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THE CRYSTAL STRUCTURE OF $[XeF^+][AsF_6^-]$[1]

Allan Zalkin, Donald L. Ward, Richard N. Biagioni
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ABSTRACT

Crystals of $XeF^+AsF_6^-$ are monoclinic, space group $P2_1/n$, a = 6.308(3), b = 6.275(3), c = 16.023(5) Å, β = 99.97(5)°, V = 624.66 Å^3, Z = 4, D_x = 3.61 g cm^{-3}, at 24°C. X-ray diffraction data were measured with counter methods and MoKα radiation. With anisotropic temperature factors for all atoms, R = 0.033 for 777 independent reflections (I > 3σ). The structure consists of ion pairs connected by a bridging fluorine atom into $FXeFAsF_5$ units. The FXeF portion of the molecule is linear (angle = 178.9(7)°) with Xe-F = 1.873(6) Å (terminal) and 2.212(5) Å (bridging). The $FAsF_5$ portion of the molecule is roughly octahedral with cis F-As-F angles ranging from 85.3(3)° to 94.6(5)°; the 5 terminal As-F distances range from 1.676(5) Å to 1.690(8) Å and the bridging As-F distance is 1.813(6) Å. The bridge Xe-F-As angle is 134.8(2)°.

INTRODUCTION

In an earlier paper Sladky et al. described[2] the preparation of the 1:1 $XeF_2 - AsF_5$ complex and showed that it lost AsF_5 readily under vacuum at ordinary temperatures to yield a 2:1 complex. The crystal structure of the 2:1 complex has already been described[3] and is consistent with formulation as the salt $Xe_2F_3^+AsF_6^-$. The structure of the 1:1 complex was of interest to us because of an earlier finding[4] that the structure of the 1:1 $XeF_2 - RuF_5$ complex was consistent with the $XeF^+RuF_6^-$ formulation and our expectation that the formulation of the 1:1 $XeF_2 - AsF_5$ complex would be similar. Moreover, a preliminary study of 1:1 $XeF_2 - AsF_5$ by others[5] had failed to find evidence of AsF_6^- and the authors concluded that this indicated that the complex was a molecular, possibly fluorine-bridged, adduct.

We give here the preparative conditions for 1:1 $XeF_2 - AsF_5$ complex, and the structure determined by single crystal x-ray diffraction.

EXPERIMENTAL SECTION

Preparation of $XeFAsF_6$. XeF_2 (0.684 g, 4.04 mmoles) prepared as described by Williamson,[6] was transferred in the dry atmosphere of a Vacuum Atmospheres Corporation Drilab, to a weighed quartz bulb (~ 60 cc capacity) joined to a Brass Whitey valve (IKS4) with a Teflon-gasketed Swagelock fitting. Bromine pentafluoride (Matheson Co., East Rutherford) sufficient (~ 2 ml) to dissolve the XeF_2 was transferred to the bulb under vacuum. The resultant, almost colorless solution of XeF_2 in BrF_5 was exposed to arsenic pentafluoride gas (Ozark Mahoning, Tulsa, Okla.) to maintain a pressure of ~ 900 torr. A small Teflon-coated stirrer bar driven by an external magnet kept the solution mixed. The solution rapidly became pale yellow-green but the system was left for ~ 1 hr. to ensure full take up of AsF_5 gas. The solution was cooled to -22.8° (CCl_4 slush) and the bulk of the BrF_5 was removed in a dynamic vacuum. (At the beginning of this evaporation the valve was opened slowly to ensure smooth boiling and to avoid frothing). The crystalline solid so obtained was dried by intermittent opening and closing of the valve, for a few minutes, with the sample at room temperature. The solid remaining in the trap was almost white, with a pale yellow-green tint. It amounted to 1.372 g, indicating an AsF_5 uptake of 0.688 g (4.05 mmoles) which corresponds to a XeF_2:AsF_5 molar ratio of 1:1.002.

X-ray powder photographs were obtained by rapidly transferring the solid to a 0.5 mm quartz X-ray capillary in the Drilab. The capillary was sealed with a small flame. The data given in Table I were indexed using the single crystal information.

Single crystals of $XeFAsF_6$ were grown by sublimation of small samples of the powder sealed, as in the X-ray powder sample case, in thin-walled quartz X-ray capillaries. These capillaries were placed in an electrically heated tube which provided for a temperature gradient along the capillary of 1 or 2° at a temperature of ~ 70°. The capillaries were left in this heater overnight and were then inspected for single crystal development using a polarizing microscope.

Raman spectra were obtained from a sample packed as for the X-ray powder sample, but in a 1.5 mm diam. quartz capillary. The sample was cooled by a cold nitrogen stream shrouded by a dry room temperature stream and the temperature was thereby maintained at ~ -100°. The spectrum of $Xe_2F_3AsF_6$ was obtained in the same way. The spectra are compared in Fig. 1.

X-ray Measurements. The crystal selected for diffraction measurements was 0.09 x 0.11 x 0.15 mm in size. Precession photographs established the lattice to be monoclinic. Absent reflections h01 ($h + 1 \neq 2n$) and 0k0 ($k \neq 2n$) indicate space group $P2_1/n$. Setting angles for the Picker FACS-I diffractometer for 12 reflections above $2\theta = 45°$ (λ 0.70926 Å for $MoK\alpha_1$) were used for least-squares adjustment of the cell dimensions. Intensities were measured with graphite-monochromatized MoKα radiation and θ-2θ technique for all reflections in the half-sphere with k non-negative and $2\theta<55°$. After averaging equivalent pairs there were 1447 unique ones of which 783 had $I > 3\sigma$. Measurements were made with a scan rate of 1°/min, backgrounds counted for 10 sec each at an offset of 1° from each end of the scan, and a scan length of 1.5° in 2θ plus the $\alpha_1 - \alpha_2$ divergence. Three standard reflections showed slight changes during the experiment, and a compensating correction (ranging 1.00 to 1.10) was applied to the intensities. Correction for absorption[7] ($\mu = 103.4\ cm^{-1}$) was made by analytical integration with the crystal shape described by eight faces. Effects of extinction were evident in the data, and an empirical isotropic correction was applied which increased the structure factors by 84% and 36% for the strongest and seventh-strongest reflections, respectively. The six strongest reflections were assigned zero weight because they failed to give good agreement even with this correction. A term $(0.03I)^2$ was added to the variance of I derived from counting statistics. Zero weight was also assigned to reflections with $I < 3\sigma$. Atomic scattering factors of Doyle and Turner[8] with anomalous scattering corrections of Cromer and Liberman[9] were used for neutral Xe, As, and F.

Crystal Data. $XeFAsF_6$, monoclinic, $P2_1/n$, $a = 6.308(3)$, $b = 6.275(3)$, $c = 16.023(5)$ Å, $\beta = 99.97(5)°$, $V = 624.66$ Å^3, $Z = 4$, $D_x = 3.61$ g cm^{-3}, at 24°C.

