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Tetrakis(*tert*-butylammonium) benzene-1,2,4,5-tetracarboxylate octahydrate

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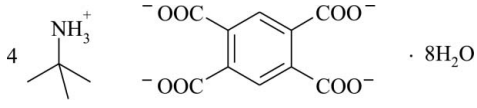
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.108; data-to-parameter ratio = 12.8.

In the crystal structure of the title compound, $4C_4H_{12}N^+\cdot C_{10}H_2O_8^{4-}\cdot 8H_2O$, there is a centre of symmetry at the centre of the benzene ring; the asymmetric unit comprises one half-anion, two cations and four water molecules. The pyromellitate tetraanion is nonplanar; it and the cations exhibit normal geometry. The two unique carboxylate groups are twisted out of the plane of the benzene ring by about 40 and 50°. The network formed by the ions and water molecules is based on eight O—H···O and six N—H···O strong hydrogen bonds.

Related literature

For related literature, see: Arora & Pedireddi (2003); Bergstrom *et al.* (2000); Su *et al.* (2001); Adams & Ramdas (1979); Kinbara *et al.* (1996); Sada *et al.* (2004); Nagahama *et al.* 2003); Wang *et al.* (2005); Ejsmont & Zaleski (2006*a*,*b*); Steiner (2002).



Experimental

Crystal data

$4C_4H_{12}N^+\cdot C_{10}H_2O_8^{4-}\cdot 8H_2O$
M_r = 690.83
Monoclinic, $P2_1/c$
a = 10.2536 (13) Å
b = 10.6867 (14) Å
c = 18.213 (2) Å
β = 100.612 (11)°
V = 1961.6 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (1) K
0.40 × 0.35 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur diffractometer
Absorption correction: none
11870 measured reflections
3450 independent reflections
2640 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
S = 1.06
3450 reflections
269 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected torsion angles (°).

C12—C11—C14—O1	−134.78 (14)	C11—C12—C15—O4	38.47 (19)
C12—C11—C14—O2	49.96 (18)	C13—C12—C15—O3	40.48 (18)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O1^{i}	0.96 (2)	1.82 (2)	2.7626 (16)	166.2 (17)
O5—H5B···O1	0.88 (2)	1.93 (2)	2.7885 (15)	168 (2)
O6—H6A···O3^{ii}	0.89 (2)	1.78 (2)	2.6606 (17)	171.9 (19)
O6—H6B···O5^{iii}	0.93 (3)	1.87 (3)	2.7994 (17)	178 (2)
O7—H7A···O2	0.92 (3)	1.75 (3)	2.6635 (15)	170 (2)
O7—H7B···O3^{iv}	0.92 (2)	1.80 (2)	2.7122 (16)	168.9 (18)
O8—H8A···O7	0.87 (3)	1.92 (3)	2.788 (2)	179 (2)
O8—H8B···O5	0.91 (3)	2.00 (3)	2.8469 (19)	156 (2)
N9—H9A···O8^{v}	0.95 (2)	1.89 (2)	2.813 (2)	164.3 (16)
N9—H9B···O7^{iii}	0.99 (2)	1.86 (2)	2.8223 (18)	164.2 (16)
N9—H9C···O6	0.947 (19)	1.88 (2)	2.8240 (19)	175.9 (16)
N10—H10A···O2^{ii}	0.947 (18)	1.868 (18)	2.8115 (17)	173.6 (15)
N10—H10B···O4	0.95 (2)	1.84 (2)	2.7805 (17)	176.2 (15)
N10—H10C···O6	0.95 (2)	2.00 (2)	2.9429 (18)	173.0 (17)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2206).

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supplementary materials

Acta Cryst. (2007). E**63**, o4315 [**doi:10.1107/S1600536807049690**]

Tetrakis(*tert*-butylammonium) benzene-1,2,4,5-tetracarboxylate octahydrate

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Comment

Supramolecular hydrogen-bonded assemblies of pyromellitic acid and ammonium (Bergstrom *et al.*, 2000) and organic amines have been examined by single-crystal X-ray diffraction and other techniques (Su *et al.*, 2001; Adams & Ramdas, 1979; Wang *et al.*, 2005; Ejsmont & Zaleski 2006*b*).

