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)		

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

EHMKE POHL, HANS J. GOSINK, REGINE HERBST-IRMER, MATHIAS NOLTEMEYER, HERBERT W. ROESKY AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie, Universität Göttingen, Tammannstrasse 4, 3400 Göttingen, Germany

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

chloride (Hursthouse, Walker, Warrens & Woollins, 1985), amino(triphenyl)phosphonium [1,2-bis(benzamid-2'-olato)phenyl-N,N',O,O']nitridoosmium(IV) (Barner, Collins, Mapes & Santarsiero, 1986) and amino(triphenyl)phosphonium [di(thiazane)-3-eno-N,S](thiosulfato)-(triphenylphosphine)platinate (Hursthouse, Short, Kelly & Woollins, 1988).
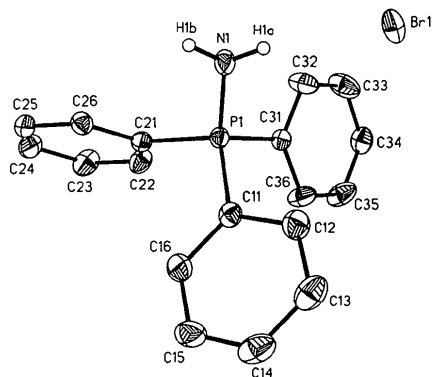

Fig. 1. Structure of (I) showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.
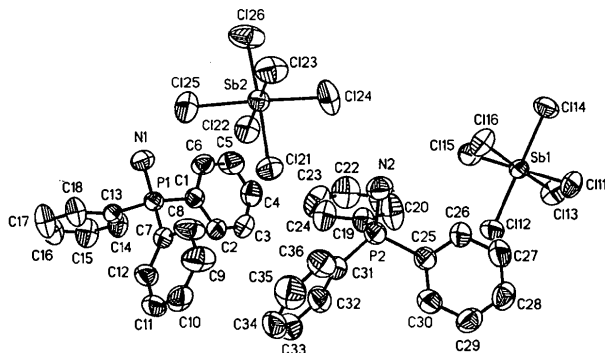

Fig. 2. Structure of (II) showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.

Experimental

Compound (I)

Crystal data

$C_{18}H_{17}NP^+.Br^-$
$M_r = 358.21$
Orthorhombic
$Pna2_1$
$a = 10.9780 (10)$ Å
$b = 9.6280 (10)$ Å
$c = 15.530 (2)$ Å
$V = 1641.5 (3)$ Å^3
$Z = 4$
$D_x = 1.449$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 56 reflections
$\theta = 10-12.5°$
$\mu = 2.595$ mm^{-1}
$T = 153.0 (10)$ K
Block
$0.4 \times 0.2 \times 0.2$ mm
Colourless

Data collection

Stoe–Siemens AED four-circle diffractometer

3385 observed reflections
$[I > 2\sigma(I)]$

Profile data from $2\theta/\omega$ scans
Absorption correction:
 Empirical
 $T_{min} = 0.783$, $T_{max} = 0.952$
3776 measured reflections
3704 independent reflections

$R_{int} = 0.0068$
$\theta_{max} = 29.98°$
$h = -15 \rightarrow 15$
$k = -11 \rightarrow 13$
$l = -21 \rightarrow 21$
3 standard reflections
 frequency: 90 min
 intensity variation: none

