with 4 mol of tetrachloropyrazine[5] (50% and 32%, respectively). The low solubility of **4** led us to **5**, whose dimerization was most conveniently studied. Vapor pressure osmometry showed that **5** at 27 °C in $CHCl_3$ (7.8–31 mM concentrations) gave molecular weights of 2665 ± 270, closer to that of dimer (2808) than of monomer (1404). In $CDCl_3$ at −18 °C, the 360-MHz ^1H NMR spectrum of monomer **5** gave δ 2.19 for the two up-methyls and δ 2.43 for the two out-methyls, whereas these respective signals moved to δ 1.69 and 2.63 in dimer. Irradiation of the δ 2.63 peak (500 MHz at −18 °C) produced −3% enhancements of the δ 1.69 peak. Irradiation of the δ 1.69 peak was unfruitful because it was partially obscured by water peaks. Control irradiations of aryl protons gave −3 to −10% enhancements.[6] Negative enhancements are frequently encountered in large molecules.[7] Intermolecular distances between up-methyls and out-methyls in CPK models of **5·5** are <5 Å. We concluded that **5·5**[2a] is structured much like **1·1**, whose crystal structure is known.[2b] Similar differences in ^1H NMR chemical shifts for **1** and **1·1** were observed.

Association constants (K_a, M^{-1}) and $-\Delta G^\circ$ values for **5** + **5** $\rightleftarrows$ **5·5** were measured by employing 360-MHz ^1H NMR spectral differences between monomer and dimer. At −46 °C, $-\Delta G^\circ$ (kcal mol^{-1}) values varied with solvent as follows: $CD_3C_6D_5$, 3.5; $CDCl_3$, 4.1; CD_2Cl_2, 4.3. At −18 °C, they varied as follows (solvent %, v/v): 100% $CDCl_3$, 4.1; 75% $CDCl_3$ −25% $(CD_3)_2CO$, 5.1; 75% $CDCl_3$ −25% CD_3NO_2, 5.3; 75% $CDCl_3$ −25% CD_3OD, >5.6 kcal mol^{-1}. Values of k_a (M^{-1}) for **5** dimerization in $CDCl_3$ changed with temperature (K) as follows: 2300 (263); 3070 (253); 4520 (241); 5270 (237); 7090 (227). The least-squares line ($r > 0.99$) of a van't Hoff plot provided $\Delta H^\circ = -3.8 \pm 0.5$ kcal mol^{-1} and $\Delta S^\circ = 1.1 \pm 3$ eu. Similar plots from data obtained in 90% $CDCl_3$ −10% $(CD_3)_2CO$ (v/v) and 90% $CDCl_3$ −10% CD_3OD (v/v) gave, respectively: ΔH°, −4.0 ± 0.5 and −3.7 ± 0.5 kcal mol^{-1}; ΔS° = 2 ± 3 eu and 3 ± 3 eu. Typical entropy changes for host–guest complexation in organic solvents usually range from −11 to −15 eu.[8] Molecular model examination of **5** and **1** suggests that each face can be solvated by up to 9 mol of $CDCl_3$, which is released to solvent upon dimerization. Decollection of many solvent molecules probably pays the entropic cost of collecting and rigidifying two monomers during dimer formation. Thus the full $-\Delta H^\circ$ values are felt in the $-\Delta G^\circ$ binding values.

The $ArCH_3$ signals at 2.62 ppm (**5·5**) and 2.48 ppm (**5**) coalesced at 12 ± 5 °C, providing a ΔG^* for dimer dissociation of 14.0 ± 0.3 kcal mol^{-1} and $k_{-1} = 106\ s^{-1}$, which coupled with K_a gives $k_1 = 1.4 \times 10^5\ M^{-1}\ s^{-1}$. We attribute the remarkably slow dissociation to the absence of incremental solvation–desolvation of faces involved in dimerization. The locking of four CH_3 groups into four cavities in the dimer[2b] inhibits monomer-to-monomer slippage. Insertion of one solvent molecule between the rigid dimer faces (clamlike opening) largely dissipates attractive forces.

