Co-crystallization of octahedral and icosahedral fluoroanions in $K_3(AsF_6)(B_{12}F_{12})$ and $Cs_3(AsF_6)(B_{12}F_{12})$. Rare examples of salts containing fluoroanions with different shapes and charges[☆]

Dmitry V. Peryshkov [a], Evgeny Goreshnik [b], Zoran Mazej [b,**], Steven H. Strauss [a,*]

[a] Department of Chemistry, Colorado State University, Fort Collins, CO 80523, USA
[b] Jožef Stefan Institute, 1000 Ljubljana, Slovenia

ABSTRACT

Crystals grown from anhydrous HF solutions of $CsAsF_6$ and $Cs_2B_{12}F_{12}$ or $KAsF_6$ and $Cs_2B_{12}F_{12}$ are shown by Raman spectroscopy and single-crystal X-ray diffraction to be the ternary salts $K_3(AsF_6)(B_{12}F_{12})$ or $Cs_3(AsF_6)(B_{12}F_{12})$. Both compounds exhibit a modified version of the anti-perovskite structure. They are rare examples of crystals that simultaneously contain octahedral and icosahedral molecular species and are also rare examples of salts containing fluoroanions with different shapes and charges. The crystallographic results show that both compounds are densely packed.

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1. Introduction

The packing of different shapes in three-dimensional space is a longstanding problem in mathematics, physics, and chemistry. Our dual interests in highly fluorinated anions [1–4] and crystal engineering [5] has resulted in the preparation of alkali metal salts with more than one complex fluoroanion, in part to examine the packing efficiencies of hard ions as a function of ion size, shape, and charge. Two such compounds, $K_3(AsF_6)(B_{12}F_{12})$ and $Cs_3(AsF_6)(B_{12}F_{12})$, are described in this report. These are rare examples of salts containing fluoroanions of different shape and charge. To our knowledge, the only other examples are $[Te(C_6H_5)_3]_3F$-$(BF_4)(SiF_6)_{0.5} \cdot 3CHCl_3$ [6], $[Co(en)_3]_2(SiF_6)(Zr_2F_{12}) \cdot 4H_2O$ [7], and $K_3Na_4(BF_4)(SiF_6)_3$ [8] (examples of salts with fluoroanions of different shape but the same charge are $Ba(BF_4)(PF_6)$, $Ba(BF_4)(AsF_6)$, and $Ba_2(BF_4)_2(AsF_6)(H_3F_4)$ [9], $[Pt_3(\mu_3\text{-}AuPPh_3)(\mu\text{-}dppm)_3](BF_4)_{0.25}$ $(PF_6)_{0.75}$ [10], $[Mo(CO)_4]_2(SbF_6)_3(Sb_2F_{11})$ [11], $Pd(L)(L')(BF_4)(PF_6)$ $(L,L' = o\text{-aminophenolates})$ [12], and $[Pd(\eta^3\text{-allyl})L]_2(BF_4)(PF_6)$

(L = a diphosphitocalix[4]arene [13]). They are also rare examples of mixed anion salts containing an icosahedral anion (other examples are $M_3I(B_{12}H_{12})$ ($M^+ = K^+$, Rb^+, Cs^+, NH_4^+) [14] and $[La(H_2O)_9](-H_3O)Cl_2[B_{12}H_{12}] \cdot H_2O$ [15]).

