

Bis(DL-valinium) pentafluorido-oxidoniobate(V)

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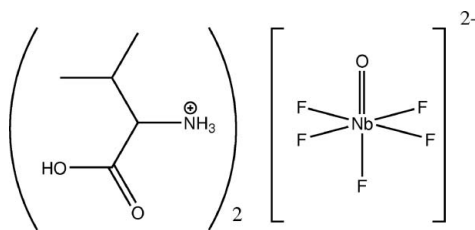
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.030; wR factor = 0.078; data-to-parameter ratio = 26.7.

The asymmetric unit of the title compound [systematic name: (*R*)-1-carboxy-2-methylpropan-1-aminium (*S*)-1-carboxy-2-methylpropan-1-aminium pentafluoridooxidoniobate(V)], $(\text{C}_5\text{H}_{12}\text{NO}_2)_2[\text{NbOF}_5]$, consists of a discrete $[\text{NbOF}_5]^{2-}$ complex anion and two protonated valinium cations. The Nb atom has a distorted octahedral geometry and the Nb—F bond *trans* to the Nb=O bond is significantly longer than the other four Nb—F bonds in the polyhedron. The valinium cations are linked to the pentafluoridooxidoniobate(V) anions *via* O—H...F, N—H...F and N—H...O hydrogen bonds.

Related literature

For related literature, see: Bondi (1964); Sarin *et al.* (1977); Zhu *et al.* (2005); Zhu & Tang (2005).



Experimental

Crystal data

$(\text{C}_5\text{H}_{12}\text{NO}_2)_2[\text{NbOF}_5]$

$M_r = 440.22$

Monoclinic, $P2_1/c$

$a = 9.8018$ (4) Å

$b = 19.4213$ (8) Å

$c = 10.1979$ (4) Å

$\beta = 116.190$ (1)°

$V = 1742.01$ (12) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.76$ mm⁻¹

$T = 296$ (2) K

$0.32 \times 0.31 \times 0.30$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Absorption correction: Gaussian (*SADABS* and *XPREP* in

SAINT; Bruker, 2003)

$T_{\min} = 0.861$, $T_{\max} = 0.907$

20336 measured reflections

5717 independent reflections

4829 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.078$

$S = 1.08$

5717 reflections

214 parameters

H-atom parameters constrained

$\Delta\rho_{\max} = 0.51$ e Å⁻³

$\Delta\rho_{\min} = -0.53$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Nb—O1	1.7172 (9)	Nb—F4	1.9339 (9)
Nb—F3	1.8906 (9)	Nb—F5	1.9648 (9)
Nb—F2	1.9331 (9)	Nb—F1	2.2270 (7)
O1—Nb—F1	174.23 (4)	F3—Nb—F1	85.23 (3)
O1—Nb—F2	99.19 (5)	F3—Nb—F2	89.84 (5)
O1—Nb—F3	100.50 (5)	F3—Nb—F4	93.19 (5)
O1—Nb—F4	98.35 (5)	F3—Nb—F5	163.95 (4)
O1—Nb—F5	95.41 (4)	F4—Nb—F1	81.83 (3)
F2—Nb—F1	80.08 (3)	F4—Nb—F5	86.54 (4)
F2—Nb—F4	161.34 (3)	F5—Nb—F1	78.84 (3)
F2—Nb—F5	85.52 (4)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2...F1	0.82	1.70	2.5179 (11)	174
N1—H1A...O3 ⁱ	0.89	2.11	2.9619 (12)	160
N1—H1B...O5 ⁱ	0.89	2.16	2.8372 (12)	133
N1—H1B...F2 ⁱⁱ	0.89	2.10	2.7103 (14)	125
N1—H1C...O1 ⁱⁱⁱ	0.89	1.90	2.7898 (14)	173
O4—H4...F1 ^{iv}	0.82	1.73	2.5314 (12)	166
N2—H2A...O3	0.89	2.32	3.1418 (13)	153
N2—H2B...F5	0.89	1.86	2.7457 (13)	178
N2—H2C...F4 ⁱⁱⁱ	0.89	1.99	2.8048 (16)	151

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: CI2394).

