Supramolecular Crystal Engineering

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# Oligomeric Pseudorotaxanes Adopting Infinite-Chain Lattice Superstructures**

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Polyrotaxanes and polypseudorotaxanes[1] are relatively new types of polymeric materials, comprising polymer chains threaded through cyclic components in the presence or absence, respectively, of bulky stoppers at both ends of their polymeric chains. They often demonstrate[2] unusual macroscopic properties compared with their small-molecule counterparts[3]—that is, rotaxanes and pseudorotaxanes, respectively—in relation to properties such as stability, solubility, and viscosity, as well as thermal, mechanical, and electronic performance. Crystallinity, if it occurs, is one of the key factors affecting the thermal, optical, mechanical, and chemical properties of polymers, but it is difficult for long polymer chains—typically those in excess of 10 monomer repeating units or those with molecular weights higher than 1000 Da—to form macroscopic single crystals, because of both 1) diffusion limitations resulting from chain entanglements and 2) the heterogeneous length distributions of most synthetic polymers. Crystal structures of synthetic polymers are thus usually obtained[4] by computational model-building and diffraction simulation of X-ray powder- or fiber-diffraction data by using structural information from single-crystal analyses of small oligomeric homologues.[5] Although obtaining macroscopic single crystals of high-molecular-weight polyrotaxanes might seem to be even less realistic than it is for simple polymer chains, a small number of polypseudorotaxane crystal superstructures are known as a result of the end-to-end linking of small-molecule pseudorotaxanes by, for example, metal coordination[6] or hydrogen bonding[7] of the chain components. Cyclodextrins have also been found to crystallize with threaded chains, either of disordered surfactants[8] or of low-molecular-weight polyethylene or polypropylene glycols, which are present in channels created by the stacked cyclodextrin tori running through the crystalline lattices.[9]

Here, we report a series of single-crystal X-ray superstructures of donor–acceptor polypseudorotaxanes. Remarkably, although the oligomeric chains are undoubtedly discrete and monodisperse, they nevertheless appear to be infinite in the crystal. These polypseudorotaxanes lie on polymeric crystalline lattices with oligomer end-groups constituting crystallographically invisible defects in their superstructures. It follows that the solid-state superstructures of the corresponding high-molecular-weight "genuine" polypseudorotaxanes can be described in full atomic detail despite the fact that single crystals of these supramolecular polymers have not been, and may never be, obtained.

A series of donor–acceptor oligorotaxanes, currently under investigation[10] for their foldamer-like properties, have been obtained from oligomeric chains containing electron-rich 1,5-dioxynaphthalene (NP) units, separated by tetraethylene glycol loops, and with cyclobis(paraquat-*p*-phenylene) ($CBPQT^{4+}$) as the ring components. The secondary structures of these foldamers are stabilized by a combination of C−H···O hydrogen-bonding interactions and π–π stacking interactions between the π-electron-rich NP units in the polymer chains and the π-electron-deficient bipyridinium ($BIPY^{2+}$) units in the $CBPQT^{4+}$ rings. Single-crystal X-ray analyses of the complexes (Figure 1a) formed between $CBPQT^{4+}$ and both 3NPP and 3NPE have revealed the anticipated formation of [2]- and [3]pseudorotaxanes, respectively, in which one $CBPQT^{4+}$ ring encircles the central NP unit in the first case, while two such rings encircle the two outer NP rings in the second case. The crystal superstructure[11] of the corresponding pseudorotaxane formed from the

