

Tableau 2. Paramètres géométriques (Å, °)

Cu(1)—O(2) $\times 2^{\text{I}}$	1,996 (5)	Cu(2)—O(1) $\times 2^{\text{ii,vi}}$	1,964 (5)
Cu(1)—O(3) $\times 2^{\text{iii,iii}}$	1,910 (4)	Cu(2)—O(2) $\times 2^{\text{iv}}$	2,393 (5)
Cu(1)—O(4) $\times 2^{\text{iv,v}}$	2,888 (5)	Cu(2)—O(4) $\times 2^{\text{iv}}$	2,000 (4)
Na—O(1 ^{vii})	2,386 (5)	Na—O(3)	2,328 (5)
Na—O(1 ^{vi})	2,770 (5)	Na—O(3 ^{ix})	2,545 (5)
Na—O(2 ^{vi})	2,464 (6)	Na—O(4)	2,360 (5)
Na—O(2 ^{viii})	2,559 (5)	Na—O(4 ^s)	2,630 (5)
P—O(1)	1,499 (5)	P—O(3)	1,550 (4)
P—O(2)	1,555 (5)	P—O(4 ⁱⁱⁱ)	1,541 (5)
O(1)—P—O(2)	107,6 (3)	O(2)—P—O(3)	108,7 (2)
O(1)—P—O(3)	110,3 (3)	O(2)—P—O(4 ⁱⁱⁱ)	109,9 (3)
O(1)—P—O(4 ⁱⁱⁱ)	114,8 (2)	O(3)—P—O(4 ⁱⁱⁱ)	105,4 (2)

Codes symétriques: (i) $-x, -y, -z$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (iv) $1 - x, -y, -z$; (v) $x - 1, y, z$; (vi) $\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$; (vii) $1 + x, y, z$; (viii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (ix) $1 - x, 1 - y, -z$; (x) $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

La mesure de la densité des cristaux de la phase- β a été effectuée par flottage dans la solution de Clerici. La densité du liquide a été mesurée en utilisant les petits morceaux de verre dont les densités sont précisément connues. Mais on n'a pas obtenu la valeur précise à cause de la haute viscosité du liquide et aussi de la très petite taille des cristaux. La collecte des données: *Rigaku AFC/MSC Diffractometer Control Program* (Rigaku Corporation, 1991). L'affinement de constantes de la maille: *Rigaku AFC/MSC Diffractometer Control Program*. La réduction des données: *Rigaku AFC/MSC Diffractometer Control Program*. La synthèse de Patterson et de Fourier: *RSSFR-5, UNICS* (Sakurai, 1971). L'affinement par moindres carrés à matrices complètes: *RSFLS-4, UNICS* (Sakurai, 1971). Les distances interatomiques et les angles: *CCPC* (Kawamura & Kawahara, 1980). Le graphique: *ORTEP* (Johnson, 1965). L'impression des tableaux de F_o et σF_o : *LISTHKL* (Yamakawa & Kawahara, 1992).

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Les listes des facteurs de structure et des facteurs d'agitation thermique anisotrope ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 71026: 8 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre. [Référence de CIF: DU1029]

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Bis(tetraphenylarsonium) Aquatetra-(cyano-C)nitridorhenate(V) Pentahydrate

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Abstract

The complex is a salt which contains discrete tetracyanonitridoarhenate(V) anions. Bond lengths and angles are normal.

Comment

Nitrido complexes of rhenium were investigated in the early 1960's (Clifford & Olsen, 1960; Chatt, Garforth, Johnson & Rowe, 1964; Lock & Wilkinson, 1964). The $\nu(\text{Re}\equiv\text{N})$ vibrations of the solid K^+ , Cs^+ and Na^+ salts of $[\text{ReN}(\text{CN})_4]^{2-}$ were different from those in solution (Johnson, 1969). In the solid, $\nu(\text{Re}\equiv\text{N})$ is split into two poorly resolved peaks and is about 100 cm^{-1} lower than that for the solution spectrum. An explanation was suggested by the results of an X-ray structural study of crystals of ' $\text{K}_2[\text{ReN}(\text{NC})_4]\cdot\text{H}_2\text{O}$ ' (Davies, Johnson, Johnson & Graham, 1969). The structure consisted of infinite chains of alternating N and Re atoms, with four cyanide ligands around each Re atom bound through the N atoms. A similar chain structure occurs in WOCl_4 (Adams & Churchill, 1968; Hess & Hartung, 1966).

