Oxidation of Ruthenium and Iridium Metal by XeF₂ and Crystal Structure Determination of [Xe₂F₃][RuF₆]·XeF₂ and [Xe₂F₃][MF₆] (M = Ru, Ir)

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Abstract

Salts containing [Xe₂F₃]⁺ cations and [MF₆]⁻ anions (M = Ru, Ir) were synthesized by the oxidation of metal with excess of XeF₂ in anhydrous hydrogen fluoride (aHF) as a solvent. Single crystals of [Xe₂F₃][RuF₆]·XeF₂, [Xe₂F₃][RuF₆] and [Xe₂F₃][IrF₆] crystallized by slow evaporation of the solvent. [Xe₂F₃][RuF₆]·XeF₂ crystallizes in a triclinic P–1 space group (a = 8.3362(1) Å, b = 8.8197(2) Å, c = 9.3026(4) Å; α = 68.27(1)°, β = 63.45(1)°, γ = 82.02°, V = 568.09(9) Å³ (Z = 2)). Discrete [Xe₂F₃]⁺, XeF₂ and [RuF₆]⁻ units are found in the asymmetric unit. [Xe₂F₃][RuF₆] and [Xe₂F₃][IrF₆] compounds are isomorphic and crystallize in a monoclinic Cc space group (α = 14.481(3) Å (Ru); 14.544(3) Å (Ir); b = 8.0837(8) Å (Ru), 8.0808(7) Å (Ir), c = 10.952(2) Å (Ru), 11.014(2) Å (Ir); β = 136.825(6)° (Ru), 139.954(7)°, V = 877.2(3) Å³ (Ru), 883.6(3) Å³ (Ir); Z = 4). The asymmetric unit in the [Xe₂F₃][MF₆] (M = Ru, Ir) consists of one [Xe₂F₃]⁺ and one [MF₆]⁻ unit.

Keywords: Noble gas fluorides, ruthenium, iridium, crystal structure

1. Introduction

XeF₂ is the most stable and easily handled noble gas fluoride and therefore its chemistry is very extensive. Basic information about XeF₂, its properties and the possibilities that it offers can be found in review paper and book and the references listed therein. One of its unexpected and interesting abilities is also binding to metal centres in order to form coordination compounds. A large variety of such compounds has been found in previous years. The formation of XeF₂ adducts with main-group and transition-metal Lewis acidic pentafluoridometalates – MF₅ is known for decades. So far three types of such compounds were found: 2XeF₂·MF₅, Xe₂F₃·MF₅ and Xe₂F₃·(MF₅)₂. The degree of ionic character in these compounds varies depending on the Lewis acidity of the respective pentafluoride. Compounds were mainly characterized by vibrational spectroscopy and can be written as salts [Xe₂F₃][MF₅], [XeF₂][MF₅] and [XeF][M₂F₁₁], especially in the case of reactions with strong Lewis acids (for example AsF₅, SbF₅, BiF₅...). The formation of 2:1 compounds was found in the cases where M was As, Sb, Bi, Ta, Ru, Os and Ir. From this type of compounds (2:1 composition) [Xe₂F₃][MF₆] (M = As, Sb) were also structurally characterized. The compounds with composition 1:1 (XeF²MF₆) and 1:2 (XeF²M₂F₁₁) were obtained for the most of the MF₅ mentioned above. Some of them were structurally characterized: [XeF][MF₅] (M = As, Sb, Bi, Ru and Ir) and [XeF][M₂F₁₁] (M = Sb, Bi). One of the latest structurally characterized examples is also and [XeF][IrSbF₁₁] with two different metals in the anion. [Xe₂F₃]⁺ and [XeF]⁺ cations were recently found with nonoctahedral anion in the xenon(II) polyfluoridotitanates(IV): [Xe₂F₃][TiF₃]₃ and [XeF][TiF₃]₈.[14]

The present study reports about the synthesis and structural characterization of three noble gas salts containing [Xe₂F₃]⁺ cation: [Xe₂F₃][RuF₆]·XeF₂, [Xe₂F₃][RuF₆] and [Xe₂F₃][IrF₆].

