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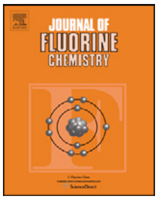

Short communication

The missing crystal structures of fluorosulfates of monovalent cations: $M(I)SO_3F$, M = Na, Rb and Tl

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This work is dedicated to Professor Zbigniew Ryszard Grabowski at his 85th birthday.

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A B S T R A C T

Anhydrous fluorosulfates of sodium(I) and thallium(I) have been structurally characterized for the first time; structure of a novel polymorphic form of rubidium(I) fluorosulfate is also reported. $NaSO_3F$ is hexagonal ($P6_3/mmc$, $a = b = 5.4812(2)$ Å, $c = 6.5172(3)$ Å, $Z = 2$), while $RbSO_3F$ and $TlSO_3F$ are orthorhombic an both adopt the *Pnma* space group, but despite similar ionic radii of metal cations they are not isostructural with each other (Rb: $a = 8.7812(7)$ Å, $b = 6.0318(6)$ Å, $c = 7.5108(6)$ Å, $Z = 4$; Tl: $a = 5.2205(3)$ Å, $b = 5.5141(2)$ Å, $c = 13.4378(7)$ Å, $Z = 4$). The three fluorosulfates studied here complete the list of fluorosulfates of monovalent metals, for which the crystal structures have been determined.

1. Introduction

Fluorosulfate anion may be considered as a derivative of hydrogensulfate anion, $HOSO_3^-$, in which the hydroxyl group has been substituted by isoelectronic F atom [1]. Fluorine substituent is much more electron withdrawing than OH moiety and it exercises significant effects on the charge distribution. In consequence, fluorosulfate anion is hard to oxidize [2] and simultaneously it is rather weakly coordinating thus giving rise to a broad range of useful ionic liquids containing organic cations. Tetrabutylammonium fluorosulfate is solid at ambient conditions, but due to its solubility in a number of organic solvents it is a versatile phase transfer catalyst. Metal fluorosulfates [3] constitute another large and important group of fluorosulfate derivatives, used predominantly in catalysis [4]. Due to substantial ionicity of metal–ligand bonding and concomitant large positive charge on metal cations, they are used mainly as solid Lewis acid catalysts [5] but also as fluorinating agents in organic chemistry [6] and oxidizers [7]. Fluorosulfates of monovalent metals constitute one important subgroup, as they are utilized for fabrication of substituted (per)fluoropolyethers [8], inorganic binders for electrode materials [9], starch sulfate esters [10] and as electrolyte additives [11–13]. While majority of monovalent metal fluorosulfates have been structurally characterized to date (Li [14], K [15], Rb [16], Cs [17–19], and Ag [20]) it is surpsiring that two important members of this series, the sodium and thallium(I) salts, have not. This communication seeks to fill this gap by reporting the crystal structures of MSO_3F (M = Na, Tl) from single crystal X-ray measurements. We also describe a novel polymorphic β-form of Rb(I) fluorosulfate.

2. Material and methodology

Fluorosulfates of Na, Rb and Tl were obtained from NaF, RbF and $TlNO_3$ precursors, respectively, by dissolution in anhydrous HSO_3F (all reagents from Aldrich). Single crystals were grown from solutions of MSO_3F in HSO_3F. We have not observed the formation of $MH(SO_3F)_2$ in any of these systems.

The single crystals of MSO_3F compounds were sealed in glass capillaries to protect them from moisture and than mounted on the goniometer of the diffractometer equipped with LT device. During measurements of all the crystals the temperature was set to 100 K to protect the crystals from possible decomposition. All measurements were performed on a KM4CCD κ-axis diffractometer with

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graphite-monochromated MoK$_\alpha$ radiation. The data were corrected for Lorentz and polarization effects. Empirical correction for absorption was applied in the case of Na salt [21]. The data corresponding to highly absorbing crystals of 2 and 3 were corrected for the absorption based on the crystals shape [22]. Data reduction and analysis were carried out with the Oxford Diffraction programs [23]. The structure was solved by direct methods [24] and refined using SHELXL [25]. The refinement was based on F2. Scattering factors were taken from Tables 6.1.1.4 and 4.2.4.2 in [26]. Further details of the crystal structures at 100 K may be obtained from Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany (fax: +49 7247 808 666; crysdata@fizkarlsruhe.de) on quoting the CSD numbers 424112 ($NaSO_3F$), 424113 (β-$RbSO_3F$) and 424114 ($TlSO_3F$). Diamond was used for structure drawings [27].

