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Synthesis of Some Bicyclophanes

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Bicyclic aromatic compounds, bicyclophanehexaenes of the general structure A, have been obtained from sixfold Witting reactions between aromatic bisphosphonium salts and 1,3,5-benzenetricarbaldehyde. Hydrogenation gave bicyclophanes of structure B. The synthesis of bicyclophanes from ylids prepared from the bisphosphonium salts from 1,3- and 1,4-bis(bromomethyl)benzene, 1,3-bis(bromomethyl)-5-bromobenzene, and 2,5-bis(chloromethyl)thiophene is described and the structures of the bicyclophanes discussed.

Cage compounds having a framework large enough to define a sizeable cavity in the centre of the molecule have been found to possess unusual properties, for example as host molecules in host–guest complexes.[1,2] Whether such a cavity would be stable or collapse in the absence of guest molecules should depend on the rigidity of the bonding framework. Studies of rigid cage compounds might provide information on the factors governing formation and stability of cavities in molecules.

Molecules containing aromatic rings linked by double bonds are conformationally restricted. We have recently prepared a number of [2$_4$]cyclophanetetraenes.[3,4]

The macrocyclic ring in [2$_4$]paracyclophanetetraene, *7A*, is relatively planar and the rotation of the benzene rings around the single bonds in *7A* and some closely related compounds is rapid.[5] On hydrogenation of the olefinic bonds, the cyclophanes show increased flexibility and conformational isomerism occurs, arising from *gauche* and sometimes *anti* orientations of the substitutents at the ethane bridges.[6] In line with these observations, cage cyclophanes or bicyclophanes with bridging double bonds might be compounds possessing stable cavities.

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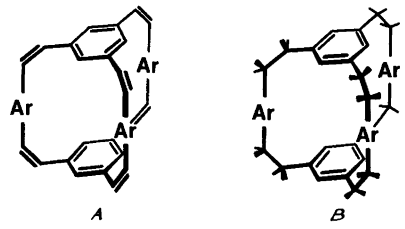
The [2$_4$]cyclophanetetraenes mentioned above can be synthesized by fourfold Witting reactions between aromatic dialdehydes and biphosphonium salts.[3,4] The conditions have been optimized to yield the highest possible *cis/trans* ratio.[3,4]

RESULTS AND DISCUSSION

We found that a mixture of 1,3,5-benzenetricarbaldehyde (2 equiv. and a biphosphonium salt from a bis(bromomethyl)arene (3 equiv.) in dry dimethylformamide at −40 °C on treatment with lithium ethoxide gave a crude product which, on chromatography followed by sublimation, afforded a bicyclophane* with unsaturated bridges (*A*, 1.4–

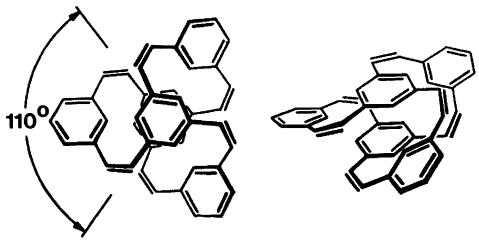


A *B*

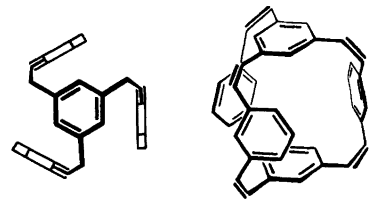
* We suggest the name bicyclophane for compounds in which two aromatic rings are joined by three bridges, all containing aromatic rings. The name should contain the types and number of bridges between the aromatic rings, followed by the types and number of aromatics, starting with the triply-bridged rings. The name should end with the suffix -bicyclophane. This suggestion does not lead to an unambiguous naming of bicyclophanes, for which the IUPAC nomenclature must be used, but is intended to simplify the naming of complex multicyclic compounds of this type. According to this suggestion, the compounds *1A*, *2A* and *3A* should be named [2$_6$](1,3,5)$_2$(1,3$_3$-bicyclophanehexaene, [2$_6$](1,3,5)$_2$(1,4)$_3$bicyclophanehexaene and [2$_6$](1,3,5)$_2$(2,5-thiopheno)$_3$bicyclophanehexaene, respectively. The IUPAC name of *2A* is heptacyclo[12.12.8^{1,14}.2^{6,9}.2^{19,21}.2^{29,32}.1^{3,25}.1^{12,16}]dotetraconta-1,3(41),4,6,8,10,12(42),13,15,17,19,21,23,25,27,29,31,33,35,37,39-heneicosaene.

