Crystal Engineering

Absolute Helical Arrangement of Stacked Benzene Rings: Heterogeneous Double-Helical Interaction Comprising a Hydrogen-Bonding Belt and an Offset Parallel Aromatic–Aromatic-Interaction Array**

Isao Azumaya, Daisuke Uchida, Takako Kato, Akihiro Yokoyama, Aya Tanatani, Hiroaki Takayanagi, and Tsutomu Yokozawa**

Compounds that form helical structures in the crystalline state or in solution[1] have attracted much attention because living things utilize helical structures to store genetic information, and because compounds with a helical-ordered structure have provided new materials in solid- or liquid-crystal engineering.[2] There are several types of helical structure: a single strand that folds helically owing to solvophobic effect[3] or guest ligation,[4] a double or multiple helix of strands derived from cation or anion ligation,[5] or hydrogen bonding,[6] and a helical super-structure formed by conformationally helical or discotic small molecules through columnar stacking supported by hydrogen bonds,[7] for other examples see ref. [8]. In helical structures, a parallel aromatic–aromatic interaction[9] is often a very important driving force to stabilize the structure, and sometimes hydrogen bonding or other weak intermolecular interactions assist the formation of the highly ordered helical arrangement. Such a helical structure is intrinsically chiral, that is, it has a right-handed helix (*P*-helix) or a left-handed helix (*M*-helix), which are enantiomeric. Asymmetric synthesis to produce one enantiomeric helix is important, especially in self-assembling systems,[5–7] in foldamers,[1a] or in polymers with a helical structure induced by catalytic chiral initiation.[10] There are generally two kinds of driving forces to determine which helicity a compound will adopt, they are, an internal or an external chiral source. For example, the helicity of DNA is derived from the internal chirality of the chiral carbon atom in the deoxyribose chain. On the other hand, one of oligophenylacetylenes prepared by Moore and co-workers folded into a single helical form upon ligation of a chiral ligand.[4b,c] In this context, absolute asymmetric assembly of an achiral molecule is especially intriguing. In the course of our investigation[11] of spontaneous optical resolution,[12] we found a discotic compound, tris(2-hydroxyethyl)-1,3,5-benzenetricarboxylate (**1**), which crystallized as chiral crystals in which the nearly planar molecules were helically arranged within individual single crystals. The helicity of the triester **1** was derived from the helical arrangement of the molecules, supported by an array of hydrogen bonding through a single moiety and offset aromatic stacking of the adjacent molecules.

The triester **1**, which has an almost planar and highly symmetrical primary structure, was synthesized and crystallized from methanol/chloroform to give slender prismatic single crystals. A preliminary X-ray crystallographic analysis (Figure 1) revealed that the crystal belonged to the noncentrosymmetric space group $P6_1$ (or $P6_5$). This result was surprising, since the majority of achiral organic compounds tend to pack into centrosymmetric crystals. The most interesting feature is that the planar, discotic molecules were arranged in a columnar stack with a progressive helical twist between adjacent molecules to form helical superstructures in the crystal lattice. The chirality of the crystals was intrinsically derived from the helical arrangement of the molecules, assisted by the twist of the terminal hydroxy groups with respect to the plane of the central benzene ring, which leads to the generation of conformational chirality.

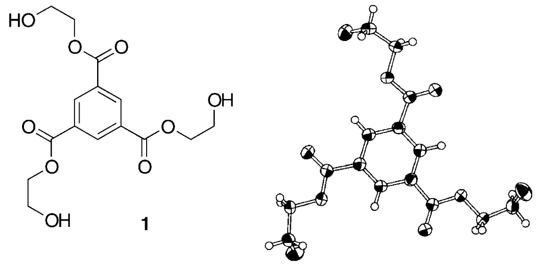

Figure 1. ORTEP drawing of the crystal structure of **1**. The thermal ellipsoids are set at 50% probability.

