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New class of coordination compounds with noble gas fluorides as ligands to metal ions

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Abstract

A new class of coordination compounds of the type $[M^{n+}(L)_p](AF_6^-)_n$ and $[M^{n+}(L)_r](BF_4^-)_n$, where M is Mg, Ca, Sr, Ba, Cd, Pb, lanthanides, A is P, As, Sb, Bi and L is XeF_2, XeF_4, XeF_6, KrF_2, was studied. A review of all known coordination compounds with L is XeF_2 is given: (a) synthetic routes for the preparation of these compounds; (b) analysis of their crystal structures (molecular, dimer, chain, double chain, layer, strongly interconnected double layers and three-dimensional network); (c) the influence of the ligand XeF_2 (small formula volume, linear, semi-ionic, charge of $-0.5e$ on each F ligand); (d) the influence of the central metal ion; (e) the influence of the anions: AF_6^- and BF_4^- (the formula volume, Lewis basicity). On the basis of all properties of the metal ions, ligand and anions the obtained variety of the structures is analyzed.

Keywords: Xenon fluorides; Ligands to metal ion; Arsenates(V); Antimonates(V); Phosphates(V)

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1. Introduction

Metal(II) and many metal(III) fluorides react with Lewis acid AF_5 (A = P, As, Sb, Bi) in anhydrous HF (aHF), as a solvent, yielding compounds of the type $M^{n+}(AF_6^-)_n$, where n is the oxidation state of the metal. These salts have rather low lattice energy as a consequence of the anion volume (>100 Å^3) [1]. Thus almost "naked" metal centers surrounded with weakly interacting AF_6^- anions can coordinate not only strong ligands, but also weak ligands of the solvent. Therefore the compounds $M^{n+}(AF_6^-)_n$ represent excellent starting materials for the preparation of a new class of the coordination compounds of the type $[M^{n+}(L)_p](AF_6)_n$ with noble gas fluorides, such as XeF_2, XeF_4, XeF_6 and KrF_2 as possible ligands (L). The first compound in which noble gas fluoride, XeF_2, was observed to be bound directly to the metal center was $[Ag(XeF_2)_2](AsF_6)$ [2]. In the last years, a whole series of the coordination compounds with XeF_2 as a ligand to the metal ion was isolated and many crystal structures of these compounds were determined [3–10].

In this paper, a review of all known coordination compounds with XeF_2 as a ligand is given together with the reaction systematics, Raman spectra, available structures, metal-fluoro-ligand polyhedra and the influence of the cation and the anion on the type of the structure. Further, the problems connected with XeF_4, XeF_6 and KrF_2 as possible ligands to metal ions are elucidated. Finally the first coordination compound of XeF_2 with $M(BF_4)_2$ is mentioned.

2. Results and discussion

2.1. Syntheses

In the salts $M^{n+}(AF_6^-)_n$, AF_6^- anions are weakly coordinated to metal centers which are therefore practically "naked". AF_6^- are also poor Lewis bases, so that even poor Lewis base solvent, such as aHF, can dissolve compounds $M^{n+}(AF_6^-)_n$, yielding at least solvated cations $[M^{n+}(HF)_m]$ and AF_6^- anions (Eq. (1)). Addition of a relatively weak Lewis base, XeF_2, to this solution, however, introduces a stronger Lewis base than aHF. XeF_2, by virtue of its semi-ionic character and its relatively small formula volume

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(65 Å^3), competes effectively with AF_6^- in providing Coulomb energy (Eq. (2)). The charge on F ligands in XeF_2 is nearly $-0.5e$:

$$M^{n+}(AF_6^-)_n + mHF \xrightarrow[22\,^\circ C]{aHF} [M(HF)_m]^{n+} + nAF_6^- \quad (1)$$

$$[M(HF)_m]^{n+} + nAF_6^- + zXeF_2 \xrightarrow[22\,^\circ C]{aHF} [M(XeF_2)_p]^{n+} + nAF_6^- + (z-p)XeF_2 \quad (2)$$

However, there are two requirements which have to be fulfilled in order that the reaction (2) would proceed: (a) metal cations should not be sufficiently strong Lewis acids to withdraw F^- from XeF_2 and to generate $Xe_2F_3^+AF_6^-$ or $XeF^+AF_6^-$ salts. (b) XeF_2 dissolved in aHF is relatively strong oxidizing agent; therefore it is essential that metal cations are resistant towards further oxidation. If metal cations are oxidized they are converted to even stronger Lewis acids and the possibility that they will withdraw F^- from XeF_2 molecule is even greater.