2 % yield).[7] Although the yields were low, the simplicity of this one-step synthesis makes it an easy route to this type of compound. Catalytic hydrogenation of the "unsaturated" bicyclophanes furnished the "saturated" bicyclophanes of type *B* in quantitative yield. The bisphosphonium salts from 1,3- and 1,4-bis(bromomethyl)benzene, 1,3-bis(bromomethyl)-5-bromobenzene, and 2,5-bis(chloromethyl)thiophene gave, together with 1,3,5-benzenetricarbaldehyde, the bicyclophanes *1A*, *2A*, *4A* and *3A*, respectively. Hydrogenation of the bicyclophanes *1A*, *2A* and *3A* afforded the bicyclophanes *1B*, *2B* and *3B*, respectively.

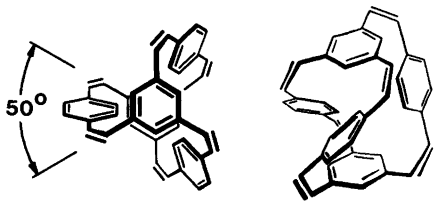
The mass spectra of the bicyclophanes of type *A* confirmed their proposed gross structures, showing base peaks at *m/e* values of the expected molecular



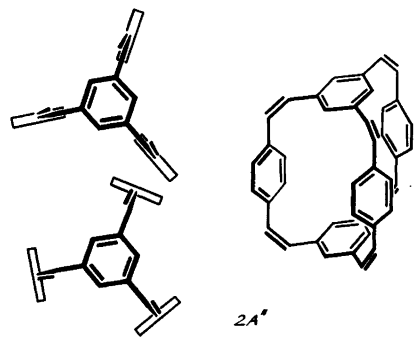
1A'



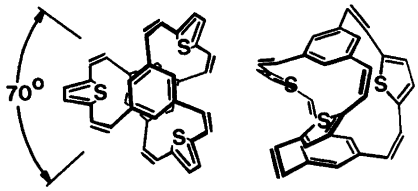
1A''



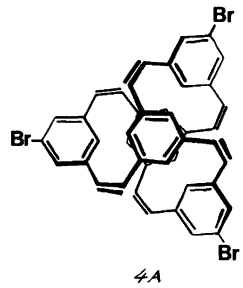
2A'



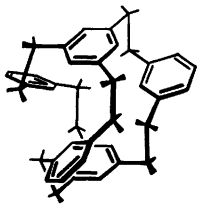
2A''



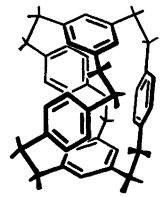
3A



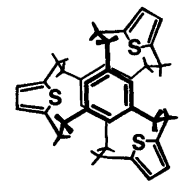
4A



1B



2B



3B

weights. Doubly-charged molecular ions were the second most abundant in these spectra, while almost no fragmentation was observed. The bicyclophanes of type *B* gave rise to molecular ion base peaks at *m/e* twelve units higher than those of type *A* and fragmentation from cleavage of the sp^3-sp^3 C–C bonds.

Inspection of molecular models (CPK and Dreiding) revealed that the "unsaturated" bicyclophanes (type *A*) are conformationally mobile to a certain extent and able to adopt conformations ranging between two extremes. In one of these, the distance between the two trisubstituted rings is as short as possible and a maximum is reached for the twist angle (*i.e.* the angle between the start and end of a bridge when viewing the molecule from a point on the threefold axis of symmetry). A large twist angle leads to a minimal cavity in the centre of the molecule (see *1A'*, *2A'* and *3A*). In the other extreme conformation, the twist angle is zero and the distance between the trisubstituted rings as well as the size of the cavity in the centre are as large as possible (see *1A"* and *2A"*).

The *m*-phenylene bridged bicyclophane *1A* thus has two extreme conformations *1A'* and *1A"* (two projections are shown for each of these with twist angles of 110 and 0°, respectively).