We measured the solid-state CD spectrum (in KBr) of several crystals of **1**.[13] The Cotton effect was characteristic of a helical structure of a chromophore.[14] As expected, we found two enantiomeric crystals which showed mirror-image curves in the region between 200–320 nm (Figure 2); one (red line in Figure 2) showed a negative Cotton effect at around 210 nm, a large positive one at 224 nm, a negative one at 248 nm, and two faint positive ones at 288 and 298 nm, and the other showed Cotton effects with the opposite sign at the corresponding wavelengths. Because one torsion angle of OC–aromatic ring unit was −2.7(4)° and the others were 0.1(4)° and 1.2(4)°, the Cotton effects of the crystal could be derived from the helical arrangement of the benzene rings of the molecule as well as torsion of one carbonyl group with respect to the central benzene ring.

The absolute structure could be determined[15] by comparison of the Flack parameter[16] for each space group ($P6_1$ or $P6_5$) using the least-squares method. For example, if the

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[*] Dr. I. Azumaya, T. Kato, Prof. Dr. H. Takayanagi
Faculty of Pharmaceutical Sciences
Kitasato University
5-9-1 Shirokane, Minato-ku, Tokyo 108-8641 (Japan)
Fax: (+81) 334-42-5707
E-mail: azumayai@pharm.kitasato-u.ac.jp

D. Uchida, Dr. A. Yokoyama, Dr. A. Tanatani, Prof. Dr. T. Yokozawa
Department of Applied Chemistry
Kanagawa University
Kanagawa-ku, Yokohama 221-8686 (Japan)
Fax: (+81) 45-413-9770
E-mail: yokozt01@kanagawa-u.ac.jp

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Supporting information (view of the left and right handed helixes) for this article is available on the WWW under http://www.angewandte.org or from the author.

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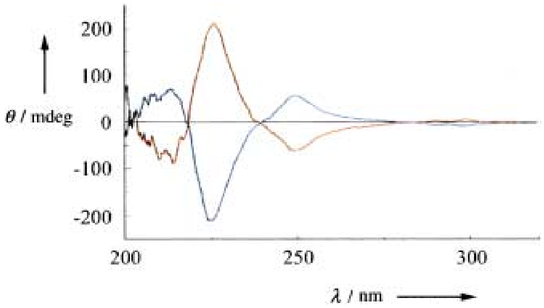

Figure 2. CD spectra of two enantiomeric crystals of **1** ($P6_1$: red line; $P6_5$: blue line) in KBr. A mixture of 250 μg of each crystal and 100 mg of KBr was well ground and formed into a transparent disk with a radius of 5 mm.

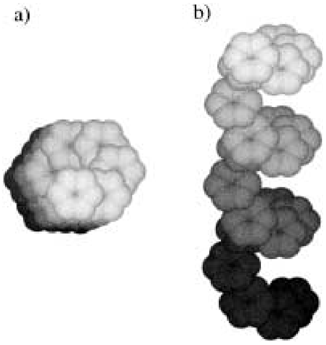

Figure 4. Top view (a) and tilted view (b) of the crystal packing of **1** showing the helical arrangement of the central benzene rings of the molecules. Neighboring benzene rings are stacked in an offset mode.

crystal that showed a positive Cotton effect at 224 nm was in the space group $P6_1$, the Flack parameter was 0.003 and in $P6_5$, it was 0.942, so the crystal was assigned to $P6_1$. On the other hand, if the crystal that showed a negative Cotton effect at 224 nm was in the space group $P6_1$, the Flack parameter was 1.036 and in $P6_5$, it was −0.041, so the crystal was assigned to $P6_5$. This correspondence between the Flack parameter and the sign of the Cotton effect was confirmed by examining at least three crystals for each enantiomer.

A further investigation of the crystal structure of **1** revealed that the arrangement of the molecule showed unique features. Only one of the three hydroxyethyloxycarbonyl groups formed a hydrogen bonding array between adjacent molecules, that is, the hydroxy group of a hydroxyethyloxycarbonyl group formed a hydrogen bond with the carbonyl group of a hydroxyethyloxycarbonyl group of the next molecule (Figure 3). This single-hydrogen-bond belt forms a helical strand, which was right-handed for $P6_1$ and left-handed for $P6_5$.[17] Another remarkable feature is that the central benzene rings of the molecules exhibit circular offset stacking to form an enantiomeric single helix (Figure 4). These heterogeneous helical weak interactions have the same helicity. The attractive interaction between two aromatic rings in this structure would be strongest among various types of parallel stacking because an aromatic hydrogen center is located just above the center of the adjacent benzene ring.[9d,g–i] Because the unit cell has six molecules along the