2. Results and discussion

2.1. Synthesis

Multigram-scale syntheses of $K_2B_{12}F_{12}$ and $Cs_2B_{12}F_{12}$ were recently reported [1]. The combination of stoichiometric amounts of $Cs_2B_{12}F_{12}$ and $CsAsF_6$ in HF resulted in the formation of crystals of the double salt $Cs_3(AsF_6)(B_{12}F_{12})$. However, the combination of stoichiometric amounts of $Cs_2B_{12}F_{12}$ and $KAsF_6$ resulted only in the formation of crystals of the two double salts $K_3(AsF_6)(B_{12}F_{12})$ and $Cs_3(AsF_6)(B_{12}F_{12})$. Crystals of $K_xCs_{3-x}(AsF_6)(B_{12}F_{12})$, $K_xCs_{2-x}B_{12}F_{12}$, or $K_xCs_{1-x}AsF_6$ were not observed in spite of the co-existence of K^+ and Cs^+ cations in numerous crystalline compounds including $Cs_2K(Bi(SCN)_xI_{6-x}$ [16], $Cs_{0.6}K_{0.4}TiOAsO_4$ [17], $KCs_3Ni_4(Fe(CN)_6)_3$ [18], and $Cs_3K_2Na_4[Cs_2K(H_2O)_7Pd_2WO(H_2O)(A\text{-}\alpha\text{-}SiW_9O_{34})_2] \cdot 5H_2O$ [19]. There is a practical application for the co-existence of K^+ and Cs^+ cations in crystalline compounds: the extraction of Cs^+ into ionic compounds that are partially depleted in K^+ has been studied for the purpose of nuclear waste remediation [18,20–22].
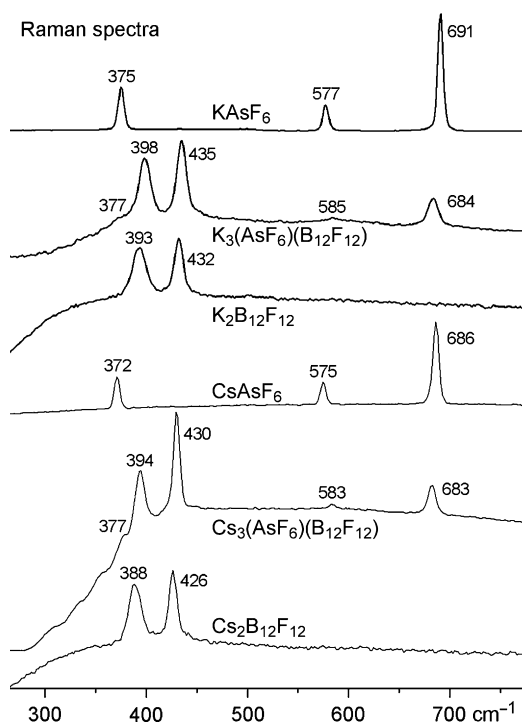
Fig. 1. Solid-state Raman spectra (632.8 nm excitation).

Table 1
Crystallographic and data-collection parameters.

Compound	$K_3(AsF_6)(B_{12}F_{12})$	$Cs_3(AsF_6)(B_{12}F_{12})$
Empirical formula	$AsB_{12}F_{18}K_3$	$AsB_{12}Cs_3F_{18}$
Formula weight ($g\,mol^{-1}$)	663.9	945.3
Habit, color	Prism, colorless	Cube, colorless
Crystal system	cubic	cubic
Space group	$Fm\bar{3}c$	$Ia\bar{3}$
a (Å)	15.5340(3)	16.1206(7)
V (Å^3)	3748.4(1)^a	4189.3(3)
Z	8	8
T (K)	120(2)	200(2)
ρ_{calc} ($g\,cm^{-3}$)	2.3530(1)^a	2.9978(2)
$R(F)$ ($I > 2\sigma(I)$)	0.0186	0.0655
$wR(F^2)$ (all data)	0.0505	0.1794
Minimum, maximum $\Delta\rho$ (eÅ^{-3})	−0.17, 0.49	−2.62, 4.61

^{a} At 200 K, the unit cell volume and calculated density of $K_3(AsF_6)(B_{12}F_{12})$ were 3774.3(6)Å^3 and 2.3369(4)$g\,cm^{-3}$, respectively.

2.2. Raman spectroscopy

Raman spectra of $K_3(AsF_6)(B_{12}F_{12})$, $Cs_3(AsF_6)(B_{12}F_{12})$, $K_2B_{12}F_{12}$, $Cs_2B_{12}F_{12}$, $KAsF_6$, and $CsAsF_6$ are shown in Fig. 1. These are the first Raman spectra reported for any salt of the $B_{12}F_{12}^{2-}$ anion. The bands at 687 ± 4, 580 ± 4, and 375 ± 3 cm^{-1} in the compounds containing the AsF_6^- anion are assigned to the ν_1 (A_{1g} symmetry) and ν_2 (E_g symmetry) As–F stretching normal modes and the ν_5 (T_{2g} symmetry) bending normal modes, respectively [23]. The bands at 431 ± 5, and 393 ± 5 cm^{-1} in the compounds containing the $B_{12}F_{12}^{2-}$ anion are assigned to the ν_2 (A_{1g} symmetry) and ν_5 (H_g symmetry) cage normal modes [24–27] (the former was predicted to be at 442 [25] or at 425 cm^{-1} [26] and the latter was predicted to be at 386 cm^{-1} [25] for the "free" $B_{12}F_{12}^{2-}$ anion).