2. Experimental

2.1. General Experimental Procedure and Reagents

Volatile materials (anhydrous HF, F₂) were handled in an all PTFE vacuum line equipped with PTFE (polyte-
trafluoroethylene) valves. The manipulations of the non-volatile materials were carried out in a glove-box (M. Braun). The residual water in the atmosphere within the glove-box never exceeded 1 ppm. The reactions were carried out in FEP (tetrafluoroethylene-hexafluoropropylene; Polytetra GmbH, Germany) reaction vessels (height 250–300 mm with inner diameter 16 mm and outer diameter 19 mm) equipped with PTFE valves and PTFE coated stirring bars. T-shaped reaction vessels from PTFE, which were constructed as described earlier, were used for the crystallization process. Prior to their use all reaction vessels were passivated with elemental fluorine. Fluorine was used as supplied (Solvay Fluor and Derivate GmbH, Germany). Anhydrous HF (Linde, 99.995%) was treated with K$_3$NiF$_6$ (Advance Research Chemicals, Inc.) for several hours prior to use. XeF$_2$ was synthesized by photochemical reaction between Xe and F$_2$. Caution: aHF, F$_2$ and XeF$_2$ must be handled with great care in a well-ventilated fume hood, and protective gear must be worn at all times.

2.2. Synthesis and Characterization Procedures

Synthetic procedures for the ruthenium and iridium compounds were the same. Metal powder (Ru: 0.215 g, 2.13 mmol, Ir: 0.430 g, 2.20 mmol) was added into a reaction vessel inside the glove-box. The aHF was condensed into the reaction vessel at –196 °C at the vacuum line. Large excess of XeF$_2$ (mole ratio M : XeF$_2$ was approximately 1:10) was weighed into another reaction vessel in a glove-box. The aHF was condensed into the reaction vessel at –196 °C at the vacuum line. Large excess of XeF$_2$ (mole ratio M : XeF$_2$ was approximately 1:10) was weighed into another reaction vessel inside the glove-box. Anhydrous HF was added to the XeF$_2$ and the reaction vessel was warmed up to room temperature. These two reaction vessels (one with the suspension of the metal, and another with dissolved XeF$_2$) were attached in a T-shape manner and additional valve was used in order to provide completely closed system. The XeF$_2$ solution was then poured into cold reaction vessel (–196 °C) with suspension of the metal powder (Ru, Ir). The reaction vessel was left to slowly warm up to room temperature. The reaction solution turned immediately green in the case of ruthenium but the reaction with iridium proceeded at room temperature for several days (light gray solid product). Products of the oxidation were isolated by removal of aHF and excessive XeF$_2$ under dynamic vacuum at room temperature for several days became slightly yellow with some grey precipitate – light grey solid product after its isolation. The grey solid product of ruthenium but the reaction with iridium proceeded at room temperature for several days became slightly yellow with some grey precipitate – light grey solid product after its isolation. The grey solid product of the reaction was obtained. The Raman spectra were recorded at room temperature with a Horiba Jobin Yvon LabRam-HR spectrometer equipped with an Olympus BXFM-ILHS microscope and CCD detector. The samples were excited by the 632.8 nm emission line of a He-Ne laser. Samples for measurement were transferred into the quartz capillary inside glove-box.

The crystallographic parameters and summaries of data collection for all compounds are presented in Table 1. Single-crystal data were collected on a Rigaku AFC7 diffractometer using graphite monochromatized MoK$\alpha$ radiation at 200 K. Crystals were immersed into perfluorinated oil in glove-box and further on selected under the microscope. An empirical multi-scan absorption correction was applied. All structures were solved by direct methods using SIR-92 and SHELXS-97 programs (teXan crystallographic software package of Molecular Structure Corporation) and refined with SHELXL-97, implemented in program package WinGX. Full-matrix least-squares refinements based on $F^2$ were carried out for the positional and thermal parameters for all non-hydrogen atoms. The figures were prepared using DIAMOND 3.1 software. Further details of the crystal-structure investigation may be obtained from the Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany, on quoting the depository number: CSD-430805 for [XeF$_2$] [RuF$_6$], CSD-430806 for [XeF$_2$] [IrF$_6$] and CSD-430807 for [XeF$_2$] [RuF$_6$]:XeF$_2$, respectively.