Determination of Structure. Analysis of a Patterson map indicated two sets of heavy atoms in general positions, a result incompatible with the preconceived opinion of the composition of the material.[10] For this reason the subsequent analysis was carried out using the diffraction data to establish the composition. The peaks for the heavy atoms were of appropriate relative height to correspond to Xe and As, and other peaks were found which corresponded to six F atoms around the As atom. Fourier maps phased with these eight atoms revealed the seventh fluorine atom. Another smaller peak was tested as a possible fluorine atom, but it was rejected by the least-squares refinement. A later electron density map, prior to the absorption correction and with $F = \Sigma|\Delta F|/\Sigma F_o = 0.10$, showed no extra peaks as high as one-third the lowest fluorine atom. The final refinement by full-matrix least-squares reduced $R_w = [\Sigma w(\Delta F)^2/\Sigma w F_o^2]^{1/2}$, the quantity minimized, to

0.036. The final R was 0.033 for 777 reflections and 0.076 for 1447 reflections including those of zero weight. In the last cycle no parameter shifted more than 0.01σ. The goodness-of-fit was 1.13. Final parameters are listed in Table II.

DISCUSSION

The crystal structure, Fig. 2(a) consists of an ordered arrangement of discrete $FXeFAsF_5$ units (Fig. 3), the closest contact between units being 2.92 Å, a contact between F(1) and F(7). Bond distances and angles of this unit are listed in Tables III and IV. The amplitudes of thermal motion are considerable, and distances corrected for this motion according to the riding model are also listed in Table III. Table V gives the angles between principal axes of thermal motion of fluorine atoms and their bond vectors. In each case except Xe-F(6) the minimum amplitude of motion is nearly parallel to the bond, and in every case the largest amplitude is nearly perpendicular to the bond. These results lend credibility to the physical reality of this model of the thermal motion and support the propriety of the riding model. It may be noted that the consistency of As-F bond lengths (omitting the bridging bond which is not expected to be the same) is improved by the thermal correction.

The $FXeFAsF_5$ unit is remarkably similar in shape to the analogous unit in $FXeFRuF_5$[4] with which it is compared in Fig. 4. In both cases the bridging fluorine is more equally shared between Xe and As(Ru) than in the corresponding $[XeF_5^+][MF_6^-]$ compounds.[3,4]

In spite of the close similarity of molecular geometry and the fact that the arsenic and ruthenium compounds crystallize with the same space group symmetry, the unit cell shapes are dissimilar and, moreover, the molecular packing is quite different. In $FXeFAsF_5$ the long dimensions of the formula units are all nearly parallel to each other, while in $FXFRuF_5$ there are two orientations nearly perpendicular to each other. The molecular volumes differ by less than 3%, reflecting the slightly larger size of Ru compared with As, and there is little difference in the shortest intermolecular distances.

Inspection of Fig. 2(b) reveals that in $FXeFRuF_5$ each XeF species is surrounded by a roughly cubic arrangement of RuF_6 species; the Xe...Ru distances are: 3.82, 4.47, 4.54, 4.82, 4.88, 4.91, 4.99. 5.66 Å. In $FXeFAsF_5$ (Fig. 2(a)) each XeF species has six AsF_6 species at Xe...As distances of 3.72, 4.38, 4.44, 4.46, 4.52, and 4.80 Å. This arrangement can be viewed as a grossly distorted octahedral coordination of the cation. The next nearest arsenic neighbor of the Xe atom is at 5.84 Å. Thus to a first approximation we can represent the $FXeFRuF_5$ as developed from a CsCl (8:8 coordination) structure type whereas we can represent $FXeFAsF_5$ as derived from a NaCl type (6:6 coordination). Such representations conform with the common structure types for hexafluoroarsenates and hexafluororuthenates.[11] Thus $NO^+[AsF_6]^-$ is of NaCl type[12] whereas $NO^+[RuF_6]^-$ is of CsCl type.[13] Moreover, although the XeF_5^+ species is essentially the same in $XeF_5^+AsF_6^-$ (ref 3) and $XeF_5^+RuF_6^-$ (ref 4) the coordination is different. The coordination number differences are not to be associated with the non-transition-element transition-element content however. Thus $NO^+[SbF_6]^-$ is of CsCl type[13] like $NO^+[RuF_6]^-$, and $XeF_5^+AuF_6^-$ has the same structure[14] as $XeF_5^+AsF_6^-$. Indeed X-ray powder photographs[15] indicate that $FXeFSbF_5$ is probably isostructural with $FXeFRuF_5$.

It is probable that the difference in coordinating capability of the anions is associated with differences in polarizability of the F ligands in the MF_6^-. In those hexafluorometallates which can be unambiguously described as salts $A^+MF_6^-$, the small less polarizable (harder) cations such as Li^+ and Na^+ generally prefer a NaCl type lattice, whereas the more polarizable (softer) cation, e.g., Cs^+, prefer the CsCl type. The data for selected AsF_6^-, AuF_6^-, RuF_6^- and SbF_6^- salts,[11,14,16-20] Table VI, shows the pattern. The data indicates that the effective molecular volume of the anions increases in the sequence $AsF_6^- < AuF_6^- < RuF_6^- < SbF_6^-$. One also notes that even with a large cation such as IF_6^+, the AsF_6^- and AuF_6^- salts adopt the $NaCl^-$ type lattice - presumably because the F ligands of that cation are not very polarizable (i.e., are hard). However, even with this cation, one sees that the CsCl lattice becomes the preferred one with the larger (and hence more polarizable) SbF_6^- anion.

The similarity of the $FXeFAsF_5$ and $FXeFRuF_5$ units (Fig. 4) suggests that the bonding of the xenon is essentially the same for both. The bonding in XeF_2, following Coulson,[21] can be conveniently represented as a resonance hybrid of the canonical forms $(F\text{-}Xe)^+F^-$ and $F^-(Xe\text{-}F)^+$. This kind of representation permits one to make rough thermodynamic stability estimates.[22] In the $FXeFMF_5$ compounds one can represent the equivalent canonical forms as $[F\text{-}Xe]^+[F\text{-}MF_5]^-$ and $F^-[XeFMF_5]^+$. The components F^-, $[Xe\text{-}F]^+$ and $[F\text{-}AsF_5]^-$ in the listed canonical forms can each be visualized as providing each atom with its 'ideal' complement of eight valence electrons.[23] The component $[XeFMF_5]^+$ cannot provide for this, without writing non-bonded forms such as $[(Xe\text{-}F)^+AsF_5]$ and the unrealistic $[Xe(F\text{-}AsF_5)^+]$. We therefore believe that the canonical form $F^-[XeFMF_5]^+$ is not important. As has been argued previously[4] we prefer to represent the formula units simply as $[FXe]^+[MF_6]^-$ and allow that the short distance between the Xe of the cation and one of the F atoms of the anion is a consequence of the cation having its positive charge centered largely at the Xe atom. This positive charge location is a consequence of the electron density in the Xe-F bond of the cation having depleted the xenon valence shell electron density (in comparison with atomic Xe). Moreover the formation of the bond in the cation must create an electron-density hole on the far side of the Xe atom (i.e., trans to the bond). One therefore anticipates that the $Xe\text{-}F^+$ species should show its highest effective positive charge when viewed along the axis towards the Xe atom. Thus the cation ought to possess a unique axis in its polarizing effects, as observed in these structures. Representation of $FXeFMF_6$ as the salts $XeF^+MF_6^-$, as for the XeF_3^+ and XeF_5^+ salts[24,4] (allowing for the polarizing effects of the cations) predicts the observed disposition of cation to anion. The model is simple, and sufficient.

