In conclusion, by determining the solution structure of **1**, we have demonstrated that *N*,*N*′-linked oligoureas of general formula **B** belong to the growing family of non-natural non-peptide oligomers with defined and predictable secondary structures. Although heptaurea **1** forms a (*P*)2.5 helix of approximately 5.1 Å pitch that is closely related to the $(P)2.6_{14}$ helix of approximately 5 Å pitch of corresponding γ^4-peptides,[4a] it is worth noting that both NH groups within the same urea linkage may participate in intramolecular hydrogen bonding to the same C=O group. The knowledge of the three-dimensional structure of **1** is likely to be useful for the de novo design of oligoureas with controlled shape and defined biological activities.

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Self-Assembling Organic Nanotubes from Enantiopure Cyclo-*N*,*N*′-Linked Oligoureas: Design, Synthesis, and Crystal Structure

Vincent Semetey, Claude Didierjean, Jean-Paul Briand, André Aubry, and Gilles Guichard*

Assembly of self-complementary cyclo-oligomeric subunits through noncovalent processes (for example, hydrogen bonding, aromatic stacking) has emerged as a powerful strategy to generate artificial organic nanotubular structures.[1, 2] Highly functionalized tubular assemblies based on peptides have attracted much interest recently in this area. Ghadiri and co-workers have compellingly demonstrated that 24- and 30-membered-ring cyclo-α-peptides with an even number of alternating D- and L-amino acids stack in an antiparallel β-sheet-like arrangement to form hydrogen-bonded tubular structures, that is, "peptide nanotubes".[2–4] Remarkably, related cyclic peptides consisting exclusively of β-amino acids[5, 6] (16- and 12-membered ring), of alternating α- and β-amino acids[7] (14-membered ring), or of vinylogous δ-amino acids[8] (18-membered ring) also form tubular stacks.

We have shown previously[9] that linear *N*,*N*′-linked oligoureas **A** consisting of homochiral residues adopt a stable 2.5-helical secondary structure in solution. The helix is characterized by the simultaneous presence of 12- and 14-membered hydrogen-bonded rings resulting from the capacity of the urea group to establish self-complementary bidirec-

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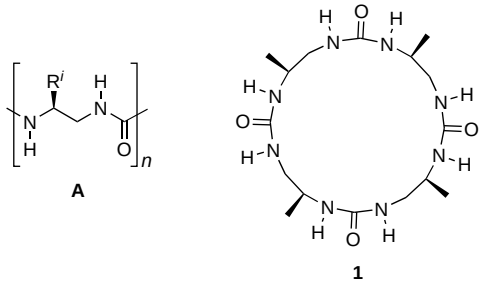

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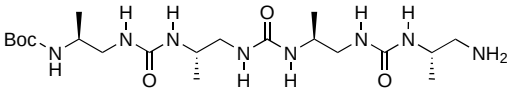
tional intermolecular hydrogen bonds. We envisioned that the corresponding macrocyclic derivatives might lead to flat ring structures with a high degree of self-complementarity that would allow self-assembling processes to occur. Herein we report the design and the synthesis of the C_4-symmetric (all-*S*) cyclotetraurea **1** bearing side chains of alanine, and demonstrate by single-crystal X-ray analysis that **1** forms square-shaped nanotubes.

The general stepwise strategy used to synthesize the 20-membered-ring macrocycle **1** is outlined in Scheme 1. Reaction of the succinimidyl carbamate derivative **2**[10] with the monoprotected benzylated diamine **3** yielded the trisubstituted urea **4**. Removal of the Boc group followed by urea formation with **2** gave the bisurea **5**. Repetition of this two-step sequence gave the fully protected triurea **6** in an overall yield of 45% from **3**. Following quantitative removal of the Alloc group, the resulting amine **7** was converted into the key activated linear precursor **8** by treatment with di(*N*-succinimidyl carbonate). At this stage, the temporary protection of the urea with a benzyl group was mandatory. Initial attempts to convert the linear oligotriurea precursor **11** under the same conditions were not successful and led exclusively to the formation of the cyclic biuret derivative **12**.



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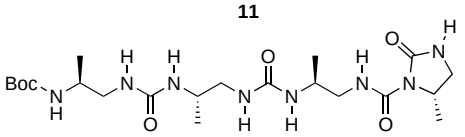

