Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[(2-Ammonioethyl)aminocarbonyl]-benzoate hemihydrate

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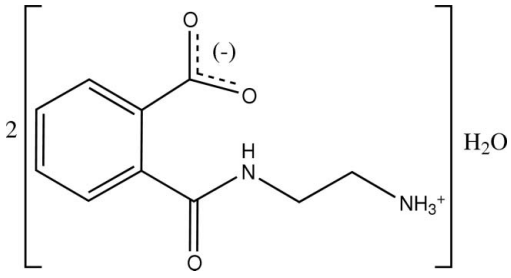
Received 30 April 2007; accepted 1 May 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.010 Å; R factor = 0.050; wR factor = 0.153; data-to-parameter ratio = 6.6.

The title amino acid-like structure, $C_{10}H_{12}N_2O_3\cdot0.5H_2O$, consists of two zwitterionic residues and one water molecule in the asymmetric unit. The carboxylate group is twisted from the plane of the attached benzene ring by angles of 35.6 (4) and 36.2 (5)° in the two residues. From the benzene ring, the side-chain conformations are observed to be *trans*/*gauche*-I/*gauche*-II and *trans*/*gauche*-II/*gauche*-II. The crystal structure is stabilized by an intricate three-dimensional hydrogen-bonding network. The amino and carboxylate groups are connected through intra- and intermolecular hydrogen bonds, forming $S(10)$, $C(4)$, $C_2^1(4)$ and $C_3^3(8)$ motifs. The chains run along the a axis of the unit cell. Hydrophobic layers across z = $\frac{1}{4}$ and $\frac{3}{4}$ are sandwiched between the hydrophilic layers across z = $\frac{1}{2}$ and 1.

Related literature

For related literature on hydrogen-bond motifs, see: Etter *et al.* (1990). For related literature on values of bond lengths and angles, see: Allen (1987). For information on the importance of this type of compounds in chemical syntheses and reactions, see: Salemme (1977); McCord & Fridovich, (1969); Fujimori *et al.* (2006); Tahara *et al.* (2007); Van Kuilenburg *et al.* (2006) and March (1977). For a related structure, see: Anitha *et al.* (2005).



Experimental

Crystal data

$C_{10}H_{12}N_2O_3\cdot0.5H_2O$
M_r = 217.23
Orthorhombic, $Pca2_1$
a = 9.0609 (9) Å
b = 8.6714 (12) Å
c = 27.0214 (19) Å
V = 2123.1 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.23 × 0.19 × 0.15 mm

Data collection

Nonius MACH3 sealed-tube diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.983, T_{max} = 0.998
1986 measured reflections
1905 independent reflections
1430 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.153
S = 1.15
1905 reflections
288 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11···O17^{i}	0.86	2.03	2.843 (7)	157
N12—H12A···O21^{i}	0.89	2.61	3.397 (8)	148
N12—H12B···O11	0.89	2.07	2.920 (7)	158
N12—H12C···O11^{ii}	0.89	2.27	3.035 (8)	144
N21—H21···O27^{iii}	0.86	2.01	2.831 (7)	159
N22—H22A···O1W^{iii}	0.89	1.88	2.768 (7)	174
N22—H22B···O12^{iii}	0.89	1.82	2.706 (8)	171
N22—H22C···O21	0.89	2.00	2.804 (7)	149
O1W—H1W···O21	0.94 (5)	1.93 (8)	2.782 (7)	149 (12)
O1W—H2W···O22^{i}	0.92 (5)	1.82 (5)	2.706 (7)	160 (9)

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL/PC* (Bruker, 2000); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

SA and SN thank the Department of Science and Technology, Government of India, for establishing the single-crystal diffractometer facility at the School of Physics, Madurai Kamaraj University, Madurai, through the FIST programme, and the University Grants Commision for the SAP programme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2313).

 doi:10.1107/S1600536807021599

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supplementary materials

Acta Cryst. (2007). E**63**, o2875-o2876 [**doi:10.1107/S1600536807021599**]

