

## 2차원 La-BDC 배위 고분자: $[\text{La}_4(\text{BDC})_6(\text{H}_2\text{O})_5](\text{H}_2\text{O})$ 의 수열합성 및 구조(BDC=benzene-1,3-dicarboxylate)

金惠眞 · 閔東元 · 許鉉秀 · 李順遠\*  
성균관대학교 화학과  
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## Two-Dimensional Lanthanum-BDC Coordination Polymer: Hydrothermal Synthesis and Structure of $[\text{La}_4(\text{BDC})_6(\text{H}_2\text{O})_5](\text{H}_2\text{O})$ (BDC=benzene-1,3-dicarboxylate)

Hye Jin Kim, Dongwon Min, Hyun Sue Hoe, and Soon W. Lee\*  
Department of Chemistry, Sungkyunkwan University, Suwon 440-746, Korea  
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**요 약.** 1,2-Bis(4-pyridyl)ethane 존재 하에서,  $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ 와 benzene-1,3-dicarboxylic acid ( $\text{H}_2\text{BDC}$ )가 수열반응하여  $[\text{La}_4(\text{BDC})_6(\text{H}_2\text{O})_5](\text{H}_2\text{O})$  (1) 실험식을 갖는 2차원 lanthanum-BDC 배위 고분자가 합성되었다. 화합물 1의 구조 분석 결과, 이 고분자는 분명히 구별되는 4개의 La 금속들을 함유하고 있었다. 3개의 La 금속들은 7-배위를 하고 있으며, 이들은 “모자 쪼음(capped)” 삼각프리즘, “모자 쪼음” 정팔면체, 그리고 오각쌍뿔 등의 다른 구조들을 가지고 있다. 나머지 La 금속은 8-배위, 사각 반프리즘(antiprismatic) 구조를 가지고 있다.

**ABSTRACT.** The hydrothermal reaction between lanthanum(III) nitrate ( $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ) and benzene-1,3-dicarboxylic acid ( $\text{H}_2\text{BDC}$ ) in the presence of 1,2-bis(4-pyridyl)ethane gave a 2-D lanthanum-BDC coordination polymer with an empirical formula of  $[\text{La}_4(\text{BDC})_6(\text{H}_2\text{O})_5](\text{H}_2\text{O})$  (1). X-ray structure analysis of compound 1 revealed that this polymer contains four distinct La metals. The three La metals are 7-coordinate with three different structures: a capped trigonal prism, a capped octahedron, and a pentagonal bipyramid. The remaining La metal has a 8-coordinate, square antiprismatic structure.

### INTRODUCTION

Recently, extended frameworks of coordination polymers, based on transition metals and multifunctional bridging groups, have got considerable attractions.<sup>1,2</sup> In particular, the synthesis as well as the structural characterization of lanthanum(III)-carboxylate coordination polymers is a rapidly growing area due to their applications to ceramic materials.<sup>3</sup>

We have recently become interested in preparing coordination polymers with anionic multifunctional ligands. For instance, we have obtained a 3-D zinc

and a 3-D cobalt coordination-polymer by hydrothermal reactions.<sup>4,5</sup> As a continuation of our research, we set out to prepare lanthanum coordination-polymer with benzene-1,3-dicarboxylic acid ( $\text{H}_2\text{BDC}$ ) by the hydrothermal reaction. When we treated lanthanum(III) nitrate with  $\text{H}_2\text{BDC}$  in the presence of 1,2-bis(4-pyridyl)ethane, we obtained a two-dimensional lanthanum-carboxylate coordination polymer with an empirical formula of  $[\text{La}_4(\text{BDC})_6(\text{H}_2\text{O})_5](\text{H}_2\text{O})$  (1). Herein, we report the preparation and structure of the polymer 1, which has an open-framework structure built from 7- or 8-coordinate

lanthanum metals and multidentate benzene-1,3-dicarboxylate (BDC<sup>2-</sup>).

## EXPERIMENTAL SECTION

La(NO<sub>3</sub>)<sub>3</sub> · 6H<sub>2</sub>O and 1,2-bis(4-pyridyl)ethane (BPE) were purchased from Aldrich company and used without further purification. Benzene-1,3-dicarboxylic acid (or isophthalic acid, H<sub>2</sub>BDC) was purchased from Fluka company and recrystallized from ethanol. IR spectra were recorded with a Nicolet 320 FT-IR spectrophotometer as KBr pellets in the range 4000~400 cm<sup>-1</sup>.