Similar measurements applied to **1** + **1** $\rightleftarrows$ **1·1** at 500 MHz in $CDCl_3$ provided these values at 12 °C: $K_a = 87\,000 \pm 30\%\ M^{-1}$; $-\Delta G^\circ$, 6.6 ± 0.2 kcal mol^{-1}. Similar experiments applied to mixtures of **1** and **5** at −18 °C (500 MHz) in $CDCl_3$ provided the following: for **1** + **1** $\rightleftarrows$ **1·1**, $K_a = 650\,000 \pm 30\%\ M^{-1}$ and $-\Delta G^\circ = 6.9 \pm 0.2$ kcal mol^{-1}; for **1** + **5** $\rightleftarrows$ **1·5**, $K_a = 263\,000 \pm 30\%\ M^{-1}$ and $-\Delta G^\circ = 6.3 \pm 0.2$ kcal mol^{-1}; for **5** + **5** $\rightleftarrows$ **5·5**, $K_a = 3100 \pm 30\%\ M^{-1}$ and $-\Delta G^\circ = 4.1 \pm 0.2$ kcal mol^{-1}. In mixtures of **2** with either **1** or **5**, no **2·1** or **2·5** formation was observed.[9] Extrapolations of our $-\Delta G^\circ$ values using Diederich's[10] correlations ($-\Delta G^\circ$ changes for a cyclophane host binding pyrene with solvent changes) suggest that, in pure CH_3OH, $-\Delta G^\circ$ values for **1·1** formation could be ≈11 kcal mol^{-1} and, in H_2O ≈14 kcal mol^{-1}.

Our study, coupled with others (e.g., Diederich's,[8d] Still's,[11] and Whitlock's[12]), indicates that, given appropriate preorganization, complementarity, and binding surface sizes of host–guest systems, high and variable binding free energies are observed in organic solvents, with solvaphobic driving forces playing highly significant roles.

---

(3) New compounds' elemental analyses were within 0.3% of theory; ^1H NMR spectra were as expected; mass spectra contained substantial M^+ or $M + H^+$ peaks.

(4) Tunstad, L. A.; Tucker, J. A.; Dalcanale, E.; Weiser, J.; Bryant, J. A.; Sherman, J. C.; Helgeson, R. C.; Knobler, C. B.; Cram, D. J. *J. Org. Chem.* **1989**, *54*, 1305–1312.

(5) Allison, C. G.; Chambers, R. D.; MacBride, A. A. H.; Musgrave, W. K. R. *J. Chem. Soc. C* **1970**, 1023–1024.

(6) We thank Michael Geckle for help with NOE experiments.

(7) Derome, A. E. *Modern NMR Techniques for Chemical Research*; Pergamon Press: Oxford, 1987.

(8) (a) Cram, D. J.; Stewart, K. D.; Goldberg, I.; Trueblood, K. N. *J. Am. Chem. Soc.* **1985**, *107*, 2574–2575. (b) Tucker, J. A.; Knobler, C. B.; Trueblood, K. N.; Cram, D. J. *Ibid.* **1989**, *111*, 3688–3699. (c) Conceill, J.; Lacombe, L.; Collet, A. *Ibid.* **1986**, *108*, 4230–4232. (d) Diederich, F. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 362–386.

(9) We estimate that $-\Delta G^\circ$ for dimerizations involving **1** as low as ~2.2 kcal mol^{-1} could have been detected, which provides our best model for $-\Delta G^\circ$ values that *might have been* detected for **2·2** or **3·3**, should they exist at all.

(10) Smithrud, D.; Diederich, F. *J. Am. Chem. Soc.*, in press.

(11) Chapman, K. T.; Still, C. W. *J. Am. Chem. Soc.* **1989**, *111*, 3075–3079.