2-[(2-Ammonioethyl)aminocarbonyl]benzoate hemihydrate

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Comment

Amino acids are classic compounds and exhibit various interesting biological activities. As enzymes (biopolymers of α-aminoacids), they catalyze various biochemical transformation with high efficiency by adopting appropriate aminoacid sequences (Salemme, 1977; McCord & Fridovich, 1969). The monomer γ-aminobutyric acid (GABA) serves as neurotransmitter while δ-aminoolevulinic acid is involved in the biosynthesis of heme unit (Fujimori *et al.*, 2006; Tahara *et al.*, 2007). β-amino-iso-butyric acid is a metabolite in the catabolism of pyrimidine bases (Van Kuilenburg *et al.*, 2006). In addition, the aminoacids being bifunctional in nature can act as synthons for a variety of organic compounds (March, 1977). The title compound, (I) is an amino acid-like structure containing a protonated ammonium (NH_3^+) and a deprotonated carboxylate (COO^-) groups with the amide group in the middle.

The aymmetric part of the unit cell contains two zwitterionic residues (A & B) of 2-[(2-Ammonium-ethylamino)-carbonyl]-benzoate and one lattice water molecule (Fig 1). In both the residues of A and B, the carboxylate group is twisted from the plane of the attached benzene ring with the angles of 35.6 (4) and 36.2 (5)°, respectively. As the title compound has amino acid-like structure it is more appropriate to detail the conformation in the same nomenclature followed in amino acid complexes (Allen, 2002). Thus, from the phenyl ring, the side chain conformations are observed to be *trans*/*gauche* I/ *gauche* II and *trans*/*gauche* II/*gauche* II for residues A and B, respectively (Table 1). The C–O bond distances in the carboxylate groups are nearly equal and lie midway between the usual single and double C–O bond distances as found in many zwitterionic amino acid complexes (Anitha *et al.*, 2005).

In both the residues, the side chain is folded back to form intramolecular ring motif of S(10). This amino-carboxylate interaction is very similar to the head-to-tail sequence observed in amino acid complexes. The crystal structure is stabilized by an intricate three-dimensional hydrogen bonding network (Fig 2). N11—H11···O17 and N21—H21···O27 H-bond interactions in residues A & B respectively, lead to C(4) chain motifs running along the *a* axis of the unit cell. In residue A, intramolecular N12—H12B···O11 hydrogen bond and an intermolecular N12—H12C···O11 hydrogen bond lead to a distinct $C_2^1(4)$ chain motif running along the a axis of the unit cell. In residue B, the amino and carboxylate groups interact through the water molecule forming a $C_3^3(8)$ chain motif. Apart from these, the residues A and B are connected through other N—H···O hydrogen bonds bringing amino and carboxylate groups to close proximity in the unit cell thereby forming hydrophilic layers across z = 1/2 and 1 with hydrophobic layers across z = 1/4 and 3/4. These alternate hydrophobic and hydrophilic layers are often observed in amino acid complexes (e.g., Anitha *et al.*, 2005).

Experimental

Diethylphthalate was added to stoichiometric excess of ethylenediamine in ether. The mixture was refluxed for 3 h and then evaporated on a water bath yielding a jelly-like mass which was dissolved in water and evaporated once again after washing with chloroform. The resulting mass when treated with ethanol to form a precipitate which was filtered, washed with ether

and dried in a vacuum dessicator. The dried precipitate was dissolved in water and, on slow evaporation of this aqueous solution, the title compound was obtained as colourless blocks (yield: 70%; melting point: 160°).

Refinement

The H atoms of the water molecules were located in a difference Fourier map and refined isotropically. All the other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 (aromatic) or 0.97 (CH_2) Å and N—H = 0.86 (NH) or 0.89 (NH_3) Å with $U_{iso}(H) = 1.2U_{eq}$ (parent atom) for aromatic, CH_2 or NH, or $1.5U_{eq}$ (parent atom) for NH_3. In addition to the 1905 unique reflections, 81 Friedel pairs were measured. However, owing to the abosence of atoms with significant anamalous dispersion effects, these data were merged.

Figures

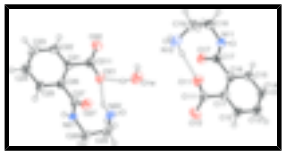

Fig. 1. The molecular structure of the title compound (I) with the atom numbering scheme and 50% probability displacement ellipsoids. H-bonds are shown as dashed lines.

