Contents lists available at SciVerse ScienceDirect

Journal of Molecular Structure



journal homepage: www.elsevier.com/locate/molstruc

Organic crystals bearing both channels and cavities formed from tripodal adamantane molecules

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HIGHLIGHTS

- Three adamantane-cored tripodal molecules were synthesized.
- X-ray crystallographic analyses were performed on single-crystals of tripodal molecules.
- Three tripodal molecules afforded molecular organic networks bearing internal spaces.
- Two crystals exhibited the formation of both 1D channels and cavities.
- One crystal showed the generation of only cavities in the crystalline lattices.

ARTICLE INFO

Article history: Received 26 October 2012 Received in revised form 11 March 2013 Accepted 18 March 2013 Available online 26 March 2013

Keywords: Adamantane Tripodal molecule Channel structure Cavity Crystal packing

1. Introduction

The construction of channels and cavities in molecular organic networks are important subjects in the fields of crystal engineering and materials science [1–4]. Organic materials which have inner spaces are of considerable interest due to their ability to recognize molecules selectively, stabilize unstable guest species, and perform chemical transformations [5–15]. Especially, C₃-symmetric organic

GRAPHICAL ABSTRACT



ABSTRACT

Three adamantane-based tripodal molecules bearing either a benzene, pyridine, or toluene unit (1-3) form molecular organic networks (1a-3a) with internal spaces, via intermolecular non-covalent interactions such as CH/ π , CH/N, and CH/O interactions in the solid state. Crystals of **1a** and **2a** formed both onedimensional channels and cavities, where guest molecules were encapsulated. The channels were derived from the alignment of the hexagonal cavities formed from six component molecules, while cavities formed between the 2D layers. In contrast, **3a** contained only cavities built from the six component molecules, which correspond to the spaces which connected to form channels in **1a** and **2a**.

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molecules with rigid frameworks are useful building blocks for the production of organic crystals with channels or cavities [16–19]. For example, tris(o-phenylenedioxy)cyclotriphosphazene forms a porous crystal with a permanent one-dimensional channel, where gases, linear π -conjugated molecules, and polymers can be encapsulated [20–22]. Substituted 1,3,5-triazine molecules provide channel-type structures, and can accommodate large molecules such as fullerenes in addition to small aromatic molecules [23,24]. However, examples of molecular organic networks containing both channels and cavities are quite limited. The generation of these higher orders of network structures containing inner spaces is an intrinsically important task for the development



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^{0022-2860/\$ -} see front matter \odot 2013 Elsevier B.V. All rights reserved. http://dx.doi.org/10.1016/j.molstruc.2013.03.033

of host-guest systems for the molecular recognition of gasses, organic molecules, and others [25-36]. Thus, we envisioned molecules with non-planar $C_{3\nu}$, to bent planar C_3 symmetric compounds would be appropriate for the preparation of crystalline materials which have both channels and cavities. This is because these molecules may form a cavity separate from the channels due to their three-dimensional shape, whilst maintaining the channel structures. Tri-substituted adamantane is a suitable skeleton for the design of C_{3v} -symmetric molecules. Recently, we presented hydrogen-bonded organic networks with channels that were built from adamantane-based tripodal molecules containing dimethoxyphenol moieties [37]. Herein we report three different adamantane-centered tripodal molecules bearing either a benzene, pyridine, or toluene moiety and the molecular organic networks they form through intermolecular non-covalent interactions such as CH/ π , CH/N, and CH/O interactions.

