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THE CRYSTAL STRUCTURE OF $[\text{XeF}_5^+]_2\text{PdF}_6^{2-}$

by

K. Leary, D. H. Templeton, A. Zalkin and N. Bartlett*

ABSTRACT

Crystals of $[\text{XeF}_5^+]_2\text{PdF}_6^{2-}$ are yellow needles. The unit cell is orthorhombic with $a = 9.346(6)$, $b = 12.786(7)$, $c = 9.397(6)$ Å, $V = 1122.9$ Å³, $Z = 4$, $D_c = 3.91$ gcm⁻³. Structure refinement has proceeded satisfactorily in space group $Pca2_1$ (No. 29) using three dimensional graphite monochromatized X-ray Mo K α data. With anisotropic temperature factors for all atoms, a final conventional R factor of 0.0256 (including zero weight data) for 1464 independent reflections, was obtained.

The asymmetric structural unit contains two crystallographically distinct XeF_5^+ ions and a PdF_6^{2-} ion. The PdF_6^{2-} ion is almost octahedral and only one Pd-F interatomic distance (= 1.860(6)Å) departs significantly from the average Pd-F distance of 1.893 Å. The XeF_5^+ ions each approximate to C_{4v} symmetry and are not significantly different in size, shape or coordination. The cations are characterized by $F_{\text{axial}}-\text{Xe}-F_{\text{equatorial}}$ angles of $\sim 79^\circ$, and average axial and equatorial bond lengths of 1.81 Å and 1.84 Å, respectively. The individual Xe-F distances of the same type (axial or equatorial) do not depart significantly from the average values for the type.

The coordination of the XeF_5^+ ions, by the F atoms of anions, in this structure, is similar to the cation coordination observed in $\text{XeF}_5^+\text{AsF}_6^-$.

INTRODUCTION

In the course of experiments¹ aimed at a more convenient synthesis of palladium tetrafluoride² we obtained the XeF_6 adducts of PdF_4 , $2\text{XeF}_6 \cdot \text{PdF}_4$ and $4\text{XeF}_6 \cdot \text{PdF}_4$. These stoichiometries are similar to those reported previously by Pullen and Cady³ for the XeF_6 adducts of the germanium and tin tetrafluorides. Although Raman data¹ indicated that the PdF_4 adducts were $[\text{XeF}_5^+]_2\text{PdF}_6^{2-}$ and $[\text{Xe}_2\text{F}_{11}^+]_2\text{PdF}_6^{2-}$, respectively, full structural investigations were desirable. Such ionic formulations are consistent with XeF_6 being a good base⁴. It was also of value for the appraisal of other XeF_5^+ salts^{4,5,6}, to know the geometry of the cation in a doubly-charged anion environment. Suitable single crystals of the 2:1 adduct for high quality X-ray structural investigation proved to be readily preparable. The structure has been determined and is reported in this paper.

EXPERIMENTAL

$2\text{XeF}_6 \cdot \text{PdF}_4$ was prepared as described in a forthcoming paper¹. Suitable crystalline samples were obtained by heating the compound in a Monel bomb to 400° under fluorine pressure (1000 psi). The bomb was cooled slowly to room temperature (~20°/hr). Yellow, needle-shaped crystals were found adhering to the walls of the bomb. Suitable sized specimens were loaded into 0.2 mm o.d. quartz capillaries, which had been drawn down to smaller diameter at the sealed end. Crystals

were wedged into the drawn-down capillary ends with the aid of Pyrex push rods. All manipulations were carried out in a Vacuum Atmospheres Corporation Dri Lab, provided with a nitrogen atmosphere. The capillaries were temporarily sealed with a plug of Kel-F grease then removed from the drybox and sealed by drawing down in a small flame.

Crystal Data.- The crystal chosen for data collection was a yellow needle with dimensions $0.3 \times 0.2 \times 0.1$ mm. Weissenberg photographs showed $F_{16}PdXe_2$ (m.w. 673.0) to be orthorhombic, with $a = 9.346(6)$, $b = 12.786(7)$, $c = 9.397(6)$ Å, $V = 1122.9$ Å³, $z = 4$, and $D_c = 3.91$ gcm⁻³. The unit cell volume satisfies Zachariason's criterion⁷ for close packed fluoride lattices, the effective volume per fluorine atom being 17.6 Å³. The systematic absences, $0\ k\ \ell$, $\ell = 2n + 1$, and $h\ 0\ \ell$, $h = 2n + 1$, indicated the space group⁸ to be either $Pca2_1$ (No. 29) or $Pcam$ (No. 57).