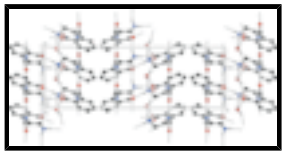

Fig. 2. Packing diagram of the molecules viewed down the b-axis. H atoms not involved in the H-bonds (dashed lines) are omitted for clarity.

2-(2-Ammonioethylaminocarbonyl)benzoate hemihydrate

Crystal data

$C_{10}H_{12}N_2O_3 \cdot 0.5H_2O$	$D_x = 1.359$ Mg m^{-3}
$M_r = 217.23$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Orthorhombic, $Pca2_1$	Cell parameters from 25 reflections
$a = 9.0609$ (9) Å	$\theta = 10.3$–12.8°
$b = 8.6714$ (12) Å	$\mu = 0.10$ mm^{-1}
$c = 27.0214$ (19) Å	$T = 293$ (2) K
$V = 2123.1$ (4) Å^3	Block, colourless
$Z = 8$	0.23 × 0.19 × 0.15 mm
$F_{000} = 920$	

Data collection

Nonius MACH3 sealed tube diffractometer	$R_{int} = 0.015$
Radiation source: fine-focus sealed tube	$\theta_{max} = 24.9$°
Monochromator: graphite	$\theta_{min} = 2.4$°
$T = 293(2)$ K	$h = -10 \rightarrow 0$
ω–2θ scans	$k = -10 \rightarrow 0$

Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.983, T_{max} = 0.998
1986 measured reflections
1905 independent reflections
1430 reflections with $I > 2\sigma(I)$

$l = -1 \rightarrow 32$
3 standard reflections
every 60 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.153$
$S = 1.15$
1905 reflections
288 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 1.9315P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
C11	0.0958 (7)	0.8470 (7)	0.4129 (3)	0.0314 (16)
C111	0.1390 (7)	0.8364 (8)	0.4668 (3)	0.0369 (16)
O11	0.1460 (6)	0.7046 (6)	0.48551 (18)	0.0524 (13)
O12	0.1588 (8)	0.9593 (6)	0.4879 (2)	0.074 (2)
C12	0.0061 (8)	0.9674 (7)	0.3967 (3)	0.0384 (16)
H12	−0.0214	1.0451	0.4185	0.046*
C13	−0.0423 (9)	0.9717 (9)	0.3481 (3)	0.0472 (19)
H13	−0.1049	1.0504	0.3378	0.057*
C14	0.0006 (8)	0.8629 (9)	0.3157 (3)	0.0472 (19)

