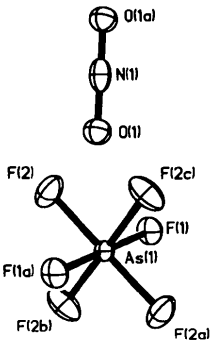

Fig. 1. General view of $NO_2^+.AsF_6^-$ (*SHELXTL-Plus* graphic) showing the atom-numbering scheme [F(1), As(1), F(1*a*) and the anion are on a mirror plane and one twofold axis perpendicular to this plane bisects the angle F(2)—As(1)—F(2*b*) and another passes through N].

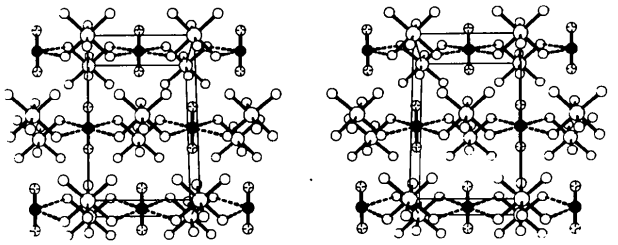

Fig. 2. Stereoscopic view [*SCHAKAL* (Keller, 1986) graphic] of the unit cell (**a** vertical, **b** horizontal).

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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Table 1. *Final atomic parameters for* K_2ReOCl_5

	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Re	0·09817 (2)	0·25	0·17657 (5)	0·0189 (2)	0·0184 (2)	0·0197 (2)	0	0·0008 (3)	0
K	0·36028 (11)	0·00114 (15)	0·18049 (27)	0·0368 (8)	0·0407 (8)	0·0417 (9)	0·0005 (9)	0·0074 (8)	−0·0022 (6)
Cl(1)	0·11004 (11)	0·01200 (13)	0·19374 (26)	0·0344 (9)	0·0205 (6)	0·0376 (9)	−0·0008 (7)	−0·0007 (9)	0·0009 (6)
Cl(2)	0·22408 (17)	0·25	−0·07091 (36)	0·0362 (14)	0·0350 (13)	0·0224 (13)	0	0·0076 (10)	0
Cl(3)	0·24656 (16)	0·25	0·40380 (36)	0·0254 (11)	0·0354 (13)	0·0237 (12)	0	−0·0063 (10)	0
Cl(4)	0·00167 (16)	0·25	0·47183 (37)	0·0290 (13)	0·0329 (12)	0·0349 (15)	0	0·0136 (11)	0
O	−0·00077 (42)	0·25	0·0284 (11)	0·031 (4)	0·035 (4)	0·042 (5)	0	−0·014 (4)	0

anions, which have crystallographic point symmetry m (C_s), the Re—O bond length is 1·655 (6) Å. The Re—Cl bond length [2·502 (2) Å], which is *trans* to the oxo O atom, is longer than the Re—Cl_{cis} distances [2·371 (2)–2·382 (2) Å].

Experimental. The $K_2[ReCl_5O]$ complex was prepared as described by Jakób & Jeżowska (1933). The structure of this compound was earlier determined by photographic methods (Głowiak, 1967) to R = 0·087 for 290 reflexions. D_m by flotation. A yellow-green crystal with dimensions 0·15 × 0·20 × 0·25 mm was used on a Syntex $P2_1$ diffractometer with monochromated Mo $K\alpha$ radiation. Lattice parameters were determined from setting angles of 15 reflexions measured in the range $20 < 2\theta < 26°$. 2θ–ω scans. Intensities of 3299 reflexions measured up to 2θ = 70° (h −20→20, k 0→15, l −10→10); two standard reflexions showed fluctuations of ±5%. The refinement was started with the coordinates from the earlier work (Głowiak, 1967). Scattering factors for K^+, Re, Cl and O atoms and anomalous-dispersion corrections were from *International Tables for X-ray Crystallography* (1974, Vol. IV). An absorption correction was made following the *DIFABS* procedure (Walker & Stuart, 1983), the corrections being between 0·803 and 1·202. The symmetry-related reflexions were averaged to give 994 data with $I > 3\sigma(I)$; R_{int} = 0·0228. All atoms refined (on F) anisotropically (Sheldrick, 1976); final R = 0·0254, wR = 0·0197, where $w = 1/\sigma^2(F_o)$. In final cycle, the largest shift/e.s.d. was 0·05. A final difference Fourier map had a maximum of 0·90 and a minimum of −1·50 e Å^{-3}. No extinction correction.

Final atomic parameters are given in Table 1 and distances and angles in Table 2.* Fig. 1 shows the $[ReCl_5O]^{2-}$ anion and the numbering scheme.

Related literature. For a review of the structures of oxo complexes, see Głowiak (1981).

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* Lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53333 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and potassium coordination distances* (Å) *in* $K_2[ReCl_5O]$

Re—O	1·655 (6)	Re—Cl(1)	2·379 (2)
Re—Cl(2)	2·371 (2)	Re—Cl(3)	2·502 (2)
Re—Cl(4)	2·382 (2)		
O—Re—Cl(1)	94·7 (2)	O—Re—Cl(2)	97·2 (2)
O—Re—Cl(3)	179·4 (2)	O—Re—Cl(4)	95·1 (2)
Cl(1)—Re—Cl(1^{i})	170·6 (1)	Cl(1)—Re—Cl(2)	89·3 (1)
Cl(1)—Re—Cl(3)	85·3 (1)	Cl(1)—Re—Cl(4)	89·7 (1)
Cl(2)—Re—Cl(3)	83·4 (1)	Cl(2)—Re—Cl(4)	167·7 (1)
Cl(3)—Re—Cl(4)	84·3 (1)		
K···Cl(1)	3·321 (3)	K···Cl(1^{ii})	3·339 (3)
Cl(1^{iii})	3·518 (3)	Cl(1^{iv})	3·422 (3)
Cl(2)	3·512 (3)	Cl(2^{v})	3·220 (3)
Cl(3)	3·275 (3)	Cl(3^{vi})	3·436 (3)
Cl(4^{vi})	3·409 (3)	Cl(4^{vii})	3·276 (3)

Symmetry code: (i) x, 0·5 − y, z; (ii) 0·5 − x, −y, z − 0·5; (iii) 0·5 − x, −y, 0·5 + z; (iv) 0·5 + x, y, 0·5 − z; (v) 0·5 − x, y − 0·5, 0·5 + z; (vi) 0·5 − x, y − 0·5, z − 0·5; (vii) 1·5 − x, 1 − y, 1·5 + z.

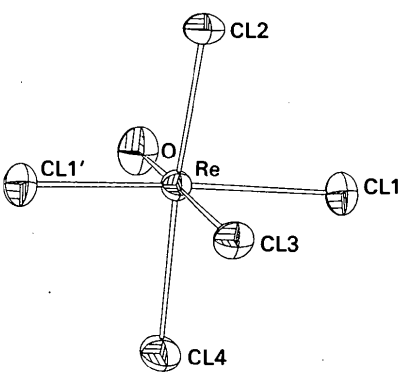

Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 50% probability level.

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