X-ray Measurements.- Diffraction data were collected at room temperature ($T = 24.5 \pm 1.5^\circ$) on a Picker automatic four circle diffractometer using Mo $K\alpha$ radiation, ($\lambda = 0.7107$ Å). Accurate cell dimensions were obtained by measuring the 2θ angle for the Mo $K\alpha_1$ peak for the highest angle reflection observable along each of the principal axes. The c axis was oriented along ϕ .

Intensity data were collected and corrected as previously described⁹. The absorption coefficient $\mu = 78.7$ cm⁻¹. Although the crystal was larger than the optimum size, no absorption correction was applied. The only other difference from the previously described procedure for the data treatment⁹ was in the choice for the value of q , the arbitrary factor, employed to prevent the relative errors for large counts

becoming unrealistically small. A value $q = 0.04$ was assumed here. Three standards were checked over 200 reflections; their intensities increased slightly over the period of data collection.

Scattering factors for neutral F, Xe, and Pd were taken from Doyle and Turner¹⁰ and Cromer and Waber¹¹. Cromer and Liberman's values¹² of $\Delta f'$ and $\Delta f''$ were used for anomalous dispersion corrections.

A complete set of $\underline{h} \underline{k} \underline{l}$ and $\underline{h} \underline{k} \bar{\underline{l}}$ data were collected for $2\theta \leq 45^\circ$ and amounted to 1464 unique reflections. In the least-squares refinements 1410 data which satisfied the condition $I \geq 3\sigma(I)$ were given non-zero weight.

Structure Refinement.- At the outset, it was assumed that the structure would be centric, so the two acentric data sets were averaged to give a single centric data set. A three dimensional Patterson synthesis revealed the positions of two of the three heavy atoms, but it also indicated that the non-centric group ($\underline{Pca}2_1$, #29) was appropriate. Full matrix least-squares¹³ and a subsequent Fourier synthesis yielded the positions of the third heavy atom and several of the fluorine atoms. The refinement proceeded routinely down to $\underline{R} = 7.5\%$ with all 19 atoms placed and the heavy atoms assigned anisotropic thermal parameters. At this point, the structure seemed to have settled into a false minimum, since bond lengths and angles gave very poor agreement with previous structures of this type. After much experimentation involving location of atoms across symmetry elements (and pseudo-symmetry elements), to generate a chemically acceptable arrangement, the \underline{R} value dropped to 6.0%. Subsequent least-squares refinement with all atoms anisotropic, but using the data appropriate for the

centric space group, yielded an \underline{R} value of 2.0%. At this point the data were re-ordered to satisfy the non-centric symmetry and to take advantage of anomalous dispersion. Two polarities must be considered. Least-square refinement yielded $\underline{R} = 2.60\%$ (including zero weight data), $\underline{R} = 2.41\%$ (excluding zero weight data) for the polarity with mostly negative \underline{z} coordinates. The magnitudes of the \underline{z} parameters of the two forms did not differ by more than one standard deviation. We describe here the arbitrarily chosen structure with the mostly positive \underline{z} atomic parameters. A final difference Fourier showed that the largest residual electron density was $-1.7 \text{ e}/\text{\AA}^3$. The final value of $\underline{R}_2 = 0.0267$ and \underline{R} (including zero weight data) = 0.0256. The final conventional $\underline{R} = 2.37\%$. The standard deviation of an observation of unit weight was 0.994. The largest shift of any parameter, divided by the estimated standard deviation on the last cycle was less than 0.003. Table I gives the final positional and thermal parameters for this refinement. The F_o , standard deviations, and ΔF data are given in the microfilm version of this paper as Table II¹⁴.

Description of the Structure.- The asymmetric structural unit contains two crystallographically distinct XeF_5 groups and a PdF_6 group. The structural unit is illustrated in Figure 1.

The PdF_6 group is almost octahedral. As may be determined from Table III, only one Pd-F [Pd-F(2) - 1.860(6)] departs significantly from the average Pd-F distance of 1.893 Å. All cis F-Pd-F angles are within 5° of 90°.

The two XeF_5 groups are similar, each being an approximately \underline{C}_{4v} symmetry species, with an $F_{\text{axial}}-\text{Xe}-F_{\text{equatorial}}$ angle of $\sim 79^\circ$. The

average Xe-F axial interatomic distance of the two crystallographically distinct species is 1.813 Å and the axial Xe-F distance for each species does not depart significantly from this mean. The mean of all Xe-F equatorial interatomic distances is 1.843 Å. Again there is no significant departure from this mean value for the Xe-F equatorial distances in each species.

Each Xe atom of each XeF_5 group makes one short contact with one PdF_6 group [i.e., $\text{Xe}(2)\text{-F}(3)$, $\text{Xe}(1)\text{-F}(4)$] and two somewhat longer contacts to a second PdF_6 group. These $\text{Xe}\dots\text{F}$ contacts link XeF_5 and PdF_6 groups into 'rings' containing two species of each kind as shown in Figure 2. These 'rings' are linked by the further involvement of the PdF_6 groups in adjoining 'rings'. There is a striking resemblance of the four member 'ring' in this structure to that previously observed in $\text{XeF}_5^+\text{AsF}_6^-$ (ref. 6).

