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Anhydrous quinolinium dihydrogen citrate

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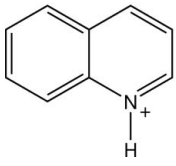
Key indicators: single-crystal X-ray study; T = 130 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.091; data-to-parameter ratio = 12.0.

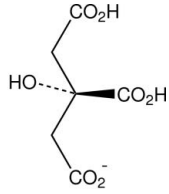
In the title compound, $C_9H_8N^+\cdot C_6H_7O_7^-$, the dihydrogen citrate anions form a convoluted two-dimensional hydrogen-bonded substructure through head-to-tail interactions with the interstitial π-stacked quinolinium cations linked to it peripherally through cyclic $R_1^2(5)$ $N^+—H\cdots O$(carboxyl/hydroxyl) hydrogen-bond interactions. The loss of a proton from one of the β-carboxylic acid groups of the citric acid generates a chiral centre in the anion, but these form a racemate in the centrosymmetric crystal structure.

Related literature

There is a similarity between the structure of the title compound and those of the quinolinium hydrogen salts of fumaric acid (Shan *et al.*, 2003) and L-tartaric acid (Smith, Wermuth & White, 2006).

For related literature, see: Glusker *et al.* (1965); Smith *et al.* (2004, 2007); Smith, Wermuth & Healy (2006); Tapscott (1982); Yathirajan *et al.* (2005).





Experimental

Crystal data

$C_9H_8N^+\cdot C_6H_7O_7^-$
M_r = 321.28
Monoclinic, $P2_1/n$
a = 7.5202 (6) Å
b = 11.9267 (10) Å
c = 17.1484 (14) Å
β = 93.217 (1)°
V = 1535.6 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 130 (2) K
0.50 × 0.35 × 0.30 mm

Data collection

Bruker SMART CCD detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.95, T_{max} = 0.97
7847 measured reflections
2695 independent reflections
2431 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.092
S = 1.07
2695 reflections
225 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O31	0.88 (2)	2.17 (2)	2.9215 (15)	144 (2)
N1—H1···O62	0.88 (2)	2.10 (2)	2.8219 (15)	140 (2)
O51—H51···O11^{i}	0.90 (2)	1.62 (2)	2.5208 (16)	180 (3)
O31—H31···O12	0.85 (2)	1.85 (2)	2.6337 (13)	151.4 (17)
O61—H61···O11^{ii}	0.99 (2)	2.57 (2)	3.1758 (14)	119.6 (14)
O61—H61···O12^{ii}	0.99 (2)	1.58 (2)	2.5641 (12)	174 (2)

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2355).

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supplementary materials

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Comment

The structures of the quinolinium carboxylates and sulfonates are not prevalent in the crystallographic literature and most of the examples are 1:1 salts, mainly with the aromatic acids e.g. 5-sulfosalicylic acid (Smith *et al.*, 2004), 3,5-dinitrosalicylic acid (Smith, Wermuth & White, 2006), and picrylsulfonic acid (Smith, Wermuth & Healy, 2006). These compounds often feature π-associated cation-anion stacks with peripheral hydrogen bonding giving three-dimensional framework structures. With the quinolinium salts of the aliphatic carboxylic acids, most examples are anhydrous acid salts of polyprotic analogues, e.g. fumaric acid (Shan *et al.*, 2003) and *L*-tartaric acid (Smith *et al.*, (2006). The 1:1 stoichiometric reaction of quinoline with citric acid in isopropyl alcohol was expected to give a similar acid citrate and this was confirmed in the structure determination of $C_9H_8N^+$ $C_6H_7O_7^-$ (I), reported here.

Figure 1 shows the quinolinium cation and the dihydrogen citrate anion in which one of the β-carboxylic acid groups rather than the α-group has lost the proton. It is more usual for the α-group to be associated with the first dissociation constant (Tapscott, 1982) and is seen in typical structures such as sodium dihydrogen citrate (Glusker *et al.*, 1965), and sildenafil dihydrogen citrate (Yathirajan *et al.*, 2005). In (I), this results in the generation a chiral centre at C31 in the anion species but these form a racemete in the centrosymmetric crystal. These anions form a convoluted two-dimensional hydrogen-bonded substructure through head-to-tail carboxylic acid···carboxylate interactions, one linear, the other three-centred cyclic [$R^2_1(4)$] (Table 1). The partially overlapping quinolinium cations [C5–C10: minimum ring centroid and perpendicular separations of 3.840 (1) and 3.560 (1) Å respectively] form π-associated stacks which extend down the *a* cell direction. The anion substructures accommodate these stacks (Fig. 2). which are linked to the anionic substructure by symmetric three-centre $R^2_1(5)$ $N^+H\cdots O$(carboxyl, hydroxyl) hydrogen-bonding associations. The result is a three-dimensional framework structure which in addition has 66.8 $Å^3$ potential solvent accessible voids.

