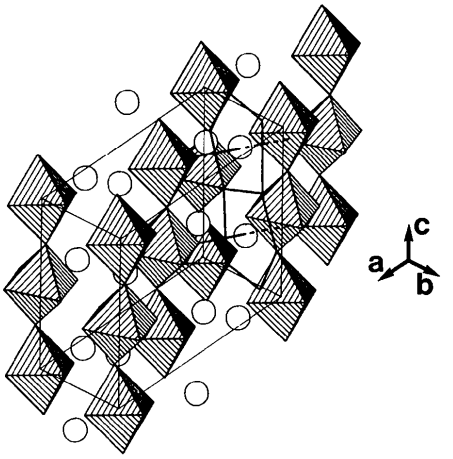

Fig. 1. Perspective view of chains of *trans*-linked AlF_6 octahedra and of CaF_7 polyhedra in α-$CaAlF_5$. Ca ions are represented as open circles.

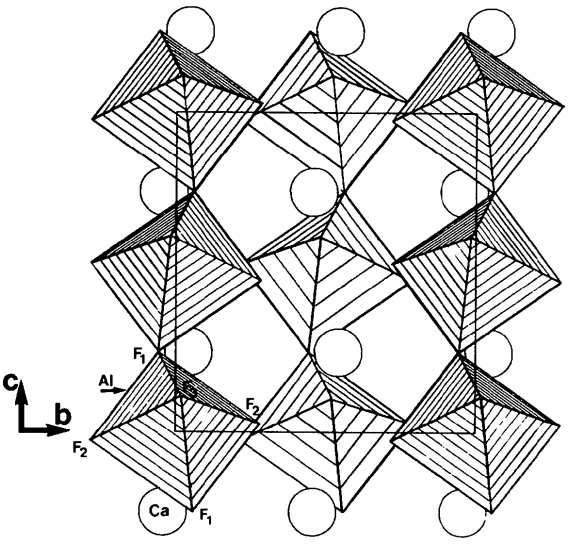

Fig. 2. (100) projection of α-$CaAlF_5$.

Al angle is bent to 157·5° (Fig. 2). Ca atoms exhibit a pentagonal bipyramidal coordination which is formed by edge-sharing chains of CaF_7 polyhedra running along [001] (Fig. 1).

As claimed many years ago, α-$CaAlF_5$ is isotypic with $CaCrF_5$. Our refinement confirms the results of Kun Wun & Brown (1973) [refinement of $CaCrF_5$ in $C2/c$ from the data given by Dumora, Von der Mühll & Ravez (1971) (non-centrosymmetric space group Cc) but with better agreement for the reliability factor.

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Structure of *catena*-Poly{bis[4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedionato-κ^2O,O']copper-μ-(4,4′-bipyridine)-$\kappa N:\kappa N'$}–*N*,*N*-dimethylformamide (1/2)

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Abstract. $[Cu(C_8H_4F_3O_2S)_2(C_{10}H_8N_2)].2C_3H_7NO$, $M_r = 808·25$, triclinic, $P\bar{1}$, $a = 9·637$ (2), $b = 9·820$ (2), $c = 11·505$ (2) Å, $\alpha = 73·40$ (1), $\beta = 65·41$ (1), $\gamma = 69·29$ (1)°, $V = 913·76$ (3) Å^3, $Z = 1$, $D_x = 1·469$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 7·82$ cm^{-1}, $F(000) = 413$, $T = 293$ K, $R = 0·064$, wR

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal factors* ($Å^2 \times 10^3$)

