recouvrement des nuages π et à une distance moyenne de 3,3 Å. Ces dimères forment des chaînes selon la direction de l'axe c par recouvrement entre les molécules (II) et (I) + c séparées par une distance plus grande de 3,5 Å (Fig. 2). La cohésion cristalline est essentiellement assurée par de multiples interactions de van der Waals.

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Acta Cryst. (1989). **C45**, 1986–1988

Structure of Hexaamminecobalt(III) Chloride Hexachlorotellurate(IV) 1·64-Water

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Abstract. $[Co(NH_3)_6]Cl(TeCl_6).1·64H_2O$, M_r = 566·43, triclinic, $P\overline{1}$, a = 7·808 (4), b = 10·932 (5), c = 11·094 (7) Å, α = 100·98 (6), β = 89·8 (1), γ = 105·01 (3)°, V = 896·9 Å^3, Z = 2, D_x = 2·122 g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 33·83 cm^{-1}, $F(000)$ = 556, T = 293 K, R = 0·0412 for 2113 reflexions ($F > 3\sigma_F$). The $TeCl_6^{2-}$ anions show a distorted octahedral structure with three short Te—Cl distances (mean 2·46 Å) and three longer Te—Cl distances (mean 2·61 Å). The idealized point symmetry of the anion is $3m$. Due to the noncentrosymmetric crystal field this statical distortion conforms to the symmetry rules for the stereochemistry of the lone-pair electrons in AX_6E systems.

Experimental. TeO_2 was dissolved in concentrated hydrochloric acid. Upon addition of a solution of $[Co(NH_3)_6]Cl_3$ (prepared according to Brauer, 1981), the following reaction was expected:

$$2[Co[NH_3)_6]^{3+} + 3TeCl_6^{2-} \rightarrow [Co(NH_3)_6]_2(TeCl_6)_3.$$

With a twofold excess of TeO_2 single crystals of orange color grew from the clear solution. An energy dispersive X-ray analysis yielded a Te/Co ratio of 1:1 and a Te/Cl ratio of about 1:8. These results contradicted the above reaction.

The crystal used for the data collection was placed in a capillary in order to protect it from moisture. The instability of the crystals (due to a loss of crystal water) prevented a longer-lasting lattice examination by film methods. The intensity variation of standard reflexions during the diffractometer measurements

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Table 1. *Summary of data collection and structure refinement*

Crystal shape	Faces {100}, {010}, {110}
Crystal volume (mm^3)	1.947×10^{-2}
Diffractometer	Siemens AED-2, graphite-monochromated Mo $K\alpha$ radiation
Determination of cell parameters:	
number of reflexions used	21
θ range (°)	9·0–20·8
Intensity data collection:	
max. $\sin\theta/\lambda$(Å^{-1})	0·5392
range of h,k,l	$\bar{8},\bar{11},0$ to 8,11,11
scan mode	ω
min. scan angle (°)	1·6
max. scan time (s)	36
standard reflexions	$\bar{1}\bar{4}1$, $14\bar{1}$
intensity variation	< 4·2%
measured reflexions	2499
unique reflexions	2312
observed reflexions	2113 with $F > 3\sigma_F$
R_{int}	0·025
Numerical absorption correction:	
min./max. transmission	0·5/0·64
Structure refinement (on F):	
R, wR [$w = 1/\sigma^2(F_o)$]	0·0412, 0·0419
S	4·32
number of parameters	155
max. final shift/e.s.d.	0·001
min./max. final $\Delta\rho$ excursions (e Å^{-3})	$-1.169 \le \Delta \le 0.941$

Table 2. *Positional and equivalent isotropic displacement parameters for atoms in* $[Co(NH_3)_6]Cl(TeCl_6).1{\cdot}64H_2O$

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{A}_{ij}$, where $\mathbf{A}_{ij}$ is the dot product of the ith and jth direct-space unit-cell vectors.

