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Bis(acetonitrile-*N*)diaquatrichloroerbium(III) Acetonitrile Solvate

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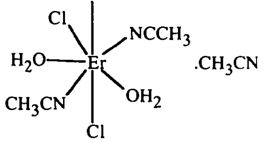
Abstract

The metal atom in the title compound, [$ErCl_3(MeCN)_2(H_2O)_2$].MeCN, is seven-coordinate and has a distorted pentagonal-bipyramidal geometry, with two of the chlorines in the axial positions.

Comment

Thionyl chloride dehydration of MCl$_3$.6H$_2$O in the presence of tetrahydrofuran (thf) invariably leads to the formation of the solvates MCl$_3$(thf)$_x$. However, there are examples where incomplete removal of water is observed. Typically, we have previously isolated the 'mixed' solvates [$ThCl_4(thf)_3(H_2O)$] (Spry *et al.*, 1997) and [$YCl_3(H_2O)_2(MeCN)_2$] (Willey *et al.*, 1996). In this report, we describe [$ErCl_3(MeCN)_2(H_2O)_2$].MeCN, (I), as obtained from the reaction of $ErCl_3.6H_2O/SOCl_2$/MeCN.



(I)

The [$ErCl_3(MeCN)_2(H_2O)_2$] complex in (I) is represented in Fig. 1. The Er—Cl bond distances range from 2.6132 (9) to 2.6443 (9) Å, with a mean value of 2.6255 (9) Å. These values compare well with the mean Er—Cl bond distances of 2.590 (2) Å in $ErCl_3$(triethylene glycol) (Rodgers, Voss & Etzenhouser, 1988) and 2.632 (3) Å in [$ErCl_2$(12-crown-4)(H_2O)$_2$]Cl (Rodgers, Rollins & Benning, 1988). The Er—N bond distances of 2.436 (3) and 2.455 (3) Å [mean 2.446 (3) Å] are slightly longer than those found in [$ErFe(CN)_6$].4H$_2$O [mean 2.420 (5) Å] (Dommann *et al.*, 1990), but shorter than those in [$ErCl(H_2O)_4$(terpy)]Cl$_2$, terpy is terpyridyl (Keppert *et al.*, 1994). Similarly, the Er—O(H_2O) distances of 2.318 (3) and 2.369 (3) Å [mean 2.344 (3) Å] are slightly shorter than those present in [$ErCl(H_2O)_4$(terpy)]Cl$_2$ [mean 2.353 (7) Å], but are significantly longer than those found in [$ErCl_2$(12-crown-4)(H_2O)$_2$]Cl [mean 3.311 (5) Å].

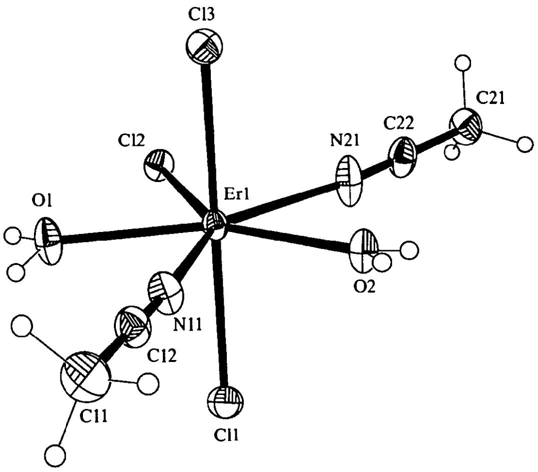


Fig. 1. View of the title complex showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