There are a number of different synthetic routes for the preparation of these coordination compounds. The number of XeF_2 molecules per metal cation (p) is the highest when the solvent aHF is pumped away after the reaction is completed, but the coordination compound is still mixed with an excess of XeF_2. In this case it was proven by Raman spectroscopy that in many cases at $p = 6$ there was no more free XeF_2 present (the stretching band ν_1 of XeF_2 at 496 cm^{-1} was absent). The compounds with high "p" value are usually not stable in a dynamic vacuum, slowly losing XeF_2 and yielding white solids with $5 \geq p \geq 2$. In the case of $[Ca(XeF_2)_4](AsF_6)_2$ [7], which is stable at room temperature, the compound $[Ca(XeF_2)_{2.5}](AsF_6)_2$ could be obtained by pumping the 1:4 compound in a dynamic vacuum at 60 °C to the constant weight [7].

Another synthetic approach, for the preparation of pure compounds, is the synthesis where the use of the exact stoichiometric amounts of $M^{n+}(AF_6^-)_n$ and XeF_2 was employed. XeF_2 could be synthesized also during the reaction in situ, as was done in the case of the synthesis of $[Ag(XeF_2)_2](AsF_6)$ [2].

In the case of Lewis acid PF_5, which does not form $XeF^+PF_6^-$ or stable $XeF_2{\cdot}PF_5$ adduct, the reaction could be performed by direct reaction between binary fluoride ($M^{n+}F_n$), XeF_2 and gaseous PF_5 in aHF. Because solubility of PF_5 in aHF is poor, a high pressure of PF_5 should be employed. Corresponding $M(PF_6)_2$ is formed in situ and it reacts further with the present XeF_2 yielding $[M(XeF_2)_p](PF_6)_2$ [11].

Recently also the first coordination compound with BF_4^- anion $[Cd(XeF_2)](BF_4)_2$ was isolated and its structure was determined [12]. The compound could be synthesized by using the same synthetic approach as in the case of PF_5 or by the reaction between $Cd(BF_4)_2$ and XeF_2 in aHF.

Another example of special synthetic approach is the procedure in the case of the lanthanides. When $Ln(AsF_6)_3$ are prepared in dry form, they are losing AsF_5 yielding compounds of the type $LnF_x(AsF_6)_{3-x}$, where $x = 1, 1.5$. In order to have $Ln(AsF_6)_3$ in the solution, the exact amount of AsF_5 should be added to LnF_3. An excess of AsF_5 would form $Xe_2F_3^+AsF_6^-$ salt after addition of XeF_2 to the solution.

What about the other noble gas fluorides as ligands to the metal ion? XeF_4 is a weaker Lewis base as XeF_2. It is a planar molecule and its interaction with metal center in $M^{n+}(AF_6^-)_n$ is possible. This was proven by the reaction between $Mg(AsF_6)_2$ and XeF_4 in aHF at room temperature. Now the only evidence for the existence of the compound $[Mg(XeF_4)](AsF_6)_2$ [13] is its Raman spectrum showing bands which by the comparison with the spectrum of XeF_3^+ [14] could be attributed to bands of the coordinated XeF_4: $\nu(Xe\text{–}F_{2(ax)})$ is 550(10) cm^{-1} and $\nu(Xe\text{–}F_{eq})$ is 622(5.8) cm^{-1}.

XeF_6 is the strongest oxidizer and the strongest base among binary fluorides of xenon. Because XeF_6 is so much better Lewis base as XeF_2, the metal cations, which are able to coordinate XeF_2, withdraw F^- from XeF_6 yielding $Xe_2F_{11}^+AF_6^-$. This was exemplified by the reactions between $M(AsF_6)_2$, M = Mg, Ca, Sr, and XeF_6 in aHF:

$$M(AsF_6)_2 + nXeF_6 \xrightarrow[22\,^\circ C]{aHF} MF_2 + 2Xe_2F_{11}AsF_6 + (n-4)XeF_6, \quad n > 22 \quad (3)$$

KrF_2 is also a linear molecule as XeF_2. It is a very strong oxidizing agent, but it is a relatively weak Lewis base. The coordination compounds of the type $[M(KrF_2)_n](AF_6)_2$ should be possible.

