Acta Cryst. (1991). C47, 426–427

Structure of Trichlorotetrakis(tetrahydrofuran)europium(III)

BY SHAN-HUO LIN, ZHEN-CHAO DONG, JIN-SHUN HUANG, QIAN-ER ZHANG AND JIA-XI LU

Fuzhou Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, *People's Republic of China*

(*Received* 1 *November* 1989; *accepted* 2 *July* 1990)

Abstract. $[EuCl_3(C_4H_8O)_4]$, $M_r = 546{\cdot}75$, monoclinic, $C2/c$, $a = 35{\cdot}555\ (5)$, $b = 8{\cdot}201\ (3)$, $c = 17{\cdot}550\ (6)$ Å, $\beta = 116{\cdot}08\ (6)°$, $V = 4596{\cdot}2\ (4)$ Å^3, $Z = 8$, $D_x = 1{\cdot}58$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 31{\cdot}00$ cm^{-1}, $F(000) = 2192$, $T = 293$ K, $R = 0{\cdot}041$, $wR = 0{\cdot}050$ for 2053 reflections with $I > 3\sigma(I)$. The central Eu ion is surrounded by three Cl and four O atoms which define a slightly distorted pentagonal bipyramid. An approximate non-crystallographic plane of symmetry passes through the atoms Eu, Cl(1), Cl(2), and Cl(3) and relates pairs of C_4H_8O rings. The average interatomic Eu—Cl and Eu—O distances are 2·628 (3) and 2·453 (7) Å respectively.

Experimental. The title compound was obtained as an unexpected product from the reaction of $EuCl_3$ with tetrahydrofuran (THF, C_4H_8O) in a stream of H_2S. A pale-yellow single crystal with approximate dimensions 0·15 × 0·31 × 0·25 mm was mounted in a glass capillary with its long axis roughly parallel to the φ axis of the goniometer. Data collection was performed on an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$ radiation, graphite monochromator). From least-squares refinement on the basis of 25 automatically centered reflections in the range $22 \leq 2\theta \leq 28°$, a triclinic cell with $a = 8{\cdot}194\ (5)$, $b = 17{\cdot}538\ (3)$, $c = 18{\cdot}241\ (6)$ Å, $\alpha = 115{\cdot}34\ (2)$, $\beta = 103{\cdot}09\ (6)$, $\gamma = 89{\cdot}95\ (4)°$ was initially obtained. The intensities were collected at 293 K using the ω–2θ scan technique with a variable rate of 1 to 7° min^{-1} and a scan range of $(0{\cdot}45 + 0{\cdot}35\tan\theta)°$ in the range $2 \leq 2\theta \leq 50°$. The index range was $-9 \leq h \leq 9$, $-20 \leq k \leq 20$, $-21 \leq l \leq 0$. Three standard reflections measured at exposure intervals of 1 h exhibited no significant change. 8332 reflections were collected, of which 4594 had $I > 3\sigma(I)$. Lorentz and polarization corrections were applied. An empirical absorption correction based on a series of ψ scans was also applied. Relative transmission coefficients ranged from 0·527 to 1·000 with an average value of 0·807. Subsequently, we found a transformation to a monoclinic C-centered unit cell (see *Abstract*). Intensities of equivalent reflections were averaged, resulting in 3590 unique reflections of which 2053 had $I > 3\sigma$(I).

From the systematic absences and from subsequent least-squares refinement, the space group was determined to be $C2/c$. The structure was solved using the Patterson method which revealed the position of the Eu atom. Subsequent difference Fourier syntheses yielded the location of all remaining atoms except for the H atoms. H atoms were located according to their ideal molecular geometries and added to the structure-factor calculations with their positions and isotropic temperature factors fixed. Full-matrix least-squares refinement of 218 variables on F yielded $R = 0{\cdot}041$, $wR = 0{\cdot}050$, $w = 4F_o^2/|\sigma^2(F_o^2) + 0{\cdot}04F_o^2|$. Scattering factors for neutral atoms and f', f'' were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72–151). The maximum ratio of parameter shift/e.s.d. in the final refinement cycle was 0·03 and the minimum, maximum values in the final difference Fourier synthesis were $-0{\cdot}31$, $+0{\cdot}56$ e Å^{-3}. $S = 1{\cdot}10$. All calculations were made with a VAX computer using the VAX/*SDP* program package (Frenz, 1978).* A view of the $[EuCl_3(C_4H_8O)_4]$ structure, based on the atomic coordinates in Table 1, is given in Fig. 1. Table 2 lists the interatomic Eu—Cl and Eu—O distances, as well as the bond angles centered at the Eu atom. Least-squares-plane analysis suggests that atoms Eu, Cl(1), Cl(2), Cl(3) and Eu, Cl(3), O(1), O(2), O(3), O(4) form least-squares planes $\pi 1$ and $\pi 2$, respectively. Taking into account the relative positions of the four C_4H_8O rings, we found that there exists a pseudo-mirror through $\pi 1$, as depicted in Fig. 1.