Supplementary Material Available. A listing of structure factors (6 pages). Ordering information is given on any current masthead page.

ACKNOWLEGEMENTS

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Table I X-ray powder data for $[XeF]^+[AsF_6]^-$ (from a sample in 0.3 mm diam. quartz capillary using Cu K_α radiation).

$hk\ell$	$1/d^2$ calc.	$1/d^2$ obs.	Rel. intensity
002	.0161	.0170	vw
$11\bar{1}$	.0518	.0515	s
111	.0588	.0586	s
004	.0642	.0648	s
?	----	.0706	vvw
112	.0744	.0774	m
$11\bar{3}$	.0768		
014	.0896	.0885	vw
113	.0980	.0988	mw
020	.1016		
200	.1036		
021	.1056	.1080	m
$20\bar{2}$	.1056		
$10\bar{5}$	.1086		
120	.1275	.1241	w
$12\bar{1}$	.1280		
$11\bar{5}$	.1340	.1347	w
121	.1350		
$20\bar{4}$	.1396	.1396	w
006	.1445		
105	.1440		
023	.1377		

CONTD.

Table I (CONTD.)

212	.1592	.1599	w
024	.1658	.1679	w
115	.1693		
204	.1961	.1970	w
$10\bar{7}$	.1979		
220	.2052	.2059	w
$20\bar{6}$	.2058		
124	.2059		
$12\bar{5}$	.2102		
$21\bar{6}$	.2312	.2349	w
$22\bar{4}$	.2412	.2426	w
008	.2570	.2550	mw
$31\bar{1}$	.2520		
$13\bar{1}$	.2549		
131	.2620	.2635	mw
$31\bar{3}$	.2629		
224	.2977	.3019	w
027	.2983		
$31\bar{5}$	.3059	.3077	w
$20\bar{8}$	.3041		
133	.3012		
$11\bar{9}$	.3447	.3484	vw
119	.4083	.4090	w
208	.4171		
400	.4145		
040	.4062		
00, 10	.4015		

Table II Coordinates and Thermal Parameters for $[XeF^+][AsF_6^-]^a$

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Xe	.25918(9)	.21684(12)	.62266(3)	3.94(3)	4.32(3)	3.51(2)	-.46(3)	.67(2)	.07(2)
As	.23792(13)	.27638(18)	.39041(5)	3.32(4)	3.83(5)	3.56(3)	-.19(4)	.46(3)	-.21(3)
F(1)	.4950(8)	.2815(14)	.3763(4)	4.4(3)	9.4(5)	8.1(4)	-.6(3)	2.4(3)	.0(3)
F(2)	.2308(13)	.5447(14)	.3816(5)	8.6(5)	4.8(4)	11.8(6)	.5(4)	2.6(4)	1.1(3)
F(3)	.1413(12)	.2335(16)	.2889(4)	8.9(4)	15.2(8)	3.9(3)	-3.3(5)	.1(3)	-1.8(4)
F(4)	-.0092(9)	.2721(13)	.4177(5)	4.0(3)	8.7(5)	8.7(4)	-.2(3)	2.1(3)	-.2(3)
F(5)	.2512(14)	.0141(13)	.4127(6)	9.1(5)	4.1(4)	13.9(6)	.2(4)	1.6(4)	.9(4)
F(6)	.3466(12)	.3220(15)	.5013(3)	7.1(3)	13.1(6)	3.8(2)	-5.4(5)	.1(2)	.5(2)
F(7)	.1843(13)	.1334(19)	.7260(4)	11.2(6)	15.5(7)	4.8(3)	-5.2(5)	3.0(3)	1.3(4)

[a] Estimated standard deviation of the least significant digit(s) is given in parentheses here and in the following tables. The form of the temperature factor is $\exp[-0.25(h^2 a^{*2} B_{11} + 2hka^* b^* B_{12} + \ldots)]$. For space group $P2_1/n$ (an alternate setting of $P2_1/c$) the general positions are: x, y, z; -x, -y, -z; 1/2 + x, 1/2 - y, 1/2 + z; 1/2 - x, 1/2 + y, 1/2 - z.

Table III Interatomic Distances in $[XeF^+][AsF_6^-]$, Å

Xe-F(6)	2.212(5)	[2.25][a]	As-F(3)	1.657(6)	[1.72]
Xe-F(7)	1.873(6)	[1.94]	As-F(4)	1.690(5)	[1.73]
As-F(1)	1.676(5)	[1.72]	As-F(5)	1.683(8)	[1.74]
As-F(2)	1.690(8)	[1.74]	As-F(6)	1.813(6)	[1.86]

[a] Values in square brackets are corrected for thermal motion accordingly to the riding model.

Table IV Bond Angles (deg)

F(6)-Xe	-F(7)	178.9(7)	F(2)-As	-F(5)	172.7(6)
Xe	-F(6)-As	134.8(2)		-F(6)	85.7(5)
F(1)-As	-F(2)	89.0(4)	F(3)-As	-F(4)	93.0(4)
	-F(3)	94.1(4)		-F(5)	92.7(5)
	-F(4)	172.9(5)		-F(6)	179.3(7)
	-F(5)	91.9(4)	F(4)-As	-F(5)	86.8(4)
	-F(6)	85.3(3)		-F(6)	87.6(4)
F(2)-As	-F(3)	94.6(5)	F(5)-As	-F(6)	87.1(5)
	-F(4)	91.4(5)			

Table V Angles (deg) between Bond Vectors and Principal Axes of the Thermal Ellipsoids, and Root-Mean-Square Displacements (Å)

Atom	Vector	Angles			Rms Displacements		
		1	2	3	1	2	3
F(1)	F(1)-As	7	85	85	.215	.325	.346
F(2)	F(2)-As	5	87	86	.242	.323	.390
F(3)	F(3)-As	2	90	88	.209	.324	.458
F(4)	F(4)-As	8	84	85	.211	.329	.336
F(5)	F(5)-As	7	89	83	.225	.340	.423
F(6)	F(6)-As	18	73	85	.215	.230	.458
F(6)	F(6)-Xe	63	27	90	.215	.230	.458
F(7)	F(7)-Xe	4	86	89	.200	.332	.492

Table VI Selected Formula Unit Volumes (Å^3) and Lattice Type (N = NaCl type and C = CsCl type) for Selected $A^+MF_6^-$ Salts

M / A	As	Au	Ru	Sb
Na	116.0, (N_R)[a]	?	120.4, (N_R)[b]	137.0, (N)[b]
IF_6	214, (N)[c]	219, (N)[d]	?	223, (C)[d,e]
Cs	138.4, (C_R)[f]	141.3(C_R)[d]	142.6(C_R)[b]	147.1, (C_R)[g]

? indicates that the structure type is not known. Subscript R indicates a rhombohedral cell, absence indicates cubic.

