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A New Pentachlorotellurate(IV): catena-Poly[hexakis(acetonitrile)aluminium tris-[tetrachlorotellurate(IV)- μ -chloro] acetonitrile]

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Abstract

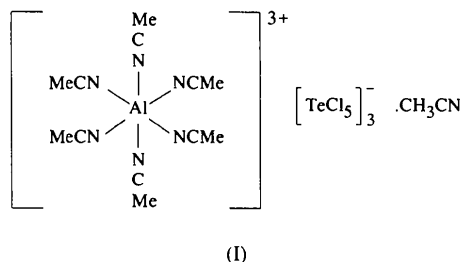
In the title compound, [Al(CH₃CN)₆][TeCl₅]₃.CH₃CN, the polymeric anions form infinite helical chains from apex-sharing distorted octahedral TeCl₆ units. These anionic chains are stabilized by [Al(CH₃CN)₆]³⁺ counterions.

Comment

From tetra- to hexahalide, all tellurium(IV) halide structures are based on octahedrally or pseudo-octahedrally coordinated tellurium (Hazell, 1966; Adams & Lock, 1967; Collins & Webster, 1972; Krebs, Bonmann & Gretenkord, 1992). TeCl₄ has been described as TeCl₃Cl⁻, with Te and Cl atoms arranged within a three-dimensional cubic array to form distorted Te-centred octahedra displaying three short and three long Te—Cl bonds (Buss & Krebs, 1971). This structural arrangement has also been encountered in TeCl₃X moieties with X = AlCl₄ (Krebs, Buss & Altena, 1971), SbF₆, AsF₆ (Christian, Collins, Gillespie & Sawyer, 1985), ClO₄ (Favier, 1991), MoCl₄ (Beck, 1991), etc. On the other hand, A₂TeCl₆ salts (where A = K, Rb or NH₄) contain discrete and regular octahedral hexa-

chlorotellurate(IV) anions (Webster & Collins, 1973; Armstrong, Dufort & Powell, 1991).

Between the three-dimensional condensed tellurium tetrachlorides and the discrete molecular species of tellurium hexachloride, intermediate polymeric moieties are formed, depending on the number of Cl atoms per Te atom. In [H₉O₄]_n[Te₂Cl₉]_n, with a Cl/Te ratio of 4.5, the Te₂Cl₁₀ dimeric units (two TeCl₆ octahedra condensed by edge-sharing) polymerize into infinite [Te₂Cl₉]_nⁿ⁻ zigzag chains through Te—Cl—Te bridges (Krebs, Bonmann & Gretenkord, 1992). [PCL₄][TeCl₅] contains distorted octahedral TeCl₆ units fused by corner-sharing within an infinite [TeCl₅]_nⁿ⁻ chain (Collins & Webster, 1972; Krebs, Buss & Berger, 1973). This assertion is corroborated by the structure of the title compound, (I), which mainly consists of an infinite pseudo-helical anionic chain of [TeCl₅]_nⁿ⁻ stabilized by *n*/3 discrete [Al(CH₃CN)₆]³⁺ units.



The structure also contains free molecules of acetonitrile. The helical unit comprises three non-equivalent Te atoms in distorted octahedral environments. Each

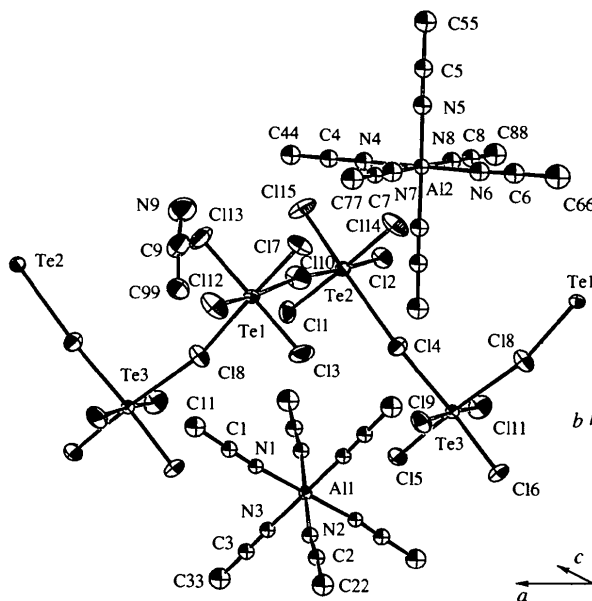


Fig. 1. The molecular structure of the title compound. H atoms have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level.