2.2. Review of the structures $[M^{n+}(XeF_2)_p](AF_6^-)_n$ and $[M(XeF_2)_r](BF_4)_2$

XeF_2 ligand in the coordination compounds of the type $[M^{n+}(XeF_2)_p](AF_6^-)_n$ is either a nonbridging ligand interacting only with one metal center, or a bridging ligand, connecting two metal centers. The number of XeF_2 molecules (bridging and nonbridging) around one metal center could range from one nonbridging XeF_2 molecule in $[Cd(XeF_2)](BF_4)_2$ [12] to up to nine XeF_2 molecules (five nonbridging and four bridging) around one Ca atom in $[Ca_2(XeF_2)_9](AsF_6)_4$ while the other Ca atom in this compound is coordinated by four bridging XeF_2 molecules and four AsF_6 units [15]. The coordination sphere of metal centers could be composed only of XeF_2 molecules (one homoleptic Ca atom in $[Ca_2(XeF_2)_9](AsF_6)_4$ or XeF_2 molecules and AF_6^- anions). The metal centers could be connected: (a) only by XeF_2 molecules, (b) by XeF_2 and AF_6^- anions, (c) only by AF_6^- anions. In the case of BF_4^- anion in the compound $[Cd(XeF_2)](BF_4)_2$ the metal centers are connected only by BF_4^- anions in the double-layer structure (Table 1).

Table 1
Structural diversity in $[M^{n+}(XeF_2)_p](AF_6^-)_n$ and $[M(XeF_2)_r](BF_4)_2$

Connections between metal ions	Compounds
XeF_2 molecules	$[Ca_2(XeF_2)_9](AsF_6)_4$ [15], $[Cd_2(XeF_2)_{10}](SbF_6)_4$ [16], $[Ca(XeF_2)_4](AsF_6)_2$ [7], $[Cd(XeF_2)_4](AsF_6)_2$ [10]
XeF_2 molecules and AF_6^- units	$[Ca(XeF_2)_{2.5}](AsF_6)_2$ [7], $[Pb(XeF_2)_3](AsF_6)_2$ [5], $[Sr(XeF_2)_3](AsF_6)_2$ [5], $[Nd(XeF_2)_{2.5}](AsF_6)_3$ [4], $[Ba(XeF_2)_5](SbF_6)_2$ [6], $[Ag(XeF_2)_2]AsF_6$ [2], $[Ag(XeF_2)_2]PF_6$ [8]
AF_6^- units	$[Mg(XeF_2)_2](AsF_6)_2$ [9], $[Mg(XeF_2)_2](SbF_6)_2$ [18], $[Nd(XeF_2)_3](SbF_6)_3$ [18]
BF_4^- units	$[Cd(XeF_2)](BF_4)_2$ [12]
Molecular structure	$[Mg(XeF_2)_4](AsF_6)_2$ [9]

The structures of these compounds depend upon effective nuclear charge on the metal center (Lewis acidity of the metal center), character of the M–F bonds (ionic, covalent), and Lewis basicity of the AF_6^- anions. A variety of the structures was found in this type of compounds: molecular structure (e.g. $[Mg(XeF_2)_4](AsF_6)_2$ [9]), dimer structure (e.g. $[Cd_2(XeF_2)_{10}](SbF_6)_4$ [16], Fig. 1) chain structure (e.g. $[Ca(XeF_2)_5](PF_6)_2$ [11], Fig. 2), double-chain (e.g. $[Nd(XeF_2)_{2.5}](AsF_6)_3$ [4]), layer structure (e.g. $[Ca(XeF_2)_4](AsF_6)_2$ [7]), strongly interconnected double layers (e.g. $[Cd(XeF_2)](BF_4)_2$, Fig. 3; $[M(XeF_2)_3](AsF_6)_2$, M = Pb, Sr [5], Fig. 4), or three-dimensional network (e.g. $[Ca(XeF_2)_{2.5}](AsF_6)_2$ [7]).