The reported structure for ' $\text{K}_2[\text{ReN}(\text{NC})_4]\cdot\text{H}_2\text{O}$ ' explained the infrared spectral problem and is often quoted in the literature (Dehnicke & Strähle, 1981). The structure is not reliable, however. The diffraction data were collected with use of $\text{Cu K}\alpha$ radiation, were used without absorption correction, and the 300 reflections were estimated visually. The reported C—N and Re—N bond distances differ significantly from accepted values and the cyanide ligands were coordinated through the N atoms, rather than the normal C atoms.

It is our opinion that the data are not sufficient or precise enough to say that the cyanide ligands were bound through the N atoms, particularly in light of reported structures of Tc and Re cyanide complexes where the cyanide ligands are C-bound (Baldas, Boas, Colmanet & Mackay, 1990; Purcell, Roodt, Basson & Leipoldt, 1990; Purcell, Potgieter, Damoense & Leipoldt, 1991; Purcell, Damoense & Leipoldt, 1992).

No further information has appeared and, in a personal communication to Rouschias (1974), Johnson admitted difficulties in the refinement, and opined that only the Re...Re separation was known with any certainty. We have re-examined the chemistry, but so far have not obtained crystals of $K_2[ReN(CN)_4] \cdot H_2O$ suitable for single-crystal X-ray studies. We have, however, determined the structure of the title compound. Since this paper was submitted for publication we have become aware of another very recent structure determination of the title compound, but which was carried out at room temperature (Purcell, Potgieter, Damoense & Leipoldt, 1992).

$ReNCl_2(PPh_3)_2$ was prepared according to the method of Chatt, Falk, Leigh & Paske (1969) and converted to $K_2[ReN(CN)_4] \cdot H_2O$, (1), by reaction with excess potassium cyanide in methanol (Johnson, 1969). $[AsPh_4]_2[ReN(CN)_4(H_2O)] \cdot 5H_2O$, (2), was prepared by mixing stoichiometrically equivalent amounts of aqueous solutions of $[AsPh_4]Cl$ (232 mg, 0.554 mmol in 10 mL) and (1) (111 mg, 0.277 mmol in 10 mL). Bright yellow crystals of (2) were obtained by recrystallization from a dilute aqueous solution at room temperature. Crystals of (2) are stable in a capped vial, but decompose slowly in air, by loss of water of crystallization.

The anion is shown in Fig. 1. The results observed here are difficult to compare completely with the previous structure determination of the title compound (Purcell, Potgieter, Damoense & Leipoldt, 1992), which we shall refer to as *ReA*, since no atomic coordinates were published. The unit cell for the title compound is about 2.2% smaller than for *ReA*, although the decreases are non-uniform, the decrease in *a* being 1.0, *b* 0.7 and *c* 0.5%. In general, the errors in bond lengths and angles for our compound are about half those reported for *ReA*. With no atom parameters for *ReA*, we cannot make a certain comparison of bond lengths and angles, but if it is assumed that C(1), C(2), C(3) and C(4) in our compound are C(4), C(2), C(1) and C(3) in *ReA*, then most distances and angles are in excellent agreement, and only five differ by more than 1.8σ [$\sigma = (\sigma_1^2 + \sigma_2^2)^{1/2}$]. Of these only one can be considered significant: the Re—O distance in our compound is smaller by 4.3σ than in *ReA* [2.463 (3) versus 2.496 (7) Å].

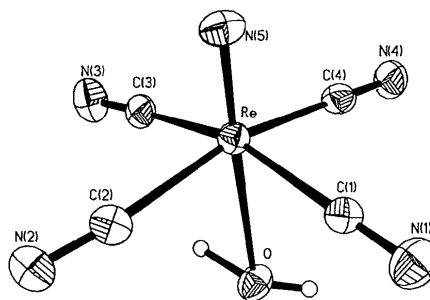


Fig. 1. The molecular anion, showing the atom numbering.

