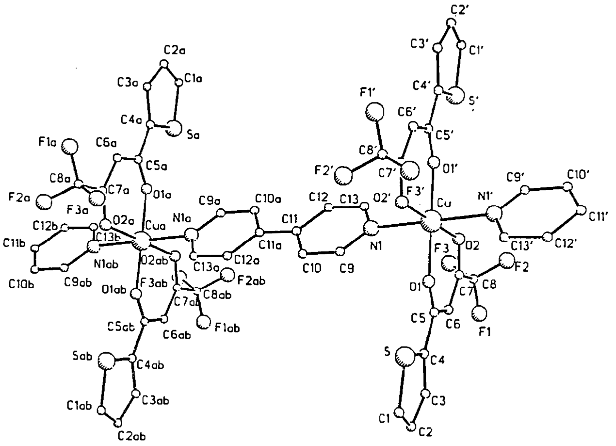

Fig. 1. The asymmetric unit in the complex (without solvent).

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.

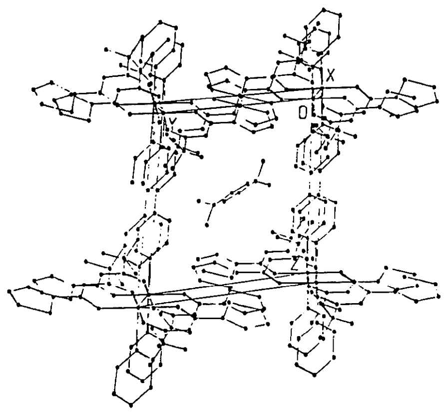

Fig. 2. The molecular packing in the unit cell.

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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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Table 1. *Positional and equivalent isotropic displacement parameters and their e.s.d.'s for* $[ZrCl_3(dme)]_2O$

$$B_{eq} = (1/3)\Sigma_i\Sigma_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Zr	0·000	0·04170 (8)	0·09075 (4)	3·16 (2)
Cl(1)	0·2765 (3)	0·0595 (2)	0·0998 (1)	6·15 (6)
Cl(2)	0·000	−0·1347 (3)	0·1456 (2)	6·9 (1)
O(1)	0·000	0·000	0·000	4·4 (2)
O(2)	0·000	0·2249 (6)	0·0715 (5)	5·2 (2)
O(3)	0·000	0·1386 (7)	0·1905 (4)	5·8 (2)
C(1)	0·062 (2)	0·280 (1)	0·016 (1)	7·2 (6)
C(2)	−0·043 (3)	0·298 (1)	0·123 (1)	8·7 (7)
C(3)	0·044 (4)	0·256 (1)	0·179 (1)	8·6 (9)
C(4)	0·053 (3)	0·094 (2)	0·251 (1)	7·5 (6)

Table 2. *Interatomic bond distances* (Å) *and angles* (°) *for* $[ZrCl_3(dme)]_2O$

Zr—Cl(1)	2·417 (3)	O(2)—C(1)	1·42 (2)
Zr—Cl(2)	2·386 (3)	O(2)—C(2)	1·42 (2)
Zr—O(1)	1·914 (1)	O(3)—C(3)	1·47 (2)
Zr—O(2)	2·225 (7)	O(3)—C(4)	1·42 (2)
Zr—O(3)	2·338 (8)	C(2)—C(3)	1·46 (3)
Cl(1)—Zr—Cl(1)′	166·6 (1)	Zr—O(3)—C(3)	109·8 (9)
Cl(1)—Zr—Cl(2)	92·42 (7)	Zr—O(3)—C(4)	124·9 (9)
Cl(1)—Zr—O(1)	95·53 (6)	C(3)^a—O(3)—C(4)^a	114 (1)
Cl(1)—Zr—O(2)	85·80 (7)	C(3)^a—O(3)—C(4)^b	125 (1)
Cl(1)—Zr—O(3)	83·70 (6)	O(2)—C(2)—C(3)	104 (2)
Cl(2)—Zr—O(1)	102·79 (9)	O(3)—C(3)—C(2)	108 (2)
Cl(2)—Zr—O(2)	162·3 (3)	Zr—O(1)—Zr′	180·00 (0)
Cl(2)—Zr—O(3)	91·8 (2)	Zr—O(2)—C(1)	127·0 (8)
O(1)—Zr—O(2)	94·9 (3)	Zr—O(2)—C(2)	118·1 (8)
O(1)—Zr—O(3)	165·4 (2)	C(1)^a—O(2)—C(2)^a	114 (1)
O(2)—Zr—O(3)	70·5 (3)	C(1)^a—O(2)—C(2)^b	102 (1)

