3-(1*H*,1*H*,2*H*,2*H*-Perfluorooctyl)-1-vinyl-4-imidazoline-2-thione

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The title compound, $C_{13}H_9F_{13}N_2S$, was obtained by reaction of sulfur with the corresponding quaternary salt in the presence of K_2CO_3. The quaternary salt in turn was obtained by alkylation of 1-vinylimidazole. The crystal structure contains two independent molecules with disordered fluoroalkyl chains with occupancy ratios of 0.7:0.3 and 0.57:0.43.

3D view
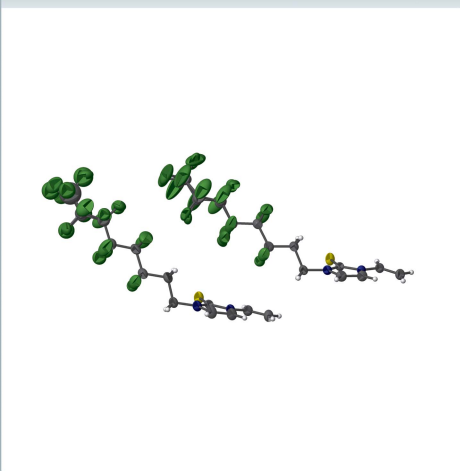
Chemical scheme
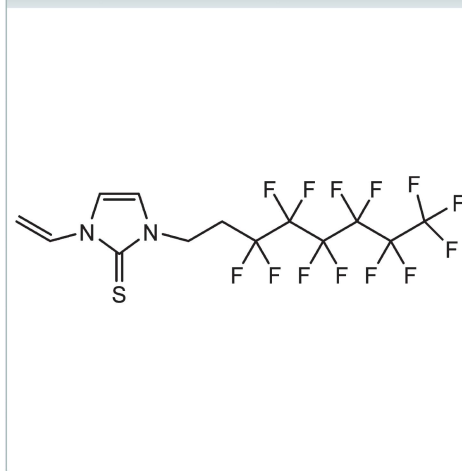
Structure description

Polyfluorinated compounds are utilized as surfactants and dispersants with numerous applications (Kovalchuk *et al.*, 2014; Krafft & Riess, 2009). Imidazoline-2-thiones (Laus *et al.*, 2013) are versatile building blocks whose properties have been reviewed (Trzhtsinskaya & Abramova, 1991). The key advantages of these thiones are simple synthesis and simple derivatization. The vinyl substituent makes the title molecule polymerizable, thus giving access to functionalized imidazolium-containing polymers (Anderson & Long, 2010). It was intended to combine these structural features in order to derive new materials with advantageous properties.

The most striking feature of the title molecule (Fig. 1) is the extensive disorder of the large substituent. The major component of the disordered fluoroalkyl chain adopts the typical helical conformation (Fournier *et al.*, 2010; Jang *et al.*, 2003) with an average CCCC twist angle (deviation from 180°) of 11°.

Specific C—F···F—C interactions have been identified in fluorous molecules (Baker *et al.*, 2012; Omorodion *et al.*, 2015). Here, a short F9D···F12C contact of 2.797 (6) Å is observed between the two chains (Fig. 2). In addition, C—H···S and C—H···F hydrogen bonds (Table 1) cross-link the rod-shaped molecules.

Related structures have been reported recently (Hummel *et al.*, 2017), notably a 4-(1*H*,1*H*,2*H*,2*H*-perfluorooctyl)-1,2,4-triazolium salt (Adamer *et al.*, 2011) and a
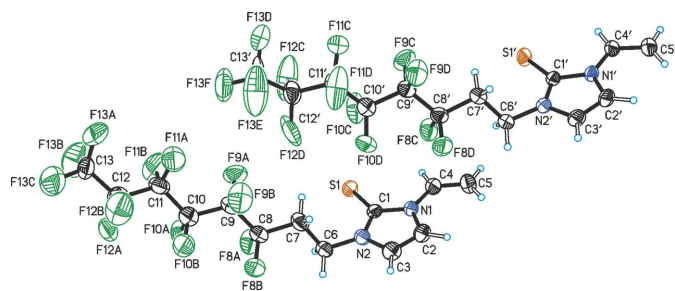
Figure 1
Asymmetric unit of the title compound, showing the atom labels and 50% probability displacement ellipsoids for non-H atoms. Minor disorder components are omitted for clarity.

1-(1*H*,1*H*,2*H*,2*H*-perfluorooctyl)-1,2,3-triazole (Omorodion *et al.*, 2015) have been described.