Te atom is surrounded by four terminal Cl atoms (one short Te—Cl bond of 2.33 Å and three longer ones at around 2.44 Å). Two bridging Cl atoms lie in the range 2.66–2.99 Å. Except for the Te— μ_2 -Cl bond of 2.99 Å, which is relatively long, these distances are close to those found in $[\text{PCL}_4][\text{TeCl}_5]$ or $[\text{H}_9\text{O}_4]_n[\text{Te}_2\text{Cl}_9]_n$. Furthermore, similar angular distortions are observed in these compounds. Two non-equivalent $[\text{Al}(\text{CH}_3\text{CN})_6]^{3+}$ counterions sit inside channels delimited by four $[\text{TeCl}_5]_n^-$ units. The Al atoms, Al1 and Al2, lying in sites 4a and 4c, respectively, are in slightly distorted octahedral environments, as are Mn (Weller, Mai & Dehnicke, 1996), Fe (Stork-Blaisse, Verschuur & Romers, 1972), Co and Ni (Veith, Gödicke & Huch, 1989) in related cations. The N—Al—N angles are all close to 90°, while Al—N bond lengths range from 1.92 (1) to 1.99 (2) Å.

Experimental

The serendipitous synthesis of the title compound occurred during the course of an electrochemical synthesis of mixed tungsten(IV)/tellurium(IV) chloride complexes in acetonitrile. The electrochemical cell was operated at 300 $\mu\text{A cm}^{-2}$ with platinum electrodes with an equimolar concentrated solution of WCl_6 and TeCl_4 in CH_3CN . After a few days, long yellow needles ($10 \times 0.5 \times 0.5$ mm) appeared in abundance on both electrodes. Qualitative electron microprobe analysis revealed the presence of Te, Cl and surprisingly of Al in the approximate ratio 3/15/1. The Al in the crystals appears to come from partial electrolytic dissolution of the Al-alloy electrode supports. Reagents as well as products were handled in a glove box under dry argon or on vacuum line (1.33 Pa).

Crystal data

$[\text{Al}(\text{C}_2\text{H}_3\text{N})_6][\text{TeCl}_5]_3 \cdot \text{C}_2\text{H}_3\text{N}$

$M_r = 1228.91$

Orthorhombic

Pnma

$a = 19.923$ (5) Å

$b = 41.216$ (9) Å

$c = 10.068$ (3) Å

$V = 8267$ (4) Å³

$Z = 8$

$D_x = 1.975$ Mg m⁻³

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71070$ Å

Cell parameters from 23 reflections

$\theta = 12.23$ – 22.14°

$\mu = 3.115$ mm⁻¹

$T = 293$ (2) K

Needle

$0.48 \times 0.26 \times 0.10$ mm

Yellow

Data collection

Enraf–Nonius CAD-4

diffractometer

ω – $\frac{2}{3}\theta$ scans

Absorption correction:

numerical (Sheldrick, 1976)

