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Crystal structure of dicyclohexylammonium *N*-phthaloylglycinate

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ABSTRACT Hydrogen bonds link the dicyclohexylammonium cations to the *N*-phthaloylglycinato anions in crystalline dicyclohexylammonium *N*-phthaloylglycinate to give rise to a linear chain structure. Crystals belong to the triclinic $P\bar{1}$ space group, with a = 8.9837 (8), b = 9.6253 (7), c = 12.3863 (7) Å, α = 100.029 (5), β = 93.394 (6), γ = 90.411 (7)°.

ABSTRAK Ikatan hidrogen menyambung kation-kation disikloheksilamonium kepada anion-anion *N*-ftaloglisinato dalam hablur disikloheksilamonium *N*-ftaloglisinat untuk mewujudkan suatu struktur rantai linear. Sebatian ini menghablur dalam ruang kumpulan $P\bar{1}$, dengan a = 8.9837 (8), b = 9.6253 (7), c = 12.3863 (7) Å, α = 100.029 (5), β = 93.394 (6), γ = 90.411 (7)°.

(dicyclohexylammonium *N*-phthaloylglycinate, crystal structure)

INTRODUCTION

In an extension to the studies on the structural characterization of the dicyclohexylammonium salts of biologically-active carboxylic acids [1, 2], this paper reports the crystal structure of the dicyclohexylammonium derivative of *N*-phthaloylglycine, a plant auxin [3].

EXPERIMENTAL

Phthalic anhydride was condensed with glycine to give *N*-phthaloylglycine [4]; the protected amino acid was treated with a molar equivalent of dicyclohexylamine in ethanol to yield the ammonium carboxylate. Single crystals were grown from its solution in the same solvent, and a 0.29 x 0.29 x 0.29 mm cube was used for the diffraction measurements.

Measurements was made on an Enraf-Nonius CAD4 diffractometer (graphite-monochromatized Mo-$K\alpha$ radiation, 0.71073 Å). Unit-cell dimensions were calculated from the 25 strongest reflections in the $10° \leq \theta \leq 12°$ range. The set of 3959 reflections was measured up to $2\theta_{max} = 45°$ ($0 \leq h \leq 10$, $-11 \leq k \leq 11$, $-14 \leq l \leq 14$) by ω-scans. Peak profiles were calculated [5]. The structure was solved by direct phase determination [6] and a θ-dependent correction [7] was applied to the data following isotropic refinement. Of the data 3701 (R_{int} = 0.031) independent data, the 1820 satisfying the $I \geq 3\sigma(I)$ criterion were used for solution and refinement. Non-H atoms were refined anisotropically; H-atoms were generated (C-H = 0.95 Å, B = 5 Å^2) and were allowed to ride on the parent C- or N-atoms. Full-matrix least-squares refinements on F with 253 variables converged to R = 0.095; R_w = 0.059 and S = 0.492 for the $w = [\sigma(F)^2 + (0.02F)^2 + 1]^{-1}$ [8] weighting scheme. Residual peaks ranged from -0.11 (3) to 0.17 (3) eÅ^{-3}. Scattering factors were taken from the *International Tables for X-ray Crystallography* [9]. All computations were performed on a MicroVAX minicomputer with the *MolEN* structure determination package. [10]. Atomic coordinates are listed in Table 1, the structure is shown as an *ORTEP* [11] plot at the 50% probability level in Fig. 1.

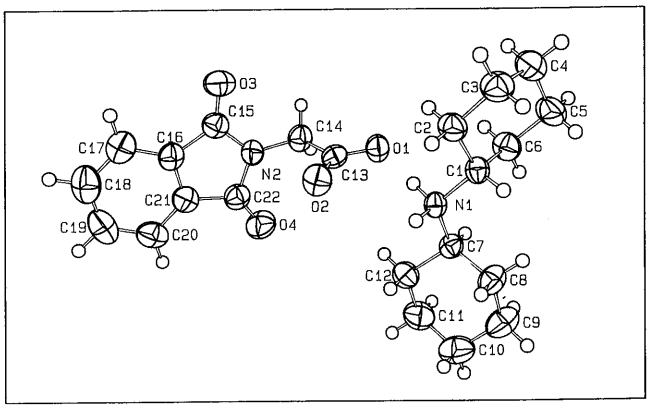

Figure 1: Atomic labeling scheme for dicyclohexylammonium *N*-phthaloylglycinate.