(12) Sheridan, R. E.; Whitlock, H. W., Jr. *J. Am. Chem. Soc.* **1986**, *108*, 7120–7121.

Spontaneous Assembly of a Double-Helical Binuclear Complex of 2,2′:6′,2″:6″,2‴:6‴,2⁗:6⁗,2′′′′′-Sexipyridine

Edwin C. Constable* and Michael D. Ward

University Chemical Laboratory
Lensfield Road, Cambridge CB2 1EW, United Kingdom

Derek A. Tocher

Department of Chemistry, University College London
20 Gordon Street, London WC1H OAJ, United Kingdom

Received July 26, 1989

Double-helical molecules have attracted interest since DNA was demonstrated to adopt this molecular topology.[1] Until recently, this geometry was rare in inorganic coordination compounds,[2] although we[3] and others[4] have demonstrated the spontaneous assembly of double-helical complexes containing polypyridine ligands. We have probed the structural requirements for the formation of double-helical complexes and have demonstrated that it arises when a conjugated polydentate ligand interacts with a metal ion that is too small for the bonding cavity occurring in a *planar* ligand configuration. We have also shown that π-stacking interactions play a crucial role in dictating the stability of the double-helical geometry.

---

(1) Watson, J. D.; Crick, F. H. C. *Nature (London)* **1953**, *171*, 737. Pauling, L.; Corey, R. B.; Branson, H. R. *Proc. Natl. Acad. Sci. U.S.A.* **1951**, *37*, 205. Florey, P. J.; Miller, W. G. *J. Mol. Biol.* **1966**, *15*, 284. Brewster, J. H. *Top. Curr. Chem.* **1974**, *47*, 29. Mislow, K.; Gust, D.; Finnocchiaro, P.; Böttcher, R. J. *Top. Curr. Chem.* **1974**, *47*, 1. Meurer, K. P.; Vögtle, F. *Top. Curr. Chem.* **1985**, *127*, 1.

(2) Stoddart, F. *Nature (London)* **1988**, *334*, 10. Libman, J.; Tor, Y.; Stanzer, A. *J. Am. Chem. Soc.* **1987**, *109*, 5880. Stuckmeier, G.; Thewalt, U.; Furhop, J.-H. *J. Am. Chem. Soc.* **1976**, *98*, 278.

(3) Constable, E. C.; Drew, M. G. B.; Ward, M. D. *J. Chem. Soc., Chem. Commun.* **1987**, 1600. Constable, E. C.; Holmes, J. M. *Inorg. Chim. Acta* **1987**, *126*, 187. Barley, M.; Constable, E. C.; Corr, S. A.; Drew, M. G. B.; McQueen, R. C. S.; Nutkins, J. C.; Ward, M. D. *J. Chem. Soc., Dalton Trans.* **1988**, 2655. Constable, E. C.; Drew, M. G. B.; Forsyth, G.; Ward, M. D. *J. Chem. Soc., Chem. Sommun.* **1988**, 1450. Constable, E. C.; Drew, M. G. B.; Forsyth, G.; Ward, M. D. *Polyhedron*, in press. Constable, E. C.; Holmes, J. M.; Raithby, P. R. *J. Chem. Soc., Dalton Trans.*, submitted.

(4) Lehn, J.-M.; Rigault, A. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 1095. Lehn, J.-M. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 89. Lehn, J.-M.; Rigault, A.; Siegel, J.; Harrowfield, J.; Chevrier, B.; Moras, D. *Proc. Natl. Acad. Sci. U.S.A.* **1987**, *84*, 2565. Lehn, J.-M.; Sauvage, J.-P.; Simon, J.; Ziessel, R.; Piccinni-Leopardi, C.; Germain, G.; Declercq, J.-P.; VanMeerssche, M. *Nouv. J. Chim.* **1983**, *7*, 413. Gisselbrecht, J.-P.; Gross, M.; Lehn, J.-M.; Sauvage, J.-P.; Ziessel, R.; Piccinni-Leopardi, C.; Arrieta, J. M.; Germain, G.; Van Meerssche, M. *Nouv. J. Chim.* **1984**, *8*, 661.

