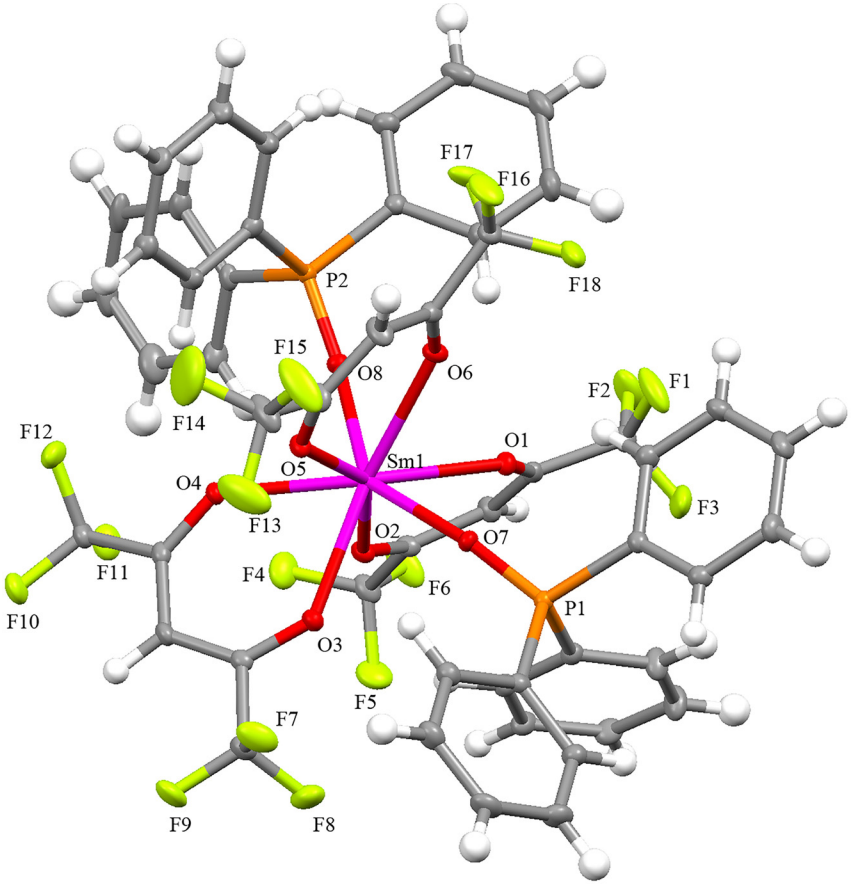
Wanbin Li, Jinke Jiang, Xin Wang and Qiang Liu*

Crystal structure of tris(hexafluoroacetylacetonato-κ^2O,O′) bis(triphenylphosphine oxide-κ^1O) samarium(III), $C_{51}H_{33}F_{18}O_8P_2Sm$



https://doi.org/10.1515/ncrs-2024-0198
Received May 4, 2024; accepted May 23, 2024;
published online June 18, 2024

Abstract

$C_{51}H_{33}F_{18}O_8P_2Sm$, monoclinic, $P2_1/c$ (no. 14), a = 12.8193(9) Å, b = 13.4721(8) Å, c = 30.280(2) Å, β = 91.227(3)°, V = 5,228.2(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0370, $wR_{ref}(F^2)$ = 0.1025, T = 100.0 K.

CCDC no.: 2351578

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.40 × 0.30 × 0.25 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.30 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	172,838, 11,988, 0.089
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 9,105
$N(param)_{refined}$:	751
Programs:	Bruker,[1] SHELX, [2,4] Olex2 [3]

***Corresponding author: Qiang Liu**, College of Biology and Pharmaceutical Engineering, Jilin Agricultural Science and Technology University, 132101 Jilin, China, E-mail: jlnkliu@163.com. https://orcid.org/0009-0006-0399-1083
Wanbin Li, Jinke Jiang and Xin Wang, College of Biology and Pharmaceutical Engineering, Jilin Agricultural Science and Technology University, 132101 Jilin, China

