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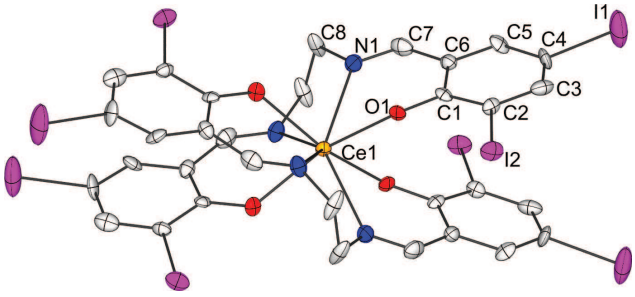
Crystal structure of bis(6,6′-((ethane-1,2-diylbis(azanylylidene))bis(methanylylidene))bis(2,4-diiodophenolato-κ^4*N*,*N*′,*O*,*O*′)cer(IV), $C_{32}H_{20}CeI_8N_4O_4$



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Abstract

$C_{32}H_{20}CeI_8N_4O_4$, tetragonal, $P\bar{4}b2$ (no. 117), $a = 11.1898(8)$ Å, $b = 11.1898(8)$ Å, $c = 16.6870(19)$ Å, $V = 2089.4(4)$ Å^3, $Z = 2$; $R_{gt}(F) = 0.0566$, $wR_{ref}(F^2) = 0.0939$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All commercially available reagents were used as supplied. The title compound was synthesized with the following procedure. 3,5-diiodosalicylaldehyde 0.0350 g (0.094 mmol) and 0.003 mL ethylendiamine (0.047 mmol) were stirred in a mixture of 20 mL ethanol and 10 mL DMF at room temperature for 20 min. Then, 0.1 g cerium(III) nitrate was added to the above solution and the resulting yellow mixture was further stirred for another 30 min and filtered. The blocked-shaped crystals of the title compound were obtained after 3 days by slow evaporation.

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Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.10 × 0.18 × 0.24 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	7.04 mm^{-1}
Diffractometer, scan mode:	Multiwire proportional, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8392, 1844, 0.092
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1440
$N(param)_{refined}$:	112
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3], Diamond [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ce1	0.0000	0.5000	0.0000	0.0242(3)
I1	0.01371(14)	0.23830(14)	0.44341(7)	0.1072(6)
I2	0.30338(8)	0.55816(9)	0.22720(7)	0.0456(3)
O1	0.1181(8)	0.4406(7)	0.1025(5)	0.028(2)
N1	−0.0731(10)	0.2970(9)	0.0539(7)	0.037(3)
C1	0.0991(11)	0.3973(12)	0.1740(7)	0.025(3)
C2	0.1664(11)	0.4358(11)	0.2386(9)	0.029(3)
C3	0.1426(12)	0.3903(12)	0.3168(10)	0.044(4)
H3	0.1898	0.4148	0.3596	0.053*
C4	0.0501(15)	0.3102(14)	0.3299(9)	0.047(5)
C5	−0.0096(11)	0.2670(11)	0.2668(8)	0.036(3)
H5	−0.0662	0.2072	0.2746	0.043*
C6	0.0114(15)	0.3101(11)	0.1892(8)	0.031(4)
C7	−0.0651(13)	0.2625(13)	0.1260(10)	0.044(4)
H7	−0.1141	0.1990	0.1404	0.052*
C8	−0.1700(13)	0.2392(11)	0.0072(10)	0.054(5)
H8A	−0.1392	0.2091	−0.0433	0.064*
H8B	−0.2037	0.1730	0.0371	0.064*

Comment

The investigation on lanthanide complexes represents one of the most active areas of material science and environmental chemistry [5, 6]. In this field, a large number of tetradentate Schiff-base ligands have been introduced for the isolation of lanthanide complexes with different structures and

properties [7, 8]. However, up to present, related research about cerium(VI) complexes constructed from halogenated Schiff base ligands are still rare. In order to enrich the knowledge on halogenated Schiff base cerium(IV) complexes the title compound was synthesised and characterised.

Single-crystal X-ray diffraction reveals that the title compound crystallizes in the tetragonal space group. The cerium atom is located on a twofold special position of this space group in a distorted square antiprism environment. The coordination is defined by four oxygen atoms and four nitrogen atoms from two tetradentate ligands (*cf*. the figure). According to the analysis of result of *PLATON* software, the crystal structure is stabilized by weak C—I$\cdots\pi$ and face-to-face π-π stacking interactions [C2—I2$\cdots$*Cg*1 ($1/2-x$, $1/2+y$, z), I$\cdot$*Cg* 3.574(6) Å; *Cg*1$\cdots$*Cg*1($-x$, $1-y$, z), centroid-to-centroid distance = 3.734(8) Å, where *Cg*1 is the centroid of the C1—C6 ring].

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References

1. Agilent Technologies: CrysAlis^{PRO} Software system, version 1.171.38.41. Agilent Technologies UK Ltd, Oxford, UK (2015).
2. 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. **42** (2009) 339–341.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
4. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 3.2i. Crystal Impact, Bonn, Germany (2012).
5. Kong, L.; Tang, J.; Liu, J.; Wang, Y.; Wang, L.; Cong, F.: Fluorescent nanoblocks of lanthanide complexes on nano silicon dioxide and carbon nanotube donors with ligand–antenna integration (ALI) structure. Mater. Sci. Eng. C **29** (2009) 85–91.
6. Shang, Q.; Li, Y.; Yang, E. C.; Zhao, X. J.: Three isostructural 4–sulfophthalate–based lanthanide complexes: syntheses, crystal structures, and luminescent properties. Z. Anorg. Allg. Chem. **641** (2015) 2515–2519.
7. Gao, B.; Zhang, Q.; Yan, P.; Hou, G.; Li, G.: Crystal engineering of salen type cerium complexes tuned by various cerium counterions. CrystEngComm **15** (2013) 4167–4175.
8. Kubono, K.; Hirayama, N.; Kokusen, H.; Yokoi, K.: X-ray crystallographic approach to the design of phenolic schiff base reagents for the mutual separation of lanthanoids. Anal. Sci. **17** (2001) 193–197.