

КРАТКИЕ СООБЩЕНИЯ

UDC 548.73:547.13:546.65

YTTRIUM AND SAMARIUM COMPLEXES WITH A LINKED
1,4,7-TRIAZACYCLONONANE-ARYLOXIDE ANCILLARY LIGANDJ. Cui^{1,2}, I.C. Santos^{1,3}, J.M. Carretas¹¹*Centro de Ciências e Tecnologias Nucleares, Instituto Superior Técnico, Universidade de Lisboa, 2696-066, Bobadela LRS, Portugal*

E-mail: carretas@ctn.ist.utl.pt

²*Department of Chemistry and Biochemistry, 251 Nieuwland Science Hall, University of Notre Dame, Notre Dame, USA*³*Centro de Química Estrutural, Instituto Superior Técnico, Universidade de Lisboa, Lisboa, Portugal*

Received May, 8, 2013

The reaction of yttrium trichloride with the stoichiometric amount of the potassium salt of the 1-(3,5-di-*tert*-butyl-2-hydroxybenzyl)-4,7-diisopropyl-1,4,7-triazacyclononane ligand (ⁱPr₂—TACN—CH₂C₆H₂^tBu₂OH) (TACN = 1,4,7-triazacyclononane) in DME affords in good yield complex [ⁱPr₂—TACN—CH₂C₆H₂^tBu₂O]YCl₂ (**1**). This complex is characterized by elemental analysis and ¹H nuclear magnetic resonance; the solid state structure is determined by the single crystal X-ray diffraction analysis. In complex **1**, the metal centre is six-coordinated by the aryloxy oxygen atom, the three nitrogen atoms of the TACN ligand, and two chlorine atoms. The X-ray structure of isomorphous samarium complex [ⁱPr₂—TACN—CH₂C₆H₂^tBu₂O]SmCl₂ (**2**) is also determined.

Keywords: 1,4,7-triazacyclononane (TACN), yttrium, samarium, synthesis, crystal structures.

1,4,7-Triazacyclonane ligands have been successfully used as 6-electron fac-tridentate ligands for a wide range of metals, and various neutral and anionic TACN derivatives have appeared in the literature [1—12]. This macrocycle can be derivatized at the nitrogen atoms to give access to ligands with one or more pendant functionalities. Hence, these pendant-arm macrocycles offer the ability to modify and tune the electronic and stereochemical properties of a range of metal centres, and may be regarded as potent protecting groups for catalytic reactions [13—15]. We were therefore interested to explore the potential of the aryloxy functionality as a pendant arm linked with the 1,4,7-triazacyclonane ligand in the chemistry of rare earth elements. Only a few complexes with monoanionic TACN macrocycles with aryloxy pendant arms have been reported [5, 10] but none involving the rare earth metals.

This work reports the synthesis and structural characterization of an Y(III) complex with a linked 1,4,7-triazacyclonane-aryloxy ligand: [ⁱPr₂—TACN—CH₂C₆H₂^tBu₂O]YCl₂. The solid state structure of the analogous Sm(III) complex [ⁱPr₂—TACN—CH₂C₆H₂^tBu₂O]SmCl₂ has also been determined.

Experimental. All preparations and subsequent manipulations were carried out using the standard Schlenk and glove-box techniques in a dinitrogen atmosphere. 1,2-Dimethoxyethane, *n*-hexane, and tetrahydrofuran were distilled from sodium benzophenone under nitrogen. Dichloromethane was distilled from P₂O₅. All solvents were degassed prior to use. CDCl₃ was dried over P₂O₅ and distilled. YCl₃(THF)_{2,5} [16], SmCl₃ [17], and the ⁱPr₂—TACN—CH₂C₆H₂^tBu₂OH ligand precursor [18] were

prepared as described in the literature. $\text{KOC}_6\text{H}_2^i\text{Bu}_2\text{CH}_2\text{—TACN—}^i\text{Pr}_2$ was prepared by the reaction of $^i\text{Pr}_2\text{—TACN—CH}_2\text{C}_6\text{H}_2^i\text{Bu}_2\text{OH}$ with an excess of KH in THF.