0002-7863/90/1512-1256$02.50/0

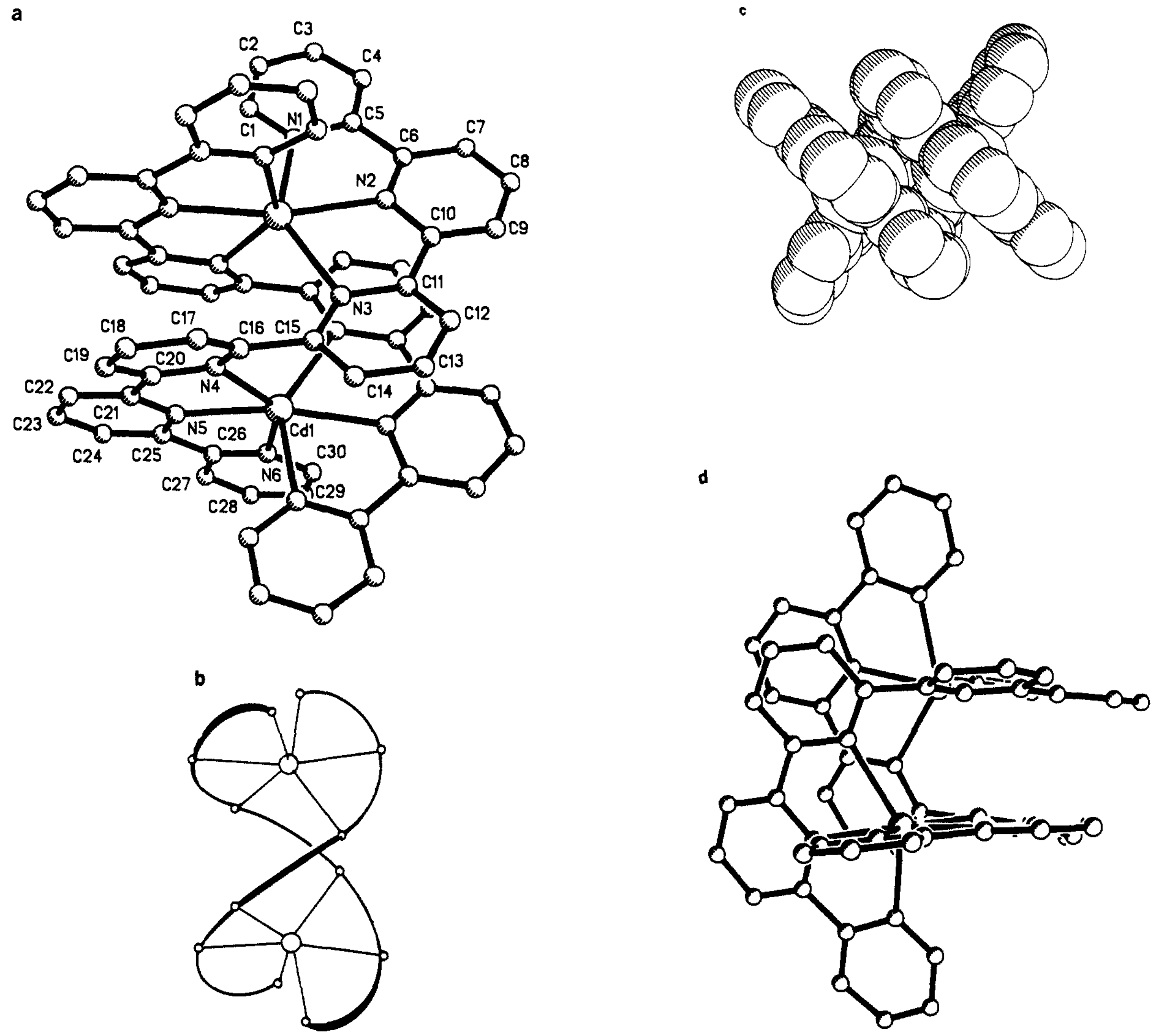
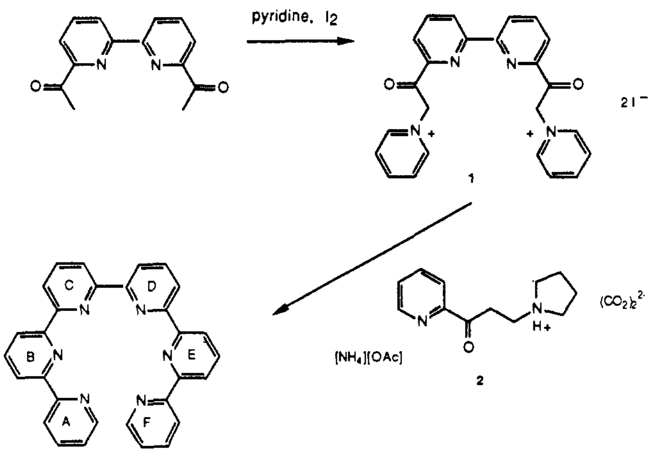


