

Bis(diisopropylammonium)
2,6-naphthalenedicarboxylate

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Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.059

wR factor = 0.141

Data-to-parameter ratio = 19.1

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

In the title compound, $[\{(\text{CH}_3)_2\text{CH}\}_2\text{NH}_2]_2 [\text{C}_{10}\text{H}_6\text{-2,6-}(\text{CO}_2)_2]$, two ammonium $-\text{NH}_2$ groups interact with two carboxyl $-\text{CO}_2$ groups around an inversion center to give rise to a ten-membered $\leftarrow\text{O}-\text{C}-\text{O}\cdots\text{H}-\text{N}-\text{H}\cdots\text{O}-\text{C}-\text{O}\cdots\text{H}-\text{N}-\text{H}\leftarrow$ ring. As the dicarboxylate dianion also lies on an inversion center, a linear hydrogen-bonded chain structure is formed.

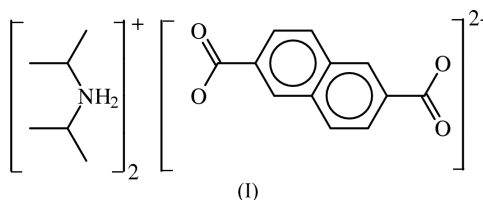
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Comment

Dicyclohexylammonium monocarboxylates typically adopt an arrangement in which two $[\text{R}_2\text{NH}_2]^+$ cations are linked by hydrogen bonds to two $[\text{R}'\text{CO}_2]^-$ anions across a center of inversion, the arrangement giving rise to a ten-membered $\leftarrow\text{O}-\text{C}-\text{O}\cdots\text{H}-\text{N}-\text{H}\cdots\text{O}-\text{C}-\text{O}\cdots\text{H}-\text{N}-\text{H}\leftarrow$ ring (Ng, 1993, 1995, 1996, 1997; Ng & Hook, 1999; Subramanian *et al.*, 2000; Ng *et al.*, 2001); an exception is the trifluoroacetate, which adopts a chain structure (Ng *et al.*, 1999). For the corresponding dicarboxylic acid salts, only the adipate has been characterized, but the ammonium dicarboxylate crystallizes as a hydrate (Yang *et al.*, 2000), and it does not display the expected twin ten-membered-ring motif.



This motif is now documented in the bis(diisopropylammonium) salt of 2,6-naphthalenedicarboxylic acid, (I) (Fig. 1); the crystal structure of the parent acid is known from powder-diffraction measurements (Kaduk & Golab, 1999). $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds lead to the formation of a chain structure (Fig. 2 and Table 2) as the flat dicarboxylate dianion lies on an inversion center.

Experimental

Diisopropylamine (1 g, 10 mmol) was weighed into a beaker, and ethanol (20 ml) was poured into it. 2,6-Naphthalenedicarboxylic acid (1.1 g, 5 mmol) was added to the solution. The mixture was heated until the solid material that had formed dissolved completely. The solvent was allowed to evaporate over several days to furnish a tan-colored syrup from which the desired colorless product separated as parallelepiped-shaped crystals.

Crystal data

$2C_6H_{16}N^+ \cdot C_{12}H_6O_4^{2-}$
 $M_r = 418.56$
 Triclinic, $P\bar{1}$
 $a = 7.8718$ (7) Å
 $b = 8.5597$ (7) Å
 $c = 10.1327$ (9) Å
 $\alpha = 67.749$ (1)°
 $\beta = 88.179$ (1)°
 $\gamma = 75.389$ (1)°
 $V = 609.92$ (9) Å³

$Z = 1$
 $D_x = 1.140$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 1895
 reflections
 $\theta = 2.2$ – 28.3 °
 $\mu = 0.08$ mm⁻¹
 $T = 298$ (2) K
 Parallelepiped, colorless
 $0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker AXS area-detector
 diffractometer
 φ and ω scans
 Absorption correction: none
 5309 measured reflections
 2756 independent reflections

1084 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.045$
 $\theta_{max} = 28.3$ °
 $h = -10 \rightarrow 10$
 $k = -11 \rightarrow 10$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.059$
 $wR(F^2) = 0.141$
 $S = 0.72$
 2756 reflections
 144 parameters