2.2.1. The influence of the metal center

The influence of the metal center will be shown on the examples of the compounds $[M(XeF_2)_4](AsF_6)_2$ with M = Mg, Ca, Cd. The crystal structure of $[Mg(XeF_2)_4](AsF_6)_2$ represents the first molecular structure found in the system $M^{n+}(AF_6^-)_n/XeF_2/aHF$. The type of the structure is a consequence of a small Mg^{2+} ion and therefore low coordination number of Mg (CN = 6) and the covalency of the Mg–F bond. The Mg–F(Xe) and Mg–F(As) distances in the $[Mg(XeF_2)_4](AsF_6)_2$ are practically the same. This indicates that F ligands from monodentate AsF_6^- can, in terms of relative Lewis basicity, compete effectively with F^- ligands of XeF_2 molecules in coordinating to the Mg^{2+} ion. The electron charge transfer from the XeF_2 molecule to the cation due to the covalent character of the Mg–F bond renders the XeF_2 molecule less capable of bridging two magnesium cations [9].

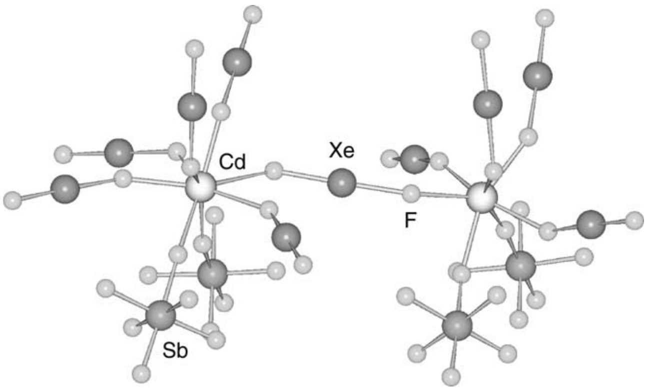

Fig. 1. Dimer in the structure of $[Cd_2(XeF_2)_{10}](SbF_6)_4$.

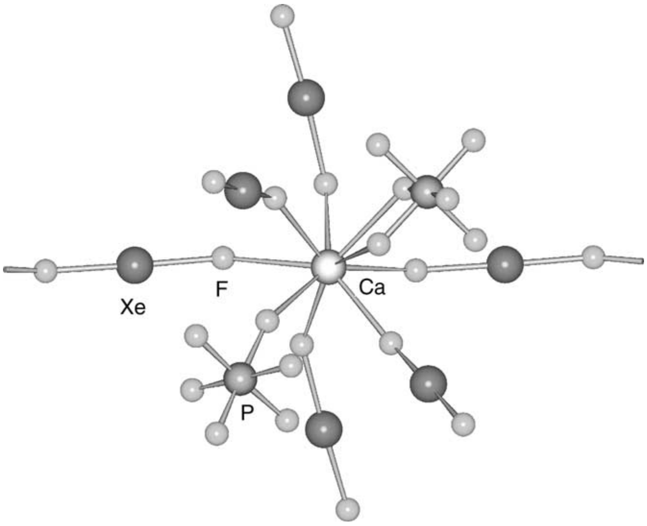

Fig. 2. Coordination sphere of Ca in the chain structure of $[Ca(XeF_2)_5](PF_6)_2$.

In accordance with the much higher electron affinity of Cd^{2+} (16.91 eV [17]) than that of Ca^{2+} (11.87 eV [17]) a higher charge transfer from XeF_2 molecule to the metal ion, and therefore a higher degree of covalency in the M–F bond, is expected in the case of Cd compound than in the case of

