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# Dimethylammonium terephthalate

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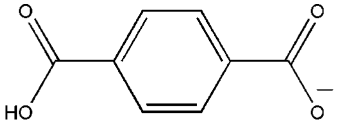
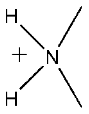
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.047; $wR$ factor = 0.137; data-to-parameter ratio = 18.2.

The asymmetric unit of the title compound, $C_2H_8N^+\cdot C_8H_5O_4^-$, comprises half each of a dimethylammonium cation and a terephthalate anion. The cation lies on a twofold rotation axis, and the anion on an inversion centre. The crystal packing is stabilized by a bifurcated N—H···O hydrogen bond and a linear, apparently symmetric, O—H···O hydrogen bond. Experimental evidence suggests that the latter interaction might be a (disordered) asymmetric hydrogen bond.

## Related literature

For related literature, see: Catti & Ferraris (1976); Cobbledick & Small (1972); Farrugia (1999); Kaduk (2000); Kalsbeek & Larsen (1991).





## Experimental

### Crystal data

$C_2H_8N^+\cdot C_8H_5O_4^-$
$M_r$ = 211.21
Monoclinic, $C2/c$
$a$ = 16.804 (5) Å
$b$ = 7.885 (4) Å
$c$ = 10.484 (5) Å
$\beta$ = 126.161 (5)°
$V$ = 1121.5 (9) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 293 (2) K
0.48 × 0.40 × 0.38 mm

### Data collection

Bruker SMART APEX2 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min}$ = 0.958, $T_{max}$ = 0.966
3305 measured reflections
1345 independent reflections
985 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.036

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.137
$S$ = 1.05
1345 reflections
74 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···O1$^{i}$ | 0.88 (4) | 1.59 (4) | 2.467 (2) | 176 (6) |
| N1—H1*NA*···O2$^{ii}$ | 0.90 | 2.01 | 2.7630 (17) | 140 |
| N1—H1*NA*···O1$^{iii}$ | 0.90 | 2.48 | 3.107 (3) | 127 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2049).

## References

Bruker (2004). *APEX2* (Version 1.08), *SAINT* (Version 7.03) and *SADABS* (Version 2.11). Bruker AXS Inc., Madison, Wisconsin, USA.
Catti, M. & Ferraris, G. (1976). *Acta Cryst.* B**32**, 2754–2756.
Cobbledick, R. E. & Small, R. W. H. (1972). *Acta Cryst.* B**28**, 2924–2928.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Kaduk, J. A. (2000). *Acta Cryst.* B**56**, 474–485.
Kalsbeek, N. & Larsen, S. (1991). *Acta Cryst.* C**47**, 1005–1009.
Sheldrick, G. M. (1990). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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# supplementary materials

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# Dimethylammonium terephthalate

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## Comment

The ionic title compound, (I), was isolated as a side-product in the synthesis of metal-organic framework materials. The asymmetric unit comprises one half-dimethylammonium cation and one half-terephthalate anion. In the terephthalate anions, the two carboxyl groups are essentially coplanar with the phenyl ring.