Synthesis and crystallization

3-(1*H*,1*H*,2*H*,2*H*-Perfluorooctyl)-1-vinylimidazolium iodide:
A mixture of 1-vinylimidazole (5.0 g, 53.1 mmol) and 1*H*,1*H*,2*H*,2*H*-perfluorooctyl iodide (26.0 g, 54.9 mmol) was stirred at 70°C for 72 h. After cooling, the mixture was dissolved in MeOH (25 ml) and the product precipitated by addition of Et$_2$O (250 ml). The product was filtered off, washed with Et$_2$O and dried in high vacuum overnight, affording 23.0 g (76%) of colourless product; m.p. 434 K. ^{1}H NMR (300 MHz, CD$_3$OD): δ 9.54 (*t*, *J* = 1.9 Hz, 1H), 8.07 (*t*, *J* = 1.9 Hz, 1H), 7.93 (*t*, *J* = 1.9 Hz, 1H), 7.30 (*dd*, *J* = 15.6, 8.7 Hz, 1H), 5.98 (*dd*, *J* = 15.6, 2.8 Hz, 1H), 5.49 (*dd*, *J* = 8.7, 2.8 Hz, 1H), 4.72 (*t*, *J* = 7.2 Hz, 2H), 3.06 (*tt*, *J* = 18.0, 7.2 Hz, 2H) p.p.m. ^{13}C NMR (75 MHz, CD$_3$OD): δ 137.3, 129.8, 124.7, 124.4–109.0 (*m*, 6C), 121.1, 110.6, 43.5 (*t*, *J* = 5.3 Hz), 31.9 (*t*, *J* = 21.4 Hz) p.p.m. IR (neat): *ν* 3117, 2991, 1652, 1568, 1142, 1119, 1071, 1020, 735, 699, 640, 620, 524 cm^{-1}.
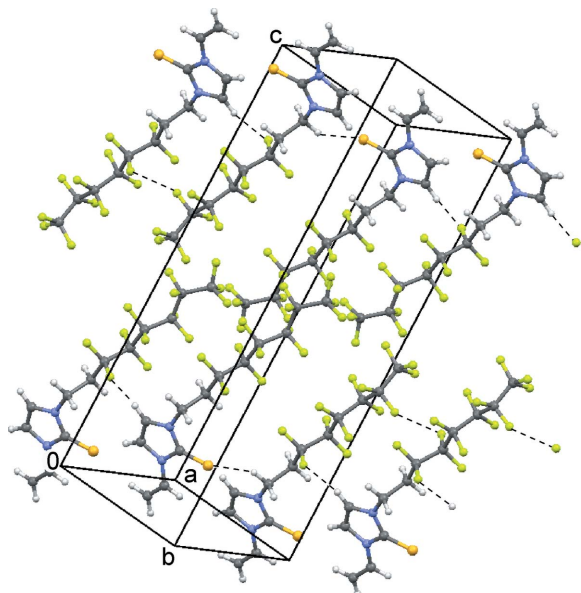

Figure 2
Crystal packing of the title compound. Minor disorder components are omitted for clarity.

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C3—H3⋯F8A^i	0.95	2.47	3.201 (5)	133
C3′—H3′⋯F8C^i	0.95	2.45	3.233 (4)	140
C6′—H6′1⋯S1^i	0.99	2.71	3.616 (3)	152
C4—H4⋯S1	0.95	2.80	3.179 (4)	105
C4′—H4′⋯S1′	0.95	2.83	3.191 (3)	104
C7′—H7′2⋯F9C	0.99	2.52	2.880 (5)	101
C7—H7B⋯F9A	0.99	2.54	2.875 (5)	100

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

Table 2
Experimental details.

Crystal data	
Chemical formula	C$_{13}$H$_9$F$_{13}$N$_2$S
M_r	472.28
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	193
a, b, c (Å)	7.425 (1), 9.952 (2), 24.359 (4)
α, β, γ (°)	85.076 (5), 83.431 (5), 88.720 (5)
V (Å^3)	1781.4 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.31
Crystal size (mm)	0.22 × 0.18 × 0.04
Data collection	
Diffractometer	Bruker D8 QUEST PHOTON 100
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.449, 0.490
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	44091, 6372, 4851
R_{int}	0.043
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.599
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.067, 0.176, 0.99
No. of reflections	6372
No. of parameters	679
No. of restraints	1685
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.86, −0.64

Computer programs: *APEX2* (Bruker 2014), *SAINT* (Bruker 2014), *SHELXL2014/6* (Sheldrick, 2015), *XP* in *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008), *Mercury* (Macrae *et al.*, 2008).

3-(1*H*,1*H*,2*H*,2*H*-Perfluorooctyl)-1-vinylimidazoline-2-thione: A mixture of 3-(1*H*,1*H*,2*H*,2*H*-perfluorooctyl)-1-vinylimidazolium iodide (80.0 g, 141 mmol), elemental sulfur (4.6 g, 144 mmol) and K$_2$CO$_3$ (19.9 g, 144 mmol) in MeOH (240 ml) was refluxed for 3 h. The solvent was evaporated and the residue extracted thrice with Et$_2$O (total 700 ml). The organic phase was treated with charcoal, filtered and the solvent was evaporated. The residue was dried in high vacuum overnight, affording 60.5 g (91%) of the product as an off-white powder; m.p. 359 K. Single crystals suitable for structure determination were fashioned by slow evaporation of a solution in CHCl$_3$. ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.53 (*dd*, *J* = 16.1, 9.0 Hz, 1H), 6.99 (*d*, *J* = 2.6 Hz, 1H), 6.75 (*d*, *J* = 2.6 Hz, 1H), 5.16 (*dd*, *J* = 16.1, 1.8 Hz, 1H), 4.96 (*dd*, *J* = 9.0, 1.8 Hz, 1H), 4.36 (*t*, *J* = 6.9 Hz, 2H), 2.70 (*tt*, *J* = 18.6, 6.9 Hz, 2H) p.p.m. ^{13}C NMR (75 MHz, CDCl$_3$): δ 163.4, 130.3, 124–105 (*m*, 6C),

118.6, 112.8, 101.4, 40.5 (t, J = 5.1 Hz), 29.6 (t, J = 21.5 Hz) p.p.m. IR (neat): ν 3133, 3104, 2995, 1643, 1234, 1182, 1139, 1118, 1077, 1062, 894, 694, 647, 509 cm^{-1}.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The terminal C_4F_9 units of the fluoroalkyl chains of both molecules are disordered over two sets of sites, and their disorder components were found to have relative occupancies of approximately 0.7:0.3 and 0.57:0.43 in the two molecules. Occupancies were fixed at these values in the final refinement cycles. The refinement of the disordered fragments was carried out with distance restraints (SAME/SADI) for all chemically equivalent 1,2- and 1,3-distances. All other non-H atoms were refined anisotropically, except for those in the minor disorder component of the first molecule, which were refined isotropically.

