

interactions between the π orbitals of the two groups. Similarly to other systems with polarized double bonds (Ganazzoli, Meille & Gronchi, 1986), the enamionone ring shows extensive π delocalization involving both the carbonyl and the aminic N which displays the features of sp^2 hybridization. The N1–C5–C7–C8 torsion angle is $-4.0(4)^\circ$, while C5–C7–C8–C9 and C4–N1–C5–C7 measure $-177.5(2)$ and $179.6(2)^\circ$ respectively. As a consequence the deviations of atoms C9, C8, O3, C7, C5, N1, C4 from the least-squares planes they define are within $0.19(3)$ Å, while HN and C6 are at distances of $0.08(2)$ and $0.138(4)$ Å respectively from the same plane. It is noteworthy that the bond lengths and angles of the enamionone ring in the title compound are insignificantly different from the corresponding values found in *cis-s-cis* enamionones coordinated to Pd (Iida, Yuasa, Kibayashi & Iitaka, 1981) possibly also because of the large standard deviations involved in that structural analysis.

The carboxypropyl chain bond lengths and angles are also in the expected ranges but its conformation is of interest. The C3–C4–N1–C5, the C2–C3–C4–N1 and the O1–C1–C2–C3 torsion angles are $93.1(3)$, $172.8(2)$ and $171.2(2)^\circ$ respectively while C1–C2–C3–C4 is $-71.8(3)^\circ$. This arrangement brings O1, O2 and HO1 close to the enamionone plane and is probably due to the formation of dimeric structures (Fig. 1) in which the enamionone heteroatoms of one molecule are hydrogen bonded to the carboxyl O atoms of the molecule related by the $(-x+1, -y, -z)$ symmetry operator to the original one. The shorter hydrogen bonds involve both intra- and intermolecularly the ketone rather than the carboxyl carbonyl

oxygen as acceptor. Since ketonic carbonyls are known (Taylor & Kennard, 1984) to be better acceptors than un-ionized carboxyls, this feature may account for the formation of the peculiar dimers found in the crystal structure of the title compound.

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Anti-Cancer Compounds. Structures of the Dimethyl[2-(1,8-naphthalenedicarboxido)ethyl]ammonium Cation with Cl^- , PtCl_4^{2-} , PtCl_6^{2-} and $\text{PtCl}_3[\text{HN}(\text{CH}_3)_2]^-$ Anions

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(Received 18 April 1988; accepted 1 August 1988)

Abstract. (1): Dimethyl[2-(1,8-naphthalenedicarboximido)ethyl]ammonium chloride, $\text{C}_{16}\text{H}_{17}\text{N}_2\text{O}_2^+\text{Cl}^-$, $M_r = 304.6$, monoclinic, $P2_1/c$, $a = 13.261(1)$, $b = 12.099(1)$, $c = 10.693(2)$ Å, $\beta = 118.02(1)^\circ$, $V = 1514.4(3)$ Å³, $Z = 4$, $D_m = 1.34$, $D_x = 1.336$ g cm⁻³, $\text{Cu K}\alpha$, $\lambda = 1.5418$ Å, $\mu = 2.63$ cm⁻¹, $F(000) = 640$,

0108-2701/89/010067-05\$03.00

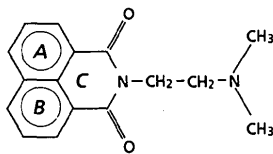
$T = 293$ K, $R = 0.040$ for 2531 reflections. (2): Dimethyl[2-(1,8-naphthalenedicarboximido)ethyl]ammonium tetrachloroplatinate(II), $(\text{C}_{16}\text{H}_{17}\text{N}_2\text{O}_2^+)_2\text{[PtCl}_4\text{]}^{2-}$, $M_r = 875.5$, monoclinic, $P2_1/c$, $a = 12.548(1)$, $b = 8.957(1)$, $c = 14.829(1)$ Å, $\beta = 105.95(1)^\circ$, $V = 1602.6(2)$ Å³, $Z = 2$, $D_m = 1.82$,

