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Structure of 8-Dimethylamino-1-dimethylammonionaphthalene Hydrogen Squarate

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Abstract. (8-Dimethylamino-1-naphthyl)dimethylammonium 2-hydroxy-3,4-dioxocyclobut-1-en-olate, $C_{14}H_{19}N_2^+ \cdot C_4HO_4^-$, $M_r = 328.37$, orthorhombic, *Pbca*, $a = 18.319$ (3), $b = 14.868$ (2), $c = 12.219$ (1) Å, $V = 3328.1$ (8) Å³, $Z = 8$, $D_x = 1.311$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 1.0$ cm⁻¹, $F(000) = 1392$, $T = 150$ K, $R = 0.036$ for 3003 observed reflections with $I \geq 2.5\sigma(I)$. In the crystal structure of the title compound bis(dimethylamino)naphthalene acts as a proton sponge by accepting a proton from squaric acid. In the 1-dimethylamino-8-dimethylammonionaphthalene cation a strong asymmetric intramolecular hydrogen bond is formed with $N \cdots N$, $N-H$ and $N-H \cdots N$ 2.583 (2), 1.08 (2) Å and 157 (2)° respectively. The $N-H$ donor also is involved in a weak interaction with a squarate carbonyl group. The hydrogen squarate anions form

planar strongly hydrogen-bonded cyclic dimers across centres of inversion with $O \cdots O$, $O-H$ and $O-H \cdots O$ 2.477 (2), 0.97 (2) Å and 170 (2)° respectively. In contrast to the free base which displays severe puckering of the aromatic system and opposite deviations of the N atoms of 0.40 Å from the best plane, the aromatic system including both N atoms of the cation is planar.

Introduction. The NHN hydrogen bonds in homo-conjugated nitrogen bases play an important role in so-called proton sponges (Alder, Bowman, Steele & Wintermann, 1968; Hibbert, 1974; de Groot & Sikkema, 1976; Awwal & Hibbert, 1977; Alder, Goode, Miller, Hibbert, Hunte & Robbins, 1978; Glowiak, Malarski, Sobczyk & Grech, 1987). Among the NHN systems the bi-centre bases with 1,8-bis(dimethylamino)naphthalene (DMAN) as a representative proton sponge are of particular interest.

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In protonated DMAN strong hydrogen bonds are formed with extremely low frequency for the stretching vibration $\nu(\text{NHN})$ at about 500–600 cm⁻¹ (Hamann & Linton, 1976; Chojnacki, Laskowski & Malarski, 1983; Grech, Malarski & Sobczyk, 1985).

A search of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) yielded the structures of six complexes containing the 1-dimethylamino-8-dimethylammonionaphthalene cation. These complexes with their REFCODES are: the isomorphous [DMANH]⁺·[tris(hexafluoroacetylacetonato)]³⁻ Cu²⁺ and Mg²⁺ complexes [NAFACU and NAFAMG respectively, Truter & Vickery (1972)], DMANH⁺·Br⁻ [BUTNOT, Pyzalska, Pyzalski & Borowiak (1983)], [DMANH]⁺·[1-oxo-2-phenyl-1,2-dicarbododecaborate]⁻ [FIJWAW, Brown, Clegg, Colquhoun, Daniels, Stephenson & Wade (1987)], [DMANH]⁺·[2,4-dinitroimidazolate]⁻ [GADHEY, Glowiak, Malarski, Sobczyk & Grech (1987)], and [DMANH]⁺·[oxopentafluorotellurium]⁻ [GEKZIF, Miller, Abney, Rappé, Anderson & Strauss (1988)]. The N—H···N geometries of the DMANH⁺ fragments listed in Table 1 show that the N···N distances span a range of 2.554 (5)–2.65 (2) Å which is an indication of strong intramolecular hydrogen bonding.

It is remarkable that the N—H distances are much larger than usually found in N—H···N hydrogen bonds. The tendency of the H atom to move to a central position is often met with in strong possibly symmetrical hydrogen bonds and it implies that in this particular N—H···N system the potential is not the usual asymmetric double well, but approaches the symmetric double well and possibly the symmetric single well. This interpretation is supported by the central position of the H atom in BUTNOT as required by the space-group symmetry and by the curious finding in the isomorphous Cu and Mg complexes (NAFACU and NAFAMG) where the proton in the Cu complex is at a distance of 1.27 (17) Å from N(1) but in the Mg complex at 1.25 (11) Å from N(2). In order to obtain accurate structural information about the asymmetry of the potential in [DMANH]⁺ complexes we decided to undertake a low-temperature analysis of the complex of strongly basic DMAN (p*K* = 12.3) with the strongly acidic squaric acid (p*K* = 1.5).