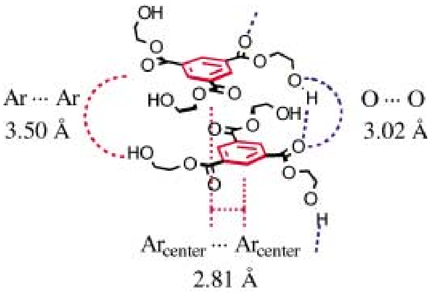

Figure 3. Schematic representation of the relative position of adjacent molecules in the crystal packing of **1**; red: benzene separations, blue: -hydrogen-bonding distances.

c axis, which gives rise to a complete turn in 21.0 Å, the distance between adjacent benzene rings is 3.50 Å. The exterior width of the array of molecules is about 12.5 Å, which is just over half of that of DNA (ca. 20 Å).

The stacked structure of **1** is reminiscent of the layered crystal structures of *N*,*N*′,*N*′′-tris(2-methoxyethyl)benzene-1,3,5-tricarboxamide (**2**) which was reported to crystallize as chiral crystals belonging to space group $P2_1$.[7a] However, the triamide **2** showed straight stacking of the central benzene ring along the *b* axis, with complete overlap, and the distance between the central benzene rings is 3.60 Å. Further, the helical structure is derived from triple helices formed by hydrogen bonding between the secondary amide hydrogen and the carbonyl oxygen of the neighboring molecule. On the other hand, the helicity of the triester **1** was derived from a heterogeneous double-helical interaction, that is, a relatively weak, single hydrogen-bonding belt involving one of the hydroxyethyloxycarbonyl groups and an offset parallel aromatic–aromatic interaction between Ar−H and the center of the neighboring benzene ring to give the offset stacking. Our triester is the first example of an absolute helical arrangement of a single benzene ring in a parallel offset-stacking array that undergoes spontaneous resolution in the crystal, assisted by progressive hydrogen bonding; that is, the spontaneous resolution is driven by two different weak helical interactions which result in the formation of Ar−H···π and OH···O=C arrays. Such helical chirality induction of an achiral compound is of great interest, since such a system could be a simple model for spontaneous helical structure formation in nature.

Experimental Section

1: Preparation of 2-(*tert*-Butyldimethylsilyloxy)ethanol: dropwise, over 1 h, a solution of *tert*-butyldimethylsilyl chloride (15.1 g, 0.10 mol) in dry THF (500 mL) was added to a stirred solution of ethylene glycol (37.2 g, 0.60 mol) and imidazole (6.81 g, 0.10 mol) in dry THF (500 mL) at room temperature, and the mixture was stirred at room temperature for 6 h. After addition of water, THF was removed by evaporation under reduced pressure, and the residue was extracted with ethyl acetate. The organic layer was washed with water, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (elution solvents: ethyl acetate/hexane = 1/4) to give 2-(*tert*-

butyldimethylsilyloxy)ethanol as a colorless oil (14.1 g, 80 %): ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 3.74$–3.40 (m, 4 H), 2.47–2.33 (m, 1 H), 0.85 (s, 9 H), 0.03 ppm (s, 6 H); ^{13}C NMR (50 MHz, $CDCl_3$): $\delta =$ 64.2, 63.7, 25.9, −5.4 ppm.

