CRYSTAL STRUCTURE OF A 3D COORDINATION POLYMER: SODIUM LANTHANIDE TEREPHTHALATE N, N-DIMETHYLFORMAMIDE SOLVATE

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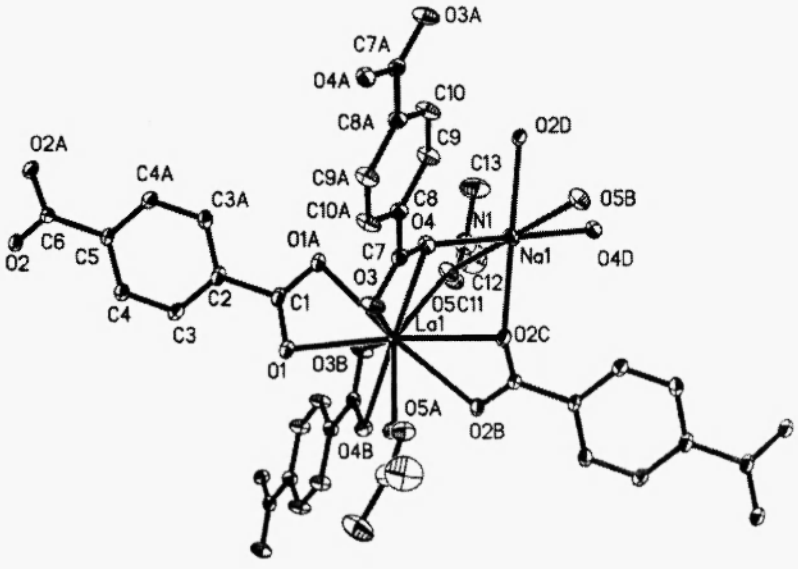

Figure 1. *ORTEP* plot showing coordination environment of lanthanide and sodium ions at the 50% probability level. Hydrogen atoms are omitted for clarity. Selected bond distances and angles: La1-O1 2.5481(16), La1-O3 2.5962(16), La1-O5 2.6078(18), La1-O4 2.6322(17), La1-O2b 2.6711(16), Na1-O4 2.2361(15), Na1-O2e 2.3896(18), Na1-O2b 2.3896(18), Na1-O5d 2.5054(17), Na1-La1d 3.6734(6) Å, O1-La1-O1a 51.38(8), O1-La1-O3 77.99(6), O3-La1-O3a 149.99(9), O1-La1-O5 127.84(6), O3-La1-O5 118.44(5), O5-La1-O5a 147.76(9), O1-La1-O4 110.16(5), O3-La1-O4 49.65(5), O5a-La1-O4 110.90(5), O3a-La1-O4a 49.65(5), O4-La1-O4a 179.15(6), O1-La1-O2b 160.70(6), O3a-La1-O2b 122.74(6), O5-La1-O2b 69.57(6), O1-La1-O2c 134.34(6), O3-La1-O2c 122.74(6), O5a-La1-O2c 69.57(6), O4-La1-O2c 113.62(5), O2b-La1-O2c 48.57(7), O4d-Na1-O5d 77.10(6), O4-Na1-O5d 102.90(6), O2e-Na1-O5d 75.91(6)°. Symmetry transformation: *a* = *-x+1, y, -z+3/2; b* = *-x+1, y-1, -z+3/2; c* = *x, y-1, z; d* = *-x+1, -y, -z+1; e* = *x, -y+1, z-1/2; f* = *-x+1, y+1, -z+3/2; g* = *x, y+1, z.*

COMMENT

Terephthlate, acting as a multidentate ligand, has received great interest in the assembly of metal-organic coordination polymers. Up to now, several coordination polymers have been constructed by metal ions with this versatile ligand [1–5], some of which show unique structure and properties [1–4]. In this paper, we report a 3D coordination polymer formed by sodium, lanthanide and terephthalate in obtained from a DMF solution at room temperature. The crystallographic study shows the lanthanide centre to lie on a crystallographic 2-fold axis which bisects the terephthlate ligand. The lanthanide atom is ten-coordinate and exists in an environment defined by four bidentate carboxylate groups derived from four terephthalate ligands and two DMF molecules. The sodium ion is distorted octahedral and is coordinated by four monodentate carboxylate groups and two DMF molecules. The assembly of the 3D structure can be viewed as two steps: 1) the ten-coordination lanthanide atom connected by four terephthalate ligands forms a 2D structure viewed along *c*-axis; and 2) the 2D structure connected by sodium ions through three oxygen atoms with two from carboxylate groups from two terephthalate ligands and one from DMF molecule generates a 3D structure viewed along the *c*-axis (Figure 2b).