In addition to the ionic interactions, the crystal structure is held together by a network of two types of hydrogen bonds. The first one is a bifurcated one involving N1—H1NA in the amine anion and both O atoms in the carboxyl group (Table 1). The nature of the second type of hydrogen bond is ambiguous. Certainly there exists a short hydrogen bond interaction between the O1-carboxyl group and its crystallographically equivalent one, the $O1\cdots O1^{i}$ distance of 2.467 (2) Å [symmetry code: (i) $-x + 2$, $-y$, $-z + 2$] falls within the normal range for symmetric hydrogen bonds (Cobbledick & Small, 1972; Catti & Ferraris, 1976), but a slightly better refinement was obtained by allowing the hydrogen to move off the center of symmetry. Two alternative structural models have been studied. Placing atom H1 on the inversion center gives a symmetric structure. Full-matrix least-squares refinement converged to a stable solution. The residual difference Fourier map has a largest peak and hole of 0.214 and -0.189 e $Å^{-3}$, respectively. The final *R* was 0.0474. The crystallographic symmetry constrains the hydrogen-bond angle to 180 ° and the O1—H1 distance to 1.232 (1) Å. A second structural model is one with the half occupied H1-atom site displaced from the inversion center towards O1. A Fourier map section in the O1/C1/O2 plane (MAPVIEW; Farrugia, 1999) showed a broad peak centered at the crystallographic inversion center and slightly elongated (nearly) along the $O\cdots O^{i}$ direction, a fact which might support this latter hypothesis. Free refinement of the *x*, *y*, *z* and $U_{iso}$ parameters for the H1 atom converged to an asymmetric hydrogen-bonding model, which is the one finally reported here, with a largest Fourier peak and hole of 0.212 and -0.176 e $Å^{-3}$ and the final *R* of 0.0470, respectively. This type of hydrogen bonding geometry is closely comparable to what reported in similar structures (Kalsbeek & Larsen, 1991; Kaduk, 2000). Nevertheless, the limited data quality and resolution mean that we cannot unambiguously determine the nature of this hydrogen bonding interaction.

The terephthalate anions link each other by the O—H···O short hydrogen-bond into a 1-D polymeric chain. Then the terephthalate anions and dimethylammonium cations are connected by N—H···O hydrogen-bonds into 3-D supermolecule structure (Table 1).

## Experimental

The title compound (I) was synthesized *via* solvent-thermal method under autogenous pressure. A mixture of terephthalic acid (0.332 g, 2 mmol), zinc acetate dihydrate (0.11 g, 0.5 mmol), $N^1$,$N^1$,$N^2$,$N^2$-tetrakis(pyridin-4-ylmethyl)ethane-1,2-diamine (0.212 g, 0.5 mmol) and DMF 12 ml was sealed in a Teflon-lined stainless steel Parr vessel, which was heated at 453 K for 72 h. After slow cooling to ambient temperature, colorless blockshaped crystals of the title compound were separated mechanically as a side-product. Analysis found: C 56.8, H 6.2, N 6.5%; $C_{10}H_{13}NO_4$ requires: C 56.86, H 6.20, N 6.63%.

### Refinement

The H1 atom was located in difference Fourier maps. the other H atoms were visible in difference Fourier maps but were placed in calculated positions with C—H= 0.93 Å (CH), C—H= 0.96 Å ($CH_3$) and N—H= 0.90 Å ($NH_2$), $U_{iso}$(H)= 1.5 times $U_{eq}$(C) for $CH_3$ and $U_{iso}$(H)= 1.2 times $U_{eq}$(C) or $U_{eq}$(N) for CH or $NH_2$. All other non-H atoms were refined anisotropically. The maximum positive peak of 0.212 e $Å^{-3}$ in the final difference electron density map is located 0.74 Å from atom C3.

### Figures

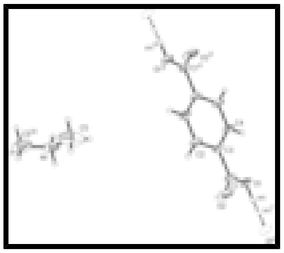

Fig. 1. A view of of (I) with the atomic labeling scheme. Displacement ellipsoids are shown at the 30% probability level. The $O1\cdots O1^{i}$ interaction is presented as an asymmetric, disordered H-bond, drawn in broken lines. Symmetry codes: (i) -*x* + 2, -*y*, -*z* + 2; (ii) 1.5 - *x*, 0.5 - *y*, 1 - *z*], (iii) - *x*, *y*, 0.5 - *z*].