2. Experimental

2.1. General

All the reagents and solvents used were commercially available and employed as received without further purification. The ¹H NMR and ¹³C NMR spectra were recorded on a Bruker AV-400M spectrometer at 27 °C using CDCl3 as the solvent and TMS as an internal reference. Melting points were determined on an ASONE ATM-01 melting point apparatus and were uncorrected. FAB mass was performed on a JEOL JMS-700 using an m-nitrobenzylalcohol (NBA) matrix. Elemental analyses were performed with a Perkin-Elmer 2400 elemental analyzer. IR spectra were recorded with a Jasco FT/IR-6300 instrument. Xray data for the crystals **1a-3a** were collected on a CCD diffractometer with graphite monochromated Mo K α (λ = 0.71073 Å) radiation. Data collection was carried out at 100 K using a Japan Thermal Eng. Col., Ltd. Cryostat system equipped with a liquid nitrogen generator. The crystal structures were solved by direct methods (SHELXS 97, Sheldrick, 1997) [38]. Refinements were carried out by full-matrix least squares on F^2 , with anisotropic temperature factors for non-H atoms. In all structures, H atoms were included as their calculated positions. SHELXTL was used for refinement of the structure and structure analysis. Column chromatography was performed using a Wakogel C200, and thin-layer chromatography was carried out on 0.25 mm Merck precoated silica gel glass plates. Gel permeation Chromatography (GPC) was performed using a recycling preparative HPLC system (LC-9204, Japan Analytical Industry Co., Ltd.) fitted with a JAIGEL H series column (Japan Analytical Industry Co., Ltd.).

2.2. Synthesis

2.2.1. 1,3,5-Tris(4-benzoyloxy-3,5-dimethoxyphenyl)adamantane (1)

To a solution of 1,3,5-tris(4-hydroxy-3,5-dimethoxyphenyl)adamantane (0.59 g, 1.00 mmol) in dry THF (20 mL), benzoyl chloride (0.46 g, 3.30 mmol) was added under an argon atmosphere at 0 °C. After 10 min, triethylamine (1.5 mL) was added into the solution, and stirred for 2 d at room temperature. After evaporation to dryness, the residue was dissolved in CHCl₃, and washed with a saturated aqueous NaHCO₃ solution, then H₂O, and finally brine. The organic layer was dried over anhydrous Na₂SO₄ and filtered. Evaporation of the solvent followed by silica gel column chromatography (eluent: CHCl₃) and gel permeation chromatography (JAIGEL 1H+2H, CHCl₃) afforded **1** as a white solid (0.74 g, 0.82 mmol) in 82% yield. m.p. 170–172 °C. FT-IR (ATR, cm⁻¹): 2932, 2850, 1739, 1599, 1514, 1450, 1412, 1327, 1262, 1212, 1125, 1059, 704. ¹H NMR (400 MHz, CDCl₃, 27 °C) δ 8.24 (d, 6H, J = 7.6 Hz), 7.60 (t, 3H, J = 7.2 Hz), 7.48 (t, 6H, J = 8.0 Hz), 6.75 (s, 6H), 3.84 (s, 18H), 2.59 (s, 1H), 2.20–2.06 (m, 12H). ¹³C NMR (100 MHz, CDCl₃, 27 °C) δ 164.60, 152.01, 148.25, 133.24, 130.29, 129.33, 128.33, 127.18, 102.24, 56.25, 48.27, 41.42, 38.83, 30.06. MS (FAB, m/z) calcd for C₅₅H₅₃O₁₂ (M+H⁺) 905.35, found 905.1. Anal. Calcd for C₅₅H₅₂O₁₂·0.1H₂O: C, 72.85; H, 5.80. Found: C, 72.56; H, 5.78.

2.2.2. 1,3,5-Tris(4-isonicotinoyloxy-3,5-dimethoxyphenyl) adamantane (2)