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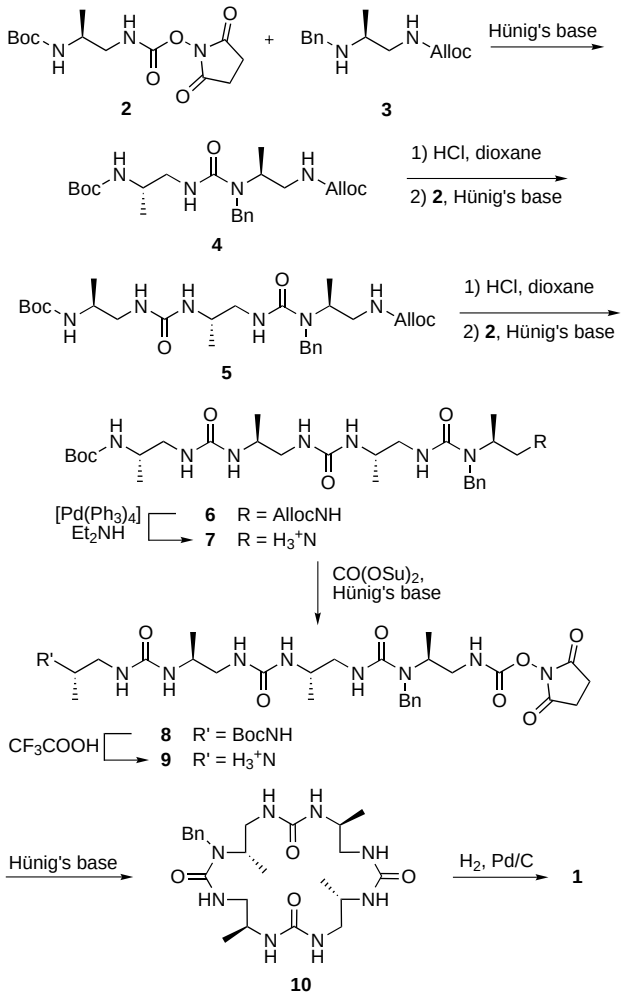

Scheme 1. Synthesis of **1**. Alloc = allyloxycarbonyl, Boc = *tert*-butoxycarbonyl, Bn = benzyl, Hünig's base = diisopropylethylamine, Su = succinimidyl.

The Boc protecting group was selectively removed upon treatment of crude **8** with CF_3COOH to give the trifluoroacetate salt **9**. Slow addition of a solution of **9** in MeCN to a dilute solution of Hünig's base in MeCN resulted in the formation of the expected monobenzylated cylic oligotetraurea **10** in a yield of 62% from **7** after HPLC purification (C_{18} column, reversed phase). Cyclotetraurea **1** was obtained in 92% yield from **10** following deprotection of the urea group by hydrogenation in EtOH. Single crystals of **1** suitable for X-ray studies were grown by slow evaporation of the EtOH solution.

X-ray crystallographic analysis[11] revealed that cyclourea **1** adopts a C_4-symmetric conformation in the solid state and stacks to form a hollow tubular structure (Figure 1). The four

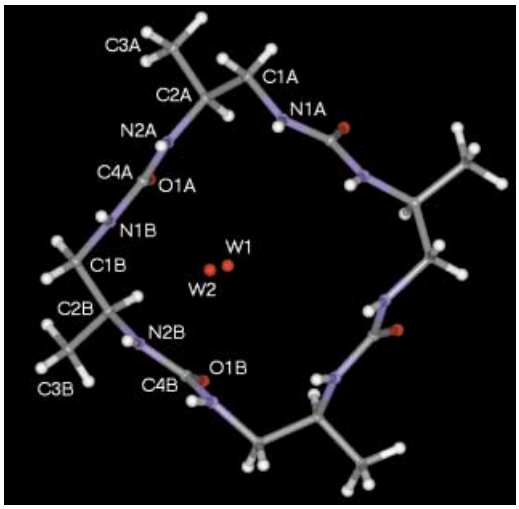

Figure 1. Ball and stick representation of **1** showing the numbering scheme. The torsion angles [°] of the main chain are : N1A-C1A-C2A-N2A 57.9(2), C1A-C2A-N2A-C4A −156.7(2), C2A-N2A-C4A-N1B 177.4(2), N2A-C4A-N1B-C1B 172.6(2), C4A-N1B-C1B-C2B 78.0(3).

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urea fragments in the macrocyle present an all-*trans*, planar conformation with all the urea carbonyl groups pointing down and all the NH groups pointing up (Figure 1). The macrocyle exhibits a square shape with a cross-section of 6.052(7) Å (distance between C1A and C1B).

A difference map analysis revealed the presence of two peaks located on the fourfold symmetry axis. The distance between the two peaks was 1.42 Å. NMR experiments gave no evidence for the presence of any organic solvent molecules in the crystal, thus the two peaks were treated as disordered water molecules W1 and W2. The internal van der Waals diameter (approximately 3.5 Å) of the tubular structure is actually large enough to accommodate such molecules. The distance between the two water molecules is too short to have both sites occupied at the same time. There is no short atomic contact favoring hydrogen bonding between the water molecules W1 and W2 and the macrocyle.