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 Zhu, G., Liu, Y.-Y., Wang, G. & Tang, Z.-X. (2005). *Acta Cryst.* **E61**, m1566–m1567.
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supplementary materials

Acta Cryst. (2007). E63, m2086 [doi:10.1107/S1600536807028346]

Bis(DL-valinium) pentafluoridooxidoniobate(V)

M. A. Pushilin, A. V. Gerasimenko and R. L. Davidovich

Comment

Recently, a significant number of organic-inorganic hybrid materials have been prepared and studied because of their important structure-dependent properties, such as non-linear optical activity, piezoelectricity and ferroelectricity. The early transition metal fluorine complexes with out-of-centre distortions in metal oxide fluoride anions are of a special interest. We report here the crystal structure of a new organic-inorganic hybrid compound *bis*(DL-valinium) pentafluorooxononiobate(V), $(C_5H_{12}NO_2)_2[NbOF_5]$ with an isolated ordered $[NbOF_5]^{2-}$ complex anion, (I).

The asymmetric unit of the crystal structure (I) consists of a discrete anionic complex $[NbOF_5]^{2-}$, and two protonated valinium cations (Fig. 1). The Nb atom in $[NbOF_5]^{2-}$ is coordinated by five F atoms and one O atom forming a distorted octahedron. The Nb—F bond *trans* to the Nb—O bond is significantly longer than other four Nb—F bonds in the polyhedron (Table 1).

The Nb atom is displaced from the equatorial plane of the octahedron in the direction to the axial O atom by 0.283 Å. The bond lengths and angles in the Nb-octahedron are comparable with corresponding values observed for discrete and ordered $[NbOF_5]^{2-}$ anions (Sarin *et al.*, 1977; Zhu *et al.*, 2005; Zhu & Tang, 2005).

In the valinium cation the amine group is protonated and carries a positive charge. Four valinium cations are linked by N—H \cdots O intermolecular hydrogen bonds to form a tetrameric associate (Fig. 2) which is linked with $[NbOF_5]^{2-}$ anions by N—H \cdots F, N—H \cdots O and O—H \cdots F hydrogen bonds forming layers parallel to the *ac* plane. Note that there are two very strong O—H \cdots F hydrogen bonds between hydroxyl groups of the valinium cations and the axial fluorine atom F1 of the $[NbOF_5]^{2-}$ anions (Table 2), and a weak interaction C1 \cdots F5ⁱⁱⁱ [2.840 (1) Å] which is little less than the sum of van der Waals radii C and F atoms (3.17 Å) (Bondi, 1964). The layers are connected *via* van der Waals interactions.

Experimental

The title compound was synthesized by the reaction of Nb₂O₅ (1.33 g, 5 mmol) in a solution of hydrofluoric acid (48%, 40 ml) with DL-valine (2.34 g, 20 mmol), in molar stoichiometric ratio DL-valine: Nb₂O₅ = 4:1. The obtained solution was allowed to evaporate slowly by normal temperature. After few days, colorless crystals suitable for X-ray diffraction were obtained. Then they were separated from solution, washed with small amount of acetone and dried in air.

Refinement

After checking their presence in the difference map, all H atoms were placed in geometrically idealized positions and refined in the riding-model approximation, with C—H = 0.96 or 0.98 Å, N—H = 0.89 Å and O—H = 0.82 Å, and $U_{iso}(H) = 1.2$ or 1.5 times $U_{eq}(C,N,O)$.

Figures

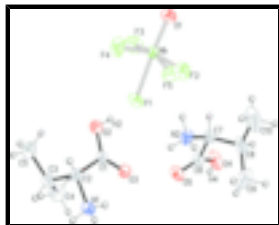


Fig. 1. View of the asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

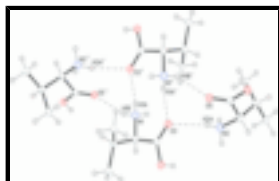


Fig. 2. The tetrameric associate of valinium cations. Hydrogen bonds are shown as dashed lines.