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$D_x = 1.813 \text{ g cm}^{-3}$, Mo $K\alpha$, $\lambda = 0.71069 \text{ \AA}$, $\mu = 50.01 \text{ cm}^{-1}$, $F(000) = 896$, $T = 293 \text{ K}$, $R = 0.029$ for 2796 reflections. (3): Dimethyl[2-(1,8-naphthalenedicarboximido)ethyl]ammonium hexachloroplatinate(IV), $(C_{16}H_{17}N_2O_2^+)_2[PtCl_6]^{2-}$, $M_r = 946.1$, monoclinic, $P2_1/c$, $a = 9.717 (1)$, $b = 12.748 (1)$, $c = 14.228 (6) \text{ \AA}$, $\beta = 105.89 (1)^\circ$, $V = 1695.1 (7) \text{ \AA}^3$, $Z = 2$, $D_m = 1.86$, $D_x = 1.853 \text{ g cm}^{-3}$, Mo $K\alpha$, $\lambda = 0.71069 \text{ \AA}$, $\mu = 49.10 \text{ cm}^{-1}$, $F(000) = 964$, $T = 293 \text{ K}$, $R = 0.026$ for 3162 reflections. (4): Dimethyl[2-(1,8-naphthalenedicarboximido)ethyl]ammonium trichloro-(dimethylamino)platinate(II), $(C_{16}H_{17}N_2O_2^+)[PtCl_3(C_2H_7N)]^-$, $M_r = 614.6$, monoclinic, $P2_1/c$, $a = 16.009 (2)$, $b = 6.586 (1)$, $c = 20.385 (3) \text{ \AA}$, $\beta = 91.49 (1)^\circ$, $V = 2148.6 (5) \text{ \AA}^3$, $Z = 4$, $D_m = 1.89$, $D_x = 1.900 \text{ g cm}^{-3}$, Mo $K\alpha$, $\lambda = 0.71069 \text{ \AA}$, $\mu = 72.50 \text{ cm}^{-1}$, $F(000) = 1188$, $T = 293 \text{ K}$, $R = 0.052$ for 2911 reflections. In all four crystals the cation contains a regular pattern of long and short bonds in the *A* and *B* naphthyl rings, as observed previously.

Introduction. Two classes of compounds which exhibit anti-cancer properties are the platinum drugs (e.g. *cis*-platin) and intercalators (e.g. 9-aminoacridine). Each class disrupts the normal function of DNA. The platinum compounds bind selectively to the nucleic acid bases in a monodentate or bidentate fashion, and they may form intra- or inter-strand crosslinks. The intercalators sandwich between the stacked nucleic acid bases causing partial unwinding of the helical structure.

We have been attempting to combine both of the above functionalities into a single molecule, which would be a derivative of *cis*-platin with one or both amines being at the end of a suitable length tether to a known intercalator. The intercalator chosen was *N*-[2-(dimethylamino)ethyl]-1,8-naphthalenedicarboximide (below) which was generously provided by Dr W. A.



Denny of the University of Auckland Medical School Cancer Research Laboratory. Although platinum-intercalator compounds are known where the amine is either primary or secondary (Bowler, Hollis & Lippard, 1984) none is available for a tertiary amine. The difficulty of coordinating a tertiary amine to Pt is well known, and this factor has thus far thwarted our attempts to synthesize the target molecules. During our experimentation we have succeeded in crystallizing the parent intercalator as its hydrochloride salt (1) and have determined its crystal structure. We have also isolated and crystallized the $PtCl_4^{2-}$, $PtCl_6^{2-}$ and $PtCl_3-$

$[HN(CH_3)_2]^+$ salts and determined their structures for comparison with the structure of the parent compound.