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full crystallographic data

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Crystal data

$C_{13}H_9F_{13}N_2S$	$Z = 4$
$M_r = 472.28$	$F(000) = 936$
Triclinic, $P\bar{1}$	$D_x = 1.761$ Mg m^{-3}
$a = 7.425$ (1) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 9.952$ (2) Å	Cell parameters from 9960 reflections
$c = 24.359$ (4) Å	$\theta = 2.3$–25.0°
$\alpha = 85.076$ (5)°	$\mu = 0.31$ mm^{-1}
$\beta = 83.431$ (5)°	$T = 193$ K
$\gamma = 88.720$ (5)°	Plate, colourless
$V = 1781.4$ (5) Å^3	0.22 × 0.18 × 0.04 mm

Data collection

Bruker D8 QUEST PHOTON 100 diffractometer	44091 measured reflections
Radiation source: Incoatec Microfocus	6372 independent reflections
Multi layered optics monochromator	4851 reflections with $I > 2\sigma(I)$
Detector resolution: 10.4 pixels mm^{-1}	$R_{int} = 0.043$
φ and ω scans	$\theta_{max} = 25.2°$, $\theta_{min} = 2.2°$
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)	$h = -8 \rightarrow 8$
	$k = -11 \rightarrow 11$
$T_{min} = 0.449$, $T_{max} = 0.490$	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.067$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.176$	
$S = 0.99$	H-atom parameters constrained
6372 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 3.9492P]$ where $P = (F_o^2 + 2F_c^2)/3$
679 parameters	
1685 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.86$ e Å^{-3}
	$\Delta\rho_{min} = -0.64$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.40414 (12)	0.52354 (9)	0.88826 (5)	0.0443 (3)	
N1	0.7681 (4)	0.5518 (3)	0.89347 (12)	0.0340 (7)	
N2	0.6520 (4)	0.7057 (3)	0.83893 (13)	0.0370 (7)	
C1	0.6094 (4)	0.5939 (3)	0.87344 (15)	0.0327 (7)	
C2	0.9074 (5)	0.6388 (4)	0.87116 (17)	0.0441 (9)	
H2	1.0305	0.6323	0.8783	0.053*	
C3	0.8345 (5)	0.7331 (4)	0.83786 (18)	0.0462 (10)	
H3	0.8969	0.8063	0.8170	0.055*	
C4	0.7800 (5)	0.4409 (4)	0.93323 (15)	0.0394 (8)	
H4	0.6720	0.3928	0.9457	0.047*	
C5	0.9282 (5)	0.3992 (4)	0.95423 (17)	0.0476 (10)	
H5A	1.0391	0.4446	0.9428	0.057*	
H5B	0.9247	0.3235	0.9809	0.057*	
C6	0.5221 (5)	0.7852 (4)	0.80941 (15)	0.0408 (9)	
H6A	0.5709	0.8765	0.7983	0.049*	
H6B	0.4082	0.7948	0.8343	0.049*	
C7	0.4820 (5)	0.7189 (4)	0.75785 (15)	0.0453 (9)	
H7A	0.5961	0.7096	0.7331	0.054*	
H7B	0.4341	0.6274	0.7691	0.054*	
C8	0.3448 (4)	0.8008 (3)	0.72632 (13)	0.0406 (8)	
F8A	0.1985 (3)	0.8280 (3)	0.76251 (10)	0.0623 (7)	
F8B	0.4136 (4)	0.9224 (2)	0.70743 (11)	0.0669 (7)	
C9	0.2799 (5)	0.7328 (3)	0.67842 (13)	0.0449 (9)	
F9A	0.2100 (5)	0.6147 (3)	0.69848 (12)	0.0874 (10)	
F9B	0.4259 (4)	0.7087 (3)	0.64324 (12)	0.0849 (10)	
C10	0.1424 (5)	0.8101 (4)	0.64541 (14)	0.0553 (11)	
F10A	0.0164 (6)	0.8699 (5)	0.67659 (17)	0.0861 (16)	0.7
F10B	0.2389 (7)	0.9168 (4)	0.6158 (2)	0.0916 (17)	0.7
C11	0.0614 (8)	0.7358 (6)	0.6021 (2)	0.053 (3)	0.7
F11A	0.1752 (8)	0.6416 (6)	0.5817 (2)	0.0975 (19)	0.7
F11B	−0.0764 (8)	0.6655 (5)	0.6313 (2)	0.111 (2)	0.7
C12	−0.0167 (8)	0.8189 (5)	0.5552 (2)	0.070 (2)	0.7
F12A	−0.0968 (7)	0.9311 (4)	0.57322 (19)	0.0761 (14)	0.7
F12B	0.1245 (7)	0.8610 (6)	0.51811 (19)	0.1042 (17)	0.7
C13	−0.1448 (7)	0.7494 (6)	0.5229 (2)	0.056 (3)	0.7
F13A	−0.0747 (10)	0.6335 (5)	0.5060 (3)	0.092 (2)	0.7
F13B	−0.3064 (7)	0.7206 (7)	0.5497 (2)	0.121 (2)	0.7
F13C	−0.1745 (9)	0.8273 (5)	0.47815 (19)	0.1055 (17)	0.7
F10E	0.1515 (15)	0.9404 (7)	0.6429 (5)	0.075 (3)*	0.3

