References

Byriel, K. A., Lynch, D. E., Smith, G. & Kennard, C. H. L. (1991). *Aust. J. Chem.* **44**, 1459–1464.

Choi, C. S. & Abel, J. E. (1972). *Acta Cryst.* **B28**, 193–201.

Coffey, S. (1977). *Rodd's Chemistry of Carbon Compounds*, Vol. III, p. 48. Amsterdam: Elsevier.

Etter, M. C. (1991). Private communication.

Etter, M. C. & Frankenbach, G. M. (1989). *Chem. Mater.* **1**, 10–12.

Etter, M. C., Frankenbach, G. M. & Adsmond, D. A. (1990). *Mol. Cryst. Liq. Cryst.* **187**, 25–39.

Etter, M. C., Frankenbach, G. M. & Bernstein, J. (1989). *Tetrahedron Lett.* **30**, 3617–3620.

Fair, M. C. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.

Issa, Y. M., Hindaway, A. M., Issa, R. M. & Nassar, A. M. G. (1980). *Rev. Roum. Chim.* **25**, 1535–1541.

Lai, T. F. & Marsh, R. E. (1967). *Acta Cryst.* **22**, 885–893.

Lechat, J. (1984). *Acta Cryst.* **A40**, C-264.

Lynch, D. E., Smith, G., Byriel, K. A. & Kennard, C. H. L. (1992*a*). *Acta Cryst.* **C48**, 533–536.

Lynch, D. E., Smith, G., Byriel, K. A. & Kennard, C. H. L. (1992*b*). *Z. Kristallogr.* **200**, 73–82.

Lynch, D. E., Smith, G., Byriel, K. A. & Kennard, C. H. L. (1992*c*). *J. Chem. Soc. Chem. Commun.* pp. 300–301.

Lynch, D. E., Smith, G., Byriel, K. A., Kennard, C. H. L. & Whittaker, A. K. (1994). *Aust. J. Chem.* **47**, 309–319.

Lynch, D. E., Smith, G., Byriel, K. A., Kennard, C. H. L., Whittaker, A. K. & Hanna, J. V. (1994). *Aust. J. Chem.* **47**, 1401–1411.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures.* Univ. of Cambridge, England.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.

Stecher, P. G. (1968). *The Merck Index*, 8th ed., p. 53. Rahway: Merck.

Ueda, M., Onishi, H. & Nagu, T. (1986). *Acta Cryst.* **C42**, 462–464.

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A Hydroxide Inclusion Complex of a Methylene-Bridged Tetrapyrimidinium Macrocycle

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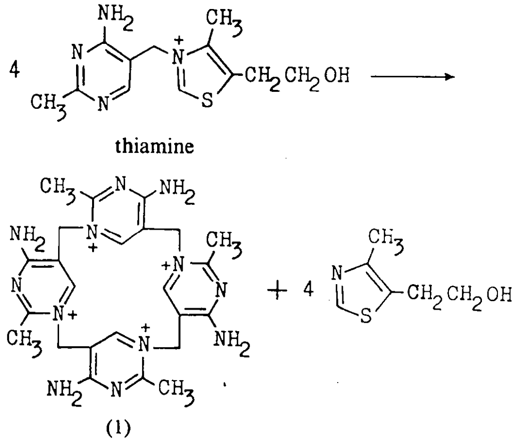
Abstract

In the structure of 4,10,16,22-tetraamino-6,12,18,24-tetramethyl-5,11,17,23-tetraaza-1,7,13,19-tetraazoniapentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacosa-1(25),3,5,7(26),9,11,13(27),15,17,19(28),21,23-dodecaene dichloride dihydroxide octahydrate, $C_{24}H_{32}N_{12}^{4+}.2OH^-.2Cl^-.8H_2O$, the tetracationic macrocycle consists of four substituted pyrimidinium rings connected by four methylene bridges. The cation captures two hydroxide anions, one on either side of the plane through the macrocycle, *via* pyrimidinium ring–OH^- electrostatic interactions and C—H···O hydrogen bonds, while two chloride anions are located outside the macrocycle. The orientation of the OH^- ions is affected by hydrogen bonds between OH^- and a chloride ion and OH^- and a water molecule.

