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2-{*N*′-[3-(Dimethylammonio)propyl]-oxamido}benzoate

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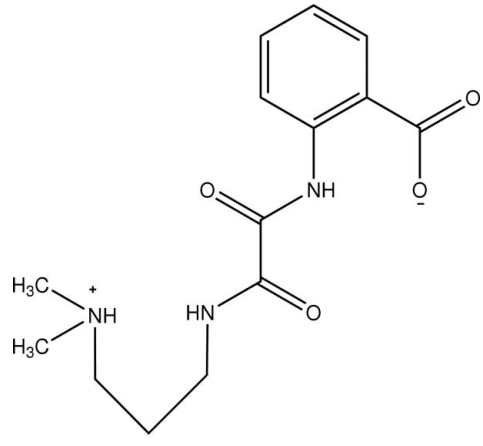
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.139; data-to-parameter ratio = 13.4.

The title compound, $C_{14}H_{19}N_3O_4$, exists as a zwitterion, with a tertiary N atom protonated and a carboxyl group deprotonated. In the crystal structure, the planar oxamide group displays a *transoid* conformation. The molecules link to each other *via* hydrogen bonding, resulting in an extended supramolecular chain along the *b* axis.

Related literature

For general background, see: Ojima & Nonoyama (1988); Matović *et al.* (2005); Pei *et al.* (1991); Zang *et al.* (2003). For related structures, see: Perić *et al.* (2001); Su *et al.* (1999); Sun *et al.* (2006). For synthesis, see: Matović *et al.* (2005).



Experimental

Crystal data

$C_{14}H_{19}N_3O_4$
M_r = 293.32
Monoclinic, $P2_1/c$
a = 14.780 (5) Å
b = 8.762 (3) Å
c = 11.576 (4) Å
β = 104.193 (5)°
V = 1453.4 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.24 × 0.23 × 0.10 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: none
7492 measured reflections
2628 independent reflections
1374 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.139
S = 1.03
2628 reflections
196 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2	0.86	1.98	2.637 (3)	132
N3—H3A···O2^{i}	0.912 (19)	1.693 (18)	2.604 (3)	177 (3)

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2306).

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supplementary materials

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2-{*N*'-[3-(Dimethylammonio)propyl]oxamido}benzoate

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Comment

N,*N*'-disubstituted oxamidate derivatives are known to be versatile organic ligands, which can chelate as well as bridge the metal ions to construct discrete and extend structures (Ojima & Nonoyama, 1988). Compared with the symmetrical substituted derivatives, the design and synthesis of those unsymmetrical substituted are rather limited owing to the relative difficulty of synthesizing new compounds (Matović *et al.*, 2005; Pei *et al.*, 1991; Zang *et al.*, 2003). Herein, we report the synthesis and X-ray structural of the title oxamidate compound.

The title compound occurs as a zwitterion with the tertiary N atom (N3) protonated and the carboxyl group deprotonated (Fig. 1). There is an intramolecular hydrogen bond between amido nitrogen atom (N1) and carboxyl oxygen atom (O2). The zwitterion has a *transoid* conformation, and six atoms of oxamido group are almost coplanar, which is similar to other oxamido compounds (Perić *et al.*, 2001; Su *et al.*, 1999; Sun *et al.*, 2006). The dihedral angle between the oxamide and the benzene ring is 27.05 (12)°, and that between the benzene ring and carboxyl group is 23.9 (4)°.

As shown in Fig. 2, the zwitterions are linked into a 1-D ribbon along the *b* axis by the hydrogen bonding (Table 1).

Experimental

All reagents were of AR grade and obtained commercially without further purification. The title compound was prepared according to Matović *et al.* (2005). A THF (THF= tetrahydrofuran) solution (8 ml) of ethyl oxalyl chloride (1.11 ml, 10 mmol) was added dropwise into a THF solution (10 ml) of anthranilic acid (1.37 g, 10 mmol) with continuous stirring. The mixture was stirred quickly for 1 h and then 20 ml absolute ethanol was further added and the mixture was added dropwise into the absolute ethanol solution (10 ml) of 3-dimethylamino-propylamine (1.02 g, 10 mmol) with stirring and kept the temperature at 273 K for 8 h. The title compound was precipitated as a white powder and washed with absolute ethanol for several times and dried under vacuum. Yield: 1.79 g (75%). Colorless crystals of the compound suitable for X-ray analysis were obtained from an ethanol/water (1:1) mixture by slow evaporation for one week at room temperature.