Experimental. Crystals obtained: (1) by ethyl acetate diffusion into a concentrated aqueous solution, (2) by methanol diffusion into a conc. HCl solution of (1) at room temperature, (3) as for (2) but at 278 K, (4) by diethyl ether diffusion into a chloroform/dimethylformamide solution. Crystal sizes: (1) $0.60 \times 0.21 \times 0.30 \text{ mm}$, (2) $0.50 \times 0.16 \times 0.24 \text{ mm}$, (3) $0.30 \times 0.16 \times 0.10 \text{ mm}$, (4) $0.18 \times 0.24 \times 0.16 \text{ mm}$. Nonius CAD-4 diffractometer, (1) Ni-filtered Cu $K\alpha$ radiation, $\lambda = 1.5418 \text{ \AA}$, (2) Zr-filtered Mo $K\alpha$ radiation, $\lambda = 0.71069 \text{ \AA}$, (3) and (4) graphite-monochromated Mo $K\alpha$ radiation, $\lambda = 0.71069 \text{ \AA}$. $2\theta/\omega$ scans, variable scan speeds, 60 s max. time, scan width (1) 1.0° in θ , (2) 0.9° , (3) 1.0° , (4) 0.8° . Lattice parameters refined using 25 reflections in the ranges: (1) $39 \leq 2\theta \leq 50^\circ$, (2) $27 \leq 2\theta \leq 34^\circ$, (3) $32 \leq 2\theta \leq 36^\circ$, (4) $23 \leq 2\theta \leq 26^\circ$. $[(\sin\theta)/\lambda]_{\max}$ (1) 0.601, (2) 0.682, (3) 0.704, (4) 0.639 \AA^{-1} . Range of hkl : $hk \pm l$ for (1) and (3), $\pm hk - l$ for (2), $\pm hkl$ for (4). For (1) 3031 independent reflections measured ($0 \leq h \leq 16$, $0 \leq k \leq 15$, $-13 \leq l \leq 13$), 2531 observed [criterion $I > 3\sigma(I)$]. For (2) 3632 measured ($-21 \leq h \leq 21$, $0 \leq k \leq 12$, $-20 \leq l \leq 0$), 2796 observed. For (3) 4216 measured ($0 \leq h \leq 14$, $0 \leq k \leq 18$, $-21 \leq l \leq 21$), 3162 observed. For (4) 4052 measured ($-21 \leq h \leq 21$, $0 \leq k \leq 9$, $0 \leq l \leq 20$), 2911 observed. Standard reflections: three checked every 100 reflections, no significant variations. (1) $\bar{3}43, \bar{5}20, \bar{4}2\bar{1}$, (2) $640, 9\bar{1}7, 257$, (3) $2, \bar{6}, \bar{1}0, 3, \bar{1}0, \bar{2}, 64\bar{8}$, (4) $\bar{8}20, \bar{9}\bar{1}\bar{1}, 037$. Corrections for Lorentz and polarization. Absorption corrections: none for (1), empirical absorption curves derived from azimuthal scans for (2), (3) and (4) (North, Phillips & Mathews, 1968). Max./min. transmission: (2) 0.999/0.799, (3) 0.999/0.937, (4) 0.971/0.787.

Structure solution: Patterson and ΔF electron density maps and full-matrix least-squares refinement on F using SHELX76 (Sheldrick, 1976). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). For (1) H atoms located and refined isotropically. Parameters in least-squares refinement: (1) 258, (2) 205, (3) 214, (4) 244. Weights $w = [\sigma^2(F) + gF^2]^{-1}$, g for: (1) 2.29×10^{-3} , (2) 2.70×10^{-3} , (3) 2.70×10^{-3} , (4) 1.18×10^{-2} . Final R factors: (1) $R = 0.040$, $wR = 0.051$, (2) $R = 0.029$, $wR = 0.034$, (3) $R = 0.026$, $wR = 0.030$, (4) $R = 0.052$, $wR = 0.054$. $(\Delta/\sigma)_{\max}$ in final refinement cycle: (1) -0.138 [z of C(1'')], max. H 0.305 [x of H(7)], (2) 0.095 [y of C(8b')], (3) -0.023 [y of Cl(2)], (4) 0.041 [x of C(2')]. $S =$ (1) 1.42, (2) 0.83, (3) 0.50, (4) 0.37. Max. and min. heights in final Fourier synthesis (1) 0.24, -0.20 , (2) 0.64, -0.99 , (3) 0.75, -1.07 , (4) 2.51, -3.73 e \AA^{-3} . Largest peak: (1) between C(8) and C(8b'), (2) 0.99 \AA from C(3), (3) and (4) close to Pt atom.

Table 1. Atomic positions ($\times 10^5$ for Cl, $\times 10^4$ for other atoms) and equivalent isotropic thermal parameters for (1)

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
Cl	98721 (4)	6820 (3)	23413 (5)	3.42 (3)
N(1)	6920 (1)	3980 (1)	-874 (2)	3.3 (1)
N(2)	9681 (1)	2839 (1)	-515 (2)	2.9 (1)
O(1 <i>b'</i>)	6569 (2)	4980 (2)	-2818 (2)	7.4 (2)
O(8 <i>b''</i>)	7293 (1)	3062 (2)	1130 (2)	5.0 (1)
C(1)	4957 (2)	4550 (2)	-2506 (2)	4.5 (1)
C(2)	4185 (3)	5049 (3)	-3735 (3)	6.1 (2)
C(3)	3006 (3)	5064 (3)	-4083 (4)	7.2 (2)
C(4)	2644 (2)	4626 (3)	-3222 (4)	6.2 (2)
C(4 <i>a</i>)	3406 (2)	4088 (2)	-1952 (3)	5.1 (1)
C(5)	3060 (2)	3603 (3)	-1027 (4)	6.2 (2)
C(6)	3823 (3)	3085 (3)	192 (4)	6.5 (2)
C(7)	4984 (2)	3054 (2)	526 (4)	5.3 (1)
C(8)	5354 (2)	3526 (2)	-348 (2)	3.8 (1)
C(8 <i>a</i>)	4585 (2)	4049 (2)	-1606 (2)	4.0 (1)
C(1 <i>b'</i>)	6187 (2)	4540 (2)	-2120 (2)	4.5 (1)
C(8 <i>b''</i>)	6583 (2)	3497 (2)	47 (2)	3.6 (1)
C(1')	8149 (2)	3975 (2)	-444 (2)	3.4 (1)
C(2')	8531 (2)	2824 (1)	-585 (2)	3.1 (1)
C(1'')	10604 (2)	3183 (2)	900 (2)	3.7 (1)
C(2'')	9938 (2)	1736 (2)	-918 (2)	3.7 (1)