F10F	−0.0086 (12)	0.7804 (11)	0.6841 (4)	0.077 (3)*	0.3
C11A	0.0840 (11)	0.7482 (11)	0.5963 (3)	0.061 (9)*	0.3
F11E	0.2058 (11)	0.7957 (8)	0.5557 (3)	0.061 (2)*	0.3
F11F	0.1012 (18)	0.6148 (10)	0.6003 (6)	0.071 (4)*	0.3
C12A	−0.1085 (10)	0.7732 (12)	0.5828 (4)	0.059 (4)*	0.3
F12E	−0.170 (2)	0.8957 (10)	0.5935 (6)	0.102 (6)*	0.3
F12F	−0.2213 (18)	0.6889 (10)	0.6146 (5)	0.104 (4)*	0.3
C13A	−0.1427 (17)	0.7621 (17)	0.5233 (4)	0.19 (3)*	0.3
F13G	−0.0388 (17)	0.8340 (14)	0.4839 (6)	0.107 (4)*	0.3
F13H	−0.3131 (15)	0.7907 (16)	0.5150 (7)	0.124 (5)*	0.3
F13I	−0.114 (3)	0.6331 (14)	0.5142 (13)	0.148 (12)*	0.3
S1′	1.14881 (11)	−0.08928 (10)	0.93033 (4)	0.0420 (3)	
N1′	1.5143 (3)	−0.0605 (3)	0.93303 (11)	0.0304 (6)	
N2′	1.3884 (4)	0.1043 (3)	0.88608 (12)	0.0320 (6)	
C1′	1.3513 (4)	−0.0151 (3)	0.91672 (14)	0.0302 (7)	
C2′	1.6495 (4)	0.0316 (4)	0.91273 (15)	0.0380 (8)	
H2′	1.7741	0.0238	0.9182	0.046*	
C3′	1.5710 (5)	0.1328 (4)	0.88420 (16)	0.0396 (8)	
H3′	1.6299	0.2106	0.8658	0.048*	
C4′	1.5358 (4)	−0.1827 (3)	0.96593 (14)	0.0338 (8)	
H4′	1.4367	−0.2430	0.9717	0.041*	
C5′	1.6825 (5)	−0.2188 (4)	0.98903 (16)	0.0414 (9)	
H5′1	1.7843	−0.1611	0.9842	0.050*	
H5′2	1.6869	−0.3027	1.0106	0.050*	
C6′	1.2529 (5)	0.1929 (3)	0.86435 (14)	0.0365 (8)	
H6′1	1.3028	0.2848	0.8562	0.044*	
H6′2	1.1469	0.1976	0.8928	0.044*	
C7′	1.1912 (5)	0.1455 (3)	0.81163 (14)	0.0385 (8)	
H7′1	1.2986	0.1317	0.7845	0.046*	
H7′2	1.1295	0.0579	0.8205	0.046*	
C8′	1.0628 (4)	0.2470 (3)	0.78577 (12)	0.0365 (8)	
F8C	0.9324 (3)	0.2841 (2)	0.82534 (9)	0.0545 (6)	
F8D	1.1538 (3)	0.3607 (2)	0.76671 (10)	0.0587 (7)	
C9′	0.9688 (4)	0.1972 (3)	0.73893 (12)	0.0379 (8)	
F9C	0.8781 (4)	0.0847 (2)	0.75896 (11)	0.0676 (8)	
F9D	1.0966 (3)	0.1600 (3)	0.69965 (10)	0.0686 (8)	
C10′	0.8368 (5)	0.2946 (4)	0.71214 (14)	0.0537 (11)	
F10C	0.6801 (6)	0.2835 (7)	0.75193 (19)	0.0753 (17)	0.57
F10D	0.8782 (11)	0.4206 (5)	0.7110 (4)	0.096 (3)	0.57
C11′	0.7630 (9)	0.2554 (8)	0.6605 (2)	0.044 (3)	0.57
F11C	0.7459 (16)	0.1231 (8)	0.6598 (7)	0.091 (3)	0.57
F11D	0.8918 (6)	0.2935 (8)	0.6201 (2)	0.102 (2)	0.57
C12′	0.5851 (8)	0.3183 (7)	0.6460 (2)	0.053 (2)	0.57
F12C	0.4525 (7)	0.2527 (9)	0.6777 (3)	0.128 (3)	0.57
F12D	0.5822 (18)	0.4423 (8)	0.6606 (6)	0.154 (7)	0.57
C13′	0.5365 (10)	0.3114 (10)	0.5882 (3)	0.120 (6)	0.57
F13D	0.5385 (15)	0.1849 (7)	0.5766 (5)	0.163 (9)	0.57
F13E	0.6497 (11)	0.3793 (11)	0.5506 (3)	0.159 (4)	0.57

F13F	0.3713 (9)	0.3592 (9)	0.5831 (4)	0.105 (2)	0.57
F10G	0.9634 (10)	0.3818 (7)	0.6787 (3)	0.082 (3)	0.43
F10H	0.7523 (13)	0.3770 (10)	0.7450 (4)	0.103 (4)	0.43
C11B	0.7302 (11)	0.2438 (9)	0.6693 (3)	0.056 (5)	0.43
F11G	0.5858 (10)	0.1941 (10)	0.7016 (3)	0.106 (3)	0.43
F11H	0.8144 (19)	0.1406 (15)	0.6462 (8)	0.089 (5)	0.43
C12B	0.6696 (11)	0.3427 (9)	0.6244 (3)	0.068 (4)	0.43
F12G	0.8066 (11)	0.3687 (11)	0.5844 (3)	0.117 (3)	0.43
F12H	0.6144 (17)	0.4608 (11)	0.6416 (6)	0.091 (4)	0.43
C13B	0.5192 (11)	0.3016 (10)	0.5930 (3)	0.074 (6)	0.43
F13J	0.567 (2)	0.1878 (12)	0.5705 (6)	0.159 (11)	0.43
F13K	0.4823 (18)	0.3900 (11)	0.5520 (4)	0.126 (4)	0.43
F13L	0.3649 (11)	0.2818 (18)	0.6255 (4)	0.215 (10)	0.43