H14	−0.0314	0.8677	0.2831	0.057*
C15	0.0909 (8)	0.7453 (8)	0.3305 (3)	0.0394 (17)
H15	0.1204	0.6716	0.3075	0.047*
C16	0.1391 (7)	0.7337 (7)	0.3788 (2)	0.0285 (14)
C17	0.2387 (8)	0.6051 (7)	0.3933 (3)	0.0327 (15)
O17	0.3729 (5)	0.6252 (5)	0.3991 (2)	0.0405 (12)
N11	0.1742 (6)	0.4662 (6)	0.3996 (2)	0.0338 (13)
H11	0.0817	0.4580	0.3930	0.041*
C18	0.2517 (8)	0.3301 (7)	0.4167 (3)	0.0396 (17)
H18A	0.3518	0.3322	0.4040	0.048*
H18B	0.2034	0.2393	0.4034	0.048*
C19	0.2568 (9)	0.3176 (8)	0.4715 (3)	0.0502 (19)
H19A	0.1567	0.3202	0.4842	0.060*
H19B	0.2990	0.2186	0.4803	0.060*
N12	0.3421 (7)	0.4391 (7)	0.4955 (2)	0.0555 (16)
H12A	0.3406	0.4250	0.5282	0.083*
H12B	0.3028	0.5305	0.4883	0.083*
H12C	0.4349	0.4360	0.4848	0.083*
C21	0.9036 (7)	0.6445 (7)	0.6746 (3)	0.0307 (15)
C211	0.8532 (7)	0.6411 (8)	0.6216 (3)	0.0374 (17)
O22	0.8559 (7)	0.5169 (6)	0.5986 (2)	0.0663 (17)
O21	0.8076 (6)	0.7632 (5)	0.60255 (17)	0.0516 (14)
C22	0.9971 (7)	0.5287 (7)	0.6914 (3)	0.0384 (16)
H22	1.0258	0.4504	0.6700	0.046*
C23	1.0483 (8)	0.5286 (8)	0.7402 (3)	0.0457 (19)
H23	1.1119	0.4512	0.7509	0.055*
C24	1.0048 (8)	0.6429 (9)	0.7725 (3)	0.049 (2)
H24	1.0389	0.6422	0.8049	0.059*
C25	0.9078 (8)	0.7622 (8)	0.7564 (2)	0.0372 (17)
H25	0.8767	0.8385	0.7782	0.045*
C26	0.8603 (7)	0.7620 (7)	0.7069 (2)	0.0289 (15)
C27	0.7617 (7)	0.8920 (6)	0.6915 (2)	0.0266 (13)
O27	0.6264 (5)	0.8757 (5)	0.6901 (2)	0.0443 (12)
C28	0.7510 (8)	1.1631 (7)	0.6643 (3)	0.0399 (17)
H28A	0.8188	1.2496	0.6643	0.048*
H28B	0.6719	1.1865	0.6873	0.048*
N21	0.8299 (5)	1.0244 (6)	0.68173 (19)	0.0309 (13)
H21	0.9239	1.0294	0.6858	0.037*
C29	0.6868 (8)	1.1457 (9)	0.6131 (3)	0.053 (2)
H29A	0.5994	1.0814	0.6150	0.064*
H29B	0.6567	1.2463	0.6012	0.064*
N22	0.7921 (6)	1.0761 (6)	0.5768 (2)	0.0480 (14)
H22A	0.8650	1.1423	0.5708	0.072*
H22B	0.7447	1.0552	0.5488	0.072*
H22C	0.8292	0.9894	0.5893	0.072*
O1W	0.5242 (6)	0.7194 (6)	0.5655 (2)	0.0600 (14)
H1W	0.600 (11)	0.751 (12)	0.587 (4)	0.15 (5)*
H2W	0.485 (9)	0.625 (7)	0.574 (4)	0.10 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.025 (3)	0.034 (4)	0.035 (4)	−0.003 (3)	0.005 (3)	0.008 (3)
C111	0.041 (4)	0.036 (4)	0.034 (4)	0.005 (3)	−0.009 (3)	−0.001 (3)
O11	0.073 (3)	0.047 (3)	0.037 (3)	0.014 (3)	−0.007 (2)	0.003 (2)
O12	0.117 (6)	0.048 (3)	0.057 (4)	−0.008 (3)	−0.032 (4)	−0.012 (3)
C12	0.041 (4)	0.033 (4)	0.041 (4)	0.001 (3)	−0.004 (4)	0.008 (3)
C13	0.051 (4)	0.038 (4)	0.052 (4)	0.001 (4)	−0.016 (4)	0.018 (4)
C14	0.045 (4)	0.057 (5)	0.039 (5)	−0.002 (4)	−0.016 (4)	0.016 (4)
C15	0.039 (4)	0.038 (4)	0.041 (4)	−0.006 (3)	0.001 (3)	−0.006 (3)
C16	0.025 (3)	0.030 (3)	0.031 (4)	−0.007 (3)	0.003 (3)	0.001 (3)
C17	0.031 (4)	0.035 (3)	0.032 (3)	0.002 (3)	−0.001 (3)	−0.008 (3)
O17	0.020 (2)	0.045 (3)	0.057 (3)	−0.003 (2)	−0.001 (2)	0.007 (3)
N11	0.021 (3)	0.033 (3)	0.048 (3)	−0.002 (2)	0.000 (3)	−0.001 (3)
C18	0.036 (3)	0.029 (3)	0.054 (5)	0.002 (3)	−0.005 (3)	0.003 (3)
C19	0.054 (4)	0.042 (4)	0.055 (5)	0.012 (3)	0.003 (4)	0.012 (3)
N12	0.062 (4)	0.046 (3)	0.058 (4)	0.008 (3)	−0.007 (3)	0.010 (3)
C21	0.030 (3)	0.026 (3)	0.036 (4)	−0.006 (3)	−0.007 (3)	−0.003 (3)
C211	0.030 (3)	0.041 (4)	0.041 (4)	0.004 (3)	−0.001 (3)	−0.001 (3)
O22	0.092 (4)	0.052 (3)	0.056 (3)	0.023 (3)	−0.021 (3)	−0.022 (3)
O21	0.073 (3)	0.044 (3)	0.038 (3)	0.010 (2)	−0.014 (3)	0.002 (2)
C22	0.033 (3)	0.027 (3)	0.056 (5)	0.001 (3)	0.000 (4)	−0.002 (3)
C23	0.040 (4)	0.041 (4)	0.057 (5)	0.011 (3)	0.000 (4)	0.016 (4)
C24	0.057 (5)	0.052 (5)	0.037 (5)	0.000 (4)	−0.007 (4)	0.012 (3)
C25	0.041 (4)	0.042 (4)	0.029 (4)	0.009 (3)	−0.009 (3)	0.006 (3)
C26	0.022 (3)	0.033 (3)	0.032 (4)	−0.002 (3)	−0.001 (3)	0.004 (3)
C27	0.021 (3)	0.033 (3)	0.026 (3)	−0.002 (3)	0.002 (3)	0.004 (3)
O27	0.024 (2)	0.044 (3)	0.065 (3)	−0.001 (2)	0.000 (2)	0.005 (3)
C28	0.033 (3)	0.029 (3)	0.058 (5)	0.004 (3)	0.009 (3)	0.001 (3)
N21	0.024 (3)	0.028 (3)	0.041 (3)	−0.001 (2)	−0.006 (2)	0.003 (2)
C29	0.037 (4)	0.058 (5)	0.065 (5)	0.009 (4)	−0.007 (4)	0.021 (4)
N22	0.050 (3)	0.044 (3)	0.050 (3)	−0.003 (3)	−0.011 (3)	0.011 (3)
O1W	0.046 (3)	0.061 (3)	0.073 (4)	−0.001 (3)	−0.003 (3)	0.014 (3)