The conformation *1A"* with D_3 symmetry is chiral and the models show that the enantiomers can interconvert *via* *1A"* which has C_{3h} symmetry.

In the ^{1}H NMR spectrum of the bicyclophane *1A* one peak appeared at unusually low field (δ 8.86). This peak arises from the three *m*-phenylene protons *ortho* to both the ethylene bridges (H_A in Table 1). A similar low field shift has been observed for the corresponding H_A-protons in [2$_2$]metacyclophanediene *5A* (δ 7.90),[8] which has the *anti* conformation depicted in *5A*.[9] The average value of the shifts

Table 1. Chemical shifts for the protons in bicyclophanes *1–4* and some closely related cyclophanes *5–9*.

Compound	H_A	H_B	H_C	H_D	H_E	H_F	H_G	and	$H_{G'}$	H_H	and	$H_{H'}$
1A	8.86	7.08	7.33	7.33			6.31		6.18			
1B	6.05	7.04	7.24	6.36						2.71		2.65
2A				6.98	7.08		6.65		6.42			
2B				6.67	6.51						2.81	
3A				7.10		6.81	6.55		6.28			
3B				6.66		6.46				2.83		2.65
4A	8.70	7.32		7.26				6.22				
5A	7.90	7.01	6.60					6.22				
5B	4.20	7.30	7.00									
6A	7.89	7.13	7.07					6.32				
7A					7.32			6.45				
7B					6.72						2.85	
8A					7.24	6.80	6.55		6.38			
8B					6.75	5.69				2.97		2.86
9B	5.88	6.93	7.13		6.81							2.73

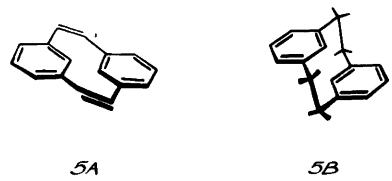
of the peaks due to the H_A and H_D protons in *1A* is $(\delta_{H_A}+2\delta_{H_D})/3 = 7.84$. Of the possible conformations of *1A* only the compressed one *1A'* has the H_A and H_D protons located in an environment similar to that of the H_A protons in the cyclophane *5A*. Because the *m*-phenylene rings in *1A'* are tilted, the H_D protons are more shielded than the H_A protons in *5A*. However, since the H_A protons in *1A'* are deshielded by two aromatic rings their resonances appear at a lower field than those of the H_A protons in the cyclophanediene *5A*. It seems clear that the chiral conformation *1A'* best represents the structure of the bicyclophane *1A*. Whether the activation energy of the interconvertion between the enantiomers is large enough to determine by NMR methods remains to be tested on a properly substituted derivative.

It is interesting to note that the resonances of the H_A protons in [2$_4$]metacyclophanetetraene (*6A*) appear at δ 7.89.[4] The favoured conformation of compound *6A* must therefore be closely related to that of the bicyclophane conformation *1A'*.

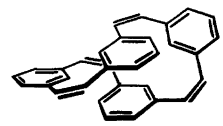
The tribromo-derivative of the *m*-phenylene bridged bicyclophane, compound *4A*, shows essentially the same NMR spectrum as *1A* and should behave similarly and prefer the conformation shown in *4A*.

Models of the *p*-phenylene bridged bicyclophane *2A* show that the compressed chiral D_3 conformation *2A'* is virtually strain-free if the bond angles in the bridging double bonds are close to 120°. However, the corresponding bond angles in crystalline [2$_4$]paracyclophanetetraene *7A* are 132°.[5] If the bond angles are of similar magnitude in the bicyclophane *2A* the conformation *2A''* with D_{3h} symmetry is favoured. The rotation of the *p*-phenylene rings should be more restricted in the compressed conformation *2A'* than in *2A''*. It may be noted that if the *p*-phenylene rings are oriented tagentially in the D_{3h} conformation *2A''*, the cavity in the centre of the molecule is large enough to accommodate small molecules such as methane or ethane.