Related literature. Three preferred coordination polyhedra for this coordination number have been observed. $[Ce^{III}Cl_2(THF)_5]^+$ in $[Ce^{III}Cl_2(THF)_5][Ce^{IV}Cl_5(THF)]$ (Jin, Jin, Wang & Chen, 1988) and

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles for C and O atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53369 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/91/020426-02$03.00

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$B_{eq} = (4/3)\Sigma_i\Sigma_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Eu	0·12445 (1)	0·04622 (7)	0·15819 (3)	4·63 (1)
Cl(1)	0·06728 (9)	−0·1826 (4)	0·1026 (2)	7·78 (9)
Cl(2)	0·18124 (9)	0·2750 (4)	0·1903 (2)	7·40 (9)
Cl(3)	0·1238 (1)	0·0797 (6)	0·3070 (2)	11·7 (1)
O(1)	0·0681 (2)	0·238 (1)	0·1329 (4)	7·6 (2)
O(2)	0·1568 (2)	−0·1084 (9)	0·0803 (4)	6·9 (2)
O(3)	0·0957 (2)	0·152 (1)	0·0110 (4)	6·9 (2)
O(4)	0·1771 (2)	−0·141 (1)	0·2509 (4)	7·9 (2)
C(1)	0·0739 (4)	0·339 (2)	0·165 (1)	15·1 (6)
C(2)	0·0381 (4)	0·437 (2)	0·1827 (9)	12·8 (5)
C(3)	0·0065 (4)	0·329 (2)	0·131 (1)	15·1 (6)
C(4)	0·0265 (4)	0·198 (2)	0·113 (1)	16·4 (6)
C(5)	0·1923 (4)	−0·060 (2)	0·0737 (9)	15·6 (5)
C(6)	0·2088 (4)	−0·185 (2)	0·0449 (8)	12·6 (5)
C(7)	0·1779 (4)	−0·326 (2)	0·0218 (8)	13·4 (5)
C(8)	0·1430 (4)	−0·259 (2)	0·0327 (8)	13·3 (5)
C(9)	0·0615 (6)	0·092 (3)	−0·0596 (9)	19·3 (7)
C(10)	0·0484 (5)	0·190 (3)	−0·1312 (8)	14·0 (7)
C(11)	0·0802 (5)	0·315 (2)	−0·1066 (7)	16·7 (6)
C(12)	0·1046 (6)	0·294 (3)	−0·021 (1)	21·8 (8)
C(13)	0·2166 (4)	−0·084 (2)	0·3160 (9)	13·4 (6)
C(14)	0·2269 (5)	−0·193 (3)	0·3826 (8)	14·1 (6)
C(15)	0·1989 (5)	−0·348 (3)	0·3536 (9)	16·9 (7)
C(16)	0·1679 (5)	−0·309 (2)	0·272 (1)	16·0 (7)

Table 2. *Interatomic* Eu—Cl *and* Eu—O *distances* (Å) *and bond angles around the* Eu *atom* (°)