**Preparation of [La<sub>4</sub>(BDC)<sub>6</sub>(H<sub>2</sub>O)<sub>2</sub>](H<sub>2</sub>O) (1).** A mixture of La(NO<sub>3</sub>)<sub>3</sub> · 6H<sub>2</sub>O (0.261 g, 0.602 mmol), H<sub>2</sub>BDC (0.100 g, 0.602 mmol), BPE (0.11 g, 0.602 mmol), and deionized H<sub>2</sub>O (6.0 mL, 0.333 mol) in the mole ratio of 1:1:1:553 was heated in a 23-mL capacity Teflon-lined reaction vessel at 180 °C for 2 days, and then cooled to room temperature by air-cooling. The crystalline product was collected by filtration, washed with water (2 × 5 mL) and ethanol (3 × 5 mL), and then air-dried to give yellow crystals of compound 1. IR (KBr): 3373 (br. OH), 3074 (s), 1608 (s), 1543 (s), 1479 (s), 1443 (s), 1385 (s), 747 (s), 713 (s) cm<sup>-1</sup>.

**X-ray structure determination.** All X-ray data were collected with the use of a Siemens P4 diffractometer equipped with a Mo X-ray tube and a graphite-crystal monochromator. The orientation matrix and unit-cell parameters were determined by least-squares analyses of the setting angles of 34 reflections in the range 15.0 < 2θ < 25.0°. Three check-reflections were measured every 100 reflections throughout data collection and showed no noticeable variations in intensity. Intensity data were corrected for Lorentz and polarization effects. Decay corrections were also made. The intensity data were empirically corrected for absorption with ψ-scan data. All calculations were carried out with the use of SHELXTL programs.<sup>9</sup>

A yellow crystal of approximate dimensions 0.20 × 0.18 × 0.08 mm, shaped as a block, was used for crystal- and intensity-data collection. The unit-cell parameters and systematic absences, *h*0*l* (*l* = 2*n* + 1)

Table 1. X-ray data collection and structure refinement

Formula	C <sub>48</sub> H <sub>36</sub> O <sub>30</sub> La <sub>4</sub>
<i>fw</i>	1648.41
temperature, K	296(2)
crystal system	monoclinic
space group	<i>P</i> 2 <sub>1</sub> / <i>c</i>
<i>a</i> , Å	16.753(2)
<i>b</i> , Å	14.605(2)
<i>c</i> , Å	21.521(3)
β, deg	91.242(6)
<i>V</i> , Å <sup>3</sup>	5265(1)
<i>Z</i>	4
<i>d</i> <sub>calc</sub> , g cm <sup>-3</sup>	2.080
μ, mm <sup>-1</sup>	3.278
<i>T</i> <sub>min</sub>	0.2140
<i>T</i> <sub>max</sub>	0.3009
<i>F</i> (000)	3168
No. of reflections measured	9553
No. of reflections unique	9218
No. of reflections with <i>I</i> ≥ 2( <i>I</i> )	5598
No. of parameters refined	739
2θ range (°)	3.5–50.0
scan type	ω
scan speed	variable
GOF (goodness-of-fit on <i>F</i> <sup>2</sup> )	1.011
Max., min. in Δρ (e Å <sup>-3</sup> )	0.888, -1.060
<i>R</i>	0.0485
<i>wR</i> <sub>2</sub>	0.0909

$$^a wR_2 = \frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]}^{1/2}$$

and 0*kl* (*k* = 2*n* + 1), unambiguously indicated *P*2<sub>1</sub>/*c* as a space group. The structure was solved by direct methods. All non-hydrogen atoms were refined anisotropically. The C-H hydrogen atoms were generated in ideal positions and refined in a riding model. The hydrogen atoms in the aqua ligands and in the free water molecule could not be located.

Details on crystal data, intensity collection, and structure refinements are given in Table 1. Final atomic coordinates and some selected bond distances and bond angles are shown in Tables 2 and 3, respectively.