Refinement

Refinement on F^2
Final $R = 0.0258$ for $F > 4\sigma(F)$, $R = 0.0327$ for all data
$wR = 0.0547$ for $F > 4\sigma(F)$, $wR = 0.0598$ for all data
$S = 1.066$
3701 reflections
216 parameters
Coordinates of H atoms attached to N refined with distance restraints; those of C—H H atoms not refined
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.0241P)^2 + 0.6395P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.248$ e Å^{-3}
$\Delta\rho_{min} = -0.230$ e Å^{-3}
Extinction correction: SHELXL92
Extinction coefficient: 0.0050 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute structure: $x = -0.016$ (7) (Flack, 1983)
Floating-origin restraints were used (Flack & Schwarzenbach, 1988)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for* (I)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br(1)	0.38157 (2)	0.27359 (2)	0.50000 (2)	0.02694 (9)
P(1)	0.14371 (4)	0.49898 (7)	0.65684 (4)	0.0173 (2)
N(1)	0.1273 (2)	0.3578 (2)	0.60192 (13)	0.0234 (8)
C(11)	0.1653 (2)	0.6551 (2)	0.59526 (13)	0.0209 (9)
C(12)	0.2659 (2)	0.6611 (3)	0.5394 (2)	0.0295 (11)
C(13)	0.2886 (2)	0.7828 (3)	0.4941 (2)	0.0374 (11)
C(14)	0.2121 (2)	0.8956 (3)	0.5018 (2)	0.0356 (12)
C(15)	0.1116 (2)	0.8897 (3)	0.5560 (2)	0.0346 (12)
C(16)	0.0882 (2)	0.7688 (3)	0.6029 (2)	0.0282 (10)
C(21)	0.0109 (2)	0.5193 (2)	0.72264 (13)	0.0191 (9)
C(22)	0.0224 (2)	0.5529 (3)	0.8098 (2)	0.0261 (10)
C(23)	-0.0819 (2)	0.5672 (3)	0.8600 (2)	0.0336 (12)
C(24)	-0.1958 (2)	0.5483 (3)	0.8241 (2)	0.0303 (11)
C(25)	-0.2077 (2)	0.5154 (3)	0.7374 (2)	0.0253 (9)
C(26)	-0.1045 (2)	0.5011 (3)	0.68582 (14)	0.0210 (9)
C(31)	0.2764 (2)	0.4780 (2)	0.72269 (13)	0.0187 (9)
C(32)	0.3141 (2)	0.3456 (3)	0.7437 (2)	0.0314 (13)
C(33)	0.4146 (3)	0.3265 (3)	0.7973 (2)	0.0390 (15)
C(34)	0.4753 (2)	0.4401 (3)	0.8297 (2)	0.0291 (10)
C(35)	0.4378 (2)	0.5722 (3)	0.8101 (2)	0.0351 (13)
C(36)	0.3386 (2)	0.5924 (3)	0.7553 (2)	0.0294 (11)

Table 2. *Geometric parameters* (Å, °) *for* (I)

P(1)—N(1)	1.615 (2)	P(1)—C(11)	1.797 (2)
P(1)—C(21)	1.791 (2)	N(1)—H(1A)	0.842 (22)
P(1)—C(31)	1.791 (2)	N(1)—H(1B)	0.839 (22)
N(1)—P(1)—C(21)	107.58 (10)	C(31)—P(1)—C(11)	106.93 (10)
N(1)—P(1)—C(31)	107.31 (10)	P(1)—N(1)—H(1A)	119.6 (21)
C(21)—P(1)—C(31)	110.39 (10)	P(1)—N(1)—H(1B)	118.4 (21)
N(1)—P(1)—C(11)	115.96 (11)	H(1A)—N(1)—H(1B)	113.9 (30)
C(21)—P(1)—C(11)	108.64 (11)		

Table 3. *Hydrogen-bonding geometry* (Å, °) *for* (I)

D	H	A	H···A	D···A	D—H···A
N(1)	H(1A)	Br(1)	2.481 (22)	3.310 (2)	168.4 (2.7)
N(1)	H(1B)	Br(1^i)	2.560 (23)	3.373 (2)	163.5 (2.8)

Symmetry code: (i) $-0.5 + x, 0.5 - y, z$.

Compound (II)

Crystal data

$C_{18}H_{17}NP^+.SbCl_6^-$
$M_r = 612.75$
Monoclinic
$P2_1/n$
$a = 14.3940\ (10)$ Å
$b = 16.7120\ (10)$ Å
$c = 20.177\ (2)$ Å
$\beta = 90.440\ (10)°$
$V = 4853.5\ (7)$ Å^3
$Z = 8$

$D_x = 1.677$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 34 reflections
$\theta = 10-12.5°$
$\mu = 1.868$ mm^{-1}
$T = 293.0\ (10)$ K
Block
$0.5 \times 0.4 \times 0.3$ mm
Colourless

