Journal of Fluorine Chemistry 132 (2011) 767–771

Contents lists available at ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

Syntheses, structures and Raman spectra of $Cd(BF_4)(AF_6)$ (A = Ta, Bi) compounds

Kristian Radan, Matic Lozinšek, Evgeny Goreshnik, Boris Žemva *

Jožef Stefan Institute, Jamova cesta 39, SI-1000, Ljubljana, Slovenia

ARTICLE INFO

Article history:
Received 14 April 2011
Received in revised form 25 May 2011
Accepted 30 May 2011
Available online 6 June 2011

Keywords:
Tetrafluoroborate
Hexafluorotantalate(V)
Hexafluorobismuthate(V)
Crystal structure
Ternary salts
Mixed fluoroanions
Raman spectra

We dedicate this paper to our colleague fluorine chemist and a good friend Professor Alain Tressaud in honour of his receiving the 2011 ACS Award for Creative Work in Fluorine Chemistry.

ABSTRACT

The compounds, $Cd(BF_4)(TaF_6)$ and $Cd(BF_4)(BiF_6)$, have been synthesized and characterized by single-crystal X-ray diffraction and Raman spectroscopy. Both isostructural compounds crystallize in the monoclinic $P2_1/c$ space group with $a = 8.2700(6)$ Å, $b = 9.3691(6)$ Å, $c = 8.8896(7)$ Å, $\beta = 94.196(3)°$, $V = 686.94(9)$ Å^3 for $Cd(BF_4)(TaF_6)$ and $a = 8.3412(8)$ Å, $b = 9.4062(8)$ Å, $c = 8.9570(7)$ Å, $\beta = 93.320(5)°$, $V = 701.58(11)$ Å^3 for $Cd(BF_4)(BiF_6)$. Eight fluorine atoms ($4\,BF_4^- + 4\,AF_6^-$) form a surrounding around the cadmium atom in the shape of distorted square antiprism. These compounds are not isostructural with mixed-anion analogues of Ca, Sr, Ba and Pb studied earlier.

1. Introduction

To enable the coordination with weak fluoroligands like HF [1], AsF_3 [2], XeF_2 [3] and XeF_4 [4], the metal center needs to be practically naked. This is facilitated by the use of weakly coordinating fluoroanions [5,6], such as PF_6^-, AsF_6^- and SbF_6^-. It turns out that fluoroanions of different geometries can be packed very efficiently in the crystal lattice as it was shown in our recent report about the preparation and structural investigation of the first salts of divalent alkaline earth metals containing two perfluorinated anions with the same charge—tetrahedral BF_4^- and octahedral PF_6^- or AsF_6^- [7]. Examples of salts simultaneously containing tetrahedral and octahedral anions are known but most have complex cations [8]. Recently the salts with octahedral AsF_6^- and icosahedral $B_{12}F_{12}^{2-}$ anions have also been published [9].

The crystal-chemical analysis of known structures and the formulation of crystallographic rules is an important aspect in the quest for crystal structure prediction [10]. Therefore, we have synthesized and characterized, by single-crystal X-ray diffraction and by Raman spectroscopy, a variety of $M^{II}(BF_4)(A^VF_6)$ derivatives of Ca, Sr, Ba and Pb with a wide range of octahedral anions [11]. It is amazing, that all these compounds except the PF_6^- derivatives are isostructural. An attempt to prepare similar types of cadmium salts resulted in the synthesis of two new $Cd(BF_4)(TaF_6)$ and $Cd(BF_4)(BiF_6)$ compounds that crystallize as a different structural type. The syntheses and characterization of these salts are described in this report.