Numbers in parentheses are e.s.d.'s in the least significant digits.

polarized light microscopy. Pale-green needle, 0·40 × 0·15 × 0·15 mm, mounted in a capillary under argon. Intensities measured with Rigaku AFC5R diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω–2θ scans, scan speed 8° min^{-1}, maximum four-scan repetition to obtain $F/\sigma(F) > 25$. Unit-cell dimensions determined from least-squares analysis of 25 reflections with $20 < 2\theta < 30°$. Laue class and unit-cell dimensions confirmed with axial photographs. Data collected to $(\sin\theta)/\lambda = 0{\cdot}647$ Å^{-1}, $0 \le h \le 24$; $0 \le k \le 14$; $0 \le l \le 10$. Three standard reflections ($2\bar{4}0$, $0\bar{2}8$ and $42\bar{3}$), measured every 150 reflections, showed small (<0·2%) random variations. 1375 reflections measured, 1282 unique ($R_{int} = 0{\cdot}01$), 716 considered observed with $F_o^2 > 3\sigma(F_o^2)$. Analytical absorption correction based on ψ scans (North, Phillips & Matthews, 1968) varied from 0·89 to 1·00. Data corrected for Lorentz and polarization effects.

Systematic absences suggested *Cmca* or *C2cb* (non-standard setting of *Aba*2) as possible space groups. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986). The parameters were first refined in space group *Cmca*, but this attempt resulted in disordered dme ligands. The C atoms located off the mirror plane were refined at 0·5 occupancy. An attempt to remove the disorder in *Aba*2 failed. The data were subjected to full-matrix least-squares refinement in *Cmca* employing the Enraf–Nonius (1979) *Structure Determination Package*. wR minimized, where $w = 1/\sigma^2(|F_o|)$. All atoms refined anisotropically for a total of 74 variables. H atoms not included. The final $R = 0{\cdot}048$, $wR = 0{\cdot}067$, $S = 1{\cdot}48$. At convergence, $(\Delta/\sigma)_{max} = 0{\cdot}05$, $\Delta\rho_{max} = 0{\cdot}97$ and $\Delta\rho_{min} = -0{\cdot}45$ e Å^{-3} on final difference Fourier map. Atomic scattering factors and anomalous-dispersion correction from *International Tables for X-ray Crystallography* (1974, Vol. IV). No extinction correction applied.

Table 1 gives positional and equivalent isotropic displacement parameters, Fig. 1 shows the molecule

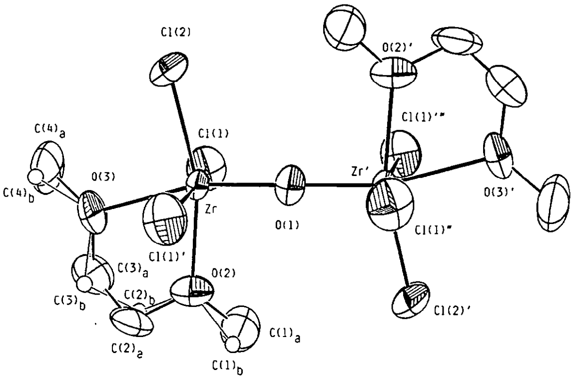

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the $[ZrCl_3(dme)]_2O$ molecule. Atoms are represented by displacement ellipsoids at the 50% level. Small circles represent the alternative orientation of the disordered dme ligand. For clarity the alternative orientation of the dme ligand around Zr′ is not shown.

