

The wires reported here are suitable for low-loss optical wave guiding, and will be promising components in future microphonic devices for various applications, such as optical communications and optical sensing. Owing to their excellent uniformity, large length, high flexibility, and strength, these wires can be manipulated and assembled with high accuracy and used as micro- or nanoscale tools in physical, chemical, biological, micro-electronic and materials research. □

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Synthesis of a Möbius aromatic hydrocarbon

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The defining feature of aromatic hydrocarbon compounds is a cyclic molecular structure stabilized by the delocalization of π electrons that, according to the Hückel rule, need to total $4n + 2$ ($n = 1, 2, \dots$); cyclic compounds with $4n$ π electrons are antiaromatic and unstable. But in 1964, Heilbronner predicted¹ on purely theoretical grounds that cyclic molecules with the topology of a Möbius band—a ring constructed by joining the ends of a rectangular strip after having given one end half a twist—should be aromatic if they contain $4n$, rather than $4n + 2$, π electrons. The prediction stimulated attempts to synthesize Möbius aromatic hydrocarbons, but twisted cyclic molecules are destabilized by large ring strains, with the twist also suppressing overlap of the p orbitals involved in electron delocalization and stabilization. In larger cyclic molecules, ring strain is less pronounced but the structures are very flexible and flip back to the less-strained Hückel topology^{2,3}. Although transition-state species⁴, an unstable intermediate⁵ and a non-conjugated cyclic molecule⁶, all with a Möbius topology, have been documented, a stable aromatic Möbius system has not yet been realized. Here we report that combining a ‘normal’ aromatic structure (with p orbitals orthogonal to the ring plane) and a ‘belt-like’ aromatic structure (with p orbitals within the ring plane) yields a Möbius compound stabilized by its extended π system.

Numerous theoretical calculations have been performed to predict the properties of potential Möbius aromatic systems^{7–12}. The twist in the π system is usually introduced by a suitable arrangement of E and Z double bonds. *Trans*-benzene¹³ is the smallest conceivable antiaromatic Möbius annulene. It is only a very shallow minimum on the energy hypersurface, with an energy as much as 107 kcal mol⁻¹ above benzene¹⁴. The next-higher Möbius homologue, *trans*-cyclooctatetraene, indeed comes energetically closer to its Hückel (all-*cis* and global minimum) isomer¹⁴. Nevertheless, with a relative energy of 21.3 kcal mol⁻¹, it is expected to be extremely difficult to synthesize. Moreover, there is almost no conjugation between the E and the neighbouring Z double bonds

Table 1 Relative stabilities

E_{ref}	[16]annulene		E_{ref}	Möbius stabilized [16]annulene	
	Topology	Sym.		Topology	Sym.
0.0	Hückel	S ₄	0.0	Möbius	C ₁
2.0	Hückel	C ₁	0.3	Möbius	C ₂
5.1	Möbius	C ₁	2.8	Möbius	C ₂
7.6	Möbius	C ₂	2.8	Möbius	C ₁
15.8	Möbius	C ₂	6.7	Möbius	C ₁
51.4	Möbius	C ₂	7.0	Hückel	C _s
			8.3	Möbius	C ₁

Calculated relative stabilities (E_{ref} in kcal mol⁻¹) of the most stable isomers of the parent [16]annulene and of our modified [16]annulene at the B3LYP/6-31G* level of DFT.