2. Results and discussion

2.1. Synthesis

The compounds of the $M^{II}(BF_4)(A^VF_6)$ family crystallize from the anhydrous HF (aHF) solutions in which M^{2+}, BF_4^- and AF_6^- ions are present. This is achieved by dissolving an equimolar amount of the corresponding $M(BF_4)_2$ and $M(AF_6)_2$ salts in aHF as in the case of $Cd(BF_4)(BiF_6)$ (i) or by preparing the AF_6^- anion in situ in the reaction between AF_5 and MF_2 in a 2:1 mole ratio as it is demonstrated in the synthesis of $Cd(BF_4)(TaF_6)$ derivative (ii). However, both reaction routes are equivalent and yield the desired product, $M(BF_4)(AF_6)$.

$$Cd(BF_4)_2 + Cd(BiF_6)_2 \rightarrow 2Cd(BF_4)(BiF_6) \quad (i)$$

$$Cd(BF_4)_2 + CdF_2 + 2TaF_5 \rightarrow 2Cd(BF_4)(TaF_6) \quad (ii)$$

* Corresponding author. Tel.: +386 1 477 35 40; fax: +386 1 477 31 55.
E-mail address: boris.zemva@ijs.si (B. Žemva).

0022-1139/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.jfluchem.2011.05.033

2.2. Crystal structure

The crystallographic data and the selected interatomic distances and bond angles are listed in Table 1 and Table 2, respectively. In the structures of $Cd(BF_4)(AF_6)$ (A = Ta, Bi), the coordination surrounding of the cadmium atom is comprised of eight fluorine atoms in the shape of slightly distorted square antiprisms (Fig. 1). The dihedral angle characteristic of a twisted square antiprism is 43° in the tantalum and 42° in the bismuth derivative instead of 45° for an ideal square anti-prismatic geometry. The Cd–F distances lie in the range of 2.271(8)–2.441(9) Å with an average bond length of 2.324 Å for the tantalum compound and in the range of 2.27(2)–2.39(2) Å, (2.32 Å average) for the bismuth compound. Four F atoms belonging to 4 BF_4^- units, and other four are from 4 AF_6^- anions. The F atoms from the BF_4^- anions are closer to the Cd atom with an average distance of 2.298 Å compared to the F atom distances for the TaF_6^- anions, which have an average distance of 2.350 Å. A similar situation is observed in the bismuth compound with average Cd–F distances of 2.29 Å and 2.35 Å for BF_4^- and BiF_6^-, respectively. This is in agreement with the fact that the F atoms of the BF_4^- anion bear higher partial charge than F atoms of the AF_6^-. The sum of all eight bond valences [12] for the Cd atom in $Cd(BF_4)(TaF_6)$ is 2.02 with contributions of 1.073 from BF_4^- and 0.947 from TaF_6^-. Similarly, for the bismuth compound, the F atoms of the BF_4^- anions contribute 1.098 and the BiF_6^- groups contribute 0.947, which amounts to 2.045 for the sum of all eight Cd–F bond valences.

Each BF_4^- anion is coordinated to four cadmium centres, resulting in similar B–F bond lengths for all four bonds (Table 2), with an average of 1.39 Å in the tantalum compound and 1.40 Å in the bismuth one. The F–B–F angles lie in the range 108.2–110.5° with an average of 109.5° and in 105–113° range, that also averages to 109° in $Cd(BF_4)(TaF_6)$ and $Cd(BF_4)(BiF_6)$, respectively.

Contrary to the earlier studied Ca, Sr, Ba and Pb derivatives [11], where the AF_6^- anions exhibit coordination numbers 5 or 5 + 1, in the case of the cadmium salts, each AF_6^- unit is connected only to 4 Cd atoms. The terminal F5 and F9 atoms are *cis* to one another (Fig. 1). The Ta–F(terminal) bonds of 1.852(9) Å and 1.849(11) Å are noticeably shorter than the Ta–F(bridging) bonds which lie in range l.907(9)–1.949(9) Å. As a result, TaF_6^- anion is distorted from O_h symmetry. Such a distortion is smaller in the bismuth derivative, where Bi–F bond lengths for the terminal F5 and F9 atoms are 1.99(2) Å and 1.94(2) Å, and for the bridging F atoms they are in the range of 1.96(2)–2.03(2) Å. The average Ta–F and Bi–F bond lengths are 1.901 Å and 1.99 Å, respectively. In the TaF_6^- anion, the F–Ta–F *cis*-angles range from 87.2° to 93.5° with an average of 90.0°, while the average *trans*-angle is 176.0°. Similarly, the F–Bi–F average *trans*-angle is 175.3° and the average *cis*-angle is 90.0° for a range of 86.2–94.3°.