EXPERIMENTAL

A mixture of $LaCl_3 \cdot 9H_2O$ (0.30 g, 1.0 mmol), terephthalic acid (0.34 g, 2.0 mmol) and NaOH (0.16 g, 4.0 mmol) was mixed in 25 ml DMF. The solution was allowed to stand for a week and colourless crystals of

the title complex were obtained. A 0.40 × 0.36 × 0.33 mm crystal was used in the diffraction measurements. The intensities were corrected for absorption effects [6].

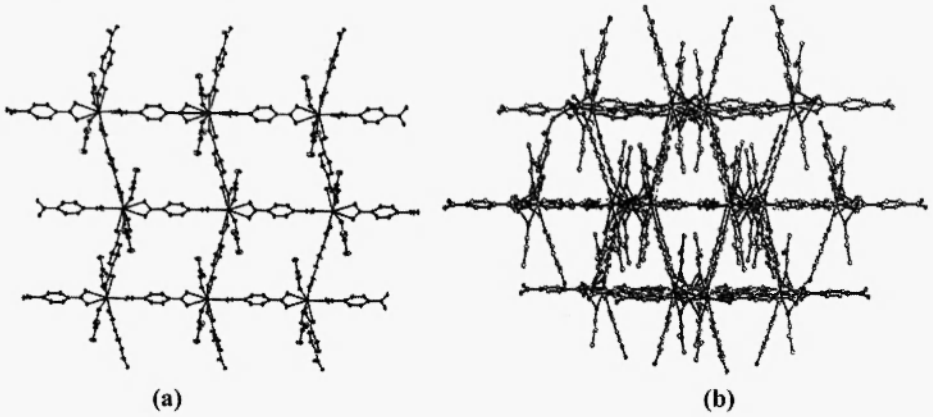

Figure 2. *ORTEP* plot showing 2D structure of $[La(terephthalate)_2(DMF)_2]^-$ along the *c* axis at the 30% probability level **(a)** and 3D structure of $Na[La(terephthalate)_2(DMF)_2]$ along *c* axis at the 30% probability level **(b)**. Hydrogen atoms are omitted for clarity.

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Table 1. Crystal data for Crystal data for the title complex

Empirical formula	$C_{22}H_{22}La\ N_2NaO_{10}$	Formula weight	636.32
Crystal system	Monoclinic	Space group	*C2/c*
Unit cell dimensions	*a* 16.942(3)	Volume, Å^3	2481.9(9
	b 11.735(2) *β* 101.08(3) °	*Z*	4
	c 12.721(3) Å	D_{calc}, g cm^{-3}	1.703
Diffractometer	Enraf–Nonius CAD4	Temperature, K	293
μ (Mo-*K*α), mm^{-1}	1.797	θ range, °	2.1 – 26.0
Reflections collected	2654	*F*(000)	1264
Independent reflections	2440 (R_{int} = 0.016)	Reflections with $I>2\sigma(I)$	2324
No. parameters refined	167	*R* (all data)	0.021
Final *R* [$I>2\sigma(I)$]	0.0191	*wR* (all data)	0.0505
w	$[\sigma^2(F_o^2)+(0.0332P)^2+1.6101P]^{-1}$	where $P = (F_o^2 + 2F_c^2)/3$	
Goodness-of-fit on F^2	1.05	ρ, *e*Å^{-3}	-0.84 to 0.85
Programs	*SHELXS*-97, *SHELXL*-97 [7, 8]	CCDC deposition no.	192643

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