The structure observed here is also very similar to that observed for isomorphous bis[tetraphenylarsonium] aquatetracyanonitridotechnetate(V) pentahydrate (Baldas, Boas, Colmanet & Mackay, 1990). The cells are oriented differently; the cell reported here is related to that of the Tc compound by the matrix 001/0–10/100. In addition, there is an origin shift. The cell for our compound is 2.9% smaller than that for the Tc compound, but the decrease is much more uniform than for *ReA* (0.9% on *a*, 0.8% on *b*, 1.1% on *c*). Again, the cell is smaller because the data were measured at 165 (2) K instead of 298 K, since bond lengths and angles are generally very similar in the two compounds.

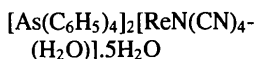
The $Tc \equiv N$ distance [1.596 (10) Å] is significantly shorter than the $Re \equiv N$ distance [1.655 (4) Å, 5.4σ] and the Tc—O distance [2.559 (9) Å] is significantly longer than the Re—O distance [2.463 (3) Å, 10.1σ]. It is argued that the strong *trans* influence of the $Tc \equiv N$ system causes the long Tc—O distance (Baldas, Boas, Colmanet & Mackay, 1990), but non-bonding repulsions may be equally important in determining the structure. The N(5)...C distances in the Re compound average 2.874 Å (*versus* 2.844 Å in the Tc compound), which is significantly shorter than the van der Waals distance [3.20–3.25 Å (Bondi, 1964)]. It is normal for distances between atoms bound to a mutual atom to be shorter than the normal van der Waals distance, the difference varying with the strength of the bonds. Thus, we expect the N(5)...C distances for the Tc compound to be slightly smaller than for the Re compound since the N atom is more strongly bound to the Tc atom. [The Bragg–Slater radii of the two metals are identical (Huheey, 1978).] A contrasting effect is seen for the O...C distances (average 2.990 Å for the Re compound, 3.038 Å for the Tc compound). The van der Waals distance is 3.15–3.20 Å (Bondi, 1964). In this case the O atom bound to Tc is involved in the weaker bond and so the O...C distances are slightly longer for the Tc compound.

In order to confirm that the cyanide groups were bound through the C atoms an alternative series of refinements was undertaken in which the C atoms of

the cyanide groups were given N-atom scattering factors and *vice versa*. The residuals were $R = 0.061$ and $wR = 0.034$. In addition, U_{eq} increased for the C atoms [C(1N) 0.052, C(2N) 0.054, C(3N) 0.051, C(4N) 0.056 Å²], whereas those for the N atoms were reduced [N(1C) 0.023, N(2C) 0.018, N(3C) 0.020, N(4C) 0.024 Å²]. All these results are consistent with the alternative refinement being the wrong solution.

Experimental

Crystal data



$M_r = 1179.0$

Monoclinic

$P2_1/n$

$a = 15.330 (3) \text{ \AA}$

$b = 19.808 (4) \text{ \AA}$

$c = 16.915 (3) \text{ \AA}$

$\beta = 101.638 (15)^\circ$

$V = 5031 (2) \text{ \AA}^3$

$Z = 4$

$D_x = 1.557 \text{ Mg m}^{-3}$

$D_m = 1.53 (1) \text{ Mg m}^{-3}$

Density measured by suspension in $\text{CCl}_4/\text{cyclohexane}$

Ag $K\alpha$ radiation

$\lambda = 0.56086 \text{ \AA}$

Cell parameters from 17 reflections

$\theta = 13.05\text{--}17.45^\circ$

$\mu = 3.774 \text{ mm}^{-1}$

$T = 167 (2) \text{ K}$

Plate

$0.22 \times 0.21 \times 0.11 \text{ mm}$

Yellow

Data collection

Siemens R3m/V diffractometer

θ -2 θ scans

Absorption correction:

refined from ΔF (DI-

FABS; Walker & Stuart, 1983)