It should be noted that the three F-ligands of the PdF_6 groups which make close approach to the Xe atom do so more or less symmetrically with respect to the pseudo four-fold axis of each XeF_5 group. Thus the $\text{F}_{\text{axial}}\text{-Xe}\dots\text{F}$ (close contact) angles all lie within the range 135-146°. The manner in which the XeF_5 and PdF_6 groups are arranged in the crystal is illustrated in the stereogram, which is Figure 2c.

DISCUSSION

Structure Analysis.- The analysis was hampered by the fact that the structure deviates only a little from space group Pcan (No. 60, with an unconventional setting)⁸. The coordinates of Xe and F listed in Table I can be grouped as pairs related by the transformation x,

-7-

$1/2 - y$, $1/2 - z$ with discrepancies no greater than 0.046 for F and 0.023 for Xe, while Pd would be related to itself by this operation if its y were increased by 0.008. If this correspondence were exact, the symmetry would be P_{can}, a centric space group. This pseudosymmetry escaped notice at first because there are clear violations of the extinction rule for the n glide. The structure consisting of Xe(2) atoms is nearly the inverse of that of the Xe(1) atoms, and thus has the same Patterson vectors. As a result, it was easy to find vectors for two sets of four heavy atoms per unit cell in the Patterson maps, but a third set could not be found. If one tries to interpret these vectors in space group P_{cam} (the centric group consistent with the extinction rules) either set of four atoms can be placed on the mirror planes, but then the other must be in a general set of positions. This interpretation was ruled out by the absence of vectors for such atoms related by the mirror. Had the similarity to space group P_{can} been noticed, it would have been easy to assign four Pd atoms to a special position (on the two-fold axis) and eight Xe atoms to a general position.

The next complication is that Pd hardly contributes to the phasing of any reflection with $h + k$ odd, nor Xe to any reflection with $l = 0$ and $h + k$ odd. For other reflections the heavy atoms give good phasing for the centric pseudostructure, but the deviations from this symmetry will be controlled by the accident of the slight displacements of the trial structure. Thus the heavy atom positions are not a very helpful guide to the correct positions of the F atoms, and false minima with relative good agreement indices are possible. A similar situation

was encountered in the refinement of the structure of $\text{Na}_4\text{XeO}_6 \cdot 6\text{H}_2\text{O}$ (ref. 16).

A third difficulty is the question of absolute polarity. The structure is too nearly centric for there to be any dramatic differences in the Bijvoet pairs, and the differences in agreement indices are not very decisive. It is even more difficult to exclude the possibility of two orientations mixed by twinning. Had the measurements been confined to one octant, as is often the custom, the polar dispersion effect^{17,18} would have caused a serious uncertainty. With full data that effect disappears, and nearly the same results are obtained with either assumption of polarity.

Chemical Aspects.- Hexafluoropalladates(IV) have long been known¹⁸ and a regular octahedral PdF_6^{2-} geometry was indicated by the isomorphism of the alkali fluoropalladates with the fluoroplatinates, of which the crystal structure of K_2PtF_6 was known from X-ray single crystal structure analysis¹⁹. Hitherto, however, the only structure determination reported for a PdF_6^{2-} salt was that of Bartlett and Quail²⁰, derived for K_2PdF_6 from X-ray powder data. Although the precision of their structure is uncertain, the quoted Pd-F bond length of 1.86 Å is roughly compatible with the Pd-F bond lengths observed in the $[\text{XeF}_5]_2\text{PdF}_6$ structure (see Figure 1 and Table III). It is not known to what extent the unsymmetrical strongly polarizing cations of the xenon salt perturb the anion from ideality. A bond length of 1.86 Å in the alkali salts is quite plausible and a value between 1.86 and 1.90 Å seems certain.

The structure of $[\text{XeF}_5]_2\text{PdF}_6$ is of most interest at this time for the geometry of the cation and the cation coordination. The cation has

previously been established by X-ray single crystal analysis in the salts $XeF_5^+MF_6^-$ [M = Pt and Ru (ref. 5), As (ref. 6)] and crystalline XeF_6 itself has been formulated²¹ as $XeF_5^+F^-$. Table IV gives the bond lengths and angles for the various structures examined so far. It can