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

	x	y	z	U_{eq}
Cu	0	0	0	39 (1)
S	3101 (2)	−4964 (2)	163 (3)	73 (1)
F1	−4720 (6)	−2560 (6)	2650 (8)	139 (4)
F2	−5188 (6)	−577 (7)	1431 (7)	126 (4)
F3	−4892 (8)	−626 (10)	3138 (8)	158 (5)
N1	−9 (7)	−95 (6)	1937 (6)	54 (3)
N2*	6861 (14)	6250 (10)	5519 (9)	107 (6)
O1	853 (5)	−2176 (4)	174 (4)	48 (2)
O2	−2316 (5)	−223 (5)	869 (5)	57 (2)
O3*	7431 (11)	4308 (8)	4590 (9)	137 (6)
C1	3399 (10)	−6808 (8)	729 (9)	71 (5)
C2	2070 (10)	−7180 (8)	1474 (9)	70 (4)
C3	694 (9)	−5978 (7)	1641 (8)	56 (4)
C4	1090 (8)	−4653 (7)	967 (7)	45 (3)
C5	102 (7)	−3132 (7)	860 (6)	42 (3)
C6	−1524 (8)	−2842 (7)	1488 (7)	52 (3)
C7	−2575 (8)	−1443 (7)	1430 (7)	48 (3)
C8	−4351 (9)	−1328 (8)	2185 (9)	73 (4)
C9	1093 (9)	−1055 (8)	2400 (9)	64 (4)
C10	1134 (9)	−1064 (8)	3594 (8)	63 (4)
C11	−8 (8)	−22 (7)	4372 (7)	50 (3)
C12	−1152 (9)	976 (8)	3867 (9)	67 (4)
C13	−1118 (9)	906 (9)	2667 (9)	67 (4)
C14*	7003 (42)	5273 (17)	4950 (20)	385 (26)
C15*	7886 (32)	7117 (35)	5229 (22)	299 (24)
C16*	5354 (25)	6590 (36)	6536 (21)	291 (22)

* N2, O3, C14, C15 and C16 are the atoms of a DMF molecule.

= 0·064 for 2956 observed [$I \geq 3\sigma(I)$] reflections. The Cu^{II} complex has a chain structure with a repeated unit of bis[4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedionato-κ^2O,O']copper-μ-(4,4′-bipyridine)-$\kappa N:\kappa N'$. Each copper(II) ion has a slightly distorted octahedral environment, the basal plane is composed of O atoms of two 4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedionato-O,O' groups, and the axial positions are occupied by two N atoms of two 4,4′-bipyridine moieties, respectively.

Experimental. The complex was prepared by slow addition of 4,4′-bipyridine (1 mmol) in ethanol (20 ml) into a solution of bis[4,4,4-trifluoro-1-(2-thienyl)-1,3-butanedionato-O,O']copper(II) (2 mmol) in ethanol (30 ml) yielding a green solution. The mixture was stirred and refluxed for half an hour, green crystals were obtained.

Tabular single crystals were grown from a DMF solution of the complex at room temperature. Crystal 0·35 × 0·40 × 0·55 mm, *R3M/E* diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; cell parameters from 25 reflections in θ range 3·5–12°; data collected by ω–2θ scans in θ range 1–23·5°. h 0 to 11, k −12 to 12, l −13 to 13; 5408 measured reflections, 2956 with $I \geq 3\sigma(I)$, $R_{int} = 0{\cdot}012$; Lorentz–polarization correction, absorption correction not applied; three standard reflections monitored every 200 reflections, no decay. Cu atom located from Patterson syntheses, and light atoms found with successive Fourier syntheses. Refinement (on F) with 261 parameters performed by block-diagonal least-squares methods, using anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms; final $R = 0{\cdot}064$, $wR = 0{\cdot}064$, $S = 1{\cdot}10$, max. $\Delta/\sigma = 0{\cdot}082$, $-0{\cdot}47 < \Delta\rho < 0{\cdot}63$ e $Å^{-3}$; $w = 1/[\sigma(F) + (gF^2)^2]$, with $g = 0{\cdot}0006$. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations with *SHELXTL* (Sheldrick, 1983) on an Eclipse S/140 computer. Fig. 1 is a view of the asymmetric unit in the complex prepared using *SHELXTL* and Fig. 2 shows the molecular packing. Final atomic coordinates are presented in Table 1* and selected bond lengths and angles in Table 2.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