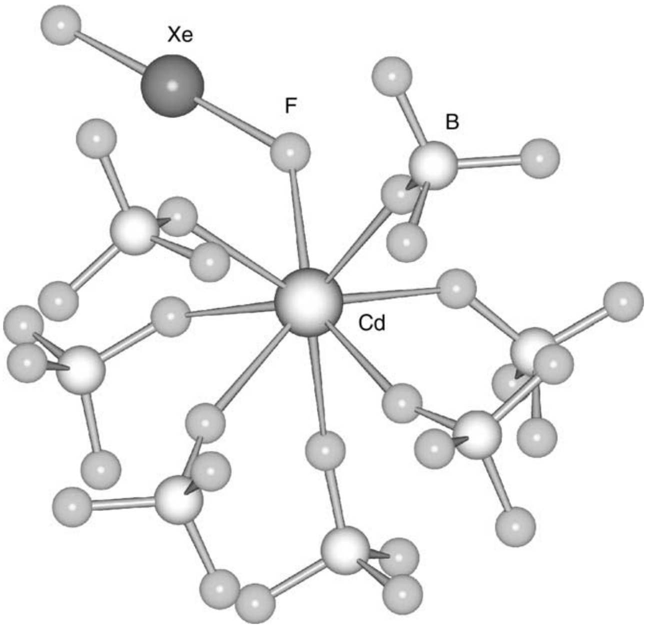

Fig. 3. Coordination sphere of Cd in the first structure with XeF_2 ligand and BF_4^- anions.

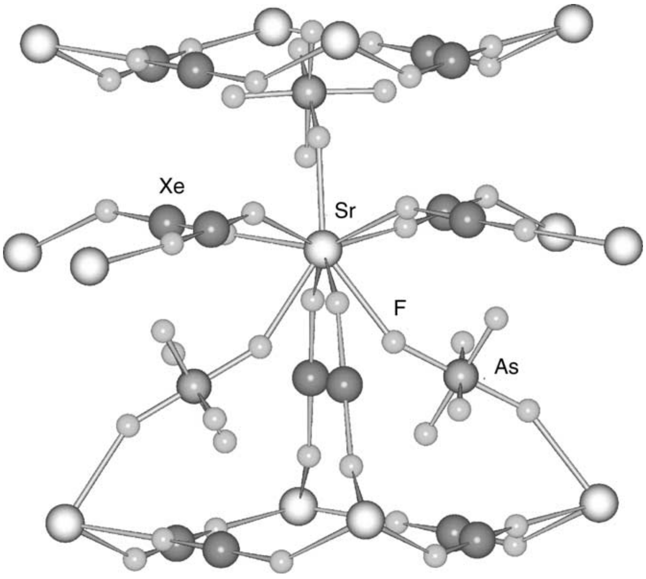

Fig. 4. Part of interconnected double layers in the structure of [Sr(Xe-F_2)$_3$](AsF_6)$_2$.

Ca compound. This makes bridging interactions in the Cd structure less favorable. In the compound [Cd(Xe-F_2)$_4$](AsF_6)$_2$ there are only two bridging XeF_2 molecules, resulting in the chain arrangement of crystal packing [10], while in [Ca (XeF_2)$_4$](AsF_6)$_2$ there are four bridging XeF_2 molecules yielding the layer structure [7]. It is evident that with changing only the cation three different crystal structures were obtained: molecular, chain and layer structure. In all three structures only XeF_2 molecules are connecting the metal centers.

2.2.2. *The influence of the anion*

The effect of the anion is much less pronounced what could be seen in the case of the isostructural compounds [Mg(XeF_2)$_2$](AF_6)$_2$, A = As [9], Sb [18]. The next example are the compounds Nd(XeF_2)$_{2.5}$(AsF_6)$_3$, **1** [4] and Nd(XeF_2)$_3$(SbF_6)$_3$, **2** [18]. AsF_6^- is a better Lewis base as SbF_6^-, which means that the interactions of the six AsF_6^- anions with Nd^{3+} ion are stronger as similar interactions with the SbF_6^- anions. As a consequence of this the interactions of the negatively charged F ligands of the XeF_2 molecules are stronger in the case of the compound **2**. This results in a shift of the negative charge on the XeF_2 molecules towards the metal center and therefore in a lower ability of XeF_2 ligands in this compound to act as bridging ligands. In the case of the compound **1** the interactions of XeF_2 ligands are weaker providing one XeF_2 molecule to be able to act as a bridging ligand and thus yielding a double-chain. This is in accordance also with the Raman spectra of the compounds 1:3, showing that the bond M–F(Xe) is stronger in the case of SbF_6^- anion (ν(Xe–F) 576 cm^{-1}) than in the case of AsF_6^- anion (ν(Xe–F) is 568 cm^{-1}).

