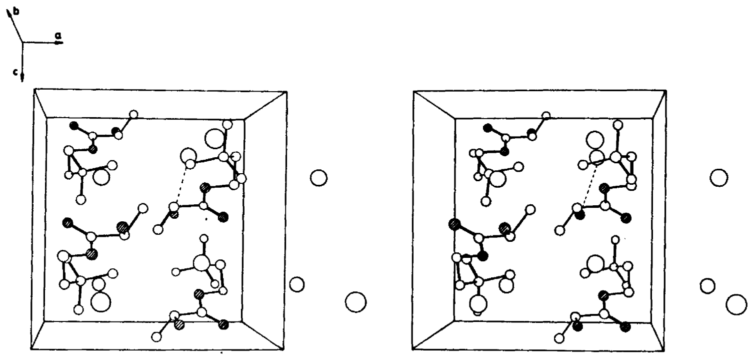

Fig. 2. A stereo view of the packing of lactoylcholine iodide. The possible hydrogen bond O12..$I^-_{(1-x,y-\frac{1}{2},\frac{1}{2}-z)}$ is indicated by a broken line. Oxygen atoms are shaded.

squares techniques to a final R value of 0.05, using the X-RAY-system.[5] All hydrogen atoms bonded to carbon atoms were found in a difference fourier map at the calculated positions (distance C–H equal to 1.0 Å) and were introduced in the final refinement as a fixed contribution ($U = 6.3$). Also the position of the hydroxyl hydrogen atom H12 was suggested in the difference fourier map, and the positional parameters of this atom have been refined. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\{1 + [(F_o - B)/A]^2\}$, $B = 25.0$ and $A = 20.0$. The X-ray atomic scattering factors used for hydrogen were those of Stewart, Davidson and Simpson[6] and for all other atoms those of Cromer and Mann.[7] All atoms but I^- were treated as uncharged. The final list of structure factors is available on request.

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Propellicene or Bi-2,13-pentahelicenylene

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Aromatic compounds having structures with interesting topologies, *e.g.* the helicenes,[1,2] cyclophanes,[3-5] and circulenes[6-9] have long fascinated the synthetic chemists. We now wish to report the synthesis of a new hydrocarbon, $C_{44}H_{24}$, which has the geometry of a two-bladed propeller. The compound, which we would like to call propellicene or bi-2,13-pentahelicenylene[12] is built from two pentahelicene units, with the terminal rings linked together at C_2 and C_{13} by single bonds (Fig. 1).

We recently described a procedure, using the Wittig reaction, whereby various [2.2.2.2]-cyclophanes and related compounds can be prepared.[10,11] Such compounds may serve as precursors for topologically interesting compounds like those mentioned above.

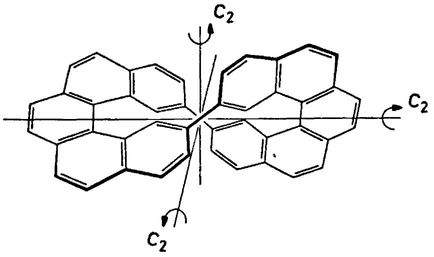
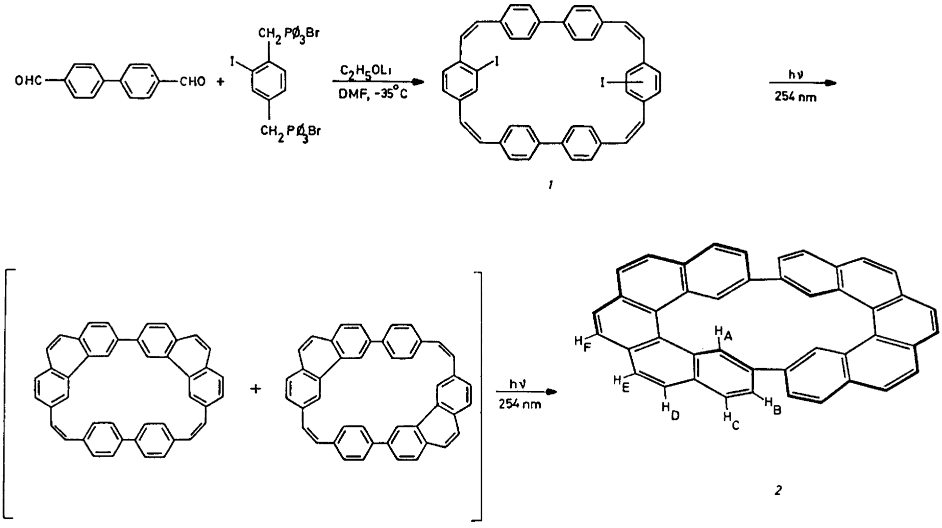


Fig. 1. Proposed structure of propellicene or bi-2,13-pentahelicenylene with the three symmetry axes.



Scheme 1.

One example of the versatility of this procedure is the two-step synthesis of the title compound from relatively simple starting materials. Thus di-iodo[2.2.0.2.2.0]paracyclophanetetraene[13] was obtained as a mixture of two isomers in 4.5 % yield from 4,4′-biphenyldicarbaldehyde and the bistriphenylphosphonium salt from 1,4-bis-(bromomethyl)-2-iodobenzene. When irradiated with light from a low pressure mercury lamp, the isomeric compound mixture, *1*, gave 70 % yield of propellicene, *2* (Scheme 1).