For the seven-coordinate Er^{III} centre, the metal geometry approximates to distorted pentagonal bipyramidal. The atoms in the equatorial (eq) plane have an r.m.s. deviation from the least-squares plane of only 0.018 Å. The axial (ax) Cl atoms have an Cl—Er—Cl angle of 170.55 (3)°. This non-linearity may be due to the Cl$_{ax}$—Cl$_{eq}$ repulsions, with a consequent bending of the Cl$_{ax}$—Er—Cl$_{eq}$ angles to 95.18 (3) and 93.82 (3)°. The equatorial inter-ligand angles are also slightly distorted from the ideal (72°), with the largest angles unsurprisingly involving the single equatorial Cl atom, Cl2—Er—N21 72.85 (8) and Cl2—Er—O1 76.38 (7)°. Similar variations are observed in the seven-coordinate $ErCl_3$(triethylene glycol), *viz* Cl$_{ax}$—Er—Cl$_{ax}$ 169.82 (7), and Cl$_{eq}$—Er—O$_{glycol}$ 80.2 (1) and 79.5 (1)°.

Experimental

The title compound was obtained from the $ErCl_3.6H_2O/SOCl_2$/MeCN system following heating at reflux for 24 h.

After removal of the solvent *in vacuo*, the resulting solid was washed with diethyl ether (2 × 25 ml) to give a pink powder. Recrystallization from MeCN gave pink-tinged block crystals.

Crystal data

$[ErCl_3(C_2H_3N)_2(H_2O)_2].C_2H_3N$
$M_r = 432.80$
Monoclinic
$P2_1/n$
$a = 9.6642 (6)$ Å
$b = 9.5221 (6)$ Å
$c = 16.1965 (4)$ Å
$\beta = 107.036 (2)°$
$V = 1425.1 (1)$ Å^3
$Z = 4$
$D_x = 2.017$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 6151 reflections
$\theta = 2.21–28.75°$
$\mu = 6.438$ mm^{-1}
$T = 180 (2)$ K
Block
0.20 × 0.14 × 0.10 mm
Pink

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996*a*)
$T_{min} = 0.276$, $T_{max} = 0.525$
8331 measured reflections
3346 independent reflections

2796 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 28.75°$
$h = -12 \rightarrow 12$
$k = -9 \rightarrow 12$
$l = -21 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.052$
$S = 0.985$
3346 reflections
151 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0249P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.86$ e Å^{-3}
$\Delta\rho_{min} = -1.285$ e Å^{-3} (0.80 Å from Er1)
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Er1—O1	2.318 (3)	Er1—Cl1	2.6132 (9)
Er1—O2	2.369 (3)	Er1—Cl3	2.6189 (9)
Er1—N21	2.436 (3)	Er1—Cl2	2.6443 (9)
Er1—N11	2.455 (3)		
O2—Er1—N21	69.1 (1)	N21—Er1—Cl2	72.85 (8)
O1—Er1—N11	69.9 (1)	Cl1—Er1—Cl2	95.18 (3)
O2—Er1—N11	71.8 (1)	Cl3—Er1—Cl2	93.82 (3)
Cl1—Er1—Cl3	170.55 (3)	N11—C12—C11	179.3 (4)
O1—Er1—Cl2	76.38 (7)	N21—C22—C21	179.0 (4)

The temperature of the crystal was controlled using an Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). Data were collected over a hemisphere of reciprocal space, by a combination of three sets of exposures. Each set had a different φ angle for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 5.01 cm. Coverage of the unique set was over 91% complete to at least 28° in θ. Crystal decay was monitored by repeating the initial frames at the end of the data collection and analysing the duplicate reflections. H atoms were added at calculated positions and refined using a riding model; the H atoms in the two water molecules were inserted from the electron density map but restrained to make all four O—H distances the same. Anisotropic displacement parameters were used for all non-H atoms. The wide variations in the anisotropic displacement parameters for C13 (the acetonitrile methyl C atom) probably indicate disorder. H atoms were given isotropic displacement parameters equal to 1.2 (1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached.

Data collection: *SMART* (Siemens, 1994*a*). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL/PC* (Siemens, 1994*b*). Program(s) used to refine structure: *SHELXL96* (Sheldrick, 1996*b*). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1345). Services for accessing these data are described at the back of the journal.

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