Comment

Macrocyclic molecules as anion receptors have received much attention because of their selective complexation with a variety of chemically and biologically important anions (Lehn, 1988). Polyammonium macrocycles have been studied extensively in order to find species capable of binding anions (Graf & Lehn, 1976; Dietrich, Hosseini, Lehn & Sessions, 1981). However, stable anion complexes of macrocyclic ligands remain less common than their metal-ion complexes. Recently, a new class of cyclic polypyrimidinium cations has been reported (Cramer & Carrie, 1990; Cramer, Fermin, Kuwabara, Kirkup, Selman, Aoki, Adeyemo & Yamazaki, 1991; Hu, 1993). These were produced by the oligomerization of thiamine resulting from thiazole displacement by the pyrimidine ring. The two isolated polypyrimidinium cations are the cyclic tetramer 16-pyrimidinium crown-4 (1) and the hexamer 24-pyrimidinium crown-6 (Cramer & Carrie, 1990; Cramer *et al.*, 1991). The cyclic cations hold Cl^-, NO_3^- or HgI_4^{2-} anions, depending upon their cavity size, using a combination of weak non-covalent interactions. We report here the crystal structure of a hydroxide inclusion complex of (1), [16-pyrimidinium crown-4].2OH.2Cl.$8H_2O$.



(1)

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Thiamine.Cl.HCl and $Ni(AcO)_2.4H_2O$ were dissolved in water and mixed in a 2:1 molar ratio to give a green solution. Vapour diffusion of acetone into this solution resulted in a colour change to yellow. Crystals formed after several weeks; these decomposed rapidly when removed from the solution.

The bond lengths and angles of (1) are in agreement with those found in protonated thiamine (Cramer, Maynard & Ibers, 1981). Complex (1) adopts a sterically favourable conformation with the opposite pyrimidinium rings *cis* and the adjacent ones *trans* with respect to the plane of 16-membered ring; this minimizes the steric hindrance of the exocyclic substitutents. The dihedral angles between the planes through opposite pyrimidinium rings are 55.8 (3) (rings I and III) and 57.7 (2)° (rings II and IV). This conformation allows each pair of opposite pyrimidinium rings to act as 'opening pincers' and grip a hydroxide anion on either side of the macrocycle through pyrimidinium ring–OH^- electrostatic contacts and C—H···O hydrogen bonds. The contact distances from O(1) to rings I and III are 3.21 (1) and 3.19 (1) Å and those from O(2) to II and IV are 3.28 (1) and 3.18 (1) Å. The two chloride anions are located outside the macrocycle.

In the polypyrimidinium–anion complexes reported previously, the anions bind to the macrocycles symmetrically, with a twofold axis for the tetrameric Cl^- and NO_3^- complexes and a *pseudo*-threefold axis for the hexameric HgI_4^{2-} complex. It is interesting to note that in the present structure the cation host includes the OH^- guests in an asymmetric manner, *i.e.* both O—H bonds are at oblique angles to the plane of the macrocycle with the H atoms pointing outwards, rather than perpendicular to it and symmetrical. Furthermore, O(1) acts as the acceptor atom for two hydrogen bonds [from C(24) and C(44) on the macrocycle] while O(2) only accepts a hydrogen bond from C(34); no hydrogen bond is observed between O(2) and C(14). The asymmetric binding of the OH^- anions distorts the framework of the macrocycle. The average deviation of the four bridging C atoms from their least-squares plane (1.13 Å) is larger than that found in the chloride complex (0.91 Å; Hu, 1993). It can be seen from Fig. 1 that the orientation of the OH^- anions to the macrocycle is influenced by the hydrogen bonds between O(1) and O(*W*2) and between O(2) and Cl(1) in which the guests act as donors. Cl(1) also blocks the hydrogen-bonding position of O(2) to C(14). The asymmetric orientation of the OH^- anions results in reduction of the complex symmetry. This structure, together with those of the polypyrimidinium–anion complexes reported previously, shows that the complexation of various anions by this type of macrocyclic ligand is possible through electrostatic and hydrogen-bonding interactions.