We report here the preparation and structural characterization of the title compound, which is an organic salt of pyromellitic acid and *tert*-butylamine. The crystal structure consists of *tert*-butylammonium cations, centrosymmetric pyromelitate(4-) tetra-anions and water molecules. The geometry of the *tert*-butylammonium cations is normal and compares well with those found in other crystal structures which include this cation (Kinbara *et al.*, 1996; Sada *et al.*, 2004; Nagahama *et al.*, 2003; Ejsmont & Zaleski 2006*a*). The centrosymmetric pyromellitate(4-) anion is not planar. The dihedral angles between the benzene ring and the C14/O1/O2 and C15/O3/O4 carboxylate groups are 53.06 (9) and 40.2 (1)°, respectively. The geometrical parameters for the pyromellitate anion agree well with corresponding values found in crystal structures containing these units (*e.g.* Arora & Pedireddi, 2003; Adams & Ramdas, 1979; Bergstrom *et al.*, 2000; Ejsmont & Zaleski 2006*b*; Su *et al.*, 2001; Wang *et al.*, 2005).

In the crystal structure, there are eight O—H···O and six N—H···O hydrogen bonds (Fig. 1 and 2) between *tert*-butylammonium cations, pyromelitate(4-) tetra-anions and water molecules; these interactions are very strong (Steiner, 2002).

Experimental

Crystals of the title compound were grown by slow evaporation of an aqueous solution containing *tert*-butylamine and pyromellitic acid in a 4:1 stoichiometric ratio at room temperature.

Refinement

H atoms bonded to O, N and C_{ar} were located in a difference map and freely refined. O—H = 0.87 (3) − 0.96 (2) Å; N—H = 0.95 (2) − 0.99 (2) Å; C_{ar}—H = 0.96 (2) Å. Methyl H atoms were positioned geometrically and refined as riding with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(methyl C).

Figures

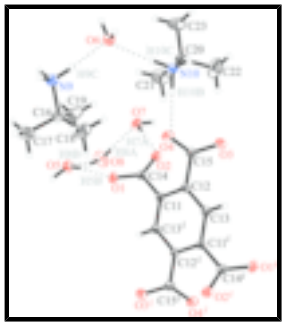

Fig. 1. The molecular structure of the title compound, showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $2-x$, $1-y$, $-z$].

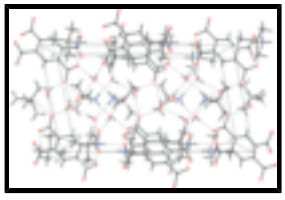

Fig. 2. The packing diagram of the title compound. Dashed lines indicate hydrogen bonds.

Tetrakis(*tert*-butylammonium) benzene-1,2,4,5-tetracarboxylate octahydrate

Crystal data

$4C_4H_{12}N^+\cdot C_{10}H_2O_8^{4-}\cdot 8H_2O$	F_{000} = 756
M_r = 690.83	D_x = 1.170 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3450 reflections
a = 10.2536 (13) Å	θ = 3.3–25.0º
b = 10.6867 (14) Å	μ = 0.10 mm^{-1}
c = 18.213 (2) Å	T = 298 (1) K
β = 100.612 (11)º	Cube, colourless
V = 1961.6 (4) Å^3	0.40 × 0.35 × 0.20 mm
Z = 2	

Data collection

Oxford Diffraction Xcalibur diffractometer	2640 reflections with $I > 2\sigma(I)$
Radiation source: Enhance (Mo) X-ray Source	R_{int} = 0.039
Monochromator: graphite	θ_{max} = 25.0º
T = 298(1) K	θ_{min} = 3.3º
ω scans	h = −9→12
Absorption correction: none	k = −12→12
11870 measured reflections	l = −21→20
3450 independent reflections	