Cu—N1	2·201 (8)	Cu—O1	1·983 (4)
Cu—O2	2·096 (5)	Cu—N1*a*	2·201 (8)
Cu—O1*a*	1·983 (4)	Cu—O2*a*	2·096 (5)
S—C1	1·703 (8)	S—C4	1·719 (6)
F1—C8	1·285 (11)	F2—C8	1·321 (13)
F3—C8	1·285 (14)	N1—C9	1·333 (11)
N1—C13	1·340 (10)	N2—C14	1·249 (30)
N2—C15	1·404 (40)	N2—C16	1·434 (21)
O1—C5	1·268 (8)	O2—C7	1·245 (8)
O3—C14	1·023 (21)	C1—C2	1·321 (12)
C2—C3	1·414 (9)	C3—C4	1·403 (10)
C4—C5	1·463 (8)	C5—C6	1·385 (9)
C6—C7	1·392 (8)	C7—C8	1·542 (10)
C9—C10	1·388 (16)	C10—C11	1·402 (10)
C11—C12	1·399 (11)	C11—C11*a*	1·464 (18)
C12—C13	1·389 (16)		

N1—Cu—O1	89·8 (2)	N1—Cu—O2	88·7 (2)
O1—Cu—O2	90·1 (2)	N1—Cu—N1*a*	180·0 (1)
O1—Cu—N1*a*	90·2 (2)	O2—Cu—N1*a*	91·3 (2)
N1—Cu—O1*a*	90·2 (2)	O1—Cu—O1*a*	180·0 (1)
O2—Cu—O1*a*	89·9 (2)	N1—Cu—O2*a*	91·3 (2)
O1—Cu—O2*a*	89·9 (2)	O2—Cu—O2*a*	180·0 (1)
C1—S—C4	91·4 (4)	Cu—N1—C9	123·1 (5)
Cu—N1—C13	119·0 (6)	C9—N1—C13	117·8 (8)
C14—N2—C15	129·4 (19)	C14—N2—C16	112·9 (24)
C15—N2—C16	117·4 (20)	Cu—O1—C5	127·1 (3)
Cu—O2—C7	121·0 (4)	S—C1—C2	113·0 (6)
C1—C2—C3	114·1 (7)	C2—C3—C4	110·6 (6)
S—C4—C3	110·9 (4)	S—C4—C5	117·9 (5)
C3—C4—C5	131·1 (6)	O1—C5—C4	114·9 (5)
O1—C5—C6	125·6 (5)	C4—C5—C6	119·5 (6)
C5—C6—C7	124·2 (6)	O2—C7—C6	130·2 (6)
O2—C7—C8	112·7 (5)	C6—C7—C8	117·1 (6)
F1—C8—F2	105·6 (10)	F1—C8—F3	106·8 (8)
F2—C8—F3	106·3 (7)	F1—C8—C7	115·8 (6)
F2—C8—C7	110·5 (7)	F3—C8—C7	111·4 (10)
N1—C9—C10	123·1 (7)	C9—C10—C11	120·2 (7)
C10—C11—C12	115·8 (8)	C10—C11—C11*a*	121·9 (8)
C12—C11—C11*a*	122·3 (7)	C11—C12—C13	120·5 (7)
N1—C13—C12	122·6 (8)	N2—C14—O3	160·8 (40)

Related literature. Recent research on organic conductors has involved *DA* (donor–acceptor) com-

* Lists of structure factors, thermal parameters, calculated H-atom coordinates, least-squares planes and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53675 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