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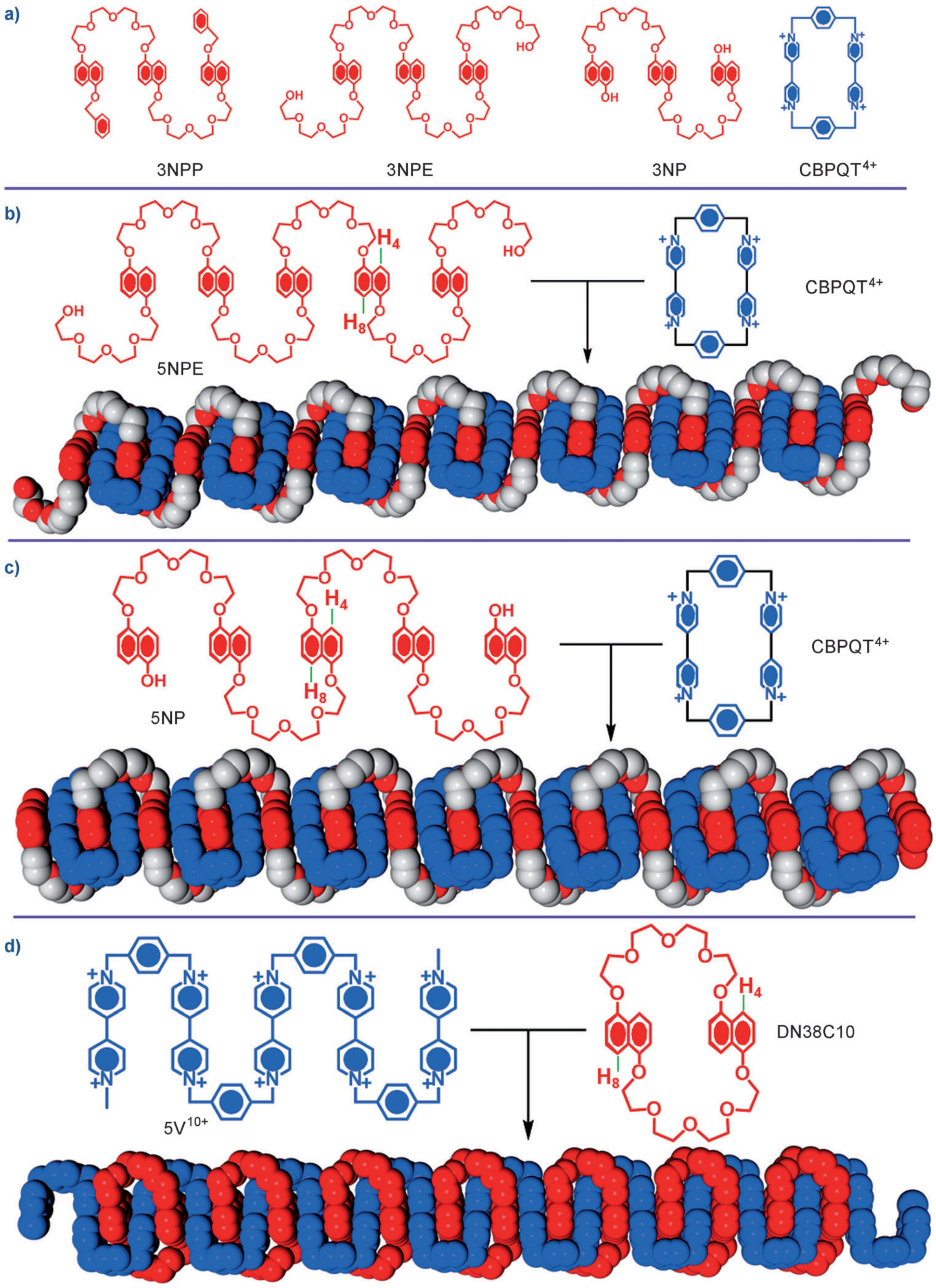

*Figure 1.* a) Structural formulas and space-filling representations of the X-ray single-crystal superstructures of the apparently infinite polypseudorotaxanes formed between CBPQT·4 $PF_6$ and b) 5NPE and c) 5NP, and d) between DN38C10 and 5V·10 $PF_6$.