Carbon, hydrogen, and nitrogen analyses were performed in-house using an automatic analyser CE Instruments EA 1110. ^1H nuclear magnetic resonance (NMR) spectra were recorded on a Varian 300 MHz spectrometer. ^1H spectra were referenced internally to residual protio-solvent (^1H) resonances and reported relative to tetramethylsilane.

Synthesis of yttrium and samarium complexes. To a suspension of $\text{YCl}_3(\text{THF})_{2.5}$ (0.290 g, 0.773 mmol) in DME a solution of $\text{KOC}_6\text{H}_2^i\text{Bu}_2\text{CH}_2\text{—TACN—}^i\text{Pr}_2$ (0.363 g, 0.773 mmol) in the same solvent was slowly added. After stirring overnight at room temperature, KCl was separated by centrifugation and the solvent was removed under vacuum. The white solid was washed with *n*-hexane and dried. The compound was crystallized from a concentrated dichloromethane solution to give crystals suitable for the X-ray diffraction analysis. Yield: 85.3 % (0.389 mg, 0.659 mmol). $\text{YCl}_2\text{C}_{27}\text{H}_{48}\text{N}_3\text{O} \cdot 2\text{CH}_2\text{Cl}_2$ requires C, 45.81 %; H, 6.89 %; N, 5.53 %. Found: C, 45.98 %; H, 7.01 %; N, 5.42 %. ^1H NMR (δ ; ppm; CDCl_3 ; 293 K) 7.21 (d, 1H, $J_{\text{HH}} = 2.4$ Hz, $\text{C}_6\text{H}_2^i\text{Bu}_2$), 6.79 (d, 1H, $J_{\text{HH}} = 2.4$ Hz, $\text{C}_6\text{H}_2^i\text{Bu}_2$), 4.85 (d, 1H, $J_{\text{HH}} = 12.9$ Hz, NCH_2Ph), 4.12 (sept, 1H, $J_{\text{HH}} = 6.6$ Hz, $^i\text{Pr CH}$), 3.92 (sept, 1H, $J_{\text{HH}} = 6.6$ Hz, $^i\text{Pr CH}$), 3.59 (m, 1H, NCH_2), 3.32 (d, 1H, $J_{\text{HH}} = 12.9$ Hz, NCH_2Ph), 3.30—2.50 (overlapping, m, 10H, NCH_2), 2.40 (m, 1H, NCH_2), 1.60 (d, 6H, $J_{\text{HH}} = 6.6$ Hz, $^i\text{Pr Me}$), 1.43 (s, 9H, $\text{C}_6\text{H}_2^i\text{Bu}_2$), 1.24 (s, 9H, $\text{C}_6\text{H}_2^i\text{Bu}_2$), 1.06 (d, 6H, $J_{\text{HH}} = 6.6$ Hz, $^i\text{Pr Me}$).

To a suspension of SmCl_3 (0.125 g, 0.486 mmol) in DME a solution of $\text{KOC}_6\text{H}_2^i\text{Bu}_2\text{CH}_2\text{—TACN—}^i\text{Pr}_2$ (0.228 g, 0.486 mmol) in the same solvent was added. The work-up was similar to that described above and crystals suitable for X-ray diffraction were also obtained.

