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The Crystal Structures of the Adducts $SbCl_5 \cdot POCl_3$, $SbCl_5 \cdot (CH_3)_3PO$ and $NbCl_5 \cdot POCl_3$

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A further refinement of the crystal structures of $SbCl_5 \cdot POCl_3$ and $SbCl_5 \cdot (CH_3)_3PO$ is reported, together with the structure determination of $NbCl_5 \cdot POCl_3$. The three compounds are isotypic.

The crystal structures of $SbCl_5 \cdot POCl_3$[1] and $SbCl_5 \cdot (CH_3)_3PO$ [2] have already been determined and refined by Fourier methods. These refinements have now been carried a stage further using a least-square method which has also been used in the refinement of the structure of $NbCl_5 \cdot POCl_3$.

EXPERIMENTAL

Refinement method. The structures have been refined by the least squares method using a programme for the Swedish electronic computer FACIT EDB devised by Åsbrink and Brändén [3]. This programme is based mainly on the schemes outlined by Cruickshank [4] in his lecture at the Manchester Summer-school devoted to Modern Methods of X-ray Crystallography. The programme refines atomic coordinates, isotropic vibration parameters and individual scalefactors for each layer-line by minimizing the function $\Sigma\, w(|F_o| - |F_c|)^2$. The "block-diagonal approximation" is used (3×3 matrices for atomic coordinates and 1×1 for individual vibration parameters).

In concluding the scale-factor shifts an "over-all" isotropic vibration parameter is introduced as a semi-dummy parameter. In the corresponding matrix all off-diagonal terms except interaction terms between scale-factor shifts and the "over-all" vibration parameter shift are neglected. The solution of these equations gives scale-factor shifts for each scale-factor and an "over-all" thermal parameter shift with allowance for their interaction. The individual vibration parameter-shifts obtained from the 1×1 matrices are corrected to allow for their interaction with the scale-factor shifts by applying a "back-shift" correction suggested by Cruickshank and described by Hoch and Mills [5].

The weights have been computed using either the scheme proposed by Hughes [6]:

$$w = 1/|4F_{o,\min}|^2 \quad \text{for } |F_o| \leq 4\,|F_{o,\min}|$$
$$w = 1/|F_o|^2 \quad \text{for } |F_o| \geq 4\,|F_{o,\min}|$$

or that suggested by Cruickshank [4]:

$$w = 1/(a + |F_o| + c|F_o|^2)$$

At the end of each refinement cycle, an analysis of the weights is made by computing $\overline{w\Delta^2}$ (normalized) as a function of both sin Θ and $|F_o|$.

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The parameter-shifts have been treated by "shift-factor" according to the formula:

$z_k = \alpha y_k + \beta z_{k-1}$ where
z_k = applied shift in cycle k
y_k = calculated shift in cycle k
z_{k-1} = applied shift in cycle $k-1$

In the refinement of these structures rapid convergence was obtained with $\alpha = 0.8$ and $\beta = 0.2$.

The e.s.d.'s have been calculated using the formula: $\sigma^2(x) = a^{ii}(\Sigma\ w\Delta^2)/(m-n)$ which is explained in Cruickshank's paper [4].

The isotropic vibration parameter is defined as B in the expression $\exp[-(B \sin^2\Theta/\lambda^2)]$ for the temperature factor.

Experimental data for $NbCl_5 \cdot POCl_3$. Single crystals of the hygroscopic compound were prepared in sealed capillary tubes by a zone-melting technique. The unit-cell dimensions of the orthorhombic crystals were determined from quartz-calibrated rotation and zero-layer Weissenberg photographs. (a = 4.910 Å for α-quartz, $CuK\alpha_1$ = 1.54050 Å). The values are:

a = 8.07 ± 0.01 Å
b = 16.23 ± 0.03 Å
c = 8.83 ± 0.02 Å

The structure is isostructural with that of $SbCl_5 \cdot POCl_3$[1] and it was assumed that they share the same space-group $P\ m\ n\ b$ (Extinctions $h\ 0\ l$ for $h + l$ odd and $h\ k\ 0$ for k odd.)