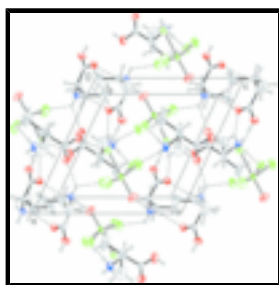


Fig. 3. Part of the polymeric layer of (I), viewed parallel to the *ac* plane. Hydrogen bonds are shown as dashed lines.

(*R*)-1-carboxy-2-methylpropan-1-aminium (*S*)-1-carboxy-2-methylpropan-1-aminium pentafluoridooxidoniobate(V)

Crystal data

(C₅H₁₂NO₂)₂[NbOF₅]

M_r = 440.22

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 9.8018 (4) Å

b = 19.4213 (8) Å

c = 10.1979 (4) Å

β = 116.190 (1)°

V = 1742.01 (12) Å³

Z = 4

*F*₀₀₀ = 896

D_x = 1.679 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 5444 reflections

θ = 3.1–31.4°

μ = 0.76 mm⁻¹

T = 296 (2) K

Prism, colourless

0.32 × 0.31 × 0.30 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

Monochromator: graphite

Detector resolution: 8.33 pixels mm⁻¹

T = 296(2) K

5717 independent reflections

4829 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.037

θ_{max} = 31.5°

ω scans $\theta_{\min} = 3.1^\circ$
 Absorption correction: gaussian $h = -14 \rightarrow 14$
 (SADABS and XPREP in SAINT; Bruker, 2003)
 $T_{\min} = 0.861$, $T_{\max} = 0.907$ $k = -28 \rightarrow 24$
 20336 measured reflections $l = -14 \rightarrow 14$

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
 Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
 $R[F^2 > 2\sigma(F^2)] = 0.030$ H-atom parameters constrained
 $wR(F^2) = 0.078$ $w = 1/[\sigma^2(F_o^2) + (0.0347P)^2 + 0.5786P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.08$ $(\Delta/\sigma)_{\max} = 0.003$
 5717 reflections $\Delta\rho_{\max} = 0.51 \text{ e } \text{\AA}^{-3}$
 214 parameters $\Delta\rho_{\min} = -0.53 \text{ e } \text{\AA}^{-3}$
 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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Nb	0.235617 (10)	0.589755 (5)	0.515321 (10)	0.02581 (2)
F1	0.45989 (8)	0.58700 (4)	0.71168 (8)	0.03843 (19)
F2	0.18570 (9)	0.53106 (5)	0.64105 (9)	0.0547 (2)
F3	0.19702 (10)	0.66821 (5)	0.60355 (10)	0.0596 (3)
F4	0.35143 (9)	0.64148 (5)	0.43684 (9)	0.0515 (2)
F5	0.32661 (10)	0.50686 (5)	0.47622 (9)	0.0497 (2)
O1	0.06771 (12)	0.58416 (5)	0.35884 (11)	0.0463 (3)
O2	0.71168 (9)	0.61373 (5)	0.71263 (9)	0.0353 (2)
H2	0.6328	0.6029	0.7167	0.053*
O3	0.80342 (9)	0.52104 (5)	0.85526 (9)	0.03338 (19)
O4	0.42829 (11)	0.38945 (7)	1.01660 (10)	0.0536 (3)
H4	0.4779	0.3968	1.1043	0.080*
O5	0.62023 (9)	0.44382 (6)	1.00016 (10)	0.0413 (2)

