

Lanthanide complexes of chiral 3+3 macrocycles derived from (1*R*, 2*R*)-1,2-diaminocyclohexane and 2,6-diformyl-4-methylphenol.

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Streszczenie

The enantiopure amine macrocycle H_3L , as well as the parent macrocyclic Schiff base H_3L1 , the 3 + 3 condensation product of (1*R*,2*R*)-1,2-diaminocyclohexane and 2,6-diformyl-4-methylphenol, are able to form mononuclear complexes with lanthanide(III) ions. The lanthanide(III) complexes of H_3L have been studied in solution using NMR spectroscopy and electrospray mass spectrometry. The NMR spectra indicate the presence of complexes of low C_1 and C_2 symmetry. The 1H and ^{13}C NMR signals of the Lu(III) complex obtained from H_3L have been assigned on the basis of COSY, TOCSY, NOESY, ROESY and HMQC spectra. The NMR data reveal unsymmetrical binding of lanthanide(III) ion and the presence of a dynamic process corresponding to rotation of Lu(III) within the macrocycle. The $[Ln(H_4L)(NO_3)_2](NO_3)_2$ ($Ln = Sm(III), Eu(III), Dy(III), Yb(III)$ and $Lu(III)$) complexes of the cationic ligand H_4L^+ have been isolated in pure form. The X-ray analysis of the $[Eu(H_4L)(NO_3)_2](NO_3)_2$ complex confirms the coordination mode of the macrocycle determined on the basis of NMR results. In this complex the europium(III) ion is bound to three phenolate oxygen atoms and two amine nitrogen atoms of the monoprotonated macrocycle H_4L^+ , as well as to two axial bidentate nitrate anions. In the presence of a base, mononuclear La(III), Ce(III) and Pr(III) complexes of the deprotonated form of the ligand L^{3-} can be obtained. When 2 equivalents of Pr(III) are used in this synthesis $Na_3[Pr_2L(NO_3)_2(OH)_2]_2NO_3 \cdot 5H_2O$ is obtained. The NMR, ES MS and an X-ray crystal model of this complex show coordination of two Pr(III) ions by the macrocycle L. The X-ray crystal structure of the free macrocycle H_3L1 has also been determined. In contrast to macrocyclic amine H_3L , the Schiff base H_3L1 adopts a cone-type conformation resembling calixarenes.

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