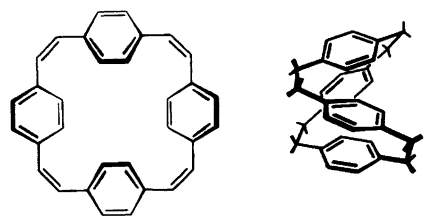
The NMR spectrum of the bicyclophane *2A* (see Table 1) does not show any anomalous shifts. The protons H_E of the *p*-phenylene rings resonate at a δ-value very similar to that of the corresponding protons in [2$_4$]paracyclophanetetraene *7A*. The chemical shifts of the aromatic protons in various conformations of the bicyclophane *2A* were calculated using the Bovey-Johnson nomogram.[10] Satisfactory agreement between calculated and



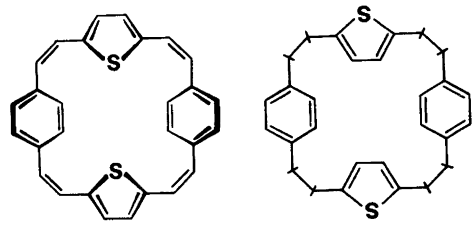
5A 5B



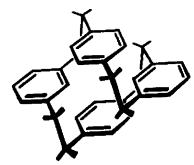
6A



7A 7B



8A 8B



9B

observed shift values was obtained for both the D_{3h} conformation *2A''* and for the twisted, compressed D_3 conformation with twist angles up to 60 – 70°. Larger angles lead to large upfield shifts of the calculated value for H_D. Thus it is not possible, from NMR data alone, to establish which of the possible conformations the bicyclophane *2A* prefers. In order to solve this problem, an X-ray structure determination is necessary.

The NMR spectrum of the thiophenylene bridged bicyclophane *3A* showed singlets for the arene protons at δ 6.80 and 7.10. The former is assigned to the thiophene protons H_F by analogy with the shifts in the cyclophane *8A*[11] and the latter to the benzene protons H_D. The shift of the H_D-protons in *3A* lies between the shifts of the same type of proton in *1A* and *2A* (see Table 1). Space filling models (CPK) show that in the compressed conformation with D_3 symmetry, which is depicted in *3A*, the sulfur atoms point towards the centre of the molecule. In the other extreme conformation, with maximum separation of the trisubstituted rings (twist angle 0°), the thiophene rings can rotate.

Since the bicyclophane *1A* most probably adopts the twisted compressed conformation *1A'*, it seems likely that the bicyclophanes *2A* and *3A* behave similarly and adopt their respective compressed conformations depicted in *2A'* and *3A*. The twist angles in the virtually strain-free conformations are approximately 110° (*1A'*), 70° (*3A*) and 50° (*2A'*) as judged from space filling (CPK) models. It is interesting to note that the observed chemical shifts for the H_D protons of these bicyclophanes are roughly linearly dependant on the estimated twist angle.

All the "unsaturated" bicyclophanes (type *A*) are extremely high-melting colourless solids and are only slightly soluble in organic solvents. These facts indicate that the bicyclophanes are rather rigid with ordered structures of high symmetry. The "saturated" bicyclophanes (type *B*) have much lower melting points, are fairly soluble, and should therefore be more flexible.

The NMR spectrum of the *m*-phenylene bridged bicyclophane *1B* showed that the resonances of the protons *ortho* to the bridges, H_A and H_D, are shifted upfield on hydrogenation of *1A*, indicating that these protons become shielded by the neighbouring aromatic rings (see Table 1). The effect is larger for H_A which is sandwiched between the trisubstituted benzene rings. A similar effect is observed in [2$_4$]-metacyclophane *5B* and [2$_4$]metaparametapara-cyclophane *9B*[4] (see Table 1). In the bicyclophane *2B* two of the *m*-phenylene rings exert deshielding effects on the nearby H_A protons of the third ring. A similar effect is operative in compound *9B*. Therefore, although the overall shielding effects in compounds *1B* and *9B* are smaller than in [2$_2$]metacyclophane, *5B*, the former two compounds most likely adopt compressed conformations closely analogous to that observed for compound *5B*.

The *p*-phenylene bridged bicyclophane *2B* should have a helix structure similar to *1B* but less compressed. The NMR spectrum shows smaller but significant upfield shifts of the aromatic protons (see Table 1), indicative of mutual shielding from the di- and trisubstituted benzene rings.