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N1	1.06725 (10)	0.52398 (5)	0.83578 (10)	0.0287 (2)
H1A	1.0846	0.5149	0.9274	0.043*
H1B	1.1551	0.5320	0.8326	0.043*
H1C	1.0213	0.4881	0.7791	0.043*
N2	0.51056 (12)	0.42889 (6)	0.71323 (11)	0.0347 (2)
H2A	0.5830	0.4557	0.7772	0.052*
H2B	0.4513	0.4536	0.6353	0.052*
H2C	0.5531	0.3948	0.6859	0.052*
C1	0.81841 (11)	0.56954 (6)	0.78715 (11)	0.0255 (2)
C2	0.96776 (11)	0.58594 (6)	0.78260 (12)	0.0261 (2)
H2D	0.9470	0.5943	0.6807	0.031*
C3	1.04893 (13)	0.64997 (7)	0.87360 (14)	0.0345 (3)
H3	1.1549	0.6476	0.8893	0.041*
C4	1.05203 (18)	0.65008 (8)	1.02461 (15)	0.0487 (4)
H4A	1.1080	0.6894	1.0788	0.073*
H4B	1.1001	0.6088	1.0758	0.073*
H4C	0.9499	0.6522	1.0142	0.073*
C5	0.98365 (18)	0.71604 (8)	0.79079 (18)	0.0501 (4)
H5A	0.8774	0.7188	0.7667	0.075*
H5B	0.9963	0.7165	0.7026	0.075*
H5C	1.0359	0.7547	0.8505	0.075*
C6	0.50189 (13)	0.41305 (6)	0.94618 (13)	0.0324 (3)
C7	0.41666 (13)	0.39942 (7)	0.78283 (13)	0.0328 (3)
H7	0.3197	0.4242	0.7448	0.039*
C8	0.38306 (15)	0.32244 (7)	0.74971 (13)	0.0407 (3)
H8	0.3218	0.3076	0.7989	0.049*
C9	0.5257 (2)	0.27905 (9)	0.8098 (2)	0.0689 (6)
H9A	0.4985	0.2312	0.7958	0.103*
H9B	0.5834	0.2882	0.9123	0.103*
H9C	0.5857	0.2902	0.7596	0.103*
C10	0.2884 (3)	0.31069 (12)	0.58804 (18)	0.0818 (6)
H10A	0.1964	0.3371	0.5548	0.123*
H10B	0.2637	0.2627	0.5705	0.123*
H10C	0.3449	0.3248	0.5361	0.123*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Nb	0.02638 (4)	0.02999 (5)	0.02207 (4)	-0.00066 (3)	0.01161 (3)	-0.00177 (3)
F1	0.0257 (3)	0.0612 (5)	0.0272 (3)	-0.0028 (3)	0.0105 (2)	-0.0045 (3)
F2	0.0510 (4)	0.0752 (6)	0.0440 (4)	-0.0150 (4)	0.0265 (3)	0.0105 (4)
F3	0.0600 (4)	0.0565 (5)	0.0594 (5)	0.0131 (4)	0.0237 (4)	-0.0211 (4)
F4	0.0620 (4)	0.0553 (5)	0.0488 (4)	-0.0135 (4)	0.0351 (3)	0.0027 (3)
F5	0.0647 (4)	0.0452 (4)	0.0345 (3)	0.0173 (4)	0.0175 (3)	-0.0055 (3)
O1	0.0400 (5)	0.0512 (6)	0.0327 (5)	-0.0015 (4)	0.0023 (4)	-0.0012 (4)
O2	0.0263 (3)	0.0379 (4)	0.0435 (4)	0.0028 (3)	0.0169 (3)	0.0086 (4)
O3	0.0301 (3)	0.0401 (5)	0.0309 (3)	-0.0010 (3)	0.0143 (3)	0.0079 (3)
O4	0.0571 (4)	0.0776 (7)	0.0329 (4)	-0.0320 (5)	0.0260 (3)	-0.0153 (4)