Experimental. Crystals of the title compound were obtained from a solution in acetonitrile of equimolar amounts of DMAN and squaric acid. A yellowish rod-shaped crystal of dimensions 0.65 × 0.39 × 0.35 mm was used for data collection on an Enraf-Nonius CAD-4 diffractometer with Zr-filtered Mo *K*α radiation. Lattice parameters were determined from the setting angles of 25 reflections in the range 14.06 < θ < 17.78°. The diffracted inten-

Table 1. *Hydrogen-bond geometry of the DMANH⁺ fragments retrieved from the Cambridge Structural Database*

REFCODE	N···N (Å)	N—H (Å)	N—H···N (°)
NAFACU	2.65 (2)	1.27 (17)	148 (12)
NAFAMG	2.60 (1)	1.25 (11)	134 (8)
BUTNOT	2.554 (5)	1.31 (1)	153 (3)
FIJWAW	2.577 (3)	1.22 (3)	140 (3)
GADHEY	2.606 (3)	1.18 (3)	160 (3)
GEKZIF	2.574 (-)	1.17 (-)	159 (-)

sities of 3812 unique reflections were collected at liquid-nitrogen temperature using the ω -2 θ scan mode, $\omega = (0.65 + 0.35 \tan \theta)^\circ$, $2\theta_{\max} = 55^\circ$ and $0 \leq h \leq 23$, $0 \leq k \leq 19$, $0 \leq l \leq 15$ of which 3003 reflections were above the 2.5 $\sigma(I)$ level. Three periodically measured standard reflections (402, 023 and 230), measured every hour, showed an average deviation of less than 3% during the duration of the data collection.

Intensities were corrected for Lp effects, but not for absorption. The structure was solved by direct methods of *SHELXS86* (Sheldrick, 1986). The H atoms were located on difference Fourier maps and included in the refinement with individual isotropic thermal parameters. Anisotropic full-matrix least-squares refinement on *F* of 298 parameters converged at $R = 0.036$ and $wR = 0.040$ with $w = 3.0273/\sigma^2(F_o)$, $S = 0.86$, $(\Delta/\sigma)_{\text{av}} = 0.0007$, $(\Delta/\sigma)_{\text{max}} = 0.0037$. The isotropic extinction parameter converged to 0.0005. Maximum and minimum residual densities in the final difference map were 0.29 and -0.18 e Å⁻³ respectively. The scattering factors were those of Cromer & Mann (1968) and anomalous-dispersion terms from Cromer & Liberman (1970). Calculations were performed with *SHELX76* (Sheldrick, 1976) (refinement) and the *EUCLID* package (Spek, 1982) (geometry and illustrations) on a MicroVAXII computer.

Discussion. The final atomic coordinates with equivalent isotropic thermal parameters are given in Table 2* and bond distances and angles in Table 3. One proton of squaric acid is transferred to the N(1) atom of DMAN and the geometry of the intramolecular N(1)—H···N(2) hydrogen bond [N···N, N—H and N—H···N being 2.583 (2), 1.08 (2) Å and 157 (2)° respectively] clearly establishes this bridge as a truly asymmetric one. The weak N(1)—H···O(4) link is part of an asymmetric bifurcate N(1)—H···N(2), O(4) hydrogen bond with N···O 3.085 (2) Å

* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53461 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters (\AA^2) and their e.s.d.'s in parentheses

	x	y	z	U_{eq}^*
N(1)	0.34652 (6)	0.41957 (7)	0.47150 (9)	0.0160 (3)
N(2)	0.39342 (6)	0.40790 (7)	0.6703 (1)	0.0175 (3)
C(1)	0.35479 (7)	0.51811 (8)	0.4782 (1)	0.0161 (4)
C(2)	0.33529 (7)	0.57058 (9)	0.3912 (1)	0.0189 (4)
C(3)	0.34188 (8)	0.66496 (9)	0.3979 (1)	0.0219 (4)
C(4)	0.36864 (7)	0.70400 (9)	0.4905 (1)	0.0206 (4)
C(5)	0.38999 (7)	0.65111 (9)	0.5817 (1)	0.0175 (4)
C(6)	0.41694 (8)	0.69244 (9)	0.6781 (1)	0.0216 (4)
C(7)	0.43625 (8)	0.6422 (1)	0.7672 (1)	0.0232 (4)
C(8)	0.42893 (7)	0.54781 (9)	0.7640 (1)	0.0212 (4)
C(9)	0.40296 (7)	0.50550 (8)	0.6724 (1)	0.0166 (3)
C(10)	0.38299 (7)	0.55540 (8)	0.5773 (1)	0.0153 (3)
C(11)	0.39994 (8)	0.3787 (1)	0.3945 (1)	0.0217 (4)
C(12)	0.27016 (8)	0.39134 (9)	0.4460 (1)	0.0223 (4)
C(13)	0.46392 (8)	0.3599 (1)	0.6793 (2)	0.0283 (4)
C(14)	0.34049 (9)	0.3769 (1)	0.7539 (1)	0.0249 (4)
C(15)	0.32389 (7)	0.06421 (9)	0.6216 (1)	0.0203 (4)
C(16)	0.38838 (7)	0.01957 (9)	0.5801 (1)	0.0203 (4)
C(17)	0.41831 (8)	0.09990 (9)	0.5348 (1)	0.0201 (4)
C(18)	0.35340 (8)	0.15067 (9)	0.5736 (1)	0.0218 (4)
O(1)	0.27025 (5)	0.04163 (7)	0.67508 (9)	0.0261 (3)
O(2)	0.40744 (6)	-0.06563 (7)	0.5867 (1)	0.0292 (3)
O(3)	0.47464 (6)	0.12345 (7)	0.48311 (9)	0.0264 (3)
O(4)	0.33388 (6)	0.22894 (7)	0.5693 (1)	0.0327 (3)