## RESULTS AND DISCUSSION

**Preparation.** The title compound has been prepared by the hydrothermal reaction. Lanthanum(III)

Table 2. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (eq) <sup>a</sup>
La(1)	2790(1)	-678(1)	6132(1)	20(1)
La(2)	3730(1)	1830(1)	4872(1)	18(1)
La(3)	2255(1)	4312(1)	4027(1)	20(1)
La(4)	1345(1)	6845(1)	5261(1)	18(1)
O(1)	4510(4)	1142(5)	5738(3)	26(2)
O(2)	4085(4)	132(5)	6415(3)	27(2)
O(3)	3927(5)	2663(6)	8814(3)	53(3)
O(4)	3305(5)	1433(6)	8450(3)	41(2)
O(5)	3646(5)	3229(5)	5488(3)	40(2)
O(6)	3347(5)	4330(6)	4797(3)	41(2)
O(7)	2657(4)	7414(5)	5696(3)	26(2)
O(8)	3633(5)	7925(5)	6314(3)	42(2)
O(9)	1514(5)	5359(5)	5712(3)	33(2)
O(10)	1553(4)	4231(5)	5017(3)	34(2)
O(11)	2754(4)	1246(5)	5742(3)	24(2)
O(12)	2272(4)	775(4)	6634(3)	29(2)
O(13)	2355(4)	2382(5)	4463(3)	27(2)
O(14)	1424(4)	2928(4)	3831(3)	30(2)
O(15)	1026(5)	-1671(5)	4851(4)	46(2)
O(16)	1681(5)	-657(6)	5436(4)	52(2)
O(17)	571(4)	6178(5)	4407(3)	28(2)
O(18)	991(4)	5158(5)	3727(3)	30(2)
O(19)	1059(5)	7612(5)	1298(3)	40(2)
O(20)	1789(5)	6476(5)	1722(3)	34(2)
O(21)	2330(4)	6187(4)	4448(3)	21(2)
O(22)	2785(5)	5767(5)	3546(3)	33(2)
O(25)	4931(5)	902(6)	4471(3)	47(2)
O(26)	5056(6)	2750(7)	4837(4)	71(3)
O(27)	-80(4)	6562(6)	5519(3)	51(2)
O(28)	1811(5)	4275(5)	2848(3)	37(2)
O(29)	3229(4)	-707(5)	7311(3)	34(2)
O(30)	4451(7)	5768(8)	4429(5)	91(4)
O(23)	3249(5)	10418(5)	4427(3)	43(2)
O(24)	3417(5)	9288(5)	5108(3)	37(2)
C(1)	4527(6)	1571(6)	6793(4)	21(2)
C(2)	4135(6)	1522(7)	7342(4)	23(2)
C(3)	4246(6)	2155(7)	7814(4)	26(2)
C(4)	4786(6)	2860(7)	7725(4)	30(3)
C(5)	5220(7)	2917(7)	7181(4)	36(3)
C(6)	5075(6)	2270(7)	6707(4)	27(2)
C(7)	4376(5)	896(6)	6282(4)	16(2)
C(8)	3791(7)	2084(8)	8406(4)	31(3)
C(9)	3628(6)	4762(6)	5844(4)	28(2)
C(10)	3416(6)	5655(6)	5716(4)	23(2)
C(11)	3517(6)	6318(6)	6177(4)	26(2)
C(12)	3865(7)	6087(7)	6752(4)	35(3)
C(13)	4055(8)	5185(8)	6872(5)	46(3)