3. Results and Discussion

The oxidation power of XeF$_2$ was used in order to prepare previously mentioned Ru(V) and Ir(V) compounds. XeF$_2$ dissolved in anhydrous hydrogen fluoride (aHF) was also used as selective inorganic fluorinating reagent for oxidation and fluorination of Ir and RuF$^3$ almost three decades ago with final products being IrF$_2$ and RuF$_2$. Ruthenium metal was oxidized rapidly with vigorous reaction being observed during the warming of the reaction vessel from –196 °C to room temperature. A clear, slightly green solution was obtained. We used a slightly modified procedure for the preparation of the ruthenium compound $(\text{XeF}_2)_n\text{MF}_6$ (M = Ru, Ir). The product was further used for the synthesis of [Ba$(\text{XeF}_2)_2$]$_2$[RuF$_6$]$_2$. The same reaction with iridium proceeded for several weeks. The solution was red-brown at the beginning and after several days became slightly yellow with some grey precipitate – same as the solid product after its isolation. The grey solid obtained by the oxidation of Ir metal with XeF$_2$ was re-dissolved in aHF and a clear, slightly green solution was obtained. Products of the reactions were also monitored by Raman spectroscopy, which shows (Figure 1) that in both cases the compound with composition $(\text{XeF}_2)_n\text{MF}_6$ was obtained. An alternative method used for the preparation of related compounds with Ru(V) (KRuF$_6$, LiRuF$_6$) and Ir(V) (KIrF$_6$, LiIrF$_6$) with al-
kaline metals is the room temperature oxidation with elemental fluorine in the presence of Lewis base (KF) in aHF. One of the recently published ways to prepare soluble iridium compounds, which seems to be important for modern “urban mining”, is the reaction of the metal with tetrafluorobromates (MBrF₄; M = K, Rb, Cs).

Figure 1. Raman spectra of \([\text{Xe}_2\text{F}_3\text{]}\)\([\text{RuF}_6\text{]}\)·\(\text{XeF}_2\) (a) and \([\text{Xe}_2\text{F}_3\text{]}\)\([\text{IrF}_6\text{]}\)·\(\text{XeF}_2\) (b)

For solid \(\text{XeF}_2\) the band at 497 cm\(^{-1}\) is characteristic. The bands at 506 cm\(^{-1}\), 515 cm\(^{-1}\) in ruthenium compound and 506 cm\(^{-1}\) and 516 cm\(^{-1}\) in iridium compound can be attributed to the \(\text{XeF}_2\) weakly associated with \([\text{Xe}_2\text{F}_3\text{]}\)\(^+\) cations and \([\text{MF}_6\text{]}\)\(^-\) anions in the \([\text{Xe}_2\text{F}_3\text{]}\)\([\text{MF}_6\text{]}\)·\(\text{XeF}_2\) product. Similar positions and assignment of these bands were observed in some other cases in the system \(\text{XeF}_2\)·\(\text{MF}_6\) (M = Sb, Ta, Nb). The weakly associated \(\text{XeF}_2\) was found in the melt of the compounds. “Free” \(\text{XeF}_2\) was also found in the compounds \(\text{XeF}_2\)·\(\text{XeF}_6\)·\(\text{AsF}_5\) and \(\text{XeF}_2\)·\(2(\text{XeF}_6)\)·\(2(\text{AsF}_5)\), where the Raman bands depend on the interaction of the so called “free” \(\text{XeF}_2\) molecule with cations and anions and consequent distortion of its shape. “Free” \(\text{XeF}_2\) in \(\text{XeF}_2\)·\(2(\text{XeF}_6)\)·\(2(\text{AsF}_5)\) is probably in a completely symmetric environment, therefore the band assigned to it coincides with the symmetric stretching frequency in molecular \(\text{XeF}_2\) (497 cm\(^{-1}\)). On the other hand Raman spectrum of \(\text{XeF}_2\)·\(\text{XeF}_6\)·\(\text{AsF}_5\) doesn’t show a symmetric vibration of molecular \(\text{XeF}_2\) but two bands at 557 cm\(^{-1}\) and 429 cm\(^{-1}\) which represent a distorted \(\text{XeF}_2\) molecule meaning that \(\text{XeF}_2\) in this compound can be far from “free”. Linear distortion of \(\text{XeF}_2\) was found also in the Raman spectrum of \(\text{XeF}_2\)·\(\text{XeF}_6\)·\(\text{AsF}_5\)·\(\text{RuF}_6\). Bands at 578 cm\(^{-1}\) and 586 cm\(^{-1}\) in the ruthenium compound and bands at 578 cm\(^{-1}\) and 587 cm\(^{-1}\) in iridium compound can be confidently assigned to the \(\text{Xe-F}_2\) stretch vibrations of the \([\text{Xe}_2\text{F}_3\text{]}\)\(^+\) cation. They are in the region that is characteristic for such vibrations (from ca. 575 cm\(^{-1}\) to 600 cm\(^{-1}\)). The bands at 646 cm\(^{-1}\), 667 cm\(^{-1}\) and 265 cm\(^{-1}\) for the ruthenium compound and 662 cm\(^{-1}\) and 243 cm\(^{-1}\) for the iridium can be assigned to the vibration of the \([\text{MF}_6\text{]}\)\(^-\) anions. Products with additional \(\text{XeF}_2\) are not stable under...
dynamic vacuum at room temperature. They release XeF₂, which leads to the formation of the [XeF₃][MF₆] and further on to the [XeF][MF₆] (M = Ru, Ir) compounds. According to the mass balance of the reactions and Raman spectra, the [XeF][MF₆] (M = Ru, Ir) salts seem to be stable at room temperature under dynamic vacuum. Raman analysis of the slow removal of XeF₂ under dynamic vacuum in the case of ruthenium is shown in Figure 2. In the spectrum shown on the Figure 2a (black colour) all three phases can be found: [XeF₃][RuF₆]·XeF₂, [XeF₃][RuF₆] and [XeF][RuF₆]. With the release of the XeF₂, phases [XeF₃][RuF₆] and [XeF][RuF₆] are found (Figure 2b (blue colour)). Prolonged pumping on the vacuum system (overnight) ended up with those published previously.4