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
$R[F^2 > 2\sigma(F^2)] = 0.038$	$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.109$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
3450 reflections	$\Delta\rho_{min} = -0.20$ e Å^{-3}
269 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.020 (2)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > 2$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}
O1	0.67535 (9)	0.52249 (10)	0.05853 (6)	0.0391 (3)
O2	0.83267 (10)	0.59772 (10)	0.14832 (6)	0.0389 (3)
O3	1.15184 (10)	0.38914 (11)	0.18681 (6)	0.0446 (3)
O4	0.94210 (10)	0.32573 (11)	0.16479 (6)	0.0441 (3)
O5	0.42524 (12)	0.61807 (11)	0.06639 (7)	0.0412 (3)
O6	0.70600 (12)	0.09759 (12)	0.31228 (7)	0.0421 (3)
O7	0.68835 (11)	0.71383 (11)	0.23518 (7)	0.0404 (3)
O8	0.49939 (17)	0.85094 (13)	0.13795 (10)	0.0695 (5)
N9	0.49549 (14)	0.09957 (13)	0.18813 (8)	0.0365 (3)
N10	0.94341 (14)	0.25188 (13)	0.31131 (8)	0.0337 (3)
C11	0.89899 (12)	0.51179 (12)	0.04206 (7)	0.0233 (3)
C12	1.01639 (12)	0.44950 (12)	0.07267 (7)	0.0241 (3)
C13	1.11574 (13)	0.44063 (13)	0.03004 (8)	0.0256 (3)
C14	0.79351 (13)	0.54353 (13)	0.08678 (8)	0.0262 (3)
C15	1.03681 (13)	0.38363 (13)	0.14770 (8)	0.0287 (3)

C16	0.51441 (16)	0.18556 (16)	0.12506 (9)	0.0436 (4)
C17	0.3934 (2)	0.1708 (2)	0.06386 (12)	0.0757 (7)
H17A	0.3892	0.0865	0.0453	0.114*
H17B	0.3995	0.2277	0.0239	0.114*
H17C	0.3149	0.1888	0.0836	0.114*
C18	0.6396 (2)	0.1463 (2)	0.09859 (13)	0.0690 (6)
H18A	0.7143	0.1567	0.1384	0.103*
H18B	0.6513	0.1972	0.0569	0.103*
H18C	0.6326	0.0600	0.0837	0.103*
C19	0.5257 (2)	0.31708 (18)	0.15633 (14)	0.0803 (7)
H19A	0.6031	0.3232	0.1948	0.120*
H19B	0.4482	0.3362	0.1769	0.120*
H19C	0.5328	0.3753	0.1171	0.120*
C20	0.94694 (15)	0.36444 (15)	0.36155 (9)	0.0399 (4)
C21	0.8410 (2)	0.4540 (2)	0.32339 (12)	0.0776 (7)
H21A	0.8619	0.4802	0.2765	0.116*
H21B	0.8376	0.5258	0.3547	0.116*
H21C	0.7564	0.4126	0.3148	0.116*
C22	1.0831 (2)	0.4206 (2)	0.36966 (15)	0.0862 (8)
H22A	1.1477	0.3619	0.3941	0.129*
H22B	1.0872	0.4957	0.3989	0.129*
H22C	1.1015	0.4402	0.3211	0.129*
C23	0.9167 (2)	0.3219 (2)	0.43570 (10)	0.0664 (6)
H23A	0.9850	0.2659	0.4592	0.100*
H23B	0.8327	0.2796	0.4278	0.100*
H23C	0.9132	0.3933	0.4673	0.100*
H5A	0.3778 (19)	0.5768 (18)	0.0226 (12)	0.059 (6)*
H5B	0.507 (2)	0.5916 (19)	0.0704 (12)	0.074 (7)*
H6A	0.751 (2)	0.026 (2)	0.3165 (11)	0.071 (7)*
H6B	0.660 (2)	0.104 (2)	0.3518 (14)	0.087 (8)*
H7A	0.737 (2)	0.666 (2)	0.2081 (15)	0.105 (8)*
H7B	0.751 (2)	0.7683 (19)	0.2594 (11)	0.062 (6)*
H8A	0.558 (2)	0.807 (3)	0.1686 (14)	0.091 (8)*
H8B	0.458 (2)	0.791 (3)	0.1067 (14)	0.095 (8)*
H9A	0.4809 (18)	0.015 (2)	0.1731 (11)	0.072 (6)*
H9B	0.4187 (19)	0.1320 (17)	0.2084 (11)	0.059 (5)*
H9C	0.5688 (18)	0.1004 (15)	0.2283 (11)	0.048 (5)*
H10A	1.0180 (17)	0.2004 (17)	0.3286 (9)	0.049 (5)*
H10B	0.9470 (16)	0.2775 (16)	0.2620 (12)	0.051 (5)*
H10C	0.8628 (19)	0.2081 (18)	0.3101 (10)	0.060 (6)*
H13	1.1952 (16)	0.3948 (14)	0.0502 (9)	0.039 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0223 (6)	0.0613 (7)	0.0355 (6)	−0.0029 (5)	0.0098 (4)	−0.0080 (5)
O2	0.0332 (6)	0.0546 (7)	0.0324 (6)	−0.0075 (5)	0.0151 (5)	−0.0156 (5)
O3	0.0352 (6)	0.0600 (8)	0.0353 (6)	−0.0029 (5)	−0.0024 (5)	0.0185 (5)