Its structure follows from NMR, MS and UV data. The ^{1}H NMR spectrum in deuteriochloroform is of first order at 270 MHz showing an AMX-pattern and singlets; H_A at δ 8.56 (1), H_B 7.12 (1), H_C 7.80 (1), $J_{AB} = 1.5$ Hz, $J_{AC} < 0.5$ Hz, $J_{BC} = 8$ Hz, H_D and H_E 7.91 (2) broad singlet and H_F 7.96 (1). The corresponding chemical shifts for the protons in pentahelicene are: H_A 8.47, H_B 7.47, H_C 7.89, H_D and H_E 7.83 and H_F 7.79.[14] The shift difference for H_B is clearly caused by the proximity of the other pentahelicene unit in propellicene.

The mass spectrum of propellicene is very simple (Fig. 2). The molecular ion is the base peak. Fragmentations are due to the loss of hydrogens and splitting of the molecule in half. Doubly charged and triply charged ions are frequent. The doubly charged molecular

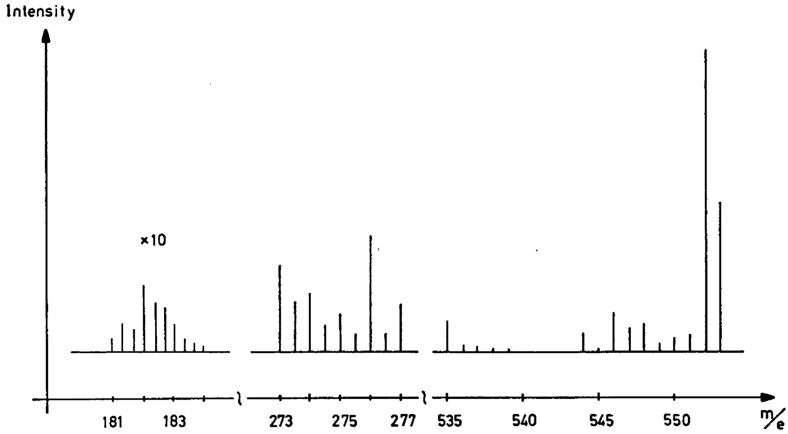


Fig. 2. Mass spectrum of propellicene or bi-2,13-pentahelicenylene.

ion and the singly charged ion with the same mass to charge ratio can be distinguished by their isotopic pattern.

The UV spectrum of propellicene in cyclohexane shows two distinct maxima at 241 nm (log $\varepsilon = 5.11$) and 284 nm (4.85) and shoulders at 225, 233, 305, 315, and 340 nm. Pentahelicene absorbs at somewhat shorter wavelengths [15] which indicates that there is some change in molecular geometry between the two compounds or possibly that there is some interaction between the helicene units in propellicene.

Propellicene has three perpendicular C_2-axes of symmetry and thus belongs to the same symmetry point group, D_2, as a two-bladed propeller (Fig. 1). The molecule is chiral and the two enantiomers should be stable and separable. The barrier for interconversion must be higher than that of pentahelicene.[1] Further work on the stereochemistry of propellicene and on the interaction between the molecular halves is under way.

Experimental. NMR spectra were recorded on a Bruker WH 270, mass spectra on an AEI MS 902, UV spectra on a Beckman DK 2 and IR spectra on a Beckman IR 9 instrument. Photochemical experiments were run in a Rayonet reactor model RPR 100.

Wittig reaction. 4,4′-Biphenyldicarbaldehyde [11] (0.01 mol) and the bistriphenylphosphonium salt from 1,4-bis-(bromomethyl)-2-iodo benzene [16] (0.01 mol) were suspended in dry dimethylformamide (250 ml) in a three-necked flask, equipped with a mechanical stirrer and a dropping funnel. The flask was kept at −35 °C in a thermostated cooling bath and flushed with nitrogen. A solution of lithium ethoxide in ethanol was added dropwise to allow for consumption of the red ylid between successive additions. The addition was stopped when no formation of an ylid could be detected, usually after 24 h. The yellow reaction mixture was diluted with an equal amount of water and extracted with diethyl ether three times. The ethereal solution was washed with water several times, dried over sodium sulfate and the solvent evaporated. The residue was chromatographed on silica gel with carbon tetrachloride as eluent. The first yellowish fraction contained di-iodo[2.2.0.2.2.0]paracyclophanetetraene, *1*, as a mixture of isomers which was recrystallised from carbon tetrachloride to give 0.18 g, 4.5 % of a yellow solid melting at 180−85 °C. ^{1}H NMR: δ 7.84 (1, broad singlet, protons adjacent to iodine), 7.46−7.07 (10, complex multiplet, aromatic protons) and 6.65−48 (4, complex multiplet, olefinic protons). MS: *m/e* 812 (100 %), 686 (20), 685 (20), 684 (48), 555 (20), 554 (40), 553 (28), 552 (44), 407 (12), 406.5 (8), 406 (12), 277 (20), 276 (20), 275 (12), 274 (12). Abs. mass; found 812.044 ± 0.003; calc for $C_{14}H_{30}I_2$ 812.046.

Propellicene. A water-cooled solution of cyclophane *1* (100 mg) in benzene (150 ml) was irradiated in a quartz cell with light from a low pressure mercury lamp (254 nm) for 3 h. The solvent was distilled off, and the residue was chromatographed on silica gel with carbon tetrachloride as eluent. A white crystalline compound identified as propellicene *2* (48 mg, 70 %) was isolated. Abs. mass; found 552.190 ± 0.003; calc. for $C_{44}H_{24}$ is 552.188.

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