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.0390 (3)	0.2574 (3)	0.68588 (14)	0.0271 (9)
C2	0.0215 (3)	0.2907 (3)	0.64508 (12)	0.0208 (8)
C3	−0.0380 (3)	0.3073 (3)	0.60621 (13)	0.0244 (8)
H3	−0.1108	0.2938	0.6058	0.029*
C4	0.0086 (3)	0.3432 (3)	0.56846 (13)	0.0218 (8)
C5	−0.0636 (3)	0.3635 (4)	0.52794 (14)	0.0308 (9)
C6	0.3593 (3)	0.6359 (3)	0.54033 (14)	0.0300 (9)
C7	0.3322 (3)	0.5256 (3)	0.53772 (13)	0.0209 (8)
C8	0.3374 (3)	0.4777 (3)	0.49681 (13)	0.0240 (8)
H8	0.3558	0.5140	0.4712	0.029*
C9	0.3155 (3)	0.3767 (3)	0.49372 (12)	0.0215 (8)
C10	0.3009 (3)	0.3319 (3)	0.44761 (13)	0.0271 (8)
C11	0.6309 (4)	0.3244 (5)	0.62619 (16)	0.0463 (14)
C12	0.5189 (3)	0.2878 (3)	0.63363 (14)	0.0284 (9)
C13	0.5044 (3)	0.2214 (4)	0.66784 (14)	0.0325 (10)
H13	0.5627	0.2008	0.6854	0.039*
C14	0.4055 (3)	0.1842 (3)	0.67698 (12)	0.0218 (8)
C15	0.3961 (3)	0.1033 (3)	0.71196 (13)	0.0256 (8)
C16	0.2521 (3)	0.4608 (3)	0.74992 (12)	0.0203 (7)
C17	0.2337 (3)	0.3592 (3)	0.75557 (14)	0.0270 (9)
H17	0.2237	0.3174	0.7306	0.032*
C18	0.2302 (3)	0.3201 (3)	0.79784 (14)	0.0304 (9)
H18	0.2182	0.2511	0.8017	0.036*
C19	0.2441 (4)	0.3807 (3)	0.83450 (14)	0.0327 (10)
H19	0.2405	0.3534	0.8633	0.039*
C20	0.2632 (3)	0.4807 (3)	0.82910 (13)	0.0308 (9)
H20	0.2735	0.5221	0.8542	0.037*
C21	0.2673 (3)	0.5212 (3)	0.78678 (13)	0.0249 (8)
H21	0.2806	0.5901	0.7831	0.030*
C22	0.1194 (3)	0.5398 (3)	0.67927 (13)	0.0214 (8)
C23	0.0936 (3)	0.5531 (3)	0.63445 (13)	0.0238 (8)
H23	0.1460	0.5476	0.6129	0.029*
C24	−0.0086 (3)	0.5742 (3)	0.62157 (14)	0.0272 (9)
H24	−0.0262	0.5822	0.5911	0.033*
C25	−0.0852 (3)	0.5836 (3)	0.65299 (14)	0.0304 (9)
H25	−0.1553	0.5971	0.6441	0.037*
C26	−0.0593 (3)	0.5732 (4)	0.69747 (15)	0.0384 (11)
H26	−0.1112	0.5813	0.7191	0.046*
C27	0.0429 (3)	0.5510 (4)	0.71037 (14)	0.0329 (10)
H27	0.0602	0.5435	0.7408	0.040*
C28	0.3280 (3)	0.6190 (3)	0.69280 (12)	0.0207 (8)
C29	0.2923 (3)	0.7065 (3)	0.71240 (14)	0.0283 (9)
H29	0.2253	0.7081	0.7252	0.034*
C30	0.3542 (4)	0.7904 (3)	0.71315 (16)	0.0369 (11)
H30	0.3300	0.8496	0.7265	0.044*
C31	0.4525 (4)	0.7881 (3)	0.69419 (15)	0.0361 (11)
H31	0.4956	0.8454	0.6951	0.043*
C32	0.4873 (4)	0.7022 (3)	0.67402 (15)	0.0348 (10)
H32	0.5536	0.7012	0.6606	0.042*
C33	0.4251 (3)	0.6175 (3)	0.67338 (14)	0.0270 (9)
H33	0.4491	0.5586	0.6597	0.032*
C34	0.1652 (3)	−0.0167 (3)	0.61110 (13)	0.0223 (8)
C35	0.1315 (3)	0.0299 (3)	0.64927 (14)	0.0287 (9)
H35	0.1418	0.0992	0.6531	0.034*
C36	0.0829 (4)	−0.0246 (4)	0.68168 (16)	0.0409 (12)
H36	0.0614	0.0073	0.7080	0.049*
C37	0.0656 (4)	−0.1251 (4)	0.67605 (16)	0.0391 (11)