Table 1

Crystallographic data for $1 \cdot 2\text{CH}_2\text{Cl}_2$ and $2 \cdot 2\text{CH}_2\text{Cl}_2$

Parameter	$1 \cdot 2\text{CH}_2\text{Cl}_2$	$2 \cdot 2\text{CH}_2\text{Cl}_2$
Empirical formula	$\text{C}_{29}\text{H}_{52}\text{Cl}_6\text{N}_3\text{OY}$	$\text{C}_{29}\text{H}_{52}\text{Cl}_6\text{N}_3\text{OSm}$
Formula weight, g	760.35	821.79
Color / shape	Colourless / prism	Colourless / prism
Temperature, K	150(2)	150(2)
Wavelength	0.71073	0.71069
Crystal system	Monoclinic	Monoclinic
Space group	P_{21}/c	P_{21}/c
Unit cell dimensions <i>a</i> , <i>b</i> , <i>c</i> , Å; β , deg.	17.8418(9), 13.5777(7), 15.7961(8); 109.677(2)	17.8610(4), 13.6270(3) 15.8130(4); 109.7910(10)
Volume, Å ³	3603.2(3)	3621.42(15)
<i>Z</i>	4	4
Density calc., g/cm ⁻³	1.402	1.507
Absorption coefficient, mm ⁻¹	2.090	2.090
<i>F</i> (000)	1584	1676
Crystal size, mm	0.18×0.16×0.14	0.20×0.18×0.10
θ range for data collection, deg.	2.59—25.32	2.78—26.37
Reflections collected / unique	25866 / 6834 [$R_{\text{int}} = 0.0712$]	68298 / 7388 [$R_{\text{int}} = 0.0439$]
Completeness to θ , deg.	0.998 (25.52°)	0.998 (26.37°)
Transmission max / min	0.7585 / 0.7048	0.8182 / 0.6799
Data / restraints / parameters	6834 / 0 / 371	7388 / 0 / 371
Goodness-of-fit on F^2	0.984	1.046
<i>R</i> indices [$I > 2\sigma(I)$]	$R_1 = 0.0422$, $wR_2 = 0.0822$	$R_1 = 0.0222$, $wR_2 = 0.0532$
<i>R</i> indices (all data)	$R_1 = 0.0735$, $wR_2 = 0.0895$	$R_1 = 0.0275$, $wR_2 = 0.0550$
Largest difference peak and hole, Å ⁻³	-0.472 / 0.654	-0.847 / 0.958

Table 2

Selected bond lengths (Å) and angles (deg.) for $1 \cdot 2\text{CH}_2\text{Cl}_2$ and $2 \cdot 2\text{CH}_2\text{Cl}_2$

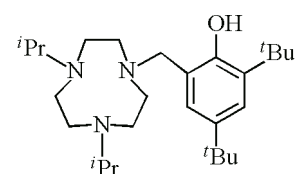
1 · 2CH ₂ Cl ₂										
Y ₁ —N ₁	2.498(2)	O ₁ —Y ₁ —N ₁	77.34(8)	Cl ₁ —Y ₁ —N ₁	100.32(6)	N ₁ —Y ₁ —N ₂	71.28(8)			
Y ₁ —N ₂	2.575(3)	O ₁ —Y ₁ —N ₂	147.92(8)	Cl ₁ —Y ₁ —N ₂	85.07(6)	N ₁ —Y ₁ —N ₃	69.87(8)			
Y ₁ —N ₃	2.581(2)	O ₁ —Y ₁ —N ₃	104.15(8)	Cl ₁ —Y ₁ —N ₃	156.20(7)	N ₂ —Y ₁ —N ₃	71.31(8)			
Y ₁ —Cl ₁	2.5769(8)	O ₁ —Y ₁ —Cl ₁	94.25(6)	Cl ₂ —Y ₁ —N ₁	157.71(6)	N ₁ —C ₁₃ —C ₁₄	117.1(3)			
Y ₁ —Cl ₂	2.5933(9)	O ₁ —Y ₁ —Cl ₂	98.03(6)	Cl ₂ —Y ₁ —N ₂	113.55(6)	Y ₁ —O ₁ —C ₁₉	145.2(2)			
Y ₁ —O ₁	2.080(2)	Cl ₁ —Y ₁ —Cl ₂	101.77(3)	Cl ₂ —Y ₁ —N ₃	90.62(6)					
2 · 2CH ₂ Cl ₂										
Sm ₁ —N ₁	2.5713(18)	O ₁ —Sm ₁ —N ₁	75.56(6)	Cl ₁ —Sm ₁ —N ₁	100.08(4)	N ₁ —Sm ₁ —N ₂	69.81(6)			
Sm ₁ —N ₂	2.6186(18)	O ₁ —Sm ₁ —N ₂	144.71(6)	Cl ₁ —Sm ₁ —N ₂	85.69(4)	N ₁ —Sm ₁ —N ₃	67.76(6)			
Sm ₁ —N ₃	2.6424(18)	O ₁ —Sm ₁ —N ₃	103.62(6)	Cl ₁ —Sm ₁ —N ₃	154.75(4)	N ₂ —Sm ₁ —N ₃	69.47(6)			
Sm ₁ —Cl ₁	2.6392(6)	O ₁ —Sm ₁ —Cl ₁	93.94(4)	Cl ₂ —Sm ₁ —N ₁	156.31(4)	N ₁ —C ₁₃ —C ₁₄	117.60(18)			
Sm ₁ —Cl ₂	2.6559(6)	O ₁ —Sm ₁ —Cl ₂	98.84(4)	Cl ₂ —Sm ₁ —N ₂	115.63(4)	Sm ₁ —O ₁ —C ₁₉	146.80(13)			
Sm ₁ —O ₁	2.1257(15)	Cl ₁ —Sm ₁ —Cl ₂	103.29(2)	Cl ₂ —Sm ₁ —N ₃	91.92(4)					