The conformation of the dihydrogen citrate anions is maintained by the presence of an intramolecular hydroxyl–carboxyl hydrogen bond [O31—H···O12, 2.6337 (13) Å].

Experimental

Compound (I) was synthesized by heating 1 mmol quantities of citric acid and quinoline in 50 ml of 2-propanol for 10 min under reflux. Colourless needles (m.p. 403 K) were obtained after partial room-temperature evaporation of solvent.

Refinement

Hydrogen atoms potentially involved in hydrogen-bonding interactions were located by difference methods and their positional and isotropic displacement parameters were refined. Other H atoms were included in the refinement at calculated positions [C—H (aromatic) = 0.95 Å and C–H (aliphatic) = 0.99 Å] using a riding-model approximation with U_{iso}(H) = 1.2U_{eq}(C).

Figures

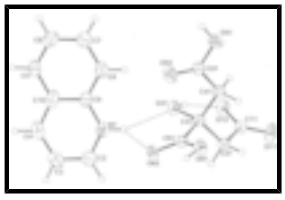


Fig. 1. The molecular configuration and atom-numbering scheme for the quinolinium cation and the dihydrogen citrate anion in (I). Non-H atoms are shown as 30% probability displacement ellipsoids.

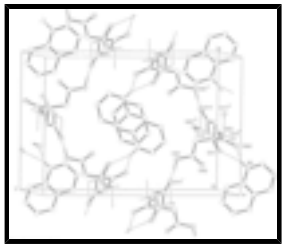


Fig. 2. A perspective view of the packing of (I) in the unit cell viewed down the approximate *a* axial direction, showing the π-stacked quinolinium cations inside the citrate convoluted sheet substructures. Hydrogen-bonding interactions are shown as dashed lines and non- interactive hydrogen atoms are omitted: symmetry codes: (iii) *x* + 1, *y*, *z*; (iv) -*x* - 1/2, *y* - 3/2, -*z* + 1/2; For other codes see Table 1.

quinolinium dihydrogen citrate

Crystal data

$C_9H_8N^+·C_6H_7O_7^-$	F_{000} = 672
M_r = 321.28	D_x = 1.390 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 403 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.5202 (6) Å	Cell parameters from 4204 reflections
b = 11.9267 (10) Å	θ = 2.9–27.5º
c = 17.1484 (14) Å	μ = 0.11 mm^{-1}
β = 93.217 (1)º	*T* = 130 (2) K
V = 1535.6 (2) Å^3	Cut block, colourless
Z = 4	0.50 × 0.35 × 0.30 mm

Data collection

Bruker SMART CCD detector diffractometer	2695 independent reflections
Radiation source: sealed tube	2431 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
T = 130(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	*h* = −8→8
T_{min} = 0.95, T_{max} = 0.97	*k* = −12→14
7847 measured reflections	*l* = −20→20

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.032$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.092$	$\Delta\rho_{max} = 0.18$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
2695 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
225 parameters	Extinction coefficient: 0.0085 (16)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

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}
O31	0.69503 (12)	0.31070 (7)	0.07462 (5)	0.0428 (3)
O11	0.44066 (13)	0.60432 (9)	0.13920 (7)	0.0622 (4)
O12	0.40951 (11)	0.43148 (8)	0.09764 (6)	0.0472 (3)
O51	0.85983 (16)	0.25801 (10)	0.30537 (6)	0.0627 (4)
O52	0.98000 (17)	0.22265 (10)	0.19299 (6)	0.0688 (4)
O61	1.08487 (11)	0.46033 (8)	0.13149 (5)	0.0455 (3)
O62	1.01275 (11)	0.35650 (8)	0.02589 (5)	0.0431 (3)
C11	0.50345 (15)	0.51775 (10)	0.11240 (7)	0.0350 (4)
C21	0.69856 (15)	0.51305 (10)	0.09419 (7)	0.0347 (3)
C31	0.78566 (15)	0.39947 (10)	0.11505 (7)	0.0332 (3)
C41	0.78738 (17)	0.38158 (11)	0.20312 (7)	0.0398 (4)
C51	0.88543 (17)	0.27801 (11)	0.23168 (8)	0.0427 (4)
C61	0.97426 (15)	0.40111 (10)	0.08600 (7)	0.0346 (3)
N1	0.82320 (16)	0.18706 (10)	−0.05817 (7)	0.0497 (4)
C2	0.8459 (2)	0.22022 (14)	−0.13027 (9)	0.0562 (5)