$T_{\text{min}} = 0.130$, $T_{\text{max}} = 0.182$

10303 measured reflections

9615 independent reflections

9615 observed reflections

$[-3\sigma_I \leq I \leq 3\sigma_I]$

$R_{\text{int}} = 0.055$

$\theta_{\text{max}} = 20^\circ$

$h = 0 \rightarrow 18$

$k = 0 \rightarrow 24$

$l = -20 \rightarrow 20$

3 standard reflections

monitored every 97

reflections

intensity variation: $\pm 2.1\%$

Refinement

Refinement on F

Final $R = 0.0583$

$wR = 0.0315$

$S = 1.41$

9615 reflections

595 parameters

H-atom parameters not refined

$w = 1/\sigma^2$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 1.15 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -1.38 \text{ e \AA}^{-3}$

Extinction correction: not applied

tors (Table 2.2B) and anomalous-dispersion corrections for N, O, As and Re (Table 2.3.1). Calculations employed *SHELXTL PC* (Sheldrick, 1990) and Laser 386 or IBM 486 computers.

Table 1. Fractional atomic coordinates and equivalent isotropic displacement coefficients (Å²)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Re	0.0302 (1)	0.1695 (1)	0.7643 (1)	0.020
As(2)	0.0587 (1)	0.55941 (1)	0.8276 (1)	0.024
As(1)	0.0583 (1)	0.1905 (1)	0.3947 (1)	0.021
O	-0.0932 (2)	0.1648 (2)	0.8375 (2)	0.029
C(1)	0.1010 (3)	0.1713 (3)	0.8845 (3)	0.025
C(2)	0.0022 (3)	0.2736 (3)	0.7664 (3)	0.026
C(3)	-0.0728 (3)	0.1702 (2)	0.6619 (3)	0.024
C(4)	0.0167 (3)	0.0638 (3)	0.7738 (3)	0.026
N(1)	0.1384 (3)	0.1716 (2)	0.9506 (3)	0.037
N(2)	-0.0185 (3)	0.3293 (2)	0.7584 (2)	0.031
N(3)	-0.1290 (3)	0.1751 (2)	0.6057 (3)	0.036
N(4)	0.0099 (3)	0.0063 (2)	0.7790 (3)	0.035
N(5)	0.1158 (3)	0.1715 (2)	0.7184 (2)	0.030
O(1)	0.3242 (2)	0.2039 (2)	0.0175 (2)	0.036
O(2)	0.3690 (2)	0.0928 (2)	0.1131 (2)	0.040
O(3)	0.8827 (2)	0.0772 (2)	0.9514 (2)	0.036
O(4)	0.1847 (2)	0.0592 (2)	0.0658 (2)	0.038
O(5)	1.0035 (2)	0.0874 (2)	0.0942 (2)	0.043
C(1A)	0.0884 (3)	0.1088 (2)	0.4535 (3)	0.022
C(2A)	0.0937 (3)	0.1055 (3)	0.5365 (3)	0.028
C(3A)	0.1102 (3)	0.0432 (3)	0.5752 (3)	0.033
C(4A)	0.1221 (3)	-0.0136 (3)	0.5317 (3)	0.035
C(5A)	0.1177 (3)	-0.0102 (3)	0.4490 (3)	0.033
C(6A)	0.1001 (3)	0.0520 (2)	0.4087 (3)	0.030
C(1B)	0.1304 (3)	0.1939 (2)	0.3162 (3)	0.025
C(2B)	0.0950 (4)	0.2040 (3)	0.2351 (3)	0.034