The square-shaped cycloureas stack along the crystallographic fourfold axis of the crystal through backbone–backbone hydrogen-bonding interactions so that the two NH hydrogen atoms of each urea fragment interact with a urea oxygen atom of a neighboring macrocycle (Figure 2).

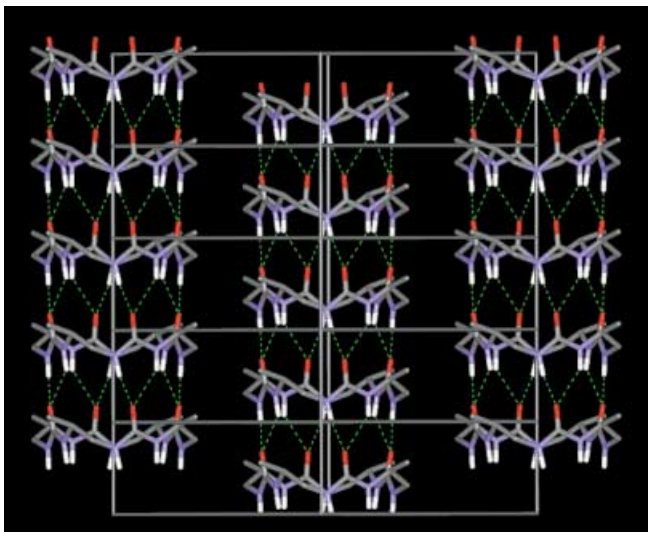

Figure 2. View of the nanotubular organization of **1** along the channel axis. The intermolecular hydrogen bonds are marked as dashed lines. The solvent molecules and H atoms, except those of the NH groups, are omitted for clarity. The edges of the unit cells are represented in gray.

One of the intermolecular N···O distances has standard dimensions (2.798(2) Å), while the other (3.281(2) Å) is just above the limit generally considered for hydrogen-bonding interactions (Figure 2). This stacking of the macrocyles differs from that observed in the crystal structure of cystine-based macrocyclic bisureas that also exhibits a backbone–backbone urea-type hydrogen-bonding interaction.[12] As a consequence of the absence of a local symmetry for the motif C1A–C1B (Figure 1), the urea N–H and C=O bonds in **1** are not parallel to the channel axis and generate two different N···O contacts. Each cyclourea is perpendicular to and centered on the crystallographic quaternary axis in the crystal of **1**. The stacking of the cyclourea units along the crystallographic *c* axis gives rise to an infinite nanotube. The crystal obtained exhibits a body-centered Bravais lattice, and thus the X-ray structure consists of an array of nanotubes in which each tubular stack is surrounded by four close neighbors at a distance of $a\sqrt{2}/2$ Å ($a = 15.333(3)$ Å is one of the unit cell parameters). Neighboring tubes are all arranged in the same direction.[13]

The nanotubes are held together in the crystal by loose van der Waals contacts. The intermolecular distances between equivalent C1A atoms and equivalent C3A atoms are 4.097(7) and 4.218(10) Å, respectively.

In summary, we have reported a versatile approach to the synthesis of 20-membered cyclic oligoureas bearing proteinogenic side chains. The cyclic units consisting of homochiral residues self-assemble in the crystal state to form hydrogen-bonded polar nanotubes. The structure of the tubular stack with all the carbonyl and NH groups pointing, respectively, in the same direction is reminiscent of the nanotubes formed by cyclotetra-β-peptides with homochiral residues.[5a] Particularly noteworthy is the presence of electronic density inside the tubular cavity which was modeled as disordered water molecules at overlapping sites. Further work to explore other tubular arrangements made from cycloureas with different side-chain functionalities and different stereochemistry is on going.

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[11] Crystal data: $C_{16}H_{32}N_8O_4$, $M_r = 400.48\ g\,mol^{-1}$, colorless prism, crystal size $0.3 \times 0.1 \times 0.1\ mm^3$, $a = b = 15.333(3)$, $c = 4.724(1)$ Å, $V = 1110.6(4)$ Å^3, $T = 293$ K, tetragonal, space group $I4$, $Z = 2$, $\rho_{calcd} = 1.226\ Mg\,m^{-3}$, $\mu = 0.763\ mm^{-1}$. Nonius Mach3 diffractometer, $\lambda = 1.54178$ Å. 4293 measured reflections, 1052 unique ($R_{int} = 0.017$), 953 with $I > 2\sigma(I)$, the structure was solved by direct methods and refined by full-matrix least squares on F^2 for all data to $R_1 = 0.0411$ [$I > 2\sigma(I)$] and $wR_2 = 0.1036$ [$I > 2\sigma(I)$], 93 parameters. CCDC 175843 contains 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 CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

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Drastic Luminescence Response to Carbon Monoxide from a Ru^{II} Complex Containing a Hemilabile Phosphane Pyrene Ether**