Crystal structure determination. Colorless crystals of [ⁱPr₂—TACN—CH₂C₆H₂^tBu₂O]YCl₂ · 2CH₂Cl₂ and [ⁱPr₂—TACN—CH₂C₆H₂^tBu₂O]SmCl₂ · 2CH₂Cl₂ suitable for X-ray diffraction were both obtained from dichloromethane. A summary of the crystallographic data and structure refinements is shown in Table 1, and selected bond lengths and angles of the complexes are listed in Table 2. Crystallographic data were collected using graphite-monochromated MoK_α (α = 0.71073 Å) radiation on a Bruker AXS APEX II area detector diffractometer equipped with an Oxford Cryosystem open-flow nitrogen cryostat, and data were collected at 150 K. Cell parameters were retrieved using the Bruker SMART software and refined using Bruker SAINT on all observed reflections. Absorption corrections were applied using SADABS [19]. The structures were solved by direct methods using SIR 97 [20] and refined using the full-matrix least squares refinement against F² using SHELXL-97 [21]. All programs are included in the WINGX-version 1.64.05 package of programs [22]. All non-hydrogen atoms were refined anisotropically and all hydrogen atoms were placed in idealized positions and allowed to refine riding on the parent carbon atom. The molecular structures were drawn with ORTEP3 for Windows [22].

Data for the complexes were deposited in CCDC under the deposit numbers 905740 and 93207 and can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Results and discussion. The reaction of yttrium trichloride with the potassium salt of the 1-(3,5-di-*tert*-butyl-2-hydroxybenzyl)-4,7-diisopropyl-1,4,7-triazacyclononane) ligand (Fig. 1) in DME gave a good yield of a white product, insoluble in hydrocarbon solvents and soluble in polar solvents such as DME, THF, or dichloromethane. The results of the elemental analysis of the crystals are compatible with the formulation [ⁱPr₂—TACN—CH₂C₆H₂^tBu₂O]YCl₂ · 2CH₂Cl₂. The ¹H NMR spectrum at room temperature corroborated the existence of this complex. Of special note is the presence of two resonances at 4.85 ppm and 3.32 ppm assigned to the benzylic protons NCH₂Ph, instead of only one resonance at 3.76 ppm present the coordination of the aryloxide arm. The observation of two resonances for the CH protons of the isopropyl groups shows that the complex displays a C₁ symmetry in the solution, which is consistent with the solid state structure.