The same upfield shift of the aromatic protons is observed in the thiophene bridged bicyclophane *3B* as compared to *3A*. The effect is smaller for the thiophene protons, H_F, than for the protons in the benzene rings, H_D, which is consistent with the assumption that the sulfur atoms point towards the centre of the molecule. The shift difference for the thiophene protons in the [2$_4$](2,5)thiophenoparacyclophanes *8A* and *8B* is much larger[11] (see Table 1) and this is interpreted as being due to different orientations of the thiophene rings in the two compounds.

Several conformations are possible for the bicyclophanes *1B*, *2B* and *3B*. The orientation of the aromatic rings linked by $-CH_2-CH_2-$ bridges must be *gauche* in most cases. Conformations with *anti*-orientations at some bridges are possible in *1B* but seem to be of higher energy than the all-*gauche* conformations, due to steric interactions. Neglecting *anti*-orientations at the $-CH_2-CH_2-$ bridges, the number of different conformations due to *gauche*$^+$ or *gauche*$^-$ orientations is still sixteen (including mirror images). The interconversion of conformers should occur mainly over *syn* barriers, as the other possibility, over *anti* conformations, is less plausible. The barriers to rotation of the aromatic rings are assumed to be low.

The temperature-dependence of the ^{1}H NMR spectra of the bicyclophanes *1B* and *2B* has been investigated and reported elsewhere.[12] Both bicyclophanes show the same type of behaviour. The AA'BB'-patterns for the protons in the saturated bridges broaden on cooling the samples and reappear as ABCD-patterns. The signals for the aromatic protons in *1B* also broaden and then resharpen to the original pattern. These observations are consistent with the assumption of

rapidly interconverting symmetrical conformations (D_3-symmetry).

The analysis of the ^{1}H NMR spectra of the bicyclophanes *1*–*4*, by comparison with spectra of similar but simpler cyclophanes, has led us to the conclusion that these molecules adopt conformations in which the central cavity is minimized. Furthermore, several of the cyclophanes prefer one or only a few, often highly symmetrical, conformations out of the large number of theoretically possible ones.

EXPERIMENTAL

The *bistriphenylphosphonium* salts were prepared from 1,3-bis(bromomethyl)benzene, 1,4-bis(bromomethyl)benzene, 1,3-bis(bromomethyl)-5-bromobenzene (*cf.* below) or 2,5-bis(chloromethyl)thiophene[11] by warming with triphenylphosphine (2 mol equiv.) in DMF up to *ca.* 150 °C. The salts crystallized on cooling and were collected and dried under vacuum at 110 °C before use.

1,3,5-Benzenetricarbaldehyde was prepared *via* Rosenmund reduction of 1,3,5-benzenetricarbonyl chloride using a slightly modified procedure as compared with that previously described.[13] The acid chloride (100 g) was dissolved in dry xylene (1 l). Palladium on barium sulphate catalyst (5 %, 25 g) and finely powdered thiourea (500 mg) were added. The mixture was stirred under nitrogen using a high speed stirrer. A stream of hydrogen was passed directly into the reaction mixture which was then heated to reflux temperature whereupon evolution of hydrogen chloride began. Addition of hydrogen, heating and stirring were continued until no further hydrogen chloride was evolved (approx. 10 h). On cooling, crystals were slowly formed. These and the catalyst were filtered off (filtrate: A) and boiled with water (3.5 l) until all the volatile components had steam distilled. After hot filtration the solution was cooled. The trialdehyde crystallized as long colourless needles, which were collected by filtration (filtrate: B), to give 27 g, m.p. 152 °C (Lit[13] 152 °C). Filtrate A was evaporated to 75 ml volume. The solid formed was collected by filtration (filtrate: C) and dissolved in the hot filtrate B which was then evaporated to approx. 1 l volume. On cooling, an additional 9 g of the trialdehyde was obtained, m.p. 150–151 °C (total yield 59 %). Filtrate C on evaporation gave 1,3-benzenedicarbaldehyde (9.5 g, 19 %).

