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Triethylaminium–trimesate–trimesic acid–water (1/1/1/2)

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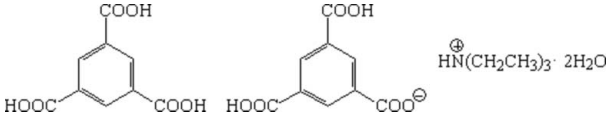
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; disorder in solvent or counterion; R factor = 0.045; wR factor = 0.117; data-to-parameter ratio = 11.6.

The asymmetric unit of the title compound, $C_6H_{16}N^+\cdot C_9H_5O_6^-\cdot C_9H_6O_6\cdot 2H_2O$, consists of a protonated triethylamine cation, one singly deprotonated trimesate anion, one trimesic acid molecule, and two water molecules. The trimesic acid molecules form stacks, while pairs of parallel anions also have π–π interactions; the perpendicular distances between adjacent parallel benzene rings are 3.291 (1), 3.347 (1) and 3.390 (1) Å. Intermolecular hydrogen bonds are found, with all O—H and N—H groups as donors, forming a three-dimensional network.

Related literature

For related structures containing trimesic acid, see: Zaworotko (2001); Duchamp & Marsh (1969); Herbstein *et al.* (1987); Kolotuchin *et al.* (1999); Liu *et al.* (2001).



Experimental

Crystal data

$C_6H_{16}N^+\cdot C_9H_5O_6^-\cdot C_9H_6O_6\cdot 2H_2O$
M_r = 557.50
Triclinic, $P\overline{1}$
a = 7.3762 (10) Å
b = 9.4733 (12) Å
c = 20.754 (3) Å
α = 87.502 (2)°
β = 87.584 (2)°
γ = 67.711 (2)°
V = 1340.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.52 × 0.40 × 0.30 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.938, T_{max} = 0.970
7392 measured reflections
5168 independent reflections
3702 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.117
S = 0.93
5168 reflections
445 parameters
37 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O9	0.88 (2)	1.90 (2)	2.771 (2)	166 (2)
O4—H4*O*···O13^{i}	0.917 (15)	1.651 (16)	2.5653 (18)	174 (2)
O6—H6*O*···O3^{ii}	0.916 (15)	1.703 (16)	2.5895 (18)	162 (2)
O8—H8*O*···O11^{iii}	0.907 (15)	1.697 (15)	2.5993 (19)	173 (2)
O10—H10*O*···O2	0.924 (16)	1.557 (17)	2.4671 (17)	167 (3)
O12—H12*O*···O14^{iv}	0.925 (15)	1.651 (15)	2.5740 (18)	176 (2)
O13—H13*A*···O1	0.901 (14)	1.936 (15)	2.832 (2)	173 (2)
O13—H13*B*···O5^{v}	0.908 (15)	1.951 (15)	2.8577 (18)	176 (3)
O14—H14*A*···O7^{vi}	0.894 (15)	1.935 (16)	2.8141 (19)	167 (3)
O14—H14*B*···O1	0.889 (14)	1.896 (16)	2.7433 (18)	159 (2)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2096).

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supplementary materials

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Triethylaminium-trimesate-trimesic acid-water (1/1/1/2)

J.-M. Chen, J.-J. Sun, W.-W. Huang, Y.-N. Lao and S.-P. Yang

Comment

On account of its robustness and the presence of three exodentate carboxylic groups, trimesic acid is often used in crystal engineering (Zaworotko, 2001). The compound self-assembles into an infinite interpenetrated framework with a honeycomb motif, through carboxylic acid dimer interactions, with the graph set $R_2^2(8)$ (Duchamp & Marsh, 1969). A non-interpenetrated framework can also be synthesized through the inclusion of neutral guests (Herbstein *et al.*, 1987). Extended frameworks of trimesic acid with neutral guests (Kolotuchin *et al.*, 1999; Liu *et al.*, 2001) or organic cations have also been found, but large voids usually involve interpenetration to stabilize the structure (Zaworotko, 2001).

Recently we have isolated the title compound, (I), and report here its preparation and crystal structure. The asymmetric unit consists of a protonated triethylamine cation, one singly deprotonated trimesic acid anion, one neutral trimesic acid molecele, and two water molecules. Fig. 1 shows the asymmetric unit with the atom numbering. A range of intermolecular hydrogen bonds are found (Table 1 and Fig. 2). The two benzene rings of the asymmetric unit are not parallel with each other. The acid molecules form stacks with π···π interactions (Fig. 2); the perpendicular distances in the stacks are 3.291 (1) and 3.347 (1) Å. Pairs of trimesate anions, parallel by inversion symmetry, also have π···π interactions, with a perpendicular distance of 3.390 (1) (Fig. 3).

Experimental

An ethanol solution (10 ml) of trimesic acid (2 mmol, 0.432 g) was mixed with a methanol solution (20 ml) of $ZnCl_2·6H_2O$ (1 mmol, 0.240 g) and triethylamine (1 mmol, 0.10 g). The mixture was stirred for 1 h at room temperature and then filtered. Single crystals of (I) were obtained from the filtrate after 5 d.

Refinement

The cation is disordered, with each methylene group having two alternative sites; the occupancy factors refined to 0.548:0.452 (4). C-bound H atoms were positioned geometrically and refined as riding, with C—H = 0.96 or 0.97 Å and U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C). Other H atoms were located in a difference map. The N-bound H atom was refined freely, while O-bound H atoms were refined with all O—H distances restrained to be equal; the H···H distances in the two water molecules were also restrained to be equal.