O4	0.0426 (6)	0.0577 (8)	0.0334 (6)	−0.0121 (5)	0.0106 (5)	0.0134 (5)
O5	0.0348 (7)	0.0478 (7)	0.0420 (7)	0.0020 (5)	0.0095 (5)	−0.0114 (5)
O6	0.0402 (7)	0.0474 (7)	0.0402 (7)	0.0031 (5)	0.0109 (5)	0.0062 (5)
O7	0.0346 (6)	0.0460 (7)	0.0437 (7)	−0.0025 (5)	0.0154 (5)	−0.0113 (5)
O8	0.0800 (11)	0.0405 (8)	0.0778 (11)	0.0127 (7)	−0.0123 (8)	−0.0113 (8)
N9	0.0369 (8)	0.0351 (8)	0.0379 (8)	0.0023 (6)	0.0078 (7)	0.0033 (6)
N10	0.0317 (7)	0.0408 (8)	0.0300 (7)	0.0017 (6)	0.0091 (6)	0.0057 (6)
C11	0.0220 (7)	0.0277 (7)	0.0210 (7)	−0.0022 (5)	0.0064 (5)	−0.0022 (6)
C12	0.0244 (7)	0.0281 (7)	0.0199 (7)	−0.0018 (5)	0.0046 (5)	−0.0006 (6)
C13	0.0228 (7)	0.0301 (7)	0.0241 (7)	0.0034 (6)	0.0045 (6)	0.0010 (6)
C14	0.0253 (7)	0.0302 (8)	0.0249 (7)	−0.0013 (6)	0.0092 (6)	0.0000 (6)
C15	0.0310 (8)	0.0322 (8)	0.0236 (7)	0.0021 (6)	0.0070 (6)	0.0015 (6)
C16	0.0457 (10)	0.0438 (10)	0.0457 (10)	0.0074 (7)	0.0203 (8)	0.0141 (8)
C17	0.0710 (14)	0.1013 (18)	0.0539 (13)	0.0199 (12)	0.0093 (11)	0.0291 (12)
C18	0.0605 (13)	0.0804 (14)	0.0755 (15)	0.0137 (10)	0.0375 (11)	0.0119 (12)
C19	0.1085 (18)	0.0387 (11)	0.1109 (19)	−0.0014 (11)	0.0656 (16)	0.0128 (12)
C20	0.0452 (9)	0.0413 (9)	0.0354 (9)	0.0053 (7)	0.0131 (7)	0.0007 (7)
C21	0.1051 (18)	0.0670 (14)	0.0625 (14)	0.0419 (13)	0.0202 (12)	0.0100 (12)
C22	0.0731 (15)	0.0911 (17)	0.1015 (19)	−0.0340 (13)	0.0346 (14)	−0.0485 (15)
C23	0.0989 (16)	0.0653 (13)	0.0395 (11)	0.0107 (11)	0.0249 (11)	0.0012 (10)