Figure 1. (a) A view of one of the two enantiomeric $[Cd_2L_2]^{4+}$ cations in $[Cd_2L_2][PF_6]_4{\cdot}4MeCN$: Cd(1)-N(4), 2.376 (8) Å; Cd(1)-N(6), 2.355 (9) Å; Cd(1)-N(2A), 2.301 (6) Å; Cd(1)-N(5), 2.301 (7) Å; Cd(1)-N(1A), 2.335 (6) Å; Cd(1)-N(3A), 2.403 (6) Å (atoms labeled "A" are generated by symmetry). (b) A cartoon diagram of the same cation. (c) An alternative space-filling depiction of the cation, emphasizing the double-helical character. (d) A view illustrating the π-stacking interactions.

Scheme I



Studies with 2,2′:6′,2″:6″,2‴:6‴,2⁗-quinquepyridine[3] led us to propose that 2,2′:6′,2″:6″,2‴:6‴,2⁗:6⁗,2′′′′′-sexipyridine (L) should form double helical complexes with second- and third-row transition elements in which each metal is in an N_6 environment. The required ligand was prepared as indicated in Scheme I,[5] with the key step being the simultaneous preparation of rings 2 and 4 in a Kröhnke-type synthesis.[6] The reaction of L with a variety of metal ions was investigated, and complexes of stoichiometry $\{M_2L_2\}$ (from FAB mass spectroscopy) were obtained with copper(II), manganese(II), iron(II), palladium(II), ruthenium(II), and cadmium(II). The reaction of cadmium(II) acetate with L in methanol, followed by the addition of ammonium hexafluorophosphate and recrystallization from acetonitrile, yields $[Cd_2L_2][PF_6]_4{\cdot}4MeCN$,[7] which molecular modeling indicates should possess a double-helical binuclear structure.

We have structurally characterized the complex, and the crystal and molecular structure of the $[Cd_2L_2]^{2+}$ cation is shown in Figure 1a.[8] The cation possesses a binuclear double-helical geometry,

---

(5) A mixture of **1** (1.0 g, 1.53 mmol),[9] **2** (0.91 g, 3.06 mmol),[10] and ammonium acetate (4 g) in 50 mL of aqueous methanol (1:1, 50 mL) was heated to reflux for 12 h. The crude product was collected by filtration, dried, added to toluene, and heated to reflux for 10 min with activated charcoal. The suspension was filtered hot through Celite, to give a yellow solution. Most of the solvent was removed on a rotary evaporator, and the residue was triturated with methanol and cooled to −40 °C; L precipitated and was collected by filtration as a cream-colored powder (114 mg, 16%): EI mass spectrum, *m/z* 464. The compound is too insoluble to obtain satisfactory ^1H NMR spectra in any solvent other than CF_3CO_2D, in which very broad resonances are observed.