	Site	x	y	z	U_{eq}(Å^2)
Te	2(i)	0·06967 (6)	−0·13799 (5)	0·22260 (5)	0·0293
Cl(1)	2(i)	0·3214 (3)	0·9098 (2)	0·0680 (2)	0·0478
Cl(2)	2(i)	0·0937 (3)	0·1053 (2)	0·2747 (2)	0·0459
Cl(3)	2(i)	0·1574 (4)	0·1671 (3)	0·9373 (3)	0·0667
Cl(4)	2(i)	0·1585 (4)	0·1784 (3)	0·6266 (3)	0·0697
Cl(5)	2(i)	0·3243 (3)	0·9040 (3)	0·3941 (3)	0·0576
Cl(6)	2(i)	0·0508 (4)	0·6276 (3)	0·1706 (4)	0·0884
Co	2(i)	0·3183 (1)	0·7224 (1)	0·6978 (1)	0·0283
N(1)	2(i)	0·3357 (8)	0·9096 (6)	0·7290 (7)	0·0366
N(2)	2(i)	0·3011 (9)	0·5352 (6)	0·6664 (7)	0·0371
N(3)	2(i)	0·1319 (9)	0·6957 (6)	0·8201 (6)	0·0361
N(4)	2(i)	0·1358 (8)	0·6926 (6)	0·5644 (6)	0·0345
N(5)	2(i)	0·4963 (8)	0·2508 (7)	0·1718 (6)	0·0379
N(6)	2(i)	0·4981 (8)	0·2552 (7)	0·4261 (7)	0·0375
Cl(7)	2(i)	0·2641 (3)	0·4594 (2)	0·3653 (2)	0·0436
O(1)	2(i)	0·340 (2)	0·545 (1)	0·957 (1)	0·1408
O(2)*	2(i)	−0·506 (2)	−0·365 (2)	0·182 (2)	0·1185

* Site-occupancy factor 0·64 (3).

was a function of the decreasing quality of the crystal. A summary of data collection and structure refinement is given in Table 1.

Table 3. *Bond lengths* (Å) *and bond angles* (°)

Distances between anions	
Cl(1)—Cl(1)	3·493 (3)
—Cl(3)	3·884 (3)
Cl(2)—Cl(7)	3·691 (2)
—Cl(4)	3·841 (4)
—Cl(3)	3·936 (4)
Cl(4)—Cl(5)	4·046 (4)
Cl(5)—Cl(5)	3·555 (4)
Cl(6)—Cl(7)	3·734 (4)
—Cl(6)	4·194 (6)
Cation—anion distances	
N(1)—Cl(2)	3·314 (5)
—Cl(5)	3·361 (6)
—Cl(1)	3·428 (6)
N(2)—Cl(7)	3·280 (8)
—Cl(7)	3·398 (6)
N(3)—Cl(1)	3·319 (6)
—Cl(2)	3·437 (6)
N(4)—Cl(7)	3·303 (5)
—Cl(5)	3·310 (6)
—Cl(7)	3·398 (6)
—Cl(2)	3·436 (6)
N(5)—Cl(1)	3·417 (6)
—Cl(2)	3·434 (5)
N(6)—Cl(7)	3·387 (6)
—Cl(5)	3·394 (7)
—Cl(2)	3·433 (6)
Distances within the cation	
Co—N(1)	1·978 (5)
—N(2)	1·978 (5)
—N(3)	1·990 (6)
—N(4)	1·979 (6)
—N(5)	1·977 (6)
—N(6)	1·984 (6)
Distances to O atoms	
O(1)—O(2)	2·66 (2)
—O(2)	2·84 (2)
—N(5)	2·90 (1)
—O(1)	3·13 (1)
—N(3)	3·17 (1)
—N(2)	3·22 (2)
—Cl(6)	3·30 (1)
—Cl(6)	3·42 (1)
O(2)—Cl(7)	3·27 (2)
—Cl(4)	3·40 (2)
—N(2)	3·41 (2)
—Cl(6)	3·44 (1)
—Cl(3)	3·45 (1)