Data collection

Stoe–Siemens AED four-circle-diffractometer
Profile data from $2\theta/\omega$ scans
Absorption correction:
  Empirical
$T_{min} = 0.233$, $T_{max} = 0.283$
6622 measured reflections
6320 independent reflections

5329 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0204$
$\theta_{max} = 22.50°$
$h = -15 \rightarrow 15$
$k = -17 \rightarrow 17$
$l = 0 \rightarrow 21$
3 standard reflections
  frequency: 90 min
  intensity variation: none

Refinement

Refinement on F^2
Final $R = 0.0302$ for $F > 4\sigma(F)$, $R = 0.0392$ for all data
$wR = 0.0625$ for $F > 4\sigma(F)$, $wR = 0.0667$ for all data
$S = 1.079$
6320 reflections
500 parameters
Coordinates of H atoms attached to N refined with distance restraints; those of C—H H atoms not refined

Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.0225P)^2 + 5.7476P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.003$
$\Delta\rho_{max} = 0.428$ e Å^{-3}
$\Delta\rho_{min} = -0.348$ e Å^{-3}
Extinction correction: *SHELXL92*
Extinction coefficient: 0.00046 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

For both compounds: Data collection: Stoe *DIF*4. Cell refinement: Stoe *DIF*4. Data reduction: Stoe *REDU*4. Program(s) used to solve structure: *SHELXS92* (Sheldrick, 1990a). Program(s) used to refine structure: *SHELXL92* (Sheldrick, 1992). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990b). Software used to prepare material for publication: *SHELXL92*.