(6) Krohnke, F. *Synthesis* **1976**, 1.

(7) FAB mass spectrum: *m/z* 1587 $\{Cd_2L_2(PF_6)_3\}$ 1442, 1297, 1040, 721, 576 (based upon ^{112}Cd). ^1H NMR: 10 resonances (δ 6.91 d, 7.20 t, 7.25 t, 7.90 t, 8.05 t, 8.20 d, 8.38 d, 8.56 dd, 8.64 d, 8.66 d).

with no short contacts to the hexafluorophosphate counterions. The double-helical geometry is emphasized in the sketch of the molecule presented in Figure 1b. The two cadmium atoms are in irregular six-coordinate N_6 environments, with all Cd–N distances in the expected range 2.301 (6)–2.403 (6) Å. The intramolecular Cd(1)–Cd(1A) distance is 4.173 (4) Å and is dictated by the conformation of the ligand set. It is worthy of note that this metal–metal distance is actually shorter than that of 4.503 (2) Å observed in the complex $[Cu_2(L^1)_2(OAc)][PF_6]_3$ (L^1 = 2,2′:6′,2″:6″,2‴:6‴,2⁗-quinquepyridine)[3] and is dictated by the double-helical arrangement of the ligands. The double-helical geometry is achieved by a twist of 57.3° between rings C and D such that each ligand presents a terpyridyl donor set to each cadmium ion. Each terpyridyl moiety is approximately planar, with interplanar angles between adjacent rings in the range 2.0–13.2°. A stacking interaction is observed between terpyridyl fragments of the two ligands, with closest interplanar contacts between rings B and C′ (3.736 Å) and rings D and E′ (3.561 Å); all other interplanar contacts are greater than 4.0 Å. These structural features are seen in the space-filling representation of the cation presented in Figure 1c. We consider that the stacking interaction observed between the aromatic ligands is the most important feature in the formation of the double-helical geometry, and this interaction is emphasized in Figure 1d. This is the first example of a double-helical binuclear complex incorporating two near-octahedral six-coordinate metal ions coordinated to the helicate-forming ligand, although the binding of ancillary ligands in the case of nickel(II) and cobalt(II) complexes of quinquepyridine allows the formation of two six-coordinate sites. In contrast, the complex cation $[Pd_2(quinquepy)_2]^{4+}$ contains two *five*-coordinate metal ions.[11] The ^1H NMR spectrum of the complex indicates 10 magnetically distinct proton resonances, strongly suggesting that the double-helical geometry (which results in local D_2 symmetry about each metal ion) is maintained in solution. The complex is electrochemically inert, merely exhibiting irreversible absorption processes at moderate negative potentials.

The crystal-structural analysis confirms our predictions that the sexidentate ligand L allows the preparation of binuclear double-helical complexes with second-row (and third-row) transition elements, and we are currently investigating the properties of the ruthenium(II) complexes.[12]

Acknowledgment. We thank the Science and Engineering Research Council for support.

Registry No. 1, 87007-78-1; **2**, 51928-95-1; L, 124561-86-0; $[Cd_2L_2][PF_6]_4 \cdot 4MeCN$, 124561-89-3.

Supplementary Material Available: Details of the crystal structure determination and tables of aromatic positional and isotropic equivalent thermal parameters, anisotropic thermal parameters, bond distances, and bond angles for $[Cd_2(C_{30}H_{20}N_6)_2][PF_6]_4 \cdot 4CH_3CN$ (6 pages); listing of observed and calculated structure factors for $[Cd_2(C_{30}H_{20}N_6)_2][PF_6]_4 \cdot 4CH_3CN$ (24 pages). Ordering information is given on any current masthead page.