Crystal data: $C_{22}H_{30}NO_4$, FW = 386.50, triclinic, $P\bar{1}$ (No. 2), a = 8.9837 (8), b = 9.6253 (7), c = 12.3863 (7)Å, α = 100.029 (5), β = 93.394 (6), γ = 90.411 (7)°, V = 1052.7 (1) Å^3, $F(000)$ = 416, Z = 2, D_x = 1.219 g cm^{-3}, μ = 0.786 cm^{-1}.

RESULTS AND DISCUSSION

Dicyclohexylammonium *N*-phthaloylylycinate adopts a linear chain structure in which the anions are linked to

Table 1. Atomic coordinates and equivalent[a] isotropic temperature factors.

Atom	*x*	*y*	*z*	B_{eq}(Å²)
O1	0.6913 (3)	1.1582 (3)	0.5644 (3)	4.15 (7)
O2	0.9291 (3)	1.1445 (3)	0.6220 (3)	4.72 (8)
O3	0.9911 (4)	1.5170 (3)	0.7764 (3)	5.17 (8)
O4	0.8552 (4)	1.1114 (4)	0.8895 (3)	5.98 (9)
N1	0.7788 (4)	0.9389 (4)	0.4115 (3)	3.04 (7)
N2	0.8909 (4)	1.3129 (4)	0.8171 (3)	3.88 (8)
C1	0.7421 (4)	0.9750 (4)	0.3011 (3)	3.26 (9)
C2	0.8318 (5)	1.1046 (5)	0.2900 (4)	4.0 (1)
C3	0.8001 (5)	1.1442 (5)	0.1773 (4)	4.8 (1)
C4	0.6339 (6)	1.1613 (5)	0.1519 (4)	5.4 (1)
C5	0.5462 (6)	1.0314 (5)	0.1667 (4)	5.5 (1)
C6	0.5765 (5)	0.9974 (5)	0.2819 (4)	4.4 (1)
C7	0.7040 (5)	0.8102 (4)	0.4396 (3)	3.32 (9)
C8	0.7258 (6)	0.6826 (5)	0.3523 (4)	4.6 (1)
C9	0.6651 (7)	0.5497 (5)	0.3860 (5)	6.1 (1)
C10	0.7309 (7)	0.5278 (6)	0.4964 (5)	6.5 (2)
C11	0.7102 (7)	0.6553 (5)	0.5828 (4)	5.9 (1)
C12	0.7715 (6)	0.7890 (5)	0.5513 (4)	4.4 (1)
C13	0.8002 (5)	1.1894 (4)	0.6315 (4)	3.5 (1)
C14	0.7684 (5)	1.2914 (5)	0.7348 (4)	4.2 (1)
C15	0.9959 (5)	1.4215 (5)	0.8279 (4)	4.0 (1)
C16	1.1084 (5)	1.3942 (5)	0.9129 (4)	3.9 (1)
C17	1.2332 (5)	1.4703 (6)	0.9583 (4)	5.0 (1)
C18	1.3160 (6)	1.4204 (6)	1.0402 (5)	5.9 (1)
C19	1.2739 (6)	1.2998 (6)	1.0759 (4)	5.9 (1)
C20	1.1492 (6)	1.2215 (6)	1.0310 (4)	5.2 (1)
C21	1.0665 (5)	1.2723 (5)	0.9488 (4)	4.0 (1)
C22	0.9279 (5)	1.2161 (5)	0.8843 (4)	4.3 (1)

[a] $B_{eq} = 4/3\ [a^2B_{1,1} + b^2B_{2,2} + c^2B_{3,3} + ab\ (\cos\gamma)\ B_{1,2} + ac\ (\cos\beta)\ B_{1,3} + bc\ (\cos\alpha)\ B_{2,3}]$

Table 2. Bond distances (Å) and angles (°).