Figures

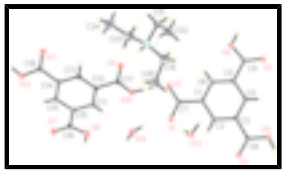

Fig. 1. The asymmetric unit of (I) with 30% probability displacement ellipsoids.

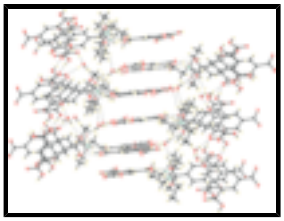

Fig. 2. Perspective view of the packing of the title compound, showing stacks of parallel trimesic acid molecules. Hydrogen bonds are shown as dashed lines.

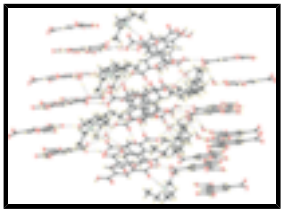

Fig. 3. Perspective view of the packing of the title compound, showing the stacking of parallel pairs of trimesate anions. Hydrogen bonds are shown as dashed lines.

Triethylaminium–trimesate–trimesic acid–water (1/1/1/2)

Crystal data

$C_6H_{16}N^+·C_9H_5O_6^-·C_9H_6O_6·2H_2O$	$Z = 2$
$M_r = 557.50$	$F_{000} = 588$
Triclinic, $P\overline{1}$	$D_x = 1.382$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.3762$ (10) Å	Cell parameters from 2512 reflections
$b = 9.4733$ (12) Å	$\theta = 4.7$–$27.1°$
$c = 20.754$ (3) Å	$\mu = 0.12$ mm^{-1}
$\alpha = 87.502$ (2)°	$T = 298$ (2) K
$\beta = 87.584$ (2)°	Block, colorless
$\gamma = 67.711$ (2)°	0.52 × 0.40 × 0.30 mm
$V = 1340.1$ (3) Å^3	

Data collection

Bruker SMART CCD diffractometer	5168 independent reflections
Radiation source: fine-focus sealed tube	3702 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.062$
$T = 298(2)$ K	$\theta_{max} = 26.0°$
φ and ω scans	$\theta_{min} = 2.0°$
Absorption correction: multi-scan (SADABS; Bruker, 1998)	$h = -9 \rightarrow 9$
$T_{min} = 0.938$, $T_{max} = 0.970$	$k = -11 \rightarrow 11$
7392 measured reflections	$l = -25 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.93$	$(\Delta/\sigma)_{max} < 0.001$
5168 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
445 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
37 restraints	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0096 (16)

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 $F^2 > 2\text{sigma}(F^2)$ 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)
N1	0.6420 (3)	0.7995 (2)	0.24948 (8)	0.0526 (4)	
H1N	0.619 (3)	0.738 (2)	0.2793 (11)	0.065 (7)*	
C19	0.7540 (8)	0.7096 (5)	0.1922 (2)	0.0771 (17)	0.548 (4)
H19A	0.6683	0.6742	0.1692	0.093*	0.548 (4)
H19B	0.7933	0.7763	0.1630	0.093*	0.548 (4)
C20	0.9343 (4)	0.5737 (3)	0.21166 (13)	0.0899 (9)	
H20A	0.9981	0.5191	0.1740	0.135*	0.548 (4)
H20B	0.8964	0.5075	0.2407	0.135*	0.548 (4)
H20C	1.0227	0.6084	0.2326	0.135*	0.548 (4)
H20X	1.0720	0.5410	0.2014	0.135*	0.452 (4)
H20Y	0.8667	0.5652	0.1744	0.135*	0.452 (4)
H20Z	0.9135	0.5107	0.2464	0.135*	0.452 (4)
C21	0.4411 (6)	0.9081 (5)	0.2288 (2)	0.0699 (14)	0.548 (4)
H21A	0.3827	0.9826	0.2619	0.084*	0.548 (4)
H21B	0.4543	0.9624	0.1893	0.084*	0.548 (4)
C22	0.3090 (4)	0.8233 (3)	0.21796 (13)	0.0830 (8)	
H22A	0.1830	0.8945	0.2049	0.124*	0.548 (4)
H22B	0.2942	0.7709	0.2572	0.124*	0.548 (4)
H22C	0.3658	0.7506	0.1848	0.124*	0.548 (4)