Distances within the anion	
Te—Cl(1)	2·620 (2)
—Cl(2)	2·569 (2)
—Cl(3)	2·430 (3)
—Cl(4)	2·454 (3)
—Cl(5)	2·647 (3)
—Cl(6)	2·483 (3)
Cl(1)—Cl(2)	3·614 (3)
—Cl(3)	3·611 (3)
—Cl(5)	3·631 (4)
—Cl(6)	3·636 (3)
Cl(2)—Cl(3)	3·547 (3)
—Cl(5)	3·608 (3)
—Cl(4)	3·579 (3)
Cl(3)—Cl(6)	3·470 (4)
—Cl(4)	3·472 (5)
Cl(5)—Cl(6)	3·699 (4)
—Cl(4)	3·639 (3)
Cl(6)—Cl(4)	3·490 (4)
Angles within the anion	
Cl(1)—Te—Cl(2)	88·28 (6)
—Cl(3)	91·22 (8)
—Cl(5)	87·16 (7)
—Cl(6)	90·86 (9)
—Cl(4)	177·97 (8)
Cl(2)—Te—Cl(3)	90·36 (8)
—Cl(5)	87·53 (7)
—Cl(4)	90·86 (8)
—Cl(6)	179·12 (8)
Cl(3)—Te—Cl(6)	89·9 (1)
—Cl(4)	90·63 (9)
—Cl(5)	177·38 (8)
Cl(5)—Te—Cl(6)	92·2 (1)
—Cl(4)	90·96 (9)
Cl(6)—Te—Cl(4)	90·0 (1)
Angles within the cation	
N(1)—Co—N(3)	90·8 (2)
—N(4)	90·2 (2)
—N(5)	90·9 (3)
—N(6)	90·4 (3)
—N(2)	180·0 (4)
N(3)—Co—N(4)	91·0 (2)
—N(5)	90·1 (2)
—N(2)	89·3 (2)
—N(6)	178·7 (2)
N(4)—Co—N(6)	88·5 (2)
—N(2)	89·8 (3)
—N(5)	178·5 (2)
N(5)—Co—N(6)	90·5 (2)
—N(2)	89·1 (3)
N(6)—Co—N(2)	89·5 (3)

The initial position of Te was obtained from the Patterson function synthesis. Subsequent ΔF synthesis yielded the positions of the Co, Cl, N and O atoms (two sites for water molecules had to be considered). The final composition showed a mixed chloride tellurate $[Co(NH_3)_6]Cl(TeCl)_6.(2-x)H_2O$; a site-occupancy refinement of the O(2) atom resulted in the value 0·64 (3) indicating an x of 0·36. Hydrogen sites could not be found in the final ΔF synthesis. Scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974). The program used was *STRUCSY* from the diffractometer software package. Atomic parameters are given in Table 2,* bond lengths and some important bond angles in Table 3. A projection of the structure is given in Fig. 1. According to Shannon (1976), octahedral distortion is defined by $\Delta = (1/6)\sum[(R_i - \bar{R})/\bar{R}]^2$, where $\bar{R}$ = average bond length and R_i = an individual bond length; for the $TeCl_6^{2-}$ anion in the described structure, Δ is calculated to be 106.97×10^{-5}.