H37	0.0307	−0.1618	0.6981	0.047*
C38	0.0988 (4)	−0.1722 (3)	0.63858 (17)	0.0368 (11)
H38	0.0880	−0.2415	0.6351	0.044*
C39	0.1485 (3)	−0.1182 (3)	0.60565 (15)	0.0294 (9)
H39	0.1707	−0.1507	0.5796	0.035*
C40	0.3647 (3)	0.0297 (3)	0.57120 (13)	0.0224 (8)
C41	0.4061 (3)	−0.0538 (3)	0.59245 (14)	0.0267 (9)
H41	0.3619	−0.0968	0.6085	0.032*
C42	0.5119 (3)	−0.0740 (3)	0.59010 (15)	0.0319 (10)
H42	0.5399	−0.1313	0.6043	0.038*
C43	0.5770 (3)	−0.0114 (3)	0.56728 (14)	0.0281 (9)
H43	0.6493	−0.0262	0.5656	0.034*
C44	0.5369 (3)	0.0731 (3)	0.54681 (14)	0.0295 (9)
H44	0.5820	0.1168	0.5317	0.035*
C45	0.4307 (3)	0.0937 (3)	0.54850 (14)	0.0252 (8)
H45	0.4031	0.1511	0.5343	0.030*
C46	0.1785 (3)	0.0315 (3)	0.51598 (12)	0.0218 (8)
C47	0.1257 (3)	0.1066 (4)	0.49337 (14)	0.0339 (10)
H47	0.1148	0.1690	0.5072	0.041*
C48	0.0890 (4)	0.0901 (5)	0.45038 (16)	0.0471 (13)
H48	0.0537	0.1416	0.4347	0.056*
C49	0.1034 (4)	0.0001 (5)	0.43058 (15)	0.0476 (14)
H49	0.0771	−0.0108	0.4014	0.057*
C50	0.1563 (4)	−0.0753 (4)	0.45277 (16)	0.0421 (13)
H50	0.1661	−0.1375	0.4387	0.051*
C51	0.1952 (3)	−0.0601 (3)	0.49568 (14)	0.0307 (9)
H51	0.2325	−0.1113	0.5108	0.037*
F1	0.0243 (2)	0.2248 (3)	0.71790 (10)	0.0567 (9)
F2	−0.1038 (3)	0.1829 (2)	0.67658 (10)	0.0541 (9)
F3	−0.0955 (3)	0.3290 (2)	0.70225 (10)	0.0568 (9)
F4	−0.0206 (2)	0.3335 (2)	0.49080 (8)	0.0447 (7)
F5	−0.0828 (2)	0.4606 (2)	0.52377 (10)	0.0469 (7)
F6	−0.1566 (2)	0.3199 (3)	0.53083 (10)	0.0549 (9)
F7	0.4439 (2)	0.6498 (2)	0.56637 (10)	0.0479 (8)
F8	0.2814 (2)	0.68766 (19)	0.55757 (10)	0.0470 (7)
F9	0.3794 (2)	0.6778 (2)	0.50152 (9)	0.0434 (7)
F10	0.3363 (2)	0.3879 (2)	0.41476 (8)	0.0407 (7)
F11	0.1983 (2)	0.3177 (2)	0.43881 (8)	0.0393 (6)
F12	0.3468 (2)	0.2432 (2)	0.44438 (8)	0.0394 (6)
F13[a]	0.6353 (6)	0.4154 (8)	0.6114 (7)	0.079 (6)
F14[b]	0.6733 (11)	0.273 (2)	0.5933 (8)	0.097 (9)
F15[c]	0.6943 (5)	0.3174 (13)	0.6608 (3)	0.078 (5)
F16	0.4865 (2)	0.0764 (2)	0.73089 (10)	0.0474 (8)
F17	0.3549 (3)	0.0219 (2)	0.69421 (10)	0.0533 (9)
F18	0.3338 (2)	0.1306 (2)	0.74416 (9)	0.0468 (7)
O1	0.1177 (2)	0.30045 (19)	0.65074 (8)	0.0204 (5)
O2	0.1025 (2)	0.3647 (2)	0.56262 (9)	0.0218 (6)
O3	0.3066 (2)	0.48902 (19)	0.57424 (9)	0.0221 (6)
O4	0.3022 (2)	0.31569 (19)	0.52426 (8)	0.0211 (5)
O5	0.4524 (2)	0.3217 (2)	0.60646 (9)	0.0224 (6)
O6	0.3204 (2)	0.2090 (2)	0.65909 (9)	0.0228 (6)
O7	0.2948 (2)	0.43052 (19)	0.66324 (8)	0.0196 (5)
O8	0.2093 (2)	0.16784 (19)	0.58018 (8)	0.0201 (5)
P1	0.25195 (8)	0.50705 (7)	0.69407 (3)	0.01806 (19)
P2	0.22798 (7)	0.05981 (7)	0.57076 (3)	0.01752 (19)