C(3B)	0.1527 (4)	0.2086 (3)	0.1806 (3)	0.045
C(4B)	0.2442 (5)	0.2045 (3)	0.2108 (4)	0.054
C(5B)	0.2799 (4)	0.1933 (3)	0.2922 (4)	0.047
C(6B)	0.2223 (3)	0.1885 (2)	0.3448 (3)	0.033
C(1C)	0.0842 (3)	0.2686 (2)	0.4621 (3)	0.022
C(2C)	0.1416 (3)	0.3176 (2)	0.4418 (3)	0.027
C(3C)	0.1618 (3)	0.3738 (3)	0.4920 (3)	0.034
C(4C)	0.1255 (3)	0.3810 (3)	0.5601 (3)	0.033
C(5C)	0.0673 (3)	0.3317 (3)	0.5784 (3)	0.031
C(6C)	0.0462 (3)	0.2753 (2)	0.5288 (3)	0.028
C(1D)	0.1064 (3)	0.5563 (2)	0.7325 (3)	0.026
C(2D)	0.0518 (3)	0.5638 (3)	0.6571 (3)	0.031
C(3D)	0.0880 (4)	0.5644 (3)	0.5886 (3)	0.038
C(4D)	0.1790 (3)	0.5576 (3)	0.5946 (3)	0.034
C(5D)	0.2342 (3)	0.5505 (3)	0.6698 (3)	0.036
C(6D)	0.1980 (3)	0.5492 (3)	0.7390 (3)	0.034
C(1E)	0.1081 (3)	0.4879 (2)	0.8978 (3)	0.024
C(2E)	0.1333 (3)	0.4995 (3)	0.9801 (3)	0.033
C(3E)	0.1678 (3)	0.4464 (3)	1.0302 (3)	0.038
C(4E)	0.1761 (3)	0.3828 (3)	0.9981 (3)	0.037
C(5E)	0.1501 (3)	0.3715 (3)	0.9158 (3)	0.040
C(6E)	0.1178 (3)	0.4249 (2)	0.8654 (3)	0.031
C(1F)	0.0875 (3)	0.6437 (2)	0.8829 (3)	0.028
C(2F)	0.1596 (3)	0.6805 (3)	0.8694 (3)	0.035
C(3F)	0.1803 (4)	0.7406 (3)	0.9106 (3)	0.042
C(4F)	0.1284 (4)	0.7633 (3)	0.9633 (3)	0.043
C(5F)	0.0564 (4)	0.7268 (3)	0.9768 (4)	0.049
C(6F)	0.0357 (3)	0.6649 (3)	0.9363 (3)	0.040
C(1G)	-0.0681 (3)	0.5522 (2)	0.7973 (3)	0.022
C(2G)	-0.1172 (3)	0.6110 (3)	0.7807 (3)	0.032
C(3G)	-0.2093 (3)	0.6068 (3)	0.7566 (3)	0.033
C(4G)	-0.2513 (3)	0.5436 (3)	0.7483 (3)	0.030
C(5G)	-0.2013 (3)	0.4855 (3)	0.7642 (3)	0.033
C(6G)	-0.1087 (3)	0.4895 (3)	0.7898 (3)	0.028
C(1H)	-0.0646 (3)	0.1906 (2)	0.3420 (3)	0.023
C(2H)	-0.1171 (3)	0.2454 (3)	0.3519 (3)	0.028
C(3H)	-0.2062 (3)	0.2452 (3)	0.3130 (3)	0.033
C(4H)	-0.2406 (3)	0.1913 (3)	0.2651 (3)	0.034
C(5H)	-0.1869 (3)	0.1370 (3)	0.2552 (3)	0.036
C(6H)	-0.0979 (3)	0.1363 (2)	0.2939 (3)	0.029

The structure was solved by direct methods. Reflections with $-3\sigma_I \leq I \leq 3\sigma_I$ were treated by the method of French & Wilson (1978). H atoms were placed in calculated positions, with fixed temperature factors, and constrained to ride on the atoms to which they were attached. *International Tables for X-ray Crystallography* (1974, Vol. IV) provided the atomic scattering fac-

Table 2. Geometric parameters (Å, °)