Refinement

The H atom on protonated tertiary nitrogen atom N3 was located in a different Fourier map and refined with a restraint of N—H = 0.91 Å, final U_{iso}(H) value being 0.046 (9) Å^2. Other H atoms were placed in calculated positions with C—H = 0.97 Å (methylene), 0.96 Å (methyl), 0.93 Å (aromatic) and N—H = 0.86 Å, and included in the final cycles of refinement in riding mode. Torsion angles for methyl groups were refined with U_{iso}(H) = 1.5U_{eq}(C). For other H atoms, U_{iso}(H) = 1.2U_{eq}(C,*N*).

Figures

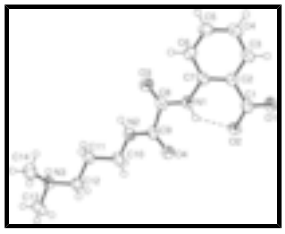

Fig. 1. The molecular structure of the title compound, with 50% probability displacement ellipsoids. Dashed line indicates hydrogen bond.

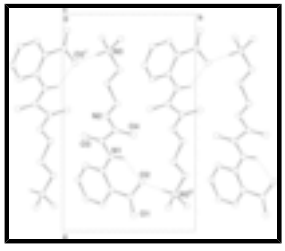

Fig. 2. The packing diagram for (I), viewed down the *c* axis. The H-bonds are shown as dashed lines; H atoms not involved in hydrogen bonding were omitted for clarity [symmetry codes: (i) 1 − *x*, *y* − 1/2, 3/2 − *z*; (ii) 1 − *x*, *y* + 1/2, 3/2 − *z*].

2-{*N*'-[3-(Dimethylammonio)propyl]oxamido}benzoate

Crystal data

$C_{14}H_{19}N_3O_4$	F_{000} = 624
M_r = 293.32	D_x = 1.341 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1356 reflections
a = 14.780 (5) Å	θ = 2.4–22.7º
b = 8.762 (3) Å	μ = 0.10 mm^{-1}
c = 11.576 (4) Å	*T* = 298 (2) K
β = 104.193 (5)º	Block, colourless
V = 1453.4 (9) Å^3	0.24 × 0.23 × 0.10 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	1374 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.059
Monochromator: graphite	θ_{max} = 25.2º
T = 298(2) K	θ_{min} = 1.4º
φ and ω scans	*h* = −17→17
Absorption correction: none	*k* = −10→8
7492 measured reflections	*l* = −13→11
2628 independent reflections	

Refinement

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

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.139$

$S = 1.03$

2628 reflections

196 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.0967P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.23$ e Å^{-3}

$\Delta\rho_{min} = -0.21$ e Å^{-3}

Extinction correction: none

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}
C1	0.8415 (2)	0.5075 (3)	0.5747 (3)	0.0353 (7)
C2	0.81197 (19)	0.3653 (3)	0.4998 (3)	0.0337 (7)
C3	0.8755 (2)	0.3010 (3)	0.4428 (3)	0.0394 (8)
H3	0.9354	0.3413	0.4566	0.047*
C4	0.8514 (2)	0.1781 (4)	0.3659 (3)	0.0460 (9)
H4	0.8943	0.1380	0.3272	0.055*
C5	0.7636 (2)	0.1161 (3)	0.3474 (3)	0.0454 (8)
H5	0.7471	0.0340	0.2955	0.055*
C6	0.6995 (2)	0.1739 (3)	0.4047 (3)	0.0419 (8)
H6	0.6409	0.1292	0.3929	0.050*
C7	0.72259 (19)	0.2997 (3)	0.4806 (3)	0.0335 (7)
C8	0.5848 (2)	0.2933 (3)	0.5658 (3)	0.0377 (8)
C9	0.53513 (19)	0.3990 (3)	0.6361 (3)	0.0361 (7)
C10	0.41422 (19)	0.4063 (4)	0.7457 (3)	0.0462 (9)
H10A	0.4076	0.5132	0.7235	0.055*
H10B	0.4460	0.4002	0.8295	0.055*
C11	0.31874 (19)	0.3339 (3)	0.7258 (3)	0.0402 (8)
H11A	0.3249	0.2307	0.7571	0.048*
H11B	0.2900	0.3286	0.6410	0.048*
C12	0.25722 (19)	0.4261 (3)	0.7870 (3)	0.0398 (8)