The significance of the Raman spectra is that they demonstrate that the samples of $K_3(AsF_6)(B_{12}F_{12})$ and $Cs_3(AsF_6)(B_{12}F_{12})$ used to record the Raman spectra were in fact the ternary compounds as formulated. These samples did not contain an observable amount of the binary salt precursors. Interestingly, both the AsF_6^- and $B_{12}F_{12}^{2-}$ bands soften on going from the potassium salt to the cesium salt, at least in the 300–700 cm^{-1} region of the spectra.

2.3. Crystal structures

Crystallographic results and structure refinement parameters are listed in Table 1. The structures of $K_3(AsF_6)(B_{12}F_{12})$ and $Cs_3(AsF_6)(B_{12}F_{12})$, like those of $K_3I(B_{12}H_{12})$ and $Cs_3I(B_{12}H_{12})$ [14], can be described as variants of the Pm3m K_3OCl anti-perovskite structure [28], with the As atom of AsF_6^- replacing the Cl$^-$ ions and the B_{12} centroids replacing the O^{2-} ions. Selected distances and angles are listed in Table 2. The packing of the ions in $K_3(AsF_6)(B_{12}F_{12})$ is shown in Figs. 2 and 3. The F atoms of the AsF_6^- anions are badly disordered and must be modeled by at least eight partial occupancies with unrealistic thermal parameters and an abnormally short As–F2 distance of 1.671(5) Å. Therefore, the As–F2 and K···F2 distances are not listed in Table 2 and the F2 atoms have been omitted from Figs. 2 and 3.

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

| Parameter	$K_3(AsF_6)(B_{12}F_{12})^a$	$Cs_3(AsF_6)(B_{12}F_{12})^b$
As–F	–	$1.702(8) \times 6$ (F = F3)
B–F	$1.381(2) \times 12$	$1.39(1) \times 6$, $1.40(1) \times 6$
B–B range	1.788(2)–1.790(2)	1.779(16)–1.798(13)
B–B ave.	1.790	1.789
B···(B$_{12}$ centroid)	1.702	
M···F	$2.589(1) \times 4$	$3.001(6) \times 2$ (F = F2)
		$3.153(5) \times 2$ (F = F1)
		$3.178(5) \times 2$ (F = F1)
		$3.246(8) \times 2$ (F = F3)
		$3.269(8) \times 2$ (F = F3)
M···As	$3.884(1) \times 2$	$4.064(1) \times 2$
M···(B$_{12}$ centroid)	5.492×4	5.343×2
		6.080×2
As···As	$7.767(1) \times 6$	$8.060(1) \times 6$
(B$_{12}$ centroid)···(B$_{12}$ centroid)	7.767×6	8.060×6
As···(B$_{12}$ centroid)	6.726×8	
M···F–B	$151.0(1) \times 4$ (F = F1)	$117.1(5) \times 2$ (F = F1)
		$117.9(5) \times 2$ (F = F1)
		$174.3(5) \times 2$ (F = F2)
M···F–As	–	$105.3(3) \times 2$ (F = F3)
		$106.2(3) \times 2$ (F = F3)