H22X	0.2339	0.8178	0.1822	0.124*	0.452 (4)
H22Y	0.2528	0.9234	0.2355	0.124*	0.452 (4)
H22Z	0.3077	0.7487	0.2506	0.124*	0.452 (4)
C23	0.7499 (8)	0.8860 (6)	0.2784 (2)	0.0730 (15)	0.548 (4)
H23A	0.8837	0.8169	0.2858	0.088*	0.548 (4)
H23B	0.7545	0.9649	0.2477	0.088*	0.548 (4)
C24	0.6599 (5)	0.9584 (3)	0.34048 (16)	0.1043 (10)	
H24A	0.7349	1.0128	0.3561	0.156*	0.548 (4)
H24B	0.6592	0.8808	0.3717	0.156*	0.548 (4)
H24C	0.5279	1.0282	0.3335	0.156*	0.548 (4)
H24X	0.6094	1.0617	0.3546	0.156*	0.452 (4)
H24Y	0.8002	0.9222	0.3364	0.156*	0.452 (4)
H24Z	0.6233	0.8951	0.3715	0.156*	0.452 (4)
C19X	0.8584 (8)	0.7341 (7)	0.2313 (3)	0.0764 (19)	0.452 (4)
H19X	0.8826	0.7961	0.1962	0.092*	0.452 (4)
H19Y	0.9315	0.7417	0.2679	0.092*	0.452 (4)
C21X	0.5205 (9)	0.7914 (8)	0.1950 (2)	0.0740 (19)	0.452 (4)
H21X	0.5767	0.6908	0.1769	0.089*	0.452 (4)
H21Y	0.5215	0.8656	0.1614	0.089*	0.452 (4)
C23X	0.5773 (11)	0.9525 (7)	0.2768 (3)	0.084 (2)	0.452 (4)
H23X	0.4354	0.9930	0.2813	0.101*	0.452 (4)
H23Y	0.6127	1.0189	0.2463	0.101*	0.452 (4)
C1	0.5708 (2)	0.30422 (18)	0.13104 (7)	0.0342 (4)	
C2	0.6654 (3)	0.17636 (19)	0.09499 (8)	0.0357 (4)	
H2	0.776 (3)	0.095 (2)	0.1118 (8)	0.037 (5)*	
C3	0.6069 (2)	0.16946 (18)	0.03272 (8)	0.0334 (4)	
C4	0.4541 (2)	0.29113 (19)	0.00659 (8)	0.0342 (4)	
H4	0.415 (2)	0.2824 (19)	−0.0352 (9)	0.041 (5)*	
C5	0.3570 (2)	0.41939 (18)	0.04285 (7)	0.0324 (4)	
C6	0.4151 (3)	0.42441 (19)	0.10511 (8)	0.0344 (4)	
H6	0.354 (3)	0.509 (2)	0.1298 (9)	0.044 (5)*	
C7	0.6419 (3)	0.3127 (2)	0.19719 (8)	0.0400 (4)	
C8	0.7172 (3)	0.02888 (19)	−0.00336 (8)	0.0402 (4)	
C9	0.1953 (2)	0.55055 (19)	0.01377 (8)	0.0361 (4)	
O1	0.80455 (19)	0.21718 (15)	0.21294 (6)	0.0516 (4)	
O2	0.5304 (2)	0.41917 (16)	0.23152 (6)	0.0600 (4)	
O3	0.8326 (2)	−0.08334 (16)	0.02274 (7)	0.0698 (5)	
O4	0.6772 (2)	0.03685 (15)	−0.06430 (6)	0.0553 (4)	
H4O	0.752 (3)	−0.054 (2)	−0.0827 (12)	0.094 (9)*	
O5	0.14604 (18)	0.55484 (14)	−0.04160 (6)	0.0466 (3)	
O6	0.1127 (2)	0.66205 (14)	0.05408 (6)	0.0522 (4)	
H6O	0.012 (3)	0.742 (2)	0.0357 (11)	0.076 (7)*	
C10	0.7878 (2)	0.25947 (18)	0.53374 (8)	0.0361 (4)	
C11	0.7343 (3)	0.2969 (2)	0.46974 (8)	0.0366 (4)	
H11	0.714 (2)	0.2227 (19)	0.4464 (8)	0.038 (5)*	
C12	0.7067 (2)	0.44105 (18)	0.44425 (7)	0.0331 (4)	
C13	0.7304 (2)	0.5473 (2)	0.48242 (8)	0.0338 (4)	
H13	0.713 (2)	0.649 (2)	0.4658 (8)	0.037 (5)*	
C14	0.7848 (2)	0.51107 (18)	0.54653 (8)	0.0332 (4)	