[a] Ref 16

[b] Ref 11

[c] Ref 17

[d] Ref 14

[e] Ref 18

[f] Ref 19

[g] Ref 20

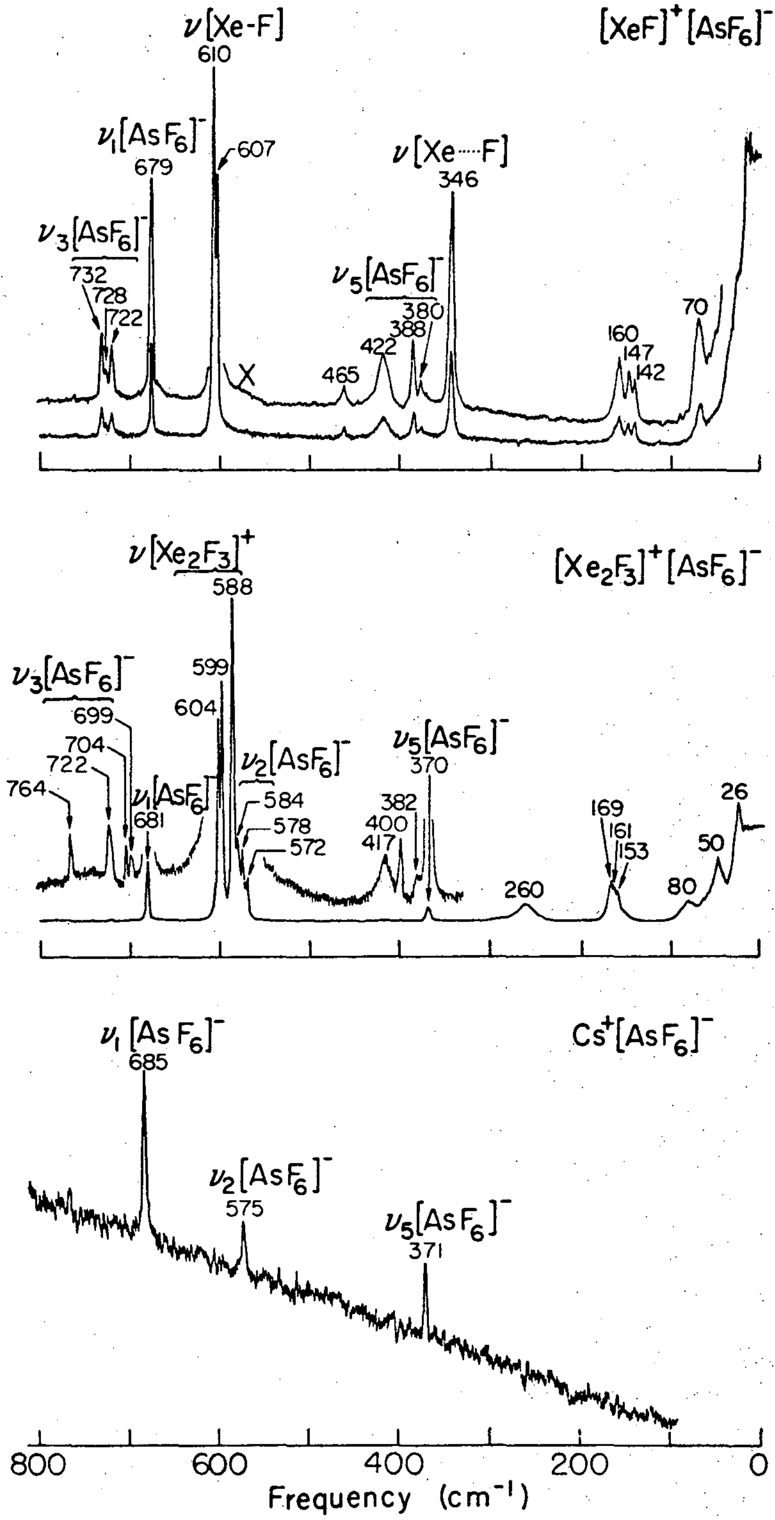
FIGURE CAPTIONS

Fig. 1. The spectra of $XeFAsF_6$ and $Xe_2F_3AsF_6$ are seen to be similar and the counterparts of the $\underline{\nu}_1$ and $\underline{\nu}_5$ modes are seen, from comparison with the $CsAsF_6$ spectrum to be present in both. $Xe_2F_3AsF_6$ consists of well separated ions $Xe_2F_3^+$ and AsF_6^- (Ref. 3) whereas in $XeFAsF_6$ there is a unique interaction of cation and anion. This interaction is probably responsible for the relatively intense band at 346 cm^{-1} in $XeFAsF_6$. Similar assignments, but with a different bonding interpretation have been given by Gillespie and Landa (Inorg. Chem. <u>12</u>, 1385 (1973)); our spectrum of $XeFAsF_6$ shows less $Xe_2F_3AsF_6$ impurity (X) than theirs and fine structure and weak bands previously unrecorded.

Fig. 2. Stereoviews of the crystal structure (a) of $[XeF^+][AsF_6^-]$ and (b) $[XeF^+][RuF_6^-]$, drawn with Johnson's ORTEP.

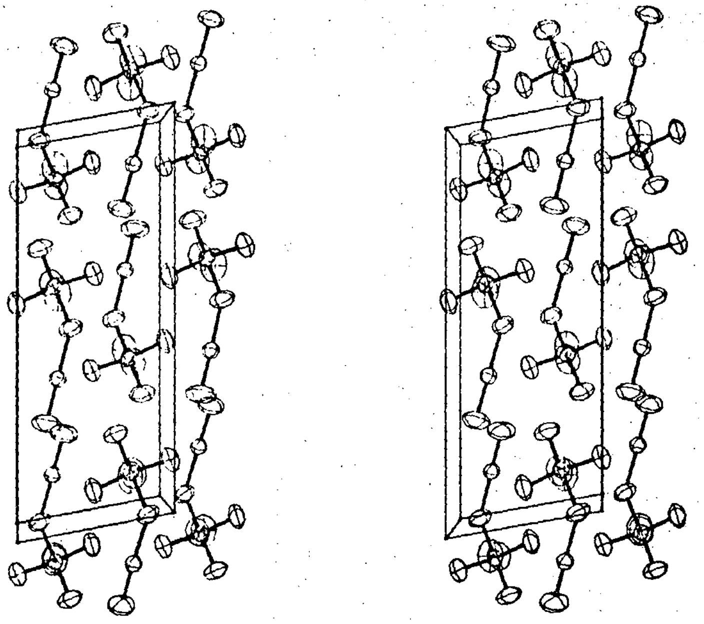
Fig. 3. Stereoview of the molecular unit with 50% probability ellipsoids.