O5	0.0319 (3)	0.0568 (6)	0.0338 (4)	-0.0099 (4)	0.0132 (3)	-0.0030 (4)
N1	0.0251 (3)	0.0345 (5)	0.0286 (4)	0.0003 (3)	0.0137 (3)	-0.0015 (3)
N2	0.0446 (4)	0.0319 (5)	0.0296 (4)	-0.0027 (4)	0.0183 (3)	0.0046 (4)
C1	0.0239 (4)	0.0307 (5)	0.0220 (4)	-0.0017 (4)	0.0102 (3)	-0.0015 (4)
C2	0.0240 (4)	0.0300 (5)	0.0253 (4)	-0.0019 (4)	0.0117 (3)	-0.0007 (4)
C3	0.0298 (4)	0.0339 (6)	0.0387 (5)	-0.0045 (4)	0.0140 (4)	-0.0042 (5)
C4	0.0579 (7)	0.0480 (8)	0.0354 (6)	-0.0046 (6)	0.0163 (5)	-0.0123 (6)
C5	0.0543 (7)	0.0331 (7)	0.0589 (8)	-0.0042 (6)	0.0213 (6)	0.0017 (6)
C6	0.0347 (4)	0.0356 (6)	0.0284 (5)	-0.0025 (4)	0.0153 (4)	-0.0026 (4)
C7	0.0312 (4)	0.0395 (6)	0.0270 (5)	-0.0003 (4)	0.0121 (4)	0.0034 (4)
C8	0.0515 (6)	0.0426 (7)	0.0284 (5)	-0.0182 (5)	0.0182 (4)	-0.0031 (5)
C9	0.0978 (12)	0.0320 (8)	0.0783 (11)	0.0037 (8)	0.0402 (9)	0.0043 (7)
C10	0.1092 (13)	0.0918 (13)	0.0339 (7)	-0.0555 (11)	0.0221 (8)	-0.0165 (8)

Geometric parameters (Å, °)

Nb—O1	1.7172 (9)	C2—H2D	0.98
Nb—F3	1.8906 (9)	C3—C5	1.5140 (19)
Nb—F2	1.9331 (9)	C3—C4	1.527 (2)
Nb—F4	1.9339 (9)	C3—H3	0.98
Nb—F5	1.9648 (9)	C4—H4A	0.96
Nb—F1	2.2270 (7)	C4—H4B	0.96
O2—C1	1.3065 (13)	C4—H4C	0.96
O2—H2	0.82	C5—H5A	0.96
O3—C1	1.2170 (15)	C5—H5B	0.96
O4—C6	1.3053 (17)	C5—H5C	0.96
O4—H4	0.82	C6—C7	1.5214 (16)
O5—C6	1.2008 (15)	C7—C8	1.5361 (19)
N1—C2	1.4920 (14)	C7—H7	0.98
N1—H1A	0.89	C8—C10	1.510 (2)
N1—H1B	0.89	C8—C9	1.511 (2)
N1—H1C	0.89	C8—H8	0.98
N2—C7	1.5018 (17)	C9—H9A	0.96
N2—H2A	0.89	C9—H9B	0.96
N2—H2B	0.89	C9—H9C	0.96
N2—H2C	0.89	C10—H10A	0.96
C1—C2	1.5188 (15)	C10—H10B	0.96
C2—C3	1.5456 (16)	C10—H10C	0.96
O1—Nb—F1	174.23 (4)	C4—C3—H3	106.7
O1—Nb—F2	99.19 (5)	C2—C3—H3	106.7
O1—Nb—F3	100.50 (5)	C3—C4—H4A	109.5
O1—Nb—F4	98.35 (5)	C3—C4—H4B	109.5
O1—Nb—F5	95.41 (4)	H4A—C4—H4B	109.5
F2—Nb—F1	80.08 (3)	C3—C4—H4C	109.5
F2—Nb—F4	161.34 (3)	H4A—C4—H4C	109.5
F2—Nb—F5	85.52 (4)	H4B—C4—H4C	109.5
F3—Nb—F1	85.23 (3)	C3—C5—H5A	109.5
F3—Nb—F2	89.84 (5)	C3—C5—H5B	109.5
F3—Nb—F4	93.19 (5)	H5A—C5—H5B	109.5