3. Results and discussion

Sodium fluorosulfate adopts the $P6_3/mmc$ space group and is the only hexagonal fluorosulfate of monovalent cation known to date (Fig. 1, Table 1). $NaSO_3F$ contains disordered fluorosulfate anions in that manner the whole moiety looks like trigonal bipyramid sharing three oxygen atom as the base. However the central S atom is split over two positions with occupancies yielding 50% each; so is the terminal F atom. The crystal structure of $NaSO_3F$ is related to that of the disordered high-temperature form of Na_2SO_4 (Fig. 1). The $NaSO_3F$-like structure is obtained from hexagonal Na_2SO_4 if half of Na atoms (the ones at special position 2/3, 1/3, 1/4) are removed. The Na^+ cation in $NaSO_3F$ is coordinated by six O atoms in the form of a distorted octahedron, with six Na–O separations of 2.4004(10) Å and the O–Na–O angles varying

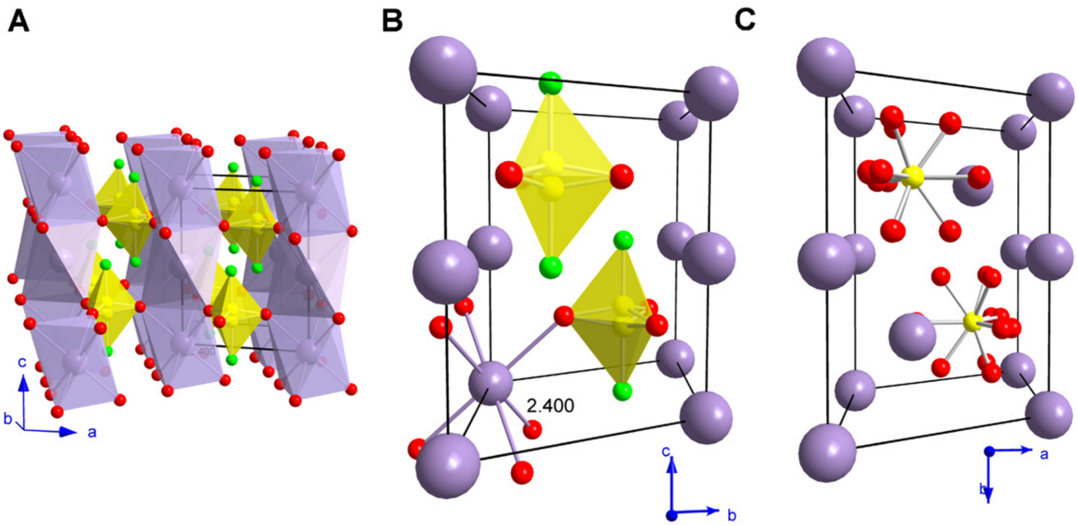

Fig. 1. Three views of the crystal structure of $NaSO_3F$ and its relation to that of the high-temperature form of Na_2SO_4. (A) $NaSO_3F$: the projection emphasizing the presence of infinite chains composed of face-sharing [NaO_6] octahedra, running parallel to crystallographic *c* axis. (B) $NaSO_3F$: the crystallographic unit cell (note the disorder of the SO_3F anions) and the first coordination sphere of Na^+. (C) The crystallographic unit cell of the high-temperature form of Na_2SO_4. S – yellow, O – red, Na – large violet, and F – small green balls. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

Table 1

Crystal data for $NaSO_3F$, β-$RbSO_3F$ and $TlSO_3F$.