The single crystal was rotated around the c-axis and Weissenberg photographs were taken with CuK- and MoK-radiation. 13 zones (0—12) were recorded with Nb-filtered, MoK α-radiation using a multiple-film technique with iron foils between the films. The layer lines were correlated using a method described by Magnéli [7]. The 1135 independent intensities were estimated visually and corrected for Lorentz and polarization effects. We are indebted to S. Sandborg for help with the X-ray work.

RESULTS

$SbCl_5 \cdot POCl_3$. The final atomic parameters obtained by Fourier refinement[1] were used as a starting point for the least-quares refinement employing 1124 independent observed intensities. 21 reflexions were rejected during the refinement, 7 of these because of extinction errors and 10 owing to the assignment of incorrect indices while the remaining 4 were very weak. (The programme rejected a reflexion if $|\Delta| \geq 128$ or $|F_c| \leq 0.3|F_o|$.) Atomic coordinates, individual isotropic vibration parameters and individual scale-factors for each layer line were refined. The weights were calculated using Cruickshank's scheme with $a = 18$ and $c = 0.0095$.

After five cycles of refinement all shifts were less than one tenth of the e.s.d.'s and the usual disagreement index R for the observed reflexions had decreased from 0.203 to 0.125. The largest shift in the atomic coordinates was 0.029 Å. The weight-analysis obtained in the last cycle of refinement is given in Table 1 and the final atomic parameters and their e.s.d.'s are given in Table 2. Tables of calculated and observed $F(h,k,l)$ can be obtained from this Institute.

For 14 of the atomic coordinates, the differences between the parameters obtained from Fourier refinement and from least-squares refinement are less than the e.s.d.'s. Of the remaining six coordinates, only two, namely the y-coordinates of the two Cl-atoms in general positions, show differences more than 1.5 times the e.s.d.'s. These differences may have arisen because only an

Table 1. Weight-analysis in $SbCl_5 \cdot POCl_3$.

sin Θ-interval	$\overline{w\Delta^2}$	number of reflexions in each interval
0.00—0.27	1.08	240
0.27—0.35	0.98	230
0.35—0.39	1.19	188
0.39—0.43	0.97	148
0.43—0.47	0.82	114
0.47—0.50	0.99	97
0.50—0.52	0.79	56
0.52—0.59	0.59	30
$\|F_o\|$-interval		
0— 23	1.10	449
23— 45	0.77	369
45— 68	0.92	143
68— 90	1.06	76
90—113	0.81	31
113—135	2.32	22
135—225	2.44	13

Table 2. Final atomic parameters and their e.s.d.'s for $SbCl_5 \cdot POCl_3$.

		x	y	z	B (Å^2)	$\sigma(x)$ Å	$\sigma(y)$ Å	$\sigma(z)$ Å	$\sigma(B)$ Å^2
Sb	(1)	0.25000	0.14500	0.07972	3.029	—	0.0016	0.0018	0.023
Cl	(2)	0.25000	0.25863	0.92004	3.839	—	0.0076	0.0082	0.116
Cl	(3)	0.25000	0.02088	0.20502	4.996	—	0.0100	0.0101	0.168
Cl	(4)	0.25000	0.22479	0.29381	5.082	—	0.0099	0.0104	0.171
Cl	(5)	0.96179	0.13874	0.05956	4.739	0.0069	0.0061	0.0066	0.109
O	(6)	0.25000	0.07193	0.87770	3.656	—	0.0204	0.0216	0.360
P	(7)	0.25000	0.07404	0.71325	3.199	—	0.0072	0.0072	0.111
Cl	(8)	0.25000	0.46442	0.86926	4.917	—	0.0095	0.0104	0.162
Cl	(9)	0.05717	0.13032	0.63227	5.151	0.0078	0.0066	0.0079	0.122

"over-all" temperature-factor and one scale-factor were included in the Fourier refinement leading to errors in the signs of some structure-factors and to some distortion of the maxima in the Fourier synthesis. However, Fourier and least-squares refinements made in other laboratories[8] have also yielded different results, even when the same parameters have been refined. The differences in the atomic coordinates do not produce any significant alteration (3 times e.s.d.) in the bond lengths or bond angles of the molecule.