Table 4. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

$U_{eq} = \frac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Sb(1)	0.12802 (2)	0.22996 (2)	0.875917 (14)	0.0487 (2)
Cl(11)	−0.03560 (7)	0.22566 (8)	0.88310 (6)	0.0693 (6)
Cl(12)	0.11563 (9)	0.32889 (8)	0.79347 (7)	0.0755 (8)
Cl(13)	0.12652 (10)	0.12931 (8)	0.79422 (7)	0.0810 (10)
Cl(14)	0.13996 (9)	0.13084 (9)	0.95975 (7)	0.0820 (8)
Cl(15)	0.29123 (8)	0.23702 (9)	0.87096 (7)	0.0749 (6)
Cl(16)	0.12907 (9)	0.33025 (8)	0.96012 (7)	0.0772 (8)
Sb(2)	0.37830 (2)	0.71300 (2)	1.107288 (15)	0.0511 (2)
Cl(21)	0.27434 (8)	0.76906 (8)	1.02761 (6)	0.0667 (7)
Cl(22)	0.48881 (8)	0.68884 (8)	1.02445 (6)	0.0721 (8)
Cl(23)	0.26714 (9)	0.73870 (10)	1.18969 (7)	0.0838 (8)
Cl(24)	0.31206 (10)	0.58640 (8)	1.08729 (9)	0.0987 (10)
Cl(25)	0.44417 (9)	0.84172 (8)	1.12219 (7)	0.0781 (9)
Cl(26)	0.48060 (10)	0.66181 (11)	1.18771 (8)	0.1015 (9)
P(1)	0.61656 (7)	0.87033 (6)	0.91920 (5)	0.0449 (6)
N(1)	0.6189 (3)	0.8766 (3)	0.9993 (2)	0.058 (2)
C(1)	0.6249 (3)	0.7706 (2)	0.8881 (2)	0.048 (2)
C(2)	0.5871 (3)	0.7506 (3)	0.8269 (2)	0.060 (3)
C(3)	0.5978 (3)	0.6745 (3)	0.8024 (3)	0.067 (3)
C(4)	0.6437 (3)	0.6179 (3)	0.8387 (3)	0.072 (3)
C(5)	0.6813 (4)	0.6370 (3)	0.8989 (3)	0.077 (4)
C(6)	0.6727 (3)	0.7134 (3)	0.9239 (2)	0.062 (3)
C(7)	0.5092 (3)	0.9133 (2)	0.8914 (2)	0.046 (2)
C(8)	0.4275 (3)	0.8903 (4)	0.9209 (3)	0.080 (3)
C(9)	0.3446 (3)	0.9227 (4)	0.9002 (3)	0.088 (3)
C(10)	0.3427 (3)	0.9782 (3)	0.8502 (3)	0.073 (3)
C(11)	0.4228 (4)	1.0002 (3)	0.8202 (3)	0.078 (4)
C(12)	0.5064 (3)	0.9677 (3)	0.8411 (2)	0.066 (3)
C(13)	0.7141 (3)	0.9265 (2)	0.8899 (2)	0.047 (2)
C(14)	0.7715 (3)	0.8962 (3)	0.8421 (2)	0.068 (3)
C(15)	0.8468 (4)	0.9420 (3)	0.8212 (3)	0.082 (3)
C(16)	0.8640 (3)	1.0138 (3)	0.8477 (3)	0.078 (3)
C(17)	0.8067 (4)	1.0447 (3)	0.8944 (3)	0.087 (3)
C(18)	0.7322 (3)	1.0013 (3)	0.9159 (3)	0.070 (3)
P(2)	0.11079 (8)	0.64211 (7)	0.90609 (6)	0.0538 (7)
N(2)	0.0990 (3)	0.6280 (3)	0.9858 (2)	0.067 (3)
C(19)	0.2189 (3)	0.5963 (3)	0.8852 (2)	0.060 (3)
C(20)	0.2238 (4)	0.5163 (3)	0.8711 (4)	0.103 (4)
C(21)	0.3096 (5)	0.4795 (4)	0.8636 (4)	0.131 (5)
C(22)	0.3884 (4)	0.5219 (4)	0.8697 (3)	0.102 (4)
C(23)	0.3849 (4)	0.6014 (4)	0.8811 (3)	0.092 (4)
C(24)	0.3000 (3)	0.6392 (3)	0.8883 (3)	0.076 (3)
C(25)	0.0127 (3)	0.5938 (3)	0.8680 (2)	0.053 (3)
C(26)	−0.0112 (3)	0.5176 (3)	0.8886 (3)	0.071 (3)
C(27)	−0.0847 (4)	0.4775 (3)	0.8590 (3)	0.079 (3)
C(28)	−0.1351 (3)	0.5154 (3)	0.8105 (3)	0.071 (3)
C(29)	−0.1135 (3)	0.5907 (3)	0.7904 (2)	0.069 (3)
C(30)	−0.0391 (3)	0.6301 (3)	0.8188 (2)	0.064 (3)
C(31)	0.1113 (3)	0.7453 (3)	0.8817 (2)	0.057 (3)
C(32)	0.1497 (4)	0.7689 (3)	0.8222 (2)	0.072 (4)
C(33)	0.1470 (4)	0.8478 (4)	0.8043 (3)	0.087 (4)
C(34)	0.1054 (4)	0.9028 (4)	0.8443 (4)	0.089 (4)
C(35)	0.0684 (4)	0.8793 (4)	0.9038 (4)	0.094 (4)
C(36)	0.0711 (4)	0.8010 (3)	0.9227 (3)	0.073 (4)

Table 5. *Geometric parameters* (Å, °) *for* (II)

P(1)—N(1)	1.619 (4)	P(2)—N(2)	1.636 (4)
P(1)—C(1)	1.786 (4)	P(2)—C(19)	1.788 (5)
P(1)—C(7)	1.790 (4)	P(2)—C(31)	1.794 (5)
P(1)—C(13)	1.793 (4)	P(2)—C(25)	1.794 (4)
N(1)—H(1A)	0.771 (22)	N(2)—H(2A)	0.773 (22)
N(1)—H(1B)	0.771 (22)	N(2)—H(2B)	0.773 (22)
N(1)—P(1)—C(1)	114.2 (2)	N(2)—P(2)—C(19)	105.5 (2)
N(1)—P(1)—C(7)	107.4 (2)	N(2)—P(2)—C(31)	114.1 (2)
C(1)—P(1)—C(7)	109.0 (2)	C(19)—P(2)—C(31)	110.0 (2)
N(1)—P(1)—C(13)	106.5 (2)	N(2)—P(2)—C(25)	105.6 (2)
C(1)—P(1)—C(13)	108.6 (2)	C(19)—P(2)—C(25)	113.0 (2)
C(7)—P(1)—C(13)	111.3 (2)	C(31)—P(2)—C(25)	108.7 (2)
P(1)—N(1)—H(1A)	116.1 (38)	P(2)—N(2)—H(2A)	113.0 (42)
P(1)—N(1)—H(1B)	121.1 (38)	P(2)—N(2)—H(2B)	116.8 (41)
H(1A)—N(1)—H(1B)	111.3 (53)	H(2A)—N(2)—H(2B)	115.7 (59)