Fig. 4. The formula units of $XeFAsF_6$ and $XeFRuF_6$ (Ref. 4), the former with 50% probability ellipsoids, the latter with 30%.



XBL 778-6003

Fig. 1



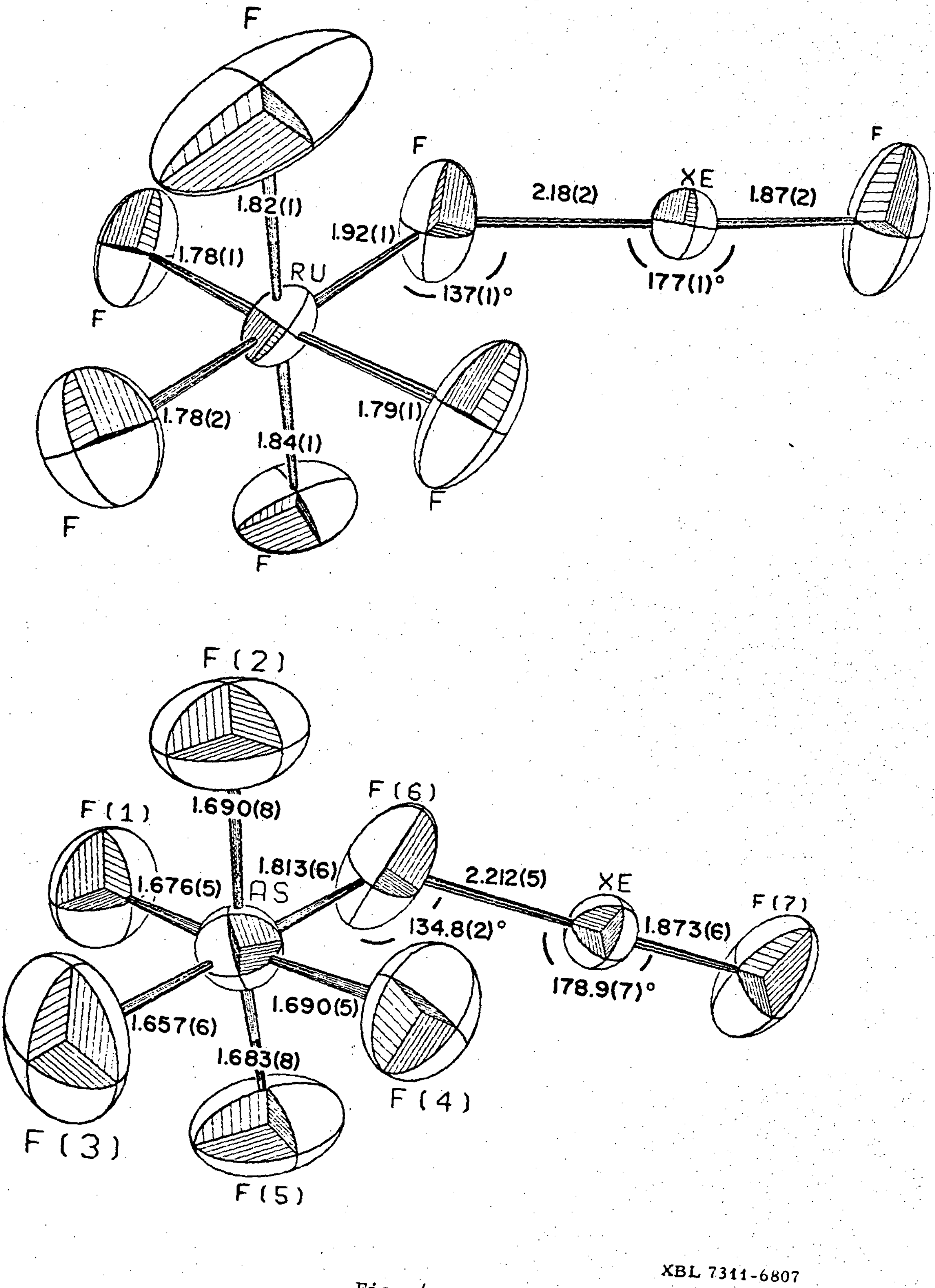
XBL 7311-1427

Fig. 2(a)

```
OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (X 3.0) FOR
 (XEF+)(ASF6-)                                                 F(0,0,0) = 1799

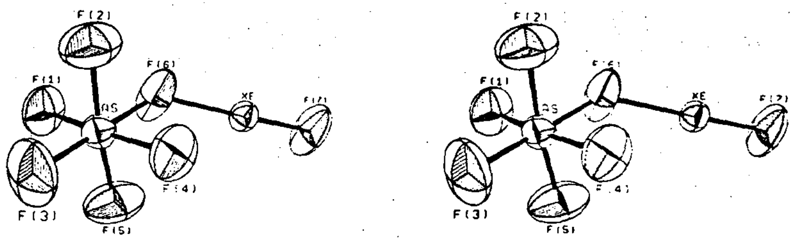
FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS.
SG = ESTIMATED STANDARD DEVIATION OF FOB.  DEL = /FOB/ - /FCA/.
* INDICATES ZERO WEIGHTED DATA.

  L FOB SG DEL   L FOB SG DEL   L FOB SG DEL   L FOB SG DEL   L FOB SG DEL 
   H,K= 0,   0    H,K= 0,   3  14  41  3   5  15  29  9  -7*-12  85  6   5 
  2 112  5   0   1   3 10  -6* 15  52  6   6  17 117  4   1 -11  44 11  -2*
  4 999 32 117*  2  13  6  -3* 16  20  7   2* 19  48  5   6 -10  26  5   2*
  6 312  6  12   3 136  3   1    H,K= 0,   6    H,K= 1,   1  -9 174  5   6 
  8 698 15  64   4  55  2   1   0  77  9  -5 -20   0 26 -12* -8  87  2  -1 
 10 298  5   2   5  30  5   9   1 121  2  -3 -19  22 26  10* -7 202 10   2 
 12 307  5   7   6  96  2   3   2  20  8  14*-18   2118 -21* -6  82  4   4 
 14  89  5 -11   7  77  2   1   3 141  3  -1 -17 141  5  -5  -5 182  9   5 
 16 137  2   3   8 123  3   2   4  77  3  -1 -16  20168  -9* -4 127  6   2 
 18  99  2   1   9  31  3  -3   5  91  2   0 -15  31  4  -5  -3 208  9   8 
 20  55  2   3  10  74  2  -5   6  34  7  10*-14  15 44 -16* -2  78  8  -1 
   H,K= 0,   1  11 106  3   3   7 129  3  -0 -13 249  7  -0  -1 155  6   3 
  1   0 20  -3* 12  54  6  -7   8  52  4  -0 -12   0 22 -18*  0 161  5  -2 
  2  39 12  -8* 13  38  3  -5   9  45  7  -4 -11  97  4  -4   1 171  5   4 
  3  70  2   7  14  88  3  -3  10  19 12  -8*-10 125  5  -3   2  99  5  -6 
  4 120  6  -2  15  43  3  -4  11  94  2  -1  -9 500 20  34   3 194  9   8 
  5   1 26  -6* 16  41  3   9  12  33  8   3* -8  33  4  -3   4 105  3   1 
  6 194  4   6  17  30  5   7  13  31 10  14* -7 237  7   8   5 173  6   0 
  7  63  2   0  18  63  3  -3    H,K= 0,   7  -6 124  6  -2   6   0 12  -4*
  8 159  3  -0  19  41  4   8   1  27 11  19* -5 446 26  27   7  21  6   4*
  9   6 11  -3*   H,K= 0,   4   2  22 10   6* -4   0 38  -3*  8  44  5  -1 
 10 193  4   7   0 391  7 -20   3  28 16   2* -3 443 23   7   9 178  5   3 
 11  43  2  -9   1 247  4  -8   4   8 18   0* -2 176  9   3  10  41  4   5 
 12  96  2  -0   2  55  2   1   5   0 20  -3* -1 632 40-129* 11  30 12  12*
