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Bis(dicyclohexylammonium) 2,6-Pyridinedicarboxylate Dihydrate

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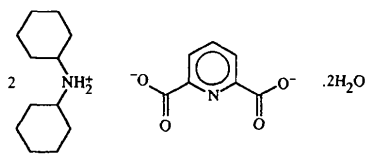
Abstract

In the title compound, $2C_{12}H_{24}N^+.C_7H_3NO_4^{2-}.2H_2O$, one of the ammonium cations is linked to the dianion [N···O = 2.717 (3), 2.846 (3) Å] to form an eight-membered ring; this monoanionic entity is linked to an adjacent entity through the second ammonium cation and a disordered water molecule [N···O = 2.871 (3), 2.908 (3); O···O 2.602 (6), 2.778 (6) Å] to form a linear chain. The second water molecule links two chains through the disordered water molecule [O···O = 2.674 (7), 2.825 (9) Å] into a ribbon running parallel to the *a* axis.

Comment

Whereas dicyclohexylamine reacts with monocarboxylic acids to yield ammonium carboxylates, as displayed by dicyclohexylammonium *O*-(*N*,*N*-dimethylthiocarbamoyl)acetate (Ng, 1993), this secondary amine affords an ammonium hydrogen dicarboxylate when reacted with dicarboxylic acids. The acidic proton in the dicyclohexylammonium hydrogen trithiocarbodiglycolate (Ng, 1995) and in dicyclohexylammonium hydrogen oxalate (Ng, 1994) is held by one of the two carboxylate ends of the anion. The amine reacts with 2,6-pyridinedicarboxylic acid to give a bis(dicyclohexylammonium) derivative containing two water molecules, (I). Bond dimensions involving the planar anion are similar to those of the parent acid, which crystallizes as a monohydrate that adopts a hydrogen-bonded sheet structure (Takusagawa, Hirotsu & Shimada, 1973). The dicyclohexylammonium–water interactions [2.871 (3), 2.908 (3) Å] are longer than those [2.738 (6), 2.741 (6) Å] found in dicyclohexylammonium *N*,*N*-dimethyldithiocarbamoylacetate hydrate (Ng, 1992), which adopts a three-dimensional network structure.



(I)

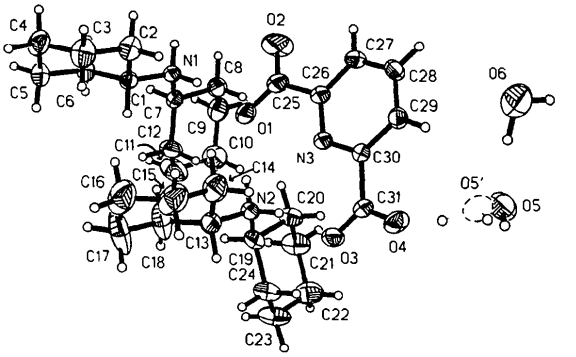


Fig. 1. *ZORTEP* (Zsolnai & Pritzkow, 1996) plot of bis(dicyclohexylammonium) 2,6-pyridinedicarboxylate dihydrate at the 30% probalility level. H atoms are drawn as spheres of arbitrary radii.

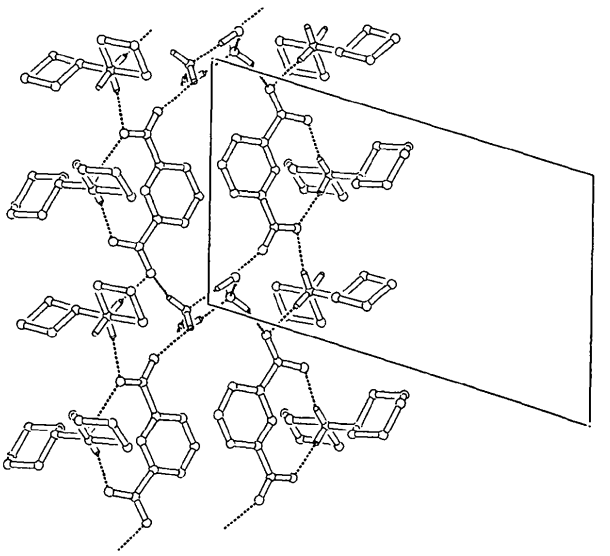


Fig. 2. Plot showing the hydrogen-bonding interactions when viewed along **b**.