The difference between the compounds **1** and **2** is really very subtle. The compound **1** exist also in the mole ratio 1:3 but is losing half of the XeF_2 molecule per formula when it is crystallized out of the aHF solution yielding only the compound **1** (see Table 2).

2.3. *Raman spectra*

Raman spectroscopy of the compounds [M^{n+}(XeF_2)$_p$](AF_6^-)$_n$ is an important tool of characterization of these compounds. The high polarizability of xenon usually results in the Raman active modes of xenon fluorides and their complexes having intense Xe–F stretching modes. Modes involving A–F and M–F vibrations are usually far less intense. The totally symmetric stretching mode for XeF_2 [19] and symmetrical XeF_2 in complexes such as XeF_2·(XeF_5AsF_6)$_2$ [20] is at or near 497 cm^{-1}. When XeF_2 is distorted, that means it is on the ionization path way toward $XeF^+ + F^-$, the band at 497 cm^{-1} is replaced by two bands. The band at higher frequency is labeled as the short-bond Xe–F stretch (ν(Xe–F)) and that at lower frequency as the long-bond Xe–F stretch (ν(Xe$\cdots$F)). The frequency for ν(Xe–F)$^+$ should be at or higher than 600 cm^{-1} [21].

Table 2
Stretching modes of XeF_2 as a nonbridging and a bridging ligand[a]

Metal ion	Anion	Number of XeF_2	ν(Xe–F) (cm^{-1})	ν(XeF_2) (cm^{-1})	Ref.
Mg^{2+}	AsF_6^-	4	565(10)		[9]
Mg^{2+}	AsF_6^-	2	578(10), 585(sh)		[9]
Mg^{2+}	SbF_6^-	2	572(10) 582(sh)		[18]
Ca^{2+}	AsF_6^-	4	546(7.1)	533(10)	[7]
Ca^{2+}	AsF_6^-	2.5		519(10), 528(sh)	[7]
Ca^{2+}	PF_6^-	5	542(10)	520(4.0)	[11]
Sr^{2+}	AsF_6^-	3		531(10)	[5]
Pb^{2+}	AsF_6^-	3		514(10)	[5]
Ba^{2+}	SbF_6^-	5		510(10), 521(58)	[6]
Cd^{2+}	AsF_6^-	4	547(10)	521(5.1)	[10]
Cd^{2+}	SbF_6^-	5	544(10)		[16]
Nd^{3+}	AsF_6^-	2.5	575(10), 584(8.9)	534(1.5)	[4]
Nd^{3+}	AsF_6^-	3	568(10)		[4]
Nd^{3+}	SbF_6^-	3	566(9.1), 576(10)		[18]

[a] Intensities are in parentheses.

The strength of the interactions of nonbridging XeF_2 molecules with the metal center is expressed by the position of ν(Xe–F) band which is in the range from 544 to 584 cm^{-1} (Table 2), while the stretching band of the bridging XeF_2 should be close to the vibrations of the free XeF_2 molecule (at 497 cm^{-1}). All $\nu(XeF_2)$ vibration modes of the bridging XeF_2 molecules are in the range from 500 to 535 cm^{-1}. The enhancement of this stretching frequency is probably associated with the high Coulomb field (between two M^{n+} cations) where this semi-ionic molecule is in.

The AF_6^- octahedra are usually deformed due to the formation of strong fluorine bridges between M atoms via AF_6^- units and interactions of F atoms in AF_6^- units with positive Xe atoms of XeF_2 molecules. The O_h symmetry is reduced and instead of three Raman active modes more bands could be assigned to AF_6^- vibrations. Some bands are split and some forbidden bands can appear.

3. Conclusion

Noble gas fluorides XeF_2, XeF_4 and XeF_6 were used as possible ligands to metal ions in the systems $M^{n+}(AF_6^-)_n$ with M is Mg, Ca, Sr, Ba, Cd, Pb and lanthanides, A is P, As, Sb, Bi/aHF and $M(BF_4)_2$/aHF. XeF_2 with its relatively small formula volume, linear shape, semi-ionic character and the charge of each F ligand nearly $-0.5e$, competes effectively with AF_6^-, and to lower extend with BF_4^-, in providing Coulomb energy. A whole series of new coordination compounds of the type $[M^{n+}(XeF_2)_p](AF_6^-)_n$ and $[Cd(XeF_2)](BF_4)_2$ was isolated. The influence of the metal ion, its effective nuclear charge, character of M–F bond and Lewis basicity of the AF_6^- anions was analyzed in the above-mentioned series.