The reaction of the same ligand KOC₆H₂^tBu₂CH₂—TACN—ⁱPr₂ with samarium trichloride in DME was also studied and led to a product which was not fully characterized. The elemental analysis data revealed a solid not

Fig. 1. Ligand precursor (ⁱPr₂—TACN—CH₂C₆H₂^tBu₂OH)

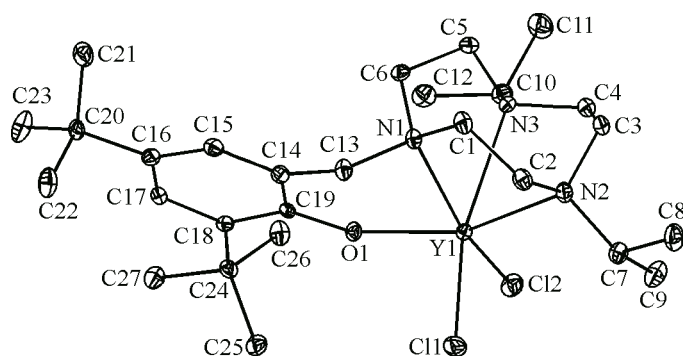


Fig. 2. ORTEP diagram of $[\text{}^i\text{Pr}_2\text{—TACN—CH}_2\text{C}_6\text{H}_2\text{}^t\text{Bu}_2\text{O}]\text{YCl}_2 \cdot 2\text{CH}_2\text{Cl}_2$ with 40 % probability ellipsoids

completely pure, and due to samarium paramagnetism no resolvable NMR spectrum was obtained. Nevertheless, crystals suitable for X-ray diffraction were obtained.

The $[\text{}^i\text{Pr}_2\text{—TACN—CH}_2\text{C}_6\text{H}_2\text{}^t\text{Bu}_2\text{O}]\text{YCl}_2$ complex crystallized from dichloromethane in the $P21/c$ space group with two molecules of dichloromethane included in the lattice. The X-ray molecular structure is shown in Fig. 2. The molecule consists of a monomeric six-coordinated complex with the yttrium atom surrounded by two chlorine atoms, the oxygen atom of the aryloxy arm, and three nitrogen atoms of the TACN moiety in a distorted octahedral configuration. A clear view of the inner coordination is given in Fig. 3. The three nitrogen atoms of the TACN moiety occupy a triangular face while the two chlorine and one oxygen atom form a staggered triangular face on the other side of the central yttrium atom. The two staggered triangular faces are not parallel but are inclined at an angle of $13.12(5)^\circ$, showing therefore a considerable distortion. The asymmetry in the structure of the complex is also revealed, for example, in the angle $\text{N}(1)\text{—Y}(1)\text{—Cl}(1)$ $100.32(6)^\circ$ vs $\text{N}(1)\text{—Y}(1)\text{—Cl}(2)$ $157.71(6)^\circ$. This may be associated with minimizing the steric interference between the chloride ligands and the ${}^i\text{Pr}$ groups on the TACN moiety. The asymmetry retained in the solution, as displayed by ${}^1\text{H}$ NMR, is consistent with these structural considerations. The Y—N distances for the TACN nitrogens are: $\text{Y}_1\text{—N}_1 = 2.498(2)$ Å, $\text{Y}_1\text{—N}_2 = 2.575(3)$ Å, and $\text{Y}_1\text{—N}_3 = 2.581(2)$ Å. The Y—N average distance of $2.551(2)$ Å is longer than the corresponding values in the yttrium complexes with TACN-amide ligands $[\text{}^i\text{Pr}_2\text{—TACN}(\text{CH}_2)_2\text{N}^t\text{Bu}]\text{Y}(\text{CH}_2\text{SiMe}_3)_2$ ($2.463(5)$ Å) and $[\text{}^i\text{Pr}_2\text{—TACNSiMe}_2\text{N}^t\text{Bu}]\cdot\text{Y}(\text{CH}_2\text{SiMe}_3)_2$ ($2.480(4)$ Å) [10]. The Y—O distance could not be compared with analogous values because there are no references in the literature to the existence of yttrium TACN-aryloxy complexes. However, the Y—O distance in the $[\text{}^i\text{Pr}_2\text{—TACN—CH}_2\text{C}_6\text{H}_2\text{}^t\text{Bu}_2\text{O}]\text{YCl}_2$ complex ($2.080(2)$ Å) is comparable to the Y—O average distances in the hexacoordinated complexes $[\text{Y}({}^t\text{Bu}_2\text{O}_2\text{NN}') \times (\text{C}_6\text{H}_4\text{NMe}_2)]$ ($2.149(18)$ Å) and $[\text{Y}({}^t\text{Bu}_2\text{O}_2\text{NN}')\text{(N}(\text{SiMe}_3)_2)]$ ($2.127(4)$ Å) (${}^t\text{Bu}_2\text{O}_2\text{NN}' = \text{Me}_2\text{NCH}_2\text{CH}_2\text{N}(\text{CH}_2\text{OC}_6\text{H}_2\text{}^t\text{Bu}_2)_2$) [23].

