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Key indicators

Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.034
 wR factor = 0.095
Data-to-parameter ratio = 12.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Tetramethylammonium hydrogen 4,5-dichlorophthalate

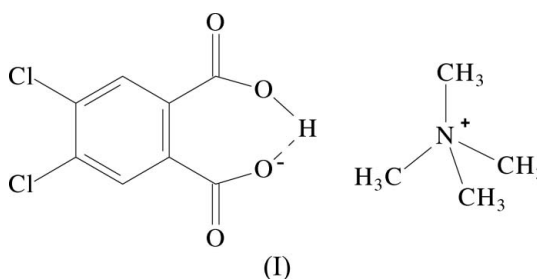
In the title compound, $\text{C}_4\text{H}_{12}^+\cdot\text{C}_8\text{H}_3\text{Cl}_2\text{O}_4^-$, the hydrogen 4,5-dichlorophthalate ions are linked to the tetramethylammonium ions through $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, generating two edge-fused motifs, viz. $R_2^1(6)$ and $S(7)R_1^2(7)$. The cation and all atoms of the anion lie on a mirror plane.

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Comment

Tetramethylammonium salts are of general interest in preparative chemistry. Their enhanced solubility in aprotic solvents in comparison to the corresponding alkali metal salts, as well as their higher reactivity due to a lower degree of solvation of their anions in solutions in non-polar solvents, make them a powerful and versatile tool (Malchus & Jansen, 1998). We have been interested in hydrogen-bonding systems formed by organic amines and carboxylic acids (Büyükgüngör & Odabaşoğlu, 2002, 2003, 2006*a,b*; Odabaşoğlu, Büyükgüngör & Lönnecke, 2003; Odabaşoğlu, Büyükgüngör, Turgut *et al.*, 2003; Odabaşoğlu & Büyükgüngör, 2006*a,b,c*). The present work is part of a structural study of compounds of organic ammonium systems with hydrogen-bond donors and we report here the structure of the title compound, (I) (Fig. 1 and Table 1).



A mirror plane passes through atoms N1, C10 and C11 of the cation and all atoms of the anion. The tetramethylammonium ions are linked to the hydrogen 4,5-dichlorophthalate ions, which have an $S(7)$ motif, through $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2), forming two edge-fused $R_2^1(6)$ motifs (Etter, 1990). These motifs are connected by two $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, generating $R_1^2(7)S(7)$ motifs (Fig. 2). The two $R_2^1(6)S(7)R_1^2(7)$ motifs generate a three-dimensional network by $\text{C9}-\text{H9B}\cdots\text{O1}^{\text{iv}}$ and $\text{C9}-\text{H9C}\cdots\text{O4}^{\text{iii}}$ hydrogen bonds (Fig. 3). There are no specific aromatic $\pi-\pi$ interactions between the aromatic rings in (I).

Experimental

The title compound, (I), was prepared by mixing 4,5-dichlorophthalic acid and tetramethylammonium hydroxide in a 1:1 molar ratio in

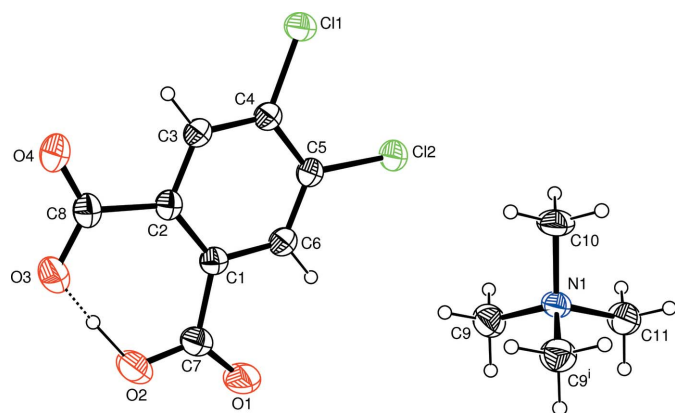


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates the intramolecular O—H...O hydrogen bond. [Symmetry code: (i) $x, -y + \frac{1}{2}, z$].