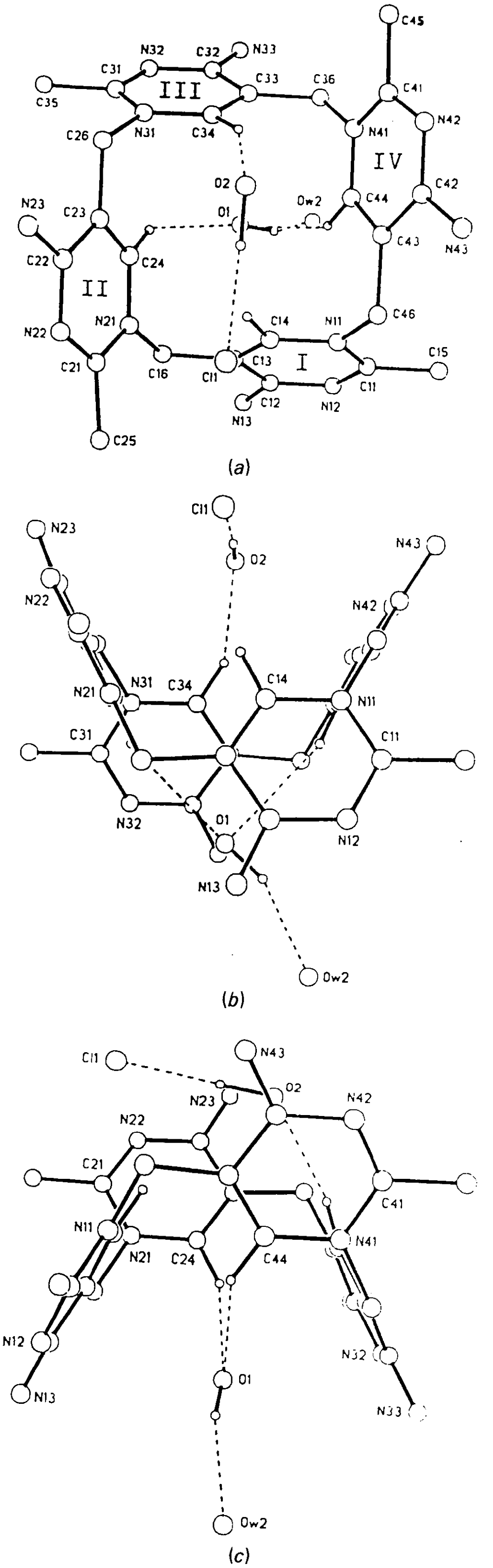

Fig. 1. Structure of (1) showing its interactions with OH^- anions; (*a*) a top view, (*b*) a side view and (*c*) a side view after a rotation of 90° from (*b*) around the axis perpendicular to the macrocycle plane. Broken lines denote hydrogen bonds.

Experimental

Crystal data

$C_{24}H_{32}N_{12}^{4+}.2OH^{-}.2Cl^{-}.8H_2O$
$M_r = 737.64$
Monoclinic
$P2_1/c$
$a = 10.740$ (2) Å
$b = 17.606$ (4) Å
$c = 19.775$ (4) Å
$\beta = 102.39$ (2)°
$V = 3652$ (1) Å^3
$Z = 4$
$D_x = 1.34$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 21 reflections
$\theta = 5.2$–$13.4°$
$\mu = 0.24$ mm^{-1}
$T = 294$ K
Tabular
$0.55 \times 0.50 \times 0.42$ mm
Light yellow