In the case of XeF_4, as a ligand, up to now only one compound, $[Mg(XeF_4)](AsF_6)_2$, was isolated. XeF_6 is too strong Lewis base so that the above-mentioned metal cations are enough strong Lewis acid to withdraw F^- from XeF_6 thus yielding $Xe_2F_{11}^+$ and XeF_5^+ salts and corresponding binary fluorides.

4. Experimental details

4.1. General experimental procedure

Volatile materials (aHF, AsF_5, PF_5, SbF_5, BF_3, XeF_2, XeF_4, XeF_6, KrF_2) were manipulated in an all-Teflon vacuum line equipped with Teflon valves. Nonvolatile materials that were sensitive to traces of moisture were handled in an argon atmosphere in a dry box (maximum water content was less than 1 ppm). PFA reaction vessels, equipped with a Teflon valve and a Teflon-covered mixing bar were used for the syntheses. T-shaped reaction vessels constructed from PFA tubes of different diameters joined at right angle and equipped with a Teflon were used for crystallization.

4.2. Reagents

Metal binary fluorides were used as purchased. Their purity was checked by elemental analysis. Anhydrous HF (Fluka, purum) was treated with K_2NiF_6 (Ozark-Mahoning, 99%) for several days prior to use. PF_5 was prepared by high-pressure fluorination of P_2O_5, as previously described [22]. AsF_5 was prepared by high-pressure fluorination of As_2O_3, as previously described for PF_5. Their purity was checked by IR spectroscopy. SbF_5 (Merck >98%) was distilled prior to use. BiF_5 was prepared by fluorination of BiF_3 with elemental fluorine at pressure 50 bar and temperature 350 °C. The purity of BiF_5 was checked by Raman spectroscopy and X-ray powder diffraction pattern. BF_3 (Ucar, 99.5%) was used as purchased. $M(AF_6)_2$ (M = alkaline earth metal, Pb; A = As, Sb) were prepared by the reaction between MF_2 and AsF_5 or SbF_5 in aHF as described earlier [23,24].

XeF_2 was prepared by the photochemical reaction between Xe and F_2 at room temperature [25]. XeF_4 was prepared by photochemical reaction between xenon and excess fluorine at room temperature in the presence of NiF_2 as a catalyst [26]. XeF_6 was prepared by the reaction between xenon and fluorine under pressure in the presence of nickel difluoride as a catalyst at 120 °C [27]. KrF_2 was prepared as described elsewhere [28].

4.3. Preparation procedure

The coordination compounds of the type $[M^{n+}(XeF_2)_p](AF_6^-)_n$ were prepared by the reaction of the corresponding $M^{n+}(AF_6^-)_n$ with excess of XeF_2 in aHF. After the reaction was completed solvent and excess of XeF_2 were pumped away. $[Cd(XeF_2)](BF_4)_2$ was prepared by the reaction of $Cd(BF_4)_2$ and excess of XeF_2 in aHF as a solvent. In the case of phosphates and borates also direct reaction of binary fluorides, XeF_2, aHF and PF_5 or BF_3 under pressure were used.

4.4. Raman spectroscopy

Raman spectra of powdered samples in sealed quartz capillaries were recorded on a Renishaw Raman Imaging Microscope System 1000, with the 632.8 nm exciting line of a He–Ne laser. The course of the reaction was followed by recording Raman spectra directly in the PFA reaction vessels.

4.5. X-ray powder diffraction

X-ray powder diffraction patterns of samples in sealed quartz capillaries were obtained with a 114 mm diameter Debye–Scherrer camera with X-ray film, using CuKα radiation (λ = 154.18 pm) with a Ni filter.

4.6. Crystal structure determination

Single-crystal data were collected using a Mercury CCD area detector coupled to a Rigaku AFC7 diffractometer with graphite-monochromated MoKα radiation. All details regarding crystal structure determinations are given elsewhere [5–7,9–13,15,16,18].

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