### Dimethylammonium terephthalate

*Crystal data*

$C_2H_8N^+\cdot C_8H_5O_4^-$

$M_r = 211.21$

Monoclinic, *C*2/*c*

Hall symbol: -C 2yc

$a = 16.804$ (5) Å

$b = 7.885$ (4) Å

$c = 10.484$ (5) Å

$\beta = 126.161$ (5)°

$V = 1121.5$ (9) $Å^3$

$Z = 4$

$F_{000} = 448$

$D_x = 1.251$ Mg $m^{-3}$

Mo *K*α radiation
λ = 0.71069 Å

Cell parameters from 588 reflections

θ = 3.6–22.8°

$\mu = 0.10$ $mm^{-1}$

*T* = 293 (2) K

Block, colourless

0.48 × 0.40 × 0.38 mm

*Data collection*

Bruker SMART APEX2 CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

*T* = 293(2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2004)

$T_{min} = 0.958$, $T_{max} = 0.966$

3305 measured reflections

1345 independent reflections

985 reflections with $I > 2\sigma(I)$

$R_{int} = 0.036$

$\theta_{max} = 28.2°$

$\theta_{min} = 3.0°$

$h = -19 \rightarrow 22$

$k = -6 \rightarrow 10$

$l = -13 \rightarrow 13$

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H atoms treated by a mixture of independent and constrained refinement |
| $R[F^2 > 2\sigma(F^2)] = 0.047$ | $w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.4384P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.137$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.05$ | $\Delta\rho_{max} = 0.21$ e Å$^{-3}$ |
| 1345 reflections | $\Delta\rho_{min} = -0.18$ e Å$^{-3}$ |
| 74 parameters | Extinction correction: none |
| 1 restraint | |
| Primary atom site location: structure-invariant direct methods | |
| Secondary atom site location: difference Fourier map | |

### *Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

### *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.88542 (10) | 0.1522 (2) | 0.82777 (15) | 0.0429 (4) | |
| C2 | 0.73871 (12) | 0.3105 (2) | 0.61160 (16) | 0.0528 (5) | |
| H2 | 0.7306 | 0.3520 | 0.6863 | 0.063* | |
| C3 | 0.81474 (9) | 0.20024 (19) | 0.65670 (14) | 0.0390 (4) | |
| C4 | 0.82574 (12) | 0.1401 (2) | 0.54521 (17) | 0.0532 (5) | |
| H4 | 0.8769 | 0.0656 | 0.5750 | 0.064* | |
| C5 | 0.0816 (3) | 0.3853 (4) | 0.2722 (3) | 0.1501 (16) | |
| H5A | 0.0569 | 0.4584 | 0.1830 | 0.225* | |
| H5B | 0.1292 | 0.3086 | 0.2817 | 0.225* | |
| H5C | 0.1121 | 0.4524 | 0.3665 | 0.225* | |
| H1 | 0.983 (4) | 0.011 (8) | 0.960 (5) | 0.082 (15)* | 0.50 |
| N1 | 0.0000 | 0.2885 (3) | 0.2500 | 0.0755 (8) | |
| H1NA | 0.0247 | 0.2208 | 0.3346 | 0.091* | 0.50 |
| H1NB | −0.0247 | 0.2208 | 0.1654 | 0.091* | 0.50 |
| O1 | 0.94143 (8) | 0.02566 (16) | 0.85670 (11) | 0.0537 (4) | |