---

[8] Crystal data for $C_{68}H_{52}N_{16}F_{24}P_4Cd_2$: monoclinic, $C2/c$, a = 20.466 (8) Å, b = 18.872 (6) Å, c = 20.617 (5) Å, β = 111.98 (2)°, V = 7384 Å^3, Z = 4, D(calcd) = 1.71 g cm^{-3}, λ (Mo Kα) = 0.710 73 Å (graphite monochromator), μ = 7.7 cm^{-1}. A Nicolet R3m/V diffractometer was used to collect 7732 reflections (5° ≤ 2θ ≤ 50°) on a pale yellow crystal 0.28 × 0.26 × 0.70 mm. Of these, 6512 were independent and 3968 observed [$I \geq 3\sigma(I)$]. The data were corrected for Lorentz and polarization effects, and an empirical absorption correction was applied. The cadmium atom was located by direct methods. The remaining non-hydrogen atoms were located by iterative least-squares refinement and difference Fourier synthesis. All non-hydrogen atoms of the cations and anions were refined anisotropically. The hydrogen atoms of the cation were included in idealized positions. R = 0.0599, R' = 0.0634. All computations used SHELXTL-PLUS: Sheldrick, G., University of Göttingen, Federal Republic of Germany, 1986.

[9] Constable, E. C.; Ward, M. D., manuscript in preparation.

[10] Ison, R. R.; Franks, F. M.; Soh, K. S. *J. Pharm. Pharmacol.* **1973**, *25*, 887.

[11] Constable, E. C.; Ward, M. D.; Tocher, D. A., unpublished results.

[12] Constable, E. C.; Ward, M. D.; Tocher, D. A. *Inorg. Chem.*, manuscript submitted.

[2$_2$](1,5)Cyclooctatetraenophane

Leo A. Paquette* and Mark A. Kesselmayer

Evans Chemical Laboratories
The Ohio State University
Columbus, Ohio 43210

Received November 8, 1989

The view has long been held that [2$_n$]cyclophanes constructed of decks possessing $4n$ π-electrons or a combination of decks with $4n$ and ($4n$ + 2) π-electrons will differ intrinsically in chemical properties from structural counterparts where both decks have ($4n$ + 2) π-electrons.[1,2] The ability to alter cavity size reversibly by means of redox reactions holds particular fascination.[3,4] Unusual opportunities for metal complexation are also offered. For these and many other reasons, the parent cyclooctatetraenophanes constitute attractive synthetic targets.[5] Since they are unknown, we have sought to develop new and versatile synthetic technology for their acquisition. Herein we detail the successful preparation of the first member of this series, [2$_2$](1,5)-cyclooctatetraenophane (**9**) and present an early glimpse at the properties inherent to this fascinating molecule.

The route to **9** began with the [6.3.3]propellenedione **1**, a tricyclic compound readily available[6] from *cis*-5-cyclooctene-1,2-dione.[7] Its conversion to acetonide **2**[8] was efficiently accomplished (83%) by dihydroxylation with catalytic (0.1 mol %) osmium tetraoxide and direct acetonide formation.[9] Lithium aluminum hydride reduction of **2**, subsequent 2-fold xanthate elimination to give **3**, and mild acidic hydrolysis provided the diol as a mixture of olefin isomers (56% overall). The formation of two regioisomeric dienes at this point is not of long-range consequence, since they converge to a single intermediate at a later stage.