$T_{\min} = 0.125$, $T_{\max} = 0.632$

6116 measured reflections

6116 independent reflections

2320 reflections with

$I > 3\sigma(I)$

$\theta_{\max} = 23.0^\circ$

$h = 0 \rightarrow 21$

$k = 0 \rightarrow 44$

$l = 0 \rightarrow 10$

3 standard reflections

frequency: 60 min

intensity decay: none

Refinement

Refinement on F^2

$R(F) = 0.038$

$wR(F^2) = 0.079$

$S = 1.013$

2316 reflections

286 parameters

H atoms: see below

$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 1.04$ e Å⁻³

$\Delta\rho_{\min} = -0.51$ e Å⁻³

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

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

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Te1	0.95452 (5)	0.64674 (2)	0.16984 (13)	0.0443 (3)
Te2	0.73447 (5)	0.65638 (2)	0.83355 (13)	0.0427 (3)
Te3	0.83336 (5)	0.55391 (2)	0.51049 (12)	0.0455 (3)
Al1	0	0	0	0.038 (2)
Al2	0.0222 (4)	1/4	0.3380 (9)	0.054 (2)
Cl1	0.6707 (2)	0.61914 (12)	0.9835 (5)	0.076 (2)
Cl2	0.6404 (2)	0.65998 (11)	0.6874 (5)	0.0702 (15)
Cl3	1.0089 (3)	0.60675 (12)	0.0259 (7)	0.135 (3)
Cl4	0.7908 (2)	0.60079 (10)	0.6719 (5)	0.0686 (14)
Cl5	0.7910 (3)	0.51685 (11)	0.6637 (6)	0.088 (2)
Cl6	0.8718 (3)	0.50996 (11)	0.3734 (5)	0.099 (2)
Cl7	1.0047 (3)	0.68905 (12)	0.0386 (6)	0.096 (2)
Cl8	0.9011 (2)	0.59775 (12)	0.3134 (6)	0.085 (2)
Cl9	0.7229 (2)	0.55150 (13)	0.3971 (6)	0.094 (2)
Cl10	0.8371 (3)	0.65511 (12)	0.9973 (6)	0.104 (2)
Cl11	0.9404 (2)	0.55175 (12)	0.6328 (6)	0.087 (2)
Cl12	1.0484 (2)	0.64465 (14)	0.3090 (7)	0.108 (2)
Cl13	0.9100 (3)	0.68833 (13)	0.3202 (6)	0.122 (2)
Cl14	0.7959 (2)	0.69438 (13)	0.6906 (6)	0.103 (2)
Cl15	0.6928 (3)	0.69997 (13)	0.9528 (6)	0.108 (2)
N1	−0.0300 (5)	0.0214 (3)	−0.1642 (14)	0.041 (3)
C1	−0.0471 (8)	0.0351 (4)	−0.2519 (17)	0.049 (5)
C11	−0.0720 (8)	0.0531 (4)	−0.3654 (18)	0.078 (6)
N2	−0.0796 (6)	−0.0276 (3)	−0.0039 (13)	0.046 (4)
C2	−0.1267 (8)	−0.0417 (4)	0.0065 (18)	0.052 (5)
C22	−0.1893 (8)	−0.0594 (4)	0.0116 (19)	0.089 (7)
N3	0.0502 (6)	−0.0313 (3)	−0.1008 (13)	0.043 (4)
C3	0.0819 (7)	−0.0496 (4)	−0.1566 (18)	0.047 (5)
C33	0.1191 (8)	−0.0731 (4)	−0.2271 (19)	0.084 (7)
N4	0.0781 (9)	1/4	0.175 (2)	0.062 (6)
C4	0.1109 (11)	1/4	0.075 (3)	0.058 (8)
C44	0.1512 (11)	1/4	−0.033 (2)	0.071 (8)
N5	0.0206 (6)	0.2972 (3)	0.3350 (16)	0.063 (4)
C5	0.0253 (8)	0.3247 (4)	0.3365 (19)	0.060 (5)
C55	0.0359 (8)	0.3599 (4)	0.341 (2)	0.088 (6)
N6	−0.0327 (9)	1/4	0.501 (2)	0.070 (7)
C6	−0.0522 (14)	1/4	0.603 (3)	0.078 (9)
C66	−0.0885 (14)	1/4	0.725 (3)	0.118 (13)
N7	−0.0583 (11)	1/4	0.226 (2)	0.073 (7)
C7	−0.1052 (11)	1/4	0.162 (3)	0.050 (7)
C77	−0.1599 (12)	1/4	0.077 (3)	0.092 (10)
N8	0.1023 (10)	1/4	0.450 (2)	0.059 (6)
C8	0.1457 (12)	1/4	0.521 (3)	0.056 (7)
C88	0.2005 (12)	1/4	0.608 (3)	0.093 (10)
N9	0.3045 (11)	0.1978 (5)	0.677 (3)	0.147 (9)
C9	0.2917 (11)	0.1706 (6)	0.664 (3)	0.103 (9)
C99	0.2737 (10)	0.1387 (4)	0.663 (2)	0.109 (8)

Table 2. Selected geometric parameters (Å, °)

Te1—Cl3	2.447 (5)	Al2—N7	1.96 (2)
Te1—Cl7	2.405 (5)	Al2—N8	1.95 (2)
Te1—Cl8	2.702 (5)	N1—C1	1.10 (2)
Te1—Cl12	2.339 (5)	C1—Cl11	1.45 (2)
Te1—Cl13	2.452 (5)	N2—C2	1.11 (2)
Te2—Cl11	2.500 (5)	C2—C22	1.45 (2)