C3	0.8327 (2)	0.14309 (16)	−0.19125 (9)	0.0625 (6)
C4	0.7987 (2)	0.03385 (16)	−0.17563 (9)	0.0628 (6)
C5	0.7391 (2)	−0.11459 (14)	−0.07793 (11)	0.0498 (5)
C6	0.7164 (2)	−0.14220 (15)	−0.00272 (12)	0.0689 (6)
C7	0.7289 (2)	−0.06036 (14)	0.05581 (11)	0.0655 (6)
C8	0.7652 (2)	0.04851 (13)	0.03880 (9)	0.0572 (5)
C9	0.78666 (18)	0.07852 (11)	−0.03881 (8)	0.0443 (4)
C10	0.77402 (18)	−0.00213 (13)	−0.09901 (9)	0.0646 (6)
H51	0.931 (3)	0.203 (2)	0.3252 (13)	0.080 (8)*
H21A	0.76380	0.57310	0.12360	0.0420*
H21B	0.70910	0.52770	0.03780	0.0420*
H31	0.586 (3)	0.3302 (15)	0.0746 (10)	0.064 (5)*
H41A	0.84280	0.44780	0.22940	0.0480*
H41B	0.66280	0.37690	0.21870	0.0480*
H61	1.208 (3)	0.4490 (17)	0.1150 (12)	0.088 (6)*
H1	0.832 (3)	0.2372 (17)	−0.0209 (12)	0.074 (6)*
H2	0.87120	0.29670	−0.14060	0.0680*
H3	0.84730	0.16670	−0.24340	0.0750*
H4	0.79150	−0.01900	−0.21710	0.0750*
H5	0.73150	−0.17110	−0.11700	0.0780*
H6	0.69180	−0.21770	0.01050	0.0830*
H7	0.71170	−0.08110	0.10830	0.0790*
H8	0.77580	0.10320	0.07910	0.0690*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O31	0.0297 (5)	0.0399 (5)	0.0592 (6)	−0.0029 (4)	0.0066 (4)	−0.0124 (4)
O11	0.0347 (5)	0.0547 (6)	0.0974 (8)	0.0032 (5)	0.0045 (5)	−0.0326 (6)
O12	0.0253 (5)	0.0451 (5)	0.0711 (6)	−0.0022 (4)	0.0030 (4)	−0.0105 (4)
O51	0.0738 (7)	0.0627 (7)	0.0537 (6)	0.0255 (6)	0.0220 (5)	0.0206 (5)
O52	0.0789 (8)	0.0706 (7)	0.0585 (6)	0.0392 (6)	0.0184 (6)	0.0070 (5)
O61	0.0257 (5)	0.0609 (6)	0.0498 (5)	−0.0017 (4)	0.0023 (4)	−0.0123 (4)
O62	0.0353 (5)	0.0516 (6)	0.0434 (5)	−0.0009 (4)	0.0107 (4)	−0.0081 (4)
C11	0.0282 (6)	0.0404 (7)	0.0360 (6)	0.0017 (5)	−0.0021 (5)	−0.0018 (5)
C21	0.0284 (6)	0.0361 (6)	0.0396 (6)	−0.0019 (5)	0.0014 (5)	0.0006 (5)
C31	0.0260 (6)	0.0348 (6)	0.0389 (6)	−0.0006 (5)	0.0036 (5)	−0.0034 (5)
C41	0.0353 (7)	0.0429 (7)	0.0419 (7)	0.0068 (5)	0.0097 (5)	0.0026 (5)
C51	0.0393 (7)	0.0436 (7)	0.0459 (7)	0.0043 (6)	0.0079 (5)	0.0036 (6)
C61	0.0278 (6)	0.0377 (6)	0.0385 (6)	0.0021 (5)	0.0027 (5)	0.0005 (5)
N1	0.0546 (7)	0.0443 (7)	0.0497 (7)	0.0104 (5)	−0.0025 (5)	−0.0084 (6)
C2	0.0492 (9)	0.0582 (9)	0.0615 (9)	0.0132 (7)	0.0050 (7)	0.0075 (7)
C3	0.0566 (10)	0.0827 (12)	0.0487 (8)	0.0153 (8)	0.0072 (7)	0.0000 (8)
C4	0.0558 (9)	0.0776 (12)	0.0550 (9)	0.0078 (8)	0.0032 (7)	−0.0235 (8)
C5	0.0378 (7)	0.0543 (9)	0.0575 (8)	0.0062 (6)	0.0034 (6)	−0.0165 (7)
C6	0.0579 (10)	0.0494 (9)	0.1007 (14)	0.0040 (7)	0.0172 (9)	0.0010 (9)
C7	0.0676 (11)	0.0598 (10)	0.0701 (10)	0.0139 (8)	0.0130 (8)	0.0081 (8)
C8	0.0667 (10)	0.0535 (9)	0.0513 (8)	0.0139 (7)	0.0023 (7)	−0.0055 (7)