Application of the Swern protocol to **3** gave **4** (69%) with minimal complication stemming from overoxidation or cleavage.[10] As shown in Scheme I, submission of **4** to the Cook–Weiss procedure[6] served admirably to generate the pivotal pentacyclic diketone **5** (85%). This highly crystalline solid was next subjected to the same two-step reduction–elimination sequence utilized previously on **3**. As anticipated, the resulting tetraene **6** (70%) could be allylically tetrabrominated when heated (20 min) with 4 equiv of *N*-bromosuccinimide and a catalytic quantity of AIBN in carbon tetrachloride solution. The unpurified product mixture was directly converted into the unusual bis(semibullvalene) **7′** ⇄ **7″** by coupling with nickel carbonyl (34% isolated).[11] The simplicity of the ^1H and ^{13}C NMR spectra of this hydrocarbon[12] attests to the highly fluxional character of both of its divinyl-

---

[1] Boekelheide, V. *Top. Curr. Chem.* **1983**, *113*, 87.

[2] Garbe, J. E.; Boekelheide, V. *J. Am. Chem. Soc.* **1983**, *105*, 7384.

[3] Müllen, K. *Chem. Rev.* **1984**, *84*, 603.

[4] Heinz, W.; Räder, H.-J.; Müllen, K. *Tetrahedron Lett.* **1989**, *30*, 159.

[5] 1,2-Bridging (Paquette, L. A.; Wang, T.-Z. *J. Am. Chem. Soc.* **1988**, *110*, 8192. Paquette, L. A.; Wang, T.-Z.; Luo, J.; Cottrell, C. *Ibid.* **1987**, *109*, 3730), 1,3-bridging (Wang, T.-Z.; Paquette, L. A. *Tetrahedron Lett.* **1988**, *29*, 41. Paquette, L. A.; Wang, T.-Z.; Luo, J.; Cottrell, C. E.; Clough, A. E.; Anderson, L. B. *J. Am. Chem. Soc.* **1990**, *112*, 239. 1,4-bridging (Paquette, L. A.; Trova, M. P. *Ibid.* **1988**, *110*, 8197; *Tetrahedron Lett.* **1986**, *27*, 1895), and 1,5-bridging (Paquette, L. A.; Trova, M. P. *Ibid.* **1987**, *28*, 4354. Paquette, L. A.; Trova, M. P.; Luo, J.; Clough, A. E.; Anderson, L. B. *J. Am. Chem. Soc.* **1990**, *112*, 228.

[6] Mitschk, R.; Oehldrich, J.; Takahashi, K.; Cook, J. M. *Tetrahedron* **1981**, *37*, 4521.

[7] Yates, P.; Lewars, E. G.; McCabe, P. H. *Can. J. Chem.* **1972**, *50*, 1548.

[8] All of the illustrated compounds were obtained in analytically pure form and characterized satisfactorily by IR, 300-MHz ^1H NMR, ^{13}C NMR, MS, and combustion data.

[9] The dihydroxy dione possesses considerable water solubility and is consequently more tedious to isolate.

[10] (a) Christl, M.; Kraft, A. *Angew. Chem., Int. Ed. Engl.* **1988**, *27*, 1369. (b) Tobe, Y.; Furukawa, T.; Kobiro, K.; Kakiuchi, K.; Odaira, Y. *J. Org. Chem.* **1989**, *54*, 488. (c) Kawada, K.; Gross, R. S.; Watt, D. S. *Synth. Commun.* **1989**, *19*, 777.

[11] Paquette, L. A.; Trova, M. P.; Luo, J.; Clough, A. E.; Anderson, L. B. *J. Am. Chem. Soc.* **1990**, *112*, 228.

[12] ^1H NMR (300 MHz, $CDCl_3$): δ 5.16 (t, J = 3.8 Hz, 2 H), 4.99 (t, J = 3.8 Hz, 2 H), 4.18 (t, J = 3.8 Hz, 4 H), 4.09 (t, J = 3.8 Hz, 4 H), 1.60–1.40 (m, 8 H). ^{13}C NMR (75 MHz, $CDCl_3$): (ppm) 120.37, 118.62, 94.08, 91.55, 65.19, 26.75.

0002-7863/90/1512-1258\$02.50/0