Table 3. Atomic positions ($\times 10^5$ for Cl, $\times 10^4$ for other atoms) and equivalent isotropic thermal parameters for (3)

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
Pt	0	0	0	2.04 (1)
Cl(1)	3148 (15)	17778 (9)	3119 (10)	3.2 (1)
Cl(2)	16356 (15)	534 (11)	-9217 (10)	3.7 (1)
Cl(3)	18805 (14)	-3505 (12)	13683 (9)	3.3 (1)
O(1 <i>b'</i>)	6566 (5)	6288 (3)	1666 (3)	4.3 (2)
O(8 <i>b''</i>)	8367 (4)	4022 (3)	-140 (3)	4.3 (2)
N(1)	7466 (4)	5137 (3)	779 (3)	3.0 (2)
N(2)	10523 (4)	6967 (3)	1744 (3)	3.0 (2)
C(1)	5519 (5)	4596 (5)	1463 (4)	2.9 (2)
C(2)	4664 (7)	4796 (5)	2059 (5)	4.0 (2)
C(3)	3715 (7)	3991 (7)	2216 (5)	5.4 (3)
C(4)	3627 (6)	3040 (5)	1736 (5)	4.4 (2)
C(4 <i>a</i>)	4500 (5)	2837 (4)	1097 (4)	3.4 (2)
C(5)	4443 (6)	1863 (5)	602 (5)	4.1 (2)
C(6)	5362 (7)	1680 (5)	39 (5)	4.3 (2)
C(7)	6353 (6)	2443 (5)	-67 (4)	3.9 (2)
C(8)	6412 (5)	3409 (4)	398 (4)	3.0 (2)
C(8 <i>a</i>)	5478 (5)	3632 (4)	974 (3)	2.8 (2)
C(1 <i>b'</i>)	6516 (5)	5415 (4)	1322 (3)	3.1 (2)
C(8 <i>b''</i>)	7485 (5)	4187 (4)	315 (4)	2.9 (2)
C(1')	8486 (6)	5968 (4)	689 (4)	3.5 (2)
C(2')	9799 (6)	5914 (4)	1588 (4)	3.3 (2)
C(1'')	12072 (6)	6866 (5)	2308 (5)	4.6 (2)
C(2'')	9725 (9)	7711 (5)	2245 (5)	5.1 (3)

Table 2. Atomic positions ($\times 10^4$) and equivalent isotropic thermal parameters for (2)

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
Pt	5000	5000	0	2.80 (1)
Cl(1)	4793 (1)	2483 (1)	255 (1)	4.1 (1)
Cl(2)	3515 (1)	5493 (2)	586 (1)	4.8 (1)
O(1 <i>b'</i>)	9223 (3)	8015 (4)	3037 (3)	4.4 (2)
O(8 <i>b''</i>)	7405 (3)	3636 (5)	2453 (4)	5.4 (3)
N(1)	8360 (3)	5802 (5)	2693 (3)	3.2 (2)
N(2)	5921 (3)	8356 (5)	1863 (3)	3.4 (2)
C(1)	10218 (4)	5887 (5)	3771 (3)	2.6 (2)
C(2)	11164 (4)	6693 (6)	4210 (4)	3.5 (3)
C(3)	12082 (4)	5954 (8)	4791 (4)	4.3 (3)
C(4)	12063 (5)	4441 (9)	4937 (4)	4.2 (3)
C(4 <i>a</i>)	11101 (4)	3581 (6)	4483 (4)	3.2 (2)
C(5)	11058 (6)	2005 (6)	4592 (4)	4.6 (3)
C(6)	10139 (6)	1241 (6)	4109 (4)	4.6 (3)
C(7)	9205 (6)	1964 (6)	3536 (5)	4.2 (3)
C(8)	9220 (4)	3501 (5)	3427 (4)	2.9 (2)
C(8 <i>a</i>)	10177 (4)	4347 (5)	3895 (3)	2.6 (2)
C(1 <i>b'</i>)	9259 (4)	6672 (5)	3160 (4)	3.1 (2)
C(8 <i>b''</i>)	8255 (4)	4247 (6)	2818 (4)	3.2 (2)
C(1')	7425 (5)	6549 (7)	1999 (4)	4.2 (3)
C(2')	6604 (4)	7195 (6)	2493 (4)	4.0 (3)
C(1'')	6560 (9)	9818 (6)	1950 (7)	5.3 (4)
C(2'')	4847 (4)	8607 (7)	2114 (5)	4.8 (3)

