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Diphenylguanidinium hydrogen oxalate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.088
wR factor = 0.199
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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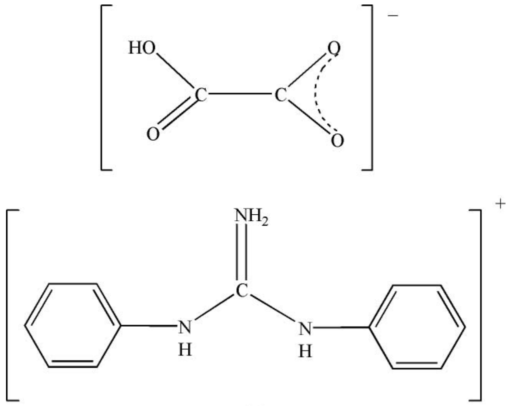
In the crystal structure of the title compound, $C_{13}H_{14}N_3^+\cdot C_2HO_4^-$, each cation is linked to four adjacent anions by intermolecular N—H···O hydrogen bonds [N···O = 2.870 (4)–2.895 (4) Å], and each anion is also linked to two adjacent anions by O—H···O hydrogen bonds [O···O = 2.526 (3) Å] to form a two-dimensional grid network parallel to (100).

Comment

Directional intermolecular interactions are the primary tools for assembling intriguing supramolecular structures, and hydrogen bonding is currently the most efficient (Zaworotko, 1997; Braga & Grepioni, 2000). To extend this research, we were interested in the hydrogen bonding in the crystal structure of the title compound, $C_{15}H_{15}N_3O_4$, (I), which we report here.



(I)

In the title compound (Fig. 1), the O3—C15 bond length [1.295 (4) Å] differs significantly from the O4—C15 bond length [1.210 (4) Å], indicating this carboxyl group is not deprotonated (Table 1). In the anion, the C15/O3/H3/O4 carboxyl group, with an r.m.s. deviation from planarity of 0.0143 Å, forms a dihedral angle of 31.7 (3)° with the other carboxylate group (C14/O1/O2). In the diphenylguanidinium cation, one phenyl ring (C1–C6), with an r.m.s. deviation from planarity of 0.0030 Å, forms a dihedral angle of 14.1 (3)° with the other phenyl ring (C8–C13) (r.m.s. deviation from planarity of 0.0078 Å). In the crystal structure, each cation is linked to four adjacent anions by intermolecular N—H···O hydrogen bonds, and each anion is also linked to two adjacent anions by O—H···O hydrogen bonds to form a two-dimensional grid network parallel to (100) (Fig. 2 and Table 2). The hydrogen-bonding pattern, as shown in Fig. 2, contains the

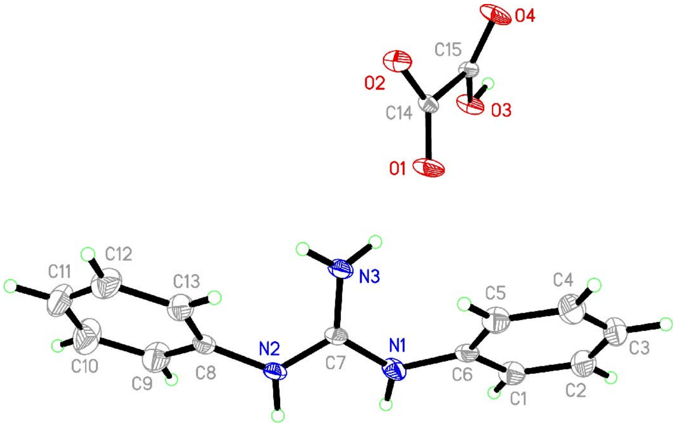


Figure 1
The structure of (I), with the atom numbering, showing displacement ellipsoids at the 30% probability level.

