Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Low-temperature structure of a twinned crystal of tris(2,4-pentanedionato)(1,10-phenanthroline)-samarium(III)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.012 Å
R factor = 0.073
wR factor = 0.201
Data-to-parameter ratio = 15.4

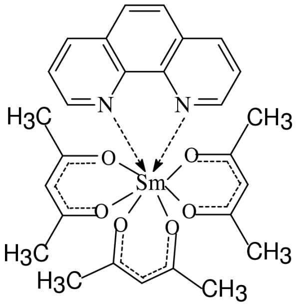
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 August 2001
Accepted 3 September 2001
Online 20 September 2001

The crystal of the title complex, [Sm($C_5H_7O_2$)$_3$($C_{12}H_8N_2$)], was found to be a non-merohedral twin. The samarium coordination is in the form of a distorted square antiprism, with the six O and two N atoms of the ligands lying at the apices.

Comment

In a continuation of work on the structures of MOCVD (metal–organic chemical vapour deposition) precursors (Patnaik *et al.*, 1996; Urs *et al.*, 2000, 2001), the structure of a samarium complex, (I), has been analysed. The crystal was found to be a non-merohedral twin with two components. The twinning arises from a rotation of 180° around the normal to (100) in real space. This twin law was used to process the data using the program *TwinSolve* (Rigaku, 1999). The eightfold coordination of the Sm atom is in the form of a distorted square antiprism. The ligands span the opposite edges of the two square faces of the coordination antiprism (*s* edges) (Hoard & Silverton, 1963), as observed in rare earth metal complexes of europium (Watson *et al.*, 1972), lanthanum (Kuz'mina *et al.*, 1997), cerium and praseodymium (Christidis *et al.*, 1998). The title complex has only one molecule in the asymmetric unit, as in the europium and praseodymium complexes, and is found to be isostructural with the praseodymium complex. The Sm—O bond distances are in the range 2.341 (5)–2.388 (5) Å and the Sm—N distances are 2.589 (5) and 2.641 (5) Å. The angle between the least-squares mean planes fitted to the two square faces of the coordination antiprism is 2.8 (1)° (Nardelli, 1995).



(I)

Experimental

The title complex was synthesized in a single step. Samarium chloride solution (10 ml, 2 mmol) was mixed with acetylacetone (0.62 ml, 6 mmol) in the presence of 50 ml of ethanol. The solution was heated to boiling and 2 *N* sodium hydroxide was added to the hot solution (to achieve pH = 6–7), followed by the addition of 1,10-phenanthroline (0.396 g, 2 mmol) in 10 ml of ethanol. The mixture was stirred for 2 h at room temperature. The precipitate thus formed was filtered, washed with water and recrystallized from aqueous ethanol. Crystals were obtained by slow evaporation at low temperature from a solution in aqueous ethanol.

Crystal data

$[Sm(C_5H_7O_2)_3(C_{12}H_8N_2)]$
$M_r = 627.87$
Monoclinic, $P2_1/n$
$a = 9.432$ (2) Å
$b = 20.957$ (5) Å
$c = 14.728$ (3) Å
$\beta = 98.899$ (9)°
$V = 2876$ (1) Å^3
$Z = 4$
$D_x = 1.45$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 23142 reflections
$\theta = 3.0$–25.0°
$\mu = 2.08$ mm^{-1}
$T = 173$ (2) K
Prism, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku AFC-8 diffractometer coupled with Mercury CCD
ω scans
23 142 measured reflections
23 142 independent reflections
4253 reflections with $I > 2\sigma(I)$
$\theta_{max} = 25.0°$
$h = -11 \rightarrow 10$
$k = -24 \rightarrow 24$
$l = -15 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.201$
$S = 1.32$
5098 reflections
332 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 55.2376P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 2.48$ e Å^{-3}
$\Delta\rho_{min} = -1.88$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Sm1—O1	2.341 (5)	Sm1—O3	2.383 (5)
Sm1—O6	2.351 (5)	Sm1—O5	2.388 (5)
Sm1—O4	2.372 (5)	Sm1—N2	2.589 (5)
Sm1—O2	2.378 (5)	Sm1—N1	2.641 (5)
O1—Sm1—O2	72.40 (16)	O6—Sm1—O5	72.33 (17)
O4—Sm1—O3	71.83 (17)	N2—Sm1—N1	62.65 (16)

Data collection: *CrystalClear* (Rigaku, 1999); data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

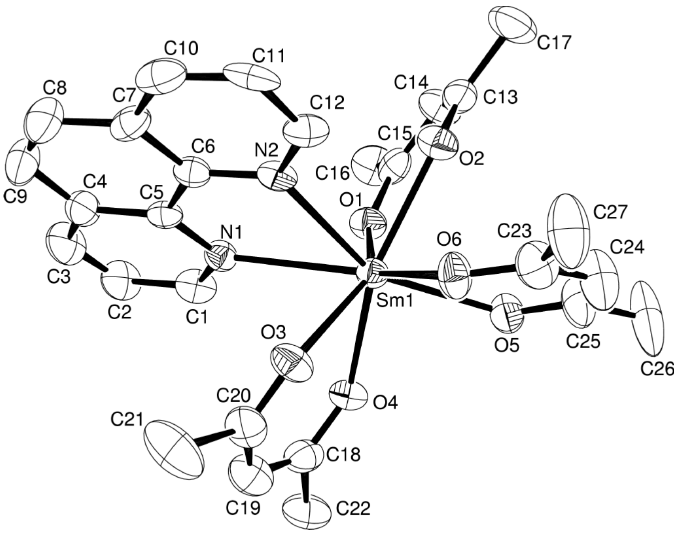


Figure 1
View of the title Sm complex (Johnson, 1965) with the atomic numbering scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

The authors thank Dr T. E. Concolino of the Molecular Structure Corporation, Texas, USA, for help with the data collection. UKU and KS thank CSIR, India, for a research associateship and research fellowship, respectively.

References

Christidis, P. C., Tossidis, I. A., Paschalidis, D. G. & Tzavellas, L. C. (1998). *Acta Cryst.* C**54**, 1233–1236.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837.
Hoard, J. L. & Silverton, J. V. (1963). *Inorg. Chem.* **2**, 235–243.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Kuz'mina, N. P., Chugarov, N. V., Pisarevsky, A. P. & Martynenko, L. I. (1997). *Koord. Khim.* **23**, 450–454.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Patnaik, S., Guru Row, T. N., Raghunathan, L., Devi, A., Goswami, J., Shivashankar, S. A., Chandrasekaran, S. & Robinson, W. T. (1996). *Acta Cryst.* C**52**, 891–894.
Rigaku (1999). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Urs, U. K., Anitha, K. C, Raghunathan, K. L., Shivashankar, S. A., Robinson, W. T. & Guru Row, T. N. (2001). *Acta Cryst.* E**57**, m242–m243.
Urs, U. K., Shalini, K., Shivashankar, S. A. & Guru Row, T. N. (2000). *Acta Cryst.* C**56**, e448–e449.
Watson, W. H., Williams, R. J. & Stemple, N. R. (1972). *J. Inorg. Nucl. Chem.* **34**, 501–508.

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