Table 4. Atomic positions ($\times 10^5$ for Pt, $\times 10^4$ for other atoms) and equivalent isotropic thermal parameters for (4)

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
Pt	14268 (3)	53429 (7)	34842 (2)	2.41 (2)
Cl(1)	1354 (2)	3649 (4)	4467 (2)	3.4 (2)
Cl(2)	1212 (2)	8398 (5)	4006 (2)	3.6 (2)
Cl(3)	1579 (3)	6962 (6)	2500 (2)	4.7 (2)
O(1 <i>b'</i>)	2856 (6)	2698 (16)	1082 (6)	5.1 (7)
O(8 <i>b''</i>)	3999 (6)	8300 (20)	194 (6)	5.5 (7)
N(1)	3450 (5)	5441 (18)	634 (5)	3.5 (5)
N(2)	1310 (5)	7910 (15)	593 (5)	2.7 (4)
N(3)	1524 (6)	2547 (14)	3038 (4)	2.6 (4)
C(1)	4291 (10)	3124 (24)	1293 (8)	4.7 (7)
C(2)	4370 (13)	1380 (26)	1644 (8)	5.7 (8)
C(3)	5158 (12)	694 (29)	1893 (10)	6.0 (8)
C(4)	5843 (13)	1829 (33)	1796 (9)	6.5 (8)
C(4 <i>a</i>)	5810 (9)	3680 (28)	1395 (9)	5.6 (8)
C(5)	6498 (9)	4900 (31)	1240 (10)	5.9 (8)
C(6)	6441 (10)	6681 (35)	883 (9)	5.9 (8)
C(7)	5625 (8)	7369 (28)	609 (8)	5.1 (8)
C(8)	4939 (8)	6099 (25)	771 (8)	4.2 (7)
C(8 <i>a</i>)	5023 (8)	4330 (23)	1148 (7)	4.1 (7)
C(1 <i>b'</i>)	3472 (8)	3711 (25)	991 (8)	4.4 (7)
C(8 <i>b''</i>)	4105 (9)	6765 (24)	531 (7)	4.0 (7)
C(1')	2619 (7)	6097 (25)	339 (7)	4.3 (7)
C(2')	2207 (7)	7494 (20)	797 (6)	3.3 (5)
C(1'')	997 (9)	9684 (23)	984 (7)	3.9 (6)
C(2'')	759 (8)	6111 (23)	676 (7)	4.1 (6)
C(N3 <i>a</i>)	2251 (9)	2357 (25)	2591 (7)	4.7 (7)
C(N3 <i>b</i>)	707 (10)	2108 (22)	2683 (8)	4.6 (7)

Discussion. Final atomic parameters for the four structures are given in Tables 1-4.* Comparison of bond lengths and angles is presented in Tables 5 and 6 respectively. Atomic numbering of the cation is shown in Fig. 1. In each of the four structures the aliphatic side chain adopts an orientation such that maximum extension away from the naphthalimide ring system is

achieved. A common pattern of long and short bond lengths is found in *A* and *B* rings of the cation. Specifically the C(1)-C(2), C(3)-C(4), C(5)-C(6) and C(7)-C(8) bonds are shorter than the expected aromatic bond whilst C(2)-C(3), C(4)-C(4*a*), C(4*a*)-C(8*a*), C(4*a*)-C(5), C(6)-C(7) and C(8)-C(8*a*) are longer. This pattern of bond lengths has been observed in other *N*-substituted naphthalimide molecules

* Lists of anisotropic thermal parameters, H-atom parameters for (1) and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51293 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 5. Bond lengths (Å) for the salts of the *N*-1'-ethyl-1,8-naphthalenedicarboximido-2'-dimethylammonium cation