Table 2. Continued

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (eq) <sup>a</sup>
C(14)	3926(7)	4519(8)	6426(5)	43(3)
C(15)	3550(6)	4063(7)	5347(5)	29(2)
C(16)	3261(7)	7285(7)	6055(4)	27(3)
C(17)	1782(6)	3875(7)	6083(4)	25(2)
C(18)	1795(7)	4172(7)	6691(4)	33(3)
C(19)	2000(8)	3566(7)	7164(4)	41(3)
C(20)	2204(7)	2668(7)	7026(4)	31(3)
C(21)	2204(6)	2378(7)	6407(4)	22(2)
C(22)	1956(6)	2989(7)	5949(4)	26(2)
C(23)	1596(6)	4530(7)	5565(4)	24(2)
C(24)	2411(6)	1404(7)	6248(4)	20(2)
C(25)	1299(7)	1382(7)	4121(5)	31(3)
C(26)	1431(6)	710(7)	4558(4)	26(2)
C(27)	1049(7)	-118(7)	4522(5)	35(3)
C(28)	497(9)	-274(8)	4033(6)	61(4)
C(29)	350(10)	395(9)	3601(7)	78(5)
C(30)	752(8)	1230(8)	3641(6)	57(4)
C(31)	1732(6)	2278(7)	4141(4)	21(2)
C(32)	1246(7)	-872(8)	4969(5)	38(3)
C(33)	514(6)	6584(7)	3344(4)	25(2)
C(34)	1(6)	7329(8)	3447(4)	30(3)
C(35)	-133(6)	7960(8)	2962(4)	37(3)
C(36)	257(6)	7871(7)	2411(4)	28(3)
C(37)	779(6)	7150(7)	2317(4)	22(2)
C(38)	900(6)	6504(7)	2777(4)	21(2)
C(39)	705(6)	5916(7)	3857(4)	24(2)
C(40)	1246(7)	7083(7)	1727(4)	26(3)
C(41)	2721(6)	7366(7)	3758(4)	25(2)
C(42)	2928(6)	7990(6)	4215(4)	23(2)
C(43)	3013(7)	8917(7)	4075(4)	32(3)
C(44)	2862(7)	9217(7)	3464(4)	36(3)
C(45)	2631(8)	8584(8)	3008(5)	43(3)
C(46)	2604(7)	7665(7)	3151(4)	30(3)
C(47)	2604(6)	6407(7)	3921(4)	22(2)
C(48)	3245(6)	9580(7)	4566(5)	27(2)

<sup>a</sup>Equivalent isotropic *U* defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

nitrate ( $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ ) reacts with benzene-1,3-dicarboxylic acid ( $\text{H}_2\text{BDC}$ ) at 180 °C in the presence of 1,2-bis(4-pyridyl)ethane (BPE) to give  $[\text{La}_4(\text{BDC})_6(\text{H}_2\text{O})_3](\text{H}_2\text{O})$  (**1**), a 3-D polymer (eq 1). Compound **1** has been obtained as yellow crystals. It is air- and moisture-stable and is insoluble in common organic solvents.

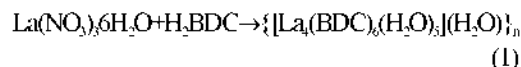


Table 3. Selected bond distances (Å) and bond angles (°)

La1-O16	2.362(8)	La1-O20#1	2.424(7)	La1-O24#2	2.464(6)
La1-O8#2	2.508(7)	La1-O2	2.533(7)	La1-O12	2.542(6)
La1-O29	2.626(6)	La1-O11	2.933(7)	La1-O7#2	2.948(7)
La2-O23#2	2.405(7)	La2-O3#3	2.422(7)	La2-O5	2.442(7)
La2-O1	2.467(6)	La2-O13	2.577(7)	La2-O25	2.590(8)
La2-O26	2.598(9)	La2-O11	2.654(6)	La3-O4#3	2.432(8)
La3-O6	2.442(7)	La3-O10	2.460(7)	La3-O14	2.485(7)
La3-O18	2.524(7)	La3-O22	2.533(7)	La3-O28	2.629(6)
La3-O21	2.887(6)	La3-O13	2.974(7)	La4-O9	2.391(7)
La4-O15#4	2.396(7)	La4-O19#5	2.426(6)	La4-O17	2.430(6)
La4-O27	2.497(7)	La4-O7	2.511(7)	La4-O21	2.614(6)
La4-O7-La1#4	119.4(3)	La2-O11-La1	120.0(2)	La2-O13-La3	116.6(2)
La4-O21-La3	122.4(2)				

Symmetry transformations used to generate equivalent atoms: #1-x, -y+1/2, z+1/2; #2-x, y-1, z; #3-x, -y+1/2, z-1/2; #4-x, y-1, z; #5-x, -y+3/2, z-1/2; #6-x, -y-3/2, z-1/2

The IR spectrum of **1** shows the expected absorption peaks for the asymmetric and symmetric stretching bands of carboxylates ( $\text{BDC}^2$ ) in the ranges of  $1600\sim 1550\text{ cm}^{-1}$  and  $1400\sim 1350\text{ cm}^{-1}$ , respectively.<sup>7</sup> It shows no absorption bands for the free  $\text{H}_2\text{BDC}$  ( $1700\sim 1680\text{ cm}^{-1}$ ), suggesting the complete deprotonation of  $\text{H}_2\text{BDC}$  to  $\text{BDC}^{2-}$ .