(TABLE IV here)

be seen that within the quoted standard deviations, there are barely any significant differences between XeF_5^+ species in different lattices, including XeF_5^+ in crystalline XeF_6 . As has already been pointed out above, the two crystallographically distinguishable XeF_5^+ species in $[XeF_5]_2PdF_6$ are not significantly different. As may be seen from Table IV, the precision of the XeF_5^+ description from this structure is quite high. The common geometry of XeF_5 , in all of the quoted structures, itself suggests a discrete cation, but as has been pointed out previously^{5,22} the close similarity to the neutral molecule IF_5 provides compelling evidence. Indeed as we have discussed in an earlier paper⁹ the strictly isoelectronic relatives of XeF_5^+ are remarkably similar in shape to it. Table V presents the latest data. This highlights, even more dramatically than before, the constancy of the bond angle $F_{axial}-E-F_{equatorial}$.⁹

(TABLE V here)

The coordination behavior of the XeF_5^+ species not only supports the discrete nature of the species but also provides evidence of steric activity of the Xe(VI) non-bonding valence-electron pair. Figures 1 and 2 show that each XeF_5 species in $[XeF_5]_2PdF_6$ is coordinated to three F ligands of two PdF_6 groups, such that the three F ligands lie

approximately on a conical surface, the axis of which is coincident with the symmetry axis of the XeF_5^+ . But if we describe XeF_5^+ as a pseudo-octahedral species in which the nonbonding valence-electron pair occupies the Xe-coordination site trans to the axial bond, the position of the F ligands "on the conical surface" is seen to be appropriate, since then the Xe-valence 'pair' is avoided, and the positive charge of the cation (which we can anticipate will be centered essentially at the xenon atom) is least shielded. This model does not, of course, allow us to predict that three F ligands would coordinate to each XeF_5^+ and indeed in $\text{XeF}_5^+ \text{PtF}_6^-$ and $\text{XeF}_5^+ \text{RuF}_6^-$ (ref. 5) the cation coordination in anion F-ligands is four - not three.

It is not yet clear why certain anions (like PtF_6^- and RuF_6^-) provide four F ligands to coordinate to XeF_5^+ , whereas PdF_6^{2-} and AsF_6^- (ref. 6) provide three. The similarity of the XeF_5^+ coordination in the PdF_6^{2-} and AsF_6^- salts is striking. As a consequence, essentially the same ring (shown for $[\text{XeF}_5]_2 \text{PdF}_6$ in Figure 2) of two XeF_5 groups linked to two MF_6 groups occurs in each structure. It is of relevance to the hypothesis of steric activity of the non-bonding Xe(VI) valence electron 'pairs' that the XeF_5 groups do not share a common axis in the ring but avoid one another (see the stereogram, Figure 2b).

It appears probable that the $2\text{XeF}_5 \cdot \text{MF}_4$ (M = Ge, Sn) complexes reported³ by Pullen and Cady, will prove to be structurally related to $\text{XeF}_5^+ \text{PdF}_6^{2-}$. The ability of XeF_6 to donate F^- to a tetrafluoride to form $\text{XeF}_5^+ \text{PdF}_6^{2-}$ indicates that it is a moderately good base. It

-11-

remains, however, to be seen if $\text{XeF}_5^+ \text{MF}_6^{3-}$ salts can be derived from the metal trifluorides.

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14. Table II will appear following these pages in the microfilm edition of this volume of the journal. Single copies may be obtained from the Business Operations Office, Books and Journals Division, American Chemical Society, 1155 Sixteenth Street, N.W., Washington D. C. 20036. Remit check or money order for \$....., for photocopy or \$2.00 for microfiche, referring to code number INORG -00-0000.
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TABLE I

Positional and Thermal Parameters for $[\text{XeF}_5^+]_2\text{PdF}_6^{2-}$

With Estimated Standard Deviations in Parentheses.