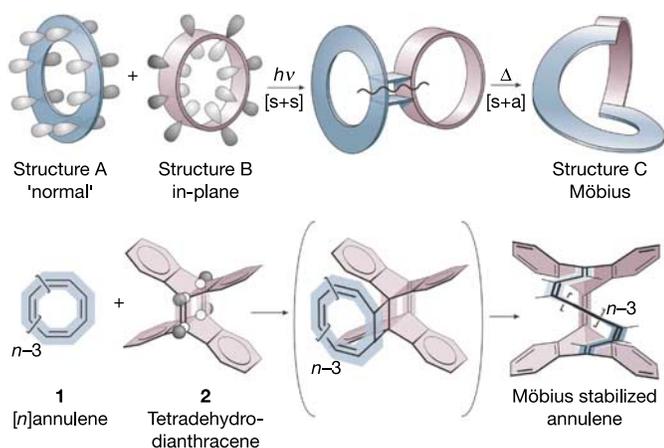


Figure 1 Strategy to stabilize the Möbius structure of annulenes. Möbius bands (structure C) contain a 'normal' trilateral planar conjugated part (shown cyan) and a pyramidalized 'in-plane' part (magenta). The strain arises from the latter. Combining a normal conjugated ring (structure A; for example, $[n]$ annulene **1**) with a rigid 'prefabricated' pyramidalized in-plane conjugated building block (structure B; for example, tetrahydrodianthracene **2**) by metathesis leads to rings that are more stable in the twisted Möbius configuration than in the untwisted Hückel configuration. A supra supra allowed $[2 + 2]$ addition ($[s + s]$) followed by a thermal supra antara cycloreversion ($[s + a]$) leads to a Möbius system.

because of the large deformation of the ring induced by the twist. Castro and Schleyer¹⁵ have published an extensive study of various isomers and conformations of higher annulenes. One of the most interesting structures is a $[16]$ annulene with an $E,Z,Z,Z,Z,Z,E,E,Z,E,E,Z,Z,Z,Z$ arrangement of conjugated bonds that exhibits distinct Möbius aromaticity. The relative energy is $15.3 \text{ kcal mol}^{-1}$ above the global minimum isomer. In view of the large number of conceivable isomers—many of which are more stable than this strongly conjugated Möbius compound—the directed synthesis or isolation of this compound is expected to be a very difficult task.

Our synthetic approach is therefore based on a general strategy that stabilizes Möbius structures (Fig. 1). In 'normal' aromatic structures like benzene, the p orbitals are orthogonal with respect to the ring plane (Fig. 1, structure A, cyan) whereas in belt-like systems (for example, carbon nanotubes) the p orbitals are perpendicular with respect to the surface of a cylinder (Fig. 1, structure B, magenta). A Möbius system (Fig. 1, structure C) includes both types of conjugation: a 'normal' part with trigonal planar sp^2 -hybridized atoms, and a belt-like system with pyramidalized sp^2 atoms. The strain arises from pyramidalization. Thus, introducing a 'prefabricated' pyramidalized building block (Fig. 1, bottom, magenta) in a ring should stabilize the Möbius- versus Hückel-type isomers.

We chose bianthraquinodimethane as this rigid in-plane conjugated part. The steric hindrance of the inner *ortho* protons of the anthracene units enforces the pyramidalization of the central quinoid bond. Ring enlargement metathesis of tetrahydrodianthracene (TDDA) **2** (refs 16–18) with cyclooctatetraene **1** ($n = 1$) should lead to the twisted annulenes.

To test our strategy, preliminary theoretical calculations were performed on the Möbius stabilized $[16]$ annulene. The geometrically fixed anthraquinodimethane part reduces the number of conceivable E/Z isomers to 12, each of which can have 9 *s-cis/s-trans* conformations. We optimized all 108 isomers using the PM3 method¹⁹, and performed density functional theory (DFT) calculations²⁰ at the B3LYP/6-31G* level^{21,22} of theory on the most stable conformation of the 12 most stable isomers. In Table 1 the relative energies of our Möbius stabilized annulene are compared with the results of Castro and Schleyer¹⁵ on the parent $[16]$ annulene.