A bridging function of both tetrahedral and octahedral anions result in 3-D networks (Fig. 2). This packing appears to be very efficient because the compound that crystallizes is the mixed-anion salt $Cd(BF_4)(AF_6)$ and not $Cd(BF_4)_2$ or $Cd(AF_6)_2$. $Cd(BF_4)(AF_6)$ are, in fact, more densely packed with 84.9% of space occupied by atoms in the unit cell for the tantalum and 85.9% for the bismuth as compared to the packing index of 83.8% in $Cd(BF_4)_2$ [13].

Table 1
Crystal data and structure refinement.

Compound	$Cd(BF_4)(TaF_6)$	$Cd(BF_4)(BiF_6)$	$Cd(BF_4)(BiF_6)$
Fw ($g\,mol^{-1}$)	494.16	522.19	522.19
T (K)	200	200	293
Space group	P 2_1/c	P 2_1/c	P 2_1/c
a (Å)	8.2700(6)	8.3412(8)	8.3735(10)
b (Å)	9.3691(6)	9.4062(8)	9.4150(11)
c (Å)	8.8896(7)	8.9570(7)	8.9678(11)
β (°)	94.196(3)	93.320(5)	93.604(7)
V (Å^3)	686.94(9)	701.58(11)	705.59(15)
Z	4	4	4
ρ_{calc} ($g\,cm^{-3}$)	4.778	4.944	4.916
λ (Å)	0.71069	0.71069	0.71069
μ (mm^{-1})	19.158	28.223	28.062
GOF indicator	1.237	1.33	1.118
$R1$	0.0539	0.0998	0.0891
$wR2$ ($I > 2.00\sigma(I)$)	0.1103	0.2936	0.2316
Min., max. $\Delta\rho$ (eÅ^{-3})	−2.449, 3.457	−6.333, 5.7	−4.041, 3.525

$R1 = \Sigma||F_o|-|F_c||/\Sigma|F_o|$, $wR2 = [\Sigma(w(F_o^2 - F_c^2)^2/\Sigma(w(F_o^2)^2]^{1/2}$, $GOF = [\Sigma(w(F_o^2 - F_c^2)^2/(N_o - N_p)]^{1/2}$, where N_o = no. of reflections and N_p = no. of refined parameters.

Table 2
Selected interatomic distances (Å) and bond angles (°).

Parameter	$Cd(BF_4)(TaF_6)$[a]	$Cd(BF_4)(BiF_6)$[b]
Cd1–F1^{iii}	2.303(9)	2.28(2)
Cd1–F2^{i}	2.271(8)	2.27(2)
Cd1–F3^{ii}	2.291(10)	2.31(2)
Cd1–F4	2.328(9)	2.30(2)
Cd1–F6^{iii}	2.365(9)	2.39(2)
Cd1–F7^{ii}	2.441(9)	2.36(2)
Cd1–F8^{iv}	2.320(9)	2.33(2)
Cd1–F10	2.273(9)	2.30(2)
B1–F1	1.41(2)	1.46(4)
B1–F2	1.38(2)	1.32(4)
B1–F3	1.39(2)	1.41(4)
B1–F4	1.38(2)	1.42(4)
A1–F5	1.852(9)	1.99(2)
A1–F6	1.933(9)	2.02(2)
A1–F7	1.918(9)	2.03(2)
A1–F8	1.907(9)	1.96(2)
A1–F9	1.849(11)	1.94(2)
A1–F10	1.949(9)	1.99(2)
B1–F1–Cd1^{v}	170.4(10)	164(2)
B1–F2–Cd1^{i}	145.5(9)	154(2)
B1–F3–Cd1^{vi}	147.8(9)	147(2)
B1–F4–Cd1	169.5(10)	179(2)
A1–F6–Cd1^{v}	153.3(5)	142.8(11)
A1–F7–Cd1^{vi}	163.2(5)	162.4(7)
A1–F8–Cd1^{vii}	156.5(5)	153.1(7)
A1–F10–Cd1	149.5(5)	148.2(8)

Symmetry codes used to generate equivalent atoms correspond to those in Fig. 1.
[a] A = Ta; [b] A = Bi.