$SbCl_5 \cdot (CH_3)_3PO$. Starting with the final atomic parameters obtained by Fourier refinement[2] a least-squares refinement has been made using 420 observed independent intensities. No reflections were rejected during the refinement. (A correction for extinction had already been made[2].) Atomic coordinates, individual isotropic vibration parameters and individual scale-factors for each layer line were refined. The weights were calculated using Cruickshank's weighting-scheme with $a = 20$ and $c = 0.0070$.

Table 3. Weight-analysis in $SbCl_5 \cdot (CH_3)_3PO$.

sinΘ-interval	$\overline{w\Delta^2}$	number of reflexions in each interval
0.00 – 0.46	1.12	128
0.46 – 0.58	0.83	91
0.58 – 0.66	0.76	75
0.66 – 0.73	1.23	51
0.73 – 0.79	1.18	28
0.79 – 0.84	0.84	19
0.84 – 0.88	0.74	14
0.88 – 0.99	1.18	14
$\lvert F_o \rvert$-interval		
0 – 35	1.15	102
35 – 70	0.94	211
70 – 105	0.87	71
105 – 140	1.46	21
140 – 175	0.65	7
175 – 210	1.56	6
210 – 350	1.32	2

Table 4. Final atomic parameters and their e.s.d.'s for $SbCl_5 \cdot (CH_3)_3PO$.

		x	y	z	B (Å²)	$\sigma(x)$ Å	$\sigma(y)$ Å	$\sigma(z)$ Å	$\sigma(B)$ Å²
Sb	(1)	0.25000	0.14578	0.06936	3.621	–	0.0037	0.0041	0.060
Cl	(2)	0.25000	0.26220	0.92157	4.431	–	0.0143	0.0174	0.292
Cl	(3)	0.25000	0.02680	0.21235	4.751	–	0.0150	0.0168	0.336
Cl	(4)	0.25000	0.22399	0.28816	5.184	–	0.0158	0.0177	0.353
Cl	(5)	0.96646	0.14253	0.05780	5.292	0.0116	0.0108	0.0123	0.216
O	(6)	0.25000	0.07950	0.88952	3.787	–	0.0340	0.0347	0.771
P	(7)	0.25000	0.08017	0.71331	3.839	–	0.0147	0.0169	0.302
C	(8)	0.25000	0.47540	0.85277	6.942	–	0.0715	0.0713	1.811
C	(9)	0.07341	0.13337	0.64474	7.017	0.0542	0.0483	0.0557	1.246

After seven cycles of refinement all shifts were less than one tenth of the e.s.d. and the R-value had dropped from 0.130 to 0.108. The largest shift in the atomic coordinates was 0.24 Å. The weight-analysis obtained in the last cycle of refinement is given in Table 3 and the final atomic parameters and their e.s.d's are given in Table 4. (The coordinates quoted here have been transformed to agree with the space-group $P\,m\,n\,b$). Tables of calculated and observed $F(h,k,l)$ can be obtained from this Institute.