3. 1. Crystal Structure Determination of [XeF₃][RuF₆]·XeF₂ and [XeF₃][MF₆] (M = Ru, Ir)

Three compounds in this system were structurally characterized. Summary of crystal data and refinement results for [XeF₃][RuF₆]·XeF₂ and [XeF₃][MF₆] (M = Ru, Ir) are presented in Table 1 and selected distances and angles are found in Table 2. Several unsuccessful attempts were made in order to prepare suitable single crystals of the [XeF₃][IrF₆]·XeF₂. So far we were only able to resolve the structure of [XeF₃][IrF₆].

The structure of [XeF₃][RuF₆]·XeF₂ consists of discrete [XeF₃]⁺, XeF₂ and [RuF₆]⁻ units (Figure 3). XeF₂ molecules and [XeF₃]⁺ cations are oriented roughly perpendicularly to each other. When viewed along (–3 –4 3) direction alternating cationic and anionic layers could be seen. Anions are separated by XeF₂ molecules (Figure 4). The compound is structurally related to [Kr₆F₅]SbF₆·Krf₂ in which the crystal packing consists of alternating cation and equally populated anion/KrF₂ layers.31

XeF₂ molecules are nearly linear with distances being 1.980(7) and 1.992(7) Å and angle F10-Xe-F11 of 178.9(4)°. [XeF₃]⁺ cation exhibit a planar, V shape configuration with nearly symmetrical Xe-F bonds (2.139(7) and 2.152(7) Å) and a Xe-F₆ angle of 154.3(4)°. The [RuF₆]⁻ anions are slightly distorted octahedra with Ru–F bond distances in the range from 1.904 to 1.98(2) Å.

Table 1. Crystal data and structure refinement for [XeF₃][RuF₆]·XeF₂ and XeF₃MF₆ (M = Ru, Ir)