Geometric parameters (Å, °)

O1—C14	1.2464 (16)	C13—H13	0.964 (16)
O2—C14	1.2598 (17)	C16—C18	1.511 (2)
O3—C15	1.2619 (17)	C16—C19	1.513 (3)
O4—C15	1.2386 (17)	C16—C17	1.516 (3)
O5—H5A	0.96 (2)	C17—H17A	0.9600
O5—H5B	0.88 (2)	C17—H17B	0.9600
O6—H6A	0.89 (2)	C17—H17C	0.9600
O6—H6B	0.93 (3)	C18—H18A	0.9600
O7—H7A	0.92 (3)	C18—H18B	0.9600
O7—H7B	0.92 (2)	C18—H18C	0.9600
O8—H8A	0.87 (3)	C19—H19A	0.9600
O8—H8B	0.91 (3)	C19—H19B	0.9600
N9—C16	1.511 (2)	C19—H19C	0.9600
N9—H9A	0.95 (2)	C20—C22	1.502 (3)
N9—H9B	0.99 (2)	C20—C23	1.510 (2)
N9—H9C	0.947 (19)	C20—C21	1.517 (2)
N10—C20	1.508 (2)	C21—H21A	0.9600
N10—H10A	0.947 (18)	C21—H21B	0.9600
N10—H10B	0.95 (2)	C21—H21C	0.9600
N10—H10C	0.95 (2)	C22—H22A	0.9600
C11—C13^{i}	1.3902 (19)	C22—H22B	0.9600
C11—C12	1.3985 (18)	C22—H22C	0.9600
C11—C14	1.5075 (18)	C23—H23A	0.9600
C12—C13	1.3932 (19)	C23—H23B	0.9600
C12—C15	1.5171 (18)	C23—H23C	0.9600
C13—C11^{i}	1.3902 (19)		