For 13 of the atomic coordinates, the differences between the parameters obtained from Fourier refinement and from the least-squares refinement are larger than the e.s.d.'s These differences probably arise owing to a combination of the effects mentioned in connection with the refinement of $SbCl_5 \cdot POCl_3$ and the fact that the Fourier refinement was terminated when the shifts were smaller, but of the same order of magnitude, as the e.s.d.'s. Further Fourier refinement might give shifts which, when added together from successive cycles of refinement, could be larger than the e.s.d.'s, thus giving closer agreement between the two methods. One other factor was the difficulty of

Table 5. Weight-analysis in $NbCl_5 \cdot POCl_3$.

sin Θ-interval	$\overline{w\Delta^2}$	number of reflexions in each interval
0.00 – 0.30	1.12	306
0.30 – 0.38	1.14	275
0.38 – 0.44	0.98	212
0.44 – 0.48	0.84	140
0.48 – 0.52	0.64	117
0.52 – 0.55	0.94	58
0.55 – 0.65	0.89	15
$\|F_o\|$-interval		
0 – 25	0.94	486
25 – 50	1.04	418
50 – 75	1.00	120
75 – 100	0.74	56
100 – 125	2.09	26
125 – 225	0.99	17

Table 6. Final atomic parameters and their e.s.d.'s for $NbCl_5 \cdot POCl_3$.

	x	y	z	B (Å^2)	$\sigma(x)$ Å	$\sigma(y)$ Å	$\sigma(z)$ Å	$\sigma(B)$ Å^2
Nb (1)	0.25000	0.14611	0.08525	2.119	–	0.0019	0.0019	0.024
Cl (2)	0.25000	0.26120	0.92455	2.909	–	0.0065	0.0068	0.097
Cl (3)	0.25000	0.02157	0.20776	4.359	–	0.0093	0.0094	0.153
Cl (4)	0.25000	0.22639	0.29339	4.244	–	0.0091	0.0093	0.149
Cl (5)	0.96646	0.13920	0.05964	4.278	0.0068	0.0060	0.0064	0.105
O (6)	0.25000	0.07590	0.87727	2.861	–	0.0190	0.0192	0.317
P (7)	0.25000	0.07487	0.71279	2.024	–	0.0060	0.0057	0.084
Cl (8)	0.25000	0.46300	0.86664	3.964	–	0.0086	0.0087	0.136
Cl (9)	0.05759	0.13033	0.62857	3.920	0.0069	0.0055	0.0061	0.094

locating the carbon atoms accurately in the Fourier synthesis; consequently the largest differences occur in the parameters of these atoms.

Since the intensities for this structure had been obtained from a very small crystal using Cu*K*-radiation, the number of observed reflexions are less than half the number observed for the other structures reported here, where the intensities were recorded with Mo*K*-radiation using a larger crystal. This is reflected in the e.s.d.'s of the parameters which are approximately twice as large for this structure.

$NbCl_5 \cdot POCl_3$. The final atomic coordinates obtained by Fourier refinement of $SbCl_5 \cdot POCl_3$[1], and a vibration parameter of 2.73 Å^2 for all atoms were used as a starting point for a least squares refinement employing 1135 observed intensities. 12 very weak reflexions for which $|F_c| \leq 0.3\ |F_o|$ were rejected from the refinement. Atomic coordinates, individual isotropic vibration parameters and individual scale-factors for each layer-line were refined. (The observed structure factors were first compared with the calculated values to bring them approximately into an absolute scale.) The weights were calculated using Cruickshank's scheme with $a = 18$ and $c = 0.0087$.

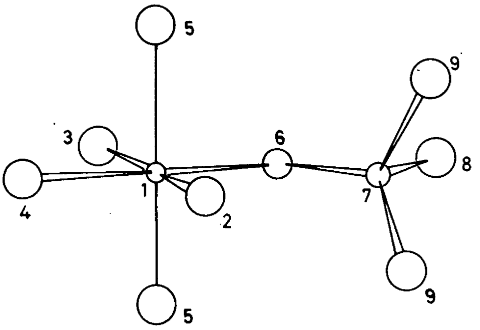

Fig. 1. The structures of $SbCl_5 \cdot POCl_3$, $NbCl_5 \cdot POCl_3$ and $SbCl_5 \cdot (CH_3)_3PO$. Generalized figure.