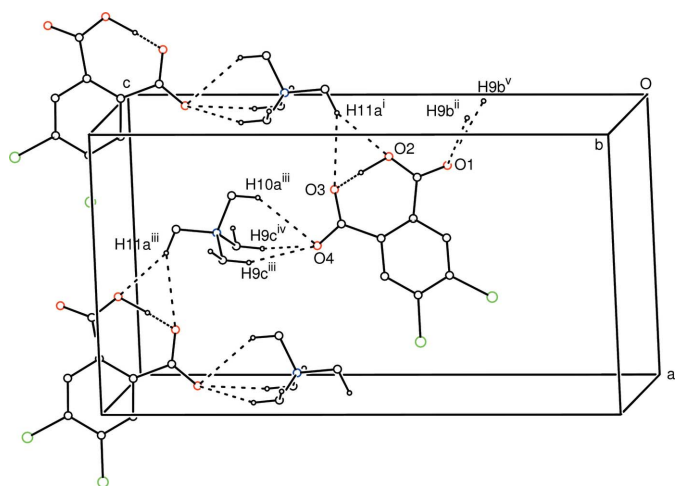


Figure 2
Part of the crystal structure of (I), showing the formation of $R_2^2(6)S(7)R_2^2(6)$ motifs. Dashed lines indicate hydrogen bonds. [Symmetry codes: (i) $x - \frac{1}{2}, -y, z + \frac{1}{2}$; (ii) $x + \frac{1}{2}, y + \frac{1}{2}, z$; (iii) $1 - x, -y, z + \frac{1}{2}$; (iv) $x, -y, z + \frac{1}{2}$; (v) $x + \frac{1}{2}, y, z$].

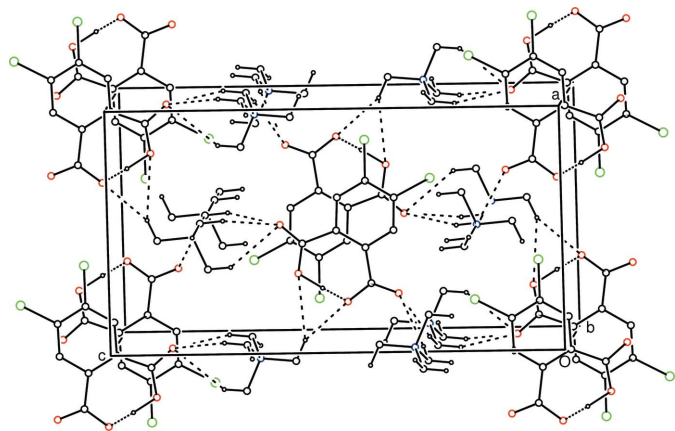


Figure 3
A packing diagram of (I), showing the hydrogen-bonding scheme and C—H...O interactions as dashed lines. H atoms not involved in inter- and intramolecular interactions have been omitted for clarity.

50 ml methanol at 323–333 K for 15 min. Crystals of (I) were obtained by slow evaporation of the solvent (m.p. 493–495 K).

Crystal data

$C_4H_{12}N^+ \cdot C_8H_3Cl_2O_4^-$
 $M_r = 308.15$
Orthorhombic, $Pnma$
 $a = 10.2800$ (6) Å
 $b = 6.9120$ (3) Å
 $c = 19.0486$ (10) Å
 $V = 1353.51$ (12) Å³

$Z = 4$
 $D_x = 1.512$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.49$ mm⁻¹
 $T = 296$ K
Rod, colourless
 $0.66 \times 0.35 \times 0.17$ mm

Data collection

Stoe IPDS-2 diffractometer
 ω scans
Absorption correction: integration
(*X-RED32*; Stoe & Cie, 2002)
 $T_{\min} = 0.783$, $T_{\max} = 0.931$

18362 measured reflections
1449 independent reflections
1239 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.065$
 $\theta_{\max} = 26.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.095$
 $S = 1.03$
1449 reflections
116 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + 1 + 0.2573P + (0.0562P)^2]$
where $P = 0.33F_o^2 + 0.67F_c^2$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.27$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³
Extinction correction: *SHELXL97*
Extinction coefficient: 0.026 (4)

Table 1

Selected geometric parameters (Å, °).

C1—C2	1.410 (3)	C7—O2	1.258 (3)
C1—C7	1.528 (3)	C8—O4	1.215 (3)
C2—C8	1.529 (3)	C8—O3	1.278 (3)
C7—O1	1.212 (3)	C9—N1	1.483 (2)
C9 ⁱ —N1—C9	109.2 (2)	C9 ⁱ —N1—C10	109.69 (13)
C9 ⁱ —N1—C11	109.68 (13)	C11—N1—C10	108.86 (19)
C6—C1—C7—O1	0.0	C3—C2—C8—O4	0.0

Symmetry code: (i) $x, -y - \frac{1}{2}, z$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O2—H3A...O3	1.18 (5)	1.20 (5)	2.380 (3)	177 (4)
C11—H11A...O2 ⁱⁱ	0.96	2.44	3.337 (3)	156
C11—H11A...O3 ⁱⁱ	0.96	2.82	3.691 (3)	151
C10—H10A...O4 ⁱⁱⁱ	0.96	2.75	3.571 (4)	145
C9—H9C...O4 ⁱⁱⁱ	0.96	2.60	3.466 (3)	150
C9—H9B...O1 ^{iv}	0.96	2.76	3.659 (3)	157

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

All C-bound H atoms were refined using the riding-model approximation, with C—H = 0.93 Å for aromatic and 0.96 Å for methyl H atoms [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{methyl C})$]. The O-bound H atom was located in a difference Fourier map and refined freely.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s)

used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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