C9	0.0384 (7)	0.0441 (8)	0.0502 (8)	0.0100 (6)	−0.0003 (6)	−0.0074 (6)
C10	0.0538 (9)	0.0512 (9)	0.0894 (13)	0.0006 (7)	0.0090 (8)	−0.0255 (8)

Geometric parameters (Å, °)

O31—C31	1.4188 (15)	C21—H21A	0.9900
O11—C11	1.2347 (16)	C41—H41B	0.9900
O12—C11	1.2656 (15)	C41—H41A	0.9900
O31—H31	0.85 (2)	C2—C3	1.392 (2)
O51—C51	1.3108 (17)	C3—C4	1.357 (3)
O52—C51	1.1979 (18)	C4—C10	1.404 (2)
O51—H51	0.90 (2)	C5—C10	1.418 (2)
O61—C61	1.3140 (15)	C5—C6	1.351 (3)
O62—C61	1.2095 (15)	C6—C7	1.399 (3)
O61—H61	0.99 (2)	C7—C8	1.362 (2)
N1—C9	1.3681 (18)	C8—C9	1.396 (2)
N1—C2	1.318 (2)	C9—C10	1.410 (2)
N1—H1	0.88 (2)	C2—H2	0.9500
C11—C21	1.5182 (16)	C3—H3	0.9500
C21—C31	1.5381 (17)	C4—H4	0.9500
C31—C41	1.5246 (17)	C5—H5	0.9500
C31—C61	1.5295 (16)	C6—H6	0.9500
C41—C51	1.5063 (18)	C7—H7	0.9500
C21—H21B	0.9900	C8—H8	0.9500
C31—O31—H31	103.4 (12)	C51—C41—H41A	109.00
C51—O51—H51	112.3 (14)	C51—C41—H41B	109.00
C61—O61—H61	109.1 (12)	H41A—C41—H41B	108.00
C2—N1—C9	123.35 (13)	N1—C2—C3	119.92 (15)
C9—N1—H1	118.5 (13)	C2—C3—C4	119.50 (15)
C2—N1—H1	118.1 (13)	C3—C4—C10	120.94 (15)
O11—C11—O12	122.24 (11)	C6—C5—C10	120.61 (16)
O11—C11—C21	120.11 (11)	C5—C6—C7	120.51 (16)
O12—C11—C21	117.64 (10)	C6—C7—C8	121.17 (17)
C11—C21—C31	112.87 (10)	C7—C8—C9	118.94 (15)
C41—C31—C61	111.59 (10)	N1—C9—C10	118.29 (13)
O31—C31—C41	110.90 (10)	C8—C9—C10	121.08 (13)
O31—C31—C21	110.91 (9)	N1—C9—C8	120.62 (13)
O31—C31—C61	106.21 (9)	C5—C10—C9	117.68 (14)
C21—C31—C41	109.48 (10)	C4—C10—C5	124.32 (15)
C21—C31—C61	107.67 (9)	C4—C10—C9	118.00 (14)
C31—C41—C51	114.43 (11)	N1—C2—H2	120.00
O51—C51—C41	111.53 (11)	C3—C2—H2	120.00
O51—C51—O52	123.89 (13)	C2—C3—H3	120.00
O52—C51—C41	124.54 (13)	C4—C3—H3	120.00
O61—C61—C31	112.41 (10)	C3—C4—H4	120.00
O61—C61—O62	124.66 (11)	C10—C4—H4	120.00
O62—C61—C31	122.86 (11)	C6—C5—H5	120.00
C11—C21—H21A	109.00	C10—C5—H5	120.00
C11—C21—H21B	109.00	C5—C6—H6	120.00