^{a} M = K.
^{b} M = Cs.
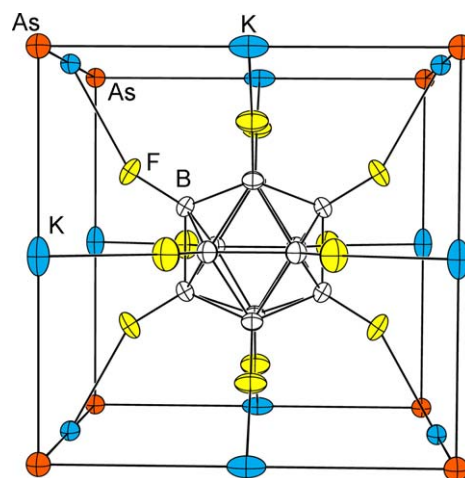
Fig. 2. Crystal packing in $K_3(AsF_6)(B_{12}F_{12})$ (50% probability ellipsoids). The disordered F atoms attached to the AsF_6^- anions have been omitted for clarity.
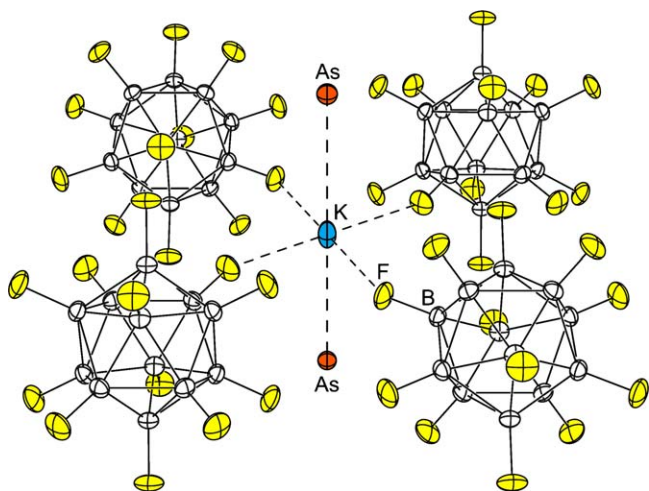
Fig. 3. The environment around the K^+ ion in $K_3(AsF_6)(B_{12}F_{12})$ (50% probability ellipsoids). The disordered F atoms attached to the AsF_6^- anions have been omitted for clarity. The four $K\cdots F(B)$ distances are 2.589(1) Å.

The $B_{12}F_{12}{}^{2-}$ anion in $K_3(AsF_6)(B_{12}F_{12})$ has crystallographic D_{2h} site symmetry, and the drawing in Fig. 3 has been oriented so that one of the C_2 axes is nearly perpendicular to the plane of the page. The K^+ cation and the four F atoms from four $B_{12}F_{12}{}^{2-}$ anions are co-planar; they form a square with the K^+ cation in the middle. The four $K\cdots F1$ distances of 2.589(1) Å produce a bond-valence [29] sum of 0.797. Therefore, assuming a total bond-valence of 1.000 for K^+, the unknown $K\cdots F2$ distances can only contribute a bond-valence sum of 0.203, which would imply two $K\cdots F2$ distances of ca. 2.84 Å or four $K\cdots F2$ distances of ca. 3.10 Å.

There is no disorder in the structure of $Cs_3(AsF_6)(B_{12}F_{12})$. The packing of the ions is shown in Fig. 4. Unlike the K^+ ions in $K_3(AsF_6)(B_{12}F_{12})$, the Cs^+ ions do not lie directly on the $As\cdots As$ vectors. The CsF_{10} coordination sphere, shown in Fig. 5, can be described as a distorted bicapped square antiprism. The four $Cs\cdots F3$ distances to the two closest AsF_6^- anions, 3.246(8) ($\times 2$) and 3.269(8) Å ($\times 2$), are longer than the six $Cs\cdots F1/F2$ distances to the two closest $B_{12}F_{12}{}^{2-}$ anions, 3.001(6) ($\times 2$), 3.153(5) ($\times 2$), and 3.178(5) Å ($\times 2$), and the sum of all ten $Cs\cdots F$ bond valences [30] is 1.071, with 0.745 contributed by four F1 and two F2 atoms and 0.326 contributed by four F3 atoms.
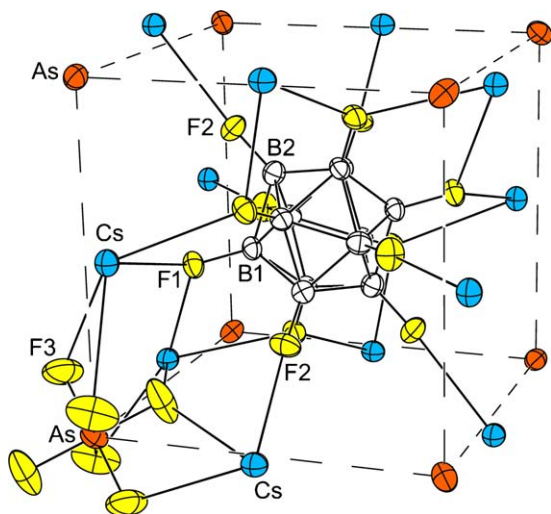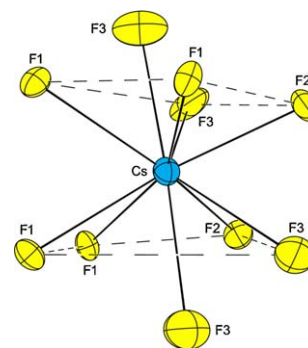

