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# SHORT-FORMAT PAPERS

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## Structure of Nitryl Hexafluoroarsenate(V)

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**Abstract.** $NO_2^+.AsF_6^-$, $M_r = 234{\cdot}92$, monoclinic, $C2/m$, $a = 9{\cdot}053\ (2)$, $b = 5{\cdot}790\ (2)$, $c = 5{\cdot}077\ (2)$ Å, $\beta = 90{\cdot}09\ (3)°$, $V = 266{\cdot}1\ (2)$ Å$^3$, $Z = 2$, $D_x = 2{\cdot}932$ Mg m$^{-3}$, $F(000) = 220$, $\lambda$(Mo $K\alpha$) = 0·71073 Å, $\mu = 6{\cdot}45$ mm$^{-1}$, $T = 291\ (1)$ K, final $R = 0{\cdot}021$ for 263 unique observed [$F \geq 3{\cdot}0\sigma(F)$] diffractometer data. In the crystal, the cation and anion reside on centers of symmetry (2/*m*). The N—O distance in the linear cation is 1·159 (3) Å and the As—F distances in the anion are 1·717 (2) and 1·722 (2) Å. The anion is nearly an ideal octahedron with 180° angles and maximum deviations of 0·5(1)° from the 90° angles. Four symmetrically equivalent F atoms around the nitrogen have a short N···F contact of 2·600 (2) Å with O—N···F angles between 85·9 (1) and 94·1 (1)° and F···N···F angles between 80·2 (1) and 99·8 (1)° besides the 180° angles. Through these short N···F contacts infinite two-dimensional nets form parallel to *y* and *z* and eight-membered ···N···F—As—F···N···F—As—F··· heterocycles are formed.

**Experimental.** Single crystals were prepared from a saturated solution of $NO_2^+.AsF_6^-$ in anhydrous HF at 243 K in a KEL-F reactor equipped with a stainless-steel valve. Over a period of ten days, the HF was removed, stepwise, at this temperature until crystallization began. The solution was then decanted and the crystals dried by pumping out the HF in a high vacuum. Crystal size ~0·09 × 0·12 × 0·12 mm, $D_m$ not determined, $\omega/2\theta$ scan, scan speed 1·50–14·65° min$^{-1}$ in $\theta$, scan width (1·2 + dispersion)°; Nicolet *R*3*m*/*V* diffractometer equipped with an LT-1 low-temperature device, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 28 reflections up to $2\theta = 36{\cdot}63°$; $\omega$ scans of low-order reflections along the three crystal axes showed acceptable mosaicity; six standard reflections (200, 040, 002, $\bar{2}00$, $0\bar{4}0$, $00\bar{2}$) recorded every 2·5 h, only random deviations over 18·65 h of X-ray exposure; 939 reflections measured, $3{\cdot}0 \leq \theta \leq 50{\cdot}0°$, $-10 \leq h \leq 10$, $-6 \leq k \leq 6$, $-6 \leq l \leq 4$; after averaging ($R_{int} = 0{\cdot}024$): 263 unique reflections, 263 with $F \geq 3{\cdot}0\sigma(F)$; Lorentz–polarization correction, no absorption correction; systematic absences (*hkl*) $h + k = 2n + 1$ conform to space groups $C2/m$, $C2$ and $Cm$; structure solution in $C2/m$ *via* direct methods, $\Delta F$ syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all atoms; refinement on $F$ with 263 reflections and 30 refined parameters; $w = 1{\cdot}0/[\sigma^2(F) + (0{\cdot}000686F^2)]$ which led to featureless analysis of variance in terms of $\sin\theta$ and $F_o$; $S = 1{\cdot}046$, $R = 0{\cdot}021$, $wR = 0{\cdot}021$, $(\Delta/\sigma)_{max} = 0{\cdot}004$, no extinction correction; largest peak in final $\Delta F$ map $\pm 0{\cdot}5\ (1)$ e Å$^{-3}$; atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974, Vol. IV); programs: *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987); *PCK*83 (Williams, 1984), *PLATON* (Spek, 1982) and *MISSYM* (Le Page, 1987). The anion and cation with the numbering scheme are shown in Fig. 1 and

