Supplementary Material (ESI) for Chemical Communications

Supplementary Information

Lanthanide-induced helical arrays of [{Co(III) sepulchrate} ∩ {*p*-sulfonatocalix[4]arene}] supermolecules

Christopher B. Smith,[a]* Leonard J. Barbour,[b] Mohamed Makha,[a] Colin L. Raston,[a]* Alexandre N. Sobolev[a]

[a]*School of Biomedical, Biomolecular and Chemical Sciences, University of Western Australia, Crawley, W.A. 6009, Australia. Email: cbsmith@cyllene.uwa.edu.au*
[b]*Department of Chemistry, University of Stellenbosch, 7602 Matieland, South Africa.*

Synthesis of I

A hot (~80°) solution of [Co(diOHsar)]Cl_3 **2**[1] (17.1 mg, 3.6×10^{-5} mol) in water (2 cm^3) was added to a hot (~80°) solution of *p*-sulfonatocalix[4]arene tetrasodium salt **1** (10 mg, 1.2×10^{-5} mol) and $Pr(O_3SCF_3)_3$ (14.1 mg, 2.4×10^{-5} mol) in water (2 cm^3). The pH was adjusted to 4-5 using 1M aqueous NaOH solution and the solution cooled slowly over 24 h. Small orange crystals formed (3 mg) which were suitable for X-ray diffraction.

X-Ray crystallography of I

The X-ray diffracted intensities were measured from a single crystal (0.45 x 0.42 x 0.33 mm) at 153 K on a Bruker SMART CCD instrument using a monochromatized Mo-K_α (λ = 0.71073 Å) X-ray source. Data were corrected for Lorentz and polarization effects and absorption correction applied using multiple symmetry equivalent reflections. The structures were solved by direct method and refined on F^2 using Bruker SHELXTL crystallographic package.[2] A full matrix least-squares refinement procedure was used, minimizing $w(F_o^2-F_c^2)$, with $w = [\sigma^2(F_o^2)+(AP)^2+BP]^{-1}$, where $P = (F_o^2+2F_c^2)/3$. Agreement factors ($R = \Sigma||F_o|-|F_c||/\Sigma|F_o|$, $wR2 = \{\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$ and GOF = $\{\Sigma[w(F_o^2-F_c^2)^2]/(n-p)\}^{1/2}$ are cited, where n is the number of reflections and p the total number of parameters refined).

Crystal/refinement details: $C_{42}H_{57.5}CoN_6O_{20.75}Pr_{0.17}S_4$, M = 1189.12, F(000) = 9894 e, Tetragonal, $I4_1/a$ (No. 88), Z = 16, T = 153 K, a = 25.47(5), c = 41.51(7) Å, V = 26929(68) $Å^3$; D_c = 1.173 g cm^{-3}; $\sin\theta/\lambda_{max}$ = 0.587; N(unique) = 10610 (merged from 64020, R_{int} = 0.1624, R_σ = 0.1175), N_o ($I > 2\sigma(I)$) = 5316; R = 0.1695, $wR2$ = 0.3882 (A,B = 0.25, 250.0), GOF = 1.019; $|\Delta\rho_{max}|$ = 2.4(2) e $Å^{-3}$.

1. R. J. Geue, T. W. Hambley, J. M. Harrowfield, A. M. Sargeson and M. R. Snow, *J. Am. Chem. Soc.,* 1984, **106**, 5478.

2. G. M. Sheldrick, SHELX-97: Structure solution and refinement programs, University of Göttingen, 1997; Bruker SMART, SAINT, SADABS & SHELXTL. v5.1., 1997, Bruker AXS Inc., Madison, Wisconsin, USA.