Te2—C12	2.388 (5)	N3—C3	1.13 (2)
Te2—C110	2.628 (5)	C3—C33	1.41 (2)
Te2—C114	2.454 (5)	N4—C4	1.20 (3)
Te2—C115	2.315 (5)	C4—C44	1.35 (3)
Te3—C14	2.663 (5)	N5—C5	1.14 (2)
Te3—C15	2.329 (5)	C5—C55	1.46 (2)
Te3—C16	2.403 (5)	N6—C6	1.09 (3)
Te3—C19	2.482 (5)	C6—C66	1.43 (4)
Te3—C111	2.464 (5)	N7—C7	1.14 (3)
Al1—N1	1.967 (14)	C7—C77	1.38 (3)
Al1—N2	1.952 (12)	N8—C8	1.12 (3)
Al1—N3	1.922 (12)	C8—C88	1.40 (3)
Al2—N4	1.99 (2)	N9—C9	1.15 (2)
Al2—N5	1.947 (13)	C9—C99	1.37 (2)
Al2—N6	1.97 (2)		
C13—Te1—C18	89.3 (2)	C15—Te3—C111	87.6 (2)
C17—Te1—C113	89.0 (2)	C15—Te3—C14	87.5 (2)
C112—Te1—C13	88.6 (2)	C16—Te3—C19	89.3 (2)
C112—Te1—C17	91.3 (2)	C16—Te3—C111	89.1 (2)
C112—Te1—C18	88.1 (2)	C19—Te3—C14	91.6 (2)
C112—Te1—C113	86.9 (2)	N2—Al1—N1	89.9 (5)
C113—Te1—C18	92.9 (2)	N3—Al1—N1	90.9 (5)
C11—Te2—C110	90.3 (2)	N3—Al1—N2	91.2 (5)
C12—Te2—C11	90.6 (2)	N5—Al2—N4	89.8 (5)
C12—Te2—C114	89.4 (2)	N5—Al2—N6	90.2 (5)
C115—Te2—C11	88.9 (2)	N5—Al2—N7	88.7 (5)
C115—Te2—C12	89.4 (2)	N5—Al2—N8	91.3 (5)
C115—Te2—C110	88.2 (2)	N7—Al2—N6	91.4 (9)
C115—Te2—C114	89.3 (2)	N8—Al2—N4	91.1 (8)
C15—Te3—C16	90.1 (2)	N8—Al2—N6	88.4 (9)
C15—Te3—C19	87.5 (2)		

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A Copper(II) Complex of a New Chiral Tridentate Imidazoline Ligand

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Abstract

The title compound, {2,6-bis[(4*R*,5*R*)-1,3-dimethyl-4,5-diphenylimidazolidin-2-yl-*N*¹]pyridine-*N*}chloro-copper(II) hexafluorophosphate 0.5-methanol solvate, [CuCl(C₃₉H₄₁N₅)]PF₆·0.5CH₃OH, is the first example of a metal complex containing the new chiral ligand, 2,6-bis[(4*R*,5*R*)-1,3-dimethyl-4,5-diphenylimidazolidin-2-yl]pyridine. Complexation of the ligand to the Cu atom results in only a slight distortion from idealized C₂ symmetry in the crystal in spite of its less symmetrical environment.

Comment

The compound 2,6-bis[(4*R*,5*R*)-1,3-dimethyl-4,5-diphenylimidazolidin-2-yl]pyridine has been prepared in an enantiomerically pure form as part of a study to evaluate its properties as a chiral ligand for asymmetric catalysis (Pfaltz, 1996). The structure of a copper complex, (I), has been determined in order to establish the geometry of the ligand when it is coordinated to the metal.

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1985). Full-matrix least-squares refinements made by the *SHELXL93* program (Sheldrick, 1993) were carried out using anisotropic displacement parameters for Te, Al, Cl atoms and for C and N atoms of the free solvent molecule. H atoms were included in idealized positions with displacement parameters fixed to 1.2 times those of the C atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: own program. Molecular graphics: *ZORTEP* (Zsolnai & Pritzkow, 1994).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1301). Services for accessing these data are described at the back of the journal.

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