Related literature. Symmetry rules for the stereochemistry of the lone-pair electrons in AX_6E systems are discussed by Abriel (1986), Abriel &

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* Lists of anisotropic displacement parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52090 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

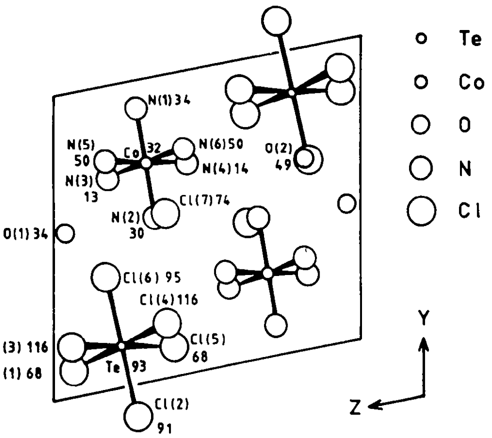

Fig. 1. Projection of the crystal structure of [Co(NH$_3$)$_6$]Cl(TeCl$_6$).$(2-x)$H$_2$O along [100]. Heights of atoms are indicated as percentages of the *a* length. The unit cell is outlined.

Zehnder (1987) and Abriel (1987). The last paper gives basic structural data for all compounds containing AX_6^{2-} ions (A = Se, Te; X = Cl, Br, I) known to date including Δ values for octahedral distortion. Extended symmetry rules considering SbX_6^{3-} and BiX_6^{3-} species are given by du Bois & Abriel (1988). Relevant interatomic distances for hydrogen-bonding contacts [here O(1)···N(5) and O(1)···O(2)] are listed by Wells (1986).

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Structure of 2,3,4-Tris(diethyl dithiophosphato-*S*,*S'*)-1-iodo-2,3-μ_2-methanoato-4-(pyridine)-tetrakis(μ_3-sulfido)-copper(I)trimolybdenum(IV)

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Abstract. [CuMo$_3$S$_4$(I)(CHO$_2$)(C$_4$H$_{10}$O$_2$PS$_2$)$_3$(C$_5$H$_5$N)], M_r = 1286, triclinic, $P\bar{1}$, a = 11·460 (8), b = 14·237 (8), c = 15·046 (8) Å, α = 112·35 (5), β = 90·09 (5), γ = 111·77 (5)°, V = 2079·3 Å^3, Z = 2, D_x = 2·05 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ_m = 27·4 cm^{-1}, F(000) = 1256, R = 0·058 for 3372 observed unique reflections $I \geq 5\sigma(I)$. The structure contains discrete molecules with a central CuMo$_3$S$_4$ distorted cubic cluster (mean Mo—S 2·329, Cu—S 2·280, Mo···Mo 2·744, Mo···Cu 2·856 Å). Octahedral coordination at each Mo is completed by two S atoms of bidentate chelating diethyl dithiophosphate ligands (mean Mo—S 2·531 Å), and by a terminal pyridine ligand for one Mo (Mo—N 2·368 Å) and an HCOO$^-$ bridging ligand for the other two Mo (mean Mo—O 2·187 Å); an I$^-$ ligand completes tetrahedral geometry at the Cu atom.

Experimental. The title compound was prepared by the method described by Wu, Lu, Zhu, Wu & Lu (1987). The crystal measured 0·50 × 0·25 × 1·00 mm. Data were collected using a CAD-4 κ-geometry diffractometer using Mo $K\alpha$ radiation at *ca* 296 K. $\omega/2\theta$ scan, scan speed varied from 2 to 7° min^{-1} (in ω), the scan width was $(0·50 + 0·35\tan\theta)°$. Cell constants were obtained by least-squares analysis on 25 diffraction maxima ($26 < 2\theta < 27°$). The intensities were corrected for absorption using empirical scan data (maximum and minimum transmission factors 1·06 and 0·86, respectively), and Lorentz and polarization factors to give a total of 7703 intensities, to a maximum 2θ of 50° ($0 \leq h \leq 13$, $-16 \leq k \leq 15$, $-17 \leq l \leq 17$). Max. $(\sin\theta)/\lambda$ = 0·59 Å^{-1}, 4331 reflections with $I < 5\sigma(I)$ are considered unobserved. 3372 reflections with $I \geq 5\sigma(I)$ used in the refinement. Three standard reflections were measured periodically, no random deviations.

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