Cerrie W. Rogers and Michael O. Wolf*

The rapid, reversible nature of the coordinative bonding between metals and Lewis-basic small molecules makes molecular chemosensors based on this phenomenon quite promising. Indeed, recent reviews contain numerous reports of sensors based on metal – ligand interactions.[1–3] Of particular potential are metal complexes designed such that the analyte-binding event changes the luminescence properties of an intramolecular lumophore. One approach is to use a modular design in which a metal-based analyte receptor is covalently linked to a separate lumophore. The emission of the lumophore is influenced by the binding of the analyte, usually through On – Off switching of energy- or electron-transfer quenching mechanisms.[1, 2, 4] Pyrene, which emits intense indigo-blue fluorescence in dilute solution[5] and blue-green excimer emission in concentrated solution,[5, 6] is a popular lumophore for such molecule-based sensors, particularly those based on photoinduced electron transfer (PET) processes.[2, 7] Switching between pyrene monomer and excimer emission has also been used to obtain a sensor response, whereby the ability of pyrene moieties to interact with one another is influenced by the binding of analyte to the receptor.[2, 8]

We used a [$RuCl_2$(POR-*P*,*O*)$_2$] complex (where POR is an ether-substituted phosphane) as the basis for a modular molecular sensor. This type of complex reacts rapidly and reversibly with numerous Lewis-basic small molecules, including CO,[9–11] a small-molecule analyte for which effective new sensor materials may be useful, by displacement of the labile ether moiety. Incorporation of a pyrene group in the hemilabile ligand leads to metal-based reactivity towards small molecules and pyrene-based luminescence. Here a Ru^{II} complex containing the hemilabile phosphane ether ligand 4-{2-(diphenylphosphanyl)phenoxy}butylpyrene (POC4Pyr) is described. The complex [$RuCl_2$(POC4Pyr-*P*,*O*)$_2$] (**1**, see Scheme 1) reacts with carbon monoxide to produce a significant luminescence response in which monomer-to-excimer emission switching is observed. This is the first example of the use of the hemilabile-ligand approach to obtain a molecular sensor with a room-temperature luminescence response, although we have previously reported a Ru bipyridyl complex containing a hemilabile ligand that exhibits small-molecule-dependent luminescence at low temperature.[12]

Complex **1** was prepared by reaction of POC4Pyr with $RuCl_3 \cdot x\,H_2O$ in boiling deaerated ethanol/toluene. The burgundy-pink complex is mildly sensitive to oxidation by air, both in solution and as a solid. The $^{13}C\{^1H\}$ and $^{31}P\{^1H\}$ NMR data of **1** (Tables 1 and 2) are analogous to those of [$RuCl_2$(POMe-*P*,*O*)$_2$] (POMe = (2-methoxyphenyl)diphenylphosphane), which has been crystallographically characterized[9] and found to contain two *P*,*O*-coordinated phosphane ether ligands with the phosphane moieties *cis* to each other.

The absorption spectrum of complex **1** is essentially a combination of those of [$RuCl_2$(POMe-*P*,*O*)$_2$] and the pyrenyl ligand POC4Pyr. The color of the complex is caused by a weak

Table 1. Summary of $^{13}C\{^1H\}$ NMR spectroscopic data[a] for **1** – **3**.

Complex	CO	δ/ppm (multiplicity) [*J*/Hz] *ortho*[b]	*ipso*	*ipso'*
1	–	161.7 (t) [5.2]	134.1 (d) [25.3]	ND[c]
2	197.6 (t) [13.5]	160.5 (t) [2.3]	132.8 (t) [24.4]	118.8 (t) [23.9]
3	193.7 (t) [10.9]	159.0 (t) [2.3]	131.8 (t) [24]	119.3 (t) [23.5]

[a] In CD_2Cl_2. [b] Phenyl C atom bound to oxygen. [c] ND = not determined.

Table 2. Comparison of **1** – **3** with analogous POMe complexes.[a]

Complex	Color	$\delta(^{31}P\{^1H\})$/ppm	$\tilde{\nu}$(C=O)/cm^{-1}
tcc-**1**	red	63.7[b]	–
ttt-**2**	greenish yellow	27.1[b]	2005[c]
cct-**3**	greenish yellow	13.9[b]	2005, 2058[c]
tcc-[$RuCl_2$(POMe-*P*,*O*)$_2$]	red	62.7[d]	–
ttt-[$RuCl_2(CO)_2$(POMe-*P*)$_2$]	yellow	26.7	1962
cct-[$RuCl_2(CO)_2$(POMe-*P*)$_2$]	white	10.6	2000, 2060

[a] From ref. [9]; $^{31}P\{^1H\}$ NMR spectra measured in $CDCl_3$ solution; IR spectra measured in mineral-oil mulls. [b] In CD_2Cl_2. [c] In $CHCl_3$. [d] In $CDCl_3$.

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