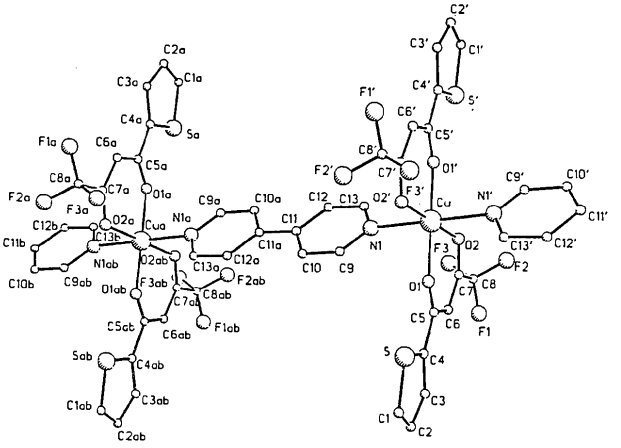

Fig. 1. The asymmetric unit in the complex (without solvent).

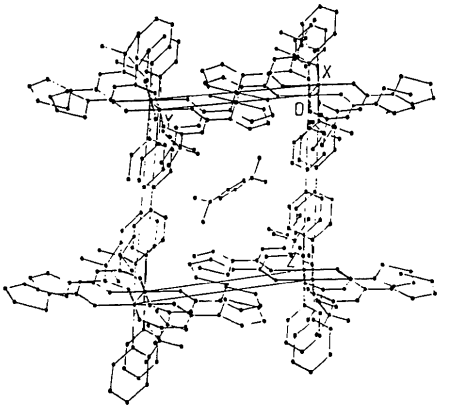

Fig. 2. The molecular packing in the unit cell.

pounds with high conductivities (Williams, Wang, Emge, Beno, Leung, Carlson, Thorn, Schultz & Whangbo, 1987). Such complexes with Cu^{2+}, Ni^{2+}, Pt^{2+} and Au^{+} are found to be planar in the crystal structures without exception. In the title complex, the *DA* moiety also possesses a large degree of electron delocalization associated with a planar structure and may, therefore, exhibit the property of 'high' conductivity; further research will be undertaken.

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Structure of $[ZrCl_3(CH_3OCH_2CH_2OCH_3)]_2O$

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Abstract. μ-Oxo-bis[*mer*-{1,2-bis(dimethoxy)ethane-*O*,*O*′}trichlorozirconium(IV)], $[Zr_2(Cl)_6(O)(C_4H_{10}O_2)_2]$, $M_r = 591{\cdot}40$, orthorhombic, *Cmca*, $a = 8{\cdot}683\ (2)$, $b = 11{\cdot}954\ (2)$, $c = 20{\cdot}361\ (3)$ Å, $V = 2113\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}859$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 17{\cdot}49$ cm^{-1}, $F(000) = 1160$, $T = 294$ K, $R = 0{\cdot}048$ for 716 unique observed reflections. The molecule is located around a 2/*m* symmetry site and consists of two distorted octahedra sharing an oxygen corner. The plane of the molecule, coplanar with the mirror plane, is defined by the Zr atoms, the bridging O atom, the 1,2-bis(dimethoxy)ethane (dme) ligands, and two of the six Cl ligands. The remaining Cl ligands are located above and below the Zr atoms. The twofold symmetry axis passes through the bridging O atom. Each Zr is six coordinate, bound to three Cl ligands and three O atoms in a *mer* configuration. Two of the O atoms come from the dme ligand that forms a five-membered chelate ring with Zr. The Zr—O_{dme} distances are 2·225 (7) and 2·334 (8) Å. The Zr—O—Zr angle is 180° and the Zr—O_{bridge} distance is 1·914 (1) Å. The Zr—Cl distances range from 2·386 (3) to 2·417 (3) Å.

Experimental. Compound prepared by the reaction of $ZrCl_4$ (2·0 g) with 1,2-bis(dimethoxy)ethane (30 mL) under argon at 195 K. Crystals obtained by warming the reaction mixture to room temperature, filtering through Celite, and layering with isomers of hexane. The quality of the crystal was confirmed by

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