	(1)	(2)	(3)	(4)
Pt—Cl(1)	—	2.313 (1)	2.314 (1)	2.298 (3)
Pt—Cl(2)	—	2.305 (1)	2.322 (1)	2.306 (3)
Pt—Cl(3)	—	—	2.319 (1)	2.291 (3)
Pt—N(3)	—	—	—	2.06 (1)
O(1b')—C(1b')	1.206 (3)	1.22 (1)	1.21 (1)	1.21 (1)
O(8b')—C(8b')	1.217 (3)	1.19 (1)	1.23 (1)	1.23 (1)
N(1)—C(1b')	1.401 (3)	1.39 (1)	1.40 (1)	1.35 (1)
N(1)—C(8b')	1.387 (3)	1.43 (1)	1.38 (1)	1.38 (1)
N(1)—C(1')	1.470 (2)	1.49 (1)	1.48 (1)	1.51 (2)
N(2)—C(2')	1.491 (2)	1.50 (1)	1.50 (1)	1.51 (2)
N(2)—C(1'')	1.491 (3)	1.52 (1)	1.51 (1)	1.51 (2)
N(2)—C(2'')	1.489 (2)	1.51 (1)	1.52 (1)	1.49 (2)
N(3)—C(N3a)	—	—	—	1.50 (2)
N(3)—C(N3b)	—	—	—	1.51 (2)
C(1)—C(2)	1.369 (3)	1.39 (1)	1.36 (1)	1.36 (3)
C(1)—C(8a)	1.408 (4)	1.39 (1)	1.40 (1)	1.45 (2)
C(1)—C(1b')	1.483 (3)	1.47 (1)	1.48 (1)	1.49 (2)
C(2)—C(3)	1.428 (5)	1.40 (1)	1.44 (1)	1.42 (3)
C(3)—C(4)	1.332 (5)	1.37 (1)	1.38 (1)	1.34 (3)
C(4)—C(4a)	1.414 (4)	1.43 (1)	1.43 (1)	1.47 (3)
C(4a)—C(5)	1.400 (5)	1.42 (1)	1.42 (1)	1.41 (3)
C(4a)—C(8a)	1.428 (3)	1.42 (1)	1.43 (1)	1.41 (2)
C(5)—C(6)	1.370 (5)	1.36 (1)	1.37 (1)	1.38 (3)
C(6)—C(7)	1.408 (3)	1.40 (1)	1.41 (1)	1.48 (3)
C(7)—C(8)	1.369 (4)	1.39 (1)	1.39 (1)	1.43 (2)
C(8)—C(8a)	1.401 (3)	1.43 (1)	1.41 (1)	1.40 (2)
C(8)—C(8b')	1.479 (3)	1.46 (1)	1.47 (1)	1.48 (2)
C(1')—C(2')	1.513 (3)	1.53 (1)	1.54 (1)	1.48 (2)

Table 6. Bond angles (°) for the salts of the *N*-1'-ethyl-1,8-naphthalenedicarboximido-2'-dimethylammonium cation