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
Sm1	0.26477 (2)	0.32591 (2)	0.60233 (2)	0.01607 (6)
F14A[d]	0.6552 (10)	0.339 (3)	0.5862 (5)	0.071 (11)
F13A[e]	0.6393 (12)	0.4152 (15)	0.6436 (11)	0.114 (9)
F15A[f]	0.6994 (10)	0.253 (2)	0.6405 (11)	0.064 (8)

[a]Occupancy: 0.58(3), [b]occupancy: 0.64(5), [c]occupancy: 0.72(3), [d]occupancy: 0.36(5), [e]occupancy: 0.42(3), [f]occupancy: 0.28(3).

1 Source of material

The hexafluoroacetylacetone **(6 FAAT)** (0.312 g, 1.5 mmol) was dissolved in 10 ml ethanol, and 1 ml aqueous solution of samarium acetate monohydrate (0.164 g, 0.5 mmol) was added dropwise into the solution. The mixtures were stirred for 2 h at room temperature, and then add 10 ml of ethanol solution of triphenylphosphine oxide (**TPPO**) (0.279 g, 1.0 mmol). After stirring overnight and heating to concentrate the solution, a crystalline precipitate is formed. The complexes were dissolved in ethyl acetate/ethanol (1:1) and stilled several days. Crystals were obtained in the form of single crystals which are suitable for X-ray measurements.

2 Experimental details

The data were collected using Bruker,[1] the structure of crystal was solved by SHELXT[2] program within Olex2[3] and refined by SHELXL.[4]

3 Comment

Rare earth organic complexes have extensive application value in fields such as fluorescence, biology, and magnetic materials, and have received increasing attention.[5–8] The title compound consists of three **FAAT** anions, two **TPPO**s, and one metal samarium(III) ion. The action of **TPPO** is to saturate the remaining coordination number of samarium ions.[9]

Acknowledgment: This work was supported by the Jilin Agricultural Science and Technology University Students Science and Technology Innovation Training Program Project (GJ202211439019) and the Key Discipline of Biology.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

References

1. Bruker. *SAINT and SADABS*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2000.
2. Sheldrick, G. M. *SHELXT* – Integrated Space-Group and Crystal-Structure Determination. *Acta Crystallogr.* **2015**, *A71*, 3–8.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. OLEX2: A Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Crystallogr.* **2009**, *42*, 339–341.
4. Sheldrick, G. M. Crystal Structure Refinement with *SHELXL*. *Acta Crystallogr.* **2015**, *C71*, 3–8.
5. Binnemans, K. Lanthanide-Based Luminescent Hybrid Materials. *Chem. Rev.* **2009**, *109*, 4283–4374.
6. Aulsebrook, M. L.; Graham, B.; Grace, M. R.; Tuck, K. L. Lanthanide Complexes for Luminescence-Based Sensing of Low Molecular Weight Analytes. *Coord. Chem. Rev.* **2018**, *375*, 191–220.
7. Ortu, F. Rare Earth Starting Materials and Methodologies for Synthetic Chemistry. *Chem. Rev.* **2022**, *122*, 6040–6116.
8. Richardson, G. M.; Howarth, J.; Evans, M. J.; Edwards, A. J.; Cameron, S. A.; Anker, M. D. Reactivity of a β-Diketiminate Ytterbium(II) Hydride with Cyclopentadiene Derivatives. *J. Coord. Chem.* **2022**, *75*, 1954–1966.
9. Liu, Q.; Cai, P. J.; Wan, X. Y.; Zhang, S.; Du, K.; Yin, Q. Neodymium Ternary Complexes with 2,4,6-Tri(2-Pyridyl)-1,3,5-Triazine: Synthesis, Crystal Structure and Fluorescence Property. *J. Mol. Struct.* **2019**, *1193*, 151–157.