Experimental

The compound was obtained as crystals on slow evaporation of an ethanol solution containing dicyclohexylamine and 2,6-pyridinedicarboxylic acid in a 2:1 molar ratio.

Crystal data

$2C_{12}H_{24}N^+.C_7H_3NO_4^{2-}.2H_2O$
M_r = 565.78
Monoclinic
$P2_1/n$
a = 10.579 (1) Å
b = 18.1356 (9) Å
c = 17.507 (2) Å
β = 106.044 (6)°
V = 3228.1 (6) Å^3
Z = 4
D_x = 1.164 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10–11°
μ = 0.080 mm^{-1}
T = 298 (2) K
Irregular block
0.53 × 0.53 × 0.53 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.922, T_{max} = 0.958
5858 measured reflections
5664 independent reflections
3326 reflections with $I > 2\sigma(I)$
R_{int} = 0.0181
θ_{max} = 24.97°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 20$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.195
S = 1.107
5664 reflections
370 parameters
H atoms: riding model with $U(H) = 1.5U_{eq}(C,N)$; water H atoms were calculated, U = 0.10 Å^2
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.370 e Å^{-3}
$\Delta\rho_{min}$ = −0.267 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C25	1.246 (4)	N1···O1	2.871 (3)
O2—C25	1.217 (4)	N1···O4^{i}	2.908 (3)
O3—C31	1.235 (3)	N2···O1	2.846 (3)
O4—C31	1.245 (3)	N2···O3	2.717 (3)
N1—C1	1.499 (3)	O5···O2^{ii}	2.602 (6)
N1—C7	1.496 (3)	O5···O4	2.778 (6)
N2—C13	1.493 (3)	O5···O6	2.674 (7)
N2—C19	1.499 (3)	O5′···O2^{ii}	2.656 (6)
N3—C26	1.337 (3)	O5′···O4	2.710 (5)
N3—C30	1.339 (3)	O6···O5^{iii}	2.825 (9)
C1—N1—C7	116.8 (2)	O1—C25—O2	124.0 (3)
C13—N2—C19	119.6 (2)	O1—C25—C26	118.6 (3)
C26—N3—C30	118.7 (2)	O2—C25—C26	117.5 (3)
N1—C1—C2	108.2 (2)	N3—C26—C25	117.2 (2)
N1—C1—C6	112.2 (2)	N3—C26—C27	122.0 (2)
N1—C7—C8	109.1 (2)	N3—C30—C29	122.2 (2)
N1—C7—C12	112.3 (2)	N3—C30—C31	115.5 (2)
N2—C13—C14	108.5 (2)	O3—C31—O4	124.6 (2)
N2—C13—C18	112.7 (3)	O3—C31—C30	117.8 (2)
N2—C19—C20	108.1 (2)	O4—C31—C30	117.7 (2)
N2—C19—C24	110.8 (2)		

Symmetry codes: (i) $1 + x, y, z$; (ii) $x - 1, y, z$; (iii) $-x, -y, -z$.

Data collection: *CAD-4-VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD-4-VAX/PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLUTON* (Spek, 1994), *ZORTEP* (Zsolnai & Pritzkow, 1996). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KH1129). Services for accessing these data are described at the back of the journal.

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Characterization of Quinoline Derivatives. I. 6,7-Dihydro-8-(4-methyl-1-piperazinyl)[1]-benzoxepino[4,5-*c*]quinoline 0.13-Hydrate

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Abstract

The title compound, $C_{22}H_{23}N_3O.0.13H_2O$, is a novel potent and selective serotonin 5-HT_3 receptor antagonist. Three independent molecules constitute the asymmetric unit. While two of these molecules show only small differences in their metric and conformational parameters, the third differs from the other two, mainly as a result of the conformation of the oxepine ring. The conformations of the seven-membered ring determine quite different orientations of the oxepine-fused benzene ring, whose role is important in the exploitation of biological activity.