Synthesis of bicyclophanehexaenes. 1,3,5-Benzenetricarbaldehyde (8.1 g, 0.05 mol) and a bisphosphonium salt from a bis(halomethyl)arene (0.075 mol) were added to dry DMF (250 ml) and the mixture was stirred under nitrogen at −40 °C. A freshly prepared solution of lithium ethoxide (approx. 0.3 M) was added dropwise at such a rate that the coloured ylid was consumed before the next drop was added. When no further colour was observed on addition of the base (after several hours) the reaction mixture was poured into water (*ca.* 250 ml) and filtered. The solid obtained was triturated with hot ethanol which dissolved the triphenylphosphineoxide formed. The aqueous phase was extracted with dichloromethane which was then washed with water, dried and the solvent evaporated. The residue together with the solid above was chromatographed on silica gel (tetrachloromethane as eluent except for the trithiabicyclophane, *A*, where dichloromethane was used). The bicyclophanehexaenes were eluted first. On concentration of the eluate to a small volume (*ca.* 10 ml) the cage cyclophanes crystallized. Filtration furnished essentially pure bicyclophanehexaenes, 1.5–2 %, which could be further purified by gradient vacuum sublimation.

[2$_6$](1,3,5)$_2$(1,3)$_3$Bicyclophanehexaene, 1A. Prisms (xylene), m.p. >360 °C. MS (70 eV): *m/e* 534 (M^+, 100 %), 277 (10, M^{2+}), 91 (2). Mol. wt., obs. 534.237; calc. for $C_{22}H_{30}$ 534.235. UV ($CHCl_3$): 297 nm (ε 83500), 265 sh (48700).

[2$_6$](1,3,5)$_2$(1,4)$_3$Bicyclophanehexaene, 2A. Prism (subl. or recryst. from quinoline), m.p. >550 °C. Anal. $C_{42}H_{30}$:C,H. MS (70 eV): *m/e* 534 (M^+, 100 %), 277 (M^{2+}, 6), 115 (4), and 91 (4). Mol. wt., obs. 534.233; calc. for $C_{42}H_{30}$ 534.235. UV ($CHCl_3$): 288 nm (ε 32100) and 260 sh (36000).

[2$_6$](1,3,5)$_2$(2,5-Thiopheno)$_3$bicyclophanehexaene, 3A. M.p. >360 °C from sublimation. MS (36 eV): *m/e* 552 (M^+, 100 %). Mol. wt. obs. 552.109; calc. for $C_{36}H_{24}S_3$ 552.108. UV ($CHCl_3$): 272 nm (ε 36300), 300 sh (19000), and 343 (15500).

[2$_6$](1,3,5)$_2$(5-Bromo,1,3)$_3$bicyclophanehexaene, 4A, was prepared in a small amount from 1,3,5-benzenetricarbaldehyde and the bistriphenylphosphonium salt from 1,3-bis(bromomethyl)-5-bromobenzene (see below) and its ^{1}H NMR spectrum recorded. (see Table 1).

1-Bromo-3,5-dimethylbenzene was prepared from diazotized 3,5-dimethylaniline. B.p. 80–82 °C/10 mmHg (Lit.[14] 88–89 °C/12 mmHg). ^{1}H NMR (60 MHz, $CDCl_3$): δ 2.18 (6H, broad s), 6.75 (1H, m), 7.00 (2H,m).

1,3-Bis(bromomethyl)-5-bromobenzene. 1-Bromo-3,5-dimethylbenzene (13,1 g), *N*-bromosuccinimide (recrystallized from acetic acid, 27.4 g) and benzoyl peroxide (700 mg) were reflexed in analytical grade tetrachloromethane (150 ml) under nitrogen. When all the bromosuccinimide was consumed the reaction mixture was filtered. The filtrate was evaporated to give an oil. On addition of ligroin, crystals separated which were recrystallized from ethanol, 5.7 g (22 %), m.p. 97–99 °C. Anal. $C_8H_7Br_3$: C, H.

^{1}H NMR (60 MHz, $CDCl_3$); δ 4.41 (4 H, s), 7.30 (1H, broad t, J 1.5 Hz), 7,47 (2H, d, J 1.5 Hz).

Synthesis of the bicyclophanes with saturated bridges, B. A small amount of a bicyclophanehexaene (*ca.* 10 mg) was mixed with palladium on charcoal (10 %, 5 mg) in benzene and stirred under hydrogen at atmospheric pressure until all the starting material had disappeared (usually 24 h). The mixture was filtered and the solvent evaporated to give a quantitative yield of the bicyclophane.