ATOM	X	Y	Z	B11	B22	B33	B12	B13	B23
PD	.17525(7)	.24185(6)	.250°	2.70(3)	2.53(3)	1.13(2)	-.25(3)	-.02(3)	.22(3)
XE(1)	.53535(7)	.12057(5)	.0332(1)	3.11(3)	3.24(3)	1.75(3)	.75(2)	-.10(2)	.29(3)
XE(2)	.51278(7)	.38670(6)	.4738(1)	2.85(3)	3.20(3)	1.82(3)	-.58(3)	-.20(2)	.49(3)
F(1)	.0394(6)	.2865(5)	.3880(8)	4.0(3)	3.1(3)	1.3(3)	.9(2)	.5(2)	-.2(3)
F(2)	.0268(6)	.2145(5)	.1235(8)	3.8(3)	2.5(3)	1.5(3)	-.1(2)	-.5(2)	-.0(2)
F(3)	.3261(7)	.2720(6)	.3796(8)	3.8(3)	5.4(4)	1.7(3)	-1.7(3)	-.9(2)	1.1(3)
F(4)	.3101(6)	.1917(6)	.1150(8)	3.5(3)	6.8(4)	1.8(3)	1.7(3)	.9(2)	1.0(3)
F(5)	.1555(7)	.1085(5)	.3352(7)	4.1(3)	2.4(3)	2.0(3)	.1(2)	.4(2)	.7(2)
F(6)	.1809(7)	.3767(5)	.1659(8)	4.6(3)	2.6(3)	2.4(3)	-.8(2)	-.2(2)	.9(2)
F(7)	.4920(9)	-.0171(5)	-.0011(1)	8.7(4)	2.2(3)	4.9(5)	.6(3)	-1.6(3)	.8(3)
F(8)	.7048(8)	.0634(7)	.0830(9)	4.7(4)	8.6(5)	5.0(5)	3.6(4)	-1.1(3)	.3(4)
F(9)	.6348(8)	.1241(6)	-.1368(8)	4.2(3)	5.8(4)	2.9(3)	1.7(3)	1.7(3)	.4(3)
F(10)	.6289(9)	.2376(6)	.0990(8)	5.5(4)	6.5(4)	2.7(4)	-1.5(3)	-1.7(3)	-.5(3)
F(11)	.5049(9)	.0869(8)	.2209(9)	7.3(5)	8.9(5)	1.9(4)	3.5(4)	.7(3)	2.6(4)
F(12)	.6658(8)	.4658(6)	.4212(9)	4.9(4)	6.8(5)	4.9(5)	-2.6(3)	-.1(3)	1.7(4)
F(13)	.4460(8)	.5180(5)	.519(1)	6.5(4)	2.9(3)	5.3(5)	-.1(3)	.2(4)	-.9(3)
F(14)	.6301(8)	.2836(7)	.4013(9)	5.0(4)	6.0(4)	3.8(4)	-.2(4)	1.6(3)	-.9(3)
F(15)	.4669(9)	.4216(7)	.288(1)	8.2(5)	7.1(5)	2.6(4)	-4.0(4)	-1.7(3)	2.6(4)
F(16)	.6188(8)	.3897(6)	.6381(8)	4.2(3)	4.4(3)	2.4(3)	-.7(3)	-1.4(2)	-.1(3)

* Fixed Parameter.

TABLE III

Interatomic Distances (Å) and Angles (Deg.) Within the $[\text{XeF}_5^+]_2\text{PdF}_6^{2-}$
Asymmetric Structural Unit

Distances		Angles			
Xe(1)-F(8)	1.806(7)	F(8)-Xe(1)-F(1)	134.21(42)	F(12)-Xe(2)-F(2)	135.92(39)
F(7)	1.836(7)	F(4)	146.39(44)	F(3)	142.70(45)
F(9)	1.849(7)	F(5)	140.21(40)	F(6)	140.82(42)
F(10)	1.841(7)	F(7)	81.45(40)	F(13)	79.79(39)
F(11)	1.838(8)	F(9)	78.06(35)	F(14)	80.20(42)
F(4)	2.418(6)	F(10)	79.93(40)	F(15)	77.98(39)
F(5)	2.582(6)	F(11)	78.02(38)	F(16)	78.02(37)
F(1)	2.617(7)				
		F(7)-Xe(1)-F(9)	88.93(52)	F(13)-Xe(2)-F(15)	85.40(46)
Xe(2)-F(12)	1.820(7)	F(11)	84.93(48)	F(16)	88.25(49)
F(13)	1.842(7)				
F(14)	1.845(8)	F(10)-Xe(1)-F(9)	91.82(41)	F(14)-Xe(2)-F(15)	87.80(49)
F(15)	1.855(8)	F(11)	86.63(48)	F(16)	90.28(41)
F(16)	1.835(7)				
F(3)	2.445(7)	F(1)-Pd-F(2)	89.66(33)	F(3)-Pd-F(4)	90.23(36)
F(6)	2.559(7)	F(3)	89.85(37)	F(5)	89.10(35)
F(2)	2.639(7)	F(5)	85.25(34)	F(6)	93.53(35)
		F(6)	91.70(34)		
Pd-F(1)	1.902(6)			F(4)-Pd-F(5)	92.45(37)
F(2)	1.860(6)	F(2)-Pd-F(4)	90.28(36)	F(6)	90.59(36)
F(3)	1.902(7)	F(5)	91.59(32)		
F(4)	1.900(7)	F(6)	85.75(35)		
F(5)	1.893(6)				
F(6)	1.898(6)				

TABLE IV
The XeF_5^+ Ion

	Xe-F_{ax} ‡	Average ‡ XeF_{eq}	Average ‡ $\text{F}_{\text{ax}}-\text{Xe}-\text{F}_{\text{eq}}$	
XeF_6	(t)* 1.84(4) Å	1.86(3)	77.2(18)°	(a)
	(h) 1.76(3)	1.92(2)	80.0(6)	
$\text{XeF}_5^+ \text{AsF}_6^-$	1.76(2)	1.82(3)	80.4(15)	(b)
$\text{XeF}_5^+ \text{RuF}_6^-$	1.79(1)	1.85(1)	79.0(6)	(c)
$\text{XeF}_5^+ \text{PtF}_6^-$	1.81(8)	1.88(8)	79.5(40)	(d)
$[\text{XeF}_5^+]_2 \text{PdF}_6^{2-}$	1.81(1)	1.84(1)	79.2(4)	present work

* (t) indicates tetramer and (h) hexamer. ‡ number in parentheses are the estimated standard deviations for the least significant digit.