supplementary materials

F3—Nb—F5	163.95 (4)	C3—C5—H5C	109.5
F4—Nb—F1	81.83 (3)	H5A—C5—H5C	109.5
F4—Nb—F5	86.54 (4)	H5B—C5—H5C	109.5
F5—Nb—F1	78.84 (3)	O5—C6—O4	125.39 (12)
C1—O2—H2	109.5	O5—C6—C7	122.76 (12)
C6—O4—H4	109.5	O4—C6—C7	111.81 (10)
C2—N1—H1A	109.5	N2—C7—C6	107.23 (9)
C2—N1—H1B	109.5	N2—C7—C8	112.56 (11)
H1A—N1—H1B	109.5	C6—C7—C8	111.78 (10)
C2—N1—H1C	109.5	N2—C7—H7	108.4
H1A—N1—H1C	109.5	C6—C7—H7	108.4
H1B—N1—H1C	109.5	C8—C7—H7	108.4
C7—N2—H2A	109.5	C10—C8—C9	111.80 (16)
C7—N2—H2B	109.5	C10—C8—C7	110.84 (12)
H2A—N2—H2B	109.5	C9—C8—C7	112.54 (11)
C7—N2—H2C	109.5	C10—C8—H8	107.1
H2A—N2—H2C	109.5	C9—C8—H8	107.1
H2B—N2—H2C	109.5	C7—C8—H8	107.1
O3—C1—O2	124.96 (10)	C8—C9—H9A	109.5
O3—C1—C2	122.72 (9)	C8—C9—H9B	109.5
O2—C1—C2	112.31 (10)	H9A—C9—H9B	109.5
N1—C2—C1	107.69 (9)	C8—C9—H9C	109.5
N1—C2—C3	110.49 (8)	H9A—C9—H9C	109.5
C1—C2—C3	113.45 (10)	H9B—C9—H9C	109.5
N1—C2—H2D	108.4	C8—C10—H10A	109.5
C1—C2—H2D	108.4	C8—C10—H10B	109.5
C3—C2—H2D	108.4	H10A—C10—H10B	109.5
C5—C3—C4	112.13 (13)	C8—C10—H10C	109.5
C5—C3—C2	111.61 (10)	H10A—C10—H10C	109.5
C4—C3—C2	112.49 (11)	H10B—C10—H10C	109.5
C5—C3—H3	106.7		

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>
O2—H2...F1	0.82	1.70	2.5179 (11)	174
N1—H1A...O3 ⁱ	0.89	2.11	2.9619 (12)	160
N1—H1B...O5 ⁱ	0.89	2.16	2.8372 (12)	133
N1—H1B...F2 ⁱⁱ	0.89	2.10	2.7103 (14)	125
N1—H1C...O1 ⁱⁱⁱ	0.89	1.90	2.7898 (14)	173
O4—H4...F1 ^{iv}	0.82	1.73	2.5314 (12)	166
N2—H2A...O3	0.89	2.32	3.1418 (13)	153
N2—H2B...F5	0.89	1.86	2.7457 (13)	178
N2—H2C...F4 ⁱⁱⁱ	0.89	1.99	2.8048 (16)	151