Table 6. *Hydrogen-bonding geometry* (Å, °) *for* (II)

D	H	A	H···A	D···A	D—H···A
N(1)	H(1A)	Cl(14^i)	2.824 (26)	3.563 (4)	161.3 (4.7)
N(1)	H(1B)	Cl(25)	2.853 (26)	3.594 (4)	161.7 (4.7)
N(2)	H(2A)	Cl(16^i)	2.778 (24)	3.537 (5)	167.7 (5.3)
N(2)	H(2B)	Cl(24)	3.030 (31)	3.740 (5)	154.0 (5.1)

Symmetry code: (i) $1 - x, 1 - y, 2 - z$; (ii) $-x, 1 - y, 2 - z$.

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Structure of Potassium Silanolate at 153 K

FRANK PAUER AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie, Universität Göttingen, Tammannstrasse 4, 3400 Göttingen, Germany

Abstract

The structure of tetrapotassium tetrakis(2-methyl-2-sila-2-propanolate), (KOSiMe₃)₄, is reported. The cubane-like tetramer lies on a position of crystallographic symmetry 23; the Me₃SiO unit and the K atom lie on a crystallographic threefold axis.

Comment

The structure of (KOSiMe₃)₄ has been determined previously from powder diffraction data. It was published in the

space group $P\bar{4}3m$ [a = 8.844 (1) Å (Weiss, Hoffmann & Grützmacher, 1990)]. The single-crystal X-ray diffraction data show that after doubling the axes [a = 17.573 (2) Å] additional weak *uuu* reflections are present. This leads to an *F*-centred lattice and the space group $F\bar{4}3c$. Omitting the reflections with *uuu* indices gives the primitive cell mentioned above. Presumably, these *uuu* reflections were overlooked in the powder diffraction experiment because of their relative weakness. The good agreement of the single-crystal data with the $F\bar{4}3c$ model makes it very probable that this is the correct space group. This may also be true for the isostructural Rb and Cs species. The change of space group reduces the symmetry of the tetramer from $T_d(\bar{4}3m)$ to $T(23)$ and involves a rotation of the Me₃Si group by 19.5° about the threefold axis. There are no close intermolecular contacts.
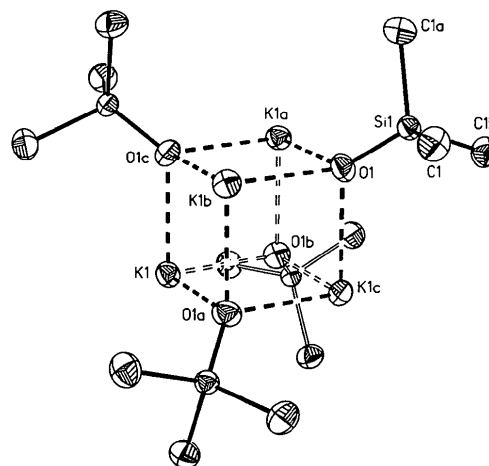

Fig. 1. Structure of the title compound showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.

Experimental

Crystal data

4K⁺.4C₃H₉OSi⁻	Cell parameters from 60
M_r = 513.2	reflections
Cubic	θ = 8–55°
$F\bar{4}3c$	μ = 0.845 mm⁻¹
a = 17.573 (2) Å	T = 153 (2) K
V = 5426.7 (11) Å³	Cube
Z = 8	0.5 × 0.5 × 0.5 mm
D_x = 1.256 Mg m⁻³	Colourless
Mo $K\alpha$ radiation	
λ = 0.71072 Å	

Data collection

Stoe–Siemens AED diffractometer	R_{int} = 0.0263
	θ_{max} = 27.46°
Profile data from $2\theta/\omega$ scans	h = −22 → 22
Absorption correction:	k = −22 → 22
none	l = −13 → 13