(a) Reference 21; (b) Reference 6; (c) Reference 5(b);

(d) Reference 5(a).

TABLE V

Distances (Å) and $F_{ax}-E-F_{eq}$ Angle (Deg.) for the Isoelectronic Species

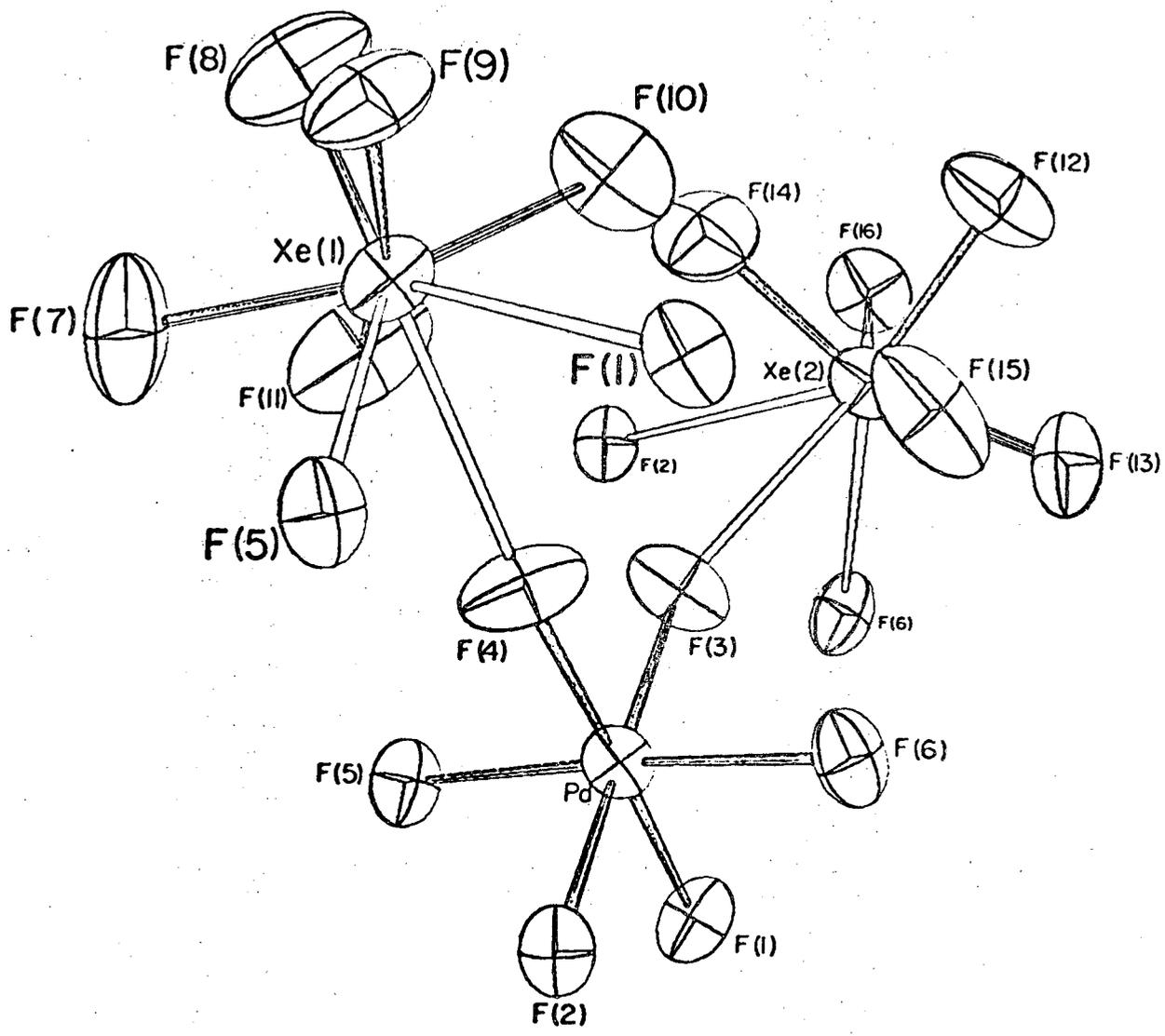
EF_5 (E = Sb, Te, I, Xe), Averaged to C_{4v} symmetry for Comparison