Empirical formula	$NaSO_3F$	$RbSO_3F$	$TlSO_3F$
M	122.05	184.53	303.43
T	100(2) K	100(2) K	100(2) K
λ	0.71073 Å	0.71073 Å	0.71073 Å
Crystal system, space group	Hexagonal, $P6_3/mmc$	Orthorhombic, $Pnma$	Orthorhombic, $Pnma$
Unit cell dimensions	a = 5.4812(2) Å	a = 8.7812(7) Å	a = 5.2205(3) Å
	b = 5.4812(2) Å	b = 6.0318(6) Å	b = 5.5141(2) Å
	c = 6.5172(3) Å	c = 7.5108(6) Å	c = 13.4378(7) Å
V	169.568(12) Å^3	397.82(6) Å^3	386.83(3) Å^3
Z, D_x	2, 2.390 g cm^{-3}	4, 3.081 g cm^{-3}	4, 5.210 g cm^{-3}
μ	0.940 mm^{-1}	12.851 mm^{-1}	42.170 mm^{-1}
$F_{(0\,0\,0)}$	120	344	520
Crystal size	0.15 mm × 0.10 mm × 0.10 mm	1.00 mm × 0.09 mm × 0.09 mm	0.39 mm × 0.36 mm × 0.17 mm
θ range for data collection	4.29–27.92°	3.57–29.99°	3.03–29.50°
Reflections collected/unique	3267/100 [R_{int} = 0.0170]	6458/634 [R_{int} = 0.0617]	3680/591 [R_{int} = 0.0433]
Completeness	100%	100%	99.5%
Absorption correction	Semi-empirical	Analytical	Analytical
Max. and min. transmission	0.912 and 0.801	0.415 and 0.027	0.035 and 0.005
Refinement method	Full-matrix LSQ on F^2	Full-matrix LSQ on F^2	Full-matrix LSQ on F^2
Data/restraints/parameters	100/0/14	634/0/35	591/0/35
Goodness-of-fit on F^2	1.302	1.247	1.193
Final R indices [$I > 2\sigma(I)$]	$R1$ = 0.0176, $wR2$ = 0.0554	$R1$ = 0.0329, $wR2$ = 0.0707	$R1$ = 0.0269, $wR2$ = 0.0708
R indices (all data)	$R1$ = 0.0177, $wR2$ = 0.0555	$R1$ = 0.0412, $wR2$ = 0.0746	$R1$ = 0.0279, $wR2$ = 0.0714
Extinction coefficient	0.27(4)	0.0142(19)	0.0060(6)
Largest diff. peak and hole	0.254 and −0.273 e A^{-3}	1.682 and −0.821 e A^{-3}	3.011 and −2.132 e A^{-3}

Table 2

Bonding and some non-bonding metal-F/O contacts in structures of $NaSO_3F$, β-$RbSO_3F$ and $TlSO_3F$. For valence bond sums calculation for $TlSO_3F$ refer to ESI.

$NaSO_3F$		β-$RbSO_3F$		$TlSO_3F$	
Na(1)···O(2)	2.4004(10)	Rb(1)···O(3)#1	2.918(3)	Tl(1)···O(1)	3.0479(33)
Na(1)···F(1)	3.1699(2)	Rb(1)···O(2)|F(2)#2	2.929(4)	Tl(1)···O(1)#1	2.9806(76)
		Rb(1)···O(3)#3	2.970(3)	Tl(1)···O(2)	3.2298(61)
		Rb(1)···O(1)|F(1)	2.984(4)	Tl(1)···O(2)#2	2.9456(57)
		Rb(1)···O(3)#4	3.089(3)	Tl(1)···O(2)#3	2.9717(62)
		Rb(1)···O(1)|F(1)#3	3.3104(16)	Tl(1)···F(1)#1	3.7013(74)
				Tl(1)···F(1)#2	3.5735(47)
				Tl(1)···F(1)#4	3.1934(66)
		Symmetry used to generate equivalent atoms: #1 $-x+1, y+1/2, -z+1$; #2 $x-1/2, y, -z+1/2$; #3 $-x+1/2, -y, z-1/2$; #4 $x, y, z-1$.		Symmetry used to generate equivalent atoms: #1 $-x+1, 0.5+y, -z+1$; #2 $x-1, y, z$; #3 $x-0.5, y, -z+1.5$; #4 $-x+2, y+0.5, -z+1$	

between *ca.* 79° and *ca.* 101° (Fig. 1, Table 2). The Na–O bond length is comparable to those found for other oxo-derivatives of Na(I) with coordination number of 6 (*cf.* $NaNO_3$ 2.400–2.410 Å, low-temperature Fddd form of Na_2SO_4: 2.334–2.538 Å). The $[NaO_6]$ octahedra share faces while forming infinite 1D $[NaO_{6/2}]$ chains. The Na–F separation of 3.1699(2) Å is too long to be considered a bonding distance.