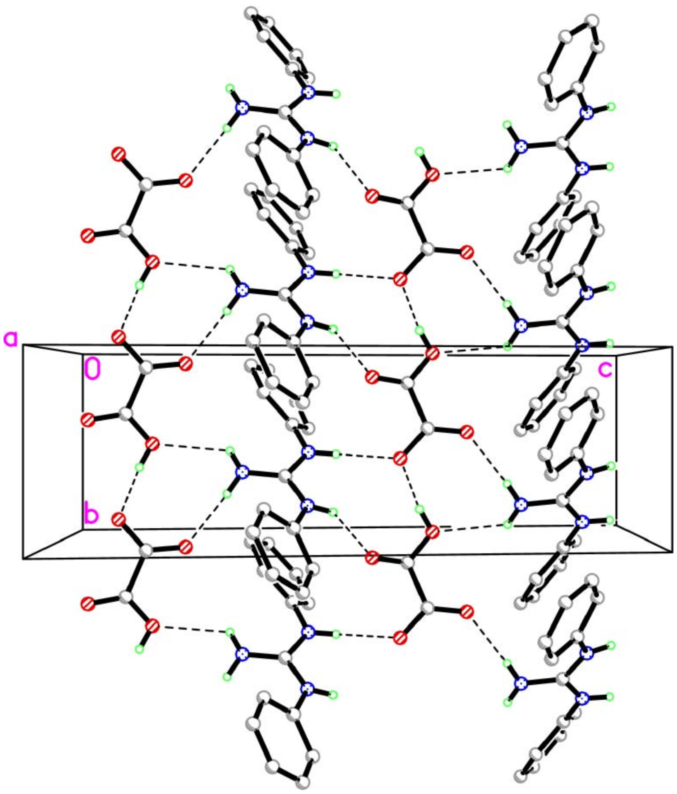


Figure 2
The grid network formed by hydrogen-bonding interactions, which are shown as dashed lines. H atoms not involved in these interactions have been omitted for clarity.

graph-set motifs (Etter, 1990; Grell *et al.*, 2000) $R_3^3(8)$, $R_3^3(10)$ and $R_4^4(18)$.

Experimental

An ethanol solution (10 ml) of diphenylguanidine (0.1 mmol, 0.21) was added dropwise to a stirred aqueous solution (10 ml) of oxalic acid (0.2 mmol, 0.18 g) at 253 K. The reaction mixture was filtered and the filtrate was allowed to stand for approximately two weeks until colorless single crystals formed.

Crystal data

$C_{13}H_{14}N_3^+\cdot C_2HO_4^-$
$M_r = 301.30$
Monoclinic, $P2_1/c$
$a = 15.315$ (7) Å
$b = 5.573$ (3) Å
$c = 17.487$ (8) Å
$\beta = 99.863$ (9)°
$V = 1470.3$ (12) Å^3
$Z = 4$

$D_x = 1.361$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1357 reflections
$\theta = 2.4$–24.4°
$\mu = 0.10$ mm^{-1}
$T = 298$ (2) K
Plate, colorless
0.39 × 0.23 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.962$, $T_{\max} = 0.992$
7305 measured reflections

2640 independent reflections
2271 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 25.2°$
$h = -16 \rightarrow 18$
$k = -6 \rightarrow 6$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.088$
$wR(F^2) = 0.199$
$S = 1.32$
2640 reflections
200 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 1.2264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C14	1.225 (4)	N1—C7	1.338 (4)
O2—C14	1.262 (4)	N2—C7	1.324 (4)
O3—C15	1.295 (4)	N3—C7	1.321 (4)
O4—C15	1.210 (4)		
N3—C7—N2	122.3 (3)	O1—C14—O2	127.2 (3)
N3—C7—N1	120.5 (3)	O4—C15—O3	125.1 (3)
N2—C7—N1	117.2 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3*B*···O1	0.86	2.05	2.878 (4)	161
N3—H3*C*···O3$^{\rm i}$	0.86	2.39	2.895 (4)	118
N2—H2···O2$^{\rm ii}$	0.86	2.03	2.875 (4)	168
N1—H1···O4$^{\rm iii}$	0.86	2.11	2.870 (4)	148
O3—H3···O2$^{\rm iv}$	0.82	1.72	2.526 (3)	167

Symmetry codes: (i) $x, y+1, z$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (iv) $x, y-1, z$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms at C—H distances of 0.93 Å with $U_{\rm iso} = 1.2U_{\rm eq}$(parent atom), N—H distances of 0.86 Å with $U_{\rm iso} = 1.2U_{\rm eq}$(parent atom), and O—H distances of 0.82 Å with $U_{\rm iso} = 1.2U_{\rm eq}$(parent atom).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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