	SbF_5^{2-} ^a	TeF_5^- ^b	IF_5 ^c	XeF_5^+
M-F _{ax}	1.916(4)	1.862(4)	1.817(10)	1.813(7)
M-F _{eq}	2.075(3)	1.952(4)	1.873(5)	1.843(8)
$F_{ax}-E-F_{eq}$	97.4(1)	78.8(2)	80.9(2)	79.2(4)

a R. R. Ryan and D. T. Cromer, Inorg. Chem. 11, 2322 (1972).

b S. H. Mastin, R. R. Ryan and L. B. Asprey, ibid. 9, 2100 (1970).

c G. R. Jones, R. D. Burbank and N. Bartlett, ibid. 9, 2264 (1970).



XBL 724-6188

Figure 1(a): The formula unit in $[\text{XeF}_5^+]_2\text{PdF}_6^{2-}$

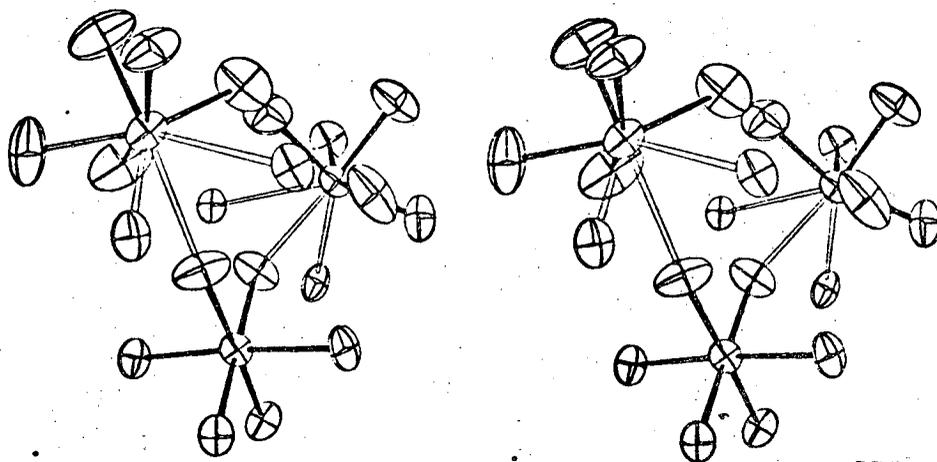
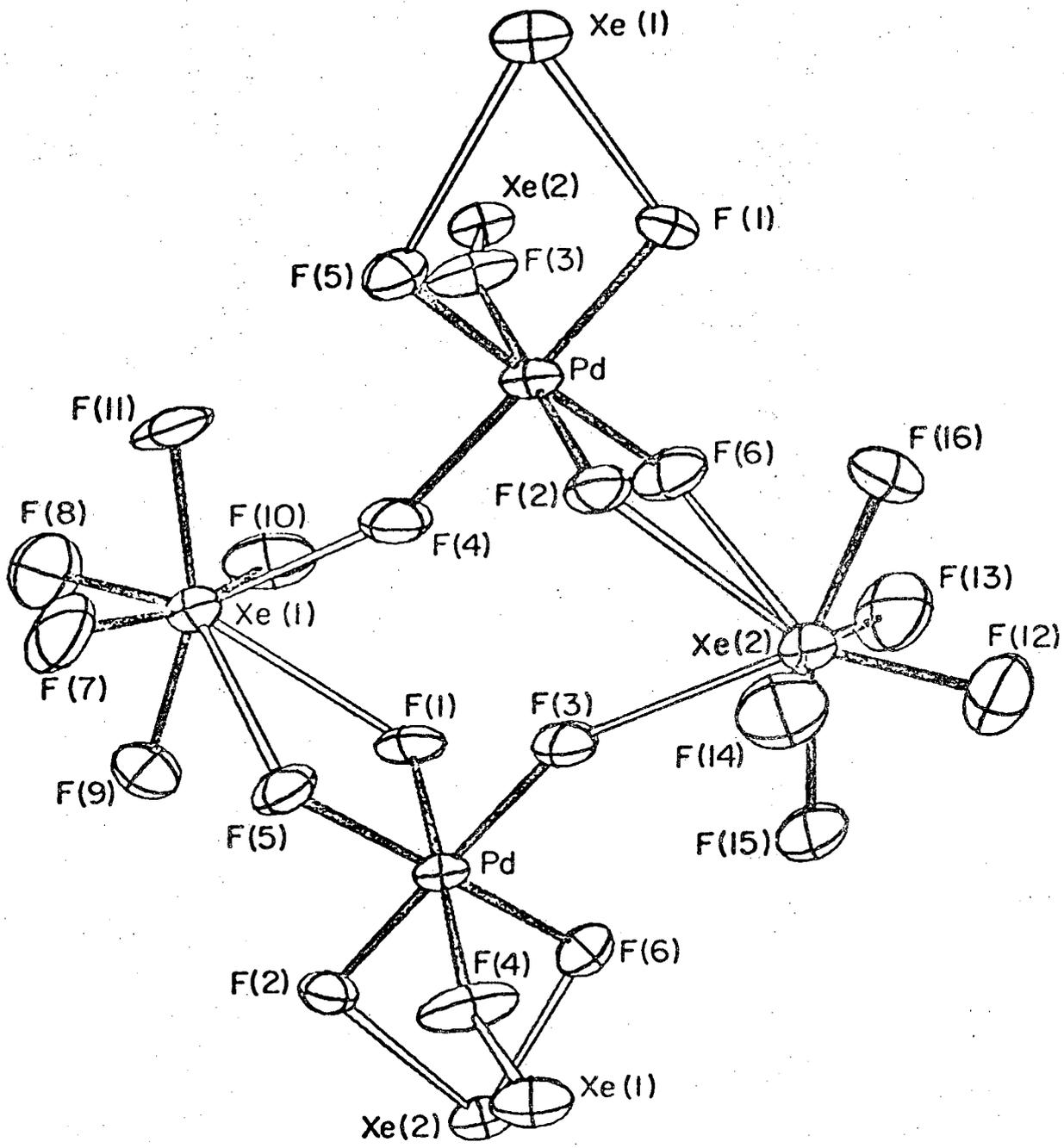