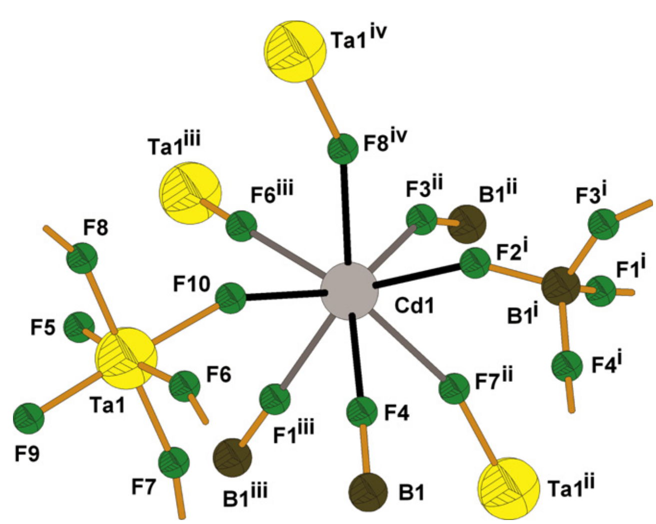

Fig. 1. Coordination surrounding the Cd atom in $Cd(BF_4)(TaF_6)$. Bonds shown in black are in front and bonds shown in gray are behind the plane of the figure. Symmetry codes: (i) $-x+2$, $-y+1$, $-z+1$; (ii) $-x+2$, $y+1/2$, $-z+1/2$; (iii) x, $-y+1/2$, $z-1/2$; (iv) $-x+1$, $y+1/2$, $-z+1/2$; (v) x, $-y+1/2$, $z+1/2$; (vi) $-x+2$, $y-1/2$, $-z+1/2$; (vii) $-x+1$, $y-1/2$, $-z+1/2$.

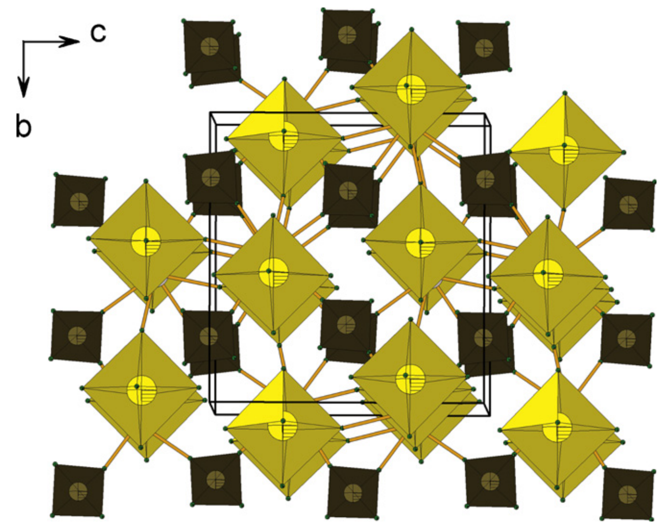

Fig. 2. Packing of $Cd(BF_4)(AF_6)$.

Table 3
Raman frequencies (cm^{-1}), intensities and tentative assignments.

$Cd(BF_4)(TaF_6)$		$Cd(BF_4)(BiF_6)$	
Raman Frequencies	Assignments	Raman Frequencies	Assignments
1054(1)	ν_3 BF_4^-	1010(2)	ν_3 BF_4^-
1016(2)		803(9)	ν_1 BF_4^-
805(19)	ν_1 BF_4^-	620(71)	ν Bi–F
723(100)	ν_1 TaF_6^-	605(82)	
661(11)	ν_2 TaF_6^-	584(100)	
618(3)		544(4)	ν_4 BF_4^-
538(2)	ν_4 BF_4^-	533(2)	
530(3)		498(17)	ν_2 BiF_6^-
514(5)		364(1)	ν_2 BF_4^-
368(1)	ν_2 BF_4^-	356(1)	
357(1)		253(23)	ν_5 BiF_6^-
290(35)	ν_5 TaF_6^-	236(17)	
263(9)		154(1)	lattice mode
158(3)	lattice mode	117(1)	
120(1)			

2.3. Raman spectroscopy

The Raman spectra of both compounds are shown in Fig. 3. Raman frequencies, intensities and tentative assignments are given in Table 3. From the Raman spectra, it is evident that distorted BF_4^- and AF_6^- (A = Ta, Bi) anions are present. The main reason for these distortions originates from the polymeric 3-D structure in which many fluorine atoms are bridged to different Cd cations. In the BF_4^- anion, all four F atoms are bridged to four different Cd cations while in the AF_6^- anion four F atoms are bridged to different Cd cations and two *cis* F atoms are terminal.