β-$RbSO_3F$ takes orthorhombic *Pnma* structure (Fig. 2, Table 1), and it is isostructural to its K (Fig. 2) and NH_4 analogs. The Rb^+ cation in β-$RbSO_3F$ is coordinated by eight O atoms, with the Rb–O separations of 2× 2.918(3) Å, 1× 2.929(4) Å, 2× 2.970(3) Å, 1× 2.984(4) Å, and 2× 3.089(3) Å (Fig. 2 and Table 2; we have not taken into account the long Rb–O/F separation of 3.3104(16) Å). The Rb–O bond lengths are comparable to those found for other oxo-derivatives of Rb(I) with coordination number of 8 (*cf.* $RbHSO_4$ 2.964–3.270 Å). β-$RbSO_3F$ is disordered and contains fluorosulfate anions exhibiting substitutional O|F disorder. Thus the anion has two ordered O atoms, as well as two mixed O|F sites with 0.5:0.5 occupancy ratios of O and F atoms; such type of O/F disorder is typical, for example, for KSO_3F and $AgSO_3F$. The $[RbO_8]$ polyhedra share edges and corners while forming complex structure. The β polymorph has considerably smaller volume, by nearly 10%, than the known triclinic α-$RbSO_3F$ [16]. The less compact α-form is characerized by a larger coordination number of rubidium (11) as compared to β-$RbSO_3F$ (8).

$TlSO_3F$ adopts the orthorhombic *Pnma* space group with 4 formula units per unit cell (Fig. 3, Table 1); structural disorder is absent. The volume of one formula unit, 96.706(5) $Å^3$, is very similar to those of fluorosulfates of K^+ and H_3O^+ (both also *Pnma*). However, $TlSO_3F$ is not isostructural with its potassium and oxonium analogs. Crystal structure of $TlSO_3F$ with most closely resembles that of tetragonal $CsSO_3F$ [19] (Fig. 3). The coordination sphere of Tl(I) is highly irregular with seven O atoms in its vicinity, and the Tl–O separations of 2× 2.9456(57) Å, 2× 2.9717(62) Å, 1× 2.9806(76) Å, and 2× 3.0479(33) Å (Fig. 3C, Table 2). The range of Tl–O bond lengths is typical for oxo-connections of this cation with coordination number of 7 (*cf.* orthorhombic Tl_2SO_4 2.905–3.261 Å). One Tl–F contact at 3.1934(66) Å falls below the sum of the van der Waals radii of these atoms (3.43 Å). Irregular coordination sphere of Tl(I) originates from the stereochemical activity of the $6sp^2$ lone pair, as typical for most compounds of monovalent thallium. The $[TlO_7]$ polyhedra share edges to form a complex structure.

The binding mode (ligand dentity) of fluorosulfate anion [28,29] is of interest. Fluorosulfate anion is tridentate for $NaSO_3F$ and $TlSO_3F$, and it does not use its F atom for binding to metal. The situation is different for $RbSO_3F$, where F atom is engaged in ionic bonding to metal cation on equal pace with all three oxygen atoms. The terminal F atoms stick toward each other in $NaSO_3F$ at the F···F separation of *ca.* 2.89 Å (which is slightly shorter than twice the van der Waals radius of F atom, 2.94 Å). In case of $TlSO_3F$, the terminal F atoms form 1D channels with equidistant F atoms at *ca.* 3.105 Å. For $RbSO_3F$ the situation is more complex due to O/F disorder; still, the 1D channels made up of F atoms can be distinguished in the structure, with the closest F···F separation of