After nine cycles of refinement all shifts were less than one tenth of the standard deviations and the R-value had decreased from 0.225 to 0.130. The weight-analysis obtained in the last cycle of refinement is given in Table 5, and the final atomic parameters and their e.s.d.'s are given in Table 6. Tables of a calculated and observed $F(h,k,l)$ values can be obtained from this Institute.

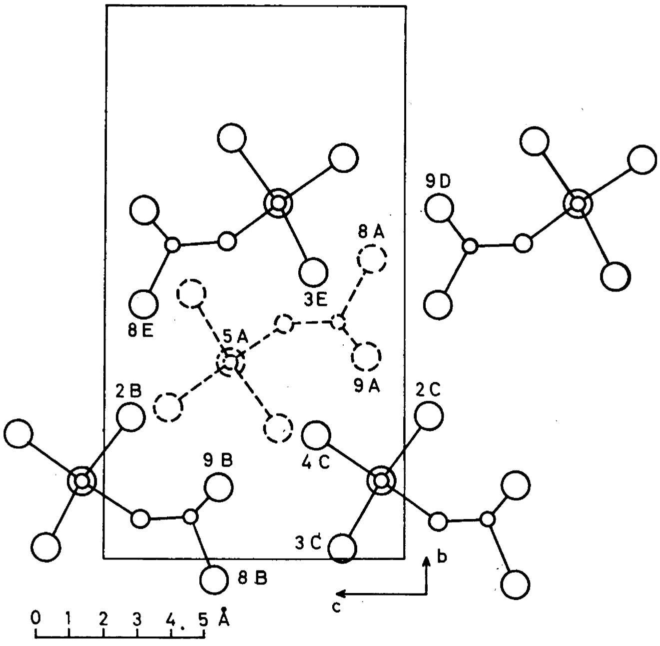

Fig. 2. Projection of the structure along 100, showing packing distances. The dotted molecule is in the plane $x = 1/4$. The other molecules are in $x = 3/4$.

Table 7. Bond lengths and bond angles with their e.s.d. and packing distances in $SbCl_5 \cdot POCl_3$.

Bond length	Distance (Å)	e.s.d.(Å)	Bond length	Distance (Å)	e.s.d.(Å)
Sb(1)–Cl(2)	2.35	0.01	Sb(1)–O(6)	2.17	0.02
Sb(1)–Cl(3)	2.33	0.01	P (7)–O(6)	1.47	0.02
Sb(1)–Cl(4)	2.32	0.01	P (7)–Cl(8)	1.95	0.01
Sb(1)–Cl(5)	2.33	0.01	P (7)–Cl(9)	1.95	0.01

Bond angle	Angle(°)	e.s.d.(°)	Bond angle	Angle(°)	e.s.d.(°)
Cl(2)–Sb(1)–Cl(4)	93.0	0.3	Cl(5)–Sb(1)–O(6)	84.9	0.6
Cl(2)–Sb(1)–Cl(5)	89.3	0.3	Cl(5)–Sb(1)–Cl(5)	169.8	0.4
Cl(2)–Sb(1)–O(6)	86.2	0.6	Sb(1)–O(6)–P(7)	145.0	1.6
Cl(3)–Sb(1)–Cl(4)	95.7	0.4	O(6)–P(7)–Cl(8)	110.9	1.0
Cl(3)–Sb(1)–Cl(5)	89.9	0.3	O(6)–P(7)–Cl(9)	112.5	1.0
Cl(3)–Sb(1)–O(6)	85.1	0.6	Cl(8)–P(7)–Cl(9)	107.4	0.6
Cl(4)–Sb(1)–Cl(5)	95.1	0.3	Cl(9)–P(7)–Cl(9)	105.9	0.6

