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Di-μ-fluoro-bis[bis(dimethyl sulfoxide)-trifluorozirconium(IV)]

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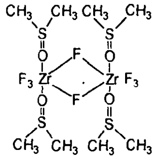
Abstract

The title compound, $[ZrF_4(C_2H_6OS)_2]_2$, is a centrosymmetric fluorine-bridged dimer. The seven-coordinate Zr atoms are in a pentagonal bipyramidal environment with F atoms above and below the pentagonal plane. The average Zr—F bridging distance of 2.167 (4) Å is significantly longer than the terminal distances which range from 1.973 (4) to 2.001 (4) Å.

Comment

Dimethyl sulfoxide (DMSO) is known to be both a good ligand and solvent for metal compounds (Reynolds, 1970). For the Group 4 elements (Ti, Zr, Hf) several adducts are known, for example MF_4.2DMSO (M = Ti, Zr) (Muetterties, 1960), $TiCl_4$.2DMSO (Schläfer & Schaffernicht, 1960) and $ZrCl_4$.2(or 3)DMSO (Makhija & Westland, 1978), together with several solvates such as $ZrCl_4$.9DMSO (Schläfer & Wille, 1965). Suprisingly there is no definitive structural information for any of these adducts. An earlier IR investigation (Clark & Errington, 1967) of the 1:2 adduct formed between ZrF_4 and dimethyl sulfoxide was inconclusive; the three observed Zr—F stretching modes suggested that the complex has low symmetry and eliminated the *trans*-octahedral structure. The present study of the dimethyl sulfoxide adduct (I) establishes a centrosymmetric dimeric structure with fluorine bridges (see Fig. 1). Each Zr atom is seven-coordinated with a pentagonal bipyramidal stereochemistry, F3 and F4 being in axial sites. The Zr—F bridging bonds [2.180 (4) and 2.153 (4) Å] are significantly longer than the terminal bonds [1.973 (4), 1.983 (4) and 2.001 (4) Å]. The dimethyl sulfoxide ligands coordinate to the Zr *via* their O atoms [Zr—O 2.216 (5) and 2.217 (5) Å].



(I)

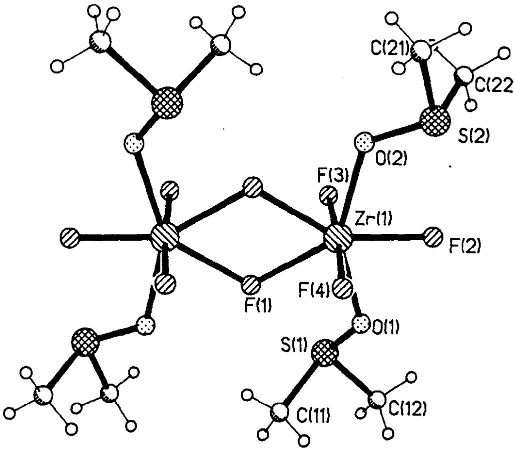


Fig. 1. View of the molecule showing the labelling of the non-H atoms.

Experimental

Crystal data

$[Zr_2F_8(C_2H_6OS)_4]$
M_r = 323.48
Monoclinic
$P2_1/n$
a = 8.831 (2) Å
b = 11.955 (2) Å
c = 10.834 (2) Å
β = 108.12 (3)°
V = 1087.1 (4) Å^3
Z = 4

D_x = 1.976 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 23 reflections
θ = 8–11°
μ = 1.420 mm^{-1}
T = 293 (2) K
Transparent blocks
0.43 × 0.18 × 0.17 mm
Colourless

Data collection

Siemens $R3m$ diffractometer
ω-2θ scans

$R_{\rm int}$(on F^2) = 0.0577
$\theta_{\rm max}$ = 25.05°

Absorption correction:
Gaussian
$T_{\min} = 0.58$, $T_{\max} = 0.87$
2009 measured reflections
1917 independent reflections
1537 observed reflections
$[I > 2\sigma(I)]$

$h = -9 \rightarrow 10$
$k = 0 \rightarrow 14$
$l = -9 \rightarrow 12$
3 standard reflections
monitored every 200
reflections
intensity variation: 10%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0527$
$wR(F^2) = 0.1497$
$S = 1.068$
1917 reflections
123 parameters
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.0967P)^2 + 2.2710P]$
where $P = (F_o^2 + 2F_c^2)/3$

$\Delta\rho_{\max} = 0.74$ e Å^{-3}
$\Delta\rho_{\min} = -2.01$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8, 6.1.1.4)

Methyl H atoms were positioned from stereochemical considerations; a single orientation parameter was refined for each Me group as was an isotropic U [0.065 (7) Å^2] common to all methyl H atoms. Data collection: Siemens *R3m* system. Cell refinement: Siemens *R3m* system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXS90* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*.