C15	0.8140 (2)	0.36655 (19)	0.57152 (8)	0.0350 (4)
H15	0.845 (3)	0.344 (2)	0.6155 (9)	0.044 (5)*
C16	0.8095 (3)	0.1082 (2)	0.56246 (9)	0.0439 (4)
C17	0.6458 (3)	0.4833 (2)	0.37579 (8)	0.0381 (4)
C18	0.8045 (3)	0.6313 (2)	0.58549 (8)	0.0391 (4)
O7	0.8630 (2)	0.06852 (15)	0.61700 (6)	0.0634 (4)
O8	0.7639 (3)	0.02296 (16)	0.52346 (7)	0.0686 (5)
H8O	0.771 (3)	−0.069 (2)	0.5401 (11)	0.088 (8)*
O9	0.6254 (2)	0.60924 (16)	0.35290 (6)	0.0559 (4)
O10	0.6160 (2)	0.37868 (15)	0.34633 (6)	0.0569 (4)
H10O	0.593 (4)	0.403 (3)	0.3030 (8)	0.104 (9)*
O11	0.7628 (2)	0.76037 (15)	0.56492 (7)	0.0643 (4)
O12	0.8720 (2)	0.58578 (15)	0.64318 (6)	0.0531 (4)
H12O	0.889 (3)	0.668 (2)	0.6609 (10)	0.072 (7)*
O13	1.1077 (2)	0.20607 (16)	0.12245 (7)	0.0526 (4)
H13A	1.013 (3)	0.201 (3)	0.1503 (10)	0.071 (7)*
H13B	1.029 (3)	0.285 (2)	0.0976 (11)	0.099 (9)*
O14	1.0765 (2)	0.19261 (16)	0.30336 (7)	0.0539 (4)
H14A	1.076 (4)	0.115 (3)	0.3294 (12)	0.111 (10)*
H14B	0.967 (2)	0.209 (2)	0.2825 (10)	0.069 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0631 (12)	0.0497 (10)	0.0427 (10)	−0.0195 (9)	−0.0050 (8)	0.0116 (8)
C19	0.103 (4)	0.074 (3)	0.044 (3)	−0.023 (3)	0.009 (3)	0.003 (2)
C20	0.0819 (19)	0.091 (2)	0.0786 (19)	−0.0140 (16)	0.0173 (15)	−0.0045 (16)
C21	0.066 (3)	0.057 (3)	0.074 (3)	−0.010 (2)	−0.020 (2)	0.014 (2)
C22	0.0768 (18)	0.0886 (19)	0.0835 (18)	−0.0305 (15)	−0.0244 (15)	0.0113 (15)
C23	0.077 (4)	0.065 (3)	0.082 (3)	−0.033 (3)	−0.001 (3)	0.000 (2)
C24	0.117 (3)	0.095 (2)	0.114 (3)	−0.051 (2)	−0.013 (2)	−0.0345 (19)
C19X	0.067 (4)	0.088 (4)	0.077 (4)	−0.035 (3)	−0.005 (3)	0.014 (3)
C21X	0.085 (4)	0.090 (5)	0.045 (3)	−0.031 (4)	−0.022 (3)	0.017 (3)
C23X	0.085 (5)	0.063 (4)	0.105 (5)	−0.027 (4)	−0.006 (4)	−0.001 (3)
C1	0.0424 (10)	0.0340 (9)	0.0245 (8)	−0.0120 (8)	−0.0072 (7)	0.0004 (7)
C2	0.0410 (10)	0.0313 (9)	0.0302 (9)	−0.0078 (8)	−0.0110 (7)	0.0027 (7)
C3	0.0400 (10)	0.0288 (9)	0.0295 (8)	−0.0101 (7)	−0.0074 (7)	−0.0018 (7)
C4	0.0409 (10)	0.0345 (9)	0.0257 (9)	−0.0116 (8)	−0.0100 (7)	0.0003 (7)
C5	0.0362 (9)	0.0307 (9)	0.0286 (8)	−0.0106 (7)	−0.0065 (7)	0.0021 (7)
C6	0.0386 (10)	0.0327 (9)	0.0284 (9)	−0.0090 (8)	−0.0033 (7)	−0.0032 (7)
C7	0.0520 (11)	0.0408 (10)	0.0235 (8)	−0.0128 (9)	−0.0088 (8)	0.0006 (7)
C8	0.0443 (10)	0.0333 (10)	0.0363 (10)	−0.0055 (8)	−0.0107 (8)	−0.0053 (8)
C9	0.0386 (10)	0.0345 (9)	0.0330 (9)	−0.0111 (8)	−0.0059 (7)	0.0027 (7)
O1	0.0567 (8)	0.0528 (8)	0.0326 (7)	−0.0043 (7)	−0.0203 (6)	−0.0019 (6)
O2	0.0721 (10)	0.0593 (9)	0.0278 (7)	0.0010 (7)	−0.0125 (6)	−0.0115 (6)
O3	0.0803 (11)	0.0413 (8)	0.0567 (9)	0.0155 (8)	−0.0264 (8)	−0.0118 (7)
O4	0.0695 (9)	0.0453 (8)	0.0351 (7)	−0.0012 (7)	−0.0117 (6)	−0.0127 (6)
O5	0.0498 (8)	0.0461 (7)	0.0347 (7)	−0.0073 (6)	−0.0150 (6)	0.0066 (6)

O6	0.0570 (9)	0.0360 (7)	0.0460 (8)	0.0037 (7)	−0.0132 (7)	−0.0027 (6)
C10	0.0399 (10)	0.0330 (9)	0.0315 (9)	−0.0094 (8)	−0.0063 (7)	0.0025 (7)
C11	0.0455 (10)	0.0338 (9)	0.0294 (9)	−0.0131 (8)	−0.0049 (7)	−0.0041 (7)
C12	0.0363 (9)	0.0365 (9)	0.0242 (8)	−0.0113 (8)	−0.0016 (7)	0.0005 (7)
C13	0.0372 (10)	0.0336 (9)	0.0295 (9)	−0.0126 (8)	−0.0039 (7)	0.0051 (7)
C14	0.0362 (9)	0.0346 (9)	0.0289 (8)	−0.0132 (7)	−0.0057 (7)	0.0007 (7)
C15	0.0404 (10)	0.0365 (9)	0.0259 (9)	−0.0118 (8)	−0.0083 (7)	0.0037 (7)
C16	0.0560 (12)	0.0356 (10)	0.0391 (10)	−0.0158 (9)	−0.0110 (9)	0.0051 (8)
C17	0.0408 (10)	0.0440 (10)	0.0245 (8)	−0.0105 (8)	−0.0027 (7)	−0.0002 (8)
C18	0.0445 (10)	0.0383 (10)	0.0372 (10)	−0.0179 (8)	−0.0100 (8)	0.0017 (8)
O7	0.1019 (12)	0.0465 (8)	0.0437 (8)	−0.0297 (8)	−0.0271 (8)	0.0170 (6)
O8	0.1237 (14)	0.0427 (9)	0.0523 (9)	−0.0445 (9)	−0.0285 (9)	0.0100 (7)
O9	0.0822 (10)	0.0535 (8)	0.0320 (7)	−0.0261 (8)	−0.0127 (7)	0.0138 (6)
O10	0.0888 (11)	0.0504 (8)	0.0273 (7)	−0.0199 (8)	−0.0148 (7)	−0.0045 (6)
O11	0.1039 (12)	0.0386 (8)	0.0593 (9)	−0.0352 (8)	−0.0273 (8)	0.0061 (7)
O12	0.0762 (10)	0.0484 (8)	0.0385 (7)	−0.0258 (7)	−0.0218 (7)	−0.0012 (6)
O13	0.0586 (9)	0.0473 (8)	0.0397 (8)	−0.0056 (7)	−0.0004 (7)	−0.0076 (6)
O14	0.0740 (10)	0.0496 (9)	0.0431 (8)	−0.0267 (8)	−0.0266 (7)	0.0026 (6)