To a solution of 1,3,5-tris(4-hydroxy-3,5-dimethoxyphenyl)adamantane (0.59 g, 1.00 mmol) in dry THF (20 mL), isonicotinoyl chloride hydrochloride (0.59 g, 3.30 mmol) was added under an argon atmosphere at 0 °C. After 10 min, triethylamine (2.5 mL) was added into the solution, and stirred for 3 d at room temperature. After evaporation to dryness, the residue was dissolved in CHCl₃, and washed with a saturated aqueous NaHCO₃ solution, then H₂O, and finally brine. The organic layer was dried over anhydrous Na₂SO₄ and filtered. Evaporation of the solvent followed by silica gel column chromatography (eluent: CHCl₃:-MeOH = 50:1) and gel permeation chromatography (JAIGEL 1H+2H, CHCl₃) afforded **2** as a white solid (0.58 g, 0.64 mmol) in 64% yield. m.p. 232–233 °C. FT-IR (ATR, cm⁻¹): 2931, 2847, 1748. 1587, 1508, 1451, 1413, 1324, 1248, 1212, 1124, 847, 687. ¹H NMR (400 MHz, CDCl₃, 27 °C) & 8.85 (s, 6H), 8.05 (d, 6H, J = 5.6 Hz), 6.75 (s, 6H), 3.86 (s, 18H), 2.62 (s, 1H), 2.22–2.08 (m, 12H). ¹³C NMR (100 MHz, CDCl₃, 27 °C) δ 163.22, 151.75, 150.55, 148.63, 136.54, 126.61, 123.40, 102.02, 56.16, 48.20, 41.33, 38.87, 29.98. MS (FAB, m/z) calcd for $C_{52}H_{50}N_3O_{12}$ (M+H⁺) 908.33, found 908.9. Anal. Calcd for C52H49N3O12·1H2O: C, 67.45; H, 5.55; N, 4.54. Found: C, 67.14; H, 5.41; N, 4.53.

2.2.3. 1,3,5-Tris(4-(4-methylbenzoyloxy)-3,5-dimethoxyphenyl) adamantane (3)

To a solution of 1.3.5-tris(4-hvdroxy-3.5-dimethoxyphenyl)adamantane (0.59 g. 1.00 mmol) in dry THF (20 mL), p-toluoyl chloride (0.51 g, 3.30 mmol) was added under an argon atmosphere at 0 °C. After 10 min, triethylamine (1.5 mL) was added into the solution, and stirred for 2 d at room temperature. After evaporation to dryness, the residue was dissolved in CHCl₃, and washed with a saturated aqueous NaHCO₃ solution, then H₂O, and finally brine. The organic layer was dried over anhydrous Na₂SO₄ and filtered. Evaporation of the solvent followed by silica gel column chromatography (eluent: CHCl₃) and gel permeation chromatography (JAIGEL 1H+2H, CHCl₃) afforded **3** as a white solid (0.67 g, 0.71 mmol) in 71% yield. m.p. 163-165 °C. FT-IR (ATR, cm⁻¹): 2933, 2849, 1733, 1598, 1515, 1452, 1411, 1325, 1263, 1211, 1128, 1061, 816, 746. ¹H NMR (400 MHz, CDCl₃, 27 °C) δ 8.13 (d, 6H, J = 8.0 Hz), 7.29 (d, 6H, J = 8.4 Hz), 6.73 (s, 6H), 3.83 (s, 18H), 2.59 (s, 1H), 2.43 (s, 9H), 2.18–2.05 (m, 12H). ¹³C NMR (100 MHz, CDCl₃, 27 °C) δ 164.69, 152.09, 148.20, 144.02, 130.40, 129.07, 127.30, 126.63, 102.32, 56.31, 48.31, 41.48, 38.86, 30.11, 21.65. MS (FAB, m/z) calcd for C₅₈H₅₉O₁₂ (M+H⁺) 947.39, found 946.8. Anal. Calcd for C₅₈H₅₈O₁₂·0.1H₂O: C, 73.42; H, 6.18. Found: C, 73.08; H, 6.10.

2.3. Crystallization

The appropriate tripodal molecule (0.02 mmol) was stirred in tetrahydrofuran (10.0 mL) for **1** or acetonitrile (10.0 mL) for **2** and **3** at room temperature for 1 h. Colorless single crystals of **1a–3a** were obtained by slow evaporation of the solvent after a few days.

3. Results and discussion

The organic networks (**1a–3a**) were formed from an adamantane-cored tripodal molecule (**1–3**), in which either benzene, pyridine, or toluene units were linked through ester groups to a triaryladamantane derivative (Fig. 1). The esterification of 1,3, 5-tris(4-hydroxy-3,5-dimethoxyphenyl)adamantane with the corresponding acid chloride compounds in the presence of triethylamine gave the desired adamantane-cored tripodal molecules bearing arylester groups. Colorless single crystals of **1a** were obtained by slow evaporation of a tetrahydrofuran solution of **1**. Crystals **2a** and **3a** were isolated by the slow evaporation of an acetonitrile solution of the compounds **2** and **3**, respectively. X-ray crystallographic analyses were carried out on single-crystals of **1a–3a** (Table 1).