The tetrahedral BF_4^- anion (T_d) has four normal modes of vibration, which are all Raman-active whereas only ν_3 and ν_4 are infrared-active. In the solid state, ν_3 and ν_4 may split into two or three bands because of site symmetry lowering. The undistorted AF_6^- anion (O_h) has six normal modes of vibration. Vibrations ν_1, ν_2 and ν_5 are formally Raman-active whereas only ν_3 and ν_4 are infrared-active. The vibration ν_6 is formally inactive [14].

The Raman-active vibrations of the octahedral (O_h) TaF_6^- in the crystalline $CsTaF_6$ are at 692(s), $\nu_1(A_{1g})$; 581(w), $\nu_2(E_g)$; and 272(m) cm^{-1}, $\nu_5(F_{2g})$ [15]. The multiple Raman bands assigned to the TaF_6^- anion in the compound $Cd(BF_4)(TaF_6)$ (see Table 3) could be explained on the basis of lowering the symmetry from O_h to C_{2v} because of the distortion of the octahedral TaF_6^- anion by four fluorine bridges to the different Cd cations.

The Raman-active vibrations of the octahedral (O_h) BiF_6^- are found at 590 cm^{-1}, (ν_1); 547 cm^{-1}, (ν_2); 247, 231 cm^{-1}, (ν_5) in the crystalline $KBiF_6$ [16]. Because of the four fluorine bridges with different Cd cations, the O_h symmetry is lowered to C_{2v} resulting in more Raman bands. The tentative assignments were made by making comparisons with the spectra of the similarly distorted BiF_6^- anion in $KrF^+BiF_6^-$ and $XeF^+BiF_6^-$ salts [17,18]. The splittings around 600 cm^{-1} can only arise from vibrational coupling among the four structural units in the unit cell.

The remaining bands can be attributed to the vibrations of BF_4^- anion. The tetrahedral BF_4^- (T_d) has Raman bands at 1070 (ν_3), 777 (ν_1), 533 (ν_4), and 360 (ν_2) cm^{-1} [19]. Lowering of the anion site symmetry splits these modes. Clearly even greater complexity should be anticipated for the BF_4^- anion Raman spectra of $Cd(BF_4)(AF_6)$: 1054, 1016 (ν_3 related), 805 (ν_1 related), 538, 530, 514 (ν_4 related), 368, 357 (ν_2 related) cm^{-1} for the tantalum