A bifunctional ligand BPE was added in hopes of its role as a flexible "spacer", but it does not appear to be involved in the reaction. However, we cannot exclude the possibility that BPE acts as a base to deprotonate  $\text{H}_2\text{BDC}$  during the reaction. The incorporation of the  $\text{BDC}^{2-}$  instead of BPE into the product suggests that the  $\text{BDC}^{2-}$  has a higher coordinating power compared with BPE in this reaction.

**Structure.** Seven-coordinate compounds are relatively rare in transition-metal chemistry. The relative instability of these species has been attributed to the fact that the additional energy of the seventh bond is offset by the increased ligand-ligand repulsions, weaker bonds, and generally reduced ligand-field-stabilization energy as a result of non-octahedral geometry.<sup>8</sup> Three geometries now established: (1) a pentagonal bipyramid; (2) a capped trigonal prism in which a seventh ligand has been added to a rectangular face; (3) a capped octahedron in which a seventh ligand has been added to a triangular face.

The monomeric unit of the polymer  $\{[\text{La}_i(\text{BDC})_6$

$(\text{H}_2\text{O})_5(\text{H}_2\text{O})_n\}$  (**1**) is shown in Fig. 1. Compound **1** exhibits a two-dimensional structure, which contains four crystallographically independent lanthanum ions with four distinct coordination geometries. The three La metals are 7-coordinate with three different structures: a capped trigonal prism (La1), a capped octahedron (La3), and a pentagonal bipyramid (La4). The remaining La metal (La2) has a 8-coordinate, square antiprismatic structure (Chart 1).

The local coordination geometry around La1 is a 7-coordinate, capped trigonal prism, which is constructed by seven oxygen atoms coming from the six carboxylate oxygen atoms and one aqua oxygen atom. The trigonal-prism core comprises two triangles (triangle 1: O2, O8, and O24; triangle 2: O12, O20, and O16), with their dihedral angle of  $17.1^\circ(2)$ . The oxygen atom (O29) in the aqua ligand acts as a capping agent on the rectangular face.

The coordination environment around La2 is a square antiprism, which consists of six carboxylate oxygen and two aqua oxygen atoms. The square-antiprism core comprises two squares (square 1: O3, O26, O5, and O13; square 2: O25, O1, O11, and O23), with their dihedral angle of  $3.5^\circ(3)$ . The coordination geometry of La3 is a capped octahedron, which contains six carboxylate oxygen atoms and one aqua oxygen atom. The coordination sphere of La4 can be described as pentagonal bipyramidal.

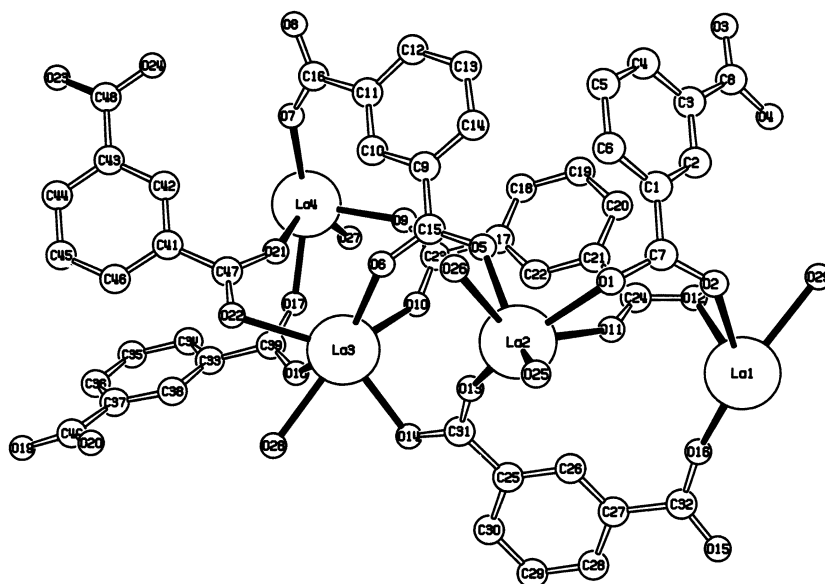


Fig. 1. PLUTO drawing of the local coordination environments of the La metals in compound 1.

