.3 (4)
C1—C2—C3	115.1 (3)	F2—C13—F1	106.3 (4)
C1—C2—H2A	108.5	F3—C13—C14	114.7 (4)
C3—C2—H2A	108.5	F2—C13—C14	112.2 (3)
C1—C2—H2B	108.5	F1—C13—C14	110.0 (4)
C3—C2—H2B	108.5	O2—C14—O1	129.4 (4)
H2A—C2—H2B	107.5	O2—C14—C13	116.3 (4)
C4—C3—C2	113.3 (3)	O1—C14—C13	114.3 (3)

C4—C3—H3A	108.9	O3—C15—O4	129.2 (3)
C2—C3—H3A	108.9	O3—C15—C16	115.6 (3)
C4—C3—H3B	108.9	O4—C15—C16	115.2 (3)
C2—C3—H3B	108.9	F6′—C16—F5	125.8 (6)
H3A—C3—H3B	107.7	F6′—C16—F4	73.7 (7)
C3—C4—C5	114.2 (3)	F5—C16—F4	106.1 (7)
C3—C4—H4A	108.7	F6′—C16—F5′	108.7 (6)
C5—C4—H4A	108.7	F5—C16—F5′	28.5 (5)
C3—C4—H4B	108.7	F4—C16—F5′	126.8 (6)
C5—C4—H4B	108.7	F6′—C16—F6	34.0 (5)
H4A—C4—H4B	107.6	F5—C16—F6	105.1 (7)
N1—C5—C4	110.9 (2)	F4—C16—F6	105.5 (8)
N1—C5—C6	110.5 (2)	F5′—C16—F6	80.1 (7)
C4—C5—C6	109.7 (2)	F6′—C16—F4′	106.3 (6)
N1—C5—H5	108.6	F5—C16—F4′	77.2 (6)
C4—C5—H5	108.6	F4—C16—F4′	34.9 (6)
C6—C5—H5	108.6	F5′—C16—F4′	104.1 (6)
O7—C6—O8	125.6 (3)	F6—C16—F4′	132.7 (6)
O7—C6—C5	117.4 (3)	F6′—C16—C15	112.9 (4)
O8—C6—C5	116.9 (3)	F5—C16—C15	115.0 (5)
N4—C7—C8	113.0 (3)	F4—C16—C15	115.2 (5)
N4—C7—H7A	109.0	F5′—C16—C15	112.0 (4)
C8—C7—H7A	109.0	F6—C16—C15	109.0 (5)
N4—C7—H7B	109.0	F4′—C16—C15	112.3 (4)
C8—C7—H7B	109.0	C5—N1—H1C	109.5
H7A—C7—H7B	107.8	C5—N1—H1D	109.5
C7—C8—C9	115.2 (3)	H1C—N1—H1D	109.5
C7—C8—H8A	108.5	C5—N1—H1E	109.5
C9—C8—H8A	108.5	H1C—N1—H1E	109.5
C7—C8—H8B	108.5	H1D—N1—H1E	109.5
C9—C8—H8B	108.5	C1—N2—H2C	109.5
H8A—C8—H8B	107.5	C1—N2—H2D	109.5
C10—C9—C8	113.6 (3)	H2C—N2—H2D	109.5
C10—C9—H9A	108.8	C1—N2—H2E	109.5
C8—C9—H9A	108.8	H2C—N2—H2E	109.5
C10—C9—H9B	108.8	H2D—N2—H2E	109.5
C8—C9—H9B	108.8	C11—N3—H3C	109.5
H9A—C9—H9B	107.7	C11—N3—H3D	109.5
C9—C10—C11	113.9 (3)	H3C—N3—H3D	109.5
C9—C10—H10A	108.8	C11—N3—H3E	109.5
C11—C10—H10A	108.8	H3C—N3—H3E	109.5
C9—C10—H10B	108.8	H3D—N3—H3E	109.5
C11—C10—H10B	108.8	C7—N4—H4C	109.5
H10A—C10—H10B	107.7	C7—N4—H4D	109.5
N3—C11—C10	110.6 (3)	H4C—N4—H4D	109.5
N3—C11—C12	110.0 (2)	C7—N4—H4E	109.5
C10—C11—C12	110.4 (3)	H4C—N4—H4E	109.5
N3—C11—H11	108.6	H4D—N4—H4E	109.5

C10—C11—H11	108.6		
N2—C1—C2—C3	−56.8 (4)	F3—C13—C14—O2	8.0 (5)
C1—C2—C3—C4	−62.9 (4)	F2—C13—C14—O2	130.0 (4)
C2—C3—C4—C5	173.4 (3)	F1—C13—C14—O2	−111.8 (4)
C3—C4—C5—N1	58.8 (3)	F3—C13—C14—O1	−173.0 (4)
C3—C4—C5—C6	−178.8 (3)	F2—C13—C14—O1	−51.0 (5)
N1—C5—C6—O7	14.6 (4)	F1—C13—C14—O1	67.2 (4)
C4—C5—C6—O7	−108.0 (3)	O3—C15—C16—F6′	78.6 (8)
N1—C5—C6—O8	−168.6 (3)	O4—C15—C16—F6′	−98.9 (8)
C4—C5—C6—O8	68.8 (4)	O3—C15—C16—F5	−127.4 (8)
N4—C7—C8—C9	−58.7 (4)	O4—C15—C16—F5	55.0 (9)
C7—C8—C9—C10	−58.1 (4)	O3—C15—C16—F4	−3.5 (11)
C8—C9—C10—C11	177.0 (3)	O4—C15—C16—F4	178.9 (10)
C9—C10—C11—N3	61.2 (3)	O3—C15—C16—F5′	−158.3 (7)
C9—C10—C11—C12	−176.7 (3)	O4—C15—C16—F5′	24.1 (8)
N3—C11—C12—O5	−167.2 (3)	O3—C15—C16—F6	114.9 (9)
C10—C11—C12—O5	70.4 (4)	O4—C15—C16—F6	−62.7 (9)
N3—C11—C12—O6	16.8 (4)	O3—C15—C16—F4′	−41.6 (7)
C10—C11—C12—O6	−105.6 (3)	O4—C15—C16—F4′	140.9 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*E*···O7^{i}	0.89	1.96	2.838 (4)	168
N4—H4*D*···O1^{i}	0.89	2.63	3.080 (4)	112
N4—H4*D*···F2^{i}	0.89	2.54	3.079 (4)	120
N4—H4*D*···O3^{i}	0.89	2.05	2.842 (4)	147
N3—H3*E*···O8^{ii}	0.89	1.98	2.866 (4)	172
N3—H3*D*···O4^{iii}	0.89	1.98	2.864 (4)	171
N3—H3*C*···O6^{iv}	0.89	2.40	3.148 (4)	142
N3—H3*C*···O5^{iv}	0.89	2.23	3.064 (4)	157
N2—H2*E*···O6^{v}	0.89	1.97	2.853 (4)	174
N2—H2*D*···O3^{vi}	0.89	1.94	2.800 (4)	163
N2—H2*C*···O4^{vii}	0.89	2.12	2.934 (4)	151
N1—H1*E*···O5^{iv}	0.89	1.99	2.870 (4)	172
N1—H1*C*···O7^{i}	0.89	2.52	3.212 (4)	136
N1—H1*C*···O8^{i}	0.89	2.04	2.901 (4)	163

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