Data collection

Nicolet $R3m/E$ diffractometer
ω scans
Absorption correction: empirical
$T_{\min} = 0.89$, $T_{\max} = 0.91$
6787 measured reflections
6420 independent reflections
2692 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.013$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 23$
2 standard reflections monitored every 98 reflections intensity variation: <2.4%

Refinement

Refinement on F
$R = 0.078$
$wR = 0.086$
$S = 3.36$
2692 reflections
433 parameters
H-atom parameters not refined
Unit weights applied

$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.64$ e Å^{-3}
$\Delta\rho_{\min} = -0.34$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N(11)	0.1085 (7)	0.6759 (4)	0.3244 (4)	0.043 (3)
N(12)	0.0084 (7)	0.5582 (4)	0.3163 (4)	0.046 (3)
N(13)	−0.0062 (8)	0.4843 (4)	0.2213 (4)	0.061 (3)
C(11)	0.0439 (9)	0.6203 (5)	0.3510 (5)	0.048 (4)
C(12)	0.0340 (8)	0.5488 (5)	0.2529 (4)	0.041 (3)
C(13)	0.1003 (8)	0.6053 (5)	0.2225 (4)	0.041 (3)
C(14)	0.1335 (8)	0.6678 (5)	0.2600 (4)	0.038 (3)
C(15)	0.0131 (10)	0.6299 (6)	0.4196 (5)	0.059 (4)
C(16)	0.1317 (8)	0.5915 (5)	0.1534 (4)	0.044 (3)
N(21)	0.2031 (6)	0.6550 (4)	0.1311 (3)	0.038 (3)
N(22)	0.2183 (7)	0.7639 (4)	0.0661 (4)	0.046 (3)
N(23)	0.4058 (7)	0.8272 (4)	0.0753 (4)	0.050 (3)
C(21)	0.1489 (8)	0.7102 (5)	0.0863 (4)	0.044 (3)
C(22)	0.3439 (9)	0.7686 (5)	0.0941 (4)	0.041 (4)
C(23)	0.4050 (8)	0.7130 (5)	0.1418 (4)	0.040 (3)
C(24)	0.3317 (8)	0.6559 (5)	0.1575 (4)	0.043 (3)
C(25)	0.0104 (9)	0.7107 (6)	0.0622 (5)	0.056 (4)
C(26)	0.5448 (9)	0.7213 (5)	0.1730 (5)	0.051 (4)
N(31)	0.5935 (6)	0.6611 (4)	0.2250 (4)	0.045 (3)
N(32)	0.7011 (7)	0.5458 (4)	0.2564 (4)	0.051 (3)
N(33)	0.7200 (8)	0.4975 (5)	0.3638 (4)	0.061 (3)
C(31)	0.6584 (9)	0.5992 (6)	0.2107 (5)	0.053 (4)
C(32)	0.6770 (8)	0.5531 (5)	0.3202 (5)	0.045 (3)
C(33)	0.6078 (8)	0.6167 (5)	0.3393 (4)	0.042 (3)
C(34)	0.5690 (8)	0.6687 (5)	0.2894 (5)	0.045 (3)
C(35)	0.6896 (11)	0.5909 (7)	0.1406 (5)	0.079 (5)
C(36)	0.5778 (9)	0.6192 (5)	0.4097 (4)	0.048 (4)
N(41)	0.5003 (7)	0.6871 (4)	0.4188 (4)	0.047 (3)
N(42)	0.4795 (7)	0.8085 (4)	0.4635 (4)	0.054 (3)
N(43)	0.2843 (8)	0.8658 (4)	0.4469 (4)	0.061 (3)
C(41)	0.5487 (9)	0.7511 (6)	0.4532 (5)	0.049 (4)
C(42)	0.3526 (9)	0.8063 (5)	0.4354 (5)	0.048 (4)
C(43)	0.2938 (8)	0.7425 (5)	0.3965 (4)	0.041 (3)
C(44)	0.3705 (8)	0.6838 (5)	0.3904 (4)	0.042 (3)
C(45)	0.6891 (10)	0.7561 (6)	0.4815 (6)	0.072 (5)
C(46)	0.1537 (8)	0.7452 (5)	0.3632 (4)	0.042 (3)
Cl(1)	0.1292 (3)	0.8533 (2)	0.1992 (2)	0.067 (1)
Cl(2)	0.3049 (3)	0.5304 (2)	0.4757 (1)	0.063 (1)
O(1)	0.3584 (8)	0.5158 (5)	0.2889 (5)	0.120 (5)
O(2)	0.4152 (8)	0.8269 (5)	0.2778 (5)	0.104 (4)
O(*W*1)	0.3882 (10)	0.4470 (8)	0.1714 (6)	0.177 (7)
O(*W*2)	0.3435 (8)	0.3824 (5)	0.3849 (5)	0.109 (4)
O(*W*3)	0.1547 (8)	0.2808 (6)	0.3163 (5)	0.123 (5)
O(*W*4)	0.0890 (10)	0.4049 (6)	0.4398 (4)	0.124 (5)
O(*W*5)	0.3919 (7)	0.5063 (4)	0.0518 (4)	0.087 (4)
O(*W*6)	0.1362 (11)	0.0390 (6)	0.4896 (6)	0.143 (6)
O(*W*7)	0.0035 (5)	0.3985 (3)	0.0893 (3)	0.032 (2)
O(*W*8)	0.1885 (5)	0.2821 (3)	0.1728 (3)	0.040 (2)