Tris[2-(*tert*-butyldimethylsilyloxy)ethyl]-1,3,5-benzenetricarboxylate: dropwise, over 3 h, a solution of 1,3,5-benzenetricarbonyl trichloride (5.88 g, 22 mmol) in dry CH_2Cl_2 (100 mL) was added to a stirred solution of 2-(*tert*-butyldimethylsilyloxy)ethanol (14.1 g, 80 mmol; see above) and triethylamine (8.06 g, 80 mmol) in dry CH_2Cl_2 (200 mL) at room temperature, and the mixture was stirred at room temperature for 3 days. After addition of water, the mixture was extracted with CH_2Cl_2, and the organic layer was washed with water, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (elution solvents: ethyl acetate/hexane = 1/4) to give tris-[2-(*tert*-butyldimethylsilyloxy)ethyl]-1,3,5-benzenetricarboxylate as a yellow oil (13.6 g, 90 %): ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 8.89$ (s, 3 H), 4.45 (t, $J = 5.0$ Hz, 6 H), 3.96 (t, $J = 5.0$ Hz, 6 H), 0.90 (s, 27 H), 0.09 ppm (s, 18 H); ^{13}C NMR (125 MHz, $CDCl_3$): $\delta = 164.9$, 134.7, 131.3, 66.7, 61.1, 25.8, 18.3, −5.31 ppm; IR (neat) 2954, 2930, 2884, 2857, 1733, 1472 cm^{-1}.

Tris(2-hydroxyethyl)-1,3,5-benzenetricarboxylate (**1**): A mixture of tris[2-(*tert*-butyldimethylsilyloxy)ethyl]-1,3,5-benzenetricarboxylate (13.6 g, 20 mmol; see above), 6 M hydrochloric acid (1 mL), and THF (200 mL) was stirred at room temperature for 6 h, and concentrated under reduced pressure. The crude solid obtained was washed with hexane and dried in vacuo to give **1** as a colorless solid (6.11 g, 90 %): ^{1}H NMR (600 MHz, [D$_6$]DMSO): $\delta = 8.74$ (s, 3 H), 4.99 (t, $J = 5.4$ Hz, 3 H), 4.39 (t, $J = 4.9$ Hz, 6 H), 3.75 ppm (t, $J = 5.0$ Hz, 6 H); ^{13}C NMR (151 MHz, [D$_6$]DMSO): $\delta = 164.5$, 133.8, 131.3, 67.5, 59.1 ppm; IR (KBr): $\tilde{\nu} = 3327$, 2955, 1722, 1441, 1242, 1078, 1025 cm^{-1}; Elemental analysis calcd (%) for $C_{15}H_{18}O_9$: C 52.63, H 5.30; found: C 52.60, H 5.34.

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[15] Crystal data of **1a** ($P6_1$): $C_{15}H_{18}O_9$, $M_r = 342.30$, hexagonal, space group $P6_1$, $a = 11.304(1)$, $c = 20.997(4)$ Å, $V = 2323.5(6)$ Å^3, $Z = 6$, $\rho_{calcd} = 1.468$ g cm^{-3}, $T = 296$ K, crystal size $0.20 \times 0.20 \times 0.20$ mm^3, $R = 0.043$, $R_w = 0.055$, GOF = 1.23 for 2817 reflections with $I > 0.0\sigma(I)$. The Flack absolute structure parameter has a value of 0.003(0.161), while the inverted stereochemistry requires a value of 0.942(0.154). Crystal data of **1b** ($P6_5$): $C_{15}H_{18}O_9$, $M_r = 342.30$, hexagonal, space group $P6_5$, $a = 11.3048(9)$, $c = 21.011(3)$ Å, $V = 2325.5(5)$ Å^3, Z = 6, $\rho_{calcd} = 1.466$ g cm^{-3}, $T = 296$ K, crystal size $0.20 \times 0.20 \times 0.20$ mm^3, $R = 0.047$, $R_w = 0.067$, GOF = 1.29 for 2781 reflections with $I > 0.0\sigma(I)$. The Flack absolute structure parameter has a value of −0.041(0.174), while the inverted stereochemistry requires a value of 1.036(0.164). CCDC-218393 (**1a**) and CCDC-218394 (**1b**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB21EZ, UK; fax: (+44)1223-336-033; or deposit@ccdc.cam.ac.uk).

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[17] Only one of the hydroxy groups contributes to the formation of a single-helical hydrogen-bonding array of $-C{=}O\cdots HOCH_2CH_2OC{=}O\cdots HO-$ (see Supporting Information). The two other hydroxy groups form weak hydrogen bonds with carbonyl groups on the next molecule, but do not form a hydrogen-bonding array.

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