Geometric parameters (Å, °)

N1—C23X	1.474 (6)	C1—C6	1.382 (2)
N1—C21X	1.494 (5)	C1—C7	1.506 (2)
N1—C23	1.498 (5)	C2—C3	1.391 (2)
N1—C19X	1.514 (6)	C2—H2	0.952 (17)
N1—C19	1.514 (5)	C3—C4	1.380 (2)
N1—C21	1.515 (4)	C3—C8	1.487 (2)
N1—H1N	0.88 (2)	C4—C5	1.389 (2)
C19—C20	1.513 (6)	C4—H4	0.942 (19)
C19—H19A	0.970	C5—C6	1.387 (2)
C19—H19B	0.970	C5—C9	1.484 (2)
C20—C19X	1.475 (7)	C6—H6	0.924 (19)
C20—H20A	0.960	C7—O1	1.245 (2)
C20—H20B	0.960	C7—O2	1.259 (2)
C20—H20C	0.960	C8—O3	1.204 (2)
C20—H20X	0.960	C8—O4	1.303 (2)
C20—H20Y	0.960	C9—O5	1.2141 (19)
C20—H20Z	0.960	C9—O6	1.316 (2)
C21—C22	1.508 (5)	O4—H4O	0.917 (15)
C21—H21A	0.970	O6—H6O	0.916 (15)
C21—H21B	0.970	C10—C15	1.383 (2)
C22—C21X	1.531 (6)	C10—C11	1.394 (2)
C22—H22A	0.960	C10—C16	1.482 (2)
C22—H22B	0.960	C11—C12	1.386 (2)
C22—H22C	0.960	C11—H11	0.933 (18)
C22—H22X	0.960	C12—C13	1.373 (2)
C22—H22Y	0.960	C12—C17	1.500 (2)
C22—H22Z	0.960	C13—C14	1.396 (2)
C23—C24	1.493 (6)	C13—H13	0.971 (17)

C23—H23A	0.970	C14—C15	1.383 (2)
C23—H23B	0.970	C14—C18	1.479 (2)
C24—C23X	1.491 (7)	C15—H15	0.945 (19)
C24—H24A	0.960	C16—O7	1.211 (2)
C24—H24B	0.960	C16—O8	1.306 (2)
C24—H24C	0.960	C17—O9	1.221 (2)
C24—H24X	0.960	C17—O10	1.279 (2)
C24—H24Y	0.960	C18—O11	1.205 (2)
C24—H24Z	0.960	C18—O12	1.308 (2)
C19X—H19X	0.970	O8—H8O	0.907 (15)
C19X—H19Y	0.970	O10—H10O	0.924 (16)
C21X—H21X	0.970	O12—H12O	0.925 (15)
C21X—H21Y	0.970	O13—H13A	0.901 (14)
C23X—H23X	0.970	O13—H13B	0.908 (15)
C23X—H23Y	0.970	O14—H14A	0.894 (15)
C1—C2	1.381 (2)	O14—H14B	0.889 (14)
C23X—N1—C21X	113.4 (4)	C23—C24—H24C	109.5
C23X—N1—C23	47.1 (3)	H24A—C24—H24C	109.5
C21X—N1—C23	146.2 (3)	H24B—C24—H24C	109.5
C23X—N1—C19X	113.2 (4)	C23X—C24—H24X	109.5
C21X—N1—C19X	111.3 (4)	C23—C24—H24X	133.5
C23—N1—C19X	68.2 (3)	H24A—C24—H24X	53.5
C23X—N1—C19	141.5 (3)	H24B—C24—H24X	116.9
C21X—N1—C19	64.2 (3)	H24C—C24—H24X	56.8
C23—N1—C19	111.7 (3)	C23X—C24—H24Y	109.5
C19X—N1—C19	47.9 (3)	C23—C24—H24Y	63.5
C23X—N1—C21	65.5 (3)	H24A—C24—H24Y	56.9
C21X—N1—C21	49.7 (3)	H24B—C24—H24Y	94.7
C23—N1—C21	110.2 (3)	H24C—C24—H24Y	155.6
C19X—N1—C21	145.0 (3)	H24X—C24—H24Y	109.5
C19—N1—C21	109.6 (3)	C23X—C24—H24Z	109.5
C23X—N1—H1N	107.4 (15)	C23—C24—H24Z	116.1
C21X—N1—H1N	102.7 (14)	H24A—C24—H24Z	116.4
C23—N1—H1N	109.5 (14)	H24C—C24—H24Z	94.7
C19X—N1—H1N	108.1 (15)	H24X—C24—H24Z	109.5
C19—N1—H1N	110.5 (15)	H24Y—C24—H24Z	109.5
C21—N1—H1N	105.1 (15)	C20—C19X—N1	114.8 (4)
C20—C19—N1	112.6 (3)	C20—C19X—H19X	108.6
C20—C19—H19A	109.1	N1—C19X—H19X	108.6
N1—C19—H19A	109.1	C20—C19X—H19Y	108.6
C20—C19—H19B	109.1	N1—C19X—H19Y	108.6
N1—C19—H19B	109.1	H19X—C19X—H19Y	107.5
H19A—C19—H19B	107.8	N1—C21X—C22	111.0 (4)
C19X—C20—C19	48.5 (3)	N1—C21X—H21X	109.4
C19X—C20—H20A	136.2	C22—C21X—H21X	109.4
C19—C20—H20A	109.5	N1—C21X—H21Y	109.4
C19X—C20—H20B	113.7	C22—C21X—H21Y	109.4
C19—C20—H20B	109.5	H21X—C21X—H21Y	108.0
H20A—C20—H20B	109.5	N1—C23X—C24	115.2 (5)

