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Ethylenediammonium phthalate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C—C) = 0.003 Å
R factor = 0.039
wR factor = 0.108
Data-to-parameter ratio = 11.4

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

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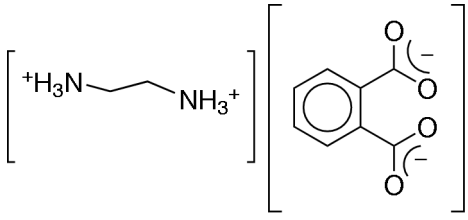
The phthalate salt of diprotonated ethylenediamine, $C_2H_{10}N_2^{+}\cdot C_8H_4O_4^{-}$, was obtained during ongoing studies of the hydrolytic chemistry of phthalimides for the synthesis of efficient sensitizers for Eu^{III} and Tb^{III} photoluminescence.

Comment

The phthalate salt $[NH_3(CH_2)_2NH_3][1,2\text{-}C_6H_4(COO)_2]$, (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

The title compound was prepared by heating 13.5 mmol of phthalic anhydride in 30 ml of pyridine to 363 K and then adding 65 mmol of ethylenediamine dissolved in 10 ml of pyridine. Deposition of a white solid occurred immediately and the resulting mixture was refluxed for 24 h. The white solid was then filtered off and washed with acetone and dichloromethane. Hydrolysis of the material thus obtained in wet dimethyl sulfoxide and subsequent crystallization yielded rectangular crystals. Analysis calculated for $C_{10}H_{14}N_2O_4$: C 55.0, H 5.1, N 7.1%; found: C 55.0, H 5.0, N 7.1%.

Crystal data

$C_2H_{10}N_2^{+}\cdot C_8H_4O_4^{-}$
M_r = 226.23
Orthorhombic, *Pbca*
a = 21.825 (3) Å
b = 12.384 (1) Å
c = 8.048 (1) Å
V = 2175.2 (5) Å^3
Z = 8
D_x = 1.382 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 23 reflections
θ = 11.7–16.0°
μ = 0.11 mm^{-1}
T = 293 (2) K
Plate, colourless
0.40 × 0.30 × 0.13 mm

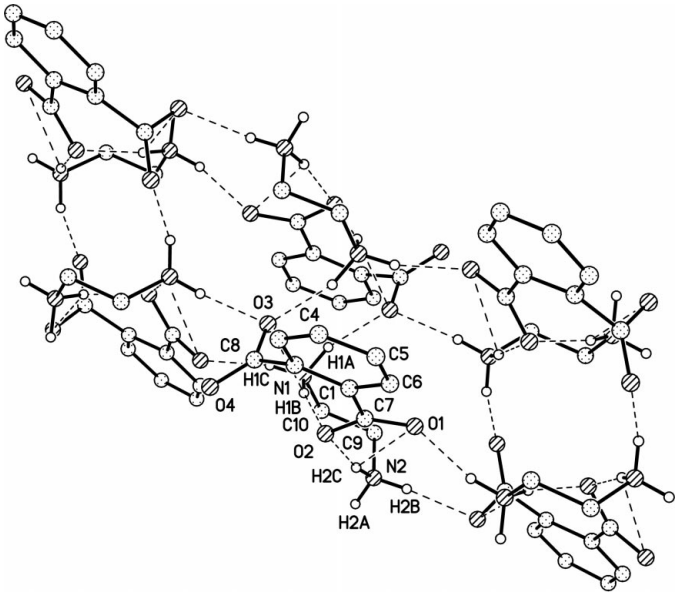


Figure 1
A portion of the hydrogen-bonded network in (I).

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
1934 measured reflections
1934 independent reflections
1187 reflections with $I > 2\sigma(I)$
$\theta_{max} = 25.1°$
$h = 0 \rightarrow 26$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 9$
2 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.108$
$S = 1.01$
1934 reflections
169 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.1370P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.98 (3)	1.85 (3)	2.802 (3)	161 (2)
N1—H1B···O2^{ii}	0.95 (3)	1.87 (3)	2.788 (2)	163 (2)
N1—H1C···O1^{iii}	0.93 (3)	1.85 (3)	2.765 (3)	166 (3)
N2—H2A···O4	0.95 (2)	1.89 (3)	2.776 (3)	155 (2)
N2—H2B···O3^{iv}	0.90 (3)	1.94 (3)	2.835 (2)	173 (2)
N2—H2C···O2^{ii}	1.04 (3)	1.74 (3)	2.766 (2)	166 (2)

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

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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