H5A—O5—H5B	104.5 (18)	C16—C17—H17C	109.5
H6A—O6—H6B	108.7 (18)	H17A—C17—H17C	109.5
H7A—O7—H7B	102.0 (19)	H17B—C17—H17C	109.5
H8A—O8—H8B	102 (2)	C16—C18—H18A	109.5
C16—N9—H9A	113.4 (12)	C16—C18—H18B	109.5
C16—N9—H9B	107.0 (11)	H18A—C18—H18B	109.5
H9A—N9—H9B	110.3 (15)	C16—C18—H18C	109.5
C16—N9—H9C	112.7 (10)	H18A—C18—H18C	109.5
H9A—N9—H9C	106.9 (15)	H18B—C18—H18C	109.5
H9B—N9—H9C	106.3 (16)	C16—C19—H19A	109.5
C20—N10—H10A	109.5 (10)	C16—C19—H19B	109.5
C20—N10—H10B	110.1 (11)	H19A—C19—H19B	109.5
H10A—N10—H10B	108.3 (14)	C16—C19—H19C	109.5
C20—N10—H10C	109.4 (11)	H19A—C19—H19C	109.5
H10A—N10—H10C	111.6 (16)	H19B—C19—H19C	109.5
H10B—N10—H10C	107.9 (15)	C22—C20—N10	107.26 (14)
$C13^{i}$—C11—C12	119.11 (12)	C22—C20—C23	111.87 (17)
$C13^{i}$—C11—C14	117.65 (12)	N10—C20—C23	108.46 (14)
C12—C11—C14	122.84 (12)	C22—C20—C21	111.55 (18)
C13—C12—C11	118.32 (12)	N10—C20—C21	106.86 (14)
C13—C12—C15	118.47 (12)	C23—C20—C21	110.61 (15)
C11—C12—C15	123.08 (11)	C20—C21—H21A	109.5
$C11^{i}$—C13—C12	122.56 (13)	C20—C21—H21B	109.5
$C11^{i}$—C13—H13	118.9 (10)	H21A—C21—H21B	109.5
C12—C13—H13	118.4 (9)	C20—C21—H21C	109.5
O1—C14—O2	125.20 (12)	H21A—C21—H21C	109.5
O1—C14—C11	118.42 (12)	H21B—C21—H21C	109.5
O2—C14—C11	116.18 (12)	C20—C22—H22A	109.5
O4—C15—O3	125.17 (13)	C20—C22—H22B	109.5
O4—C15—C12	118.27 (12)	H22A—C22—H22B	109.5
O3—C15—C12	116.51 (12)	C20—C22—H22C	109.5
C18—C16—N9	108.00 (14)	H22A—C22—H22C	109.5
C18—C16—C19	111.58 (16)	H22B—C22—H22C	109.5
N9—C16—C19	106.88 (15)	C20—C23—H23A	109.5
C18—C16—C17	111.39 (17)	C20—C23—H23B	109.5
N9—C16—C17	107.07 (14)	H23A—C23—H23B	109.5
C19—C16—C17	111.64 (17)	C20—C23—H23C	109.5
C16—C17—H17A	109.5	H23A—C23—H23C	109.5
C16—C17—H17B	109.5	H23B—C23—H23C	109.5
H17A—C17—H17B	109.5		
$C13^{i}$—C11—C12—C13	1.7 (2)	C12—C11—C14—O1	−134.78 (14)
C14—C11—C12—C13	−170.81 (12)	$C13^{i}$—C11—C14—O2	−122.65 (14)
$C13^{i}$—C11—C12—C15	−173.97 (12)	C12—C11—C14—O2	49.96 (18)
C14—C11—C12—C15	13.5 (2)	C13—C12—C15—O4	−137.18 (14)
C11—C12—C13—$C11^{i}$	−1.8 (2)	C11—C12—C15—O4	38.47 (19)
C15—C12—C13—$C11^{i}$	174.11 (12)	C13—C12—C15—O3	40.48 (18)

C13^{i}—C11—C14—O1	52.61 (18)	C11—C12—C15—O3	−143.86 (13)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5A···O1^{ii}	0.96 (2)	1.82 (2)	2.7626 (16)	166.2 (17)
O5—H5B···O1	0.88 (2)	1.93 (2)	2.7885 (15)	168 (2)
O6—H6A···O3^{iii}	0.89 (2)	1.78 (2)	2.6606 (17)	171.9 (19)
O6—H6B···O5^{iv}	0.93 (3)	1.87 (3)	2.7994 (17)	178 (2)
O7—H7A···O2	0.92 (3)	1.75 (3)	2.6635 (15)	170 (2)
O7—H7B···O3^{v}	0.92 (2)	1.80 (2)	2.7122 (16)	168.9 (18)
O8—H8A···O7	0.87 (3)	1.92 (3)	2.788 (2)	179 (2)
O8—H8B···O5	0.91 (3)	2.00 (3)	2.8469 (19)	156 (2)
N9—H9A···O8^{vi}	0.95 (2)	1.89 (2)	2.813 (2)	164.3 (16)
N9—H9B···O7^{iv}	0.99 (2)	1.86 (2)	2.8223 (18)	164.2 (16)
N9—H9C···O6	0.947 (19)	1.88 (2)	2.8240 (19)	175.9 (16)
N10—H10A···O2^{iii}	0.947 (18)	1.868 (18)	2.8115 (17)	173.6 (15)
N10—H10B···O4	0.95 (2)	1.84 (2)	2.7805 (17)	176.2 (15)
N10—H10C···O6	0.95 (2)	2.00 (2)	2.9429 (18)	173.0 (17)