1a Crystallized in the trigonal system, in the centrosymmetric space group P-3c1, and included one molecule of **1** and three molecules of tetrahydrofuran in the asymmetric unit. In the structure, the tripodal molecules were assembled into two-dimensional (2D) layers via CH/ π interactions [39] between the phenyl groups (the distance between the carbon atom and the ring center of the phenyl ring was 4.07 Å) (Fig. 2a) [40,41]. These layers were further organized as three-dimensional networks via CH/O interactions between the methylene protons of the adamantane units and the oxygen atoms of carbonyl groups at a distance of 3.45 Å. measured between the methylene carbon and the carbonyl oxygen atoms (Fig. 2b). An isolated cavity A was formed between the individual 2D layers, where disordered tetrahydrofuran molecules were enclathrated. A hexagonal cavity **B** formed from the cyclic hexamers of the tripodal molecule 1, and these cavities formed the channel framework (Fig. 2c). Disordered tetrahydrofuran molecules were accommodated within the hexagonal cavity. Thus, both channels



Fig. 1. The three adamantane-centered tripodal molecules 1–3.

Table

|--|

_	Crystal	1a	2a	3a
	Formula	C _{66.99} H _{75.99} O ₁₅	$C_{56}H_{49}N_5O_{13.5}$	$C_{36}H_{29}N_{3.5}O_{6.25}$
	Crystal system	Trigonal	Trigonal	Trigonal
	Space group	P3c1	P3c1	RĪ
	a (Å)	15.7084 (10)	15.425 (2)	18.3248 (18)
	b (Å)	15.7084 (10)	15.425 (2)	18.3248 (18)
	c (Å)	25.9732 (18)	25.083 (5)	35.862 (3)
	α (°)	90	90	90
	β(°)	90	90	90
	γ(°)	120	120	120
	$V(Å^3)$	5550.3 (6)	5168.3 (16)	10429.0 (17)
	Dc (Mg m ⁻³)	1.342	1.295	1.167
	Ζ	4	4	12
	T (K)	100	100	100
	$R_1, wR_2 [I > 2\sigma(I)]$	0.0789, 0.2398	0.0552, 0.1660	0.1004, 0.3152
	CCDC number	903301	903302	903303

and cavities were observed in the crystalline lattices, with solvent molecules included in both the channels and cavities.

2a Crystallized in the trigonal system in the centrosymmetric space group P-3c1, and included one molecule of **2**, one molecule of water, and two molecules of acetonitrile in the asymmetric unit.



Fig. 2. Crystal structure of **1a**. (a) The 2D layer structure, (b) the 3D network structure viewed from the side, along the b axis, and (c) a space-filling model of the 3D network structure viewed from the top. The guest molecules were omitted for clarity. The red dotted lines show the CH/ π interactions.



Fig. 3. Crystal structure of **2a**. (a) The 2D layer structure, (b) the 3D network structure viewed from the side, along the b axis, and (c) a space-filling model of the 3D network structure viewed from the top. The guest molecules were omitted for clarity. The red dotted lines show the CH/O interactions.

In **2a**, the tripodal molecules assembled into 2D layers via CH/O interactions between the methylene protons of the adamantane units and the oxygen atoms of carbonyl groups. The distance between the carbon atom and the oxygen atom was 3.45 Å (Fig. 3a). These layers were further organized into 3D networks via CH/O interactions between the carbon atom of one methoxy groups and the oxygen atom of another at a distance of 3.39 Å (Fig. 3b). Similarly, a cavity **A** was generated between the 2D layers, where acetonitrile molecules were enclathrated via CH/N interactions between the phenyl protons and the nitrogen atoms of acetonitrile where the distance between the carbon atom and



Fig. 4. Crystal structure of **3a**. (a) The 2D layer structure, (b) the 3D network structure viewed from the side, along the b axis, and (c) a space-filling model of the 3D network structure viewed from the top. The guest molecules were omitted for clarity. The red dotted lines show the CH/ π interactions.

the nitrogen atom was 3.58 Å. A hexagonal cavity **B** was produced from the cyclic hexamers of the tripodal molecule **2**, and forms a channel framework (Fig. 3c). Acetonitrile and water molecules were encapsulated within the hexagonal cavity and disordered over two identical sites.

