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2,2′-Iminobis(ethylammonium) phthalate

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Key indicators

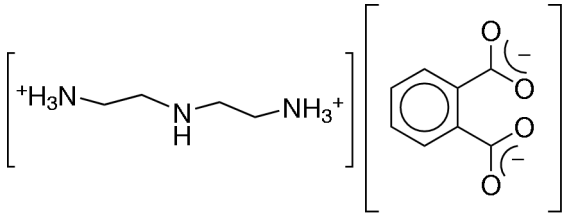
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.119
Data-to-parameter ratio = 11.1

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

The phthalate salt of diprotonated diethylenetriamine, $C_4H_{15}N_3^{+}\cdot C_8H_4O_4^{-}$, was obtained during ongoing studies of the hydrolytic chemistry of phthalimides for the synthesis of efficient sensitizers for Eu^{III} and Tb^{III} photoluminescence.

Comment

The phthalate salt $[NH_3(CH_2)_2NH(CH_2)_2NH_3][1,2\text{-}C_6H_4(COO)_2]$, (I), was obtained in the course of studies of the hydrolytic processes of phthalimides to phthalates through phthalamate intermediates which are of interest as efficient sensitizers for Eu^{III} and Tb^{III} photoluminescence (Barrett *et al.*, 1995, 1998). The structure consists of sheets of cations sandwiched between two sheets of anions. There is extensive hydrogen bonding between the cations and anions.



(I)

Experimental

Compound (I) was obtained by heating a mixture of 3.5 mmol of phthalic anhydride with 1.9 mmol of diethylenetriamine in an oil bath until the mixture had formed a uniform brown melt. After the melt solidified, it was ground to a powder and dissolved in aqueous ethanol from which the product crystallized. Analysis calculated for $C_{12}H_{19}N_3O_4$: C 53.1, H 7.1, N 15.6%; found: C 53.6, H 7.1, N 15.6%.

Crystal data

$C_4H_{15}N_3^{+}\cdot C_8H_4O_4^{-}$
M_r = 269.30
Monoclinic, $P2_1/c$
a = 11.7634 (8) Å
b = 11.025 (1) Å
c = 12.0322 (7) Å
β = 115.378 (1)°
V = 1409.0 (2) Å^3
Z = 4
D_x = 1.269 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 15.1–23.2°
μ = 0.10 mm^{-1}
T = 293 (2) K
Plate, colourless
0.53 × 0.40 × 0.13 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
2335 measured reflections
2218 independent reflections
1371 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 24.0°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 12$
$l = -13 \rightarrow 12$
2 standard reflections
frequency: 120 min
intensity decay: 3.4%

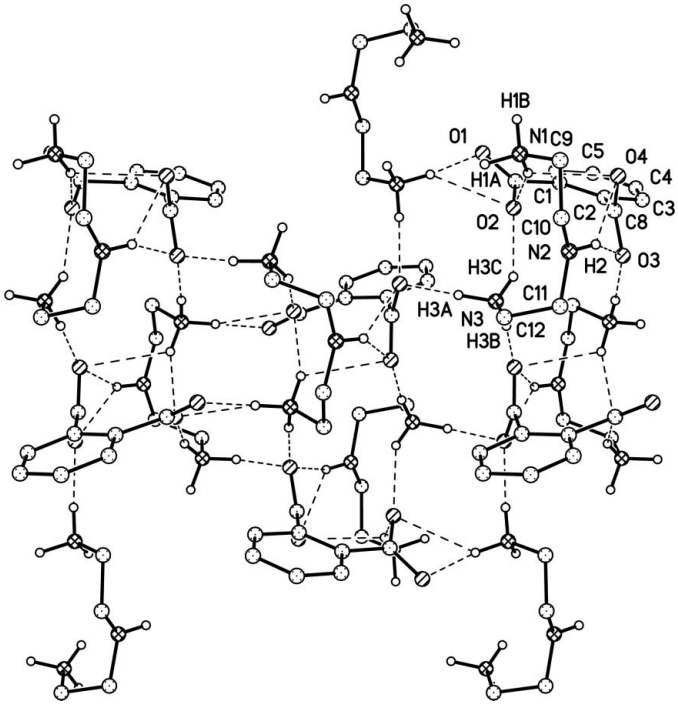


Figure 1
A portion of the hydrogen-bonded network in (I).

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.119$
$S = 1.02$
2218 reflections
200 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.1498P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	1.01 (3)	1.75 (3)	2.755 (3)	171 (2)
N1—H1A···O2^{i}	1.01 (3)	2.53 (3)	3.271 (3)	129.9 (19)
N1—H1B···O3^{ii}	0.95 (3)	1.88 (4)	2.826 (3)	176 (3)
N1—H1C···O2	0.95 (3)	2.06 (3)	2.954 (3)	156 (2)
N1—H1C···O4	0.95 (3)	2.56 (3)	3.151 (3)	121 (2)
N2—H2···O3	0.83 (3)	2.38 (3)	3.042 (3)	137 (2)
N2—H2···O4	0.83 (3)	2.51 (3)	3.143 (3)	134 (2)
N3—H3A···O3^{iii}	1.03 (3)	1.73 (4)	2.752 (3)	170 (3)
N3—H3B···O4^{iv}	0.96 (3)	1.81 (3)	2.759 (3)	169 (2)
N3—H3C···O2	0.82 (3)	2.19 (3)	2.890 (3)	143 (3)

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

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *XCAD*4 (Harms & Wocadlo, 1987); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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