Geometric parameters (Å, °)

C11—C12	1.394 (9)	C21—C26	1.397 (8)
C11—C16	1.403 (9)	C21—C211	1.503 (10)
C11—C111	1.512 (10)	C211—O22	1.245 (8)
C111—O12	1.222 (8)	C211—O21	1.249 (8)
C111—O11	1.252 (8)	C22—C23	1.398 (11)
C12—C13	1.383 (10)	C22—H22	0.9300
C12—H12	0.9300	C23—C24	1.378 (11)
C13—C14	1.344 (11)	C23—H23	0.9300
C13—H13	0.9300	C24—C25	1.425 (10)
C14—C15	1.368 (10)	C24—H24	0.9300
C14—H14	0.9300	C25—C26	1.406 (8)

C15—C16	1.379 (9)	C25—H25	0.9300
C15—H15	0.9300	C26—C27	1.497 (8)
C16—C17	1.487 (9)	C27—O27	1.235 (7)
C17—O17	1.239 (8)	C27—N21	1.330 (7)
C17—N11	1.349 (8)	C28—N21	1.476 (8)
N11—C18	1.449 (8)	C28—C29	1.507 (12)
N11—H11	0.8600	C28—H28A	0.9700
C18—C19	1.486 (10)	C28—H28B	0.9700
C18—H18A	0.9700	N21—H21	0.8600
C18—H18B	0.9700	C29—N22	1.495 (9)
C19—N12	1.459 (9)	C29—H29A	0.9700
C19—H19A	0.9700	C29—H29B	0.9700
C19—H19B	0.9700	N22—H22A	0.8900
N12—H12A	0.8900	N22—H22B	0.8900
N12—H12B	0.8900	N22—H22C	0.8900
N12—H12C	0.8900	O1W—H1W	0.94 (5)
C21—C22	1.390 (9)	O1W—H2W	0.92 (5)
C12—C11—C16	118.7 (6)	C22—C21—C211	118.8 (6)
C12—C11—C111	119.9 (6)	C26—C21—C211	121.6 (6)
C16—C11—C111	121.3 (6)	O22—C211—O21	122.2 (7)
O12—C111—O11	126.9 (7)	O22—C211—C21	119.2 (6)
O12—C111—C11	115.8 (6)	O21—C211—C21	118.5 (6)
O11—C111—C11	117.2 (6)	C21—C22—C23	120.7 (6)
C13—C12—C11	120.2 (7)	C21—C22—H22	119.6
C13—C12—H12	119.9	C23—C22—H22	119.6
C11—C12—H12	119.9	C24—C23—C22	120.1 (6)
C14—C13—C12	120.5 (7)	C24—C23—H23	120.0
C14—C13—H13	119.8	C22—C23—H23	120.0
C12—C13—H13	119.8	C23—C24—C25	120.4 (7)
C13—C14—C15	120.4 (7)	C23—C24—H24	119.8
C13—C14—H14	119.8	C25—C24—H24	119.8
C15—C14—H14	119.8	C26—C25—C24	118.5 (7)
C14—C15—C16	121.3 (7)	C26—C25—H25	120.7
C14—C15—H15	119.3	C24—C25—H25	120.7
C16—C15—H15	119.3	C21—C26—C25	120.6 (6)
C15—C16—C11	118.8 (6)	C21—C26—C27	122.9 (6)
C15—C16—C17	119.7 (6)	C25—C26—C27	116.5 (6)
C11—C16—C17	121.4 (6)	O27—C27—N21	123.6 (6)
O17—C17—N11	122.3 (6)	O27—C27—C26	121.0 (5)
O17—C17—C16	121.6 (6)	N21—C27—C26	115.3 (5)
N11—C17—C16	116.1 (6)	N21—C28—C29	113.5 (6)
C17—N11—C18	123.9 (5)	N21—C28—H28A	108.9
C17—N11—H11	118.0	C29—C28—H28A	108.9
C18—N11—H11	118.0	N21—C28—H28B	108.9
N11—C18—C19	113.2 (6)	C29—C28—H28B	108.9
N11—C18—H18A	108.9	H28A—C28—H28B	107.7