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0262 (5)	0.0372 (5)	0.0702 (7)	−0.0085 (4)	−0.0110 (4)	0.0010 (4)
N1	0.0251 (14)	0.0314 (15)	0.0463 (17)	−0.0011 (11)	−0.0066 (12)	−0.0047 (13)
N2	0.0303 (15)	0.0325 (16)	0.0481 (18)	−0.0015 (12)	−0.0055 (13)	−0.0010 (13)
C1	0.0276 (17)	0.0286 (17)	0.043 (2)	−0.0019 (13)	−0.0062 (14)	−0.0072 (15)
C2	0.0226 (17)	0.046 (2)	0.063 (3)	−0.0060 (15)	−0.0010 (17)	−0.0026 (19)
C3	0.0284 (18)	0.044 (2)	0.063 (3)	−0.0086 (16)	0.0035 (17)	0.0045 (19)
C4	0.0344 (19)	0.038 (2)	0.046 (2)	−0.0018 (15)	−0.0068 (16)	−0.0021 (16)
C5	0.040 (2)	0.050 (2)	0.053 (2)	0.0072 (18)	−0.0094 (18)	−0.0008 (19)
C6	0.041 (2)	0.0322 (19)	0.050 (2)	0.0037 (15)	−0.0080 (17)	−0.0012 (16)
C7	0.047 (2)	0.040 (2)	0.048 (2)	0.0122 (17)	−0.0056 (18)	−0.0048 (17)
C8	0.042 (2)	0.0345 (19)	0.044 (2)	0.0041 (16)	−0.0007 (17)	−0.0015 (16)
F8A	0.0455 (13)	0.0904 (19)	0.0524 (14)	0.0194 (13)	−0.0045 (11)	−0.0209 (13)
F8B	0.0813 (18)	0.0425 (14)	0.0796 (18)	−0.0141 (12)	−0.0314 (15)	0.0126 (12)
C9	0.055 (2)	0.035 (2)	0.043 (2)	0.0083 (17)	−0.0015 (18)	−0.0031 (16)
F9A	0.148 (3)	0.0412 (14)	0.0799 (19)	−0.0284 (16)	−0.053 (2)	0.0146 (13)
F9B	0.0703 (19)	0.120 (3)	0.0662 (18)	0.0339 (18)	−0.0012 (15)	−0.0359 (17)
C10	0.064 (3)	0.047 (2)	0.058 (3)	0.006 (2)	−0.016 (2)	−0.007 (2)
F10A	0.099 (3)	0.091 (3)	0.082 (3)	0.064 (3)	−0.049 (3)	−0.051 (3)
F10B	0.122 (4)	0.061 (3)	0.096 (4)	−0.036 (3)	−0.058 (3)	0.037 (2)
C11	0.079 (6)	0.033 (4)	0.048 (4)	0.007 (3)	−0.012 (3)	−0.005 (3)
F11A	0.126 (5)	0.089 (4)	0.091 (4)	0.053 (4)	−0.047 (4)	−0.054 (3)
F11B	0.149 (5)	0.107 (4)	0.081 (3)	−0.065 (4)	−0.041 (3)	0.030 (3)
C12	0.095 (6)	0.060 (4)	0.059 (4)	0.000 (4)	−0.022 (4)	−0.001 (3)
F12A	0.102 (4)	0.059 (2)	0.075 (3)	0.033 (3)	−0.041 (3)	−0.015 (2)
F12B	0.102 (4)	0.127 (4)	0.080 (3)	−0.033 (3)	−0.013 (3)	0.022 (3)
C13	0.064 (5)	0.063 (5)	0.046 (4)	0.008 (3)	−0.025 (3)	−0.010 (3)
F13A	0.116 (5)	0.085 (4)	0.089 (4)	0.007 (3)	−0.042 (3)	−0.044 (3)
F13B	0.086 (4)	0.164 (6)	0.112 (5)	−0.026 (4)	−0.003 (3)	−0.006 (4)
F13C	0.133 (5)	0.100 (4)	0.087 (4)	−0.020 (3)	−0.039 (3)	0.013 (3)
S1′	0.0220 (4)	0.0404 (5)	0.0627 (6)	−0.0056 (4)	−0.0066 (4)	0.0047 (4)
N1′	0.0218 (13)	0.0340 (15)	0.0357 (16)	−0.0020 (11)	−0.0032 (11)	−0.0049 (12)