Figure 1(b): Stereogram of the formula unit in $[\text{XeF}_5^+]_2\text{PdF}_6^{2-}$



XBL 725-6264

Figure 2(a): The $XeF_5^+AsF_6^-$ -like rings in $[XeF_5^+]_2PdF_6^{2-}$

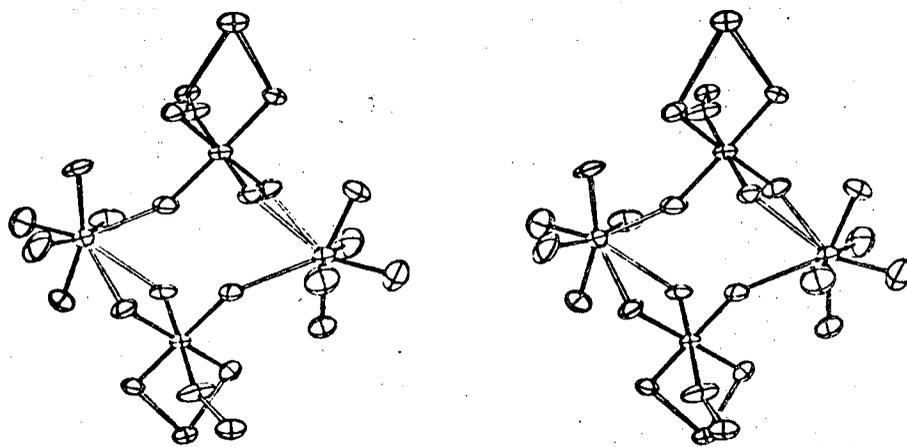


Figure 2(b): Stereogram of the $\text{XeF}_5^+\text{AsF}_6^-$ -like rings in $[\text{XeF}_5^+]_2\text{PdF}_6^{2-}$

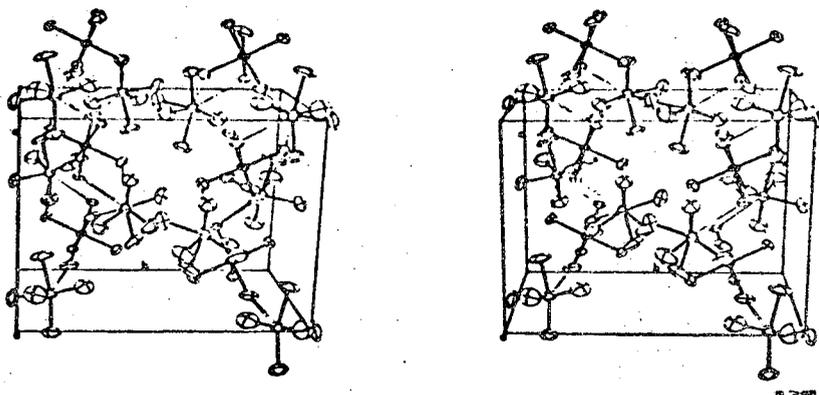


Figure 2(c): Stereogram of the $[\text{XeF}_5^+]_2[\text{PdF}_6]^{2-}$ unit cell, showing the packing of linked $\text{XeF}_5^+\text{AsF}_6^-$ -like rings.

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