	(1)	(2)	(3)	(4)
Cl(1)—Pt—Cl(2)	—	89.2 (1)	90.3 (0)	90.6 (1)
Cl(1)—Pt—Cl(3)	—	—	89.7 (1)	176.6 (1)
Cl(2)—Pt—Cl(3)	—	—	88.6 (1)	91.1 (1)
Cl(1)—Pt—N(3)	—	—	—	87.5 (2)
Cl(2)—Pt—N(3)	—	—	—	175.4 (3)
Cl(3)—Pt—N(3)	—	—	—	91.1 (3)
C(1b')—N(1)—C(8b')	124.6 (3)	125.3 (4)	125.6 (4)	127 (1)
C(1b')—N(1)—C(1')	118.1 (2)	116.0 (5)	115.1 (4)	118 (1)
C(8b')—N(1)—C(1')	117.1 (2)	116.6 (4)	119.3 (4)	115 (1)
C(2')—N(2)—C(1'')	112.1 (2)	110.3 (5)	111.2 (4)	109 (1)
C(2')—N(2)—C(2'')	110.1 (1)	110.2 (4)	110.4 (4)	113 (1)
C(1'')—N(2)—C(2'')	110.7 (2)	109.6 (5)	111.5 (5)	110 (1)
Pt—N(3)—C(N3a)	—	—	—	114 (1)
Pt—N(3)—C(N3b)	—	—	—	108 (1)
C(N3a)—N(3)—C(N3b)	—	—	—	112 (1)
C(2)—C(1)—C(8a)	120.1 (2)	120.6 (4)	122.4 (5)	120 (2)
C(2)—C(1)—C(1b')	120.0 (3)	119.3 (4)	118.8 (5)	121 (2)
C(8a)—C(1)—C(1b')	119.9 (2)	120.0 (4)	118.8 (5)	119 (1)
C(1)—C(2)—C(3)	119.3 (3)	119.7 (5)	119.1 (6)	122 (2)
C(2)—C(3)—C(4)	121.4 (3)	121.1 (5)	120.3 (6)	119 (2)
C(3)—C(4)—C(4a)	121.3 (3)	120.3 (5)	120.4 (6)	122 (2)
C(4)—C(4a)—C(5)	123.2 (3)	122.2 (5)	121.7 (5)	126 (2)
C(4)—C(4a)—C(8a)	117.8 (3)	117.9 (5)	118.7 (5)	118 (2)
C(5)—C(4a)—C(8a)	119.0 (3)	119.9 (6)	119.6 (5)	116 (2)
C(4a)—C(5)—C(6)	121.6 (2)	119.4 (6)	119.7 (5)	124 (2)
C(5)—C(6)—C(7)	118.9 (3)	122.1 (5)	121.2 (6)	120 (2)
C(6)—C(7)—C(8)	121.1 (3)	119.5 (6)	120.1 (6)	114 (2)
C(7)—C(8)—C(8a)	120.7 (2)	120.4 (5)	120.4 (5)	123 (1)
C(7)—C(8)—C(8b')	119.7 (2)	119.4 (5)	119.9 (5)	116 (1)
C(8a)—C(8)—C(8b')	119.5 (2)	120.1 (4)	119.7 (5)	120 (1)
C(1)—C(8a)—C(4a)	120.1 (2)	120.3 (5)	119.0 (5)	119 (1)
C(1)—C(8a)—C(8)	121.3 (2)	121.2 (4)	122.0 (4)	120 (1)
C(4a)—C(8a)—C(8)	118.6 (2)	118.5 (5)	118.9 (5)	121 (1)
O(1b')—C(1b')—N(1)	119.8 (2)	119.2 (5)	119.7 (5)	123 (1)
O(1b')—C(1b')—C(1)	123.4 (2)	123.8 (5)	123.5 (5)	121 (1)
N(1)—C(1b')—C(1)	116.8 (2)	117.0 (4)	116.7 (5)	117 (1)
O(8b')—C(8b')—N(1)	119.7 (2)	119.5 (5)	120.4 (5)	121 (1)
O(8b')—C(8b')—C(8)	122.5 (2)	124.0 (5)	122.8 (5)	123 (1)
N(1)—C(8b')—C(8)	117.8 (2)	116.1 (4)	116.8 (5)	116 (1)
N(1)—C(1')—C(2')	110.3 (2)	110.2 (4)	108.6 (4)	109 (1)
N(2)—C(2')—C(1')	111.5 (1)	108.0 (4)	109.3 (4)	112 (1)

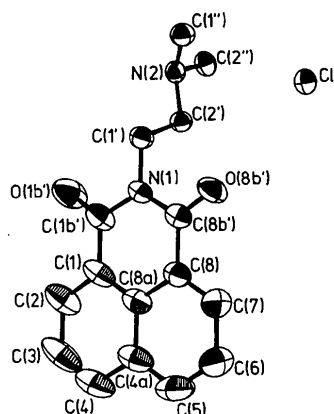


Fig. 1. Geometry and atomic numbering for (1).

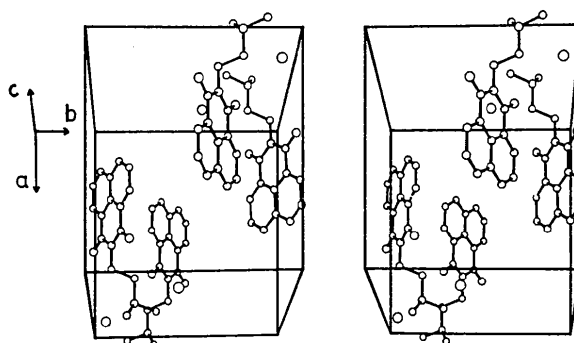


Fig. 2. Unit-cell packing for (1).