N2′	0.0275 (14)	0.0293 (15)	0.0398 (16)	−0.0032 (11)	−0.0055 (12)	−0.0034 (12)
C1′	0.0248 (16)	0.0299 (17)	0.0370 (19)	−0.0010 (13)	−0.0055 (14)	−0.0071 (14)
C2′	0.0228 (17)	0.042 (2)	0.050 (2)	−0.0082 (15)	−0.0018 (15)	−0.0045 (17)
C3′	0.0302 (18)	0.036 (2)	0.052 (2)	−0.0099 (15)	−0.0017 (16)	−0.0023 (17)
C4′	0.0279 (17)	0.0335 (18)	0.040 (2)	−0.0002 (14)	−0.0042 (15)	−0.0028 (15)
C5′	0.0353 (19)	0.043 (2)	0.047 (2)	0.0026 (16)	−0.0072 (16)	−0.0031 (17)
C6′	0.0377 (19)	0.0275 (17)	0.045 (2)	0.0031 (14)	−0.0091 (16)	−0.0043 (15)
C7′	0.041 (2)	0.0318 (19)	0.043 (2)	0.0041 (15)	−0.0080 (16)	−0.0044 (15)
C8′	0.0368 (19)	0.0301 (18)	0.041 (2)	−0.0023 (15)	−0.0009 (16)	−0.0010 (15)
F8C	0.0446 (13)	0.0722 (16)	0.0489 (13)	0.0176 (11)	−0.0077 (10)	−0.0196 (12)
F8D	0.0648 (15)	0.0399 (13)	0.0732 (17)	−0.0168 (11)	−0.0268 (13)	0.0135 (11)
C9′	0.0382 (19)	0.0344 (19)	0.040 (2)	−0.0004 (15)	−0.0008 (16)	−0.0021 (15)
F9C	0.0842 (19)	0.0488 (14)	0.0737 (17)	−0.0302 (13)	−0.0348 (15)	0.0141 (12)
F9D	0.0536 (15)	0.100 (2)	0.0551 (15)	0.0205 (14)	−0.0059 (12)	−0.0324 (14)
C10′	0.061 (3)	0.043 (2)	0.060 (3)	0.009 (2)	−0.021 (2)	−0.009 (2)
F10C	0.054 (3)	0.131 (5)	0.041 (3)	0.040 (3)	−0.006 (2)	−0.023 (3)
F10D	0.142 (8)	0.035 (3)	0.129 (8)	0.021 (4)	−0.090 (6)	−0.018 (4)
C11′	0.042 (5)	0.056 (7)	0.035 (5)	0.005 (5)	−0.006 (4)	−0.005 (5)
F11C	0.111 (9)	0.048 (4)	0.131 (9)	0.019 (4)	−0.072 (7)	−0.029 (4)
F11D	0.048 (3)	0.211 (8)	0.040 (3)	−0.001 (4)	0.001 (2)	0.013 (4)
C12′	0.041 (5)	0.067 (6)	0.053 (5)	0.016 (4)	−0.009 (4)	−0.010 (4)
F12C	0.036 (3)	0.262 (10)	0.082 (4)	−0.001 (4)	−0.007 (3)	0.013 (5)
F12D	0.239 (13)	0.113 (9)	0.140 (11)	0.128 (9)	−0.136 (9)	−0.063 (8)
C13′	0.109 (11)	0.181 (13)	0.078 (10)	0.030 (10)	−0.070 (8)	0.023 (10)
F13D	0.202 (15)	0.092 (7)	0.238 (17)	0.035 (7)	−0.185 (14)	−0.050 (8)
F13E	0.143 (7)	0.262 (11)	0.070 (4)	0.013 (7)	−0.046 (5)	0.043 (5)
F13F	0.086 (5)	0.141 (6)	0.101 (6)	0.042 (5)	−0.057 (4)	−0.028 (5)
F10G	0.100 (6)	0.035 (4)	0.121 (7)	−0.027 (4)	−0.074 (5)	0.030 (4)
F10H	0.124 (9)	0.089 (8)	0.116 (9)	0.070 (7)	−0.075 (7)	−0.064 (7)
C11B	0.055 (8)	0.055 (11)	0.060 (9)	−0.004 (7)	−0.012 (7)	−0.002 (7)
F11G	0.075 (5)	0.158 (8)	0.085 (5)	−0.063 (5)	−0.026 (4)	0.041 (5)
F11H	0.087 (9)	0.098 (9)	0.102 (9)	0.048 (7)	−0.058 (7)	−0.070 (8)
C12B	0.064 (9)	0.082 (10)	0.055 (8)	0.005 (7)	−0.004 (7)	0.000 (7)
F12G	0.090 (6)	0.179 (9)	0.076 (5)	−0.022 (6)	−0.016 (5)	0.034 (6)
F12H	0.132 (8)	0.054 (5)	0.102 (9)	0.018 (6)	−0.085 (6)	−0.007 (5)
C13B	0.065 (11)	0.070 (10)	0.090 (15)	−0.007 (9)	−0.012 (10)	−0.027 (9)
F13J	0.137 (14)	0.26 (3)	0.102 (11)	0.050 (14)	−0.040 (9)	−0.115 (14)
F13K	0.130 (10)	0.136 (9)	0.117 (9)	−0.001 (8)	−0.071 (8)	0.036 (7)
F13L	0.062 (6)	0.44 (3)	0.131 (10)	−0.063 (10)	−0.036 (6)	0.087 (14)

Geometric parameters (Å, º)

S1—C1	1.679 (3)	S1′—C1′	1.676 (3)
N1—C1	1.370 (4)	N1′—C1′	1.372 (4)
N1—C2	1.395 (4)	N1′—C2′	1.393 (4)
N1—C4	1.412 (5)	N1′—C4′	1.415 (4)
N2—C1	1.357 (4)	N2′—C1′	1.363 (4)
N2—C3	1.385 (5)	N2′—C3′	1.386 (4)