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

Fig. 1

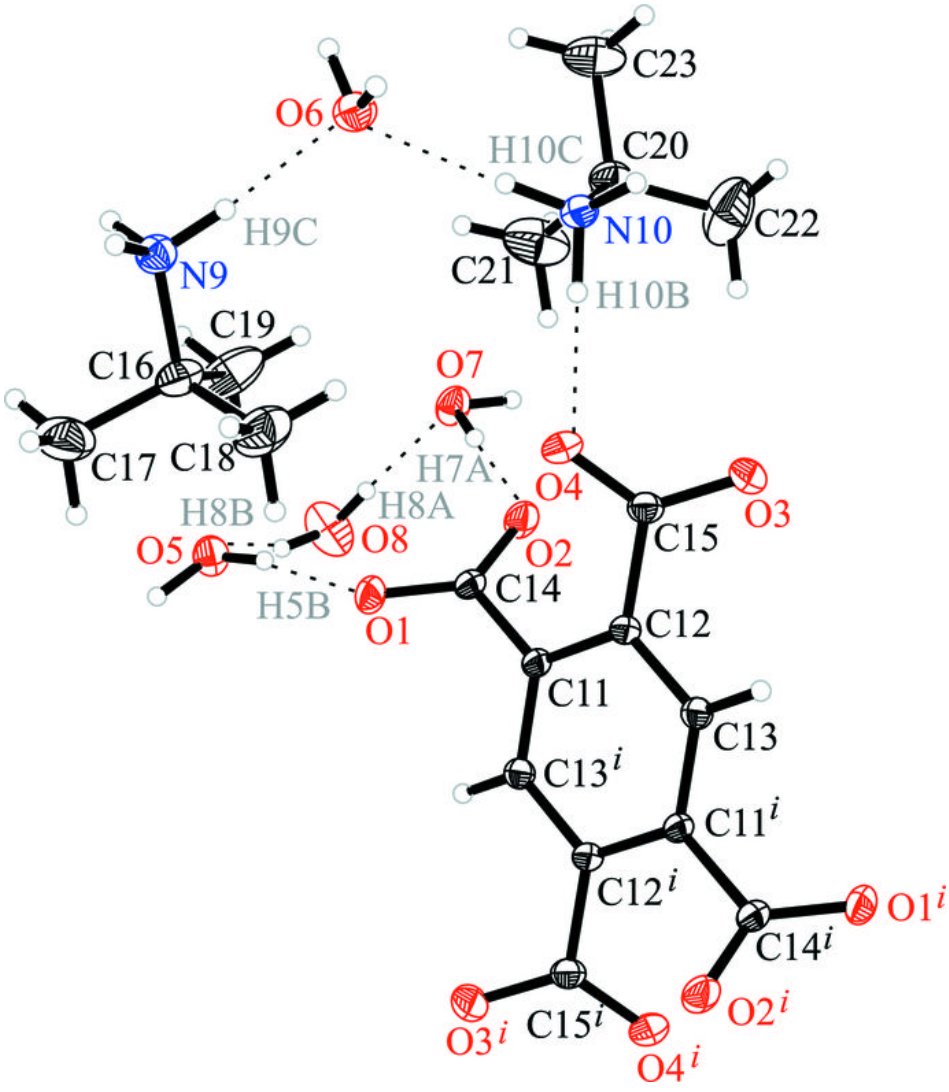
C23
O6
H10C
C20
H9C
N10
C22
N9
C21
H10B
C19
C16
O7
C17
C18
H7A
O4
O3
H8B
H8A
O8
C15
O5
O2
C14
H5B
O1
C12
C11
C13
C13^{i}
C11^{i}
C12^{i}
O1^{i}
C14^{i}
O2^{i}
O3^{i}
C15^{i}
O4^{i}

Fig. 2