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Bis(dimethylammonium) terephthalate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.134
Data-to-parameter ratio = 15.2

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

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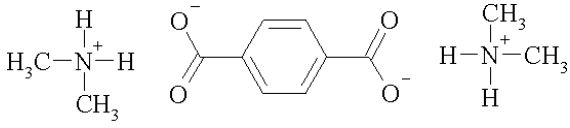
The asymmetric unit of the title compound, $2C_2H_8N^+\cdot C_8H_4O_4^{2-}$, comprises two crystallographically independent dimethylammonium cations and two half-terephthalate anions. The latter are each disposed about an inversion centre. N—H···O hydrogen bonds link the ions into a three-dimensional framework.

Comment

The title compound, (I), was isolated as a side-product in the synthesis of metal-organic framework materials. The asymmetric unit (Fig. 1) comprises two crystallographically independent dimethylammonium cations and halves of two terephthalate anions, the latter each disposed about an inversion centre. Cations and anions are connected by typical N—H···O hydrogen bonds into a three-dimensional framework (Table 1). The structure consists of two independent interpenetrating frameworks, each comprising two kinds of cations and two kinds of anions connected by hydrogen bonds



(I)

Experimental

The typical quantities of reagents were 0.2 mmol neodimium chloride or holmium nitrate and 0.3 mmol of terephthalic acid in 5 ml of DMSO in one vessel and 2 ml of dimethylamine in another vessel. For a typical synthesis of metal-organic framework materials, two DMFA solutions were prepared in glass vessels: a metal salt with terephthalic acid and the other containing only dimethylamine. The former glass vessel had only a small hole, whereas the latter was open. Such a technique is needed for the slow diffusion of amine into the reaction vessel for slow crystallization of metal-organic framework materials. Glass vessels of these solutions were placed into a desiccator held under vacuum. After a few days, colourless needles were formed. These crystals were mounted in a glass capillary under water-free conditions.

Crystal data

$2C_2H_8N^+\cdot C_8H_4O_4^{2-}$
M_r = 256.30
Monoclinic, $P2_1/n$
a = 9.642 (2) Å
b = 11.103 (2) Å
c = 13.272 (3) Å
β = 91.01 (3)°
V = 1420.6 (5) Å^3
Z = 4
D_x = 1.198 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20 reflections
θ = 10–14°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colourless
0.5 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.968, T_{max} = 0.982
2910 measured reflections
2790 independent reflections
1759 reflections with $I > 2\sigma(I)$

R_{int} = 0.050
θ_{max} = 26.0°
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 16$
2 standard reflections
frequency: 7200 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.134
S = 1.05
2790 reflections
184 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2 + 0.1137P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.083 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H5···O3^{i}	0.95 (2)	1.83 (2)	2.761 (3)	167 (2)
N1—H6···O2	0.96 (3)	1.78 (3)	2.731 (3)	171 (2)
N2—H1···O1	0.95 (3)	1.77 (3)	2.700 (3)	165 (2)
N2—H2···O4^{ii}	1.02 (3)	1.68 (3)	2.693 (3)	179 (3)

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

N-bound H atoms, *i.e.* H1, H2, H5 and H6, were located in a difference map and refined isotropically. As the N1—H5 distance was too long under free refinement, it was restrained to 0.95 (2) Å. C-bound H atoms were included in the riding-model approximation, with C—H = 0.93 Å (for benzene) and C—H = 0.96 Å (for methyl), and U_{iso}(H) = 1.2U_{eq}(CH) and U_{iso}(methyl H) = 1.5U_{eq}(CH$_3$).

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Crystal Impact, 2000); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

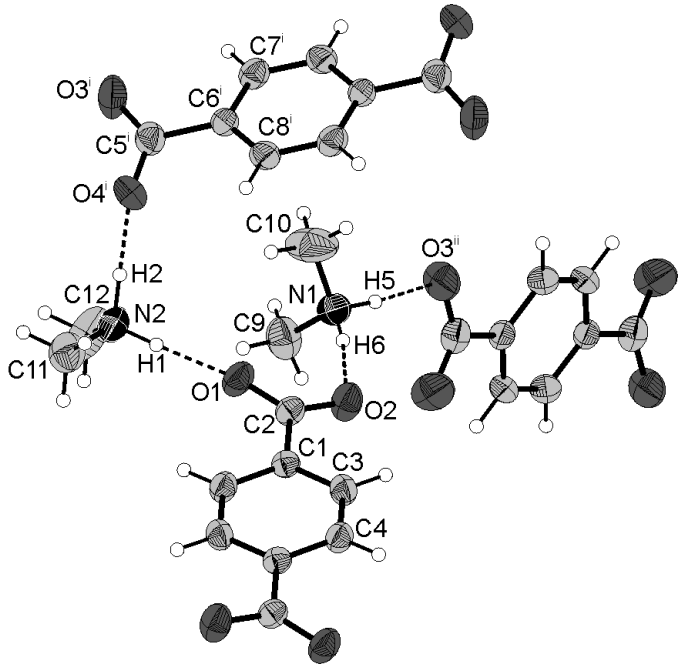


Figure 1
A view of the association between ions in dimethylammonium terephthalate, showing the atomic numbering scheme. Molecules of only one framework are shown. The second framework is hidden for simplicity. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $x + \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $x + 1, y, z$.]

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