Table 2. *Selected geometric parameters* (Å, °)

N(11)—C(11)	1.37 (1)	N(31)—C(31)	1.36 (1)
N(11)—C(14)	1.36 (1)	N(31)—C(34)	1.36 (1)
N(11)—C(46)	1.47 (1)	N(31)—C(26)	1.49 (1)
C(11)—N(12)	1.30 (1)	C(31)—N(32)	1.32 (1)
C(11)—C(15)	1.47 (1)	C(31)—C(35)	1.50 (1)
N(12)—C(12)	1.35 (1)	N(32)—C(32)	1.35 (1)
C(12)—C(13)	1.43 (1)	C(32)—C(33)	1.44 (1)
C(12)—N(13)	1.32 (1)	C(32)—N(33)	1.32 (1)
C(13)—C(14)	1.33 (1)	C(33)—C(34)	1.34 (1)
C(13)—C(16)	1.49 (1)	C(33)—C(36)	1.49 (1)
N(21)—C(21)	1.36 (1)	N(41)—C(41)	1.36 (1)
N(21)—C(24)	1.37 (1)	N(41)—C(44)	1.39 (1)
N(21)—C(16)	1.48 (1)	N(41)—C(36)	1.49 (1)
C(21)—N(22)	1.32 (1)	C(41)—N(42)	1.30 (1)
C(21)—C(25)	1.46 (1)	C(41)—C(45)	1.49 (1)
N(22)—C(22)	1.35 (1)	N(42)—C(42)	1.36 (1)
C(22)—C(23)	1.42 (1)	C(42)—C(43)	1.43 (1)
C(22)—N(23)	1.32 (1)	C(42)—N(43)	1.33 (1)
C(23)—C(24)	1.35 (1)	C(43)—C(44)	1.34 (1)
C(23)—C(26)	1.50 (1)	C(43)—C(46)	1.51 (1)
C(11)—N(11)—C(14)	120.1 (7)	C(31)—N(31)—C(34)	119.0 (7)
C(11)—N(11)—C(46)	122.1 (8)	C(31)—N(31)—C(26)	123.2 (8)
C(14)—N(11)—C(46)	117.7 (7)	C(34)—N(31)—C(26)	117.8 (7)
N(11)—C(11)—N(12)	120.8 (9)	N(31)—C(31)—N(32)	123.3 (9)
N(11)—C(11)—C(15)	120.4 (8)	N(31)—C(31)—C(35)	119.8 (8)
N(12)—C(11)—C(15)	118.8 (9)	N(32)—C(31)—C(35)	116.8 (9)
C(11)—N(12)—C(12)	119.8 (8)	C(31)—N(32)—C(32)	117.8 (8)
N(12)—C(12)—N(13)	115.7 (8)	N(32)—C(32)—N(33)	115.5 (8)
N(12)—C(12)—C(13)	121.7 (8)	N(32)—C(32)—C(33)	122.2 (8)