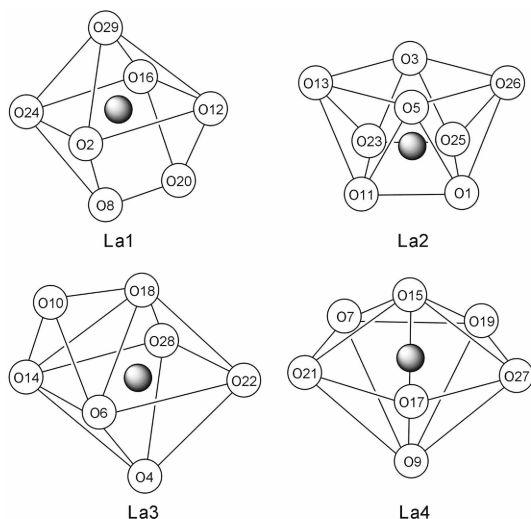


Chart 1. Coordination modes of La metals.

The pentagonal plane is composed of four carboxylate oxygen atoms and one water oxygen atom. The axial sites are occupied by carboxylate oxygen atoms coming from another two BDC<sup>2-</sup> ligands. The equatorial plane, defined by five oxygen atoms (O7, O19, O27, O17, and O21) in the pentagon and La4, is somewhat planar with the average atomic displacement of 0.1632 Å.

The bonding parameters mentioned above indicate the flexibility in the coordination of lanthanide ions, which might facilitate the formation of infinite frameworks of polymer **1** with unusual coordination numbers and geometries. The shortest La...La distances {La1...La2: 4.8407(9); La2...La3: 4.7283(9); La3...La4: 4.8213(9) Å} indicate no direct La-La interactions.

Fig. 2 shows an extremely complicated 2-D polymeric structure of **1**. All carboxylate groups participate in connecting La metals to give a compact polymeric structure like a double helix, which does not appear to have any desirable channels. Whereas four carboxylate oxygen atoms (O7, O11, O13, and O21) participate in connecting La metals by acting as  $\mu_2$ -O bridging ligands, five aqua ligands (O25-O29) act simply as monodentate ligands.

In summary, we have structurally characterized  $\{[La_4(BDC)_6(H_2O)_4](H_2O)\}_n$  (**1**), which was prepared by the hydrothermal reaction of  $La(NO_3)_3 \cdot 6H_2O$  with benzene-1,3-dicarboxylic acid ( $H_2BDC$ ) in the presence of 1,2-bis(4-pyridyl)ethane. The structure of polymer **1** is unique in that it contains four distinct 7- or 8-coordinate La ions with a different coordination geometry for each La: a capped trigonal

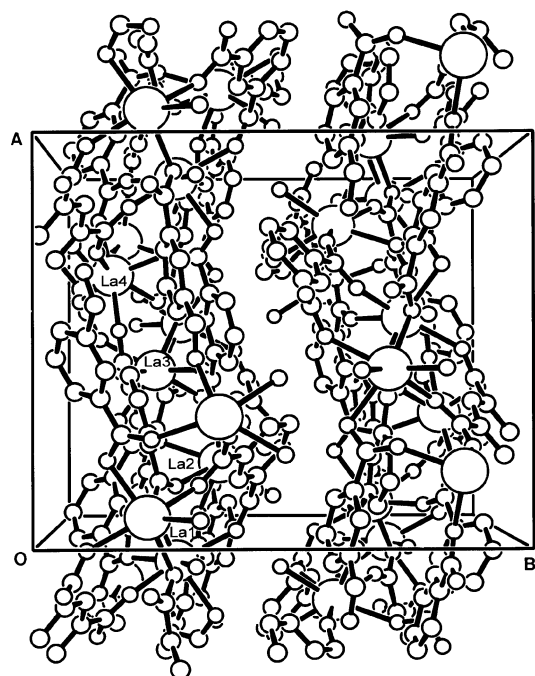


Fig. 2. A perspective view of the building blocks along the *c*-axis.

prism, a capped octahedron, a pentagonal bipyramid, or a square antiprism. Unfortunately, this polymer does not exhibit any desirable pores or channels of appropriate sizes.

#### SUPPLEMENTARY MATERIAL

Tables of full bond distances and bond angles, anisotropic thermal parameters, and atomic coordinates of hydrogen atoms are available from the corresponding author Soon W. Lee.

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