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

Fig. 1

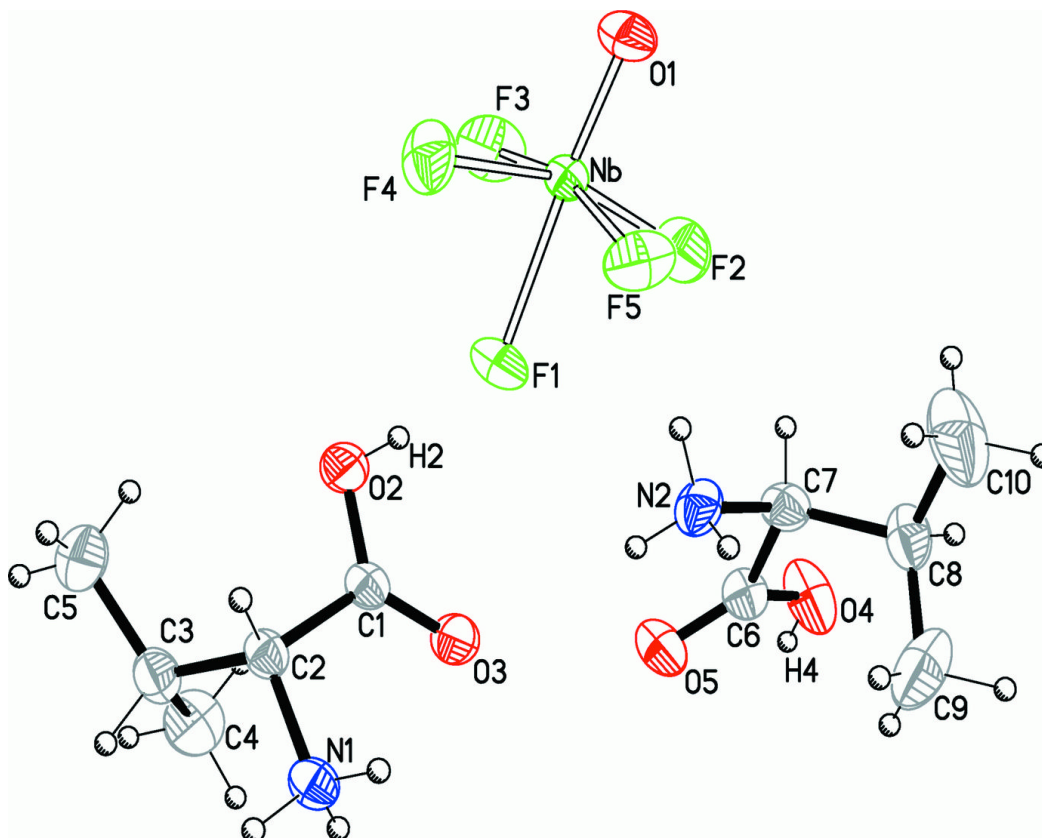


Fig. 2

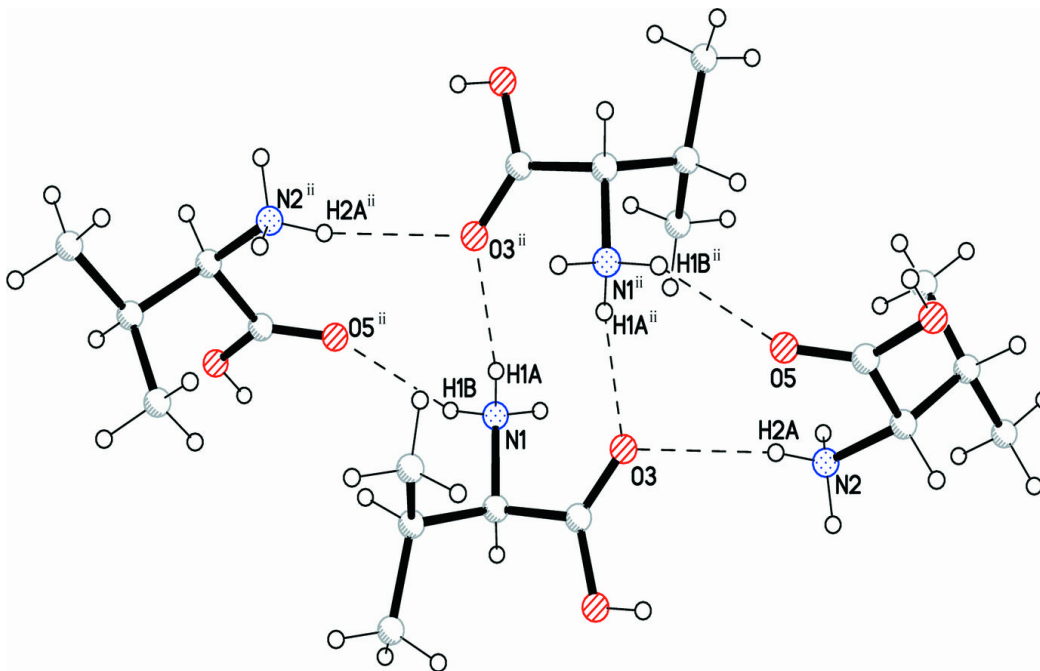


Fig. 3