N(13)—C(12)—C(13)	122.6 (9)	N(33)—C(32)—C(33)	122.3 (9)
C(12)—C(13)—C(14)	116.0 (8)	C(32)—C(33)—C(34)	115.8 (9)
C(12)—C(13)—C(16)	120.2 (8)	C(32)—C(33)—C(36)	119.5 (8)
C(14)—C(13)—C(16)	123.8 (8)	C(34)—C(33)—C(36)	124.6 (9)
N(11)—C(14)—C(13)	121.5 (8)	N(31)—C(34)—C(33)	121.9 (9)
C(13)—C(16)—N(21)	112.5 (7)	C(33)—C(36)—N(41)	112.0 (7)
C(21)—N(21)—C(24)	119.8 (7)	C(41)—N(41)—C(44)	118.7 (8)
C(21)—N(21)—C(16)	124.0 (7)	C(41)—N(41)—C(36)	124.2 (7)
C(24)—N(21)—C(16)	116.2 (7)	C(44)—N(41)—C(36)	117.1 (7)
N(21)—C(21)—N(22)	121.3 (8)	N(41)—C(41)—N(42)	123.6 (8)
N(21)—C(21)—C(25)	118.8 (8)	N(41)—C(41)—C(45)	118.9 (9)
N(22)—C(21)—C(25)	119.9 (8)	N(42)—C(41)—C(45)	117.5 (9)
C(21)—N(22)—C(22)	120.1 (7)	C(41)—N(42)—C(42)	118.3 (8)
N(22)—C(22)—N(23)	116.9 (7)	N(42)—C(42)—N(43)	116.8 (8)
N(22)—C(22)—C(23)	120.7 (8)	N(42)—C(42)—C(43)	122.1 (8)
N(23)—C(22)—C(23)	122.4 (8)	N(43)—C(42)—C(43)	121.1 (8)
C(22)—C(23)—C(24)	117.1 (8)	C(42)—C(43)—C(44)	116.3 (8)
C(22)—C(23)—C(26)	119.0 (8)	C(42)—C(43)—C(46)	119.6 (8)
C(24)—C(23)—C(26)	123.9 (8)	C(44)—C(43)—C(46)	124.0 (7)
N(21)—C(24)—C(23)	120.7 (8)	N(41)—C(44)—C(43)	120.9 (8)
C(23)—C(26)—N(31)	112.7 (8)	C(43)—C(46)—N(11)	112.9 (7)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
C(24)—H(C24)···O(1)	2.63	3.55 (1)	146
C(34)—H(C34)···O(2)	2.26	3.22 (1)	152
C(44)—H(C44)···O(1)	2.63	3.56 (1)	146
O(1)—H(O1)···O(*W*2)	2.13	3.04 (1)	153
O(2)—H(O2)···Cl(1)	2.09	3.165 (9)	177