C19X—C20—H20C	62.5	N1—C23X—H23X	108.5
C19—C20—H20C	109.5	C24—C23X—H23X	108.5
H20A—C20—H20C	109.5	N1—C23X—H23Y	108.5
H20B—C20—H20C	109.5	C24—C23X—H23Y	108.5
C19X—C20—H20X	109.5	H23X—C23X—H23Y	107.5
C19—C20—H20X	134.8	C2—C1—C6	119.40 (15)
H20A—C20—H20X	55.1	C2—C1—C7	119.65 (15)
H20B—C20—H20X	115.7	C6—C1—C7	120.93 (15)
H20C—C20—H20X	55.5	C1—C2—C3	120.39 (15)
C19X—C20—H20Y	109.5	C1—C2—H2	119.8 (10)
C19—C20—H20Y	62.2	C3—C2—H2	119.7 (10)
H20A—C20—H20Y	55.9	C4—C3—C2	119.95 (15)
H20B—C20—H20Y	98.4	C4—C3—C8	122.76 (15)
H20C—C20—H20Y	152.0	C2—C3—C8	117.28 (15)
H20X—C20—H20Y	109.5	C3—C4—C5	119.93 (15)
C19X—C20—H20Z	109.5	C3—C4—H4	118.2 (11)
C19—C20—H20Z	115.1	C5—C4—H4	121.8 (11)
H20A—C20—H20Z	114.3	C6—C5—C4	119.63 (15)
H20C—C20—H20Z	98.3	C6—C5—C9	120.97 (15)
H20X—C20—H20Z	109.5	C4—C5—C9	119.39 (14)
H20Y—C20—H20Z	109.5	C1—C6—C5	120.68 (16)
C22—C21—N1	111.1 (3)	C1—C6—H6	118.4 (11)
C22—C21—H21A	109.4	C5—C6—H6	120.9 (11)
N1—C21—H21A	109.4	O1—C7—O2	125.67 (16)
C22—C21—H21B	109.4	O1—C7—C1	118.21 (15)
N1—C21—H21B	109.4	O2—C7—C1	116.11 (15)
H21A—C21—H21B	108.0	O3—C8—O4	123.44 (16)
C21—C22—C21X	49.2 (3)	O3—C8—C3	121.67 (16)
C21—C22—H22A	109.5	O4—C8—C3	114.89 (15)
C21X—C22—H22A	134.5	O5—C9—O6	123.69 (16)
C21—C22—H22B	109.5	O5—C9—C5	123.40 (15)
C21X—C22—H22B	115.6	O6—C9—C5	112.91 (14)
H22A—C22—H22B	109.5	C8—O4—H4O	109.0 (17)
C21—C22—H22C	109.5	C9—O6—H6O	112.0 (15)
C21X—C22—H22C	61.4	C15—C10—C11	119.67 (16)
H22A—C22—H22C	109.5	C15—C10—C16	119.61 (15)
H22B—C22—H22C	109.5	C11—C10—C16	120.68 (16)
C21—C22—H22X	133.7	C12—C11—C10	120.09 (16)
C21X—C22—H22X	109.5	C12—C11—H11	122.9 (10)
H22A—C22—H22X	51.6	C10—C11—H11	116.9 (10)
H22B—C22—H22X	116.7	C13—C12—C11	119.80 (15)
H22C—C22—H22X	58.7	C13—C12—C17	119.67 (15)
C21—C22—H22Y	61.2	C11—C12—C17	120.50 (15)
C21X—C22—H22Y	109.5	C12—C13—C14	120.67 (15)
H22A—C22—H22Y	58.9	C12—C13—H13	121.6 (10)
H22B—C22—H22Y	95.0	C14—C13—H13	117.7 (10)
H22C—C22—H22Y	155.5	C15—C14—C13	119.27 (15)
H22X—C22—H22Y	109.5	C15—C14—C18	122.61 (15)
C21—C22—H22Z	116.4	C13—C14—C18	118.11 (15)