O1-C13	1.245 (5)	C7-C12	1.525 (6)
O2-C13	1.244 (5)	C8-C9	1.520 (7)
O3- C15	1.206 (6)	C9-C10	1.507 (8)
O4-C22	1.210 (6)	C10-C11	1.501 (8)
N1-C1	1.488 (6)	C11-C12	1.517 (7)
N1-C7	1.508 (5)	C13-C14	1.513 (6)
N2-C14	1.442 (6)	C15-C16	1.474 (7)
N2-C15	1.387 (6)	C16-C17	1.371 (7)
N2-C22	1.384 (6)	C16-C21	1.383 (7)
C1-C2	1.510 (6)	C17-C18	1.378 (8)
C1-C6	1.515 (6)	C18-C19	1.371 (8)
C2-C3	1.522 (7)	C19-C20	1.378 (8)
C3-C4	1.523 (7)	C20-C21	1.385 (7)
C4-C5	1.516 (7)	C21-C22	1.484 (7)
C5-C6	1.528 (7)	N1···O1	2.733 (5)
C7-C8	1.510 (6)	N1···O2'	2.783 (5)
C1-N1-C7	117.9 (3)	O1-C13-C14	115.2 (4)
C14-N2-C15	124.2 (4)	O2-C13-C14	118.2 (4)
C14-N2-C22	123.1 (4)	N2-C14-C13	114.3 (4)
C15-N2-C22	112.1 (4)	O3-C15-N2	125.0 (5)
N1-C1-C2	108.8 (4)	O3-C15-C16	129.0 (5)
N1-C1-C6	111.8 (4)	N2-C15-C16	106.0 (4)
C2-C1-C6	111.3 (4)	C15-C16-C17	130.7 (5)
C1-C2-C3	110.8 (4)	C15-C16-C21	108.2 (4)
C2-C3-C4	111.9 (4)	C17-C16-C21	121.1 (5)
C3-C4-C5	111.1 (5)	C16-C17-C18	117.7 (6)
C4-C5-C6	111.3 (4)	C17-C18-C19	121.1 (6)
C1-C6-C5	109.1 (4)	C18-C19-C20	122.1 (6)
N1-C7- C8	110.6 (4)	C19-C20-C21	116.5 (6)
N1-C7-C12	107.3 (4)	C16-C21-C20	121.5 (5)
C8-C7-C12	111.4 (4)	C16-C21-C22	107.9 (4)
C7-C8-C9	110.9 (4)	C20-C21-C22	130.6 (5)
C8-C9-C10	112.4 (5)	O4-C22-N2	125.0 (5)
C9-C10-C11	111.1 (5)	O4-C22-C21	129.2 (5)
C10-C11-C12	112.2 (5)	N2-C22-C21	105.8 (4)
C7-C12-C11	111.0 (4)	O1···N1···O2'	126.1 (2)

Symmetry transformation ('): 2 - *x*, 2 - *y*, 1 - *z*.

the cation across an inversion center by short hydrogen bonds (2.733(5), 2.783(5) Å). In the ammonium counterion, the alkyl groups which adopt chair conformations subtend an angle of 117.9 (3)° at the nitrogen atom; the angle has been opened up from the idealized tetrahedral angle of 109.5° in response to the steric bulk of the cyclohexyl rings. The carbon-nitrogen-carbon angle as well as the nitrogen-oxygen interactions are similar to those found in other dicyclohexylammonium [1,2, 12] derivatives.

In the carboxylato anion, the negative charge is delocalized over the -CO_2 entity as the two carbon-oxygen bond distances are equal (1.244(5), 1.245(5) Å). The anion retains the basic conformation adopted by the parent acid, which crystallizes as a monohydrate. The carbon-oxygen single-bond distance (1.322 (6) Å) exceeds the double-bond distance (1.196 (7) Å) in the acid; the water molecule in the parent acid hydrate acts as a hydrogen-bond donor to the imido oxygen atoms [13]. Bond dimensions of the phthalimido portion of the *N*-phthaloylglycinato anion of the dicyclohexylammonium derivative compare well with those of phthalimide [14].

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