H atoms treated by a mixture of
 independent and constrained
 refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0622P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.18$ e Å⁻³
 $\Delta\rho_{min} = -0.22$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.245 (3)	C4—C6 ⁱ	1.396 (3)
O2—C1	1.248 (2)	C4—C4 ⁱ	1.416 (4)
N1—C7	1.495 (3)	C5—C6	1.364 (3)
N1—C10	1.491 (3)	C6—C4 ⁱ	1.396 (3)
C1—C2	1.509 (3)	C7—C8	1.504 (3)
C2—C3	1.349 (3)	C7—C9	1.505 (3)
C2—C5	1.402 (3)	C10—C11	1.505 (3)
C3—C4	1.424 (3)	C10—C12	1.518 (3)
C7—N1—C10	117.7 (2)	C4 ⁱ —C4—C3	117.7 (2)
O1—C1—O2	125.4 (2)	C6—C5—C2	120.9 (2)
O1—C1—C2	117.5 (2)	C5—C6—C4 ⁱ	121.0 (2)
O2—C1—C2	117.1 (2)	N1—C7—C8	110.9 (2)
C3—C2—C5	119.2 (2)	N1—C7—C9	107.9 (2)
C3—C2—C1	121.4 (2)	C8—C7—C9	112.2 (2)
C5—C2—C1	119.5 (2)	N1—C10—C11	111.4 (2)
C2—C3—C4	122.1 (2)	N1—C10—C12	107.5 (2)
C6 ⁱ —C4—C4 ⁱ	119.2 (2)	C11—C10—C12	111.0 (2)
C6 ⁱ —C4—C3	123.1 (2)		

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1n1 \cdots O1	0.90	1.82	2.716 (2)	173
N1—H1n2 \cdots O2 ⁱ	0.87	1.88	2.746 (2)	169

Symmetry code: (i) $-x, 1 - y, -z$.

The ammonium H atoms were located and refined subject to the restraint $N-H$ 0.86 (1) Å. The aliphatic and aromatic H atoms were positioned geometrically ($C-H$ 0.93 Å for the aromatic H atoms, $C-H$ 0.98 Å for the methine H atom and $C-H$ 0.96 Å for the methyl H atoms), and were included in the refinement in the riding model

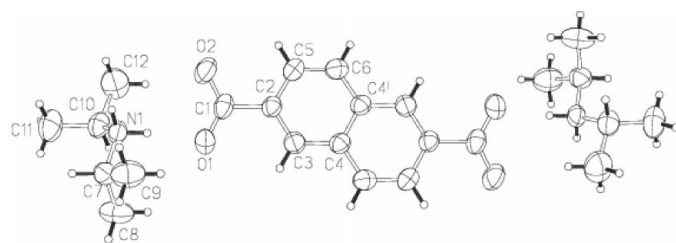


Figure 1

ORTEPII (Johnson, 1976) view of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

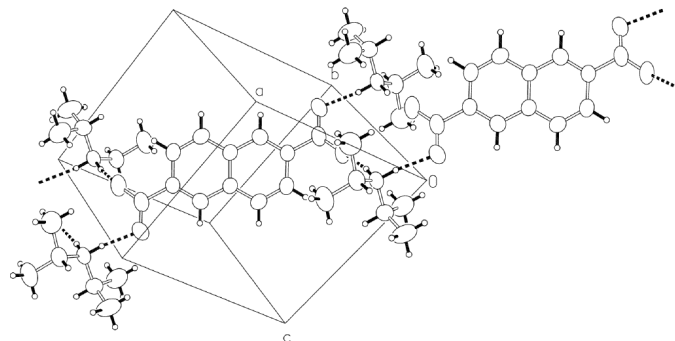


Figure 2

ORTEPII (Johnson, 1976) drawing, illustrating the hydrogen-bonded chain structure.

approximation; their respective displacement factors were set to 1.2, 1.2 and 1.5 times U_{eq} of the parent C atom.

Data collection: SMART (Bruker, 2001); cell refinement: SMART; data reduction: SAINT (Bruker, 2001); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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