|                         | [XeF₃][RuF₆]·XeF₂ | [XeF₃][RuF₆] | [XeF₃][IrF₆] |
|-------------------------|-------------------|--------------|--------------|
| Empirical formula       | RuXeF₁₃           | RuXeF₉       | IrXeF₉      |
| Formula weight          | 703.94            | 534.67       | 625.8        |
| Wavelength, MoKα        | 0.71069 Å         | 0.71069 Å    | 0.71069 Å   |
| Crystal system,         | triclinic         | monoclinic   | monoclinic  |
| Space group             | P–1               | Cc           | Cc           |
| Temperature, K          | 200               | 200          | 200          |
| Unit cell dimensions    |                   |              |              |
| a, Å                    | 8.3362(1)         | 14.481(3)    | 14.544(3)    |
| b, Å                    | 8.8197(2)         | 8.0837(8)    | 8.0808(7)    |
| c, Å                    | 9.3026(4)         | 10.952(2)    | 11.014(2)    |
| α, °                    | 68.27(1)          | 90           | 90           |
| β, °                    | 63.45(1)          | 136.825(6)   | 136.954(7)   |
| γ, °                    | 82.02(2)          | 90           | 90           |
| V, Å³                   | 568.09(9)         | 877.2(3)     | 883.6(3)     |
| Z                       | 2                 | 4            | 4            |
| Calculated density, g/cm³ | 4.115            | 4.048        | 4.704        |
| Absorption coeff., mm⁻¹ | 10.289            | 9.477        | 22.745       |
| F(000)                  | 610               | 932          | 1064         |
| Crystal size, mm        | 0.08×0.06×0.04    | 0.10×0.10×0.08 | 0.10×0.07×0.05 |
| Colour                  | colourless        | colourless   | colourless   |
| Theta range for data collection, deg | 2.487–28.6986 | 3.2522–28.5637 | 3.1256–27.9323 |
| Limiting indices        | –8 ≤ h ≤ 11, –10 ≤ k ≤ 11 | –18 ≤ h ≤ 16, –10 ≤ k ≤ 16 | –18 ≤ h ≤ 17, –6 ≤ k ≤ 17, –6 ≤ l ≤ 14 |
| Measured reflections    | 2259              | 1174         | 1129         |
| Used in refinement      | 1837              | 890          | 1074         |
| Free parameters         | 137               | 111          | 111          |
| Goodness-of-fit on F²   | 1.134             | 1.134        | 1.067        |
| R indices               | R₁ = 0.0608       | R₁ = 0.0371  | R₁ = 0.0382  |
|                         | wR₁ = 0.1775      | wR₁ = 0.0849 | wR₁ = 0.0963 |
| Largest diff. peak and hole, e Å⁻³ | 1.758 and –3.063 | 1.579 and –1.236 | 1.699 and –2.329 |
Table 2. Selected distances (Å) and angles (°) in [XeF$_3$][RuF$_6$] · XeF$_2$, [XeF$_3$][MF$_6$] (M = Ru, Ir)

|            | [XeF$_3$] | [XeF$_3$] | [XeF$_3$] |
|------------|------------|------------|------------|
|            | [RuF$_6$]  | [RuF$_6$]  | [IrF$_6$]  |
| Xe1-F1     | 2.139(7)   | 2.09(1)    | 2.12(2)    |
| Xe2-F1     | 2.152(7)   | 2.17(1)    | 2.15(2)    |
| Xe1-F2     | 1.913(8)   | 1.90(2)    | 1.88(2)    |
| Xe2-F3     | 1.919(6)   | 1.92(2)    | 1.96(2)    |
| Xe3-F10    | 1.980(7)   |            |            |
| Xe3-F11    | 1.992(7)   |            |            |
| Xe1-F1-Xe2 | 154.3(4)   | 161.5(5)   | 161.3(8)   |
| F10-Xe3-F11|            | 178.9(4)   |            |

Figure 3. Asymmetric unit in [XeF$_3$][RuF$_6$] · XeF$_2$ with thermal ellipsoids drawn at 50 % probability level.

The Xe centres from both XeF$_2$ and [XeF$_3$]$^+$ moieties interact with fluorine atoms from another structural units. The shortest F···Xe distances of 3.190(7) and 3.216(7) Å respectively correspond to slightly elongated Ru1-F9 bond (1.861(7) Å). There are four XeF$_2$ molecules bound to one anion via Xe···F contacts of 3.301(7)–3.373(7) Å. Terminal F2 and F3 atoms from each [XeF$_3$]$^+$ also form longer contacts with Xe centres from other cations. These contacts are slightly longer (from 3.219(7) to 3.278(8) Å, correspondingly) than those in the case of XeF$_2$ molecules.

Weak Xe···F(Xe) and Xe···F(Ru) interactions connect above mentioned units into three-dimensional network (Figure 4) if contacts shorter than 3.4 Å are taken into consideration. The sum of the van der Waals radius for xenon (2.16 Å) and fluorine (1.35 Å) is 3.51 Å. The oxidizing power of XeF$_2$ was demonstrated by oxidation of ruthenium and iridium metal in aHF as a solvent. Products of the oxidation belong to the family of general Xe···F contacts in the range above 3 Å can be found.