H21A—C21—H21B	108.00	C7—C6—H6	120.00
C31—C21—H21A	109.00	C6—C7—H7	119.00
C31—C21—H21B	109.00	C8—C7—H7	119.00
C31—C41—H41A	109.00	C7—C8—H8	121.00
C31—C41—H41B	109.00	C9—C8—H8	121.00
C2—N1—C9—C10	0.5 (2)	C31—C41—C51—O52	11.94 (19)
C9—N1—C2—C3	0.1 (2)	C31—C41—C51—O51	−170.43 (11)
C2—N1—C9—C8	179.57 (14)	N1—C2—C3—C4	−0.8 (2)
O12—C11—C21—C31	−40.13 (15)	C2—C3—C4—C10	1.0 (2)
O11—C11—C21—C31	141.26 (12)	C3—C4—C10—C9	−0.4 (2)
C11—C21—C31—C41	−64.53 (13)	C3—C4—C10—C5	−179.63 (15)
C11—C21—C31—C61	173.98 (10)	C6—C5—C10—C9	0.9 (2)
C11—C21—C31—O31	58.17 (13)	C10—C5—C6—C7	−0.7 (2)
O31—C31—C41—C51	62.88 (13)	C6—C5—C10—C4	−179.79 (15)
O31—C31—C61—O61	−164.52 (10)	C5—C6—C7—C8	−0.4 (2)
C21—C31—C61—O61	76.61 (12)	C6—C7—C8—C9	1.3 (2)
C21—C31—C61—O62	−100.34 (13)	C7—C8—C9—C10	−1.0 (2)
C41—C31—C61—O61	−43.55 (14)	C7—C8—C9—N1	180.00 (14)
C21—C31—C41—C51	−174.41 (10)	N1—C9—C10—C4	−0.4 (2)
C61—C31—C41—C51	−55.31 (14)	N1—C9—C10—C5	178.95 (13)
C41—C31—C61—O62	139.50 (12)	C8—C9—C10—C4	−179.42 (14)
O31—C31—C61—O62	18.52 (15)	C8—C9—C10—C5	−0.10 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O31	0.88 (2)	2.17 (2)	2.9215 (15)	144 (2)
N1—H1···O62	0.88 (2)	2.10 (2)	2.8219 (15)	140 (2)
O51—H51···O11^{i}	0.90 (2)	1.62 (2)	2.5208 (16)	180 (3)
O31—H31···O12	0.85 (2)	1.85 (2)	2.6337 (13)	151.4 (17)
O61—H61···O11^{ii}	0.99 (2)	2.57 (2)	3.1758 (14)	119.6 (14)
O61—H61···O12^{ii}	0.99 (2)	1.58 (2)	2.5641 (12)	174 (2)
C7—H7···O51^{i}	0.95	2.50	3.314 (2)	144
C8—H8···O31	0.95	2.55	3.2358 (18)	129
C21—H21B···O12^{iii}	0.99	2.49	3.4087 (16)	154