N2—C6	1.449 (4)	N2′—C6′	1.440 (4)
C2—C3	1.333 (6)	C2′—C3′	1.335 (5)
C2—H2	0.9500	C2′—H2′	0.9500
C3—H3	0.9500	C3′—H3′	0.9500
C4—C5	1.310 (5)	C4′—C5′	1.311 (5)
C4—H4	0.9500	C4′—H4′	0.9500
C5—H5A	0.9500	C5′—H5′1	0.9500
C5—H5B	0.9500	C5′—H5′2	0.9500
C6—C7	1.529 (4)	C6′—C7′	1.526 (4)
C6—H6A	0.9900	C6′—H6′1	0.9900
C6—H6B	0.9900	C6′—H6′2	0.9900
C7—C8	1.525 (4)	C7′—C8′	1.521 (4)
C7—H7A	0.9900	C7′—H7′1	0.9900
C7—H7B	0.9900	C7′—H7′2	0.9900
C8—F8B	1.348 (4)	C8′—F8D	1.350 (3)
C8—F8A	1.356 (4)	C8′—F8C	1.354 (4)
C8—C9	1.527 (4)	C8′—C9′	1.526 (4)
C9—F9A	1.327 (4)	C9′—F9D	1.338 (4)
C9—F9B	1.333 (4)	C9′—F9C	1.346 (4)
C9—C10	1.525 (4)	C9′—C10′	1.523 (4)
C10—F10E	1.296 (6)	C10′—F10D	1.295 (5)
C10—F10A	1.302 (4)	C10′—F10H	1.297 (6)
C10—F10B	1.394 (5)	C10′—F10C	1.426 (5)
C10—F10F	1.400 (6)	C10′—F10G	1.429 (6)
C10—C11A	1.502 (6)	C10′—C11B	1.506 (6)
C10—C11	1.524 (5)	C10′—C11′	1.512 (6)
C11—F11A	1.337 (5)	C11′—F11D	1.327 (7)
C11—F11B	1.350 (6)	C11′—F11C	1.328 (6)
C11—C12	1.514 (5)	C11′—C12′	1.514 (6)
C12—F12A	1.337 (5)	C12′—F12D	1.313 (6)
C12—F12B	1.352 (6)	C12′—F12C	1.328 (6)
C12—C13	1.515 (6)	C12′—C13′	1.500 (6)
C13—F13C	1.320 (5)	C13′—F13D	1.313 (7)
C13—F13B	1.324 (5)	C13′—F13F	1.321 (6)
C13—F13A	1.333 (5)	C13′—F13E	1.321 (7)
C11A—F11E	1.325 (7)	C11B—F11H	1.324 (7)
C11A—F11F	1.327 (7)	C11B—F11G	1.333 (7)
C11A—C12A	1.514 (7)	C11B—C12B	1.508 (6)
C12A—F12E	1.328 (7)	C12B—F12H	1.322 (7)
C12A—F12F	1.333 (7)	C12B—F12G	1.339 (7)
C12A—C13A	1.514 (7)	C12B—C13B	1.507 (6)
C13A—F13H	1.324 (7)	C13B—F13K	1.322 (6)
C13A—F13G	1.329 (7)	C13B—F13J	1.323 (7)
C13A—F13I	1.330 (7)	C13B—F13L	1.324 (7)
C1—N1—C2	109.7 (3)	C1′—N1′—C2′	109.8 (3)
C1—N1—C4	123.3 (3)	C1′—N1′—C4′	123.5 (3)
C2—N1—C4	126.9 (3)	C2′—N1′—C4′	126.7 (3)

C1—N2—C3	109.8 (3)	C1′—N2′—C3′	109.9 (3)
C1—N2—C6	124.1 (3)	C1′—N2′—C6′	124.2 (3)
C3—N2—C6	126.1 (3)	C3′—N2′—C6′	125.6 (3)
N2—C1—N1	105.5 (3)	N2′—C1′—N1′	105.2 (3)
N2—C1—S1	126.7 (3)	N2′—C1′—S1′	126.7 (2)
N1—C1—S1	127.8 (3)	N1′—C1′—S1′	128.1 (3)
C3—C2—N1	106.8 (3)	C3′—C2′—N1′	107.1 (3)
C3—C2—H2	126.6	C3′—C2′—H2′	126.4
N1—C2—H2	126.6	N1′—C2′—H2′	126.4
C2—C3—N2	108.2 (3)	C2′—C3′—N2′	108.0 (3)
C2—C3—H3	125.9	C2′—C3′—H3′	126.0
N2—C3—H3	125.9	N2′—C3′—H3′	126.0
C5—C4—N1	125.2 (4)	C5′—C4′—N1′	124.8 (3)
C5—C4—H4	117.4	C5′—C4′—H4′	117.6
N1—C4—H4	117.4	N1′—C4′—H4′	117.6
C4—C5—H5A	120.0	C4′—C5′—H5′1	120.0
C4—C5—H5B	120.0	C4′—C5′—H5′2	120.0
H5A—C5—H5B	120.0	H5′1—C5′—H5′2	120.0
N2—C6—C7	111.1 (3)	N2′—C6′—C7′	112.1 (3)
N2—C6—H6A	109.4	N2′—C6′—H6′1	109.2
C7—C6—H6A	109.4	C7′—C6′—H6′1	109.2
N2—C6—H6B	109.4	N2′—C6′—H6′2	109.2
C7—C6—H6B	109.4	C7′—C6′—H6′2	109.2
H6A—C6—H6B	108.0	H6′1—C6′—H6′2	107.9
C8—C7—C6	111.9 (3)	C8′—C7′—C6′	111.6 (3)
C8—C7—H7A	109.2	C8′—C7′—H7′1	109.3
C6—C7—H7A	109.2	C6′—C7′—H7′1	109.3
C8—C7—H7B	109.2	C8′—C7′—H7′2	109.3
C6—C7—H7B	109.2	C6′—C7′—H7′2	109.3
H7A—C7—H7B	107.9	H7′1—C7′—H7′2	108.0
F8B—C8—F8A	104.9 (3)	F8D—C8′—F8C	105.8 (3)
F8B—C8—C7	109.9 (3)	F8D—C8′—C7′	109.4 (3)
F8A—C8—C7	108.6 (3)	F8C—C8′—C7′	109.5 (3)
F8B—C8—C9	109.5 (3)	F8D—C8′—C9′	109.0 (2)
F8A—C8—C9	108.5 (3)	F8C—C8′—C9′	107.7 (2)
C7—C8—C9	115.0 (3)	C7′—C8′—C9′	115.0 (3)
F9A—C9—F9B	107.7 (3)	F9D—C9′—F9C	106.3 (3)
F9A—C9—C10	108.6 (3)	F9D—C9′—C10′	108.9 (3)
F9B—C9—C10	107.7 (3)	F9C—C9′—C10′	108.0 (3)
F9A—C9—C8	108.1 (3)	F9D—C9′—C8′	108.2 (2)
F9B—C9—C8	107.2 (3)	F9C—C9′—C8′	107.9 (2)
C10—C9—C8	117.3 (3)	C10′—C9′—C8′	117.0 (3)
F10A—C10—F10B	103.6 (4)	F10D—C10′—F10C	102.9 (5)
F10E—C10—F10F	103.6 (5)	F10H—C10′—F10G	101.8 (5)
F10E—C10—C11A	117.7 (7)	F10H—C10′—C11B	116.3 (6)
F10F—C10—C11A	99.7 (6)	F10G—C10′—C11B	101.8 (5)
F10A—C10—C11	111.0 (4)	F10D—C10′—C11′	114.7 (5)
F10B—C10—C11	105.5 (4)	F10C—C10′—C11′	101.8 (4)