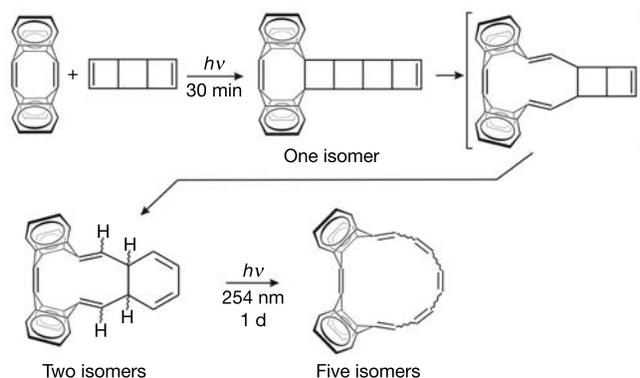


Figure 2 Reaction of *syn*-tricyclooctadiene with tetrahydrodianthracene.

Whereas in the parent $[16]$ annulene the two most stable isomers have Hückel topology, the six most stable isomers of our modified $[16]$ annulene are Möbius-type systems including the global minimum. Hence, the theoretical data confirm our Möbius stabilizing strategy, and on the basis of these thermodynamic data, the synthesis of a Möbius system should be feasible.

Unfortunately, all attempts to combine TDDA with cyclooctatetraene in a ring enlargement metathesis reaction failed. Even though benzene and a number of alkenes upon irradiation react with TDDA to form metathesis products, cyclooctatetraene under the same conditions only furnished Diels–Alder adducts, isomers of cyclooctatetraene and 9,9'-bianthracene. To circumvent these problems, we used *syn*-tricyclooctadiene (TCOD) as a synthetic equivalent of cyclooctatetraene²³. Irradiation of TDDA and TCOD in benzene with a high-pressure mercury lamp, using a quartz apparatus, gave as the main products two 1,3-cyclohexadiene derivatives (Fig. 2; see Supplementary Information for a detailed experimental procedure).

In the first step, a $[2 + 2]$ cycloaddition product is formed, which could be isolated and characterized by NMR. This ladder-shaped compound, containing four fused cyclobutane rings, undergoes cycloreversion to give an unstable intermediate, which again opens in an electrocyclic reaction to form a C_2 and a C_s symmetric 1,3-cyclohexadiene. Both isomers were isolated, and characterized by X-ray structure analysis. Only minor amounts of the desired fully ring opened products were detected. As it is known that the photochemical stationary equilibrium between 1,3-cyclohexadienes and hexatrienes is shifted towards the ring opened product at shorter wavelengths, we irradiated the reaction mixture for 24 h with a low-pressure mercury lamp and obtained a mixture of isomers in an overall combined yield of about 50%. Five ring opened isomers were isolated by high-performance liquid chromatography. Two of them exhibit Möbius topology (C_1 and C_2 symmetry) and one has Hückel topology (C_s symmetry). The two Möbius structures were elucidated by NMR, and confirmed by X-ray analysis (for details of the X-ray data of the C_2 Möbius compound, see Supplementary Information; Cambridge Crystallographic Data Centre deposition number 223927). Both geometries are in very good agreement with DFT calculations. Only the C_2 symmetry isomer is significantly conjugated. The structural parameters of the Hückel C_s structure are not accurate because of disorder (<10% of a second isomer), but the data confirm the structural assignment by NMR. We use the Hückel structure as a reference to investigate the properties of our new Möbius C_2 compound (Fig. 3).

The C_2 symmetry Möbius compound forms red crystals, whereas the C_s symmetry Hückel compound is colourless (for ultraviolet data, see Supplementary Information). In Fig. 3, the most important geometry parameters calculated at the B3LYP/6-31G* level of