Eu—Cl(1)	2·620 (4)	Eu—O(1)	2·430 (8)
Eu—Cl(2)	2·630 (3)	Eu—O(2)	2·486 (8)
Eu—Cl(3)	2·636 (4)	Eu—O(3)	2·481 (6)
Eu—O(4)	2·413 (7)		
Cl(1)—Eu—Cl(2)	171·1 (1)	Cl(3)—Eu—O(1)	75·4 (2)
Cl(1)—Eu—Cl(3)	95·8 (1)	Cl(3)—Eu—O(2)	143·7 (2)
Cl(1)—Eu—O(1)	87·3 (2)	Cl(3)—Eu—O(3)	144·6 (3)
Cl(1)—Eu—O(2)	84·4 (2)	Cl(3)—Eu—O(4)	75·0 (3)
Cl(1)—Eu—O(3)	88·1 (2)	O(1)—Eu—O(2)	140·7 (3)
Cl(1)—Eu—O(4)	92·4 (2)	O(1)—Eu—O(3)	69·7 (2)
Cl(2)—Eu—Cl(3)	93·1 (1)	O(1)—Eu—O(4)	150·2 (3)
Cl(2)—Eu—O(1)	94·2 (2)	O(2)—Eu—O(3)	71·7 (2)
Cl(2)—Eu—O(2)	89·1 (2)	O(2)—Eu—O(4)	68·7 (2)
Cl(2)—Eu—O(3)	84·2 (2)	O(3)—Eu—O(4)	140·2 (3)
Cl(2)—Eu—O(4)	90·8 (3)		

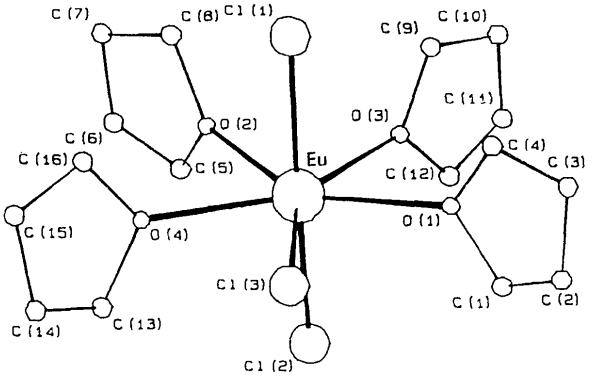

Fig. 1. Molecular diagram of $[EuCl_3(C_4H_8O)_4]$. (H atoms omitted.)

$[TbF_7]^{3-}$ in the salt $Cs_3[TbF_7]$ (Hoppe & Roeder, 1961) have pentagonal bipyramidal geometry. A monocapped octahedron is characteristic of $[Y(C_6H_5COCHCOCH_3)_3(H_2O)]$ (Cotton & Legzdins, 1968), while a monocapped trigonal prism is characteristic of $[Y(CH_3COCHCOCH_3)_3(H_2O)]$ (Cunningham, Sands, Wagner & Richardson, 1969).

References

COTTON, F. A. & LEGZDINS, P. (1968). *Inorg. Chem.* 7, 1777–1783.

CUNNINGHAM, J. A., SANDS, D. E., WAGNER, W. F. & RICHARDSON, M. F. (1969). *Inorg. Chem.* **8**, 22–28.

FRENZ, B. A. (1978). *The Enraf–Nonius CAD4 SDP–A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64–71. Delft Univ. Press.

HOPPE, R. & ROEDER, K.-M. (1961). *Z. Anorg. Allg. Chem.* **312**, 277–281.

JIN, Z. S., JIN, S. C., WANG, X. Y. & CHEN, W. Q. (1988). *JieGau HuaXue*, pp. 181–183.

Acta Cryst. (1991). C**47**, 427–429

Structure of 2,2,2-Tricarbonyl-1,1-bis(ethylene)-μ-(η^5:η^5-fulvalene)-2-methylrhodiumtungsten

BY MARY P. NEU, MICHAEL E. SMITH, THOMAS R. BOUSSIE AND DAWN A. GROENKE

Department of Chemistry, University of California, Berkeley, California, 94720, USA

(*Received* 24 *April* 1990; *accepted* 17 *July* 1990)

Abstract. $[RhW(C_{10}H_8)(CH_3)(C_2H_4)_2(CO)_3]$, M_r = 570·10, monoclinic, $P2_1/c$, a = 7·3358 (8), b = 10·8640 (15), c = 21·6310 (20) Å, β = 90·924 (8)°, V = 1723·7 (3) Å^3, Z = 4, D_m = 2·16, D_x = 2·196 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 77·7 cm^{-1}, $F(000)$ = 1080, T = 298 K, R = 0·021, wR = 0·029 for 4453 observed reflections with $I \geq 3\sigma(I)$. The compound is a bimetallic fulvalene complex with

0108-2701/91/020427-03$03.00