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

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

	x	y	z	$U_{\rm eq}$
Zr1	0.03818 (7)	0.05068 (5)	0.35310 (5)	0.0237 (2)
S1	−0.0959 (2)	−0.20149 (14)	0.2155 (2)	0.0338 (4)
S2	0.1519 (2)	0.31475 (13)	0.3198 (2)	0.0326 (4)
F1	−0.0185 (5)	−0.0903 (3)	0.4586 (4)	0.0410 (10)
F2	0.0812 (6)	0.1088 (3)	0.1940 (4)	0.0463 (11)
F3	−0.1949 (5)	0.0742 (4)	0.2786 (5)	0.0541 (12)
F4	0.2662 (5)	0.0166 (4)	0.4337 (5)	0.0536 (12)
O1	0.0143 (6)	−0.0988 (4)	0.2276 (5)	0.0385 (11)
O2	0.0766 (7)	0.2321 (4)	0.3933 (5)	0.0434 (12)
C11	0.0163 (9)	−0.3047 (6)	0.3232 (7)	0.041 (2)
C12	−0.0905 (10)	−0.2651 (7)	0.0682 (7)	0.051 (2)
C21	0.2215 (10)	0.4252 (6)	0.4344 (8)	0.047 (2)
C22	−0.0108 (10)	0.3831 (7)	0.2059 (8)	0.051 (2)

Table 2. *Selected geometric parameters* (Å, °)

Zr1—F4	1.973 (4)	S1—O1	1.546 (5)
Zr1—F3	1.983 (4)	S1—C11	1.774 (8)
Zr1—F2	2.001 (4)	S1—C12	1.781 (7)
Zr1—F1$^{\rm i}$	2.153 (4)	S2—O2	1.544 (5)
Zr1—F1	2.180 (4)	S2—C22	1.776 (8)
Zr1—O1	2.216 (5)	S2—C21	1.785 (7)
Zr1—O2	2.217 (5)	F1—Zr1$^{\rm i}$	2.153 (4)
F4—Zr1—F3	175.5 (2)	F4—Zr1—O2	92.4 (2)
F4—Zr1—F2	90.7 (2)	F3—Zr1—O2	91.0 (2)
F3—Zr1—F2	92.9 (2)	F2—Zr1—O2	76.8 (2)
F4—Zr1—F1$^{\rm i}$	89.9 (2)	F1$^{\rm i}$—Zr1—O2	69.6 (2)
F3—Zr1—F1$^{\rm i}$	88.6 (2)	F1—Zr1—O2	134.3 (2)
F2—Zr1—F1$^{\rm i}$	146.3 (2)	O1—Zr1—O2	152.8 (2)
F4—Zr1—F1	88.7 (2)	O1—S1—C11	106.7 (3)
F3—Zr1—F1	86.8 (2)	O1—S1—C12	102.9 (3)
F2—Zr1—F1	148.9 (2)	C11—S1—C12	97.3 (4)
F1$^{\rm i}$—Zr1—F1	64.8 (2)	O2—S2—C22	105.5 (4)
F4—Zr1—O1	89.7 (2)	O2—S2—C21	103.0 (3)
F3—Zr1—O1	88.6 (2)	C22—S2—C21	99.9 (4)
F2—Zr1—O1	76.0 (2)	Zr1$^{\rm i}$—F1—Zr1	115.2 (2)
F1$^{\rm i}$—Zr1—O1	137.6 (2)	S1—O1—Zr1	129.0 (3)
F1—Zr1—O1	72.8 (2)	S2—O2—Zr1	126.1 (3)

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

The complex was prepared by refluxing ZrF_4 in dimethyl sulfoxide (Muetterties, 1960) and allowed to crystallize upon cooling. The complex decomposes slowly in air and therefore the data were collected with the crystal mounted in a Lindemann tube; intensities were corrected for crystal decomposition, indicated by a 10% reduction in the intensities of the standard reflections in the course of the experiment.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71530 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1073]

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Dimeric μ-Hydroxy-diphenyl-(trichloroacetato)tin

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Abstract

The title compound, di-μ-hydroxy-bis[diphenyl(trichloroacetato-*O*)tin], $[Sn(C_2Cl_3O_2)(C_6H_5)_2(OH)]_2$, is a centrosymmetric dimer, with distorted trigonal-bipyramidally coordinated Sn atoms bridged by OH groups. The latter are hydrogen bonded to the unidentate carboxylates.

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