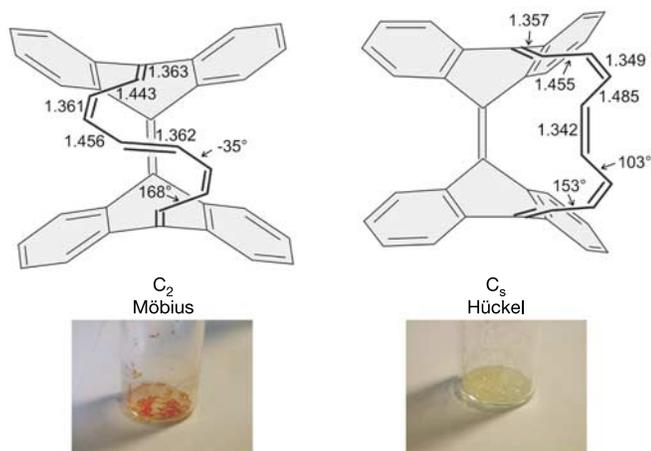


Figure 3 X-ray structures and photographs of the crystals of the C_2 Möbius and the C_s Hückel isomer. The structure plots are idealized for clarity (thermal ellipsoids are removed, and the bond orders are indicated by single and double lines). Structural parameters for the Hückel isomer are not reliable because of disorder (see text). Therefore the bond lengths (Å) and C=C=C dihedral angles for the polyene bridge shown in the figure are determined by DFT calculations (B3LYP/6-31G*). For the complete set of X-ray data and calculations, see Supplementary Information.

DFT are given. Whereas the rigid bianthraquinodimethane part is almost identical in both isomers, the geometry of the polyene bridge differs substantially. Bond-length equalization, which is used as an aromaticity probe, is more pronounced in the Möbius compound than in the Hückel structure. The HOMA value²⁴, which quantifies this effect, is 0.50 in the polyene bridge (9 bonds) of the Möbius isomer, and 0.05 in the Hückel isomer. For comparison, the prototype aromatic benzene has a HOMA value of 0.98. The total HOMA (all bonds included) of the Möbius isomer is 0.35, and the HOMA of the Hückel structure is 0.17. A value of 0.35 corresponds to the HOMA in the benzene rings of C_{60} , and is somewhat lower than the corresponding value of the central benzene ring in phenanthrene (0.40). The Hückel isomer escapes from the 16-electron antiaromaticity by twisting the central double bond in the polyene bridge by 103° with respect to the two neighbouring double bonds. The dihedral angles in the Möbius system, however, still differ 12° and 35° from the ideal *s-cis* or *s-trans* conformation, still allowing considerable conjugation.

Aromaticity is a multidimensional property²⁵, and arguments exclusively based on geometric parameters may be misleading. For example, the maximum bond-length difference between single and double bonds in our C_2 Möbius compound (0.095 Å) is still quite large, comparable to the value for the non-aromatic anti-bismethano-[14]annulene²⁶. We therefore also investigated a second, independent, aromaticity measure based on energy considerations, by using the ‘indene–isoindene method’ (ref. 27) to estimate the stabilization caused by conjugation in the Möbius structure. At the B3LYP/6-31G* level of DFT we computed a stabilization energy due to conjugation, ISE_{II} , of $4.04 \text{ kcal mol}^{-1}$ for the C_2 Möbius compound (20% of the ISE_{II} of benzene) and an antiaromatic destabilization energy of $-2.22 \text{ kcal mol}^{-1}$ for the Hückel C_s structure. Both values are uncorrected, and can be expected to be more positive if *syn-anti* corrections are included²⁸. We also find that the strain energy of the Möbius compound is slightly higher than that of the Hückel isomer, clearly indicating that it is the more efficient conjugation that makes the Möbius compound more stable than the Hückel compound.

Taken together, the observed trends in bond-length equalization and stabilization energy indicate that the Möbius structure is moderately aromatic, whereas the Hückel structure is non-

aromatic. In the C_2 Möbius structure, all C–C bonds are conjugated, yet the molecule has only one π surface. The properties investigated here finally confirm Heilbronner’s prediction for Möbius twisted $[4n]$ annulenes. □

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Authors’ contributions R.H. together with D.A. conceived the experiment; D.A. carried it out; R.H. wrote the Letter; and A.S. and O.O. determined the X-ray structure of the Möbius C_2 compound.

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