 13  19 23  10*  3 292  8   0   6  18  7   7*  0  38  7  21  12  74  6  -5 
 14 129  3  -2   4 343 10  -1   7  37  3  -1   1 641 67 -24* 13 127  4   4 
 15   0 21 -14*  5 159  4   5   8   8 15  -5*  2 188 10   4  14  13 24 -10*
 16  39  4   6   6 113  3   4   9  12 18   5*  3 517 58   5  15  33  9  -3*
 17   0 16  -7*  7 237  6   0  10  20 16  17*  4  65  9  -6  16  22 30  -7*
 18 106  2  -2   8 199  5  -1    H,K= 0,   8   5 513 35  18  17 101  4   5 
 19   0 48 -16*  9  81  6   1   0   8 23   3*  6 146  5  -3  18   0 29 -10*
 20  19 16  -1* 10  85  5  -9   1  60  3  -0   7 237  6   8  19  31 11   1*
   H,K= 0,   2  11 145  3  -3   2  22 10  19*  8  37 14  -6*   H,K= 1,   3 
  0 672 23-156* 12 115  2   1   3  72  3   2   9 557 20  37 -19  10 19   2*
  1 233  5  -7  13  32  6   4*  4  17 23  14* 10 103  4   4 -18  49  3   5 
  2  39 17  -5* 14  55  4  -5    H,K= 1,   0  11 115  2   1 -17  86  2  -0 
  3 165  4  -2  15  90  3   2 -19  57  7  -6  12   0 17 -13*-16  52  7   1 
  4 538 28 -17  16  46  4  -4 -17  95  2  -1  13 224  4   1 -15  33 10  14*
  5 101  2   0  17  11 16   4*-15  78  5  -3  14  35 12  -7*-14  67  6  -3 
  6 135  3   3    H,K= 0,   5 -13 152  3  -2  15  18 20 -11*-13 185  8   0 
  7 205  5   4   1  48  4   2 -11  47  4  11  16   0 18 -24*-12  60  2  -2 
  8 446 14  14   2  22 14  13* -9 256  5   3  17 128  3  -1 -11  57 11   4*
  9  71  2  -6   3  63  3   2  -7 243  4  20  18  11 23  -6*-10 152  9  -8 
 10 193  3   6   4  46 13  11* -5 176  4   5  19  12 16   5* -9 275 14   5 
 11 114  3  -1   5  32 17  -7* -3 105  2   8    H,K= 1,   2  -8  84  3   1 
 12 231  4  -4   6  25 33 -12* -1  65  2  -1 -20  31  9  -2* -7 155  7  -2 
 13  21 33  -9*  7  57  5  -2   1  85  8  -8 -19  47  4   2  -6 214 10   4 
 14  97  2   1   8  37  6  -9   3  26 43  17*-18   4 20  -1* -5 346 22  -0 
 15  65  3  -2   9  23 10  -0*  5 181  4  -0 -17  95  4   8  -4 107  3   0 
 16 104  3  -3  10  37  6   1   7 120  2  -4 -16  37  3   0  -3 276 12  -5 
 17   6 17   6* 11  59  4  -2   9 249  4   2 -15  67  3  -1  -2 302 17 -15 
 18  69  3  -3  12  26  7  -4* 11  31 10   3*-14   0 27  -3* -1 388 16 -31 
 19  32  6  -7* 13  21 12  -6* 13 154  6  -2 -13 126  3   6   0  24  4   9 
```



XBL 7311-6807

Fig. 4



XBL 7311-1428

Fig. 3

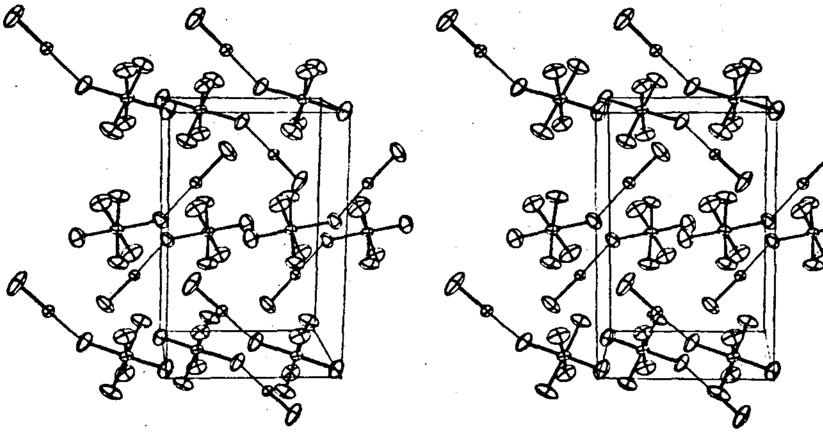


Fig. 2(b)

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STRUCTURE FACTORS CONTINUED FOR
 (XEF+)(ASF6-)                                                    PAGE 2