Re—O	2.463 (3)	Re—N(5)	1.655 (4)
Re—C(1)	2.102 (4)	Re—C(2)	2.108 (5)
Re—C(3)	2.096 (4)	Re—C(4)	2.114 (5)
C(1)—N(1)	1.149 (6)	C(2)—N(2)	1.149 (7)
C(3)—N(3)	1.150 (6)	C(4)—N(4)	1.149 (7)
As(2)—C(1D)	1.897 (5)	As(2)—C(1E)	1.904 (5)
As(2)—C(1F)	1.923 (5)	As(2)—C(1G)	1.913 (4)
As(1)—C(1A)	1.908 (5)	As(1)—C(1B)	1.893 (5)
As(1)—C(1C)	1.916 (5)	As(1)—C(1H)	1.914 (4)
O—Re—C(1)	79.2 (2)	O—Re—C(2)	81.3 (2)
O—Re—C(3)	83.6 (2)	O—Re—C(4)	80.0 (2)
O—Re—N(5)	177.7 (1)	N(5)—Re—C(1)	98.7 (2)
N(5)—Re—C(2)	99.7 (2)	N(5)—Re—C(3)	98.5 (2)
N(5)—Re—C(4)	99.1 (2)	C(1)—Re—C(2)	92.0 (2)
C(1)—Re—C(3)	162.7 (2)	C(1)—Re—C(4)	89.0 (2)
C(2)—Re—C(3)	83.4 (2)	C(2)—Re—C(4)	160.8 (2)
C(3)—Re—C(4)	90.0 (2)	Re—C(1)—N(1)	178.7 (4)
Re—C(2)—N(2)	172.0 (4)	Re—C(3)—N(3)	175.5 (4)
Re—C(4)—N(4)	179.6 (5)	C(1D)—As(2)—C(1E)	109.4 (2)
C(1D)—As(2)—C(1F)	110.5 (2)	C(1E)—As(2)—C(1F)	108.6 (2)
C(1D)—As(2)—C(1G)	108.4 (2)	C(1E)—As(2)—C(1G)	111.5 (2)
C(1F)—As(2)—C(1G)	108.4 (2)	C(1A)—As(1)—C(1B)	106.4 (2)
C(1A)—As(1)—C(1C)	112.0 (2)	C(1B)—As(1)—C(1C)	108.0 (2)
C(1A)—As(1)—C(1H)	110.8 (2)	C(1B)—As(1)—C(1H)	109.5 (2)
C(1c)—As(1)—C(1H)	110.0 (2)	As(1)—C(1A)—C(2A)	121.6 (4)
As(1)—C(1A)—C(6A)	116.5 (4)	As(1)—C(1B)—C(2B)	122.2 (4)
As(1)—C(1B)—C(6B)	116.4 (4)	As(1)—C(1C)—C(2C)	118.6 (4)
As(1)—C(1C)—C(6C)	119.7 (4)	As(2)—C(1D)—C(2D)	120.8 (4)
As(2)—C(1D)—C(6D)	119.4 (4)	As(2)—C(1E)—C(2E)	119.9 (4)
As(2)—C(1E)—C(6E)	118.9 (3)	As(2)—C(1F)—C(2F)	119.4 (4)
As(2)—C(1F)—C(6F)	118.4 (4)	As(2)—C(1G)—C(2G)	118.3 (4)
As(2)—C(1G)—C(6G)	120.3 (3)	As(1)—C(1H)—C(2H)	119.2 (3)
As(1)—C(1H)—C(6H)	118.9 (4)		

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Lists of structure factors, anisotropic displacement coefficients, H-atom coordinates, bond distances and angles, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71050 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1026]

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Structures of Amino(triphenyl)phosphonium Bromide and Amino(triphenyl)phosphonium Hexachloroantimonate

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Abstract

The structures of amino(triphenyl)phosphonium bromide and amino(triphenyl)phosphonium hexachloroantimonate are stabilized by hydrogen bonds.

Comment

Amino(triphenyl)phosphonium bromide (I) and amino(triphenyl)phosphonium hexachloroantimonate (II) have been structurally characterized. There are two formula units of (II) in the asymmetric unit. Both compounds form hydrogen bonds from the amino H atoms to the anions. The positions of the amino H atoms were refined with distance restraints for the N—H distances. The N—Br distances in (I) are 3.310 (2) and 3.373 (2) Å; the N—Cl distances in (II) are 3.594 (4), 3.563 (4), 3.740 (5) and 3.537 (5) Å. All other distances and angles are generally as expected. They correspond well with values found in amino(triphenyl)phosphonium