[2$_6$](1,3,5)$_3$Bicyclophane, 1B. M.p. 205–206 °C from acetic acid. MS (36 eV): *m/e* 546 (M^+, 100 %), 441 (16), 427 (11), 233 (16), 231 (13), 223 (10), 221 (13), 220 (11), 219 (44), 218 (10), 217 (14), 207 (21), 206 (16), 205 (48), 204 (10) and 203 (12), only fragments with *m/e* > 200 are listed. Mol. wt., obs. 546.323; calc. for $C_{42}H_{42}$ 546.329.

[2$_6$](1,3,5)$_2$(1,4)$_3$Bicyclophane, 2B. M.p. 207–208 °C, from acetic acid. MS (70 eV): *m/e* 546 (M^+, 100 %), 455 (8), 441 (7), 233 (13), 221 (11), 219 (22), 207 (12), 205 (18), 131 (9), 119 (21), 117 (18), 105 (28), 91 (13) and metastables for *m/e* 546→455 and 546→441. Mol. wt., obs. 546.328; calc. for $C_{42}H_{42}$ 546.329.

Calculation of the chemical shifts of the H_E and H_D protons of [2$_6$](1,3,5)$_2$(1,4)$_3$bicyclophanehexaene, 2A. The Bovey-Johnson nomogram[10] was used to calculate the expected chemical shifts of the protons on the aromatic rings at various twist angles. The influence of the bridging double bonds was considered to be small and was neglected. The distances from one H to the neighbouring aromatic rings were measured using Dreiding models. In case of the D_{3h} conformation (twist angle 0°) the mean values for δ_{H_E} and δ_{H_D} were used as calculated from the two extreme conformations depicted in *2A''*. δ_{H_E} is 7.2 for *p*-divinylbenzene and δH_D is 7.3 for *m*-divinylbenzene.[15] These were taken as standard values for unperturbed shifts. δ_{H_E} was then calculated to be: δ_{H_E} (twist angle): 7.05(0°), 6.95(50°), 6.85(70°) and 6.65(90°); δ_{H_D} (twist angle): 7.30(0°), 7.25(50°), 7.05(70°) and 6.38(90°).

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REFERENCES

1. Cram, J. C. and Cram, J. M. *Acc. Chem. Res. 11* (1978) 8.
2. Lehn, J.-M. *Acc. Chem. Res. 11* (1978) 49.
3. Thulin, B., Wennerström, O., Somfai, I. and Chmielarz, B. *Acta Chem. Scand. 31* (1977) 135.
4. Thulin, B., Wennerström, O. and Somfai, I. *Acta Chem. Scand. B 32* (1978) 109.
5. Ljungström, E., Lindquist, O. and Wennerström, O. *Acta Crystallogr. B 34* (1978) 1889.
6. Olsson, T., Tanner, D., Thulin, B., Wennerström, O. and Liljefors T. *Tetrahedron 37* (1981) 3473.
7. Högberg, H.-E., Thulin, B. and Wennerström, O. *Tetrahedron Lett.* (1977) 931.
8. Mitchell, R. H. and Boekelheide, V. *J. Am. Chem. Soc. 96* (1970) 1547.
9. Hanson, A. W. and Röhrl, M. *Acta Crystallogr. B 28* (1972) 2032.
10. Johnson, C. E. and Bovey, F. A. *J. Chem. Phys. 29* (1958) 1012.
11. Strand, A., Thulin, B. and Wennerström, O. *Acta Chem. Scand. B 31* (1977) 521; Tanner, D. and Wennerström, O. *Unpublished results.*
12. Olsson, T., Tanner, D., Thulin, B. and Wennerström, O. *Tetrahedron 37* (1981) 3485.
13. Ried, W. and Königstein, F.-J. *Chem. Ber. 92* (1959) 2532.
14. Fieser, L. F. and Heyman, J. *J. Am. Chem. Soc. 64* (1942) 376.
15. Wiley, R. H. and Grawford, T. H. *J. Polym. Sci. Part B 3* (1965) 99.

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