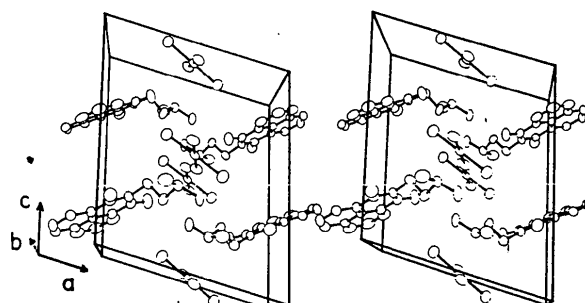


Fig. 3. Unit-cell packing for (2).

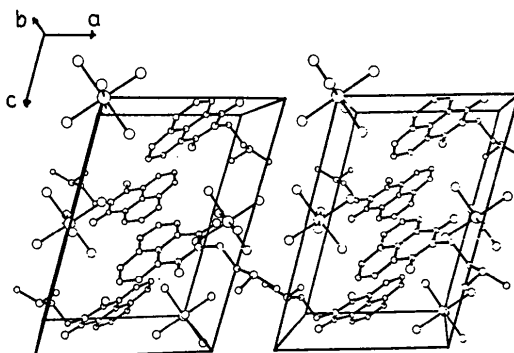


Fig. 4. Unit-cell packing for (3).

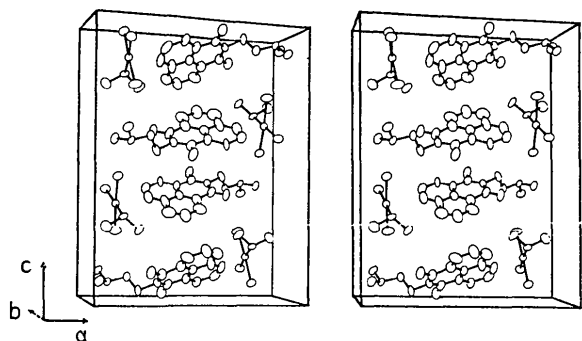


Fig. 5. Unit-cell packing for (4).

(Sobolev, Chetkina, Gol'der, Federov & Zavodnik, 1973; Grigor'eva, Chetkina, Neigauz & Gol'der, 1975; Grigor'eva & Chetkina, 1977).

The imide ring *C* consists of three consecutive aromatic carbons, two carbonyl carbons, and a tertiary N. In each structure the observed C(1*b'*)–N(1)–C(8*b'*) angle at N(1) deviates from the tetrahedral angle (the

angles average 125.6° in the four compounds) indicating considerable electron withdrawal by the adjacent imide carbonyls. The cations in each structure are essentially planar.

The unit-cell packing arrangements for compounds (1) to (4) are illustrated in Figs. 2 to 5.

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Acta Cryst. (1989). **C45**, 71–73

1,3,3,5,5,7,9,9,11,11-Decamethyl-2,4,6,8,10,12-hexaoxa-1,3,5,7,9,11-hexasila-bicyclo[5.5.2]tetradecane

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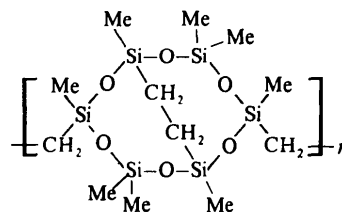
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(Received 14 December 1987; accepted 18 May 1988)

Abstract. C₁₂H₃₄O₆Si₆, *M_r* = 442.9, orthorhombic, *Pcan*, *a* = 12.083 (1), *b* = 12.083 (1), *c* = 17.702 (1) Å, *V* = 2584.3 (3) Å³, *Z* = 4, *D_x* = 1.138 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 0.34 mm⁻¹, *F*(000) = 952, *T* = 297 K, *R* = 0.047 for 969 observed reflections. The molecule has a twofold symmetry axis; the conformation of the hexasiloxane ring is a twisted saddle, the dihedral angle between the wings being 98.4 (3)°. The SiCCSi bridge has a synclinal conformation with a torsion angle of 67.3 (5)°. The crystal structure is loose with molecules forming layers coplanar to the *ab* and *bc* planes.

Introduction. Earlier we determined the crystal structure of a cyclotetrasiloxane with an intracyclic –CH₂CH₂– bridge (I) (Ovchinnikov, Shklover,

Struchkov, Astapova & Zhdanov, 1986). In this work the molecular and crystal structure of the cyclohexasiloxane analogue (II) is reported. Like (I), molecule (II) represents a single unit of the carbocyclosiloxane polymer (see below), a structural study of which is proposed in the future.



Experimental. Irregular-shaped single crystal 0.8 × 0.6 × 0.4 mm used for measurements of unit-cell parameters (12 reflections with 28 ≤ 2θ ≤ 32°) and

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