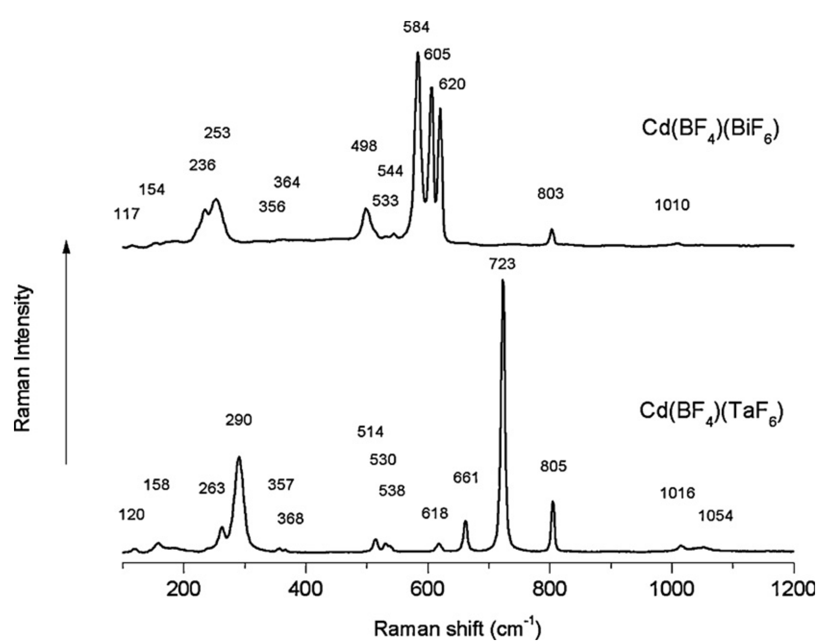

Fig. 3. Raman spectra of $Cd(BF_4)(TaF_6)$ (randomly orientated crystal—with respect to the laser beam) and $Cd(BF_4)(BiF_6)$ (powdered sample).

compound and 1010 (ν_3 related), 803 (ν_1 related), 544, 533 (ν_4 related), 364, 356 (ν_2 related) cm^{-1} for the bismuth compound.

3. Conclusions

The cadmium derivatives are the first examples of only the third structural type for $M^{2+}(BF_4)(AF_6)$ compounds. Two points are particularly noteworthy. Despite the significant difference in ionic radii between calcium and barium, all previously studied $M(BF_4)(AF_6)$ (M = Ca, Sr, Ba, Pb; A = As, Sb, Bi, V, Nb, Ta, Ru) compounds belong to the same structural type and crystallize in an orthorhombic Pnma space group. Both $Ba(BF_4)(PF_6)$ and $Sr(BF_4)(PF_6)$ represent a different structural type—hexagonal, $P\bar{6}2m$ [7,11]. On the other hand, calcium [20], strontium [21] and cadmium [13] tetrafluoroborates are isotypical, but $Ba(BF_4)_2$ [22] is not isostructural with the three previous salts. One may conclude that parent $Ba(BF_4)(AsF_6)$ structural type appears to be extremely energetically favorable and very efficient packing, allowing metal derivatives with such a big difference in ionic radii to exist. Even the calcium atoms in these compounds adopt coordination number 9, higher than that in the structure of calcium fluoride. Replacing the Ca central atom with the Cd atom with a respective decrease in the ionic radius from 1.18 Å to 1.1 Å [23], leads to a decrease in the coordination number from 9 to 8, which is more suitable for the Cd atom [13] and therefore, to the formation of a completely new crystal structure type.

4. Experimental

4.1. General experimental procedure

Volatile materials (aHF, BF_3) were manipulated in a Teflon, FEP (fluorinated ethylene propylene) and nickel vacuum system and line, which were used as described previously [24]. All non-volatile reagents, sensitive to traces of moisture, were stored and handled in an argon atmosphere in a glovebox with maximum water content of less than 1 ppm (M. Braun, Garching, Germany). The reaction vessels were made of 16 mm i.d. (19 mm o.d.) FEP tubing and equipped with Teflon valves and Teflon-coated stirring bars were used for the syntheses. Crystals were grown in a reactor comprised of Teflon valve attached to a T-shaped FEP crystallization vessel which had a larger diameter "reaction" arm constructed from 16 mm i.d. (19 mm o.d.) FEP tube to which a 6 mm i.d. (8 mm o.d.) FEP tube was attached at right angles. The latter was used as a narrower "crystallization" arm. Reaction and crystallization vessels were passivated with F_2 prior to use.

4.2. Reagents

CdF_2 (Alfa Aesar, 99.9%, metals basis), TaF_5 (Alfa Aesar, 99.9%, metals basis), BF_3 (Union carbide, 99.5%) and F_2 (Solvay Fluor, 98–99 vol%) were used as purchased. The solvent anhydrous HF (aHF; Fluka, purum) was treated with K_2NiF_6 (Ozark-Mahoning, 99%) for several days prior to use. BiF_5 was synthesized by high pressure fluorination of BiF_3 (ABCR, 99.5%) at 350 °C in a nickel reaction vessel, similar to the method used for the preparation of AsF_5 [25]. Caution: aHF and BF_3 must be handled in a well-ventilated fume hood, and protective clothing must be worn at all times.