  L FOB SG DEL   L FOB SG DEL   L FOB SG DEL   L FOB SG DEL   L FOB SG DEL
  1 326  8 -17 -15   0 18  -2* 11  26  8  17*-16  16 19   5*  1 125  5   5
  2 310 15 -12 -14  78  4   5  12  35 20  -6*-15   0 73 -10*  2  65  3   1
  3 306 16  -9 -13  60  7  -6  13  19 23  -2*-14 148  4  -0   3  77  1   1
  4  37  2  -3 -12  61  3  -1    H,K=  1,  7 -13   8 24   0*  4 373 35 -11
  5 369 25  -4 -11  22 14   6*-10  69 10   1 -12 112  3  -1   5 139  8 -11
  6 244  9  -2 -10 134  7  -2  -9  33 15  13*-11  32  4 -12   6  42  4  -5
  7 170  4  -0  -9  88  6  -5  -8  33  8  -8*-10 200  3   1   7 124  5   2
  8  92  3   3  -8  55  4  -1  -7   0 29 -10* -9   5 20 -13*  8 355 26   5
  9 293 12   1  -7  33 28  -6* -6 107  5   3  -8  13  6   1*  9  49  5  -3
 10 142  5   0  -6 202  6   5  -5  27 15   3* -7  17  5  -6* 10  87  8   7
 11  62 11  -4  -5 124  7  -1  -4  32  6  -2* -6 313  6   7  11  76  8  -2
 12  43  4  -4  -4  56 12   8* -3  14 17  -6* -5  50  2  -1  12 192  7  -3
 13 163  6   3  -3  99  5  10  -2 131  5  -2  -4   9 21  -2* 13  36 10  -4*
 14  80  3   0  -2 233  9   2  -1  36 12  12* -3  47  7   9  14  80  5   4
 15  18 31   5* -1 128  4  -2   0  15 18   5* -2 180  4   1  15  57  3  -0
 16  42  5  -1   0  31  9  16*  1  19 26  -5* -1  15 36 -11* 16  82  2  -2
 17  79  3  -1   1 117  4   1   2 132  6  -2   0   0 35 -14* 17  10 20   3*
 18  34 39  -4*  2 234  4   2   3  31 18  12*  1  72  8   4  18  53  8   1
   H,K=  1,  4   3 100  5   5   4  27 32   8*  2 399 37 -20     H,K=  2,  3
-18   0 22  -8*  4  23 29  11*  5  21 22  -5*  3  56  6  -4 -19  36 15   3*
-17  55  6   4   5 132  5  -2   6 107  4   1   4  48  3   2 -18  63  7  -7
-16  51  9  -4   6 213  5   5   7  12 25   5*  5  21  4   4 -17  32 13  -5*
-15  30  8   5*  7  40 10  -4*  8  29 29  -1*  6 119  4  -6 -16  28 11   0*
-14  13 24   4*  8  43  6   1   9  32  8  10*  7  23 12  -2*-15  46 12   4*
-13  64  5  -7   9  94  4  -5     H,K=  1,  8   8 115  2   1 -14  96  3  -5
-12  81  7   4  10 130  4  -1  -3  22 26   8*  9  12 18  -3*-13  52  3   2
-11   0 25 -14* 11  24  7   7* -2   8 23   2* 10 136  3  -2 -12  56  4  -4
-10  20 38  -9* 12  46  4   0  -1   9 24   2* 11  38 14   3*-11  85  4   2
 -9  87  8  -3  13  59  4  -3   0  16 20   6* 12  54 11  -0*-10  93  4  -3
 -8  82  7   3  14  72  3  -0   1   0 35 -11* 13   0 21  -4* -9  50  4  -6
 -7  19 22  12* 15   7 17   6*  2   9 19   3* 14 101  3  -1  -8  65  3   5
 -6   0 14 -18*   H,K=  1,  6    H,K=  2,  0  15   3 15 -15* -7  82  2  -2
 -5  87  3  -1 -14  20 22   8*-20  39  6  -2  16  68  5   6  -6 128  2   2
 -4  89  6   8 -13  25  7   0*-18  93  2  -2  17   2 23  -1* -5  85  4  -1
 -3   8 17  -1*-12  47  5  -3 -16 128  2   2  18  79  3   3  -4  18  9  -0*
 -2  27  7  -2*-11  29 14  23*-14 165  4  -7    H,K=  2,  2  -3  86  3  -4
 -1   0 18  -5*-10  21 25   6*-12 248  4   6 -20  32  7   1* -2  92  2   0
  0  40 16  -3* -9  13 18 -11*-10 256  4   5 -19  25 29 -16* -1  67  3  -0
  1  22 27   7* -8  59  5   2  -8 522 10  33 -18  73  6   3   0  15  7   3*
  2  46  4   2  -7  11 16   2* -6 238  4   3 -17  12 24  10*  1  50  3  -0
  3  12 14  -4* -6  28  8  16* -4 638 11  -4*-16  94  3  -4   2 123  7   1
  4  56  2  -2  -5  23  6   6* -2 161  6   3 -15  59  7  -4   3 130  4   2
  5  72  2   2  -4  64  2  -3   0 670 25-187*-14 138  4  -1   4  15 19  -1*
  6  49  3   4  -3  12 22   8*  2  78  6   0 -13   0 35  -4*  5  29  3   6
  7  44  5  12  -2  20 16  19*  4 548 15 -23 -12 200  4  -1   6  71  2  -2
  8  56 15  -1* -1  19 26  10*  6 100  2  -0 -11 112  3  -2   7  78  3   4
  9  75 11  -8   0  70  7   5   8 486 12  16 -10 177  3   7   8  75  4   3
 10  38  6  -8   1   0 17  -3* 10 125  2   2  -9  77  4  -6   9  35 17   5*
 11  26 10  21*  2  16 19   6* 12 250  4  -0  -8 332  9   3  10  44  7 -15
 12  63  5  -6   3   0 17  -4* 14  80  3  -1  -7 160  4  -2  11  73  4  -4
 13  64  6  -2   4  49  3  -5  16 102  3  -2  -6  87  2   2  12  41  3  -5
 14  31 19   5*  5  22 29  14* 18  67  4   0  -5  16  5   3* 13  25 29   6*
 15  21 25   5*  6  19 25  -3*   H,K=  2,  1  -4 405 13 -25  14  65  7  -2
 16  44  8   1   7  21 15  1C*-20  18 16   4* -3 134  4  -1  15  41 10  -3*
 17  55  3   6   8  49  7   4 -19  28 34  10* -2  69  2  -3  16  41  6   3
   H,K=  1,  5   9  10 34  -7*-18 113  2  -0  -1 145  2  -7  17  20 27   8*
-16  40 14  -3* 10  26  6  -0*-17   8 25 -10*  0 380 12 -24     H,K=  2,  4
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STRUCTURE FACTORS CONTINUED FOR
 (XEF+)(ASF6-)                                                    PAGE 3