$$*U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Table 3. Bond distances (\AA) and bond angles ($^\circ$) with e.s.d.'s in parentheses

N(1)—C(1)	1.475 (2)	C(6)—C(7)	1.366 (2)
N(1)—C(11)	1.487 (2)	C(7)—C(8)	1.410 (2)
N(1)—C(12)	1.493 (2)	C(8)—C(9)	1.369 (2)
N(2)—C(9)	1.462 (2)	C(9)—C(10)	1.427 (2)
N(2)—C(13)	1.480 (2)	O(1)—C(15)	1.227 (2)
N(2)—C(14)	1.481 (2)	O(2)—C(16)	1.316 (2)
C(1)—C(2)	1.367 (2)	O(3)—C(17)	1.260 (2)
C(1)—C(10)	1.428 (2)	O(4)—C(18)	1.219 (2)
C(2)—C(3)	1.411 (2)	C(15)—C(16)	1.447 (2)
C(3)—C(4)	1.363 (2)	C(15)—C(18)	1.513 (2)
C(4)—C(5)	1.418 (2)	C(16)—C(17)	1.426 (2)
C(5)—C(6)	1.418 (2)	C(17)—C(18)	1.486 (2)
C(5)—C(10)	1.430 (2)		
C(1)—N(1)—C(11)	111.9 (1)	N(2)—C(9)—C(8)	120.8 (1)
C(1)—N(1)—C(12)	112.8 (1)	N(2)—C(9)—C(10)	118.1 (1)
C(11)—N(1)—C(12)	111.7 (1)	C(8)—C(9)—C(10)	121.1 (1)
C(9)—N(2)—C(13)	111.9 (1)	C(1)—C(10)—C(5)	116.8 (1)
C(9)—N(2)—C(14)	112.0 (1)	C(1)—C(10)—C(9)	125.6 (1)
C(13)—N(2)—C(14)	111.7 (1)	C(5)—C(10)—C(9)	117.7 (1)
N(1)—C(1)—C(2)	119.8 (1)	O(1)—C(15)—C(16)	135.6 (1)
N(1)—C(1)—C(10)	118.0 (1)	O(1)—C(15)—C(18)	136.5 (1)
C(2)—C(1)—C(10)	122.2 (1)	C(16)—C(15)—C(18)	87.8 (1)
C(1)—C(2)—C(3)	120.0 (1)	O(2)—C(16)—C(15)	129.5 (1)
C(2)—C(3)—C(4)	120.2 (1)	O(2)—C(16)—C(17)	136.7 (1)
C(3)—C(4)—C(5)	121.0 (1)	C(15)—C(16)—C(17)	93.8 (1)
C(4)—C(5)—C(6)	120.6 (1)	O(3)—C(17)—C(16)	137.9 (1)
C(4)—C(5)—C(10)	119.9 (1)	O(3)—C(17)—C(18)	132.4 (1)
C(6)—C(5)—C(10)	119.6 (1)	C(16)—C(17)—C(18)	89.7 (1)
C(5)—C(6)—C(7)	121.0 (1)	O(4)—C(18)—C(15)	136.3 (1)
C(6)—C(7)—C(8)	119.8 (1)	O(4)—C(18)—C(17)	134.9 (1)
C(7)—C(8)—C(9)	120.8 (1)	C(15)—C(18)—C(17)	88.7 (1)

and N—H...O 108 (1) $^\circ$. The four-atom configuration is planar as follows from the sum of angles around the H atom [360 (2) $^\circ$]. The crystal structure is built up of hydrogen-bonded dimers of the squaric acid anions across a centre of symmetry and this dimer is linked, at either side, to DMANH $^+$ cations by weak