4.3. Synthesis of $Cd(BF_4)_2$

$Cd(BF_4)_2$ was prepared as previously described [13] from CdF_2 (0.630 g, 4.19 mmol) and excess of BF_3 (0.963 g, 14.20 mmol) in aHF (2.4 mL). After 3 days of stirring at room temperature, volatiles were pumped off and the yield of the white product was 1.229 g (4.30 mmol, expected: 1.198 g).

4.4. Synthesis of $Cd(BiF_6)_2$

CdF_2 (9 mg, 0.06 mmol) and BiF_5 (36 mg, 0.12 mmol) in aHF (1.4 mL) were continuously stirred overnight in a FEP reaction vessel. After the removal of the solvent on the vacuum line, the yield of $Cd(BiF_6)_2$ was 46 mg (0.06 mmol, expected: 45 mg).

4.5. Synthesis/crystallization of $Cd(BF_4)(TaF_6)$

$Cd(BF_4)_2$ (30 mg, 0.10 mmol), CdF_2 (16 mg, 0.11 mmol) and TaF_5 (56 mg, 0.20 mmol) were weighed into the large diameter arm of the crystallization vessel inside the glovebox. Solvent aHF (2.0 mL) was added on the vacuum line. After 3 h of stirring at room temperature, a solution with some white precipitate was obtained. Only the precipitate-free, clear, colourless solution was decanted into the narrower arm of the crystallization vessel. The crystals were grown over a period of 4 days by slow evaporation of the solvent from the narrow arm into the wider arm between which a temperature gradient of ca. 16 °C was maintained. Anhydrous HF was pumped off on the vacuum line and the crystals were isolated and immersed in the perfluorinated oil (ABCR, AB102850, Perfluorodecalin, 98%, *cis* and *trans*) inside the glovebox. A colourless single-crystal of 0.08 mm × 0.07 mm × 0.05 mm dimensions was selected under the microscope and quickly transferred into a cold nitrogen stream of the X-ray diffractometer.

4.6. Synthesis/crystallization of $Cd(BF_4)(BiF_6)$

The same procedure as described for the synthesis/crystallization of $Cd(BF_4)(TaF_6)$ was used for $Cd(BiF_6)_2$ (27 mg, 0.04 mmol) and $Cd(BF_4)_2$ (11 mg, 0.04 mmol) in aHF (1.4 mL). A colourless single-crystal of 0.1 mm × 0.08 mm × 0.05 mm dimensions has been used for XRD experiment.

4.7. Raman spectroscopy

The Raman spectra of the single crystals sealed in quartz capillary were obtained on Horiba Jobin-Yvon LabRAM HR spectrometer using the 632.81 nm excitation line of a 25 mW He–Ne laser. The unit cell of the crystal in capillary was checked on the diffractometer to confirm the identity of the compound. Spectra of powdered samples in capillaries were also taken and correspond completely with the ones obtained on the single-crystal. We report the Raman spectrum for $Cd(BF_4)(TaF_6)$ obtained on single-crystal. The choice was made on the basis of lower baseline and more intense signals. In the single-crystal Raman spectrum of $Cd(BF_4)(BiF_6)$, the ratio of intensities for the three most intense peaks (ν Bi–F) varies, therefore we report the spectrum obtained on the powdered sample.

4.8. X-ray structural determination

Data was collected on a Rigaku AFC7 diffractometer equipped with a Mercury CCD area detector using graphite-monochromatized Mo Kα radiation at 200 K. The data were corrected for Lorentz and polarization effects. A multi-scan absorption correction was applied to all data sets. The structures were solved by direct methods using SIR-92 [26] program implemented in the program package TeXsan [27] and refined with SHELX-97 [28] software (program packages TeXsan and WinGX) [29]. The figures were prepared using DIAMOND 3.1 software [30]. Because of the reproducible twinning of $Cd(BF_4)(BiF_6)$ crystals, it was impossible to accurately apply an absorption correction which resulted in rather poor quality of the data. Some details of the data collection, data processing, and refinement are given in Table 1.

Further details of the crystal structure investigation may be obtained from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany, on quoting the deposition numbers CSD-422897 for $Cd(BF_4)(TaF_6)$, CSD-422898 and CSD-422899 for $Cd(BF_4)(BiF_6)$ at 200 K and room temperature respectively.