C19—C18—H18A	108.9	C27—N21—C28	122.8 (5)
N11—C18—H18B	108.9	C27—N21—H21	118.6
C19—C18—H18B	108.9	C28—N21—H21	118.6

H18A—C18—H18B	107.8	N22—C29—C28	113.3 (5)
N12—C19—C18	114.0 (7)	N22—C29—H29A	108.9
N12—C19—H19A	108.7	C28—C29—H29A	108.9
C18—C19—H19A	108.7	N22—C29—H29B	108.9
N12—C19—H19B	108.7	C28—C29—H29B	108.9
C18—C19—H19B	108.7	H29A—C29—H29B	107.7
H19A—C19—H19B	107.6	C29—N22—H22A	109.5
C19—N12—H12A	109.5	C29—N22—H22B	109.5
C19—N12—H12B	109.5	H22A—N22—H22B	109.5
H12A—N12—H12B	109.5	C29—N22—H22C	109.5
C19—N12—H12C	109.5	H22A—N22—H22C	109.5
H12A—N12—H12C	109.5	H22B—N22—H22C	109.5
H12B—N12—H12C	109.5	H1W—O1W—H2W	112 (7)
C22—C21—C26	119.6 (6)		
C12—C11—C111—O12	−35.1 (9)	C22—C21—C211—O22	22.1 (9)
C16—C11—C111—O12	147.7 (7)	C26—C21—C211—O22	−158.8 (6)
C12—C11—C111—O11	142.7 (7)	C22—C21—C211—O21	−159.2 (6)
C16—C11—C111—O11	−34.6 (9)	C26—C21—C211—O21	19.9 (10)
C16—C11—C12—C13	1.9 (10)	C26—C21—C22—C23	−0.1 (10)
C111—C11—C12—C13	−175.5 (6)	C211—C21—C22—C23	179.1 (6)
C11—C12—C13—C14	−2.2 (11)	C21—C22—C23—C24	0.9 (11)
C12—C13—C14—C15	1.0 (11)	C22—C23—C24—C25	−0.2 (11)
C13—C14—C15—C16	0.7 (11)	C23—C24—C25—C26	−1.2 (11)
C14—C15—C16—C11	−1.0 (10)	C22—C21—C26—C25	−1.4 (9)
C14—C15—C16—C17	−179.3 (6)	C211—C21—C26—C25	179.5 (6)
C12—C11—C16—C15	−0.3 (9)	C22—C21—C26—C27	179.1 (6)
C111—C11—C16—C15	177.0 (6)	C211—C21—C26—C27	0.0 (9)
C12—C11—C16—C17	177.9 (6)	C24—C25—C26—C21	2.0 (10)
C111—C11—C16—C17	−4.8 (9)	C24—C25—C26—C27	−178.4 (6)
C15—C16—C17—O17	102.6 (8)	C21—C26—C27—O27	81.8 (9)
C11—C16—C17—O17	−75.6 (8)	C25—C26—C27—O27	−97.7 (8)
C15—C16—C17—N11	−77.3 (8)	C21—C26—C27—N21	−100.9 (7)
C11—C16—C17—N11	104.5 (7)	C25—C26—C27—N21	79.6 (7)
O17—C17—N11—C18	4.2 (11)	O27—C27—N21—C28	−5.1 (11)
C16—C17—N11—C18	−175.9 (6)	C26—C27—N21—C28	177.7 (6)
C17—N11—C18—C19	86.8 (9)	C29—C28—N21—C27	−67.1 (9)
N11—C18—C19—N12	−65.4 (8)	N21—C28—C29—N22	−46.0 (8)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O17^{i}	0.86	2.03	2.843 (7)	157
N12—H12A···O21^{i}	0.89	2.61	3.397 (8)	148
N12—H12B···O11	0.89	2.07	2.920 (7)	158
N12—H12C···O11^{ii}	0.89	2.27	3.035 (8)	144
N21—H21···O27^{iii}	0.86	2.01	2.831 (7)	159
N22—H22A···O1W^{iii}	0.89	1.88	2.768 (7)	174
N22—H22B···O12^{iii}	0.89	1.82	2.706 (8)	171