3a Crystallized in the trigonal system in the centrosymmetric space group R-3, and included one molecule of **3**, one molecule of water, and three molecules of acetonitrile in the asymmetric unit. In **3a**, the tripodal molecules were arranged into 2D layers

via CH/π interactions between the methylene protons of the adamantane units and the phenyl groups, with a distance between the carbon atom and the ring center of the phenyl ring of 4.14 Å, and between the methyl protons of the methoxy groups and the phenyl groups with a distance between the carbon atom and the ring center of the phenyl ring of 4.18 Å (Fig. 4a). These layers were extended into 3D networks via CH/π interactions between the methyl protons of the methoxy groups and the phenyl groups, with a distance between the carbon atom and the ring center of the phenyl ring of 3.66 and 4.35 Å (Fig. 4b). A hexagonal cavity **A** arose from the cyclic hexamers of the tripodal molecule **3** (Fig. 4c), where multiple acetonitrile molecules were enclathrated due to CH/N interactions between the phenyl protons and the nitrogen atoms of nitrile groups with distances between the carbon atoms and the nitrogen atoms from 3.55 to 3.47 Å. The 2D layers were assembled into 3D networks with a half molecule shift along the *c* axis. This means the hexagonal cavity could not produce channels along the c axis. Thus, in **3a**, only cavities were formed in the organic networks, where acetonitrile and water molecules were included.

Each organic crystal had different types of internal spaces in the crystalline lattices, where multiple guest molecules were accommodated. In **1a** and **2a**, both channels and cavities were generated. The channels were built from the arrangement, directly along *c* axis, of the hexagonal cavities consisting of six component molecules, while the cavities were formed between the individual 2D layers. In contrast, only cavities were produced in the crystal **3a**. This relatively large cavity was derived from six component molecules, which correspond to the cavities inducing into channels observed for **1a** and **2a**. The cavity observed between the 2D layers in **1a** and **2a** was not found in **3a**. These differences were probably due to the influence of steric methyl groups in the tripodal molecule **3** influencing the crystal packing. Namely, the nature of the arylester units was crucial for the formation of organic networks bearing both channels and cavities.

4. Conclusions

In this paper, we have constructed three different organic networks containing internal spaces from adamantane-centered tripodal molecules with **arylester** parts via intermolecular noncovalent interactions. Two crystals showed the formation of both channels and cavities in the crystalline lattices, showing that these tripodal adamantanes were versatile building blocks for shaping both channels and cavities. The creation of two different internal spaces in the crystalline materials is of use for the selective inclusion and molecular recognition of different kinds of guest molecules. Studies on the production of more complicated network structures bearing channels and cavities, and their chemical and physical properties are now in progress.

Supplementary data

Additional material comprised of the full X-ray data collection details and final refinement parameters, including anisotropic thermal parameters and full list of the bond lengths and angles, have been deposited with the Cambridge Crystallographic Data Centre in the CIF format as CCDC-903301, 903302, and 903303. Copies of the data can be obtained free of charge on the application to CCDC, 12 Union Road, Cambridge CB21EZ, UK, (Fax: +44 1233 336 033; e-mail: deposit@ccdc.cam.ac.uk). Supplementary data

associated with this article can be found in the online version, at doi:http://dx.doi.org/10.1016/j.molstruc.2013.03.033.

Acknowledgment

This work was supported by a Grant-in-Aid for Young Scientists (B) from the Ministry of Education, Culture, Sports, Science, and Technology of Japan (No. 22750113).

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