pentameric 5NPE and CBPQT$^{4+}$ reveals, however, an apparently infinite polypseudorotaxane with the NP-containing oligomers extending indefinitely throughout the superstructure (Figure 1 b) and accommodating CBPQT$^{4+}$ rings on every alternate NP unit. Furthermore, crystallization of CBPQT·4 $PF_6$ with a completely different NP-containing oligomer—namely 5NP (Figure 1 c), where the terminal polyether chains present in 5NPE are now absent—results in the identical polypseudorotaxane superstructure[12] being observed (Figure 1 c) with the same space group and very similar unit-cell parameters for the crystal. Although these pseudorotaxanes appear to crystallize as supramolecular polymers, detailed spectroscopic analyses confirm (see Figures S1–S4 in the Supporting Information) that the oligomeric chains are indeed the pure, monodisperse oligomers 5NPE and 5NP, respectively.

It is clear that the NP chains in these superstructures are not high-molecular-weight polymers: rather, one in every six NP units present in the superstructure must, in reality, be absent. It is not immediately apparent where the vacancies occur: either encircled NP units (Figure 2 a) or those (Figure 2 b) in between adjacent CBPQT$^{4+}$ rings are missing. Moreover, because the solid-state superstructure appears to be polymeric, there is evidently no positional correlation between vacancies from chain to chain, since such a feature would result in an ordered, discrete-chain superstructure, which is not observed. The occupancies of the two crystallographically unique 5NPE superstructures (Figure 2 a,b) are thus mingled so that they sum to a 1/6th vacancy. Site occupancy factors for the encircled and non-encircled NP units were determined to be 0.900 and 0.767, respectively, thus indicating a preference for the vacancies to occupy regions between the CBPQT$^{4+}$ rings, rather than the cavities inside them.

There are two possible scenarios for the adoption of a “polymeric” crystal superstructure by the complexes formed between 5NP and CBPQT$^{4+}$. One (Figure 3 a) has only the polyether end-loops missing, so that the encircled “polymer chain” alternates between three and two CBPQT$^{4+}$ rings per oligomer. The other scenario (Figure 3 b) comprises only three CBPQT$^{4+}$ rings per oligomer, thus leaving the terminal polyether loops and terminal NP units vacant from between every clutch of three CBPQT$^{4+}$ rings. In the first case, the atoms of the polyether loops are absent in only 1/5th of their

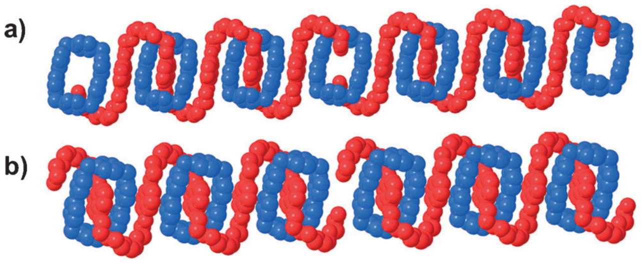

***Figure 2.*** Space-filling representations of the infinite-chain lattice superstructures of the complexes formed between 5NPE and CBPQT$^{4+}$ in which the vacancies occur a) within the cavities of the rings and b) between the rings.

sites, whereas, in the second case, the polyether loops and NP units are absent in 1/3rd of their sites. Both models were tested and a better refinement was achieved for the first one. Since other spectroscopic data suggest that the supramolecular species can exist in multiple states,[13] it is possible that this crystal comprises a mixture of two and three CBPQT$^{4+}$ rings on the 5NP units. In any event, the single-stranded polypseudorotaxanes[14] are organized into densely packed parallel-layered superstructures (see Figures S5 and S6 in the Supporting Information), with the spaces between the layers being filled with solvent molecules and counterions.