H12A	0.2923	0.4511	0.8671	0.048*
H12B	0.2405	0.5211	0.7441	0.048*
C13	0.1151 (2)	0.4356 (4)	0.8607 (3)	0.0593 (11)
H13A	0.1554	0.4706	0.9340	0.089*
H13B	0.0665	0.3734	0.8777	0.089*
H13C	0.0880	0.5217	0.8135	0.089*
C14	0.1101 (2)	0.2974 (4)	0.6764 (3)	0.0513 (9)
H14A	0.0570	0.2423	0.6884	0.077*
H14B	0.1451	0.2334	0.6360	0.077*
H14C	0.0893	0.3866	0.6292	0.077*
N1	0.65712 (15)	0.3637 (3)	0.5368 (2)	0.0376 (7)
H1	0.6642	0.4589	0.5547	0.045*
N2	0.46907 (16)	0.3289 (3)	0.6754 (2)	0.0460 (7)
H2	0.4583	0.2341	0.6585	0.055*
N3	0.17012 (16)	0.3439 (3)	0.7937 (2)	0.0366 (6)
H3A	0.1880 (18)	0.2573 (18)	0.837 (2)	0.046 (9)*
O1	0.92471 (14)	0.5334 (2)	0.6149 (2)	0.0555 (7)
O2	0.77507 (13)	0.5939 (2)	0.58818 (19)	0.0461 (6)
O3	0.56068 (15)	0.1614 (2)	0.5449 (2)	0.0553 (7)
O4	0.55702 (13)	0.5330 (2)	0.6540 (2)	0.0500 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0358 (18)	0.0429 (18)	0.0285 (19)	−0.0025 (14)	0.0101 (15)	0.0036 (15)
C2	0.0326 (17)	0.0388 (17)	0.0301 (19)	0.0015 (13)	0.0084 (15)	0.0052 (14)
C3	0.0334 (17)	0.0502 (19)	0.037 (2)	0.0040 (14)	0.0120 (16)	0.0007 (16)
C4	0.046 (2)	0.054 (2)	0.043 (2)	0.0096 (16)	0.0200 (18)	−0.0051 (17)
C5	0.049 (2)	0.0469 (19)	0.040 (2)	0.0044 (16)	0.0110 (18)	−0.0097 (16)
C6	0.0395 (19)	0.0470 (19)	0.038 (2)	−0.0047 (14)	0.0075 (17)	−0.0086 (16)
C7	0.0325 (17)	0.0398 (17)	0.0291 (19)	0.0044 (13)	0.0091 (15)	0.0029 (14)
C8	0.0328 (17)	0.0424 (18)	0.039 (2)	0.0023 (14)	0.0110 (16)	0.0034 (16)
C9	0.0282 (16)	0.0434 (18)	0.036 (2)	0.0001 (14)	0.0056 (15)	0.0014 (16)
C10	0.0375 (19)	0.058 (2)	0.048 (2)	−0.0008 (15)	0.0190 (17)	−0.0008 (18)
C11	0.0347 (17)	0.0447 (18)	0.044 (2)	0.0019 (14)	0.0155 (16)	0.0024 (16)
C12	0.0345 (17)	0.0401 (18)	0.046 (2)	−0.0016 (13)	0.0126 (16)	0.0038 (15)
C13	0.049 (2)	0.070 (2)	0.068 (3)	0.0075 (18)	0.031 (2)	−0.008 (2)
C14	0.0417 (19)	0.068 (2)	0.042 (2)	−0.0041 (17)	0.0056 (18)	0.0009 (18)
N1	0.0324 (14)	0.0404 (15)	0.0424 (17)	−0.0034 (11)	0.0138 (13)	−0.0063 (13)
N2	0.0410 (16)	0.0436 (15)	0.061 (2)	−0.0060 (12)	0.0264 (15)	−0.0056 (14)
N3	0.0325 (14)	0.0412 (16)	0.0376 (17)	0.0005 (12)	0.0115 (13)	0.0058 (13)
O1	0.0293 (13)	0.0661 (15)	0.0691 (18)	−0.0068 (11)	0.0079 (12)	−0.0197 (13)
O2	0.0377 (12)	0.0439 (12)	0.0578 (16)	0.0002 (10)	0.0138 (12)	−0.0144 (11)
O3	0.0600 (15)	0.0385 (13)	0.0772 (19)	−0.0097 (11)	0.0352 (14)	−0.0068 (12)
O4	0.0460 (14)	0.0407 (13)	0.0689 (18)	−0.0055 (10)	0.0252 (13)	−0.0074 (12)