Packing distances (Å)		Packing distances (Å)	
Cl(2)B–Cl(5)A	4.01	Cl(4)C–Cl(9)A	3.72
Cl(2)B–Cl(8)E	3.41	Cl(4)C–Cl(9)B	3.74
Cl(2)C–Cl(9)A	3.61	Cl(5)A–Cl(5)A	3.41
Cl(3)E–Cl(5)A	3.92	Cl(5)A–Cl(8)E	3.66
Cl(3)C–Cl(8)B	3.91	Cl(5)A–Cl(9)B	3.85
Cl(3)E–Cl(9)A	3.80	Cl(8)A–Cl(9)D	3.75
Cl(4)C–Cl(5)A	3.68		

Two cycles of refinement were then carried out, using weights calculated according to Hughes' weighting-scheme and including 452 accidentally absent reflexions for which $\sin \Theta/\lambda \leq 0.65$. For these reflexions, F_o was set equal to $F_{o,\mathrm{min.}}$ (the value of F obtained from the threshold intensity of the actual layer-line). The quantity $|F_o|-|F_c|$ was set equal to zero in the sums when $|F_c| \leq |F_o|$ and equal to $|F_o|-|F_c|$ when $|F_c| \geq |F_o|$. Only 64 of these reflexions contributed to the sums. All shifts in the parameters were less than one fifth of the e.s.d. The R-value calculated for all 1587 reflexions was 0.185.

DESCRIPTION OF THE CRYSTAL STRUCTURES

The bond lengths and angles with their e.s.d.'s are given together with the packing distances in Tables 7 ($SbCl_5 \cdot POCl_3$), 8 ($SbCl_5 \cdot (CH_3)_3PO$) and 9 ($NbCl_5 \cdot POCl_3$). The symbols refer to the numbers in Figs. 1 and 2 which describe all three isotypic structures.

DISCUSSION OF THE STRUCTURES

1. The differences in the P—O and Sb—O bond lengths of adducts with $POCl_3$ and $(CH_3)_3PO$, reported earlier[2], have been confirmed, and the values for $NbCl_5 \cdot POCl_3$ are consistent.

Table 8. Bond lengths and bond angles with their e.s.d. and packing distances in $SbCl_5\cdot(CH_3)_3PO$.

Bond length	Distance (Å)	e.s.d. (Å)	Bond length	Distance (Å)	e.s.d. (Å)
Sb(1)—Cl(2)	2.34	0.02	Sb(1)—O(6)	1.94	0.04
Sb(1)—Cl(3)	2.35	0.02	P(7)—O(6)	1.56	0.04
Sb(1)—Cl(4)	2.34	0.02	P(7)—C(8)	1.84	0.07
Sb(1)—Cl(5)	2.33	0.02	P(7)—C(9)	1.80	0.08

Bond angle	Angle(°)	e.s.d.(°)	Bond angle	Angle(°)	e.s.d.(°)
Cl(2)—Sb(1)—Cl(4)	90.1	0.6	Cl(5)—Sb(1)—O(6)	87.2	1.0
Cl(2)—Sb(1)—Cl(5)	89.7	0.6	Cl(5)—Sb(1)—Cl(5)	174.3	0.7
Cl(2)—Sb(1)—O(6)	90.7	1.1	Sb(1)—O(6)—P(7)	144.9	2.3
Cl(3)—Sb(1)—Cl(4)	91.3	0.6	O(6)—P(7)—C(8)	108.1	2.7
Cl(3)—Sb(1)—Cl(5)	90.2	0.6	O(6)—P(7)—C(9)	109.9	2.9
Cl(3)—Sb(1)—O(6)	87.9	1.1	C(8)—P(7)—C(9)	111.0	4.0
Cl(4)—Sb(1)—Cl(5)	92.8	0.6	C(9)—P(7)—C(9)	106.9	4.0