N22—H22C···O21	0.89	2.00	2.804 (7)	149
O1W—H1W···O21	0.94 (5)	1.93 (8)	2.782 (7)	149 (12)
O1W—H2W···O22^{i}	0.92 (5)	1.82 (5)	2.706 (7)	160 (9)

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

Fig. 1

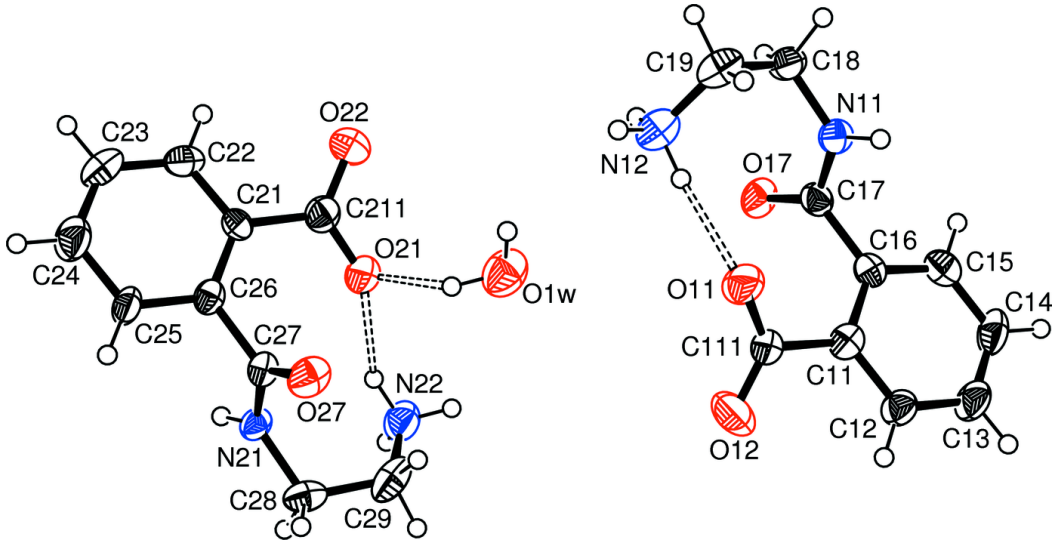
C19
C18
N11
N12
O17
C17
C16
C15
C14
O11
C111
C11
C12
C13
O12
C23
C22
O22
C21
C211
O21
O1w
C24
C26
C25
C27
O27
N22
N21
C28
C29

Fig. 2