The crystal for data collection was sealed in a glass capillary with a small quantity of mother liquor. The structure was solved by direct methods and refined by block-diagonal least squares. The H atoms were located on a difference map except for eight of those attached to water molecules. All non-H atoms were refined anisotropically and the H atoms were fixed with isotropic displacement parameters (U = 0.06 Å^2) in the final cycles of refinements. All calculations were performed using the *SHELXTL* crystallographic software package (Sheldrick, 1983).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1097). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cramer, R. E. & Carrie, M. J. J. (1990). *Inorg. Chem.* **29**, 3902–3904.
Cramer, R. E., Fermin, V., Kuwabara, E., Kirkup, R., Selman, M., Aoki, K., Adeyemo, A. & Yamazaki, H. (1991). *J. Am. Chem. Soc.* **113**, 7033–7034.
Cramer, R. E., Maynard, R. B. & Ibers, J. A. (1981). *J. Am. Chem. Soc.* **103**, 76–81.
Dietrich, B., Hosseini, M. W., Lehn, J. M. & Sessions, R. B. (1981). *J. Am. Chem. Soc.* **103**, 1282–1283.
Graf, E. & Lehn, J. M. (1976). *J. Am. Chem. Soc.* **98**, 6403–6405.
Hu, N.-H. (1993). *Jiegou Huaxue*, **12**, 60–64.
Lehn, J. M. (1988). *Angew. Chem. Int. Ed. Engl.* **27**, 89–112.
Sheldrick, G. M. (1983). *SHELXTL User's Manual.* Revision 4. Nicolet XRD Corporation, Madison, Wisconsin, USA.

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Camphene-1-carboxylic Acid: Stereochemistry of the Nametkin Shift

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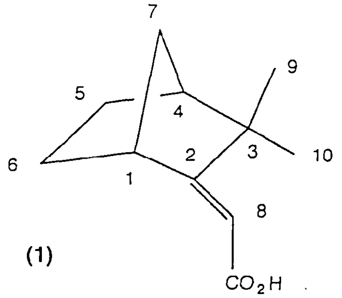
Abstract

The title compound, 2,2-dimethyl-3-methylenebicyclo[2.2.1]heptane-1-carboxylic acid, is a hydrogen-bonded dimer, $(C_{11}H_{16}O_2)_2$. This structure determination provides some evidence for the idea that, in the Nametkin Shift, migration of the *exo* methyl group may be marginally preferred.

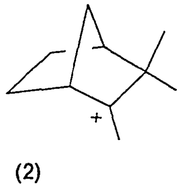
Comment

The Nametkin rearrangement refers to 3,2-methyl shifts in norbornyl cations (Nametkin & Brussoff, 1927). Both the methyl and the 3,2-hydride shifts have been well documented, and a general preference for an *exo* shift is observed (Berson, Hammons, McRowe, Bergman, Remanik & Houston, 1967; David, Everling, Kilian, Stothers & Vaughan, 1973), although authentic examples of *endo* shifts exist (Bushell & Wilder, 1967; Dadson, Hutchinson & Money, 1990). Attention has centred on the reasons for the preferred *exo* shifts and, in particular, on the nature of the transition state for migration.

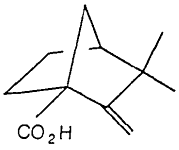
A crystal structure determination of camphene-8-carboxylic acid, (1), revealed that the methyl groups were unsymmetrically disposed with respect to the double bond (Moews, Knox & Vaughan, 1978). Thus, the dihedral angle C8—C2—C3—C9 was 65.1°, compared with a value of 55.9° for C8—C2—C3—C10. From this, the authors concluded that the observed conformations of the *gem*-dimethyl groups could result, in the corresponding cation (2) of the Nametkin rearrangement (shown as the classical ion for convenience), in a preferred *exo,exo* 3,2-methyl migration, since the p orbital at C2 and the C3—C9 [*i.e.* the C3—C(Me)$_{exo}$] bond are closer to being parallel. Camphene-8-carboxylic acid exists, as is normal, as a hydrogen-bonded dimer in the solid state. However, this structure carries a carboxylic acid group on the double bond, which is the part of the molecule under closest scrutiny. Accordingly, we have undertaken an X-ray structure determination of camphene-1-carboxylic acid (3) in which the carboxylic acid group is located at the bridgehead position.



(1)



(2)



(3)

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