Packing distances (Å)		Packing distances (Å)	
Cl(2)B—Cl(5)A	4.01	Cl(4)C—C(9)A	3.78
Cl(2)B—C(8)E	3.60	Cl(4)C—C(9)B	3.79
Cl(2)C—C(9)A	3.74	Cl(5)A—Cl(5)A	3.51
Cl(3)E—Cl(5)A	4.10	Cl(5)A—C(8)E	3.72
Cl(3)C—C(8)B	3.95	Cl(5)A—C(9)B	3.83
Cl(3)E—C(9)A	3.97	C(8)A—C(9)D	4.13
Cl(4)C—Cl(5)A	3.72		

Table 9. Bond lengths and bond angles with their e.s.d. and packing distances in $NbCl_5\cdot POCl_3$.

Bond length	Distance (Å)	e.s.d.(Å)	Bond length	Distance (Å)	e.s.d.(Å)
Nb(1)—Cl(2)	2.35	0.01	Nb(1)—O(6)	2.16	0.02
Nb(1)—Cl(3)	2.29	0.01	P(7)—O(6)	1.45	0.02
Nb(1)—Cl(4)	2.25	0.01	P(7)—Cl(8)	1.95	0.01
Nb(1)—Cl(5)	2.30	0.01	P(7)—Cl(9)	1.94	0.01

Bond angle	Angle(°)	e.s.d.(°)	Bond angle	Angle(°)	e.s.d.(°)
Cl(2)—Nb(1)—Cl(4)	91.9	0.3	Cl(5)—Nb(1)—O(6)	83.7	0.5
Cl(2)—Nb(1)—Cl(5)	88.8	0.3	Cl(5)—Nb(1)—Cl(5)	167.4	0.3
Cl(2)—Nb(1)—O(6)	84.6	0.5	Nb(1)—O(6)—P(7)	148.8	1.2
Cl(3)—Nb(1)—Cl(4)	97.2	0.3	O(6)—P(7)—Cl(8)	111.8	0.8
Cl(3)—Nb(1)—Cl(5)	90.2	0.3	O(6)—P(7)—Cl(9)	112.2	0.8
Cl(3)—Nb(1)—O(6)	86.3	0.6	Cl(8)—P(7)—Cl(9)	107.1	0.5
Cl(4)—Nb(1)—Cl(5)	96.2	0.3	Cl(9)—P(7)—Cl(9)	106.1	0.5

Packing distances (Å)		Packing distances (Å)	
Cl(2)B—Cl(5)A	4.00	Cl(4)C—Cl(9)A	3.69
Cl(2)B—Cl(8)E	3.31	Cl(4)C—Cl(9)B	3.70
Cl(2)C—Cl(9)A	3.54	Cl(5)A—Cl(5)A	3.50
Cl(3)E—Cl(5)A	3.93	Cl(5)A—Cl(8)E	3.63
Cl(3)C—Cl(8)B	3.88	Cl(5)A—Cl(9)B	3.79
Cl(3)E—Cl(9)A	3.79	Cl(8)A—Cl(9)D	3.72
Cl(4)C—Cl(5)A	3.65		

2. The bond angles in the $POCl_3$ molecules differ from regular tetrahedral coordination; the O—P—Cl bond angles are slightly larger and the Cl—P—Cl bond angles slightly smaller than for regular coordination.

3. The deviations from regular octahedral coordination around Sb and Nb will be discussed in another connection [9].

4. The Nb—Cl bond lengths exhibit a surprising variation, far beyond the significance limits. However, the variation, between 2.25 Å and 2.35 Å, may be compared with the values of 2.25 Å and 2.30 Å for the non-bridging Nb—Cl bonds in Nb_2Cl_{10} [10]. Variations of this magnitude have not been reported in $SbCl_5$ adducts. The length of the bridge bonds in Nb_2Cl_{10} is 2.56 Å.

5. In both $POCl_3$ adducts, very short Cl—Cl packing distances are found; the shortest distance is 3.31 Å in the $NbCl_5$ adduct and 3.41 Å in the $SbCl_5$ adduct.

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