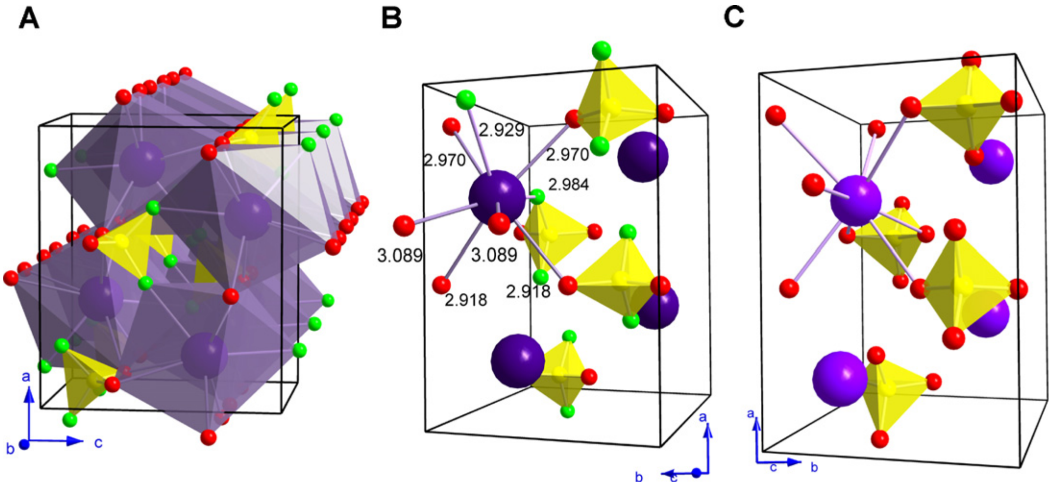

Fig. 2. Three views of the crystal structure of β-$RbSO_3F$ and its relation to that of KSO_3F. (A) β-$RbSO_3F$: the projection emphasizing the presence of $[Rb(O/F)_8]$ polyhedra. (B) The crystallographic unit cell of β-$RbSO_3F$ and the first coordination sphere of Rb; note the substitutional O/F disorder as emphasized here by drawing of 2 F atoms at one S atom. (C) The crystallographic unit cell of related KSO_3F. S – yellow, O – red, K – light violet, Rb – dark violet, and F – small green balls. Here, substitutional O/F disorder has not been marked. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

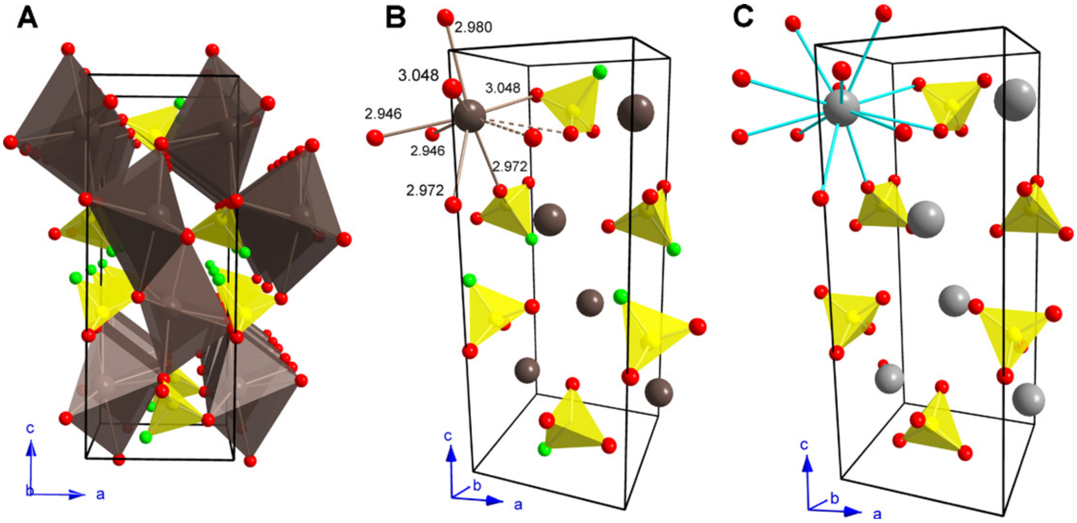

Fig. 3. Two views of the crystal structure of $TlSO_3F$ and its relation to that of the tetragonal form of $CsSO_3F$. (A) $TlSO_3F$: the projection emphasizing the presence of the $[TlO_7]$ polyhedra linked in infinite 1D chains. (B) $TlSO_3F$: the crystallographic unit cell and the first coordination sphere of Tl^+. (C) The crystallographic unit cell of the tetragonal form of $CsSO_3F$. Here, substitutional O/F disorder has not been marked. S – yellow, O – red, Tl – large brown, Cs – large gray, and F – small green balls. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

3.401 Å. Similar 'self-assemble' of F sublattice has been frequently observed for fluorosulfates and for related triflates; for $AgSO_3F$ and $LiSO_3F$ the F atoms arrange themselves in 2D sheets, while for KSO_3F and NH_4SO_3F the 1D arrangement is seen.

The data of crystal structures of all monovalent metal cations synthesized to date, supplemented by information for related H^+ [30], H_3O^+ [31], NH_4^+ [32] and NMe_4^+ [33] derivatives, are jointly shown in Table 3. The structures were arranged according to the increasing volume per formula unit, V_{FU}. The V_{FU} ranges from 76.7(4) $Å^3$ for HSO_3F to 114.43(7) $Å^3$ for the monoclinic form of $CsSO_3F$, while the coordination number of cation varies from 1 + 1 for HSO_3F to as much as 12 for the tetragonal form of $CsSO_3F$. It can be concluded that fluorosulfates of monovalent inorganic cations [34] exhibit large structural diversity, with one triclinic (P-1), three monoclinic ($P2_1/m$, C2/m, P21/a), three orthorhombic ($P2_12_12_1$, and two *Pnma*), one tetragonal ($I4_1/amd$) and one hexagonal ($P6_3/mmc$) distinct structure types. This diversity comes not only from the varying volume of cation but it is also related to the possibility of O/F disorder (as for example for Na, K, Rb and ammonium salts) and – as in the case of HSO_3F – to the reduced dentity of the fluorosulfate anion.