The adoption of polymeric lattices by oligomers in previous work can be ascribed[15] to the heterogeneous nature of the oligomeric chains and consequent disorder in the crystal,[16] whereas in the present study we show that polymeric lattices can be preferred even for rigorously pure, homogeneous oligomers. It would appear that the critical chain length for the formation of the polymer lattice in the crystal occurs between 3NPE and 5NPE. The fact that there must be a minimum critical chain length for the formation of the polymer lattice was confirmed by crystallizing the shorter 3NP oligomer with CBPQT$\cdot$4PF$_6$ to afford a [2]pseudorotaxane in which the ring encircles only the NP unit so as to maximize donor–acceptor interactions. This superstructure (see Figure S7 in the Supporting Information) is completely distinct from that found for the polypseudorotaxanes based on 5NPE and 5NP. Crystals of complexes incorporating longer, but still monodisperse, NP oligomers such as the 7-, 9- and 11-mers, grown in the presence of CBPQT$\cdot$4PF$_6$, had unit-cell dimensions identical to those of the complex formed between 5NP and CBPQT$^{4+}$. This finding indicates that these higher homologues also crystallize to form the same polymer lattice.

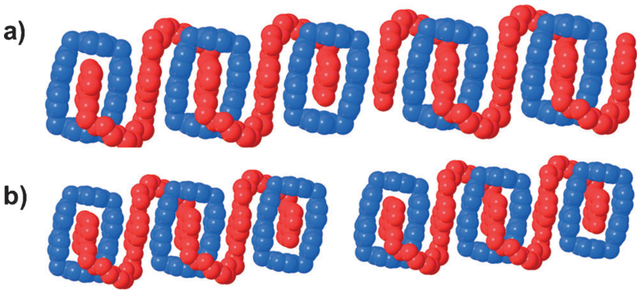

***Figure 3.*** Space-filling representations of the infinite-chain lattice superstructures of the complexes formed between 5NP and CBPQT$^{4+}$, showing the presence of a) vacant polyether end loops and b) vacancies between CBPQT$^{4+}$ rings.

To test the generality of the principle of polypseudorotaxane formation in donor–acceptor complexes, the electronic properties of the oligomeric and ring components were reversed. Even though the electron-deficient oligomer[17] 5V$^{10+}$ is much more compact and rigid than its 5NPE counterpart,[18] single-crystal X-ray analysis[19] of the complex formed between 5V$\cdot$10PF$_6$ and 1,5-dioxynaphtho[38]crown-10 (DN38C10)[20] once again reveals (Figure 1 d) a polypseudorotaxane in which the DN38C10 ring encircles every other BIPY$^{2+}$ unit in the oligomer, which once again adopts a zigzag conformation. This solid-state superstructure is analogous to that observed for the complex formed between 5NP and CBPQT$\cdot$4PF$_6$. To produce discrete 5V$^{10+}$ chains, one in every five paraphenylene linkers has to be absent. The atoms in the paraphenylene rings were thus refined with a site occupancy factor of 0.80. In this case, the oligomeric chains contain five BIPY$^{2+}$ units and are either encircled by two or three DN38C10 rings in a crystal, comprising a mixture of both pseudorotaxanes (Figure 4). Once again, there are no long-range, longitudinal correlations between chains, leading to a pseudopolymeric superstructure. The single-strand polypseudorotaxane is organized into a densely packed parallel-layered superstructure (see Figure S10 in the Supporting Information), with the space between the layers being filled with solvent molecules and counterions. The requirement for a minimum critical chain length for polypseudorotaxane formation in the crystal is also evident here, from a comparison with the discrete [2]pseudorotaxane observed[21] previously in the solid-state superstructure of 3V$\cdot$6PF$_6$$\subset$DN38C10 comprising three BIPY$^{2+}$ units and a DN38C10 ring.

