Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tris[4-(3-methyl-4-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-ylidene)isobutanolato-κ^2O,O']bis(triphenylphosphine oxide-κO)europium(III)

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.009 Å
R factor = 0.048
wR factor = 0.110
Data-to-parameter ratio = 18.0

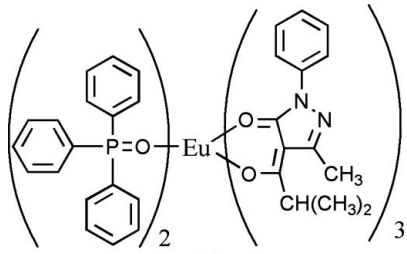
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 19 September 2005
Accepted 24 October 2005
Online 31 October 2005

In the title complex, $[Eu(C_{14}H_{15}N_2O_2)_3(C_{18}H_{15}OP)_2]$, the Eu^{III} ion is surrounded by eight O atoms, six from the β-diketonate ligands and two from the triphenylphosphine oxides, with a distorted triangular dodecahedral coordination.

Comment

Metal complexes are good electroluminescent materials (Tang & Vanslyke, 1987). As some europium and terbium complexes have been well studied (Kido & Okamota, 2002), we prepared the title Eu^{III} complex, (I), and present its structure here.



(I)

The molecular structure of (I) is shown in Fig. 1. The Eu^{III} ion is surrounded by eight O atoms, six from the β-diketonate ligands and two from the triphenylphosphine oxides. Using the criterion established by Haigh (1995), the coordination polyhedron can be described as a distorted triangular dodecahedron. The Eu—O distances (Table 1) are normal (Pettinari *et al.*, 2004). They are a little longer than the Tb—O distances [2.260 (2)–2.373 (2) Å] found in the related Tb^{III} complex (Xin *et al.*, 2003).

Experimental

An aqueous solution (10 ml) of $EuCl_3$ (1 mmol) was added dropwise to an ethanol solution (50 ml) of 1-phenyl-3-methyl-4-isobutyryl-5-pyrazolone (3 mmol), triphenylphosphine oxide (2 mmol) and NaOH (3 mmol). The solution was refluxed for 1 h to yield a white precipitate. Colorless crystals of (I) were obtained by recrystallization from an ethanol solution.

Crystal data

$[Eu(C_{14}H_{15}N_2O_2)_3(C_{18}H_{15}OP)_2]$
M_r = 1438.34
Monoclinic, $P2_1/n$
a = 13.3872 (14) Å
b = 23.187 (2) Å
c = 23.130 (2) Å
β = 91.890 (2)°
V = 7175.7 (13) Å^3
Z = 4
D_x = 1.331 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5765 reflections
θ = 2.3–21.3°
μ = 0.98 mm^{-1}
T = 294 (2) K
Block, colorless
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.815, T_{max} = 0.855
41976 measured reflections

15550 independent reflections
8316 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
θ_{max} = 27.0°
$h = -7 \rightarrow 17$
$k = -29 \rightarrow 29$
$l = -27 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.110
S = 0.98
15550 reflections
865 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.82 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Eu1—O1	2.391 (3)	Eu1—O5	2.380 (3)
Eu1—O2	2.358 (3)	Eu1—O6	2.389 (3)
Eu1—O3	2.296 (3)	Eu1—O7	2.318 (3)
Eu1—O4	2.449 (3)	Eu1—O8	2.513 (3)
O4—Eu1—O8	70.67 (11)	O3—Eu1—O2	87.09 (11)
O1—Eu1—O4	70.84 (11)	O7—Eu1—O1	93.59 (11)
O7—Eu1—O8	70.94 (11)	O3—Eu1—O1	97.94 (11)
O7—Eu1—O6	71.13 (11)	O7—Eu1—O2	102.06 (11)
O5—Eu1—O6	71.42 (10)	O6—Eu1—O8	123.95 (11)
O2—Eu1—O8	72.16 (11)	O5—Eu1—O4	128.21 (10)
O3—Eu1—O4	72.85 (10)	O6—Eu1—O4	135.92 (11)
O5—Eu1—O1	74.30 (10)	O5—Eu1—O8	138.68 (10)
O3—Eu1—O5	75.46 (10)	O2—Eu1—O4	140.62 (11)
O2—Eu1—O5	75.58 (11)	O1—Eu1—O8	140.75 (11)
O2—Eu1—O6	77.26 (12)	O7—Eu1—O5	142.00 (10)
O7—Eu1—O4	77.61 (11)	O3—Eu1—O7	142.51 (11)
O3—Eu1—O8	77.77 (11)	O3—Eu1—O6	145.92 (11)
O6—Eu1—O1	80.79 (11)	O2—Eu1—O1	147.06 (11)

The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the C—C bonds to fit the electron density. Other H atoms were positioned geometrically and treated as riding (C—H = 0.98 Å or 0.93 Å), with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

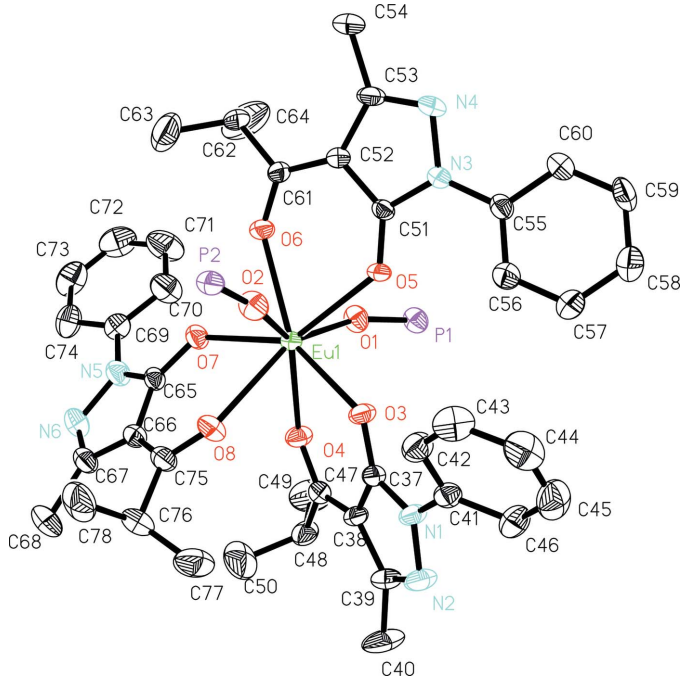


Figure 1
The molecular structure of (I); displacement ellipsoids are drawn at the 30% probability level, and the phenyl rings of triphenylphosphine oxide and H atoms have been omitted for clarity.

This work was supported by the Ministry of Education of China (Z02873) and the Bureau of Education of Jiangxi Province (Z0283).

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