| | | | | |
|---|---|---|---|---|
| O2 | 0.88617 (10) | 0.23126 (18) | 0.92810 (12) | 0.0756 (5) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0390 (7) | 0.0493 (8) | 0.0237 (6) | 0.0042 (6) | 0.0094 (5) | 0.0071 (6) |
| C2 | 0.0541 (9) | 0.0687 (11) | 0.0268 (7) | 0.0241 (8) | 0.0190 (7) | 0.0103 (7) |
| C3 | 0.0337 (7) | 0.0454 (8) | 0.0231 (6) | 0.0054 (6) | 0.0085 (5) | 0.0078 (5) |
| C4 | 0.0488 (9) | 0.0680 (11) | 0.0295 (7) | 0.0285 (8) | 0.0158 (6) | 0.0145 (7) |
| C5 | 0.212 (4) | 0.136 (3) | 0.0814 (19) | −0.100 (3) | 0.075 (2) | −0.0276 (17) |
| N1 | 0.118 (2) | 0.0552 (13) | 0.0380 (10) | 0.000 | 0.0376 (12) | 0.000 |
| O1 | 0.0473 (6) | 0.0667 (8) | 0.0266 (5) | 0.0200 (5) | 0.0103 (5) | 0.0140 (5) |
| O2 | 0.0919 (10) | 0.0798 (10) | 0.0252 (5) | 0.0354 (8) | 0.0181 (6) | 0.0076 (5) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—O2 | 1.2164 (19) | C5—N1 | 1.462 (3) |
| C1—O1 | 1.2791 (19) | C5—H5A | 0.9600 |
| C1—C3 | 1.5024 (18) | C5—H5B | 0.9600 |
| C2—C3 | 1.378 (2) | C5—H5C | 0.9600 |
| C2—C4$^{i}$ | 1.3877 (19) | N1—H1NA | 0.9000 |
| C2—H2 | 0.9300 | N1—H1NB | 0.9000 |
| C3—C4 | 1.371 (2) | O1—H1 | 0.88 (4) |
| C4—H4 | 0.9300 | | |
| O2—C1—O1 | 124.34 (12) | N1—C5—H5B | 109.5 |
| O2—C1—C3 | 120.03 (14) | H5A—C5—H5B | 109.5 |
| O1—C1—C3 | 115.63 (13) | N1—C5—H5C | 109.5 |
| C3—C2—C4$^{i}$ | 120.05 (14) | H5A—C5—H5C | 109.5 |
| C3—C2—H2 | 120.0 | H5B—C5—H5C | 109.5 |
| C4$^{i}$—C2—H2 | 120.0 | C5—N1—C5$^{ii}$ | 117.1 (4) |
| C4—C3—C2 | 119.32 (12) | C5—N1—H1NA | 108.0 |
| C4—C3—C1 | 121.42 (13) | C5$^{ii}$—N1—H1NA | 108.0 |
| C2—C3—C1 | 119.24 (13) | C5—N1—H1NB | 108.0 |
| C3—C4—C2$^{i}$ | 120.63 (14) | C5$^{ii}$—N1—H1NB | 108.0 |
| C3—C4—H4 | 119.7 | H1NA—N1—H1NB | 107.3 |
| C2$^{i}$—C4—H4 | 119.7 | C1—O1—H1 | 109 (4) |
| N1—C5—H5A | 109.5 | | |
| C4$^{i}$—C2—C3—C4 | 0.0 (3) | O2—C1—C3—C2 | −12.7 (2) |
| C4$^{i}$—C2—C3—C1 | 178.31 (15) | O1—C1—C3—C2 | 166.73 (15) |
| O2—C1—C3—C4 | 165.59 (17) | C2—C3—C4—C2$^{i}$ | 0.0 (3) |
| O1—C1—C3—C4 | −14.9 (2) | C1—C3—C4—C2$^{i}$ | −178.28 (15) |

Symmetry codes: (i) $-x+3/2, -y+1/2, -z+1$; (ii) $-x, y, -z+1/2$.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| O1—H1···O1$^{iii}$ | 0.88 (4) | 1.59 (4) | 2.467 (2) | 176 (6) |
| N1—H1NA···O2$^{iv}$ | 0.90 | 2.01 | 2.7630 (17) | 140 |
| N1—H1NA···O1$^{v}$ | 0.90 | 2.48 | 3.107 (3) | 127 |

Symmetry codes: (iii) −*x*+2, −*y*, −*z*+2; (iv) −*x*+1, *y*, −*z*+3/2; (v) *x*−1, −*y*, *z*−1/2.

**Fig. 1**