  L FOB SG DEL    L FOB SG DEL    L FOB SG DEL    L FOB SG DEL    L FOB SG DEL
-18  30 18  -5*  5  21 25 -12*  H,K=  3,   0  16  20 30  10* -2 231  7  -3
-17   0 16  -1*  6  15 22 -13*-19  58 10  -5  17  87 10   2  -1 315  8 -11
-16  46 10   2*  7  62  3   9 -17  88  3  -2   H,K=  3,   2   0  35  9  18*
-15  90  3   4   8  24  9  -3*-15  78  3  -7 -19  42  5  -6   1 198 10   6
-14  62 14  -8*  9  17 32   0*-13 175  4  -4 -18  20 29  19*  2 219  6   4
-13  19 22  17* 10  26 31   4*-11  18  6   9*-17  82  4   2   3 233 11   0
-12 115  3   8  11  55  7   4  -9 233  4  -1 -16  43  5  -3   4  31  5   2
-11 147  3  -3  12  30  9   5* -7  70  1   2 -15  63  2  -1   5 237 10   3
-10  91  4  -7  13   0 27 -11* -5 143  3   2 -14  10 18   5*  6 174  8  -2
 -9  55 15  -2* 14  34 11   8* -3 158  3   4 -13 138  4  -1   7 146  5  -3
 -8 159  6  -3   H,K=  2,   6  -1  57  3   1 -12  50  8  -1   8  17 36  -4*
 -7 208  4   0 -13  16 23   9*  1 125  2   1 -11  35 16  -1*  9 158  8  -1
 -6  91  3   3 -12  33 38   5*  3  70  2  -2 -10  27 18  -2* 10  98  3   3
 -5  85  2   1 -11  95  2  -0   5 126  3  -4  -9 158  8   2  11  73  3   1
 -4 278  7  -1 -10  18 24  -6*  7  33  4  -3  -8  76  5  -9  12  14 20  -3*
 -3 253  6  -1  -9  16 23 -15*  9 122  3   1  -7  73  4   5  13 107  2   0
 -2  60  6  -4  -8  45 11  -2* 11  45  9  -6* -6  11 22  -4* 14  61  6  -6
 -1 162  4  -2  -7 121  4   0  13 100  3  -0  -5 124  3   0  15  23  9   8*
  0 273  5  -4  -6  18 22   1* 15   0 31  -8* -4  60  2  -3  16  29 14   9*
  1 198  7  -1  -5  65  5   3  17  78  3   2  -3  51  2   2   H,K=  3,   4
  2  13 19   9* -4  66  2   0   H,K=  3,   1  -2   0 20  -6*-17  48  5   1
  3 176  6   4  -3 131  3  -1 -20   0 28 -13* -1  93  2  -1 -16  54  3  -3
  4 250 14  -1  -2   0 20  -5*-19   6 21  -5*  0  91  3  -3 -15  29 30   8*
  5 169  5   1  -1  88  2   2 -18   9 23 -11*  1 141  4   2 -14  16 20  10*
  6  68  9  10   0  62  5  -4 -17 125  3  -2   2  27  6   1*-13  68  4  -8
  7 161  6   1   1 117  2   0 -16  26  7  -4*  3 111  3   6 -12  64  5  -0
  8 171  9  -3   2  15 17  12*-15  25 13  -1*  4  68  3   3 -11  20 11   2*
  9  80  7   1   3  98  2  -0 -14   0 30 -18*  5 114  3  -2 -10  19 15  -4*
 10  45  8  -5   4  55  6  -8 -13 254  5   1   6  28  8   8* -9  84  2  -3
 11 103  6  -1   5  97  3   3 -12  37 11  -5*  7  12 18   1* -8  62  6 -11
 12  94  8  -1   6  16 23  -2*-11  15 17  10*  8  50 14   3* -7  27 28   3*
 13  37  5  -2   7  96  3  -1 -10  88  4  -2   9  89  7  -3  -6  40 30   4*
 14  44 12  -3*  8  49  7   1  -9 407 10  16  10  16 27   0* -5  67  8   1
 15  66  7  -4   9  53  8   0  -8  48  6   1  11  14 20 -15* -4  61  5   7
 16  43  7   2  10  24 13   2* -7 118  2  -1  12  43  7   5  -3  34  5  13
  H,K=  2,   5  11  73  6   1  -6  93  2  -3  13  83  3   1  -2  13 21  11*
-16  22 25   4* 12  31  7   6* -5 564 12 -16  14  23 27   1* -1  28 26   3*
-15  49 12   4*  H,K=  2,   7  -4  76  2   0  15  29  5  19   0  50  3   4
-14  36  6  -3 -10   7 30   7* -3 260  6  -2  16  25 16   5*  1   9 23   4*
-13  34  7  -1* -9  31  5  18  -2 106  3  -5  17  65 13   4*  2  30 23  14*
-12  31 13   2* -8  15 17  -2* -1 481 34 -22   H,K=  3,   3   3  26 37  19*
-11  50  6  -3  -7  35  7  -1*  0  12 15   9*-18  30 11  -4*  4  35 37  -9*
-10  40  8  -3* -6  10 17   2*  1 304 10  -7 -17  74  5   2   5  42 20  -4*
 -9  32  7  -5* -5  20  8  10*  2 108  3   1 -16  55  5   3   6  28 37  -6*
 -8  31  4  -5  -4  16 22   4*  3 355 19 -13 -15  15 23   5*  7   3 18  -1*
 -7  59  7   1  -3  21 25  -4*  4  63  3  -7 -14  40  6  -9   8  52  4   1
 -6  24 30 -14* -2  14 27   4*  5 320 13  -6 -13 175  6   2   9  39  8  -5*
 -5  47 17 -11* -1  12 22  -3*  6  97  2  -1 -12  91  3  -0  10  16 26 -12*
 -4  23 36  -2*  0   7 28  -2*  7 223  6   0 -11   9 34   8* 11  16 20  13*
 -3  57 16   0*  1  10 25  -1*  8   0 22  -5*-10 114  7  -5  12  33 13  -5*
 -2  33 38  -2*  2   5 24  -7*  9 242  5   3  -9 248 10   3  13  42  5  -3
 -1  75 10   5   3  23 19  -0* 10  52 17   1* -8 104  5   5  14  23 28  -1*
  0  16 31   9*  4  19 23  18* 11 124  4  -3  -7  77  3  -3   H,K=  3,   5
  1  56 20   2*  5   0 25  -3* 12   0 16  -3* -6 173  5   3 -15  10 24   7*
  2   0 37 -25*  6  20 26  10* 13 155  4  -0  -5 350 13  -9 -14  54  6  -1
  3  45 12 -13*  7  24 29  -6* 14  29 17  -6* -4 143  3  -3 -13  58  5   0
  4  18 34  -1*  8   6 20   1* 15  33 12   2* -3 188  5  -2 -12  71  2  -4
```

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STRUCTURE FACTORS CONTINUED FOR
 (XEF+)(ASF6-)                                                      PAGE 4

  L FOB SG DEL    L FOB SG DEL    L FOB SG DEL    L FOB SG DEL    L FOB SG DEL
-11  20  9  15* -2 102  5  -0   9   0 14  -4* -5  42 14  10* -9  34 14   1*
-10 106  4   3  -1  12 21  -6* 10  65 12  -3  -4  59  5   1  -8  32 15  -0*
 -9  88  4   1   0  18 24  -4* 11   4 22 -11* -3  27 17   7* -7  43  8  -2
 -8  80  5   1   1  21 25  -0* 12  59  4   6  -2  36  7   4* -6  23 31   4*
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 -6  16 27   4*-15  11 18   2*  6  28 37  -1* -4 194  4  -0   0  61  3   6
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STRUCTURE FACTORS CONTINUED FOR
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 (XEF+)(ASF6-)                                                              PAGE 6

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  -4   6 23  -9*
  -3  12 18  -4*
  -2   0 17  -9*
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