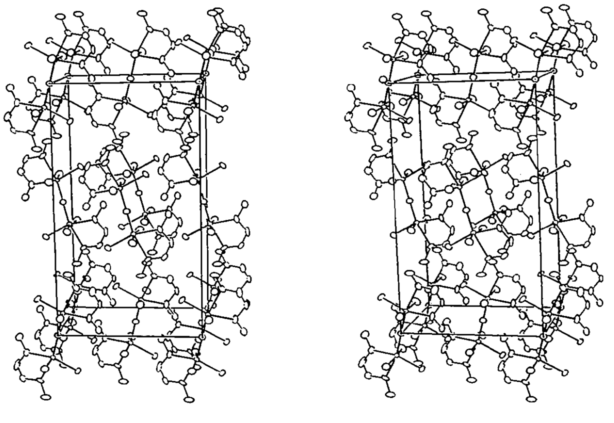

Fig. 2. Unit-cell diagram for $[ZrCl_3(dme)]_2O$. Atoms are represented by displacement ellipsoids at the 20% level. Axes orientation: **b** across, **c** down, **a** towards the viewer. For clarity each molecule is shown with one, arbitrarily chosen, orientation of the disordered dme ligands.

with the numbering scheme, and Table 2 gives selected interatomic bond distances and angles. A unit-cell diagram is shown in Fig. 2.*

Related literature. Structural work on related organometallic complexes $[Zr(C_5H_5)_2Cl]_2O$ (Clarke & Drew, 1974) and $[Zr(C_5H_5)_2CH_3]_2O$ (Hunter, Hrncir, Bynum, Penttila & Atwood, 1983) has been reported.

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* Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53720 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Orthorhombic Crystal Form of *trans*-Carbonylchlorobis(triphenylphosphine)rhodium(I) Dichloromethane Solvate

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Abstract. $[RhCl(C_{18}H_{15}P)_2(CO)].CH_2Cl_2$, $M_r = 775{\cdot}9$, orthorhombic, $Pbca$, $a = 23{\cdot}345\,(3)$, $b = 20{\cdot}644\,(7)$, $c = 7{\cdot}965\,(3)$ Å, $V = 3821{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}35$ g cm^{-3}, monochromated Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 7{\cdot}6$ cm^{-1}, $F(000) = 1576$, $T = 298$ K, $R = 0{\cdot}056$ and $wR = 0{\cdot}063$ for 587 observed reflections with $|F^2| > 3\sigma(F^2)$. The geometry at Rh is close to square planar and bond lengths and angles are similar to those in the previous determinations of the unsolvated structure.

Experimental. Large, very thin plates of *trans*-$[Rh(PPh_3)_2(CO)Cl]$ were isolated from an attempted purification of *trans*-$[Rh_2(PPh_3)_2(CO)_2\{\mu\text{-}S(CH_2)_4S\}]$ and a fragment *ca* $0{\cdot}3 \times 0{\cdot}1 \times 0{\cdot}008$ mm was used for data collection. Unit-cell parameters by least-squares fit of 25 reflections in the range $6{\cdot}7 < \theta < 11{\cdot}3^\circ$, space group $Pbca$ from systematic absences of $0kl$, k odd; $h0l$, l odd; $hk0$, h odd, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, θ–2θ scan, $\Delta\theta = (0{\cdot}8 + 0{\cdot}35\tan\theta)^\circ$, maximum scan time 1 min, 2087 unique reflections measured for $2 < \theta < 20^\circ$ and h $0\rightarrow7$, k $0\rightarrow19$, l $0\rightarrow22$, 587 observed reflections with $|F^2| > 3\sigma(F^2)$, $\sigma(F^2) = [\sigma^2(I) + (0{\cdot}04I)^2]^{1/2}/Lp$. Two standard reflections remeasured every 30 min showed no significant change, Lorentz–polarization corrections, no absorption or extinction corrections. The structure was solved by routine heavy-atom methods and showed disorder between carbonyl and chloride ligands. The carbonyl C and O atoms were fixed at idealized positions with Rh—C = 1·82 and C—O = 1·14 Å (Rheingold & Geib, 1987). Rh atom anisotropic, otherwise non-H atoms were refined isotropically by

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