F10E—C10—C9	115.7 (6)	F10D—C10′—C9′	114.5 (4)
F10A—C10—C9	113.1 (3)	F10H—C10′—C9′	114.9 (4)
F10B—C10—C9	105.0 (3)	F10C—C10′—C9′	101.8 (3)
F10F—C10—C9	96.0 (5)	F10G—C10′—C9′	99.4 (4)
C11A—C10—C9	118.1 (4)	C11B—C10′—C9′	118.0 (4)
C11—C10—C9	117.1 (3)	C11′—C10′—C9′	117.9 (4)
F11A—C11—F11B	104.6 (5)	F11D—C11′—F11C	106.8 (6)
F11A—C11—C12	110.0 (5)	F11D—C11′—C10′	103.5 (5)
F11B—C11—C12	107.1 (5)	F11C—C11′—C10′	112.9 (9)
F11A—C11—C10	111.7 (4)	F11D—C11′—C12′	108.3 (5)
F11B—C11—C10	104.1 (4)	F11C—C11′—C12′	106.4 (8)
C12—C11—C10	118.1 (4)	C10′—C11′—C12′	118.3 (5)
F12A—C12—F12B	105.7 (5)	F12D—C12′—F12C	107.2 (6)
F12A—C12—C11	110.6 (4)	F12D—C12′—C13′	112.4 (9)
F12B—C12—C11	107.1 (5)	F12C—C12′—C13′	104.0 (6)
F12A—C12—C13	109.1 (5)	F12D—C12′—C11′	106.6 (7)
F12B—C12—C13	106.2 (4)	F12C—C12′—C11′	107.6 (6)
C11—C12—C13	117.4 (4)	C13′—C12′—C11′	118.4 (5)
F13C—C13—F13B	106.1 (4)	F13D—C13′—F13F	107.0 (6)
F13C—C13—F13A	107.2 (5)	F13D—C13′—F13E	108.4 (7)
F13B—C13—F13A	106.3 (5)	F13F—C13′—F13E	107.8 (6)
F13C—C13—C12	109.5 (5)	F13D—C13′—C12′	109.4 (8)
F13B—C13—C12	115.6 (5)	F13F—C13′—C12′	111.6 (7)
F13A—C13—C12	111.6 (5)	F13E—C13′—C12′	112.5 (7)
F11E—C11A—F11F	107.3 (7)	F11H—C11B—F11G	106.7 (7)
F11E—C11A—C10	102.0 (7)	F11H—C11B—C10′	111.1 (10)
F11F—C11A—C10	112.6 (9)	F11G—C11B—C10′	100.5 (6)
F11E—C11A—C12A	112.4 (7)	F11H—C11B—C12B	109.1 (11)
F11F—C11A—C12A	103.9 (10)	F11G—C11B—C12B	109.6 (7)
C10—C11A—C12A	118.4 (7)	C10′—C11B—C12B	118.9 (7)
F12E—C12A—F12F	105.0 (7)	F12H—C12B—F12G	106.1 (7)
F12E—C12A—C11A	112.6 (10)	F12H—C12B—C13B	103.9 (9)
F12F—C12A—C11A	110.3 (10)	F12G—C12B—C13B	103.1 (7)
F12E—C12A—C13A	104.1 (12)	F12H—C12B—C11B	114.2 (10)
F12F—C12A—C13A	107.2 (10)	F12G—C12B—C11B	110.2 (7)
C11A—C12A—C13A	116.8 (7)	C13B—C12B—C11B	118.1 (6)
F13H—C13A—F13G	106.8 (7)	F13K—C13B—F13J	106.7 (6)
F13H—C13A—F13I	106.8 (7)	F13K—C13B—F13L	106.2 (6)
F13G—C13A—F13I	106.8 (8)	F13J—C13B—F13L	108.4 (7)
F13H—C13A—C12A	112.5 (12)	F13K—C13B—C12B	114.3 (8)
F13G—C13A—C12A	117.5 (12)	F13J—C13B—C12B	108.8 (10)
F13I—C13A—C12A	105.7 (18)	F13L—C13B—C12B	112.2 (8)

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C3—H3⋯F8A[i]	0.95	2.47	3.201 (5)	133
C3′—H3′⋯F8C[i]	0.95	2.45	3.233 (4)	140

C6′—H6′1···S1[i]	0.99	2.71	3.616 (3)	152
C4—H4···S1	0.95	2.80	3.179 (4)	105
C4′—H4′···S1′	0.95	2.83	3.191 (3)	104
C7′—H7′2···F9*C*	0.99	2.52	2.880 (5)	101
C7—H7*B*···F9*A*	0.99	2.54	2.875 (5)	100

Symmetry code: (i) *x*+1, *y*, *z*.