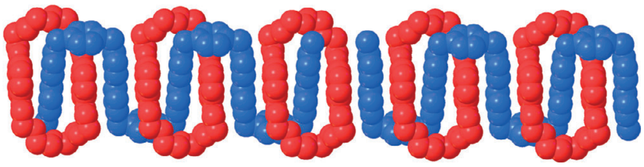

***Figure 4.*** Space-filling representations of the infinite-chain lattice superstructures of the complexes formed between 5V$\cdot$10PF$_6$ and DN38C10, showing a vacant paraphenylene linker.

Thus, above a certain chain length, the superstructures derived from mixed pseudorotaxane components adopt the same crystal lattices as those based on authentic high-molecular-weight polypseudorotaxanes. The crystal superstructures of these polypseudorotaxanes can be employed to predict the corresponding crystal structures of related polyrotaxanes, even when the latter cannot be obtained in a single-crystalline form.

## Experimental Section

Complex between 5NPE and CBPQT$\cdot$4PF$_6$: Colorless solutions of 5NPE in MeCN (4 mM, 0.2 mL) and CBPQT$\cdot$4PF$_6$ in MeCN (4 mM, 0.6 mL) were mixed together to produce a deep-red solution, which was passed through a Pall syringe filter (pore size 0.45 μm) into VWR culture tubes ($6\times50$ mm). The tubes were allowed to stand at room temperature in a closed scintillation vial containing $i$Pr$_2$O (5 mL). After 1 week, red crystals appeared in the tubes, from which a red tabular crystal, with dimensions of about $0.16\times0.08\times0.05$ mm$^3$, was selected and mounted using oil (Infineum V8512) on a glass fiber and transferred to the cold gas stream cooled by liquid N$_2$ on a Bruker APEX-II CCD with graphite monochromated Mo$_{K\alpha}$ radiation. The structure was solved by direct methods and refined subsequently using OLEX2 software.[22]

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[11] Crystal data for the complex formed between 5NPE and CBPQT$\cdot$4PF$_6$: C$_{153.333}$H$_{172}$F$_{48}$N$_{16}$O$_{20}$P$_8$, $M_r = 3718.83$, monoclinic, space group $P2_1/c$, $a = 14.0613(6)$, $b = 13.9204(5)$, $c = 21.2119(8)$ Å, $\beta = 97.593(3)°$, $V = 4115.6(3)$ Å$^3$, $T = 100(2)$ K, $Z = 1$, $\rho_{\text{cald}} = 1.500$ g cm$^{-3}$, $\mu(\text{Mo}_{K\alpha}) = 0.209$, $F(000) = 1916$. Independent measured reflections 33289. $R1 = 0.068$, $wR2 = 0.249$ for 2466 independent observed reflections $[2\theta \leq 41.6°, I > 2\sigma(I)]$. CCDC 872021 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

[12] Crystal data for the complex formed between 5NP and CBPQT$\cdot$4PF$_6$: C$_{153.6}$H$_{163.2}$F$_{48}$N$_{16}$O$_{17.6}$P$_8$, $M_r = 3674.76$, monoclinic, space group $P2_1/c$, $a = 14.0020(5)$, $b = 13.7439(4)$, $c = 21.1562(7)$ Å, $\beta = 97.422(2)°$, $V = 4037.2(2)$ Å$^3$, $T = 100(2)$ K, $Z = 1$, $\rho_{\text{cald}} = 1.500$ g cm$^{-3}$, $\mu(\text{Cu-K}\alpha) = 1.907$, $F(000) = 1890$. Independent measured reflections 17230. $R1 = 0.167$, $wR2 = 0.425$ for 3957 independent observed reflections $[2\theta \leq 117.8°, I > 2\sigma(I)]$. CCDC 872023 contains the supplementary crystallographic data for this paper..