Geometric parameters (Å, °)

C1—O1	1.224 (3)	C10—C11	1.512 (4)

C1—O2	1.279 (3)	C10—H10A	0.9700
C1—C2	1.520 (4)	C10—H10B	0.9700
C2—C3	1.393 (4)	C11—C12	1.516 (4)
C2—C7	1.407 (4)	C11—H11A	0.9700
C3—C4	1.386 (4)	C11—H11B	0.9700
C3—H3	0.9300	C12—N3	1.494 (3)
C4—C5	1.375 (4)	C12—H12A	0.9700
C4—H4	0.9300	C12—H12B	0.9700
C5—C6	1.379 (4)	C13—N3	1.489 (3)
C5—H5	0.9300	C13—H13A	0.9600
C6—C7	1.399 (4)	C13—H13B	0.9600
C6—H6	0.9300	C13—H13C	0.9600
C7—N1	1.408 (3)	C14—N3	1.485 (4)
C8—C9	1.534 (4)	C14—H14A	0.9600
C8—O3	1.217 (3)	C14—H14B	0.9600
C9—O4	1.222 (3)	C14—H14C	0.9600
C8—N1	1.345 (3)	N1—H1	0.8600
C9—N2	1.325 (3)	N2—H2	0.8600
C10—N2	1.451 (3)	N3—H3A	0.912 (19)
O1—C1—O2	125.0 (3)	C12—C11—H11A	109.6
O1—C1—C2	119.3 (3)	C10—C11—H11B	109.6
O2—C1—C2	115.7 (3)	C12—C11—H11B	109.6
C3—C2—C7	118.4 (3)	H11A—C11—H11B	108.1
C3—C2—C1	118.0 (3)	N3—C12—C11	113.0 (2)
C7—C2—C1	123.5 (2)	N3—C12—H12A	109.0
C4—C3—C2	121.4 (3)	C11—C12—H12A	109.0
C4—C3—H3	119.3	N3—C12—H12B	109.0
C2—C3—H3	119.3	C11—C12—H12B	109.0
C5—C4—C3	119.4 (3)	H12A—C12—H12B	107.8
C5—C4—H4	120.3	N3—C13—H13A	109.5
C3—C4—H4	120.3	N3—C13—H13B	109.5
C4—C5—C6	120.9 (3)	H13A—C13—H13B	109.5
C4—C5—H5	119.6	N3—C13—H13C	109.5
C6—C5—H5	119.6	H13A—C13—H13C	109.5
C5—C6—C7	120.1 (3)	H13B—C13—H13C	109.5
C5—C6—H6	120.0	N3—C14—H14A	109.5
C7—C6—H6	120.0	N3—C14—H14B	109.5
C6—C7—C2	119.7 (3)	H14A—C14—H14B	109.5
C6—C7—N1	121.0 (3)	N3—C14—H14C	109.5
C2—C7—N1	119.3 (3)	H14A—C14—H14C	109.5
O3—C8—N1	126.7 (3)	H14B—C14—H14C	109.5
O3—C8—C9	121.6 (3)	C8—N1—C7	127.7 (2)
N1—C8—C9	111.6 (3)	C8—N1—H1	116.2
O4—C9—N2	125.0 (3)	C7—N1—H1	116.2
O4—C9—C8	121.9 (3)	C9—N2—C10	122.5 (3)
N2—C9—C8	113.0 (3)	C9—N2—H2	118.8
N2—C10—C11	110.3 (3)	C10—N2—H2	118.8
N2—C10—H10A	109.6	C14—N3—C13	110.1 (2)
C11—C10—H10A	109.6	C14—N3—C12	114.5 (2)