A packing diagram along the $b$-axis is presented in Figure 6. A less bent structure of the [XeF$_3$]$^+$ cation is found in the case of [XeF$_3$][MF$_6$] (M = Ru, Ir) compared to the [XeF$_3$][RuF$_6$] · XeF$_2$. The Xe···F angle is 161.5(5)° in the ruthenium compound and 161.3(8)° in the iridium compound. These angles are in agreement with those in isostructural monoclinic [XeF$_3$][SbF$_6$] (Xe···F = 160.3 °). Bridging angle of Xe···F···Xe in [XeF$_3$]$^+$ cations vary from 139.8° as observed in trigonal [XeF$_3$] [AsF$_6$]$^+$ to the widest one being 164.3° as observed in [XeF$_3$][Ti$_2$F$_3$]$_2$. The oxidizing power of XeF$_2$ was demonstrated by oxidation of ruthenium and iridium metal in aHF as a solvent. Products of the oxidation belong to the family of non-linear structure of the [XeF$_3$]$^+$ cation with a bridging angle of 168°.

Figure 4. Packing diagram of [XeF$_3$][RuF$_6$] along $b$-axis.

Figure 5. Asymmetric units in [XeF$_3$][RuF$_6$] and [XeF$_3$][IrF$_6$] with thermal ellipsoids drawn at 50 % probability level.

Figure 6. Packing diagram of [XeF$_3$][RuF$_6$] along $b$-axis.

4. Conclusions

The oxidizing power of XeF$_2$ was demonstrated by oxidation of ruthenium and iridium metal in aHF as a solvent. Products of the oxidation belong to the family of no-
ble gas compounds with \([\text{Xe}_2\text{F}_3]^+\) cations. Salt with composition \([\text{Xe}_2\text{F}_3][\text{RuF}_6]\cdot \text{XeF}_2\) was structurally characterized. The synthesis and characterization with Raman spectroscopy of the new salt \([\text{Xe}_2\text{F}_3][\text{IrF}_6]\cdot \text{XeF}_2\) are also reported. Single crystal structures of \([\text{Xe}_2\text{F}_3][\text{RuF}_6]\) and \([\text{Xe}_2\text{F}_3][\text{IrF}_6]\) were determined.

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Povzetek
Z oksidacijo kovine (Ru, Ir) s presežnim XeF₂ v brezvodnem vodikovem fluoridu (aHF) kot topilu smo pripravili spojine s kationi [Xe₆F₃⁺] in anioni [MF₆⁻] (M = Ru, Ir). Kristale [Xe₆F₃]([RuF₆] ∙ XeF₂ + [Xe₆F₃][RuF₆] in [Xe₆F₃][IrF₆]), ki so bili primerni za rentgensko strukturno analizo, smo pripravili s počasnimi izhlapevanjem topila. [Xe₆F₃][RuF₆] ∙ XeF₂ kristalizira v triklinem kristalnem sistemu; prostorska skupina P-1 (a = 8,3362(1) Å, b = 8,8197(2) Å, c = 9,3026(4) Å; α = 68,27(1)°, β = 63,45(1)°, γ = 82,02°, V = 568,09(9) Å³ (Z = 2)). V asimetrični enoti spojine se nahajajo [Xe₆F₃⁺], XeF₂ in [RuF₆]⁻. Spojini [Xe₆F₃][RuF₆] in [Xe₆F₃][IrF₆] sta izostrukturni in kristalizirata v monoklinskem kristalnem sistemu; prostorska skupina Cc (a = 14,481(3) Å (Ru); 14,544(3) Å (Ir); b = 8,0837(8) Å (Ru), 8,0808(7) Å (Ir), c = 10,952(2) Å (Ru), 11,014(2) Å (Ir); β = 136,825(6)° (Ru), 139,954(7)°, V = 877,2(3) Å³ (Ru), 883,6(3) Å³ (Ir); Z = 4). Asimetrični enota [Xe₆F₃][MF₆] (M = Ru, Ir) je sestavljena iz [Xe₆F₃⁺] in [MF₆⁻] ionov.