Fig. 5. The CsF_{10} coordination sphere of the Cs^+ cations in $Cs_3(AsF_6)(B_{12}F_{12})$ (50% probability ellipsoids; F1 and F2 atoms are bonded to B1 and B2, and F3 atoms are bonded to As). The coordination geometry is a distorted bicapped square antiprism.

The formula unit volumes of $K_3(AsF_6)(B_{12}F_{12})$ and $Cs_3(AsF_6)(B_{12}F_{12})$ at 200 K differ by 415.0 Å^3, or 17.3 Å^3 per alkali metal cation. Interestingly, this is 6% smaller than the difference in volumes of cubes with edge lengths equal to twice the ionic radii of K^+ (i.e., 1.33 Å $\times$ 2) and Cs^+ (1.67 Å $\times$ 2), which is 18.4 Å^3. This implies that the packing efficiency of the cesium salt may be slightly more efficient than the packing efficiency of the potassium salt, and this may be due to the significant AsF_6^- disorder in the potassium salt. Another way to assess the packing efficiency of the cesium salt is to add the molar volumes of $CsAsF_6$, 83 cm^3 mol^{-1} [31] and $Cs_2(H_2O)B_{12}F_{12}$, 227 cm^3 mol^{-1} [32], and to compare that sum, 310 cm^3 mol^{-1}, with the molar volume of $Cs_3(AsF_6)(B_{12}F_{12})$, which, at 316 cm^3 mol^{-1}, is nearly identical.

The co-existence of real or idealized icosahedral and octahedral/cubic moieties in the same crystal, and even within the same molecule, has recently been reviewed [33]. The $B_{12}F_{12}{}^{2-}$ anion in $K_3(AsF_6)(B_{12}F_{12})$ is an example of this phenomenon. The three C_2 axes of the icosahedral anion form the axes of an octahedron inscribed in the B_{12} cluster. A related example is the compound $C_{60}(C(COOEt)_2)_6$, a hexakis cycloadduct of I_h-C_{60} in which the six quaternary C atoms attached to the parent icosahedral fullerene also define three idealized C_2 axes and form an idealized octahedron [34].

3. Experimental

3.1. Materials

The salts $K_2B_{12}F_{12}$ and $Cs_2B_{12}F_{12}$ were prepared as previously described [1]. Liquid anhydrous HF (aHF; Fluka, Purum) was treated with K_2NiF_6 (Ozark Mahoning) for several hours prior to use. Potassium fluoride (Ventron, 99.9%) was dried under vacuum at 200 °C to remove possible traces of moisture. Cesium fluoride (Aldrich, 99.9%) and F_2 (Solvay Fluor, 98–99% by volume) were used as received. Arsenic pentafluoride was prepared from As_2O_3 (Fluka, 99.5%) and F_2 as previously described [35]. The salts $KAsF_6$ and $CsAsF_6$ were prepared from KF or CsF and AsF_5 in aHF in a similar manner to the synthesis of $Pd(AsF_6)_2$ from PdF_2 and AsF_5 in aHF [36]. The volatile materials (AsF_5 and aHF) were handled in an all-Teflon vacuum line equipped with Teflon valves. All other materials were handled in an argon-filled glovebox (≤ 2 ppm H_2O vapor).

3.2. Synthesis of $M_3(AsF_6)(B_{12}F_{12})$

The reactions were carried out in T-shaped FEP tubes equipped with Teflon valves and were passivated with F_2 prior to use. The "upright" of the T was a 6 mm o.d. tube and the "crossbar" was a 19 mm o.d. tube (ca. 250–300 mm long) to which the valve was attached. Reaction mixtures were stirred with Teflon-coated stir bars.

Fig. 4. The crystal packing of $Cs_3(AsF_6)(B_{12}F_{12})$ (50% probability ellipsoids). The F3 atoms have been omitted for clarity except for one AsF_6^- anion.