C21X—C22—H22Z	109.5	C10—C15—C14	120.49 (15)
H22A—C22—H22Z	115.9	C10—C15—H15	120.6 (11)
H22C—C22—H22Z	95.0	C14—C15—H15	118.9 (11)
H22X—C22—H22Z	109.5	O7—C16—O8	123.58 (17)
H22Y—C22—H22Z	109.5	O7—C16—C10	122.95 (17)
C24—C23—N1	113.7 (3)	O8—C16—C10	113.46 (15)
C24—C23—H23A	108.8	O9—C17—O10	125.42 (16)
N1—C23—H23A	108.8	O9—C17—C12	120.53 (16)
C24—C23—H23B	108.8	O10—C17—C12	114.04 (15)
N1—C23—H23B	108.8	O11—C18—O12	123.21 (16)
H23A—C23—H23B	107.7	O11—C18—C14	122.05 (16)
C23X—C24—C23	46.9 (3)	O12—C18—C14	114.74 (15)
C23X—C24—H24A	134.0	C16—O8—H8O	115.7 (16)
C23—C24—H24A	109.5	C17—O10—H10O	112.2 (17)
C23X—C24—H24B	115.6	C18—O12—H12O	106.3 (14)
C23—C24—H24B	109.5	H13A—O13—H13B	96.5 (18)
H24A—C24—H24B	109.5	H14A—O14—H14B	98.8 (19)
C23X—C24—H24C	63.6		
C23X—N1—C19—C20	118.9 (6)	C3—C4—C5—C6	0.3 (2)
C21X—N1—C19—C20	−145.4 (5)	C3—C4—C5—C9	178.90 (15)
C23—N1—C19—C20	71.4 (4)	C2—C1—C6—C5	−1.8 (2)
C19X—N1—C19—C20	45.4 (4)	C7—C1—C6—C5	176.52 (15)
C21—N1—C19—C20	−166.2 (3)	C4—C5—C6—C1	1.1 (2)
N1—C19—C20—C19X	−46.3 (4)	C9—C5—C6—C1	−177.51 (15)
C23X—N1—C21—C22	−148.1 (5)	C2—C1—C7—O1	12.8 (2)
C21X—N1—C21—C22	48.5 (4)	C6—C1—C7—O1	−165.46 (16)
C23—N1—C21—C22	−163.4 (3)	C2—C1—C7—O2	−168.32 (16)
C19X—N1—C21—C22	116.1 (6)	C6—C1—C7—O2	13.4 (2)
C19—N1—C21—C22	73.3 (4)	C4—C3—C8—O3	−170.31 (18)
N1—C21—C22—C21X	−47.4 (3)	C2—C3—C8—O3	10.7 (3)
C23X—N1—C23—C24	47.1 (4)	C4—C3—C8—O4	8.9 (3)
C21X—N1—C23—C24	112.6 (6)	C2—C3—C8—O4	−170.15 (16)
C19X—N1—C23—C24	−151.2 (5)	C6—C5—C9—O5	176.00 (16)
C19—N1—C23—C24	−171.7 (4)	C4—C5—C9—O5	−2.6 (3)
C21—N1—C23—C24	66.2 (4)	C6—C5—C9—O6	−3.3 (2)
N1—C23—C24—C23X	−46.6 (4)	C4—C5—C9—O6	178.04 (15)
C19—C20—C19X—N1	47.4 (4)	C15—C10—C11—C12	−0.3 (3)
C23X—N1—C19X—C20	172.6 (4)	C16—C10—C11—C12	177.35 (16)
C21X—N1—C19X—C20	−58.4 (5)	C10—C11—C12—C13	−0.7 (3)
C23—N1—C19X—C20	158.0 (6)	C10—C11—C12—C17	−178.95 (15)
C19—N1—C19X—C20	−47.9 (4)	C11—C12—C13—C14	0.9 (3)
C21—N1—C19X—C20	−107.6 (6)	C17—C12—C13—C14	179.25 (15)
C23X—N1—C21X—C22	−63.8 (5)	C12—C13—C14—C15	−0.3 (3)
C23—N1—C21X—C22	−110.5 (6)	C12—C13—C14—C18	−178.94 (16)
C19X—N1—C21X—C22	167.2 (4)	C11—C10—C15—C14	0.9 (3)
C19—N1—C21X—C22	158.6 (5)	C16—C10—C15—C14	−176.72 (16)
C21—N1—C21X—C22	−47.5 (4)	C13—C14—C15—C10	−0.7 (3)
C21—C22—C21X—N1	48.3 (4)	C18—C14—C15—C10	177.94 (16)
C21X—N1—C23X—C24	165.6 (4)	C15—C10—C16—O7	−5.3 (3)

C23—N1—C23X—C24	−47.9 (4)	C11—C10—C16—O7	177.07 (19)
C19X—N1—C23X—C24	−66.4 (6)	C15—C10—C16—O8	174.17 (17)
C19—N1—C23X—C24	−117.1 (6)	C11—C10—C16—O8	−3.4 (3)
C21—N1—C23X—C24	151.9 (6)	C13—C12—C17—O9	3.2 (3)
C23—C24—C23X—N1	48.3 (4)	C11—C12—C17—O9	−178.48 (17)
C6—C1—C2—C3	1.1 (3)	C13—C12—C17—O10	−175.64 (16)
C7—C1—C2—C3	−177.21 (15)	C11—C12—C17—O10	2.7 (2)
C1—C2—C3—C4	0.2 (3)	C15—C14—C18—O11	−173.63 (18)
C1—C2—C3—C8	179.31 (15)	C13—C14—C18—O11	5.0 (3)
C2—C3—C4—C5	−0.9 (3)	C15—C14—C18—O12	7.0 (3)
C8—C3—C4—C5	−179.94 (15)	C13—C14—C18—O12	−174.39 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O9	0.88 (2)	1.90 (2)	2.771 (2)	166 (2)
O4—H4O···O13^{i}	0.917 (15)	1.651 (16)	2.5653 (18)	174 (2)
O6—H6O···O3^{ii}	0.916 (15)	1.703 (16)	2.5895 (18)	162 (2)
O8—H8O···O11^{iii}	0.907 (15)	1.697 (15)	2.5993 (19)	173 (2)
O10—H10O···O2	0.924 (16)	1.557 (17)	2.4671 (17)	167 (3)
O12—H12O···O14^{iv}	0.925 (15)	1.651 (15)	2.5740 (18)	176 (2)
O13—H13A···O1	0.901 (14)	1.936 (15)	2.832 (2)	173 (2)
O13—H13B···O5^{v}	0.908 (15)	1.951 (15)	2.8577 (18)	176 (3)
O14—H14A···O7^{vi}	0.894 (15)	1.935 (16)	2.8141 (19)	167 (3)
O14—H14B···O1	0.889 (14)	1.896 (16)	2.7433 (18)	159 (2)