Acknowledgements

The authors gratefully acknowledge the Slovenian Research Agency (ARRS) for financial support of the present study within the research program: P1-0045 Inorganic Chemistry and Technology.

References

[1] Z. Mazej, H. Borrmann, K. Lutar, B. Žemva, Inorg. Chem. 37 (1998) 5912–5914.
[2] M. Tramšek, B. Žemva, J. Fluorine Chem. 127 (2006) 1275–1284.
[3] T. Bunič, M. Tramšek, E. Goreshnik, B. Žemva, Collect. Czech. Chem. Commun. 73 (2008) 1645–1654.
[4] G. Tavčar, B. Žemva, Angew. Chem. Int. Ed. 48 (2009) 1432–1434.
[5] S.H. Strauss, Chem. Rev. 93 (1993) 927–942.
[6] I. Krossing, I. Raabe, Angew. Chem. Int. Ed. 43 (2004) 2066–2090.
[7] M. Lozinšek, T. Bunič, E. Goreshnik, A. Meden, M. Tramšek, G. Tavčar, B. Žemva, J. Solid State Chem. 182 (2009) 2897–2903.
[8] K.W. Muir, L. Manojlović-Muir, J. Fullard, Acta Crystallogr. C52 (1996) 295–297.
[9] D.V. Peryshkov, E. Goreshnik, Z. Mazej, S.H. Strauss, J. Fluorine Chem. 131 (2010) 1225–1228.
[10] A. Meden, Acta Chim. Slov. 53 (2006) 148–152.
[11] In preparation.
[12] N.E. Brese, M. O'Keeffe, Acta Crystallogr. B47 (1991) 192–197.
[13] G. Tavčar, B. Žemva, Inorg. Chem. 44 (2005) 1525–1529.
[14] K. Nakamoto, Infrared and Raman Spectra of Inorganic and Coordination Compounds, Part A, 6th ed., John Wiley & Sons, Hoboken, New Jersey, 2009.
[15] O. Lewin Keller, A. Chetham-Strode, Inorg. Chem. 5 (1966) 367–372.
[16] A.I. Popov, A.V. Scharabarin, V.F. Sukhoverkhov, N.A. Tchumaevsky, Z. Anorg, Allg. Chem. 576 (1989) 242–254.
[17] R.J. Gillespie, D. Martin, G.J. Schrobilgen, J. Chem. Soc. Dalton Trans. (1980) 1898–1903.
[18] H.St.A. Elliott, J.F. Lehmann, H.P.A. Mercier, H.D.B. Jenkins, G.J. Schrobilgen, Inorg. Chem. 49 (2010) 8504–8523.
[19] A.S. Quist, J.B. Bates, G.E. Boyd, J. Chem. Phys. 54 (1971) 4896–4901.
[20] T.H. Jordan, B. Dickens, L.W. Schroeder, W.E. Brown, Acta Crystallogr. B31 (1975) 669–672.
[21] E. Goreshnik, A. Vakulka, B. Žemva, Acta Crystallogr. C66 (2010) e9.
[22] T. Bunič, G. Tavčar, E. Goreshnik, B. Žemva, Acta Crystallogr. C63 (2007) i75–i76.
[23] R.D. Shannon, Acta Crystallogr. A32 (1976) 751–767.
[24] H. Borrmann, K. Lutar, B. Žemva, Inorg. Chem. 36 (1997) 880–882.
[25] Z. Mazej, B. Žemva, J. Fluorine Chem. 126 (2005) 1432–1434.
[26] A. Altomare, M. Cascarano, C. Giacovazzo, A. Guagliardi, J. Appl. Crystallogr. 26 (1993) 343–350.
[27] TeXsan for Windows-Single Crystal Structure Analysis Software, v. 1. 06, Molecular Structure Corp, Woodlands, TX, USA, 1997–1999.
[28] G.M. Sheldrick, Acta Crystallogr. A64 (2008) 112–122.
[29] L.J. Farrugia, J. Appl. Crystallogr. 32 (1999) 837–838.
[30] DIAMOND, v. 3. 1., Crystal Impact GbR, Bonn, Germany, 2004–2005.