Single crystals of $Cs_3(AsF_6)(B_{12}F_{12})$ were prepared by stirring $CsAsF_6$ (0.13 mmol) and $Cs_2B_{12}F_{12}$ (0.13 mol) in aHF (8 mL) in the 19 mm o.d. part of the FEP apparatus at 20–25 °C. The reaction mixture contained some undissolved solids. The clear, colorless supernatant was decanted into the 6 mm o.d. arm of the apparatus. Evaporation of aHF from this solution was carried out by maintaining a temperature gradient of ca. 10 °C between the 6 mm o.d. and the 19 mm o.d. tubes for 3 weeks. When all of the aHF had condensed in the 19 mm o.d. tube, colorless crystals of $Cs_3(AsF_6)(B_{12}F_{12})$ remained in the 6 mm o.d. tube, which were ultimately collected in the glovebox.

The same procedure was carried out with a mixture of $KAsF_6$ (0.14 mmol) and $Cs_2B_{12}F_{12}$ (0.14 mmol) in an attempt to prepare single crystals of $KCs_2(AsF_6)(B_{12}F_{12})$. Instead of the desired product, a mixture of crystals of the single-cation salts $K_3(AsF_6)(B_{12}F_{12})$ and $Cs_3(AsF_6)(B_{12}F_{12})$ was obtained.

3.3. Raman spectroscopy

Single crystals of $K_3(AsF_6)(B_{12}F_{12})$ and $Cs_3(AsF_6)(B_{12}F_{12})$ and microcrystalline samples of $KAsF_6$, $CsAsF_6$, $K_2B_{12}F_{12}$, and $Cs_2B_{12}F_{12}$ were sealed inside 0.3 mm quartz capillaries in the glovebox and their $1\ cm^{-1}$ resolution Raman spectra were recorded using a Renishaw Raman Imaging Microscope System 1000 (632.8 nm excitation; 50 mW laser power).

3.4. X-ray crystallography

X-ray diffraction data from a crystal of $K_3(AsF_6)(B_{12}F_{12})$ was collected on a Bruker Kappa APEX II CCD diffractometer at 120 K employing Mo Kα radiation (graphite monochromator). Selected details of the crystallographic experiment are listed in Table 2. Unit cell parameters were obtained from least-squares fits to the angular coordinates of all reflections, and intensities were integrated from a series of frames (ω and φ rotation) covering more than a hemisphere of reciprocal space. Absorption and other corrections were applied using SADABS [37]. The structure was solved using direct methods and refined (on F^2, using all data) by a full-matrix, weighted least-squares process. Standard Bruker control and integration software (APEX II) was employed [38], and Bruker SHELXTL software was used for structure solution, refinement, and molecular graphics [39]. All atoms were refined using anisotropic atomic displacement parameters. The F atoms of the AsF_6^- anions were badly disordered and appeared to be located at eight partial occupancies at the corners of a cube around the As atom, with unrealistic thermal parameters and an abnormally short As–F2 distance of 1.671(5) Å. Attempts to model the disorder with multiple octahedral AsF_6^- anions were unsuccessful.

X-ray diffraction data from a crystal of $Cs_3(AsF_6)(B_{12}F_{12})$ covered with ABCR FO5960 fluorinated oil was collected on a Rigaku AFC7 diffractometer equipped with a Mercury CCD area detector at 200 K employing Mo Kα radiation (graphite monochromator). The data were corrected for Lorentz and polarization effects. Selected details of the crystallographic experiment are listed in Table 2. A multi-scan absorption correction was applied to the data. The structure was solved by direct methods using SIR-92 [40] (TeXsan program package [41]) and refined (on F^2, using all data) by a full-matrix, weighted least-squares process using SHELXL-97 [42]. Molecular graphics were created using Bruker SHELXTL software [39].

4. Supplementary data

Crystallographic data for $K_3(AsF_6)B_{12}F_{12}$ and $Cs_3(AsF_6)B_{12}F_{12}$ (excluding structure factors) have been deposited with the Inorganic Crystal Structure Database (deposition numbers CSD-421902 ($K_3(AsF_6)B_{12}F_{12}$) and CSD-421903 ($Cs_3(AsF_6)B_{12}F_{12}$)).

Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, fax: +44 1223 336033, e-mail: deposit@ccdc.cam.ac.uk.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jfluchem.2010.05.009.

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