N2—C10—H10B	109.6	C13—N3—C12	110.8 (2)
C11—C10—H10B	109.6	C14—N3—H3A	107.4 (18)
H10A—C10—H10B	108.1	C13—N3—H3A	106.8 (17)
C10—C11—C12	110.4 (2)	C12—N3—H3A	107.0 (18)
C10—C11—H11A	109.6		
O1—C1—C2—C3	−23.4 (4)	O3—C8—C9—O4	−177.5 (3)
O2—C1—C2—C3	154.9 (3)	O3—C8—C9—N2	3.5 (4)
O1—C1—C2—C7	159.7 (3)	N1—C8—C9—N2	−174.2 (3)
O2—C1—C2—C7	−22.0 (4)	N1—C8—C9—O4	4.8 (4)
C7—C2—C3—C4	1.9 (4)	N2—C10—C11—C12	−173.0 (2)
C1—C2—C3—C4	−175.2 (3)	C10—C11—C12—N3	−167.4 (3)
C2—C3—C4—C5	−1.5 (5)	O3—C8—N1—C7	−2.7 (5)
C3—C4—C5—C6	−0.3 (5)	C9—C8—N1—C7	174.9 (3)
C4—C5—C6—C7	1.6 (5)	C2—C7—N1—C8	−154.0 (3)
C5—C6—C7—C2	−1.1 (4)	C6—C7—N1—C8	27.0 (4)
C5—C6—C7—N1	177.8 (3)	O4—C9—N2—C10	0.0 (5)
C3—C2—C7—C6	−0.6 (4)	C8—C9—N2—C10	178.9 (3)
C1—C2—C7—C6	176.3 (3)	C11—C10—N2—C9	151.3 (3)
C3—C2—C7—N1	−179.6 (2)	C11—C12—N3—C14	−58.1 (3)
C1—C2—C7—N1	−2.7 (4)	C11—C12—N3—C13	176.7 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	1.98	2.637 (3)	132
N3—H3A···O2^{i}	0.912 (19)	1.693 (18)	2.604 (3)	177 (3)

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

Fig. 1

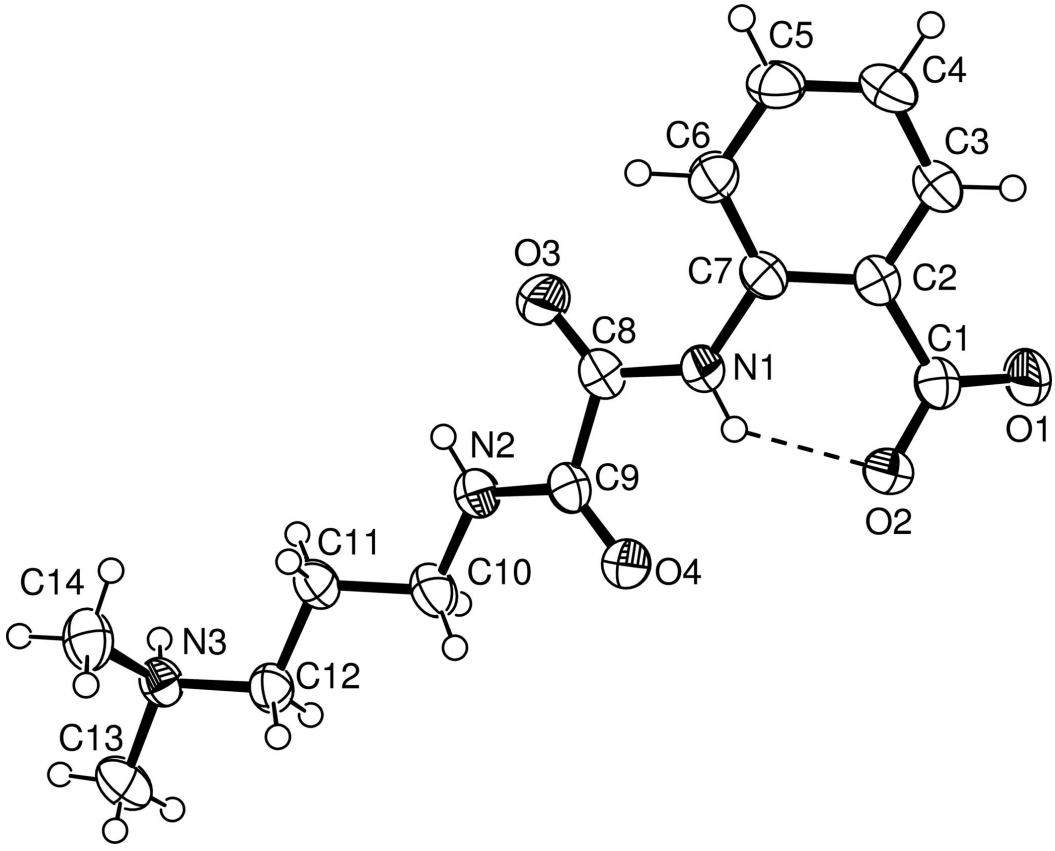

Fig. 2