[13] Multiple states for the complexes formed between 5NP and CBPQT$\cdot$4PF$_6$ were confirmed by HRMS. For example, [5NP + 2CBPQT$\cdot$4PF$_6$]: calcd for C$_{154}$H$_{160}$F$_{30}$N$_8$O$_{22}$P$_5$: 1065.9952 for $[M-3\text{PF}_6]^{3+}$, found 1065.9896; while [5NP + 3CBPQT$\cdot$4PF$_6$]: calcd for C$_{190}$H$_{192}$F$_{54}$N$_{12}$O$_{22}$P$_9$: 1432.7017 for $[M-3\text{PF}_6]^{3+}$, found 1432.7034.

[14] In both pseudopolymeric superstructures, whereas the NP units are essentially planar and strain-free in the oligomers, the BIPY$^{2+}$ units in the cyclophanes are distorted by the strain imposed by the CBPQT$^{4+}$ ring itself. Thus, the CH$_2$–N$^+$ bonds in the cyclophanes subtend angles of about 156° and 159°, respectively, in the complexes involving 5NPE and 5NP units, that is, deviations of 24° and 21°, respectively, from the colinearity associated with the conventional planar geometries of BIPY$^{2+}$ units. The mean planes of the NP and BIPY$^{2+}$ units are oriented parallel to another. The supramolecular assemblies in both cases are stabilized by a) C-H$\cdots$O interactions (F. M. Raymo, M. D. Bartberger, K. N. Houk, J. F. Stoddart, *J. Am. Chem. Soc.* **2001**, *123*, 9264–9267) between the protons α to nitrogen atoms in the BIPY$^{2+}$ units and some of the oxygen atoms in the polyether loops and b) C-H$\cdots\pi$ interactions between the *peri*-hydrogen atoms of the encircled NP units and the planes of the paraphenylene linkers in the CBPQT$^{4+}$ rings, as well as donor–acceptor interactions between the electron-rich NP units and the electron-deficient BIPY$^{2+}$ units. While the separations between the encircled NP units and their interacting BIPY$^{2+}$ units are 3.41 Å for the complex involving 5NPE, and 3.40 Å for the one involving 5NP, the separations between the alongside NP units and the two BIPY$^{2+}$ units on neighboring CBPQT$^{4+}$ rings are 3.41 Å and 3.34 Å, respectively, for the first and second complexes.

[15] There is only one previous example (D. J. Williams, H. M. Colquhoun, C. A. ÓMahoney, *J. Chem. Soc. Chem. Commun.* **1994**, 1643–1644) of a polymeric lattice being adopted by well-defined oligomers, namely that involving *m*-deciphenyl (*m*-undeciphenyl). Crystals of these oligomers show generalized longitudinal disorder in a polymeric lattice with atomic positions in the structure of *m*-deciphenyl refining satisfactorily to the expected occupancies. It is noteworthy that the formation of the polymer-type lattice by this system is evidently destabilized by high concentrations of chain ends, since discrete molecules are observed (P. W. Rabideau, A. Sygula, R. K. Dhar, F. R. Fronc-

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[17] 5V·10PF$_6$ was prepared (see Scheme S1 in the Supporting Information) and characterized by $^1$H NMR spectroscopy (Figure S8) and HRMS (Figure S9).

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[19] Crystal data for the complex formed between 5V·10PF$_6$ and DN38C10: $C_{85.6}H_{98.4}F_{24}N_{12}O_{10}P_4$, $M_r = 2035.24$, triclinic, space group $P\bar{1}$, $a = 13.4967(2)$, $b = 13.5574(2)$, $c = 14.5137(3)$ Å, $\alpha = 75.5240(10)$, $\beta = 72.3740(10)$, $\gamma = 75.1900(10)°$, $V = 2403.66(7)$ Å$^3$, $T = 100(2)$ K, $Z = 1$, $\mu(\text{Cu-K}\alpha) = 1.679$, $F(000) = 1052$. Independent measured reflections 8415. $R1 = 0.117$, $wR2 = 0.350$ for 6092 independent observed reflections $[2\theta \leq 34.8°, I > 2\sigma(I)]$. CCDC 872024 contains the supplementary crystallographic data for this paper..

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