Table 3
Comparison of the unit cell parameters for fluorosulfates of monovalent metal cations, MSO_3F (M = Li···Cs, Ag, Tl) with H^+, H_3O^+, NH_4^+ and NMe_4^+ derivatives shown for comparison; V_{FU} and CN_C stand for volume per formula unit (FU) and coordination number of a cation, respectively. Arrangement from the left to the right is according to the increasing V_{FU}. For H_3O^+, NH_4^+ and NMe_4^+ salts CN_C is defined by the smallest polyhedron made of S atoms and needed to enclose the cation. RT, room temperature and ND, not determined.

M	H	Li	Ag	Na	K	Tl	H_3O
Reference	[24]	[14]	[20]	This work	[15]	This work	[25]
Space group	$P2_12_12_1$ (no. 19)	$C2/m$ (no. 12)	$P2_1/m$ (no. 11)	$P6_3/mmc$ (no. 194)	*Pnma* (no. 62)	*Pnma* (no. 62)	*Pnma* (no. 62)
T (K)	123	RT?	100	100	RT?	100	RT?
a (Å)	4.868(4)	8.54(2)	5.4128(10)	5.4812(2)	8.62	5.2205(3)	8.055(5)
b (Å)	6.736(5)	7.62(1)	8.1739(14)	5.4812(2)	5.84	5.5141(2)	6.465(4)
c (Å)	9.359(7)	4.98(2)	7.5436(17)	6.5172(3)	7.35	13.4378(7)	7.459(4)
α (°)	90	90	90	90	90	90	90
β (°)	90	90.0(3)	94.599(18)	90	90	90	90
γ (°)	90	90	90	120	90	90	90
Z (FU)	4	4	4	2	4	4	4
V_{FU} ($Å^3$)	76.7(4)	81.0(16)	83.17(3)	84.784(3)	92.50	96.706(5)	97.1(4)
CN_C	1 + 1	4	6	6	8	7	6
M	Rb	NH_4	NMe_4	Rb	Cs	Cs	Cs
Reference	This work	[26]	[27]	[16]	[17]	[18]	[19]
Space group	*Pnma* (no. 62)	*Pnma* (no. 62)	ND	*P-1* (no. 2)	$I4_1/a$ (no. 88)[a]	$I4_1/amd$ (no. 141)	$P2_1/a$ (no.14)
T (K)	100	RT?	RT?	RT?	RT?	RT?	?
a (Å)	8.7812(7)	8.972(10)	8.38	5.503(2)	5.611(1)	5.6317(2)	7.7243(6)
b (Å)	6.0318(6)	5.996(10)	8.38	5.623(1)	5.611(1)	5.6317(2)	8.1454(6)
c (Å)	7.5108(6)	7.542(10)	5.86	7.603(3)	14.133(9)	14.1991(9)	7.7839(7)
α (°)	90	90	90	100.64(2)	90	90	90
β (°)	90	90	90	106.72(2)	90	90	110.832(7)
γ (°)	90	90	90	93.22(2)	90	90	90
Z (FU)	4	4	4	2	4	4	4
V_{FU} ($Å^3$)	99.455(7)	101.43(10)	102.88	109.97(13)	111.3(3)	112.59(4)	114.43(7)
CN_C	8	6	ND	11	ND	12	10

[a] Likely isostructural to the $I4_1/amd$ form.

4. Conclusions

Anhydrous fluorosulfates of sodium(I) and thallium(I) have been structurally characterized for the first time. The β-form of $RbSO_3F$ is isostructural to KSO_3F and much more densely packed than the known triclinic α-form. $TlSO_3F$ and $NaSO_3F$ adopt unique structures types, not been seen before for any fluorosulfate derivative, thus contributing to enrichment of the structural diversity in this family of compounds. It is now of interest whether fluorosulfates of Au(I), Cu(I), In(I), Ga(I) and possibly even that of Al(I) could be prepared and what crystal structures they would adopt.

Electronic supplementary information (ESI)

This paper is associated by ESI where further projections of parking motiffs and thermal ellipsoids are shown.

Acknowledgements

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.jfluchem.2012.05.004.

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