hydrogen bonds. A perspective view of this structural entity together with atom numbering is shown in Fig. 1. The hydrogen bonds linking the squaric acid anions are close to linear [O—H...O 170 (2) $^\circ$], very strong and also asymmetric as indicated by the O...O and O—H distances of 2.477 (2) and 0.97 (2) \AA respectively. Contrary to the structure of GADHEY (Glowiak *et al.*, 1987) in which the [DMANH] $^+$ fragments are to a larger extent isolated from the imidazolate anions, the structure of the title compound displays a weak hydrogen-bond interaction between the oppositely charged entities. Both the squaric acid anion and the naphthalene ring are planar with σ_{plane} values of 0.012 and 0.016 \AA respectively and are almost perpendicular [inter-ring angle 83.5 (5) $^\circ$]. This orientation has also been observed in GADHEY. Comparison of the geometries of [DMANH] $^+$ of the title compound and of GADHEY with that of unprotonated DMAN (Einspahr, Robert, Marsh & Roberts, 1973) reveals that protonation and the resulting formation of an intramolecular hydrogen bond has a pronounced effect, particularly on the torsion angles. Whereas in [DMANH] $^+$ the aromatic ring is planar and N(1) and N(2) are coplanar (deviations 0.00 and 0.06 \AA respectively), the situation in DMAN is different. The naphthalene ring is moderately puckered (σ_{plane} 0.106 \AA) and the N atoms are at opposite sides of the plane at 0.40 \AA . The puckering of DMAN primarily originates from a twist about the central C—C bond: C(4)—C(5)—C(10)—C(1) and C(6)—C(5)—C(10)—C(9) torsion angles are 8.9 and 10.5 $^\circ$ respectively, whereas in the title compound these angles are 0.6 (2) and 0.3 (2) $^\circ$ respectively. Clearly this twist forces the N atoms to opposite positions with respect to the ring which results in an N...N distance of 2.792 (8) \AA which is appreciably larger than the distance of about 2.44 \AA of the hypothetical planar structure (Einspahr *et al.*, 1973) derived from the structure of perdeuterionaphthalene (Pawley & Yeats, 1969). Another difference relates to the conformation of the dimethylamino groups. In [DMANH] $^+$ of the title compound and also of GADHEY the torsion angles about the two N—C bonds are such that at each N atom the methyl groups point away from the central part of the ring with *gauche* $^-$ and *gauche* $^+$ confor-

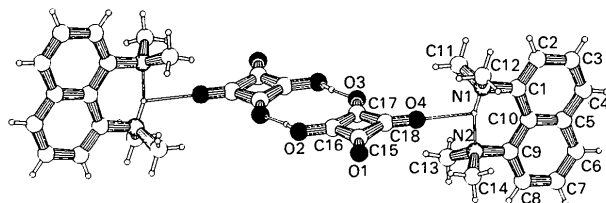


Fig. 1. Perspective view of the complex with atom numbering. A symmetry-related complex is also shown.

mations with respect to the C(1)—C(2) and C(8)—C(9) bonds respectively. These conformations bring the N(1)—H proton and the N(2) lone pair into a favourable position to form an intramolecular hydrogen bond. However, in DMAN these conformations are such that at each N atom one methyl group is almost eclipsed with either C(1)—C(2) or C(8)—C(9) (torsion angles -18 and -19.4° respectively) while the other methyl groups have torsion angles of 120 and 119° respectively.

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Structure of Lantadene A, the Major Triterpenoid of *Lantana Camara*, Red Variety*

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Abstract. Lantadene A, [22β(Z)]-2-methylisocrotonoyloxy-3-oxoolean-12-en-28-oic acid, is the major triterpenoid constituent of *L. Camara*, red variety. Mixed toxin preparation from lantana leaves has been found to exist in two molecular forms, of which only one was found to be hepatotoxic to guinea pigs. The structure of form (I), which is not hepatotoxic to guinea pigs, is reported. C₃₅H₅₂O₅, *M_r* = 552.35, orthorhombic, *P*2₁2₁2₁, *a* = 12.494 (2), *b* = 15.790 (2), *c* = 16.367 (2) Å, *V* = 3229.0 Å³, *Z* = 4,

D_x = 1.14 g cm⁻³, *D_m* = 1.15 g cm⁻³, λ(Cu Kα) = 1.5418 Å, μ = 5.9 cm⁻¹, *F*(000) = 1208, *T* = 296 (1) K, *R* = 0.061 for 3152 unique observed reflections. The *A/B* and *B/C* rings are *trans* fused while the *D/E* rings are *cis* fused. The packing of the molecule is stabilized by O—H⋯O hydrogen bonds.

Introduction. Ingestion of lantana foliage causes cholestasis and hepatotoxicity in animals (Sharma, Makkar & Dawra, 1988). Consensus did not exist regarding the capacity of lantadene A, which is the major constituent of *L. Camara*, red variety (Sharma, Dawra & Makkar, 1987), to elicit hepato-

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