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

Fig. 1

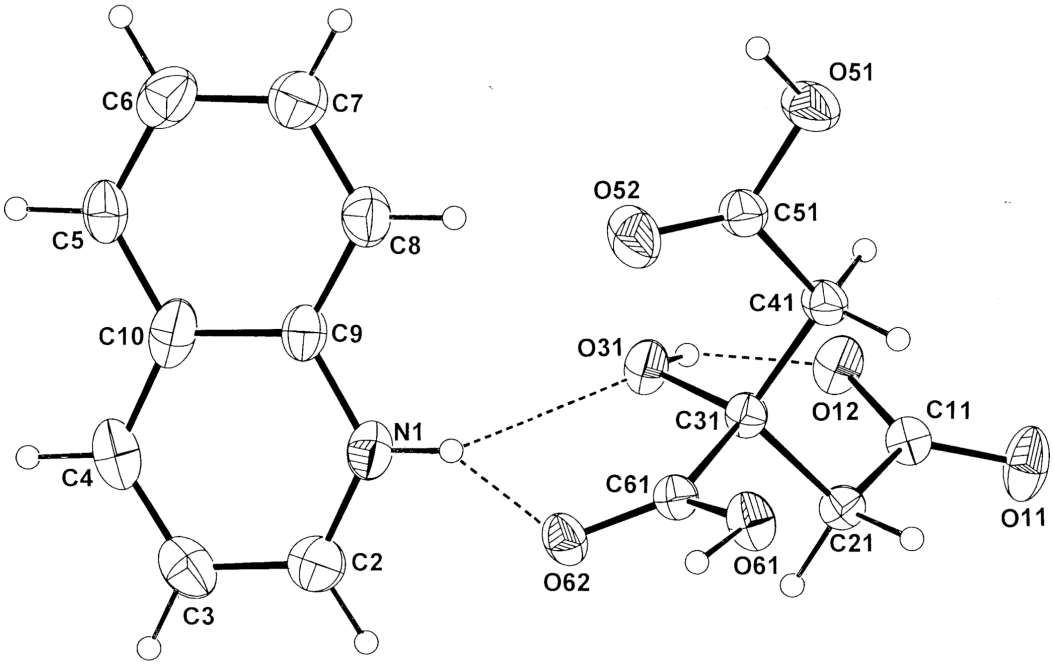
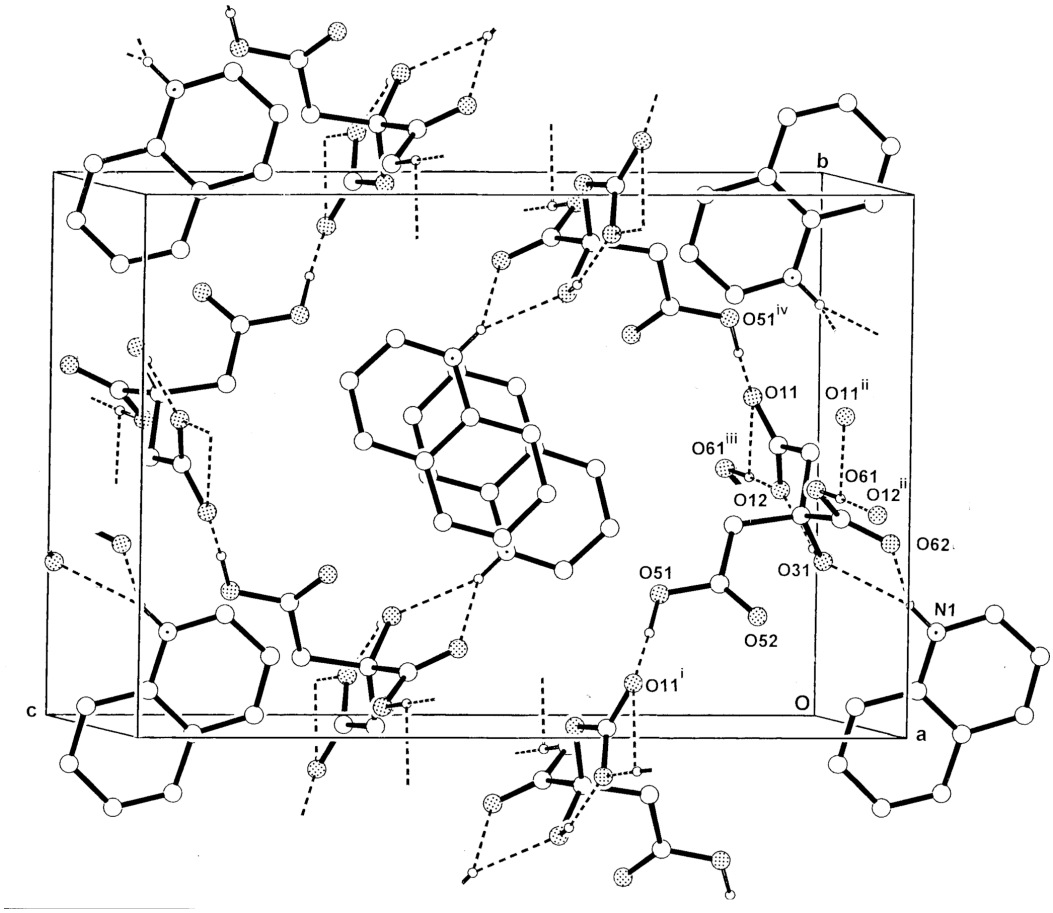

C6
C7
C5
C8
C10
C9
N1
C4
C2
C3
O51
O52
C51
C41
O31
O12
C11
C31
C61
O11
C21
O62
O61

Fig. 2


b
c
a
O
O51iv
O11
O11ii
O61iii
O61
O12
O12ii
O62
O31
O51
O52
N1
O11i