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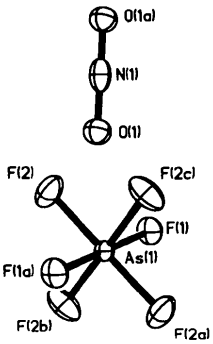


Fig. 1. General view of $NO_2^+.AsF_6^-$ (*SHELXTL-Plus* graphic) showing the atom-numbering scheme [F(1), As(1), F(1*a*) and the anion are on a mirror plane and one twofold axis perpendicular to this plane bisects the angle F(2)—As(1)—F(2*b*) and another passes through N].

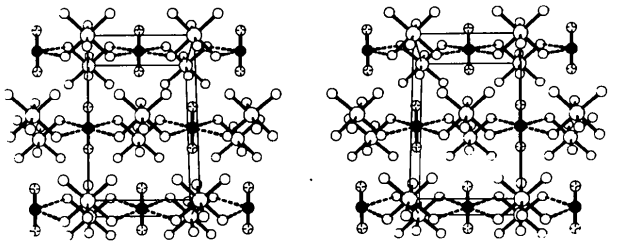

Fig. 2. Stereoscopic view [*SCHAKAL* (Keller, 1986) graphic] of the unit cell (**a** vertical, **b** horizontal).

a stereoscopic view of the unit cell in Fig. 2. Positional parameters and the equivalent values of the anisotropic temperature factors are given in Table 1.* Bond lengths, bond angles and short interionic distances are given in Table 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53343 (3 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* ($Å^2 \times 10^4$)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

| | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|
| As(1) | 0·0 | 0.0 | 0·0 | 170 |
| F(1) | 0·1201 (3) | 0·0 | −0·2614 (4) | 297 |
| F(2) | 0·1028 (2) | 0·2108 (4) | 0·1540 (4) | 433 |
| N(1) | 0·5 | 0·0 | 0·5 | 300 |
| O(1) | 0·38718 (3) | 0·0 | 0·4121 (6) | 313 |

Table 2. *Bond distances* (Å), *bond angles* (°) *and a short interionic contact* (Å)

| | | | |
|---|---|---|---|
| As(1)—F(1) | 1·717 (2) | N(1)—O(1) | 1·159 (3) |
| As(1)—F(2) | 1·722 (2) | N(1)···F(2)$^i$ | 2·600 (2) |
| F(1)—As(1)—F(2) | 90·5 (1) | F(2)—As(1)—F(2*b*) | 89·7 (1) |
| F(1)—As(1)—F(2*b*) | 89·5 (1) | | |

Symmetry code: (i) 0·5 + *x*, 0·5 − *y*, *z*.

**Related literature.** In $NO_2^+.ClO_4^-$ (Truter, Cruickshank & Jeffrey, 1960) N—O 1·084 (9) Å and O—N—O 175·2 (14)° were found and in $(NO_2^+)_2S_3O_{10}^{2-}$ (Cruickshank, 1964), N—O between 1·08 and 1·14 Å and O—N—O 166° were found.

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# Structure of Dipotassium Pentachlorooxorhenate(V)

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**Abstract.** $K_2[ReCl_5O]$, $M_r = 457·7$, orthorhombic, *Pnma*, $a = 13·258$ (7), $b = 9·960$ (6), $c = 6·806$ (4) Å, $V = 898·7$ (9) Å$^3$, $Z = 4$, $D_m = 3·38$, $D_x = 3·382$ (4) Mg m$^{-3}$, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 16·6$ mm$^{-1}$, $F(000) = 824$, $T = 304$ (1) K, final $R = 0·0254$ for 994 observed data. In the $[ReCl_5O]^{2-}$

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