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

Fig. 1

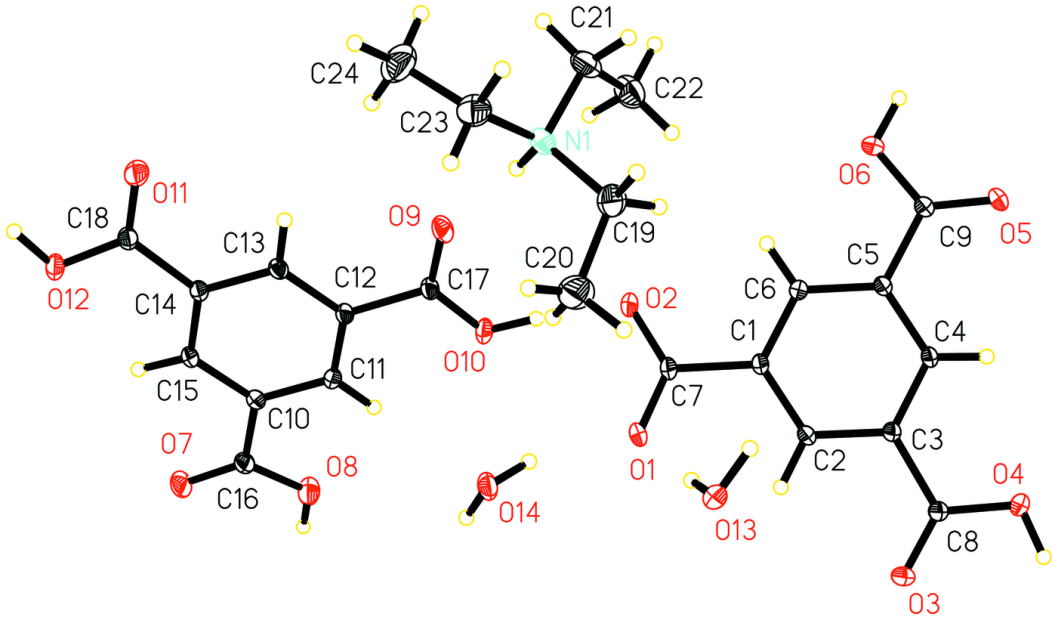
C21
C24
C22
C23
N1
O11
C18
O9
C19
C13
C20
C12
C17
O12
C14
O2
C6
C5
O6
C9
O5
C1
C4
O10
C11
C15
C7
C10
C3
O7
O8
C2
O4
O1
O14
O13
C8
O3

Fig. 2

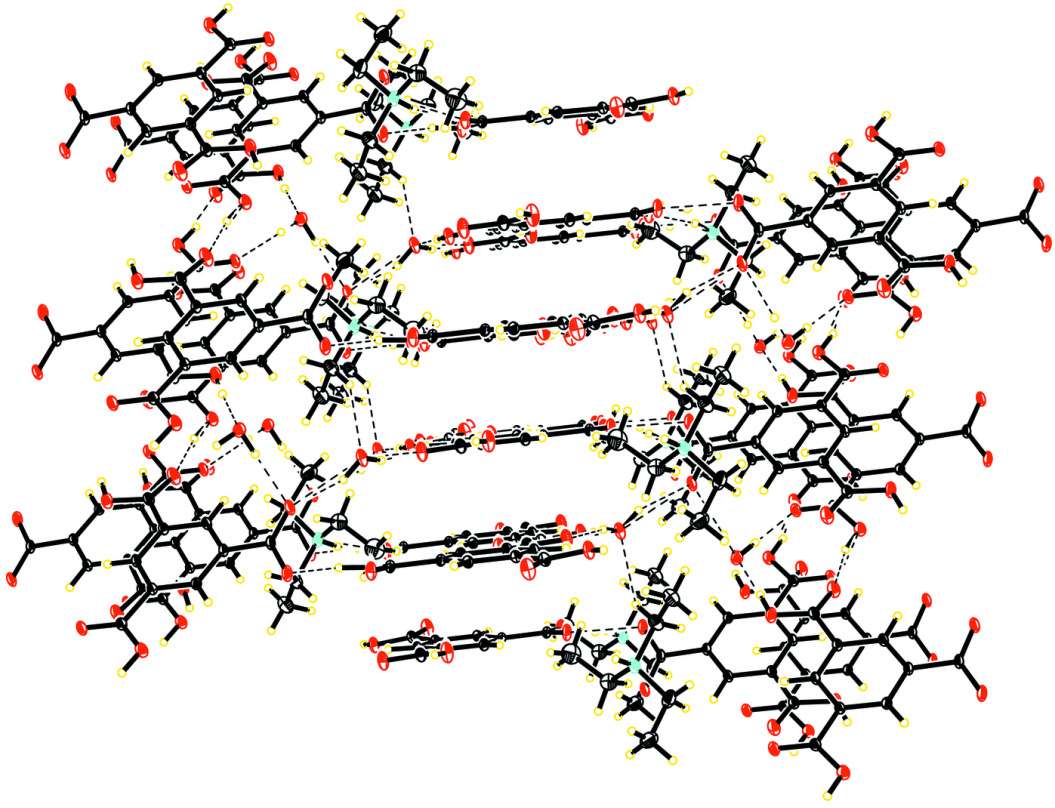

Fig. 3