The $[\text{}^i\text{Pr}_2\text{—TACN—CH}_2\text{C}_6\text{H}_2\text{}^t\text{Bu}_2\text{O}]\text{SmCl}_2$ complex crystallized from dichloromethane in the $P21/c$ space group with two molecules of dichloromethane included in the lattice. This complex is isostructural with **1** and the X-ray crystal structure of **2** is shown in Fig. 4 with the samarium atom surrounded by two chlorine atoms, the aryloxy oxygen atom, and three nitrogen atoms of TACN.

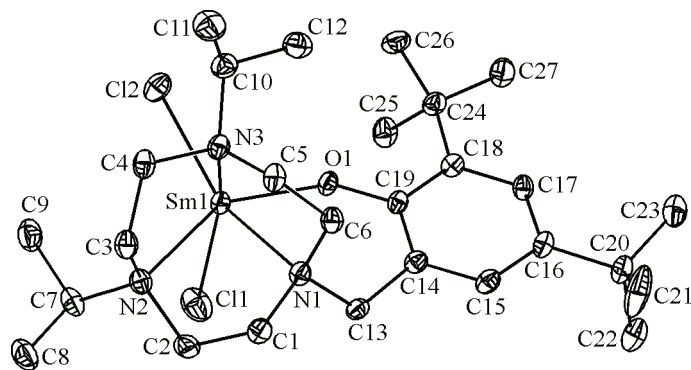


Fig. 3. ORTEP diagram of $[\text{}^i\text{Pr}_2\text{—TACN—CH}_2\text{C}_6\text{H}_2\text{}^t\text{Bu}_2\text{O}]\text{SmCl}_2 \cdot 2\text{CH}_2\text{Cl}_2$ with 40 % probability ellipsoids

There are no references in the literature for Sm—N bond distances in hexacoordinated samarium complexes with a TACN ligand. However, the Sm—N average distance of 2.6108(18) Å in **2** is comparable with the corresponding distances in the nine-coordinated samarium(III) complexes: SmL¹ (L¹ = 1,4,7-Tris(2-aminoethyl)-1,4,7-triazacyclononane) (2.679(4) Å) [24] and SmL² (L² = 1,4,7-Tri[ethyl(6-methylpyridin-2yl(phenyl)phosphinate)]-1,4,7-triazacyclonane) 2.60(2) Å [25].

The bond distances in isomorphous complexes **1** and **2**, with differences of 0.06 Å for M—N, 0.06 Å for M—Cl and 0.05 Å for M—O, are in line with the 0.06 Å difference in the ionic radii of their Y(III) and Sm(III) central metal atoms [26].

Acknowledgements. Jinlan Cui thanks the Fundação para a Ciência e a Tecnologia (FCT) for a post-doc grant (SFRH/BPD/21006/2004). The authors are grateful to Dr. Noémia Marques and Dr. Joaquim Marçalo for their support in this work.

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