

Hexafluoroiodium(VII) hexafluoroarsenate

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Received 20 April 2006

Accepted 12 May 2006

Online 15 June 2006

The structure of hexafluoroiodium(VII) hexafluoroarsenate, IF_6AsF_6 , has been determined by X-ray diffraction using a single crystal grown from a saturated solution in anhydrous HF. IF_6AsF_6 crystallizes in the cubic space group $Pa\bar{3}$ with a simple NaCl-like ionic packing. The I and As atoms occupy the $4a$ and $4b$ Wyckoff positions, respectively, with $\bar{3}$ symmetry.

Comment

Until now, the single-crystal X-ray structure of $\text{IF}_6\text{Sb}_2\text{F}_{11}$ represented the only detailed crystallographic study of salts containing the IF_6^+ cation (Lehmann *et al.*, 2004). IF_6AsF_6 was first prepared by the reaction of IF_7 with AsF_5 (Seel & Detmer, 1958, 1959). Powder X-ray diffraction studies of IF_6AsF_6 were performed later (Beaton, 1966; Christe & Sawodny, 1967). We have determined the structure of IF_6AsF_6 by single-crystal diffraction techniques to obtain more precise structural data and to allow a comparison of the cation geometry with that previously found in $\text{IF}_6\text{Sb}_2\text{F}_{11}$.

IF_6AsF_6 crystallizes in the cubic space group $Pa\bar{3}$, in agreement with the published data based on X-ray powder diffraction experiments. The structure consists of alternating IF_6^+ and AsF_6^- octahedra packed in an NaCl-like arrangement (Fig. 1). The closest As...I distance is 4.705 (1) Å. The I and As atoms are both located at sites with $\bar{3}$ symmetry (the $4a$ and $4b$ Wyckoff positions, respectively), resulting in six equal I—F distances and six equal As—F distances. The I—F bond lengths [1.7744 (17) Å] are in the same range as those in $\text{IF}_6\text{Sb}_2\text{F}_{11}$ (1.767–1.782 Å at 173 K; Lehmann *et al.*, 2004). The As—F bond lengths [1.7200 (17) Å] are very similar to those in CsAsF_6 (1.714 Å; Loss & Röhr, 1998). The neighbouring IF_6^+ and AsF_6^- octahedra are mutually tilted, possibly because of packing effects. The geometry of the IF_6^+ cation in IF_6AsF_6 is almost identical to that in $\text{IF}_6\text{Sb}_2\text{F}_{11}$.

Bond-valence analysis of IF_6AsF_6 gives bond-valence sums of 6.972 v.u. (bond valence units) for I and 4.578 v.u. for As (Bresle & O'Keeffe, 1991), with contributions of 1.162 v.u. per F1 atom for the former and 0.763 v.u. per F2 atom for the latter.

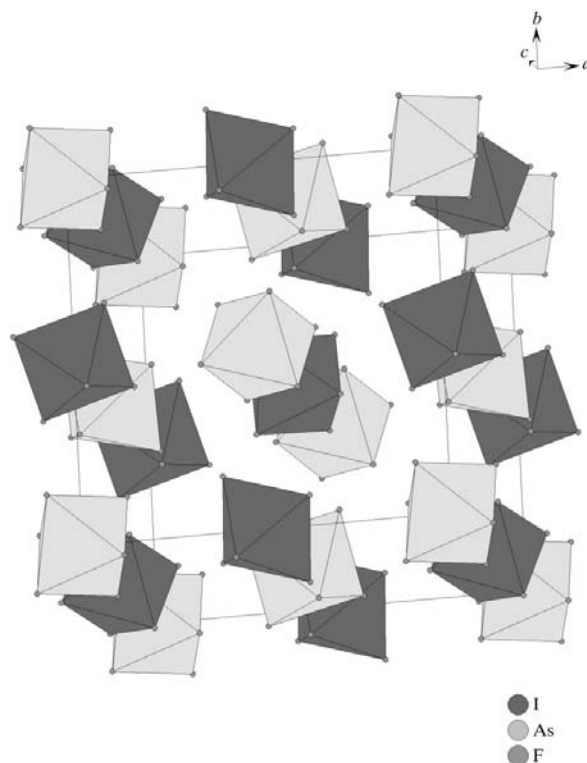


Figure 1

A view of the unit-cell contents of IF_6AsF_6 . IF_6^+ and AsF_6^- ions are shown as light- and dark-grey octahedra, respectively.

Experimental

IF_6AsF_6 was synthesized by the oxidation of iodine pentafluoride by $[\text{Ag}(\text{HF})_n]^{2+}$, prepared by reaction of AgF_2 with AsF_5 in anhydrous HF: $2[\text{Ag}(\text{HF})_n]^{2+} \cdot (\text{AsF}_6)_2^- + \text{IF}_5 \Rightarrow 2(\text{Ag}^+ \cdot \text{AsF}_6^-) + \text{IF}_6\text{AsF}_6 + \text{AsF}_5$. Colourless block-shaped crystals of the title compound were grown from a saturated solution in anhydrous HF. Raman spectra recorded on single crystals were in agreement with literature data for IF_6AsF_6 (Beaton, 1966; Christe & Sawodny, 1967).

Crystal data

AsF_{12}I
 $M_r = 429.82$
Cubic, $Pa\bar{3}$
 $a = 9.409$ (2) Å
 $V = 832.9$ (3) Å³
 $Z = 4$

$D_x = 3.428$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 7.96$ mm⁻¹
 $T = 200$ K
Chunk, colourless
 $0.3 \times 0.3 \times 0.2$ mm

Data collection

Rigaku Mercury CCD
diffractometer
 ω scans
Absorption correction: multi-scan
(Blessing, 1995)
 $T_{\min} = 0.115$, $T_{\max} = 0.2$

3317 measured reflections
354 independent reflections
230 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\text{max}} = 29.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.021$
 $wR(F^2) = 0.063$
 $S = 1.10$
354 reflections
24 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0225P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.72$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.60$ e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.0023 (4)

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Version 3.1; Bergerhoff *et al.*, 1996); software used to prepare material for publication: *WinGX* (Version 1.70; Farrugia, 1999), *enCIFer* (Version 1.2; Allen *et al.*, 2004) and *TEXAN for Windows* (Molecular Structure Corporation, 1999).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BC3005). Services for accessing these data are described at the back of the journal.

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